

A UNIQUE CASE OF POLYMORPH CHARACTERIZATION BY PXRD

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The unit cells and crystal structures of the two polymorphic forms reported here display a number of similarities. Both forms contain dense layers of molecules with extensive hydrogen bonding/electrostatic interactions, while interactions between layers are relatively weak. The regions between layers also contain void spaces of ca. 50 Å³/molecule. The PXRD patterns of the two polymorphs are also similar but easily distinguished by a few unique low angle peaks in the range 5-10 degrees (2-theta); where one form has peaks the other form has none. There are other samples with closely related patterns, which with conventional instrumentation, exhibit either very weak, or no peaks in the range 5-10 degrees (2-theta), but the rest of the pattern is quite similar to the two known polymorphs. Further experiments with a high power/resolution diffractometer revealed small peaks in the 5-10 degrees (2-theta) region with *d*-spacings characteristic of one or both of the two polymorphs. In fact, such materials display a whole range of patterns with variable peak heights and widths in the low angle region. Based on single crystal diffraction patterns and powder pattern indexing, the observed variation can be understood as arising from materials containing varying amounts of layer stacking faults.