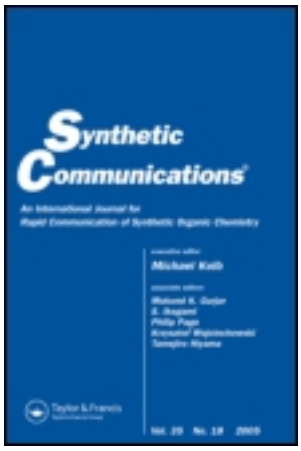

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A New Simple and Efficient Synthesis of *N*-Aryl Phthalimides in Ionic Liquid [bmim][PF_6]

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ABSTRACT

The room temperature ionic liquid [bmim][PF_6], namely 1-butyl-3-methyl-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.[1] Accordingly, the synthesis of these derivatives has developed remarkably in recent years. Well known methods are: dehydrative condensation of an anhydride and

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amine catalyzed conc. H_2SO_4 in acetic anhydride at refluxing.[2] Direct *N*-alkylation of phthaloyl dichloride with azide in the presence of PPh_3 in CH_2Cl_2[3] and *N*-alkylation of imides using of alcohol promoted by PPh_3 and DIAP in THF[4] 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.

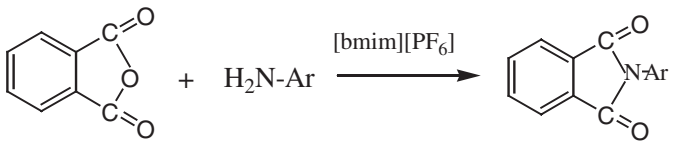
Recently, ionic liquids have gained increasing interest in organic synthesis. This is mainly because their negligible vapor pressure, easy of handling and potential for recycling, circumvents many of the problems associated with volatile organic solvents. With the continuing depletion of natural resources 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.[5]

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_6] (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_6] used here is prepared according to the literature.[6]



1 2 3

Scheme 1.

Table 1. The *N*-aryl phthalic anhydride with aromatic amines in ionic liquids [bmim]PF_6[a].

Entry	Ar (1)	Reaction time (h)	M.p. (°C)	Lit. mp (°C)	Yields[b] (%)
3a	C_6H_5	8	206–208	206[7]	93
3b	*o*-$MeOC_6H_4$	8	155–156	155–156[8]	92
3c	*p*-MeC_6H_4	8	203–204	204[8]	97
3d	*p*-$NO_2C_6H_4$	8	264–266	264–266[8]	91
3e	*p*-ClC_6H_4	8	192–194	194–195[8]	90
3f	*p*-BrC_6H_4	8	202–204	203–204[8]	90
3g	α-$C_{10}H_7$	8	182–184	184–185[8]	94

[a]All of these compounds were confirmed by IR and compared with authentic samples.
[b]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_6] (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.[7,8]

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