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Gold-Catalyzed Divergent Ring-Opening Rearrangement of Cyclopropenes Enabled by Dichotomous Gold–Carbenes

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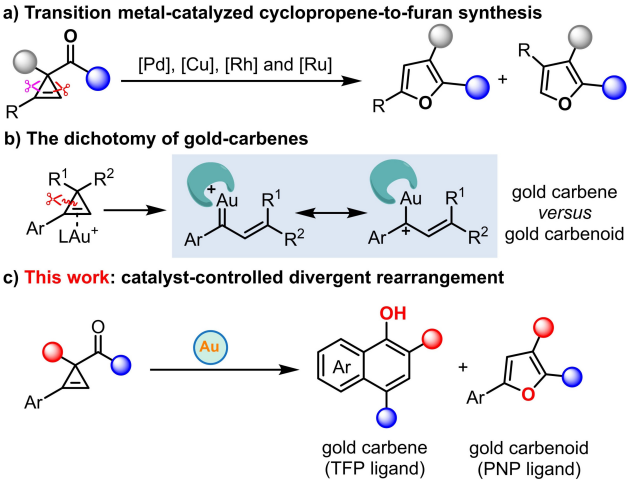
Dedicated to Professor A. Stephen K. Hashmi on the occasion of his 60th birthday.

Abstract: The gold-catalyzed ring-opening rearrangement of cyclopropenes affords an efficient route to either polysubstituted naphthols or aryl-substituted furans. Owing to the unique dichotomy of gold–carbenes, this protocol provides a switchable reaction selectivity between naphthols and furans enabled by the use of TFP–Au(MeCN)SbF_6 (tri(2-furyl) phosphine) or PNP($AuNTf_2$)$_2$ (bis(diphenylphosphino)(isopropyl) amine) as catalysts respectively. It is proposed that the gold–carbene intermediate might be involved in the cyclopropene→naphthol rearrangement while the gold-carbocation is more likely to be involved in the cyclopropene→furan rearrangement.

Owing to their special reactivity and unique ring-strain, ring-opening rearrangements of cyclopropene have attracted attention during the past decades.[1] Among these, a transition metal-catalyzed controllable rearrangement for the concise synthesis of furan derivatives using metal–carbene intermediates has been actively pursued. Thus, the direct employment of toxic diazo compounds as carbene precursors can be avoided (Scheme 1a).[2] For example, a series of transition metal catalysts, such as Pd, Cu, Rh and Ru have been widely explored to realize the synthetically useful cyclopropene-to-furan rearrangement.[3] On the other hand, gold is regarded as an elegant π-acid catalyst for the activation of π-bonds.[4] Gold-catalyzed ring-opening rearrangements of cyclopropenes have attracted considerable interest from experimental and theoretical chemists,[5] and gold–carbene has long been regarded as an important intermediate in gold-catalyzed reactions but the true structure of "gold–carbene" is still a subject of controversy.[6] Interestingly, the isolation of gold-coordinated carbocations generated in the ring-opening reactions of cyclopropenes revealed a gold–carbene resonance structure,[7] whose reactivity



Scheme 1. The dichotomy of gold–carbenes in the divergent ring-opening rearrangement.

differs from compounds in which Au activation of π-bonds exists (Scheme 1b). Notwithstanding these seminal findings, gold-catalyzed organic transformations of cyclopropenes with controlled gold–carbene or gold–carbenoid features is of considerable interest.[8] As the electronic feature of gold catalysts with different ligands and counterions can be well tuned, we undertook exploration of the gold-catalyzed divergent reactivity of the ring-opening rearrangements of cyclopropenes. Inspired by the seminal work of Shi et al.[9] and Zhang's group,[10] we hypothesized that the choice of an appropriate gold-catalyst would be crucial to the achievement of different chemoselectivity.[11] As our interests in homogenous gold catalysis,[12] we herein describe a divergent ring-opening rearrangement of cyclopropenes[13] to polysubstituted naphthols with TFP–Au(MeCN)SbF_6 (tri(2-furyl) phosphine) as the catalyst. The use of PNP($AuNTf_2$)$_2$ (bis(diphenylphosphino)(iso-

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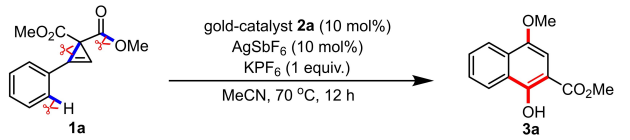
propyl) amine) as a catalyst converts the same substrates into furan skeletons.

To explore the rearrangement reaction of cyclopropene, we tested some gold-catalysts, silver salts and other additives (see Table 1 and Supporting Information for details). Finally, it is found that the optimized reaction conditions include dimethyl 2-phenylcycloprop-2-ene-1,1-dicarboxylate (**1a**) as the carbene precursor with chloro(tri(2-furyl) phosphine) gold(I) (**2a**) as the catalyst, $AgSbF_6$ as a chloride-abstractor and KPF_6 as an additive in MeCN at 70 °C for 12 h. This furnished the polysubstituted 1-naphthol (**3a**) in 81% yield. The structure of **3a** was clearly proved by X-ray single crystal diffraction (see Supporting Informations). Notably, synthesis of this kind of naphthalene-1,4-diol skeleton is very challenging but the structure is prevalent in a series of bioactive molecules, such as Karamomycin C, Rubinaphthin A and Mollugin (Scheme 2).[14] Without the addition of either gold-catalyst or silver additives under the optimized conditions, this reaction does not proceed (entries 2 and 3). A 33% yield was obtained in the absence of KPF_6 (entry 4). The use of $AgNTf_2$ instead of $AgSbF_6$ reduces the yield of **3a** to 50% (entry 5) while $Ph_3PAuNTf_2$ produces only 5% of the target product (entry 6). This may indicate that the furan-based phosphine ligand is important for the formation of a polysubstituted 1-naphthol (**3a**).

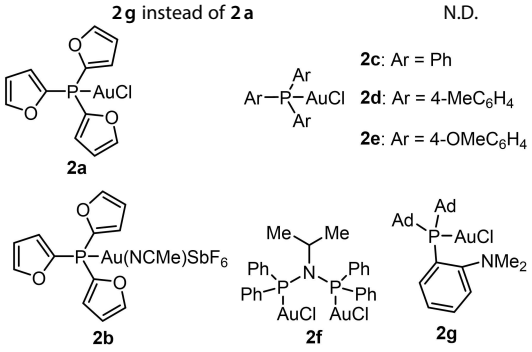
We prepared and isolated the possible cationic-type gold-catalyst, $TFPAu(MeCN)SbF_6$ (**2b**). As shown in entry 7 in Table 1, with **2b** as gold-catalyst in the absence of silver salt additives, the desired reaction can proceed smoothly to afford product (**3a**) in 80% yield, almost equivalent to the optimized reaction conditions which gave an 81% yield (entry 1). This eliminates the catalytic influence of $AgSbF_6$ or AgCl generated in situ in the standard reaction conditions. Different gold-catalysts (**2c**–**2g**) were also investigated (entries 8–12) but were not found to improve the catalytic efficiency.

With the optimized reaction conditions in hand (entry 1, Table 1), we then examined the substrate scope of this cyclopropene→1-naphthol transformation (Scheme 3). A series of cyclopropenes bearing different alkyl group substituents on the phenyl rings were investigated and they uniformly provided the corresponding alkyl substituted naphthols (**3b**–**3i**) in moderate to good yields. Importantly, several compounds with halogen substituents on the phenyl ring (**3j**–**3o**) tolerated the reaction conditions well. In particular, the bromo- and iodo-substituents (**3l**, **3m**) remain unchanged during the ring-opening rearrangement. It was also found that the substituents on the *ortho*-, *meta*- and *para*-position of the phenyl group (**3j**, **3n**, **3o**) have little influence on the efficiency of the reaction. When naphthalene-substituted cyclopropenes (**3p** and **3q**) were employed, the cyclopropene ring-opening rearrangement gave rise to polysubstituted phenanthrene compounds in a synthetically useful yield of 64% while polysubstituted anthracenes were not detected, implying the good regioselectivity of this protocol. Interestingly, it was found that the electron-rich methoxy group (**3r**, **3s**), and the trimethylsilyl group (**3t**, **3u**) are also tolerated, and undertake this rearrangement readily, affording the desired products in acceptable yields. Esters other than the methyl ester were also studied and it was found that they can participate in this transformation to produce the products (**3v**, **3w**) in 45–71% yields. The 3-thiophene-substituted cyclopropenes are competent substrates, and regioselectively afford the products (**3x**) in 42% yield. Notably, all the examples described here and illustrated in Scheme 3 show specific regioselectivity during the rearrangement. It was expected that the electronic effect on the aromatic rings would play an important role in controlling the reaction regioselectivity, and indeed when strongly electron-withdrawing groups such as $-CF_3$, –COOMe and $-NO_2$ were present in the phenyl groups, this ring-opening rearrangement reaction to naphthol became very sluggish and little of the desired product was obtained.

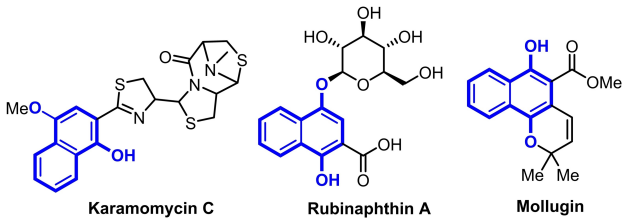
Table 1. Optimization of reaction conditions.[a]



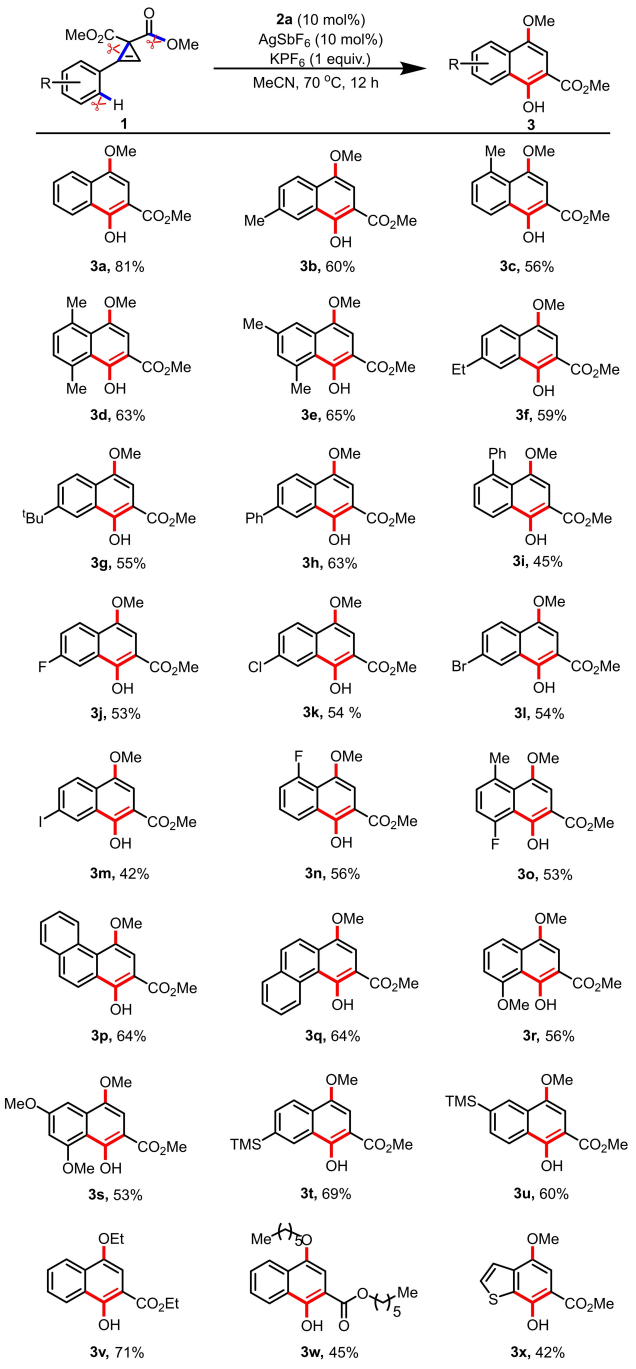
Entry	Variation of conditions	Yield[b]
1	None	**81%**
2	No gold-catalyst	N.D.
3	No Ag salt	N.D.
4	No KPF_6	33%
5	$AgNTf_2$ instead of $AgSbF_6$	50%
6	$Ph_3PAuNTf_2$ instead of **2a**	5%
7	**2b** instead of **2a**	80%
8	**2c** instead of **2a**	70%
9	**2d** instead of **2a**	44%
10	**2e** instead of **2a**	48%
11	**2f** instead of **2a**	25%
12	**2g** instead of **2a**	N.D.



[a] Standard conditions: gold catalyst (10 mol%), **1a** (0.1 mmol, 1 equiv.), KPF_6 (0.1 mmol, 1 equiv.), $AgSbF_6$ (10 mol%), MeCN (1 mL), 70 °C, 12 h. [b] Yield of isolated product. N.D. = not detected.



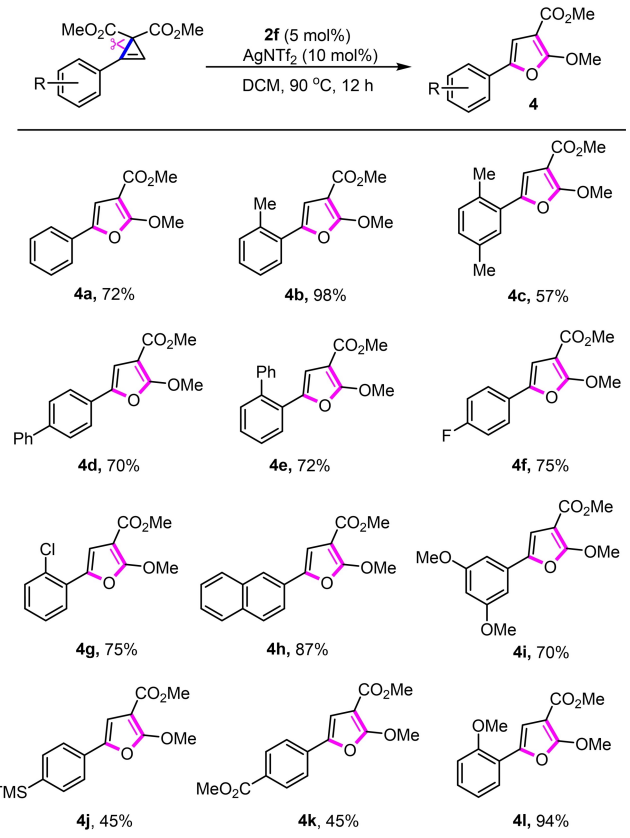
Scheme 2. The occurrence of naphthalene-1,4-diol skeletons in biologically important molecules.



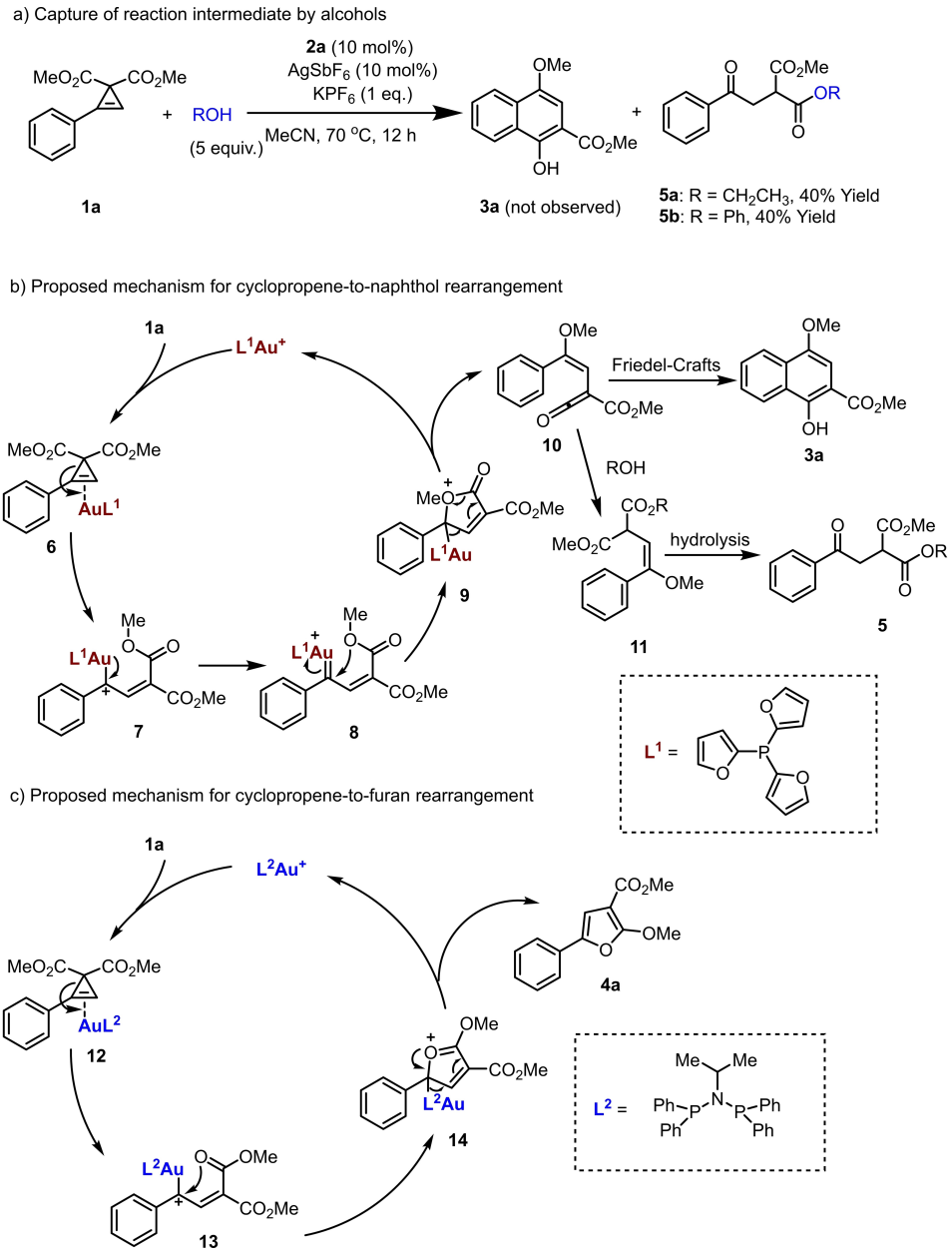
Scheme 3. Reaction scope. Reaction conditions: gold catalyst **2a** (10 mol%), $AgSbF_6$ (0.01 mmol), **1** (0.1 mmol), KPF_6 (0.1 mmol), MeCN (1 mL), 70 °C, 12 h.

Subsequently, we studied optimization of the reaction conditions which achieve the cyclopropene-to-furan rearrangement. It was interesting to find that the combination of gold-catalyst **2f** and $AgNTf_2$ is able to change the reaction product from polysubstituted naphthol to aryl-substituted furan (see Supporting Information for details). This is probably due to the different ligand and gold affinity index (GAI) of two counterions (for SbF_6^- is 0 and of NTf_2^- is 2.9.).[11c,d] A 5 mol% loading of $PNP(AuCl)_2$ (**2f**) as gold catalyst together with 10 mol% $AgNTf_2$, can totally avoid the naphthol-type rearrangement, affording exclusively the furan-based products (Scheme 4). The optimized reaction conditions for the cyclopropene→furan conversion should include 5 mol% gold-catalyst (**2f**) together with 10 mol% $AgNTf_2$ at 90 °C for 12 h. This gave the desired product (**4a**) regio-exclusively in 72% yield. Several aryl-substituted cyclopropenes were subjected to the standard conditions, and they follow this rearrangement reaction readily, delivering the aryl-substituted furans (**4a**–**4l**) in yields of up to 98%. Surprisingly, we found that an electron-withdrawing group on the phenyl rings can tolerate the cyclopropene-to-furan transformation well, giving rise to the desired product (**4k**) in moderate yield, but failed to undergo the gold-catalyzed cyclopropene→naphthol reaction. This may suggest that the two rearrangements proceed by different reaction pathway.

To gain insight into a possible reaction mechanism of the cyclopropene-to-naphthol rearrangement, we used alcohol as a nucleophilic agent to capture the intermediates potentially formed during the reaction process. As shown in Scheme 5a, when 5 equivalents of ethanol and phenol were added into the reaction mixture, the cyclopropene→naphthol rearrangement was significantly suppressed, and none of the desired naphthol (**3a**) was detected by GC-MS. However, new products, 1-ethyl 3-methyl 2-(2-oxo-2-phenylethyl) malonate (**5a**) and 1-methyl 3-phenyl 2-(2-oxo-2-phenylethyl) malonate (**5b**) were isolated



Scheme 4. Scope of furan production. Reaction conditions: gold catalyst **2f** (5 mol%), **1** (0.1 mmol), $AgNTf_2$ (10 mol%), DCM (1 mL), 90 °C, 12 h.



Scheme 5. Possible mechanism for divergent ring-opening rearrangement of cyclopropenes.

both in 40% yield. This suggests that a carbonyl cation or ketene type intermediate may be involved in this transformation.

Based on our experimental observations, we propose a plausible mechanism for both the cyclopropene-to-naphthol rearrangement and the cyclopropene-to-furan rearrangement. As shown in Scheme 5b, the cationic LAu^+ initially generates in situ a complex with the π bond in cyclopropene (**1a**). This produces complex (**6**), which can undergo the cleavage of C1–C3 and ring opening to form the gold carbene intermediate (**8**) through resonance from intermediate **7**.[15] The generated intermediate (**9**) may undergo a rearrangement process to generate a ketene intermediate (**10**), which undergoes an intramolecular Friedel-Crafts-like reaction to give the product (**3a**).[16] This would also account for the failure of electron-withdrawing groups on the phenyl rings. The addition of alcohols led to the formation of intermediate (**11**), which is subsequently hydrolyzed to give a product (**5**). As proposed in Scheme 5c, similar to the initial steps of the cyclopropene→naphthol conversion, the cyclopropene→furan transformation also starts with the π-activation of cyclopropene, initiating a ring-opening to deliver the resonance-stabilized carbocation intermediate (**13**) as the dominant intermediate. The subsequent intramolecular nucleophilic attack of carbonyl group on the carbocation leads to intermediate (**14**), and rearomatization gives the product

(**4a**).[17] Among these two distinct pathways, the electronic effect of ligands might play an important role to the transition-state of intermediates. For the use of gold-catalyst **2a** that equipped with an electron-rich furan-ring substituted phosphine ligand (**L**[1]), the strong π-donation from gold to C1 would favor the formation of gold–carbene intermediate. When the gold-catalyst equipped with PNP ligands (**2f**), the relatively electron-poor ligands because of the electronegativity of nitrogen atom, would reduce the π-donation from gold to C1 and thus the gold-carbocation form might be more favorable.

In summary, we have developed a protocol for the divergent gold-catalyzed transformations of cyclopropenes to either polysubstituted naphthols or aryl-substituted furans. With the same reactants, the use of $TFPAu(MeCN)SbF_6$ generally affords the cyclopropene→naphthol rearrangement while the use of $PNP(AuNTf_2)_2$ as gold catalyst delivers aryl-substituted furans. A key factor may be due to the unique dichotomy of gold–carbene and gold–carbenoid. Gold-catalyzed organic transformations of cyclopropenes with controlled gold–carbene or gold–carbenoid is of considerable interest in organic synthesis.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Deposition Number 2197633 (for **3a**) contains the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service.

Keywords: divergence · gold catalysis · gold–carbene · homogeneous catalysis · rearrangement

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