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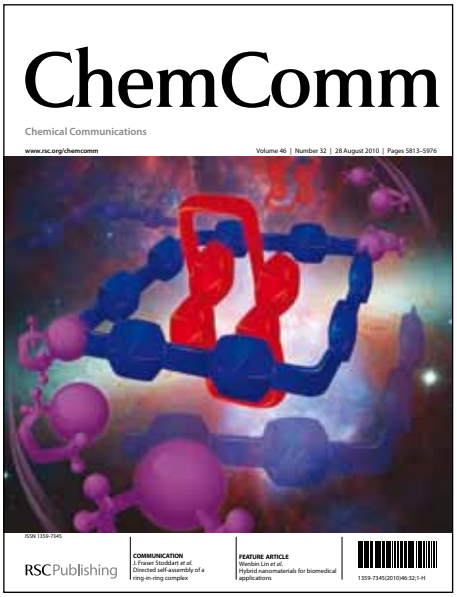

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ARTICLE TYPE

Rhodium-catalyzed Tandem Pauson-Khand Type Reactions of 1,4 Enynes Tethered by a Cyclopropyl Group

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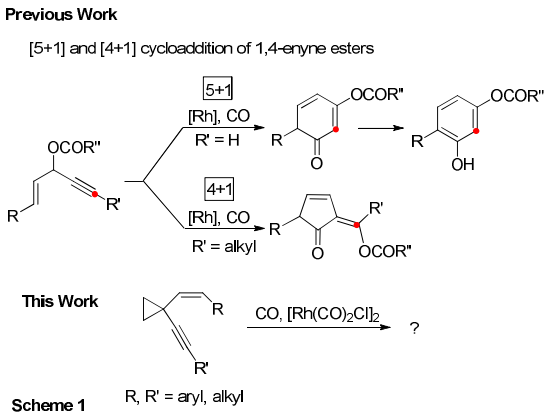
The tandem Pauson-Khand type reactions of 1,4 enynes tethered by a cyclopropyl group with two molecules of CO proceed smoothly in the presence of $[Rh(CO)_2Cl]_2$ under CO atmosphere to give the corresponding 6-hydroxy-2,3-dihydro-1*H*-inden-1-one derivatives in moderate yields under mild conditions.

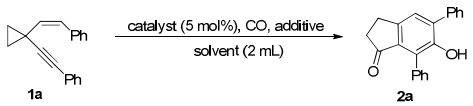
Pauson-Khand reaction (PKR) has attracted considerable attention since its discovery by Pauson and Khand.[1] This reaction has been recognized as a new route to synthetically useful cyclopentanone units. In the last few decades, significant advances have been made in this area.[2] Besides cobalt, other transition metals such as zirconium, titanium, ruthenium, palladium and rhodium can also be used in Pauson-Khand reaction and variants of Pauson-Khand type reactions are prevalent in recent literature.[3] On the other hand, transition metal catalyzed cyclizations of enynes have been studied extensively in the last few decades with only few examples for 1,4 enynes.[4,5] To the best of our knowledge, Pauson-Khand type reactions of 1,4 enynes have not been reported thus far.

Very recently, Fukuyama reported a novel Rh-catalyzed cycloaddition of 1,4 enyne esters,[6] which involves an acyloxy migration as a key step (Scheme 1). Cyclopropane derivatives are versatile building blocks in organic chemistry.[7] Because of their unique structures and relatively high reactivities, they have been widely utilized in organic synthesis. Considering the tendency of strain releasing of cyclopropyl group and on the basis of previous study of rhodium catalyzed reactions of cyclopropane derivatives of our group,[8] Wender's and Yu's group,[9] we envisaged that introducing a cyclopropyl group at the 1,4 enyne functionality will induce the ring opening of the cyclopropyl group in a Rh(I)-catalyzed Pauson-Khand type reaction (Scheme 1). Herein, we wish to report a novel tandem Pauson-Khand type reaction of 1,4 enynes tethered with a cyclopropyl group.



Scheme 1

Table 1. Selected Examples of Reaction Conditions Optimization.



1a 2a

entry[a]	catalyst	additive	time (h)	solvent	P (atm)	T (°C)	yield (%)[b]
1	$[Rh(CO)_2Cl]_2$	-	8	*p*-xylene	1	100	35
2[c]	$[Rh(CO)_2Cl]_2$	-	8	*p*-xylene	1	120	55
3	$[Rh(CO)_2Cl]_2$	-	18	*p*-xylene	1	140	60
4	$[Rh(CO)_2Cl]_2$	-	18	*p*-xylene (4 mL)	1	140	53
5	$[Rh(CO)_2Cl]_2$	-	18	TCE	1	120	25
6	$[Rh(CO)_2Cl]_2$	-	24	Nonane	1	140	33
7	$[Rh(CO)_2Cl]_2$	-	18	decane/*p*-xylene (3/1)	1	140	55
8	$[Rh(CO)_2Cl]_2$	-	12	toluene	1	100	52
9	$[Rh(CO)_2Cl]_2$	NMO	12	*p*-xylene	1	140	no reaction
10	$[Rh(CO)_2Cl]_2$	-	18	*p*-xylene	30	140	complex
11	$[Rh(CO)_2Cl]_2$	-	18	*p*-xylene	< 1[d]	140	50
12	$[Rh(CO)_2Cl]_2$ (10 mol%)	-	18	*p*-xylene	1	140	54

[a] The reaction was performed in a 25 mL flame and vacuum dried Schlenk tube. **1** (0.2 mmol) and the catalyst (4 mg, 5 mol%) was added and the tube was evacuated and filled with CO for 5 times, then the solvent was added and the tube was allowed to stir in an oil bath. [b] Isolated yields. [c] The unreacted starting material was recovered in 23% yield as E/Z isomeric mixtures (E/Z = 1/4). [d] CO and Ar mixed atmosphere.

We initiated our investigations by seeking the optimal conditions for the tandem Pauson-Khand type reaction of 1,4 enyne **1a** in the presence of rhodium catalyst and the selected results are indicated in Table 1. With $[Rh(CO)_2Cl]_2$ (5 mol %) as the catalyst, the product **2a** was afforded in 35% yield (100 °C, 8 h). The yield of **2a** could be improved to 55% or 60% when the reaction was carried out at 120 °C or when the reaction time was extended to 18 h at 140 °C (Table 1, entries 1-3). Upon further screening of various solvents such as TCE (1,1,2,2-tetrachloroethane), nonane, toluene, and mixed solvent (n-decane/p-xylene = 3/1), no improvement in yield was observed (Table 1, entries 5-8). The additive effect of NMO has been also examined.[10] However, this additive did not improve the outcome of our reaction (Table 1, entry 9). Increasing or reducing the

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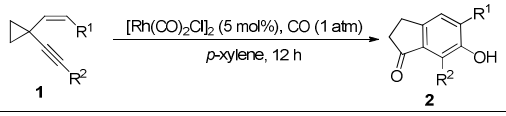
† Electronic Supplementary Information (ESI) available: Experimental procedures, characterization data of new compounds, and CCDC 889520, See DOI: 10.1039/b000000x/.

pressure of CO and increasing the employed amount of Rh(I) catalyst did not give better results either (Table 1, entries 10, 11 and 12, for more detailed information, see Table SI-1 in the Supporting Information).

To evaluate the generality of the reaction, different substrates with varying substituents have been synthesized and investigated under the standard reaction conditions. As can be seen from Table 2, as for a variety of 1,4 enynes with different aromatic substituents at the alkenyl terminal, the reaction proceeded smoothly to give the desired products in moderate yields (Table 2, entries 1-8). When R^1 is an alkyl group (Et or *n*-Pr), similar results were obtained (Table 2, entries 9-10). The reaction was also successful when the alkynyl terminal has different aromatic groups (Table 2, entries 11-12) or alkyl group (Table 2, entry 13). Only 21% yield of **2a** was obtained with the *trans* substrate **1a'**, indicating that the *trans* substrate is less reactive than that of the *cis* one. The structure of the product was further confirmed by the X-ray diffraction of product **2c**. The ORTEP drawing and the CIF data are presented in the Supporting Information.[11]

Table 2. Substrate Scope of the Pauson-Khand Type Reaction

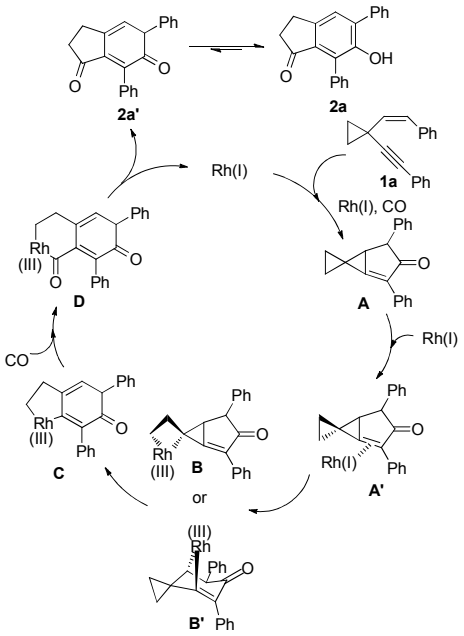


entry[a]	**1**, R^1	R^2	T (°C)	yield (%)[b]
1	**1b**, *p*-Me-C_6H_4	Ph	130	**2b**, 51
2	**1c**, *p*-O_2N-C_6H_4	Ph	140	**2c**, 45
3[c]	**1d**, *p*-F-C_6H_4	Ph	100	**2d**, 47
4[c]	**1e**, *p*-Cl-C_6H_4	Ph	100	**2e**, 42
5[c]	**1f**, *p*-MeO-C_6H_4	Ph	100	**2f**, 44
6	**1g**, *o*-Me-C_6H_4	Ph	130	**2g**, 46
7	**1h**, *m*-Me-C_6H_4	Ph	130	**2h**, 52
8	**1i**, 1-naphthyl	Ph	100	**2i**, 43
9	**1j**, Et	Ph	140	**2j**, 48
10	**1k**, *n*-Pr	Ph	130	**2k**, 41
11	**1l**, Ph	*p*-Me-C_6H_4	130	**2l**, 55
12	**1m**, Ph	*p*-O_2N-C_6H_4	100	**2m**, 40
13	**1n**, Ph	*n*-butyl	100	**2n**, 49
14	**1a'**, *trans*-Ph	Ph	130	**2a**, 21

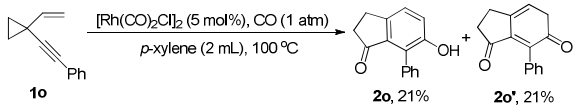
[a] The reaction was performed in a 25 mL flame and vacuum dried Schlenk tube. **1** (0.2 mmol) and the catalyst (4 mg, 5 mol%) was added and the tube was evacuated and filled with CO for 5 times, then the solvent was added, and the tube was allowed to stir in an oil bath. [b] Isolated yields. [c] When the reaction was performed at 140 °C, the reaction gave complex product mixtures because of the unknown side reactions. [d] 73% starting materials can be recovered as E/Z isomeric mixtures (E/Z = 7.6/1).

A plausible reaction mechanism is shown in Scheme 2 using **1a** as a model substrate. **1a** first undergoes a Pauson-Khand reaction in the presence of Rh(I) catalyst to give an spiropentane intermediate **A** and regenerate the Rh(I) species. Coordination of the double bond with the Rh(I) species forms intermediate **A'**. Oxidative addition of intermediate **A** by Rh(I) produces intermediate **B** (or intermediate **B'** according to one referee's suggestion), which undergoes a subsequent *β*-carbon elimination to afford intermediate **C**.[12] Migratory insertion of another CO molecule generates intermediate **D**, which produces compound **2a'** via reductive elimination. Compound **2a'** can tautomerize to product **2a**.

Scheme 2. A Plausible Reaction Mechanism

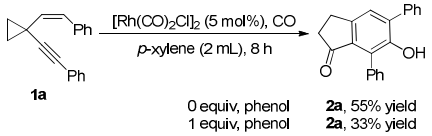


Schene 3. Pauson-Khand Type Reaction of **1o**



To verify the reaction pathway, we attempted to determine and isolate the possible reaction intermediates. To our delight, when the Pauson-Khand type reaction of **1o** was conducted under the standard conditions (Scheme 3). Products **2o** and **2o'** were isolated respectively, suggesting the formation of product **2a'** in Scheme 2.

Scheme 4. Control Experiment in the Tandem Pauson-Khand Reaction



Moreover, the control experiment shown in Scheme 4 revealed that adding 1.0 equiv of phenol can significantly impair the reaction outcome, giving **2a** in 33% yield. This result can explain the moderate yield of this reaction because the generated phenolic product can inhibit the reaction proceeding.

In summary, we have developed a novel tandem Pauson-Khand type reaction of 1,4 enynes tethered by a cyclopropyl group catalyzed by $[Rh(CO)_2Cl]_2$, affording the corresponding 6-hydroxy-2,3-dihydro-1*H*-inden-1-one derivatives in moderate yields under mild conditions. A plausible reaction mechanism has been also proposed. Current efforts are in progress to use this new methodology to synthesize biologically active products.

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support (21072206, 20472096, 20872162, 20672127, 21121062 and 20732008).

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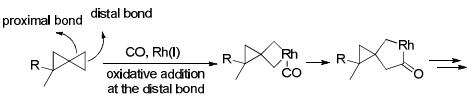
11. The crystal data of **2c** have been deposited in CCDC with number 889520.
12. Murakami reported similar oxidative addition and *β*-carbon elimination cascade of spiropentane units. The difference is the site selectivity in the oxidative addition step. In our mechanism, the oxidative addition takes place at the proximal bond, whereas in Murakami's reaction, the distal bond was cleaved at first. T. Matsuda, T. Tsuboi, M. Murakami, *J. Am. Chem. Soc.*, **2007**, *129*, 12596-12597.



Oxidative addition at distal bond in Mulakami's mechanism

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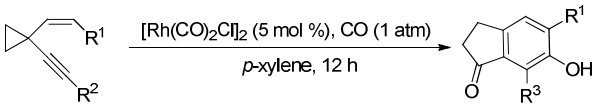
Rhodium-catalyzed tandem Pauson-Khand type reactions of 1,4 enynes tethering a cyclopropyl group



R^1 = H, alkyl, aryl; R^2 = alkyl, aryl.
yield: 40% to 60%

Novel 6-hydroxy-2,3-dihydro-1*H*-inden-1-one derivatives can be synthesized by the tandem Pauson-Khand type reactions of 1,4 enynes tethering a cyclopropyl group in the presence of $[Rh(CO)_2Cl]_2$ under CO atmosphere.

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