ChemComm

COMMUNICATION

OF CHEMISTR

ROYAL SOCIETY

View Article Online

Check for updates

Cite this: DOI: 10.1039/c9cc08559a

Received 2nd November 2019, Accepted 6th December 2019

DOI: 10.1039/c9cc08559a

rsc.li/chemcomm

Relay Rh(μ)/Pd(0) dual catalysis: synthesis of α -quaternary β -keto-esters *via* a [1,2]-sigmatropic rearrangement/allylic alkylation cascade of α -diazo tertiary alcohols⁺

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A relay Rh(μ)/Pd(0) dual catalysis that enables domino [1,2]sigmatropic rearrangement/allylic alkylation of α -diazo tertiary alcohols is described. This transformation represents a highly efficient method for the one-pot synthesis of α -quaternary β -ketoesters under mild conditions, in which two separate C-C σ -bonds at the carbenic center were formed in a straightforward manner.

The efficient construction of quaternary carbon centers remains a critical challenge in organic synthesis.¹ Among various methodologies, the gem-difunctionalization of diazo compounds^{2,3,9} via metal-carbenoids is a well-documented process for accessing quaternary carbon centers. This directly introduces two different functional groups at a carbenic center (Scheme 1). Recent milestones in transition metal catalyzed cross-couplings involving metalcarbenoid migratory insertions have mostly been successful3-8 (Scheme 1, strategy A). Pioneered by Van Vranken^{4a} and Barluenga, 3a,4b,c the research groups of Wang, 3c,d,5 Liang, 6a,b Yu,⁷ and Rovis^{8a} have further developed the migratory insertion and coupling cascade reaction of *in situ* generated palladium, rhodium, or copper carbenoids, which realizes the efficient construction of quaternary carbon centers. Remarkably, Hu^{9,10} and others¹¹ also established the method of electrophilic trapping of metal-carbenoid induced ylides or zwitterionic intermediates (Scheme 1, strategy B), which provides an alternative route to quaternary carbon centers. Despite the apparent advances, these two strategies are mostly limited to donor/acceptor (D/A) diazo compounds. Therefore, developing new and efficient catalytic systems to address other types of diazo compounds under mild conditions is highly desired.

Previously, we demonstrated for the first time the crosscompatible Rh(u)/Pd(0) dual-catalysis system.¹² This promotes the divergent reaction of β -keto- α -diazo-carbonyl compounds (acceptor/acceptor diazo compounds) with allylic carboxylates

Strategy A R-[M] R=[M] R=ary, vinyl, allyl, alkynyl, acyl, benzyl, allenyl R = ary, vinyl, allyl, alkynyl, acyl, benzyl, allenyl R = ary, vinyl, allyl, alkynyl, acyl, benzyl, allenyl Quaternary carbon Quaternary carbon M = Pd, Rh, Au Nu = O, N, S, ArH

Scheme 1 Metal-carbenoids for the construction of quaternary carbon centers.

for the selective construction of cyclic quaternary carbon centers (Scheme 2a).^{12a,c,d} In this cooperative dual catalysis process, the catalytically generated Rh(II)-carbenoids (A) and π -allyl Pd(II) intermediates (B) are involved. The sigmatropic rearrangement is recognized as one of the most attractive fashions for the formation of quaternary carbon centers.¹³⁻¹⁶ In particular, a series of practical tandem semipinacol rearrangements developed by Tu and co-workers14,15 have been successfully applied in the total synthesis of natural products. Maruoka^{16a,b} and Feng^{16c} have also explored Lewis acid catalyzed homologation of aldehydes or ketones with diazo compounds. This results in a carbon-chain extension or ring expansion at a carbonyl group, involving a single catalyst. However, some chemical transformations would be inefficient or unattainable with mono-catalysis. Recently, Gong and co-workers established a highly compatible rhodium/bifunctional urea dual-catalysis system¹⁷ that rendered a [1,2]-sigmatropic rearrangement/Michael addition cascade of α-diazo alcohols with nitroalkenes.

Inspired by the above studies, we envisioned that the Rh(π)-catalyst induced [1,2]-sigmatropic rearrangement¹⁸ of α -diazo tertiary alcohols (1) would efficiently generate the enol ester intermediates (E). Subsequently, Pd(0)-catalyzed allylic alkylation with allyl *tert*-butyl carbonate (2) would access acyclic

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 $[\]dagger$ Electronic supplementary information (ESI) available. See DOI: 10.1039/ c9cc08559a

(a) Previous work: Cyclic quaternary centers construction via Rh(II)/Pd(0) dual catalysis



(b) This work: Acyclic quaternary centers construction via Rh(II)/Pd(0) dual catalysis



 α -quaternary allylated β -keto-esters (3), in which two different C–C bond formations on the carbenic center were formed in a candid manner (Scheme 2b).

At the outset, the redox-compatible $Rh(\pi)/Pd(0)$ dual catalytic system¹² established by our group was selected to investigate the reaction of an α -diazo tertiary alcohol (1a) with an allyl *tert*-butyl carbonate (2a) (Table 1).

As expected, when the reaction was carried out at room temperature in the presence of $Rh_2({}^tBuCO_2)_4$ (1.0 mol%), $[Pd(allyl)Cl]_2$ (2.0 mol%), and Xantphos (2.2 mol%), the desired product (**3aa**) could be isolated in 10% yield (entry 1). We were pleased to find that the addition of K_2CO_3 (2.0 equiv.)

Table 1 Optimization of the reaction conditions ^a						
	$\begin{array}{c} Ph & Ph \\ HO & \\ N_2 \end{array} + \begin{array}{c} OBoc \\ OBoc \end{array}$		[Rh] (2.0 mol%) [Pd] (4.0 mol%) Ligand (2.2 mol%) Toluene, r.t, 2h		Ph Ph Ph	
	1a	2a			3aa	
Entry	Rh_2L_4	[Pd]	Liga	and	Base	3aa ^b (%)
1	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)C	l] ₂ Xan	tphos	_	10
2	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)C	2l]₂ Xan	tphos	K_2CO_3	94
3	$Rh_2(^tBuCO_2)_4$	$Pd(PPh_3)_4$		-	K_2CO_3	0
4	$Rh_2(^tBuCO_2)_4$	PdCl ₂	Xan	tphos	K_2CO_3	<5
5	$Rh_2(^tBuCO_2)_4$	$Pd(OAc)_2$	Xan	tphos	K_2CO_3	86
6	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)C	$[2]_2 P(2-$	furyl)3	K_2CO_3	38
7	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)C	$\left\ \mathbf{J}_{2} \right\ _{2}$ Seg	phos	K_2CO_3	89
8	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)C	$\left\ \mathbf{J}_{2} \right\ _{2}$ Dpp	of	K_2CO_3	92
9	$RhCl(PPh_3)_3$	[Pd(allyl)C	$\left\ {l} \right\ _2$ Xan	tphos	K_2CO_3	71
10	$Rh_2(OAc)_4$	[Pd(allyl)C	ll]₂ Xan	tphos	K_2CO_3	76
11	$Rh_2(^tBuCO_2)_4$	[Pd(allyl)0	∑l]₂ Xan	tphos	Cs ₂ CO ₃	98

 a Reaction conditions: 1a (0.12 M, 1.2 equiv.), 2a (0.1 M, 1.0 equiv.), Rh₂L₄ (1.0 mol%), [Pd] (4.0 mol%), based on Pd, and the ligand (2.2 mol%) in toluene at room temperature. b Isolated yields.

significantly improved the yield affording 3aa in 94% yield, which may facilitate the Pd(0) catalyzed allylic alkylation (entry 2). Further optimization showed that the reaction efficiency was largely affected by changing the Pd catalysts (entries 3-5). For example, Pd(PPh₃)₄ and PdCl₂ were unable to promote the reaction, probably because of the deactivation effect within this catalytic system.¹⁹ Various phosphine ligands were also tested (entries 6–8). However, the utilization of mono-dentate phosphine $P(2-furyl)_3$ (entry 6) had a deleterious effect. The other bidentate phosphine ligands (entries 7 and 8) did not give better results than those observed with Xantphos. In addition, when other Rh catalysts such as RhCl(PPh₃)₃ and Rh₂(OAc)₄ were used, the yields were diminished too (entries 9 and 10). After considerable investigations, a protocol based on $Rh_2(^tBuCO_2)_4$, $[Pd(allyl)Cl]_2$, Xantphos, and Cs₂CO₃ in toluene at room temperature afforded 3aa in 98% yield (entry 11).

With the ideal conditions in hand, we then tested the scope of α -diazo tertiary alcohols, which could easily be prepared from the commercially available α -diazo ethyl acetate and ketones (Table 2).²⁰ Various α-diazo alcohols bearing different substituents at the para positions of the phenyl rings performed well, affording the corresponding products (3ba-3fa) in good to excellent yields. Notably, the halogen substituents were all tolerated in the presence of a palladium catalyst, thus providing the opportunity for further transformations through coupling reactions (3da-3fa). In addition to the carbon-chain extension, this approach could also be effectively utilized for the ring expansion of cyclic ketones (3ga-3ja). Interestingly, the reaction proceeded well over substrates derived from dissymmetrical ketones, in which two different migration products can simultaneously be obtained. While using the chain dissymmetrical di-aryl ketones, the electron-rich aryl group migration is dominant in the selective formal C-C insertion process (31 vs. 31').

Table 2 Scope of α-diazo tertiary alcohols^a



^{*a*} Reaction conditions: **1** (0.12 M, 1.2 equiv.), **2a** (0.10 M, 1.0 equiv.), $Rh_2({}^tBuCO_2)_4$ (1.0 mol%), $[Pd(allyl)Cl]_2$ (2.0 mol%), and Xantphos (2.2 mol%) in toluene at room temperature. Yields represent isolated yields.

Table 3 Scope of functionalized allyl tert-butyl carbonates^a



^{*a*} Reaction conditions: **1a** (0.12 M, 1.2 equiv.), **2** (0.10 M, 1.0 equiv.), $Rh_2({}^{t}BuCO_2)_4$ (1.0 mol%), [Pd(allyl)Cl]₂ (2.0 mol%), and Xantphos (2.2 mol%) in toluene at room temperature. Yields represent isolated yields. ^{*b*} Reaction carried out at 60 °C.

In contrast, the alkyl group migrates prior to the aryl group (**3m** *vs.* **3m**'), when the α -diazo alcohol (derived from benzo-cyclohexanone) was employed. The migration process revealed in this case is similar to that in previous reports.^{13b,c}

Next, we turned our attention to study an array of functionalized allyl tert-butyl carbonates. As shown in Table 3, the preparative scope was in general observed regardless of whether electron-donating (2c-f) or electron-withdrawing (2g-k) groups were present or not. In particular, substrates having fluoro (2g), chloro (2h-j), and bromo (2k) groups were well-tolerated, giving the corresponding products (3ag-3ak) in excellent yields. Similarly, the 1-naphthyl or 2-thiophenyl substituted allyl carbonate (2l or 2m) was also successfully incorporated into the product, 3al or 3am obtained in high yield. Although the reaction rate decreased in the case of the substituents on the ortho-positions of the aromatic ring of γ -substituted allyl carbonates (2d, 2h, and 2l), the efficiency of this transformation was not noticeably affected by the position of the substituents. Gratifyingly, this method could be extended to 3-methyl-substituted allyl carbonate (2n) (R = CH₃), in which the palladium complex did not rapidly undergo β-hydride elimination.

Asymmetric dual transition-metal catalysis is an enduring challenging subject,²¹ in part due to the redox-compatibility of the catalysts, catalyst deactivation, and the need for balanced kinetics. This is required to suppress undesired catalytic pathways and to come up with a controlled reaction sequence.¹⁹ Next, we also attempted to explore an asymmetric relay Rh(π)/Pd(0) dual catalysis for gaining access to chiral acyclic α -quaternary allylated β -ketoesters. First, the reaction was



Scheme 3 Asymmetric one-pot synthesis of chiral acyclic α -quaternary allylated β -keto-esters.

conducted at room temperature by combining $Rh_2({}^{t}BuCO_2)_4$ with Ito's {[Pd(allyl)Cl]₂-(*R*)-BINAP} catalyst, 22a,b which was the most enantioselective catalyst for the asymmetric allylic alkylation of cyclic 1,3-diketones. 22c The response of the reaction was only the racemic product **3aa** in 80% yield. After a series of efforts (see Table S1, ESI†), chiral ligand **L8** shows moderate reactivity and enantio-selectivity, affording **3aa** in 59% yield and 53% ee (Scheme 3).

In summary, we have developed a highly efficient relay $Rh(\pi)/Pd(0)$ dual catalysis for the one-pot synthesis of acyclic α -quaternary allylated β -ketoesters. Under mild conditions, this binary catalyst system rendered domino [1,2]-sigmatropic rearrangement/allylic alkylation of α -diazo tertiary alcohols, in which two separate C–C σ -bonds on the same carbon atom were formed in an upfront manner. Further extension of the $Rh(\pi)/Pd(0)$ dual-catalyzed system with diazo compounds is underway in our laboratory.

The current work was made possible by the support of the National Natural Science Foundation of China (No. 21402154), the Natural Science Foundation of Shaanxi Province (No. 2019JQ647 and 2018JQ2072), the Chinese Universities Scientific Fund (No. 2452016093), and the Youth Training Project of Northwest A&F University (No. 2452017034).

Conflicts of interest

There are no conflicts to declare.

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