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Electronic Supporting Information

Solid-state Conformations of Pharmaceutical Polymorphs in Solution: Validation and Invalidation by NMR?

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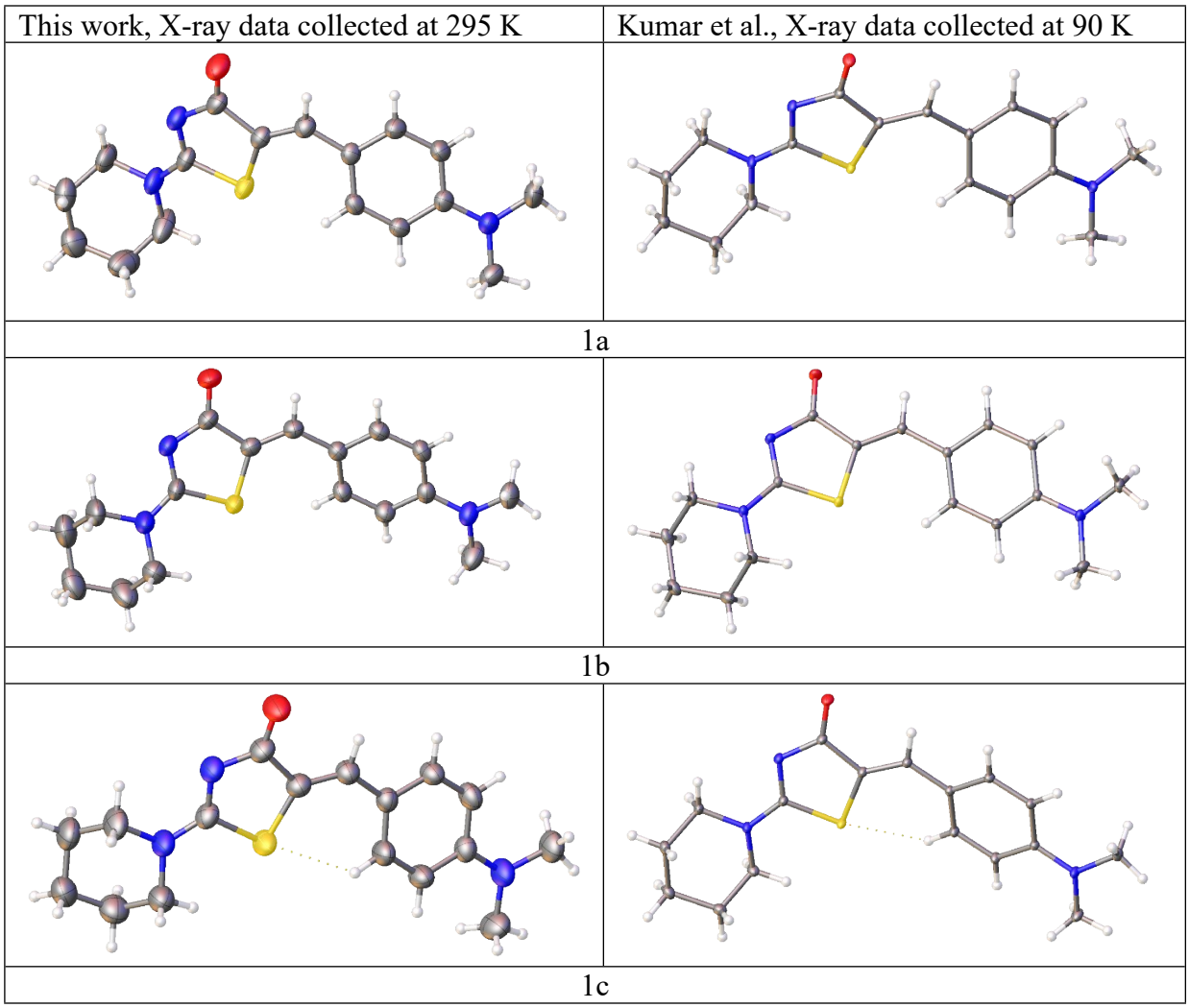

Figure S1. ORTEP of polymorphs 1a, 1b, and 1c at 50% probability level drawn in Olex2−1.3 program.

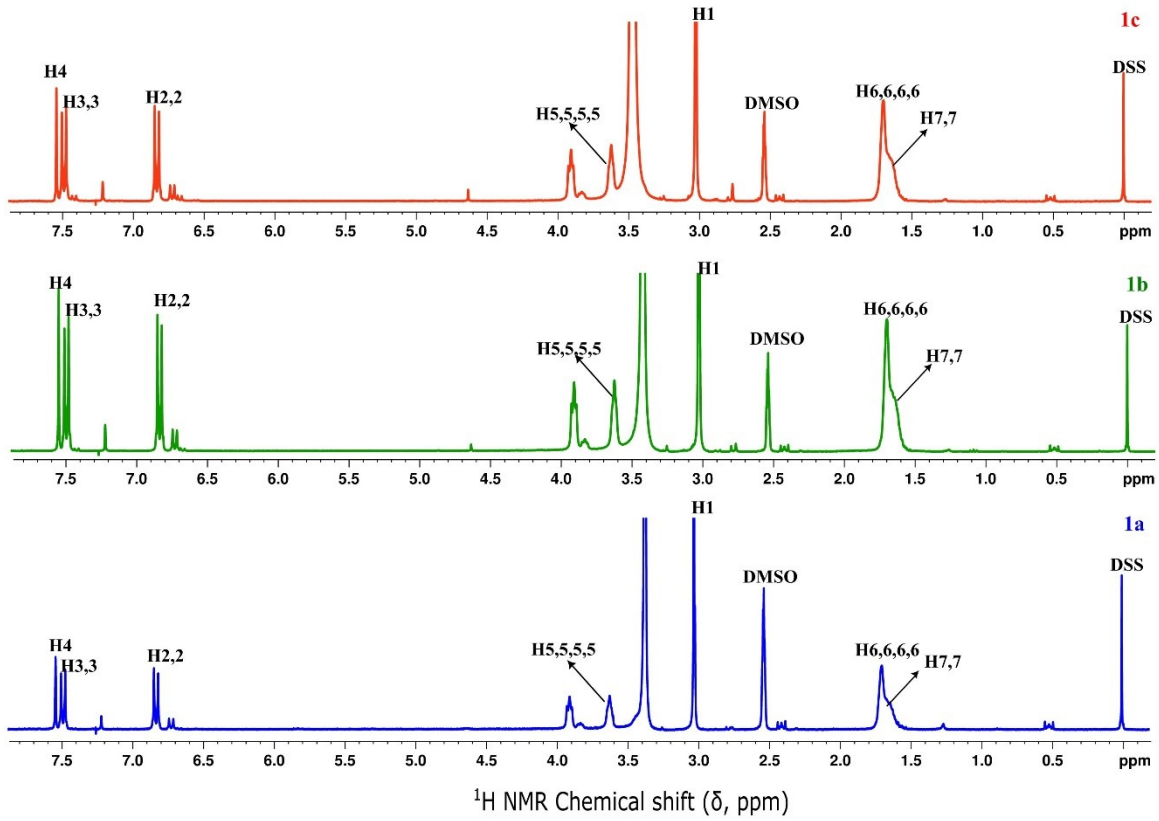

Figure S2. ^{1}H solution NMR spectra of polymorphs 1a (blue), 1b (green), and 1c (red).

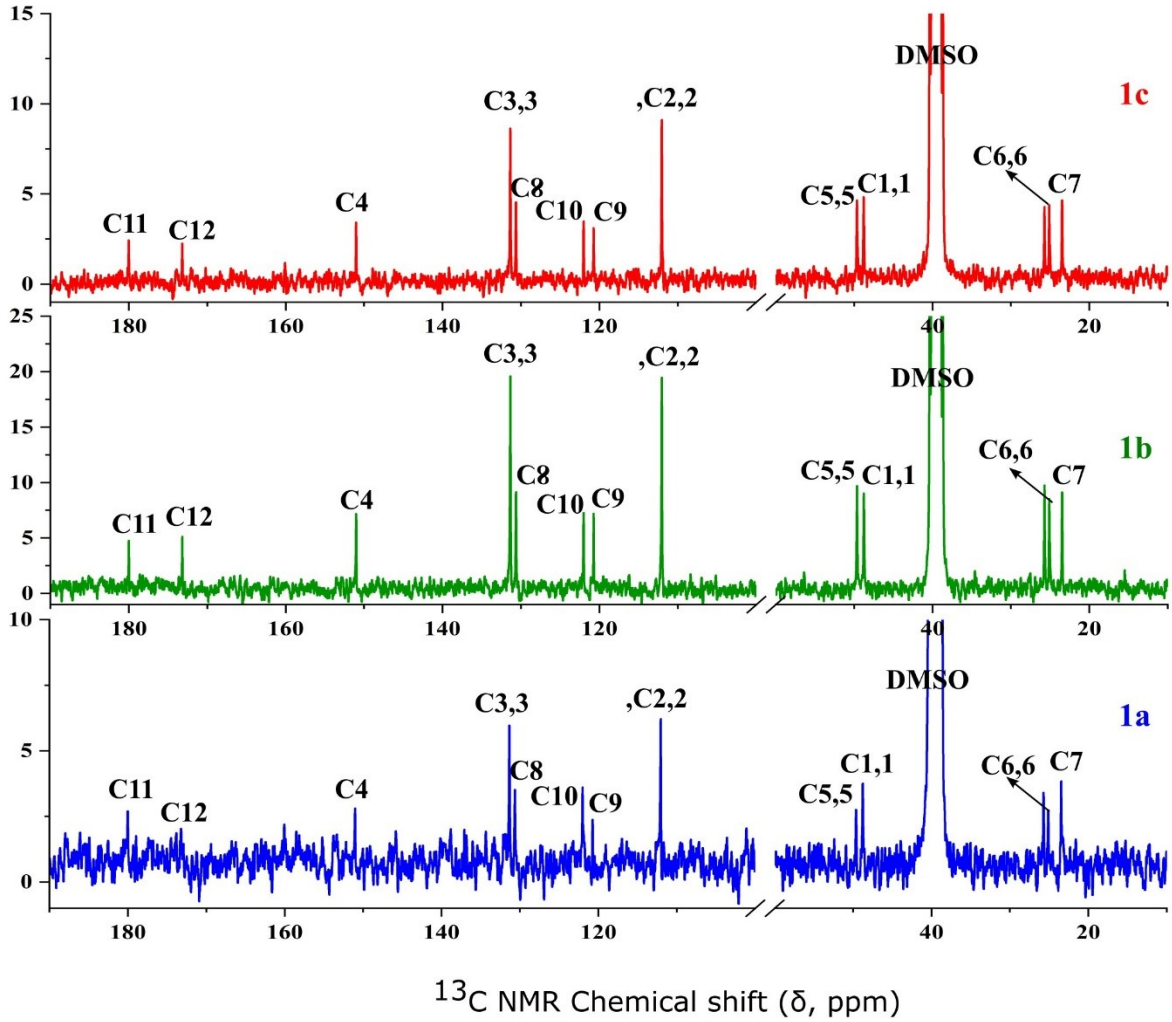

Figure S3. Solution-state 1D ^{13}C NMR spectra of the polymorphs 1a (blue), 1b (green), and 1c (red) in DMSO. Peaks are referenced with respect to DSS (this study).

Table S1. Crystallographic data of polymorphs 1a, 1b, and 1c were determined in this study at ~295 K. The unit cell values from ref. 20 for identical space group crystal structures for 90-100 K data are given in the footnote.[a,b,c]

Compound	**1a**	**1b**	**1c**
Emp. formula	C17 H21 N3 O S	C17 H21 N3 O S	C17 H21 N3 O S
Formula wt.	315.43	315.43	315.43
Cryst. System	Monoclinic	Monoclinic	Orthorhombic
Space group	$P2_1/n$	Cc	$P2_12_12_1$
T (K)	295	296	296
***a* (Å)**	12.7047(9)	13.9971(4)	7.325(5)
***b* (Å)**	8.0199(5)	16.3389(5)	12.993(6)

***c* (Å)**	16.4944(12)	7.7318(2)	16.832(11)
α (deg)	90	90	90
β (deg)	105.648(3)	114.482(1)	90
γ (deg)	90	90	90
Z	4	4	4
V (Å^3)	1618.33(19)	1609.26(8)	1602.0(17)
Rflns collected	3737	3686	3922
Unique reflections	3201	3378	2559
Obsd rflns	3729	3636	3803
Parameters	201	201	201
R_1	0.0819	0.0598	0.0634
***w*R_2**	0.2389	0.1518	0.0634
GOF	1.166	1.134	1.018
Diffractometer	Bruker	Bruker	Bruker

[a] Unit cell values of 1a: a = 12.6934(3), b = 7.7894(2), c = 16.5608(5), β = 107.02.

[b] Unit cell values of 1a: a = 13.9696(12), b = 16.2266(14), c = 7.5176(7), β = 114.336(1).

[c] Unit cell values of 1a: a = 7.1796(3), b = 12.9968(5), c = 16.8109(6).

Table S2. ^{1}H Chemical shift values of the polymorphs 1a, 1b, and 1c derived from Kumar's spectra in Figure S4 (of ref. 20) at 400 MHz.

Polymorph	Chemical Shift (□, ppm)			
	H1,1	H2,2	H3,3	H4
1a	2.9951	6.7959	7.4530	7.5059
1b	2.9951	6.7955	7.4513	7.5056
1c	2.9925	6.7918	7.4477	7.5055

Table S3. ^{1}H Chemical shift values of the polymorphs 1a, 1b, and 1c in DMSO (this paper).

Polymorph	Chemical Shift (□, ppm)

	H1,1	H2,2	H3,3	H4	H5,5,5,5	H6,6,6,6	H7,7,7,7
1a	2.995	6.795	7.442	7.506	3.590	1.666	1.614
1b	2.992	6.791	7.440	7.505	3.586	1.664	1.612
1c	2.992	6.792	7.449	7.504	3.587	1.664	1.614

Table S4. ^{13}C Chemical shift values of the polymorphs 1a, 1b, and 1c.

Polymorph	Chemical Shift (□, ppm)											
	C1,1	C2,2	C3,3	C4	C5,5	C6,6	C7	C8	C9	C10	C11	C12
1a	48.8	112.0	131.4	151.0	49.6	25.1	23.5	130.6	120.7	122.0	180.0	173.2
1b	48.8	112.0	131.3	151.0	49.6	25.1	23.5	130.6	120.7	122.0	180.0	173.2
1c	48.8	112.0	131.4	151.0	49.6	25.1	23.5	130.6	120.7	122.0	180.0	173.2