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Structure of *N-tert*-Butoxycarbonyl-L-phenylalanine Benzyl Ester

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Abstract. $C_{21}H_{25}NO_4$, $M_r = 355.4$, monoclinic, $P2_1$, $a = 5.206$ (2), $b = 17.294$ (4), $c = 10.972$ (2) Å, $\beta = 98.91$ (1)°, $V = 976$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}21$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0.68$ mm^{-1}, $F(000) = 380$, $T = 293$ K, final $R = 0.071$ for 1186 observed reflections. The structure is stabilized in the **a** direction by means of intermolecular hydrogen bonds [$N(1)\cdots O(2^{i}) = 3.01$ (1) Å, (i) = $x + 1$, y, z]. The urethane amide bond adopts the *trans* conformation [H(1)—N(1)—C(5)═O(2) = 169 (3)°]. The butoxycarbonyl (Boc) moiety is directed away from both the phenylalanine aromatic ring and the benzyl ester ring, in contrast to the arrangement observed in Bocphenylalanine phenacyl ester [Vlassi, Germain, Matsoukas, Psachoulia, Voliotis & Leban (1987). *Acta Cryst.* C**43**, 2173–2175]. The orientation assumed by the Boc group may be the result of steric restrictions imposed by both rings.

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
C(1)	205 (10)	7186	3121 (5)	5.53 (13)
C(2)	−837 (13)	7430 (5)	4271 (6)	6.63 (18)
C(3)	1770 (12)	7833 (5)	2663 (7)	7.00 (18)
C(4)	−1886 (11)	6904 (6)	2108 (6)	6.44 (16)
C(5)	1583 (10)	5897 (4)	3900 (5)	4.85 (13)
C(6)	3531 (10)	4695 (4)	4750 (5)	5.06 (12)
C(7)	4278 (11)	4683 (5)	6127 (5)	5.43 (14)
C(8)	7183 (13)	5143 (5)	7813 (5)	6.55 (17)
C(9)	9352 (12)	5730 (5)	8104 (5)	6.01 (17)
C(10)	10276 (16)	6176 (6)	7244 (7)	7.57 (23)
C(11)	12311 (17)	6695 (7)	7596 (10)	8.96 (25)
C(12)	13391 (17)	6757 (7)	8835 (10)	9.24 (30)
C(13)	12518 (16)	6304 (7)	9672 (8)	9.04 (28)
C(14)	10532 (15)	5785 (6)	9331 (6)	7.78 (21)
C(15)	5265 (11)	4099 (5)	4194 (5)	5.56 (14)
C(16)	4456 (11)	3988 (4)	2818 (5)	5.56 (14)
C(17)	2451 (17)	3513 (6)	2356 (8)	8.28 (25)
C(18)	1698 (21)	3422 (9)	1091 (9)	10.09 (33)
C(19)	3023 (23)	3801 (7)	316 (8)	9.42 (30)
C(20)	5075 (22)	4258 (8)	763 (7)	10.03 (32)
C(21)	5764 (18)	4359 (6)	2004 (6)	8.05 (20)
N(1)	3725 (8)	5453 (4)	4215 (4)	5.26 (13)
O(1)	2201 (6)	6573 (4)	3425 (4)	5.55 (10)
O(2)	−616 (6)	5703 (4)	4043 (4)	5.89 (11)
O(3)	3261 (10)	4274 (4)	6793 (4)	7.25 (13)
O(4)	6263 (8)	5154 (4)	6518 (3)	6.11 (11)
H(1)	5589 (98)	5677 (30)	4222 (42)	8.95 (90)

Experimental. Crystals were grown from an ethyl acetate/petroleum ether solution, melting point 342 K. Crystal size 0.23 × 0.34 × 0.87 mm, Siemens Kristaloflex 805 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, 20 reflections with $16 \leq \theta \leq 27°$ used for determining lattice parameters, data collected using ω–2θ scans up to $2\theta = 110°$. One standard reflection monitored after every 50 measurements showed no significant deviation from its mean intensity. 1283 unique reflections measured of which 1186 with $I > 2.0\sigma(I)$ were used in refinement. Range of hkl: $-5 \leq h \leq 5$, $0 \leq k \leq 18$, $0 \leq l \leq 11$. Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986) and refined by the full-matrix least-squares technique (*SHELX*76; Sheldrick, 1976). Final $R = 0.071$, $wR = 0.065$, $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F) + 0.04508F^2]^{-1}$, 240 parameters refined, $S = 0.44$, final accuracy of the refinement is limited by the poor quality of the crystals, $(\Delta/\sigma)_{\max}$ (for non-H atoms) = 0.63, max. and min. electron densities in final difference synthesis 0.18 and $-0{\cdot}35$ e Å^{-3}. All non-H atoms refined with anisotropic thermal parameters; H atoms included using riding model (C—H 1.08 Å) with fixed isotropic temperature factor $U = 0.137$ Å^2 for phenyl ring H and $U = 0.103$ Å^2 for the other H atoms, except that bonded to atom N(1), which was found from ΔF synthesis and refined isotropically. Atomic scattering factors as incorporated in *SHELX*76; for torsion angles and other geometrical calculations the programs *PARST* (Nardelli, 1983) and *XANADU* (Roberts & Sheldrick, 1975) were used. Atomic coordinates and

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Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

C(2)—C(1)	1.509 (8)	C(3)—C(1)	1.515 (8)
C(4)—C(1)	1.511 (8)	O(1)—C(1)	1.486 (6)
N(1)—C(5)	1.353 (6)	O(1)—C(5)	1.339 (6)
O(2)—C(5)	1.226 (6)	C(7)—C(6)	1.500 (7)
C(15)—C(6)	1.556 (7)	N(1)—C(6)	1.447 (6)
O(3)—C(7)	1.197 (6)	O(4)—C(7)	1.333 (6)
C(9)—C(8)	1.516 (8)	O(4)—C(8)	1.428 (6)
C(10)—C(9)	1.363 (9)	C(14)—C(9)	1.394 (8)
C(11)—C(10)	1.396 (11)	C(12)—C(11)	1.392 (12)
C(13)—C(12)	1.340 (13)	C(14)—C(13)	1.377 (11)
C(16)—C(15)	1.515 (7)	C(17)—C(16)	1.364 (9)
C(21)—C(16)	1.364 (9)	C(18)—C(17)	1.392 (11)
C(19)—C(18)	1.346 (14)	C(20)—C(19)	1.358 (13)
C(21)—C(20)	1.365 (9)	N(1)—H(1)	1.044 (48)
C(3)—C(1)—C(2)	110.6 (5)	C(4)—C(1)—C(2)	113.2 (4)
C(4)—C(1)—C(3)	111.0 (5)	O(1)—C(1)—C(2)	110.0 (4)
O(1)—C(1)—C(3)	102.0 (4)	O(1)—C(1)—C(4)	109.5 (5)
O(1)—C(5)—N(1)	110.8 (4)	O(2)—C(5)—N(1)	124.1 (4)
O(2)—C(5)—O(1)	125.0 (4)	C(15)—C(6)—C(7)	108.0 (4)
N(1)—C(6)—C(7)	113.5 (4)	N(1)—C(6)—C(15)	111.2 (4)
O(3)—C(7)—C(6)	123.6 (4)	O(4)—C(7)—C(6)	112.5 (4)
O(4)—C(7)—O(3)	123.9 (4)	O(4)—C(8)—C(9)	109.0 (5)
C(10)—C(9)—C(8)	124.4 (5)	C(14)—C(9)—C(8)	116.8 (5)
C(14)—C(9)—C(10)	118.7 (6)	C(11)—C(10)—C(9)	120.4 (6)
C(12)—C(11)—C(10)	119.6 (8)	C(13)—C(12)—C(11)	119.7 (7)
C(14)—C(13)—C(12)	121.0 (7)	C(13)—C(14)—C(9)	120.4 (7)
C(16)—C(15)—C(6)	112.7 (4)	C(17)—C(16)—C(15)	121.6 (5)
C(21)—C(16)—C(15)	120.2 (5)	C(21)—C(16)—C(17)	118.1 (6)
C(18)—C(17)—C(16)	121.3 (8)	C(19)—C(18)—C(17)	118.8 (9)
C(20)—C(19)—C(18)	120.5 (8)	C(21)—C(20)—C(19)	120.3 (8)
C(20)—C(21)—C(16)	120.8 (7)	C(6)—N(1)—C(5)	120.7 (4)
C(5)—O(1)—C(1)	120.6 (3)	C(8)—O(4)—C(7)	115.8 (4)
H(1)—N(1)—C(5)	121 (3)	H(1)—N(1)—C(6)	117 (3)
N(1)—C(5)—O(1)—C(1)	−174.2 (5)	N(1)—C(6)—C(15)—C(16)	−62.1 (7)
O(1)—C(5)—N(1)—C(6)	179.9 (5)	N(1)—C(6)—C(7)—O(4)	−37.3 (8)
O(2)—C(5)—N(1)—H(1)	−169.1 (33)	C(6)—C(7)—O(4)—C(8)	−176.7 (6)
C(15)—C(6)—N(1)—C(5)	138.0 (6)	C(9)—C(8)—O(4)—C(7)	−177.7 (6)
C(7)—C(6)—N(1)—C(5)	−100·0 (7)		

D—H···*A*	*D*—H	*D*···*A*	H···*A*	*D*—H···*A*
N(1)—H(1)···O(2')	1.04 (5)	3.01 (1)	2.02 (5)	159 (4)

Symmetry code: (i) $x + 1, y, z$.

equivalent isotropic temperature factors are listed in Table 1;* selected bond distances, angles and torsion angles are given in Table 2. Atom numbering follows that shown in Fig. 1.

Related literature. For the preparation and properties of the title compound and related derivatives see Nagasawa, Kuroiwa, Narita & Isowa (1973); Matsoukas, Moharir, & Findlay (1983); Findlay, Dalzeet, Matsoukas & Moharir (1984); Matsoukas, Semertzidis, Hondrelis, Nastopoulos, Voliotis & Leban (1989), Semertzidis, Matsoukas, Nastopoulos, Hondrelis, Voliotis & Leban (1989); Matsoukas, Goghari, Scanlon, Franklin & Moore (1985); Matsoukas, Bigam, Zhou & Moore (1990). Knowledge of the conformation of the title compound and related phenylalanine derivatives is important for the design and synthesis of effective inhibitors for diagnostic and therapeutic applications.

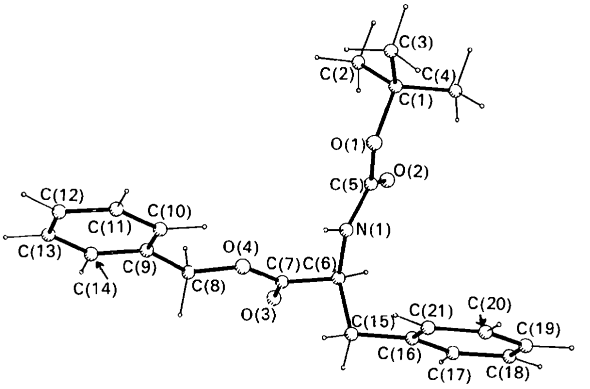

Fig. 1. Perspective view (*PLUTO*; Motherwell & Clegg, 1978) of the molecule with the atom-numbering scheme.

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*Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54406 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0261]