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O_2 促进下五元环烯胺的 C—H 亚胺化

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摘要　报道了一个 O_2 促进下五元环烯胺的 C—H 亚胺化反应. 在微波辐射下, 季酮酸或 1,3-环戊二酮衍生的烯胺酮、芳酮醛水合物和芳胺三组分参与的无金属催化反应进展顺利, 以良好的产率和完全的立体选择性得到了一系列 *α*-烯胺基-*α*-芳酰基亚胺衍生物. 在该反应过程中, 空气充当绿色氧化剂, 水是唯一的副产物.
关键词　三组分; 区域选择性亚胺化; 微波辐射

O_2-Enabled C—H Imination of Five-Membered Cyclic Enamines

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Abstract　An O_2-enabled C—H imination of five-membered cyclic enamines is reported. Metal-free catalytic three-component reactions of tetronic acid- or 1,3-cyclopentanedione-derived enaminones with arylglyoxals and arylamines worked well under microwave irradiation, furnishing a series of *α*-enamino-*α*-aroyl imines with good yields and complete stereoselectivity. In this reaction process, air serves as the green oxidant with water as the sole byproduct.
Keywords　three component; regioselective imination; microwave irradiation

1　Introduction

Imines frequently serve as versatile intermediates and precursors for the synthesis of various dyes, pigments, polymers, natural products, agrochemicals and pharmaceuticals.[1] A classical method for the formation of imines involves acid-catalyzed condensation between aldehydes or ketones and amines. The following multistep Staudinger/aza-Wittig reaction is typically used, which starts from azides and phosphines via iminophosphorane formation, followed by reaction with aldehydes or ketones (Scheme 1, a).[2] Another redox-neutral imination reaction from azides and alcohols into imines is catalyzed by Ni without adding any external redox reagents (Scheme 1, b).[3] Recent efforts have been made to establish several alternative and reliable methods for the fabrication of imines, which mainly include oxidation of amines,[4] oxidative dehydrogenative coupling of alcohols and amines,[5] hydroamination of alkynes by amines,[6] hydrogenative coupling of nitriles and amines,[7] transfer hydrogenative cross-coupling of nitriles with alcohols[8] and hydrogenation of nitriles.[9] Generally, most of the methods for imine synthesis require transition metal catalysts or stoichiometric amounts of sacrificial oxidants or reductants. Thus, the development of new metal-, oxidant- and reductant-free synthetic approaches for assembling functionalized imines would be particularly useful, given their great application in organic chemistry and pharmaceutical science.

Enaminones, including other analogous electron-deficient enamines such as enamino esters, are an important class of molecules in synthetic organic chemistry and are often used as versatile building blocks for organic synthesis, particularly in heterocyclic chemistry, due to their special structural features.[10] These utilities continue to stimulate interest in the development of new synthetic strategies for accessing polyfunctionalized enaminones by direct C—H functionalization. To date, a number of catalytic and/or oxidative C—H functionalizations, including acyloxylation,[11] selenylation,[12] arylation,[13] alkenylation,[14] sulfenylation,[15] sulfonylation,[16] trifluoromethylation,[17] fluorination[18] and amination,[19] have been well documented. However, there is no report on the oxidative C—H imination of cyclic enamines. On the other hand, the organic synthesis reaction involving hydroxyalkylation,[20a] alkylation,[20a] heteroarylation,[20h] alkenylation[20i] enabled by O_2 has been widely reported, which fully reflect the

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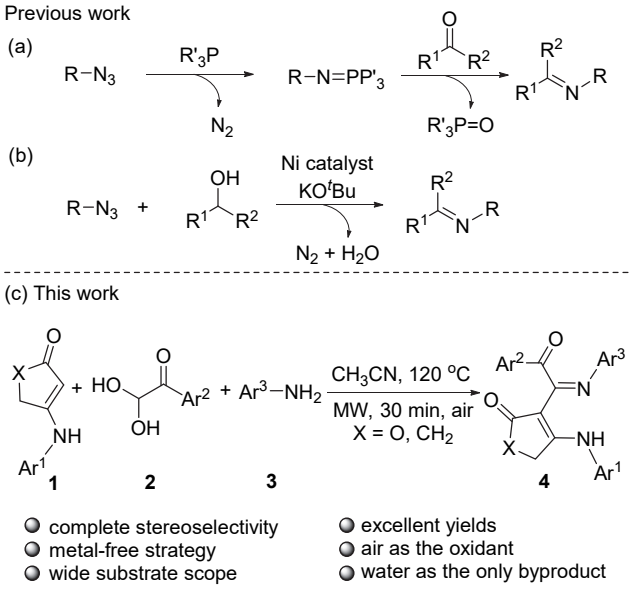
advantages of O_2 as oxidant.[20] Herein, we report a new O_2-enabled oxidative C—H imination of cyclic five-membered enamines under microwave irradiation starting from tetronic acid- or 1,3-cyclopentanedione-derived enaminones **1**, arylglyoxals **2** and arylamines **3**, in which a series of *α*-enamino-*α*-aroyl imines **4** were obtained in good yield with complete stereoselectivity (Scheme 1, c). Notable features of this transformation are the metal-free strategy, wide substrate scope, excellent yields, use of air as the oxidant and water as the only byproduct.

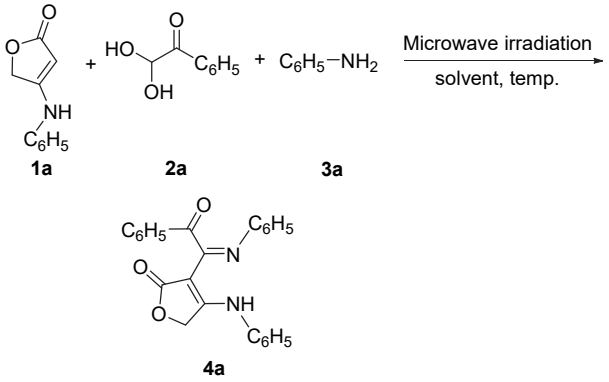


Scheme 1 Methods for the formation of imines

2 Results and discussion

Our study was began with the reaction of 4-(phenylamino)furan-2(5*H*)-one (**1a**), 2,2-dihydroxy-1-phenylethanone (**2a**) and aniline (**3a**). The parameter optimization, including solvents and reaction temperatures, is depicted in Table 1. First, the effect of the solvent on this reaction was studied. It was found that the reaction could proceed in ethyl acetate (EA) at 100 ℃ under air conditions, leading to imine product **4a** in 27% yield (Entry 1). Exchanging EA with EtOH or 1,2-dichloroethane (DCE) as a solvent resulted in a similar inferior result (Entries 2, 3). The use of benzene as the reaction medium completely suppressed the reaction process (Entry 4). A remarkable increase in the yields was observed when 1,4-dioxane, tetrahydrofuran (THF) and MeCN were independently adopted (Entries 5～7). Among these three solvents, the latter proved to be the best choice, as the highest yield was obtained (68%, Entry 7). Next, the influence of reaction temperature was also examined (Entries 8～12). Increasing the reaction temperature to 120 ℃ was beneficial for this transformation, providing 73% yield of **4a** (Entry 10). Further elevating the reaction temperature to 130 ℃ gave a slightly decreased yield (Entry 12). Subsequently, the reaction was carried out under O_2 conditions, but the yield of **4a** was not improved (Entry 13). The desired product **4a** was not observed under Ar conditions, indicating that O_2 plays a key role in the success of this reaction (Entry 14). To simplify the operation, the reaction was run under air conditions.

Table 1 Optimization for the synthesis of product **4a**[a]



Entry	Solvent	Temp./℃	Yield[b]/%
1	EA	100	27
2	EtOH	100	29
3	DCE	100	33
4	Benzene	100	Trace
5	1,4-Dioxane	100	43
6	THF	100	58
7	MeCN	100	68
8	MeCN	110	70
9	MeCN	115	71
10	MeCN	120	73
11	MeCN	125	72
12	MeCN	130	71
13	MeCN	120	73[c]
14	MeCN	120	ND[d]

[a] Reaction conditions: all the reactions were performed with 0.1 mmol of **1a**, 0.1 mmol of **2a**, 0.1 mmol of **3a**, 2.0 mL of solvent in the sealed reaction tube under microwave radiation conditions for 30 min. [b] Isolated yield. [c] O_2 (101 kPa) conditions. [d] ND (not detected) under Ar conditions.

With these optimized conditions in hand, the scope of this regioselective imination process was examined by using various easily available 4-(arylamino)furan-2(5*H*)-ones **1**, arylglyoxals **2** and aromatic amines **3**. As revealed in Table 2, a variety of substituent-diverse 4-(arylamino)-furan-2(5*H*)-ones bearing 4-F (**1b**), 3,5-Cl_2 (**1c**), 4-Br (**1d**) and 4-Me (**1e**) groups in the phenyl group were compatible. Both *para*-chloro (**2c**) and *para*-methyl (**2d**) substituents from arylglyoxals were accommodated as well. Alternatively, 2-thenyl glyoxal (**2b**) showed high reactivity, giving product **4e** in 75% yield. Moreover, aromatic amines **3** with 4-chloro- (**3b**), 4-methoxy- (**3c**), 3,4-dichloro- (**3d**), 4-bromo- (**3e**) and 3,5-dichloro- (**3f**) substituents all worked well to provide the desired *α*-enamino-*α*-aroyl imines **4a**～**4h** in good to excellent yields.

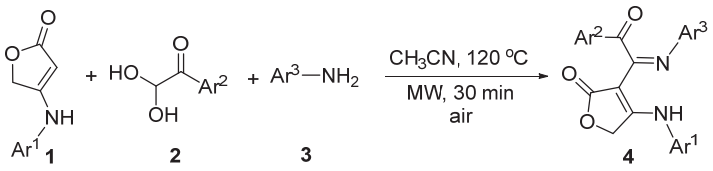
To expand the synthetic utility of this methodology, *N*-aryl 3-aminocyclopent-2-en-1-ones **1f**～**1j** as five-membered cyclic enamines were investigated. As expected, the reactions of 3-aminocyclopent-2-en-1-ones with arylgly-

oxals **2** and aromatic amines **3** worked readily, furnishing products **4i**～**4p** in 74%～83% yields. As shown in Table 3, both electron-rich [*e.g.*, 3,5-Cl_2 (**1f**)] and electron-deficient [*e.g.*, 4-methyl (**1g**), 3-methyl (**1h**, and 4-MeO (**1i**)] groups residing in the phenyl ring of 3-aminocyclopent-2-en-1-ones were suitable. It is worth mentioning that the *N*-benzyl counterpart **1j** was well suited for this transformation, providing product **4p** in 75% yield. Similarly, the electronic properties of both aryl rings (Ar^2 and Ar^3) of substrates **2** and **3** have little impact on the yields. Various electronically neutral (H), rich (*e.g.*, methyl and methoxy), or poor (*e.g.*, chloro, bromo) compounds were all tolerated.

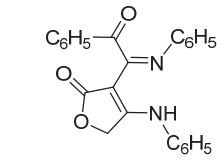
The structural elucidation was unambiguously determined by IR, NMR and HRMS spectroscopic analysis. As shown in Figure 1, the structure of **4f** was further confirmed by X-ray diffraction analysis.

Control reaction was conducted to understand the mechanism of this transformation (Scheme 2). The preformed (*Z*)-1-phenyl-2-(phenylimino)ethanone was subjected to the reaction with 4-(phenylamino)furan-2(5*H*)-one (**1a**), providing (*Z*)-3-(2-oxo-2-phenyl-1-(phenylimino)ethyl)-4-(phenylamino)furan-2(5*H*)-one (**4a**) in 73% yield.

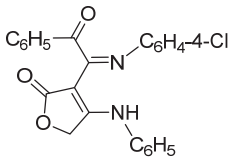
Table 2 Substrate scope for the synthesis of **4a**～**4h**



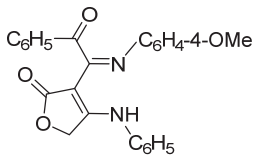
1 **2** **3** **4**



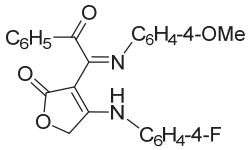
4a (73%)



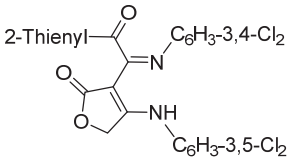
4b (76%)



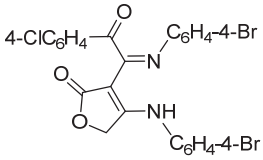
4c (83%)



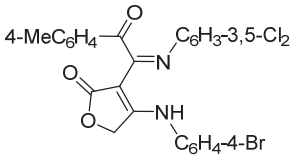
4d (78%)



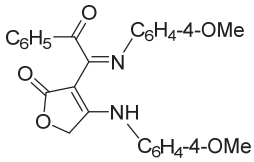
4e (75%)



4f (84%)

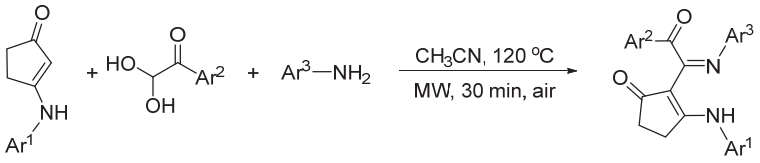


4g (82%)

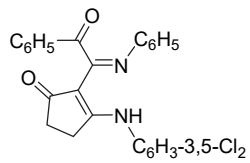


4h (78%)

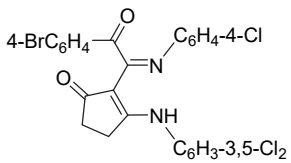
Table 3 Substrate scope for the synthesis of **4i**～**4p**



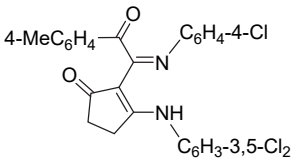
1 **2** **3** **4**



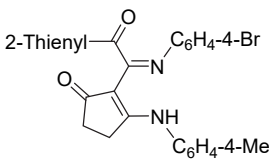
4i (76%)



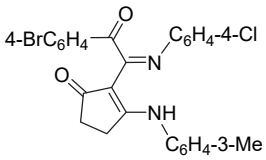
4j (78%)



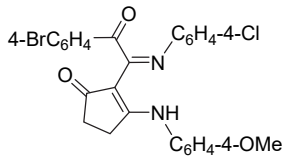
4k (83%)



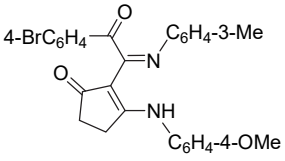
4l (83%)



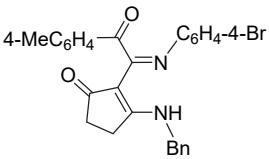
4m (74%)



4n (75%)



4o (77%)



4p (75%)

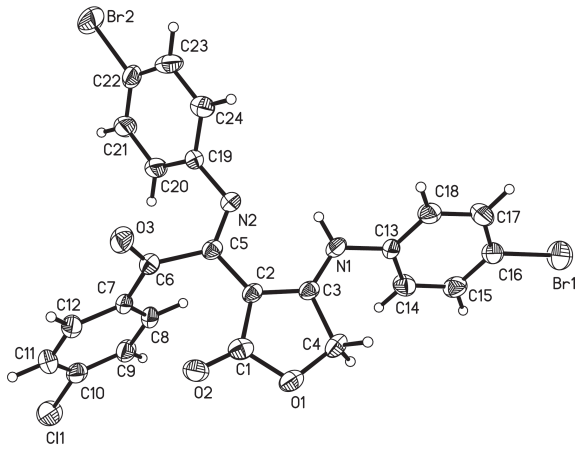
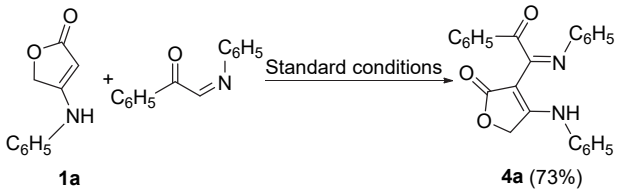
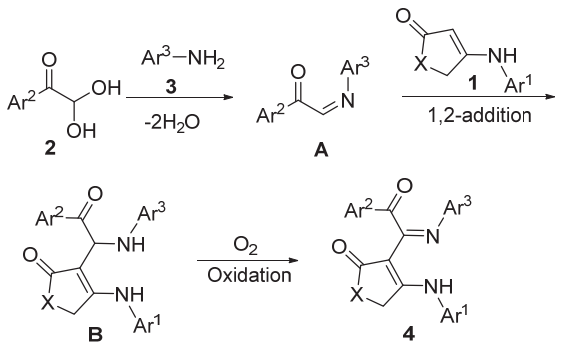


Figure 1 ORTEP (Oak Ridge thermal ellipsoid plot) drawing of **4f**



Scheme 2 Control reaction

Based on the above result, a plausible reaction mechanism is presented in Scheme 3. Arylglyoxal **2** is subjected to arylamine **3** to give access to intermediate **A**, followed by 1,2-addition with enaminone **1** to yield intermediate **B**, which undergoes oxidation of O_2 from air to afford final product **4**.



Scheme 3 Mechanism hypothesis for forming **4**

3 Conclusions

In summary, starting from available tetronic acid- or 1,3-cyclopentanedione-derived enaminones, arylglyoxals and arylamines, a new O_2-enabled C—H imination of five-membered cyclic enamines was developed. Intermolecular and intramolecular variants of this reaction were shown, and a wide range of *α*-enamino-*α*-aroyl imines were synthesized. This protocol features a metal-free strategy, wide substrate scope, good yields, and air behaves as the green oxidant with water as the sole byproduct.

4 Experimental section

4.1 General information

Microwave irradiation was carried out with Initiator 2.5 Microwave Synthesizers from Biotage, Uppsala, Sweden. Melting points were determined in open capillaries and were uncorrected. IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets. ^{1}H NMR (^{13}C NMR) spectra were measured on a Bruker DPX 400 MHz spectrometer in DMSO-d_6 with TMS as internal standard. HRMS (ESI) was determined by using microTOF-QII HRMS/MS instruments (BRUKER). X-ray crystallographic analysis was performed with a Siemens SMART CCD and a Siemens P4 diffractometer.

4.2 General procedure for the synthesis of **4**

In a typical experimental procedure, 4-(phenylamino)-furan-2(5*H*)-one (**1a**) (0.1 mmol, 0.0175 g, 1.0 equiv.), 2,2-dihydroxy-1-phenylethanone (**2a**) (0.1 mmol, 0.0152 g, 1.0 equiv.) and aniline (**3a**) (0.1 mmol, 0.0093 g, 1.0 equiv.) were introduced into a 10-mL reaction vial, and then CH_3CN (2.0 mL) was successively added. Subsequently, the mixture was stirred at 120 ℃ under microwave radiation for 30 min. After completion of the reaction (monitored by thin-layer chromatography), the reaction mixture was brought to room temperature, diluted with cold water, and then extracted with EtOAc. The extract was washed with 10% $Na_2S_2O_3$ solution, dried over anhydrous Na_2SO_4 and evaporated. The residue was purified by column chromatography (eluents: petroleum ether/ethyl acetate, V∶V=18∶1) to afford pure product **4a**. Compounds **4b**～**4p** were synthesized according to above method.

(*Z*)-3-(2-Oxo-2-phenyl-1-(phenylimino)ethyl)-4-(phenylamino)furan-2(5*H*)-one (**4a**): Yellow solid, 0.0279 g, 73% yield. m.p. 215～217 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.68 (s, 1H, NH), 7.76～7.74 (m, 2H, ArH), 7.60 (t, J=7.2 Hz, 1H, ArH), 7.50～7.45 (m, 4H, ArH), 7.41 (d, J=7.6 Hz, 2H, ArH), 7.32 (t, J=7.2 Hz, 1H, ArH), 7.16 (t, J=7.6 Hz, 2H, ArH), 6.96 (t, J=7.6 Hz, 1H, ArH), 6.88 (d, J=7.2 Hz, 2H, ArH), 5.37 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 193.5, 170.7, 168.4, 163.5, 148.3, 137.9, 135.0, 134.6, 130.2, 129.4, 129.0, 128.9, 126.8, 124.9, 122.5, 122.0, 93.0, 66.8; IR (KBr) v: 3455, 3063, 1734, 1681, 1625, 1587, 1564, 1506, 1480, 1456, 1417 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{17}N_2O_3$ $[M-H]^-$ 381.1239 found 381.1246.

(*Z*)-3-(1-((4-Chlorophenyl)imino)-2-oxo-2-phenylethyl)-4-(phenylamino)furan-2(5*H*)-one (**4b**): Yellow solid, 0.0316 g, 76% yield. m.p. 219～221 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.55 (s, 1H, NH), 7.76 (d, J=7.6 Hz, 2H, ArH), 7.62 (t, J=7.2 Hz, 1H, ArH), 7.49 (t, J=7.6 Hz, 4H, ArH), 7.43 (d, J=7.6 Hz, 2H, ArH), 7.32 (t, J=7.2 Hz, 1H, ArH), 7.22 (d, J=8.8 Hz, 2H, ArH), 6.89 (d, J=8.4 Hz, 2H, ArH), 5.36 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 193.3, 170.7, 168.6, 164.0, 147.3, 137.7, 134.9, 134.7, 130.1, 129.5, 129.0, 128.9, 128.8, 127.0, 123.7, 122.8, 93.0, 66.8; IR (KBr) v: 3465, 3068, 1738,

1679, 1625, 1595, 1584, 1556, 1503, 1481, 1457, 1420, 1342 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{16}ClN_2O_3$ $[M-H]^-$ 415.0850, found 415.0867.

(*Z*)-3-(1-((4-Methoxyphenyl)imino)-2-oxo-2-phenylethyl)-4-(phenylamino)furan-2(5*H*)-one (**4c**): Yellow solid, 0.0342 g, 83% yield. m.p. 170～172 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.83 (s, 1H, NH), 7.76 (d, J=7.2 Hz, 2H, ArH), 7.61 (t, J=7.6 Hz, 1H, ArH), 7.48 (t, J=7.6 Hz, 4H, ArH), 7.40 (d, J=7.6 Hz, 2H, ArH), 7.31 (t, J=7.6 Hz, 1H, ArH), 6.88 (d, J=8.8 Hz, 2H, ArH), 6.75 (d, J=8.8 Hz, 2H, ArH), 5.37 (s, 2H, CH_2), 3.64 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 194.2, 170.7, 168.0, 162.9, 156.9, 141.0, 138.0, 134.9, 134.6, 130.2, 129.4, 129.0, 126.6, 123.4, 122.2, 114.3, 93.1, 66.8, 55.6; IR (KBr) v: 3446, 1737, 1681, 1624, 1595, 1585, 1563, 1508, 1456, 1417, 1340 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{19}N_2O_4$ $[M-H]^-$ 411.1345, found 411.1364.

(*Z*)-4-((4-Fluorophenyl)amino)-3-(1-((4-methoxyphenyl)-imino)-2-oxo-2-phenylethyl)furan-2(5*H*)-one (**4d**): Yellow solid, 0.0335 g, 78% yield. m.p. 168～170 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.62 (s, 1H, NH), 7.76～7.74 (m, 2H, ArH), 7.60～7.59 (m, 1H, ArH), 7.48 (s, 4H, ArH), 7.33～7.32 (m, 2H, ArH), 6.87～6.85 (m, 2H, ArH), 6.75～6.73 (m, 2H, ArH), 5.25 (s, 2H, CH_2), 3.63 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 194.2, 170.8, 168.5, 162.7, 160.6 ($^1J_{CF}$=242.3 Hz), 156.8, 141.0, 134.9, 134.5 ($^4J_{CF}$=3.1 Hz), 129.4, 129.0, 125.3 ($^3J_{CF}$=8.6 Hz), 123.4, 116.8 ($^2J_{CF}$=22.8 Hz), 114.2, 93.0, 66.6, 55.6; IR (KBr) v: 3447, 3069, 1744, 1734, 1680, 1632, 1600, 1564, 1511, 1441, 1405, 1342 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{18}FN_2O_4$ $[M-H]^-$ 429.1251, found 429.1268.

(*Z*)-4-((3,5-Dichlorophenyl)amino)-3-(1-((3,4-dichlorophenyl)imino)-2-oxo-2-(thiophen-2-yl)ethyl)furan-2(5*H*)-one (**4e**): Pale yellow solid, 0.0395 g, 75% yield. m.p. 189～191 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.28 (s, 1H, NH), 8.09 (d, J=4.8 Hz, 1H, ArH), 7.70 (d, J=3.2 Hz, 1H, ArH), 7.59 (d, J=10.8 Hz, 3H, ArH), 7.47 (d, J=8.8 Hz, 1H, ArH), 7.22～7.19 (m, 2H, ArH), 6.86 (d, J=7.2 Hz, 1H, ArH), 5.39 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 184.6, 170.4, 168.9, 162.9, 148.4, 141.9, 140.2, 137.7, 136.3, 135.1, 131.1, 130.7, 129.5, 126.9, 126.6, 124.0, 122.4, 122.2, 93.9, 66.8; IR (KBr) v: 3433, 3075, 1756, 1661, 1634, 1588, 1563, 1511, 1463, 1446, 1409, 1347, 1319 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{11}Cl_4N_2O_3S$ $[M-H]^-$ 524.9215, found 524.9237.

(*Z*)-4-((4-Bromophenyl)amino)-3-(1-((4-bromophenyl)-imino)-2-(4-chlorophenyl)-2-oxoethyl)furan-2(5*H*)-one (**4f**): Yellow solid, 0.0482 g, 84% yield. m.p. 183～185 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.42 (s, 1H, NH), 7.77 (d, J=8.4 Hz, 2H, ArH), 7.65 (d, J=8.8 Hz, 2H, ArH), 7.56 (d, J=8.4 Hz, 2H, ArH), 7.41～7.36 (m, 4H, ArH), 6.81 (d, J=8.8 Hz, 2H, ArH), 5.32 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 192.3, 170.8, 168.6, 163.2, 147.6, 139.7, 137.2, 133.6, 132.9, 131.8, 130.8, 129.8, 125.3, 124.0, 119.5, 117.3, 93.3, 67.0; IR (KBr) v: 3451, 3073, 1715, 1704, 1688, 1673, 1608, 1584, 1568, 1552, 1507, 1485, 1463 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{14}Br_2ClN_2O_3$ $[M-H]^-$ 572.9039, found 572.9055.

(*Z*)-4-((4-Bromophenyl)amino)-3-(1-((3,5-dichlorophenyl)imino)-2-oxo-2-(*p*-tolyl)ethyl)furan-2(5*H*)-one (**4g**): Yellow solid, 0.0444 g, 82% yield. m.p. 194～196 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.47 (s, 1H, NH), 7.65 (s, 1H, ArH), 7.63 (s, 1H, ArH), 7.59 (d, J=1.6 Hz, 2H, ArH), 7.56 (t, J=1.6 Hz, 1H, ArH), 7.29 (d, J=8.0 Hz, 2H, ArH), 7.24 (d, J=8.8 Hz, 2H, ArH), 6.89 (d, J=8.8 Hz, 2H, ArH), 5.38 (s, 2H, CH_2), 2.34 (s, 3H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 192.6, 170.5, 168.5, 163.6, 146.9, 145.6, 140.6, 135.1, 132.4, 130.1, 129.1, 129.1, 128.8, 126.3, 123.8, 122.1, 94.1, 66.8, 21.8; IR (KBr) v: 3421, 3112, 1651, 1601, 1520, 1435, 1373, 1350, 1311 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{16}BrCl_2N_2O_3$ $[M-H]^-$ 540.9722, found 540.9711.

(*Z*)-4-((4-Methoxyphenyl)amino)-3-(1-((4-methoxyphenyl)imino)-2-oxo-2-phenylethyl)furan-2(5*H*)-one (**4h**): Pale yellow solid, 0.0345 g, 78% yield. m.p. 211～213 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.53 (s, 1H, NH), 7.75 (d, J=6.8 Hz, 2H, ArH), 7.60～7.58 (m, 1H, ArH), 7.49～7.48 (m, 2H, ArH), 7.38 (d, J=7.6 Hz, 2H, ArH), 7.02 (d, J=8.0 Hz, 2H, ArH), 6.85 (d, J=8.0 Hz, 2H, ArH), 6.73 (d, 2H, J=8.0 Hz, ArH), 5.22 (s, 2H, CH_2), 3.79 (s, 3H, OCH_3), 3.63 (s, 3H, OCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 194.4, 170.9, 168.5, 162.9, 158.2, 156.7, 141.3, 135.0, 134.5, 130.7, 129.4, 129.0, 124.7, 123.3, 115.2, 114.2, 92.4, 66.4, 55.9, 55.5; IR (KBr) v: 3445, 3070, 1668, 1595, 1583, 1461, 1389, 1343 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{21}N_2O_5$ $[M-H]^-$ 441.1451, found 441.1466.

(*Z*)-3-((3,5-Dichlorophenyl)amino)-2-(2-oxo-2-phenyl-1-(phenylimino)ethyl)cyclopent-2-enone (**4i**): Yellow solid, 0.0340 g, 76% yield. m.p. 195～197 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 12.21 (s, 1H, NH), 7.69～7.66 (m, 4H, ArH), 7.56 (t, J=7.6 Hz, 2H, ArH), 7.44 (t, J=7.6 Hz, 2H, ArH), 7.14 (t, J=8.0 Hz, 2H, ArH), 6.94 (t, J=7.6 Hz, 1H, ArH), 6.84 (d, J=7.6 Hz, 2H, ArH), 3.10～3.09 (m, 2H, CH_2), 2.29～2.28 (m, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.6, 193.9, 177.1, 164.0, 147.9, 140.8, 135.4, 135.0, 134.1, 129.2, 128.7, 128.7, 126.3, 124.7, 123.3, 122.1, 109.3, 33.0, 26.4; IR (KBr) v: 3452, 3051, 1748, 1704, 1689, 1682, 1621, 1586, 1558, 1507, 1488, 1449, 1418 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{17}Cl_2N_2O_2$ $[M-H]^-$ 447.0667, found 447.0651.

(*Z*)-2-(2-(4-Bromophenyl)-1-((4-chlorophenyl)imino)-2-oxoethyl)-3-((3,5-dichlorophenyl)amino)cyclopent-2-enone (**4j**): Pale yellow solid, 0.0437 g, 78% yield. m.p. 214～216 ℃; ^1H NMR (400 MHz, DMSO-d_6) δ: 11.96 (s, 1H, NH), 7.70～7.67 (m, 4H, ArH), 7.62～7.59 (m, 3H, ArH), 7.22 (d, J=8.4 Hz, 2H, ArH), 6.83 (d, J=8.8 Hz, 2H, ArH), 3.09～3.08 (m, 2H, CH_2), 2.29 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.7, 193.1, 181.2, 177.3, 164.0, 147.0, 140.4, 134.9, 134.4, 132.6, 130.5, 128.9, 128.7, 128.46, 126.6, 123.8, 123.5, 109.3, 32.9, 26.5; IR (KBr) v: 3434, 3073, 1686, 1680, 1621, 1584, 1560, 1479, 1448, 1421, 1337 cm^{-1}; HRMS (ESI) calcd for $C_{25}H_{15}BrCl_3N_2O_2$ $[M-H]^-$ 558.9383, found 558.9367.

(*Z*)-2-(1-((4-Chlorophenyl)imino)-2-oxo-2-(*p*-tolyl)ethyl)-

3-((3,5-dichlorophenyl)amino)cyclopent-2-enone (**4k**): Pale yellow solid, 0.0412 g, 83% yield. m.p. 190～192 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.08 (s, 1H, NH), 7.67～7.66 (m, 2H, ArH), 7.59～7.57 (m, 3H, ArH), 7.25 (d, J=8.0 Hz, 2H, ArH), 7.20 (d, J=8.8 Hz, 2H, ArH), 6.84 (d, J=8.8 Hz, 2H, ArH), 3.08～3.07 (m, 2H, CH_2), 2.33 (s, 3H, CH_3), 2.27 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.4, 193.3, 177.2, 164.7, 147.1, 144.8, 140.6, 134.9, 133.0, 129.9, 128.8, 128.7, 128.6, 126.4, 123.8, 123.4, 109.3, 33.0, 26.4, 21.7; IR (KBr) v: 3405, 3065, 1663, 1587, 1554, 1518, 1445, 1420, 1388, 1341 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{18}Cl_3N_2O_2$ [M－H]$^-$ 495.0434, found 495.0421.

(*Z*)-2-(1-((4-Bromophenyl)imino)-2-oxo-2-(thiophen-2-yl)ethyl)-3-(*p*-tolylamino)cyclopent-2-enone (**4l**): Yellow solid, 0.0397 g, 83% yield. m.p. 189～191 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.03 (s, 1H, NH), 7.99～7.97 (m, 1H, ArH), 7.59～7.57 (m, 1H, ArH), 7.38～7.35 (m, 4H, ArH), 7.29 (d, J=8.4 Hz, 2H, ArH), 7.14～7.12 (m, 1H, ArH), 6.82 (d, J=8.8 Hz, 2H, ArH), 3.02～2.99 (m, 2H, CH_2), 2.34 (s, 3H, CH_3), 2.29～2.28 (m, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.0, 185.8, 177.5, 163.6, 147.8, 142.7, 136.6, 136.3, 135.3, 135.2, 131.7, 130.4, 129.1, 124.2, 124.1, 116.9, 107.8, 33.1, 26.4, 21.0; IR (KBr) v: 3462, 3093, 1658, 1608, 1558, 1516, 1469, 1427, 1410, 1335 cm^{-1}; HRMS (ESI) calcd for $C_{24}H_{18}BrN_2O_2S$ [M－H]$^-$ 477.0273, found 477.0259.

(*Z*)-2-(2-(4-Bromophenyl)-1-((4-chlorophenyl)imino)-2-oxoethyl)-3-(*m*-tolylamino)cyclopent-2-enone (**4m**): Gray solid, 0.0374 g, 74% yield. m.p. 170～172 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.05 (s, 1H, NH), 7.68 (d, J=8.4 Hz, 2H, ArH), 7.64 (d, J=8.4 Hz, 2H, ArH), 7.37 (t, J=8.4 Hz, 1H, ArH), 7.29 (d, J=6.0 Hz, 2H, ArH), 7.21 (d, J=8.8 Hz, 2H, ArH), 7.15 (d, J=7.2 Hz, 1H, ArH), 6.83 (d, J=8.4 Hz, 2H, ArH), 3.07～3.06 (m, 2H, CH_2), 2.36 (s, 3H, CH_3), 2.29～2.27 (m, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.4, 193.2, 177.3, 164.4, 147.2, 139.7, 137.7, 134.5, 132.5, 130.5, 129.8, 128.8, 128.7, 128.4, 127.8, 124.6, 123.8, 121.1, 108.3, 32.9, 26.6, 21.3; IR (KBr) v: 3440, 3066, 1684, 1625, 1583, 1481, 1439, 1397, 1340, 1307 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{19}BrClN_2O_2$ [M－H]$^-$ 505.0319 found 505.0304.

(*Z*)-2-(2-(4-Bromophenyl)-1-((4-chlorophenyl)imino)-2-oxoethyl)-3-((4-methoxyphenyl)amino)cyclopent-2-enone (**4n**): Pale yellow solid, 0.0392 g, 75% yield. m.p. 175～177 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.85 (s, 1H, NH), 7.68 (d, J=8.4 Hz, 2H, ArH), 7.63 (d, J=8.4 Hz, 2H, ArH), 7.43 (d, J=8.8 Hz, 2H, ArH), 7.20 (d, J=8.0 Hz, 2H, ArH), 7.04 (d, J=8.8 Hz, 2H, ArH), 6.81 (d, J=8.0 Hz, 2H, ArH), 3.80 (s, 3H, OCH_3), 2.94 (s, 2H, CH_2), 2.26 (s, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.3, 193.3, 177.7, 164.3, 158.4, 147.4, 134.5, 132.5, 130.5, 128.7, 128.6, 128.3, 126.1, 123.8, 115.0, 107.9, 55.9, 32.8, 26.3; IR (KBr) v: 3446, 3051, 1697, 1670, 1607, 1583, 1568, 1538, 1511, 1482, 1458, 1398 cm^{-1}; HRMS (ESI) calcd for $C_{26}H_{19}BrClN_2O_3$ [M－H]$^-$ 521.0268, found 521.0253.

(*Z*)-2-(2-(4-Bromophenyl)-2-oxo-1-(*m*-tolylimino)ethyl)-3-((4-methoxyphenyl)amino)cyclopent-2-enone (**4o**): White solid, 0.0387 g, 77% yield. m.p. 163～165 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 12.00 (s, 1H, NH), 7.67 (d, J=8.0 Hz, 2H, ArH), 7.62 (d, J=8.0 Hz, 2H, ArH), 7.43 (d, J=8.4 Hz, 2H, ArH), 7.05～6.99 (m, 3H, ArH), 6.75 (d, J=7.6 Hz, 1H, ArH), 6.64 (s, 1H, ArH), 6.57 (d, J=7.6 Hz, 1H, ArH), 3.80 (s, 3H, OCH_3), 2.95 (s, 2H, CH_2), 2.28～2.23 (m, 2H, CH_2), 2.14 (s, 3H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 199.2, 193.6, 177.5, 163.7, 158.4, 148.3, 138.1, 134.8, 132.4, 130.6, 130.5, 128.7, 128.1, 126.0, 125.3, 122.8, 118.9, 115.1, 108.0, 55.9, 32.8, 26.2, 21.3; IR (KBr) v: 3433, 3053, 1683, 1673, 1607, 1586, 1551, 1512, 1482, 1466, 1396, 1329 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{22}BrN_2O_3$ [M－H]$^-$ 501.0814, found 501.0805.

(*Z*)-3-(Benzylamino)-2-(1-((4-bromophenyl)imino)-2-oxo-2-(*p*-tolyl)ethyl)cyclopent-2-enone (**4p**): Pale yellow solid, 0.0365 g, 75% yield. m.p. 223～225 ℃; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 10.55 (t, J=6.4 Hz, 1H, NH), 7.54 (d, J=8.4 Hz, 2H, ArH), 7.43 (d, J=4.4 Hz, 4H, ArH), 7.35～7.32 (m, 1H, ArH), 7.27 (d, J=8.4 Hz, 2H, ArH), 7.23 (d, J=8.0 Hz, 2H, ArH), 6.66 (d, J=8.4 Hz, 2H, ArH), 4.75 (d, J=6.4 Hz, 2H, CH_2), 2.93～2.89 (m, 2H, CH_2), 2.32 (s, 3H, CH_3), 2.25～2.20 (m, 2H, CH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 198.7, 193.6, 179.2, 164.8, 148.3, 144.5, 138.0, 133.2, 131.5, 129.8, 129.3, 128.8, 128.0, 127.8, 124.1, 116.3, 107.1, 47.7, 32.6, 25.2, 21.7; IR (KBr) v: 3446, 3031, 1668, 1607, 1553, 1488, 1468, 1448, 1402, 1356, 1332 cm^{-1}; HRMS (ESI) calcd for $C_{27}H_{22}BrN_2O_2$ [M－H]$^-$ 485.0865, found 485.0849.

Supporting Information ^{1}H NMR and ^{13}C NMR of all new compounds **4a**～**4p** and crystal data for **4f**. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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