International Journal for Multidisciplinary Research (IJFMR)
E-ISSN: 2582-2160 ● Website: www.ijfmr.com ● Email: editor@ijfmr.com

Preparation of a Schiff base compound derived from 2-Mercapto salicylaldehyde & Hydroxamic acid

Dr. Amit Kumar

Assistant Professor
Department of Chemistry
Dr. S.K.S. Women's College, Motihari (E. Champaran)
B.R.A. Bihar University, Muzaffarpur

Abstract:
The Schiff base compound α(2-mercapto Salicylidine) imino aceto hydroxamic acid has been prepared by the condensation of α□-amino aceto hydroxamic acid and 2-mercapto Salicylaldehyde under suitable condition. The production of violet colouration with Fe(III) salts and green colour with Cu (II) salts indicates the presence of hydroxamic acid.

Keywords: Schiff base compound, condensation, formation, filtration, ester, molar-ratio, characterization, molecule, preparation, foul smell.

Introduction:

Schiff bases are important organic compounds which are obtained by the condensation of a carbonyl group and a primary amine group under suitable conditions. A considerable amount of Schiff bases having nitrogen and oxygen atoms as their donor sites have been prepared but at least Schiff bases containing Sulphur atom in addition to nitrogen and oxygen atoms have been prepared due to their foul smell. Therefore, in this paper, we report the Schiff base containing nitrogen, oxygenand sulphur atoms. α (2-mercapto salicylidine) iminio aceto hydroxamic acid has been prepared by the condensation of □-amino aceto hydroxamic acid and 2-mercapto salicylaldehyde under suitable conditions.

Preparation of the ligand and the complexes:

The preparation of Schiff base ligand, □ (2-mercaptoSalicylidine) imino aceto hydroxamic acid has been done in three steps as detailed below:

(A) Preparation of glycine ethyl ester hydrochloride:

Glycine ethyl ester hydrochloride was prepared by fischer-speir 1 by the interaction of glycine and ethyl alcohol at reflux termperature in presence of HCl gas.

$H_2N\text{-}CH_2\text{-}COOH + CH_3\text{-}CH_2\text{-}OH$ (Dry HCl) □ $H_2N\text{-}CH_2\text{-}COOC_2H_5$ HCl+ H_2O

Procedure: A mixture cotaining 0.1 mole (7.5 grams) of glycine (□ -amino aceto carboxylic acid) and

0.1 mole (4.6 grams) of dry ethyl alcohol was gently boiled under reflux on water-bath for two and half an hour at room temperature . Then, a stream of dry HCl gas was passed in it. The excess of HClwas removed by evaporation and the product was recrystallised with ethanol. The melting point of the compound was recorded between 1420-1430C. The compound was furhter analysed and found to contain the following percentage of the element present in the compound. C=34.15%, H=7.18%, N=10.10% and Chlorine = 25.20 % which indicates the molecular formulae - $C_4H_{10}NO_2Cl$ respectively.

B) Preparation of glycine Hydroxamic acid (-amino aceto hydroxamic acid):

Glycine hydroxamic acid also known as -amino acetohydroxamic acid was prepared by Safir and Williams2by the reaction of glycine ethyl ester hydrochloride and hydroxylamine hydrochloride.

H_2N - CH_2 - $COOC_2H_5$ HCl + H_2N - OH. HCl Dry- HCl H_2N - CH_2- CONHOH + CH_3 - CH_2 - OH

Procedure: A mixture containing 0.1 mole (14 grams) of glycine ethyl ester hydrochloride and 0.1 mole (7 grams) of hydroxylamine Hydrocholoride was dissolved in minimum volume of water to produce better yield. Then 0.33 mole (13.2 grams) of NaOH was dissolved in water and the solution was diluted up to 26.4 ml by the addition of water to produce the strength of the solution equal to 12.5

(N) NaOH. into The solution was added thought dropping funnel to the ice-cooled solution containing glycine ethyl ester hydrochloride and hydroxylamine hydrochloride drop wise during one and half an hour with continuous stirring and shaking . The resulting solution was acidified with the addition of

8.5 ml of 12 (N) HCl on cooling in ice-bath. Then the product was obtained as white crystalline solidon chilling . If by erroneously, a little excess of acid was added to the solution, the precipitate dissolves and the product has been obtained by neutralising the solution by the addition of two or three pellets of NaOH into it. The productt was separated by filteration and washed with a small amount of water and then recrystallised with aqueous ethanoliic solutin. The yield was found to be about 4.0 grams (45%) . Then the melting point of the compound was recorded and found to be between 1280-1300 C Then the compound was further analysed and found to contain. C=26.28%, Hydrogen = 6.68% and nitrogen 31.16% respectively. Which correspoond the molecular formula $C_2H_6N_2O_2$

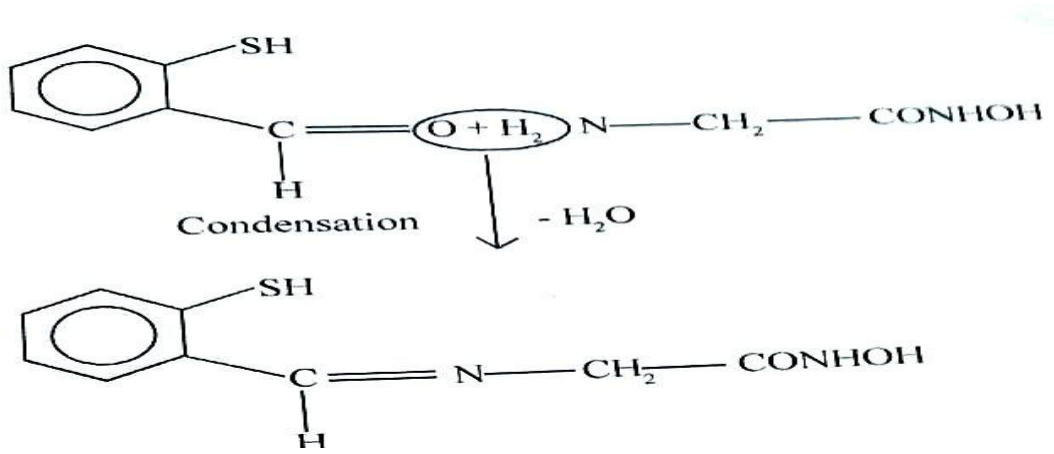
(C) Preparation of Schiff base ligand, (2-mercapto Salicylidine)imino aceto hydroxamic acid.

:The Schiff base ligand (2-mercapto Salicylidine) imino aceto hydroxamic acid has been prepared bythe condensation of 2-mercapto Salicylaldehyde (1 mole) and amino aceto hydroxamic acid (1 mole) at room temperature .

Procedure: A mixture containing 01. mole (9.0 grams) of amino aceto hydroxamic acid and 0.1 mole (14 grams)of 2-mercapto Salicylaldehyde was completely dissolved in minimum volume of ethyl alcohol . The resulting solution was heated under reflux for about three hours on water bath. The resulting solution on crystallisation produced another crop of Schiff base which was then separated by filteration and washed with cold water and finally dried over in an oven. The melting point of the compound was recorded and found to be between 1480-1500C. The compound was further analysed and found to contain.

Table No. 1 Elements	Found%	Calculated %
Carbon	51.10	51.42
Hydrogen	4.80	4.76
Nitrogen	`13.36	13.33
Sulphur	15.10	15.23

Which corresponds the molecular formula $C_9H_{10}N_2O_2S$ The expected chemical reaction taking place during the course of the preparation of the Schiff base ligand (2-mercapto Salicylidine) imino aceto hydroxamic acid by the condensation of -amino aceto hydroxamic acid and 2-mercapto salicylaldehyde taken into 1:1 molar ratio may be presented as below:



 (2-mercapto salicylidine) imino aceto hydroxamic acid Identification of the compound was confirmed by the following tools. 1) Elemental analysis of the compound. 2) Estimation of elements (non-metals) present in the compound. 3) Nuclear Magnetic resonance (NMR) spectroscopy and 4) Infra-red spectroscopy.

Estimation of elements (non-metals) present in the compound.

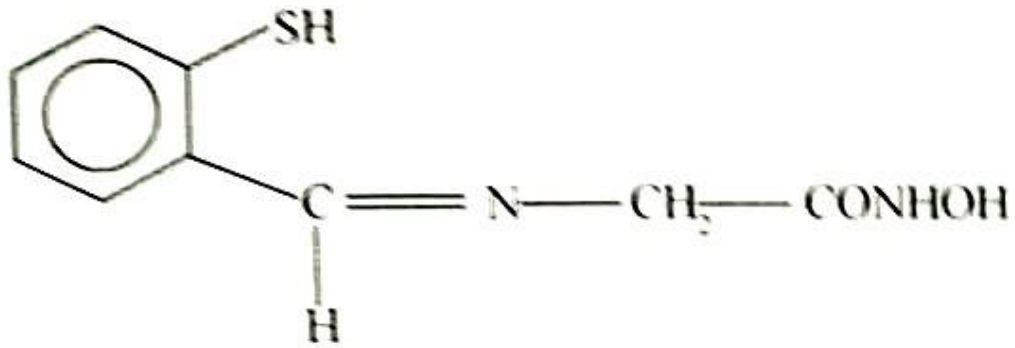
(i) **Hydrogen, Carbon and Nitrogen:**- Hydrogen, Carbon and Nitrogen was estimated by semi-macro-duma's method.

(ii) **Sulphur** - sulphur was estimated gravimetrically by BaSO4

(iii) **1HMNR of the protons present in the compound** (CDCl3) - , 730 -780 (m, 4H, ArH), 580- 590 (m,1H,NH), 5.30-5.40 (m,1H, aldimino), 2.80-2.90 (b, 1H, enolic), 430-460 (b,1H,mercapto), 1.60-170 (b,2H, alkane).

(iv) **Infra-red spectroscopy:** Infra-red spectra of the groups present in the compound:- (1) 3240-3260 cm-1 broad and unsymmetrical due to (combined OH+H- bonded) (2) 2960-2980 cm-1 (Sharp + Weak) due to Ar (C-H) group. (3) 2490-2520 cm-1 (Sharp + Medium) due to (S-H) group. (4) 1640-1660 cm-1 (Strong + Medium) due to Azomethine (>C=N- group. (5) 1470-1490cm-1 (Sharp + Medium) due to (>C=N-OH) group. (6) 1430-1440cm-1 (Sharp + Medium) due to (C-O) group. (7) 1310-1320cm-1 (Sharp + Medium) due to Oximinio (-OH) group. (8) 1030-1050cm-1 (Sharp + Medium) due to (C-S) group. (9) 990-1010cm-1 (Sharp + Medium) due to (N-O) group.

On the basis of the elemental analyis, I.R. spectra and 1HNMR spectroscopy the following structure has been suggested for the Schiff base compound prepared.



 (2-mercapto salicylidine) imino aceto hydroxamic acid

Acknowledgement: I am thankful to the Principal & Department of Chemistry of Dr.S.K.S,College.Women's Motihari. B.R.A.Bihar University Muzaffarpur for providing me available library and laboratory facilties. I am also thankful to faculty members and non-teaching staff of

chemistry department for providing decorous behaviour during the course of the experimental work for this paper.

REFERENCES:

1. H. Schiff- Annal Phys, 150 (1869) 193.
2. H. Lossen- Annal Phys, 150 (1869) 314
3. F.G. Mann and B.C. Saunders- Practiical Organic Chemistry.
4. Vogel, A.E. : Practical, (E.L.B.S. Longman group Ltd., Organic Chemistry 1063 (Reprint) 1971.5. N.D.C. Hodges: Annal Phys, 182, (1876) 182.
6. Handzsch and C.H., Desch, A. annal Phys 323 (1902) 453.
7. John Dyer: Applications of absorption spectroscopy of Organic compounds prentice/Hall of IndiaPrivate Limited, New Delhi, 1974.