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N^1,N^2-Bis[(2-chloro-6-methylquinolin-3-yl)methylidene]ethane-1,2-diamine

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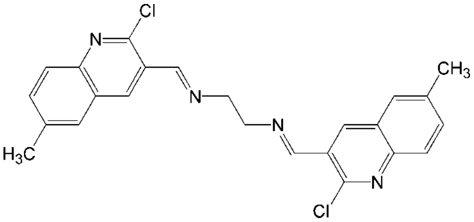
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.168; data-to-parameter ratio = 14.8.

The title molecule, $C_{24}H_{20}Cl_2N_4$, lies on an inversion center in an extended *trans* conformation. In the crystal, weak C—H···Cl interactions connect the molecules into chains along [010].

Related literature

For general background to Schiff bases, see: Schiff (1864); Huiyan *et al.* (2009); Kano *et al.* (2003); Liu *et al.* (2010); Salhi *et al.* (2009); Wang *et al.* (2008); Yong & Zheng (2009). For related structures, see: Assey *et al.* (2010); Dipesh *et al.* (2007).



Experimental

Crystal data

$C_{24}H_{20}Cl_2N_4$
M_r = 435.34
Triclinic, $P\overline{1}$
a = 4.4088 (8) Å
b = 7.2008 (11) Å
c = 16.9383 (18) Å
α = 84.236 (11)°
β = 87.924 (12)°
γ = 78.698 (14)°
V = 524.57 (14) Å^3
Z = 1
Cu $K\alpha$ radiation
μ = 2.93 mm^{-1}
T = 295 K
0.46 × 0.37 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Ruby Gemini detector
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
T_{min} = 0.378, T_{max} = 1.000
3137 measured reflections
2011 independent reflections
1710 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.168
S = 1.05
2011 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···Cl^{i}	0.93	2.86	3.780 (2)	170

Symmetry code: (i) $x, y-1, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

RJB wishes to acknowledge the NSF-MRI program (grant CHE-0619278) for funds to purchase the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5149).

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supplementary materials

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N^1,N^2-Bis[(2-chloro-6-methylquinolin-3-yl)methylidene]ethane-1,2-diamine

R. Prasath, P. Bhavana, A. M. Butcher, R. J. Butcher and **J. P. Jasinski**

Comment

Quinoline Schiff base complexes are important class of compounds owing to their applications in the fields of environmental (Salhi *et al.*, 2009), catalytic (Kano *et al.*, 2003), DNA binding (Yong *et al.*, 2009) and polymeric applications (Huiyan *et al.*, 2009). Quinoline appended Schiff base complexes are also known for their photophysical properties (Liu *et al.*, 2010; Wang *et al.*, 2008). Related structures have already appeared in the literature (Assey *et al.*, 2010; Dipesh *et al.*, 2007). Herein we report the synthesis and crystal structure of the title compound, (I).

In the title compound, $C_{24}H_{20}Cl_2N_4$, the molecule is in an extended *trans* conformation and is located on a center of inversion between C12 and C12(-x, 1-y, -z). In the crystal structure, weak C—H···Cl interactions connect molecules into chains along [010].

Experimental

A mixture of 2-chloro-3-formyl-6-methylquinoline (0.2 g, 1 m*M*) and ethylenediamine (0.03 ml, 0.5 m*M*) was stirred in dichloromethane for 3 h at room temperature. The solvent from the reaction mixture was removed under reduced pressure, and the resulting solid was dried and purified by column chromatography using a 1:3 mixture of ethyl acetate and hexane. Recrystallization was by slow evaporation of a dichloromethane solution of (I) which yielded white coloured needle type crystals. *M*.p. 485–487 K. Yield: 83%.

Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with a C—H distances of 0.93, 0.96 and 0.97 Å; $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$.

Figures

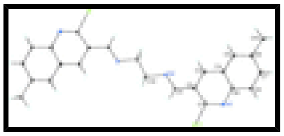

Fig. 1. The molecular structure of the title compound with unique part of molecule labeled. Ellipsoids drawn at 30% probability level. The unlabeled atoms are related by the symmetry operator (-x, 1-y, -z).

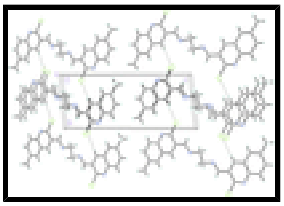

Fig. 2. Part of the crystal structure viewed along the *a* axis showing the intermolecular C—H···Cl interactions as dashed lines.

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Crystal data

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$M_r = 435.34$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 4.4088$ (8) Å

$b = 7.2008$ (11) Å

$c = 16.9383$ (18) Å

$\alpha = 84.236$ (11)°

$\beta = 87.924$ (12)°

$\gamma = 78.698$ (14)°

$V = 524.57$ (14) Å^3

$Z = 1$

$F(000) = 226$

$D_x = 1.378$ Mg m^{-3}

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 2032 reflections

$\theta = 5.3$–73.4°

$\mu = 2.93$ mm^{-1}

$T = 295$ K

Plate, colorless

0.46 × 0.37 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Ruby Gemini detector

Radiation source: Enhance (Cu) X-ray Source

graphite

Detector resolution: 10.5081 pixels mm^{-1}

ω scans

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)

$T_{\text{min}} = 0.378$, $T_{\text{max}} = 1.000$

3137 measured reflections

2011 independent reflections

1710 reflections with $I > 2\sigma(I)$

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

$\theta_{\text{max}} = 73.6°$, $\theta_{\text{min}} = 5.3°$

$h = -5\rightarrow5$

$k = -8\rightarrow8$

$l = -20\rightarrow21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.168$

$S = 1.05$

2011 reflections

136 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.1105P)^2 + 0.063P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.44$ e Å^{-3}

$\Delta\rho_{\text{min}} = -0.29$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
Cl	0.37544 (18)	1.10262 (8)	0.15067 (4)	0.0756 (3)
N1	0.7058 (5)	0.8882 (3)	0.25986 (11)	0.0533 (5)
N2	0.2488 (4)	0.5727 (3)	0.07293 (11)	0.0532 (5)
C1	0.5343 (5)	0.8821 (3)	0.20006 (13)	0.0500 (5)
C2	0.4730 (5)	0.7140 (3)	0.17101 (12)	0.0459 (5)
C3	0.6152 (5)	0.5444 (3)	0.21010 (12)	0.0463 (5)
H3A	0.5844	0.4305	0.1934	0.056*
C4	0.8067 (5)	0.5410 (3)	0.27498 (12)	0.0449 (5)
C5	0.9619 (5)	0.3710 (3)	0.31671 (13)	0.0507 (5)
H5A	0.9386	0.2546	0.3009	0.061*
C6	1.1455 (5)	0.3738 (3)	0.37978 (13)	0.0540 (5)
C7	1.1733 (6)	0.5531 (4)	0.40297 (14)	0.0597 (6)
H7A	1.2940	0.5570	0.4464	0.072*
C8	1.0298 (6)	0.7198 (3)	0.36399 (14)	0.0579 (6)
H8A	1.0559	0.8348	0.3806	0.069*
C9	0.8427 (5)	0.7190 (3)	0.29892 (12)	0.0470 (5)
C10	1.3155 (7)	0.1939 (4)	0.42378 (16)	0.0696 (7)
H10A	1.2724	0.0859	0.4004	0.104*
H10B	1.2489	0.1886	0.4784	0.104*
H10C	1.5339	0.1922	0.4206	0.104*
C11	0.2710 (5)	0.7195 (3)	0.10311 (13)	0.0498 (5)
H11A	0.1572	0.8358	0.0823	0.060*
C12	0.0528 (5)	0.5922 (3)	0.00365 (13)	0.0517 (5)
H12A	0.1677	0.6255	−0.0439	0.062*
H12B	−0.1257	0.6933	0.0090	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.1104 (6)	0.0382 (4)	0.0809 (5)	−0.0165 (3)	−0.0379 (4)	−0.0011 (3)
N1	0.0706 (11)	0.0410 (10)	0.0537 (10)	−0.0204 (8)	−0.0112 (9)	−0.0083 (7)
N2	0.0627 (11)	0.0470 (10)	0.0525 (10)	−0.0123 (8)	−0.0189 (8)	−0.0078 (8)

C1	0.0654 (13)	0.0386 (11)	0.0499 (11)	−0.0168 (9)	−0.0107 (9)	−0.0064 (8)
C2	0.0540 (11)	0.0426 (11)	0.0454 (10)	−0.0174 (8)	−0.0046 (8)	−0.0085 (8)
C3	0.0602 (12)	0.0397 (10)	0.0450 (10)	−0.0212 (9)	−0.0062 (9)	−0.0086 (8)
C4	0.0556 (11)	0.0415 (10)	0.0421 (10)	−0.0185 (8)	−0.0041 (8)	−0.0068 (8)
C5	0.0639 (13)	0.0427 (11)	0.0497 (11)	−0.0183 (9)	−0.0072 (9)	−0.0063 (9)
C6	0.0629 (13)	0.0520 (13)	0.0494 (11)	−0.0170 (10)	−0.0066 (9)	−0.0024 (9)
C7	0.0728 (14)	0.0613 (14)	0.0507 (12)	−0.0221 (11)	−0.0205 (10)	−0.0085 (10)
C8	0.0767 (15)	0.0493 (12)	0.0550 (12)	−0.0235 (10)	−0.0149 (11)	−0.0132 (9)
C9	0.0592 (11)	0.0425 (11)	0.0444 (10)	−0.0190 (8)	−0.0036 (8)	−0.0097 (8)
C10	0.0848 (17)	0.0598 (15)	0.0646 (15)	−0.0155 (13)	−0.0223 (13)	0.0021 (12)
C11	0.0599 (12)	0.0417 (11)	0.0499 (11)	−0.0125 (9)	−0.0124 (9)	−0.0047 (8)
C12	0.0600 (12)	0.0471 (12)	0.0498 (11)	−0.0117 (9)	−0.0158 (9)	−0.0053 (9)

Geometric parameters (Å, °)

Cl—C1	1.747 (2)	C6—C7	1.415 (3)
N1—C1	1.295 (3)	C6—C10	1.504 (3)
N1—C9	1.366 (3)	C7—C8	1.361 (4)
N2—C11	1.242 (3)	C7—H7A	0.9300
N2—C12	1.462 (3)	C8—C9	1.401 (3)
C1—C2	1.428 (3)	C8—H8A	0.9300
C2—C3	1.375 (3)	C10—H10A	0.9600
C2—C11	1.473 (3)	C10—H10B	0.9600
C3—C4	1.405 (3)	C10—H10C	0.9600
C3—H3A	0.9300	C11—H11A	0.9300
C4—C5	1.414 (3)	C12—C12^{i}	1.508 (4)
C4—C9	1.422 (3)	C12—H12A	0.9700
C5—C6	1.368 (3)	C12—H12B	0.9700
C5—H5A	0.9300		

C1—N1—C9	117.53 (17)	C6—C7—H7A	118.8
C11—N2—C12	117.90 (19)	C7—C8—C9	120.3 (2)
N1—C1—C2	126.0 (2)	C7—C8—H8A	119.9
N1—C1—Cl	115.36 (15)	C9—C8—H8A	119.9
C2—C1—Cl	118.65 (16)	N1—C9—C8	119.18 (18)
C3—C2—C1	116.02 (18)	N1—C9—C4	122.22 (19)
C3—C2—C11	121.40 (18)	C8—C9—C4	118.6 (2)
C1—C2—C11	122.6 (2)	C6—C10—H10A	109.5
C2—C3—C4	120.86 (18)	C6—C10—H10B	109.5
C2—C3—H3A	119.6	H10A—C10—H10B	109.5
C4—C3—H3A	119.6	C6—C10—H10C	109.5
C3—C4—C5	123.30 (18)	H10A—C10—H10C	109.5
C3—C4—C9	117.38 (19)	H10B—C10—H10C	109.5
C5—C4—C9	119.33 (18)	N2—C11—C2	121.6 (2)
C6—C5—C4	121.50 (19)	N2—C11—H11A	119.2
C6—C5—H5A	119.3	C2—C11—H11A	119.2
C4—C5—H5A	119.3	N2—C12—C12^{i}	109.9 (2)
C5—C6—C7	117.9 (2)	N2—C12—H12A	109.7
C5—C6—C10	121.9 (2)	C12^{i}—C12—H12A	109.7

C7—C6—C10	120.2 (2)	N2—C12—H12B	109.7
C8—C7—C6	122.4 (2)	C12^{i}—C12—H12B	109.7
C8—C7—H7A	118.8	H12A—C12—H12B	108.2
C9—N1—C1—C2	0.3 (4)	C10—C6—C7—C8	178.5 (2)
C9—N1—C1—Cl	−178.07 (16)	C6—C7—C8—C9	0.9 (4)
N1—C1—C2—C3	−1.1 (4)	C1—N1—C9—C8	−179.9 (2)
Cl—C1—C2—C3	177.21 (16)	C1—N1—C9—C4	1.2 (3)
N1—C1—C2—C11	179.1 (2)	C7—C8—C9—N1	−178.8 (2)
Cl—C1—C2—C11	−2.6 (3)	C7—C8—C9—C4	0.1 (4)
C1—C2—C3—C4	0.4 (3)	C3—C4—C9—N1	−1.8 (3)
C11—C2—C3—C4	−179.78 (19)	C5—C4—C9—N1	178.25 (19)
C2—C3—C4—C5	−179.1 (2)	C3—C4—C9—C8	179.34 (19)
C2—C3—C4—C9	0.9 (3)	C5—C4—C9—C8	−0.6 (3)
C3—C4—C5—C6	−179.8 (2)	C12—N2—C11—C2	−177.51 (19)
C9—C4—C5—C6	0.2 (3)	C3—C2—C11—N2	−8.3 (3)
C4—C5—C6—C7	0.8 (3)	C1—C2—C11—N2	171.5 (2)
C4—C5—C6—C10	−179.0 (2)	C11—N2—C12—C12^{i}	−156.5 (2)
C5—C6—C7—C8	−1.3 (4)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3A···Cl^{ii}	0.93	2.86	3.780 (2)	170

Symmetry codes: (ii) x, $y-1$, z.

Fig. 1

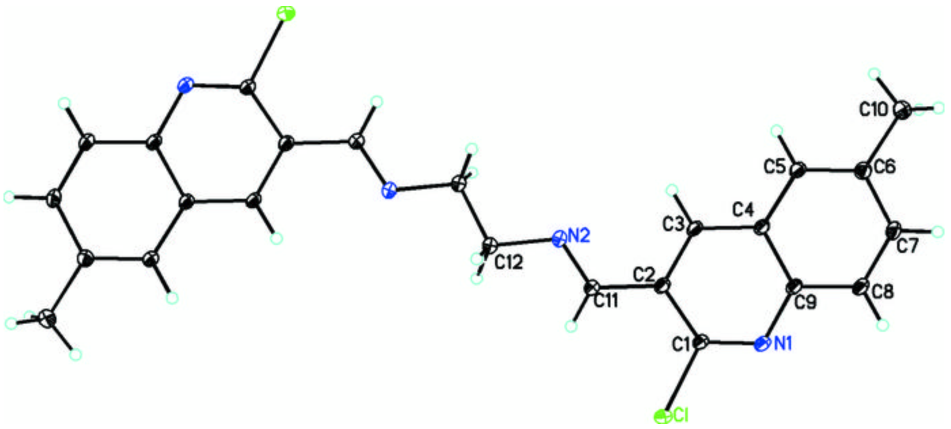

Fig. 2