Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A new salen ligand: 2,2′-[(spiro[4.4]-nonane-1,6-diyl)dinitrilomethylidyne]-bis(4,6-dichlorophenol)

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Received 27 April 2007; accepted 10 May 2007

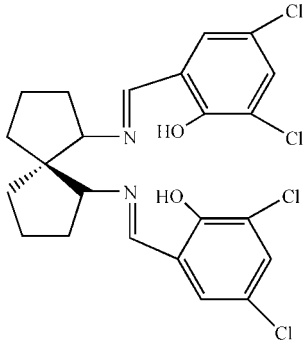
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.086; wR factor = 0.277; data-to-parameter ratio = 18.5.

The title compound, $C_{23}H_{22}Cl_4N_2O_2$, was synthesized by the reaction of 1,1′-spiro[4,4]nonane-1,6-diamine and 3,5-dichlorosalicylaldehyde. It is a new Schiff base containing a spirocyclic backbone and an approximate non-crystallographic C_2 axis. Two intramolecular O—H···N hydrogen bonds determine the relative orientation of the two benzene rings. Intermolecular π–π stacking interactions between the phenyl rings and weak intermolecular C—H···Cl hydrogen bonds stabilize the structure. Cp1 and Cp2 are the centroids of the planes defined by atoms C11–C16 and C18–C23. The angle between the planes is only 0.8° and interplanar distances are Cp1···Cp2(x, y, $z+1$) = 4.023 Å and Cp1···Cp2($x+1$, y, $z+1$) = 4.032 Å.

Related literature

For information on the application of salen complexes to asymmetric catalysis, see Larrow *et al.* (1994), Li *et al.* (2006) and Lo *et al.* (2006). For related structures, see Zhou *et al.* (1999), Li *et al.* (2005) and Altona *et al.* (1971).

For related literature, see: Cram & Steinberg (1954); Ebeling *et al.* (2002).



Experimental

Crystal data

$C_{23}H_{22}Cl_4N_2O_2$
M_r = 500.23
Monoclinic, $P2_1/n$
a = 8.052 (2) Å
b = 33.489 (6) Å
c = 8.881 (3) Å
β = 107.310 (5)°
V = 2286.5 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 294 (2) K
0.50 × 0.50 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.686, T_{max} = 0.869
20717 measured reflections
5224 independent reflections
2703 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.277
S = 1.01
5224 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N2	0.82	1.88	2.597 (3)	146
O1—H1···N1	0.82	1.86	2.589 (3)	148
C2—H2A···Cl1^{i}	0.98	3.10	4.005 (3)	154
C7—H7B···Cl1^{ii}	0.97	2.87	3.734 (4)	149
C14—H14···Cl2^{iii}	0.93	3.01	3.886 (3)	158
C6—H6···Cl3^{iv}	0.98	3.10	4.027 (3)	158
C3—H3A···Cl3^{v}	0.97	3.00	3.874 (4)	151
C21—H21···Cl4^{vi}	0.93	3.01	3.886 (3)	158
C23—H23···Cl4^{vii}	0.93	3.16	4.023 (3)	154

Symmetry codes: (i) $x-1, y, z-1$; (ii) $x, y, z-1$; (iii) $-x+1, -y+1, -z+2$; (iv) $x+1, y, z+1$; (v) $x, y, z+1$; (vi) $-x-1, -y+1, -z-1$; (vii) $-x, -y+1, -z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

This project was supported by the Student Innovation Foundation of Sichuan University and sponsored by the Scientific Research Foundation for Returned Overseas Chinese Scholars, State Education Ministry of China. We also thank Sichuan University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2310).

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supplementary materials

Acta Cryst. (2007). E**63**, o3049-o3050 [**doi:10.1107/S1600536807023148**]

A new salen ligand: 2,2'-[(spiro[4.4]nonane-1,6-diyl)dinitrilomethylidyne]bis(4,6-dichlorophenol)

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Comment

Chiral salen transition metal complexes have been widely and successfully applied in various asymmetric catalytic reactions (Larrow *et al.*, 1994; Li *et al.*, 2006; Lo *et al.*, 2006). Compared with a normal chiral salen ligand, derived from diamine with chiral carbons, such as cyclohexanediamine or 1, 2-diphenylethyldiamine, chiral salen with spiro scaffold has never been reported. In a continuation of our studies of salen complexes (Zhou *et al.*, 1999; Li *et al.*, 2005), we report here the synthesis and structure of the title compound. The rigid structure indicates its potential usage as a ligand in asymmetric synthesis.

The novel salen ligand (I) possesses a spirocyclic backbone, Fig. 1, and contains a non-crystallgraphic C_2 axis through the C1 atom, Fig. 1. The two five–membered rings adopt a conformation intermediate between envelope and half-chair forms but closer to the latter as reported for spiro[4,4]nonane-1,6-dione (Altona *et al.*, 1971) and nearly perpendicular to each other with a dihedral angle of 86.1 (3)° between the respective ring planes, which adds to the rigidity of the molecule. The two imine moieties are well separated and provide potential sites for coordination with metal ions. Furthermore, there are two intramolecular O—H···N hydrogen bonds in the ligand that define the conformation of the two benzene rings.

In the crystal structure there are intermolecular π-π stacking interactions between the C12–C19 and C15–C22 rings as well as a number of weak Cl···H–C hydrogen bonds, Fig 2.

Experimental

The salen ligand, 2,6-bis(3,5-dichlorosalicylaldimine)-1,1`-spiro[4,4]nonane was prepared by condensation of 3,5-dichlorosalicylaldehyde with 1, 1`-spiro[4,4]nonane-1,6-diamine, which was prepared by literature methods (Cram & Steinberg, 1954; Ebeling *et al.*, 2002). Crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol /methylene chloride (10:1) solution of (I). MS (EI) m/*z*: 500(M^+). Anal calculated for C23H22Cl4N2O2: C, 55.20; H, 4.40; N, 5.60. Found: C, 55.12; H, 4.48; N, 5.61.

Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C) for aromatic, 0.97 Å, U_{iso}(H) = 1.2U_{eq}(C) for CH_2, O—H = 0.82 Å, U_{iso}(H) = 1.5U_{eq}(O) for the OH groups.

Figures

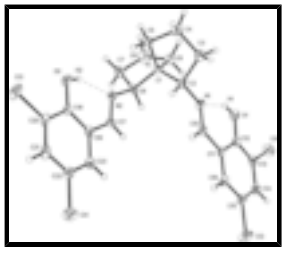


Fig. 1. A perspective view, with displacement ellipsoids drawn at the 30% probability level.

2,6-bis(3,5-dichlorosalicylaldimine)-1,1'-spiro[4,4]nonane

Crystal data

$C_{23}H_{22}Cl_4N_2O_2$	F_{000} = 1032
M_r = 500.23	D_x = 1.453 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 7024 reflections
a = 8.052 (2) Å	θ = 1–27.5º
b = 33.489 (6) Å	μ = 0.54 mm^{-1}
c = 8.881 (3) Å	T = 294 (2) K
β = 107.310 (5)º	Plate, colourless
V = 2286.5 (10) Å^3	0.50 × 0.50 × 0.26 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	5224 independent reflections
Radiation source: fine-focus sealed tube	2703 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.080
T = 294(2) K	θ_{max} = 27.6º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→10
T_{min} = 0.686, T_{max} = 0.869	k = −43→43
20717 measured reflections	l = −11→11

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.086	H-atom parameters constrained
$wR(F^2)$ = 0.277	$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2 + 10.5P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
5224 reflections	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
282 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, p.p.m.): 1.30–1.96 (m, 12H, CH_2), 3.13 (m, 2H, CH), 7.14 (m, 2H, Ar—H), 7.34 (m, 2H, Ar—H), 5.0 (br, 2H, –OH), 8.18 (s, 2H, HC═N).

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.54922 (14)	0.61810 (3)	1.00270 (11)	0.0636 (3)
Cl2	0.23820 (16)	0.48030 (3)	0.76711 (14)	0.0695 (4)
Cl3	−0.44144 (15)	0.61516 (4)	−0.57224 (11)	0.0691 (3)
Cl4	−0.27100 (15)	0.47871 (3)	−0.24418 (13)	0.0658 (3)
O1	0.3179 (3)	0.65320 (7)	0.7145 (3)	0.0504 (8)
H1	0.2512	0.6616	0.6317	0.076*
O2	−0.1879 (3)	0.65165 (8)	−0.2998 (3)	0.0511 (8)
H2	−0.1096	0.6603	−0.2252	0.077*
N1	0.0794 (3)	0.65024 (8)	0.4446 (3)	0.0370 (8)
N2	0.0376 (3)	0.65020 (8)	−0.0213 (3)	0.0374 (8)
C1	0.0581 (4)	0.69371 (8)	0.2094 (3)	0.0267 (7)
C2	−0.0370 (4)	0.66977 (10)	0.3067 (3)	0.0347 (9)
H2A	−0.1101	0.6495	0.2388	0.042*
C3	−0.1546 (5)	0.70092 (12)	0.3505 (5)	0.0506 (11)
H3A	−0.2567	0.6883	0.3661	0.061*
H3B	−0.0924	0.7149	0.4465	0.061*
C4	−0.2054 (5)	0.72931 (13)	0.2129 (4)	0.0581 (12)
H4A	−0.1865	0.7567	0.2495	0.070*
H4B	−0.3274	0.7260	0.1548	0.070*
C5	−0.0928 (4)	0.71947 (10)	0.1091 (4)	0.0383 (9)
H5A	−0.0485	0.7437	0.0752	0.046*
H5B	−0.1591	0.7049	0.0164	0.046*
C6	0.1552 (4)	0.66987 (10)	0.1156 (3)	0.0329 (8)
H6	0.2296	0.6499	0.1842	0.040*
C7	0.2688 (4)	0.70132 (11)	0.0676 (4)	0.0460 (10)

H7A	0.2046	0.7147	−0.0291	0.055*
H7B	0.3722	0.6892	0.0526	0.055*
C8	0.3169 (5)	0.73070 (14)	0.2072 (5)	0.0641 (13)
H8A	0.4389	0.7281	0.2662	0.077*
H8B	0.2955	0.7580	0.1697	0.077*
C9	0.2034 (4)	0.72019 (11)	0.3102 (4)	0.0392 (9)
H9A	0.1555	0.7442	0.3424	0.047*
H9B	0.2702	0.7060	0.4038	0.047*
C10	0.0692 (4)	0.61313 (10)	0.4599 (3)	0.0335 (8)
H10	−0.0101	0.5987	0.3813	0.040*
C11	0.1808 (4)	0.59175 (10)	0.6003 (3)	0.0346 (8)
C12	0.3006 (4)	0.61407 (10)	0.7220 (3)	0.0354 (9)
C13	0.3999 (4)	0.59259 (11)	0.8535 (4)	0.0401 (9)
C14	0.3822 (4)	0.55176 (11)	0.8680 (4)	0.0424 (10)
H14	0.4496	0.5383	0.9567	0.051*
C15	0.2626 (5)	0.53137 (11)	0.7486 (4)	0.0440 (10)
C16	0.1630 (4)	0.55106 (11)	0.6149 (4)	0.0386 (9)
H16	0.0840	0.5369	0.5351	0.046*
C17	0.0223 (4)	0.61260 (9)	−0.0209 (3)	0.0331 (8)
H17	0.0912	0.5980	0.0640	0.040*
C18	−0.1022 (4)	0.59139 (10)	−0.1519 (3)	0.0341 (9)
C19	−0.2007 (4)	0.61212 (10)	−0.2847 (3)	0.0352 (9)
C20	−0.3175 (4)	0.59040 (11)	−0.4050 (3)	0.0395 (9)
C21	−0.3378 (4)	0.54951 (12)	−0.3938 (4)	0.0446 (10)
H21	−0.4160	0.5355	−0.4749	0.054*
C22	−0.2402 (4)	0.53002 (11)	−0.2603 (4)	0.0408 (9)
C23	−0.1231 (4)	0.54973 (10)	−0.1397 (4)	0.0385 (9)
H23	−0.0581	0.5359	−0.0509	0.046*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0608 (6)	0.0764 (7)	0.0372 (4)	−0.0104 (5)	−0.0103 (4)	0.0129 (5)
Cl2	0.0771 (7)	0.0500 (5)	0.0698 (6)	0.0244 (5)	0.0041 (5)	0.0005 (5)
Cl3	0.0704 (6)	0.0789 (7)	0.0382 (4)	0.0185 (5)	−0.0142 (5)	−0.0151 (6)
Cl4	0.0741 (6)	0.0474 (5)	0.0617 (6)	0.0015 (5)	−0.0014 (5)	−0.0138 (5)
O1	0.0647 (16)	0.0420 (14)	0.0342 (12)	−0.0015 (10)	−0.0010 (12)	−0.0001 (12)
O2	0.0674 (17)	0.0440 (14)	0.0312 (12)	0.0062 (10)	−0.0015 (12)	−0.0042 (12)
N1	0.0397 (14)	0.0396 (15)	0.0271 (12)	0.0079 (11)	0.0031 (11)	−0.0004 (12)
N2	0.0376 (14)	0.0449 (16)	0.0253 (12)	−0.0064 (11)	0.0025 (11)	0.0032 (12)
C1	0.0236 (12)	0.0258 (13)	0.0265 (13)	−0.0021 (12)	0.0010 (11)	−0.0012 (12)
C2	0.0297 (15)	0.0418 (17)	0.0253 (14)	0.0102 (12)	−0.0031 (12)	0.0005 (14)
C3	0.0442 (17)	0.058 (2)	0.0572 (19)	0.0088 (17)	0.0267 (15)	0.0075 (17)
C4	0.0477 (19)	0.070 (3)	0.064 (2)	0.0217 (18)	0.0290 (17)	0.0284 (18)
C5	0.0374 (16)	0.0386 (17)	0.0334 (15)	0.0108 (13)	0.0022 (14)	0.0107 (15)
C6	0.0286 (14)	0.0412 (17)	0.0258 (14)	−0.0050 (12)	0.0033 (12)	0.0019 (14)
C7	0.0397 (16)	0.055 (2)	0.0505 (18)	−0.0076 (16)	0.0237 (14)	−0.0055 (16)
C8	0.056 (2)	0.075 (3)	0.068 (2)	−0.024 (2)	0.0286 (19)	−0.0329 (19)

C9	0.0301 (15)	0.0456 (19)	0.0414 (16)	−0.0057 (15)	0.0098 (13)	−0.0082 (14)
C10	0.0346 (15)	0.0391 (17)	0.0249 (13)	0.0034 (12)	0.0059 (12)	−0.0012 (14)
C11	0.0360 (15)	0.0434 (18)	0.0252 (13)	0.0061 (13)	0.0102 (12)	0.0046 (14)
C12	0.0392 (16)	0.0430 (19)	0.0256 (13)	0.0017 (12)	0.0119 (12)	0.0021 (14)
C13	0.0350 (16)	0.057 (2)	0.0259 (14)	0.0013 (14)	0.0055 (13)	0.0101 (16)
C14	0.0440 (17)	0.055 (2)	0.0275 (14)	0.0114 (14)	0.0093 (14)	0.0158 (16)
C15	0.0451 (18)	0.0452 (19)	0.0425 (17)	0.0156 (15)	0.0143 (15)	0.0126 (16)
C16	0.0347 (16)	0.051 (2)	0.0290 (15)	0.0086 (14)	0.0075 (13)	0.0005 (15)
C17	0.0336 (15)	0.0359 (17)	0.0260 (13)	−0.0024 (12)	0.0031 (12)	0.0088 (14)
C18	0.0339 (15)	0.0411 (17)	0.0238 (13)	−0.0043 (13)	0.0030 (12)	−0.0020 (14)
C19	0.0385 (16)	0.0424 (18)	0.0233 (13)	0.0025 (12)	0.0073 (13)	−0.0018 (14)
C20	0.0379 (16)	0.056 (2)	0.0204 (13)	0.0046 (14)	0.0029 (13)	−0.0021 (16)
C21	0.0350 (16)	0.067 (2)	0.0293 (15)	−0.0035 (15)	0.0064 (13)	−0.0143 (17)
C22	0.0396 (17)	0.0437 (19)	0.0362 (16)	0.0000 (15)	0.0067 (14)	−0.0034 (15)
C23	0.0401 (17)	0.0437 (19)	0.0276 (14)	−0.0008 (13)	0.0038 (13)	−0.0006 (15)

Geometric parameters (Å, °)

Cl1—C13	1.727 (3)	C7—C8	1.540 (5)
Cl2—C15	1.735 (4)	C7—H7A	0.9700
Cl3—C20	1.734 (3)	C7—H7B	0.9700
Cl4—C22	1.748 (4)	C8—C9	1.514 (6)
O1—C12	1.321 (4)	C8—H8A	0.9700
O1—H1	0.8200	C8—H8B	0.9700
O2—C19	1.338 (4)	C9—H9A	0.9700
O2—H2	0.8200	C9—H9B	0.9700
N1—C10	1.255 (4)	C10—C11	1.486 (4)
N1—C2	1.458 (4)	C10—H10	0.9300
N2—C17	1.265 (4)	C11—C16	1.380 (5)
N2—C6	1.457 (4)	C11—C12	1.427 (4)
C1—C6	1.526 (5)	C12—C13	1.402 (4)
C1—C9	1.527 (4)	C13—C14	1.385 (5)
C1—C2	1.539 (5)	C14—C15	1.383 (5)
C1—C5	1.539 (4)	C14—H14	0.9300
C2—C3	1.535 (5)	C15—C16	1.386 (4)
C2—H2A	0.9800	C16—H16	0.9300
C3—C4	1.506 (5)	C17—C18	1.473 (4)
C3—H3A	0.9700	C17—H17	0.9300
C3—H3B	0.9700	C18—C19	1.395 (4)
C4—C5	1.510 (6)	C18—C23	1.413 (5)
C4—H4A	0.9700	C19—C20	1.399 (4)
C4—H4B	0.9700	C20—C21	1.386 (5)
C5—H5A	0.9700	C21—C22	1.376 (5)
C5—H5B	0.9700	C21—H21	0.9300
C6—C7	1.537 (5)	C22—C23	1.368 (4)
C6—H6	0.9800	C23—H23	0.9300
C12—O1—H1	109.5	C7—C8—H8B	110.4
C19—O2—H2	109.5	H8A—C8—H8B	108.6
C10—N1—C2	119.3 (3)	C8—C9—C1	106.0 (3)

C17—N2—C6	119.1 (3)	C8—C9—H9A	110.5
C6—C1—C9	101.3 (3)	C1—C9—H9A	110.5
C6—C1—C2	117.1 (3)	C8—C9—H9B	110.5
C9—C1—C2	113.3 (3)	C1—C9—H9B	110.5
C6—C1—C5	114.9 (2)	H9A—C9—H9B	108.7
C9—C1—C5	110.4 (2)	N1—C10—C11	121.7 (3)
C2—C1—C5	100.3 (2)	N1—C10—H10	119.2
N1—C2—C3	112.5 (3)	C11—C10—H10	119.2
N1—C2—C1	113.7 (3)	C16—C11—C12	120.7 (3)
C3—C2—C1	103.7 (3)	C16—C11—C10	120.1 (3)
N1—C2—H2A	108.9	C12—C11—C10	119.1 (3)
C3—C2—H2A	108.9	O1—C12—C13	120.6 (3)
C1—C2—H2A	108.9	O1—C12—C11	122.6 (3)
C4—C3—C2	105.2 (3)	C13—C12—C11	116.8 (3)
C4—C3—H3A	110.7	C14—C13—C12	122.4 (3)
C2—C3—H3A	110.7	C14—C13—Cl1	119.0 (2)
C4—C3—H3B	110.7	C12—C13—Cl1	118.7 (3)
C2—C3—H3B	110.7	C15—C14—C13	118.9 (3)
H3A—C3—H3B	108.8	C15—C14—H14	120.5
C3—C4—C5	106.9 (3)	C13—C14—H14	120.5
C3—C4—H4A	110.3	C14—C15—C16	121.0 (3)
C5—C4—H4A	110.3	C14—C15—Cl2	119.1 (3)
C3—C4—H4B	110.3	C16—C15—Cl2	119.8 (3)
C5—C4—H4B	110.3	C11—C16—C15	120.1 (3)
H4A—C4—H4B	108.6	C11—C16—H16	120.0
C4—C5—C1	106.4 (3)	C15—C16—H16	120.0
C4—C5—H5A	110.4	N2—C17—C18	121.4 (3)
C1—C5—H5A	110.4	N2—C17—H17	119.3
C4—C5—H5B	110.4	C18—C17—H17	119.3
C1—C5—H5B	110.4	C19—C18—C23	120.4 (3)
H5A—C5—H5B	108.6	C19—C18—C17	120.7 (3)
N2—C6—C1	112.4 (2)	C23—C18—C17	118.9 (3)
N2—C6—C7	111.7 (3)	O2—C19—C18	122.4 (3)
C1—C6—C7	103.7 (3)	O2—C19—C20	119.5 (3)
N2—C6—H6	109.6	C18—C19—C20	118.1 (3)
C1—C6—H6	109.6	C21—C20—C19	121.7 (3)
C7—C6—H6	109.6	C21—C20—Cl3	118.9 (2)
C6—C7—C8	104.0 (3)	C19—C20—Cl3	119.4 (3)
C6—C7—H7A	111.0	C22—C21—C20	118.8 (3)
C8—C7—H7A	111.0	C22—C21—H21	120.6
C6—C7—H7B	111.0	C20—C21—H21	120.6
C8—C7—H7B	111.0	C23—C22—C21	122.0 (3)
H7A—C7—H7B	109.0	C23—C22—Cl4	119.4 (3)
C9—C8—C7	106.6 (3)	C21—C22—Cl4	118.6 (3)
C9—C8—H8A	110.4	C22—C23—C18	119.1 (3)
C7—C8—H8A	110.4	C22—C23—H23	120.5
C9—C8—H8B	110.4	C18—C23—H23	120.5
C10—N1—C2—C3	119.2 (4)	C10—C11—C12—O1	−0.7 (5)
C10—N1—C2—C1	−123.3 (3)	C16—C11—C12—C13	1.3 (5)

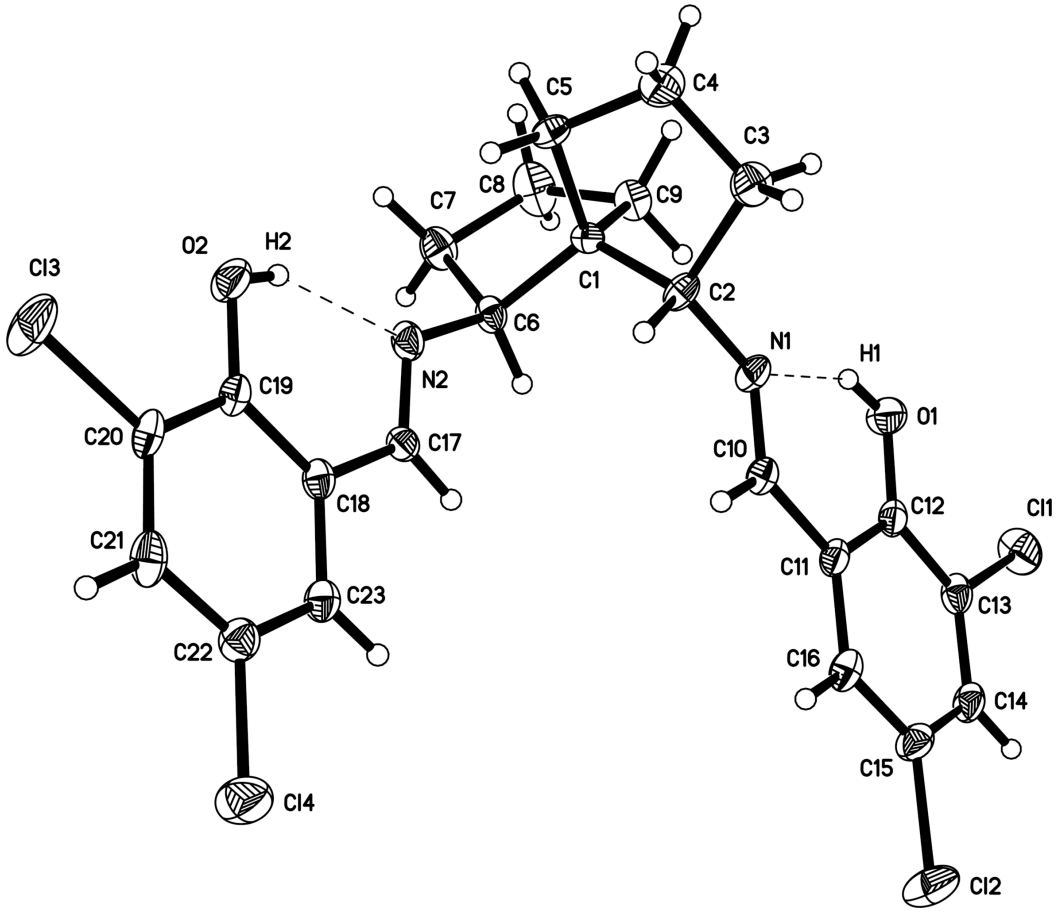
C6—C1—C2—N1	71.0 (3)	C10—C11—C12—C13	178.6 (3)
C9—C1—C2—N1	−46.3 (4)	O1—C12—C13—C14	178.1 (3)
C5—C1—C2—N1	−164.0 (3)	C11—C12—C13—C14	−1.2 (5)
C6—C1—C2—C3	−166.5 (2)	O1—C12—C13—Cl1	−1.7 (5)
C9—C1—C2—C3	76.1 (3)	C11—C12—C13—Cl1	179.0 (3)
C5—C1—C2—C3	−41.5 (3)	C12—C13—C14—C15	0.2 (6)
N1—C2—C3—C4	156.4 (3)	Cl1—C13—C14—C15	180.0 (3)
C1—C2—C3—C4	33.1 (3)	C13—C14—C15—C16	0.8 (6)
C2—C3—C4—C5	−10.6 (4)	C13—C14—C15—Cl2	−179.3 (3)
C3—C4—C5—C1	−15.9 (4)	C12—C11—C16—C15	−0.3 (5)
C6—C1—C5—C4	161.9 (3)	C10—C11—C16—C15	−177.7 (3)
C9—C1—C5—C4	−84.3 (3)	C14—C15—C16—C11	−0.7 (6)
C2—C1—C5—C4	35.4 (3)	Cl2—C15—C16—C11	179.4 (3)
C17—N2—C6—C1	−109.3 (3)	C6—N2—C17—C18	176.3 (3)
C17—N2—C6—C7	134.5 (3)	N2—C17—C18—C19	3.4 (5)
C9—C1—C6—N2	−163.8 (3)	N2—C17—C18—C23	−175.3 (3)
C2—C1—C6—N2	72.4 (3)	C23—C18—C19—O2	178.6 (3)
C5—C1—C6—N2	−44.8 (4)	C17—C18—C19—O2	−0.1 (5)
C9—C1—C6—C7	−42.9 (3)	C23—C18—C19—C20	−1.0 (5)
C2—C1—C6—C7	−166.7 (2)	C17—C18—C19—C20	−179.7 (3)
C5—C1—C6—C7	76.1 (3)	O2—C19—C20—C21	−178.9 (3)
N2—C6—C7—C8	155.0 (3)	C18—C19—C20—C21	0.7 (5)
C1—C6—C7—C8	33.7 (3)	O2—C19—C20—Cl3	0.7 (5)
C6—C7—C8—C9	−11.1 (4)	C18—C19—C20—Cl3	−179.7 (3)
C7—C8—C9—C1	−15.7 (4)	C19—C20—C21—C22	0.1 (6)
C6—C1—C9—C8	36.1 (3)	Cl3—C20—C21—C22	−179.5 (3)
C2—C1—C9—C8	162.4 (3)	C20—C21—C22—C23	−0.6 (6)
C5—C1—C9—C8	−86.1 (3)	C20—C21—C22—Cl4	178.1 (3)
C2—N1—C10—C11	−178.2 (3)	C21—C22—C23—C18	0.3 (6)
N1—C10—C11—C16	178.4 (3)	Cl4—C22—C23—C18	−178.4 (3)
N1—C10—C11—C12	1.0 (5)	C19—C18—C23—C22	0.5 (5)
C16—C11—C12—O1	−178.0 (3)	C17—C18—C23—C22	179.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N2	0.82	1.88	2.597 (3)	146
O1—H1···N1	0.82	1.86	2.589 (3)	148
C2—H2A···Cl1^{i}	0.98	3.10	4.005 (3)	154
C7—H7B···Cl1^{ii}	0.97	2.87	3.734 (4)	149
C14—H14···Cl2^{iii}	0.93	3.01	3.886 (3)	158
C6—H6···Cl3^{iv}	0.98	3.10	4.027 (3)	158
C3—H3A···Cl3^{v}	0.97	3.00	3.874 (4)	151
C21—H21···Cl4^{vi}	0.93	3.01	3.886 (3)	158
C23—H23···Cl4^{vii}	0.93	3.16	4.023 (3)	154

Symmetry codes: (i) *x*−1, *y*, *z*−1; (ii) *x*, *y*, *z*−1; (iii) −*x*+1, −*y*+1, −*z*+2; (iv) *x*+1, *y*, *z*+1; (v) *x*, *y*, *z*+1; (vi) −*x*−1, −*y*+1, −*z*−1; (vii) −*x*, −*y*+1, −*z*.

Fig. 1


C5
C4
C3
C8
C9
C7
O2
H2
Cl3
C1
C2
C6
N1
H1
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C19
O1
C20
C17
C10
C18
C12
Cl1
C21
C11
C23
C13
C22
C16
C14
C15
Cl4
Cl2