Supplementary Material (ESI) for Chemical Communications
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A highly diastereoselective, catalytic three-component assembly reaction for the synthesis of spiro-pyrrolidinyloxindoles

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General Information.

All reactions were carried out under an atmosphere of argon or nitrogen in flame-dried glassware with magnetic stirring. THF, Et_2O, CH_2Cl_2 and toluene were purified by passage through a bed of activated alumina.[1] Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego.[2] Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and *p*-anisaldehyde, ceric ammonium nitrate stain, or phosphomolybic acid followed by heating.

Melting points were obtained on a Melt-temp 3 instrument and are uncorrected. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ^{1}H-NMR spectra were recorded on a Varian Inova 500 (500 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 7.26 ppm). Data are reported as (ap = apparent, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad; coupling constant(s) in Hz; integration. Proton-decoupled ^{13}C-NMR spectra were recorded on a Varian Inova 500 (125 MHz) spectrometer and are reported in ppm using solvent as an internal standard ($CDCl_3$ at 77.0 ppm).

Mass spectra data were obtained on either a Thermo-Finnigan MAT900 high resolution mass spectrometer, a VG 70-250SE HRMS or Thermo-Finnigan LCQ Advantage HPLC-MS in the Northwestern University Analytical Services Laboratory. Single crystal X-ray data were collected using a Bruker SMART-1000 diffractometer equipped with a CCD detector and processed using SAINT-NT from Bruker. Ethyl diazoacetate (EDA, **2a**), *t*-butyl diazoacetate (**2b**), isatin and 5-chloroisatin were all purchased from Aldrich Chemical Company and used without purification. Copper(I) trifluoromethanesulfonate was prepared using a modification to the method of Kochi and coworkers.[3]

Imines were prepared by combining the corresponding aldehyde (freshly distilled), appropriately substituted aniline, and either activated 4Å molecular sieves or anhydrous magnesium sulfate in dichloromethane.[4] The reactions were stirred for 16 h, then filtered and concentrated *in vacuo*. If necessary, the resulting imines were re-crystallized from a suitable organic solvent mixture to afford imines that were >95% pure by ^{1}H NMR analysis.

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[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.
[2] Perrin, D. D. and Armarego, W. L. *Pruification of Laboratory Chemicals;* 3rd ed., Pergamon Press, Oxford, 1998
[3] Salomon, R. G.; Kochi, J. K. *J. Am. Chem. Soc.* **1973**, *95*, 1889-1897.
[4] (a) Antilla, J. C.; Wulff, W. D. *J. Am. Chem. Soc.* **1999**, *121*, 5099-5100. (b) Derdau, V.; Snieckus, V. *J. Org. Chem.* **2001**, *66*, 1992-1998. (c) Casarrubios, L.; Perez, J. A.; Brookhart, M.; Templeton, J. L. *J. Org. Chem.* **1996**, *61*, 8358-8359.

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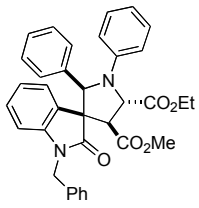
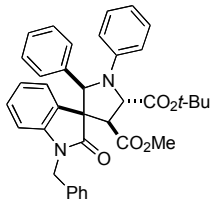
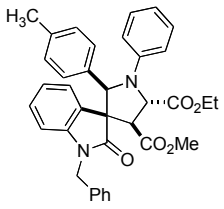
.**General Experimental Procedure for the Copper(I)-Catalysed Reaction:**
To a solution of imine **1a-i** (1.3 mmol) dipolarophile **3a-d** (0.41 mmol) and catalyst (0.04-0.08 mmol) in CH_2Cl_2 (3.0 mL) heated at reflux was added a solution of a diazo ester **2a or b** (1.3 mmol) in CH_2Cl_2 (4.5 mL) via syringe pump over 3 h. After complete addition, the reaction was heated for an additional 1-2 hours, cooled and then filtered through a silica gel plug using CH_2Cl_2 (50 mL). The solvent was removed and diastereoselectivities were assigned by analysis of ^{1}H NMR (500 MHz) spectra and correlated with GC integrations. The reaction mixture was purified by flash column chromatography (mixtures of ether/hexanes or EtOAc/hexanes as eluent), usually by dry loading the sample, to afford the desired cycloaddition product.



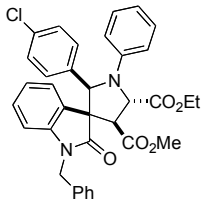
(4a): Purified with 40% ether/hexanes, yielding 181 mg (79%) of **4a** as a light bown oil. R_f = 0.26 (40:60 ether/hexanes); IR (film) 2980, 1742, 1716, 1606 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37-6.34 (m, 19H); 5.62-5.60 (m, 2H); 4.98-4.76 (m, 2H); 4.23-4.21 (d, *J*=7.9, 1H); 4.21-4.02 (m, 2H); 3.18 (s, 3H); 0.99-0.95 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.5, 169.9, 169.0, 163.2, 162.5, 145.5, 144.2, 143.8, 142.7, 138.1, 135.7, 134.6, 129.3, 129.1, 128.8, 128.6, 127.8, 127.6, 127.5, 122.4, 120.4, 119.2, 118.6, 114.0, 71.3, 70.0, 62.1, 52.8, 52.6, 44.5, 14.7; Mass calculated for $C_{35}H_{36}N_2O_5$, 560.6. Found: 561.7 $[M+H]^+$, 583.8 $[M+Na]^+$, 1144.2 $[(2M)+Na]^+$.



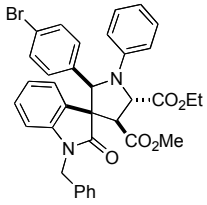
(4b): Purified with 40% ether/hexanes, yielding 178 mg (74%) of **4b** as a white solid (m. p. 100-102 °C). R_f = 0.32 (40/60 ether/hexanes); IR (film) 2977, 2947, 2906, 1739, 1719, 1605 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39-6.38 (m, 19H); 5.47-5.41 (b, m, 2H); 4.98-4.72 (m, 2H); 4.19-4.17 (d, *J*=7.9, 1H); 3.18 (s, 3H); 1.19 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.4, 171.5, 169.1, 142.7, 135.7, 134.8, 128.8, 128.6, 127.8, 127.7, 127.6, 126.5, 126.0, 122.3, 120.3, 119.4, 108.8, 82.3, 71.7, 66.7, 61.6, 53.9, 52.2, 44.2, 27.8; LRMS (electrospray): Mass calculated for $C_{37}H_{36}N_2O_5$, 588.7. Found: 589.6 $[M+H]^+$, 611.6 $[M+Na]^+$, 1200.2 $[(2M)+Na]^+$.



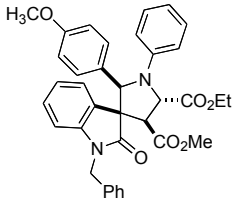
(5): Purified with 40% ether/hexanes, yielding 169 mg (72%) of **5** as a waxy solid. R_f = 0.27 (40/60 ether/hexanes); IR (film) 2953, 2950, 2946, 1742, 1716, 1606 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43-6.39 (m, 18H); 5.61-5.59 (d, *J*=7.9 Hz, 1H); 5.54 (s, 1H); 4.91-4.81 (m, 2H); 4.23-4.21 (d, *J*=7.9, 1H); 4.13-3.97 (m, 2H); 3.18 (s, 3H); 0.95-0.91 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.3, 172.4, 169.0, 145.6, 142.8, 137.2, 135.7, 131.6, 128.8, 128.7, 128.6, 128.5, 127.7, 127.6, 127.5, 126.5, 125.9, 122.3, 120.3, 119.3, 108.9, 71.6, 65.6, 61.6, 61.5, 53.9, 52.2, 44.1, 21.3, 14.1; LRMS (electrospray): Mass calculated for $C_{36}H_{34}N_2O_5$, 575.7. Found 575.7 $[M]^+$, 576.7 $[M+H]^+$.



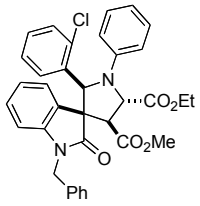
(6): Purified with 40% ether/hexanes, yielding 157 mg (60%) of **6** as a waxy white solid. R_f = 0.26 (40/60 ether/hexanes); IR (film) 2950, 2942, 2936, 2910, 1742, 1716, 1609 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.39-6.40 (m, 18H); 5.61-5.59 (d, *J*=7.9 Hz, 1H); 5.50 (s, 1H); 4.93-4.76 (m, 2H); 4.22-4.20 (d, *J*=7.9, 1H); 4.10-3.95 (m, 2H); 3.18 (s, 3H); 0.94-0.92 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.9, 172.3, 168.8, 145.2, 142.8, 135.6, 133.4, 133.4, 129.2, 129.0, 128.9, 128.7, 128.0, 127.9, 127.6, 126.0, 125.8, 122.5, 120.8, 119.3, 109.2, 71.1, 65.6, 61.6, 61.4, 53.8, 52.3, 44.2, 14.1; LRMS (electrospray): Mass calculated for $C_{35}H_{31}ClN_2O_5$, 595.1. Found: 596.7 $[M+H]^+$, 618.9 $[M+Na]^+$, 1213.3 $[(2M)+Na]^+$.



(7): Purified with 40% ether/hexanes, yielding 144 mg (55%) of **7** as an off-white solid (m.p. 95-96 °C). R_f = 0.20 (33/67 ether/hexanes); IR (film) 2953, 2950, 2946, 2933, 2890, 1742, 1716, 1609, 1590 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.41-6.46 (m, 18H); 5.61-5.59 (d, *J*=7.9 Hz, 1H); 5.51 (s, 1H); 4.94-4.80 (m, 2H); 4.25-4.22 (d, *J*=7.9, 1H); 4.09-3.97 (m, 2H); 3.18 (s, 3H); 0.98-0.94 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0, 172.2, 168.8, 145.1, 142.8, 135.5, 133.9, 131.0, 129.3, 129.2, 127.9, 128.7, 127.9, 127.6, 125.9, 125.8, 122.5, 121.7, 120.8, 119.3, 109.2, 71.1, 65.6, 61.6, 61.3, 53.8, 52.3, 44.2, 14.1; LRMS (electrospray): Mass calculated for $C_{35}H_{31}BrN_2O_5$, 639.6. Found: 640.8 $[M+H]^+$, 662.8 $[M+Na]^+$, 1301.5 $[(2M)+Na]^+$.

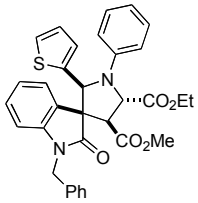


(8): Purified with 40% ether/hexanes, yielding 111 mg (46%) of **8** as a colorless oil. R_f = 0.27 (50/50 ether/hexanes); IR (film) 2953, 2950, 2933, 1741, 1716, 1605 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-6.38 (m, 18H); 6.13 (s, 1H); 5.62-5.59 (d, *J*=7.9 Hz 1H); 5.00-4.81 (m, 2H); 4.37-4.33 (d, *J*=7.9, 1H); 4.22-3.99 (m, 2H); 3.82 (s, 3H); 3.20 (s, 3H); 1.01-0.98 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.1, 172.8, 159.0, 145.9, 142.2, 135.7, 129.1, 129.0, 128.9, 128.7, 128.0, 127.9, 127.8, 127.6, 126.4, 126.0, 122.1, 120.6, 119.1, 113.3, 109.2, 71.1, 65.6, 61.6, 61.5, 55.4, 52.1, 44.3, 14.0; LRMS (electrospray): Mass calculated for $C_{36}H_{33}N_2O_6$ 590.7. Found: 591.6 $[M+H]^+$.

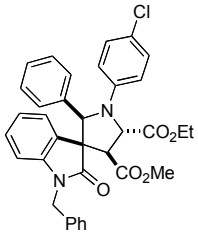


(9): Purified with 35% ether/hexanes, yielding 130 mg (53%) of **11** as an off-white solid (m.p. 151-153 °C). R_f = 0.15 (30/70 ether/hexanes); IR (film) 2970, 2940, 2921, 2901, 1739, 1710, 1605 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-6.39 (m, 18H); 6.13 (s, 1H); 5.62-5.59 (d, *J*=7.9 Hz 1H); 5.00-4.81 (m, 2H); 4.37-4.33 (d, *J*=7.9, 1H); 4.22-3.99 (m, 2H); 3.16 (s, 3H); 1.01-0.98 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0, 172.3, 169.1, 145.1, 143.1, 135.8, 133.3, 129.5, 129.3, 128.8, 127.7, 127.5,

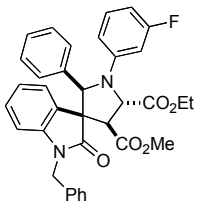
126.4, 125.7, 121.8, 120.6, 119.0, 109.0, 67.0, 65.6, 62.1, 62.0, 61.6, 54.2, 53.0, 52.2, 52.3, 44.4, 14.3; LRMS (electrospray): Mass calculated for $C_{35}H_{31}ClN_2O_5$, 595.1. Found: 595.8 $[M]^+$, 618.8 $[M+Na]^+$, 1213.2 $[(2M)+Na]^+$.



(10): Purified with 40% ether/hexanes, yielding 100 mg (43%) of **10** as a powdery off-white solid (m.p. 195-198 °C). R_f = 0.22 (40/60 ether/hexanes); IR (film) 2954, 2923, 1741, 1717, 1601 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-6.38 (m, 18H); 5.60-5.58 (d, *J*=7.9 Hz, 1H); 5.52 (s, 1H); 4.94-4.76 (m, 2H); 4.23-4.22 (d, *J*=7.9, 1H); 4.14-4.02 (m, 2H); 3.18 (s, 3H); 1.04-1.01 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.8, 172.1, 168.9, 145.4, 143.1, 138.2, 135.6, 129.2, 128.9, 128.6, 127.8, 127.4, 126.7, 126.4, 126.1, 125.9, 125.6, 122.6, 121.1, 119.6, 109.1, 68.2, 65.6, 61.6, 61.4, 53.6, 52.3, 44.2, 14.1; LRMS (electrospray): Mass calculated for $C_{35}H_{31}ClN_2O_5$, 566.7. Found: 568.6 $[M+H]^+$.



(11): Purified with 40% ether/hexanes, yielding 122 mg (50%) of **11** as a powdery off-white solid (m.p. 189-193 °C). R_f = 0.24 (40/60 ether/hexanes); IR (film) 2955, 2931, 2926, 2890, 1742, 1716, 1608 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-6.38 (m, 18H); 5.60-5.58 (d, *J*=7.9 Hz, 1H); 5.52 (s, 1H); 4.94-4.76 (m, 2H); 4.23-4.22 (d, *J*=7.9, 1H); 4.14-4.02 (m, 2H); 3.18 (s, 3H); 1.04-1.01 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0, 172.1, 168.8, 144.2, 142.8, 135.6, 134.2, 129.0, 128.8, 128.6, 127.9, 127.8, 127.6, 127.5, 126.1, 125.8, 125.5, 122.4, 120.3, 109.0, 71.8, 65.6, 61.7, 61.5, 53.9, 52.3, 44.2, 14.3; LRMS (electrospray): Mass calculated for $C_{35}H_{31}ClN_2O_5$, 595.1. Found: 595.8 $[M]^+$, 618.8 $[M+Na]^+$, 1213.2 $[(2M)+Na]^+$.



(12): Purified with 40% ether/hexanes, yielding 97 mg (41%) of **11** as a colourless oil. R_f = 0.26 (40/60 ether/hexanes); IR (film) 2946, 2921, 1743, 1717, 1606, 1603 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.36-6.38 (m, 18H); 5.59-5.57 (d, *J*=7.9 Hz, 1H); 5.50 (s, 1H); 4.94-4.75 (m, 2H); 4.23-4.22 (d, *J*=7.9, 1H); 4.17-4.06 (m, 2H); 3.17 (s, 3H); 1.02-1.01 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0, 172.1, 168.7, 135.6, 134.2, 129.8, 129.7, 129.0, 128.8, 128.0, 127.8, 127.6, 126.1, 125.8, 122.4, 114.6, 109.2, 109.0, 107.2, 107.0, 106.3, 106.1, 71.8, 65.6, 61.7, 53.9, 52.3, 44.2, 14.3; LRMS (electrospray): Mass calculated for $C_{35}H_{31}FN_2O_5$, 578.6. Found: 601.3 $[M+Na]^+$, 1180.2 $[(2M)+Na]^+$.

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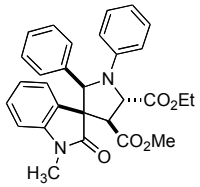
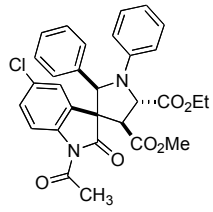
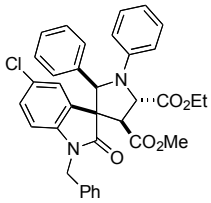


(13): Purified with 40% ether/hexanes, yielding 137 mg (69%) of **13** as a light yellow oil. R_f = 0.11 (40/60 ether/hexanes); IR (film) 2930, 2923, 1741, 1719, 1606 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-7.41 (m, 14H); 5.61-5.60 (d, *J*=7.9 Hz, 1H); 5.49 (s, 1H); 4.19-4.17 (d, *J*=7.9 Hz, 1H); 4.13-3.99 (m, 2H); 3.28 (s, 3H); 3.12 (s, 3H); 0.93-0.96 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.0, 172.5, 169.0, 145.5, 143.5, 134.7, 128.9, 128.6, 127.6, 127.5, 127.3, 126.3, 125.7, 122.3, 120.3, 119.1, 151.2, 71.5, 65.7, 61.7, 61.5, 53.6, 52.4, 26.7, 13.9; LRMS (electrospray): Mass calculated for $C_{29}H_{28}N_2O_5$ 484.5. Found 485.6 $[M+H]^+$.



(14): Purified with 40% ether/hexanes, yielding 136 mg (56%) of **14** as a viscous yellow oil. R_f = 0.45 (40:60 ether/hexanes); IR (film) 2921, 1747, 1726, 1725, 1588 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.89-6.68 (m, 14H); 5.58-5.57 (d, *J*=7.9 1H); 5.53 (s, 1H); 4.22-4.20 (d, *J*=7.9 Hz, 1H); 4.13-4.02 (m, 2H); 3.30 (s, 3H); 2.74 (s, 3H); 0.99-0.96 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.7, 172.3, 170.5, 168.6, 145.1, 139.0, 134.2, 129.3, 128.7, 128.2, 128.0, 127.7, 127.1, 126.7, 125.4, 125.2, 124.9, 120.9, 120.0, 119.4, 119.2, 116.1, 72.5, 65.4, 62.2, 62.0, 61.7, 62.7, 52.7, 27.0, 14.1; LRMS (electrospray): Mass calculated for $C_{30}H_{27}ClN_2O_6$ 546.9. Found: 547.8 $[M+H]^+$, 1116.9 $[(2M)+Na]^+$



(15): Purified with 25% EtOAc/hexanes, yielding 207 mg (84%) of **15** as a white solid. R_f = 0.24 (25:75 EtOAc/hexanes); IR (film) 3054, 2985, 1742, 1710, 1598 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.40-6.34 (m, 18H); 5.59-5.58 (d, *J*=7.9 Hz, 1H); 5.56 (s, 1H); 4.94-4.75 (m, 2H); 4.24-4.25 (d, *J*=7.9, 1H); 4.14-3.98 (m, 2H); 3.28 (s, 3H); 1.01-0.97 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.8, 172.3, 168.7, 145.2, 141.4, 135.2, 134.3, 129.5, 129.0, 128.9, 128.8, 128.7, 128.2, 128.0, 127.9, 127.7, 127.5, 126.0, 120.8, 119.4, 118.8, 115.3, 109.8, 71.6, 65.5, 61.6, 53.8, 52.5, 44.3, 14.1; LRMS (electrospray): Mass calculated for $C_{35}H_{31}ClN_2O_5$, 595.1. Found: 595.8 $[M]^+$, 618.8 $[M+Na]^+$, 1213.2 $[(2M)+Na]^+$.

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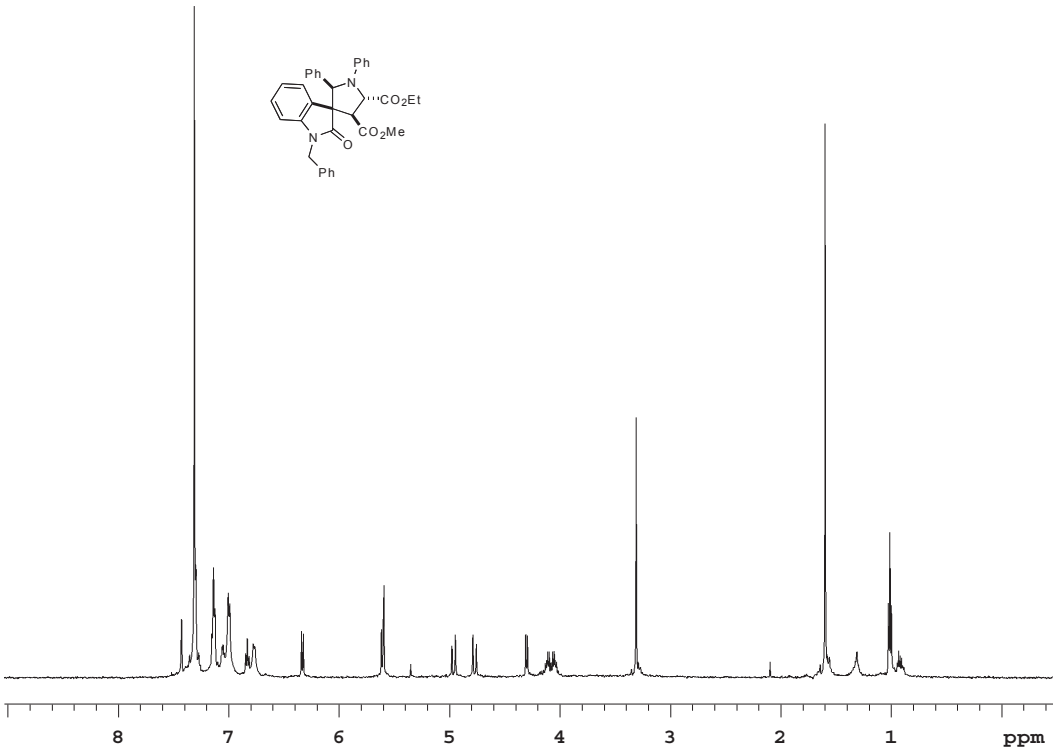

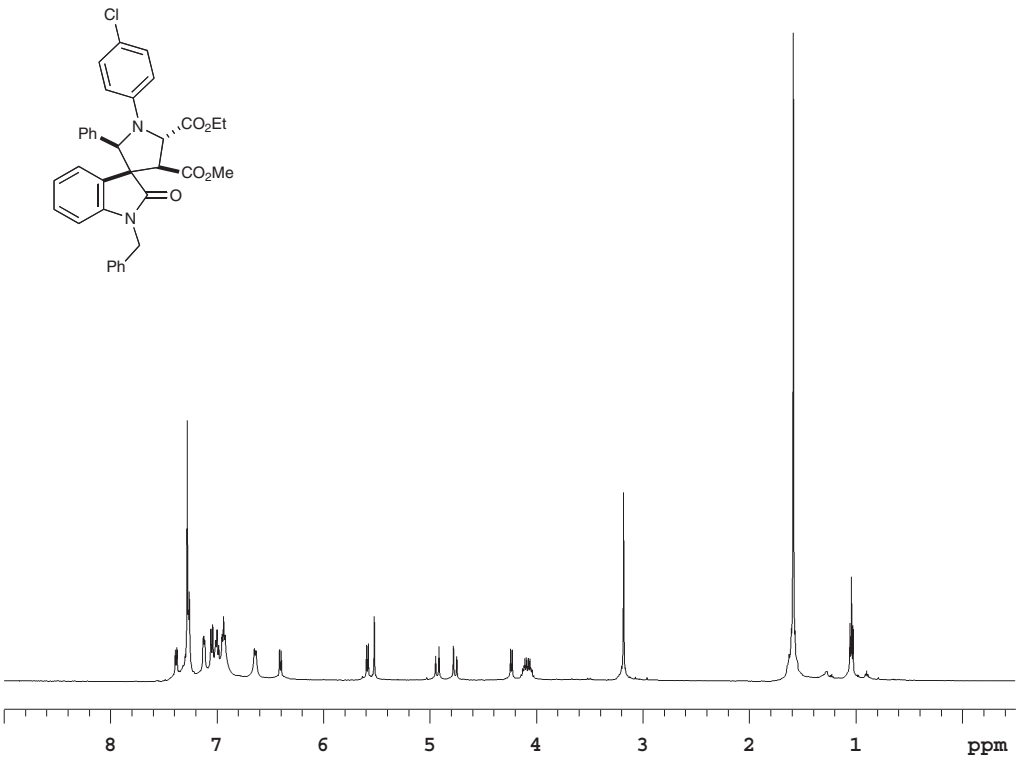

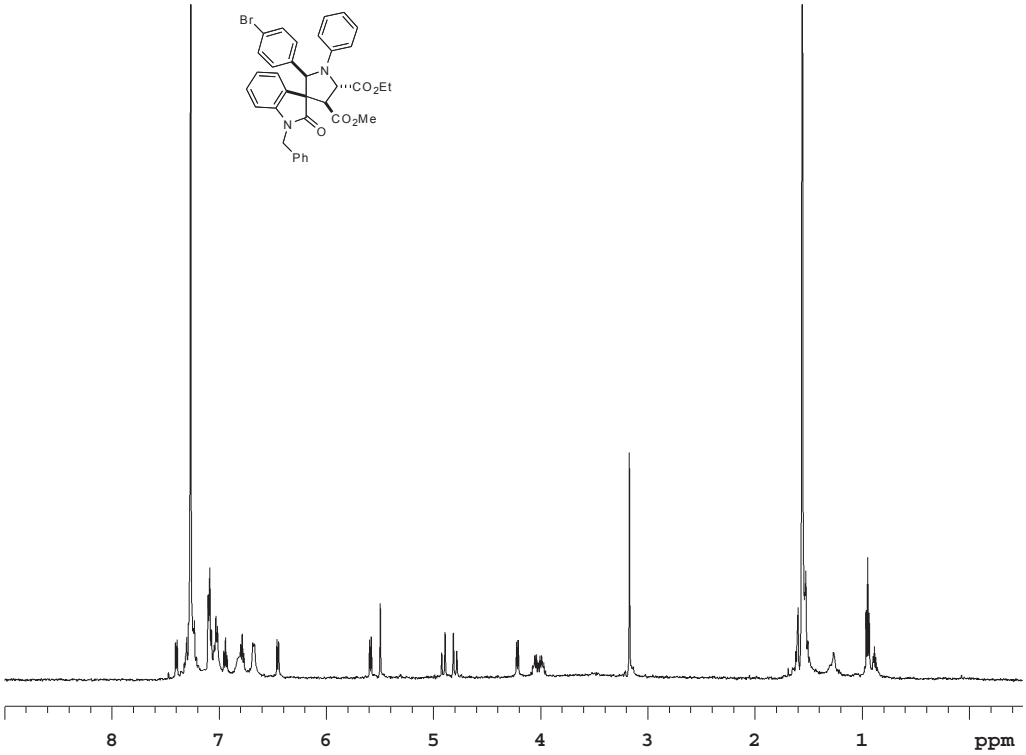
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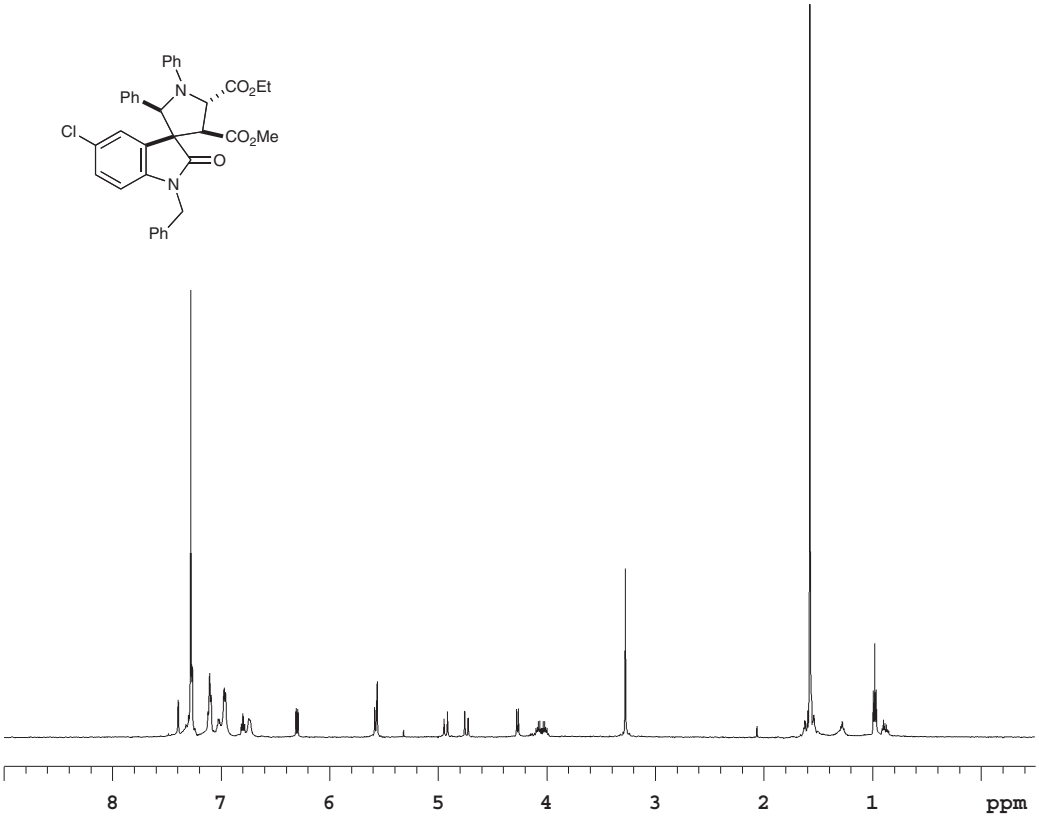
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Supplementary Material (ESI) for Chemical Communications
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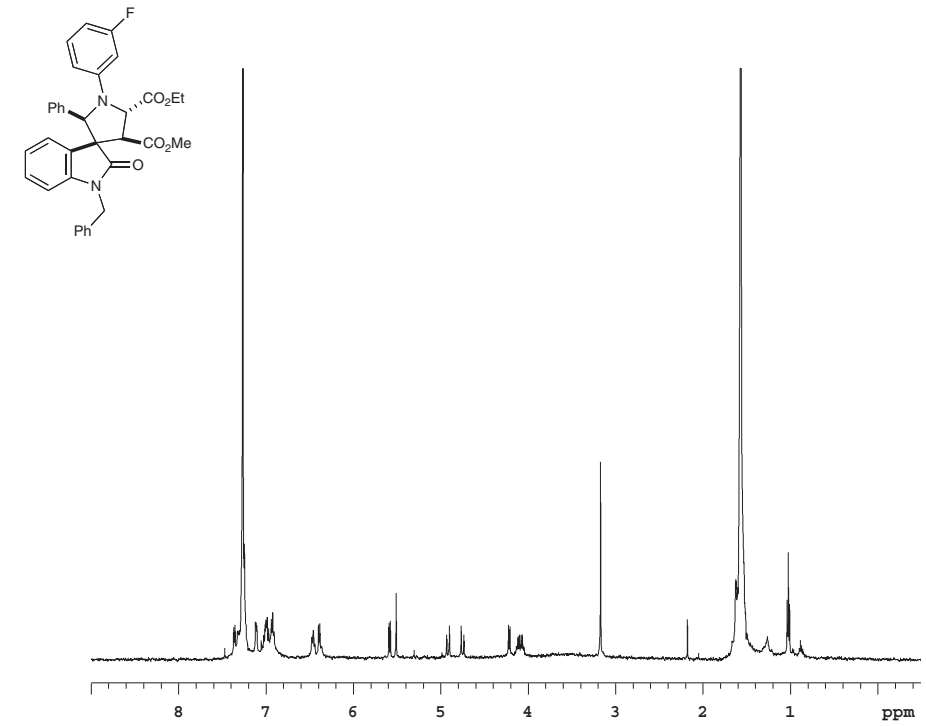

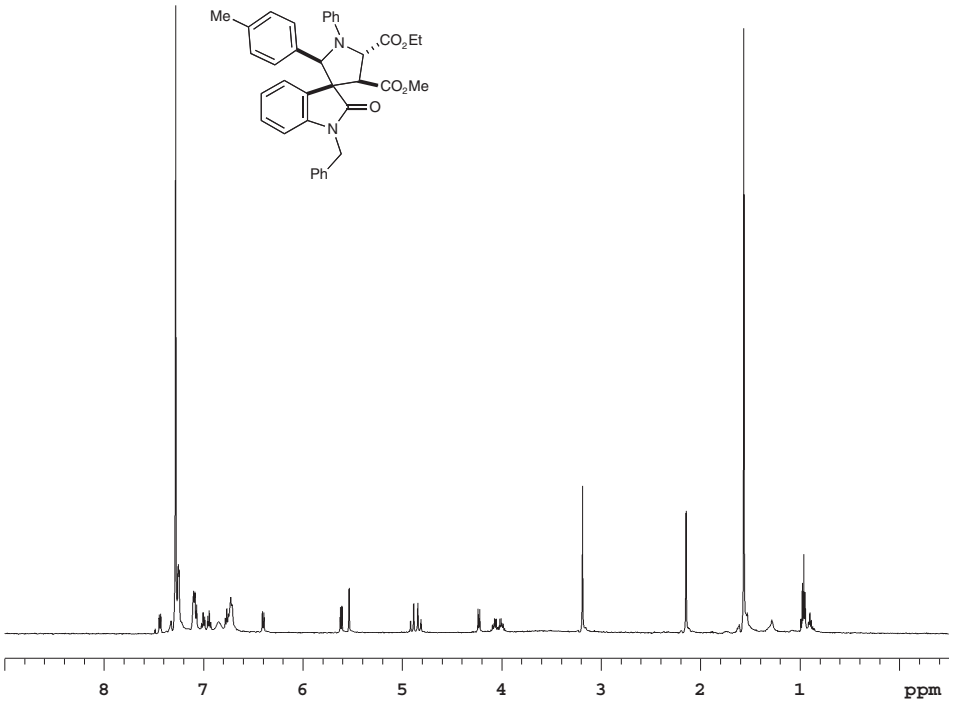

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Crystallographic Data for compound **9**

Data Collection

A colorless plate crystal of $C_{35}H_{31}ClN_2O_5$ having approximate dimensions of 0.348 x 0.220 x 0.010 mm was mounted using oil (Infineum V8512) on a glass fiber. All measurements were made on a CCD area detector with graphite monochromated MoK\(radiation.

Cell constants and an orientation matrix for data collection corresponded to a Monoclinic cell with dimensions:

a = 13.1699(14) Å
b = 16.1972(17) Å ® = 103.160(2)°
c = 14.1817(14) Å
V = 2945.7(5) Å3

For Z = 4 and F.W. = 595.07, the calculated density is 1.342 g/cm^3. Based on systematic absences, and the successful solution and refinement of the structure, the space group was determined to be:

P2(1)/c

The data were collected at a temperature of 153(2)K with a theta range for data collection of 1.59 to 28.90°. Data were collected in 0.3° oscillations with 25 second exposures. The crystal-to-detector distance was 50.00 mm with the detector at the 28° swing position.

Data Reduction

Of the 26840 reflections which were collected, 7120 were unique (Rint = 0.1034). Data were collected using Bruker SMART detector and processed using SAINT from Bruker.

The linear absorption coefficient, mu, for MoK\a radiation is 0.177 mm-1. An analytical absorption correction was applied. Minimum and maximum transmission factors were: 0.9560 and 0.9982, respectively. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods1 and expanded using Fourier techniques2. The non-hydrogen atoms were refined anisotropically.
Hydrogen atoms were included in idealized positions, but not refined. The final cycle of full-matrix least-squares refinement3 on F2 was based on 7120
reflections and 390 variable parameters and converged (largest parameter shift was 0.001 times its esd) with unweighted and weighted agreement factors of:

R1 = ©| |F_o|-|F_c| |/©|F_o| = 0.0524

$wR^2 = \{\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2} = 0.1000$

The weighting scheme was calc.
calc $w=1/[\sigma^2(F_o^2)+(0.0513P)^2 + 0.0000P]$ where $P=(F_o^2+2F_c^2)/3$

The standard deviation of an observation of unit weight 4 was 0.929. The weighting scheme was based on counting statistics and included a factor to downweight the intense reflections. Plots of $\sum w (|F_o| - |F_c|)^2$ versus $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.321 and -0.256 e-/Å3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber5. Anomalous dispersion effects were included in Fcalc6; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley7. The values for the mass attenuation coefficients are those of Creagh and Hubbell8. All calculations were performed using the Bruker SHELXTL9 crystallographic software package.

References

(1) Bruker SMART, Version 5.054 Bruker Analytical X-ray Instruments, Inc.: Madison, WI, 2000

(2) Saint-Plus, version 6.02A; Bruker Analytical X-ray Instruments, Inc.: Madison, WI, 2000.

(3) Sheldrick, G.M. SHELXTL Version 6.14; Bruker Analytical X-ray Instruments, Inc.: Madison, WI, 2003

(4) Full-Matrix Least-Squares refinement on F^2:

$$wR^2 = \{\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$$

(5) GooF = $S = \{\sum[w(F_o^2-F_c^2)^2]/(n-p)\}^{1/2}$ n = number of reflections; p = total number of reflections refined

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallograph
Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography",
Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8,
pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography",

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