Crystal structure of Boc-L-Ile- Δ Phe-Ile-OCH₃, C₂₇H₄₁N₃O₆ · H₂O

J. Makker, S. Dey, P. Kumar and T. P. Singh*

All India Institute of Medical Sciences, Department of Biophysics, Ansari Nagar, New Delhi-110029, India

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Abstract

C₂₇H₄₃N₃O₇, orthorhombic, *P*2₁2₁2 (No. 18), *a* = 12.202(1) Å, *b* = 27.790(1) Å, *c* = 9.128(1) Å, *V* = 3095.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.054, *wR*_{ref}(*F*²) = 0.180, *T* = 293 K.

Source of material

The title peptide *N*-butyloxycarbonyl-isoleucyl-dehydro-phenylalanine-isoleucine has been synthesised using the mixed anhydride coupling and the azalactone method according to [1] and [2], respectively. The peptide was crystallised from its solution in acetone-water mixture (4:1) by slow evaporation.

Experimental details

The two H atoms attached to O5 could not be located.

Discussion

The peptide Boc-L-Ile- Δ Phe-L-Ile-OCH₃ was synthesised as part of the program on peptide design with α,β -dehydro-residues [3].

The structure contains a peptide and a hydrogen bonded water molecule. It adopts a poorly folded conformation with φ,ψ torsion angles: $\varphi_1 = -124.9(3)^\circ$, $\psi_1 = 162.4(2)^\circ$, $\varphi_2 = 51.0(3)^\circ$, $\psi_2 = 36.8(3)^\circ$, $\varphi_3 = -117.9(3)^\circ$, $\psi_3^T = 167.3(2)^\circ$. The structure of the peptide does not form an intramolecular hydrogen bond. However, two intra residue hydrogen bonds are present that determine the conformation of (*i*+3) substituted Ile in the structure. The water molecule is also involved in an intermolecular hydrogen bond with carbonyl oxygen atom of Δ Phe. There are two more intermolecular hydrogen bonds involving NH groups of first two amino acids and carbonyl oxygen atoms of symmetry related last two amino acids in the structure.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.3 × 0.4 × 0.6 mm
Wavelength:	Cu <i>K</i> α radiation (1.54180 Å)
μ :	6.59 cm ⁻¹
Diffractionmeter, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{\max}$:	150.64°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3311, 3306
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3181
<i>N</i> (<i>param</i>) _{refined} :	335
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(01A)	4c	1.1675	0.9932	1.5315	0.08
H(01B)	4c	1.2735	1.0021	1.6245	0.08
H(01C)	4c	1.1965	0.9586	1.6616	0.08
H(02A)	4c	1.4492	0.9301	1.4723	0.08
H(02B)	4c	1.3862	0.9013	1.5937	0.08
H(02C)	4c	1.4168	0.9555	1.6195	0.08
H(03A)	4c	1.2464	0.9776	1.2904	0.08
H(03B)	4c	1.3670	0.9581	1.2902	0.08
H(03C)	4c	1.3389	1.0061	1.3721	0.08
H(1)	4c	1.1336	0.8340	1.4521	0.08
H(1A)	4c	1.2488	0.7936	1.2258	0.08
H(1B)	4c	1.2404	0.7172	1.3302	0.08
H(1GA)	4c	1.1431	0.7574	1.5820	0.08
H(1GB)	4c	1.0794	0.7263	1.4671	0.08
H(1DA)	4c	1.1220	0.6800	1.6699	0.08
H(1DB)	4c	1.2471	0.6903	1.6469	0.08
H(1DC)	4c	1.1827	0.6592	1.5324	0.08
H(1GC)	4c	1.3957	0.7637	1.3777	0.08
H(1GD)	4c	1.3751	0.7274	1.5064	0.08
H(1GE)	4c	1.3434	0.7818	1.5246	0.08
H(2)	4c	1.1543	0.7335	1.0920	0.08
H(2B)	4c	0.9142	0.6826	0.9356	0.08
H(2DA)	4c	1.0925	0.6611	1.2376	0.08
H(2DB)	4c	0.9913	0.6071	0.8536	0.08
H(2EA)	4c	1.1733	0.5882	1.2934	0.08
H(2EB)	4c	1.0678	0.5335	0.9123	0.08
H(2N)	4c	1.1693	0.5262	1.1270	0.08
H(3)	4c	1.0498	0.8111	0.9367	0.08
H(3A)	4c	0.8458	0.8447	0.8644	0.08
H(3B)	4c	0.9098	0.9248	0.9044	0.08
H(3GA)	4c	1.0705	0.8942	1.0841	0.08
H(3GB)	4c	1.0962	0.8878	0.9197	0.08
H(3GC)	4c	0.8708	0.9215	1.1523	0.08
H(3GD)	4c	0.9042	0.8671	1.1596	0.08
H(3GE)	4c	0.7986	0.8826	1.0739	0.08
H(3DA)	4c	1.0648	0.9738	0.9929	0.08
H(3DB)	4c	1.1578	0.9482	0.9036	0.08
H(3DC)	4c	1.1594	0.9469	1.0753	0.08
H(4TA)	4c	0.9645	0.9287	0.4808	0.08
H(4TB)	4c	0.8592	0.9007	0.4313	0.08
H(4TC)	4c	0.9702	0.8732	0.4517	0.08

* Correspondence author (e-mail: tps@aiims.aiims.ac.in)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(01)	4c	1.226(1)	0.9779(2)	1.584(1)	0.22(1)	0.087(3)	0.144(6)	−0.030(5)	0.008(7)	−0.058(4)
C(02)	4c	1.3946(8)	0.9322(3)	1.548(2)	0.179(9)	0.132(6)	0.25(1)	−0.058(6)	−0.10(1)	−0.022(8)
C(03)	4c	1.313(1)	0.9746(3)	1.347(1)	0.186(8)	0.099(4)	0.149(7)	−0.038(5)	−0.005(7)	0.010(4)
C(0)	4c	1.2895(6)	0.9470(2)	1.4827(7)	0.132(5)	0.071(2)	0.108(4)	−0.048(3)	−0.002(4)	−0.023(3)
O(0)	4c	1.2214(3)	0.9057(1)	1.4571(4)	0.090(2)	0.064(1)	0.081(2)	−0.025(1)	0.018(2)	−0.027(1)
C(0P)	4c	1.2518(3)	0.8694(1)	1.3690(4)	0.052(2)	0.061(2)	0.061(2)	−0.016(1)	0.011(1)	−0.015(1)
O(0P)	4c	1.3277(3)	0.8704(1)	1.2845(5)	0.094(2)	0.093(2)	0.118(3)	−0.044(2)	0.057(2)	−0.031(2)
N(1)	4c	1.1846(2)	0.83195(8)	1.3874(3)	0.038(1)	0.050(1)	0.046(1)	−0.0058(8)	0.0072(9)	−0.0105(9)
C(1A)	4c	1.1942(2)	0.78808(8)	1.3028(2)	0.029(1)	0.049(1)	0.035(1)	−0.0021(8)	0.0024(8)	−0.0055(9)
C(1B)	4c	1.2333(2)	0.7448(1)	1.3965(3)	0.033(1)	0.059(1)	0.046(1)	0.009(1)	−0.007(1)	−0.004(1)
C(1G2)	4c	1.1501(3)	0.7309(1)	1.5136(4)	0.061(2)	0.071(2)	0.051(1)	0.016(1)	0.003(1)	0.014(1)
C(1D)	4c	1.1780(6)	0.6860(2)	1.5984(6)	0.113(4)	0.095(3)	0.087(3)	0.025(3)	0.006(3)	0.037(3)
C(1G1)	4c	1.3475(3)	0.7554(2)	1.4569(5)	0.050(2)	0.098(3)	0.081(2)	0.012(2)	−0.027(2)	−0.013(2)
C(1P)	4c	1.0851(2)	0.77740(8)	1.2286(2)	0.029(1)	0.0419(9)	0.0321(9)	0.0002(8)	−0.0004(8)	0.0038(8)
O(1P)	4c	0.9988(2)	0.79462(7)	1.2674(2)	0.0326(9)	0.064(1)	0.0415(8)	0.0090(7)	0.0023(7)	−0.0010(7)
N(2)	4c	1.0916(2)	0.74551(7)	1.1150(2)	0.027(1)	0.0415(8)	0.0397(9)	−0.0003(7)	−0.0046(7)	−0.0018(7)
C(2A)	4c	0.9978(2)	0.73181(8)	1.0344(3)	0.029(1)	0.043(1)	0.040(1)	0.0002(8)	−0.0070(9)	0.0019(9)
C(2B)	4c	0.9746(2)	0.6865(1)	0.9966(3)	0.043(1)	0.045(1)	0.057(1)	−0.004(1)	−0.017(1)	−0.001(1)
C(2G)	4c	1.0318(3)	0.64182(9)	1.0382(4)	0.050(2)	0.044(1)	0.065(2)	−0.005(1)	−0.016(1)	0.003(1)
C(2D1)	4c	1.0882(3)	0.6357(1)	1.1715(4)	0.069(2)	0.048(1)	0.074(2)	−0.007(1)	−0.027(2)	0.011(1)
C(2D2)	4c	1.0272(5)	0.6034(1)	0.9428(6)	0.105(3)	0.052(2)	0.095(3)	0.009(2)	−0.043(3)	−0.009(2)
C(2E1)	4c	1.1373(4)	0.5921(1)	1.2044(6)	0.075(2)	0.064(2)	0.104(3)	−0.004(2)	−0.031(2)	0.031(2)
C(2E2)	4c	1.0748(6)	0.5594(2)	0.9760(8)	0.135(4)	0.052(2)	0.123(4)	0.016(2)	−0.031(4)	−0.009(2)
C(2N)	4c	1.1330(5)	0.5548(2)	1.1062(8)	0.103(3)	0.056(2)	0.131(4)	0.018(2)	−0.030(3)	0.017(2)
C(2P)	4c	0.9259(2)	0.77173(8)	0.9753(3)	0.032(1)	0.041(1)	0.039(1)	0.0019(8)	−0.0069(9)	−0.0011(8)
O(2P)	4c	0.8263(2)	0.76643(7)	0.9689(3)	0.030(1)	0.056(1)	0.069(1)	−0.0006(7)	−0.0122(9)	0.0051(9)
N(3)	4c	0.9795(2)	0.81051(8)	0.9291(2)	0.033(1)	0.0444(9)	0.0406(9)	0.0047(8)	−0.0003(8)	0.0066(8)
C(3A)	4c	0.9244(2)	0.85187(9)	0.8665(3)	0.047(1)	0.048(1)	0.036(1)	0.014(1)	0.003(1)	0.0039(9)
C(3B)	4c	0.9403(5)	0.8977(1)	0.9595(4)	0.160(5)	0.051(2)	0.048(2)	0.039(2)	−0.020(2)	−0.007(1)
C(3G1)	4c	1.0574(9)	0.9082(2)	0.988(1)	0.222(9)	0.073(3)	0.168(7)	−0.018(4)	−0.116(7)	−0.010(4)
C(3G2)	4c	0.8721(8)	0.8917(2)	1.0994(5)	0.243(9)	0.121(4)	0.048(2)	0.107(6)	0.017(3)	0.002(2)
C(3D)	4c	1.113(1)	0.9466(6)	0.990(3)	0.17(1)	0.34(2)	0.74(7)	−0.00(2)	0.01(2)	0.35(4)
C(3P)	4c	0.9617(2)	0.85891(9)	0.7093(3)	0.042(1)	0.046(1)	0.036(1)	0.0049(9)	0.0030(9)	0.0018(9)
O(3P)	4c	1.0387(2)	0.83898(9)	0.6540(2)	0.062(1)	0.076(1)	0.048(1)	0.022(1)	0.013(1)	0.0067(9)
O(4)	4c	0.8979(2)	0.88975(9)	0.6396(2)	0.067(1)	0.080(1)	0.0421(9)	0.028(1)	0.005(1)	0.0194(9)
C(4)	4c	0.9252(4)	0.8989(2)	0.4881(3)	0.084(3)	0.086(2)	0.041(1)	0.023(2)	0.006(2)	0.021(1)
O(5)	4c	1.2153(3)	0.8068(2)	0.8712(5)	0.055(2)	0.143(3)	0.112(3)	0.006(2)	−0.003(2)	0.051(3)

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