

N,N'-Bis-(2,4-dichlorobenzylidene)-2,2-dimethylpropane-1,3-diamine

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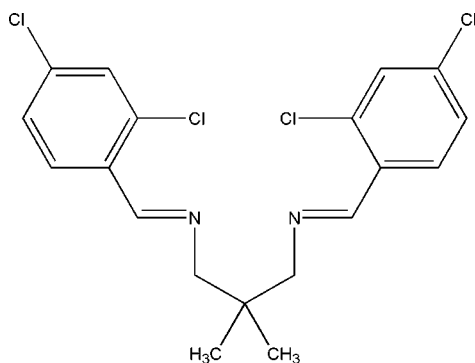
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.030; wR factor = 0.099; data-to-parameter ratio = 43.4.

The molecule of the title Schiff base compound, $\text{C}_{19}\text{H}_{18}\text{Cl}_4\text{N}_2$, has crystallographic twofold rotation symmetry, with one C atom lying on the rotation axis. The dihedral angle between the two symmetry-related benzene rings is $84.70(2)^\circ$. The plane of the $-\text{C}=\text{N}-\text{C}-$ group is twisted away from the benzene ring by $7.5(1)^\circ$. In the crystal structure, weak intermolecular $\text{Cl}\cdots\text{Cl}$ [$3.4851(3)$ Å] contacts link neighbouring molecules into a two-dimensional network parallel to the bc plane.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Li *et al.* (2005); Bomfim *et al.* (2005); Glidewell *et al.* (2005, 2006); Sun *et al.* (2004); Fun *et al.* (2008).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{18}\text{Cl}_4\text{N}_2$	$V = 1903.05(5)$ Å ³
$M_r = 416.15$	$Z = 4$
Orthorhombic, <i>Pcca</i>	Mo $K\alpha$ radiation
$a = 30.7633(4)$ Å	$\mu = 0.63$ mm ⁻¹
$b = 5.4012(1)$ Å	$T = 100.0(1)$ K
$c = 11.4532(1)$ Å	$0.43 \times 0.25 \times 0.23$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	86683 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2005)	6565 independent reflections
$T_{\min} = 0.774$, $T_{\max} = 0.872$	5678 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	115 parameters
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.19$	$\Delta\rho_{\max} = 0.51$ e Å ⁻³
4992 reflections	$\Delta\rho_{\min} = -0.30$ e Å ⁻³

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2702).

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supplementary materials

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Comment

Schiff bases are one of most prevalent mixed-donor ligands in the field of coordination chemistry. They play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism, and supramolecular architectures. Crystal structures of Schiff bases derived from substituted benzaldehydes and closely related to the title compound have been reported earlier (Li *et al.*, 2005; Bomfim *et al.*, 2005; Glidewell *et al.*, 2005, 2006; Sun *et al.*, 2004; Fun *et al.*, 2008).

The molecule of the title Schiff base compound has crystallographic twofold rotation symmetry (Fig. 1). Bond lengths are within normal ranges (Allen *et al.*, 1987). The plane of the —C=N—C— group is twisted away from the benzene ring by $7.5(1)^\circ$.

In the crystal structure, weak intermolecular $\text{Cl}\cdots\text{Cl}$ contacts [$\text{Cl1}\cdots\text{Cl2}(x, 1-y, -1/2+z) = 3.4851(3) \text{ \AA}$] link neighbouring molecules into a two-dimensional network parallel to the *bc* plane (Fig.2).

Experimental

The synthetic method has been described earlier (Fun *et al.*, 2008). Single crystals suitable for *X*-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

Refinement

H atoms were positioned geometrically and refined using a riding model, with $\text{C—H} = 0.93 \text{ \AA}$ for aromatic and 0.97 \AA for methylene and 0.96 \AA for methyl H atoms.

Figures

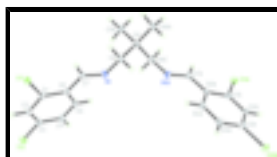


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability ellipsoids for non-H atoms. Atoms labelled with the suffix A are generated by the symmetry operation $(1/2 - x, 2 - y, z)$.

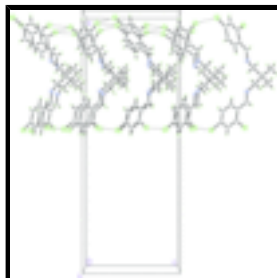


Fig. 2. Part of the crystal structure of the title compound, viewed down the *b*-axis. Dashed lines indicate intermolecular $\text{Cl}\cdots\text{Cl}$ interactions.

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Crystal data

$C_{19}H_{18}Cl_4N_2$	$F_{000} = 856$
$M_r = 416.15$	$D_x = 1.452 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pcca</i>	Mo $K\alpha$ radiation
Hall symbol: -P 2a 2ac	$\lambda = 0.71073 \text{ \AA}$
$a = 30.7633 (4) \text{ \AA}$	Cell parameters from 9929 reflections
$b = 5.4012 (1) \text{ \AA}$	$\theta = 3.6\text{--}41.1^\circ$
$c = 11.4532 (1) \text{ \AA}$	$\mu = 0.63 \text{ mm}^{-1}$
$V = 1903.05 (5) \text{ \AA}^3$	$T = 100.0 (1) \text{ K}$
$Z = 4$	Block, colourless
	$0.43 \times 0.25 \times 0.23 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	6565 independent reflections
Radiation source: fine-focus sealed tube	5678 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.028$
$T = 100.0(1) \text{ K}$	$\theta_{\text{max}} = 42.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.3^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -52 \rightarrow 57$
$T_{\text{min}} = 0.774$, $T_{\text{max}} = 0.872$	$k = -9 \rightarrow 10$
86683 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.5965P]$
$S = 1.19$	where $P = (F_o^2 + 2F_c^2)/3$
4992 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
115 parameters	$\Delta\rho_{\text{max}} = 0.51 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.053693 (7)	1.12278 (4)	0.346996 (17)	0.01671 (5)
Cl2	0.046757 (8)	0.37440 (4)	0.653936 (18)	0.02040 (6)
N1	0.19231 (2)	0.96758 (14)	0.31674 (6)	0.01660 (12)
C1	0.08049 (2)	0.88739 (14)	0.42126 (6)	0.01301 (12)
C2	0.05625 (3)	0.74765 (14)	0.49989 (6)	0.01467 (12)
H2A	0.0270	0.7822	0.5127	0.018*
C3	0.07667 (3)	0.55568 (15)	0.55865 (7)	0.01504 (12)
C4	0.12041 (3)	0.50138 (16)	0.54095 (7)	0.01807 (13)
H4A	0.1336	0.3713	0.5807	0.022*
C5	0.14395 (3)	0.64492 (16)	0.46298 (8)	0.01748 (13)
H5A	0.1732	0.6098	0.4510	0.021*
C6	0.12480 (2)	0.84188 (14)	0.40166 (7)	0.01397 (12)
C7	0.15133 (2)	0.99635 (15)	0.32244 (7)	0.01527 (12)
H7A	0.1379	1.1157	0.2763	0.018*
C8	0.21668 (3)	1.13674 (14)	0.24238 (7)	0.01553 (13)
H8A	0.2317	1.2566	0.2909	0.019*
H8B	0.1966	1.2262	0.1925	0.019*
C9	0.2500	1.0000	0.16598 (9)	0.01351 (16)
C18	0.22667 (3)	0.81059 (16)	0.08866 (7)	0.01774 (13)
H18A	0.2044	0.8922	0.0443	0.027*
H18B	0.2138	0.6850	0.1368	0.027*
H18C	0.2472	0.7358	0.0364	0.027*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01438 (9)	0.01789 (9)	0.01786 (9)	0.00404 (6)	0.00073 (5)	0.00121 (5)
Cl2	0.02045 (10)	0.02338 (10)	0.01738 (9)	-0.00623 (7)	0.00240 (6)	0.00349 (6)
N1	0.0117 (3)	0.0205 (3)	0.0176 (3)	-0.0015 (2)	0.0016 (2)	0.0025 (2)
C1	0.0116 (3)	0.0143 (3)	0.0131 (3)	0.0006 (2)	0.0001 (2)	-0.0008 (2)
C2	0.0117 (3)	0.0175 (3)	0.0149 (3)	-0.0002 (2)	0.0016 (2)	-0.0010 (2)
C3	0.0143 (3)	0.0170 (3)	0.0139 (3)	-0.0031 (2)	0.0004 (2)	0.0006 (2)
C4	0.0136 (3)	0.0197 (3)	0.0209 (3)	-0.0004 (2)	-0.0016 (2)	0.0046 (3)
C5	0.0108 (3)	0.0207 (3)	0.0209 (3)	0.0005 (2)	-0.0002 (2)	0.0044 (3)
C6	0.0102 (3)	0.0168 (3)	0.0149 (3)	-0.0004 (2)	0.0005 (2)	0.0009 (2)

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C7	0.0120 (3)	0.0178 (3)	0.0160 (3)	-0.0004 (2)	0.0010 (2)	0.0015 (2)
C8	0.0120 (3)	0.0163 (3)	0.0183 (3)	-0.0012 (2)	0.0021 (2)	0.0009 (2)
C9	0.0113 (4)	0.0144 (4)	0.0148 (4)	-0.0023 (3)	0.000	0.000
C18	0.0169 (3)	0.0176 (3)	0.0187 (3)	-0.0033 (3)	-0.0036 (2)	-0.0008 (2)

Geometric parameters (Å, °)

C11—C1	1.7376 (8)	C5—H5A	0.93
C12—C3	1.7311 (8)	C6—C7	1.4783 (11)
N1—C7	1.2720 (10)	C7—H7A	0.93
N1—C8	1.4566 (10)	C8—C9	1.5369 (10)
C1—C2	1.3917 (11)	C8—H8A	0.97
C1—C6	1.4033 (11)	C8—H8B	0.97
C2—C3	1.3866 (11)	C9—C18	1.5316 (10)
C2—H2A	0.93	C9—C18 ⁱ	1.5316 (10)
C3—C4	1.3917 (11)	C9—C8 ⁱ	1.5369 (10)
C4—C5	1.3869 (12)	C18—H18A	0.96
C4—H4A	0.93	C18—H18B	0.96
C5—C6	1.4043 (11)	C18—H18C	0.96
C7—N1—C8	117.61 (7)	N1—C7—H7A	119.7
C2—C1—C6	121.94 (7)	C6—C7—H7A	119.7
C2—C1—C11	117.35 (6)	N1—C8—C9	112.00 (6)
C6—C1—C11	120.71 (6)	N1—C8—H8A	109.2
C3—C2—C1	118.49 (7)	C9—C8—H8A	109.2
C3—C2—H2A	120.8	N1—C8—H8B	109.2
C1—C2—H2A	120.8	C9—C8—H8B	109.2
C2—C3—C4	121.66 (7)	H8A—C8—H8B	107.9
C2—C3—C12	119.21 (6)	C18—C9—C18 ⁱ	109.36 (9)
C4—C3—C12	119.12 (6)	C18—C9—C8 ⁱ	108.71 (4)
C5—C4—C3	118.74 (8)	C18 ⁱ —C9—C8 ⁱ	109.73 (4)
C5—C4—H4A	120.6	C18—C9—C8	109.73 (4)
C3—C4—H4A	120.6	C18 ⁱ —C9—C8	108.71 (4)
C4—C5—C6	121.76 (7)	C8 ⁱ —C9—C8	110.59 (9)
C4—C5—H5A	119.1	C9—C18—H18A	109.5
C6—C5—H5A	119.1	C9—C18—H18B	109.5
C1—C6—C5	117.40 (7)	H18A—C18—H18B	109.5
C1—C6—C7	122.39 (7)	C9—C18—H18C	109.5
C5—C6—C7	120.19 (7)	H18A—C18—H18C	109.5
N1—C7—C6	120.65 (7)	H18B—C18—H18C	109.5
C6—C1—C2—C3	-1.08 (11)	C11—C1—C6—C7	2.99 (11)
C11—C1—C2—C3	178.81 (6)	C4—C5—C6—C1	-0.64 (13)
C1—C2—C3—C4	0.20 (12)	C4—C5—C6—C7	177.81 (8)
C1—C2—C3—C12	-178.30 (6)	C8—N1—C7—C6	-176.18 (7)
C2—C3—C4—C5	0.42 (13)	C1—C6—C7—N1	172.02 (8)
C12—C3—C4—C5	178.92 (7)	C5—C6—C7—N1	-6.36 (12)
C3—C4—C5—C6	-0.19 (13)	C7—N1—C8—C9	-134.46 (8)
C2—C1—C6—C5	1.29 (11)	N1—C8—C9—C18	59.07 (9)

C11—C1—C6—C5	-178.59 (6)	N1—C8—C9—C18 ⁱ	178.63 (7)
C2—C1—C6—C7	-177.13 (7)	N1—C8—C9—C8 ⁱ	-60.85 (5)

Symmetry codes: (i) $-x+1/2, -y+2, z$.

Fig. 1

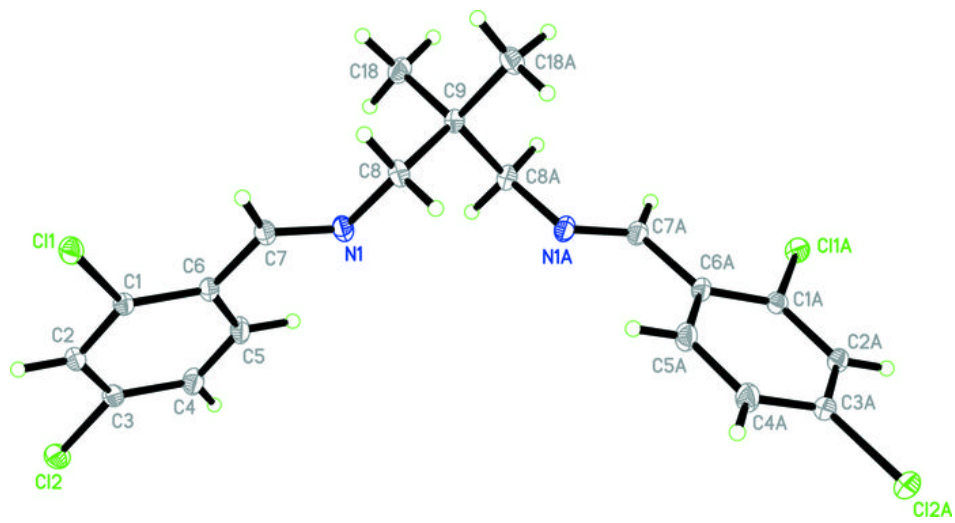


Fig. 2

