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Structure of 3,3-Diphenyl-*N*-(1-phenylethyl)propylamine (Fendiline) Hydrochloride

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Abstract. $C_{23}H_{26}N^+.Cl^-$, $M_r = 351·92$, monoclinic, $P2_1/n$, $a = 11·762$ (2), $b = 11·009$ (3), $c = 15·661$ (1) Å, $\beta = 93·34$ (2)°, $V = 2024$ Å^3, $Z = 4$, $D_x = 1·15$ g cm^{-3}, $\lambda(Cu K\alpha) = 1·54178$ Å, $\mu = 16·77$ cm^{-1}, $F(000) = 752$, room temperature, $R = 0·044$ for 1301 observed reflections. The dihedral angle between the two rings of the diphenyl group is 79 (1)°. The aliphatic chain is nearly maximally extended. The molecules are held together by N—H···Cl hydrogen bonds.

Introduction. First prepared some 25 years ago (Harsanyi, Korbonits & Kiss, 1964), fendiline is used in therapeutics in the treatment of ischemic cardiopathy. According to the World Health Organization, it belongs to calcium antagonists type V (prenylamine-like) (Vanhoutte & Paoletti, 1987). We here report the structure of the racemic mixture of fendiline hydrochloride.

Experimental. White crystal (from methanol), dimensions $0·15 \times 0·12 \times 0·10$ mm. Density not determined. Unit-cell parameters and intensity data obtained on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ in ω/θ scan mode ($0 < \theta < 65°$). Cell dimensions refined by least-squares fitting of θ values of 25 reflections. No appreciable drop in intensity of two standard reflections ($0\bar{2}\bar{2}$ and $00\bar{4}$) checked every 5400 s. 3442 independent reflections collected in $\pm h, k, l$ range $-13, 0, 0$ to $13, 12, 18$; 1301 with $I \geq 3\sigma(I)$ used in subsequent calculations. Intensities corrected for Lorentz and polarization effects but not for absorption. Scattering factors for non-H atoms from *International Tables for X-ray Crystallography* (1974) and for H from Stewart, Davidson & Simpson

(1965). The Wilson statistical test showed a centro-symmetric intensity distribution. Structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and standard Fourier techniques. H atoms located by ΔF synthesis. Block-diagonal-matrix least-squares refinement on F of observed reflections, $w = 1$ if $|F_o| < P$, $P = [F_o^2(\text{max.})/10]^{1/2}$, $w = (P/F_o)^2$ if $|F_o| > P$; anisotropic thermal parameters for all non-H atoms and isotropic ones for H. Final $R = 0·044$, $wR = 0·054$, $S = 0·9497$ (1301 reflections, 330 parameters). In final cycle mean and max. Δ/σ 0·1 and 0·5. Residual electron density within $\pm 0·3$ e Å^{-3}. Calculations carried out on a Mini 6-92 CII-Honeywell–Bull computer (programs *CRISTA*, *CRISAF*, *CRISEC*, *UTIL*, Laboratory of Crystal-lography, University of Bordeaux I, Talence).*

Discussion. The atomic parameters are given in Table 1 with the numbering scheme shown in Fig. 1. Bond distances and angles, close to expected values, are listed in Table 2. A view of the crystal structure is shown in Fig. 2.

The dihedral angle between the two rings of the diphenyl group is 79 (1)°. The phenyl ring C(2)–C(7) is oriented at 65 (1) and 56 (1)° towards the phenyl rings C(12)–C(17) and C(18)–C(23) respectively. The aliphatic chain C(11)–C(10)–C(9)–N(1)–C(8)–C(24) is nearly maximally extended, the torsion angles C(11)–C(10)–C(9)–N(1), C(10)–C(9)–N(1)–C(8)

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51391 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates (×10⁴) and equivalent isotropic temperature factors*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å²)
N(1)	4412 (3)	1004 (4)	1089 (2)	4·8 (2)
C(2)	3437 (4)	630 (4)	2413 (3)	5·0 (2)
C(3)	3315 (5)	1395 (5)	3103 (3)	6·2 (3)
C(4)	2284 (5)	1516 (5)	3469 (3)	7·2 (3)
C(5)	1365 (5)	872 (6)	3155 (4)	8·2 (3)
C(6)	1456 (5)	103 (6)	2473 (4)	8·0 (4)
C(7)	2490 (5)	−28 (5)	2108 (3)	6·6 (3)
C(8)	4547 (4)	539 (4)	1996 (3)	5·2 (2)
C(9)	3901 (4)	2237 (5)	1022 (3)	5·8 (3)
C(10)	3889 (4)	2687 (6)	99 (3)	7·2 (3)
C(11)	2962 (4)	3571 (5)	−95 (3)	6·3 (3)
C(12)	1782 (4)	3075 (4)	−3 (3)	5·0 (2)
C(13)	1034 (5)	3762 (5)	418 (4)	8·0 (4)
C(14)	−60 (5)	3393 (8)	523 (4)	11·0 (5)
C(15)	−424 (5)	2325 (8)	203 (4)	10·7 (5)
C(16)	285 (6)	1628 (6)	−215 (4)	9·9 (4)
C(17)	1399 (5)	1978 (6)	−316 (4)	7·5 (3)
C(18)	3084 (4)	4214 (5)	−941 (3)	5·4 (2)
C(19)	3157 (4)	5470 (5)	−937 (3)	6·6 (3)
C(20)	3250 (5)	6092 (6)	−1667 (4)	7·8 (3)
C(21)	3286 (5)	5521 (6)	−2423 (4)	8·2 (4)
C(22)	3208 (5)	4260 (6)	−2472 (3)	8·2 (4)
C(23)	3102 (5)	3627 (5)	−1710 (3)	6·8 (3)
C(24)	5061 (5)	−721 (6)	1980 (4)	8·5 (4)
Cl(25)	6687 (1)	937 (1)	205 (1)	5·8 (1)

Table 2. *Bond distances (Å) and angles (°)*

N(1)–C(8)	1·510 (6)	C(11)–C(18)	1·517 (7)
N(1)–C(9)	1·486 (6)	C(12)–C(13)	1·360 (7)
C(2)–C(3)	1·384 (7)	C(12)–C(17)	1·370 (7)
C(2)–C(7)	1·390 (7)	C(13)–C(14)	1·368 (9)
C(2)–C(8)	1·496 (6)	C(14)–C(15)	1·339 (10)
C(3)–C(4)	1·378 (7)	C(15)–C(16)	1·333 (10)
C(4)–C(5)	1·360 (8)	C(16)–C(17)	1·384 (9)
C(5)–C(6)	1·371 (8)	C(18)–C(19)	1·385 (7)
C(6)–C(7)	1·382 (8)	C(18)–C(23)	1·368 (7)
C(8)–C(24)	1·514 (8)	C(19)–C(20)	1·342 (8)
C(9)–C(10)	1·527 (7)	C(20)–C(21)	1·344 (8)
C(10)–C(11)	1·480 (7)	C(21)–C(22)	1·393 (8)
C(11)–C(12)	1·506 (7)	C(22)–C(23)	1·393 (8)

C(8)–N(1)–C(9)	113·3 (3)	C(11)–C(12)–C(13)	118·1 (4)
C(3)–C(2)–C(7)	117·9 (4)	C(11)–C(12)–C(17)	124·9 (4)
C(3)–C(2)–C(8)	120·9 (4)	C(13)–C(12)–C(17)	117·0 (5)
C(7)–C(2)–C(8)	121·2 (4)	C(12)–C(13)–C(14)	122·2 (6)
C(2)–C(3)–C(4)	121·2 (5)	C(13)–C(14)–C(15)	119·9 (6)
C(3)–C(4)–C(5)	119·5 (5)	C(14)–C(15)–C(16)	119·6 (6)
C(4)–C(5)–C(6)	120·5 (5)	C(15)–C(16)–C(17)	121·3 (6)
C(5)–C(6)–C(7)	119·8 (5)	C(12)–C(17)–C(16)	119·9 (5)
C(2)–C(7)–C(6)	120·7 (5)	C(11)–C(18)–C(19)	118·0 (4)
N(1)–C(8)–C(2)	110·0 (4)	C(11)–C(18)–C(23)	123·8 (4)
N(1)–C(8)–C(24)	108·3 (4)	C(19)–C(18)–C(23)	118·2 (4)
C(2)–C(8)–C(24)	115·4 (4)	C(18)–C(19)–C(20)	120·9 (5)
N(1)–C(9)–C(10)	110·2 (4)	C(19)–C(20)–C(21)	121·3 (5)
C(9)–C(10)–C(11)	112·0 (4)	C(20)–C(21)–C(22)	120·7 (5)
C(10)–C(11)–C(12)	114·4 (4)	C(21)–C(22)–C(23)	117·4 (5)
C(10)–C(11)–C(18)	112·4 (4)	C(18)–C(23)–C(22)	121·6 (5)
C(12)–C(11)–C(18)	112·7 (4)		

Fig. 1. View of the molecule showing the numbering of the atoms.

coordinates are given in Table 1 is *R* according to the sequence rule (Cahn & Ingold, 1951). The distances between the protonated nitrogen N(1) and the centres of the phenyl rings C(2)–C(7), C(12)–C(17) and C(18)–C(23) are 3·679 (4), 4·944 (4) and 6·196 (5) Å respectively.
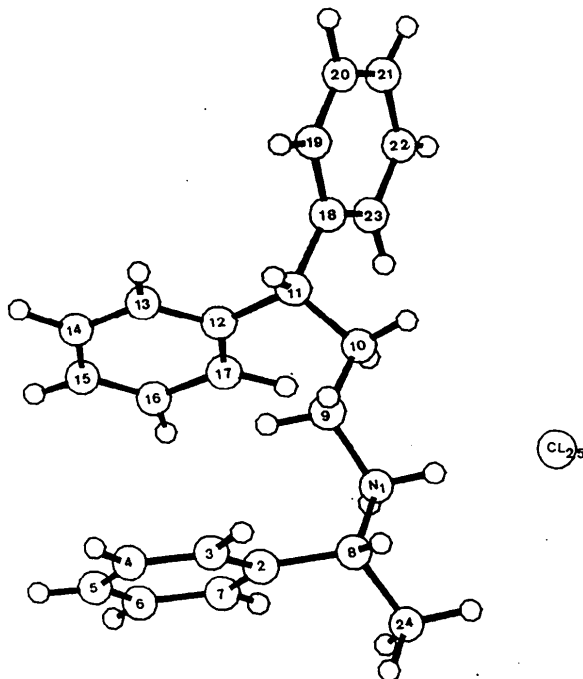
and C(9)–N(1)–C(8)–C(24) being 153 (1), 175 (1) and 170 (1)° respectively, a conformation found in other diphenylpropylamine calcium antagonists such as prenylamine (E. F. Paulus, unpublished), terodiline (Carlström & Hacksell, 1983) or bepidril (Declercq & Van Meerssche, 1984). The configuration around the asymmetric carbon atom C(8) of the molecule whose
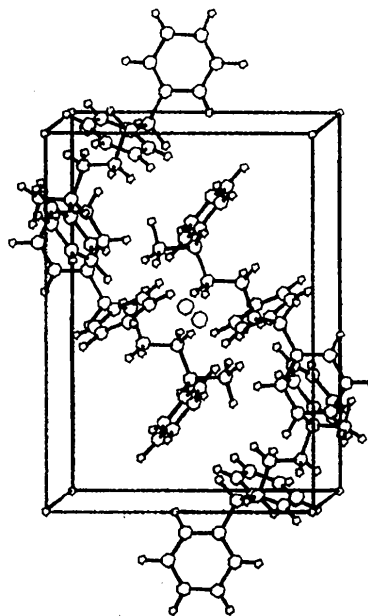

Fig. 2. Contents of the unit cell.

The molecules are held together by N—H···Cl hydrogen bonds. The geometry of this bond is $N(1)···Cl(25)(1-x,y,z) = 3·169$ (4), $H(201)···Cl(25) = 2·27$ (4) Å, $N(1)—H(201)···Cl(25) = 164$ (4)°.

The authors wish to thank the 'Societa Prodotti Antibiotici' (Milan, Italy) for the gift of the sample.

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Structure of 6-Amino-4,4,5,7,8-pentamethyldihydrocoumarin

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Abstract. $C_{14}H_{19}NO_2$, $M_r = 233·3$, triclinic, $P\bar{1}$, $a = 9·925$ (2), $b = 11·193$ (3), $c = 11·915$ (3) Å, $\alpha = 95·39$ (2), $\beta = 91·09$ (2), $\gamma = 97·38$ (2)°, $V = 1304·7$ (3) Å^3, $Z = 4$, $F(000) = 480$, $D_x = 1·19$ (1), $D_m = 1·18$ (1) g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu(Mo\ K\alpha) = 0·43$ cm^{-1}, $T = 292$ K. Final $R = 0·045$ for 3099 observed reflections. The statistically significant difference in bond length C(6)—N between the two independent molecules can be explained in terms of the differences in the intermolecular interactions of the N atoms. The conformation of the lactone ring in both independent molecules is intermediate between half-chair and sofa and very similar to that observed in 4,4,5,7,8-pentamethyldihydrocoumarin. In both these compounds similar large distortions of valency angles occur in the phenyl rings to accommodate the over-methylation.

Introduction. The crystal structure of the title compound (hereafter referred to as I) opens a series of papers on our studies of structural properties of coumarin derivatives. Similar compounds were synthesized and used by Milstien & Cohen (1972) who introduced the concept of stereopopulation control to show the importance of conformational restrictions in enzyme-catalysed reactions. The enzyme enhancements of such reactions often range from 10^{10} to 10^{18}. Among a number of factors which make such rate enhancement possible Milstien & Cohen (1972) indi-

cated the importance of the restrictions in conformational freedom of the substrate resulting from its interactions with the enzyme. The coumarin derivatives were the model compounds chosen to demonstrate this process. Milstien & Cohen (1972) and Borchardt & Cohen (1972) described the influence of substituents – three methyl groups – restricting the freedom of conformational changes of a side chain with acid group and so promoting rapid lactonization. To support their work, two X-ray diffraction studies on the structures of pentamethyl-o-hydroxydihydrocinnamyl alcohol and of 4,4,5,7,8-pentamethyldihydrocoumarin were carried out by Karle & Karle (1972). The present study was intended to supply additional structural data for the studies on stereopopulation control as well as to study the influence of substituents on minor conformational changes.

Experimental. Crystals suitable for X-ray analysis were obtained from an ethanol–acetone solution. They were colourless elongated prisms. The crystal used for data collection was a piece cut off a larger crystal, its dimensions being $0·3 \times 0·3 \times 0·3$ mm. It was mounted on a CAD-4 diffractometer, and graphite-monochromated Mo $K\alpha$ radiation was used. The unit-cell parameters were determined by a least-squares fit to 16 automatically centred reflections ($12·0 \le 2\theta \le 31·0°$). The 2θ–θ scan method was applied with a variable scan speed ranging between $2·0$ and $20·0°$ min^{-1} depending