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

Table 1: Data collection and handling.

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

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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, $V = 2421.2(2)$ Å³, $Z = 4$; $R_{\text{gt}}(F) = 0.0387$, $wR_{\text{ref}}(F^2) = 0.0754$, $T = 293$ 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

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...N hydrogen bonds between the hydroxyl group and the Schiff base nitrogen atom, with the distance of O(1)–H(1)...N(1) and O(2)–H(2)...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...I hydrogen bonds

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
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 π – π interactions to join adjacent molecules. [interactions Cg1... Cg1(−X, 1 − Y, 1 − Z) and Cg3... 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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References

- Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.38.41, Agilent Technologies UK Ltd, Oxford, UK (2015).
- Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
- Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
- Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany (2012).
- Venkataramanan, N. S.; Kuppuraj, G.; Rajagopal, S.: Metal–salen complexes as efficient catalysts for the oxygenation of heteroatom containing organic compounds–synthetic and mechanistic aspects. *Coord. Chem. Rev.* **249** (2005) 1249–1268.
- Mukherjee, P.; Biswas, C.; Drew, M. G. B.; Ghosh, A.: Structural variations in Ni (II) complexes of salen type di-Schiff base ligands. *Polyhedron* **26** (2007) 3121–3128.
- Li, G.; Zhou, W. M.; Zhu, L. C.; Wang, L.: Crystal structure of (Z)-1-(((3-acetyl-2-hydroxyphenyl)amino)methylene)naphthalen-2(1H)-one, C₁₉H₁₅NO₃. *Z. Kristallogr. NCS* **231** (2016) 315–317.
- Hadjoudis, E.; Rontoyianni, A.; Ambroziak, K.; Dziembowska, T.; Mavridis, I. M.: Photochromism and thermochromism of solid trans-*N,N'*-bis-(salicylidene)-1,2-cyclohexanediamines and trans-*N,N'*-bis-(2-hydroxy-naphylidene)-1,2-cyclohexanediamine. *J. Photochem. Photobiol., A* **162** (2004) 521–530.
- Köysal, Y.; Bülbül, H.; Kantar, E. N.; Coşkun, E.; Sahin, S.; Dege, N.: Crystal structure of (E)-4-nitro-2-((2-phenoxyphenylimino)methyl)phenol, C₁₉H₁₄N₂O₄. *Z. Kristallogr. NCS* **232** (2016) 9–10.
- Sui, Y.; Hu, R. H.; Luo, Z. G.; Lin, W. H.; Liu, D. S.: Synthesis, structure and properties of an erbium(III) complex with chiral salen-type Schiff base ligand. *Z. Anorg. Allg. Chem.* **641** (2015) 1566–1570.