when it is compared with that of thromboxane B_2. The difference of the torsion angle between the bicycloheptane and the carboxyl arm confers no extra stability in the free molecule and may simply be the result of the hydrogen bonding. Clarification of the mode of action of these molecules must await the structure of the receptor.

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Structure of 9-Chloro-7-(2-chlorophenyl)-3,5-dihydro-[1,2,4]triazino[4,3-a][1,4]-benzodiazepin-2(1H)-one*

BY HELEN J. KEMMISH AND THOMAS A. HAMOR

Department of Chemistry, University of Birmingham, Birmingham B15 2TT, England

Abstract. $C_{17}H_{12}Cl_2N_4O$, $M_r = 359 \cdot 2$, triclinic, $P\bar{1}$, $a = 8 \cdot 777$ (8), $b = 12 \cdot 715$ (4), $c = 14 \cdot 883$ (4) Å, $\alpha = 95 \cdot 69$ (2), $\beta = 83 \cdot 62$ (3), $\gamma = 93 \cdot 46$ (3)°, $V = 1640 \cdot 5$ Å^3, $Z = 4$, $D_x = 1 \cdot 454$ g cm^{-3}, λ(Mo $K\alpha$) = $0 \cdot 71069$ Å, $\mu = 3 \cdot 99$ cm^{-1}, $F(000) = 736$, $T = 293$ K, $R = 0 \cdot 039$ for 2783 observed reflections. The seven-membered heterocyclic ring has a cycloheptatriene-like boat conformation with bow and stern angles $60 \cdot 5$ (8) and $33 \cdot 8$ (8)°, and $59 \cdot 8$ (8) and $35 \cdot 9$ (8)°, respectively, in the two independent molecules. The triazino ring is near planar in both molecules. The angles between the 7-phenyl ring and the fused benzo moiety are $88 \cdot 9$ (8) and $82 \cdot 0$ (8)°. Corresponding bond lengths and angles in the two molecules are generally similar and agree well with accepted values.

Introduction. The title compound (Szmuszkovicz, 1973) is related to the classical psychoactive 5-phenyl-1,4-benzodiazepin-2-ones such as diazepam, but differs from them in containing a six-membered hetero ring fused across the N(1)—C(2) bond of the parent system. Many of these diazepam derivatives exhibit a high degree of biological activity and the present compound binds to the benzodiazepine receptor *in vitro* about eight times more strongly than diazepam.

Experimental. Crystals were grown from amyl acetate. A crystal of size $0 \cdot 3 \times 0 \cdot 4 \times 0 \cdot 7$ mm was mounted on an Enraf–Nonius CAD-4 diffractometer and cell dimensions determined from the setting angles of 25 reflections in the range $10 < \theta < 16°$. Intensity data were measured with graphite-monochromated Mo $K\alpha$ radiation. 5121 unique reflections were scanned by ω–2θ scans up to $\theta = 24°$, 2783 [$I > 2 \cdot 5\sigma(I)$] reflections were considered observed and used in the analysis, index range h -9 to 10, k -14 to 14, l 0 to 16. Two standard reflections measured every 2 h showed no significant variation in intensity. No absorption corrections were applied.

The structure was determined by direct methods and refined by least squares using anisotropic thermal parameters for the heavier atoms. H atoms were initially located from a difference synthesis and then

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^2$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Molecule A				
Cl(2)	2032 (1)	5121 (1)	5896 (1)	88
Cl(9)	-4792 (1)	4914 (1)	7038 (1)	71
O	3835 (3)	3983 (2)	9440 (2)	60
N(1)	1159 (3)	5587 (2)	8597 (2)	45
N(3)	3820 (4)	5737 (2)	9363 (2)	49
N(4)	3210 (4)	6663 (2)	9148 (2)	49
N(6)	1373 (4)	7540 (2)	7573 (2)	49
C(1)	1798 (6)	4611 (4)	8780 (5)	69
C(2)	3244 (4)	4758 (3)	9221 (3)	46
C(4)	1942 (4)	6539 (3)	8786 (2)	44
C(5)	1239 (6)	7506 (3)	8552 (3)	52
C(7)	507 (4)	6879 (3)	7139 (3)	42
C(8)	-2003 (4)	5898 (3)	7187 (3)	45
C(9)	-3070 (4)	5155 (3)	7513 (3)	50
C(10)	-2760 (5)	4570 (3)	8196 (3)	56
C(11)	-1375 (4)	4741 (3)	8544 (3)	51
C(12)	-283 (4)	5489 (3)	8236 (2)	41
C(13)	-598 (4)	6084 (3)	7538 (2)	40
C(1')	598 (4)	6953 (3)	6139 (3)	46
C(2')	1250 (5)	6209 (4)	5524 (3)	61
C(3')	1336 (7)	6328 (6)	4588 (4)	88
C(4')	740 (8)	7185 (7)	4305 (5)	97
C(5')	83 (7)	7928 (6)	4910 (5)	90
C(6')	20 (5)	7833 (4)	5819 (4)	65
Molecule B				
Cl(2)	-7096 (1)	-416 (1)	3924 (1)	80
Cl(9)	-619 (1)	-598 (1)	2821 (1)	65
O	-9659 (3)	-2558 (2)	730 (2)	53
N(1)	-6575 (3)	-675 (2)	1264 (2)	39
N(3)	-9259 (4)	-843 (2)	524 (2)	48
N(4)	-8454 (4)	146 (2)	595 (2)	47
N(6)	-6491 (3)	1597 (2)	2074 (2)	46
C(1)	-7345 (5)	-1717 (3)	1159 (4)	57
C(2)	-8862 (4)	-1738 (3)	796 (2)	40
C(4)	-7194 (4)	174 (3)	962 (2)	41
C(5)	-6347 (5)	1233 (3)	1107 (3)	51
C(7)	-5718 (4)	1111 (3)	2573 (2)	40
C(8)	-3303 (4)	211 (3)	2601 (3)	41
C(9)	-2362 (4)	-604 (3)	2362 (2)	44
C(10)	-2789 (5)	-1442 (3)	1765 (3)	51
C(11)	-4162 (4)	-1438 (3)	1402 (3)	47
C(12)	-5133 (4)	-611 (3)	1624 (2)	39
C(13)	-4707 (4)	230 (3)	2248 (2)	37
C(1')	-5772 (4)	1495 (3)	3552 (3)	44
C(2')	-6325 (4)	847 (3)	4224 (3)	55
C(3')	-6334 (6)	1206 (6)	5128 (4)	79
C(4')	-5807 (7)	2215 (6)	5374 (4)	90
C(5')	-5261 (6)	2853 (5)	4720 (5)	87
C(6')	-5242 (5)	2509 (4)	3825 (4)	60

refined isotropically. Weights, $w = 1/[\sigma^2(F) + 0.001F^2]$, which resulted in a satisfactory weighting analysis, were used in the least-squares refinement. The refinement was carried out on F values in two blocks and converged to $R = 0.039$, $wR = 0.048$ with maximum shift/e.s.d. ratio < 0.1. The residual electron density in a final difference synthesis was within $\pm 0.3 \text{ e Å}^{-3}$. No correction for secondary extinction was applied. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Computations were carried out on the University of Birmingham Honeywell DPS 8/70 computer and on the CDC 7600 at the University of Manchester Regional Computer Centre with the *SHELX*76 (Sheldrick, 1978) and *PLUTO* (Motherwell & Clegg, 1978) programs.

Discussion. Final atomic parameters for the two independent molecules (labelled A and B) are listed in Table 1,* and bond lengths, bond angles and selected torsion angles in Table 2. The atomic numbering scheme is shown in Fig. 1.

The bond lengths and angles of the two molecules are generally in close agreement with each other and are also similar to values found previously in analogous structures (Hamor & Martin, 1983). The N(1)—C(4) bond is shortened from the normal single-bond value of *ca* 1.47 Å to 1.375 (4) or 1.373 (5) Å, due to electron delocalization between N(1) and N(4). Consistent with this the N(4)—C(4) formal double bond is very slightly longer than a normal C=N double bond [*cf.* N(6)—C(7)]. There is a near-planar disposition of bonds about N(1) and C(4), so that the geometry of this bond resembles that of a double bond. The disposition of bonds about N(3) is also near planar and the triazino ring is planar to within ± 0.05 (1) Å for both molecules.

The seven-membered ring adopts a cyclo-heptatriene-like boat conformation, as is commonly found in this class of compounds (Hamor & Martin, 1983). The 'boat' can be described in terms of the angles between the central plane, consisting of atoms

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52369 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
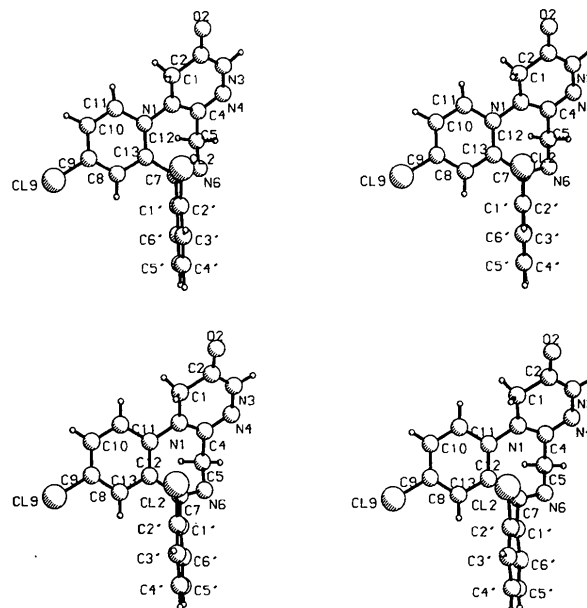

Fig. 1. Stereoscopic views of the two independent molecules in a direction perpendicular to the mean plane through atoms C(8)—C(13); upper diagram, molecule A, lower diagram, molecule B.

$C_{17}H_{12}Cl_2N_4O$

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses and selected torsion angles* (°) *with e.s.d.'s ca* 0·7°

	Molecule A	Molecule B		Molecule A	Molecule B
Cl(9)—C(9)	1·737 (4)	1·742 (4)	C(7)—C(1')	1·494 (5)	1·487 (5)
Cl(2')—C(2')	1·745 (5)	1·741 (4)	C(8)—C(9)	1·374 (5)	1·359 (5)
O—C(2)	1·228 (5)	1·222 (4)	C(8)—C(13)	1·391 (5)	1·395 (5)
N(1)—C(1)	1·454 (6)	1·453 (5)	C(9)—C(10)	1·378 (6)	1·380 (5)
N(1)—C(4)	1·375 (4)	1·373 (5)	C(10)—C(11)	1·371 (6)	1·375 (6)
N(1)—C(12)	1·423 (5)	1·422 (5)	C(11)—C(12)	1·383 (5)	1·385 (5)
N(3)—N(4)	1·401 (5)	1·406 (4)	C(12)—C(13)	1·405 (5)	1·406 (5)
N(3)—C(2)	1·322 (5)	1·323 (5)	C(1')—C(2')	1·355 (6)	1·387 (6)
N(4)—C(4)	1·285 (5)	1·284 (5)	C(1')—C(6')	1·402 (7)	1·382 (6)
N(6)—C(5)	1·454 (5)	1·460 (5)	C(2')—C(3')	1·410 (8)	1·376 (7)
N(6)—C(7)	1·274 (5)	1·283 (5)	C(3')—C(4')	1·358 (12)	1·367 (10)
C(1)—C(2)	1·486 (7)	1·489 (6)	C(4')—C(5')	1·349 (10)	1·357 (10)
C(4)—C(5)	1·496 (6)	1·507 (5)	C(5')—C(6')	1·364 (9)	1·360 (8)
C(7)—C(13)	1·479 (5)	1·481 (5)			
C(1)—N(1)—C(4)	119·7 (3)	119·8 (3)	C(8)—C(9)—C(10)	120·3 (4)	120·2 (4)
C(1)—N(1)—C(12)	116·6 (3)	116·8 (3)	C(9)—C(10)—C(11)	119·0 (4)	119·5 (4)
C(4)—N(1)—C(12)	123·7 (3)	123·2 (3)	C(10)—C(11)—C(12)	122·1 (4)	121·5 (4)
N(4)—N(3)—C(2)	127·1 (3)	127·2 (3)	N(1)—C(12)—C(11)	119·0 (3)	119·0 (3)
N(3)—N(4)—C(4)	115·9 (3)	115·8 (3)	N(1)—C(12)—C(13)	122·0 (3)	122·1 (3)
C(5)—N(6)—C(7)	116·3 (3)	116·1 (3)	C(11)—C(12)—C(13)	118·9 (3)	118·8 (3)
N(1)—C(1)—C(2)	114·7 (4)	114·7 (4)	C(7)—C(13)—C(8)	118·2 (3)	118·8 (3)
O—C(2)—N(3)	123·4 (4)	122·7 (3)	C(7)—C(13)—C(12)	123·3 (3)	122·6 (3)
O—C(2)—C(1)	119·6 (3)	120·6 (4)	C(8)—C(13)—C(12)	118·4 (3)	118·6 (3)
N(3)—C(2)—C(1)	117·0 (4)	116·6 (3)	C(7)—C(1')—C(2')	122·5 (4)	121·5 (3)
N(1)—C(4)—N(4)	125·5 (3)	125·6 (3)	C(7)—C(1')—C(6')	119·2 (3)	121·0 (4)
N(1)—C(4)—C(5)	116·6 (3)	116·9 (3)	C(2')—C(1')—C(6')	118·3 (4)	117·5 (4)
N(4)—C(4)—C(5)	117·8 (3)	117·5 (3)	Cl(2')—C(2')—C(1')	119·8 (4)	119·7 (3)
N(6)—C(5)—C(4)	110·3 (3)	109·7 (3)	Cl(2')—C(2')—C(3')	120·0 (4)	119·3 (4)
N(6)—C(7)—C(13)	126·1 (3)	125·5 (3)	C(1')—C(2')—C(3')	120·2 (5)	120·9 (4)
N(6)—C(7)—C(1')	115·9 (3)	117·7 (3)	C(2')—C(3')—C(4')	119·6 (6)	120·1 (6)
C(13)—C(7)—C(1')	117·9 (3)	116·8 (3)	C(3')—C(4')—C(5')	120·7 (7)	119·4 (6)
C(9)—C(8)—C(13)	121·3 (4)	121·4 (3)	C(4')—C(5')—C(6')	120·3 (7)	121·1 (6)
Cl(9)—C(9)—C(8)	120·2 (3)	119·9 (3)	C(1')—C(6')—C(5')	120·9 (5)	121·0 (5)
Cl(9)—C(9)—C(10)	119·5 (3)	119·9 (3)			

	Molecule A	Molecule B
C(12)—N(1)—C(4)—C(5)	−2·8	−5·7
N(1)—C(4)—C(5)—N(6)	−71·5	−70·0
C(4)—C(5)—N(6)—C(7)	72·5	72·6
C(5)—N(6)—C(7)—C(13)	−0·7	0·1
N(6)—C(7)—C(13)—C(12)	−40·7	−43·5
C(7)—C(13)—C(12)—N(1)	−1·9	−0·4
C(13)—C(12)—N(1)—C(4)	42·3	44·3
C(13)—C(7)—C(1')—C(2')	−72·9	−63·4
N(6)—C(7)—C(1')—C(2')	109·1	119·6

N(1), C(4), N(6), C(7) and the 'bow' and 'stern' planes, consisting of atoms C(4), C(5), N(6) and N(1), C(12), C(13), C(7), respectively. The bow angles of 60·5 (8) and 59·8 (8)° and the stern angles of 33·8 (8) and 35·9 (8)° fall within the ranges found for benzodiazepin-2-ones (58–64 and 32–40°) (Hamor & Martin, 1983), rather than showing some flattening of the bow angle, as has been found for compounds having a five-membered ring fused onto the N(1)—C(2) bond of the parent system, for example 54·9° in 8-chloro-6-(2-chlorophenyl)-2-methyl-4*H*-imidazo[1,2-*a*][1,4]benzodiazepine and 53·4° in 8-chloro-6-(2-chlorophenyl)-1-(4-pyridyl)-[1,2,4]triazolo[4,3-*a*][1,4]benzodiazepine (Kemmish & Hamor, 1989). The deviation of the seven-membered ring from an ideal cycloheptatriene boat with mirror symmetry and zero torsion angles about the three double bonds in terms of the 'deviation parameter', Δ, defined in Hamor & Martin (1983), is 1·8° for molecule A and 2·8° for molecule B. The corresponding values for the two benzodiazepines cited above are similar, 2·1 and 4·6°, respectively.

The phenyl ring is steeply inclined to the plane of the fused benzo moiety in both molecules, interplanar angles 88·9 (8) and 82·0 (8)° respectively. The C(7)—C(1') bond lengths (see Table 2) correspond to single bonds between sp^2-hybridized C atoms, thus excluding the possibility of any significant conjugation between the phenyl ring and the N(6)—C(7) double bond.

Each of the independent molecules is hydrogen bonded to a corresponding centrosymmetrically related molecule. The distances and angles involved are: molecule A N(3)⋯O 2·853 (5), H(3)⋯O 1·94 (4) Å and angle H(3)—N(3)⋯O 10 (2)°; molecule B N(3)⋯N(4) 2·973 (4), H(3)⋯N(4) 2·09 (4) Å and angle H(3)—N(3)⋯N(4) 14 (2)°. Other intermolecular contact distances correspond to normal van der Waals interactions.

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Fumarate de Bis{[(1*H*-benzimidazolyl-1)-3 méthyl-1 propyl][hydroxy-2 (hydroxy-4 méthoxy-3 phényl)-2 éthyl]ammonium} Hexahydrate

PAR P. TOFFOLI ET N. RODIER

Laboratoire de Chimie Minérale, Faculté des Sciences Pharmaceutiques et Biologiques, 5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

R. CEOLIN

Laboratoire de Chimie Physique, Faculté de Pharmacie, 2 bis Boulevard Tonnelé, 37042 Tours CEDEX, France

K. DAO DUONG

Service des Recherches Analytiques, Laboratoires Roger Bellon, 159 Avenue A. Peretti, 92200 Neuilly sur Seine, France

ET M. JOANNIC

Service de Chimie, Rhône-Poulenc Santé, 90 Rue Marcel Bourdarias, 94143 Alfortville CEDEX, France

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Abstract. $2C_{20}H_{26}N_3O_3^+ \cdot C_4H_2O_4^{2-} \cdot 6H_2O$, $M_r = 935 \cdot 05$, triclinic, $P\bar{1}$, $a = 7 \cdot 7568$ (6), $b = 11 \cdot 515$ (1), $c = 14 \cdot 749$ (2) Å, $\alpha = 108 \cdot 604$ (9), $\beta = 103 \cdot 234$ (8), $\gamma = 94 \cdot 758$ (8)°, $V = 1197 \cdot 9$ (5) Å^3, $Z = 1$, $D_x = 1 \cdot 296$ Mg m^{-3}, $\lambda(Cu\ K\alpha) = 1 \cdot 5418$ Å, $\mu = 0 \cdot 787$ mm^{-1}, $F(000) = 500$, $T = 295$ (1) K, $R = 0 \cdot 037$ for 3409 independent reflections. The benzimidazolyl group and the phenyl ring are planar. Their dihedral angle is 83·8 (5)°. The chain linking them is almost planar. The fumarate anion presents $\bar{1}$ symmetry. Its two halves are in different planes, separated by a distance of 0·27 Å. The cations form layers which spread out along the (011) planes. Numerous O—H···O, N—H···O and O—H···N hydrogen bonds are involved in the cohesion of the structure.

Introduction. L'imoxitérol, bronchodilatateur béta-mimétique, est un mélange (70:30) des deux α-{[(1*H*-benzimidazolyl-1)-3 méthyl-1 propyl]aminométhyl} hydroxy-4 méthoxy-3 benzèneméthanols racémiques. L'étude cristallographique du fumarate du constituant minoritaire a été entreprise dans le but de connaître les configurations *R* ou *S* des deux atomes de carbone asymétriques présents dans sa molécule. Elle a eu aussi pour objectif de préciser la géométrie de cette dernière et, ainsi, de faciliter la recherche d'éventuelles relations entre la structure et l'activité pharmacologique.

Partie expérimentale. Cristal parallélépipédique: 0,12 × 0,15 × 0,40 mm. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que: $15,70 \le \theta \le 41,58°$. Diffractomètre Enraf–Nonius CAD-4. $0,023 \le (\sin\theta)/\lambda \le 0,578$ Å^{-1}. $0 \le h \le 8$, $-13 \le k \le 13$, $-16 \le l \le 15$. Quelques réflexions *hkl* dont il était impossible de mesurer les intensités, ont été remplacées par les réflexions équivalentes $\bar{h}k\bar{l}$. Réflexions de contrôle: $\bar{1}32$, $25\bar{3}$ et $41\bar{5}$. Variations non significatives des intensités au cours des mesures. $\sigma(I)/I$ moyen (contrôle): 0,0020. 3866 réflexions indépendantes mesurées, 457 inobservées $[I < 3\sigma(I)]$. Méthodes directes, programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Affinement basé