

One Pot Synthesis of γ -Iminolactones by Three Component Reactions Between Alkyl Isocyanides, Acetylenic Esters and Benzyl Phenyl Ketone and its α -Chloro Derivative

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The 1:1 intermediate generated by the addition of alkyl isocyanides to acetylenic esters is trapped by benzyl phenyl ketone and its derivatives to form γ -iminolactone derivatives.

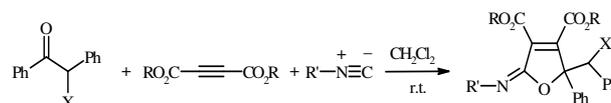
Key Words: Isocyanide, Acetylenic ester, Benzyl phenyl ketone, 2-Chloro-2-phenyl acetophenone.

INTRODUCTION

Isocyanide-based multicomponent reactions (IMCRs) now occupy a position of importance in synthetic organic chemistry, mainly in the synthesis of various heterocyclic systems like amino furans, iminopyrans and iminolactones from readily available starting materials¹⁻³. The reaction of 1:1 zwitterionic species, generated *in situ* by the addition of isocyanide to dialkyl acetylenedicarboxylates can be trapped by a variety of electrophiles such as aldehydes, 1,3-diketones, isocyanates, isothiocyanates, N-tosylimines and proton donors for synthesis of heterocyclic compounds⁴⁻⁷. Recently, the reaction between isocyanides and acetylenic esters in the presence of some α -dicarbonyl compounds which leads to γ -iminolactones has been reported⁸⁻¹¹. Hence, it was interesting to investigate the reactivity of isocyanide-acetylenic ester zwitterions towards some ketones. We now report the reaction of benzyl phenyl ketone **1** and its α -chloro derivative with dialkyl acetylene dicarboxylates **2** in the presence of alkyl isocyanides **3** which leads to γ -iminolactones (**Scheme-I**).

The structures of **4a-i** were deduced from their ¹H and ¹³C NMR and IR spectra and their mass spectrometric data. The ¹H NMR spectrum of **4a** exhibited a sharp signal for *tert*-butyl ($\delta = 1.29$ ppm), two singlets for two methoxy groups ($\delta = 3.76$ and 3.77 ppm), two doublets for diastereotopic protons of CH₂ at 3.62 and 4.03 ppm (²J_{HH} = 14 Hz) and a multiplet at 7.14-7.52 ppm for aromatic protons. The ¹³C NMR spectrum of **4a** displayed 19 sharp lines in agreement with the proposed structure.

The ¹H and ¹³C NMR spectra of **4b-i** are similar to that of **4a**, except for the signals of alkoxy groups. The ¹H NMR spectrum of **4d** exhibited a multiplet for cyclohexyl CH proton



1	2	3	4	Yield (%)
4	X	R	R'	
a	H	Me	^t Bu	60
b	H	Et	^t Bu	70
c	H	^t Bu	^t Bu	75
d	H	Me	cyclohexyl	60
e	H	Et	cyclohexyl	70
f	Cl	Me	^t Bu	85
g	Cl	Et	^t Bu	88
h	Cl	Me	cyclohexyl	75
i	Cl	Et	cyclohexyl	80

Scheme-I: Synthesis of γ -iminolactones through three component reaction

($\delta = 3.67$ ppm) and a multiplet for methylene groups (1.19-1.70 ppm), two doublets for two diastereotopic protons of CH₂ groups at about 3.59 and 3.96 (²J_{HH} = 13.8 Hz), two singlets for two methoxy groups (3.74 and 3.76 ppm) and a multiplet for aromatic protons (7.16-7.26 ppm). The ¹³C NMR spectrum of **4d** displayed 23 sharp lines in agreement with the proposed structure. This spectrum indicated six signals for cyclohexyl ($\delta = 25.2, 25.3, 26.2, 33.7, 33.8, 43.2$ and 57.1 ppm), two signals for two methoxy groups (53.1 and 53.2 ppm) and two signals for two carbonyl groups (162.2 and 162.6 ppm). The structural assignments of **4a-i** were supported by the IR spectra which have strong absorption bands at about 1750 and 1720 cm⁻¹ for carbonyl groups of esters. The nature of these compounds as 1:1:1 adducts was apparent from their mass spectra, which displayed, in each case, the molecular ion peak at appropriate m/z value.

EXPERIMENTAL

Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. NMR spectra were recorded with BRUCKER DRX-400 AVANCE spectrometer (at 400.1 MHz for ^1H and 100.6 MHz for ^{13}C) with CDCl_3 as solvent. Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a FT-IR, Bruker, VECTOR22 spectrometer. TLC was carried out on Fluka silica gel TLC-cards. All other reagents and solvents were used as received from commercial suppliers. All of the coupling constants are given in hertz.

General procedure: To a magnetically stirred solution of benzyl phenyl ketone (0.39 g, 2 mmol) and dialkyl acetylene dicarboxylate (2 mmol) in CH_2Cl_2 (10 mL), dropwise, 2 mmol of *tert*-butyl isocyanide in CH_2Cl_2 (3 mL) was added at -10°C over 10 min. The mixture was allowed to stir at room temperature for 2 days. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel, 230-400 mesh) column chromatography using hexane:ethyl acetate (70:30) as eluent. The solvent was removed under reduced pressure and the products **4a-i** were obtained.

Dimethyl-2-benzoyl-5-(*tert*-butyl imino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4a): Yellow powder, m.p. $103-105^\circ\text{C}$, yield 60%; IR (KBr, ν_{max} , cm^{-1}): 1751 and 1729 ($\text{C}=\text{O}$), 1674 ($\text{C}=\text{N}$), 1656 ($\text{C}=\text{C}$). ^1H NMR (300.1 MHz, CDCl_3): δ 1.29 (s, 9H, CMe_3), 3.62 (d, $^2J_{\text{HH}}$ 14.0 Hz, 1H, CH), 3.76 and 3.77 (2s, 6H, 2OCH_3), 4.03 (d, $^2J_{\text{HH}}$ 14.0 Hz, 1H, CH), 7.14-7.52 (m, 10H, aromatic protons); ^{13}C NMR (75.5 MHz, CDCl_3): δ 29.8 (CMe_3), 42.6 (CH_2), 52.6 (NCMe_3), 54.4 (2OCH_3), 92.8 (quaternary carbon of furan), 125.8, 127.1, 128.0, 128.8, 128.5, 130.6, 134.4, 137.8 (aromatic carbons), 139.5 ($\text{C}-\text{C}=\text{N}$), 143.9 ($\text{C}=\text{C}-\text{C}=\text{N}$), 152.4 ($\text{C}=\text{N}$), 161.7 and 162.5 ($2\text{C}=\text{O}$ ester). EI-MS, m/z (%): 421 (421 (M^+ , 2), 406 (13), 374 (10), 330 (56), 274 (100), 242 (80), 202 (23), 105 (92), 91(59), 57 (69). Anal. calcd. (%) for $\text{C}_{25}\text{H}_{27}\text{NO}_5$: C 71.24, H 6.46, N 3.32. Found (%): C 71.12, H 6.41, N 3.29.

Diethyl-2-benzoyl-5-(*tert*-butyl imino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4b): Yellow powder, m.p. $64-66^\circ\text{C}$, yield 70%. IR (KBr, ν_{max} , cm^{-1}): 1743 and 1718 ($2\text{C}=\text{O}$), 1678 ($\text{C}=\text{N}$), 1652 ($\text{C}=\text{C}$). ^1H NMR (300.1 MHz, CDCl_3): δ 1.24 and 1.26 (t, $^3J_{\text{HH}}$ 7.0 Hz, 6H, 2CH_3), 1.29 (s, 9H, CMe_3), 3.62 and 4.01 (d, $^2J_{\text{HH}}$ 13.9 Hz, 2H, 2CH), 4.16-4.25 (m, 4H, 2OCH_2), 7.14-7.53 (m, 10H, aromatic protons); ^{13}C NMR (75.5 MHz, CDCl_3): δ 13.8 and 13.9 (2CH_3), 42.7 (CH_2), 54.3 (NCMe_3), 61.6 and 61.6 (2OCH_2), 92.8 (quaternary carbon of furan), 125.9, 127.0, 127.9, 128.2, 128.4, 130.7, 134.5, 137.8 (aromatic carbons), 139.6 ($\text{C}-\text{C}=\text{N}$), 143.7 ($\text{C}=\text{C}-\text{C}=\text{N}$), 152.5 ($\text{C}=\text{N}$), 161.3 and 162.1 ($2\text{C}=\text{O}$ ester). EI-MS, m/z (%): 449 (M^+ , 1), 434 (11), 358 (43), 302 (100), 256 (94), 228 (50), 202 (18), 105 (80), 91 (53), 57 (68). Anal. calcd. (%) for $\text{C}_{27}\text{H}_{31}\text{NO}_5$: C 72.14, H 6.95, N 3.11. Found (%): C 72.03, H 6.93, N 3.05.

Di(*tert*-butyl) 2-benzoyl-5-(*tert*-butyl imino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4c): Yellow powder, m.p. $82-84^\circ\text{C}$, yield 75%. IR (KBr, ν_{max} , cm^{-1}): 1743 and 1718 ($2\text{C}=\text{O}$), 1674 ($\text{C}=\text{N}$), 1656 ($\text{C}=\text{C}$). ^1H NMR (300.1 MHz, CDCl_3): δ 1.25 (s, 9H, CMe_3), 1.45 (s, 18H, 2CMe_3), 3.58 and 3.98 (d, $^2J_{\text{HH}}$ 13.8 Hz, 2H, 2CH), 7.16-7.52 (m, 10H, aromatic protons); ^{13}C NMR (75.5 MHz, CDCl_3): δ 27.9 and 28.1

(2OCMe_3), 29.9 (NCMe_3), 42.9 (CH_2), 53.9 (NCMe_3), 92.5 (quaternary carbon of furan), 126.0, 126.8, 127.8, 128.1, 128.2, 130.7, 134.8, 137.9 (aromatic carbons), 140.1 ($\text{C}-\text{C}=\text{N}$), 143.1 ($\text{C}=\text{C}-\text{C}=\text{N}$), 152.6 ($\text{C}=\text{N}$), 160.8 and 161.3 ($2\text{C}=\text{O}$ ester). EI-MS, m/z (%): 506 (M^+ + 1, 59), 490 (2), 450 (3), 394 (14), 302 (23), 246 (50), 105 (14), 91 (80), 57 (100). Anal. calcd. (%) for $\text{C}_{31}\text{H}_{39}\text{NO}_5$: C 73.6, H 7.77, N 2.77. Found (%): C 73.52, H 7.72, N 2.73.

Dimethyl-2-benzoyl-5-(cyclohexyl imino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4d): Yellow powder, m.p. $123-125^\circ\text{C}$, yield 60%. IR (KBr, ν_{max} , cm^{-1}): 1739 and 1719 ($2\text{C}=\text{O}$), 1675 ($\text{C}=\text{N}$), 1656 ($\text{C}=\text{C}$). ^1H NMR (500.1 MHz, CDCl_3): δ 1.19-1.70 (m, 10H, 5CH_2), 3.59 and 3.96 (d, $^2J_{\text{HH}}$ 13.8 Hz, 2H, 2CH), 3.65-3.69 (m, 1H, NCH), 3.76 (s, 6H, 2OCH_3), 7.16-7.26 (m, 1H, aromatic proton), 7.34 (t, $^3J_{\text{HH}}$ 7.1 Hz, 1H, aromatic proton), 7.39 (t, $^3J_{\text{HH}}$ 7.1 Hz, 2H, aromatic protons), 7.49 (d, 2H, $^3J_{\text{HH}}$ 7.1 Hz, aromatic protons). ^{13}C NMR (125.8 MHz, CDCl_3): δ 25.2, 25.3, 26.2, 33.7, 33.8 (5CH_2 , cyclohexyl), 43.2 (CH_2), 53.1 and 53.2 (2OCH_3), 57.1 (NCH), 92.1 (quaternary carbon of furan), 126.2, 127.5, 128.4, 128.9, 129.0, 131.0, 134.7, 136.4 (aromatic carbons), 139.5 ($\text{C}-\text{C}=\text{N}$), 145.8 ($\text{C}=\text{C}-\text{C}=\text{N}$), 155.0 ($\text{C}=\text{N}$), 162.2 and 162.6 ($2\text{C}=\text{O}$ ester). EI-MS, m/z (%): 447 (M^+ , 14), 356 (53), 274 (89), 242 (53), 203 (20), 105 (100), 83 (39). Anal. calcd. (%) for $\text{C}_{27}\text{H}_{29}\text{NO}_5$: C 72.46, H 6.53, N 3.13. Found (%): C 72.38, H 6.51, N 3.10.

Diethyl-2-benzoyl-5-(cyclohexyl imino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4e): Yellow powder, m.p. $115-117^\circ\text{C}$, yield 70%. IR (KBr, ν_{max} , cm^{-1}): 1750 and 1719 ($2\text{C}=\text{O}$), 1679 ($\text{C}=\text{N}$), 1658 ($\text{C}=\text{C}$). ^1H NMR (500.1 MHz, CDCl_3): δ 1.23 and 1.24 (t, $^3J_{\text{HH}}$ 7.1 Hz, 6H, 2CH_3), 1.28-1.76 (m, 10H, 5CH_2), 3.60 and 4.00 (d, $^2J_{\text{HH}}$ 13.8 Hz, 2H, 2CH), 3.64-3.68 (m, 1H, NCH), 4.19-4.24 (m, 4H, 2OCH_2), 7.16-7.23 (m, 1H, -Ph), 7.34 (t, $^3J_{\text{HH}}$ 7.0 Hz, 1H, -Ph), 7.39 (t, $^3J_{\text{HH}}$ 7.0 Hz, 2H, aromatic protons), 7.51 (d, $^3J_{\text{HH}}$ 7.0 Hz, 2H, aromatic protons). ^{13}C NMR (125.8 MHz, CDCl_3): δ 14.2 and 14.3 (2CH_3), 25.1, 25.2, 26.2, 33.7, 33.8 (5CH_2 , cyclohexyl), 43.2 (CH_2), 56.9 (NCH), 62.1 and 62.2 (2OCH_2), 92.0 (quaternary carbon of furan), 126.3, 127.5, 128.4, 128.8, 128.9, 131.0, 134.8, 136.4 (aromatic carbons), 139.6 ($\text{C}-\text{C}=\text{N}$), 145.5 ($\text{C}=\text{C}-\text{C}=\text{N}$), 155.1 ($\text{C}=\text{N}$), 161.8 and 162.1 ($2\text{C}=\text{O}$ ester). EI-MS, m/z (%): 475 (M^+ , 15), 430 (8), 384 (65), 302 (100), 256 (70), 228 (44), 105 (97), 91 (44). Anal. calcd. (%) for $\text{C}_{29}\text{H}_{33}\text{NO}_5$: C 73.24, H 6.99, N 2.95. Found (%): C 73.19, H 6.95, N 2.92.

Dimethyl-5-(*tert*-butylimino)-2-[chloro(phenyl)methyl]-2-phenyl-2,5-dihydro-3,4-furandicarboxylate (4f): White powder, m.p. $146-148^\circ\text{C}$, yield 85%. IR (KBr, ν_{max} , cm^{-1}): 1761 and 1731 ($2\text{C}=\text{O}$), 1690 ($\text{C}=\text{N}$), 1645 ($\text{C}=\text{C}$). ^1H NMR (400.1 MHz, CDCl_3): δ 1.29 (9H, s, CMe_3), 3.89 and 3.92 (2s, 6H, 2OCH_3), 6.26 (s, 1H, CHCl), 7.18-7.66 (m, 10H, aromatic protons). ^{13}C NMR (100.6 MHz, CDCl_3): δ 30.1 (CMe_3), 52.9 and 53.0 (2OCH_3), 54.8 (NCMe_3), 65.1 (CHCl), 94.7 (quaternary carbon of furan), 125.8, 127.8, 128.4, 128.4, 128.5, 129.4, 136.3, 136.9 (aromatic carbons), 138.2 ($\text{C}-\text{C}=\text{N}$), 142.7 ($\text{C}=\text{C}-\text{C}=\text{N}$), 151.7 ($\text{C}=\text{N}$), 161.9 and 162.5 ($2\text{C}=\text{O}$). EI-MS, m/z (%): 455 (M^+ , 1), 330 (41), 315 (17), 300 (4), 274 (100), 242 (41), 125 (30), 105 (30), 77(9), 57 (23). Anal. calcd. (%) for $\text{C}_{25}\text{H}_{26}\text{NO}_5\text{Cl}$ (455.94): C 65.86, H 5.75, N 3.07. Found (%): C 65.79, H 5.71, N 3.11.

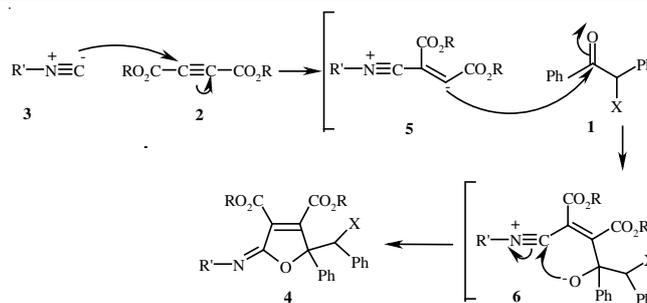
Diethyl-5-(*tert*-butylimino)-2-[chloro(phenyl)methyl]-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4g): White powder, m.p. 150-152 °C, yield 88 %. IR (KBr, ν_{\max} , cm^{-1}): 1747 and 1719 (2C=O), 1679 (C=N), 1651 (C=C). ^1H NMR (400.1 MHz, CDCl_3): δ 1.20 and 1.21 (t, 6H, $^3J_{\text{HH}}$ 7.2 Hz, 2CH₃), 1.51 (s, 9H, CMe₃), 4.08-4.27 (m, 4H, 2OCH₂), 6.26 (s, 1H, CHCl), 7.30-7.67 (m, 10H, aromatic protons). ^{13}C NMR (100.6 MHz, CDCl_3): δ 13.7 and 13.8 (2CH₃), 30.1 (CMe₃), 54.9 (NCMe₃), 61.6 and 61.9 (2OCH₂), 63.5 (CHCl), 94.5 (quaternary carbon of furan), 126.3, 128.1, 128.4, 128.5, 129.1, 135.7, 137.5 (aromatic carbons), 137.6 (C-C=N), 142.6 (C=C-C=N), 151.7 (C=N), 161.3 and 160.9 (2C=O). EI-MS, m/z (%): 484 ($M^+ + 1$, 93), 392 (10), 358 (48), 302 (100), 256 (55), 228 (24), 125 (60), 105 (65), 77(12), 57 (48). Anal. calcd. (%) for C₂₇H₃₀NO₅Cl (483.99): C 67.01, H 6.25, N 2.89. Found (%): C 66.95, H 6.22, N 2.85.

Dimethyl-2-[chloro(phenyl)methyl]-5-(cyclohexylimino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4h): White powder, m.p. 165-167 °C, yield 75 %. IR (KBr, ν_{\max} , cm^{-1}): 1747 and 1719 (2C=O), 1679 (C=N), 1651 (C=C). ^1H NMR (400.1 MHz, CDCl_3): δ 1.25-1.90 (m, 10H, 5CH₂), 3.70 and 3.74 (2s, 6H, 2OCH₃), 3.98-4.03 (m, 1H, NCH), 6.26 (s, 1H, CHCl), 7.30-7.64 (m, 10H, aromatic protons). ^{13}C NMR (100.6 MHz, CDCl_3): δ 24.6, 24.7, 25.7, 33.4, 33.6 (5CH₂, cyclohexyl), 52.8 and 52.9 (2OCH₃), 56.8 (NCH), 63.5 (CHCl), 93.5 (quaternary carbon of furan), 126.0, 128.2, 128.5, 128.6, 129.2, 129.5, 135.5, 135.7 (aromatic carbons), 137.1 (C-C=N), 144.4 (C=C-C=N), 153.8 (C=N), 161.4 and 161.4 (2C=O). EI-MS, m/z (%): 483 ($M^+ + 2$, 1), 481 (M^+ , 3), 445 (4), 386 (3), 356 (60), 324 (15), 274 (100), 242 (53), 125 (27), 105 (73), 83 (6), 77 (14). Anal. calcd. (%) for C₂₇H₂₈NO₅Cl (481.97): C 67.29, H 5.86, N 2.91. Found (%): C 67.24, H 5.91, N 2.95.

Diethyl-2-[chloro(phenyl)methyl]-5-(cyclohexylimino)-2-phenyl-2,5-dihydro-3,4-furan dicarboxylate (4i): White powder, m.p. 157-159 °C, yield 80 %. IR (KBr, ν_{\max} , cm^{-1}): 1754 and 1744 (2C=O), 1680 (C=N), 1649 (C=C). ^1H NMR (400.1 MHz, CDCl_3): δ 1.18 and 1.21 (t, $^3J_{\text{HH}}$ 7.2 Hz, 6H, 2CH₃), 1.45-1.90 (m, 10H, 5CH₂), 3.98 and 4.02 (m, 1H, NCH), 4.11-4.28 (m, 4H, 2OCH₂), 6.26 (s, 1H, CHCl), 7.28-7.65 (m, 10H, aromatic protons). ^{13}C NMR (100.6 MHz, CDCl_3): δ 13.7 and 13.8 (2CH₃), 24.6, 24.7, 25.7, 33.4, 33.6 (5CH₂, cyclohexyl), 56.6 (NCH), 61.7 and 62.0 (2OCH₃), 63.6 (CHCl), 93.4 (quaternary carbon of furan), 126.1, 128.2, 128.5, 128.6, 129.1, 135.6, 135.9 (aromatic carbons), 137.1 (C-C=N), 144.0 (C=C-C=N), 153.8 (C=N), 161.0 and 161.1 (2C=O). EI-MS, m/z (%): 483 ($M^+ + 1$, 59), 474 (10), 384 (100), 338 (24), 302 (80), 256 (49), 228 (28), 125 (51), 105 (51), 83 (4), 77 (6). Anal. calcd. (%) for C₂₉H₃₂NO₅Cl (510.03): C 68.29, H 6.32, N 2.75. Found (%): C 68.32, H 6.27, N 2.79.

RESULTS AND DISCUSSION

On the basis of the well established chemistry of isocyanides¹²⁻¹⁴, it is reasonable to assume that compound **4** results from the initial addition of isocyanide to the acetylenic diester to form the zwitter ionic intermediate **5**, which is added to the carbonyl group of compound **1**. Then, nucleophilic addition to nitrilinium moiety **6** leads to the formation of iminolactones **4** (Scheme-II).



Scheme-II: Mechanism of the three component reaction

Conclusion

In conclusion, a one-pot, three-component reaction of isocyanides and dialkyl acetylene dicarboxylates with benzyl phenyl ketone and its α -chloro derivative leading to novel iminolactone compounds in moderate yields has been described. The products are highly functionalized molecules potentially amenable to further manipulation. Also, the present method carries some advantages including mild and neutral reaction conditions without any activation or modification.

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