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CF₃SOCl-promoted intramolecular cyclization of β -diketones: An efficient synthesis of flavones

Dong-Wei Sun ^{a, b}, Yong-Yan Zhou ^{a, **}, Min Jiang ^b, Tang Nian ^a, Jin-Tao Liu ^{b, *}

^a Key Laboratory of Sulfur Hexafluoride of China Southern Power Grid Co., Ltd., Electric Power Research Institute of Guangdong Power Grid Co., Ltd., Guangzhou, Guangdong, 510080, China

^b Key Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Road, Shanghai, 200032, China

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ABSTRACT

An efficient intramolecular cyclization reaction of β -diketones containing a phenyl group with an orthohydroxyl substituent was achieved. Using CF₃SOCl as an additive, the reaction took place under transition-metal-free and mild conditions. A series of flavones were synthesized in moderate to excellent yields.

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1. Introduction

Flavonoids have attracted considerable attention in recent decades owing to their important role as key structural units in many natural products [1] with pharmaceutical and biological activities including bactericidal, anti-cancer, anti-viral, anti-oxidants, anti-HIV, ovipositor stimulant phytoalexins, DNA cleavage, vasodilator, antimutagenic, anti-inflammatory and anti-allergic [2]. At present, more than 10000 flavonoids have been identified from plants, and the number is increasing rapidly [3]. Among various flavonoids, flavones play an important role in the field of medicine, and many drug molecules contain the chromone skeleton (2-phenyl-4*H*chromen-4-one), such as Flavone-8-acetic acid [4], Flavoxate [5], Flavopirydol [6], Nobiletin [7], and Luteolin [8] (Fig. 1). Therefore, the development of efficient protocol for the synthesis of flavones is of great significance for drug development.

Over the past decade, various efficient approaches towards the synthesis of different flavones have been developed. So far, flavones can be made from 1,3-diketones [9], chalcones [10], and salicy-laldehydes [11]. Among these methods, the intramolecular

https://doi.org/10.1016/j.tet.2021.132226 0040-4020/© 2021 Elsevier Ltd. All rights reserved. cyclization of 1-(2-hydroxyphenyl)-3-arylpropane-1,3-diones is an important one. Usually, an promoter was needed in this cyclization reaction, such as sulfuric acid/glacial acetic acid [12], Co^{III}(salpr)(OH) [13], FeCl₃ [14], P₂O₅ [15], CuCl₂ [16], ZnO nanoparticles [17], Wells-Dawson heteropolyacids [18] and silica-supported Preyssler heteropolyacid [19]. However, most methods have more or less deficiencies, such as complex reaction conditions, low yield, and the use of toxic or expensive reagents. Therefore, it is still desirable to develop new synthetic methods for flavones. During our study on the base-promoted trifluoromethylthiolation/bifunctionalization reaction of 1,3-diketones with trifluoromethanesulfinyl chloride (CF₃SOCl) [20] (Scheme 1, eq 1), it was surprisingly found that the reaction of 1-(2-hydroxyphenyl)-3aylpropane-1,3-dione in the absence of base gave 2-phenyl-4Hchromen-4-one as the major product (Scheme 1, eq 2). Considering the importance of flavones, we studied this reaction in detail, and the results are reported in this paper.

2. Results and discussion

Initially, 1-(2-hydroxyphenyl)-3-phenylpropane-1,3-dione (**1a**) was treated with 1.2 equivalent of CF_3SOCI in dichloromethane (DCM) at room temperature for 10 h. It was found that instead of the expected trifluoromethylthiolation/bifunctionalization

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^{*} Corresponding author.

^{**} Corresponding author.

E-mail address: jtliu@sioc.ac.cn (J.-T. Liu).

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Fig. 1. Representative natural products and drug molecules.



Scheme 1. Previous and this work.

Table 1

Optimization of the reaction conditions.



entry ^a	additive	1a/additive	solvent	yield (2a , %) ^b
1	CF ₃ SOCl	1/1.2	DCM	65
2	CF ₃ SOCl	1/1.5	DCM	81
3	CF ₃ SOCl	1/2.0	DCM	89
4	CF ₃ SOCl	1/2.5	DCM	87
5	CF ₃ SOCl	1/3.0	DCM	85
6	CF ₃ SOCl	1/2.0	toluene	73
7	CF ₃ SOCl	1/2.0	THF	51
8	CF ₃ SOCl	1/2.0	DMF	80
9	CF ₃ SOCl	1/2.0	DMSO	24
10	CF ₃ SOCl	1/2.0	CH ₃ CN	70
11	CF ₃ SOCl	1/2.0	DCE	71
12	-	-	DCM	NR
13	CF ₃ SO ₂ Cl	1/2.0	DCM	84
14	TsCl	1/2.0	DCM	46
15	MsCl	1/2.0	DCM	38
16	$(COCl)_2$	1/2.0	DCM	20
17	SOCl ₂	1/2.0	DCM	26

 $^a\,$ Reaction conditions: $1a\,(0.2\,mmol),$ CF_3SOCI (0.24–0.6 mmol), solvent (0.5 mL), room temperature.

^b Isolated yield.

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product, intramolecular cyclization product 2a was obtained as major product (65% yield, Table 1, entry 1). Inspired by this result, we commenced the screening of different conditions to find a suitable condition to improve the yield of 2a. Changing the ratio of reactants indicated that the yield of 2a could be improved to 89% with a ratio of 1:2.0 (1a/CF₃SOCI) (entries 2–3). However, lower vield of **2a** was obtained when the amount of CF₃SOCl was further increased (entries 4-5). When other solvents such as toluene. tetrahydrofuran (THF), N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), acetonitrile and 1,2-dichloromethane were used instead of DCM, the yield of **2a** dropped remarkably (entries 6–11). Control experiments indicated that additive CF₃SOCl was important for the reaction (entry 12). Other additives, such as trifluoromathanesulfonyl chloride (CF₃SO₂Cl), tosyl chloride (TsCl), methanesulfonyl chloride (MsCl), oxalyl chloride [(COCl)₂] and thionyl chloride (SOCl₂), were also tried, but could not give better result (entries 13-17). Therefore, the optimal conditions for the formation of 2a were set as follows: 1.0 equiv of 1a, 2.0 equiv of CF₃SOCl, in DCM at room temperature.

With the optimized reaction conditions in hand, we next studied the scope of β -diketones **1** containing a phenyl group with an ortho-hydroxyl substituent. As shown in Scheme 2, a series of β diketones 1 containing both electron-withdrawing and electrondonating groups could react smoothly with CF₃SOCl to give the corresponding cyclization products in moderate to high yields. The substituent in the phenyl group has a great influence on the yield of **2**. For example, high yields were obtained with substrates bearing methyl (2b-2d, 2l-2m) or methoxy (2e-2g) group, but the reaction of halogen substituted B-diketones gave the corresponding products 2h-2i, 2n-2p in moderate yields. Only 48% yield was obtained with β -diketone **1k** containing a trifluoromethyl group. Furthermore, almost no desired product was formed in the reaction of β diketone 1j containing a nitro substituent, with a large amount of 1j recovered. It is worth mentioning that β -diketone **1q** containing a furyl group was also a good substrate for this reaction, affording the expected product **2q** in good yield.

Based on previous research [20] and above experimental results, a plausible mechanism was proposed for this intramolecular cyclization reaction as showed in Scheme 3. It is known that β -diketones exist in both keto and enol forms. The enol tautomer reacted with CF₃SOCl to give intermediate **A**. Subsequent intramolecular nuclephilic attack of oxygen atom in the *orth*-hydroxyl group on the carbon connecting OS(O)CF₃ group afforded cyclization product **2** with the release of a CF₃SO₂H molecule.

3. Conclusion

In conclusion, we have developed an efficient protocol for the synthesis of flavones. Using readily available CF₃SOCl as a promoter, intramolecular cyclization of β -diketones containing a phenyl group with an *ortho*-hydroxyl substituent was achieved under transition-metal-free and mild conditions. A series of flavones prepared by this method with high yields demonstrated its potential applications in organic synthesis and drug development.

4. Experimental

4.1. General

Unless otherwise mentioned, solvents and reagents were purchased from commercial sources and used as received. Solvents were freshly distilled by standard procedure prior to use. ¹H NMR spectra were recorded in CDCl₃ on a Bruker AM 400 spectrometer (400 MHz) with TMS as internal standard. ¹⁹F NMR spectra were taken on a Bruker AM 400 (376 MHz) spectrometer with CFCl₃ as

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[a] Reaction conditions: 1 (0.2 mmol), CF₃SOCl (0.4 mmol), DCM (0.5 mL), rt, 10 h, isolated yield.

Scheme 2. Scope of the intramolecular cyclization reaction of 1^a.

external standard.

4.2. General procedure for the preparation of substrates [20]

Acyl chloride (10 mmol, 2.0 equiv) was added to a stirred solution of 2-hydroxyacetophenone (5 mmol) dissolved in pyridine (5 mL) at room temperature. After 2-hydroxyacetophenone was consumed in about 2 h as determined by TLC, a large excess of ice water was added, and then the pH of the solution was adjusted to 6.0 with concentrated HCl. The precipitated solid was collected by



Scheme 3. Proposed mechanism for the intramolecular cyclization of β -diketones.

filtration, giving crude ester products S-1.

To a solution of S-1 (2.5 mmol) in pyridine (10 mL) was added NaOH (3.75 mmol, 1.5 equiv). The mixture was stirred at 50 °C for about 2 h. After the reaction was finished as indicated by TLC, a large excess of ice water was added, and the pH of the solution was adjusted to 6.0 with concentrated HCl. The resulting mixture was extracted with ethyl acetate for 3 times. The combined organic phase was washed with brine, dried over anhydrous Na₂SO₄, filtrated, and concentrated under reduced pressure. The residue was purified by recrystallization from alcohol to give the desired product.

4.3. General procedure for the intramolecular cyclization reaction

A solution of 1-(2-hydroxyphenyl)-3-phenylpropane-1,3-dione (0.2 mmol, **1a**) and CF₃SOCl (0.4 mmol, 2.0 equiv) in DCM (0.5 mL) was stirred at room temperature for 10 h. After the reaction was completed, the reaction mixture was quenched with ice water. The resulting mixture was extracted with DCM for three times. The combined organic solution was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 3:1 v/v) to give product **2a**.

4.3.1. 2-Phenyl-4H-chromen-4-one (2a)

White solid, 39.6 mg, yield: 89%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, J = 7.6 Hz, 1H), 7.93 (d, J = 5.0 Hz, 2H), 7.70 (t, J = 7.6 Hz, 1H), 7.61–7.49 (m, 4H), 7.42 (t, J = 7.2 Hz, 1H), 6.83 (s, 1H).

4.3.2. 2-(o-Tolyl)-4H-chromen-4-one (2b)

White solid, 43.0 mg, yield: 91%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.28–8.20 (m, 1H), 7.73–7.64 (m, 1H), 7.56–7.46 (m, 2H), 7.45–7.37 (m, 2H), 7.34–7.28 (m, 2H), 6.48 (s, 1H), 2.48 (s, 3H).

4.3.3. 2-(m-Tolyl)-4H-chromen-4-one (2c)

White solid, 43.9 mg, yield: 93%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.22 (dd, J = 8.0, 1.6 Hz, 1H), 7.73–7.67 (m, 3H), 7.57 (d, J = 7.9 Hz, 1H), 7.43–7.39 (m, 2H), 7.34 (d, J = 7.9 Hz, 1H), 6.81 (s, 1H), 2.45 (s, 3H).

4.3.4. 2-(p-Tolyl)-4H-chromen-4-one (2d)

White solid, 44.4 mg, yield: 94%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.21 (s, 1H), 7.81 (s, 2H), 7.62 (d, J = 47.1 Hz, 2H), 7.40–7.31 (m, 3H), 6.80 (s, 1H), 2.43 (s, 3H).

4.3.5. 2-(2-Methoxyphenyl)-4H-chromen-4-one (**2e**)

White solid, 46.9 mg, yield: 93%. This is a known compound.[10]

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¹H NMR (400 MHz, CDCl₃) δ 8.22 (dd, J = 8.0, 1.6 Hz, 1H), 7.90 (dd, J = 7.8, 1.7 Hz, 1H), 7.67 (ddd, J = 8.6, 7.1, 1.7 Hz, 1H), 7.52 (d, J = 7.9 Hz, 1H), 7.50–7.44 (m, 1H), 7.40 (dd, J = 11.0, 4.0 Hz, 1H), 7.14 (s, 1H), 7.13–7.07 (m, 1H), 7.04 (d, J = 8.4 Hz, 1H), 3.93 (s, 3H).

4.3.6. 2-(3-Methoxyphenyl)-4H-chromen-4-one (2f)

White solid, 47.4 mg, yield: 94%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, J = 6.4 Hz, 1H), 7.69 (s, 1H), 7.59–7.47 (m, 2H), 7.43 (s, 3H), 7.08 (s, 1H), 6.81 (s, 1H), 3.89 (s, 3H).

4.3.7. 2-(4-Methoxyphenyl)-4H-chromen-4-one (2g)

White solid, 48.4 mg, yield: 96%. This is a known compound.[10] ¹H NMR (400 MHz, CDCl₃) δ 8.28–8.12 (m, 1H), 7.88 (d, *J* = 8.9 Hz, 2H), 7.70–7.61 (m, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 8.9 Hz, 2H), 6.74 (s, 1H), 3.88 (s, 3H).

4.3.8. 2-(4-Fluorophenyl)-4H-chromen-4-one (2h)

White solid, 35.6 mg, yield: 74%. This is a known compound.[11] ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 7.9 Hz, 1H), 7.93 (dd, *J* = 8.7, 5.2 Hz, 2H), 7.70 (t, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 8.3 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 8.5 Hz, 2H), 6.77 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ –107.46 to –107.54 (m, 1F).

4.3.9. 2-(4-Chlorophenyl)-4H-chromen-4-one (2i)

White solid, 41.6 mg, yield: 81%. This is a known compound.[11] ¹H NMR (400 MHz, CDCl₃) δ 8.22 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.86 (d, *J* = 8.6 Hz, 2H), 7.70 (ddd, *J* = 8.7, 7.2, 1.6 Hz, 1H), 7.56 (d, *J* = 8.5 Hz, 1H), 7.50 (d, *J* = 8.6 Hz, 2H), 7.44–7.41 (m, 1H), 6.79 (s, 1H).

4.3.10. 2-(4-(Trifluoromethyl)phenyl)-4H-chromen-4-one (2k)

White solid, 27.9 mg, yield: 48%. This is a known compound.[11] ¹H NMR (400 MHz, CDCl₃) δ 8.23 (dd, *J* = 7.9, 1.5 Hz, 1H), 8.04 (d, *J* = 8.2 Hz, 2H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.73 (ddd, *J* = 8.6, 7.2, 1.6 Hz, 1H), 7.59 (d, *J* = 8.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 6.86 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ –63.04 (s, 3F).

4.3.11. 6-Methyl-2-phenyl-4H-chromen-4-one (2l)

White solid, 42.5 mg, yield: 90%. This is a known compound.[3] ¹H NMR (400 MHz, CDCl₃) δ 8.01 (s, 1H), 7.94–7.87 (m, 2H), 7.52–7.45 (m, 5H), 6.81 (s, 1H), 2.46 (s, 3H).

4.3.12. 5-Methyl-2-phenyl-4H-chromen-4-one (2m)

White solid, 43.9 mg, yield: 93%. This is a known compound.[3] ¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, J = 8.1 Hz, 1H), 7.92–7.89 (m, 2H), 7.52–7.48 (m, 3H), 7.37 (s, 1H), 7.24–7.22 (m, 1H), 6.79 (s, 1H), 2.50 (s, 3H).

4.3.13. 7-Fluoro-2-phenyl-4H-chromen-4-one (2n)

White solid, 32.2 mg, yield: 67%. This is a known compound.[3] ¹H NMR (400 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.8, 6.4 Hz, 1H), 7.91–7.84 (m, 2H), 7.58–7.46 (m, 3H), 7.27–7.21 (m, 1H), 7.14 (td, *J* = 8.5, 2.3 Hz, 1H), 6.79 (s, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ –102.82 to –102.88 (m, 1F).

4.3.14. 6-chloro-2-phenyl-4H-chromen-4-one (20)

White solid, 40.0 mg, yield: 78%. This is a known compound.[3] ¹H NMR (400 MHz, CDCl₃) δ 8.15 (d, J = 2.5 Hz, 1H), 7.87 (dd, J = 8.0, 1.6 Hz, 2H), 7.60 (dd, J = 8.9, 2.6 Hz, 1H), 7.57–7.42 (m, 4H), 6.79 (s, 1H).

4.3.15. 7-bromo-2-phenyl-4H-chromen-4-one (**2p**)

White solid, 48.18 mg, yield: 80%. This is a known compound.[3] ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.5 Hz, 1H), 7.94–7.81 (m, 2H), 7.76 (d, *J* = 1.2 Hz, 1H), 7.56–7.46 (m, 4H), 6.80 (s, 1H).

4.3.16. 2-(Furan-2-yl)-4H-chromen-4-one (**2q**)

White solid, 34.8 mg, yield: 82%. This is a known compound [21]. ¹H NMR (400 MHz, CDCl₃) δ 8.20 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.72–7.63 (m, 1H), 7.61 (s, 1H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 3.4 Hz, 1H), 6.73 (s, 1H), 6.60 (dd, *J* = 3.3, 1.6 Hz, 1H).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2021.132226.

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