Synthesis of dihydrobenzofurans and dihydroindoles by intramolecular Friedel-Crafts cyclizations of aryloxy- and arylamino-substituted propargylic alcohols

Xiangsheng Xu,* Yanfeng Lu, Guo Hong, Zhiteng Zhao, and Xiaoqing Li

College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou 310 014, China
E-mail: future@zjut.edu.cn

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Abstract

A $FeCl_3$-catalyzed intramolecular Friedel-Crafts cyclization of aryloxy- and arylamino-substituted propargylic alcohols has been studied. This transformation provides a versatile and direct strategy for the construction of dihydrobenzofurans and dihydroindoles from easily accessible starting materials.

Keywords: Propargylic alcohols, Meyer-Schuster rearrangement, intramolecular Friedel-Crafts reaction, dihydrobenzofurans, dihydroindoles

Introduction

Both dihydrobenzofuran and dihydroindole moieties are important structural unit frequently found in many natural and synthetic compounds that exhibit a variety of biological properties.[1,2] Although several general methods for their synthesis have been developed,[3-12] they often suffer from the use of expensive transition-metal catalysts or toxic reagents and multistep preparation of the starting materials. Hence, efficient and general methods for the synthesis of both dihydrobenzofurans and dihydroindoles from easily accessible starting materials using inexpensive and low-toxicity catalysts are still needed.

Propargylic alcohols are uniquely versatile intermediates in organic synthesis because of their easy generation and multifarious reactions.[13-19] One of the most important applications of propargylic alcohols is the synthesis of allenes by the coupling of a nucleophiles with allenic carbocation intermediates, which were generated *in situ* by the Meyer-Schuster rearrangement. The allenes generated *in situ* could then undergo various cascade reactions to form many useful structural frameworks.[20-26] In a recent report, the Zhou group have developed an intramolecular Friedel-Crafts (IFC) reaction that provides a facile and versatile method for one-step construction

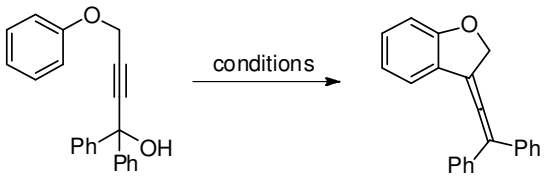
of a variety of six membered ring skeletons from benzylamino- or aryl-substituted propargylic alcohols.[27,28] Very recently, our group has reported the application of propargylic alcohols for the construction of substituted fluorenes, in which five-membered rings were constructed by IFC reactions.[29] It was envisaged that the similar IFC reaction of propargylic alcohols may also be applied to construct a five-membered heterocyclic skeleton. Herein, we report a concise and versatile route to the synthesis of dihydrobenzofurans and dihydroindoles by $FeCl_3$-catalyzed IFC cyclizations of aryloxy- and arylamino-substituted propargylic alcohols.

Results and Discussion

We began our investigation with the IFC reaction of **1a** with 1 equiv. of various Lewis acids in the presence of 4Å molecular sieve (MS) in dioxan at 40 ^oC (Table 1, entries 1-7), $FeCl_3$ was proven to be optimal, albeit with a low yield (entry 1). Brønsted acids, such as TsOH and TFA were also tested, but no desired product was detected (entries 8 and 9). A series of other desiccants, such as CaH_2 and $MgSO_4$, were also tested (entries 10, 11), and they displayed higher efficiency for the reaction. No significant formation of **2a** was detected when other solvents, including $MeNO_2$, toluene, MeCN and DCM were used (entries 12-15). Decreasing the catalyst from 1 equivalent to 20 mol% had a drastic effect as only 14% of **2a** was isolated (entry 16), indicating that stoichiometric catalyst was required for the transformation to occur. However, the yield slightly decreased when the reaction using 1.2 molar equivalent of $FeCl_3$ (entry 17). In the cases in which the product is not detected or it is obtained in lower yield, the reactions give a large amount of unidentified side-products.

With the optimized reaction conditions in hand, the scope of the $FeCl_3$-catalyzed IFC reaction was examined. As shown in Table 2, aryloxy-substituted propargylic alcohols bearing electron-donoring Me and MeO substitution at the phenolic position gave yields of 69% and 36% respectively (Entries 2, 3). Introduction of moderately electron-withdrawing Cl on the *para*-position of phenolic position was well tolerated and the product was obtained in 67% yield (entry 4). It is noteworthy that our findings sharply differ from the results reported by the Zhou group, in which benzylamino-substituted propargylic alcohols bearing a chloride group resulted in the isolation of the Meyer-Schuster rearranged ene-ketone as the main product.[27,28] It was also found that the substituents of arenes at the propargylic position play a significant role on the reactivity. By using propargylic alcohol bearing *p*-Me group, the reaction gives only a mixture of unidentified side-products, while the *p*-Cl substituted propargylic alcohol afforded a yield of 34% (entries 5, 6). When one of the R^2 is an aliphatic substituent, no product was obtained (entry 7).

Table 1. Optimization of reaction conditions for access to **2a** [a]

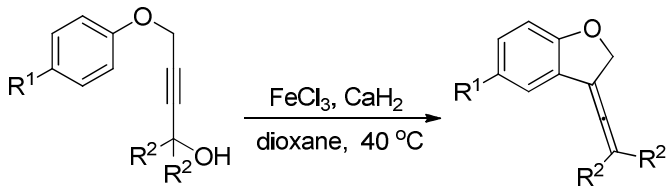


1a **2a**

Entry	Catalyst	Solvent	Desiccant	Yield (%) [b]
1	$FeCl_3$	dioxan	4Å MS	25
2	$TiCl_4$	dioxan	4Å MS	15
3	$BF_3{\cdot}Et_2O$	dioxan	4Å MS	13
4	$AlCl_3$	dioxan	4Å MS	nd
5	$Sc(OTf)_3$	dioxan	4Å MS	trace
6	$Sm(OTf)_3$	dioxan	4Å MS	nd
7	$ZnBr_2$	dioxan	4Å MS	nd
8	TsOH	dioxan	4Å MS	nd
9	TFA	dioxan	4Å MS	nd
10	$FeCl_3$	dioxan	CaH_2	59
11	$FeCl_3$	dioxan	$MgSO_4$	47
12	$FeCl_3$	$MeNO_2$	CaH_2	nd
13	$FeCl_3$	toluene	CaH_2	nd
14	$FeCl_3$	MeCN	CaH_2	8
15	$FeCl_3$	DCM	CaH_2	nd
16	$FeCl_3$ [c]	dioxan	CaH_2	14
17	$FeCl_3$ [d]	dioxan	CaH_2	53

[a] Reaction conditions: propargylic alcohol **1a** (0.2 mmol), Lewis acid (0.2 mmol) and desiccant in solvent (2 mL) at 40 °C for 4 h under N_2.
[b] Isolated yield, nd = not detected.
[c] 20 mol% of catalyst.
[d] 120 mol% of catalyst.

Table 2. Cyclization reactions of aryl- substituted propargylic alcohols [a]



1 **2**

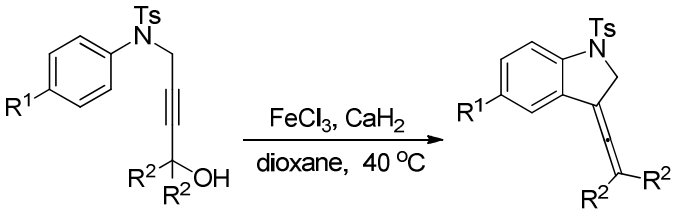
Entry	Products	Yield (%)[b]
1	**2a** R^1 = H, R^2 = Ph	59
2	**2b** R^1 = Me, R^2 = Ph	69
3	**2c** R^1 = OMe, R^2 = Ph	36
4	**2d** R^1 = Cl, R^2 = Ph	67
5	**2e** R^1 = H, R^2 = *p*-MeC_6H_4	nd
6	**2f** R^1 = H, R^2 = *p*-ClC_6H_4	34
7	**2g** R^1 = H, R^2 = Me/Ph	nd

[a] Reaction conditions: propargylic alcohols **1** (0.2 mmol), $FeCl_3$ (0.2 mmol) and CaH_2(12 mg) in dioxan (2 mL) at 40 °C for 4 h under N_2.
[b] Isolated yield; nd = not detected.

To expand the utility of this methodology, several arylamino-substituted propargylic alcohols were also examined under the optimal conditions (Table 3). Generally, they exhibited higher reactivity than aryloxy-substituted propargylic alcohols. Good to excellent yields were obtained for these substrates with Cl, Me and MeO substituents (entries 2-4). Substrate with Br substituent was also subjected to the process, thus affording the dihydroindole **4e** with 82% yield (entry 5). Similar trends in the reactivity were observed with the substituents of arenes at propargylic position. No desired product was isolated in the case of propargylic alcohol with Me substituent, while propargylic alcohol with F substituent can provide the desired product in 52% yields (entries 6-7).

On the basis of previous reports[5] and our experimental results, a plausible mechanism is outlined in Scheme 1. Propargyl alcohols **1** and **3** are first converted into the allenylic cations **I** in the presence of Lewis acid *via* a Meyer-Schuster rearrangement.[4] Then, benzene ring acts as the nucleophile to trap the cation **I** and form the corresponding dihydrobenzofuran and dihydroindole.

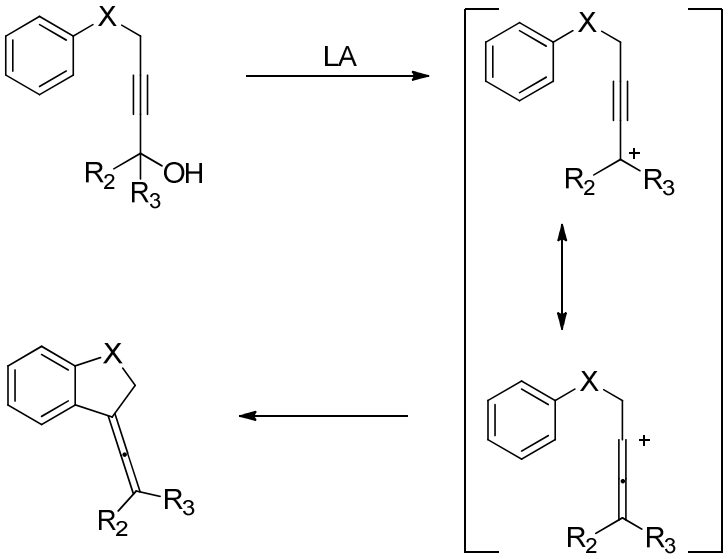
Table 3. Cyclization reactions of arylamino-substituted propargylic alcohols [a]



3 **4**

Entry	Products	Yield (%)[b]
1	**4a** R^1 = H, R^2 = Ph	61
2	**4b** R^1 = Me, R^2 = Ph	75
3	**4c** R^1 = OMe, R^2 = Ph	94
4	**4d** R^1 = Cl, R^2 = Ph	80
5	**4e** R^1 = Br, R^2 = Ph	82
6	**4f** R^1 = H, R^2 = *p*-MeC_6H_4	nd
7	**4g** R^1 = H, R^2 = *p*-FC_6H_4	52

[a] Reaction conditions: propargylic alcohols **3** (0.2 mmol), $FeCl_3$ (0.2 mmol) and CaH_2 (12 mg) in dioxan (2 mL) at 40 °C for 4 h under N_2.
[b] Isolated yield, nd = not detected.



4 or **5** **(I)**

Scheme 1. Proposed mechanism for intramolecular Friedel-Crafts reaction.

Conclusions

In summary, we report a facile and versatile method for synthesis of dihydrobenzofuran and dihydroindole derivatives *via* $FeCl_3$ catalyzed intramolecular Friedel-Crafts cyclizations of aryloxy- and arylamino-substituted propargylic alcohols. Further experiments on the derived dihydrobenzofuran and dihydroindole and the construction of other five-membered and seven-membered ring skeletons using this protocol are in progress.

Experimental Section

General. All organic solvent were pre-dried prior to use. The ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C in $CDCl_3$ at 500 and 125 MHz, respectively, with TMS as the internal standard. Chemical shifts (δ) are expressed in ppm and coupling constants *J* are given in Hz. High resolution mass spectra (HRMS) were obtained on a TOF MS instrument with ESI source. The propargylic alcohols were synthesized according to literature method.[30]

General experimental procedure and spectroscopic data. In a Schlenk tube, propargylic alcohols **1** or **3** (0.2 mmol), $FeCl_3$ (0.2 mmol) and CaH_2 (12 mg) were added and charged with N_2 three times. Anhydrous dioxan (2 mL) was then added. After being stirred at 40 ^oC for 4 h, the mixture was diluted with DCM (20 mL), washed with water and saturated brine, dried over Na_2SO_4, and then evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (eluent: light petroleum/ ethyl acetate = 60 : 1) to give the products.
3-(2,2-Diphenylvinylidene)-2,3-dihydrobenzofuran (2a). Pale yellow solid; ^1H NMR: δ 7.32 (m, 5H), 7.26 – 7.24 (m, 4H), 7.16 (dd, *J* 8.5, 7.5 Hz, 2H), 6.92 – 6.89 (m, 3H), 4.85 (s, 2H); ^{13}C NMR: δ 201.3, 157.7, 134.9, 129.4, 129.1, 128.6, 128.5, 121.6, 119.8, 115.5, 103.0, 69.3; HRMS (ESI): *m/z* $[M+H]^+$calcd for $C_{22}H_{17}O$: 297.1274; found: 297.1275; IR (KBr) *v*/cm^{-1} 3061, 2962, 1951, 1691, 1595, 1490, 1446, 1367, 1210, 1088, 700.
3-(2,2-Diphenylvinylidene)-5-methyl-2,3-dihydrobenzofuran (2b). Yellow solid; ^1H NMR: δ 7.29 – 7.28 (m, 5H), 7.24 – 7.22 (m, 4H), 6.92 (d, J 8.3 Hz, 2H), 6.80 – 6.77 (m, 2H), 4.79 (s, 2H), 2.21 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 201.3, 155.5, 134.9, 130.8, 129.9, 129.0, 128.5, 128.4, 119.6, 115.6, 103.1, 69.6, 20.5; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H1_9O$: 311.1430; found: 311.1433; IR (KBr) *v*/cm^{-1} 3063, 2914, 1948, 1604, 1587, 1511, 1453, 1367, 1237, 1040, 697.
3-(2,2-Diphenylvinylidene)-5-methoxy-2,3-dihydrobenzofuran (2c). Yellow oil; ^1H NMR: δ 7.32 (m, 5H), 7.22 (dd, J 6.7, 3.0 Hz, 4H), 6.86 – 6.85 (m, 2H), 6.69 – 6.68 (m, 2H), 4.80 (s, 2H), 3.69 (s, 3H); ^{13}C NMR: δ 200.5, 153.5, 150.6, 133.9, 128.0, 127.5, 127.4, 118.4, 116.2, 113.5, 102.1, 69.6, 54.6; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{19}O_2$: 327.1380; found: 327.1383; IR (KBr) *v*/cm^{-1} 3059, 2962, 1954, 1695, 1589, 1505, 1444, 1367, 1203, 1032, 699.

5-Chloro-3-(2,2-diphenylvinylidene)-2,3-dihydrobenzofuran (2d). Pale yellow solid; ^{1}H NMR: δ 7.34 (m, 5H), 7.24 – 7.22 (m, 4H), 7.04 – 7.03 (m, 2H), 6.79 – 6.78 (m, 2H), 4.82 (s, 2H); ^{13}C NMR: δ 201.2, 156.0, 134.7, 129.3, 128.9, 128.7, 128.5, 126.5, 120.1, 116.9, 102.4, 69.4; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{16}ClO$: 331.0884; found: 331.0887; IR (KBr) *v*/cm^{-1} 3057, 2925, 1946, 1690, 1589, 1496, 1448, 1367, 1221, 1060, 696.

3-[2,2-Bis(4-chlorophenyl)vinylidene]-2,3-dihydrobenzofuran (2f). Pale yellow solid; ^{1}H NMR: δ 7.29 – 7.27 (m, 4H), 7.16 (dd, J 8.6, 7.4 Hz, 2H), 7.12 – 7.10 (m, 3H), 6.93 (t, *J* 7.4 Hz, 1H), 6.88 (dd, *J* 8.6, 0.8 Hz, 2H), 4.84 (s, 2H); ^{13}C NMR: δ 201.1, 157.4, 134.8, 133.1, 130.2, 129.5, 128.8, 121.7, 118.2, 115.4, 103.7, 68.8; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{15}Cl_2O$: 365.0494; found: 365.0498; IR (KBr) *v*/cm^{-1} 3059, 2931, 1951, 1694, 1592, 1509, 1457, 1367, 1232, 1059, 697.

3-(2,2-Diphenylvinylidene)-1-tosylindoline (4a). White solid; ^{1}H NMR: δ 7.54 (d, *J* 8.3 Hz, 2H), 7.29 – 7.25 (m, 8H), 7.17 (dd, *J* 9.9, 3.5 Hz, 2H), 7.08 (dd, *J* 9.6, 7.7 Hz, 6H), 4.59 (s, 2H), 2.43 (s, 3H); ^{13}C NMR: δ 202.2, 143.6, 138.3, 135.8, 134.7, 129.4, 129.2, 129.0, 128.9, 128.4, 128.1, 127.7, 119.0, 102.7, 55.5, 21.5; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{29}H_{24}NO_2S$: 450.1522; found: 450.1525; IR (KBr) *v*/cm^{-1} 3057, 2914, 1956, 1597, 1492, 1452, 1347, 1230, 1162, 1109, 695.

3-(2,2-Diphenylvinylidene)-5-methyl-1-tosylindoline (4b). White solid; ^{1}H NMR: δ 7.53 (d, *J* 8.2 Hz, 2H), 7.28 – 7.23 (m, 7H), 7.06 – 7.04 (m, 4H), 6.91 (d, *J* 5.8 Hz, 4H), 4.53 (s, 2H), 2.40 (s, 3H), 2.28 (s, 3H); ^{13}C NMR: δ 202.3, 143.5, 138.0, 135.7, 135.4, 134.6, 129.7, 129.4, 128.9, 128.8, 128.4, 128.3, 127.7, 118.7, 102.6, 55.4, 21.5, 21.2; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{30}H_{26}NO_2S$: 464.1679; found: 464.1681; IR (KBr) *v*/cm^{-1} 3030, 2922, 1957, 1599, 1501, 1448, 1351, 1230, 1164, 1107, 697.

3-(2,2-Diphenylvinylidene)-5-methoxy-1-tosylindoline (4c). White solid; ^{1}H NMR: δ 7.53 (d, *J* 8.2 Hz, 2H), 7.25 – 7.23 (m, 7H), 7.05 – 7.03 (m, 4H), 6.92 (d, *J* 8.9 Hz, 2H), 6.65 (d, *J* 8.9 Hz, 2H), 4.52 (s, 2H), 3.73 (s, 3H), 2.39 (s, 3H); ^{13}C NMR: δ 202.3, 159.2, 143.4, 135.8, 134.6, 130.5, 130.4, 129.3, 128.8, 128.3, 128.2, 127.7, 118.6, 114.1, 102.5, 55.7, 55.2, 21.4; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{30}H_{26}NO_3S$: 480.1628; found: 480.1630; IR (KBr) *v*/cm^{-1} 3058, 2919, 1944, 1603, 1508, 1456, 1347, 1251, 1162, 1098, 696.

5-Chloro-3-(2,2-diphenylvinylidene)-1-tosylindoline (4d). Pale yellow solid; ^{1}H NMR: δ 7.50 (d, *J* 8.3 Hz, 2H), 7.29 – 7.22 (m, 7H), 7.06 (dd, *J* 8.4, 1.9 Hz, 6H), 6.94 – 6.93 (m, 2H), 4.53 (s, 2H), 2.39 (s, 3H); ^{13}C NMR: δ 202.3, 143.9, 136.5, 135.2, 134.4, 134.0, 130.2, 129.6, 129.2, 128.8, 128.6, 128.4, 127.6, 119.0, 102.1, 55.2, 21.5; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{29}H_{23}ClNO_2S$: 484.1133; found: 484.1136; IR (KBr) *v*/cm^{-1} 3058, 2925, 1946, 1597, 1488, 1454, 1354, 1223, 1165, 1099, 698.

5-Bromo-3-(2,2-diphenylvinylidene)-1-tosylindoline (4e). White solid; ^{1}H NMR: δ 7.50 (d, J 8.3 Hz, 2H), 7.31 – 7.27 (m, 5H), 7.23 – 7.18 (m, 4H), 7.06 (dd, *J* 8.0, 1.4 Hz, 4H), 6.88 – 6.86 (m, 2H), 4.53 (s, 2H), 2.39 (s, 3H); ^{13}C NMR: δ 202.4, 144.0, 137.12, 135.3, 134.5, 132.3, 130.6, 129.7, 128.9, 128.7, 128.5, 127.7, 122.2, 119.1, 102.1, 55.2, 21.6; HRMS (ESI): *m/z* $[M+H]^+$

calcd for $C_{29}H_{23}{}^{79}BrNO_2S$: 528.0627; found: 528.0631; IR (KBr) *v*/cm^{-1} 3037, 2921, 1948, 1596, 1486, 1458, 1350, 1220, 1157, 1099, 697.

3-[2,2-Bis-(4-fluorophenyl)vinylidene]-1-tosylindoline (4g). White solid; ^{1}H NMR: δ 7.51 (d, *J* 8.3 Hz, 2H), 7.25 – 7.22 (m, 3H), 7.15 (t, *J* 7.7 Hz, 2H), 7.05 – 7.00 (m, 5H), 6.96 – 6.93 (m, 4H), 4.55 (s, 2H), 2.41 (s, 3H); ^{13}C NMR: δ 201.8, 163.8, 161.8, 143.7, 138.3, 135.6, 130.6, 130.6, 130.6, 129.5, 129.0, 129.0, 128.1, 127.7, 117.4, 115.6, 115.4, 103.2, 55.3, 21.5; HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{29}H_{22}F_2NO_2S$: 486.1334; found: 486.1337; IR (KBr) *v*/cm^{-1} 3051, 2926, 1954, 1597, 1481, 1447, 1357, 1232, 1161, 1102, 695.

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