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4-Hydroxy-4-phenylhexanamide, an Anticonvulsant Molecule

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Abstract. $C_{12}H_{17}NO_2$, orthorhombic, *Pcca*, $a = 23 \cdot 025$ (4), $b = 10 \cdot 366$ (2), $c = 10 \cdot 069$ (4) Å, $V = 2403 \cdot 6$ Å^3, $Z = 8$, $D_m = 1 \cdot 14$ (by flotation), $D_c = 1 \cdot 15$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71073$ Å, $\mu(\text{Mo } K\alpha) = 8 \cdot 39$ mm^{-1}. The structure was solved by application of direct methods, and refined by full-matrix least squares to a final R of $0 \cdot 082$ for 707 reflections with $I \geq 2\sigma(I)$. The bond distances in the benzene ring are rather short owing to libration. There is an intermolecular hydrogen bond $[O(1)\cdots O(2) = 2 \cdot 768$ (6) Å].

Introduction. The title compound [also known as γ-hydroxy-γ-ethyl-γ-phenylbutyramide (HEPB)] has long been known for its anticonvulsant activity. It was previously named EPP because it was believed to be 5-ethyl-5-phenyl-2-pyrrolidinone when it was designed and synthesized to penetrate the blood–brain barrier to inhibit γ-aminobutyric acid–α-oxoglutaric acid transaminase (GABA-T). In spite of the many papers

published about its biological activity, its structure has only recently been revised by proton and carbon magnetic resonance (Joseph-Nathan, Massieu, Carvajal & Tapia, 1978) to establish its structural formula as HEPB.

The geometrical configuration of this substance may be an aid to the understanding of its mechanism of action at a molecular level. For this reason and to confirm unambiguously its structural formula a crystal structure determination was undertaken.

Crystals of adequate size for X-ray analysis were obtained, after many different trials, from H_2O–EtOH by slow evaporation at room temperature. A crystal of irregular shape with maximum and minimum linear dimensions of 0·6 and 0·3 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. 17 centred reflections and least-squares refinement produced the unit-cell dimensions and the orientation matrix for data collection. The θ–2θ scan technique at a rate of

1·4–6·7° min^{-1}, determined by fast prescan of 6·7° min^{-1}, was used. 1731 reflections were collected in the range $0° < \theta < 22°$ using graphite-monochromated Mo $K\alpha$ radiation; of these, 707 have $I \geq 2\sigma(I)$. The intensity of a standard reflection was essentially constant over the period of the experiment.

Intensities were corrected for Lorentz and polarization effects but not for absorption or extinction. The atomic scattering factors used were those of Cromer & Waber (1974) and anomalous-dispersion correction coefficients were those of Cromer & Ibers (1974).

The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1978), adapted to a PDP-45 computer with 48K 16-bit words, using 160 reflections with $E > 1·62$. All the non-hydrogen atoms, except four of the benzene ring, appeared clearly in the E map calculated from the solution with the highest figure of merit. A subsequent difference synthesis showed the rest of the molecule. The structure was refined isotropically to an R value of 0·13 and then anisotropically to $R = 0·10$. At this stage all H atoms were placed on stereochemical grounds and assigned an isotropic temperature factor of 4·0 Å^2. Further anisotropic refinement gave a final $R = 0·082$ and $R_w = 0·078$. Analysis of the minimization function in terms of F_{obs} intervals showed unit weights to be a convenient choice. Atomic coordinates together with the equivalent isotropic temperature factors (Hamilton, 1959) are listed in Table 1.* A final difference map was essentially featureless.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35624 (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 and isotropic thermal parameters*

|	x	y	z	B_{Iso} (Å^2)
O(1)	0·4023 (2)	0·1483 (6)	0·1549 (5)	4·2
O(2)	0·4337 (2)	0·1083 (5)	0·6477 (5)	3·7
N(1)	0·5236 (3)	0·1597 (7)	0·5809 (6)	4·1
C(1)	0·2760 (4)	0·1805 (10)	0·1011 (10)	6·2
C(2)	0·3034 (3)	0·1385 (8)	0·2301 (9)	4·1
C(3)	0·3628 (3)	0·1918 (7)	0·2561 (8)	3·3
C(4)	0·3864 (3)	0·1409 (8)	0·3893 (7)	3·2
C(5)	0·4474 (3)	0·1808 (8)	0·4218 (8)	3·6
C(6)	0·4668 (3)	0·1448 (8)	0·5597 (8)	3·1
C(7)	0·3644 (3)	0·3409 (7)	0·2556 (8)	3·4
C(8)	0·3311 (5)	0·4086 (10)	0·3383 (14)	9·3
C(9)	0·3308 (6)	0·5382 (11)	0·3424 (16)	11·1
C(10)	0·3641 (5)	0·6079 (9)	0·2648 (14)	8·8
C(11)	0·4019 (6)	0·5438 (10)	0·1854 (14)	11·2
C(12)	0·4005 (6)	0·4112 (10)	0·1802 (11)	8·8
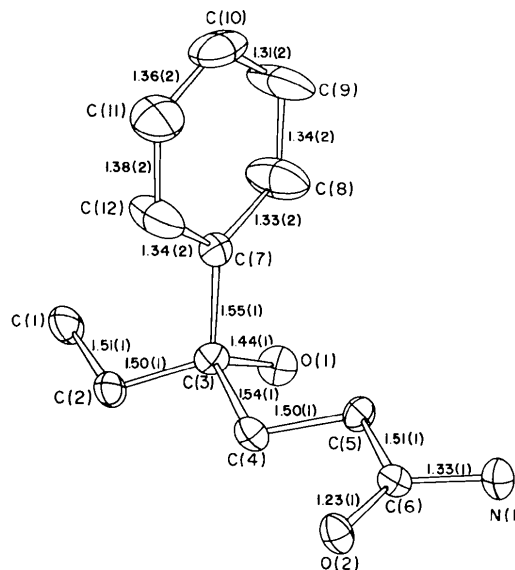
Fig. 1. The title compound, showing the bond distances (Å) and numbering scheme.

Table 2. *Interatomic angles (°) with e.s.d.'s in parentheses*

C(1)–C(2)–C(3)	115·2 (9)	C(5)–C(6)–N(1)	114·2 (9)
C(2)–C(3)–C(4)	110·3 (8)	O(2)–C(6)–N(1)	122·0 (9)
C(2)–C(3)–C(7)	112·9 (8)	C(3)–C(7)–C(8)	120·8 (11)
C(2)–C(3)–O(1)	109·7 (8)	C(3)–C(7)–C(12)	124·0 (11)
C(4)–C(3)–C(7)	109·7 (8)	C(8)–C(7)–C(12)	115·1 (12)
C(4)–C(3)–O(1)	106·7 (7)	C(7)–C(8)–C(9)	123·3 (14)
C(7)–C(3)–O(1)	107·3 (8)	C(8)–C(9)–C(10)	122·0 (17)
C(3)–C(4)–C(5)	115·1 (7)	C(9)–C(10)–C(11)	117·1 (15)
C(4)–C(5)–C(6)	114·3 (8)	C(10)–C(11)–C(12)	119·9 (16)
C(5)–C(6)–O(2)	123·9 (9)	C(7)–C(12)–C(11)	122·4 (14)

Discussion. The molecule is depicted in Fig. 1, which also shows bond distances. Angles between bonded atoms are given in Table 2. The benzene ring is planar to within experimental accuracy with a mean deviation of the atoms from the best least-squares plane through them of 0·02 (1) Å.

All distances are within the expected range, except for those of the benzene ring which are rather short. This anomaly is not a consequence of a biased least-squares refinement: reflections $>2\sigma(I)$ were used giving a ratio of 5·2 reflections per independent parameter and the minimization function showed no trends when displayed as a function of F_{obs} intervals. Fig. 1, in which the thermal ellipsoids are drawn to a 45% probability scale, shows rather high thermal motion for C(8) to C(12); in fact, the U_{11}, U_{33} and U_{13} elements of the thermal tensors of these atoms are consistently larger than all the others. This may be interpreted as a libration of the benzene ring (which forms a dihedral angle of nearly 45° with either the *ab* or *bc* planes) about the C(3)–C(7) bond.
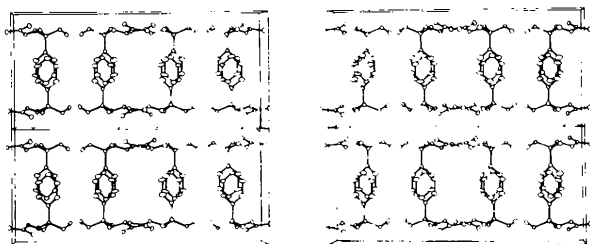
Fig. 2. A stereoview of the extended structure.

of 3·66 Å. In a direction normal to the benzene-ring plane, there is no overlap between neighbouring rings which indicates that an eventual $\pi-\pi$ intermolecular interaction is not favoured; the packing seems to be dominated by the steric requirements of the hydrogen-bonding scheme.

This mode of motion causes the distance between the mean atomic positions to be less than the corresponding mean interatomic separation, which is the quantity of chemical significance (Johnson, 1970). The rather high temperature factors of many atoms of the molecule are probably the reason for the low percentage of observed reflections collected.

A stereopicture of the extended structure is shown in Fig. 2. The benzene rings of all molecules in the unit cell are pointing inwards along the **b** direction while the carbon chains are located along the **a** direction near the edge of the cell. This arrangement favours an intermolecular hydrogen-bond interaction between O(1) acting as donor and O(2) as acceptor. The relevant interatomic distance is 2·768 (6) Å. The benzene rings of neighbouring molecules are almost parallel with a dihedral angle of 2·8° and a mean interplanar distance

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The Structure of Bis(4-hydroxybutyl) 4,4'-Methylenebis(phenylcarbamate): a Model Compound for Diol-Linked MDI Units in Polyurethane Elastomers

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Abstract. $C_{23}H_{30}N_2O_6$, orthorhombic, $P2_12_12$, $a = 16·511$ (1), $b = 13·970$ (1), $c = 4·778$ (1) Å, $Z = 2$, $D_m = 1·29$, $D_c = 1·296$ Mg m^{-3}. The structure was refined to $R = 0·075$ ($R_w = 0·068$), based on diffractometer data collected at room temperature. The two halves of the molecule are related by a twofold axis through the central CH_2. The central $C-CH_2-C$ bridge angle is 105·2 (1·0)°, and the planes of the phenyl rings are mutually inclined at 98·2°. The urethane groups are planar and are inclined at 13·6° to their adjacent phenyl rings. The $(CH_2)_4$ chains are also planar (all *trans*), and are approximately *gauche* to the adjacent urethane. The terminal OH groups form hydrogen bonds to the C=O and N—H groups of different neighboring molecules, leading to a chain of hydrogen bonds along the b axis. The title compound serves as a model for the diol-linked 4,4'-methylenediphenyl diisocyanate (MDI) units which form the hard segments in certain polyurethane elastomers.

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