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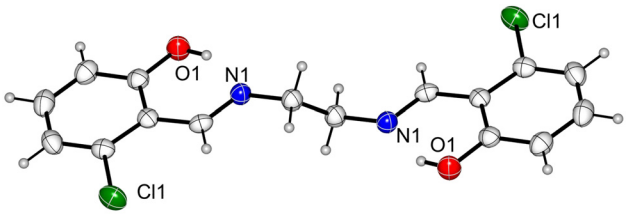
Crystal structure of 2,2′-[ethane-1,2-diylbis(azanylylidenemethylylidene)] bis(6-chlorophenol), $C_{16}H_{14}Cl_2N_2O_2$



https://doi.org/10.1515/ncrs-2020-0642
Received December 27, 2020; accepted xxx;
published online February 11, 2021

Abstract

$C_{16}H_{14}Cl_2N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 6.8492(14) Å, b = 11.515(2) Å, c = 10.018(2) Å, β = 107.11(3)°, V = 755.2(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0592, $wR_{ref}(F^2)$ = 0.1369, T = 293(2) K.

CCDC no.: 2052614

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.

Source of material

2-Chloro-6-hydroxybenzaldehyde and 30 ml of anhydrous methanol were added in a conical flask, monitored with a temperature probe and warmed up to 60 °C with stirring. Then 1.0 mmol of ethylenediamine was dropwisely added. Over 30 min the mixture gradually turned to light yellow, and was refluxed for another 16 h. The obtained solution was filtered and the filtrate was left to crystallise in an open vessel at room temperature. Colorless plates crystals were isolated from the yellow mother liquor.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.44 mm^{-1}
Diffractometer, scan mode:	Bruker D8/APEX2, ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3010, 1550, 0.060
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1110
$N(param)_{refined}$:	101
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Experimental details

The structure was solved with the Olex2 program [2] as an interface with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with O–H = 0.84 (phenolic hydroxyl), 0.95 Å (benzene), and with $U_{iso}(H) = 1.2\ U_{eq}(C)$ for H atoms on phenolic hydroxyl and benzene.

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cl1	0.24538 (13)	0.73733 (6)	0.50866 (9)	0.0438 (3)
O1	0.2004 (3)	0.30936 (16)	0.3632 (2)	0.0393 (5)
H1	0.173519	0.341051	0.286672	0.059*
N1	0.1417 (3)	0.47566 (19)	0.1863 (2)	0.0308 (5)
C1	0.2225 (3)	0.5104 (2)	0.4306 (3)	0.0270 (6)
C2	0.2387 (4)	0.3907 (2)	0.4641 (3)	0.0313 (6)
C3	0.2967 (4)	0.3548 (3)	0.6029 (3)	0.0373 (7)
H3	0.307500	0.276026	0.624193	0.045*
C4	0.3378 (4)	0.4354 (3)	0.7083 (3)	0.0401 (7)
H4	0.378407	0.410409	0.800626	0.048*
C5	0.3202 (4)	0.5531 (3)	0.6799 (3)	0.0380 (7)
H5	0.345733	0.607048	0.752092	0.046*
C6	0.2644 (4)	0.5887 (2)	0.5435 (3)	0.0309 (6)
C7	0.1744 (4)	0.5486 (2)	0.2867 (3)	0.0290 (6)
H7	0.167144	0.627733	0.267050	0.035*
C8	0.1024 (4)	0.5202 (2)	0.0449 (3)	0.0321 (6)
H8A	0.208052	0.493401	0.005740	0.039*
H8B	0.105723	0.604428	0.047011	0.039*

Comment

Schiff bases are the condensation products of primary amines and carbonyl compounds and structurally characterized by carbon-nitrogen double bond group [5–8]. Owing to the special pharmacological and physiological properties, this class of compounds has been developed as a research focus for many fields such as magnetism, catalysis, electrochemistry, chemical sensor and supramolecular chemistry etc. [9–12]. As a part of our current research interest in halogenated Schiff-base compounds, in this work, we report a new hydrazone-based compound.

The centrosymmetric title molecule is shown in the figure. There is an intramolecular hydrogen bond [O(1)–H(1)···N(1)] in the asymmetric unit, which forms a S(6) ring plane between the phenolic oxygen atom and the diamine nitrogen atom. The S(6) ring is parallel to the aryl moiety. The whole molecule exhibits a centrosymmetric "Z-type" geometry. Further analysis revealed that intermolecular C–H···π hydrogen bonds and aromatic face-to-face π–π interactions play important roles for the crystal packing. [Interactions: C(7)–H(7)···Cg(1) with distance = 3.332(2) Å; Cg1···Cg1′ (′ = -x, 1-y, 1-z): = 3.734(2) and Cg1···Cg1″ (″ = 1-x, 1-y, 1-z): 3.753(2) Å [Cg1 is the centroid of the C1–C6 ring]. All bond lengths and angles are in the expected ranges [13].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: Fund for Yunnan Fundamental Research Projects (Grant No. 2019FH001(-091)).
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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