This article was downloaded by: [Moskow State Univ Bibliote] On: 13 February 2014, At: 06:39 Publisher: Taylor & Francis Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information: <u>http://www.tandfonline.com/loi/lsyc20</u>

# A New Simple and Efficient Synthesis of N-Aryl Phthalimides in Ionic Liquid [bmim][PF<sub>6</sub>]

Mei-Yun Zhou<sup>a</sup>, Yi-Qun Li Ph.D.<sup>a</sup> & Xin-Ming Xu<sup>a</sup> <sup>a</sup> Department of Chemistry, Jinan University, Guangzhou, P.R. China Published online: 16 Aug 2006.

To cite this article: Mei-Yun Zhou , Yi-Qun Li Ph.D. & Xin-Ming Xu (2003) A New Simple and Efficient Synthesis of N-Aryl Phthalimides in Ionic Liquid [bmim][PF<sub>6</sub>], Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 33:21, 3777-3780, DOI: <u>10.1081/SCC-120025187</u>

To link to this article: http://dx.doi.org/10.1081/SCC-120025187

### PLEASE SCROLL DOWN FOR ARTICLE

Taylor & Francis makes every effort to ensure the accuracy of all the information (the "Content") contained in the publications on our platform. However, Taylor & Francis, our agents, and our licensors make no representations or warranties whatsoever as to the accuracy, completeness, or suitability for any purpose of the Content. Any opinions and views expressed in this publication are the opinions and views of the authors, and are not the views of or endorsed by Taylor & Francis. The accuracy of the Content should not be relied upon and should be independently verified with primary sources of information. Taylor and Francis shall not be liable for any losses, actions, claims, proceedings, demands, costs, expenses, damages, and other liabilities whatsoever or howsoever caused arising directly or indirectly in connection with, in relation to or arising out of the use of the Content.

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms & Conditions of access and use can be found at <a href="http://www.tandfonline.com/page/terms-and-conditions">http://www.tandfonline.com/page/terms-and-conditions</a>



MARCEL DEKKER, INC. • 270 MADISON AVENUE • NEW YORK, NY 10016

©2003 Marcel Dekker, Inc. All rights reserved. This material may not be used or reproduced in any form without the express written permission of Marcel Dekker, Inc.

SYNTHETIC COMMUNICATIONS<sup>®</sup> Vol. 33, No. 21, pp. 3777–3780, 2003

## A New Simple and Efficient Synthesis of *N*-Aryl Phthalimides in Ionic Liquid [bmim][PF<sub>6</sub>]

Mei-Yun Zhou, Yi-Qun Li,\* and Xin-Ming Xu

Department of Chemistry, Jinan University, Guangzhou, P.R. China

#### ABSTRACT

The room temperature ionic liquid [bmim][ $PF_6$ ], namely 1-butyl-3methyl-imidazolium hexafluorophosphate, is used as an alternative solvent to classic solvents for the synthesis of a series of *N*-aryl phthalimides in good to excellent yields.

Key Words: N-Aryl phthalimides; Ionic liquid; Synthesis.

Imide derivatives are an important class of substrates for biological and chemical applications.<sup>[1]</sup> Accordingly, the synthesis of these derivatives has developed remarkably in recent years. Well known methods are: dehydrative condensation of an anhydride and

DOI: 10.1081/SCC-120025187 Copyright © 2003 by Marcel Dekker, Inc. 0039-7911 (Print); 1532-2432 (Online) www.dekker.com

<sup>\*</sup>Correspondence: Yi-Qun Li, Associate Professor, Ph.D., Assistant head of department, Department of Chemistry, Jinan University, 601 Huangpu Dadaoxl, Guangzhou 510632, P.R. China; Fax: 86-20-85221697; E-mail: tlyq@ jnu.edu.cn.

<sup>3777</sup> 

3778

©2003 Marcel Dekker, Inc. All rights reserved. This material may not be used or reproduced in any form without the express written permission of Marcel Dekker, Inc.

#### Zhou, Li, and Xu

amine catalyzed conc.  $H_2SO_4$  in acetic anhydride at refluxing.<sup>[2]</sup> Direct *N*-alkylation of phthaloyl dichloride with azide in the presence of PPh<sub>3</sub> in CH<sub>2</sub>Cl<sub>2</sub><sup>[3]</sup> and *N*-alkylation of imides using of alcohol promoted by PPh<sub>3</sub> and DIAP in THF<sup>[4]</sup> etc. These methods have not been entirely satisfactory owing to such drawbacks as using of organic solvents. It is necessary to develop an alterative solvent for the synthesis of imides under mild and environmentally benign conditions.

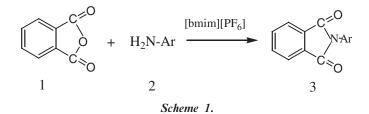
Recently, ionic liquids have gained increasing interest in organic synthesis. This is mainly because their negligible vapor pressure, easy of handling and potential for recycling, circumvents many of the problems associated with volatile organic solvents. With the continuing depletion of natural resources and growing environmental awareness in the practices of both the chemical industry and academia, the replacement of deleterious molecular solvents with environmentally more benign has emerged. Examples of their application in organic reactions have been summarized in a number of recent review articles.<sup>[5]</sup>

In this article we describe a new and efficient synthetic approach to N-aryl phthalimides from phthalic anhydride and aromatic amines in ionic liquids, [bmim][PF<sub>6</sub>] (Sch. 1). The ionic liquids used here not only overcome many of the problems brought from volatile organic solvents, but also be recovered for reuse.

In summary, the synthetic approach reported here is an attractive addition to the existing methodologies since the procedure is simple, the yields are excellent, the product purification is convenient, and the ionic liquids can be reused and moreover it is benign to the environment.

#### **EXPERIMENTAL**

Melting points were uncorrected. Infrared spectra were recorded using KBr pellets on a Bruker Equinox 66 spectrometer. All chemicals used were of commercial grade without further purification. The ionic liquids, [bmim][PF<sub>6</sub>] used here is prepared according to the literature.<sup>[6]</sup>



MARCEL DEKKER, INC. • 270 MADISON AVENUE • NEW YORK, NY 10016

©2003 Marcel Dekker, Inc. All rights reserved. This material may not be used or reproduced in any form without the express written permission of Marcel Dekker, Inc.

#### N-Aryl Phthalimides in Ionic Liquid

#### 3779

**Table 1.** The *N*-aryl phthalic anhydride with aromatic amines in ionic liquids  $[\text{bmim}]PF_6^{a}$ .

Entry	Ar (1)	Reaction time (h)	M.p. (°C)	Lit. mp (°C)	Yields <sup>b</sup> (%)
3a 3b 3c 3d 3e 3f	$C_6H_5$ $o-MeOC_6H_4$ $p-MeC_6H_4$ $p-NO_2C_6H_4$ $p-CIC_6H_4$ $p-BrC_6H_4$	8 8 8 8 8	206–208 155–156 203–204 264–266 192–194 202–204	$\begin{array}{r} 206^{[7]}\\ 155-156^{[8]}\\ 204^{[8]}\\ 264-266^{[8]}\\ 194-195^{[8]}\\ 203-204^{[8]} \end{array}$	93 92 97 91 90 90
31 3g	$\alpha$ -C <sub>10</sub> H <sub>7</sub>	8	182–184	$184 - 185^{[8]}$	90 94

<sup>a</sup>All of these compounds were confirmed by IR and compared with authentic samples.

<sup>b</sup>Isolated yields.

#### **General Procedure**

To a 25 mL round-bottomed flask, a mixture of phthalic anhydride (1, 7.5 mmol) and an aromatic amine (5.0 mmol) in [bmim][PF<sub>6</sub>] (2 mL) was added and stirred for 8 h at 80°C as indicated in Table 1. On completion of the reaction as monitored by TLC, EtOH (95%, 5 mL) was added to the reaction mixture and was heated to reflux for 10 min to extract the ionic liquid. After cooling, the product was filtered off and washed with EtOH for several times to afford the pure *N*-aryl phthalimide as a white solid. The ionic liquid can be reused for the next run prior removing the ethanol and drying in vacuum. The melting points and IR of compound **3a–g** agreed well with the reported data.<sup>[7,8]</sup>

#### ACKNOWLEDGMENT

The Project Sponsored by the National Nature Science Foundation of China (20272018), the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry of China and the Nature Science Foundation of Guangdong Province (974021, 021166).

#### REFERENCES

1. (a) Da Settimo, A.; Primofiore, G.; Da Settimo, F.; Simorini, F.; La Motta, C.; Martinelli, A.; Boldrine, E. Eur. J. Med. Chem. **1996**,  $\mathbb{H}^{+}$ 

MARCEL DEKKER, INC. • 270 MADISON AVENUE • NEW YORK, NY 10016

©2003 Marcel Dekker, Inc. All rights reserved. This material may not be used or reproduced in any form without the express written permission of Marcel Dekker, Inc.

#### 3780

#### Zhou, Li, and Xu

*31*, 69; (b) Langmuir, M.E.; Yang, J.R.; Moussa, A.M.; Laura, R.; Lecompte, K.A. Tetrahedron Lett. **1995**, *36*, 3989; (c) Mayer, A.; Neuenhofer, S. Angew. Chem. Int. Ed. Engl. **1994**, *33*, 1044; (d) Rusiecki, V.K.; Warne, S.A. Biorg. Med. Chem. Lett. **1993**, *3*, 707; (e) Reddy, P.Y.; Kondo, S.; Toru, T.; Ueno, Y. J. Org. Chem. **1997**, *62*, 2652.

- 2. Prapas, A.G. J. Org. Chem. 1959, 24, 388.
- 3. Aubert, M.T.; Farnier, M.; Guilard, R. Tetrahedron 1991, 47, 53.
- 4. Walker, M.A. J. Org. Chem. 1995, 60, 5352.
- (a) Wasserscheid, P.; Keim, W. Angew. Chem. Int. Ed. 2000, 39, 3722; (b) Sheldon, R. Chem. Commun. 2001, 2399.
- (a) Bonhôte, P.; Dias, A.P.; Papageorgiou, N.; Kalyanasundaram, K.; Grätzel, M. Inorg. Chem. **1996**, *35*, 1168; (b) Suarez, P.A.Z.; Dulius, J.E.L.; Einloft, S.; de Souza, R.F.; Dupont, J. Polyhedron **1996**, *15*, 1217.
- Mary, L.S.; Florence, L.S.; Elizabeth P.S. J. Am. Chem. Soc., 1928, 50, 47.
- 8. Ian Heilbron, Bunbury, H.M. *Dictionary of Organic Compounds* (*Chinese*), Scientific Press: China, 1996; 292.

Received in Japan March 17, 2003