Single-Step Synthesis of Pyridine Derivatives

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Supporting Information

General Procedures. All reactions were performed in oven-dried or flame-dried round bottomed flasks, modified Schlenk (Kjeldahl shape) flasks, or glass pressure vessels. The flasks were fitted with rubber septa and reactions were conducted under a positive pressure of argon. Stainless steel syringes or cannulae were used to transfer air- and moisture-sensitive liquids. Flash column chromatography was performed as described by Still et al. using silica gel (60-Å pore size, 32–63 µm, standard grade, Sorbent Technologies) or non-activated alumina gel (80–325 mesh, chromatographic grade, EM Science).¹ Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel or neutral alumina gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light and/or by exposure to an ethanolic phosphomolybdic acid (PMA), an acidic solution of *p*-anisaldehyde (anis), an aqueous solution of ceric ammonium molybdate (CAM), an aqueous solution of potassium permanganate (KMnO₄) or an ethanolic solution of ninhydrin followed by heating (<1 min) on a hot plate (~250 °C). Organic solutions were concentrated on Büchi R-200 rotary evaporators at ~10 Torr (house vacuum) at 25–35 °C, then at ~0.5 Torr (vacuum pump) unless otherwise indicated.

Materials. Commercial reagents and solvents were used as received with the following exceptions: Dichloromethane, diethyl ether, tetrahydrofuran, acetonitrile, and toluene were purchased from J.T. Baker (CycletainerTM) and were purified by the method of Grubbs et al. under positive argon pressure.² 2-chloropyridine was distilled from calcium hydride and stored sealed under an argon atmosphere. The starting amides were prepared by acylation of the corresponding anilines³ or via

¹ Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923–2925.

² Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.

³ For a general procedure, see: DeRuiter, J.; Swearingen, B. E.; Wandrekar, V.; Mayfield, C. A. *J. Med. Chem.* **1989**, *32*, 1033–1038.

previously reported copper-catalyzed C–N bond-forming reactions.^{4,5} Ethoxy acetylene (**2a**) was purchased from Aldrich as a solution in hexanes and purified by kugelrohr distillation before use (% wt. in hexanes determined by ¹H NMR analysis, ~47% wt.). Silyloxy acetylenes **2b** and **2c** were prepared according to Sun, J.; Kozmin, S. A. *Angew. Chem. Int. Ed.* **2006**, *45*, 4991–4993. Ynamide **2d** was prepared according to Buissonneaud, D.; Cintrat, J.-C. *Tetrahedron Lett.* **2006**, *47*, 3139–3143. Silyl enol ether **3b** was prepared according to Schaumann, E.; Tries, F. *Synthesis* **2002**, 191–194.

Instrumentation. All reaction conducted at 140 °C were performed in a CEM Discover Lab Mate microwave reactor. Proton nuclear magnetic resonance (¹H NMR) spectra were recorded with a Varian inverse probe 500 INOVA spectrometer or a Bruker 400 AVANCE spectrometer. Chemical shifts are recorded in parts per million from internal tetramethylsilane on the δ scale and are referenced from the residual protium in the NMR solvent (CHCl₃: δ 7.27, C₆HD₅: δ 7.16). Data is reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, q = quartet, m = multiplet), integration, coupling constant(s) in Hertz, assignment]. Carbon-13 nuclear magnetic resonance spectra were recorded with a Varian 500 INOVA spectrometer or a Bruker 400 AVANCE spectrometer and are recorded in parts per million from internal tetramethylsilane on the δ scale and are referenced from the carbon resonances of the solvent (CDCl₃: δ 77.2, benzene-*d*₆: δ 128.0). Data is reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, q = quartet, m = multiplet), coupling constant(s) in Hertz, assignment]. Infrared data were obtained with a Perkin-Elmer 2000 FTIR and are reported as follows: [frequency of absorption (cm⁻¹), intensity of absorption (s = strong, m = medium, w = weak, br = broad), assignment]. Chiral HPLC analysis was performed on an Agilent 1100 Series HPLC with a Whelk-O1 (*S,S*) column. We thank Dr. Li Li at the Massachusetts Institute of Technology Department of Chemistry instrumentation facility for obtaining mass spectroscopic data.

⁴ For the general procedure used for the synthesis of all *N*-vinyl amides, see: Jiang, L.; Job, G. E.; Klapars, A.; Buchwald, S. L. *Org. Lett.* **2003**, *5*, 3667–3669.

⁵ For related reports, see: (a) Wolfe, J. P.; Wagaw, S.; Marcoux, J.-F.; Buchwald, S. L. *Acc. Chem. Res.* **1998**, *31*, 805–818. (b) Hartwig, J. F. *Acc. Chem. Res.* **1998**, *31*, 852–860. (c) Muci, A. R.; Buchwald, S. L. *Top. Curr. Chem.* **2002**, *219*, 131–209. (d) Beletskaya, I. P.; Cheprakov, A. V. *Coordin. Chem. Rev.* **2004**, *248*, 2337–2364. (e) Dehli, J. R.; Legros, J.; Bolm, C. *Chem. Commun.* **2005**, 973–986.

6-Cyclohexyl-4-ethoxy-2,3-dimethylpyridine (4a, Table 1):

Trifluoromethanesulfonic anhydride (50 μL , 0.30 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1b** (50 mg, 0.28 mmol, 1 equiv) and 2-chloropyridine (52 μL , 0.55 mmol, 2.0 equiv) in dichloromethane (900 μL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ethyl ethynyl ether (**2a**, 82 mg, 0.55 mmol, 2.0 equiv, 47% wt. in hexane) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 20% EtOAc/1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the pyridine derivative **4a** as a pale yellow solid (55 mg, 85%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 6.48 (s, 1H, ArH), 4.06 (q, 2H, $J = 7.0$ Hz, OCH_2CH_3), 2.66–2.58 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 2.46 (s, 3H, CH_3), 2.10 (s, 3H, CH_3), 1.98–1.93 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.86–1.80 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.77–1.71 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.48–1.38 (m, 7H, $^6\text{C}_6\text{H}_{11}$, OCH_2CH_3), 1.32–1.24 (m, 1H, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 164.7, 163.4, 156.5, 117.2, 101.0, 63.5, 47.0, 33.5, 26.8, 26.3, 22.8, 14.8, 10.9.

FTIR (neat) cm^{-1} : 3231 (w), 3064 (w), 2955 (s), 2865 (s), 1726 (w), 1634 (m), 1594 (w), 1535 (s), 1498 (s), 1475 (s), 1240 (m).

HRMS (ESI): calcd for $\text{C}_{15}\text{H}_{24}\text{NO}$ $[\text{M}+\text{H}]^+$: 234.1852, found: 234.1844.

TLC (20% EtOAc/hexanes), R_f : 0.27 (UV, KMnO_4).

2-Cyclohexyl-4-ethoxy-5,7-dimethoxyquinoline (4b**, Table 1):**

Trifluoromethanesulfonic anhydride (83 μL , 0.50 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (120 mg, 0.456 mmol, 1 equiv) and 2-chloropyridine (52 μL , 0.55 mmol, 1.2 equiv) in dichloromethane (1.5 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , ethyl ethynyl ether (**2a**, 128 mg, 0.91 mmol, 2.0 equiv, 50% wt. in hexane) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 30% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline derivative **4b** as a white solid (110 mg, 75%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 6.97 (br s, 1H, ArH), 6.51 (s, 1H, ArH), 6.44 (d, 1H, $J = 2.3$ Hz, ArH), 4.22 (q, 2H, $J = 7.0$ Hz, OCH_2CH_3), 3.93 (s, 3H, OCH_3), 3.91 (s, 3H, OCH_3), 2.82–2.72 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 2.04–1.99 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.90–1.85 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.80–1.74 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.60–1.51 (m, 5H, $^6\text{C}_6\text{H}_{11}$, OCH_2CH_3), 1.50–1.41 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.32 (tt, 1H, $J = 12.7, 3.5$ Hz, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, $\text{DMF}-d_7$, 20°C) δ : 168.6, 164.0, 161.0, 158.2, 152.8, 107.6, 100.2, 98.2, 98.2, 64.4, 56.3, 55.7, 47.9, 33.1, 26.7, 26.3, 14.7.

FTIR (neat) cm^{-1} : 2929 (m), 2851 (w), 1725 (w), 1615 (s), 1591 (s), 1451 (m), 1404 (m), 1382 (m), 1206 (m), 1155 (s).

HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_3$ $[\text{M}+\text{H}]^+$: 316.1907, found: 316.1904.

TLC (40% EtOAc/hexanes), R_f : 0.54 (UV, KMnO_4).

nOe data:

7-Butyl-6-phenyl-8-(triisopropylsilyloxy)-3,4-dihydro-2H-pyrano[3,2-*b*]pyridine (4c, Table 1):

Trifluoromethanesulfonic anhydride (72 μ L, 0.43 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1d** (80 mg, 0.39 mmol, 1 equiv) and 2-chloropyridine (75 μ L, 0.79 mmol, 2.0 equiv) in dichloromethane (1.3 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynol **2b**⁶ (200 mg, 0.788 mmol, 2.00 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 10 min, triethylamine (500 μ L) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 20% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the pyridine derivative **4c** as a clear oil (110 mg, 63%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 7.40–7.30 (m, 5H, ArH), 4.18 (t, 2H, J = 5.1 Hz, CH₂CH₂CH₂O), 2.95 (t, 2H, J = 6.6 Hz, CH₂CH₂CH₂O), 2.56–2.50 (m, 2H, CH₂CH₂CH₂CH₃), 2.15–2.09 (m, 2H, CH₂CH₂CH₂O), 1.45–1.38 (m, 2H, CH₂CH₂CH₂CH₃), 1.35–1.27 (m, 3H, Si(CH(CH₃)₂)₃), 1.25–1.15 (m, 2H, CH₂CH₂CH₂CH₃), 1.11 (d, 18H, J = 7.4 Hz, Si(CH(CH₃)₂)₃), 0.76 (t, 3H, J = 7.3 Hz, CH₂CH₂CH₂CH₃).

¹³C NMR (100 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 152.4, 149.9, 141.6, 141.0, 140.5, 129.1, 128.1, 127.3, 125.9, 65.8, 32.3, 28.3, 27.2, 23.0, 22.7, 18.3, 14.6, 13.9.

FTIR (neat) cm⁻¹: 2981 (w), 2927 (s), 2852 (m), 1731 (w), 1588 (s), 1575 (s), 1468 (m), 1328 (m), 1217 (m), 1123 (s).

HRMS (ESI): calcd for C₁₈H₂₂NO₂ [M–Si(^{*i*}Pr)₃+2H]⁺: 284.1645, found: 284.1644.

TLC (20% EtOAc/hexanes), R_f : 0.21 (UV, KMnO₄).

⁶ For preparation of **2b** and **2c**, see Sun, J.; Kozmin, S. A. *Angew. Chem. Int. Ed.* **2006**, 45, 4991–4993

2-sec-Butyl-3-butyl-4-(triisopropylsilyloxy)-5,6,7,8-tetrahydroquinoline (4d, Table 1):

Trifluoromethanesulfonic anhydride (80 μL , 0.49 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1e** (80 mg, 0.44 mmol, 1 equiv) and 2-chloropyridine (84 μL , 0.88 mmol, 2.0 equiv) in dichloromethane (1.5 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynone **2b** (123 mg, 0.485 mmol, 1.10 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 10 min, triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 3% EtOAc and 0.5% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the pyridine derivative **4d** as a clear oil (134 mg, 73%).

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 2.92–2.79 (m, 3H), 2.70–2.50 (m, 4H), 1.85–1.70 (m, 5H), 1.62–1.53 (m, 1H), 1.43–1.30 (m, 7H), 1.21 (d, 3H, $J = 6.8$ Hz, CH₃CH₂CHCH₃), 1.10 (d, 18H, $J = 7.5$ Hz, Si(CH(CH₃)₂)₃), 0.95–0.92 (m, 3H), 0.79 (t, 3H, $J = 7.4$ Hz, CH₃CH₂CHCH₃).

¹³C NMR (125 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 162.3, 159.8, 155.3, 123.7, 120.4, 38.0, 33.5, 33.0, 30.1, 26.4, 24.4, 23.4, 23.3, 22.9, 21.0, 18.2, 14.6, 14.3, 12.7.

FTIR (neat) cm⁻¹: 3257 (w), 3090 (w), 2958 (s), 2933 (s), 2869 (s), 1631 (w), 1612 (w), 1502 (s), 1464 (m), 1427 (m), 1210 (w).

HRMS (ESI): calcd for C₂₆H₄₈NOSi [M+H]⁺: 418.3500, found: 418.3510.

TLC (20% EtOAc/hexanes), R_f: 0.76 (UV, KMnO₄).

2-Cyclohexyl-5,7-dimethoxy-3-phenyl-4-(triisopropylsilyloxy)quinoline (4e, Table 1):

Trifluoromethanesulfonic anhydride (69 μ L, 0.42 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (100 mg, 0.380 mmol, 1 equiv) and 2-chloropyridine (43 μ L, 0.46 mmol, 1.2 equiv) in dichloromethane (1.3 mL) at -78 $^{\circ}$ C. After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0 $^{\circ}$ C, the ynone **2c**⁶ (115 mg, 0.418 mmol, 1.10 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μ L) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 4% EtOAc and 0.5% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the quinoline derivative **4e** as a clear oil (154 mg, 78%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}$ C) δ : 7.41–7.30 (m, 5H, ArH), 6.97 (d, 1H, J = 2.4 Hz, ArH), 6.41 (d, 1H, J = 2.4 Hz, ArH), 3.95 (s, 3H, OCH₃), 3.82 (s, 3H, OCH₃), 2.58 (tt, 1H, J = 11.1, 3.5 Hz, ^cC₆H₁₁), 1.75–1.55 (m, 7H, ^cC₆H₁₁), 1.28 (qt, 1H, J = 13.0, 3.4 Hz, ^cC₆H₁₁), 1.12–1.00 (m, 2H, ^cC₆H₁₁), 0.83 (d, 18H, J = 7.2 Hz, Si(CH(CH₃)₂)₃), 0.75–0.67 (m, 3H, Si(CH(CH₃)₂)₃).

¹³C NMR (100 MHz, CDCl₃, 20 $^{\circ}$ C) δ : 166.7, 160.2, 157.9, 157.5, 152.6, 137.3, 132.8, 128.2, 127.2, 123.9, 110.4, 100.3, 97.5, 55.6, 55.0, 42.8, 32.3, 26.6, 26.1, 18.1, 14.2.

FTIR (neat) cm⁻¹: 3057 (w), 2942 (s), 2865 (s), 1619 (s), 1567 (s), 1465 (m), 1451 (m), 1375 (s), 1358 (s), 1250 (m), 1207 (s).

HRMS (ESI): calcd for C₃₂H₄₆NO₃Si [M+H]⁺: 520.3241, found: 520.3245.

TLC (10% EtOAc/hexanes), R_f: 0.32 (UV, KMnO₄).

3-(2-Cyclohexyl-5,6-dimethyl-3-phenyl-pyridin-4-yl)-oxazolidin-2-one (4f, Table 1):

Trifluoromethanesulfonic anhydride (52 μL , 0.31 mmol, 1.1 equiv) was added via syringe drop-wise to a stirred mixture of amide **1b** (51 mg, 0.28 mmol, 1 equiv) and 2-chloropyridine (54 μL , 0.57 mmol, 2.0 equiv) in dichloromethane (700 μL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , and a solution of ynamide **2d**⁷ (59 mg, 0.32 mmol, 1.1 equiv) in dichloromethane (250 μL) was added via cannula. After 20 minutes, aqueous sodium hydroxide solution (1.0 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between dichloromethane (10 mL) and water (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane (2×10 mL) and the combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 50% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 2 cm) to give the pyridine derivative **4f** as a white solid (80 mg, 81%).

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 7.46–7.38 (m, 3H, ArH), 7.31–7.27 (m, 1H, ArH), 7.20–7.17 (m, 1H, ArH), 4.24 (td, 1H, $J = 8.8, 5.6$ Hz, NCH₂CH₂O), 3.79 (td, 1H, $J = 8.4, 8.0$ Hz, NCH₂CH₂O), 3.52 (app. q, 1H, $J = 8.4$ Hz, NCH₂CH₂O), 3.08 (td, 1H, $J = 9.0, 5.6$ Hz, NCH₂CH₂O), 2.57 (s, 3H, ArCH₃), 2.48 (tt, 1H, $J = 11.4, 3.4$ Hz, ^cC₆H₁₁), 2.19 (s, 3H, ArCH₃), 1.85–1.70 (m, 3H, ^cC₆H₁₁), 1.65–1.54 (m, 3H, ^cC₆H₁₁), 1.47–1.44 (m, 1H, ^cC₆H₁₁), 1.29–1.12 (m, 2H, ^cC₆H₁₁), 1.00 (qt, 1H, $J = 12.4, 3.2$ Hz, ^cC₆H₁₁).

¹³C NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 161.9, 157.9, 156.7, 141.1, 136.4, 132.1, 129.7, 129.0, 128.8, 128.1, 127.9, 126.9, 62.8, 46.2, 42.1, 32.5, 32.4, 25.9, 23.6, 13.8

FTIR (neat) cm⁻¹: 2925 (s), 2852 (m), 1752 (s), 1643 (m), 1448 (m).

HRMS (ESI): calcd for C₂₂H₂₇N₂O₂ [M+H]⁺: 351.2067, found: 351.2054.

TLC (50% EtOAc/1% Et₃N/hexanes), R_f: 0.25 (UV, CAM).

⁷ For preparation of **2d** see Buissonneaud, D.; Cintrat, J.-C. *Tetrahedron Lett.* **2006**, 47, 3139–3143.

3-(2-sec-Butyl-3-phenyl-5,6,7,8-tetrahydroquinolin-4-yl)oxazolidin-2-one (4g, Table 1):

Trifluoromethanesulfonic anhydride (80 μ L, 0.49 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1e** (80 mg, 0.44 mmol, 1 equiv) and 2-chloropyridine (84 μ L, 0.88 mmol, 2.0 equiv) in dichloromethane (1.5 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamide **2d**⁷ (91 mg, 0.49 mmol, 1.1 equiv) was added as a solid in one portion and the reaction flask was rapidly purged and sealed under an argon atmosphere. After 1 h, triethylamine (500 μ L) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 30% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the pyridine derivative **4g** as a white solid (126 mg, 81%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$, minor atropisomer noted by *) δ : 7.48–7.37 (m, 3H, ArH; 3H, ArH*), 7.33–7.28 (m, 1H, ArH; 1H, ArH*), 7.20–7.16 (m, 1H, ArH; 1H, ArH*), 4.28–4.21 (m, 1H, NCH₂CH₂O; 1H, NCH₂CH₂O*), 3.86–3.80 (m, 1H, NCH₂CH₂O; 1H, NCH₂CH₂O*), 3.59–3.48 (m, 1H, NCH₂CH₂O; 1H, NCH₂CH₂O*), 3.14–3.03 (m, 1H, NCH₂CH₂O; 1H, NCH₂CH₂O*), 3.03–2.80 (m, 3H, CH₂CH₂CH₂CH₂; 3H, CH₂CH₂CH₂CH₂*), 2.69–2.60 (m, 1H, CH₃CH₂CHCH₃; 1H, CH₃CH₂CHCH₃*), 2.59–2.51 (m, 1H, CH₂CH₂CH₂CH₂; 1H, CH₂CH₂CH₂CH₂*), 1.97–1.62 (m, 5H, CH₂CH₂CH₂CH₂, CH₃CH₂CHCH₃; 5H, CH₂CH₂CH₂CH₂*, CH₃CH₂CHCH₃*), 1.60–1.37 (m, 1H, CH₃CH₂CHCH₃; 1H, CH₃CH₂CHCH₃*), 1.23 (d, 3H, J = 6.7 Hz, CH₃CH₂CHCH₃), 0.99 (d, 3H, J = 6.8 Hz, CH₃CH₂CHCH₃*), 0.82 (t, 3H, J = 7.4 Hz, CH₃CH₂CHCH₃*), 0.58 (t, 3H, J = 7.3 Hz, CH₃CH₂CHCH₃).

¹³C NMR (125 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 162.4, 162.2, 158.5, 158.5, 156.5, 156.5, 141.0, 140.9, 136.5, 136.4, 132.8, 132.7, 130.1, 129.9, 129.1, 129.1, 129.0, 129.0, 128.2, 128.1, 127.9, 127.7, 62.9, 46.1, 45.9, 39.7, 38.8, 38.8, 33.1, 29.9, 29.1, 24.4, 22.9, 22.9, 22.5, 20.9, 20.9, 12.7, 12.3.

FTIR (neat) cm⁻¹: 2959 (m), 2933 (m), 1753 (s), 1603 (w), 1575 (w), 1481 (w), 1440 (m), 1409 (m), 1230 (m), 1181 (w).

HRMS (ESI): calcd for C₂₂H₂₇N₂O₂ [M+H]⁺: 351.2067, found: 351.2057.

TLC (30% EtOAc/hexanes), R_f: 0.29 (UV, KMnO₄).

3-(2-Morpholino-3-phenylquinolin-4-yl)oxazolidin-2-one (4h**, Table 1):**

Trifluoromethanesulfonic anhydride (40 μL , 0.24 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of urea **1f** (46 mg, 0.22 mmol, 1 equiv) and 2-chloropyridine (25 μL , 0.27 mmol, 1.2 equiv) in dichloromethane (750 μL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamide **2d** (83 mg, 0.44 mmol, 1.1 equiv) was added as a solid in one portion and the reaction flask was rapidly purged and sealed under an argon atmosphere. The resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a microwave reactor and heated to 140°C . After 20 min, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature before triethylamine (300 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 40% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline derivative **4h** as a white solid (58 mg, 70%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ :	7.93–7.90 (m, 1H, ArH), 7.73–7.69 (m, 1H, ArH), 7.66 (qd, 1H, $J = 6.9, 1.5$ Hz, ArH), 7.63–7.58 (m, 1H, ArH), 7.55–7.47 (m, 3H, ArH), 7.45–7.41 (m, 2H, ArH), 4.46–4.40 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$), 4.08–4.02 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$), 3.64–3.52 (m, 5H, $\text{NCH}_2\text{CH}_2\text{O}$, $\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$), 3.22–3.13 (m, 4H, $\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$), 3.04–2.99 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$).
^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ :	159.6, 157.8, 147.8, 140.4, 135.6, 130.3, 129.6, 129.5, 128.8, 128.7, 128.6, 128.3, 128.2, 125.3, 122.5, 122.2, 66.7, 63.2, 49.7, 46.1.
FTIR (neat) cm^{-1} :	3490 (w), 3060 (m), 2965 (m), 2893 (m), 2849 (m), 2248 (w), 1755 (s), 1587 (s), 1491 (s), 1409 (s), 1238 (s), 1117 (s).
HRMS (ESI):	calcd for $\text{C}_{22}\text{H}_{22}\text{N}_3\text{O}_3$ $[\text{M}+\text{H}]^+$: 376.1656, found: 376.1668.
TLC (40% EtOAc/hexanes), R_f :	0.28 (UV, KMnO_4).

3-(2-Cyclohexyl-5,7-dimethoxy-3-phenylquinolin-4-yl)oxazolidin-2-one (4i**, Table 1):**

Trifluoromethanesulfonic anhydride (69 μL , 0.42 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (100 mg, 0.380 mmol, 1 equiv) and 2-chloropyridine (43 μL , 0.46 mmol, 1.2 equiv) in dichloromethane (1.3 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamide **2d** (78 mg, 0.42 mmol, 1.1 equiv) was added as a solid in one portion, and the reaction flask was rapidly purged and sealed under an argon atmosphere. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 40% EtOAc in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline derivative **4i** as a white solid (140 mg, 85%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 7.49–7.40 (m, 4H, ArH), 7.23–7.19 (m, 1H, ArH), 7.11 (d, 1H, $J = 2.3$ Hz, ArH), 6.57 (d, 1H, $J = 2.3$ Hz, ArH), 4.33–4.28 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$), 3.97 (s, 3H, OCH_3), 3.96–3.89 (m, 4H, OCH_3 , $\text{NCH}_2\text{CH}_2\text{O}$), 3.76–3.70 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$), 3.24–3.18 (m, 1H, $\text{NCH}_2\text{CH}_2\text{O}$), 2.67–2.60 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.91–1.74 (m, 3H, $^6\text{C}_6\text{H}_{11}$), 1.63–1.60 (m, 2H, $^6\text{C}_6\text{H}_{11}$), 1.35–1.10 (m, 4H, $^6\text{C}_6\text{H}_{11}$), 1.09–0.99 (m, 1H, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 166.4, 160.9, 157.8, 156.0, 152.3, 138.5, 136.3, 133.4, 130.3, 128.8, 128.7, 127.9, 127.8, 111.4, 101.2, 99.9, 62.9, 56.6, 55.8, 47.8, 43.2, 32.5, 32.2, 26.5, 26.5, 26.0.

FTIR (neat) cm^{-1} : 2924 (m), 2850 (w), 1745 (s), 1617 (s), 1573 (s), 1479 (w), 1446 (w), 1423 (m), 1407 (m), 1249 (m).

HRMS (ESI): calcd for $\text{C}_{26}\text{H}_{29}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: 433.2122, found: 433.2107.

TLC (40% EtOAc/hexanes), R_f : 0.31 (UV, KMnO_4).

Methyl 2-cyclohexyl-4-(2-oxooxazolidin-3-yl)-3-phenylquinoline-6-carboxylate (4j, Table 1):

Trifluoromethanesulfonic anhydride (36 μL , 0.21 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1g** (50 mg, 0.19 mmol, 1 equiv) and 2-chloropyridine (36 μL , 0.38, 2.0 equiv) in dichloromethane (540 μL) at -78°C . After five minutes, the reaction mixture was allowed to warm to 0°C for five minutes, cooled back to -78°C , and, after five minutes, a solution of ynamide **2d** (72 mg, 0.38 mmol, 2.0 equiv) in dichloromethane (100 μL) was added via cannula. After an additional five minutes, the reaction mixture was allowed to warm back to 0°C . After 20 minutes, trifluoroacetic acid (150 μL) was added to the reaction mixture to remove excess ynamide. After 15 minutes, saturated aqueous sodium bicarbonate solution (7 mL) was added to quench excess acid. The mixture was diluted with water (10 mL) and extracted with dichloromethane (3×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 50% EtOAc and 2% Et_3N in hexanes; SiO_2 : 15×3 cm) to give the quinoline derivative **4j** as a white solid (69 mg, 84%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 8.53 (d, 1H, $J = 1.5$ Hz, ArH), 8.33 (dd, 1H, $J = 9.0, 1.5$ Hz ArH), 8.20 (d, 1H, $J = 9.0$ Hz, ArH), 7.56–7.46 (m, 4H, ArH), 7.26–7.25 (m, 1H, ArH), 4.49 (td, 1H, $J = 9.0, 6.5$ Hz, $\text{NCH}_2\text{CH}_2\text{O}$), 4.06–3.98 (m, 4H, $\text{NCH}_2\text{CH}_2\text{O}$, OCH_3), 3.81 (td, 1H, $J = 9.0, 7.0$ Hz, $\text{OCH}_2\text{CH}_2\text{N}$), 3.30 (td, 1H, $J = 9.0, 6.5$ Hz, $\text{OCH}_2\text{CH}_2\text{N}$), 2.79–2.73 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.98–1.81 (m, 3H, $^6\text{C}_6\text{H}_{11}$), 1.72–1.61 (m, 4H, $^6\text{C}_6\text{H}_{11}$), 1.35–1.27 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.22–1.14 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.09–1.03 (m, 1H, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 168.9, 166.8, 157.2, 150.8, 140.4, 135.5, 135.2, 130.4, 129.8, 129.5, 128.7, 128.6, 128.4, 125.6, 123.5, 63.2, 52.7, 47.2, 43.6, 32.7, 32.3, 26.5, 26.4, 26.0

FTIR (neat) cm^{-1} : 2928 (m), 2853 (w), 1758 (s), 1720 (s), 1451 (m), 1413 (m), 1259 (s).

HRMS (ESI): calcd for $\text{C}_{26}\text{H}_{27}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: 431.1965, found: 431.1958.

TLC (8% EtOAc/2% Et_3N /DCM), R_f : 0.26 (UV, CAM).

X-ray Structure of **4j**: See page S37.

4-(2-Cyclohexyl-5,6-dimethyl-3-(trimethylsilyl)pyridin-4-yl)morpholine (4k, Table 1):

Trifluoromethanesulfonic anhydride (66 μL , 0.39 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1b** (65 mg, 0.38 mmol, 1 equiv) and 2-chloropyridine (68 μL , 0.72, 2.0 equiv) in dichloromethane (1.0 mL) at $-78\text{ }^{\circ}\text{C}$. After five minutes, the reaction mixture was placed in an ice-water bath, allowed to warm to $0\text{ }^{\circ}\text{C}$, and a solution of ynamine **2e** (143 μL , 0.72 mmol, 2.00 equiv) in dichloromethane (200 μL) was added via cannula. After 20 minutes, triethylamine (0.5 mL) was added to the reaction mixture to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane ($2 \times 10\text{ mL}$). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et₃N in hexanes; SiO₂: $12 \times 3\text{ cm}$) to give the pyridine derivative **4k** as an off-white solid (106 mg, 85%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 3.84 (t, 4H, $J = 4.5\text{ Hz}$, N(CH₂CH₂)₂O), 3.16 (br s, 4H, O(CH₂CH₂)₂N), 2.93 (tt, 1H, $J = 11.5, 3.5\text{ Hz}$, ^cC₆H₁₁), 2.46 (s, 3H, CH₃), 2.30 (s, 3H, CH₃), 1.82–1.71 (m, 7H, ^cC₆H₁₁), 1.36–1.34 (m, 3H, ^cC₆H₁₁), 0.39 (s, 9H, Si(CH₃)₃).

¹³C NMR (125 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 170.1, 162.2, 159.9, 128.7, 126.9, 66.9, 49.7, 44.9, 33.4, 26.8, 26.1, 23.7, 16.1, 4.2.

FTIR (neat) cm⁻¹: 2923 (s), 2854 (m), 1529 (m), 1450 (m), 1367 (m), 1260 (s), 1115 (s).

HRMS (ESI): calcd for C₂₀H₃₅N₂OSi [M+H]⁺: 347.2513, found: 347.2507.

TLC (35% EtOAc/1% Et₃N/hexanes), R_f: 0.42 (UV, CAM).

4-(5-Phenyl-6-(trimethylsilyl)thieno[3,2-*b*]pyridin-7-yl)morpholine (4l**, Table 1):**

Trifluoromethanesulfonic anhydride (89 μL , 0.54 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1h** (100 mg, 0.492 mmol, 1 equiv) and 2-chloropyridine (233 μL , 2.45 mmol, 5.00 equiv) in dichloromethane (1.6 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamine **2e** (270 mg, 1.48 mmol, 3.00 equiv) was added via syringe, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a microwave reactor and heated to 140°C . After 1 min, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature before triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 20% EtOAc was 1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the pyridine derivatives **4l** as a pale yellow oil (138 mg, 76%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 7.74–7.72 (m, 1H, ArH), 7.62–7.60 (m, 1H, ArH), 7.50–7.47 (m, 2H, ArH), 7.45–7.40 (m, 3H, ArH), 3.98–3.95 (m, 4H, $\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$), 3.42 (br s, 4H, $\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$), 0.05 (s, 9H, $\text{Si}(\text{CH}_3)_3$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 181.1, 166.3, 161.8, 158.7, 144.6, 130.6, 129.7, 128.3, 128.3, 125.9, 125.8, 67.0, 51.9, 2.6.

FTIR (neat) cm^{-1} : 2955 (m), 2894 (m), 2856 (m), 1572 (w), 1515 (s), 1471 (s), 1445 (m), 1337 (s), 1259 (s), 1113 (s).

HRMS (ESI): calcd for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{OSSi}$ $[\text{M}+\text{H}]^+$: 369.1451, found: 369.1452.

TLC (30% EtOAc/hexanes), R_f : 0.44 (UV, KMnO_4).

7-(4-Methoxyphenyl)-5-phenylthieno[3,2-*b*]pyridine (4m**, Table 1):**

Trifluoromethanesulfonic anhydride (80 μL , 0.49 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1h** (90 mg, 0.44 mmol, 1 equiv) and 2-chloropyridine (50 μL , 0.53 mmol, 1.2 equiv) in dichloromethane (1.5 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the acetylene **2f** (117 mg, 0.886 mmol, 2.00 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 10% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the pyridine derivative **4m** as a pale yellow oil (90 mg, 64%).

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 8.13–8.09 (m, 2H, ArH), 7.82–7.77 (m, 3H, ArH), 7.73 (s, 1H, ArH), 7.70 (d, 1H, $J = 5.5$ Hz, ArH), 7.55–7.50 (m, 2H, ArH), 7.46 (tt, 1H, $J = 7.4, 1.3$ Hz, ArH), 7.12–7.09 (m, 2H, ArH), 3.92 (s, 3H, OCH₃).

¹³C NMR (100 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 160.6, 157.3, 156.5, 144.7, 140.0, 131.2, 130.9, 130.5, 129.5, 129.0, 129.0, 127.5, 126.1, 115.7, 114.7, 55.6.

FTIR (neat) cm⁻¹: 3061 (w), 2931 (w), 2836 (w), 1996 (w), 1608 (m), 1565 (m), 1513 (s), 1357 (m), 1294 (m), 1257 (s), 1031 (m).

HRMS (ESI): calcd for C₂₀H₁₆NOS [M+H]⁺: 318.0947, found: 318.0944.

TLC (20% EtOAc in hexanes), R_f : 0.37 (U V, KMnO₄).

nOe data:

2-cyclohexyl-4-(4-methoxyphenyl)-3-methyl-6-nitroquinoline (4n, Table 1):

Trifluoromethanesulfonic anhydride (65 μ L, 0.39 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1i** (87 mg, 0.35 mmol, 1 equiv) and 2-chloropyridine (66 μ L, 0.70, 2.0 equiv) in dichloromethane (1.0 mL) at -78 $^{\circ}$ C. After five minutes, the reaction mixture was allowed to warm to 0 $^{\circ}$ C for five minutes, and a solution of alkyne **2g** (56 mg, 0.38 mmol, 1.1 equiv) in dichloromethane (200 μ L) was added via cannula. After 20 minutes, aqueous sodium hydroxide solution (1.0 mL, 1 N) was added to the reaction mixture to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 10% EtOAc and 1% Et₃N in hexanes; SiO₂: 14×3 cm) to give the quinoline **4n** as a white solid (116 mg, 87%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}$ C) δ : 8.36–8.23 (m, 2H, ArH), 8.14 (dd, 1H, $J = 9.0, 0.5$ Hz, ArH), 7.17 (dt, 2H, $J = 9.0, 2.5$ Hz, ArH), 7.10 (dt, 2H, $J = 9.0, 2.5$ Hz, ArH), 3.95 (s, 3H, OCH₃), 3.13 (tt, 1H, $J = 11.0, 3.5$ Hz, ^cC₆H₁₁), 2.29 (s, 3H, ArCH₃), 1.96–1.82 (m, 7H, ^cC₆H₁₁), 1.51–1.41 (m, 3H, ^cC₆H₁₁).

¹³C NMR (100 MHz, CDCl₃, 20 $^{\circ}$ C) δ : 169.9, 159.7, 148.7, 148.3, 145.0, 130.9, 130.7, 129.3, 128.6, 126.2, 123.4, 121.5, 114.5, 55.6, 43.7, 32.0, 26.9, 26.3, 16.6

FTIR (neat) cm⁻¹: 2929 (s), 2853 (m), 1609 (m), 1525 (s), 1483 (s), 1339 (s).

HRMS (ESI): calcd for C₂₃H₂₅N₂O₃ [M+H]⁺: 377.1860, found: 377.1858.

TLC (35% EtOAc/1% Et₃N/hexanes), R_f: 0.55 (UV, CAM).

nOe data :

2-Cyclohexyl-4-(4-methoxy-phenyl)-6-nitro-quinoline (4o, Table 1):

Trifluoromethanesulfonic anhydride (42 μ L, 0.25 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1i** (56 mg, 0.23 mmol, 1 equiv) and 2-chloropyridine (43 μ L, 0.45 mmol, 2.0 equiv) in dichloromethane (760 μ L) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , and acetylene **2f** (33 μ L, 0.25 mmol, 1.1 equiv) was added via syringe. After 20 minutes, aqueous sodium hydroxide solution (0.5 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between dichloromethane (10 mL) and water (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane (2×10 mL) and the combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et₃N in hexanes; SiO₂: 15×2 cm) to give the quinoline derivative **4o** as a yellow solid (34 mg, 42%) and the ynone **S10** as a pale yellow oil (15.3 mg, 32%).

¹H NMR (500 MHz, CDCl₃, 20°C) δ : 8.87 (m, 1H, *J* = 2.5 Hz, ArH), 8.44 (dd, 1H, *J* = 9.5, 2.5 Hz, ArH), 8.20 (d, 1H, *J* = 9.5 Hz ArH), 7.46 (dt, 2H, *J* = 9.0, 2.5 Hz, ArH), 7.39 (s, 1H, ArH), 7.12 (dt, 2H, *J* = 9.0, 2.5 Hz, ArH), 3.94 (s, 3H, OCH₃), 2.98 (tt, 1H, *J* = 12.0, 3.5 Hz, ⁶C₆H₁₁), 2.09–2.05 (m, 2H, ⁶C₆H₁₁), 1.95–1.92 (m, 2H, ⁶C₆H₁₁), 1.82–1.80 (m, 1H, ⁶C₆H₁₁), 1.73–1.65 (m, 2H, ⁶C₆H₁₁), 1.49 (qt, 2H, *J* = 12.5, 3.5 Hz, ⁶C₆H₁₁), 1.35 (qt, 1H, *J* = 12.5, 3.5 Hz, ⁶C₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20°C) δ : 170.6, 160.6, 151.0, 150.6, 145.2, 131.3, 131.0, 129.4, 124.9, 123.2, 122.8, 121.8, 114.8, 55.7, 48.0, 32.8, 26.6, 26.2.

FTIR (neat) cm⁻¹: 2929 (s), 2853 (m), 1609 (s), 1530 (m), 1491 (s), 1340 (s).

HRMS (ESI): calcd for C₂₂H₂₃N₂O₃ [M+H]⁺: 363.1703, found: 363.1708.

TLC (35% EtOAc/1% Et₃N/hexanes), *R*_f: 0.41 (UV, CAM).

Characterization of the byproduct S10:

¹H NMR (500 MHz, CDCl₃, 20°C) δ : 7.54 (dt, 2H, *J* = 8.5, 2.0 Hz, ArH), 6.90 (dt, 2H, *J* = 9.0, 2.0 Hz, ArH), 3.85 (s, 3H, OCH₃), 2.50 (tt, 1H, *J* = 11.0, 3.5 Hz, ⁶C₆H₁₁), 2.08–2.04 (m, 2H, ⁶C₆H₁₁), 1.84–1.81 (m, 2H, ⁶C₆H₁₁), 1.70–1.67 (m, 1H, ⁶C₆H₁₁), 1.54–1.46 (m, 2H, ⁶C₆H₁₁), 1.35 (qt, 2H, *J* = 12.5, 3.0 Hz, ⁶C₆H₁₁), 1.25 (qt, 1H, *J* = 12.0, 3.0 Hz, ⁶C₆H₁₁).

^{13}C NMR (125 MHz, CDCl_3 , 20 °C) δ :	191.9, 161.7, 135.3, 114.5, 112.2, 92.7, 87.3, 55.6, 52.4, 28.6, 26.0, 25.7.
FTIR (neat) cm^{-1} :	2931 (s), 2854 (m), 2192 (s), 1658 (s), 1603 (s), 1510 (s), 1253 (s).
HRMS (ESI):	calcd for $\text{C}_{16}\text{H}_{18}\text{NaO}_2$ $[\text{M}+\text{Na}]^+$: 265.1199, found: 265.1209.
TLC (35% EtOAc/1% Et_3N /hexanes), R_f :	0.56 (UV, CAM).

5,7-dimethoxy-2-phenylquinoline (4p**, Table 1):**

Trifluoromethanesulfonic anhydride (55 μL , 0.33 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (80 mg, 0.30 mmol, 1 equiv) and 2-chloropyridine (35 μL , 0.37 mmol, 1.2 equiv) in dichloromethane (1.0 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the vinyl ether **3a** (24 mg, 0.33 mmol, 1.1 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, aqueous sodium hydroxide solution (1 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 10% EtOAc in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline product **4p** as a pale yellow solid (81 mg, 99%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ :	8.36 (d, 1H, $J = 8.5$ Hz, ArH), 7.16 (d, 1H, $J = 8.7$ Hz, ArH), 7.00 (d, 1H, $J = 2.1$ Hz, ArH), 6.47 (d, 1H, $J = 2.3$ Hz, ArH), 3.97 (s, 3H, OCH ₃), 3.95 (s, 3H, OCH ₃), 2.87 (tt, 1H, $J = 12.0, 3.5$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$), 2.06–2.0 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.94–1.86 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.82–1.76 (m, 1H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.62 (qd, 2H, $J = 12.6, 3.0$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.48 (qt, 2H, $J = 12.6, 3.2$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.35 (qt, 1H, $J = 12.8, 3.5$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$).
^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ :	167.8, 156.2, 135.3, 131.2, 127.9, 116.5, 115.4, 99.7, 97.5, 55.9, 55.8, 47.8, 33.1, 26.8, 26.3.
FTIR (neat) cm^{-1} :	2929 (s), 2852 (m), 1643 (m), 1624 (s), 1608 (s), 1580 (s), 1511 (w), 1452 (m), 1397 (m), 1205 (s), 1151 (s).
HRMS (ESI):	calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 272.1645, found: 272.1644.
TLC (20% EtOAc/hexanes), R_f :	0.28 (UV, KMnO_4).

2-Cyclohexyl-5,7-dimethoxy-4-methylquinoline (4q, Table 1):

Trifluoromethanesulfonic anhydride (76 μL , 0.46 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (110 mg, 0.418 mmol, 1 equiv) and 2-chloropyridine (48 μL , 0.50 mmol, 1.2 equiv) in dichloromethane (1.4 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3c** (109 mg, 0.836 mmol, 2.00 equiv) was added via syringe, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a preheated oil bath at 45°C and maintained at that temperature. After 1 h, aqueous sodium hydroxide solution (1 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 3% EtOAc and 1% Et₃N $\rightarrow$ 10% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the quinoline product **4q** as a white solid (70 mg, 59%).

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 6.98 (s, 1H, ArH), 6.88 (s, 1H, ArH), 6.44 (s, 1H, ArH), 3.92 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 2.83–2.74 (m, 4H, CH₂, ^cC₆H₁₁), 2.02–1.96 (m, 2H, ^cC₆H₁₁), 1.91–1.84 (m, 2H, ^cC₆H₁₁), 1.80–1.74 (m, 1H, ^cC₆H₁₁), 1.59 (qd, 2H, $J = 12.7, 3.3$ Hz, ^cC₆H₁₁), 1.51–1.40 (m, 2H, ^cC₆H₁₁), 1.32 (qt, 1H, $J = 12.5, 3.4$ Hz, ^cC₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 166.8, 160.4, 158.7, 151.4, 145.9, 119.6, 115.4, 100.5, 97.9, 55.6, 55.6, 47.5, 33.0, 26.7, 26.3, 24.6.

FTIR (neat) cm⁻¹: 2998 (w), 2927 (s), 2851 (m), 1692 (w), 1619 (s), 1593 (s), 1452 (m), 1405 (m), 1252 (m), 1155 (m).

HRMS (ESI): calcd for C₁₈H₂₄NO₂ [M+H]⁺: 286.1802, found: 286.1804.

TLC (20% EtOAc/hexanes), R_f : 0.46 (UV, KMnO₄).

2-Cyclohexyl-5,7-dimethoxy-4-phenylquinoline (**4r**, Table 1):

Trifluoromethanesulfonic anhydride (71 μL , 0.42 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1c** (101 mg, 0.382 mmol, 1 equiv) and 2-chloropyridine (44 μL , 0.46, 1.2 equiv) in dichloromethane (1.3 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath, allowed to warm to 0°C , and after five minutes silylenol ether **3d** (158 μL , 0.768 mmol, 2.01 equiv) was added via syringe. After 5 min, the reaction mixture was heated to 45°C in an oil bath. After an hour, the reaction mixture was allowed to cool to ambient temperature and aqueous sodium hydroxide solution (2.0 mL, 1 N) was added to neutralize the trifluoromethanesulfonate salts. The reaction mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 10% EtOAc in hexanes; Al_2O_3 : 15×3 cm) on neutral alumina to give the quinoline derivative **4r** as a white solid (90 mg, 62%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 7.40–7.36 (m, 3H, ArH), 7.32–7.30 (m, 2H, ArH), 7.10 (d, 1H, $J = 2.5$ Hz, ArH), 6.96 (s, 1H, ArH), 6.42 (d, 1H, $J = 2.5$ Hz, ArH), 3.96 (s, 3H, OCH_3), 3.48 (s, 3H, OCH_3), 2.87 (tt, 1H, $J = 12.0, 3.5$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$), 2.06–2.03 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.89–1.87 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.79–1.76 (m, 1H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.65–1.57 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.50–1.41 (m, 2H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.35–1.26 (m, 1H, $^{\text{C}}\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 166.6, 160.9, 157.4, 151.4, 148.1, 143.2, 128.4, 127.0, 126.8, 119.6, 113.4, 100.6, 98.6, 55.7, 55.3, 47.6, 33.0, 26.7, 26.3.

FTIR (neat) cm^{-1} : 2927 (s), 2851 (m), 1619 (s), 1587 (s), 1563 (m), 1451 (m), 1403 (s), 1207 (s).

HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{26}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 348.1958, found: 348.1957.

TLC (35% EtOAc/1% Et_3N /hexanes), R_f : 0.57 (UV, CAM).

4-Cyclohexyl-7,9-dimethoxy-2,3-dihydro-1H-cyclopenta[c]quinoline (4s, Table 1):

Trifluoromethanesulfonic anhydride (76 μL , 0.46 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (110 mg, 0.418 mmol, 1 equiv) and 2-chloropyridine (48 μL , 0.51 mmol, 1.2 equiv) in dichloromethane (1.4 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3e** (131 mg, 0.836 mmol, 2.00 equiv) was added via syringe, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a preheated oil bath at 45°C and maintained at that temperature. After 1 h, the reaction mixture was allowed to cool to ambient temperature and aqueous sodium hydroxide solution (1 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 5% EtOAc and 0.5% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the quinoline product **4s** as a white solid (69 mg, 53%).

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 7.00 (s, 1H, ArH), 6.41 (s, 1H, ArH), 3.92 (s, 3H, OCH₃), 3.89 (s, 3H, OCH₃), 3.52–3.44 (m, 2H, CH₂CH₂CH₂), 3.04–2.96 (m, 2H, CH₂CH₂CH₂), 2.88–2.80 (m, 1H, ^cC₆H₁₁), 2.18–2.10 (m, 2H, CH₂CH₂CH₂), 1.94–1.70 (m, 7H, ^cC₆H₁₁), 1.48–1.36 (m, 3H, ^cC₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 163.3, 160.0, 157.4, 150.2, 149.9, 133.0, 113.9, 100.1, 97.5, 55.7, 55.6, 44.6, 35.4, 31.4, 30.5, 26.9, 26.2, 24.5.

FTIR (neat) cm⁻¹: 2998 (w), 2928 (s), 2850 (m), 1621 (s), 1583 (s), 1508 (w), 1450 (m), 1414 (w), 1360 (m), 1251 (m), 1205 (s).

HRMS (ESI): calcd for C₂₀H₂₆NO₂ [M+H]⁺: 312.1958, found: 312.1961.

TLC (20% EtOAc/hexanes), R_f: 0.59 (UV, KMnO₄).

6-Cyclohexyl-1,3-dimethoxy-7,8,9,10-tetrahydrophenanthridine (4t**, Table 1):**

Trifluoromethanesulfonic anhydride (76 μL , 0.46 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1c** (110 mg, 0.418 mmol, 1 equiv) and 2-chloropyridine (79 μL , 0.84 mmol, 2.0 equiv) in dichloromethane (1.4 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3f** (142 mg, 0.836 mmol, 2.00 equiv) was added via syringe, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a preheated oil bath at 45°C and maintained at that temperature. After 1 h, the reaction mixture was allowed to cool to ambient temperature and aqueous sodium hydroxide solution (1 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 5% EtOAc and 0.5% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline product **4t** as a white solid (83 mg, 61%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 6.96 (d, 1H, $J = 2.5$ Hz, ArH), 6.43 (d, 1H, $J = 2.5$ Hz, ArH), 3.93 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 3.42–3.48 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2$), 3.00–2.93 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 2.87–2.82 (m, 2H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2$), 1.92–1.73 (m, 11H, $^6\text{C}_6\text{H}_{11}$, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2$), 1.48–1.37 (m, 3H, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 165.6, 159.2, 158.6, 149.5, 143.6, 125.6, 114.9, 101.0, 98.2, 55.6, 55.6, 41.8, 32.1, 30.5, 27.1, 26.7, 26.4, 23.1, 22.6.

FTIR (neat) cm^{-1} : 2926 (s), 2851 (m), 1617 (s), 1577 (m), 1449 (m), 1406 (w), 1307 (w), 1245 (m), 1206 (s), 1157 (s).

HRMS (ESI): calcd for $\text{C}_{21}\text{H}_{28}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 326.2115, found: 326.2105.

TLC (20% EtOAc/hexanes), R_f : 0.59 (UV, KMnO_4).

2-Phenylquinoline (**4u**, Table 1):

Trifluoromethanesulfonic anhydride (92 μL , 0.56 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1j** (100 mg, 0.507 mmol, 1 equiv) and 2-chloropyridine (58 μL , 0.61 mmol, 1.2 equiv) in dichloromethane (1.7 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the vinyl ether **3a** (73 mg, 1.0 mmol, 2.0 equiv) was added via syringe. The resulting solution was allowed to warm to ambient temperature. After 1 h, aqueous sodium hydroxide solution (1 mL, 1N) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 5% EtOAc in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the quinoline product **4u**⁸ as a white solid (77 mg, 74%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 8.25 (d, 1H, $J = 8.5$ Hz, ArH), 8.21–8.16 (m, 3H, ArH), 7.90 (d, 1H, $J = 8.5$ Hz, ArH), 7.85 (d, 1H, $J = 8.2$ Hz, ArH), 7.75 (ddd, 1H, $J = 8.5, 7.0, 1.5$ Hz, ArH), 7.57–7.52 (m, 3H, ArH), 7.48 (tt, 1H, $J = 7.3, 1.2$ Hz, ArH).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 157.5, 148.4, 139.8, 137.0, 129.9, 129.9, 129.5, 129.0, 127.8, 127.7, 127.3, 126.5, 119.2.

FTIR (neat) cm^{-1} : 3189 (s), 3055 (w), 2091 (s), 1617 (w), 1597 (s), 1491 (m), 1447 (s).

HRMS (EI): calcd for $\text{C}_{15}\text{H}_{11}\text{N}$ $[\text{M}]^+$: 205.0886, found: 205.0885.

Analysis: calcd for $\text{C}_{15}\text{H}_{11}\text{N}$: C, 87.77; H, 5.40; N, 6.82, found: C, 87.55; H, 5.37; N, 6.84.

TLC (20% EtOAc–hexanes), R_f: 0.51 (UV, CAM).

⁸ For a two-step synthesis of **4u**, see Movassaghi, M.; Hill, M. D. *J. Am. Chem. Soc.* **2006**, *128*, 4592–4593.

2-Phenyl-5,6,7,8-tetrahydroquinoline (**4v**, Table 1):

Trifluoromethanesulfonic anhydride (57 μL , 0.34 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1k** (63 mg, 0.31 mmol, 1 equiv) and 2-chloropyridine (59 μL , 0.63 mmol, 2.0 equiv) in dichloromethane (1.1 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3b**⁹ (189 mg, 0.626 mmol, 2.00 equiv) was added, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a microwave reactor and heated to 140°C . After 20 min, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature before triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 3% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the pyridine derivative **4v**⁸ as a pale yellow solid (46 mg, 71%).

^1H NMR (500 MHz, CDCl_3 , 20°C): 7.97–7.94 (m, 2H, ArH), 7.48–7.36 (m, 5H, ArH), 3.00 (t, 2H, $J = 6.4$ Hz, CH_2), 2.82 (t, 2H, $J = 6.4$ Hz, CH_2), 1.98–1.91 (m, 2H, CH_2), 1.89–1.83 (m, 2H, CH_2).

^{13}C NMR (125 MHz, CDCl_3 , 20°C): 157.4, 154.9, 140.1, 137.6, 130.9, 128.8, 128.5, 127.0, 118.1, 33.1, 28.8, 23.4, 23.0.

FTIR (neat): 3061 (w), 3032 (w), 2935 (s), 2860 (m), 1590 (m), 1566 (m), 1460 (s), 1434 (m), 1253 (m).

HRMS (ESI): calcd for $\text{C}_{15}\text{H}_{16}\text{N}$ $[\text{M}+\text{H}]^+$: 210.1277, found: 210.1279.

TLC (20% EtOAc–hexanes), R_f : 0.48 (UV, KMnO_4).

⁹ For preparation of **3b** see Schaumann, E.; Tries, F. *Synthesis* **2002**, 191–194.

5-Phenylthieno[3,2-*b*]pyridine (**4w**, Table 1):

Trifluoromethanesulfonic anhydride (63 μL , 0.38 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1h** (70 mg, 0.34 mmol, 1 equiv) and 2-chloropyridine (39 μL , 0.41 mmol, 1.2 equiv) in dichloromethane (1.2 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3b** (208 mg, 0.688 mmol, 2.00 equiv) was added, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a microwave reactor and heated to 140°C . After 20 min, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature before triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 10% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×1.5 cm) on neutralized silica gel to give the pyridine product **4w**⁸ as a pale yellow solid (55 mg, 76%).

^1H NMR (500 MHz, CDCl_3 , 20°C):	8.26 (d, 1H, $J = 8.6$ Hz, ArH), 8.08 (d, 2H, $J = 7.3$ Hz, ArH), 7.78 (d, 1H, $J = 5.5$ Hz, CHS), 7.73 (d, 1H, $J = 8.5$ Hz, ArH), 7.65 (d, 1H, $J = 5.5$ Hz, CHCHS), 7.52 (t, 2H, $J = 7.0$ Hz, ArH), 7.45 (t, 1H, $J = 7.3$ Hz, ArH).
^{13}C NMR (125 MHz, CDCl_3 , 20°C):	156.4, 155.5, 139.8, 131.7, 131.0, 131.0, 129.0, 128.9, 127.4, 125.5, 116.5.
FTIR (neat):	3071 (s), 1906 (w), 1564 (s), 1544 (s), 1397 (s), 1280 (s), 1158 (s).
HRMS (ESI):	calcd for $\text{C}_{13}\text{H}_{10}\text{NS}$ $[\text{M}+\text{H}]^+$: 212.0528, found: 212.0534.
TLC (20% EtOAc–hexanes), R_f :	0.53 (UV, KMnO_4).

5-(Thiophen-2-yl)thieno[3,2-*b*]pyridine (4x**, Table 1):**

Trifluoromethanesulfonic anhydride (69 μL , 0.42 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **11** (80 mg, 0.38 mmol, 1 equiv) and 2-chloropyridine (72 μL , 0.76 mmol, 2.0 equiv) in dichloromethane (1.3 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the enol ether **3b** (231 mg, 0.764 mmol, 2.00 equiv) was added, and the resulting solution was allowed to warm to ambient temperature for 5 minutes before the reaction vessel was placed into a microwave reactor and heated to 140°C . After 20 min, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature before triethylamine (500 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 5% EtOAc and 0.5% Et₃N in hexanes; SiO₂: 15×1.5 cm) on neutralized silica gel to give the pyridine derivative **4x** as a pale yellow solid (61 mg, 74%).

¹H NMR (500 MHz, CDCl₃, 20°C): 8.18 (d, 1H, $J = 8.5$ Hz, ArH), 7.76 (d, 1H, $J = 5.5$ Hz, ArH), 7.68–7.65 (m, 2H, ArH), 7.59 (dd, 1H, $J = 5.5$, 0.6 Hz, ArH), 7.42 (dd, 1H, $J = 5.1$, 1.1 Hz, ArH), 7.14 (dd, 1H, $J = 5.0$, 3.7 Hz, ArH).

¹³C NMR (125 MHz, CDCl₃, 20°C): 156.1, 150.6, 145.3, 131.5, 131.1, 131.0, 128.2, 127.6, 125.3, 125.1, 115.1.

FTIR (neat): 3098 (w), 2733 (w), 2453 (w), 1996 (w), 1564 (s), 1548 (s), 1437 (m), 1395 (s), 1159 (m).

HRMS (ESI): calcd for C₁₁H₈NS₂ [M+H]⁺: 218.0093, found: 218.0096.

TLC (20% EtOAc–hexanes), *R*_f: 0.50 (UV, KMnO₄).

4-Cyclohexyl-8-methoxy-2,3-dihydro-1H-cyclopenta[c]quinoline (4y, Table 1):

Trifluoromethanesulfonic anhydride (81 μL , 0.48 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1a** (101 mg, 0.434 mmol, 1 equiv) and 2-chloropyridine (50 μL , 0.52, 1.2 equiv) in dichloromethane (1.5 mL) at -78°C . After five minutes, the reaction mixture was allowed to warm to 0°C for five minutes, and silylenol ether **3g** (198 μL , 0.868 mmol, 2.00 equiv) was added via syringe. The reaction vessel was placed in a microwave reactor and heated to 140°C . After one hour, the reaction vessel was removed from the microwave reactor and allowed to cool to ambient temperature. Aqueous sodium hydroxide solution (1 mL, 1 N) was added to the reaction mixture to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The solution was concentrated under reduced pressure, and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×3 cm) on neutralized silica gel to give the quinoline derivative **4y** as a white solid (91 mg, 75%).

^1H NMR (400 MHz, CDCl_3 , 20°C) δ : 7.98 (d, 1H, $J = 9.5$ Hz, ArH), 7.27–7.25 (m, 1H, ArH), 6.98 (d, 1H, $J = 3.0$ Hz, ArH), 3.93 (s, 3H, OCH_3), 3.22 (t, 2H, $J = 7.5$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 3.13 (t, 2H, $J = 7.5$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 2.86 (tt, 1H, $J = 11.5, 3.5$ Hz, $^c\text{C}_6\text{H}_{11}$), 2.27 (p, 2H, $J = 7.5$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 1.91–1.89 (m, 4H, $^c\text{C}_6\text{H}_{11}$), 1.83–1.76 (m, 3H, $^c\text{C}_6\text{H}_{11}$), 1.45–1.39 (m, 3H, $^c\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 160.9, 157.1, 148.4, 143.3, 135.4, 131.0, 125.8, 120.2, 102.2, 55.6, 44.7, 31.8, 31.5, 31.3, 26.9, 26.3, 24.2.

FTIR (neat) cm^{-1} : 3323 (w), 2924 (s), 2855 (s), 1621 (s), 1592 (s), 1505 (s), 1459 (s), 1344 (m).

HRMS (ESI): calcd for $\text{C}_{19}\text{H}_{24}\text{NO}$ $[\text{M}+\text{H}]^+$: 282.1852, found: 282.1866.

TLC (35% EtOAc/1% Et_3N /hexanes), R_f : 0.62 (UV, CAM).

Methyl 4-cyclohexyl-2,3-dihydro-1H-cyclopenta[c]quinoline-8-carboxylate (4z**, Table 1):**

Trifluoromethanesulfonic anhydride (62 μL , 0.37 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1g** (87 mg, 0.33 mmol, 1 equiv) and 2-chloropyridine (63 μL , 0.67, 2.0 equiv) in dichloromethane (800 μL) at -78°C . After five minutes, the reaction mixture was allowed to warm to 0°C for five minutes, cooled back to -78°C , and after an additional five minutes a solution of silylenol ether **3g** (134 μL , 0.67 mmol, 2.0 equiv) in dichloromethane (300 μL) was added via cannula. The reaction mixture was allowed to warm to 0°C for five minutes, and the reaction vessel was placed in a microwave reactor and heated to 140°C . After one hour, the reaction mixture was allowed to cool to ambient temperature and aqueous sodium hydroxide solution (1 mL, 1N) was added to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL). The layers were separated and the aqueous layer extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×3 cm) to give the quinoline derivative **4z** as an off-white solid (49 mg, 48%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 8.52 (d, 1H, $J = 2.0$ Hz, ArH), 8.19 (dd, 1H, $J = 9.0, 2.0$ Hz, ArH), 8.09 (d, 1H, $J = 8.5$ Hz, ArH), 3.99 (s, 3H, OCH_3), 3.33 (t, 2H, $J = 7.0$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 3.15 (t, 2H, $J = 7.0$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 2.91 (tt, 1H, $J = 12.0, 3.0$ Hz, $^{\text{C}}\text{C}_6\text{H}_{11}$), 2.30 (p, 2H, $J = 7.0$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_2$), 1.92–1.91 (m, 4H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.84–1.77 (m, 3H, $^{\text{C}}\text{C}_6\text{H}_{11}$), 1.49–1.37 (m, 3H, $^{\text{C}}\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 167.3, 165.9, 151.2, 149.5, 136.2, 129.8, 127.6, 127.5, 126.7, 124.4, 52.4, 45.0, 31.7, 31.4, 31.4, 26.8, 26.2, 24.2.

FTIR (neat) cm^{-1} : 2926 (s), 2850 (m), 1722 (s), 1452 (m), 1262 (s), 1250 (s).

HRMS (ESI): calcd for $\text{C}_{20}\text{H}_{24}\text{NO}_2$ $[\text{M}+\text{H}]^+$: 310.1802, found: 310.1801.

TLC (35% EtOAc/1% Et_3N /hexanes), R_f : 0.51 (UV, CAM).

4-cyclohexyl-8-nitro-2,3-dihydro-1H-cyclopenta[c]quinoline (4aa, Table 1):

Trifluoromethanesulfonic anhydride (77 μL , 0.45 mmol, 1.1 equiv) was added via syringe drop-wise to a stirred mixture of amide **1i** (103 mg, 0.413 mmol, 1 equiv) and 2-chloropyridine (78 μL , 0.82 mmol, 2.0 equiv) in dichloromethane (1.0 mL) at -78°C . After five minutes, the reaction mixture was allowed to warm to 0°C for five minutes, cooled back to -78°C , and, after five minutes, a solution of silyl ether **3g** (163 mg, 0.822 mmol, 1.99 equiv) in dichloromethane (400 μL) was added via cannula. The reaction mixture was allowed to warm to 0°C for five minutes, and the reaction vessel was placed in a microwave reactor and heated to 140°C . After one hour, the reaction mixture was allowed to cool to ambient temperature and aqueous sodium hydroxide solution (1 mL, 1N) was added to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between dichloromethane (10 mL) and water (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane ($2 \times 10\text{ mL}$) and the combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et₃N in hexanes; SiO₂: $15 \times 3\text{ cm}$) to give the quinoline derivative **4aa** as a yellow solid (33 mg, 27%).

¹H NMR (400 MHz, CDCl₃, 20°C) δ : 8.67 (d, 1H, $J = 2.8$, ArH), 8.33 (dd, 1H, $J = 9.2\text{ Hz}$, 2.8 Hz ArH), 8.11 (m, 1H, $J = 9.2\text{ Hz}$, ArH), 3.32 (t, 2H, $J = 7.6\text{ Hz}$, CH₂CH₂CH₂), 3.14 (t, 2H, $J = 7.4\text{ Hz}$, CH₂CH₂CH₂), 2.89 (tt, 1H, $J = 11.6, 3.2\text{ Hz}$, ^cC₆H₁₁), 2.30 (p, 2H, $J = 7.6\text{ Hz}$, CH₂CH₂CH₂), 1.91–1.86 (m, 4H, ^cC₆H₁₁), 1.81–1.71 (m, 3H, ^cC₆H₁₁), 145.–1.27 (m, 3H, ^cC₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20°C) δ : 167.6, 152.0, 149.8, 144.7, 137.6, 131.2, 124.1, 121.6, 121.4, 45.1, 31.8, 31.5, 31.4, 26.8, 26.2, 24.2.

FTIR (neat) cm⁻¹: 2929 (s), 2853 (m), 1600 (m), 1528 (m), 1492 (m), 1340 (s).

HRMS (ESI): calcd for C₁₈H₂₁N₂O₂ [M+H]⁺: 297.1598, found: 297.1592.

TLC (9% EtOAc/1% Et₃N/hexanes), R_f: 0.37 (UV, CAM).

6-Cyclohexyl-2-methoxy-7H-indeno[2,1-c]quinoline (4bb, Table 1):

Trifluoromethanesulfonic anhydride (81 μL , 0.48 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1a** (102 mg, 0.438 mmol, 1 equiv) and 2-chloropyridine (83 μL , 0.88, 2.0 equiv) in dichloromethane (1.0 mL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C . After 5 min, the mixture was cooled back to -78°C and after another 5 min a solution of the silylenol ether **3h** (216 μL , 0.875 mmol, 2.00 equiv) in dichloromethane (460 μL) was added via cannula. After 5 min, the reaction vessel was placed in an ice-water bath and warmed to 0°C , and after an additional 5 min, the reaction vessel was placed in the microwave reactor and heated to 140°C . After an hour, the reaction vessel was removed from the microwave reactor and cooled to ambient temperature before adding aqueous sodium hydroxide (1 mL) to the reaction mixture to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et_3N in hexanes; SiO_2 : 15×3 cm) to give the quinoline derivative **4bb** as an off-white solid (95 mg, 66%).

^1H NMR (500 MHz, CDCl_3 , 20°C) δ : 8.34 (d, 1H, $J = 8.0$ Hz, ArH), 8.11 (d, 1H, $J = 9.0$ Hz, ArH), 7.93 (d, 1H, $J = 2.5$ Hz, ArH), 7.72 (d, 1H, $J = 7.5$, ArH), 7.55 (td, 1H, $J = 7.5$, 1.0 Hz, ArH), 7.48 (td, 1H, $J = 7.5$, 1.0 Hz, ArH), 7.39 (dd, 1H, $J = 9.5$, 2.5 Hz, ArH), 4.07 (s, 2H, CH_2), 4.06 (s, 3H, OCH_3), 3.11–3.06 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 2.00–1.89 (m, 6H, $^6\text{C}_6\text{H}_{11}$), 1.85–1.81 (m, 1H, $^6\text{C}_6\text{H}_{11}$), 1.55–1.43 (m, 3H, $^6\text{C}_6\text{H}_{11}$).

^{13}C NMR (125 MHz, CDCl_3 , 20°C) δ : 160.6, 157.5, 144.6, 144.4, 143.1, 141.6, 134.8, 131.7, 127.7, 127.3, 125.4, 124.4, 123.8, 120.0, 102.4, 55.7, 44.4, 36.0, 31.9, 27.0, 26.3

FTIR (neat) cm^{-1} : 2929 (s), 2848 (m), 1643 (w), 1625 (m), 1568 (m), 1508 (s), 1221 (s).

HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{24}\text{NO}$ $[\text{M}+\text{H}]^+$: 330.1852, found: 330.1845.

TLC (35% EtOAc/1% Et_3N /hexanes), R_f : 0.42 (UV, CAM).

6-Cyclohexyl-2-methoxy-11H-indeno[1,2-c]quinoline (4cc, Table 1):

Trifluoromethanesulfonic anhydride (81 μL , 0.48 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1a** (101 mg, 0.433 mmol, 1 equiv) and 2-chloropyridine (49 μL , 0.52, 1.2 equiv) in dichloromethane (1.5 mL) at -78°C . After five minutes, the reaction mixture was placed in an ice-water bath, allowed to warm to 0°C for five minutes, and a solution of the silylenol ether **3i** (220 μL , 0.866 mmol, 2.00 equiv) in dichloromethane (200 μL) was added via cannula. The reaction vessel was placed in a microwave reactor and heated to 140°C . After one hour, the reaction mixture was allowed to cool to ambient temperature and sodium hydroxide solution (1 mL, 1 N) was added to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between dichloromethane (10 mL) and water (10 mL), and the aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The solution was concentrated under reduced pressure, and the residue was purified by flash column chromatography (eluent: 9% EtOAc and 1% Et₃N in hexanes; SiO₂: 15×3 cm) to give the quinoline derivative **4cc** as an off-white solid (112 mg, 78%).

¹H NMR (500 MHz, CDCl₃, 20°C) δ : 8.03 (d, 1H, $J = 9.0$ Hz, ArH), 7.96 (d, 1H, $J = 7.5$ Hz, ArH), 7.67 (d, 1H, $J = 8.0$ Hz, ArH), 7.48 (td, 1H, $J = 7.5, 0.5$ Hz, ArH), 7.38 (td, 1H, $J = 7.5, 0.5$ Hz, ArH), 7.32 (dd, 1H, $J = 9.0, 2.5$ Hz, ArH), 7.20 (d, 1H, $J = 3.0$ Hz, ArH), 4.17 (s, 2H, CH₂), 3.98 (s, 3H, OCH₃), 3.52 (tt, 1H, $J = 11.5, 3.0$ Hz, C₆H₁₁), 2.17–2.15 (m, 2H, C₆H₁₁), 2.03–1.99 (m, 2H, C₆H₁₁), 1.96–1.87 (m, 3H, C₆H₁₁), 1.66–1.57 (m, 2H, C₆H₁₁), 1.50–1.41 (m, 1H, C₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20°C) δ : 159.7, 157.6, 148.5, 143.5, 142.5, 141.3, 133.3, 131.5, 127.5, 126.6, 125.6, 125.2, 123.3, 121.1, 101.7, 55.8, 43.9, 36.0, 31.5, 27.1, 26.5.

FTIR (neat) cm⁻¹: 3432 (br), 2927 (s), 2850 (m), 1622 (m), 1574 (w), 1477 (w), 1220 (s).

HRMS (ESI): calcd for C₂₃H₂₄NO [M+H]⁺: 330.1852, found: 330.1843.

TLC (35% EtOAc/1% Et₃N/hexanes), R_f: 0.44 (UV, CAM).

(Z)-2-(Cyclohexyl(4-methoxyphenylamino)methylene)cyclopentanone (8, equation 4):

Trifluoromethanesulfonic anhydride (82 μ L, 0.49 mmol, 1.1 equiv) was added drop-wise via syringe to a stirred mixture of amide **1a** (103 mg, 0.439 mmol, 1 equiv) and 2-chloropyridine (50 μ L, 0.53, 1.2 equiv) in dichloromethane (1.5 mL) at -78°C . After five minutes, the reaction mixture was placed in an ice-water bath, allowed to warm to 0°C for five minutes, and silylenol ether **3e** (158 μ L, 0.885 mmol, 2.01 equiv) was added via syringe. After five minutes, the reaction mixture was allowed to warm to ambient temperature. After 20 minutes, aqueous sodium hydroxide solution (2 mL) was added to the reaction mixture to neutralize the trifluoromethanesulfonate salts. The mixture was partitioned between water (10 mL) and dichloromethane (10 mL), and the layers were separated. The aqueous layer was extracted with dichloromethane (2×10 mL). The combined organic layers were washed with brine (10 mL), were dried over anhydrous sodium sulfate, and were filtered. The volatiles were removed under reduced pressure, and the residue was purified by flash column chromatography (eluent: 19% EtOAc and 1% Et₃N in hexanes; SiO₂: 13×3 cm) to give the vinylogous amide **8** as a pale-yellow oil (102 mg, 78%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 12.48 (br s, 1H, NH), 7.00 (d, 2H, $J = 8.5$ Hz, ArH), 6.88 (d, 2H, $J = 8.5$ Hz, ArH), 3.83 (s, 3H, OCH₃), 2.77 (t, 2H, $J = 7.0$ Hz, CH₂CH₂CH₂), 2.50–2.46 (m, 1H, ^cC₆H₁₁), 2.35 (t, 2H, $J = 8.0$ Hz, CH₂CH₂CH₂), 1.88 (p, 2H, $J = 7.5$ Hz, CH₂CH₂CH₂), 1.74–1.58 (m, 7H, ^cC₆H₁₁), 1.15–1.06 (m, 3H, ^cC₆H₁₁).

¹³C NMR (125 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 204.6, 164.9, 157.9, 132.3, 127.8, 114.3, 103.1, 55.7, 40.5, 38.8, 28.9, 28.7, 26.4, 25.9, 21.5.

FTIR (neat) cm⁻¹: 2930 (s), 2852 (m), 1618 (s), 1576 (s), 1512 (s), 1450 (m), 1240 (s), 1211 (s).

HRMS (ESI): calcd for C₁₉H₂₆NO₂ [M+H]⁺: 300.1958, found: 300.1947.

TLC (20% EtOAc/1% Et₃N/hexanes), R_f: 0.25 (UV, CAM).

3-(3,8-Diphenyl-2-(thiophen-2-yl)quinolin-4-yl)oxazolidin-2-one (4dd, equation 5):

Trifluoromethanesulfonic anhydride (52 μ L, 0.32 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1m** (80 mg, 0.29 mmol, 1 equiv) and 2-chloropyridine (33 μ L, 0.34 mmol, 1.2 equiv) in dichloromethane (950 μ L) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamide **2d** (59 mg, 0.32 mmol, 1.1 equiv) was added as a solid in one portion and the reaction flask was rapidly purged and sealed under an argon atmosphere. The resulting solution was allowed to warm to ambient temperature. After 1 h, triethylamine (500 μ L) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 40% EtOAc/1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the quinoline derivative **4dd** as a pale yellow solid (73 mg, 57%).

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 7.87–7.81 (m, 4H, ArH), 7.69–7.59 (m, 3H, ArH), 7.57–7.52 (m, 3H, ArH), 7.50–7.44 (m, 2H, ArH), 7.28–7.26 (m, 2H, ArH), 6.74 (dd, 1H, J = 5.1, 3.9 Hz, ArH), 6.28 (dd, 1H, J = 3.9, 1.1 Hz, ArH), 4.51–4.45 (m, 1H, NCH₂CH₂O), 4.07–4.01 (m, 1H, NCH₂CH₂O), 3.90–3.84 (m, 1H, NCH₂CH₂O), 3.41–3.35 (m, 1H, NCH₂CH₂O).

¹³C NMR (125 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 157.3, 151.4, 146.1, 145.9, 141.3, 140.8, 138.9, 135.9, 132.2, 131.5, 131.2, 129.9, 129.9, 129.6, 129.2, 129.1, 128.9, 127.9, 127.8, 127.6, 124.7, 121.9, 63.2, 47.2.

FTIR (neat) cm⁻¹: 3060 (w), 2916 (w), 2249 (w), 1753 (s), 1601 (w), 1579 (m), 1479 (s), 1421 (s), 1231 (m).

HRMS (EI): calcd for C₂₈H₂₁N₂O₂S [M+H]⁺: 449.1318, found: 449.1317.

TLC (50% EtOAc in hexanes), R_f : 0.43 (UV, KMnO₄)

Characterization of the product **9** (see text):

¹H NMR (500 MHz, CDCl₃, 20 $^{\circ}\text{C}$) δ : 8.71 (d, 1H, J = 8.3 Hz, ArH), 8.59 (dd, 2H, J = 7.9, 4.6 Hz, ArH), 8.22 (d, 1H, J = 8.2 Hz, ArH), 7.89 (t, 1H, J = 7.5 Hz, ArH), 7.78–7.65 (m, 4H, ArH), 7.58 (dd, 1H, J = 5.1, 1.0 Hz, ArH), 7.25 (dd, 1H, J = 5.2, 3.6 Hz, ArH).

^{13}C NMR (125 MHz, CDCl_3 , 20 °C) δ : 154.2, 143.9, 142.7, 133.8, 130.8, 130.4, 129.5, 129.1, 128.3, 128.1, 127.6, 127.6, 127.2, 124.9, 123.7, 122.5, 122.1.

FTIR (neat) cm^{-1} : 3070 (m), 1956 (w), 1812 (w), 1734 (w), 1610 (m), 1577 (m), 1562 (s), 1519 (m), 1484 (s), 1458 (s), 1430 (s).

HRMS (EI): calcd for $\text{C}_{17}\text{H}_{12}\text{NS}$ $[\text{M}+\text{H}]^+$: 262.0685, found: 262.0683.

TLC (20% EtOAc in hexanes), R_f : 0.51 (UV, KMnO_4).

(S)-1-(2-sec-butyl-3-phenyl-5,6,7,8-tetrahydroquinolin-4-yl)azetidin-2-one (4ee, equation 6):

Trifluoromethanesulfonic anhydride (15 μL , 0.091 mmol, 1.1 equiv) was added via syringe over 1 min to a stirred mixture of amide **1n**¹⁰ (15 mg, 0.083 mmol, 1 equiv) and 2-chloropyridine (16 μL , 0.17 mmol, 2.0 equiv) in dichloromethane (280 μL) at -78°C . After 5 min, the reaction mixture was placed in an ice-water bath and warmed to 0°C , the ynamide **2h** (16 mg, 0.091 mmol, 1.1 equiv) was added as a solid in one portion and the reaction flask was rapidly purged and sealed under an argon atmosphere. After 1 h, triethylamine (100 μL) was introduced to neutralize the trifluoromethanesulfonate salts. Dichloromethane (5 mL) was added to dilute the mixture and the layers were separated. The organic layer was washed with brine (2 mL), was dried over anhydrous sodium sulfate, and was filtered. The volatiles were removed under reduced pressure and the residue was purified by flash column chromatography (eluent: 20% EtOAc and 1% Et₃N in hexanes; SiO₂: 15 $\times$ 1.5 cm) on neutralized silica gel to give the pyridine derivative **4ee** as a pale yellow oil (19 mg, 69%). The enantiomeric excess of pyridine **4ee** was determined to be 94% ee by chiral HPLC analysis [Whelk-O1 (*S,S*); 0.5 mL/min; 1% *i*PrOH in hexanes containing 0.2% Et₃N; t_R (minor) = 55.0 min, t_R (major) = 61.2 min].

¹H NMR (500 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 7.46–7.41 (m, 2H, ArH), 7.41–7.36 (m, 1H, ArH), 7.23–7.18 (m, 2H, ArH), 2.95 (t, 2H, J = 6.3 Hz, NCH₂CH₂CO), 2.85–2.62 (m, 7H, NCH₂CH₂CO, CH₂CH₂CH₂CH₂, CH₃CH₂CHCH₃), 1.96–1.73 (m, 5H, CH₂CH₂CH₂CH₂, CH₃CH₂CHCH₃), 1.50–1.40 (m, 1H, CH₃CH₂CHCH₃), 1.11 (d, 3H, J = 6.8 Hz, CH₃CH₂CHCH₃), 0.67 (t, 3H, J = 7.5 Hz, CH₃CH₂CHCH₃).

¹³C NMR (125 MHz, CDCl₃, 20 $^\circ\text{C}$) δ : 166.0, 161.6, 158.1, 140.9, 136.9, 130.9, 129.9, 129.7, 128.6, 128.6, 127.7, 126.6, 41.0, 38.6, 36.5, 33.2, 29.6, 25.3, 22.9, 22.7, 21.0, 12.5.

FTIR (neat) cm⁻¹: 2959 (m), 2933 (m), 2870 (w), 1757 (s), 1602 (w), 1570 (w), 1547 (m), 1432 (m), 1405 (m), 1379 (m), 1192 (m).

HRMS (ESI): calcd for C₂₂H₂₇N₂O [M+H]⁺: 335.2118, found: 335.2115.

¹⁰ The enantiomeric excess of amide **1n** was determined to be 94% ee by chiral HPLC analysis [Whelk-O1 (*S,S*); 1.0 mL/min; 3% *i*PrOH in hexanes; t_R (major) = 28.6 min, t_R (minor) = 32.5 min].

Crystal Structure of quinoline 4j (Table 1).

Table S1. Crystal data and structure refinement for **4j**.

Identification code	07069	
Empirical formula	C ₂₆ H ₂₆ N ₂ O ₄	
Formula weight	430.49	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 16.767(13) Å	α = 90°.
	b = 6.055(5) Å	β = 97.944(14)°.
	c = 20.972(16) Å	γ = 90°.
Volume	2109(3) Å ³	
Z	4	
Density (calculated)	1.356 Mg/m ³	
Absorption coefficient	0.092 mm ⁻¹	
F(000)	912	
Crystal size	0.40 x 0.10 x 0.05 mm ³	
Theta range for data collection	1.96 to 29.57°.	
Index ranges	-23 ≤ h ≤ 23, -8 ≤ k ≤ 8, -29 ≤ l ≤ 29	
Reflections collected	43401	
Independent reflections	5922 [R(int) = 0.0672]	
Completeness to theta = 29.57°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9954 and 0.9642	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5922 / 0 / 290	
Goodness-of-fit on F ²	1.032	
Final R indices [I > 2σ(I)]	R1 = 0.0415, wR2 = 0.0997	
R indices (all data)	R1 = 0.0617, wR2 = 0.1107	
Largest diff. peak and hole	0.412 and -0.238 e. Å ⁻³	

Table S2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\approx^2 \times 10^3$) for **4j**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	177(1)	5105(2)	2289(1)	14(1)
C(2)	204(1)	4446(2)	2912(1)	14(1)
C(3)	822(1)	5357(2)	3373(1)	14(1)
C(4)	1280(1)	7561(2)	2609(1)	15(1)
C(5)	1794(1)	9275(2)	2466(1)	18(1)
C(6)	1759(1)	10043(2)	1857(1)	18(1)
C(7)	1237(1)	9063(2)	1356(1)	16(1)
C(8)	741(1)	7375(2)	1478(1)	15(1)
C(9)	733(1)	6652(2)	2111(1)	14(1)
C(10)	-1044(1)	5488(2)	1535(1)	16(1)
C(11)	-1328(1)	1920(2)	1262(1)	19(1)
C(12)	-468(1)	2003(2)	1587(1)	18(1)
C(21)	-427(1)	2949(2)	3090(1)	15(1)
C(22)	-1224(1)	3624(2)	2990(1)	17(1)
C(23)	-1829(1)	2233(2)	3125(1)	20(1)
C(24)	-1642(1)	145(2)	3360(1)	22(1)
C(25)	-852(1)	-536(2)	3464(1)	21(1)
C(26)	-248(1)	850(2)	3330(1)	18(1)
C(31)	926(1)	4662(2)	4065(1)	15(1)
C(32)	1654(1)	3147(2)	4215(1)	21(1)
C(33)	1770(1)	2439(2)	4914(1)	25(1)
C(34)	1845(1)	4415(2)	5359(1)	23(1)
C(35)	1126(1)	5918(2)	5213(1)	22(1)
C(36)	1013(1)	6642(2)	4517(1)	20(1)
N(1)	-420(1)	4267(2)	1812(1)	14(1)
N(3)	1327(1)	6868(2)	3226(1)	16(1)
O(1)	-1157(1)	7422(1)	1608(1)	22(1)
O(2)	-1551(1)	4209(1)	1145(1)	18(1)
O(3)	929(1)	8872(2)	219(1)	25(1)
O(4)	1343(1)	12056(1)	689(1)	22(1)
C(13)	1158(1)	9920(2)	692(1)	17(1)
C(14)	1217(1)	13123(2)	73(1)	26(1)

Table 3. Bond lengths [$\approx$] and angles [$^\circ$] for **4j**.

C(1)-C(2)	1.3626(19)	C(8)-C(9)	1.3999(19)
C(1)-C(9)	1.4070(17)	C(10)-O(1)	1.1994(17)
C(1)-N(1)	1.4088(16)	C(10)-O(2)	1.3405(15)
C(2)-C(3)	1.4265(17)	C(10)-N(1)	1.3448(16)
C(2)-C(21)	1.4789(17)	C(11)-O(2)	1.4472(18)
C(3)-N(3)	1.3117(16)	C(11)-C(12)	1.507(2)
C(3)-C(31)	1.498(2)	C(12)-N(1)	1.4481(18)
C(4)-N(3)	1.3514(18)	C(21)-C(26)	1.3852(19)
C(4)-C(9)	1.4032(18)	C(21)-C(22)	1.3855(19)
C(4)-C(5)	1.4073(18)	C(22)-C(23)	1.3777(19)
C(5)-C(6)	1.353(2)	C(23)-C(24)	1.377(2)
C(6)-C(7)	1.4040(18)	C(24)-C(25)	1.376(2)
C(7)-C(8)	1.3632(18)	C(25)-C(26)	1.3744(19)
C(7)-C(13)	1.475(2)	C(31)-C(36)	1.5226(19)
		C(31)-C(32)	1.5245(19)
		C(32)-C(33)	1.514(2)

C(33)-C(34)	1.511(2)	O(2)-C(11)-C(12)	104.65(10)
C(34)-C(35)	1.507(2)	N(1)-C(12)-C(11)	100.69(9)
C(35)-C(36)	1.511(2)	C(26)-C(21)-C(22)	118.79(11)
O(3)-C(13)	1.1942(17)		
O(4)-C(13)	1.3303(18)	C(26)-C(21)-C(2)	121.82(11)
O(4)-C(14)	1.4342(18)	C(22)-C(21)-C(2)	119.34(12)
		C(23)-C(22)-C(21)	120.75(13)
C(2)-C(1)-C(9)	120.88(11)	C(24)-C(23)-C(22)	119.82(13)
C(2)-C(1)-N(1)	120.17(11)	C(25)-C(24)-C(23)	119.86(12)
C(9)-C(1)-N(1)	118.95(11)	C(26)-C(25)-C(24)	120.39(13)
C(1)-C(2)-C(3)	117.53(11)	C(25)-C(26)-C(21)	120.38(12)
C(1)-C(2)-C(21)	119.38(10)	C(3)-C(31)-C(36)	111.72(11)
C(3)-C(2)-C(21)	122.98(11)	C(3)-C(31)-C(32)	110.60(10)
N(3)-C(3)-C(2)	122.76(12)	C(36)-C(31)-C(32)	109.93(11)
N(3)-C(3)-C(31)	115.60(10)	C(33)-C(32)-C(31)	111.25(11)
C(2)-C(3)-C(31)	121.63(11)	C(34)-C(33)-C(32)	111.21(12)
N(3)-C(4)-C(9)	122.39(11)	C(35)-C(34)-C(33)	110.96(11)
N(3)-C(4)-C(5)	118.43(11)	C(34)-C(35)-C(36)	111.23(11)
C(9)-C(4)-C(5)	119.17(12)	C(35)-C(36)-C(31)	111.17(12)
C(6)-C(5)-C(4)	120.44(12)	C(10)-N(1)-C(1)	122.86(11)
C(5)-C(6)-C(7)	120.19(12)	C(10)-N(1)-C(12)	112.02(10)
C(8)-C(7)-C(6)	120.61(12)	C(1)-N(1)-C(12)	125.04(10)
C(8)-C(7)-C(13)	117.63(11)	C(3)-N(3)-C(4)	119.24(11)
C(6)-C(7)-C(13)	121.61(12)	C(10)-O(2)-C(11)	108.87(10)
C(7)-C(8)-C(9)	119.94(11)	C(13)-O(4)-C(14)	115.84(11)
C(8)-C(9)-C(4)	119.45(12)	O(3)-C(13)-O(4)	124.19(12)
C(8)-C(9)-C(1)	123.56(11)	O(3)-C(13)-C(7)	124.98(12)
C(4)-C(9)-C(1)	116.94(12)	O(4)-C(13)-C(7)	110.78(11)
O(1)-C(10)-O(2)	122.90(11)		
O(1)-C(10)-N(1)	127.54(12)		
O(2)-C(10)-N(1)	109.56(11)		

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($\approx 2 \times 10^3$) for **4j**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	11(1)	11(1)	20(1)	-1(1)	0(1)	0(1)
C(2)	11(1)	11(1)	19(1)	1(1)	2(1)	1(1)
C(3)	12(1)	13(1)	18(1)	0(1)	1(1)	2(1)
C(4)	12(1)	13(1)	20(1)	1(1)	1(1)	1(1)
C(5)	14(1)	17(1)	21(1)	1(1)	1(1)	-3(1)
C(6)	14(1)	16(1)	23(1)	2(1)	3(1)	-3(1)
C(7)	14(1)	14(1)	20(1)	2(1)	3(1)	2(1)
C(8)	13(1)	14(1)	19(1)	1(1)	1(1)	1(1)
C(9)	12(1)	11(1)	20(1)	1(1)	1(1)	1(1)
C(10)	14(1)	14(1)	19(1)	2(1)	1(1)	-1(1)
C(11)	18(1)	12(1)	24(1)	-1(1)	-2(1)	0(1)
C(12)	17(1)	12(1)	25(1)	-4(1)	0(1)	1(1)
C(21)	14(1)	14(1)	16(1)	-1(1)	1(1)	-3(1)
C(22)	17(1)	16(1)	19(1)	0(1)	1(1)	0(1)
C(23)	14(1)	23(1)	23(1)	-1(1)	2(1)	-2(1)
C(24)	20(1)	21(1)	24(1)	1(1)	4(1)	-8(1)
C(25)	24(1)	14(1)	23(1)	2(1)	2(1)	-4(1)
C(26)	16(1)	15(1)	21(1)	0(1)	1(1)	0(1)
C(31)	13(1)	16(1)	18(1)	1(1)	1(1)	-1(1)
C(32)	20(1)	21(1)	23(1)	4(1)	3(1)	6(1)
C(33)	27(1)	24(1)	24(1)	6(1)	1(1)	8(1)

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C(34)	19(1)	28(1)	21(1)	5(1)	-2(1)	-2(1)
C(35)	24(1)	22(1)	18(1)	0(1)	-1(1)	0(1)
C(36)	24(1)	17(1)	20(1)	0(1)	-2(1)	2(1)
N(1)	14(1)	10(1)	19(1)	0(1)	-1(1)	-1(1)
N(3)	13(1)	15(1)	19(1)	2(1)	1(1)	0(1)
O(1)	21(1)	11(1)	33(1)	1(1)	-2(1)	1(1)
O(2)	18(1)	12(1)	23(1)	1(1)	-4(1)	-1(1)
O(3)	34(1)	20(1)	20(1)	1(1)	4(1)	-2(1)
O(4)	28(1)	15(1)	22(1)	5(1)	3(1)	-3(1)
C(13)	14(1)	15(1)	23(1)	3(1)	4(1)	1(1)
C(14)	34(1)	19(1)	24(1)	9(1)	5(1)	2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\approx 2 \times 10^3$) for **4j**.

	x	y	z	U(eq)
H(5)	2167	9897	2800	21
H(6)	2090	11248	1769	22
H(8)	402	6690	1134	19
H(11A)	-1368	1090	852	22
H(11B)	-1679	1209	1544	22
H(12A)	-373	942	1948	22
H(12B)	-83	1712	1279	22
H(22)	-1354	5062	2827	21
H(23)	-2373	2713	3056	24
H(24)	-2058	-823	3451	26
H(25)	-724	-1973	3629	25
H(26)	296	365	3403	21
H(31)	436	3817	4142	19
H(32A)	1579	1823	3936	26
H(32B)	2143	3928	4120	26
H(33A)	1307	1525	4999	30
H(33B)	2263	1524	5003	30
H(34A)	1887	3905	5811	27
H(34B)	2341	5242	5308	27
H(35A)	1202	7235	5495	26
H(35B)	637	5136	5306	26
H(36A)	1481	7540	4434	25
H(36B)	525	7577	4431	25
H(14A)	1617	12593	-190	38
H(14B)	1273	14724	131	38
H(14C)	675	12781	-143	38

Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 16 repetitions
 OBSERVE H1, 499.7417206 MHz
 DATA PROCESSING
 FT size 262144
 Total time 2 min, 8 sec

Relax. delay 0.700 sec
 Pulse 36.7 degrees
 Acq. time 2.000 sec
 Width 31397.2 Hz
 48 repetitions
 OBSERVE C13, 125.6601366 MHz
 DECOUPLE H1, 499.7442194 MHz
 Power 34 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 752 hr, 28 min, 15 sec

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
7 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

PULSE SEQUENCE
 Relax. delay 5.000 sec
 Pulse 69.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 616 repetitions
 OBSERVE C13, 125.7832297 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 2.0 Hz
 FT size 131072
 Total time 69 minutes

Solvent: CDCl₃
 Ambient temperature
 File: mh-VI-212
 INOVA-500 "Zippy"
 PULSE SEQUENCE
 Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 7 repetitions
 OBSERVE H1, 499.7417206 MHz
 DATA PROCESSING
 FT size 262144
 Total time 2 min, 8 sec

Current Data Parameters
 NAME MH-VI-251
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070418
 Time 18.05
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 667
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2580.3
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SF01 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127535 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

S46/S112

Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 6 repetitions
 OBSERVE H1, 499.7417206 MHz
 DATA PROCESSING
 FT size 262144
 Total time 2 min, 8 sec

מד

```

INNOVA-3000 11/06/94
PULSE SEQUENCE
Relax. delay 0.750 sec
Pulse 65.4 degrees
Acq. time 1.736 sec
Width 37735.8 Hz
320 repetitions
OBSERVE C13, 125.7832771 MHz
DECOUPLE H1, 500.2332753 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 13 minutes

```

PULSE SEQUENCE
 Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 8 repetitions
 OBSERVE H1, 499.7417206 MHz
 DATA PROCESSING
 FT size 131072
 Total time 2 min, 8 sec

F2 - Processing parameters	
SI	65536
SF	100.6127583 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

F2 - Acquisition Parameters

Date_ 20070323
 Time 9.11
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 57
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====

NUC1 1H
 P1 13.88 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 65536
 SF 400.1300052 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F2 - Acquisition Parameters

Date_ 20070323
 Time 9.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 133
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2896.3
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 65536
 SF 100.6127633 MHz
 EM
 WDW 0
 SSB 1.00 Hz
 LB 0
 GB 0
 PC 1.40

ppm

200 180 160 140 120 100 80 60 40 20 0

4g

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
10 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

Relax. delay 3.000 sec
 Pulse 36.7 degrees
 Acq. time 2.000 sec
 Width 31397.2 Hz
 96 repetitions
 OBSERVE C13, 125.6601386 MHz
 DECOUPLE H1, 499.7442194 MHz
 Power 34 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 13913 hr, 35 min, 56 sec

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
10 repetitions
OBSERVE HI, 499.7417206 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 8 sec

4h

PULSE SEQUENCE
 Relax. delay 0.753 sec
 Pulse 65.4 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 1000000 repetitions
 OBSERVE C13, 125.7832320 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 38 dB
 continuously on
 VALT2-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 6942.2 hours

PULSE SEQUENCE
 Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 16 repetitions
 OBSERVE H1, 499.7417206 MH
 DATA PROCESSING
 FT size 262144
 Total time 2 min, 8 sec

Relax. delay 0.763 sec
Pulse 65.0 degrees
Acq. time 1.736 sec
Width 37735.8 Hz
128 repetitions
OBSERVE C13, 125.7832349 MHz
DECOUPLE H1, 500.2332753 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.3 Hz
FT size 131072
Total time 5 minutes

date May 1 2007
 solvent CDCl3
 file /data/export/~
 home/movassag/MVoa~
 hm/bullwinkle/DA-I~
 I-259-2.fid
 ACQUISITION
 sfrq 499.746
 tn H1
 at 3.001
 np 63050
 sw 10504.2
 fb not used
 bs 2
 tpwr 56
 pw 8.6
 d1 2.000
 tof 1519.5
 nt 32
 ct 24
 alock n
 gain not used
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -249.9
 wp 6246.7
 vs 126
 sc 0
 wc 250
 hzmm 24.99
 ls 33.57
 rfl 1233.6
 rfp 0
 th 7
 ins 100.000
 ai cdc ph

DEC. & VT
 dfrq 125.672
 dn C13
 dpwr 30
 dof 0
 dm nnn
 dmm w
 dmf 10000
 dseq
 dres 1.0
 homo n
 PROCESSING
 wtfile
 proc ft
 fn 262144
 math f
 werr
 wexp
 wbs
 wnt
 wft

SAMPLE		DEC. & VT	
date	Apr 26 2007	dfrq	500.233
solvent	CDC13	dn	H1
file	/data/export/~	dpwr	37
home	/movassag/MVo	dof	-500.0
hm	/rocky/OA-II-242~	dm	y
	carbon.fid	dmm	w
		dmf	10000
ACQUISITION		dseq	
sfrq	125.796	dres	1.0
tn	Cl3	homo	n
at	1.736	PROCESSING	
np	131010	lb	0.30
sw	37735.8	wtfile	
fb	not used	proc	ft
bs	4	fn	131072
ss	1	math	f
tpwr	53	werr	
pw	6.8	wexp	
dl	0.763	wbs	
tof	631.4	wnt	
nt	1000		
ct	232		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2516.2		
wp	30189.9		
vs	404		
sc	0		
wc	250		
hzm	120.76		
is	500.00		
rfl	16005.3		
rfp	9711.2		
th	37		
ins	1.000		
ai	ph

```

date      Mar 2 2007
solvent   CDC13
file      /data/export/~
home/movassag/MVoac~
hm/bullwinkle/OA-I~
I-181.fid
ACQUISITION
sfrq      489.746
tn        H1
at        3.001
np        63050
sw        10504.2
fb        not used
bs        2
tpwr      56
pw        8.9
d1        2.000
tof       1519.5
nt        32
ct        8
alock     not used
gain      not used
flags
il        n
in        n
dp        y
hs        nn
DISPLAY
sp        -250.0
wp        6246.6
vs        63
sc        0
wc        250
hzmm      24.99
ls        33.57
rfl       1233.8
rfp       0
th        7
ins       100.000
ai        cdc
ph

```

```

DEC. & VT
dfrq      125.672
dn        C13
dpwr      30
dm        0
dmm       nnn
dmf       10000
dseq      1.0
dres      n
PROCESSING
wtfile
proc      ft
fn        131072
math      f
werr      wft
wexp      wft
wbs       wft
wnt       wft

```

Solvent: CDCl3
 Ambient temperature
 INOVA-500 "bullwinkle"
 Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 5 repetitions
 OBSERVE H1, 499.7417206 MHz
 DATA PROCESSING
 FI size 131072
 Total time 2 min, 8 sec

Relax. delay 0.750 sec
 Pulse 36.7 degrees
 Acq. time 2.000 sec
 Width 31397.2 Hz
 104 repetitions
 OBSERVE C13, 125.6601352 MHz
 DECOUPLE H1, 499.7442194 MHz
 Power 34 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 7663 hr, 35 min, 56 sec

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
5 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

Current Data Parameters
 NAME MH-VI-248
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070419
 Time 18.33
 INSTRUM spect
 PROBD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 48
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 8192
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.52 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127577 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

F2 - Acquisition Parameter:

Date_ 20070503
 Time 10.24
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 9
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====

NUC1 1H
 P1 15.07 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 65536
 SF 400.1300082 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

F2 - Acquisition Parameters

Date_ 20070503
 Time 20.14
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 117
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 6502
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====

NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters

SI 65536
 SF 100.6127532 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

SAMPLE date May 21 2007
 solvent CDC13
 file ACQUISITION exp
 sfreq 499.746
 tn H1
 at 3.001
 np 63050
 sw 10504.2
 fb not used
 bs 4
 tpwr 56
 pw 8.6
 dt 2.000
 tof 1519.5
 nt 32
 ct 8
 alock n
 gain not used
 il FLAGS
 in n
 dp y
 hs nn
 sp -252.6
 wp 6239.0
 vs 29
 sc 0
 wc 250
 hzmm 24.96
 ls 33.57
 rfl 4865.7
 rfp 3633.1
 th 7
 ins 100.000
 ai Cdc ph

DEC. & VT 125.672
 dn C13
 dpwr 30
 dof 0
 dm nm
 dmm w
 dmf 10000
 dseq 1.0
 dres n
 homo 22.0
 temp
 DEC2
 dfrq2 0
 dn2 1
 dpwr2 0
 dof2 0
 dm2 n
 dmm2 C
 dmf2 200
 dseq2 1.0
 dres2 n
 homo2
 DEC3
 dfrq3 0
 dn3 1
 dpwr3 0
 dof3 0
 dm3 n
 dmm3 C
 dmf3 200
 dseq3 1.0
 dres3 n
 homo3
 wtfile
 proc ft
 fn 262144
 math f
 werr
 wexp
 wbs
 wnt wft

PROCESSING

SAMPLE DEC. & VT
 date May 21 2007 dfrq 500.233
 solvent CDC13 dn H1
 file /data/export/~ dpwr 37
 home/movassag/MVoa~ dof -500.0
 hm/rocky/0A-II-298~ dm y
 -2carbon.fid dmm w
 ACQUISITION dmf 10000
 sfrq 125.796 dseq
 tn C13 dres 1.0
 at 1.736 homo n
 np 131010 PROCESSING
 sw 37735.8 lb 0.30
 fb not used wfile
 bs 8 proc ft
 ss 1 fn 131072 f
 tpwr 53 math
 pw 6.9
 d1 0.763 werr
 tof 631.4 wexp
 nt 2000 wbs
 ct 760 wnt
 alock n
 gain not used
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -2515.9
 wp 30189.9
 vs 1188
 sc 0
 wc 250
 hzmm 120.76
 is 500.00
 rfl 16002.4
 rfp 9715.0
 th 11
 ins 1.000
 ai ph

SAMPLE		DEC. & VT	
date	May 19 2007	dfrq	125.672
solvent	CDC13	dn	C13
file	/data/export/~	dpwr	30
home	/movassag/MVoa~	dof	0
hm	/bullwinkle/OA-I~	dm	nnn
	I-299.fid	dmm	w
ACQUISITION		dmf	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	temp	22.0
sw	10504.2	PROCESSING	
fb	not used	wfile	
bs	2	proc	ft
tpwr	56	fn	262144
pw	8.6	math	f
dl	2.000	werr	
tof	1519.5	wexp	
nt	32	wbs	
ct	8	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-249.9		
wp	6246.7		
vs	29		
sc	0		
wc	250		
hzmm	24.99		
is	33.57		
rfl	1233.8		
rfl	0		
th	7		
ins	100.000		
al	cdc	ph

SAMPLE date May 19 2007 DEC. & VT 500.233
 solvent CDC13 dn H1
 file /data/export/~ dpwr 37
 home/movassag/MVoa~ dof -500.0
 hm/rocky/OA-II-299~ dm y
 .fid dm 10000
 ACQUISITION sfreq 125.796 dseq
 tn C13 dres 1.0
 at 1.736 homo n
 np 131010 PROCESSING
 sw 3735.8 lb 0.30
 fb not used wfile
 bs 10 proc ft
 ss 1 fn 131072 f
 tpwr 53 math
 pw 6.9
 d1 0.763 werr
 tof 631.4 wexp
 nt 5000 wbs
 ct 1300 wnt
 alock n
 gain not used
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -2515.9
 wp 30189.9
 vs 628
 sc 0
 wc 250
 hzmm 120.776
 is 500.00
 rfl 16002.4
 rfp 9715.0
 th 4
 ins 1.000
 al ph

PULSE SEQUENCE
 Relax. delay 0.200 sec
 Pulse 69.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 816 repetitions
 OBSERVE C13, 125.7832268 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 26 minutes

Relax. delay 2.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
15 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 1 min, 20 sec

Relax. delay 0.050 sec
Pulse 36.7 degrees
Acq. time 2.000 sec
Width 31397.2 Hz
192 repetitions
OBSERVE C13, 125.6601366 MHz
DECOUPLE H1, 499.7442194 MHz
Power 34 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 131072
Total time 571 hr, 54 min, 55 sec

SAMPLE DEC. & VT
 date Apr 26 2007 dfrq 125.672
 solvent CDCl3 dn C13
 file /data/export/~ dpwr 30
 home/movassag/MVoac~ dof 0
 hm/bullwinkle/OA-I~ dm nnn
 I-270.fid dmm w
 ACQUISITION dmf 10000
 sfrq 499.746 dseq
 tn 499.746 dres 1.0
 at 3.001 homo n
 np 63050 PROCESSING
 sw 10504.2 wfile
 fb not used ft
 bs 2 fn 262144 f
 tpwr 56 math
 pw 8.6
 d1 2.000 werr
 tof 1519.5 wexp
 nt 32 wbs
 ct 8 wnt
 alock n
 gain not used wft
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -249.9
 wp 6246.7
 vs 24
 sc 0
 wc 250
 hzmm 24.99
 ls 33.57
 rfl 1233.8
 rfp 0
 th 7
 ins 1.000
 ai cdc ph

```

SAMPLE      DEC. & VT
date Apr 26 2007  dfrq 500.233
solvent CDCl3      dn   H1
file /data/export/~ dpwr 37
home/movassag/MVoa~ dof -500.0
hm/rocky/OA-II-270~ dm   y
carbon.fid      dmm   w
ACQUISITION  dmf      10000
sfrq 125.796  dseq
tn 013      dres 1.0
at 1.736    homo n
np 131010   PROCESSING
sw 3735.8   lb 0.30
fb not used wf file
bs 4        proc ft
ss 1        fn 131072
tpwr 53     math f
pw 6.9
dl 0.763    werr
tof 631.4    wexp
nt 1000     wbs
ct 96       wnt
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -2516.2
wp 30189.9
vs 162
sc 0
wc 250
hzmm 120.76
is 500.00
rfl 16005.3
th 9711.2
ins 11
ai 1.000
ph

```

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
11 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

PULSE SEQUENCE
 Relax. delay 0.200 sec
 Pulse 59.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 496 repetitions
 OBSERVE C13, 125.7832332 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 VALT2-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 16 minutes

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
6 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

PULSE SEQUENCE
 Relax. delay 0.200 sec
 Pulse 69.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 48 repetitions
 OBSERVE C13, 125.7832291 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 1 minutes

4u

Solvent: CDCl3
Ambient temperature
INOVA-500 "bullwinkle"
PULSE SEQUENCE
Pulse 85.8 degrees
Acq. time 3.277 sec
Width 9998.8 Hz
16 repetitions
OBSERVE H1, 499.7537710 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 52 sec

S83/S112

4u

PULSE SEQUENCE
 Relax. delay 0.763 sec
 Pulse 65.4 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 100 repetitions
 OBSERVE C13, 125.7832332 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 44 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 4 minutes

584/S112

ppm

0

20

40

60

80

100

120

140

160

180

200

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
4 repetitions
OBSERVE : H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

S85/S112

4v

PULSE SEQUENCE
 Relax. delay 0.763 sec
 Pulse 65.4 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 24 repetitions
 OBSERVE C13, 125.7832303 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 1 minute

4w

Solvent: CDCl3
Ambient temperature
File: mh-III-113
INOVA-500 "zippy"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 3.277 sec
Width 9998.8 Hz
16 repetitions
OBSERVE H1, 499.7446549 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 8 sec

4w

PULSE SEQUENCE
 Relax. delay 0.763 sec
 Pulse 65.4 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 16 repetitions
 OBSERVE C13, 125.7832458 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 1 minute

ppm
 588/S112

4x

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
4 repetitions
OBSERVE H1, 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

PULSE SEQUENCE
 Relax. delay 0.763 sec
 Pulse 65.4 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 56 repetitions
 OBSERVE C13, 125.7832332 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 2 minutes

4y

F2 - Acquisition Parameters

Date_ 20070503
 Time 20.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 14
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 287.4
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====

NUC1 1H
 P1 13.88 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 65536
 SF 400.1300154 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

ph

SAMPLE		DEC. & VT	
date	Feb 17 2007	dfrq	125.672
solvent	CDC13	dn	C13
file	/data/export/~	dpwr	30
home	/movassag/MVoa~	dof	0
hm	/bullwinkle/DA-1~	dm	nnn
I	1-166fr13-20.fid	dmm	w
ACQUISITION		dmm	10000
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050	PROCESSING	
sw	10504.2	wtfile	
fb	not used	proc	ft
bs	2	fn	131072
tpwr	56	math	f
pw	8.9		
d1	2.000	werr	
tof	1519.5	wexp	
nt	32	wbs	
ct	20	wnt	
alock	n		wft
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.0		
wp	6246.6		
vs	39		
sc	0		
wc	250		
hzmm	24.99		
is	33.57		
rfl	1233.8		
rfl	0		
rflp	0		
th	g		
ins	1.000		
ai	cdc	ph

4z

```

SAMPLE      DEC. & VT
date Mar 2 2007 dfrq 499.744
solvent CDCl3 dn H1
file /data/export/~ dpwr 34
home/movassag/MVoa~ dof 0
hm/bullwinkle/OA-I~ dm YVY
I-185carbon.fid dmm w
ACQUISITION dmf 10000
sfrq 125.672 dseq
tn 125.672 dres 1.0
at 2.000 homo n
np 125588 lb PROCESSING
sw 31397.2 lb 1.00
fb not used wtfile
bs 4 proc ft
tpwr 58 fn 131072
pw 6.7 math f
d1 3.000
tof 0 werr
nt 1000 wexp
ct 76 wbs
alock n wnt
gain not used
FLAGS
il n
in n
dp y
hs nm
DISPLAY
sp -2531.5
wp 30158.3
vs 629
sc 0
wc 250
hzmm 120.83
is 500.00
rfl 13470.9
rfp 9701.0
th 24
ins 100.000
ai cdc ph

```

4aa

F2 - Acquisition Parameters

Date_ 20070209
 Time 9.15
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 322.5
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====

NUC1 1H
 P1 13.88 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters

SI 65536
 SF 400.1300154 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

4aa

SAMPLE		DEC. & VT	
date	May 19 2007	dfrq	500.233
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	37
home	/movassag/MVoa~	dof	-500.0
hm/rocky	0A-II-151~	dm	y
carbon.fid		dmm	w
ACQUISITION		dmf	10000
sfrq	125.796	dseq	1.0
tn	C13	dres	n
at	1.736	homo	PROCESSING
np	131010	lb	0.30
sw	37735.8	wfile	ft
fb	not used	proc	131072
bs	10	fn	f
ss	1	math	
tpwr	53	werr	
pw	6.9	wexp	
d1	0.763	wbs	
tof	631.4	wnt	
nt	4000		
ct	840		
alock	not used		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nm		
DISPLAY			
sp	-2515.9		
wp	30189.9		
vs	287		
sc	0		
wc	250		
hzmm	120.76		
is	500.00		
rfl	16004.7		
rfl	9715.0		
th	20		
ins	1.000		
al	ph

date	Jan 29 2007	DEC. & VI	125.672
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home	/movassag/MVoa~	dof	0
hm	pullwinkle/OA-I~	dm	nnn
I-133fr7-12.fid	dmm	w	10000
ACQUISITION	dmf		
sfrq	499.746	dseq	
tn	H1	dres	1.0
at	3.001	homo	n
np	63050		
sw	10504.2	wtfile	PROCESSING
fb	not used	proc	ft
bs	2	fn	131072
tpwr	56	math	f
pw	8.9		
d1	2.000	werr	
tof	1519.5	wexp	
nt	32	wbs	
ct	16	wnt	wft
alock	not used		
gain			
il	n		
in	n		
dp	y		
hs	nn		
sp	-250.0		
wp	6246.6		
vs	57		
sc	0		
wc	250		
hzmm	24.99		
is	33.57		
rfl	4865.5		
rfp	3633.1		
th	60		
ins	1.000		
ai	cdc	ph

```

SAMPLE      date      Feb 7 2007
solvent      CDCl3
file /data/export/~
home/movassag/MVoa~
hm/bullwinkle/OA-I~
I-133carbon.fid
ACQUISITION dmm      10000
sfrq      125.672
tn         C13
at         2.000
np         125588
sw         31397.2
fb         not used
bs         10
tpwr      58
pw         6.7
d1         3.000
tof         0
nt         1000
ct         140
alock      n
gain       not used
flags      n
il         n
in         n
dp         y
hs         nn

DISPLAY
sp      -2529.6
wp      30158.3
vs      536
sc       0
wc       250
hzmm     120.63
is       500.00
rfl      13472.8
rfp      9704.7
th        68
ins      100.000
ai      cdc
ph

```

SAMPLE May 1 2007 DEC. & VT
 solvent CDC13 dfrq 125.672
 file /data/export/~ dn C13
 home/movassag/MVoaa~ dpwr 30
 hm/bullwinkle/OA-I~ dof 0
 I-99clean.fid dm nnn
 ACQUISITION dmf 10000 w
 sfrq 499.746 dseq 1.0
 tn 3.001 dres n
 at 63050 homo
 np 10504.2 wtfile
 sw not used proc ft
 fb 2 fn 262144
 bs 56 math f
 tpwr 8.6
 pw 2.000 werr
 d1 1519.5 wexp
 tof 32 wbs
 nt 14 wnt
 ct gain not used wft
 alock
 gain not used
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp ~249.9
 wp 6246.7
 vs 34
 SC 0
 WC 250
 hzmm 24.88
 ls 33.57
 rfl 4865.5
 rfp 3633.1
 th 3.000
 ins
 ai cdc ph

```

date      Feb 7 2007
SAMPLE    DEC. & VT
solvent    CDCl3
file       /data/export/~
home/movassag/MVoa~
hm/rocky/OA-II-99g~
carbon.fid
ACQUISITION
sfrq      125.796
in         C13
at         1.736
np         131010
sw         37735.8
fb         not used
bs         8
ss         1
tpwr       53
pw         6.9
d1         0.763
tof        631.4
nt         1000
ct         0
alock      not used
gain       not used
il         n
in         n
dp         y
hs         nn
DISPLAY
sp         -2515.9
wp         30189.9
vs         802
sc         0
wc         250
hzmm       120.76
is         500.00
rf1        16003.0
th         9715.0
ins        17
ai         1.000
ph

```

SAMPLE DEC. & VT
 date Apr 30 2007 dfreq 125.672
 solvent CDC13 dn C13
 file /data/export/~ dpwr 30
 home/movassag/MVoa~ dof 0
 hm/bullwinkle/OA-I~ dm nnn
 I-276fr37-52.fid dmm w
 ACQUISITION dmf 10000
 sfrq 499.746 dseq
 tn h1 dres 1.0
 at 3.001 homo n
 np 63050
 sw 10504.2 wfile
 fb not used proc ft
 bs 2 fn 262144 f
 tpwr 56 math
 pw 8.6
 dl 2.000 werr
 tof 1519.5 wexp
 nt 32 wbs
 ct 8 wnt
 alock n
 gain not used
 FLAGS
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -249.9
 wp 7246.2
 vs 53
 sc 0
 wc 250
 hzmm 28.96
 is 33.57
 rfl 4867.1
 rfp 3633.1
 th 7
 ins 100.000
 ai cdc ph

8

SAMPLE DEC. & VT
 date May 1 2007 dfrq 500.233
 solvent CDC13 dn H1
 file /data/export/~ dpwr 37
 home/movassag/MVoa~ dof -500.0
 hm/rocky/0A-II-274~ dm y
 carbon.fid dmm w
 ACQUISITION dmf 10000
 sfrq 125.796 dseq
 tn C13 dres 1.0
 at 1.736 homo n
 np 131010 PROCESSING
 sw 37735.8 lb 0.30
 fb not used wfile
 bs 4 proc ft
 ss 1 fn 131072 f
 tpwr 53 math
 pw 6.9 werr
 dl 0.763 wepp
 tof 631.4 wbs
 nt 2000 wnt
 ct 0
 alock n
 gain not used
 il n
 in n
 dp y
 hs nn
 DISPLAY
 sp -2515.9
 wp 30189.9
 vs 556
 sc 0
 wc 250
 hzmm 120.76
 ls 500.00
 rfl 16003.5
 rfp 9715.0
 th 20
 ins 1.000
 al ph

4dd

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
2 repetitions
OBSERVE H1 499.7417206 MHz
DATA PROCESSING
F1 size 262144
Total time 2 min, 8 sec

4dd

PULSE SEQUENCE
 Relax. delay 0.050 sec
 Pulse 69.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 208 repetitions
 OBSERVE C13, 125.7832314 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT size 131072
 Total time 6 minutes

9

Relax. delay 5.000 sec
Pulse 89.0 degrees
Acq. time 3.001 sec
Width 10504.2 Hz
7 repetitions
OBSERVE H1 499.7417206 MHz
DATA PROCESSING
FT size 262144
Total time 2 min, 8 sec

Relax. delay 0.050 sec
 Pulse 36.7 degrees
 Acq. time 2.000 sec
 Width 31397.2 Hz
 216 repetitions
 OBSERVE C13, 125.6601362 MHz
 DECOUPLE H1, 499.7442194 MHz
 Power 34 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 571 hr, 54 min, 55 sec

PULSE SEQUENCE
 Relax. delay 5.000 sec
 Pulse 89.0 degrees
 Acq. time 3.001 sec
 Width 10504.2 Hz
 8 repetitions
 OBSERVE H1 499.7417206 MHz
 DATA PROCESSING
 F1 size 262144
 Total time 2 min, 8 sec

PULSE SEQUENCE
 Relax. delay 0.763 sec
 Pulse 69.0 degrees
 Acq. time 1.736 sec
 Width 37735.8 Hz
 192 repetitions
 OBSERVE C13, 125.7832337 MHz
 DECOUPLE H1, 500.2332753 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Resol. enhancement -0.0 Hz
 FT size 131072
 Total time 8 minutes

=====
 Area Percent Report
 =====

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 C, Sig=300,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	55.044	MM	2.1536	81.62379	6.31687e-1	2.8197
2	61.242	MM	2.5261	2813.19092	18.56096	97.1803

Totals : 2894.81470 19.19264

Results obtained with enhanced integrator!

=====
 *** End of Report ***

=====
 Area Percent Report
 =====

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 C, Sig=300,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	56.095	VV	1.5770	9719.56055	85.58348	37.3632
2	58.588	VB	2.3944	1.62942e4	89.23138	62.6368

Totals : 2.60138e4 174.81486

Results obtained with enhanced integrator!

=====
 *** End of Report ***

=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.603	MM	1.1097	3.83898e4	576.55585	96.7799
2	32.488	MM	1.1118	1277.32971	19.14890	3.2201

Totals : 3.96672e4 595.70474

Results obtained with enhanced integrator!

=====
*** End of Report ***

=====
 Area Percent Report
 =====

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.778	MM	0.9852	2.24874e4	380.43207	49.6411
2	31.971	MM	1.1508	2.28126e4	330.39188	50.3589

Totals : 4.53000e4 710.82394

Results obtained with enhanced integrator!

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 *** End of Report ***