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2,4-Bis(4-chlorophenyl)-2-methyl-2,3-dihydro-1H-1,5-benzodiazepine

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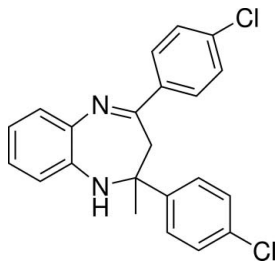
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Key indicators: single-crystal X-ray study; $T = 213$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.097; data-to-parameter ratio = 14.1.

In the title compound, $\text{C}_{22}\text{H}_{18}\text{Cl}_2\text{N}_2$, the seven-membered heterocyclic ring is in a boat-shaped conformation. The molecules are linked by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonding into one-dimensional chains along the [010] direction.

Related literature

For related structures see: Braun *et al.* (2000); Hormaza *et al.* (2004); Cabral *et al.* (1982); Braun & Muller (2004); Samanta *et al.* (1999); Kaluski *et al.* (1989). For related literature, see: Atwal *et al.* (1987); Aversa *et al.* (1986); Chimirri *et al.* (1990); Di Braccio *et al.* (2001); El-Sayed *et al.* (1999); Kaluski *et al.* (1989); Landquist (1984); Low *et al.* (2003); Merluzzi *et al.* (1990); Reddy *et al.* (2000); Schutz (1982); Tranquillini *et al.* (1997).



Experimental

Crystal data

$\text{C}_{22}\text{H}_{18}\text{Cl}_2\text{N}_2$

$M_r = 381.28$

Monoclinic, $P2_1/c$

$a = 14.095$ (3) Å

$b = 9.9845$ (15) Å

$c = 14.859$ (3) Å

$\beta = 116.728$ (3)°

$V = 1867.7$ (6) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.36$ mm⁻¹

$T = 213$ (2) K

$0.48 \times 0.38 \times 0.20$ mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan
(Jacobson, 1998)

$T_{\min} = 0.834$, $T_{\max} = 0.921$

17639 measured reflections
3404 independent reflections
3103 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.097$

$S = 1.14$

3404 reflections

241 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\max} = 0.20$ e Å⁻³

$\Delta\rho_{\min} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{N1}^i$	0.83 (2)	2.50 (2)	3.306 (2)	165 (2)

Symmetry code: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2040).

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2,4-Bis(4-chlorophenyl)-2-methyl-2,3-dihydro-1*H*-1,5-benzodiazepine

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Comment

1,5-benzodiazepine derivatives have been attracting great interest because of their potential pharmacological use as hypnotic agents (Landquist *et al.*, 1984; Schutz *et al.*, 1982), against cancer (Atwal *et al.*, 1987; Merluzzi *et al.*, 1990), viral infections (Merluzzi *et al.*, 1990) and cardiovascular disorders (Di Braccio *et al.*, 2001; Tranquillini *et al.*, 1997). They are also of interest in the development of new classes of fused heterocyclic compounds, as exemplified by triazolo- (Aversa *et al.*, 1986), oxadiazolo- (Chimirri *et al.*, 1990), oxazino- (El-Sayed *et al.*, 1999) or furanobenzodiazepines (Reddy *et al.*, 2000). A literature search for 1,5-benzodiazepines substituted at C7 and C9 leads to (2-(4-Nitro-phenyl)-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepine) (Braun *et al.*, 2000), (2,4-Bis-(2,5-dipropoxy-phenyl)-2,3-dihydro-1*H*-1,5-benzodiazepine) (Hormaza *et al.*, 2004), 4,5:15,16-Dibenzo-2,7:13,18-dimethano-7,13-dimethyl-3,6,14, 17,23,24-hexa-azatricyclo(17.3.1.18,12) tetracos-1(23),2,8, 11,12 (24),17,19,21-octaene (V) (de O. Cabral *et al.*, 1982) and (VI) (4-(4-Methoxy-phenyl)-2-(4-nitro-phenyl)-2,3-dihydro-1*H*-1,5-benzodiazepine) (Braun *et al.*, 2004), which are related to the title compound. In view of the importance of such compounds we have determined the structure of the title compound (I).

In the crystal structure of the title compound (I) the seven-membered heterocyclic is in an boat-shaped conformation with an approximate mirror plane through C8 and the mid-point of the C1—C2 bond (Fig. 1). A similar boat structure is also observed in 1-Benzyl-4-(4-nitrophenyl)-2,3-dihydro-1*H*-1,5-benzodiazepine (Low *et al.*, 2003) and 2,2,4-Trimethyl-2,3-dihydro-1*H*-1,5-benzodiazepine (Samanta *et al.*, 1999). The atoms N1 and N2 are not coplanar with the adjacent aryl ring, as shown both by the N1—C1—C2—N2 torsion angle (4.1 (3) °), and by the deviations of the two N atoms (−0.015 (1) Å for N1 and −0.154 (2) Å for N2) from the plane of the aryl ring (C1/C2/C3/C4/C5/C6).

The phenyl rings C10 to C15 and C16 to C21 are rotated out of the plane of the seven membered ring, which is probably due to the steric affects of the phenyl groups. This phenomenon is also found in 2,4-diphenyl-2,3-dihydro-1*H*-1,5-benzodiazepine (Kaluski *et al.*, 1989).

The molecules are connected *via* intermolecular N—H⋯N hydrogen bonding between N2 which act as a donor and the H atom attached to N1 into chains that elongate in the [010] direction (Fig. 2).

Experimental

Phenylenediamine (0.11 g, 1.0 mmol), montmorillonite K10 (0.3 g) and 4-chloroacetophenone (0.34 g, 2.2 mmol) were mixed and afterwards allowed to stay at room temperature for 24 h. On completion of the reaction, which was monitored by TLC, montmorillonite was filtered off and washed with 3.5 ml of acetone. The filtrate was evaporated to dryness and the residue was subjected to column chromatography over silica gel using petroleum ether-acetone (4:1) as eluent to afford compound I in 87% yield (0.33 g). The crystal was obtained by evaporation of the solvent from a solution of I in acetone-petroleum ether (1:1). ¹H NMR (CDCl₃, 400 MHz): δ 1.74 (s, 3H, CH₃), 2.88 (d, 1H, J=12.8 Hz), 3.07 (d, 1H, J=12 Hz), 3.43 (s, 1H, NH), 6.84–7.53 (m, 12H, ArH). ¹³C NMR (CDCl₃, 100 MHz): δ 30.2, 43.3, 74.1, 122.0, 122.5, 127.1, 127.5,

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128.7, 128.8, 129.0, 133.4, 136.5, 138.0, 140.3, 146.2, 166.6. UV λ_{max} (EtOH): 262 nm, 365 nm. IR (KBr, cm^{-1}): 3272 (*s*), 1590 (*m*), 1559(*m*), 1474(*s*), 1436(*w*), 1397(*m*), 1328(*m*), 1243(*s*), 1212(*m*), 1096(*m*), 1011(*m*).

Refinement

C—H atoms were positioned with idealized geometry and were refined isotropic using a riding model with C—H = 0.94 Å for aromatic, C—H = 0.98 Å for methylene and C—H = 0.97 Å for methyl H atoms and $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ for aromatic and methylene H atoms and $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. The N—H H atom was located in difference map and was refined isotropic ($U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{N})$) with the N—H bond length restraint to 0.83 (2) Å.

Figures

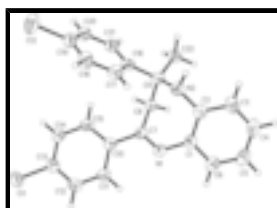


Fig. 1. Crystal structure of compound (I) with labelling and displacement ellipsoid drawn at the 50% probability level.

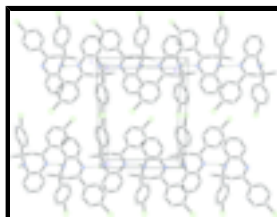


Fig. 2. Crystal structure of compound (I) with view in the direction of the *c* axis (N—H...N hydrogen bonding is shown as dashed lines).

2,4-Bis(4-chlorophenyl)-2-methyl-2,3-dihydro-1*H*-1,5-benzodiazepine

Crystal data

$\text{C}_{22}\text{H}_{18}\text{Cl}_2\text{N}_2$

$M_r = 381.28$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 14.095$ (3) Å

$b = 9.9845$ (15) Å

$c = 14.859$ (3) Å

$\beta = 116.728$ (3)°

$V = 1867.7$ (6) Å³

$Z = 4$

$F_{000} = 792$

$D_x = 1.356$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71070$ Å

Cell parameters from 6453 reflections

$\theta = 3.1$ – 25.3 °

$\mu = 0.36$ mm⁻¹

$T = 213$ (2) K

Block, yellow

$0.48 \times 0.38 \times 0.20$ mm

Data collection

Rigaku Mercury
diffractometer

Radiation source: fine-focus sealed tube

3103 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

Monochromator: graphite $\theta_{\max} = 25.4^\circ$
 $T = 213(2)$ K $\theta_{\min} = 3.1^\circ$
 ω scans $h = -16 \rightarrow 16$
 Absorption correction: multi-scan (Jacobson, 1998) $k = -12 \rightarrow 12$
 $T_{\min} = 0.834$, $T_{\max} = 0.921$ $l = -17 \rightarrow 16$
 17639 measured reflections Standard reflections: ?
 3404 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.043$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.097$ $w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 0.8045P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.14$ $(\Delta/\sigma)_{\max} = 0.001$
 3404 reflections $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
 241 parameters $\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.45977 (4)	0.38308 (5)	0.58336 (4)	0.05107 (17)
Cl2	0.49691 (4)	0.97041 (7)	0.31844 (4)	0.0594 (2)
N1	0.00610 (12)	0.71966 (15)	0.37243 (11)	0.0301 (3)
N2	0.00367 (12)	0.97254 (17)	0.27959 (12)	0.0315 (4)
H2	-0.0114 (17)	1.033 (2)	0.2376 (18)	0.041 (6)*
C1	-0.08515 (14)	0.80205 (18)	0.33118 (13)	0.0296 (4)
C2	-0.08722 (14)	0.92813 (19)	0.28826 (13)	0.0312 (4)
C3	-0.18201 (16)	0.9998 (2)	0.24741 (15)	0.0409 (5)
H3	-0.1844	1.0846	0.2189	0.049*

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C4	-0.27317 (16)	0.9482 (2)	0.24808 (16)	0.0476 (5)
H4	-0.3362	0.9986	0.2210	0.057*
C5	-0.27147 (16)	0.8233 (2)	0.28832 (16)	0.0453 (5)
H5	-0.3332	0.7883	0.2887	0.054*
C6	-0.17828 (15)	0.7497 (2)	0.32819 (14)	0.0369 (4)
H6	-0.1777	0.6634	0.3536	0.044*
C7	0.09696 (14)	0.77208 (18)	0.42989 (13)	0.0281 (4)
C8	0.10919 (14)	0.91792 (17)	0.45824 (13)	0.0286 (4)
H8A	0.1795	0.9315	0.5151	0.034*
H8B	0.0561	0.9413	0.4812	0.034*
C9	0.09695 (14)	1.01437 (18)	0.37237 (13)	0.0287 (4)
C10	0.18912 (14)	0.67966 (18)	0.47382 (12)	0.0281 (4)
C11	0.17155 (15)	0.54254 (19)	0.47762 (14)	0.0330 (4)
H11	0.1020	0.5107	0.4562	0.040*
C12	0.25524 (15)	0.45329 (19)	0.51245 (14)	0.0361 (4)
H12	0.2426	0.3612	0.5145	0.043*
C13	0.35711 (15)	0.49938 (19)	0.54418 (14)	0.0347 (4)
C14	0.37761 (15)	0.6339 (2)	0.54494 (15)	0.0387 (5)
H14	0.4478	0.6648	0.5690	0.046*
C15	0.29383 (15)	0.72347 (19)	0.50997 (14)	0.0349 (4)
H15	0.3077	0.8156	0.5106	0.042*
C16	0.19604 (15)	1.00558 (17)	0.35502 (13)	0.0298 (4)
C17	0.20002 (16)	0.9223 (2)	0.28171 (14)	0.0357 (4)
H17	0.1393	0.8735	0.2391	0.043*
C18	0.29232 (17)	0.9102 (2)	0.27055 (15)	0.0411 (5)
H18	0.2944	0.8527	0.2213	0.049*
C19	0.38053 (16)	0.9830 (2)	0.33215 (15)	0.0383 (5)
C20	0.37924 (16)	1.0662 (2)	0.40541 (15)	0.0391 (5)
H20	0.4401	1.1154	0.4473	0.047*
C21	0.28728 (15)	1.07658 (19)	0.41658 (14)	0.0351 (4)
H21	0.2864	1.1329	0.4669	0.042*
C22	0.07973 (16)	1.15650 (19)	0.40215 (15)	0.0358 (4)
H22A	0.0843	1.2207	0.3553	0.054*
H22B	0.1339	1.1763	0.4698	0.054*
H22C	0.0101	1.1621	0.4002	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0413 (3)	0.0425 (3)	0.0614 (4)	0.0102 (2)	0.0160 (3)	-0.0041 (2)
C12	0.0425 (3)	0.0919 (5)	0.0528 (4)	0.0102 (3)	0.0294 (3)	0.0096 (3)
N1	0.0302 (8)	0.0306 (8)	0.0304 (8)	-0.0029 (7)	0.0143 (7)	-0.0013 (6)
N2	0.0355 (9)	0.0345 (9)	0.0247 (8)	0.0000 (7)	0.0136 (7)	0.0030 (7)
C1	0.0294 (9)	0.0332 (10)	0.0263 (9)	-0.0017 (8)	0.0126 (7)	-0.0062 (7)
C2	0.0322 (10)	0.0382 (10)	0.0239 (9)	0.0013 (8)	0.0132 (8)	-0.0032 (8)
C3	0.0412 (12)	0.0471 (12)	0.0336 (11)	0.0102 (9)	0.0161 (9)	0.0033 (9)
C4	0.0316 (11)	0.0665 (15)	0.0417 (12)	0.0116 (10)	0.0138 (9)	-0.0004 (11)
C5	0.0319 (11)	0.0618 (14)	0.0450 (12)	-0.0034 (10)	0.0198 (9)	-0.0064 (10)

C6	0.0353 (11)	0.0427 (11)	0.0345 (10)	-0.0050 (9)	0.0174 (8)	-0.0057 (8)
C7	0.0319 (10)	0.0311 (10)	0.0249 (9)	-0.0026 (8)	0.0161 (8)	0.0013 (7)
C8	0.0334 (10)	0.0286 (9)	0.0251 (9)	-0.0013 (8)	0.0145 (8)	-0.0017 (7)
C9	0.0353 (10)	0.0274 (9)	0.0255 (9)	-0.0031 (8)	0.0155 (8)	-0.0021 (7)
C10	0.0331 (9)	0.0286 (9)	0.0233 (9)	-0.0034 (8)	0.0133 (7)	-0.0010 (7)
C11	0.0329 (10)	0.0312 (10)	0.0337 (10)	-0.0052 (8)	0.0141 (8)	0.0000 (8)
C12	0.0409 (11)	0.0282 (10)	0.0375 (11)	-0.0027 (8)	0.0161 (9)	-0.0003 (8)
C13	0.0342 (10)	0.0366 (11)	0.0322 (10)	0.0037 (8)	0.0139 (8)	-0.0005 (8)
C14	0.0288 (10)	0.0383 (11)	0.0471 (12)	-0.0041 (8)	0.0153 (9)	0.0001 (9)
C15	0.0368 (11)	0.0289 (10)	0.0382 (11)	-0.0052 (8)	0.0163 (9)	0.0001 (8)
C16	0.0381 (10)	0.0250 (9)	0.0291 (9)	0.0005 (8)	0.0177 (8)	0.0031 (7)
C17	0.0398 (11)	0.0367 (11)	0.0332 (10)	-0.0024 (9)	0.0187 (9)	-0.0033 (8)
C18	0.0470 (12)	0.0477 (12)	0.0347 (11)	0.0060 (10)	0.0237 (9)	-0.0025 (9)
C19	0.0367 (11)	0.0463 (12)	0.0367 (11)	0.0072 (9)	0.0207 (9)	0.0109 (9)
C20	0.0360 (11)	0.0371 (11)	0.0415 (11)	-0.0017 (9)	0.0150 (9)	0.0030 (9)
C21	0.0398 (11)	0.0310 (10)	0.0355 (10)	-0.0017 (8)	0.0178 (9)	-0.0044 (8)
C22	0.0453 (11)	0.0288 (10)	0.0394 (11)	0.0010 (8)	0.0244 (9)	-0.0011 (8)

Geometric parameters (Å, °)

C11—C13	1.7387 (19)	C10—C15	1.395 (3)
C12—C19	1.746 (2)	C10—C11	1.397 (3)
N1—C7	1.288 (2)	C11—C12	1.380 (3)
N1—C1	1.413 (2)	C11—H11	0.9400
N2—C2	1.415 (2)	C12—C13	1.374 (3)
N2—C9	1.474 (2)	C12—H12	0.9400
N2—H2	0.83 (2)	C13—C14	1.373 (3)
C1—C6	1.395 (3)	C14—C15	1.383 (3)
C1—C2	1.405 (3)	C14—H14	0.9400
C2—C3	1.392 (3)	C15—H15	0.9400
C3—C4	1.389 (3)	C16—C21	1.391 (3)
C3—H3	0.9400	C16—C17	1.391 (3)
C4—C5	1.379 (3)	C17—C18	1.389 (3)
C4—H4	0.9400	C17—H17	0.9400
C5—C6	1.384 (3)	C18—C19	1.374 (3)
C5—H5	0.9400	C18—H18	0.9400
C6—H6	0.9400	C19—C20	1.376 (3)
C7—C10	1.484 (3)	C20—C21	1.383 (3)
C7—C8	1.504 (2)	C20—H20	0.9400
C8—C9	1.545 (2)	C21—H21	0.9400
C8—H8A	0.9800	C22—H22A	0.9700
C8—H8B	0.9800	C22—H22B	0.9700
C9—C16	1.533 (2)	C22—H22C	0.9700
C9—C22	1.538 (3)		
C7—N1—C1	119.37 (15)	C12—C11—C10	120.72 (17)
C2—N2—C9	117.82 (14)	C12—C11—H11	119.6
C2—N2—H2	111.2 (15)	C10—C11—H11	119.6
C9—N2—H2	108.5 (15)	C13—C12—C11	119.85 (18)
C6—C1—C2	119.58 (17)	C13—C12—H12	120.1

supplementary materials

C6—C1—N1	117.11 (17)	C11—C12—H12	120.1
C2—C1—N1	123.18 (16)	C14—C13—C12	120.95 (18)
C3—C2—C1	118.60 (18)	C14—C13—C11	120.68 (15)
C3—C2—N2	122.00 (18)	C12—C13—C11	118.37 (15)
C1—C2—N2	119.14 (16)	C13—C14—C15	119.25 (18)
C4—C3—C2	121.1 (2)	C13—C14—H14	120.4
C4—C3—H3	119.5	C15—C14—H14	120.4
C2—C3—H3	119.5	C14—C15—C10	121.25 (17)
C5—C4—C3	120.2 (2)	C14—C15—H15	119.4
C5—C4—H4	119.9	C10—C15—H15	119.4
C3—C4—H4	119.9	C21—C16—C17	117.94 (17)
C4—C5—C6	119.57 (19)	C21—C16—C9	120.21 (16)
C4—C5—H5	120.2	C17—C16—C9	121.77 (16)
C6—C5—H5	120.2	C18—C17—C16	121.03 (18)
C5—C6—C1	120.9 (2)	C18—C17—H17	119.5
C5—C6—H6	119.6	C16—C17—H17	119.5
C1—C6—H6	119.6	C19—C18—C17	119.32 (18)
N1—C7—C10	116.84 (16)	C19—C18—H18	120.3
N1—C7—C8	122.24 (16)	C17—C18—H18	120.3
C10—C7—C8	120.76 (15)	C18—C19—C20	121.10 (18)
C7—C8—C9	114.58 (14)	C18—C19—C12	120.02 (16)
C7—C8—H8A	108.6	C20—C19—C12	118.88 (16)
C9—C8—H8A	108.6	C19—C20—C21	119.11 (19)
C7—C8—H8B	108.6	C19—C20—H20	120.4
C9—C8—H8B	108.6	C21—C20—H20	120.4
H8A—C8—H8B	107.6	C20—C21—C16	121.49 (18)
N2—C9—C16	108.87 (14)	C20—C21—H21	119.3
N2—C9—C22	109.92 (15)	C16—C21—H21	119.3
C16—C9—C22	112.02 (15)	C9—C22—H22A	109.5
N2—C9—C8	108.48 (14)	C9—C22—H22B	109.5
C16—C9—C8	109.36 (14)	H22A—C22—H22B	109.5
C22—C9—C8	108.12 (14)	C9—C22—H22C	109.5
C15—C10—C11	117.90 (17)	H22A—C22—H22C	109.5
C15—C10—C7	122.66 (16)	H22B—C22—H22C	109.5
C11—C10—C7	119.42 (16)		
C7—N1—C1—C6	-139.58 (17)	C8—C7—C10—C11	156.48 (16)
C7—N1—C1—C2	44.5 (2)	C15—C10—C11—C12	-2.5 (3)
C6—C1—C2—C3	2.6 (3)	C7—C10—C11—C12	176.09 (16)
N1—C1—C2—C3	178.39 (16)	C10—C11—C12—C13	0.3 (3)
C6—C1—C2—N2	-171.65 (16)	C11—C12—C13—C14	2.2 (3)
N1—C1—C2—N2	4.1 (3)	C11—C12—C13—C11	-177.79 (15)
C9—N2—C2—C3	112.9 (2)	C12—C13—C14—C15	-2.2 (3)
C9—N2—C2—C1	-73.0 (2)	C11—C13—C14—C15	177.73 (15)
C1—C2—C3—C4	-0.4 (3)	C13—C14—C15—C10	-0.1 (3)
N2—C2—C3—C4	173.62 (18)	C11—C10—C15—C14	2.4 (3)
C2—C3—C4—C5	-0.9 (3)	C7—C10—C15—C14	-176.10 (17)
C3—C4—C5—C6	0.1 (3)	N2—C9—C16—C21	160.02 (16)
C4—C5—C6—C1	2.1 (3)	C22—C9—C16—C21	38.2 (2)
C2—C1—C6—C5	-3.5 (3)	C8—C9—C16—C21	-81.6 (2)

N1—C1—C6—C5	-179.51 (16)	N2—C9—C16—C17	-23.3 (2)
C1—N1—C7—C10	177.88 (14)	C22—C9—C16—C17	-145.10 (17)
C1—N1—C7—C8	2.4 (2)	C8—C9—C16—C17	95.1 (2)
N1—C7—C8—C9	-73.0 (2)	C21—C16—C17—C18	0.2 (3)
C10—C7—C8—C9	111.68 (18)	C9—C16—C17—C18	-176.54 (17)
C2—N2—C9—C16	158.23 (15)	C16—C17—C18—C19	-0.8 (3)
C2—N2—C9—C22	-78.7 (2)	C17—C18—C19—C20	0.8 (3)
C2—N2—C9—C8	39.3 (2)	C17—C18—C19—C12	-179.33 (15)
C7—C8—C9—N2	45.0 (2)	C18—C19—C20—C21	-0.2 (3)
C7—C8—C9—C16	-73.60 (19)	C12—C19—C20—C21	179.98 (15)
C7—C8—C9—C22	164.18 (15)	C19—C20—C21—C16	-0.5 (3)
N1—C7—C10—C15	159.47 (16)	C17—C16—C21—C20	0.5 (3)
C8—C7—C10—C15	-25.0 (2)	C9—C16—C21—C20	177.27 (17)
N1—C7—C10—C11	-19.1 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots N1 ⁱ	0.83 (2)	2.50 (2)	3.306 (2)	165 (2)

Symmetry codes: (i) $-x, y+1/2, -z+1/2$.

Fig. 1

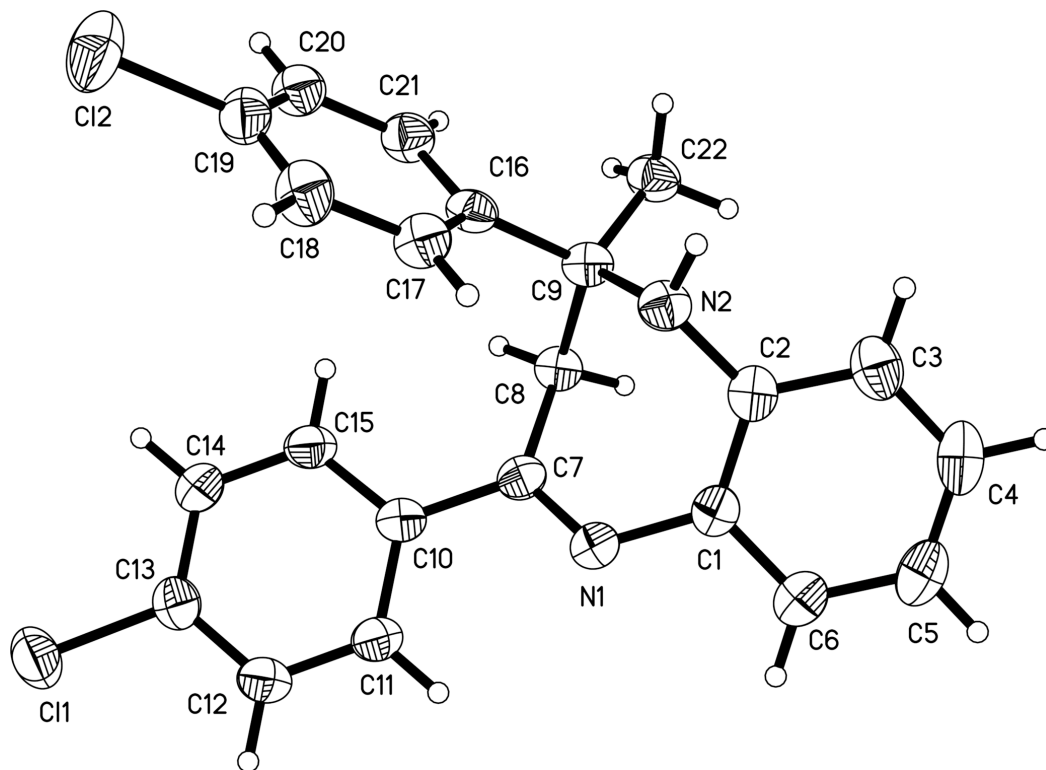


Fig. 2

