Crystal structure of Boc-Leu-ΔPhe-ΔPhe-Ile-OCH_3, $C_{36}H_{48}N_4O_7$

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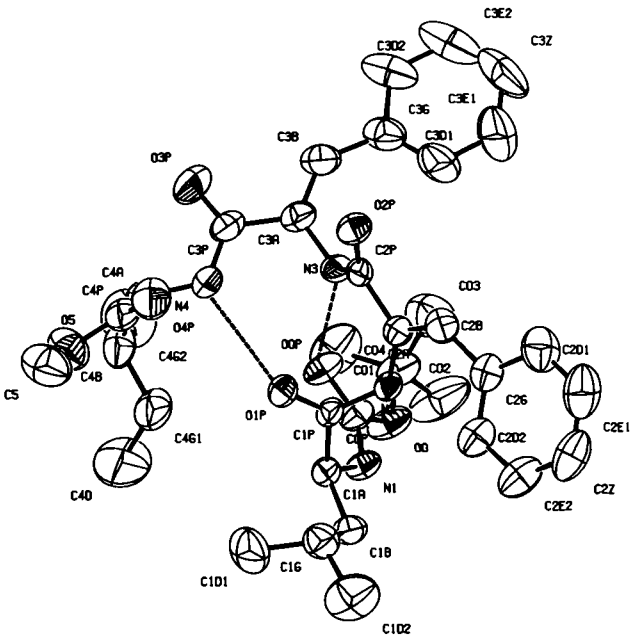

Abstract

$C_{36}H_{48}N_4O_7$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 11.753(3)$ Å, $b = 14.99(1)$ Å, $c = 21.464(3)$ Å, $V = 3781.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.058$, $wR_{ref}(F^2) = 0.183$, $T = 293$ K.

Source of material

The title peptide 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 method.

Discussion

The peptide adopts a 3_{10} helical conformation which deviates substantially at Ile residue indicating the strong steric effects of a branched β-carbon residue. However, the two intramolecular hydrogen bonds involving NH's of residues 3 and 4, and O's of Boc group and residue 1 are well formed [N3–H3···O0P = 2.878(5) Å and N4–H4···O1P = 3.049(4) Å]. The tetrapeptides [3-6] with two ΔPhe residues at (i+2) and (i+3) positions give rise to folded conformations irrespective of any residue at (i+1) and (i+4) positions. Two intermolecular hydrogen bonds characterize the packing: the former involves NH of residue 1 and O2P of residue 2 while the second one is between the NH of residue 2 and O4P of residue 4. Relevant geometric parameters together with those of the intramolecular hydrogen bonds are included in the deposit CIF data. It may be noted that in all cases the H···A distances are within 3 Å.

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Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.1 × 0.2 × 0.3 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	6.44 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	150.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4218, 4216
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3275
$N(param)_{refined}$:	425
Programs:	SDP [7], SHELXS-97 [8], SHELXL-97 [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(02A)	4a	−0.3197	−0.2506	−0.2945	0.08
H(02B)	4a	−0.2539	−0.2483	−0.2310	0.08
H(02C)	4a	−0.3860	−0.2629	−0.2317	0.08
H(03A)	4a	−0.2840	−0.4774	−0.1985	0.08
H(03B)	4a	−0.3656	−0.4030	−0.1732	0.08
H(03C)	4a	−0.2335	−0.3891	−0.1708	0.08
H(04A)	4a	−0.3730	−0.4792	−0.2988	0.08
H(04B)	4a	−0.3819	−0.3919	−0.3386	0.08
H(04C)	4a	−0.4606	−0.4049	−0.2804	0.08
H(1)	4a	0.0014	−0.3947	−0.3141	0.08
H(1A)	4a	0.0105	−0.5439	−0.3745	0.08
H(1BA)	4a	0.1857	−0.4579	−0.3081	0.08
H(1BB)	4a	0.1599	−0.4465	−0.3791	0.08
H(1G)	4a	0.2518	−0.6035	−0.3259	0.08
H(1DA)	4a	0.1506	−0.6390	−0.4173	0.08
H(1DB)	4a	0.2808	−0.6578	−0.4261	0.08
H(1DC)	4a	0.2261	−0.5719	−0.4550	0.08
H(1DD)	4a	0.3816	−0.4863	−0.3281	0.08
H(1DE)	4a	0.3666	−0.4790	−0.4005	0.08
H(1DF)	4a	0.4215	−0.5646	−0.3713	0.08
H(2)	4a	0.0545	−0.5210	−0.2176	0.08
H(2B)	4a	0.1079	−0.6910	−0.1050	0.08
H(2DA)	4a	0.2364	−0.6273	−0.0325	0.08
H(2DB)	4a	0.2626	−0.5560	−0.2122	0.08
H(2EA)	4a	0.4059	−0.5564	−0.0129	0.08
H(2EB)	4a	0.4342	−0.4904	−0.1895	0.08
H(2Z)	4a	0.5054	−0.4882	−0.0920	0.08
H(3)	4a	−0.1522	−0.6224	−0.2197	0.08
H(3B)	4a	−0.3821	−0.7767	−0.1646	0.08
H(3DA)	4a	−0.1804	−0.6342	−0.0904	0.08
H(3DB)	4a	−0.4710	−0.7644	−0.0716	0.08
H(3EA)	4a	−0.1969	−0.5832	0.0088	0.08
H(3E2)	4a	−0.4848	−0.7094	0.0293	0.08
H(3Z)	4a	−0.3463	−0.6233	0.0706	0.08
H(4)	4a	−0.1417	−0.7372	−0.3023	0.08
H(4A)	4a	−0.2787	−0.8558	−0.3631	0.08
H(4B)	4a	−0.2484	−0.7896	−0.4592	0.08
H(4GA)	4a	−0.1523	−0.6397	−0.4006	0.08
H(4GB)	4a	−0.0758	−0.7139	−0.4304	0.08
H(4GC)	4a	−0.4119	−0.7561	−0.4049	0.08
H(4GD)	4a	−0.3755	−0.6753	−0.4468	0.08
H(4GE)	4a	−0.3520	−0.6731	−0.3749	0.08

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(4DA)	4*a*	−0.0987	−0.5963	−0.4995	0.08
H(4DB)	4*a*	−0.2310	−0.6097	−0.4982	0.08
H(4DC)	4*a*	−0.1526	−0.6829	−0.5278	0.08
H(5A)	4*a*	−0.0014	−0.9650	−0.4959	0.08
H(5B)	4*a*	−0.0143	−1.0096	−0.4302	0.08
H(5C)	4*a*	0.0662	−0.9271	−0.4388	0.08

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(01)	4*a*	−0.2984(4)	−0.3728(3)	−0.2569(3)	0.052(2)	0.044(2)	0.108(4)	0.002(2)	0.024(2)	−0.003(2)
C(02)	4*a*	−0.3160(6)	−0.2750(4)	−0.2532(5)	0.090(4)	0.058(3)	0.24(1)	−0.004(3)	0.066(6)	−0.020(5)
C(03)	4*a*	−0.2951(7)	−0.4142(5)	−0.1944(3)	0.113(5)	0.114(5)	0.100(4)	0.005(4)	0.029(4)	0.001(4)
C(04)	4*a*	−0.3864(5)	−0.4161(4)	−0.2973(4)	0.052(3)	0.098(4)	0.164(7)	0.011(3)	−0.012(4)	−0.013(5)
O(0)	4*a*	−0.1858(2)	−0.3790(2)	−0.2869(2)	0.050(1)	0.035(1)	0.107(2)	−0.000(1)	0.017(2)	0.006(1)
C(0P)	4*a*	−0.1365(3)	−0.4572(2)	−0.2965(2)	0.048(2)	0.031(1)	0.058(2)	−0.001(1)	−0.006(2)	0.009(2)
O(0P)	4*a*	−0.1817(2)	−0.5313(2)	−0.2906(1)	0.048(1)	0.036(1)	0.083(2)	−0.007(1)	−0.003(1)	0.011(1)
N(1)	4*a*	−0.0283(3)	−0.4472(2)	−0.3145(2)	0.044(2)	0.030(1)	0.063(2)	−0.004(1)	0.003(1)	0.005(1)
C(1A)	4*a*	0.0407(3)	−0.5222(2)	−0.3347(2)	0.049(2)	0.035(2)	0.041(2)	−0.003(1)	0.000(2)	0.000(1)
C(1B)	4*a*	0.1611(3)	−0.4893(2)	−0.3452(2)	0.050(2)	0.043(2)	0.054(2)	−0.006(2)	0.009(2)	0.002(2)
C(1G)	4*a*	0.2501(4)	−0.5608(3)	−0.3604(2)	0.060(2)	0.060(2)	0.063(2)	0.000(2)	0.009(2)	−0.003(2)
C(1D1)	4*a*	0.2245(6)	−0.6121(4)	−0.4202(3)	0.110(4)	0.083(3)	0.078(3)	0.013(3)	0.020(3)	−0.023(3)
C(1D2)	4*a*	0.3653(5)	−0.5189(5)	−0.3655(4)	0.069(3)	0.102(5)	0.152(6)	0.009(4)	0.024(4)	−0.005(5)
C(1P)	4*a*	0.0378(3)	−0.5989(2)	−0.2878(2)	0.042(2)	0.028(1)	0.044(2)	−0.004(1)	−0.000(1)	−0.003(1)
O(1P)	4*a*	0.0296(2)	−0.6764(1)	−0.3054(1)	0.068(2)	0.028(1)	0.051(1)	−0.003(1)	0.004(1)	−0.005(1)
N(2)	4*a*	0.0473(3)	−0.5763(2)	−0.2275(1)	0.049(2)	0.026(1)	0.042(1)	−0.002(1)	−0.003(1)	0.000(1)
C(2A)	4*a*	0.0456(3)	−0.6415(2)	−0.1799(2)	0.050(2)	0.026(1)	0.043(2)	0.004(1)	0.004(2)	−0.001(1)
C(2B)	4*a*	0.1232(4)	−0.6478(2)	−0.1350(2)	0.066(2)	0.040(2)	0.048(2)	0.006(2)	−0.004(2)	0.003(2)
C(2G)	4*a*	0.2292(3)	−0.5976(2)	−0.1248(2)	0.050(2)	0.044(2)	0.061(2)	0.007(2)	−0.009(2)	−0.005(2)
C(2D1)	4*a*	0.2748(5)	−0.5986(4)	−0.0646(2)	0.080(3)	0.084(3)	0.066(3)	0.008(3)	−0.024(3)	0.001(2)
C(2D2)	4*a*	0.2904(4)	−0.5568(3)	−0.1716(2)	0.057(2)	0.050(2)	0.077(3)	0.001(2)	−0.012(2)	0.001(2)
C(2E1)	4*a*	0.3768(5)	−0.5569(5)	−0.0532(3)	0.078(4)	0.115(5)	0.094(4)	0.002(4)	−0.040(3)	−0.022(4)
C(2E2)	4*a*	0.3932(4)	−0.5174(3)	−0.1576(3)	0.057(2)	0.060(3)	0.107(4)	−0.005(2)	−0.013(3)	0.007(3)
C(2Z)	4*a*	0.4363(5)	−0.5161(4)	−0.1001(3)	0.059(3)	0.072(3)	0.123(5)	−0.002(2)	−0.033(3)	−0.017(3)
C(2P)	4*a*	−0.0558(3)	−0.7035(2)	−0.1762(1)	0.057(2)	0.027(1)	0.034(1)	−0.003(1)	0.006(1)	−0.005(1)
O(2P)	4*a*	−0.0511(3)	−0.7729(2)	−0.1469(1)	0.071(2)	0.030(1)	0.057(2)	−0.004(1)	0.001(1)	0.007(1)
N(3)	4*a*	−0.1516(3)	−0.6745(2)	−0.2028(1)	0.056(2)	0.030(1)	0.046(1)	−0.005(1)	0.007(1)	0.004(1)
C(3A)	4*a*	−0.2533(3)	−0.7269(2)	−0.2044(2)	0.052(2)	0.040(2)	0.061(2)	−0.006(2)	0.005(2)	0.004(2)
C(3B)	4*a*	−0.3220(4)	−0.7380(3)	−0.1565(2)	0.065(3)	0.058(2)	0.068(3)	−0.008(2)	0.016(2)	0.008(2)
C(3G)	4*a*	−0.3231(5)	−0.7022(3)	−0.0935(2)	0.080(3)	0.053(2)	0.070(3)	0.006(2)	0.022(2)	0.009(2)
C(3D1)	4*a*	−0.2439(7)	−0.6504(4)	−0.0672(3)	0.125(5)	0.094(4)	0.075(3)	−0.010(4)	0.051(4)	−0.023(3)
C(3D2)	4*a*	−0.4149(6)	−0.7268(5)	−0.0561(3)	0.094(4)	0.104(4)	0.085(4)	0.023(3)	0.039(3)	0.036(3)
C(3E1)	4*a*	−0.253(1)	−0.6203(6)	−0.0071(3)	0.199(9)	0.122(6)	0.094(4)	−0.018(7)	0.029(6)	−0.052(4)
C(3E2)	4*a*	−0.4220(8)	−0.6944(6)	0.0052(3)	0.130(6)	0.134(6)	0.076(4)	0.055(5)	0.046(4)	0.044(4)
C(3Z)	4*a*	−0.3417(9)	−0.6430(5)	0.0296(3)	0.179(8)	0.121(6)	0.057(3)	0.042(6)	0.046(5)	−0.004(4)
C(3P)	4*a*	−0.2783(4)	−0.7769(3)	−0.2638(2)	0.058(2)	0.048(2)	0.068(2)	−0.019(2)	0.009(2)	−0.005(2)
O(3P)	4*a*	−0.3657(3)	−0.8197(3)	−0.2692(2)	0.078(2)	0.099(3)	0.101(3)	−0.048(2)	0.016(2)	−0.027(2)
N(4)	4*a*	−0.2008(3)	−0.7698(2)	−0.3085(2)	0.061(2)	0.049(2)	0.051(2)	−0.020(2)	−0.003(2)	−0.002(1)
C(4A)	4*a*	−0.2133(4)	−0.8157(3)	−0.3677(2)	0.070(2)	0.043(2)	0.053(2)	−0.016(2)	−0.007(2)	−0.006(2)
C(4B)	4*a*	−0.2422(5)	−0.7530(3)	−0.4216(2)	0.091(3)	0.052(2)	0.062(2)	0.002(2)	−0.027(3)	−0.006(2)
C(4G1)	4*a*	−0.1492(7)	−0.6845(4)	−0.4331(3)	0.149(6)	0.057(3)	0.069(3)	−0.032(3)	−0.025(3)	0.015(2)
C(4G2)	4*a*	−0.3555(7)	−0.7106(6)	−0.4111(4)	0.116(5)	0.124(6)	0.128(6)	0.041(5)	−0.028(5)	0.010(5)
C(4D)	4*a*	−0.159(1)	−0.6393(5)	−0.4953(3)	0.22(1)	0.101(5)	0.114(5)	−0.029(6)	−0.038(7)	0.061(4)
C(4P)	4*a*	−0.1113(4)	−0.8745(2)	−0.3779(2)	0.081(3)	0.034(2)	0.053(2)	−0.009(2)	−0.005(2)	0.009(2)
O(4P)	4*a*	−0.0503(4)	−0.9026(2)	−0.3376(2)	0.105(3)	0.052(2)	0.068(2)	0.001(2)	−0.015(2)	0.009(1)
O(5)	4*a*	−0.0968(4)	−0.8955(2)	−0.4379(2)	0.114(3)	0.072(2)	0.060(2)	0.011(2)	0.007(2)	0.004(2)
C(5)	4*a*	−0.0039(6)	−0.9541(5)	−0.4518(3)	0.118(5)	0.083(4)	0.088(4)	0.017(4)	0.025(4)	0.013(3)

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