

N,N'-Bis(2,6-dichlorobenzyl)ethylene-diiimine

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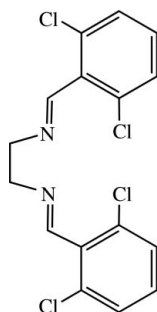
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Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.054; wR factor = 0.127; data-to-parameter ratio = 15.7.

In the centrosymmetric title compound, $\text{C}_{16}\text{H}_{12}\text{Cl}_4\text{N}_2$, the asymmetric unit is one half-molecule. Weak van der Waals interactions between the molecules are effective in the molecular packing. This is the first reported structure of a chloro-substituted benzaldehyde derivative that can potentially form a tetradentate ligand.

Related literature

For related literature, see: Helldörfer *et al.* (2003); Richmond *et al.* (1988).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{Cl}_4\text{N}_2$	$V = 824.25 (15) \text{ \AA}^3$
$M_r = 374.08$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 3.9704 (5) \text{ \AA}$	$\mu = 0.71 \text{ mm}^{-1}$
$b = 14.3246 (13) \text{ \AA}$	$T = 290 (2) \text{ K}$
$c = 14.4926 (14) \text{ \AA}$	$0.2 \times 0.1 \times 0.1 \text{ mm}$
$\beta = 90.201 (9)^\circ$	

Data collection

Oxford Diffraction XcaliburII with SapphireIII CCD diffractometer	5106 measured reflections
Absorption correction: numerical (<i>X-RED</i> and <i>X-SHAPE</i> ; Stoe & Cie, 1997)	1585 independent reflections
$T_{\min} = 0.749$, $T_{\max} = 0.853$	621 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.098$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$	101 parameters
$wR(F^2) = 0.127$	H-atom parameters constrained
$S = 0.80$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
1585 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2093).

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supplementary materials

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N,N'-Bis(2,6-dichlorobenzyl)ethylenediimine

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Comment

In the present study the structure of the title compound, which can be used as tetradentate ligand coordinating to metals and/or for the preparation of crystalline polymers, is investigated. Previously, novel coordination polymers containing silver ions bridged by the monochloro-substituted equivalent of the title compound has been studied (Richmond *et al.*, 1988). In addition, various macrocyclic compounds can be synthesized by template condensation of ethane-1,2-diimine or related blocks (Helldörfer *et al.*, 2003).

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. Relatively weak intermolecular van der Waals interactions are present between neighboring molecules, stabilizing the crystal structure. Two benzyl rings, which are connected through the diimine group, lie in two parallel planes with a dihedral angle of zero. The two carbon and two nitrogen atoms in the ethane-1,2-diimine chain are co-planar, with an N1—C8—C8—N1 torsion angle of 180.0 (0)°, as there is a centre of symmetry between the two central carbon atoms.

Experimental

2,6-dichlorobenzaldehyde (100 mmol) in absolute ethanol (30 ml) was added to ethylenediamine (50 mmol) and stirred for 24 h. The colorless crystalline solid was filtered and washed with ether and hexane. Very small crystals were obtained by recrystallization from dichloromethane. It was not possible to obtain larger crystals.

Refinement

All H atoms were geometrically positioned and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The very small crystals led to weak data and a low ratio of observed to unique reflections.

Figures

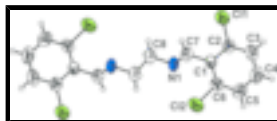


Fig. 1. Molecular structure of (I) showing the atom labelling scheme, with 50% probability displacement ellipsoids.

N,N'-Bis(2,6-dichlorobenzyl)ethylenediimine

Crystal data

$\text{C}_{16}\text{H}_{12}\text{Cl}_4\text{N}_2$

$M_r = 374.08$

Monoclinic, $P2_1/c$

$F_{000} = 380$

$D_x = 1.507 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

supplementary materials

Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 3.9704 (5) \text{ \AA}$	Cell parameters from 5106 reflections
$b = 14.3246 (13) \text{ \AA}$	$\theta = 4.0\text{--}32.2^\circ$
$c = 14.4926 (14) \text{ \AA}$	$\mu = 0.71 \text{ mm}^{-1}$
$\beta = 90.201 (9)^\circ$	$T = 290 (2) \text{ K}$
$V = 824.25 (15) \text{ \AA}^3$	Block, colourless
$Z = 2$	$0.2 \times 0.1 \times 0.1 \text{ mm}$

Data collection

Oxford Diffraction XcaliburII with SapphireIII CCD diffractometer	1585 independent reflections
Radiation source: fine-focus sealed tube	621 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.098$
Detector resolution: 12 pixels mm^{-1}	$\theta_{\text{max}} = 26.0^\circ$
$T = 290(2) \text{ K}$	$\theta_{\text{min}} = 4.0^\circ$
ω scans at different φ	$h = -4 \rightarrow 4$
Absorption correction: numerical X-RED and X-SHAPE (Stoe & Cie, 1997)	$k = -15 \rightarrow 17$
$T_{\text{min}} = 0.749, T_{\text{max}} = 0.853$	$l = -15 \rightarrow 17$
5106 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2]$
$wR(F^2) = 0.127$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.80$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1585 reflections	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
101 parameters	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97, $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.016 (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 $F^2 > 2\sigma(F^2)$ is used only for calculat-

ing 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.8556 (3)	0.96684 (8)	0.13206 (7)	0.0833 (5)
Cl2	0.7366 (3)	1.24856 (7)	0.37687 (7)	0.0899 (5)
N1	0.7752 (7)	1.0293 (2)	0.3933 (2)	0.0582 (9)
C1	0.7773 (8)	1.1105 (2)	0.2499 (2)	0.0482 (9)
C2	0.7342 (9)	1.0791 (3)	0.1596 (2)	0.0552 (10)
C3	0.5925 (11)	1.1355 (3)	0.0922 (3)	0.0721 (12)
H3	0.5629	1.1129	0.0325	0.086*
C4	0.4978 (12)	1.2233 (4)	0.1134 (3)	0.0813 (14)
H4	0.4055	1.2611	0.0676	0.098*
C5	0.5349 (11)	1.2583 (3)	0.2017 (3)	0.0746 (12)
H5	0.4673	1.3187	0.2160	0.089*
C6	0.6755 (9)	1.2008 (3)	0.2678 (2)	0.0577 (10)
C7	0.9218 (9)	1.0470 (2)	0.3199 (3)	0.0547 (10)
H7	1.1297	1.0194	0.3086	0.066*
C8	0.9438 (9)	0.9702 (2)	0.4596 (2)	0.0604 (11)
H8A	1.1373	0.9405	0.4313	0.072*
H8B	0.7917	0.9218	0.4807	0.072*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1191 (11)	0.0660 (8)	0.0647 (7)	0.0050 (7)	-0.0062 (7)	-0.0082 (6)
Cl2	0.1344 (11)	0.0710 (8)	0.0645 (8)	0.0043 (7)	-0.0006 (7)	-0.0138 (6)
N1	0.055 (2)	0.072 (2)	0.0469 (18)	0.0036 (18)	-0.0077 (16)	0.0153 (17)
C1	0.050 (2)	0.052 (2)	0.042 (2)	-0.0046 (19)	0.0016 (18)	0.0082 (18)
C2	0.059 (3)	0.054 (2)	0.052 (2)	-0.0002 (19)	0.004 (2)	0.009 (2)
C3	0.093 (3)	0.076 (3)	0.047 (2)	-0.006 (3)	-0.010 (2)	0.013 (2)
C4	0.095 (4)	0.084 (4)	0.064 (3)	0.007 (3)	-0.009 (3)	0.025 (3)
C5	0.091 (3)	0.058 (3)	0.075 (3)	0.009 (2)	0.009 (3)	0.013 (2)
C6	0.066 (3)	0.061 (3)	0.046 (2)	-0.002 (2)	0.0076 (19)	0.000 (2)
C7	0.055 (2)	0.057 (3)	0.052 (2)	0.000 (2)	-0.009 (2)	0.005 (2)
C8	0.065 (3)	0.067 (3)	0.049 (2)	0.001 (2)	-0.011 (2)	0.0113 (19)

Geometric parameters (\AA , $^\circ$)

Cl1—C2	1.727 (4)	C3—H3	0.9300
Cl2—C6	1.738 (3)	C4—C5	1.382 (5)
N1—C7	1.241 (4)	C4—H4	0.9300
N1—C8	1.443 (4)	C5—C6	1.381 (5)
C1—C6	1.381 (5)	C5—H5	0.9300
C1—C2	1.393 (4)	C7—H7	0.9300
C1—C7	1.477 (4)	C8—C8 ⁱ	1.515 (6)

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C2—C3	1.386 (5)	C8—H8A	0.9700
C3—C4	1.348 (5)	C8—H8B	0.9700
C7—N1—C8	118.2 (3)	C6—C5—H5	121.0
C6—C1—C2	116.3 (3)	C4—C5—H5	121.0
C6—C1—C7	124.1 (3)	C5—C6—C1	123.1 (3)
C2—C1—C7	119.6 (3)	C5—C6—C12	116.9 (3)
C3—C2—C1	121.5 (4)	C1—C6—C12	120.0 (3)
C3—C2—C11	119.5 (3)	N1—C7—C1	122.2 (4)
C1—C2—C11	118.9 (3)	N1—C7—H7	118.9
C4—C3—C2	119.7 (4)	C1—C7—H7	118.9
C4—C3—H3	120.1	N1—C8—C8 ⁱ	108.6 (4)
C2—C3—H3	120.1	N1—C8—H8A	110.0
C3—C4—C5	121.4 (4)	C8 ⁱ —C8—H8A	110.0
C3—C4—H4	119.3	N1—C8—H8B	110.0
C5—C4—H4	119.3	C8 ⁱ —C8—H8B	110.0
C6—C5—C4	117.9 (4)	H8A—C8—H8B	108.4
C6—C1—C2—C3	-0.9 (5)	C2—C1—C6—C5	0.6 (6)
C7—C1—C2—C3	178.5 (3)	C7—C1—C6—C5	-178.7 (4)
C6—C1—C2—C11	-180.0 (3)	C2—C1—C6—C12	-176.9 (3)
C7—C1—C2—C11	-0.6 (5)	C7—C1—C6—C12	3.7 (5)
C1—C2—C3—C4	1.0 (6)	C8—N1—C7—C1	-176.9 (3)
C11—C2—C3—C4	-179.9 (4)	C6—C1—C7—N1	54.5 (5)
C2—C3—C4—C5	-0.8 (7)	C2—C1—C7—N1	-124.8 (4)
C3—C4—C5—C6	0.6 (7)	C7—N1—C8—C8 ⁱ	108.0 (5)
C4—C5—C6—C1	-0.5 (6)	N1—C8—C8 ⁱ —N1 ⁱ	180.000 (4)
C4—C5—C6—C12	177.1 (3)		

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

Fig. 1

