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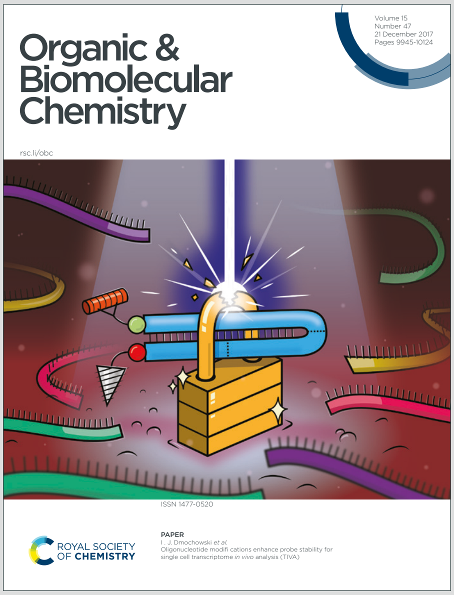

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ARTICLE

Synthesis of polymethylene-linked bis(cyclobutane-fused chromanones) mediated by gold-photocatalysis

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Junying Wang,[a] Vladislav A. Voloshkin,[a] Sayan Dutta,[b] Yuan Zhao,[a] Tommaso Ruggiero,[a] Kristof Van Hecke,[a] Bholanath Maity,[b] Luigi Cavallo,[b] and Steven P. Nolan*

We report a gold-mediated energy transfer strategy for the sequential double [2+2] photocycloaddition of coumarins with dienes, providing access to polymethylene-linked bis(cyclobutane-fused chromanones). The reaction proceeds under mild conditions and affords structurally complex products in moderate to good yields. Optimization studies, control experiments, triplet-energy analysis, and DFT calculations support a triplet energy transfer pathway involving diradical intermediates.

In the past few decades, the field of photocatalysis has made significant progress and has become an important tool in organic synthesis.[1–4] Although photoredox catalysis has received widespread attention,[5–8] electron transfer (ET) mechanisms still dominate and research on energy transfer (EnT) catalysis remains insufficient. In recent years, considerable effort has been devoted to the development of high-triplet-energy photocatalysts capable of transferring photoenergy to organic substrates under mild and sustainable conditions through EnT processes.[9–12] Photocatalytic reactions utilizing the triplet-triplet energy transfer (TTEnT) mechanism have emerged as a powerful strategy for the construction of complex molecular scaffolds, particularly in the synthesis of functionalized substrates.[13–15] In this context, gold photocatalysis has attracted increasing attention because of the unique photophysical properties of Au complexes and their potential applications in photoredox and energy-transfer transformations.[16]

Tetrahydro-cyclobuta[c]chromanones are important structural motifs that have garnered widespread attention in the fields of natural products, bioactive and pharmaceutical compounds. For instance, Melicodenine G is a compound with anti-proliferative activity, particularly demonstrating significant inhibitory effects on DLD-1 human colon cancer cells (Figure 1).[17] Lindleyanin is a novel cyclobutane-type lignan isolated from pleurospermum lindleyanum, a plant used in traditional medicine to treat hypertension and coronary heart disease.[18] In addition, a variety of compounds containing the tetrahydro-cyclobuta[c]chromanone scaffold exhibit important pharmacological effects, such as cyclohoehneliacoumarin[19] and monoterpene coumarin.[20] The long-chain, methyl-branched scaffold is also a prevalent motif in natural products[21–23] and can serve as a frequent precursor in drug development.[24,25] Existing methods for synthesizing the cyclobuta[c]chromanone core include direct excitation with high-pressure mercury lamps,[26–28] Lewis acid catalysis,[29] and photosensitizer-mediated intermolecular cycloaddition reactions.[30–32] However, to the best of our knowledge, current strategies for the synthesis of polymethylene-linked bis(cyclobutane-fused chromanones) remain limited.

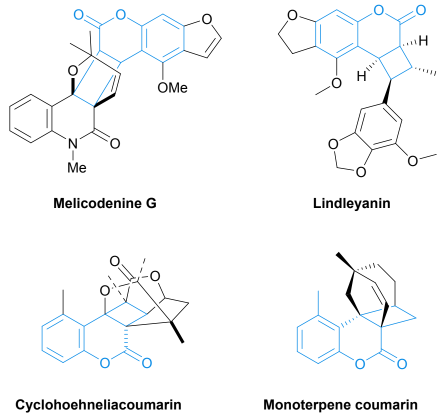

Figure 1. Representative examples of compounds containing cyclobuta[c]chromanone scaffolds.

In 2021, our group developed high-triplet-energy gold photocatalysts that enable efficient [2+2] photocycloadditions, even surpassing iridium systems.[33–38] Subsequently, we reported a gold-mediated synthesis of cyclobuta[c]chromanone from coumarins (Scheme 1a).[39] In contrast to monofunctional alkenes, dienes enable two consecutive intermolecular [2+2] photocycloaddition events, providing direct access to linked bis(cyclobutane-fused chromanone) architectures. This transformation raises additional mechanistic questions, including the competition between productive diene addition and coumarin homodimerization, the fate of the initially formed dimeric species, and the origin of the observed bis-cycloaddition selectivity. Herein, we report a gold-mediated sequential double [2+2] photocycloaddition of coumarins with

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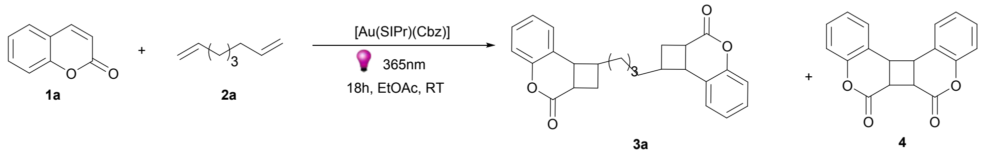
[a] Department of Chemistry, Centre for Sustainable Chemistry, Ghent University, Krijgslaan 289, S3, 9000 Ghent, Belgium. E-mail: steven.nolan@ugent.be
[b] Physical Science and Engineering Division, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia.

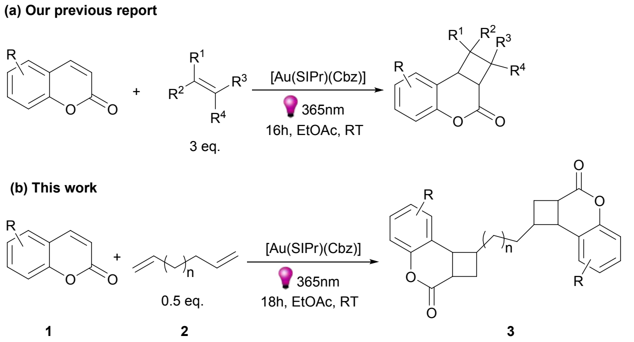
dienes for the synthesis of polymethylene-linked bis(cyclobutane-fused chromanones) (Scheme 1b).

Table 1. Optimization of reaction conditions.[a]



[a]Unless otherwise noted, the reaction was conducted with **1a** (0.20 mmol), **2a** and [Au(SIPr)(Cbz)] in EtOAc for 18 h under 365 nm lamp (18 W). [b]NMR yield. [c]Under 365 nm Lamps (30 W+18 W). [d]reaction time 24 h. [e]THF instead of EtOAc. [f]MeCN instead of EtOAc.

Entry	Catalyst (mol%)	Diene (eq.)	Conc. (M)	Yield[b] (**3a**, %)	Yield[b] (**4,** %)
1	[Au(SIPr)(Cbz)] (1)	0.5	0.1	74	16
2	[Au(SIPr)(Cbz)] (0.5)	0.5	0.1	55	39
3	[Au(SIPr)(Cbz)] (1.5)	0.5	0.1	20	71
4	[Au(SIPr)(Cbz)] (2)	0.5	0.1	19	71
5	[Au(SIPr)(Cbz)] (1)	0.5	0.2	61	23
6	[Au(SIPr)(Cbz)] (1)	0.5	0.3	39	46
7	[Au(SIPr)(Cbz)] (1)	0.5	0.05	48	38
8	[Au(SIPr)(Cbz)] (1)	0.6	0.1	72	<10
9[c]	[Au(SIPr)(Cbz)] (**1**)	**0.6**	**0.1**	**80**	**-**
10[c]	[Au(SIPr)(Cbz)] (1)	0.5	0.1	76	11
11[c, d]	[Au(SIPr)(Cbz)] (1)	0.6	0.1	78	-
12[c, e]	[Au(SIPr)(Cbz)] (1)	0.6	0.1	65	<10
13[c, f]	[Au(SIPr)(Cbz)] (1)	0.6	0.1	13	49
14[c]	[Au(IPr)(Cbz)] (1)	0.6	0.1	77	-
15[c]	[Ir(dF(CF$_3$)ppy)$_2$(dtppy)]PF$_6$(1)	0.6	0.1	20	80
16[c]	Thioxanthone(1)	0.6	0.1	15	75
17[c]	Benzophenone(1)	0.6	0.1	15	75



Scheme 1. Previous report and this work.

Based on conditions reported in our previous work,[39] we initiated our investigations with the model coumarin (**1a**), 0.5 equivalents 1,7-octadiene (**2a**) with 1 mol% of [Au(SIPr)(Cbz)] under 365 nm irradiation in EtOAc. To our delight, after 18 hours of irradiation 74% NMR yield of the desired compound **3a** was observed, and the coumarin dimer **4** was formed in 16% yield (Table 1, entry 1). We have previously shown that coumarin dimer formation occurs fast under these conditions, but then it is converted into product of [2+2]-cycloaddition. Encouraged by this result, we further optimized the reaction conditions. To maximize the yield of **3a** and minimize the amount of remaining coumarin dimer, the catalyst loading was first screened. However, both increasing and decreasing the catalyst loading led to a noticeable reduction in the yield of the desired product. (Table 1, entries 2-4). Adjusting the concentration of the reaction system still resulted in reduction in the yield of the target compound (Table 1, entries 5-7). Noteworthily, when we increased the amount of diene to 0.6 eq., the yield of **3a** remained relatively constant at 72%, and the yield of **4** decreased to below 10% (Table 1, entry 8). Interestingly, increasing the irradiation intensity by using 30 W and 18 W lamps together afforded **3a** in 80% yield, with no detectable dimer formation (Table 1, entry 9). Extending the reaction time from 18 to 24 h, the yield of **3a** showed no improvement (Table 1, entry 11).

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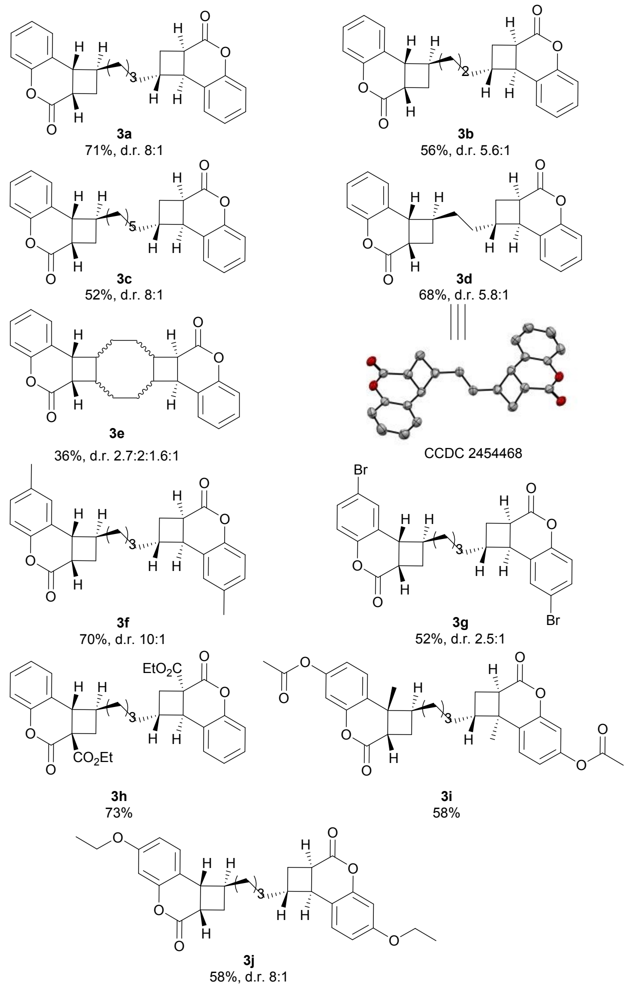
Finally, we examined the influence of the reaction solvent. When the reaction was carried out in tetrahydrofuran (THF), the desired product was obtained in a slightly lower yield of 65% (Table 1, entry 12). In contrast, switching the solvent to acetonitrile resulted in a significant drop in efficiency, affording only 13% of compound **3a** (Table 1, entry 13). We also explored the effect of different photosensitizers. Notably, the use of [Au(IPr)(Cbz)] as the photocatalyst delivered the product in 77% yield under otherwise identical conditions (Table 1, entry 14). A commonly used iridium photosensitizer with one of the highest triplet-energy value (E_T = 61.8 kcal/mol), [Ir(dF(CF$_3$)ppy)$_2$(dtbpy)]PF$_6$, as well as one of the most frequently used organic sensitizers thioxanthone and benzophenone, were examined. All three led to much lower yields of the product and high dimer yields, highlighting the superior efficiency of the gold sensitizers for this transformation. (Table 1, entry 15-17).

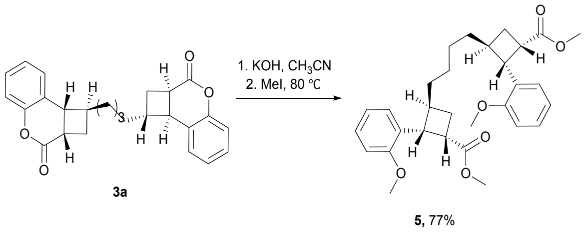


Scheme 2. Substrate scope. Unless otherwise noted, standard reaction conditions: 1 (0.40 mmol), 2 (0.24 mmol) and [Au(SIPr)(Cbz)] (0.004 mmol, 1 mol%) in EtOAc for 18 h under 365 nm LEDs (30W + 18W); yields are of isolated products; the diastereomers ratio was determined by ^{1}H NMR analysis of the crude reaction mixture.

With the optimized reaction conditions in hand (Table 1, entry 9), we continued to examine several representative dienes (Scheme 2). The model substrate, 1,7-octadiene, was successfully converted to product **3a** in 71% isolated yield. Investigating 1,6-heptadiene, 1,9-decadiene, and 1,5-hexadiene yielded the corresponding products **3b**, **3c**, and **3d** in 52–68% yield. The structure of compound **3d** was determined using X-ray diffraction analysis on single crystals (see SI). However, when short-chain dienes such as 1,3-butadiene were employed, only coumarin dimer formation was observed (see SI). Further exploration with 2,5-dimethylhexa-2,4-diene and 1,3-cyclooctadiene did not afford the desired products, which may be attributed to competitive quenching of the catalyst by the conjugated diene motifs. Beyond acyclic dienes, cyclic dienes were also examined. Upon reaction with 1,5-cyclooctadiene, the desired product **3e** was obtained as a mixture of diastereomers in a ratio of 2.7:2:1.6:1.

The influence of substituents on the coumarins scaffold was also investigated (Scheme 2). Methyl-substituted coumarin afforded the desired product **3f** in good yield of 70% with high diastereomeric ratio of 10:1. The reaction also proceeded with more sterically hindered 6-bromo-3-acetylcoumarin affording **3g** in 52% yield. Ester groups were well tolerated, the products **3h** and **3i** were obtained in yields of 73% and 58% respectively. The corresponding product **3j** with the 7-ethoxy-substituted coumarin was obtained in 58% yield. (Scheme 2).

As a preliminary demonstration of the synthetic manipulability of the bis-chromanone products, **3a** was transformed via basic hydrolysis followed by methylation with methyl iodide, affording the functionalized linked cyclobutane derivative **5** in 77% yield (Scheme 3). This result illustrates the potential of these products for further structural diversification, particularly in view of the established interest in cyclobutane-containing scaffolds in medicinal chemistry. [40–43]



Scheme 3. Conversion of 3a to cyclobutane derivative 5 in one step.

The relevant triplet energies were compared to rationalize the performance of the gold photocatalysts (Table 2). The triplet energy of [Au(SIPr)(Cbz)] is sufficient to sensitize coumarin **1a**, whereas the higher triplet energy of dimer **4** indicates that its activation by triplet energy transfer is energetically more demanding. Guided by these triplet-energy considerations, we employed density functional theory (DFT) calculations to gain further insight into the reaction mechanism (see the Computational Methodology in the SI). These calculations suggest that the triplet excited state of

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[Au(SIPr)(Cbz)] initiates the reaction by transferring energy to coumarin (**1a**), thereby generating the diradical ^{T}**1a** (Figure 2).[44] The subsequent addition of **2a** to the C$_{\alpha}$ position of ^{T}**1a** ($\Delta G^{\ddagger}$ = 10.4 kcal/mol) is preferred over addition to the C$_{\beta}$ position ($\Delta G^{\ddagger}$ = 14.2 kcal/mol). Radical pair recombination from the intermediate ^{T}**INT-1$_{exo}$** demands a high energy barrier of 40.2 kcal/mol on the triplet surface, whereas recombination on the open-shell singlet surface proceeds via a much lower barrier of 4.6 kcal/mol, furnishing the remarkably stable intermediate **INT-2**. Subsequently, **INT-2** reacts with ^{T}**1a** in a similar manner to deliver the desired product **3a**. On the other hand, generation of the homo-coupled product **4** via addition of **1a** to the C$_{\alpha}$ position of ^{T}**1a**, followed by radical pair recombination, needs surmounting a slightly higher energy barrier of 12.1 kcal/mol (Figure S2). Therefore, even if **4** is formed, it may dissociate again into **1a** and ^{T}**1a** via energy transfer from the triplet excited state of the gold photocatalyst, a moderately endoergic process (ΔE = 10.5 kcal/mol) (Figure S3).[16,39] Furthermore, oxidative and reductive quenching of the photoexcited [Au(SIPr)(Cbz)] by **4** are thermodynamically uphill by 19.9 and 62.0 kcal/mol, respectively (Figure S4), suggesting neither of these electron transfer pathways is operative.

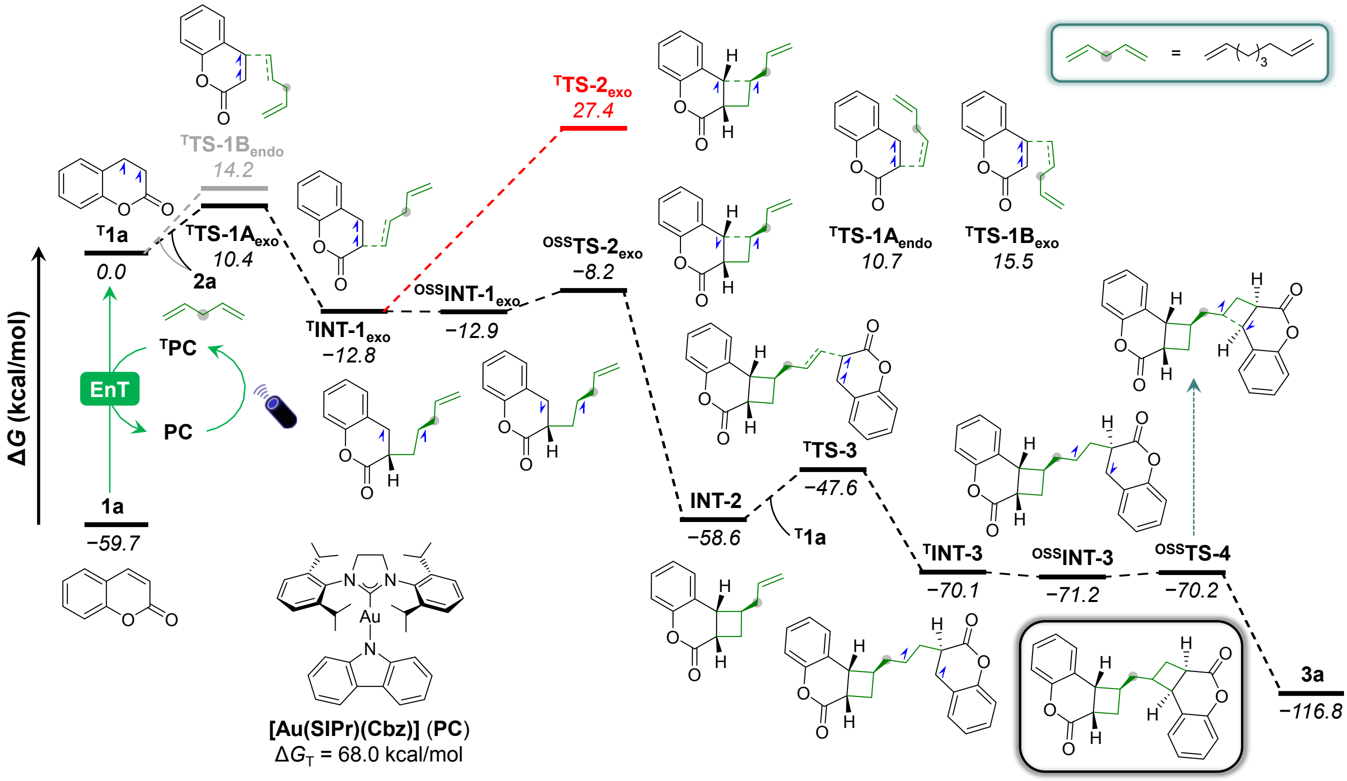

Figure 2. Free energy profile for the formation of **3a** at the ωB97X-D(SMD, EtOAc)/def2-TZVPP//ωB97X-D/def2-SVP level of theory.

Table 2. Comparison of triplet energies relevant to this transformation.

Species	E$_T$/ kcal/mol
[Au(SIPr)(Cbz)]	67.9
[Au(IPr)(Cbz)]	67.6
[Ir(dF(CF$_3$)ppy)$_2$(dtppy)]PF$_6$	61.8
Thioxanthone	65.5
Benzophenone	68.6
1a	59.7
4	74.6

To confirm the DFT-predicted reaction mechanism, several mechanistic experiments were conducted (see SI). Control experiments were conducted to verify that both a sensitizer and light irradiation are necessary for the reaction to proceed. We also carried out a control experiment using the dimer of coumarin with diene, showing that the dimer could be converted into the product under the reaction conditions.

In summary, a gold-mediated EnT photocatalytic approach for the synthesis of polymethylene-linked bis(cyclobutane-fused chromanones) has been developed. The method proceeds under mild conditions and enables the formation of structurally complex products in moderate to good yields. This study provides further insight into gold-mediated energy transfer photocycloadditions of coumarins with alkenes and highlights the unique potential of gold sensitizers in photocatalysis. Further studies aimed at probing the versatility of gold-mediated photocatalysts are ongoing in our laboratory.

Conflicts of interest

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There are no conflicts to declare.

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Data availability statement

All data included and leading to conclusions presented in this manuscript are included in the ESI.