

2,2'-[Hexane-1,6-diylbis(nitrilomethylidyne)]diphenol

Iran Sheikhshoae^{a*} and
Mahboubeh A. Sharif^b^aDepartment of Chemistry, Faculty of Science, Shahid-bahonar University of Kerman, Kerman, Iran, and ^bDepartment of Chemistry, Islamic Azad University, Qom branch, Qom, Iran

Correspondence e-mail: i_shoae@yahoo.com

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Key indicators

Single-crystal X-ray study

T = 120 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.053

wR factor = 0.110

Data-to-parameter ratio = 18.9

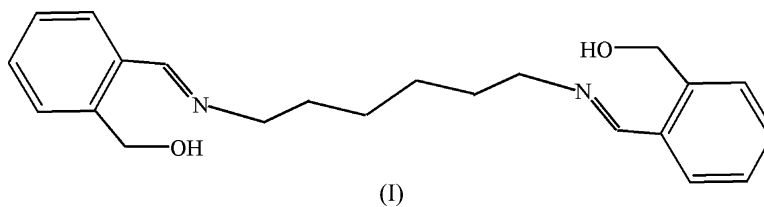
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2$, is a new potential tetradentate N_2O_2 Schiff base ligand which has been synthesized by the condensation reaction between hexane-1,6-diamine and salicylaldehyde, and characterized by common spectroscopic methods (FT-IR, ^1H NMR and ^{13}C NMR) and CHN elemental analyses. The molecule possesses crystallographically imposed C_i symmetry. Two intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds stabilize the molecular conformation.

Comment

Over the past 25 years, extensive research has surrounded the synthesis and use of Schiff base compounds (Schiff, 1864) in inorganic chemistry, as they have important medicinal and pharmaceutical applications. These compounds show biological activities including antibacterial (Karia & Parsania, 1999), antifungal (Singh & Dash, 1988), anticancer (Desai *et al.*, 2001) and herbicidal activities (Samadhiya & Halve, 2001). Furthermore, Schiff bases are utilized as starting materials in the synthesis of compounds of industrial (Aydogan *et al.*, 2001) and biological interest such as β -lactams (Taggi *et al.*, 2002). Schiff bases are also becoming increasingly important in the dye and plastic industries as well as for liquid-crystal technology and mechanistic investigation of drugs used in pharmacology, biochemistry and physiology (Casaszar *et al.*, 1985).

We have recently reported the successful use of some Schiff base compounds in the construction of polyvinyl chloride powder (PVC)-based membrane selective sensors for Co^{2+} , Ag^+ , Fe^{3+} and Hg^{2+} (Mashhadizadeh & Sheikhshoae, 2003*a,b*; Shamspur *et al.*, 2003, 2005). Studies on the properties of Schiff bases as NLO materials have been also reported (Jalali-Heravi *et al.*, 2000; Sheikhshoae & Mashhadizadeh, 2003; Sheikhshoae *et al.*, 2004, 2006). In view of the importance of this class of compounds, we have undertaken the synthesis of the title compound, (I), which represents a new potential tetradentate Schiff base ligand, and report here its crystal structure. The biological and analytical uses of this compound are under study.



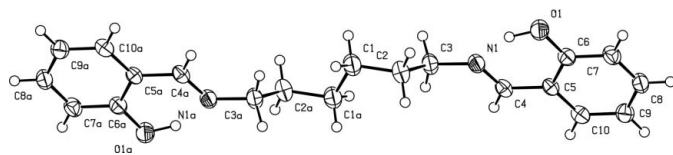


Figure 1

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. Atoms labelled with the suffix a are related by $(-x, -y, 1 - z)$.

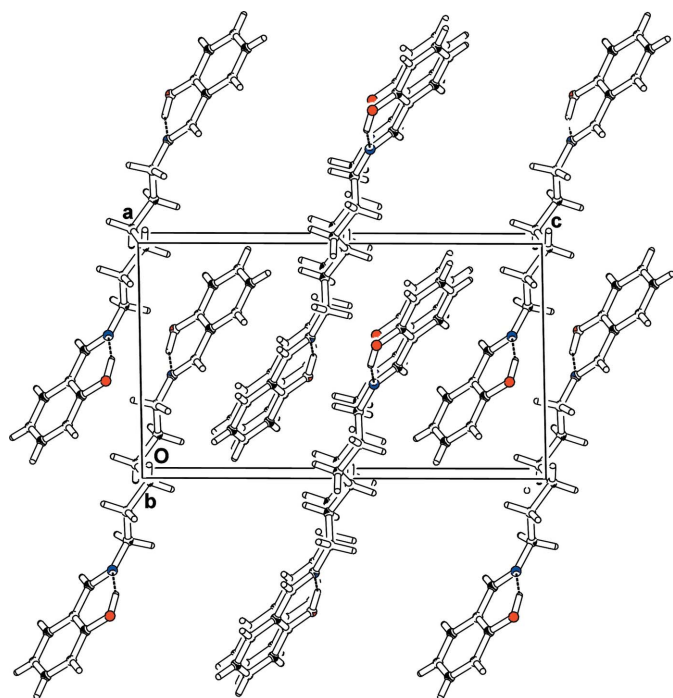


Figure 2

Crystal packing of (I) viewed along the b axis. Intramolecular hydrogen bonds are shown as dashed lines.

expected for this type of compound (Allen *et al.*, 1987). The molecular conformation is stabilized by two intramolecular O—H...N hydrogen bonds (Table 1). The crystal packing (Fig. 2) is mainly determined by van der Waals interactions.

Experimental

Salicylaldehyde and hexane-1,6-diamine were of reagent grade (Merck) and were used without further purification. The solvents methanol, CHCl_3 and ethyl acetate were dried before use by standard methods. A mixture of salicylaldehyde (0.002 mol) in methanol (10 ml) and hexane-1,6-diamine (0.001 mol) in methanol (10 ml) was refluxed for 1.5 h. The colour of the reaction mixture changed to yellow. The precipitate was collected and washed with a small amount of cold methanol. Yellow crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate/chloroform (50:50 *v/v*) solution (yield 72%; m.p. 351 K). Analysis calculated for $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2$: C 74.074, H 7.407, N 8.641%; found: C 74.02, H 7.11, N 8.34%. The structure of the Schiff base ligand was also confirmed by ^1H NMR, ^{13}C NMR, UV-vis and FT-IR spectroscopy. A comparison of the FT-IR spectra of salicylaldehyde and (I) reveals that the absorption band of the CO group at 1669 cm^{-1} is

absent in (I) and a new absorption band at 1624 cm^{-1} due to the C=N group appeared upon condensation.

Crystal data

$\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2$	$Z = 2$
$M_r = 324.41$	$D_x = 1.238\text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.3654(12)\text{ \AA}$	$\mu = 0.08\text{ mm}^{-1}$
$b = 5.7915(7)\text{ \AA}$	$T = 120(2)\text{ K}$
$c = 16.042(2)\text{ \AA}$	Block, yellow
$\beta = 91.140(3)^\circ$	$0.55 \times 0.40 \times 0.25\text{ mm}$
$V = 869.95(19)\text{ \AA}^3$	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2063 independent reflections
φ and ω scans	1433 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.056$
5880 measured reflections	$\theta_{\text{max}} = 28.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0012P)^2 + 0.515P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.27\text{ e \AA}^{-3}$
2063 reflections	$\Delta\rho_{\text{min}} = -0.23\text{ e \AA}^{-3}$
109 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O1—H1O...N1	0.98	1.68	2.5634 (16)	147

The H atom of the hydroxyl group was located in a difference Fourier synthesis, while the H atoms attached to C atoms were placed in calculated positions. All H atoms were refined in the riding-model approximation with O—H = 0.98 \AA , C—H = 0.93 \AA (aromatic and methine H) and 0.97 \AA (methylene H), and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{O})$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1998); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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