COMPARISON OF SPECIMEN PREPARATION TECHNIQUES FOR X-RAY POWDER DIFFRACTION DATA OF ALPHA TEGAFUR

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Different X-ray powder diffraction (XRPD) specimen preparation techniques were explored to compare the effect of preferred orientation in the XRPD data for the pure alpha Tegafur polymorph. Tegafur is the active ingredient in UFT, used in the first-line treatment of certain gastric and colo-rectal cancers. A commercial sample of Tegafur was recrystallized, and IR and DSC data were collected. A PANalytical X'Pert Pro MPD was used for the XRPD experiments. Three types of specimen preparation techniques were used: 1mm capillary, cavity mount, and zero-background. Scanning step sizes used were 0.0167°, 0.0090°, and 0.0167°, respectively. The DICVOL04 indexing and GSAS Rietveld refinement programs were used for XRPD data analysis.

The IR result is consistent with the literature reported data. The DSC data shows an endothermic melting point peak at 172 °C preceded by a thermal event indicating a polymorphic phase transition at about 161 °C. The indexing and refinement results of the XRPD patterns for pure alpha Tegafur are consistent with a triclinic crystal system and P-1 space group. The intensity ratios for crystal planes [110]/[2-20]/[121] are 80/100/60, 100/38/28, and 100/30/9 for the capillary, zero-background, and cavity mount data respectively. The preferred orientation texture indexes, determined by the refinement using spherical harmonics method, are 1.0, 2.4, and 8.0 for the capillary, and zero- background, and cavity mount samples respectively.

Recrystallized alpha Tegafur consists of needle shaped particles, elongated along the [110] axis of alpha Tegafur. This is the result of hydrogen-bonded dimer stacking being the preferred method of crystal growth. The preferred orientation phenomenon for XRPD analysis is particularly significant for the commonly used cavity mount (Bragg-Brentano) geometry. The intensity distribution of different specimen preparations showed that the capillary technique produced very little preferred orientation effect. However, the zero-background technique is the most time efficient one. The pros and cons of these three specimen preparation techniques and their XRPD experimental results will be discussed.