solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SE1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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11-(4-Methylpiperazin-1-yl)-5*H*-pyrido-[4,3-*b*][1,5]benzodiazepine

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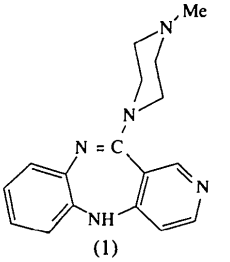
Abstract

The determination of the crystal structure of the title compound, $C_{17}H_{19}N_5$, has been undertaken as part of our studies of dopamine receptors. The diazepine ring is in a boat conformation. The dihedral angle between the two aromatic rings is 125.35 (6)°. The distances between the methylpiperazine N atom and the centres of the two aromatic rings are 5.999 (4) and 7.712 (4) Å. There is no hydrogen bond.

Comment

The title compound, (1), was prepared as part of our study of dopamine receptors. The structures of the related compounds 11-formyl-5-(4-methylpiperazin-1-yl)-11*H*-pyrido[2,3-*b*][1,5]benzodiazepine and of 6-(4-methylpiperazin-1-yl)-11-methyl-11*H*-pyrido[2,3-*b*]-[1,4]benzodiazepine (Dupont, Englebert, Dideberg, Liégeois & Delarge, 1991) have been reported previously. Other new analogous compounds are under investigation.



(1)

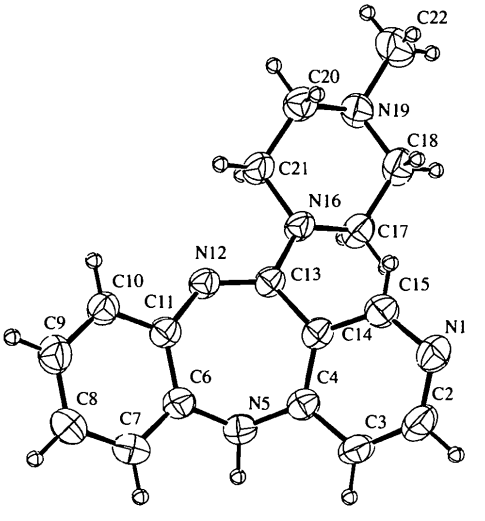


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

Crystal data

$C_{17}H_{19}N_5$
$M_r = 293.37$

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å

Orthorhombic
Pcab
a = 9.8098 (5) Å
b = 16.5788 (10) Å
c = 19.1759 (8) Å
V = 3118.7 (3) Å^3
Z = 8
D_x = 1.250 Mg m^{-3}

Cell parameters from 48 reflections
θ = 31.5–38.61°
μ = 0.617 mm^{-1}
T = 293 (2) K
Prismatic
0.53 × 0.38 × 0.19 mm
Colourless
Crystal source: Laboratory of Medicinal Chemistry, Liège

Data collection

Stoe Siemens AED four-circle diffractometer
ω scans
Absorption correction: ψ scan
T_{min} = 0.75, T_{max} = 0.85
2134 measured reflections
2134 independent reflections

1636 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 57.58°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 20$
2 standard reflections
frequency: 60 min
intensity decay: 2.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0370
$wR(F^2)$ = 0.1027
S = 1.224
2131 reflections
203 parameters
H atoms treated using a riding model except H(N5) which was fixed
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.1626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.148 e Å^{-3}
$\Delta\rho_{min}$ = −0.162 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0072 (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)

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

	x	y	z	U_{eq}
N1	−0.2308 (2)	0.94798 (10)	0.61831 (8)	0.0515 (5)
C2	−0.3149 (2)	0.88699 (12)	0.60314 (10)	0.0523 (5)
C3	−0.2852 (2)	0.82595 (12)	0.55769 (9)	0.0471 (5)
C4	−0.1576 (2)	0.82361 (10)	0.52549 (9)	0.0393 (4)
N5	−0.12575 (15)	0.76411 (8)	0.47642 (7)	0.0459 (4)
C6	−0.0789 (2)	0.79057 (10)	0.40982 (9)	0.0412 (4)
C7	−0.1474 (2)	0.76692 (13)	0.35018 (10)	0.0577 (6)
C8	−0.1055 (2)	0.79119 (13)	0.28503 (10)	0.0617 (6)
C9	0.0059 (2)	0.84118 (12)	0.27891 (10)	0.0531 (5)
C10	0.0754 (2)	0.86488 (10)	0.33757 (8)	0.0415 (4)
C11	0.0362 (2)	0.83957 (10)	0.40392 (8)	0.0373 (4)
N12	0.12394 (13)	0.85947 (8)	0.45971 (7)	0.0391 (4)
C13	0.0811 (2)	0.88215 (10)	0.52015 (8)	0.0360 (4)
C14	−0.0635 (2)	0.88327 (10)	0.54433 (9)	0.0371 (4)
C15	−0.1080 (2)	0.94425 (11)	0.58869 (9)	0.0429 (5)
N16	0.17548 (14)	0.91386 (9)	0.56580 (7)	0.0416 (4)
C17	0.1763 (2)	0.89719 (12)	0.64087 (9)	0.0471 (5)
C18	0.2341 (2)	0.96830 (13)	0.67948 (10)	0.0512 (5)
N19	0.37189 (14)	0.98554 (9)	0.65519 (8)	0.0471 (4)
C20	0.3679 (2)	1.00373 (12)	0.58129 (10)	0.0500 (5)
C21	0.3116 (2)	0.93342 (12)	0.54006 (9)	0.0479 (5)
C22	0.4337 (2)	1.0518 (2)	0.69402 (11)	0.0732 (7)

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

N1—C15	1.334 (2)	C11—N12	1.412 (2)
N1—C2	1.337 (2)	N12—C13	1.289 (2)
C2—C3	1.367 (3)	C13—N16	1.378 (2)
C3—C4	1.396 (2)	C13—C14	1.493 (2)
C4—N5	1.399 (2)	C14—C15	1.391 (2)
C4—C14	1.400 (2)	N16—C21	1.460 (2)
N5—C6	1.426 (2)	N16—C17	1.466 (2)
C6—C7	1.383 (3)	C17—C18	1.503 (3)
C6—C11	1.395 (2)	C18—N19	1.458 (2)
C7—C8	1.376 (3)	N19—C20	1.449 (2)
C8—C9	1.376 (3)	N19—C22	1.459 (3)
C9—C10	1.373 (3)	C20—C21	1.513 (3)
C10—C11	1.394 (2)		
C15—N1—C2	115.5 (2)	N12—C13—N16	117.63 (14)
N1—C2—C3	124.5 (2)	N12—C13—C14	126.34 (15)
C2—C3—C4	119.6 (2)	N16—C13—C14	115.86 (14)
C3—C4—N5	121.2 (2)	C15—C14—C4	117.7 (2)
C3—C4—C14	117.2 (2)	C15—C14—C13	119.8 (2)
N5—C4—C14	121.6 (2)	C4—C14—C13	122.5 (2)
C4—N5—C6	117.22 (14)	N1—C15—C14	125.2 (2)
C7—C6—C11	119.4 (2)	C13—N16—C21	118.95 (13)
C7—C6—N5	119.8 (2)	C13—N16—C17	123.74 (14)
C11—C6—N5	120.81 (15)	C21—N16—C17	111.66 (13)
C8—C7—C6	121.5 (2)	N16—C17—C18	109.7 (2)
C9—C8—C7	119.4 (2)	N19—C18—C17	110.3 (2)
C8—C9—C10	119.8 (2)	C20—N19—C18	109.17 (13)
C9—C10—C11	121.6 (2)	C20—N19—C22	110.7 (2)
C10—C11—C6	118.2 (2)	C18—N19—C22	111.7 (2)
C10—C11—N12	116.91 (15)	N19—C20—C21	111.1 (2)
C6—C11—N12	124.60 (15)	N16—C21—C20	109.19 (15)
C13—N12—C11	123.41 (14)		
C3—C4—N5—C6	125.3 (2)	C11—N12—C13—C14	6.8 (3)
C14—C4—N5—C6	−54.6 (2)	N5—C4—C14—C13	−7.3 (2)
C4—N5—C6—C7	−123.0 (2)	N12—C13—C14—C15	−144.6 (2)
C4—N5—C6—C11	57.6 (2)	N16—C13—C14—C15	30.6 (2)
N5—C6—C11—N12	7.6 (3)	N12—C13—C14—C4	37.1 (2)
C10—C11—N12—C13	140.4 (2)	N16—C13—C14—C4	−147.7 (2)
C6—C11—N12—C13	−45.8 (2)	N12—C13—N16—C21	10.7 (2)
C11—N12—C13—N16	−168.36 (15)	N12—C13—N16—C17	−140.5 (2)

Data collection: *DIF*4 (Stoe & Cie, 1987*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1987*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). 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, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: PA1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stoe & Cie (1987*a*). *DIF4. Diffractometer Control Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1987*b*). *REDU4. Data Reduction Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.

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2-Aminoanthraquinone

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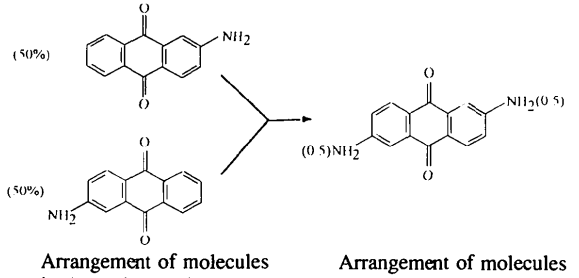
Abstract

The molecule of 2-aminoanthraquinone, $C_{14}H_9NO_2$, is nearly planar, with the non-H atoms exhibiting a mean distance of 0.022 Å from their best plane. The statistical disorder of the 2-aminoanthraquinone molecules is located around the centre of symmetry in space group $P2_1/c$. Weak intermolecular hydrogen bonds (N—H···N and N—H···O) link the molecules into a three-dimensional network.

Comment

This paper reports on the statistically disordered structure of 2-aminoanthraquinone. The planar molecules possess a centre of symmetry and have an occupancy factor of 0.5 for the randomly disordered NH_2 groups [atom H(2) is also disordered]. This accounts for the C—N bond distance of 1.222 (9) Å which is about 0.1 Å shorter than the C—N distances in other diaminoanthraquinone derivatives (Bailey & Brown, 1967*a*,*b*; Brown & Mitchell, 1982; Chippendalle, Mathias, Aujla, Harris, Packer & Say, 1983; Kashino, Senoo & Haisa, 1988). One type of molecular disorder is presented in the scheme below. The C—C and C=O bond distances are comparable to those observed in non-substituted anthraquinone (Lonsdale, Milledge & Sayed, 1966; Lonsdale, Walley & Sayed, 1966; Lonsdale, 1966; Murty, 1960; Prakash, 1967).



Arrangement of molecules in the real crystals — Arrangement of molecules resulting from X-ray analysis

(I)

The crystal structure consists of the two parallel sheets of planar 2-aminoanthraquinone molecules (Fig. 2). The distance between two successive parallel planes is 3.488 (6) Å, which is slightly longer than the van der Waals distance (3.4 Å) for aromatic C atoms (Pauling, 1960). The angle between the planes of two neighbouring sheets is 56.2 (5)°. The shortest intermolecular contacts between N and H, and O and H atoms are 2.36 (7)

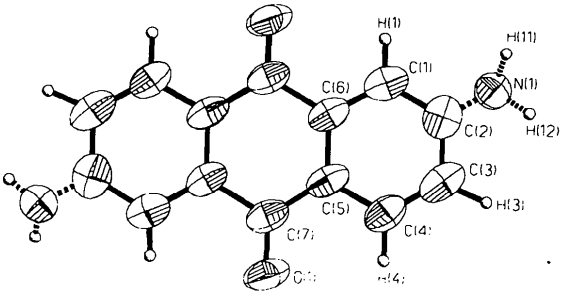


Fig. 1. View of the title compound showing the numbering scheme with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radii. Atom H(2) has been omitted for clarity.

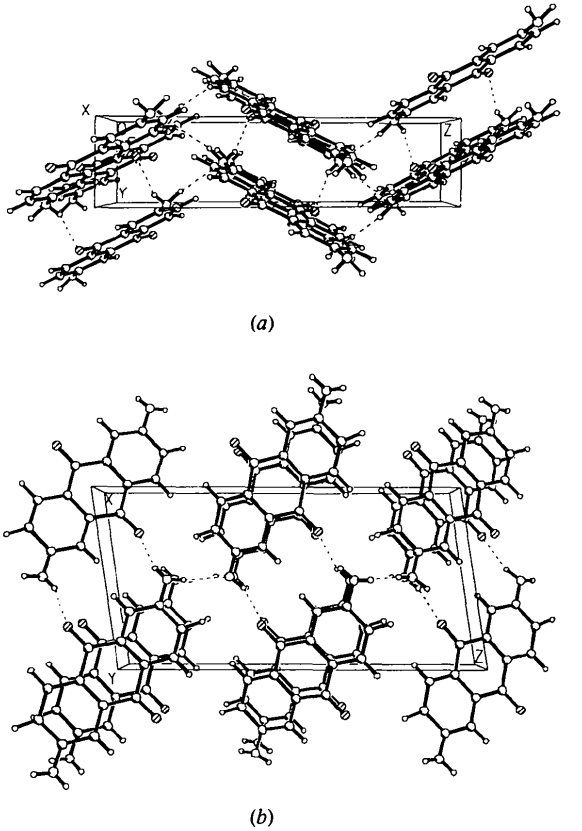


Fig. 2. Packing of the molecules in the unit cell shown by (*a*) a *bc* projection and (*b*) an *ac* projection.