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Iodine/Et_3SiH: a novel reagent system for the synthesis of 3-aryl-1*H*-indenes from 1,3-diaryl propargyl alcohols

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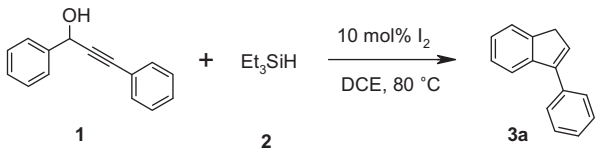
ABSTRACT

1,3-Diaryl propargyl alcohols undergo smooth intramolecular Friedel–Crafts cyclization with triethylsilane in the presence of 10 mol % iodine 3-aryl-1*H*-indene derivatives in good yields in short reaction times. This is the first example on the synthesis of substituted indenes from 1,3-diaryl propargyl alcohols. The use of inexpensive and readily available molecular iodine makes this method quite simple, more convenient, and practical.

The chemistry of alcohols occupies a vital role in organic synthesis. In particular, 'π-activated' alcohols are attracted as proelectrophiles, capable of reacting with various nucleophiles, and their ability to undergo nucleophilic substitution reactions contributes largely to their synthetic value.[1] The use of 'π-activated' alcohols such as benzylic, propargylic, and allylic alcohols makes the C–OH bond activation easier via the formation of stabilized positively charged intermediates.[2] Notably, indene scaffolds are attractive targets for the synthesis of some biologically active molecules.[3] Consequently, transition metal-catalyzed approaches such as Ni- and Co-catalyzed carboannulation of alkynes with *o*-halophenyl aldehydes or *o*-iodophenyl malonates, rhenium-,[4] and palladium-[5] catalyzed annulations and gold(I)-catalyzed intramolecular carboalkoxylations[6] have been introduced for the synthesis of indene derivatives. In addition, substituted indene derivatives were prepared via the Pd-catalyzed annulation of alkynes,[7] and palladium-catalyzed carboannulation of internal alkynes by substituted aryl halides.[8] Subsequently, Lewis acid-catalyzed ring expansion of substituted cyclopropanes and cyclopropenes,[9] and intramolecular hydroarylation of phenyl-substituted alkenes[10] have also been reported for the preparation of indene scaffolds. Recently, $FeCl_3$ has also been utilized to accomplish the synthesis of indenes.[11] Though these methods are quite effective for the synthesis of simple indenes; they have certain drawbacks in the preparation of highly substituted indenes. Many of these methods involve multi-step reaction sequences[12] and often require expensive metal catalysts and strong acidic conditions. Therefore, the development of a simple, convenient, and metal-free catalytic system for the synthesis of indene scaffolds is highly desirable.

Recently, molecular iodine has received considerable interest in organic synthesis because of its low cost and ready availability. The mild Lewis acidity associated with iodine has enhanced its use in organic synthesis to perform several organic transformations using stoichiometric levels to catalytic amounts.[13] A catalytic amount of iodine is able to activate the hydroxyl group and the elimination processes are usually accompanied by rearrangements or intramolecular cyclizations.

Following our interest in the catalytic uses of iodine,[14] we herein, report a direct one-pot method for the synthesis of indenes via an intramolecular Friedel–Crafts cyclization of aryl-substituted propargylic alcohols. Initially, we attempted the deoxygenation of diaryl-substituted propargyl alcohols with triethylsilane using a catalytic amount of molecular iodine. Interestingly, indene derivatives were formed instead of the expected deoxygenation. Thus, treatment of 1,3-diphenyl-2-propyn-1-ol (**1**) with triethylsilane (**2**) in the presence of 10 mol % molecular iodine in 1,2-dichloroethane at 80 °C for 1 h gave the corresponding 3-phenyl-1*H*-indene **3a** in 85% yield (Scheme 1).



Scheme 1. Preparation of 3-phenyl-1*H*-indene.

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Table 1
Molecular iodine-catalyzed cyclization of propargyl alcohols with triethylsilane

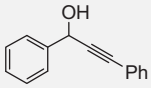
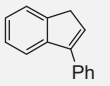
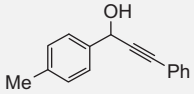
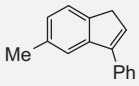
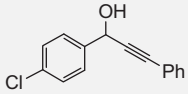
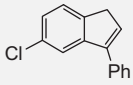
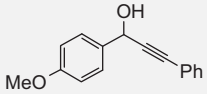
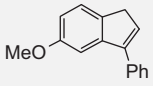
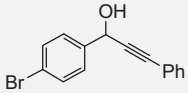
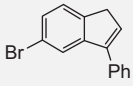
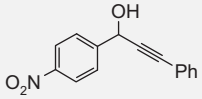
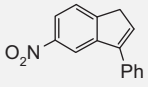
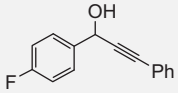
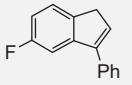
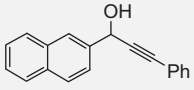
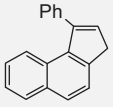
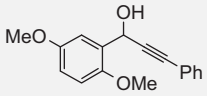
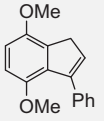
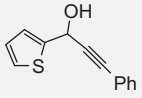
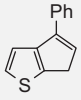
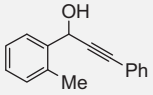
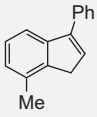
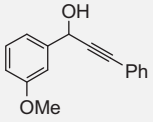
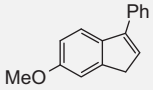
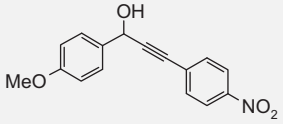
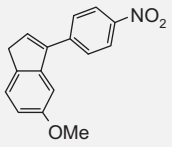
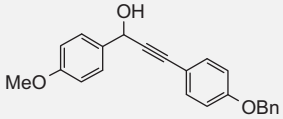
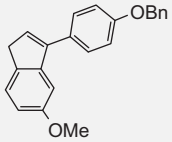
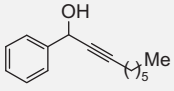
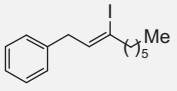
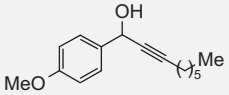
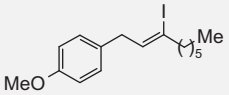
Entry	Propargyl alcohol (**1**)	Triethylsilane (**2**)	Product (**3**)[a]	Time (h)	Yield (%)[b]
a		Et_3SiH		1.0	85
b		Et_3SiH		0.5	86
c		Et_3SiH		1.5	80
d		Et_3SiH		0.5	88
e		Et_3SiH		1.5	82
f		Et_3SiH		2.0	78
g		Et_3SiH		1.5	80
h		Et_3SiH		1.5	84
i		Et_3SiH		1.0	82
j		Et_3SiH		1.5	85
k		Et_3SiH		2.0	75
l		Et_3SiH		1.5	82
m		Et_3SiH		2.0	80
n		Et_3SiH		1.5	85
o		Et_3SiH		2.5	80[c]

Table 1 (*continued*)

Entry	Propargyl alcohol (**1**)	Triethylsilane (**2**)	Product (**3**)[a]	Time (h)	Yield (%)[b]
p		Et_3SiH		2.0	78[c]

[a] All products were charecterized by NMR, IR, and mass spectroscopy.
[b] Yield refers to pure products after chromatography.
[c] Stoichiometric amount of iodine was used.

The starting secondary propargyl alcohols were prepared using known procedures.[15] Encouraged by this result; we turned our attention to various substituted propargylic alcohols. Interestingly, a wide range of substituted aryl propargyl alcohols participated well in this reaction (entries b–g, Table 1). Notably, sterically hindered propargyl alcohols such as 2,5-dimethoxyphenyl and 2-naphthyl derivatives were also effective for this conversion (entries h and i, Table 1). Furthermore, the propargyl alcohol derived from thiophene-2-carboxaldehyde also gave the product in good yield (entry j, Table 1). To know the effect of *ortho*-substituent, the reaction was performed with the propargyl alcohol derived from *o*-tolualdehyde and phenylacetylene (entry k, Table 1). It was observed that the cycloannulation of *o*-tolyl propargyl alcohol requires a long reaction time when compared to *p*-tolylpropargyl alcohol (entry b, Table 1). This may be due to the steric factors of the *ortho*-substituent (entry k, Table 1).

To further evaluate the role of the substituents on this reaction, several propargyl alcohols with different aryl substituents were subjected to the present reaction conditions. The reactions with substrates bearing electron- donating groups on the aromatic ring are faster than those of electron-deficient substrates (Table 1). To realize the regioselectivity, the reaction was performed with *m*-methoxyphenyl propargyl alcohol (entries l, Table 1). Interestingly, the cycloannulation was observed regioselectively at *para* to methoxy group (entries l, Table 1). Next, we attempted the reactions with secondary propargyl alcohols prepared from *p*-substituted phenyl acetylenes (entries m and n, Table 1). In the case of *p*-benzyloxysusbtituted propargyl alcohol (entry n, Table 1), the desired product was obtained in high yield in a shorter reaction time compared to *p*-nitrosubstituted one. Finally, we examined the reaction of triethylsilane with alkyl-substituted propargyl alcohols. However, vinyl iodides were obtained exclusively instead of the formation of indene derivatives when alkyl-substituted propargyl alcohols were treated with 1 equiv of iodine and 2 equiv of triethylsilane at 80 °C in dichloroethane (entries o and p, Table 1). The formation of indene was successful only with propargylic alcohols derived from aromatic aldehydes and phenylacetylenes. In all the cases, the yields are generally high and the reaction times are quite reasonable.

No indene formation was observed with alkyl-substituted propargyl alcohols. Similarly, no cycloannulation was observed with tertiary propargyl alcohols. The catalytic efficiency of various Lewis acids such as $BiCl_3$, $ZnCl_2$, and $FeCl_3{\cdot}6H_2O$ was tested for this reaction.

For instance, treatment of 1,3-diphenyl-2-propyn-1-ol (**1**) with triethylsilane (**2**) in the presence of 10 mol % of $FeCl_3{\cdot}6H_2O$, $BiCl_3$ or $ZnCl_2$ gave the 3-phenyl-1*H*-indene **3a** in 50%, 45%, and 35% yields, respectively. As a solvent, dichloroethane appeared to give the best results. However, in the absence of either iodine or triethylsilane, the reactions did not proceed even after 12 h. This clearly indicates that both molecular iodine and triethylsilane are essential to facilitate the reaction. The products were characterized by NMR, IR, and mass spectroscopy and also by comparison with authentic samples.[11] The scope and generality of this process is illustrated with respect to various propargylic alcohols and the results are presented in Table 1.[16]

In summary, we have developed a novel and efficient catalytic process for the synthesis of substituted indenes from aryl-substituted propargylic alcohols by means of a tandem isomerization, reduction followed by intramolecular Friedel–Crafts type cyclization.[17] The remarkable features of this procedure are high conversions, excellent control of geometry of olefins, operational simplicity, and ready availability of reagents at low cost.

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16. *Experimental procedure:* A mixture of triethylsilane (2.0 mmol), aryl propargyl alcohol (1.0 mmol) and iodine (10 mol %) in dichloroethane (5 mL) was stirred in 1,2-dichloroethane at 80 °C for the appropriate time (Table 1). After completion of the reaction as indicated by TLC, the mixture was diluted with water (10 mL) and extracted with dichloromethane (2 × 10 mL). The combined organic layers were washed with saturated $Na_2S_2O_3$ solution (2 × 10 mL) followed by brine (10 mL) solution and dried over anhydrous Na_2SO_4. Removal of solvent followed by purification on silica gel column chromatography using hexane as an eluent afforded the pure product. The spectral data of the products were identical with the data reported in the literature.[11] *Spectral data for selected products: Compound* **3a***: 3-phenyl-1H-indene:* pale yellow liquid, IR (neat): υ_{max} 3028, 2906, 1602, 1491, 1443, 1215, 1041 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.43–7.38 (m, 2H), 7.28–7.11 (m, 7H), 6.01 (t, J = 6.7 Hz, 1H), 3.63 (d, J = 6.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 142.8, 137.5, 135.8, 135.6, 129.2 (×2), 128.5 (×2), 128.3 (×2), 128.1, 128.0 (×2), 120.9, 43.6; ESI-MS: m/z: 193 [M+H]. *Compound* **3b***: 5-methyl-3-phenyl-1H-indene:* colorless liquid, IR (neat): υ_{max} 3017, 2917, 1510, 1440, 1215, 1051 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.41–7.30 (m, 2H), 7.20–6.94 (m, 6H), 5.92 (t, J = 6.7 Hz, 1H), 3.51 (d, J = 6.7 Hz, 2H), 2.19 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 142.8, 137.5 (×2), 135.8, 135.6, 129.2 (×2), 128.5, 128.3, 128.0–128.2 (×5), 43.6, 21.0; ESI-MS: m/z: 224 [M+NH_4]. *Compound* **3d***: 5-methoxy-3-phenyl-1H-indene:* pale yellow liquid, IR (neat): υ_{max} 2924, 2853, 1596, 1510, 1450, 1254, 1029 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.42–7.08 (m, 6H), 6.81–6.75 (m, 2H), 5.97 (t, J = 6.9 Hz, 1H), 3.71 (s, 3H), 3.54 (d, J = 6.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 158.2, 144.6, 142.9, 137.7, 130.8, 130.2, 129.4 (×2), 128.6, 128.2, 128.1 (×2), 114.3, 114.0, 55.2, 43.1; ESI-MS: m/z: 222 [M^+].
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