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A simple synthesis of *E*-9-aryl-5-arylidene-1-oxo-1,2,3,4,5,6,7,8-octahydroxanthenes and their lower analogues from *E*,*E*-α,α′-diarylidenecycloalkanones

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Abstract. A simple and efficient synthesis of *E*-9-aryl-5-arylidene-1-oxo-1,2,3,4,5,6,7,8-octahydroxanthenes and their lower analogues has been developed by amberlyst–15 catalysed cyclocondensation of *E*,*E*-α,α′-diarylidenecyclohexanones and *E*,*E*-α,α′-diarylidenecyclopentanones, respectively, with cyclohexan-1,3-diones. The products were obtained in moderate to good yield and their structures were confirmed from analytical and spectral data.

Keywords. Cyclocondensation; amberlyst–15; *E*,*E*-α,α′-diarylidenecycloalkanones; xanthene derivatives and their lower analogues.

1. Introduction

Xanthenes derivatives belong to an important class of organic compounds possessing a wide range of biological and pharmaceutical properties such as antiviral,[1] antibacterial,[2] anti-inflammatory,[3] antinociceptive,[4] antidepressant and antimalarial[5] activities. They constitute a structural unit in a number of natural products,[6] and santalin pigments occurring in a number of plant species are major sources for xanthenes.[7] They find important applications in industries, viz., as leuco-dyes,[8] as pH sensitive fluorescent materials for the visualization of biomolecular assemblies,[9] in laser technology,[10] and in photodynamic therapy.[11] Another important application of them is in the construction of new chiral bidentate phosphine ligands having potential to be used in catalytic processes.[12] A very simple methodology for synthesizing a common type of xanthene derivatives, viz., 1,8-dioxo-octahydroxanthenes, is the acid[13] or base[14] catalysed condensation of aromatic aldehydes with cyclohexan-1,3-diones. The sequence of reactions taking place in such synthesis is Knoevenagel condensation, Michael addition and then cyclization through water elimination. α,β-Unsaturated ketones and related compounds are known to undergo cyclocondensation with 1,3-dicarbonyl compounds under acid or base catalysed conditions,[15,16] which mechanistically follow the second and third steps of the above said synthetic route to 1,8-dioxo-octahydroxanthenes. However, there is no report of any cyclocondensation reaction involving *E*,*E*-α,α′-diarylidenecycloalkanones and 1,3-diketones in the literature, and this encouraged us to undertake the present work. As a part of our recent interest in developing newer applications of the sulphonated polystyrene resin amberlyst–15 in organic synthesis,[17] we first tried the targeted reaction in refluxing acetonitrile using this heterogeneous catalyst. Applicability of several other catalytic conditions was also investigated in this connection. Among all these the first mentioned catalytic condition was found to be most effective in producing xanthene derivatives and their lower analogues. Our work on the synthesis of these hitherto unknown compounds is presented herein.

2. Experimental

All melting points were recorded on a Köfler block and are uncorrected. IR spectra were recorded on a Perkin–Elmer FT–IR spectrophotometer (Spectrum BX II) in KBr pellets. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker AV–300 and Bruker–400 Avance NMR spectrometers. Analytical samples were routinely dried *in vacuo* at room temperature. Mass spectra were measured with Jeol the M Station JMS.700 (FAB–MS) and MicroMass ESI–TOF (HRMS) instruments. Microanalytical data were recorded on three Perkin–Elmer 2400 Series II C, H, N analyzers. Column chromatography was performed with silica gel (100–200

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mesh) and TLC with silica gel G made of SRL Pvt. Ltd. Petroleum ether had the boiling range 60–80°C. Amberlyst–15 used was made of Fluka Chemika. *E*,*E*-α,α′-diarylidenecycloalkanones were synthesized either by a previous method[18] or by a recent amberlyst–15 catalysed microwave assisted method developed by us,[17a] and they were properly characterized.

2.1 *General procedure for condensation of E,E-α,α′-diarylidenecycloalkanones with cyclic 1,3-dicarbonyl compounds*

To a solution of a mixture of an *E*,*E*-α,α′-diarylidenecycloalkanone (**1/2/3**) (1 mmol) and a 1,3-cyclohexadione (1 mmol) in anhy. acetonitrile (25 mL), amberlyst–15 (100 mg) was added and the mixture was refluxed for the time period mentioned in table 1. The catalyst was then removed by filtration and the filtrate was concentrated. The concentrate was subjected to chromatography over silica gel using mixtures of petroleum ether and ethyl acetate of increasing polarity as eluents. The pure product thus obtained was crystallized from chloroform–petroleum ether. All the products were obtained as colourless needles. They were characterized from their spectral data which are given below:

2.1a *Compound* ***5a***: IR (KBr): 1661 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 and 1.12 (each s, 3H, >CMe_2), 1.56–1.64 (m, 2H, H_2–7), 2.05 (t, 2H, J = 5.8 Hz, H_2–6), 2.16 (d, 1H, J = 16.5 Hz, H_A–4), 2.25 (d, 1H, J = 16.5 Hz, H_B–4), 2.52–2.62 (m, 1H, H_A–8), 2.54 (s, 2H, H_2–2), 2.67–2.77 (m, 1H, H_B–8), 4.20 (s, 1H, H–9), 6.95 (1H, br. s, Ar–CH=), 7.16–7.35 (10H, m, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.40, 27.07, 27.36, 27.62, 29.27, 32.08, 40.27, 41.30, 50.79, 112.54, 118.15, 122.01, 126.45, 126.50, 128.07, 128.18, 128.26, 129.19, 130.22, 137.36, 141.81, 144.09, 163.93, 197.09 (C=O); FABMS: 397.31 $[M+H]^+$; Anal. Calcd for $C_{28}H_{28}O_2$: C, 84.81; H, 7.12. Found: C, 84.56; H, 7.35%.

2.1b *Compound* ***5b***: IR (KBr): 1660 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 and 1.11 (each s, 3H, >CMe_2), 1.56–1.64 (m, 2H, H_2–7), 2.04 (t, 2H, J = 5.9 Hz, H_2–6), 2.16 (d, 1H, J = 16.4 Hz, H_A–4), 2.24 (d, 1H, J = 16.4 Hz, H_B–4), 2.28 and 2.35 (each s, 3H, 2 × Ar–Me), 2.50–2.60 (m, 1H, H_A–8), 2.52 (s, 2H, H_2–2), 2.67–2.77 (m, 1H, H_B–8), 4.16 (s, 1H, H–9), 6.91(s, 1H, Ar–CH=), 7.05–7.26 (m, 8H, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.06, 21.18, 22.49, 27.19, 27.49, 27.66, 29.33, 32.15, 39.92, 41.39, 50.90, 112.71, 117.10, 121.87, 128.20, 128.58, 128.86, 128.96, 129.20, 129.71, 134.57, 135.96, 136.32, 141.26, 163.90, 197.00 (C=O); Anal. Calcd for $C_{30}H_{32}O_2$: C, 84.87; H, 7.60. Found: C, 84.65; H, 7.45%.

2.1c *Compound* ***5c***: IR (KBr): 1661 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.01 and 1.12 (each s, 3H, >CMe_2), 1.56–1.65 (m, 2H, H_2–7), 2.01 (br. s, 2H, H_2–6), 2.16 (d, 1H,J = 16.5 Hz, H_A–4), 2.25 (d, 1H, J = 16.5 Hz, H_B–4), 2.49–2.59 (m, 1H, H_A–8), 2.52 (s, 2H, H_2–2), 2.63-2.73 (m, 1H, H_B–8), 4.18 (1H, s, H–9), 6.88 (1H, br. s, Ar–CH=), 7.23–7.33 (m, 8H, Ar–H); Anal. Calcd for $C_{28}H_{26}Cl_2O_2$: C, 72.26; H, 5.63. Found: C, 71.98; H, 5.77%.

2.1d *Compound* ***5d***: IR (KBr): 1662 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.02 and 1.09 (each s, 3H, >CMe_2), 1.56–1.64 (m, 2H, H_2–7), 2.04 (t, 2H, J = 5.7 Hz, H_2–6), 2.16 (d, 1H, J = 16.3 Hz, H_A–4), 2.24 (d, 1H, J = 16.3 Hz, H_B–4), 2.52 (s, 2H, H_2–2), 2.52–2.62 (m,1H, H_A–8), 2.68–2.78 (m, 1H, H_B–8), 3.77 and 3.83 (each s, 3H, 2 × OMe) 4.14 (s, 1H, H–9), 6.80 (d, J = 8.5 Hz, 2H, Ar–H), 6.88 (1H, s, Ar–CH=), 6.90 (d, J = 8.5 Hz, 2H, Ar–H), 7.21 (d, J = 8.5 Hz, 2H, Ar–H), 7.27 (d, J = 8.5 Hz, 2H, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.50, 27.19, 27.43, 27.60, 29.32, 32.13, 39.40, 41.37, 50.88, 55.16, 55.26, 112.79, 113.64, 117.66, 121.47, 128.84, 129.25, 130.02, 130.50, 136.51, 141.90, 158.19, 158.30, 163.84, 197.28 (C=O); Anal. Calcd for $C_{30}H_{32}O_4$: C, 78.92; H, 7.06. Found: C, 78.60; H, 7.23%.

Table 1. Optimization of cyclocondensation reaction of **2a** and dimedone.

Entry	Catalyst	Reaction condition	Yield (%)[a] of **5a**
1.	Amberlyst–15	Anhy. MeCN, reflux, 16 h (condition–I)	52
2.	Amberlyst–15	Amberlyst-15, MW (540 w), 5 min[b]	5
3.	HOAc	HOAc, reflux, 16 h	12
4.	Et_3N	EtOH, reflux, 16 h	0[c]
5.	Et_3N	No solvent, 100°C, 10 h	0[c]

[a]Yield refers to isolated product
[b]Beyond this time the reaction mixture charred
[c]Here a product other than **5a** was formed, which could not be characterized so far

2.1e *Compound* ***5e***: IR (KBr): 1661 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.04 and 1.12 (each s, 3H, >CMe_2), 1.56–1.64 (m, 2H, H_2–7), 2.04 (2H, t, J = 5.7 Hz, H_2–6), 2.18 (d, 1H, J = 16.5 Hz, H_A–4), 2.25 (d, 1H, J = 16.5 Hz, H_B–4), 2.52 (s, 2H, H_2–2), 2.52–2.62 (m, 1H, H_A–8), 2.67–2.77 (m, 1H, H_B–8), 4.11 (s, 1H, H–9), 5.90 and 5.97 (each s, 2H, 2 x -O-CH_2-O-), 6.76 (s, 1H, Ar–CH=), 6.71–6.84 (m, 6H, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.39, 27.15, 27.49, 29.20, 32.08, 39.87, 41.28, 50.82, 100.76, 100.94, 107.88, 108.09, 108.60, 109.31, 112.52, 117.71, 121.48, 121.74, 123.12, 129.15, 131.43, 138.27, 141.75, 146.07, 146.17, 147.40, 147.55, 163.85, 197.20 (C=O); Anal. Calcd for $C_{30}H_{28}O_6$: C, 74.36; H, 5.82. Found: C, 74.45; H, 5.89%.

2.1f *Compound* ***5f***: IR (KBr): 1660 cm^{-1} (C=O); ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.86–1.97 (m, 2H), 2.22–2.35 (m, 4H), 2.58–2.69 (m, 2H), 2.83–3.04 (m, 4H), 4.53 (s, 1H, H–9), 7.26 (s, 1H, Ar–CH=), 7.40-7.55 (10H, m, Ar–H), Anal. Calcd for $C_{26}H_{24}O_2$: C, 84.75; H, 6.57. Found: C, 84.64; H, 6.46%.

2.1g *Compound* ***5g***: IR (KBr): 1662 cm^{-1} (C=O); ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.95–2.15 (m, 4H), 2.33 and 2.40 (each s, 3H, 2 × Ar–CH_3), 2.33–2.73 (m, 8H), 4.23 (s, 1H, H–9), 6.96 (br. s, 1H, Ar–CH=), 7.09–7.30 (m, 8H, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.41, 21.04, 21.16, 22.45, 27.13, 27.64, 36.99, 39.73, 114.02, 117.93, 121.79, 128.20, 128.81, 128.93, 129.15, 129.57, 134.46, 135.97, 136.28, 141.27, 141.87, 165.61, 197.39 (C=O); Anal. Calcd for $C_{28}H_{28}O_2$: C, 84.81; H, 7.12. Found: C, 84.63; H, 7.20%.

2.1h *Compound* ***5h***: IR (KBr): 1661 cm^{-1} (C=O); ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.95–2.13 (m, 4H), 2.35–2.80 (m, 8H), 4.25 (s, 1H, H–9), 6.93 (br. s, 1H, Ar–CH=), 7.25–7.38 (m, 8H, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.40, 22.31, 27.04, 27.59, 36.93, 39.67, 113.57, 118.04, 121.13, 128.32, 128.43, 129.70, 130.48, 130.53, 132.27, 132.35, 135.67, 141.93, 142.60, 165.77, 197.35 (C=O); Anal. Calcd for $C_{26}H_{22}Cl_2O_2$: C, 71.40; H, 5.07. Found: C, 71.28; H, 4.96%.

2.1i *Compound* ***4a***: IR (KBr): 1661 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09 and 1.13 (each 3H, s, >CMe_2), 2.18–2.40 (m, 4H), 2.56 (d, 1H, J = 16.6 Hz, H_A–2), 2.63 (d, 1H, J = 16.6 Hz, H_B–2), 2.76-2.96 (m, 2H), 4.47(s, 1H, H–8), 6.49 (s, 1H, Ar–CH=), 7.16–7.41(m, 10H, Ar–H). TOFMSES$^+$ 405.1832 [Cald. for $(M+Na)^+$: 405.1830]. Anal. Calcd for $C_{27}H_{26}O_2$: C, 84.78; H, 6.85. Found: C, 84.55; H, 6.61%.

2.1j *Compound* ***4b***: IR (KBr): 1658 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.08 and 1.13 (each s, 3H, >CMe_2), 2.18–2.37 (m, 4H), 2.28 (s, 3H, Ar–CH_3), 2.34 (s, 3H, Ar–CH_3), 2.52 (br. s, 2H, H_2–2), 2.74-2.94 (m, 2H), 4.42 (s, 1H, H–8), 6.45 (s, 1H, Ar–CH=), 7.05-7.16 (m, 6H, Ar–H), 7.29 (d, 2H, J = 7.6 Hz, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 21.05, 21.18, 27.02, 27.75, 28.10, 28.93, 32.14, 38.07, 41.65, 51.03, 112.78, 116.36, 124.77, 127.91, 128.02, 129.00, 129.18, 134.87, 135.97, 136.79, 140.45, 146.65, 164.88, 197.57(C=O); Anal. Calcd for $C_{29}H_{30}O_2$: C, 84.84; H, 7.37. Found: C, 84.59; H, 7.30%.

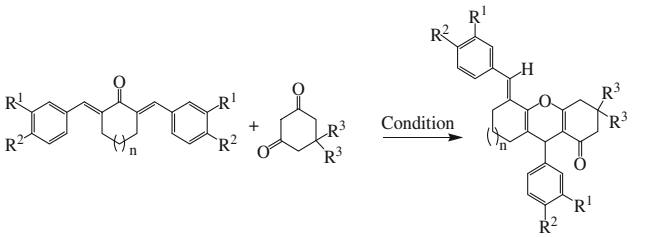
2.1k *Compound* ***4c***: IR (KBr): 1660 cm^{-1} (C=O); ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.07 and 1.13 (each s, 3H, >CMe_2), 2.17–2.42 (m, 4H), 2.57 (br. s, 2H, H_2–2), 2.69–2.89 (m, 2H), 4.44 (s,1H, H–8), 6.44 (s, 1H, Ar–CH=), 7.16-7.54 (m, 8H, Ar–H). Anal. Calcd for $C_{27}H_{24}Cl_2O_2$: C, 71.84; H, 5.36. Found: C, 71.56; H, 5.50%.

2.1l *Compound* ***4d***: IR (KBr): 1658 cm^{-1} (C=O); ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.00–2.11 (m, 2H), 2.30 (s, 3H, Ar–CH_3), 2.36 (s, 3H, Ar–CH_3), 2.20–2.50 (m, 4H), 2.62–2.76 (m, 2H), 2.80–2.93 (m, 2H), 4.47 (s, 1H, H–8), 6.48 (s, 1H, Ar–CH=), 7.07–7.18 (m, 6H, Ar–H), 7.31 (d, 2H, J = 8.2 Hz, Ar–H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.51, 21.04, 21.16, 27.04, 27.99, 28.13, 37.22, 38.00, 114.10, 116.37, 124.72, 127.97, 128.03, 128.99, 129.17, 134.89, 135.98, 136.75, 140.45, 146.70, 166.56, 197.66 (C=O); TOFMSES$^+$ 405 [Cald. for $(M+Na)^+$: 405]; Anal. Calcd for $C_{27}H_{26}O_2$: C, 84.78; H, 6.85. Found: C, 85.03; H, 6.72.

2.1m *Compound* ***4e***: IR (KBr): 1659 cm^{-1} (C=O); ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.00–2.10 (m, 2H), 2.20–2.45 (m, 4H), 2.64–2.76 (m, 2H), 2.80–2.90 (m, 2H), 4.46 (s, 1H, H–8), 6.45 (s, 1H, Ar–CH=), 7.15–7.30 (m, 8H, Ar–H); Anal. Calcd for $C_{25}H_{20}Cl_2O_2$: C, 70.93; H, 4.76. Found: C, 80.06; H, 4.62%.

3. Results and discussion

Our endeavour to carry out cyclocondensation of *E*,*E*-α,α'-diarylidenecycloalkanones with cyclohexan–1,3-diones (scheme 1) started with the amberlyst–15 catalysed reaction of *E*, *E*-2,6-dibenzylidenecyclohexanone (**2a**) with dimedone. This reaction done in anhy. acetonitrile under reflux condition (condition–I, table 1)



1: n=0; **2**: n=1; **3**: n=2

4: n=0; **5**: n=1; **6**: n=2

a: R^1=R^2=H; **b**: R^1=H, R^2=Me; **c**: R^1=H, R^2= Cl;
d: R^1=H, R^2=OMe; **e**: R^1, R^2= -OCH_2O-

Scheme 1. Cyclocondensation of *E*,*E*-*α*,*α*′-diarylidenecycloalkanones with cyclohexan-1,3-diones.

was found to give the targeted cyclocondensation product **5a** in moderate yield in 16 h. The reaction of the same reactants was then studied under several other conditions shown in table 1, but none of them was found to be effective for synthesis of **5a**. We then investigated the outcome of the reaction of four other *E*, *E*-2,6-diarylidenecyclohexanones (**2b–e**) and five *E*, *E*-2,5-diarylidenecyclopentanones (**1a–e**) with dimedone and that of some of the compounds of the series **1** and **2** with cyclohexan–1,3-diones under condition–I. It was observed that the yield of **5** from substrates of the series **2** were in general good when the latter contained an electron withdrawing or a weakly electron donating substituent but those were only moderate with substrates having an electron donating substituent (table 2).

Table 2. Amberlyst–15 catalysed synthesis of *E*-9-aryl-5-arylidene-1-oxo-1,2,3,4,5,6,7,8-octahydroxanthenes (**5**) and their lower analogues (**4**).

Entry	Reactants	Product	Time (h)	Yield (%)[a]	m.p. (°C)
1.	**2a**	**5a**	12	52	167–168
2.	**2b**	**5b**	10	61	166–168
3.	**2c**	**5c**	12	51	157–159
4.	**2d**	**5d**	16	36	158–159
5.	**2e**	**5e**	16	34	169–170

Table 2. (continued).

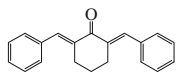
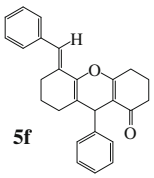
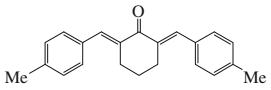
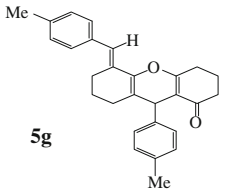
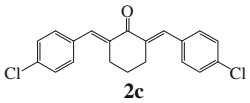
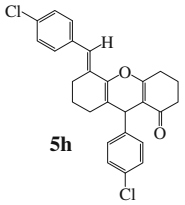
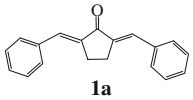
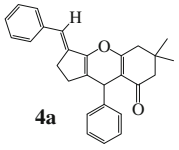
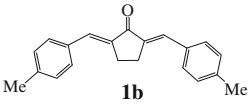
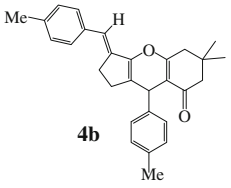
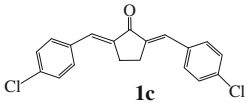
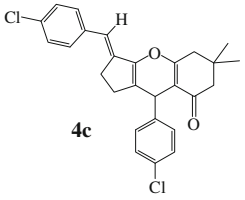
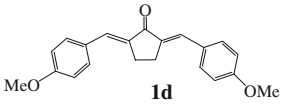
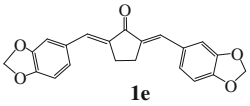
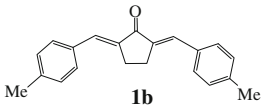
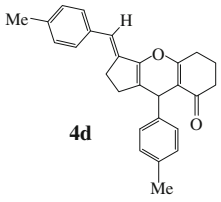
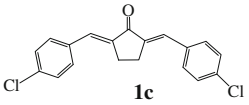
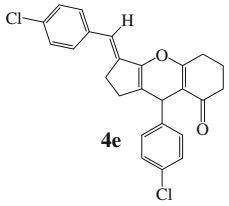
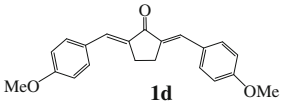
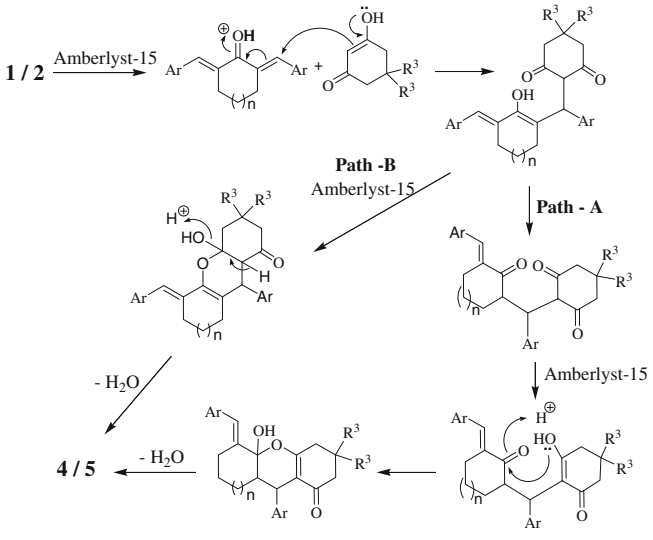
Entry	Reactants		Product	Time (h)	Yield (%)[a]	m.p. (°C)
6.	 **2a**		 **5f**	15	51	167–170
7.	 **2b**		 **5g**	15	68	163–165
8.	 **2c**		 **5h**	15	70	212–214
9.	 **1a**		 **4a**	15	59	199–201
10.	 **1b**		 **4b**	15	51	228–230
11.	 **1c**		 **4c**	16	43	232–234
12.	 **1d**		No reaction	16	–	–
13.	 **1e**		No reaction	16	–	–

Table 2. (continued).

Entry	Reactants	Product	Time (h)	Yield (%)[a]	m.p. (°C)
14.	 **1b** 	 **4d**	15	52	228–230
15.	 **1c** 	 **4e**	15	45	229–231
16.	 **1d** 	No reaction	16	–	–

[a]Yields refer to isolated products

Analogously, with the substrates of the series **1**, the yield of cyclocondensation product **4** was found to be very much dependent on the substituent in the arylidene moiety (table 2). With a view to getting more analogues of **4** and **5**, we then attempted the above cyclocondensation reaction (under conditions–I) by variation of reactants in the following ways: (i) reaction of each of *E*, *E*-2,7-dibenzylidenecycloheptanone (**3**) and *E*, *E*-dibenzylideneacetone with each of dimedone and cyclohexane–1,3-dione and (ii) reaction of each of **1** and **2** with the acyclic 1,3-diketone acetylacetone. But, in none of these cases any significant extent of reaction was found to occur. Plausible mechanistic paths for the formation of **4** and **5** from **1** and **2**, respectively, have been delineated in scheme 2. It was evident from the structures of the cyclocondensation products that one of the enone double bonds of the substrates did not undergo any reaction. So the exocyclic double bond of each of **4** and **5** has been assigned*E*-configuration (as that is in **1** and **2**).



Scheme 2. Plausible mechanism for formation of **4**/**5**.

4. Conclusion

A series of *E*-9-aryl-5-arylidene-1-oxo-1,2,3,4,5,6,7,8-octahydroxanthenes (**5**) and their lower analogues (**4**) have been synthesized in moderate yield by using a simple methodology. All the synthesized compounds are hitherto unknown compounds and might have interesting biological activities.

Supplementary information

The electronic supplementary material contains ^{1}H NMR, ^{13}C NMR and mass spectra of a number of compounds of the series **4** and **5**, which can be seen in www.ias.ac.in/chemsci.

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