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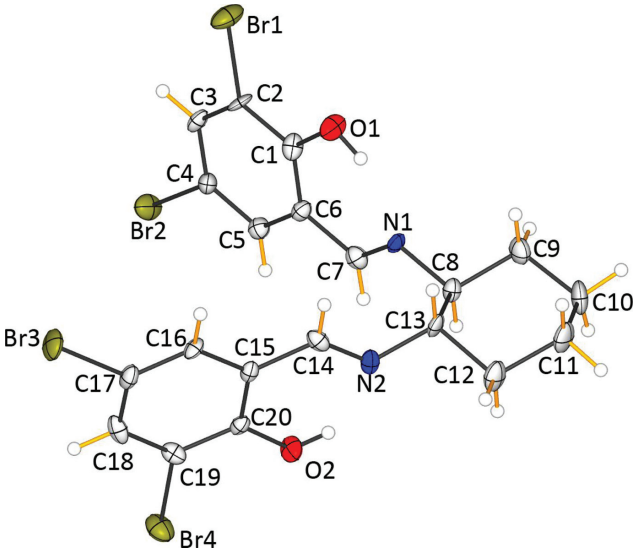
Crystal structure of rac-*trans*-*N*,*N*′-bis(3,5-dibromosalicylidene)-1,2-cyclohexanediamine, $C_{20}H_{18}Br_4N_2O_2$



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Abstract

$C_{20}H_{18}Br_4N_2O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 9.1119(6)$ Å, $b = 17.8211(12)$ Å, $c = 13.5228(8)$ Å, $\beta = 90.809(2)°$, $V = 2195.7(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0448$, $wR_{ref}(F^2) = 0.0963$, $T = 273(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.18 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	7.35 mm^{-1}
Diffractometer, scan mode:	CCD, φ and ω
θ_{max}, completeness:	26.4°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7499, 4348, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3643
$N(param)_{refined}$:	255
Programs:	Bruker [1], SHELX [2], Olex2 [3], Diamond [4]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.45469(6)	−0.16449(3)	0.13869(4)	0.03128(15)
Br2	0.71811(6)	0.12188(3)	0.12434(4)	0.02764(14)
Br3	0.10675(5)	0.03553(3)	−0.36191(4)	0.02908(15)
Br4	0.64831(6)	0.18303(3)	−0.42078(4)	0.02950(15)
C1	0.6642(5)	−0.1210(3)	−0.0044(4)	0.0192(10)
C2	0.5853(5)	−0.0971(3)	0.0782(3)	0.0164(10)
C3	0.6020(5)	−0.0257(3)	0.1152(3)	0.0175(10)
H3	0.5483	−0.0106	0.1697	0.021*
C4	0.6985(5)	0.0235(3)	0.0714(3)	0.0176(10)
C5	0.7784(5)	0.0021(3)	−0.0097(3)	0.0184(10)
H5	0.8435	0.0356	−0.0384	0.022*
C6	0.7615(5)	−0.0697(3)	−0.0485(3)	0.0168(10)
C7	0.8476(5)	−0.0929(3)	−0.1334(4)	0.0199(11)
H7	0.9143	−0.0595	−0.1605	0.024*
C8	0.9238(5)	−0.1804(3)	−0.2541(3)	0.0175(10)
H8	0.9989	−0.1419	−0.2642	0.021*
C9	0.9986(6)	−0.2548(3)	−0.2320(4)	0.0247(12)
H9A	1.0581	−0.2499	−0.1723	0.030*
H9B	0.9245	−0.2927	−0.2202	0.030*
C10	1.0948(6)	−0.2800(3)	−0.3160(4)	0.0283(12)
H10A	1.1345	−0.3293	−0.3014	0.034*
H10B	1.1764	−0.2454	−0.3226	0.034*
C11	1.0071(6)	−0.2828(3)	−0.4132(4)	0.0286(13)
H11A	0.9346	−0.3225	−0.4097	0.034*
H11B	1.0727	−0.2943	−0.4669	0.034*
C12	0.9293(6)	−0.2080(3)	−0.4347(4)	0.0277(12)
H12A	0.8719	−0.2122	−0.4954	0.033*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H12B	1.0017	−0.1688	−0.4435	0.033*
C13	0.8283(5)	−0.1875(3)	−0.3490(3)	0.0180(10)
H13	0.7542	−0.2267	−0.3404	0.022*
C14	0.6165(5)	−0.1116(3)	−0.3607(3)	0.0187(10)
H14	0.5629	−0.1551	−0.3495	0.022*
C15	0.5395(5)	−0.0400(3)	−0.3704(3)	0.0149(10)
C16	0.3859(5)	−0.0370(3)	−0.3647(3)	0.0158(10)
H16	0.3322	−0.0808	−0.3563	0.019*
C17	0.3151(5)	0.0311(3)	−0.3715(3)	0.0199(11)
C18	0.3917(5)	0.0970(3)	−0.3867(3)	0.0212(11)
H18	0.3426	0.1426	−0.3918	0.025*
C19	0.5436(5)	0.0938(3)	−0.3943(3)	0.0169(10)
C20	0.6210(5)	0.0266(3)	−0.3855(3)	0.0151(10)
N1	0.8329(4)	−0.1585(2)	−0.1712(3)	0.0163(8)
N2	0.7567(4)	−0.1155(2)	−0.3673(3)	0.0186(9)
O1	0.6454(4)	−0.1908(2)	−0.0387(3)	0.0263(8)
H1	0.6958	−0.1969	−0.0878	0.039*
O2	0.7674(3)	0.0268(2)	−0.3914(3)	0.0229(8)
H2	0.7975	−0.0165	−0.3939	0.034*

Source of material

All hydrogen atoms were placed in calculated positions and refined using a riding model.

Experimental details

All commercially available reagents were used as supplied. According to classical organic synthesis of Schiff-bases, 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 methanol. The obtained suspension was then stirred at 60 °C for 3 h and filtrated. Colorless crystals of the title compound slowly appear after one week.

Comment

Salen type Schiff base represent a class of compounds that can act as efficient tetradentate ligands to construct various complexes with rare earth and transition metals [5–9]. In order to explore the relationship between molecular structure and physicochemical properties of halogenated Schiff-bases, we report a new bromo-substituted salen-type ligand.

The unit cell content is constructed by two pairs of related racemic molecules, and overall exhibiting centrosymmetric configuration. The bond lengths and angles are within the normal ranges and are closed to the related structures of a chloro [10] and iodo [11] analogue.

There are two strong intramolecular O—H···N hydrogen bonds between the hydroxyl group and the N-atom, with the O—H···N distances 1.8326(1) and 1.8397(1) Å, respectively. Further research showed that C—H···π hydrogen bonds and aromatic face-to-face π-π interactions play important roles to construct a three-dimensional framework. [C(3)—H(3)···Cg(3) distance = 2.85 Å; Cg1···Cg1 ($1-x$, $-y$, $-z$) and Cg3···Cg3 ($1-x$, $-y$, $1-z$) centroid-to-centroid distance = 3.824(3) and 3.495(2) Å, respectively, where Cg1 is the centroid of the C1—C6 ring and Cg3 is the centroid of the C15—C20 ring.]

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