

Fig. 2. A stereodiagram of the unit-cell contents (Motherwell & Clegg, 1978).

References

- ANDRETTI, G. D., GARBARCZYK, J. & KRÓLIKOWSKA, M. (1981). *Cryst. Struct. Commun.* **10**, 789–792.
 GARBARCZYK, J. (1984). *Makromol. Chem.* **185**, 2033–2041.
International Tables for X-ray Crystallography. (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

- KITAIGORODSKI, A. I. (1976). *Kryształy Molekularne*. Warszawa: PWN.
 LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 RATAJCZAK-SITARZ, M., KATRUSIAK, A., KAŁUSKI, Z. & GARBARCZYK, J. (1987). *Acta Cryst.* **C43**, 2389–2391.
 ROZSONDAI, B., MOORE, J. H., GREGORY, D. C. & HARGITTAI, I. (1977). *Acta Chim. Acad. Sci. Hung.* **94**, 321–331.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 TABOR, B. J., MAGRÉ, E. P. & BOON, J. (1971). *Eur. Polym. J.* **7**, 1127.

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Structure of 1,5-Diazabicyclo[5.4.0]undecan-6-one

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Abstract. $C_9H_{16}N_2O$, $M_r = 168.24$, monoclinic, $P2_1/c$, $a = 12.287$ (2), $b = 5.167$ (1), $c = 15.186$ (2) Å, $\beta = 107.97$ (1)°, $V = 917.09$ (2) Å³, $Z = 4$, $D_x = 1.216$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 6.08$ cm⁻¹, $F(000) = 368$, room temperature, $R = 0.067$ for 1546 reflexions with $I > 3\sigma(I)$. The seven-membered and six-membered rings are in chair conformations. Molecules related by the centre of inversion form hydrogen-bonded dimers, $H \cdots O$ 2.14 (5) Å, $N-H \cdots O$ 132 (2)°. The atoms participating in the hydrogen bond form an eight-membered ring which is almost flat (except for the H atoms).

Introduction. The title compound was obtained first by Paquett & Scott (1968). Recently a more simple and efficient synthesis of this heterocyclic system has been reported (Guryn, 1987). This compound is now used as a substrate in the synthesis of hypotensive drugs which act on the central nervous system (Guryn, Szadowska, Czarnecka & Wejman, 1987).

The present structural work has been undertaken to obtain more detailed information about the bond system and conformation of the 1,5-diazabicycloundecanone rings.

Experimental. Colourless, needle-shaped crystals obtained from acetone, dimensions 0.25 × 0.25 × 0.4 mm; cell parameters and intensity data measured on an Enraf–Nonius CAD-4 diffractometer. Orientation matrix from 25 carefully centred reflexions with $\theta_{\text{max}} = 51^\circ$, graphite-monochromatized $\text{Cu } K\alpha$, radiation range of h , k and l $-15 \rightarrow 15$, $0 \rightarrow 6$, $0 \rightarrow 8$ respectively; total of 2162 independent reflexions measured to $(\sin\theta)/\lambda = 0.61$ Å⁻¹; data not corrected for absorption (controls: orientation matrix and intensity $1\bar{1}5$ with respect to $P2_1/a$), $\mu r = 0.15$; 1546 reflexions with $I > 3\sigma(I)$ used in refinement. Solution by direct methods using *SHELX76* (Sheldrick, 1976); refinement on F in space group $P2_1/c$ by full-matrix least-squares procedure; after initial anisotropic refine-

ment a difference synthesis at $R = 0.12$ revealed positions of all H atoms, in subsequent cycles of refinement H atoms allowed to refine isotropically; max. and min. $\Delta\rho$ in final difference map were 0.24 and -0.23 e \AA^{-3} ; max. $\Delta/\sigma < 0.18$ for both positional and thermal parameters, overall max. $\Delta/\sigma < 0.377$, $S = 0.638$, $R = 0.067$. Unit weights. Atomic scattering factors from *SHELX76*.

Table 1. Final positional and isotropic thermal parameters ($\times 10^4$) with standard deviations in parentheses

	$U_{iso} = \frac{1}{3}\sum U_{ii}$			$U_{iso}(\text{\AA}^2)$
	<i>x</i>	<i>y</i>	<i>z</i>	
C1	2212 (3)	-301 (6)	5029 (2)	595 (19)
C2	1415 (3)	1996 (7)	4992 (3)	681 (21)
C3	639 (3)	2515 (8)	4013 (3)	747 (24)
C4	1329 (3)	2659 (8)	3337 (3)	617 (20)
C5	2371 (3)	-290 (8)	2602 (2)	630 (21)
C6	3307 (3)	1372 (7)	2436 (2)	591 (19)
C7	4419 (3)	1222 (7)	3212 (2)	590 (19)
C8	3765 (2)	2111 (5)	4595 (2)	434 (14)
C9	2844 (3)	-44 (5)	4309 (2)	476 (15)
N1	2009 (2)	286 (5)	3402 (2)	527 (14)
N2	4408 (2)	2615 (5)	4054 (2)	534 (14)
O1	3915 (2)	3331 (4)	5326 (1)	575 (12)

Table 2. Interatomic distances (\AA)

C1 C2	1.529 (5)	C2 C3	1.522 (5)
C3 C4	1.521 (5)	C4 N1	1.469 (4)
N1 C9	1.452 (4)	C9 C1	1.528 (4)
C9 C8	1.551 (4)	C8 N2	1.329 (3)
N2 C7	1.470 (4)	C7 C6	1.506 (5)
C6 C5	1.517 (5)	C5 N1	1.448 (4)
C8 O1	1.240 (3)	O1 H102*	2.13 (5)

* Symmetry-related atoms.

Table 3. Bond angles ($^\circ$)

C1 C2 C3	112.0 (3)	C2 C3 C4	110.8 (3)
C3 C4 N1	109.7 (3)	C4 N1 C9	112.5 (2)
N1 C9 C1	108.8 (3)	C9 C1 C2	111.5 (3)
C4 N1 C5	115.5 (3)	C1 C9 C8	110.5 (2)
N1 C5 C6	117.9 (3)	C5 C6 C7	113.0 (3)
C6 C7 N2	114.0 (3)	C7 N2 C8	127.4 (3)
N2 C8 C9	119.3 (2)	C8 C9 N1	115.0 (2)
C9 N1 C5	117.5 (3)	C9 C8 O1	120.6 (2)
N2 C8 O1	120.1 (2)	C8 O1 H102*	120 (1)
N2 H102 O1*	131 (4)		

* Symmetry-related atoms.

Table 4. Torsion angles ($^\circ$)

C1 C2 C3 C4	-50.2 (4)	C1 C9 N1 C5	-159.9 (3)
C2 C3 C4 N1	55.0 (4)	C1 C9 C8 O1	0.8 (4)
C3 C4 N1 C9	-62.8 (4)	C1 C9 C8 N2	179.8 (3)
C4 N1 C9 C1	62.3 (4)	C2 C1 C9 C8	71.8 (3)
N1 C9 C1 C2	-55.3 (3)	C4 N1 C5 C6	72.4 (4)
C9 C1 C2 C3	50.6 (4)	C4 N1 C9 C8	-62.2 (4)
N1 C5 C6 C7	61.3 (4)	C3 C4 N1 C5	158.5 (3)
C5 C6 C7 N2	-73.6 (4)	N1 C9 C8 O1	124.4 (3)
C6 C7 N2 C8	63.7 (4)	C7 N2 C8 O1	174.2 (3)
C7 N2 C8 C9	-4.7 (4)	O1 C8 N2 H102	34 (3)
N2 C8 C9 N1	-56.6 (4)	C8 N2 H102 O1*	-110 (4)
C8 C9 N1 C5	75.7 (4)	N2 H102 O1* C8*	107 (4)
C9 N1 C5 C6	-64.1 (4)	H102 O1* C8* N2*	-18 (1)

* Symmetry-related atoms.

Discussion. The final positional parameters are listed in Table 1,* bond lengths in Table 2, angles in Table 3 and torsion angles in Table 4. Fig. 1 shows the numbering scheme and Fig. 2 shows the molecules in the unit cell.

The title compound is composed of six- and seven-membered rings fused together. Both rings have the chair conformation (see Table 4). The six-membered ring (C1, C2, C3, C4, N1, C9) possesses an approximate plane of symmetry which is parallel to the C3C4 and C1C9 bonds and which crosses the ring through the C2 and N1 atoms ($\Delta m = 0.4^\circ$). The six-membered ring also possesses an approximate twofold axis which is perpendicular to the symmetry plane and which passes through the middle of the C3C4 and C1C9 bonds ($\Delta_2 = 0.1^\circ$). The seven-membered ring (C5, C6, C7, C8, C9, N1, N2) possesses a quasi symmetry plane only. This plane passes through the C5

* 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 44405 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

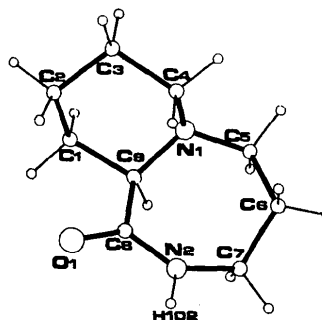


Fig. 1. The atom-numbering scheme.

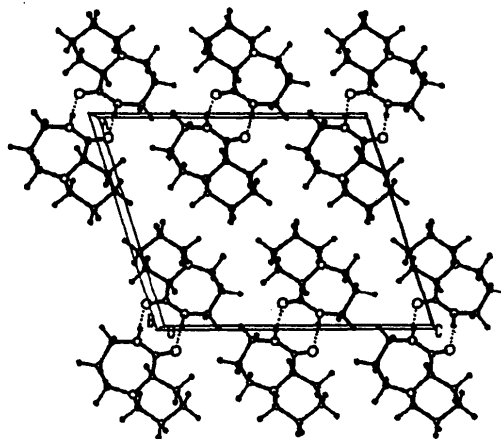


Fig. 2. Arrangement of the molecules in the unit cell.

atom and divides the C8N2 bond into two equal parts ($A_m = 6.4^\circ$).

The neighbouring molecules, related by the centre of inversion, form hydrogen-bonded dimers. The unique hydrogen bond is made between atoms H102 and O1, with a distance H102...O1 of 2.14 (5) Å and an angle N2-H102...O1 of 132 (2)°. The participating atoms (O1, C8, N2, H102 and symmetry-related ones) in the hydrogen bond form an eight-membered ring which is almost flat (except for the H102 atoms).

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Structure du (Méthyl-1 pipérazinyl-4)-5 Pyrido[2,3-*b*][1,5]benzothiazépine

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Abstract. $C_{17}H_{18}N_4S$, $M_r = 310.42$, m.p. = 410 K, triclinic, $P\bar{1}$, $a = 8.249$ (3), $b = 9.999$ (3), $c = 11.029$ (4) Å, $\alpha = 70.26$ (13), $\beta = 76.16$ (19), $\gamma = 70.09$ (21)°, $V = 797.10$ Å³, $Z = 2$, $D_x = 1.293$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 17.02$ cm⁻¹, $F(000) = 328$, $T = 290$ K, final $R = 0.049$ for 1987 observed reflections. The structure was solved by direct methods. The thiazepine ring is in a boat conformation while the piperazine ring is in the normal chair conformation. The dihedral angle between the two aromatic rings is 111°. The cohesion of the crystal is the result of van der Waals interactions.

Introduction. La (méthyl-1 pipérazinyl-4)-5 pyrido[2,3-*b*][1,5]benzothiazépine (Fig. 1) a été brevetée par Hoffmann & Bowley (1979) pour ses propriétés antihistaminiques et orexigéniques. Malgré l'analogie de structure avec des substances comme la clozapine, et surtout la clothiapine, cette pyridobenzothiazépine ne semble pas posséder d'activité psychotrope. Elle s'intègre cependant tout-à-fait dans le travail entrepris au laboratoire sur les neuroleptiques.

Partie expérimentale. Cristallisé dans une solution 50–50% méthanol-acétone. Cristal incolore: 0,45 × 0,65 × 0,53 mm. Paramètres de la maille déterminés à partir de 49 réflexions ($37 \leq \theta \leq 43^\circ$). Diffractomètre Siemens, 2146 réflexions mesurées, $4 \leq \theta \leq 55^\circ$, Cu $K\alpha$ monochromatisée au graphite, balayage ω , 2004

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References

- GURYN, R. (1987). *Pol. J. Chem.* In the press.
 GURYN, R., SZADOWSKA, A., CZARNECKA, E. & WEJMAN, J. (1987). *Acta Pol. Pharm.* In the press.
 PAQUETT, L. A. & SCOTT, M. K. J. (1968). *J. Org. Chem.* **33**, 2379–2384.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

réflexions indépendantes ($\bar{8} \leq h \leq 8$, $\bar{9} \leq k \leq 10$, $0 \leq l \leq 11$), $R_{\text{int}} = 0,012$. Deux réflexions de référence: $2049 \leq F_o(\bar{1}\bar{3}\bar{9}) \leq 2132$ et $3755 \leq F_o(\bar{2}\bar{6}\bar{2}) \leq 3872$. Correction d'absorption par la méthode semi-empirique de North, Phillips & Mathews (1968). Facteurs de transmission minimum et maximum: 0,44 et 0,53. Structure déterminée avec *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Affinement sur F avec *SHELX76* (Sheldrick, 1976). Facteurs de température anisotropes pour tous les atomes non-hydrogène, H placés suivant une

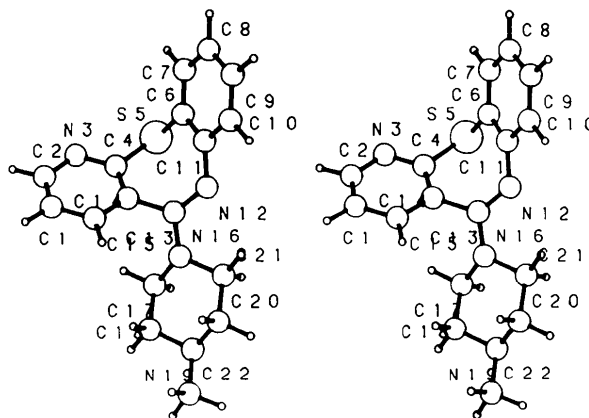


Fig. 1. Vue stéréographique de la molécule avec la numérotation des atomes.