Imine Acylation via Benzotriazole Derivatives: The Preparation of Enaminones

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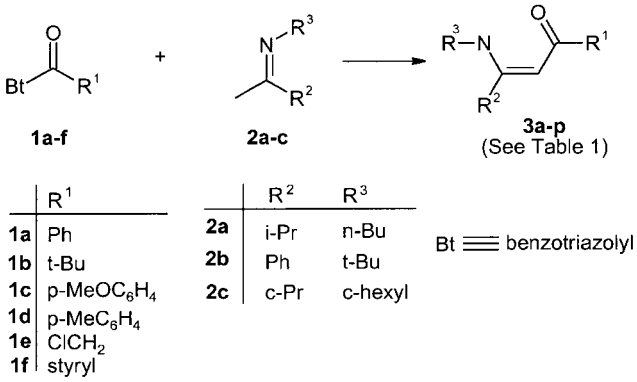
Abstract: Metalated ketimines **2a−c** are converted into enaminones **3a−p** in good to excellent yields by acylbenzotriazoles **1a−f**.

Key words: imine, acylation, lithiation, benzotriazole, enaminone

Azolides in general and acylbenzotriazoles in particular are powerful acylating reagents.[1] 1-(Trifluoroacetyl)benzotriazole is a convenient and effective trifluoroacetylating reagent for amines and alcohols.[2] *N*-Acetylbenzotriazole is superior in many ways to other reagents for the acetylation of proteins.[3] Stable *N*-formylbenzotriazole is convenient for *N*- and *O*- formylation of amines and alcohols, respectively.[4] 1,1′-(1,2-Dioxoethane-1,2-diyl)bis-1*H*-benzotriazole provides asymmetrical oxamides.[5] A 1-benzotriazolylcarbonyl group offers convenient protection of a primary amine in the synthesis of terminal peptides.[6] Oligomeric *N*-acylbenzotriazoles are used in medicinal chemistry as matrices for binding pharmacologically active substrates with hydroxyl or amino groups.[7] Poly(1-acroylbenzotriazole) with amines, alcohols, phenols or hydrazines give the corresponding polyesters and polyamides.[8a-c] Asymmetrical ketones are formed from acylbenzotriazoles with alkylaluminum chlorides[9] or organozinc agents.[10]

Enaminones are important synthetic intermediates, particularly in heterocyclic chemistry.[11] Heterocycles prepared from enaminones include carbazolequinone alkaloids,[12] tricyclic benzo[*a*]quinolizines,[13] pyrroles,[14] benzodiazepines,[15] pyrimidines,[16a,b] pyrazoles,[17a,b] isoxazoles[18] and quinolines.[19] The preparation of enaminones is well documented.[11,20,21] A common method for the synthesis of enaminones not involving C−C bond formation is to react ammonia or a primary or secondary amine with a 1,3-diketone.[11] Alternatively imine anion can be acylated at the β-carbon by esters to give enaminones:[21] in the published procedure,[21] 1.5 moles of imine was reacted with one mole of ester to give enaminones (over 11 examples) in 70% yield based on esters, or 47% yield based on imine (no imine is reported to be recovered). We now report that the analogous acylation by *N*-acylbenzotriazoles produces enaminones from imines in significantly higher conversions.

The *N*-acylbenzotriazoles **1a−f** were prepared from the corresponding acyl chloride and benzotriazole following literature procedures in 80−95% yields.[22a,b] The acylation reactions were accomplished by treatment of the ketimines **2a−c**[23] (1.0 equivalent) with LDA (2.0 equivalents) in THF at 0 °C, followed by the addition, at −78 °C, of a THF solution of a *N*-acylbenzotriazole **1a−f** (1.0 equivalent). The solution was allowed to warm up to room temperature overnight. After aqueous workup, enaminones **3a−p** were isolated as the only products in good to excellent yields (Scheme, Table).



Scheme

The structures of compounds **3a−p** were supported by NMR spectroscopy, elemental analysis and/or high-resolution mass spectrometry. For example, for compound **3a**, characteristic signals in the ^{1}H NMR spectrum were observed at δ 1.25 (s, 9H), 5.61 (s, 1H), 7.34−7.53 (m, 8H), 7.80−7.98 (m, 2H) and 11.85 (br s, 1H) which were assigned to the *t*-Bu protons, the vinylic proton, the protons from the two phenyl groups and NH group, respectively. The carbonyl group appears at δ 187.5 (CO) in the ^{13}C NMR spectrum. These signals along with the downfield shift of the NH group suggest six-membered intramolecular hydrogen bonding −N−H---O = C−, indicating that the compounds **3a−p** exist in the stable *Z* configuration enamino tautomeric form **3β** (Figure). A characteristic feature of this procedure is the total C-C versus C-N regioselectivity: the enaminones **3a−p** were the sole products isolated; no compounds corresponding to the *N*-acylation have been detected in the GC/MS spectra, the single peak had the corresponding mass.

In conclusion, enaminones **3a−p** are prepared via acylation of metalated ketimines **2a−c** using acylbenzotriazoles **1a−f** as acylating agents. With the exception of enaminones **3l−n** for which the chloromethyl group at-

Table Preparation of Enaminones **3a–p**

Compd.	SM **1**	Imine **2**	R^1	R^2	R^3	Yield[a] (%)
3a	**a**	**b**	Ph	Ph	*t*-Bu	85
3b	**a**	**a**	Ph	*i*-Pr	*n*-Bu	65
3c	**a**	**c**	Ph	*c*-Pr	*c*-hexyl	90
3d	**b**	**a**	*t*-Bu	*i*-Pr	*n*-Bu	73
3e	**b**	**b**	*t*-Bu	Ph	*t*-Bu	89
3f	**b**	**c**	*t*-Bu	*c*-Pr	*c*-hexyl	90
3g	**c**	**a**	*p*-$MeOC_6H_4$	*i*-Pr	*n*-Bu	70
3h	**c**	**b**	*p*-$MeOC_6H_4$	Ph	*t*-Bu	71
3i	**c**	**c**	*p*-$MeOC_6H_4$	*c*-Pr	*c*-hexyl	85
3j	**d**	**a**	*p*-MeC_6H_4	*i*-Pr	*n*-Bu	78
3k	**d**	**b**	*p*-MeC_6H_4	Ph	*t*-Bu	66
3l	**e**	**a**	$ClCH_2$	*i*-Pr	*n*-Bu	35
3m	**e**	**b**	$ClCH_2$	Ph	*t*-Bu	33
3n	**e**	**c**	$ClCH_2$	*c*-Pr	*c*-hexyl	29
3o	**f**	**a**	styryl	*i*-Pr	*n*-Bu	61
3p	**f**	**c**	styryl	*c*-Pr	*c*-hexyl	75

[a] Isolated yields.

tached to carbonyl causes some side reactions, the yields are good to excellent (average 77% for thirteen examples).

3α **3**β

Figure

Mps were determined on a hot stage apparatus and are uncorrected. ^1H (300 MHz) and ^{13}C (75 MHz) NMR spectra were recorded in $CDCl_3$ with TMS or $CDCl_3$ as internal reference. Elemental analyses were performed on a Carlo Erba-1106 instrument. High resolution mass spectra were measured on a Kratos/AE1-MS 30 mass spectrometer. THF and Et_2O were distilled from sodium/benzophenone under N_2 immediately prior to use. All reactions with air-sensitive compounds were carried out under an Ar atm.

Enaminones 3a–p; General Procedure

To a stirred solution of LDA (5 mmol) in THF (10 mL) at −10 °C was added dropwise the solution of ketimine **2a–c** (2.5 mmol) in THF (15 mL), and the resulting solution was stirred at 0 °C for 30 min. After cooling to −78 °C, a solution of benzotriazole derivative **1a–f** (2.5 mmol) in THF (10 mL) was added dropwise. The reaction was allowed to warm up to r.t. while stirring overnight, quenched by the addition of sat. NH_4Cl, and extracted with EtOAc. The organic extracts were combined, washed with sat. Na_2CO_3 and brine, and dried (Na_2SO_4). After evaporation under vacuum, the residue was purified by flash chromatography (hexanes/EtOAc, 1:10) to afford the desired product **3a–p**.

(*Z*)-3-(*tert*-Butylamino)-1,3-diphenylprop-2-en-1-one (3a)

Yield: 85%; mp: 117–118 °C.

^1H NMR: δ = 1.25 (s, 9H), 5.61 (s, 1H), 7.34–7.52 (m, 8H), 7.80–7.98 (m, 2H), 11.85 (br s, 1H).

^{13}C NMR: δ = 31.8, 54.0, 95.1, 127.0, 127.8, 128.1, 128.2, 128.9, 130.6, 137.5, 140.3, 167.0, 187.5.

Anal. Calcd for $C_{19}H_{21}NO$ (279.39): C, 81.68; H, 7.58; N, 5.01. Found: C, 81.76; H, 7.70; N, 5.34.

(*Z*)-3-(Butylamino)-4-methyl-1-phenylpent-2-en-1-one (3b)

Yield: 65%; oil.

^1H NMR: δ = 0.99 (t, *J* = 7.2 Hz, 3H), 1.24 (d, *J* = 6.7 Hz, 6H), 1.44–1.60 (m, 2H), 1.60–1.78 (m, 2H), 2.78–2.89 (m, 1H), 3.34–3.41 (m, 2H), 5.73 (s, 1H), 7.35–7.42 (m, 3H), 7.84–7.89 (m, 2H), 11.74 (br s, 1H).

^{13}C NMR: δ = 13.8, 20.1, 21.3, 28.8, 32.3, 42.1, 86.6, 126.8, 128.1, 130.2, 140.8, 174.5, 188.0.

HRMS (CI): *m/z* calcd for $C_{16}H_{24}NO$ (M+1): 246.1858. Found: 246.1856.

(*Z*)-3-Cyclohexylamino-3-cyclopropyl-1-phenylprop-2-en-1-one (3c)

Yield: 90%; mp: 78–80 °C.

^1H NMR: δ = 0.90–0.99 (m, 2H), 0.99–1.05 (m, 2H), 1.20–1.60 (m, 5H), 1.60–1.80 (m, 2H), 1.80–1.90 (m, 2H), 1.90–2.10 (m, 2H), 3.75–3.90 (m, 1H), 5.35 (s, 1H), 7.30–7.50 (m, 3H), 7.70–7.90 (m, 2H), 11.81 (br s, 1H).

^{13}C NMR: δ = 7.9, 11.9, 24.6, 25.4, 33.9, 51.5, 85.4, 126.7, 128.0, 130.1, 140.9, 168.5, 187.4.

Anal. Calcd for $C_{18}H_{23}NO$ (269.39): C, 80.25; H, 8.61; N, 5.20. Found: C, 79.89; H, 8.93; N, 5.21.

(*Z*)-5-(Butylamino)-2,2,6-trimethylhept-4-en-3-one (3d)

Yield: 73%; oil.

^1H NMR: δ = 0.94 (t, *J* = 7.2 Hz, 3H), 1.00–1.30 (m, 15H), 1.30–1.50 (m, 2H), 1.52–1.70 (m, 2H), 2.62–2.80 (m, 1H), 3.20–3.30 (m, 2H), 5.19 (s, 1H), 11.24 (br s, 1H).

^{13}C NMR: δ = 13.8, 20.2, 21.3, 28.1, 28.7, 32.3, 41.5, 41.9, 84.6, 173.4, 203.9.

Anal. Calcd for $C_{14}H_{27}NO$ (225.38): N, 6.21. Found: N, 6.16.

(*Z*)-1-(*tert*-Butylamino)-4,4-dimethyl-1-phenylpent-1-en-3-one (3e)

Yield: 89%; mp: 123–124 °C.

^1H NMR: δ = 1.15 (s, 9H), 1.16 (s, 9H), 5.02 (s, 1H), 7.26–7.39 (m, 5H), 11.23 (br s, 1H).

^{13}C NMR: δ = 27.9, 31.7, 41.6, 53.5, 93.8, 127.6, 128.3, 128.6, 137.8, 166.0, 203.9.

Anal. Calcd for $C_{17}H_{25}NO$ (259.39): N, 5.40. Found: N, 5.20.

(*Z*)-1-Cyclohexylamino-1-cyclopropyl-4,4-dimethylpent-1-en-3-one (3f)

Yield: 90%; oil.

^{1}H NMR: δ = 0.70−0.80 (m, 2H), 0.90−1.00 (m, 2H), 1.12 (s, 9H), 1.20−1.50 (m, 5H), 1.50−1.70 (m, 2H), 1.70−1.90 (m, 2H), 1.90−2.10 (m, 2H), 3.60−3.80 (m, 1H), 4.84 (s, 1H), 11.28 (br s, 1H).

^{13}C NMR: δ = 7.5, 11.8, 24.8, 25.4, 28.1, 34.0, 41.4, 51.3, 83.7, 167.2, 203.4.

Anal. Calcd for $C_{16}H_{27}NO$ (249.40): N, 5.62. Found: N, 5.89.

(*Z*)-3-(Butylamino)-1-(4-methoxyphenyl)-4-methylpent-2-en-1-one (3g)
Yield: 70%; mp: 68−70 °C.

^{1}H NMR: δ = 0.96 (t, *J* = 5.0 Hz, 3H), 1.21 (d, *J* = 2.2 Hz, 6H), 1.40−1.60 (m, 2H), 1.60−1.78 (m, 2H), 2.75−2.90 (m, 1H), 3.30−3.40 (m, 2H), 3.83 (s, 3H), 5.67 (s, 1H), 6.88 (d, *J* = 8.8 Hz, 2H), 7.83 (d, *J* = 8.8 Hz, 2H), 11.68 (br s, 1H).

^{13}C NMR: δ = 13.7, 20.0, 21.2, 28.7, 32.2, 41.9, 55.2, 85.9, 113.2, 128.4, 133.5, 161.2, 173.9, 187.1.

Anal. Calcd for $C_{17}H_{25}NO_2$ (275.39): N, 5.09. Found: N, 5.41.

(*Z*)-3-(*tert*-Butylamino)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (3h)
Yield: 71%; mp: 98−100 °C.

^{1}H NMR: δ = 1.21 (s, 9H), 3.82 (s, 3H), 5.56 (s, 1H), 6.85 (d, *J* = 8.8 Hz, 2H), 7.38−7.50 (m, 5H), 7.80 (d, *J* = 8.8 Hz, 2H), 11.72 (br s, 1H).

^{13}C NMR: δ = 21.3, 31.8, 55.2, 94.6, 113.2, 127.7, 128.2, 128.5, 128.7, 132.9, 137.8, 161.6, 166.4, 186.7.

Anal. Calcd for $C_{20}H_{23}NO_2$ (309.41): N, 4.53. Found: N, 4.66.

(*Z*)-3-(Cyclohexylamino)-3-cyclopropyl-1-(4-methoxyphenyl)-prop-2-en-1-one (3i)
Yield: 85%; oil.

^{1}H NMR: δ = 0.80−0.95 (m, 2H), 0.95−1.00 (m, 2H), 1.20−1.56 (m, 6H), 1.58−1.76 (m, 2H), 1.78−1.88 (m, 2H), 1.92−2.05 (m, 2H), 3.81 (s, 3H), 5.33 (s, 1H), 6.87 (d, *J* = 9.0 Hz, 2H), 7.81 (d, *J* = 9.0 Hz, 2H), 11.65 (br s, 1H).

^{13}C NMR: δ = 7.6, 11.8, 20.6, 24.4, 25.3, 29.6, 33.8, 51.2, 55.1, 84.8, 113.1, 128.3, 133.4, 161.2, 167.9, 186.6.

Anal. Calcd for $C_{19}H_{25}NO_2$ (299.42): N, 4.68. Found: N, 4.97.

(*Z*)-3-(Butylamino)-4-methyl-1-(4-methylphenyl)pent-2-en-1-one (3j)
Yield: 78%; oil.

^{1}H NMR: δ = 0.96 (t, *J* = 7.5 Hz, 3H), 1.22 (d, *J* = 6.8 Hz, 6H), 1.40−1.60 (m, 2H), 1.60−1.76 (m, 2H), 2.37 (s, 3H), 2.72−2.90 (m, 1H), 3.30−3.40 (m, 2H), 5.70 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 11.70 (br s, 1H).

^{13}C NMR: δ = 13.7, 20.0, 21.2, 21.3, 28.7, 32.2, 42.0, 86.3, 126.8, 128.7, 138.3, 140.3, 174.2, 187.9.

Anal. Calcd for $C_{17}H_{25}NO$ (259.39): N, 5.40. Found: N, 5.28.

(*Z*)-3-(*tert*-Butylamino)-1-(4-methylphenyl)-3-phenylprop-2-en-1-one (3k)
Yield: 66%; mp: 100−102 °C.

^{1}H NMR: δ = 1.22 (s, 9H), 2.36 (s, 3H), 5.57 (s, 1H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.30−7.55 (m, 5H), 7.77 (d, *J* = 8.2 Hz, 2H), 11.90 (br s, 1H).

^{13} C NMR: δ = 21.4, 31.8, 53.9, 94.9, 127.0, 127.8, 128.2, 128.8, 137.5, 140.9, 166.7, 187.4.

Anal. Calcd for $C_{20}H_{23}NO$ (293.41): N, 4.77. Found: N, 4.57.

(*Z*)-4-(Butylamino)-1-chloro-5-methylhex-3-en-2-one (3l)
Yield: 35%; oil.

^{1}H NMR: δ = 0.95 (t, *J* = 7.2 Hz, 3H), 1.17 (d, *J* = 6.8 Hz, 6H), 1.36−1.56 (m, 2H), 1.56−1.70 (m, 2H), 2.70−2.84 (m, 1H), 3.20−3.40 (m, 2H), 3.98 (s, 2H), 5.50 (s, 1H), 11.30 (br s, 1H).

^{13}C NMR: δ = 13.6, 19.9, 20.9, 28.6, 31.9, 47.2, 86.3, 175.4, 187.8.

HRMS (CI): *m/z* calcd for $C_{11}H_{21}ClNO$ (M+1): 218.1312. Found: 218.1319.

(*Z*)-4-(*tert*-Butylamino)-1-chloro-4-phenylbut-3-en-2-one (3m)
Yield: 33%; mp: 130−131 °C.

^{1}H NMR: δ = 1.18 (s, 9H), 4.00 (s, 2H), 5.14 (s, 1H), 7.22−7.43 (m, 5H), 11.40 (br s, 1H).

^{13}C NMR: δ = 31.6, 47.2, 54.4, 94.7, 127.8, 129.1, 136.5, 167.8, 187.6.

HRMS (CI): *m/z* calcd for $C_{14}H_{19}ClNO$ (M+1): 252.1155. Found: 252.1154.

(*Z*)-1-Chloro-4-(cyclohexylamino)-4-cyclopropylbut-3-en-2-one (3n)
Yield: 29%; mp: 61−63 °C.

^{1}H NMR: δ = 0.75−0.87 (m, 8H), 0.96−1.12 (m, 3H), 1.16−1.32 (m, 1H), 1.32−1.72 (m, 1H), 1.80−2.05 (m, 3H), 3.58 (s, 2H), 5.89 (s, 1H).

^{13}C NMR: δ = 6.6, 8.3, 8.8, 11.3, 19.8, 24.3, 33.5, 40.0, 105.4, 138.1, 157.9.

HRMS (CI): *m/z* calcd for $C_{13}H_{21}ClNO$ (M+1): 242.1312. Found: 242.1341.

(1*E*,4*Z*)-5-(Butylamino)-6-methyl-1-phenylhepta-1,4-dien-3-one (3o)
Yield: 61%; oil.

^{1}H NMR: δ = 0.96 (t, *J* = 7.2 Hz, 3H), 1.18 (d, *J* = 5.6 Hz, 6H), 1.40−1.60 (m, 2H), 1.60−1.75 (m, 2H), 2.70−2.84 (m, 1H), 3.20−3.40 (m, 2H), 5.19 (s, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 7.20−7.40 (m, 3H), 7.40−7.60 (m, 3H), 11.85 (br s, 1H).

^{13}C NMR: δ = 13.7, 20.0, 21.1, 28.5, 32.1, 42.1, 91.7, 127.6, 128.6, 128.7, 129.1, 136.1, 136.5, 174.7, 185.3.

Anal. Calcd for $C_{18}H_{25}NO$ (271.41): N, 5.16. Found: N, 5.32.

(1*Z*,4*E*)-1-(Cyclohexylamino)-1-cyclopropyl-5-phenylpenta-1,4-dien-3-one (3p)
Yield: 75%; mp: 75−77 °C.

^{1}H NMR: δ = 0.80−0.90 (m, 2H), 0.90−1.00 (m, 2H), 1.20−1.60 (m, 5H), 1.60−1.76 (m, 2H), 1.76−1.90 (m, 2H), 1.90−2.04 (m, 2H), 3.72−3.90 (m, 1H), 4.80 (s, 1H), 6.64 (d, *J* = 15.7 Hz, 1H), 7.24−7.40 (m, 4H), 7.40−7.60 (m, 2H), 12.00 (br s, 1H).

^{13}C NMR: δ = 8.08, 11.4, 24.4, 25.4, 33.7, 51.4, 90.5, 127.6, 128.6, 129.0, 136.1, 136.4, 169.0, 184.6.

Anal. Calcd for $C_{20}H_{25}NO$ (295.43): N, 4.74. Found: N, 4.69.

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