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3,4,5-Trimethylphenol and Lewis Acid Dual-Catalyzed Cascade Ring-Opening/Cyclization: Direct Synthesis of Naphthalenes

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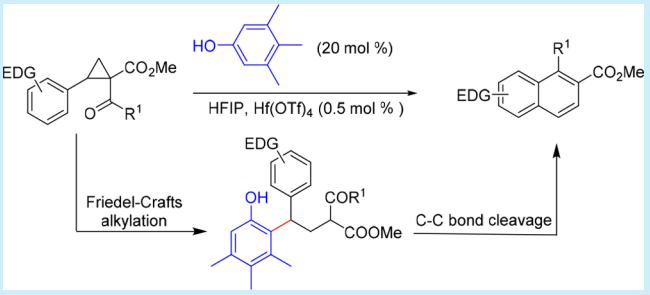
Supporting Information

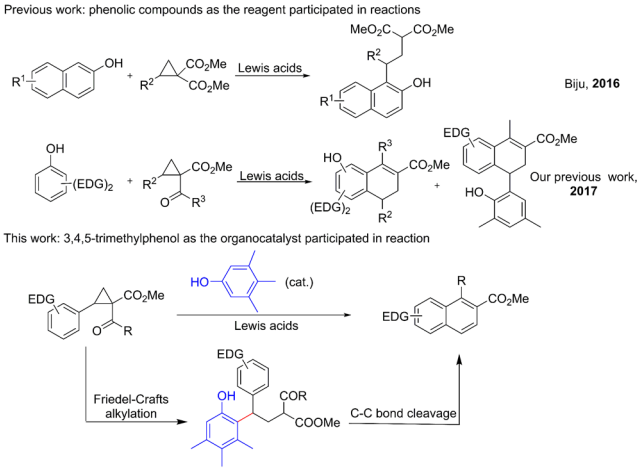
ABSTRACT: A 3,4,5-trimethylphenol and Lewis acid dual-catalyzed cascade reaction of donor−acceptor (D−A) cyclopropanes via ring-opening and cyclization is developed. In this reaction, a phenolic compound was used as a covalent catalyst for the first time. Additionally, control experiments proved that 3,4,5-trimethylphenol completed the catalytic cycle by accomplishing the C−C bond cleavage. Using this strategy, a wide variety of substituted naphthalenes has been synthesized from D−A cyclopropanes in moderate to high yields under mild conditions.



Donor−acceptor (D−A) cyclopropanes have become an important class of building blocks for the construction of cyclic and noncyclic compounds.[1] Because of their special reactivities, ring-opening,[2] cycloaddition,[3] and rearrangement reactions[4] have been developed. Among them, the ring-opening reactions of D−A cyclopropanes with carbon nucleophilies are very useful in the development of cascade reactions for the construction of carbocyclic skeletons. For example, the cascade annulation reactions of D−A cyclopropanes with indoles[5] and silyl enol ethers[6] were all developed on the basis of the corresponding ring-opening reactions. In order to develop the annulation reactions between D−A cyclopropanes and phenol derivatives, we focused our attention on the Friedel−Crafts reactions of D−A cyclopropanes with phenols first. In 2016, Biju and co-workers reported a Lewis acid-catalyzed Friedel−Crafts reaction of naphthol with D−A cycloprapanes which furnished chain products with excellent yields (Scheme 1).[7] In 2017, our group developed the [4 + 2]-annulation reaction of D−A cyclopropanes with electron-rich phenols for the preparation of dihydronaphthols and dihydronaphthalenes.[8] Recently, we also discovered that 3,4,5-trimethylphenol could act as a novel organocatalyst[9] and form a cooperative catalytic system to catalyze the rearrangement of D−A cyclopropanes.[10] This cooperative catalytic system had two attractive features: (a) 3,4,5-trimethylphenol completed the catalytic cycle by accomplishing the C−C bond cleavage, and (b) a phenolic compound was used as an organocatalyst for the first time.

The ring-opening intermediate **A** was common in most research reports (Scheme 2),[11] and the nucleophilic reagents stay on the product molecules through the whole reaction processes except the iodide. In a previous report, MgI_2 acted as a bifunctional catalyst in which the nucleophilic iodide formed a C−I bond and was then released via an S_N2 reaction (Scheme 2, i).[12] In this work, we hypothesized that, after control experiments, 3,4,5-trimethylphenol would fall off from the intermediates through an elimination reaction and at the same time accomplish the catalytic cycle (Scheme 2, ii).

Scheme 1. Reactions of D−A Cyclopropanes with Phenolic Compounds



The initial study began with the reaction of D−A cyclopropane (**1a**) catalyzed by 3,4,5-trimethylphenol (**2a**) and $Sc(OTf)_3$ (1 mol %) in 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) at 0 °C, and the expected product **3a** was successfully produced in 85% yield (Table 1, entry 1). After the substituted phenolic compounds were screened, 3,4,5-trimethylphenol **2a** was identified as the optimal organocatalyst for the reaction (Table 1, entries 1−3). In the absence of **2a**, 4,5-dihydrofuran[13] **4** isomer was produced, and no product **3a** was generated from the D−A cyclopropane (Table 1, entry 4). Several Lewis acids

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Scheme 2. Nucleophilic Reagents' Catalytic Cycle

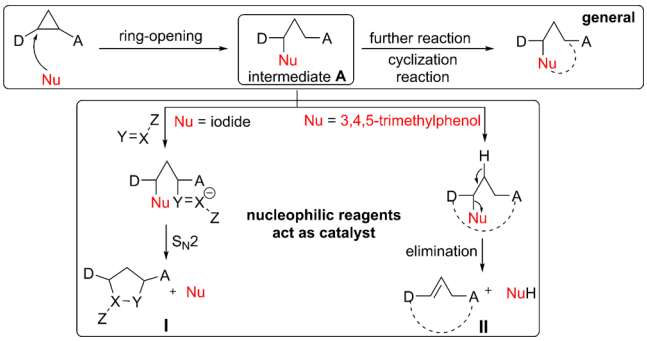
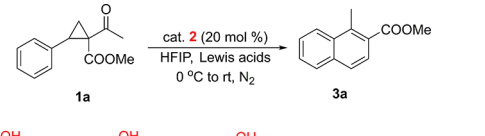
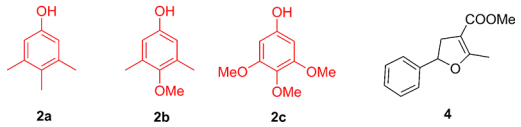


Table 1. Optimization of the Reaction Conditions[a]





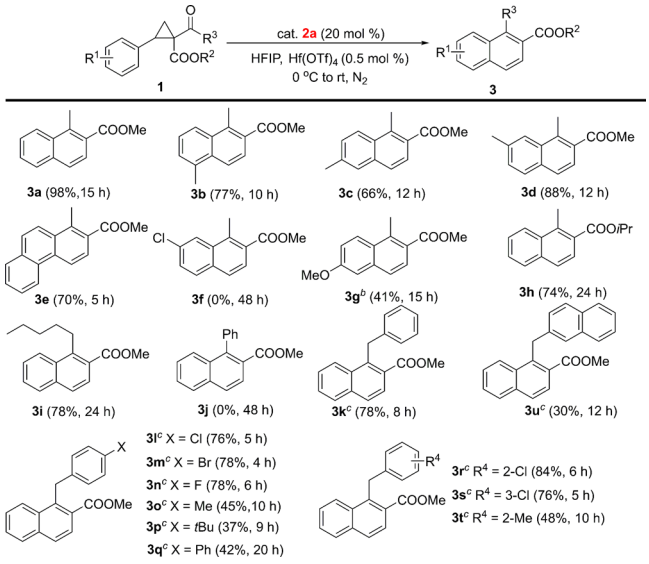
entry	Lewis acid	2	time (h)	yield (%)
1	$Sc(OTf)_3$ (1 mol %)	**2a**	8	85
2	$Sc(OTf)_3$ (1 mol %)	**2b**	48	complex
3	$Sc(OTf)_3$ (1 mol %)	**2c**	48	complex
4	$Sc(OTf)_3$ (1 mol %)		48	0
5	$Sn(OTf)_2$ (1 mol %)	**2a**	8.5	81
6	$Hf(OTf)_4$ (1 mol %)	**2a**	10	87
7	none	**2a**	48	NR
8	$Hf(OTf)_4$ (2.5 mol %)	**2a**	7	79
9	$Hf(OTf)_4$ (0.5 mol %)	**2a**	15	98
10[b]	$Hf(OTf)_4$ (0.5 mol %)	**2a**	48	41
11[c]	$Hf(OTf)_4$ (0.5 mol %)	**2a**	48	69
12[d]	$Hf(OTf)_4$ (0.5 mol %)	**2a**	26	92
13[e]	$Hf(OTf)_4$ (0.5 mol %)	**2a**	9	84

[a]Unless otherwise noted, the reaction was conducted with **1a** (0.2 mmol), **2** (0.04 mmol), and the Lewis acid in HFIP (2.0 mL) in an ice−water bath, and the temperature of the system was then allowed to rise spontaneously to room temperature. [b]10 mol % of **2a** was used. [c]1.0 mL of HFIP was used. [d]4.0 mL of HFIP was used. [e]The reaction was conducted at room temperature.

were tested in the reaction, and $Hf(OTf)_4$ was proved to be the best cocatalyst with the product yield of 87% (Table 1, entries 5−6). In particular, the presence of a Lewis acid was also proved to be necessary to activate the D−A cyclopropane (Table 1, entry 7). Subsequently, adjusting the loading of $Hf(OTf)_4$ to 0.5 mol % gave the desired product **3a** in 98% yield, and the 3,4,5-trimethylphenol **2a** could be completely recycled (Table 1, entry 9). However, reducing the dosage of 3,4,5-trimethylphenol to 10 mol % had a negative impact on the cascade reaction with a product yield of 41% (Table 1, entry 10). Further screening of the reaction temperature and the reactant concentration (Table 1, entries 11−13) demonstrated that reaction conditions of entry 9 were optimal.

With the optimized conditions established, the scope of the reaction was then investigated by using various D−A cyclopropanes **1** with different electronic and steric properties (Scheme 3). The electronic properties of substrates had

Scheme 3. Scope of the Synthesis of Naphthalenes[a]



[a]Unless otherwise noted, the reaction was conducted with **1** (0.2 mmol), **2a** (0.04 mmol), and $Hf(OTf)_4$ (0.5 mol %) in HFIP (2.0 mL) in an ice−water bath, and the temperature of the system was then allowed to rise spontaneously to room temperature. [b]60 mol % of **2a** was used. [c]The reaction was conducted at 40 °C.

noticeable influences on the reaction. In general, substrates with an electron-donating methyl group gave good yields (**3b−d**) (Scheme 3), and the substrate with a fused ring also worked well (**3e**) (Scheme 3). However, the reaction did not occur for the D−A cyclopropane with an electron-withdrawing substituent probably due to the lower nucleophilicity of the substrate (**3f**) (Scheme 3). Methoxy group substituted D−A cyclopropane gave the product only in 41% yield even when 60 mol % of the 3,4,5-trimethylphenol **2a** was used, probably because the high nucleophilicity of the substrate led to a more complex reaction (**3g**) (Scheme 3). The reaction proceeded smoothly to afford the product when substituent R^2 was the other alkyl group (**3h**) (Scheme 3). In addition, when R^3 was changed to long aliphatic chain, the desired product was also obtained with satisfying yield (**3i**) (Scheme 3). Furthermore, because of the steric and electronic influence of the phenyl group, 1-phenylnaphthalene was not obtained (**3j**) (Scheme 3). To gain more insight into the applicability of this reaction, further investigation on the scope of benzylic substrates was conducted, and it was found that the reaction temperature needed to be raised to 40 °C to obtain good results. As depicted in Scheme 3, reactions of different D−A cyclopropanes with the electron-withdrawing groups on the benzyl group proceeded smoothly to furnish the products in 76−84% yields (**3l−n** and **3r,s**) (Scheme 3). However, lower yields were obtained when the substituents of the benzyl group were replaced with the electron-donating groups (**3o−q,t,u**) (Scheme 3). In view of above situations from **3k** to **3u**, the electron-rich effect of substituents on the benzyl group might weaken the activity of the carbonyl and then reduce the rate of the nucleophilic attack.

In order to understand the reaction mechanism, we carried out several control experiments with the intermediates separated. Intermediate 4,5-dihydrofuran **4** and ring-opening product **5** were separated from the mixture of the control reaction after 2.5 h (Scheme 4, a). Then the reaction of the 4,5-

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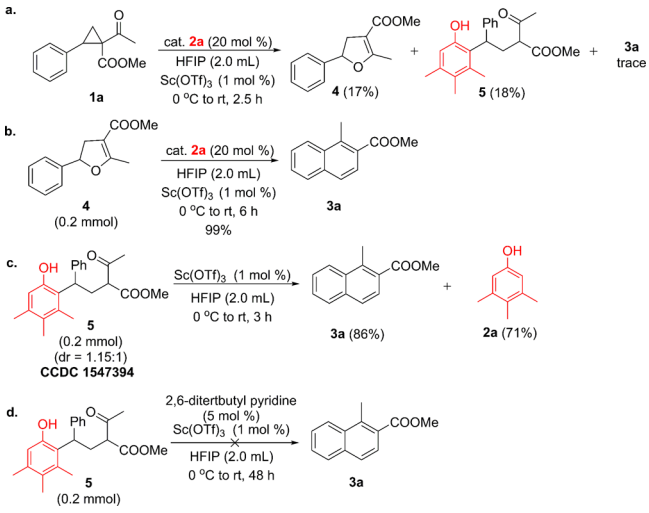
Scheme 4. Understanding the Reaction Course



dihydrofuran **4** catalyzed by 20 mol % of catalyst **2a** and 1 mol % of $Sc(OTf)_3$ gave the final product **3a** in an excellent yield of 99% after 6 h (Scheme 4, b). It was demonstrated that 4,5-dihydrofuran **4** was not only the product of the intramolecular cyclization of D−A cyclopropane **1a**, but also a significant intermediate to participate in the Friedel−Crafts alkylation. Additionally, another ring-opening intermediate product **5** also was obtained, and its structure was confirmed by X-ray crystallography (Supporting Information). With $Sc(OTf)_3$ as the catalyst in HFIP, the intermediate **5** could provide naphthalene **3a** in a yield of 86% with recovery of **2a** in a yield of 71% after 3 h (Scheme 4, c). The experimental results confirmed that 3,4,5-trimethylphenol **2a** served as an organocatalyst in this cascade reaction. The final elimination step involving C−C bond cleavage required the protonation of the phenol ring to make it a leaving group by the strong acid HOTf generated in situ. The control experiment in the presence of 2,6-ditertbutylpyridine could not produce any corresponding cyclization product (Scheme 4, d), which proved the existence and importance of HOTf. No reaction occurred even after 48 h when dihydronaphthalene obtained in our previous work[8] was tested under the new conditions, which illustrated that 3,4,5-trimethylphenol must act as the carbon nucleophile in the beginning of the ring-opening reactions to realize catalysis.

On the basis of the experimental results, a plausible mechanism for the dual-catalyzed reaction was proposed (Figure 1). There are two pathways from D−A cyclopropane **1a** to intermediate **5**. In the first pathway, the Lewis acid in HFIP induces the ring-opening reaction the cyclopropane, affording zwitterionic intermediate **I**. The intramolecular cyclization of intermediate **I** occurs to form 4,5-dihydrofuran **4**. The intermolecular Friedel−Crafts reaction between 4,5-dihydrofuran **4** or intermediate **II** with catalyst **2a** yields the ring-opening product **5**. In the second pathway, the ring-opening product **5** is directly synthesized by the Friedel−Crafts reaction between D−A cyclopropanes **1a** and catalyst **2a**. Next, elimination of one molecule of water after intramolecular Friedel−Crafts reaction of the ring-opening product **5** gives the dihydrogen naphthol **8**.[8] It should be noted that the high acidity/strong hydrogen-bond-donating property of HFIP also plays an important role in this process.[14,15] Finally, the elimination process with aromatization as one of the main

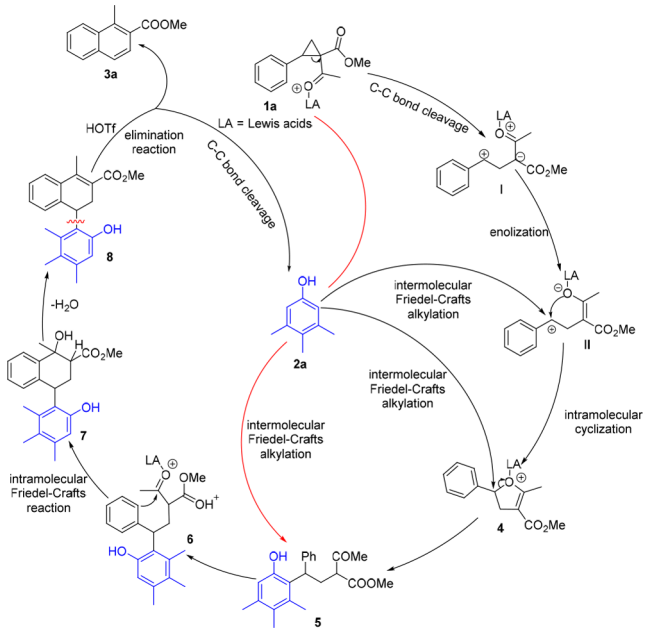


Figure 1. Plausible mechanism for the dual-catalyzed reaction.

driving forces affords the substituted naphthalene and 3,4,5-trimethylphenol **2a** to accomplish the catalytic cycle.

In conclusion, we developed a mild and efficient method to synthesize substituted naphthalenes from D−A cyclopropanes in moderate to high yields by using commercially available 3,4,5-trimethylphenol and a Lewis acid as dual cocatalysts. Additionally, we proved that 3,4,5-trimethylphenol acted as a covalent catalyst and could be recycled in the cascade reaction through control experiments. Currently, applying the phenolic organocatalyst to other synthetic systems is underway 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.7b03392.

Detailed experimental procedures, full spectroscopic data for all new compounds, and X-ray data for **5** (PDF)

Accession Codes

CCDC 1547394 contains 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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Notes

The authors declare no competing financial interest.

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