Catalytic CH Arylation of SEM-Protected Azoles Using Palladium Complexes of NHCs and Phosphines.

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Supporting Information

1. General Procedures

All reactions were performed under an atmosphere of argon, which was passed through Drierite. Nuclear Magnetic Resonance spectra were recorded at 298 K on Bruker Avance DPX 300 and 400 Fourier Transform NMR. Spectra recorded in CDCl₃ solutions were referenced to TMS; spectra recorded in other deuterated solvents were referenced to residual solvent peaks. IR spectra were taken as thin films on NaCl plates using a Perkin-Elmer 1600 FTIR spectrophotometer. Mass spectra were recorded on a JEOL Lcmate instrument (Ionization mode: APCI+). All HPLC analysis was conducted on a Waters Millennium³² Analytical system with a 996 photodiode array detector; all runs were analyzed at 254 nm, with a Zorbax Rx-C18 Column (4.6 mm x 25 cm). All HPLC methods were conducted using 30% acetonitrile / water (with 0.1 % trifluoroacetic acid) with a linear gradient to 100 % acetonitrile over 20 minutes for 1-alkylindoles and 30 min for 1-SEM-indoles; this was followed by a 5 minute wash of 100% acetonitrile and a 5 minute re-equilibration time; analyte concentrations were determined versus benzoic acid as an internal standard. Flash chromatography was carried out on SILICYCLE silica gel (230-400 mesh). Unless otherwise mentioned, all reaction vessels were flame-dried, and cooled to room temperature under an atmosphere of argon.

2. Materials:

All materials were used as received unless otherwise noted. 1-Methylindole, purchased from Aldrich, was purified via fractional distillation onto 4 Å molecular sieves; the purified material was stored under argon in the dark prior to use. Electron rich SEM-indoles slowly decompose upon standing, and were passed through a short silica plug, and dried under high vacuum prior to use. Their arylation products are stable and can be kept at room temperature for a long period of time.

3. Synthesis of the palladium complexes (The stereochemistry of these compounds still remain to be established)

3.1. Complex 1a

2 (Scheme 1) was prepared using a known protocol,¹ and alkylated using 2-iodopropane according to the following procedure:

2 (0.4 g, 2.5 mmol) was dissolved in acetonitrile (10.0 mL) and treated with 2-iodopropane (1.0 mL, 10.0 mmol). The reaction mixture was refluxed for 40 h, then cooled to room temperature, and the solvent was removed to afford an oily residue, which turned into a white solid upon addition of acetone. 3-pyrrolidinecarbamoyl-1-isopropylimidazolium iodide (**3**) was collected by filtration, washed extensively with ether, and dried under vacuum (0.71 g, 84%).

Scheme 1.

1a was obtained from **3** in two steps via a known literature procedure.¹ **4** is best purified by silica gel chromatography using ethyl acetate/ hexane (6:4) (70% from **3**, orange solid). Palladium complex **1a** is obtained as a yellow solid after silica gel chromatography (69%). The complex can also be recrystallized by slow diffusion of Et₂O into a saturated solution of **1a** in CH₂Cl₂ (92% from **3**). Finally, simple stirring of the reaction mixture in hot EtOAc followed by cooling on ice, and filtration efficiently removes the displaced imidazole impurity, and yields pure **1a**. TLC (70% EtOAc/Hexane): 0.3; ¹H NMR (CDCl₃, 300 MHz): δ 8.21-6.70 (bs, 15H), 7.08 (d, *J* = 2.1 Hz, 1H), 6.70 (d, *J* = 2.1 Hz, 1H), 5.09 (sept, *J* = 6.8 Hz, 1H), 4.02-3.73 (m, 2H), 3.52-3.88 (bs, 1H), 3.32-3.04 (bs, 1H), 2.20-1.80 (bs, 4H), 1.52 (d, *J* = 6.8 Hz, 3H), 0.76 (d, *J* = 6.8 Hz, 3H); Similar to what we have observed with other well characterized compounds of this class,¹ the pyrrolidine and phosphine signals are very broad (bs = broad signal). ¹³C NMR (CDCl₃, 100 MHz): δ 166.7, 150.2, 135.7 (bs), 128.3, 128.2, 121.2, 117.5, 54.2, 50.7, 48.3, 26.7, 25.3, 24.3, 21.9; E.A: calcd for C₂₉H₃₂I₂N₃OPPd: C (42.00), H (3.90), N (5.07); found: C (41.86), H (3.80), N (5.13).

3.2. Complex 1b

This complex was prepared in 6% overall yield using the procedure described below for **1d**. TLC (30% Et₂O/Hexane, UV): 0.4; ¹H NMR (CDCl₃, 400 MHz): δ 7.77-7.67 (m, 5H), 7.38-7.32 (m, 12H), 2.81-2.75 (m, 12H), 2.29-2.21 (m, 6H), 1.80-1.68 (m, 12H); ¹³C NMR (CDCl₃, 100 MHz): δ 135.2, 135.1, 133.7, 133.3, 130.2, 128.1, 128.0, 119.9, 119.8, 61.1, 45.1, 36.5, 30.7; LRMS (FAB⁺): calcd for C₄₁H₄₇I₂N₂PPd: 958.1 [M⁺], found: 831.08 [M-I]⁺, 704.2 [M-2I]⁺; E.A: calcd for C₄₁H₄₇I₂N₂PPd: C (51.34), H (4.94), N (2.92); found: C (50.84), H (4.23), N (3.03). Note: the combustion analysis data for carbon and hydrogen are slightly off despite repeated recrystallization.

3.3. Complex 1c³

This complex was prepared using a literature procedure.³ ¹H NMR (CDCl₃, 300 MHz): δ 6.92 (s, 8H), 6.75 (s, 4H), 2.46 (s, 12H), 1.99 (s, 24H), 1.27 (s, 6H); ¹H NMR (C₆D₆, 300 MHz): δ 6.90 (s, 8H), 6.04 (s, 4H), 2.33 (s, 12H), 2.17 (s, 24H), 1.57 (s, 6H); ¹³C NMR (CDCl₃, 100 MHz): δ 175.0, 170.1, 137.7, 136.3, 129.1, 122.5, 23.4, 21.9, 19.1; ¹³C NMR: (C₆D₆, 100 MHz) δ 174.4, 171.2, 137.3, 136.8, 136.4, 129.3, 122.1, 23.3, 21.3, 19.1; LRMS (FAB⁺): found: 773.0 [(IMes)₂PdOAc]⁺, 410.0 [(IMes)Pd]⁺.

3.4. Complex 1d: (prepared using a modified literature procedure).²

A suspension of 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (0.25 g, 0.73 mmol), potassium tert-butoxide 95% (0.09 g, 0.80 mmol), sodium iodide (0.44 g, 2.90 mmol), and palladium acetate (0.16 g, 0.73 mmol) in THF (30.0 mL) was stirred at room temperature for 18 h in the absence of any light. The solvent was evaporated at room temperature under reduced pressure, and the residue was stirred in ethyl acetate:hexane (6:4) and filtered through a short silica plug. The silica gel was washed with ether, and the solvent was evaporated to afford a red solid, which was further purified by flash chromatography using a mixture of ether and hexane (4:6). This product was dissolved in CH_2Cl_2 (5.0 mL), and treated with triphenylphosphine (42 mg, 0.16 mmol). The resulting mixture was stirred at room temperature for 2 h, followed by silica gel chromatography using 30 % ether/hexane to afford the product as an orange solid (37 mg, 6%). TLC (30% Et_2O /Hexane, UV): 0.5; ^1H NMR (CDCl_3 , 400 MHz): δ 7.38-7.18 (m, 15H), 7.07 (d, J = 1.4 Hz, 2H), 7.02 (s, 4H), 2.43 (s, 6H), 2.40 (s, 12H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 166.2, 164.3, 138.9, 136.6, 136.4, 135.8, 135.7, 134.7, 134.2, 129.9, 129.6, 127.5, 127.4, 124.7, 124.65, 22.7, 21.9; LRMS (FAB $^+$): calcd for $\text{C}_{39}\text{H}_{39}\text{I}_2\text{N}_2\text{PPd}$: 926.0 [M^+], found: 799.0 [$\text{M}-\text{I}$] $^+$, 672.1 [$\text{M}-2\text{I}$] $^+$; E.A: calcd for $\text{C}_{39}\text{H}_{39}\text{I}_2\text{N}_2\text{PPd}$: C (50.53), H (4.24), N (3.02); found: C (50.73), H (4.44), N (3.01).

4. Direct comparison of the catalysts (Figure 2).

In an 8 mL vial, sealed with an open top closure, CsOAc (0.1 g, 0.5 mmol) was dried under vacuum at 120 °C over night, and cooled to RT. SEM-indole (1.6 g, 6.5 mmol), PhI (0.9 mL, 7.8 mmol), and dimethyl acetamide (DMA) (2.6 mL) were mixed in a flame-dried vial. The vials containing dry CsOAc were charged with 0.2 mL of this solution. A solution of the appropriate catalyst in DMA (0.1 mL) was added to each vial, and the resulting reaction mixtures were heated to 125 °C. At the time point indicated on Fig. 2, the vials were cooled to 0 °C then diluted with methanol (20 mL). This solution was then analyzed by HPLC using benzoic acid as internal standard.

Typical procedure for the protection of the azoles

2-cyano pyrrole (0.5 g, 5.4 mmol) was dissolved in DMF (10.0 mL) in a flame dried flask under argon, and cooled to 0 °C. NaH (240.0 mg, 6.0 mmol, 60 % dispersion in mineral oil) was added over 5 min. After 45 min, SEMCl (5.4 mmol) was added slowly, and the reaction was slowly warmed up to room temperature and stirred for 5 h. The reaction was then quenched by slow addition of water, and diluted with ether (50 mL), water (25 mL). The two phases were separated and, the aqueous phase was extracted with ether (2 X 25 mL). The combined organic phase was washed with water (3 X 25 mL), brine (30 mL), and dried over magnesium sulfate. The solvent was evaporated under reduced pressure and the residue was purified by silica gel chromatography using ethyl acetate/hexane (5:1) to afford the product as a colorless oil in 91%.

Arylation of SEM-indole using $\text{Pd}(\text{OAc})_2$

Cesium acetate (0.1g, 0.5 mmol) was dried as described above. To each vial was added SEM-indole (62 mg, 0.25 mmol), PhI (56.0 μL , 0.5 mmol). $\text{Pd}(\text{OAc})_2$ (4.8 mg), and PPh_3 (22.0 mg) were dissolved in DMA (0.75 mL), and the appropriate amount was added to each vial. The final volume of DMA was adjusted to 0.5 mL in each vial. The reaction mixtures were heated to 125 °C for 20 h.

The yields were established by ^1H NMR analysis of the crude reaction mixture using *t*-BuOH as internal standard in the case of indole, and GC for biphenyl using dodecane as internal standard.

Table 1

Cat (mol%)	% conversion (indole)	Yield (Ph-Ph) (%) ^a
0.5	55	12
1.0	63	18
2.0	69	27
4.0	71	56

^aYield based on PhI.

Typical Arylation procedure

In an 8 mL vial, sealed with an open top closure, CsOAc (0.2g, 1.0 mmol) was dried under vacuum at 120 °C for 2 h. The vials were then cooled to room temperature under argon, and charged with the appropriate amount of the catalyst. The substrate (0.5 mmol), DMA (volume indicated for each substrate), PhI were sequentially added, and the resulting reaction mixture was heated to 125 °C for 24 h, and then cooled to RT. The reaction mixture was diluted with ether (25 mL), washed with water (3 × 10 mL), brine (10 mL), and dried over MgSO₄. The solvent was evaporated and the residue was purified by silica gel chromatography to afford the desired product.

Note: In all cases Ar-Ar byproducts are observed.

2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole

See general procedure: DMA (0.6 mL), PhI (1.5 equiv), substrate (2.0 mmol), CsOAc (2.0 mmol). TLC (9 % Et₂O/Hexane, UV): 0.51; ^1H NMR (CDCl₃, 300 MHz): δ 7.66 (m, 3H), 7.56-7.31 (m, 4H), 7.23 (dd, J = 6.6, 6.7 Hz, 1H), 7.20 (dd, J = 6.7, 6.9 Hz, 1H), 6.64 (s, 1H), 5.51 (s, 2H), 3.54 (t, J = 8.0 Hz, 2H), 0.94 (t, J = 8.0 Hz, 2H), -0.1 (s, 9H); ^{13}C NMR (CDCl₃, 100 MHz): δ 142.0, 138.6, 132.9, 129.9, 128.9, 128.7, 128.4, 122.6, 121.0, 120.9, 110.7, 103.6, 73.4, 66.3, 18.6, 0.0; IR (CDCl₃, film): ν_{max} 3082, 3060, 3025, 2924, 2849, 1942, 1871, 1802, 1745, 1601, 1493, 1451, 1372, 1028 cm⁻¹; LRMS (APCI): calcd for C₂₀H₂₅NOSi: 323.17, found: 324.11 [M + H]⁺.

2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole-5-carbonitrile

See general procedure: DMA (0.3 mL), PhI (1.4 equiv), substrate (0.5 mmol). TLC (10 % EtOAc/Hexane, UV): 0.30; ^1H NMR (CDCl₃, 300 MHz): δ 8.0 (d, J = 1.1 Hz, 1H), 7.69-7.51 (m, 7H), 6.68 (s, 1H), 5.51 (s, 2H), 3.53 (t, J = 8.1, 2H), 0.90 (t, J = 8.1 Hz, 2H), -0.2 (s, 9H); ^{13}C NMR (CDCl₃, 100 MHz): δ 144.3, 140.0, 131.5, 129.9, 129.3, 129.1, 126.2, 125.4, 121.0, 111.8, 111.7, 104.2, 103.8, 73.5, 66.7, 18.5, 0.0; LRMS (APCI): calcd for C₂₁H₂₄N₂O₂Si: 348.13, found: 349.10 [M + H]⁺. The product is contaminated by a small amount of inseparable impurity.

5-Nitro-2-phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole

See general procedure: DMA (0.6 mL), PhI (1.4 equiv), substrate (1 mmol). TLC (20 % EtOAc/Hexane, UV): 0.41; ^1H NMR (CDCl_3 , 300 MHz): δ 8.60 (d, $J = 2.2$ Hz, 1H), 8.19 (dd, $J = 2.2, 9.0$ Hz, 1H), 7.67-7.53 (m, 6H), 6.77 (s, 1H), 5.50 (s, 2H), 3.5 (t, $J = 8.1$ Hz, 2H), 0.93 (t, $J = 8.1$ Hz, 2H), -0.1 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 150.0, 143.3, 142.4, 132.5, 131.7, 129.7, 129.0, 128.8, 128.4, 120.9, 117.3, 101.2, 71.2, 66.9, 18.7, 0.0; IR (CDCl_3 , film): ν_{max} 2952, 2901, 1611, 1514, 1468, 1394, 1336, 1249, 1071 cm^{-1} ; LRMS (APCI): calcd for $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_3\text{Si}$: 368.16, found: 369.10 $[\text{M} + \text{H}]^+$.

3-*o*-Tolyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole

See general procedure: DMA (0.5 mL), 2-iodotoluene (2.0 equiv), substrate (0.5 mmol). TLC (3 % EtOAc/Hexane, UV): 0.21; ^1H NMR (CDCl_3 , 300 MHz): δ 7.58 (d, $J = 7.2$ Hz, 1H), 7.54 (d, $J = 7.1$ Hz, 1H), 7.47-7.42 (m, 1H), 7.38-7.28 (m, 4H), 7.22-7.18 (m, 2H), 5.53 (s, 2H), 3.55 (t, $J = 8.0$ Hz, 2H), 2.35 (s, 3H), 0.93 (t, $J = 8.0$ Hz, 2H), -0.3 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 137.1, 136.7, 134.4, 131.2, 130.7, 128.9, 127.2, 126.9, 126.0, 122.6, 120.7, 120.6, 117.5, 110.5, 76.2, 66.4, 21.4, 18.5, -0.1; IR (CDCl_3 , film): ν_{max} 3058, 2952, 2894, 1460, 1334, 1309, 1249, 1077; LRMS (APCI) calcd for $\text{C}_{21}\text{H}_{27}\text{NOSi}$: 337.19, found 338.28.

2-*o*-Tolyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole

TLC (3 % EtOAc/Hexane, UV): 0.25; ^1H NMR (CDCl_3 , 300 MHz): δ 7.64 (d, $J = 7.6$ Hz, 1H), 7.54 (d, $J = 7.2$ Hz, 1H), 7.38-7.18 (m, 6H), 6.45 (s, 1H), 5.28 (s, 2H), 3.24 (t, $J = 8.1$ Hz, 2H), 2.20 (s, 3H), 0.74 (t, $J = 8.1$ Hz, 2H), -0.12 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 140.5, 138.5, 137.5, 132.5, 131.9, 130.6, 129.2, 128.9, 125.9, 122.3, 120.9, 120.8, 111.0, 103.7, 73.2, 65.9, 20.6, 18.2, -0.1; IR (CDCl_3 , film): ν_{max} 3058, 2952, 2894, 1460, 1334, 1309, 1249, 1077; LRMS (APCI) calcd for $\text{C}_{21}\text{H}_{27}\text{NOSi}$: 337.19, found 338.28. (Note: the reaction mixture is best purified using 4% Et_2O in hexane)

2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole-3-carboxylic acid methyl ester

See general procedure: DMA (0.25 mL), PhI (2.0 equiv), substrate (0.25 mmol). ^1H NMR (CDCl_3 , 300 MHz): δ 8.26-8.18 (m, 1H), 7.59-7.40 (m, 6H), 7.38-7.28 (m, 2H), 5.32 (s, 2H), 3.76 (s, 3H), 3.32 (t, $J = 8.1$ Hz, 2H), 0.76 (t, $J = 8.1$ Hz, 2H), -0.08 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 166.0, 147.2, 136.9, 131.4, 131.1, 129.5, 128.4, 127.1, 123.7, 123.0, 122.4, 111.2, 106.7, 73.3, 66.4, 51.2, 18.1, -1; LRMS calcd for $\text{C}_{22}\text{H}_{27}\text{NO}_3\text{Si}$: 381.1, found 382.12 $[\text{M} + \text{H}]^+$.

3-Methyl-2-phenyl-1*H*-indole (commercially available)

See general procedure: DMA (0.3 mL), PhI (2.0 equiv), substrate (0.5 mmol). The product was contaminated with inseparable unreacted starting material (15-25%), thus it was dissolved in THF (5.0 mL), and TBAF (5.0 equiv) was added. After refluxing for 10 h, the solvent was removed, and the residue was purified by silica gel chromatography. TLC (7.5 % EtOAc/Hexane, UV): 0.48; ^1H NMR (CDCl_3 , 300 MHz): δ 8.0

(bs, 1H), 7.54-7.46 (m, 3H), 7.40 (dd, $J = 7.3, 7.3$ Hz, 2H), 7.30-7.24 (m, 2H), 7.12 (ddd, $J = 1.4, 7.0, 8.1$, 1H), 7.07 (ddd, $J = 1.4, 7.0, 8.1$, 1H), 2.41 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 136.0, 134.2, 133.8, 130.4, 129.1, 128.1, 127.6, 122.7, 119.9, 119.3, 111.0, 109.0, 10.2; LRMS (APCI) calcd for $\text{C}_{15}\text{H}_{13}\text{N}$: 207.10, found: 208.16 $[\text{M} + \text{H}]^+$.

5-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrole-2-carbonitrile

See general procedure: DMA (0.25 mL), PhI (2.0 equiv), substrate (0.5 mmol). TLC (20% EtOAc/Hexane, UV): 0.64; ^1H NMR (CDCl_3 , 300 MHz): δ 7.50 (dd, $J = 2.7, 7.2$ Hz, 2H), 7.33-7.39 (m, 3H), 6.85 (d, $J = 3.9$ Hz, 1H), 6.25 (d, $J = 3.9$ Hz, 1H), 3.61 (t, $J = 8.1$ Hz, 2H), 0.90 (t, $J = 8.1$ Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 141.2, 131.3, 129.5, 129.1, 129.0, 121.1, 114.5, 110.8, 106.0, 75.0, 67.3, 18.6, 0.0; IR (CDCl_3 , film): ν_{max} 3064, 2953, 2896, 2215, 1604, 1460, 1398, 1311, 1249 cm^{-1} ; LRMS (APCI): calcd for $\text{C}_{17}\text{H}_{22}\text{N}_2\text{OSi}$ (298.15), found: 299.22 $[\text{M} + \text{H}]^+$. The product is contaminated by a small amount of inseparable impurity (4-6%).

5-Pyridin-3-yl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrole-2-carbonitrile

See general procedure: DMA (0.2 mL), 3-iodopyridine (1.2 equiv), substrate (0.5 mmol). TLC (20% EtOAc/Hexane, UV): 0.4; ^1H NMR (CDCl_3 , 300 MHz): δ 8.78-8.76 (m, 1H), 8.62 (dd, $J = 1.5, 5.0$ Hz, 1H), 7.95 (dm, $J = 7.9$ Hz, 1H), 7.36 (dd, $J = 5.0, 8.0$ Hz, 1H), 6.90 (d, $J = 4.0$ Hz, 1H), 6.38 (d, $J = 4.0$ Hz, 1H), 5.34 (s, 2H), 3.71 (t, $J = 8.1$ Hz, 2H), 0.99 (t, $J = 8.1$ Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 150.6, 150.4, 137.4, 136.5, 127.4, 123.7, 121.0, 114.0, 111.7, 107.2, 75.0, 67.5, 18.6, -0.2; IR (ν , cm^{-1}) 2953, 2896, 2218, 1570, 1460, 1428, 1313, 1249, 1085 cm^{-1} ; LRMS (APCI): calcd for $\text{C}_{16}\text{H}_{21}\text{N}_3\text{OSi}$: 299.44 $[\text{M}]^+$, found: 300.61 $[\text{M} + \text{H}]^+$.

Arylation of [1-(trimethylsilyl)-ethoxymethyl]-pyrrol-3-yl]-ethanone:

This reaction afforded a mixture of three products, which were assigned structures **5**, **6**, and **7** based on the following NMR data.

5: $J = 3.1$ Hz suggested a vicinal relationship between the pyrrole protons.

6: $J = 1.9$ Hz is a typical coupling constant for 1,3-protons on pyrroles ring. To discriminate between the two possibilities (Ph at 4 or 5 positions), nOe studies were conducted. In addition, as

the C2 proton in **6** was buried under aromatic (phenyl) protons, the product was deprotected and the nOe results were confirmed.

These nOe data are in agreement with the Ph group being at the 5 position.

Our ^1H -NMR spectrum for **8** did not match the literature data. This discrepancy is attributed to a mistake in the latter paper, wherein the ^1H NMR spectra for 5-phenyl-3-acetyl pyrrole and 2-phenyl-3-acetyl pyrrole have been inadvertently switched, as confirmed by personal communication with the authors.⁴

7: Based on the above data, it is our assumption that the bis-arylation product results from reaction at the 2 and 5 positions.

The full characterization data are presented below:

1-{5-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrol-3-yl}-ethanone

See general procedure: DMA (0.3 mL), PhI (1.4 equiv), substrate (0.5 mmol). TLC (20 % EtOAc/Hexane, UV): 0.20; ^1H NMR (CDCl_3 , 300 MHz): δ 7.51-7.31 (m, 6H), 6.66 (d, $J = 1.9$ Hz, 1H), 5.25 (s, 2H), 3.50 (t, $J = 8.1$ Hz, 2H), 2.44 (s, 3H), 0.90 (t, $J = 8.1$ Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 192.9, 138.7, 132.0, 129.3, 128.9, 128.2, 127.9, 126.2, 109.7, 77.03, 66.8, 27.8, 18.5, 0.0; IR (CDCl_3 , film): ν_{max} 3116, 3072, 2953, 2895, 1612, 1604, 1559, 1519, 1403, 1364, 1249, 1177, 1086 cm^{-1} ; LRMS (APCI): calcd for $\text{C}_{18}\text{H}_{25}\text{NO}_2\text{Si}$: 315.17, found: 316.22 $[\text{M} + \text{H}]^+$.

1-{2,5-Diphenyl-1-[2-(trimethyl-silyl)-ethoxymethyl]-pyrrol-3-yl}-ethanone

See general procedure: DMA (0.3 mL), PhI (1.4 equiv), substrate (0.5 mmol). TLC (20 % EtOAc/Hexane, UV): 0.4; ^1H NMR (CDCl_3 , 300 MHz): δ 7.63-7.59 (m, 2H), 7.56-7.35 (m, 8H), 6.80 (s, 1H), 4.93 (s, 2H), 3.29 (t, $J = 8.1$ Hz, 2H), 2.13 (s, 3H), 0.84 (t, $J = 8.1$ Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 195.0, 139.8, 135.9, 132.5, 132.3, 131.6, 129.4, 128.9, 128.6, 128.1, 124.6, 110.3, 73.8, 66.0, 29.7, 18.6, 0.0; IR (CDCl_3 , film): ν_{max} 2953, 2895, 1724, 1606, 1494, 1355, 1249, 1211, 1087; LRMS (APCI): calcd for $\text{C}_{24}\text{H}_{29}\text{NO}_2\text{Si}$: 391.20, found 392.26 $[\text{M} + \text{H}]^+$.

1-[2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrol-3-yl]-ethanone

See general procedure: DMA (0.3 mL), PhI (1.4 equiv), substrate (0.5 mmol). TLC (20 % EtOAc/Hexane, UV): 0.25; ^1H NMR (CDCl_3 , 300 MHz): δ 7.51-7.42 (m, 3H), 7.41-7.37 (m, 2H), 6.84 (d, J = 3.1 Hz, 1H), 6.75 (d, J = 3.1 Hz, 1H), 5.0 (s, 2H), 3.39 (t, J = 8.1 Hz, 2H), 2.12 (s, 3H), 0.86 (t, J = 8.1 Hz, 2H), 0.0 (s, 9H); LRMS (APCI) calcd for $\text{C}_{18}\text{H}_{25}\text{NO}_2\text{Si}$: 315.17, found 316.22 (Minor product of the reaction mixture).

2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-7-azaindole

See general procedure: DMA (0.6 mL), PhI (1.4 equiv), substrate (1 mmol). TLC (20% EtOAc/Hexane, UV): 0.67; ^1H NMR (CDCl_3 , 300 MHz): δ 8.39 (dd, J = 1.5, 4.8 Hz, 1H), 7.93 (dd, J = 1.4, 7.7 Hz, 1H), 7.81 (d, J = 6.8 Hz, 2H), 7.53-7.48 (m, 3H), 7.13 (dd, J = 4.7, 7.8 Hz, 1H), 6.61 (s, 1H), 5.52 (s, 2H), 3.74 (t, J = 8.2 Hz, 2H), 1.01 (t, J = 8.2 Hz, 2H), -0.3 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 150.0, 143.3, 142.4, 132.5, 129.7, 129.0, 128.8, 128.4, 120.9, 117.3, 101.2, 71.2, 66.9, 18.7, 0.0; IR (CDCl_3 , film): ν_{max} 3061, 2951, 2894, 1572, 1477, 1426, 1249, 1073 cm^{-1} . LRMS (APCI): calcd for $\text{C}_{19}\text{H}_{24}\text{N}_2\text{OSi}$: 324.49, found: 325.24 $[\text{M} + \text{H}]^+$. The product is contaminated by a small amount of inseparable impurity (See ^1H -NMR spectrum).

5-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-imidazole.

See general procedure: DMA (0.5 mL), PhI (2.0 equiv), substrate (0.5 mmol). ^1H NMR (CDCl_3 , 300 MHz): δ 7.71 (s, 1H), 7.60 (d, J = 6.9 Hz, 2H); 7.48-7.39 (m, 3H), 7.19 (s, 1H), 5.42 (s, 2H), 3.57 (t, J = 8.2 Hz, 2H), 0.99 (t, J = 8.2 Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 139.4, 133.9, 129.8, 129.1, 129.0, 128.8, 128.3, 74.5, 66.8, 18.4, 0.0; IR (CDCl_3 , film): ν_{max} 3063, 2952, 2894, 1606, 1486, 1484, 1362, 1249, 1075 cm^{-1} ; LRMS (APCI): calcd for $\text{C}_{15}\text{H}_{22}\text{N}_2\text{OSi}$: 274.15, found (LR-APCI) 275.10 $[\text{M} + \text{H}]^+$.

2,5-Diphenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-imidazole.

See general procedure: DMA (1.0 mL), PhI (2.0 equiv), substrate (0.5 mmol), Ag_2CO_3 (2.0 equiv). TLC (45 % EtOAc/Hexane, UV): 0.55; ^1H NMR (CDCl_3 , 300 MHz): δ 7.80 (d, J = 6.9 Hz, 2H), 7.59 (d, J = 7.0 Hz, 2H), 7.49-7.36 (m, 6H), 7.18 (s, 1H), 5.42 (s, 2H), 3.45 (t, J = 8.1 Hz, 2H), 0.93 (t, J = 8.1 Hz, 2H), 0.0 (s, 9H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 150.5, 135.7, 131.0, 130.4, 129.3, 129.27, 129.16, 129.1, 128.9, 128.4, 127.9, 73.8, 66.0, 18.66, 0.0; IR (CDCl_3 , film): ν_{max} 3062, 2952, 2894, 1606, 1486, 1362, 1249, 1074 cm^{-1} ; LRMS calcd for $\text{C}_{21}\text{H}_{26}\text{N}_2\text{OSi}$: 350.18, found: 351.26 $[\text{M} + \text{H}]^+$.

3-Phenyl-imidazo[1,2-a]pyridine

See general procedure: DMA (0.2 mL), PhI or PhBr (1.2 equiv), substrate (0.35 mmol). TLC (EtOAc, UV): 0.38; ^1H NMR (CDCl_3 , 300 MHz): δ 8.31 (d, J = 6.8 Hz, 1H), 7.69 (s, 1H), 7.68 (d, J = 7.2 Hz, 1H), 7.55-7.39 (m, 5H), 7.17 (dd, J = 6.8, 7.3 Hz, 1H), 6.78 (dd, J = 6.8, 7.0 Hz, 1H); ^{13}C NMR (CDCl_3 , 100 MHz): δ 146.4, 132.8, 129.6, 128.5, 128.4, 126.1, 124.6, 123.7, 118.6, 113.0; IR

(CDCl₃, film): $\nu_{\max}$ 3056, 2982, 1636, 1604, 1500, 1482, 1354, 1298 cm⁻¹; LRMS (APCI): calcd for C₁₃H₁₀N₂: 194.08, found: 195.04 [M + H]⁺.

3-Phenyl-imidazo[1,2-a]pyridine-2-carboxylic acid ethyl ester

See general procedure: DMA (0.6 mL), PhI (1.2 equiv), substrate (0.5 mmol). TLC (EtOAc, UV): 0.49; ¹H NMR (CDCl₃, 300 MHz): δ 7.94 (d, J = 5.2 Hz, 1H), 7.73 (d, J = 6.9 Hz, 1H), 7.29-7.22 (m, 5H), 7.25 (dd, J = 5.2, 7.0 Hz, 1H), 6.80 (dd, J = 6.9, 7.0 Hz, 1H), 4.35 (q, J = 5.3 Hz, 2H), 1.30 (t, J = 5.3 Hz, 3H);

¹³C NMR (CDCl₃, 100 MHz): δ 163.8, 144.6, 133.3, 130.4, 129.7, 129.1, 128.4, 126.4, 124.4, 119.4, 114.0, 61.4, 14.9; IR (CDCl₃, film): $\nu_{\max}$ 3056, 2982, 1720, 1637, 1508, 1446, 1279, 1234, 1183 cm⁻¹; LRMS (APCI): calcd for C₁₆H₁₆N₂O₂: 266.11, found: 267.05 [M + H]⁺.

3-(4-Methoxycarbonylphenyl)-imidazo[1,2-a]pyridine-2-carboxylic acid ethyl ester

See general procedure: DMA (0.4 mL), 4-BrPhCO₂Me (1.2 equiv), substrate (0.5 mmol). TLC (60 % EtOAc/Hexane, UV): 0.15; ¹H NMR (CDCl₃, 300 MHz): δ 8.23 (d, J = 6.6 Hz, 2H), 7.92 (dd, J = 1.1, 7.0 Hz, 1H), 7.75 (dd, J = 1.1, 7.0 Hz, 1H), 7.57 (d, J = 6.6 Hz, 2H), 7.27 (m, 1H), 6.84 (dd, J = 6.9, 7.0 Hz, 1H), 4.38 (q, J = 7.1 Hz, 2H), 3.97 (s, 3H), 1.33 (t, J = 7.1 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz): δ 166.8, 163.0, 144.9, 133.8, 133.0, 131.2, 131.0, 130.3, 128.4, 126.8, 124.1, 119.5, 114.4, 61.6, 52.9, 15.0; LRMS (APCI): calcd

for C₁₈H₁₆N₂O₄: 324.11, found: 325.21 [M+H]⁺.

References

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3. Konnick, M. M.; Guzei, I. A.; Stahl, S. S. *J. Am. Chem. Soc.* **2004**, *126*, 10212-10213
4. Katritzky, A. R.; Zhu, L.; Lang, H.; Denisko, O.; Wang, Z. *Tetrahedron* **1995**, *51*, 13271-13275.

Compound **1a**, Fig.1

The pyrrolidine and phosphine signals are very broad. The stereochemistry of the complex still remains to be established.

Compound **1b**, Fig. 1

The stereochemistry of **1b** has not yet been confirmed

proton 400MHz
ol-19gl-1b-c

Current Data Parameters
NAME BT-II-129C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090818
Time 23:27
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 32768
ID
SOLVENT Acetone
NS 6217
DS 4
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.742452 sec
RG 16384
DW 22.650 usec
DE 36.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL F1
NUC1 13C
P1 17.50 usec
PL1 0.00 dB
SFO1 100.569800 MHz

CHANNEL F2
CPDPRG2 waltz16
NUC2 1H
PCPD2 96.25 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 399.921599 MHz

F2 - Processing parameters
SI 65536
SF 100.5599210 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Compound **1c**, Fig 1 (Proton and Carbon NMR spectra in C₆D₆)

Compound **1c**, Fig 1 (Proton and Carbon spectra in CDCl₃)

Compound **1d**, Fig 1

Current Data Parameters
NAME BT-II-11H-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050809
Time 8.14
INSTRUM spect
PROBHD 5 mm Mh1-zrpg30
PULPROG zgpg30
TO 32768
SOLVENT Aceton
NS 10229
DS 0
SWH 22075.0153 Hz
FIDRES 0.673677 Hz
AQ 0.7422452 sec
RG 16384
DW 22.650 usec
DE 6.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 17.50 usec
PL1 -1.00 dB
SFO1 100.569800 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 96.25 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 399.9215997 MHz

F2 - Processing parameters
SI 65536
SF 100.559240 MHz
WDW EM
SSB 0 Hz
LB 3.00 Hz
GB 0
PC 1.40

Table 1, entry 2. **2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole**

proton 400Mhz
ol-tabl-1c

Current Data Parameters
NAME BF-II-16-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080726
Time 8.41
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 32768
FIDRES 0.742452 sec
RG 16384
DM 22.650 usec
DE 3.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL F1 13C
NUC1 13C
P1 17.50 usec
PL1 0.00 dB
SFO1 100.5699800 MHz

CHANNEL F2 1H
CPDPRG2 waltz16
NUC2 1H
P2 96.25 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 399.9215997 MHz

F2 - Processing parameters
SI 65536
SF 100.5599210 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Table 1, Entry 3. **5-Cyano-2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole**

Current Data Parameters
NAME: BT-II-24-1C13
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20090716
Time: 17.39
INSTRUM: spect
PROBHD: 5 mm Bb1Hnucl
PULPROG: zgpg30
TD: 32768
SOLVENT: Residual
NS: 500
DS: 0
SWH: 22075.055 Hz
FIDRES: 0.673677 Hz
AQ: 0.7422452 sec
RG: 16384
DM: 22.650 usec
DE: 3800.0 Hz
TE: 300.2 K
D1: 0.2500000 sec
d11: 0.0300000 sec
d12: 0.0002000 sec

CHANNEL F1
NUC1: 13C
P1: 17.50 usec
PL1: 1.00 dB
PC1: 100.569900 MHz
SFO1: 100.569900 MHz

CHANNEL F2
NAME: waitc16
NUC2: 1H
P2: 96.25 usec
PL2: 0.00 dB
PCP2: 23.00 dB
PL12: 23.00 dB
PL13: 23.00 dB
SFO2: 399.921597 MHz

F2 - Processing parameters
SI: 65536
SF: 100.559240 MHz
WDW: EM
SSB: 0
LB: 3.00 Hz
GB: 0
PC: 1.40

Table 1, entry 4. **5-Nitro-2-phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole**

Table 1, entry 5 (2-*o*-Tolyl-1-[2-(trimethyl-silyl)-ethoxymethyl]-indole)

Table 1, entry 5 (3-*o*-Tolyl-1-[2-(trimethyl-silyl)-ethoxymethyl]-indole)

Current Data Parameters
NAME BT-11-11-2013
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080923
Time 13.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
FIDRES 0.673677 Hz
AQ 0.742452 sec
RG 16384
DM 22.650 usec
DE 3.00 Hz
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 17.00 usec
PL1 -1.00 dB
SFO1 100.5699800 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 96.20 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 399.9215997 MHz

F2 - Processing parameters
SF 100.5599240 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Table 1, entry 6. **2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-indole-3-carboxylic acid methyl ester**

Table 1, entry 8. **5-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrole-2-carbonitrile.**

Table 1, entry 9. **5-Pyridin-3-yl-1-[2-(trimethylsilyl)-ethoxymethyl]-pyrrole-2-carbonitrile**

..... 22-23, 25

11

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Date_      2000/10
Time      17.55

```

INSTIKUM spect
PROBHD 5 mm Multinuel

SOLVENT	Aceton
NO	2.579

FIDRES	0.673677 Hz
AO	0.7422452 sec

DW	27.630 use
DE	6.00 use

d12	0.00002000 sec
-----	----------------

P1 17.50 use

===== CHANNEL F2 =====

PCPDZ	96.25 use
PT2	0.00 dB

F2 - Processing parameters

WUM	SSB	0
0	0	0

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Table 1, Entry 10. **1-(2,5-Diphenyl-1-[2-(trimethyl-silyl)-ethoxymethyl]-pyrrol-3-yl)-ethanone**

proton 400Mhz
ol-tabl-9ac

Current Data Parameters
NAME BT-II-24-3aCl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080805
Time 9.33
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 32768
FIDRES 0.3213
SOLVENT CDCl3
DS 2341
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.742452 sec
RG 16384
DM 22.650 usec
DE 6.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL F1 1JC
P1 17.50 usec
PL1 0.00 dB
SFO1 100.5699800 MHz

CHANNEL F2 1H
CPDPRG2 waltz16
NUC2 1H
PCPD2 96.25 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 399.9215997 MHz

F2 - Processing parameters
SI 65536
SF 100.5599210 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Table 1, entry 10. **1-{2-phenyl-1-[2-(trimethyl-silyl)-ethoxymethyl]-pyrrol-3-yl}-ethanone**

Current Data Parameters
NAME BT-II-24-3DC13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080805
Time 8.43
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TOPOPROG 32768
SOLVENT Acetone
NS 2042
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.742452 sec
RG 16384
DM 22.650 usec
DE 6.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL F1 13C
NUC1 13C
P1 17.50 usec
PL1 -1.00 dB
SFO1 100.569800 MHz

CHANNEL F2 1H
NUC2 1H
P2 96.25 usec
PL2 0.00 dB
P12 23.00 dB
P13 23.00 dB
SFO2 399.9610997 MHz

F2 - Processing parameters
SI 65536
SF 100.5599240 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Table 2, entry 1. **2-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-7-azaindole**

```

***** 150.10
***** 141.79
***** 142.09
*****
*****
*****
***** 132.47
***** 131.70
***** 110.08
***** 103.71
***** 102.66
***** 128.76
***** 128.44
*****
***** 120.53
*****
***** 117.26
*****

```

```

***** 201.27

*****

***** 77.52
***** 77.50
***** 77.28
***** 71.29

***** 66.56

```

..... 20 75

***** 2, 53

```
Current Data Parameters
NAME          BT-II-21-2E
EXPNO         1
PROCNO        1
```

F2 - Acquisition Parameters
Date 20050613

```
Time 14.45
INSTRUM spect
```

PROBHD 5 mm Multinuc1
PULPROG zqpy30

ID	32168
SOLVENT	Aceton

DS 0 22075 055 Hz

FIDRES	0.673677 Hz
AQ	0.7422452 sec

RG	16384
DW	22.650 use

```
DE      6.00 use
TE      300.0 K
```

d1	0.20000000	sec
d11	0.03000000	sec
d13	0.00000000	sec

```
===== CHANNEL F1 =====
```

NUC1	13C
P1	17.50 use

PL1	-1.00 dB
SFO1	100.5699800 MHz

----- CHANNEL F2 -----

CLUSTER	WAVELENGTH
NUC2	1H
PCPD?	0.6 25

PL2	0.00 dB
PL12	23.00 dB

PL13	23.00	dB
SFO2	399.9215997	MHz

F2 - Processing parameters

Channel	Frequency (MHz)
CH 1	100.5599240
CH 2	100.5599240
CH 3	100.5599240
CH 4	100.5599240
CH 5	100.5599240
CH 6	100.5599240
CH 7	100.5599240
CH 8	100.5599240
CH 9	100.5599240
CH 10	100.5599240
CH 11	100.5599240
CH 12	100.5599240
CH 13	100.5599240
CH 14	100.5599240
CH 15	100.5599240
CH 16	100.5599240
CH 17	100.5599240
CH 18	100.5599240
CH 19	100.5599240
CH 20	100.5599240
CH 21	100.5599240
CH 22	100.5599240
CH 23	100.5599240
CH 24	100.5599240
CH 25	100.5599240
CH 26	100.5599240
CH 27	100.5599240
CH 28	100.5599240
CH 29	100.5599240
CH 30	100.5599240
CH 31	100.5599240
CH 32	100.5599240
CH 33	100.5599240
CH 34	100.5599240
CH 35	100.5599240
CH 36	100.5599240
CH 37	100.5599240
CH 38	100.5599240
CH 39	100.5599240
CH 40	100.5599240
CH 41	100.5599240
CH 42	100.5599240
CH 43	100.5599240
CH 44	100.5599240
CH 45	100.5599240
CH 46	100.5599240
CH 47	100.5599240
CH 48	100.5599240
CH 49	100.5599240
CH 50	100.5599240
CH 51	100.5599240
CH 52	100.5599240
CH 53	100.5599240
CH 54	100.5599240
CH 55	100.5599240
CH 56	100.5599240
CH 57	100.5599240
CH 58	100.5599240
CH 59	100.5599240
CH 60	100.5599240
CH 61	100.5599240
CH 62	100.5599240
CH 63	100.5599240
CH 64	100.5599240
CH 65	100.5599240
CH 66	100.5599240
CH 67	100.5599240
CH 68	100.5599240
CH 69	100.5599240
CH 70	100.5599240
CH 71	100.5599240
CH 72	100.5599240
CH 73	100.5599240
CH 74	100.5599240
CH 75	100.5599240
CH 76	100.5599240
CH 77	100.5599240
CH 78	100.5599240
CH 79	100.5599240
CH 80	100.5599240
CH 81	100.5599240
CH 82	100.5599240
CH 83	100.5599240
CH 84	100.5599240
CH 85	100.5599240
CH 86	100.5599240
CH 87	100.5599240
CH 88	100.5599240
CH 89	100.5599240
CH 90	100.5599240
CH 91	100.5599240
CH 92	100.5599240
CH 93	100.5599240
CH 94	100.5599240
CH 95	100.5599240
CH 96	100.5599240
CH 97	100.5599240
CH 98	100.5599240
CH 99	100.5599240
CH 100	100.5599240

SSB	0
TR	3.00 Hz

GB	0
PC	1.40

Table 2, entry 2. **2,5-Diphenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-imidazole.**

Table2 Entry 3. **5-Phenyl-1-[2-(trimethylsilyl)-ethoxymethyl]-imidazole**

01-tab1-12c
400 MHz

Current Data Parameters
NAME BT-1-296-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040411
Time 9.19
INSTRUM spect
PROBHD 5 mm Multinuc1
PULPROG zgpg30
TD 32768
FIDRES 0.1200
SOLVENT Aceton
DS 0
SWH 22075.055 Hz
FIDRES 0.673677 Hz
AQ 0.742452 sec
RG 16384
DM 22.650 usec
DE 3.00 usec
TE 300.2 K
D1 0.25000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

CHANNEL F1 13C
NUC1 13C
P1 17.50 usec
PL1 0.00 dB
SFO1 100.5699800 MHz

CHANNEL F2 1H
CDPRG2 waltz16
NUC2 1H
PCPG2 96.25 usec
P2 0.00 dB
PL2 23.00 dB
PL13 23.00 dB
SFO2 399.9215997 MHz

F2 - Processing parameters
SI 65536
SF 100.5599210 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Table 3, entry 1. 3-Phenyl-imidazo[1,2-*a*]pyridine.

Table 3, entry 2. **3-Phenyl-imidazo[1,2-*a*]pyridine-2-carboxylic acid methyl ester.**

Table 3, entry 3. 3-(4-Methoxycarbonylphenyl)-imidazo[1,2-*a*]pyridine-2-carboxylic acid ethyl ester.