

SIMULATED PXRD PATTERNS IN STUDIES OF THE PHASE COMPOSITION AND THERMAL BEHAVIOR OF BULK CRYSTALLINE SOLIDS

Shawn Yin, Raymond P. Scaringe, John Dimarco, and Jack Z. Gougoutas
Solid State Science Group, Pharmaceutical Research Institute,
Bristol-Myers Squibb Company

The discovery and identification of crystal forms is essential in the pharmaceutical industry because the crystal structure of the API (Active Pharmaceutical Ingredient) can affect efficacy, unit process operation, safety, and patent issues. Powder X-ray Diffraction (PXRD) is probably the most common technique used to identify the crystalline components of bulk materials. When atomic parameters are available from single crystal structure determination, the PXRD pattern expected for bulk samples can be simulated by standard methods.

Our presentation examines the use of simulated PXRD patterns as reference standards for each individual form (polymorph, solvates, salts, etc.). Small differences between the observed and simulated patterns can indicate the presence of different forms or be due to preferred orientation. In some cases, spinning capillary data are required to completely eliminate preferred orientation.

The simulated patterns also serve as reference standards for thermodynamic studies of bulk phase transformations at elevated temperature. The use of low-temperature single crystal data in the interpretation of higher temperature PXRD patterns will also be discussed.