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Regioselective mono-aza-Michael additions of divinyl ketones with 3-(arylimino)indolin-2-ones: Synthesis of N-enone-functionalized 3-(arylimino)indolin-2-ones

Xiao Chen and Zheng Li

Abstract

Selective mono-aza-Michael additions of divinyl ketones with 3-(arylimino)indolin-2-ones in the presence of cesium carbonate are described. *N*-Enone-functionalized 3-(arylimino)indolin-2-ones were efficiently synthesized in satisfactory yield. The salient features of this protocol are high regioselectivity, high yield, and mild conditions.

Keywords

3-(arylimino)indolin-2-one, divinyl ketone, high regioselectivity, mono-aza-Michael addition, synthesis

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Introduction

The indolin-2-one framework bearing various functional groups at the *N*-position is a privileged heterocyclic motif that is found in many bioactive natural products and pharmaceutically active compounds.[1,2] One important method for the *N*-functionalization of indolin-2-one is the aza-Michael addition. However, the 3-position is more active than the 1-position for indolin-2-one making the Michael addition to C-3 more favorable.[3–5] Therefore, 3-substituted indolin-2-ones may be better candidates for aza-Michael additions. 3-(Arylimino)indolin-2-ones, synthesized by reactions of isatins with aromatic amines,[6] may act as good aza-Michael donors. For example, Imanzadeh et al.[7] reported the aza-Michael additions of 3-(arylimino)indolin-2-ones with fumaric esters; Zari[8] described the thiourea catalyzed asymmetric aza-Michael additions of 3-(arylimino)indolin-2-ones with unsaturated 1,4-diketoesters; Metsala[9] investigated these aza-Michael reactions by means of density functional theory (DFT) calculations; and Metsala[10] also performed conformational analyses across reaction paths for these aza-Michael reactions.

Divinyl ketones (e.g. 1,4-dien-3-ones) have been used as Michael acceptors in recent years to synthesize spiro-, cyano-, and other important compounds.[11–19] However, divinyl ketones as Michael acceptors have problems of regioselective control and can undergo 1,2-addition and two kinds of 1,4-addition reactions.

Herein, we report the regioselective mono-aza-Michael additions of divinyl ketones with 3-(arylimino)indolin-2-ones under mild, transition metal-free and acid-free conditions to synthesize *N*-enone-functionalized 3-(arylimino)indolin-2-ones bearing both C=C and C=N functional groups.

Results and discussion

In previous work, the reactions of 3-(arylimino)indolin-2-ones with enones had been reported, which could give the aza-Michael addition products in satisfactory yields.[20] In order to compare enones and divinyl ketones, the aza-Michael additions of 3-(arylimino)indolin-2-ones with divinyl ketones were investigated. Initially, (1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one (**1a**) was selected as a substrate and reacted with 1 equiv. of (*E*)-3-(phenylimino)indolin-2-one (**2a**), which was synthesized by reaction of isatin with aniline, and isolated from the *E*-*Z* mixture (ca. 87:13). The reaction was attempted using a variety of bases at different temperatures in various solvents. When the reaction was carried out using KOH as the base in CH_2Cl_2, one product was isolated in 52% yield and was identified as (*E*)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one (**3aa**). This result indicated that the reaction of **1a** with 1 equiv. of **2a** proceeded by only a single 1,4-conjugate addition. No 1,2-adduct was observed. When **1a** was reacted with 2 equiv. of **2a**, the product still was **3aa**, and no double 1,4-conjugate addition product was isolated. These results implied that the reaction between **1a** and **2a** was highly regioselective for a carbon, carbon double-bond of **1a** (Scheme 1).

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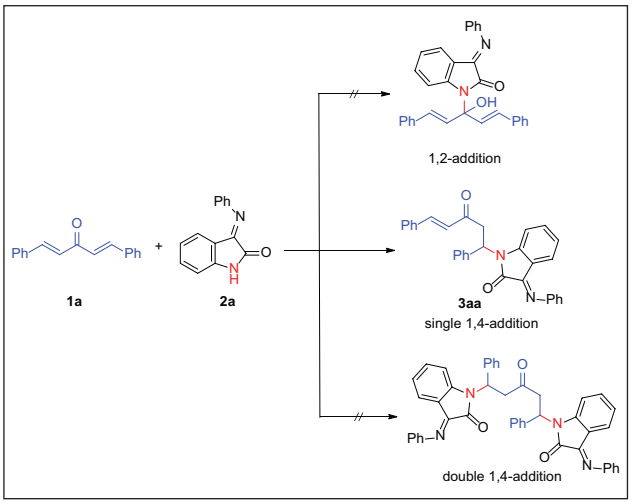
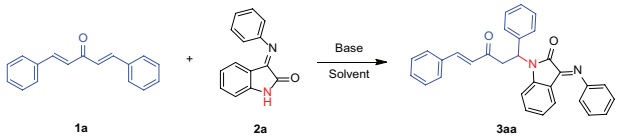


Scheme 1. Regioselective addition of **1a** with **2a**.

In order to optimize further the reaction conditions for the model reaction, other bases including inorganic bases, such as NaOH, K_2CO_3, Na_2CO_3, CsF, and Cs_2CO_3, and organic bases, such as Et_3N, 1,4-diazabicyclo[2.2.2] octane (DABCO), 4-dimethylaminopyridine (DMAP), and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), were examined for the reaction (Table 1, entries 2–5 and Supporting information, Table S1). Of these, Cs_2CO_3 as a base gave the best yield of the desired product **3aa** (entry 4). The effect of the amount of base on the yield of **3aa** was also tested (entries 4, 6–8). It was found that when the amount of Cs_2CO_3 was reduced to 0.5 equiv., the highest yield of **3aa** was obtained (entry 6). The choice of solvent also had an effect on the yield of the reaction. Dimethylformamide (DMF), dimethyl sulfoxide (DMSO), MeOH, 1,4-dioxane, and tetrahydrofuran (THF) were not suitable for the reaction (entries 14–18). Meanwhile, the reactions in MeCN and $ClCH_2CH_2Cl$ gave **3aa** in moderate yields (Supporting information, Table S1). The highest yield was obtained using CH_2Cl_2 as the solvent (entry 6). In addition, increasing the temperature from room temperature to 50 °C caused a reduction in the yield of **3aa** because of an increase in by-products (entry 9). Changing the ratio of **1a** to **2a** from 1:1 to 1:2 did not improve the yield of **3aa** (entry 10).

With the optimized reaction conditions in hand, the generality of this reaction was examined for the reactions of various divinyl ketones with 3-(arylimino)indolin-2-ones in CH_2Cl_2 at room temperature using Cs_2CO_3 as the base. The reactions were first tested using various divinyl ketones (**1a–i**) and **2a**. As shown in Table 2, divinyl ketones bearing electron-donating groups (Me, MeO) and electron-withdrawing groups (CF_3, Cl, Br) on the aromatic rings gave the corresponding mono-aza-Michael addition products in satisfactory yields (**3aa–3ha**). It was also found that *ortho*-substituted divinyl ketones (**3ba**) afforded slightly lower yield than *meta*- and *para*-substituted divinyl ketones because of the increased steric hindrance. In addition, a divinyl ketone bearing a thiophen-2-yl heterocycle also gave the corresponding product in a moderate yield (**3ia**). Aliphatic divinyl ketones did not produce the desired products.

Table 1. Optimization of the reaction conditions.[a]



Entry	Base (equiv.)	Solvent	Yield (%)[b]
1	KOH (1)	CH_2Cl_2	52
2	NaOH (1)	CH_2Cl_2	40
3	CsF (1)	CH_2Cl_2	35
4	Cs_2CO_3 (1)	CH_2Cl_2	67
5	DBU (1)	CH_2Cl_2	43
6	Cs_2CO_3 (0.5)	CH_2Cl_2	70
7	Cs_2CO_3 (1.5)	CH_2Cl_2	69
8	Cs_2CO_3 (2)	CH_2Cl_2	65
9[c]	Cs_2CO_3 (0.5)	CH_2Cl_2	Trace
10[d]	Cs_2CO_3 (0.5)	CH_2Cl_2	53

[a]Reaction conditions: **1a** (0.3 mmol), **2a** (0.36 mmol), and base (appropriate amount) in solvent (3 mL) at room temperature for 8 h.
[b]Isolated yields.
[c]Reaction condition: 50 °C.
[d]Reaction condition: **1a**:**2a** = 1:2.

The scope of the reactions of **1a** with various 3-(arylimino)indolin-2-ones (**2b–m**) was also examined (Table 3). The reactions tolerated a wide range of indolin-2-ones bearing substituted arylimino groups (R^2 = Me, MeO, F, and Cl) and afforded the corresponding products in satisfactory yields (**3ab–3ag**). 3-(Arylimino)indolin-2-ones bearing disubstituted arylimino groups also allowed the easy synthesis of the corresponding products in moderate to high yields (**3ah, 3ai**). 3-(Arylimino)indolin-2-ones bearing substituents on the indolin-2-one rings (R^3 = 5-Cl, 5-Me) also gave the corresponding products in good yield (**3aj–3am**).

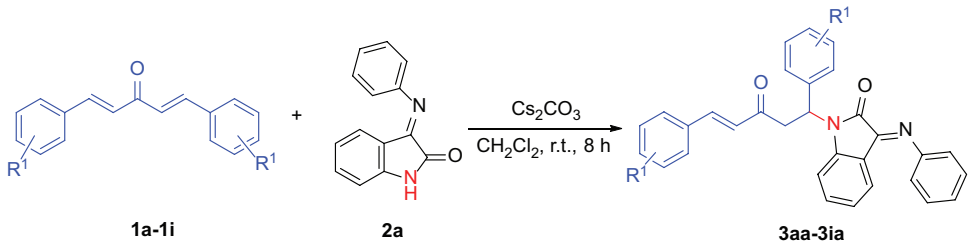
The reaction of **1a** with **2a** was also extended to the gram scale (Scheme 2). The reaction of **1a** (1.17 g) and **2a** (1.33 g) in CH_2Cl_2 (40 mL) in the presence of Cs_2CO_3 (0.41 g) was performed under the optimized conditions to give **3aa** (1.51 g) in a 66% isolated yield. The success of this gram scale reaction further showed the potency of optimized conditions for bulk processes.

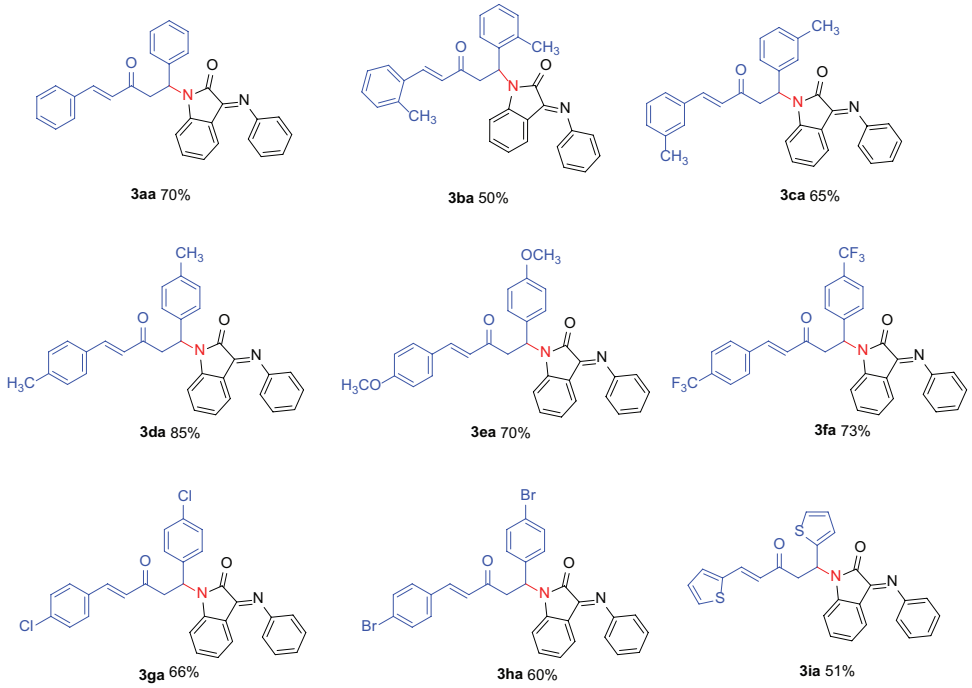
A plausible mechanism is proposed for the reaction of **1a** with **2a** (Scheme 3). 3-(Phenylimino)indolin-2-one (**2a**) is deprotonated by the Cs_2CO_3 to form 3-(phenylimino)indolin-2-one anion **A**. Then, the 1,4-addition of **A** to the divinyl ketone **1a** affords the intermediate **B**. Intermediate **B** then undergoes protonation to give the enolic intermediate **C**. **C** readily tautomerizes to produce the final product **3aa**.

Conclusion

In conclusion, an efficient method for mono-aza-Michael additions of symmetric divinyl ketones with 3-(arylimino) indolin-2-ones using cesium carbonate as the base has been developed. This protocol has advantages of high regioselectivity, high yields, and mild conditions. This will be a useful alternative for the synthesis of *N*-enone-functionalized indolin-2-ones. Owing to the compatibility

Table 2. The reactions of various divinyl ketones with **2a**.[a]





[a]Reaction conditions: **1a–i** (0.3 mmol), **2a** (0.36 mmol), and Cs_2CO_3 (0.15 mmol) in CH_2Cl_2 (3 mL) at room temperature for 8 h.

of multiple functional groups, such as C=C, C=O, C=N, and aryl rings, with the reaction, the resulting compounds will have many applications for the synthesis of other useful fine chemicals.

Experimental

General information

^{1}H NMR and ^{13}C NMR spectra were obtained with Mercury-400BB and Mercury-600BB instruments using $CDCl_3$ as solvent and Me_4Si as the internal standard. High-resolution mass spectra (HRMS) (ESI) were obtained with a Bruker Daltonics APEX II 47e and Orbitrap Elite mass spectrometer. Divinyl ketones[21,22] and 3-(arylimino)indolin-2-ones[6,23] were synthesized according to literature procedures.

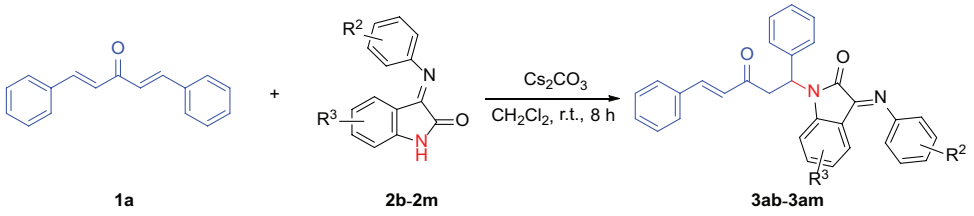
The general procedure for the mono-aza-Michael additions of divinyl ketones with 3-(arylimino)indolin-2-ones: synthesis of **3aa–3ia** *and* **3ab–3am**

A mixture of divinyl ketone (0.3 mmol), 3-(arylimino)indolin-2-one (0.36 mmol), and cesium carbonate (0.15 mmol) in methylene chloride (3 mL) was stirred under air at room temperature for 8 h. Then, the resulting mixture was extracted with ethyl acetate (2 × 5 mL). The combined organic layers were dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The residues were isolated by column chromatography using petroleum ether and ethyl acetate (v/v 5:1) as eluent to give pure product. Analytical data for the products are given below.

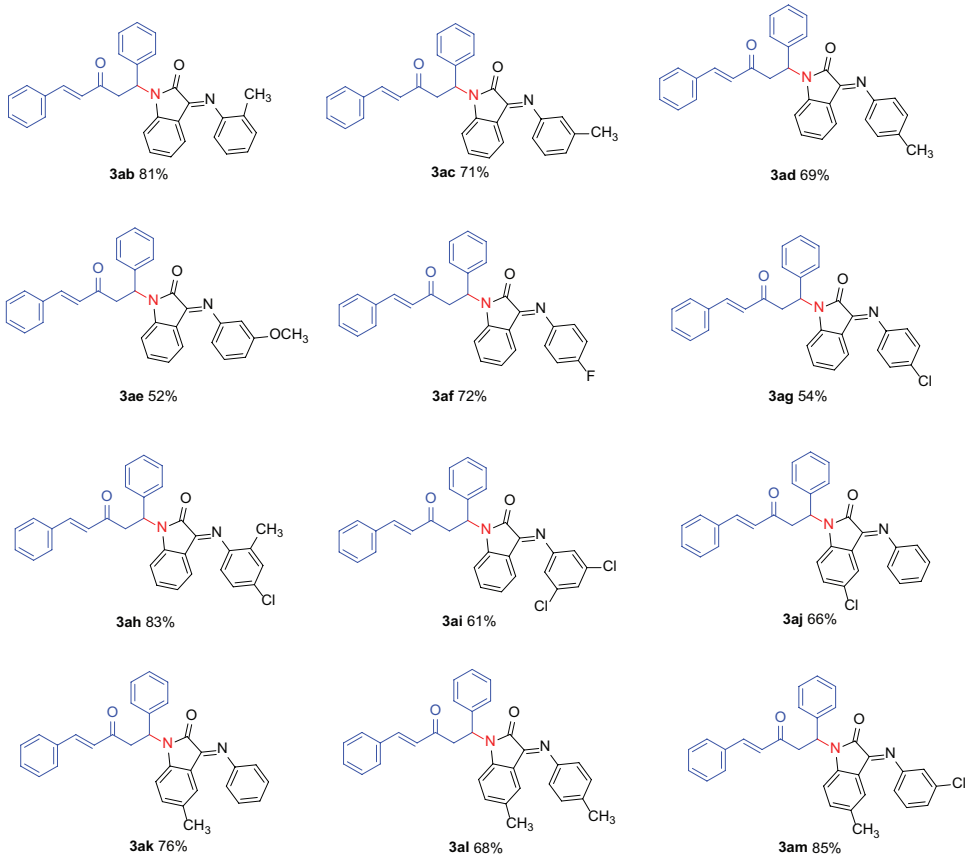
(E)-*1-((E)-3-Oxo-1,5-diphenylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3aa**): Orange solid (96 mg, 70%). m.p. 103–105 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, J = 16.2 Hz, 1H), 7.59–7.52 (m, 4H), 7.41–7.36 (m, 7H), 7.34–7.28 (m, 2H), 7.22 (t, J = 7.5 Hz, 1H), 7.04 (d, J = 8.1 Hz, 1H), 6.96 (d, J = 7.8 Hz, 2H), 6.80 (d, J = 16.2 Hz, 1H), 6.70 (d, J = 7.8 Hz, 1H), 6.58 (d, J = 7.7 Hz, 1H), 5.82 (dd, J = 8.9, 5.4 Hz, 1H), 4.47 (dd, J = 17.5, 8.9 Hz, 1H), 3.56 (dd, J = 17.5, 5.6 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.6, 154.2, 150.4, 147.6, 143.8, 138.6, 134.3, 134.0, 130.7, 129.4, 129.0, 128.9, 128.5, 128.2, 127.3, 126.2, 125.8, 125.1, 122.5, 117.7, 115.9, 110.5, 53.3, 43.0. HRMS (ESI): Calcd for $C_{31}H_{24}N_2O_2$ $[M+H]^+$: 457.1911; found: 457.1910.

(E)-*1-((E)-3-Oxo-1,5-di-o-tolylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ba**): Orange solid (72 mg, 50%). m.p. 76–78 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, J = 16.0 Hz, 1H), 7.67 (dd, J = 7.1, 1.9 Hz, 1H), 7.57 (d, J = 7.9 Hz, 1H), 7.40 (dd, J = 8.4, 7.4 Hz, 2H), 7.28 (td,

Table 3. The reactions of **1a** with various 3-(arylimino)indolin-2-ones.[a]



1a **2b-2m** **3ab-3am**



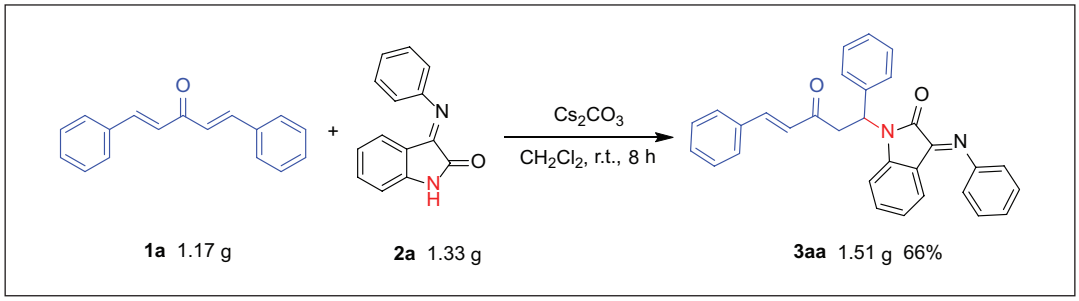
3ab 81% **3ac** 71% **3ad** 69%

3ae 52% **3af** 72% **3ag** 54%

3ah 83% **3ai** 61% **3aj** 66%

3ak 76% **3al** 68% **3am** 85%

[a]Reaction conditions: **1a** (0.3 mmol), **2b–m** (0.36 mmol), and Cs_2CO_3 (0.15 mmol) in CH_2Cl_2 (3 mL) at room temperature for 8 h. Isolated yields.

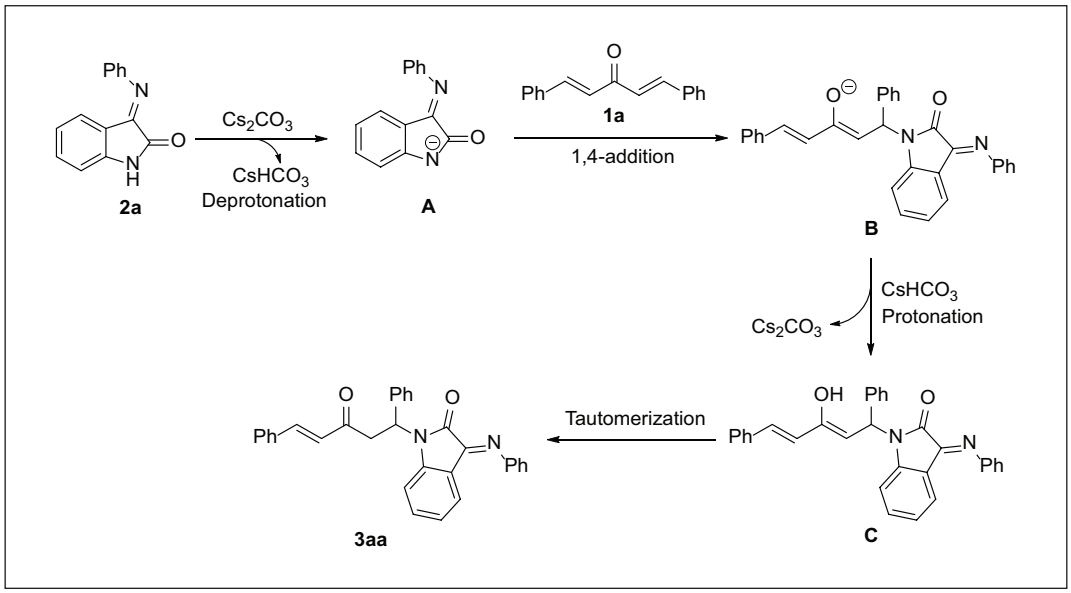


1a 1.17 g **2a** 1.33 g **3aa** 1.51 g 66%

Scheme 2. Gram-scale synthesis of **3aa.**

J=7.5, 1.5 Hz, 2H), 7.25 (d, J=1.4 Hz, 1H), 7.23 (d, J=1.7 Hz, 1H), 7.22–7.19 (m, 4H), 6.96 (dd, J=8.5, 1.2 Hz, 2H), 6.85 (d, J=8.1 Hz, 1H), 6.74 (d, J=16.1 Hz, 1H), 6.69 (td, J=7.7, 0.9 Hz, 1H), 6.58 (dd, J=7.7, 1.3 Hz, 1H), 5.92 (dd, J=9.9, 4.5 Hz, 1H), 4.58 (dd, J=17.3, 9.9 Hz, 1H), 3.24 (dd, J=17.3, 4.5 Hz, 1H), 2.48 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 197.0, 164.0, 154.3, 150.5, 148.0, 141.4, 138.4 136.7, 135.2, 134.1, 133.4, 131.2, 131.0, 130.5, 129.5, 128.3, 127.3, 126.9, 126.8, 126.6, 126.5, 126.2, 125.3, 122.6, 117.8, 116.0, 110.7, 51.3, 42.8, 20.0, 19.7. HRMS (ESI): Calcd for $C_{33}H_{28}N_2O_2$ $[M+H]^+$: 485.2224; found: 485.2222.

(E)-*1-((E)-3-Oxo-1,5-di*-m-*tolylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ca**): Orange solid (94 mg,



Scheme 3. The proposed mechanism for formation of product **3aa.**

65%). m.p. 80−82 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, *J* = 16.0 Hz, 1H), 7.67 (dd, *J* = 7.1, 1.9 Hz, 1H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.40 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.28 (td, *J* = 7.5, 1.5 Hz, 2H), 7.25 (d, *J* = 1.4 Hz, 1H), 7.23 (d, *J* = 1.7 Hz, 1H), 7.22–7.19 (m, 4H), 6.96 (dd, *J* = 8.5, 1.2 Hz, 2H), 6.85 (d, *J* = 8.1 Hz, 1H), 6.74 (d, *J* = 16.1 Hz, 1H), 6.69 (td, *J* = 7.7, 0.9 Hz, 1H), 6.58 (dd, *J* = 7.7, 1.3 Hz, 1H), 5.92 (dd, *J* = 9.9, 4.5 Hz, 1H), 4.58 (dd, *J* = 17.3, 9.9 Hz, 1H), 3.24 (dd, *J* = 17.3, 4.5 Hz, 1H), 2.48 (s, 3H), 2.44 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 197.0, 164.0, 154.3, 150.5, 148.0, 141.4, 138.4, 136.7, 135.2, 134.1, 133.4, 131.2, 131.0, 130.5, 129.5, 128.3, 127.3, 126.9, 126.8, 126.6, 126.5, 126.2, 125.3, 122.6, 117.8, 116.0, 110.7, 51.3, 42.8, 20.0, 19.7. HRMS (ESI): Calcd for $C_{33}H_{28}N_2O_2$ $[M+H]^+$: 485.2224; found: 485.2223.

(E)-*1-((*E*)-3-Oxo-1,5-di*-p-*tolylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3da**): Orange solid (123 mg, 85%). m.p. 175 °C−176 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 (d, *J* = 16.2 Hz, 1H), 7.47 (d, *J* = 8.2 Hz, 2H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.39 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.29 (td, *J* = 7.9, 1.3 Hz, 1H), 7.22 (dt, *J* = 7.5, 1.2 Hz, 1H), 7.19 (t, *J* = 8.2 Hz, 4H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.95 (dd, *J* = 8.5, 1.1 Hz, 2H), 6.76 (d, *J* = 16.2 Hz, 1H), 6.69 (td, *J* = 7.7, 1.0 Hz, 1H), 6.57 (dd, *J* = 7.8, 1.2 Hz, 1H), 5.80 (dd, *J* = 8.9, 5.5 Hz, 1H), 4.41 (dd, *J* = 17.3, 8.9 Hz, 1H), 3.54 (dd, *J* = 17.3, 5.5 Hz, 1H), 2.37 (s, 3H), 2.33 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.6, 154.3, 150.4, 147.6, 143.9, 141.2, 137.9, 135.7, 134.0, 131.5, 129.7, 129.6, 129.3, 128.5, 127.2, 126.1, 125.1, 124.9, 122.4, 117.7, 115.9, 110.6, 53.1, 42.9, 21.5, 21.1. HRMS (ESI): Calcd for $C_{33}H_{28}N_2O_2$ $[M+H]^+$: 485.2224; found: 485.2226.

(E)-*1-((*E*)-1,5-Bis(4-methoxyphenyl)-3-oxopent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ea**): Orange solid (108 mg, 70%). m.p. 186 °C−187 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 16.2 Hz, 1H), 7.49 (dd, *J* = 8.9, 7.2 Hz, 4H), 7.41–7.36 (m, 2H), 7.30 (td, *J* = 7.8, 1.3 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.93 (dd, *J* = 8.5, 1.2 Hz, 2H), 6.89 (dd, *J* = 8.8, 7.1 Hz, 4H), 6.72–6.63 (m, 2H), 6.55 (dd, *J* = 7.7, 1.3 Hz, 1H), 5.77 (dd, *J* = 8.7, 5.6 Hz, 1H), 4.36 (dd, *J* = 17.2, 8.7 Hz, 1H), 3.83 (s, 3H), 3.77 (s, 3H), 3.53 (dd, *J* = 17.2, 5.6 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 163.6, 161.9, 159.4, 154.4, 150.5, 147.7, 143.7, 134.1, 130.8, 130.3, 129.4, 128.8, 127.0, 126.2, 125.2, 123.7, 122.5, 117.7, 115.9, 114.5, 114.3, 110.6, 55.5, 55.4, 52.8, 43.0. HRMS (ESI): Calcd for $C_{33}H_{28}N_2O_4$ $[M+H]^+$: 517.2122; found: 517.2120.

(E)-*1-((*E*)-3-Oxo-1,5-bis(4-(trifluoromethyl)phenyl) pent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3fa**): Orange solid (130 mg, 73%). m.p. 98 °C−100 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.65 (d, *J* = 16.3 Hz, 1H), 7.64–7.62 (m, 5H), 7.59 (s, 1H), 7.40 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.34 (td, *J* = 7.9, 1.3 Hz, 1H), 7.25–7.20 (m, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 6.97–6.94 (m, 2H), 6.86 (d, *J* = 16.3 Hz, 1H), 6.75 (td, *J* = 7.7, 0.9 Hz, 1H), 6.63 (dd, *J* = 7.7, 1.2 Hz, 1H), 5.83 (dd, *J* = 8.7, 5.5 Hz, 1H), 4.49 (dd, *J* = 17.7, 8.7 Hz, 1H), 3.61 (dd, *J* = 17.7, 5.4 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.1, 163.8, 153.9, 150.3, 147.2, 142.6, 142.2, 137.7, 134.2, 132.3 (q, *J* = 32.7 Hz), 130.7 (q, *J* = 32.6 Hz), 129.5, 128.7, 127.9, 127.7, 126.5, 126.1 (q, *J* = 3.6 Hz), 126.0 (q, *J* = 3.7 Hz), 125.5, 124.9, 124.8, 123.0, 117.7, 116.0, 110.3, 53.0, 43.3. HRMS (ESI): Calcd for $C_{33}H_{22}F_6N_2O_2$ $[M+H]^+$: 593.1658; found: 593.1657.

(E)-*1-((*E*)-1,5-Bis(4-chlorophenyl)-3-oxopent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ga**): Orange solid (104 mg, 66%). m.p. 186 °C−187 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, *J* = 16.2 Hz, 1H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.6 Hz, 2H), 7.40 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.35–7.31 (m, 3H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 8.5 Hz, 1H), 6.96–6.93 (m, 2H), 6.75 (d, *J* = 16.2 Hz, 1H), 6.72 (dd, *J* = 7.6, 0.9 Hz, 1H), 6.60 (d, *J* = 7.7 Hz, 1H), 5.74 (dd, *J* = 8.7, 5.6 Hz, 1H), 4.40 (dd, *J* = 17.5, 8.7 Hz, 1H), 3.54 (dd, *J* = 17.5, 5.6 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.4, 163.8, 154.1, 150.4, 147.4, 142.6, 137.2, 136.9, 134.3, 134.2, 132.8, 129.7, 129.5, 129.4, 129.3, 128.9, 126.4, 126.1, 125.4, 122.9,

117.8, 116.0, 110.4, 52.8, 43.2. HRMS (ESI): Calcd for $C_{31}H_{22}Cl_2N_2O_2$ $[M+H]^+$: 525.1131; found: 525.1131.

(E)-*1-((*E*)-1,5-Bis(4-bromophenyl)-3-oxopent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ha**): Orange solid (110 mg, 60%). m.p. 189 °C−190 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, *J* = 16.2 Hz, 1H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.49 (d, *J* = 8.6 Hz, 2H), 7.44 (d, *J* = 8.6 Hz, 2H), 7.42–7.37 (m, 4H), 7.32 (td, *J* = 7.8, 1.3 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.00 (d, *J* = 8.1 Hz, 1H), 6.94 (d, *J* = 7.3 Hz, 2H), 6.76 (d, *J* = 16.2 Hz, 1H), 6.73 (t, *J* = 7.2 Hz, 1H), 6.60 (d, *J* = 7.7 Hz, 1H), 5.72 (dd, *J* = 8.6, 5.6 Hz, 1H), 4.40 (dd, *J* = 17.5, 8.6 Hz, 1H), 3.53 (dd, *J* = 17.5, 5.6 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.4, 163.8, 154.1, 150.4, 147.3, 142.7, 137.8, 134.2, 133.2, 132.4, 132.3, 129.9, 129.5, 129.2, 126.4, 126.2, 125.4, 125.3, 122.9, 122.4, 117.8, 116.0, 110.4, 52.9, 43.2. HRMS (ESI): Calcd for $C_{31}H_{22}Br_2N_2O_2$ $[M+H]^+$: 613.0121; found: 613.0120.

(E)-*1-((*E*)-3-Oxo-1,5-di(thiophen-2-yl)pent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ia**): Orange solid (71 mg, 51%). m.p. 55 °C−57 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 15.8 Hz, 1H), 7.42 (d, *J* = 5.1 Hz, 1H), 7.40 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.34 (td, *J* = 7.8, 1.3 Hz, 1H), 7.31 (d, *J* = 3.2 Hz, 1H), 7.24 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.22–7.18 (m, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 7.07 (dd, *J* = 5.0, 3.6 Hz, 1H), 6.99–6.92 (m, 3H), 6.73 (td, *J* = 7.7, 0.9 Hz, 1H), 6.61–6.55 (m, 2H), 6.10 (dd, *J* = 8.6, 5.4 Hz, 1H), 4.31 (dd, *J* = 17.3, 8.6 Hz, 1H), 3.61 (dd, *J* = 17.2, 5.5 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 195.7, 163.2, 154.1, 150.5, 147.1, 141.1, 139.7, 136.6, 134.2, 132.4, 129.6, 129.5, 128.5, 127.1, 126.4, 126.3, 125.5, 125.3, 124.4, 122.7, 117.8, 116.1, 110.5, 48.5, 43.8. HRMS (ESI): Calcd for $C_{27}H_{20}N_2O_2S_2$ $[M+H]^+$: 469.1039; found: 469.1037.

(E)-*1-((*E*)-3-Oxo-1,5-diphenylpent-4-en-1-yl)-3-(o-tolylimino)indolin-2-one* (**3ab**): Orange solid (114 mg, 81%). m.p. 69 °C−70 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 16.2 Hz, 1H), 7.61–7.52 (m, 4H), 7.42–7.36 (m, 5H), 7.34–7.29 (m, 2H), 7.27 (dd, *J* = 5.9, 2.2 Hz, 1H), 7.22–7.16 (m, 1H), 7.12 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 6.85–6.76 (m, 2H), 6.70 (t, *J* = 7.6 Hz, 1H), 6.47 (d, *J* = 7.7 Hz, 1H), 5.84 (dd, *J* = 8.7, 5.4 Hz, 1H), 4.44 (dd, *J* = 17.4, 8.7 Hz, 1H), 3.60 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.10 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.5, 154.1, 149.2, 147.3, 143.9, 138.7, 134.3, 134.0, 130.9, 130.7, 129.0, 129.0, 128.5, 128.2, 127.3, 126.7, 126.1, 125.8, 125.1, 122.7, 116.6, 116.2, 110.5, 53.3, 43.0, 17.7. HRMS (ESI): Calcd for $C_{32}H_{26}N_2O_2$ $[M+H]^+$: 471.2067; found: 471.2065.

(E)-*1-((*E*)-3-Oxo-1,5-diphenylpent-4-en-1-yl)-3-(m-tolylimino)indolin-2-one* (**3ac**): Orange solid (103 mg, 71%). m.p. 69 °C−70 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.3 Hz, 1H), 7.59–7.52 (m, 4H), 7.40–7.37 (m, 5H), 7.35 (s, 1H), 7.32 (d, *J* = 1.3 Hz, 1H), 7.31–7.29 (m, 1H), 7.28 (d, *J* = 3.1 Hz, 1H), 7.03 (dd, *J* = 7.8, 5.4 Hz, 2H), 6.80 (d, *J* = 16.3 Hz, 1H), 6.77 (d, *J* = 7.6 Hz, 1H), 6.72 (td, *J* = 7.7, 1.0 Hz, 1H), 6.61 (dd, *J* = 7.7, 1.3 Hz, 1H), 5.81 (dd, *J* = 9.0, 5.4 Hz, 1H), 4.47 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.55 (dd, *J* = 17.5, 5.4 Hz, 1H), 2.35 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.6, 154.0, 150.4, 147.5, 143.8, 139.3, 138.6, 134.3, 133.9, 130.7, 129.2, 129.0, 128.9, 128.5, 128.1, 127.3, 126.2, 125.9, 125.8, 122.5, 118.1, 115.9, 114.6, 110.5, 53.3, 43.0, 21.5. HRMS (ESI): Calcd for $C_{32}H_{26}N_2O_2$ $[M+H]^+$: 471.2067; found: 471.2066.

(E)-*1-((*E*)-3-Oxo-1,5-diphenylpent-4-en-1-yl)-3-(p-tolylimino)indolin-2-one* (**3ad**): Orange solid (100 mg, 69%). m.p. 70 °C−72 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.2 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.55–7.52 (m, 2H), 7.40–7.35 (m, 5H), 7.32–7.28 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.1 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 2H), 6.80 (d, *J* = 16.2 Hz, 1H), 6.75–6.72 (m, 2H), 5.82 (dd, *J* = 8.9, 5.4 Hz, 1H), 4.46 (dd, *J* = 17.4, 8.9 Hz, 1H), 3.56 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.39 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.8, 154.0, 147.8, 147.6, 143.9, 138.8, 135.0, 134.3, 133.9, 130.8, 130.0, 129.0, 129.0, 128.5, 128.2, 127.4, 126.1, 125.9, 122.5, 118.0, 116.1, 110.5, 53.3, 43.1, 21.1. HRMS (ESI): Calcd for $C_{32}H_{26}N_2O_2$ $[M+H]^+$: 471.2067; found: 471.2065.

(E)-*3-((3-Methoxyphenyl)imino)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3ae**): Orange red solid (76 mg, 52%). m.p. 54 °C−56 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.3 Hz, 1H), 7.56 (d, *J* = 7.3 Hz, 2H), 7.54 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.40–7.36 (m, 5H), 7.32–7.27 (m, 3H), 7.06–7.03 (m, 1H), 6.80 (d, *J* = 16.3 Hz, 1H), 6.77–6.74 (m, 1H), 6.73 (dd, *J* = 7.6, 0.9 Hz, 1H), 6.67 (dd, *J* = 7.7, 1.3 Hz, 1H), 6.53 (dd, *J* = 3.7, 1.6 Hz, 2H), 5.79 (dd, *J* = 8.9, 5.4 Hz, 1H), 4.47 (dd, *J* = 17.5, 9.0 Hz, 1H), 3.78 (s, 3H), 3.55 (dd, *J* = 17.5, 5.3 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.7, 160.7, 154.4, 151.8, 147.7, 144.0, 138.8, 134.4, 134.2, 130.8, 130.4, 129.1, 128.6, 128.3, 127.4, 126.5, 125.9, 122.7, 115.9, 111.3, 110.6, 109.7, 103.2, 55.5, 53.5, 43.2. HRMS (ESI): Calcd for $C_{32}H_{26}N_2O_3$ $[M+H]^+$: 487.2016; found: 487.2014.

(E)-*3-((4-Fluorophenyl)imino)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3af**): Orange solid (102 mg, 72%). m.p. 76 °C−78 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.2 Hz, 1H), 7.56 (d, *J* = 7.4 Hz, 2H), 7.53 (dd, *J* = 7.3, 2.3 Hz, 2H), 7.41–7.35 (m, 5H), 7.32 (d, *J* = 7.8 Hz, 2H), 7.10 (t, *J* = 8.6 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 1H), 6.94 (dd, *J* = 8.8, 4.8 Hz, 2H), 6.79 (d, *J* = 16.2 Hz, 1H), 6.77–6.72 (m, 1H), 6.68 (d, *J* = 7.7 Hz, 1H), 5.80 (dd, *J* = 9.1, 5.2 Hz, 1H), 4.48 (dd, *J* = 17.5, 9.1 Hz, 1H), 3.53 (dd, *J* = 17.5, 5.2 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.5, 160.5 (d, *J* = 244.4 Hz), 154.6, 147.7, 146.2 (d, *J* = 2.9 Hz), 143.9, 138.6, 134.2, 134.2, 130.7, 129.0, 128.9, 128.5, 128.2, 127.2, 125.8 (d, *J* = 25.3 Hz), 122.5, 119.5 (d, *J* = 8.0 Hz), 116.3, 116.2, 115.7, 110.6, 53.4, 43.0. HRMS (ESI): Calcd for $C_{31}H_{23}FN_2O_2$ $[M+H]^+$ 475.1816, found 475.1816.

(E)-*3-((4-Chlorophenyl)imino)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3ag**): Orange solid (79 mg, 54%). m.p. 150 °C−151 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.3 Hz, 1H), 7.58–7.52 (m, 4H), 7.40−7.39 (m, 3H), 7.38−7.35 (m, 4H), 7.35–7.31 (m, 2H), 7.05 (d, *J* = 8.1 Hz, 1H), 6.91 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 16.3 Hz, 1H), 6.77–6.75 (m, 1H), 6.67 (d, *J* = 7.7 Hz, 1H), 5.79 (dd, *J* = 9.1, 5.2 Hz, 1H), 4.48 (dd, *J* = 17.4, 9.1 Hz, 1H), 3.53 (dd, *J* = 17.5, 5.2 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.5, 154.7, 148.8, 147.9, 144.0, 138.6, 134.5, 134.3, 130.9, 130.7, 129.7, 129.1, 129.1, 128.6, 128.3, 127.4, 126.2, 125.9, 122.7, 119.4, 115.8, 110.8, 53.5,

43.1. HRMS (ESI): Calcd for $C_{31}H_{23}ClN_2O_2$ $[M+H]^+$: 491.1521; found: 491.1520.

(E)-*3-((4-Chloro-2-methylphenyl)imino)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3ah**): Orange solid (125 mg, 83%). m.p. 61 °C−62 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.2 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.53 (dd, *J* = 7.4, 2.3 Hz, 2H), 7.41–7.35 (m, 5H), 7.34–7.31 (m, 2H), 7.26 (d, *J* = 2.4 Hz, 1H), 7.16 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 16.2 Hz, 1H), 6.75 (t, *J* = 7.6 Hz, 1H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.57 (dd, *J* = 7.7, 1.2 Hz, 1H), 5.82 (dd, *J* = 8.9, 5.4 Hz, 1H), 4.43 (dd, *J* = 17.3, 8.9 Hz, 1H), 3.56 (dd, *J* = 17.3, 5.4 Hz, 1H), 2.08 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.3, 154.5, 147.6, 147.5, 143.9, 138.6, 134.4, 134.3, 130.8, 130.2, 129.1, 129.0, 128.5, 128.5, 128.3, 127.3, 127.3, 126.8, 125.8, 125.7, 122.9, 118.1, 116.1, 110.7, 53.4, 43.0, 17.7. HRMS (ESI): Calcd for $C_{32}H_{25}ClN_2O_2$ $[M+H]^+$: 505.1677; found: 505.1675.

(E)-*3-((3,5-Dichlorophenyl)imino)-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3ai**): Orange solid (96 mg, 61%). m.p. 56 °C−57 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, *J* = 16.2 Hz, 1H), 7.56–7.52 (m, 4H), 7.41–7.36 (m, 6H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.20 (t, *J* = 1.9 Hz, 1H), 7.06 (d, *J* = 8.1 Hz, 1H), 6.85 (d, *J* = 1.8 Hz, 2H), 6.82–6.77 (m, 2H), 6.64 (dd, *J* = 7.8, 1.2 Hz, 1H), 5.78 (dd, *J* = 9.3, 5.1 Hz, 1H), 4.46 (dd, *J* = 17.4, 9.3 Hz, 1H), 3.51 (dd, *J* = 17.4, 5.1 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.2, 155.4, 152.2, 148.1, 144.1, 138.4, 136.0, 135.0, 134.3, 130.9, 129.2, 129.1, 128.6, 128.4, 127.3, 126.5, 125.8, 123.0, 117.2, 116.4, 115.4, 111.0, 53.6, 43.0. HRMS (ESI): Calcd for $C_{31}H_{22}Cl_2N_2O_2$ $[M+H]^+$: 525.1131; found: 525.1130.

(E)-*5-Chloro-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3aj**): Orange solid (97 mg, 66%). m.p. 57 °C−59 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.2 Hz, 1H), 7.54 (d, *J* = 7.4 Hz, 4H), 7.44–7.35 (m, 8H), 7.32–7.26 (m, 2H), 7.00–6.94 (m, 3H), 6.80 (d, *J* = 16.1 Hz, 1H), 6.55 (d, *J* = 2.1 Hz, 1H), 5.76 (dd, *J* = 9.3, 5.0 Hz, 1H), 4.49 (dd, *J* = 17.6, 9.2 Hz, 1H), 3.51 (dd, *J* = 17.6, 5.0 Hz, 1H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.8, 163.2, 153.1, 149.8, 146.1, 144.1, 138.3, 134.3, 133.6, 130.9, 129.6, 129.2, 129.1, 128.6, 128.4, 128.1, 127.3, 126.1, 125.8, 125.8, 117.7, 116.8, 111.8, 53.7, 43.0. HRMS (ESI): Calcd for $C_{31}H_{23}ClN_2O_2$ $[M+H]^+$: 491.1521; found: 491.1520.

(E)-*5-Methyl-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)-3-(phenylimino)indolin-2-one* (**3ak**): Orange solid (107 mg, 76%). m.p. 47 °C−49 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.3 Hz, 1H), 7.58–7.51 (m, 4H), 7.40–7.34 (m, 6H), 7.33–7.27 (m, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.10 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.00–6.90 (m, 3H), 6.80 (d, *J* = 16.2 Hz, 1H), 6.36 (s, 1H), 5.80 (dd, *J* = 8.9, 5.4 Hz, 1H), 4.44 (dd, *J* = 17.4, 8.9 Hz, 1H), 3.55 (dd, *J* = 17.4, 5.3 Hz, 1H), 2.01 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 163.7, 154.5, 150.5, 145.4, 143.9, 138.7, 134.5, 134.3, 132.1, 130.8, 129.4, 129.0, 128.5, 128.2, 127.3, 126.8, 125.8, 125.2, 119.0, 117.7, 115.9, 110.4, 53.2, 43.0, 20.8. HRMS (ESI): Calcd for $C_{32}H_{26}N_2O_2$ $[M+H]^+$: 471.2067; found: 471.2065.

(E)-*5-Methyl-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)-3-(*p*-tolylimino)indolin-2-one* (**3al**): Orange solid (99 mg, 68%). m.p. 67 °C−69 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.2 Hz, 1H), 7.57–7.51 (m, 4H), 7.40–7.34 (m, 5H), 7.32–7.29 (m, 1H), 7.22–7.18 (m, 2H), 7.12–7.09 (m, 1H), 6.92–6.88 (m, 3H), 6.79 (d, *J* = 16.2 Hz, 1H), 6.56 (s, 1H), 5.80 (dd, *J* = 8.9, 5.5 Hz, 1H), 4.44 (dd, *J* = 17.4, 8.9 Hz, 1H), 3.55 (dd, *J* = 17.4, 5.5 Hz, 1H), 2.39 (s, 3H), 2.04 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.8, 154.1, 147.7, 145.4, 143.9, 138.8, 135.0, 134.4, 132.0, 130.7, 129.9, 129.2, 129.0, 128.5, 128.2, 127.3, 126.6, 126.6, 125.9, 118.1, 116.1, 110.3, 53.3, 43.1, 21.1, 20.9. HRMS (ESI): Calcd for $C_{33}H_{28}N_2O_2$ $[M+H]^+$: 485.2224; found: 485.2223.

(E)-*3-((3-Chlorophenyl)imino)-5-methyl-1-((*E*)-3-oxo-1,5-diphenylpent-4-en-1-yl)indolin-2-one* (**3am**): Orange solid (129 mg, 85%). m.p. 73 °C−75 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.3 Hz, 1H), 7.56–7.51 (m, 4H), 7.40–7.35 (m, 6H), 7.31 (dd, *J* = 7.7, 3.1 Hz, 2H), 7.14 (dd, *J* = 8.1, 1.0 Hz, 1H), 6.98 (t, *J* = 2.0 Hz, 1H), 6.93 (d, *J* = 8.2 Hz, 1H), 6.84 (ddd, *J* = 8.0, 2.0, 1.0 Hz, 1H), 6.79 (d, *J* = 16.2 Hz, 1H), 6.41 (s, 1H), 5.79 (dd, *J* = 9.1, 5.3 Hz, 1H), 4.43 (dd, *J* = 17.4, 9.1 Hz, 1H), 3.53 (dd, *J* = 17.4, 5.3 Hz, 1H), 2.05 (s, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 196.9, 163.5, 155.1, 151.6, 145.7, 143.9, 138.6, 135.1, 135.0, 134.3, 132.3, 130.8, 130.6, 129.1, 129.0, 128.5, 128.3, 127.3, 126.9, 125.9, 125.1, 118.1, 116.1, 115.7, 110.6, 53.4, 43.0, 20.9. HRMS (ESI): Calcd for $C_{32}H_{25}ClN_2O_2$ $[M+H]^+$: 505.1677; found: 505.1676.

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Supplemental material

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