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Copper catalyzed C–O bond formation by direct C–H activation of THF with phenols: an approach to the synthesis of phenyl tetrahydrofuranyl ethers

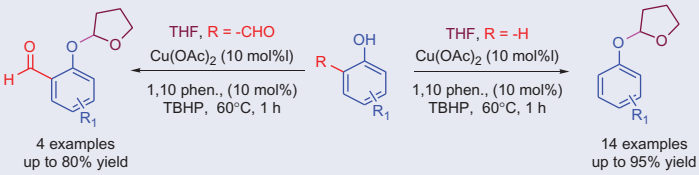
Manoj Kumar Vala[a,b], Suryakumari Adurthi[a,b], and Sudhakar Chithaluri[a]

[a]Department of Chemistry, GITAM Deemed to be University, Rudraram, India; [b]Department of Chemistry, TARA Govt Degree College, Sangareddy, India

ABSTRACT

A direct oxidative transformation of phenol derivatives to the corresponding tetrahydrofuranyl ethers via C–H bond activation, in the presence of copper catalyst and desired products were achieved in good yields using TBHP as an oxidant.

GRAPHICAL ABSTRACT



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C–H activation; $Cu(OAc)_2$; phenol substrates; tetrahydrofuranyl ethers

Introduction

The transition-metal catalyzed synthetic protocols of carbon–heteroatom bond formation have been much more advanced in the trends of synthetic organic chemistry.[1] Apart from the well-established classical cross-coupling strategies are versatile methods for C–O bond formations that have found broad applications in organic synthesis, protecting groups and related disciplines.[2] In particular, transition-metal-catalyzed reactions have attracted significant interest in the synthetic community.[3]

Copper has been widely studied in organic synthesis because of its promise as a high efficiency catalyst, and Cu-catalyzed C–H activation reactions have been described.[2d,4] Recently, the direct functionalization of O–H bonds in simple phenols and alcohols has received significant attention in organic synthesis because of their potential possibility for diverse transformation into a variety of useful derivitatives.[5] In the past five decades, a multitude of different strategies has been developed for the protection of important functional groups in organic compounds.[6] Tetrahydropyranylation[7] and tetrahydrofuranylation[8,9] have become widely used methods in organic synthesis for the protection of hydroxyl groups.

CONTACT Sudhakar Chithaluri schithal@gitam.edu; sudha1iict@gmail.com Department of Chemistry, GITAM Deemed to be University, Rudraram, Hyderabad 502329, India.

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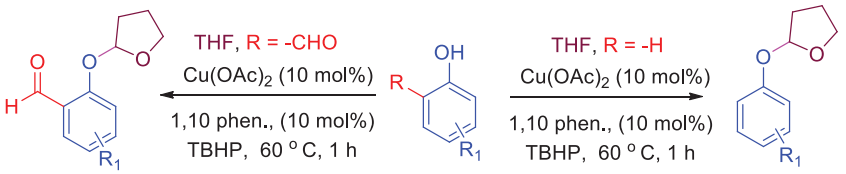


Scheme 1. Synthesis of 2-phenoxytetrahydrofuran derivatives Reaction conditions: Phenol derivative (1 mmol), $Cu(OAc)_2$ (10 mol%), 1,10 phen. (10 mol%), TBHP in water (3 equiv.), THF 3 mL, 60 °C, 1 h.

A very important method for protecting alcohol functional groups in synthesis involves their conversion to acetals.[10] Conversion to tetrahydropyranyl (THP) ethers is very common and these are generally prepared from an acid-catalyzed addition of the alcohol to dihydropyran (DHP). Tetrahydropyranyl (THP) and tetrahydrofuranyl (THF) ethers make ideal protecting groups because of their ease of preparation and stability toward a broad range of reaction conditions, such as strongly basic media, hydrides, and acylating and alkylating agents.

There have been several methods have been reported for the synthesis of THF ethers.[9] In addition, the activation of tetrahydrofuran has recently been reported with a variety of one-electron oxidants, including p-TsOH,[11] cerium (IV) reagents,[12] peroxodisulfates,[13] $CrCl_2$,[14] alkylperoxy-λ3-iodane,[15] Manganese powder,[16] aluminum triflate,[17] allyl chloride[18] and Mn_3O_4 (SMONP).[19] Unfortunately, there are issues associated with all of these methods, including the use of expensive reagents, elevated temperatures, strongly acidic conditions, toxic reagents, and low levels of functional group tolerance.

Results and discussion

In this context, we look for the efficient and environmentally benign direct coupling of phenol derivatives with tetrahydrofurans in the presence of external ligand for the synthesis of 2-phenoxytetrahydrofuran derivatives via O–H bond activation (Scheme 1).

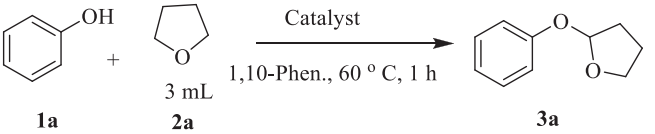
During these investigations, desired product was observed, under oxidative cross coupling of phenols with THF in the presence of copper catalyst, 1, 10-phenonthroline ligand and TBHP as an external oxidant. This is a ligand-assisted copper-catalyzed tetrahydrofuranylation of phenols and alcohols under mild conditions (Scheme 1). Interestingly, this transformation represents a direct cross coupling of sp^3 C–H bond and O–H bond.

In the initial experiments, simple phenol was chosen as a model substrate and treated with excess amount of THF, using 10 mol% of CuI as catalyst and 10 mol% of 1, 10 phenanthroline as a ligand, TBHP as the external oxidant (Table 1, entry 1). A small amount of carbamate product was formed at 60 °C for 1 h. In these reaction conditions hydroxyl group of phenol participated in the oxidative coupling with the THF and afforded the 2-phenoxytetrahydrofuran, which was the desired product. Further purification of the product and analysis by ^1H, ^{13}C NMR and ESI mass revealed that the coupled product is indeed the 2-phenoxytetrahydrofuran. Based on this initial result, further optimization was carried out under different reaction conditions (Table 1). Absence of any coupled product in blank experiments clearly shows the importance of both catalyst and oxidant for this reaction (Table 1, entry 7, 8). It has been observed that both Cu(I) and Cu(II) salts are quite active for the coupling reaction (Table 1, entries 2–6), among the different

copper salts, $Cu(OAc)_2$ provided relatively higher yields of the product (Table 1, entry 6). Then the role of different oxidants was evaluated, and no product formation was observed with the use of H_2O_2, m-CPBA and NaOCl as oxidants (Table 1, entries 9–11).

Further, optimization experiments were carried out with tetrahydrofuran (THF) and simple phenol as the reaction partners in presence of $Cu(OAc)_2$ by using various ligands (Table 2). Among them, bidentate ligands viz., 2,2′-bipyridine (BPY) and 1,10-phenanthroline (phen.) have shown relatively higher activity. In these optimization studies, the absence of the product in control experiments also clearly indicates that the ligand is essential for the activation of phenol by the metal ion (Table 2, entry 3). During these investigations various ligands were further tested (Table 2). Whereas, the product yield was not increased when the reaction was carried out in presence of other ligands DMAP, DMEDA, and TMEDA (Table 2, entries 4–6). As shown in the table,

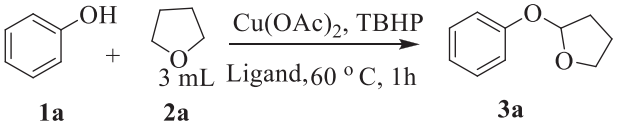
Table 1. Catalyst optimization studies of reaction conditions.[a]



Entry	Catalyst	Oxidant	Yield[%][b]
1	CuI	TBHP	20
2	CuBr	TBHP	25
3	$CuBr_2$	TBHP	42
4	$CuCl_2$ $2H_2O$	TBHP	48
5	Cu(OAc)	TBHP	64
6	$Cu(OAc)_2$	TBHP	83
7	–	TBHP	NR
8	$Cu(OAc)_2$	–	NR
9	$Cu(OAc)_2$	NaOCl	NR
10	$Cu(OAc)_2$	*m*-CPBA	NR
11	$Cu(OAc)_2$	H_2O_2	NR

[a]Reaction conditions: Phenol (1 mmol), catalyst (10 mol%), 1,10- phenanthroline (10 mol%), THF (3 mL, 30 mmol), 3 equiv., TBHP(tert-Butyl hydroperoxide—70 wt % in H_2O), 60 °C, 1 h. [b]Isolated yields.

Table 2. Ligand optimization studies of 2-phenoxytetrahydrofuran formation.[a]



Entry	Ligand	Yield [%][b]
1	Triethylamine	10
2	Ethylene diamine	14
3	–	NR
4	DMAP	25
5	DMEDA	30
6	TMEDA	33
7	2-aminomethyl pyridine	42
8	2,2′-bipyridine	48
9	1,10-Phenanthroline	83

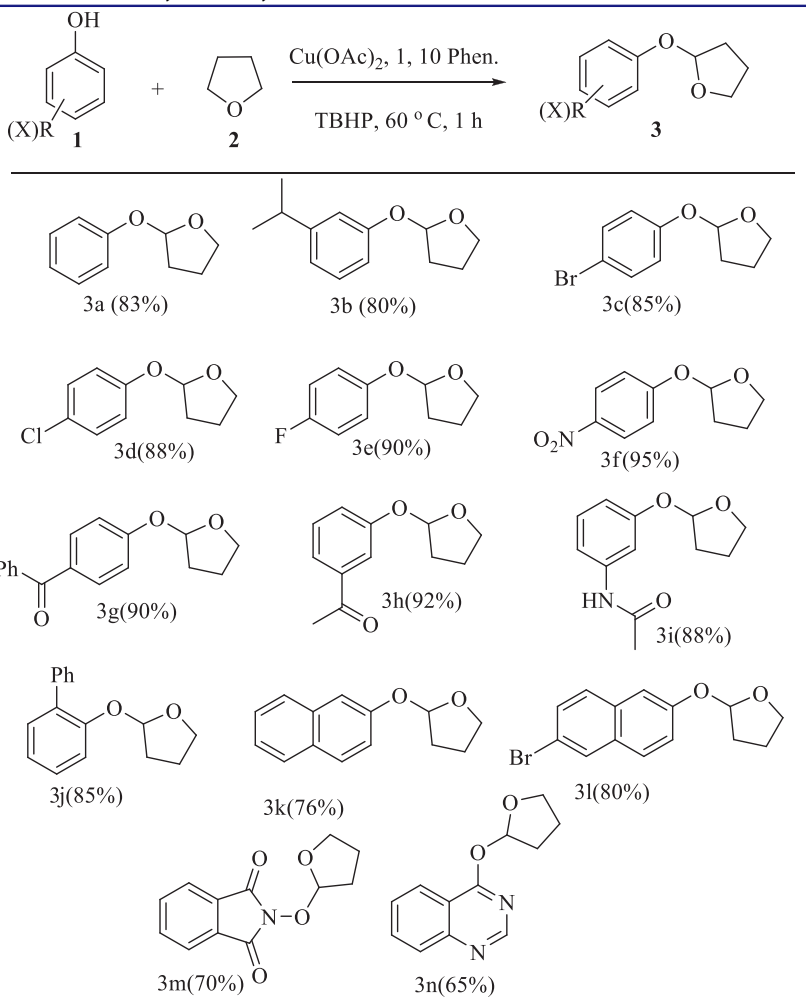
[a]Reaction conditions: Phenol (1 equiv.), catalyst (10 mol%), THF (3 mL), TBHP in water (3 equiv.), ligand (10 mol%), 60° C, 1 h. [b]Isolated yields.

most of the ligands have shown some activity, still 1, 10-phenanthroline proved to be the best one (Table 2, entry 9).

With these optimization studies, the general scope of the present oxidative cross coupling protocol was carried out for different phenol substrates using 10 mol% of $Cu(OAc)_2$, 3 mL of tetrahydrofuran source, 3 equiv. of TBHP in water solution, 10 mol% of 1,10 phenanthroline at 60 °C for 1 h and the results are summarized in Table 3. Simple phenol and substrates having electron-donating groups at meta-position provided the 2-phenoxytetrahydrofuran products in good yields (Table 3, 3a and 3b).

Similarly, substrates having electron withdrawing groups at para- position provided the 2-phenoxytetrahydrofuran derivatives in high yields (Table 3, 3c–3g). In addition, that there was a negligible meta-electron withdrawing group influence on the product where moderate yield of the product was observed (Table 3, 3h and 3i). A similar

Table 3. Synthesis of tetrahydrofuranyl ether derivatives.[a,b]

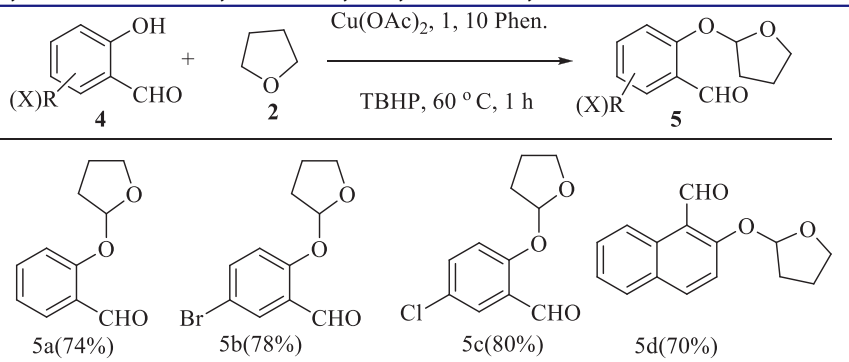


[a]Reaction conditions: Phenol substrate (1 mmol), catalyst (10 mol%), TBHP (3 equiv.), ligand (10 mol%), THF (solvent, 3 mL). [b]Isolated yields.

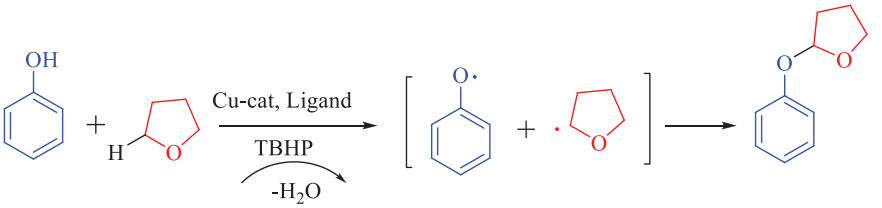
reactivity pattern was also observed for other derivatives (Table 3, 3j). (Table 3, 3k–3n). Further, to test the generality of this reaction with naphthalene, hetero aromatic phenols such as 2-hydroxyisoindoline-1,3-dione and quinazolin-4-ol were used. These derivatives provided the corresponding tetrahydrofuranyl ether products in moderate to good yields Furthermore; the present coupling protocol was also extended to salicylaldehyde substrates with tetrahydrofuran (THF) as an ether source, whose resulting products have more importance like as protecting groups in synthetic organic chemistry. Moreover, the recent reports on coupling chemistry involving tetrahydrofuran were mainly with simple phenol derivatives and rarely reported with salicylaldehyde substrates. In the present investigation, the reaction of salicylaldehyde substrate with THF under optimized reaction conditions provided 70–80% yields of the 2-((tetrahydrofuran-2-yl)oxy)benzaldehyde product (Table 4, 5a–5d).

From the above results, ligand plays an important role in the activation of phenol and salicylaldehyde substrates with a subsequent coupling of tetrahydrofuran. The reaction between simple phenol and THF under 1:0; 1:1; 1:2 and 1:3 metal to ligand ratios resulted in 0%, 83%, 60% and 35% yields of the tetrahydrofuranyl product respectively, which clearly confirms the participation of ligand with metal ion in activation of phenol. Similarly, no product formation was observed in the presence of radical scavenger TEMPO, it has strongly supported that the present system undergoes via radical mechanism (Scheme 2).[19,20]

Table 4. Synthesis of 2-((tetrahydrofuran-2-yl) oxy) benzaldehyde derivatives.[a,b]



[a]Reaction conditions: Salicylaldehyde substrate (1 mmol), catalyst (10 mol%), TBHP (3 equiv.), ligand (10 mol%), THF (solvent, 3 mL). [b]Isolated yields.



Scheme 2. Plausible mechanism for 2-phenoxytetrahydrofuran formation

Conclusion

The present copper-catalyzed oxidative C–O bond formation protocol in which the simple phenols and salicylaldehydes were directly coupled with simple tetrahydrofurans to generate the unsymmetrical acetal scaffolds. It has been shown that both phenol- and salicylaldehyde substrates were easily coupled under mild reaction conditions with THF, resulting in tetrahydrofuranyl ethers, which are useful for protecting groups. Generally, this alcohol functionality has affords protected by temporarily converting into simple derivatives in which most often as ethers. This protocol provides a new route for the protection of alcohols. The present method is useful in the synthesis of salicylaldehyde tetrahydrofuranyl ethers, which are not easily available by other methods. Although in some cases the yields are moderate, this new approach involving the oxidative cross-coupling chemistry via C–H bond activation.

Experimental section

General procedure for the synthesis of 2-phenoxytetrahydrofuran

In a reaction vessel $Cu(OAc)_2$ (0.1 mmol), 1,10-phenanthroline (0.1 mmol) was dissolved with 3 mL of THF source. The reaction mixture stirred for five minutes and added the phenol (1.0 mmol) substrates. To the above reaction mixture, 3.0 equiv., of TBHP (tert-Butyl hydroperoxide −70 wt % in H_2O) was added dropwise, with stirring over a period of 5 min. Then the reaction temperature was increased to 60 °C and stirred for one hour. After cooling to room temperature, the reaction mixture was directly subjected to purification with column chromatography on silica gel using 5–10% ethyl acetate and hexane mixture to afford the required 2-phenoxy tetrahydrofuran derivatives.

2-Phenoxytetrahydrofuran(3a): yield (**83**%); m.p.: 18–20 °C; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.34 (t, $J = 7.7$ Hz, 2H, 2xCH_{Ar}), 7.12 (d, $J = 8.1$ Hz, 2H, 2xCH_{Ar}) , 7.04 (m, $J = 7.3$ Hz, 1H, CH_{Ar}), 5.88 (t, $J = 4.34$ Hz, 1H, O–CH–O), 4.15–3.97 (m, 2H, OCH_2), 2.26–2.0 (m, 4H, –CH_2–CH_2–); **^{13}C NMR** (75 MHz, $CDCl_3$): δ 157.0 (O–C_{Ar}), 129.2 (CH_{Ar}), 121.4 (CH_{Ar}), 116.4 (CH_{Ar}), 102.1 (O–CH–O), 67.9 (O–CH_2), 32.6 (CH_2), 23.3 (CH_2); **IR (KBr)**: 28,351,529,138,012,500,000,000,000,000 cm^{-1}; **HRMS (ESI)** calcd for $C_{10}H_{13}O_2$ $(M + H)^+$ 165.09101, found 165.09060.

Full experimental detail, ^{1}H and ^{13}C NMR spectra. This material can be found via the "Supplementary Content" section of this article's webpage.

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