An Intramolecular Hetero Diels-Alder (Povarov) Approach to the Synthesis of the Alkaloids Luotonin A and Camptothecin

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Experimental Procedure

General Synthetic Methods

THF, hexanes, benzene and toluene were distilled from sodium metal/benzophenone ketyl under argon. CH₂Cl₂ and MeCN were distilled from CaH₂ under argon. All other solvents were obtained as ACS grade from commercial suppliers and used directly. All reagents, unless otherwise stated, were used as received from commercial suppliers (Aldrich, Fischer, Strem or Lancaster). All glassware, unless otherwise stated, was flame dried under a stream of dry nitrogen. All reactions, unless stated otherwise, were performed under an atmosphere of dry nitrogen. Melting points were obtained on a Fisher-Johns melting point apparatus and are uncorrected. FT-IR spectra were obtained on a Perkin-Elmer Spectrum 1000 spectrometer, with samples loaded as films on NaCl plates. ¹H and ¹³C NMR spectra were obtained on Varian Mercury 300 or Unity 400 or 500 spectrometers as solutions in deuterated solvents (normally CDCl₃ or CD₃OD, obtained from Cambridge Isotope Labs) and referenced to their corresponding solvent peaks (e.g., CDCl₃, δ 7.26 ppm; CD₃OD, δ 3.31 ppm for proton resonances and CDCl₃, δ 77.23 ppm; CD₃OD, δ 49.15 ppm; for carbon resonances). Low-resolution mass spectra (MS) were recorded on a Bell and Howell 21-490 spectrometer and high-resolution mass spectra (HRMS) were recorded on an AEI MS3074 spectrometer. Flash column chromatography on silica gel (60 Å, 230-400 mesh, low acidity, obtained from Silicycle Inc.) was performed with reagent grade solvents. Analytical thin-layer chromatography (TLC) was performed on pre-coated silica gel plates (Alugram SIL G/UV₂₅₄ purchased from Rose Scientific Ltd or Silicycle Inc.), visualized with a UV₂₅₄ lamp (Spectroline, Longlife filter) and stained with 20% ninhydrin or KMnO₄.

6-Dimethoxymethyl-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile **5**

Pyruvic aldehyde dimethylacetal (3.63 mL, 0.03 mol) and dimethylformamide dimethylacetal (7.31 mL, 0.45 mol) in anhydrous DMF (20 mL) were heated at 80 °C for 3 hours, after which time, the solvent was removed under reduced pressure. A solution of the crude enamine in DMF (10 mL) was added dropwise to a stirred solution containing sodium methoxide (3.56 g, 0.07 mol) and 2-cyanoacetamide (2.77 g, 0.03 mol) in DMF (40 mL). The resulting mixture was heated at 100 °C for 3 hours and then concentrated under reduced pressure. CH₂Cl₂ (100 mL) and water (60 mL) were added to the residue, and the pH adjusted to 7 by the addition of HCl (1 M soln.). The CH₂Cl₂ layer was separated and the aqueous phase was extracted with CH₂Cl₂ (4 x 100 mL). The combined organic extracts were washed with brine (1 x 50 mL), dried (MgSO₄) and concentrated under reduced pressure. The crude product was recrystallized (EtOAc/Hexanes) to give 6-dimethoxymethyl-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile **5** (1.80 g, 29%) as a pale yellow solid. m.p. = 197-199 °C; *R_f* = 0.03 (50% EtOAc:Hexanes); IR (film) 3218, 1651, 1385, 1202, 1114, 913 cm⁻¹; δ_H (300 MHz, CDCl₃) 6.33 (1H, s), 5.30 (1H, s), 3.45 (6H, s), 2.48 (3H, s); δ_C (75 MHz, CDCl₃) 161.4, 161.0, 147.6, 114.7, 106.4, 104.4, 98.3, 53.9, 21.5; MS (EI) *m/e* (rel intensity) 208 (39%), 177 (100%), 119 (51%), 75 (94%); HRMS (EI) *m/e* C₁₀H₁₂N₂O₃ calcd. 208.0848, found 208.0844.

6-Dimethoxymethyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile

To a suspension of 6-dimethoxymethyl-4-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile **5** (400 mg, 1.92 mmol) in a mixture of water (0.15 mL) and toluene (15 mL) was added potassium carbonate (531 mg, 3.84 mmol), lithium bromide (334 mg, 3.84 mmol), TBAB (61 mg, 0.19

mmol) and propargyl bromide (321 μ L, 2.88 mmol, 80% soln. in toluene). The reaction mixture was heated for 1 hour at 100 $^{\circ}$ C. The inorganic residues were removed by filtration over Celite, washing thoroughly with CH_2Cl_2 and the filtrate concentrated under reduced pressure. Flash chromatography (20% EtOAc:Hexanes) gave 6-dimethoxymethyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile (404 mg, 86%) as a pale yellow solid. m.p. = 78-80 $^{\circ}$ C; R_f = 0.23 (50% EtOAc:Hexanes); IR(film) 3306, 2940, 2253, 2228, 1655, 1604, 1552, 1450, 1389, 1353, 1187, 1124, 1063, 912 cm^{-1} ; δ_{H} (500 MHz, CDCl_3) 6.49 (1H, s), 5.46 (1H, s), 5.00 (2H, d, J = 2.5 Hz), 3.40 (6H, s), 2.46 (3H, s), 2.29 (1H, t, J = 2.5 Hz); δ_{C} (125 MHz, CDCl_3) 159.8, 158.6, 146.6, 114.6, 108.3, 104.8, 98.4, 77.4, 72.5, 53.7, 33.2, 21.3; MS (EI) m/e (rel intensity) 246 (29%), 231 (100%), 201 (19%), 185 (17%); HRMS (EI) m/e $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_3$ calcd. 246.1004, found 246.1002.

6-Formyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile 6

To 6-dimethoxymethyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile (74 mg, 0.30 mmol) was added a mixture of HCl (0.5 mL, conc.), glacial acetic acid (1.5 mL) and water (2.5 mL). The reaction was heated for 1 hour at 60 $^{\circ}$ C then allowed to cool to room temperature. After being poured into water (20 mL), NaHCO_3 (20 mL sat. aq. soln.) and ethyl acetate (4 x 20 mL) were added. The combined organic extracts were washed with brine (2 x 10 mL, sat. aq. soln.), dried over MgSO_4 , filtered and the solvent removed *in vacuo* to give 6-formyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile **6** (57 mg, 95%) as a white solid. m.p. = 174-176 $^{\circ}$ C; IR (film) 3422, 1638, 1266 cm^{-1} ; δ_{H} (300 MHz, $\text{CDCl}_3/\text{CD}_2\text{Cl}_2$) 9.74 (1H, s), 6.79 (1H, s), 5.28 (2H, d, J = 2.0 Hz), 2.57 (3H, s), 2.29 (1H, t, J = 2.5 Hz); δ_{C} (75 MHz, $\text{CDCl}_3/\text{CD}_2\text{Cl}_2$) (1 carbon missing) 184.1, 158.5, 157.9, 141.0, 119.4, 113.7, 77.3, 73.1, 32.8, 21.1; MS (EI) m/e (rel intensity) 200 (61%), 172 (100%), 143 (50%); HRMS (EI) m/e $\text{C}_{11}\text{H}_8\text{N}_2\text{O}_2$ calcd. 200.0586, found 200.0583.

7-Methyl-9-oxo-9,11-dihydroindolizino[1,2-*b*]quinoline-8-carbonitrile **7a**

To 6-formyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydropyridine-3-carbonitrile **6** (40 mg, 0.20 mmol) in acetonitrile (10 mL) was added aniline (18 μ L, 0.20 mmol) followed by Dy(OTf)₃ (12 mg, 0.02 mmol). The reaction mixture was heated at 50 °C for 16 hours, then allowed to cool. Removal of the solvent followed by recrystallization from hot methanol gave 7-methyl-9-oxo-9,11-dihydroindolizino[1,2-*b*]quinoline-8-carbonitrile **7a** (39 mg, 71%) as a fine yellow powder. m.p. $\geq$ 300 °C; δ_{H} (300 MHz, CD₂Cl₂+CD₃OD) 8.50 (1H, s), 8.21 (1H, d, J = 9.0 Hz), 8.00 (1H, d, J = 8.5 Hz), 7.86 (1H, ddd, J = 8.5, 7.0, 1.5 Hz), 7.71 (1H, ddd, J = 8.0, 7.0, 1.0 Hz), 7.32 (1H, s), 5.30 (2H, s), 2.64 (3H, s); δ_{C} (75 MHz, CDCl₃+CD₃OD) 162.5, 150.9, 148.8, 131.7, 131.0, 129.2, 129.0, 128.6, 128.2, 103.2, 77.2, 50.4, 21.3; MS (EI) m/e (rel intensity) 273 (100%), 244 (34%), HRMS (EI) m/e C₁₇H₁₁N₃O calcd. 273.0902, found 273.0903. (Literature: Fortunak, J. M. D.; Mastrocola, A. R.; Mellinger, M.; Sisti, N. J.; Wood, J. L.; Zhuang, Z.-P. *Tetrahedron Lett.* **1996**, 37, 5679-5682).

2-Methoxy-7-methyl-9-oxo-9,11-dihydroindolizino[1,2-*b*]quinoline-8-carbonitrile **7b**

To 6-formyl-4-methyl-2-oxo-1-prop-2-ynyl-1,2-dihydro-pyridine-3-carbonitrile **6** (50 mg, 0.25 mmol) in acetonitrile (10 mL) was added *p*-anisidine (31 mg, 0.25 mmol) followed by Dy(OTf)₃ (15 mg, 0.025 mmol). The reaction mixture was heated at 50 °C for 16 hours, then allowed to

cool. Removal of the solvent followed by recrystallization from hot methanol gave 2-methoxy-7-methyl-9-oxo-9,11-dihydroindolizino[1,2-*b*]quinoline-8-carbonitrile **7b** (49 mg, 64%) as a fine yellow powder. m.p. ≥ 300 °C; δ_{H} (300 MHz, $\text{CDCl}_3 + \text{CD}_3\text{OD}$) 8.39 (1H, s), 8.11 (1H, d, $J = 9.0$ Hz), 7.52 (1H, m), 7.33 (1H, s), 7.26 (1H, m), 5.31 (2H, s), 4.02 (3H, s), 2.67 (3H, s); MS (EI) m/e (rel intensity) 303 (100%), 260 (22%); HRMS (EI) m/e $\text{C}_{18}\text{H}_{13}\text{N}_3\text{O}_2$ calcd. 303.1008, found 303.1015. (Literature: Fortunak, J. M. D.; Mastrocola, A. R.; Mellinger, M.; Sisti, N. J.; Wood, J. L.; Zhuang, Z.-P. *Tetrahedron Lett.* **1996**, 37, 5679-5682).

2-Amino-*N*-prop-2-ynylbenzamide **9**

To a stirred solution of isatoic anhydride (7.90 g, 48 mmol) in DMF (40 mL) at 40-50 °C, was added a solution of propargylamine (4.98 mL, 73 mmol) in DMF (20 mL) dropwise. The resulting mixture was stirred at 40-50 °C for 3 hours, allowed to cool, and the DMF removed by rotary evaporation. Water (200 mL) was added and the pH adjusted to 9 using sodium hydroxide (10 mL, 1.0 M). The resulting solid was extracted using CH_2Cl_2 (3 x 100 mL) and washed with brine (1 x 50 mL). The combined organic extracts were dried over MgSO_4 , decolourized using charcoal, filtered and the solvent removed *in vacuo* to give 2-amino-*N*-prop-2-ynylbenzamide **9** (5.47 g, 65%) as a white solid. m.p. = 98-100 °C; IR (film) 3426, 3289, 2252, 1638, 1618, 1587, 1529, 1266, 1159 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 7.33 (1H, dd, $J = 8.0, 1.5$ Hz), 7.22 (1H, ddd, $J = 8.5, 7.5, 1.5$ Hz), 6.69-6.61 (2H, m), 6.29 (1H, bs), 5.55 (2H, bs), 4.19 (1H, dd, $J = 5.0, 2.5$ Hz), 2.27 (1H, t, $J = 2.5$ Hz); δ_{C} (75 MHz, CDCl_3) 168.9, 148.9, 132.6, 127.2, 117.3, 116.5, 114.9, 79.6, 71.7, 29.4; MS (EI) m/e (rel intensity) 174 (54%), 120 (100%), 119 (54%), 92 (57%); HRMS (EI) m/e $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}$ calcd. 174.0793, found 174.0790.

Acetic acid (2-prop-2-ynylcarbamoylphenylcarbamoyl)-methyl ester **10**

To a stirred solution of 2-amino-*N*-prop-2-ynylbenzamide **9** (0.80 g, 4.60 mmol) and triethylamine (962 μ L, 6.90 mmol) in benzene (20 mL), was added a solution of acetoxyacetyl chloride (544 μ L, 5.06 mmol) in benzene (4 mL) dropwise at 40 °C. After the reaction mixture was allowed to cool to room temperature and stand overnight, the solvent was removed under reduced pressure and the resulting residue was shaken with water (10 mL). The precipitate was collected by filtration and recrystallised (EtOH) to give acetic acid (2-prop-2-ynylcarbamoylphenylcarbamoyl)-methyl ester **10** (0.861 mg, 68%) as a white solid. m.p. = 167-169 °C; IR (film) 3273, 1741, 1686, 1626, 1596, 1526, 1448, 1227, 1055, 914 cm^{-1} ; δ_{H} (300 MHz, CDCl_3) 11.62 (1H, bs), 8.61 (1H, d, J = 8.0 Hz), 7.49 (2H, m), 7.11 (1H, t, J = 7.5 Hz), 6.57 (1H, bs), 4.71 (2H, s), 4.21 (2H, dd, J = 5.0 Hz, 2.5 Hz), 2.33 (3H, s), 2.31 (1H, t, J = 2.5 Hz); δ_{C} (100 MHz, CDCl_3) 170.0, 168.3, 166.2, 138.6, 133.0, 126.6, 123.4, 121.5, 119.9, 78.9, 72.2, 62.8, 29.6, 20.7; MS (EI) m/e (rel intensity) 274 (19%), 201 (100%), 146 (75%); HRMS (EI) m/e $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_4$ calcd. 274.0954, found 274.0953.

Acetic acid 4-prop-2-ynylimino-4*H*-benzo[*d*][1,3]oxazin-2-ylmethyl ester **11**

To a stirred solution of acetic acid (2-prop-2-ynylcarbamoylphenylcarbamoyl)-methyl ester **10** (300 mg, 1.10 mmol) in CH_2Cl_2 (20 mL), was added triphenylphosphine (1.44 g, 5.47 mmol),

iodine (1.39 g, 5.47 mmol) and Hünig's base (1.89 mL, 10.9 mmol). The reaction mixture was stirred for 5 hours and quenched by the addition of sodium carbonate (20 mL, sat. aq. soln.) and extracted with CH₂Cl₂ (3 x 30 mL), dried (MgSO₄), filtered and concentrated under reduced pressure. Column chromatography (10% EtOAc:Hexanes, pre-treated with 2% triethylamine) gave acetic acid 4-prop-2-ynylimino-4*H*-benzo[*d*][1,3]oxazin-2-ylmethyl ester **11** (250 mg, 89%) as a light brown solid. m.p. = 97-99 °C; *R*_f = 0.66 (50% EtOAc:Hexanes); IR (film) 3243, 1735, 1681, 1652, 1462, 1439, 1259, 1221, 1046 cm⁻¹; δ_H (300 MHz, CDCl₃) 8.12 (1H, dd, *J* = 8.0, 1.0 Hz), 7.56 (1H, ddd, *J* = 9.0, 7.0, 1.5 Hz), 7.42-7.33 (2H, m), 4.94 (2H, s), 4.25 (2H, d, *J* = 2.5 Hz), 2.25 (1H, t, *J* = 2.5 Hz), 2.22 (3H, s); δ_C (75 MHz, CDCl₃) 170.2, 154.3, 147.9, 140.8, 133.5, 128.7, 126.4, 126.1, 119.3, 81.2, 70.3, 61.4, 35.5 20.6; MS (EI) *m/e* (rel intensity) 256 (14%), 183 (100%), 84 (18%); HRMS (EI) *m/e* C₁₄H₁₂N₂O₃ calcd. 256.0848, found 256.0854.

Acetic acid 4-oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-ylmethyl ester **12**

A solution of acetic acid 4-prop-2-ynylimino-4*H*-benzo[*d*][1,3]oxazin-2-ylmethyl ester **11** (300 mg, 1.20 mmol) in piperidine (3.0 mL) and ethylacetate (12 mL) was stirred for 1 hour at room temperature and then concentrated under reduced pressure. The crude imine was re-dissolved in ethyl acetate (15 mL), SiO₂ (2.50 g, 42.0 mmol) was added and the reaction mixture stirred overnight. The suspension was filtered and the filtrate concentrated under reduced pressure. Flash chromatography (20% EtOAc:Hexanes, pre-treated with 2% triethylamine) gave acetic acid 4-oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-ylmethyl ester **12** (256 mg, 85%) as a white solid. m.p. = 106-108 °C; *R*_f = 0.65 (50% EtOAc:Hexanes); IR (film) 3265, 1749, 1687, 1607, 1475, 1229, 1041, 774 cm⁻¹; δ_H (400 MHz, CDCl₃) 8.29 (1H, dd, *J* = 8.0, 1.5 Hz), 7.77 (1H, ddd, *J* = 8.0, 7.0, 1.5 Hz), 7.70 (1H, ddd, *J* = 8.0, 1.5, 0.5 Hz), 7.51 (1H, ddd, *J* = 8.0, 7.0, 1.0 Hz), 5.33 (2H, s), 4.96 (2H, d, *J* = 2.5 Hz), 2.35 (1H, t, *J* = 2.5 Hz), 2.23 (3H, s); δ_C (100 MHz, CDCl₃) 169.9, 161.0, 150.0, 146.6, 134.6, 127.7, 127.6, 127.0, 120.8, 77.2, 73.0, 63.8, 32.2, 20.7; MS

(EI) m/e (rel intensity) 256 (35%), 213 (77%), 197 (100%), 185 (28%), 129 (30%); HRMS (EI) m/e C₁₄H₁₂N₂O₃ calcd. 256.0848, found 256.0851.

2-Hydroxymethyl-3-prop-2-ynyl-3H-quinazolin-4-one

To a stirred solution of acetic acid 4-oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-ylmethyl ester **12** (600 mg, 2.34 mmol) in THF (30 mL), was added sodium hydroxide solution (1.0 M, 4.69 mL, 4.69 mmol) dropwise, followed by water (3.8 mL). The reaction mixture was stirred for 1 hour and then concentrated under reduced pressure. The residue was treated with water (40 mL), the pH adjusted to 4.5 by the addition of HCl (1.0 M, 4-5 mL) and then extracted with ethyl acetate (3 x 40 mL). The combined organic extracts were dried (MgSO₄) and concentrated under reduced pressure. Purification by column chromatography on silica (40% EtOAc:Hexanes pretreated with 2% triethylamine) gave 2-hydroxymethyl-3-prop-2-ynyl-3H-quinazolin-4-one (417 mg, 83%) as a white solid. R_f = 0.50 (50% EtOAc:Hexanes); m.p. = 126-128 °C ; IR(film) 3425, 1654, 1609, 1466, 1427, 1384, 1266, 1273, 1024 cm⁻¹; δ_H (400 MHz, CDCl₃) 8.25 (1H, dd, J = 8.0, 1.5 Hz), 7.76 (1H, ddd, J = 8.0, 7.0, 1.5 Hz), 7.65 (1H, d, J = 8.0 Hz), 7.48 (1H, dd, J = 8.0, 7.0 Hz), 4.84 (2H, d, J = 2.5 Hz), 4.82 (2H, s), 2.64 (1H, br s), 2.34 (1H, t, J = 2.5 Hz); δ_C (100 MHz, CDCl₃) 160.8, 154.3, 145.7, 134.7, 127.2, 127.1, 126.6, 120.3, 76.6, 73.1, 60.1, 30.7; MS (EI) m/e (rel intensity) 214 (52%), 197 (39%), 185 (39%), 175 (100%); HRMS (EI) m/e C₁₂H₁₀N₂O₂ calcd. 214.0742, found 214.0741.

4-Oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-carbaldehyde **13**

To a stirred solution of 2-hydroxymethyl-3-prop-2-ynyl-3*H*-quinazolin-4-one (100 mg, 0.467 mmol) and pyridine (378 μ L, 4.67 mmol) in CH_2Cl_2 (30 mL), was added Dess-Martin periodinane (218 mg, 0.514 mmol) at 0 $^\circ\text{C}$. The reaction mixture was stirred for 15 minutes at 0 $^\circ\text{C}$, followed by a further 1 hour at room temperature. Additional Dess-Martin periodinane (218 mg, 0.514 mmol) was added, and the reaction mixture stirred for 4 hours at room temperature. Filtration over Celite, removal of the solvent *in vacuo*, and column chromatography on silica (20% EtOAc:Hexanes) gave 4-oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-carbaldehyde **13** (77 mg, 78%) as a white solid. m.p. = 169 $^\circ\text{C}$ (decomp.); IR (film) 3423, 3053, 2986, 2305, 1724, 1685, 1420, 1265 cm^{-1} ; δ_{H} (400 MHz, CDCl_3) 9.76 (1H, s), 8.37 (1H, d, $J = 7.5$ Hz), 7.92-7.86 (2H, m), 7.68 (1H, ddd, $J = 8.0, 6.0, 3.0$ Hz); 5.40 (2H, d, $J = 2.5$ Hz), 2.27 (1H, t, $J = 2.5$ Hz); δ_{C} (75 MHz, CDCl_3) 187.5, 160.5, 146.1, 145.9, 135.0, 130.2, 128.7, 127.4, 121.7, 78.1, 72.2, 31.5; MS (EI) m/e (rel intensity) 212 (49%), 184 (100%), 155 (27%), 129 (36%); HRMS (EI) m/e $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_2$ calcd. 212.0586, found 212.0591.

Luotonin A

To 4-oxo-3-prop-2-ynyl-3,4-dihydroquinazolin-2-carbaldehyde **13** (50 mg, 0.236 mmol) in acetonitrile (10 mL) was added aniline (22 μ L, 0.236 mmol) and $\text{Dy}(\text{OTf})_3$ (15 mg, 0.024 mmol). The reaction mixture was stirred at room temperature for 24 hours then water (20 mL) was added

and the mixture extracted with CH₂Cl₂ (3 x 30 mL). The combined organic extracts were washed with brine (1 x 40 mL), dried over MgSO₄, filtered and the solvent removed *in vacuo*. Column chromatography on silica (20% EtOAc:Hexanes) provided Luotonin A (67 mg, 51%) as a pale yellow solid. m.p. = 278-280 °C (decomp.); IR (film) 3445, 3054, 2987, 2306, 1633, 1421, 1265, 1018, 896 cm⁻¹; δ_{H} (500 MHz, CDCl₃ + CD₃OD (3:1)) 8.33 (1H, s) 8.17 (2H, d, *J* = 8.5 Hz), 7.85 (1H, d, *J* = 8.0 Hz), 7.78 (1H, d, *J* = 8.0 Hz), 7.67 (2H, m), 7.50 (1H, t, *J* = 8.0 Hz), 7.40 (1H, t, *J* = 7.0 Hz), 5.16 (2H, s); δ_{C} (125 MHz, CDCl₃) 160.7, 152.3, 150.3, 148.9 (2 carbons), 134.5, 131.7, 130.5, 129.8, 129.3, 128.6, 128.3, 128.0, 127.8, 127.3, 125.9, 120.6, 47.1; MS (EI) *m/e* (rel intensity) 285 (100%), 257 (15%), 207 (19%); HRMS (EI) *m/e* C₁₈H₁₁N₃O calcd. 285.0902, found 285.0900. (Literature: Mhaske, S. B.; Argade, N. P. *J. Org. Chem.* **2004**, *69*, 4563-4566 and references therein).