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Crystal Structure of N,N-Disalicylidene-(R,S)(S,R)-1,2-ethanediamine

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N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine crystallizes in orthorhombic space group Pbca with $a = 9.5634(6)$, $b = 14.2917(9)$, $c = 16.9181(8)$ Å and $Z = 4$. The crystal structure was solved by direct methods and refined by full-matrix least squares to final values $R1 = 0.0 + 399$ and $wR2 = 0.1004$ with 2755 reflections ($I > 2\sigma(I)$). The N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine molecules exhibit intramolecular N-H···O and are connected by C-H···O and C-H···π interactions to form a 2D supramolecular network.

INTRODUCTION

Schiff bases have been investigated extensively for many years due to their importance in chemistry and multitude of applications [1], such as in the development of catalysis, magnetism, molecular architectures and materials chemistry. The coordinate properties of salicylaldehyde Schiff bases are similar to those of 8-hydroxy-quinoline, because of the latter's hydroxyl group(s), coordination nitrogen atom and delocalized π-system [2]. In this paper, we wish to report the crystal structure of N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine.

METHODS

The N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine ligand was prepared as previously reported [3]. The slow evaporation of the methanolic solution of the N,N-
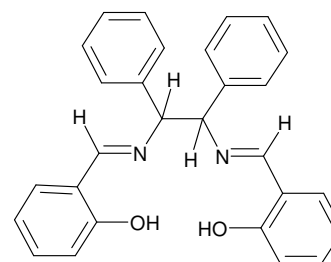
N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine

disalicylidene-(R,S)(S,R)-1,2-ethanediamine at room temperature yielded suitable crystals for X-ray analysis by full-matrix least squares using the program SHELXTL-97.

The crystal structure was solved by direct methods and refined [4]. All H atoms were calculated geometrically.

RESULTS AND DISSCUSION

The corresponding crystal data and structure refinements

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are summarized in Table 1, and all atomic coordinates and equivalent isotropic displacement parameters are given in Table 2. Obviously, the crystal structure is orthorhombic, space group Pbca with a = 9.5634(6), b = 14.2917(9), c = 16.9181(8) Å, Z = 4, $R1$ = 0.0399 and $wR2$ = 0.1004 with 2755 reflections [$I > 2\sigma(I)$].

As seen in Fig. 1, there are classical intramolecular N-H···O hydrogen bonds between the hydrogen atoms of the OH groups and nitrogen atoms in N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine. The corresponding distances and angles for the hydrogen bonds are given in Table 3. A search was also generally made for non-classical C-H···O approaches in this compound and it revealed that there are C-H···O interactions, weak hydrogen bonding, between the hydrogen atoms of phenyl rings and oxygen atoms belonging to phenolic rings with distances of O···HC(ph) = 2.495 Å {HC(ph)···O = 3.391 Å and <O···H-C = 161.81°}. The C-H···O distance range in this compound is much shorter than similar compounds previously reported [5], which suggests strong interactions within this class of weak noncovalent contacts. Consequently, C-H···O interactions cause the monomeric units o form a two-dimensional network as illustrated in Fig. 2. Additionally, several C-H···π interactions with an average distance of 3.30 Å further reinforce the crystal structure.

Supplementary Material

Complete bond lengths and angles, coordinates and

Table 1. Crystal Data and Structure Refinement for N,N-Disalicylidene-(R,S)(S,R)-1,2-ethanediamine

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Empirical formula	$C_{28}H_{24}N_2O_2$
Formula weight	420.50
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 9.5634(6)Å
	b = 14.2917(9) Å
	c =16.9181(8)Å
Volume	2312.3(2) Å^3
Z, Calculated density	4, 1.208 mg m^{-3}
Absorption coefficient (MoKα)	0.076 mm^{-1}
F(000)	888
Crystal size	0.470 × 0.413 × 0.330 mm
θ range for data collection	2.41 to 27.86 deg.
Limiting indices	-12 ≤ h ≤ 12, -18 ≤ k ≤ 18, -22 ≤ l ≤ 22
Reflections collected/unique	37008/2755 [R(int) = 0.0731]
Completeness to theta = 25.02	95.5%
Absorption correction	Integration
Max. and min. transmission	0.9801 and 0.9606
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2755/0/153
Goodness-of-fit on F^2	1.050
Final R indices [for 1829 rfls with I > 2σ(I)]	R1 = 0.0399, wR2 = 0.1004
R indices (all data)	R1 = 0.0655, wR2 = 0.1102
Largest diff. peak and hole	0.126 and -0.140 e. Å^{-3}
$(\Delta/\delta)_{max}$	0.000

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters
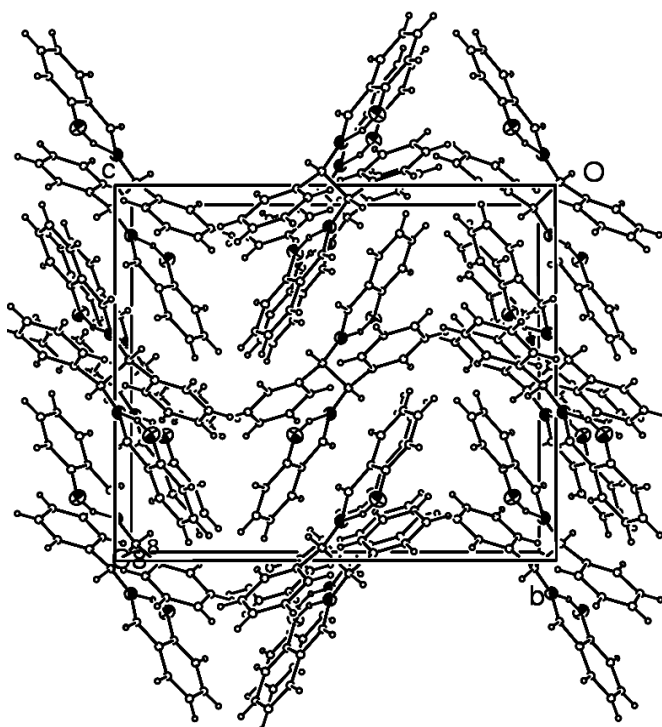($\mathring{A}^2 \times 10^4$) for N,N-Disalicylidene-(R,S)(S,R)-1,2-ethanediamine

	x ($\times 10^5$)	y ($\times 10^5$)	z ($\times 10^5$)	U(eq)*
C(1)	60653(13)	24634(9)	41959(7)	591(3)
C(2)	74109(13)	25808(9)	38842(8)	602(3)
C(3)	79558(16)	19165(11)	33713(9)	745(4)
C(4)	71817(19)	11566(11)	31629(10)	838(5)
C(5)	5855(2)	10241(11)	34642(11)	908(5)
C(6)	53071(17)	16743(10)	39751(10)	789(4)
C(7)	54496(13)	31668(9)	47072(8)	596(3)
C(8)	53294(12)	46530(8)	53071(7)	537(3)
C(9)	62922(12)	51710(9)	58587(7)	566(3)
C(10)	76010(14)	54714(10)	56135(9)	715(4)
C(11)	84094(15)	60292(12)	61032(11)	835(5)
C(12)	79235(17)	62888(11)	68350(10)	815(5)
C(13)	66407(17)	59946(10)	70843(9)	756(4)
C(14)	58323(14)	54387(9)	66000(8)	651(3)
N(1)	60741(10)	39321(7)	48669(6)	564(3)
O(1)	81878(10)	33355(8)	40685(7)	766(3)

*U(eq) is defined as one-third of the trace of the orthogonalized Uij tensor.

displacement parameters have been deposited at the Cambridge Crystallography Data Center. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK upon request, quoting the deposition number
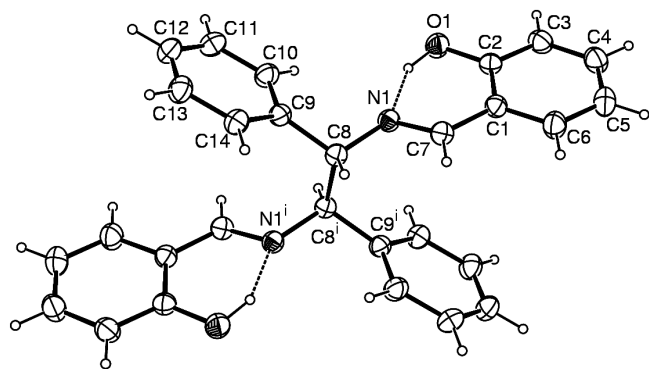
Fig. 1. The ORTEP diagram of N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine, showing hydrogen bonding.

Fig. 2. Unit cell plot of N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine.

Table 3. Hydrogen Bond Distances and Angles (in Å and °, respectively) of N,N-Disalicylidene-(R,S)(S,R)-1,2-ethanediamine

D-H	d(D-H)	D(H…A)	d(D…A)	DHA	A
O(1)-H(1)	0.96(2)	1.69(2)	2.576(19)	152	N(1)

291312 for N,N-disalicylidene-(R,S)(S,R)-1,2-ethanediamine.

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