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Polyvinyl alcohol immobilized N-ethylsulfamic acid(PVA-NHSO₃H): Mild and Efficient Catalyst for Acetylation of Phenols, Alcohols and Amines under Solvent-Free Conditions

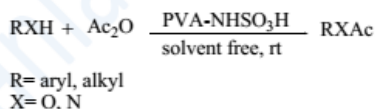
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Background: Acylation of hydroxyl groups is one of the most frequently used transformations in organic synthesis. Among the various protecting groups used for the hydroxyl function, acetyl is the most common group in view of its easy introduction, being stable to the acidic reaction conditions, and also easily removable by mild alkaline hydrolysis [1]. The poor nucleophilic properties of hydroxy compounds necessitate activation. Various activators employed for this purpose have been used for the acetylation of alcohols.[2] Although there is currently a number of methods available, they have one or more disadvantages, such as long reaction times, the use of halogenated solvents, and the use of hazardous materials. Some methods require the use of an excess of acylating agent. Thus, the development of new catalytic methods is highly desirable. In this research, we wish to report PVA-NHSO₃H as an efficient and very mild catalyst for acylation of phenols, alcohols and amines using acetic anhydride as acetylating agent.

Methods: General Procedure: PVA-NHSO₃H (0.018 gr) was added to a mixture of phenols/ alcohols/ or amines (1.0 mmol) and Ac₂O (1.0 mmol). The reaction mixture was stirred at r.t. for the appropriate amount of time. After completion of the reaction (by TLC), the reaction mixture was diluted with saturated NaHCO₃ (10 mL) and extracted with EtOAc (3 × 10 mL). Concentration of the combined organic layer under vacuum gave a crude mass, which was purified by thin layer chromatography (silica gel); which gave the corresponding acetylated product.

Results: During the course of our studies, we report an alternative method for the acylation of phenols, alcohols and amines based on treatment with acetic anhydride in the presence of PVA-NHSO₃H without the necessity for use of solvents (Scheme 1).



Scheme 1

Conclusion: We have reported a catalytic method for acetylation of phenols, alcohols and amines using PVA-NHSO₃H as an efficient, reusable heterogeneous catalyst. High yields, working at room temperature and short reaction time with an easy workup procedure are advantages of this reaction.

Keywords: Phenols; Alcohol; Amines; Acetic anhydride; Polyvinyl alcohol immobilized N-ethylsulfamic acid(PVA-NHSO₃H).

References

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