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Ytterbium Triflate Promoted One-Pot Three Component Synthesis of 3,4,5-Trisubstituted-3,6-dihydro-2*H*-1,3-oxazines

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Abstract An efficient one-pot synthesis of 3,4,5-trisubstituted-3,6-dihydro-2*H*-1,3-oxazines from acetylene dicarboxylates, aromatic and aliphatic amines, and formaldehyde is described. The six member *N*,*O*-heterocyclic nucleus was constructed via $Yb(OTf)_3$ promoted domino hydroamination/Prins reaction/cyclization/dehydration reactions.

Keywords Heterogeneous catalysis · Multicomponent reactions · Lanthanides · Oxazine derivatives · Ytterbium triflate

1 Introduction

The development of simple routes to widely used chemicals is one of the most challenging task in organic synthesis [1]. In this context, domino reactions including multicomponent processes have been employed as a powerful mean to reach this goal [2–8]. Multicomponent reactions (MCRs) in fact allow to get access to molecular structure complexity and diversity from readily available starting materials in the same reaction vessel by a one-step procedure [9–17]. Despite the fact that it is approaching its century "birthday" [18], the Mannich reaction still remains one of the most important C–C bond-forming reactions for production of nitrogen-containing molecules [19–21]. 1,3-Oxazine derivatives represent a group of compounds of particular interest for their antibiotic [22–24], anticancer [25], analgesic [26], and anticonvulsant [27] activities. Although several different methods for the preparation of title compounds have already been reported in the literature [28–31], very few have been accomplished by means of a the MCRs-based methodology. Recently Jiang and coworkers reported the synthesis of 3,4,5-trisubstituted-3,6-dihydro-2H-1,3-oxazines from alkynoates, anilines, and formaldehyde via a Bronsted acid (e.g. HCl) promoted domino hydroamination/Prins reaction/cyclization/dehydration process [32]. A recent alternative to this process is represented by the use of sulfamic acid as catalyst of the title process [33]. However many of the proposed methodologies suffer major or minor drawbacks such as harsh reaction conditions, low yields, tedious work-up procedures, low selectivity leading to mixture of differently sized rings, co-occurrence of several side reactions, and need of chromatography for purification of adducts.

During the last two decades, rare earth metal triflates have been found to be unique Lewis acids since they are water tolerant reusable catalysts and can effectively promote several carbon–carbon and carbon–heteroatom bonds formation reactions in high yields, including MCRs [34]. In the course of our investigations, we have recently seen that rare earth metal triflates are able to efficiently promote a variety of valuable and high yielding C–C bond-forming reactions in the context of a green chemical approach [35]. In continuation of our ongoing studies aimed at developing mild and practical protocols for the synthesis of useful building blocks and/or biologically active compounds by using lanthanides as catalysts, and employing water as the solvent or solvent-free conditions, we wish to report herein

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that 1,3-oxazine derivatives can be effectively synthesized by a MCR catalyzed by $Yb(OTf)_3$ hydrate in satisfactory yields using acetylene dicarboxylates, aromatic and aliphatic amines, and formaldehyde (Scheme 1).

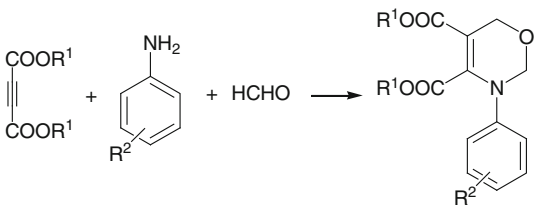
2 Results and Discussion

In a preliminary experiment, dimethyl acetylene dicarboxylate ($R^1 = Me$) (1 equiv.), aniline ($R^2 = H$) (1 equiv.), and formaldehyde (2 equiv. of fresh 37% aqueous solution) were added 10% mol $Yb(OTf)_3$ hydrate. The resulting reaction mixture was vigorously stirred at room temperature for 6 h. After the addition of 1 N NaOH to precipitate Yb^{3+} as the hydroxide and filtration, the desired adduct dimethyl 3-phenyl-3,6-dihydro-2*H*-1,3-oxazine-4,5-dicarboxylate (entry 1) was obtained in 72% yield after extraction with Et_2O and purification by SiO_2 gel column chromatography. Saponification of the ester functions in the adducts occurred to a very limited extent during the described recovery procedure and was virtually undetectable. To investigate the advantages and limitations of our methodology, other amines, having electron-withdrawing and donating substituents in the aromatic ring (entries 2–6 and 10–14), as well as aliphatic ones like *n*-butylamine (entries 7 and 15) or cyclohexylamine (entries 8 and 16), and diethyl acetylene dicarboxylate ($R^1 = Et$), as an alternative source of alkynoate were used as reactants. The results of the performed condensations are reported in the Table 1. See Sect. 1.

From data reported in the Table 1 it is evident that both aromatic amines having electron donating or withdrawing substituents as well as aliphatic aldehydes reacted nearly to the same extent furnishing the desired adducts in yields ranging from 50 to 72%. Compared to the existing methodologies, our process is effective in avoiding the use of strong mineral acids and an excess of HCHO. The catalyst was always recovered by precipitation as $Yb(OH)_3$, filtrated, and transformed into the triflate salt as already described [36]. Recycled in this way, the catalyst could be reused several times without any significant loss of activity. For example the reaction leading to product of entry 1 was repeated four additional times with the recovered Yb^{3+} with yields in oxazine derivative of 71, 69, 64, and 68% respectively.

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 of 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 [37].

From a mechanistic point of view, it could be hypothesized that Yb^{3+} first coordinates the alkynoate thus enhancing its reactivity towards the hydroamination reaction, and subsequent coordination to the oxygen atom of formaldehyde led to the formation of the desired adduct by Prins reaction and consequent dehydration and cyclization. The "hard" feature as a Lewis acid of Yb^{3+} would result in a great enhancement of the electrophilicity of the intermediates involved in the steps of the MCR we are dealing with all requiring a coordination to an oxygen atom.



Scheme 1

3 Conclusions

As a conclusion, in this manuscript we have demonstrated that dimethyl- or diethyl acetylene dicarboxylate, formaldehyde, and differently substituted aromatic and aliphatic amines undergo an efficient condensation reaction under the catalysis of $Yb(OTf)_3$ hydrate yielding 1,3-oxazine derivatives. The simple workup procedure, mild reaction conditions, and satisfactory yields make our methodology a valid and alternative contribution to the already existing processes in the field of this kind of multicomponent process. Moreover, this protocol introduces a practical and viable technology of solvent-free reactions. Further investigations to broaden the scope of this methodology are in progress in our laboratories and will be reported in due course.

4 Experimental

4.1 General Remarks

All reagents, including $Yb(OTf)_3$ hydrate, were obtained from commercial sources (Aldrich Chemical Co.) and used without further purification. All solvents were of analytical grade. All extracts were dried over anhydrous Na_2SO_4. ^1H and ^{13}C NMR spectra were recorded on a Bruker AC 200 (^1H NMR, 200 MHz; ^{13}C NMR, 50.32 MHz) $CDCl_3$ was used as the solvent and tetramethylsilane as an internal standard. Chemical shifts are reported in δ (ppm). Reactions were routinely monitored by TLC using Merck silica gel F_{254} plates and visualization of TLC spots with a

Table 1 Yb(OTf)3 catalyzed synthesis of 1,3 oxazine derivatives

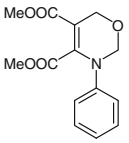
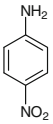
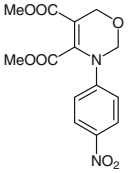
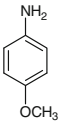
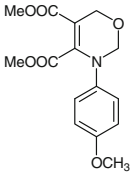
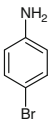
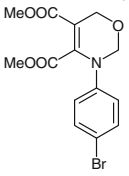
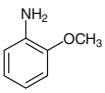
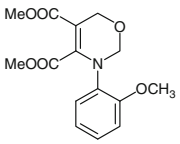
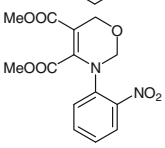
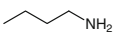
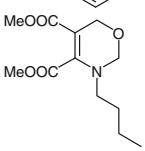
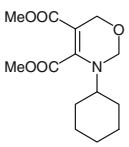
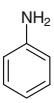
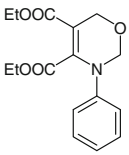
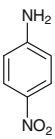
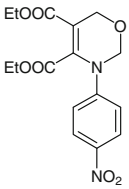
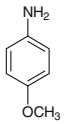
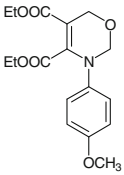
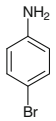
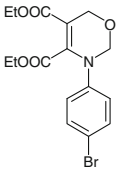
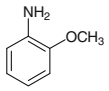
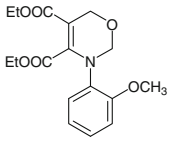
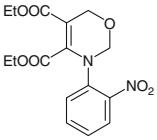
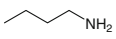
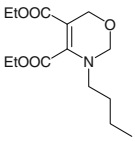
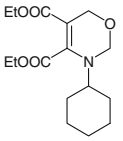
Entry	Alkynoate	Amine	Product	Yield (%)[a]
1	R^1 = Me			72
2	R^1 = Me			60
3	R^1 = Me			67
4	R^1 = Me			70
5	R^1 = Me			65
6	R^1 = Me			50
7	R^1 = Me			60
8	R^1 = Me			60
9	R^1 = Et			70
10	R^1 = Et			60

Table 1 continued

Entry	Alkynoate	Amine	Product	Yield (%)[a]
11	R^1 = Et			65
12	R^1 = Et			68
13	R^1 = Et			58
14	R^1 = Et			60
15	R^1 = Et			60
16	R^1 = Et			60

[a] Yields of pure isolated products, characterized by IR, elemental analysis, ^{1}H NMR, and ^{13}C NMR

freshly prepared 7% EtOH solution of phosphomolybdic acid. Silica gel 40 (0.063–0.200 mm) from Merck was used for column chromatography. Melting points were measured on a Büchi melting point apparatus and are uncorrected prior crystallization with *n*-hexane. Elemental analyses were carried out on a Carlo Erba 1106 elemental analyzer. The purity of the intermediates and the final products was confirmed by combustion analysis.

4.2 Synthesis of 1,3-Oxazine Derivatives. General Procedure

$Yb(OTf)_3$ hydrate (0.1 mmol) was added to a stirred suspension of alkynoate (1.0 mmol), aromatic amine (1.1 mmol), and formaldehyde 37% (2.1 mmol). The resulting mixture was vigorously stirred for 6 h at room temperature, few drops of a solution of NaOH 1 N were added, and the precipitate formed was filtered under vacuum. The filtrate was extracted twice with Et_2O (5 mL) and 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 column chromatography (eluent CH_2Cl_2/MeOH 99:1) yielding the desired product.

4.2.1 Dimethyl 3-phenyl-3,6-dihydro-2H-1,3-oxazine-4, 5-dicarboxylate (1)

Pale yellow solid (m.p. = 191–192 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{14}H_{15}NO_5$ C 60.64; H 5.45; N 5.05; O 28.85. Found: C 60.59; H 5.43; N 5.01; O 28.81.

4.2.2 Dimethyl 3-(4′-nitro)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (2)

Orange solid (m.p. = 203–205 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{14}H_{14}N_2O_7$

C 52.18; H 4.38; N 8.69; O 34.75. Found: C 52.12; H 4.33; N 8.61; O 34.78.

4.2.3 Dimethyl 3-(4′-methoxy)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (3)

Pale reddish solid (m.p. = 162–164 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{15}H_{17}NO_6$ C 58.63; H 5.45; N 4.56; O 31.24. Found: C 58.65; H 5.41; N 4.52; O 31.21.

4.2.4 Dimethyl 3-(4′-bromo)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (4)

Pale red solid (m.p. = 172–173 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{14}H_{14}BrNO_5$ C 47.21; H 3.96; N 3.93; O 22.46. Found: C 47.15; H 3.93; N 3.88; O 22.40.

4.2.5 Dimethyl 3-(2′-methoxy)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (5)

Red solid (m.p. = 165–167 °C). IR = 1,650 cm^{-1}. ^1H NMR δ 3.72 (s, 3H), 3.88 (s, 3H), 3.92 (s, 3H), 4.76 (s, 2H), 4.97 (s, 2H), 6.52–7.84 (m, 4H). ^{13}C NMR δ 51.1, 52.2, 55.7, 67.8, 83.0, 114.1, 116.7, 125.4, 126.2, 130.4, 130.9, 131.4, 132.4, 151.3, 166.2, 166.3. Anal. Calcd. for $C_{15}H_{17}NO_6$ C 58.63; H 5.45; N 4.56; O 31.24. Found: C 58.68; H 5.40; N 4.50; O 31.18.

4.2.6 Dimethyl 3-(2′-nitro)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (6)

Orange solid (m.p. = 186–188 °C). IR = 1650, 1550, 1305 cm^{-1}. ^1H NMR δ 3.74 (s, 3H), 3.92 (s, 3H), 4.81 (s, 2H), 5.02 (s, 3H), 7.29–7.97 (m, 4H). ^{13}C NMR δ 51.2, 52.1, 67.4, 78.8, 115.8, 120.3, 127.0, 130.5, 131.0, 131.5, 135.8, 165.5, 166.8. Anal. Calcd. for $C_{14}H_{14}N_2O_7$ C 52.18; H 4.38; N 8.69; O 34.75. Found: C 52.14; H 4.37; N 8.62; O 34.70.

4.2.7 Dimethyl 3-nbutyl-3,6-dihydro-2H-1,3-oxazine-4, 5-dicarboxylate (7)

Pale yellow solid (m.p. = 168–169 °C). IR = 1,650 cm^{-1}. ^1H NMR δ 0.94 (t, 3H, J = 5.1 Hz), 1.39 (m, 2H), 1.92 (m, 2H), 3.41 (t, 3H, J = 6.8 Hz), 3.74 (s, 3H), 3.90 (s, 3H), 4.17 (s, 2H), 4.81 (s, 2H). ^{13}C NMR δ 13.7, 20.1, 31.3, 51.2, 52.0, 52.3, 67.5, 84.3, 110.2, 131.9, 164.5, 166.3. Anal. Calcd. for $C_{12}H_{19}NO_5$ C 56.02; H 7.44; N 5.44; O 31.09. Found: C 56.00; H 7.38; N 5.39; O 31.02.

4.2.8 Dimethyl 3-cyclohexyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (8)

Pale yellow solid (m.p. = 191–192 °C). IR = 1,650 cm^{-1}. ^1H NMR 1.09–1.33 (m, 10H), 2.89–2.96 (m, 1H), 3.75 (s, 3H), 3.87 (s, 3H), 4.31 (s, 2H), 4.82 (s, 2H). ^{13}C NMR δ 25.6, 26.7, 32.7, 51.1, 52.3, 57.5, 67.2, 82.3, 110.7, 131.0, 163.9, 166.3. Anal. Calcd. for $C_{14}H_{21}NO_5$ C 59.35; H 7.47; N 4.94; O 28.24. Found: C 59.31; H 7.42; N 4.99; O 28.20.

4.2.9 Diethyl 3-phenyl-3,6-dihydro-2H-1,3-oxazine-4, 5-dicarboxylate (9)

Pale yellow solid (m.p. = 234–235 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{16}H_{19}NO_5$ C 62.94; H 6.27; N 4.59; O 26.20. Found: C 62.99; H 6.21; N 4.54; O 26.29.

4.2.10 Diethyl 3-(4′-nitro)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (10)

Orange solid (m.p. = 245–247 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{16}H_{18}N_2O_7$ C 54.86; H 5.18; N 8.00; O 31.97. Found: C 54.80; H 5.21; N 7.93; O 31.90.

4.2.11 Diethyl 3-(4′-methoxy)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (11)

Pale reddish solid (m.p. = 189–191 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{17}H_{21}NO_6$ C 60.89; H 6.31; N 4.18; O 28.63. Found: C 60.81; H 6.34; N 4.12; O 28.69.

4.2.12 Diethyl 3-(4′-bromo)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (12)

Pale red solid (m.p. = 189–190 °C). Analytical data were in full agreement for those already reported in the literature for the same compound [32]. Anal. Calcd. for $C_{16}H_{18}BrNO_5$ C 50.02; H 4.72; N 3.65; O 20.82. Found: C 50.07; H 4.67; N 3.60; O 20.78.

4.2.13 Diethyl 3-(2′-methoxy)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (13)

Red solid (m.p. = 171–173 °C. IR = 1,650 cm^{-1}. ^1H NMR δ 1.19 (t, 3H, J = 7.1 Hz), 1.28 (t, 3H, J = 7.0 Hz), 3.86 (s, 3H), 4.11 (q, 2H, J = 7.1 Hz), 4.22 (q, 2H,

J = 7.0 Hz), 4.75 (s, 2H), 4.92 (s, 2H), 6.50–7.78 (m, 4H). ^{13}C NMR δ 14.0, 14.1, 55.4, 60.2, 61.3, 68.0, 82.9, 113.7, 116.6, 125.4, 126.3, 130.5, 131.1, 131.4, 132.4, 151.3, 166.3, 166.5. Anal. Calcd. for $C_{17}H_{21}NO_6$ C 60.89; H 6.31; N 4.18; O 28.63. Found: C 60.85; H 6.37; N 4.11; O 28.65.

4.2.14 Diethyl 3-(2′-nitro)phenyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (14)

Orange solid (m.p. = 227–228 °C). IR = 1655, 1553, 1307 cm^{-1}. ^{1}H NMR δ 1.21 (t, 3H, J = 7.2 Hz), 1.29 (t, 3H, J = 7.1 Hz), 4.09 (q, 2H, J = 7.2 Hz), 4.18 (q, 2H, J = 7.1 Hz), 4.79 (s, 2H), 5.04 (s, 2H), 7.31–8.04 (m, 4H). ^{13}C NMR δ 14.2, 14.3, 55.4, 60.2, 61.5, 67.4, 78.8, 116.4, 120.2, 126.0, 130.4, 130.9, 131.5, 131.8, 135.9, 166.0, 166.9. Anal. Calcd. for $C_{16}H_{18}N_2O_7$ C 54.86; H 5.18; N 8.00; O 31.97. Found: C 54.82; H 5.14; N 7.98; O 31.92.

4.2.15 Diethyl 3-nbutyl-3,6-dihydro-2H-1,3-oxazine-4, 5-dicarboxylate (15)

Pale yellow solid (m.p. = 182–184 °C). IR = 1,650 cm^{-1}. ^{1}H NMR δ 0.95 (t, 3H, J = 5.2 Hz), 1.20 (t, 3H, J = 7.0 Hz), 1.31 (t, 3H, J = 7.0 Hz), 1.94 (m, 2H), 1.40 (m, 2H), 3.42 (t, 2H, J = 6.7 Hz), 4.10 (q, 2H, J = 7.0 Hz), 4.21 (q, 2H, J = 7.0 Hz), 4.25 (s, 2H), 4.88 (s, 2H). ^{13}C NMR δ 13.8, 14.2, 14.3, 20.2, 31.5, 51.9, 60.1, 61.2, 67.7, 84.2, 110.8, 132.9, 165.7, 166.5. Anal. Calcd. for $C_{14}H_{23}NO_5$ C 58.93; H 8.12; N 4.91; O 28.04. Found: C 58.90; H 8.09; N 4.88; O 28.01.

4.2.16 Diethyl 3-cyclohexyl-3,6-dihydro-2H-1, 3-oxazine-4,5-dicarboxylate (16)

Pale yellow solid (m.p. = 199–202 °C). IR = 1,650 cm^{-1}. ^{1}H NMR δ 1.10–1.95 (m, 10H), 1.18 (t, 3H, J = 7.1 Hz), 1.32 (t, 3H, J = 7.1 Hz), 2.92–2.96 (m, 1H), 4.10 (q, 2H, J = 7.1 Hz), 4.22 (q, 2H, J = 7.1 Hz), 4.44 (s, 2H), 4.76 (s, 2H). ^{13}C NMR δ 14.0, 14.1, 25.4, 26.7, 32.9, 57.4, 60.2, 61.1, 67.3, 82.0, 111.4, 131.9, 164.5, 166.6. Anal. Calcd. for $C_{16}H_{25}NO_5$ C 61.72; H 8.09; N 4.50; O 25.69. Found: C 61.77; H 8.01; N 4.44; O 25.65.

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