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A general and efficient method for synthesis of enaminones and enamino esters catalyzed by NbCl₅ under solvent-free conditions

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Abstract A general and simple procedure was developed for the synthesis of β -enaminones and β -enamino esters by reacting 1,3-dicarbonyl compounds with amines in the presence of catalytic amounts of niobium pentachloride. The reaction proceeds smoothly at room temperature under solvent-free conditions and leads to chemo- and regioselective formation of enamine derivatives in high to excellent yields.

Keywords 1,3-Dicarbonyl compounds $\cdot \beta$ -Enaminones $\cdot \beta$ -Enamino esters \cdot Niobium pentachloride

Introduction

 β -Enaminones and β -enamino esters have been extensively used as key intermediates in organic synthesis [1–6]. In particular, they have been employed as synthons of a wide variety of heterocycles [7], pharmaceutical compounds having anti-epileptic [8], molluscicidal and larvicidal activities [9], and naturally occurring alkaloids [10]. The conventional approach for the preparation of β -enaminones and β -enamino esters is direct condensation of 1,3-dicarbonyl compounds with amines in refluxing aromatic solvent with azeotropic removal of water. In recent years, it has been reported that the use of catalysts enables the reaction to be performed under milder conditions. A variety of catalysts

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P. Wang · G.-T. Cheng Hebei Chemical and Pharmaceutical Vocational Technology College, Shijiazhuang 050026, China such as $CoCl_2 \cdot 6H_2O$ [11], $Cu(NO_3)_2 \cdot 3H_2O$ [12], $InBr_3$ [13], ZrCl₄ [14], ZrOCl₂ $\cdot 8H_2O$ [15], $SnCl_4 \cdot 5H_2O$ [16], NH_4 $Ce(NO_3)_5$ [17, 18], $Ni(OAc)_2$ [19], $Zn(OAc)_2 \cdot 2H_2O$ [20], I_2 [21, 22], B_2O_3/Al_2O_3 [23], $SbCl_3/SiO_2$ [24], $HCIO_4/SiO_2$ [25], P_2O_5/SiO_2 [26], silica chloride [27], sulfamic acid [28], L-proline [29], phosphotungstic acid [30], and Ag [31] or Cu nanoparticles [32] have been used to promote this transformation. Despite these advances, there were always one or more drawbacks with these procedures, such as the use of expensive or less readily available reagents, rigorous reaction conditions, longer reaction times, unsatisfactory yields, low selectivity, or the use of toxic solvents that limit these methods to small-scale synthesis. Thus, searching for a new catalyst and procedure is still of practical importance.

Recently, for environmental and economic reasons, attention has been focused on catalytic reactions under solvent-free conditions [33, 34]. NbCl₅ has been considered as an effective Lewis acid catalyst for a variety of organic transformations, such as trimethylsilyl protection of hydroxyl groups [35], synthesis of α -aminophosphonates [36], quinoxaline derivatives [37], diaminotriarylmethanes [38], bis(indolyl)alkanes [39], 1,1-diacetates [40], and 1,5-benzodiazepine derivatives [41]. As part of our continuing interest in the development of new synthetic methodologies [42–47], herein, we wish to report a mild and efficient method for the chemo- and regioselective examination of 1,3-dicarbonyl compounds in the presence of catalytic amounts of NbCl₅ (Scheme 1).

Results and discussion

The Lewis acid-catalyzed condensation of amines with β ketoesters or β -diketones has recently been described. Vohra and coworkers reported that the reaction of

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Scheme 1

acetylacetone and aniline was performed in CH_2Cl_2 in the presence of $Zn(OAc)_2 \cdot 2H_2O$ and $MgSO_4$; the corresponding product (Table 1, **3a**) was obtained in 86 % yield after 2 days [20]. When this reaction was carried out in the presence of NbCl₅ under solvent-free conditions at room temperature, the desired product was formed in 96 % yield within 5 min. The effect of the amount of catalyst on the

Table 1 Synthesis of β -enaminones and β -enamino esters catalyzed by NbCl₅

Entry	Amine	\mathbb{R}^2	R ³	Product	Time/min	Yield/% ^a	M.p. (lit. m.p.)/°C
1	PhNH ₂	Me	Me	3 a	5	96	49-50 (49-50 [34])
2	2-MeC ₆ H ₄ NH ₂	Me	Me	3b	5	95	38-39 (39-40 [34])
3	4-MeC ₆ H ₄ NH ₂	Me	Me	3c	5	96	58-59 (59-60 [34])
4	2-MeOC ₆ H ₄ NH ₂	Me	Me	3d	15	94	50-51 (51-52 [34])
5	4-MeOC ₆ H ₅ NH ₂	Me	Me	3e	4	95	41-43 (41-42 [13])
6	4-EtOC ₆ H ₅ NH ₂	Me	Me	3f	5	93	39-40 (40-41 [13])
7	3-ClC ₆ H ₄ NH ₂	Me	Me	3g	120	81	39-40 (40-42 [13])
8	4-ClC ₆ H ₄ NH ₂	Me	Me	3h	110	82	60-62 (61-62 [34])
9	2-BrC ₆ H ₄ NH ₂	Me	Me	3j	300	80	72–73 (71–72 [34])
10	4-BrC ₆ H ₄ NH ₂	Me	Me	3i	150	75	48-50 (49-51 [49])
11	3-CF ₃ C ₆ H ₄ NH ₂	Me	Me	3k	360	72	62.3-62.8 (62-63 [48])
12	Furan-2-amine	Me	Me	31	6	94	Oil (oil [32])
13	1-Naphthylamine	Me	Me	3m	60	85	61-63 (62-64 [23])
14	CH ₃ (CH ₂) ₃ NH ₂	Me	Me	3n	3	95	Oil (oil [13])
15	H ₂ C=CHCH ₂ NH ₂	Me	Me	30	5	93	Oil (oil [13])
16	Morpholine	Me	Me	3p	120	75	45-47 (46-47 [13])
17	Piperidine	Me	Me	3q	120	72	45-46 (46-47 [13])
18	(R)-PhCHMeNH ₂	Me	Me	3r	5	93	Oil (oil [13])
19	PhNH ₂	CH ₂ CH ₂ CH ₂		3s	50	80	179–180 (178–179 [26])
20	CH ₃ (CH ₂) ₃ NH ₂	CH ₂ CH ₂ CH ₂		3t	10	95	Oil (oil [26])
21	PhNH ₂	Ph	Me	3u	60	82	96-97 (95-97 [13])
22	PhNH ₂	OMe	Me	3v	7	96	46-47 (45-46 [13])
23	2-MeOC ₆ H ₄ NH ₂	OMe	Me	3w	20	90	28-30 (29-30 [11])
24	4-MeC ₆ H ₄ NH ₂	OMe	Me	3x	6	94	57-58 (56-57 [34])
25	4-ClC ₆ H ₄ NH ₂	OMe	Me	3у	150	80	60-61 (61-62 [13])
26	4-BrC ₆ H ₄ NH ₂	OMe	Me	3z	200	84	48-50 (44-48 [49])
27	PhCH ₂ NH ₂	OMe	Me	3aa	4	95	36-37 (37-38 [13])
28	Cyclohexanamine	OMe	Me	3ab	5	93	Oil (oil [13])
29	H ₂ C=CHCH ₂ NH ₂	OMe	Me	3ac	5	92	Oil (oil [13])
30	PhNH ₂	OEt	Me	3ad	5	96	Oil (oil [13])
31	PhNH ₂	OCH ₂ CHMe ₂	Me	3ae	20	93	Oil
32	PhNH ₂	OCH ₂ CH ₂ OMe	Me	3af	20	94	Oil
33	PhNH ₂	OCH ₂ CH=CH ₂	Me	3ag	20	96	Oil
34	PhNH ₂	OMe	Et	3ah	20	92	Oil
35	4-H ₂ NC ₆ H ₄ NH ₂	Me	Me	3ai	20	85	185–187 (186–188 [23])
36	H ₂ N(CH ₂) ₃ NH ₂	Me	Me	3aj	10	96	51-52 (52-53 [13])
37	H ₂ N(CH ₂) ₆ NH ₂	Me	Me	3ak	30	95	82-84
38	$H_2N(CH_2)_6NH_2$	OMe	Me	3al	20	94	71–73
39	$H_2N(CH_2)_2NH_2$	OCH ₂ CHMe ₂	Me	3am	12	96	52–54
40	$H_2N(CH_2)_2NH_2$	OCH ₂ CH=CH ₂	Me	3an	10	95	81-83

^a Isolated yields

yield was investigated. It was found that there was no obvious difference between 1 mol% and 2 mol% catalyst loading. The use of lower catalyst loading (0.1 mol%) required a longer time period (1 h) to afford a comparable result. So we inferred that 1 mol% of NbCl₅ was sufficient for this reaction. This reaction was also examined in various solvents, such as dichloromethane, acetonitrile, ethanol, and methanol, in the presence of 1 mol% of NbCl₅. The results showed that the presence of additional solvents made the reaction rate lower. Therefore, we chose to perform this reaction under solvent-free conditions in the presence of 1 mol% NbCl₅ at room temperature.

Splendidly, the scope of this protocol was extensively broad and versatile, spanning a wide range of amines and 1,3-dicarbonyl compounds. As shown in Table 1, a series of aromatic amines containing both electron-withdrawing and donating substituents were treated with acetylacetone, and the corresponding β -enaminones were obtained in good to excellent yields. Aromatic amines with electronwithdrawing groups such as -Cl, -Br, and -CF₃ showed weaker reactivity than those containing electron-neutral or electron-donating groups. It should be noted that orthosubstituted anilines such as 2-methoxyaniline (Table 1, entry 4) and 2-bromoaniline (Table 1, entry 9) gave lower yields than the others, which may be due to the steric hindrance effect. The mildness of the reaction conditions permits furan-2-amine to undergo the reaction successfully to produce the desired product in high yield without formation of any polymerization product (Table 1, entry 12). Aliphatic amines and allylic amine were shown to be reactive to undergoing the title reaction, and a shorter reaction time was required compared to aromatic amines. It is noteworthy that optically active (R)-1-phenylethanamine was converted successfully into the corresponding β enaminone (Table 1, entry 18) without any racemization or inversion by measuring its optical rotation and comparing with the literature value [13]. The reactions worked also well when secondary amines like morpholine and piperidine were utilized. In addition, cyclic 1,3-dicarbonyl compounds such as cyclohexane-1,3-dione underwent similar reactions, leading to the corresponding β -enaminone in high yields (Table 1, entries 19 and 20). In the case of unsymmetrical diketones such as 1-phenylbutane-1,3dione, the regiochemistry was controlled by the more reactive carbonyl group, which underwent the attack by aniline to give exclusively 1-phenyl-3- (phenylamino)but-2-en-1-one (3u).

With the successful enamination of diketones, we further studied the reaction of amines and methyl acetoacetate under similar conditions. It was found that the corresponding β -enamino esters were obtained in high to excellent yields. The different reactivities of the anilines were dependent on the substituents on the benzene ring. It could also be concluded that anilines bearing electronwithdrawing groups required longer time and gave lower yields. The use of other β -ketoesters such as ethyl acetoacetate, isobutyl acetoacetate, 2-methoxyethyl acetoacetate, and allyl acetoacetate was also effective. The alkoxy (-R₂) moiety present had little influence on the reaction, and generally high yields were obtained.

The method was found to be chemoselective. Since a keto carbonyl group is more electrophilic than an ester group, the amine attacks only at the ketone carbonyl for β -ketoesters. The *Z* selectivity in the products derived from acyclic diketones and β -ketoesters was confirmed by ¹H NMR spectra. The proton of the –NH group appearing at a lower field ($\delta > 8.6$ ppm) indicated the formation of an intramolecular bond, which stabilized the products. However, the β -enaminones derived from cyclic diketones such as 1,3-cyclohexadione displayed ¹H NMR spectra having the signals for the non-hydrogen bonded proton of the –NH group in the region of $\delta = 4.5-6.5$ ppm, thus indicating the *E* configuration [10].

Encouraged by the above interesting results, we also attempted to prepare bis-enaminones or bis-enamino esters to further broaden the scope. To our delight, the reaction of diamines with two equiv. of 1,3-dicarbonyl compounds resulted in the expected products (Table 1, **3aj–3an**) in high yield. Moreover, the reaction of acetylacetone and aniline was carried out on a scale of 100 mmol. As expected, the reaction proceeded nicely to afford the desired product in 92 % yield in 10 min.

Finally, in order to show the merit of the present work we compared this method with the reported results in the literature (Table 2), which showed that NbCl₅ is the most efficient catalyst with respect to the reaction time, the amount of catalyst, and yield of product. In addition, the toxicity of niobium salts is not high (LD_{50} orally in rats 725 mg Nb/kg) [50].

Conclusion

We developed a simple and efficient method of synthesis of β -enaminones and β -enamino esters by the reaction of 1,3dicarbonyl compounds with amines in the presence of catalytic amounts of NbCl₅ under solvent-free conditions. The mild conditions, short times, simple workup, high yield, and chemo- and regioselectivity are the salient features of this procedure.

Experimental

All solvents and chemicals were obtained commercially and were used as received. Melting points were determined

Table 2 Comparison of the efficiency of NbCl5 with reported catalysts for the synthesis of 4-(phenylamino)pent-3-en-2-one (3a)

Catalyst/solvent	Catalyst load	Time	Yield/%	Ref.
CoCl ₂ ·6H ₂ O	5 mol%	15 min	95	[11]
Cu(NO ₃) ₂ ·3H ₂ O/solvent-free	10 mol%	10 min	92	[12]
InBr ₃ /solvent-free	1 mol%	10 min	94	[13]
ZrCl ₄ /solvent-free	1 mol%	12 min	96	[14]
ZrOCl ₂ ·8H ₂ O/solvent-free	2 mol%	10 min	95	[15]
SnCl ₄ ·5H ₂ O/solvent-free	2 mol%	15 min	95	[16]
Ni(OAc) ₂ /solvent-free	5 mol%	9 min	98	[19]
Zn(OAc) ₂ ·2H ₂ O/CH ₂ Cl ₂	5 mol%	2 days	86	[20]
I ₂ /solvent-free	20 mol%	3 min	79	[22]
B ₂ O ₃ /Al ₂ O ₃ /solvent-free	15 % (w/w)	2 h	87	[23]
HClO ₄ ·SiO ₂ /solvent-free	50 mg/mol	14 min	98	[25]
Silica chloride/solvent-free	10 % (w/w)	5 min	91	[27]
L-Proline/solvent-free	5 mol%	4 h	85	[29]
Phosphotungstic acid/solvent-free	1 mol%	15 min	95	[30]
Ag nanoparticles/MeOH	20 mol%	8 h	90	[31]
Cu nanoparticles/MeOH	10 mol%	2.5 h	92	[32]
NbCl ₅ /solvent-free	1 mol%	5 min	96	This work

by using an X-4 apparatus. IR spectra were recorded using a Bruker-TENSOR 27 spectrometer. NMR spectra were taken with a Bruker DRX-500 spectrometer at 500 MHz (¹H) and 125 MHz (¹³C) using CDCl₃ as the solvent. Elemental analyses were obtained on a Vario EL III CHNOS elemental analyzer, and the results obtained agreed favorably with calculated values.

Typical experimental procedure for the preparation of enaminones or enamino esters

A mixture of amine (5 mmol), 1,3-dicarbonyl compound (5 mmol), and NbCl₅ (0.05 mmol) in a round-bottom flask was stirred with a magnetic stirrer at room temperature. When the two substrates were not liquids, the mixture was ground with a pestle in the mortar. After completion of the reaction as monitored by thin-layer chromatography (TLC), the mixture was diluted with 10 cm^3 ethyl acetate and washed with 20 cm³ water, and the aqueous layer was then extracted with ethyl acetate $(2 \times 10 \text{ cm}^3)$. The combined organic layer was dried over MgSO4 and concentrated under vacuum to give the product in almost pure form. Further purification was carried out by recrystallization from hexane-ether if the product was solid or by short column chromatography on silica gel eluting with ethyl acetate/petroleum ether (2:8 v/v).

Isobutyl 3-(phenylamino)-2-butenoate (3ae, C14H19NO2) IR (KBr): $\bar{\nu} = 3,202, 2,960, 2,937, 1,651, 1,622, 1,596,$ 1,583, 1,504, 1,384, 1,271, 1,230, 1,161, 1,055, 1,008, 908, 786, 752, 698 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): $\delta = 0.96$ (d, J = 7.0 Hz, 6H), 1.92–1.98 (m, 1H), 1.99 (s, 3H), 3.88 (d, J = 7.0 Hz, 2H), 4.72 (s, 1H), 7.08 (d, J = 7.5 Hz, 2H), 7.15 (t, J = 7.5 Hz, 1H), 7.32 (t, J = 7.5 Hz, 2H), 10.38 (br s, 1H, NH) ppm; ¹³C NMR $(125 \text{ MHz}, \text{CDCl}_3)$: $\delta = 19.3, 20.4, 26.1, 69.2, 86.2, 124.5,$ 125.0, 129.2, 139.5, 159.0, 170.6 ppm.

2-Methoxyethyl 3-(phenylamino)-2-butenoate (**3af**, C₁₃H₁₇NO₃)

IR (KBr): $\bar{v} = 3,257, 3,188, 3,034, 2,927, 1,651, 1,616,$ 1,597, 1,585, 1,501, 1,440, 1,385, 1,359, 1,271, 1,230, 1,165, 1,064, 1,028, 983, 786, 752, 698 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): $\delta = 1.96$ (s, 3H), 3.37 (s, 3H), 3.59 (t, J = 7.5 Hz, 2H), 4.24 (t, J = 7.5 Hz, 2H), 4.75 (s, 1H), 7.05 (d, J = 7.5 Hz, 2H), 7.12 (t, J = 7.5 Hz, 1H), 7.29 (t, J = 7.5 Hz, 2H), 10.38 (br s, 1H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 20.2$, 58.9, 61.8, 70.9, 85.8, 124.3, 125.0, 129.0, 139.2, 159.2, 170.0 ppm.

Allyl 3-(phenylamino)-2-butenoate (**3ag**, C₁₃H₁₅NO₂)

IR (KBr): $\bar{v} = 3,257, 3,188, 3,033, 2,928, 1,660, 1,614,$ 1,597, 1,585, 1,504, 1,441, 1,385, 1,354, 1,269, 1,228, 1,161, 1,053, 1,029, 995, 786, 752, 698 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): $\delta = 2.02$ (s, 3H), 4.64 (d, J = 5.5 Hz, 2H), 4.77 (s, 1H), 5.24 (dd, J = 10.5, 1.5 Hz, 1H), 5.36 (dd, J = 17.5, 1.5 Hz, 1H), 5.96-6.04 (m, 1H), 7.10 (d, J)J = 7.5 Hz, 2H), 7.17 (t, J = 7.5 Hz, 1H), 7.34 (t, J = 7.5 Hz, 2H), 10.40 (br s, 1H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 20.3$, 63.6, 85.7, 117.2, 124.5, 125.1, 129.1, 133.4, 139.3, 159.4, 170.0 ppm.

Methyl 3-(phenylamino)-2-pentenoate (**3ah**, C₁₂H₁₅NO₂) IR (KBr): $\bar{\nu} = 3,312, 2,978, 2,947, 1,654, 1,612, 1,595, 1,583, 1,501, 1,379, 1,267, 1,228, 1,164, 1,037, 1,003, 895, 786, 750, 698 cm⁻¹; ¹H NMR (500 MHz, CDCl₃):$ $<math>\delta = 1.03$ (t, J = 7.5 Hz, 3H), 2.32 (q, J = 7.5 Hz, 2H), 3.69 (s, 3H), 4.74 (s, 1H), 7.09 (d, J = 7.5 Hz, 2H), 7.17 (t, J = 7.5 Hz, 1H), 7.32 (t, J = 7.5 Hz, 2H), 10.31 (br s, 1H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 12.4, 25.5, 50.3, 83.3, 125.1, 125.3, 129.1, 139.2, 165.0, 171.1 ppm.$

4,4'-(1,6-Hexanediyldiimino)bis(3-penten-2-one)

$(3ak, C_{16}H_{28}N_2O_2)$

White solid; IR (KBr): $\bar{\nu} = 2,992, 2,934, 2,860, 1,606, 1,568, 1,431, 1,356, 1,290, 1,222, 1,103, 1,034, 984, 765 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): <math>\delta = 1.39-1.42$ (m, 4H), 1.59 (quin, J = 7.0 Hz, 4H), 1.90 (s, 6H), 1.99 (s, 6H), 3.22 (q, J = 7.0 Hz, 4H), 4.95 (s, 2H), 10.86 (br s, 2H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 18.8, 26.5, 28.7, 29.9, 42.8, 95.1, 163.1, 194.7$ ppm.

Dimethyl 3,3'-(1,6-hexanediyldiimino)bis(2-butenoate) (**3al**, C₁₆H₂₈N₂O₄)

White solid; IR (KBr): $\bar{\nu} = 3,276, 2,936, 2,860, 1,647, 1,593, 1,427, 1,265, 1,226, 1,163, 1,111, 1,049, 932, 781 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): <math>\delta = 1.39$ (quin, J = 7.0 Hz, 4H), 1.58 (quin, J = 7.0 Hz, 4H), 1.91 (s, 6H), 3.17 (q, J = 7.0 Hz, 4H), 3.70 (s, 6H), 4.83 (s, 2H), 9.74 (br s, 2H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 19.4, 26.5, 30.3, 42.8, 49.8, 81.5, 162.0, 170.9$ ppm.

Diisobutyl 3,3'-(1,2-ethanediyldiimino)bis(2-butenoate) (**3am**, $C_{18}H_{32}N_2O_4$)

White solid; IR (KBr): $\bar{\nu} = 3,267, 2,966, 1,643, 1596,$ 1496, 1367, 1265, 1226, 1,168, 1,114, 1,055, 1,006, 787 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): $\delta = 0.92$ (d, J = 6.5 Hz, 12H), 1.91 (s, 6H), 2.28–2.34 (m, 2H), 3.36 (t, J = 6.0 Hz, 4H), 3.80 (d, J = 6.5 Hz, 4H), 4.51 (s, 2H), 8.64 (br s, 2H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): = 19.3, 28.0, 43.9, 68.8, 71.5, 83.6, 161.3, 170.7 ppm.

Diallyl 3,3'-(1,2-ethanediyldiimino)bis(2-butenoate) (**3an**, $C_{16}H_{24}N_2O_4$)

White solid; IR (KBr): $\bar{\nu} = 3,271$, 3,086, 2,883, 1,649, 1,589, 1,497, 1,259, 1,228, 1,172, 1,111, 1,053, 997, 783 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): $\delta = 1.92$ (s, 6H), 3.36 (d, J = 6.0 Hz, 4H), 4.54 (d, J = 6.0 Hz, 4H), 4.55 (s, 2H), 5.19 (dd, J = 10.5, 1.5 Hz, 2H), 5.29 (dd, J = 17.5, 1.5 Hz, 2H), 5.90–5.98 (m, 2H), 8.64 (br s, 2H, NH) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 19.3$, 43.8, 63.4, 83.3, 117.1, 133.5, 161.7, 170.1 ppm.

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