Annulation of 2,3-diphenyl-4H-chromen-4-ones via photo-induced hydrogen evolution†

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An efficient photo-induced transition-metal-free direct hydrogen evolution and annulation of 2,3-diphenyl-4H-chromen-4-ones in EtOH–H2O (19 : 1, v/v) at room temperature was described. The reported methodology provided a quick and easy access to the synthesis of dibenzo[a,c]xanthene derivatives, which eliminates the use of any catalysts, oxidants and additives.

Introduction

In recent years, photochemical reactions have drawn great attention due to their environmentally friendly properties, sustainability and high atom efficiency compared to conventional transition-metal catalyzed reactions.1,2 Furthermore, photons have been recognized as an ideal clean reagent for organic reactions.3,4 Photochemical reactions have been successfully applied to the synthesis of phenanthrene derivatives and polycyclic aromatic hydrocarbons (PAHs),5,6 which are difficult to achieve via traditional chemical conditions.

Chromones are the core structures of flavonoids and isoflavonoids and have been extensively studied as biologically active substrates as well as drug molecules.7,8 Owing to their tremendous significance in biological and pharmaceutical activities,9 great effort has been made in the development of various chromone derivatives. Recently, transition-metal catalyzed C–H activation and annulation have been successfully applied for the synthesis of polycyclic derivatives of chromones. Yoshikai et al. reported Pd[db]2 catalyzed cyclization of 3-iodoflavones with 2-(trimethylsilyl)phenyl triflate in the presence of CsF and P(o-tolyl)3 at 110 °C for 24 h to give dibenzo[a,c]xanthene derivative 2a in 79% (Scheme 1a).10

Alternatively, the irradiation of 2,3-diphenyl-4H-chromen-4-one 1a in acetone with a medium pressure mercury lamp under the air atmosphere for 1 h yielded trace amount of 2a (5%, Scheme 1b).11 It was proposed that 2a was generated via the well-known stilbene-phenanthrene type photocyclization of 2,3-diphenyl-4H-chromen-4-one 1a. Generally speaking, photocyclization of stilbene analogues always require the presence of oxidant (O2 or I2, KI, CuCl2, TCNE et al.) for the reaction to proceed smoothly.12-14 Luckily, we were able to obtain the cyclization products of 2,3-di(hetero)aryl-4H-chromen-4-ones without the requirement of any oxidant or additives.15 However, the annulation product for less reactive 2,3-diphenyl-4H-chromen-4-one substrate was not obtained. Following our investigation in the development of photo-induced transition-metal-free cross-coupling reaction16 as well as the direct oxidative annulation,15,17,18 we would like to extend an efficient photo-induced intramolecular hydrogen evolution and annulation to 2,3-diphenyl-4H-chromen-4-ones substrate, which provides access to dibenzo[a,c]xanthene analogues (Scheme 1c).

Results and discussion

Initially, the photo-induced oxidative annulation of 2,3-diphenyl-4H-chromen-4-one (1a) was optimized and the
corresponding data were presented in Table 1. Thus, irradiation of 1a in acetonitrile (100 mL) with a 500 W high-pressure mercury lamp at ambient temperature under argon atmosphere for 3 h gave 2a in the yield of 49% (entry 1). Annulation of 1a in acetone or dichloromethane did not improve the yield of 2a (38–43%, entries 2–3). While, similar yield of 2a was obtained in ethanol (56%, entry 4). It was interesting to find out that the yields of 2a were significantly affected with the presence of H2O (entries 5–8). The amount of additional water played a critical role towards the cyclization, since 2a was obtained in lower yields with either too much or too little water. The optimal yield was obtained when EtOH/H2O (19:1, v/v) was chosen as solvent (63%, entry 7). Meanwhile, the concentration of 1a was also explored (entries 7, 9–10) and 2a was obtained in highest yield when the reaction was performed in 5 mM scale (63%, entry 11). When the cyclization was performed under open air, 2a was obtained in 32% yield (entry 13). Thus, the irradiation of 5 mM 1a in EtOH–H2O (19:1, v/v) at ambient temperature for 3 h under the argon atmosphere was determined to be the optimal condition.

With the optimized conditions in hand, the tolerance of various functional groups (methyl, tert-butyl, fluoro, chloro, hydroxyl and methoxyl) has been explored and the yields were summarized in Table 2. Generally speaking, the substrates bearing electron-donating groups (e.g., Me, OMe, t-Bu) gave the corresponding products 2a–2p in higher good yields (59–81%) comparing to those (2q–2x, 54–69%) bearing electron-withdrawing groups (e.g., F, Cl). Notably, the substrates bearing free hydroxyl group also yielded the corresponding annulation products 2y and 2z in high yields, which are better than the yields of 2l and 2e, respectively. There is a significant difference in the irradiation time as well. Substrates containing EWG usually require longer reaction time than those with EDG. The structures of 2 were characterized by 1H NMR, 13C NMR, HRMS and IR.

Based on our experimental data and a literature report, a plausible mechanism for the formation of 2a is depicted in Scheme 2. Initially, the irradiation of 2,3-diphenyl-4H-chromen-4-one (1a) with a high-pressure mercury lamp generate intermediate A, followed by a thermal suprafacial [1,5]-H shift24 to give intermediate B. Spontaneous rearomatization of benzene ring is believed to be the force driving for [1,5]-sigmatropic shift. Subsequently, keto–enol isomerization of benzene ring was obtained along with the elimination of hydrogen molecule via syn-elimination from intermediate C, which could be easily explained by the restoration of aromaticity for the benzene ring and the entire conjugated system. To our delight, we have successfully detected the hydrogen via GC and the detail experiment was shown in ESI-Fig. 2.† The annihilation product 2a was also produced under the open air (oxygen, Table 1, entry 13), and it indicated that the photochemical cyclization proceed through the S1 state.

Encouraged by the further certified the proposed mechanism, we turned our attention to the reductive carbonyl. 1a was reduced to 3 under the presence of LiAlH4, AlCl3 in THF at 0 °C for 30 min to give 3 (yield 44%, Scheme 3).25 As expected, no stable product of 3 was obtained in the presence of t-BuAlCl2.

Table 1 Optimization for the annulation of 1a

| Entry | Concн (mM) | Solvent (v/v) | Time (h) | Conv (%) | Yield (%) |
|-------|-----------|---------------|----------|----------|-----------|
| 1     | 5         | MeCN          | 3        | 73       | 49        |
| 2     | 5         | Me2CO         | 3        | 69       | 38        |
| 3     | 5         | CH2Cl2        | 3        | 64       | 43        |
| 4     | 5         | EtOH          | 3        | 75       | 56        |
| 5     | 5         | EtOH–H2O (7:1) | 3     | 83       | 38        |
| 6     | 5         | EtOH–H2O (13:1) | 3      | 72       | 45        |
| 7     | 5         | EtOH–H2O (19:1) | 3    | 94       | 63        |
| 8     | 5         | EtOH–H2O (25:1) | 3    | 71       | 49        |
| 9     | 3         | EtOH–H2O (19:1) | 3      | 96       | 37        |
| 10    | 7         | EtOH–H2O (19:1) | 3    | 70       | 41        |
| 11    | 5         | EtOH–H2O (19:1) | 5      | 100      | 51        |
| 12    | 5         | EtOH–H2O (19:1) | 1      | 54       | 36        |
| 13    | 5         | EtOH–H2O (19:1) | 3      | 79       | 32†       |

* 0.5 mmol 1a in various solvent (100 mL, 5 mM) was irradiated with a 500 W high-pressure mercury lamp at room temperature in the argon atmosphere. † Isolated yield. ‡ In the open air atmosphere.
annulation product 4 was detected at the optimal reaction conditions, which could be explained to the lack of keto–enol tautomerization.

**Conclusions**

In summary, we have demonstrated a simple photo-induced direct hydrogen evolution and annulation for the synthesis of...
14H-dibenzo[a,c]xanthen-14-one derivatives in 95% EtOH under room temperature. Comparing to conventional transition-metal-catalyzed annulation and oxidative photo-cyclization reaction, the reported methodology offers several notable advantages, including simple operation, mild reaction condition, no catalysts, oxidants and additives. Meanwhile, the failure of the cyclization of 3 and detection the H2 generated by the annulation of 1 in sufficiently proved the rationality of the proposed mechanism.

**Experimental sections**

Unless otherwise noted, commercial reagents were purchased from Energy Chemical. All experiments were determined by thin layer chromatography (TLC). TLC used silica gel 60 GF254 plate. Column chromatography (200–300 mesh) was performed on silica gel. 1H and 13C NMR spectra were recorded on Bruker 44336 2-phenyl-4 chromen-4-one, which using 2-phenyl-4 chromen-4-one (1 mmol), I2 (304 mg, 1.2 mmol) and MeCN (10 mL) as solvent a series of 3-iodo

General procedure for the synthesis of compounds 2a–2z

Substrate 2,3-diphenyl-4H-chromen-4-one 1a (149 mg, 0.5 mmol) was dissolved in EtOH–H2O (100 mL, 19:1, v/v) at ambient temperature in a quartz tube, and was degassed for 5 min via ultrasound, deaerated for 20 min by bubbling argon and irradiated with a high pressure mercury lamp (500 W) at room temperature for 3 h. Then the reaction mixture were removed under reduced pressure and purified by column chromatography (ethyl acetate/petroleum ether, 1:50) to give 2a (63%).

14H-dibenzo[a,c]xanthen-14-one (2a).10 1H NMR (400 MHz, CDCl3) δ 10.19 (d, J = 8.4 Hz, 1H), 8.77 (d, J = 8.2 Hz, 1H), 8.68 (t, J = 8.5 Hz, 2H), 8.46 (d, J = 7.9 Hz, 1H), 7.87–7.68 (m, 6H), 7.51–7.47 (m, 1H); 13C NMR (100 MHz, CDCl3) δ 178.33, 155.21, 154.42, 133.92, 133.80, 130.62, 128.99, 128.57, 127.75, 127.50, 127.32, 126.71, 126.68, 124.71, 124.10, 123.99, 123.97, 122.90, 122.32, 117.50, 112.70.

6-methoxy-12-methyl-14H-dibenzo[a,c]xanthen-14-one (2b). White solid. Yield: 72%, Mp: 229.3–231.9 °C. 1H NMR (400 MHz, CDCl3) δ 10.11 (d, J = 8.4 Hz, 1H), 8.42 (d, J = 8.4, 5.7 Hz, 2H), 8.14 (s, 1H), 7.82 (d, J = 2.3 Hz, 1H), 7.69 (dd, J = 11.3, 4.1 Hz, 1H), 7.63–7.53 (m, 1H), 7.48 (dd, J = 8.5, 2.0 Hz, 1H), 7.41 (d, J = 8.4 Hz, 1H), 7.17 (dd, J = 9.0, 2.4 Hz, 1H), 3.97 (s, 3H), 2.49 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 178.10, 161.54, 155.33, 152.54, 135.81, 134.79, 134.76, 129.75, 128.64, 128.55, 128.26, 125.96, 123.59, 122.27, 117.97, 117.18, 116.91, 110.89, 104.35, 55.57, 21.18; IR (KBr), ν (cm−1) 2927, 1610, 1446, 1247, 1029, 95, 821, 658, 530; HRMS (ESI): calc. for C23H16O3 [M + H]+ 341.1178, found 341.1177.

6-Methoxy-12,16-dimethyl-14H-dibenzo[a,c]xanthen-14-one (2c). White solid. Yield: 76%, Mp: 222.4–224.2 °C. 1H NMR (400 MHz, CDCl3) δ 9.93 (s, 1H), 8.45 (d, J = 8.4 Hz, 1H), 8.31 (d, J = 8.4 Hz, 1H), 8.15 (s, 1H), 7.81 (s, 1H), 7.50–7.38 (m, 3H), 7.17 (d, J = 9.0 Hz, 1H), 3.99 (s, 3H), 2.60 (s, 3H), 2.50 (s, 3H); 13C NMR (100 MHz, CDCl3) δ 178.20, 161.59, 155.54, 152.61, 138.68, 135.97, 134.74, 134.32, 129.82, 127.75, 127.63, 125.98, 124.71, 123.67, 122.20, 117.65, 117.20, 116.57, 110.83, 104.14, 55.57, 22.21, 21.21; IR (KBr), ν (cm−1) 3101, 2914, 1612, 1442, 1238, 1130, 1029, 827, 622, 495; HRMS (ESI): calc. for C22H16O3 [M + H]+ 341.1334, found 341.1336.

3-(tert-Butyl)-6-methoxy-12-methyl-14H-dibenzo[a,c]xanthen-14-one (2d). Yellow solid. Yield: 81%, Mp: 255.6–258.1 °C. 1H NMR (400 MHz, CDCl3) δ 10.08 (d, J = 8.9 Hz, 1H), 8.60 (d, J = 8.9 Hz, 1H), 8.51 (d, J = 2.3 Hz, 1H), 8.20 (s, 1H), 8.01 (d, J = 2.3 Hz, 1H), 7.83 (dd, J = 8.9, 2.0 Hz, 1H), 7.51 (d, J = 2.8 Hz, 2H), 7.28 (dd, J = 9.1, 2.4 Hz, 1H), 4.05 (s, 3H), 2.50 (s, 3H), 1.51 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 178.21, 161.58, 155.19, 152.77, 149.03, 136.20, 134.80, 134.37, 127.69, 127.53, 126.93, 126.74,
126.22, 126.09, 123.73, 118.36, 118.16, 117.27, 116.16, 111.13, 105.19, 77.48, 77.16, 76.84, 75.58, 75.35, 31.60, 21.18; IR (KBr), ν (cm⁻¹) 2941, 1623, 1396, 1259, 1168, 1047, 854, 800, 576; HRMS (ESI): calc. for C₂₇H₂₄O₃ [M + H⁺] 381.1855, found 381.1855.

3-(tert-Butyl)-6,12-dimethyl-14H-dibenzo[a,c]xanthen-14-one (2j). Yellow solid. Yield: 78%; Mₚ: 260.2-262.8 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.02 (d, J = 8.9 Hz, 1H), 8.53 (d, J = 1.6 Hz, 1H), 8.35 (d, J = 8.3 Hz, 1H), 8.29 (s, 1H), 8.11 (s, 1H), 7.80 (dd, J = 8.9, 1.9 Hz, 1H), 7.43 (dd, J = 8.5, 1.9 Hz, 1H), 7.36 (dd, J = 8.0, 4.2 Hz, 2H), 2.57 (s, 3H), 2.46 (s, 3H), 1.54 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 178.14, 154.87, 152.56, 148.92, 140.63, 134.69, 134.19, 134.04, 128.67, 127.51, 127.02, 129.93, 126.45, 125.88, 123.96, 123.55, 122.43, 121.86, 110.84, 111.22, 111.84, 35.15, 31.64, 22.42, 21.11; IR (KBr), ν (cm⁻¹) 3050, 2932, 2649, 1431, 1402, 1284, 1193, 1039, 850, 808, 556; HRMS (ESI): calc. for C₂₇H₂₄O₃ [M + H⁺] 381.1855, found 381.1855.

6-Methyl-14H-dibenzo[a,c]xanthen-14-one (2k). Yellow solid. Yield: 70%; Mₚ: 185.7-187.6 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.10 (d, J = 8.3 Hz, 1H), 8.38 (d, J = 8.1 Hz, 1H), 8.34 (dd, J = 7.8, 1.3 Hz, 1H), 8.23 (d, J = 8.2 Hz, 1H), 8.13 (s, 1H), 7.66-7.61 (m, 2H), 7.52 (dd, J = 11.0, 3.9 Hz, 1H), 7.45 (d, J = 8.2 Hz, 1H), 7.38 (t, J = 7.4 Hz, 1H), 7.26 (d, J = 8.2 Hz, 1H), 2.47 (t, 3H), 1.17, 1.06, 1.02; IR (KBr), ν (cm⁻¹) 3045, 2916, 1635, 1488, 1398, 1284, 1124, 1033, 923, 806, 763, 636, 522; HRMS (ESI): calc. for C₂₃H₂₂O₂ [M + Na⁺] 383.3091, found 333.0893.

3-(tert-Butyl)-6-methyl-14H-dibenzo[a,c]xanthen-14-one (2l). Yellow solid. Yield: 75%; Mₚ: 212.2-214.4 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.06 (d, J = 8.8 Hz, 1H), 8.60 (d, J = 1.8 Hz, 1H), 8.53 (d, J = 8.3 Hz, 1H), 8.45-8.38 (m, 2H), 7.82 (dd, J = 8.8, 2.0 Hz, 1H), 7.74-7.69 (m, 1H), 7.60 (d, J = 8.2 Hz, 1H), 7.48 (d, J = 8.2 Hz, 1H), 7.46-7.41 (m, 1H), 2.65 (s, 3H), 1.53 (s, 9H); ¹³C NMR (150 MHz, CDCl₃) δ 178.27, 155.14, 154.51, 149.22, 140.94, 134.31, 133.62, 128.88, 127.57, 127.10, 126.97, 126.73, 126.64, 125.47, 125.11, 124.07, 122.66, 121.93, 118.17, 117.58, 112.12, 35.20, 31.64, 22.51; IR (KBr), ν (cm⁻¹) 3099, 2956, 1635, 1463, 1411, 1296, 1122, 1012, 873, 763, 644, 559, 470; HRMS (ESI): calc. for C₂₃H₂₄O₂ [M + Na⁺] 383.3091, found 383.1516.

2,6-Dimethyl-14H-dibenzo[a,c]xanthen-14-one (2m). Yellow solid. Yield: 75%; Mₚ: 218.5-221.1 °C. ¹H NMR (600 MHz, CDCl₃) δ 0.97 (s, 1H), 8.53 (d, J = 7.8 Hz, 1H), 8.23 (t, J = 8.1 Hz, 1H), 8.08 (s, 1H), 7.65 (dd, J = 11.1, 4.0 Hz, 1H), 7.45 (d, J = 8.1 Hz, 1H), 7.38 (t, J = 7.4 Hz, 1H), 7.31 (d, J = 8.1 Hz, 1H), 7.24 (d, J = 9.2 Hz, 1H), 2.54 (s, 3H), 2.46 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 178.11, 155.29, 154.21, 140.76, 138.21, 133.86, 133.44, 129.12, 128.30, 127.81, 127.39, 126.52, 124.87, 124.44, 124.93, 123.75, 123.22, 122.04, 121.05, 117.44, 111.66, 22.34, 22.16; IR (KBr), ν (cm⁻¹) 3028, 2914, 1637, 1415, 1307, 1172, 1033, 873, 761, 667, 576; HRMS (ESI): calc. for C₂₃H₂₄O₂ [M + Na⁺] 374.1048, found 374.1048.

11-Methoxy-6-methyl-14H-dibenzo[a,c]xanthen-14-one (2n). White solid. Yield: 59%; Mₚ: 190.2-191.8 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.15 (d, J = 8.2 Hz, 1H), 8.54 (d, J = 8.2 Hz, 1H), 8.40 (d, J = 8.8 Hz, 1H), 8.32 (s, 1H), 8.27 (d, J = 8.8 Hz, 1H), 7.70 (t, J = 7.4 Hz, 1H), 7.62-7.60 (m, 1H), 7.41 (d, J = 8.1 Hz, 1H), 6.96 (d, J = 7.4 Hz, 1H), 6.90 (s, 1H), 3.92 (s, 3H), 2.58 (s, 3H); ¹³C NMR
(150 MHz, CDCl₃) δ 177.70, 164.23, 156.09, 155.24, 140.79, 133.78, 129.43, 128.92, 128.33, 128.09, 127.89, 127.31, 126.38, 123.80, 122.72, 122.27, 121.72, 117.99, 114.04, 111.91, 99.62, 55.95, 22.42; IR (KBr), υ (cm⁻¹) 3016, 2918, 1622, 1444, 1249, 1031, 923, 783, 736, 630, 540; HRMS (ESI): calc. for C₂₃H₂₁F₂O₂ [M + Na]⁺ 397.1178, found 397.1175.

11-Methoxy-2,6-dimethyl-14H-dibenzo[a,c]xanthen-14-one (2q). Yellow solid. Yield: 70%, M_p: 221.4-223.8 °C. ¹H NMR (600 MHz, CDCl₃) δ 8.48 (dd, J = 8.3 Hz, 1H), 8.33 (s, 1H), 8.29 (dd, J = 8.8 Hz, 1H), 7.44 (dd, J = 11.3, J = 8.8 Hz, 2H), 7.01-0.65 (m, 2H), 3.95 (s, 3H), 2.61 (d, J = 8.9 Hz, 6H). IR (KBr), υ (cm⁻¹) 3105, 2964, 1917, 1610, 1458, 1324, 1026, 813, 636, 536; HRMS (ESI): calc. for C₂₃H₂₁F₂O₂ [M + H]⁺ 359.1083, found 359.1089.

3-Fluoro-11-methoxy-6-methyl-14H-dibenzo[a,c]xanthen-14-one (2q). White solid. Yield: 64%, M_p: 254.4-257.2 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (s, 1H), 7.88 (dd, J = 8.3 Hz, 1H), 7.39 (m, 3H), 2.60 (s, 6H); 13C NMR (100 MHz, CDCl₃) δ 157.96, 156.10, 154.47, 148.17, 130.39, 129.50, 128.52, 128.09, 127.96, 126.73, 125.14, 123.85, 122.58, 122.21, 121.37, 118.05, 114.06, 111.83, 99.67, 55.98, 22.45, 22.23; IR (KBr), υ (cm⁻¹) 2912, 1627, 1444, 1257, 1172, 1022, 825, 690, 576, 466; HRMS (ESI): calc. for C₂₃H₂₁F₂O₂ [M + Na]⁺ 377.1154, found 377.1157.
(d, $\delta = 9.37$ Hz), 124.64 (d, $\delta = 3.72$ Hz), 122.47, 120.21, 117.98, 115.81 (d, $\delta = 23.56$ Hz), 114.23, 111.9537, 111.9448, 108.21 (d, $\delta = 22.61$ Hz), 99.72, 56.02, 22.25; IR (KBr), $\nu\ (\text{cm}^{-1})$ 2974, 1897, 1623, 1417, 1271, 1166, 1051, 821, 607, 430; HRMS (ESI): calc. for C$_{21}$H$_{17}$F$_{2}$O$_{3}$ [M + Na]$^+$ 391.0835, found 391.0835.

3-(tert-Butyl)-6-fluoro-11-methoxy-14H-dibenzo[a,c]xanthene-14-one (2x). White solid. Yield: 69%, $M_p$: 254.2–256.7 °C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 10.01 (d, $\delta = 8.9$ Hz, 1H), 8.47 (dd, $\delta = 8.9$, 6.0 Hz, 1H), 8.38 (s, 1H), 8.22 (d, $\delta = 8.9$ Hz, 1H), 8.18–8.11 (m, 1H), 7.85–7.77 (m, 1H), 7.37 (d, $\delta = 6.6$ Hz, 1H), 6.92 (dd, $\delta = 8.9$, 2.0 Hz, 1H), 6.83 (s, 1H), 3.90 (s, 3H), 1.51 (s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 177.43, 164.26, 164.03 (d, $\delta = 3.55$ Hz), 120.56, 118.26, 117.79, 115.89 (d, $\delta = 23.63$ Hz), 114.11, 111.95, 108.18 (d, $\delta = 22.75$ Hz), 99.56, 55.94, 35.18, 31.56; IR (KBr), $\nu\ (\text{cm}^{-1})$ 3341, 2912, 2345, 1608, 1436, 1244, 806, 721, 578; HRMS (ESI): calc. for C$_{20}$H$_{15}$NO$_{3}$ [M + Na]$^+$ 390.0841, found 390.0841.

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