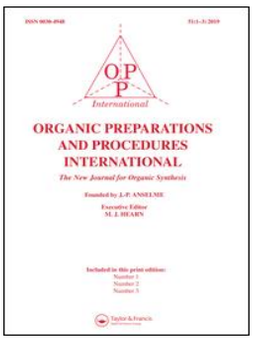

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The New Journal for Organic Synthesis

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/uopp20

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To cite this article: Farzaneh Mohamadpour (2020): Glycine as a Green Catalyst for the Preparation of Xanthenes, Organic Preparations and Procedures International, DOI: 10.1080/00304948.2020.1834343

To link to this article: https://doi.org/10.1080/00304948.2020.1834343

Published online: 29 Dec 2020.

ORGANIC PREPARATIONS AND PROCEDURES INTERNATIONAL
https://doi.org/10.1080/00304948.2020.1834343

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EXPERIMENTAL PAPER

Glycine as a Green Catalyst for the Preparation of Xanthenes

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ARTICLE HISTORY Received 7 March 2020; Accepted 18 July 2020

Xanthenes and their analogues have been receiving attention owing to their biological activities. These include antiviral,[1] anti-inflammatory[2] and antiplasmodial properties.[3] These heterocyclic molecules have also been widely used as pH sensitive fluorescent materials for visualization of luminescent dyes,[4–5] laser technology,[6–7] biomolecules[8–9] and sensitizers in photodynamic therapy.[10–11] Some of the more well-known frameworks with biological properties are shown in Figure 1.

To date, methods for the synthesis of xanthenes have been reported using multicomponent reactions (MCRs) in the presence of catalysts. Among the catalysts are ceric ammonium nitrate,[12] [Hbim]BF_4,[13] strontium triflate,[14] sulfonic acid functionalized LUS-1,[15] Fe_3O_4@SiO_2–SO_3H,[16] nano-alumina sulfuric acid,[17] zinc oxide nanoparticles,[18] $NaHSO_4$·SiO_2,[19] trityl chloride,[20] Fe_3O_4 nanoparticles,[21] sulfonic acid-functionalized phthalimide,[22] sulfamic acid,[23] boron sulphonic acid,[24] trichloromelamine,[25] [cmmim][BF_4],[26] NO_2–Fe(III)Pc/C,[27] diatomite-SO_3H,[28] phosphosulfonic acid,[29] silica sulfuric acid,[30] [H-NMP][HSO_4],[31] $Mg(BF_4)_2$ doped in [BMIm][BF_4],[32] [(n-propyl)$_2NH_2$][HSO_4],[33] SiO_2-Pr-SO_3H,[34] AHS@MMT,[35] wet cyanuric chloride,[36] and 1,3-disulfonic acid imidazolium hydrogen sulfate.[37] Notwithstanding the value of specific applications, some of these methods have limitations. Chief among these are long time reactions, low yields, the use of strongly acidic conditions, difficult work-up, and toxic and expensive catalysts. To address some of these concerns, we have recently reported on the use of glycine as a natural, bio-based and biodegradable catalyst.[38] We now report on the use of glycine for the MCR preparation of xanthenes.

In a preliminary experiment, we carried out the three-component condensation of β-naphthol (1.0 mmol), benzaldehyde (1.0 mmol) and dimedone (1.0 mmol) in the presence of glycine (10 mol%) under solvent-free conditions at 80 °C. The desired product **4h** was obtained in 89% yield. Encouraged by this result, we chose this reaction as a model to optimize conditions for the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones (**4a-p**). Only a trace of product could be detected in the absence of the catalyst even after 120 min (Table 1, entry 1). After exploring several catalyst loadings, we found that 10 mol% of glycine is optimal (Table 1, entry 3). The use of more catalyst did not alter either reaction time or yield of the product (Table 1, entry 10). We also investigated several different temperatures for the model reaction (Table 1). We were satisfied

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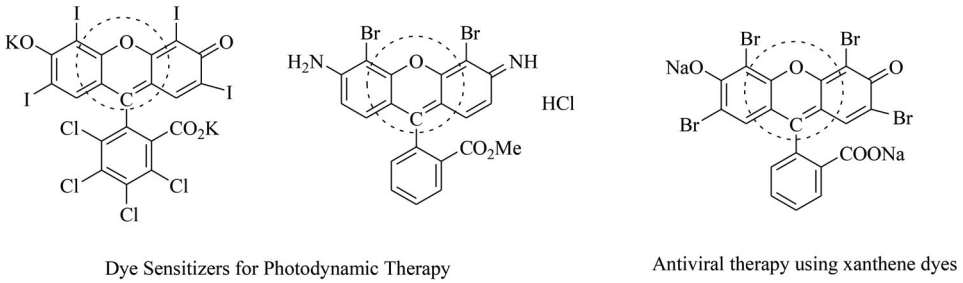
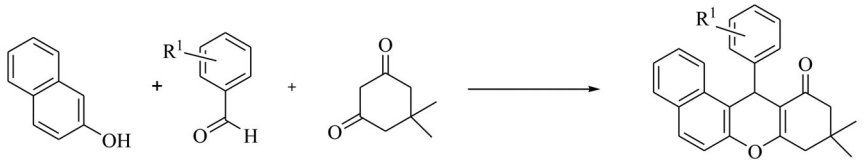


Figure 1. Pharmaceutically active compounds with xanthene units.

Table 1. Optimization of the reaction condition for the synthesis of **4h**.[a]



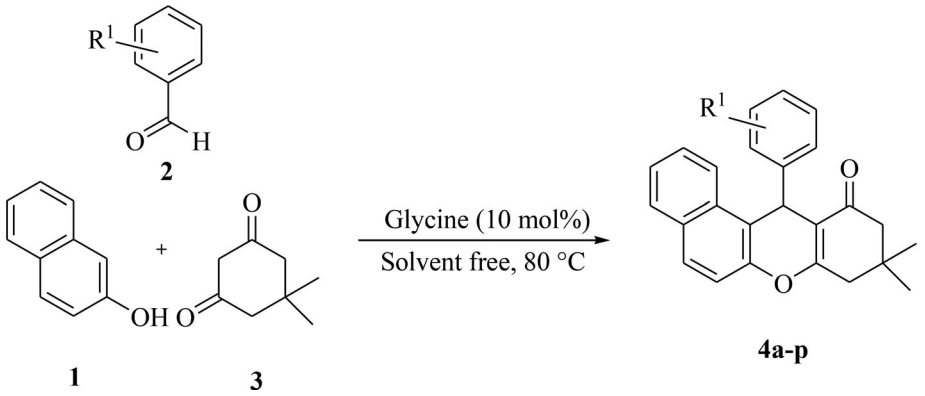
Entry	Glycine (mol %)	Temperature (°C)	Time (min)	ISOLATED YIELDS (%)
1	Catalyst free	80	120	TRACE
2	5	80	25	67
3	**10**	**80**	**15**	**89**
4	10	rt	120	TRACE
5	10	40	60	16
6	10	50	45	38
7	10	60	25	53
8	10	70	15	68
9	10	90	15	89
10	15	80	15	91

[a]Reaction conditions: *β*-naphthol (1.0 mmol); benzaldehyde (1.0 mmol); dimedone (1.0 mmol) and glycine were heated at specified temperatures for the appropriate time.

to find that the reaction proceeded smoothly, and almost complete conversion of reactants was observed at 80 °C to afford the desired product (**4h**) in 89% yield within 15 min (Table 1, entry 3).

Having optimized reaction conditions, we then synthesized a series 12-aryl-tetrahydrobenzo[α]xanthene-11-ones via *β*-naphthol (**1**, 1.0 mmol), aldehyde derivatives (**2**, 1.0 mmol) and dimedone (**3**, 1.0 mmol) (**4a-p**) using 10 mol% glycine as the catalyst under solvent-free conditions at 80 °C (Scheme 1). The results are summarized in Table 2.

We then turned our attention toward the synthesis of 1,8-dioxo-octahydroxanthene derivatives and, herein, initially, we chose benzaldehyde (1.0 mmol) and dimedone (2 mmol) as the standard substrates to search for suitable reaction conditions. When a systematic screening was made, we found that, at 70 °C, the substrates were transformed into the desired product **5e** in an excellent yield (Table 3, entry 3). With these optimized conditions in hand, we examined the scope of this process capitalizing on the ready availability of the inexpensive starting materials. As revealed in Table 4, a range of useful 1,8-dioxo-octahydroxanthene derivatives can be synthesized in high yields (Scheme 2).

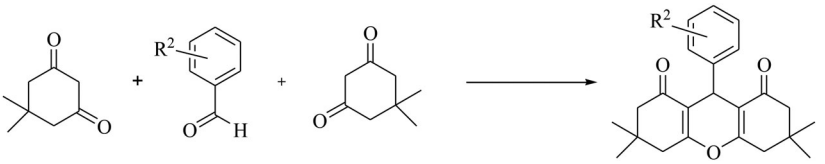


Scheme 1. Synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones.

Table 2. Glycine catalyzed synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones.

Entry	R^1	Product	Time (min)	Isolated Yields (%)	M.p. °C	M.p. °C
1	3-Cl-C_6H_4	**4a**	20	83	170-172	172-173[21]
2	4-Me-C_6H_4	**4b**	15	86	173-174	171-173[37]
3	4-OH-C_6H_4	**4c**	20	79	221-223	222-223[37]
4	3-O_2N-C_6H_4	**4d**	10	88	167-169	167-169[16]
5	4-OMe-C_6H_4	**4e**	20	82	203-205	202-204[37]
6	3-F-C_6H_4	**4f**	10	91	225-227	223-228[21]
7	4-Cl-C_6H_4	**4g**	25	77	178-180	176-178[37]
8	C_6H_5	**4h**	15	89	149-151	148-150[37]
9	3-Me-C_6H_4	**4i**	10	88	176-178	178-180[23]
10	3-Br-C_6H_4	**4j**	20	81	163-165	161-164[20]
11	3,4-$(OMe)_2$-C_6H_3	**4k**	25	84	195-197	196-198[21]
12	4-F-C_6H_4	**4l**	10	89	183-185	184-185[16]
13	4-Br-C_6H_4	**4m**	20	78	186-188	184-186[37]
14	2-Cl-C_6H_4	**4n**	20	85	180-181	179-180[30]
15	2-OMe-C_6H_4	**4o**	15	87	166-168	165-167[33]
16	4-O_2N-C_6H_4	**4p**	10	83	174-176	175-178[20]

Table 3. Optimization of the reaction condition for the synthesis of **5e**.[a]

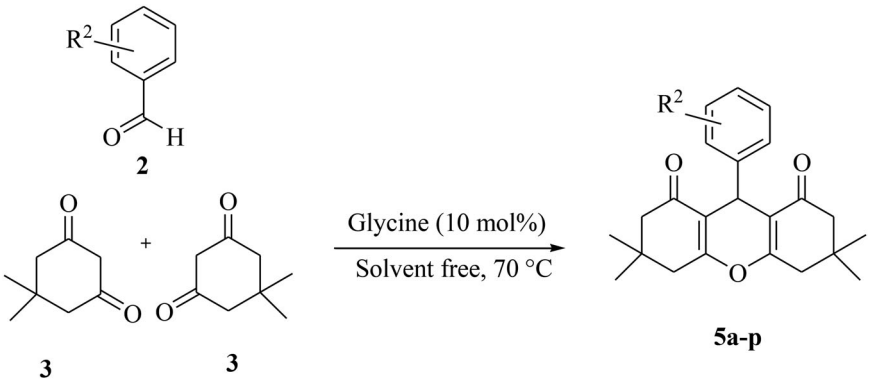


Entry	Glycine (mol %)	Temperature (°C)	Time (min)	Isolated Yields (%)
1	Catalyst free	70	120	trace
2	5	70	20	73
3	**10**	**70**	**10**	**91**
4	10	rt	120	trace
5	10	40	50	27
6	10	50	25	46
7	10	60	15	65
8	10	80	10	91
9	15	70	10	90

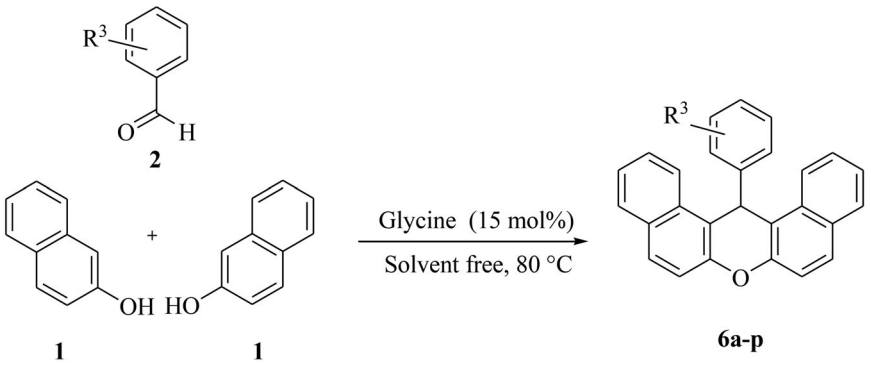
[a]Reaction conditions: benzaldehyde (1.0 mmol), dimedone (2.0 mmol) and glycine were heated at various temperatures for the appropriate time.

Table 4. Glycine catalyzed synthesis of 1,8-dioxo-octahydroxanthenes.

Entry	R^2	Product	Time (min)	Isolated Yields (%)	M.p. °C	M.p. °C
1	3-Br-C_6H_4	**5a**	20	82	193-195	192-194[26]
2	3-OMe-C_6H_4	**5b**	15	88	168-170	166-168[15]
3	4-F-C_6H_4	**5c**	10	92	194-196	193-195[37]
4	4-Br-C_6H_4	**5d**	20	78	238-240	239-241[16]
5	C_6H_5	**5e**	10	91	204-206	206-208[37]
6	2-Cl-C_6H_4	**5f**	10	85	225-227	224-227[16]
7	4-OH-C_6H_4	**5g**	25	76	244-246	246-248[26]
8	2-O_2N-C_6H_4	**5h**	10	89	242-244	240-242[25]
9	4-Me-C_6H_4	**5i**	10	90	216-218	216-218[26]
10	4-Cl-C_6H_4	**5j**	20	81	237-239	235-238[16]
11	4-OMe-C_6H_4	**5k**	20	86	240-242	241-243[16]
12	4-O_2N-C_6H_4	**5l**	10	87	220-222	222-224[26]
13	2-OH-C_6H_4	**5m**	20	80	201-203	202-204[24]
14	3,4-$(OMe)_2$-C_6H_3	**5n**	20	83	175-177	174-176[26]
15	3-O_2N-C_6H_4	**5o**	15	86	171-173	171-172[16]
16	2,4-Cl_2-C_6H_3	**5p**	20	79	246-248	248-250[15]



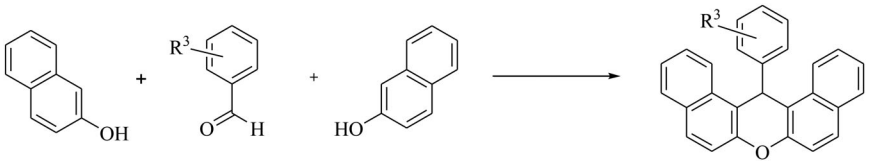
Scheme 2. Synthesis of 1,8-dioxo-octahydroxanthenes.



Scheme 3. Synthesis of 14-aryl-14*H*-dibenzo[*α,j*]xanthenes.

In continuation of our work on the preparation of xanthenes, we explored the use of glycine for the synthesis of 14-aryl-14*H*-dibenzo[*α,j*]xanthenes in a one-pot three-component domino reaction of *β*-naphthol (**1**, 2.0 mmol) and aromatic aldehydes (**2**, 1.0 mmol) (Scheme 3) . We chose *β*-naphthol (2.0 mmol) and benzaldehyde (1.0 mmol)

Table 5. Optimization of the reaction condition for the synthesis of **6c**.[a]



Entry	Glycine (mol %)	Temperature (°C)	Time (min)	Isolated Yields (%)
1	Catalyst free	80	120	trace
2	5	80	45	37
3	10	80	25	61
4	**15**	**80**	**15**	**88**
5	15	rt	120	trace
6	15	40	70	21
7	15	50	55	38
8	15	60	45	53
9	15	70	25	76
10	15	90	15	89
11	20	80	15	90

[a]Reaction conditions: β-naphthol (2.0 mmol), benzaldehyde (1.0 mmol) and glycine were heated at specified temperatures for the appropriate time.

Table 6. Glycine catalyzed synthesis of 14-aryl-14*H*-dibenzo[α,*j*]xanthenes.

Entry	R^3	Product	Time (min)	Isolated Yields (%)	M.p. °C	M.p. °C
1	4-Br-C_6H_4	**6a**	25	78	295-297	297-298[37]
2	4-OMe-C_6H_4	**6b**	15	86	202-204	204-205[37]
3	C_6H_5	**6c**	15	88	182-184	183-184[28]
4	4-Me-C_6H_4	**6d**	15	85	228-230	227-228[37]
5	3-O_2N-C_6H_4	**6e**	10	90	213-215	212-213[31]
6	2-Cl-C_6H_4	**6f**	20	82	212-214	212-213[28]
7	3-Br-C_6H_4	**6g**	25	79	193-195	191-193[16]
8	2-O_2N-C_6H_4	**6h**	10	88	288-290	290-291[29]
9	4-Cl-C_6H_4	**6i**	25	83	290-292	289-290[28]
10	3-Me-C_6H_4	**6j**	15	87	195-197	197-198[31]
11	2-OMe-C_6H_4	**6k**	10	89	257-259	258-259[36]
12	2,4-Cl_2-C_6H_3	**6l**	25	76	256-258	255-256[29]
13	4-O_2N-C_6H_4	**6m**	15	84	307-309	308-309[31]
14	4-F-C_6H_4	**6n**	10	87	241-243	240-242[18]
15	3-OMe-C_6H_4	**6o**	10	85	168-170	170-172[35]
16	4-OH-C_6H_4	**6p**	25	74	139-141	138-140[31]

as the model substrates. To obtain the optimized reaction conditions, we changed temperature and the amount of catalyst. The results are summarized in Table 5. We found that the reaction proceeded smoothly, and almost complete conversion of reactants was observed at 80 °C to afford the desired product (**6c**) in 88% yields within 15 min (Table 5, entry 4) with 15 mol% catalyst loading.

We ascertained the scope of the reaction, using a number of aromatic aldehydes (Table 6). We were pleased to find that all substrates were converted to the corresponding products in good to excellent yields (74-90%).

We made a comparison of the abilities of some catalysts previously reported in the literature for the synthesis of compounds **4**, **5** and **6**. The results are presented in Tables 7, 8, and 9.

Table 7. Comparison of catalytic ability with catalysts reported in the literature for synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives.[a]

Entry	Catalyst	Conditions	Time/Yield (%)[References]
1	CAN	Microwave irradiation, 120 °C	120 min/85[12]
2	$Sr(OTf)_2$	1,2-Dichloroethane, 80 °C	300 min/85[14]
3	Fe_3O_4@SiO_2-SO_3H	Solvent-free, 110 °C	30 min/95[16]
4	$NaHSO_4/SiO_2$	CH_2Cl_2, Reflux	300 min/91[19]
5	Fe_3O_4 MNPs	Solvent-free, 110 °C	60 min/94[21]
6	$B(HSO_4)_3$	Solvent-free, 120 °C	10 min/87[24]
7	NO_2—FePc/C	EtOH, Reflux	30 min/91[27]
8	DSIMHS	Solvent-free, 55 °C	20 min/93[37]
9	**Glycine**	**Solvent-free, 80 °C**	**15 min/89** [This work]

[a]Based on the three-component reaction of β-naphthol (1.0 mmol); benzaldehyde (1.0 mmol) and dimedone (1.0 mmol).

Table 8. Comparison of catalytic ability with catalysts reported in the literature for synthesis of 1,8-dioxo-octahydroxanthene derivatives.[a]

Entry	Catalyst	Conditions	Time/Yield (%)[References]
1	$[Hbim]BF_4$	Microwave irradiation	45 min/85[13]
2	Mesoporous silica LUS-1	Solvent-free, 140 °C	15 min/90[15]
3	Fe_3O_4@SiO_2-SO_3H	Solvent-free, 110 °C	4 min/94[16]
4	Trichloromelamine	Solvent-free, 110 °C	30 min/82[25]
5	$[cmmim][BF_4]$	Microwave irradiation	2 min/92[26]
6	Phospho sulfonic acid	Solvent-free, 80 °C	50 min/86[29]
7	$[BMim][BF_4]$	$Mg(BF_4)_2$, 80 °C	30 min/97[32]
8	DSIMHS	Solvent-free, 55 °C	4 min/95[37]
9	**Glycine**	**Solvent-free, 70 °C**	**10 min/91**[This work]

[a]Based on the three-component reaction of dimedone (2.0 mmol) and benzaldehyde (1.0 mmol).

Table 9. Comparison of catalytic ability with catalysts reported in the literature for synthesis of 14-aryl-14*H*-dibenzo[α,*j*]xanthenes derivatives.[a]

Entry	Catalyst	Conditions	Time/Yield (%)[References]
1	Fe_3O_4@SiO_2-SO_3H	Solvent-free, 110 °C	30 min/94[16]
2	SFP	Solvent-free, 90 °C	30 min/98[22]
3	$B(HSO_4)_3$	Solvent-free, 120 °C	10 min/91[24]
4	Diatomite-SO_3H	Solvent-free, 90 °C	10 min/93[28]
5	Phospho sulfonic acid	Solvent-free, 80 °C	15 min/84[29]
6	$[H\text{-}NMP][HSO_4]$	Solvent-free, 110 °C	12 min/94[31]
7	$[BMim][BF_4]$	$Mg(BF_4)_2$, 80 °C	15 min/95[32]
8	SiO_2-Pr-SO_3H	Solvent-free, 125 °C	20 min/98[34]
9	DSIMHS	Solvent-free, 90 °C	3 min/94[37]
10	**Glycine**	**Solvent-free, 80 °C**	**15 min/88**[This work]

[a]Based on the three-component reaction of β-naphthol (2.0 mmol) and benzaldehyde (1.0 mmol).

This study reveals that glycine has shown exceptional potential as an inexpensive and readily available catalyst for the green synthesis of the xanthenes described in this paper. The methods disclosed here allow for a convenient and solvent-free approach for the preparation of these important heterocyclic compounds. We hope that these easy procedures will permit wider access to the xanthenes and promote further investigations into their chemistry and pharmacology.

Experimental section

Melting points of all compounds were determined using an Electro thermal 9100 apparatus and are uncorrected. ^{1}H NMR spectra were recorded on a Bruker DRX-400

Avance instrument with $CDCl_3$ as solvent. All reagents and solvents were purchased from Merck, Fluka and Acros chemical companies and were used without further purification. TLC was performed on silica gel using EtOAc/n-hexane (1/3) as eluting solvent. All of the compounds were known and were identified by matching their melting points with the literature values in the references cited in the tables. For the sake of completeness, representative NMR spectra are provided below.

Synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one derivatives (4)

A mixture of β-naphthol (**1**, 1.0 mmol), aromatic aldehyde derivative (**2**, 1.0 mmol), dimedone (**3**, 1.0 mmol) and glycine (10 mol%) was heated at 80 °C for the appropriate time. After completion of the reaction (TLC) the mixture was cooled to r.t. and ethanol was added. The resulting precipitate was separated by filtration, The solid was recrystallized from ethanol to afford the pure products (**4a-p**).

Synthesis of 1,8-dioxo-octahydroxanthene derivatives (5)

A mixture of dimedone (**3**, 2.0 mmol), aromatic aldehyde derivative (**2**, 1.0 mmol), and glycine (10 mol %) was heated at 70 °C for the appropriate time. After completion of the reaction (TLC) the mixture was cooled to r.t. and ethanol was added. The resulting precipitate was separated by filtration. The solid was recrystallized from ethanol to afford the pure products (**5a-p**).

Synthesis of 14-aryl-14H-dibenzo[a,j]xanthene derivatives (6)

A mixture of β-naphthol (**1**, 2.0 mmol), aromatic aldehyde derivative (**2**, 1.0 mmol) and glycine (15 mol %) was heated at 80 °C for appropriate time. After completion of the reaction (TLC) the mixture was cooled to r.t. Ethanol was added and the resulting precipitate was separated by filtration. The solid was recrystallized from ethanol to afford the pure products (**6a-p**).

9,9-Dimethyl-12-(4-methoxyphenyl)-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one (4e)

^{1}HNMR (400 MHz, $CDCl_3$): 0.99 (3H, s, CH_3), 1.12 (3H, s, CH_3), 2.16-2.35 (2H, m, CH_2), 2.58 (2H, s, CH_2), 3.71 (3H, s,OCH_3), 5.68 (1H, s, CHAr), 6.72 (2H, d, $J = 8.4$ Hz, ArH), 7.21-7.47 (5H, m, ArH), 7.85 (2H, t, $J = 9.2$ Hz, ArH), 8.01 (1H, d, $J = 8.4$ Hz, ArH).

3,3.6,6-Tetramethyl-9-(4-methoxyphenyl)-1,2,3,4,5,6,7,8-octahydroxanthene-1,8-dione (5k)

^{1}H NMR (400 MHz, $CDCl_3$): 1.01 (6H, s, $2CH_3$), 1.12 (6H, s, $2CH_3$), 2.16-2.27 (4H, q, $J = 8.2$ Hz, $2CH_2$), 2.47 (4H, s, $2CH_2$), 3.75 (3H, s, OCH_3), 4.72 (1H, s, CH), 6.77 (2H, d, $J = 8.8$Hz, ArH), 7.22 (2H, d, $J = 8.8$ Hz, ArH).

14-(4-Methylphenyl)-7,14-dihydrodibenzo[a,j]xanthene (6d)

^{1}H NMR (400 MHz, $CDCl_3$): 2.18 (3H, s, CH_3), 6.47 (1H, s, CH), 6.81(1H, d, $J = 7.6$ Hz, ArH), 6.81(1H, t, $J = 7.6$ Hz, ArH), 7.40-7.85 (12H, m, ArH), 8.42 (2H,d, $J = 8.8$ Hz, ArH).

Acknowledgment

We gratefully acknowledge financial support from the Research Council of the Apadana Institute of Higher Education.

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