Supplementary Material

Supplmentary Table S1. Complete list of refined lattice parameters of human insulin co-crystallized with phenol, as extracted via the Pawley method. (*) This sample was produced in the same conditions of crystallization with phenol and data were collected during a later experiment at RT using the ID31 beamline (ESRF) and a wavelength of λ=1.29992(3). We note that the Matthews coefficient cannot be reported for the novel $P2_{1(\alpha)}$ monoclinic phase at this stage of analysis as multiple solutions are obtained.

Space Group	pH	a (Å)	b (Å)	c (Å)	β (°)	Volume (Å^3)	Matthews coeff. (Å^3 Da^{-1})	Resolution Range
$P2_{1(\alpha)}$	5.47	114.518(5)	338.05(1)	49.366(2)	101.452(3)	1,873,033(131)		
$P2_{1(\alpha)}$	5.70	114.682(6)	337.63(2)	49.270(4)	101.555(6)	1,869,070(200)	-	112.2 -7.5
$C222_1$	5.93	60.2151(4)	221.884(1)	228.743(2)	90	3,056,190(34)		
$C222_1$	6.14	60.287(1)	221.797(6)	228.812(5)	90	3,059,569(117)		
$C222_1$	6.40	60.3633(7)	221.763(3)	228.766(3)	90	3,062,341(69)		
$C222_1$	6.54	60.3917(6)	221.759(3)	228.643(2)	90	3,062,091(59)	3.66	115 -7.5
(*)C2	6.70	103.000(1)	61.3255(6)	63.582(1)	117.195(2)	357,220(9)		
C2	6.75	103.0115(5)	61.3213(2)	63.5783(4)	117.2244(5)	357,121(3)	2.56	45.9 -5.3
$P2_{1(\beta)}$	7.01	61.4134(5)	61.8494(4)	47.8957(4)	110.7157(7)	170,164(2)		
$P2_{1(\beta)}$	7.26	61.1889(4)	61.8369(3)	47.9198(3)	110.6595(6)	169656(2)		
$P2_{1(\beta)}$	7.46	61.0920(4)	61.8279(4)	47.9302(4)	110.6253(7)	169,437(2)		
$P2_{1(\beta)}$	8.11	61.0095(4)	61.8700(3)	47.9820(3)	110.5368(5)	169,605(2)		
$P2_{1(\beta)}$	8.25	61.0130 (5)	61.8896(3)	47.99454(4)	110.4703(7)	169,787(2)	2.43	45 -4.4

Supplementary Table S2. Complete list of refined lattice parameters of human insulin co-crystallized with resorcinol, as extracted via the Pawley method. We note that the Matthews coefficient cannot be reported for the novel $P2_{1(\alpha)}$ monoclinic phase at this stage of analysis.

Space Group	pH	a (Å)	b (Å)	c (Å)	β (°)	Volume ($Å^3$)	Matthews coeff. ($Å^3$ Da^{-1})	Resolution Range
$P2_{1(\alpha)}$	5.29	114.028(8)	335.43(3)	49.211(6)	101.531(8)	1,844,227(293)		
$P2_{1(\alpha)}$	5.46	114.051(5)	334.99(1)	49.398(3)	101.488(4)	1,849,496(154)	-	112.2 -7.5
$C222_1$	5.93	60.4325(2)	220.8862(5)	228.2074(6)	90	3,046,273(13)		
$C222_1$	6.13	60.4835(4)	220.916(2)	228.303(2)	90	3,050,531(39)		
$C222_1$	6.40	60.5579(7)	220.907(3)	228.320(3)	90	3,054,395(63)		
$C222_1$	6.54	60.5864(3)	220.824(1)	228.207(1)	90	3,053,171(25)		
$C222_1$	6.75	60.6251(2)	220.7959(6)	228.1699(7)	90	3,054,228(15)		
$C222_1$	7.01	60.6819(3)	220.873(1)	228.191(1)	90	3,058,436(23)		
$C222_1$	7.26	60.7273(6)	220.792(2)	228.232(2)	90	3,060,154(51)		
$C222_1$	7.45	60.841(1)	220.881(4)	228.559(3)	90	3,071,519(85)	3.66	115 -7.5
$P2_{1(\beta)}$	7.53	60.9724(4)	61.8540(3)	47.7823(3)	110.1894(5)	169,133(2)		
$P2_{1(\beta)}$	7.74	60.9619(5)	61.8718(4)	47.8235(5)	110.2911(8)	169,188(3)		
$P2_{1(\beta)}$	8.22	61.0008(4)	62.0041(3)	47.8823(3)	110.0465(5)	170,154(2)	2.43	45 -4.4