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Reaction between alkyl isocyanides and dialkyl acetylenedicarboxylates in the presence of *N*-alkyl isatins: convenient synthesis of γ-spiro-iminolactones

Abbas Ali Esmaeili* and Masoomeh Darbanian

Department of Chemistry, University of Birjand, P.O. Box 414, Birjand, Iran

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Abstract—The highly reactive 1:1 intermediate generated in the reaction between an alkyl isocyanide and a dialkyl acetylenedicarboxylate is trapped by *N*-alkyl isatin to yield iminolactones in fairly high yields. © 2003 Elsevier Science Ltd. All rights reserved.

1. Introduction

The reactivity of nucleophilic carbenes such as isocyanides towards dimethyl acetylenedicarboxylate (DMAD) is well documented.[1,2] The reaction of isocyanides with carbon–carbon triple bonds occurs in a stepwise manner through a zwitterionic intermediate, the ultimate fate of which appears to be dictated by the nature of the original triple-bonded substrate.[3–5]

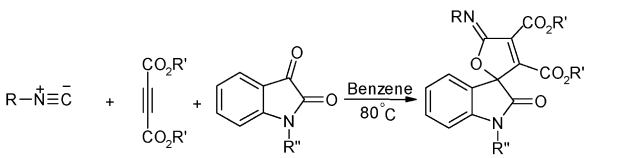
The initially formed zwitterionic intermediate has been shown to undergo further reaction with DMAD and isocyanide in different molar proportions, leading to a variety of complex heterocyclic compounds and these reactions have been the subject of detailed investigation by a number of research groups.[6–13]

In order to confirm the presence of highly reactive intermediates derived from isocyanides and acetylenic esters which are then likely to undergo addition to alkyl isatins leading to heterocycles. We initiated an investigation of the reaction of isocyanides and acetylenic esters with isatin derivatives. Previous attempts to trap such zwitterionic intermediates with olefinic dipolarophiles such as cyclohexene and dimethylfumarate had failed.[10] However, the existence of the 1:1 intermediate was indicated by the isolation of 1:1:1 heterocyclic adducts from the reaction mixture of isocyanide with DMAD in the presence of methyl isatin.

2. Results and discussion

Thus, alkyl isocyanides **1** and acetylenic esters **2** in the presence of *N*-alkyl isatin **3** undergo a cycloaddition reaction in benzene at 80°C to produce γ-spiro iminolactons **4** in good to excellent yields (Scheme 1).

On the basis of the well established chemistry of isocyanides,[3,4] it is reasonable to assume that compounds **4** result from initial addition of alkyl isocyanides to the acetylenic esters and concomitant addition to isatins leading to γ-spiro-iminolactones (Scheme 2). Structures **4** were assigned on the basis of their elemental analyses as well as, their IR, ^{1}H NMR, ^{13}C NMR and mass spectral data. The IR spectrum of **4a** showed strong absorptions at 1755, 1735 and 1720 cm^{-1} due to the ester carbonyl and amide carbonyl and at 1680 cm^{-1} due to the C=N. The ^{1}H NMR spectrum of **4a** exhibited four singlets arising from the *tert*-butyl (δ 1.34), N–CH$_3$ (δ 3.34) and methoxy groups (δ 3.65 and



1	R
a	*t*-Bu
b	cyclohexyl

2	R'
a	Me
b	Et
c	*t*-Bu
d	i-pr

3	R"
a	Me
b	Et

4	R	R'	R"	(%)Yield
a	*t*-Bu	Me	Me	95
b	cyclohexyl	Me	Me	90
c	*t*-Bu	Et	Me	85
d	cyclohexyl	Et	Me	95
e	cyclohexyl	*i*-pr	Me	85
f	cyclohexyl	*t*-Bu	Me	85
g	*t*-Bu	Me	Et	95
h	cyclohexyl	Me	Et	90
i	cyclohexyl	*i*-pr	Et	85
j	cyclohexyl	Et	Et	90

Scheme 1.

Keywords: γ-spiro-iminolactones; isocyanid; acetilenedicarboxylate; iminlactone; isatin.

* Corresponding author. Tel.: +98-561-2230516; fax: +98-561-2230515; e-mail: aa_esmaeili@yahoo.com

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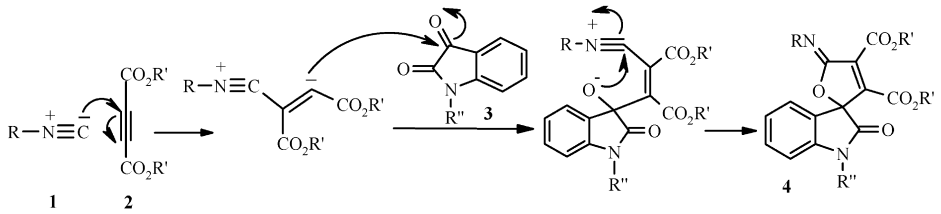

Scheme 2.

3.96). The ^{13}C NMR spectrum showed nineteen distinct resonances consistent with the γ-spiro-iminolactone structure. Partial assignments of these resonances are given in Section 3.

The characteristic signal due to the spiro carbon was described at δ 86.49. The ^{1}H and ^{13}C NMR spectra of **4b–4i** are similar to those of **4a**, except for isocyanide, esters and *N*-alkyl residues.

In conclusion, a three-component condensation reaction is required. It offers an easy and effective one-pot synthesis of iminolactones which are potentially amenable to a number of synthetic transformation.[14]

3. Experimental

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses for C, H, and N were performed using a Heraeus CHN–O-Rapid analyzer. IR spectra were measured on a Perkin–Elmer 783 Infrared spectrophotometer. ^{1}H and ^{13}C NMR spectra were measured with BRUKER DRX-500 AVANCE spectrometer at 500 and 125.77 MHz, BRUKER DRX-250 AVANCE spectrometer at 250 and 62.90 MHz, respectively. Mass spectra were recorded on a Finnigan–Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. Isocyanide and acetylenic esters **1** were obtained from Fluka (Buchs, Switzerland) and were used without further purification.

3.1. Preparation of *N*-alkyl isatin 3a and 3b

Compounds **3a** and **3b** were prepared from alkylbenzenesulfonates and potassium isatin by known methods[15] and identified as follow.[16]

3.1.1. Selected data for 3a. Purple crystals; mp 129–130°C; IR (KBr) (ν_{max}, cm^{-1}): 1730, 1750 (C=O); *m/z* (%): 161 (M^+, 20), 147 (M^++1−Me, 18); Anal. calcd for $C_9H_7NO_2$ (161.16): C, 67.07; H, 4.37; N, 8.69%. Found: C, 67.2; H, 4.5; N, 8.5%. ^{1}H NMR (90 MHz, $CDCl_3$): δ=3.23 (3H, s, N-*Me*), 6.82–7.8 (4H, m, arom.); ^{13}C NMR (22.6 MHz, $CDCl_3$): δ=26.22 (N-*Me*), 110.11 (C3a), 117.40 (C4), 123.87 (C5), 125.14 (C7), 138.57 (C6), 151.48 (C7b), 158.28 (N–C=O), 183.41(C=O of ketone).

3.1.2. Selected data for 3b. Purple crystals; mp 86–87°C; IR (KBr) (ν_{max}, cm^{-1}): 1735, 1750 (C=O); MS(CI), *m/z* (%): 175 (M^+, 87), 147 (M^++1−Et, 20), 118 (M^++1−2CO, 85); Anal. calcd for $C_{10}H_9NO_2$ (175.19): C, 68.56; H, 5.18; N, 7.99%. Found: C, 68.6; H, 5.2; N, 8.1%. ^{1}H NMR (90 MHz, $CDCl_3$): δ=1.30 (3H, t, *J*=7.2 Hz, N–CH_2*Me*), 3.80 (H, q, *J*=7.2 Hz, N–CH_2Me), 6.85–7.80 (4H, m, arom.); ^{13}C NMR (22.6 MHz, $CDCl_3$): δ=12.54 (N–CH_2*Me*), 34.98 (N–*CH*$_2$Me), 110.07 (C3a), 117.64 (C4), 123.67 (C5), 125.46 (C7), 138.37 (C6), 150.71 (C7b), 157.92 (C=O amid), 183.73 (C=O of keton).

3.2. General procedure for synthesis of dialkyl 2′-alkyl imino-2-oxo spiro[1-alkyl-indole-3, 5′-(2′,5′-dihydro furan)]3′,4′ dicarboxylate 4

A mixture of alkyl isatin (1 mmol) and dialkyl acetylendicarboxylate (1.1 mmol) in dry benzene was purged with argon at 80°C. To this mixture, isocyanide (1.1 mmol) was added via a syringe and refluxed for about 4 h. The solvent was removed under vacuum and the product **4** was crystallized out from a CH_2Cl_2–hexane mixture and washed with hexane (4×3 mL) to give white crystalline solid.

3.2.1. Selected data for 4a. (0.37 g, mp 144°C, yield 95%); IR (KBr) (ν_{max}, cm^{-1}): 1755, 1740 and 1723 (3C=O), 1680 (C=N); Anal. calcd for $C_{20}H_{22}N_2O_4$ (386.40): C, 62.17; H, 5.74; N, 7.25%. Found: C, 61.3; H, 5.7; N, 7.2%. ^{1}H NMR (250 MHz, $CDCl_3$): δ=1.34 (9H, s, C*Me*$_3$), 3.34 (3H, s, N-*Me*), 3.65 and 3.96 (6H, 2s, 2O*Me*), 6.92–7.47 (4H, m, arom.); ^{13}C NMR (62.90 MHz, $CDCl_3$): δ=27.2 (N-*Me*), 29.9 (C*Me*$_3$), 53.5 and 53.2 (2O*Me*), 55.70 (N-^{13}CMe$_3$), 86.50 (C_{spiro}), 109.3 (C), 123.0 (CH), 124.6 (C), 124.3 (C), 131.1 (CH), 138.5 (C), 139.6 (CH), 145.0 (C), 153.9 (CO amide), 160.6 and 158.6 (2CO of ester), 170.2 (C=N).

3.2.2. Selected data for 4b. (0.37 g, mp 183°C, yield 90%); IR (KBr) (ν_{max}, cm^{-1}): 1750, 1735 and 1720 (3C=O), 1678 (C=N); MS(CI), (*m/z*, %): 413 (M^++1, 35), 315 (M^+−$C_6H_{11}N$, 100), 287 (M^+−C_6H_{11}N=C=O, 35), 59 (CO_2Me, 18); Anal. calcd for $C_{22}H_{24}N_2O_6$ (412.44): C, 64.07; H, 5.87; N, 6.79%. Found: C, 63.9; H, 5.8; N, 6.8%. ^{1}H NMR (500 MHz, $CDCl_3$): δ=1.24–1.87 (10H, m, 5C*H*$_2$), 3.34 (3H, S, N-*Me*), 3.65 (1H, m, C*H*–N), 3.69 and 4.00 (6H, 2s, 2O*Me*), 6.97–7.49 (4H, m, arom.); ^{13}C NMR (125.77 MHz, $CDCl_3$): δ=26.53 (N-*Me*), 24.38, 24.44, 25.38, 32.88 and 32.89 (5*CH*$_2$), 52.56 and 52.85 (2O*Me*), 56.55 (N–*C*H), 86.61 (C_{spiro}), 108.71 (C), 123.09 (CH), 123.79 (CH), 124.09 (C), 131.33 (CH), 138.05 (C), 139.75 (CH), 144.37 (C), 153.94 (CO amid), 161.43 and 159.70 (2CO of ester), 169.88 (C=N imin).

3.2.3. Selected data for 4c. (0.35 g, mp 132°C, yield 85%); IR (KBr) (ν_{max}, cm^{-1}): 1750, 1740 and 1725 (3C=O), 1690 (C=N); MS(CI), (*m/z*, %): 415 (MH^+, 55), 399 (M^+−CH_3, 100); Anal. calcd for $C_{22}H_{26}N_2O_6$ (414.46): C, 63.76; H, 6.32; N, 6.76%. Found: C, 63.9; H, 6.2; N, 6.8%. ^{1}H NMR

(250 MHz, $CDCl_3$): δ=1.09 and 1.40 (6H, 2t, *J*=14.3, 14.3 Hz, 2OCH$_2$*Me*), 1.30 (9H, s, C*Me*$_3$), 3.29 (3H, s, N-*Me*), 4.06 (2H, m, O*CH*$_a$*H*$_b$Me), and 4.42 (2H, q, *J*=14.3 Hz, O*CH*$_2$Me), 6.90–7.46 (4H, m, arom.); ^{13}C NMR (62.90 MHz, $CDCl_3$): δ=15.91 and 16.39 (2O*CH*$_2$*Me*), 29.07 (N-*Me*), 31.87 (C*Me*$_3$), 57.49 (N–^{13}*C*Me$_3$), 64.58 and 64.10 (2O*CH*$_2$Me), 88.56 (C_{spiro}), 111.10 (C), 125.62 (CH), 126.58 (CH), 126.78 (C), 133.69 (CH), 140.25 (C), 142.23 (CH), 146.90 (C), 154.46 (CO amid), 162.90, 161.82 (2CO of ester), 171.02 (C=N iminolacton).

3.2.4. Selected data for 4d. (0.418 g, mp 159°C, yield 95%); IR(KBr) (ν_{max}, cm^{-1}): 1750, 1735 and 1730 (3C=O), 1685 (C=N); MS(CI), (*m*/*z*, %): 441 (MH$^+$, 23), 343 (M$^+$−$C_6H_{11}N$, 100), 269 (M$^+$−C_6H_{11}N–Et–OEt); Anal. calcd for $C_{24}H_{28}N_2O_6$ (440.50): C, 65.44; H, 6.41; N, 6.36%. Found: C, 65.6; H, 6.3; N, 6.4%. ^{1}H NMR (500 MHz, $CDCl_3$): δ=1.12 (3H, t, *J*=6.7 Hz, OCH$_2$*Me*), 1.43 (3H, t, *J*=6.9 Hz, OCH$_2$*Me*), 1.22–1.86 (10H, m, 5CH$_2$), 3.33(3H, s, N-*Me*), 3.65 (1H, m, N–*CH*), 4.10 (H, m, O*CH*$_2$Me), 4.47 (2H, q, *J*=6.9 Hz, O*CH*$_2$Me), 6.95–7.49 (4H, m, arom.); ^{13}C NMR (125.77 MHz, $CDCl_3$): δ=13.31 and 13.74 (2OCH$_2$*Me*), 24.34, 24.40, 25.41, 32.84 and 32.87 (5CH$_2$), 26.47 (N-*Me*), 56.40 (N–CH), 61.55 and 62.10 (2OCH$_2$), 86.57 (C_{spiro}), 108.60 (CH), 123.06 (CH), 123.99 (C), 124.14 (CH), 131.24 (CH), 138.21(C), 139.75 (C), 144.37 (C), 154.07, 159.15 and 160.99 (3C=O), 170.02 (C=N iminolacton).

3.2.5. Selected data for 4e. (0.39 g, mp 136°C, yield 85%); IR (KBr) (ν_{max}, cm^{-1}): 1755, 1740 and 1725 (3C=O), 1685 (C=N); MS(CI), (*m*/*z*, %): 470 (MH$^+$, 35), 371 (M$^+$−$C_6H_{11}N$, 47), 329 (MH$^+$−C_6H_{11}N–pr^i, 25), 287 (MH$^+$−C_6H_{11}N-2pr^i, 47), 269 (M−C_6H_{11}N–Opr^i–pr^i, 100); Anal. calcd for $C_{26}H_{32}N_2O_6$ (468.55): C, 66.65; H, 6.88; N, 5.98%. Found: C, 66.7; H, 6.8; N, 6.0%. ^{1}H NMR (500 MHz, $CDCl_3$): δ=0.97 (3H, d, *J*=6 Hz, OCHMe$_a$*Me*$_b$), 1.15 (3H, d, *J*=6 Hz, OCHMe$_a$*Me*$_b$), 1.43 (6H, d, *J*=6 Hz, OCH*Me*$_2$), 1.24–1.87 (10H, m, 5CH$_2$), 3.33 (3H, s, N-*Me*), 3.65 (1H, m, N–*CH*), 4.93 and 5.37 (2H, 2 septed, *J*=6 Hz, 2C*H*(Me)$_2$), 6.94–7.49 (4H, m, arom.); ^{13}C NMR (125.77 MHz, $CDCl_3$): δ=20.94, 21.15, 21.34 and 21.47 (2OCH*Me*$_2$), 24.19, 24.26 and 25.48 (3C*H*$_2$), 26.41 (N-*Me*), 32.75 and 32.85 (2C*H*$_2$), 56.05 (N–C*H*), 69.47 and 70.03 (2O*CH*Me$_2$), 86.49 (C_{spiro}), 108.38 (C), 123.01 (CH), 124.19 (CH), 124.28 (C), 131.10 (CH), 138.47 (C), 139.62 (CH), 144.37 (C), 153.91 (C=O amid), 158.57 and 160.61 (2C=O), 170.15 (C=N).

3.2.6. Selected data for 4f. (0.42 g, mp 185°C, yield 85%); IR (KBr) (ν_{max}, cm^{-1}): 1748, 1740 and 1723 (3C=O), 1683 (C=N); MS(CI), (*m*/*z*, %): 498 (MH$^+$, 15), 399 (M$^+$−$C_6H_{11}N$, 16), 287 (MH$^+$−C_6H_{11}N–2Bu^t, 60), 269 (M$^+$−C_6H_{11}N–Bu^t–OBu^t), 98 (M of $C_6H_{11}NH^+$, 100), 57 (M of Bu^t, 100); Anal. calcd for $C_{28}H_{36}N_2O_6$ (496.59): C, 67.72; H, 7.31; N, 5.64%. Found: C, 67.7; H, 7.2; N, 5.7%. ^{1}H NMR (500 MHz, $CDCl_3$): δ=1.25 and 1.64 (18H, 2s, 2OC*Me*$_3$), 1.24–1.84 (10H, m, 5CH$_2$), 3.31 (3H, N-*Me*), 3.64 (1H, m, N–C*H*), 6.93–7.48 (4H, m, arom.); ^{13}C NMR (125.77 MHz, $CDCl_3$): δ=24.11, 24.17, 25.55 and 26.38 (4CH$_2$ of cyclohexyl), 27.32 (N-*Me*), 27.86 and 27.86 (2OC*Me*$_3$), 32.89 (1CH$_2$ of cyclohexyl), 55.70 (N–CH), 82.86 and 83.50 (2O*C*Me$_3$), 86.26 (C_{spiro}), 108.31 (C), 122.99 (CH), 124.22 and 124.50 (2C), 130.98 (CH), 138.15 (C), 140.53 (CH), 144.36 (C), 153.83, 158.23 and 160.03 (3C=O), 170.36 (C=N).

3.2.7. Selected data for 4g. (0.38 g, mp 167°C, yield 95%); IR (KBr) (ν_{max}, cm^{-1}): 1755, 1740 and 1725 (3C=O), 1680 (C=N); Anal. calcd for $C_{21}H_{24}N_2O_6$ (400.42): C, 62.99; H, 6.04; N, 6.99%. Found: C, 62.9; H, 6.0; N, 7.0%. ^{1}H NMR (250.13 MHz, $CDCl_3$): δ=1.30 (9H, s, C*Me*$_3$), 1.34 (3H, t, *J*=7.5 Hz, N–CH$_2$*Me*), 3.63 (3H, 3s, O*Me*), 3.69 (2H, q, *J*=7.5 Hz, N–C*H*$_2$Me), 3.95(3H, s, O*Me*), 6.93–7.45 (4H, m, arom.); ^{13}C NMR (62.82 MHz, $CDCl_3$): δ=12.60 (N–CH$_2$*Me*), 29.92 (C*Me*$_3$), 35.70 (C*H*$_2$–N), 53.09 and 53.45 (2OCH$_3$), 55.65 (N–*C*Me$_3$), 86.45 (C_{spiro}), 109.45 (CH), 123.50 (CH), 123.99 (C), 124.82 (CH), 131.81 (CH), 138.4 (C), 139.58 (C), 140.07 (CH), 144.07 (C), 152.57, 160.35 and 162.43 (3C=O), 170.11 (C=N).

3.2.8. Selected data for 4h. (0.38 g, mp 152°C, yield 90%); IR (KBr) (ν_{max}, cm^{-1}): 1745, 1735 and 1715 (3C=O), 1675 (C=N); MS(CI), (*m*/*z*, %): 427 (MH$^+$, 30), 329 (M$^+$−$C_6H_{11}N$, 100); Anal. calcd for $C_{23}H_{26}N_2O_6$ (426.45): C, 67.78; H, 6.15; N, 6.57%. Found: C, 67.7; H, 6.0; N, 6.6%. ^{1}H NMR (250.13 MHz, $CDCl_3$): δ=1.24–1.87 (10H, m, 5CH$_2$), 1.35 (3H, t, *J*=7.5 Hz, N–CH$_2$*Me*), 3.64 and 3.97 (6H, 2s, 2O*Me*), 3.65 (1H, m, N–CH), 3.83 (2H, t, *J*=7.5 Hz, N–C*H*$_2$Me), 6.93–7.45 (4H, m, arom.); ^{13}C NMR (62.82 MHz, $CDCl_3$): δ=12.63 (N–CH$_2$*Me*), 25.07, 25.15, 26.06, 33.01 and 33.55 (5CH$_2$), 35.74 (N–*C*H$_2$Me), 53.15 and 53.57 (2O*Me*), 57.20 (N–CH), 86.28 (C_{spiro}), 109.49 (C), 123.56 (CH), 124.66, and 125.00 (2C), 131.94 (CH), 138.75 (C), 140.59 (CH), 144.12 (C), 154.69, 160.13 and 162.30 (3C=O), 170.10 (C=N).

3.2.9. Selected data for 4i. (0.41 g, mp 139°C, yield 85%); IR (KBr) (ν_{max}, cm^{-1}): 1745, 1740, 1720 (3C=O), 1678 (C=N); Anal. calcd for $C_{27}H_{34}N_2O_6$ (482.56): C, 67.20; H, 7.10; N, 5.81%. Found: C, 67.1; H, 7.2; N, 5.8%. ^{1}H NMR (250.13 MHz, $CDCl_3$): δ=0.97, 1.13, 1.37 and 1.39 (12H, 4d, *J*=7.5 Hz, 2O–CH*Me*$_2$), 1.27 (3H, t, *J*=7.5 Hz, N–CH$_2$*Me*), 1.24–1.75 (10H, m, 5CH$_2$), 3.62 (1H, m, N–C*H*), 3.82 (2H, q, *J*=7.5 Hz, N–C*H*$_2$Me), 4.90 and 5.31 (2H, 2septed, 2OC*H*Me$_2$), 6.92–7.45 (4H, m, arom.); ^{13}C NMR (62.89 MHz, $CDCl_3$): δ=12.88 (N–CH$_2$*Me*), 21.63, 2121.87, 22.04 and 22.10 (4Me of O–CH*Me*$_2$), 24.87, 24.95, 26.16, 33.52 and 33.57 (5CH$_2$ of cyclohexyl), 35.81 (N–*C*H$_2$Me), 56.66 (N–CH), 70.21 and 70.69 (2O–*C*HMe$_2$), 86.45 (C_{spiro}), 109.23 (CH), 123.46 (C), 125.06 (CH), 125.19 (CH), 131.81 (CH), 138.4 (C), 139.58 (C), 140.07 (C), 152.57, 160.35 and 162.43 (3C=O), 170.11 (C=N).

3.2.10. Selected data for 4j. (0.40 g, mp 164°C, yield 90%); IR(KBr) (ν_{max}, cm^{-1}): 1750, 1735 and 1725 (3C=O), 1683 (C=N); MS(CI), (*m*/*z*, %): 455 (MH$^+$, 30), 357 (M$^+$−$C_6H_{11}N$, 100), 285 (M$^+$−C_6H_{11}N–CO_2Et, 35); Anal. calcd for $C_{25}H_{30}N_2O_6$ (454.52): C, 66.06; H, 6.65; N, 6.16%. Found: C, 66.2; H, 6.7; N, 6.2%. ^{1}H NMR (250.13 MHz, $CDCl_3$): δ=1.08 (3H, t, *J*=7.3 Hz, N–CH$_2$*Me*), 1.37 (6H, 2t, *J*=7.3, 7.7 Hz, 2OCH$_2$*Me*), 1.24–1.87 (10H, m, 5CH$_2$), 3.62 (1H, m, N–CH), 3.82 (2H, m, 2OC*H*$_a$*H*$_b$Me), 4.07 (2H, m, 2OC*H*$_a$*H*$_b$Me), 4.42 (2H, q,

J=7.4 Hz, N–CH_2Me), 6.92–7.45 (4H, m, arom.); ^{13}C NMR (62.89 MHz, $CDCl_3$: δ=12.70 (N–CH_2Me), 14.05 and 14.42 (2OCH_2Me), 25.01, 25.10, 26.09, 33.10 and 33.55 (5CH_2), 35.77 (N–CH$_2$Me), 57.03 (N–CH), 62.21 and 62.77 (2OCH$_2$Me), 86.29 (C_{spiro}), 109.39 (C), 123.51 (CH), 124.90 and 125.01 (2C), 131.83 (CH), 138.88 (C), 140.47 (CH), 144.19 (C), 154.75, 160.86 and 161.71 (3C=O), 170.26 (C=N of iminolacton).

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