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Ruthenium(II)-Catalyzed Homocoupling of Weakly Coordinating Sulfoxonium Ylides via C–H Activation/Annulations: Synthesis of Functionalized Isocoumarins

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Abstract: Homocoupling of weakly coordinating sulfoxonium ylides was accomplished via ruthenium (II) catalyzed C–H activation process. This strategy provides a convenient, efficient and step-economic method to access 3-substituted isocoumarins with good functional group tolerance. The sulfoxonium ylide acts both as the convenient aromatic substrate and the acylmethylation reagent in this transformation. Moreover, the products could be transformed to diverse valuable derivatives.

Keywords: C–H Activation; Ruthenium(II) catalysis; Homocoupling; Isocoumarins; Sulfoxonium ylides

Introduction

Isocoumarins are ubiquitous structural motifs widely existed in natural products, pharmaceuticals, and bioactive compounds.[1–3] Among various isocoumarin derivatives, 3-substituted isocoumarins possess versatile pharmacophores such as anticancer,[4] anti-inflammatory,[5] antifungal,[6] antiallergic,[7] and anti-HIV[8] activities and have been received much attentions (Figure 1). Moreover, they are also valuable synthetic intermediates which can be transformed into useful molecules such as canesin, isochromenes, and alkaloids.[9] Given the substantial applications of isocoumarins, tremendous and persistent efforts have been made to seek efficient methods to assemble this scaffold. Traditional approaches to synthesis 3-substituted isocoumarins generally rely on electrophilic cyclization of substituted alkynes, or the metal-catalyzed 2-haloaromatic acid/esters with terminal alkynes.[10] However, most of these methods suffer from the utilization of halogen functionalized reagents, formation of isomeric mixtures of products, and multi-step synthesis under harsh reaction conditions, which limited their synthetic practicality and utility. Therefore, the development of efficient, expeditious and straightforward strategies for the construction of isocoumarins is highly desirable.

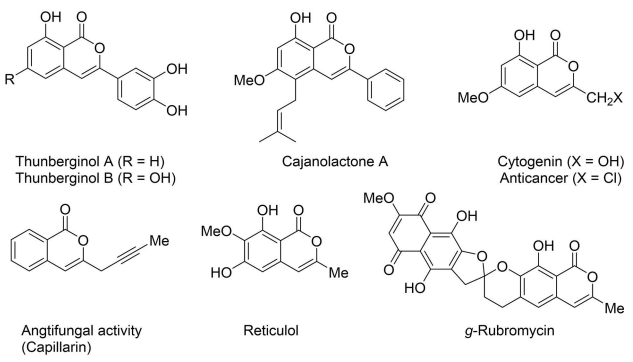


Figure 1. Representative Naturally and Pharmacologically Active 3-Substituted Isocoumarins.

During the past two decades, transition-metal-catalyzed directing group-assisted C–H bond activation have emerged as powerful tool to compose various functionalized molecules in organic synthesis.[11] Meanwhile, the synthesis of isocoumarins has been witnessed the rapid development via Ru, Rh, Co, Pd, or Ir complexes catalyzed C–H bond activations.[12] Owing to the lower cost of $[Ru(p\text{-cymene})Cl_2]_2$ (as compared to Cp*Rh(III), Cp*Ir(III), or Cp*Co(III)

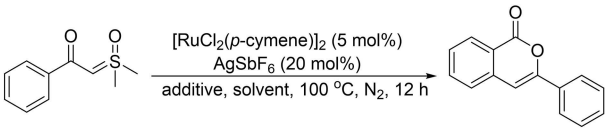
catalysts), the ruthenium(II)-catalyzed C–H activation of arenes has been increasingly explored in recent years.[13] In particular, the ruthenium(II)-catalyzed C–H activation/annulation reaction has been recognized as an important and valuable strategy for the construction of heterocyclic compounds.[14] Despite these considerable advances, very limited efforts have been made to the synthesis of 3-substituted isocouumarins via ruthenium(II)-catalyzed C–H activations. Recently, Ackermann's group described a Ru(II)-catalyzed coupling of aromatic acids with sulfoxonium ylides[15] to synthesis 3-substituted isocouumarins.[12k] Inspired by these work and as part of our interest in C–H activation reactions,[16] we recently developed a Ru(II)-catalyzed homocoupling of weakly coordinating sulfoxonium ylides to synthesize 3-substituted isocouumarins. Therefore, we wish to report the primary results of this work herein.

Results and Discussion

At the outset of our studies, we selected sulfoxonium ylide **1a** as the model substrate to optimize the reaction conditions. Our initial efforts revealed that the reaction could be activated with [Ru(*p*-cymene)Cl_2]$_2$ (5 mol%)/$AgSbF_6$ (20 mol%) in the presence of 1 equiv. AcOH in TFE at 100 °C for 12 h under N_2 (for more details see Table S1 of SI). To further optimize the reaction condition, the variation of additives such as 2,4-Me_2PhCOOH, $AdCO_2H$ (1-Adamantanecarboxylic acid), PivOH, $Zn(OTf)_2$, and $Cu(OAc)_2 \cdot H_2O$ were investigated. Unfortunately, these additives led to only poor conversion of sulfoxonium ylide **1a** (entries 1–7). Screening of solvents indicated that TFE was better than EtOH, DCE, DMF, and HFIP (entries 8–11). When switching the ruthenium catalyst to Rh(III) catalyst ([RhCp*Cl_2]$_2$) or Co(III) catalyst (Cp*Co(CO)I_2), the reaction could not be activated however (entries 12–13). Control experiment indicated that the introduction of an acid additive is necessary for this transformation (entry 14). Likely, the acid additive not only help to facilitate the C–H activation, but also promote the intramolecular cyclization process. Lowing down the temperature to 80 °C resulted in diminished yield (entry 15), and 71% of **2a** was isolated when increasing the temperature to 120 °C (entry 16).

With the optimized reaction conditions in hand, various sulfoxonium ylides were subsequently utilized to investigate the scope and generality of this homocoupling reaction (Table 2). Sulfoxonium ylides bearing electron-donating, halogen, and electron-withdrawing groups at the *para*-position of phenyl ring all coupled smoothly to afford the targeted isocoumarins with moderated to excellent yields (**2b–2h**). The molecular structure of **2b** was unambiguously confirmed by X-ray crystallography (CCDC 1917189).[17]

Table 1. Optimization of the Reaction Conditions[a].



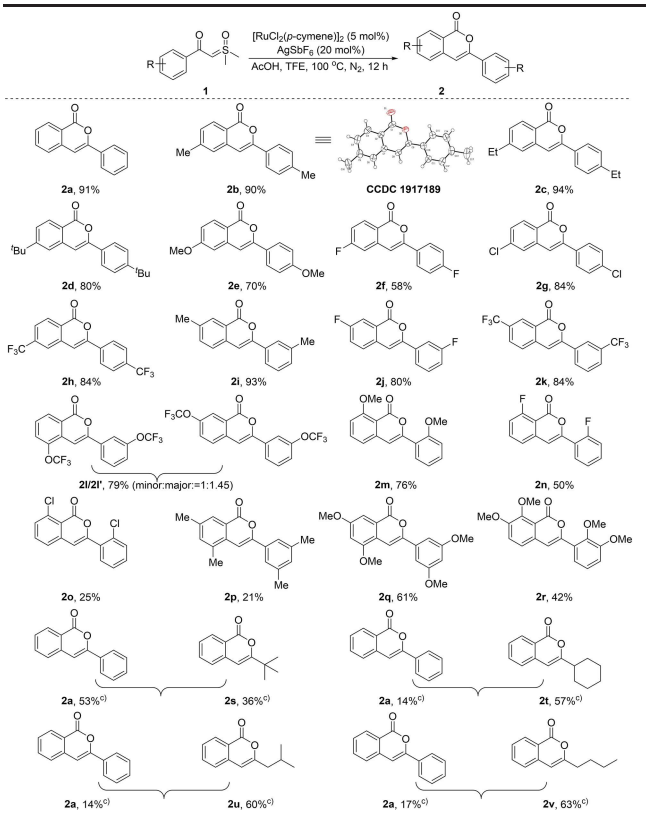
1a 2a

Entry	Additive (equiv.)	Solvent	Yield[b]
1	AcOH (1)	TFE	91
2	AcOH (2)	TFE	76
3	2,4-Me_2PhCOOH (1)	TFE	67
4	$AdCO_2H$ (1)	TFE	85
5	PivOH (1)	TFE	79
6	Zn(OTf)1 (1)	TFE	22
7	$Cu(OAc)_2 \cdot H_2O$ (1)	TFE	70
8	AcOH (1)	EtOH	trace
9	AcOH (1)	DCE	84
10	AcOH (1)	DMF	68
11	AcOH (1)	HFIP	51
12[c]	AcOH (1)	TFE	41
13[d]	AcOH (1)	TFE	trace
14		TFE	trace
15[e]	AcOH (1)	TFE	74
16[f]	AcOH (1)	TFE	71

[a] Reactions were carried out using **1a** (0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol%), $AgSbF_6$ (20 mol%), and additive (0.4 mmol) in a solvent (2 mL) for 12 h.
[b] Isolated yield.
[c] [RhCp*Cl_2]$_2$ (5.0 mol%) was applied as the catalyst.
[d] Cp*Co(CO)I_2 (5.0 mol%) was applied as the catalyst.
[e] 80 °C. [f] 120 °C.

Meta-substituted sulfoxonium ylides bearing methyl, fluroro, chloro, and trifluoromethyl at the benzene rings also proceeded well at the less hindered site, furnishing the annulated products in moderate to excellent yields (**2i–2k**). In the case of *meta*-trifluoromethoxy sulfoxonium ylide, the reaction afforded a mixture of two regioisomeric products **2l** and **2l′** with a total yield of 79% in approximately 1:1.45 ratio. Introduction of electron-donating and halogen groups at the *ortho*-position of phenyl ring could be well tolerated to this transformation (**2m–2o**). Unfortunately, the reactions using thiophene or furan rings did not take place under the standard conditions. Notably, di/tri-substituted sulfoxonium ylides were also viable for this transformation. (**2p–2r**). Besides the homocoupling reaction, we were also curious to examine the cross-coupling mode of sulfoxonium ylides. First, the coupling of **1e** with **1h** was carried out under the standard conditions, and the reaction afforded a mixture of **2e** (20%), **2h** (12%), **2eh** (21%), and **2he** (10%). The molecular structure of **2eh** and **2he** was also confirmed by ^{1}H-^{1}H NOESY spectra (for more details see the SI). In addition, coupling of **1a** with **1s** yielded a mixture of **2a** (53%) and **2s** (36%). When less steric hindered alkyl sufoxonium ylides **1t–1v**

Table 2. Scope of Sulfoxonium Ylides[a]



[a] Reactions were carried out using **1** (0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol%), $AgSbF_6$ (20 mol%), and AcOH (0.2 mmol) in TFE (2 mL) for 12 h.
[b] Isolated yield.
[c] Reactions were carried out using **1a** (0.2 mmol), **1s** (0.8 mmol).

were applied, the reaction delivered **2t–2v** as the major cross-coupling products in good yields.

Based on the above studies, we next investigated the synthetic applications of the obtained functionalized isocouumarins. In the presence of BBr_3, isocouumarins **2q** could be deprotected to the analogous thunberginol **3q** in 48% yield (Figure 2, eq 1).[18] Under the Grignard reaction condition, 3-substituted isocoumarin **2a** could be nicely transferred to the corresponding benzophenone **4a** (Figure 2, eq 2).[12j] Ammoniation of **2a** in the presence of an excess amount of aqueous ammonia readily afforded the corresponding 3-phenylisoquinolin-1(*2H*)-one **5a** in 95% yield (Figure 2, eq 3).[19] Treatment of **2a** with hydrazine hydrate delivered the ring-expanding product in 86% yield (Figure 2, eq 4).[20] To get some insights into the reaction mechanism, control experiment was performed (Figure 2, eq 5). The H/D exchange homocoupling of **1a** under the standard conditions was carried out in the presence of CD_3OD, and the result suggests that the C–H activation is

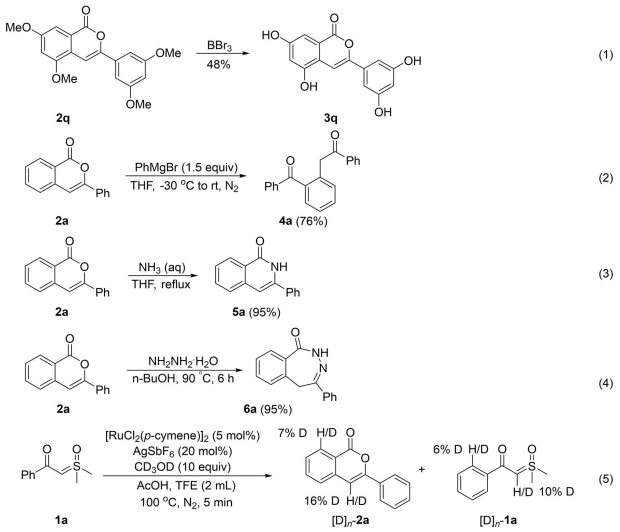
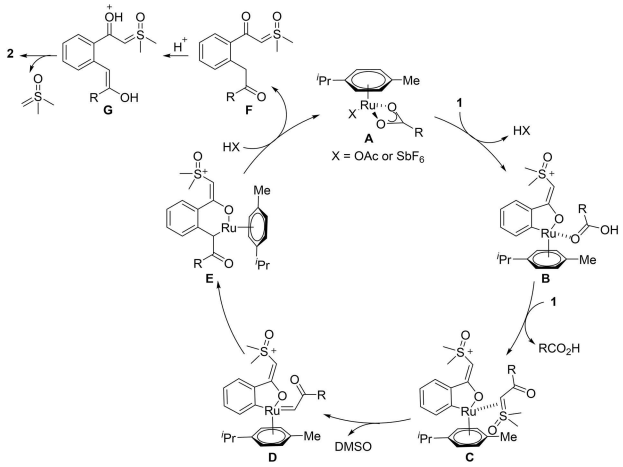


Figure 2. Derivatives of 3-Substituted Isocouumarins and Deuterium Labeling Experiment.

reversible.[12j] Furthermore, the observed deuterium incorporation at the olefinic position of $[D]_n$-**2a** may suggest enolization of the alkylation intermediate **F**, which may due to the enhanced acidity of the methylene protons.

On the basis of the above experimental results and related reports,[12j,k,21] a plausible mechanism is proposed as shown in Scheme 1. First, cyclometalation of sulfoxonium ylide **1** gives a metalacyclic intermdiate **B**. Next, the coordination of **B** with sulfoxonium ylide **1** affords ruthenium(II) species **C**, which is then transformed into a key ruthenium(II) carbene species **D** by α-elimination of DMSO. The subsequent migratory insertion of the Ru–C(Ar) bond into carbene moiety of **D** generates a six-membered ruthenacyclic intermediate **E**. Then, **E** undergoes protonolysis to



Scheme 1. Postulated Mechanism.

form an alkylation intermediate **F**, and further transfers to intermediate **G** in the presence of acid. Finally, an intramolecular nucleophilic attack of the enol oxygen furnishes isocouumarin **2**, meanwhile releasing a Corey's ylide.[22] The formation of benzyloxirane was confirmed by trapping with phenylacetaldehyde, suggesting that the Corey's ylide could form in our reaction system (for more details see the SI).[23]

Conclusion

In conclusion, we have developed a ruthenium(II)-catalyzed homocoupling of weakly coordinating sulfoxonium ylides involving C–H activation/intramolecular nucleophilic addition/elimination process. This reaction protocol provides a simple, efficient, step-economic approach with excellent functional group compatibility to access synthetically and pharmaceutically valuable 3-substituted isocouumarins. More importantly, the sufoxonium ylide acts both as the convenient aromatic substrate and the acylmethylation reagents in this transformation.

Experimental Section

General Procedure for Synthesis of 2

A pressure tube was charged with $[RuCl_2(p\text{-cymene})]_2$ (5 mol%), $AgSbF_6$ (20 mol%), AcOH (1 equiv.), sulfoxonium ylides (1, 0.4 mmol) and TFE (1 mL). The reaction mixture was stirred under N_2 condition at 100 °C for 12 h. Then, the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography with PE/EA to afford the product **2**.

3-Phenyl-1*H*-isochromen-1-one (2a);[12j] White solid, 40.2 mg, yield: 91%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, J = 8.4 Hz, 1H), 7.88–7.85 (m, 2H), 7.72–7.68 (m, 1H), 7.50–7.41 (m, 5H), 6.94 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 153.6, 137.5, 134.9, 132.0, 130.0, 129.6, 128.8, 128.2, 126.0, 125.2, 120.5, 101.8.

6-Methyl-3-(*p*-tolyl)-1*H*-isochromen-1-one (2b);[10b] White solid, 45.0 mg, yield: 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 8.4 Hz, 2H), 7.29–7.24 (m, 4H), 6.83 (s, 1H), 2.47 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 153.9, 145.9, 140.2, 137.8, 129.6, 129.5, 129.4, 129.3, 125.8, 125.2, 118.1, 101.1, 22.0, 21.4.

6-Ethyl-3-(4-ethylphenyl)-1*H*-isochromen-1-one (2c); White solid, m.p. 55–56 °C, 52.2 mg, yield: 94%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 8.4 Hz, 1H), 7.80 (d, J = 8.0 Hz, 2H), 7.33–7.28 (m, 4H), 6.88 (s, 1H), 2.77 (q, J = 7.6 Hz, 2H), 2.70 (q, J = 7.6 Hz, 2H), 1.31 (t, J = 7.6 Hz, 3H), 1.27 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 153.9, 152.0, 146.4, 137.9, 129.7, 129.6, 128.4, 128.3, 125.3, 124.6, 118.2, 101.3, 29.2, 28.7, 15.4, 15.1. HRMS (ESI): calcd for $C_{19}H_{18}O_2$ ([M + Na]$^+$) 301.1199, found 301.1200.

6-(*tert*-Butyl)-3-(4-(*tert*-butyl)phenyl)-1*H*-isochromen-1-one (2d); White solid, m.p. 139–140 °C, 53.2 mg, yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.4 Hz, 1H), 7.83 (d, J = 8.8 Hz, 2H), 7.54 (dd, J = 8.4, 2.0 Hz, 1H), 7.49–7.46 (m, 3H), 6.93 (s, 1H), 1.39 (s, 9H), 1.36 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 158.8, 153.7, 153.3, 137.7, 129.4, 129.3, 126.0, 125.8, 125.0, 122.2, 118.0, 101.7, 35.4, 34.9, 31.2, 31.0. HRMS (ESI): calcd for $C_{23}H_{26}O_2$ ([M + Na]$^+$) 357.1825, found 357.1824.

6-Methoxy-3-(4-methoxyphenyl)-1*H*-isochromen-1-one (2e); White solid, m.p. 146–147 °C, 39.3 mg, yield: 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 8.8 Hz, 1H), 7.80 (d, J = 8.8 Hz, 2H), 7.00–6.94 (m, 3H), 6.82 (d, J = 2.4 Hz, 1H), 6.75 (s, 1H), 3.91 (s, 3H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 162.2, 161.1, 154.2, 140.2, 131.8, 126.8, 124.6, 116.2, 114.2, 113.3, 107.6, 100.3, 55.6, 55.4. HRMS (ESI): calcd for $C_{17}H_{14}O_4$ ([M + Na]$^+$) 305.0784, found 305.0779.

6-Fluoro-3-(4-fluorophenyl)-1*H*-isochromen-1-one (2f); White solid, m.p. 158–159 °C, 30 mg, yield: 58%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (dd, J = 8.8, 5.6 Hz, 1H), 7.90–7.84 (m, 2H), 7.22–7.13 (m, 4H), 6.85 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8 (d, J = 255.2 Hz), 164.0 (d, J = 250.1 Hz), 161.2, 154.0, 140.1 (d, J = 10.8 Hz), 133.1 (d, J = 10.4 Hz), 127.8 (d, J = 3.3 Hz), 127.5 (d, J = 8.6 Hz), 116.8 (d, J = 2.2 Hz), 116.5 (d, J = 23.1 Hz), 116.1 (d, J = 21.9 Hz), 111.5 (d, J = 22.4 Hz), 101.0 (dd, J = 2.7, 1.7 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ −109.5, −101.6. HRMS (ESI): calcd for $C_{15}H_8F_2O_2$ ([M + Na]$^+$) 281.0385, found 281.0383.

6-Chloro-3-(4-chlorophenyl)-1*H*-isochromen-1-one (2g); White solid, m.p. 227–228 °C, 48.7 mg, yield: 84%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 8.4 Hz, 1H), 7.81 (d, J = 8.4 Hz, 2H), 7.49–7.44 (m, 4H), 6.86 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3, 153.9, 141.7, 138.7, 136.5, 131.4, 130.0, 129.2, 128.9, 126.7, 125.5, 118.8, 101.1. HRMS (ESI): calcd for $C_{15}H_8Cl_2O_2$ ([M + Na]$^+$) 312.9794, found 312.9798.

6-(Trifluoromethyl)-3-(4-(trifluoromethyl)phenyl)-1*H*-isochromen-1-one (2h); White solid, m.p. 175–176 °C, 52.9 mg, yield: 74%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, J = 8.4 Hz, 1H), 8.00 (d, J = 8.0 Hz, 2H), 7.81 (s, 1H), 7.77–7.73 (m, 3H), 7.10 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6, 153.4, 137.3, 136.6, (q, J = 32.6 Hz) 134.6, 132.2 (q, J = 32.6 Hz), 130.8, 126.0 (q, J = 3.8 Hz), 125.7, 125.0 (q, J = 3.2 Hz), 123.7 (q, J = 270.8 Hz), 123.4 (q, J = 3.6 Hz), 123.2 (q, J = 271.7 Hz), 123.1, 102.7. ^{19}F NMR (377 MHz, $CDCl_3$) δ −62.9, −63.5. HRMS (ESI): calcd for $C_{17}H_8F_6O_2$ ([M + Na]$^+$) 381.0321, found 381.0327.

7-Methyl-3-(*m*-tolyl)-1*H*-isochromen-1-one (2i);[10b] White solid, 46.3 mg, yield: 93%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.69 (s, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.53–7.50 (m, 1H), 7.37 (d, J = 8.0 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 7.21 (d, J = 7.6 Hz, 1H), 6.90 (s, 1H), 2.45 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 153.0, 138.5, 138.4, 136.2, 135.1, 132.0, 130.6, 129.3, 128.7, 125.9, 125.7, 122.2, 120.4, 101.7, 21.5, 21.4.

7-Fluoro-3-(3-fluorophenyl)-1*H*-isochromen-1-one (2j); White solid, m.p. 165–166 °C, 41 mg, yield: 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, J = 7.2, 2.0 Hz, 1H), 7.68 (d, J = 8.0 Hz, 1H), 7.59 (dt, J = 10.0, 2.0 Hz, 1H), 7.50–7.41 (m, 3H), 7.16–7.11 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (d, J = 245.3 Hz), 160.6 (d, J = 3.6 Hz), 157.3 (d, J = 251.5 Hz), 152.8

(d, J=4.0 Hz), 133.8 (d, J=8.1 Hz), 130.5 (d, J=8.2 Hz), 128.9 (d, J=7.7 Hz), 126.3 (d, J=16.7 Hz), 125.4 (d, J=3.9 Hz), 122.0 (d, J=3.8 Hz), 121.0 (d, J=3.1 Hz), 120.4 (d, J=19.6 Hz), 117.2 (d, J=21.2 Hz), 112.4 (d, J=23.8 Hz), 95.1 (d, J=5.0 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ −111.8, −120.7. HRMS (ESI): calcd for $C_{15}H_8F_2O_2$ ([M+Na]$^+$) 281.0385, found 281.0384.

7-(Trifluoromethyl)-3-(3-(trifluoromethyl)phenyl)-1*H*-isochromen-1-one (2 k); White solid, m.p. 169–170 °C, 59.8 mg, yield: 84%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 8.13 (s, 1H), 8.08 (d, J=8.0 Hz, 1H), 7.96 (dd, J=8.4, 2.0 Hz, 1H), 7.73 (d, J=7.6 Hz, 1H), 7.67 (d, J=8.0 Hz, 1H), 7.63 (t, J=8.0 Hz, 1H), 7.08 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 154.0, 139.7, 132.2, 132.7 (q, J=32.6 Hz), 131.4 (q, J=3.4 Hz), 130.7 (q, J=33.5 Hz), 129.6, 128.6, 127.3 (q, J=4.0 Hz), 127.2 (q, J=3.7 Hz), 127.0, 123.7 (q, J=270.9 Hz), 123.3 (q, J=270.8 Hz), 122.3 (q, J=3.8 Hz), 120.8, 102.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ −62.80, −62.83; HRMS (ESI): calcd for $C_{17}H_8F_6O_2$ ([M+Na]$^+$) 381.0321, found 381.0321.

7-(Trifluoromethoxy)-3-(3-(trifluoromethoxy)phenyl)-1*H*-isochromen-1-one (2 l); White solid, m.p. 103–104 °C, 24.6 mg, yield: 32%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, J=7.6 Hz, 1H), 7.86–7.83 (m, 1H), 7.75 (s, 1H), 7.67 (dt, J=8.0, 1.6 Hz, 1H), 7.56 (d, J=8.0 Hz, 1H), 7.52 (d, J=8.0 Hz, 1H), 7.35–7.32 (m, 1H), 7.18 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 153.2, 149.8, 143.9, 133.7, 130.8, 130.5, 128.7, 128.3, 126.3, 123.8, 122.7, 122.3, 120.6 (q, J=258.1 Hz), 120.4 (q, J=256.5 Hz), 118.4, 96.0. ^{19}F NMR (377 MHz, $CDCl_3$) δ −57.7, −57.8. HRMS (ESI): calcd for $C_{17}H_8F_6O_4$ ([M+Na]$^+$) 413.0219, found 413.0216.

5-(Trifluoromethoxy)-3-(3-(trifluoromethoxy)phenyl)-1*H*-isochromen-1-one (2 l'); White solid, m.p. 115–116 °C, 35.6 mg, yield: 47%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.81 (d, J=8.4 Hz, 1H), 7.71 (s, 1H), 7.59–7.58 (m, 2H), 7.51 (t, J=8.4 Hz, 1H), 7.32–7.29 (m, 1H), 6.99 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 152.4, 149.8 (q, J=1.8 Hz), 148.8 (q, J=1.9 Hz), 135.6, 133.6, 130.5, 128.2, 123.6, 122.6, 122.0, 121.2, 121.1, 120.4 (q, J=256.3 Hz), 120.3 (q, J=257.4 Hz), 118.0, 101.8. ^{19}F NMR (377 MHz, $CDCl_3$) δ −57.8, −58.0. HRMS (ESI): calcd for $C_{17}H_8F_6O_4$ ([M+Na]$^+$) 413.0219, found 413.0218.

8-Methoxy-3-(2-methoxyphenyl)-1*H*-isochromen-1-one (2 m); White solid, m.p. 168–169 °C, 43 mg, yield: 76%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (dd, J=7.6, 1.6 Hz, 1H), 7.59 (t, J=8.0 Hz, 1H), 7.38–7.34 (m, 1H), 7.28 (s, 1H), 7.07–7.02 (m, 2H), 6.98 (d, J=8.0 Hz, 1H), 6.92 (d, J=8.0 Hz, 1H), 4.01 (s, 3H), 3.95 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.5, 159.4, 157.2, 150.7, 141.0, 135.5, 130.7, 128.9, 120.8, 120.6, 118.5, 111.3, 109.7, 109.4, 107.0, 56.3, 55.6. HRMS (ESI): calcd for $C_{17}H_{14}O_4$ ([M+Na]$^+$) 305.0784, found 305.0790.

8-Fluoro-3-(2-fluorophenyl)-1*H*-isochromen-1-one (2 n); White solid, m.p. 171–172 °C, 25.7 mg, yield: 50%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (td, J=8.0, 2.0 Hz, 1H), 7.68 (td, J=8.0, 5.2 Hz, 1H), 7.43–7.38 (m, 1H), 7.31–7.25 (m, 2H), 7.20–7.14 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (d, J=265.2 Hz), 160.1 (d, J=251.6 Hz), 157.5 (d, J=5.4 Hz), 149.0 (d, J=4.9 Hz), 140.0, 136.3 (d, J=10.1 Hz), 131.5 (d, J=9.1 Hz), 128.6 (d, J=1.7 Hz), 124.7 (d, J=3.6 Hz), 122.3 (d, J=4.3 Hz), 119.6 (d, J=9.7 Hz), 116.5 (d, J=22.7 Hz), 115.7 (d, J=21.1 Hz), 109.6 (d, J=7.4 Hz), 106.5 (dd, J=16.0, 3.0 Hz). ^{19}F NMR (377 MHz, CDCl3) δ −107.1, −111.7. HRMS (ESI): calcd for $C_{15}H_8F_2O_2$ ([M+Na]$^+$) 281.0385, found 281.0383.

8-Chloro-3-(2-chlorophenyl)-1*H*-isochromen-1-one (2 o); White solid, m.p. 150–151 °C, 14.2 mg, yield: 25%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75–7.71 (m, 1H), 7.62–7.54 (m, 2H), 7.52–747 (m, 1H), 7.41–7.37 (m, 3H), 6.96 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 152.0, 140.0, 137.2, 134.6, 132.3, 131.5, 131.0, 130.9, 130.7, 130.6, 127.1, 125.2, 117.8, 107.5. HRMS (ESI): calcd for $C_{15}H_8Cl_2O_2$ ([M+Na]$^+$) 312.9794, found 312.9796.

3-(3,5-Dimethylphenyl)-5,7-dimethyl-1*H*-isochromen-1-one (2 p); White solid, m.p. 213–214 °C, 14.2 mg, yield: 21%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.51 (s, 2H), 7.38 (s, 1H), 7.06 (s, 1H), 7.01 (s, 1H), 2.53 (s, 3H), 2.43 (s, 3H), 2.39 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1, 152.7, 138.4, 137.7, 137.2, 133.9, 133.4, 132.3, 131.5, 127.3, 123.0, 120.5, 98.5, 21.4, 21.3, 18.8. HRMS (ESI): calcd for $C_{19}H_{18}O_2$ ([M+Na]$^+$) 301.1199, found 301.1193.

3-(3,5-Dimethoxyphenyl)-5,7-dimethoxy-1*H*-isochromen-1-one (2 q); White solid, m.p. 195–196 °C, 41.5 mg, yield: 61%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, J=2.4 Hz, 1H), 7.22 (s, 1H), 7.00 (d, J=2.4 Hz, 2H), 6.73 (d, J=2.4 Hz, 1H), 6.49 (t, J=2.4 Hz, 1H), 3.93 (s, 3H), 3.91 (s, 3H), 3.86 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 161.0, 160.3, 155.7, 150.8, 134.4, 122.5, 122.1, 105.1, 102.9, 101.9, 101.0, 96.8, 56.0, 55.9, 55.6. HRMS (ESI): calcd for $C_{19}H_{18}O_6$ ([M+Na]$^+$) 365.0996, found 365.0989.

3-(2,3-Dimethoxyphenyl)-7,8-dimethoxy-1*H*-isochromen-1-one (2 r); White solid, m.p. 129–130 °C, 28.5 mg, yield: 42%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (dd, J=8.0, 1.6 Hz, 1H), 7.35 (d, J=8.4 Hz, 1H), 7.25 (s, 1H), 7.23 (d, J=8.4 Hz, 1H), 7.14 (t, J=8.0 Hz, 1H), 6.97 (dd, J=8.0, 1.6 Hz, 1H), 4.01 (s, 3H), 3.95 (s, 3H), 3.92 (s, 3H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 153.2, 152.8, 150.9, 148.6, 147.1, 132.5, 126.4, 124.3, 122.3, 120.3, 120.0, 115.3, 113.4, 106.5, 61.6, 60.5, 56.7, 56.0. HRMS (ESI): calcd for $C_{19}H_{18}O_6$ ([M+Na]$^+$) 365.0996, found 365.0999.

3-(*tert*-Butyl)-1*H*-isochromen-1-one (2 s);[12j] Yellow oil, 15.0 mg, yield: 36%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27–8.25 (m, 1H), 7.69–7.65 (m, 1H), 7.47–7.38 (m, 1H), 7.39 (d, J=7.6 Hz, 1H), 6.31 (s, 1H), 1.33 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 163.1, 137.7, 134.6, 129.4, 127.6, 125.5, 120.1, 99.7, 35.6, 28.0.

3-Cyclohexyl-1*H*-isochromen-1-one (2 t);[10h] White solid, 25.8 mg, yield: 57%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, J=8.0 Hz, 1H), 7.66 (t, J=7.6 Hz, 1H), 7.44 (t, J=7.6 Hz, 1H), 7.36 (d, J=7.6 Hz, 1H), 6.23 (s, 1H), 2.47–2.41 (m, 1H), 2.05–1.73 (m, 5H), 1.50–1.23 (m, 5H).^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 162.4, 137.8, 134.7, 129.5, 127.5, 125.3, 120.3, 100.9, 41.9, 30.6, 26.0, 25.9.

3-Isobutyl-1*H*-isochromen-1-one (2 u);[10f] Yellow solid, 24.3 mg, yield: 60%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27–8.24 (m, 1H), 7.70–7.66 (m, 1H), 7.47–7.43 (m, 1H), 7.36 (d, J=8.0 Hz, 1H), 6.25 (s, 1H), 2.39 (d, J=7.6 Hz, 2H), 2.20–2.10

(m, 1H), 0.98 (d, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 157.4, 137.6, 134.7, 129.5, 127.6, 125.0, 120.1, 104.0, 42.8, 26.6, 22.3.

3-Butyl-1*H*-isochromen-1-one (2v);[12g] Colorless liquid, 25.4 mg, yield: 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26–8.24 (m, 1H), 7.70–7.65 (m, 1H), 7.47–7.43 (m, 1H), 7.35 (d, J = 8.0 Hz, 1H), 6.26 (s, 1H), 2.53 (t, J = 7.6 Hz, 2H), 1.73–1.67 (m, 2H), 1.45–1.36 (m, 2H), 0.95 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 158.3, 137.7, 134.7, 129.5, 127.5, 125.0, 120.1, 102.9, 33.2, 29.0, 22.1, 13.8.

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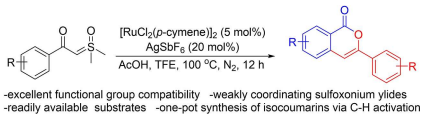
FULL PAPER

Ruthenium(II)-Catalyzed Homocoupling of Weakly Coordinating Sulfoxonium Ylides via C–H Activation/Annulations: Synthesis of Functionalized Isocoumarins



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