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Gold-Catalyzed Photoredox C(sp^2) Cyclization: Formal Synthesis of (±)-Triptolide

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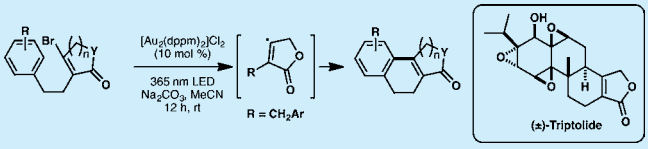
Supporting Information

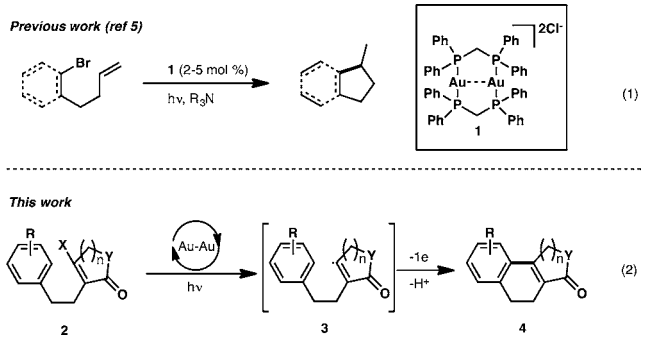
ABSTRACT: Photoexcitation of a dimeric gold complex showed the activation of a C(sp^2)−Br bond to generate a vinyl radical in a mild photoredox catalysis process. Use of $[Au_2(dppm)_2]Cl_2$ with 365 nm LEDs in a photoredox catalysis process to produce polycyclic scaffolds using vinyl radicals is reported. This method achieved the synthesis of a small library of butenolide polycyclic compounds and naphthol polycyclic compounds. The efficacy of this photoredox process was further demonstrated by accomplishing the concise formal synthesis of (±)-triptolide.



For over a century, radicals have been interesting to chemists, becoming a powerful tool for building C−C bonds.[1] Radical C−C bond construction has been very useful for complex molecule synthesis, such as for natural products.[2] Recently, photoredox catalysis has emerged as an efficient method for the generation of carbon-centered radicals through single-electron transfer (SET) processes. These light-mediated methods have shown high-yielding protocols for the generation of C−C, C−N, and C−O bonds, offering an attractive alternative to the use of potentially hazardous radical initiators (AIBN, Et_3B/O_2, peroxides) and hydrogen atom donors such as organostannane.[3]

In this context, we[4] and others[5] recently reported the use of the dimeric gold complex $[Au_2(dppm)_2]Cl_2$ **1**[6] in combination with ultraviolet A (UVA, 365 nm) light irradiation to generate radical intermediates from unactivated bromoalkanes/arenes in a mild photoredox catalysis process (Scheme 1, eq 1). Further mechanistic investigation showed that this photoredox process proceeds through an inner-sphere mechanism via either an oxidative or a reductive quenching cycle.[7] Taking advantage of this distinctive mechanism, we wondered if the generation of β-enone radicals such as **3** could be achieved through a photoredox process (Scheme 1, eq 2). Although these radical intermediates can be more reactive than alkyl radicals, one can imagine that their inherent reactivity could be harnessed for the formation of new C(sp^2)−C(sp^2) bonds. In particular, one can envisage functionalization of γ-butenolides (Y = O, n = 1). These structural motifs and their saturated derivatives are frequently encountered in natural and synthetic product molecules, and these compounds are useful building blocks.[8]

Scheme 1. Photoredox Reaction Using a Dimeric Gold Complex



Despite the rich literature in radical chemistry, these types of carbon-centered radicals have been occasionally used, and the potential broad utility of such photoredox processes in organic synthesis warrants further investigation.[9] Herein, we describe the development of a photoredox direct arylation of substituted butenolides and cyclic enones using dimeric gold complexes and its application to a short formal synthesis of (±)-triptolide.

Our investigation began with the irradiation of 3-bromobutenolide **2a** (UVA LED, 365 nm) in the presence of **1** (10 mol %) and various bases in acetonitrile (Table 1). In the presence of tertiary amine bases, the photoredox process proceeds through a reductive quenching cycle in which the amine bases serve as a sacrificial electron donor (Table 1, entries 1−4). Use of DIPEA led to an inseparable mixture of the desired compound **4a** and the dehalogenated **9** in 50% yield as a 6:4 ratio (Table 1, entry 1). Given that these radical intermediates are a more reactive species than alkyl radicals, hindered or poor hydrogen donor alkyl amine bases were tested to decrease the competitive reduction process. No significant improvements were noticed when using pempidine and DABCO (Table 1, entries 2 and 3). However, a higher yield and ratio were observed with DBU (Table 1, entry 4). Switching to sodium carbonate, we obtained the exclusive formation of **4a** in 39% yield (Table 1, entry 5). Prolonged irradiation substantially increased the reaction yield to 72% (Table 1, entry 6). Control experiments demonstrated the need

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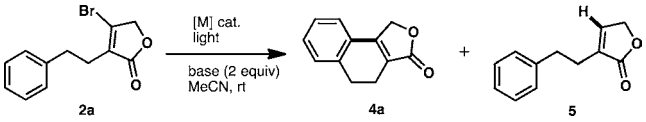
Table 1. Optimization of Reaction Conditions and Control Experiments[a]



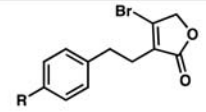
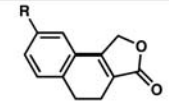
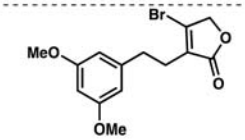
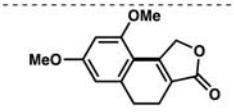
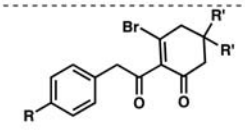
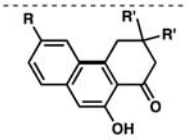
entry	photocatalyst	base	light[b]	time (h)	yield (%)[c]	4a/5[d]
1	$[Au_2(dppm)_2]Cl_2$	DIPEA	365 nm	3	50	6:4
2	$[Au_2(dppm)_2]Cl_2$	PMP	365 nm	3	58	3:1
3	$[Au_2(dppm)_2]Cl_2$	DABCO	365 nm	3	34	9:1
4	$[Au_2(dppm)_2]Cl_2$	DBU	365 nm	3	50	9:1
5	$[Au_2(dppm)_2]Cl_2$	Na_2CO_3	365 nm	3	39	1:0
6	**$[Au_2(dppm)_2]Cl_2$**	**Na_2CO_3**	**365 nm**	**12**	**72**	**1:0**
7	-	Na_2CO_3	365 nm	12	n.r.	-
8	$[Au_2(dppm)_2]Cl_2$	-	365 nm	12	n.r.	-
9	$[Au_2(dppm)_2]Cl_2$	Na_2CO_3	-	12	n.r.	-
10	$[Ru(bpy)_3](PF_6)_2$	DIPEA	CFL	24	n.r.	-
11	$[Ru(bpy)_3](PF_6)_2$	DIPEA	410 nm	24	14	3:1
12	$[Ru(bpy)_3](PF_6)_2$	Na_2CO_3	CFL	24	n.r.	-
13	$[Ru(bpy)_3](PF_6)_2$	Na_2CO_3	410 nm	24	n.r.	-
14	$Ir(ppy)_3$	DIPEA	CFL	24	n.r.	-
15	$Ir(ppy)_3$	DIPEA	410 nm	24	7	7:3
16	$Ir(ppy)_3$	Na_2CO_3	CFL	24	n.r.	-
17	$Ir(ppy)_3$	Na_2CO_3	410 nm	24	18	1:0

[a]Unless otherwise noted, reactions were conducted with **2a** (0.1 M in MeCN), catalyst (10 mol %), base (2 equiv), and light at rt. [b]365 nm LED or 410 nm LED or CFL bulb (14 W). [c]Yields of isolated products. [d]Ratio determined by ^1H NMR.

of each reagent and light (Table 1, entries 7−9). In the absence of catalyst, base, or light, only starting material was recovered. Common photocatalysts such as $Ru(bpy)_3(PF_6)_2$ and $Ir(ppy)_3$ proved to be ineffective for this radical cyclization. Only starting material was recovered after irradiation with a 14 W compact fluorescent light (24 h) (Table 1, entries 10, 12, 14, and 16). Irradiating the reaction mixture with a 410 nm LED in the presence of DIPEA afforded a mixture of **4a** and **5** in very low yields (Table 1, entries 11 and 15). Combining sodium carbonate with $Ir(ppy)_3$ produced **4a** as the only product in 18% yield after 24 h (Table 1, entry 17).

With optimized conditions in hand, the scope of the reaction was explored using various substituents on the aromatic ring (Table 2). Methyl and phenyl groups afforded the corresponding cyclized products **4b** and **4c** with 70 and 77% yields, respectively (Table 2, entries 2 and 3). The presence of an electron-withdrawing group such as chloride **2d** had no effect on the cyclization, and product **4d** was obtained in 79% yield (Table 2, entry 4). However, a decrease in yield was observed when using electron-rich arene **2e**, and cyclic product **4e** was isolated in 46% yield (Table 2, entry 5). We then applied this photoredox process to a range of 3-bromocyclohexenones. Cyclization of **2f** and subsequent tautomerization afforded naphthol **4f** with 64% yield

Table 2. Scope of the Reaction[a]

entry	substrate, 2	product, 4	yield (%)[b]
			
1	**2a**, R = H	**4a**	72
2	**2b**, R = Me	**4b**	70
3	**2c**, R = Ph	**4c**	77
4	**2d**, R = Cl	**4d**	79
			
5	**2e**	**4e**	46
			
6	**2f**, R = H, R'= H	**4f**	64
7	**2g**, R = Me, R'= H	**4g**	62
8	**2h**, R = Ph, R'= H	**4h**	58
9	**2i**, R = H, R'= Me	**4i**	67
10	**2j**, R = Cl, R'= Me	**4j**	69

[a]Reaction conditions: vinyl bromide (0.1 M in MeCN), **1** (10 mol %), Na_2CO_3 (2 equiv), 365 nm LED, 12 h, rt. [b]Yields of isolated products.

(Table 2, entry 6). Methyl and phenyl groups present on substrates **2g** and **2h** gave the corresponding cyclized naphthols **4g** and **4h** with 62 and 58% yields (Table 2, entries 7 and 8). 3-Bromocyclohexenone with a *gem*-dimethyl group at position 5 (**2i**) was cyclized to obtain the corresponding product **4i** with 67% yield (Table 2, entry 9). Presence of a chloride in the *para* position of the aromatic ring and the *gem*-dimethyl group on the cyclohexenone **2j** gave cyclized product **4j** with 69% yield (Table 2, entry 10).

Use of sodium carbonate to achieve this kind of photoredox catalysis process is not frequently encountered. Indeed, most reported studies use tertiary amine bases as sacrificial electron donors and hydrogen sources. According to the value of the redox potential of the $CO_3^{\bullet-}/CO_3^{2-}$ couple (1.5 V),[10] the carbonate species cannot turnover the catalyst in either reductive or oxidative quench pathways. For this reason, we suggest the mechanism shown in Figure 1.

Irradiation of $[Au-Au]^{2+}$ generates excited $[Au-Au]^{2+*}$, which could then reduce the C−Br bond through a SET to afford the vinyl radical **3** (oxidative quenching cycle). This radical can then cyclize to produce the tricyclic compound **5** bearing a tertiary radical. Simultaneous oxidation of **5** and reduction of the $[Au-Au]^{3+}$ complex should provide **4** and the dimeric gold photocatalyst **1**.

The utility of this photoredox process was demonstrated in a concise synthesis of (±)-triptolide **6** (Scheme 2). Isolated from

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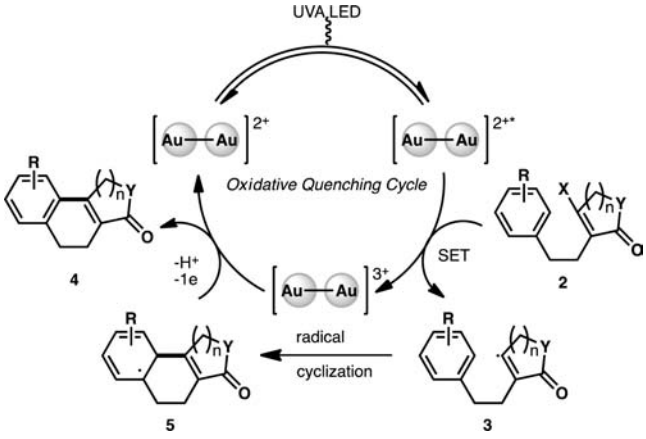
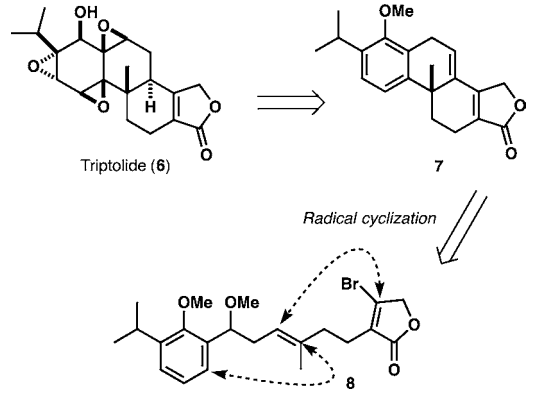


Figure 1. Proposed mechanism.

Scheme 2. Radical Cascade Cyclization as Key Step



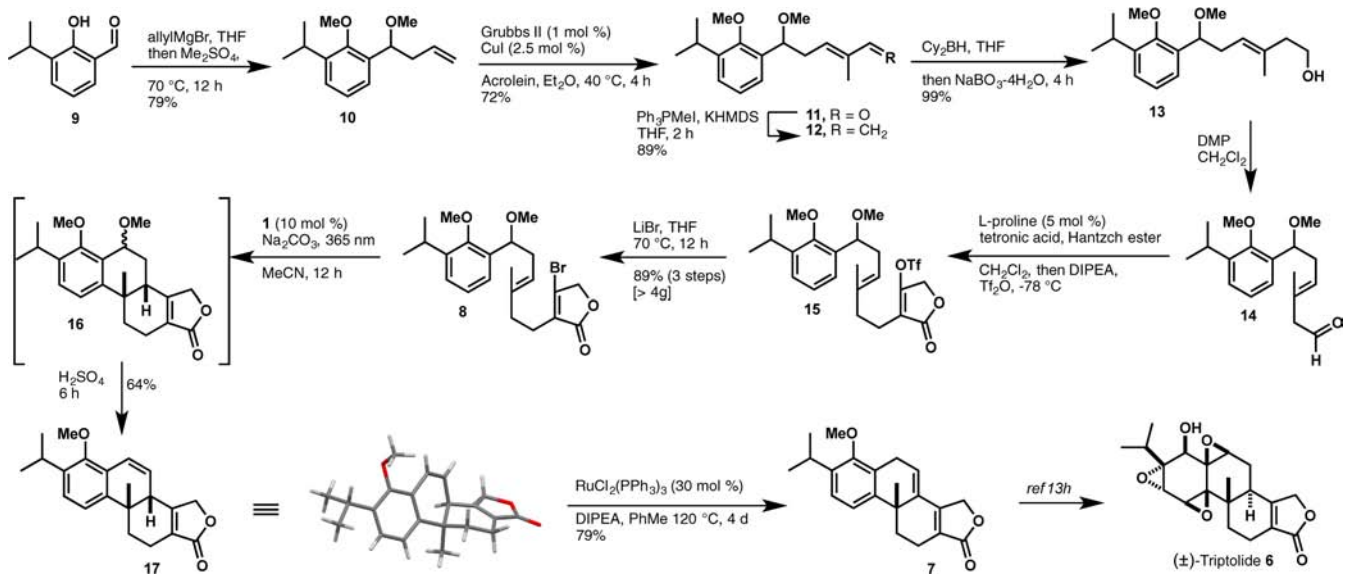
the chinese climbing vine *Tripterygium wilfordii*, **6** is a naturally occurring diterpene containing three contiguous epoxides and a butenolide ring.[11] This diterpene shows in vitro and in vivo activity against polycystic kidney disease and pancreatic tumor cells[12a−c] and also has immunosuppressive activities.[12d] Promising therapeutic properties and challenging structure of triptolide **6** have resulted in considerable attention from the synthetic community and led to clever syntheses.[13] We envisioned the application of the gold-catalyzed photoredox reaction to assemble **7**, a known intermediate, via an intramolecular radical cascade cyclization of the γ-butenolide derivative **8**.

Allyl **10** was obtained in 79% yield by treatment of aldehyde **9** with allylmagnesium bromide followed by addition of dimethyl sulfate (Scheme 3). Compound **10** was engaged in a cross-metathesis reaction using methacrolein in the presence of Grubbs second generation catalyst and a catalytic amount of copper iodide[14] to give the aldehyde **11** in 72% yield as a single *E*-isomer.[15] A carbon homologation using the Wittig reaction transformed **11** into diene **12** in 89% yield. The latter was quantitatively converted to the corresponding primary alcohol **13** through a hydroboration/oxidation sequence.[16] Dess−Martin oxidation of **13** led to aldehyde **14**, which underwent a one-pot proline-catalyzed coupling reaction with tetronic acid in the presence of Hantzsch ester[17] and triflation to afford triflate **15**. The crude material was directly heated in THF with LiBr to furnish bromobutenolide **8** in 89% yield over three steps. All of these steps were performed on a multigram scale.

Reaction of bromobutenolide **8** under our optimal conditions was followed by treatment with H_2SO_4 to provide tetracycle **17**. The relative stereochemistry of **17** was unequivocally established by X-ray analysis.[18] Radical cyclization provided **16** (1:1 dr) with a *cis* ring junction rather than a *trans* junction.[19] Epimerization at C4 aimed at obtaining the *trans* ring junction under basic or acidic conditions proved to be fruitless. Isomerization of **15** using a catalytic amount of $RuCl_2(PPh_3)_3$ and DIPEA in toluene at 120 °C provided **7** in 79% yield. Conversion of **7** into **6** was achieved in eight steps.[13d] Interception of this late-stage intermediate in their synthesis thus completes the formal total synthesis of the natural product.

In summary, a photoredox process using **1** (10 mol %) and a 365 nm LED to generate vinyl radicals from bromobutenolide and bromocyclohexenone was developed. This redox-neutral method allows the formation of polycyclic compounds. This effective reaction was demonstrated by a concise formal synthesis of (±)-triptolide **6** in nine steps (17% overall yield) from readily available **9**. Further application of this gold-catalyzed photoredox

Scheme 3. Formal Synthesis of (±)-Triptolide 6



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process, including the syntheses of other diterpenes, will be reported in due course.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b00968.

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

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Notes

The authors declare no competing financial interest.

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(18) CCDC 1471725 (**16**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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