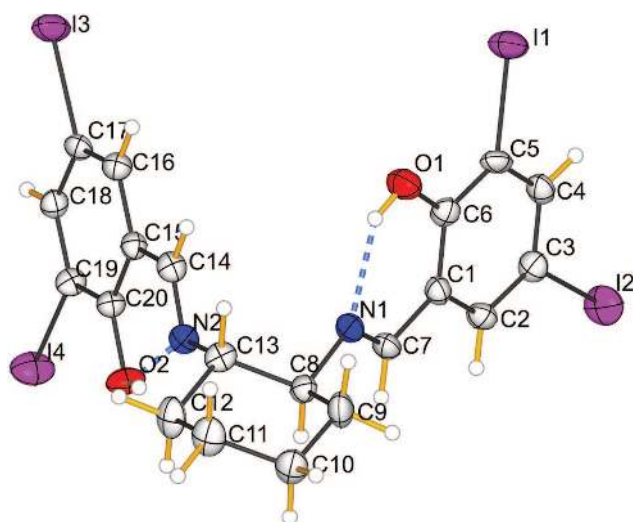


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# Crystal structure of *rac-trans-N,N'*-bis(3,5-diiodosalicylidene)-1,2-cyclohexanediamine, $C_{20}H_{18}I_4N_2O_2$



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## Abstract

$C_{20}H_{18}I_4N_2O_2$ , monoclinic,  $P2_1/c$  (no. 14),  $a = 15.8722(9)$  Å,  $b = 9.5990(5)$  Å,  $c = 16.8433(8)$  Å,  $V = 2421.2(2)$  Å<sup>3</sup>,  $Z = 4$ ;  $R_{gt}(F) = 0.0387$ ,  $wR_{ref}(F^2) = 0.0754$ ,  $T = 293$  K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

All commercially available reagents were used as supplied. The title compound was synthesized with the

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.26 × 0.12 mm
Wavelength:	Mo K $\alpha$ radiation (0.71073 Å)
$\mu$ :	5.17 mm <sup>-1</sup>
Diffractometer, scan mode:	Xcalibur, $\varphi$ and $\omega$
$\theta_{max}$ , completeness:	26.0°, >99%
$N(hkl)_{measured}$ , $N(hkl)_{unique}$ , $R_{int}$ :	11367, 4753, 0.033
Criterion for $I_{obs}$ , $N(hkl)_{gt}$ :	$I_{obs} > 2 \sigma(I_{obs})$ , 3478
$N(param)_{refined}$ :	255
Programs:	CrysAlis <sup>PRO</sup> [1], Olex2 [2], SHELX [3], Diamond [4]

following procedure: 3,5-diiodosalicylaldehyde 0.070 g (0.188 mmol) and (+-)-1,2-cyclohexanediamine 0.01 mL (0.094 mmol) were dissolved in 20 mL ethanol and 10 mL *N,N*-dimethylformamide (DMF). The yellow suspension was then stirred at 60 °C for 3 h and filtrated. The orange-yellow crystals of the title compound slowly appears after one week.

## Experimental details

All hydrogen atoms were placed in calculated positions using a riding model. Aromatic C–H distances were set to 0.93 Å and their  $U_{iso}$  set to 1.2 times the  $U_{eq}$  of the parent atom.

## Comment

Salen type Schiff bases are important ligands that can act as efficient tetradentate ligands to construct various complexes with rare earth and transition metals [5–8]. As a part of our current research interest on Schiff bases and their properties, we report herein a new salen-type ligand.

The title compound crystallizes in the monoclinic space group  $P2_1/c$ , which unit cell is filled by two pairs of racemic neutral molecules. The bond lengths and angles are within the normal ranges and are comparable to related structures [9, 10]. There are two strong intramolecular O–H $\cdots$ N hydrogen bonds between the hydroxyl group and the Schiff base nitrogen atom, with the distance of O(1)–H(1) $\cdots$ N(1) and O(2)–H(2) $\cdots$ N(2) are 2.618(4) Å and 2.566(3) Å, respectively. Besides the intramolecular hydrogen bonds, there may exist weak intermolecular C–H $\cdots$ I hydrogen bonds

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> <sup>*</sup> / <i>U</i> <sub>eq</sub>
I1	0.14672(3)	0.56134(5)	0.70889(2)	0.06458(16)
I2	−0.13281(3)	0.13082(6)	0.55975(3)	0.06588(16)
I3	0.57208(3)	0.04416(5)	0.77262(2)	0.06216(16)
I4	0.38537(3)	−0.33119(4)	0.48458(2)	0.06026(15)
O1	0.1852(3)	0.4939(5)	0.5407(2)	0.0536(11)
H1	0.1983	0.4748	0.4988	0.080*
O2	0.3200(3)	−0.0392(4)	0.4040(2)	0.0463(10)
H2	0.3132	0.0340	0.3775	0.069*
N1	0.1625(3)	0.3835(5)	0.3929(2)	0.0395(12)
N2	0.3133(3)	0.2266(5)	0.3864(2)	0.0368(11)
C1	0.0761(3)	0.3147(6)	0.4795(3)	0.0354(13)
C2	0.0064(3)	0.2329(6)	0.4855(3)	0.0398(14)
H2A	−0.0201	0.1681	0.4436	0.048*
C3	−0.0238(3)	0.2464(6)	0.5523(3)	0.0404(14)
C4	0.0164(3)	0.3407(6)	0.6157(3)	0.0413(14)
H4	−0.0042	0.3496	0.6611	0.050*
C5	0.0871(4)	0.4220(6)	0.6118(3)	0.0403(14)
C6	0.1173(3)	0.4122(6)	0.5433(3)	0.0390(14)
C7	0.1041(3)	0.3047(6)	0.4048(3)	0.0399(14)
H7	0.0776	0.2374	0.3645	0.048*
C8	0.1823(3)	0.3686(6)	0.3146(3)	0.0360(13)
H8	0.1521	0.2859	0.2841	0.043*
C9	0.1505(4)	0.4980(7)	0.2601(3)	0.0512(17)
H9A	0.0860	0.5045	0.2440	0.061*
H9B	0.1758	0.5802	0.2929	0.061*
C10	0.1767(4)	0.4956(7)	0.1819(3)	0.0530(17)
H10A	0.1587	0.5824	0.1515	0.064*
H10B	0.1455	0.4203	0.1457	0.064*
C11	0.2763(4)	0.4762(7)	0.2026(3)	0.0547(18)
H11A	0.3076	0.5566	0.2334	0.066*
H11B	0.2904	0.4686	0.1510	0.066*
C12	0.3072(4)	0.3453(7)	0.2554(3)	0.0511(16)
H12A	0.3713	0.3353	0.2697	0.061*
H12B	0.2790	0.2642	0.2230	0.061*
C13	0.2832(3)	0.3532(6)	0.3360(3)	0.0353(13)
H13	0.3129	0.4342	0.3689	0.042*
C14	0.3612(3)	0.2397(6)	0.4637(3)	0.0377(13)
H14	0.3755	0.3282	0.4867	0.045*
C15	0.3942(3)	0.1160(6)	0.5167(3)	0.0327(12)
C16	0.4509(3)	0.1343(6)	0.5998(3)	0.0384(14)
H16	0.4651	0.2232	0.6221	0.046*
C17	0.4851(3)	0.0194(6)	0.6477(3)	0.0356(13)
C18	0.4670(3)	−0.1133(6)	0.6160(3)	0.0361(13)
H18	0.4922	−0.1899	0.6492	0.043*
C19	0.4111(3)	−0.1314(6)	0.5343(3)	0.0339(13)
C20	0.3739(3)	−0.0172(6)	0.4837(3)	0.0334(13)

and aromatic face-to-face  $\pi$ – $\pi$  interactions to join adjacent molecules. [interactions Cg1 $\cdots$  Cg1(−*X*, 1 − *Y*, 1 − *Z*) and Cg3 $\cdots$  Cg3 (1 − *X*, −*Y*, 1 − *Z*) centroid-to-centroid with distance = 3.7552 Å and 3.6277 Å, respectively, where Cg1 is the centroids of the C1–C6 ring and Cg3 is the centroids of the C15–C20 ring].

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