D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O1W	0.62 (5)	2.22 (5)	2.821 (2)	162 (6)
O1W—H2OW···O2	0.87 (7)	2.25 (7)	2.821 (2)	123 (6)
O1—H1O···O1W^{i}	0.83 (4)	1.91 (4)	2.735 (2)	173 (4)
O1W—H1OW···O5^{ii}	0.84 (3)	2.43 (2)	3.156 (2)	146 (2)
O1W—H1OW···O3^{ii}	0.84 (3)	2.50 (3)	3.194 (2)	141 (2)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $x, 1 - y, z - \frac{1}{2}$.

Table 5. *Conformation of the rings and dihedral angles (°) between the ring planes*

Q_T = total puckering amplitude (Å) (Cremer & Pople, 1975), DAP = minimum displacement asymmetry parameter (Nardelli, 1983*b*), BC = boat-chair, T = twist, S = sofa. *A*, *B* and *C* denote the six-, four- and eight-membered rings, respectively.

	Q_T	DAP	Conformation
Compound (2*a*)			
A	0.731 (2)	*D*2(C3) = 0.0461 (8)	T
B	0.0503 (5)		
C	1.205 (2)	*DS*(C8) = 0.0867 (8)	BC
Compound (2*b*)			
A	0.512 (2)	*DS*(C1) = 0.0274 (9)	S
B	−0.0695 (3)		
C	1.192 (2)	*DS*(C8) = 0.0468 (7)	BC

Dihedral angles	*A*/*B*	*B*/*C*	*A*/*C*
(2*a*)	135.3 (1)	114.0 (1)	155.6 (1)
(2*b*)	114.7 (1)	126.4 (1)	147.5 (1)

The integrated intensities were obtained by a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) peak-profile analysis procedure.

Both structures were solved by direct methods and refined by anisotropic full-matrix least squares. All the H atoms were found from the $\Delta\rho$ maps and refined isotropically. The H2O*W* atom of compound (2*b*) was found too near the O1*W* atom [0.62 (5) Å], so its position was corrected by shifting it along the H2O*W*—O1*W* direction at an ZO1*W*—H2O*W* distance equal to that found for O1*W*—H1O*W*.

The calculations were carried out on the ENCORE91 and GOULD–POWERNODE 6040 computers of the Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma), and on a COMPAQ-486c portable computer.

For both compounds, data collection: local programs; cell refinement: *LQPARM* (Nardelli & Mangia, 1984); data reduction: local programs. Program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990) for (2*a*); *SIR*88 (Burla, *et al.*, 1988) for (2*b*). For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP* (Johnson, 1965); *PLUTO* (Motherwell & Clegg, 1976); software used to prepare material for publication: *PARST* (Nardelli, 1983); *PARSTCIF* (Nardelli, 1991).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1167). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 1889–1891

3-Methyl-6-(4-methylpiperazin-1-yl)-11*H*-pyrido[2,3-*b*][1,4]benzodiazepine

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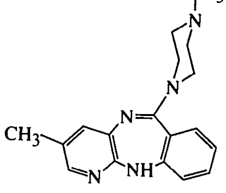
Abstract

The synthesis and crystal structure of $C_{18}H_{21}N_5$ has been undertaken as part of a study of dopamine receptors. The diazepine ring has a boat conformation. The four C atoms of the outer ring junctions are almost coplanar; the maximum deviation from their mean plane

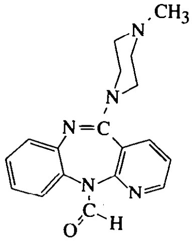
is 0.017 (1) Å. The benzene and pyridine rings lie on the same side of the diazepine ring. There is one N—H···N hydrogen bond [N···N 3.048 (2) Å].

Comment

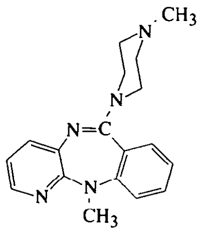
The molecular structure of the title compound, (I), may be compared, for example, with that of 11-formyl-5-(4-methylpiperazin-1-yl)-11*H*-pyrido[2,3-*b*][1,5]benzodiazepine, (II), and 6-(4-methylpiperazin-1-yl)-11-methyl-11*H*-pyrido[2,3-*b*][1,4]benzodiazepine, (III) (Dupont, Englebert, Dideberg, Liégeois & Delarge, 1991).



(I)



(II)



(III)

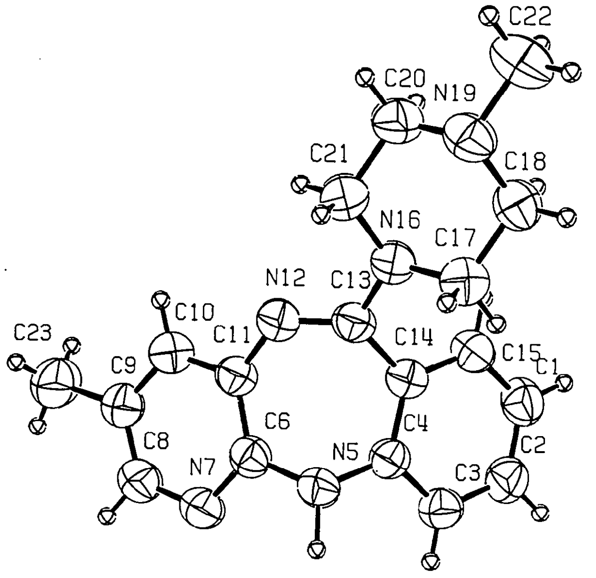


Fig. 1. *ORTEP*II plot (Johnson, 1976) of the title compound showing the molecular structure and the atom-labelling scheme. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

The diazepine ring has a boat conformation, where N5 is the 'prow' and C12 and C13 constitute the 'stern'. The piperazine ring has the normal chair conformation. The dihedral angle between the benzene and pyridine rings is 119.41 (7)° [this angle is 115.1 (1) and 115.0 (1)° in compounds (II) and (III), respectively]. The distances between the methylpiperazine N atom and the centres of the two aromatic rings are 7.727 (4) and 6.084 (4) Å [7.768 (4) and 6.107 (4) in (II); 7.726 (4) and 5.934 (4) Å in (III)]. The cohesion of the crystal is the result of both van der Waals interactions and one hydrogen bond, N5—H5···N7^{i} [N5···N7^{i} 3.048 (2), H5···N7^{i} 2.01 Å, N5—H5···N7^{i} 174°; symmetry code: (i) $1-x$, $2-y$, $-z$]. Analogues of the title compound will be the subject of future investigations.

Experimental

The compound was synthesized according to Liégeois *et al.* (1993) at the laboratory of Medicinal Chemistry of Liège. Crystals were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_{18}H_{21}N_5$
$M_r = 307.40$
Monoclinic
$P2_1/c$
$a = 9.3352\ (6)$ Å
$b = 19.2342\ (10)$ Å
$c = 9.3903\ (7)$ Å
$\beta = 93.039\ (6)°$
$V = 1683.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.213$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 48 reflections
$\theta = 37.15–41.59°$
$\mu = 0.593$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.53 × 0.49 × 0.30 mm
Colourless

Data collection

Stoe Siemens AED four-circle diffractometer
ω scans
Absorption correction: ψ scan (*EMPIR*; Stoe & Cie, 1987*b*)
$T_{\min} = 0.738$, $T_{\max} = 0.826$
2594 measured reflections
2426 independent reflections

1399 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0208$
$\theta_{\max} = 58.94°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 10$
2 standard reflections frequency: 60 min intensity decay: 2.5%

Refinement

Refinement on F^2
$R(F) = 0.0385$
$wR(F^2) = 0.1043$
$S = 0.897$
2426 reflections
211 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0059 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.2864 (2)	1.23216 (11)	0.2886 (2)	0.0680 (6)
C2	0.2286 (2)	1.18559 (11)	0.1892 (2)	0.0690 (6)
C3	0.3164 (2)	1.13997 (10)	0.1212 (2)	0.0611 (6)
C4	0.4633 (2)	1.14094 (9)	0.1501 (2)	0.0515 (5)
N5	0.5529 (2)	1.09245 (8)	0.08518 (15)	0.0564 (4)
C6	0.6304 (2)	1.04793 (10)	0.1816 (2)	0.0537 (5)
N7	0.6100 (2)	0.98021 (8)	0.1622 (2)	0.0631 (5)
C8	0.6777 (2)	0.93674 (11)	0.2541 (2)	0.0662 (6)
C9	0.7642 (2)	0.95668 (11)	0.3686 (2)	0.0629 (6)
C10	0.7889 (2)	1.02784 (11)	0.3826 (2)	0.0638 (6)
C11	0.7247 (2)	1.07516 (10)	0.2889 (2)	0.0553 (5)
N12	0.7701 (2)	1.14488 (8)	0.2929 (2)	0.0596 (5)
C13	0.6826 (2)	1.19583 (10)	0.2683 (2)	0.0554 (5)
C14	0.5234 (2)	1.19065 (9)	0.2446 (2)	0.0522 (5)
C15	0.4332 (2)	1.23465 (10)	0.3151 (2)	0.0607 (5)
N16	0.7359 (2)	1.26292 (8)	0.2752 (2)	0.0609 (5)
C17	0.6834 (2)	1.31582 (10)	0.1737 (2)	0.0636 (6)
C18	0.7088 (2)	1.38712 (10)	0.2354 (2)	0.0667 (6)
N19	0.8612 (2)	1.39806 (9)	0.2711 (2)	0.0693 (5)
C20	0.9124 (2)	1.34522 (11)	0.3719 (3)	0.0746 (6)
C21	0.8880 (2)	1.27274 (11)	0.3131 (3)	0.0733 (7)
C22	0.8875 (3)	1.46760 (11)	0.3304 (3)	0.0940 (8)
C23	0.8316 (3)	0.90336 (11)	0.4691 (3)	0.0923 (8)

Table 2. *Selected geometric parameters* (Å, °)

C4—C14	1.401 (2)	N7—C8	1.336 (2)
C4—N5	1.413 (2)	C11—N12	1.406 (2)
N5—C6	1.417 (2)	N12—C13	1.289 (2)
C6—N7	1.328 (2)	C13—N16	1.383 (2)
C6—C11	1.404 (2)	C13—C14	1.494 (3)
C14—C4—N5	119.8 (2)	C13—N12—C11	122.2 (2)
C4—N5—C6	114.66 (14)	N12—C13—N16	118.6 (2)
C11—C6—N5	120.9 (2)	N12—C13—C14	126.3 (2)
C6—N7—C8	117.8 (2)	N16—C13—C14	114.9 (2)
C6—C11—N12	123.5 (2)	C4—C14—C13	120.1 (2)
C3—C4—N5—C6	−118.4 (2)	C11—N12—C13—C14	5.4 (3)
C14—C4—N5—C6	61.7 (2)	N5—C4—C14—C13	4.6 (3)
C4—N5—C6—N7	122.6 (2)	N16—C13—C14—C15	−41.6 (3)
C4—N5—C6—C11	−58.5 (2)	N12—C13—C14—C4	−47.3 (3)
C9—C10—C11—N12	−169.8 (2)	N16—C13—C14—C4	138.3 (2)
N7—C6—C11—N12	166.4 (2)	N12—C13—N16—C17	141.4 (2)
N5—C6—C11—N12	−12.5 (3)	C14—C13—N16—C17	−43.8 (3)
C10—C11—N12—C13	−145.0 (2)	C13—N16—C17—C18	157.0 (2)
C6—C11—N12—C13	44.1 (3)		

H atoms were placed at standard calculated positions, except for atom HN5, which was obtained from a difference map. In the refinement, H atoms were constrained (included as riding atoms), except for HN5, which was kept fixed.

Data collection: *DIF*4 (Stoe & Cie, 1987*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1987*c*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1002). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Methoxy-5,6-methylenedioxy-3-(3,4,5-trimethoxyphenyl)-1,3-dihydroisobenzofuran

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Abstract

The crystal structure of the title compound consists of discrete diastereoisomeric molecules of $C_{19}H_{20}O_7$ held together by van der Waals interactions. The two H atoms on the asymmetric C atoms of the hydrofuran ring are mutually *cis*. The orientations of the three methoxy groups on the phenyl ring are consistent with minimum steric interactions.

Comment

The title compound, (2), may be prepared either by the acid-catalysed (Keay, Plaumann, Rajapaksa & Rodrigo, 1983) or by the thermally induced (Ward, Pelter, Galletti & Qianrong, 1993) elimination of methanol from the corresponding hydroxyacetal (1).

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