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Supporting information for article:

High throughput macromolecular polymorph screening via NMR and X-ray powder diffraction synergistic approach: the case of human insulin co-crystallized with resorcinol derivatives

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Table S1 Unit-cell parameters and volume with errors for every sample of HI co-crystallized with 4-chlororesorcinol and 4-bromoresorcinol measured at ID22-ESRF [Series 1: $\lambda = 1.29994(1)$ Å, Series 2: $\lambda = 1.29994(3)$ Å, Series 3: $\lambda = 1.29989(3)$] and MS-X04SA-SLS (Series 4: $\lambda = 1.3831050(6)$ Å).

Sample	pH average	Space Group	a (Å)	b (Å)	c (Å)	β (°)	Unit Cell Volume (Å ³)	Resolution range	V_m (Å ³ /Da)	V_S (%)
chl11	5.09	$P2_1(\gamma)$	87.731(1)	69.9553(8)	47.9564(8)	106.754(2)	281,826(7)	46.56-8.36	2.02	39.17
chl12	5.64	$P2_1(\delta)$	48.4206(9)	59.663(1)	47.7644(6)	94.060(2)	137,642(4)	47.90-8.46	1.97	37.71
chl13	5.93	$P2_1(\delta)$	48.8201(2)	60.0971(2)	47.7672(2)	95.3486(4)	139,536(1)	47.77-8.80	2.00	38.55
chl14	6.19	$C2$	102.947(2)	61.502(1)	63.372(2)	117.221(3)	356,802(18)	45.69-14.48	2.56	51.93
chl15	6.70	$P2_1(\beta)$	62.532(1)	61.8255(9)	47.739(1)	112.061(2)	171,052(6)	44.60-9.77	2.45	49.89
chl16	7.20	$P2_1(\beta)$	61.384(1)	61.9993(9)	47.589(1)	110.380(2)	169,775(5)	44.79-8.44	2.44	49.51
chl17	7.73	$P2_1(\beta)$	60.941(4)	62.290(3)	47.958(4)	110.670(7)	170,330(20)	45.20-12.19	2.44	49.68
chl18	8.04	$P2_1(\beta)$	60.9735(6)	62.3243(4)	47.9881(4)	110.4067(8)	170,916(2)	45.14-8.93	2.45	49.83
chl21	4.55	$P2_1(\gamma)$	87.2016(3)	69.9317(3)	47.7945(2)	106.6076(4)	279,300(2)	46.36-8.91	2.00	38.61
chl22	4.73	$P2_1(\gamma)$	87.2997(9)	69.9560(7)	48.0602(7)	106.585(1)	281,299(6)	46.55-9.69	2.02	39.06
chl23	4.91	$P2_1(\gamma)$	87.413(1)	69.9622(7)	47.8759(7)	106.717(1)	280,415(6)	46.46-9.69	2.01	38.85
chl24	5.08	$P2_1(\gamma)$	87.5622(3)	69.9699(2)	47.9156(2)	106.8409(3)	280,975(2)	46.52-9.31	2.02	38.98
chl25	5.25	$P2_1(\gamma)$	87.614(2)	70.017(1)	47.951(1)	106.883(2)	281,475(9)	46.64-15.53	2.02	39.10
chl26	5.43	$P2_1(\gamma)$	87.7280(3)	70.0154(2)	47.9842(2)	107.0077(4)	281,844(2)	46.45-9.69	2.02	39.15
chl27	<i>Amorphous</i>									
chl28	<i>Amorphous</i>									
chl29	5.98	$C2$	102.03(1)	61.698(7)	62.79(1)	116.03(2)	355,171(86)	46.07-14.46	2.55	51.72
chl210	6.19	$C2$	103.102(5)	61.428(3)	63.206(5)	116.845(7)	357,164(39)	46.16-14.47	2.56	52.00
chl211	6.33	$C2$	103.236(5)	61.464(3)	63.096(5)	116.792(6)	357,387(37)	46.26-14.49	2.56	52.04
chl212	6.52	$C2$	103.358(4)	61.442(3)	63.016(5)	116.695(7)	357,528(37)	46.37-14.42	2.57	52.05
chl213	6.69	$P2_1(\beta)$	61.543(1)	62.1547(8)	47.5918(9)	111.809(2)	169,019(5)	46.83-9.79	2.42	49.27
chl214	6.84	$P2_1(\beta)$	62.0484(5)	62.1725(4)	48.1123(5)	112.4720(8)	171,509(3)	44.58-9.85	2.46	50.03
chl215	6.93	$P2_1(\beta)$	62.0504(6)	62.1516(5)	48.1611(6)	112.460(1)	171,646(3)	44.73-9.82	2.46	50.08
chl216	7.05	$P2_1(\beta)$	62.1202(4)	62.1918(3)	48.1648(3)	112.1391(5)	172,359(2)	44.91-9.82	2.47	50.27
chl217	<i>Amorphous</i>									
chl218	<i>Amorphous</i>									
chl219	7.70	$P2_1(\beta)$	61.585(1)	62.319(1)	48.151(1)	110.577(2)	173,009(7)	45.25-9.17	2.48	50.46

chl31	4.88	$P2_1(\gamma)$	87.5028(6)	69.9512(5)	48.2212(9)	106.618(7)	282,830(3)	46.35-6.38	2.03	39.38
chl32	5.02	$P2_1(\gamma)$	87.6060(5)	69.9646(9)	47.9999(5)	106.667(3)	281,846(8)	46.38-6.20	2.02	39.17
chl33	5.21	$P2_1(\gamma)$	87.8209(2)	69.9752(8)	48.0153(2)	106.719(6)	282,594(5)	46.43-6.57	2.03	39.32
chl34	5.40	$P2_1(\gamma)$	87.7759(9)	70.0289(2)	48.0595(4)	106.809(1)	282,792(8)	46.48-7.42	2.03	39.35
chl35	5.60	$P2_1(\delta)$	48.6583(6)	59.9245(7)	47.7370(5)	94.722(1)	138,720(3)	47.75-8.82	1.99	38.18
chl36	5.73	$P2_1(\delta)$	48.7979(3)	60.0138(4)	47.7707(3)	95.2443(6)	139,313(2)	47.80-8.80	2.00	38.45
chl37	5.90	$P2_1(\delta)$	48.8577(3)	60.1125(4)	47.7789(3)	95.5491(5)	139,667(2)	47.58-9.10	2.00	38.60
chl38	6.06	$C2$	102.798(4)	61.546(2)	63.272(4)	117.077(5)	356,434(31)	45.87-9.09	2.56	51.88
chl39	6.16	$C2$	102.9223(6)	61.5132(3)	63.3498(6)	117.1939(7)	356,740(4)	45.90-14.46	2.56	51.96
chl310	6.26	$P2_1(\beta)$	62.6083(4)	61.7346(4)	47.6832(4)	112.2433(7)	170,585(2)	45.89-10.67	2.45	49.75
chl311	6.43	$P2_1(\beta)$	62.550(2)	61.774(2)	47.735(2)	112.316(3)	170,630(11)	44.35-9.02	2.45	49.75
chl312	6.61	$P2_1(\beta)$	62.413(1)	61.872(1)	47.786(1)	111.978(2)	171,123(7)	44.46-12.12	2.46	49.92
chl313	6.79	$P2_1(\beta)$	62.023(1)	62.0191(7)	47.8473(8)	111.652(2)	171,064(4)	44.96-10.74	2.45	49.89
chl314	7.02	$P2_1(\beta)$	61.496(7)	62.223(6)	47.893(6)	111.08(1)	170,995(32)	44.83-13.80	2.45	49.88
chl315	7.06	$P2_1(\beta)$	61.965(3)	62.082(2)	47.871(2)	111.490(4)	171,352(13)	44.84-12.43	2.46	50.00
chl316	<i>Amorphous</i>									
chl317	7.57	$P2_1(\beta)$	61.306(2)	62.449(1)	48.055(2)	110.883(3)	171,893(8)	45.38-10.95	2.47	50.14
chl318	7.64	$P2_1(\beta)$	61.214(5)	62.439(3)	48.063(5)	110.828(7)	171,698(23)	45.26-9.16	2.46	50.07
chl319	7.71	$P2_1(\beta)$	61.2297(5)	62.4711(4)	48.1002(5)	110.8346(8)	171,957(3)	45.04-9.16	2.47	50.15
chl320	7.94	$P2_1(\beta)$	61.1977(8)	62.5224(7)	48.0907(8)	110.769(1)	172,048(4)	45.10-9.17	2.47	50.19
chl321	8.10	$P2_1(\beta)$	61.070(1)	62.4365(9)	48.094(1)	110.735(2)	171,507(5)	45.16-9.16	2.46	50.02
chl41	5.04	$P2_1(\gamma)$	87.58(2)	69.966(4)	47.908(5)	106.764(3)	281,109(22)	53.78-7.41	2.02	39.01
chl42	5.40	$P2_1(\gamma)$	87.73(3)	70.04(2)	48.001(8)	106.886(4)	282,350(15)	53.78-5.83	2.03	39.28
chl43	5.69	$P2_1(\gamma)$	87.77(2)	67.71(3)	46.72(2)	107.295(2)	265,235(16)		1.90	35.36
		$P2_1(\delta)$	47.69(9)	59.7(1)	45.77(8)	93.93(2)	129,863(8)	47.68-6.21	1.86	33.99
chl44	6.83	$C2$	99.7(2)	61.84(8)	62.3(1)	114.56(4)	349,522(36)		2.51	50.95
		$P2_1(\gamma)$	87.5394(6)	67.9132(4)	46.7896(4)	107.647(2)	265,051(22)	45.77-8.26	1.90	35.31
chl45	6.20	$C2$	99.035(5)	61.194(3)	62.469(3)	114.902(9)	343,303(13)		2.46	50.06
chl46	7.07	$C2$	102.990(2)	61.426(2)	63.342(2)	117.1861(4)	365,422(13)	51.07-7.22	2.62	53.08
chl47	7.51	$C2$	102.23(7)	60.27(3)	63.16(3)	117.046(9)	346,637(23)		2.49	50.54
		$P2_1(\beta)$	61.89(2)	62.10(1)	47.896(8)	111.861(4)	170,648(8)	44.51-5.28	2.45	49.76
chl47	7.51	$P2_1(\beta)$	61.4307(5)	62.4119(3)	48.0548(4)	110.9231(1)	172,040(2)	44.98-4.86	2.47	50.17

Table S2 Delta variations of the normalized unit-cell parameters for the three polymorphs with increasing pH for each ligand (Series 3 measured at ESRF).

	4-chlororesorcinol			4-bromoresorcinol			
	$P2_{1(\gamma)}$	$P2_{1(\delta)}$	$C2$	$P2_{1(\beta)}$	$P2_{1(\gamma)}$	$P2_{1(\delta)}$	$P2_{1(\beta)}$
$\Delta a/a_i$ (%)	0.312	0.410	0.121	-2.457	-0.115	0.016	0.096
$\Delta b/b_i$ (%)	0.111	0.314	-0.053	1.137	0.557	0.050	1.221
$\Delta c/c_i$ (%)	-0.335	0.088	0.123	0.862	0.181	0.011	1.019
$\Delta \beta/\beta_i$ (%)	0.179	0.873	0.010	-1.344	0.116	0.022	1.044
$\Delta V/V_i$ (%)	-0.013	0.683	0.086	0.514	0.559	0.074	1.426