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Ultraviolet-light-induced aerobic oxidation of benzylic C(sp^3)-H of alkylarenes under catalyst- and additive-free conditions

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ABSTRACT

A mild and efficient system has been discovered for the synthesis of α-aryl carbonyl compounds via oxidation of benzylic C—H to C═O bonds. This ultraviolet-light-mediated oxygenation reaction exhibited excellent substrate scope including various xanthenes, thioxanthenes and 9, 10-dihydroacridines and afforded the corresponding ketones with good to excellent yields under catalyst- and additive-free conditions at room temperature.

1. Introduction

The development of green and sustainable methods for the oxidation of benzylic C—H to C═O bond for the synthesis of α-aryl carbonyl compounds under milder conditions continues to attract broad interest in modern synthetic chemistry [1]. Not only such strategies are consistent with the criterion of environmental and economic demands, but also these C—H bonds are very abundant in numerous organic compounds.

Consequently, considerable efforts have been devoted to develop more efficient synthetic methodologies or to improve the existing synthetic process to access this transformation by using various traditional toxic or corrosive metal-, peracid-, or oxoacid-based oxidants [2], which would cause serious environmental problems. Compared with the above oxidants, there is no doubt that molecular oxygen, on behalf of green and sustainable chemistry, as oxidant for this transformation is more ideal because it is abundant, inexpensive, clean, readily available, nontoxic, and more importantly, it is safe because the by-product of oxidation is water [3]. However, the molecular oxygen, which generally exists in the stable triplet ground state, has low activity towards benzylic C(sp^3)-H bonds [4]. As a result, numerous catalyst systems based on transition metals or metal-free systems, which can activate the C—H bonds or the molecular oxygen, have been devised to enable aerobic oxidation of benzylic C(sp^3)-H bonds, for example, palladium, copper, manganese, cesium, iridium, iron, and gold complexes [3c,4a-b,5-8], DDQ/TBN, NHPI/TBN, NH_4I/AcOH, and TEMPO derivatives [9,10].

In addition, in recent years, another kind of attractive catalyst system based on light-induced photocatalysis, involving photosensitive molecules, transition metal complexes, and organic dyes, has been designed and applied in the reaction [11]. For example, various copper [11c,12] and iron-based [13] photocatalytic reactions of the aerobic oxidation of C—H bonds has been established. Despite these advances, most of the above-mentioned processes suffer from some shortcomings such as use of metal catalyst, additives, hazardous reagents, tedious work-up processes, and the requirement of special apparatus, which adversely affect the economics as well as the ecofriendly nature of the reaction.

At the same time, xanthones, thioxanthones, and acridones have been widely utilized in many areas [14]. Xanthones, as a

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heterotricyclic compounds, exhibit a variety of biological activities and have been used to treat some diseases such as hypertension, convulsions, thrombosis, tumor and Alzheimer [15]. Thioxanthones are widely used in coating, microelectronics, photoresist and photoinitiator [16], and acridones play an important role in medicines for their significant antitumor activity [17]. In addition, they are also used as the raw fluorescent materials [18]. In view of their interesting properties, it remains highly desirable to develop ecologically benign strategies for synthesis of these ketone compounds, requiring milder conditions as well as avoiding the need for toxic metal catalysts or additives.

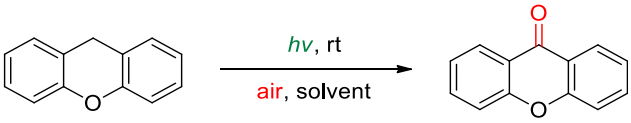
Herein, we report a new strategy for photoinduced aerobic oxidation of the benzylic C (sp^3)-H for synthesis of xanthones, thioxanthones and acridones from xanthenes, thioxanthenes and 9,10-dihydroacridines, respectively. The reactions gave corresponding ketones with good yields under mild conditions. To the best of our knowledge, the ultraviolet-induced methodology of synthesis of these compounds under catalyst- and additive-free conditions at room temperature has not been reported so far. It is noteworthy to mention that, in 2017, one benzophenone compound was involved when Wang and co-workers reported a photoinduced oxidative formylation of *N,N*-dimethylanilines [19]. Then, Jiang also disclosed visible light-induced selective aerobic oxidative transposition of vinyl halides using a tetrahalogenoferrate(III) complex catalyst [13a]. In addition, recently, we also noted that Wang and co-worker developed a strategy for selective oxidation of benzylic C—H to C═O bonds at 140 °C under 0.5 MPa of oxygen [1b]. By comparison, the use of ultraviolet light under catalyst- and additive-free at room temperature makes this reaction quite simple, mild, sustainable and practical.

2. Results and discussion

Firstly, the reaction conditions were optimized by using 9*H*-xanthene (**1a**) as a model substrate (Table 1). When the model reaction was carried out in the molecular oxygen with the illumination of LEDs (365-370 nm) in ethyl acetate at room temperature for 12 h, the target product 9*H*-xanthen-9-one (**2a**) was detected in 9% yield by internal standard method of gas chromatography (entry 1).

illumination of LEDs (365—370 nm) in ethyl acetate at room temperature for 12 h, the target product 9*H*-xanthen-9-one (**2a**) was detected in 9% yield by internal standard method of gas chromatography (entry 1). The optimization process revealed that product **2a** was detected in 23%, 11%, 7% and 6% under the irradiation of LEDs at 380—385 nm, 400—405 nm, 420—425 nm and 450—455 nm, respectively (entries 2—5). The substrate exhibited higher reactivity under irradiation of LED (380—385 nm). It was found that the ultraviolet—visible absorption intensity of the starting material **1a** was stronger at 380—385 nm than that at visible ranges. The effects of various solvents on this reaction were evaluated. The results indicated that using DCE or DMSO as a solvent is obviously better than other solvents for the reactions after 12 h. When DCE and DMSO were used as the solvents, the reaction gave desired product with 60% and 54% yields, respectively (entries 6 and 9). However, when the reaction time was prolonged to 24 h, DMSO as the solvent gave slightly higher yield (entries 17 and 18). The poor yields (4—35%) of product **2a** were obtained, when the reactions were performed in other solvents including toluene, DMF, dioxane, H_2O, DME, MeCN and THF (entries 7 and 8, 10—14). However, it was not clear why the reaction has a strong dependence on the solvent. Furthermore, the result showed that none of the desired product was observed in the absence of the molecular oxygen (entry 15). In addition, when the model reaction was performed in oil bath at 80 °C, 24% yield of **2a** was isolated in dark circumstance. This data showed that the reaction under ultraviolet irradiation was faster than that on elevated temperature (entry 16).

Table 1
Optimization of reaction conditions for product 2a.[a]



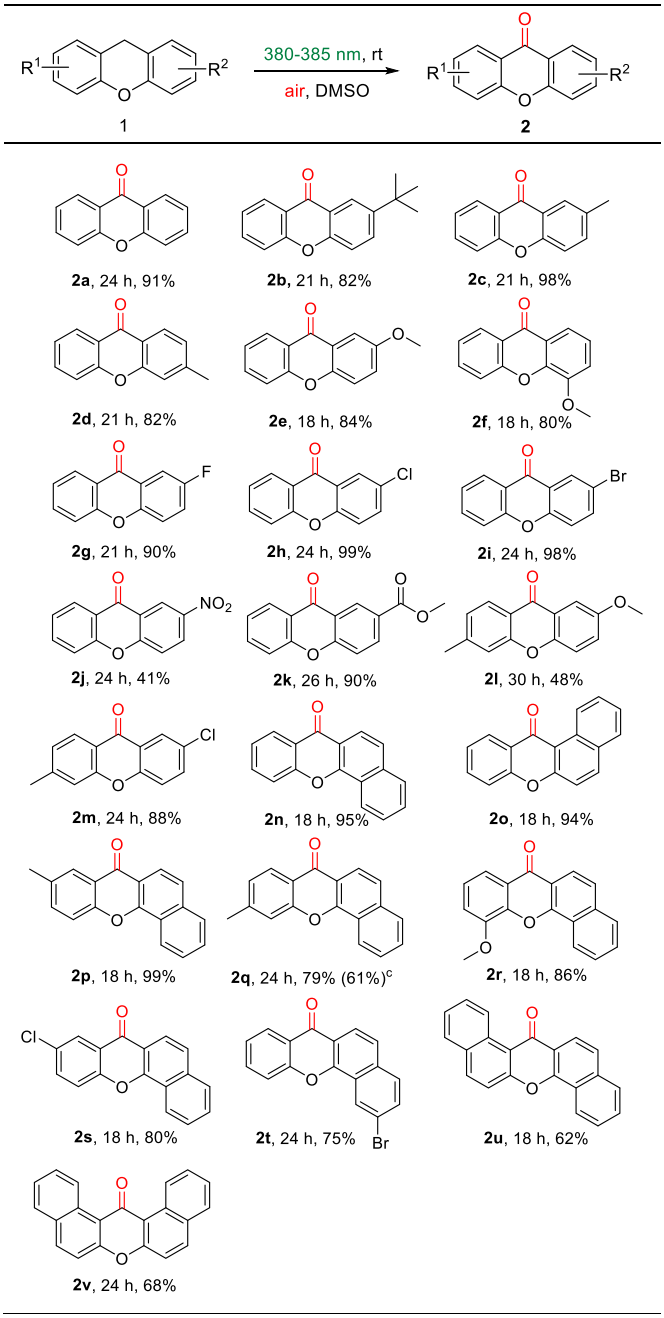
1a **2a**

Entry	Light source	Solvent	T (h)	Yield(%)[b]
1	365—370 nm	EtOAc	12	9
2	380—385 nm	EtOAc	12	23
3	400—405 nm	EtOAc	12	11
4	420—425 nm	EtOAc	12	7
5	450—455 nm	EtOAc	12	6
6	380—385 nm	DCE	12	60
7	380—385 nm	toluene	12	20
8	380—385 nm	DMF	12	23
9	380—385 nm	DMSO	12	54
10	380—385 nm	dioxane	12	29
11	380—385 nm	H_2O	12	4
12	380—385 nm	DME	12	28
13	380—385 nm	MeCN	12	35
14	380—385 nm	THF	12	27
15[c]	380—385 nm	DMSO	12	N.R.
16[d]	In dark	DMSO	12	24
17	380—385 nm	DMSO	24	94
18	380—385 nm	DCE	24	88

[a] Reaction conditions: xanthene (**1a**, 0.2 mmol), solvent (2 mL), at room temperature under light irradiation in air.
[b] Internal standard yield of gas chromatography, N.R. = no reaction.
[c] Under N_2 atmosphere.
[d] 80 °C (oil bath).

After mastering the optimized conditions, to explore the generality of the ultraviolet-induced strategy, some xanthene derivatives were examined (Table 2). A variety of xanthenes were employed under the optimal reaction conditions. Xanthenes with an electron-donating group (2-tert-butyl, 2-methyl, 3-methyl, 2-methoxy, or 4-methoxy) reacted with O_2 and gave the desired products **2b-2f** in 80—98% yields. Xanthenes bearing electron-withdrawing groups, such as 2-F, 2-Cl, 2-Br, or 2-MeO_2C, underwent smooth transformations to give the products **2g-2i** and **2k** in isolated yields of 90—99%. In addition, disubstituted xanthenes could proceed smoothly to provide the corresponding products **2l** and **2m** in 48% and 88% yields, respectively. Moreover, two of benzo-fused xanthenes **1n** and **1o** still displayed high reactivity and led to the desired products **2n** and **2o** in 95% and 94% yields. Furthermore, the benzo-fused xanthenes with an electron-donating group or electron-withdrawing group (9-methyl, 10-methyl, 11-methoxy, 9-Cl, 2-Br) were also oxidized to afford the target products **2p-2t** in 75%—99% yields. Moreover, to improve the yield of **2q**, the reaction temperature was elevated to 40 °C, however, the yield was reduced to 61%. Compared to room temperature, the amount of by-products was significantly increased which influenced the formation of the desired product. The 14*H*-dibenzo[*a,h*]xanthene **1u** and 14*H*-dibenzo[*a,j*]xanthene **1v** were well tolerated and gave the expected products **2u** and **2v** in 62% and 68% yields, respectively. At the same time, we have also observed that when the R^2 substituent was NO_2 group, the desired product was obtained with only 41% yield, probably because of the strong electron-withdrawing inductive effect of nitro group, which greatly influences the formation of the desired product. Subsequently, 9*H*-xanthen-2-amine was employed for the oxidation reaction.

Table 2
Synthesis of xanthones.[a,b]



[a] Reaction conditions: xanthenes (**1**, 0.2 mmol), DMSO (2 mL), LEDs (380–385 nm), at room temperature in air.
[b] Isolated yield.[c] at 40 °C.

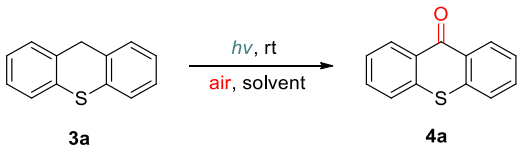
However, no product was obtained, instead of only the material was observed, even after longer reaction times (40 h).

Then, we were interested in whether the system would work for synthesis of other heterotricyclic compounds, such as thioxanthenes and 9,10-dihydroacridines while modifying the conditions slightly. At first, only 18% yield of target product was obtained when 9*H*-thioxanthene was used under the above optimized conditions. To improve the yield, a study on the effect of the solvent was undertaken. As depicted in Table 3, DCE was the optimal solvent under the irradiation of LEDs (380–385 nm) and gave the yield up to 83% yield (entry 3).

In addition, an obvious loss of the yield was observed when the reaction time was prolonged to 12 h (entries 7 and 8). For *N*-methyl-9,10-dihydroacridine compound, the reaction media DCE afforded slightly higher yield (56%) than DMSO. In addition, we also noted that the substrates thioxanthene and acridine exhibit better reaction activity than xanthene.

Under the above optimized conditions LEDs (380–385 nm) as the light source, DCE as the solvent, and at room temperature in air), the substrate scope of this reaction was examined by using various thioxanthenes and acridines. The results are summarized in Table 4. Thioxanthenes, both bearing electron-donating groups and

Table 3
Optimization of reaction conditions for product 4a.[a]



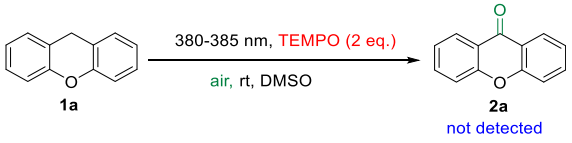
Entry	Light source	Solvent	T (h)	Yield (%)[b]
1	380–385 nm	DMSO	3	18
2	380–385 nm	DCM	3	30
3	380–385 nm	DCE	3	83
4	380–385 nm	DMF	3	N.D.
5	380–385 nm	DME	3	N.D.
6	380–385 nm	EtOAc	3	N.D.
7	380–385 nm	DCE	6	72
8	380–385 nm	DCE	12	60

[a] Reaction conditions: thioxanthene (**3a**, 0.2 mmol), solvent (2 mL), at room temperature under light irradiation in air.
[b] Internal standard yield of Gas Chromatography, N.D. = not detected.

electron-withdrawing groups, were well tolerated and gave corresponding thioxanthones **4b-4e** in 70–83% yields. *N*-methyl-9,10-dihydroacridines with a functional group, including 4-methoxy or 2-Cl, could proceed smoothly to provide the desired acridones **6b** and **6c** in 54% and 72% yields, respectively. In addition, *N*-benzyl-9,10-dihydroacridine could be also converted into the corresponding products in 88% yield in the reaction conditions.

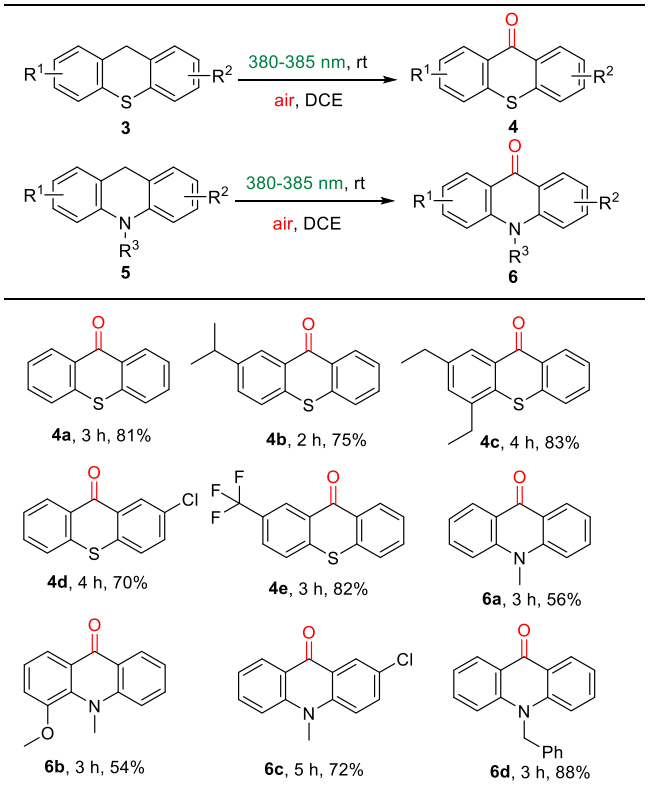
To explore the reaction mechanism, the control experiment and the isotopic tracer experiment were carried out (Schemes 1 and 2). No desired product was found when stoichiometric radical-trapping reagent 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) were added to the photoinduced reaction of **1a**. It was suggested that this transformation might proceed via a radical pathway. On the other hand, in order to find the source of oxygen in product, stoichiometric water labelled with ^{18}O were added to the isotopic tracer experiment and analyzed by MS. The result indicated that 90% oxygen of xanthone obtained from O_2 in air.

On the basis of these experiments and reported literature [20], the possible reaction mechanism is proposed (Scheme 3). Ground-state triplet oxygen $^3O_2$ was irradiated by ultraviolet light (380–385 nm), which formed to singlet oxygen $^1O_2$ with a high reactivity, then reacted with xanthene **1a** and generated a free-radical cation **I** and O_2·- through a single electron transfer (SET) process. Then the proton transfer between **I** and $O_2\cdot^-$ formed a superoxide radical HOO· and radical **II**, which combined to form intermediates **III**. Finally, the intramolecular dehydration of **III** produced the product **2a**.

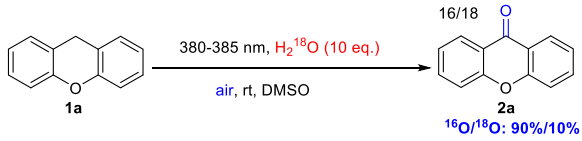


Scheme 1. Control experiment for mechanism exploration.

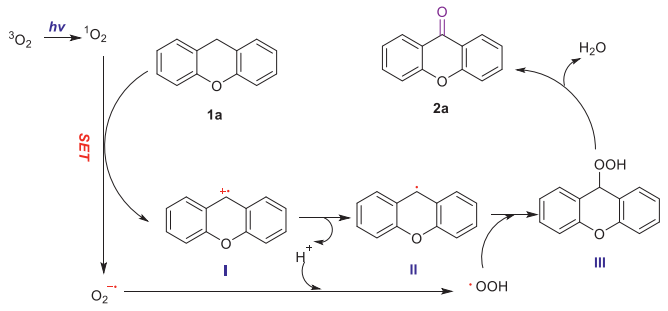
Table 4
Synthesis of thioxanthenes and 9,10-dihydroacridines.[a,b]



[a] Reaction conditions: thioxanthenes or acridines (**3** or **5**, 0.2 mmol), DCE (2 mL), LEDs (380–385 nm), at room temperature in air.
[b] Isolated yield.



Scheme 2. The isotopic tracer experiment.



Scheme 3. The possible reaction mechanism.

3. Conclusion

In summary, a new method for ultraviolet light induced oxidation of benzylic C(sp^3)-H was developed. A series of alkylarenes, including xanthenes, thioxanthenes, and 9,10-dihydroacridines were converted to the corresponding carbonyl compounds in good yields. The procedure provides a simple and mild route to form ketones under catalyst-, and additive-free at room temperature. In addition, the probable reaction mechanism clearly explained the reaction process and the role of oxygen.

4. Experimental section

Unless otherwise stated, all the reactions were performed at room temperature. All reactions were detected by GC. GC analyses were conducted on an Agilent GC7890A system with a flame ionization detector (FID) and a SE-54 capillary column. GC-MS was performed on Thermo Trace ISQ instrument with TG 5MS capillary column. Purification of reaction products was carried out by column chromatography on silica gel. ^{1}H NMR (500 MHz) and ^{13}C NMR (126 MHz) spectra were obtained on a Bruker Avance III spectrometer. $CDCl_3$ was used as the solvent with tetramethylsilane (TMS) as the internal standard. Photoinduced reactions were performed with 6W LEDs. Xanthenes (**1b-1v**) [21a], thioxanthenes (**3a-3e**) [21b] and 9,10-dihydriacridines (**5b-5d**) [21c] were prepared in our laboratory according to reported procedures. Other reagents and solvents were purchased from commercial supplier and used without any further treatment.

4.1. Typical procedure for the preparation of 9H-Xanthen-9-one (*2a*)

A 15-mL Schlenk tube equipped with a magnetic stirrer bar was charged with xanthene (**1a**; 36.4 mg, 0.2 mmol) and DMSO (2 mL). The Schlenk tube was placed in a dark box and illuminated with 6W blue LED (380–385 nm). The mixture was stirred vigorously at room temperature in air until the reaction was complete (GC). The mixture was then concentrated on a rotary evaporator, and the residue was purified by column chromatography (silica gel, PE–EtOAc) to **2a**: white solid. m.p.: 174–176 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33–8.31 (m, 2H), 7.72–7.68 (m, 2H), 7.46 (d, J = 8.5 Hz, 2H), 7.37–7.26 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.3, 156.3, 134.9, 126.8, 124.0, 122.0, 118.1. MS (EI), m/z 196.01 [M^+, 100%].

4.2. 2-(tert-Butyl)-9H-xanthen-9-one (*2b*)

White solid. m.p.: 113–116 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 8.36–8.34 (m, 1H), 8.33 (d, J = 2.5 Hz, 1H), 7.79–7.77 (m, 1H), 7.71–7.68 (m, 1H), 7.47 (d, J = 8.4 Hz, 1H), 7.42 (d, J = 8.8 Hz, 1H), 7.36 (t, J = 7.7 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 177.6, 156.3, 154.5, 147.2, 134.7, 132.9, 126.9, 123.8, 122.6, 122.0, 121.3, 118.0, 117.7, 34.9, 31.5. MS (EI), m/z 237.10 [M^+, 100%].

4.3. 2-Methyl-9H-xanthen-9-one (*2c*)

White solid. m.p.: 119–122 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.34 (d, J = 7.9Hz, 1H), 8.12 (s, 1H), 7.72–7.69 (m, 1H), 7.53 (d, J = 8.5, 1H), 7.48–7.46 (m, 1H), 7.39 (d, J = 8.8Hz, 1H), 7.36 (d, J = 7.5Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 177.4, 156.3, 154.6, 136.2, 134.8, 133.8, 126.9, 126.2, 123.8, 122.0, 121.6, 118.1, 117.9, 21.0. MS (EI), m/z 209.97 [M^+, 100%].

4.4. 3-Methyl-9H-xanthen-9-one (*2d*)

White solid. m.p.: 95–97 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, J = 8.1Hz, 1H), 8.20 (d, J = 8.3Hz, 1H), 7.71–7.67 (m, 1H), 7.45 (d, J = 8.5Hz, 1H), 7.36 (t, J = 7.5Hz, 1H), 7.17 (d, J = 8.1Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.1, 156.4, 156.3, 146.4, 134.7, 126.8, 126.6, 125.6, 123.9, 122.1, 119.8, 118.0, 117.8, 22.1.MS(EI), m/z 210.08 [M^+, 100%].

4.5. 2-Methoxy-9H-xanthen-9-one (*2e*)

White solid.m.p.: 127–129 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (d, J = 8.2 Hz, 1H), 7.71–7.67 (m, 1H), 7.46 (d, J = 8.7 Hz, 1H), 7.41 (d, J = 9.2 Hz, 1H), 7.37–7.34 (m, 1H), 7.32–7.29 (m, 1H), 3.90 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.2, 156.2, 156.1, 151.1, 134.7, 126.8, 125.0, 123.8, 122.2, 121.4, 119.5, 118.1, 106.0, 56.0. MS (EI), m/z 226.07 [M^+, 100%].

4.6. 4-Methoxy-9H-xanthen-9-one (*2f*)

White solid. m.p.: 174–175 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, J = 8.0 Hz, 1H), 7.90 (d, J = 8.1 Hz, 1H), 7.74–7.71 (m, 1H), 7.61 (d, J = 8.4 Hz, 1H), 7.40–7.37 (m, 1H), 7.30 (t, J = 8.0 Hz, 1H), 7.23 (d, J = 8.0 Hz, 1H), 4.03 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.3, 156.1, 148.8, 146.7, 134.9, 126.8, 124.2, 123.6, 122.9, 121.8, 118.4, 117.8, 115.5, 56.6. MS (EI), m/z 225.96 [M^+, 100%].

4.7. 2-Fluoro-9H-xanthen-9-one (*2g*)

White solid. m.p.: 153–155 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (d, J = 7.9 Hz, 1H), 7.97–7.95 (m, 1H), 7.75–7.72 (m, 1H), 7.50–7.47 (m, 2H), 7.46–7.42 (m, 1H), 7.38 (t, J = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.7, 158.8 (d, J = 245.8 Hz), 156.3, 152.5, 135.2, 126.8, 124.3, 123.1 (d, J = 25.3 Hz), 122.8 (d, J = 7.1 Hz), 121.2, 120.1 (d, J = 7.8 Hz), 118.1, 111.6 (d, J = 23.6 Hz). MS (EI), m/z 214.03 [M^+, 100%].

4.8. 2-Chloro-9H-xanthen-9-one (*2h*)

White solid. m.p.: 166–169 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (d, J = 8.0 Hz, 1H), 8.28 (d, J = 2.6 Hz, 1H), 7.75–7.72 (m, 1H), 7.66–7.64 (m, 1H), 7.48 (d, J = 8.5 Hz, 1H), 7.45 (d, J = 8.9 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.3, 156.2, 154.6, 135.3, 135.0, 129.9, 126.9, 126.2, 124.4, 122.8, 121.6, 119.9,

118.2. MS (EI), *m*/*z* 229.99 [M^+, 100%].

4.9. 2-Bromo-9H-xanthen-9-one (**2i**)

White solid. m.p.: 149–152 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.31 (d, *J* = 7.9 Hz, 1H), 7.78 (d, *J* = 8.9 Hz, 1H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 8.3 Hz, 1H), 7.40–7.37 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.1, 156.2, 155.1, 137.8, 135.3, 129.4, 126.9, 124.4, 123.2, 121.7, 120.1, 118.2, 117.2. MS (EI), *m*/*z* 275.97 [M^+, 95%], 273.85 [100%].

4.10. 2-Nitro-9H-xanthen-9-one (**2j**)

Brown solid. m.p.: 201–203 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 9.19 (d, *J* = 2.9 Hz, 1H), 8.55–8.53 (m, 1H), 8.33 (d, *J* = 8.0 Hz, 1H), 7.83–7.79 (m, 1H), 7.64 (d, *J* = 9.2 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.8, 159.3, 156.0, 144.0, 136.0, 129.1, 127.1, 125.4, 123.7, 121.8, 121.5, 119.8, 118.3. MS (EI), *m*/*z* 240.92 [M^+, 100%].

4.11. Methyl 9-oxo-9H-xanthene-2-carboxylate (2k)

White solid. m.p.: 217–219 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 9.02 (d, *J* = 2.2 Hz, 1H), 8.38–8.34 (m, 2H), 7.78–7.74 (m, 1H), 7.55–7.51 (m, 2H), 7.43 (t, *J* = 7.6 Hz, 1H), 3.97 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.7, 166.0, 158.9, 156.1, 135.5, 135.4, 129.5, 127.0, 126.2, 124.7, 122.0, 121.6, 118.6, 118.2, 52.5. MS (EI), *m*/*z* 254.06 [M^+, 100%].

4.12. 2-Methoxy-6-methyl-9H-xanthen-9-one (**2l**)

White solid. m.p.:137–139 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 3.1 Hz, 1H), 7.40 (d, *J* = 9.1 Hz, 1H), 7.31–7.28 (m, 1H), 7.25 (s, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 3.91 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.0, 156.4, 156.0, 151.1, 146.2, 126.6, 125.4, 124.7, 122.3, 119.4, 119.2, 117.8, 106.0, 56.0, 22.1. MS (EI), *m*/*z* 239.96 [M^+, 100%].

4.13. 2-Chloro-6-methyl-9H-xanthen-9-one (**2m**)

White solid. m.p.: 154–157 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.27 (d, *J* = 2.6 Hz, 1H), 8.19 (d, *J* = 8.1 Hz, 1H), 7.64–7.62 (m, 1H), 7.42 (d, *J* = 8.9 Hz, 1H), 7.27 (s, 1H), 7.20 (d, *J* = 8.2 Hz, 1H), 2.51 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.0, 156.3, 154.6, 147.0, 134.8, 129.7, 126.7, 126.1, 126.0, 122.9, 119.8, 119.4, 117.9, 22.2. MS (EI), *m*/*z* 243.90 [M^+, 100%].

4.14. 7H-Benzo[c]xanthen-7-one (**2n**)

White solid. m.p.: 159–161 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.1 Hz, 1H), 8.39 (d, *J* = 8.0 Hz, 1H), 8.25 (d, *J* = 8.7 Hz, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.78–7.74 (m, 1H), 7.72–7.65 (m, 4H), 7.45–7.42 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.0, 155.9, 153.8, 136.7, 134.4, 129.7, 128.2, 127.0, 126.7, 124.5, 124.2, 124.1, 123.0, 122.6, 121.6, 118.2, 117.7. MS (EI), *m*/*z* 246.08 [M^+, 100%].

4.15. 12H-Benzo[a]xanthen-12-one (**2o**)

White solid. m.p.: 152–154 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 10.07 (d, *J* = 8.8 Hz, 1H), 8.42 (d, *J* = 7.9 Hz, 1H), 8.07 (d, *J* = 9.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.76 (t, *J* = 7.8 Hz, 1H), 7.70 (t, *J* = 7.9 Hz, 1H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.51 (s, 1H), 7.49 (s, 1H), 7.42 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.5, 157.7, 154.7, 136.7, 134.0, 131.2, 130.2, 129.6, 128.4, 127.1, 126.8, 126.2, 124.4, 123.7, 118.1, 117.6, 114.6. MS (EI), *m*/*z* 246.10 [M^+, 100%].

4.16. 9-Methyl-7H-benzo[c]xanthen-7-one (**2p**)

White solid. m.p.: 162–165 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.1 Hz, 1H), 8.26 (d, *J* = 8.8 Hz, 1H), 8.17 (s, 1H), 7.91 (d, *J* = 7.7 Hz, 1H), 7.72–7.65 (m, 3H), 7.56 (s, 1H), 2.49 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.1, 154.2, 153.8, 136.6, 135.7, 134.4, 129.6, 128.2, 126.9, 126.0, 124.3, 123.9, 123.0, 122.2, 121.7, 118.0, 117.7, 21.1. MS (EI), *m*/*z* 259.95 [M^+, 100%].

4.17. 10-Methyl-7H-benzo[c]xanthen-7-one (**2q**)

White solid. m.p.: 166–169 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, *J* = 8.0 Hz, 1H), 8.25 (d, *J* = 3.1 Hz, 1H), 8.23 (d, *J* = 3.7 Hz, 1H), 7.88 (d, *J* = 7.7 Hz, 1H), 7.70–7.63 (m, 3H), 7.42 (s, 1H), 7.21 (d, *J* = 8.1 Hz, 1H), 2.52 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.8, 156.0, 153.6, 145.9, 136.6, 129.5, 128.2, 126.9, 126.4, 126.0, 124.2, 123.9, 122.9, 121.7, 120.3, 117.9, 117.7, 22.1. MS (EI), *m*/*z* 259.99 [M^+, 100%].

4.18. 11-Methoxy-7H-benzo[c]xanthen-7-one (**2r**)

White solid. m.p.: 275–277 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.77 (d, *J* = 7.1 Hz, 1H), 8.28 (d, *J* = 8.8 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.94 (d, *J* = 7.0 Hz, 1H), 7.74 (m, 3H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 7.9 Hz, 1H), 4.11 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.1, 153.6, 149.3, 146.4, 136.7, 129.7, 128.1, 127.1, 124.6, 124.3, 124.1, 123.5, 123.4, 121.6, 117.6, 117.5, 115.2, 56.7. MS (EI), *m*/*z* 275.92 [M^+, 100%].

4.19. 9-Chloro-7H-benzo[c]xanthen-7-one (**2s**)

Light yellow solid. m.p.: 224–226 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.2 Hz, 1H), 8.35 (d, *J* = 2.6 Hz, 1H), 8.24 (d, *J* = 8.7 Hz, 1H), 7.94 (d, *J* = 7.7 Hz, 1H), 7.76–7.69 (m, 4H), 7.65 (d, *J* = 8.9 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.9, 154.2, 153.8, 136.8, 134.6, 130.5, 130.0, 128.3, 127.3, 126.1, 124.6, 124.1, 123.5, 123.0, 121.5, 120.0, 117.5. MS (EI), *m*/*z* 279.86 [M^+, 100%].

4.20. 2-Bromo-7H-benzo[c]xanthen-7-one (**2t**)

White solid. m.p.: 218–221 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (s, 1H), 8.36 (d, *J* = 7.9 Hz, 1H), 8.22 (d, *J* = 8.7 Hz, 1H), 7.79–7.71 (m, 3H), 7.66–7.64 (m, 2H), 7.44 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.6, 155.7, 152.5, 135.0, 134.7, 132.9, 129.8, 126.7, 125.4, 125.3, 124.8, 123.8, 122.5, 122.2, 121.2, 118.3, 118.2. MS (EI), *m*/*z* 324.00[M^+, 85%], 189.01 [100%].

4.21. 14H-Dibenzo[a,h]xanthen-14-one (**2u**)

White solid.m.p.: 222–225 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 10.18 (d, *J* = 8.5 Hz, 1H), 8.70–8.68 (m, 1H), 8.39 (d, *J* = 8.7 Hz, 1H), 8.19 (d, *J* = 9.1 Hz, 1H), 7.97–7.92 (m, 2H), 7.82–7.79 (m, 2H), 7.76–7.69 (m, 3H), 7.63 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.4, 157.2, 152.1, 136.4, 136.2, 131.3, 130.5, 129.6, 129.5, 128.5, 128.3, 127.3, 127.1, 126.5, 124.6, 124.0, 122.8, 121.8, 119.7, 118.1, 115.5. MS (EI), *m*/*z* 296.14 [M^+, 100%].

4.22. 14H-Dibenzo[a,j]xanthen-14-one (**2v**)

Light yellow solid. m.p.: 189–192 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (d, *J* = 8.7 Hz, 2H), 8.03 (s, 2H), 7.85 (s, 2H), 7.79 (t, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.32 Hz, 2H), 7.51 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 180.3, 155.8, 135.9, 131.1, 130.6, 129.3, 128.5, 127.1, 126.2, 117.6, 116.5. MS (EI), *m*/*z* 296.02 [M^+, 100%].

4.23. 9H-Thioxanthen-9-one (**4a**)

Yellow solid. m.p.: 211–214 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (d, J = 8.1 Hz, 2H), 7.62–7.59 (m, 2H), 7.57–7.55 (m, 2H), 7.48 (t, J = 7.5 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 180.1, 137.4, 132.4, 130.0, 129.4, 126.4, 126.1. MS (EI), m/z 212.07 [M^+, 100%].

4.24. 2-Isopropyl-9H-thioxanthen-9-one (**4b**)

Yellow solid. m.p.: 75–79 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.62 (d, J = 8.2 Hz, 1H), 8.49 (s, 1H), 7.60–7.55 (m, 2H), 7.51 (s, 2H), 7.48–7.46 (m, 1H), 3.09–3.03 (m, 1H), 1.33 (d, J = 6.9 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 180.2, 147.5, 137.5, 134.6, 132.2, 131.5, 130.0, 129.4, 129.3, 127.3, 126.2, 126.1, 126.1, 34.1, 24.0.MS (EI), m/z 254.04 [M^+, 55%], 239.07 [100%].

4.25. 2,4-Diethyl-9H-thioxanthen-9-one (**4c**)

Yellow solid. m.p.: 72–75 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, J = 8.0 Hz, 1H), 8.36 (s, 1H), 7.60–7.57 (m, 1H), 7.47–7.44 (m, 1H), 7.36 (s, 1H), 2.90–2.86 (m, 2H), 2.79–2.74 (m, 2H), 1.38 (t, J = 7.6 Hz, 3H), 1.31 (t, J = 7.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 180.8, 142.2, 139.9, 137.1, 133.3, 132.1, 132.1, 129.8, 129.7, 128.9, 126.5, 126.4, 126.2, 28.7, 26.2, 15.6, 13.8.MS (EI), m/z 268.14 [M^+, 100%].

4.26. 2-Chloro-9H-thioxanthen-9-one (**4d**)

Yellow solid. m.p.: 151–154 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, J = 8.2 Hz, 1H), 8.57 (d, J = 2.3 Hz, 1H), 7.63 (t, J = 7.5 Hz, 1H), 7.56 (d, J = 8.7 Hz, 2H), 7.51–7.47 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.0, 137.0, 135.6, 132.8, 132.7, 132.7, 130.4, 130.1, 129.5, 128.9, 127.6, 126.7, 126.2. MS (EI), m/z 245.93 [M^+, 100%].

4.27. 2-(Trifluoromethyl)-9H-thioxanthen-9-one (**4e**)

Yellow solid.m.p.: 145–148 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.59 (d, J = 8.1 Hz, 1H), 7.79 (d, J = 8.5 Hz, 1H), 7.67–7.63 (m, 2H), 7.56 (d, J = 8.1 Hz, 1H), 7.51 (t, J = 7.6 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 179.1, 141.2, 136.6, 133.0, 130.1, 129.2, 129.0 128.9 (d, J = 33.6 Hz), 128.2 (d, J = 3.3 Hz), 127.3 (d, J = 4.1 Hz), 127.1, 127.0, 126.2, 123.8 (d, J = 273.4 Hz). MS (EI), m/z 279.87 [M^+, 100%].

4.28. 10-Methylacridin-9(10H)-one (**6a**)

Yellow solid.m.p.: 172–175 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.53 (d, J = 8.0 Hz, 2H), 7.69–7.66 (m, 2H), 7.47 (d, J = 8.6 Hz, 2H), 7.26 (t, J = 7.4 Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.2, 142.6, 133.9, 127.8, 122.6, 121.3, 114.8, 33.7. MS (EI), m/z 208.97 [M^+, 100%].

4.29. 4-Methoxy-10-methylacridin-9(10H)-one (**6b**)

Yellow solid.m.p.: 82–85 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.44 (d, J = 8.0 Hz, 1H), 8.12 (d, J = 7.7 Hz, 1H), 7.69 (t, J = 7.8 Hz, 1H), 7.50 (d, J = 8.5 Hz, 1H), 7.27–7.18 (m, 3H), 3.99 (s, 3H), 3.95 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.5, 150.0, 145.7, 135.8, 133.7, 127.2, 125.6, 122.9, 122.0, 121.4, 119.3, 116.2, 115.7, 56.5, 41.5. MS (EI), m/z 238.97 [M^+, 85%], 223.94 [100%].

4.30. 2-Chloro-10-methylacridin-9(10H)-one (**6c**)

Yellow solid. m.p.:170–173 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (d, J = 8.1 Hz, 1H), 8.35 (d, J = 2.6 Hz, 1H), 7.67–7.63 (m, 1H), 7.50–7.48 (m, 1H), 7.40 (d, J = 8.6 Hz, 1H), 7.32 (d, J = 9.2 Hz, 1H), 7.22 (t, J = 7.6 Hz, 1H), 3.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.9, 142.3, 140.8, 134.1, 133.8, 127.7, 127.2, 126.7, 123.1, 122.3, 121.6, 116.7, 114.9, 33.8. MS (EI), m/z 243.05 [M^+, 100%].

4.31. 10-Benzylacridin-9(10H)-one (**6d**)

Yellow solid. m.p.: 172–174 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, J = 8.1 Hz, 2H), 7.62 (t, J = 7.9 Hz, 2H), 7.37–7.28 (m, 7H), 7.21 (d, J = 7.5 Hz, 2H), 5.60 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 178.4, 142.7, 135.6, 134.2, 129.4, 127.9, 127.9, 125.8, 122.7, 121.8, 115.3, 50.9. MS (EI), m/z 284.99 [M^+, 100%].

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

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