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An alternative quinoline synthesis by via Friedländer reaction catalyzed by $Yb(OTf)_3$

Salvatore Genovese [a], Francesco Epifano [a], Maria Carla Marcotullio [b], Caroline Pelucchini [b], Massimo Curini [b,*]

[a] Dipartimento di Scienze del Farmaco, Università 'G. D'Annunzio' di Chieti-Pescara, Via dei Vestini 31, 66100 Chieti Scalo (CH), Italy
[b] Dipartimento di Chimica e Tecnologia del Farmaco, Sezione di Chimica Organica, Università degli Studi di Perugia, Via del Liceo 1, 06123 Perugia, Italy

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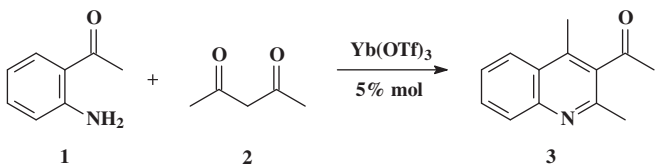
ABSTRACT

Quinolines have been synthesized in very good yields from 2-aminoarylketones and differently substituted carbonyl compounds in the presence of $Yb(OTf)_3$ as the catalyst. The method is applicable to both cyclic and acyclic carbonyl compounds with only slight differences in the experimental procedure.

Quinolines represent an important group of heterocycles. Several quinoline derivatives have been found to exert useful biological activities as anti-malarial, anti-bacterial, anti-asthmatic, anti-hypertensive, and anti-inflammatory agents.[1] In addition quinolines are valuable synthons for the preparation of nano-and meso-structures with enhanced electronic and photonic functions.[2] Because of their importance as substructures in a broad range of natural and semisynthetic products, significant efforts are directed to the development and construction[3] of new quinoline-based structures. Thus, the synthesis of quinolines is an important and useful task in organic chemistry and represents a field of research of current and growing interest. Several methods for the synthesis of the quinoline nucleus have been reported in the literature and among these several 'classic' examples in the history of organic chemistry can be cited. The Friedländer annulation, a straightforward synthesis of the title compounds,[4] is one of these evergreen processes. This reaction is a condensation followed by a cyclodehydration between 2-aminoarylketones and α-methyleneketones and it has been catalyzed by both acids and bases. For examples, in recent years, iodine,[5] Lewis acids,[6] such as $ZnCl_2$, $SnCl_2$, $Bi(OTf)_3$, $Y(OTf)_3$, $AuCl_3$, $CeCl_3{\cdot}7H_2O$, a combination of acidic catalysts and microwave irradiation,[7] ionic liquids,[8] chlorotrimethylsilane,[9] dodecylphosphonic acid,[10] 1-methylimidazolium trifluoroacetate[11] have been proposed as promoting agents for the Friedländer annulation.[12] Unfortunately many of these processes suffer major or minor limitations, such as harsh reaction conditions, low yields, tedious work-up procedure, low selectivity, co-occurrence of several side reactions.[5–11] Moreover in the case of transition metal catalyzed reactions (e.g., Ce^{+3}) there is a need of more than stoichiometric amounts of the Lewis acids to efficiently promote the process.

During the last two decades, rare earth metal triflates have been found as the unique Lewis acid in that they are water tolerant reusable catalysts and they can effectively promote several carbon–carbon and carbon–heteroatom bond formation reactions in high yields.[13] In continuation of our ongoing studies aimed at developing a mild and practical protocol for the synthesis, in water or under solvent free conditions, of useful building blocks for the synthesis of biologically active compounds using $Yb(OTf)_3$ as the catalyst, it was speculated that this lanthanide salt, which was recently shown to catalyze a wide variety of valuable and satisfactory yielding C–C bond forming reactions[13,14] might be ideal for effecting the



1 2 3

Scheme 1. $Yb(OTf)_3$ catalyzed synthesis of quinolines.

* Corresponding author. Tel.: +39 0755855106; fax: +39 0755855116.
E-mail address: curmax@unipg.it (M. Curini).

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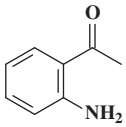
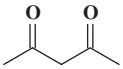
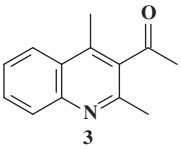
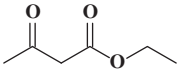
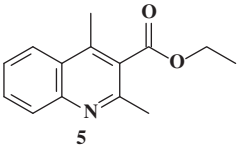
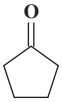
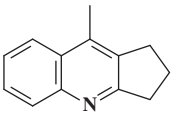
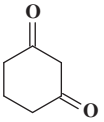
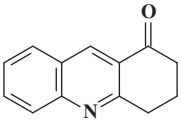
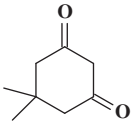
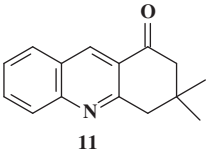
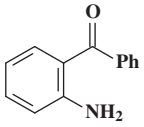
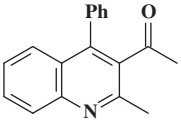
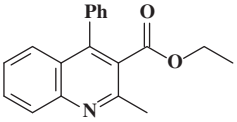
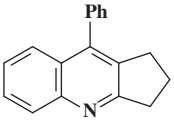
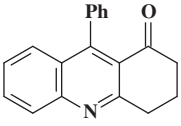
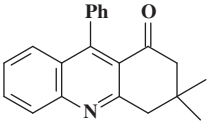
condensation of 2-aminoacetophenones and differently cyclic and acyclic carbonyl compounds, leading to the synthesis of differently substituted quinolines. So, as a part of our studies aimed at exploring the utility of lanthanide triflate promoted reactions under solvent free conditions, we decided to investigate the use of $Yb(OTf)_3$ as a catalyst for the preparation of quinolines from

Table 1
$Yb(OTf)_3$ promoted synthesis of quinolines

Reactant	Carbonylic compound	Product	Yield (%)[a]	Time (h)
 1	 2	 3	85	1
1	 4	 5	90	1
1	 6	 7	95	2
1	 8	 9	85	1
1	 10	 11	85	1
 12	2	 13	90	2
12	4	 14	80	1
12	6	 15	85	2
12	8	 16	80	2
12	10	 17	75	1

[a] Yields of pure isolated products, characterized by IR, GC–MS, ^1H NMR and ^{13}C NMR.

2-aminoaryl ketones and different carbonyl compounds, via the Friedländer annulation. The use of $Yb(OTf)_3$ to perform this kind of process has been suggested by Muchowski and Maddox that, however, carried out the process under harsh conditions (refluxing toluene) providing the desired adducts in low yields.[15] As a preliminary experiment, it was used in the reaction of 2-aminoacetophenone (**1**) (2 mmol) with acetylacetone (**2**) (2 mmol) in the presence of $Yb(OTf)_3$ 5 mol % under solvent free condition (Scheme 1).

The reaction was monitored by TLC and after 45 min at room temperature the complete conversion of the starting aromatic aminoketone was observed. After addition of few drops of NaOH 1 N to precipitate Yb(III) as the corresponding hydroxide, and running a quick work-up, consisting in dilution with H_2O and extraction with CH_2Cl_2, followed by purification by silica gel column chromatography, it was possible to isolate the condensation product in a yield of 85%. Data recorded by NMR spectroscopy and GC analysis confirm the structure of the desired product, namely 1-(2,4-dimethylquinolin-3-yl) ethanone (**3**).[16]

Encouraged by results obtained using 2-aminoarylketone as the substrate, we applied the same experimental conditions, to several differently substituted α-methylene carbonyl compounds and the corresponding quinolines were selectively obtained in good yields. Results are summarized in Table 1.

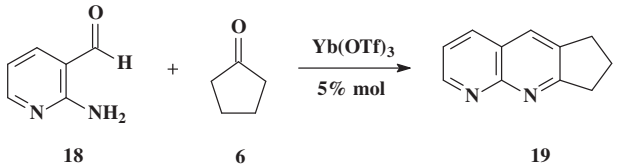
Data reported in Table 1 show that differently linear and cyclic α-methylene carbonyl compounds react without any significant difference to give the corresponding quinolines ether in very good yields ranging from 85% to 90%. Compared to the existing methodologies, our process is effective in avoiding the use of strong mineral acids, high temperatures, toxic and polluting reactants, and in some instances more than stoichiometric loading of the catalyst. De and Gibbs reported in 2005 that another Lewis acid belonging to the lanthanide series, namely $Y(OTf)_3$, was seen to effectively provide a synthetic route to the title compounds and also they reported that Yb^{+3} performed by far worse that Y^{+3} under the same experimental conditions (e.g., use of polar protic or aprotic solvents like EtOH or CH_3CN). Although we obtained similar results, herein we disclose that the use of solvent free conditions or apolar solvents greatly enhances the catalytic efficiency of $Yb(OTf)_3$ leading to quinolines in comparable or better yields but in shorter reaction times. We can hypothesize to this aim that using EtOH or other polar solvents led to a strong coordination of the solvent to Yb^{+3} thus abolishing its catalytic capacities. This solvation effect is completely avoided with the use of strongly apolar solvents like CH_2Cl_2 thus enhancing the catalytic efficiency of our lanthanide. So this latter topic could be regarded as a feature of novelty with respect to the already reported literature in the same field. At the end of each reaction the catalyst was recovered as previously described[2a] and recycled without any significant loss of its catalytic activity.

For example, the reaction leading to compound (**3**) was repeated three additional times with the recovered Lewis acid with yields of 84%, 82%, and 84%, respectively.

It's noteworthy to highlight that under the same experimental conditions inactivated ketones, like cyclohexanone do not react.

The same experimental protocol as described above was followed using other lanthanide triflates, including Y^{+3} and Sc^{+3}, or other transition metal triflates (e.g., Bi^{+3}) for the synthesis of compound (**3**). Results obtained in terms of yield in the desired adducts were in all cases worse than those provided by $Yb(OTf)_3$ ranging from 22% to 34%.

As a final consideration, loading of the catalyst less than 5 mol % did not increase reaction times significantly. The same reaction was also performed by using other metal triflates from the lanthanide series, but the results were worse than those obtained with $Yb(OTf)_3$. The reason for this discrepancy of catalytic efficiency in the lanthanide series could be explained by the fact that Yb^{+3} is the 'hardest' cation and thus the most oxophilic, due to its smaller ionic radius.[17]



18 6 19

Scheme 2. $Yb(OTf)_3$ catalyzed synthesis of naphthyridines.

To extend the applicability of our methodology we thought also about verifying the effectiveness of $Yb(OTf)_3$ to activate the formation of different substituted quinoline rings from different substrates and the same linear and cyclic carbonyl compounds employed with 2-aminoacetophenone as the reactant.

The first reaction used as a model provided the use of 2-aminobenzophenone (**12**) (2.0 mmol) with acetylacetone (**2**) (2.0 mmol) in the presence of $Yb(OTf)_3$ 5 mol % under solvent free conditions (Table 1).

Also in this case the desired product (**13**) was obtained in a very good yield of 90% after usual work-up.

On the basis of the above result, other structurally different carbonyl compounds were converted into quinolines using $Yb(OTf)_3$ in satisfactory yields ranging from 75% to 90%.

Finally, in order to generalize the process and verify its applicability to aminoarylketones containing a heterocycle, we used as the substrate 2-aminopyridine carboxaldehyde (**18**) that was condensed with cyclopentanone (**6**).

The reaction after 1 h showed complete conversion of the starting products in the corresponding adduct condensation (**19**), the structure of which was determined as described above, in quantitative yield (Scheme 2).

It is noteworthy that this latter result allowed us to have access also to the naphthyridine nucleus, another important moiety contained in several biologically active compounds,[18] by the same reaction protocol.

As conclusion, in this manuscript we have demonstrated that 2-aminoarylketones, and differently substituted carbonyl compounds undergo an efficient condensation reaction under the catalysis of $Yb(OTf)_3$ hydrate yielding quinoline and naphthyridine derivatives .The simple work-up procedure, mild reaction conditions, satisfactory to very good yields make our methodology a valid and alternative contribution to the existing processes in the field of the quinoline synthesis.

Further investigation into the scope and other applications of $Yb(OTf)_3$ promoted reactions are now in progress in our laboratories and will be reported in due course.

Acknowledgment

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16. **Experimental**. *General procedure for quinoline synthesis using Yb(OTf)$_3$* 2-Aminoaryl ketone (1.0 mmol), carbonyl compound (1.0 mmol) (dissolved in CH_2Cl_2 (1 ml) when needed) and the resulting solution was added Yb(OTf)$_3$ (5 mol %) and stirred at room temperature for the appropriate time (see Table 1). After completion of the reaction by monitoring by TLC, eluent CH_2Cl_2, few drops of NaOH 1 N were added, the precipitate formed filtered under vacuum and the filtrate solution washed with H_2O and extracted twice with CH_2Cl_2 (5 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and the solvent evaporated to dryness. The syrup obtained was finally purified by SiO_2 gel column chromatography (eluent CH_2Cl_2/MeOH 99:1) affording pure quinoline.
1-(2,4-Dimethylquinolin-3-yl)ethanone (***3***): Yellow solid (mp: 240–241 °C); Anal. Calcd for $C_{13}H_{13}NO$: C, 78.36; H, 6.58; N, 7.03; O, 8.03. Found: C, 78.70; H, 6.09; N, 6.85. Analytical data were in full agreement with those already reported in the literature for the same compound.[19]
Ethyl 2,4-dimethylquinoline-3-carboxylate (***5***): Yellow solid (mp: 271–272 °C); Anal. Calcd for $C_{14}H_{15}NO$: C, 73.34; H, 6.59; N, 6.11; O, 13.96. Found: C, 73.65; H, 6.23; N, 6.05.Analytical data were in full agreement with those already reported in the literature for the same compound.[20]
9-Methyl-2,3-dihydro-1H-cyclopenta[b]quinoline (***7***): White solid (mp: 64–66 °C); Anal. Calcd for $C_{13}H_{13}N$: C, 85.21; H, 7.15; N, 7.64. Found: C, 85.13; H, 7.25; N, 7.62. Analytical data were in full agreement with those already reported in the literature for the same compound.[19]
9-Methyl-3,4-dihydroacridin-1(2H)-one (***9***): White solid (mp: 78–79 °C); Anal. Calcd for $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63; O, 7.57. Found: C, 79.34; H, 6.19; N, 6.78. Analytical data were in full agreement with those already reported in the literature for the same compound.[21]
3,3,9-Trimethyl-3,4-dihydroacridin-1(2H)-one (***11***): Pale yellow solid (mp: 105–106 °C); Anal. Calcd For $C_{16}H_{17}NO$: C, 80.30; H, 7.16; N, 5.85; O, 6.69. Found: C, 80.45; H, 7.25; N, 5.91. Analytical data were in full agreement with those already reported in the literature for the same compound.[22]
1-(2-Methyl-4-phenylquinolin-3-yl)ethanone (***13***): Yellow solid (mp: 114–115 °C); Anal. Calcd for $C_{18}H_1NO$: C, 82.73; H, 5.79; N, 5.36; O, 6.12. Found: C, 82.81; H, 5.54; N, 5.45. Analytical data were in full agreement with those already reported in the literature for the same compound.[23]
Ethyl 2-methyl-4-phenylquinoline-3-carboxylate (***14***): Yellow solid (mp: 99–100 °C); Anal. Calcd for $C_{19}H_{17}NO_2$: C, 78.33; H, 5.88; N, 4.81; O, 10.98. Found: C, 78.43; H, 5.44; N, 4.76. Analytical data were in full agreement with those already reported in the literature for the same compound.[23]
9-Phenyl-2,3-dihydro-1H-cyclopenta[b]quinoline (***15***): White solid (mp: 129–130 °C); Anal. Calcd for $C_{18}H_{15}N$: C, 88.13l; H, 6.16; N, 5.71. Found: C, 88.42; H, 6.25; N, 5.33. Analytical data were in full agreement with those already reported in the literature for the same compound.[23]
9-Phenyl-3,4-dihydroacridin-1(2H)-one (***16***): Pale yellow solid (mp: 155–156 °C); Anal. Calcd for$C_{19}H_{15}NO$: C, 83.49; H, 5.53; N, 5.12; O, 5.85. Found: C, 83.64; H, 5.11; N, 5.43. Analytical data were in full agreement with those already reported in the literature for the same compound.[24]
3,3-Dimethyl-9-phenyl-3,4-dihydroacridin-1(2H)-one (***17***): Pale yellow solid (mp: 240–241 °C); Anal. Calcd for $C_{21}H_{19}NO$: C, 83.69; H, 6.35; N, 4.65; O, 5.31. Found: C, 83.71; H, 6.39; N, 4.61. Analytical data were in full agreement with those already reported in the literature for the same compound.[24]
7,8-Dihydro-6H-cyclopenta[b]-1,8-naphthyridine (***19***): white solid (mp: 91–93 °C).
^{1}H NMR[5]; (200 MHz, $CDCl_3$) δ 2.75 (t, 3H), 3.01 (t, 3H), 3.23 (q, 2H), 7.81–7.53(m,4H), 8.34–8.07 (m, 2H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 23.7, 26.1, 34.5, 119.2, 123.4, 127.3, 132.2, 132.5, 151.67, 152.6, 164.3; GC/MS: M^+ 170 (99) 43, 68, 79, 91, 120, 130, 158. Anal. Calcd for $C_{11}H_{10}NO$: C, 77.62; H, 5.92; N, 16.46. Found: C, 77.67; H, 5.34; N, 16.99.
The degree of purity of each product listed in the paper was analyzed by GC/MS with a Hewlett Packard 6890 gas chromatograph equipped with a 12.5 mm × 0.25 mm MetSil column couplet to HP Chem Station Software. The carrier gas was helium at a pressure of 3.5 kg/cm^2, and the column temperature was programmed from 50 to 270 °C at 10 °C/min. The chromatogram was obtained by using a reporting integrator. Mass spectra were obtained from a GCMS system operating in the EI mode at 70 eV, equipped with a 12.5 mm × 0.25 mm MetSil column and an HP5973 Mass Selective Detector, by using the same chromatographic conditions reported above. The column was connected to the mass spectrometer insource through an open-split interface heated at 250 °C.
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