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Geometric forms of 8-chloro-11-[4-(8-chloro-5*H*-dibenzo[*b*,e][1,4]diazepin-11-yl)piperazin-1-yl]-5*H*-dibenzo[*b*,e][1,4]-diazepine–acetone–pentane (2/1/1)

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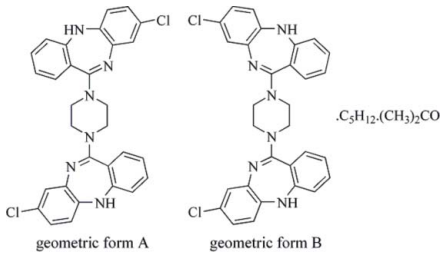
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.006 Å; disorder in solvent or counterion; R factor = 0.064; wR factor = 0.158; data-to-parameter ratio = 13.1.

The crystal structure of the title compound, $2C_{30}H_{24}Cl_2N_6\cdot C_5H_{12}\cdot C_3H_6O$, shows the presence of two geometric forms of the $C_{30}H_{24}Cl_2N_6$ molecule, each exhibiting the characteristic buckled nature of the dibenzodiazepine nucleus with the central seven-membered heterocycle in a boat conformation. Form A contains a twofold rotation axis and form B a centre of symmetry. The solvent molecules are disordered equally over two sites.

Related literature

For related literature, see: Andreasen *et al.* (1994); Capuano (1999); Capuano *et al.* (2006); Gerlach (1991); Gerson & Meltzer (1992); Hypercube Inc (2005); Petcher & Weber (1976); Veys *et al.* (1992).



Experimental

Crystal data

$2C_{30}H_{24}Cl_2N_6\cdot C_5H_{12}\cdot C_3H_6O$
M_r = 1209.13
Monoclinic, $C2/c$
a = 31.3716 (2) Å
b = 11.3509 (2) Å
c = 22.7030 (3) Å
β = 131.147 (1)°
V = 6087.77 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 123 (2) K
0.20 × 0.20 × 0.08 mm

Data collection

Enraf Nonius KAPPA CCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.94, T_{max} = 0.99
23817 measured reflections
5349 independent reflections
3743 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.158
S = 1.06
5349 reflections
408 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.83 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N5^{i}	0.85 (4)	2.33 (4)	3.154 (4)	166 (3)

Symmetry code: (i) $-x, -y, -z$.

Data collection: *COLLECT* (Bruker AXS, 2000); cell refinement: *DENZO SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); 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: HG2283).

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supplementary materials

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Geometric forms of 8-chloro-11-[4-(8-chloro-5*H*-dibenzo[*b*,*e*][1,4]diazepin-11-yl)piperazin-1-yl]-5*H*-dibenzo[*b*,*e*][1,4]diazepine-acetone-pentane (2/1/1)

B. Capuano, I. T. Crosby, G. D. Fallon, C. M. Forsyth, E. J. Lloyd, A. Podloucka and E. Yuriev

Comment

Clozapine is an atypical (Gerlach, 1991) antipsychotic drug used clinically to treat schizophrenia (Andreasen *et al.*, 1994). Clozapine, however, has been found to induce the potentially fatal blood disorder agranulocytosis. The major metabolite of clozapine, 8-chloro-11-piperazino-5*H*-dibenzo[*b,e*][1,4]diazepine (desmethylclozapine), has been implicated in this serious blood dyscrasia (Veys *et al.*, 1992, Gerson & Meltzer, 1992). A modified synthetic procedure (Capuano, 1999) was employed to synthesize desmethylclozapine as this compound was envisaged as a versatile intermediate towards clozapine-like analogues potentially devoid of any blood disorders, and for use in haematological studies to investigate the possible mechanism of clozapine-induced agranulocytosis. During the synthesis, the title compound (Scheme 1) was isolated as a by-product of commercial significance, purified and structurally characterized by X-ray diffraction to examine its solid state confirmation.

The structure of the title compound displays two geometric forms, A (Fig. 1), and B (Fig. 2). Both have half a molecule in the asymmetric unit, the other half generated by a 2-fold axis (located at the mid-point of the C—C bonds in the piperazine ring in A) or an inversion centre (located at the centroid of the piperazine ring in B). These symmetry operations generate a *cisoid* relationship of the dibenzodiazepine nucleii for A and a *transiod* relationship for B. Each unique dibenzodiazepine exhibits a typical buckled nature with the central seven-membered heterocycle in a boat arrangement. Both forms A and B have the opposite boat conformation at either end of the molecule (as required by symmetry) and consequently conversion from A to B requires inversion of the dibenzodiazepine moiety as well as rotation about the C–N linkage between the dibenzodiazepine and piperazine units. Molecular mechanics single point calculations were carried out for both geometric forms present in the unit cell (*HYPERCHEM* v7.5 (Hypercube Inc, 2005), *MM+* force field, *in vacuo*). This calculation, which accounts for the effects of crystal packing forces, determined an energy difference ΔE (E_A—E_B) of 27 kcal mol^{-1} indicating that geometric form B is the more thermodynamically stable form of (I). The observation of two distinct forms of the title compound contrasts the analogous pyridobenzoxazepine compound 8-chloro-5-[4-(8-chloropyrido [2,3-*b*][1,5]benzoxazepin-5-yl)piperazino]pyrido[2,3-*b*] [1,5]benzoxazepine which has only one form corresponding to B (Capuano *et al.*, 2006). The dihedral angle between the planes of the aromatic rings is 126.1 (1)° for A and 119.6 (1)° for B, which is comparable to the 115° observed for clozapine (Petcher & Weber, 1976). Short N2—C13 and N5—C28 bond distances, 1.280 (4) Å and 1.291 (5) Å respectively, confirm the presence of double bonds at these positions. The piperazine ring adopts a chair conformation with the tricyclic groups assuming a pseudoequatorial orientation, by virtue of the sp^2-like nature of the piperazine nitrogen atoms (Σ(°) 347.1). Alternating A and B forms are weakly associated by N1—H1A···N5 hydrogen bonds generating zigzag chains parallel to the *c* axis.

Experimental

The title compound was prepared according to a literature procedure (Capuano, 1999). Crystals suitable for X-ray diffraction were grown using the diffusion method, from an acetonic solution of the compound layered onto hexanes, affording bright

yellow prisms. Isolation of the bulk solid after drying resulted in loss of lattice solvent (acetone / hexanes) (m.p. 606–608 K dec.).

Refinement

After location of the primary molecules, residiual peaks located near the origin and the 2-fold axis (z =1/4) were assigned to disordered lattice solvent, acetone and pentane respectively. These were modelled with restrained geometries and the pentane was refined as isotropic atoms only. All H atoms for the primary molecules were initially located in the difference Fourier map. H1A and H4A (attached to N1 and N4 respectively) were freely refined but all other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å and U_{iso}(H) = 1.2–1.5 U_{eq}(C).

Figures

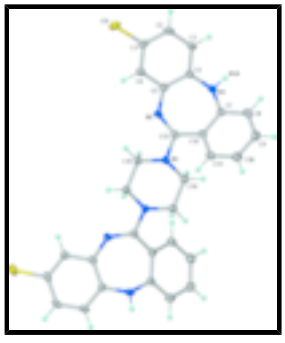


Fig. 1. A view of geometric form A. Displacement ellipsoids are drawn at the 50% probability level.

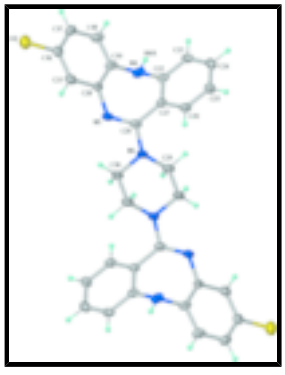


Fig. 2. A view of geometric form B. Displacement ellipsoids are drawn at the 50% probability level.

8-chloro-11-[4-(8-chloro-5*H*-dibenzo[*b*,*e*][1,4] diazepin-11-yl)piperazin-1-yl]-5*H*-dibenzo[*b*,*e*][1,4]diazepine–acetone–pentane (2/1/1)

Crystal data

$2C_{30}H_{24}Cl_2N_6{\cdot}C_5H_{12}{\cdot}C_3H_6O$	F_{000} = 2536
M_r = 1209.13	D_x = 1.319 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 23817 reflections
a = 31.3716 (2) Å	θ = 3.2–25.0º

b = 11.3509 (2) Å	μ = 0.25 mm^{-1}
c = 22.7030 (3) Å	T = 123 (2) K
β = 131.147 (1)º	Tabular, yellow
V = 6087.77 (16) Å^3	0.20 × 0.20 × 0.08 mm
Z = 4	

Data collection

Enraf Nonius KAPPA CCD diffractometer	5349 independent reflections
Radiation source: fine-focus sealed tube	3743 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.061
T = 123(2) K	θ_{max} = 25.0º
phi and ω scans	θ_{min} = 3.2º
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	h = −37→37
T_{min} = 0.94, T_{max} = 0.99	k = −13→13
23817 measured reflections	l = −26→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.065	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.158	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 21.5958P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
5349 reflections	$\Delta\rho_{max}$ = 0.83 e Å^{-3}
408 parameters	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
9 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.20014 (4)	0.30149 (8)	0.21264 (6)	0.0310 (3)	
Cl2	−0.16231 (5)	0.00760 (9)	0.07128 (7)	0.0448 (3)	
O1	−0.0158 (3)	0.1161 (6)	0.0546 (4)	0.0589 (18)	0.50
N1	0.11606 (12)	−0.1904 (3)	0.15222 (18)	0.0227 (7)	
N2	0.08616 (12)	−0.0153 (3)	0.21214 (16)	0.0204 (7)	
N3	0.04554 (11)	−0.1185 (2)	0.25006 (17)	0.0203 (7)	
N4	−0.21603 (14)	0.4267 (3)	−0.12315 (18)	0.0282 (8)	
N5	−0.10501 (12)	0.3277 (3)	−0.02192 (18)	0.0273 (7)	
N6	−0.04850 (12)	0.4712 (3)	−0.00987 (19)	0.0286 (8)	
C1	0.17546 (14)	0.1569 (3)	0.19392 (19)	0.0217 (8)	
C2	0.18875 (14)	0.0779 (3)	0.1620 (2)	0.0236 (8)	
H2	0.2104	0.1022	0.1485	0.028*	
C3	0.17007 (14)	−0.0379 (3)	0.1498 (2)	0.0226 (8)	
H3	0.1792	−0.0930	0.1281	0.027*	
C4	0.13800 (14)	−0.0741 (3)	0.16895 (19)	0.0184 (8)	
C5	0.12407 (13)	0.0067 (3)	0.20035 (19)	0.0192 (8)	
C6	0.14297 (14)	0.1234 (3)	0.21229 (19)	0.0202 (8)	
H6	0.1335	0.1795	0.2331	0.024*	
C7	0.13325 (14)	−0.2585 (3)	0.2171 (2)	0.0195 (8)	
C8	0.16246 (14)	−0.3641 (3)	0.2363 (2)	0.0251 (8)	
H8	0.1703	−0.3925	0.2050	0.030*	
C9	0.18005 (15)	−0.4278 (3)	0.3010 (2)	0.0277 (9)	
H9	0.1990	−0.5008	0.3130	0.033*	
C10	0.17021 (15)	−0.3859 (3)	0.3484 (2)	0.0279 (9)	
H10	0.1839	−0.4279	0.3942	0.033*	
C11	0.14026 (14)	−0.2824 (3)	0.3284 (2)	0.0218 (8)	
H11	0.1337	−0.2535	0.3611	0.026*	
C12	0.11958 (13)	−0.2197 (3)	0.26174 (19)	0.0192 (8)	
C13	0.08344 (13)	−0.1134 (3)	0.23741 (19)	0.0188 (8)	
C14	0.01166 (14)	−0.2250 (3)	0.2293 (2)	0.0213 (8)	
H14A	0.0355	−0.2957	0.2449	0.026*	
H14B	−0.0200	−0.2278	0.1720	0.026*	
C15	0.01207 (16)	−0.0122 (3)	0.2301 (2)	0.0275 (9)	
H15A	−0.0192	−0.0082	0.1727	0.033*	
H15B	0.0362	0.0582	0.2466	0.033*	
C16	−0.17895 (16)	0.1280 (3)	0.0112 (2)	0.0288 (9)	
C17	−0.23464 (16)	0.1655 (3)	−0.0429 (2)	0.0281 (9)	
H17	−0.2639	0.1242	−0.0496	0.034*	
C18	−0.24668 (16)	0.2646 (4)	−0.0871 (2)	0.0295 (9)	
H18	−0.2846	0.2921	−0.1239	0.035*	
C19	−0.20440 (15)	0.3242 (3)	−0.0785 (2)	0.0243 (8)	
C20	−0.14847 (15)	0.2832 (3)	−0.0256 (2)	0.0234 (8)	
C21	−0.13644 (16)	0.1835 (3)	0.0192 (2)	0.0286 (9)	
H21	−0.0988	0.1540	0.0552	0.034*	
C22	−0.18879 (14)	0.5297 (3)	−0.07650 (19)	0.0226 (8)	

C23	−0.21963 (15)	0.6224 (3)	−0.0810 (2)	0.0261 (9)	
H23	−0.2597	0.6170	−0.1150	0.031*	
C24	−0.19268 (16)	0.7223 (3)	−0.0364 (2)	0.0307 (9)	
H24	−0.2142	0.7859	−0.0406	0.037*	
C25	−0.13406 (15)	0.7302 (3)	0.0147 (2)	0.0284 (9)	
H25	−0.1154	0.7987	0.0458	0.034*	
C26	−0.10309 (15)	0.6379 (3)	0.0198 (2)	0.0266 (9)	
H26	−0.0630	0.6434	0.0550	0.032*	
C27	−0.12948 (14)	0.5367 (3)	−0.02575 (19)	0.0210 (8)	
C28	−0.09638 (14)	0.4389 (3)	−0.0221 (2)	0.0244 (8)	
C29	−0.04984 (15)	0.5690 (3)	−0.0536 (2)	0.0296 (9)	
H29A	−0.0762	0.6307	−0.0636	0.036*	
H29B	−0.0637	0.5399	−0.1048	0.036*	
C30	−0.00914 (15)	0.3787 (3)	0.0074 (2)	0.0311 (9)	
H30A	−0.0219	0.3442	−0.0420	0.037*	
H30B	−0.0082	0.3153	0.0382	0.037*	
C31	−0.0300 (7)	−0.0738 (9)	0.0164 (10)	0.050 (4)	0.50
H31A	0.0015	−0.1288	0.0511	0.075*	0.50
H31B	−0.0526	−0.0996	−0.0379	0.075*	0.50
H31C	−0.0538	−0.0720	0.0301	0.075*	0.50
C32	−0.0085 (3)	0.0391 (7)	0.0251 (5)	0.036 (2)	0.50
C33	0.0213 (7)	0.0692 (15)	0.0010 (10)	0.061 (5)	0.50
H33A	0.0431	0.1412	0.0279	0.091*	0.50
H33B	−0.0055	0.0824	−0.0556	0.091*	0.50
H33C	0.0471	0.0049	0.0139	0.091*	0.50
C34	0.0776 (6)	0.3420 (19)	0.2628 (11)	0.141 (5)*	0.50
H34A	0.1088	0.3808	0.2702	0.212*	0.50
H34B	0.0702	0.2650	0.2380	0.212*	0.50
H34C	0.0880	0.3312	0.3136	0.212*	0.50
C35	0.0249 (8)	0.4176 (17)	0.2109 (14)	0.194 (10)*	0.50
H35A	0.0299	0.4943	0.2355	0.233*	0.50
H35B	0.0133	0.4311	0.1589	0.233*	0.50
C36	−0.0151 (7)	0.3412 (14)	0.2058 (12)	0.190 (10)*	0.50
H36A	−0.0314	0.2816	0.1641	0.227*	0.50
H36B	0.0039	0.3002	0.2561	0.227*	0.50
C37	−0.0587 (7)	0.4201 (13)	0.1878 (12)	0.141 (6)*	0.50
H37A	−0.0804	0.4563	0.1355	0.169*	0.50
H37B	−0.0427	0.4827	0.2277	0.169*	0.50
C38	−0.0949 (7)	0.3330 (17)	0.1902 (12)	0.141 (5)*	0.50
H38A	−0.1282	0.3738	0.1757	0.212*	0.50
H38B	−0.0725	0.3010	0.2432	0.212*	0.50
H38C	−0.1071	0.2686	0.1533	0.212*	0.50
H1A	0.1197 (16)	−0.228 (3)	0.124 (2)	0.030 (11)*	
H4A	−0.2518 (17)	0.443 (3)	−0.159 (2)	0.026 (10)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Cl1	0.0352 (5)	0.0277 (5)	0.0341 (5)	−0.0082 (4)	0.0246 (5)	0.0011 (4)
Cl2	0.0598 (7)	0.0321 (6)	0.0560 (7)	0.0076 (5)	0.0439 (6)	0.0093 (5)
O1	0.058 (4)	0.054 (4)	0.060 (4)	0.018 (3)	0.037 (4)	0.021 (4)
N1	0.0256 (17)	0.0260 (18)	0.0223 (16)	−0.0035 (14)	0.0182 (15)	−0.0051 (14)
N2	0.0214 (15)	0.0222 (16)	0.0251 (16)	−0.0027 (13)	0.0187 (14)	−0.0014 (13)
N3	0.0198 (15)	0.0177 (15)	0.0298 (17)	−0.0021 (12)	0.0192 (14)	−0.0025 (13)
N4	0.0182 (17)	0.039 (2)	0.0171 (16)	−0.0089 (15)	0.0074 (15)	−0.0020 (15)
N5	0.0230 (17)	0.0286 (18)	0.0354 (19)	−0.0068 (14)	0.0215 (16)	−0.0116 (15)
N6	0.0188 (16)	0.0298 (18)	0.042 (2)	−0.0049 (13)	0.0221 (16)	−0.0076 (15)
C1	0.0156 (17)	0.0242 (19)	0.0181 (18)	−0.0032 (15)	0.0079 (16)	0.0050 (15)
C2	0.0174 (18)	0.036 (2)	0.0208 (19)	0.0008 (16)	0.0139 (17)	0.0047 (17)
C3	0.0199 (18)	0.031 (2)	0.0201 (19)	0.0027 (16)	0.0144 (17)	0.0013 (16)
C4	0.0181 (17)	0.0201 (18)	0.0171 (18)	−0.0002 (15)	0.0116 (16)	−0.0017 (15)
C5	0.0136 (17)	0.0249 (19)	0.0173 (17)	−0.0013 (15)	0.0093 (16)	0.0024 (15)
C6	0.0189 (18)	0.026 (2)	0.0173 (18)	0.0016 (15)	0.0125 (16)	0.0013 (15)
C7	0.0163 (17)	0.0210 (19)	0.0224 (19)	−0.0064 (15)	0.0133 (16)	−0.0051 (15)
C8	0.0225 (19)	0.025 (2)	0.033 (2)	−0.0024 (16)	0.0209 (19)	−0.0062 (17)
C9	0.0207 (19)	0.023 (2)	0.040 (2)	0.0044 (16)	0.0207 (19)	0.0022 (18)
C10	0.022 (2)	0.030 (2)	0.032 (2)	0.0030 (17)	0.0180 (19)	0.0059 (17)
C11	0.0199 (18)	0.026 (2)	0.025 (2)	−0.0020 (15)	0.0173 (17)	−0.0022 (16)
C12	0.0139 (17)	0.0211 (18)	0.0216 (19)	−0.0050 (14)	0.0112 (16)	−0.0043 (15)
C13	0.0143 (17)	0.026 (2)	0.0162 (17)	−0.0013 (15)	0.0102 (16)	−0.0042 (15)
C14	0.0177 (18)	0.0206 (19)	0.028 (2)	−0.0001 (14)	0.0160 (17)	−0.0023 (15)
C15	0.031 (2)	0.0188 (19)	0.049 (2)	0.0032 (16)	0.034 (2)	0.0024 (18)
C16	0.039 (2)	0.024 (2)	0.032 (2)	−0.0020 (18)	0.027 (2)	−0.0053 (17)
C17	0.035 (2)	0.029 (2)	0.031 (2)	−0.0103 (18)	0.026 (2)	−0.0086 (18)
C18	0.024 (2)	0.042 (2)	0.023 (2)	−0.0082 (18)	0.0155 (18)	−0.0085 (18)
C19	0.0227 (19)	0.034 (2)	0.0171 (18)	−0.0094 (16)	0.0135 (17)	−0.0088 (16)
C20	0.0241 (19)	0.024 (2)	0.027 (2)	−0.0078 (16)	0.0190 (18)	−0.0122 (16)
C21	0.027 (2)	0.030 (2)	0.031 (2)	−0.0014 (17)	0.0207 (19)	−0.0089 (18)
C22	0.0208 (18)	0.033 (2)	0.0158 (18)	−0.0055 (16)	0.0127 (16)	0.0003 (16)
C23	0.0150 (18)	0.038 (2)	0.023 (2)	0.0059 (17)	0.0117 (17)	0.0091 (18)
C24	0.032 (2)	0.034 (2)	0.032 (2)	0.0120 (18)	0.023 (2)	0.0108 (18)
C25	0.028 (2)	0.030 (2)	0.029 (2)	0.0023 (17)	0.0191 (19)	−0.0005 (17)
C26	0.0179 (18)	0.035 (2)	0.025 (2)	−0.0049 (17)	0.0130 (17)	−0.0060 (17)
C27	0.0186 (18)	0.027 (2)	0.0180 (18)	−0.0049 (15)	0.0121 (16)	−0.0049 (15)
C28	0.0174 (18)	0.033 (2)	0.0220 (19)	−0.0056 (16)	0.0128 (17)	−0.0100 (17)
C29	0.0201 (19)	0.037 (2)	0.033 (2)	−0.0040 (17)	0.0178 (19)	−0.0073 (18)
C30	0.023 (2)	0.031 (2)	0.044 (2)	−0.0042 (17)	0.024 (2)	−0.0092 (19)
C31	0.038 (7)	0.063 (8)	0.039 (9)	0.018 (6)	0.020 (6)	−0.006 (6)
C32	0.034 (5)	0.031 (5)	0.026 (5)	0.014 (4)	0.013 (4)	0.002 (4)
C33	0.040 (8)	0.102 (12)	0.034 (9)	0.029 (7)	0.022 (6)	0.004 (7)

Geometric parameters (Å, °)

Cl1—C1	1.744 (4)	C17—H17	0.9500
Cl2—C16	1.751 (4)	C18—C19	1.384 (5)
O1—C32	1.211 (10)	C18—H18	0.9500
N1—C7	1.418 (4)	C19—C20	1.402 (5)

N1—C4	1.420 (4)	C20—C21	1.397 (5)
N1—H1A	0.85 (4)	C21—H21	0.9500
N2—C13	1.280 (4)	C22—C23	1.387 (5)
N2—C5	1.403 (4)	C22—C27	1.405 (5)
N3—C13	1.396 (4)	C23—C24	1.378 (5)
N3—C15	1.462 (4)	C23—H23	0.9500
N3—C14	1.468 (4)	C24—C25	1.389 (5)
N4—C19	1.423 (5)	C24—H24	0.9500
N4—C22	1.424 (5)	C25—C26	1.381 (5)
N4—H4A	0.87 (4)	C25—H25	0.9500
N5—C28	1.291 (5)	C26—C27	1.396 (5)
N5—C20	1.404 (4)	C26—H26	0.9500
N6—C28	1.385 (4)	C27—C28	1.485 (5)
N6—C30	1.464 (5)	C29—C30^{ii}	1.524 (5)
N6—C29	1.472 (5)	C29—H29A	0.9900
C1—C2	1.379 (5)	C29—H29B	0.9900
C1—C6	1.385 (5)	C30—C29^{ii}	1.524 (5)
C2—C3	1.390 (5)	C30—H30A	0.9900
C2—H2	0.9500	C30—H30B	0.9900
C3—C4	1.395 (5)	C31—C32	1.4003 (10)
C3—H3	0.9500	C31—H31A	0.9800
C4—C5	1.397 (5)	C31—H31B	0.9800
C5—C6	1.403 (5)	C31—H31C	0.9800
C6—H6	0.9500	C32—C33	1.4008 (10)
C7—C8	1.393 (5)	C33—H33A	0.9800
C7—C12	1.406 (5)	C33—H33B	0.9800
C8—C9	1.384 (5)	C33—H33C	0.9800
C8—H8	0.9500	C34—C35	1.515 (10)
C9—C10	1.385 (5)	C34—H34A	0.9800
C9—H9	0.9500	C34—H34B	0.9800
C10—C11	1.381 (5)	C34—H34C	0.9800
C10—H10	0.9500	C35—C36	1.467 (10)
C11—C12	1.389 (5)	C35—H35A	0.9900
C11—H11	0.9500	C35—H35B	0.9900
C12—C13	1.491 (5)	C36—C37	1.450 (9)
C14—C14^{i}	1.522 (6)	C36—H36A	0.9900
C14—H14A	0.9900	C36—H36B	0.9900
C14—H14B	0.9900	C37—C38	1.534 (9)
C15—C15^{i}	1.514 (7)	C37—H37A	0.9900
C15—H15A	0.9900	C37—H37B	0.9900
C15—H15B	0.9900	C38—H38A	0.9800
C16—C21	1.374 (5)	C38—H38B	0.9800
C16—C17	1.385 (5)	C38—H38C	0.9800
C17—C18	1.383 (5)		
C7—N1—C4	116.3 (3)	C16—C21—H21	120.0
C7—N1—H1A	112 (3)	C20—C21—H21	120.0
C4—N1—H1A	113 (3)	C23—C22—C27	120.0 (3)
C13—N2—C5	123.7 (3)	C23—C22—N4	121.3 (3)

C13—N3—C15	115.9 (3)	C27—C22—N4	118.6 (3)
C13—N3—C14	120.3 (3)	C24—C23—C22	120.6 (3)
C15—N3—C14	111.1 (3)	C24—C23—H23	119.7
C19—N4—C22	113.2 (3)	C22—C23—H23	119.7
C19—N4—H4A	114 (2)	C23—C24—C25	120.1 (3)
C22—N4—H4A	107 (2)	C23—C24—H24	119.9
C28—N5—C20	123.3 (3)	C25—C24—H24	119.9
C28—N6—C30	118.4 (3)	C26—C25—C24	119.6 (4)
C28—N6—C29	121.3 (3)	C26—C25—H25	120.2
C30—N6—C29	111.5 (3)	C24—C25—H25	120.2
C2—C1—C6	121.1 (3)	C25—C26—C27	121.4 (3)
C2—C1—Cl1	120.1 (3)	C25—C26—H26	119.3
C6—C1—Cl1	118.8 (3)	C27—C26—H26	119.3
C1—C2—C3	119.1 (3)	C26—C27—C22	118.3 (3)
C1—C2—H2	120.5	C26—C27—C28	121.5 (3)
C3—C2—H2	120.5	C22—C27—C28	120.2 (3)
C2—C3—C4	120.8 (3)	N5—C28—N6	117.4 (3)
C2—C3—H3	119.6	N5—C28—C27	126.2 (3)
C4—C3—H3	119.6	N6—C28—C27	116.0 (3)
C3—C4—C5	119.8 (3)	N6—C29—C30^{ii}	110.3 (3)
C3—C4—N1	120.9 (3)	N6—C29—H29A	109.6
C5—C4—N1	119.2 (3)	C30^{ii}—C29—H29A	109.6
C4—C5—C6	119.0 (3)	N6—C29—H29B	109.6
C4—C5—N2	124.8 (3)	C30^{ii}—C29—H29B	109.6
C6—C5—N2	115.8 (3)	H29A—C29—H29B	108.1
C1—C6—C5	120.2 (3)	N6—C30—C29^{ii}	109.5 (3)
C1—C6—H6	119.9	N6—C30—H30A	109.8
C5—C6—H6	119.9	C29^{ii}—C30—H30A	109.8
C8—C7—C12	119.6 (3)	N6—C30—H30B	109.8
C8—C7—N1	121.1 (3)	C29^{ii}—C30—H30B	109.8
C12—C7—N1	119.3 (3)	H30A—C30—H30B	108.2
C9—C8—C7	120.2 (3)	C32—C31—H31A	109.5
C9—C8—H8	119.9	C32—C31—H31B	109.5
C7—C8—H8	119.9	H31A—C31—H31B	109.5
C8—C9—C10	120.6 (3)	C32—C31—H31C	109.5
C8—C9—H9	119.7	H31A—C31—H31C	109.5
C10—C9—H9	119.7	H31B—C31—H31C	109.5
C11—C10—C9	119.2 (4)	O1—C32—C31	119.4 (10)
C11—C10—H10	120.4	O1—C32—C33	117.2 (8)
C9—C10—H10	120.4	C31—C32—C33	123.4 (12)
C10—C11—C12	121.6 (3)	C32—C33—H33A	109.5
C10—C11—H11	119.2	C32—C33—H33B	109.5
C12—C11—H11	119.2	H33A—C33—H33B	109.5
C11—C12—C7	118.7 (3)	C32—C33—H33C	109.5
C11—C12—C13	121.0 (3)	H33A—C33—H33C	109.5
C7—C12—C13	120.3 (3)	H33B—C33—H33C	109.5
N2—C13—N3	116.5 (3)	C35—C34—H34A	109.5
N2—C13—C12	127.7 (3)	C35—C34—H34B	109.5

N3—C13—C12	115.6 (3)	H34A—C34—H34B	109.5
N3—C14—C14^{i}	110.0 (2)	C35—C34—H34C	109.5
N3—C14—H14A	109.7	H34A—C34—H34C	109.5
C14^{i}—C14—H14A	109.7	H34B—C34—H34C	109.5
N3—C14—H14B	109.7	C36—C35—C34	99.5 (8)
C14^{i}—C14—H14B	109.7	C36—C35—H35A	111.9
H14A—C14—H14B	108.2	C34—C35—H35A	111.9
N3—C15—C15^{i}	110.7 (3)	C36—C35—H35B	111.9
N3—C15—H15A	109.5	C34—C35—H35B	111.9
C15^{i}—C15—H15A	109.5	H35A—C35—H35B	109.6
N3—C15—H15B	109.5	C37—C36—C35	104.9 (9)
C15^{i}—C15—H15B	109.5	C37—C36—H36A	110.8
H15A—C15—H15B	108.1	C35—C36—H36A	110.8
C21—C16—C17	121.7 (4)	C37—C36—H36B	110.8
C21—C16—Cl2	119.3 (3)	C35—C36—H36B	110.8
C17—C16—Cl2	119.1 (3)	H36A—C36—H36B	108.8
C18—C17—C16	118.4 (3)	C36—C37—C38	100.1 (8)
C18—C17—H17	120.8	C36—C37—H37A	111.7
C16—C17—H17	120.8	C38—C37—H37A	111.7
C17—C18—C19	121.1 (4)	C36—C37—H37B	111.7
C17—C18—H18	119.5	C38—C37—H37B	111.7
C19—C18—H18	119.5	H37A—C37—H37B	109.5
C18—C19—C20	120.1 (3)	C37—C38—H38A	109.5
C18—C19—N4	121.8 (3)	C37—C38—H38B	109.5
C20—C19—N4	118.1 (3)	H38A—C38—H38B	109.5
C21—C20—C19	118.6 (3)	C37—C38—H38C	109.5
C21—C20—N5	117.5 (3)	H38A—C38—H38C	109.5
C19—C20—N5	123.4 (3)	H38B—C38—H38C	109.5
C16—C21—C20	120.0 (4)		

Symmetry codes: (i) −*x*, *y*, −*z*+1/2; (ii) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N5^{iii}	0.85 (4)	2.33 (4)	3.154 (4)	166 (3)

Symmetry codes: (iii) −*x*, −*y*, −*z*.

Fig. 1

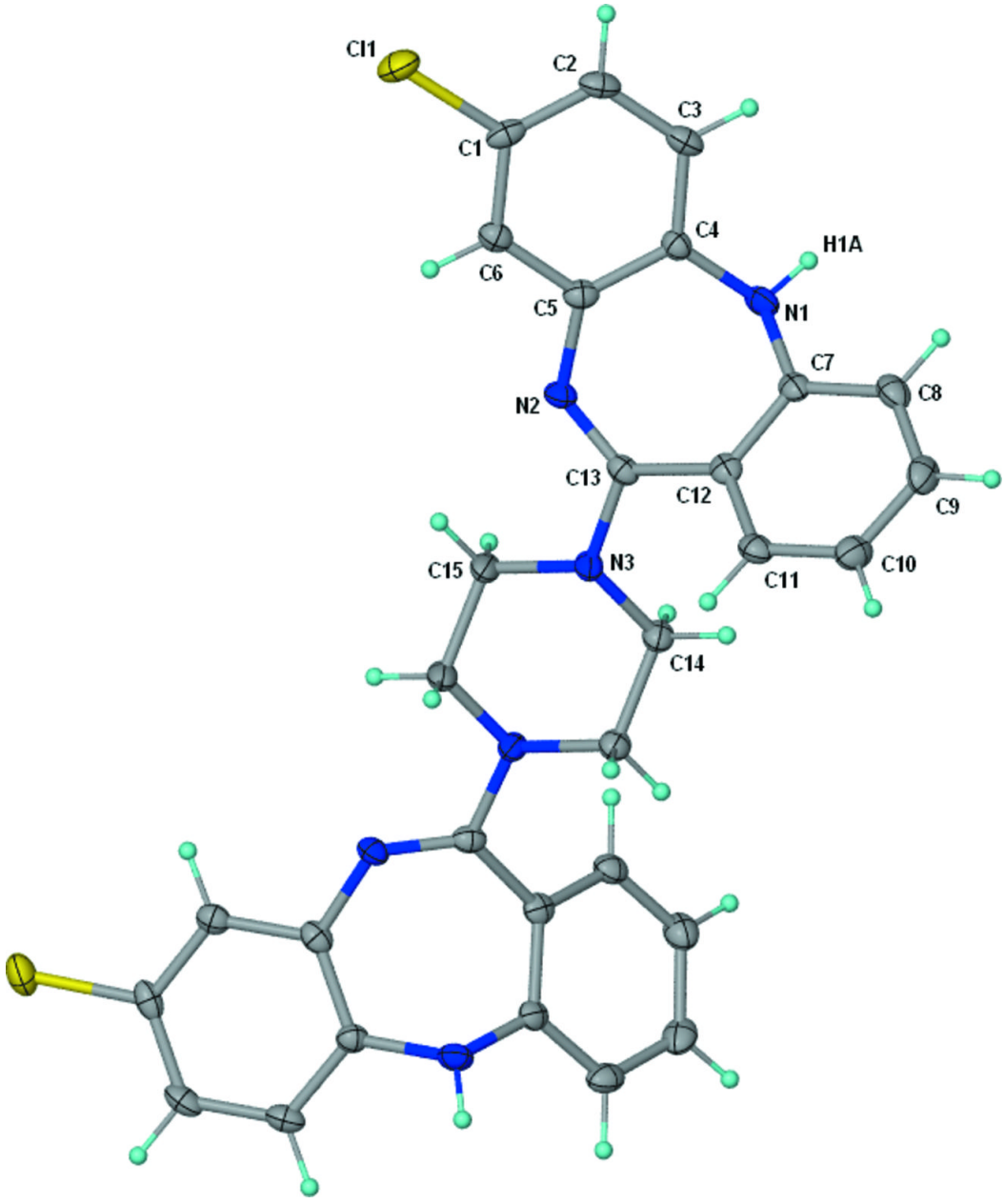
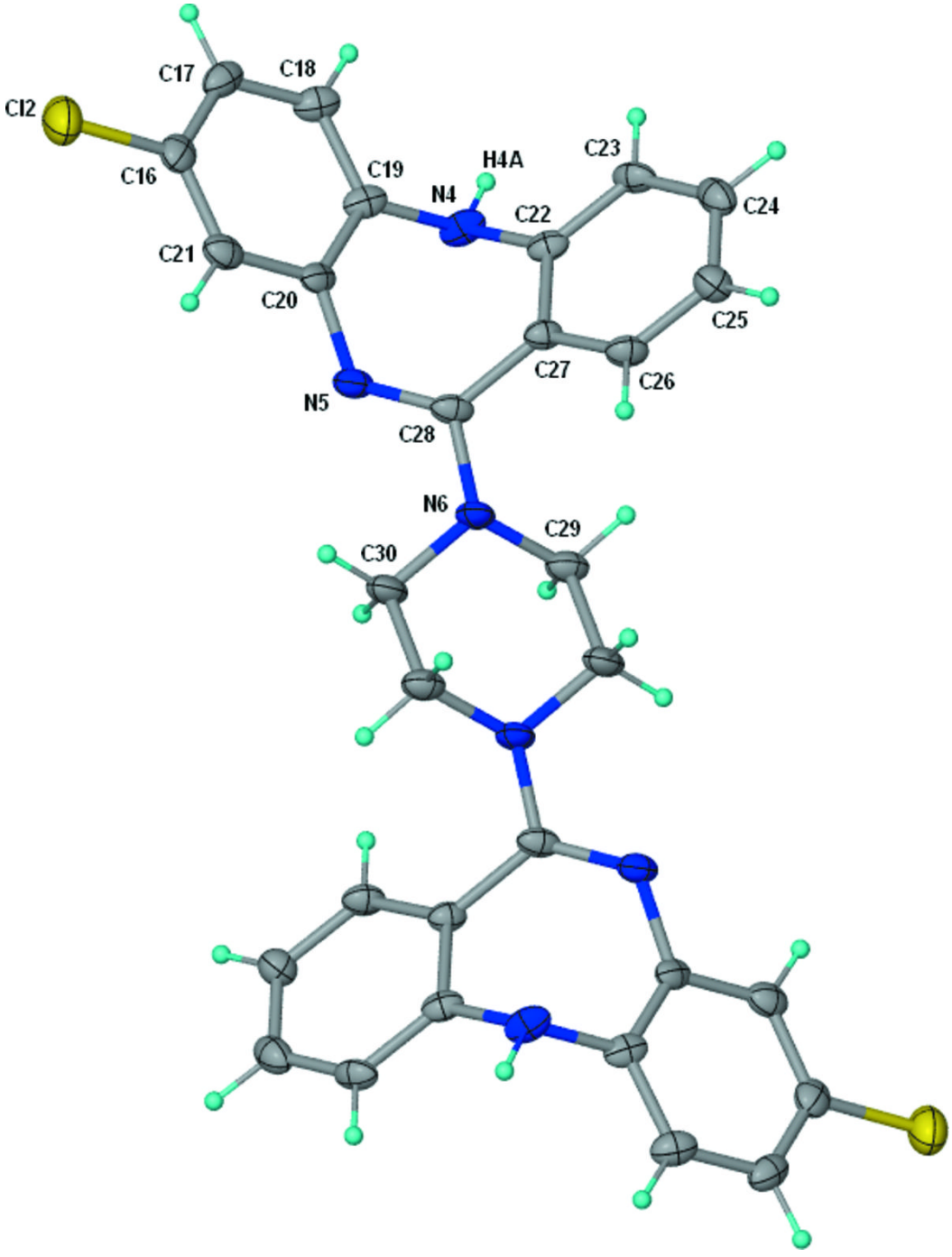


Fig. 2


C17
C18
Cl2
C16
C19
H4A
N4
C23
C22
C24
C21
C20
C25
C27
C26
N5
C28
N6
C29
C30