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Structure of *N*-(*tert*-Butoxycarbonyl)glycine

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Abstract. $C_7H_{13}NO_4$, $M_r = 175{\cdot}1$, triclinic, $P\bar{1}$, $a = 7{\cdot}268$ (2), $b = 11{\cdot}281$ (2), $c = 11{\cdot}967$ (2) Å, $\alpha = 104{\cdot}96$ (1), $\beta = 91{\cdot}94$ (1), $\gamma = 93{\cdot}61$ (2)°, $V = 944{\cdot}8$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}231$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 1{\cdot}08$ cm^{-1}, $F(000) = 376$, $T = 295$ K, $R = 0{\cdot}0442$ for 2333 unique reflections. The asymmetric unit contains two crystallographically independent molecules. The structure is stabilized along **b** by a hydrogen-bond contact between the carboxyl OH and the ester C=O [O(12*A*)···O(7*B*) = 2·650 (5), O(12*B*)···O(7*A*) = 2·652 (5) Å]. The geometry of the two independent molecules is identical, except for small differences of bond lengths and angles in the *tert*-butyl groups, which have a high thermal motion. The urethane amide bond adopts the usual *trans* conformation with O(5)—C(6)—N(8)—C(9) torsion angles of −173·9 (5) and −177·5 (5)° for the two molecules.

Experimental. Transparent prismatic crystal from ethyl acetate/petroleum ether, m.p. 360–361 K, dimensions 0·45 × 0·48 × 0·37 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice parameters determined from 25 reflections ($9 < \theta < 11°$), $\omega/2\theta$ scan technique up to $2\theta = 60°$, scan width $(1{\cdot}0 + 0{\cdot}3\tan\theta)°$, scan rate 1·50–5·49° min^{-1}, background 1/4 of the scan time at each scan limit, max. scan time 60 s, aperture $(2{\cdot}4 + 0{\cdot}9\tan\theta)$ mm. 11227 measured reflections, $-9 < h < 9$, $-15 < k < 15$, $-15 < l < 15$, 5485 averaged, mean discrepancy on $I = 1{\cdot}2\%$ for 10692 reflections, 2333 observed with $I \geq 2{\cdot}0\sigma(I)$. Reference reflections ($\bar{4}00$, $\bar{3}05$, $\bar{3}\bar{3}0$, after 2 h), orientation control reflections ($\bar{1}51$, $\bar{1}43$, $0\bar{1}6$ after 300 reflections), no significant variation during data collection. Structure solved by direct methods and refined by full-matrix least-squares techniques. All non-H atoms refined with anisotropic thermal parameters. H atoms constrained to idealized positions, riding model (C—H = 1·08 Å), except H(8*A*), H(12*A*), H(8*B*), H(12*B*)

Table 1. *Atomic parameters for molecules A and B with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j a_i a_j \mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}(Å^2)
C(1*A*)	7292 (4)	5531 (2)	13312 (2)	4·86 (8)
C(2*A*)	6520 (5)	4350 (3)	13558 (3)	6·23 (10)
C(3*A*)	6317 (5)	6617 (3)	13981 (3)	6·75 (10)
C(4*A*)	9371 (4)	5733 (3)	13545 (3)	7·00 (11)
O(5*A*)	6849 (2)	5531 (1)	12096 (1)	4·76 (5)
C(6*A*)	7491 (3)	4709 (2)	11226 (2)	3·93 (7)
O(7*A*)	8207 (2)	3781 (2)	11300 (2)	5·37 (5)
N(8*A*)	7236 (3)	5020 (2)	10224 (2)	3·84 (6)
C(9*A*)	7667 (3)	4194 (2)	9149 (2)	3·99 (7)
C(10*A*)	6501 (3)	2992 (2)	8866 (2)	3·83 (7)
O(11*A*)	5130 (2)	2802 (1)	9344 (2)	4·83 (5)
O(12*A*)	7188 (3)	2171 (2)	8027 (2)	5·66 (6)
C(1*B*)	18186 (4)	674 (2)	13324 (2)	4·96 (8)
C(2*B*)	20034 (4)	1426 (3)	13539 (3)	7·35 (12)
C(3*B*)	17173 (5)	867 (4)	14416 (3)	9·42 (16)
C(4*B*)	18480 (5)	−648 (3)	12759 (3)	7·97 (12)
O(5*B*)	17219 (2)	1199 (1)	12484 (2)	4·87 (6)
C(6*B*)	15500 (3)	805 (2)	12068 (2)	3·99 (7)
O(7*B*)	14539 (3)	50 (2)	12382 (2)	5·56 (6)
N(8*B*)	15000 (3)	1360 (2)	11262 (2)	4·70 (7)
C(9*B*)	13188 (3)	1143 (2)	10701 (2)	4·64 (7)
C(10*B*)	12066 (3)	2245 (2)	11044 (2)	4·38 (8)
O(11*B*)	12576 (3)	3199 (2)	11706 (2)	6·34 (7)
O(12*B*)	10424 (3)	2011 (2)	10493 (2)	6·15 (7)
H(8*A*)	6619 (45)	5718 (30)	10240 (28)	8·95 (18)
H(12*A*)	6577 (46)	1478 (32)	7996 (29)	8·95 (18)
H(8*B*)	15822 (47)	1938 (31)	11127 (29)	8·95 (18)
H(12*B*)	9767 (47)	2733 (30)	10860 (29)	8·95 (18)

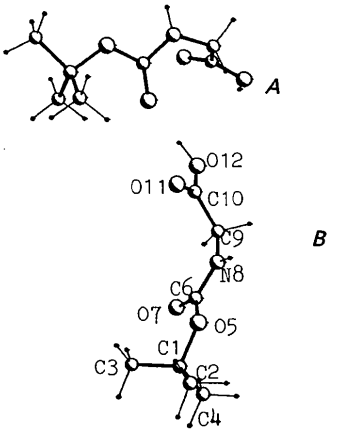

Fig. 1. A perspective view of the two molecules in the asymmetric unit.

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Table 2. *Selected geometrical parameters* (Å,°) *with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*
(*a*) Bond lengths		
C(2)—C(1)	1·515 (4)	1·520 (4)
C(3)—C(1)	1·512 (4)	1·495 (4)
C(4)—C(1)	1·519 (4)	1·502 (4)
O(5)—C(1)	1·480 (3)	1·472 (3)
C(6)—O(5)	1·322 (3)	1·334 (3)
O(7)—C(6)	1·220 (3)	1·210 (3)
N(8)—C(6)	1·343 (3)	1·330 (3)
C(9)—N(8)	1·438 (3)	1·436 (3)
C(10)—C(9)	1·507 (3)	1·505 (3)
O(11)—C(10)	1·201 (3)	1·188 (3)
O(12)—C(10)	1·314 (3)	1·321 (3)
(*b*) Valence angles		
C(3)—C(1)—C(2)	110·7 (2)	110·7 (3)
C(4)—C(1)—C(2)	112·1 (2)	109·5 (2)
C(4)—C(1)—C(3)	111·6 (2)	114·3 (3)
O(5)—C(1)—C(2)	110·9 (2)	102·4 (2)
O(5)—C(1)—C(3)	102·3 (2)	110·4 (2)
O(5)—C(1)—C(4)	108·8 (2)	109·0 (2)
C(6)—O(5)—C(1)	121·0 (2)	122·3 (2)
O(7)—C(6)—O(5)	125·6 (2)	124·5 (2)
N(8)—C(6)—O(5)	111·0 (2)	110·4 (2)
N(8)—C(6)—O(7)	123·5 (2)	125·1 (2)
C(9)—N(8)—C(6)	120·3 (2)	122·2 (2)
C(10)—C(9)—N(8)	113·0 (2)	112·4 (2)
O(11)—C(10)—C(9)	124·7 (2)	125·4 (2)
O(12)—C(10)—C(9)	111·0 (2)	110·4 (2)
O(12)—C(10)—O(11)	124·2 (2)	124·2 (2)
O(5)—C(6)—N(8)—C(9)	−173·9 (5)	−177·5 (5)

(*c*) Hydrogen-bond geometry

D—H···*A*	*D*···H	*D*···*A*	H···*A*	*D*—H···*A*
O(12*A*)—H(12*A*)···O(7*B*)	0·87 (4)	2·650 (5)	1·80 (3)	168 (3)
O(12*B*)—H(12*B*)···O(7*A*)	0·98 (3)	2·652 (5)	1·69 (3)	166 (3)

Symmetry code: (i) $-x+2, -y, -z+2$.

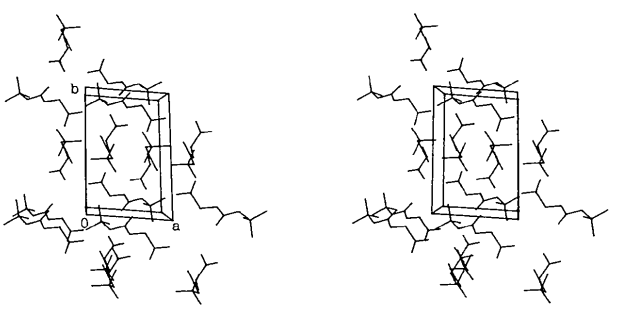

Fig. 2. A stereoview of the unit-cell packing along the *c* axis.

which were located by a difference Fourier synthesis and refined isotropically. Intensities corrected for Lp; absorption corrections and secondary-extinction corrections not applied; atomic scattering factors from *SHELX*76. $R = 0.0442$ for 2333 unique reflections, $wR = 0.0491$, $\sum w(\Delta F)^2$ minimized, $w = 0.7093/[\sigma^2(F) + 0.001097F^2]$, max. $\Delta/\sigma < 0.04$ (for non-H atoms), max. and min. electron densities in final difference map 0·19 and -0.17 e Å^{-3}. Computer programs used: *MULTAN*11/84 (Main, Germain & Woolfson, 1984), *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1986) and *PLUTO* (Motherwell & Clegg, 1978). The refined atomic coordinates and equivalent isotropic temperature factors are given in Table 1;* selected bond distances and bond angles are presented in Table 2; the molecular structure and the numbering of the atoms are shown in Figs. 1 and 2.

Related literature. For the preparation and properties of the title compound, see Nagasawa, Kuroiwa, Narita & Isowa (1973). This compound is an intermediate in the synthesis of oxytocin and enkephalin analogues.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51856 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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