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Lewis Acid Catalyzed Intramolecular [3 + 2] Cross Cycloadditions of Cobalt-Alkynylcyclopropane 1,1-Diesters with Carbonyls for Construction of Medium-Sized and Polycyclic Skeletons

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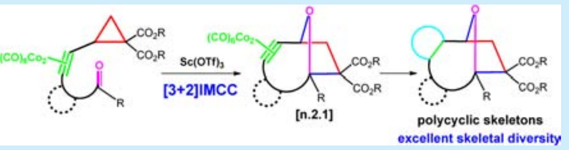
Supporting Information

ABSTRACT: A Lewis acid catalyzed intramolecular [3 + 2] cross cycloaddition of cobalt-alkynylcyclopropane 1,1-diesters with carbonyls has been successfully developed. Together with simple and efficient postcycloadditions of the cobalt-alkyne moiety, a general and efficient strategy for construction of structurally complex and diverse medium-sized skeletons and related polycycles was supplied successfully.



Developing general and efficient methods for the construction of structurally diverse and complex polycyclic skeletons is important for natural products synthesis and lead discovery. Medium-sized skeletons constitute the basic skeletons of many important biologically active natural products, often embedded within polycycles (Figure 1).[1] These medium-sized skeletons are also useful building blocks in organic synthesis. However, because of entropic and enthalpic factors, the medium-sized skeletons, and in particular the carbocyclic ones, are much more difficult to be assembled in high efficiency with conventional methods.[2] Developing efficient and general strategies for the construction of medium-sized and related polycyclic skeletons, both in terms of the efficiency for complexity and the generality for diversity, remains a challenging and attractive theme in organic synthesis.[3−6]

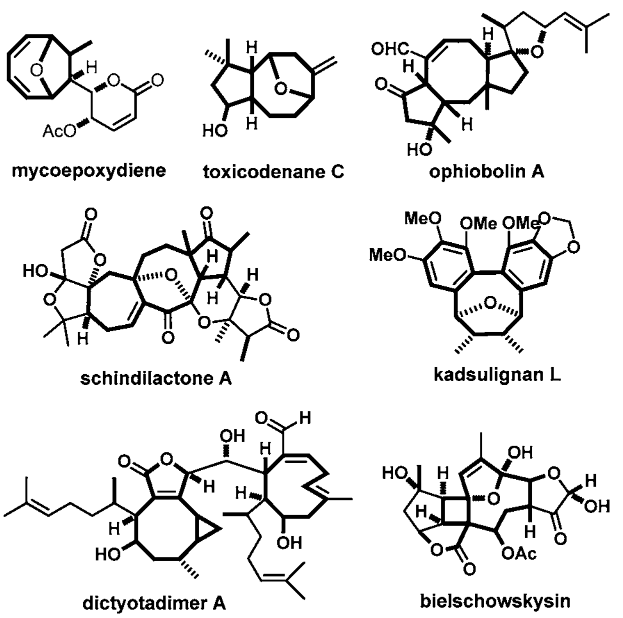


Figure 1. Representative natural products with medium-sized ring-derived polycyclic carbocycles.

We have recently developed an intramolecular cross-cycloaddition (IMCC)[7d,8] of donor−acceptor cyclopropanes,[7] which provided a general and efficient strategy for the construction of medium-sized skeletons. To develop a general and efficient strategy for construction of polycycles deriving from the medium-sized skeletons, one feasible method is to introduce a suitable functional group into the linker of the IMCC. Such a functional group should meet the following criteria: it should configurationally and conformationally favor the cycloaddition, it should activate the cyclopropane as a donor, and it should be applied for further elaboration into structurally diverse skeletons, especially cyclic ones via easy postmodifications.

Alkynyl groups have rich chemistries and can be used as donors (as a propargyl cation). Furthermore, intermolecular [3 + 2] cycloadditions of alkynylcyclopropane 1,1-diester were reported recently by Niggemann, Sakata, and Nishibayashi.[9d,e] However, this cannot be directly applied in an intramolecular version in which the two reactive sites are difficult to connect. By creatively invoking the Nicholas carbocation intermediate,[10] Christie and Jones et al. described an intermolecular [3 + 2] cycloaddition of cobalt-alkynylcyclopropane (CoACP) 1,1-diester with aldehydes or imines under promotion of $BF_3 \cdot OEt_2$.[9a,b] Kerr et al. reported a $Sc(OTf)_3$-catalyzed intermolecular [3 + 3] cycloaddition of CoACP 1,1-diester with nitrones.[9c] We reason that the cobalt-alkyne moiety can be thought as a "bent alkyne" to provide an opportunity for the two reactive sites to meet each other in the intramolecular cycloadditions, which can lead to postfunctionalizations (Scheme 1).

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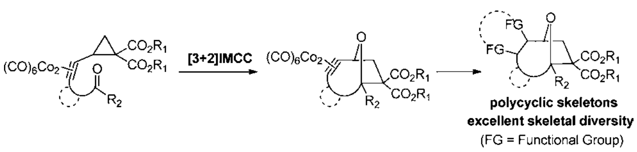
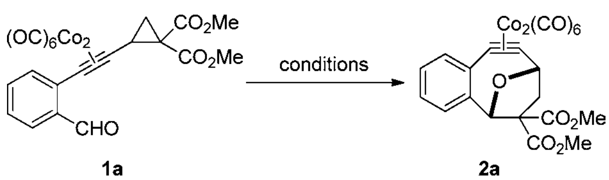
Scheme 1. [3 + 2]IMCC of CoACP 1,1-Diester for Construction of Structurally Diverse Polycyclic Skeletons



Our initial investigation started from the reaction of substrate **1a** for the concept validation. Compound **1a** was easily obtained by the methods reported in the literature.[9a−c] We were delighted to find that most of the Lewis acids could successfully promote the reaction (Table 1) to give the desired cycloadduct **2a**. The [3 + 2] IMCC proceeded smoothly under catalysis of $Sc(OTf)_3$ in CH_2ClCH_2Cl (DCE) at room temperature with an excellent yield (93%), which was used as the optimal condition (Table 1, entry 4) for most of the other examples. Structure of **2a** was determined by NMR and X-ray crystal analysis.[11,12]

Table 1. Optimization of the [3 + 2]IMCC Condition[a]



entry	catalyst	solvent	time (h)	yield (%)[b]
1	$SnCl_4$	DCE	2	86
2	$BF_3.Et_2O$	DCE	8	27
3	$Sn(OTf)_2$	DCE	17	59
4	$Sc(OTf)_3$	DCE	2	93
5	$Yb(OTf)_3$	DCE	17	40
6	$Zn(OTf)_2$	DCE	17	12
7	$Sc(OTf)_3$	CH_2Cl_2	17	76
8	$Sc(OTf)_3$	toluene	17	17
9	$Sc(OTf)_3$[c]	DCE	17	53

[a]Reaction conditions: **1a** (0.1 mmol), catalyst (0.2 equiv), solvent (2.5 mL), rt. [b]Isolated yields. [c]Used 0.1 equiv of $Sc(OTf)_3$. DCE = 1,2-dichloroethane.

Under the optimal condition, the scope of the substrates was then examined (Figure 2). [3 + 2] IMCC of the tetra-substituted cyclopropane **1b** also gave the desired cycloadduct **2b** successfully. Substrates with either electron-donating (**1d** and **1g**) or electron-withdrawing (**1e**) substituent on the benzene ring also gave the [3 + 2] IMCC cycloadducts **2d**, **2e**, and **2g**. The [3 + 2] IMCC of ketone **1f** was also successful. When the benzene rings were replaced by cycloalkenes, the [3 + 2] IMCC were successful and gave the corresponding cycloadducts in moderate to good yields (**2h**−**k**). These examples are very worthy of attention because the easy introduction[13] of various cyclic rings, and the potential postmodifications of the C═C bridges may provide an efficient and general strategy for construction of structurally diverse polycyclic skeletons in natural products (Figure 1). The successful [3 + 2] IMCC of **1l** promises to be a potential method for synthesis of the cyclooctane-fused indole alkaloid family. For the [3 + 2] IMCC of chain substrate **1m**, $Sc(OTf)_3$ failed, while $BF_3·Et_2O$ could realize the goal well. The scope of the carbocyclic ring size could be expanded to cyclononane. [3 + 2] IMCC of substrates **1n** and **1o** gave cycloadducts **2n** and **2o**, respectively, in moderate yields. However, construction of a cyclodecane substructure had failed.

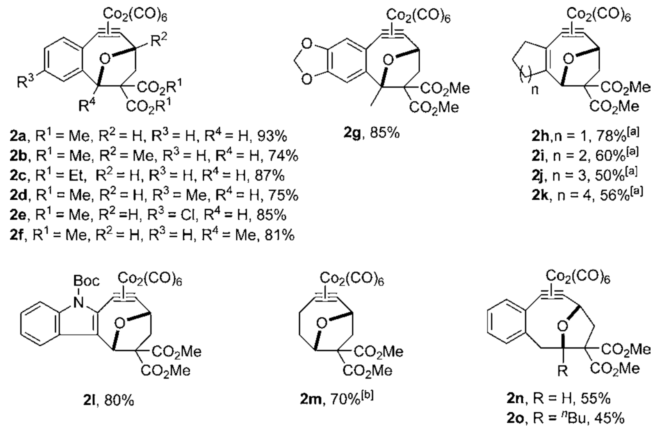
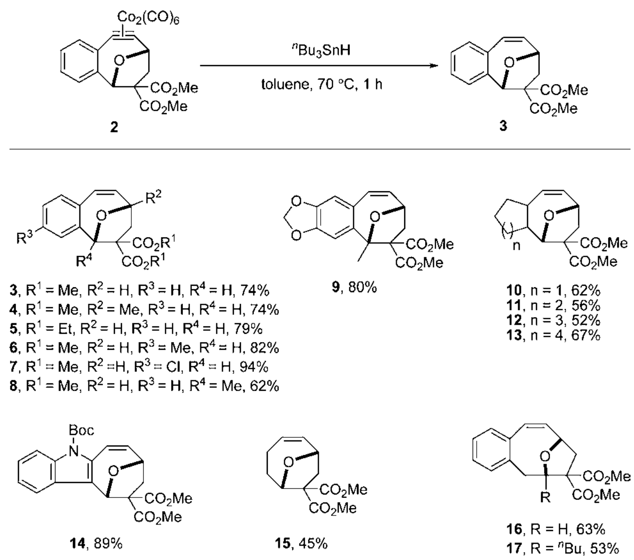


Figure 2. Lewis acid catalyzed [3 + 2] IMCC of CoACP 1,1-diesters. Key: (a) $Sc(OTf)_3$ (0.4 equiv); (b) $BF_3·Et_2O$ (3.0 equiv) was used instead of $Sc(OTf)_3$, in DCM (2.5 mL), reflux. DCM = dichloromethane.

Removal of cobalt with $^{n}Bu_3SnH$ afforded the corresponding alkene products (**3**−**17**) (Scheme 2).[12]

Scheme 2. Reductive Removal of Cobalt



The successful construction of the medium-sized carbocyclic skeletons, as well as the bridged oxa-[n.2.1] skeletons, promises to provide an efficient and general strategy in application to synthesis of a larger family of biologically important natural products (Figure 1).

To explore the potential of this strategy further, several simple and target-oriented postfunctionalizations of **2a** were carried out (Scheme 3). The cobalt-alkyne complex has rich chemistries,[10] including various cyclizations[14] and cycloadditions[15] for construction of additional rings. Pauson−Khand [2 + 2 + 1] cycloaddition[16] of **2a** with norbornene gave cycloadduct **18** (**18a**/**18b** = 3:1). The introduction of a cyclopentane ring might find its application in construction of the 5−8 bicyclic core skeleton in natural products (Figure 1). A Diels−Alder [4 + 2] cycloaddition with 2,3-dimethyl-1,4-diene introduced another benzene ring and gave 6−8−6 tricyclic product **19**, which can be applied as a potential method for synthesis of kadsulignan family (Figure 1).[5e] A furandione ring was also successfully introduced

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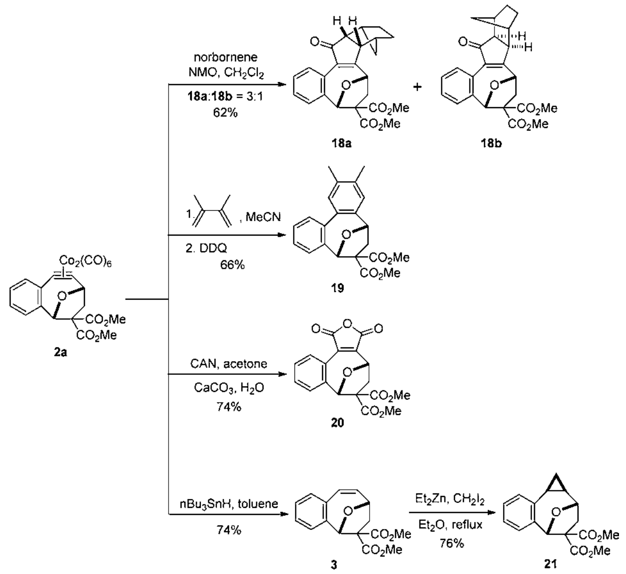
Scheme 3. Several Convenient and Target-Oriented Postfunctionalizations of 2a



to afford **20**, which supplied a potential application in construction of the bicyclic cyclooctane-furanone skeleton in natural products (Figure 1). Simmons−Smith cyclopropanation of **3** gave tricyclic product **21** with excellent stereoselectivity. The 3−8 bicyclic skeleton also exists in natural products (Figure 1).

In conclusion, we have reported the first intramolecular cycloaddition of CoACP. This constituted a general and efficient strategy for construction of medium-sized (cyclooctanoids and cyclononanoids) and related polycyclic skeletons. Features of this strategy include easy preparation of structurally diverse substrates, mild conditions, rich skeletal and substituent diversities, the inherent excellent stereochemistry of the bridged rings, and the rich and efficient postfunctionalizations (especially the introduction of additional rings). These are quite important in natural products synthesis and construction of structurally diverse libraries for lead discovery and chemical biology studies. Future investigations include the application of the strategy to total synthesis of natural products containing polycyclic skeletons and biological assay of the built compound library for lead discovery.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures and characterization data, including X-ray crystal structures of products **2a**, **3**, and **18a** (CIF). This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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(11) CCDC 1008071 (**2a**), CCDC 1008070 (**3**), and CCDC 1008072 (**18a**) contain the supplementary crystallographic data for this letter. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(12) Because the cobalt−alkyne complex **2** could not give better NMR spectra, the cobalt moieties were removed to give the corresponding alkenes **3**−**17** and spectra of which were obtained (see Supporting Information).

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