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Efficient and Clean Synthesis of 1,8-Dioxooctahydroxanthenes in Aqueous Medium

SANTOSH B. PORE

Organic Research Laboratory, Post-Graduate Department of Chemistry, Balwant College, Vita-415 311, India

Corresponding author: Tel.: +91 2347 272096; E-mail: santoshbpore@gmail.com

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In this work, an efficient synthesis of 1,8-dioxooctahydroxanthenes in aqueous medium is reported. The reaction was catalyzed by aqueous extract of plant material (pericarp of *Sapindus trifoliatus* fruit), which makes this protocol 'green' and eco-friendly. Differently substituted aromatic aldehydes underwent condensation with dimedone resulting in the formation of only the desired product in good to excellent yield. This reaction provides an alternative route to useful organic substances, employing simple reaction conditions, without organic solvents and hazardous chemical catalysts.

Keywords: 1,8-Dioxooctahydroxanthenes, *Sapindus trifoliatus*, Aqueous medium, Green catalyst.

INTRODUCTION

Xanthenes have attracted a number of research efforts in synthetic organic chemistry due to their wide range of biological and pharmacological properties like antiviral [1], antibacterial [2] and anti-inflammatory activities [3], as well as in photodynamic therapy [4] and as antagonists of the paralyzing action of zoxazolamine [5]. Xanthenes are also available from many natural sources. The prominent among them are dibenzoxanthenes, hexahydroxanthene and octahydroxanthene.

Xanthenes are frequently occurring motifs in a number of natural products [6] and have been used as versatile synthons due to the inherent reactivity of inbuilt pyran ring [7]. Octahydroxanthene derivatives containing a structural unit of benzopyrans can be used as antispasm agents [8] and fluorescent fuel [9]. Furthermore, due to their useful spectroscopic properties, they are used as dyes and pigments [10,11], in laser technologies [12] and in fluorescent materials for visualization of biomolecules [13].

Xanthenes are also known to possess many pharmacological properties. Allanxanthone C, a xanthene derivative obtained from *Allanblackia monticola* exhibits excellent biological properties [14]. Ehretianone, a quinonoid xanthene obtained from *Ehretia buxifolia* is reported to possess anti-snake venom activity [15]. A spiro-compound of xanthene, xanthene spiropiperidine, is well-known as a sedative and antihistaminic agent [16]. Cervinomycin A_1 possesses antibiotic activity against anaerobic bacteria such as *Clostridium perfringens*, *Peptococcus prevotii* and *Bacteroides fragilis* [17].

1,8-Dioxooctahydroxanthene is an important member of xanthene family. Tetramethyl derivative of this synthetically useful precursor is generally obtained by condensation of 5,5-dimethyl-1,3-cyclohexanedione (dimedone) with an aromatic aldehyde. Literature survey shows a number of routes to obtain 1,8-dioxooctahydroxanthene and its variants, employing either different starting materials or catalysts. Literature methods include catalysts like molecular iodine [18], zirconyl triflate [19], nano-TiO_2 [20], nanoparticles of iron(II, III) oxide [21], iron(III) complex [22], nano-SPA [23] and barium perchlorate [24] among others. However, contemporary research in organic chemistry desires protocols to be in conformity with green chemistry principles. An attempt to achieve 'green synthesis' of 1,8-dioxooctahydroxanthene using plant-derived catalyst with excellent results is decribed in this work.

EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. Reagent grade chemicals were purchased from s.d. Fine Chem., Spectrochem Co. and others and used as received without further purification. IR spectra were recorded on a Perkin-Elmer 1310 FT-IR spectrometer using KBr pellets. ^{1}H NMR

(300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on Varian instrument using $CDCl_3$ as solvent and TMS as internal reference. The progress of reaction was monitored by TLC run on silica gel G (Merck). Mass spectra were recorded on a Shimadzu QP 2010 GCMS with an ion source temperature of 200 ºC.

General procedure for the synthesis of 3,3,6,6-tetramethyl-9-(substituted)-1,8-dioxooctahydroxanthene: The mixture of 5,5-dimethyl-1,3-cyclohexanedione (10 mmol) and benzaldehyde (5 mmol) was mixed with aqueous extract of pericarp of *Sapindus trifoliatus* fruit (10 mL) and stirred magnetically at 80 ºC for appropriate time. The progress of the reaction was monitored at an interval of 60 minutes with the help of TLC (ethyl acetate/n-hexane in 2:8). The reaction mixture was washed by cold water to remove traces of bio-catalyst and then filtered (**Scheme-I**). The remaining solid material was washed with cold water. The solid product was recrystallized by using ethanol to yield pure product.

3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene (Table-1, entry 1): ^1H NMR (300 MHz, $CDCl_3$) δ ppm: 0.90 (s, 6H), 1.04 (s, 6H), 2.11 (d, *J* = 9 Hz, 2H), 2.27 (d, *J* = 9.1 Hz, 2H), 2.54 (d, *J* = 10.2 Hz, 2H), 2.60 (d, *J* = 10.3 Hz, 2H), 4.57 (s, 1H), 7.10 (t, J = 4.2 Hz, 1H), 7.16 (d, *J* = 4.1 Hz, 2H), 7.23 (t, *J* = 4.2 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.3, 29.3, 31.8, 32.2, 40.9, 50.7, 115.6, 126.4, 128.4, 128.9, 143.6, 161.7, 197.3. Anal. calcd. found (%) for $C_{23}H_{26}O_3$: C 78.83 (78.64), H 7.48 (7.53).

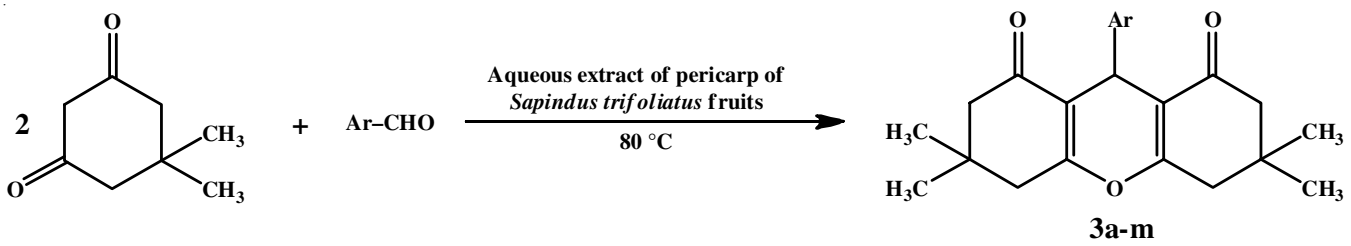
3,3,6,6-Tetramethyl-9-(4-methylphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 2): ^1H NMR (300 MHz, $CDCl_3$) δ ppm: 0.98 (s, 6H), 1.18 (s, 6H), 2.18-2.29 (m, 7H), 2.45 (s, 4H), 4.79 (s, 1H), 7.06 (d , *J* = 4.5 Hz, 2H), 7.18 (d, *J* = 4.2 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): ? (ppm) 20.9, 27.3, 28.9, 31.7, 32.6, 41.5, 51.4, 116.7, 127.9, 129.6, 135.8, 141.3, 161.4, 195.3. Anal. calcd. found (%) for $C_{24}H_{28}O_3$: C 79.09 (78.73), H 7.74 (7.69).

3,3,6,6-Tetramethyl-9-(3-nitrophenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 3): ^1H NMR (300 MHz, $CDCl_3$) δ (ppm): 1.07 (s, 6H), 1.12 (s, 6H), 2.16 (d, *J* = 9.2 Hz, 2H), 2.27 (d, *J* = 9.2 Hz, 2H), 2.54 (t, *J* = 10.9 Hz, 4H), 4.87 (s, 1H), 7.42 (t, *J* = 5.3 Hz, 1H), 7.78 (d, *J* = 4.5 Hz, 1H), 7.96 (d, *J* = 4.9 Hz, 1H), 8.12 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 26.9, 29.8, 31.8, 32.6, 41.5, 49.8, 112.9, 122.8, 123.5, 129.2, 135.3, 145.7, 148.7, 164.2, 196.3. Anal. calcd. found (%) for $C_{23}H_{25}NO_5$: C 69.86 (69.78), H 6.37 (6.52), N 3.54 (3.49).

3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 4): ^1H NMR (300 MHz, $CDCl_3$) δ ppm: 1.05 (s, 6H), 1.14 (s, 6H), 2.14 (d, *J* = 9.8 Hz, 2H), 2.25 (d, *J* = 9.9 Hz, 2H), 2.48 (t, *J* = 11.2 Hz, 4H), 4.85 (s, 1H), 7.46 (d, *J* = 4.8 Hz, 2H), 8.11 (2H, *J* = 4.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.3, 29.8, 32.1, 33.4, 41.5, 51.0, 115.3, 123.2, 130.4, 145.9, 151.6, 163.5, 195.8. Anal. calcd. found (%) for $C_{23}H_{25}NO_5$: C 69.86 (69.89), H 6.37 (6.23), N 3.54 (3.58).

3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 5): ^1H NMR (300 MHz, $CDCl_3$) δ ppm: 0.96 (s, 6H), 1.08 (s, 6H), 2.13 (d, *J* = 9.6 Hz, 2H), 2.26 (d, *J* = 9.7 Hz, 2H), 2.53 (d, 2H), 2.61 (d, *J* = 10.5 Hz, 2H), 4.54 (s, 1H), 7.21 (d, *J* = 4.9 Hz, 2H), 7.26 (d, *J* = 4.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.5, 28.8, 31.4, 32.6, 40.3, 51.2, 114.9, 127.5, 129.2, 132.4, 143.6, 162.5, 196.4. Anal. calcd. found (%) for $C_{23}H_{25}O_3Cl$: C 71.77 (71.68), H 6.55 (6.68).

3,3,6,6-Tetramethyl-9-(4-methoxyphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 6): ^1H NMR (300 MHz, $CDCl_3$) δ ppm: 1.01 (s, 6H), 1.12 (s, 6H), 2.18 (d, *J* = 12.1 Hz,



Scheme-I

TABLE-1
SYNTHESIS OF SUBSTITUTED 1,8-DIOXOOCTAHYDROXANTHENES

Entry	Ar-	Product	Time (h)	Yield (%)[b]	m.p. (°C)	
					Observed	Literature
1	C_6H_5-	**3a**	2.0	92	205-207	205-206 [19]
2	4-$CH_3C_6H_4$-	**3b**	2.5	90	214-216	216-217 [19]
3	3-$NO_2C_6H_4$-	**3c**	1.5	93	165-166	167-168 [19]
4	4-$NO_2C_6H_4$-	**3d**	1.5	93	218-220	221-223 [19]
5	4-ClC_6H_4-	**3e**	1.5	90	226-228	229-230 [28]
6	4-$MeOC_6H_4$-	**3f**	3.0	85	240-242	241-243 [19]
7	4-OHC_6H_4-	**3g**	2.5	88	242-245	246-248 [19]
8	4-OH(3-MeO)C_6H_3-	**3h**	3.0	85	226-228	225-227 [19]
9	3,4-$MeOC_6H_4$-	**3i**	3.0	80	211-214	209-212 [29]
10	4-(N,N-CH_3)C_6H_4-	**3j**	2.5	85	221-224	218-220 [28]
11	3-ClC_6H_4-	**3k**	1.5	90	177-180	179-181 [28]
12	4-CNC_6H_4-	**3l**	1.5	92	214-216	216-217 [25]
13	4-BrC_6H_4-	**3m**	2.0	90	224-227	226-229 [25]

[a]Reaction conditions: 1:2 moles of aldehyde:dimedone, room temperature, 10 mL biocatalyst; [b]Isolated yield after purification.

2H), 2.25 (d, *J* = 12.1 Hz, 2H), 2.48 (s, 4H), 3.75 (s, 3H), 4.72 (s, 1H), 6.77 (d, *J* = 6.6 Hz, 2H), 7.22 (d, *J* = 6.5 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.3, 29.3, 30.9, 32.2, 40.9, 50.8, 55.1, 113.5, 115.8, 129.3, 136.5, 157.9, 162.1, 196.5. Anal. calcd. found (%) for $C_{24}H_{28}O_4$: C 75.76 (75.94), H 7.42 (7.31).

3,3,6,6-Tetramethyl-9-(4-hydroxyphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 7): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 0.96 (s, 6H), 1.08 (s, 6H), 2.11-2.24 (m, 4H), 2.47 (s, 4H), 4.62 (s, 1H), 6.55 (d, *J* = 7.4 Hz, 2H), 7.11 (d, *J* = 7.4 Hz, 2H), 7.19 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 29.1, 29.8, 31.3, 32.6, 41.2, 50.5, 114.7, 117.2, 129.8, 135.9, 155.6, 163.5, 196.9. Anal. calcd. found (%) for $C_{23}H_{26}O_4$: C 75.38 (75.57), H 7.15 (7.31).

3,3,6,6-Tetramethyl-9-(4-hydroxy-3-methoxyphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 8): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 0.96 (s, 6H), 1.08 (s, 6H), 2.11-2.24 (m, 4H), 2.47 (s, 4H), 3.75 (s, 3H), 4.62 (s, 1H), 6.69 (dd, *J* = 8.3 Hz, 1H), 6.78 (d, *J* = 4.9 Hz, 1H), 6.83 (d, *J* = 4.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 29.1, 29.8, 31.3, 32.6, 41.2, 50.5, 114.7, 117.2, 129.8, 135.9, 155.6, 163.5, 196.9. Anal. calcd. found (%) for $C_{24}H_{28}O_5$: C 75.82 (75.76), H 7.45 (7.52).

3,3,6,6-Tetramethyl-9-(3,4-dimethoxyphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 9): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 0.98 (s, 6H), 1.08 (s, 6H), 2.12 (d, *J* = 9.6 Hz, 2H), 2.29 (d, *J* = 9.6 Hz, 2H), 2.52 (d, *J* = 10.5 Hz, 2H), 2.59 (d, *J* = 10.4 Hz, 2H), 3.71 (s, 6H), 4.47 (s, 1H), 6.68 (dd, *J* = 8.3, 1.3 Hz, 1H), 6.75 (d, *J* = 4.9 Hz, 1H), 6.81 (d, *J* = 4.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.3, 29.7, 31.5, 33.1, 51.4, 56.7, 57.3, 114.1, 115.2, 115.9, 121.3, 138.6, 147.5, 148.9, 164.3, 197.3. Anal. calcd. found (%) for $C_{25}H_{30}O_5$: C 73.15 (72.84), H 7.37 (7.45).

3,3,6,6-Tetramethyl-9-(4-N,N-dimethylphenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 10): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 0.98 (s, 6H), 1.08 (s, 6H), 2.08 (d, *J* = 9.6 Hz, 2H), 2.28 (d, *J* = 9.6 Hz, 2H), 2.52 (s, 4H), 2.89 (s, 6H), 4.51 (s, 1H), 7.13 (d, *J* = 4.9 Hz, 2H), 7.25 (d, *J* = 4.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.5, 29.7, 31.8, 32.5, 41.3, 51.2, 114.8, 121.2, 129.7, 131.4, 143.8, 163.5, 196.7. Anal. calcd. found (%) for $C_{23}H_{31}NO_3$: C 76.30 (76.28), H 7.94 (7.89), N 3.58 (3.68).

3,3,6,6-Tetramethyl-9-(3-chlorophenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 11): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 1.01 (s, 6H), 1.13 (s, 6H), 2.21 (s, 4H), 2.53 (t, *J* = 8.6 Hz, 4H), 4.76 (s, 1H), 7.11 (d, *J* = 4.5 Hz, 1H), 7.17 (t, *J* = 4.6 Hz, 2H), 7.27 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.6, 29.7, 31.8, 32.9, 41.3, 50.8, 115.7, 126.4, 127.7, 129.8, 130.5, 134.9, 147.3, 164.2, 196.7. Anal. calcd. found (%) for $C_{23}H_{25}O_3Cl$: C 71.77 (71.63), H 6.55 (6.48).

3,3,6,6-Tetramethyl-9-(4-cyanophenyl)-1,8-dioxooctahydroxanthene (Table 3, entry 12): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 1.03 (s, 6H), 1.14 (s, 6H), 2.18 (d, *J* = 9.8 Hz, 2H), 2.28 (d, *J* = 9.8 Hz, 2H), 2.53 (t, *J* = 9.1 Hz, 4H), 4.81 (s, 1H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.54 (2H, *J* = 4.8 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.5, 29.3, 32.5, 32.7, 41.3, 51.3, 111.3, 115.6, 118.7, 128.9, 132.6, 150.3, 163.2, 197.2. Anal. calcd. found (%) for $C_{24}H_{25}NO_3$: C 76.77 (76.83), H 6.71 (6.68), N 3.73 (3.69).

3,3,6,6-Tetramethyl-9-(4-bromophenyl)-1,8-dioxooctahydroxanthene (Table-1, entry 13): ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 0.98 (s, 6H), 1.08 (s, 6H), 2.08 (d, *J* = 9.6 Hz, 2H), 2.28 (d, *J* = 9.7 Hz, 2H), 2.52 (s, 4H), 4.51 (s, 1H), 7.14 (d, *J* = 4.9 Hz, 2H), 7.43 (d, *J* = 4.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 27.5, 29.7, 31.8, 32.5, 41.3, 51.2, 114.8, 121.2, 129.7, 131.4, 143.8, 163.5, 196.7. Anal. calcd. found (%) for $C_{23}H_{25}O_3Br$: C 64.34 (64.47), H 5.87 (5.82).

RESULTS AND DISCUSSION

Most of the catalysts employed for the synthesis of 1,8-dioxooctahydroxanthenes fall under the category of acidic substances (organic, mineral or Lewis acids). Some of them are easily available, cost-effective and conform well to 'Green Chemistry' parameters [25], while there are many which require special methods of preparation [26]. We have demonstrated earlier [27] that aqueous extract of pericarp of *Sapindus trifoliatus* fruits serves as an excellent catalyst to bring about a variety of organic transformations. Different substrates have been successfully made to react by this catalyst in aqueous medium. Reactions were carried out at room temperature with maximum atom efficiency, thereby producing minimum organic waste. In this context, it is also apparent that substrates with carbonyl function have inherent tendency to react, although reaction conditions may differ.

Temperature optimization: It is evident that the reaction takes place at a very slow rate when carried out at room temperature. Even after 5 h, only 30 % reactants were converted into products. The alternative was to carry out the reaction at elevated temperatures. An oil bath with temperature-control facility was used to provide necessary heat to attain required temperature in sustained manner. The results summarized in Table-2 indicated that the maximum yield of product was obtained when the reaction was carried out at 80 ºC and time duration 2 h.

TABLE-2
EFFECT OF TEMPERATURE ON REACTION TIME AND YIELD OF THE REACTION

Entry	Reaction temperature (°C)	Time (h)	Yield (%)
1	50	5	80
2	60	4	85
3	70	3	85
4	80	2	92
5	90	2	92

Different substituted benzaldehydes were made to react with dimedone at 80 ºC in presence of aqueous extract of pericarp of Soap nut fruit. Results are in agreement with general behaviour of differently substituted benzaldehydes. Benzaldehydes with electron-donating substituents required more time to react (Table-1, entries 2, 6, 8, 9) which is expected as electron-donating substituents render carbonyl carbon less reactive. Reverse tendency is recorded with benzaldehydes containing electron-attracting substituents.

Conclusion

It is demonstrated that 1,8-dioxooctahydroxanthenes can be synthesized in aqueous medium in good to excellent yield. The reaction could be efficiently catalyzed by aqueous extract of pericarp of *Sapindus trifoliatus* fruit.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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