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Supporting Information

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Zirconium Triflate-Catalyzed Direct Coupling Reaction of Lactams with Heterocyclic Arenes under Atmospheric Oxygen

Teruhisa Tsuchimoto,* Yoshitaka Ozawa, Ryoju Negoro, Eiji Shirakawa,* and Yusuke Kawakami

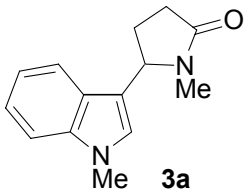
[*] Dr. T. Tsuchimoto, Y. Ozawa, R. Negoro, Prof. Dr. E. Shirakawa,[+] Prof. Dr. Y. Kawakami
Graduate School of Materials Science
Japan Advanced Institute of Science and Technology
Asahidai, Tatsunokuchi, Ishikawa 923-1292 (Japan)
Fax: (+81) 761-51-1635
E-mail: tsuchimo@jaist.ac.jp

[+] Present address:
Department of Chemistry
Graduate School of Science
Kyoto University
Sakyo-ku, Kyoto 606-8502 (Japan)
Fax: (+81) 75-753-3985
E-mail: shirakawa@kuchem.kyoto-u.ac.jp

General Remarks. All manipulations of the oxidative coupling reactions were conducted with a standard Schlenk technique under an oxygen atmosphere. Nuclear magnetic resonance spectra were taken on a Varian Gemini 300 (^{1}H, 300 MHz; ^{13}C, 75 MHz) or a Varian INOVA 400 (^{13}C, 101 MHz) spectrometer using tetramethylsilane (^{1}H and ^{13}C) as an internal standard. The preparative recycling gel permeation chromatography was performed with JAI LC-908 equipped with JAIGEL-1H and -2H (chloroform as an eluent). Preparative recycling silica gel column chromatography was performed with JAI LC-908 equipped with JAIGEL-SIL-043-15 (ethyl acetate as an eluent). Analytical gas-liquid phase chromatography was performed on a Shimadzu model GC-18A instrument equipped with a capillary column of CP-SIL 5 CB (30 m x 0.25 mm) using helium as carrier gas. High-resolution mass spectra were obtained with a Bruker Bio APEX 70e or a JEOL JMS-HX110A spectrometer. All melting points were measured with a Yanaco Micro Melting Point apparatus and uncorrected. Unless otherwise noted, reagents were commercially available and used without further purification. Zirconium triflate was prepared according to the literature procedure.[1] *N*-arylindole and -pyrrole derivatives were prepared according to literature procedures.[2] *N*-methyl-5-bromoindole and *N*-methyl-5-methoxyindole were prepared from the corresponding indole derivatives according to the reported procedure.[3] Anhydrous 1-methylpyrrolidin-2-one was purchased from Kanto Chemicals and used as received.

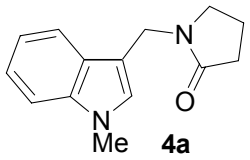
Zr(OTf)$_4$-Catalyzed Oxidative Coupling Reaction of Lactams with Heterocyclic arenes. A General Procedure. $Zr(OTf)_4$ (3.4 mg, 5.0 μmol) was placed in a 20 mL Schlenk tube, which was heated at 150 °C in vacuo for 2 h. The tube was cooled down to room temperature and was filled with oxygen. To this were added 1-methyl-2-pyrrolidinone (2.0 mL, 20.7 mmol) or 3-methyloxazolidin-2-one (2.0 mL, 23.1 mmol) and a heterocyclic arene (1.0 mmol) successively, and the resulting mixture was stirred at 130 °C. After the time specified in Table 2 and Eqs. 2–4, the mixture was diluted with diethyl ether (100 mL) and washed successively with water (10 mL x 3), saturated $NaHCO_3$ aqueous solution (5 mL) and brine (5 mL), and then dried over

anhydrous magnesium sulfate. Filtration through a pad of Celite and evaporation of the solvent followed by column chromatography on silica gel gave the corresponding coupling products as a mixture of isomers. Further isolation was performed with preparative recycling column chromatography on silica gel. The results are summarized in Table 2 and Eqs. 2–4.



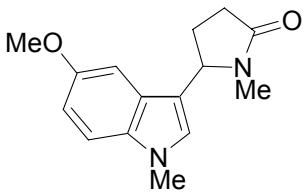
3a

1-Methyl-5-(1-methylindol-3-yl)pyrrolidin-2-one (3a). A pale yellow solid, mp 123–124 °C, R_f 0.17 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.12–2.54 (m, 1 H), 2.39–2.69 (m, 3 H), 2.70 (s, 3 H), 3.78 (s, 3 H), 4.84 (t, *J* = 7.1 Hz, 1 H), 6.98 (s, 1 H), 7.2 (ddd, *J* = 7.8, 6.8, 1.2 Hz, 1 H), 7.27 (ddd, *J* = 8.3, 6.8, 1.2 Hz, 1 H), 7.34 (d, *J* = 8.4 Hz, 1 H), 7.52 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 27.1, 27.9, 30.6, 32.7, 57.7, 109.7, 114.0, 119.1, 119.7, 122.3, 125.8, 127.1, 137.7, 175.3. HRMS (EI) Calcd for $C_{14}H_{16}N_2O$: M^+, 228.1262. Found: *m*/*z* 228.1252.

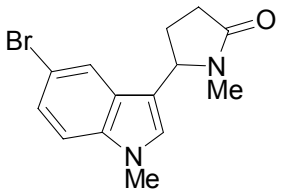


4a

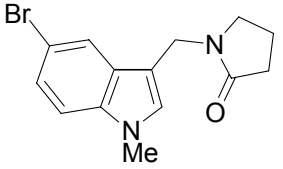
1-(1-Methylindol-3-ylmethyl)pyrrolidin-2-one (4a). A pale yellow oil, R_f 0.22 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.91 (quint, *J* = 7.8 Hz, 2 H), 2.41 (t, *J* = 8.1 Hz, 2 H), 3.27 (t, *J* = 7.2 Hz, 2 H), 3.77 (s, 3 H), 4.62 (s, 2 H), 7.03 (s, 1 H), 7.13 (ddd, *J* = 8.1, 6.6, 1.2 Hz, 1 H), 7.25 (ddd, *J* = 7.7, 6.8, 1.2 Hz, 1 H), 7.31 (dt, *J* = 8.1, 0.9 Hz, 1 H), 7.67 (dt, *J* = 7.8, 0.9 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 17.4, 31.2, 32.7, 37.4, 46.5, 109.3, 110.0, 119.4, 119.5, 122.0, 127.4, 128.4, 137.2, 174.6. HRMS (EI) Calcd for $C_{14}H_{16}N_2O$: M^+, 228.1262. Found: *m*/*z* 228.1255.



1-Methyl-5-(5-methoxy-1-methylindol-3-yl)pyrrolidin-2-one. A pale yellow solid, mp 92–94 °C, R_f 0.24 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.10–2.24 (m, 1 H), 2.38–2.68 (m, 3 H), 2.71 (s, 3 H), 3.75 (s, 3 H), 3.84 (s, 3 H), 4.80 (t, *J* = 7.5 Hz, 1 H), 6.91–6.94 (m, 3 H), 7.23 (dd, *J* = 7.5, 2.1 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 27.0, 27.9, 30.6, 32.9, 56.0, 57.6, 100.9, 110.5, 112.3, 113.3, 126.2, 127.6, 133.0, 154.2, 175.2. HRMS (ESI) Calcd for $C_{15}H_{18}N_2O_2Na$: M^++Na, 281.1265. Found: *m*/*z* 281.1247.

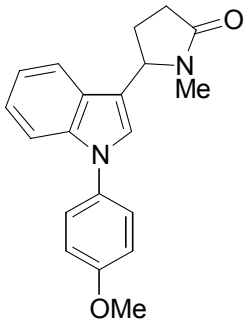


1-Methyl-5-(5-bromo-1-methylindol-3-yl)pyrrolidin-2-one. A white solid, mp 163–165 °C, R_f 0.14 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.06–2.20 (m, 1 H), 2.38–2.66 (m, 3 H), 2.70 (s, 3 H), 3.77 (s, 3 H), 4.78 (t, *J* = 7.2 Hz, 1 H), 6.97 (s, 1 H), 7.20 (d, *J* = 8.7 Hz, 1 H), 7.34 (dd, *J* = 8.7, 1.8 Hz, 1 H), 7.64 (d, *J* = 1.8 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 27.1, 28.0, 30.4, 32.9, 57.3, 111.2, 113.1, 113.6, 121.4, 125.2, 127.5, 128.1, 136.3, 175.2. HRMS (ESI) Calcd for $C_{14}H_{15}BrN_2ONa$: M^++Na, 329.0264. Found: *m*/*z* 329.0261.

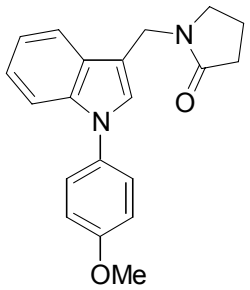


1-(5-Bromo-1-methylindol-3-ylmethyl)pyrrolidin-2-one. A pale yellow oil, R_f 0.20 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.94 (quint, *J* = 7.5 Hz, 2 H), 2.41 (t, *J* = 8.1 Hz, 2 H), 3.27 (t, *J* = 6.9 Hz, 2 H), 3.75 (s, 3 H), 4.55 (s, 2 H), 7.04 (s, 1 H),

7.17 (d, J = 8.7 Hz, 1 H), 7.32 (dd, J = 8.7, 1.8 Hz, 1 H), 7.77 (d, J = 1.8 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 17.5, 31.0, 32.8, 37.1, 46.6, 109.7, 110.9, 113.1, 121.7, 124.9, 129.1, 129.6, 135.8, 174.8; HRMS (ESI) $C_{14}H_{15}BrN_2ONa$: M^++Na, 329.0264. Found: *m*/*z* 329.0270.

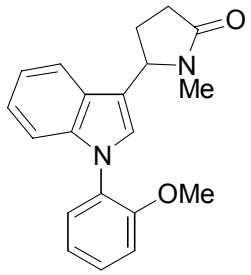


1-Methyl-5-[1-(4-methoxyphenyl)indol-3-yl]pyrrolidin-2-one. A pale yellow oil, R_f 0.34 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.19–2.32 (m, 1 H), 2.44 (m, 3 H), 2.77 (s, 3 H), 3.89 (s, 3 H), 4.91 (t, J = 6.8 Hz, 1 H), 7.04 (dt, J = 9.0, 2.8 Hz, 2 H), 7.16 (td, J = 7.4, 1.1 Hz, 1 H), 7.20 (s, 1 H), 7.25 (td, J = 7.7, 1.4 Hz, 1 H), 7.39 (dt, J = 9.0, 2.8 Hz, 2 H), 7.45 (d, J = 8.4 Hz, 1 H), 7.58 (d, J = 7.8 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.9, 28.1, 30.5, 55.6, 57.7, 110.9, 114.9, 115.9, 119.2, 120.4, 122.9, 126.0, 126.3, 126.4, 132.3, 137.4, 158.6, 175.3; HRMS (ESI) Calcd for $C_{20}H_{20}N_2O_2Na$: M^++Na, 343.1421. Found: *m*/*z* 343.1415.

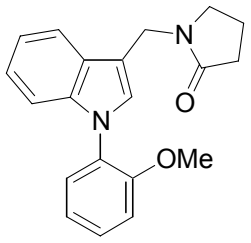


1-[1-(4-Methoxyphenyl)indol-3-ylmethyl]pyrrolidin-2-one. A pale yellow oil, R_f 0.40 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.94 (quint, J = 7.7 Hz, 2 H), 2.43 (t, J = 8.1 Hz, 2 H), 3.33 (t, J = 7.1 Hz, 2 H), 3.88 (s, 3 H), 4.68 (s, 2 H), 7.03 (dt, J = 8.7, 0.9 Hz, 2 H), 7.14–7.25 (m, 2 H), 7.25 (s, 1 H), 7.38 (dt, J = 8.7, 0.9 Hz, 2 H), 7.43–7.45 (m, 1 H), 7.72–7.75 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ17.4, 31.1,

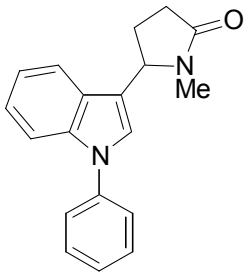
37.4, 46.6, 55.5, 11.5, 111.9, 114.8, 119.6, 120.4, 122.7, 125.9, 127.7, 127.9, 132.5, 136.8, 158.4, 174.7; HRMS (EI) Calcd for $C_{20}H_{20}N_2O_2$: M^+, 320.1524. Found: *m*/*z* 320.1500.



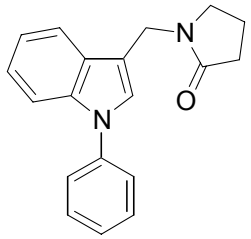
1-Methyl-5-[1-(2-methoxyphenyl)indol-3-yl]pyrrolidin-2-one. A white solid, mp 51–52 °C, R_f 0.20 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ2.21–2.35 (m, 1 H), 2.45–2.71 (m, 3 H), 2.78 (s, 3 H), 3.78 (s, 3 H), 4.92 (dd, *J* = 7.7, 6.6 Hz, 1 H), 7.05–7.24 (m, 6 H), 7.37–7.45 (m, 2 H), 7.55–7.58 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ26.9, 28.0, 30.5, 55.7, 57.7, 111.4, 112.5, 115.3, 118.9, 120.2, 120.9, 122.6, 126.0, 127.59, 127.64, 128.0, 128.8, 137.8, 154.5, 175.3; HRMS (EI) Calcd for $C_{20}H_{20}N_2O_2$: M^+, 320.1524. Found: *m*/*z* 320.1505.



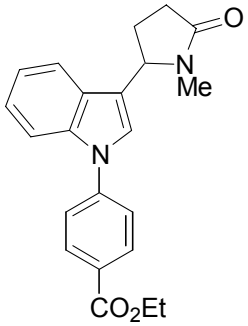
1-[1-(2-Methoxyphenyl)indol-3-ylmethyl]pyrrolidin-2-one. A pale yellow oil, R_f 0.29 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.94 (quint, *J* = 7.5 Hz, 2 H), 2.43 (t, *J* = 8.1 Hz, 2 H), 3.33 (t, *J* = 7.1 Hz, 2 H), 3.79 (s, 3 H), 4.69 (s, 2 H), 7.05–7.22 (m, 5 H), 7.25 (s, 1 H), 7.36–7.42 (m, 2 H), 7.72–7.75 (m, 1 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ17.5, 31.3, 37.6, 46.5, 55.7, 110.9, 111.3, 112.4, 119.4, 120.1, 120.9, 122.3, 127.4, 127.91, 127.85, 128.6, 128.8, 137.2, 154.3, 174.5; HRMS (ESI) Calcd for $C_{20}H_{20}N_2O_2Na$: M^++Na, 343.1421. Found: *m*/*z* 343.1414.



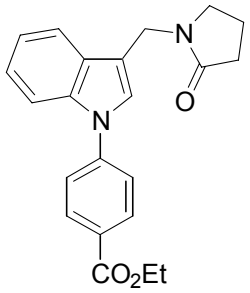
1-Methyl-5-(1-phenylindol-3-yl)pyrrolidin-2-one. A pale yellow oil, R_f 0.43 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) 2.19–2.33 (m, 1 H), 2.45–2.71 (m, 3 H), 2.78 (s, 3 H), 4.92 (dd, *J* = 8.4, 6.3 Hz, 1 H), 7.18 (ddd, *J* = 8.0, 7.4, 1.1 Hz, 1 H), 7.24–7.30 (m, 2 H), 7.35–7.41 (m, 1 H), 7.47–7.60 (m, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ26.9, 28.1, 30.5, 57.7, 111.1, 116.5, 119.3, 120.7, 123.1, 124.4, 126.0, 126.7, 126.8, 129.8, 136.9, 139.4, 175.3; HRMS (ESI) Calcd for $C_{19}H_{18}N_2ONa$: M^++Na, 313.1315. Found: *m*/*z* 313.1282.



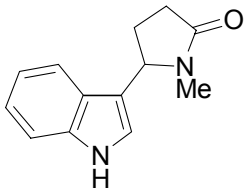
1-(1-Phenylindol-3-ylmethyl)pyrrolidin-2-one. A pale yellow oil, R_f 0.48 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.95 (quint, *J* = 7.5 Hz, 2 H), 2.44 (t, *J* = 8.1 Hz, 2 H), 3.33 (t, *J* = 7.1 Hz, 2 H), 4.69 (s, 2 H), 7.16–7.22 (m, 1 H), 7.22–7.28 (m, 1 H), 7.32 (s, 1 H), 7.32–7.39 (m, 1 H), 7.48–7.57 (m, 5 H), 7.74–7.77 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ17.4, 31.1, 37.4, 46.6, 110.6, 112.6, 119.7, 120.6, 122.9, 124.3, 126.6, 127.4, 128.2, 129.7, 136.3, 139.6, 174.7; HRMS (ESI) Calcd for $C_{19}H_{18}N_2ONa$: M^++Na, 313.1315. Found: *m*/*z* 313.1282.



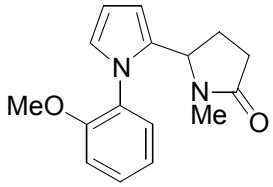
1-Methyl-5-[1-(4-ethoxycarbonylphenyl)indol-3-yl]pyrrolidin-2-one. A pale yellow oil, R_f 0.20 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.43 (t, *J* = 7.2 Hz, 3 H), 2.18–2.31 (m, 1 H), 2.45–2.73 (m, 3 H), 2.78 (s, 3 H), 4.43 (q, *J* = 7.2 Hz, 2 H), 4.92 (dd, *J* = 8.4, 6.3 Hz, 1 H), 7.19–7.24 (m, 1 H), 7.27–7.33 (m, 1 H), 7.29 (s, 1 H), 7.55–7.60 (m, 3 H), 7.64 (d, *J* = 8.4 Hz, 1 H), 8.21 (dt, *J* = 8.7, 2.1 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ14.2, 26.8, 28.1, 30.4, 57.6, 61.2, 111.1, 117.8, 119.6, 121.3, 123.3, 123.6, 125.4, 127.1, 128.4, 131.4, 136.5, 143.1, 166.0, 175.3; HRMS (EI) Calcd for $C_{22}H_{22}N_2O_3$: M^+, 362.1629. Found: *m*/*z* 362.1652.



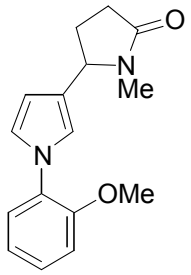
1-[1-(4-Ethoxycarbonylphenyl)indol-3-ylmethyl]pyrrolidin-2-one. A pale yellow oil, R_f 0.29 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.43 (t, *J* = 7.2 Hz, 3 H), 1.96 (quint, *J* = 7.7 Hz, 2 H), 2.44 (t, *J* = 8.0 Hz, 2 H), 3.33 (t, *J* = 7.5 Hz, 2 H), 4.42 (q, *J* = 7.2 Hz, 2 H), 4.69 (s, 2 H), 7.20–7.25 (m, 1 H), 7.26–7.31 (m, 1 H), 7.36 (s, 1 H), 7.58 (dt, *J* = 8.4, 1.8 Hz, 2 H), 7.62 (d, *J* = 8.1 Hz, 1 H), 7.76 (d, *J* = 7.5 Hz, 1 H), 8.20 (dt, *J* = 8.4, 1.5 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 17.5, 31.1, 37.3, 46.6, 61.1, 110.7, 113.9, 120.0, 121.3, 123.2, 123.4, 126.8, 128.1, 128.7, 131.3, 135.9, 143.3, 166.0, 174.8; HRMS (EI) Calcd for $C_{22}H_{22}N_2O_3$: M^+, 362.1629. Found: *m*/*z* 362.1628.



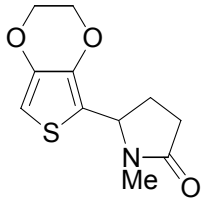
1-Methyl-5-(indol-3-yl)pyrrolidin-2-one.[4] A pale yellow solid, mp 136–137 °C, R_f 0.15 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.15–2.29 (m, 1 H), 2.40–2.67 (m, 3 H), 2.72 (s, 3 H), 4.87 (t, *J* = 7.2 Hz, 1 H), 7.12 (ddd, *J* = 8.0, 7.0, 1.1 Hz, 1 H), 7.14 (s, 1 H), 7.23 (ddd, *J* = 8.4, 7.3, 1.2 Hz, 1 H), 7.42 (d, *J* = 8.1 Hz, 1 H), 7.53 (dd, *J* = 8.1, 0.6 Hz, 1 H), 8.76 (br s, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.8, 27.9, 30.6, 57.8, 111.7, 115.2, 118.4, 120.0, 122.5, 122.6, 125.3, 137.0, 175.4.



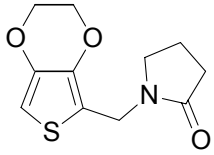
1-Methyl-5-[1-(2-methoxyphenyl)pyrrol-2-yl]pyrrolidin-2-one. A pale yellow oil, R_f 0.18 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.70–2.34 (m, 4 H), 2.66 (s, 3 H), 3.79 (s, 3 H), 4.32 (t, *J* = 8.0 Hz, 1 H), 6.13 (dd, *J* = 3.6, 1.5 Hz, 1 H), 6.29 (dd, *J* = 3.2, 2.9 Hz, 1 H), 6.67 (dd, *J* = 2.7, 2.1 Hz, 1 H), 7.02–7.07 (m, 2 H), 7.23 (dd, *J* = 3.6, 1.7 Hz, 1 H), 7.42 (td, *J* = 7.9, 1.8 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.6, 27.9, 29.7, 55.5, 56.8, 106.6, 108.4, 111.9, 120.9, 123.4, 128.1, 129.3, 130.0, 133.4, 155.0, 174.9; HRMS (ESI) Calcd for $C_{16}H_{18}N_2O_2Na$: M^++Na, 293.1264. Found: *m*/*z* 293.1242.



1-Methyl-5-[1-(2-methoxyphenyl)pyrrol-3-yl]pyrrolidin-2-one. A pale yellow solid, mp 61–63 °C, R_f 0.20 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.99–2.10 (m, 1 H), 2.36–2.63 (m, 3 H), 2.74 (s, 3 H), 3.85 (s, 3 H), 4.49–4.58 (m, 1 H), 6.17 (dd, *J* = 3.0, 1.8 Hz, 1 H), 6.93 (t, *J* = 2.0 Hz, 1 H), 6.98 (t, *J* = 2.6 Hz, 1 H), 7.01–7.04 (m, 2H), 7.25–7.31 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ27.8, 28.0, 30.5, 55.7, 58.1, 107.1, 112.4, 120.2, 121.0, 123.1, 124.2, 125.4, 127.6, 129.8, 152.5, 175.1; HRMS (ESI) Calcd for $C_{16}H_{18}N_2O_2Na$: M^++Na, 293.1264. Found: *m*/*z* 293.1241.

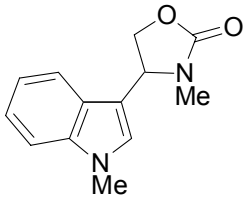


1-Methyl-5-(2,3-dihydrothieno[3,4-*b*]-1,4-dioxin-5-yl)pyrrolidin-2-one. A pale yellow oil, R_f 0.25 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.99–2.14 (m, 1 H), 2.36–2.67 (m, 3 H), 2.71 (s, 3 H), 4.17–4.23 (m, 4 H), 4.79–4.83 (m, 1 H), 6.29 (d, *J* = 0.3 Hz, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ26.9, 27.7, 30.2, 56.1, 64.6, 64.7, 98.1, 116.5, 139.6, 141.5, 174.7; HRMS (ESI) Calcd for $C_{11}H_{13}NO_3SNa$: M^++Na, 262.0513. Found: *m*/*z* 262.0516.



1-(2,3-Dihydrothieno[3,4-*b*]-1,4-dioxin-5-ylmethyl)pyrrolidin-2-one. A pale yellow oil, R_f 0.30 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ1.93–2.04 (m, 2

H), 2.40 (t, J = 8.1 Hz, 2 H), 3.35 (t, J = 7.1 Hz, 2 H), 4.17–4.23 (m, 4 H), 4.47 (s, 2 H), 6.25 (s, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ17.6, 30.8, 37.0, 46.5, 64.5, 64.7, 98.2, 111.8, 139.7, 141.3, 174.9; HRMS (ESI) Calcd for Calcd for $C_{11}H_{13}NO_3SNa$: M^++Na, 262.0513. Found: *m*/*z* 262.0516.



3-Methyl-4-(1-methylindol-3-yl)oxazolidin-2-one. A brown solid, mp 121–123 °C, R_f 0.83 (ethyl acetate). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.71 (s, 3 H), 3.80 (s, 3 H), 4.36 (dd, J = 8.9, 7.7 Hz, 1 H), 4.60 (t, J = 8.9 Hz, 1 H), 4.97 (dd, J = 9.2, 7.7 Hz, 1 H), 7.08 (s, 1 H), 7.15 (ddd, J = 7.9, 6.8, 1.2 Hz, 1 H), 7.29 (ddd, J = 8.0, 6.9, 1.1 Hz, 1 H), 7.36 (dt, J = 8.4, 0.6 Hz, 1 H), 7.60 (dt, J = 8.0, 0.9 Hz, 1 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 28.8, 32.8, 55.4, 68.4, 109.9, 110.3, 119.0, 120.1, 122.6, 125.4, 128.3, 137.8, 158.6; HRMS (ESI) Calcd for $C_{13}H_{14}N_2O_2Na$: M^++Na, 253.0952. Found: *m*/*z* 253.0956.

References

[1] M. Schmeißer, P. Sartori, B. Lippsmeier, *Chem. Ber*. **1970**, *103*, 868–879.

[2] a) A. Klapars, J. C. Antilla, X. Huang, S. L. Buchwald, *J. Am. Chem. Soc.* **2001**, *123*, 7727–7729; b) A. Klapars, X. Huang, S. L. Buchwald, *J. Am. Chem. Soc.* **2002**, *124*, 7421–7428.

[3] H. Heaney, S. V. Ley, *Org. Synth. Coll. Vol. 6* **1988**, 104.

[4] C. Kleber, Z. Andrade, R. C. F. Matos, *Synlett* **2003**, 1189.