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A Tin(IV) Chloride Promoted Tandem C−O Bond Cleavage/Nazarov Cyclization/Nucleophilic Addition Reaction of 1,1-Disubstituted Allylic Ethers toward the Synthesis of Multisubstituted Indenes

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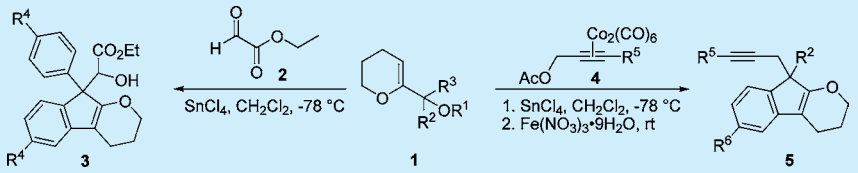
Supporting Information



ABSTRACT: A novel $SnCl_4$-promoted tandem reaction toward multisubstituted indenes via a sequential C−O bond cleavage/Nazarov cyclization/nucleophilic addition reaction has been developed to afford a series of multisubstituted indenes with an all-carbon quaternary center in moderate to good yields.

Multisubstituted indenes, as important skeletal moieties, broadly exist in a number of natural products and bioactive molecules, including phenindamine, phelligridin G, and wrightiadione, several of which have shown diverse biological activities including demonstrating antihistamine, antihypertension, and bronchodilation properties (Figure 1).[1]

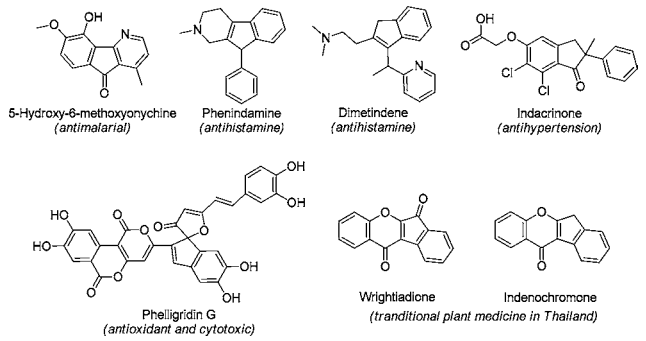
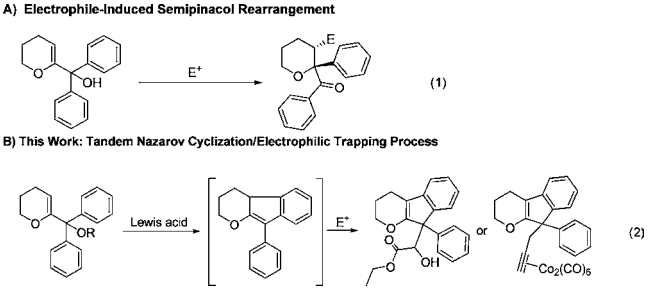


Figure 1. Representative natural products and medicines containing indene moieties.

Moreover, they also have been used as key intermediates in the synthesis of a variety of natural products and pharmaceuticals as well as special ligands in organometallic chemistry.[2] Because of the above-mentioned special utilities, development of corresponding synthetic methodologies for the construction of the indene skeleton has attracted the interest of organic and medicinal chemists for years. Accordingly, a variety of methods for the construction of polysubstituted indenes have been developed,[3−6] including the cyclization of phenyl-substituted allylic alcohols or phenylvinyl derivatives,[4] the ring expansion of suitable cyclopropene derivatives, and the dehydration/reduction of an indanone.[5] Recently, transition-metal-catalyzed cycloadditions of alkynes have been developed that lead to indene derivatives.[6] Despite the above achievements, it is also true that only very limited references are available for the synthesis of pyran-fused indene types of compounds, which are also a key moiety of bioactive molecules. Examples of these molecules include indenochromone and wrightiadione compounds (Figure 1),[7] which have been used as plant-based medicines in Thailand. Therefore, it is still desirable to further explore this topic.

Dihydropyran-type diphenylmethanol and its trimethylsilyl ether derivatives are a group of versatile synthetic intermediates, and they have been successfully applied to several electrophile-induced semipinacol rearrangement sequences,[8] which are very effective for the construction of compounds with a quaternary carbon stereocenter as well as the total synthesis of related natural products (Scheme 1, eq 1).[8,9] Furthermore, they also possess the advantage of easy accessibility, as these

Scheme 1. $SnCl_4$-Promoted Tandem Nazarov Cyclization/Electrophilic Trapping Process



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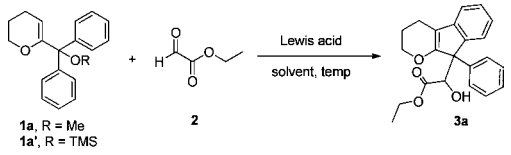
compounds could be readily prepared by the addition of dihydropyran to benzophenone in the presence of a strong base. Considering the above-mentioned features of this type of compound, we envision that, in the presence of a proper catalyst, a Nazarov type cyclization may occur to give an enol ether intermediate, which would go through a nucleophilic addition reaction to afford a pyran-fused indene product with an all-carbon quaternary center. In this paper, we report a novel method that delivers a diverse array of multisubstituted indenes from 1,1-disubstituted allylic ethers through a $SnCl_4$-promoted tandem intramolecular cyclization−electrophilic addition sequence. To our knowledge, this document describes the first robust and efficient protocol that uses Nicholas reagents or highly active aldehydes to prepare substituted indenes containing newly formed quaternary carbon centers through a tandem process (Scheme 1, eq 2).

Following the above-mentioned assumption, we first tested the feasibility of such a tandem reaction using 1,1-diphenylallyl silyl ether **1a′** (R = TMS) as the model substrate, which was prepared from a dihydropyran-type diphenylmethanol by trimethyl chlorosilane protection.[8] Initially, in the presence of different Lewis acids, the desired reaction between compounds **1a′** and **2** was tested with CH_2Cl_2 as the solvent at −78 °C. Like most Lewis acid catalyzed reactions, the use of the proper promoter is the key factor for such a tandem reaction. For example, when the most commonly used Lewis acids, i.e., $AlCl_3$ and $BF_3{\cdot}OEt_2$, were applied to the reaction, only the one promoted by $AlCl_3$ could give a trace amount of the expected product (Table 1, entry 1), which was not observed in the presence of $BF_3{\cdot}OEt_2$ (Table 1, entry 2). Similarly, only a small amount of product **3a** was detected with $EtAlCl_2$ as the promoter (Table 1, entry 3). Additionally, a very complex reaction mixture was obtained if $In(OTf)_3$ or TMSOTf was used (Table 1, entries 4 and 5). While different than the above-mentioned results, 30% to 60% yields of the desired product **3a** were obtained by using Lewis acids such as $Sc(OTf)_3$, $SnCl_4$, and $TiCl_4$ (Table 1, entries 6 to 8), among which $SnCl_4$ gave the highest yield (60%). Encouraged by these results, we further investigated the solvent effect for this reaction using $SnCl_4$ as the promoter. Among the solvents screened (Table 1, entries 9 to 14), none of them gave a higher yield than that of CH_2Cl_2 (Table 1, entry 5). Moreover, elevating the reaction temperature to 0 °C also afforded **3a** in a lower yield (55%) (Table 1, entry 15). As the TMS protecting group was labile in the acidic conditions, we also tested the transformation of substrate **1a** with a methyl protecting group for the tertiary hydroxyl group. To our delight, this variation further increased the yield of **3a** to 66%. Therefore, the conditions outlined in Table 1, entry 16 were chosen as the optimized conditions for subsequent investigation.

With the optimized reaction conditions in hand (Table 1, entry 16), we then examined the scope of substrates that could be used in this tandem Nazarov cyclization/nucleophilic addition reaction (Scheme 2), and most of the substrates gave the expected products in good yields. With ethyl 2-oxoacetate as the electrophile, substrates **1b** and **1c** with electron-withdrawing substituents on the benzene moiety were

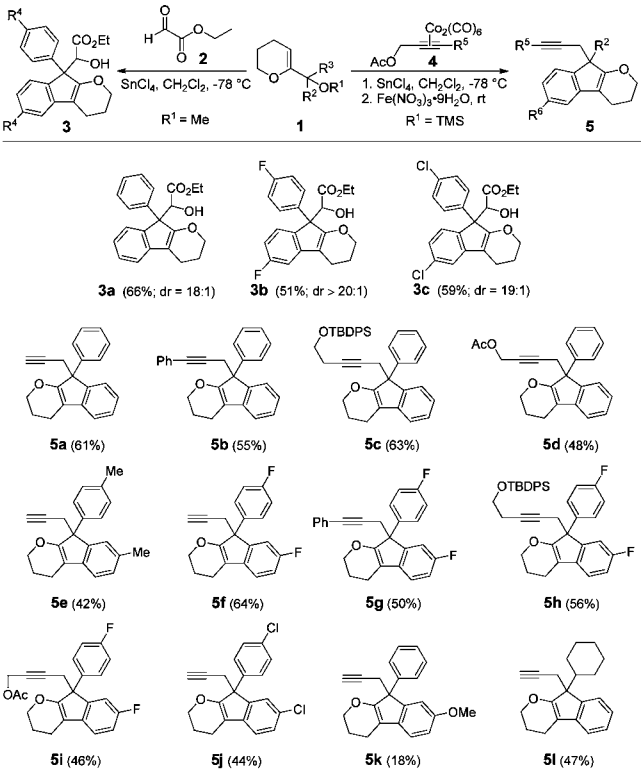
Table 1. Optimization of the Reaction Conditions for the Tandem Nazarov Cyclization/Aldol Reaction[a]



entry	promoter	solvent	temp (°C)	R	yield[b] (%)
1	$AlCl_3$	CH_2Cl_2	−78	TMS	trace
2	$BF_3{\cdot}OEt_2$	CH_2Cl_2	−78	TMS	–[c]
3	$EtAlCl_2$	CH_2Cl_2	−78	TMS	trace
4	$In(OTf)_3$	CH_2Cl_2	−78	TMS	–[d]
5	TMSOTf	CH_2Cl_2	−78	TMS	–[d]
6	$Sc(OTf)_3$	CH_2Cl_2	−78	TMS	30
7	$SnCl_4$	CH_2Cl_2	−78	TMS	60
8	$TiCl_4$	CH_2Cl_2	−78	TMS	54
9	$SnCl_4$	toluene	−78	TMS	51
10	$SnCl_4$	DCE	0	TMS	19
11	$SnCl_4$	$CHCl_3$	0	TMS	–[c]
12	$SnCl_4$	CCl_4	0	TMS	36
13	$SnCl_4$	CH_3CN	0	TMS	–[c]
14	$SnCl_4$	THF	−78	TMS	–[c]
15	$SnCl_4$	CH_2Cl_2	0	TMS	55
16	$SnCl_4$	CH_2Cl_2	−78	CH_3	66

[a]Reaction conditions: **1a** or **1a′** (0.2 mmol, 1 equiv), **2** (1.05 equiv), promoter (1.05 equiv), solvent (2.0 mL), argon atmosphere, 15 min. [b]Isolated yield. [c]No desired product. [d]Complicated mixtures.

Scheme 2. Investigation of the Scope of Substrates for the Tandem Nazarov Cyclization/Aldol Reaction[a] or Nicholas Reaction[b]



[a]Reaction conditions: **1** (0.2 mmol, 1 equiv), **2** (1.05 equiv), promoter (1.05 equiv), CH_2Cl_2 (2.0 mL), argon atmosphere, 15 min; the yields of the products are isolated yields. [b]Reaction conditions: **1′** (0.3 mmol, 1 equiv), **4** (1.1 equiv), promoter (1.05 equiv), CH_2Cl_2 (2.0 mL), argon atmosphere, 10 min; the yields of the products are isolated yields.

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transformed smoothly into the desired cyclic products **3b** and **3c**, respectively, in good yields.

In order to demonstrate the versatility of this reaction, the Nicholas reagent,[10] another typical useful electrophile, was examined under the same conditions. Different from the reaction with ethyl 2-oxoacetate, substrates protected by trimethylsilyl (TMS) provided the desired products in slightly higher yields than the methyl-protected substrate. Among the Ac-protected Co-complexed propargylic species screened, most of them could afford the expected products in good yields after the two-step reaction (iron(III) nitrate was used for the oxidative decomplexation of the alkyne from the dicobalt unit) with the TMS-protected substrates. Similar to the Nazarov cyclization/aldol reaction process, one general feature of this transformation was that substrates with electron-withdrawing groups on the phenyl ring led to the desired indene products in good yield, while the one with an electron-donating group, i.e., methyl group, afforded the expected product **5e** in a slightly lower yield. Although the O−Si bond of the TMS-protected substrate was easier to cleave, which may lead to the semipinacol rearrangement product, the overall yields were still higher than those with the substrates using methyl as the protecting group. It should be noted that, similar to the previous work of Tu,[11] preactivation of the $Co_2(CO)_6$−alkyne complex with $SnCl_4$ was crucial for the formation of the expected products. Furthermore, the structure of the product **5f** was confirmed by X-ray crystallography (Figure 2).[12] Additionally, when the substrate with a phenyl group and a cyclohexyl group attached to the tertiary hydroxyl group was applied to the reaction, it also could give the expected product **5l** in a 47% overall yield. Ethylene oxide was also tested as the electrophile to trap the reactive nucleophilic intermediate. Unfortunately, no desired product could be detected under the optimized conditions.

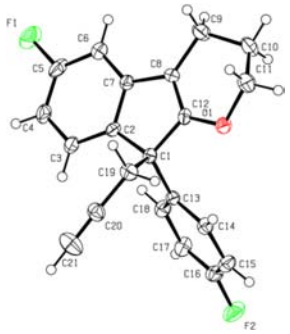
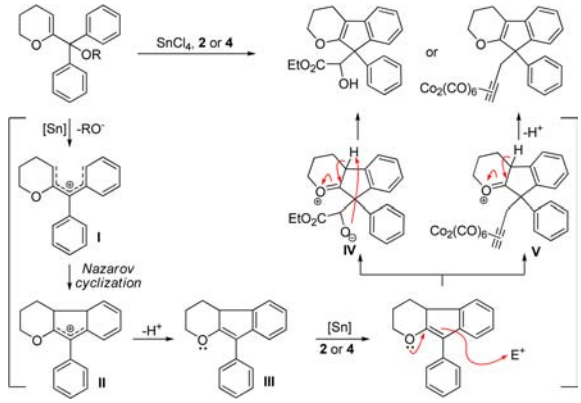


Figure 2. X-ray crystal structure of **5f** (CCDC 1422319, mp 79.6−80.3 °C, recrystallized in CH_2Cl_2).

Based on the experimental results, we proposed a plausible reaction mechanism for this tandem C−O bond cleavage/Nazarov cyclization/nucleophilic addition reaction (Scheme 3). The first step was a tin(IV)-induced C−O bond cleavage to generate pentadienyl cation intermediate **I**, which underwent a conrotatory ring closure to give a cyclic carbocation **II**.[13] After the ring closure, the carbocation intermediate **II** went through a deprotonation to reestablish the aromaticity and afford enol ether intermediate **III**. Next, under the promotion of the Lewis acid, the nucleophilic addition of intermediate **III** with ethyl 2-oxoacetate or the Nicholas reagent afforded intermediate **IV** or **V**, respectively. Finally, a proton shift of intermediate **IV** or a deprotonation of intermediate **V** gave the desired product.

Scheme 3. Proposed Mechanism for the Nazarov Cyclization/Nucleophilic Addition Reaction



In summary, we have successfully developed a novel and versatile Lewis acid induced tandem reaction toward the synthesis of multisubstituted indenes via a sequential C−O bond cleavage/Nazarov cyclization/nucleophilic addition reaction, in which a quaternary carbon center was successfully constructed. This method may provide an alternative choice for the construction of multisubstituted indenes. Further application of this method is ongoing in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02610.

Experimental procedures, characterization data and copies of the ^1H and ^{13}C NMR spectra for all new compounds (PDF)

X-ray crystal structure data for compound **5f** (CIF)

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Author Contributions

§C.Y. and Z.-L.X. contributed equally.

Notes

The authors declare no competing financial interest.

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