有机化学 DOI: 10.6023/cjoc202205021 研究论文
Chinese Journal of Organic Chemistry ARTICLE

P(NMe$_2$)$_3$介导1,2-二羰基化合物与α,β-不饱和酮的[1+4]环化反应及多取代2,3-二氢呋喃的合成

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摘要 在P(NMe$_2$)$_3$作用下, 1,2-二羰基化合物与α,β-不饱和酮顺利发生分子间[1+4]环化反应, 生成带有全碳季碳中心的多取代2,3-二氢呋喃类化合物, 从而以较高的收率及较宽的底物范围提供了合成该类化合物的新方法. 在某些情况下, [1+2]环化反应竞争发生生成多取代的环丙烷化合物. 借助密度泛函理论(DFT)计算手段, 讨论了反应机理及有关[1+2]环化反应与[1+4]环化反应的化学选择性.

关键词 [1+4]环化反应; 1,2-二羰基化合物; [1+2]环化反应; 2,3-二氢呋喃; Kukhtin-Remirez加合物

Synthesis of Polysubstituted 2,3-Dihydrofurans via P(NMe$_2$)$_3$-Mediated [1+4] Annulation of 1,2-Dicarbonyl Compounds with α,β-Unsaturated Ketones

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Abstract A [1+4] annulation of 1,2-dicarbonyl compounds with α,β-unsaturated ketones has been readily realized under the mediation of P(NMe$_2$)$_3$, producing polysubstituted 2,3-dihydrofurans bearing a quaternary carbon center in fair to good yields with a broad substrate scope. This reaction accordingly constitutes a new synthetic method for such compounds. In some cases, a [1+2] annulation reaction competitively occurs, delivering polysubstituted cyclopropanes. With the aid of density functional theory (DFT) calculation, a plausible mechanism for the [1+4] annulation and the chemoselectivity between [1+2] and [1+4] annulations has been discussed.

Keywords [1+4] annulation; 1,2-dicarbonyl compounds; [1+2] annulation; 2,3-dihydrofuran; Kukhtin-Remirez adduct

1 Introduction

The 2,3-dihydrofuran motif, which contains a quaternary carbon chiral center, widely exists in natural products and pharmaceuticals, as well as some important intermediates in medicine and organic synthesis.[1-2] Therefore, the development of efficient synthetic methods has attracted considerable interest from organic chemists. Many methods have been reported for the synthesis of 2,3-dihydrofurans, in which [1+4] cyclization is one of the most effective synthesis strategies.[3-4] A variety of strategies to construct five-membered rings through [1+4] cyclization have been successfully realized by using transition-metal catalytic or other non-metallic methods. For example, Kurahashi and coworkers[4e] reported a nickel-catalyzed [1+4] cycloaddition of enones with methylenecyclopropanes, leading to dihydrofurans. The reaction outcome is attributed to the unique reactivity of methylenecyclopropane, which is incorporated into a five-membered ring as a one-carbon synthon. Among the metal-free methods, nucleophilic phosphine catalysis plays an important role in the formation of C(1) synthons. Electron-deficient allenes, maleimides and modified Morita-Baylis-Hillman (MBH) adducts are often used as C(1) synthons in the phosphine-catalyzed [1+4] annulation reactions.[4f]

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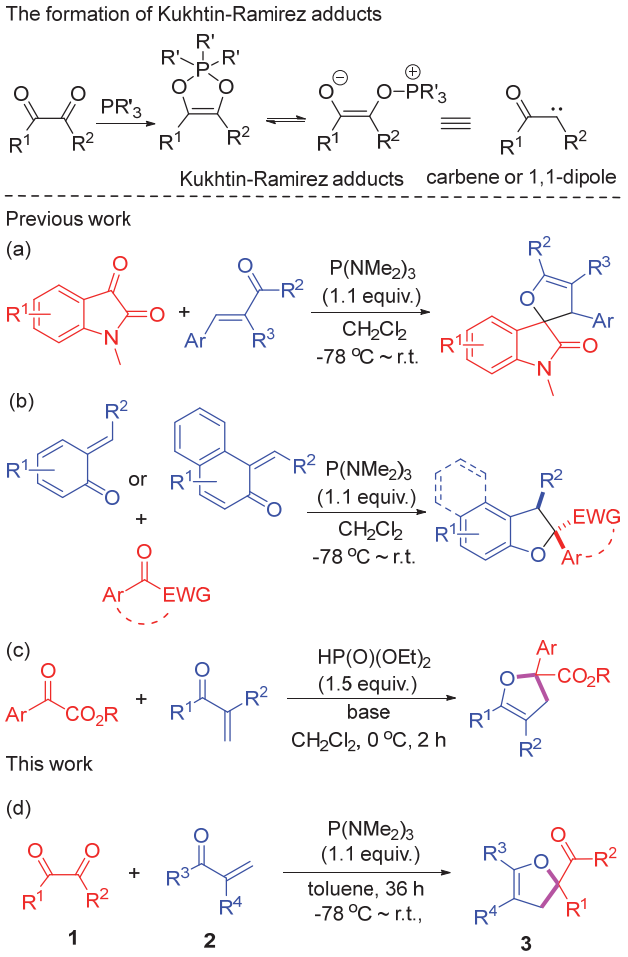
Received May 16, 2022; revised July 5, 2022; published online July 13, 2022.
Project supported by the National Natural Science Foundation of China (Nos. 21472096, 22171143).
国家自然科学基金(Nos. 21472096, 22171143)资助项目.



Scheme 1 Phosphorus reagent-mediated [1＋4] annulation reactions of 1,2-dicarbonyls, leading to dihydrofurans.

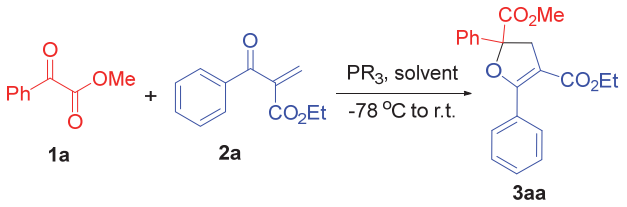
On the contrary, 1,2-dicarbonyl compounds are usually used as C(1) synthons in stoichiometric phosphine-mediated [1＋4] annulations. The Kukhtin-Ramirez adducts[5-6] of 1,2-dicarbonyl compounds and phosphines exhibit rich and unique reactivity. Recent studies have revealed that they can serve as 1,1-dipoles or carbene analogues to realize a series of metal-free annulation and insertion reactions.[7-11] An array of Kukhtin-Ramirez adduct-initiated [1＋4] annulations of different C(4) synthons have been reported.[9-11] Among them, He's group[9b] and Ashfeld's group[11] have both reported the [1＋4] annulation of cyclic 1,2-dicarbonyl compounds with *α*,*β*-unsaturated ketones in the presence of $P(NMe_2)_3$ leading to spirodihydrofurans. He's group[9b] reported the first [1＋4] annulation reaction between isatins and enones as a practical approach to construct spirooxindole-dihydrofurans (Scheme 1, a), while Ashfeld and coworkers[11] successfully realized the [1＋4] annulation of cyclic 1,2-dicarbonyls with *o*-quinone methides, providing an elegant access to spirodihydrobenzofurans with a quaternary center at C(2) (Scheme 1, b). Although above encouraging progress has been made, the [1＋4] annulation reaction of acyclic 1,2-dicarbonyl compounds with *α*,*β*-unsaturated ketones has not yet been reported. Recently, He's group[12] reported a diethyl phosphite mediated reductive [1＋4] annulation of *α*-ketoesters with *α*,*β*-unsaturated ketones, leading to polysubstituted dihydrofurans (Scheme 1, c). Inspired by the previous work,[9-12] and as a continuation of our efforts to develop [1＋4] annulation reactions, we have further studied a $P(NMe_2)_3$-mediated [1＋4] annulation reaction of acyclic 1,2-dicarbonyl compounds with *α*,*β*-unsaturated ketones based on the chemistry of Kukhtin-Ramirez adducts (Scheme 1, d). Herein we report the relevant results.

2 Results and discussion

We initialized the investigation with **1a** and **2a** as the model substrates. An expected [1＋4] annulation product **3aa** was collected in 43% yield after the model reaction was stirred in CH_2Cl_2 at －78 ℃ to room temperature for 36 h (Table 1, Entry 1). This result showed that a [1＋4] annulation was successfully realized when *α*,*β*-unsaturated ketone **2a** was selected as an electrophilic reagent to react with *in situ* generated Kukhtin-Ramirez adduct. The reaction conditions were then screened to further improve the reaction efficiency. The results showed that phosphites $P(OMe)_3$ and $P(OEt)_3$, and phosphine PPh_3 were all ineffective in the reaction (Table 1, Entries 2～4). Thus, $P(NMe_2)_3$ was chosen as the preferred trivalent phosphorus reagent in the reaction. Seven common solvents were further investigated in the reaction. The results showed that the solvents had a significant impact on the yield of the reaction (Table 1, Entries 5～11). Almost no annulation product **3aa** was detected in the reaction run in ethanol for 36 h (Table 1, Entry 5). The protic ethanol presumably quenched the *in situ* generated basic intermediate Kukhtin-Ramirez adduct from **1a** and $P(NMe_2)_3$,[7a] leading to the failure of the annulation reaction. In the solvents such as tetrahydrofuran (THF), Et_2O, and $CHCl_3$, no improvements in the product yield were achieved (Table 1, Entries 6～8). The yield of **3aa** was even lowered to 7% when THF was used as the solvent (Table 1, Entry 6). When toluene, 1,4-dioxane and acetonitrile were used as the solvent, moderate yields could be obtained (Table 1, Entries 9～11). In toluene, the yield of **3a** was further increased to 71% (Table 1, Entry 11). Toluene was therefore chosen as the preferred solvent. Finally, the reaction time was also investigated to improve the reaction efficiency. A series of parallel experiments were conducted for different reaction times such as 12, 24 and 48 h (Table 1, Entries 12～14). Results showed that the isolated yield of **3aa** was even slightly lowered to 63% when the reaction time was elongated to 48 h (Table 1, Entry 14). Thus, we chosen the reaction conditions listed in Table 1 Entry 11 as the preferred.

With the optimal conditions in hand, the substrate scope of the [1＋4] annulation reaction was investigated (Table 2). First, methyl benzoylformate **1a** as 1,2-dicarbonyl compound was employed, and a series of *α*,*β*-unsaturated ketones **2** with varied substituent R^3 were examined (Table 2, **3ab**～**3ap**). When R^3 were substituted phenyl groups

Table 1 Survey on the model reaction conditions[a]



Entry	PR_3	Solvent	Time/h	Yield[b]/%
1	$P(NMe_2)_3$	CH_2Cl_2	36	43
2	PPh_3	CH_2Cl_2	36	Trace
3	$P(OMe)_3$	CH_2Cl_2	36	Trace
4	$P(OEt)_3$	CH_2Cl_2	36	Trace
5	$P(NMe_2)_3$	Ethanol	36	Trace
6	$P(NMe_2)_3$	THF	36	7
7	$P(NMe_2)_3$	Et_2O	36	41
8	$P(NMe_2)_3$	$CHCl_3$	36	23
9	$P(NMe_2)_3$	1,4-Dioxane	36	57
10	$P(NMe_2)_3$	CH_3CN	36	60
11	$P(NMe_2)_3$	Toluene	36	71
12	$P(NMe_2)_3$	Toluene	12	23
13	$P(NMe_2)_3$	Toluene	24	40
14	$P(NMe_2)_3$	Toluene	48	63

[a] Typical condition: under N_2 atmosphere, $P(NMe_2)_3$ (0.22 mmol, 1.1 equiv.) was added dropwise by a microsyringe to a stirred mixture of **1a** (0.20 mmol, 1.0 equiv.) and **2a** (0.22 mmol, 1.1 equiv.) in solvent (2.0 mL) at −78 ℃. Then the resulting mixture was warmed up slowly to room temperature by removing the cooling bath and stirred at room temperature for 36 h. [b] Isolated yield.

with an electron-donating or electron-withdrawing group at the *ortho*, *meta* or *para* position, the corresponding ketones **2b**～**2j** readily delivered the annulation products **3ab**～**3aj** in moderate yields. 3,4,5-Trimethoxyphenyl-substituted ketone **2k** also yielded its normal cyclization product **3ak**. 2-Naphthyl-substituted (**2i**) and 2-thienyl-substituted (**2j**) ketones smoothly afforded their corresponding dihydrofurans **3ai** and **3aj** in the reactions with benzoylformate **2a**. Alkyl groups like methyl, cyclohexyl and *n*-pentyl (R^3= Me, cyclohexyl, *n*-pentyl) were all tolerated in the reactions with benzoylformate **2a**. Their corresponding substituted ketones **2n**～**2p** uneventfully delivered normal annulation products **3an**～**3ap** in modest yields. The yield of **3an** was relatively lower. The reason for the low yield of **3an** may be attributed to the existence of *α*-acidic hydrogens of the ketone **2n**, since the *in situ* generated Kukhtin-Ramirez adduct in the reaction is strongly basic.[7a] Next, the scope of 1,2-dicarbonyl compound substrate **1** was further examined in the reaction with *α*,*β*-unsaturated ketone **2a** employed (Table 2).

A series of substituted benzoylformates **1b**～**1i** were first screened. Results showed that benzoylformates **1b** ～ **1i** bearing halo or methyl substituent on the benzene ring were all effective in the reactions with *α*,*β*-unsaturated ketone **2a**, readily delivering their corresponding products **3ba**～**3ia** in modest yields. Other 1,2-dicarbonyls such as benzil (**1j**), diethyl ketomalonate (**1k**) and 9,10-phenanthraquinone (**1l**) were also tested. The [1＋4] annulation reaction of benzil **1j** and ketone **2a** smoothly produced the normal product **3ja** in 45% yield. Diethyl ketomalonate **1k** readily reacted with ketones **2a**, **2q**, and **2r**, respectively, affording their corresponding products **3ka**, **3kq** and **3kr** in excellent to moderate yields. 9,10-Phenanthraquinone **1l** was also effective in the reaction with ketone **2a**, affording a spiro compound in 34% yield. Diethyl ketomalonate **1k** could also react with cyclic ketone 2-methylene cyclohexanone (**2s**), producing a bicyclic compound **3ks** in 68% yield (Table 2).

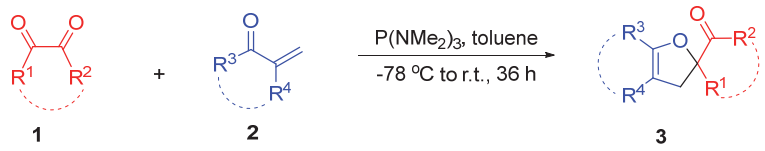
The scope of *α*,*β*-unsaturated ketones **2** was further examined (Table 3). Under similar conditions, *α*,*β*-unsaturated ketones **2q**, **2r** and **2t** (R^4=Me, Ph, H), however, chemoselectively underwent a [1＋2] annulation reaction with some 1,2-dicarbonyls. For example, ketone **2q** (R^3=Ph, R^4=Me) reacted with benzoylformates **1a** and **1m**, respectively, affording the corresponding cyclopropanation products **4aq** and **4mq** in moderate yields and low diastereoselectivities (Table 3). Ketone **2r** (R^3=R^4=Ph) delivered cyclopropane **4ar** in 49% yield in the reaction with benzoylformate **1a**. Ketone **2t** (R^3=Ph, R^4=H) also delivered cyclopropanation products **4at** and **4kt** in relatively low yields in the reactions with benzoylformate **1a** and ketomalonate **1k** under the similar conditions (Table 3). Finally, as a highly active 1,2-dicarbonyl substrate, *N*-CH_3 isatin **1n** was also examined in the reaction with ketone **2a**, delivering both [1＋4] annulation product **3na** and [1＋2] annulation product **4na** in good combined yield (Scheme 2).

All new compounds obtained in this study have been identified by NMR (^1H and ^{13}C) and HRMS. The structures of representative compounds **3ad** (CCDC: 2084148) and **4na** (CCDC: 2084150) have been further confirmed by X-ray single crystal diffraction analysis.

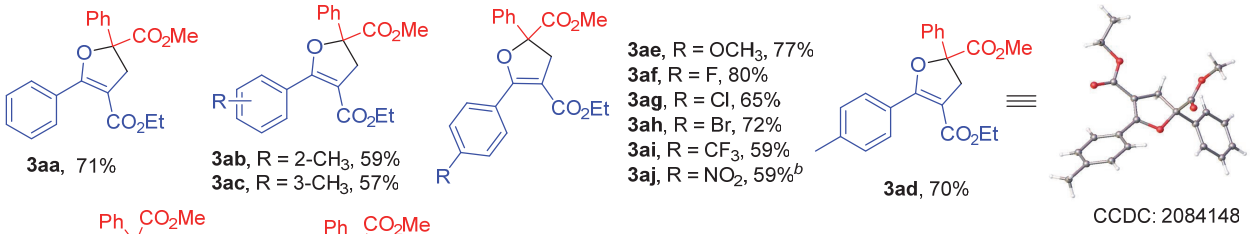
On the basis of above experimental results and closely related reports,[9a-9b] a plausible reaction mechanism is shown in Scheme 3. First, 1,2-dicarbonyl compound **1** reacts with $P(NMe_2)_3$ to form reactive Kukhtin-Ramirez adduct. In its form of zwitterions, the adduct engages in Michael addition with *α*,*β*-unsaturated ketone **2** to generate a resonance-stabilized zwitterions **I**. When its resonance contributor **Ia** acts as an oxygen anion nucleophile and undergoes an intramolecular displacement (path a), the [1＋4] annulation reaction occurs, leading to dihydrofuran **3**. On the other hand, when its resonance contributor **Ib** acts as a carbanion nucleophile and engages in an intramolecular displacement (path b), the [1＋2] annulation reaction occurs, leading to cyclopropane **4** (Scheme 3).

To rationalize the chemoselectivity between competitive [1＋2] annulation and [1＋4] annulation, we performed relevant DFT calculations on selected annulation reactions (Scheme 4). We first calculated the Gibbs free energy difference between the transition state **TS-1** for the [1＋2] annulation and the transition state **TS-2** for the [1＋4] annulation in the reaction of *N*-CH_3 isatin **1n** with ethyl 2-benzoylacrylate **2a**. The result showed that the Gibbs free energy of **TS-2** is marginally 0.08 kJ/mol higher than that of **TS-1** (Scheme 4). This result implies that the [1＋2] annu-

Table 2 [1+4] annulation of 1,2-dicarbonyl compounds **1** and α,β-unsaturated ketones **2**[a]



1 **2** **3**



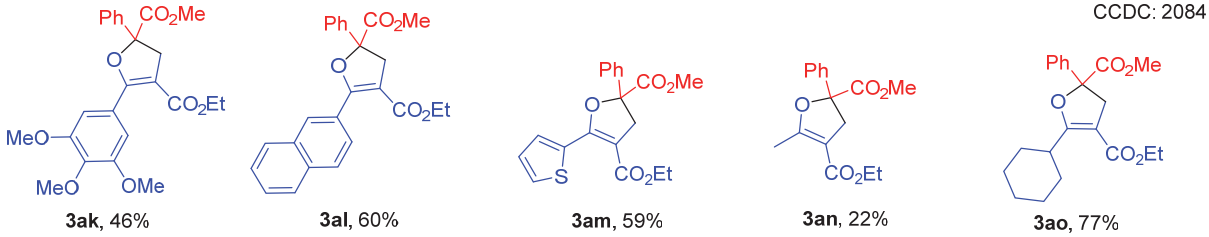
3aa, 71%

3ab, R = 2-CH_3, 59%
3ac, R = 3-CH_3, 57%

3ae, R = OCH_3, 77%
3af, R = F, 80%
3ag, R = Cl, 65%
3ah, R = Br, 72%
3ai, R = CF_3, 59%
3aj, R = NO_2, 59%[b]

3ad, 70%

CCDC: 2084148



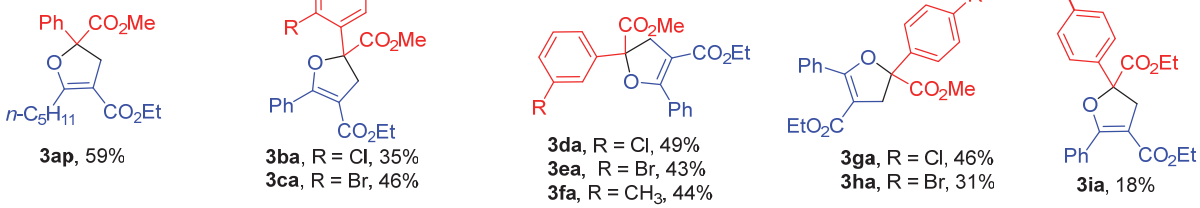
3ak, 46%

3al, 60%

3am, 59%

3an, 22%

3ao, 77%



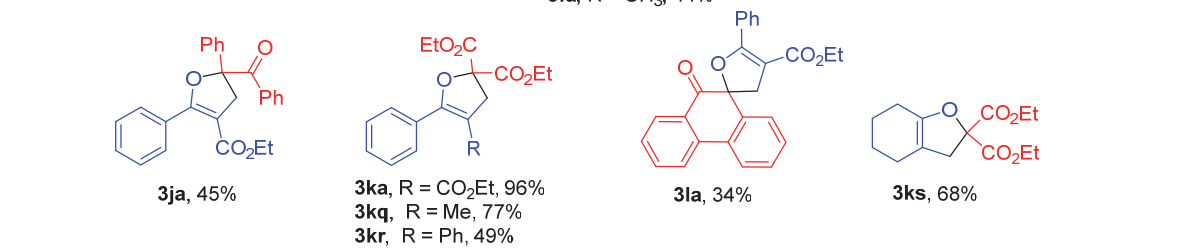
3ap, 59%

3ba, R = Cl, 35%
3ca, R = Br, 46%

3da, R = Cl, 49%
3ea, R = Br, 43%
3fa, R = CH_3, 44%

3ga, R = Cl, 46%
3ha, R = Br, 31%

3ia, 18%



3ja, 45%

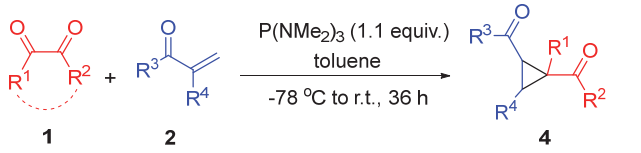
3ka, R = CO_2Et, 96%
3kq, R = Me, 77%
3kr, R = Ph, 49%

3la, 34%

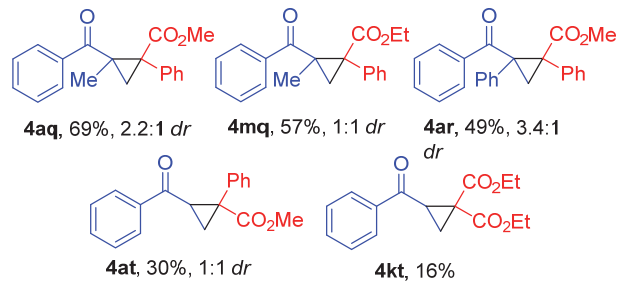
3ks, 68%

[a] For detail, see Experimental section. Isolated yield. [b] CH_2Cl_2 was used instead of solvent toluene.

Table 3 [1+2] annulations of 1,2-dicarbonyl compounds **1** and α,β-unsaturated ketones **2**[a]



1 **2** **4**



4aq, 69%, 2.2:1 *dr*

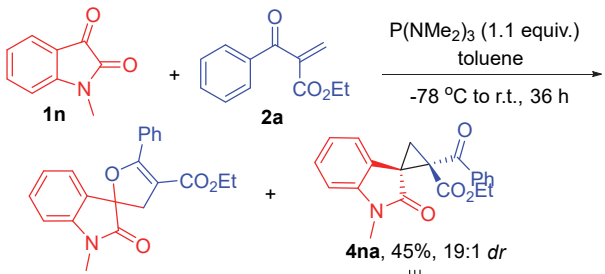
4mq, 57%, 1:1 *dr*

4ar, 49%, 3.4:1 *dr*

4at, 30%, 1:1 *dr*

4kt, 16%

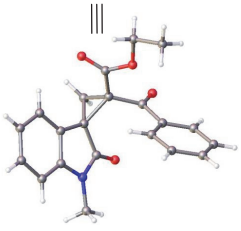
[a] For detail, see Experimental Section. Isolated yield and *dr* ratio was determined by ^{1}H NMR assay.



1n **2a**

3na, 38%

4na, 45%, 19:1 *dr*



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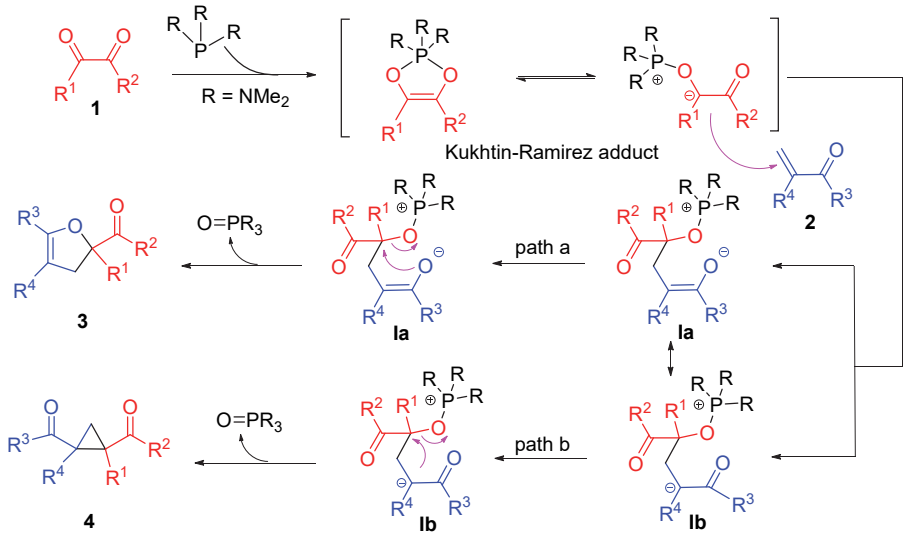
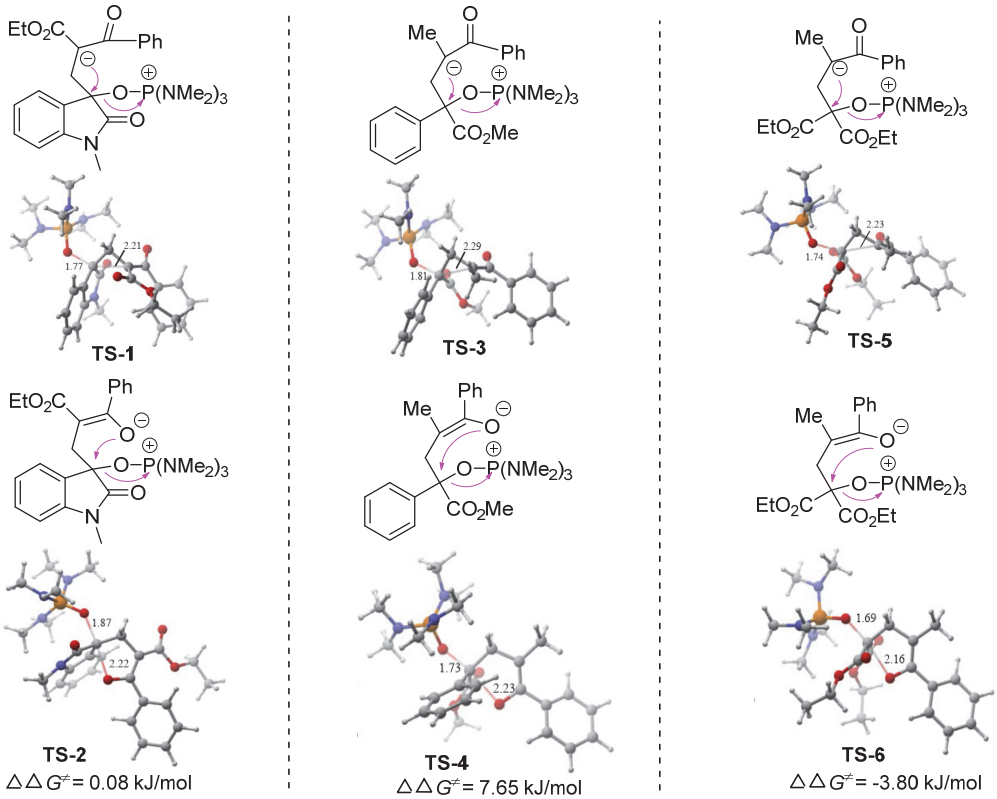
Scheme 2 Reaction of *N*-CH_3 isatin **1n** and ethyl 2-benzoylacrylate **2a**



Scheme 3 A plausible mechanism for formation of compounds **3** and **4**

lation product and the [1+4] annulation product can be competitively formed in the reaction. Next, we calculated the energy difference between the transition state **TS-3** for the [1+2] annulation and the transition state **TS-4** for the [1+4] annulation in the reaction of methyl benzoylformate **1a** with *α*,*β*-unsaturated ketone **2q**. It was found that the Gibbs free energy of **TS-4** is substantially 1.83 kcal/mol higher than that of **TS-3**. This result implies that the formation of cyclopropane product is highly favored. For the reaction of diethyl ketomalonate **1k** with *α*,*β*-unsaturated ketone **2q**, the energy difference between the transition state **TS-5** for the [1+2] annulation and the transition state **TS-6** for the [1+4] annulation was also calculated. The result showed that the Gibbs free energy of the transition state **TS-6** was 0.91 kcal/mol lower than that of the transition state **TS-5**. This result implies that the dihydrofuran product **3kq** is favored in the reaction of **1k** and **2q** (Scheme 4). The above theoretical calculation results are basically consistent with the actual experimental results (Tables 2 and 3, Scheme 2).



Scheme 4 DFT calculations

3 Conclusions

In summary, we have successfully developed a P(NMe$_2$)$_3$-mediated reductive [1＋4] annulation of 1,2-dicarbonyls **1** and *α*,*β*-unsaturated ketones **2**. It provides a metal-free and functional group-compatible strategy to synthesize versatile 2,3-dihydrofuran motif with an all-carbon quaternary carbon center. In some cases, [1＋2] and [1＋4] annulations chemoselectively occurred. A plausible mechanism has been proposed. Also, by the DFT calculations, the chemoselectivity between the [1＋2] annulation and the [1＋4] annulation is rationalized.

4 Experimental section

Solvents were purified prior to use according to conventional procedures. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard. HRMS spectra were acquired in the ESI mode (positive ion) with the mass analyzer of TOF used. Column chromatography was performed on silica gel (200～300 mesh) using a mixture of petroleum ether/ethyl acetate as eluent. *α*-Ketoesters **1a**, **1g** and **1i** are commercially available, other *α*-ketoesters and *α*,*β*-unsaturated ketones were prepared according to literature procedures.[13-17]

4.1 General procedure for P(NMe$_2$)$_3$-mediated annulation of 1,2-dicarbonyls **1** and *α*,*β*-unsaturated ketones **2**

Under a N_2 atmosphere and at －78 ℃, to a stirred solution of 1,2-dicarbonyl compound **1** (0.20 mmol) and *α*,*β*-unsaturated ketone **2** (0.22 mmol) in toluene (2 mL) was dropwise added P(NMe$_2$)$_3$ (0.22 mmol, 36 mg) by means of a microsyringe. The resulting reaction mixture was then warmed up to room temperature by removing the cooling bath and stirred at room temperature for 36 h. TLC monitoring indicated that **1** was completely consumed. The solvent was then removed on a rotary evaporator under reduced pressure, and the residue was subjected to column chromatographic isolation on silica gel by gradient elution with petroleum ether (60～90 ℃)/ethyl acetate (*V*∶*V*=20∶1～10∶1) to give products **3** and **4**.

4-Ethyl 2-methyl 2,5-diphenyl-2,3-dihydrofuran-2,4-dicarboxylate (**3aa**): Following the general procedure, **1a** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3aa** (50 mg, 71% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.94 (dd, *J*=7.7, 1.8 Hz, 2H), 7.58 (d, *J*=7.1 Hz, 2H), 7.48～7.32 (m, 6H), 4.14 (dq, *J*=7.1, 3.2 Hz, 2H), 4.03 (d, *J*=15.6 Hz, 1H), 3.78 (s, 3H), 3.50 (d, *J*=15.6 Hz, 1H), 1.22 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 172.4, 164.6, 163.4, 139.7, 130.8, 129.7, 129.4, 128.7, 128.6, 127.8, 125.3, 102.4, 88.8, 60.1, 53.3, 43.0, 14.3. HRMS-ESI calcd for $C_{21}H_{21}O_5$ $[M+H]^+$ 353.1384, found 353.1383.

4-Ethyl 2-methyl 2-phenyl-5-(*o*-tolyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ab**): Following the general procedure, **1a** (0.2 mmol) and **2b** (0.22 mmol) were employed to give product **3ab** (43 mg, 59% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.56 (d, *J*=7.0 Hz, 2H), 7.41～7.30 (m, 5H), 7.23 (d, *J*=8.3 Hz, 2H), 4.07～3.95 (m, 3H), 3.77 (s, 3H), 3.50 (d, *J*=15.5 Hz, 1H), 2.34 (s, 3H), 1.05 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (101 MHz, CDCl3) *δ*: 172.2, 164.8, 164.3, 139.9, 137.2, 130.1, 130.0, 129.9, 129.6, 128.7, 128.6, 125.3, 125.3, 104.5, 89.73, 59.9, 53.3, 41.9, 19.7, 14.1. HRMS-ESI calcd for $C_{22}H_{23}O_5$ $[M+H]^+$ 367.1540, found 367.1543.

4-Ethyl 2-methyl 2-phenyl-5-(*m*-tolyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ac**): Following the general procedure, **1a** (0.2 mmol) and **2c** (0.22 mmol) were utilized to give product **3ac** (42 mg, 57% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.57 (s, 2H), 7.42 (d, *J*=7.5 Hz, 2H), 7.25～7.09 (m, 5H), 4.05～3.93 (m, 2H), 3.87 (d, *J*=15.6 Hz, 1H), 3.62 (s, 3H), 3.34 (d, *J*=15.6 Hz, 1H), 2.26 (s, 3H), 1.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 172.4, 164.6, 163.6, 139.8, 137.4, 131.5, 130.1, 129.3, 128.7, 128.5, 127.7, 126.9, 125.3, 102.2, 88.6, 60.1, 53.3, 43.0, 21.5, 14.3. HRMS-ESI calcd for $C_{22}H_{23}O_5$ $[M+H]^+$ 367.1540, found 367.1547.

4-Ethyl 2-methyl 2-phenyl-5-(*p*-tolyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ad**): Following the general procedure, **1a** (0.2 mmol) and **2d** (0.22 mmol) were used to give product **3ad** (51 mg, 70% yield) as a white solid, m.p. 79～81 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.81 (d, *J*=8.0 Hz, 2H), 7.52 (d, *J*=7.9 Hz, 2H), 7.36～7.26 (m, 3H), 7.18 (d, *J*=8.6 Hz, 2H), 4.09 (dq, *J*=7.0, 3.4 Hz, 2H), 3.97 (d, *J*=15.6 Hz, 1H), 3.71 (s, 3H), 3.42 (d, *J*=15.6 Hz, 1H), 2.34 (s, 3H), 1.18 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 172.4, 164.6, 163.5, 141.1, 139.8, 129.6, 128.7, 128.5, 128.2, 127.5, 126.5, 125.2, 101.7, 88.6, 60.0, 53.3, 43.1, 21.7, 14.3. HRMS-ESI calcd for $C_{22}H_{23}O_5$ $[M+H]^+$ 367.1540, found 367.1545.

4-Ethyl 2-methyl 2-phenyl-5-(4-methoxyphenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ae**): Following the general procedure, **1a** (0.2 mmol) and **2e** (0.22 mmol) were employed to give product **3ae** (59 mg, 77% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.99 (d, *J*=8.9 Hz, 2H), 7.57 (d, *J*=7.0 Hz, 2H), 7.42～7.32 (m, 3H), 6.94 (d, *J*=9.0 Hz, 2H), 4.15 (dq, *J*=7.1, 3.2 Hz, 2H), 4.01 (d, *J*=15.5 Hz, 1H), 3.86 (s, 3H), 3.77 (s, 3H), 3.47 (d, *J*=15.5 Hz, 1H), 1.24 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 172.5, 164.8, 163.3, 161.6, 139.8, 131.5, 128.7, 128.5, 127.9, 127.6, 125.3, 121.7, 113.2, 88.4, 60.0, 55.4, 53.3, 43.1, 14.4. HRMS-ESI calcd for $C_{22}H_{23}O_6$ $[M+H]^+$ 383.1489, found 383.1491.

4-Ethyl 2-methyl 2-phenyl-5-(4-fluorophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3af**): Following the general procedure, **1a** (0.2 mmol) and **2f** (0.22 mmol) were employed to give product **3af** (59 mg, 80% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 8.00 (dd, *J*=8.6, 5.6 Hz, 2H), 7.56 (d, *J*=7.5 Hz, 2H), 7.43～7.32 (m, 3H), 7.11 (t, *J*=8.7 Hz, 2H), 4.15 (dq, *J*=7.1, 2.6 Hz, 2H), 4.02 (d, *J*=15.7 Hz, 1H), 3.77 (s, 3H), 3.49 (d, *J*=15.7 Hz, 1H), 1.24 (t, *J*=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 171.2, 163.4, 163.0 (d, *J*=252.5 Hz), 161.2, 138.4, 130.9 (d, *J*=

9.0 Hz), 129.0, 127.6, 124.3 (d, J=3.0 Hz), 124.2, 113.8 (d, J=22.0 Hz), 101.2, 87.7, 59.1, 52.2, 41.8, 13.1. HRMS-ESI calcd for $C_{21}H_{20}FO_5$ $[M+H]^+$ 371.1289, found 371.1292.

4-Ethyl 2-methyl 2-phenyl-5-(4-chlorophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ag**): Following the general procedure, **1a** (0.2 mmol) and **2g** (0.22 mmol) were employed to give product **3ag** (50 mg, 65% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (d, J=8.5 Hz, 2H), 7.56 (d, J=7.6 Hz, 2H), 7.44～7.31 (m, 5H), 4.15 (dq, J=7.1, 2.5 Hz, 2H), 4.02 (d, J=15.8 Hz, 1H), 3.77 (s, 3H), 3.50 (d, J=15.8 Hz, 1H), 1.24 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.3, 164.4, 162.1, 139.5, 136.8, 131.1, 128.8, 128.7, 128.1, 127.8, 125.2, 102.9, 88.7, 60.3, 53.4, 43.0, 14.3. HRMS-ESI calcd for $C_{21}H_{20}ClO_5$ $[M+H]^+$ 387.0994, found 387.0997.

4-Ethyl 2-methyl 2-phenyl-5-(4-bromophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ah**): Following the general procedure, **1a** (0.2 mmol) and **2h** (0.22 mmol) were employed to give product **3ah** (62 mg, 72% yield) as a white solid, m.p. 75～77 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, J=8.4 Hz, 2H), 7.59～7.59 (m, 4H), 7.33～7.43 (m, 3H), 4.15 (dq, J=7.0, 2.4 Hz, 2H), 4.02 (d, J=15.8 Hz, 1H), 3.77 (s, 3H), 3.49 (d, J=15.8 Hz, 1H), 1.24 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.2, 164.3, 162.0, 139.4, 131.2, 131.0, 128.7, 128.6, 128.1, 125.2, 125.1, 102.9, 88.8, 60.2, 53.3, 42.9, 14.3. HRMS-ESI calcd for $C_{21}H_{20}BrO_5$ $[M+H]^+$ 431.0489, found 431.0485.

4-Ethyl 2-methyl 2-phenyl-5-(4-(trifluoromethyl)phenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ai**): Following the general procedure, **1a** (0.2 mmol) and **2i** (0.22 mmol) were employed to give product **3ai** (49 mg, 58% yield) as a white solid, m.p. 78～80 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.08 (d, J=8.2 Hz, 2H), 7.69 (d, J=8.3 Hz, 2H), 7.57 (d, J=7.0 Hz, 2H), 7.46～7.33 (m, 3H), 4.16 (dq, J=7.1, 2.1 Hz, 2H), 4.05 (d, J=15.9 Hz, 1H), 3.79 (s, 3H), 3.54 (d, J=15.9 Hz, 1H), 1.24 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.2, 164.2, 161.6, 139.4, 132.8, 132.5, 130.1, 128.9, 128.7, 127.6, 125.2, 124.8 (q, J=28.1 Hz), 104.1, 89.2, 60.4, 53.4, 42.9, 14.3. HRMS-ESI calcd for $C_{22}H_{20}F_3O_5$ $[M+H]^+$ 421.1257, found 421.1257.

4-Ethyl 2-methyl 2-phenyl-5-(4-nitrophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3aj**): Following the general procedure, **1a** (0.2 mmol) and **2j** (0.3 mmol) were employed to give product **3aj** (46 mg, 58% yield) as a white solid, m.p. 108～110 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.28 (d, J=9.0 Hz, 2H), 8.15 (d, J=8.9 Hz, 2H), 7.59～7.52 (m, 2H), 7.45～7.36 (m, 3H), 4.17 (dq, J=7.1, 1.7 Hz, 2H), 4.05 (d, J=16.1 Hz, 1H), 3.79 (s, 3H), 3.56 (d, J=16.1 Hz, 1H), 1.25 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.0, 164.0, 160.5, 148.8, 139.1, 135.3, 130.8, 128.8, 125.2, 123.0, 105.4, 89.3, 60.6, 53.5, 42.9, 14.3. HRMS-ESI calcd for $C_{21}H_{20}NO_7$ $[M+H]^+$ 398.1234, found 398.1232.

4-Ethyl 2-methyl 2-phenyl-5-(3,4,5-trimethoxylphenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ak**): Following the general procedure, **1a** (0.2 mmol) and **2k** (0.22 mmol) were employed to give product **3ak** (47 mg, 63% yield) as a white solid, m.p. 107～110 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, J=7.0 Hz, 2H), 7.43～7.34 (m, 5H), 4.16 (q, J=7.1 Hz, 2H), 4.02 (d, J=15.7 Hz, 1H), 3.92 (s, 6H), 3.90 (s, 3H), 3.77 (s, 3H), 3.49 (d, J=15.7 Hz, 1H), 1.25 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.4, 164.6, 162.8, 152.6, 140.4, 139.6, 128.7, 128.6, 125.3, 124.4, 107.4, 102.1, 88.5, 61.0, 60.2, 56.4, 53.3, 43.3, 14.4. HRMS-ESI calcd for $C_{24}H_{27}O_8$ $[M+H]^+$ 443.1700, found 443.1708.

4-Ethyl 2-methyl 5-(naphthalen-2-yl)-2-phenyl-2,3-dihydrofuran-2,4-dicarboxylate (**3al**): Following the general procedure, **1a** (0.2 mmol) and **2l** (0.22 mmol) were employed to give product **3al** (48 mg, 60% yield) as a white solid, m.p. 122～124 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.52 (s, 1H), 8.02 (d, J=8.6 Hz, 1H), 7.95 (d, J=7.6 Hz, 1H), 7.92～7.84 (m, 2H), 7.63 (d, J=7.4 Hz, 2H), 7.60～7.50 (m, 2H), 7.45～7.34 (m, 3H), 4.23～4.13 (m, 2H), 4.10 (d, J=15.7 Hz, 1H), 3.81 (s, 3H), 3.57 (d, J=15.7 Hz, 1H), 1.23 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.4, 164.6, 163.3, 139.7, 134.4, 132.5, 130.3, 129.0, 128.7, 128.6, 127.7, 127.5, 127.3, 126.7, 126.4, 126.3, 125.3, 102.7, 88.8, 60.2, 53.4, 43.2, 14.3. HRMS-ESI calcd for $C_{25}H_{23}O_5$ $[M+H]^+$ 403.1540, found 403.1540.

4-Ethyl 2-methyl 2-phenyl-5-(thiophen-2-yl)-2,3-dihydrofuran-2,4-dicarboxylate (**3am**): Following the general procedure, **1a** (0.2 mmol) and **2m** (0.22 mmol) were employed to give product **3am** (42 mg, 59% yield) as a pale yellow solid, m.p. 29～31 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.31 (d, J=3.4 Hz, 1H), 7.57 (t, J=6.6 Hz, 3H), 7.38 (dt, J=14.9, 7.0 Hz, 3H), 7.18～7.12 (m, 1H), 4.23 (q, J=7.0 Hz, 2H), 4.05 (d, J=15.9 Hz, 1H), 3.76 (s, 3H), 3.46 (d, J=15.9 Hz, 1H), 1.31 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.1, 164.6, 157.0, 139.7, 132.7, 130.7, 128.7, 128.6, 127.3, 125.1, 100.4, 88.6, 60.2, 53.3, 43.1, 14.5. HRMS-ESI calcd for $C_{19}H_{19}O_5S$ $[M+H]^+$ 359.0948, found 359.0949.

4-Ethyl 2-methyl 5-methyl-2-phenyl-2,3-dihydrofuran-2,4-dicarboxylate (**3an**): Following the general procedure, **1a** (0.2 mmol) and **2n** (0.22 mmol) were employed to give product **3an** (13 mg, 22% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (d, J=7.2 Hz, 2H), 7.41～7.32 (m , 3H), 4.16 (q, J=7.1 Hz, 2H), 3.79 (d, J=15.1 Hz, 1H), 3.75 (d, J=0.8 Hz, 3H), 3.25 (d, J=15.1 Hz, 1H), 2.35 (s, 3H), 1.27 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.2, 166.2, 165.4, 140.0, 128.7, 128.5, 125.1, 102.1, 89.4, 59.7, 53.3, 41.5, 14.5, 14.2. HRMS-ESI calcd for $C_{16}H_{19}O_5$ $[M+H]^+$ 291.1227, found 291.1224.

4-Ethyl 2-methyl 5-cyclohexyl-2-phenyl-2,3-dihydrofuran-2,4-dicarboxylate (**3ao**): Following the general procedure, **1a** (0.2 mmol) and **2o** (0.22 mmol) were employed to give product **3ao** (55 mg, 77% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (d, J=7.0 Hz, 2H), 7.41～7.29 (m, 3H), 4.14 (q, J=7.1 Hz, 2H), 3.77 (d, J=15.0 Hz, 1H), 3.73 (s, 3H), 3.37 (tt, J=11.8, 2.9 Hz, 1H), 3.20 (d, J=15.0 Hz, 1H), 1.86～1.76 (m, 4H), 1.75～1.61 (m, 3H), 1.41～1.29 (m, 3H), 1.26 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 173.6, 172.4, 165.3, 140.2, 128.6, 128.4, 125.1, 99.8, 89.0, 59.7, 53.1, 41.6, 36.9, 29.6, 29.5, 26.0,

25.9, 14.5. HRMS-ESI calcd for $C_{21}H_{27}O_5$ $[M+H]^+$ 359.1853, found 359.1856.

4-Ethyl 2-methyl 5-pentyl-2-phenyl-2,3-dihydrofuran-2,4-dicarboxylate (**3ap**): Following the general procedure, **1a** (0.2 mmol) and **2p** (0.22 mmol) were employed to give product **3ap** (41 mg, 59% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (d, J=7.2 Hz, 2H), 7.40～7.30 (m, 3H), 4.15 (q, J=7.1 Hz, 2H), 3.80 (d, J=15.1 Hz, 1H), 3.74 (s, 3H), 3.24 (d, J=15.1 Hz, 1H), 2.76 (t, J=7.5 Hz, 2H), 1.41～1.22 (m, 9H), 0.90 (t, J=5.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.3, 170.0, 165.3, 140.1, 128.6, 128.5, 125.1, 101.6, 89.2, 59.8, 53.2, 41.6, 31.5, 27.7, 26.6, 22.5, 14.5, 14.1. HRMS-ESI calcd for $C_{20}H_{27}O_5$ $[M+H]^+$ 347.1853, found 347.1848.

4-Ethyl 2-methyl 5-phenyl-2-(2-chlorophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ba**): Following the general procedure, **1b** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ba** (27 mg, 35% yield) as a white solid, m.p. 97～98 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (dd, J=7.8, 2.0 Hz, 2H), 7.70 (dd, J=7.3, 2.2 Hz, 1H), 7.50～7.42 (m, 4H), 7.34 (ddd, J=6.9, 4.6, 2.0 Hz, 2H), 4.46 (d, J=16.1 Hz, 1H), 4.22～4.11 (m, 2H), 3.81 (s, 3H), 3.28 (d, J=16.1 Hz, 1H), 1.25 (t, J=7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.1, 164.4, 161.6, 139.1, 130.8, 130.7, 130.4, 129.5, 129.5, 127.8, 127.0, 126.2, 103.4, 87.9, 60.1, 53.5, 42.1, 14.2. HRMS-ESI calcd for $C_{21}H_{19}ClO_5Na$ $[M+Na]^+$ 409.0921, found 409.0817.

4-Ethyl 2-methyl 5-phenyl-2-(2-bromophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ca**): Following the general procedure, **1c** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ca** (40 mg, 46% yield) as a white solid, m.p. 105～106 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (dd, J=7.6, 2.2 Hz, 2H), 7.66 (dd, J=7.8, 1.7 Hz, 1H), 7.59 (dd, J=7.9, 1.3 Hz, 1H), 7.43 (d, J=6.3 Hz, 3H), 7.35 (td, J=7.6, 1.2 Hz, 1H), 7.20 (td, J=7.6, 1.7 Hz, 1H), 4.48 (d, J=16.1 Hz, 1H), 4.13 (qd, J=7.1, 1.7 Hz, 2H), 3.76 (s, 3H), 3.25 (d, J=16.1 Hz, 1H), 1.20 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 169.0, 164.4, 161.6, 140.7, 133.8, 130.8, 129.8, 129.5, 129.0, 127.9, 127.5, 126.5, 120.2, 103.4, 89.0, 60.2, 53.6, 42.5, 14.3. HRMS-ESI calcd for $C_{21}H_{19}BrO_5Na$ $[M+Na]^+$ 453.0416, found 453.0312.

4-Ethyl 2-methyl 5-phenyl-2-(3-chlorophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3da**): Following the general procedure, **1d** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3da** (38 mg, 49% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 (dd, J=7.5, 2.2 Hz, 2H), 7.65～7.61 (m, 1H), 7.52～7.44 (m, 4H), 7.37～7.31 (m, 2H), 4.18 (qd, J=7.1, 3.4 Hz, 2H), 4.08 (d, J=15.6 Hz, 1H), 3.80 (s, 3H), 3.51 (d, J=15.7 Hz, 1H), 1.26 (t, J=7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.8, 164.3, 163.1, 141.6, 134.7, 130.9, 130.0, 129.7, 129.1, 128.7, 127.8, 125.6, 123.5, 102.4, 88.1, 60.2, 53.4, 43.1, 14.2. HRMS-ESI calcd for $C_{21}H_{19}ClO_5Na$ $[M+Na]^+$ 409.0921, found 409.0816.

4-Ethyl 2-methyl 5-phenyl-2-(3-bromophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ea**): Following the general procedure, **1e** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ea** (37 mg, 43% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94 (dd, J=7.7, 1.9 Hz, 2H), 7.74 (t, J=1.9 Hz, 1H), 7.54～7.39 (m, 5H), 7.29～7.20 (m, 1H), 4.14 (qq, J=7.1, 3.7 Hz, 2H), 4.02 (d, J=15.7 Hz, 1H), 3.78 (s, 3H), 3.46 (d, J=15.6 Hz, 1H), 1.22 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.9, 164.3, 163.1, 141.8, 131.7, 130.9, 130.3, 129.6, 129.1, 128.4, 127.8, 124.0, 122.8, 102.3, 88.0, 60.2, 53.5, 43.1, 14.2. HRMS-ESI calcd for $C_{21}H_{19}BrO_5Na$ $[M+Na]^+$ 453.0416, found 453.0311.

4-Ethyl 2-methyl 5-phenyl-2-(3-methylphenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3fa**): Following the general procedure, **1f** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3fa** (32 mg, 44% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.76 (dd, J=7.6, 2.2 Hz, 2H), 7.26～7.20 (m, 3H), 7.20～7.16 (m, 2H), 7.10～7.05 (m, 1H), 6.95 (d, J=7.8 Hz, 1H), 3.95 (qq, J=7.1, 3.7 Hz, 2H), 3.85 (d, J=15.6 Hz, 1H), 3.56 (d, J=1.3 Hz, 3H), 3.31 (d, J=15.6 Hz, 1H), 2.17 (s, 3H), 1.02 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.5, 164.5, 163.4, 139.6, 138.4, 130.7, 129.7, 129.4, 129.3, 128.6, 127.8, 125.8, 122.3, 102.4, 88.8, 60.1, 53.2, 42.9, 21.6, 14.3. HRMS-ESI calcd for $C_{22}H_{22}O_5Na$ $[M+Na]^+$ 389.1467, found 389.1363.

4-Ethyl 2-methyl 5-phenyl-2-(4-chlorophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ga**): Following the general procedure, **1g** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ga** (36 mg, 46% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 (d, J=7.8 Hz, 2H), 7.52 (d, J=8.5 Hz, 2H), 7.44 (d, J=7.8 Hz, 3H), 7.36 (d, J=8.5 Hz, 2H), 4.14 (ddt, J=10.5, 7.1, 3.4 Hz, 2H), 4.01 (d, J=15.6 Hz, 1H), 3.77 (s, 3H), 3.45 (d, J=15.6 Hz, 1H), 1.22 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.0, 164.3, 163.2, 138.2, 134.6, 130.8, 129.6, 129.1, 128.8, 127.8, 126.8, 102.3, 88.2, 60.1, 53.4, 43.0, 14.2. HRMS-ESI calcd for $C_{21}H_{19}ClO_5Na$ $[M+Na]^+$ 409.0921, found 409.0817.

4-Ethyl 2-methyl 5-phenyl-2-(4-bromophenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ha**): Following the general procedure, **1h** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ha** (27 mg, 31% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.97 (dd, J=7.9, 1.9 Hz, 2H), 7.55 (d, J=8.7 Hz, 2H), 7.48 (t, J=8.0 Hz, 5H), 4.18 (dq, J=7.2, 3.8 Hz, 2H), 4.05 (d, J=15.6 Hz, 1H), 3.80 (s, 3H), 3.48 (d, J=15.7 Hz, 1H), 1.25 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 171.9, 164.3, 163.2, 138.7, 131.8, 130.8, 129.6, 129.1, 127.8, 127.1, 122.8, 102.3, 88.2, 60.1, 53.4, 43.0, 14.2. HRMS-ESI calcd for $C_{21}H_{19}BrO_5Na$ $[M+Na]^+$ 453.0416, found 453.0312.

2,4-Diethyl 5-phenyl-2-(4-methylphenyl)-2,3-dihydrofuran-2,4-dicarboxylate (**3ia**): Following the general procedure, **1i** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ia** (13 mg, 18% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.99～7.90 (m, 2H), 7.46 (d, J=8.3 Hz, 2H), 7.44～7.37 (m, 3H), 7.18 (d, J=8.0 Hz, 2H), 4.20 (q, J=7.1 Hz, 2H), 4.13 (tq, J=7.2, 3.5 Hz, 2H), 4.00 (d, J=15.6 Hz, 1H), 3.48 (d, J=15.6 Hz, 1H), 2.32 (s,

3H), 1.22 (dt, J=10.7, 7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 172.0, 164.6, 163.5, 138.3, 136.8, 130.7, 129.7, 129.5, 129.3, 127.7, 125.2, 102.3, 88.8, 62.2, 60.0, 42.8, 21.1, 14.3, 14.1. HRMS-ESI calcd for $C_{23}H_{24}O_5Na$ [M+Na]$^+$ 403.1624, found 403.1518.

Ethyl 5-benzoyl-2,5-diphenyl-4,5-dihydrofuran-3-carboxylate (**3ja**): Following the general procedure, **1j** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ja** (36 mg, 45% yield) as a pale yellow solid, m.p. 62～65 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (dd, J=8.4, 1.2 Hz, 2H), 7.77 (dt, J=6.9, 1.4 Hz, 2H), 7.54～7.45 (m, 3H), 7.43～7.28 (m, 8H), 4.44 (d, J=16.0 Hz, 1H), 4.18～4.10 (m, 2H), 3.24 (d, J=16.0 Hz, 1H), 1.22 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 197.0, 164.7, 162.3, 141.2, 134.3, 133.1, 130.7, 130.7, 129.5, 129.30, 129.2, 128.2, 128.2, 127.8, 124.2, 102.8, 94.0, 60.1, 43.6, 14.3. HRMS-ESI calcd for $C_{26}H_{23}O_4$ [M + H]$^+$ 399.1591, found 399.1583.

Triethyl 5-phenylfuran-2,2,4(3*H*)-tricarboxylate (**3ka**): Following the general procedure, **1k** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3ka** (73 mg, 96% yield) as a white solid, m.p. 77～79 ℃. ^{1}H NMR (400 MHz, CDCl3) δ: 7.85 (dd, J=8.1, 1.5 Hz, 2H), 7.47～7.35 (m, 3H), 4.32 (q, J=7.1 Hz, 4H), 4.14 (q, J=7.1 Hz, 2H), 3.67 (s, 2H), 1.32 (t, J=7.1 Hz, 6H), 1.22 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 167.4, 164.1, 163.1, 130.9, 129.7, 128.7, 127.73, 102.1, 86.4, 62.8, 60.3, 38.9, 14.1. HRMS-ESI calcd for $C_{19}H_{23}O_7$ [M+H]$^+$ 363.1438, found 363.1439.

Diethyl 4-methyl-5-phenylfuran-2,2(3*H*)-dicarboxylate (**3kq**): Following the general procedure, **1k** (0.2 mmol) and **2q** (0.22 mmol) were employed to give product **3kq** (47 mg, 77% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, J=7.2 Hz, 2H), 7.36 (t, J=7.4 Hz, 2H), 7.29 (t, J=7.3 Hz, 1H), 4.30 (q, J=7.1 Hz, 4H), 3.36 (s, 2H), 1.89 (s, 3H), 1.31 (t, J=7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 168.8, 147.2, 130.6, 128.1, 127.3, 104.6, 85.0, 62.3, 44.2, 14.0, 12.2. HRMS-ESI calcd for $C_{17}H_{21}O_5$ [M+H]$^+$ 305.1384, found 305.1386.

Diethyl 4,5-diphenylfuran-2,2(3*H*)-dicarboxylate (**3kr**): Following the general procedure, **1k** (0.2 mmol) and **2r** (0.22 mmol) were employed to give product **3kr** (36 mg, 49% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (dt, J=7.5, 2.5 Hz, 2H), 7.35～7.26 (m, 4H), 7.24～7.17 (m, 4H), 4.33 (q, J=7.1 Hz, 4H), 3.76 (s, 2H), 1.33 (t, J=7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 168.4, 149.0, 134.1, 130.5, 129.2, 128.5, 128.4, 128.3, 127.3, 126.7, 109.1, 85.4, 62.6, 42.6, 14.1. HRMS-ESI calcd for $C_{22}H_{23}O_5$ [M+H]$^+$ 367.1540, found 367.1547.

Ethyl 10'-*oxo*-5-phenyl-3*H*,10'*H*-spiro[furan-2,9'-phenanthrene]-4-carboxylate (**3la**): Following the general procedure, **1l** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **3la** (27 mg, 34% yield) as a pale yellow solid, m.p. 128～131 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.09～8.05 (m, 3H), 7.97 (d, J=8.0 Hz, 1H), 7.92 (dd, J=7.7, 1.2 Hz, 1H), 7.72 (td, J=7.7, 1.5 Hz, 1H), 7.64 (dd, J=7.6, 1.5 Hz, 1H), 7.51～7.38 (m, 6H), 4.08 (dq, J=7.1, 2.4 Hz, 2H), 3.58 (d, J=15.8 Hz, 1H), 3.17 (d, J=15.8 Hz, 1H), 1.15 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 195.5, 165.6, 164.4, 138.9, 137.2, 135.1, 130.8, 129.8, 129.6, 129.4, 129.0, 128.8, 128.5, 128.1, 127.7, 125.3, 124.4, 123.4, 100.3, 88.4, 60.0, 45.1, 14.2. HRMS-ESI calcd for $C_{26}H_{21}O_4$ [M+H]$^+$ 397.1434, found 397.1437.

Diethyl 4,5,6,7-tetrahydrobenzofuran-2,2(3*H*)-dicarboxylate (**3ks**): Following the general procedure, **1k** (0.2 mmol) and **2s** (0.22 mmol) were employed to give product **3ks** (37 mg, 68% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.27 (q, J=8.3 Hz, 4H), 3.10 (s, 2H), 2.16～2.09 (m, 2H), 2.0～1.94 (m, 2H), 1.72～1.65 (m, 2H), 1.64～1.56 (m, 2H), 1.28 (t, J=7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 168.8, 150.2, 105.0, 86.3, 62.3, 40.5, 23.1, 22.7, 22.7, 22.6, 14.1. HRMS-ESI calcd for $C_{14}H_{21}O_5$ [M+H]$^+$ 269.1384; found 269.1387.

Ethyl 1'-methyl-2'-*oxo*-5-phenyl-3H-spiro[furan-2,3'-indoline]-4-carboxylate (**3na**): Following the general procedure, **1n** (0.2 mmol) and **2a** (0.22 mmol) were employed to give products **3na** (27 mg, 38% yield) as a pale white solid, m.p. 43～45 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83 (dt, J=6.7, 1.5 Hz, 2H), 7.48 (d, J=7.4 Hz, 1H), 7.43～7.34 (m, 4H), 7.11 (td, J=7.6, 0.9 Hz, 1H), 6.86 (d, J=7.8 Hz, 1H), 4.17 (dq, J=7.1, 1.8 Hz, 2H), 3.67 (d, J=15.6 Hz, 1H), 3.38 (d, J=15.6 Hz, 1H), 3.23 (s, 3H), 1.23 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 174.4, 164.4, 164.3, 143.6, 130.7, 130.7, 129.7, 129.5, 129.2, 127.7, 124.0, 123.7, 108.7, 102.5, 83.7, 60.2, 40.8, 26.5, 14.3. HRMS-ESI calcd for $C_{21}H_{20}NO_4$ [M + H]$^+$ 350.1387, found 350.1387.

Ethyl 2-benzoyl-1'-methyl-2'-oxospiro[cyclopropane-1,3'-indoline]-2-carboxylate (**4na**): Following the general procedure, **1n** (0.2 mmol) and **2a** (0.22 mmol) were employed to give product **4na** (39 mg, 45% yield, *dr* 19∶1) as a white solid, m.p. 136～138 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (d, J=7.2 Hz, 2H), 7.54～7.45 (m, 2H), 7.41～7.32 (m, 3H), 7.07 (td, J=7.7, 0.9 Hz, 1H), 6.92 (d, J=7.8 Hz, 1H), 4.15～4.07 (m, 2H), 3.09 (s, 3H), 2.72 (d, J=5.0 Hz, 1H), 2.50 (d, J=5.0 Hz, 1H), 1.01 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 190.5, 172.5, 166.6, 144.9, 136.4, 133.2, 128.8, 128.7, 128.4, 124.5, 123.5, 122.4, 108.4, 62.5, 47.9, 39.0, 26.7, 24.8, 14.0. HRMS-ESI calcd for $C_{21}H_{20}NO_4$ [M+H]$^+$ 350.1387, found 350.1389.

Methyl 2-benzoyl-2-methyl-1-phenylcyclopropane-1-carboxylate (**4aq**): Following the general procedure, **1a** (0.2 mmol) and **2q** (0.22 mmol) were employed to give product **4aq** (42 mg, 69% yield, *dr* 2.2∶1). For the major isomer: 29 mg, 48% yield, as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.10～8.06 (m, 2H), 7.60～7.55 (m, 1H), 7.54～7.46 (m, 4H), 7.44～7.38 (m, 2H), 7.38～7.32 (m, 1H), 3.43 (s, 3H), 2.46 (d, J=4.9 Hz, 1H), 1.68 (d, J=4.9 Hz, 1H), 1.18 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 199.0, 172.3, 135.8, 135.4, 133.0, 131.1, 128.7, 128.6, 128.4, 127.7, 52.5, 39.9, 39.4, 25.9, 20.9; For the minor isomer: 13 mg, 21% yield, as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82～7.77 (m, 2H), 7.51～7.46 (m, 1H), 7.40 (ddd, J=8.0, 4.0, 2.1 Hz, 4H), 7.17～7.09 (m, 3H), 3.80 (s, 3H), 2.49 (d,

J=5.3 Hz, 1H), 1.77 (d, J=5.3 Hz, 1H), 1.62 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 198.8, 170.7, 136.8, 134.2, 132.4, 130.3, 129.2, 128.3, 128.0, 127.7, 52.8, 42.9, 37.9, 22.0, 20.0. HRMS-ESI calcd for $C_{19}H_{19}O_3$ $[M+H]^+$ 295.1329, found 295.1330.

Ethyl 2-benzoyl-2-methyl-1-phenylcyclopropane-1-carboxylate (**4mq**): Following the general procedure, **1m** (0.2 mmol) and **2q** (0.22 mmol) were employed to give product **4mq** (35 mg, 57% yield, *dr* 1∶1). For the major isomer: 18 mg, 29% yield, as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86～7.77 (m, 2H), 7.48 (t, J=7.4 Hz, 1H), 7.45～7.37 (m, 4H), 7.16～7.08 (m, 3H), 4.34～4.18 (m, 2H), 2.49 (d, J=5.3 Hz, 1H), 1.75 (d, J=5.3 Hz, 1H), 1.63 (s, 3H), 1.33 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 198.8, 170.1, 136.8, 134.3, 132.4, 130.2, 129.2, 128.2, 127.9, 127.6, 61.7, 43.0, 37.7, 21.6, 20.1, 14.4. For the minor isomer: 17 mg, 28% yield, as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15～8.05 (m, 2H), 7.57 (t, J=7.3 Hz, 1H), 7.51 (dd, J=7.3, 5.6 Hz, 4H), 7.41 (t, J=7.4 Hz, 2H), 7.34 (t, J=7.2 Hz, 1H), 3.94 (dq, J=10.8, 7.1 Hz, 1H), 3.80 (dq, J=10.8, 7.1 Hz, 1H), 2.47 (d, J=4.9 Hz, 1H), 1.65 (d, J=4.9 Hz, 1H), 1.17 (s, 3H), 0.96 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 198.8, 171.7, 135.8, 135.6, 132.9, 131.1, 128.9, 128.6, 128.3, 127.6, 61.4, 40.1, 39.3, 25.4, 20.8, 13.8. HRMS-ESI calcd for $C_{20}H_{21}O_3$ $[M+H]^+$ 309.1485; found 309.1490.

Methyl 2-benzoyl-1,2-diphenylcyclopropane-1-carboxylate (**4ar**): Following the general procedure, **1a** (0.2 mmol) and **2r** (0.22 mmol) were employed to give product **4ar** (35 mg, 49% yield, *dr* 3.4∶1). For the major isomer: 27 mg, 38% yield, as a white solid. m.p. 149～151 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67～7.60 (m, 2H), 7.57～7.51 (m, 2H), 7.44～7.40 (m, 2H), 7.37～7.31 (m, 3H), 7.28 (d, J=8.3 Hz, 3H), 7.26～7.20 (m, 3H), 3.33 (s, 3H), 2.82 (d, J=5.5 Hz, 1H), 2.51 (d, J=5.5 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 197.1, 169.8, 137.6, 136.3, 134.4, 131.9, 131.8, 130.4, 128.6, 128.4, 127.9, 127.8, 127.8, 52.5, 47.3, 42.8, 22.2; For the minor isomer: 8 mg, 11% yield, as a white solid. m.p. 94～97 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02～7.98 (m, 2H), 7.51～7.46 (m, 1H), 7.38 (t, J=7.6 Hz, 2H), 7.24～7.19 (m, 4H), 7.12～7.01 (m, 6H), 3.66 (s, 3H), 2.71 (d, J=5.7 Hz, 1H), 2.67 (d, J=5.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 195.8, 172.2, 135.8, 134.6, 133.7, 132.9, 131.5, 129.6, 128.5, 128.4, 128.3, 127.5, 127.4, 127.1, 52.8, 48.3, 41.3, 22.3. HRMS-ESI calcd for $C_{24}H_{21}O_3$ $[M+H]^+$ 357.1485, found 357.1479.

Methyl 2-benzoyl-1-phenylcyclopropane-1-carboxylate (**4at**): Following the general procedure, **1a** (0.2 mmol) and **2t** (0.22 mmol) were employed to give an inseparable diastereomeric mixture **4at** (17 mg, 30% yield, *dr* 1∶1) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, J=7.3 Hz, 2H), 7.97 (d, J=7.3 Hz, 2H), 7.60 (q, J=7.2 Hz, 2H), 7.56～7.45 (m, 6H), 7.43～7.31 (m, 3H), 7.22～7. 17 (m, 3H), 7.11 (dd, J=6.4, 3.0 Hz, 2H), 3.80～3.75 (m, 1H), 3.72 (s, 3H), 3.61 (s, 3H), 3.15 (dd, J=8.3, 6.4 Hz, 1H), 2.41～2.37 (m, 2H), 1.94 (dd, J=8.2, 4.0 Hz, 1H), 1.74 (dd, J=8.4, 4.6 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 196.3, 194.1, 173.3, 170.3, 138.5, 138.0, 137.7, 133.8, 133.3, 130.8, 129.1, 128.8, 128.8, 128.4, 128.3, 128.1, 127.8, 53.2, 52.7, 41.6, 40.3, 32.7, 32.2, 20.4, 19.8; HRMS-ESI calcd for $C_{18}H_{17}O_3$ $[M+H]^+$ 281.1172, found 281.1173.

Diethyl 2-benzoylcyclopropane-1,1-dicarboxylate (**4kt**): Following the general procedure, **1k** (0.2 mmol) and **2t** (0.22 mmol) were employed to give product **4kt** (9 mg, 16% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 (d, J=7.9 Hz, 2H), 7.59 (t, J=7.1 Hz, 1H), 7.49 (t, J=7.4 Hz, 2H), 4.30～4.21 (m, 2H), 4.13 (q, J=6.9 Hz, 2H), 3.54 (t, J=7.6 Hz, 1H), 2.26～2.20 (m, 1H), 1.75 (dd, J=8.3, 4.0 Hz, 1H), 1.30 (t, J=7.0 Hz, 3H), 1.13 (t, J=7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 194.7, 169.2, 166.0, 137.2, 133.6, 128.8, 128.5, 62.5, 61.8, 39.4, 31.1, 20.6, 14.1,13.9; HRMS-ESI calcd for $C_{16}H_{19}O_5$ $[M+H]^+$ 291.1227, found 291.1229.

Supporting Information Copies of ^{1}H NMR and ^{13}C NMR spectra of new compounds **3** and **4**; X-ray crystallographic data for compounds **3ad** (CCDC: 2084148) and **4na** (CCDC: 2084150). The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn/.

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