

Table 1 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the first cycle of humidity variations (gradual dehydration and rehydration from rH of 95% to 60 %). Data collection was conducted utilizing a laboratory X- ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	rH level	Space group	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	Volume ($\times 10^4$ Å ³)	χ^2	R_{wp} (%)
1 st	95%	$P 2_12_12_1$	18.608 (2)	30.254 (3)	39.794 (6)	90	90	90	2.240(3)	2.16216	4.8158
	90%	$P 2_12_12_1$	18.611 (2)	30.254 (3)	39.786 (5)	90	90	90	2.224(0)	2.36047	5.0821
	85%	$P 2_12_12_1$	18.616 (3)	30.257 (2)	39.772 (4)	90	90	90	2.240(3)	2.96565	6.0041
	80%	$P 2_12_12_1$	18.546 (1)	30.178 (2)	39.733 (3)	90	90	90	2.223(9)	3.39206	6.6083
	75%	$P 2_12_12_1$	18.515 (1)	30.115 (3)	39.703(5)	90	90	90	2.213(8)	2.88178	5.6379
	70%	$P 2_12_12_1$	18.453 (3)	29.952 (4)	39.978 (7)	90	90	90	2.220(9)	2.84206	5.7221
	68%	$P 2_12_12_1$	18.60(2)	29.70 (4)	40.21 (5)	90	90	90	2.222(5)	3.03643	6.0739
	66%	$P 2_12_12_1$	18.60 (3)	29.69 (6)	40.20 (6)	90	90	90	2.222(0)	3.28065	6.558
	64%	$P 2_12_12_1$	18.59 (3)	29.65 (4)	39.96 (5)	90	90	90	2.204(1)	2.92732	5.8551
	62%	$P 2_12_12_1$	18.59 (3)	29.54 (5)	39.85 (6)	90	90	90	2.189(7)	3.0151	6.211
	60%	$P 2_12_12_1$	18.6 (1)	29.0 (2)	39.3 (3)	90	90	90	2.128(8)	1.32418	8.4831
	62%	$P 2_12_12_1$	18.51(3)	29.54 (4)	39.64 (5)	90	90	90	2.168(2)	3.30002	6.6207
	64%	$P 2_12_12_1$	18.50 (2)	29.53 (3)	39.65 (4)	90	90	90	2.167(9)	3.12757	6.7134
	66%	$P 2_12_12_1$	18.58 (4)	29.65 (6)	40.13 (6)	90	90	90	2.212(1)	3.34487	6.5371
	68%	$P 2_12_12_1$	18.60 (4)	29.66 (7)	40.16 (8)	90	90	90	2.216(9)	3.16505	6.811
	70%	$P 2_12_12_1$	18.63 (4)	29.68(6)	40.17(8)	90	90	90	2.222(9)	2.92445	6.2678
	75%	$P 2_12_12_1$	18.64(3)	29.63(6)	40.16(9)	90	90	90	2.219(0)	2.28835	6.0315
	80%	$P 2_12_12_1$	18.530(1)	30.150(2)	39.684(4)	90	90	90	2.217(2)	3.12224	6.1605
	85%	$P 2_12_12_1$	18.531(1)	30.151(2)	39.680(4)	90	90	90	2.217(0)	3.61022	7.1434
	90%	$P 2_12_12_1$	18.566(1)	30.192(2)	39.696(4)	90	90	90	2.225(2)	3.42726	6.8164
95%	$P 2_12_12_1$	18.605(1)	30.249(2)	39.788(4)	90	90	90	2.239(2)	3.1479	7.1291	

Table 2 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during first cycle (gradual dehydration and rehydration from rH of 95% to 60 %) of humidity alteration experiments . Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Decreasing rH (%)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
95	0	0	0	0
90	-0.02%	0.00%	0.02%	0.71%
85	-0.04%	-0.01%	0.06%	0.00%
80	0.33%	0.25%	0.15%	0.76%
75	0.50%	0.46%	0.23%	1.21%
70	0.83%	1.00%	-0.46%	0.89%
68	0.04%	1.83%	-1.05%	0.80%
66	0.04%	1.86%	-1.02%	0.80%
64	0.10%	2.00%	-0.42%	1.61%
62	0.10%	2.36%	-0.14%	2.28%
60	0.04%	4.14%	1.24%	5.00%
Increasing rH (%)				
62	0.53%	2.36%	0.39%	3.21%
64	0.58%	2.39%	0.36%	3.26%
66	0.15%	2.00%	-0.84%	1.25%
68	0.04%	1.96%	-0.92%	1.07%
70	-0.12%	1.90%	-0.94%	0.80%
75	-0.17%	2.06%	-0.92%	0.94%
80	0.42%	0.34%	0.28%	1.03%
85	0.41%	0.34%	0.29%	1.03%
90	0.23%	0.20%	0.25%	0.67%
95	0.02%	0.02%	0.02%	0.04%

Table 3 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the second cycle of humidity variations (gradual dehydration and rehydration from rH of 95% to 40 %). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	rH level	Space group	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	Volume ($\times 10^4 \text{ \AA}^3$)	χ^2	R_{wp} (%)	
2 nd	95%	$P 2_1 2_1 2_1$	18.600(1)	30.248(2)	39.796(3)	90	90	90	2.239(1)	3.47757	6.8994	
	90%	$P 2_1 2_1 2_1$	18.586(1)	30.238(2)	39.794(3)	90	90	90	2.236(5)	3.35836	6.6651	
	85%	$P 2_1 2_1 2_1$	18.560(1)	30.202(2)	39.794(4)	90	90	90	2.230(8)	3.22097	6.4099	
	80%	$P 2_1 2_1 2_1$	18.538(1)	30.163(2)	39.785(4)	90	90	90	2.224(7)	3.25397	6.3941	
	75%	$P 2_1 2_1 2_1$	18.538(3)	30.038(5)	39.67(1)	90	90	90	2.209(3)	2.73115	5.4227	
	70%	$P 2_1 2_1 2_1$	18.66(3)	29.71(4)	39.77(7)	90	90	90	2.206(4)	1.86652	4.1898	
	65%	$P 2_1 2_1 2_1$	18.97(3)	28.72(4)	39.94(4)	90	90	90	2.177(2)	2.06036	3.963	
	60%	$P 2_1 2_1 2_1$	19.00(2)	28.68(3)	40.04(4)	90	90	90	2.183(3)	2.19177	4.2082	
	58%	$P 2_1 2_1 2_1$	19.01(2)	28.67(3)	40.05(3)	90	90	90	2.184(0)	2.19686	4.2259	
	56%	$P 2_1 2_1 2_1$	19.01(2)	28.66(2)	40.07(3)	90	90	90	2.183(8)	2.23612	4.2915	
	54%	$P 2_1 2_1 2_1$	19.00(2)	28.66(2)	40.08(3)	90	90	90	2.183(7)	2.28719	4.4447	
	52%	$P 2_1 2_1 2_1$	19.00(2)	28.66(2)	40.07(3)	90	90	90	2.182(8)	2.31801	4.4795	
	50%	$P 2_1 2_1 2_1$	18.98(2)	28.65(2)	40.10(3)	90	90	90	2.182(7)	2.32553	4.5029	
	48%	$P 2_1 2_1 2_1$	18.98(2)	28.65(2)	40.09(3)	90	90	90	2.181(5)	2.3587	4.5652	
	46%	$P 2_1 2_1 2_1$	18.97(2)	28.64(2)	40.10(3)	90	90	90	2.179(3)	2.35062	4.5686	
	44%	$P 2_1 2_1 2_1$	18.95(2)	28.61(2)	40.07(3)	90	90	90	2.173(8)	2.36094	4.5734	
	42%	$P 2_1 2_1 2_1$	18.93(2)	28.57(2)	40.07(3)	90	90	90	2.168(2)	2.34585	4.5646	
	40%	$P 2_1 2_1 2_1$	18.89(2)	28.55(3)	40.04(4)	90	90	90	2.160(3)	2.35451	4.5792	
		42%	$P 2_1 2_1 2_1$	18.87(2)	28.54(2)	40.07(3)	90	90	90	2.159(3)	2.38986	4.6497
		44%	$P 2_1 2_1 2_1$	18.85(2)	28.54(2)	40.07(3)	90	90	90	2.156(9)	2.3174	4.5389
		46%	$P 2_1 2_1 2_1$	18.84(2)	28.53(2)	40.10(3)	90	90	90	2.156(7)	2.31803	4.5449
		48%	$P 2_1 2_1 2_1$	18.83(2)	28.52(2)	40.11(3)	90	90	90	2.156(0)	2.27241	4.4692
		50%	$P 2_1 2_1 2_1$	18.86(2)	28.52(2)	40.08(3)	90	90	90	2.156(2)	2.35265	4.595
		52%	$P 2_1 2_1 2_1$	18.82(2)	28.52(2)	40.10(3)	90	90	90	2.153(5)	2.24071	4.4698
		54%	$P 2_1 2_1 2_1$	18.81(2)	28.52(2)	40.13(3)	90	90	90	2.154(0)	2.253	4.4599
		56%	$P 2_1 2_1 2_1$	18.80(2)	28.53(3)	40.16(4)	90	90	90	2.154(8)	2.23115	4.4322
		58%	$P 2_1 2_1 2_1$	18.81(2)	28.55(2)	40.29(4)	90	90	90	2.164(9)	2.29175	4.4948
		60%	$P 2_1 2_1 2_1$	18.79(2)	28.55(3)	40.39(4)	90	90	90	2.168(3)	2.16776	4.2013
		65%	$P 2_1 2_1 2_1$	18.80(2)	28.59(3)	40.57(5)	90	90	90	2.181(9)	2.18389	4.2282
		70%	$P 2_1 2_1 2_1$	18.82(2)	28.67(3)	40.86(5)	90	90	90	2.205(8)	2.11422	4.0871
	75%	$P 2_1 2_1 2_1$	18.80(2)	28.65(3)	41.20(4)	90	90	90	2.220(7)	2.10207	3.8417	
	80%	$P 2_1 2_1 2_1$	18.502(2)	30.094(3)	39.654(7)	90	90	90	2.208(1)	2.98497	5.6471	
	85%	$P 2_1 2_1 2_1$	18.543(1)	30.101(2)	39.582(2)	90	90	90	2.209(4)	3.12913	5.9189	
	90%	$P 2_1 2_1 2_1$	18.554(1)	30.113(2)	39.592(2)	90	90	90	2.212(1)	3.63613	6.5986	
	95%	$P 2_1 2_1 2_1$	18.590(1)	30.154(2)	39.643(4)	90	90	90	2.222(3)	3.48532	6.6332	

Table 4 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during second cycle (gradual dehydration and rehydration from rH of 95% to 40 %) of humidity alteration experiments . Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Decreasing rH (%)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
95	0	0	0	0
90	0.08%	0.03%	0.01%	0.13%
85	0.22%	0.15%	0.01%	0.40%
80	0.33%	0.28%	0.03%	0.67%
75	0.33%	0.69%	0.32%	1.34%
70	-0.32%	1.78%	0.07%	1.47%
65	-1.99%	5.05%	-0.36%	2.77%
60	-2.15%	5.18%	-0.61%	2.50%
58	-2.20%	5.22%	-0.64%	2.46%
56	-2.20%	5.25%	-0.69%	2.50%
54	-2.15%	5.25%	-0.71%	2.50%
52	-2.15%	5.25%	-0.69%	2.55%
50	-2.04%	5.28%	-0.76%	2.55%
48	-2.04%	5.28%	-0.74%	2.59%
46	-1.99%	5.32%	-0.76%	2.68%
44	-1.88%	5.42%	-0.69%	2.95%
42	-1.77%	5.55%	-0.69%	3.17%
40	-1.56%	5.61%	-0.61%	3.53%
Increasing rH (%)				
42	-1.45%	5.65%	-0.69%	3.57%
44	-1.34%	5.65%	-0.69%	3.71%
46	-1.29%	5.68%	-0.76%	3.71%
48	-1.24%	5.71%	-0.79%	3.71%
50	-1.40%	5.71%	-0.71%	3.71%
52	-1.18%	5.71%	-0.76%	3.84%
54	-1.13%	5.71%	-0.84%	3.80%
56	-1.08%	5.68%	-0.91%	3.80%
58	-1.13%	5.61%	-1.24%	3.35%
60	-1.02%	5.61%	-1.49%	3.17%
65	-1.08%	5.48%	-1.94%	2.59%
70	-1.18%	5.22%	-2.67%	1.52%
75	-1.08%	5.28%	-3.53%	0.85%
80	0.53%	0.51%	0.36%	1.38%
85	0.31%	0.49%	0.54%	1.34%
90	0.25%	0.45%	0.51%	1.21%
95	0.05%	0.31%	0.38%	0.76%

Table 5 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the third cycle of humidity variations (gradual dehydration and rehydration from rH of 95% to 30 %). Data collection was conducted utilizing a laboratory X- ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	rH level	Space group	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	Volume ($\times 10^4 \text{ \AA}^3$)	χ^2	R_{wp} (%)
3 rd	95%	$P 2_12_12_1$	18.565(3)	30.147(4)	39.671(8)	90	90	90	2.220(3)	1.6348	5.3146
	90%	$P 2_12_12_1$	18.566(2)	30.148(3)	39.667(5)	90	90	90	2.220(3)	1.767	5.5943
	85%	$P 2_12_12_1$	18.558(2)	30.149(2)	39.669(4)	90	90	90	2.219(6)	2.02902	6.0833
	80%	$P 2_12_12_1$	18.492(2)	30.113(3)	39.772(7)	90	90	90	2.214(7)	2.59601	7.398
	75%	$P 2_12_12_1$	18.422(1)	29.954(2)	39.509(4)	90	90	90	2.180(2)	2.30815	6.6603
	70%	$P 2_12_12_1$	18.65(1)	27.87(2)	39.79(3)	90	90	90	2.068(8)	2.92153	8.658
	65%	$P 2_12_12_1$	18.68(3)	28.37(3)	38.80(8)	90	90	90	2.057(9)	1.61858	4.8382
	60%	$P 2_12_12_1$	18.71(2)	28.03(3)	39.39(4)	90	90	90	2.066(3)	2.10157	6.1237
	55%	$P 2_12_12_1$	18.75(1)	28.02(2)	39.40(3)	90	90	90	2.065(2)	2.15575	6.2425
	50%	$P 2_12_12_1$	18.68(1)	28.01(2)	39.41(3)	90	90	90	2.063(4)	2.1062	6.0991
	45%	$P 2_12_12_1$	18.65(1)	28.01(1)	39.44(2)	90	90	90	2.061(4)	2.05593	5.9688
	40%	$P 2_12_12_1$	18.64(1)	28.01(2)	39.46(3)	90	90	90	2.060(9)	2.01858	5.8527
	38%	$P 2_12_12_1$	18.61(1)	28.00(1)	39.50(2)	90	90	90	2.060(1)	2.65468	5.408
	36%	$P 2_12_12_1$	18.60(1)	28.00(2)	39.51(2)	90	90	90	2.059(0)	2.61	5.3119
	34%	$P 2_12_12_1$	18.58(1)	28.01(2)	39.54(3)	90	90	90	2.058(2)	2.52081	5.1194
	32%	$P 2_12_12_1$	18.57(1)	28.02(2)	39.53(3)	90	90	90	2.058(5)	2.53057	5.1263
	30%	$P 2_12_12_1$	18.66(1)	28.21(2)	39.78(3)	90	90	90	2.095(4)	2.48697	5.0404
	32%	$P 2_12_12_1$	18.66(1)	28.20(2)	39.72(3)	90	90	90	2.091(2)	2.57818	5.1927
	34%	$P 2_12_12_1$	18.50(2)	28.22(3)	40.25(4)	90	90	90	2.102(3)	2.19388	4.3437
	36%	$P 2_12_12_1$	18.61(2)	28.12(2)	40.22(4)	90	90	90	2.105(4)	2.5865	5.13
	38%	$P 2_12_12_1$	18.63(1)	28.17(2)	40.18(4)	90	90	90	2.110(5)	2.5877	5.1263
	40%	$P 2_12_12_1$	18.68(2)	28.22(3)	40.55(5)	90	90	90	2.138(6)	1.95362	5.4814
	45%	$P 2_12_12_1$	18.68(2)	28.21(3)	40.58(5)	90	90	90	2.139(9)	1.90567	5.36
	50%	$P 2_12_12_1$	18.68(2)	28.21(3)	40.57(6)	90	90	90	2.139(6)	1.95078	5.5192
	55%	$P 2_12_12_1$	18.75(1)	28.23(2)	40.61(4)	90	90	90	2.151(2)	1.95188	5.5587
	60%	$P 2_12_12_1$	18.78(2)	28.23(3)	40.58(5)	90	90	90	2.153(0)	2.07886	5.7439
	65%	$P 2_12_12_1$	18.83(2)	28.27(2)	40.54(4)	90	90	90	2.158(3)	1.92407	5.6027
	70%	$P 2_12_12_1$	18.86(2)	28.34(3)	40.49(4)	90	90	90	2.165(6)	1.75268	5.0722
	75%	$P 2_12_12_1$	18.84(5)	28.47(7)	40.3(1)	90	90	90	2.166(3)	1.55231	4.4297
	80%	$P 2_12_12_1$	18.433(2)	29.929(3)	39.447(7)	90	90	90	2.176(3)	2.69095	7.7158
85%	$P 2_12_12_1$	18.440(2)	29.938(3)	39.437(6)	90	90	90	2.177(2)	2.45895	7.1064	
90%	$P 2_12_12_1$	18.452(2)	29.946(3)	39.429(6)	90	90	90	2.178(9)	2.55539	7.3557	
95%	$P 2_12_12_1$	18.469(2)	29.961(3)	39.439(7)	90	90	90	2.182(5)	2.58562	7.417	

Table 6 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during third cycle (gradual dehydration and rehydration from rH of 95% to 30 %) of humidity alteration experiments . Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Decreasing rH (%)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
95	0	0	0	0
90	-0.01%	0.00%	0.01%	0.00%
85	0.04%	-0.01%	0.01%	0.05%
80	0.39%	0.11%	-0.25%	0.27%
75	0.77%	0.64%	0.41%	1.80%
70	-0.46%	7.55%	-0.30%	6.85%
65	-0.62%	5.89%	2.20%	7.34%
60	-0.78%	7.02%	0.71%	6.94%
55	-1.00%	7.06%	0.68%	6.98%
50	-0.62%	7.09%	0.66%	7.07%
45	-0.46%	7.09%	0.58%	7.16%
40	-0.40%	7.09%	0.53%	7.21%
38	-0.24%	7.12%	0.43%	7.21%
36	-0.19%	7.12%	0.41%	7.25%
34	-0.08%	7.09%	0.33%	7.30%
32	-0.03%	7.06%	0.36%	7.30%
30	-0.51%	6.43%	-0.27%	5.63%
Increasing rH (%)				
32	-0.51%	6.46%	-0.12%	5.81%
34	0.35%	6.39%	-1.46%	5.32%
36	-0.24%	6.72%	-1.38%	5.18%
38	-0.35%	6.56%	-1.28%	4.95%
40	-0.62%	6.39%	-2.22%	3.69%
45	-0.62%	6.43%	-2.29%	3.65%
50	-0.62%	6.43%	-2.27%	3.65%
55	-1.00%	6.36%	-2.37%	3.11%
60	-1.16%	6.36%	-2.29%	3.02%
65	-1.43%	6.23%	-2.19%	2.79%
70	-1.59%	5.99%	-2.06%	2.48%
75	-1.48%	5.56%	-1.59%	2.43%
80	0.71%	0.72%	0.56%	1.98%
85	0.67%	0.69%	0.59%	1.94%
90	0.61%	0.67%	0.61%	1.89%
95	0.52%	0.62%	0.58%	1.71%

Table 7 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the first cycle of temperature variations at 95% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	95% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	β ($^\circ$)		
1 st	H e a t i n g	294.15	$P 2_1 2_1 2_1$	18.512(2)	30.050(3)	39.534(6)	90	2.1993(5)	1.44654
		298.15	$P 2_1 2_1 2_1$	18.513(2)	30.068(3)	39.574(6)	90	2.2030(5)	1.48737
		302.15	$P 2_1 2_1 2_1$	18.516(2)	30.070(3)	39.567(7)	90	2.2030(5)	1.48046
		306.15	$P 2_1 2_1 2_1$	18.525(3)	30.057(3)	39.605(6)	90	2.2053(5)	1.43477
		310.15	$P 2_1 2_1 2_1$	18.533(2)	30.098(3)	39.665(6)	90	2.2126(5)	1.51579
		314.15	$P 2_1 2_1 2_1$	18.533(2)	30.087(3)	39.660(6)	90	2.2115(5)	1.48301
		318.15	$P 2_1 2_1 2_1$	18.539(2)	30.074(3)	39.685(6)	90	2.2127(5)	1.52983
	C o o l i n g	318.15	$P 2_1 2_1 2_1$	18.539(2)	30.074(3)	39.685(6)	90	2.2127(5)	1.52983
		314.15	$P 2_1 2_1 2_1$	18.533(2)	30.063(3)	39.657(7)	90	2.2096(5)	1.69219
		310.15	$P 2_1 2_1 2_1$	18.527(2)	30.067(3)	39.644(6)	90	2.2085(5)	1.64987
		306.15	$P 2_1 2_1 2_1$	18.524(2)	30.051(3)	39.597(5)	90	2.2042(5)	1.42922
		302.15	$P 2_1 2_1 2_1$	18.526(2)	30.060(3)	39.604(6)	90	2.2056(5)	1.49202
		298.15	$P 2_1 2_1 2_1$	18.518(2)	30.061(3)	39.568(7)	90	2.2028(5)	1.49769
		294.15	$P 2_1 2_1 2_1$	18.515(2)	30.081(3)	39.577(7)	90	2.2043(5)	1.45927

Table 8 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during first cycle at 95% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	-0.01%	-0.06%	-0.10%	-0.17%
302.15	-0.02%	-0.07%	-0.08%	-0.17%
306.15	-0.07%	-0.02%	-0.18%	-0.27%
310.15	-0.11%	-0.16%	-0.33%	-0.60%
314.15	-0.11%	-0.12%	-0.32%	-0.55%
318.15	-0.15%	-0.08%	-0.38%	-0.61%
Decreasing temperature (K)				
314.15	-0.11%	-0.04%	-0.31%	-0.47%
310.15	-0.08%	-0.06%	-0.28%	-0.42%
306.15	-0.06%	0.00%	-0.16%	-0.22%
302.15	-0.08%	-0.03%	-0.18%	-0.29%
298.15	-0.03%	-0.04%	-0.09%	-0.16%
294.15	-0.02%	-0.10%	-0.11%	-0.23%

Table 9 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the second cycle of temperature variations at 85% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	85% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	θ ($^\circ$)		
2 nd	H e a t i n g	294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.446(2)	29.952(3)	39.448(7)	90	2.1796(5)	1.4798
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.451(2)	29.952(4)	39.466(7)	90	2.1811(5)	1.38136
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.461(2)	29.978(3)	39.467(7)	90	2.1843(5)	1.48822
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.461(2)	29.948(3)	39.492(6)	90	2.1835(4)	1.5732
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.468(1)	29.948(3)	39.526(5)	90	2.1862(4)	1.57598
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.467(2)	29.891(3)	39.532(7)	90	2.1822(5)	1.37569
		318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.476(2)	29.837(4)	39.536(7)	90	2.1795(5)	1.31912
	C o o l i n g	318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.476(2)	29.837(4)	39.536(7)	90	2.1795(5)	1.31912
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.464(2)	29.891(3)	39.506(7)	90	2.1804(5)	1.38748
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.460(2)	29.938(3)	39.493(7)	90	2.1827(5)	1.39353
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.457(2)	29.952(3)	39.486(6)	90	2.1830(5)	1.56033
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.454(2)	29.957(3)	39.486(7)	90	2.1830(5)	1.45087
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.454(2)	29.986(3)	39.483(7)	90	2.1849(5)	1.56413
		294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.455(2)	29.991(3)	39.466(7)	90	2.1844(5)	1.42842

Table 10 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during second cycle at 85% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	-0.03%	0.00%	0.01%	-0.07%
302.15	-0.08%	-0.09%	-0.05%	-0.22%
306.15	-0.08%	0.01%	-0.11%	-0.18%
310.15	-0.12%	0.01%	-0.20%	-0.30%
314.15	-0.11%	0.20%	-0.21%	-0.12%
318.15	-0.16%	0.38%	-0.22%	0.00%
Decreasing temperature (K)				
314.15	-0.10%	0.20%	-0.15%	-0.04%
310.15	-0.08%	0.05%	-0.11%	-0.14%
306.15	-0.06%	0.00%	-0.10%	-0.16%
302.15	-0.04%	-0.02%	-0.10%	-0.16%
298.15	-0.04%	-0.11%	-0.09%	-0.24%
294.15	-0.05%	-0.13%	-0.05%	-0.22%

Table 11 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the third cycle of temperature variations at 75% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	75% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	β ($^\circ$)		
3 rd	H e a t i n g	294.15	$P 2_12_12_1$	18.386(3)	29.644(7)	39.36(1)	90	2.1457(9)	1.87608
		298.15	$P 2_12_12_1$	18.389(2)	29.494(5)	39.419(8)	90	2.1280(6)	1.50289
		302.15	$P 2_12_12_1$	18.398(2)	29.381(5)	39.280(9)	90	2.1233(7)	1.23852
		306.15	$P 2_12_12_1$	18.385(2)	29.359(5)	39.240(9)	90	2.1181(7)	1.33646
		310.15	$P 2_12_12_1$	18.445(3)	29.301(6)	39.00(1)	90	2.1082(8)	1.17672
		314.15	$P 2_12_12_1$	18.420(4)	29.248(9)	38.78(2)	90	2.089(1)	1.47927
		318.15	$P 2_12_12_1$	18.566(6)	29.25(1)	38.54(3)	90	2.093(2)	1.30883
	C o o l i n g	318.15	$P 2_12_12_1$	18.566(6)	29.25(1)	38.54(3)	90	2.093(2)	1.30883
		314.15	$P 2_12_12_1$	18.41(1)	28.92(2)	37.84(4)	90	2.015(3)	1.2103
		310.15	$P 2_12_12_1$	18.42(1)	28.92(6)	37.83(5)	90	2.016(3)	1.16639
		306.15	$P 2_12_12_1$	18.39(1)	28.92(3)	37.89(5)	90	2.016(4)	1.41163
		302.15	$P 2_12_12_1$	18.422(7)	28.95(2)	37.89(3)	90	2.021(2)	1.24583
		298.15	$P 2_12_12_1$	18.48(2)	29.10(3)	38.26(6)	90	2.058(4)	1.26835
		294.15	$P 2_12_12_1$	18.51(1)	29.18(3)	38.45(5)	90	2.077(4)	1.28104

Table 12 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during third cycle at 75% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	-0.02%	0.51%	-0.15%	0.82%
302.15	-0.07%	0.89%	0.20%	1.04%
306.15	0.01%	0.96%	0.30%	1.29%
310.15	-0.32%	1.16%	0.91%	1.75%
314.15	-0.18%	1.34%	1.47%	2.64%
318.15	-0.98%	1.33%	2.08%	2.46%
Decreasing temperature (K)				
314.15	-0.13%	2.44%	3.86%	6.09%
310.15	-0.18%	2.44%	3.89%	6.04%
306.15	-0.02%	2.44%	3.73%	6.04%
302.15	-0.20%	2.34%	3.73%	5.81%
298.15	-0.51%	1.84%	2.79%	4.09%
294.15	-0.67%	1.57%	2.31%	3.20%

Table 13 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the fourth cycle of temperature variations at 65% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	65% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	β ($^\circ$)		
4 th	H e a t i n g	294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.603(2)	30.194(4)	39.751(8)	90	2.2328(6)	1.30225
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.63(3)	28.41(3)	38.5(2)	90	2.040(9)	1.37074
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.585(9)	28.25(1)	37.92(2)	90	1.991(2)	1.77596
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.727(7)	28.40(1)	38.12(2)	90	2.028(2)	1.80224
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.790(9)	28.46(2)	38.20(3)	90	2.043(2)	1.84656
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.788(8)	28.50(1)	38.26(2)	90	2.049(2)	1.81797
		318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.56(2)	27.99(5)	38.73(6)	90	2.012(5)	2.2769
	C o o l i n g	318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.56(2)	27.99(5)	38.73(6)	90	2.012(5)	2.2769
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.791(9)	28.52(1)	38.29(3)	90	2.052(2)	1.86604
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.81(1)	28.31(2)	37.60(5)	90	2.003(3)	1.98207
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.738(9)	28.33(2)	37.89(3)	90	2.011(2)	1.80924
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.876(9)	28.28(1)	38.02(2)	90	2.030(2)	1.8166
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.903(9)	28.31(1)	38.04(2)	90	2.036(2)	1.75858
		294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.90(1)	28.30(1)	38.16(2)	90	2.042(2)	1.75277

Table 14 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during fourth cycle at 65% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	-0.15%	5.91%	3.15%	8.63%
302.15	0.10%	6.44%	4.61%	10.83%
306.15	-0.67%	5.94%	4.10%	9.17%
310.15	-1.01%	5.74%	3.90%	8.50%
314.15	-0.99%	5.61%	3.75%	8.23%
318.15	0.23%	7.30%	2.57%	9.89%
Decreasing temperature (K)				
314.15	-1.01%	5.54%	3.68%	8.10%
310.15	-1.11%	6.24%	5.41%	10.29%
306.15	-0.73%	6.17%	4.68%	9.93%
302.15	-1.47%	6.34%	4.35%	9.08%
298.15	-1.61%	6.24%	4.30%	8.81%
294.15	-1.60%	6.27%	4.00%	8.55%

Table 15 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the fifth cycle of temperature variations at 55% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	55% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	β ($^\circ$)		
5th	H e a t i n g	294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.85(1)	28.46(2)	38.20(3)	90	2.051(3)	1.96494
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.89(1)	28.35(2)	38.08(3)	90	2.040(3)	2.01351
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.779(5)	28.169(3)	38.24(1)	90	2.0231(9)	1.75577
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.661(4)	28.057(4)	38.16(2)	90	1.9984(9)	1.81216
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.530(5)	28.137(4)	37.42(1)	90	1.9515(9)	1.95521
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.603(9)	28.24(2)	37.67(3)	90	1.979(2)	1.8181
		318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.601(9)	28.23(2)	37.66(3)	90	1.978(2)	1.81633
	C o o l i n g	318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.601(9)	28.23(2)	37.66(3)	90	1.978(2)	1.81633
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.608(9)	28.23(2)	37.70(3)	90	1.980(2)	1.78902
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.70(1)	28.37(2)	37.95(3)	90	2.015(2)	1.74104
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.73(1)	28.42(2)	38.05(6)	90	2.026(2)	1.75037
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.73(1)	28.41(2)	38.13(3)	90	2.030(2)	1.77696
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.71(4)	28.38(2)	38.16(7)	90	2.027(6)	1.84282
		294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.74(1)	28.42(2)	38.16(3)	90	2.033(2)	1.73924

Table 16 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during fifth cycle at 55% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	-0.21%	0.39%	0.31%	0.54%
302.15	0.38%	1.02%	-0.10%	1.36%
306.15	1.00%	1.42%	0.10%	2.56%
310.15	1.70%	1.13%	2.04%	4.85%
314.15	1.31%	0.77%	1.39%	3.51%
318.15	1.32%	0.81%	1.41%	3.56%
Decreasing temperature (K)				
314.15	1.28%	0.81%	1.31%	3.46%
310.15	0.80%	0.32%	0.65%	1.76%
306.15	0.64%	0.14%	0.39%	1.22%
302.15	0.64%	0.18%	0.18%	1.02%
298.15	0.74%	0.28%	0.10%	1.17%
294.15	0.58%	0.14%	0.10%	0.88%

Table 17 Complete list of refined lattice parameters of Octreotide polycrystalline sample as extracted *via* the Pawley method from the HighScore Plus software for the sixth cycle of temperature variations at 45% rH level (gradual increase and decrease from 294.15 K to 318.15 K). Data collection was conducted utilizing a laboratory X-ray powder diffractometer (X'Pert Pro) equipped with an MHC-trans humidity chamber from Anton Paar [$\lambda = 1.540598 \text{ \AA}$, room temperature] (Malvern Panalytical Company).

Cycle	45% rH	T(K)	Space Group	Unit-cell parameters				V ($\times 10^4 \text{ \AA}^3$)	χ^2
				a (\AA)	b (\AA)	c (\AA)	β ($^\circ$)		
6 th	H e a t i n g	294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.659(9)	28.29(1)	37.87(3)	90	1.999(2)	1.67975
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.65(1)	28.22(2)	38.40(2)	90	2.022(2)	1.61717
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.72(7)	28.36(9)	38.33(2)	90	2.03(1)	1.8045
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.610(7)	28.20(1)	38.18(2)	90	2.00(1)	1.80194
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.65(3)	28.25(4)	38.11(6)	90	2.009(5)	1.3271
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.59(2)	28.23(3)	38.03(5)	90	1.996(4)	1.22282
		318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.58(2)	28.17(3)	38.00(4)	90	1.990(3)	1.24969
	C o o l i n g	318.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.58(2)	28.17(3)	38.00(4)	90	1.990(3)	1.24969
		314.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.57(2)	28.14(3)	38.00(4)	90	1.987(4)	1.2147
		310.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.60(2)	28.21(3)	37.99(4)	90	1.994(4)	1.24997
		306.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.66(8)	28.0(1)	38.21(2)	90	2.00(2)	1.24943
		302.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.7(1)	28.2(2)	38.2(3)	90	2.02(2)	1.24911
		298.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.6(1)	28.2(1)	38.3(2)	90	2.02(2)	1.26409
		294.15	<i>P</i> 2 ₁ 2 ₁ 2 ₁	18.68(9)	28.1(1)	38.3(2)	90	2.02(2)	1.3444

Table 18 Evolution of normalized unit-cell parameters of Octreotide polycrystalline sample during sixth cycle at 45% level of temperature alteration experiments (gradual increase and decrease from 294.15 K to 318.15 K). Delta variations were calculated between samples measured at the lowest and highest rH values as $\Delta(x_i - x_f)/x_i$ %.

Increasing temperature (K)	$\Delta a/a_i$ (%)	$\Delta b/b_i$ (%)	$\Delta c/c_i$ (%)	$\Delta V/V_i$ (%)
294.15	0	0	0	0
298.15	0.05%	0.25%	-1.40%	-1.15%
302.15	-0.33%	-0.25%	-1.21%	-1.55%
306.15	0.26%	0.32%	-0.82%	-0.05%
310.15	0.05%	0.14%	-0.63%	-0.50%
314.15	0.37%	0.21%	-0.42%	0.15%
318.15	0.42%	0.42%	-0.34%	0.45%
Decreasing temperature (K)				
314.15	0.48%	0.53%	-0.34%	0.60%
310.15	0.32%	0.28%	-0.32%	0.25%
306.15	-0.01%	1.03%	-0.90%	-0.05%
302.15	-0.22%	0.32%	-0.87%	-1.05%
298.15	0.32%	0.32%	-1.14%	-1.05%
294.15	-0.11%	0.67%	-1.14%	-1.05%