

An Investigation of the Polymorphism of a Potent Nonsteroidal Anti-inflammatory Drug Flunixin

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Crystal Growth

Example: 250 mg of FLX was suspended in 5 mL ethyl acetate. The mixture was stirred overnight and the remaining solid was removed by pipette filtration. A vial containing the clear solution was covered with perforated parafilm. Slow evaporation led to single crystals in about a week.

For slow cooling, a saturated solution in a vial covered by cover glass was concentrated to 1/2, 1/3, 1/4 of the original volume by gentle heating on a hot plate. The heat source was removed and the vial was sealed, whereupon the solution cooled to room temperature.

Crystal structure determination by single-crystal X-ray diffraction (SCXR)

Data collection was carried out at 90 K and 293 K on a Nonius kappaCCD diffractometer with MoK α radiation ($\lambda = 0.71073 \text{ \AA}$).¹ Cell refinement and data reduction were done using SCALEPACK and DENZO-SMN.² Structure solution and refinement were carried out using the SHELXS97 and SHELXL97 programs, respectively.^{3,4}

Powder X-ray diffraction

Powder X-ray diffraction (PXRD) data for each sample were collected on a Rigaku X-ray diffractometer with CuK α radiation (40 kV, 40 mA, $\lambda = 1.5406 \text{ \AA}$) between 5.0 - 50.0° (2 θ) at ambient temperatures. The finely ground sample was placed on a quartz plate in an aluminum holder.

Thermal Analysis

The thermal behavior of FLX was investigated by differential scanning calorimetry (DSC). These experiments were performed on TA Instruments DSCQ20-1250. For DSC, Tzero[®] pans and aluminum hermetic lids with a pinhole were used at a heating rate of 10 °C/min.

Spectroscopic Studies (FT-IR and Raman)

IR spectrum was recorded with the sample dispersed in KBr pellets using a Perkin-Elmer FT-IR spectrometer (Perkin Elmer, US), while Raman spectrum was recorded on a sample compressed in a gold-coated sample holder using a Thermo Electron DXR Laser Confocal Microscopy Raman Spectrometer (Thermo Fisher, US).

Figure S1 FT-IR spectra of the two forms of FLX

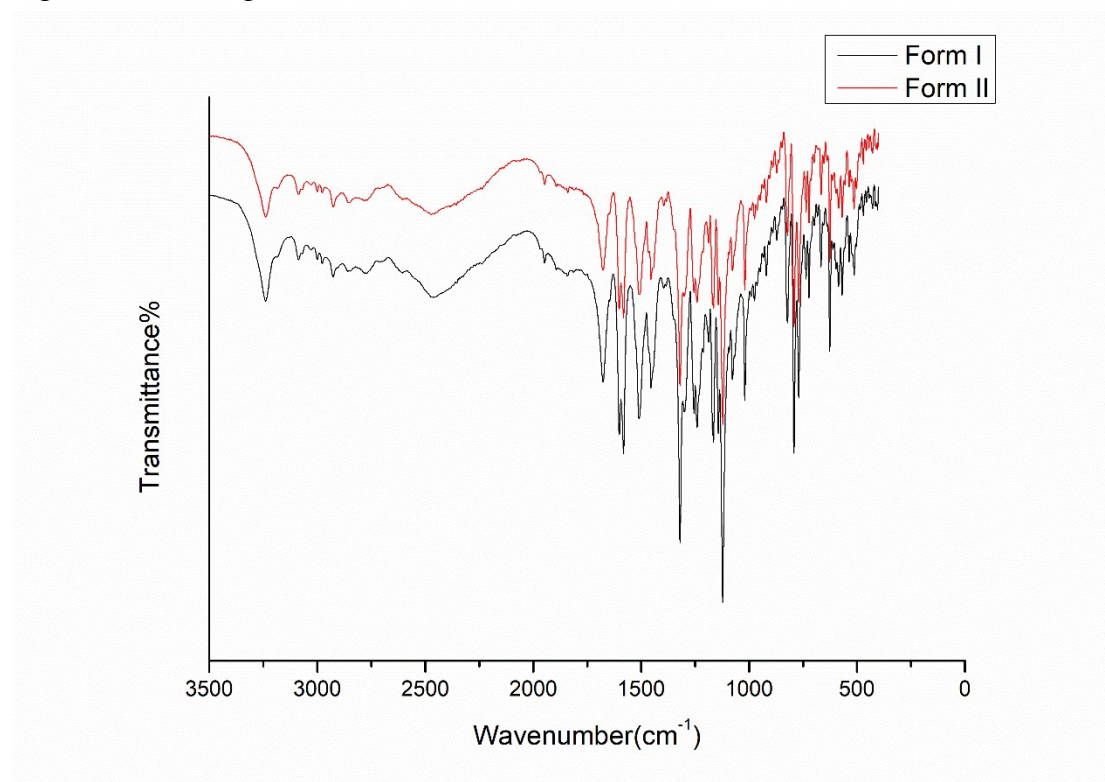
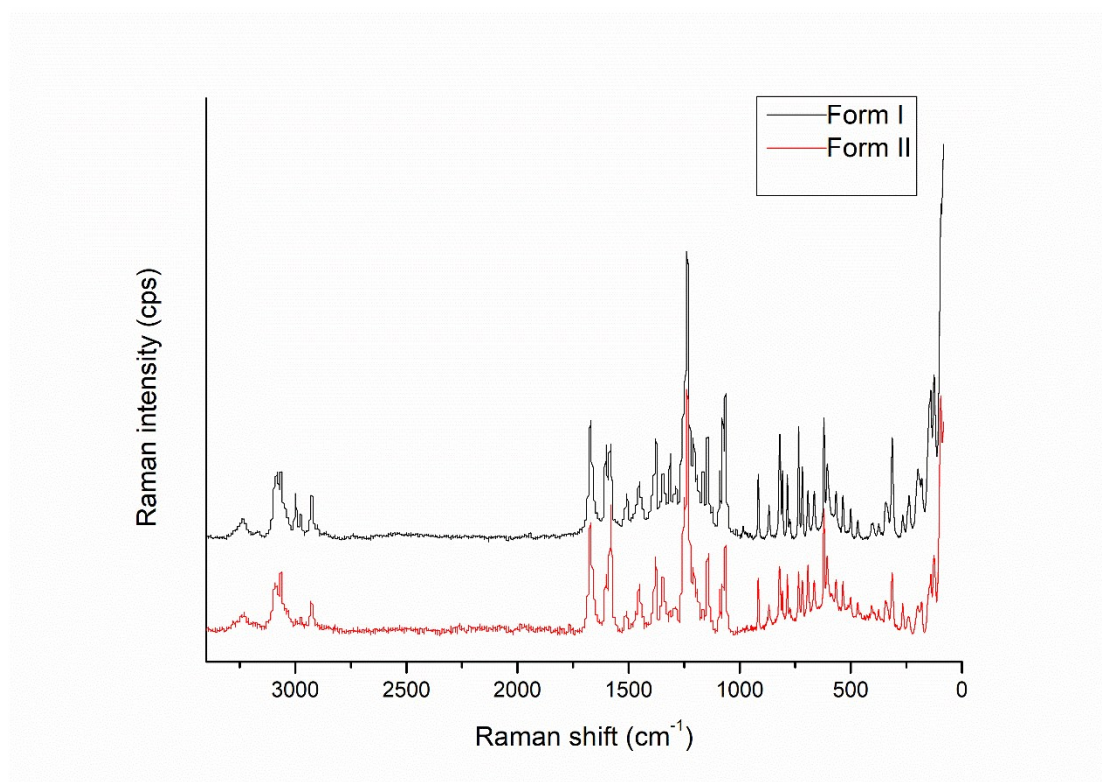


Figure S2 Raman spectra of the two forms of FLX



References

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