

Efficient Synthesis and Characterization of 4-(1, 3-Dioxoisindolin-2yl) Benzaldehyde

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ABSTRACT

The titled 4-(1,3-dioxo-2,3-dihydro-1H-inden-2-yl)benzaldehyde has been synthesized from phthalic anhydride and p-toluidine followed by use of H₂O₂ in ethanol. Compounds characterized on the basis of 1H-NMR.

Keywords: Phthalic anhydride and H₂O₂ in ethanol

I. INTRODUCTION

Benzaldehyde (C₆H₅CHO) is a chemical compound consisting of a benzene ring with an aldehyde substituent. It is the simplest representative of the aromatic aldehydes and one of the most industrially used members of this family of compounds. It is commonly employed as a commercial food flavourant or industrial solvent, benzaldehyde is used chiefly in the synthesis of other organic compounds, ranging from pharmaceuticals to plastic additives.

EXPERIMENTAL

The uncorrected M.P. Of compounds were taken in an open capillary in a paraffin bath and compared with those in the literature values. 1H-NMR and 13C-NMR were recorded on a 300 MHz spectrometer in DMSO solvent.

RESULTS AND DISCUSSION**Synthesis of 2-(p-tolyl)isoindoline-1,3-dione (a)**

To phthalic anhydride (1 mmol) and p-toluidine which were refluxed in glacial acetic acid for 3 hrs. The progress of the reaction was monitored using TLC. This reaction was then quenched in water. The crude product was filtered and washed several times with water and then dried, mp 180-185⁰C and 84% yield. 1H-NMR (DMSO) δ-3.251(s, 3H), δ-7.281-7.935 (m, 8H, Ph). 13C-NMR δ-21, 123, 126, 129, 129, 131, 134, 137, 167.

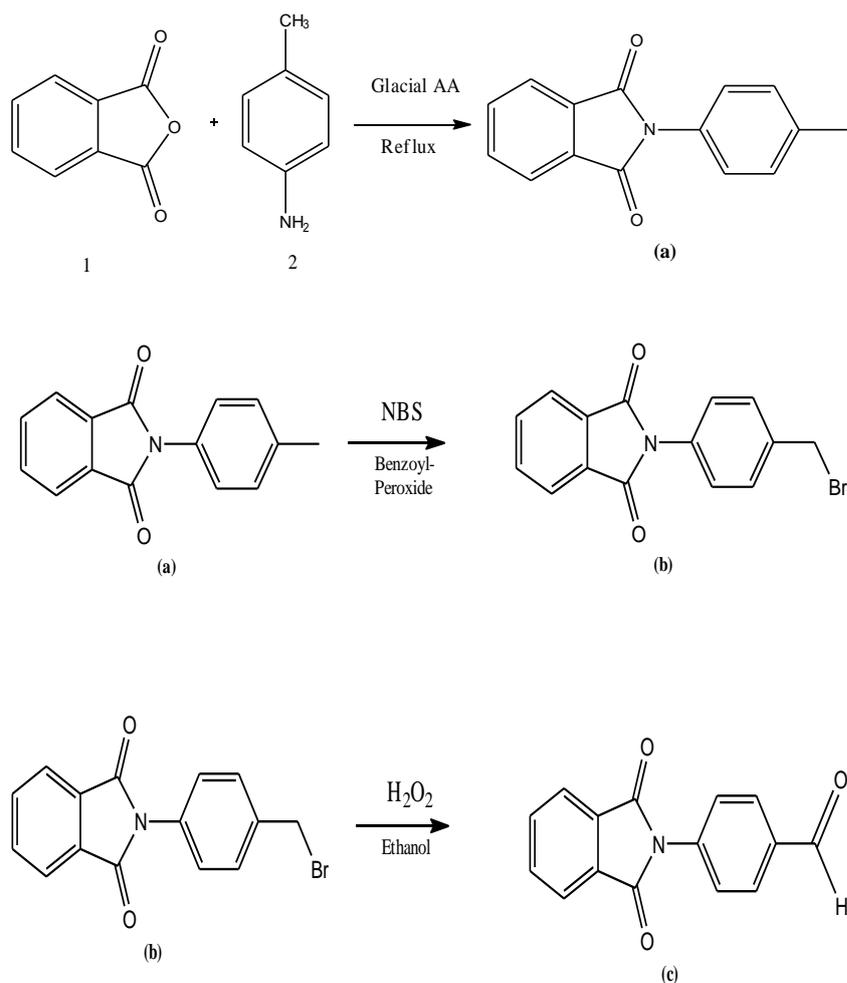
Synthesis of 2-(4-(bromomethyl) phenyl) isoindoline-1,3-dione (b)

The product obtained in the first step is then subjected to bromination by using NBS in presence of benzoyl-peroxide as catalyst in CCl₄. The reaction mixture is refluxed for 2 hrs and it is monitored by TLC. The reaction product found as a white mass. The mixture was brought to room temperature, and CCl₄ was then evaporated, filtered and washed with CCl₄ and water successively. The crude product was then dried for 2 hours. The dried product, mp 198-200⁰C, was not dissolving even in methanol so we could not able to predict the compound using NMR spectroscopic technique. However compound gave positive Bleistein's test which confirmed the presence of bromine.

Synthesis of 4-(1,3-dioxoisoindolin-2-yl) benzaldehyde (c)

The brominated product (b) was then oxidised to benzaldehyde by use of H₂O₂ in ethanol as oxidant⁵ at reflux in 3h. In other solvents, such as tetrahydrofuran, chloroform, and methylene chloride, much longer time was and the conversion was poorer required.

The ethanol is then evaporated and dried for 2hrs in an hot air oven. 1H NMR (DMSO) δ-10.068 (s, 1H), δ-7.388-7.839 (m, 8H, Ph). 13C-NMR δ-123, 130, 134, 135, 166, 167, 191.



SCHEME 1

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