Dark Secrets of “Crystal Packing and Pharmaceutical Properties of Salts of Diclofenac”

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The aforementioned abstract, submitted to session 1.1.3 of this ACA meeting, compares the crystal packing motifs in 6 salts of the anti-inflammatory drug diclofenac and relates structure to pharmaceutical properties. Data for all 6 structures were collected at 120K with Mo radiation from a rotating anode generator to 27.5 deg in theta by highly experienced colleagues at the U.K. National Crystallography Service in Southampton. Three of the data sets, even from small crystals, look very good while the other 3 gave R(obs) > 0.1 with a low fraction of observed data.

Salt	Crystal size / mm	R(obs)	wR2	Max diff peak
tBut	0.08, 0.06, 0.02	0.1093	0.2942	0.60
cHex	0.20, 0.07, 0.01	0.1166	0.2843	0.69
Benz	0.28, 0.04, 0.02	0.1083	0.2082	0.47
Adam	0.32, 0.02, 0.02	0.0399	0.0845	0.27
AMP1	0.36, 0.26, 0.10	0.0584	0.1568	1.14
AMP2	0.09, 0.08, 0.06	0.0339	0.0820	0.33

Removing the (mostly unobserved) high-angle data above 25.25 deg for the first 3 structures changed the tabulated results to:

tBut		0.0979	0.2666	0.49
cHex		0.0962	0.2060	0.56
Benz		0.0945	0.1812	0.37

Shoehorning R(obs) below 0.1 is not just psychologically satisfying; future researchers may use these results as part of a larger study in structural systematics and may well apply a CSD filter of R < 0.1 . A conundrum remains about the large difference electron density peak in the AMP1 salt.