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2,9-Dichloro-1,10-phenanthroline

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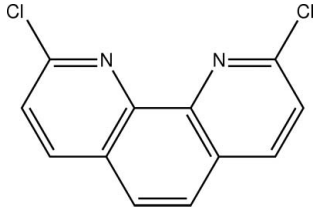
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.022; wR factor = 0.061; data-to-parameter ratio = 16.0.

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).

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

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supplementary materials

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2,9-Dichloro-1,10-phenanthroline

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Comment

(type here to add)

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%)

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).

Figures

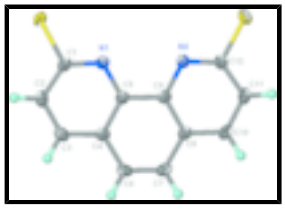

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of $C_{12}H_6Cl_2N_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2,9-Dichloro-1,10-phenanthroline

Crystal data

$C_{12}H_6Cl_2N_2$	F_{000} = 504
M_r = 249.09	D_x = 1.634 Mg m^{-3}
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 5870 reflections
a = 19.4035 (3) Å	θ = 2.7–28.3°
b = 4.4330 (1) Å	μ = 0.61 mm^{-1}
c = 11.7695 (2) Å	*T* = 100 K
V = 1012.36 (3) Å^3	Plate, colourless

$Z = 4$	0.36 × 0.18 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer	2315 independent reflections
Radiation source: fine-focus sealed tube	2248 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 100$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.1°$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -25\rightarrow25$
$T_{min} = 0.811$, $T_{max} = 0.988$	$k = -5\rightarrow5$
8646 measured reflections	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
2315 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
145 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1097 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.01 (4)
Secondary atom site location: difference Fourier map	

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

	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 (Å^2)

	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, °)

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)		

Fig. 1