

An efficient glucose-based ligand for Heck and Suzuki coupling reactions in aqueous media

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Received: 2 January 2013 / Accepted: 17 July 2013
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Abstract The glucose-based ligand, *N*-salicylidene-*D*-glucosamine (Sal-*D*-glsmN), was readily obtained by reaction of salicylaldehyde (Hsal) with the *D*-glucosamine hydrochloride. Ligand Sal-*D*-glsmN was found to be an efficient ligand in the palladium-catalyzed Suzuki and Heck C–C coupling reactions in aqueous medium under aerobic condition. It was found that the use of Sal-*D*-glsmN/Pd(OAc)₂ system as a catalyst, aryl halides undergo Suzuki and Heck cross-couplings, respectively, with arylboronic acids and olefins to give the desired products in moderate to excellent yields.

Keywords Homogeneous catalysis ·
C–C coupling · Heck reaction · Suzuki reaction

Introduction

In recent years, replacing conventional reagents with environment friendly reagents have vigorously been studied from the standpoint of green chemistry and water is preferred in studies as an attractive solvent for chemical

reactions due to low cost, non-flammability, non-toxicity, and environmental hazards [1–3].

Palladium-catalyzed cross-coupling reactions such as Suzuki and Heck have been demonstrated to be one of the most widely used standard tools for the construction of carbon–carbon bonds [4–8]. In general, improvement of palladium-catalyzed reactions is greatly based on the reactivity of the palladium catalyst using efficient supporting ligands [9–14]. Therefore, during the past decades many efforts have been made to find most efficient ligands, in which phosphine-based ligands are used as the most common ligands for Suzuki and Heck coupling reactions [15–17]. However, most of the phosphine-based ligands are water and/or air sensitive. Therefore, it is highly desirable to design the water soluble, efficient, and phosphine-free ligands to dispense the use of expensive and water and/or air-sensitive phosphines for palladium-catalyzed coupling reactions.

Sugars as the most naturally abundant organic molecules with many hydroxyl groups, which tend to increase the solubility of these molecules in water, are suitable resources for synthesis of water soluble ligand with base of sugar [18]. Furthermore, the structural diversity of sugar and the high density of functional groups offer a wide variety of opportunities for successful utilization of sugars in many technically important fields such as asymmetric induction in a reaction using sugar derivatives as chiral auxiliaries [19–21]. The very cheap, especially high environmental friendly nature of sugar molecules has driven us to explore their capabilities as ligands for Heck and Suzuki coupling reactions in aqueous medium.

To develop a general catalyst for palladium-catalyzed cross-coupling reactions, we decided to apply a green system Pd(OAc)₂/Glucose-based ligand for the preparation of polysubstituted alkenes by Suzuki cross-couplings of

Electronic supplementary material The online version of this article (doi:10.1007/s13738-013-0316-3) contains supplementary material, which is available to authorized users.

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aryl halides with arylboronic acids and Heck reaction of olefins with aryl halides.

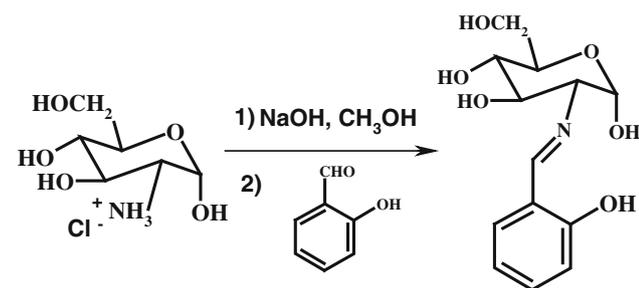
Results and discussion

The synthetic path for the glucose-based ligand, *N*-salicylidene-*D*-glucosamine (Sal-*D*-glsmN) is depicted in Scheme 1. Ligand Sal-*D*-glsmN was obtained by deprotonation of glucosamine hydrochloride with NaOH in MeOH following the addition of salicylaldehyde and stirring the reaction mixture at 35 °C for 2 h [22].

The next investigation is to study the catalytic performance of the synthesized glucose-based ligand in the palladium-catalyzed Heck reaction in aqueous medium. We chose the palladium-catalyzed Heck coupling of iodobenzene with *n*-butyl acrylate as the test reaction for optimization of the reaction conditions. A wide variety of solvents has been used for Pd-catalyzed Heck reactions and the selection of solvents with low toxicity is of primary interest. Water alone or in association with *i*PrOH or DMF represents the most economical and safety-conscious solvent combination [23, 24]. As shown in Fig. 1, (2:1) mixture of H₂O/DMF or H₂O/*i*PrOH was found to be the best of choice in presence of KOH as base. It is well known that the addition of a base can improve the reaction efficiency for some catalytic system. Subsequently, the reaction was investigated in the presence of different bases in (2:1) mixture of H₂O/DMF as solvent. Interestingly, it was found that some inorganic bases, such as K₂CO₃ and KOH, promoted the Heck coupling reaction, giving excellent yields. On the other hand, NEt₃ as an organic base had little effect on the reaction.

To broaden the catalytic applicability of the Sal-*D*-glsmN ligand, we subsequently examined Heck coupling reactions of iodobenzene and bromobenzene with various olefins (Table 1).

The results indicated that the combination of Pd(OAc)₂ and the Sal-*D*-glsmN was efficient for the Heck coupling reaction of a series of alkenes and aryl halides. The



Scheme 1 The synthetic path of glucose-based ligand *N*-salicylidene-*D*-glucosamine

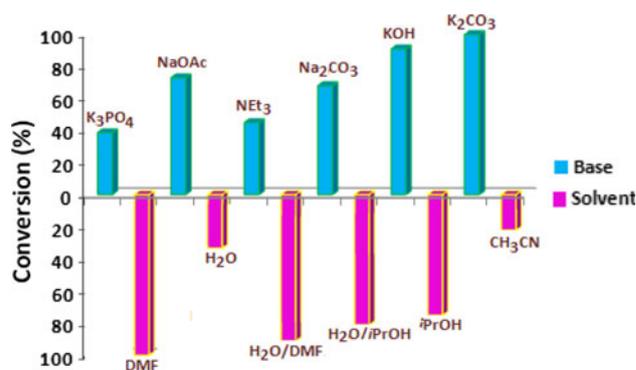


Fig. 1 The effect of base and solvent on the Heck reaction. Reaction conditions: 1.0 mmol of iodobenzene, 1.2 mmol of *n*-butyl acrylate, 2 mmol of bases, 1.0 mol % Pd(OAc)₂, (2 mol %) ligand Sal-*D*-glsmN, 80 °C, 3-mL solvent, 2 h

electronic nature of substituents bearing olefins is initially investigated. Various olefins bearing either electron-donating groups or electron-withdrawing groups such as methoxy, methyl and cyano groups, could be efficiently converted to the corresponding coupling products in good to high yields. A mixture of the two stereoisomers (*Z*) and (*E*) was obtained for the reaction of acrylonitrile (Table 1, entry 5). It is well known that electron-poor aryl halides undergo Heck reactions more easily than electron-rich substrates and this general trend has been observed for both aryl bromides and chlorides (Table 1, entries 11–18). It is worth noting that the most electron-poor aryl bromide, 4-acetylbromobenzene, showed a good reactivity and was successfully coupled with styrene and *n*-butylacrylate in good yields (Table 1, entries 11, 12). However, the Heck coupling of chlorobenzenes was difficult to proceed under the same reaction conditions and trace amounts of coupling product was observed (Table 1, entries 17–19).

In general, coupling of aryl iodides and bromides with olefins was found to give products in good to excellent yields (47–97 %). The reduced yields of chlorobenzenes are mostly a consequence of non-reactive nature of this substrate.

It is clear that ligands with coordination to palladium during the catalytic process of the Heck reaction could improve the stability of palladium complexes and, therefore, the catalytic activity induced by ligands is indeed better than ligandless conditions [25]. To study the coordination of ligand Sal-*D*-glsmN to palladium and complex formation during the catalytic process, the electronic spectra of Pd(OAc)₂, ligand Sal-*D*-glsmN and Pd(OAc)₂/Sal-*D*-glsmN ligand mixture were recorded in H₂O/DMF (1:1 v/v) solution and are presented in Fig. 2. It was found that after addition of 1 equiv. of Pd(OAc)₂ to the solution of ligand Sal-*D*-glsmN, the band at 317 nm of ligand slightly shifted to longer wavelength along with decreasing in its intensity. Conversely, the band at $\lambda_{\text{max}} = 388$ nm

Table 1 Heck reaction between aryl halides and olefins

Entry	ArX	Olefin	Yield (%) ^a (TON, TOF(h ⁻¹)) ^b	
			H ₂ O/DMF	H ₂ O/ <i>i</i> PrOH
1			88 (88, 44)	66 (66, 33)
2			97 (97, 48.5)	81 (81, 40.5)
3			96 (96, 48)	73 (73, 36.5)
4			95 (95, 47.5)	67 (67, 33.5)
5			97 nd (97, 48.5)	72 (72, 36)
6			85 (85, 42.5)	70 (70, 35)
7			83 (83, 41.5)	65 (65, 32.5)
8			79 (79, 39.5)	56 (56, 28)
9			73 (73, 36.5)	54 (54, 27)
10			72 (72, 36)	54 (54, 27)
11			70 (70, 35)	63 (63, 31.5)
12			76 (76, 38)	61 (61, 30.5)
13			61 (61, 30.5)	52 (51, 25.5)
14			59 (59, 29.5)	50 (50, 25)
15			55 (55, 27.5)	47 (47, 23.5)
16			64 (64, 32)	51 (51, 25.5)
17			27 (27, 13.5)	trace
18			30 (30, 15)	trace
19			13 (13, 6.5)	trace

Reaction conditions: 1.0 mmol of aryl halide, 1.2 mmol of olefins, 2 mmol of K₂CO₃, 1.0 mol % Pd(OAc)₂, (2.0 mol %) ligand Sal-D-glsmN, 80 °C, 3-mL solvent, 2 h

Isolated yield

^a TON = (mmol of product)/mmol of catalyst, TOF = TON/reaction time

^b Ratio (*Z*:*E*) is 23:77

exhibits a distinct hypsochromic shift followed by gradual increase in this band intensity. On the basis of spectral results, one could assume the formation of new species of type Pd-Sal-D-glsmN as a catalytic complex involved in the reaction.

In order to show the merit and efficiency of the present catalytic system in comparison with recently reported protocols, we compared the results of the stilbene synthesis from iodobenzene and styrene in the presence of palladium catalysts. As shown in Table 2, our catalytic system is superior to some of the previously reported catalysts in terms of reaction conditions. In contrast to similar previously reported systems, the catalytic system presented in this paper does not suffer from the harsh reaction conditions, such as high reaction temperature (Table 2, entries 2–4 and 6), long reaction time (Table 2, entries 2–4, 6, 7, 10, 11, and 12), and using large amounts of hazardous solvents (Table 2, entries 2–6). In addition, the attractive features of this catalytic system, such as low cost, and ease of use, make it particularly suitable for the coupling reactions.

The operationally simple, air-stable, and water soluble nature of the catalytic system Pd(OAc)₂/Sal-D-glsmN inspired us to investigate its scope in the Suzuki reaction. It is known that the nature of the solvent and base is an important factor for determining the efficiency of the Suzuki cross-coupling reaction. Therefore, the influence of various solvents and bases was firstly investigated for the Pd(OAc)₂ catalyzed Suzuki coupling reaction of iodobenzene with phenylboronic acid in presence of Sal-D-glsmN ligand. The results in Fig. 3 show that (3:1) mixture of H₂O/*i*PrOH performed most efficiently as solvent system among the various solvents screened, whereas KOH is the

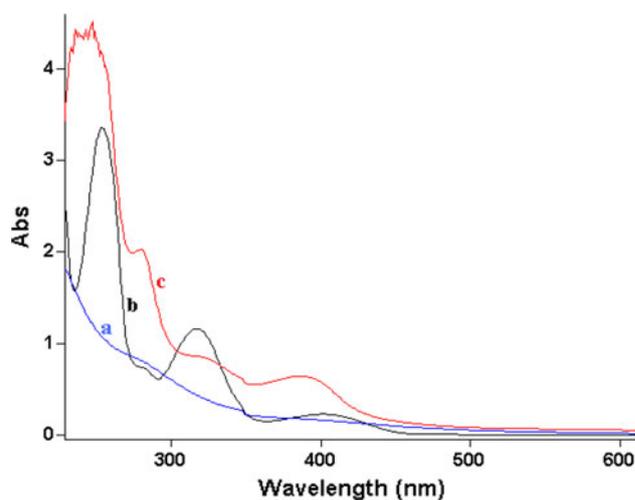
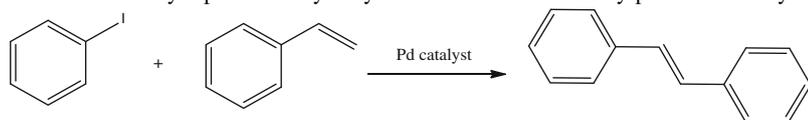


Fig. 2 UV-vis spectra of **a** 0.02-M solution of Pd(OAc)₂, **b** ligand Sal-D-glsmN, **c** Pd(OAc)₂/ligand Sal-D-glsmN mixture in H₂O/DMF (1:1 v/v)

Table 2 Recently reported catalytic systems for Heck reaction by palladium catalysts

Entry	Catalyst	Conditions	Yield (%)	TON	Ref.
1	Pd(OAc) ₂ -Sal-D-glsmN	1 mol % catalyst/K ₂ CO ₃ /H ₂ O:DMF(2:1)/2 h/80 °C	88	88	Present work
2	N,N',N'',O-tetradentate Pd(II)	0.001 mol % catalyst/LiOH/DMF/15 h/140 °C	95	95,000	[26]
3	<i>Trans</i> -[PdCl ₂ (PI) ₂]	0.001 mol % catalyst/K ₂ CO ₃ /DMF/24 h/140 °C	92	92,000	[27]
4	<i>Trans</i> -[PdBr ₂ (MPI) ₂]	0.001 mol % catalyst/K ₂ CO ₃ /DMF/24 h/140 °C	98	98,000	[27]
5	Aminophosphine-pd(II)	1 mol % catalyst/K ₂ CO ₃ /DMF/30 min/100 °C	75.2	75	[28]
6	Oxazine-Pd(II)	0.004 mol % catalyst/NEt ₃ /DMAc/8 h/120 °C	94	23,500	[29]
7	Kraft lignin Pd NPs	0.002 mol % catalyst/K ₂ CO ₃ /H ₂ O/8 h/100 °C	100	45,000	[30]
8	Poly(<i>N</i> -vinyl-2-pyrrolidone) Pd NPs	7.5 mol % catalyst/K ₂ CO ₃ /H ₂ O/1 h/60 °C	98	13	[31]
9	Pd NPs-nSTDP	0.01 mol % catalyst/K ₂ CO ₃ /H ₂ O:DMF(1:3)/9 h/85 °C	95	9,500	[32]
10	PNP-SSS	1.2 mol % catalyst/K ₂ CO ₃ /H ₂ O/1.5 h/100 °C	95	79	[33]
11	Pd/Cu (4:1) NPs	0.025 mol % catalyst/NEt ₃ /MeOH/18 h/reflux	91	5,921	[34]
12	Pd/Cu (5:1) NPs	0.025 mol % catalyst/NEt ₃ /MeOH/18 h/reflux	82	5,335	[34]
13	Diazoketiminato-pd(II)	0.03 mol % catalyst/K ₂ CO ₃ /MeOH/4 h/reflux	95	3,333	[35]

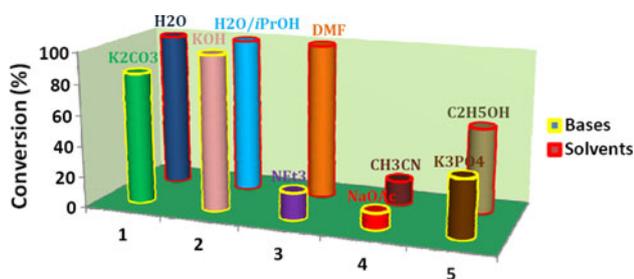


Fig. 3 The effect of base and solvent on the Suzuki reaction. Reaction conditions: 1.0 mmol of iodobenzene, 1.2 mmol of phenylboronic acid, 2 mmol of bases, 0.5 mol % Pd(OAc)₂, (1 mol %) ligand Sal-D-glsmN, 85 °C, 3-mL solvent

best choice as compared to the other bases in (3:1) mixture of H₂O/*i*PrOH as solvent. The activity towards cross-coupled product biphenyl was decreased when KOH was replaced with K₂CO₃, NEt₃, NaOAc, and K₃PO₄.

To evaluate the catalytic performance of the Sal-D-glsmN ligand, a kinetic study of the Suzuki reaction between iodobenzene and phenylboronic acid was performed in the presence of Pd(OAc)₂ and KOH at 85 °C in (3:1) mixture of H₂O/*i*PrOH.

Figure 4 demonstrates the kinetic curves of the reaction time vs. the isolated yield using in situ-generated catalysts from Pd(OAc)₂.

The catalytic applicability of the Sal-D-glsmN ligand was explored with a range of aryl halides and arylboronic acids and the results are summarized in Table 3.

As shown in Table 3, the Suzuki coupling of various phenylboronic acids with aryl iodide and bromide

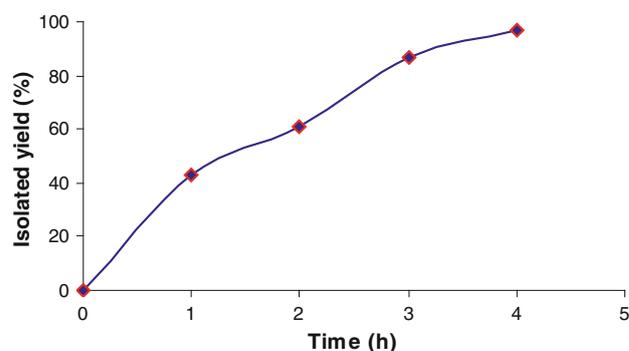
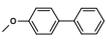
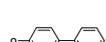
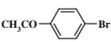
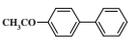


Fig. 4 Kinetic profile of Pd(OAc)₂/Sal-D-glsmN catalyzed Suzuki reaction in (2:1) mixture of H₂O/*i*PrOH. Reaction conditions: 1.0 mmol of iodobenzene, 1.2 mmol of phenylboronic acid, 2 mmol of KOH, 0.5 mol % Pd(OAc)₂, 1 mol % ligand Sal-D-glsmN, 85 °C, 3 mL (3:1) mixture of H₂O/*i*PrOH

provided good to excellent yield of the products. The reaction between iodobenzene and phenylboronic acid was quantitative and faster with the use of 0.5 mol % of Pd(OAc)₂ in (3:1) mixture of H₂O/*i*PrOH. High catalytic activity was observed in the coupling of phenylboronic acid and 4-methoxyphenylboronic acid with both aryl iodide and bromide. 2-methoxyphenylboronic acid was less reactive in comparison to phenylboronic acid and 4-methoxy phenylboronic acid, indicating that the steric affect of the substrates play a significant role in this system (Table 3, entries 1 and 5). In addition, Pd(OAc)₂/Sal-D-glsmN system showed good activity for the Suzuki reaction of phenylboronic acids with aryl iodide and aryl bromide in pure water. It is worth noting that the most

Table 3 Suzuki reaction between aryl halides and aryl boronic acids

Entry	ArX	Olefin	Yield (%) ^a (TON) ^b	
			H ₂ O	H ₂ O/ <i>i</i> PrOH
1			96 (192)	98 (196)
2			51 (102)	69(138)
3			79 (158)	92 (184)
4			90 (180)	95 (190)
5			45 (90)	61(122)
6			81 (162)	86 (172)
7			96 (192)	98 (196)
8			25 (50)	16 (32)

Reaction conditions: 1.0 mmol of aryl halide, 1.2 mmol of arylboronic acid, 2 mmol of KOH, 0.5 mol % Pd(OAc)₂, (1.0 mol %) ligand Sal-D-glsmN, 85 °C, 4 h, 3-mL solvent

^a Isolated yield

^b TON = (mmol of product)/mmol of catalyst

electron-poor aryl bromide, 4-acetylbromobenzene, quantitatively gave the coupling product in both solvents H₂O/*i*PrOH and H₂O (Table 3, entry 7). Only poor yields were obtained with inactive aryl chloride as substrate (Table 3, entry 8).

Recently, Khalafi-Nezhad and coworkers [6, 33] reported the immobilizing of the palladium nanoparticles on silica-starch substrate (PNP-SSS) and its use for the Heck and Suzuki reactions in water medium. The catalytic results, including those for Heck and Suzuki couplings are comparable to those reported in this paper. The advantage of our catalytic system in comparison with that published by Khalafi-Nezhad is simple catalyst preparation. However, the method described here involves non-reusability of catalyst as a disadvantage.

Conclusion

In conclusion, we have developed an efficient and environmentally friendly water-soluble glucose-based ligand, which is readily accessible from inexpensive and commercial

materials. The in situ-generated catalysts from Pd(OAc)₂ and the Sal-D-glsmN ligand demonstrated excellent catalytic activities towards the Suzuki reaction of aryl halides and various phenylboronic acids while using pure water or H₂O/*i*PrOH as the green solvent. In addition, catalytic system Sal-D-glsmN/Pd(OAc)₂ was used in Heck coupling reactions of iodobenzene and bromobenzene with various olefins in aqueous medium. Under aerobic conditions, good to excellent yields of coupled product were obtained.

Experimental section

General remarks

All the reactions were carried out under air. Chemicals and solvents were purchased from the Fluka and Merck Chemical companies and used without purification. The elemental analysis (carbon, hydrogen, and nitrogen) of compounds were obtained from a Carlo ERBA Model EA 1108 analyzer. UV-Vis spectra were recorded by a CARY 100 Bio VARIAN UV-vis spectrophotometer. The ¹H and ¹³C NMR spectra were recorded on a Bruker FT-NMR 500 MHz spectrometer. Chemical shifts are reported in ppm relative to TMS. The reaction products of Heck reaction were determined and analyzed by a HP Agilent 6890 gas chromatograph equipped with a HP-5 capillary column (phenyl methyl siloxane 30 m · 320 μm · 0.25 μm) and a flame-ionization detector. All products were isolated by short chromatography on a silica gel 60 (0.063–0.20 mesh ASTM) column using hexane/ethyl acetate as eluent.

Synthesis of glucose-based ligand, *N*-salicylidene-D-glucosamine

Ligand *N*-salicylidene-D-glucosamine (Sal-D-glsmN) was prepared in a similar manner according to the reported procedure with some modification [10]. To a MeOH suspension (20 mL) of glucosamine hydrochloride (0.5 g, 2.32 mmol), finely grinded-solid NaOH (0.1 g, 2.55 mmol) was added in a single portion. The mixture was stirred at room temperature, and after 30 min NaCl was filtered off. Salicylaldehyde (2.55 mmol, 0.27 mL) was slowly added to the filtrate, and the reaction mixture was stirred for 2 h at 35 °C. The solution was then concentrated in vacuum to 5 mL and put into an ice bath, the yellow solid filtered off, washed with cooled methanol, and then dried under vacuum. Yield 420 mg(64 %). ¹H NMR(d₆-DMSO): δ 8.21 (t, 1 H), 7.22 (m, 1H,), 7.10 (m, 1H), 6.68 (m, 1H), 6.59 (m, 1H,), 3.10-3.83 (multiple peaks, 7H); elemental

analysis calcd for $C_{13}H_{17}NO_6$ (283.11): C 55.12, H 6.05, N 4.94; found C 55.05, H 5.97, N 5.01.

General procedure for the Suzuki reactions

All Suzuki reactions were carried out without an inert atmosphere. A mixture of aryl halide (1.0 mmol), arylboronic acid (1.2 mmol), KOH (2 mmol), $Pd(OAc)_2$ (0.5 mol %), and ligand Sal-D-glsmN (1 mol %) in H_2O (3 mL) or (3:1) mixture of $H_2O/iPrOH$ (3 mL) was allowed to react in a sealed tube at 85 °C. The reaction mixtures was added to brine (15 mL) and extracted three times with diethyl ether (3×15 mL). The combined organic phase was analyzed by GC. The further purification of the product was achieved by flash chromatography on a silica gel column using hexane/ethyl acetate (5:1).

General procedure for the Heck reactions

All Heck reactions were carried out under air. A mixture of aryl halide (1.0 mmol), olefins (1.2 mmol), K_2CO_3 (2 mmol), $Pd(OAc)_2$ (1 mol %), and ligand Sal-D-glsmN (2 mol %) in (2:1) mixture of $H_2O/iPrOH$ or H_2O/DMF (3 mL) was allowed to react in a sealed tube at 80 °C. The reaction mixtures were added to brine (15 mL) and extracted three times with diethyl ether (3×15 mL). The combined organic phase was analyzed by GC. The further purification of the product was achieved by flash chromatography on a silica gel column using hexane/ethyl acetate (5:1).

Acknowledgments Authors thank the Research Council of Sharif University of Technology and University of Maragheh for financial support of this work.

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