Supporting Information
for DOI: 10.1055/s-0036-1588729
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A merged aldol condensation, alkene isomerization, cycloaddition / cycloreversion sequence employing oxazinone intermediates for the synthesis of substituted pyridines

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Supporting Information

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General Information. All reactions were carried out under an atmosphere of nitrogen in flame-dried or oven-dried glassware with magnetic stirring unless otherwise indicated. THF, toluene, and Et₂O were degassed with argon and purified by passage through a column of molecular sieves and a bed of activated alumina. Dichloromethane (CH₂Cl₂) was distilled from CaH₂ prior to use. All reagents were used as received unless otherwise noted. Flash column chromatography was performed using SiliCycle siliaflash P60 silica gel (230–400 mesh). Analytical thin layer chromatography was performed on SiliCycle 60Å glass plates. Visualization was accomplished with UV light, anisaldehyde, ceric ammonium molybdate, potassium permanganate followed by heating. Film infrared spectra were recorded using a Digilab FTS 7000 or Shimadzu IRTracer-100 FTIR spectrophotometer. Optical rotations were determined on either a Perkin-Elmer 341 polarimeter at 25 °C. ¹H NMR spectra were recorded on a Varian Mercury 400 (400 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 7.26 ppm) or tetramethylsilane (0.00 ppm). Proton-decoupled ¹³C-NMR spectra were recorded on a Mercury 400 (100 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 77.00 ppm). All compounds were judged to be homogeneous (>95% purity) by ¹H and ¹³C NMR spectroscopy. Mass spectra data analysis was obtained through positive electrospray ionization (w/ NaCl) on a Bruker 12 Tesla APEX–Qe FTICR-MS with an Apollo II ion source.

2-methoxy-2-oxoethyl 2-azidoacetate. A dry flask was charged with chloroacetic acid (2.06 g, 21.8 mmol) and dissolved in water (20 mL). NaN₃ (2.70 g, 41.6 mmol) was added and the reaction vessel was heated to 40 °C for 24 h. The reaction was diluted and made acidic with 1 M HCl (25 mL) and extracted with Et₂O (3 x 50 mL). The combined organic layers were washed with brine (10 mL), dried (Na₂SO₄), filtered, and concentrated in vacuo. The resulting oil (2.034 g, 92% yield) was >95% pure azidoacetic acid as judged by ¹H NMR and used without further purification.

A portion of the azidoacetic acid (0.890 g, 8.80 mmol) was dissolved in butanone (15 mL) and methyl bromoacetate (1.26 mL, 13.2 mmol) and K₂CO₃ (1.82 g, 13.2 mmol) were added in succession to the reaction mixture. The reaction vessel was warmed to 40 °C and stirred for 22 h. The reaction was cooled to RT, diluted with Et₂O, and filtered (to remove inorganic salts). The filtrate was washed with H₂O (10 mL) and sat. aq. NaHCO₃ (10 mL). The organic layer was removed and the aqueous portion was extracted with additional Et₂O (3 x 10 mL). The combined organic layers were washed with brine (10 mL), dried with Na₂SO₄, filtered, and concentrated in vacuo. The resulting product (2-methoxy-2-oxoethyl 2-azidoacetate) was obtained as a colorless oil (1.35 g, 89% yield) was >95% pure as judged by ¹H NMR and used without further purification. Spectral data agrees with published values.³

5-methoxy-3,6-dihydro-2H-1,4-oxazin-2-one (5a). A dry flask was charged with 2-methoxy-2-oxoethyl 2-azidoacetate (3.20 g, 18.5 mmol), fitted with a Dean-Stark apparatus and condenser, and flushed with nitrogen. The starting material was dissolved in toluene (100 mL) and PPh₃ (4.85 g, 18.5 mmol) was added. After visible evolution of N₂ ceased, the reaction was heated to 90 °C. After 16 h at 90 °C, the reaction was cooled to RT and concentrated in vacuo. The resulting black residue was purified by Kügelrohr distillation (1 mmHg, 150 °C) to afford the title compound 5a (0.650 g, 27% yield) as a clear colorless oil: IR (film) 1678, 1437, 1188, 1119, 719, 694 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.82 (s, 2H), 4.23 (s, 2H), 3.79 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 167.1, 159.8, 64.9, 53.6, 47.4; HRMS Exact mass calc’d for C₉H₇NNaO₃⁺ [M+Na⁺] = 152.0318, found 152.0319.

methyl 2-(2-chloroacetoxy)propanoate. Methyl lactate (4.59 mL, 48.1 mmol) was dissolved in CH₂Cl₂ (100 mL). Pyridine (7.75 mL, 96.2 mmol) was introduced to the solution and the reaction vessel was cooled to 0 °C. Chloroacetyl chloride (4.17 mL, 52.9 mmol) was added

dropwise to the solution over 1 h. After stirring for 2.5 h, the reaction was warmed to RT, diluted with 1.0M HCl (50 mL) and extracted with CH₂Cl₂ (50 mL). The organic layer was washed with sat. aq. NaHCO₃ (50 mL), dried (Na₂SO₄), filtered, and concentrated in vacuo. The resulting residue (8.26 g) was purified by flash column chromatography on silica gel (gradient elution: 0→40% EtOAc in hexane) to afford the title compound (7.32 g, 83% yield) as clear oil:

**TLC** (40% EtOAc in hexane), Rₜ = 0.6 (KMnO₄); **IR** (film) 1744, 1167, 1094, 1045, 980, 789, 704 cm⁻¹; **¹H NMR** (400MHz, CDCl₃): δ 5.20 (q, J = 7.0 Hz, 1H), 4.19 (s, 2H), 3.97 (s, 3H), 1.57 (d, J = 7.0 Hz, 3H); **¹³C NMR** (100MHz, CDCl₃): δ 170.4, 166.7, 69.9, 52.4, 40.5, 16.7; **HRMS** exact mass calc’d for C₆H₉ClO₄Na⁺ [M+Na⁺] = 203.0082, found 203.0083.

Methyl 2-(2-azidoacetoxoxy)propanoate. Methyl 2-(2-chloroacetoxoxy)propanoate (10.2 g, 41.9 mmol) was dissolved in butanone (160 mL), NaN₃ (5.45 g, 83.8 mmol) was added, and the reaction was heated to 80 °C. After 16 h, the reaction was cooled to RT, diluted with Et₂O, filtered, and the filtrate was concentrated in vacuo. The resulting clear oil (7.75 g, 99% yield) was >95% pure as judged by ¹H NMR and used without further purification.

5-methoxy-6-methyl-3,6-dihydro-2H-1,4-oxazin-2-one (5b). Prepared in a manner analogous to the preparation of 5a. The concentrated residue was purified by flash column chromatography on silica gel (gradient elution: 20→100% EtOAc in hexane) to afford product 5b (180 mg, 37% yield) as a colorless oil: **IR** (film) 1748, 1695, 1456, 1206, 1080, 1043, 773 cm⁻¹; **¹H NMR** (400MHz, CDCl₃): δ 4.92 (q, J = 1.6 Hz, 1H), 4.26 (s, 2H), 3.76 (s, 3H), 1.57 (d, J = 3.9 Hz, 3H); **¹³C NMR** (100MHz, CDCl₃): δ 167.7, 162.5, 72.70, 53.6, 47.7, 18.4; **HRMS** exact mass calc’d for C₆H₆NO₃Na⁺ [M+Na⁺] = 166.0475, found 166.0475.

Methyl 2-(2-chloroacetoxoxy)-2-phenylacetate. Methyl mandelate (5.00 g, 30 mmol) was dissolved in CH₂Cl₂ (100 mL). Pyridine (4.84 mL, 0.060 mol) was introduced to the solution and the reaction vessel was cooled to 0 °C. Chloroacetyl chloride (2.60 mL, 30 mmol) was added dropwise to the solution over 1 h. After 2 h, the reaction was warmed to RT, diluted with 1M HCl (30 mL) and extracted with CH₂Cl₂ (30 mL). The organic layer was then washed with NaHCO₃ (30 mL), dried (Na₂SO₄), filtered, and concentrated in vacuo. The resulting residue (8.25 g) was purified by flash column chromatography on silica gel (gradient elution: 0→30% EtOAc in hexane) to afford the title compound (6.47 g, 89% yield) as a clear oil: **TLC** (40% EtOAc in hexane) Rₜ = 0.6 (CAM); **IR** (film) 1748, 1217, 1155, 1038, 735 cm⁻¹; **¹H NMR**
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(400MHz, CDCl$_3$): $\delta$ 7.46 (m, J = 3.2 Hz, 2H), 7.41 (m, J = 3.2 Hz, 3H), 6.01 (s, 1H), 4.21 (q, J = 15.2 Hz, 2H), 3.74 (s, 3H); $^{13}$C NMR (100MHz, CDCl$_3$): $\delta$ 168.5, 166.8, 133.0, 129.6, 128.9, 127.7, 75.6, 52.8, 40.6; HRMS exact mass calc’d for C$_{11}$H$_{11}$ClO$_4$Na$^+$ [M+Na$^+$] = 265.0204, found 265.0239.

Methyl 2-(2-azidoacetoxy)-2-phenylacetate. Methyl 2-((chlorocarbonyl)oxy)-2-phenylacetate (5.27 g, 21.8 mmol) was dissolved in butanone (140 mL), and NaN$_3$ (3.79 g, 58 mmol) was added, and the reaction was heated to 80 °C. After 24 h, the reaction vessel was cooled to RT, diluted with Et$_2$O, filtered, and the filtrate was concentrated in vacuo. The resulting clear oil (5.10 g, 94% yield) was >95% pure as judged by $^1$H NMR and used without further purification.

5-methoxy-6-phenyl-3,6-dihydro-2H-1,4-oxazin-2-one (5c). Prepared using a procedure modified slightly from the preparation of 5a and 5b: following addition of PPh$_3$, the reaction was heated to reflux (110 °C) for 24 h. The concentrated residue was purified by flash column chromatography on silica gel (gradient elution: 5→50% EtOAc in Hexanes with 50% isocratic toluene additive) to afford the title compound (264 mg, 57% yield) as slightly yellow oil: TLC (15% EtOAc, 35% hexanes, 50% toluene), $R_f$ = 0.33 (CAM); IR (film) 1754, 1705, 1378, 1050 cm$^{-1}$; $^1$H NMR (400MHz, CDCl$_3$) $\delta$ 7.43 (m, J = 6.2 Hz, 3H), 7.33 (m, J = 7.4 Hz, 2H), 5.85 (s, 1H), 4.41 (dd, J = 20.7 Hz, 2H), 3.80 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.6, 161.1, 134.3, 129.4, 126.4, 76.7, 53.9, 47.7; HRMS Exact mass calc’d for C$_{11}$H$_{11}$NO$_3$ [M+Na$^+$] = 228.0631, found 228.0632.

General procedure for the domino reaction leading to tricyclic pyridine products 13-15: dihydrooxazinone 5a, 5b, or 5c (0.3–0.6 mmol) was dissolved in toluene (0.12–0.15M) and DBU (1.5 equiv) was added. The reaction vessel was heated in an oil bath to a gentle reflux (110 °C) and 2-alkynylbenzaldehyde 10, 11, or 12 (1.5 equiv) in 1.0 mL of toluene was introduced slowly to the reaction over 2 h (using a syringe pump). After stirring for 16-22 h at 110 °C, the reaction was cooled to RT, transferred to a separatory funnel, and partitioned between sat. aq. NH$_4$Cl (10 mL) and EtOAc (10 mL). The organic layer was removed and the aqueous portion was extracted.
with EtOAc (3 x 10 mL). The combined organic layers were washed with brine (10 mL), dried (Na$_2$SO$_4$), filtered through Celite, and concentrated in vacuo. The resulting residue was purified by flash column chromatography on silica gel.


4-butyl-2-methoxy-9H-indeno[2,1-b]pyridine (13b). Pyridine 13b was prepared from 5a and 11 following the general procedure. Purification by flash column chromatography on silica gel (gradient elution: 3%→6% EtOAc in hexanes with an isocratic 2% AcOH additive) afforded the title compound 13c (7.7 mg, 13%) as a light yellow oil: TLC (10% CHCl$_3$, 5% EtOAc, 85% hexane) $R_f = 0.24$ (KMnO$_4$); IR (film): 2956, 1597, 1570, 1381, 1050 cm$^{-1}$; $^1$H NMR (400MHz, CDCl$_3$) $\delta$ 7.70 (d, J = 7.8 Hz, 1H), 7.55 (d, J = 7.4 Hz, 1H), 7.37 (t, J = 7.4 Hz, 1H), 7.28 (t, J = 8.2 Hz, 1H), 6.53 (s, 1H), 4.00 (s, 3H), 3.89 (s, 2H), 2.97 (t, J = 7.9 Hz, 2H), 1.74 (m, J = 2.3 Hz, 2H), 1.70 (t, J = 2.0 Hz, 2H), 1.51 (m, J = 7.4 Hz, 2H), 0.97 (t, J = 7.0 Hz, 3H); $^13$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.9, 163.3, 147.7, 140.7, 139.7, 138.6, 128.6, 128.4, 128.3, 126.4, 126.1, 125.8, 124.7, 121.8, 109.4, 53.8, 38.7; HRMS exact mass calc’d for C$_{17}$H$_{19}$NONa$^+ [M + Na]^+$ = 276.1359, found 276.1360.

2-methoxy-4-phenyl-9H-indeno[2,1-b]pyridine (13c). Pyridine 13c was prepared from 5a and 12 following the representative procedure described in the manuscript for the preparation of 13a. Purification by flash column chromatography on silica gel (gradient elution: 0→20% EtOAc in hexane) afforded the title compound 13b (6.0 mg, 10% yield) as a yellow-tinted powder: TLC (10% EtOAc/Hexanes) $R_f = 0.55$ (KMnO$_4$); IR (film): 2945, 1591, 1460, 1374, 1351, 1214 cm$^{-1}$; $^1$H NMR (400MHz, CDCl$_3$) $\delta$ 7.51 (m, J = 7.4 Hz, 4H), 7.19 (t, J = 7.4 Hz, 2H), 7.08 (t, J = 7.4 Hz, 1H), 6.97 (d, J = 7.4 Hz, 1H), 6.60 (s, 1H), 4.03 (s, 3H), 3.94 (s, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 163.6, 163.3, 147.7, 140.7, 139.7, 138.6, 128.6, 128.4, 128.3, 126.4, 126.1, 125.8, 124.7, 121.8, 109.4, 53.8, 38.7; HRMS exact mass calc’d for C$_{19}$H$_{15}$NONa [M+Na]$^+$ = 296.1045, found 296.1044.
2-methoxy-3-methyl-9H-indeno[2,1-b]pyridine (14a). Pyridine 14a was prepared from 5b and 10 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 10 in toluene to the reaction mixture was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0→20% EtOAc in hexanes) to afford the title compound 14a (11 mg, 47% yield) as a light yellow oil: TLC (40% EtOAc/hexanes) R_f = 0.71 (KMnO_4); IR (film) 1576, 1457, 1393, 1339 cm\(^{-1}\); \(^1\)H NMR (400MHz, CDCl\(_3\)) \(\delta\) 7.73 (s, 1H), 7.61 (d, J=7.4 Hz, 1H), 7.51 (d, J=7.4 Hz, 1H), 7.35 (t, J = 7.4 Hz, 1H), 7.25 (t, J=7.4 Hz, 1H), 4.04 (s, 3H), 3.84 (s, 2H), 2.27 (s, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 162.2, 159.4, 140.7, 140.2, 130.2, 128.3, 126.8, 125.7, 124.9, 119.1, 118.7, 53.67, 38.3, 16.4; HRMS Exact mass calc’d for C\(_{14}\)H\(_{14}\)NO\(^+\) [M+H]\(^+\) = 212.1070, found 212.1071.

4-butyl-2-methoxy-3-methyl-9H-indeno[2,1-b]pyridine (14b). Pyridine 14b was prepared from 5b and 11 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 11 in toluene to the reaction mixture was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0→10% EtOAc in hexane) afforded the title compound (31 mg, 54% yield) as a light yellow oil: TLC (5% EtOAc in hexane) R_f = 0.34 (KMnO_4); IR (film) 1576, 1453, 1377, 1339 cm\(^{-1}\); \(^1\)H NMR (400MHz, CDCl\(_3\)) \(\delta\) 7.74 (d, J = 7.4 Hz, 1H), 7.55 (d, J = 7.4 Hz, 1H), 7.37 (t, J = 7.4 Hz, 1H), 7.28 (t, J=7.4 Hz, 1H), 4.02 (s, 3H), 3.85 (s, 2H), 3.05 (t, J = 7.4 Hz, 2H), 2.24 (s, 3H), 1.58 (m, J = 7.4 Hz, 4H), 1.02 (t, J = 7.4 Hz, 3H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 161.8, 159.3, 146.8, 141.1, 140.9, 126.8, 126.7, 125.1, 124.9, 119.1, 118.7, 53.67, 38.3, 16.4; HRMS Exact mass calc’d for C\(_{18}\)H\(_{22}\)NO\(^+\) [M+H]\(^+\) = 268.1696, found 268.1697.

2-methoxy-3-methyl-4-phenyl-9H-indeno[2,1-b]pyridine (14c). Pyridine 14c was prepared from 5b and 12 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 12 in toluene to the reaction mixture was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0→50% EtOAc in hexane with an isocratic 50% toluene additive) afforded the title compound (46 mg, 54% yield) as a light yellow oil: TLC (20% EtOAc/hexanes) R_f = 0.29 (KMnO_4); IR (film) 1567, 1374, 1345 cm\(^{-1}\); \(^1\)H NMR (400MHz, CDCl\(_3\)) \(\delta\) 7.55 (m, J=7.4 Hz, 4H), 7.28 (m, J = 7.4 Hz, 2H), 7.15 (t, J=7.4 Hz, 1H),
7.00 (t, J=7.4 Hz, 1H), 6.33 (d, J=7.4 Hz, 1H), 4.08 (s, 3H), 3.90 (s, 2H), 1.99 (s, 3H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)) \(\delta\) 162.0, 159.0, 146.0, 140.9, 140.4, 138.0, 128.9, 128.3, 127.9, 126.6, 126.4, 125.3, 124.6, 121.3, 116.7, 53.9, 38.4, 12.6; \(\text{HRMS}\) Exact mass calc’d for C\(_{20}\)H\(_{18}\)NO\(^+\) [M + H]\(^+\) = 288.1383, found 288.1384.

2-methoxy-3-phenyl-9H-indeno[2,1-b]pyridine (15a). Pyridine 15a was prepared from 5c and 10 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 10 in toluene to the reaction mixture was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0\(\rightarrow\)40% EtOAc in hexane) afforded the title compound (18 mg, 34% yield) as a yellow-tinted oil: \(\text{TLC}\) (10% EtOAc/hexanes) \(R_f=0.40\) (KMnO\(_4\)); \(\text{IR}\) (film) 1560, 1457, 1391, 1343 cm\(^{-1}\); \(^{1}\text{H NMR}\) (400MHz, CDCl\(_3\)) \(\delta\) 7.94 (s, 1H), 7.66 (m, J = 7.8 Hz, 1H), 7.60 (m, J = 7.8 Hz, 2H), 7.54 (m, J = 7.8 Hz, 1H), 7.47 (m, J = 7.8 Hz, 2H), 7.44 (m, J = 7.8 Hz, 1H), 7.33 (m, J = 7.8 Hz, 1H), 4.05 (s, 3H), 3.94 (s, 2H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)): \(\delta\) 161.3, 160.7, 140.6, 139.9, 137.5, 130.3, 129.4, 128.9, 127.4, 127.0, 125.0, 122.8, 119.4, 53.9, 38.5; \(\text{HRMS}\) Exact mass calc’d for C\(_{19}\)H\(_{16}\)NO\(^+\) [M+H]\(^+\) = 274.1226, found 274.1228.

4-butyl-2-methoxy-3-phenyl-9H-indeno[2,1-b]pyridine (15b). Pyridine 15b was prepared from 5c and 11 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 11 in toluene to the reaction mixture was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0\(\rightarrow\)10% EtOAc in hexane with an isocratic 50% toluene additive) afforded the title compound (25 mg, 50% yield) as a light yellow oil: \(\text{TLC}\) (10% EtOAc/hexanes) \(R_f=0.3\) (KMnO\(_4\)); \(\text{IR}\) (film) 1564, 1454, 1341 cm\(^{-1}\); \(^{1}\text{H NMR}\) (400MHz, CDCl\(_3\)) \(\delta\) 7.70 (d, J = 7.8 Hz, 1H), 7.59 (d, J = 7.5 Hz, 1H), 7.46 (t, J = 7.0 Hz, 2H), 7.40 (t, J = 7.4 Hz, 2H), 7.30 (m, J = 7.0 Hz, 3H), 3.95 (s, 2H), 3.93 (s, 3H), 2.76 (q, J = 8.2 Hz, 2H), 1.54 (m, J = 6.6 Hz, 2H), 1.30 (m, J = 7.4 Hz, 2H), 0.80 (t, J = 7.4 Hz, 3H); \(^{13}\text{C NMR}\) (100 MHz, CDCl\(_3\)): \(\delta\) 161.5, 147.1, 141.0, 136.2, 130.3, 128.1, 127.1, 127.0, 126.1, 125.0, 122.8, 119.4, 53.9, 38.5; \(\text{HRMS}\) Exact mass calc’d for C\(_{23}\)H\(_{24}\)NO [M+H]\(^+\) = 352.1672, found 352.1672.

2-methoxy-3,4-diphenyl-9H-indeno[2,1-b]pyridine (15c). Pyridine 15c was prepared from 5c and 12 following the representative procedure described in the manuscript for the preparation of 13a. Use of the syringe pump was unnecessary; addition of 12 in toluene to the reaction mixture
was performed by dropwise addition over 15 min. Purification by flash column chromatography on silica gel (gradient elution: 0→20% EtOAc in hexane with an isocratic 50% toluene additive) afforded the title compound (11 mg, 22% yield) as a yellow-tinted oil: **TLC** (10% EtOAc/hexanes) $R_f = 0.3$ (KMnO$_4$); **IR** (film) 1564, 1456, 1344 cm$^{-1}$; **$^1$H NMR** (400MHz, CDCl$_3$): $\delta$ 7.54 (d, J = 1.6 Hz, 1H), 7.29 (m, J = 1.6 Hz, 3H), 7.18 (m, J = 1.6 Hz, 3H), 7.13 (m, J = 1.6 Hz, 5H), 7.09 (t, J = 1.6 Hz, 1H), 6.43 (d, J = 7.8 Hz, 1H), 4.01 (s, 3H), 4.00 (s, 2H). **$^{13}$C NMR** (400 MHz, CDCl$_3$): $\delta$ 161.4, 161.0, 140.8, 140.2, 137.2, 135.4, 130.9, 129.1, 128.2, 127.5, 127.4, 126.8, 126.6, 126.5, 125.6, 124.7, 122.1, 121.7, 77.2, 54.1, 38.7; **HRMS** Exact mass calc’d for C$_{50}$H$_{38}$N$_2$O$_2$ $^+$ [M + M + Na]$^+$ = = 721.2826, found = 721.282568.
N

O

MeO

5a

400 MHz
CDCl₃

8 7 6 5 4 3 2 1 ppm

N

O

MeO

5a

100 MHz
CDCl₃

200 180 160 140 120 100 80 60 40 20 ppm
Supporting Information

5b

400 MHz
CDCl$_3$

5b

100 MHz
CDCl$_3$

ppm

ppm
Supporting Information

![Chemical Structures and NMR Spectra](image-url)

**Chemical Structure 5c**
- 400 MHz
- CDCl₃

**NMR Spectra**
- 0 ppm to 2 ppm
- 8 ppm to 7 ppm
- 6 ppm to 5 ppm
- 4 ppm to 3 ppm
- 2 ppm to 1 ppm

**Chemical Structure 5c**
- 100 MHz
- CDCl₃

**NMR Spectra**
- 200 ppm to 180 ppm
- 160 ppm to 140 ppm
- 120 ppm to 100 ppm
- 80 ppm to 60 ppm
- 40 ppm to 20 ppm
Supporting Information

15a
400 MHz
CDCl₃

NOMe
Ph

15a
100 MHz
CDCl₃

NOMe
Ph
$^{1}H$ NMR spectra of compound 15b (400 MHz, CDCl$_3$) and $^{13}C$ NMR spectrum of compound 15b (100 MHz, CDCl$_3$).