Preparation of A Schiff Base Compound, 2(2-hydroxy benzylidine) imino benzo Hydroxamic Acid

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Abstract : 2-amino benzohydroxamic acid has been prepared by the interaction of methyl benzoate and hydroxylamine hydrochloride under suitable conditions. After that, the Schiff base compound 2(2-hydroxy benzylidine) has been prepared by the condensation of 2-amino hydroxamic acid and 2-hydroxy benzaldehyde. This Schiff base contains hydroxamic acid in its moiety. This is the special feature of this Schiff base compound.

Key-words : Solution, Reflux, Condensation, Suction, Crude product, Mixture, Filtrate, Filteration, Compound, Preparation, Distillation, etc.

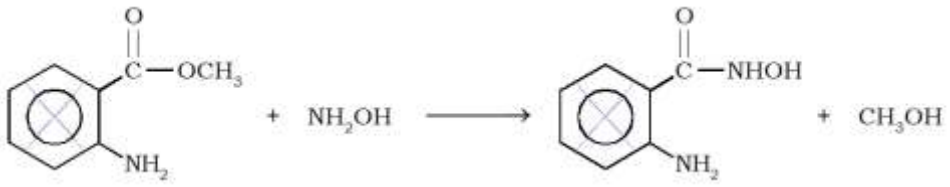
I. INTRODUCTION

A large number of Schiff base compounds have been prepared since 1869, but at least Schiff bases have been prepared containing hydroxamic acid in its moiety. Therefore, in this paper, I report the preparation of Schiff base 2(2-hydroxy benzylidine) imino benzo hydroxamic acid, which contains 2-amino benzohydroxamic acid in its moiety.

II. THE SCHIFF BASE COMPOUND 2(2-HYDROXY BENZYLIDINE) IMINO BENZO HYDROXAMIC ACID HAS BEEN PREPARED INTO TWO STEPS:

(A) Preparation of 2-amino benzo hydroxamic acid:

First of all, A.W. Scott and B.L. Wood prepared an acidic compound by the reaction of hydroxylamine on methyl anthranilate under reflux temperature.



This compound, 2-amino benzohydroxamic acid produced deep violet colouration with the solution of Fe(II) salts and green colour with the solution of Cu(II) salts, which are the characteristic tests to indicate the presence of hydroxamic acid.

This compound was found to be fairly stable upto 140°c and was kept for three months at room temperature without any noticeable decomposition.

Procedure: 48g of sodium hydroxide was completely dissolved in 300ml of water and this solution was cooled under ice-bath. 41.6g of hydroxylamine hydrochloride was gradually added with constant stirring to the solution of sodium hydroxide. After that 45.2g of methyl benzoate (methyl anthranilate) (39ml, sp. Gr.

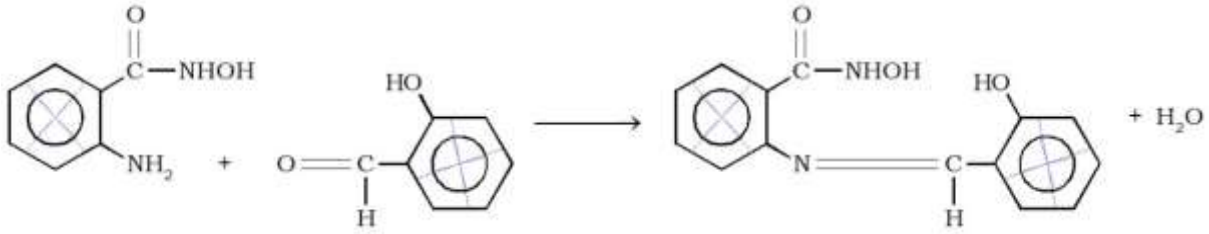
1.16) was gradually added to the resulting solution along with enough alcohol for the proper dilution of the solution. The resulting or while solution was allowed to stand for three days at room temperature. Then, the solution was distilled under the reduced pressure until sodium salt of hydroxamic acid was precipitated leaving about 100ml of mother liquor in the flask. The salt was filtered by suction and washed with ether. The filtrate was then made acidic with *dil.*HCl and then free hydroxamic acid was precipitated. The crude product was recrystallized from ether. It was light brown in colour. The melting point of the compound was recorded and found to be 149°c. The yield of the compound was found to be nearly 65%.

The compound was further analysed and found to contain carbon=55.15%, hydrogen=5.25% and nitrogen=18.45% which corresponds the molecular formula $C_7H_8N_2O_2$.

(B) Preparation of the Schiff base compound 2(2-hydroxy benzylidine) imino benzo hydroxamic acid:

This Schiff base compound has been prepared by the condensation reaction of 2-hydroxy benzaldehyde (salicylaldehyde) and 2-amino benzolydroxamic acid at reflux temperature.

This Schiff base compound has been obtained by the condensation of 2-amino benzohydroxamic acid in aqueous alcoholic solution and 2-hydroxy benzaldehyde under suitable conditions



2(2-hydroxy benzylidine) imino benzo hydroxamic acid

Procedure: 12.4g (0.1 mole) of 2-hydroxy benzaldehyde was completely dissolved in minimum volume of ethyl alcohol and 17.4g (0.1 mole) of 2-amino benzohydroxamic acid was dissolved in glacial acetic acid. Both the solutions were mixed together gradually and the resulting solution was vigorously and constinuously stirred until a brownish yellow precipitate appeared in the solution. This was then refluxed for half an hour.

A clear brownish yellow solution was obtained which on cooling produced brownish crystals of Schiff base. The compound was separated by filteration, washed with cold water, followed by a little alcohol and then recrystallized with methyl alcohol. The compound was found to be soluble in acetone and the melting point of the compound was recorded and found to be 130°c.

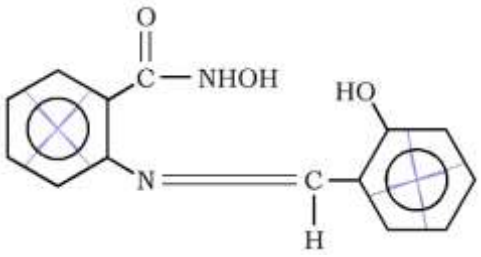
The compound was further analysed and found to contain carbon=65.24%, hydrogen=4.66% and nitrogen=10.95% which corresponds the molecular formula $C_{14}H_{12}N_2O_3$.

The identification of the compound was confirmed by I.R. and ^{1}HNMR spectroscopy.

I.R. (KBr):ν, 1320cm^{-1}(C-N), 1510cm^{-1}(C=C Ar), 1540cm^{-1}(C-O, phenolic), 1840cm^{-1}(C=O), 2740cm^{-1}(C-H), 3410cm^{-1}(NH+OH).

^{1}HNMR ($CDCl_3$):δ, 5.95(S, 3H, NH), 730-810(m, 8H, ArH), 4.30(S, 1H, $-\overset{H}{\underset{|}{C}}=N$), 536-538(b, 2H, OH phenolic).

Thus on the basis of elemental analysis, I.R. and ^{1}HNMR spectroscopy, the Schiff base compound has been assigned the following structure:



2(2-hyroxy benzylidine) iminobenzohydroxamic acid

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