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2,3-Dihydro-5,7-diphenyl-1,4-diazepinium Perchlorate Hemihydrate

By Douglas Lloyd

Department of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST, Scotland

and George Ferguson* and Barbara L. Ruhl

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

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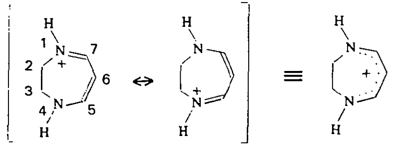
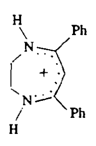
Abstract. $C_{17}H_{17}N_2^+.ClO_4^-.0{\cdot}5H_2O$, $M_r = 357{\cdot}8$, triclinic, $P\bar{1}$, $a = 9{\cdot}158\,(2)$, $b = 13{\cdot}115\,(2)$, $c = 8{\cdot}372\,(2)$ Å, $\alpha = 95{\cdot}15\,(2)$, $\beta = 107{\cdot}28\,(2)$, $\gamma = 72{\cdot}50\,(2)°$, $V = 915{\cdot}7\,(6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}70926$ Å, $\mu = 2{\cdot}3$ cm^{-1}, $F(000) = 374$, $T = 293$ K, $R = 0{\cdot}074$, $wR = 0{\cdot}083$ for 2007 observed data. The crystal structure contains discrete diazepinium cations, perchlorate anions and water molecules linked by a network of hydrogen bonds. The cation contains a five-membered delocalized 1,5-diazapentadienium chain [N—C—C—C—N; mean C—C 1·395 (6), mean C—N 1·332 (6) Å] in a helical conformation. The perchlorate anion and water molecules are disordered.

Introduction. An X-ray structure determination of the 2,3-dihydro-1,4-diazepinium cation (I) (Ferguson, Ruhl, Wieckowski, Lloyd & McNab, 1984) has shown that it consists of a delocalized vinamidinium (3-amino-2-propeniminium) system [N(4),C(5–7),N(1)], whose ends are linked through an ethylene bridge [C(2,3)]. Phenyl substituents attached to a dihydrodiazepinium ring are known to interact electronically with the vinamidinium system (Lloyd, Mackie, McNab, Tucker & Marshall, 1976; Butler, Lloyd, McNab, Marshall & Tucker, 1989); this is evident from both the spectroscopic and chemical properties. It was of interest, therefore, to study structural details of a phenyl derivative, and the 5,7-diphenyl derivative (II) has accordingly been investigated.



(I)



(II)

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* E-mail address: CHMFERG@VM.UOGUELPH.CA

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Table 1. *Final fractional coordinates* ($\times 10^4$) *and thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	Occ.	x	y	z	B_{eq}(Å^2)
Cl		−336 (2)	−3106 (1)	6638 (2)	6·7 (1)
O(1)	0·97	−19 (12)	−2560 (7)	5642 (14)	19 (1)
O(2)	0·65	151 (13)	−4191 (7)	5721 (13)	11 (1)
O(3)	0·39	−784 (17)	−2004 (8)	7512 (16)	9 (1)
O(4)	0·72	−1924 (13)	−2889 (11)	6186 (23)	18 (1)
O(5)	0·86	815 (18)	−3404 (10)	7967 (19)	19 (1)
O(6)	0·40	−870 (30)	−3649 (13)	7387 (27)	14 (1)
O(*W*1)	0·25	492 (21)	−378 (13)	625 (21)	7·3 (8)
O(*W*2)	0·25	−918 (26)	−905 (18)	−1249 (28)	9·4 (8)
N(1)		2673 (5)	2126 (4)	141 (6)	5·2 (2)
C(2)		1416 (6)	2758 (5)	−1211 (7)	5·7 (3)
C(3)		1403 (6)	3906 (5)	−1139 (7)	5·2 (3)
N(4)		2579 (5)	4073 (3)	−1822 (5)	4·4 (2)
C(5)		4097 (6)	3488 (4)	−1477 (6)	3·8 (2)
C(6)		4827 (6)	2604 (4)	−394 (6)	6·4 (2)
C(7)		4216 (6)	2058 (4)	465 (6)	4·0 (2)
C(51)		5121 (6)	3875 (4)	−2208 (6)	3·7 (2)
C(52)		6260 (6)	3152 (5)	−2873 (6)	4·8 (2)
C(53)		7205 (6)	3518 (5)	−3550 (7)	5·2 (3)
C(54)		7074 (7)	4595 (6)	−3558 (7)	5·4 (3)
C(55)		5956 (7)	5305 (5)	−2889 (6)	5·1 (3)
C(56)		4977 (6)	4954 (4)	−2239 (6)	4·4 (2)
C(71)		5319 (6)	1306 (4)	1813 (6)	4·2 (2)
C(72)		6719 (7)	1520 (4)	2853 (7)	4·9 (3)
C(73)		7726 (8)	834 (5)	4103 (9)	6·0 (3)
C(74)		7421 (8)	−85 (5)	4384 (8)	5·9 (3)
C(75)		6073 (9)	−304 (5)	3408 (8)	6·8 (4)
C(76)		5006 (7)	376 (4)	2115 (7)	5·5 (3)

Table 2. *Interatomic distances* (Å) *and angles* (°), *and hydrogen-bond geometry* (Å,°)

N(1)—C(2)	1·454 (6)	C(52)—C(53)	1·374 (7)
N(1)—C(7)	1·335 (6)	C(53)—C(54)	1·381 (8)
C(2)—C(3)	1·497 (8)	C(54)—C(55)	1·379 (8)
C(3)—N(4)	1·437 (6)	C(55)—C(56)	1·377 (7)
N(4)—C(5)	1·328 (6)	C(71)—C(72)	1·411 (7)
C(5)—C(6)	1·406 (6)	C(71)—C(76)	1·394 (7)
C(5)—C(51)	1·481 (7)	C(72)—C(73)	1·360 (8)
C(6)—C(7)	1·384 (6)	C(73)—C(74)	1·372 (9)
C(7)—C(71)	1·479 (7)	C(74)—C(75)	1·359 (9)
C(51)—C(52)	1·398 (7)	C(75)—C(76)	1·394 (8)
C(51)—C(56)	1·383 (7)		

C(2)—N(1)—C(7)	124·0 (4)	C(72)—C(71)—C(76)	117·8 (5)
N(1)—C(2)—C(3)	112·6 (5)	C(71)—C(72)—C(73)	120·7 (6)
C(2)—C(3)—N(4)	112·5 (5)	C(72)—C(73)—C(74)	121·3 (6)
C(3)—N(4)—C(5)	125·4 (4)	C(73)—C(74)—C(75)	119·1 (6)
N(4)—C(5)—C(6)	126·2 (4)	C(74)—C(75)—C(76)	121·6 (6)
N(4)—C(5)—C(51)	115·6 (4)	C(75)—C(76)—C(71)	119·5 (6)
C(51)—C(5)—C(6)	118·0 (4)	O(1)—Cl—O(2)	94·5 (6)
C(5)—C(6)—C(7)	131·9 (5)	O(1)—Cl—O(3)	83·1 (6)
C(6)—C(7)—N(1)	124·8 (4)	O(1)—Cl—O(4)	105·9 (9)
C(6)—C(7)—C(71)	119·5 (4)	O(1)—Cl—O(5)	113·0 (10)
N(1)—C(7)—C(71)	115·6 (4)	O(1)—Cl—O(6)	169·3 (13)
C(5)—C(51)—C(52)	120·4 (4)	O(2)—Cl—O(3)	177·6 (5)
C(5)—C(51)—C(56)	120·9 (5)	O(2)—Cl—O(4)	101·4 (8)
C(52)—C(51)—C(56)	118·7 (5)	O(2)—Cl—O(5)	93·6 (7)
C(51)—C(52)—C(53)	120·1 (5)	O(2)—Cl—O(6)	82·4 (8)
C(52)—C(53)—C(54)	121·1 (6)	O(3)—Cl—O(4)	79·9 (8)
C(53)—C(54)—C(55)	118·7 (5)	O(3)—Cl—O(5)	86·8 (8)
C(54)—C(55)—C(56)	121·0 (5)	O(3)—Cl—O(6)	100·1 (8)
C(55)—C(56)—C(51)	120·5 (5)	O(4)—Cl—O(5)	136·9 (11)
C(7)—C(71)—C(72)	120·6 (5)	O(4)—Cl—O(6)	65·0 (11)
C(7)—C(71)—C(76)	121·6 (5)	O(5)—Cl—O(6)	77·5 (12)

N(1)···O(3$^{\text{i}}$)	3·02 (1)	N(1)—H(1)···O(3$^{\text{i}}$)	141
N(1)···O(*W*2$^{\text{ii}}$)	2·95 (2)	N(1)—H(1)···O(*W*2$^{\text{ii}}$)	151
H(1)···O(3$^{\text{i}}$)	2·22	N(4)—H(4)···O(2$^{\text{iii}}$)	146
H(1)···O(*W*2$^{\text{ii}}$)	2·08		
N(4)···O(2$^{\text{iii}}$)	3·07 (1)		
H(4)···O(2$^{\text{iii}}$)	2·24		
O(*W*1)···O(3$^{\text{iv}}$)	3·38 (2)		
O(*W*1)···O(3$^{\text{i}}$)	3·41 (2)		

The superscripts refer to the following equivalent positions: (i) $-x$, $-y$, $1-z$; (ii) $-x$, $-y$, $-z$; (iii) x, $1+y$, $-1+z$; (iv) x, y, $-1+z$.

Experimental. Pale-yellow crystals, crystal size 0·20 × 0·25 × 0·33 mm, CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, 25 reflections with θ in range $10 < \theta < 15°$ used to determine lattice parameters; for data collection $2 < \theta < 27°$, h 0 to 11, k −16 to 16, l −10 to 10, ω–2θ scans, ω-scan width $(0·80 + 0·35\tan\theta)°$; intensities of three reflections monitored every 100 min of exposure time showed no significant decay; Lorentz and polarization corrections (no absorption correction required). 3989 unique reflections, 2007 with $I > 3\sigma(I)$ labelled observed and used in structure solution and refinement. There were very few reflections with $\theta > 20°$ hence the rather low observed/total reflection ratio (50%). The structure was solved by the heavy-atom method. Subsequent electron-density maps phased with the Cl-atom contributions served to reveal the cation structure as well as six peaks around the Cl atom (corresponding to disordered perchlorate O atoms); oxygen occupancy factors were assigned from relative peak heights. There were also two smaller peaks in the density maps adjacent to an inversion centre; from their positions in the cell, their distances to adjacent O and N atoms and their peak densities they were taken to be disordered water molecules with 0·25 occupancies (with the crystal composition corresponding overall to a hemihydrate). In the full-matrix least-squares refinement cycles (on F), the non-H atoms (apart from the water molecules) were allowed anisotropic motion and H atoms were included with idealized geometry (C—H, N—H, 0·95 Å) and an overall isotropic temperature factor. At convergence $R = 0·074$, $wR = (\sum w\Delta^2/\sum wF_o^2)^{1/2} = 0·083$ for 2007 observed data, $w = 1/(\sigma F_o^2)$. We attribute the relatively high R factors to the disorder of the perchlorate O atoms and the water molecules; in the final difference map there were small maxima 0·5 e Å^{-3} in the vicinity of the disordered perchlorate O atoms and water molecules. Maximum shift/e.s.d. = 0·35 for U_{33} of O(5), mean shift/e.s.d. = 0·04. Atomic scattering factors were from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs used included *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1979) and *ORTEP*II (Johnson, 1976). Final fractional coordinates with equivalent isotropic temperature factors are in Table 1. Table 2 contains ion dimensions and details of hydrogen bonding.* A view (Johnson, 1976) of the cation with the crystallographic numbering scheme is shown in Fig. 1.

Discussion. The crystal structure contains discrete substituted diazepinium cations, perchlorate anions and water molecules; the cation N—H moieties are linked to the disordered perchlorate O atoms and water molecules by N—H···O hydrogen bonds (2·95–3·07 Å). The perchlorate O atoms appear to be quite disordered in the crystal lattice; each of the six main sites whose coordinates are given in Table 1 probably represents several partial atoms lying in the same volume element in the lattice.

In the substituted dihydrodiazepinium cation (Fig. 1) the bond lengths of the N(4)—C(5)—C(6)—C(7)—N(1) system [mean C—C 1·395 (6), mean C—N 1·332 (6) Å] (Table 2) are consistent with a fully delocalized system and these atoms define a portion of a helix with N(4) 0·078 (6), C(5) −0·087 (6), C(6) −0·020 (6), C(7) 0·115 (6) and N(1) −0·087 (6) Å from the best plane through them. These displacements correspond to torsion angles N(4)—C(5)—C(6)—C(7) 5·9 (6) and C(5)—C(6)—C(7)—N(1) 16·0 (6)°. The remaining two atoms of the seven-membered ring are −0·528 (6) [C(2)] and +0·363 (6) Å [C(3)] from the plane of the delocalized atoms, corresponding to an N(1)—C(2)—C(3)—N(4) torsion angle of 80·5 (7)°. The angles between the five-atom vinamidinium plane and each of the phenyl ring planes are 46·1 (6) and 38·2 (6)°; the torsion angles C(7)—C(6)—C(5)—C(51) and C(5)—C(6)—C(7)—C(71) are −179·6 (6) and −166·3 (6)°, respectively. The cation thus has approximate twofold symmetry, with the twofold axis passing through C(6) and the C(2)—C(3) midpoint. The ring valency angles at the N and sp^2-hybridized C atoms have been increased from the normal 120° to a mean value of 126·5 (4)° (Table 2). The sp^3-hybridized C atoms have N—C—C angles close to those expected [mean value 112·6 (5)°].

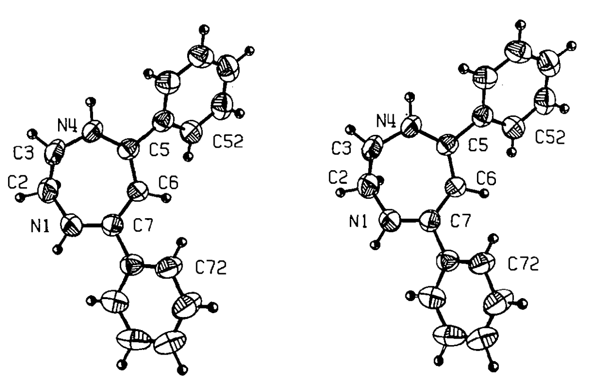
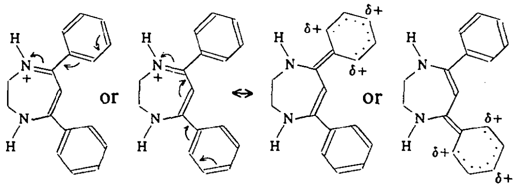


Fig. 1. A stereoview of the $C_{17}H_{17}N_2^+$ ion.

The overall structure of the dihydrodiazepinium ring in (II) thus closely resembles that of its unsubstituted analogue (I). The mean C—N distances of the vinamidinium system [N(4)—C(5)] and [N(1)—C(7)] are, however, appreciably longer [1·332 (6) Å] than in the case of the unsubstituted cation [1·306 (4) Å]. 'Curved-arrow' analysis of (II) suggests that electronic interaction between phenyl and dihydrodiazepinium rings is electron donating from the phenyl group when the latter is located at the 5 or 7 positions; this is confirmed by spectroscopic evidence (Lloyd, Mackie, McNab, Tucker & Marshall, 1976).



(II)

Contributions from such forms should result in a lengthening of the N(4)—C(5) and N(1)—C(7) bonds. This interaction between the rings is akin to that possible between the two phenyl rings in biphenyl-like systems. In accord with this, the bonds linking the phenyl groups to the dihydrodiazepinium ring [mean 1·480 (7) Å] are comparable with the value reported for the central bond of 91 biphenyl-like systems with *ortho* H atoms (1·484 Å) by Brock & Minton (1989); these authors also showed that there is no correlation between the central C—C distance and rotation angle ψ. Similar steric factors apply in the case of (II) as with biphenyl itself (Trotter, 1961; Charbonneau & Delugeard, 1977) with potential overlap between *ortho* H atoms. The angles between the planes of the phenyl groups and that of the five-atom vinamidinium plane [46·1 (6) and 38·2 (6)°] are very similar to those observed for biphenyl either in solution [32 (2)°; Eaton & Steele, 1973] or in the gas phase [45 (10)°; Bastiansen, 1949]; the crystal forces which make biphenyl planar in its crystalline form will be less operative in the case of (II) since here the seven-membered ring is not intrinsically planar. It may also be significant that the *ipso–ortho* bonds of the phenyl groups are longer [mean 1·397 (7) Å] than the other bonds of these rings [mean 1·375 (7) Å] in accord with electronic interaction with the vinamidinium system and concomitant setting up of pentadienium systems in the phenyl rings as indicated.

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* Lists of structure factors, calculated H-atom coordinates, anisotropic thermal parameters and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53610 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-[2-(*o*-Carboxybenzamido)ethyl]phenoxyacetic Acid Dihydrate

BY RASHMI S. HEGDE, AHMED MEHANNA* AND DONALD J. ABRAHAM*†

Department of Medicinal Chemistry, School of Pharmacy, University of Pittsburgh, Pittsburgh, PA 15261, USA

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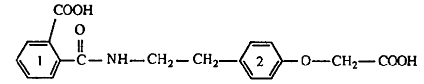
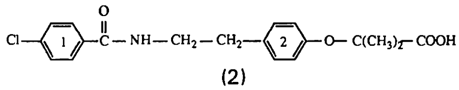
Abstract. $C_{18}H_{17}NO_6.2H_2O$, $M_r = 379\cdot386$, monoclinic, $P2_1/c$, $a = 7\cdot161\ (5)$, $b = 12\cdot997\ (2)$, $c = 20\cdot290\ (3)$ Å, $\beta = 99\cdot22\ (2)^\circ$, $V = 1861\cdot4$ Å^3, $Z = 4$, $D_x = 1\cdot354$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1\cdot5418$ Å, $\mu = 9\cdot2$ cm^{-1}, $F(000) = 800$, $T = 293$ K, final $R(F) = 0\cdot051$ for 2262 observed reflections. The structure was solved by direct methods and compared to the previously determined structure of the analogous drug molecule, bezafibrate. The structural characteristics of the two compounds are then correlated to their relative activities as allosteric effectors of hemoglobin. The molecule adopts a synplanar–antiplanar conformation unlike other phenoxyacetic acids.

Introduction. 4-[2-(*o*-Carboxybenzamido)ethyl]phenoxyacetic acid, AM49 (1), is an analog of the antihyperlipoproteinemic agent bezafibrate, BZF (2).



(1)



(2)

BZF has been shown to bind to the central water cavity of deoxyhemoglobin (Perutz, Fermi, Abraham, Poyart & Bursaux, 1986) and acts as an allosteric effector of hemoglobin. The crystal structure of bezafibrate has previously been reported [$P2_12_12_1$; $a = 10\cdot319\ (1)$, $b = 17\cdot823\ (2)$, $c = 19\cdot842\ (1)$ Å] (Hegde, Sawzik, McClure & Abraham, 1988) and later confirmed by Djinovic, Globokar & Zupet (1989) [$P2_12_12_1$; $a = 10\cdot222\ (2)$, $b = 17\cdot826\ (3)$, $c = 19\cdot921\ (4)$ Å]. In a continuing effort to design compounds with enhanced allosteric activity, AM49 introduces an ortho acid functionality tailored to interact electrostatically with the positive charge on Lys99α of hemoglobin. The crystal structure of AM49 is reported here along with a comparison of its features with those of the parent compound BZF.

Experimental. The title compound was crystallized from methanol at room temperature. Data were collected on a Rigaku AFC5 diffractometer equipped with a rotating-anode X-ray source. The crystal had dimensions $0\cdot2 \times 0\cdot2 \times 0\cdot5$ mm. Cell parameters were measured on the diffractometer using 25 reflections in the 2θ range 50–80°. Range of indices $0 < h < 9$, $0 < k < 16$, $-24 < l < 24$ ($\theta < 60^\circ$). Empirical absorption correction was applied based on ψ scans of three reflections with minimum and maximum transmission coefficients of $0\cdot84$ and $1\cdot00$. Three standard reflections ($03\bar{1}$, $13\bar{1}$, 112) measured every 150 reflections showed no decrease in intensity (99·7, 100·3

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* Present address: Department of Medicinal Chemistry, School of Pharmacy, Virginia Commonwealth University, 548A Smith Building, Box 581, MCV Station, Richmond, VA 23298-0581, USA.

† To whom correspondence should be addressed.

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