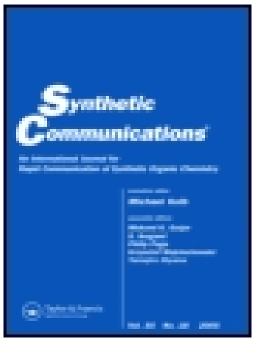

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Chemoselective formation of C–N bond in wet acetonitrile using amberlyst®-15(H) as a recyclable catalyst

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ABSTRACT
An economically efficient and environmentally benign protocol for the chemoselective one-pot synthesis of diversely N-substituted amides has been developed in good yield through the reaction of benzylic secondary alcohols as well as aliphatic tertiary alcohols and alkyl/aryl nitriles. Commercially available Amberlyst®-15(H) has been utilized at 80 °C as an air-stable and reusable heterogeneous inexpensive solid acid catalyst without any anhydrous and inert environment. The attractive features of the present synthetic protocol are mild reaction conditions, short reaction time, excellent chemoselectivity, high atom economy and tolerance of various sensitive moieties.

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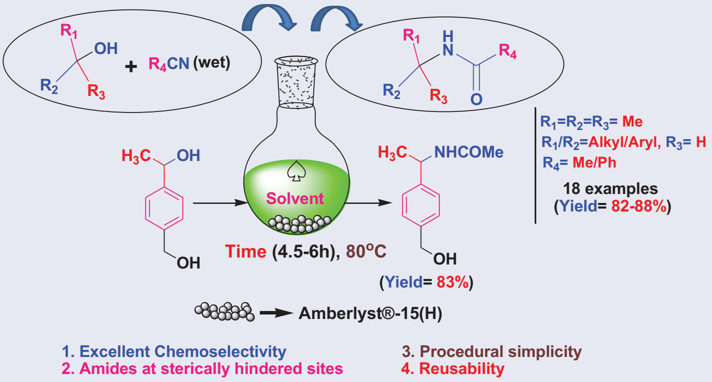
KEYWORDS
Alcohols; amides; chemoselectivity; heterogeneous catalysis; nitriles

GRAPHICAL ABSTRACT



Formation of C–N bond in aliphatic skeletons, especially from alkanols, is a formidable synthetic operation. Reaction of nitriles with substituted alkenes or alcohols using concentrated sulfuric acid in glacial acetic acid was classically used for this purpose through the formation of substituted amides. In order to avoid the involvement of such a strong mineral acid, several modifications have been proposed including other acidic substances like Tf_2O,[1] $BF_3.OEt_2$,[2] P_2O_5-SiO_2,[3] Bronsted acid,[4] Fe-montmorillonite K10,[5] Nafion-H,[6,7] Nafion-H along with microwave irradiation,[8] formic acid under reflux,[9]

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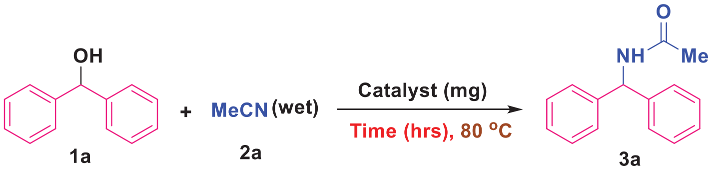


Scheme 1. Reaction of benzhydrol (**1a**) using different catalysts and reaction medium.

$HBF_4.Et_2O$,[10] 2,4-dinitrobenzenesulfonic acid,[11] TfOH,[12] $HClO_4$-functionalized silica-coated nanoparticles,[13] KI-TBHP[14] and liquid HF.[15] Ritter reaction using *tert*-butyl acetate instead of *tert*-butyl alcohol[16] was also reported to be catalyzed by metal-triflate[17] and pentafluorophenyl ammonium triflate (PFPAT),[18] Ca(II)-catalyzed reaction under microwave irradiation,[19] Molecular I_2,[20] Bi-salts,[21] $FeCl_3.6H_2O$[22] and ionic liquids[23] have also been employed for this kind of transformation. Recently a photochemical modification[24] has evolved as one of the successful alternative methods. However, most of these methods suffer from several disadvantages such as use of corrosive acid catalysts, toxic and moisture-sensitive reagents, use of expensive compounds and materials, elevated reaction temperatures, prolonged reaction time, susceptibility with acid-labile and bulky functional groups and concomitant formation of several by-products arising out of different side reactions including rearrangement. But the main drawback of most of the existing methods is the decomposition of the catalysts during aqueous work-up leading to tedious protocols for isolation, separation and recovery of the products.

Recyclable resins[25–28] bearing acidic sites offer the advantages not only due to their subtle catalytic attributes but also from the standpoint of reusability along with physical and chemical stability. Further advantages are associated with their heterogeneous nature in terms of their facile separation from the reaction mixture and easier isolation of the products. Keeping in mind the aforesaid attributes, we report herein an admirable catalytic application of Amberlyst®-15(H) for the formation of C–N bond in wet acetonitrile.

Results and discussion

To check the applicability of Amberlyst®-15(H) the reaction was carried out with benzhydrol (**1a**, 1 mmol), wet MeCN (**2a**, 1 mmol) in presence of various catalysts along with different solvent systems at 80 °C to produce N-benzhydrylacetamide (**3a)** (Scheme 1). Results are presented in Table 1.

From the results shown in Table 1, we standardized the reaction following the condition as specified in entry 8 which afforded 88% of the corresponding amide **3a**. The reactions did neither occur in anhydrous acetonitrile (entry 9) nor in water alone; wet acetonitrile came out as the suitable reaction medium where the amount of water is very crucial for optimum performance (0.1–0.3%). Best result was obtained with commercially available acetonitrile containing 0.1% of water. Use of toluene as a solvent in place of acetonitrile led to inferior results (entries 6 and 7). DMF was found not at all suitable as the solvent in this present protocol (entry 10).

Table 1. Optimization of the reaction conditions[a] using different catalysts and solvents at 80 °C.

Entry	Catalyst	Amount (mg)	Solvent	Time (h)	Yield of 3a (%)[b]
1	Dowex-50	40	Toluene	6	–
2	Dowex-50	50	DMF	8	–
3	Dowex-50	50	Acetonitrile	8	32
4	Amberlite (IR-45)	40	Toluene	6	24
5	Amberlite (IR-45)	50	DMF	8	–
6	Amberlyst®-15(H)	40	Toluene	6	40
7	Amberlyst®-15(H)	50	Toluene	6	46
8	*Amberlyst®-15(H)*	*50*	*Wet Acetonitrile (0.1% of water)*	*6*	*88*
9	Amberlyst®-15(H)	50	Acetonitrile (Anhydrous)	8	–
10	Amberlyst®-15(H)	50	DMF	8	–
11	Urea nitrate	80	Toluene	8	20
12	Alumina (acidic)	60	Acetonitrile	6	–
13	Ni- Alumina	60	Acetonitrile	6	–

[a]Reaction conditions: **1a** (1.0 mmol), **2a** (1.0 mmol), catalyst and time (as indicated), solvent (3 mL). [b]yield of isolated product.

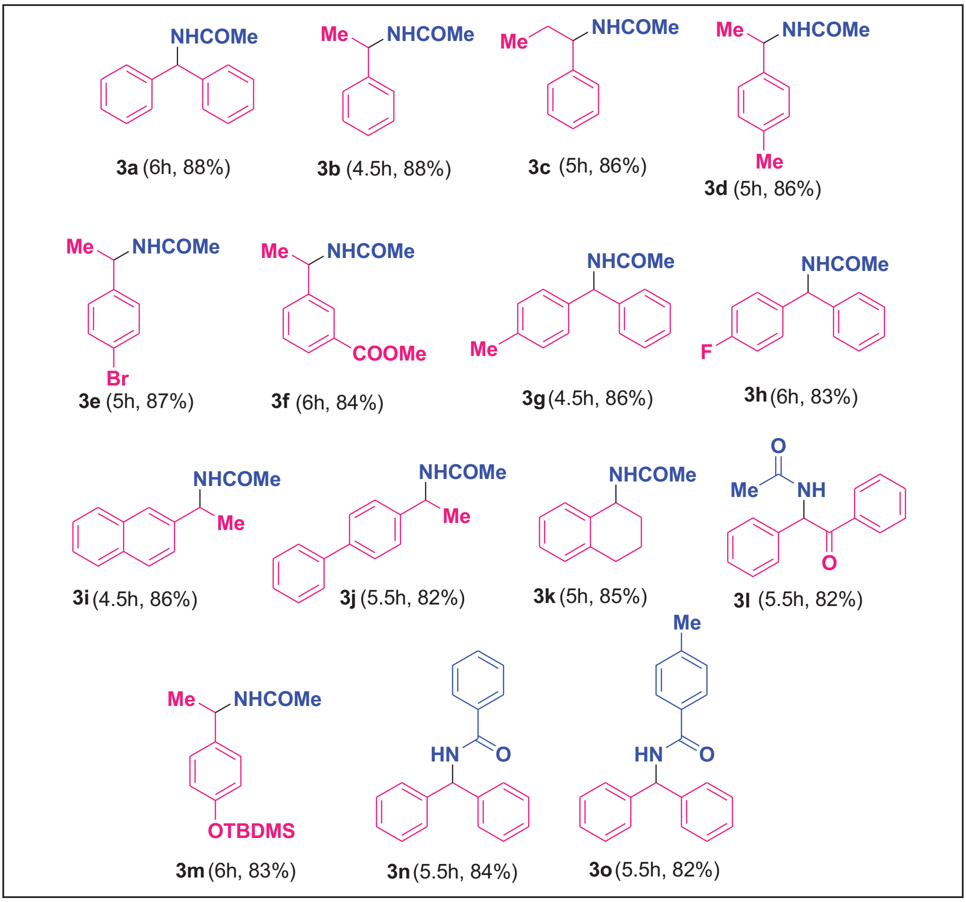
Before going to Amberlyst®-15(H), the same reaction was attempted with another well-known resin Dowex-50 where no conversion took place in toluene and DMF (entries 1 and 2) and only 32% of the product was obtained in acetonitrile solvent (entry 3). Similarly Amberlite (IR-45) produced only 24% of the required product in toluene (entry 4) and no reaction occurred in DMF (entry 5) leading to exclusive recovery of the substrate. Looking for another potential alternative of Amberlyst®-15(H), the reaction was carried out with urea nitrate where the product was obtained only in trace amount (entry 11). Even other acidic supports such as acidic alumina and Ni-alumina also failed in this case to provide the desired product (entries 12 and 13). The reaction was also carried out with 1-phenylethanol (**1b**) under optimized reaction condition at 80 °C using 1 drop of conc. H_2SO_4 as the catalyst instead of Amberlyst®-15(H) where a mixture of **3b** and styrene (**4a**) was obtained in 1:1.9 ratio (Scheme 2), but **3b** was obtained exclusively (88%) from **1b** using Amberlyst®-15(H).

In this way the essentiality, efficacy and applicability of Amberlyst®-15(H) as the solid acid resin was firmly established for the conversion of secondary alcohols to their corresponding N-acyl derivatives. Thus the present study led to the advent of a utilitarian and eco-compatible protocol for C–N bond formation using easily accessible substrates and catalyst. Moreover the same product **3a** was obtained when methyl and ethyl ethers of **1a** were used as the substrates in place of **1a**.

The catalyst i.e., Amberlyst®-15(H) was recovered by filtration, repeatedly washed with ethyl acetate, dried and further reused consecutively with marginal loss of its catalytic activity (Figure 1).

This protocol was further extended to substituted secondary and tertiary alcohols (Scheme 3 and Scheme 4) where a good number of substituted amides were obtained with satisfactory yield.

List of the products obtained in Scheme 3 using Amberlyst®-15(H)



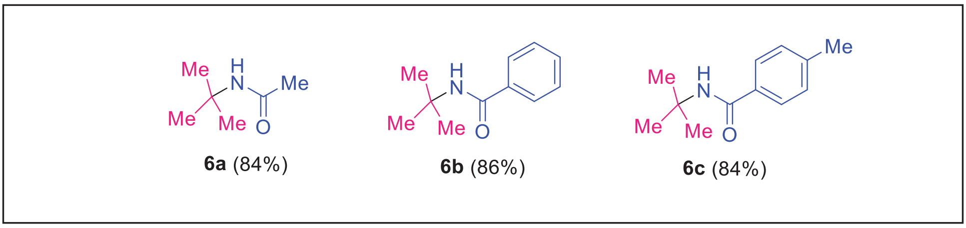
3a (6h, 88%) **3b** (4.5h, 88%) **3c** (5h, 86%) **3d** (5h, 86%)

3e (5h, 87%) **3f** (6h, 84%) **3g** (4.5h, 86%) **3h** (6h, 83%)

3i (4.5h, 86%) **3j** (5.5h, 82%) **3k** (5h, 85%) **3l** (5.5h, 82%)

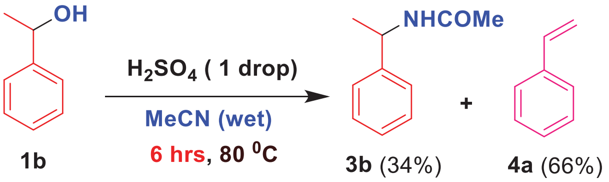
3m (6h, 83%) **3n** (5.5h, 84%) **3o** (5.5h, 82%)

List of the products formed in Scheme 4 using Amberlyst®-15(H)



6a (84%) **6b** (86%) **6c** (84%)

Under the optimized condition unsubstituted benzhydrol (**1a**) produced N-benzhydrylacetamide (**3a**) in satisfactory yield (yield refers to that of the isolated pure product fully characterized spectroscopically). This condensation reaction also took place very efficiently with both ring-unsubstituted and alkyl/aryl-substituted secondary benzylic alcohols to afford **3b**, **3c**, **3d**, **3g**, **3i** and **3j** with good yield. Secondary benzylic carbinols bearing halogen substituents in the aromatic ring responded smoothly to produce **3e**



Scheme 2. Reaction of 1-phenylethanol with 1 drop H_2SO_4 under optimized condition.

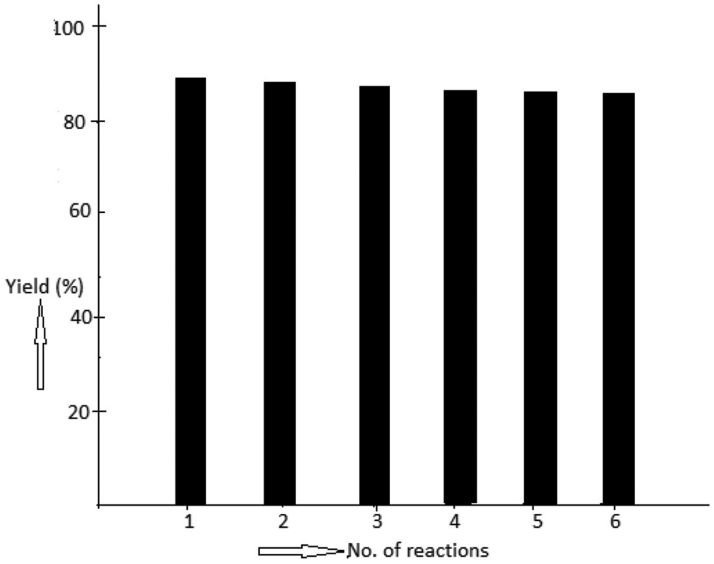
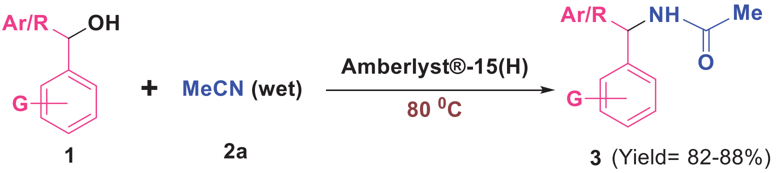
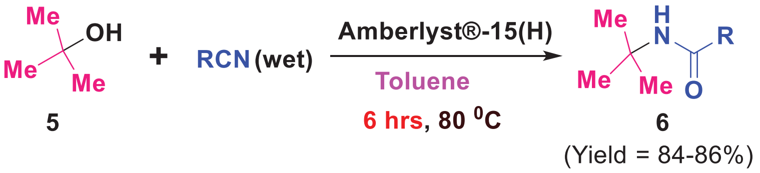


Figure 1. Recycling of Amberlyst®-15(H) using **1a** in moist MeCN at 80 °C for 6 h; % of yield was yield of isolated product **3a**.

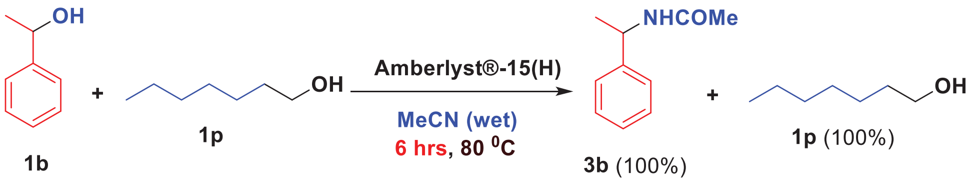


Scheme 3. Reaction of substituted secondary benzylic alcohols with wet acetonitrile under optimized condition.

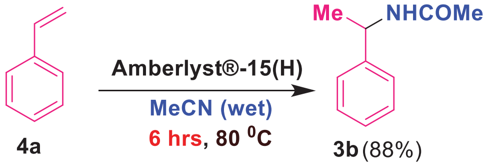


Scheme 4. Reaction of *tert*-butanol with alkyl/aryl nitriles.

and **3h** with 87% and 83% yields respectively. Hydrolyzable functional group -COOMe also survived in the present protocol to produce **3f** in 84% yield. The reaction gave quite impressive results with α-tetralol molecule forming **3k** with 85% yield. α-Hydroxyketone also reacted under this protocol and formed **3l** (82%) which is



Scheme 5. Intermolecular competition experiment (1).



Scheme 6. Reaction of styrene with wet acetonitrile under optimized condition.

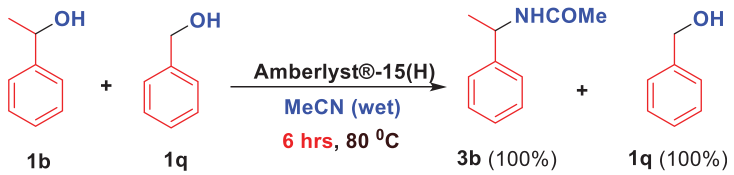
otherwise difficult to prepare. TBDMS group also survived under this procedure without any O-Si bond cleavage and **3 m** was obtained in 83% yield. When benzhydrol (**1a**) was reacted with benzonitrile and 4-methylbenzonitrile as a reagent in place of acetonitrile using toluene as the solvent it produced **3n** and **3o** respectively with satisfactory yield. No such reaction took place with aromatic or aliphatic primary alcohols where the substrates were recovered unchanged. When a mixture of equimolecular amounts of **1a** and benzyl alcohol was reacted under the optimized condition, only **1a** was converted to **3a** selectively leaving benzyl alcohol totally unaffected. Even with dialkyl secondary alcohol the reaction was inefficient to react under this protocol. Therefore this protocol is very much selective for only secondary alcohols where the aromatic ring is connected to the carbinol carbon. *tert*-Butanol responded efficiently under the present protocol (Scheme 4) giving N-tert-butyl-amides (**6a–c**) without any cleavage of any C-C bonds.

To substantiate the selectivity between aromatic and aliphatic alcohols, the competition reaction was carried out under optimized condition at 80 °C with equimolar mixtures of 1-phenylethanol (**1b**) and 1-heptanol (**1p**) where the product **3b** was obtained as expected from **1b**; leaving **1p** intact (Scheme 5). Absence of any signal at δ 3.24 ppm in ^{1}H NMR spectrum of the reaction mixture further confirmed the fact that the aliphatic $-C\underline{H}_2OH$ has not been transformed into $-C\underline{H}_2NHCOCH_3$.

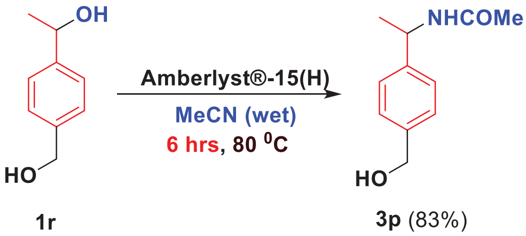
As a continuity of this theme, styrene (**4a**) was subjected to the optimized condition at 80 °C and produced N-(1-phenylethyl)-acetamide (**3b**) as the sole product (Scheme 6).

Equimolecular mixture of 1-phenylethanol (**1b**) and benzyl alcohol (**1q**), under optimized condition at 80 °C produced **3b** exclusively as the product obtained from **1b** leaving behind **1q** unaffected (Scheme 7).

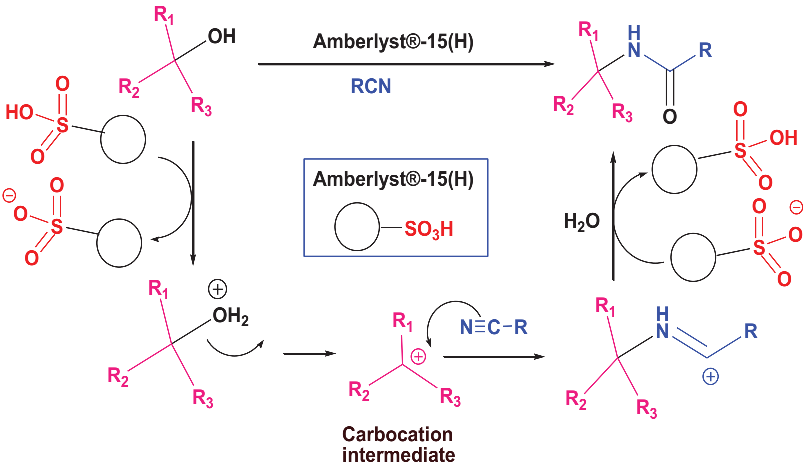
Intramolecular completion experiment with the substrate **1r** (containing both the aromatic primary and secondary carbinol moieties in the same molecule) under this protocol, produced **3p** exclusively (Scheme 8) with 83% yield. Excellent and subtle chemoselectivity was thus conclusively substantiated as the most important attribute of the present Amberlyst$^{®}$-15(H)-catalyzed protocol.



Scheme 7. Intermolecular competition experiment (2).



Scheme 8. Intramolecular competition experiment.



Scheme 9. Proposed mechanism for the reaction.

Both ^{1}H and ^{13}C NMR spectroscopic studies confirmed the occurrence of **3p**. A doublet at δ 1.46 in ^{1}H NMR (δ 21.8 in ^{13}C NMR) and a singlet at δ 1.96 in ^{1}H NMR (δ 23.6 in ^{13}C NMR) established the presence of one alkyl-methyl and one acetamino-methyl moieties respectively. A singlet at δ 4.51 in ^{1}H NMR (δ 71.9 in ^{13}C NMR) indicated the unperturbed benzylic primary carbinol moiety.

The aforesaid observations indicate that this subtle chemoselectivity might originate due to preferential formation of the relatively more stable carbocationic intermediate. The result obtained in the study delineated in Scheme 6 also indicated towards the involvement of carbocationic intermediate. Furthermore, when the reaction was carried out using enantiopure *R*-1-phenylethanol under the optimized reaction condition, the product **3b** was obtained as a racemic mixture. This observation also supported the formation of planar carbocation during the course of the reaction. On the basis of the above-mentioned outcomes, a plausible mechanistic scheme has been put forward (Scheme 9) involving the formation of carbocation through acid-catalyzed dehydration of alcohol with concomitant nucleophilic attack by nitrile followed by another nucleophilic attack by water to end up with the product amide. This mechanism also accounts for the catalytic role of Amberlyst®-15(H) in terms of Bronsted acidity and recyclability. No reaction took place with highly substituted triphenylmethanol due to lower stability of propeller-shaped triphenylmethyl carbocation as well as increased steric crowding.

Conclusion

In conclusion, commercially available Amberlyst®-15(H) has been effectively utilized as an air stable and recyclable heterogeneous inexpensive solid acid catalyst for the construction of C–N bond through chemoselective formation of diversely N-substituted amides using benzylic secondary alcohols as well as aliphatic tertiary alcohols and alkyl/aryl nitriles under environmentally acceptable conditions without any necessity of anhydrous and inert environment. Use of reagents, solvents and catalyst of negligible toxicity, mild reaction conditions, tolerance of various sensitive moieties, excellent chemoselectivity, wide substrate scope, high atom economy, formation of the most innocuous by-product (namely water), procedural simplicity, good yields and recyclability of the catalyst are the outstanding features of the present method with greater applicability compared to many existing protocols.

Experimental

A. To a mixture of benzhydrol **1a** (184 mg, 1.0 mmol) and wet MeCN **2a** (4 mL) the catalyst Amberlyst®-15(H) (50 mg) was added. The reaction mixture was stirred for 6 hours at 80 °C till the reaction was complete (monitored with TLC). Then the reaction mixture was cooled to room temperature, excess MeCN was removed under reduced pressure, the catalyst was filtered out keeping a cotton plug on a funnel and washed repeatedly by ethyl acetate (3 × 10 mL) to dissolve and collect the product. The product was thoroughly washed with water (2 × 10 mL) to remove any unused MeCN. The aqueous reaction mixture was then repeatedly extracted with ethyl acetate (3 × 5 mL). The combined organic extracts were washed with water (3 × 10 mL) and dried over anhydrous Na_2SO_4. The crude product was obtained by removal of the solvent under reduced pressure and purified by filtration chromatography on a short column of silica gel using 1–4% ethyl acetate-hexane as eluent to afford **3a**[21] (198 mg, Yield 88%, mp143–144 °C).

B. To a mixture of *tert*-butanol **5** (74 mg, 1.0 mmol), and PhCN **2b** (103 mg, 1.0 mmol) in 4 mL toluene, the catalyst Amberlyst®-15(H) (50 mg) was added. The reaction mixture was stirred for 6 hours at 80 °C till the reaction was complete (monitored with TLC). Then the reaction mixture was cooled to room temperature, excess toluene was removed under reduced pressure, keeping a cotton plug on a funnel the catalyst was filtered out and washed repeatedly by ethyl acetate (3 × 10 mL) to dissolve and collect the product. The product was thoroughly washed with water (2 × 10 mL). The aqueous reaction mixture was then repeatedly extracted with ethyl acetate (3 × 5 mL).

The combined organic extracts were washed with water (3 × 10 mL) and dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure furnished the product **6b**[29] (152 mg, Yield 86%, mp132–133 °C) without any necessity of further purification.

Supporting information

This part contains experimental procedure, physical characteristics as well as spectral (^1H and ^{13}C NMR) and HRMS/analytical data (wherever applicable) for the products. This material can be found via the "Supplementary Information" section of this article's webpage.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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