

# Efficient Synthesis and Characterization of 4-(1,3-dioxoisindolin-2-yl) Benzoic Acid

Sharad S. Sankhe\*, Nitesh R. Chindarkar

Organic Research Laboratory, Patkar-Varde College of Science, Goregaon (West), Mumbai, Maharashtra, India

## ABSTRACT

The titled 4-(1,3-dioxoisindolin-2-yl)benzoic acid has been synthesized from phthalic anhydride and methyl 4-aminobenzoate followed by use of NaOH in ethanol. Compounds characterized on the basis of IR and <sup>1</sup>H-NMR.

**Keywords :** Phthalic Anhydride, Methyl 4-Amino Benzoate and NaOH in Ethanol

## I. INTRODUCTION

Benzoic acid is a colourless crystalline solid and a simple aromatic carboxylic acid. The name is derived from gum benzoin, which was for a long time its only known source. Benzoic acid occurs naturally in many plants and it serves as an intermediate in the biosynthesis of many secondary metabolites. Salts of benzoic acid are used as food preservatives and benzoic acid is an important precursor for the industrial synthesis of many other organic substances. The salts and esters of benzoic acid are known as benzoates.

## II. EXPERIMENTAL

The uncorrected M.Ps. of compounds were taken in an open capillary in a paraffin bath and compared with those in the literature values. <sup>1</sup>H-NMR and <sup>13</sup>C-NMR were recorded on a 300 MHz spectrometer in CDCl<sub>3</sub> solvent.

## III. RESULTS AND CONCLUSION

### Synthesis of methyl 4-aminobenzoate (a)

4-Aminobenzoic acid (5 g) was dissolved in methanol (50 mL) saturated initially with dry hydrogen chloride gas. The solution was refluxed on water bath for 2 hrs

and the reaction mixture was concentrated to half of its original volume. The solution was quenched into cold water (50 mL) to get a clear solution which was neutralized with solid sodium bicarbonate to get a precipitate which was filtered, washed with cold water and dried to obtain the desired compound as white colored solid (4.7 g, 85%) m.p. 111-113 °C. IR : 3416, 3120, 1714, 1608, 1400, 1113 and 857 cm<sup>-1</sup>

### Synthesis of methyl 4-(1,3-dioxoisindolin-2-yl)benzoate (b)

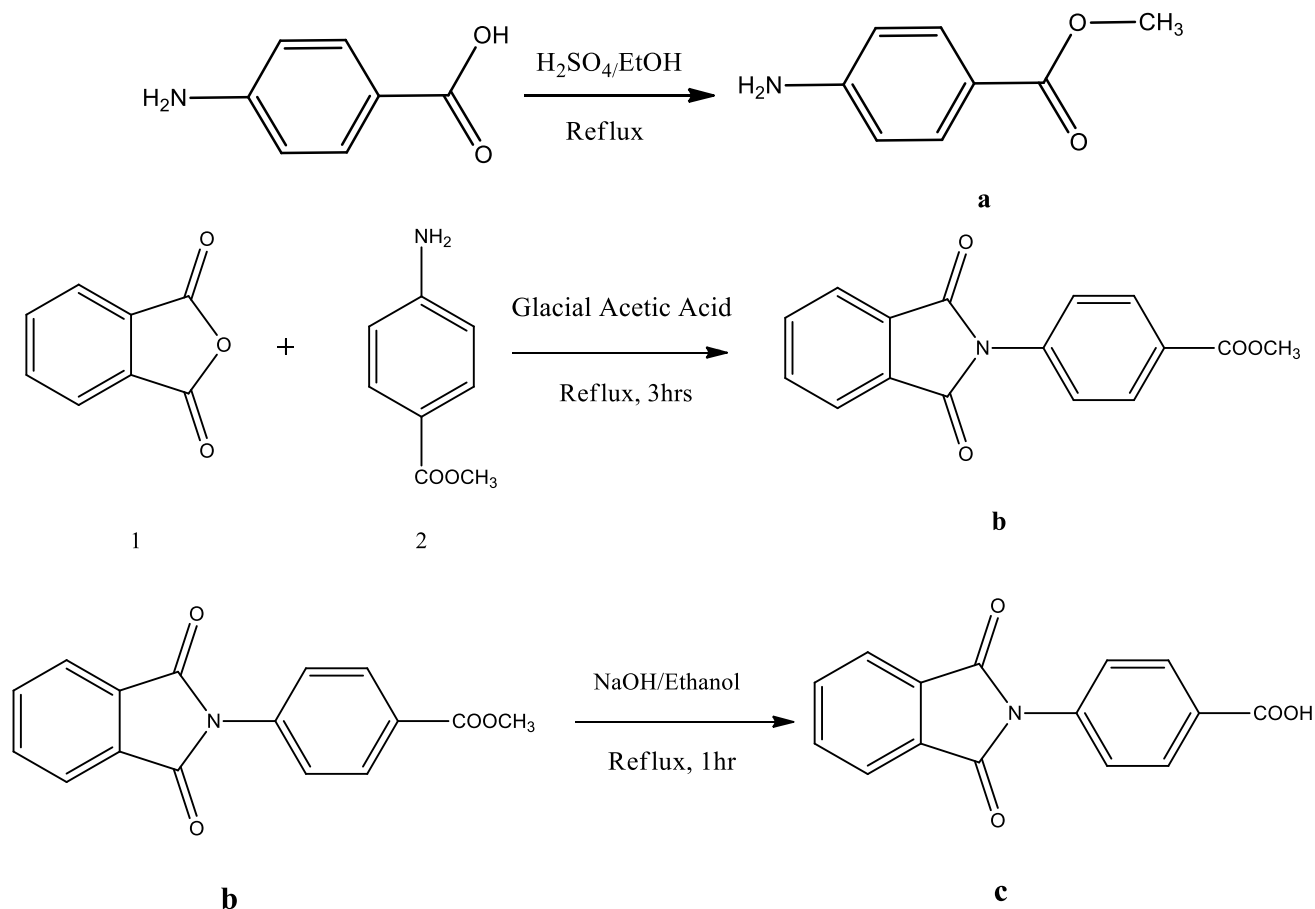
Phthalic anhydride (1 mmol) and methyl 4-aminobenzoate (1 mmol) were refluxed in glacial acetic acid for 3 hrs. The progress of the reaction was monitored using TLC. This reaction was then quenched in water. The crude product was filtered and washed several times with water and then dried, mp 175-180°C and 70% yield. <sup>1</sup>H-NMR (CDCl<sub>3</sub>) δ-3.941(s, 3H), δ-7.275-8.182 (m, 8H, Ph). <sup>13</sup>C-NMR δ-52, 123, 125, 129, 130, 131, 134, 135, 166, 166.

### Synthesis of 4-(1,3-dioxoisindolin-2-yl)benzoic acid (c)

The product obtained in the first step is then dissolved in 2N NaOH solution. The reaction mixture is refluxed for 1 hr and it is monitored by TLC. The reaction product found as a white mass. The mixture was brought to room temperature, and ethanol was

then evaporated. The crude product was then dried for 2 hours in hot air oven. The dried product, mp ~220°C and 42% yield. <sup>1</sup>H-NMR (CDCl<sub>3</sub>) δ-10.94 (s,

H), δ-7.38-7.90 (m, 8H, Ph). <sup>13</sup>C-NMR δ-123, 124, 125, 130, 132, 137, 167, 169



**Scheme 1.**

#### IV. ACKNOWLEDGMENT

Authors thanks to Rajesh Kenny, Suyog Marathe, Jitendra Patil, S.S. & L.S. Patkar College and A.P.Shah Institute of Technology, Thane, for support.

#### V. REFERENCES

- [1]. R K Sharma, Chetana Sharma & Prerna, Indian Journal of Chemistry, 51B, 2012, 1489-1493
- [2]. K.Shelke, G. Kakade, B.Shingate, M.Shingare, Rasayan J.Chem, 1, 2008, 489-494
- [3]. Saikat Das Sharma, Parasa Hazarika, Dilip Konwar, Tetrahedron Letters, 49, 2008, 2216-2220
- [4]. Subhasis Samai, Ganesh Chandra Nandi, Pallavi Singh, M.S.Singh, Tetrahedron, 65, 2009, 10155-10165
- [5]. Sayyed Sultan Qasim, Shaikh Nasreen, Syed shahed ali, International Journal of Applied Biology and Pharmaceutical Technology, 2, 2011, 12-18
- [6]. Behrooz Maleki, Hossein Keshvari and Ali Mohammad, Oriental Journal of Chemistry, 28, 2012, 28, 1207-1212

- [7]. Li-Min Wang, Yong-Hong Wang, He Tian, Yin-Fang Yao, Jue-Hua Shao, Bo Liu, *Journal of Fluorine Chemistry*, 127, 2006, 1570-1573
- [8]. A.Puratchikody and Mukesh Doble, *Bioinorganic & Medicinal Chemistry*, 15, 2007, 1083-1090
- [9]. Hass H. B.; Bender, M.L. *Am. Chem.Soc* (1949), 71, 1767-1769
- [10]. Frazen, V.Org, *Synth*, (1973), 5, 872-874
- [11]. Jun Matsui, Ian A. Nicholls, Isao Karube, and Klaus Mosbach, *J. Org. Chem.*,16, (1996) pp 5414–5417
- [12]. Kiyoshi Tanemura, Tsuneo Suzuki, Yoko Nishida, Koko Satsumabayashi, Takaaki Horaguchi, (2003), 32, No.10
- [13]. Jingting Tang, Jinlong Zhu, Zongxuan Shen and Yawen Zhang, *Tetrahedron Letters* 48 (2007) 1919-1921