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p-TSA Catalyzed Synthesis of Tetrahydrobenzo[*g*]quinolines *via* Multicomponent Reaction using Pyrazole Aldehyde, Lawsone, β-Ketoesters and Ammonium Acetate

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An efficient protocol has been developed for the multicomponent synthesis of tetrahydrobenzo[*g*]quinoline-3-carboxylate derivatives using *p*-toluenesulfonic acid (*p*-TSA) as catalyst through one-pot four component condensation reaction of pyrazole aldehyde, Lawsone, β-ketoesters and ammonium acetate in ethanol. The synthesized compounds were characterized by spectroscopic techniques.

Keywords: Tetrahydrobenzoquinoline, Pyrazole aldehydes, Lawsone, *p*-Toluenesulfonic acid.

INTRODUCTION

The multicomponent reactions were the gateway to get a number of different drug-like molecules with different structures. They involve condensation reactions of two or more reactants in a single step to form new products that contain the essential parts of all the reactants by the formation of number of new bonds. The shortest reaction time, minimal effort and limited squander were the added advantages of multicomponent reactions. They involve powerful synthetic strategies, which gives access to big series of compounds with diverse functionalities [1]. Anti-tumor, antioxidant, antimalarial, anticancer, antiproliferative, anti-inflammatory, antiprotozoal, antimicrobial medications, as well as chemotherapeutic substances, have been shown to have enhanced biological activity, when they contain a quinone moiety. Photosensitizing behaviour is exhibited by molecules with lawsone moeity [2]. Tetrahydrobenzo[*g*]quinoline molecules have such a high cytotoxity against human cancer cells [3]. Their neuroprotective properties were the best compared to all other drugs, provided these molecules has no neurotoxicity. This multipotent therapeutic activity of tetrahydrobenzo[*g*]quinoline molecules were proved to cure the Alzheimer's disease efficiently [4]. The pyrazole group has been found to have a wide range of biological activities like antidepressant, anti-inflammatory, anti-obesity, analgesic, antipsychotic, H_2 receptor, antimalarial, antitumoral, anticancer and antifungal [5-8]. Literature revealed the biological properties and many methodologies to synthesize tetrahydrobenzoquinoline [9-22]. Herein, a protocol for the synthesize of tetrahydrobenzo[*g*]quinoline-3-carboxylate by four-component condensation of methyl/ethyl acetoacetate, pyrazole aldehydes and ammonium acetate in ethanol is reported.

EXPERIMENTAL

All the reagents and chemicals were purchased from Merck and Sigma-Aldrich company. The progress of the reaction was monitored using aluminium TLC plates (Silica gel 60 F_{254}, Merck) and the spots were visualized under UV chamber.

^{1}H NMR and ^{13}C NMR were recorded on a Bruker (300 MHz and 75 Hz) spectrometer using $CDCl_3$ solvent. FTIR spectra was recorded on a Perkin Elmer Spectrometer of the range (4000-400 cm^{-1}) using KBr pellet. Thermo Finnigan LCQ Advantage MAX 6000 ESI spectrometer was used to record HRMS. Melting points were determined using open capillaries and are uncorrected.

Synthesis of tetrahydrobenzoquinoline: *p*-Toluenesulfonic acid (*p*-TSA) was added to a mixture of lawsone (2-hydroxy-1,4-naphthoquinone (1 mmol) and ethyl acetoacetate (2 mmol) taken in a single-necked round bottomed flask and was stirred for 10 min at 60 ºC in ethanol. To the clear solution, pyrazole aldehyde (1 mmol) and ammonium acetate (2 mmol) were added and refluxed at 70 ºC for 4 h. The reaction was monitored using TLC. After the complete consumption of the

reactants, the reaction mixture was cooled to room temperature and poured into ice water. The obtained thick brown precipitate was filtered and dried. The crude product was purified by column chromatography to afford pure tetrahydrobenzo[*g*]quinolines (**Scheme-I**).

Ethyl 4-(1,3-diphenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]quinoline-3-carboxylate (4a): Reddish brown solid;R_f = 0.42 (30% EAPE); m.p.: 234-236 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 0.96 (t, 3H, *J* = 6.0 Hz), 3.74 (q, 2H, *J* = 9.0 Hz), 2.33 (s, 3H), 5.60 (s, 1H), 6.9 (s, 1H), 7.22-8.08 (m, 15H); ^{13}C NMR (75 MHz $CDCl_3$) δ: 14.04, 19.40, 27.88, 60.0, 105.54, 118.77, 118.90, 126.10, 126.20, 126.57, 126.76, 127.37, 127.83, 128.10, 129.15, 129.24, 130.13, 132.78, 132.92, 134.31, 134.92, 136.92, 139.88, 142.61, 151.76, 166.87, 179.95, 182.54; FT-IR (KBr, ν_{max}, cm^{-1}): 3429, 3346, 2925, 2856, 1630, 1485, 1352, 1219, 1093,1052, 759, 693; HRMS (ESI) $[M]^+$ *m/z*: 515.18; Anal. calcd. (found) % for $C_{32}H_{25}N_3O_4$: C, 74.55 (74.51); H, 4.89 (4.86); N, 8.15 (8.12); O, 12.41 (12.35).

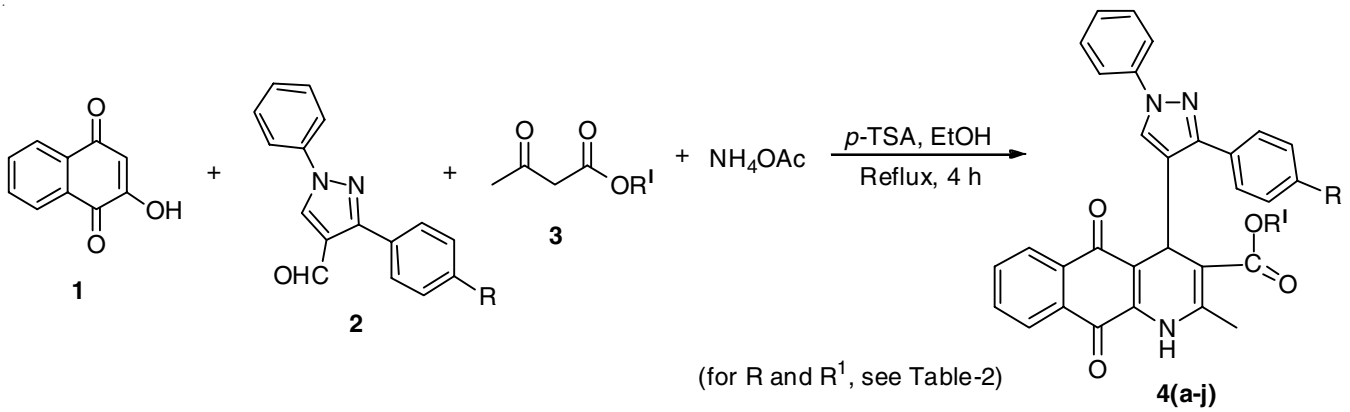
Ethyl 4-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo-[*g*]quinoline-3-carboxylate (4b): Reddish brown solid; R_f = 0.30 (30% EAPE); m.p.: 250-252 ºC; ^{1}H NMR (300 MHz $CDCl_3$) δ: 1.12 (t, 3H, *J* = 7.23 Hz), 3.94 (q, 2H, *J* = 6.8 Hz), 2.28 (s, 3H), 4.75 (s, 1H), 7.67(s, 1H), 7.43-7.99 (m, 9H), 7.60-7.72 (m, 2H), 7.68-7.70 (m, 2H,), 9.06 (s, 1H); ^{13}C NMR (75 MHz $CDCl_3$) δ: 14.11, 18.77, 35.88, 61.2, 102.6, 117.2, 118.68, 118.9, 120.4, 123.5, 126.15, 126.80, 126.81, 127.54, 127.56, 128.7, 129.3, 129.3, 129.4, 129.4, 130.13, 131.78, 134.13, 135.42, 139.7, 144.61, 150.1, 167.87, 178.95, 183.34; FT-IR (KBr, ν_{max}, cm^{-1}): 3431, 3343, 2915, 2856, 1634, 1480, 1344, 1222, 1083,1015, 762, 675; HRMS (ESI) $[M]^+$ *m/z*: 549.15; Anal. calcd. (found) % for $C_{32}H_{24}N_3O_4Cl$: C, 69.88 (69.60); H, 4.40 (4.33); Cl, 6.45 (6.30); N, 7.64 (7.62); O, 11.63 (11.54).

Ethyl 4-(3-(4-bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4c): Red solid; R_f = 0.23 (30% EAPE); m.p.: 268-270 ºC; ^{1}H NMR (300 MHz $CDCl_3$) δ: 1.16 (t, 3H, *J* = 7.22 Hz), 3.67 (q, 2H, *J* = 6.1 Hz), 2.27 (s, 3H), 4.05 (s, 1H), 7.68 (s, 1H), 7.40-7.79 (m, 9H), 7.63 (m, 2H), 7.77 (m, 2H,), 9.08 (s, 1H); ^{13}C NMR (75 MHz $CDCl_3$) δ: 14.2, 18.2, 36.8, 61.8, 102.4, 117.5, 118.6, 118.8, 120.8, 123.3, 126.4, 126.6, 126.6, 127.6, 127.6, 128.5 129.5, 129.5, 129.8, 129.8, 130.2, 132.7, 134.13, 135.5, 139.8, 144.3, 150.6, 167.7, 178.5, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3435, 3340, 2910, 2866, 1654, 1478, 1354, 1220, 1073, 1025, 742, 670; HRMS (ESI) $[M]^+$ *m/z*: 593.10; Anal. calcd. (found) % for $C_{32}H_{24}N_3O_4Cl$: C, 64.66 (64.63); H, 4.07 (4.00); Br, 13.44 (13.37); N, 7.07 (7.03); O, 10.77 (10.74).

Ethyl 4-(3-(4-methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4d): Dark red solid; R_f = 0.35 (30% EAPE); m.p: 252-254 ºC; ^{1}H NMR (300MHz $CDCl_3$) δ: 1.13 (t, 3H, *J* = 7.12 Hz), 2.27 (s, 3H), 3.80 (s, 3H), 4.05 (q, 2H, *J* = 7.05 Hz), 4.78 (s, 1 H), 7.65 (s, 1H), 7.03-7.55 (m, 9H), 7.60 (m, 2H), 7.72 (m, 2H), 9.07 (s, 1H); ^{13}C NMR (75 MHz $CDCl_3$) δ: 14.2, 18.7, 37.8, 55.5, 61.9, 102.5, 114.5, 117.6, 119.8, 120.8, 123.0, 125.3, 126.2, 126.6, 127.6, 127.8, 128.5 129.3, 129.5, 129.8, 130.2, 131.7, 134.9, 135.0, 139.7, 144.0, 150.7, 160.6, 167.7, 178.5, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3435, 3340, 2910, 2866, 1654, 1478, 1354, 1220, 1073,1025, 742, 670; HRMS (ESI) $[M]^+$ *m/z*: 545.20; Anal. calcd. (found) % for $C_{33}H_{27}N_3O_5$: C, 72.65 (72.62); H, 4.99 (4.95); N, 7.70 (7.64); O, 14.66 (14.62).

Ethyl 2-methyl-4-(3-(4-nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4e): Red solid; R_f = 0.28 (30% EAPE); m.p.: 258-260 ºC; ^{1}H NMR (300MHz $CDCl_3$) δ: 1.11 (t, 3H, J = 7.32 Hz), 2.27 (s, 3H), 4.1 (q, 2H, *J* = 7.05 Hz), 4.88 (s, 1 H), 7.63 (s, 1H), 7.03-7.55 (m, 9H), 7.62 (m, 2H), 7.75 (m, 2H,), 9.09 (s, 1H); ^{13}C NMR (75 MHz $CDCl_3$) δ: 14.4, 18.5, 37.5, 61.9, 102.9, 117.5, 119.9, 120.7, 123.0, 124.4, 124.7, 126.3, 126.4, 126.6, 126.9, 129.3, 129.5, 130.2, 131.9, 135.6, 135.7, 139.8, 144.4, 148.3, 150.7, 167. 7, 178.5, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3433, 3338, 2908, 2864, 1644, 1488, 1350, 1222, 1075, 1021, 740, 669; HRMS (ESI) $[M]^+$ *m/z*: 560.17; Anal. calcd. (found) % for $C_{32}H_{24}N_4O_6$: C, 68.56 (68.52); H, 4.32 (4.30); N, 9.99 (9.96); O, 17.12 (17.09).

Methyl 4-(1,3-diphenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]quinoline-3-carboxylate (4f): Dark red solid; R_f = 0.45 (30% EAPE); m.p.: 222-224 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.27 (s, 3H), 3.61 (s, 3H), 4.78 (s, 1 H),7.65 (s, 1H), 7.43-7.85 (m, 10H), 7.63 (m, 2H), 7.70 (m, 2H,), 9.07 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.4, 18.5, 37.5, 61.9, 102.9, 117.5, 119.9, 120.7, 123.0,



Scheme-I

124.4, 124.7, 126.3, 126.4, 126.6, 126.9, 129.3, 129.5, 130.2, 131.9, 135.6, 135.7, 139.8, 144.4, 148.3, 150.7, 167. 7, 178.5, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3428, 3330, 2905, 2860, 1645, 1485, 1344, 1228, 1070, 1025, 745, 677; HRMS (ESI) $[M]^+$ *m/z*: 501.17; Anal. calcd. (found) % for $C_{31}H_{23}N_3O_4$: C, 74.24 (74.21); H, 4.62 (4.65); N, 8.38 (8.26); O, 12.76 (12.56).

Methyl 4-(3-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4g): Reddish brown solid; R_f = 0.38 (30% EAPE); m.p.: 229-234 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.20 (s, 3H), 3.64 (s, 3H), 4.88 (s, 1 H), 7.69 (s, 1H), 7.50-7.98 (m, 9H), 7.60 (m, 2H), 7.75 (m, 2H,), 9.10 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 18.6, 37.3, 52.4, 104.8, 117.0, 120.1, 120.9, 123.6, 126.1, 126.3, 126.8, 126.9, 129.3, 129.5, 130.2, 131.9, 134.5, 135.0, 135.1, 139.5, 144.4, 149.3, 150.7, 167. 7, 178.5, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3440, 3326, 2915, 2850, 1655, 1479, 1334, 1218, 1089, 1019, 749, 687; HRMS (ESI) $[M]^+$ *m/z*: 535.17; Anal. calcd. (found) % for $C_{31}H_{22}N_3O_4Cl$: C, 69.47 (69.43); H, 4.14 (4.11); Cl, 6.61 (6.55); N, 7.84 (7.80); O, 11.94 (11.92).

Methyl 4-(3-(4-bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4h): Dark red solid; R_f = 0.26 (30% EAPE); m.p. 258-263 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.26 (s, 3H), 3.65 (s, 3H), 4.80 (s, 1 H), 7.64 (s, 1H), 7.55-7.78 (m, 9H), 7.62 (m, 2H), 7.73 (m, 2H,), 9.08 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 18.5, 37.5, 52.1, 104.5, 117.3, 119.8, 120.4, 123.0, 123.1, 126.2, 126.3, 126.7, 126.8, 129.3, 129.4, 130.5, 131.7, 132.0, 132.1, 135.0, 135.1, 139.8, 144.1, 149.4, 150.1, 167. 5, 178.4, 183.1; FT-IR (KBr, ν_{max}, cm^{-1}): 3435, 3329, 2924, 2853, 1656, 1482, 1342, 1225, 1092, 1021, 753, 691; HRMS (ESI) $[M]^+$ *m/z*: 579.08; Anal. calcd. (found) % for $C_{31}H_{22}N_3O_4Br$: C, 64.15 (64.11); H, 3.82 (3.80); Br, 13.77 (13.71); N, 7.24 (7.21); O, 11.03 (11.00).

Methyl 4-(3-(4-methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl)-2-methyl-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4i): Reddish brown solid; R_f = 0.40 (30% EAPE); m.p.: 225-227 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.22 (s, 3H), 3.61 (s, 3H), 3.82 (s, 3H), 4.88 (s, 1 H), 7.63 (s, 1H), 7.50-7.69 (m, 9H), 7.63 (m, 2H), 7.75 (m, 2H,), 9.04 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 18.8, 37.4, 52.5, 104.1, 114.6, 114.7, 117.4, 119.7, 119.8, 120.5, 123.2, 126.3, 126.7, 128.4, 128.5, 129.4, 129.5, 130.2, 131.8, 135.1, 135.2, 139.8, 144.4, 149.6, 160.1, 167.6, 178.6, 183.4; FT-IR (KBr, ν_{max}, cm^{-1}): 3430, 3325, 2920, 2855, 1656, 1480, 1340, 1224, 1090, 1020, 755, 695; HRMS (ESI) $[M]^+$ *m/z*: 531.18; Anal. calcd. (found) % for $C_{32}H_{25}N_3O_5$: C, 72.31 (72.28); H, 4.74 (7.41); N, 7.91 (7.88); O, 15.05 (15.02).

Methyl 2-methyl-4-(3-(4-nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl)-5,10-dioxo-1,4,5,10-tetrahydrobenzo[*g*]-quinoline-3-carboxylate (4j): Red solid; R_f = 0.33 (30% EAPE); m.p.: 248-250 ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.25 (s, 3H), 3.62 (s, 3H), 4.80 (s, 1 H), 7.65 (s, 1H), 7.52-7.68 (m, 9H), 7.60 (m, 2H), 7.72 (m, 2H,), 9.08 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 18.5, 37.5, 52.6, 104.4, 117.6, 119.7, 119.8, 120.7, 123.0, 124.5, 124.6, 126.2, 126.7, 126.8, 129.2, 129.3, 130.0, 131.6, 135.2, 135.3, 139.2, 139.8, 144.0, 147.6, 149.6, 149.9, 167.4, 178.3, 183.5; FT-IR (KBr, ν_{max}, cm^{-1}): 3428, 3323, 2925, 2850, 1655, 1485, 1343, 1222, 1091, 1022, 750, 690; HRMS (ESI) $[M]^+$ *m/z*: 546.15; Anal. calcd. (found) % for $C_{31}H_{22}N_4O_6$: C, 68.13 (68.10); H, 4.06 (4.03); N, 10.25 (10.21); O, 17.56 (17.20).

RESULTS AND DISCUSSION

With interest in multicomponent reactions, herein, a method is developed for the synthesis of tetrahydrobenzoquinoline derivatives. This strategy begins with multicomponent reaction of 2-hydroxy-1,4-naphthoquinone and ethyl acetoacetate in ethanol at room temperature. To a above mixture, pyrazole aldehyde and ammonium acetate were added. The reaction mixture was refluxed for 4 h at 70 ºC and the crude product was purified using column chromatographic technique. About 85% of yield could be obtained in this methodology.

In order to understand the reactivity of the reagent, and to improve the yield of the products, we have undertaken an optimization study for the synthesis of compound **4a**. Thus, reaction parameters such as temperature, time and catalysts were considered. Amidst the catalysts used, *p*-TSA is found to be the most efficient. Various solvents were used and found that in ethanol, all the reactants got dissolved uniformly and the reaction went on smoothly. From room temperature, the temperature was gradually raised. At lower temperatures, the yield was noticed to be very low. The maximum yield of 85% was obtained on reaching 70 ºC, above which there is no noticeable increase of the yield. When the reaction mixture was refluxed, the reaction time was found to be minimized (Table-1). The optimal conditions for the four components reaction was surged for different pyrazole aldehyde also and the results are given in Table-2.

TABLE-1
OPTIMIZATION OF THE REACTION CONDITIONS

Entry	Catalyst	Solvent	Temp. (°C)	Time (h)	Yield (%)
1	$Yb(OTf)_3$	EtOH	50	12	30
2	$InCl_3$	EtOH	50	10	20
3	CAN	EtOH	50	11	30
4	I_2	EtOH	50	10	30
5	*p*-TSA	EtOH	50	8	50
6	*p*-TSA	THF	50	8	30
7	*p*-TSA	CH_3CN	50	8	30
8	*p*-TSA	H_2O	50	8	50
9	*p*-TSA	EtOH + H_2O	50	8	70
10	*p*-TSA	Neat	60	10	15
11	*p*-TSA	EtOH	R.T	10	5
12	*p*-TSA	EtOH	40	10	55
13	*p*-TSA	EtOH	70	4	85

Reagents and condition: 2-Hydroxy-1,4-naphthoquinone (**1**) (1 mmol), pyrazole aldehyde (**2**) (1 mmol), ethylacetoacetate (**3**) (2 mmol) and ammonium acetate (2 mmol).

The ^{1}H NMR spectrum of compound **4a** exhibited a three proton triplet at 0.96 ppm (J = 6.0 Hz) and two proton quartet at 3.74 ppm (J = 9.0 Hz) was assigned to ethyl group. The three proton singlet at 2.33 ppm was assigned to methyl group. The peak at 5.60 ppm was assigned to pyridine ring proton.

TABLE-2
SYNTHESIS OF TETRAHYDROBENZOQUINOLINE CARBOXYLATE DERIVATIVES

Entry	R	R^1	Compound (**4a-j**)[a]	Time (h)	Yield[b] (%)
1	H	Et	**4a**	4.0	85
2	Cl	Et	**4b**	5.0	83
3	Br	Et	**4c**	4.5	80
4	OMe	Et	**4d**	4.5	85
5	NO_2	Et	**4e**	7.0	70
6	H	Me	**4f**	4.0	85
7	Cl	Me	**4g**	5.5	82
8	Br	Me	**4h**	4.5	80
9	OMe	Me	**4i**	4.5	85
10	NO_2	Me	**4j**	6.0	72

[a]All products were characterized by FTIR, ^{1}H NMR, ^{13}C NMR and HRMS. [b]Isolated yield.

The peak at 6.9 ppm was attributed to N-H proton. The peaks at 7.22-8.08 ppm were attributed to aromatic protons. In the ^{13}C NMR spectrum, the peaks the ranging between 105-151 ppm was assigned to aromatic carbon. The peak at 19.4 ppm was assigned to methyl group carbon. The peak at 27.8 ppm was attributed to pyridine ring carbon, while the peak at 166 ppm was attributed to carbonyl carbon in ethyl acetate group. The peaks at 179.9 ppm and 182.5 ppm were assigned to the carbonyl carbons in quinazolinone ring. The mass spectrum revealed the molecular ion peak (M^+) at *m/z* 515. The formation of the product was further confirmed by elemental analysis.

Conclusion

An efficient protocol for the synthesis of new tetrahydrobenzoquinoline using low cost, easily available *p*-toluenesulfonic acid (*p*-TSA) catalyst through one pot four components reaction of pyrazole aldehyde, lawsone, 1,3-dicarbonyl compound and ammonium acetate using the solvent ethanol under reflux condition within a short interval of time with remarkable high yield. This current procedure offers a simple operation conditions and easy workup procedures. The synthesized compound contains many biologically active moieties. On the whole, we report a explicit technique for the synthesis of tetrahydrobenzo-[*g*]quinoline-3-carboxylate by the conventional method.

CONFLICT OF INTEREST

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

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