Efficient synthesis and characterization of 2-(4-((phenylimino)methyl)phenyl)isoindoline-1,3-dione

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***Abstract*—The titled [2-(4phenylimino)methyl)phenyl)isoindoline-1,3-dione] has been synthesized from phthalic anhydride and p-toluidene followed by use of H_2O_2 in glacial acetic acid and aniline to from a Schiff base which is then characterized on the basis of 1H-NMR.**

Keywords— Phthalic anhydride, H_2O_2 in glacial acetic acid, aniline, Schiff base

I. INTRODUCTION

Schiff bases are aldehyde- or ketone-like compounds in which the carbonyl group is replaced by an imine group. They are widely used for industrial purposes and also exhibit a broad range of biological activities.

Schiff's bases have been playing vital roles in pharmaceuticals, rubber additives[1-2], as amino protective groups in the synthetic organic chemistry and several biologically active organic compounds[3-4]. They are also used as liquid crystals[5] in analytical[6], medicinal[7-8]and polymer chemistry[9-10]. They are most promising antimalarial, antibacterial, antifungal, and antiviral compounds. The imine group present in such compounds has been shown to be critical to their biological activities.

II. 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.

III. 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 180-185^{0}C 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 CCl_4. 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 CCl_4 was then evaporated, filtered and washed with CCl_4 and water successively. The crude product was then dried for 2 hours. The dried product, mp 198-200^{0}C, 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.

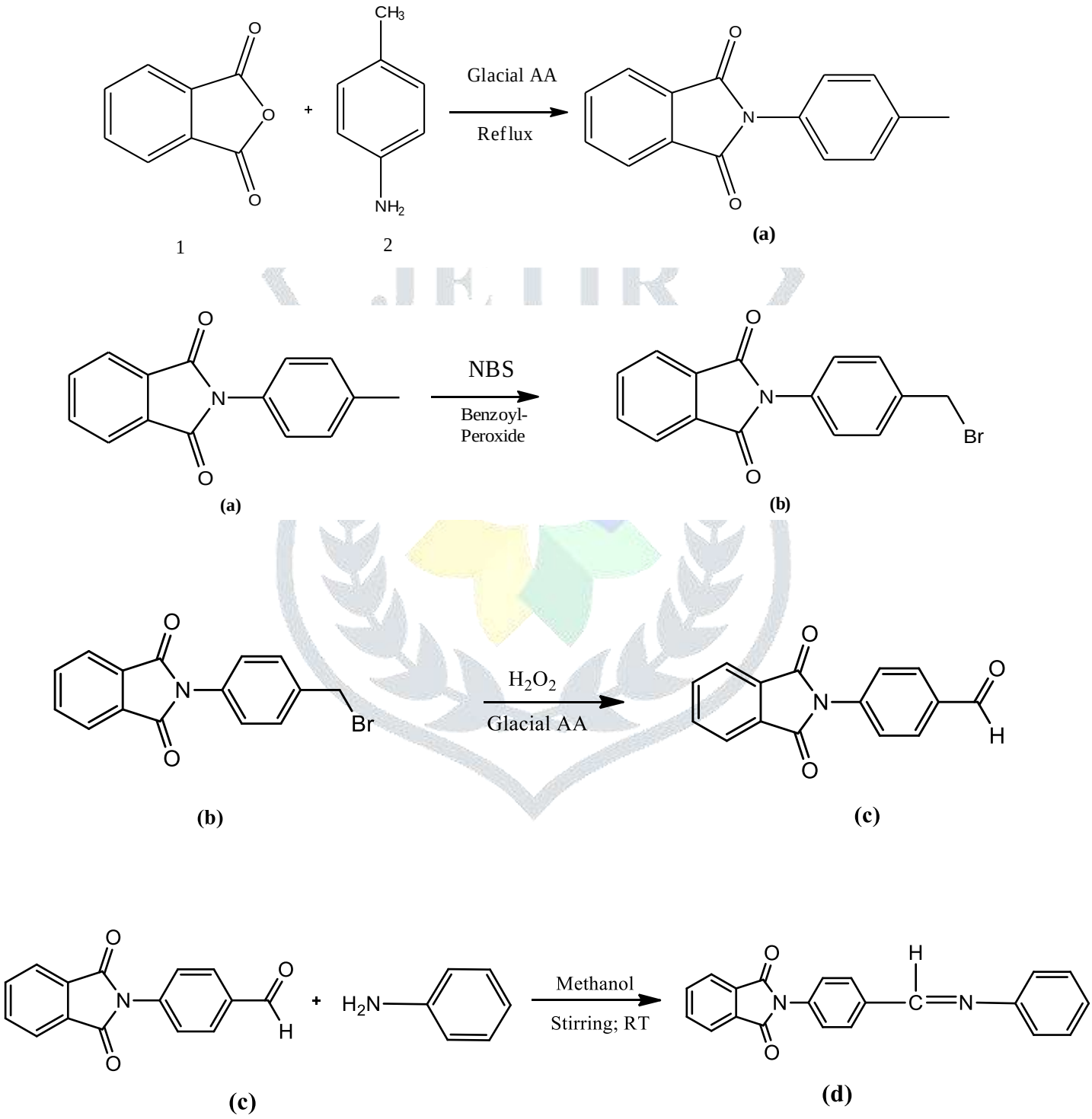
Synthesis of 4-(1,3-dioxoisoindolin-2-yl)benzaldehyde (c)

The brominated product (b) was then oxidised to benzaldehyde by use of H_2O_2 in ethanol as oxidant[12] at reflux in 3h. In other solvents, such as tetrahedron, chloroform, and methylene chloride, much longer time was required and the conversion was poorer. Melting point of the isolated compound is 134^{0}C and 30%

yield. 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.

Synthesis of 2-(4-((phenylimino)methyl)phenyl)isoindoline-1,3-dione (d)

Aniline dissolved in absolute ethanol and then added slowly to solution of (c) in an absolute ethanol. The resulting mixture was then stirred with refluxion for 12 hours. The progress of the reaction was monitored by TLC. Then the mixture is filtered washed with cold ethanol and then recystallized from ethanol.
1H NMR (DMSO) δ-8.64 (s, 1H), δ-7.88-7.06 (m, 13H, Ph). 13C-NMR δ-122,123,124,127,130,152,167.



1 2 (a) (b) (c) (d)

SCHEME

IV. ACKNOWLEDGMENT

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