

Supporting Information

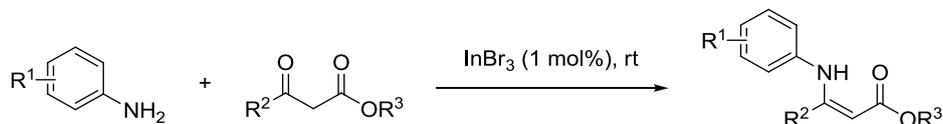
Oxidative Cross-Coupling/Cyclization to Build Polysubstituted Pyrroles from Terminal Alkynes and β -Enamino Esters

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General Information

Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (boiling point is between 60-90 °C). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum to the indicated solvent, and they are listed as volume/volume ratios. NMR spectra were recorded on a Bruker spectrometer at 400 MHz (^1H NMR), 100 MHz (^{13}C NMR). Tetramethylsilane was used as an internal standard. All ^1H NMR spectra were reported in delta (δ) units, parts per million (ppm) downfield from the internal standard. Coupling constants are reported in Hertz (Hz). High resolution mass spectra (HRMS) were measured with a Waters Micromass GCT instrument, accurate masses are reported for the molecular ion ($[\text{M}+\text{H}]^+$). Selective ratios were recorded with a Varian GC 2000 gas chromatography instrument with a FID detector. GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. For the ReactIR kinetic experiments, the reaction spectra were recorded using an IC 15 from Mettler-Toledo AutoChem. Data manipulation was carried out using the iC IR software, version 4.2. For the Raman experiments, the FT Raman spectra of all samples were recorded on a Bruker MultiRam spectrometer (Bruker Optics, Ettlingen, Germany) with a liquid-nitrogen cooled InGaAs detector. A Nd:YAG-laser with an exciting line of 1064 nm was applied as light source for the excitation of Raman scattering. The spectra were recorded over a range of 3600-0 cm^{-1} using an operating spectral resolution of 4 cm^{-1} .

Preparation of β -Enamino Esters



Following a modified procedure of Y. Wang,¹ a mixture of the amine (30 mmol), β -dicarbonyl compound (30 mmol) and $InBr_3$ (0.3 mmol) was stirred at room temperature for the appropriate time. After completion of the reaction as indicated by TLC, the reaction mixture was diluted with H_2O (60 mL) and extracted with EtOAc (3×60 mL). The combined organic layers were dried over $MgSO_4$, concentrated, and the resulting product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 150:1) to afford pure β -enamino esters in good yields.

General Procedure for Preparation of Polysubstituted Pyrroles

A mixture of phenylacetylene **1a** (0.25 mmol), (Z)-methyl-3-(2, 6-diisopropylphenyl-amino)but-2-enoate **2a** (0.5 mmol), Ag_2CO_3 (0.5 mmol), and DBU (0.5 mmol) in DMSO (6 mL) was stirred under N_2 at 80 °C. After completion of the reaction, as indicated by GC or GC-MS, the mixture was quenched with diluted hydrochloride (2 mL, 2 M), the solution was extracted with ethyl acetate (3×5 mL). The organic layers were combined, and dried over sodium sulfate. The pure product was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 150:1) to afford **3a** in 71% yield. The spectroscopic data of all the products are presented below. All the known compounds had satisfactory spectroscopic features which were analogous to spectroscopic data reported in the literature.

One-pot Procedure

In a glovebox, InBr₃ (1.8 mg, 0.005 mmol) was added into a dried Schlenk tube. Then 2,6-diisopropylaniline (88.7 mg, 0.5 mmol) and methyl 3-oxobutanoate (58.1 mg, 0.5 mmol) were added subsequently under N₂. The mixture was stirred vigorously for 30 min at room temperature until became solid, and 6 mL DMSO were added to allow efficient stirring. The reaction vessel was evacuated and backfilled with N₂ three times, then 1-ethynyl-2-methoxybenzene **1b** (33.1 mg, 0.25 mmol), Ag₂CO₃ (137.9 mg, 0.5 mmol) and DBU (76.1 mg, 0.5 mmol) were added respectively. The mixture was stirred at 80 °C. After completion of the reaction, as indicated by GC or GC-MS, the mixture was quenched with diluted hydrochloride (2 mL, 2 M), the solution was extracted with ethyl acetate (3 × 5 mL). The organic layers were combined, and dried over sodium sulfate. The pure product was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate, 100:1) to afford **3b** in 75% yield.

Procedure for the in Situ IR Investigation

Procedure for the stepwise stoichiometric reaction between **1a** and Ag₂CO₃:

An oven-dried three necked reaction vessel was fitted with a magnetic stirring bar. The IR probe was inserted through an adapter into the middle neck; the other two necks were capped by septa for injections and a nitrogen line. The reaction vessel was kept at 80 °C and was allowed to be vacuumed and purged with nitrogen for three times. Then 6.0 mL DMSO and phenylacetylene **1a** (0.25 mmol) were added in via syringes. And the data collection was started, followed by the addition of Ag₂CO₃ (0.5 mmol). In situ IR spectra were recorded over the course of the reaction. As shown in Figure S2, when an equivalent of Ag₂CO₃ was added into the DMSO solution of **1a**, the peak of **1a** (765 cm⁻¹) consumed quickly upon the accumulation of the peak of AgHCO₃ (802 cm⁻¹)² and a new component **I** (754 cm⁻¹). It is reasonable to figure out

that component **I** might be the silver acetylide complex **I**.

ConcIRT spectra of the authentic samples of phenylacetylene **1a**:

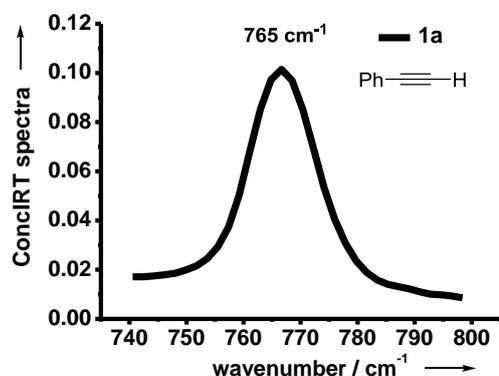


Figure S1. Standard IR spectra of phenylacetylene **1a**.

Three-dimensional Fourier transform IR (3D-FTIR) profile:

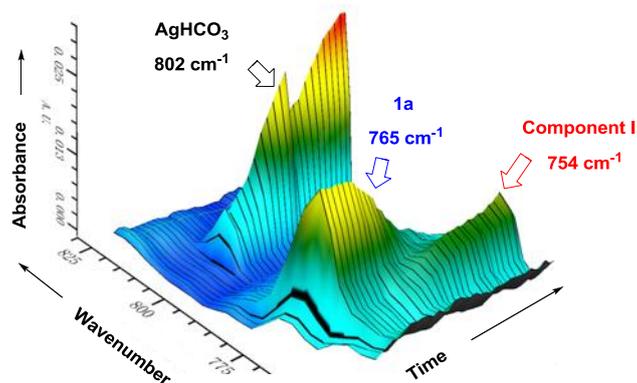


Figure S2. Three-dimensional Fourier transform IR (3D-FTIR) profile of the stoichiometric reaction between **1a** with Ag₂CO₃ in DMSO at 80 °C

Procedure for the Raman Investigation

A mixture of phenylacetylene **1a** (10.2 mg, 0.1 mmol) and Ag₂CO₃ (55.1 mg, 0.2 mmol) in d⁶-DMSO (0.5 mL) was stirred under N₂ at 80 °C for 20 min. A white precipitate was generated. After completion of the reaction, the mixture was filtered,

and the solid was washed by ethanol (2×3 mL). After removed ethanol under reduced pressure, the solid was filled in a small metallic discs and monitored by the FT Raman spectroscopy. As shown in Figure S3, the peak for the C≡C stretching mode of phenylacetylene **1a** (2110 cm^{-1}) is red shifted to 1983 cm^{-1} . This suggests that alkynyl carbon binds covalently with silver and that the C≡C bond is weakened by electron transfer from the silver to the π^* orbital of **1a**.³ The above results clearly revealed that silver acetylide (complex **I**) was formed easily by the reaction of **1a** and Ag_2CO_3 .

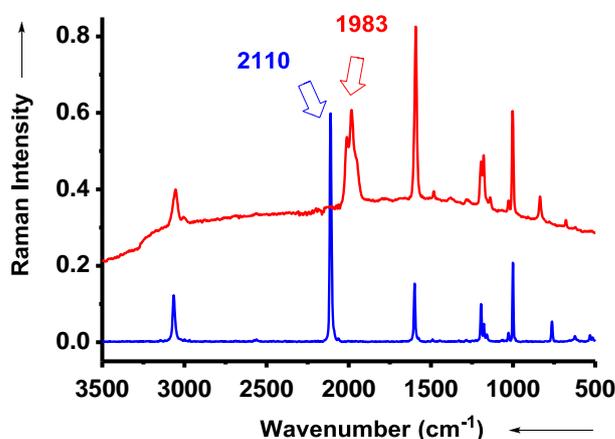


Figure S3. Raman spectra of the white precipitate (top) and **1a** (bottom).

The ^1H NMR Investigation

In addition, the stoichiometric reaction between phenylacetylene **1a** with Ag_2CO_3 in d^6 -DMSO at room temperature was also measured by NMR spectroscopy. Interestingly, no ^1H NMR signal could be observed, which indicated that the polymeric silver acetylide (complex **I**) was completely insoluble in DMSO at room temperature. However, when the filtrate of the mixture of **1a**, Ag_2CO_3 and DBU in d^6 -DMSO was monitored by ^1H NMR spectroscopy, the aryl-H peak of **1a** could be observed. This result might indicate that DBU could promote the monomerization of the polymeric silver acetylide yielding the active nonpolymeric species. Meanwhile, compared to the spectrum of **1a**, the disappearance of the acetylenic proton signal further supported

the formation of C-Ag bond (Figure S4).

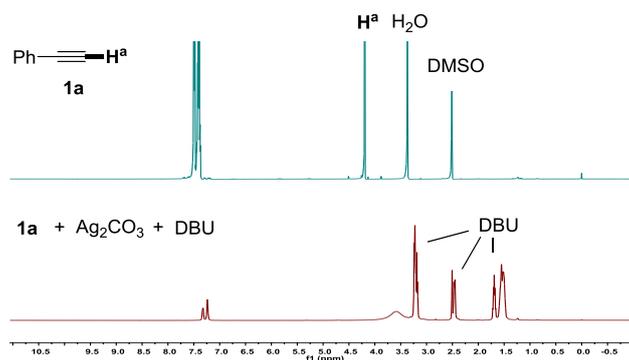


Figure S4. ^1H NMR spectroscopy. The filtrate of the reaction of **1a**, Ag_2CO_3 and DBU in d^6 -DMSO (bottom) and **1a** (top).

Procedure for the ^1H NMR spectroscopy of the reaction of **1a, Ag_2CO_3 and DBU:**

A mixture of phenylacetylene **1a** (10.2mg, 0.1 mmol), Ag_2CO_3 (55.1 mg, 0.2 mmol) and DBU (30.4 mg, 0.2 mmol) in d^6 -DMSO (0.5 mL) was stirred under N_2 at 80 °C for 30 min. After completion of the reaction, the solid was filtered off. Then a syringe equipped with a long needle was used to transfer the filtrate to a sealed NMR tube, and monitored by the ^1H NMR spectroscopy (Figure S4).

^1H NMR spectroscopy of the reaction of (Z)-methyl 3-(phenylamino)but-2-enoate

(2b) with DBU: A mixture of **2b** (9.6 mg, 0.05 mmol) and DBU (7.6 mg, 0.05 mmol) in d^6 -DMSO (0.3 mL) was stirred under N_2 at 80 °C for 1 h. After completion of the reaction, a syringe equipped with a long needle was used to transfer the solution to a sealed NMR tube, and monitored by the ^1H NMR spectroscopy. As shown in Figure S5, about half of **2b** was deprotonated in the presence of DBU.

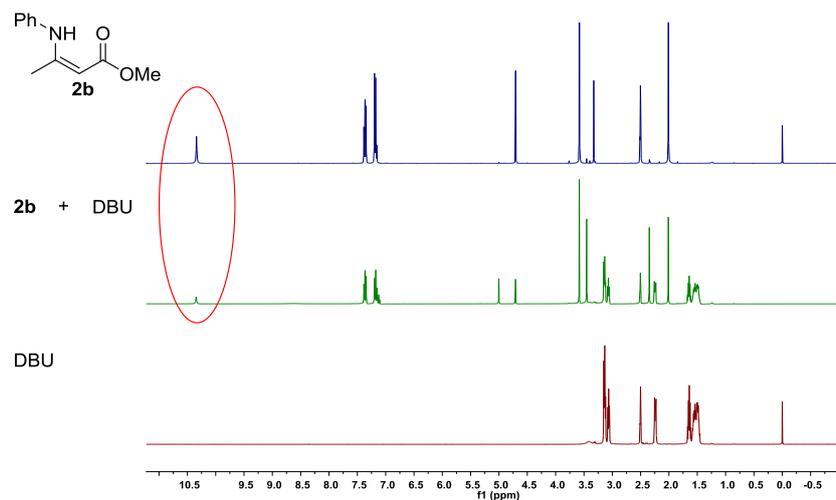
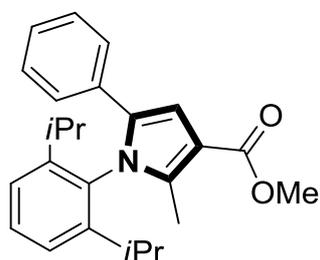


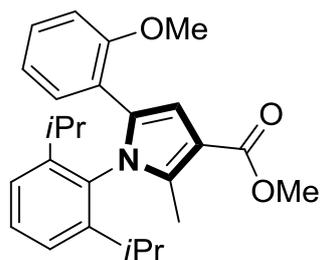
Figure S5. ^1H NMR spectroscopy. The reaction of **2b** with DBU in d^6 -DMSO (middle), **2b** (top) and DBU (bottom).

Analytical Data of Products

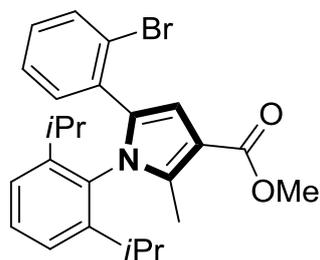


Methyl-1-(2,6-diisopropylphenyl)-2-methyl-5-phenyl-1H-pyrrole-3-carboxylate

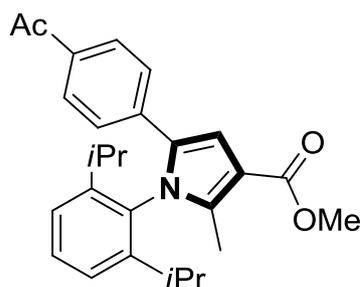
(3a). ^1H NMR (400 MHz, CDCl_3): δ 7.45 (t, $J = 7.6$ Hz, 1H), 7.24 (d, $J = 7.6$ Hz, 2H), 7.12-7.05 (m, 3H), 7.04-6.97 (m, 3H), 3.87 (s, 3H), 2.41 (m, $J = 6.8$ Hz, 2H), 2.27 (s, 3H), 1.10 (d, $J = 6.8$ Hz, 6H), 0.83 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.3, 147.0, 138.9, 133.9(1), 133.8(6), 132.4, 130.1, 128.3, 126.9, 126.6, 124.7, 112.6, 109.6, 51.2, 28.3, 24.5, 23.5, 12.8. HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{30}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 376.2277; found: 376.2281.



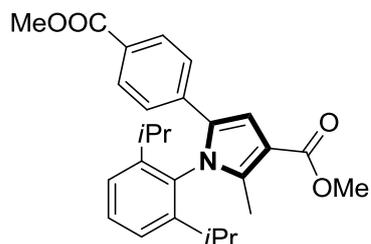
Methyl-1-(2,6-diisopropylphenyl)-5-(2-methoxyphenyl)-2-methyl-1H-pyrrole-3-carboxylate (3b). ^1H NMR (400 MHz, CDCl_3): δ 7.35 (t, $J = 7.6$ Hz, 1H), 7.15 (d, $J = 8.0$ Hz, 2H), 7.11-7.02 (m, 2H), 6.84 (d, $J = 8.0$ Hz, 1H), 6.72-6.65 (m, 1H), 6.59-6.50 (m, 1H), 3.85 (s, 3H), 3.80 (s, 3H), 2.47 (m, $J = 6.8$ Hz, 2H), 2.28 (s, 3H), 1.08 (d, $J = 6.8$ Hz, 6H), 0.89 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.4, 157.4, 147.1, 138.0, 133.6, 130.0, 129.7, 128.6, 128.3, 124.3, 121.0, 119.5, 113.0, 112.1, 111.0, 55.7, 51.0, 28.1, 24.9, 23.5, 13.3. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{32}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 406.2382; found: 406.2385.



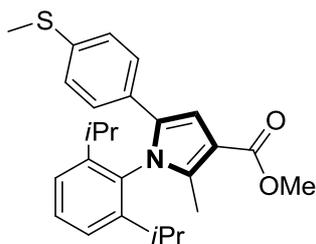
Methyl-5-(2-bromophenyl)-1-(2,6-diisopropylphenyl)-2-methyl-1H-pyrrole-3-carboxylate (3c). ^1H NMR (400 MHz, CDCl_3): δ 7.61-7.56 (m, 1H), 7.34 (t, $J = 7.8$ Hz, 1H), 7.14 (d, $J = 7.6$ Hz, 2H), 7.04 (s, 1H), 6.99-6.88 (m, 2H), 6.86-6.81 (m, 1H), 3.86 (s, 3H), 2.50 (m, $J = 6.8$ Hz, 2H), 2.29 (s, 3H), 1.09 (d, $J = 6.8$ Hz, 6H), 0.97 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.3, 147.3, 138.3, 133.8, 132.8, 132.7, 131.3, 131.1, 129.9, 128.7, 126.3, 125.2, 124.4, 113.9, 112.1, 51.2, 28.1, 25.1, 23.7, 13.6. HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{29}\text{BrNO}_2$ $[\text{M}+\text{H}]^+$: 454.1382; found: 454.1387.



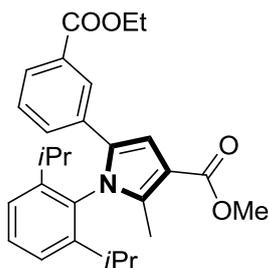
Methyl-5-(4-acetylphenyl)-1-(2,6-diisopropylphenyl)-2-methyl-1H-pyrrole-3-carboxylate (3d). ^1H NMR (400 MHz, CDCl_3): δ 7.68 (d, $J = 8.8$ Hz, 2H), 7.50 (t, $J = 7.6$ Hz, 1H), 7.28 (d, $J = 7.6$ Hz, 2H), 7.19 (s, 1H), 7.08 (d, $J = 8.8$ Hz, 2H), 3.89 (s, 3H), 2.50 (s, 3H), 2.39 (m, $J = 6.8$ Hz, 2H), 2.28 (s, 3H), 1.11 (d, $J = 6.8$ Hz, 6H), 0.84 (d, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 197.6, 166.0, 146.8, 140.2, 136.8, 134.6, 133.8, 132.3, 130.5, 128.6, 126.0, 124.9, 113.3, 111.6, 51.3, 28.4, 26.7, 24.4, 23.6, 12.8. HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{32}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 418.2382; found: 418.2385.



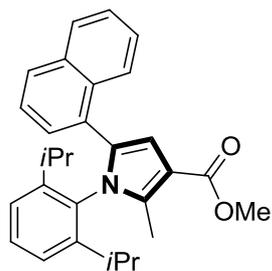
Methyl-1-(2,6-diisopropylphenyl)-5-(4-(methoxycarbonyl)phenyl)-2-methyl-1H-pyrrole-3-carboxylate (3e). ^1H NMR (400 MHz, CDCl_3): δ 7.79-7.72 (m, 2H), 7.49 (t, $J = 7.8$ Hz, 1H), 7.26 (d, $J = 8.0$ Hz, 2H), 7.16 (s, 1H), 7.08-7.02 (m, 2H), 3.88 (s, 3H), 3.84 (s, 3H), 2.38 (m, $J = 6.8$ Hz, 2H), 2.27 (s, 3H), 1.10 (d, $J = 6.8$ Hz, 6H), 0.83 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 167.1, 166.0, 146.9, 140.0, 136.7, 133.8, 132.5, 130.5, 129.7, 127.6, 126.0, 124.9, 113.2, 111.4, 52.2, 51.3, 28.4, 24.4, 23.6, 12.8. HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{32}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 434.2331; found: 434.2332.



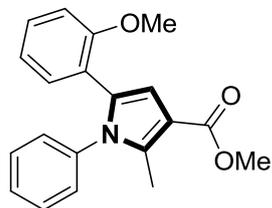
Methyl-1-(2,6-diisopropylphenyl)-2-methyl-5-(4-(methylthio)phenyl)-1H-pyrrole-3-carboxylate (3f). ^1H NMR (400 MHz, CDCl_3): δ 7.46 (t, $J = 7.8$ Hz, 1H), 7.24 (d, $J = 8.0$ Hz, 2H), 7.00-6.95 (m, 3H), 6.94-6.89 (m, 2H), 3.87 (s, 3H), 2.46-2.34 (m, 5H), 2.26 (s, 3H), 1.09 (d, $J = 6.8$ Hz, 6H), 0.85 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.2, 147.0, 138.8, 136.6, 133.9, 133.3, 130.2, 129.3, 127.1, 126.4, 124.7, 112.7, 109.4, 51.1, 28.3, 24.4, 23.6, 15.9, 12.8. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{32}\text{NO}_2\text{S}$ $[\text{M}+\text{H}]^+$: 422.2154; found: 422.2150.



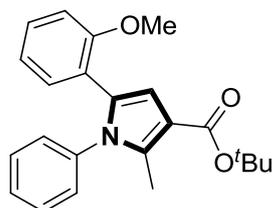
Methyl-1-(2,6-diisopropylphenyl)-5-(3-(ethoxycarbonyl)phenyl)-2-methyl-1H-pyrrole-3-carboxylate (3g). ^1H NMR (400 MHz, CDCl_3): δ 7.76 (d, $J = 8.0$ Hz, 1H), 7.63 (s, 1H), 7.47 (t, $J = 7.6$ Hz, 1H), 7.31-6.24 (m, 3H), 7.12 (t, $J = 7.8$ Hz, 1H), 7.09 (s, 1H), 4.26 (q, $J = 7.2$ Hz, 2H), 3.88 (s, 3H), 2.40 (m, $J = 6.8$ Hz, 2H), 2.28 (s, 3H), 1.32 (t, $J = 7.0$ Hz, 3H), 1.10 (d, $J = 6.8$ Hz, 6H), 0.84 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.5, 166.2, 146.9, 139.3, 133.6, 132.7, 132.6, 131.2, 130.5, 130.3, 128.5, 127.7, 127.5, 124.8, 112.9, 110.3, 61.1, 51.2, 28.4, 24.5, 23.5, 14.6, 12.8. HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{34}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 448.2488; found: 448.2481.



Methyl-1-(2,6-diisopropylphenyl)-2-methyl-5-(naphthalen-1-yl)-1H-pyrrole-3-carboxylate (3h) ^1H NMR (400 MHz, CDCl_3): δ 8.31 (d, $J = 8.4$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 1H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.49-7.40 (m, 2H), 7.31-7.23 (m, 1H), 7.12 (t, $J = 7.8$ Hz, 1H), 7.07 (d, $J = 8.0$ Hz, 2H), 7.01 (d, $J = 6.8$ Hz, 1H), 6.96 (s, 1H), 3.89 (s, 3H), 2.50 (m, $J = 6.8$ Hz, 2H), 2.34 (s, 3H), 1.07 (d, $J = 6.8$ Hz, 6H), 0.66 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.5, 147.3, 138.6, 134.1, 133.2, 130.9, 129.7, 129.6, 128.4, 127.9, 126.5, 126.3, 125.9, 124.4, 124.3, 113.3, 112.3, 51.2, 28.2, 25.1, 23.5, 13.7. HRMS (ESI) calcd for $\text{C}_{29}\text{H}_{32}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 426.2433; found: 426.2428.

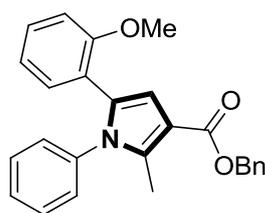


Methyl-5-(2-methoxyphenyl)-2-methyl-1-phenyl-1H-pyrrole-3-carboxylate (3i). ^1H NMR (400 MHz, CDCl_3): δ 7.31-7.23 (m, 3H), 7.22-7.15 (m, 2H), 7.09-7.03 (m, 2H), 6.88-6.82 (m, 1H), 6.69 (s, 1H), 6.63 (d, $J = 8.0$ Hz, 1H), 3.83 (s, 3H), 3.41 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.3, 157.0, 138.5, 137.5, 132.3, 131.0, 129.5, 128.5, 128.1, 127.8, 122.0, 120.4, 112.4, 110.8, 110.5, 54.9, 51.1, 13.0. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 322.1443; found: 322.1441.



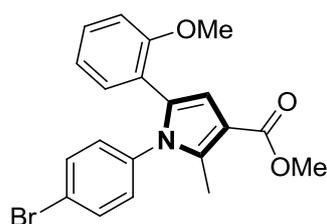
Tert-butyl-5-(2-methoxyphenyl)-2-methyl-1-phenyl-1H-pyrrole-3-carboxylate

(3j). ^1H NMR (400 MHz, CDCl_3): δ 7.30-7.15 (m, 5H), 7.09-7.03 (m, 2H), 6.90-6.83 (m, 1H), 6.66 (s, 1H), 6.62 (d, $J = 8.0$ Hz, 1H), 3.40 (s, 3H), 2.41 (s, 3H), 1.57 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 165.4, 157.0, 138.6, 136.7, 132.4, 131.2, 130.7, 129.5, 129.1, 128.5, 128.2, 127.7, 122.2, 120.5, 114.4, 111.1, 110.5, 65.9, 54.9, 28.8, 13.1. HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{26}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 364.1913; found: 364.1915.



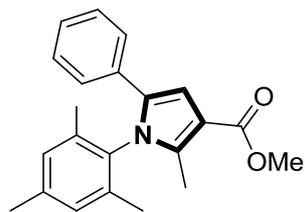
Benzyl-5-(2-methoxyphenyl)-2-methyl-1-phenyl-1H-pyrrole-3-carboxylate (3k).

^1H NMR (400 MHz, CDCl_3): δ 7.45 (d, $J = 7.2$ Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 2H), 7.33-7.15 (m, 6H), 7.09-7.04 (m, 2H), 6.85 (t, $J = 7.6$ Hz, 1H), 6.75 (s, 1H), 6.62 (d, $J = 8.0$ Hz, 1H), 5.31 (s, 2H), 3.39 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 165.6, 157.0, 138.5, 137.8, 137.3, 132.4, 131.1, 129.6, 128.7, 128.5, 128.2, 128.1, 128.0, 127.8, 122.0, 120.5, 112.4, 110.9, 110.5, 65.4, 54.9, 13.1. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{24}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 398.1756; found: 398.1757.

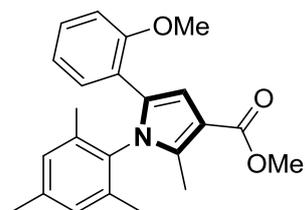


Methyl-1-(4-bromophenyl)-5-(2-methoxyphenyl)-2-methyl-1H-pyrrole-3-carboxylate (3l).

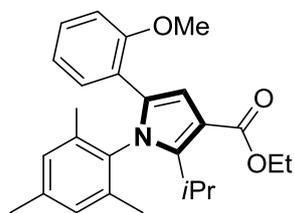
^1H NMR (400 MHz, CDCl_3): δ 7.40 (d, $J = 8.8$ Hz, 2H), 7.25-7.18 (m, 2H), 6.94 (d, $J = 8.8$ Hz, 2H), 6.91-6.85 (m, 1H), 6.70-6.62 (m, 2H), 3.83 (s, 3H), 3.43 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.2, 156.8, 137.6, 137.3, 132.3, 131.7, 131.0, 129.8, 129.7, 121.6, 120.6, 112.8, 111.0, 110.6, 54.9, 51.1, 12.9. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{19}\text{BrNO}_3$ $[\text{M}+\text{H}]^+$: 400.0548; found: 400.0549.



Methyl-1-mesityl-2-methyl-5-phenyl-1H-pyrrole-3-carboxylate (3m). ^1H NMR (400 MHz, CDCl_3): δ 7.16-7.09 (m, 3H), 7.08-7.02 (m, 2H), 6.91 (s, 2H), 6.89 (s, 1H), 3.85 (s, 3H), 2.31 (s, 3H), 2.23 (s, 3H), 1.87 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.3, 138.8, 137.6, 136.4, 134.1, 133.1, 132.7, 129.4, 128.4, 126.9, 126.7, 112.7, 109.8, 51.1, 21.3, 17.9, 11.8. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{24}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 334.1807; found: 334.1805.

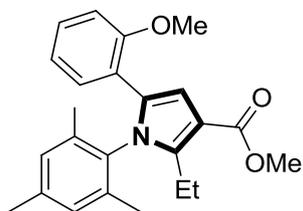


Methyl-1-mesityl-5-(2-methoxyphenyl)-2-methyl-1H-pyrrole-3-carboxylate (3n). ^1H NMR (400 MHz, CDCl_3): δ 7.18-7.10 (m, 1H), 6.97-6.90 (m, 1H), 6.85-6.79 (m, 3H), 6.78-6.68 (m, 2H), 3.84 (s, 3H), 3.64 (s, 3H), 2.24 (s, 3H), 2.23 (s, 3H), 1.90 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.5, 157.4, 138.1, 137.2, 136.5, 134.0, 131.7, 129.7, 129.0, 121.8, 120.1, 112.2, 111.8, 110.8, 55.2, 51.0, 21.3, 18.0, 12.1. HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{26}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 364.1913; found: 364.1910.



Ethyl-2-isopropyl-1-mesityl-5-(2-methoxyphenyl)-1H-pyrrole-3-carboxylate (3o). ^1H NMR (400 MHz, CDCl_3): δ 7.16-7.09 (m, 1H), 6.96-6.90 (m, 1H), 6.86 (s, 1H), 6.79 (s, 2H), 6.76-6.67 (m, 2H), 4.30 (q, $J = 7.2$ Hz, 2H), 3.65 (s, 3H), 2.61 (m, $J = 6.8$ Hz, 1H), 2.23 (s, 3H), 1.94 (s, 6H), 1.40-1.33 (m, 9H); ^{13}C NMR (100 MHz,

CDCl₃): δ 165.4, 157.5, 146.2, 138.0, 136.9, 134.2, 131.9, 128.9, 128.8(3), 128.8(1), 121.9, 119.9, 113.7, 111.7, 110.6, 59.7, 55.2, 27.3, 21.3, 20.7, 18.2, 14.8. HRMS (ESI) calcd for C₂₆H₃₂NO₃ [M+H]⁺: 406.2382; found: 406.2382.



Methyl-2-ethyl-1-mesityl-5-(2-methoxyphenyl)-1H-pyrrole-3-carboxylate (3p). ¹H NMR (400 MHz, CDCl₃): δ 7.17-7.10 (m, 1H), 6.96-6.86 (m, 1H), 6.81 (s, 1H), 6.80 (s, 2H), 6.77-6.67 (m, 2H), 3.83 (s, 3H), 3.62 (s, 3H), 2.68 (q, *J* = 7.2 Hz, 2H), 2.24 (s, 3H), 1.90 (s, 6H), 0.94 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.1, 157.4, 143.2, 138.0, 136.5, 133.8, 131.7, 129.5, 129.0, 128.9, 121.8, 120.0, 112.1, 111.5, 110.7, 55.2, 51.0, 21.3, 19.5, 18.2, 14.2. HRMS (ESI) calcd for C₂₄H₂₈NO₃ [M+H]⁺: 378.2069; found: 378.2072.

Reference:

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3. P. Maity, T. Wakabayashi, N. Ichikuni, H. Tsunoyama, S. Xie, M. Yamauchi and T. Tsukuda, *Chem. Commun.*, 2012, **48**, 6085-6087.

Spectrum:

