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Original Article

Synthesis of 4-Hydroxy-3-nitro Benzaldehyde using Green chemistry principles

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ABSTRACT

Green chemistry experiments are introduced not to drastically replace the conventional ones rather, they are considered complementary to the existing protocols.

This not only provides a wider view of various techniques but also imbibes inquest in innovative minds for future development and growth of the subject in general with due emphasis to green chemistry context. Green chemistry involves use of chemicals which are easily available and their sources are ever existing rather than depleting. It involves mainly least step synthesis procedures.It mainly gives the product in very high yield. Green chemistry is increasingly seen as a powerful tool that researchers must use to evaluate the environmental impact. Substances and the form of a substance used in a chemical process should be chosen to minimize potential for chemical accidents, including releases, explosions, and fires.

Keywords: Green chemistry , chemicals , considered complementary

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1.Introduction

Nitration is a general class of chemical process for the introduction of a nitro group into an organic chemical compound.

Typical nitration syntheses apply so-called "mixed acid", a mixture of concentrated nitric acid and sulfuric acids. This mixture produces the nitronium ion (NO2+), which is the active species in aromatic nitration.

In this project along with nitrating mixture I have used calcium nitrate as nitrating agent. The solvent used for this reaction is glacial acetic acid. This agent is eco-friendly and non hazardous unlike strong acids such as conc. Nitric acid and conc. Sulfuric acid.

Nitration is rapid. Ecofriendly nitration of phenols and its derivatives without nitric acid is achieved. Reagents and byproducts (calcium acetate) in this reaction are useful agrochemicals, environmentally benign and thus eco-friendly. Regioselective nitration is achieved.

REQUIREMENTS:-

Para hydroxy benzaldehyde
Glacial acetic acid
Calcium nitrate
Cold water

EXPERIMENTAL WORK UP:-

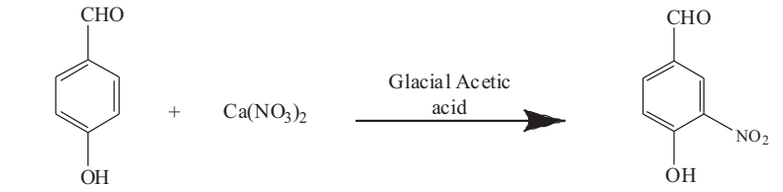
Para hydroxy benzaldehyde 2gm was taken in a dry 50ml round bottom flask fitted with a reflux condenser. A mixture of calcium nitrate 28.640mmol (4.7gm) and glacial acetic acid (10ml) was prepared and then added. The reaction mixture was refluxed and then monitored by TLC (50:50, chloroform: pet ether). After 3 hours the heating was stopped and the mixture

was cooled. The mixture was then poured into ice cold water with vigorous stirring. The crude solid obtained was filtered washed thoroughly with water and dried. The dried product was re-crystallised from methanol to give light yellow product.

RESULT:-

Practical yield of the product:-2.089 gm
Percentage yield of the product:- 75%

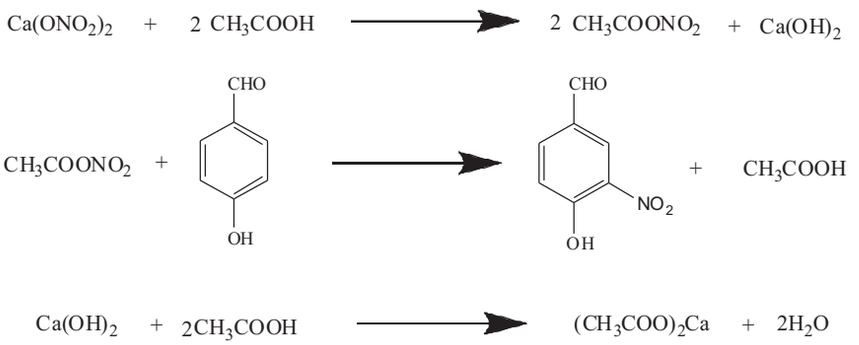
REACTION:-



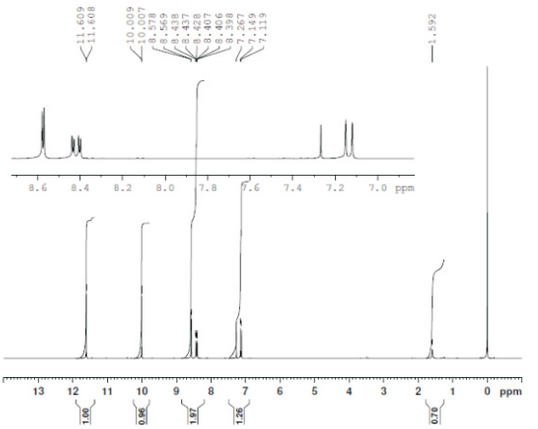
4-hydroxy benzaldehyde

4-hydroxy-3-nitro benzaldehyde

MECHANISM:-

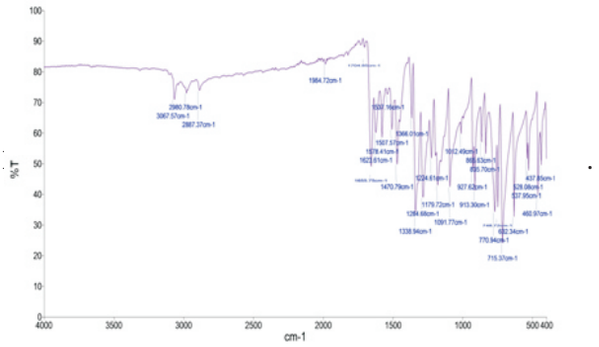


1H NMR spectra:-



1H NMR spectra of 4-Hydroxy-3-nitro Benzaldehyde.

IR spectra:-



CONCLUSION:-

The procedure using calcium nitrate as nitrating agent gave good yield as compared to method using conc. sulfuric acid - fuming nitric acid.

Milder reaction condition are necessary for active compounds such as phenols, amines, pyrroles. Highly accelerated and safe nitration of phenolic compounds has been found to be feasible with a mixture of calcium nitrate and acetic acid as an efficient nitration agent. This method is compatible with the green chemistry approach.

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