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Analysis of N-citralbenzenamines by $NaBH_4/B(OH)_3$ system

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Abstract

Citral as α, β- unsaturated carbonyl compound has been reacted with structurally different ahilines with sodium borohydride in the presents of boric acid for the synthesis of their corresponding N-citralbenzenamines with good yields ($75-85^0$) within 5 min in CH_3 CNat room temperature.

Keywords: Analysis, Carbonyl, N-citralbenzenamines, temperature

Introduction

Reduction of α, β-unsaturated carbonyl compounds can follow two pathways: addition to carbonyl group (1,2-reduction) to give allylic products. $NaBH_4$ (common reducing agent) uses for the 2-dreduction of conjugated carbonyl compounds under different reducing system [1-7]. On the other hands, in the synthetic project we needed some N-citralbenzenamines. Also, recently was reported a convenient system for direct reductive amination of aldehydes by $NaBH_4/B(OH)_3$ system.

Therefore synthesis of N-citralbenzenamines with this hope, the deductive amination of citralas an α, β- unsaturated carbonyl compound follow addition to carbonyl group (1,2-addition) to give the corresponding α,β-unsaturated N-benzenamines.

Material and Method

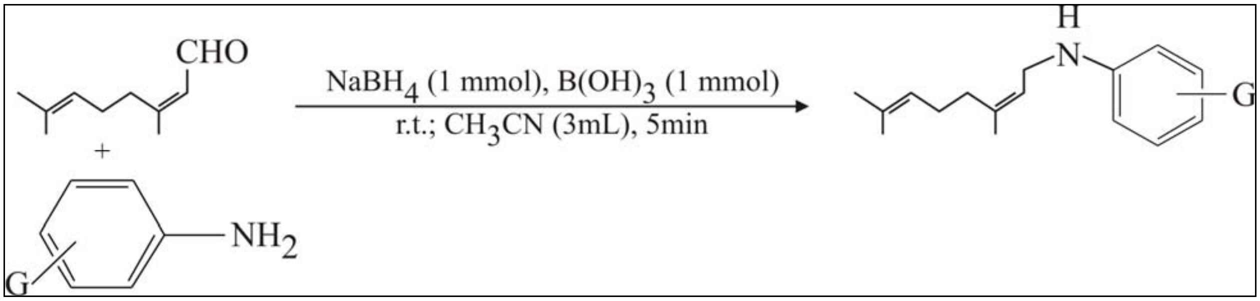
The products were characterized by their ^{1}H NMR (400MHz Bruker) or IR (Perkin Elmer FT-IR RXI) and comparison with authentic samples (melting or boiling points). TLC was applied for the purity determination of substrates, products and reaction monitoring over silica gel 60 F_{254} aluminum sheet.

Reductive amination of citral and aniline with $NaBH_4/Al(OH)_3$, A typical procedure

In a round- bottomed flask (10mL) equipped with a magnetic stirrer, a solution of citral (0.152g, 1 mmol) aniline (0.093g, 1 mmol) and Al $(OH)_3$ (0.078,1 mmol) in CH_3CH (3mL) was prepared. The resulting mixture was stirred for 5 min at room temperature. Then the $NaBH_4$ (0.036g. 1mmol) was added to the reaction mixture and stirred at room temperature. TLC monitored the progress of the reaction (eluent; CCI_4/Ether:5/2). The reaction was filtered after completion within 5 min. Evaporation of the solvent and short column chromotography of the resulting crude material over silica gel (eluent; CCl_4/Ether: 5/2) afforded the N-(3, 7-dimethylocta-2, 6-dienyl) benzenamine (0.195g, 85%)

Results and Discussion

We have done the reductive amination reactions based on the optimized reaction that has been reported in the literature. These reactions were carried out with molar ratio of citral (1 mmol), anilines (1 mmol), B $(OH)_3$ (1 mmol) and $NaBH_4$ (1 mmol) in CH_3CH (3 mL) at room temperature. The reactions were completed within 5 min with 75-85% yields of product as shown in scheme 1.



G:H (85%). 4–Br (80%). 4–Me (80%). 2–MeO (75%), 4–MeO (80%). 4–NO_2(75%)

Scheme 1

In this reactions N (3, 7-dimethylocta-2,6-dienyl) benzenamines (N-citralbenzenamines) as major products have been produced more than 75%, butgeraniol and unreacted anlinesas by products less than 15%.

Conclusion

In this investigation, we have shown that the combination reducing system of $NaBH_4$/B $(OH)_3$ in CH_3CN can be used for reductive amination of citral with a variety of anilines to their corresponding N-citralbenzenaminesin good yields (75-80%) within 5 min at room temperature. Reduction reactions were carried out with 1 molar equivalents of $NaBH_4$ in the presence of 1 molar amounts of $B(OH)_3$.

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