

Synthesis of Naphthopyranopyrimidines Using Formic Acid as an Effective Catalyst under Solvent-free Conditions

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(Received 23 February 2018, Accepted 10 May 2018)

An environmentally friendly, one-pot, three-component synthesis of 8,10-dimethyl-12-aryl-12*H*-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-diones was achieved *via* coupling aromatic of aldehydes, β -naphthol and 6-amino-1,3-dimethyl uracil in the presence of formic acid as a catalyst under solvent-free conditions at 90 °C. This method has several advantages, such as efficiency, good yield, short reaction time, cleaner reaction profiles, ease of product isolation, operational simplicity and inexpensive, effective and catalyst.

Keywords: Naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine, Aldehyde, β -Naphthol, 6-Amino-1,3-dimethyl uracil, Formic acid

INTRODUCTION

Naphthopyranopyrimidines represent an important class of organic compounds with diverse biological activities such as analgesic [1], fungicidal [2,3] antitumor [4,5], hypolipidemic [6], molluscicidal [7] and antifungal [8-11]. These compounds indicate promising physiological [12], anticonvulsant behavior [13] and hypotensive effect [14]. Recently, the biological activity of these molecules has been reported (Fig. 1) as antagonists for neuropeptide S receptor (NPSR) that displays the role of a novel drug targets for the treatment of sleep, anxiety, and addiction disorders [14]. Because of the pharmacological and biological importance of *N*- and *O*-heterocycles as drug compounds, some synthetic approaches including multi-component strategies have been developed for the synthesis of naphthopyranopyrimidine derivatives [15,16].

The pollution of nature is a serious problem in the design of chemical reactions. "Green chemistry" encourages chemists to modify the chemical processes until decrease generation and consumption of dangerous substances in chemical reactions [17].

In recent years, multi-component reactions (MCRs)

have appeared as highly notable synthetic techniques for the rapid production of structurally various and complex heterocyclic compounds [18]. These reactions present a considerable number of advantages beyond conventional multi-step synthesis including great degree of atom economy, simplicity of execution, formation of several new bonds in a one-pot reaction, reduction in the issue of work-up steps, excellent yields and extraction and purification of operations [19].

Literature reveals only a few methods for the synthesis of naphthopyranopyrimidines *via* multi-component reaction of β -naphthol, 1,3-dimethylbarbituric acid and diverse aromatic aldehydes catalyzed by InCl_3 [20], Heteropolyacid (HPA) [21], $\text{Al}(\text{H}_2\text{PO}_4)_3$ [22] and L-proline [23]. As a portion of our continued interest in the synthesis of various heterocyclic compounds of biological importance [24-28], particularly diverse heterocycle fused uracil derivatives, we report herein a simple, efficient and green method for the synthesis of naphthopyranopyrimidines using formic acid as a green catalyst.

EXPERIMENTAL

Materials and Methods

Melting points and IR spectra of all compounds were

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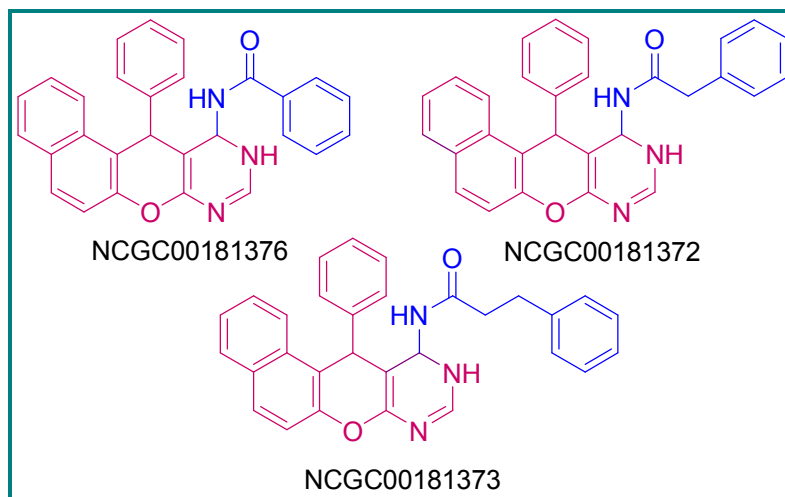


Fig. 1. A few naphthopyranopyrimidine-based biologically active molecules.

quantified on an Electro thermal 9100 apparatus and FT-IR-JASCO-460 plus spectrometer, respectively. ^1H NMR and ^{13}C NMR spectra of some compounds were recorded on a Bruker DRX-400 Avance instrument in DMSO at 400, and 100 MHz. Mass spectra were taken on an Agilent Technology (HP) spectrometer operating at an ionization potential of 70 eV. All reagents were purchased from Merck (Darmstadt, Germany) and Fluka (Buchs, Switzerland) and used without further purification.

General Procedure for Preparation of 8,10-Dimethyl-12-aryl-12H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-diones

A mixture of aldehyde 1 (1.0 mmol), β -naphthol 2 (1.0 mmol), 6-amino-1,3-dimethyluracil 3 (1 mmol) and formic acid (0.2 ml) was stirred at 90 °C under solvent-free conditions. The completion of reaction was monitored by TLC. The crude reaction mixture was washed with ethanol (5 ml), and then precipitate was filtered to separate the product. Then, the crude product was recrystallized from ethanol to obtain the pure product.

Characterization Data of Selected Compound

12-Phenyl-8,10-dimethyl-12H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-dione (4a). White solid; IR (KBr, cm^{-1}): 3105, 2950, 1704, 1665, 1643, 1592, 1484, 1231, 1178; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.16 (s,

3H, CH_3), 3.51 (s, 3H, CH_3), 5.67 (s, 1H, CH), 7.08 (t, J = 7.2 Hz, 1H, Ar-H), 7.19 (t, J = 7.2 Hz, 2H, Ar-H), 7.34 (d, J = 7.8 Hz, 2H, Ar-H), 7.46 (t, J = 7.4 Hz, 1H, Ar-H), 7.51 (t, J = 7.0 Hz, 1H, Ar-H), 7.08 (d, J = 8.8 Hz, 1H, Ar-H), 7.95 (d, J = 7.8 Hz, 1H, Ar-H), 8.00 (d, J = 9.2 Hz, 1H, Ar-H), 8.03 (d, J = 8.4 Hz, 1H, Ar-H).

12-(3-Nitrophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione (4i). White solid; IR (KBr, cm^{-1}): 3103, 2951, 1705, 1663, 1639, 1596, 1479, 1232, 1177; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.17 (s, 3H, CH_3), 3.54 (s, 3H, CH_3), 5.93 (s, 1H, CH), 7.50-7.54 (m, 3H, Ar-H), 7.63 (d, J = 7.8 Hz, 1H, Ar-H), 7.70 (d, J = 8.8 Hz, 1H, Ar-H), 7.99-8.23 (m, 4H, Ar-H), 8.76 (d, J = 8.4 Hz, 1H, Ar-H).

12-(3-Hydroxyphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione (4k). White solid; IR (KBr, cm^{-1}): 3412, 3100, 2945, 1697, 1666, 1639, 1585, 1486, 1234, 1179; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.19 (s, 3H, CH_3), 3.53 (s, 3H, CH_3), 5.61 (s, 1H, CH), 6.49 (d, J = 8.0 Hz, 1H, Ar-H), 6.69 (s, 1H, Ar-H), 6.75 (dd, J = 7.4 Hz, J = 1.2 Hz, 1H, Ar-H), 6.99 (t, J = 8.0 Hz, 1H, Ar-H), 7.50 (t, J = 6.8 Hz, 1H, Ar-H), 7.55 (t, J = 7.8 Hz, 1H, Ar-H), 7.65 (d, J = 9.2 Hz, 1H, Ar-H), 7.98 (d, J = 7.6 Hz, 1H, Ar-H), 8.01-8.04 (m, 2H, Ar-H), 9.27 (s, 1H, OH).

12-(4-Methylphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-

dione (4l). White solid; IR (KBr, cm^{-1}): 3139, 2956, 2922, 1709, 1673, 1649, 1596, 1485, 1227, 1178; ^1H NMR (400 MHz), (DMSO- d_6): δ = 2.16 (s, 3H, CH_3), 3.17 (s, 3H, CH_3), 3.53 (s, 3H, CH_3), 5.65 (s, 1H, CH), 7.00 (d, J = 8.0 Hz, 2H, Ar-H), 7.23 (d, J = 8.0 Hz, 2H, Ar-H), 7.46-7.55 (m, 2H, Ar-H), 7.63 (d, J = 8.8 Hz, 1H, Ar-H), 7.95-8.05 (m, 3H, Ar-H).

12-(4-Methoxyphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10H)-dione (4m). White solid; IR (KBr, cm^{-1}): 3121, 3024, 2956, 1708, 1668, 1645, 1595, 1485, 1225, 1177; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.18 (s, 3H, CH_3), 3.53 (s, 3H, CH_3), 3.64 (s, 3H, OCH_3), 5.64 (s, 1H, CH), 6.75 (d, J = 8.8 Hz, 1H, Ar-H), 7.25 (d, J = 8.8 Hz, 1H, Ar-H), 7.48 (t, J = 8.0 Hz, 1H, Ar-H), 7.53 (t, J = 7.5 Hz, 1H, Ar-H), 7.63 (d, J = 8.8 Hz, 1H, Ar-H), 7.97 (d, J = 8.4 Hz, 1H, Ar-H), 8.01 (d, J = 8.8 Hz, 1H, Ar-H), 8.04 (d, J = 8.4 Hz, 1H, Ar-H).

12-(4-Cyanophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10H)-dione (4n). White solid; IR (KBr, cm^{-1}): 3079, 2959, 2235, 1708, 1670, 1646, 1598, 1483, 1229, 1180; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.17 (s, 3H, CH_3), 3.53 (s, 3H, CH_3), 5.65 (s, 1H, CH), 7.47-7.55 (m, 2H, Ar-H), 7.58 (d, J = 8.4 Hz, 2H, Ar-H), 7.65 (d, J = 8.8 Hz, 1H, Ar-H), 7.69 (d, J = 7.4 Hz, 2H, Ar-H), 7.98 (d, J = 8.4 Hz, 1H, Ar-H), 8.01 (d, J = 8.0 Hz, 1H, Ar-H), 8.05 (d, J = 9.2 Hz, 1H, Ar-H).

12-(3-Methoxyphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10H)-dione (4r). White solid; IR (KBr, cm^{-1}): 3160, 2959, 1710, 1664, 1646, 1598, 1487, 1230, 1177; ^1H NMR (400 MHz), (DMSO- d_6): δ = 3.18 (s, 3H, CH_3), 3.52 (s, 3H, CH_3), 5.67 (s, 1H, CH), 6.68 (dd, J = 8.0 Hz, J = 2.4 Hz, 1H, Ar-H), 6.84 (d, J = 7.6 Hz, 1H, Ar-H), 6.94 (s, 1H, Ar-H), 7.11 (t, J = 7.6 Hz, 1H, Ar-H), 7.49 (t, J = 7.4 Hz, 1H, Ar-H), 7.63 (d, J = 9.2 Hz, 1H, Ar-H), 7.97 (d, J = 8.4 Hz, 1H, Ar-H), 8.01 (d, J = 8.8 Hz, 1H, Ar-H), 8.06 (d, J = 8.4 Hz, 1H, Ar-H); ^{13}C NMR (100 MHz, DMSO- d_6): δ = 28.25, 29.43, 35.82, 55.34, 90.42, 111.52, 115.34, 117.36, 117.43, 120.85, 124.19, 125.92, 127.92, 129.09, 129.78, 130.06, 130.69, 131.80, 146.25, 147.23, 150.54, 152.73, 159.41, 161.56; MS m/z (%): 193.1 (11), 236.1 (22), 293.1 (100), 400.2 (M^+ ,

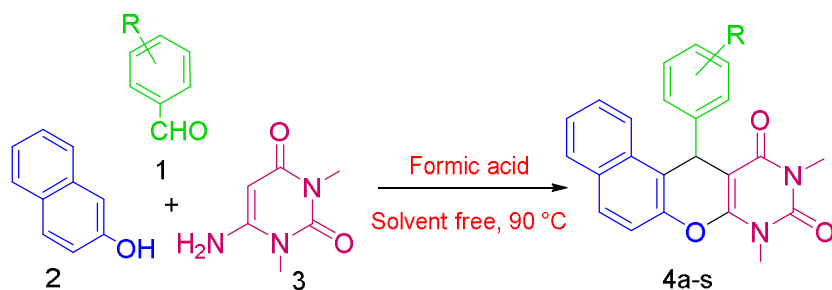
16).

12-(2-Methylphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10H)-dione (4s). White solid; IR (KBr, cm^{-1}): 3152, 3021, 2958, 1704, 1664, 1647, 1596, 1481, 1236, 1174; ^1H NMR (400 MHz), (DMSO- d_6): δ = 2.87 (s, 3H, CH_3), 3.16 (s, 3H, CH_3), 3.52 (s, 3H, CH_3), 5.70 (s, 1H, CH), 6.95-7.11 (m, 4H, Ar-H), 7.47 (t, J = 7.4 Hz, 1H, Ar-H), 7.53 (t, J = 7.0 Hz, 1H, Ar-H), 7.59 (d, J = 8.8 Hz, 1H, Ar-H), 7.79 (d, J = 8.4 Hz, 1H, Ar-H), 7.95 (d, J = 6.8 Hz, 1H, Ar-H), 7.98 (d, J = 8.8 Hz, 1H, Ar-H); ^{13}C NMR (100 MHz, DMSO- d_6): δ = 19.96, 28.27, 29.50, 32.77, 91.04, 117.51, 118.46, 123.57, 125.88, 126.77, 126.82, 127.88, 129.25, 129.80, 129.99, 130.79, 130.93, 131.74, 135.50, 143.54, 147.20, 150.46, 152.70, 161.79; MS m/z (%): 193.1 (10), 236.1 (27), 293.1 (100), 384.2 (M^+ , 17).

RESULTS AND DISCUSSION

In order to optimize the reaction conditions, as a model reaction, the coupling of β -naphthol (1.0 mmol) with benzaldehyde (1.0 mmol) and 6-amino-1,3-dimethyl uracil (1.0 mmol) was studied in the presence of various amounts of formic acid as catalyst ranging 50-100 °C under solvent-free conditions (Scheme 1). The results are summarized in Table 1. As Table 1 demonstrates, the best results were afforded when the reaction was carried out using 0.2 mL catalyst at 90 °C. To confirm that heat cannot thermodynamically perform the reaction in the absence of catalyst, the model reaction was assayed at 100 °C under catalyst-free conditions. In these conditions, the desired product was not gained after 24 h. This observation clearly verified that heat cannot improve the reaction without catalyst. Therefore, the presence of catalyst (*e.g.* formic acid) is essential for the reaction.

The performance and scope of the catalyst were evaluated by the reaction of β -naphthol 2 with divers arylaldehydes 1 and 6-amino-1,3-dimethyl uracil 3 using 0.2 mL of formic acid at 90 °C under the optimized reaction conditions. The corresponding results are indicated in Table 2. As it can be seen in Table 2, all reactions proceeded efficiently to afford the desired 8,10-dimethyl-12-aryl-12H-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-diones (4a-s) in good yields and in short reaction times. Thus, formic



Scheme 1. Formic acid catalyzed one-pot synthesis of naphthopyranopyrimidines

Table 1. Effect of the Catalyst Amount and Temperature on the Reaction between Benzaldehyde, β -Naphthol, and 6-Amino-1,3-dimethyl Uracil

Entry	Time (min)	Catalyst (ml)	Temperature (°C)	Isolated yield (%)
1	24 (h)	None	100	0
2	60	0.05	100	Trace
3	50	0.1	100	65
4	20	0.2	100	90
5	20	0.3	100	88
6	24 (h)	0.2	50	0
7	45	0.2	80	53
8	20	0.2	90	92

acid was efficient catalyst for this one-pot three-component reaction.

The results are summarized in Table 2. As envisaged, aromatic aldehydes including electron-donating and/ or electron-withdrawing groups were applied in this work to create the corresponding desired products in good to excellent yields.

In order to demonstrate the elegant properties of our research, we have compared our results achieved from the synthesis of 12-(4-chlorophenyl)-8,12-dihydro-8,10-dimethyl-9*H*-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10*H*)-dione, catalyzed by formic acid with other results reported in the previous works. As shown in Table 3,

formic acid can be treated as an efficient and green catalyst.

A probable mechanism for the formation of naphthopyranopyrimidines is portrayed supported by the literature [20] (Scheme 2). At first, formic acid activates the carbonyl functional group of aldehyde. In the next step, the reaction continues through the *ortho*-quinone methide intermediate (*o*-QM) A, generated by the nucleophilic addition of β -naphthol 2 to aldehyde 1. This intermediate is also protonated by formic acid to be activated for the next step. Michael addition of 6-amino-1,3-dimethyluracil 3 provides the intermediate B. Finally, naphthopyranopyrimidines 4a-s is synthesized by intramolecular cyclization and removing NH_3 .

Table 2. The Synthesis of Naphthopyranopyrimidines 4a-s Using Formic Acid (0.2 ml) under Solvent-free Conditions at 90 °C

Entry	R	Product	Time (min)	Isolated yield (%) ^a	M.p. (°C)	
					Found	Reported [Ref.]
1	H	4a	20	92	223-225	223-225 [29]
2	2-Cl	4b	35	80	270-273	270-272 [30]
3	3-Cl	4c	30	88	225-228	222-224 [29]
4	4-Cl	4d	20	95	268-270	274-276 [29]
5	2,4-di Cl	4e	40	85	218-220	219-221 [29]
6	4-F	4f	15	95	300-303	303-305 [30]
7	4-Br	4g	20	95	242-245	243-245 [29]
8	2-NO ₂	4h	30	87	281-284	288-290 [30]
9	3-NO ₂	4i	25	93	304-306	310-312 [21]
10	4-NO ₂	4j	25	95	277-280	283-285 [31]
11	3-OH	4k	25	92	295-297	293-294 [20]
12	4-Me	4l	30	85	202-205	200-202 [29]
13	4-OMe	4m	20	93	254-257	257-258 [20]
14	4-CN	4n	20	93	275-278	276-280 [29]
15	2-Naphthyl	4o	30	89	197-200	193-195 [29]
16	2-F	4p	30	87	278-281	287-289 [31]
17	3-Br	4q	28	88	227-230	232-234 [23]
18	3-OMe	4r	35	93	211-213	This work
19	2-Me	4s	40	89	261-263	This work

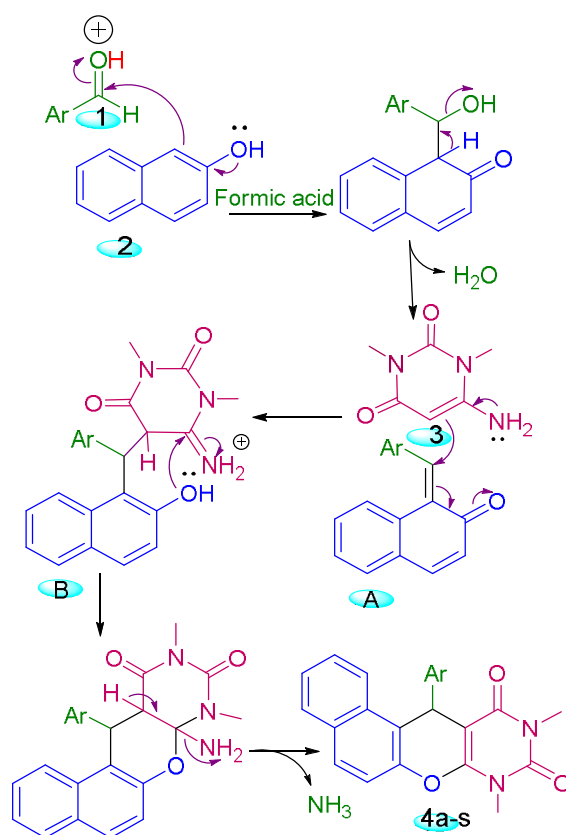
CONCLUSIONS

In summary, the formic acid was explored to be an efficient and green catalyst for the synthesis of naphthopyranopyrimidines through the multi-component reactions of aldehydes, β -naphthol, 6-amino-1,3-dimethyluracil. The reactions were performed under

environmentally benign conditions. The procedure offers several advantages including the use of eco-friendly, commercially available and inexpensive catalyst, good yields, short reaction time, and simple work-up procedure, which make it a useful and attractive protocol for the synthesis of these compounds.

Table 3. Comparison of the Results with the Reported Catalysts in Literature for the Synthesis of 12-(4-Chlorophenyl)-8,12-dihydro-8,10-dimethyl-9*H*-naphtho[1',2':5,6]pyrano[2,3-*d*]pyrimidine-9,11-(10*H*)-dione from the Reaction between 4-Chlorobenzaldehyde, β -Naphthol and 6-Amino-1,3-dimethyl uracil

Entry	Catalyst	Time (min)	Reaction conditions	Yield (%)	[Ref.]
1	InCl ₃	25	120 °C/Solvent free	77	[20]
2	Heteropolyacid (HPA)	26	100 °C/neat	90	[21]
3	Al(H ₂ PO ₄) ₃	40	110 °C/Solvent free	89	[22]
4	L-proline	30	100 °C/Solvent free	95	[23]
5	Formic acid	20	90 °C/Solvent free	95	This work



Scheme 2. The proposed mechanism for the synthesis of naphthopyranopyrimidines using formic acid

ACKNOWLEDGMENTS

Financial support from the Research Council of the University of Sistan and Baluchestan is gratefully acknowledged.

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