

Multicomponent, solvent-free synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]-xanthen-11-one derivatives catalysed by cyanuric chloride

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Abstract. An efficient and direct protocol for the preparation of 12-aryl-8,9,10,12-tetrahydro-benzo[*a*]xanthen-11-one derivatives employing a three-component one-pot reaction of aryl aldehydes, 2-naphthol and cyclic 1,3-dicarbonyl compounds in the presence of a catalytic amount of cyanuric chloride (2,4,6-trichloro-1,3,5-triazine, TCT) under solvent-free conditions is described. The desired products are obtained in high yields with short reaction times.

Keywords. Xanthenes; aldehydes; 2-naphthol; multicomponent reactions; cyanuric chloride; solvent-free condition.

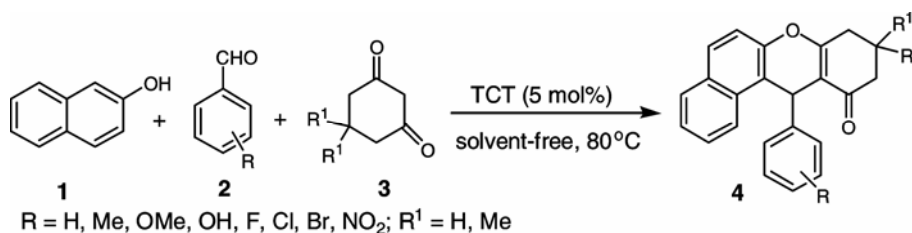
1. Introduction

Xanthenes and its derivatives are known as an important class of heterocyclic compounds widely used as leuco-dye,¹ in laser technology,² and pH sensitive fluorescent materials.³ They possess a broad range of useful pharmacological activities, including antibacterial,⁴ antiviral,⁵ and anti-inflammatory activities.⁶ These compounds are also utilized as antagonists for paralyzing action of zoxazolamine⁷ and in photodynamic therapy.⁸ The high profile of biological applications of compounds with xanthene structures has prompted extensive studies for their synthesis.^{9–12} A few methods have been developed for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one derivative; the most common method is the condensation of an aryl aldehyde, 2-naphthol and cyclic 1,3-dicarbonyl compound in boiling 1,2-dichloroethane in the presence of NaHSO₄·SiO₂¹³ or strontium triflate.¹⁴ However, in spite of their potential utility, some of these methods suffer drawbacks such as the use of toxic and hazardous solvents, unsatisfactory product yields, expensive catalyst, and prolonged reaction times. Hence, the development of new and simple synthetic

methods for the preparation of heterocyclic compounds containing xanthone fragment remains an interesting challenge.

Recently, multicomponent reactions (MCRs) have attracted considerable attention due to significant advantages such as simplicity of operation, reduction of isolation and purification steps, and minimization of costs, time, and waste production.¹⁵ MCRs are particularly useful to provide expedient approaches to a wide range of compounds of biological and pharmaceutical interest. In the context of MCR, ortho-quinone methides (*o*-QMs) have been utilized in many elegant tandem processes.¹⁶ Recently, we demonstrated that a mixture of aldehydes, 2-naphthol and amide when heated at 100°C in the presence of cyanuric chloride under solvent-free condition, afforded amidolky naphthol derivatives.¹⁷ This reaction proceeds through *in situ* formation of ortho-quinone methide intermediates. Previously, we also reported the synthesis 1,8-dioxo-octahydro-xanthenes by treatment of aromatic aldehydes and 5,5-dimethylcyclohexane-1,3-dione.¹⁸ Due to our interest in the multicomponent syntheses and in continuation of our work on the development of new synthetic methodologies,¹⁹ we report here, a simple and facile protocol for the synthesis of a series of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one

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Scheme 1. Synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one derivatives.

derivatives from aldehydes, 2-naphthol and cyclic 1,3-dicarbonyl compounds using cyanuric chloride as a catalyst under solvent-free conditions (scheme 1).

2. Experimental

Melting points were recorded on X-4 apparatus (Beijing Tech Instrument Co., Ltd) and are uncorrected. IR spectra were recorded on a Shimadzu FTIR-8900 spectrophotometer with KBr optics. ^1H NMR and ^{13}C NMR spectra were recorded with Varian 400 spectrometer using TMS as an internal standard. Mass spectra were recorded on a ThermoFinnigan LCQ Advantage instrument with an ESI source (4.5 KeV). Elemental analyses were carried out using a Vario EL III CHNOS Elemental Analyzer.

2.1 Typical procedure for the preparation of 4b

2,4,6-Trichloro-1,3,5-triazine (TCT) (0.05 mmol) was added to a mixture of 2-naphthol (1 mmol), 4-methylbenzaldehyde (1.0 mmol) and dimedone (1.2 mmol) and the reaction mixture was heated at 80°C . The progress of the reaction was monitored by TLC. After completion of the reaction, water (10 mL) was added and the mixture was extracted with ethyl acetate (3×10 mL). The extract was dried (MgSO_4) and evaporated to give the crude product. The crude product was purified by chromatography over silica gel using ethyl acetate/cyclohexane as eluent to afford pure product.

2.1a 9,9-Dimethyl-12-p-tolyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (4b): White solid, m.p. $175\text{--}176^\circ\text{C}$; IR (KBr) 3014, 2870, 1647, 1618, 1597, 1400, 1371, 1226, 1186, 1147, 1028, 812 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): $\delta = 0.98$ (s, 3H), 1.12 (s, 3H), 2.25 and 2.29 (AB system, $J = 16.4$ Hz, 2H), 2.57 (s, 2H), 5.67 (s, 1H), 6.98 (d, $J = 7.6$ Hz, 2H),

7.22–7.38 (m, 4H), 7.43 (d, $J = 8.0$ Hz, 1H), 7.76 (d, $J = 7.6$ Hz, 2H), 8.01 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 21.1, 27.3, 29.3, 32.6, 34.3, 41.4, 50.9, 114.4, 117.1, 117.9, 123.7, 124.9, 127.0, 128.3, 128.4, 128.7, 129.0, 131.4, 131.5, 135.7, 141.9, 147.7, 163.8, 197.0$; ESI-MS $m/z = 369$ ($\text{M} + 1$) $^+$; Anal. Calcd for $\text{C}_{26}\text{H}_{24}\text{O}_2$: C, 84.75; H, 6.57. Found: C, 85.02; H, 6.38.

2.1b 12-(4-Fluorophenyl)-9,9-dimethyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (4e): White solid, m.p. $185\text{--}186^\circ\text{C}$; IR (KBr) 3037, 2954, 2881, 1651, 1618, 1595, 1508, 1398, 1375, 1226, 1184, 1026, 839 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): $\delta = 0.96$ (s, 3H), 1.12 (s, 3H), 2.24 and 2.31 (AB system, $J = 16.4$ Hz, 2H), 2.57 (s, 2H), 5.70 (s, 1H), 6.85 (t, $J = 8.8$ Hz, 2H), 7.28–7.46 (m, 5H), 7.78 (t, $J = 8.0$ Hz, 2H), 7.93 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 27.1, 29.4, 32.2, 34.0, 41.4, 50.9, 114.1, 114.9, 115.1, 117.1, 117.4, 123.6, 125.0, 127.1, 128.5, 129.1, 129.9, 131.3, 131.5, 140.6, 147.7, 163.9, 196.9$; ESI-MS $m/z = 373$ ($\text{M} + 1$) $^+$; Anal. Calcd for $\text{C}_{25}\text{H}_{21}\text{FO}_2$: C, 80.62; H, 5.68. Found: C, 80.80; H, 5.45.

2.1c 12-(3,4-Dichlorophenyl)-9,9-dimethyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (4g): White solid, m.p. $181\text{--}182^\circ\text{C}$; IR (KBr) 3138, 2962, 1649, 1637, 1618, 1596, 1558, 1398, 1384, 1373, 1234, 1202, 1070, 819 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): $\delta = 0.98$ (s, 3H), 1.12 (s, 3H), 2.24 and 2.30 (AB system, $J = 16.4$ Hz, 2H), 2.58 (s, 2H), 5.70 (s, 1H), 7.19–7.24 (m, 1H), 7.30–7.48 (m, 5H), 7.78–7.85 (m, 2H), 7.86 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 27.2, 29.2, 32.3, 34.1, 41.3, 50.7, 113.3, 116.3, 117.1, 123.2, 125.1, 127.3, 128.0, 129.4, 130.1, 130.3, 131.0, 131.5, 132.2, 144.9, 147.7, 164.3, 196.8$; ESI-MS $m/z = 423$ ($\text{M} + 1$) $^+$; Anal. Calcd for $\text{C}_{25}\text{H}_{20}\text{Cl}_2\text{O}_2$: C, 70.93; H, 4.76. Found: C, 71.05; H, 4.68.

2.1d 12-(4-Bromophenyl)-9,9-dimethyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4h**): White solid, m.p. 208–209°C; IR (KBr) 3064, 1643, 1618, 1593, 1514, 1483, 1400, 1375, 1220, 1174, 1008, 837 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 0.97 (s, 3H), 1.12 (s, 3H), 2.25 and 2.30 (AB system, J = 16.4 Hz, 2H), 2.57 (s, 2H), 5.67 (s, 1H), 7.20–7.46 (m, 7H), 7.78 (d, J = 7.6 Hz, 2H), 7.90 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 27.1, 29.4, 32.3, 34.3, 41.4, 50.9, 113.7, 116.9, 117.1, 120.2, 123.4, 125.0, 127.1, 128.5, 129.1, 130.2, 131.2, 131.3, 1315, 143.8, 147.7, 164.1, 19.9; ESI-MS m/z = 433 (M + 1)⁺; Anal. Calcd for C₂₅H₂₁BrO₂: C, 69.29; H, 4.88. Found: C, 69.53; H, 4.52.

2.1e 9,9-Dimethyl-12-(3-nitrophenyl)-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4i**): White solid, m.p. 166–167°C; IR (KBr) 3139, 2958, 2912, 1649, 1618, 1597, 1400, 1375, 1348, 1224, 1176, 1085, 812 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 0.94 (s, 3H), 1.12 (s, 3H), 2.23 and 2.31 (AB system, J = 16.4 Hz, 2H), 2.59 (s, 2H), 5.80 (s, 1H), 7.32–7.39 (m, 3H), 7.34 (d, J = 7.6 Hz, 1H), 7.77–7.80 (m, 3H), 7.85 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 8.14 (d, J = 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 27.1, 29.3, 32.2, 34.7, 41.3, 50.7, 113.1, 116.0, 117.3, 121.6, 123.1, 123.2, 125.2, 127.4, 128.7, 129.1, 129.6, 130.9, 131.6, 134.8, 146.8, 147.8, 148.3, 164.6, 196.9; ESI-MS m/z = 400 (M + 1)⁺; Anal. Calcd for C₂₅H₂₁NO₄: C, 75.17; H, 5.30; N, 3.51. Found: C, 75.32; H, 5.12; N, 3.70.

2.1f 12-[4-(9,9-Dimethyl-8-oxo-8,9,10,11-tetrahydro-7H-benzo[c]xanthen-7-yl)-phenyl]-9,9-dimethyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4k**): White solid, m.p. 310–311°C; IR (KBr) 3055, 1670, 1618, 1508, 1375, 1226, 1174, 1130, 954, 819 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 0.79 (s, 6H), 1.04 (s, 3H), 1.06 (s, 3H), 2.10–2.24 (m, 4H), 2.46 (s, 2H), 2.50 (s, 2H), 5.56 (s, 1H), 5.60 (s, 1H), 7.09 (d, J = 8.0 Hz, 4H), 7.28–7.34 (m, 6H), 7.69–7.87 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 27.2, 29.0, 32.2, 33.8, 41.3, 50.9, 114.3, 117.0, 117.6, 123.9, 124.8, 126.9, 128.1, 128.2, 128.6, 131.3, 131.4, 142.3, 147.9, 163.9, 197.0; ESI-MS m/z = 631 (M + 1)⁺; Anal. Calcd for C₄₄H₃₈O₄: C, 83.78; H, 6.07. Found: C, 83.96; H, 5.91.

2.1g 12-p-Tolyl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4m**): White solid, m.p. 305–

306°C; IR (KBr) 3024, 2951, 2893, 2829, 1651, 1618, 1508, 1456, 1402, 1336, 1226, 1188, 1176, 1126, 999, 954 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 1.91–1.94 (m, 2H), 2.21 (s, 3H), 2.31–2.48 (m, 2H), 2.56–2.76 (m, 2H), 5.70 (s, 1H), 6.98 (d, J = 8.0 Hz, 1H), 7.03 (d, J = 8.0 Hz, 1H), 7.19 (d, J = 8.0 Hz, 1H), 7.25 (d, J = 8.0 Hz, 1H), 7.31–7.44 (m, 3H), 7.76 (t, J = 8.0 Hz, 2H), 7.97 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 20.3, 21.1, 27.2, 27.7, 34.2, 37.1, 115.7, 117.0, 117.8, 123.7, 124.9, 127.0, 128.3, 128.4, 128.8, 129.0, 131.4, 135.7, 142.2, 147.7, 163.9, 165.6, 196.6; ESI-MS m/z = 341 (M + 1)⁺; Anal. Calcd for C₂₄H₂₀O₂: C, 84.68; H, 5.92. Found: C, 84.82; H, 6.05.

2.1h 12-(4-Fluophenyl)-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4q**): White solid, m.p. 210–212°C; IR (KBr) 3134, 1652, 1618, 1600, 1506, 1438, 1398, 1382, 1359, 1226, 1188, 1178, 999, 846 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 1.95–2.07 (m, 2H), 2.31–2.44 (m, 2H), 2.54–2.71 (m, 2H), 5.66 (s, 1H), 6.61 (d, J = 8.4 Hz, 1H), 6.66 (d, J = 8.4 Hz, 1H), 7.14 (d, J = 8.4 Hz, 1H), 7.18 (d, J = 8.4 Hz, 1H), 7.30–7.42 (m, 3H), 7.76 (t, J = 8.4 Hz, 2H), 7.94 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 27.1, 31.1, 33.9, 37.0, 114.7, 114.9, 115.1, 117.0, 125.0, 127.0, 128.5, 129.0, 129.9, 130.0, 131.2, 131.5, 140.8, 147.7, 164.0, 165.7, 197.1; ESI-MS m/z = 345 (M + 1)⁺; Anal. Calcd for C₂₃H₁₇FO₂: C, 80.22; H, 4.98. Found: C, 80.13; H, 5.12.

2.1i 12-(4-Bromophenyl)-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (**4t**): White solid, m.p. 208–209°C; IR (KBr) 2941, 2899, 1651, 1616, 1595, 1485, 1400, 1385, 1229, 1190, 1130, 1010, 833 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ = 1.98–2.08 (m, 2H), 2.31–2.44 (m, 2H), 2.56–2.73 (m, 2H), 5.69 (s, 1H), 7.17–7.22 (m, 1H), 7.28–7.45 (m, 6H), 7.77–7.80 (m, 2H), 7.87 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ = 20.3, 27.1, 34.2, 36.9, 115.0, 116.9, 117.0, 120.1, 123.4, 125.0, 127.1, 128.5, 129.1, 130.3, 131.2, 131.4, 144.1, 147.7, 164.2, 165.8, 197.1; ESI-MS m/z = 405 (M + 1)⁺; Anal. Calcd for C₂₃H₁₇BrO₂: C, 68.16; H, 4.23. Found: C, 68.32; H, 4.08.

3. Results and discussion

Initial studies were conducted using the model reaction of 2-naphthol (1 mmol), 4-chlorobenzaldehyde

Table 1. The synthesis of 12-(4-chlorophenyl)-9,9-dimethyl-8,9,10,12-tetrahydrobenzo-[a]xanthen-11-one (**4f**) under different conditions^a.

Entry	Catalyst	Catalyst loading (mol%)	Time (h)	Yield (%) ^b
1	No		12	Trace
2	Benzeneboronic acid	10	1	48
3	Sulfamic acid	10	1	59
4	<i>N</i> -Bromosuccinimide	10	1	46
5	ZrOCl ₂ ·8H ₂ O	10	1	75
6	KAl(SO ₄) ₂ ·12H ₂ O	10	1	68
7	KAl(SO ₄) ₂ ·12H ₂ O/SiO ₂	10	1	70
8	SbCl ₃ /SiO ₂	10	1	52
9	SbCl ₃ /montmorillonite K-10	10	1	55
10	TCT	1	1	75
11	TCT	5	0.5	92
12	TCT	10	0.5	90
13	TCT	5	1	0 ^c
14	TCT	5	1	80 ^d
15	TCT	5	3	36 ^e

^aThe reaction was carried out according to general experimental procedure. ^bIsolated yields. ^cThe reaction was carried out at room temperature. ^dThe reaction carried out at 50°C. ^eThe reaction carried out in boiling 1,2-dichloroethane.

(1 mmol), and 5,5-dimethyl-1,3-cyclohexanedione (1.2 mmol) under various conditions. The results are summarized in table 1. Only a trace amount of the product was obtained when the reaction was carried out in the absence of catalyst even after 12 h (table 1, entry 1). TCT was the most effective catalyst in terms of yield of product (92%) while other catalysts such as benzeneboronic acid, sulfamic acid, *N*-bromosuccinimide, ZrOCl₂·8H₂O, KAl(SO₄)₂·12H₂O, SbCl₃/SiO₂, and SbCl₃/montmorillonite K-10, the yield of the product was lower (46–75%). Further, TCT is a stable, non-volatile, inexpensive, commercially available and easy-to-handle reagent.²⁰ It has been utilized for many synthetic transformations.²¹ Optimization of the reaction conditions was undertaken using TCT as a catalyst. The result showed that 5 mol% of TCT was sufficient to get a fairly high yield (table 1, entry 11). With 1 mol% of TCT, a lower yield was observed even after 1 h. An higher amount of the catalyst (10 mol%) scale did not improve the yield. We also tested the effect of reaction temperature on the catalysed reaction. When it was carried out at 80°C, the maximum yield was obtained in a short duration. Lower yield was obtained when the reaction was carried out in boiling 1,2-dichloroethane (table 1, entry 15).

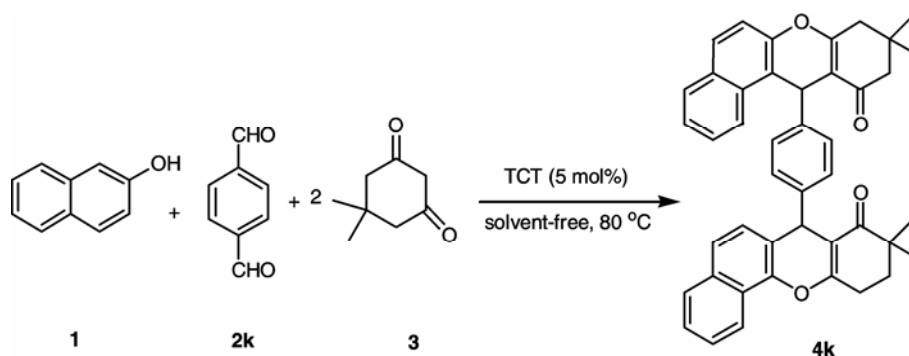
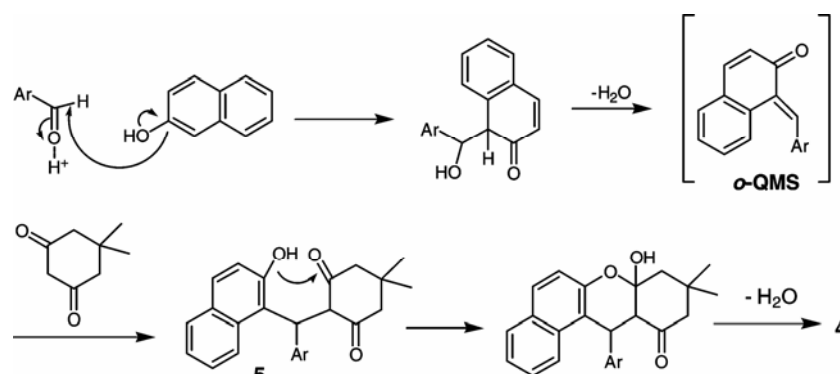
After optimizing the conditions, we next evaluated the generality and scope of this TCT catalysed reaction. An array of structurally and electronically

divergent aromatic aldehydes, cyclic 1,3-dicarbonyl compounds and 2-naphthol were tested and the results are summarized in table 2. Unlike previously reported method where 4–7 h were required for completion of this reaction in the presence of NaHSO₄·SiO₂ in refluxing 1,2-dichloroethane,¹³ the present methodology afforded high yields of the products **4a–4v** within short times (30–70 min). In general, aldehydes bearing electron-donating groups need slightly longer times to complete the reaction as compared to aldehydes containing electron-withdrawing groups. Various functional groups such as NO₂, Cl, Br, OH and OCH₃ were tolerated. This reaction was further explored for the synthesis of *bis*-benzo[a]xanthen-11-one compound (table 2, entry **4k**) in high yields by the reaction of terephthalaldehyde with 2 equiv. 2-naphthol and 5,5-dimethyl-1,3-cyclohexanedione under similar conditions (scheme 2).

A possible mechanism for the formation of **4a–4v** is proposed in scheme 3. Adventitious entry of moisture leads to the release of HCl. The *in situ* generated HCl acts as a protic acid to activate the carbonyl oxygen. The reaction proceeds through the intermediate *o*-QMs, formed *in situ* by the reaction of 2-naphthol with aromatic aldehydes as reported in the literature.²² Subsequent addition of dimedone to the *o*-QMs forms intermediate **5**, followed by cyclization to give the corresponding products **4**, accompanied by loss of H₂O.

Table 2. Synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one derivatives.

Entry	Aldehyde	R	Time (min)	Yield (%) ^a	m.p. (°C)
a	PhCHO	Me	50	90	149–150 ²³
b	4-MeC ₆ H ₄ CHO	Me	70	87	175–176 ²³
c	4-MeOC ₆ H ₄ CHO	Me	70	86	207–208 ¹³
d	4-OHC ₆ H ₄ CHO	Me	50	84	151–152 ¹³
e	4-FC ₆ H ₄ CHO	Me	30	91	185–186
f	4-ClC ₆ H ₄ CHO	Me	30	92	188–189 ¹³
g	3,4-Cl ₂ C ₆ H ₃ CHO	Me	30	92	181–182
h	4-BrC ₆ H ₄ CHO	Me	30	93	186–187
i	3-NO ₂ C ₆ H ₄ CHO	Me	35	85	166–167
j	4-NO ₂ C ₆ H ₄ CHO	Me	30	93	175–176 ²³
k	4-CHOC ₆ H ₄ CHO	Me	50	84	310–311
l	PhCHO	H	50	88	189–190 ¹³
m	4-MeC ₆ H ₄ CHO	H	70	86	205–206 ²³
n	4-MeOC ₆ H ₄ CHO	H	70	85	181–182 ²³
o	4-OHC ₆ H ₄ CHO	H	70	83	269–270
p	3-OMe-4-OHC ₆ H ₃ CHO	H	60	85	193–194 ²³
q	4-FC ₆ H ₄ CHO	H	30	92	210–212
r	3-ClC ₆ H ₄ CHO	H	30	91	209–210 ²⁴
s	4-ClC ₆ H ₄ CHO	H	30	91	205–206 ²³
t	4-BrC ₆ H ₄ CHO	H	30	90	208–209
u	3-NO ₂ C ₆ H ₄ CHO	H	30	92	235–236 ²³
v	4-NO ₂ C ₆ H ₄ CHO	H	30	90	234–235 ²³

^aYields refer to isolated yield.**Scheme 2.** Synthesis of *bis*-benzo[a]xanthen-11-one compound (**4k**).**Scheme 3.** A proposed mechanism.

3. Conclusions

In conclusion, we have described a very simple, convenient and practical method for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one derivatives through a three-component one-pot reaction of aryl aldehydes, 2-naphthol and cyclic 1,3-dicarbonyl compounds. The procedure clearly demonstrates that TCT is an excellent catalyst for the synthesis of xanthene-based compounds.

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