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利用可见光引发 1,6-烯炔的增环酰化双官能化制备 1-茚酮衍生物

林 凤[†,a,b] 张 艳[†,b] 吴 明[b] 刘会艳[*,b]
郝文娟[b] 姜 波[*,a,b]
([a]江苏师范大学敬文书院 江苏徐州 221116)
([b]江苏师范大学化学与材料科学学院 江苏徐州 221116)

摘要 报道了一种新型光催化 1,6-烯炔参与的增环酰化−双官能化反应, 立体选择性地合成具有环季碳中心的酰基取代 1-茚酮衍生物, 收率中等至良好. 该光催化反应通过调整酰基自由基前体类型, 实现了不饱和烃的两类酰化官能化反应. 当使用芳酰氯为双官能团试剂时, 其与 1,6-烯炔发生增环氯酰化反应; 而使用酰基肟酯作为酰基自由基前体时, 与醇和 1,6-烯炔发生三组分增环烷氧基化酰化反应. 目前方法展现出良好官能团相容性、广泛底物范围和温和反应条件.
关键词 可见光催化反应; 增环氯酰化反应; 增环烷氧基化酰化反应; 1,6-烯炔衍生物

Visible-Light-Induced Annulative Acylative Difunctionalization of 1,6-Enynes for Accessing 1-Indanones

Lin, Feng[†,a,b] Zhang, Yan[†,b] Wu, Ming[b] Liu, Huiyan[*,b]
Hao, Wen-Juan[b] Jiang, Bo[*,a,b]
([a] *C.W. Chu College, Jiangsu Normal University, Xuzhou, Jiangsu 221116*)
([b] *School of Chemistry & Materials Science, Jiangsu Normal University, Xuzhou, Jiangsu 221116*)

Abstract A new photocatalytic annulative acylative difunctionalization of 1,6-enynes is reported, enabling stereoselective access to acylated 1-indanones with cyclic quaternary centers in moderate to good yields. This photocatalysis enables two types of acylation of unsaturated hydrocarbons by adjusting the categories of acyl radical precursors. Aroyl chlorides as bifunctional reagents react with 1,6-enynes to realize annulative chloroacylation, while acyl oxime esters are used as acyl radical precursors, which undergo a three-component annulative alkoxyacylation by treatment with 1,6-enynes and alcohols. The current method demonstrates good functional group compatibility, a broad substrate scope and mild reaction conditions
Keywords photoredox catalysis; annulative chloroacylation; annulative alkoxyacylation; 1,6-enynes

1 Introduction

The ketone group is a versatile and useful structural motif that is frequently distributed in a myriad of naturally occurring products and synthetically bioactive compounds.[1] Moreover, ketones could act as fundamental feedstocks in the preparation of natural products, active pharmaceutical ingredients, and other important derivatives in organic synthesis because of their synthetic accessibility and intrinsic ability to be converted to other diverse functional groups.[2] Consequently, chemists have made long-standing efforts to establish various efficient synthetic tactics for the synthesis of functionalized ketones.[3] Important methods for the synthesis of ketones include the oxidation of alcohols,[4] electrophilic acylation (acylation of carbon-centered nucleophiles),[5] the addition of acyl radicals across unsaturated systems,[6] and the acylation of carbon-centered radicals (radical acylation)[7] (Scheme 1a). Among them, the addition of acyl radicals across unsaturated carbon-carbon bonds has become a primary and powerful shortcut for the fabrication of ketones, benefiting from controllable radical-transfer direction, rich sources of acyl radicals, and availability of the reaction.[8] Specifically, a variety of acyl radical precursors, including aroyl

* Corresponding authors. E-mail: liuhuiyan72@163.com; jiangchem@jsnu.edu.cn
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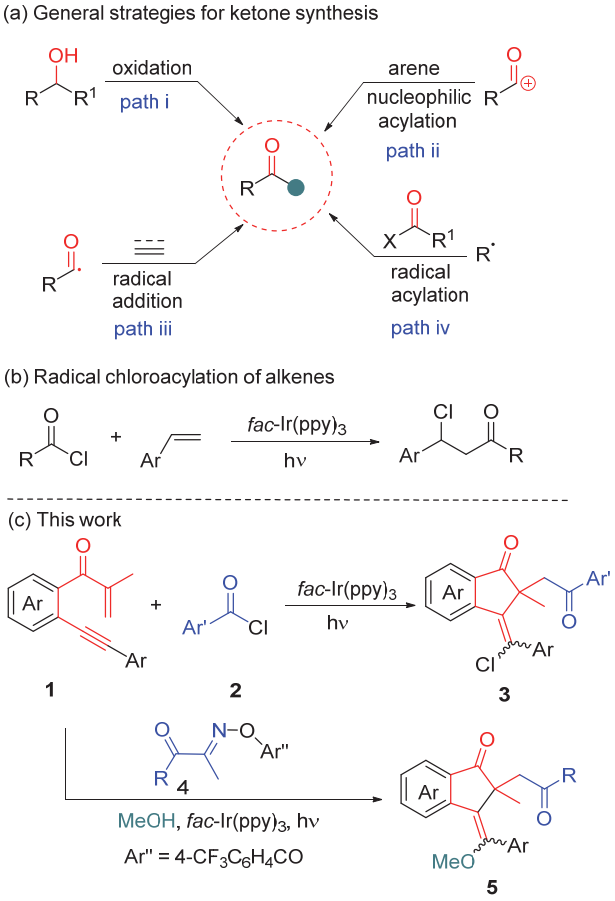
† 共同第一作者(The authors contributed equally to this work).



Scheme 1 Annulative acylated difunctionalization of 1,6-enynes

chlorides,[9] aryl anhydrides,[10] acylsilanes,[11] 2-pyridylthioesters,[12] carboxylic acids,[13] aldehydes,[14] α-keto acids,[15] α-hydroxylketones,[16] 4-acyl-1,4-dihydropyridines[17] and acyl oxime esters,[18] have been exploited, enabling single electron transfer (SET) under visible-light photocatalysis to access acylated products via acyl radical species. Despite these achievements, the development of new and efficient approaches for the generation of functionalized ketones with structural diversity via acyl radical-triggered transformations is highly desirable.

Visible-light-driven photoredox catalysis (VLPC) has become a flourishing and powerful tool for initiating various synthetic transformations under usually mild conditions by virtue of the impressive characteristics of visible light, including green, sustainability, and renewable energy sources.[19] Generally, photoredox catalysis can produce significant radical species with frequently unusual or unconventional reactivities via a single-electron transfer process, which are engaged in a remarkable variety of unusual chemical transformations to derive biologically important molecules that are difficult to obtain via thermal processes. Over the years, many state-of-the-art and seminal works have demonstrated the tremendous potential of visible light photoredox catalysis in organic synthesis.[20] Notably, Ngai,[21] Oh[22] and co-workers independently reported elegant photocatalytic acylation-functionalization of alkenes using aroyl chlorides as acyl radical precursors, providing a practical pathway for producing β-functionalized ketones. However, a survey of the literature reveals that there are no reports on the photoredox catalysis of 1,6-enynes with *in situ* generated acyl radical species to form acylated 1-indanones. To continue our interest in VLPC,[23] herein, we report a new and external oxidant-free annulative acylative difunctionalization of 1,6-enynes to synthesize 1-indanones via the photocatalysis of easily accessible substrates under very mild conditions. The use of aroyl chlorides as bifunctional reagents of chlorine and acyl sources enabled the chloroacylation of 1,6-enynes via an atom transfer radical cyclization (ATRC) approach, whereas annulative alkoxyacylation of 1,6-enynes can be realized by exploiting acyl oxime esters as acyl radical precursors and alcohols as alkoxylation reagents.

2 Results and discussion

At the beginning of our study, the preformed 1,6-enyne **1a** and *p*-tolyl chloride (**2a**) were selected as model substrates to screen the reaction conditions (Table 1), and the results are summarized in Table 1. The reaction proceeded readily in ethyl acetate (EtOAc) under argon conditions with the irradiation of 10 W blue LEDs by using *fac*-Ir(ppy)$_3$ (2 mol%) as a photocatalyst and 2,6-dichloropyridine (20 mol%) as a base, and the expected 1-indanone **3a** was provided in 85% yield with a >19∶1 *E*/*Z* ratio (Table 1, Entry 1). The *E*/*Z* ratio was determined through ^{1}H NMR analysis and by analogy with **3b** as a case study (Figure 1).[24] Decreasing or increasing the catalytic amount of *fac*-Ir(ppy)$_3$ did not improve the yield of **3a** (Entries 2, 3). Subsequent screening of several other organic bases, such as 2,4,6-collidine, 2,6-lutidine and 4-dimethylaminopyridine (DMAP) (Entries 4～6), revealed that none of them improved in the yield. The decrease in the loading of 2,6-dichloropyridine resulted in a marked decrease in the yield of **3a** (56%, Entry 7). The reaction did not proceed when ethyl acetate (EtOAc) was changed to tetrahydrofuran (THF) as the reaction medium (Entry 8). The reaction became messy under air conditions, and only a trace amount of **3a** was detected (Entry 9), implying that

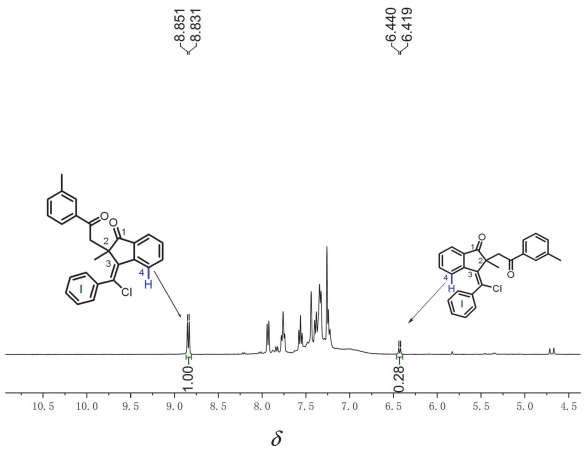
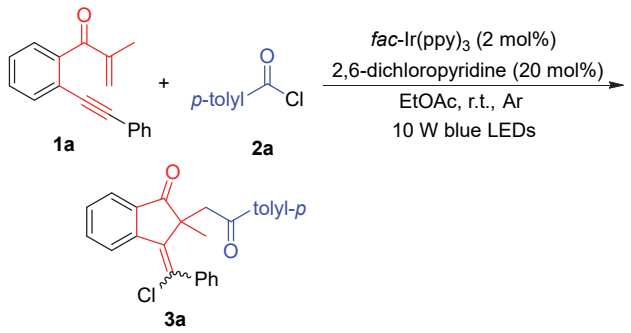


Figure 1 Determination of *Z*/*E* configuration of compound **3b**

Table 1 Condition optimization for product **3a**[a]



Entry	Variation of the established conditions	Yield[b]/%
1	None	85
2	1.0 mol% *fac*-Ir(ppy)$_3$	77
3	5.0 mol% *fac*-Ir(ppy)$_3$	85
4	2,4,6-Collidine (20 mol%) as the base	30
5	2,6-Lutidine (20 mol%) as the base	45
6	DMAP (20 mol%) as the base	0
7	2,6-Dichloropyridine (10 mol%)	56
8	THF instead of EtOAc	NR
9	In the air	Trace
10	Ru(bpy)$_3$Cl$_2$•6H$_2$O instead of *fac*-Ir(ppy)$_3$	Trace
11	Without *fac*-Ir(ppy)$_3$	0
12	No light	0

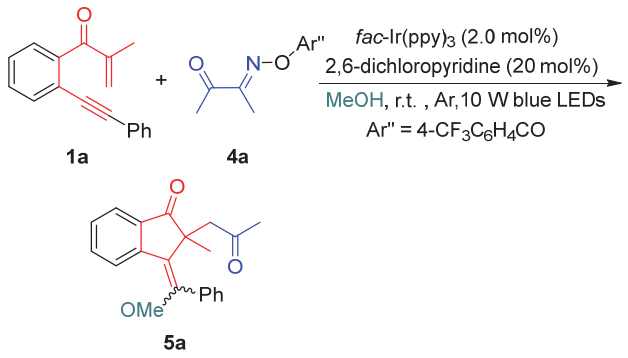
[a] Reaction conditions: **1a** (0.2 mmol), **2a** (2.0 equiv.), *fac*-Ir(ppy)$_3$ (2 mol%), 10 W Blue LEDs, EtOAc (2.5 mL) under Ar condition at 25 ℃ for 24 h. [b] Isolated yield based on **1a**. ppy=2-phenylpyridine; bpy=bipyridine, NR=no reaction.

O_2 could seriously deteriorate this transformation. Exchanging *fac*-Ir(ppy)$_3$ with Ru(bpy)$_3$Cl$_2$ completely suppressed the reaction process (Entry 10). No product **3a** was observed without the *fac*-Ir(ppy)$_3$ catalyst or light irradiation (Entries 11, 12), suggesting that both the photocatalyst and visible light are the keys to driving this transformation.

With these established reaction conditions in hand, the generality of photocatalytic annulative chloroacylation with respect to 1,6-enynes and aroyl chlorides was probed (Scheme 2). First, the scope of aroyl chlorides was evaluated, and several aroyl chlorides with different electronic properties at both aromatic rings were found to be compatible as illustrated in Scheme 2. Electronically rich (*e.g.* *meta*-methyl **2b**, *meta*-methoxy **2c**, *para*-*tert*-butyl **2d**), neutral (H **2e**) and poor (*para*-bromo **2f**) groups at different positions (*para* and *meta*) in the arene ring were well accommodated, enabling visible-light-induced annulation to furnish products **3b**～**3f** in 41%～66% yields and between 1∶1 and 5∶1 *E*/*Z* ratios. Similarly, 1,6-enyne bearing methoxy (**1b**), chloro (**1c**), or bromo (**1d**) group at the *para*-position in the aromatic ring directly bound to the alkyne unit (Ar) was workable for this catalytic protocol, delivering products **3g**～**3i** in acceptable yields, albert with moderate *E*/*Z* ratios. Notably, swapping aroyl chloride with *p*-toluenesulfonyl chloride (TsCl) **2g** enabled a completely stereoselective pathway to generate sulfonylated 1-indanone **3j** as a single *E*-stereoisomer in 85% yield.

After achieving photocatalytic annulative chloroacylation of 1,6-enynes, we turn our attention to evaluate the feasibility of annulative alkoxyacylation of 1,6-enynes.[25] The reaction of 1,6-enyne **1a** with benzoyl chloride was conducted in MeOH under standard conditions (Scheme 3). However, the reaction did not proceed through alkoxyacylation, probably because of the esterification between benzoyl chloride and MeOH in the presence of bases. To avoid esterification, we selected acyl oxime esters as acyl radical precursors for alkoxyacylation.[18] To our delight, the reaction of **1a** with acyl oxime ester **4a** proceeded smoothly along the direction of alkoxylation, and desired product **5a** was obtained in 45% yield with a 6∶1 *Z*/*E* ratio (Scheme 3). After simple optimization of the reaction conditions, the yield of **5a** was improved to 65% when the reaction worked in the presence of 1 mol% *fac*-Ir(ppy)$_3$ without any base (Table 2, Entry 3). Changing other reaction parameters, including the solvent (*e.g.*, EtOH), inorganic base (*e.g.*, K_2CO_3 and Na_2CO_3), and photocatalyst (*e.g.*, Eosin Y and Ru(bpy)$_3$Cl$_2$•6H$_2$O) did not enhance the efficiency of this transformation (Entries 4～8). Moreover, a lower conversion of **1a** into **5a** was detected under air conditions (Table 2, Entry 9). No reactions occurred without *fac*-Ir(ppy)$_3$ or light irradiation (Entries 10, 11).

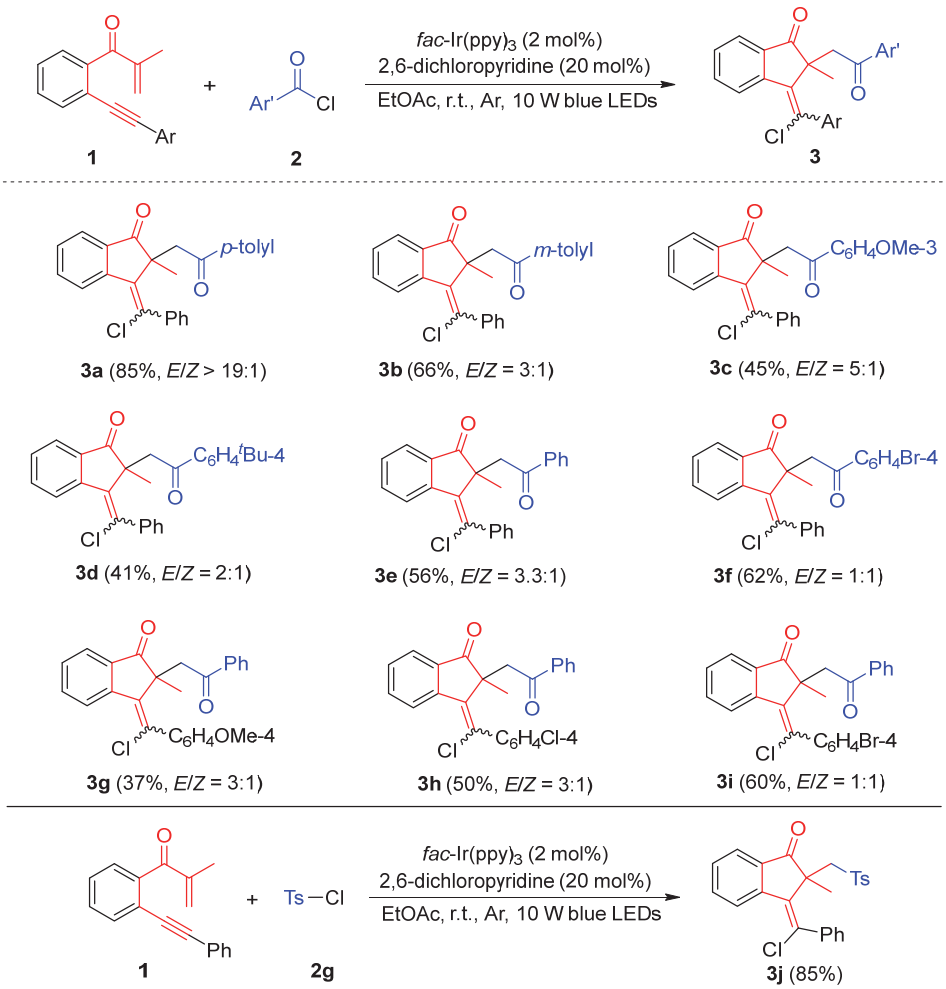
Table 2 Condition optimization for product **5a**[a]



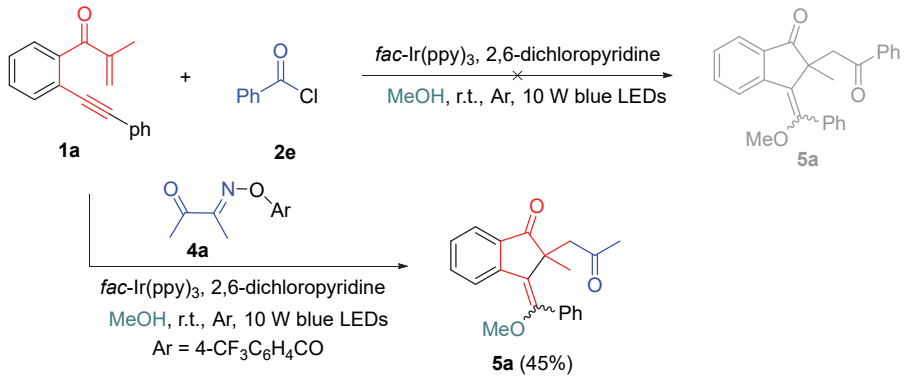
Entry	Variation of the established conditions	Yield[b]/%
1	None	45
2	1.0 mol% of *fac*-Ir(ppy)$_3$	45
3	1.0 mol% of *fac*-Ir(ppy)$_3$ without base	65
4	EtOH instead of MeOH	Trace
5	K_2CO_3 (2.0 equiv.)	21
6	Na_2CO_3 (2.0 equiv.)	53
7	Eosin Y instead of *fac*-Ir(ppy)$_3$	32
8	Ru(bpy)$_3$Cl$_2$•6H$_2$O instead of *fac*-Ir(ppy)$_3$	Trace
9	In the air	55
10	Without *fac*-Ir(ppy)$_3$	0
11	No light	0

[a] Reaction conditions: **1a** (0.2 mmol), **4a** (1.5 equiv.), *fac*-Ir(ppy)$_3$ (1 mol%), 10 W Blue LEDs, MeOH (2.0 mL) under Ar conditions at 25 ℃ for 24 h. [b] Isolated yield based on **1a**. ppy=2-phenylpyridine; bpy=bipyridine.

Following these acceptable conditions, we extended our studies to the scope of 1,6-enynes and acyl oxime esters for annulative alkoxyacylation (Scheme 4). Substituents inc-

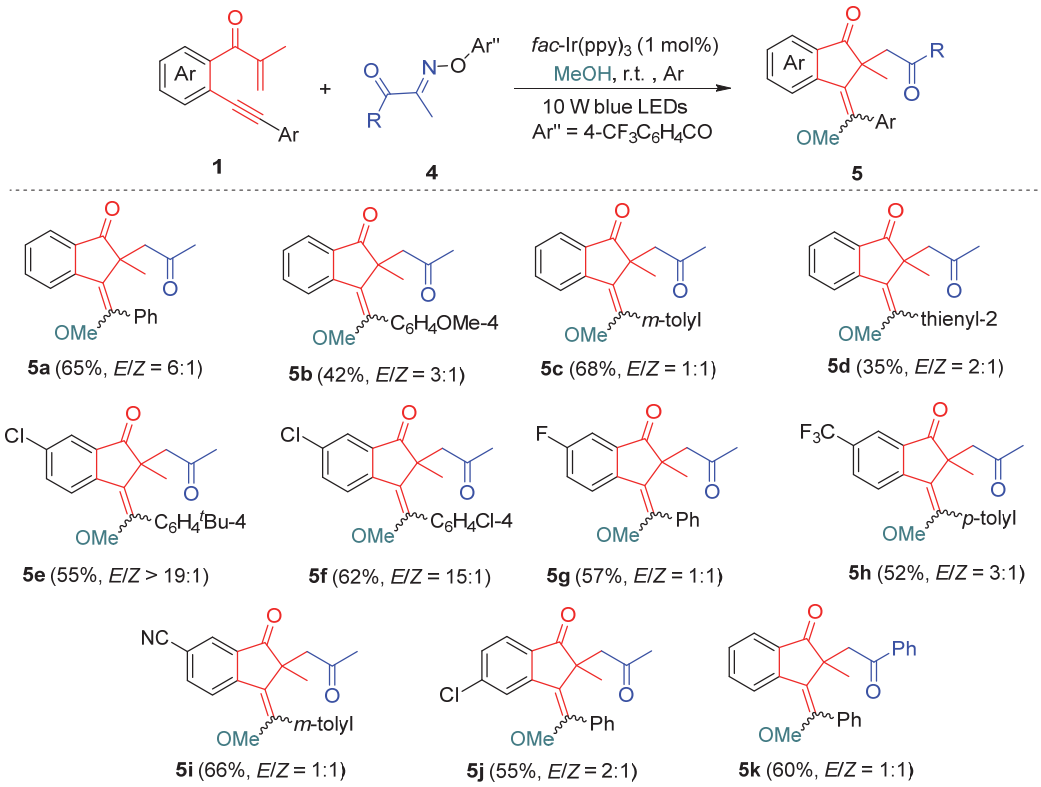


Scheme 2 Annulative chloro-functionalization of 1,6-enynes



Scheme 3 Attempts at the alkoxyacylation of 1,6-enynes

luding methoxy and methyl at different positions of the arylalkynyl (Ar) moiety were converted into corresponding products **5b** and **5c** in 42% and 68% yields, respectively. Notably, the 2-thienyl counterpart showed high reactivity, providing 2-thienyl-substituted 1-indanone **5d** with inseparable stereoisomers in 35% yield. Next, various functional groups, such as chloro, fluoro, trifluoromethyl, and cyano, at the C4 or C5 position in the internal arene ring were proven to be applicable for this alkoxyacylation, as products **5e**～**5j** with acceptable yields were afforded. Among these groups, two challenging cases with trifluoromethyl and cyano functionalities as strongly electronically deficient groups were effective for this catalytic protocol, demonstrating the good compatibility of this photocatalysis. Swapping the acetyl group with a benzoyl group in the oxime ester substrate led to the formation of product **5k** with equal amounts of stereoisomers in 60% yield. Moreover, a scale-up reaction of **1a** with **4a** was conducted with



Scheme 4 Annulative alkoxyacylation of 1,6-enynes

a 1.0 mmol scale, furnishing product **5a** in a slightly decreased 60% yield. The resulting 1-indanones were fully identified via NMR spectroscopy and HRMS data. Furthermore, the stereo-structure of **5g** was confirmed by X-ray diffraction analysis (Figure 2, CCDC 2387944). Moreover, *Z*/*E*-configuration was confirmed by ^{1}H NMR analysis (Figure 1). Using compound **3b** as a case study, when exocyclic double bond is in *Z*-configuration, the proton at the C4 position of the 1-indenone ring is shielded by the benzene ring **I**, resulting in an upfield shift of its ^{1}H NMR absorption to appear as a doublet at δ 6.43.[25] In contrast, when the exocyclic double bond adopts in *E*-configuration, there is no shielding effect between the proton at C4 position and the benzene ring **I**, thus remains unshifted and appears as a doublet at δ 8.84. In the case of compound **3b**, *E*-isomer predominates as the major stereoisomer.

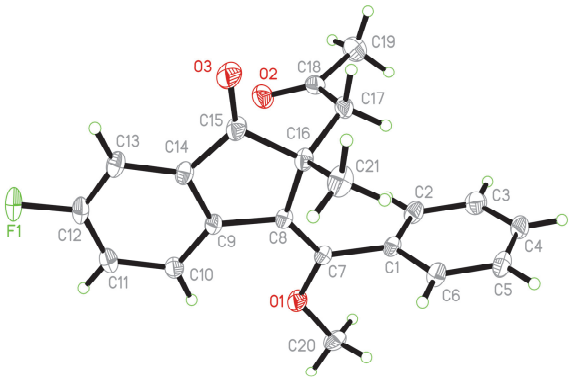


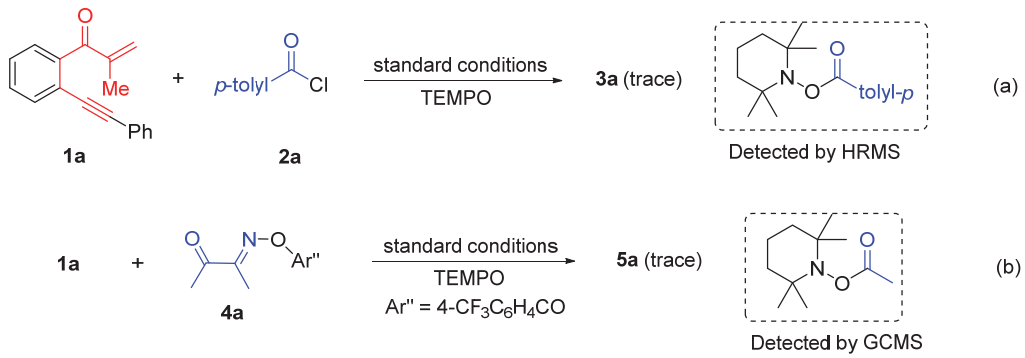
Figure 2 X-Ray structure of **5g**

To elucidate this mechanism of photocatalysis, the reactions of **1a** with **2a** (or **4a**) in the presence of 2,2,6,6-tetramethyl-piperidine-*N*-oxyl (TEMPO, 2.0 equiv.) were carried out under standard conditions (Schemes 5a and 5b). As a result, this process was severely inhibited, and TEMPO-acyl adducts were detected by HRMS and GC-MS, respectively, indicating that an acyl radical process may be involved.

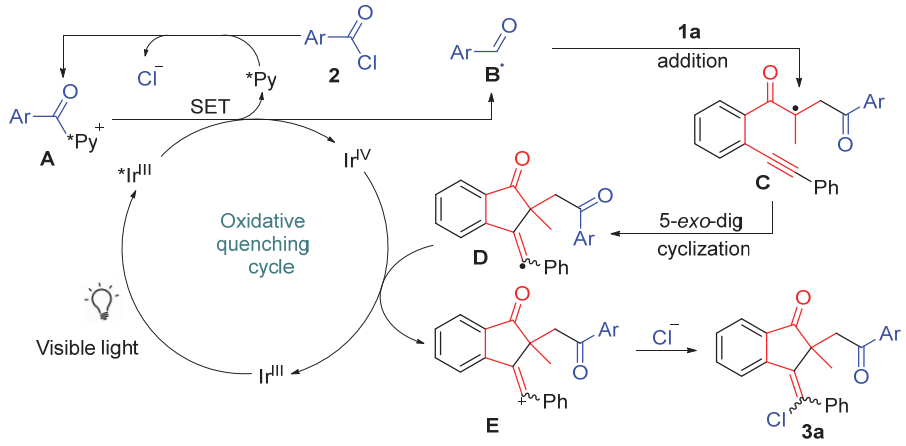
Combined with these observations and literature reports,[21,22,24,26] a reasonable reaction pathway for the formation of products **3** is proposed in Scheme 6. Upon exposure to visible light, the excited state of the *Ir(III) complex, which is rapidly generated from the normal Ir(III) complex, reduces *N*-acylpyridinium ion **A**, derived from aroyl chloride **2** and 2,6-dichloropyridine, via single electron transfer (SET) to generate acyl radical **B**,[27] together with Ir(IV) species and 2,6-dichloropyridine. Next, acyl radical **B** undergoes regioselective addition and 5-*exo*-*dig*-cyclization to produce alkenyl radical **D**. Intermediate **D** is oxidized by Ir(IV) species to yield alkenyl cation **E** and regenerate the Ir(III) complex for the next catalytic cycle. The former cation **E** is captured by the chloride anion to afford the final product **3a**. The formation of **5a** is similar to the above proposed pathway.

3 Conclusions

In summary, we have developed a new photocatalytic annulative acylative difunctionalization of 1,6-enynes under mild conditions, enabling stereoselective access to acylated 1-indanones with a quaternary center in moderate to



Scheme 5 Control experiments



Scheme 6 Proposed pathway toward product **3a**

good yields. An annulative chloroacylation of 1,6-enynes was realized by taking advantage of aroyl chlorides as bifunctional reagents of chlorine and acyl sources, whereas the three-component annulative alkoxyacylation of 1,6-enynes produced acylated 1-indanones with acyl oxime esters as acyl radical precursors and alcohols as the alkoxylation reagents. The current method provides a unified and operationally simple strategy for preparing 1-indanones with substitution diversity, featuring good functional group compatibility and a broad substrate scope. Further application of photocatalytic acylative functionalization for the forging of important cyclic structures is underway in our laboratory.

4 Experimental section

4.1 Materials and methods

^{1}H NMR (^{13}C NMR) spectra were measured on a Bruker DPX 400 MHz spectrometer in $CDCl_3$ with chemical shift (δ) given relative to TMS as internal standard. HRMS (APCI and ESI) was determined by using microTOF-QII HRMS/MS instrument (BRUKER).

4.2 General procedure for the synthesis of products **3a** and **5a**

To a dry Schlenk reaction tube equipped with a magnetic stir bar, were added 1,6-enyne **1a** (49.2 mg, 0.2 mmol), 4-methylbenzoyl chloride (**2a**, 61.6 mg, 0.4 mmol), and *fac*-Ir(ppy)$_3$ (0.004 mmol, 2.6 mg). The Schlenk tube was closed with a septum, evacuated and refilled with Ar conditions. 2,6-Dichloropyridine (7.6 mg, 0.04 mmol) and dry EtOAc (2.5 mL) was added via syringe. The mixture was placed in LH LABWARE photosynthesis instrument by 10 W Blue-LED lamp and stirred at room temperature and monitored by thin-layer chromatography (TLC) for 24 h. The mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (elution with petroleum ether to 30∶1 ether/EtOAc). to afford the desired product **3a** (85% yield) as a yellow solid.

To a dry Schlenk reaction tube equipped with a magnetic stir bar, was added 1,6-enyne **1a** (49.2 mg, 0.2 mmol), acyl oxime ester **4a** (82 mg, 0.03 mmol), *fac*-Ir(ppy)$_3$ (0.002 mmol, 1.3 mg, 1 mol%). The Schlenk tube was closed with a septum, evacuated and refilled with Ar conditions. MeOH (2.0 mL) was added via syringe. The mixture was placed in LH LABWARE photosynthesis instrument by 10W Blue-LED lamp and stirred at room temperature and monitored by TLC for 24 h. The mixture was concentrated under reduced pressure and purified by via column chromatography on silica gel (elution with petroleum ether to 10∶1 ether/EtOAc) to afford the desired product **5a** (65% yield) as a yellow solid.

4.3 Scale-up reaction of **1a**

To a dry Schlenk reaction tube equipped with a magnetic stir bar, were added 1,6-enyne **1a** (246 mg, 1 mmol),

acyl oxime ester **4a** (410 mg, 0.15 mmol), and *fac*-Ir(ppy)$_3$ (0.01 mmol, 6.5 mg, 1 mol%). The Schlenk tube was closed with a septum, evacuated and refilled with Ar. MeOH (4.0 mL) was added via syringe. The mixture was placed in LH LABWARE photosynthesis instrument by 10 W Blue-LED lamp and stirred at room temperature and monitored by TLC for 24 h. The mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (elution with petroleum ether to 10∶1 ether/EtOAc) to afford the desired product **5a** (192 mg, 60% yield) as a yellow solid.

4.4 Compound characterization

(*E*)-3-(Chloro(phenyl)methylene)-2-methyl-2-(2-oxo-2-(*p*-tolyl)ethyl)-2,3-dihydro-1*H*-inden-1-one (**3a**): White solid, 68.1 mg, 85% yield. m.p. 138～140 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.84 (d, J=8.0 Hz, 1H), 7.93 (d, J=7.6 Hz, 1H), 7.80～7.72 (m, 1H), 7.58～7.53 (m, 1H), 7.51 (d, J=7.6 Hz, 2H), 7.49～7.39 (m, 1H), 7.38～7.29 (m, 2H), 7.22～7.13 (m, 3H), 7.12～7.06 (m, 1H), 3.32 (d, J=17.2 Hz, 1H), 2.76 (d, J=18.8 Hz, 1H), 2.39 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.1, 196.1, 146.8, 144.1, 139.7, 138.6, 135.9, 134.6, 133.3, 129.5, 129.1 (d, J=9.1 Hz), 124.1, 122.7, 52.1, 46.0, 21.8. HRMS (ESI) calcd for $C_{26}H_{22}ClO_2$ $[M+H]^+$ 401.1308, found 401.1304.

(*E*)-3-(Chloro(phenyl)methylene)-2-methyl-2-(2-oxo-2-(*m*-tolyl)ethyl)-2,3-dihydro-1*H*-inden-1-one (**3b**, major): Yellow solid, 52.9 mg, 66% yield. m.p. 131～133 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.84 (d, J=8.0 Hz, 1H), 7.93 (d, J=7.6 Hz, 1H), 7.86～7.72 (m, 2H), 7.58～7.54 (m, 1H), 7.46 (d, J=17.6 Hz, 2H), 7.42～7.31 (m, 5H), 7.23 (d, J=7.2 Hz, 1H), 3.32 (d, J=18.8 Hz, 1H), 2.78 (d, J=18.8 Hz, 1H), 2.34 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.0, 196.6, 146.8, 139.7, 138.2, 134.6, 134.0, 129.5, 129.0, 128.7, 128.3, 127.5, 125.4, 124.1, 52.1, 46.1, 25.7, 21.4. HRMS (ESI) calcd for $C_{26}H_{22}ClO_2$ $[M+H]^+$ 401.1308, found 401.1313.

(*E*)-3-(Chloro(phenyl)methylene)-2-(2-(3-methoxyphenyl)-2-oxoethyl)-2-methyl-2,3-dihydro-1*H*-inden-1-one (**3c**, major): Yellow solid, 37.9 mg, 45% yield. m.p. 140～142 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.85 (d, J=8.0 Hz, 1H), 7.94 (d, J=7.6 Hz, 1H), 7.81～7.74 (m, 1H), 7.59～7.54 (m, 1H), 7.49～7.41 (m, 1H), 7.38～7.32 (m, 2H), 7.28～7.21 (m, 3H), 7.18～7.03 (m, 3H), 3.78 (s, 3H), 3.34 (d, J=18.8 Hz, 1H), 2.77 (d, J=18.8 Hz, 1H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 205.9, 196.3, 159.7, 146.7, 139.6, 138.4, 137.0, 135.8, 134.7, 129.5, 129.3, 129.0, 127.5, 124.1, 120.9, 120.3, 111.6, 55.4, 52.1, 46.1, 25.6. HRMS (ESI) calcd for $C_{26}H_{22}ClO_3$ $[M+H]^+$ 417.1257, found 417.1266.

(*E*)-2-(2-(4-(*tert*-Butyl)phenyl)-2-oxoethyl)-3-(chloro(phenyl)methylene)-2-methyl-2,3-dihydro-1*H*-inden-1-one (**3d**, major): Yellow solid, 36.3 mg, 41% yield. m.p. 135～137 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.84 (d, J=8.0 Hz, 1H), 7.99～7.85 (m, 2H), 7.79～7.73 (m, 1H), 7.59～7.51 (m, 3H), 7.50～7.42 (m, 2H), 7.37 (d, J=8.0 Hz, 4H), 3.32 (d, J=18.8 Hz, 1H), 2.78 (d, J=18.4 Hz, 1H), 1.33 (s, 9H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.0, 196.1, 157.0, 146.8, 139.7, 138.6, 135.9, 134.6, 134.0, 129.5, 128.2, 128.1, 125.6, 125.3, 124.1, 52.1, 46.1, 35.2, 31.2, 25.6, 21.8. HRMS (ESI) calcd for $C_{29}H_{28}ClO_2$ $[M+H]^+$ 443.1778, found 443.1780.

(*E*)-3-(Chloro(phenyl)methylene)-2-methyl-2-(2-oxo-2-phenylethyl)-2,3-dihydro-1*H*-inden-1-one (**3e**, major): Yellow solid, 43.3 mg, 56% yield. m.p. 142～144 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.85 (d, J=8.4 Hz, 1H), 7.95～7.91 (m, 1H), 7.89～7.68 (m, 2H), 7.61 (d, J=7.7 Hz, 2H), 7.60～7.49 (m, 3H), 7.47～7.41 (m, 1H), 7.39～7.30 (m, 4H), 3.35 (d, J=18.8 Hz, 1H), 2.76 (d, J=18.8 Hz, 1H), 1.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 205.9, 196.4, 146.7, 139.6, 138.5, 135.7, 134.6, 133.2, 129.5, 129.0, 128.4, 128.1, 127.5, 127.2, 124.1, 52.1, 46.0, 25.7. HRMS (ESI) calcd for $C_{25}H_{20}ClO_2$ $[M+H]^+$ 387.1152, found 387.1146.

(*E*)-2-(2-(4-Bromophenyl)-2-oxoethyl)-3-(chloro(phenyl)methylene)-2-methyl-2,3-dihydro-1*H*-inden-1-one (**3f**, major): Yellow solid, 57.7 mg, 62% yield. m.p. 129～131 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07～7.76 (m, 3H), 7.57～7.51 (m, 2H), 7.50～7.37 (m, 4H), 7.37～7.27 (m, 2H), 7.19 (d, J=4.4 Hz, 1H), 6.94 (d, J=7.6 Hz, 1H), 3.82 (d, J=18.4 Hz, 1H), 3.76 (d, J=18.4 Hz, 1H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.5, 196.4, 146.3, 144.6, 136.6, 135.8, 134.8, 134.1, 133.4, 131.9, 130.4, 128.7, 128.4, 128.2, 125.2, 124.2, 122.2, 121.2, 47.1, 45.4, 25.3. HRMS (ESI) calcd for $C_{25}H_{20}BrClO_2$ $[M+H]^+$ 465.0257, found 465.0268.

(*E*)-3-(Chloro(4-methoxyphenyl)methylene)-2-methyl-2-(2-oxo-2-phenylethyl)-2,3-dihydro-1*H*-inden-1-one (**3g**, major): Yellow solid, 30.8 mg, 37% yield. m.p. 110～112 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.83 (d, J=8.0 Hz, 1H), 7.99～7.81 (m, 2H), 7.78～7.73 (m, 1H), 7.65 (d, J=7.6 Hz, 2H), 7.57～7.51 (m, 2H), 7.45～7.34 (m, 3H), 7.34～7.27 (m, 1H), 3.77 (s, 3H), 3.37 (d, J=18.8 Hz, 1H), 2.85 (d, J=18.8 Hz, 1H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.2, 196.4, 159.9, 147.1, 135.8, 134.6, 133.2, 129.4, 128.4, 128.1, 127.5, 124.1, 113.9, 55.4, 52.2, 46.0, 25.7. HRMS (ESI) calcd for $C_{26}H_{22}ClO_3$ $[M+H]^+$ 417.1257, found 417.1258.

(*E*)-3-(Chloro(4-chlorophenyl)methylene)-2-methyl-2-(2-oxo-2-phenylethyl)-2,3-dihydro-1*H*-inden-1-one (**3h**, major): Yellow solid, 42.1 mg, 50% yield. m.p. 125～127 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.81 (d, J=8.0 Hz, 1H), 8.00～7.80 (m, 2H), 7.80～7.74 (m, 1H), 7.64 (d, J=7.6 Hz, 2H), 7.60～7.53 (m, 2H), 7.45 (d, J=7.2 Hz, 1H), 7.43～7.34 (m, 3H), 7.31 (d, J=8.0 Hz, 1H), 3.41 (d, J=18.8 Hz, 1H), 2.80 (d, J=18.4 Hz, 1H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 205.5, 196.2, 146.5, 139.2, 138.0, 135.8, 135.5, 135.1, 134.7, 133.5, 129.7, 128.6, 128.5, 128.2, 128.0, 127.5, 125.7, 124.2, 52.1, 46.0, 25.7. HRMS (ESI) calcd for $C_{25}H_{19}Cl_2O_2$ $[M+H]^+$ 421.0762, found 421.0768.

(*E*)-3-((4-Bromophenyl)chloromethylene)-2-methyl-2-(2-oxo-2-phenylethyl)-2,3-dihydro-1*H*-inden-1-one (**3i**,

major): Yellow solid, 55.8 mg, 60% yield. m.p. 124～126 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.92～7.83 (m, 2H), 7.76 (d, J=7.6 Hz, 1H), 7.61～7.49 (m, 2H), 7.47～7.42 (m, 1H), 7.40 (d, J=9.2 Hz, 1H), 7.35 (d, J=8.0 Hz, 2H), 7.33～7.28 (m, 3H), 7.22～7.16 (m, 1H), 7.07 (d, J=7.6 Hz, 1H), 3.74 (s, 1H), 3.30 (s, 1H), 1.35 (d, J=7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.4, 143.6, 143.1, 137.1, 134.0, 131.9, 131.6, 129.6, 129.5, 128.7, 128.5, 128.4, 128.3, 125.2, 123.9, 121.2, 46.9, 45.2, 25.3. HRMS (ESI) calcd for $C_{25}H_{19}BrClO_2$ $[M+H]^+$ 465.0257, found 465.0263

(*E*)-3-(Chloro(phenyl)methylene)-2-methyl-2-(tosylmethyl)-2,3-dihydro-1*H*-inden-1-one (**3j**): Yellow solid, 74.1 mg, 85% yield. m.p. 134～136 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.92 (d, J=4.0 Hz, 1H), 7.82～7.76 (m, 2H), 7.61 (s, 1H), 7.57～7.47 (m, 4H), 7.47～7.38 (m, 3H), 7.21 (d, J=8.0 Hz, 2H), 3.50 (d, J=16.0 Hz, 1H), 3.15 (d, J=12.0 Hz, 1H), 2.40 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 203.5, 147.1, 144.6, 139.1, 137.2, 135.5, 131.3, 129.9, 128.5, 127.9, 127.4, 124.2, 61.9, 51.2, 25.7, 21.6. HRMS (ESI) calcd for $C_{25}H_{22}ClO_3S$ $[M+H]^+$ 437.0978, found 437.0986.

(*E*)-3-(Methoxy(phenyl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5a**, major): Yellow solid, 41.7 mg, 65% yield. m.p. 105～107 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.37 (d, J=8.0 Hz, 1H), 8.22 (d, J=8.4 Hz, 1H), 7.81 (d, J=7.6 Hz, 1H), 7.75 (d, J=8.0 Hz, 1H), 7.63～7.67 (m, 1H), 7.48～7.50 (m, 2H), 7.37～7.38 (m, 2H), 3.43 (s, 3H), 2.85 (d, J=18.4 Hz, 1H), 2.06 (d, J=18.4 Hz, 1H), 1.81 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 208.0, 205.0, 152.3, 148.7, 134.9, 134.1, 134.0, 130.6, 130.0, 129.6, 128.6, 127.3, 127.1, 123.7, 121.40, 55.9, 50.6, 48.6, 29.1, 26.1. HRMS (ESI) calcd for $C_{21}H_{21}O_3$ $[M+H]^+$ 321.1491, found 321.1490.

(*E*)-3-(Methoxy(4-methoxyphenyl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5b**, major): Yellow oil, 29.4 mg, 42% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.36 (d, J=8.0 Hz, 1H), 7.80 (d, J=7.6 Hz, 1H), 7.61～7.65 (m, 1H), 7.33～7.37 (m, 1H), 7.21 (d, J=7.6 Hz, 2H), 6.99 (d, J=7.6 Hz, 2H), 3.89 (s, 3H), 3.41 (s, 3H), 2.86 (d, J=18.4 Hz, 1H), 2.15 (d, J=18.4 Hz, 1H), 1.83 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.2, 204.2, 159.6, 151.5, 148.0, 134.0, 133.1, 130.5, 129.8, 126.3, 126.2, 125.3, 122.8 120.5, 113.1, 54.9, 54.6, 49.9, 47.8, 28.3, 25.2. HRMS (ESI) calcd for $C_{22}H_{23}O_4$ $[M+H]^+$ 351.1596, found 351.1593.

(*E*)-3-(Methoxy(*m*-tolyl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5c**, major): colorless oil, 45.5 mg, 68% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.36 (d, J=8.0 Hz, 1H), 7.80 (d, J=7.6 Hz, 1H), 7.73 (d, J=7.2 Hz, 1H), 7.61～7.66 (m, 1H), 7.35～7.39 (m, 2H), 7.22～7.20 (m, 2H), 3.35 (s, 3H), 3.20 (d, J=18.4 Hz, 1H), 2.83 (d, J=18.4 Hz, 1H), 2.53 (s, 3H), 2.17 (s, 3H), 1.48 (s, 3H). HRMS (ESI) calcd for $C_{22}H_{23}O_3$ $[M+H]^+$ 335.1647, found 335.1642.

(*E*)-3-(Methoxy(thiophen-2-yl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5d**, major): Yellow solid, 22.8 mg, 35% yield. m.p. 98～100 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.22 (d, J=8.0 Hz, 1H), 7.75 (d, J=8.0 Hz, 2H), 7.52～7.59 (m, 2H), 7.21～7.23 (m, 1H), 7.15～7.18 (m, 1H), 3.46 (s, 3H), 3.21 (d, J=18.4 Hz, 1H), 2.35 (d, J=18.4 Hz, 1H), 2.04 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.7, 206.0, 147.7, 143.9, 134.9, 134.2, 130.7, 130.5, 129.0, 127.8, 127.2, 125.7, 123.9, 123.5, 55.8, 50.6, 50.5 29.8, 23.0; IR (KBr) ν: 1712.8, 1599.6, 1524.1, 1359.6 cm^{-1}. HRMS (ESI) calcd for $C_{19}H_{19}SO_3$ $[M+H]^+$ 327.1055, found 327.1054.

(*Z*)-3-((4-(*tert*-Butyl)phenyl)(methoxy)methylene)-6-chloro-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5e**, major): yellow oil, 45.2 mg, 55% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (s, 1H), 7.49 (d, J=7.2 Hz, 2H), 7.26-7.23 (m, 2H), 7.07 (d, J=8.8 Hz, 1H), 6.12 (d, J=8.4 Hz, 1H), 3.63 (d, J=17.2 Hz, 1H), 3.36 (s, 3H), 3.18 (d, J=17.6 Hz, 1H), 2.05 (s, 1H), 1.39 (s, 9H), 1.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.0, 206.0, 153.3, 153.0, 146.7, 136.2, 133.9, 132.5, 130.5, 126.3, 124.6, 123.4, 123.4, 121.1, 56.1, 50.8, 50.6, 34.1, 31.4, 29.8, 22.9. HRMS (ESI) calcd for $C_{25}H_{28}ClO_3$ $[M+H]^+$ 411.1727, found 411.1724.

(*E*)-6-Chloro-3-((4-chlorophenyl)(methoxy)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5f**, major): Colorless solid, 48.2 mg, 62% yield. m.p. 108～110 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (d, J=8.4 Hz, 1H), 7.75 (s, 1H), 7.58 (d, J=8.4 Hz, 1H), 7.27～7.29 (m, 2H), 7.17～7.22 (m, 2H), 3.41 (s, 3H), 2.88 (d, J=18.4 Hz, 1H), 2.07 (d, J=18.4 Hz, 1H), 1.84 (s, 3H), 1.07 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.3, 204.8, 151.4, 146.7, 135.3, 134.8, 133.4, 131.9, 131.8 130.3, 129.8, 129.7 128.3, 123.4, 121.1, 116.0, 115.8, 55.9, 50.8, 48.9, 29.1, 25.9. HRMS (ESI) calcd for $C_{21}H_{19}Cl_2O_3$ $[M+H]^+$ 389.0711, found 389.0713.

(*E*)-6-Fluoro-3-(methoxy(phenyl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5g**, major): Yellow oil, 38.6 mg, 57% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.33～8.37 (m, 1H), 7.48～7.50 (m, 3H), 7.42～7.41 (m, 2H), 7.34～7.37 (s, 2H), 3.41 (s, 3H), 2.83 (d, J=18.4 Hz, 1H), 2.46 (d, J=18.8 Hz, 1H), 2.06 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.2, 204.9, 162.1 ($^1J_{CF}$=247.6 Hz), 151.7, 144.8 ($^4J_{CF}$=2.4 Hz), 136.7 ($^3J_{CF}$=7.0 Hz), 136.8 133.8, 130.0, 129.9, 128.9, 128.6, 125.0 ($^3J_{CF}$=7.6 Hz), 122.2 ($^2J_{CF}$=23.0 Hz), 109.4 ($^2J_{CF}$=21.8 Hz), 55.9, 50.8, 49.2, 29.8, 26.0. 376; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −114.0. HRMS (ESI) calcd for $C_{21}H_{20}FO_3$ $[M+H]^+$ 339.1396, found 339.1391.

(*E*)-3-(Methoxy(*p*-tolyl)methylene)-2-methyl-2-(2-oxopropyl)-6-(trifluoromethyl)-2,3-dihydro-1*H*-inden-1-one (**5h**, major): Yellow oil, 41.8 mg, 52% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96 (s, 1H), 7.36～7.26 (m, 5H), 3.42 (s, 3H), 2.86 (d, J=19.2 Hz, 1H), 2.46 (s, 3H), 2.13 (d, J=19.2 Hz, 1H), 2.06 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.0, 206.1, 154.7, 151.5, 140.1, 134.8, 131.1 (J_{CF}=3.2 Hz), 130.3, 129.4, 128.5 (J_{CF}=32.7 Hz), 127.5, 124.2 (J_{CF}=270.8 Hz), 123.8, 121.0 (J_{CF}=3.9

Hz), 120.8, 56.0, 50.8, 50.5, 29.7, 22.7, 21.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −62.6. HRMS (ESI) calcd for $C_{23}H_{22}F_3O_3$ $[M+H]^+$ 403.1521, found 403.1520.

(*E*)-1-(Methoxy(*p*-tolyl)methylene)-2-methyl-3-oxo-2-(2-oxopropyl)-2,3-dihydro-1*H*-indene-5-carbonitrile (**5i**, major): Yellow solid, 47.4 mg, 66% yield. m.p. 113～115 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.45 (d, J=8.4 Hz, 1H), 7.87 (s, 1H), 7.74 (d, J=8.0 Hz, 1H) 7.32～7.30 (m, 2H), 7.14 (d, J=7.6 Hz, 2H), 3.43 (s, 3H), 2.85 (d, J=18.8 Hz, 1H), 2.47 (s, 3H), 2.13 (d, J=19.2 Hz, 1H), 1.85 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 206.4, 206.0, 152.0, 140.6, 137.2, 135.0,130.6, 130.0, 129.5, 128.0, 127.8, 124.1, 120.8, 118.7, 110.0, 56.2, 50.9, 50.4, 29.7, 25.7, 21.7. HRMS (ESI) calcd for $C_{23}H_{22}NO_3$ $[M+H]^+$ 360.1600, found 360.1605.

(*E*)-5-Chloro-3-(methoxy(phenyl)methylene)-2-methyl-2-(2-oxopropyl)-2,3-dihydro-1*H*-inden-1-one (**5j**, major): yellow oil, 39.0 mg, 55% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.36 (s, 1H), 7.72 (d, J=8.0 Hz, 1H), 7.49～7.55 (m, 5H), 7.33 (d, J=8.0 Hz, 2H), 3.44 (s, 3H), 2.82 (d, J=18.8 Hz, 1H), 2.06 (d, J=19.2 Hz, 1H), 1.82 (s, 3H), 1.07 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 207.2, 204.2, 159.6, 151.5, 148.0, 134.0, 133.1, 130.5, 126.3, 126.2, 125.3, 122.8, 120.5, 113.1, 54.8, 54.6, 49.9, 47.8, 28.3, 25.2. HRMS (ESI) calcd for $C_{21}H_{20}ClO_3$ $[M+H]^+$ 355.1101, found 355.1104.

(*E*)-3-(Methoxy(phenyl)methylene)-2-methyl-2-(2-oxo-2-phenylethyl)-2,3-dihydro-1*H*-inden-1-one (**5k**, major): yellow oil, 45.9 mg, 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.43 (d, J=8.0 Hz, 1H), 7.87 (d, J=7.2 Hz, 1H), 7.62～7.65 (m, 2H), 7.40～7.53 (m, 6H), 7.22～7.26 (m, 4H), 3.39 (s, 3H), 3.29 (d, J=18.4 Hz, 1H), 2.76 (d, J=18.8 Hz, 1H), 1.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 208.0, 196.5, 152.3, 148.7, 136.1, 134.8, 134.1, 133.8, 133.0, 130.1, 129.4, 128.5, 128.4, 128.1, 127.1, 123.9, 123.7, 55.8, 48.6, 46.3, 23.1. HRMS (ESI) calcd for $C_{26}H_{23}O_3$ $[M+H]^+$ 383.1647, found 383.1643.

Supporting Information ^{1}H NMR and ^{13}C NMR spectra of products **3a**～**3j** and **5a**～**5k**. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn/.

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