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Structure of 4-Hydroxy-3-phenylbutanamide Monohydrate, $C_{10}H_{13}NO_2.H_2O$*

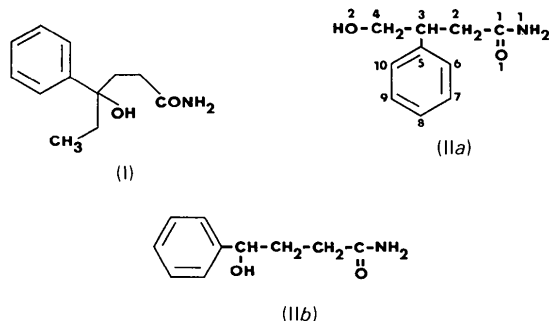BY M. SORIANO-GARCÍA,† R. A. TOSCANO, T. ALCOCER, R. CRUZ AND F. GARCÍA

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Abstract. $M_r = 197.2$, monoclinic, $C2/c$, $a = 16.546 (5)$, $b = 6.445 (2)$, $c = 19.936 (5)$ Å, $\beta = 100.77 (2)°$, $V = 2089 (1)$ Å^3, $Z = 8$, $D_x = 1.25$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.726$ mm^{-1}, $F(000) = 848$, $T = 293$ K. Final $R = 0.039$ for 1313 observed reflections. The X-ray study confirms that in the solid state the structure of the title compound is similar to that inferred from chemical and spectroscopic evidence. Steric hindrance from different chemical groups is minimized by the adoption of a staggered configuration at the central C(2)—C(3) bond. The crystal structure is stabilized by a three-dimensional network of O—H···O and N—H···O hydrogen bonds.

Introduction. Crystal structure studies on a series of derivatives of the anticonvulsant molecule γ-hydroxy-γ-phenylcaproamide (I) (4-hydroxy-4-phenyl-hexanamide) (Carvajal, Russek, Tapia & Massieu, 1964; Joseph-Nathan, Massieu, Carvajal & Tapia, 1978) have been undertaken in our laboratory to investigate the influence of different substituents on its pharmacological activity.

4-Hydroxy-3-phenylbutanamide (IIa) and 4-hydroxy-4-phenylbutanamide (IIb) were obtained by condensation of styrene oxide with malonic ester in the presence of sodium ethoxide, followed by successive hydrolysis and decarboxylation of the resulting product and preparation of the amide derivative (Bavin, Hansell & Spickett, 1964).

X-ray analysis of (IIa) was undertaken to confirm the proposed structure and to obtain details of its molecular conformation.

Experimental. Cube-shaped crystal 0.32 × 0.37 × 0.34 mm, Nicolet R3 four-circle diffractometer, graphite-monochromated Cu $K\alpha$, lattice parameters from 15 machine-centered reflections with $10.9 < 2\theta < 24.6°$; 1610 reflections with $3 < 2\theta < 115°$ for two octants, 1313 independent with $I > 2.5\sigma(I)$, index range $h \pm 17$, k 0/6, l 0/21, ω-scan mode, variable scan speed, scan width $1.0°(\theta)$, two standard reflections monitored every 50 measurements, Lp correction, absorption ignored; structure solved by direct methods using *SHELXTL* (Sheldrick, 1981); least-squares refinement of all non-H atoms treated anisotropically, H atoms riding on the bonded C, coordinates of H atoms bonded to N and O refined with fixed isotropic temperature factor $U = 0.06$ Å^2, function minimized $\sum w(\Delta F)^2$, $w = |\sigma^2(F_o) + 0.001 \times (F_o)^2|^{-1}$, $(\Delta/\sigma)_{max.} = 0.5$; residual electron

density within $\pm 0\cdot 2\,e\,\text{Å}^{-3}$, isotropic extinction parameter $X = 0\cdot 00606$; final $R = 0\cdot 039$, $wR = 0\cdot 070$; scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Fig. 1 shows the molecular conformation of one of the enantiomers of (II*a*). Final atomic coordinates are in Table 1 and bond distances and angles in Table 2. Calculations of the least-squares planes through the phenyl ring, the butyl and the amide groups, the deviations of individual atoms from the planes and interplanar angles have been deposited.*

The phenyl ring shows normal geometry, $1\cdot 380$ Å and $120\cdot 0°$ for the mean $C(sp^2)–C(sp^2)$ bond distance and the mean internal angle. This mean bond distance is close to that given by Sutton (1965) $[1\cdot 395\ (5)$ Å] and Brisse & Sygusch (1974) $[1\cdot 379$ Å].

The phenyl ring and amide group are planar within the limit of experimental error and are perpendicular to each other [dihedral angle $91\cdot 7\ (4)°$].

The Newman projection along the C(3)–C(5) bond (Fig. 2) shows that the projected angle between the amide and hydroxyl groups at C(3) is $123\cdot 0\ (4)°$, which helps to reduce the *gauche* interaction between these two groups. Non-bonded interactions between amide and hydroxyl groups, and the phenyl ring are minimized across the C(2)–C(3) bond by the staggered conformation adopted.

Details of the hydrogen-bonding scheme have been deposited.* The amino group takes part in two hydrogen bonds with the O atoms of two symmetry-related water molecules as the acceptors. The O(2) hydroxyl group interacts with the O(1) carbonyl group of an adjacent molecule *via* a diagonal translation in the *ac* plane. The water molecule is also hydrogen bonded to carbonyl and hydroxyl O atoms. The packing of the molecules in the crystal structure is illustrated in Fig. 3.

* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and the results of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39678 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
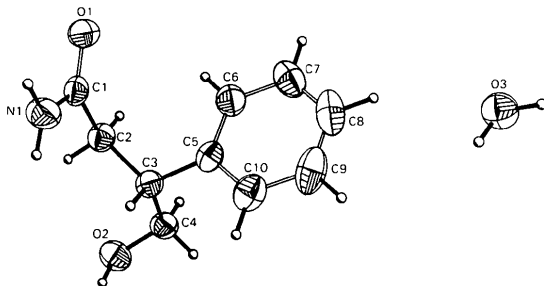
Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic temperature factors* $(\text{Å}^2 \times 10^3)$

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
N(1)	1722 (1)	940 (3)	425 (1)	50 (1)
O(1)	1040 (1)	−1828 (2)	698 (1)	47 (1)
O(2)	−217 (1)	5676 (2)	985 (1)	51 (1)
O(3)	3318 (1)	242 (2)	5070 (1)	53 (1)
C(1)	1104 (1)	81 (3)	653 (1)	37 (1)
C(2)	473 (1)	1524 (3)	857 (1)	40 (1)
C(3)	819 (1)	3018 (2)	1442 (1)	37 (1)
C(4)	128 (1)	4488 (3)	1567 (1)	43 (1)
C(5)	1193 (1)	2005 (3)	2114 (1)	37 (1)
C(6)	963 (1)	52 (3)	2308 (1)	48 (1)
C(7)	1266 (1)	−731 (4)	2954 (1)	60 (1)
C(8)	1798 (1)	396 (4)	3413 (1)	67 (1)
C(9)	2052 (1)	2323 (4)	3226 (1)	68 (1)
C(10)	1749 (1)	3113 (3)	2583 (1)	52 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(1)	1·315 (2)	O(1)–C(1)	1·240 (2)
O(2)–C(4)	1·417 (2)	C(1)–C(2)	1·510 (2)
C(2)–C(3)	1·538 (2)	C(3)–C(4)	1·540 (2)
C(3)–C(5)	1·515 (2)	C(5)–C(6)	1·390 (2)
C(5)–C(10)	1·381 (2)	C(6)–C(7)	1·387 (3)
C(7)–C(8)	1·356 (3)	C(8)–C(9)	1·384 (4)
C(9)–C(10)	1·384 (3)		
N(1)–C(1)–O(1)	121·8 (2)	N(1)–C(1)–C(2)	117·1 (2)
O(1)–C(1)–C(2)	121·2 (2)	C(1)–C(2)–C(3)	114·3 (1)
C(2)–C(3)–C(4)	109·0 (1)	C(2)–C(3)–C(5)	115·7 (1)
C(4)–C(3)–C(5)	108·4 (1)	O(2)–C(4)–C(3)	113·4 (1)
C(3)–C(5)–C(6)	123·2 (1)	C(3)–C(5)–C(10)	119·1 (2)
C(6)–C(5)–C(10)	117·5 (1)	C(5)–C(6)–C(7)	121·2 (2)
C(6)–C(7)–C(8)	120·5 (2)	C(7)–C(8)–C(9)	119·4 (2)
C(8)–C(9)–C(10)	120·2 (2)	C(5)–C(10)–C(9)	121·2 (2)

Fig. 1. The molecular conformation of (II*a*), showing atom numbering. The thermal ellipsoids are drawn at 50% probability level.
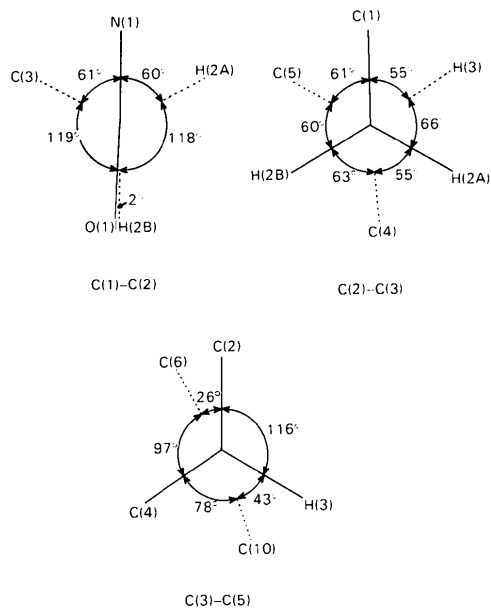

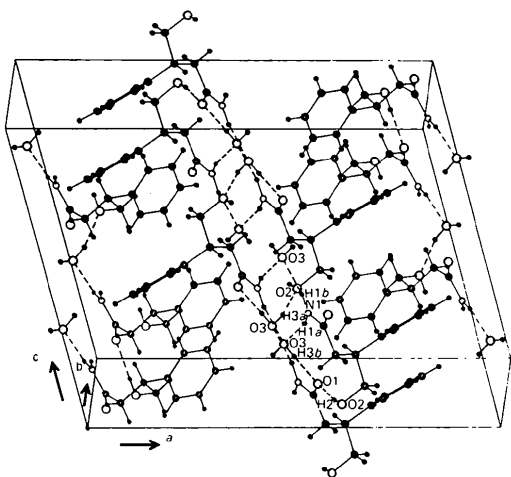Fig. 2. Newman projections of selected C—C bonds. Estimated standard deviations of torsion angles $0\cdot 3$–$0\cdot 5°$.

Fig. 3. A perspective drawing of the contents of one unit cell. The dashed lines indicate the intermolecular hydrogen bonds.

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Structure of 2,5-Dimethyl-1,3,4,6-tetraazacycl[3.3.3]azine,* C₁₀H₉N₅

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Abstract. $M_r = 199.22$, monoclinic, $P2_1/m$, $a = 4.022$ (1), $b = 17.640$ (1), $c = 6.568$ (1) Å, $\beta = 96.02$ (1)°, $V = 463.42$ Å³, $Z = 2$ (special positions), $D_x = 1.43$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.54178$ Å, $\mu = 6.73$ cm⁻¹, $F(000) = 208$, $T = 298$ K, final $R_w = 0.045$ for 770 observed $[I \geq 2.5\sigma(I)]$ reflections. The structure is planar. By comparison with the unmethylated compound the presence of methyl groups has no influence on either the bond lengths and angles, or the planarity of the molecule.

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* 2,5-Dimethyl-1,3,4,6,9b-pentaazaphenalene.
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Introduction. The title compound belongs to the family of azacyclazines, which are particularly interesting for their special aromatic character, due to the π-electron delocalization of the nitrogens onto the whole molecule. Its crystal structure has been studied by X-ray diffraction and compared with the corresponding unmethylated derivative (Lindqvist, Ljungström, Andréasson & Ceder, 1978) to characterize the influence of methyl substituents on geometrical properties.

Experimental. Red-purple crystals obtained by slow evaporation from a chloroform solution. Crystal $0.31 \times 0.43 \times 0.27$ mm, D_m not measured, 24 reflections ($62° \leq 2\theta \leq 80°$) used for measuring lattice parameters. 963 intensities collected, four-circle Enraf–