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Ir(III)催化新型三组分串联三氟乙氧基化反应并一锅法构建复杂酰胺化合物

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摘要 开发了 Ir(III)催化的三组分串联反应来构建独特的三氟乙氧基酰胺化合物, 同时氟化物可以继续与醇反应制备复杂的螺旋异多酰胺酮衍生物; 也可通过一锅法直接制备螺旋异多酰胺酮衍生物. 通过条件控制高效地生成各种酰胺化合物.

关键词 三组分串联反应; 氟化反应; 螺环化作用; 2-芳基氧唑

Ir(III)-Catalyzed Novel Three-Component Cascade Trifluoroethoxylation and One-Pot Method to Construct Complex Amide Compounds

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Abstract Ir(III)-catalyzed three-component cascade reaction to construct unique trifluoroethoxylation amide compounds has been developed, meanwhile the fluorinated compounds could continue to react with alcohols to prepare complex spiro isoindolinone derivatives in one-pot. The highly efficient approaches produce various amide compounds by condition-controlled.

Keywords three-component cascade reaction; fluorination; spirocyclization; 2-aryloxazole

1 Introduction

Maleimide frameworks are important N-heterocycles structural units that have widely access to a large variety of natural products and drug molecules.[1] With the recent development, several groups have been committed to study the C—H bond activation reaction of maleimides due to its high activity, low cost and easy availability.[2] Great progress has been made in the study of alkylation, alkenylation, [4＋1] annulation and [4＋2] annulation through transition-metal-catalyzed C—H bond activation using extensive directing groups (Scheme 1a).[3-4]

In recent years, multicomponent C—H functionalization has aroused extensive attention according to its atom- and step-economy.[5] Therefore some forts have been devoted to giving access to complex compounds from simple precursors.[6] Meanwhile, 2-aryloxazoles serving as the directing group have been used in C—H activation reactions because of their unique reactivity.[7] Nevertheless, transition-metal-catalyzed multicomponent C—H bond activation from 2-aryloxazole is still very limited, and only a few examples are provided, for example, Cui,[8a] Kapur[8b] and our groups[8c] separately reported 2-aryloxazol-directed three component C—H bond activation reaction to build isoquinolone derivatives. In 2021, Liu's group[9] described an oxazolinyl-assisted Rh(III)-catalyzed oxidative cyclization of oxazolines with cyclopropanols for synthesis of isoindolinones. Herein, we disclose a new Ir(III)-catalyzed three-component cascade reaction to construct unique fluorinated amide compounds and one-pot spirocyclization to prepare complex spiro isoindolinone derivatives from 2-aryloxazoles using different conditions (Scheme 1b).

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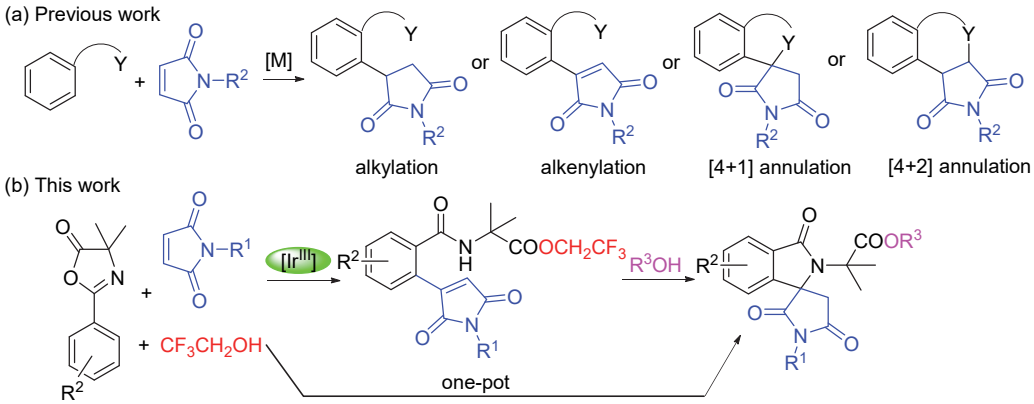
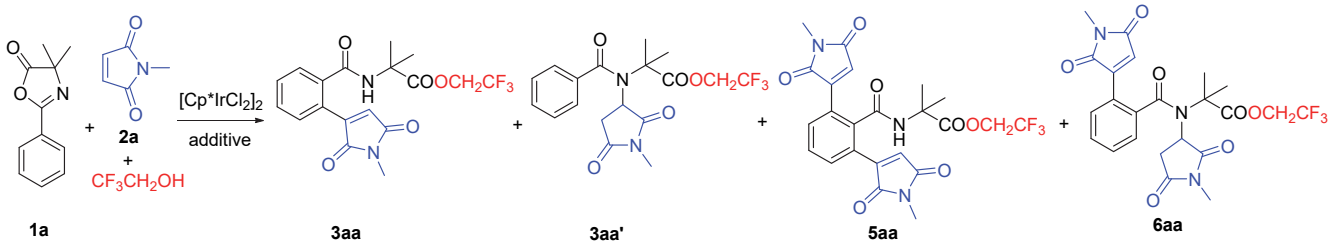


Scheme 1 C—H bond activation from maleimides

2 Results and discussion

Our exploration using 4,4-dimethyl-2-phenyl-oxazol-5(4*H*)-one (**1a**) and *N*-methylmaleimide (**2a**) as model substrates in trifluoroethanol (TFE) to optimize the three-component reaction conditions (Table 1) was started. Initially, no desired product was observed when choosing different oxidant (Entries 1～3). To our delight, fluorinated amide product **3aa** could be isolated in 37% yield in the presence of $[Cp^*IrCl_2]_2$, together with some other products **3aa'**, **5aa** and **6aa** (Entry 4), while the reaction could not proceed smoothly when choosing other metal catalysts.[10] Silver salt screening showed that AgOTf gave the increased result (Entries 5～8). Increasing the loading of

Table 1 Optimization of the reaction conditions[a]



Entry	Oxidant	Ag/Additive	Solvent	Yield[b]/%	
				3aa	**3aa'**
1	AgOAc	$AgSbF_6$	TFE	ND	ND
2	$Cu(OAc)_2•H_2O$	$AgSbF_6$	TFE	ND	ND
3	$Cu(acac)_2$	$AgSbF_6$	TFE	Trace	ND
4	$Cu(OTf)_2$	$AgSbF_6$	TFE	37	Trace
5	$Cu(OTf)_2$	$AgNTf_2$	TFE	40	Trace
6	$Cu(OTf)_2$	$AgBF_4$	TFE	44	Trace
7	$Cu(OTf)_2$	CH_3SO_3Ag	TFE	38	Trace
8	$Cu(OTf)_2$	AgOTf	TFE	47	Trace
9[c]	$Cu(OTf)_2$	AgOTf	TFE	72	Trace
10[c]	$Cu(OTf)_2$	AgOTf	MeOH	ND	ND
11[c]	$Cu(OTf)_2$	AgOTf	EtOH	ND	ND
12[c]	$Cu(OTf)_2$	AgOTf	DCE	ND	ND
13[c]	$Cu(OTf)_2$	AgOTf	CCl_3CH_2OH	ND	ND
14[c]	$Cu(OTf)_2$	AgOTf/PivOH	TFE	66	Trace
15[c]	$Cu(OTf)_2$	AgOTf/NaOAc	TFE	Trace	ND
16[c]	$Cu(OTf)_2$	$AgOTf/Ag_2O$	TFE	40	Trace
17[c,d]	$Cu(OTf)_2$	AgOTf/CuO	TFE	74	Trace
18[c,e]	$Cu(OTf)_2$	AgOTf/CuO	TFE	35	Trace
19[f]	—	$AgNTf_2$/1-AdCOOH	TFE	ND	79

[a] Unless otherwise stated, reaction conditions are as follows: **1a** (0.05 mmol), **2a** (1.5 equiv.), TFE (0.5 mL), $[Cp^*IrCl_2]_2$ (5 mol%), oxidant (1.0 equiv.), Ag salt (20 mol%), additive (1.0 equiv.), 100 ℃, overnight, under Ar. [b] Isolated yield. [c] $Cu(OTf)_2$ (2.0 equiv.), 6 h. [d] **2a** (1.2 equiv.), CuO (0.5 equiv.), 120 ℃, 5 h. [e] **2a** (2.5 equiv.), CuO (0.5 equiv.), 120 ℃, 24 h. [f] $[Ru(p\text{-cymene})Cl_2]_2$ (5 mol%), 1-AdCOOH (0.5 equiv.).

$Cu(OTf)_2$ and shortening the reaction time resulted in a higher yield (Entry 9). However, no reaction occurred when other solvents were used instead of TFE (Entries 10～13). The addition of CuO and the increase of temperature can promote the reaction activity (Entries 14～17). The increase of the amount of **2a** and prolonged time gave **3aa**, **5aa** and **6aa** in 35%, 26% and 29% yields, respectively (Entry 18).[10] It is worth mentioning that only product **3aa'** was detected in 79% yield using [Ru(*p*-cymene)$Cl_2]_2$, 1-AdCOOH and $AgNTf_2$ (Entry 19).

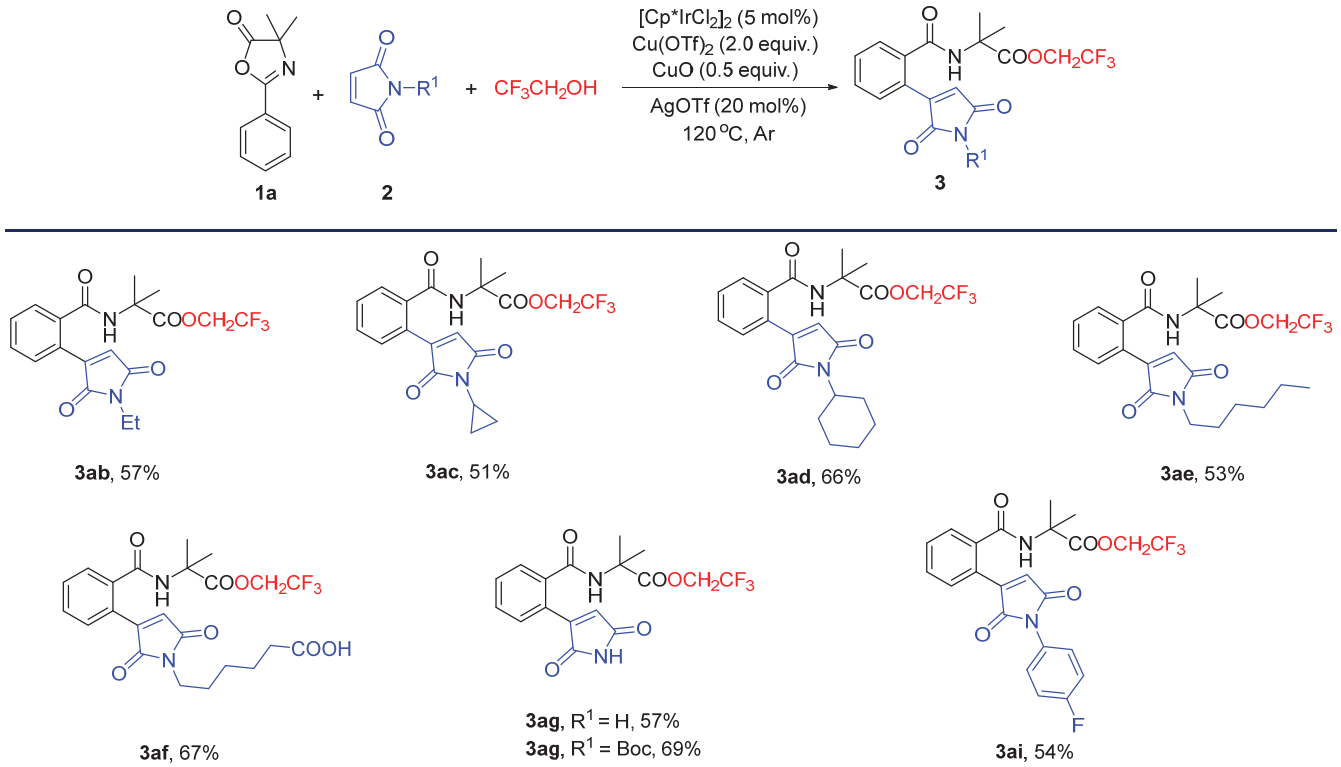
With the optimized reaction conditions in hand, the substrate scope of maleimides was explored in Table 2. Diverse *N*-alkyl-substituents of maleimides (**2b**～**2d**), such as ethyl, cyclohexyl, straight chain hexyl and acid were tolerated to afford the corresponding products in moderate yields. N—H and N-Boc protected maleimides were also suitable substrates, giving the same product **3ag** in 57% and 69% yields, respectively. *N*-Aryl substituents (**2h** and **2i**) reacted smoothly to produce the desired products in moderate yields. Unfortunately, other aryl substituted substrates were sluggish in the system, and only ring opening by-product was obtained.[10]

Next, the substrate scope of 2-aryloxazoles was explored and shown in Table 3. A series of substituents at *para*- and *meta*-position of phenyl ring (**1b**～**1h**) were well tolerated, affording the three component products in moderate yields. Only little *ortho*-position of phenyl ring substituent products were detected because of the steric hindrance.[10] Moreover, thiophene was also a suitable substrate, giving the heteroaryl product **3ia** in 46% yield. Cyclohexyl substituted **1j** proceeded smoothly to afford the corresponding product **3ja** in 47% yield.

Subsequently, **3aa** could be transformed to spirocyclization **4aa** and **4ab** in the presence of NaOAc and ROH in 99% and 93% yields, respectively (Table 4), which underwent transesterification and *aza*-Michael spiro cyclization. Notably, one-pot spirocyclization could work successfully to construct the complex isoindolinone derivatives (**4ba**～**4da**) from phenyloxazols and *N*-methylmaleimides.

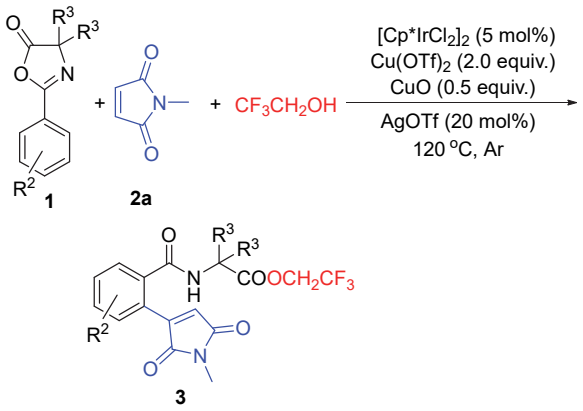
To investigate the reaction mechanism, some experiments were carried out (Scheme 2). **1a** could completely open the ring to form amide compound **7** in the presence of $Cu(OTf)_2$ and TFE (Scheme 2a), and compound **7** could continue to be converted into product **3aa** by reacting with **2a** under standard conditions (Scheme 2b), which showed that **7** was the intermediate of the reaction. The intermolecular competition reaction between the Me- and CF_3-substituents at the *para*-position of the aryl ring with **2a** under standard conditions indicated that the electrically different substituent exhibited similar reaction activity in this reaction (Scheme 2c). Further, 54% deuterium was observed at both *ortho*-positions under standard conditions when the reaction was performed in the absence of **2a** using CD_3OD as the solvent, showing the possibility of the reaction pathway to proceed via *ortho*-C—H bond activation (Scheme 2d).

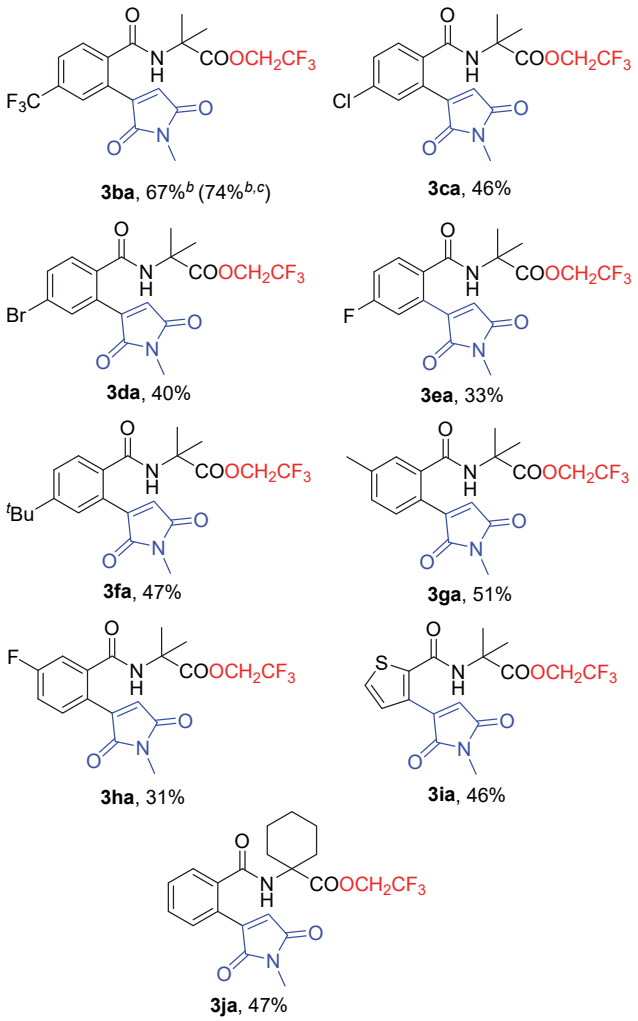
Table 2 Substrate scope of maleimides[a]



[a] Reaction conditions: **1a** (0.1 mmol), **2** (1.2 equiv.), TFE (1.0 mL), $[Cp^*IrCl_2]_2$ (5 mol%), $Cu(OTf)_2$ (2.0 equiv.), AgOTf (20 mol%), CuO (0.5 equiv.), 120 ℃, 6～10 h, under Ar, isolated yield.

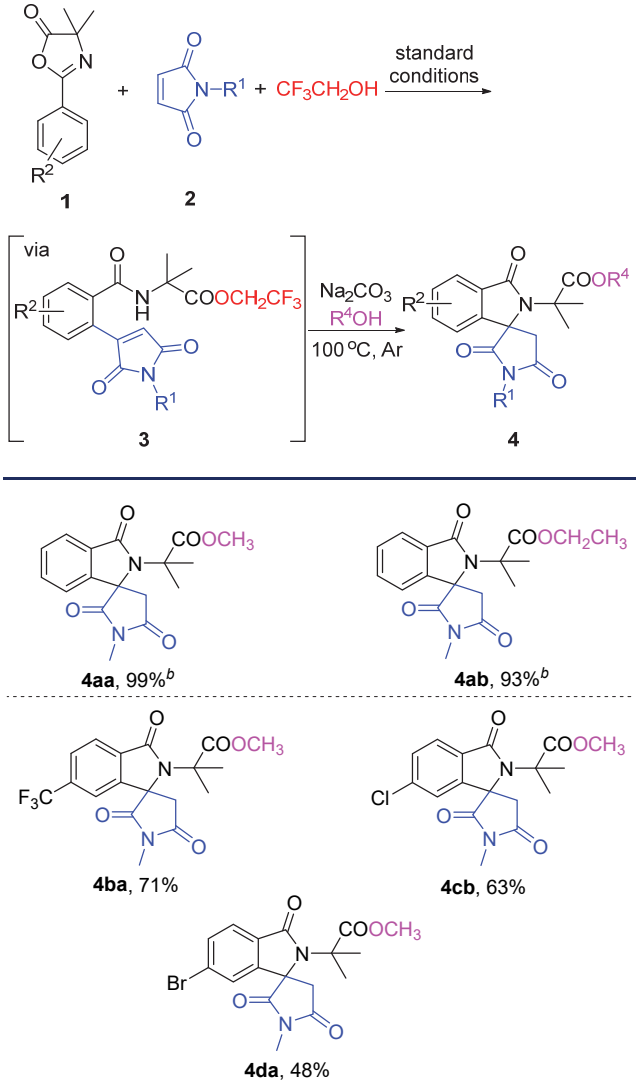
Table 3 Substrate scope of 2-aryloxazole[a]





[a] Reaction conditions: **1** (0.1 mmol), **2a** (1.2 equiv.), TFE (1.0 mL), $[Cp^*IrCl_2]_2$ (5 mol%), $Cu(OTf)_2$ (2.0 equiv.), AgOTf (20 mol%), CuO (0.5 equiv.), 120 ℃, 6～10 h, under Ar. [b] **1** (0.1 mmol), **2a** (1.5 equiv.), no CuO, 5 h. [c] **1** (0.05 mmol).

Table 4 One-pot synthesis of spirocyclization **4**[a]

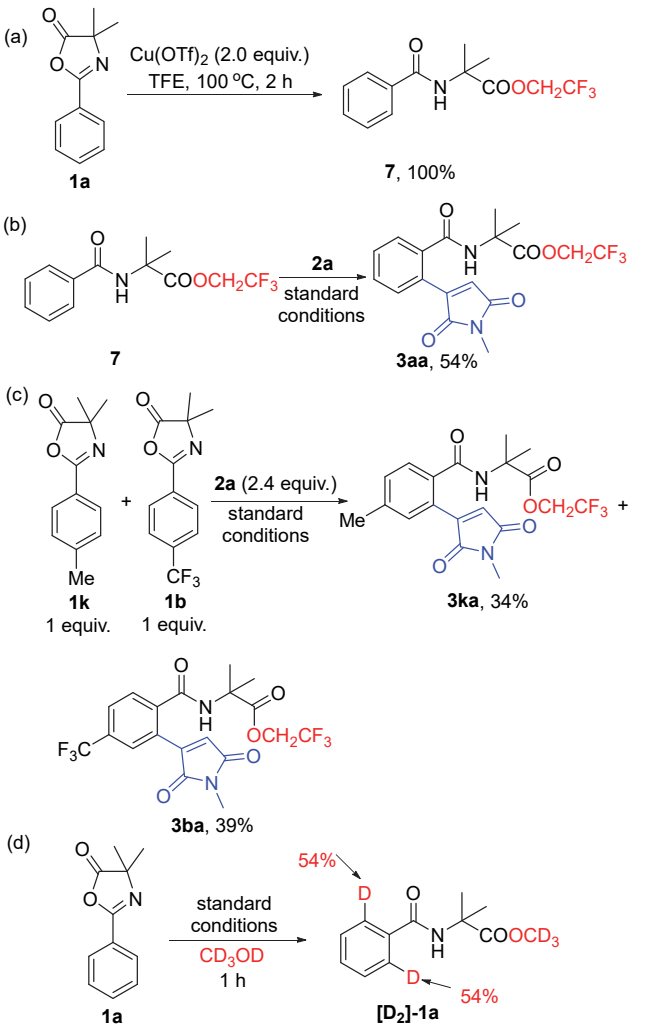


[a] Reaction conditions: From **1** to **4** in one-pot, **1** (0.05 mmol), **2** (1.5 equiv.), TFE (0.5 mL), $[Cp^*IrCl_2]_2$ (5 mol%), $Cu(OTf)_2$ (2.0 equiv.), AgOTf (20 mol%), 100 ℃, 6 h, under Ar. Subsequently, removed solvent, Na_2CO_3 (2.5 equiv) and MeOH (0.5 mL) were directly added to the mixture, 100 ℃, 1～2 h, under Ar. [b] Conditions: from **3** to **4**: **3** (0.025 mmol), NaOAc (1.2 equiv.), ROH (0.3 mL), 100 ℃, 1 h, under Ar.

On the basis of control experiment and the reported literatures,[11] a plausible reaction mechanism was proposed in Scheme 3. Initially, **1a** is transformed into **7** in TFE. Subsequently, coordination of nitrogen atom of ring opening product **7** to the active iridium species undergoes C—H bond activation to afford an iridium cyclic intermediate **I**, which coordinates **2a** to give complex **II**. Then, insertion of maleimide into the C—Ir bond affords complex **III**, and *β*-H elimination provides alkenylated product **3aa** along with Ir(I) species. Finally, the reduced Ir(I) is oxidized to regenerate Ir(III) participating in the next catalytic cycle. **3aa** might undergo transesterification and *aza*-Michael spiro cyclization to generate **4aa**.

3 Conclusions

In summary, a novel Ir(III)-catalyzed efficient synthesis method has been developed to construct the fluorinated compounds and complex spiro isoindolinone derivatives. The former undergoes three-component reaction, and the latter proceeds to undergo transesterification and *aza*-Mi-



Scheme 2 Study on characteristics and mechanism

chael spiro cyclization. The method showed a broad substrate scope and functional group tolerance using 2,2,2-trifluoroethanol as both solvent and reactant.

4 Experimental section

4.1 Instruments and reagents

High-resolution mass spectra (HRMS) were obtained with a Waters-Q-TOF-Premier (ESI). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 400 MHz and 100 MHz instrument. Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Solvents were dried over sodium (for THF and ether) and CaH_2 (for toluene, DCM and DCE) by refluxing for overnight and freshly distilled prior to use.

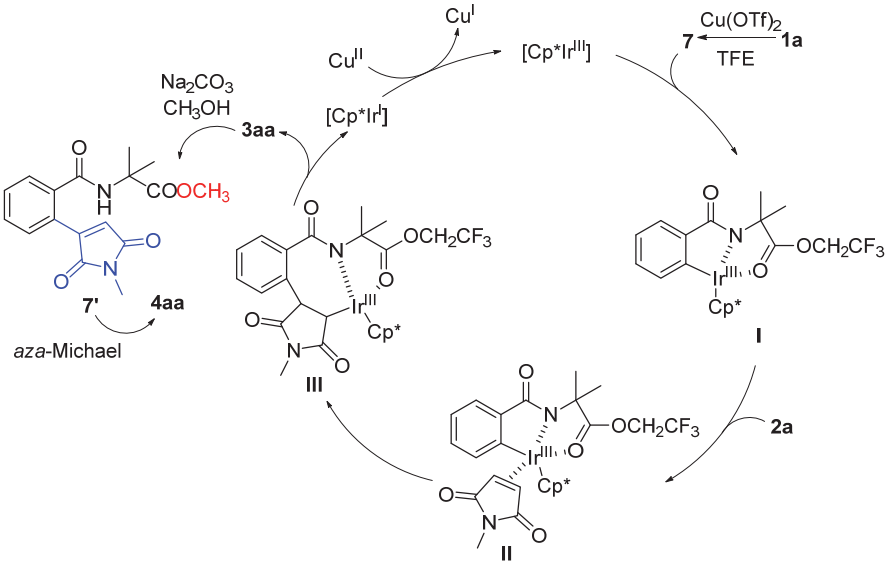
4.2 General procedure for the synthesis of 2-aryloxazole

2-Amino-2-methylpropionic acid (2 g, 19.4 mmol) was dissolved in 1 mol/L NaOH aqueous solution (20 mL). The mixture was cooled to 0 ℃, and then benzoyl chloride (2.30 mL, 19.4 mmol) and 1 mol/L NaOH aqueous solution (20 mL) were added dropwise simultaneously. The resulting mixture was stirred overnight at room temperature. Then the mixture was cooled to 0 ℃, 1 mol/L HCl (60 mL) was added to the reaction mixture and stirred for 30 min. The resulting solid was collected by filtration and washed with water and Et_2O. The crude product was obtained as a white solid.

A solution with crude product (1.0 equiv., 2.0 mmol) and dichloromethane (DCM) (20 mL) was cooled to 0 ℃. Then *N*,*N*-dicyclohexylcarbodiimide (DCC) (1.2 equiv., in DCM 10 mL) was added dropwise and stirred for 30 min. The was stirred overnight at room temperature. The mixture was filtered over a pad of Celite and the filtrate was concentrated *in vacuo*. Purification of the residue by column chromatography [*V*(petroleum ether)∶*V*(ethyl acetate)=15∶1] gave the product **1**.

4.3 General procedure for the synthesis of product 3

A mixture of **1** (0.1 mmol), **2** (1.2 equiv.), TFE (1.0 mL), $[Cp^*IrCl_2]_2$ (5 mol%), AgOTf (20 mol%), Cu-



Scheme 3 Possible mechanism of three-component reaction

$(OTf)_2$ (2.0 equiv.), CuO (0.5 equiv.) were added to an oven dried high-pressure tube under Ar atmosphere. The reaction mixture was stirred at 120 ℃ for 5～10 h [monitored by thin-layer chromatography (TLC)]. After cooling to room temperature, and purification of the mixture by column chromatography [*V*(petroleum ether)∶*V*(ethyl acetate)=4∶1] to give the product **3**.

2,2,2-Trifluoroethyl 2-methyl-2-(2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)propanoate (**3aa**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63 (dd, J=7.5, 1.6 Hz, 1H), 7.52 (tt, J=8.0, 4.0 Hz, 2H), 7.46～7.42 (m, 1H), 6.63 (d, J=1.3 Hz, 1H), 6.54 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 3.04 (d, J=1.3 Hz, 3H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 169.3, 168.9, 166.9, 145.8, 134.5, 130.0, 129.7, 129.3, 127.2, 126.3, 125.4, 121.8(q, J=277.3 Hz), 59.9 (q, J=36.5 Hz), 55.7, 23.5, 22.9; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.83 (t, J=8.5 Hz); HRMS (ESI) calcd for $C_{18}H_{18}F_3N_2O_5$ $[M+H]^+$ 399.1162, found 399.1163.

2,2,2-Trifluoroethyl 2-methyl-2-(*N*-(1-methyl-2,5-dioxopyrrolidin-3-yl)benzamido)propanoate (**3aa'**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (dd, J=10.0, 2.5 Hz, 2H), 7.42 (td, J=7.6, 1.5 Hz, 1H), 7.34 (td, J=7.5, 1.3 Hz, 1H), 7.13～7.09 (m, 1H), 4.52 (q, J=8.5 Hz, 3H), 3.17 (dd, J=18.6, 9.5 Hz, 1H), 3.04 (s,3H), 2.90 (d, J=4.7 Hz, 1H), 2.86 (d, J=4.7 Hz, 1H), 1.66 (s, 3H), 1.57 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 178.0 (d, J=2.5 Hz), 175.2, 171.6, 167.4, 135.3 (d, J=2.8 Hz), 133.4 (d, J=1.9 Hz), 130.0, 127.5, 127.2 (d, J=2.2 Hz), 126.3, 122.0 (q, J=277.1 Hz), 60.2 (q, J=36.4 Hz), 55.5, 42.1, 36.0, 24.5 (d, J=2.0 Hz), 24.2 (d, J=2.0 Hz), 23.1; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.74 (t, J=8.5 Hz); HRMS (ESI) calcd for $C_{18}H_{20}F_3N_2O_5$ $[M+H]^+$ 401.1319, found 401.1329.

2,2,2-Trifluoroethyl 2-(2-(1-ethyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ab**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63 (dd, J=6.8, 1.9 Hz, 1H), 7.53 (td, J=6.6, 1.6 Hz, 2H), 7.44 (dd, J=7.1, 1.8 Hz, 1H), 6.60 (s, 1H), 6.55 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 3.59 (q, J=7.2 Hz, 2H), 1.65 (s, 6H), 1.21 (t, J=7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 169.1, 168.8, 166.9, 145.7, 134.5, 130.0, 129.8, 129.2, 127.3, 126.2, 125.3, 123.5～118.7 (m), 60.0 (d, J=36.6 Hz), 55.7, 32.0, 23.5, 13.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.87 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{19}H_{20}F_3N_2O_5$ $[M+H]^+$ 413.1319, found 413.1328.

2,2,2-Trifluoroethyl 2-(2-(1-cyclopropyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ac**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62 (dd, J=7.2, 1.7 Hz, 1H), 7.52 (pd, J=7.5, 1.7 Hz, 2H), 7.42 (dd, J=7.2, 1.7 Hz, 1H), 6.57 (s, 1H), 6.55 (s, 1H), 4.54 (q, J=8.4 Hz, 2H), 2.54 (tt, J=7.1, 4.1 Hz, 1H), 1.65 (s, 6H), 0.94～0.88 (m, 4H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 169.7, 169.3, 166.9, 145.4, 134.4, 130.0, 129.8, 129.3, 127.4, 126.2, 125.0, 124.7～118.9 (m), 60.0 (q, J=36.7 Hz), 55.7, 23.5, 19.5, 3.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.87 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{20}H_{20}F_3N_2O_5$ $[M+H]^+$ 425.1319, found 425.1327.

2,2,2-Trifluoroethyl 2-(2-(1-cyclohexyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ad**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61 (dd, J=6.8, 2.1 Hz, 1H), 7.52 (td, J=7.0, 1.7 Hz, 2H), 7.45～7.41 (m, 1H), 6.56 (s, 1H), 6.54～6.51 (m, 1H), 4.52 (q, J=8.4 Hz, 2H), 3.92 (tt, J=12.4, 3.9 Hz, 1H), 2.04 (dd, J=12.5, 3.6 Hz, 2H), 1.88～1.80 (m, 2H), 1.74～1.66 (m, 2H), 1.64 (s, 6H), 1.35～1.17 (m, 4H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 169.3, 169.0, 166.9, 145.2, 134.7, 129.9, 129.8, 129.1, 127.3, 126.2, 125.2, 121.8 (q, J=277.2 Hz), 60.0 (q, J=36.7 Hz), 55.7, 50.0, 29.0, 24.9, 24.1, 23.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.87 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{23}H_{26}F_3N_2O_5$ $[M+H]^+$ 467.1788, found 467.1795.

2,2,2-Trifluoroethyl 2-(2-(1-hexyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ae**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63 (dd, J=7.2, 1.7 Hz, 1H), 7.52 (d, J=2.2 Hz, 2H), 7.45 (d, J=1.9 Hz, 1H), 6.60 (s, 1H), 6.56 (s, 1H), 4.52 (d, J=8.4 Hz, 2H), 3.51 (t, J=7.3 Hz, 2H), 1.64 (s, 6H), 1.62 (d, J=1.8 Hz, 2H), 1.29 (d, J=4.0 Hz, 6H), 0.88 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 169.3, 169.0, 166.9, 145.6, 134.5, 130.0, 129.8, 129.2, 127.3, 126.2, 125.2, 121.8 (q, J=277.3 Hz), 60.0 (d, J=36.6 Hz), 55.7, 37.2, 30.3, 27.5, 25.3, 23.5, 21.4, 12.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.88 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{23}H_{28}F_3N_2O_5$ $[M+H]^+$ 469.1945, found 469.1951.

6-(3-(2-((2-Methyl-1-*oxo*-1-(2,2,2-trifluoroethoxy)-propan-2-yl)carbamoyl)phenyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)hexanoic acid (**3af**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62 (d, J=1.9 Hz, 1H), 7.57～7.48 (m, 2H), 7.46～7.41 (m, 1H), 6.60 (s, 1H), 6.57 (s, 1H), 4.52 (q, J=8.4 Hz, 2H), 4.45 (d, J=8.5 Hz, 2H), 3.53 (t, J=7.1 Hz, 2H), 2.42 (t, J=7.5 Hz, 2H), 1.73～1.66 (m, 2H), 1.64 (s, 6H), 1.41～1.32 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 170.8, 169.3, 168.9, 166.9, 145.8, 134.5, 130.0, 129.8, 129.3, 127.3, 126.2, 125.1, 121.9 (qd, J=277.1, 17.4 Hz), 59.5 (dq, J=133.1, 36.6 Hz), 55.7, 36.8, 32.3, 27.1, 24.9, 23.5, 23.1; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.86 (dt, J=13.1, 8.1 Hz); HRMS (ESI) calcd for $C_{23}H_{26}F_3N_2O_7$ $[M+H]^+$ 499.1687, found 499.1690.

2,2,2-Trifluoroethyl 2-(2-(2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ag**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64 (dd, J=6.7, 2.1 Hz, 1H), 7.55 (dtt, J=12.0, 7.4, 3.7 Hz, 2H), 7.47～7.40 (m, 2H), 6.61 (d, J=1.4 Hz, 1H), 6.57 (s, 1H), 4.54 (q, J=8.4 Hz, 2H), 1.65 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 168.6, 168.5, 166.7, 146.8, 134.4, 130.1, 129.9, 129.5, 126.9, 126.3, 126.1, 121.8 (d, J=277.4 Hz), 60.0 (q, J=36.7 Hz), 55.7, 23.5; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.82 (t, J=8.5 Hz); HRMS (ESI) calcd for $C_{17}H_{16}F_3N_2O_5$ $[M+H]^+$ 385.1006, found 385.1014.

2,2,2-Trifluoroethyl 2-(2-(1-(4-fluorophenyl)-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ai**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (dd, J=7.2, 1.7 Hz, 1H), 7.62～7.52 (m, 2H), 7.50 (dd, J=7.2, 1.7

Hz, 1H), 7.34 (dd, J=8.8, 4.9 Hz, 2H), 7.15 (t, J=8.6 Hz, 2H), 6.75 (s, 1H), 6.61 (s, 1H), 4.45 (q, J=8.4 Hz, 2H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 167.9, 167.6, 166.8, 161.5, 159.9, 146.2, 134.4, 130.2, 129.9, 129.6, 127.2, 127.1, 127.0, 126.3, 125.0, 121.8 (q, J=277.4 Hz), 115.1, 114.9, 59.8 (q, J=36.6 Hz), 55.7, 23.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.90 (t, J=8.3 Hz), −113.42 (dt, J=8.6, 3.7 Hz); HRMS (ESI) calcd for $C_{23}H_{19}F_4N_2O_5$ $[M+H]^+$ 479.1225, found 479.1232.

2,2,2-Trifluoroethyl-2-methyl-2-(2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)-4-(trifluoromethyl)benzamido)propanoate (**3ba**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.80～7.73 (m,2H), 7.72 (d, J=1.6 Hz, 1H), 6.73 (s, 1H), 6.57 (s, 1H), 4.54 (q, J=8.3 Hz, 2H), 3.05 (s,3H), 1.66 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.3, 168.7, 168.5, 165.7, 144.1, 137.7, 132.6～131.5 (m), 128.0, 126.9, 126.6, 126.6 (d, J=4.0 Hz), 126.0 (d, J=3.9 Hz), 60.1 (q, J=36.7 Hz), 56.0, 23.4, 23.0 (d, J=1.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ: −63.13, −73.88 (t, J=8.4 Hz). HRMS (ESI) calcd for $C_{19}H_{17}F_6N_2O_5$ $[M+H]^+$ 467.1036, found 467.1045.

2,2,2-Trifluoroethyl 2-(4-chloro-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ca**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, J=8.2 Hz, 1H), 7.48 (dd, J=8.2, 2.1 Hz, 1H), 7.43 (d, J=2.1 Hz, 1H), 6.65 (s, 1H), 6.54 (s, 1H), 4.52 (q, J=8.4 Hz, 2H), 3.04 (s, 3H), 1.63 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 168.9, 168.5, 166.0, 144.5, 136.1, 132.8, 129.6, 129.1, 128.9, 127.6 (d, J=2.1 Hz), 126.1, 121.8 (q, J=277.3 Hz), 60.0 (q, J=36.7 Hz), 55.8, 23.5, 22.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.86 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{18}H_{17}ClF_3N_2O_5$ $[M+H]^+$ 433.0773, found 433.0777.

2,2,2-Trifluoroethyl-(4-bromo-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3da**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.65 (dt, J=8.4, 2.8 Hz, 1H), 7.59 (d, J=1.9 Hz, 1H), 7.49 (d, J=8.3 Hz, 1H), 6.65 (d, J=1.6 Hz, 1H), 6.54 (d, J=13.9 Hz, 1H), 4.52 (q, J=8.4 Hz, 2H), 3.04 (d, J=1.0 Hz, 3H), 1.64 (d, J=1.6 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.3, 168.8, 168.5, 166.0, 144.4, 133.3, 132.4, 132.1, 129.1, 127.7, 126.1, 124.2, 123.1～118.4 (m), 60.0 (q, J=36.7 Hz), 55.8, 23.5, 23.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.86 (t, J=8.3 Hz); HRMS (ESI) calcd for $C_{18}H_{17}BrF_3N_2O_5$ $[M+H]^+$ 477.0267, found 477.0274.

2,2,2-Trifluoroethyl2-(4-fluoro-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ea**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.63 (dd, J=8.5, 5.3 Hz, 1H), 7.23～7.14 (m, 2H), 6.66 (s, 1H), 6.50 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 3.04 (s, 3H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.4, 168.9, 168.5, 166.0, 162.5 (d, J=253.0 Hz), 144.5, 130.8 (d, J=3.6 Hz), 129.7 (d, J=8.7 Hz), 128.5 (d, J=8.9 Hz), 126.2, 121.8 (d, J=277.4 Hz), 117.0 (d, J=23.3 Hz), 115.9 (d, J=21.3 Hz), 60.0 (q, J=36.6 Hz), 55.8, 23.5, 22.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.87 (t, J=8.4 Hz), −107.85 (d, J=9.0 Hz); HRMS (ESI) calcd for $C_{18}H_{17}F_4N_2O_5$ $[M+H]^+$ 417.1068, found 417.1068.

2,2,2-Trifluoroethyl 2-(4-(*tert*-butyl)-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3fa**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58～7.50 (m, 2H), 7.41 (d, J=1.9 Hz, 1H), 6.61 (s, 1H), 6.51 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 3.03 (s, 3H), 1.63 (s, 6H), 1.33 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 169.4, 169.1, 166.8, 153.7, 146.6, 131.6, 127.1, 126.9, 126.3, 126.1, 125.0, 121.8 (q, J=277.2 Hz), 59.9 (q, J=36.6 Hz), 55.6, 33.9, 30.0, 23.6, 22.9; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.83 (t, J=8.2 Hz); HRMS (ESI) calcd for $C_{22}H_{26}F_3N_2O_5$ $[M+H]^+$ 455.1788, found 455.1790.

2,2,2-Trifluoroethyl-2-methyl-2-(5-methyl-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)propanoate (**3ga**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41 (s, 1H), 7.35 (s, 2H), 6.59 (s, 1H), 6.48 (s, 1H), 4.54 (q, J=8.4 Hz, 2H), 3.03 (s, 3H), 2.44 (s, 3H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.5, 169.4, 169.1, 167.0, 145.6, 139.9, 134.5, 130.6, 129.7, 127.0, 124.9, 124.2, 121.8 (d, J=277.3 Hz), 60.1, 55.7, 23.5, 22.9, 20.3; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.82 (t, J=8.5 Hz); HRMS (ESI) calcd for $C_{19}H_{20}F_3N_2O_5$ $[M+H]^+$ 413.1319, found 413.1324.

2,2,2-Trifluoroethyl 2-(5-fluoro-2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)-2-methylpropanoate (**3ha**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.46 (dd, J=8.5, 5.2 Hz, 1H), 7.33 (dd, J=8.4, 2.6 Hz, 1H), 7.23 (dd, J=8.2, 2.6 Hz, 1H), 6.63 (s, 1H), 6.54 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 3.03 (s, 3H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.33, 169.11, 168.93, 165.65, 163.13, 161.45, 144.50, 136.70 (d, J=6.7 Hz), 131.87 (d, J=8.2 Hz), 125.63, 116.99 (d, J=21.4 Hz), 114.05 (d, J=23.4 Hz), 60.06 (q, J=36.6 Hz), 55.92, 27.18, 23.48, 22.96; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.84, −108.30 (d, J=6.7 Hz); HRMS (ESI) calcd for $C_{18}H_{17}F_4N_2O_5$ $[M+H]^+$ 417.1068, found 417.1074.

2,2,2-Trifluoroethyl 2-methyl-2-(3-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)thiophene-2-carboxamido)-propanoate (**3ia**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, J=5.2 Hz, 1H), 7.36 (d, J=5.1 Hz, 1H), 6.86 (s, 1H), 6.52～6.47 (m, 1H), 4.55 (q, J=8.4 Hz, 2H), 3.04 (s, 3H), 1.64 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.3, 169.3, 168.9, 160.3, 138.8, 135.4, 129.7, 129.0, 126.8, 126.1, 121.8 (d, J=277.4 Hz), 60.0 (q, J=36.6 Hz), 56.0, 23.6, 23.0; ^{19}F NMR (565 MHz, $CDCl_3$) δ: −73.80 (t, J=8.4 Hz); HRMS (ESI) calcd for $C_{16}H_{16}F_3N_2O_5S$ $[M+H]^+$ 405.0727, found 405.0732.

2,2,2-trifluoroethyl 1-(2-(1-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-3-yl)benzamido)cyclohexane-1-carboxylate (**3Ja**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.64 (dd, J=7.1, 1.8 Hz, 1H), 7.59～7.50 (m, 2H), 7.46 (dd, J=6.7, 2.3 Hz, 1H), 6.63 (s, 1H), 6.33 (s, 1H), 4.51 (q, J=8.5 Hz, 2H), 3.03 (s, 3H), 2.09 (dt, J=13.5, 4.4 Hz, 2H), 1.97 (ddd, J=14.1, 11.1, 4.0 Hz, 2H), 1.73 (dq, J=13.4, 4.3 Hz, 2H), 1.59～1.52 (m, 2H), 1.45～1.33 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 170.8, 169.3, 169.0, 166.9, 145.6, 134.4, 130.0, 129.8, 129.3, 127.3, 126.3, 125.7,

122.0 (q, J=277.5 Hz), 59.6 (q, J=36.5 Hz), 58.0, 31.2, 23.9, 22.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: −73.74 (t, J=8.5 Hz); HRMS (ESI) calcd for $C_{21}H_{22}F_3N_2O_5$ $[M+H]^+$ 439.1475, found 439.1481.

4.4 General procedure for the synthesis of product **4** in one-pot

A mixture of **1** (0.05 mmol), **2** (1.5 equiv.), $[Cp^*IrCl_2]_2$ (5 mol%), AgOTf (20 mol%), $Cu(OTf)_2$ (2.0 equiv.), TFE (0.5 mL) were added to an oven dried high-pressure tube under Ar atmosphere. The reaction mixture was stirred at 100 ℃ for 5～10 h (monitored by TLC). After cooling to room temperature, the mixture was concentrated *in vacuo*. Subsequently, Na_2CO_3 (2.5 equiv.) and ROH (0.5 mL) were directly to the mixture, and the reaction mixture was stirred at 100 ℃ for 1～2 h under Ar. Purification of the residue by column chromatography [V(petroleum ether)∶V(ethyl acetate)=3∶1] gives the **4**.

4.5 General procedure for the synthesis of product **4** from **3**

A mixture of **3** (0.025 mmol), NaOAc (1.2 equiv.) and ROH (0.3 mL) were added to an oven dried high-pressure tube under Ar atmosphere. And the reaction mixture was stirred at 100 ℃ for 1～2 h. Purification of the residue by column chromatography [V(petroleum ether)∶V(ethyl acetate)=3∶1] gives the **4**.

Methyl 2-methyl-2-(1'-methyl-2',3,5'-trioxospiro[isoindoline-1,3'-pyrrolidin]-2-yl)propanoate (**4aa**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (d, J=7.4 Hz, 1H), 7.54 (dtd, J=24.9, 7.5, 1.1 Hz, 2H), 7.17 (d, J=7.6 Hz, 1H), 3.76 (s, 3H), 3.60 (d, J=19.3 Hz, 1H), 3.20 (s, 3H), 3.15 (s, 1H), 1.73 (s, 3H), 1.53 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 173.7, 172.9, 172.1, 167.5, 144.8, 132.1, 129.0, 128.8, 123.3, 118.0, 67.0, 59.7, 51.9, 39.9, 24.8, 22.8, 22.7; HRMS (ESI) calcd for $C_{17}H_{19}N_2O_5$ $[M+H]^+$ 331.1288, found 331.1294.

Ethyl 2-methyl-2-(1'-methyl-2',3,5'-trioxospiro[isoindoline-1,3'-pyrrolidin]-2-yl)propanoate (**4ab**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85～7.80 (m, 1H), 7.55 (dtd, J=24.2, 7.5, 1.2 Hz, 2H), 7.17 (d, J=7.5 Hz, 1H), 4.27～4.16 (m, 2H), 3.62 (d, J=19.3 Hz, 1H), 3.21 (s, 3H), 3.17 (d, J=19.5 Hz, 1H), 1.75 (s, 3H), 1.53 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 173.8, 172.4, 172.2, 167.5, 144.8, 132.1, 129.1, 128.7, 123.2, 118.0, 67.0, 60.8, 59.8, 40.0, 24.8, 22.8, 22.7, 13.0; HRMS (ESI) calcd for $C_{18}H_{21}N_2O_5$ $[M+H]^+$ 345.1445, found 345.1450.

Methyl 2-methyl-2-(1'-methyl-2',3,5'-trioxo-6-(trifluoromethyl)spiro[isoindoline-1,3'-pyrrolidin]-2-yl)propanoate (**4ba**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95 (d, J=7.9, 0.8 Hz, 1H), 7.83～7.79 (m, 1H), 7.43～7.40 (m, 1H), 3.77 (s, 3H), 3.64 (d, J=19.4 Hz, 1H), 3.24 (s, 3H), 3.19 (d, 1H), 1.74 (s, 3H), 1.54 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 172.9, 172.6, 171.5, 166.1, 145.0, 134.1 (d, J=33.1 Hz), 132.4, 126.3～126.1 (m), 124.0, 122.1 (d, J=273.2 Hz), 115.7～115.2 (m), 67.1, 60.1, 52.0, 39.6, 25.1, 22.7, 22.6; HRMS (ESI) calcd for $C_{18}H_{18}F_3N_2O_5$ $[M+H]^+$ 399.1162, found 399.1175.

Methyl 2-(6-chloro-1'-methyl-2',3,5'-trioxospiro[isoindoline-1,3'-pyrrolidin]-2-yl)-2-methylpropanoate (**4cb**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (d, J=8.1 Hz, 1H), 7.50 (dd, J=8.1, 1.6 Hz, 1H), 7.16 (d, J=1.6 Hz, 1H), 3.76 (s, 3H), 3.60 (d, J=19.4 Hz, 1H), 3.22 (s, 3H), 3.17 (d, J=19.4 Hz, 1H), 1.72 (s, 3H), 1.52 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 173.1, 172.9, 171.6, 166.4, 146.1, 138.4, 129.4, 127.5, 124.5, 118.7, 66.7, 59.9, 52.0, 39.8, 25.0, 22.8, 22.7; HRMS (ESI) calcd for $C_{17}H_{18}ClN_2O_5$ $[M+H]^+$ 365.0899, found 365.0904.

Methyl 2-(6-bromo-1'-methyl-2',3,5'-trioxospiro[isoindoline-1,3'-pyrrolidin]-2-yl)-2-methylpropanoate (**4da**): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70～7.64 (m, 2H), 7.32 (d, J=1.3 Hz, 1H), 3.76 (s, 3H), 3.60 (d, J=19.4 Hz, 1H), 3.22 (s, 3H), 3.18 (d, J=19.4 Hz, 1H), 1.72 (s, 3H), 1.52 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 173.1, 172.7, 171.6, 166.6, 146.2, 132.3, 128.0, 126.6, 124.6, 121.6, 66.6, 59.9, 52.0, 39.7, 25.0, 22.8, 22.6; HRMS (ESI) calcd for $C_{17}H_{18}BrN_2O_5$ $[M+H]^+$ 409.0394, found 409.0398.

4.6 General procedure for the synthesis of product **7** from **1a**

1a (0.1 mmol) and $Cu(OTf)_2$ (2.0 equiv.) were stirred in TFE (1.0 mL) under Ar atmosphere at 100 ℃ for 2 h. After completion, the reaction mixture was purified by column chromatography (petroleum ether/ethyl acetate, V∶V=10∶1) to afford 2,2,2-trifluoroethyl 2-benzamido-2-methylpropanoate (**7**). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77～7.72 (m, 2H), 7.54～7.48 (m, 1H), 7.42 (t, J=7.4 Hz, 2H), 6.57 (s, 1H), 4.53 (q, J=8.4 Hz, 2H), 1.67 (s, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 171.8, 166.0 (d, J=2.3 Hz), 132.8, 130.7, 127.5, 125.9, 125.2～117.8 (m), 60.0 (q, J=36.6 Hz), 55.4, 23.8; HRMS (ESI) calcd for $C_{13}H_{15}F_3NO_3$ $[M+H]^+$ 290.1004, found 290.1006.

Supporting Information Procedure for synthetic transformation of **3aa**, **5aa**, and gram-scale experiment of **3na**, mechanistic experiments, ^{1}H NMR, ^{13}C NMR, ^{19}F NMR and HRMS spectra of **3aa**～**3ai**, **3ba**～**3ja**, and **4aa**～**4da**. The Supporting Information is available free of charge via the Internet at http://sioc-Journal.cn/.

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