

Supplementary Information

High Throughput Phase Diagram Mapping via Powder Diffraction:

A case-study of HEWL versus pH.

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Tables

Table 1(a). Measured pH values of the buffer solution before addition of protein (*first column*), pH of protein solutions after crystallisation was complete (*second column*), lattice parameters for tetragonal HEWL (sp. Group: $P4_32_12$) as they were extracted from LeBail analysis from the low temperature experiment (4°C) and the volume per molecule.

pH _(i)	pH _(f)	a (Å)	c (Å)	V/molecule (Å ³)
8.00	6.56	78.8341(29)	38.0783(29)	29581.1(22)
7.80	6.51	78.8004(8)	38.1544(9)	29615.0(7)
7.54	6.41	78.7988(4)	38.1607(5)	29618.6(4)
7.39	6.36	78.8089(5)	38.1747(6)	29637.1(4)
7.23	6.32	78.8151(4)	38.1748(5)	29641.9(3)
7.01	6.23	78.8314(4)	38.1633(5)	29645.2(3)
6.81	6.18	78.8393(4)	38.1629(5)	29650.8(3)
6.60	6.16	78.8509(4)	38.1599(5)	29657.2(3)
6.43	6.02	78.8894(4)	38.1411(4)	29671.6(3)
6.22	5.88	78.9376(4)	38.1096(5)	29683.3(3)
6.03	5.73	78.9925(4)	38.0829(5)	29703.8(3)
5.83	5.55	79.0504(4)	38.0556(5)	29726.1(3)
5.62	5.33	79.1304(5)	38.0131(5)	29752.9(3)
5.45	5.22	79.1780(5)	37.9968(6)	29776.0(4)
5.22	4.99	79.2292(4)	37.9790(5)	29800.5(4)
5.03	4.80	79.2523(4)	37.9662(5)	29807.9(4)
4.82	4.60	79.2754(5)	37.9486(5)	29811.4(4)
4.60	4.42	79.2794(5)	37.9289(5)	29799.0(4)
4.40	4.17	79.2844(5)	37.9137(5)	29790.8(4)
4.21	4.05	79.3047(7)	37.9139(7)	29806.2(5)
3.96	3.83	79.2997(6)	37.8911(6)	29784.5(5)
3.77	3.68	79.3084(8)	37.8867(8)	29787.5(6)
3.55	3.48	79.3071(11)	37.8678(10)	29771.7(8)
3.35	3.33	79.3188(19)	37.8684(20)	29781.0(15)

Table 1(b). Measured pH values of the buffer solution before addition of protein (*first column*), pH of protein solutions after crystallisation was complete (*second column*) and lattice parameters for orthorhombic– (pH 6.5 - 6.26) and tetragonal– (pH 5.11 – 3.33) HEWL as they were extracted from LeBail analysis from the room temperature experiment. Two sets of unit cell dimensions are given from pH_(f) 6.14 – 5.30 due to the coexistence region of the orthorhombic– (upper) and tetragonal (lower) phase.

pH _(i)	pH _(f)	a (Å)	b (Å)	c (Å)	V/molecule (Å ³)	Orthorhombic phase fraction (%)	Tetragonal phase fraction (%)
8.00	6.50	68.4622(7)	59.1644(9)	30.5146(5)	30900.0(4)	100,00	0,00
7.80	6.39	68.4218(6)	59.0813(9)	30.5597(5)	30884.1(4)	100,00	0,00
7.54	6.30	68.5174(6)	59.2328(8)	30.4582(4)	30903.5(4)	100,00	0,00
7.39	6.28	68.5127(6)	59.2326(7)	30.4578(3)	30900.9(3)	100,00	0,00
7.23	6.26	68.5133(6)	59.2357(7)	30.4537(3)	30898.6(4)	100,00	0,00
7.01	6.14	68.4969(10)	59.2135(17)	30.4495(6)	30875.3(8)	92,66	7,34
		78.8658(26)		38.1639(32)	29671.5(30)	88,00	12,00
6.81	6.08	68.5302(7)	59.2621(7)	30.4338(3)	30899.8(4)	51,00	49,00
		78.8923(130)		38.1823(18)	29705.8(94)	34,00	66,00
6.03	5.70	68.4955(11)	59.2979(13)	30.4215(7)	30890.3(7)	18,00	82,00
		78.9669(5)		38.1305(4)	29721.6(3)	0,00	100,00
5.83	5.55	68.4866(10)	59.3306(22)	30.4130(7)	30894.7(9)	0,00	100,00
		79.0428(4)		38.1002(4)	29755.2(3)	0,00	100,00
5.62	5.30	68.4524(24)	59.3885(71)	30.4150(7)	30911.4(3)	0,00	100,00
		79.1063(6)		38.0808(7)	29787.7(5)	0,00	100,00
5.45	5.11	79.1132(4)		38.0746(4)	29788.1(3)	0,00	100,00
5.22	4.95	79.1868(4)		38.0668(4)	29837.5(3)	0,00	100,00
5.03	4.80	79.2122(4)		38.0490(4)	29842.6(3)	0,00	100,00
4.82	4.59	79.2313(5)		38.0474(5)	29855.8(4)	0,00	100,00
4.60	4.41	79.2451(4)		38.0239(4)	29847.7(3)	0,00	100,00
4.40	4.21	79.2575(5)		38.0083(5)	29844.8(4)	0,00	100,00
4.21	4.06	79.2486(5)		38.0035(5)	29834.3(4)	0,00	100,00
3.96	3.86	79.2603(5)		37.9746(5)	29820.5(4)	0,00	100,00
3.55	3.56	79.2802(8)		37.9307(9)	29801.0(7)	0,00	100,00
3.35	3.30	79.3262(5)		37.9144(6)	29822.7(4)	0,00	100,00

Table 2. Effective completeness for single (upper) and 4 combined data sets (lower) as the fraction of "peaks" having $I/\sigma(I)$ greater than some threshold. The individual columns from left to right correspond to minimum resolution (d_{\max}), maximum resolution (d_{\min}) total number of peaks in d-spacing range (total), number of peaks with $I/\sigma(I)$ less than 1 sigma ($<1\sigma$), number of peaks with $I/\sigma(I)$ between 1 and 3 sigma ($1-3\sigma$) and >3 sigma ($>3\sigma$), number of unique d-spacings in the corresponding range (accounting for (4,3,0) versus (5,0,0) etc) (Poss) and percentages representing the completeness ($\%>1\sigma$, $\%>3\sigma$, $\%Poss$).

Single Pattern									
d_{\max}	d_{\min}	total	$<1\sigma$	$1-3\sigma$	$>3\sigma$	Poss	$\%>1\sigma$	$\%>3\sigma$	$\%Poss$
55.696	12.876	46	2	1	43	45	95.65	93.48	97.83
12.876	9.229	70	13	8	49	63	81.43	70.00	90.00
9.229	7.57	77	25	6	46	73	67.53	59.74	89.02
7.57	6.571	95	33	7	55	80	65.26	57.89	87.91
6.571	5.885	126	59	16	51	93	53.17	40.48	86.92
5.885	5.378	124	63	10	51	93	49.19	41.13	83.78
5.378	4.982	128	71	17	40	103	44.53	31.25	85.83
4.982	4.663	121	63	13	45	106	47.93	37.19	82.81
4.663	4.398	129	77	13	39	113	40.31	30.23	84.96
4.398	4.173	159	99	12	48	119	37.74	30.19	79.87
4.173	3.98	144	100	13	31	115	30.56	21.53	82.14
3.98	3.811	150	100	11	39	128	33.33	26.00	81.01
3.811	3.663	158	113	16	29	132	28.48	18.35	80.00
3.663	3.53	147	102	6	39	132	30.61	26.53	78.57
3.53	3.411	163	128	9	26	135	21.47	15.95	79.88

Combined fit to four patterns

d_{\max}	d_{\min}	total	$<1\sigma$	$1-3\sigma$	$>3\sigma$	Poss	$\%>1\sigma$	$\%>3\sigma$	$\%Poss$
55.696	12.876	46	3	1	42	45	93.48	91.30	97.83
12.876	9.229	68	11	1	56	63	83.82	82.35	90.00
9.229	7.57	81	19	7	55	73	76.54	67.90	89.02
7.57	6.571	96	20	6	70	80	79.17	72.92	87.91
6.571	5.885	110	30	9	71	93	72.73	64.55	86.92
5.885	5.378	115	35	13	67	93	69.57	58.26	83.78
5.378	4.982	126	44	13	69	103	65.08	54.76	85.83
4.982	4.663	144	60	10	74	106	58.33	51.39	82.81
4.663	4.398	134	53	14	67	113	60.45	50.00	84.96
4.398	4.173	150	59	13	78	119	60.67	52.00	79.87
4.173	3.98	140	70	16	54	115	50.00	38.57	82.14
3.98	3.811	135	62	16	57	128	54.07	42.22	81.01
3.811	3.663	180	82	28	70	132	54.44	38.89	80.00
3.663	3.53	158	87	16	55	132	44.94	34.81	78.57
3.53	3.411	154	88	20	46	135	42.86	29.87	79.88