A His-Pro-Aib Peptide that Exhibits an Asx-Pro Turn-Like Structure

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Supporting Information

Table 1. Crystal data and structure refinement for **Peptide 2.**

Empirical formula	C35 H46 N6 O5	
Formula weight	630.78	
Temperature	293(2) K	
Wavelength	0.71073 ≈	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 12.1058(7) Å	α= 90 deg.
	b = 12.1453(7) Å	β= 90 deg.
	c = 23.493(1) Å	γ = 90 deg.
Volume	3454.1(3) $Å^3$	
Z	4	
Density (calculated)	1.213 Mg/m^3	
Absorption coefficient	0.082 mm^{-1}	
F(000)	1352	
Theta range for data collection	1.73 to 28.30°.	
Index ranges	-15<=h<=16, -15<=k<=14, -27<=l<=31	
Reflections collected	22959	
Independent reflections	8316 [R(int) = 0.1046]	
Completeness to theta = 28.30°	98.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8316 / 0 / 411	
Goodness-of-fit on F^2	1.028	
Final R indices [I>2sigma(I)]	R1 = 0.0639, wR2 = 0.1197	
R indices (all data)	R1 = 0.1376, wR2 = 0.1493	
Absolute structure parameter	-0.7(14)	
Extinction coefficient	0.0050(7)	
Largest diff. peak and hole	0.294 and -0.224 e. $Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å) ^{2}x 10^3) for **Peptide 2**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(3)	6690(2)	7532(2)	302(1)	30(1)
O(1)	5900(2)	7818(2)	-1593(1)	37(1)
O(4)	8573(2)	8205(2)	1370(1)	39(1)
C(7)	6263(2)	8432(2)	176(1)	26(1)
N(2)	6705(2)	9386(2)	362(1)	28(1)
N(3)	6738(2)	8478(2)	1497(1)	28(1)
N(4)	6869(2)	6204(2)	1439(1)	38(1)
O(2)	6449(2)	9264(2)	-1043(1)	51(1)
N(6)	2666(2)	9404(2)	1226(1)	32(1)
C(11)	7694(2)	9402(2)	716(1)	29(1)
N(1)	5331(2)	7917(2)	-702(1)	29(1)
N(5)	4440(2)	9130(2)	1054(1)	38(1)
C(12)	7700(3)	8616(2)	1218(1)	29(1)
C(27)	2732(3)	8717(3)	768(1)	32(1)
C(6)	5182(2)	8462(3)	-154(1)	28(1)
C(29)	1656(3)	9856(3)	1482(1)	33(1)
C(28)	3716(3)	9632(3)	1382(1)	37(1)
C(25)	4292(2)	7874(3)	189(1)	30(1)
O(5)	7129(3)	6073(2)	2384(1)	64(1)
C(26)	3815(2)	8558(3)	661(1)	30(1)
C(8)	6349(3)	10505(2)	199(1)	37(1)
C(10)	7760(3)	10616(3)	901(1)	38(1)
C(5)	5931(3)	8411(3)	-1114(1)	35(1)
C(14)	6929(3)	6635(3)	1962(1)	40(1)
C(30)	1154(2)	10776(3)	1135(1)	31(1)
C(13)	6637(3)	7856(3)	2033(1)	34(1)
C(9)	7317(3)	11214(3)	382(1)	41(1)
C(18)	5946(3)	4406(3)	1273(1)	38(1)
C(35)	1005(3)	11808(3)	1375(2)	45(1)

C(19)	5965(4)	3263(3)	1318(1)	54(1)
C(17)	7034(3)	5038(3)	1327(2)	40(1)
C(16)	7358(3)	8374(3)	2492(1)	46(1)
C(2)	6307(3)	8273(3)	-2137(1)	42(1)
C(31)	792(3)	10607(3)	582(1)	47(1)
C(15)	5436(3)	7890(4)	2214(2)	55(1)
C(32)	304(3)	11447(4)	276(2)	53(1)
C(20)	5023(4)	2652(4)	1241(2)	63(1)
C(33)	146(3)	12461(3)	522(2)	52(1)
C(21)	4044(4)	3144(4)	1127(2)	73(2)
C(1)	5660(3)	9298(3)	-2281(2)	58(1)
C(34)	508(3)	12635(3)	1065(2)	56(1)
C(3)	7548(3)	8455(4)	-2105(2)	58(1)
C(4)	6052(3)	7341(3)	-2556(1)	55(1)
C(23)	4945(3)	4913(3)	1163(2)	50(1)
C(22)	3991(3)	4288(4)	1093(2)	66(1)
C(24)	7756(3)	4896(3)	805(2)	55(1)

Table 3. Bond lengths [Å] and angles [deg] for **Peptide 2.**

O(3)-C(7)	1.245(3)
O(1)-C(5)	1.337(4)
O(1)-C(2)	1.475(4)
O(4)-C(12)	1.221(3)
C(7)-N(2)	1.349(4)
C(7)-C(6)	1.521(4)
N(2)-C(11)	1.458(4)
N(2)-C(8)	1.476(4)
N(3)-C(12)	1.346(4)
N(3)-C(13)	1.473(4)
N(4)-C(14)	1.338(4)
N(4)-C(17)	1.454(4)
O(2)-C(5)	1.223(4)
N(6)-C(28)	1.351(4)

N(6)-C(27)	1.365(4)
N(6)-C(29)	1.469(4)
C(11)-C(12)	1.518(4)
C(11)-C(10)	1.539(4)
N(1)-C(5)	1.350(4)
N(1)-C(6)	1.459(3)
N(5)-C(28)	1.317(4)
N(5)-C(26)	1.381(4)
C(27)-C(26)	1.348(4)
C(6)-C(25)	1.524(4)
C(29)-C(30)	1.511(4)
C(25)-C(26)	1.500(4)
O(5)-C(14)	1.228(4)
C(8)-C(9)	1.517(4)
C(10)-C(9)	1.517(4)
C(14)-C(13)	1.533(5)
C(30)-C(31)	1.385(4)
C(30)-C(35)	1.386(4)
C(13)-C(15)	1.515(5)
C(13)-C(16)	1.525(4)
C(18)-C(23)	1.383(5)
C(18)-C(19)	1.392(5)
C(18)-C(17)	1.530(5)
C(35)-C(34)	1.379(5)
C(19)-C(20)	1.372(6)
C(17)-C(24)	1.515(5)
C(2)-C(1)	1.511(5)
C(2)-C(3)	1.519(5)
C(2)-C(4)	1.532(5)
C(31)-C(32)	1.381(5)
C(32)-C(33)	1.373(5)
C(20)-C(21)	1.355(6)
C(33)-C(34)	1.365(5)
C(21)-C(22)	1.392(6)
C(23)-C(22)	1.392(5)
C(5)-O(1)-C(2)	121.2(3)

O(3)-C(7)-N(2)	120.8(3)
O(3)-C(7)-C(6)	119.9(3)
N(2)-C(7)-C(6)	119.1(3)
C(7)-N(2)-C(11)	121.5(2)
C(7)-N(2)-C(8)	126.2(2)
C(11)-N(2)-C(8)	112.0(2)
C(12)-N(3)-C(13)	123.5(3)
C(14)-N(4)-C(17)	122.7(3)
C(28)-N(6)-C(27)	106.5(3)
C(28)-N(6)-C(29)	126.6(3)
C(27)-N(6)-C(29)	126.9(3)
N(2)-C(11)-C(12)	116.0(2)
N(2)-C(11)-C(10)	102.5(2)
C(12)-C(11)-C(10)	112.5(2)
C(5)-N(1)-C(6)	119.8(3)
C(28)-N(5)-C(26)	105.0(2)
O(4)-C(12)-N(3)	123.7(3)
O(4)-C(12)-C(11)	119.2(3)
N(3)-C(12)-C(11)	116.9(3)
C(26)-C(27)-N(6)	106.9(3)
N(1)-C(6)-C(7)	109.4(2)
N(1)-C(6)-C(25)	110.0(2)
C(7)-C(6)-C(25)	109.1(2)
N(6)-C(29)-C(30)	113.0(2)
N(5)-C(28)-N(6)	111.9(3)
C(26)-C(25)-C(6)	113.8(3)
C(27)-C(26)-N(5)	109.6(3)
C(27)-C(26)-C(25)	126.2(3)
N(5)-C(26)-C(25)	124.2(3)
N(2)-C(8)-C(9)	103.0(2)
C(9)-C(10)-C(11)	102.3(2)
O(2)-C(5)-O(1)	125.9(3)
O(2)-C(5)-N(1)	123.6(3)
O(1)-C(5)-N(1)	110.4(3)
O(5)-C(14)-N(4)	122.3(3)
O(5)-C(14)-C(13)	119.8(3)

N(4)-C(14)-C(13)	117.8(3)
C(31)-C(30)-C(35)	118.3(3)
C(31)-C(30)-C(29)	121.6(3)
C(35)-C(30)-C(29)	120.1(3)
N(3)-C(13)-C(15)	107.9(3)
N(3)-C(13)-C(16)	110.2(3)
C(15)-C(13)-C(16)	109.8(3)
N(3)-C(13)-C(14)	112.6(2)
C(15)-C(13)-C(14)	106.2(3)
C(16)-C(13)-C(14)	110.1(3)
C(8)-C(9)-C(10)	103.2(3)
C(23)-C(18)-C(19)	118.2(3)
C(23)-C(18)-C(17)	123.1(3)
C(19)-C(18)-C(17)	118.7(3)
C(34)-C(35)-C(30)	120.0(3)
C(20)-C(19)-C(18)	121.1(4)
N(4)-C(17)-C(24)	109.7(3)
N(4)-C(17)-C(18)	112.7(3)
C(24)-C(17)-C(18)	111.9(3)
O(1)-C(2)-C(1)	109.2(3)
O(1)-C(2)-C(3)	110.1(3)
C(1)-C(2)-C(3)	113.8(3)
O(1)-C(2)-C(4)	102.2(3)
C(1)-C(2)-C(4)	111.1(3)
C(3)-C(2)-C(4)	109.8(3)
C(32)-C(31)-C(30)	121.0(3)
C(33)-C(32)-C(31)	120.2(3)
C(21)-C(20)-C(19)	120.9(4)
C(34)-C(33)-C(32)	119.1(3)
C(20)-C(21)-C(22)	119.5(4)
C(33)-C(34)-C(35)	121.4(4)
C(18)-C(23)-C(22)	120.4(4)
C(23)-C(22)-C(21)	119.9(4)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **Peptide 2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(3)	33(1)	24(1)	33(1)	-3(1)	-2(1)	7(1)
O(1)	47(1)	38(1)	24(1)	-3(1)	4(1)	-7(1)
O(4)	32(1)	45(1)	40(1)	-3(1)	-6(1)	9(1)
C(7)	29(2)	29(2)	21(1)	-2(1)	5(1)	1(1)
N(2)	31(1)	23(1)	31(1)	-1(1)	-5(1)	1(1)
N(3)	28(1)	27(1)	30(1)	-2(1)	-1(1)	5(1)
N(4)	60(2)	23(1)	30(1)	3(1)	-7(1)	0(1)
O(2)	63(2)	47(2)	42(1)	-11(1)	8(1)	-24(1)
N(6)	32(1)	34(2)	28(1)	-6(1)	-1(1)	3(1)
C(11)	29(2)	28(2)	30(2)	-3(1)	0(1)	-3(1)
N(1)	36(2)	26(1)	25(1)	-3(1)	3(1)	-4(1)
N(5)	30(1)	46(2)	39(2)	-10(1)	-5(1)	6(1)
C(12)	32(2)	26(2)	29(2)	-6(1)	-6(1)	1(1)
C(27)	34(2)	34(2)	29(2)	-8(1)	0(1)	-1(1)
C(6)	33(2)	27(2)	24(1)	-1(1)	-1(1)	3(1)
C(29)	33(2)	39(2)	27(2)	-6(1)	5(1)	4(1)
C(28)	38(2)	40(2)	32(2)	-8(1)	-7(2)	3(2)
C(25)	28(2)	31(2)	31(2)	-4(1)	-3(1)	-1(1)
O(5)	114(3)	41(2)	37(1)	11(1)	-14(2)	7(2)
C(26)	29(2)	32(2)	29(2)	-3(1)	0(1)	0(1)
C(8)	47(2)	23(2)	40(2)	2(1)	-4(2)	6(2)
C(10)	48(2)	28(2)	39(2)	-6(2)	-2(2)	-6(2)
C(5)	37(2)	37(2)	31(2)	-4(2)	-2(1)	-3(2)
C(14)	50(2)	39(2)	29(2)	3(2)	-5(2)	0(2)
C(30)	24(2)	40(2)	29(2)	0(1)	4(1)	-1(1)
C(13)	42(2)	33(2)	26(2)	0(1)	-1(1)	2(2)
C(9)	53(2)	24(2)	46(2)	-3(2)	-3(2)	-2(2)
C(18)	53(2)	32(2)	29(2)	-2(1)	7(2)	-2(2)
C(35)	56(2)	34(2)	45(2)	-9(2)	-5(2)	-5(2)

C(19)	85(3)	38(2)	38(2)	-3(2)	13(2)	-5(2)
C(17)	52(2)	26(2)	42(2)	3(2)	-4(2)	4(2)
C(16)	68(2)	43(2)	28(2)	-6(2)	-8(2)	3(2)
C(2)	51(2)	47(2)	28(2)	7(2)	5(2)	-5(2)
C(31)	53(2)	53(2)	35(2)	-7(2)	-5(2)	11(2)
C(15)	45(2)	80(3)	40(2)	16(2)	10(2)	5(2)
C(32)	51(2)	71(3)	37(2)	5(2)	-4(2)	8(2)
C(20)	88(3)	51(3)	49(2)	-3(2)	23(2)	-25(3)
C(33)	49(2)	47(2)	60(2)	24(2)	3(2)	-3(2)
C(21)	83(4)	88(4)	49(2)	-26(2)	35(3)	-44(3)
C(1)	71(3)	52(3)	53(2)	8(2)	-1(2)	2(2)
C(34)	68(3)	29(2)	71(3)	0(2)	-6(2)	-1(2)
C(3)	51(2)	70(3)	52(2)	7(2)	10(2)	-6(2)
C(4)	77(3)	60(3)	28(2)	-2(2)	1(2)	-5(2)
C(23)	50(2)	52(2)	50(2)	-10(2)	4(2)	4(2)
C(22)	49(2)	102(4)	48(2)	-21(2)	11(2)	-2(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **Peptide 2**.

	x	y	z	U(eq)
H(3A)	6152	8767	1354	34
H(4A)	6726	6633	1157	45
H(11A)	8334	9238	475	35
H(1A)	5038	7283	-763	35
H(27A)	2143	8416	567	39
H(6A)	4962	9229	-216	33
H(29A)	1827	10130	1860	39
H(29B)	1118	9269	1522	39
H(28A)	3904	10088	1684	44
H(25A)	3700	7657	-66	36

H(25B)	4604	7208	351	36
H(8A)	5678	10714	398	44
H(8B)	6224	10557	-208	44
H(10A)	7304	10754	1233	46
H(10B)	8515	10832	983	46
H(9A)	7077	11953	478	49
H(9B)	7871	11256	84	49
H(35A)	1240	11943	1745	54
H(19A)	6626	2909	1401	64
H(17A)	7436	4729	1652	48
H(16A)	7170	9138	2533	70
H(16B)	8121	8309	2386	70
H(16C)	7236	8002	2847	70
H(31A)	878	9918	415	56
H(15A)	4986	7557	1925	82
H(15B)	5213	8642	2268	82
H(15C)	5350	7495	2565	82
H(32A)	82	11326	-98	64
H(20A)	5058	1888	1268	75
H(33A)	-204	13021	321	63
H(21A)	3412	2722	1072	88
H(1B)	5828	9866	-2011	88
H(1C)	4884	9138	-2268	88
H(1D)	5855	9543	-2657	88
H(34A)	418	13326	1229	67
H(3B)	7703	9042	-1843	86
H(3C)	7822	8647	-2476	86
H(3D)	7901	7793	-1977	86
H(4B)	5268	7229	-2574	83
H(4C)	6403	6676	-2429	83
H(4D)	6324	7533	-2926	83
H(23A)	4910	5676	1135	60
H(22A)	3319	4633	1023	79
H(24A)	8431	5299	856	82
H(24B)	7374	5170	476	82
H(24C)	7921	4129	753	82

Table 6. NMR data for peptide **2**.

^{1}H NMR (C_6D_6, 499.87 Hz, 30 deg C) δ 8.974 (s,1H), 8.029 (d, J=8 Hz, 1H), 7.512 (d, J=8 Hz), 6.98-7.2 (m, 6H), 8.867 (s, 1H), 6.696 (m, 2H), 6.046 (s, 1H), 5.675 (d, J= 8 Hz), 5.526 (quin, J=7.5 Hz, 1H), 4.511 (m, 1H), 4.302 (m, 1H), 4.178 (m, 2H), 2.855 (m, 2H), 2.593 (m, 1H), 2.162 (m, 1H), 2.064 (s, 3H), 1.715 (m, 1H), 1.688 (d, J=7.0 Hz, 3H), 1.632 (m, 1H), 1.577 (s, 3H), 1.430 (s, 9 H), 1.176 (m 1H), 0.913 (sep., 6.5 Hz, 1H)

NOESY (C_6D_6, 499.87 Hz, 30 deg C) pw=9.3 @ tpwr=60, mix=0.700 sec, nt=8, ni=200, dpwr=39. Temp=30 deg C.