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Complex of [BMIm] PF₆ with PEG₁₀₀₀: a high efficient and recycle system for palladiumcatalyzed Suzuki cross-coupling and Heck reaction

Xiang Liu, Yang Mao and Ming Lu*

A complex of [BMIm] PF₆ (1-Butyl-3-methylimidazolium hexafluorophosphate) with PEG₁₀₀₀ has been successfully applied and found to be efficient for Suzuki cross-coupling and Heck reaction using Pd(OAc)₂ as the catalyst under air atmosphere. Moreover, the isolation of the products is readily performed by the extraction of diethyl ether, and the catalytic system reported here can be recycled and reused several times. Copyright © 2012 John Wiley & Sons, Ltd.

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Keywords: complex; [BMIm] PF₆; PEG₁₀₀₀; Suzuki cross-coupling; Heck reaction

Introduction

In the past years, C&bond;C cross-coupling catalyzed by expensive transition metal palladium complex along with ligands in organic solvents was applied widely in the pharmaceutical and fine chemical industries.^[1–3] Unfortunately, the problem of homogeneous catalysis is the difficulty in separating the costly catalyst from the reaction mixture and the impossibility of reusing it in consecutive reactions. The development of heterogeneous catalysts would be an attractive solution to this problem owing to their easy separation and facile recycling.^[4–8] However, most of the heterogeneous catalysts exhibit lower activity than their homogeneous counterparts. Considerable research efforts have been devoted to the development of efficient catalytic systems possessing the respective advantages of homogeneous and heterogeneous catalysis. Ionic liquids (ILs) have paved the way for alternative and new synthetic strategies, because this methodology allows not only high reaction activity but also the possibility of recycling the catalyst.^[9-13] Concerning C&bond;C cross-coupling reactions carried out in ionic liquids consisting of 1,3-dialkylimidazolium cations, some studies have already been reported.^[14-16] Regrettably, however, these processes suffer from some limitations: (i) the need for the use of toxic, expensive and air-sensitive phosphine; ^[17–19] and (ii) performing abnormal methodology, such as ultrasound or microwave.^[20-22] These limits are of significant concern in large-scale organic syntheses. Moreover, this reaction system requires a large amount of expensive ionic liquid. It would be desirable to reduce the amount of ionic liquid phase from an economic and toxicological viewpoint.^[23-25] As a result, the search for new readily available IL catalytic systems addressing these drawbacks remains an impending goal.

Polyethylene glycols (PEGs), as inexpensive, thermally stable, recoverable, and non-toxic media for catalysts, have been attracting increasing interest.^[26,27] Recently, PEGs were chosen to form complexes with metal cations, organocatalyst or ILs for some reactions such as asymmetric Michael addition reactions.^[28] The

results showed that these complexes could have potential applications in catalysis, especially PEG-ILs. In this communication, we would like to present a complex of [BMIm] PF_6 with PEG_{1000} (Scheme 1) for C&bond;C cross-coupling reactions. To the best our knowledge, there is no report on the utility of this complex, including metal palladium-catalyzed coupling reaction. On the basis of pioneering work,^[29] we explored the catalytic role of the complex in the palladium-catalyzed Suzuki reaction and Heck reaction. To our delight, this system exhibited excellent catalytic activity and stability in the presence of $Pd(OAc)_2$. In addition, the desired products were obtained simply by extraction and the residual could be reused several times.

Results and Discussion

Using the complex of PEG_{1000} –[BMIm] PF_6 as the catalytic system in the presence of inexpensive and convenient $Pd(OAc)_2$, we screened a series of general Pd-catalyzed C&bond;C bondforming reactions with the aim of studying the activity and reusability of the system. Preliminary reactions were carried out respectively in [BMIm] PF_6 , PEG_{1000} and PEG_{1000} –[BMIm] PF_6 . These results demonstrated that the Suzuki reaction (Table 1, entries 10–11) are sluggish in the pure ionic liquid or PEG_{1000} under the same reaction condition. However, the use of PEG_{1000} –[BMIm] PF_6 as the reaction medium furnished the best yield of desired product in the coupling reaction.

In fact, PEG_{1000} plays a very important part in the coupling reaction. On the one hand, used as the solvent, PEG_{1000} promotes

^{*} Correspondence to: Ming Lu, College of Chemical Engineering, Nanjing University of Science and Technology, Nanjing, Jiangsu, China. E-mail: luming302@126.com

College of Chemical Engineering, Nanjing University of Science and Technology, Nanjing, Jiangsu, China



Scheme 1. Structure of PEG₁₀₀₀-[BMIm] PF₆

ligand-free Suzuki–Miyaura reactions through reduction of Pd(II) to Pd(0), obviating the need for phosphine reducing agents. On the other hand, an excess of PEG could play a positive role in stabilizing Pd(0). In addition, the far higher efficiency of PEG_{1000} –[BMIm] PF₆ system over [BMIm] PF₆ ionic liquid alone can probably be attributed in part to the properties of PEG_{1000}

as a phase-transfer catalyst. The use of palladium complexes along with N-heterocyclic carbene as catalysts for C&bond; C cross-coupling reactions is well known and the formation of Pd-carbene species from the 1,3-disubstituted imidazolium salts in situ under basic conditions has been demonstrated, which are speculated to have implications on the success of the catalytic reaction in this kind of IL. Firm evidence for the in situ formation of a mixed palladium complex with [BMIm] BF4 at room temperature had been reported by Mathews in studies of the Suzuki reaction in ILs. Therefore, it is believed that the PEG₁₀₀₀-[BMIm] PF₆ catalytic system could enhance the coupling rate and the yield of desired product.^[30-32] It is noteworthy that PEG₁₀₀₀-[BMIm] PF₆ is not simply a mixture but a complex formed by an ion-dipole interaction. Furthermore, the formation of 1:1 self-assembled complex provided the stable structure detected by Luo and his co-workers.^[29]

From Table 1 (entries 12–13), it is clear that the efficiency of the complex was influenced by the nature of the anion. For example, the Suzuki reaction in PEG_{1000} –[BMIm] Br or PEG_{1000} –[BMIm] BF₄ always afforded low conversion. Optimization studies were also

Table 1. Suzuki reaction of <i>p</i> -methoxybenzeneboronic acid with bromobenzene catalyzed by Pd(OAc) ₂ in given media ^a						
$H_3CO \longrightarrow B(OH)_2 + Br \longrightarrow Media$ $Pd(OAc)_2, Base \longrightarrow OCH_3$						
Entry	Media	Base	Temperature / °C	Conversion ^b / %	Time / h	Yield °/ %
1	PEG ₁₀₀₀ -[BMIm] PF ₆	NaOAc	80	60	3.0	50
2	PEG ₁₀₀₀ -[BMIm] PF ₆	КОН	80	75	3.0	70
3	PEG ₁₀₀₀ -[BMIm] PF ₆	K_2CO_3	80	92	3.0	85
4	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	80	100	3.0	96
5	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	RT	10	3.0	trace
6	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	60	85	3.0	81
7	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	110	94	3.0	90
8	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	80	78	3.0	73 ^d
9	PEG ₁₀₀₀ -[BMIm] PF ₆	Cs_2CO_3	80	96	3.0	92 ^e
10	[BMIm] PF ₆	Cs_2CO_3	80	40	3.0	36 ^f
11	PEG ₁₀₀₀	Cs_2CO_3	80	81	3.0	65 ^f
12	PEG ₁₀₀₀ -[BMIm]BF ₄	Cs_2CO_3	80	88	3.0	80
13	PEG ₁₀₀₀ -[BMIm]Br	Cs ₂ CO ₃	80	60	3.0	55

^aReaction condition: 1.0 mmol of bromobenzene, 1.2 mmol of p-methoxybenzeneboronic acid, 2.0 mmol of base, 2.0 g of media, 5 mmol% of Pd (OAc)₂.

^b Determined by GC analysis, therein biphenyl as an internal standard.

^c Isolated yield.

^d 10 mmol% of Pd(OAc)₂.

e 30 mmol% of Pd(OAc)₂.

 $^{\rm f}$ Cross-coupling reaction in [BMIm] PF₆ or PEG₁₀₀₀ were carried out under the same condition.

performed to determine how bases, temperature and the amount of Pd(OAc)₂ affect the coupling reaction. As demonstrated in Table 1, the best yield was not obtained until the Suzuki reaction was carried out at 80 °C in the PEG₁₀₀₀–[BMIm] PF₆ system using Cs₂CO₃ as base and 5 mmol% Pd(OAc)₂.

With the best conditions to hand, we determined to explore the scope of Suzuki cross-coupling in PEG_{1000} -[BMIm] PF_6 with various substrates, including aryl bromides and chlorides with electron-withdrawing and electron-donating substituents. The results, given in Table 2, indicate that PEG_{1000} -[BMIm] PF_6 in the presence of palladium acetate without any ligands is among themost efficient systems for C&bond;C cross-coupling ever reported. As expected, aryl iodides and bromides give comparatively higher yields than chlorides, and there is a beneficial influence of the presence of electron-withdrawing substituents on the reaction product yield. These features are in agreement with the general behavior of Pd catalysts.

To extend the application scope of the catalytic system, we carried out another classic palladium-catalyzed C&bond;C crosscoupling, namely the Heck reaction of aryl halides with terminal olefins. Initially, we explored the conditions for the Heck reaction by using iodobenzene and methyl acrylate (Table 3, entry 4) as reactants in the Pd(OAc)₂–PEG₁₀₀₀-[BMIm] PF₆ catalytic system. The results demonstrated that Et₃N as the base was the best choice for the cross-coupling reaction and other bases such as KOH, Na₂CO₃ and K₂CO₃ were less effective. Many aryl iodides including electron-poor and electron-rich substrates could react with terminal olefins within a much shorter time (approximately 2-3h)^[8,20,33,34] and the yields of corresponding coupling products were very satisfactory (Table 3, entries 1–9). It was worth noting that the reaction can be conducted under mild conditions (60 °C) without the use of higher temperature or microware previously reported. Encouraged by these results, we tried to study the reaction activity between substituted aryl bromides and terminal olefins. Compared to aryl iodides, it required a longer reaction time and a higher amount of palladium acetate to complete the Heck reaction. Furthermore, the results in Table 3 show that the electronic nature of the aryl bromides have a clear effect on the coupling reactions. For example, the coupling reaction of electron-rich 4-methyl-bromobenzene with methyl acrylate provided 64% isolated yield (Table 3, entry 12), but for electron-deficient 4-nitro-bromobenzene the desired coupled product was obtained in 82% isolated yield (Table 3, entry 10).

Having established the scope of the new reaction system, our attention was focused on the recycling of the system. To demonstrate this issue, recycling experiments were conducted for the Suzuki cross-coupling reaction of bromobenzene with p-methoxybenzeneboronic acid, and for the Heck reaction of iodobenzene and methyl acrylate. After the product was isolated

Table 2	2. Suzuki	coupling reaction of v	various aryl halides w	vith <i>p</i> -methoxybenzeneboronic aci	d by Pd(OAc) ₂ in PEG ₁₀₀₀	-[BMIm] PF ₆ ^a
		ОСН ₃ + B(OH) ₂ +	X	$\frac{PEG_{1000}\text{-}[BMIm]PF_6}{Pd(OAc)_2,Cs_2CO_3}$	R	-OCH ₃
	Entry	Х	R	Conversion $^{\rm b}$ / %	Time / h	Yield %
	1	Ι	Н	100	2.0	98
	2	Ι	4-CH ₃	100	2.0	97
	3	Ι	4-NO ₂	100	2.0	98
	4	Br	4-OCH ₃	99	3.0	95
	5	Br	4-CH ₃	100	3.0	95
	6	Br	4-NO ₂	100	3.0	96
	7	Cl	Н	60	8.0	55 ^d
	8	Cl	2-NO ₂	80	8.0	70 ^d
	9	Cl	4-NO ₂	82	8.0	75 ^d
	10	Cl	2,4-NO ₂	87	8.0	80 ^d

^a Reaction condition: 1.0 m mol of ArX, 1.2 mmol of p-methoxybenzeneboronic acid, $2.0 \text{ mmol of } Cs_2CO_3$, 2.0 g of

 $PEG_{1000}\mbox{-}[BMIm]\ PF_6, 5\ mmol\%\ of\ Pd(OAc)_2\,,\,80\ ^oC\ (external).$

^b Determined by GC analysis, therein biphenyl as an internal standard.

^c Isolated yield.

 d 30 mmol% of Pd(OAc)₂.

able 3. Heck coupling reaction with various aryl halides with terminal olefins by $Pd(OAc)_2$ in PEG_{1000} -[BMIm] PF_6^{a}								
	 	R ₂ PEG ₁₀	₀₀₀ -[BMIm] PF ₆					
	$R_1^{/1}$	Pd(C	DAc) ₂ , Et ₃ N	$R_1 \sim R_2$				
Entry	R1	R2	Х	Time / h	Yield °/%			
1	4-NO ₂	COOMe	Ι	2	99			
2	4-NO ₂	COOEt	Ι	2	99			
3	4-NO ₂	COOn-Bu	Ι	2	99			
4	Н	COOMe	Ι	2	98			
5	Н	COOEt	Ι	2	97			
6	Н	COOn-Bu	Ι	2	97			
7	4-CH ₃	COOMe	Ι	3	93			
8	4-CH ₃	COOEt	Ι	3	91			
9	4-CH ₃	COOn-Bu	Ι	3	90			
10	4-NO ₂	COOMe	Br	6	82 ^b			
11	Н	COOEt	Br	10	70 ^b			
12	4-CH ₃	COOn-Bu	Br	24	64 ^b			

^a Reaction condition: 1.0 m mol of ArX, 2.0 mmol of terminal olefins, 2.0 mmol of Et_3N , 2.0 g of PEG-[BMIm] PF₆, 5 mmol%

of Pd(OAc)₂, 60 °C (external).

^b 20 mmol% of Pd(OAc)₂, 110 °C(external).

^c Isolated yield.



Figure 1. Recycling of $Pd(OAc)_2$ -PEG–[BMIm] PF₆ catalytic system. -•-, GC yield of Suzuki reaction between bromobenzene with *p*-methoxybenzeneboronic acid in every cycle; -**u**-, GC yield of Heck reaction between iodobenzene and methyl acrylate in every cycle

from the reaction mixture by ether (4 × 15 ml), the solidified Pd $(OAc)_2$ -PEG₁₀₀₀-[BMIm] PF₆ was subjected to a second run by charging with the same substrates without any regeneration or addition of Pd(OAc)₂. As demonstrated in Fig. 1, the reusability of Pd(OAc)₂-PEG₁₀₀₀-[BMIm] PF₆ for the Suzuki reaction would

be decreased rapidly from the fifth run. With respect to recycling in successive batch operations of the Heck reaction, the results shown in Figure 1 show that catalytic activity was maintained very well even after six cycles.

In summary, we have presented an active, air-stable and recyclable approach for ligand-free Suzuki and Heck coupling reactions using inexpensive $Pd(OAc)_2$ as the palladium catalyst in PEG_{1000} –[BMIm] PF_6 media. For the Suzuki reaction, a number of aryl halides, including aryl bromides and chlorides, were coupled to arylboronic acids smoothly and efficiently. Furthermore, the present $Pd(OAc)_2$ – PEG_{1000} –[BMIm] PF_6 system were also desired for the Heck reaction of aryl iodides and aryl bromides with terminal olefins. Other excellent properties are simple separation of wanted products and outstanding recyclability of the catalytic system.

Experimental

General Information

All chemicals were from commercial sources without any pretreatment. All reagents were of analytical grade. ¹H NMR spectra were recorded on a Bruker 500 MHz spectrometer with tetramethylsilane (TMS) as an internal standard. Melting points were recorded on a Buchi R-535 apparatus and are uncorrected. GC analysis was performed on an Agilent GC-6820 gas

chromatograph equipped with a $30\,m\times0.32\,mm\times0.5\,\mu m$ HP-Innowax capillary column and a flame ionization detector. GC-MS analyses were performed on a Saturn 2000 GC-MS instrument.

Preparation of PEG₁₀₀₀-[BMIm] PF₆

[BMIm] PF₆ (10.0 mmol) was added to a 100 ml methanol containing PEG₁₀₀₀ (10.0 mmol) and stirred at room temperature for 10 min. The solution was concentrated under vacuum to give the desired product.

General Procedure for Suzuki Cross-Coupling Reaction

Under air atmosphere, a flask was charged with aryl halides (1.0 mmol), *p*-methoxybenzeneboronic acid (1.2 mmol), Cs_2CO_3 (2.0 mmol), Pd(OAc)₂ (5 mmol%) and PEG₁₀₀₀–[BMIm] PF₆ (2.0 g). The mixture was heated to 80 °C for the indicated time. After the reaction solution was cooled to room temperature, the mixture was extracted four times with ether (4 × 15 ml). The combined ether phase was analyzed by GC-MS and then concentrated. Further purification of the product was achieved by flash chromatography on a silica gel column.

General Procedure for Heck Reaction

Typical procedure for Heck reaction: under air atmosphere, a flask was charged with aryl halides (1.0 mmol), terminal olefins (2.0 mmol), Et₃N (2.0 mmol), PEG₁₀₀₀–[BMIm] PF₆ (2.0 g) and Pd (OAc)₂ (5 mmol%). The mixture was heated to 60 °C for the indicated time and progress of the reaction was monitored by TLC. After the reaction solution was cooled to room temperature, the mixture was extracted four times with ether (4 × 15 ml). The combined ether phase was analyzed by GC or HPLC and then concentrated. Further purification of the product was achieved by flash chromatography on a silica gel column. All products are known compounds and the identity of some represent compounds was confirmed by comparison with literature spectroscopic data.

4-Methoxylbiphenyl

 $R_{\rm f}$ = 0.5 (petroleum ether). Chromatography solvent petroleum ether. ¹HNMR (500 MHz, DMSO): σ = 7.60–7.85 (m, 4H), 7.41–7.45 (m, 2H), 7.23–7.32 (s, 1H), 6.98–7.00 (m, 2H), 3.5 (s, 3H).

(E)-Methyl cinnamate

 $R_{\rm f}$ = 0.4 (petroleum ether/ethyl acetate = 100:1, v/v). Chromatography solvent petroleum ether/ethyl acetate = 150/1, v/v). ¹ H NMR (500 MHz, CDCl₃): σ = 7.73(d, *J* = 16.0 Hz, 1H), 7.28–7.55(m, 5H), 6.45 (d, *J* = 16.0 Hz, 1H), 3.83 (s, 3H).

(E)-Ethyl cinnamate

 $R_{\rm f}$ = 0.5 (petroleum ether/ethyl acetate = 100:1, v/v). Chromatography solvent petroleum ether/ethyl acetate = 150/1, v/v). ¹H NMR (500 MHz, CDCl₃): σ = 7.72 (d, *J* = 16.1 Hz, 1H), 7.379–7.55 (m, 5H), 6.44–6.47 (m, 1H), 4.26–4.30 (m, 2H) 1.34–1.37 (m, 3H).

(E)-Methyl 3-(4-nitrophenyl) acrylate

 $R_{\rm f}$ = 0.4 (petroleum ether/ethyl acetate = 20:1, v/v). Chromatography solvent petroleum ether/ethyl acetate = 50/1, v/v). ¹H NMR (500 MHz, CDCl₃): σ = 8.23–8.25 (m, 2H), 7.66–7.73 (m, 3H), 6.55 (d, J = 16.1 Hz, 1H), 3.83 (s, 3H).

(E)-Ethyl 3-(4-nitrophenyl)acrylate

 $R_{\rm f}$ =0.4 (petroleum ether/ethyl acetate = 20:1, v/v). Chromatography solvent petroleum ether/ethyl acetate = 50/1, v/v). M.p. 134.9–136.1 °C; ¹H NMR (500 MHz, DMSO): σ = 8.29–8.32 (m, 2H), 7.24–8.16 (m, 3H), 6.95 (d, *J* = 16.0 Hz, 1H), 4.25–4.30 (m, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).

Supporting Information

Supporting information may be found in the online version of this article.

References

- [1] L. X. Yin, J. Liebscher, Chem. Rev. 2007, 107, 133.
- [2] G. Bringmann, S. Rudenauer, T. Bruhn, L. Benson, R. Brun, Tetrahedron 2008, 64, 5563.
- [3] Y. D. Wang, M. Dutia, M. B. Floyd, A. S. Prashad, D. Berger, M. Lin, *Tetrahedron* 2009, 65, 57.
- [4] J. C. Park, E. J. Heo, A. Kim, M. Kim, K. H. Park, H. Song, J. Phys. Chem. C 2011, 115, 15772.
- [5] K. Karami, C. Rizzoli, M. M. Salah, J. Organomet. Chem. 2011, 696, 940.
- [6] Y. He, C. Cai, *Catal. Commun.* **2011**, *12*, 678.
- [7] H. L. Qiu, S. M. Sarkar, D. H. Lee, M. J. Jin, Green Chem. 2008, 10, 37.
- [8] X. Y. Shi, X. Y. Han, W. J. Ma, J. Fan, J. F. Wei, Appl. Organometal. Chem. 2012, 26, 16.
- [9] H. Hagiwara, K. Sato, T. Hoshi, T. Suzuki, Synlett 2011, 2545.
- [10] L. Lin, Y. C. Li, S. B. Zhang, S. H. Li, Synlett 2011, 1779
- [11] J. Dupont, R. F. D. Souza, P. A. Z. Suarez, Chem. Rev. 2002, 102, 3667.
- [12] R. Sheldon, Chem. Commun. 2001, 2399.
- [13] P. Wasserscheid, W. Keim, Angew. Chem. Int. Ed. 2000, 39, 3772.
- [14] F. McLachlan, C. J. Mathewas, P. J. Smith, T. Welton, *Organometallics* 2003, 22, 5350.
- [15] J. C. Xiao, J. M. Shreeve, J. Org. Chem. 2005, 70, 3072.
- [16] J. Carmichael, M. J. Earle, J. D. Holbrey, P. B. McCormac, K. R. Seddon, Org. Lett. **1999**, 1, 997.
- [17] J. Mathews, P. J. Smith, T. Welton, Chem. Commun. 2000, 1249.
- [18] H. Yang, C. C. Tai, Y. T. Huang, I. W. Sun, Tetrahedron 2005, 61, 4857.
- [19] M. M. Manas, R. Pleixats, A. S. Muns, Synlett 2006, 3001.
- [20] Z. Y. Du, W. W. Zhou, L. Bai, F. Wang, J. X. Wang, Synlett 2011, 369.
- [21] K. S. A. Vallin, P. Emilsson, M. Larhed, A. Hallberg, J. Org. Chem. 2002, 67, 6243.
- [22] R. Hajipour, K. Karami, F. Rafiee, Appl. Organometal. Chem. 2012, 26, 27.
- [23] Taher, J. B. Kim, J. Y. Jung, W. S. Ahn, M. J. Jin, Synlett 2009, 2477.
- [24] R. H. Wang, B. Twamley, J. M. Shreeve, J. Org. Chem. 2006, 71, 426.
- [25] J. D. Revell, A. Ganesan, Org. Lett. 2002, 4, 3071.
- [26] J. M. Harris, Polyethylene Gycol Chemistry: Biotechnological and Biomedical Applications, Plenum Press, New York, 1992.
- [27] G. K. Datta, H. V. Schenck, A. Hallberg, M. Larhed, J. Org. Chem. 2006, 71, 3896.
- [28] D. Q. Xu, S. P. Luo, Y. F. Wang, A. B. Xia, H. D. Yue, L. P. Wang, Z. Y. Xu, Chem. Commun. 2007, 4393.
- [29] S. P. Luo, S. Zhang, Y. F. Wang, A. B. Xia, G. C. Zhang, X. H. Du, D. Q. Xu, J. Org. Chem. 2010, 75, 1888.
- [30] W. A. Herrmann, Angew. Chem. Int. Ed. 2002, 41, 1290.
- [31] W. A. Herrmann, C. P. Reisinger, M. Spiegler, J. Organomet. Chem. 1998, 557, 93.
- [32] M. Zhang, J. K. Huang, M. L. Trudell, S. P. Nolan, J. Org. Chem. 1999, 64, 3804.
- [33] J. H. Li, X. C. Hu, Y. Liang, Y. X. Xie, Tetrahedron 2006, 62, 31.
- [34] G. P. Zhang, H. H. Zhou, J. Q. Hu, M. Liu, Y. F. Kuang, Green Chem. 2009, 11, 1428.