Gold-catalyzed tandem cyclization/Friedel–Crafts type reactions toward furan derivatives†

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A simple and convenient synthetic approach to furan derivatives 4 has been developed *via* gold-catalyzed tandem cyclization/Friedel–Crafts type reactions.

Furan rings occur widely as key structural subunits in numerous natural products, which exhibit interesting biological activities and also in substances of relevance for industry.[1] For this reason, the field of furan synthesis is continuously and rapidly developing.[2] Among many different approaches to multiply substituted furans,[2b,3,4] cycloisomerizations of alkynyloxiranes catalyzed by transition metals are particularly attractive. In general, traditional methods for furan synthesis from alkynyloxiranes mostly using Hg(II),[5] Mo,[6] Ru[7] and SmI_2/Pd(0/II)[8] compounds as transition-metal catalysts have some limitations. Thus, a new method for the synthesis of furans is needed.

Recently, it was reported that carbon-carbon and carbon-heteroatom bonds can be formed under mild conditions using gold catalysts.[9] Their ability to activate alkynes and promote the addition of nucleophiles has spurred growing investigations for the discovery of selective reactions.[10]

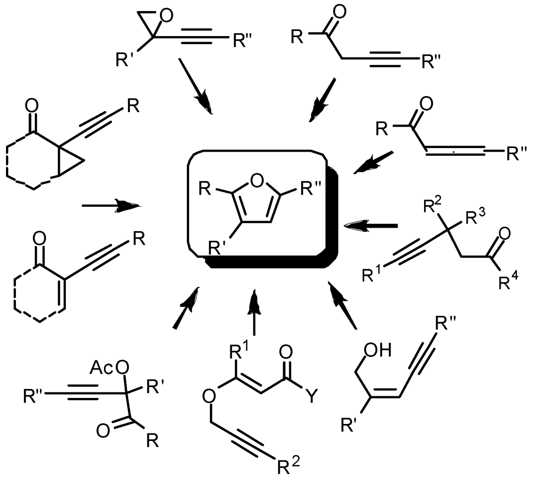
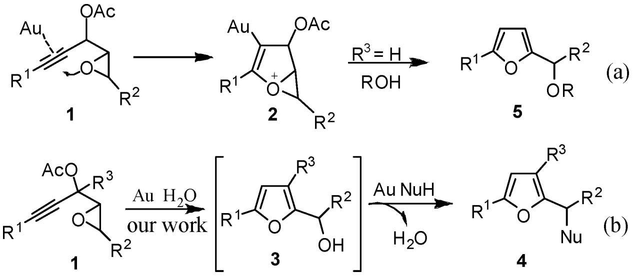


Fig. 1 Representative gold-catalyzed cycloisomerizations leading to furans.



Scheme 1 Design of a gold-catalyzed formation of furan derivatives through tandem cyclization/Friedel–Crafts type reactions.

Fig. 1 schematically illustrates representative examples of gold-catalyzed cycloisomerizations of oxoalkynyl compounds: alkynyloxiranes,[10a] alk-3-yn-1-ones,[10b] alka-2,3-dien-1-ones,[10c] alk-4-yn-1-ones,[10d] (*Z*)-alk-2-en-4-yn-1-ols,[10e,f] propargyl vinyl ethers,[10g] alk-3-yn-1-one-2-yl acetates[10h,i,j] and alkynyl enones[10k,l,m,n] that lead to substituted furans. However, most of them are intramolecular cycloisomerization reactions, examples of the corresponding highly selective intermolecular reactions by adding nucleophiles for furan synthesis are limited in gold chemistry.[10k,l,m,n] Recently, we have described the gold-catalyzed tandem cyclization of 1-oxiranyl-2-alkynyl esters **1** with nucleophiles to give 2,5-disubstituted furans **5** (Scheme 1(a)).[11] It was found that the oxonium ion **2** was an important intermediate. Thus, we envision that if water was exited in the reaction system, water may play an important role in the cyclization to give the 2-(α-hydroxyalkyl)-furans **3**. Then, under gold catalysis, the latter may afford furan derivatives **4** through an intermolecular Friedel–Crafts type reaction,[9g,12] which is an efficient approach to the formation of a carbon-carbon or carbon-heteroatom bond. (Scheme 1(b)).

Herein, we wish to report the gold-catalyzed tandem cyclization/Friedel–Crafts type reactions toward furan derivatives under very mild conditions. During this reaction, water as a nucleophile was the key step.

Initially, we started by treating 1-oxiranyl-2-alkynyl ester **1a** (0.5 mmol) and furan (5 equivs) with 2 mol% of $HAuCl_4{\cdot}4H_2O$ in wet 1,4-dioxane (2 mL) and, gratifyingly, the desired product **4a** was formed in 50% yield after 20 min, along with difurylmethane **6a** (5% yield; Table 1, entry 1). To our delight, on increasing the amount of furan to 10 equivs, a good yield (71%) of **4a** was obtained and no difurylmethane **6a** could be isolated (Table 1, entry 3). With other gold catalysts, such as $NaAuCl_4{\cdot}2H_2O$, $AuCl_3$, AuCl and Au(I) systems generated from silver salts, no superior results were obtained (Table 1, entries 4–7). $AgBF_4$ and $PtCl_2$ gave

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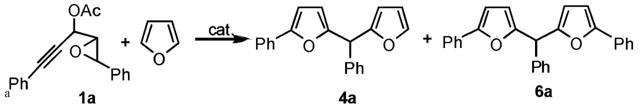
Table 1 Optimization of reaction conditions for the synthesis of **4a**



Entry	Catalyst	Furan (equivs)	Time (min)	Yield(%)[b] 6a	4a
1	$HAuCl_4{\cdot}4H_2O$	5	20	5	50
2	$HAuCl_4{\cdot}4H_2O$	8	20	<5	64
3	$HAuCl_4{\cdot}4H_2O$	10	20	0	71
4	$NaAuCl_4{\cdot}2H_2O$	10	30	<5	68
5	$AuCl_3$	10	25	0	70
6	AuCl	10	30	trace	71
7	$Au(PPh_3)Cl/AgBF_4$	10	30	0	35
8[c]	$AgBF_4$	10	120	0	0
9[d]	$PtCl_2$	10	120	0	0

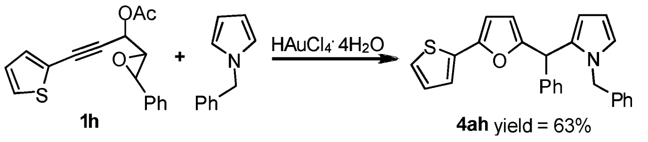
[a] Conditions: 0.5 mmol of **1a** and 10 equivs of furan with 2 mol% of catalyst in wet 1,4-dioxane (2.0 mL) at room temperature (23–25 °C). [b] Isolated yield. [c] 91% of **1a** was recovered. [d] 93% of **1a** was recovered.

no reaction. Thus, the use of 10 equivs of furan and $HAuCl_4{\cdot}4H_2O$ (2 mol%) in wet 1,4-dioxane at room temperature was found to be the most efficient and was utilized as the standard conditions.

To study the scope of this formation of furan derivatives, various 1-oxiranyl-2-alkynyl esters[13] were then submitted to the above conditions, as depicted in Table 2. Thus, a tandem cyclization/Friedel–Crafts type reactions of 1-oxiranyl-2-alkynyl esters **1a-1l** proceeded smoothly to provide the corresponding furan derivatives in moderate to good yields. The reaction works well with aromatic R^2 groups (entries 1–3). While substrates with aliphatic or H R^2 group like **1d** and **1e** gave the corresponding products in lower yields (entries 4 and 5). Various aryl substituents on the alkyne moiety, were compatible with this reaction and generally gave good yields of the corresponding furan derivatives (entries 6–8). With aliphatic substituents on the alkyne moiety like **1i**, only a moderate yield (41%) of **4i** was obtained (entry 9). Substrates with an aliphatic R^3 group like **1j** gave the corresponding products in good yield (entry 10). And we have also investigated the reactions of 1-oxiranyl-2-alkyn-1-ols with different protective groups. While substrates like **1k** and **1l** gave the furan derivatives **4a** in good yields.

Furthermore, we also investigated the scope of nucleophiles in this cyclization/Friedel–Crafts type reaction, as depicted in Table 3. It was found that, in addition to furan, 2-methylfuran, pyrrole derivatives, 1*H*-indole, pentane-2,4-dione and NaN_3 could be used as effective nucleophiles for this reaction (up to 81%) (entries 1–7).

It is very interesting that when substrate **1h** and a pyrrole derivative were then submitted to the above conditions, the corresponding product **4ah**, which contains O, S and N heteroatoms at the same time, was obtained in 63% yield (Scheme 2).

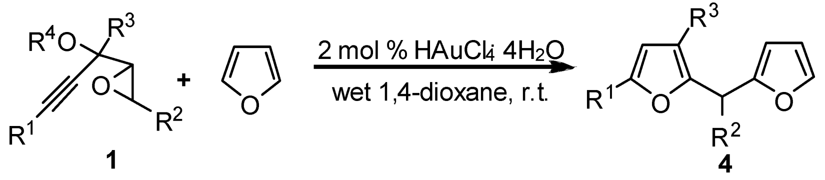


Scheme 2

In order to uncover the mechanism for the reaction, we started out our investigation by using 2-(α-hydroxyalkyl)furan **3a** and furan (5 equivs) under 2 mol% of $HAuCl_4{\cdot}4H_2O$ and the desired product **4a** was formed in excellent yield. From the above, it was found that it was a Friedel–Crafts type reaction (Scheme 3).

On the basis of the above observations, a possible reaction mechanism is proposed as shown in Scheme 4, which may involve the following steps. (i) coordination of the alkynyl moiety of **A** to the Au catalyst gives the complex **B**. (ii) The subsequent domino nucleophilic attack/anti-endo-dig cyclization affords organogold intermediate **E** through *path a* during which trace amounts of

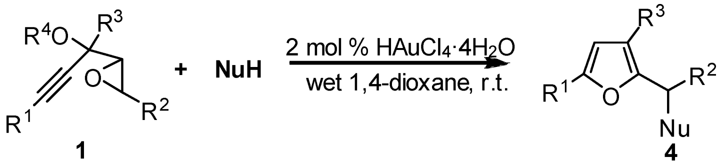
Table 2 Gold(III)-catalyzed reaction of 1-oxiranyl-2-alkynyl ester **1** with furan [a]

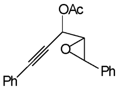
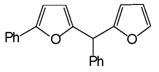
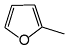
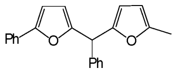
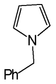
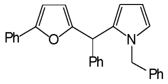
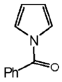
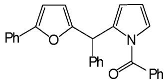
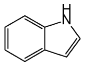
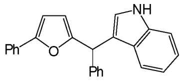
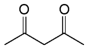
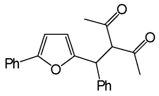
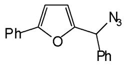
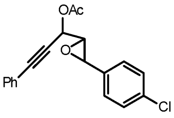
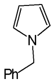
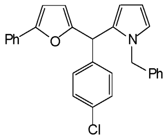


Entry	1[b]	R^1	R^2	R^3	R^4	Time (min)	4	Yields (%)[c]
1	**1a** (7:5)	Ph	Ph	H	Ac	20	**4a**	71
2	**1b** (10:7)	Ph	p-CH_3-C_6H_4	H	Ac	10	**4b**	73
3	**1c** (2:1)	Ph	p-Cl-C_6H_4	H	Ac	10	**4c**	70
4	**1d** (2:1)	Ph	CH_3	H	Ac	20	**4d**	50[d]
5	**1e** (9:7)	Ph	H	H	Ac	20	**4e**	43[d]
6	**1f** (16:7)	p-CH_3-C_6H_4	Ph	H	Ac	10	**4f**	68
7	**1g** (2:1)	p-CH_3CO-C_6H_4	Ph	H	Ac	50	**4g**	58
8	**1h** (2:1)	2-thienyl	Ph	H	Ac	10	**4h**	60
9	**1i** (5:3)	C_5H_{11}	Ph	H	Ac	60	**4i**	41
10	**1j** (1:0)	Ph	Ph	CH_3	Ac	10	**4j**	72
11	**1k** (2:1)	Ph	Ph	H	$COOCH_3$	20	**4a**	68
12	**1l** (1:1)	Ph	Ph	H	$COOC(CH_3)_3$	10	**4a**	70

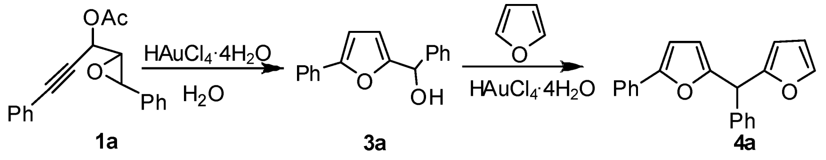
[a] Conditions: 0.5 mmol of **1** and 10 equivs of furan with 2 mol% of catalysts in wet 1,4-dioxane (2.0 mL) at room temperature (23–25 °C). [b] *Syn/anti* mixtures of the substrate were used; *syn:anti* ratio determined by ^1H NMR. [c] Isolated yield. [d] Reaction run with furan (15 equivs) and 5 mol% catalyst loading.

Table 3 Gold(III)-catalyzed reaction of 1-oxiranyl-2-alkynyl ester **1** with nucleophiles[a]



Entry	Substrates	Nucleophiles	Products	Time	Yields (%)[b]	
1	 **1a**			20 min	**4a**	71
2				15 min 10 h	**4aa**	73 77[c]
3				30 min	**4ab**	81[d]
4				60 min	**4ac**	57[e]
5				10 h	**4ad**	51[f]
6				8 h	**4ae**	38[g]
7		NaN_3		8 h	**4af**	46[h]
8	 **1c**			20 min	**4ag**	79

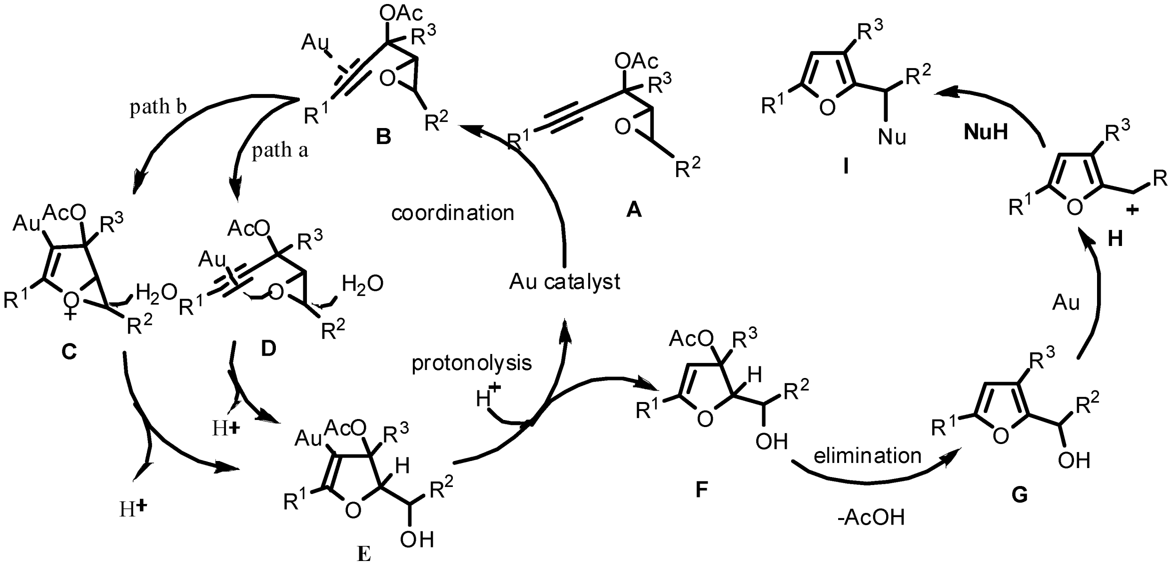
[a] Conditions: 0.5 mmol of **1a**, nucleophile (10 equivs) with 2 mol% of catalyst in wet 1,4-dioxane (2.0 mL) at room temperature (23–25 °C). [b] Isolated yield. [c] 0.5 mmol of **1a**, 1.0 mmol of 2-methylfuran with 5 mol% of catalysts in water (1.0 mL) at 80 °C for 10 h. [d] 7.5 equivs of 1-benzyl-1*H*-pyrrole was recovered. [e] 7.0 equivs of phenyl(1*H*-pyrrol-1-yl)methanone was recovered. [f] 0.5 mmol of **1a**, 0.6 mmol of 1*H*-indole with 5 mol% of catalysts in water (0.8 mL) at 80 °C for 10 h. [g] 0.5 mmol of **1a**, 1.5 mmol of pentane-2,4-dione with 5 mol% of catalysts in water (0.8 mL) at 80 °C for 8 h and 38% of desired product **4ae** was obtained, along with 20% **6a** was isolated. [h] 0.5 mmol of **1a**, 1.0 mmol of NaN_3 with 5 mol% of catalysts in water (0.8 mL) at 80 °C for 8 h.



Scheme 3

water act as the nucleophile. Alternatively, the complex **B** may form an oxonium ion **C**[14] through *path b* which is formed by the nucleophilic attack of epoxide oxygen to the gold-coordinated alkynes,[11a,c,15] then trace amounts of water as the nucleophile attack oxonium ion **C** and afford organogold intermediate **E** (iii) Protonation of **E** yields 2,3-dihydrofuran **F** and regenerates the catalyst Au. (iv) **F** undergoes direct reductive elimination of acetic acid to afford the 2-(α-hydroxyalkyl)furans **G**. (v) Under the gold catalysis, **G** eliminates one molecule water to afford ion intermediate **H**. (vi) Then nucleophilic attack on ion intermediate **H** affords the furan derivatives **I**, which is an intermolecular Friedel–Crafts-type reaction.

In summary, a novel and flexible method for the synthesis of furan derivatives has been developed through the cyclization/Friedel–Crafts type reactions of esters of 1-oxiranyl-2-alkyn-1-ols at room temperature under gold catalysis. During



Scheme 4 Proposed reaction mechanism for Au-catalyzed cyclization/Friedel–Crafts type reactions.

this reaction, water as a nucleophile was the key step. Further studies, including detailed investigations into the mechanism and the scope of this reaction, are in progress in our laboratory.

Acknowledgements

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