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Article

Synthesis of benzo[*g*]indeno[2,1-*b*]quinoline derivatives via four-component and one-pot synthesis in presence of 3-methyl-1-sulfonic acid imidazolium hydrogen sulfate

Nader Ghaffari Khaligh *

Research House of Professor Reza, Education Guilan, Rasht, District 1, 41569-17139, Iran

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ABSTRACT

A simple and facile synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives was accomplished by the one-pot condensation of 2-hydroxynaphthalene-1,4-dione, aryl aldehydes, 2*H*-indene-1,3-dione, and ammonium acetate under solvent-free conditions in the presence of a Brønsted ionic liquid catalyst, namely 3-methyl-1-sulfonic acid imidazolium hydrogen sulfate.

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1. Introduction

The four-component condensation of substituted benzaldehydes with 2-hydroxynaphthalene-1,4-dione, cyclic β -diketones, and ammonium acetate has been widely used as an efficient procedure for synthesizing fused nitrogen-containing heterocycles [1–5]. Azaheterocycles with a quinoline fragment and oxo group are generating much attention, because of their pharmacological properties such as antiasthmatic, anti-inflammatory, antimalarial, anticancer, and anthelmintic actions [6–20]. In addition, these compounds are used as ligands for the preparation of organic light-emitting diode phosphorescent complexes [21–23] and they are used in the preparation of nano- and meso-structures with enhanced electronic and photonic properties [24–26]. Indenoquinoline derivatives have a

wide range of biological activity such as 5-HT-receptor-binding, anti-inflammatory, antitumor, and antimalarial activity, and they also act as steroid reductase and acetylcholinesterase inhibitors [27–33]. Various methods have therefore been developed for the synthesis of substituted quinolines [34–38].

Task-specific ionic liquids (TSILs) are regarded as excellent alternatives to volatile organic solvents, and are used as environmentally benign catalysts and reagents in green synthesis, because of their negligible vapor pressures and non-flammable natures [39,40]. The use of TSIL sulfonic acids to catalyze organic reactions is an area of ongoing activity. These acids have been used as catalysts for Fischer esterification, dehydrocoupling of alcohols, and Pinacol rearrangements [41]. Furthermore, it has been suggested that water makes the proton of the sulfonic acid group more chemically available for reactions. A

* Corresponding author. Tel: +98-2166431738, Fax: +98-2166934046; E-mail: ngkhaligh@gmail.com, ngkhaligh@guilan.ac.ir

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sulfonic acid TSIL was used to catalyze the esterification of acetic acid with 1-heptene and various cyclic olefins [42]. 1-Butyl-3-methylimidazolium hydrogen sulfate was used to promote the one-pot, multicomponent synthesis of 1,8-dioxooctahydroxanthenes and polysubstituted quinolines [43,44].

More recently, an efficient one-pot synthesis of benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives in the presence of poly(4-vinylpyridinium) hydrogen sulfate [P(4-VPH)HSO₄] as a solid acid, under solvent-free conditions, was reported [45]. In this paper, in a continuation of our recent interest in the synthesis of heterocyclic compounds [46,47], we describe the synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives under solvent-free conditions in the presence of 3-methyl-1-sulfonic acid imidazolium hydrogen sulfate (MSAIm)HSO₄.

2. Experimental

2.1. Materials

Unless specified, all chemicals were analytical grade and purchased from the Merck, Aldrich, and Fluka chemical companies, and used without further purification. The products were characterized based on their physical constants and by comparison with authentic samples. The determination of substrate purity and reaction monitoring were performed using thin-layer chromatography (TLC) with silica-gel SIL G/UV 254 plates.

2.2. Instrumentation

The product purities were determined by gas chromatography-mass spectrometry (GC-MS), using an Agilent GC-Mass-6890 instrument operated at 70 eV. Infrared (IR) spectra were recorded with a Perkin Elmer 781 spectrophotometer, using KBr pellets for solid samples and neat liquid samples, in the range 4000–400 cm⁻¹. ¹H nuclear magnetic resonance (NMR) spectra were recorded using a Bruker Avance 300 MHz instrument. All chemical shifts are quoted in parts per million (ppm) relative to tetramethylsilane, and deuterated solvents were used. Mass spectra were recorded using a PESCiex model API 3000 instrument. Microanalyses were performed using a Vario El III CHNOS elemental analyzer. Melting points were determined, using a Büchi B-545 apparatus, in open capillary tubes.

2.3. Typical procedure for preparation of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives (**4**)

(MSAIm)HSO₄ (10 mg) [48] was added to a mixture of 2-hydroxynaphthalene-1,4-dione (**1**; 2 mmol), an aromatic aldehyde (**2**; 2 mmol), 2*H*-indene-1,3-dione (**3**; 2 mmol), and ammonium acetate (3 mmol). The mixture was vigorously mixed for an appropriate time at 55 °C under solvent-free conditions (Table 1). When THE reaction mixture was solid, the substrates and Brönsted acid ionic liquid were ground in a

Table 1

Optimization of synthesis of benzo[*g*]indeno[2,1-*b*]quinoline derivatives in presence of (MSAIm)HSO₄.

Entry	Solvent	Amount of (MSAIm)HSO ₄ (mg)	Temperature (°C)	Time (min)	Yield* (%)
1	CH ₂ Cl ₂	20	r.t.	120	34
2	CH ₂ Cl ₂	20	reflux	120	52
3	CH ₃ CN	20	r.t.	120	54
4	CH ₃ CN	20	reflux	120	62
5	CH ₃ OH	20	r.t.	120	49
6	CH ₃ OH	20	reflux	120	67
7	C ₂ H ₅ OH	20	r.t.	120	55
8	C ₂ H ₅ OH	20	reflux	120	68
9	H ₂ O	20	r.t.	120	32
10	H ₂ O	20	reflux	120	59
11	solvent-free	20	r.t.	120	72
12	solvent-free	20	55	35	82
13	solvent-free	10	55	40	82
14	solvent-free	5	55	40	77
15	solvent-free	10	90	35	82
16	solvent-free	20	90	35	83

Reaction conditions: Dimedone (1 mmol); 4-chlorobenzaldehyde (1 mmol); ethyl acetoacetate (1 mmol); ammonium acetate (1 mmol).

* GC-MS yield.

porcelain dish with a pestle at 55 °C. After completion of the reaction (monitored by TLC), the residue was diluted with EtOAc (3 × 5 mL) and the catalyst was recovered (the Brönsted acidic ionic liquid catalyst was not soluble in this solvent). The solvent was evaporated at reduced pressure and the crude solid product was crystallized from EtOH/H₂O or purified using silica-gel column chromatography. The recovered catalyst was washed with EtOAc (5 mL), dried at ambient temperature, and reused for the next run.

2.4. Biodegradability of 3-methyl-1-sulfonic acid imidazolium hydrogen sulfate

A solution containing (MSAIm)HSO₄ (2 mg/L) was added to an aerobic aqueous medium inoculated with wastewater microorganisms, and the well-mixed solution was carefully dispensed into a series of biochemical oxygen demand (BOD) bottles; all the bottles were completely full. A control with inoculum but without (MSAIm)HSO₄ was run in parallel for the determination of oxygen blanks. Duplicate samples of each series were analyzed immediately for dissolved oxygen and the remaining samples were incubated at (20 ± 1) °C in the dark. The depletion of dissolved molecular oxygen was measured over a 28 d incubation period, and reported as a percentage of the theoretical maximum. Sodium *n*-dodecyl sulfate was used as a reference material. The biodegradation was expressed as the ratio of the BOD to the chemical oxygen demand (COD), both expressed as mg of O₂ per mg of compound. The COD was measured using the dichromate reflux method [49] and the BOD was calculated by dividing the determined oxygen depletion by the concentration of (MSAIm)HSO₄.

2.5. Melting point, spectral properties (IR, ¹H NMR, and Mass),

and elemental analysis of some products

12-(4-Chlorophenyl)-12-hydro-5*H*-benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione (**4b**). Orange powder, m.p. < 320 °C; IR (KBr): $\nu_{\max}$ = 3410, 3028, 1670, 1633, 1610 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ = 5.39 (s, 1H, CH), 7.23–7.44 (m, 6H, ArH), 7.52–7.84 (m, 3, ArH), 7.96 (d, J = 8.4 Hz, 1H, ArH), 8.10–8.15 (m, 2H, ArH), 10.21 (br s, 1H, NH); MS (m/z , %): 423 (M^+ , 12), 312 (100); Anal. Calcd (%) for $\text{C}_{26}\text{H}_{14}\text{NClO}_3$: C 73.76, H 3.31, N 3.31; Found (%): C 73.69, H 3.28, N 3.35.

12-(4-Methoxyphenyl)-12-hydro-5*H*-benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione (**4g**). Brown powder, m.p. 315–317 °C; IR (KBr): $\nu_{\max}$ = 3415, 3028, 1663, 1653, 1608, 1235, 1010 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ = 3.76 (3H, s, CH₃), 5.27 (1H, s, CH), 7.28–7.70 (m, 10H, ArH), 8.08–8.11 (m, 2H, ArH), 10.35 (br s, 1H, NH); MS (m/z , %): 419 (M^+ , 40), 312 (70); Anal. Calcd (%) for $\text{C}_{27}\text{H}_{17}\text{NO}_4$: C 77.32, H 4.06, N 3.34; Found (%): C 77.27, H 3.98, N 3.41.

12-(4-Nitrophenyl)-12-hydro-5*H*-benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione (**4i**). Yellow powder, m.p. < 320 °C; IR (KBr): $\nu_{\max}$ = 3380, 3075, 1665, 1635, 1590 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ = 6.08 (s, 1H, CH), 7.45–7.69 (m, 6H, ArH), 7.76–7.94 (m, 4H, ArH), 8.08 (d, J = 8.8 Hz, 1H, ArH), 8.17 (d, J = 7.6 Hz, 1H, ArH), 10.40 (br s, 1H, NH); MS (m/z , %): 434 (M^+ , 35), 312 (75); Anal. Calcd (%) for $\text{C}_{26}\text{H}_{14}\text{N}_2\text{O}_5$: C 71.89, H 3.22, N 5.53; Found (%): C 71.81, H 3.16, N 5.61.

3. Results and discussion

The reaction conditions were optimized using the conden-

sation of 2-hydroxynaphthalene-1,4-dione (**1**), 4-chlorobenzaldehyde (**2b**), 2*H*-indene-1,3-dione (**3**), and ammonium acetate in ratios of 1:1:1.5 equivalents as a model reaction. Parameters such as the amount of ionic liquid catalyst, i.e., [MSAIm]HSO₄ (5, 10, and 20 mg), and various temperatures (room temperature and reflux) in different solvents (CH_2Cl_2 , CH_3CN , MeOH, EtOH, and H_2O) and in the absence of solvent at room temperature, 55, and 90 °C were investigated. An optimum yield of 82% was obtained in the presence of 10 mg of [MSAIm]HSO₄ at 55 °C under solvent-free conditions after 35 min (Scheme 1). The poor results obtained in solvents compared with that under solvent-free condition can be explained by competitive interactions of the solvent molecules with the active sites of the catalyst and decreased diffusion of the substrates in the presence of a solvent.

The optimized conditions were then used for the conversion of various aromatic aldehydes (**2**) to the corresponding 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives **4a–4j** (Table 2). The results show that the presence of electron-withdrawing and electron-donating substituents on the aromatic ring of the aldehyde have a slight influence on the reaction rate and yield. The rate and product yield decreased in the presence of the –OMe group in 4-methoxybenzaldehyde, as a result of the reduction in the electrophilicity of the carbonyl carbon through resonance (Table 2, entry 7), whereas the –NO₂ group, a strong electron-withdrawing group, in 4-nitrobenzaldehyde significantly increased the reaction rate and yield (Table 2, entry 9). However, a slower rate and lower yield of 12-(2-nitrophenyl)benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione was observed, as a result of intramolecular interactions and

Scheme 1. Synthesis of 12-(4-chlorophenyl)-12-hydro-5*H*-benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives in presence of P(4-VPH)HSO₄ under solvent-free condition.

Table 2

Synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives in presence of [MSAIm]HSO₄ or P(4-VPH)HSO₄ under similar conditions.

Entry	Substrate 2	Product 4	[MSAIm]HSO ₄		P(4-VPH)HSO ₄		m.p. (°C)
			Time (min)	Yield ^a (%)	Time (min)	Yield ^a (%)	
1	C ₆ H ₅ -CHO	4a	40	85	50	86	316–318
2	4-Cl-C ₆ H ₄ -CHO	4b	35	82	47	84	> 320
3	2-Cl-C ₆ H ₄ -CHO	4c	35	78	45	80	> 320
4	4-Br-C ₆ H ₄ -CHO	4d	40	80	50	82	> 320
5	4-F-C ₆ H ₄ -CHO	4e	38	79	48	81	310–312
6	4-CH ₃ -C ₆ H ₄ -CHO	4f	40	76	50	79	> 320
7	4-CH ₃ O-C ₆ H ₄ -CHO	4g	52	66	62	64	> 320
8	2-CH ₃ O-C ₆ H ₄ -CHO	4h	62	68	72	67	311–313
9	4-NO ₂ -C ₆ H ₄ -CHO	4i	25	86	35	89	> 320
10	2-NO ₂ -C ₆ H ₄ -CHO	4j	30	85	40	85	> 320

Reaction conditions: 2-hydroxynaphthalene-1,4-dione (2 mmol); aryl aldehyde (2 mmol); 2*H*-indene-1,3-dione (2 mmol); ammonium acetate (3 mmol); [MSAIm]HSO₄ (10 mg); P(4-VPH)HSO₄ (40 mg); solvent-free. ^a Isolated yield.

steric hindrance of the $-\text{NO}_2$ group in 2-nitrobenzaldehyde (Table 2, entry 10). The presence of a halogen on the aromatic ring of the aldehyde had a negligible effect on the reaction results (Table 2, entries 2–5). Aliphatic aldehydes were not good substrates, the reactions did not progress, and numerous products were obtained.

As a blank, the model reaction was carried out in the absence of a catalyst under the optimized conditions; the product yield was 30% after 6 h. The product structures were characterized on the basis of their spectroscopic properties (IR, ^1H NMR, and mass) and elemental analysis, and by comparison with authentic samples [45].

The reusability of the ionic liquid in the synthesis of 12-(4-chlorophenyl)benzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione was evaluated. When the reaction was complete, the product was separated and new substrates were added to the reaction vessel. This process was repeated for five runs; the results show that the catalyst activity did not decrease, and the yields ranged from 82% to 79% in an average reaction time of approximately 37 min. These results clearly demonstrate the practical recyclability of the $[\text{MSAIm}]\text{HSO}_4$ ionic liquid (Table 3).

To highlight the merits of the present method in comparisons with other reported methods for the same transformation [45], we have tabulated some of the results in Table 2. The results show that the new method has advantages such as low catalyst loadings, short reaction time, and similar or higher yields.

Based on literature reports [45,50,51], a possible mecha-

Table 3

Reusability of $[\text{MSAIm}]\text{HSO}_4$ in synthesis of **4b** under optimized reaction conditions.

Run	Time (min)	Isolated yield (%)
1	35	82
2	35	82
3	35	82
4	38	79
5	40	79

nism for this reaction is shown in Scheme 2. 2-Hydroxynaphthalene-1,4-dione (**1**), aldehyde (**2**), and 2*H*-indene-1,3-dione (**3**) are activated by the Brønsted ionic liquid $[\text{MSAIm}]\text{HSO}_4$. First, the carbonyl carbon of aldehyde (**2**) is attacked by 2*H*-indene-1,3-dione (**3**) or 2-hydroxynaphthalene-1,4-dione (**1**) to form a Knoevenagel intermediate (**I**) or (**III**). Intermediate (**I**) and ammonium acetate afford enamine (**II**). Alternatively, the activated 2-hydroxynaphthalene-1,4-dione (**1**) or 2*H*-indene-1,3-dione (**3**), the catalyst, and ammonium acetate give enamine (**IV**). Subsequent Michael addition of enamine (**II**) to 2-hydroxynaphthalene-1,4-dione (**1**), or enamine (**IV**) to intermediate (**III**), followed by cyclization, dehydration, and aromatization, afford the indeno[*g*]quinoline product (**4**). It should be mentioned that the increase in the reaction rate might be caused by an increase in the rate of absorption of water by the ionic liquid. It should be mentioned that the role of $[\text{MSAIm}]\text{HSO}_4$ in catalyzing organic reactions can be attributed to the C-2 hydrogen of the imidazolium moiety [52], although the H^+ of the sulfonic acid or hydrogen sulfate group also make

Scheme 2. Possible mechanism for synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione in presence of $[\text{MSAIm}]\text{HSO}_4$ under solvent-free conditions.

a significant contribution; the proton is transferred to the ionic liquid in another step.

To the best of our knowledge, no biodegradation data on (MSAIm)HSO₄ has been published, and this is the first report on the biodegradability of this ionic liquid. The biodegradability of (MSAIm)HSO₄ was evaluated using the “closed bottle test” (OECD 301 D) [53]. In this test, BOD/COD for (MSAIm)HSO₄ was 44% within 28 d of incubation, showing significant biodegradation of (MSAIm)HSO₄ (Compounds that reach a biodegradation level higher than 60% are referred to as “readily biodegradable” [54]).

4. Conclusions

In conclusion, an efficient method for the synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-triones has been developed. This is the first report of the synthesis of these compounds via one-pot, multicomponent condensation of 2-hydroxynaphthalene-1,4-dione, various aldehydes, 2*H*-indene-1,3-dione, and ammonium acetate in the presence of [MSAIm]HSO₄ as a Brønsted ionic liquid catalyst. The current method has the advantages of a simple experimental procedure, use of inexpensive materials, good to high product yields, and reusability of the ionic liquid.

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Graphical Abstract

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Nader Ghaffari Khaligh*

Research House of Professor Reza, Education Guilan, Iran

The synthesis of 12-arylbenzo[*g*]indeno[2,1-*b*]quinoline-6,11,13-trione derivatives was accomplished through a one-pot, multicomponent condensation in the presence of a Brønsted ionic liquid catalyst, namely 3-methyl-1-sulfonic acid imidazolium hydrogen sulfate, under solvent-free conditions.

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