

## Structure of Phenylalaninol

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**Abstract.**  $C_9H_{13}NO$ ,  $M_r = 151.21$ , orthorhombic,  $P2_12_12_1$ ,  $a = 4.959(1)$ ,  $b = 8.144(1)$ ,  $c = 21.257(2)$  Å,  $V = 858.5(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.170$  g cm<sup>-3</sup>,  $Cu K\alpha$  ( $\lambda = 1.5418$  Å),  $\mu = 5.71$  cm<sup>-1</sup>,  $F(000) = 328$ ,  $T = 295$  K,  $R(F) = 0.053$  for 762 unique reflections with  $I \geq 2.5\sigma(I)$ . The phenyl ring is planar and the molecules in the unit cell are stabilized by N–H...O-type hydrogen bonds. The phenylalanine group has a  $g^-$  conformation. Relevant torsion angles are:  $\psi(O1-C2-C3-N4) = 57.1(4)$ ,  $\chi_1(N4-C3-C5-C6) = -67.4(4)$ ,  $\chi_2(C3-C5-C6-C7) = 94.1(4)^\circ$ .

**Experimental.** Thin transparent colourless needles,  $0.4 \times 0.3 \times 0.2$  mm, from water, three-dimensional intensity data on Enraf–Nonius CAD-4 automatic diffractometer,  $\omega/2\theta$  scan mode with  $\Delta\omega = (0.8 + 0.14 \tan\theta)^\circ$ ; aperture width =  $(4.0 + 2.0 \tan\theta)$  mm;  $4 \leq 2\theta \leq 120^\circ$ ,  $0 \leq h \leq 5$ ,  $0 \leq k \leq 9$ ,  $0 \leq l \leq 25$ , 826 unique reflections and 762 observed with  $I \geq 2.5\sigma(I)$ ; cell constants from 16 reflections with  $22 \leq 2\theta \leq 102^\circ$ ; three standard reflections monitored every 100 reflections showed no significant variation in intensity. The maximum time spent on any reflection measurement was 30 s and the background count was half the scan time. The intensity data were corrected for direct-beam polarization, Lorentz and absorption effects (the average transmission factor was 0.932, with a maximum and minimum of 0.860 and 0.994, respectively). Structure solution by direct methods, H atoms from  $\Delta\rho$  maps, the proton attached to the oxygen could not be located. Full-matrix least-squares refinement on  $|F_o|$ , with non-H atoms anisotropic and H atoms isotropic, converged to a final  $R(|F_o|) = 0.052$ ,  $wR = 0.068$  with individual weighting scheme based on counting statistics where  $w = 4|F_o|^2/[\sigma^2(|F_o|^2)]$  and  $\sigma^2(F_o) = [\sigma^2(I) + 0.0004I^2]^{1/2}/Lp$ ;  $(\Delta/\sigma)_{\max} = 0.3$ ,  $(\Delta/\sigma)_{\text{mean}} = 0.07$ ,  $S = 6.28$  for 148 parameters, final  $\Delta\rho$  map had no peaks  $\geq 0.32$  e Å<sup>-3</sup>. All calculations on VAX 11/730 computing system using *SDP* package (Frenz, 1978). The fractional positional parameters with equivalent isotropic temperature factors for the non-H atoms

are given in Table 1.‡ A stereoview of the molecule is shown in Fig. 1, and the packing of the molecules in the unit cell in Fig. 2. The bond lengths and angles involving non-H atoms and the torsion angles are given in Table 2.

‡ Lists of structure factors, positional parameters of H atoms, bond lengths and angles involving H atoms and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51642 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters for non-H atoms with estimated standard deviations

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
O1	0.3536 (6)	0.4335 (3)	0.7263 (1)	4.38 (5)
C2	0.4622 (8)	0.5449 (5)	0.6813 (2)	3.90 (8)
C3	0.2650 (7)	0.6832 (4)	0.6689 (2)	3.14 (7)
N4	0.2043 (7)	0.7642 (3)	0.7283 (1)	3.69 (6)
C5	0.3901 (9)	0.8054 (5)	0.6221 (2)	3.83 (8)
C6	0.1975 (8)	0.9373 (4)	0.6004 (2)	3.42 (7)
C7	0.1874 (9)	1.0893 (4)	0.6306 (2)	4.13 (8)
C8	0.009 (1)	1.2086 (5)	0.6111 (2)	4.72 (9)
C9	-0.1601 (9)	1.1818 (5)	0.5609 (2)	4.73 (9)
C10	-0.153 (1)	1.0310 (5)	0.5302 (2)	4.68 (9)
C11	0.0263 (9)	0.9110 (4)	0.5500 (2)	4.08 (8)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as:

$$B_{\text{eq}} = \frac{1}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2).$$

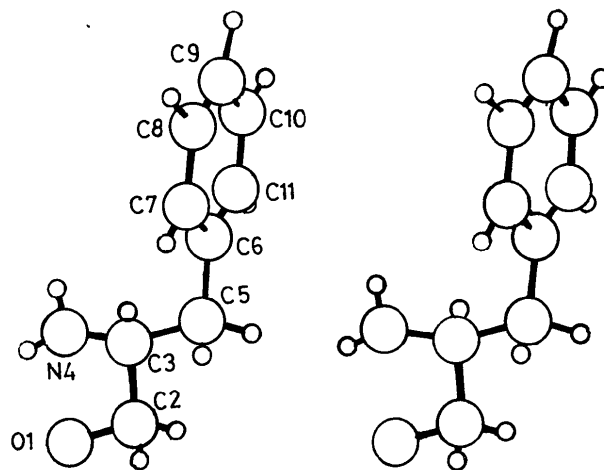


Fig. 1. Stereoview of the molecule with atom numbering.

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† DCB contribution No. 723.

Table 2. Bond distances (Å), bond angles (°) and torsion angles (°) involving non-H atoms, with e.s.d.'s in parentheses

O1—C2	1.424 (5)	C5—C6	1.510 (5)	C8—C9	1.373 (6)
C2—C3	1.515 (5)	C6—C7	1.394 (5)	C9—C10	1.391 (6)
C3—N4	1.457 (4)	C6—C11	1.384 (5)	C10—C11	1.387 (6)
C3—C5	1.537 (5)	C7—C8	1.379 (6)		

O1—C2—C3	110.3 (3)	C7—C6—C11	118.1 (3)
C2—C3—N4	108.6 (3)	C6—C7—C8	120.7 (4)
C2—C3—C5	109.5 (3)	C7—C8—C9	120.9 (4)
N4—C3—C5	110.6 (3)	C8—C9—C10	119.3 (4)
C3—C5—C6	113.8 (3)	C9—C10—C11	119.7 (4)
C5—C6—C7	121.0 (3)	C6—C11—C10	121.3 (3)
C5—C6—C11	120.9 (3)		

$\psi$ O1—C2—C3—N4	57.11 (0.37)
O1—C2—C3—C5	177.91 (0.27)
C2—C3—C5—C6	173.03 (0.29)
$\chi_1$ N4—C3—C5—C6	-67.36 (0.38)
$\chi_2$ C3—C5—C6—C7	94.06 (0.42)
C3—C5—C6—C11	-86.02 (0.42)
C5—C6—C7—C8	-179.14 (0.38)
C11—C6—C7—C8	0.93 (0.60)
C5—C6—C11—C10	179.42 (0.38)
C7—C6—C11—C10	-0.65 (0.60)
C6—C7—C8—C9	-1.19 (0.67)
C7—C8—C9—C10	1.12 (0.68)
C8—C9—C10—C11	-0.83 (0.66)
C9—C10—C11—C6	0.61 (0.64)

Table 3. Comparison of torsion angles (°) (defined in Table 2)

	$\psi$	$\chi_1$	$\chi_2$	Reference
Phenylalaninol	57.1 (4)	-67.4 (4)	94.1 (4)	(a)
Boc-glycyl-L-phenylalanine	170.3	52.0	85.8	(b)
L-Tyrosyl-L-phenylalanine	146.6	-75.1	75.9	(c)
L-Phenylalanine-L-proline H <sub>2</sub> O	-42.5 (7)	-170.6 (5)	81.6 (7)	(d)

References: (a) Present study; (b) Murali & Subramanian (1986); (c) Murali & Subramanian (1987); (d) Panneerselvam & Chacko (1989).

**Related literature.** In the unit cell the molecules are stabilized by N—H...O-type intermolecular hydrogen bonds with N...O distance of 2.764 (2) Å with sym-

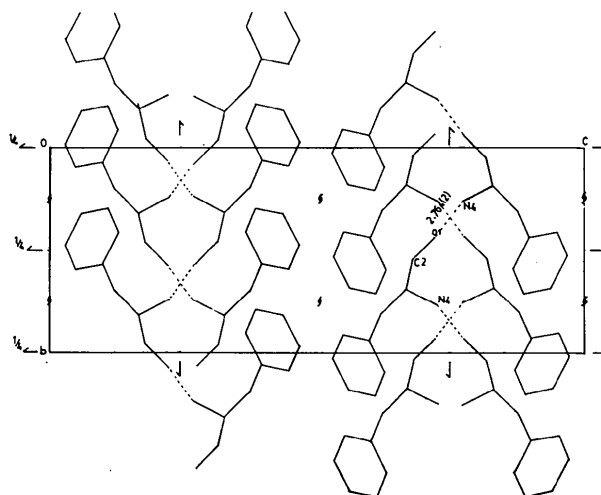


Fig. 2. Packing of the molecules in the unit cell projected down the *a* axis.

metry  $1-x, y-\frac{1}{2}, \frac{3}{2}-z$ . The backbone and side-chain torsion angles are comparable with the values observed in related structures containing a phenyl group (Murali & Subramanian, 1986, 1987; Panneerselvam & Chacko, 1989) (Table 3).

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## 1:2 Adduct of 1-Ethoxy-2-methylisoindole with *N*-Methylmaleimide

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**Abstract.** *endo*-1-Ethoxy-*N*-methyl-1,4-(*N*-methylimino)-4-(*N*-methylsuccinimido)-1,2,3,4-tetrahydro-naphthalene-2,3-dicarboximide hemimethanol solvate,

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$C_{21}H_{23}N_3O_5 \cdot \frac{1}{2}CH_3OH$ ,  $M_r = 413.46$ , triclinic,  $P\bar{1}$ ,  $a = 8.623$  (4),  $b = 9.876$  (6),  $c = 14.235$  (10) Å,  $\alpha = 108.96$  (5),  $\beta = 97.40$  (5),  $\gamma = 102.28$  (4)°,  $V =$

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