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New Synthetic Pathway for α-Benzil monoxi methio carbo hydrazide Compound

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Abstract- Benzilmonoxi methio carbo hydrazide synthesis is an important intermediate step for many hydrazine organic synthetic reactions. Synthesis of α-benzil monoximethio carbo hydrazide can be done by reaction of α-benzil monoxi me with thiocarbo hydrazide (1,3-Diamino-2-thiourea) and anhydrous sodium acetate in water, methanol (50:50v/v) at 60°C giving 73% yield (based on [α-benzilmonoxime]). However, in this article, we achieve a 93% yield (based on [α-benzil monoxime]) by carrying out the reaction in ethanolic solution (50:50 v/v) at room temperature.

Keywords- carbo hydrazide, ethanolic solution etc.

I. INTRODUCTION

The chemistry and applications of thiocarbo hydrazide in synthetic organic chemistry and biological sciences have recently been reviewed[1]. The applications of this compound include assessment process of the three-dimensional ultra structure examination techniques of inter phase nuclei and tissues; use as fogging agent; use in cool-burning pyrotechnic compounds for dissemination of smoke.

use in chemical warfare and as therapeutic agents; use in performing a highly selective heavy metal ion adsorbent and as complexing agents in solven text raction separation methods[2-6] etc. α-benzyl monoxides mythoi carbo hydrazide is an important class thiocarbo hydrazine derivatives and useful intermediates in many organic synthesis. As such, laboratory synthesis of α-benzil monoxi methio carbo hydrazine by many chemists is unavoidable. Green chemistry practice would then suggest a synthetic pathwaythat produces less solvent waste with very high-quality product yields. Currently,α-benzil monoximethio carbohydrazide can be synthesized using condensation reaction[7]. However, this method gives a 25%product yield based on α-benzil monoxime concentration. Consequently, there is a high waste solvent generation. This research article reports a new pathway that allows an93% high-quality product yield with minimal waste solvent generation. This synthesized compound is here in characterized and their synthetic pathway proposed.

II.METHODOLOGY

1. Chemicals
All the chemical materials were purchased from Lobachemie and used as received without further purification.

2.Synthesis of α-benzil monoximethio carob hydrazide according to Literature[7]:
α-Benzi monoximethio carbo hydrazine was prepared by mixing hot aqueous solution of 20.000 g of thiocarbo hydrazide (0.188 mol) with ethanolic solution of 10.000 g of α-benzilmonoxime (0.044 mol) in presence of sodium acetate (20.000 g), the mixture was refluxed for 7h on a water bath and kept overnight, a colorless solid was obtained. This was filtered and washed by hot water and dried at 100 °C. [The yield of a product was 10.165 g, 73.80% of the theoretical. Melting point is 168 °C].

3. Synthesis of Thiocarbo hydrazide(New Method)

A 500 mL two neck round bottomed flask wasconnected to a reflux apparatus and 0.10 moles ofα-benzil monoxime and 100ml ethanol were added to the flask and stirred using a magnetic stirrer at 60°C and 3mL conc HCl was added. Then dropwise added 0.11 moles of thiocarbohydrazide (in 150mLwater) solution in 45 minutes. The reaction mixture was then refluxed at 90°C for 24 hours. Recovered the methanol from reaction mixture. Finally, the solution was permitted to cool to room temperature and the resultant precipitate,α-benzyl monoximethio carbo hydrazide, was separated by filtration. The precipitate was washed with cold methanol and dried. The crude product was purified by dissolving in methanol at 60°C and cooling to room temperature. The light yellow color compound finally obtained were washed with cold methanol and dried in vacuum. The actual yield was 0.084 moles (84%).

4. Measurements

The data for ^{1}H-NMRspectrum was obtained on a Bruker AV400 NMRspectrometer at the resonance frequency of 400MHz. Fourier transform infrared (FT-IR) spectrum was carried out on a Bruker Vector 22 infrareds pectro meter using KBr pellet method. Electro spray ionization mass spectrum (ESI-MS) were recorded using a Xevo G2 QT ESI-MS. Ultra-violet spectrum was recorded on JASCO V650 spectro photometry.

III.RESULTS AND DISCUSSION

In the laboratory, researchers encounter different challenges that include non-intended results. In this work, synthesis of benzyl monoximethio carbohydrazide[7] in large amounts was desired so as to synthesize various substituted aldehydes. Characterization of synthesized compound obtained in glacial acetic acid and methanol as reaction solvents using^{1}H NMR and FTIR (see **Fig.-1**) show the products to be similar. The FTIR shows thiocarbonyl groups (C=S) with a stretch at1210 cm^{-1}, while primary amines (-NH_2) record two peaks at 3317 and 3217 cm^{-1}. The secondary amines have a peak at 3205 cm^{-1}. The morphology of a powder sample from methanol product before recrystallization shows a crystalline product whereas the sample from water product shows small crystals coagulated together. However, after recrystallization of both samples in water, the morphology was similar.

IV. CONCLUSIONS

In this article, a high yield of α-benzil monoxi methiocarbo hydrazide(84% based on [α-benzil monoxime]) is achieved when the reaction mixture is changed from water to methanol. This method saves on the loss of methanol that is relatively expensive.

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