ISSN 1070-3632, Russian Journal of General Chemistry, 2016, Vol. 86, No. 4, pp. 934–938. © Pleiades Publishing, Ltd., 2016.

One-Pot Synthesis of Xanthene Derivatives Catalyzed by Fe(III) Tetranitrophthalocyanine Immobilized on Activated Carbon[1]

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Received October 22, 2015

Abstract—Xanthene derivatives were synthesized by a multicomponent one-pot reaction of aromatic aldehydes, β-naphthol, and 5,5-dimethylcyclohexane-1,3-dione or 1,3-cyclohexanedione upon efficient and eco-friendly catalysis of Fe(III) tetranitrophthalocyanine immobilized on activated carbon in ethanol. The method tolerated a variety of functional groups and the process was carried out under mild conditions to give high yield of products (85–91%) in 30 min. The catalyst can be recycled without any loss in catalytic activity.

Keywords: xanthene, Fe(III) tetranitrophthalocyanine immobilized on activated carbon, multicomponent

DOI: 10.1134/S1070363216040307

INTRODUCTION

Xanthene derivatives demonstrate various biological and pharmaceutical activities including anti-inflammatory [1], anti-bacterial [2] and anti-viral [3, 4]. Due to their specific spectroscopic properties, the xanthene derivatives found diverse applications in dyes industry [5], laser technology [6] and pH-sensitive fluorescent materials [7].

The earlier developed methods of synthesis of tetrahydrobenzo[*a*]xanthene-11-ones by condensation of aldehydes, β-naphthol and cyclic 1,3-dicarbonyl compounds had been catalyzed by a wide variety of catalysts [10–19]. Many of those methods led to moderate yields of products upon long reaction time, harsh conditions and involved toxic organic solvents [20].

Here in, we report a simple, efficient and environmentally friendly protocol for the synthesis of xanthene derivatives via one-pot multicomponent condensation of aromatic aldehydes, β-naphthol and 5,5-dimethylcyclohexane-1,3-dione or 1,3-cyclohexanedione catalyzed by Fe(III) tetranitrophthalocyanine immobilized on activated carbon [NO_2–Fe(III)Pc/C] upon refluxing in ethanol (Scheme 1).

RESULTS AND DISCUSSION

The optimized reaction conditions have been worked out for the reaction of benzaldehyde (1 mmol), β-naphthol (1 mmol) and 5,5-dimethylcyclohexane-

Table 1. Catalysts tested in the synthesis of 9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (**1a**)

Entry	Catalyst	Time, h	*T*, °C	Yield, %
1	–	12.0	Reflux	[a]
2	Cu(II)Pc/C	3.0	Reflux	11
3	Zn(II)Pc/C	3.0	Reflux	35
4	Co(II)Pc/C	3.0	Reflux	34
5	Ni(II)Pc/C	3.0	Reflux	41
6	Mn(II)Pc/C	3.0	Reflux	40
7	Fe(III)Pc/C	3.0	Reflux	52
8	NO_2–Cu(II)Pc/C	2.0	Reflux	21
9	NO_2–Zn(II)Pc/C	2.0	Reflux	59
10	NO_2–Co(II)Pc/C	2.0	Reflux	53
11	NO_2–Ni(II)Pc/C	2.0	Reflux	54
12	NO_2–Mn(II)Pc/C	2.0	Reflux	56
13	NO_2–Fe(III)Pc/C	0.5	Reflux	91

[a] Not determined.

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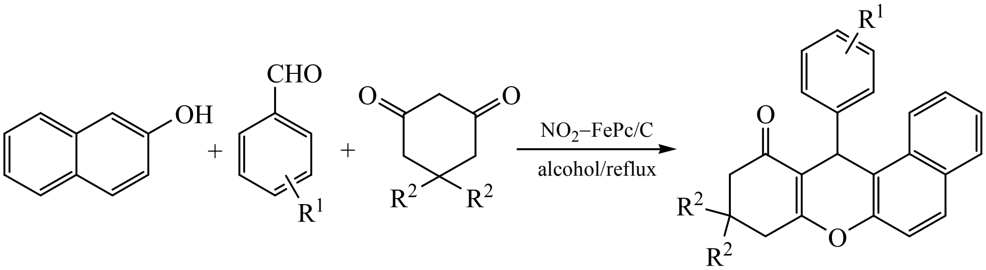
[1] The text was submitted by the authors in English.

Scheme 1. Synthesis of xanthene derivatives.



1a–1o

1a: R^1= H, R^2= CH_3; **1b**: R^1= 4-CH_3, R^2= CH_3; **1c**: R^1= 4-Cl, R^2= CH_3; **1d**; R^1= 4-OH, R^2= CH_3; **1e**: R^1 = 4-OCH_3, R^2 = CH_3; **1f**: R^1= 3-OCH_3-4-OH, R^2= CH_3; **1g**: R^1= 2,4-di-Cl, R^2= CH_3; **1h**: R^1= 4-NO_2, R^2= CH_3; **1i**: R^1 = 3-NO_2, R^2= CH_3; **1j**: R^1= H, R^2= H; **1k**: R^1= CH_3, R^2= H; **1l**; R^1= 4-Cl, R^2= H; **1m**; R^1= 4-OCH_3, R^2= H; **1n**: R^1 = 4-NO_2, R^2= H; **1o**: R^1 = 4-NO_2, R^2= H.

1,3-dione (1.2 mmol) considered as a model process. Various phthalocyanine catalysts were tested in the model reaction (Table 1). The desired product was not accumulated in the absence of catalysts. Fe(III) tetranitrophthalocyanine immobilized on activated carbon demonstrated the highest catalytic activity (Table 1, entry 13).

Among various solvents tested in the reaction (THF, $CHCl_3$, actone, CH_3CN, toluene, DMF, MeOH, and EtOH) the latter one proved to be the most efficient (Table 2). The most favorable reaction temperature and time of the process have been determined experimentally (Table 2, entry 8). The catalyst of choice, Fe(III) tetranitrophthalocyanine immobilized on activated carbon, could be recycled and used repeatedly (Table 3).

Table 2. Solvents tested in the synthesis of 9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (**1a**)

Entry	Solvent	Time, h	*T*, °C	Yield, %
1	THF	4.0	Reflux	12
2	$CHCl_3$	4.0	Reflux	14
3	Acetone	4.0	Reflux	15
4	CH_3CN	4.0	Reflux	35
5	Toluene	4.0	Reflux	38
6	DMF	4.0	Reflux	45
7	MeOH	1.0	Reflux	82
8	EtOH	0.5	Reflux	91
9	EtOH	5.0	50	63
10	EtOH	5.0	25	37

The experimental data demonstrated that the reaction proceeded smoothly with different aldehydes substituted with electron-donating or electron-withdrawing groups giving high yields of products (Table 4).

EXPERIMENTAL

Melting points were determined by an XT-4 melting point microscope without correction. IR spectra were recorded on a EQUINOX55 FT-IR spectrophotometer. ^{1}H NMR (400 M Hz) spectra were measured on a BRUKER pectrometer using $CDCl_3$ as a solvent. Mass spectra were measured on an Agilent AXIMA-CFR-plus MALDI-TOF-Ms Spectrometer. The reaction progress and purity of the products were monitored by TLC on silica gel.

General procedure for synthesis of compounds 1a–1i. A mixture of 5,5-dimethylcyclohexane-1,3-dione 0.168 g (1.2 mmol), β-naphthol 0.144 g (1 mmol), aromatic aldehyde (1 mmol), two drops of concentrated hydrochloric acid, ethanol (10 mL), and a catalytic amount of Fe(III) tetranitrophthalocyanine immobilized on activated carbon (5 mol %) was loaded

Table 3. Yield of the synthesis of **1a** catalyzed by the recycled Fe(III) tetranitrophthalocyanine immobilized on activated carbon in EtOH upon refluxing for 30 min

Run no.	Yield, %
1	91
2	91
3	90
4	90
5	89

Table 4. Yield of xanthene derivatives in the presence of Fe(III) tetranitrophthalocyanine immobilized on activated carbon

Comp. no.	R	R^1	Yield, %
1a	H	CH_3	91
1b	4-CH_3	CH_3	90
1c	4-Cl	CH_3	91
1d	4–OH	CH_3	89
1e	4-OCH_3	CH_3	88
1f	3-OCH_3–4-OH	CH_3	86
1g	2,4-Cl_2	CH_3	85
1h	4-NO_2	CH_3	89
1i	3-NO_2	CH_3	88
1j	H	H	93
1k	4-CH_3	H	90
1l	4-Cl	H	91
1m	4-OCH_3	H	88
1n	4-NO_2	H	89
1o	3-NO_2	H	86

in a 50 mL round-bottomed flask. and refluxed for ca 30 min upon stirring. The catalyst was recovered by filtration followed by washing with ethanol repeatedly and drying. The liquid filtrate was cooled and crystallized. The solid precipitated was filtered off, dried and purified by recrystallization from ethanol.

9,9-Dimethyl-12-phenyl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1a). Yield 91%, mp 148–150°C. IR spectrum, ν, cm^{-1}: 3055, 2956, 1651, 1595, 1373, 1226, 746, 698. ^{1}H NMR spectrum, δ, ppm: 0.96 s (3H, CH_3), 1.12 s (3H, CH_3), 2.23–2.33 m (2H, CH_2), 2.57 s (2H, CH_2), 5.70 s (1H, CH), 7.03–7.07 t (1H, *J* = 7.4 Hz, ArH), 7.15–7.19 t (2H, *J* = 7.6 Hz, ArH), 7.31–7.45 m (5H, ArH), 7.75–7.79 t (2H, *J* = 7.6 Hz, ArH), 7.99 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 377.1495. $C_{25}H_{22}O_2$. Calculated: 377.1517. $[M + Na^+]$.

9,9-Dimethyl-12-(4-methylphenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1b). Yield 90%, mp 173–175°C. IR spectrum, ν, cm^{-1}: 3043, 2951, 1649, 1597, 1371, 1228, 813, 756. ^{1}H NMR spectrum, δ, ppm: 0.97 s (3H, CH_3), 1.11 s (3H, CH_3), 2.19 s (3H, Ar–CH_3), 2.21–2.32 m (2H, CH_2), 2.56 s (2H, CH_2), 5.66 s (1H, CH), 6.97 d (2H, *J* = 8.0 Hz, ArH), 7.21–7.42 m (5H, ArH), 7.73–7.77 t (2H, *J* = 8.8 Hz, ArH), 8.00 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 369.1855. $C_{26}H_{24}O_2$. Calculated: 369.1855. $[M + H^+]$.

9,9-Dimethyl-12-(4-chlorophenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1c). Yield 91%, mp 179–181°C. IR spectrum, ν, cm^{-1}: 3078, 2952, 1649, 1595, 1371, 1226, 874, 752. ^{1}H NMR spectrum, δ, ppm: 0.96 s (3H, CH_3), 1.12 s (3H, CH_3), 2.22–2.33 m (2H, CH_2), 2.56 s (2H, CH_2), 5.68 s (1H, CH), 7.13 d (2H, *J* = 8.4 Hz, ArH), 7.25–7.45 m (5H, ArH), 7.76–7.79 t (2H, *J* = 7.2 Hz, ArH), 7.90 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 411.1116. $C_{25}H_{21}O_2Cl$. Calculated: 411.1128. $[M + Na^+]$.

9,9-Dimethyl-12-(4-hydroxyphenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1d). Yield 89%, mp 219–221°C. IR spectrum, ν, cm^{-1}: 3365, 3060, 2952, 1651, 1595, 1373, 1230, 819, 771. ^{1}H NMR spectrum, δ, ppm: 0.97 s (3H, CH_3), 1.11 s (3H, CH_3), 2.23–2.34 m (2H, CH_2), 2.56 s (2H, CH_2), 5.60 s (1H, OH), 5.63 s (1H, CH), 6.60 d (2H, *J* = 8.8 Hz, ArH), 7.17 d (2H, *J* = 8.4 Hz, ArH), 7.30–7.43 m (3H, ArH), 7.74–7.79 t (2H, *J* = 9.2 Hz, ArH), 7.97 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 371.1653. $C_{25}H_{22}O_3$. Calculated: 371.1647. $[M + H^+]$.

9,9-Dimethyl-12-(4-methoxyphenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1e). Yield 88%, mp 205–206°C. IR spectrum, ν, cm^{-1}: 3057, 2956, 1647, 1595, 1379, 1226, 837, 748. ^{1}H NMR spectrum, δ, ppm: 0.97 s (3H, CH_3), 1.11 s (3H, CH_3), 2.22–2.32 m (2H, CH_2), 2.56 s (2H, CH_2), 3.68 s (3H, OCH_3), 5.65 s (1H, CH), 6.70 d (2H, *J* = 8.4 Hz, ArH), 7.23–7.45 m (5H, ArH), 7.73–7.78 t (2H, *J* = 9.2 Hz, ArH), 7.98 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 385.1826. $C_{26}H_{24}O_3$. Calculated: 385.1804. $[M + H^+]$.

9,9-Dimethyl-12-(3-methoxy-4-hydroxyphenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1f). Yield 86%, mp 163–165°C. IR spectrum, ν, cm^{-1}: 3471, 3070, 2958, 1645, 1595, 1371, 1226, 877, 813, 752. ^{1}H NMR spectrum, δ, ppm: 0.97 s (3H, CH_3), 1.11 s (3H, CH_3), 2.23–2.33 m (2H, CH_2), 2.55 s (2H, CH_2), 3.81 s (3H, OCH_3), 5.48 s (1H, OH), 5.64 s (1H, CH), 6.63–6.68 m (2H, ArH), 7.00 s (1H, ArH), 7.30–7.43 m (3H, ArH), 7.74–7.78 t (2H, *J* = 9.6 Hz, ArH), 7.98 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 401.1757. $C_{26}H_{24}O_4$. Calculated: 401.1753. $[M + H^+]$.

9,9-Dimethyl-12-(2,4-dichlorophenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1g). Yield 85%, mp 179–181°C. IR spectrum, ν, cm^{-1}: 3070, 2954, 1652, 1597, 1369, 1228, 846, 752. ^{1}H NMR spectrum, δ, ppm: 0.99 s (3H, CH_3), 1.14 s (3H, CH_3), 2.21–2.34 m (2H, CH_2), 2.60 s (2H, CH_2), 5.94 s (1H, CH), 7.02–7.05 m (1H, ArH), 7.21–7.27 m (3H, ArH), 7.29–7.39 t (1H, *J* = 7.2 Hz, ArH), 7.46–7.50 t (1H, *J* = 7.4 Hz, ArH), 7.74–7.78 t (2H, *J* = 7.8 Hz, ArH), 8.12 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 423.0920. $C_{25}H_{20}O_2Cl_2$ [$M + H^+$]. Calculated: 423.0919.

9,9-Dimethyl-12-(4-nitrophenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1h). Yield 89%, mp 177–178°C. IR spectrum, ν, cm^{-1}: 3073, 2950, 1654, 1597, 1375, 1226, 810, 764. ^{1}H NMR spectrum, δ, ppm: 0.95 s (3H, CH_3), 1.14 s (3H, CH_3), 2.22–2.36 m (2H, CH_2), 2.60 s (2H, CH_2), 5.81 s (1H, CH), 7.35–7.43 m (3H, ArH), 7.52 d (2H, *J* = 8.8 Hz, ArH), 7.81–7.84 m (3H, ArH), 8.04 d (2H, *J* = 8.8 Hz, ArH). ESI-MS, *m/z*. Found: 422.1378. $C_{25}H_{21}O_4N$. Calculated: 422.1368. [$M + Na^+$].

9,9-Dimethyl-12-(3-nitrophenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1i). Yield 88%, mp 168–170°C. IR spectrum, ν, cm^{-1}: 3073, 2953, 1657, 1375, 1224, 963, 812, 764. ^{1}H NMR spectrum, δ, ppm: 0.95 s (3H, CH_3), 1.14 s (3H, CH_3), 2.22–2.35 m (2H, CH_2), 2.61 s (2H, CH_2), 5.81 s (1H, CH), 7.35–7.44 m (4H, ArH), 7.80–7.87 m (4H, ArH), 7.94 d (1H, *J* = 8.4 Hz, ArH), 8.11 s (1H, ArH). ESI-MS, *m/z*. Found: 422.1378. $C_{25}H_{21}O_4N$. Calculated: 422.1368. [$M + Na^+$].

General procedure for synthesis of compounds 1j–1o. A catalytic amount of Fe(III) tetranitrophthalocyanine immobilized on activated carbon (5 mol %) was added to a mixture of aromatic aldehyde (1 mmol), 1,3-cyclohexanedione (0.146 g, 1.2 mmol), β-naphthol (0.144 g, 1 mmol), two drops of concentrated hydrochloric acid, and ethyl alcohol (10 mL). The reaction mixture was refluxed for ca 30 min upon stirring. The catalyst was recovered by filtration followed by repeated washing with ethanol and drying. The liquid filtrate was cooled and crystallized. The solid obtained was filtered off, dried and purified by recrystallization from ethanol.

12-Phenyl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1j). Yield 93%, mp 188–190°C. IR spectrum, ν, cm^{-1}: 3053, 2955, 1651, 1594, 1373, 1226, 748, 680. ^{1}H NMR spectrum, δ, ppm: 1.95–2.08 m (2H, CH_2), 2.34–2.50 m (2H, CH_2), 2.62–2.78 m (2H, CH_2), 5.74 s (1H, CH), 7.05–7.08 t (1H, *J* = 7.4 Hz, ArH), 7.16–7.19 t (2H, *J* = 7.6 Hz, ArH), 7.32–7.44 m (5H, ArH), 7.75–7.79 t (2H, *J* = 7.0 Hz, ArH), 7.96 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 349.1199. $C_{23}H_{18}O_2$. Calculated: 349.1204. [$M + Na^+$].

12-(4-Methylphenyl)-8,9,10,12-tetrahydrobenzo-[*a*]xanthen-11-one (1k). Yield 90%. mp 201–203°C. IR spectrum, ν, cm^{-1}: 3022, 2960, 1661, 1595, 1371, 1224, 819, 746. ^{1}H NMR spectrum, δ, ppm: 1.97–2.05 m (2H, CH_2), 2.20 s (3H, CH_3), 2.33–2.48 m (2H, CH_2), 2.62–2.77 m (2H, CH_2), 5.70 s (1H, CH), 6.98 d (2H, *J* = 8.0 Hz, ArH), 7.22–7.44 m (5H, ArH), 7.74–7.78 t (2H, *J* = 8.0 Hz, ArH), 7.97 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 363.1361. $C_{24}H_{20}O_2$. Calculated: 363.1361. [$M + Na^+$].

12-(4-Chlorophenyl)-8,9,10,12-tetrahydrobenzo[*a*]-xanthen-11-one (1l). Yield 91%, mp 206–208°C. IR spectrum, ν, cm^{-1}: 3076, 2962, 1651, 1594, 1375, 1222, 856, 718. ^{1}H NMR spectrum, δ, ppm: 1.94–2.10 m (2H, CH_2), 2.34–2.45 m (2H, CH_2), 2.62–2.76 m (2H, CH_2), 5.71 s (1H, CH), 7.13 d (2H, *J* = 8.4 Hz, ArH), 7.27–7.43 m (5H, ArH), 7.77–7.80 t (2H, *J* = 7.0 Hz, ArH), 7.88 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 361.1003. $C_{23}H_{17}O_2Cl$. Calculated: 361.0095. [$M + H^+$].

12-(4-Methoxyphenyl)-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-one (1m). Yield 88%, mp 180–181°C. IR spectrum, ν, cm^{-1}: 3056, 2960, 1661, 1376, 1222, 832, 753. ^{1}H NMR spectrum, δ, ppm: 1.92–2.10 m (2H, CH_2), 2.38–2.48 m (2H, CH_2), 2.66–2.76 m (2H, CH_2), 3.69 s (3H, OCH_3), 5.69 s (1H, CH), 6.71 d (2H, *J* = 8.8 Hz, ArH), 7.23–7.44 m (5H, ArH), 7.74–7.78 t (2H, *J* = 8.8 Hz, ArH), 7.95 d (1H, *J* = 8.4 Hz, ArH). ESI-MS, *m/z*. Found: 379.1305. $C_{24}H_{20}O_3$. Calculated: 379.1310. [$M + Na^+$].

12-(4-Nitrophenyl)-8,9,10,12-tetrahydrobenzo[*a*]-xanthen-11-one (1n). Yield 89%, mp 236–238°C. IR spectrum, ν, cm^{-1}: 3069, 2953, 1664, 1373, 1224, 810, 726. ^{1}H NMR spectrum, δ, ppm: 1.94–2.07 m (2H, CH_2), 2.36–2.49 m (2H, CH_2), 2.71–2.80 m (2H, CH_2), 5.84 s (1H, CH), 7.36–7.44 m (5H, ArH), 7.51 d (2H, *J* = 8.8 Hz, ArH), 7.80–7.82 t (2H, ArH), 8.04 d (1H, *J* = 8.8 Hz, ArH). ESI-MS, *m/z*. Found: 394.1052. $C_{23}H_{17}O_4N$. Calculated: 394.1055. [$M + Na^+$].

12-(3-Nitrophenyl)-8,9,10,12-tetrahydrobenzo[*a*]-xanthen-11-one (1o). Yield 86%, mp 232–234°C. IR spectrum, ν, cm^{-1}: 3072, 2963, 1656, 1371, 1226, 954, 837, 748. ^{1}H NMR spectrum, δ, ppm: 1.94–2.10 m (2H, CH_2), 2.35–2.50 m (2H, CH_2), 2.66–2.83 m (2H, CH_2), 5.84 s (1H, CH), 7.36–7.42 m (4H, ArH), 7.80–7.84 m (4H, *J* = 8.8 Hz, ArH), 7.94 d (1H, *J* = 8.4 Hz, ArH), 8,09 s (1H, ArH). ESI-MS, *m/z*. Found: 394.1054. $C_{23}H_{17}O_4N$. Calculated: 394.1055. [$M + Na^+$].

CONCLUSIONS

The highly efficient and green method of synthesis of xanthene derivatives catalyzed by Fe(III) tetranitrophthalocyanine immobilized on activated carbon in ethanol has been developed. The simple procedure, short reaction time, efficient recovery, and non-toxicity of the catalyst are the advantages of the method.

ACKNOWLEDGMENTS

We gratefully acknowledge financial support from the Industrial Research Project of Shaanxi Science and Technology Department (2014K08-29), National Basic Science Personnel Training Fund (J1103311 and J1210057) and Scientific Research Foundation of Northwestern University.

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