

Facile Green Transesterification: An Undergraduate Laboratory Approach to Synthesize 3,5-dinitrobenzoates

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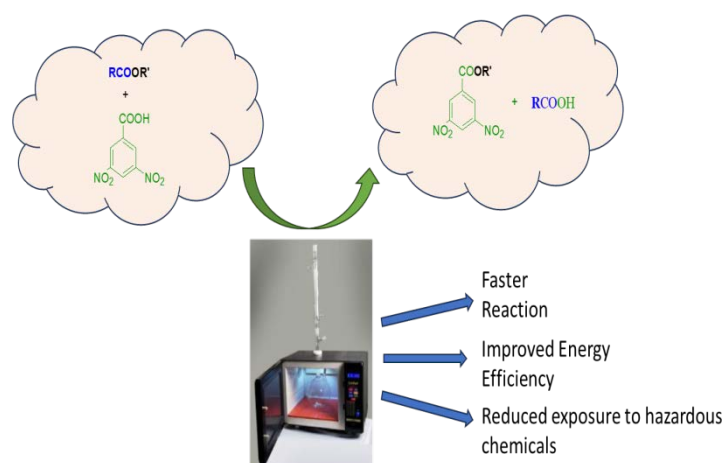
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Abstract Qualitative organic analysis is a key element of chemistry students' laboratory training worldwide, equipping them with essential skills for careers in research and development. However, traditional methods used for such analysis often do not align with the principles of green chemistry. As part of our ongoing efforts to incorporate sustainable practices into undergraduate and postgraduate chemistry labs, we report a green methodology for preparing 3,5-dinitrobenzoates of alcohol part of ester, used in the characterization of unknown esters. In the present method, the ester is directly treated with the 3,5-dinitrobenzoic acid in presence of a few drops of conc. sulphuric acid in a microwave oven at ~70°C for 5 minutes.



Keywords: qualitative organic analysis, microwave oven, green chemistry, 3,5-dinitro benzoates

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1. Introduction

Qualitative organic analysis is a fundamental aspect of the training provided to undergraduate and postgraduate students at universities in India and globally. The process involves the identification of an unknown compound through preliminary and functional group tests, followed by confirmation via the preparation of a suitable derivative. [1,2] The preparation of a suitable derivative not only aids in compound identification but teaches students essential skills in organic synthesis, which are valuable in their career as organic chemists. Certain functional group

derivatives, such as those of carboxylic acids (S-benzyl iso thiuronium salt and anhydrides), carbonyl compounds (2,4-DNP, semicarbazones) are easy to prepare, while, derivatives of other functional groups, such as alcohols (3,5-dinitro or p-nitro benzoates) and esters (3,5-dinitro benzoate of alcohol moiety) are obtained with difficulty.

Chemists continue to make efforts to modify and enhance organic qualitative analysis curriculum more sustainable, by incorporating the 12 principles of green chemistry. [3,4] At our institution, we have successfully revised the procedures for preliminary and functional group testing via spot tests. [5,6,7] In addition, the derivatization of alcohols and esters using ionic liquids and microwaves have been reported. [8] These changes

aim to maintain the quality of student training while addressing the issues of excessive chemical and energy consumption, as well as waste generation, commonly associated with traditional methods in the undergraduate laboratories.

In this recent work we have focused on the functional group esters, as it is an important component in many multistep organic syntheses as well as part of many naturally occurring fragrances of flowers and fruits e.g. bananas have isopentyl acetate, wintergreen contains methyl salicylate, and pineapples contain ethyl butyrate. Esters are important component in several cosmetics, perfumes and synthetic flavours.

2. Present Work

Literature survey indicates that not much work has been done to modify the existing procedures towards the derivative preparation of esters. Esters are usually identified by analysing their alcohol and acid components, which are converted into suitable derivatives. Alkaline hydrolysis of esters offers a convenient method for identifying the carboxylic acid component, while the alcohol component is typically identified by converting it into its 3,5-dinitrobenzoate derivative through transesterification. However, transesterification is time consuming, and students prepare it with great difficulty. Transesterification of esters to the 3,5-dinitrobenzoates is conventionally carried out by heating the ester and 3,5-dinitro benzoic acid with continuous stirring over a sand bath for 15-20 minutes. This method is time consuming and often the reactants charr before the products are formed.

In the modified method, the ester is treated with the 3,5-dinitro benzoic acid in presence of a few drops of conc. sulphuric acid in a microwave oven at 70°C for 5 minutes. Microwave-assisted reactions offer several benefits to undergraduate students, particularly in enhancing their laboratory experience and understanding of green chemistry. [9] Microwave heating significantly reduces reaction time, thus allowing students to complete experiments within a single lab session, enhancing efficiency and enabling more experiments in limited time.

3. Experimental Procedure:

Transesterification of Esters Using 3,5-Dinitrobenzoic Acid

Conventional Method: A typical procedure involves heating the ester and 3,5-dinitro benzoic acid in presence of a few drops of conc. sulphuric acid with continuous stirring over a sand bath for 15-20 minutes.

Microwave assisted method: The green method of transesterification of esters is carried out in a microwave oven by heating the ester and 3,5-dinitro benzoic acid in presence of a few drops of conc. sulphuric acid at 70°C for 5 minutes.

4. Conclusion

We have successfully developed a microwave-assisted transesterification reaction for the easy identification of esters for qualitative organic analysis. This facile protocol allows for faster reaction, improved energy efficiency, smaller amounts of reagents, reducing chemical waste and exposure to hazardous substances. We believe, the rapid and visible results of the above microwave-assisted transesterification reaction will keep students engaged, helping them better understand and appreciate the concepts of organic synthesis and green chemistry.

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