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### Efficient one pot synthesis of N-alkyl and N-aryl imides

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#### ABSTRACT

*Imide derivatives have been found to possess a broad spectrum of biological activities. A variety of methods have been reported for the preparation of this class of compound. However, in spite of their potential utility, some of the reported methods suffer from drawbacks such as long reaction times, cumbersome product isolation procedures. Thus there is still need for development of new method for synthesis of imides. In the present work we have reported use of sulphamic acid for synthesis of imides in few minutes.*

#### INTRODUCTION

The development of simple general and efficient synthetic methods for widely used organic compounds from readily available reagents is one of the major challenges in organic synthesis. Imide derivatives are among such type of organic compound with numerous applications in biological [1] synthetic [2] and polymer [3] chemistry. To date, most commonly N-substituted phthalimides are formed by direct dehydrative condensation of an amine with phthalic anhydride. [1-5] But those methods needed high temperature and long reaction times. For example, synthesis of N-phenyl-phthalimide <sup>1c</sup> needed 2 hrs at 160<sup>0</sup> C-180<sup>0</sup> C. Recently improvements in classical methods has been made using Lewis acid catalyst, [4] Phthalic anhydride and azide with in situ reduction by condensation using chlortrimethylsilane–sodium iodide [4] and microwave irradiation. Recently ionic liquids have been used for the synthesis which are costly.[6,7] Reported methods have limitations of harsh reaction conditions, [4] costly reagents, and long reaction time, poor yields.[5] Therefore a development of simple, relatively mild, efficient method for synthesis of N-alkyl and N-aryl phthalimides and succinimides is a major challenge.

Herein I am reporting a simple, efficient and practical method for the synthesis of N-alkyl and N-aryl phthalimides and succinimides using 10 mol% Sulphamic acid catalyst.

Sulphamic acid ( $\text{H}_2\text{NSO}_3\text{H}$ ), which is a common organic acid with mild acidity, involatility and incorrosivity. It is insoluble in common organic solvents. It is a very stable white crystalline solid and has already been demonstrated that sulphamic acid is composed of  $\text{H}_3\text{N}^+ \text{SO}_3^-$  zwitterionic units by both X-ray and neutron diffraction techniques.[8-9] During the last few years, sulphamic acid has emerged as an acid catalyst for acid catalyzed reaction, viz. acetalisation,[10] esterification,[11] acetylation of alcohols and phenols,[12] and tetrahydropyranylation of alcohols.[13] Very recently, sulphamic acid has been used as a chemo selective catalyst for the transesterification of  $\beta$ -ketoesters [14] and Beckman rearrangement.[15]. It has been recently reported use of sulphamic acid in Biginelli reaction.[16]

### MATERIALS AND METHODS

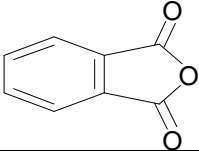
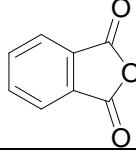
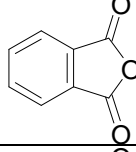
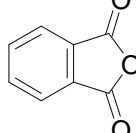
#### Typical experimental Procedure for synthesis of 3a

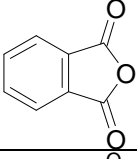
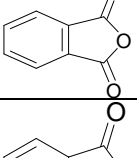
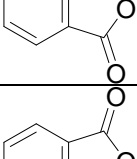
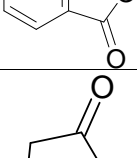
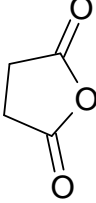
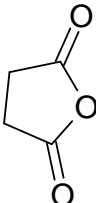
A mixture of Phthalic anhydride (10 mmol) and aniline (10 mmol) in acetic acid (5 times) was stirred at  $110^\circ\text{C}$  for appropriate time (table 1). The reaction mixture was poured into water. The solid was collected by filtration under suction and washed with ethyl acetate to give N-phenyl phthalimide as white solid.

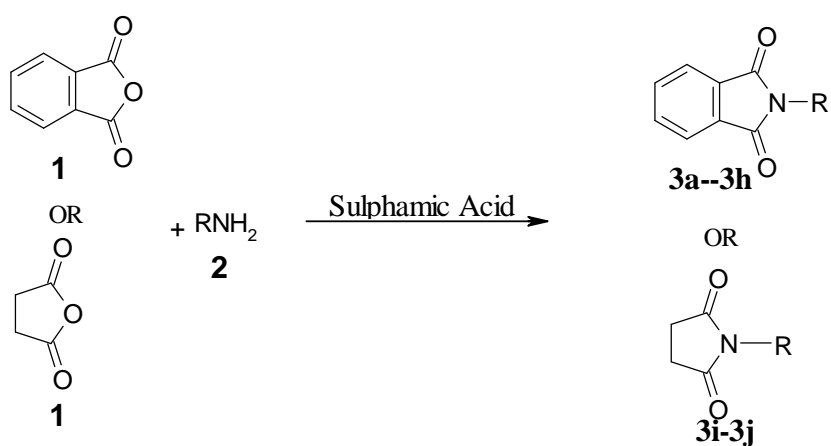
mp  $204\text{--}205^\circ\text{C}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$ : 7.69-7.92(m, 4H), 7.24-7.50 (m, 5H), .ES/MS, m/z;224 (M+1).

This procedure was followed for preparation of all N aryl Phthalimides and succinimides listed in Table 1. All compounds were identified by comparison of analytical data ( $^1\text{H NMR}$  and mass spectra) and melting points with published data.

Table 1: Synthesis of N-alkyl and N-aryl imides using sulphamic acid catalyst

Entry	Anhydride	R	Time in minutes	Yield (%)
3a		$\text{C}_6\text{H}_5\text{NH}_2$	05	98
3b		P-Cl $\text{C}_6\text{H}_5\text{NH}_2$	05	98
3c		P- $\text{NO}_2$ $\text{C}_6\text{H}_5\text{NH}_2$	05	95
3d		P- $\text{CH}_3$ $\text{C}_6\text{H}_5\text{NH}_2$	05	96

3e		m-OH C <sub>6</sub> H <sub>5</sub> NH <sub>2</sub>	15	96
3f		C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> NH <sub>2</sub>	10	95
3g		4,5 di methoxy C <sub>6</sub> H <sub>5</sub> NH <sub>2</sub>	10	86
3h		NH <sub>2</sub> CH <sub>2</sub> COOH	10	96
3i		C <sub>6</sub> H <sub>5</sub> NH <sub>2</sub>	10	90
3j		C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> NH <sub>2</sub>	10	96



Scheme 1

## RESULTS AND DISCUSSION

Initially, we studied the catalytic properties of sulphamic acid for the synthesis of N-phenyl phthalimides **3a** using Phthalic anhydride (**1**) and aniline (**2**) substrates (Scheme -1) and varying the mol % of sulphamic acid. Among the results obtained, use of 10 mol% sulphamic acid gave better yield (98%) for synthesis of **3a**.

We investigated the reaction for a series of substituted anilines with anhydrides to get the corresponding imides (Table 2). The present method was found to be effective for both electron-donating and electron-withdrawing substituted anilines. The results are summarized in table 2. All synthesized derivatives were characterized using mass and <sup>1</sup>H NMR. The easy work-up of the reaction was also the advantageous aspect of this method. It includes the pouring the reaction mixture in water which on filtration gave the imides. This method was superior in regards with yield and reaction time than the previously reported methods.

## CONCLUSION

In conclusion, we have demonstrated an efficient and simple alternative for the preparation of N-alkyl and N-aryl phthalimides and using sulphamic acid catalyst. Simple handling, short reaction time, easily and cheaply available sulphamic acid as catalyst and excellent yield are the advantages of the proposed method. Hence, the utility of sulphamic acid catalyst for synthesis of N-alkyl and N-aryl phthalimides and succinimides would be good addition to available methods.

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