

Solid-solid transition between hydrated racemic compound and anhydrous conglomerate in Na-ibuprofen: a combined XRD, Solid-State NMR, calorimetric and computational study

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Supporting Information

Table S1. Crystal Data and Structure Refinement Parameters for RS-H as obtained from the SCD experiments performed at 15, 120, 150 and 298 K

	Temperature (K)			
	15	120	150	298
Formula	$C_{13}H_{21}O_4Na$			
M	264.29			
λ (Å)	0.71073			
Cell parameters (Å, °)	a = 5.7035(7) b = 5.9553(7) c = 23.535(2) α = 82.940(9) β = 84.447(9) γ = 63.14(1)	a = 5.7227(5) b = 5.9955(5) c = 23.661(2) α = 83.000(7) β = 89.351(7) γ = 63.032(9)	a = 5.7272(6) b = 6.0052(6) c = 23.723(2) α = 83.333(9) β = 89.081(9) γ = 63.06(1)	a = 5.7808(5) b = 6.0812(5) c = 23.998(2) α = 84.691(8) β = 89.788(8) γ = 62.863(9)
V (Å ³)	707.0(1)	717.3(1)	721.8(1)	746.5(1)
Crystal system, space group	Triclinic, P-1			
Z	2			
d (g/cm ³)	1.242	1.224	1.216	1.176
μ (mm ⁻¹)	0.116	0.114	0.113	0.109
F(000)	284			
θ range (°)	4.14-32.03	4.12-25.79	4.25-26.83	4.17-26.65
Limiting indices	-8<h<8 -7<k<8 -33<l<34	-6<h<6 -7<k<6 -28<l<28	-7<h<6 -7<k<7 -29<l<29	-7<h<7 -7<k<7 -28<l<29
GOF on F ²	1.097	1.076	1.125	1.055
Data/parameters	4437/174	2357/184	2584/184	2602/187
R ₁ [I>2σ(I)]	0.0723	0.0847	0.0818	0.0987
wR ₂ (all data)	0.1972	0.1979	0.1812	0.2379

Table S2. Crystal details from ref. 25.

Crystal data		
RS-H		a = 5.7396(4) Å; α = 83.457(1) $^\circ$ b = 6.0284(4) Å; β = 89.241(1) $^\circ$ c = 23.8301(17) Å; γ = 63.154(1) $^\circ$ V = 730.20(9) Å ³ T = 173 K d (g/cm ³) = 1.202
S-H		a = 5.7767(5) Å; α = 83.151(1) $^\circ$ b = 6.0835(5) Å; β = 86.482(1) $^\circ$ c = 23.880(2) Å; γ = 61.612(1) $^\circ$ V = 733.00(11) Å ³ T = 173 K d (g/cm ³) = 1.197
Selected bond distances (Å)		
	RS-H	S-H
Na1-O1	2.343	2.327; 2.300
Na2-O2	2.313	2.302; 2.313
Na1-O3w	2.371	2.404; 2.376
Na2-O4w	2.360	2.379; 2.397

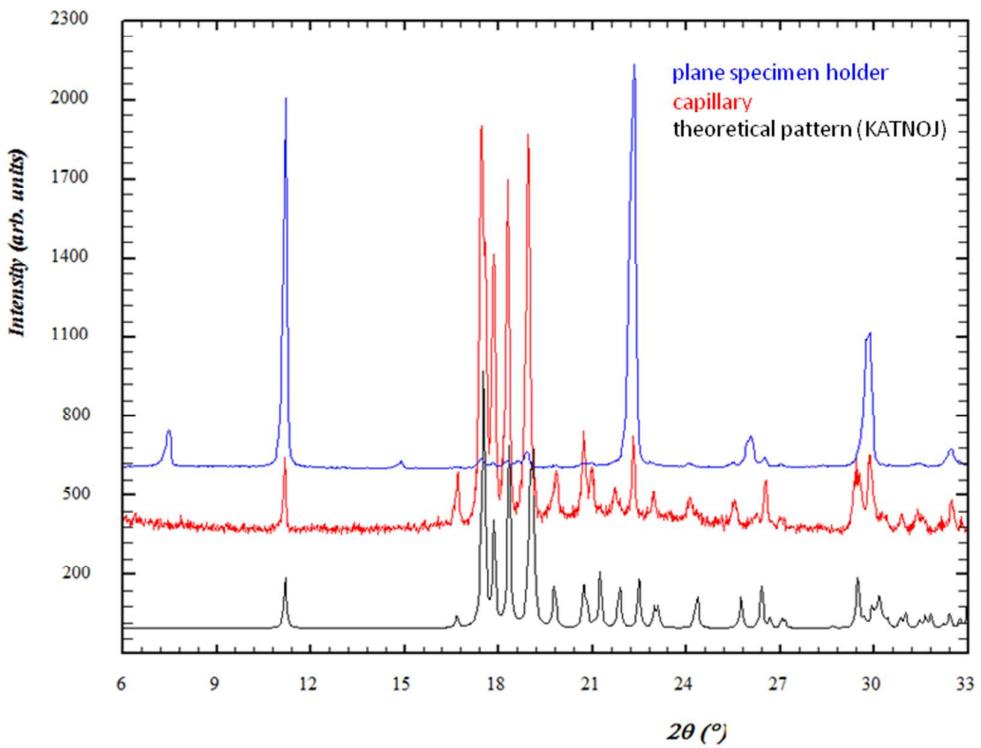


Figure S1. Comparison of the experimental XRPD patterns of S-H recorded in capillary, in a plane specimen holder and the theoretical one obtained from the single-crystal data.²⁵

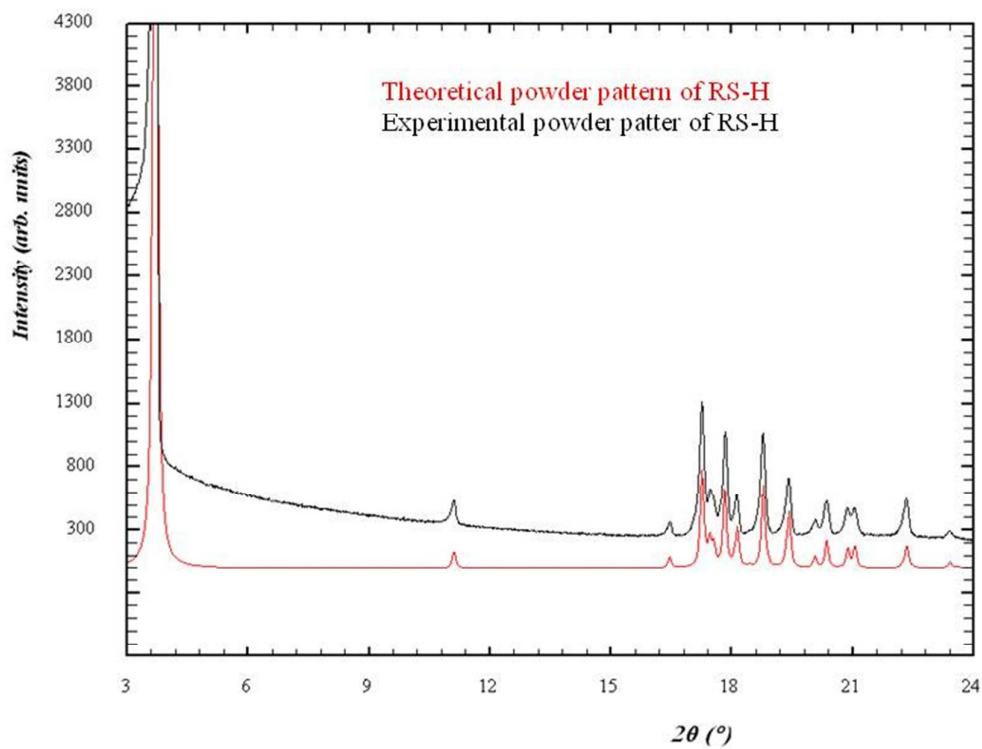


Figure S2. Comparison of the experimental XRPD patterns (after 1 hour exposure in air at rt) and the theoretical one obtained from the single-crystal data of the racemic sodium Ibuprofen di-hydrate phase collected at room temperature (RS-Hrt).

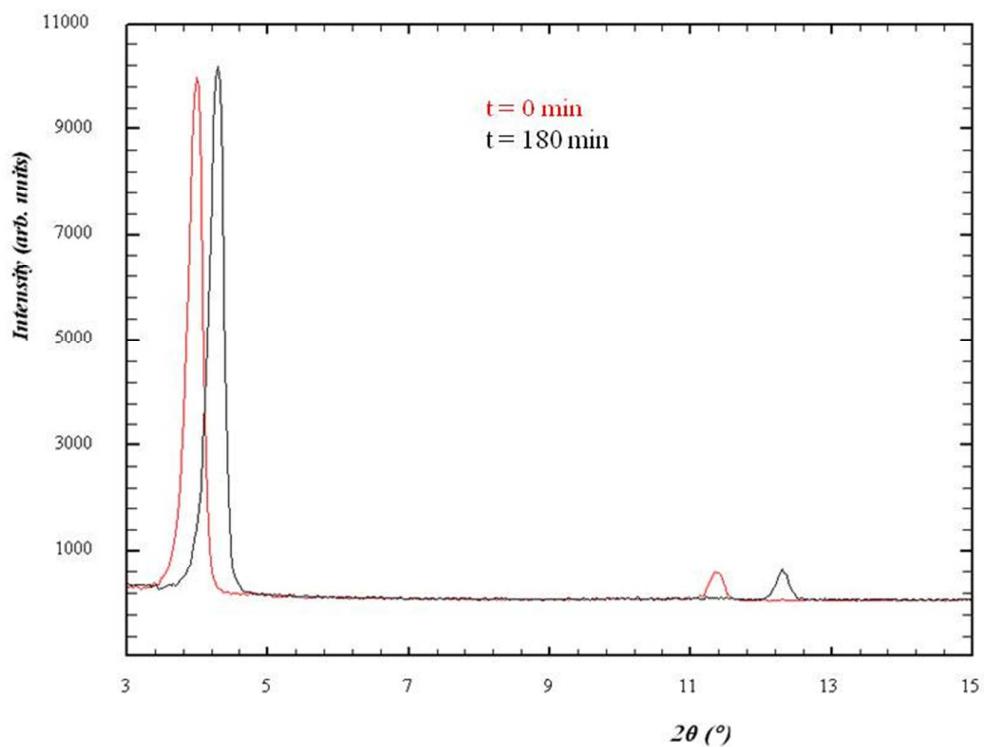


Figure S3. Comparison of the experimental XRPD patterns of RS-H ($t=0\text{min}$) and RS-A ($t=180\text{min}$, at rt in a closed chamber with P_2O_5 .

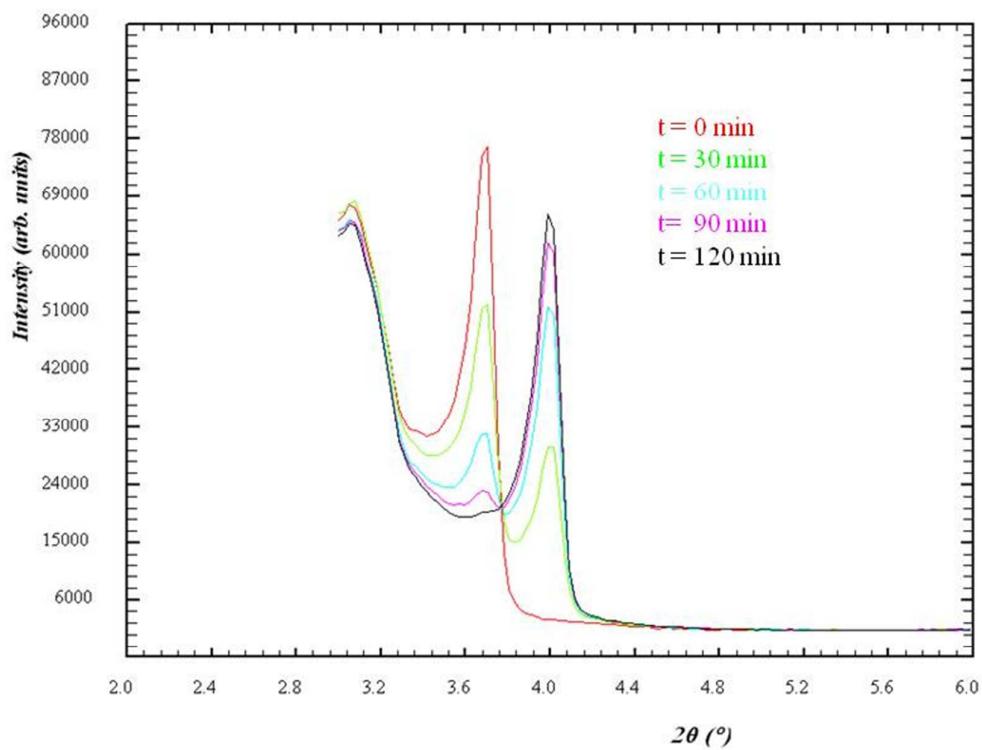


Figure S4. Experimental XRPD patterns of racemic sodium Ibuprofen recorded at rt under N_2 flow (RS-H, $t=0\text{min}$) (RS-A, $t=120\text{min}$).

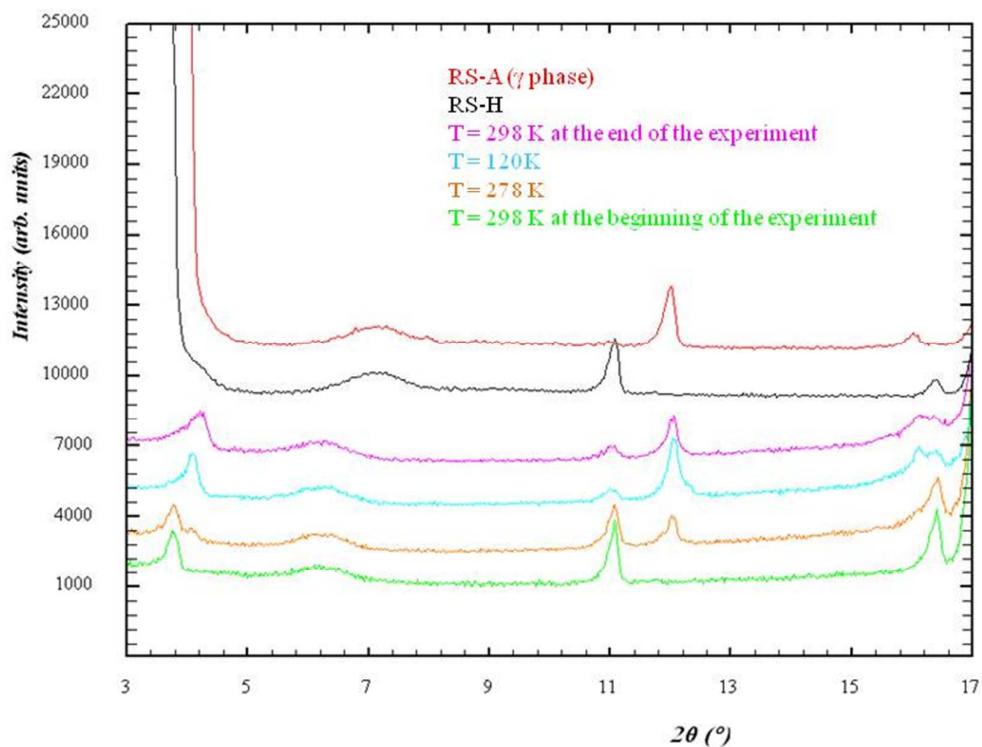


Figure S5. Experimental XRPD patterns of racemic sodium Ibuprofen recorded in vacuum at rt, 278, 120K and again at rt after heating. For comparison the patterns of RS-H and RS-A are also reported.