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PPA-Mediated C–C Bond Formation: A Synthetic Route to Substituted Indeno[2,1-*c*]quinolin-6(7*H*)-ones

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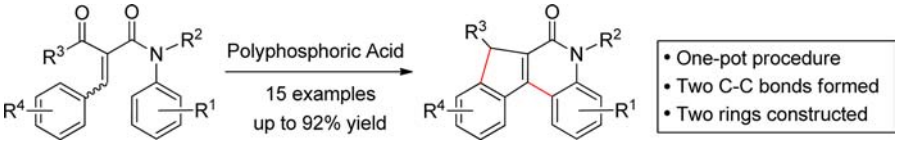
ABSTRACT



A facile and efficient synthesis of substituted indeno[2,1-*c*]quinolin-6(7*H*)-ones from a variety of α-acyl *N*-arylcinnamamides mediated by polyphosphoric acid (PPA) is described, and a mechanism involving the formation of a dicationic superelectrophile and subsequent double intramolecular nucleophilic cyclization reactions is proposed.

The vast number of bioactive natural products and pharmaceutical drugs based on the indeno[2,1-*c*]quinoline ring system exhibit a diverse range of biological properties,[1] such as 5-HT-receptor binding activity[2] and anti-inflammatory activity,[3] and also act as antitumor agents,[4] antimalarials,[1a,5] acetylcholinesterase inhibitors,[6] potential topo I/II inhibitors,[7] and steroid reductase inhibitors.[8] In addition, functionalized indeno[2,1-*c*]quinolines have been used as versatile intermediates in the synthesis of a series of other biologically important compounds.[9] The development of efficient synthetic approaches for indeno[2,1-*c*]-quinolines has been the focus of intense research for decades and continues to be an active area of research today.[10] These compounds are frequently synthesized via the aza-Bergman cyclization,[11] Friedel–Crafts-type reaction,[12] or the imino Diels–Alder reaction.[13] Recently, Lee and co-workers developed an efficient synthetic method for 7*H*-indeno[2,1-*c*]quinolines from Baylis–Hillman

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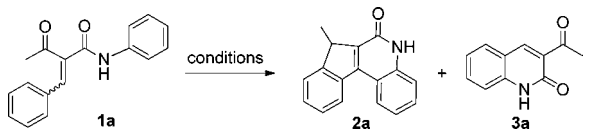
adducts in polyphosphoric acid (PPA).[14] Nevertheless, to match the increasing scientific and practical demands, it is still of continued interest and great importance to explore novel, efficient synthetic approaches for indeno-[2,1-*c*]quinolines.

During the course of our studies on the synthesis of carbo- and heterocycles based on *β*-oxo amide derivatives,[15] we successfully achieved efficient synthesis of pyrano[2,3-*b*]-quinolines from enaminones mediated by trifluoromethanesulfonic acid[16] and substituted quinolin-2(1*H*)-ones from penta-2,4-dienamides mediated by concentrated H_2SO_4.[17] In connection with this previous work and our continued efforts for the synthesis of highly valuable heterocycles through acid-mediated processes, we synthesized a series of α-acyl-*N*-arylcinnamamides from *β*-oxo amides and examined their reactivity toward acidic conditions. As a result of these studies, we have developed a facile and efficient synthesis of substituted indeno[2,1-*c*]quinolin-6(7*H*)-ones. Herein we report our preliminary results and a proposed mechanism.

The substrates, α-acylcinnamamides, were prepared by Knoevenagel condensation of commercially available *β*-oxo amides with aryl aldehydes in the presence of piperidine in acetic acid according to a reported procedure.[18] It should be mentioned that the obtained α-acylcinnamamides were pure *E*-isomers for tertiary amides or a mixture of a pair of *E*- and *Z*-isomers for secondary amides based on their ^{1}H NMR and NOE analytical data (see the Supporting Information). Then, 2-benzylidene-3-oxo-*N*-phenylbutanamide **1a** was selected as the model compound and subjected to H_2SO_4 (98%) at room temperature. As indicated by TLC, the reaction occurred and furnished two products after workup and purification by column chromatography of the resulting mixture. The main product was characterized as 7-methyl-5*H*-indeno[2,1-*c*]quinolin-6(7*H*)-one **2a**, and the by-product was characterized as 3-acetylquinolin-2(1*H*)-one **3a** on the basis of its spectroscopic and analytical data (Table 1, entry 1). Actually, we have revealed that the same compound **3a** could be synthesized from penta-2,4-dienamide in the presence of concentrated H_2SO_4 in high yield.[17]

The results encouraged us to investigate the reaction conditions with the aim of optimizing the yield of **2a**. It was observed that the reaction temperature had a dramatic effect on the reaction of **1a** in concentrated H_2SO_4. When the reaction temperature was increased to 50 °C, **2a** and **3a** were obtained in 55% and 25% yields (Table 1, entry 2), respectively, whereas further increase of the reaction temperature to 90 °C, the yield of **3a** reached 38% (Table 1, entry 3). Similarly, treatment of **1a** with PPA at 90 °C could only afford **2a** in 37% yield along with the recovery of **1a** (Table 1, entry 4). To our delight, when the reaction of **1a** was performed in PPA at 130 °C, **2a** was isolated in 92% yield (Table 1, entry 5). It seemed that further increase of the reaction temperature had no significant influence on the conversion, although the reaction rate could be accelerated (Table 1, entry 6). The reaction of **1a** could take place in CF_3SO_3H, but the conversion was very low within a short reaction time, and a complex mixture containing **2a** was formed when the reaction time was prolonged (Table 1, entries 7 and 8). No reaction was observed when **1a** was subjected to CF_3COOH at room temperature, and a complex mixture was formed at high temperature and the desired **2a** was not even detected (Table 1, entries 9 and 10).

Table 1. Reactions of **1a** under Different Acidic Conditions[a]



				yield[b] (%)	
entry	acids	temp (°C)	time (h)	**2a**	**3a**
1	H_2SO_4(98%)	rt	5.0	79	6
2	H_2SO_4(98%)	50	3.0	55	25
3	H_2SO_4(98%)	90	1.0	<5	38
4	PPA	90	12.0	37[c]	<5
5	PPA	130	12.0	92	<5
6	PPA	150	3.0	89	<5
7	CF_3SO_3H	rt	3.0	34[d]	<5
8	CF_3SO_3H	rt	5.0	complex mixture	
9	CF_3COOH	rt	10.0	no reaction	
10	CF_3COOH	reflux	10.0	complex mixture	

[a] Reaction conditions: **1a** (2.0 mmol), acid (5.0 mL). [b] Isolated yield. [c] 46% of **1a** was recovered. [d] 53% of **1a** was recovered.

Under the optimal conditions for **2a** in entry 5, Table 1, a range of reactions of substrates **1** were carried out to determine the scope of the indeno[2,1-*c*]quinolin-6(7*H*)-one synthesis, and some of the results are summarized in Table 2. It was found that the reactions of **1b**–**j** bearing variable aryl amide groups with both electron-donating (e.g., Me, MeO) and electron-withdrawing (e.g., Cl) substituents R^1 on the benzene ring in the presence of PPA at 130 °C proceeded smoothly to afford the corresponding substituted indeno[2,1-*c*]quinolin-6(7*H*)-ones **2b**–**j** in high yields (Table 2, entries 2–10). In the case of **1f**,

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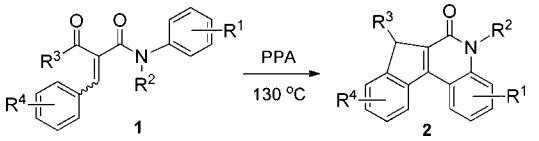
indeno[2,1-*c*]quinolin-6(7*H*)-one **2f** was exclusively obtained in 86% yield, which indicated that the cyclization reaction of **1f** occurred in a regioselective manner (Table 2, entry 6). In the same fashion, the corresponding indeno-[2,1-*c*]quinolin-6(7*H*)-one **2k** was obtained in good yield when substrate **1k** containing a propyl group (R^3) was employed (Table 2, entry 11). The versatility of this cyclization protocol was further evaluated by performing the reaction of α-acyl *N*-arylcinnamamides **1i**–**1o** bearing variable aromatic groups (R^4) under identical conditions (Table 2, entries 12–15).

Table 2. Reactions of α-Acyl *N*-Arylcinnamamides **1** with PPA[a]



entry	1	R^1	R^2	R^3	R^4	2	yield[b] (%)
1	**1a**	H	H	Me	H	**2a**	92
2	**1b**	H	Me	Me	H	**2b**	87
3	**1c**	H	Et	Me	H	**2c**	89
4	**1d**	4-Me	H	Me	H	**2d**	91
5	**1e**	2-Me	H	Me	H	**2e**	78
6	**1f**	3-Me	H	Me	H	**2f**	86
7	**1g**	2,4-Me_2	H	Me	H	**2g**	81
8	**1h**	4-Cl	H	Me	H	**2h**	75
9	**1i**	4-MeO	H	Me	H	**2i**	77
10	**1j**	2-MeO	H	Me	H	**2j**	82
11	**1k**	H	H	Pr	H	**2k**	84
12	**1l**	H	H	Me	4-Me	**2l**	67
13	**1m**	H	H	Me	2-Me	**2m**	71
14	**1n**	4-Me	H	Me	2-Me	**2n**	69
15	**1o**	2-Me	H	Me	2-Me	**2o**	72

[a] Reagents and conditions: **1** (1.0 mmol), PPA (2.0 mL), 130 °C, 10.0–15.0 h. [b] Isolated yield.

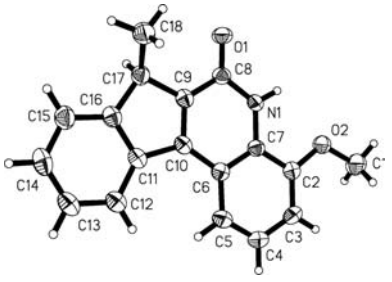
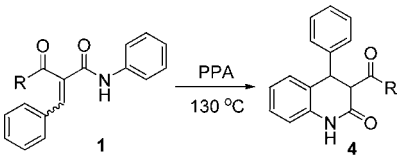


Figure 1. ORTEP drawing of **2j**.

It is worth mentioning that the structure of **2j** was elucidated by means of NMR (^1H, ^{13}C) spectra and further confirmed by the X-ray single-crystal analysis (Figure 1). The results shown above demonstrated the efficiency and synthetic value of the cyclization with respect to α-acyl *N*-arylcinnamamides **1**. Therefore, we provide a facile, efficient, and metal-free synthetic method for a highly substituted indeno[2,1-*c*]quinolin-6(7*H*)-one of type **2**.

Scheme 1. Reactions of *N*-Arylcinnamamides **1p** and **1q** with PPA

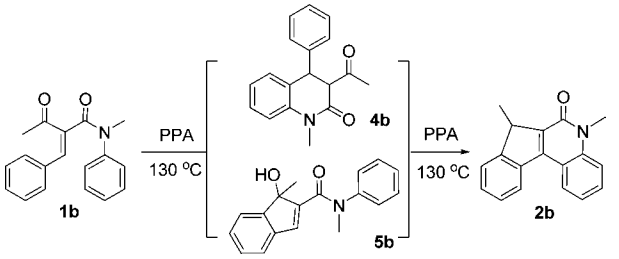


4p: R = Ph, yiled 85%

4q: R = EtO, yield 91%

To extend the scope of this indeno[2,1-*c*]quinolin-6(7*H*)-one synthesis, we prepared *N*-arylcinnamamides **1p** and **1q** with benzyl and ester groups, respectively, as shown in Scheme 1 and subjected them to PPA at 130 °C. In these cases, 3-benzoyl-4-phenyl-3,4-dihydroquinolin-2(1*H*)-one **4p** and ethyl 2-oxo-4-phenyl-1,2,3,4-tetrahydroquinoline-3-carboxylate **4q** were obtained in excellent yields (Scheme 1). The results revealed that **1p** and **1q** could only undergo a single nucleophilic cyclization to form the dihydro quinolin-2(1*H*)-one ring, and probably the further nucleophilic cyclization reaction was prohibited by the electronic and steric effect from benzyl and ester groups.

Scheme 2. Stepwise experiment with **1b** and PPA



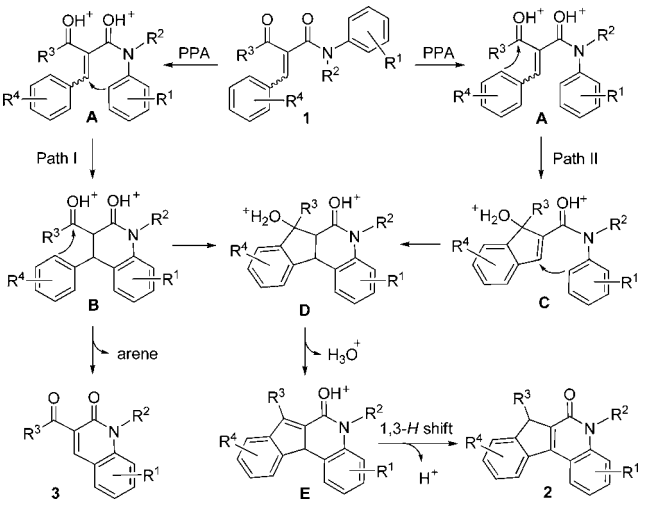
To gain insight into the mechanism of the indeno[2,1-*c*]quinolin-6(7*H*)-one synthesis, a controlled experiment was carried out. As shown in Scheme 2, (*E*)-2-benzylidene-3-oxobutanamide **1b** was subjected into PPA at 130 °C for 0.3 h, the reaction was then quenched. Two products, 3-acetyl-1-methyl-4-phenyl-3,4-dihydroquinolin-2(1*H*)-one **4b** and 1-hydroxy-*N*,1-dimethyl-*N*-phenyl-1*H*-indene-2-carboxamide **5b**, were obtained as an inseparable mixture with a ratio of 5:2 by silica gel column chromatography (determined by NMR spectra). The mixture was then investigated by means of GC–MS. From one mass spectrum, the peaks appeared at 279 and 236 were assigned to the molecular ion $[M]^+$ of **4b** and its molecular fragment ion $[M - CH_3CO]^+$, respectively. Similarly, the peaks displayed at 279, 202, and 184 on another mass spectrum could be easily assigned to the molecular ion $[M]^+$ of **5b**, and its molecular fragment ions $[M - Ph]^+$ and $[M - Ph - OH + 1]^+$, respectively. It is worth noting that the

obtained mixture of **4b** and **5b** could be converted into **2b** in 94% yield when treated with PPA at 130 °C within 2.0 h. These results indicate that **4b** and **5b** are generated simultaneously, and both of them are the intermediates in the transformation.

The formation of **4b** demonstrated that the nucleophilic addition site of aryl amide was on the C−C double bond of **1b** instead of the α-acyl group and also clearly showed that the intramolecular cyclization proceeded in a chemoselective manner, which was different from the conventional acid-catalyzed Knorr quinolin-2(1*H*)-one synthesis.[19] During the investigation of the Knorr cyclization at 1960s and 1970s, a number of cationic electrophilic reagents were noted to show greatly enhanced reactivities in the presence of superacids, which led to the concept of superelectrophilic activation.[20] At the time of that study, there was a considerable amount of uncertainty regarding the protonation site on amides. This problem has been resolved over the years and in most cases oxygen is preferred over nitrogen as the protonation site.[21] Recently, Sai et al. studied the acid-catalyzed Knorr cyclization by both experimental and computational methods, and revealed that *β*-oxo amides underwent deprotonation at the two carbonyl oxygen atoms to form distonic superelectrophiles, which gave rise to Knorr products.[22]

On the basis of the results obtained above and the reported literature,[23] a plausible mechanism for the synthesis of indeno[2,1-*c*]quinolin-6(7*H*)-ones **2** is presented in Scheme 3. Mediated by PPA, α-acylcinnamamide **1** is protonated to form a dicationic superelectrophile **A**,[20] which undergoes double-intramolecular cyclization reactions in two different pathways, path I and path II, to give the same intermediate **D**.[24] The elimination of water from **D** and the subsequent isomerization of intermediate **E** via 1,3-*H* shift under acidic conditions[25] led to the formation of the final product indeno [2,1-*c*]quinolin-6(7*H*)-one of type **2**. It is most likely that dihydroquinolinone **4** and 1*H*-indene-2-carboxamide **5** are generated from intermediates **B** and **C** upon treatment with water, and quinolin-2(1*H*)-one **3** is derived from **B** through the elimination of an arene.[17]

Scheme 3. Possible Mechanism for the PPA-Mediated Cyclization of α-Acyl-*N*-arylcinnamamides



In summary, a facile and efficient synthesis of indeno [2,1-*c*]quinolin-6(7*H*)-ones **2** is developed from α-acyl *N*-arylcinnamamides, which involves the formation of dicationic superelectrophile, and subsequent double intramolecular nucleophilic cyclization reactions in the presence of polyphosphoric acid (PPA). This protocol is associated with readily available starting materials, mild conditions, good yields, a wide range of substrate scope, dense and flexible substituted patterns, and important synthetic potential of the products.

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Supporting Information Available. Experimental details, spectral and analytical data, and copies of ^{1}H NMR and ^{13}C NMR spectra for new compounds **1**, **2**, **4**, and **5**; crystallographic data for compound **2j** (CIF). This material is available free of charge via the Internet at http://pubs.acs.org

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