

*Full Length Research Paper*

# Crystal structure of 2-[(2-(2-Hydroxyethylamino) ethylimino) methyl] phenol at 130K

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**In this research, the 2-[(2-(2-hydroxyethylamino) ethylimino) methyl] phenol (H<sub>2</sub>L) has been prepared with high yield via condensation of salicylaldehyde and 2-hydroxyethylamine. Crystal structure of H<sub>2</sub>L is determined at 130K. In the crystal structure of H<sub>2</sub>L, it is observed that there are two independent H<sub>2</sub>L-units. X-ray crystal structural analysis revealed that the H<sub>2</sub>L belonged to the orthorhombic space group of *P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>* with cell parameters; a = 7.0205(5), b = 14.1909(9), c = 21.7147(14) Å and β=90.00°.**

**Key words:** Crystal structure, imine Schiff bases, high yield.

## INTRODUCTION

Ligands such as diethylenetriamine, triethylenetetramine, tetraethylenepentaamine and *N*-2-hydroxyethyl-ethylenediamine give condensation reactions with salicylaldehyde and form polydentate ligands. These polydentate ligands are suitable for several types of coordination geometry (Kenar et al., 2001).

The preparative accessibility, diversity and structural variability make Schiff bases very attractive and have achieved recognition for their role in biological systems such as mutagenesis and carcinogenesis (Casella et al., 1982). In addition to the biochemical research, Schiff bases are also being used as analytical reagents (Shanthi et al., 1987). Schiff bases offer a versatile and flexible series of ligands capable of bind with transition, non-transition, lanthanide and actinide metal ions to give complexes with suitable properties for theoretical studies and/or practical applications (Casella and Gulloti, 1983).

The first complex of this compound is known as vanadium (IV) metal (Hakimi et al., 2011a); other complexes are formed with transition metal ions, such as V(V), Fe(III), Co(III), Cd(II) and Mn(III) (Spek, 2003).

Recently, we carried out a study on coordination chemistry of nitrogen donor groups of triazoles, such as 4-amino-5-methyl-1,2,4-triazol-3(2H)-thione (HAMTT), 3,4-diamino-5-methyl-4H-1,2,4-triazole (DAMT) and Schiff

bases using different ions e.g. Pb(II), Cr(III), V(IV) and Cd(II) (Hakimi et al., 2011b).

In this investigation, we reported the crystal structure of 2-[(2-(2-hydroxyethylamino) ethylimino) methyl] phenol at low temperature (130K).

## EXPERIMENTAL

The compound of H<sub>2</sub>L was synthesized as described in previous publication with latter changes and high yields (Hakimi et al., 2012a). Briefly, 10 ml solution of 2-hydroxyethylamine (0.20 g, 2 mmol) was added to 10 ml solution of salicylaldehyde (0.24 g, 2 mmol) in MeOH. The solution was heated by refluxing for 2 h. After roto-evaporization, recrystallization was done by dichloromethane. The as-prepared yellow crystals were collected for X-ray analysis. Yield: 0.39 g (95%); m.p: 93°C.

### Single-crystal structure determination

The data collection for 2-[(2-(2-hydroxyethylamino) ethylimino) methyl] phenol was carried out by Oxford Diffraction Xcalibur Gemini Ultra four-circle kappa geometry diffractometer with Eos CCD detector, using a graphite monochromated Mo-Kα (λ = 0.71073 Å) radiation, and by applying the CrysAlisPro Software system, Version 171.33.53 at 130K. Data reduction was done by the same program (Hakimi et al., 2012b).

The X-ray diffraction data have been corrected for Lorentz-polarization factor and scaled for absorption effects by multi-scan. The structure was solved by direct methods implemented in SHELXS-97. Refinement procedure by full-matrix least-squares methods based on *F*<sup>2</sup> values against all reflections has been

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**Table 1.** Crystal data and details of the refinement of the crystal structure of H<sub>2</sub>L.

Empirical formula	C <sub>11</sub> H <sub>16</sub> N <sub>2</sub> O <sub>2</sub>
Formula weight	208.26
Temperature (K)	130.0
Crystal system	Orthorhombic
Space group	<i>P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub></i>
Unit cell dimensions (Å, °)	a = 7.0205(5) b = 14.1909(9) c = 21.7147(14) β = 90.00
Volume (Å <sup>3</sup> )	2163.4(2)
Z	8
Density (calculated) (mgm <sup>-3</sup> )	1.279
Absorption coefficient (mm <sup>-1</sup> )	0.09
<i>F</i> (000)	896
Crystal size (mm <sup>3</sup> )	0.23 × 0.14 × 0.05
Theta range for data collection	29.3 to 3.0
Index range	-8 ≤ h ≤ 9, -19 ≤ k ≤ 11, -29 ≤ l ≤ 13
Reflections collection	8314
Independent reflections	4641 [ <i>R</i> <sub>(int)</sub> = 0.030]
Absorption correction	Multi Scan
Refinement method	Full-matrix least-squares on <i>F</i> <sup>2</sup>
Data/restraints/parameters	4641/ 0/ 282
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.025
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.048, <i>wR</i> <sub>2</sub> = 0.116
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.0614, <i>wR</i> <sub>2</sub> = 0.1216
Largest diff. peak and hole (e Å <sup>-3</sup> )	0.40/ -0.41

performed by SHELXL-97, including anisotropic displacement parameters for all non-hydrogen atoms.

Calculations concerning the molecular geometry, the choice and verification of the space group, the analysis of hydrogen bonds were performed by PLATON. The molecular graphics were done with ORTEP-3 (Patil et al., 2010). Crystal data and refinement details for H<sub>2</sub>L are as shown in Table 1. Selected bond distances and angles are listed in Table 2.

## RESULTS AND DISCUSSION

The Ortep-3 diagram of 2-((2-(2-hydroxyethylamino) ethylimino) methyl) phenol is as shown in Figure 1. First report of the preparation H<sub>2</sub>L was proposed in 1988.

The yellow crystal of H<sub>2</sub>L was obtained from the reaction between salicylaldehyde and 2-hydroxyethylamine. The compound may bind tetradentate or tridentate to a single metal ion. For most metals, tridentate coordination is practicable, but in state dimer, tetradentate coordination is shown to be practicable for most metals. In state tridentate coordination, OH alcoholic is not bound to metal ion. However, chelation may occur and it involves two adjacent rings (forming

five-membered and six-membered rings).

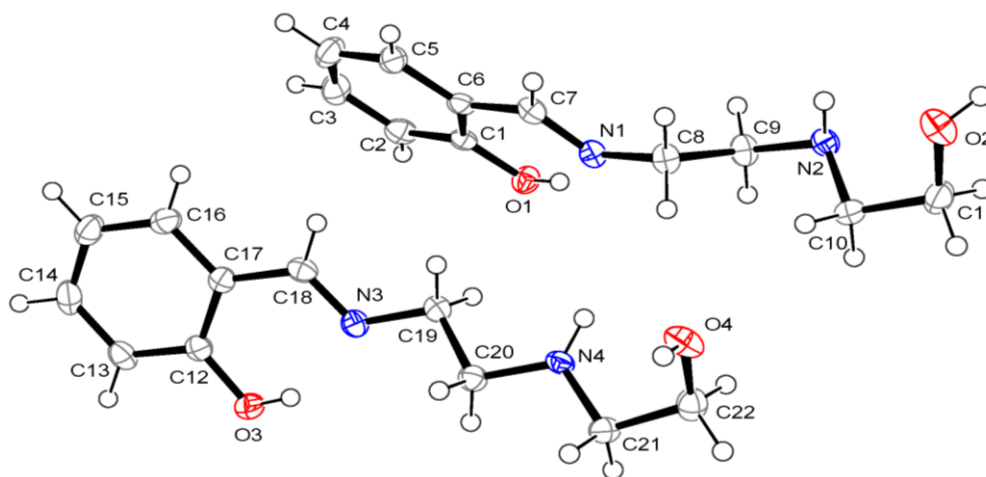
From the literature reports, it is observed that salicylidene imines form keto and enol tautomers, both in solution and the solid state. In the crystal structure, both conformers correspond to the enol form. This is evidenced by the presence of an intramolecular O–H...N hydrogen bond formed between the phenolic OH group and the imine function, and a comparative analysis of the bond lengths within the salicylidene fragment (Rabia et al., 2004).

According to the crystal structural data of H<sub>2</sub>L, two independent H<sub>2</sub>L-units are observed which are connected together via medium strong NH...O and N...OH hydrogen bondings (N1...O1–H1: 2.564 and H4–N4...O2: 2.839 and H<sub>4</sub>–O<sub>4</sub>...N2: 2.798 Å) (Figure 2). The crystal structure of H<sub>2</sub>L has been reported by others, but the quality of our X-ray results is significantly better.

In the compound of H<sub>2</sub>L, bonding distances are N1–C7 (1.274 Å) and N–C8 (1.460 Å), on the other hand, bonding distances N2–C9 and N2–C10 are 1.464 and 1.468 Å, respectively. Considerate to this point which bonding distances N2–C9, N2–C10 and N1–C8 are greater than the bonding distances N1–C7, this implies

**Table 2.** Selected bond distances (Å) and angles (°) for H<sub>2</sub>L.

Bond distances			
N(1)-C(7)	1.274(3)	N(3)-C(18)	1.279(3)
N(1)-C(8)	1.460(3)	N(3)-C(19)	1.458(3)
N(2)-C(9)	1.464(3)	N(4)-C(20)	1.456(3)
N(2)-C(10)	1.468(3)	N(4)-C(21)	1.463(3)
C(6)-C(7)	1.466(3)	C(17)-C(18)	1.451(3)
C(1)-O(1)	1.346(2)	C(12)-O(3)	1.354(2)
C(11)-O(2)	1.417(3)	C(22)-O(4)	1.419(3)
Bond angles			
C(7)-N(1)-C(8)	119.21(19)	C(18)-N(3)-C(19)	118.63(19)
C(6)-C(7)-N(1)	121.1(2)	C(17)-C(18)-N(3)	121.7(2)
C(9)-N(2)-C(10)	114.46(18)	C(20)-N(4)-C(21)	113.65(16)
O(1)-C(1)-C(6)	121.43(19)	O(3)-C(12)-C(17)	121.41(19)
H(1)-O(1)-C(1)	109.5	H(3)-O(3)-C(12)	109.5
H(2)-O(2)-C(11)	109.5	H(4)-O(4)-C(22)	115(3)

**Figure 1.** Molecular structure of H<sub>2</sub>L (50% probability for the ellipsoids).

that bonding between nitrogen 1 and carbon 7 is doublet bond, which confirms the formation of imine bonding.

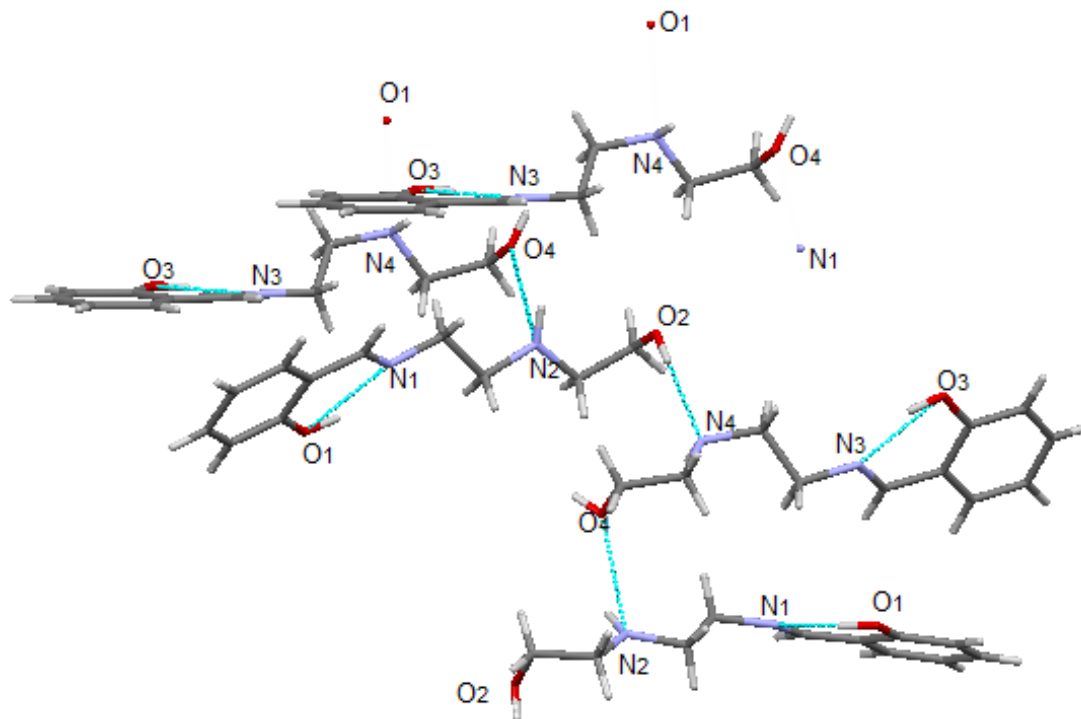
Bond angles are C7–N1–C8 (119.21), C5–C6–C7 (119.9) and also N1–C8–C9 (109.38), N2–C10–C11 (109.83); since bonding between carbon 5 and carbon 6 of aromatic ring is doublet bonding, and on the other hand, bonding between nitrogen 2 and carbon 1 is single bonding, it can be concluded from the stated angles that bonding between nitrogen 1 and carbon 7 is doublet bonding and is from the imine type.

In the compound of H<sub>2</sub>L, any molecule gives three hydrogen bonds. One hydrogen bond is between imine nitrogen atom (N1) and phenolic oxygen atom (O1) of self-molecule and second hydrogen bonding is between its amine nitrogen atom (N2) with alcoholic oxygen atom (O4) of other molecule, the third hydrogen bonding is

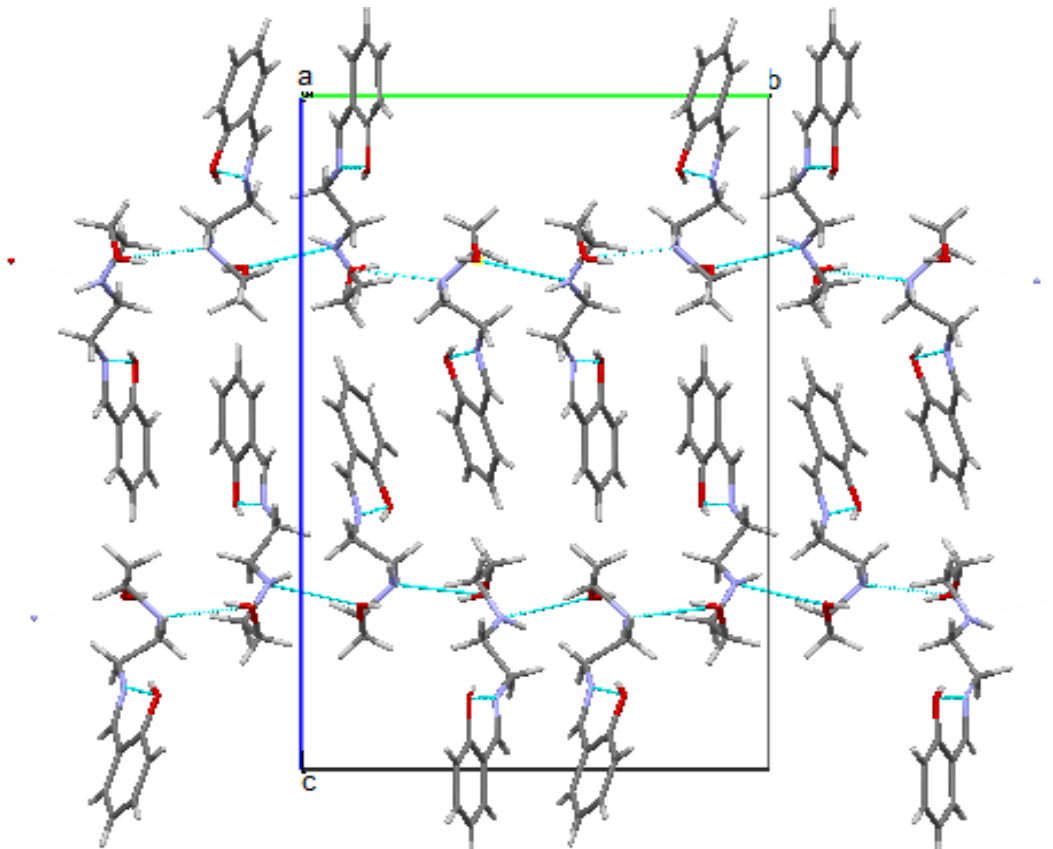
formed between alcoholic oxygen atom (O2) and amine nitrogen atom (N4) of other molecule (Figure 2). In crystal lattice, molecules are bonded together by hydrogen bondings of NH<sup>+</sup>⋯O between molecule and N<sup>+</sup>⋯OH inter molecule, so formed polymer chain which is shown in Figure 3.

## Conclusion

A ONNO Schiff base (H<sub>2</sub>L) has been prepared via condensation of salicylaldehyde and 2-hydroxy-ethylamine with high yield. The compound was used as a tetra and tridentate ligand to react with transition metal ions. The compound gave three hydrogen bonds: one intra molecular hydrogen bond and two inter-molecular



**Figure 2.** Hydrogen bond between molecules of  $H_2L$ .



**Figure 3.** A view of formation chain in direction a axis by hydrogen bonds of  $H_2L$ .

hydrogen bonds. The compound gave resonance between aromatic ring and imini group.

## REFERENCES

- Casella L, Gulloti M, Pacchioni G (1982). Coordination Modes of Histidine. 3. Stereochemistry of Copper(II) Complexes Related to Pyridoxal Catalysis. *J. Am. Chem. Soc.*, 104: 2386-2396.
- Casella L, Gulloti M (1983). Conformation of Pyridoxal Schiff Bases of Amino Acids. A Circular Dichroism Study. *J. Am. Chem. Soc.*, 105(3): 803-809.
- Hakimi M, Vahedi MH, Rezvaninezhad M, Schuh E, Mohr F (2011a). Synthesis and Characterization of Copper (I) Complexes from Triphenylphosphine and Isatin Schiff bases of Semi- and Thiosemicarbazide. *J. Sulfur Chem.*, 32: 55-61.
- Hakimi M, Maeder M, Lawrance GA (2011b). Synthesis, Characterization, and Crystal Structure of a Tricadmium Complex of 3,4-Diamino-5-Methyl-1,2,4-Triazole. *J. Coord. Chem.*, 64: 105-111.
- Hakimi M, Kukovec BM, Normohammadzadeh Z, Schuh E, Mohr F (2012a). Preparation and Crystal Structure of Lead (II) Complex with Propane-1,2-Diamine. *J. Chem. Crystallogr.*, 42: 180-185.
- Hakimi M, Kukovec BM, Minoura M (2012b). 2D *s-d* block Metal Coordination Polymer Containing Potassium, Chromium (III) and Dipicolinate Ions. Preparation and Crystal Structure., *J. Chem. Crystallogr.*, 42: 290-294.
- Kenar A, Arici C, Levent Aksu M, Nawaz Tahir M, Durmus S, Atakol O (2001). Crystal Structure of {Bis[N-(2-hydroxyethylamino)ethylsalicylaldiminato]-iron(III)} Chloride. *Anal. Sci.*, 17(1): 569-570.
- Patil M, Hunoor R, Gudasi K (2010). Transition Metal Complexes of a New Hexadentate Macroacyclic N<sub>2</sub>O<sub>4</sub>-Donor Schiff Base: Inhibitory Activity Against Bacteria and Fungi. *Eur. J. Med. Chem.*, 45: 2981-2986.
- Rabia MKM, Aly GY, Al-Mohanna MA (2004). X-ray Crystal Structure of the N-(2-Hydroxy-1-Napthalidene) Phenylglycine Schiff Base. Synthesis and Characterization of its Transition Metal Complexes. *Synth. React. Inorg. Met Org. Chem.*, 34(8): 1651-1658.
- Shanathi R, Nagaraja KS, Udupa MR (1987). Chromium(III) Complexes of *N*-Salicylideneamino Acid. *Inorganica Chim. Acta*, 133: 211-215.
- Spek AL (2003). Single-crystal Structure Validation with the Program *PLATON*. *J. Appl. Crystallogr.*, 36: 7-13.