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Rhodium-catalyzed regio- and enantioselective allylic alkylation of pyrazol-5-ones with alkynes†

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A rhodium-catalyzed asymmetric allylic alkylation of pyrazol-5-ones with internal alkynes is illustrated. In the presence of a chiral rhodium-hydride catalyst, functionalized heterocyclic products bearing an all-carbon quaternary stereogenic center were obtained in high yields with satisfactory enantioselectivities. This protocol also features good regiocontrol and a high atom economy without stoichiometric by-product formation.

Pyrazol-5-ones and related heterocycles are prevalent structural units in natural products and synthetic pharmaceutical molecules.[1] In particular, optically pure pyrazol-5-ones bearing all-carbon quaternary stereocenters exhibit remarkable biological activities in drug discovery (Fig. 1).[2] For instance, capromorelin tartrate is an orally active and potent growth hormone secretagogue (GHS).[2a,b] Spiropyrazolones I and II show good anti-tumor and anti-inflammatory properties, respectively.[2c,d] Therefore, numerous efforts have been devoted to the synthesis of these compounds.[3]

Owing to the versatility of the allyl group, transition-metal-catalyzed asymmetric allylic alkylations (AAAs) have been established as a powerful and reliable tool in organic transformations.[4,5] However, due to the existence of tautomeric forms,[6] the regio- and enantioselective allylation of pyrazol-5-ones is by no means an easy process (Scheme 1).[7] In 2013, Gong's group developed the first highly efficient asymmetric allylic alkylation of pyrazol-5-ones with allylic alcohols *via* cooperative catalysis (chiral Pd and chiral phosphoric acid)[7a] (Scheme 1a). Later, this cooperative strategy was extended by the same group to enantioselective allylic C–H alkylation of pyrazol-5-ones with allylarenes in the presence of an oxidant (Scheme 1b).[7b,e] In 2017, the Jiang group also reported a significant Pd-catalyzed asymmetric allylation of pyrazol-5-ones with alkoxyallenes in branched selectivities (Scheme 1c).[7c] Despite these important advances, the development of an effective and economic pathway to access chiral allylic pyrazol-5-ones with non-activated precursors under redox-neutral conditions is still highly appealing.

Since alkynes are easily accessible and bench-stable synthons in organic synthesis, the employment of alkynes as greener allyl precusors is becoming an attractive alternative for the buildup of molecular complexity.[8] In this context, elegant studies that employ terminal or internal alkynes in AAAs have been contributed by the Yamamoto, Breit, Krische and Dong groups.[9–11] However, the direct utilization of alkynes to construct all-carbon quaternary stereocenters is lacking. In 2017, Dong and co-workers reported the only example to generate vicinal quaternary and tertiary carbon stereocenters *via* an enantioselective coupling between α-branched aldehydes and alkynes.[9g] Besides, all the reported studies on rhodium-catalyzed AAAs with alkynes gave branched allylic products, and how to switch the regioselectivity is a challenging problem. Considering this synthetic importance together with our continuous investigation on metal-hydride catalysis,[12] we report herein a regio- and enantioselective allylation of pyrazol-5-ones with alkynes under atom-economical Rh-catalysis.

Initially, we began our investigations with coupling of pyrazol-5-one **1a** and commercially available 1-phenyl-1-propyne **2a**. As highlighted in Table 1, after examining a range of achiral bisphosphine ligands, we found that the bite angle of the ligands exhibited a significant influence on the efficiency of this allylic

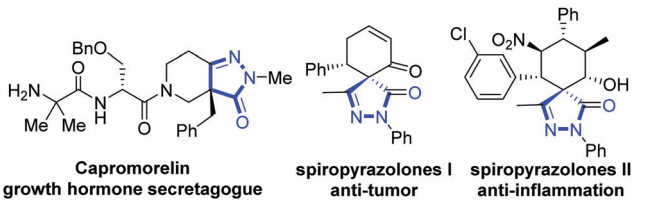

Fig. 1 Bioactive molecules containing pyrazol-5-one skeletons.

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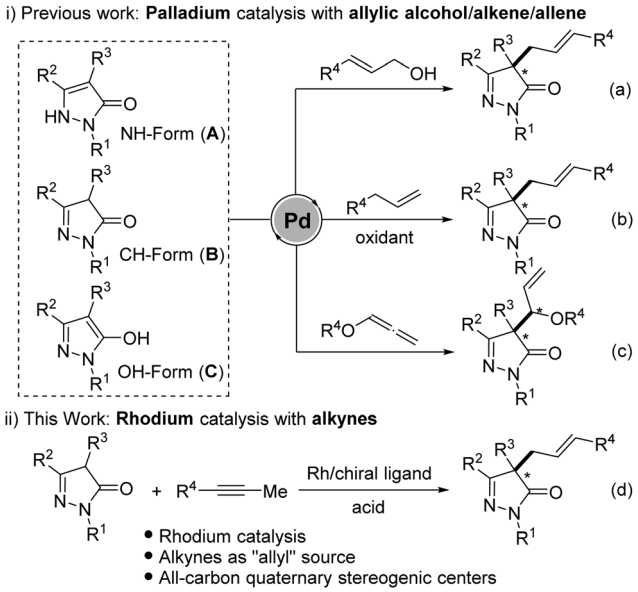
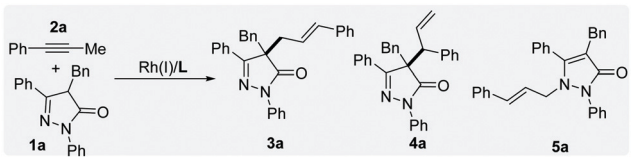


Scheme 1 Transition-metal-catalyzed AAAs of pyrazol-5-ones.

alkylation (Table 1a). The ligands with a moderate bite angle such as dppf and dppb showed good reactivities but produced a mixture of **3a**, **4a**, and **5a**. Although a higher temperature diminished the regioselectivity, an enhanced result was achieved when the reaction was conducted at 50 °C (Table 1b). Based on these preliminary results, we turned our attention to enantioselective control. The linear **3a** was obtained as a major product in 28% yield and 55 : 45 er with (*R*)-BINAP (**L1**) as the ligand and $PhCO_2H$ as the acid co-catalyst (Table 1c). Delightfully, the bulky ligand bearing the DTBM-phosphine substituent (**L3**) showed better performance in both reactivity and enantioselectivity and the reaction delivered **3a** in 68% yield and 84 : 16 er. The other axial ligands such as SEGPHOS (**L4**), MeO-BIPHEP (**L5**), and GARPHOS (**L6**) provided lower reactivities and selectivities. The good performance of the ligand **L3** was probably ascribed to its bulky substituents and comparably large dihedral angle. Futher evaluation of acids indicated that $(PhO)PO_2H$ was the best acid additive in terms of regio- and enantioselectivity as well as reactivity (Table 1d). Eventually, decreasing the temperature to 35 °C led to the product **3a** in 89% yield with >19 : 1 rr and 91 : 9 er. The disfavoring of branched selectivity might be attributed to steric hindrance.

With the optimized reaction conditions in hand, we next investigated the scope of pyrazol-5-ones (Table 2). Generally, various pyrazol-5-ones were allowed to react with alkyne **2a** and gave the corresponding products in satisfactory yields and selectivities. Pyrazol-5-ones bearing different aryl substituents at the C5 position (R^1) all proceeded smoothly under the current reaction conditions, leading to the products **3a** to **3e**

Table 1 Optimization of the reaction conditions[a]



a) Ligand bite angle effects (with $PhCO_2H$ as acid)

Increasing Bite Angle

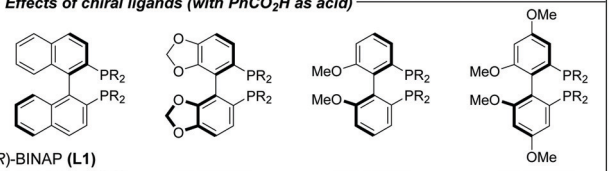
Ligand:	dppm	dppe	dppp	dppf	dppb	DPEphos	Xantphos
Yield (%) **3a/4a/5a**:	11/3/5	5/<2/<2	7/2/6	77/5/22	47/12/41	<2/<2/<2	10/4/13

b) Temperature effects (with dppf as ligand)

Increasing Temperature

Temp:	50 °C	70 °C	90 °C	110 °C
Yield (%) **3a/4a/5a**:	79/8/13	77/5/22	73/<2/26	46/<2/20

c) Effects of chiral ligands (with $PhCO_2H$ as acid)



(*R*)-BINAP (**L1**) (*R*)-DM-BINAP (**L2**) (*R*)-DTBM-BINAP (**L3**) | R = DTBM SEGPHOS (**L4**) | R = DTBM MeO-BIPHEP (**L5**) | R = DTBM GARPHOS (**L6**)

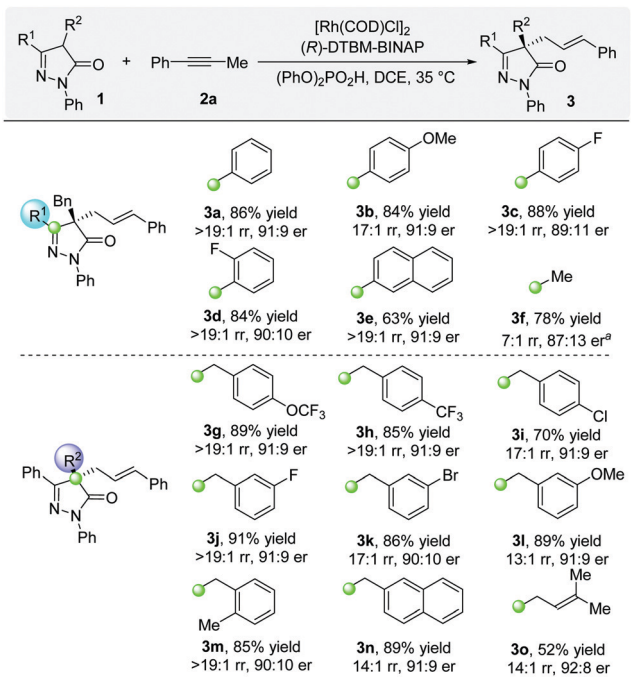
Ligand:	L1	L2	L3	L4	L5	L6
Yield (%) **3a/4a/5a**:	28/3/6	72/10/16	68/20/11	11/5/12	29/12/8	13/6/5
er of **3a**:	55:45	52:48	84:16	56:44	79:21	75:25

d) Effects of acids (with L3 as ligand)

Acid:	$PhCO_2H$	$AdCO_2H$	TsOH	$(PhO)_2PO_2H$	$(^nBuO)_2PO_2H$
Yield (%) **3a/4a/5a**:	68/20/11	58/16/9	43/<2/2	87/3/2 (89/2/2)[b]	84/9/6
er of **3a**:	84:16	85:15	90:10	90:10 (91:9)[b]	89:11

[a] Reaction conditions: **1a** (0.10 mmol), **2a** (0.20 mmol), $[Rh(COD)Cl]_2$ (2.5 mol%), ligand (5.0 mol%), acid (20 mol%), DCE (0.25 M), 50 °C, 18 h. In all cases, the yield was determined by ^1H NMR analysis with 1,3,5-trimethoxybenzene as the internal standard, and er was determined by chiral HPLC. [b] 35 °C, 3 d.

Table 2 Scope of pyrazol-5-one substrates



Reaction conditions: **1** (0.10 mmol), **2a** (0.20 mmol), $[Rh(COD)Cl]_2$ (2.5 mol%), (*R*)-DTBM-BINAP (5.0 mol%), $(PhO)_2PO_2H$ (20 mol%), DCE (0.25 M), 35 °C, 3 d. In all cases, the isolated yields were given; the rr was determined by ^1H NMR analysis of the crude reaction mixture, and er was determined by chiral HPLC. [a] The absolute configuration was assigned by comparing the optical rotation in the literature (ref. 7*a*).

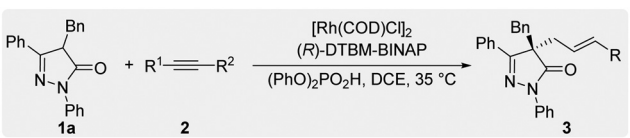
in moderate to good yields (63–88%) with good selectivities. The substrate having a methyl group at the C5 position of the pyrazolones was also compatible and delivered the product **3f** in 78% yield with 7 : 1 rr and 87 : 13 er. Substituents at the quaternary carbon position were then examined. The electronic nature of the substituents (electron-donating or electron-withdrawing groups) on the benzyl ring exerted no significant impact on the yields and selectivities (**3g–3n**). Various groups such as 4-OCF_3, 4-CF_3, 4-Cl and 3-F could be well tolerated and the corresponding products **3g–3j** were obtained in 70–91% yields with 91 : 9 er. Notably, the bromo group, which is easily eliminated under palladium catalysis, remained intact under the current reaction conditions, and the product **3k** was produced in 86% yield with 17 : 1 rr and 90 : 10 er. The substrates bearing a sterically hindered group (**3m**) or a naphthyl group (**3n**) also reacted well with **2a** and generated the products in good yields and selectivities. To our delight, prenyl-substituted pyrazol-5-one was also applicable under the current conditions and led to the product **3o** in 52% yield with 14 : 1 rr and 92 : 8 er.

Next, the generality of alkynes was tested (Table 3). The substrates possessing electron-withdrawing groups at the 4-position of the phenyl ring all reacted with **1a** smoothly and provided the allylic products in 53–79% yield with >19 : 1 rr and 91 : 9 to 92 : 8 er (entries 1–4). In comparison, the alkynes with electron-donating groups at the para position of phenyl were a bit sluggish in the current protocol (entries 5 and 6). However, good yield and regioselectivity could be observed when the methyl group was located at the meta-position of the phenyl ring (entry 7). Due to the steric hindrance, a decrease in the yield and enantioselectivity was observed when *o*-methylphenyl substituted alkyne was used (entry 8). On replacing the phenyl group with a naphthyl group, the target allylic product was furnished in 92% yield with >19 : 1 rr and 89 : 11 er (entry 9). 2-Thienyl derived alkyne was also suitable for this reaction and led to **3y** in 25% yield (entry 10). Unfortunately, subjecting 2-butyne to coupling with **2a** under the standard conditions only gave the product **3z** in 15% yield with low regio- and enantioselectivity (entry 11). Finally, 16% yield of **3a** was obtained with the terminal alkyne as a substrate and the selectivities were well maintained (entry 12).

To probe the possibility of the allene intermediate, the pre-synthesized phenylallene **6** was subjected to coupling with pyrazol-5-one **2a** under the standard conditions, and the product **3a** was indeed observed with a comparable er, albeit in a low yield (Scheme 2a). This result also suggests that alkynes could serve as good allene surrogates in some catalytic reactions.[9d] Then an isotopic-labeling study was performed. When the deuterated alkyne **2a**-d_3 was employed as a substrate, the deuterium atom was found to incorporate into the α-, β-, and γ-positions of the allyl unit (Scheme 2b). This observation indicates that a reversible hydrometallation might be involved during the allene formation. Additional control experiments showed that the products **3a** and **5a** remained intact under the standard reaction conditions, while 14% of **4a** was found to be transformed into **5a** (Scheme 2c). The isomerization reaction from **4a** to **5a** presumably resulted from an aza-cope rearrangement process.[9i] This result also indicates that the selectivity of the product **3a** does not derive from the isomerization of **4a** or **5a**.

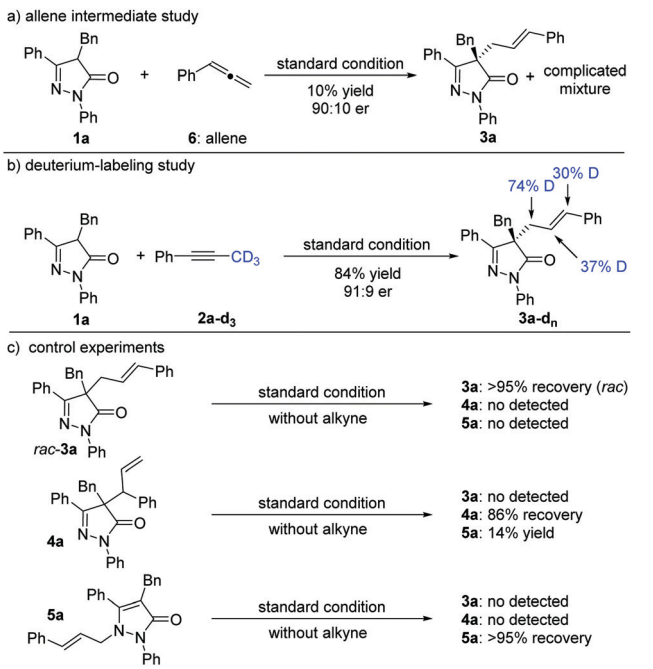
On the basis of the literature[7a–c,9] and our observations, we proposed a pathway *via* tandem Rh-catalysis (Fig. 2). First, the oxidative addition of the Brønsted acid with a Rh(I) precursor generates Rh(III)-hydride species **A**. Then the *syn*-migratory insertion of alkyne **2a** into Rh(III)–H **A** affords the vinyl-Rh intermediate **B**. The following β-hydride elimination from **B** gives the allene **6** intermediate and regenerates the Rh(III)–H

Table 3 Scope of alkyne substrates



Entry	R^1	R^2	Yield of **3** (%)	rr	er
1	4-FC_6H_4	Me	**3p**, 74	>19 : 1	91 : 9
2	4-ClC_6H_4	Me	**3q**, 79	>19 : 1	91 : 9
3	4-BrC_6H_4	Me	**3r**, 77	>19 : 1	92 : 8
4	4-$CF_3C_6H_4$	Me	**3s**, 53	>19 : 1	92 : 8
5	4-MeC_6H_4	Me	**3t**, 43	>19 : 1	89 : 11
6	4-$MeOC_6H_4$	Me	**3u**, 39	>19 : 1	88 : 12
7	3,5-$Me_2C_6H_3$	Me	**3v**, 87	>19 : 1	89 : 11
8	2-MeC_6H_4	Me	**3w**, 30	>19 : 1	87 : 13
9	2-Naphthyl	Me	**3x**, 92	>19 : 1	89 : 11
10	2-Thienyl	Me	**3y**, 25	15 : 1	90 : 10
11	Me	Me	**3z**, 15	2 : 1	54 : 46
12	Benzyl	H	**3a**, 16	>19 : 1	91 : 9

Reaction conditions: **1a** (0.10 mmol), **2** (0.20 mmol), $[Rh(COD)Cl]_2$ (2.5 mol%), (*R*)-DTBM-BINAP (5.0 mol%), $(PhO)_2PO_2H$ (20 mol%), DCE (0.25 M), 35 °C, 3 d. In all cases, the isolated yields were given; the rr was determined by ^1H NMR analysis of the crude reaction mixture, and er was determined by chiral HPLC.



Scheme 2 Mechanistic studies.

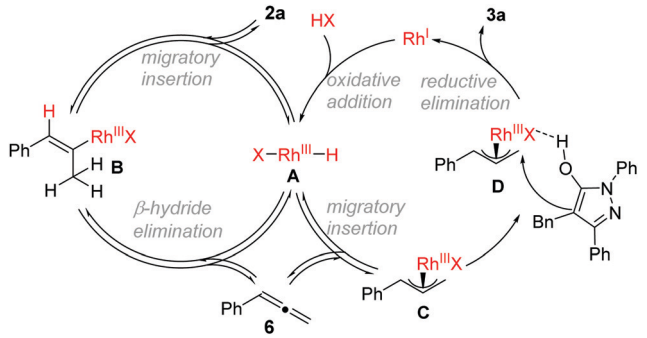


Fig. 2 Proposed mechanism.

species **A**. Next, the reinsertion of the terminal allene **6** into Rh(III)–H yields the chiral π-allyl rhodium complex **C**, which shows hydrogen-bonding interaction with the enol form of pyrazolone **1a**. Then the enol motif undergoes nucleophilic attack at the carbon center to deliver the allylic product **3a** and generate Rh(I) species.

In summary, we have described a rhodium-catalyzed enantioselective allylic alkylation of pyrazol-5-ones with internal alkynes. With the assistance of rhodium-hydride catalysis, functionalized heterocyclic products bearing an all-carbon-substituted quaternary stereogenic center were formed in high yields with good regio- and enantiocontrol. This method provides an atom-economical paradigm for exploiting asymmetric allylic alkylations of other heterocyclic ketones.

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Conflicts of interest

There are no conflicts to declare.

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