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7,8-Dichloro-1,2,3,4-tetrahydrophenazine

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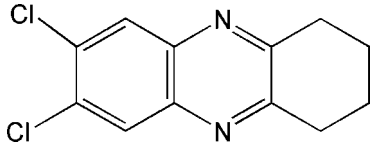
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.089; data-to-parameter ratio = 10.1.

In the structure of the title compound, $C_{12}H_{10}Cl_2N_2$, the cyclohexene ring adopts a half-chair conformation.

Related literature

For related literature, see: Brown *et al.* (2004); Farrugia (1997); Gibson *et al.* (2006); Page *et al.* (1998); Pascal & Ho (1993); Simpson & Gordon (1995); Willett *et al.* (2001); Wozniak *et al.* (1993); Wu *et al.* (2002).



Experimental

Crystal data

$C_{12}H_{10}Cl_2N_2$
M_r = 253.12
Triclinic, $P\overline{1}$
a = 6.3442 (3) Å
b = 7.3885 (3) Å
c = 11.7831 (5) Å
α = 85.720 (2)°
β = 82.122 (2)°
γ = 83.572 (2)°
V = 542.68 (4) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 5.13 mm^{-1}
T = 200 (2) K
0.35 × 0.21 × 0.08 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.267, T_{max} = 0.685
9473 measured reflections
1868 independent reflections
1810 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.05
1868 reflections
185 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *XSHELL* (Bruker, 2004); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RT2001).

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supplementary materials

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7,8-Dichloro-1,2,3,4-tetrahydrophenazine

C. H. Zambrano, R. D. Pike, R. J. P. Gibson, B. D. Barger and E. E. Dueno

Comment

One of the primary interests of our lab is the synthesis and characterization of novel substituted quinoxalines and the closely related phenazines. Quinoxalines and their derivatives have received considerable attention in the past several years due to their electronic properties (Page *et al.*, 1998; Simpson & Gordon, 1995), H-bonding ability (Pascal & Ho, 1993; Wozniak *et al.*, 1993), and their capacity to coordinate to metals (Wu *et al.*, 2002; Willett *et al.*, 2001). During our investigations, we have prepared a number of substituted quinoxalines and phenazines, which readily coordinate to copper iodide forming novel structures. Our current work involves the synthesis of new nitrogen heterocycles (Gibson, *et al.*, 2006) which may lead to novel three-dimensional structures upon coordination to cuprous salts. Here, we report the crystal structure of 7,8-dichloro-1,2,3,4-tetrahydrophenazine (I), (Figure 1).

The structure of (I) exhibits bond distances and angles that are normal, for all fall within ranges established in the literature for similar nitrogen heterocycles (Brown *et al.*, 2004). There are two molecules per unit cell, related to each other by an inversion center. The chloride substituents are almost eclipsed with respect to each other, with a torsion angle Cl1—C10—C9—Cl2 of 0.36 (16)°. Both aromatic rings are essentially planar and almost co-planar with a dihedral angle of 1.20 (18)°, based on least-squares plane calculations on C12—C11—C10—C9—C8—C7 and C7—C12—N1—C1—C6—N2. The H-saturated fragment of the ring system adopts a twisted, cyclohexyl-like conformation as evidenced by the angles depicted by atoms C5 C4 C3 110.68 (12)°, C2 C3 C4 109.92 (12)°, and the C2—C3—C4—C5 torsion angle of 63.62 (17)°, which suggest the presence of some angle and torsional strain.

Experimental

A 20 ml test tube was charged with 4,5-Dichloro-*o*-phenylenediamine (177 mg, 1 mmol) and 1,2-Cyclohexanedione (112 mg, 1 mmol). This was heated in a boiling water bath for 1 h, until the reaction mixture was homogeneous. The residue was then dissolved in boiling ethanol (100% EtOH, 15 ml). Upon cooling to 0° C, light yellow crystals of (1) were obtained (215 mg, 85% yield) mp 215–216° C.

Refinement

H atoms were treated as riding, with C—H = 1.00 with U_{iso}(H) = 1.2 U_{eq}(C) for all H atoms.

Figures

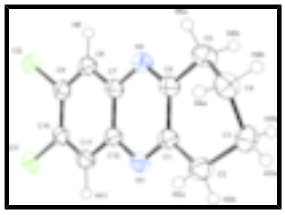


Fig. 1. *ORTEP* drawing of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres.

7,8-dichloro-1,2,3,4-tetrahydrophenazine

Crystal data

$C_{12}H_{10}Cl_2N_2$	$Z = 2$
$M_r = 253.12$	$F_{000} = 260$
Triclinic, $P\overline{1}$	$D_x = 1.549$ Mg m^{-3}
Hall symbol: -P 1	Cu $K\alpha$ radiation $\lambda = 1.54178$ Å
$a = 6.3442$ (3) Å	Cell parameters from 71 reflections
$b = 7.3885$ (3) Å	$\theta = 9.5$–41.0º
$c = 11.7831$ (5) Å	$\mu = 5.13$ mm^{-1}
$\alpha = 85.720$ (2)º	$T = 200$ (2) K
$\beta = 82.122$ (2)º	Block, yellow
$\gamma = 83.572$ (2)º	0.35 × 0.21 × 0.08 mm
$V = 542.68$ (4) Å^3	

Data collection

Bruker SMART APEX II CCD diffractometer	1868 independent reflections
Radiation source: fine-focus sealed tube	1810 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 200$(2) K	$\theta_{max} = 67.0$º
ω and ψ scans	$\theta_{min} = 3.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -7 \rightarrow 7$
$T_{min} = 0.267$, $T_{max} = 0.685$	$k = -8 \rightarrow 8$
9473 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	All H-atom parameters refined
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.0594P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
1868 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
185 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.93385 (5)	0.65523 (4)	0.28885 (3)	0.04023 (16)
Cl2	0.50704 (6)	0.83977 (5)	0.20124 (3)	0.04102 (16)
N1	0.54783 (19)	0.67203 (16)	0.69452 (11)	0.0335 (3)
N2	0.16607 (18)	0.84040 (15)	0.61792 (10)	0.0314 (3)
C1	0.3736 (2)	0.71175 (17)	0.76619 (11)	0.0313 (3)
C2	0.3837 (3)	0.6595 (2)	0.89145 (13)	0.0402 (3)
C3	0.2006 (2)	0.7516 (2)	0.97160 (12)	0.0395 (3)
C4	−0.0116 (2)	0.7388 (2)	0.92727 (12)	0.0390 (3)
C5	−0.0180 (2)	0.8423 (2)	0.81112 (13)	0.0382 (3)
C6	0.1794 (2)	0.79711 (17)	0.72703 (11)	0.0307 (3)
C7	0.3461 (2)	0.79900 (16)	0.54201 (11)	0.0294 (3)
C8	0.3393 (2)	0.83969 (17)	0.42346 (12)	0.0322 (3)
C9	0.5173 (2)	0.79438 (17)	0.34757 (11)	0.0322 (3)
C10	0.7083 (2)	0.71125 (16)	0.38628 (11)	0.0316 (3)
C11	0.7188 (2)	0.67350 (18)	0.50048 (12)	0.0340 (3)
C12	0.5361 (2)	0.71473 (17)	0.58085 (11)	0.0298 (3)
H8	0.202 (3)	0.896 (2)	0.3985 (14)	0.039 (4)*
H11	0.855 (3)	0.616 (2)	0.5315 (13)	0.037 (4)*
H2A	0.376 (3)	0.524 (3)	0.9028 (15)	0.047 (4)*
H3A	0.205 (3)	0.698 (2)	1.0494 (15)	0.037 (4)*
H4A	−0.025 (3)	0.613 (2)	0.9204 (13)	0.036 (4)*
H5A	−0.142 (3)	0.818 (3)	0.7777 (16)	0.050 (5)*
H2B	0.523 (3)	0.682 (3)	0.9123 (17)	0.057 (5)*
H3B	0.218 (3)	0.880 (2)	0.9745 (14)	0.043 (4)*
H4B	−0.127 (3)	0.781 (2)	0.9851 (15)	0.043 (4)*
H5B	−0.032 (3)	0.976 (2)	0.8240 (15)	0.046 (4)*

Atomic displacement parameters (Å^2)

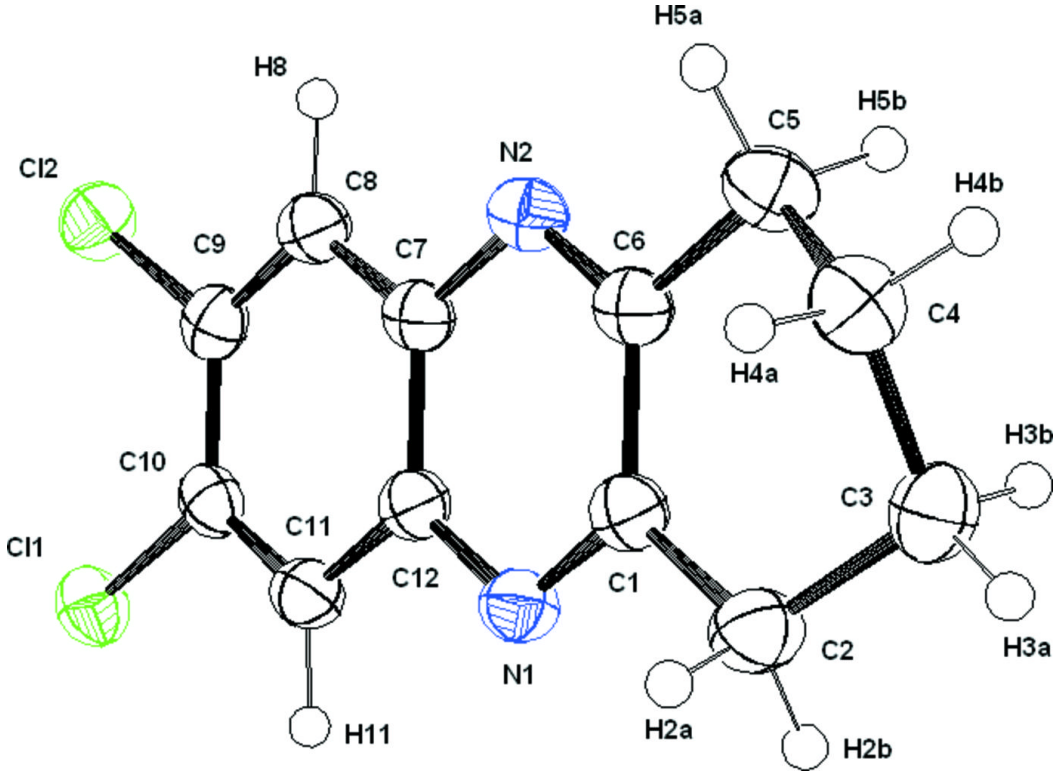
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0375 (2)	0.0407 (2)	0.0404 (2)	−0.00292 (16)	0.00363 (15)	−0.00642 (15)
Cl2	0.0478 (2)	0.0431 (2)	0.0313 (2)	−0.00231 (17)	−0.00522 (15)	0.00027 (15)
N1	0.0326 (6)	0.0321 (6)	0.0357 (6)	−0.0007 (5)	−0.0071 (5)	−0.0007 (5)
N2	0.0302 (6)	0.0283 (6)	0.0354 (6)	−0.0037 (5)	−0.0041 (5)	0.0023 (4)
C1	0.0332 (7)	0.0272 (6)	0.0337 (7)	−0.0040 (5)	−0.0056 (5)	0.0001 (5)
C2	0.0387 (8)	0.0460 (8)	0.0348 (7)	−0.0002 (7)	−0.0069 (6)	0.0016 (6)
C3	0.0451 (8)	0.0406 (8)	0.0329 (7)	−0.0077 (6)	−0.0032 (6)	−0.0014 (6)
C4	0.0387 (7)	0.0392 (8)	0.0373 (7)	−0.0057 (6)	0.0011 (6)	0.0008 (6)
C5	0.0336 (7)	0.0381 (8)	0.0402 (8)	−0.0006 (6)	−0.0004 (6)	0.0025 (6)
C6	0.0325 (6)	0.0242 (6)	0.0354 (7)	−0.0051 (5)	−0.0048 (5)	0.0013 (5)
C7	0.0297 (6)	0.0231 (6)	0.0358 (7)	−0.0046 (5)	−0.0051 (5)	0.0001 (5)
C8	0.0337 (7)	0.0274 (6)	0.0363 (7)	−0.0043 (6)	−0.0077 (5)	0.0009 (5)
C9	0.0398 (7)	0.0261 (6)	0.0318 (6)	−0.0078 (5)	−0.0059 (5)	0.0003 (5)
C10	0.0328 (6)	0.0246 (6)	0.0372 (7)	−0.0044 (5)	−0.0013 (5)	−0.0041 (5)
C11	0.0318 (7)	0.0301 (6)	0.0399 (7)	−0.0012 (6)	−0.0059 (6)	−0.0024 (5)
C12	0.0320 (6)	0.0249 (6)	0.0331 (6)	−0.0039 (5)	−0.0066 (5)	−0.0002 (5)

Geometric parameters (Å, °)

Cl1—C10	1.7394 (13)	C4—C5	1.520 (2)
Cl2—C9	1.7405 (13)	C4—H4A	0.955 (17)
N1—C1	1.3151 (19)	C4—H4B	0.970 (18)
N1—C12	1.3632 (19)	C5—C6	1.5090 (19)
N2—C6	1.3132 (18)	C5—H5A	0.97 (2)
N2—C7	1.3716 (18)	C5—H5B	1.001 (18)
C1—C6	1.4378 (19)	C7—C12	1.4104 (19)
C1—C2	1.506 (2)	C7—C8	1.412 (2)
C2—C3	1.524 (2)	C8—C9	1.368 (2)
C2—H2A	1.006 (19)	C8—H8	0.995 (18)
C2—H2B	0.99 (2)	C9—C10	1.413 (2)
C3—C4	1.524 (2)	C10—C11	1.363 (2)
C3—H3A	0.974 (18)	C11—C12	1.4133 (19)
C3—H3B	0.969 (18)	C11—H11	1.019 (17)
C1—N1—C12	116.95 (11)	C4—C5—H5A	110.3 (11)
C6—N2—C7	116.96 (12)	C6—C5—H5B	108.7 (10)
N1—C1—C6	121.78 (13)	C4—C5—H5B	107.7 (10)
N1—C1—C2	117.16 (12)	H5A—C5—H5B	108.0 (16)
C6—C1—C2	121.04 (13)	N2—C6—C1	121.97 (13)
C1—C2—C3	113.99 (13)	N2—C6—C5	117.29 (12)
C1—C2—H2A	107.4 (10)	C1—C6—C5	120.73 (13)
C3—C2—H2A	107.6 (10)	N2—C7—C12	120.84 (12)
C1—C2—H2B	110.1 (12)	N2—C7—C8	119.23 (12)
C3—C2—H2B	111.0 (12)	C12—C7—C8	119.92 (12)
H2A—C2—H2B	106.4 (16)	C9—C8—C7	119.21 (12)

C4—C3—C2	109.92 (12)	C9—C8—H8	122.7 (9)
C4—C3—H3A	112.9 (10)	C7—C8—H8	118.0 (9)
C2—C3—H3A	109.4 (10)	C8—C9—C10	120.99 (12)
C4—C3—H3B	107.7 (10)	C8—C9—Cl2	119.22 (10)
C2—C3—H3B	110.3 (10)	C10—C9—Cl2	119.79 (10)
H3A—C3—H3B	106.5 (14)	C11—C10—C9	120.61 (12)
C5—C4—C3	110.68 (12)	C11—C10—Cl1	118.89 (11)
C5—C4—H4A	109.9 (9)	C9—C10—Cl1	120.50 (10)
C3—C4—H4A	107.9 (10)	C10—C11—C12	119.75 (13)
C5—C4—H4B	113.5 (11)	C10—C11—H11	122.7 (9)
C3—C4—H4B	108.5 (10)	C12—C11—H11	117.6 (9)
H4A—C4—H4B	106.2 (14)	N1—C12—C7	121.50 (12)
C6—C5—C4	113.28 (12)	N1—C12—C11	119.01 (12)
C6—C5—H5A	108.7 (11)	C7—C12—C11	119.49 (12)
C12—N1—C1—C6	−0.29 (19)	C12—C7—C8—C9	0.80 (19)
C12—N1—C1—C2	178.13 (12)	C7—C8—C9—C10	−1.3 (2)
N1—C1—C2—C3	164.89 (13)	C7—C8—C9—Cl2	178.55 (9)
C6—C1—C2—C3	−16.7 (2)	C8—C9—C10—C11	0.3 (2)
C1—C2—C3—C4	46.64 (17)	Cl2—C9—C10—C11	−179.53 (10)
C2—C3—C4—C5	−63.51 (17)	C8—C9—C10—Cl1	−179.73 (9)
C3—C4—C5—C6	48.59 (17)	Cl2—C9—C10—Cl1	0.43 (15)
C7—N2—C6—C1	0.57 (19)	C9—C10—C11—C12	1.2 (2)
C7—N2—C6—C5	179.97 (11)	Cl1—C10—C11—C12	−178.78 (9)
N1—C1—C6—N2	−0.3 (2)	C1—N1—C12—C7	0.49 (19)
C2—C1—C6—N2	−178.61 (11)	C1—N1—C12—C11	−179.57 (12)
N1—C1—C6—C5	−179.63 (11)	N2—C7—C12—N1	−0.18 (19)
C2—C1—C6—C5	2.0 (2)	C8—C7—C12—N1	−179.40 (11)
C4—C5—C6—N2	162.46 (13)	N2—C7—C12—C11	179.88 (11)
C4—C5—C6—C1	−18.14 (19)	C8—C7—C12—C11	0.66 (19)
C6—N2—C7—C12	−0.36 (18)	C10—C11—C12—N1	178.41 (12)
C6—N2—C7—C8	178.87 (11)	C10—C11—C12—C7	−1.6 (2)
N2—C7—C8—C9	−178.43 (11)		

Fig. 1


H5a
H8
H5b
C5
N2
Cl2
C8
H4b
C9
C7
C6
C4
H4a
H3b
C10
C3
C11
Cl1
C12
C1
H3a
C2
N1
H2a
H2b
H11