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Key indicators

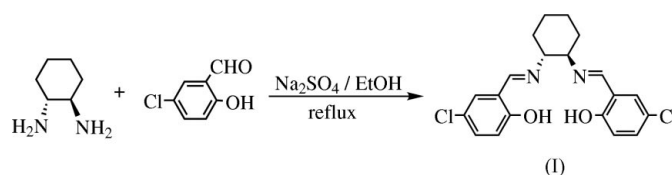
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.054
 wR factor = 0.155
Data-to-parameter ratio = 15.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(*R,R*)-*N,N'*-Bis(5-chlorosalicylidene)-
1,2-cyclohexanediamine**

In the title compound {systematic name: (*R,R*)-4,4'-dichloro-2,2'-[cyclohexane-1,2-diylbis(nitrilomethylidene)]diphenol}, $\text{C}_{20}\text{H}_{20}\text{Cl}_2\text{N}_2\text{O}_2$, there are two chiral C atoms and two intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds. The molecule lies on a twofold rotation axis. The crystal structure is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, which link the molecules into one-dimensional helical chains along the b axis.

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Comment

Chiral salen compounds are important chiral ligands widely used in asymmetric catalytic synthesis (Canail & Sherrington, 1999; Jacobsen, 2000). The structure of chiral salen compounds has a crucial effect on enantioselectivity and activity in asymmetric catalytic reactions (Nicewicz *et al.*, 2004; Yao *et al.*, 2001). Our research is focused on asymmetric synthesis catalysed by chiral salen-metal complexes (Zhu *et al.*, 2004). In order to study the relationship between the structures and properties of such salen compounds, we have synthesized the chiral ligand (*R,R*)-*N,N'*-bis(5-chloro-salicylidene)-1,2-cyclohexanediamine, (I), and present its crystal structure here.



The molecular structure of (I) (Fig. 1) contains two chiral C atoms in (*R,R*)-diastereomeric form, the molecule lying on a twofold rotation axis. Intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds (Fig. 1, Table 1) are present.

The crystal packing is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1), which link the molecules into one-dimensional helical chains along the b axis (Fig. 2).

Experimental

Under nitrogen, a mixture of (*R,R*)-1,2-cyclohexanediamine (342 mg, 3 mmol), Na_2SO_4 (2 g) and 5-chloro-2-hydroxybenzaldehyde (939 mg, 6 mmol) in absolute ethanol (10 ml) was refluxed for about 12 h to yield a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was dissolved in CH_2Cl_2 (50 ml) and washed with water (2×10 ml) and brine (10 ml). After drying over Na_2SO_4 , the solvent was removed under vacuum and a yellow solid was isolated in 85% yield (1.0 g). Yellow single crystals of (I) suitable for X-ray analysis were grown from a solution in hexane by slow evaporation of the solvent at room temperature over a period of about a week. Spectroscopic analysis:

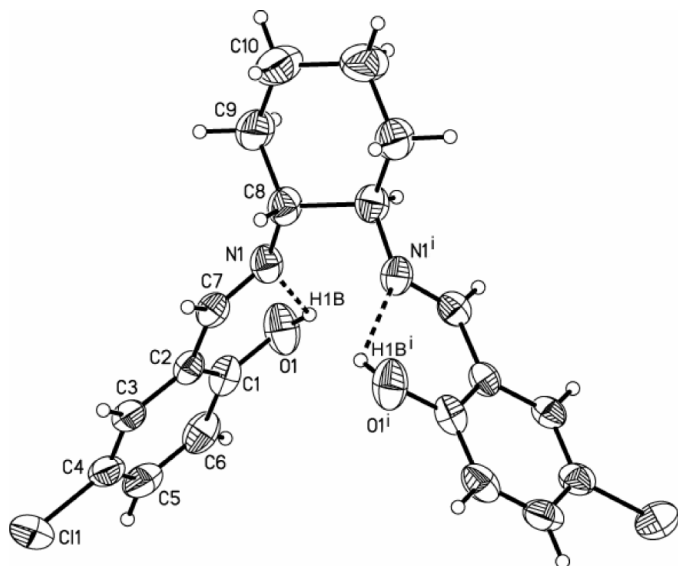


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) $1 - x, 1 - y, z$.]

^1H NMR (300 MHz, CDCl_3 , δ , p.p.m.): 13.13 (*s*, 2H), 8.10 (*s*, 2H), 7.10–7.14 (*dd*, $J = 2.0$ and 8.8 Hz, 2H), 6.98–7.04 (*d*, $J = 2.0$ Hz, 2H), 6.75–6.78 (*d*, $J = 8.8$ Hz, 2H), 3.23–3.26 (*m*, 2H), 1.82–1.87 (*m*, 4H), 1.62–1.65 (*m*, 2H), 1.36–1.42 (*m*, 2H); IR (KBr, ν , cm^{-1}): 3430, 2924, 2856, 1633, 1478, 1371, 1282, 1185, 1293, 1034, 977; analysis calculated for $\text{C}_{20}\text{H}_{20}\text{Cl}_2\text{N}_2\text{O}_2$ (%): C 61.39, H 5.15, N 7.16; found: C 61.28, H 5.24, N 7.22.

Crystal data

$\text{C}_{20}\text{H}_{20}\text{Cl}_2\text{N}_2\text{O}_2$
 $M_r = 391.28$
Orthorhombic, $P2_12_12$
 $a = 18.990$ (6) Å
 $b = 5.829$ (2) Å
 $c = 8.839$ (3) Å
 $V = 978.4$ (6) Å³
 $Z = 2$
 $D_x = 1.328$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 780 reflections
 $\theta = 2.5$ – 25.0°
 $\mu = 0.35$ mm⁻¹
 $T = 293$ (2) K
Block, yellow
 $0.30 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{\min} = 0.91$, $T_{\max} = 0.93$
5037 measured reflections

1879 independent reflections
1554 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$
 $\theta_{\max} = 26.0^\circ$
 $h = -21 \rightarrow 23$
 $k = -5 \rightarrow 7$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.155$
 $S = 1.05$
1879 reflections
118 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2 + 0.127P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.34$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³
Absolute structure: Flack (1983), with 732 Friedel pairs
Flack parameter = 0.13 (15)

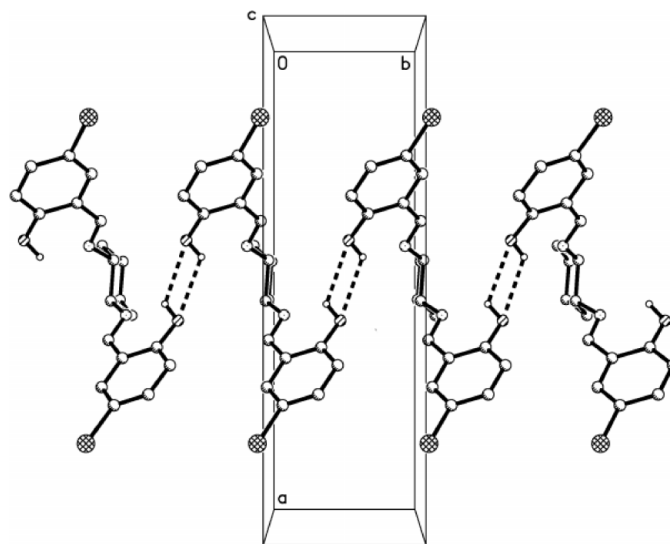


Figure 2
The one-dimensional hydrogen-bonded helical chain in (I), viewed down the *c* axis.

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1B...N1	0.85	2.03	2.599 (4)	124
O1–H1B...O1 ⁱ	0.85	2.46	2.903 (5)	114

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

All H atoms were positioned geometrically and refined using a riding model, with C–H distances in the range 0.93–0.98 Å and an O–H distance of 0.85 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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