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1,2-Gold Carbene Transfer Empowers Regioselective Synthesis of Polysubstituted Furans

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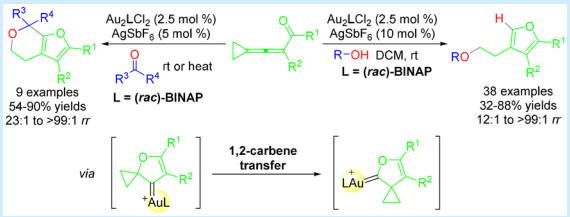
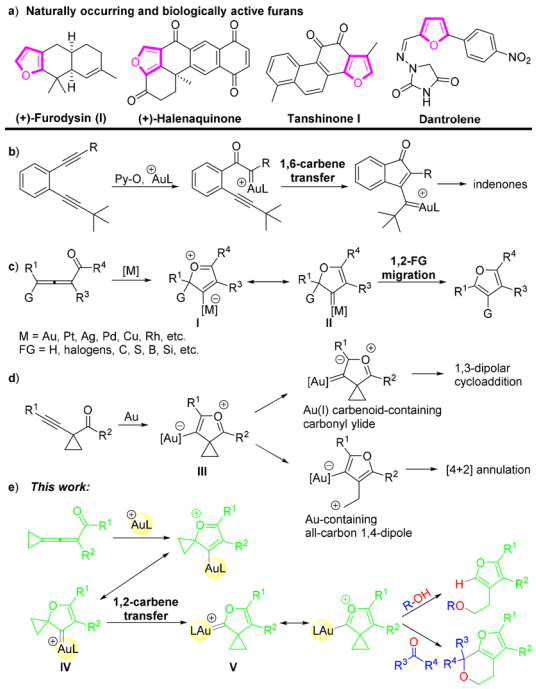
Supporting Information

ABSTRACT: A gold-catalyzed cascade cycloisomerization/ring-opening reaction of allenyl ketones bearing a cyclopropyl moiety with a wide variety of alcohols or ketones is developed. This reaction involves an unprecedented 1,2-gold carbene transfer to provide regioselective and modular access to tri- or tetrasubstituted furans in moderate to high yields and with broad substrate tolerability.



Polysubstituted furans are frequently found as key structure motifs in many bioactive natural products,[1] pharmaceutically important substances,[2] and functional materials,[3] and they also serve as versatile building blocks in organic synthesis (Scheme 1a).[4] Although much progress has been made on the synthesis of multiple substituted furans via transition-metal-catalyzed reactions,[5] development of mild and practical synthetic methods to access this privileged heterocyclic core by utilizing new catalytic properties of transition metals is still of great significance in organic chemistry.

Scheme 1. Background and Working Hypothesis



Homogeneous gold catalysis has grown explosively during the past decade to become one of the most exciting research fields for rapidly building molecular complexity.[6] In particular, considerable attention has been paid to gold-catalyzed generation of carbene species[7] from some typical substrate precursors,[8] together with exploration of their new reactivities. However, developing new reactions involving formation of gold carbene complexes based on these strategies, thereby permitting carbene species transfer from one carbon atom to another remains challenging to implement. In 2007, Toste's group[9] has demonstrated that a gold carbene can be transferred across an alkyne from a diazo substrate. Chan[10] and co-workers have disclosed a tandem process involving carbenoid transformation through an in situ formed cyclopropene intermediate. Notably, Hashmi[11] has recently reported an oxidative cyclization of diynes via 1,6-carbene transfer for the synthesis of indenones (Scheme 1b). While these types of gold carbene transfer are limited to the 1,6- or 1,7-positions, the short distance transfer such as 1,2-neighboring transfer, to the best of our knowledge, has not been reported to date.

On the other hand, allenyl ketones have been served as *cyclic metal carbene precursors* to afford a range of furans via 1,2-functional groups migration[12] (involving intermediates **I** and **II**, Scheme 1c). For example, Marshall[13] has illustrated the Rh^{I}- or Ag^{I}-catalyzed method for the synthesis of furans through a formal 1,2-hydrogen shift of allenyl ketones. A related strategy has also been explored by Hashmi[14] using Pd^{II} and $AuCl_3$ as efficient catalysts. In this context, Gevorgyan[15] and co-workers have presented fruitful results in transition-metals catalyzed cycloisomerization of allenyl ketones with various 1,2-FG (FG = alkyl, aryl, halide, acyloxy, silyl, boryl, etc.) migration as a key step in the assembly of highly substituted furans.

In addition, Zhang[16] has reported two interesting gold-catalyzed reactions using 1-(1-alkynyl)cyclopropyl ketones as an alternative substrate. The oxocarbenium species **III** was

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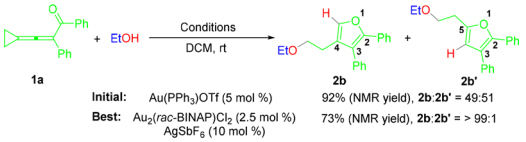
generated when the carbonyl group attacked the Au-activated C−C triple bond via 5-*endo-dig* cyclization. Further transformation of **III** would lead to conceptually novel Au-containing all-carbon 1,4-dipole or Au carbenoid-containing carbonyl ylides (Scheme 1d). In this area, we[17] have disclosed a $PdCl_2$-catalyzed cycloisomerization of cyclopropyl-tethered allenyl ketones to form furan-fused cyclobutenes with high potential via C−C bond cleavage of the strained ring. Inspired by these achievements and diverse gold catalysis, we conceived that allenyl ketones bearing a cyclopropyl moiety will be converted to cyclic gold carbene intermediate **IV** via 5-*endo-trig* cyclization in the presence of cationic gold(I) catalyst, which may be able to shift to the neighboring carbon to form cyclic gold carbene species **V** via adjacent cyclopropyl moiety consecutive scission (Scheme 1e). Here, we report the realization of 1,2-gold carbene transfer and the development of a gold(I)-catalyzed cycloisomerization/ring-opening reaction of allenyl ketones containing tethered cyclopropyl functionality with alcohols or ketones that delivers tri- or tetrasubstituted furans efficiently and regioselectively.

With this idea in mind, we initially investigated the transformation of cyclopropyl-tethered allenyl ketone **1a** with ethanol in DCM at room temperature in the presence of 5 mol % of $Au(PPh_3)OTf$. Gratifyingly, a mixture of 2,3,4-trisubstituted furan **2b** and 2,3,5-trisubstituted furan **2b′** (**2b**/**2b′** = 49:51) was afforded in 92% total yield (Scheme 2). We

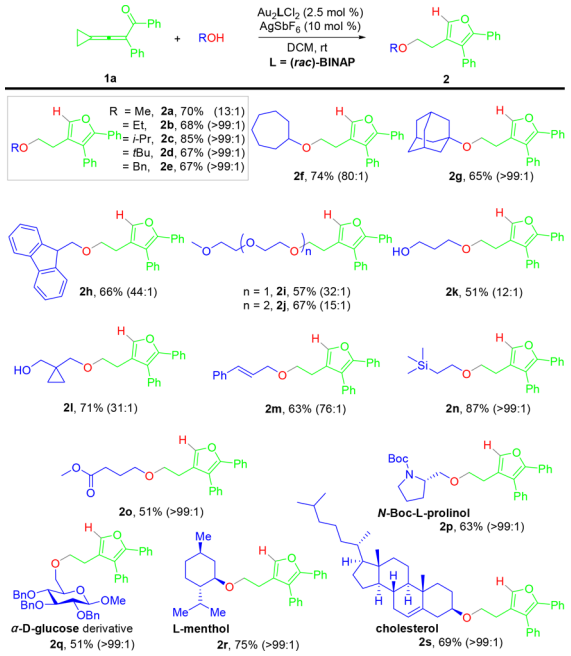
Scheme 2. Initial Attempt



presumed that the reaction involving 1,2-gold carbene transfer between cyclic carbene **IV** and **V** would yield the furan **2b**. Considering that the selective formation of **2b** is abnormal and particularly interesting, we then focused on optimizing the reaction with respect to **2b** including yield and selectivity. After an extensive survey of the reaction parameters (Table S1) including metals, anions, ligands, silver effect, and solvents, the optimal conditions were identified to be a mixture of Au_2(*rac*-BINAP)Cl_2 (2.5 mol %) and $AgSbF_6$ (10 mol %) as the catalyst, DCM as the solvent at room temperature, wherein the desired product **2b** was afforded in 73% yield, rr > 99:1.

With the above optimal conditions in hand, we examined the scope of alcohols using **1a** as the model substrate (Scheme 3). Simple aliphatic alcohols such as MeOH, EtOH, *i*PrOH, *t*BuOH, BnOH, and cycloheptanol (**2a**−**f**) afforded the desired functionalized furans in moderate to high yields. A moderate regioselectivity was observed when MeOH was employed. Sterically hindered 1-adamantanol (**2g**) was suitable for this reaction, yielding the corresponding product **2g** with good yield and selectivity. A variety of alcohols containing a fluorenyl (**2h**), polyether (**2i** and **2j**), hydroxyl (**2k**), cyclopropyl (**2l**), alkenyl (**2m**), silyl (**2n**), and ester (**2o**) functionalities were all smoothly and effectively accommodated into this process. Notably, such a gold-catalyzed carbene-transfer reaction was applied to the synthesis of furans containing bioactive moieties. Natural occurring alcohols and their derivatives such as *N*-Boc-L-prolinol (**2p**), *α*-D-glucose derivative (**2q**), L-menthol (**2r**), and cholesterol (**2s**) could be introduced to the furan linker in

Scheme 3. Varying Alcohols in Formation of 2[a,b]

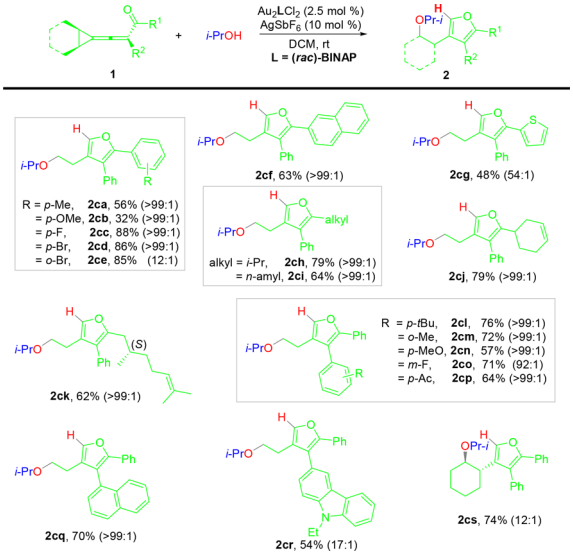


[a]The reaction was carried out using **1a** (0.2 mmol), alcohols (2 mmol), Au_2(*rac*-BINAP)Cl_2 (2.5 mol %), and $AgSbF_6$ (10 mol %) in 2 mL of DCM under N_2 atmosphere. [b]Isolated yield.

appropriate yields, rendering this late-stage modification method broadly useful. Finally, when *n*-octyl mercaptan was employed, the reaction afforded the Michael addition product in 98% yield (1.3:1 *E*/*Z* mixtures) instead of the desired furan.

Subsequently, we turned our attention to defining the capacity of different cyclopropyl-tethered allenyl ketones **1** with *i*PrOH (Scheme 4). With respect to the substituents at the R^1 position, diverse aryl, heteroaryl, and alkyl groups could be employed for this transformation, and the corresponding

Scheme 4. Varying Substrate 1 in Formation of 2[a,b]



[a]The reaction was carried out using **1** (0.2 mmol), *i*PrOH (2 mmol), Au_2(*rac*-BINAP)Cl_2 (2.5 mol %), and $AgSbF_6$ (10 mol %) in 2 mL of DCM under N_2 atmosphere. [b]Isolated yield.

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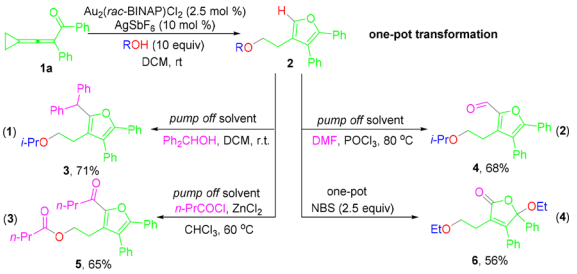
trisubstituted furans **2ca**–**ck** were achieved in modest to good yields (33–88%). The nature of the substituents had a demonstrable effect on this reaction. As for the electron-donating 4-methyl and 4-methoxy groups, **2ca** and **2cb** were afforded in modest yields. Moreover, the product **2ce** containing the sterically hindered 2-Br phenyl substituent was provided in 85% yield, however, with lower regioselectivity (rr = 12:1). The R^2 substituents with an array of substituted aromatic ring could also be successfully altered, leading to furans **2cl**–**cr** in moderate to high yields, and the electronic nature of the R^2 substituents had little effect on the reaction. Notably, cyclohexane-fused substrate could also be employed to reach the target furan scaffold (**2cs**) in high yield with excellent *trans*-stereoselectivity.

To demonstrate the synthetic value of the method, we investigated one-pot transformations using 2,3,4-trisubstituted furans **2** as the intermediates (Scheme 5). The cascade

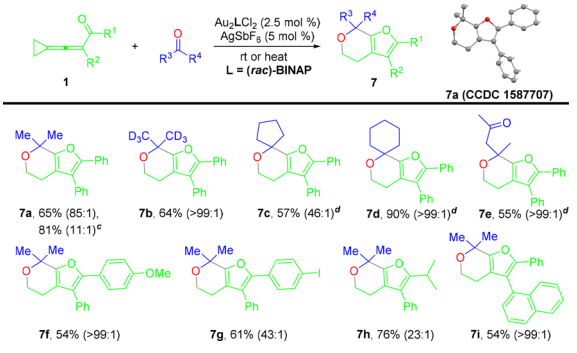
Scheme 5. Synthetic Application of Trisubstituted Furans



diarylmethylcarbocation electrophilic substitution, Vilsmeier–Haack formylation, and Friedel–Crafts acylation of **2** were achieved, respectively, affording tetrasubstituted furans **3**–**5** in 65–71% yields. Moreover, the highly functionalized isocrotonolactone **6** was obtained in 56% yield via NBS-mediated oxidative dearomatization of the furan skeleton.

With such a ring-opening reaction in hand, we then considered the possibility of extending the above protocol to synthesize cyclic tetrasubstituted furans **7** by trapping the migrated gold–carbene intermediates by ketones. After some trials, the product **7a** was obtained in 65% yield with high regioselectivity by using a mixture of Au_2(*rac*-BINAP)Cl_2 (2.5 mol %) and $AgSbF_6$ (5 mol %) as the catalyst, acetone as the reagent, and solvent (Scheme 6). The structure of **7a** was unambiguously confirmed by single-crystal X-ray diffraction

Scheme 6. Varying Substrate 1 in Formation of 2[a,b]

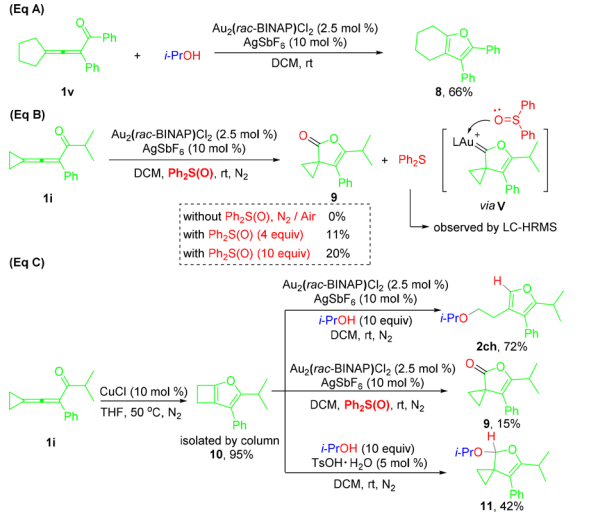


[a]The reaction was carried out using **1** (0.2 mmol), Au_2(*rac*-BINAP)Cl_2 (2.5 mol %), and $AgSbF_6$ (5 mol %) in ketones as solvent under N_2 atmosphere. [b]Isolated yield. [c]40 °C. [d]70 °C.

analysis. Thus, the scope of the reaction was briefly examined. The acetone-d_6 (**7b**), cyclopentanone (**7c**), cyclohexanone (**7d**), and acetylacetone (**7e**) could be tolerated to afford furans in moderate to high yield with excellent selectivity. Unfortunately, the reaction of isobutylaldehyde with **1a** turned to generate a complex mixture. With respect to cyclopropyl-tethered allenyl ketones **1**, typical substrates bearing substitutions at the 1- or 2-position were examined, which all delivered the products **7f**–**i** in acceptable yields.

Mechanistic studies were conducted to gain insight into the aforementioned transformations (Scheme 7). First, subjecting

Scheme 7. Mechanism Study



the cyclopentyl-substituted allenyl ketone **1v** with *i*PrOH to the reaction conditions led to the six-membered ring fused furan **8** instead of the desired ring-opening product (eq A). This observation rules out that the cyclopropyl moiety in substrate **1** plays a crucial role in the cascade cycloisomerization/carbene-transfer/ring-opening reaction. Subsequently, to determine whether this cascade reaction involves a 1,2-carbene transfer process, several parallel experiments were performed. For example, the reaction of substrate **1i** without addition of any trappers resulted in unknown polymers. To our delight, when the diphenyl sulfoxide,[18] a typical gold carbene scavenger, was added under the same conditions, the 2(3*H*)-furanone **9** and diphenyl sulfide were observed (eq B). This experiment clearly indicates the formation of gold–carbene intermediate **V** during the reaction. In addition, when the furan-fused cyclobutene **10**[17b] was subjected to reaction with *i*PrOH and diphenyl sulfoxide,the desired products **2ch** and **9**, respectively, were successfully afforded. These results indicated that furan-fused cyclobutene **10** is an important intermediate in the reactions. Moreover, the Brønsted acid showed different reactivities, and ring-contraction product **11** was obtained when TsOH (5 mol %) was employed instead of gold catalyst (eq C).

Based on the above experimental results and previous reports, a tentative mechanism is outlined in Scheme 8. First, the C–C bond of allene moiety is activated by a cationic gold(I) complex, which is then attacked by the carbonyl oxygen to form spirocyclic oxonium[19] ***Int*-A** or its resonance structure, cyclic gold carbene complex **IV** (note: oxidation compound of carbene intermediate **IV** was not observed yet in trapping experiments probably due to the relatively fast ring-expansion). Next, the competitive reaction occurs. The intermediate ***Int*-A**

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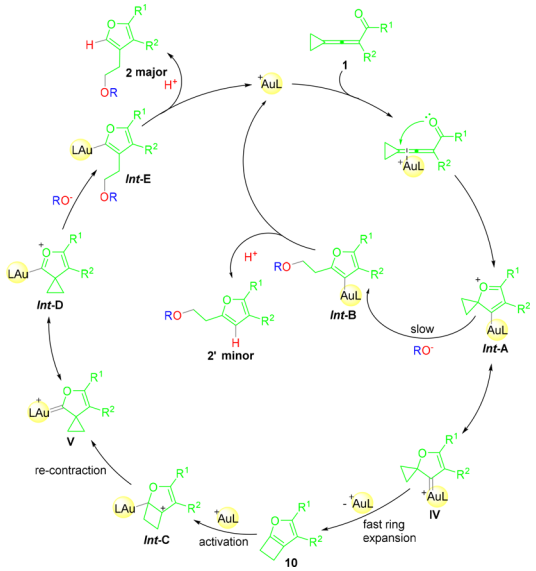
Scheme 8. Tentative Mechanism for the Selective Production of 2



could be directly transformed to 2,3,5-trisubstituted furans **2′** via the nucleophilic attack of alcohols and protonation reaction. On the other hand, the ring expansion of the cyclopropane ring of resonance form **IV** followed by elimination of the gold(I) catalyst provides the furan-fused cyclobutenes **10**. The cationic gold species may reactivate the relatively electron-rich C−C double bond of **10** to give ***Int*-C**, which may be converted to the key cyclic gold carbene intermediate **V** and its resonance structure ***Int*-D** by recontraction. Similarly, intermediate ***Int*-D** undergoes nucleophilic attack by alcohols and protonation to furnish 2,3,4-trisubstituted furan **2**. It should be noted that the reaction prefers to produce **2** instead of **2′** in our catalytic system, probably due to the relatively fast rate of ring expansion of species **IV** compared with nucleophilic attack by alcohols.

In summary, we have developed the first 1,2-gold carbene-transfer process via cascade C−C bond cleavage. This protocol provides a mild, general, and regioselective means to access a broad range of tri- or tetrasubstituted furan derivatives from allenyl ketones bearing a cyclopropyl moiety with various alcohols or ketones. The detailed mechanism as well as selection of other nucleophilic trappers is currently under investigation in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01152.

Experimental procedures and ^{1}H and ^{13}C NMR spectra for all new compounds (PDF)

Accession Codes

CCDC 1587707 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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