

PREPARATION OF SCHIFF BASE COMPOUND, α (O-HYDROXY ACETO PHENONE) IMINO ACETO HYDROXAMIC ACID

Amitabh Kumar₁
Research Scholar.

University Department of Chemistry,
 B.R.A. Bihar University, Muzaffarpur-842001 (BIHAR).

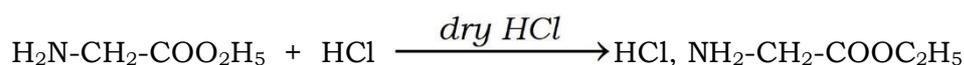
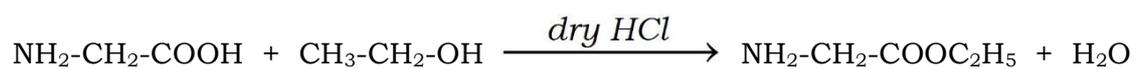
ABSTRACT: I have prepared such a Schiff base which is derivative of hydroxamic acid. I have prepared α (o-hydroxy acetophenone) imino aceto hydroxamic acid by the condensation of α -amino aceto hydroxamic acid and o-hydroxy acetophenone under suitable conditions. This compound being derivative of hydroxamic acid behaves as bi-anionic tridentate.

KEY WORDS: Derivative, base, bi-anionic, tridentate, reflux, condensation, solution, solvent.

INTRODUCTION: A large number of Schiff bases have been prepared by the condensation of a primary amine and a carbonyl group. But least Schiff bases have been prepared which may contain hydroxamic acid in its moiety. Therefore, in this paper, I report the preparation of the Schiff base α (o-hydroxy acetophenone) imino aceto hydroxamic acid which contains hydroxamic acid group in its moiety.

A.PREPARATION OF GLYCINE ETHYL ESTER HYDROCHLORIDE:

Glycine ethyl ester hydrochloride was prepared by Fischer-speir by the interaction of glycine and ethyl alcohol at reflux temperature in presence of HCl



Procedure: A mixture of 0.1 mole of glycine and 0.1 mole of dry ethyl alcohol was gently boiled under reflux on water bath for several hours at room temperature. Then, a stream of dry HCl gas was passed in it. The excess of HCl was removed by evaporation and the product was recrystallized with ethanol. The melting point of the compound was recorded between (142° - 143°). Further, the compound was

analysed and found to contain Carbon = 34.16%, Hydrogen = 7.20%, Nitrogen = 10.15% and Chlorine = 25.20% which correspond the molecular formula



B.Preparation of Glycine Hydroxamic Acid:

The acid was prepared by Safir and Williams. A slight modification was made in this method.



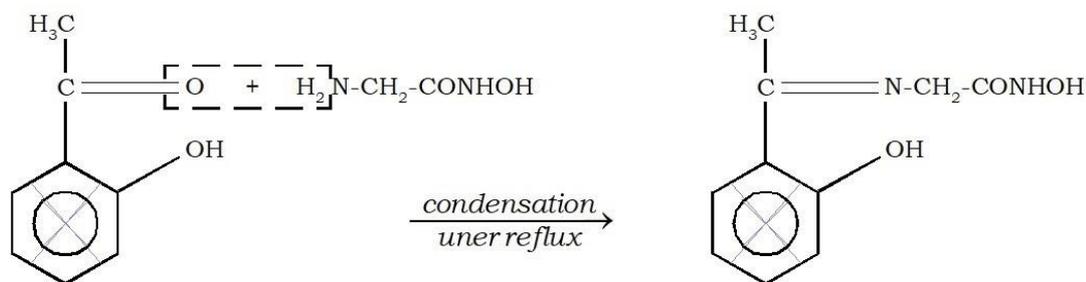
Procedure: 14g (0.1 mole) of glycine ethyl ester hydrochloride and 7g (0.1 mole) of hydroxylamine hydrochloride was dissolved in 11ml of water (Safir and Williams took 17ml of water but with this volume, the precipitation of the product was unsatisfactory because supersaturated solution gives good yield). The solution was cooled in ice bath and to this solution 26.4ml. (0.33 mole) of 12.5(N) NaOH (i.e. 13.2g of NaOH pellet was dissolved in water and the volume of the solution was made to 26.4ml) was added drop-wise during one and half hours with continuous stirring and shaking. The resulting solution was acidified with 8.5ml of 12 (N) HCl on cooling ice. The product crystallized as a white solid after chilling. If by erroneously a little excess of acid is added to the solution, the precipitate dissolves and the product can be obtained by neutralizing the solution by adding two or three pellets of NaOH into it.

It was filtered and washed with a little cold water and recrystallized with aqueous alcohol. Yield (3.5g) (39%) M.P. 142° – 143°c (dec). Jones and Sneed reported M.P. 140°c (dec). The compound was analysed and found to contain Carbon = 26.40%, Hydrogen = 6.72%, Nitrogen = 31.18% which correspond the molecular formula $C_2H_6N_2O_2$.

C.Preparation of α (o-Hydroxy Aceto Phenone) Imino Aceto Hydroxamic Acid,

LH₂:

The ligand α (o-hydroxy acetophenone) imino aceto hydroxamic acid was prepared by the condensation of α -amino aceto hydroxamic acid and o-hydroxy aceto phenome under reflux at room temperature.



Procedure: α -amino aceto hydroxamic acid was dissolved in a minimum volume of dry and cold methyl alcohol and allowed to react with methanolic solution of *o*-hydroxy acetophenone in equimolar proportion under reflux on a water bath for several hours at room temperature. During the course of reflux the solution was shaken well for more than two times. The solution thus obtained on crystallisation produced a coloured solid which was then separated by filtration, washed with acetone and finally dried over KOH in a desiccator.

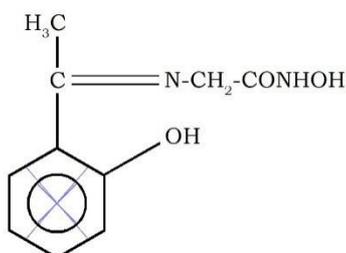
The compound was further analysed and found to contain Carbon = 57.48%, Hydrogen = 5.80%, Nitrogen = 13.52% which correspond the molecular formula $C_{10}H_{12}N_2O_3$.

The identification of this compound has been confirmed by IR and 1H NMR spectroscopy.

I.R. (KBr) : ν , 3260 cm^{-1} (OH + H-bonded), 2960 cm^{-1} (C-H Ar), 1600 cm^{-1} ($>C=N$), 1450 cm^{-1} (N-O, oxime), 1480 cm^{-1} (C-H aliphatic), 1430 cm^{-1} (C-O, phenolic), 1230 cm^{-1} (phenyl ring vibration), 1100 cm^{-1} (N-O, oxime), 800 cm^{-1} (NH + CH), 740 cm^{-1} (benzene ring out of plane), 560 cm^{-1} (M-O) and 445 cm^{-1} (M-N).

1H NMR ($CDCl_3$) : δ , 7.30-8.10 (m, 4H, ArH), 1.60-2.64 (b, 5H, alkane), 5.32 – 6.30 (b, 2H, -OH phenolic), 5.90 (m, 1H, NH).

On the basis of these results of IR and 1H NMR spectra, the following structure of the compound has been assigned:



ACKNOWLEDGEMENT: I am thankful to Dr.Nagendra Prasad, the then Head & Professor, University Department of Chemistry for providing me available library and laboratory facilities. I am also thankful to my guide Dr. S.N. Thakur, Associate Professor, University Department of Chemistry under whose invaluable guidance at every stage of the investigation the thesis could be given this shape. I am also thankful to faculty members and non-teaching staffs of Chemistry Department for providing me cordial relation with me during the course of my whole research work.

REFERENCES:

1. FischerSpeir: *J.O.C.*, 17, 506(1952).
2. S.K. Safir& J.H. Williams: *J.O.C.*, 17, 1398(1952).
3. H. Lossen: *Annal Phys.*, 150(1869)314.
4. Exner, O and Katac: *Coll. Czech. Chem. Commun.*, 28(1963)1656.
5. W. Lossen: *Annal Phys.*, 16(1883)873.
6. H. Schiff: *Annal Phys.*, 150(1869)193.
7. John Dyer: *Applications of Absorption Spectroscopy of Organic Compounds*, Prentice/Hall of India Private Limited, New Delhi, 1974.
8. Akhilendra Singh: *IJRAR*, 6(2019)614-616.
9. B. Shankar, R. Tomaret.al.: *Journal of Chemical and Pharmaceutical Research*, 6(5)2014, 925-930.
10. B.K. Rai et.al.: *Asian J. Chem.*, 21(2009)3708-3712.
11. A.K. Yadav et.al.: *J. Chemtracks*, 18(2016)99-104.
12. Sangeet and Rajneesh Kumar: *J. Chemtracks*, 19(2017)123-126.