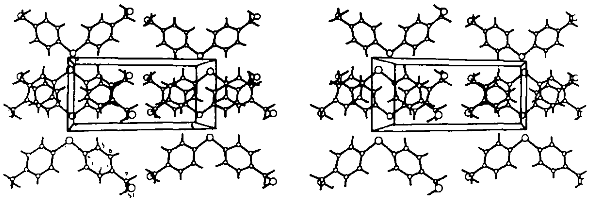

Fig. 2. A stereodiagram of the unit-cell contents (Motherwell & Clegg, 1978).

References

ANDREETTI, 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.* A**30**, 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., KALUSKI, Z. & GARBARCZYK, J. (1987). *Acta Cryst.* C**43**, 2389–2391.

ROZSONDAI, B., MOORE, J. H., GREGORY, D. C. & HARGITTAI, I. (1977). *Acta Chim. Acad. Sci. Hung.* **94**, 321–331.

SHELDRICK, 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.

Acta Cryst. (1988). C**44**, 317–319

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

BY TOMASZ A. OLSZAK, ANDRZEJ M. BRZOZOWSKI, M. J. GRABOWSKI AND A. STĘPIEŃ

Department of Crystallography, Institute of Chemistry, University of Łódź, Nowotki 18, 91-416 *Łódź, Poland*

R. GURYN

Institute of Chemistry and Technology of Drugs, School of Medicine, Narutowicza 120*a*, 90-145 *Łódź, Poland*

AND ZBIGNIEW DAUTER

Department of Biochemistry, Technical University of Gdańsk, Majakowskiego 11/12, 80-952 *Gdańsk, Poland*

(*Received* 28 *October* 1986; *accepted* 28 *September* 1987)

Abstract. $C_9H_{16}N_2O$, $M_r = 168{\cdot}24$, monoclinic, $P2_1/c$, $a = 12{\cdot}287\,(2)$, $b = 5{\cdot}167\,(1)$, $c = 15{\cdot}186\,(2)$ Å, $\beta = 107{\cdot}97\,(1)°$, $V = 917{\cdot}09\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}216$ g cm^{-3}, $\lambda(\text{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 6{\cdot}08$ cm^{-1}, $F(000) = 368$, room temperature, $R = 0{\cdot}067$ 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···O 2·14 (5) Å, N—H···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 $\theta_{\max} = 51°$, graphite-monochromatized Cu $K\alpha$, radiation range of h, k and l −15→15, 0→6, 0→8 respectively; total of 2162 independent reflexions measured to $(\sin\theta)/\lambda = 0{\cdot}61$ Å^{-1}; data not corrected for absorption (controls: orientation matrix and intensity $1\bar{1}5$ with respect to $P2_1/a$), $\mu r = 0{\cdot}15$; 1546 reflexions with $I > 3\sigma(I)$ used in refinement. Solution by direct methods using *SHELX*76 (Sheldrick, 1976); refinement on F in space group $P2_1/c$ by full-matrix least-squares procedure; after initial anisotropic refine-

0108-2701/88/020317-03$03.00

ment a difference synthesis at $R = 0\cdot12$ 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 $-0\cdot23$ e Å^{-3}; max. $\Delta/\sigma < 0\cdot18$ for both positional and thermal parameters, overall max. $\Delta/\sigma < 0\cdot377$, $S = 0\cdot638$, $R = 0\cdot067$. Unit weights. Atomic scattering factors from *SHELX*76.

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

$U_{\text{iso}} = \frac{1}{3}\sum U_{ii}$.

	x	y	z	U_{iso}(Å^2)
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* (Å)

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* (°)

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* (°)

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 positonal 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 ($\Delta m = 0\cdot4°$). 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 ($\Delta_2 = 0\cdot1°$). The seven-membered ring (C5,C6,C7,C8,C9,N1,N2) possesses a quasi symmetry plane only. This plane passes through the C5

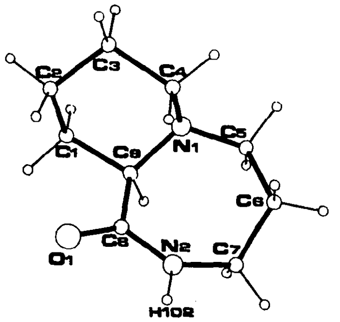

Fig. 1. The atom-numbering scheme.

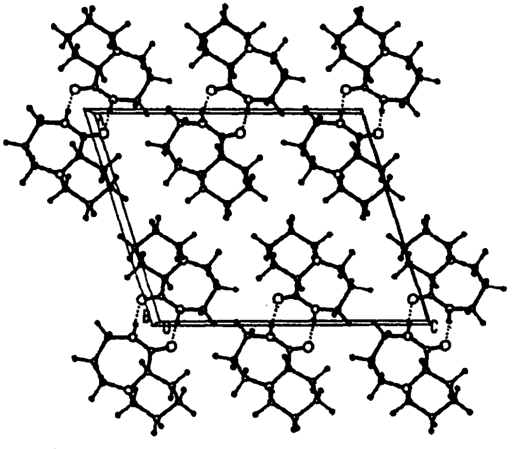

Fig. 2. Arrangement of the molecules in the unit cell.

---

* 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.

atom and divides the C8N2 bond into two equal parts ($\Delta_m = 6{\cdot}4°$).

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).

This work was supported by Project RP.II.10 from the Polish Ministry of Science and Higher Education.

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.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 319–321

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

PAR M. SBIT, L. DUPONT ET O. DIDEBERG

*Laboratoire de Cristallographie, Institut de Physique B5, Université de Liège au Sart Tilman, B-*4000 *Liège, Belgique*

ET J. F. LIÉGEOIS ET J. DELARGE

*Laboratoire de Chimie Pharmaceutique, Institut de Pharmacie F*1, *Université de Liège, rue Fusch* 3–5, *B-*4000 *Liège, Belgique*

(*Reçu le* 10 *février* 1987, *accepté le* 9 *octobre* 1987)

Abstract. $C_{17}H_{18}N_4S$, $M_r = 310{\cdot}42$, m.p. = 410 K, triclinic, $P\bar{1}$, $a = 8{\cdot}249\,(3)$, $b = 9{\cdot}999\,(3)$, $c = 11{\cdot}029\,(4)$ Å, $\alpha = 70{\cdot}26\,(13)$, $\beta = 76{\cdot}16\,(19)$, $\gamma = 70{\cdot}09\,(21)°$, $V = 797{\cdot}10$ Å^3, $Z = 2$, $D_x = 1{\cdot}293$ g cm^{-3}, $\lambda(\text{Cu}\,K\bar{\alpha}) = 1{\cdot}5418$ Å, $\mu = 17{\cdot}02$ cm^{-1}, $F(000) = 328$, $T = 290$ K, final $R = 0{\cdot}049$ 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 pyridobenzothiazepine 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 ($37 \le \theta \le 43°$). Diffractomètre Siemens, 2146 réflexions mesurées, $4 \le \theta \le 55°$, Cu $K\bar{\alpha}$ monochromatisée au graphite, balayage ω, 2004 réflexions indépendantes ($\bar{8} \le h \le 8$, $\bar{9} \le k \le 10$, $0 \le l \le 11$), $R_{\text{int}} = 0{,}012$. Deux réflexions de référence: $2049 \le F_o(\bar{1}\bar{3}\bar{9}) \le 2132$ et $3755 \le F_o(\bar{2}6\bar{2}) \le 3872$. 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 *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Affinement sur *F* avec *SHELX*76 (Sheldrick, 1976). Facteurs de température anisotropes pour tous les atomes non-hydrogène, H placés suivant une

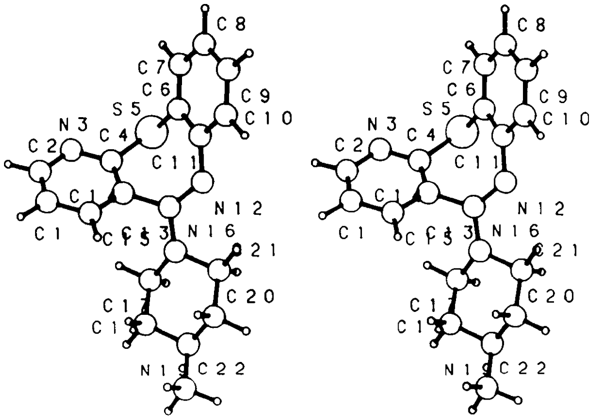

Fig. 1. Vue stéréographique de la molécule avec la numérotation des atomes.

0108-2701/88/020319-03$03.00