Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,9-Dichloro-1,10-phenanthroline

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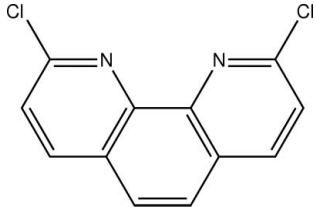
Received 25 March 2009; accepted 26 March 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.022; wR factor = 0.061; data-to-parameter ratio = 16.0.

The title molecule, $C_{12}H_6Cl_2N_2$, is almost planar (the r.m.s. deviation of C atoms is 0.04 Å). The C—N and C—C distances indicate delocalization of the π-electrons in the aromatic fused-ring system.

Related literature

For the synthesis, see: Yamada *et al.* (1990). The compound is used for the synthesis of other phenanthroline-like heterocycles; see: Hamilton *et al.* (2004); Ohira *et al.* (2005); Zong & Thummel (2004, 2005).



Experimental

Crystal data

$C_{12}H_6Cl_2N_2$
M_r = 249.09
Orthorhombic, $Pna2_1$
a = 19.4035 (3) Å
b = 4.4330 (1) Å
c = 11.7695 (2) Å
V = 1012.36 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 100 K
0.36 × 0.18 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.811, T_{max} = 0.988
8646 measured reflections
2315 independent reflections
2248 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.061
S = 1.02
2315 reflections
145 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1097 Friedel pairs
Flack parameter: −0.01 (4)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the Organization for the Prohibition of Chemical Weapons and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2404).

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supporting information

Acta Cryst. (2009). E**65**, o922 [doi:10.1107/S1600536809011180]

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S1. Experimental

A mixture of 6,7-dihydro-3*H*-[1,4]diazepino[1,2,3,4-*lmn*][1,10]phenanthroline-3,9(5*H*)-dione (1.7 g, 6.7 mmol) and phosphorus pentachloride (3 g, 14.4 mmol) was reacted in thionyl chloride (20 ml, 170 mmol) for 16 h at room temperature. The thionyl chloride was removed under reduced pressure and the residue was suspended in cold ammonium hydroxide. A light-tan precipitate was formed which was dissolved in hot benzene; crystals were obtained upon recrystallization from benzene (1.1 g 65%)

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C).

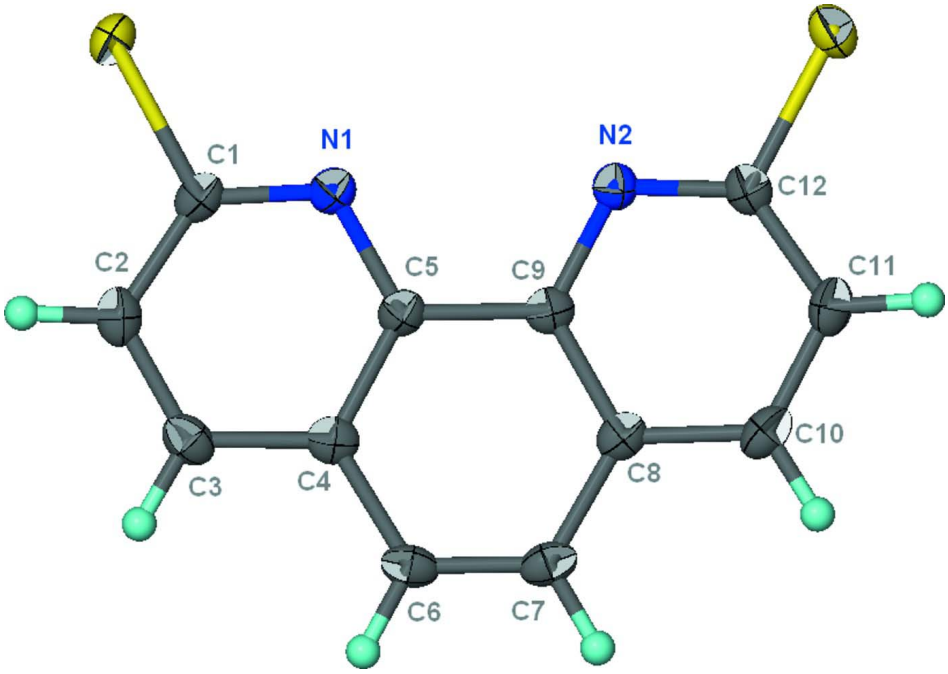

Figure 1

Thermal ellipsoid plot (Barbour, 2001) plot of $C_{12}H_6Cl_2N_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2,9-Dichloro-1,10-phenanthroline

Crystal data

$C_{12}H_6Cl_2N_2$
$M_r = 249.09$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 19.4035$ (3) Å
$b = 4.4330$ (1) Å
$c = 11.7695$ (2) Å
$V = 1012.36$ (3) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.634$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5870 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.61$ mm^{-1}
$T = 100$ K
Plate, colourless
0.36 × 0.18 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.811$, $T_{max} = 0.988$
8646 measured reflections
2315 independent reflections
2248 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -25 \rightarrow 25$
$k = -5 \rightarrow 5$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.061$
$S = 1.02$
2315 reflections
145 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack (1983), 1097 Friedel pairs
Absolute structure parameter: −0.01 (4)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.027691 (18)	0.55270 (8)	0.25010 (3)	0.01876 (10)
Cl2	0.35805 (2)	0.89704 (8)	0.45699 (4)	0.02052 (10)
N1	0.11803 (7)	0.4349 (3)	0.40752 (11)	0.0151 (3)
N2	0.24727 (7)	0.5756 (3)	0.48930 (11)	0.0153 (3)
C1	0.05604 (8)	0.3672 (3)	0.37354 (12)	0.0158 (3)
C2	0.01038 (8)	0.1645 (4)	0.42492 (13)	0.0183 (3)
H2	−0.0341	0.1269	0.3945	0.022*
C3	0.03326 (8)	0.0226 (4)	0.52172 (14)	0.0184 (3)
H3	0.0046	−0.1192	0.5595	0.022*
C4	0.09941 (8)	0.0884 (3)	0.56464 (14)	0.0159 (3)
C5	0.14049 (8)	0.2979 (4)	0.50487 (12)	0.0150 (3)
C6	0.12494 (9)	−0.0487 (3)	0.66693 (14)	0.0178 (3)

H6	0.0980	−0.1968	0.7048	0.021*
C7	0.18689 (8)	0.0307 (4)	0.70984 (13)	0.0176 (3)
H7	0.2025	−0.0587	0.7785	0.021*
C8	0.22933 (8)	0.2478 (3)	0.65313 (13)	0.0158 (3)
C9	0.20767 (8)	0.3756 (3)	0.54864 (13)	0.0150 (3)
C10	0.29315 (8)	0.3417 (4)	0.69870 (14)	0.0189 (3)
H10	0.3088	0.2647	0.7695	0.023*
C11	0.33217 (9)	0.5458 (4)	0.63927 (14)	0.0195 (3)
H11	0.3752	0.6152	0.6676	0.023*
C12	0.30611 (7)	0.6483 (3)	0.53484 (13)	0.0164 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01835 (17)	0.02130 (17)	0.01663 (17)	0.00087 (13)	−0.00441 (14)	−0.00032 (15)
Cl2	0.01900 (17)	0.02184 (16)	0.02072 (17)	−0.00573 (14)	0.00151 (15)	−0.00218 (15)
N1	0.0165 (6)	0.0163 (6)	0.0124 (6)	0.0010 (5)	0.0008 (5)	−0.0019 (5)
N2	0.0154 (6)	0.0158 (6)	0.0146 (6)	0.0001 (5)	0.0000 (5)	−0.0014 (4)
C1	0.0173 (7)	0.0174 (7)	0.0126 (7)	0.0046 (6)	−0.0004 (6)	−0.0028 (5)
C2	0.0154 (7)	0.0197 (7)	0.0199 (8)	0.0007 (6)	−0.0002 (6)	−0.0062 (6)
C3	0.0183 (8)	0.0186 (7)	0.0182 (8)	−0.0024 (6)	0.0051 (6)	−0.0023 (6)
C4	0.0192 (7)	0.0150 (7)	0.0136 (7)	0.0014 (5)	0.0028 (6)	−0.0030 (5)
C5	0.0158 (7)	0.0170 (7)	0.0121 (6)	0.0017 (5)	0.0013 (5)	−0.0018 (6)
C6	0.0220 (8)	0.0167 (7)	0.0146 (7)	0.0000 (6)	0.0060 (7)	0.0008 (6)
C7	0.0231 (8)	0.0177 (7)	0.0122 (6)	0.0047 (6)	0.0017 (6)	0.0004 (6)
C8	0.0181 (7)	0.0161 (7)	0.0132 (7)	0.0036 (6)	0.0001 (6)	−0.0022 (5)
C9	0.0178 (7)	0.0143 (6)	0.0130 (7)	0.0015 (5)	0.0014 (6)	−0.0027 (6)
C10	0.0212 (8)	0.0212 (8)	0.0145 (7)	0.0056 (6)	−0.0033 (6)	−0.0020 (6)
C11	0.0174 (8)	0.0223 (8)	0.0187 (8)	0.0003 (6)	−0.0041 (6)	−0.0052 (6)
C12	0.0156 (7)	0.0170 (7)	0.0166 (7)	−0.0003 (6)	0.0022 (6)	−0.0030 (6)

Geometric parameters (Å, º)

Cl1—C1	1.758 (2)	C4—C6	1.437 (2)
Cl2—C12	1.752 (2)	C5—C9	1.443 (2)
N1—C1	1.303 (2)	C6—C7	1.350 (2)
N1—C5	1.368 (2)	C6—H6	0.9500
N2—C12	1.302 (2)	C7—C8	1.432 (2)
N2—C9	1.365 (2)	C7—H7	0.9500
C1—C2	1.399 (2)	C8—C10	1.412 (2)
C2—C3	1.375 (2)	C8—C9	1.418 (2)
C2—H2	0.9500	C10—C11	1.372 (2)
C3—C4	1.410 (2)	C10—H10	0.9500
C3—H3	0.9500	C11—C12	1.405 (2)
C4—C5	1.412 (2)	C11—H11	0.9500
C1—N1—C5	116.68 (13)	C4—C6—H6	119.7
C12—N2—C9	116.36 (14)	C6—C7—C8	120.86 (14)

N1—C1—C2	126.86 (14)	C6—C7—H7	119.6
N1—C1—Cl1	115.78 (12)	C8—C7—H7	119.6
C2—C1—Cl1	117.36 (12)	C10—C8—C9	118.15 (14)
C3—C2—C1	116.59 (14)	C10—C8—C7	121.70 (14)
C3—C2—H2	121.7	C9—C8—C7	120.16 (14)
C1—C2—H2	121.7	N2—C9—C8	122.43 (14)
C2—C3—C4	119.74 (15)	N2—C9—C5	118.74 (14)
C2—C3—H3	120.1	C8—C9—C5	118.81 (14)
C4—C3—H3	120.1	C11—C10—C8	118.99 (15)
C3—C4—C5	118.14 (15)	C11—C10—H10	120.5
C3—C4—C6	121.77 (15)	C8—C10—H10	120.5
C5—C4—C6	120.08 (14)	C10—C11—C12	117.45 (15)
N1—C5—C4	121.97 (14)	C10—C11—H11	121.3
N1—C5—C9	118.72 (14)	C12—C11—H11	121.3
C4—C5—C9	119.29 (14)	N2—C12—C11	126.57 (15)
C7—C6—C4	120.66 (15)	N2—C12—Cl2	116.43 (12)
C7—C6—H6	119.7	C11—C12—Cl2	117.00 (12)

C5—N1—C1—C2	−1.3 (2)	C12—N2—C9—C8	−1.2 (2)
C5—N1—C1—Cl1	178.60 (10)	C12—N2—C9—C5	177.09 (13)
N1—C1—C2—C3	0.2 (2)	C10—C8—C9—N2	2.5 (2)
Cl1—C1—C2—C3	−179.66 (12)	C7—C8—C9—N2	−177.70 (13)
C1—C2—C3—C4	0.8 (2)	C10—C8—C9—C5	−175.77 (13)
C2—C3—C4—C5	−0.8 (2)	C7—C8—C9—C5	4.0 (2)
C2—C3—C4—C6	178.52 (14)	N1—C5—C9—N2	−2.1 (2)
C1—N1—C5—C4	1.3 (2)	C4—C5—C9—N2	179.17 (13)
C1—N1—C5—C9	−177.33 (14)	N1—C5—C9—C8	176.22 (13)
C3—C4—C5—N1	−0.4 (2)	C4—C5—C9—C8	−2.5 (2)
C6—C4—C5—N1	−179.63 (14)	C9—C8—C10—C11	−1.5 (2)
C3—C4—C5—C9	178.30 (14)	C7—C8—C10—C11	178.68 (15)
C6—C4—C5—C9	−1.0 (2)	C8—C10—C11—C12	−0.5 (2)
C3—C4—C6—C7	−176.22 (15)	C9—N2—C12—C11	−1.1 (2)
C5—C4—C6—C7	3.0 (2)	C9—N2—C12—Cl2	178.34 (10)
C4—C6—C7—C8	−1.5 (2)	C10—C11—C12—N2	2.0 (2)
C6—C7—C8—C10	177.72 (15)	C10—C11—C12—Cl2	−177.45 (12)
C6—C7—C8—C9	−2.1 (2)		