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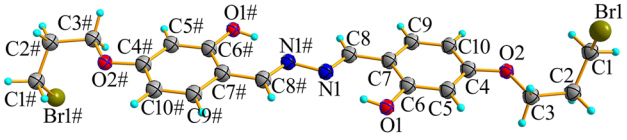
Crystal structure of 6,6′-((1*E*,1′*E*)-hydrazine-1,2-diylidenebis(methanylylidene))bis(3-(3-bromopropoxy)phenol), $C_{20}H_{22}Br_2N_2O_4$



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Abstract

$C_{20}H_{22}Br_2N_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 5.0060(12) Å, b = 9.135(2) Å, c = 11.650(3) Å, V = 518.6(2) Å^3, Z = 1, $R_{gt}(F)$ = 0.0474, w$R_{ref}(F^2)$ = 0.1270, T = 273(2) K, β = 78.492(7).

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

4-(3–Bromopropoxy)-2-hydroxybenzaldehyde was synthesized with the method specified in the literature [5], 4-(3-bromopropoxy)-2-hydroxybenzaldehyde (0.51 g, 2 mmol) and hydrazine hydrate (0.05 g, 1 mmol) were weighed and added to a 50 mL round-bottom flask. Then 30 mL of ethanol was added, heated to reflux for 9 h, cooled to precipitate yellow solid, filtered, washed three times with ethanol, dried under vacuum to afford the target compound (0.41 g), with a yield of 80 %. The yellow solid was dissolved in a mixed solution of trichloromethane and ethanol. After five days of slow evaporation, crystals at a size suitable for X-ray diffraction were obtained.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.16 × 0.14 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.94 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7312, 1806, 0.084
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1461
$N(param)_{refined}$:	128
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

2 Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1. Using Olex2 [1], the structure was solved using Charge Flipping and refined with the Shelxl [3] refinement. All hydrogen atoms were positioned geometrically, with the d (C—H) = 0.97–0.99 Å, U_{iso}(H) = 1.2 times U_{eq}(C) and U_{iso}(H) = 1.5 times U_{eq}(O).

3 Comment

Schiff base compounds have been widely used in generation of special Schiff base complexes [6, 7]. The salicylaldehyde-based Schiff base compounds synthesized based on the reaction of amines with aldehydes are easy to undergo intramolecular proton transfer. Under the excitation of light, the molecule absorbs energy for enol-keto transformation, thus providing a large Stokes shift. The Schiff base compounds based on excited state intramolecular proton transfer (ESIPT) mechanism mostly have good luminescent properties [8, 9]. We report herein the crystal structure of 6,6′-((1*E*,1′*E*)-hydrazine-1,2-diylidenebis(methanylylidene))bis(3-(3-bromopropoxy)phenol), which shows that 4-(3-bromopropoxy)-2-hydroxybenzaldehyde was condensed with hydrazine hydrate to form the Schiff base. The bond length of C8=N1 was 1.285(6) Å, all bond lengths and bond angles were within the normal ranges [10]. The two aryl units are completely parallel to each other, according to the space group symmetry.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0134 (11)	0.2705 (6)	0.9159 (5)	0.0519 (13)
H1A	0.9102	0.3442	0.9593	0.062*
H1B	1.1396	0.2263	0.9627	0.062*
C2	1.1721 (10)	0.3444 (6)	0.8014 (5)	0.0533 (14)
H2A	1.2720	0.2699	0.7578	0.064*
H2B	1.3046	0.4112	0.8183	0.064*
C3	1.0020 (11)	0.4294 (6)	0.7243 (5)	0.0505 (13)
H3A	1.1170	0.4727	0.6515	0.061*
H3B	0.8696	0.3646	0.7050	0.061*
C4	0.7011 (10)	0.6363 (5)	0.7383 (4)	0.0407 (11)
C5	0.6503 (10)	0.6333 (5)	0.6266 (4)	0.0425 (11)
H5	0.7348	0.5635	0.5798	0.051*
C6	0.4747 (10)	0.7334 (5)	0.5841 (4)	0.0383 (11)
C7	0.3445 (9)	0.8401 (5)	0.6531 (4)	0.0379 (11)
C8	0.1548 (10)	0.9443 (5)	0.6134 (4)	0.0421 (11)
H8	0.0736	1.0119	0.6633	0.051*
C9	0.5770 (11)	0.7420 (6)	0.8075 (5)	0.0523 (13)
H9	0.6130	0.7459	0.8824	0.063*
C10	0.4017 (11)	0.8404 (6)	0.7651 (4)	0.0484 (13)
H10	0.3181	0.9097	0.8127	0.058*
N1	0.0945 (8)	0.9466 (4)	0.5111 (4)	0.0415 (9)
O1	0.4293 (9)	0.7248 (5)	0.4742 (3)	0.0631 (11)
H1	0.3036	0.7793	0.4626	0.095*
O2	0.8661 (7)	0.5427 (4)	0.7888 (3)	0.0530 (9)
Br1	0.76524 (11)	0.11976 (6)	0.89428 (5)	0.0552 (3)

The molecule contains an intramolecular hydrogen bond of O1—H1···N1, C1—H1A···O1, C3—H3B···O1.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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