Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: empirical (*SHELXTL*; Sheldrick, 1984)
$T_{\min} = 0.912$, $T_{\max} = 0.968$
4631 measured reflections
2039 independent reflections (plus 2592 Friedel-related reflections)
3456 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25°$
$h = -10 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 14$
3 standard reflections every 297 reflections intensity decay: 8.60%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.181$
$S = 0.998$
4631 reflections
528 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1164P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.011$
$\Delta\rho_{\max} = 0.429$ e Å^{-3}
$\Delta\rho_{\min} = -0.284$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.2 (2)

Table 1. *Selected geometric parameters (Å, °)*

P1—O7	1.473 (6)	P1′—O7′	1.477 (7)
P1—O1	1.574 (6)	P1′—O1′	1.565 (8)
P1—C31	1.782 (5)	P1′—C21′	1.801 (5)
P1—C21	1.805 (4)	P1′—C31′	1.803 (5)
O1—C1	1.443 (9)	O1′—C1′	1.450 (11)
O7—P1—O1	115.2 (3)	O7′—P1′—O1′	114.8 (3)
O7—P1—C31	113.9 (3)	O7′—P1′—C21′	114.7 (3)
O1—P1—C31	105.0 (2)	O1′—P1′—C21′	99.0 (2)
O7—P1—C21	115.6 (3)	O7′—P1′—C31′	110.6 (3)
O1—P1—C21	99.6 (3)	O1′—P1′—C31′	110.0 (2)
C31—P1—C21	106.1 (3)	C21′—P1′—C31′	107.1 (3)
C1—O1—P1	127.2 (5)	C1′—O1′—P1′	133.7 (7)
O1—C1—C6	110.9 (7)	O1′—C1′—C6′	114.5 (3)
O7—P1—O1—C1	−25.0 (8)	O7′—P1′—O1′—C1′	81.2 (3)
C31—P1—O1—C1	101.1 (7)	C21′—P1′—O1′—C1′	−156.2 (3)
C21—P1—O1—C1	−149.3 (7)	C31′—P1′—O1′—C1′	−44.2 (2)
P1—O1—C1—C6	−77.4 (8)	P1′—O1′—C1′—C6′	−42.9 (4)
P1—O1—C1—C2	164.3 (5)	P1′—O1′—C1′—C2′	−163.4 (3)
C7—O5—C5—C4	−136.2 (7)	C7′—O5′—C5′—C4′	−110.7 (7)
C7—O5—C5—C6	−13.5 (8)	C7′—O5′—C5′—C6′	10.2 (8)
O1—C1—C6—O6	78.7 (8)	O1′—C1′—C6′—O6′	93.2 (6)
C2—C1—C6—O6	−164.1 (6)	C2′—C1′—C6′—O6′	−148.2 (6)
C2—C1—C6—C5	−45.4 (9)	C2′—C1′—C6′—C5′	−31.6 (8)
O5—C5—C6—O6	−8.4 (8)	O5′—C5′—C6′—O6′	−28.3 (8)
C4—C5—C6—O6	111.1 (8)	C4′—C5′—C6′—O6′	91.4 (8)
O5—C5—C6—C1	−131.3 (7)	O5′—C5′—C6′—C1′	−149.8 (6)
C4—C5—C6—C1	−11.8 (10)	C4′—C5′—C6′—C1′	−30.1 (10)
O1—P1—C21—C26	16.3 (4)	O1′—P1′—C21′—C26′	−9.3 (3)

The phenyl rings were constrained to be regular hexagons (C—C 1.39 Å). Methyl, tertiary carbon, phenyl and hydroxyl H atoms were constrained to calculated positions (C—H = 0.98, 1.00, 0.95 and O—H = 0.84 Å, respectively). All H-atom isotropic displacement parameters were set at 1.2 times the equivalent isotropic displacement parameter of their parent atom.

Data collection: *XSCANS* (Siemens, 1993). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. 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) and *ORTEP*-3 (Farrugia, 1997). Software used to prepare material for publication: *CIFTAB* in *SHELXL*93 and *PLATON*98 (Spek, 1998).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1226). Services for accessing these data are described at the back of the journal.

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1-Acetyl-1,2,3,4-tetrahydro-4-methyl-2,4-diphenyl-5*H*-1,5-benzodiazepine

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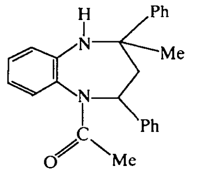
Abstract

The single-crystal X-ray diffraction study of the title compound, $C_{24}H_{24}N_2O$, confirms the boat conformation of the benzodiazepine ring. The equatorial and axial orientations of the phenyl and methyl groups, respectively, are also confirmed. The *N*-acetyl group is non-coplanar with the fused benzene ring. There are two independent molecules in the asymmetric unit.

Comment

The configuration and conformation of molecules containing a benzodiazepine ring are of interest to the understanding of their drug action. The structure of the title compound, (I), has been determined to assign the molecular conformation and the orientation of the substituents in the tetrahydrobenzodiazepine ring.



(I)

There are two independent molecules in the asymmetric unit. The seven-membered ring has a boat conformation similar to other related compounds (Xu *et al.*, 1998), with a fold (Camerman & Camerman, 1972) between C3(C3*A*) and C11(C11*A*) (Fig. 1). This is also supported by the torsion angles around the bonds in the seven-membered ring which are similar to the values for the boat form of methylcycloheptane (Hendrickson, 1967). However, the molecules do not have C_s symmetry. The average bond angles around N1 [120.0 (2)°] and N1*A* [119.9 (2)°] indicate considerable flattening of the N atoms in the seven-membered ring in each of the two molecules. The C=O distance in both the molecules are comparable to the average C=O distance [1.234 (12) Å] reported for substituted amides (Allen *et al.*, 1987) leading to the deviation from the ideal boat conformation of the seven-membered ring. The torsion angles, C2—C3—C4—C24 49.4 (3) and C2*A*—C3*A*—C4*A*—C24*A* −51.4 (3)° indicate the axial orientation of the methyl group in each of the molecules. The equatorial orientations of the two phenyl groups in both of the molecules are evident from the corresponding torsion angles, C2—C3—C4—C18 173.2 (2), C4—C3—C2—C12 176.7 (2), C2*A*—C3*A*—C4*A*—C18*A* −173.9 (2), C4*A*—C3*A*—C2*A*—C12*A* −172.7 (2)°. The non-coplanarity between the fused benzene ring and the *N*-acetyl part of the molecule is revealed by the dihedral angle between them [73.4 (9) and 82.7 (8)°, respectively]. Calculations indicate possible hydrogen bonds involving C2, C2*A* and C24*A* on the one hand and O1 and O1*A* on the other.

Experimental

The title compound was obtained by the acetylation of the parent diamine using a calculated amount of CH_3COCl and an excess of triethylamine. Diffraction quality crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{24}H_{24}N_2O$
M_r = 356.45
Orthorhombic
*Pna*2$_1$
a = 20.999 (1) Å
b = 14.3970 (5) Å
c = 12.648 (4) Å
V = 3823.8 (12) Å^3
Z = 8
D_x = 1.238 Mg m^{-3}
D_m not measured

Mo *Kα* radiation
λ = 0.71069 Å
Cell parameters from 80 reflections
θ = 1.71–24.10°
μ = 0.076 mm^{-1}
T = 173 (2) K
Cubic
0.29 × 0.29 × 0.29 mm
Colourless

Data collection

Rigaku RU300 R-AXIS IP diffractometer
ω–2θ scans
Absorption correction: none
20774 measured reflections
5754 independent reflections
5538 reflections with $I > 2\sigma(I)$

R_{int} = 0.085
θ_{max} = 24.10°
$h = -24 \rightarrow 24$
$k = -16 \rightarrow 16$
$l = -14 \rightarrow 13$
3 standard reflections every 150 reflections intensity decay: negligible

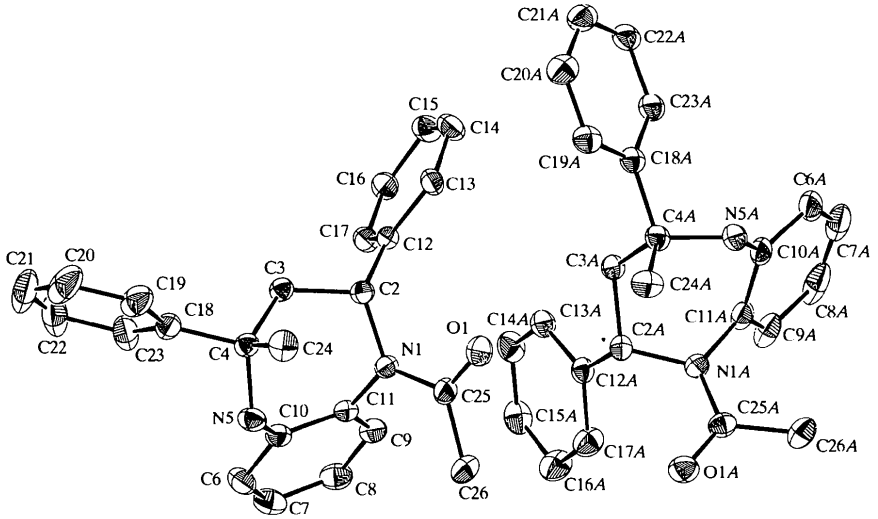


Fig. 1. *ZORTEP* (Zsolnai, 1997) plot of the two independent molecules with 30% probability displacement ellipsoids. The H atoms are omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.125$
$S = 1.222$
5754 reflections
502 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.650$ e Å^{-3}
$\Delta\rho_{\min} = -0.478$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.113 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C11—N1—C2—C3	39.2 (3)
N1—C2—C3—C4	50.4 (3)
C2—C3—C4—N5	−69.9 (2)
C3—C4—N5—C10	−5.7 (3)
C4—N5—C10—C11	53.7 (3)
N5—C10—C11—N1	−2.2 (3)
C2—N1—C11—C10	−69.4 (3)
C11*A*—N1*A*—C2*A*—C3*A*	−41.9 (3)
N1*A*—C2*A*—C3*A*—C4*A*	−49.3 (2)
C2*A*—C3*A*—C4*A*—N5*A*	68.9 (2)
C3*A*—C4*A*—N5*A*—C10*A*	8.7 (3)
C4*A*—N5*A*—C10*A*—C11*A*	−54.1 (3)
N5*A*—C10*A*—C11*A*—N1*A*	−1.5 (3)
C2*A*—N1*A*—C11*A*—C10*A*	75.8 (3)

The H atoms attached to N5 and N5*A* were located from the difference map and were refined with isotropic displacement parameters in the subsequent cycles of refinement. All the other H atoms were fixed using geometrical considerations.

Data collection: R-AXIS image plate software. Cell refinement: R-AXIS image plate software. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL*97. Geometrical calculations: *PARST*96 (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1098). Services for accessing these data are described at the back of the journal.

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2,11-Dithia[3.3.1]paracyclophane, (I), and 2,11-dithia[3.3.2]paracyclophane, (II)†

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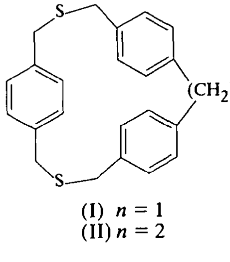
Abstract

In the title compounds [$C_{23}H_{22}S_2$, (I), and $C_{24}H_{24}S_2$, (II)], the thio-bridging chains on either side of the benzene ring (*A*) are oriented *anti* to each other. Ring *A* is located nearly perpendicular to the other benzene rings (*B* and *B'*) to give a short contact between an H atom of ring *A* and the center of ring *B* (*B'*). The thio-bridging moieties in (I) and (II) seem to be almost strain-free.

Comment

In relation to the cyclophanes, thio analogs are interesting substances and the stereochemistry of a few thiocyclophanes has been studied by means of X-ray crystallography (Lai, 1981; Keehn, 1983; Mitchell, 1983; Chan *et al.*, 1986). The title compounds, (I) and (II), are expected to show ring flexibility similar to that



(I) $n = 1$
(II) $n = 2$

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† Alternative names: (I) is 8,15-dithiatetracyclo$[15.2.2.2^{3,6}.2^{10,13}]$-pentacosa-3,5,10,12,17,19(1),20,22,24-nonaene and (II) is 9,16-dithiatetracyclo$[16.2.2.2^{4,7}.2^{11,14}]$hexacosa-4,6,11,13,18,20(1),21,23,25-nonaene.

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