Regio- and stereoselective synthesis of α,β-dehydro-β-amino esters

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A facile method for the regio- and stereoselective synthesis of α,β-dehydro-β-amino esters from acetates of Baylis-Hillman adducts with amines was reported. In the absence of any solvent and catalyst, the present strategy works well for a series of electron deficient and electron rich aromatic amines as well as aliphatic ones.

Keywords Baylis–Hillman adduct, amine, α,β-dehydro-β-amino ester, solvent-free, catalyst-free

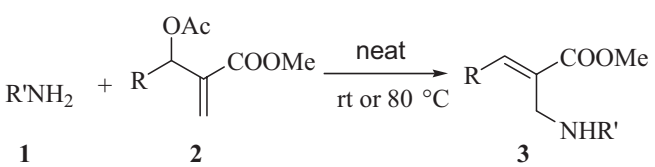
α,β-Dehydro-β-amino esters are important building blocks for the synthesis of indenoquinoline derivatives, nonproteinogenic β-amino acids and β-lactams, which facilitates the development of modern medicines.[1-4] Peptide derivatives of α,β-dehydro-β-amino acids also exhibit intramolecular hydrogen bonding, which plays a significant role in biochemical molecular recognition and mimics a β-turn,[5-8] and α,β-dehydro-β-amino acids can provide constraint after incorporation into a peptide molecule and thus renders a peptide more favourable to bonding to its target.[9,10] Note that the β-amino acids often require synthesis when they are not available commercially.[11] Hence, the development of various methodologies for the synthesis of α,β-dehydro-β-amino esters, β-amino acids or esters has been disclosed.[12-28,7] Among these protocols, using readily available Baylis–Hillman adducts as substrates provide a practical and straightforward choice. However, many suffer from long reaction times, low regioselectivity and low yields, the basic conditions, and the use of harmful solvents. Recently, palladium-catalysed synthesis of α,β-dehydro-β-amino esters from Baylis–Hillman adducts with amines contributes an interesting procedure, but the shortage of this approach is the use of expensive palladium reagent, air-sensitive phosphine ligand as well as inert atmosphere.[7] Recently, α,β-dehydro-β-amino esters have also been synthesised from acetates of Baylis–Hillman adducts with amines in the presence of cerium (IV) ammonium nitrate at nitrogen atmosphere.[20] So, the development of efficient and practical methods for the synthesis of α,β-dehydro-β-amino esters is still desirable.

Baylis–Hillman adducts are of valuable synthetic intermediates for the synthesis of a number of complex molecules, biologically active molecules as well as natural products.[29-38] Recently, we have described applications of Baylis–Hillman adducts and many excellent results were also demonstrated.[39-44] As a continuation of our previous work, herein, we disclose a facile, regio- and stereoselective synthesis of α,β-dehydro-β-amino esters by the reaction of Baylis–Hillman acetates with various primary amines under the catalyst- and solvent-free conditions. (Scheme 1). To the best of our knowledge, this is first report of the synthesis of α,β-dehydro-β-amino esters under the neat condition.

Initially, we used *p*-methoxyaniline **1a** and Baylis–Hillman adduct **2a** as model substrate. In a typical experiment, 1.5 mmol of **1a** was added to 1 mmol of Baylis–Hillman adduct **2a** under solvent- and catalyst-free conditions, and the mixture was then stirred at 80 °C until reaction completion. This model reaction finished within 30 minutes and the corresponding α,β-dehydro-β-amino ester **3a** was afforded in 91% yield (Table 1, entry 1). Furthermore, high *E*-stereoselectivity was observed from the ^{1}H NMR spectrum of **3a**. In contrast, when CH_3CN and THF were used as solvent in this model reaction, **3a** is obtained in 83% and 70% yields respectively together with prolonged reaction time (2 h). During our investigation, we also found that the reaction temperature played an important role and the reaction became quite sluggish together with low yields at room temperature.

Encouraged by these experimental results, a series of anilines **1** were treated with Baylis–Hillman adducts **2** at 80 °C under catalyst- and solvent-free conditions. As shown in Table 1, various aromatic amines underwent smooth conversion to corresponding α,β-dehydro-β-amino esters **3a–l** in good to excellent yields. Note that the behaviour of the present strategy is quite dependent on the type of substrate anilines. The substituent groups of anilines affected the conversion dramatically. As we can see, electron-donating groups such as methyl and methoxy groups add the nucleophilicity of aromatic amines and the formation of corresponding α,β-dehydro-β-amino ester thus facilitated (Table 1, entries 1–3, 6–9). In contrast, the presence of electron-withdrawing groups such as chloro and bromo groups obviously decreased the nucleophilicity of aromatic amines and the conversion became more sluggish (entries 10–12). In addition, the good *E*-stereoselectivity was demonstrated in most cases and no side products were observed. Apart from the electronic effect, the influence of steric hindrance on the transformation seemed to disappear completely (entries 6–8).

To establish the generality of the present transformation, a series of aliphatic amines were then tested with Baylis–Hillman adducts. Blending of 1.5 mmol of benzylamine **1g** and 1 mmol of Baylis–Hillman adduct **2a** at room temperature resulted in the formation of both α,β-dehydro-β-amino ester and bis-allylic tertiary amine in 45 and 50% yield respectively. Indeed, the aliphatic substrate was so reactive that the corresponding conversion was finished in 2 minutes due to the enhanced nucleophilicity of amino group. Subsequently, various experimental parameters including temperature and substrate ratio were screened. We found that the excessive use



R = aryl; R' = alkyl, aryl

Scheme 1

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Table 1 Regioselective synthesis of α,β-dehydro-β-amino esters[a]

Entry	Amine **1**	R	Time	Yield/%[b]	Product	*E*/*Z*[c]
1	4-$MeOC_6H_4NH_2$	Ph	0.5 h	91	**3a**	92:8
2	4-$MeOC_6H_4NH_2$	4-MeC_6H_4	0.5 h	93	**3b**	93:7
3	4-$MeOC_6H_4NH_2$	4-ClC_6H_4	0.5 h	98	**3c**	93:7
4	$C_6H_4NH_2$	4-ClC_6H_4	2 h	88	**3d**	77:23
5	$C_6H_4NH_2$	4-$NO_2C_6H_4$	2 h	90	**3e**	70:30
6	2-$MeOC_6H_4NH_2$	Ph	2 h	82	**3f**	93:7
7	2-$MeOC_6H_4NH_2$	4-MeC_6H_4	2 h	90	**3g**	86:14
8	2-$MeOC_6H_4NH_2$	4-ClC_6H_4	2 h	87	**3h**	85:15
9	4-$MeC_6H_4NH_2$	4-ClC_6H_4	2 h	95	**3i**	80:20
10	4-$ClC_6H_4NH_2$	4-$NO_2C_6H_4$	4 h	92	**3j**	80:20
11	4-$ClC_6H_4NH_2$	4-MeOPh	4 h	83	**3k**	84:16
12	4-$BrC_6H_4NH_2$	4-$NO_2C_6H_4$	4 h	91	**3l**	82:18
13	$C_6H_5CH_2NH_2$	Ph	2 min	95	**3m**	95:5
14	$C_6H_5CH_2NH_2$	4-MeC_6H_4	2 min	93	**3n**	93:7
15	$C_6H_5CH_2NH_2$	4-ClC_6H_4	2 min	93	**3o**	90:10
16	$C_6H_5(CH_3)CHNH_2$	Ph	2 min	92	**3p**	100:0
17	*t*-$BuNH_2$	Ph	2 min	96	**3q**	100:0
18	Cyclohexylamine	Ph	2 min	83	**3r**	86:14
19	$(CH_3)_2CHNH_2$	Ph	2 min	97	**3s**	86:14

[a]Method **A**: the reaction of aromatic amines with Baylis–Hillman adducts.
Method **B**: the reaction of aliphatic amines with Baylis–Hillman adducts.
[b]Isolated yields. [c] The ratio was determined by the ^{1}H NMR spectrum.

of benzylamine **1g** (3 equiv.) could form α,β-dehydro-β-amino ester exclusively. Then, several structurally diversed aliphatic primary amines experienced the similar conversion (Table 1, **1g–k**). As can be seen, the excellent chemoselectivity and the good stereoselectivity were achieved in all cases (Table 1, **3m–s**).

In conclusion, we have developed a facile, regio- and stereoselective synthesis of α,β-dehydro-β-amino esters in good to excellent yields from the reaction of various primary amines with Baylis–Hillman adducts under solvent- and catalyst-free conditions. We believe that our method will find its use in organic synthesis, especially in large-scale industrial preparation.

Experimental

All ^{1}H NMR spectra were measured in $CDCl_3$ and recorded on Bruker AC – 500 (500 MHz) spectrometer with TMS as the internal standard. IR spectra were measured with a Perkin–Elmer FT-IR-1600 spectrometer. Elemental analyses were performed on Elemantar Vario EL instrument.

General procedure

*Method **A*:** To magnetically stirred 1.5 mmol of aromatic amines **1** in an oven-dried glassware, 1 mmol of Baylis–Hillman adducts **2** was added. The mixtures were stirred at 80 °C until the completion of reactions (monitored by TLC). Then the mixture was purified by silica gel column chromatography eluting with hexane/ethyl acetate to afford the corresponding products (**3a–l**). All of the products were identified by IR and ^{1}H NMR spectra. The new compounds were also identified by elemental analysis.

*Method **B*:** To magnetically stirred 3 mmol of aliphatic amines **1** in an oven-dried glassware, 1 mmol of Baylis–Hillman adducts **2** was added. The mixtures were stirred at room temperature until the completion of reactions (monitored by TLC). Then the mixture was purified by silica gel column chromatography eluting with hexane/ethyl acetate to afford the corresponding products (**3m–s**). All of the products were identified by IR and ^{1}H NMR spectra. The new compounds were also identified by elemental analysis.

3a: Oil (lit.[7]); IR(KBr)/cm^{-1}: 3383, 1709, 1625; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.87 (s, 1H), 7.45–7.37 (m, 5H), 6.73 (d, 2H, *J* = 9.0 Hz), 6.50 (d, 2H, *J* = 9.0 Hz), 3.81 (s, 3H), 3.72 (s, 3H).

3b: IR (KBr)/cm^{-1}: 3380, 1694, 1605; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.84 (s, 1H), 7.35 (d, 2H, *J* = 8.0 Hz), 7.19 (d, 2H, *J* = 8.0 Hz), 6.74 (d, 2H, *J* = 9.0 Hz), 6.52 (d, 2H, *J* = 9.0 Hz), 4.07 (s, 2H), 3.81 (s, 3H), 3.73 (s, 3H), 2.36 (s, 3H). Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.28; H, 6.79; N, 4.49. Found: C, 73.53; H, 6.62; N, 4.69.

3c: Oil (lit.[7]); IR (KBr)/cm^{-1}: 3387, 1696, 1621; ^{1}H NMR (500 MHz, $CDCl_3$) 7.79 (s, 1H), 7.39 (d, 2H, *J* = 8.5 Hz), 7.35 (d, 2H, *J* = 8.5 Hz), 6.75 (d, 2H, *J* = 9.0 Hz), 6.51 (d, 2H, *J* = 9.0 Hz), 4.02 (s, 2H), 3.82 (s, 3H), 3.73 (s, 3H).

3d: Oil (lit.[21]); IR (KBr)/cm^{-1}: 3393, 1709, 1631; ^{1}H NMR (500 MHz, $CDCl_3$) 7.80 (s, 1H), 7.37 (d, 2H, *J* = 8.5 Hz), 7.33 (d, 2H, *J* = 8.5 Hz), 7.16–7.13 (m, 3H), 6.53 (d, 2H, *J* = 8.5 Hz), 4.06 (s, 2H), 3.81 (s, 3H).

3e: Oil; IR (KBr)/cm^{-1}: 3396, 1713, 1637; ^{1}H NMR (500 MHz, $CDCl_3$) 8.22 (d, 2H, *J* = 9.0 Hz), 7.87 (s, 1H), 7.59 (d, 2H, *J* = 9.0 Hz), 7.15–7.13 (m, 3H), 6.52 (d, 2H, *J* = 7.5 Hz), 4.07 (s, 2H), 3.85 (s, 3H). Anal. Calcd for $C_{17}H_{16}N_2O_4$: C, 65.37; H, 5.16; N, 8.96. Found: C, 65.19; H, 5.25; N, 8.79.

3f: Oil; IR (KBr)/cm^{-1}: 3409, 1709, 1630; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.90 (s, 1H), 7.45–7.36 (m, 5H), 6.80–6.68 (m, 3H), 6.45 (d, 2H, *J* = 7.5 Hz), 4.12 (s, 2H), 3.81 (s, 3H), 3.82 (s, 3H). Anal. Calcd for $C_{18}H_{19}NO_3$: C, 72.70; H, 6.44; N, 4.71. Found: C, 72.91; H, 6.21; N, 4.86.

3g: Oil; IR (KBr)/cm^{-1}: 3407, 1714, 1628; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.80 (s, 1H), 7.28 (d, 2H, *J* = 8.5 Hz), 7.09 (d, 2H, *J* = 8.5 Hz), 6.80–6.59 (m, 3H), 6.40 (d, 1H, *J* = 7.5 Hz), 4.04 (s, 2H), 3.73 (s, 3H), 3.72 (s, 3H), 2.27 (s, 3H). Anal. Calcd for $C_{19}H_{21}NO_3$: C, 73.28; H, 6.79; N, 4.49. Found: C, 73.50; H, 6.86; N, 4.33.

3h: Oil; IR (KBr)/cm^{-1}: 3379, 1717, 1634; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.84 (s, 1H), 7.40 (d, 2H, *J* = 8.5 Hz), 7.34 (d, 2H, *J* = 8.5 Hz), 6.83–6.70 (m, 3H), 6.46 (d, 2H, *J* = 6.5 Hz), 4.08 (s, 2H), 3.85 (s, 3H), 3.83 (s, 3H). Anal. Calcd for $C_{18}H_{18}ClNO_3$: C, 65.15; H, 5.46; N, 4.22. Found: C, 65.30; H, 5.19; N, 4.39.

3i: Oil; IR (KBr)/cm^{-1}: 3391, 1709, 1617; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.80 (s, 1H), 7.38 (d, 2H, *J* = 8.5 Hz), 7.33 (d, 2H, *J* = 8.5 Hz), 6.96 (d, 2H, *J* = 8.5 Hz), 6.46 (d, 2H, *J* = 8.5 Hz), 4.04 (s, 2H), 3.81(s, 3H), 2.23 (s, 3H). Anal. Calcd for $C_{18}H_{18}ClNO_2$: C, 68.46; H, 5.74; N, 4.43. Found: C, 68.71; H, 5.54; N, 4.26.

3j: Oil; IR (KBr)/cm^{-1}: 3390, 1709, 1634; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.24 (d, 2H, *J* = 8.5 Hz), 7.88 (s, 1H), 7.57 (d, 2H, *J* = 8.5 Hz), 7.25 (d, 2H, *J* = 8.5 Hz), 6.36 (d, 2H, *J* = 8.5 Hz), 4.05 (s, 2H), 3.86 (s, 3H). Anal. Calcd for $C_{17}H_{15}ClN_2O_4$: C, 58.88; H, 4.35; N, 8.07. Found: C, 58.63; H, 4.10; N, 8.27.

3k: Oil; IR (KBr)/cm^{-1}: 3386, 1699, 1629; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.88 (s, 1H), 7.43 (d, 2H, *J* = 9.0 Hz), 7.11 (d, 2H, *J* = 9.0 Hz), 6.91 (d, 2H, *J* = 9.0 Hz), 6.45 (d, 2H, *J* = 9.0 Hz), 4.09 (s, 2H), 3.81 (s, 3H), 3.82 (s, 3H). Anal. Calcd for $C_{18}H_{18}ClNO_3$: C, 65.15; H, 5.46; N, 4.22. Found: C, 65.28; H, 5.31; N, 4.59.

3l: Oil; IR (KBr)/cm^{-1}: 3381, 1705, 1599; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 8.24 (d, 2H, *J* = 8.5 Hz), 7.88 (s, 1H), 7.57 (d, 2H, *J* = 8.5 Hz), 7.07 (d, 2H, *J* = 9.0 Hz), 6.41 (d, 2H, *J* = 9.0 Hz), 4.05 (s, 2H), 3.86 (s, 3H). Anal. Calcd for $C_{17}H_{15}BrN_2O_4$: C, 52.19; H, 3.86; N, 7.16. Found: C, 52.39; H, 3.65; N, 7.32.

3m: Oil (lit.[20]); IR (KBr)/cm^{-1}: 3333, 1707, 1628; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.81 (s, 1H), 7.43–7.25 (m, 10H), 3.82 (s, 3H), 3.81(s, 2H), 3.59 (s, 2H), 2.04 (brs, 1H).

3n: Oil (lit.[20]); IR (KBr)/cm^{-1}: 3332, 1703, 1629; ^{1}H NMR(500 MHz, $CDCl_3$) δ (ppm): 7.83 (s, 1H), 7.39–7.36 (m, 7H), 7.17 (d, 2H, *J* = 8.0 Hz), 3.86(s, 2H), 3.85(s, 3H), 3.65(s, 2H), 2.40 (s, 3H), 2.12 (brs, 1H).

3o: Oil (lit.[7]); IR (KBr)/cm^{-1}: 3330, 1709, 1630; ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.72 (s, 1H), 7.37–7.19 (m, 9H), 3.81 (s, 3H), 3.80 (s, 2H),3.53 (s, 2H), 2.06 (brs, 1H).

3p: Oil; IR (KBr)/cm^{-1}: 3324, 1703, 1631; ^{1}H NMR (500 MHz, $CDCl_3$) (ppm): 7.76 (s, 1H), 7.39–7.19 (m, 10H), 3.82 (s, 3H), 3.77 (q, 1H, J = 6.5 Hz), 3.47 (d, 1H, J = 12 Hz), 3.41 (d, 1H, J = 12 Hz), 2.01 (brs, 1H), 1.38 (d, 3H, J = 7.0 Hz). Anal. Calcd for $C_{19}H_{21}NO_2$: C, 77.25; H, 7.16; N, 4.74. Found: C, 77.15; H, 7.33; N, 4.69.

3q: Oil (lit.[26]); IR (KBr)/cm^{-1}: 3424, 1719, 1633; ^{1}H NMR (500 MHz, $CDCl_3$) 7.79 (s, 1H), 7.58 (d, 2H, J = 7.0 Hz), 7.39–7.34 (m, 3H), 3.82 (s, 3H), 3.53 (s, 2H), 1.65 (brs, 1H), 1.16 (s, 9H).

3r: Oil (lit.[20]); IR (KBr)/cm^{-1}: 3320, 1707, 1631; ^{1}H NMR (500 MHz, $CDCl_3$) (ppm): 7.82 (s, 1H), 7.51 (d, 2H, J = 6.5 Hz), 7.41–7.21 (m, 3H), 3.83 (s, 3H), 3.62 (s, 2H), 2.46 (m, 1H), 1.82 (brs, 1H), 1.73–1.20 (m, 10H).

3s: Oil (lit.[45]); IR (KBr)/cm^{-1}: 3324, 1708, 1632; ^{1}H NMR(500 MHz, $CDCl_3$) (ppm): 7.80 (s, 1H), 7.51 (d, 2H, J = 7.5 Hz), 7.41–7.37 (m, 3H), 3.82 (s, 3H), 3.59 (s, 2H), 2.83 (m, 1H), 1.91 (brs, 1H), 1.05 (d, 6H, J = 6.5 Hz).

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