ORIGINAL ARTICLE

A C G publications

Org. Commun. XX:X (202X) X-XX

organic communications

Ferric phosphate ($FePO_4$): An efficient and reusable catalyst for synthesis of aryl-14*H*-dibenzo[*a,j*]xanthene derivatives under solvent-free conditions

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(Received August 10, 2025; Revised September 22,2025; Accepted September 24, 2025)

Abstract: A simple and efficient method has been developed for the synthesis of xanthene derivatives using various aromatic aldehydes and 2-naphthol under solvent-free conditions. In this procedure, ferric phosphate ($FePO_4$) is used as an efficient and reusable heterogeneous Lewis acid catalyst for the synthesis of various derivatives of 14-aryl-14*H*-dibenzo[*a,j*]xanthene (**3a-3m**) in excellent yields (**87-96%**). The present method affords notable advantages such as short reaction time, simple workup procedure, reusability of the catalyst and high conversions of the products. All products have been confirmed by their melting points and spectroscopic techniques such as ^{1}H NMR, ^{13}C NMR, IR spectroscopy and mass spectrometry.

Keywords 14-aryl-14*H*-dibenzo[*a,j*]xanthenes; ferric phosphate ($FePO_4$); heterogeneous Lewis acid catalyst; aromatic aldehydes; 2-naphthol; solvent-free condition.

1. Introduction

Xanthene and its derivatives are an important class of organic compounds, which show significant application in medicinal as well as in industrial chemistry. They display several biological activities including anti-inflammatory[1], anti-malarial[2], anti-cancer[3], anti-bacterial[4], anti-fungal[5] and anti-viral[6]. Additionally, some xanthene derivatives have important industrial applications, being used as dyes[7], pH-sensitive fluorescent materials for the visualization of biomolecular assemblies[8], as well as in laser technology[9] and photodynamic therapy[10]. Due to the diverse applications of xanthene and its derivatives, their syntheses have attracted increasing attention from chemists worldwide.

In view of the various important applications of xanthene and its derivatives, numerous methods have been reported for the synthesis of 14*H*-dibenzo[*a,j*]xanthene derivatives through the condensation of 2-naphthol with various aldehydes, employing different catalysts such as organo-catalysts[11-17], homogeneous Lewis acids[18-25], heterogeneous Lewis acids[26-32], homogeneous Brønsted acids[33-34], and heterogeneous Brønsted acids[35-43].

In addition, various ionic liquids[44-50] and basic conditions[51] are also used for their syntheses. Most of these reported approaches have their own one or two demerits such as use of expensive catalysts, high reaction temperature, prolonged reaction time, dreary workup procedures or low product yields.

Therefore, it is still necessary to develop novel and environmentally benign green chemical methods for synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives using reusable catalysts.

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The article was published by ACG Publications
http://www.acgpubs.org/journal/organic-communications © Month-Month 2025 EISSN:1307-6175
DOI: http://doi.org/10.25135/acg.oc.198.2508.3606
Available online: October 05, 2025

Efficient and reusable catalyst for synthesis of aryl-14*H*-dibenzo[*a,j*]xanthene derivatives

In recent years, use of heterogenous Lewis acidic catalysts have been offered significant advantages in organic transformations, due to their low cost, non-toxicity, eco-friendly nature, reusability and ease of isolation[52-54]. Among them, one of heterogenous Lewis acidic catalyst ferric phosphate ($FePO_4$) has received more attention due to their inexpensiveness, easily available, non-corrosive and environmentally benign characteristics. Owing of these advantages related to ferric phosphates ($FePO_4$), it has been used as a powerful Lewis acid catalyst for development of numerous methodologies[55-59].

In continuation of our research interest in the development of novel and environmentally benign methodologies[60-68]. Herein, we report ferric phosphate ($FePO_4$): An efficient and reusable catalyst for synthesis of aryl-14*H*-dibenzo[*a,j*]xanthene derivatives under solvent-free conditions.

2. Experimental

2.1. Chemical Material and Apparatus

All reagents and solvents were purchased from commercial sources and were used without any further purification. Thin layer chromatography (TLC) performed on Aluminum covered silica plates purchased from Merck. Melting points were determined in the Buchi R-535 apparatus. All melting points were determined in an open capillary tube and uncorrected. ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ with Gemini-300 spectrophotometer using tetramethyl silane (TMS) as internal standard. IR spectra were recorded in KBr disk using a Bruker FT-IR spectrophotometer and mass spectra were recorded on a Finnigan MAT 1020 mass spectrometer with operating at 70 eV.

2.2 General Procedure

A mixture of 2-naphthol (2 mmol) and aromatic aldehyde (1 mmol) (**3a-3m**) in presence of anhydrous ferric phosphate ($FePO_4$) (20 % mol) were stirred and heated at 90°C using oil bath for 45 minutes. After completion of reaction monitoring by TLC, reaction mixture was cooled to room temperature and ethyl alcohol was added (10 mL). Combined reaction mixture was filtered out and catalyst was separated. Collected filtrate was evaporated to obtain crude solid product which was recrystallized using ethanol. All the pure compounds were confirmed by comparing with their melting points and spectral data.

2.3. Spectral Data of Synthesized Compounds

*14-Phenyl-14H-dibenzo[a,j]xanthene (**3a**)*: White solid, m.p. 184-186 °C (Lit[35] :181 °C); IR (KBr): ν_{max} = 3072, 3019, 2885, 1622, 1593, 1512, 1485, 1405, 1253, 1030, 967, 830, 745 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.39 (d, *J* = 8.5 Hz, 2H), 7.84 (d, *J* = 7.9 Hz, 2H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.57 (t, *J* = 7.7 Hz, 2H), 7.52-7.40 (m, 4H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.15 (t, *J* = 7.5 Hz, 2H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.50 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.7, 144.9, 131.6, 131.1, 128.8, 128.7, 128.5, 128.2, 126.7, 126.2, 125.2, 122.6, 118.1, 117.4, 38.0 ppm; ESIMS: *m/z* = 381[M^{+23}], 358 [M^+].

*14-(4-Methylphenyl)-14H-dibenzo[a,j]xanthene (**3b**)*: White solid, m.p. 224-226 °C (Lit[35]: 227 °C); IR (KBr): ν_{max} = 3061, 3029, 2923, 1621, 1596, 1506, 1447, 1414, 1238, 1119, 1073, 1027, 815, 747cm^{-1} ; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.42 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.78 (d, *J* = 8.8 Hz, 2H), 7.60 (t, *J* = 8.2 Hz, 2H), 7.50 (d, *J* = 8.7 Hz, 2H), 7.42-7.30 (m, 4H), 6.96 (d, *J* = 7.7 Hz, 2H), 6.46 (s, 1H), 2.16 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.2, 142.3, 135.8, 130.6, 129.0, 128.5, 128.4, 127.9, 127.4, 126.6, 124.5, 123.1, 117.5, 117.1, 37.5, 20.1 ppm; ESIMS: *m/z* = 372 [M^+].

*14-(4-Hydroxyphenyl)-14H-dibenzo[a,j]xanthenes (**3c**)*: White solid, m.p. 136-138 °C (Lit[27]: 133-134 °C); IR (KBr): ν_{max}= 3394, 3056, 2932, 1623, 1589, 1514, 1460, 1399, 1249, 1217, 1065, 822, 740,703 cm^{-1} ; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.18 (d, *J* = 8.4 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 7.18-7.04 (m, 4H), 6.42 (d, *J* = 8.4 Hz, 2H), 6.24 (s, 1H), 5.52 (brs, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 153.7, 148.6, 137.5,

131.5, 131.3, 129.6, 129.3, 127.6, 126.7, 124.3, 122.6, 118.0, 117.3, 115.3, 37.4 ppm; ESIMS: *m/z* = 374 [M^+].

*14-(4-Methoxyphenyl)-14H-dibenzo[a,j]Xanthene (**3d**)*: White solid, m.p. 202-204 °C (Lit[50] :202-203 °C); IR (KBr): ν_{max}= 3063, 3012, 2837, 1623, 1592, 1505, 1458, 1445, 1399, 1250, 1216, 1029, 837, 745 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.33 (d, *J* = 8.4 Hz, 2H), 7.48-7.76 (m, 12H), 6.60 (d, *J* = 8.4 Hz, 2H), 6.40 (s, 1H), 3.60 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 158.2, 148.7, 137.4, 131.4, 131.0, 129.6, 128.8, 128.5, 124.2, 122.9, 118.1, 117.0, 114.1, 54.8, 37.1 ppm; ESIMS: *m/z* 388 [M^+].

*14-(2,5-Dimethoxyphenyl-14H-dibenzo[a,j]xanthene (**3e**)*: White solid, m.p. 170-172 °C (Lit[35] : 169 °C); IR (KBr):ν_{max} = 2926, 2857, 1622, 1594, 1502, 1463, 1401, 1248, 1076, 1038, 860, 810, 756 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.60 (d, *J* = 8.6 Hz, 2H), 7.80 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.6 Hz, 2H), 7.59 (t, *J* = 7.5 Hz, 2H), 7.50 (d, *J* = 8.6 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 6.92 (s, 1H), 6.83 (s, 1H), 6.78 (d, *J* = 8.6 Hz, 1H), 6.44 (d, *J* = 6.4 Hz, 1H), 4.25 (s, 3H), 3.46 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 154.1, 148.9, 148.1, 135.5, 132.0, 129.9, 129.6, 128.6, 128.4, 127.6, 124.1, 123.3, 118.3, 117.0, 112.1, 111.5, 56.0, 55.2, 30.6 ppm; ESIMS: *m/z* = 418 [M^+].

*14-(3,4-Dimethoxyphenyl)-14-H-dibenzo[a,j]xanthene (**3f**)*: White solid, m.p. 202-204 °C (Lit[22]: 208-210 °C) IR (KBr):ν_{max} = 3059, 2928, 1622, 1594, 1513, 1463, 1248, 1076, 1036, 810, 776 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.14 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 7.7 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 2H), 7.16 (t, *J* = 7.1 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H), 6.96 (s, 1H), 6.76 (d, *J* = 8.2 Hz, 1H), 6.67 (s, 1H), 6.42 (d, *J* = 8.1 Hz, 1H), 3.60 (s, 3H), 3.53 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 149.3, 147.7, 137.2, 135.1, 129.3, 129.2, 129.1, 126.9, 126.4, 124.5, 122.6, 121.2, 120.8, 120.2, 113.6, 112.2, 56.3, 56.2, 41.8 ppm; ESIMS: *m/z* 441 (M^{+23}), 418 [M^+].

*14-(4-Dimethylamino-phenyl)-14H-dibenzo[a,j]Xanthene (**3g**)*: Faint yellow solid, m.p. 196-198 °C (Lit[50]:197-199 °C); IR (KBr):ν_{max} = 3045, 2925, 1624, 1596, 1491, 1454, 1390, 1348, 1276, 1054, 748 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.08 (d, *J* = 8.4 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 9.0 Hz, 4H), 7.60 (t, *J* = 7.5 Hz, 4H), 7.42-7.37 (m, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.20 (s, 1H), 4.59 (s, 6H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 154.4, 148.3, 137.5, 135.3, 131.5, 129.9, 129.7, 128.7, 128.4, 126.9, 124.4, 122.4, 118.3, 117.2, 113.7, 55.6, 41.4 ppm; ESIMS: *m/z* 401 [M^+].

*14-(4-benzyl)-14-H-dibenzo[a,j]xanthene (**3h**)*: White solid, m.p. 182-184 °C (Lit[38]: 178-180 °C); IR (KBr):ν_{max} = 3067, 2920, 2882, 1612, 1585, 1488, 1445, 1390, 1344, 1249, 1206, 1077, 1023, 863, 749 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.22 (d, *J* = 8.5 Hz, 2H), 7.76-7.54 (m, 8H), 7.08-6.90 (m, 5H), 6.10 (d, *J* = 8.7 Hz, 2H), 5.78 (t, *J* = 4.6 Hz, 1H), 3.24 (d, *J* = 4.6 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 150.5, 137.6, 131.5, 130.7, 129.6, 128.7, 128.3, 127.1, 126.9, 126.1, 124.2, 122.3, 117.3, 115.2, 41.3, 33.2 ppm; ESIMS: *m/z* 372 [M^+].

*14-(4-Chlorophenyl)-14H-dibenzo[a,j]Xanthene (**3i**)*: Yellow solid, m.p. 286-288 °C (Lit[50] :283-287 °C); IR (KBr):ν_{max} = 3066, 2915, 2851, 1624, 1596, 1480, 1410, 1384, 1255, 1206, 1050, 844, 783, 745 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.30 (d, *J* = 8.5 Hz, 2H), 7.89 (d, *J* = 8.0 Hz, 2H), 7.80 (d, *J* = 8.8 Hz, 2H), 7.65-7.57 (m, 2H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.44-7.34 (m, 4H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.48 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.8, 143.4, 132.1, 131.3, 131.0, 129.5, 129.2, 129.0, 128.7, 127.0, 124.4, 122.4, 118.1, 116.7, 37.4 ppm; ESIMS: *m/z* = 394 [M^{+2}], 392 [M^+].

*14-(2,4-dichlorophenyl)-14H-dibenzo[a,j]xanthene (**3j**)*: Pale yellow solid, m.p. 225-227 °C (Lit[35]: 227 °C); IR (KBr):ν_{max} = 3062, 2925, 2862, 1622, 1591, 1562, 1518, 1463, 1406, 1246, 1208, 1078, 1045, 864, 816, 752 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 8.64 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 7.8 Hz, 2H), 7.79 (d, *J* = 8.7 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 2H), 7.51-7.39 (m, 4H), 7.36-7.26 (m, 2H), 6.88 (d, *J* = 6.7 Hz, 1H), 6.72 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.8, 142.2, 132.7, 132.4, 131.3, 130.7, 130.5, 129.6, 129.1, 128.6, 128.5, 127.1, 124.5, 123.1, 118.1, 117.4, 34.2 ppm; ESIMS: *m/z* 426 [M^+].

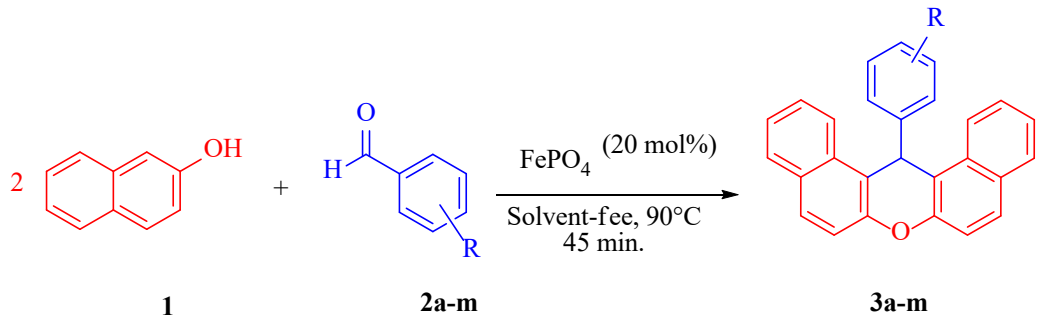
14-(4-Bromophenyl)-14H-dibenzo[a,j]Xanthene (***3k***): Pink solid, m.p. 296-298 °C (Lit[25]: 300 °C); IR (KBr):ν_{max} = 3060, 2934, 2831, 1626, 1590, 1405, 1236, 1010, 807, 739, 702 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.31 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.2 Hz, 2H), 7.78 (d, *J* = 8.7 Hz, 2H), 7.64 (t, *J* = 7.6 Hz, 2H), 7.54 (d, *J* = 8.7 Hz, 2H), 7.48-7.37 (m, 4H), 7.32-7.24 (m, 2H), 6.42 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.7, 144.1, 131.7, 131.5, 131.2, 129.9, 129.2, 129.1, 127.1, 124.4, 122.5, 120.3, 118.0, 116.7, 37.5 ppm; ESIMS: *m/z* = 438 [M^{+2}], 436 [M^+].

14-(3-Nitrophenyl)-14H-dibenzo[a,j]xanthene (***3l***): Yellow solid, m.p. 210-212 °C (Lit[35]: 214 °C); IR (KBr):ν_{max} = 3061, 3019, 1617, 1587, 1511, 1488, 1451, 1397, 1241 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.64 (d, *J* = 8.5 Hz, 2H), 8.08-7.90 (m, 4H), 7.72-7.60 (m, 4H), 7.54 (d, *J* = 8.7 Hz, 2H), 7.45 (d, *J* = 7.7 Hz, 2H), 7.21 (s, 2H), 6.78 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 148.4, 145.1, 141.4, 140.2, 131.5, 131.3, 131.1, 130.2, 129.8, 127.6, 125.1, 123.6, 118.1, 117.3, 36.2 ppm; ESIMS: *m/z* 403 [M^+].

14-(4-Nitrophenyl)-14H-dibenzo[a,j]xanthene (***3m***): Yellow solid, m.p. 312-314 °C (Lit[35] : 315 °C); IR (KBr):ν_{max} = 3061, 2932, 1625, 1598, 1512, 1456, 1402, 1210, 1027, 771, 695 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ = 8.30 (d, *J* = 8.2 Hz, 2H), 7.98 (d, *J* = 8.8 Hz, 2H), 7.87 (quasi d, *J* = 4.4 Hz, 2H), 7.80 (quasi d, *J* = 5.4 Hz, 2H), 7.68 (d, *J* = 8.7 Hz, 2H), 7.59 (t, *J* = 5.4 Hz, 2H), 7.49 (d, *J* = 8.7 Hz, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 6.60 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.2, 148.7, 146.2, 135.2, 131.0, 129.7, 129.0, 128.8, 127.3, 124.7, 123.6, 123.6, 122.1, 117.9, 115.9, 37.6 ppm; ESIMS: *m/z* 403 [M^+].

3. Results and Discussion

In a model reaction, the condensation reaction occurs in between of 2-naphthol (2 mmol) (**1**) and benzaldehyde (1 mmol) (**2a**) using anhydrous ferric phosphate ($FePO_4$) (20% mol) as Lewis acid catalyst under solvent-free condition at 90°C. The reaction was completed within 45 min. to obtain corresponding product 14-Phenyl-14*H*-dibenzo[*a,j*]xanthene (**3a**) in 92% yields (Scheme 1).



1 **2a-m** **3a-m**

Scheme 1. Ferric phosphate ($FePO_4$) catalyzed synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives

To optimize reaction conditions, preliminary experiments were carried out to study several parameters, such as catalyst, temperature, and solvent, using the model reaction. Initially, the reaction started without using any catalyst at room temperature, but desired product was not obtained even extending reaction time (Table 1, entry 1). After that, we moved to increase reaction temperature by 90°C in 240 minutes, then only trace amount of product was obtained (Table 1, entry 2). So, we understood that the role of catalyst is crucial for the progression of reaction. By considering the importance of catalyst, we decided to use inexpensive, readily available, non-toxic, highly stable and reusable anhydrous ferric phosphate ($FePO_4$) as catalyst at 90°C. Initially, 5 mol% catalyst was used for screening and 38 % yields of the corresponding product **3a** was obtained in 120 minutes (Table 1, entry 3). Next, we increased the mole ratio of catalyst from 10-20 mol%, yields of corresponding product **3a** were obtained with 65, 78 and 92% by decreasing reaction time from 90, 60 and 45 minutes respectively (Table 1, entry 4-6). However, further increasing the amount of catalyst by 25 mol% but no significant improvement was observed in the product yield (Table 1, entry 7). After optimization

of catalyst, it realized that maximum product yield was obtained within 45 minutes, when 20 mol% catalyst was used. After getting these results, we decided to optimize the reaction temperature by changing from room temperature to 80ºC by keeping same mole ratio of catalyst and reaction time but, yield of the product was not increased up to the mark (Table 1, entry 8-12). All the results are summarized in Table **1**.

Table 1. Optimization of catalyst at different temperature under solvent-free conditions

Entry	$FePO_4$ (% mol)	Temperature (ºC)	Time (min.)	Isolated Yields (%)
1	Catalyst-free	RT	300	--
2	Catalyst-free	90	240	trace
3	5	90	120	38
4	10	90	90	65
5	15	90	60	78
6	**20**	**90**	**45**	**92**
7	25	90	45	92
8	20	RT	45	23
9	20	50	45	58
10	20	60	45	69
11	20	70	45	78
12	20	80	45	87

RT = Room Temperature.

After optimization of catalyst and temperature, we moved towards screening of solvents effect by using 20% mol of catalyst. Initially, the reaction was carried out using water as green solvent but yield of the corresponding product **3a** was obtained only 32 % with extended reaction time i.e. 180 minutes (Table 2, entry 1).

Table 2. Optimization of solvents

Entry	$FePO_4$ (% mol)	Solvent	Temperature	Time (min.)	Isolated Yields (%)
1	20	Water	Reflux	180	32
2	20	DCM	Reflux	120	57
3	20	THF	Reflux	120	59
4	20	CH_3CN	Reflux	120	68
5	20	Acetone	Reflux	120	72
6	20	Ethanol	Reflux	90	84
7	**20**	**Solvent-free**	**90 ºC**	**45**	**92**

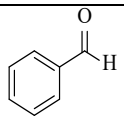
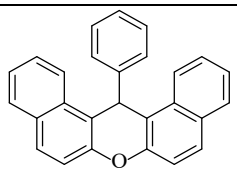
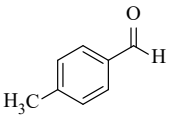
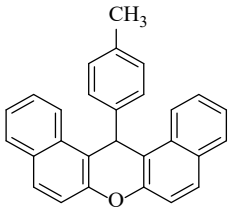
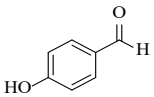
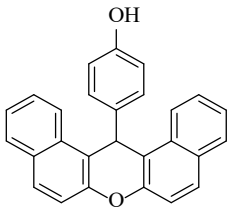
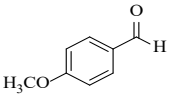
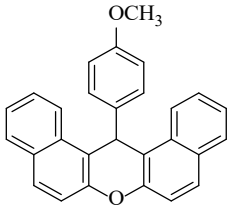
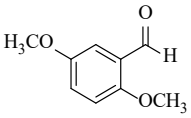
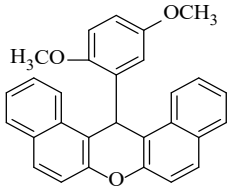
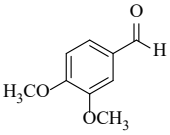
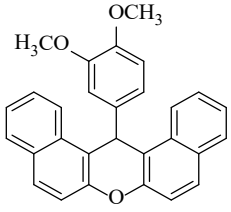
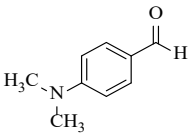
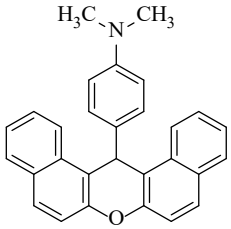
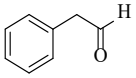
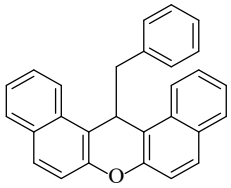
Next, we used various solvents systems such as DCM, THF, CH_3CN, as well as acetone at reflux conditions and yields of the corresponding product 3a were obtained with 54, 59, 67, 72 % respectively in 120 mins. (Table 2, entry 2-5). Later, ethanol was used at reflux condition to give corresponding product 3a in 84 % yield with reduced reaction time (Table 2, entry 6). After observation, we realized that none of the above solvents gave expected results. Hence, we decided to use solvent-free condition, which offered best results with 92 % product yield within 45 minutes (Table 2, entry 7). All the results are summarized in Table 2.

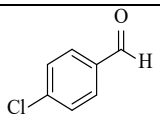
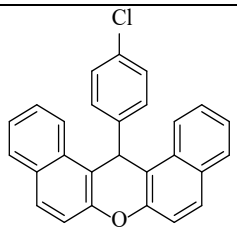
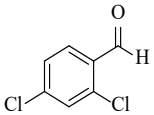
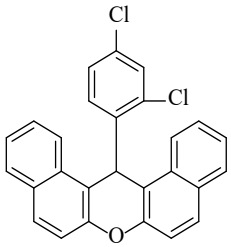
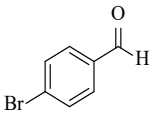
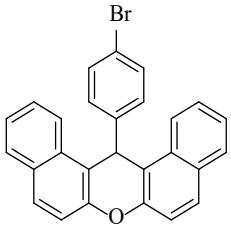
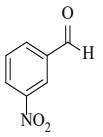
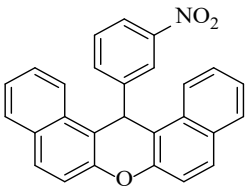
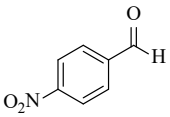
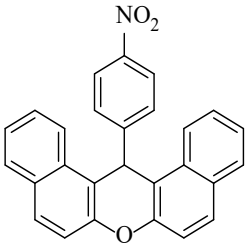
Based on these optimal reaction conditions, various aromatic aldehydes reacted with 2-napththol for the synthesis of 14-Aryl-14*H*-dibenzo[*a,j*]xanthene derivatives (**3a-3m**) to demonstrate the scope of this catalyst. This method was found to be equally effective for aromatic aldehyde having electron donating as well as withdrawing functional groups. All the reactions were carried out under solvent-free conditions using ferric phosphate ($FePO_4$) as a Lewis acid catalyst.

In general, all the reactions were clean in terms of conversion and separation of their products. All the products were confirmed by their melting points and spectroscopic methods such as ^1H, ^{13}C NMR, IR spectroscopy and mass spectrometry.

Efficient and reusable catalyst for synthesis of aryl-14*H*-dibenzo[*a,j*]xanthene derivatives

Table 3. Synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives

Sr No	Aldehyde	Product	Reaction Time (min.)	Isolated Yield (%)	M.P. ºC [Lit. M.P.][Ref]
a			45	92	184-186 [181][35]
b			45	92	224-226 [227] [35]
c			45	90	136-138 [133-134] [27]
d			45	90	202-204 [202-203] [50]
e			45	86	170-172 [169][35]
f			45	88	202-204 [208-210] [22]
g			45	87	196-198 [197-199][50]
h			45	90	182-184 [178-180][38]

i			45	93	286-288 [283-287] [50]
j			45	95	225-227 [227] [35]
k			45	93	296-298 [300] [25]
l			45	94	210-212 [214] [35]
m			45	95	312-314 [315] [35]

Recycling and reuse of catalyst is most important in green synthesis. Therefore, in the recovery procedure of $FePO_4$, ethyl alcohol was added to the reaction mixture after completion of the reaction. The catalyst was insoluble in the ethyl alcohol and separated by simple filtration. The recovered catalyst was washed with ethyl alcohol and dried using hot air oven. This catalyst was reused up to four times for further reactions without loss of its significant efficiency (Table 4).

Table 4. Recyclability study of ferric phosphate ($FePO_4$)

Sr. No.	Cycle	Reaction Time (min.)	Isolated Yields (%)
1	1	45	92
2	2	45	90
3	3	50	87
4	4	60	83

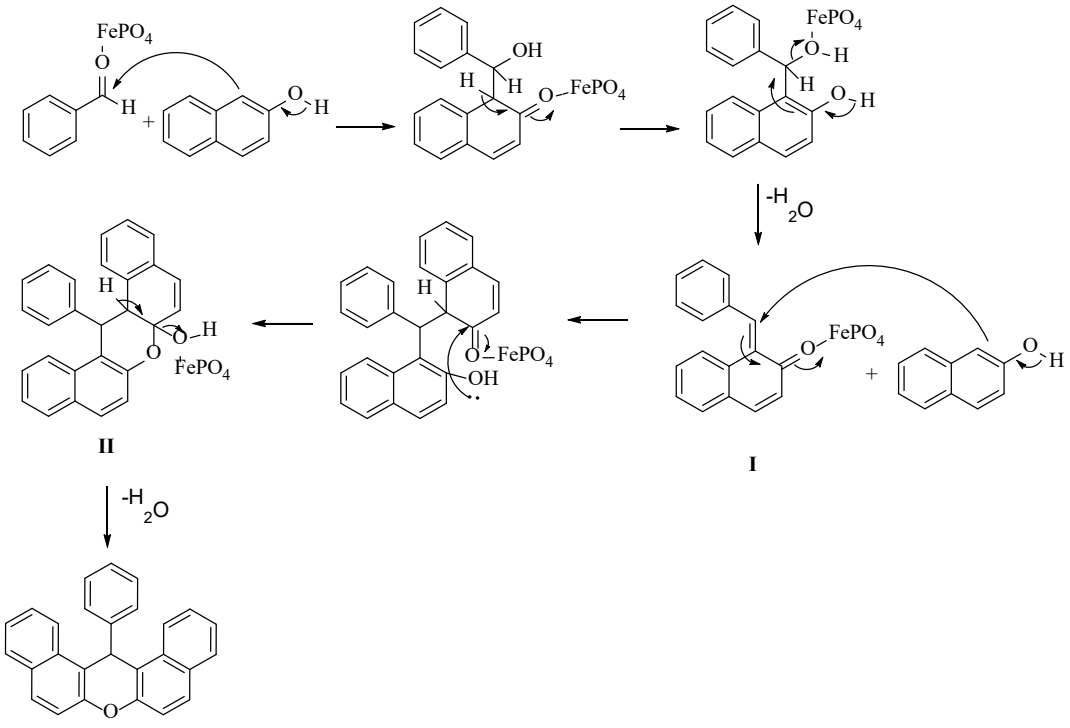
After that, ferric phosphate ($FePO_4$) catalyst has also compared with some previously reported catalyst for synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives (Table 5). The results show that the present method has more advantages from the viewpoint of product yield and reaction time.

Efficient and reusable catalyst for synthesis of aryl-14*H*-dibenzo[*a,j*]xanthene derivatives

Table 5. Comparison for different catalyst for synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives[a]

Entry	Catalyst	Conditions	Time	Yields (%)[Ref]
1	Sulfamic acid	solvent-free, 125°C	8 h	93[11]
2	I_2	neat, 90°C	2.5 h	90[18]
3	$Yb(OTf)_3$	solvent-free, 90°C	1.5 h	92[19]
4	$ln(OTf)_3$	H_2O, 100°C	8 h	82[20]
5	silica sulfuric acid	solvent-free, 80°C	45 min.	89[36]
6	$NaHSO_4•H_2O$	solvent-free, 125°C	23 min.	92[37]
7	Montmorillonite K10	solvent-free, 120°C	3 h	75[26]
8	Boric acid	neat, 120°C	2h	94[21]
9	$HClO_4$	CH_3COOH, 55°C	60 min.	98[33]
10	SaSA	solvent-free, 120°C	50 min.	94[39]
11	$FeCl_3 \cdot 6H_2O$	solvent-free, 90°C	2 h	86.6[24]
12	$CuSO_4.5H_2O$	solvent-free, 80°C	5 h	95[23]
13	$NbCl_5$	DCM, ambient temperature	48 h	90[25]
14	SA-Sn	solvent-free, 120°C	1.5h	96[42]
15	Sulfonated fructose	EtOH:H_2O, Reflux	30 min.	91[43]
16	Ionic liquid			
	i) [Dsim]Cl	solvent-free, 110°C	5 min.	90[50]
	ii) $[Msim]PF_6$	solvent-free, 110°C	7 min.	85[50]
	iii) $[Msim]BF_4$	solvent-free, 110°C	8 min.	88[50]
17	**$FePO_4$**	**solvent-free, 90°C**	**45 min.**	**92[this work]**

[a]Based on the three-component reaction of 2-naphthol (2.0 mmol) and benzaldehyde (1.0 mmol).



Scheme 2. Plausible reaction mechanism

The formation of product can be explained as shown in plausible reaction mechanism (Scheme 2). The Lewis acid has activated the carbonyl carbon of aromatic aldehydes by coordination with oxygen, which leads to formation of intermediate product **I** reacting with 2-naphthol followed by removal of water molecule. In the next step, second mole of 2-naphthol is reacting with product **I**

followed by intra molecular cyclisation to give intermediate **II**, which undergoes removal of water molecule to obtain desired product 14-substituted-14*H*dibenzo [*a,j*]xanthene obtained.

4. Conclusion

In summary, we have developed a simple and efficient method for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives from easily available and inexpensive starting materials. In this method, ferric phosphate used as heterogeneous Lewis acid catalyst for reaction of 2-napthol and various aromatic aldehyde. All reactions proceed smoothly under solvent-free conditions. This novel process has many significant advantages such as, environmentally benign, shorter reaction time, simple workup procedure, recyclability of catalyst and high conversions of the products with excellent yield.

Acknowledgements

All authors are thankful to department of chemistry, B. N. N. College, Bhiwandi and DST-FIST Delhi for providing laboratory and instrumentation facility for this work.

Supporting Information

Supporting information accompanies this paper on http://www.acgpubs.org/journal/organic-communications

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