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Synthesis of Dibenzo[*b*,*i*]xanthene-5,7,12,14(13*H*)-tetraone using *p*-Toluenesulfonic Acid as Catalyst

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This methodology involves synthesis of dibenzo xanthene tetraone by one pot multicomponent reaction of 2-hydroxy-1,4-naphthoquinone and pyrazole aldehyde in presence of *p*-toluenesulfonic acid (PTSA) as catalyst in ethylene glycol at 75-95 ºC. This protocol provides excellent yields (80-90%) involving simple workup procedures and purification techniques. By using several spectroscopic techniques the compounds were characterized.

Keywords: Xanthene, Pyrazole aldehydes, 2-Hydroxy-1,4-naphthoquinone, *p*-Toluenesulfonic acid.

INTRODUCTION

Modern discovery of drugs through synthetic organic chemistry involves multicomponent reactions (MCRs). This strategy engages synthesis of new products by combining two or more reactants imbibing most of constituents of all the reactants [1]. A large number of new drug like molecules are synthesized by this technique which are proved to have many biological applications. This technique is more advantageous over the traditional multi-step synthesis in different aspects like low reaction time, producing minimal waste, cost effective and have high atom economy utilizing easily available, low costs solvents and catalysts.

To assist the medicine discovery against COVID-19, the binding affinity of xanthene-triazole-chloroquinoline/xanthene-chloroquinoline is studied against main proteases of SARS-CoV-2 and found to be potential in inhibition. Xanthene chore is proved to inhibit corona virus [2]. The compounds having xanthene moiety have high pharmacological properties like antimicrobial, antimalarial, antibacterial, antiviral, antitumor, antiparasitic, cytotoxic, anti-inflammatory, analgesic, anti-diabetic, antihistaminic, antipsychotic, anticonvulsant [3], anti-oxidant [4], antifungal [5] activities and used as sensitizers in photodynamic therapy [6]. Xanthenes are used as food dyes and for visualization of biomolecules as chemical probes [7]. Literature survey reveals several methods for the synthesis of xanthenes [8-22].

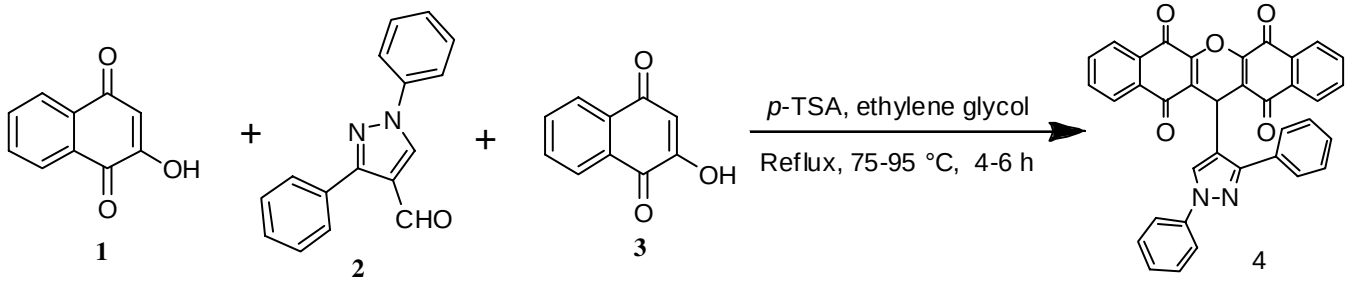
EXPERIMENTAL

The chemicals were purchased from Sigma-Aldrich USA. The reaction was monitored by TLC using precoated aluminium sheets of silica gel 60 F_{254} (Merck, Germany) of 0.2 mm thickness. Column chromatography were performed with silica gel of mesh size (230-400 Merck).

^{1}H NMR and ^{13}C NMR was recorded on a Bruker (300 MHz and 75 Hz) spectrometer using $CDCl_3$ solvent. An FTIR spectrum was recorded on a Perkin-Elmer Spectrometer of the range (4000-400 cm^{-1}) using KBr pellet. The Q-Tof-Mass spectrometer was utilized to measure HRMS.

Synthesis of compounds 4a-j: Pyrazole aldehyde (1 equiv.) and 2-hydroxy-1,4-naphthoquinone (2 equiv.) were refluxed in the presence of *p*-toluenesulfonic acid (PTSA) as catalyst in ethylene glycol (10 mL) at 90 ºC for 5 h. The residue was washed with cold water and stirred for 10 min, a brown precipitate was filtered under suction (**Scheme-I**). The crude product was purified using column chromatography on silica gel with ethylacetate:petroleum ether (50:50)] to afford pure benzoxanthene tetraone.

13-(1,3-Diphenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b*,*i*]-xanthene-5,7,12,14(13*H*)-tetraone (4a): Red solid; R_f = 0.51 (50% ethyl acetate); ^{1}H NMR: (300 MHz, $CDCl_3$, δ ppm): 3.94 (s, 1H), 7.42-7.84 (m, 19H); ^{13}C NMR (75 MHz, $CDCl_3$): 35.0, 117.1, 119.8, 123.1, 126.3, 127.4, 128.75, 129.1, 129.4, 130.9, 131.9, 133.1, 135.1, 139.5, 150.0, 160.7, 178.1, 183.2;



Scheme-I

FT-IR (KBr, ν_{max}, cm^{-1}): 3424, 3336, 3249, 2949, 2870, 2365, 1650, 1516, 1457, 1340; HRMS (ESI) $[M]^+$ *m/z*: 560.14; Anal. calcd. (found) % for $C_{36}H_{20}N_2O_5$: C, 77.14 (77.23); H, 3.60 (3.36); N, 5.00 (4.93); O, 14.27 (14.32).

13-(3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4b): Reddish solid; R_f = 0.46 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 4.17 (s, 1H), 7.26-8.37 (m,18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 40.4, 110.1, 110.9, 119.2, 119.4, 119.6, 122.1, 123.2, 123.7, 127.5, 127.9, 128.9, 129.0, 129.2, 129.7, 130.7, 134.3, 134.6, 134.7, 139.4, 143.07, 146.2, 150.5, 160.0, 180.0, 182.2; FT-IR (KBr, ν_{max}, cm^{-1}): 3433, 3148, 3065, 2929, 1669, 1597, 1510, 1452, 1354, 1249, 1105, 1044, 755, 689, 657; HRMS (ESI) $[M]^+$ *m/z*: 594.10; Anal. calcd. (found) % for $C_{36}H_{19}N_2O_5Cl$: C, 72.67 (71.55); H, 3.22 (3.11); Cl, 5.96 (6.00); N, 4.71 (4.13); O, 13.44 (12.94).

13-(3-(4-Bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4c): Reddish brown solid; R_f = 0.40 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm):3.95 (s, 1H), 7.45-7.78 (m,18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 35.1, 117.4, 120.6, 123.0, 123.1, 126.1, 126.8, 128.3, 129.3, 130.8, 131.8, 132.0, 132.1, 135.0, 139.7, 149.9, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 638.05; Anal. calcd. (found) % for $C_{36}H_{19}N_2O_5Br$: C, 67.62 (67.55); H, 3.00 (2.55); Br, 12.50 (12.30); N, 4.38 (3.99); O, 12.51 (12.11).

13-(3-(4-Methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4d): Red solid; R_f = 0.48 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 3.81 (s, 3H), 3.93 (s, 1H), 7.03 (d, 2H, *J* = 7.61), 7.41-7.74 (m, 16H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 35.1, 117.2, 120.5, 123.0, 123.1, 126.2, 126.8, 128.3, 129.3, 130.8, 131.8, 132.0, 132.1, 135.0, 139.7, 149.9, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083,1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 590.15; Anal. calcd. (found) % for $C_{37}H_{22}N_2O_6$: C, 75.25 (75.05); H, 3.75 (3.60); N, 4.74 (4.50); O, 16.25 (16.11).

13-(3-(4-Ethoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4e): Red solid; R_f = 0.44 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 1.34 (t, 3H *J* = 8.05), 3.96 (s, 1 H), 4.05 (q, 2H, *J* = 7.89), 7.08 (d, 2H, *J* = 7.51), 7.43-7.71 (m, 16H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.8, 35.1, 64.6, 114.9, 117.2, 119.9, 120.5, 123.0, 124.6, 126.2, 126.8, 128.1, 129.3, 130.8, 131.8, 135.0, 139.7, 149.9, 159.4, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 604.16; Anal. calcd. (found) % for $C_{38}H_{24}N_2O_6$: C, 75.49 (75.35); H, 4.00 (3.88); N, 4.63 (4.51); O, 15.88 (15.23).

13-(1-(2,4-Dinitrophenyl)-3-phenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4f): Dark brown solid; R_f = 0.38 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 3.94 (s, 1H), 7.49-7.84 (m, 14 H), 8.13 (d, 1H, *J* = 7.85), 8.75 (d, 1H, *J* = 7.33), 8.81 (d, 1H, *J* = 7.66); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 335.1, 117.2, 120.5, 120.6, 123.0, 124.7, 126.8, 127.5, 128.7, 129.2, 130.8, 131.8, 133.0, 135.0, 136.1, 142.1, 146.3, 149.9, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 650.11; Anal. calcd. (found) % for $C_{36}H_{18}N_4O_9$: C, 66.47 (66.33); H, 2.79 (2.52); N, 8.61 (8.12); O, 22.13 (22.01).

13-(3-(4-Chlorophenyl)-1-(2,4-dinitrophenyl)-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4g): Reddish brown solid; R_f = 0.33 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 3.95 (s, 1H), 7.52-7.98 (m, 13 H), 8.14 (d, 1H, *J* = 7.90), 8.77 (d, 1H, *J* = 7.30), 8.98 (d, 1H, *J* = 7.69); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 35.1, 117.2, 120.5, 120.6, 123.0, 124.7, 126.8, 127.6, 128.9, 129.3, 130.8, 131.1, 131.8, 134.3, 135.0, 136.1, 142.1, 146.3, 149.9, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 684.07; Anal. calcd. (found) % for $C_{36}H_{17}N_4O_9Cl$: C, 63.12 (63.01); H, 2.50 (2.32); Cl, 5.18 (5.03); N, 8.18 (7.89); O, 21.02 (20.98).

13-(3-(4-Bromophenyl)-1-(2,4-dinitrophenyl)-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4h): Dark red solid; R_f = 0.25 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 3.92 (s, 1H), 7.55-7.78 (m, 13 H), 8.11 (d, 1H, *J* = 7.65), 8.65 (d, 1H, *J* = 7.33), 8.81 (d, 1H, *J* = 7.66); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 35.1, 117.2, 120.5, 120.6, 123.0, 123.1, 124.7, 126.8, 127.6, 128.3, 130.8, 131.8, 132.0, 132.1, 135.0, 136.1, 142.1, 146.3, 149.9, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 728.02; Anal. calcd. (found) % for $C_{36}H_{17}N_4O_9Br$: C, 59.28 (59.13); H, 2.35 (2.11); Br, 10.95 (10.14); N, 7.68 (7.28); O, 19.74 (19.34).

13-(1-(2,4-Dinitrophenyl)-3-(4-methoxyphenyl)-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b,i*]xanthene-5,7,12,14(13*H*)-tetraone (4i): red solid; R_f = 0.35 (50% ethyl acetate); m.p.:

250-252 °C; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 3.81 (s, 3H), 3.94 (s, 1H), 7.05 (d, 2 H, *J* = 6.32), 7.59-7.73 (m, 11 H), 8.13 (d, 1H, *J* = 7.66), 8.75 (d, 1H, *J* = 7.58), 8.81 (d, 1H, *J* = 6.96); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 35.1, 55.8, 114.8, 117.2, 120.5, 123.0, 124.7, 125.3, 126.8, 128.5, 130.8, 131.8, 135.0, 136.1, 142.1, 149.9, 160.6, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 680.12; Anal. calcd. (found) % for $C_{37}H_{20}N_4O_{10}$: C, 65.30 (65.12); H, 2.96 (2.56); N, 8.23 (8.13); O, 23.51 (23.48).

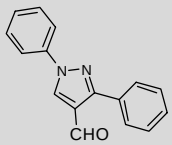
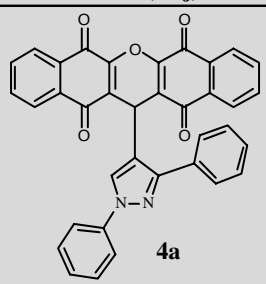
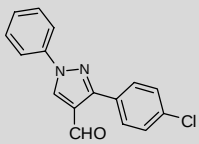
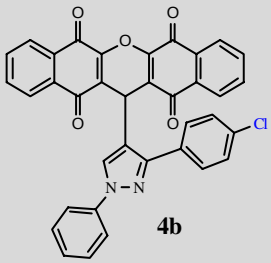
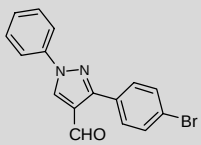
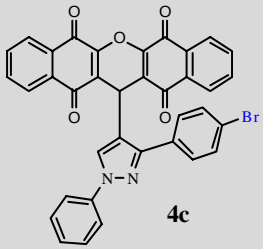
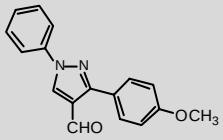
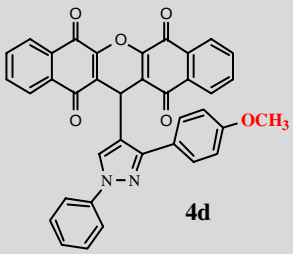
13-(1-(2,4-Dinitrophenyl)-3-(4-ethoxyphenyl)-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b*,*i*]xanthene-5,7,12,14(13*H*)-tetraone (4j): Red solid; R_f = 0.30 (50% ethyl acetate); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 1.34 (t, 3H, 3.34), 3.91 (s, 1H), 4.05 (q, 2H, 12.50), 7.15 (d, 2H, *J* = 6.38), 7.60-7.79 (m, 11 H), 8.17 (d, 1H, *J* = 7.64), 8.62 (d, 1H, *J* = 7.60), 8.85 (d, 1H, *J* = 6.87); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.8, 35.1, 64.6, 114.9, 117.2, 120.5, 120.6, 123.0, 124.7, 126.8, 127.6, 128.1, 130.8, 131.8, 135.0, 136.1, 142.1, 146.3, 149.9, 159.4, 160.8, 178.2, 183.0; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083, 1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 694.13; Anal. calcd. (found) % for $C_{38}H_{22}N_4O_{10}$: C, 65.71 (65.66); H, 3.19 (3.09); N, 8.07 (7.89); O, 23.03 (22.95).

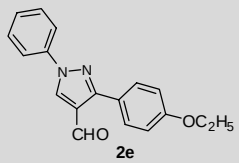
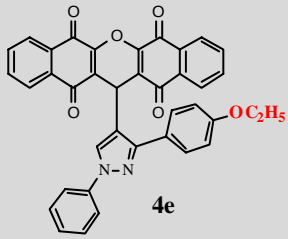
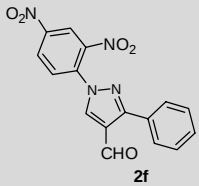
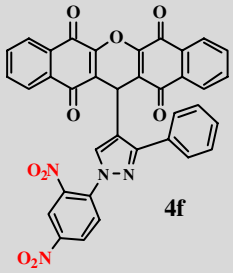
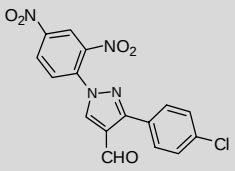
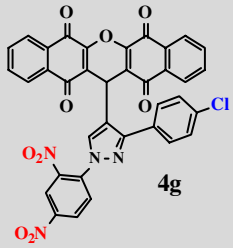
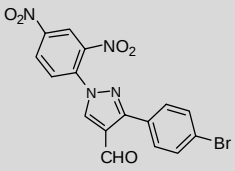
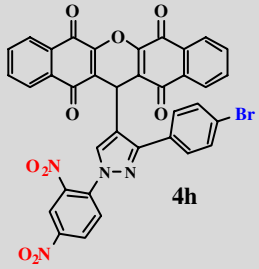
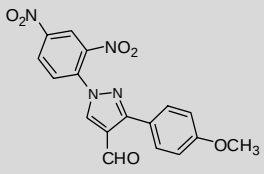
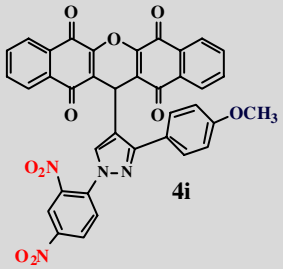
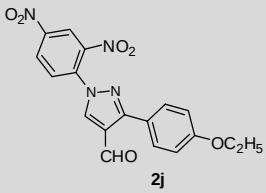
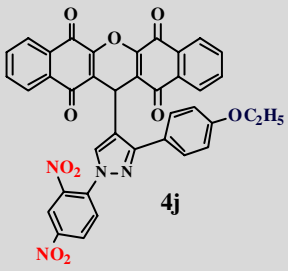
RESULTS AND DISCUSSION

Herein, a new methodology for the synthesis of dibenzoxanthene tetraone is adopted. The best results were obtained while using *p*-TSA catalyst and ethylene glycol solvent. It was found that optimum temperature for the reaction was 90 ºC. Optimizing the reaction conditions, good yields of the desired products (78-90%) were obtained. For the synthesis of 13-(1,3-diphenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo-[*b*,*i*]xanthene-5,7,12,14(13*H*)-tetraone, comparative analysis of temperature, time and yield are shown in Table-1. In aim of optimize the reaction, we carried out the reaction with a variety of catalysts and solvents (Table-2). All the synthesized products were characterized with spectroscopic techniques thoroughly.

The ^{1}H NMR spectrum of compound **4b** exhibited a singlet at 4.17 ppm was attributed to pyran ring proton. The one proton

TABLE-1
SYNTHESIS OF DIBENZOXANTHENE TETRAONE DERIVATIVES

Entry	Pyrazole aldehyde	Product (**4a-j**)	Time (h)	Temp. (°C)	Isolated yield (%)
1	 **2a**	 **4a**	4.0	75	90
2	 **2b**	 **4b**	4.5	78	88
3	 **2c**	 **4c**	5.0	77	91
4	 **2d**	 **4d**	5.5	82	80

5	 **2e**	 **4e**	4.5	80	88
6	 **2f**	 **4f**	6.0	88	82
7	 **2g**	 **4g**	5.5	90	78
8	 **2h**	 **4h**	6.0	92	75
9	 **2i**	 **4i**	5.5	95	85
10	 **2j**	 **4j**	6.0	91	83

singlet at 7.76 ppm was assigned to pyrazole ring proton. The peaks at 7.26-8.37 ppm were attributed to aromatic protons. In the ^{13}C NMR spectra, the peaks range between 110-150 ppm was assigned to aromatic carbon. The peak at 160 ppm was attributed to C-O-C group in xanthene ring. The peaks at 180 ppm and 182 ppm were assigned to four carbonyl carbons in xanthene ring. The mass spectrum revealed the molecular ion peak (M^+) at *m/z* 594. The formation of the product was further confirmed by elemental analysis.

TABLE-2
CATALYSTS SCREENING FOR THE SYNTHESIS OF DIBENZO[*b,i*]XANTHENE-5,7,12,14(13*H*)-TETRAONE USING ETHYLENE GLYCOL

Catalyst	Solvent	Time (h)	Yield (%)
–	Ethanol	24	5
Acetic acid	Ethanol	10	50
$Yb(OTf_3)$	Ethanol	12	55
Boric Acid	Ethanol	11	60
I_2	Ethanol	08	57
CAN	Ethanol	09	40
InCl3	Ethanol	10	35
$ZrOCl_2 \cdot 8H_2O$	Ethanol	8.5	40
PTSA	Ethanol	6	75
PTSA	Ethylene glycol	4-5	78-90

Conclusion

After preliminary examinations, it was found that refluxing pyrazole aldehyde and 2-hydroxy-1,4-naphthalenedione mixture (dissolved in ethylene glycol), in presence of *p*-toluenesulfonic acid (PTSA) catalyst for 4 h at 85 ºC yielded 85% of desired product 13-(1,3-diphenyl-1*H*-pyrazol-4-yl)-12*H*-dibenzo[*b*,*i*]-xanthene-5,7,12,14(13*H*)-tetraone (**4a**). Under similar conditions, the reaction with various pyrazole aldehydes were conducted and obtained the respective 13-aryl-5*H*-dibenzo[*b*,*i*]xanthene-5,7,12,14(13*H*)-tetraones (**4b-j**) in good yields. The current methodology provides a simple, easy reaction and workup procedure using easily available solvents, readily available, low cost catalyst and utilizing the simple equipments.

CONFLICT OF INTEREST

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

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