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Synthesis of Indenes by a BF₃·OEt₂-Mediated, One-Pot Reaction of Aryl Homopropargyl Alcohols, Aldehydes, and Arenes

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ABSTRACT: A new and efficient protocol to prepare indenes is reported. Assisted by boron trifluoride diethyl etherate, the one-pot reaction of aryl homopropargyl alcohols and aldehydes in the presence of arenes yielded indene derivatives. The reaction was studied with various substrates, which suggests a cascade reaction including a sequence of Prins, Friedel-Crafts, ring-opening reactions, and Friedel-Crafts to form three C-C bonds leading to indenes.

The indene framework is frequently found in natural products, pharmaceutically active compounds, functional molecules, and ligands for metal complexes. It is not surprising that syntheses of indenes and their derivatives continue to receive chemists' attention, and many methods have been developed to prepare this important carbocycle, which include the dehydration of indanols,6 intramolecular cyclization of conjugated alkenes or alkynes, arene cyclization involving various metal-catalyzed C-H activations,8 Brønsted or Lewis acid catalyzed intramolecular Friedel-Crafts cyclization of aryl-allylic carbocations, ^{6a,9} and various cascade reactions using propargyl alcohols. ¹⁰ Indenes have also been prepared by Lewis acid catalyzed cycloaddition reactions of arylidenecyclopropanes with aldehydes, acetals, or aldimines (eq 1). Here, we report that indene derivatives could also be

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prepared from homopropargyl alcohols, aldehydes and arenes, promoted by boron trifluoride diethyl etherate at room temperature (eq 2). The reactions between homopropargyl alcohols and aldehydes are known to form furans (eq 3)12 via Lewis acid catalyzed Prins cyclizations. 13,14 In the presence of aromatic solvents and boron trifluoride diethyl etherate, we found that the cascade reaction was further extended to yield indenes.

Our investigation started with the reaction of 4-phenylbut-3yn-1-ol (1a) and benzaldehyde (2a) in benzene and the presence of BF₃·OEt₂. The reaction was complete in 1 h

according to TLC analysis and the spectroscopic data (IR and ¹H, ¹³C NMR) of the major product were consistent with the reported indene derivative 3a with a 30% isolated yield. 11b The structural assignment was also confirmed by X-ray crystallography (see the Supporting Information for the ORTEP of 3a). In addition to the moieties of 1a and 2a, the incorporation of a benzene molecule into product 3a was interesting and prompted us toward further study. Several reaction factors, such as the stoichiometry of boron trifluoride diethyl etherate, Lewis acids and reaction time were screened. The results are summarized in Table 1. The yields of 3a increased as the amount of BF₃·OEt₂ increased (entries 1-4); however, more impurities were observed when 5 equiv of BF3·OEt2 were applied. Prolonging the reaction time did not improve the yield (entry 5). The reaction also proceeded to yield 3a with 10 equiv of benzene in CH_2Cl_2 (entry 6) or was promoted by trimethylsilyl triflate and

Table 1. Optimization of the Reaction Conditions

entry ^a	Lewis acid	equiv	time (h)	yield ^b (%)
1	$BF_3 \cdot OEt_2$	1.0	1	30
2	$BF_3 \cdot OEt_2$	2.0	1	40
3	$BF_3 \cdot OEt_2$	3.0	1	64
4	$BF_3 \cdot OEt_2$	5.0	1	68
5	$BF_3 \cdot OEt_2$	3.0	2	66
6	$BF_3 \cdot OEt_2$	3.0	1	28 ^c
7	TMSOTf	3.0	12	26
8	TfOH	3.0	2	19
9	$FeCl_3$	3.0	12	0^d

^aReactions were conducted with 1a (0.25 mmol), 2a (0.28 mmol) in benzene (1 mL). ^bIsolated yields. ^cReactions were conducted with 1a (0.25 mmol), 2a (0.28 mmol), benzene (2.5 mmol) in CH₂Cl₂ (1 mL). ^dUnidentified mixture.

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trifluoromethanesulfonic acid; however, the yields were lower (entries 6–8). The reaction using iron(III) chloride was sluggish to yield a complicated mixture (entry 9). Other Lewis acids, such as CF₃CO₂H, InCl₃, Fe(OTf)₂, and Zn(OTf)₂, failed to induce the reactions, and the starting materials were recovered

Various aldehydes were applied to examine the reaction scope with this optimized reaction condition (3 equiv of BF₃·OEt₂ in benzene, 0 °C to rt, Table 2). All of the para-substituted benzaldehydes provided the desired indene products (3b-i, entries 1-8) with moderate to good reaction yields (55-90%), which indicate that the reaction is not sensitive to the electronic properties of aldehydes imposed by the para-substituents. The ortho-substituted benzaldehydes also gave satisfactory yields (3j-l, entries 9-11), and the reaction was not deterred by the sterically more hindered 2,6-dichlorobenzaldehyde (entry 13). Although 3-methoxybenzaldehyde provided a fair yield (49%, entry 12), slightly better results were observed for other 3alkoxy-substituted benzaldehydes (entries 14 and 15). Other aromatic aldehydes, such as cinnamaldehyde and thiophene-2carbaldehyde, were also good substrates and converted to the corresponding indenes (3q and 3r, entries 16 and 17). Aliphatic aldehydes were compatible for this process (entries 18-21); however, their yields decreased as the steric hindrance around the carbaldehyde group increased. Overall, many aryl and alkyl aldehydes could be converted to indenes by this simple, threecomponent reaction with fair to good yields.

In contrast to the high tolerance of aldehydes, we found that this reaction is sensitive to the substituents on the homopropargyl alcohols (Table 3). The reactions of 4-(4chlorophenyl)-3-butyn-1-ol (1b) with three aldehydes 2d, 2j, and 2p, provided the indenes 3bd, 3bj, and 3bp, respectively (entries 1-3). The location of the chloro substituent was unambiguously determined by X-ray crystallography of 3bj (Figure 1). However, the reaction of methyl-substituted alcohol 1c gave a pair of regioisomers 3cg and 3cg', which suggests a competition between the phenyl and tolyl groups during the formation of the indene moiety (vide infra). Interestingly, alcohol 1d bearing an electron-donating methoxy group afforded the known conjugated enone 4,15 derived from a formal alkyne-carbonyl metathesis of 1d and benzaldehyde. 16 No indene was generated from the reaction of the homopropargyl alcohol bearing an electron-deficient p-nitrobenzene (1e, entry 6). The inertness of aliphatic 3-pentynol (1f) and methyl ether 1g under this condition (entries 7 and 8) indicates that the aromatic alkynyl moiety and the hydroxyl group are essential for the reaction.

On the basis of the above results and previous studies, 12b,c a plausible mechanism for the formation of indenes is proposed (Scheme 1). Boron trifluoride initiated the reaction of homopropargyl alcohol and aldehyde to form the oxonium cation A. The following Prins-type cyclization yielded the exocyclic vinyl cation B, which was the electrophile for the Friedel-Crafts reaction of benzene and the precursor of intermediate C. The equilibrated, ring-opening rearrangement gave the allyl carbocation **D**, which was intercepted by the more electron-rich aryl group to produce thermodynamically stable indene 3. Many aldehydes that can form oxonium cation A and allow the following Prins-type cyclization are compatible with this process. On the other hand, the R² substituent has a direct influence on the formation of vinyl cation B. Thus, the reaction was obstructed by an electron-withdrawing R² group, such as the nitro group of 1e (entry 6, Table 3); however, the electron-

Table 2. Synthesis of Indene with Various Aldehydes

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entry ^a	R¹/aldehyde	product	yield (%) ^b
1	4-Me, 2b	3b	85
2	4- <i>i</i> Bu, 2c	3c	76
3	4-OMe, 2d	3d	84
4	4-SMe, 2e	3e	55
5	4-F, 2f	3f	75
6	4-Cl, 2g	3g	90
7	4-Br, 2h	3 h	77
8	4- NO ₂ , 2i	3i	71
9	2-F, 2 j	3j	70
	2-1', 2 j 2-Cl, 2k		60
10		3k	
11	2-Br, 21	31	77
12	3-OMe, 2m	3m	49
13	CI CHO	Ph —OH	84
	Cl 2n	CI	
		CI	
		∭ 3n	
14	MeO	Ph —OH	61
	20 Br		
		30	
		Br	
		OMe	
15	ρ ΥΥ CHO	Ph —OH	70
	Br 2p		
	.	3p	
		Br	
	. 111 1	`O → Ph	=0
16	cinnamaldehyde	OH	78
	(2q)		
		3q Ph	
17	∠S CHO	Рh	50
	\ // 2r	_OH	
		∑ _S 3r	
18	propionaldehyde		45^{c}
	(2s)		
		_OH	
		∨ ∖ 3s	
19	decanal (2t)		57°
		∠ ∠ OH	
		3t	
		n-C ₉ H ₁₉	
20	CHO		31°
	2u		
	•	√-OH	
		Cy 3u	
21	> cho	-	O^c
	2v		

"Reactions were conducted with 1a (0.25 mmol), aldehyde (0.28 mmol), and BF $_3$ ·OEt $_2$ (0.75 mmol) in benzene (1 mL). ^bIsolated yields. ^cReaction time: 12 h.

donating methoxy group seems to stabilize intermediate **B** well and thwart the following Fridedel—Crafts reaction to give 4 (entry 5, Table 3). The reaction using 4-methyl-substituted 1c proceeded but gave a mixture of regioisomers due to the

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Table 3. Synthesis of Indene with Substituted Alcohols

^aReactions were conducted with 1a (0.25 mmol), aldehyde (0.28 mmol) in benzene (1 mL). ^bIsolated yields. ^cUnidentified mixture. ^dStarting materials recovered.

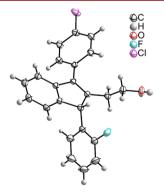


Figure 1. ORTEP of 3bj.

competition between the two aryl groups in the intramolecular Friedel—Crafts-type cyclization of $\bf D$ to form indenes $\bf 3cg$ and $\bf 3cg'$ (entry 4, Table 3). The issue of competition was relieved in $\bf 1a$ ($\bf R^2 = \bf H$) and the chloro-substituted $\bf 1b$ ($\bf R^2 = \bf Cl$), which gives a symmetrical $\bf D$ or has a deactivated chlorophenyl group for the Friedel—Crafts-type reaction, respectively.

The above mechanism was also supported by the results obtained from the reactions performed in p-xylene or 1,4-dimethoxybenzene/CH₂Cl₂ (Scheme 2). The 1 H and 13 C NMR spectra indicated that the reaction in p-xylene provided a pair of regioisomers (3w and 3w'), and the reaction in 1,4-

Scheme 1. Proposed Mechanism for the Formation of indene 3

Scheme 2. Reactions Conducted in Different Solvents

$$\begin{array}{c} Ph & Ph & Ph \\ \hline Ph & Ph & Ph \\ \hline 1a & Ph & Ph \\ \hline 1a & Ph & Ph \\ \hline 2a & Ph & Ph \\ \hline Ph & Ph & Ph \\ \hline Ph & Ph & Ph \\ \hline 1a & Ph & Ph \\ \hline Ph & Ph & Ph \\ P$$

dimethoxybenzene gave single product 3x. The results are consistent with the relative reactivity of the corresponding aryl groups toward to the allyl cation in the intermediates D1 and D2. No indene product could be found when the reaction was carried out in bromobenzene.

In summary, a new approach to access indenes is developed through a sequence of Prins, Friedel—Crafts, ring-opening and Friedel—Crafts reactions including three C—C bond formations. This method has the advantages of broad substrate scope in aldehydes, readily available substrates and a metal-free, simple one-pot procedure. The intermolecular Friedel—Crafts reaction between the vinyl cations and arenes is especially attractive because the effort needed to prepare the highly strained arylidenecyclopropanes is circumvented. Further studies on the tandem Prins/Friedel—Crafts reactions and their application in synthesis are in progress.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01929.

Experimental procedures and spectral data of compounds described herein (PDF)

Accession Codes

CCDC 1846842 and 1847193 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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