

Phase-Transfer-Catalyzed Intramolecular Hydroaryloxylation and Hydroamination of C≡C Triple Bonds: An Efficient Synthesis of Benzo[*b*]furan and 3-Methyleneisoindoline-1-one Derivatives

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Abstract

Phase-transfer-catalyzed intramolecular cyclization reaction of forming benzo[*b*]furan and 3-methyleneisoindoline-1-one derivatives has been developed. The cyclization reaction of propargylic carbonates was also described under metal-free condition and the reaction was reported by Pd and Ni before. The reaction conditions and the scope of the process are examined. The catalysts are cheap and environmentally friendly and the substrates are readily available and the procedure is simple, rapid, and general. The development of C-O and C-N bond formation processes via an overall structural isomerization represents the most atom-economical approach.

Keywords: PTC, Intramolecular, Cyclization, Metal-Free

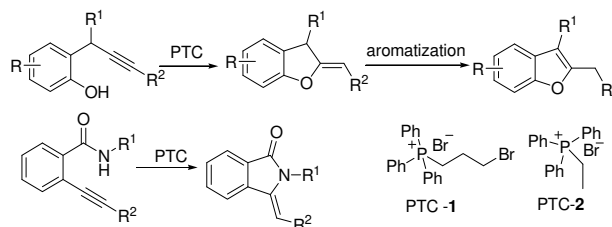
1. Introduction

The addition of heteroatomic nucleophiles to C≡C triple bonds is an important reaction that often requires a high activation energy [1,2]. The transition metal-catalyzed construction of heterocycles is one of the frontier areas in organic chemistry, because of the exceptional ability to activate the triple bond, towards intermolecular and intramolecular nucleophilic attack [3-7]. Oxygen-containing heterocyclic moieties such as benzo[*b*]furan rings represent key structural units of a variety of natural compounds with interesting biological and pharmacological activities [8-10] and a number of routes leading to differently substituted benzo[*b*]furans have been described by metal-catalyzed intramolecular ring closure [11-16]. Meanwhile, nitrogen-containing heterocyclic moiety 3-methyleneisoindoline-1-one is a core structure of numerous natural products and has also been prepared in the literature [17,18]. Compared with other methods phase-transfer catalysis has long been recognized as a versatile methodology for organic synthesis in both industrial and academic laboratories, featuring its simple reaction procedure, safe, inexpensive, environmentally friendly reagents, absence of anhydrous solvents, ease of scale-up, and metal-free conditions [19-22].

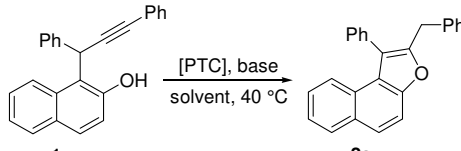
Recently, [23] we have developed a novel and efficient cyclization reaction initiated by phase-transfer catalysis for building carbon-carbon bond. Herein, we report a new mild synthesis of heterocyclic compounds under phase-transfer catalysis by carbon-oxygen or carbon-nitrogen bond formation (Scheme 1).

2. Results and Discussion

Initially, we focused on the reaction of 0.20 mmol of 1-(1,3-diphenylprop-2-ynyl)naphthalen-2-ol (**1a**), 5 mol % of PTC-1 and 2 equiv of Na₂CO₃ in DMF at 40°C in air. To our delight, the desired product benzo[*b*]furan **2a** was formed in 66% yield after 1 h (Table 1, entry 1). Encouraged by this result, we further optimized the reaction



Scheme 1. C-O bond and C-N bond formation by PTC.

Table 1. Optimization of the phase-transfer-catalyzed intramolecular cyclization of 1a^a.


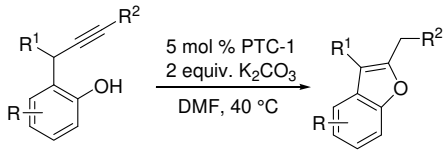
Entry	[PTC]	Base	Solvent	Time (h)	Yield (%) ^[b]
1	PTC-1	Na ₂ CO ₃	DMF	1	66
2	PTC-1	K ₂ CO ₃	DMF	0.5	85
3	PTC-1	Li ₂ CO ₃	DMF	12	50
4	PTC-1	K ₃ PO ₄	DMF	1	44
5	PTC-1	KOAc	DMF	0.5	65
6	PTC-1	K ₂ HPO ₄	DMF	1	31
7	PTC-1	KHCO ₃	DMF	0.5	67
8		K ₂ CO ₃	DMF	3	50
9		KO ^t Bu	DMF	0.5	39
10	PTC-1	K ₂ CO ₃	DMA	1	82
11	PTC-1	K ₂ CO ₃	NMP	1	77
12	PTC-1	K ₂ CO ₃	DMSO	1	68
13	PTC-1	K ₂ CO ₃	THF	3	77
14	PTC-1	K ₂ CO ₃	1,4-dioxane	1.5	53
15	PTC-1	K ₂ CO ₃	CH ₃ CN	1	83
16	PTC-2	K ₂ CO ₃	DMF	0.5	51
17	TBAI	K ₂ CO ₃	DMF	0.5	56
18	TEAB	K ₂ CO ₃	DMF	0.5	68
19	TBAC	K ₂ CO ₃	DMF	0.5	66
20	TBAF	K ₂ CO ₃	DMF	0.5	64

^aReactions were carried out on a 0.2 mmol scale in 2.0 mL of solvent in air at 40 °C with 1.0 equiv of **1a**, 2.0 equiv of base and 5 mol% equiv of PTC.

^bIsolated yield.

conditions. Other common bases, such as K₂CO₃, Li₂CO₃, K₃PO₄, KOAc, K₂HPO₄, KHCO₃ were also tested; K₂CO₃ gave the best yield of 85% (entries 2 - 7), which indicated that base played an important role in the process. In the absence of PTC condition, the product was obtained in low yield (entries 8 and 9). The effect of the solvent was also investigated. Changing solvent to DMA, NMP, DMSO, THF, 1, 4-dioxane, CH₃CN failed to improve the yield of the product **2a** (entries 10 - 15). When different PTCs were examined, the results showed that PTC-2 could also promote this cyclization, but the desired product **2a** was only obtained with 51% yield (entry 16). Tetraalkyl ammonium salts, such as Bu₄NI, Et₄NBr, Bu₄NCl, and Bu₄NF have also been applied to this process, and the yields were not better than PTC-1 (entries 17 - 20). Thus, we chose the following reaction conditions as optimum for subsequent cyclization: 0.20 mmol of **1a**, 5 mol % PTC-1, and 0.40 mmol K₂CO₃ in DMF at 40 °C in air.

With the optimized conditions in hand, the scope of this reaction was then investigated, and the results are summarized in **Table 2**. As depicted in **Table 2**, the

Table 2. Phase-transfer-catalyzed synthesis of disubstituted benzo[b]furan derivatives (2a-m)^a.


Entry	Substrate (1)/Time (h)	Product (2)	Yield ^[b]
1	1a R ¹ = Ph, R ² = Ph (0.5)	2a R ¹ = Ph, R ² = Ph	85
2	1b R ¹ = <i>o</i> -CH ₃ OC ₆ H ₄ , R ² = Ph (1.5)	2b R ¹ = <i>o</i> -CH ₃ OC ₆ H ₄ , R ² = Ph	80
3	1c R ¹ = <i>m</i> -CH ₃ C ₆ H ₄ , R ² = Ph (0.5)	2c R ¹ = <i>m</i> -CH ₃ C ₆ H ₄ , R ² = Ph	95
4	1d R ¹ = <i>p</i> -CH ₃ C ₆ H ₄ , R ² = Ph (0.5)	2d R ¹ = <i>p</i> -CH ₃ C ₆ H ₄ , R ² = Ph	81
5	1e R ¹ = 2,4-diClC ₆ H ₄ , R ² = Ph (0.5)	2e R ¹ = 2,4-diClC ₆ H ₄ , R ² = Ph	60
6	1f R ¹ = Ph, R ² = TMS (0.5)	2f R ¹ = Ph, R ² = H	75
7	1g R ¹ = Ph, R ² = CH ₃ CH ₂ CH ₂ (1)	2g R ¹ = Ph, R ² = CH ₃ CH ₂ CH ₂	20
8	1h R ¹ = CH ₃ (CH ₂) ₂ , R ² = Ph (2)	2h R ¹ = CH ₃ (CH ₂) ₂ , R ² = Ph	51
9	1i R = 4-CH ₃ C ₆ H ₄ , R ¹ = Ph (0.5)	2i R = 4-CH ₃ C ₆ H ₄ , R ¹ = Ph	76
10	1j R = 5-CH ₃ C ₆ H ₄ , R ¹ = Ph (0.5)	2j R = 5-CH ₃ C ₆ H ₄ , R ¹ = Ph	87
11	1k R = 4- ^t BuC ₆ H ₄ , R ¹ = Ph (0.5)	2k R = 4- ^t BuC ₆ H ₄ , R ¹ = Ph	64
12	1l R = 5-CH ₃ C ₆ H ₄ , R ¹ = <i>m</i> -CH ₃ C ₆ H ₄ (1)	2l R = 5-CH ₃ C ₆ H ₄ , R ¹ = <i>m</i> -CH ₃ C ₆ H ₄	72
13	1m R = 5-CH ₃ C ₆ H ₄ , R ¹ = <i>o</i> -CH ₃ OC ₆ H ₄ (2)	2m R = 5-CH ₃ C ₆ H ₄ , R ¹ = <i>o</i> -CH ₃ OC ₆ H ₄	62

^aReactions were carried out on a 0.2 mmol scale in 2.0 mL of DMF in air at 40 °C with 1.0 equiv of **1a**, 2.0 equiv of K₂CO₃ and 5 mol% equiv of PTC-1. ^bIsolated yield (%).

reaction was application to various propargylnaphthols and phenols, and for the most tested substrates, good results have also been obtained. Naphthols with an electron-donating aryl group at propargylic position cyclized smoothly to give the products in high yields, no matter substituents appeared in *ortho*, *meta* or *para* positions (Table 2, entries 2 - 4), while the electron-withdrawing aryl group naphthol (**1e**) apparently lowered yield (entry 5). The trimethylsilyl-substituted naphthol, **1f**, reacted to produce the respective naphthofuran in a moderate (75%) yield and the TMS group happened to undergo desilylation at the same time (entry 6). When a propargylnaphthol with a butyl group at the triple bond was used, the desired product **2g** was obtained in a poor yield (entry 7). On employing naphthol with a propyl group at the propargylic position, cyclization product **2h** was obtained in moderate yield (entry 8). Furthermore, the propargylphenols were also examined. The presence of electron-donating group on the aromatic ring gave moderate to good yields (entries 9 - 11). Introducing an electron-donating aryl group at the propargylic position, the reaction proceeded smoothly to give the corresponding cyclization products (entries 12 and 13).

Furthermore, to expand the scope of this reaction, we also investigated the ethynylphenol **1n-r**. As depicted in Table 3, for all the tested substrates, the phase-transfer-catalysts intramolecular cyclization of ethynylphenol could prove to be a very effective method for the synthesis of a variety of monosubstituted benzo[*b*]furans and the reaction can be tolerant of the substitution groups in aromatic ring (Table 3, entries 1 - 4). It is noteworthy that when R¹ was replaced by an alkyl group, the reaction proceeds smoothly to give the corresponding product in good yield (entry 5).

Table 3. Phase-transfer-catalyzed synthesis of monosubstituted benzo[*b*]furan derivatives (**2n-s**)^a.

Entry	Substrate (1)/Time (h)	Product (2)	Yield (%) ^[b]
1	1n R=H, R ¹ =h (1)	2n R=H, R ¹ =Ph	90
2	1o R=H ₃ , R ¹ =h (1)	2o R=CH ₃ , R ¹ =Ph	95
3	1p R=I, R ¹ =h (1)	2p R=Cl, R ¹ =Ph	90
4	1q R= <i>t</i> -Bu, R ¹ =Ph (1.5)	2q R= <i>t</i> -Bu, R ¹ =Ph	86
5	1r R=H, R ¹ =CH ₃ (CH ₂) ₄ (1.5)	2r R=H, R ¹ =CH ₃ (CH ₂) ₄	84

^aReactions were carried out on a 0.2 mmol scale in 2.0 mL of CH₃CN in air at 40°C with 1.0 equiv of **1a**, 2.0 equiv of Cs₂CO₃ and 5 mol% equiv of PTC-1. ^bIsolated yield.

The addition of nitrogen nucleophiles to triple bonds were also investigated and results are summarized in Table 4. On introducing aryl groups on the amide moiety, the desired products were generated in good to excellent yields irrespective of the presence of electron-withdrawing or electron-donating groups (entries 2 - 6). Functional groups, such as methyl, methoxyl and bromo were well tolerated in the reaction. When R¹ was the benzyl or substituted benzyl, good results still were obtained in our system (entries 7 - 9). Furthermore, we investigated the effect of various substituents on the remote end of the alkyne moiety. These substrates all worked well and gave moderate yields (entries 10 - 14).

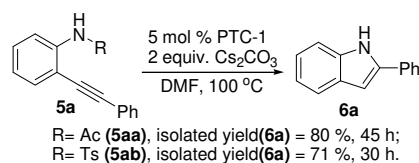
When the substrates **5a** was investigated, to our surprising, 2-phenyl-1H-indole was obtained in good yield (Scheme 2).

For the substrates **7aa**, **7ba** and **7ca**, we have reported via Pd [24] and Ni [25,26] catalyzed reaction. To our delight, the same good result could also be obtained in our system under metal-free condition (Scheme 3).

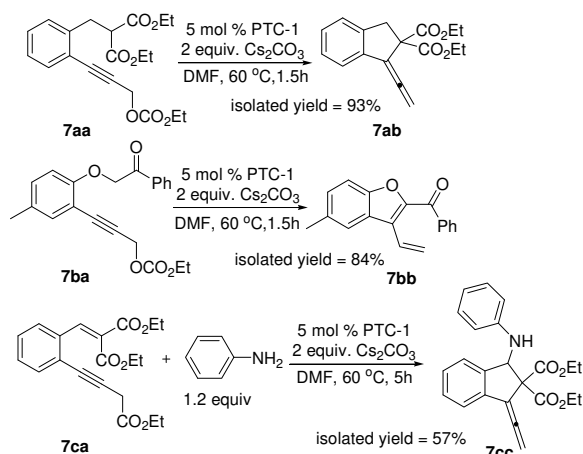
Table 4. Cyclization reaction catalyzed by TBAB^{a,b}.

Entry	R ¹	R ²	3	4	Isolated Yield (%)
1	CH ₃	Ph	3a	4a	98
2	Ph	Ph	3b	4b	91
3	m-CH ₃ C ₆ H ₄	Ph	3c	4c	84
4	p-CH ₃ C ₆ H ₄	Ph	3d	4d	80
5	p-CH ₃ OC ₆ H ₄	Ph	3e	4e	94
6	m-BrC ₆ H ₄	Ph	3f	4f	80
7	PhCH ₂	Ph	3g	4g	90
8	p-CH ₃ OPhCH ₂	Ph	3h	4h	91
9	p-ClPhCH ₂	Ph	3i	4i	87
10	Ph	p-CH ₃ C ₆ H ₄	3j	4j	65
11	Ph	p-BrC ₆ H ₄	3k	4k	64
12	Ph	m-CH ₃ C ₆ H ₄	3l	4l	63
13	Ph	o-CH ₃ C ₆ H ₄	3m	4m	67
14 ^c	Ph	CH ₂ CH ₂ CH ₃	3n	4n	70

^aReactions were carried out on a 0.2 mmol scale in 2.0 mL of solvent in air at r.t. with 1.0 equiv of **1a**, 2 equiv of base and 5 mol% equiv of TBAB for 0.5 h. ^bTBAB: Tetrabutylammonium bromide. ^cThe reaction was carried out for 5 h.



Scheme 2. Phase-transfer-catalyzed intramolecular cyclization of **5a**.



Scheme 3. Phase-transfer-catalyzed intramolecular cyclization of propargylic carbonates.

3. Experimental Part

Typical procedure for the preparation of disubstituted benzo[*b*]furan derivatives **2a**. To a solution of **1a** (65.2 mg, 0.20 mmol) in 2.0 mL of DMF was added K_2CO_3 (55.2 mg, 0.40 mmol). The mixture was allowed to stir at room temperature for 1 minute and PTC-1 (4.74 mg, 5 mol%) was added. The resulting mixture was then heated under air at 40 °C. When the reaction was considered complete as determined by TLC analysis, the reaction was allowed to cool to room temperature and quenched with a saturated aqueous solution of ammonium chloride, and the mixture was extracted with EtOAc. The combined organic extracts were washed with water and saturated brine. The organic layers were dried over Na_2SO_4 , filtered. Solvents were evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford 2,3-disubstitution benzo[*b*]furans **2a**.

Typical procedure for the preparation of 3-methyleneisindoline-1-one derivatives **4a** to a solution of **3a** (0.20 mmol) in 2.0 mL of CH_3CN was added Cs_2CO_3 (130.4 mg, 0.40 mmol). The mixture was allowed to stir at room temperature when TBAB (3.22 mg, 5 mol%) was added. When the reaction was considered complete as determined by TLC analysis, the reaction was allowed to quench with a saturated aqueous solution of ammonium chloride, and the mixture was extracted with EtOAc. The combined organic extracts were washed with water and saturated brine. The organic layers were dried over Na_2SO_4 , filtered. Solvents were evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford 3-methyleneisindoline-1-one derivatives **4a**.

4. Conclusions

In conclusion, phase-transfer-catalyzed intramolecular hy-

droaryloxylation and hydroamination reaction of corresponding O/N-containing substrates is a novel and efficient method for synthesis of heteroatomic compounds. We also described the cyclization reaction of propargylic carbonates under metal-free condition. These reactions are run under mild conditions, tolerate various functional groups, and generally a wide range of substrates undergo this process in good to excellent yields. Compared to the expensive and toxic transition-metal catalyzed reaction, this methodology showed considerable synthetic advantages in terms of mild reaction conditions, environmentally friendly catalysts and simple experimental operations. The scope and synthetic application of these reactions are currently under investigation.

5. Acknowledgements

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6. References

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