

1. General Information

Synthesis and techniques

Unless otherwise stated, all preparative scale reactions were conducted in oven dried (160°C) glassware with magnetic stirring using Schlenk-line techniques or in a glove box under an atmosphere of dry dinitrogen. Experiments on NMR tube were carried out in Teflon cap sealed NMR tubes (\varnothing 5mm). Solvents were purified by passage over an activated aluminum oxide column, followed by distillation from Na-benzophenone ketal (toluene, benzene, THF, hexanes, pentane) and degassed prior to use. Dichloromethane, CD_2Cl_2 , and α,α,α -trifluorotoluene were distilled from CaH_2 (followed by 3 freeze-pump-thaw cycles and stored over a mixture of 4Å molecular sieves). Toluene- d_8 and benzene- d_6 were degassed by 3 freeze-pump-thaw cycles and stored over activated 4Å molecular sieves. DMF was of DrySolv-quality and used as received. Solvents for chromatography and other syntheses were used as received from commercial sources and were at least of ACS reagent grade. Solvents for routine NMR spectroscopy experiments were used as received. Silica gel 60 (particle size 0.040 -0.063mm, 230 -400 mesh) was purchased from Silicycle. TLCs were run on silica gel coated aluminum plates with UV indicator (F254) obtained by EMD Chemicals, Inc. and analyzed by UV/VIS and stained using a cerium ammonium molybdate solution.

Reagents and materials

Reagents for imine synthesis were used as received without further purification unless otherwise noted 9-borabicyclo[3.3.1]nonane dimer and sodium bis(trimethylsilyl)amide (NaHMDS) were purchased from Aldrich, stored in a glove box, and used as received. $\text{Ph}_3\text{C}^+\text{B}(\text{C}_6\text{F}_5)_4^-$ was purchased from Strem stored in a glove box, and used as received. All silanes in this study were purchased from Aldrich, distilled from CaH_2 (followed by 3 freeze-pump-thaw cycles and stored over a mixture of 4Å molecular sieves) and stored in a glove box. Diphenyliodonium tetrafluoroborate,^[1] 1-H,^[2] and imine starting materials were prepared via known procedures.^[3-5] Quinoline derivatives were synthesized by a modified literature procedure by way of a Suzuki-Miyaura cross coupling from the corresponding 2-bromoquinoline and boronic acid.^[6]

Characterization

NMR spectra were recorded on Bruker Avance 300 (^1H : 300.13 ^{13}C : 75.47; TXI probe), Bruker Avance 400 (^1H : 400.13, ^{11}B : 128.38, ^{13}C : 100.62, ^{19}F : 376.50, ^{31}P : 161.98; BBI, BBFO and QNP probes), Bruker Avance 500 (^1H : 500.19, ^2H : 426.80, ^{11}B : 160.27, ^{13}C : 125.62; BBFO probe), Bruker NEO 500 (^1H : 500.19, ^2H : 426.80, ^{11}B : 160.27, ^{13}C : 125.62; BBFO probe), Bruker Avance 600 (^1H : 600.17, ^{11}B : 192.56, ^{13}C : 150.93; ^{31}P : 242.94, TBI probe) or Bruker NEO 700 (^1H : 700.13, ^{11}B : 224.63, ^{13}C : 176.05, ^{31}P : 283.42; BBFO probe) instruments operating at the denoted spectrometer frequency given in mega Hertz (MHz) for the specified nucleus. The samples were measured as solutions in the stated solvent at ambient temperature in non-spinning mode if not mentioned otherwise. To specify the signal multiplicity, the following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, qu = quintet, sept = septet, oct = octet, and m = multiplet; br. indicates a broad resonance. Shifts δ are reported in parts per million (ppm) relative to tetramethylsilane (TMS) as an external standard for ^1H and ^{13}C NMR spectra and calibrated against the solvent residual peak or in case of proteo-solvents against known solvent resonances.^[8] ^{11}B signals are calibrated against external $\text{BF}_3\cdot\text{OEt}_2$ and ^{19}F against CFCl_3 . Coupling constants J are given in Hertz (Hz). GC-MS measurements were performed on an Agilent Technologies GC 6850N/ MS 5975N VL MSD equipped with an Agilent Technologies HP-5MS column (length: 30m, 0.25mm inner diameter,

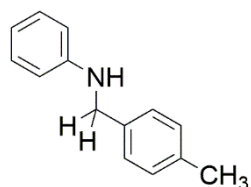
0.25 μ m coating thickness) coupled to a quadrupole mass filter. Helium was used as the carrier gas with a constant flow of 1.2mL/min. Separation of the injected species was achieved using the denoted temperature program and retention times t_R are given in minutes (min). High resolution mass-spectra (HRMS) were measured by the Queen's Mass Spectrometry and Proteomics Unit (MSPU) at Queen's University, Kingston, Ontario, Canada. Mass spectra were measured on Applied Biosystems/MDS Sciex QStar XL QqTOF or Waters ZQ Single Quad. Fragment signals are given in mass per charge number (m/z).

2. General procedure for the hydrosilylation of imines

In a glovebox, a 0.5 dram vial was charged with $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$ (0.01-0.05 eq.) and **1-H** (0.01-0.05 eq.) then 0.25 mL of DCM was added. The mixture was swirled until the colour changed from red to pale yellow. A J-Young screw-cap NMR tube or 4-dram vial was charged with the desired imine (1.0 eq.) and 0.25 mL of DCM. The solution in vial 1 was directly transferred to J-Young tube or 4-dram vial and agitated until dissolved. The hydrosilane (1.1–1.5 eq.) was added via an Eppendorf pipette directly to the tube. The reaction was monitored by ^1H NMR until complete reduction of the substrate was observed. The reaction mixture was transferred to a flask and diluted with 1 mL of DCM, followed by the addition of 5 drops of 15% aq. KOH solution and 10 mL of MeOH. After 1 h, any volatiles were removed under high vacuum and the crude mixture was purified by silica gel column chromatography to give the desired amine product. Performing the reactions in 4 dram scintillation vials with a stirbar produces results consistent with the NMR tube reactions.

Analytical data for reduced imines

N-(4-methylbenzyl)aniline (**4b**)



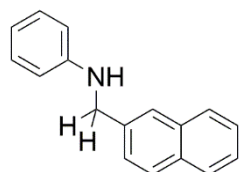
Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 21.5 h reaction time, room temperature

Yield (0.5 mmol, NMR tube) = 71 mg (71%); Yield (0.5 mmol, 4-dram vial) = 72 mg (73%).

Spectra were consistent with the literature: Sreedhar, B., Surendra Reddy, P., and Keerthi Devi, D., *J. Org. Chem.* **2009**, *22*, 8806-8809.

^1H NMR (400 MHz, CDCl_3) δ 7.38 (2H, d, $J = 7.9$ Hz), 7.26 – 7.31 (4H, m), 6.84 (1H, t, $J = 7.3$ Hz), 6.74 (2H, d, $J = 7.8$ Hz), 4.38 (2H, s), 4.06 (1H, br. s), 2.47 (3H, s)

N-(naphthalen-2-ylmethyl)aniline (**4c**)



Conditions: 3.3 mol% **1-H** + 3.3 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 2.5 h reaction time, room temperature

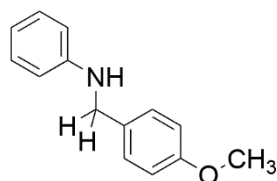
Yield (0.5 mmol) = 116 mg (95%)

Spectra were consistent with the literature: Gajare, A. S., Toyota, K., Yoshifuji, M., and Ozawa, F., *J. Org. Chem.* **2004**, *19*, 6504-6506

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 8.17-8.19 (1H, m), 8.01-8.03 (1H, m), 7.93 (1H, d, $J = 8.1$ Hz), 7.62-7.65 (3H, m), 7.54 (1H, t, $J = 8.1$ Hz), 7.34 (2H, t, $J = 7.7$ Hz), 6.90 (1H, t, $J = 7.2$ Hz), 6.77 (2H, d, $J = 8.4$ Hz), 4.80 (2H, s), 4.04 (1H, s).

$^{13}\text{C NMR}$ (126 MHz, CD_2Cl_2) δ 148.29, 134.42, 133.93, 131.60, 129.39, 128.84, 128.21, 126.38, 126.05, 125.90, 125.62, 123.65, 117.61, 112.79, 46.39.

N-(4-methoxybenzyl)aniline (4d)



Conditions: 1 mol% **1-H** + 1 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 24 h reaction time, 65 °C

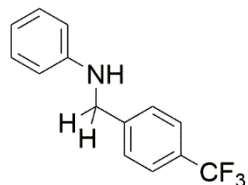
Yield (0.25 mmol) = 51 mg (96%)

Spectra were consistent with the literature: Bagan, D. B., Watile, R. A., Khedkar, M. V., Dhake, K. P., and Bhanage, B. M., *Catal. Sci. Technol.*, **2012**, *2*, 354-358

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.33 (2H, d, $J = 8.6$ Hz), 7.22 (2H, t, $J = 7.4$ Hz), 6.92 (2H, d, $J = 8.6$ Hz), 6.76 (1H, t, $J = 7.3$ Hz), 6.67 (2H, d, $J = 7.8$ Hz), 4.28 (2H, s), 3.98 (1H, br. s), 3.83 (3H, s)

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 158.99, 148.34, 131.55, 129.34, 128.90, 117.59, 114.15, 112.95, 55.38, 47.89.

N-(4-(trifluoromethyl)benzyl)aniline (4e)



Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 1 h reaction time, room temperature

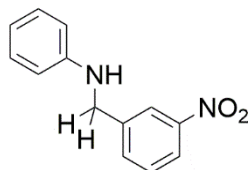
Yield (0.25 mmol, NMR tube) = 46 mg (71%); Yield (0.5 mmol, 4-dram vial) = 108 mg (86%).

Spectra match literature: Sousa, S. C. A. and Fernandes, A. C., *Adv. Synth. Catal.* **2010**, 352, 2218-2226

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.62 (2H, d, $J=8.0$ Hz), 7.51 (2H, d, $J=7.9$ Hz), 7.18-7.23 (2H, m), 6.77 (1H, tt, $J=7.4, 1.0$ Hz), 6.64 (2H, dd, $J=9.7, 1.0$ Hz), 4.43 (2H, s), 4.15 (1H, br. s)

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -63.00.

N-(3-nitrobenzyl)aniline (4f)



Conditions: 1 mol% **1-H** + 1 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 72 h reaction time, 65 °C

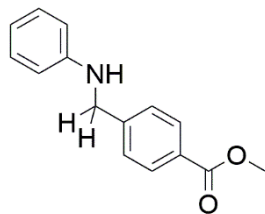
Yield (0.25 mmol)= 47 mg (82%)

Spectra were consistent with the literature: Waltz, F., Pillette, L., and Ambroise, Y., *ChemMedChem* **2011**, 6, 1775-1777

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 8.25 (1H, s), 8.13 (1H, d, $J=7.1$ Hz), 7.72 (1H, d, $J=7.6$ Hz), 7.51 (1H, t, $J=7.9$ Hz), 7.18 (2H, t, $J=8.4$ Hz), 6.75 (1H, t, $J=7.4$ Hz), 6.61 (2H, d, $J=7.8$ Hz), 4.47 (2H, s), 4.22 (1H, s).

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 148.76, 147.48, 142.17, 133.34, 129.72, 129.53, 122.42, 122.22, 118.39, 113.11, 47.70.

Methyl 4-((phenylamino)methyl)benzoate (4g)



Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 24 h reaction time, room temperature

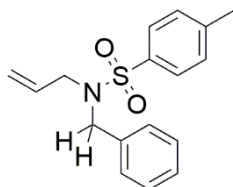
Yield (0.5 mmol)= 106 mg (87%)

Spectra were consistent with the literature: Sousa, S. C. A. and Fernandes, A. C., *Adv. Synth. Catal.* **2010**, 352, 2218-2226

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 8.04 (2H, d, $J=7.9$ Hz), 7.45 (2H, d, $J=7.8$ Hz), 7.20 (2H, t, $J=7.6$ Hz), 6.76 (1H, t, $J=7.6$ Hz), 6.63 (2H, d, $J=7.8$ Hz), 4.41 (2H, s), 4.21 (1H, br. s), 3.93 (3H, s).

^{13}C NMR (126 MHz, CDCl_3) δ 166.98, 147.85, 145.09, 129.97, 129.33, 129.07, 127.16, 117.82, 112.93, 52.09, 47.96.

N-allyl-N-benzyl-4-methylbenzenesulfonamide (4h)



Conditions: 3.3 mol% **1-H** + 3.3 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 48 h reaction time, room temperature

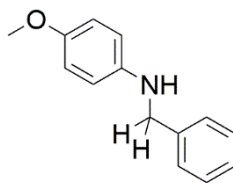
Yield (0.5 mmol)= 141 mg (86%)

Spectra were consistent with the literature: Ning, X-S., Wang, M-M., Yao, C-Z., Chen, X-M., and Kang, Y-B., *Org. Lett.*, **2016**, *11*, 2700-2703.

^1H NMR (499 MHz, CDCl_3) δ 7.75-7.78 (2H, m), 7.33-7.34 (1H, m), 7.31-7.33 (2H, m), 7.29-7.31 (1H, m), 7.26-7.30 (3H, m), 5.49 (1H, ddt, J = 16.7, 10.1, 6.5 Hz), 5.07 (1H, ddt, J = 10.1, 1.1, 1.1 Hz), 5.01 (1H, ddt, J = 17.1, 1.25, 1.25 Hz), 4.36 (2H, s), 3.78 (2H, d, J = 6.5 Hz), 2.45 (3H, s).

^{13}C NMR (126 MHz, CDCl_3) δ 143.34, 137.55, 136.04, 132.21, 129.78, 128.54, 128.49, 127.73, 127.22, 119.38, 50.23, 49.52, 21.55.

N-benzyl-4-methoxyaniline (4i)



Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 2.5 h reaction time, room temperature

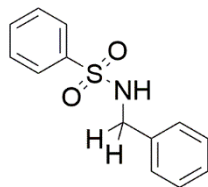
Yield (0.5 mmol)= 90 mg (84%)

Spectra were consistent with the literature: Martinez, R., Ramon, D. J., and Yus, M., *Org. Biomol. Chem.*, **2009**, *7*, 2176-2181

^1H NMR (400 MHz, CDCl_3) δ 7.32-7.41 (4H, m), 7.25-7.31 (1H, m), 6.77-6.81 (2H, m), 6.60-6.64 (2H, m), 4.30 (2H, s), 3.79 (1H, br. s), 3.75 (3H, s).

^{13}C NMR (126 MHz, CDCl_3) δ 152.22, 142.53, 139.80, 128.63, 127.58, 127.19, 114.96, 114.14, 55.80, 49.23.

N-benzylbenzenesulfonamide (4j)



Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 3 h reaction time, room temperature

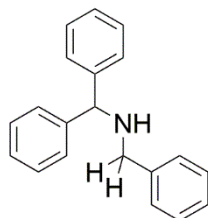
Yield (0.5 mmol)= 106 mg (85%)

Spectra were consistent with the literature: Cano, R., Ramon, D. J., Yus, M., *J. Org. Chem.*, **2011**, *14*, 5547-5557

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 7.87-7.90 (2H, m), 7.59 (1H, ttd, $J= 8.2, 1.25, 0.4$ Hz), 7.50-7.53 (2H, m), 7.26-7.30 (3H, m), 7.19-7.21 (2H, m), 4.96 (1H, t, $J= 5.7$ Hz), 4.15 (2H, d, $J= 6.3$ Hz).

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 140.03, 136.32, 132.81, 129.25, 128.81, 128.04, 127.98, 127.22, 47.38.

N-benzyl-1,1-diphenylmethanamine (4k)



Conditions: 1 mol% **1-H** + 1 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 1 h reaction time, room temperature

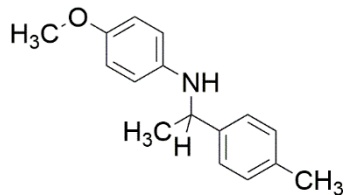
Yield (0.5 mmol)= 130 mg (96%)

Spectra were consistent with the literature: Likhar, P. R., Arundhathi, R., Lakshmi Kantam, M., and Sai Prathima, P., *Eur. J. Org. Chem.* **2009**, 5383-5389

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 7.42-7.45 (4H, m), 7.29-7.35 (8H, m), 7.24-7.28 (1H, m), 7.20-7.24 (2H, m), 4.87 (1H, s), 3.76 (2H, s)

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 144.14, 140.63, 128.64, 128.52, 128.31, 127.51, 127.18, 127.07, 66.60, 52.03.

4-methoxy-N-(1-(p-tolyl)ethyl)aniline (4l)



Conditions: 1 mol% **1-H** + 1 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 3 h reaction time, room temperature

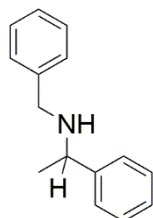
Yield (0.5 mmol)= 118 mg (96%)

Spectra were consistent with the literature: Fleischer, S., Zhou, S., Junge, K., and Beller, M., *Chem. Asian J.*, **2011**, *6*, 2240-2245

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 7.35 (2H, d, $J=7.8$ Hz), 7.22 (2H, d, $J=7.4$ Hz), 6.80 (2H, d, $J=8.7$ Hz), 6.58 (2H, d, $J=8.7$ Hz), 4.49 (1H, q, $J=6.4$ Hz), 3.86 (1H, br. s), 3.77 (3H, s), 2.42 (3H, s), 1.57 (3H, d, $J=6.6$ Hz).

$^{13}\text{C NMR}$ (126 MHz, CD_2Cl_2) δ 151.90, 142.58, 141.74, 136.33, 129.34, 125.86, 114.81, 114.60, 55.72, 53.96, 25.16, 21.11.

***N*-benzyl-1-phenylethan-1-amine (4m)**



Conditions: 5 mol% **1-H** + 5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 1 h reaction time, room temperature

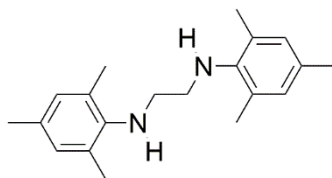
Yield (0.25 mmol)= 31 mg (60%)

Spectra were consistent with the literature: Wehn, P. M., and Du Bois, J., *Org. Lett.* **2005**, *21*, 4685-4688

$^1\text{H NMR}$ (499 MHz, CDCl_3) δ 7.25-7.41 (10H, m), 3.85 (1H, qd, $J=6.6, 2.2$ Hz), 3.71 (1H, dd, $J=13.1, 2.0$ Hz), 3.62 (1H, dd, $J=13.2, 2.2$ Hz), 1.68 (1H, br. s), 1.41 (3H, dd, $J=6.6, 2.4$ Hz)

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 145.72, 140.79, 128.60, 128.49, 128.26, 127.06, 126.97, 126.84, 57.63, 51.79, 24.63.

***N*¹,*N*²-dimesitylethane-1,2-diamine (4n)**



Conditions: 2.5 mol% **1-H** + 2.5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 0.5 h reaction time, room temperature

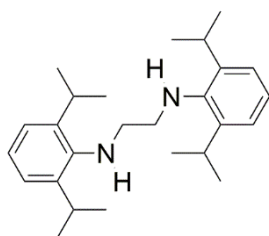
Yield (0.5 mmol)= 145 mg (97%)

Spectra were consistent with the literature: Papadaki, E., and Magrioti, V., *Tetrahedron* **2020**, 61, 151419

^1H NMR (400 MHz, CDCl_3) δ 7.05 (4H, s), 3.54 (2H, br. s), 3.37 (4H, s), 2.51 (12H, s), 2.46 (6H, s).

^{13}C NMR (101 MHz, CDCl_3) δ 143.40, 131.39, 129.74, 129.53, 49.19, 20.58, 18.42.

***N*¹,*N*²-bis(2,6-diisopropylphenyl)ethane-1,2-diamine (4o)**



Conditions: 2.5 mol% **1-H** + 2.5 mol% $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$, 8 h reaction time, room temperature

Yield (0.5 mmol)= 178 mg (92%)

Spectra were consistent with the literature: Papadaki, E., and Magrioti, V., *Tetrahedron* **2020**, 61, 151419

^1H NMR (400 MHz, CDCl_3) δ 7.10-7.21 (6H, m), 3.41 (6H, sept, J = 6.8 Hz), 3.21 (4H, s), 1.31 (24H, d, J = 6.8 Hz).

^{13}C NMR (101 MHz, CDCl_3) δ 143.47, 142.61, 123.97, 123.74, 52.45, 27.91, 24.39.

3. General procedure for the hydrosilylation of quinolines

In a glovebox, a 0.5 dram vial was charged with $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$ (0.05 eq.) and **1-H** (0.055 eq.) then 0.25 mL of DCM was added. The mixture was swirled until the colour changed from red to pale yellow. A J-Young screw-cap NMR tube was charged with the desired 2-aryl-quinoline (1.0 eq.), and 0.25 mL of DCM. The solution in vial **1** was directly transferred to the J-Young tube and agitated. PhMe_2SiH (1.5 eq.) was added *via* an Eppendorf pipette directly to the tube. The reaction was monitored by ^1H NMR, with almost complete reduction occurring in 24 h at room temperature. Once the hydrosilylation was completed, a

solution of the aldehyde (1.0 eq.) in DCM (0.3 mL) was added to the J-Young tube resulting in a colour change to yellow. Within 20 minutes, the aldehyde was completely consumed. The solution was then transferred to a round-bottom flask where MeOH (3 mL) and NaBH₄ (2 eq.) were added and the reaction was stirred under open atmosphere at room temperature for 1 h. Following this, THF (3mL), H₂O (6 mL) and acetic acid (6 mL), were added to the same flask and the mixture was stirred at room temperature for 1 h then saturated NaHCO_{3(aq)} was added dropwise until bubbling ceased. The aqueous layer was washed three times with DCM (3 x 10mL). The combined organic extracts were dried over NaSO₄, filtered and any volatiles were removed under high vacuum. The crude mixture was purified by silica gel column chromatography to give the desired products.

Analytical data for new aminoalcohols:

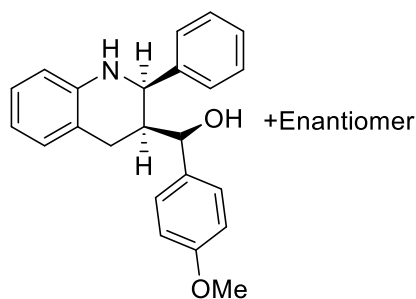
(4-methoxyphenyl)(2-phenyl-1,2,3,4-tetrahydroquinolin-3-yl)methanol (8a & 8a')

Conditions: 5.3 mol% **1-H** (18.5 mg, 0.0530 mmol), 5.0 mol% of [Ph₃C][B(C₆F₅)₄] (46.0 mg, 0.0500 mmol), 1.000 mmol of 2-phenylquinoline (207 mg), 1.500 mmol of PhMe₂SiH (230 μL), 1.000 mmol of 4-methoxybenzaldehyde (121 μL), 2.000 mmol of NaBH₄ (66.0 mg), 24 h reaction time, room temperature.

Crude diastereomeric ratio by ¹H NMR 8a:8a' = 30:70

Yield (1 mmol) 8a/8a' = 98 mg/171 mg (269 mg total, 77%, 36:64 diastereomeric ratio).

8a

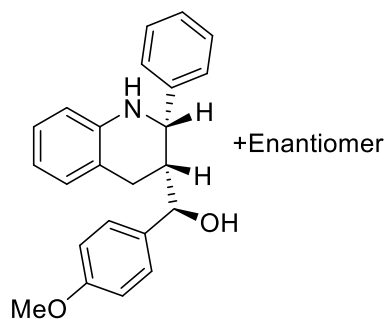


¹H NMR (400 MHz, CDCl₃) δ 7.32-7.40 (5H, m), 7.16 (2H, d, J= 8.6 Hz), 7.09 (1H, d, J= 7.6 Hz), 7.05 (1H, t, J= 7.4 Hz), 6.87 (2H, d, J= 8.7 Hz), 6.78 (1H, t, J= 7.3 Hz), 6.63 (1H, d, J= 7.8 Hz), 4.60 (1H, d, J= 4.7 Hz), 4.54 (1H, d, J= 3.4 Hz), 3.81 (3H, s), 3.03 (1H, dd, J= 17.2, 6.7 Hz), 2.88 (1H, d, J= 17.1, 5.9 Hz), 2.54 (1H, m)

¹³C NMR (126 MHz, CDCl₃) δ 158.96, 142.70, 135.15, 129.66, 128.80, 127.86, 127.71, 127.12, 126.77, 122.71, 119.6, 116.4, 114.0, 113.2, 75.0, 55.3, 55.0, 43.5, 25.6

HRMS (EI): calcd. for C₂₆H₂₃NO₂ 345.1729, found 345.1722

8a'



$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38-7.42 (2H, m), 7.28 – 7.33 (3H, m), 7.19 (2H, d, $J = 8.6$ Hz), 7.02 (1H, t, $J = 7.4$ Hz), 6.86 (2H, d, $J = 8.7$ Hz), 6.78 (1H, d, $J = 7.4$ Hz), 6.56-6.60 (2H, m), 5.08 (1H, d, $J = 3.9$ Hz), 3.94 (1H, d, $J = 10.4$ Hz), 3.81 (4H, m), 2.26 (1H, dd, $J = 16.1, 13.1$ Hz), 1.97 (1H, dd, $J = 16.2, 3.8$ Hz), 1.82 (1H, br. s).

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 159.61, 144.25, 143.21, 135.16, 129.63, 128.32, 127.96, 127.40, 127.31, 119.60, 116.50, 114.09, 113.23, 75.14, 55.42, 55.09, 43.66, 25.71

HRMS (EI): calcd. for $\text{C}_{26}\text{H}_{23}\text{NO}_2$ 345.1729, found 345.1715

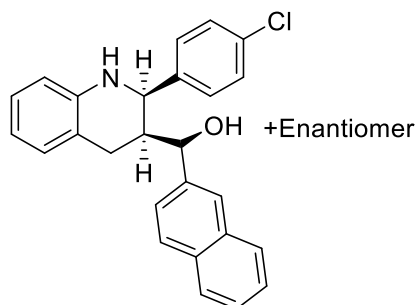
(2-(4-chlorophenyl)-1,2,3,4-tetrahydroquinolin-3-yl)(naphthalen-2-yl)methanol (8b & 8b')

Conditions: 5.5 mol% **1-H** (7.60 mg, 0.0220 mmol), 5.0 mol% of $[\text{Ph}_3\text{C}][\text{B}(\text{C}_6\text{F}_5)_4]$ (18.4 mg, 0.0200 mmol), 0.400 mmol of 2-(4-chlorophenyl)quinoline (95.9 mg), 0.600 mmol of PhMe_2SiH (92.0 μL), 0.400 mmol of 2-naphthaldehyde (62.5 mg), 0.800 mmol of NaBH_4 (30.3 mg), 24 h reaction time, room temperature.

Crude diastereomeric ratio by $^1\text{H NMR}$ 8b:8b' = 49:51

Yield (0.4 mmol) 8b/8b' = 54 mg/49 mg (103 mg total, 64%, 52:48 diastereomeric ratio)

8b

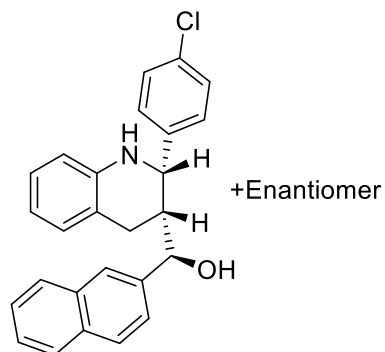


$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.79-7.85 (3H, m), 7.74 (1H, s), 7.48-7.52 (2H, m), 7.37 (2H, d, $J = 8.5$ Hz), 7.29 (2H, d, $J = 8.5$ Hz), 7.25 (1H, dd, $J = 8.4, 1.7$ Hz), 7.08 (2H, t, $J = 7.6$ Hz), 6.82 (1H, td, $J = 7.4, 1.2$ Hz), 6.64 (1H, d, $J = 7.8$ Hz), 4.78 (1H, d, $J = 4.4$ Hz), 4.54 (1H, d, $J = 3.5$ Hz), 4.17 (1H, br s), 3.54 (1H, br s), 3.04 (1H, dd, $J = 17.3, 7.0$ Hz), 2.90 (1H, dd, $J = 17.3, 5.4$ Hz), 2.66 (1H, m).

$^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 141.0, 140.4, 140.2, 133.7, 133.4, 133.0, 129.7, 129.0, 128.5, 128.2, 128.1, 127.8, 126.9, 126.3, 126.0, 125.5, 124.5, 122.7, 119.6, 115.4, 73.8, 58.6, 44.0, 25.5.

HRMS (EI): calcd. for $\text{C}_{26}\text{H}_{22}\text{NOCl}$ 399.1390, found 399.1398

8b'



¹H NMR (500 MHz, CDCl₃) δ 7.85 (2H, d, J= 8.6 Hz), 7.78-7.80 (1H, m), 7.64 (1H, s), 7.47-7.51 (3H, m), 7.38 (2H, d, J= 8.4 Hz), 7.29 (2H, d, J= 8.4 Hz), 7.01 (1H, t, J= 7.7 Hz), 6.69 (1H, d, J= 7.4 Hz), 6.53 -6.57 (2H, m), 5.12 (1H, d, J= 4.3 Hz), 4.52 (1H, br s), 4.10 (1H, d, J= 10.4), 2.72-2.79 (1H, m), 2.28 (1H, dd, J= 16.4, 13.1 Hz), 2.00 (1H, br s), 1.95 (1H, dd, J= 16.4, 4.1 Hz).

¹³C NMR (125 MHz, CDCl₃) δ 143.84, 141.63, 140.01, 133.52, 133.28, 133.12, 129.68, 129.40, 128.94, 128.45, 128.09, 127.90, 127.54, 126.55, 126.53, 126.38, 124.53, 119.24, 116.74, 113.27, 75.89, 54.45, 43.28, 25.51.

HRMS (EI): calcd. for C₂₆H₂₂NOCl 399.1390, found 399.1382

Single crystals for molecule **8b'** were grown by layering roughly 4 mL of pentane into a solution of **8b'** in roughly 0.5 mL of toluene, resulting in colorless plates.

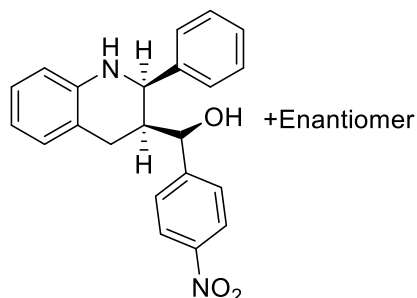
(4-nitrophenyl)(2-phenyl-1,2,3,4-tetrahydroquinolin-3-yl)methanol (8c & 8c')

Conditions: 5.3mol% **1-H** (7.55 mg, 0.0212 mmol), 5.0 mol% of [Ph₃C][B(C₆F₅)₄] (18.2 mg, 0.200 mmol), 0.4000 mmol of 2-phenylquinoline, 0.4400 mmol of Me₂PhSiH (68 μL), 0.4000 mmol of 4-nitrobenzaldehyde (60.3 mg), 0.8000 mmol of NaBH₄ (256 mg), 24 h reaction time, room temperature

Crude diastereomeric ratio by ¹H NMR 8c:8c' = 47:53

Yield (0.4 mmol) 8c/8c' = 30 mg/34 mg (64 mg total, 44%, 47:53 diastereomeric ratio)

8c

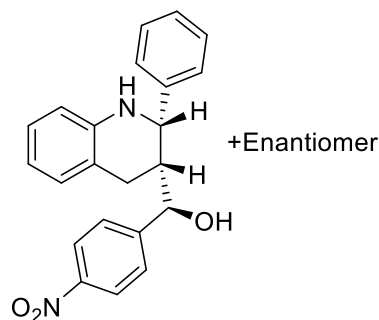


¹H NMR (500 MHz, CDCl₃) δ 8.14 (2H, d, J = 8.8 Hz), 7.47 – 7.52 (4H, m), 7.39-7.42 (1H, m), 7.35 (2H, d, J = 8.8 Hz), 7.06 (2H, m), 6.84 (1H, t, J = 7.5 Hz), 6.70 (1H, d, J = 7.9 Hz), 4.99 (1H, d, J = 1.6 Hz), 4.75 (1H, d, J = 3.3 Hz), 4.33 (1H, br. s), 4.21 (1H, br. s), 2.95 (1H, dd, J = 17.5, 7.8 Hz), 2.74 (1H, dd, J = 17.5, 2.1 Hz), 2.43-2.47 (1H, m).

¹³C NMR (100 MHz, CDCl₃) δ 151.0, 147.0, 144.1, 141.0, 129.5, 129.3, 128.3, 126.9, 126.7, 126.6, 123.5, 123.2, 120.5, 116.0, 72.6, 60.1, 44.6, 25.4.

HRMS (EI): calcd. for C₂₂H₂₀N₂O₃ 360.1474, found 360.1470

8c'



¹H NMR (400 MHz, CDCl₃) δ 8.19 (2H, d, J = 8.7 Hz), 7.41-7.45 (4H, m), 7.33 – 7.36 (3H, m), 7.04 (1H, t, J = 7.4 Hz), 6.77 (1H, d, J = 7.3 Hz), 6.57-6.62 (2H, m), 5.09 (1H, d, J = 3.8 Hz), 4.18 (1H, d, J = 9.8 Hz), 2.58-2.64 (1H, m), 2.37 (1H, dd, J = 15.9, 12.5 Hz), 1.96 (1H, dd, J = 16.0, 3.9 Hz).

¹³C NMR (126 MHz, CDCl₃) δ 150.3, 147.7, 143.8, 142.6, 129.5, 128.4, 127.9, 127.6, 127.6, 123.8, 118.7, 116.8, 113.4, 74.6, 60.4, 54.9, 21.1

HRMS (EI): calcd. for C₂₂H₂₀N₂O₃ 360.1474, found 360.1479

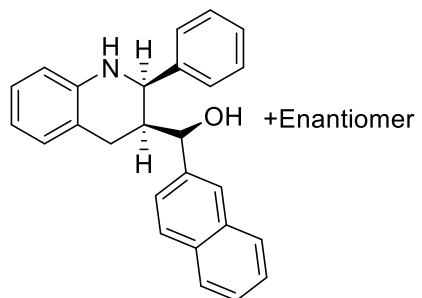
Naphthalen-2-yl(2-phenyl-1,2,3,4-tetrahydroquinolin-3-yl)methanol (8d)

Conditions: 5.5 mol% **1-H** (18.9 mg, 0.055 mmol), 5.0 mol% of [Ph₃C][B(C₆F₅)₄] (46.1 mg, 0.0500 mmol), 1.000 mmol of 2-phenylquinoline (205 mg), 1.500 mmol of PhMe₂SiH (230 μL), 1.000 mmol of 2-naphthaldehyde (156 mg), 2.000 mmol of NaBH₄ (66.0 mg), 24 h reaction time, room temperature.

Crude diastereomeric ratio by ¹H NMR **8d**:**8d'** = 40:60

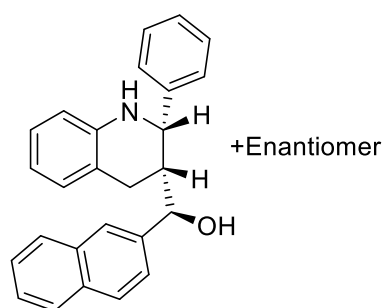
Yield (**8d'** only, 1 mmol)= 111 mg (30%)

8d



8d was not able to be isolated in pure form due to difficulties separating from impurities by column chromatography.

8d'



¹H NMR (500 MHz, CDCl₃) δ 7.82 – 7.86 (2H, m), 7.76 – 7.78 (1H, m), 7.62 (1H, s), 7.44 – 7.50 (5H, m), 7.30 – 7.36 (3H, m), 7.01 (1H, t, $J = 7.6$ Hz), 6.70 (1H, d, $J = 7.4$ Hz), 6.56 (1H, d, $J = 8.0$ Hz), 6.54 (1H, d, $J = 7.5$ Hz), 5.14 (1H, d, $J = 4.3$ Hz), 4.53 (1H, br. s), 4.16 (1H, d, $J = 10.3$ Hz), 2.74 – 2.81 (1H, m), 2.35 (1H, dd, $J = 16.3, 13.0$ Hz), 2.05 (1H, br. s), 1.96 (1H, dd, $J = 16.4, 4.2$ Hz).

¹³C NMR (125 MHz, CDCl₃) δ 144.2, 143.2, 140.3, 133.5, 133.3, 129.6, 128.8, 128.4, 128.1, 128.0, 127.9, 127.4, 127.4, 126.5, 126.4, 126.3, 124.6, 119.5, 116.6, 113.3, 75.9, 55.1, 43.4, 25.8.

HRMS (EI): calcd. for C₂₆H₂₃NO 365.1780, found 365.1787

NMR Spectra

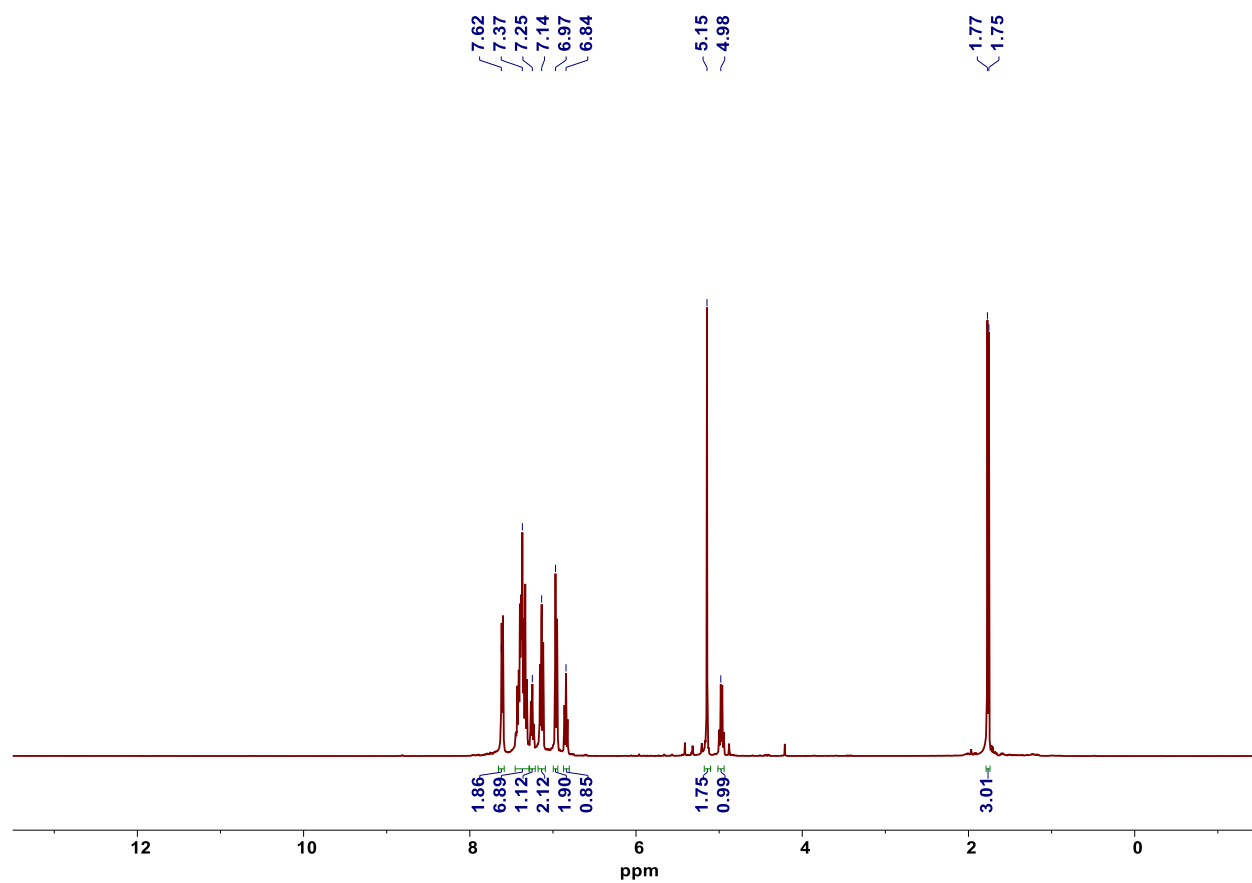


Figure S1 ^1H NMR (400 MHz) in CDCl_3 **4a** (crude sample since product was too unstable for isolation)

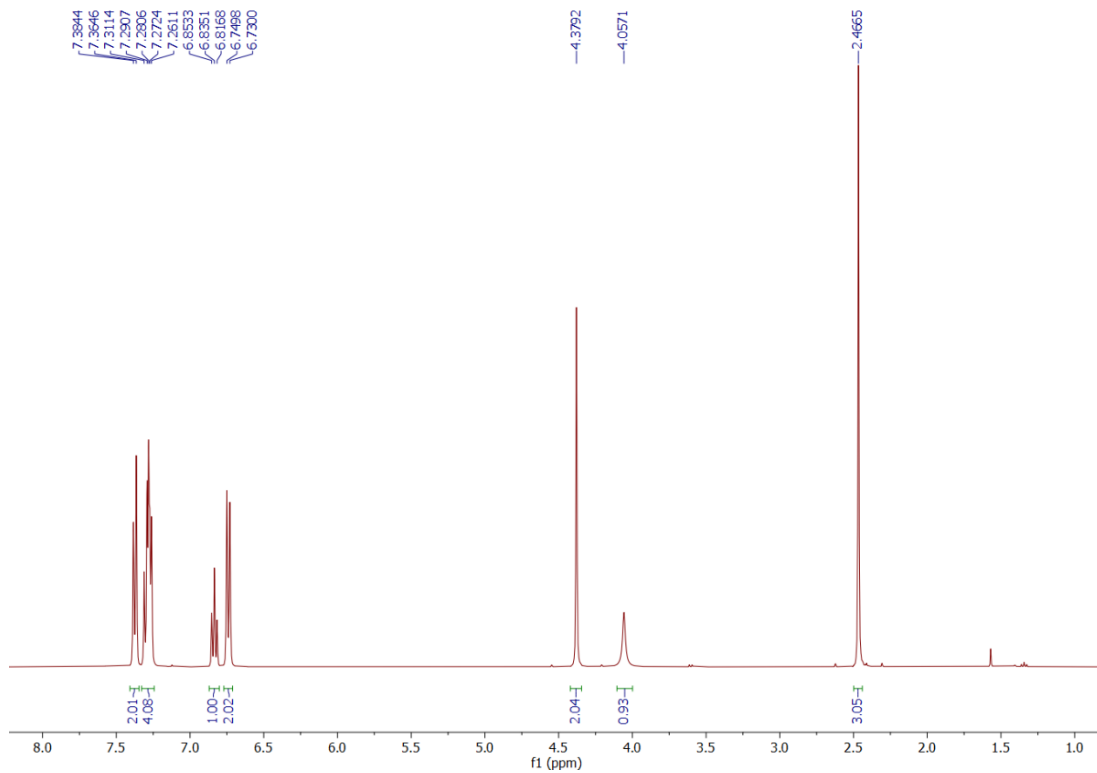


Figure S2 ^1H NMR (400 MHz) in CDCl_3 **4b**

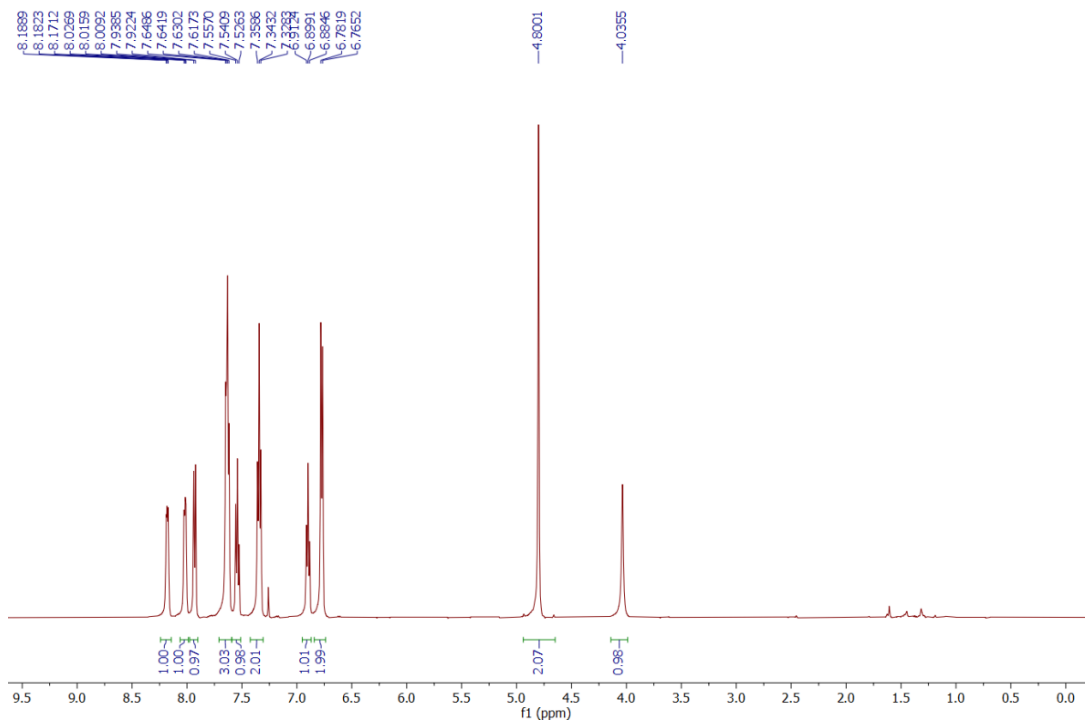


Figure S3 ^1H NMR (500 MHz) in CDCl_3 **4c**

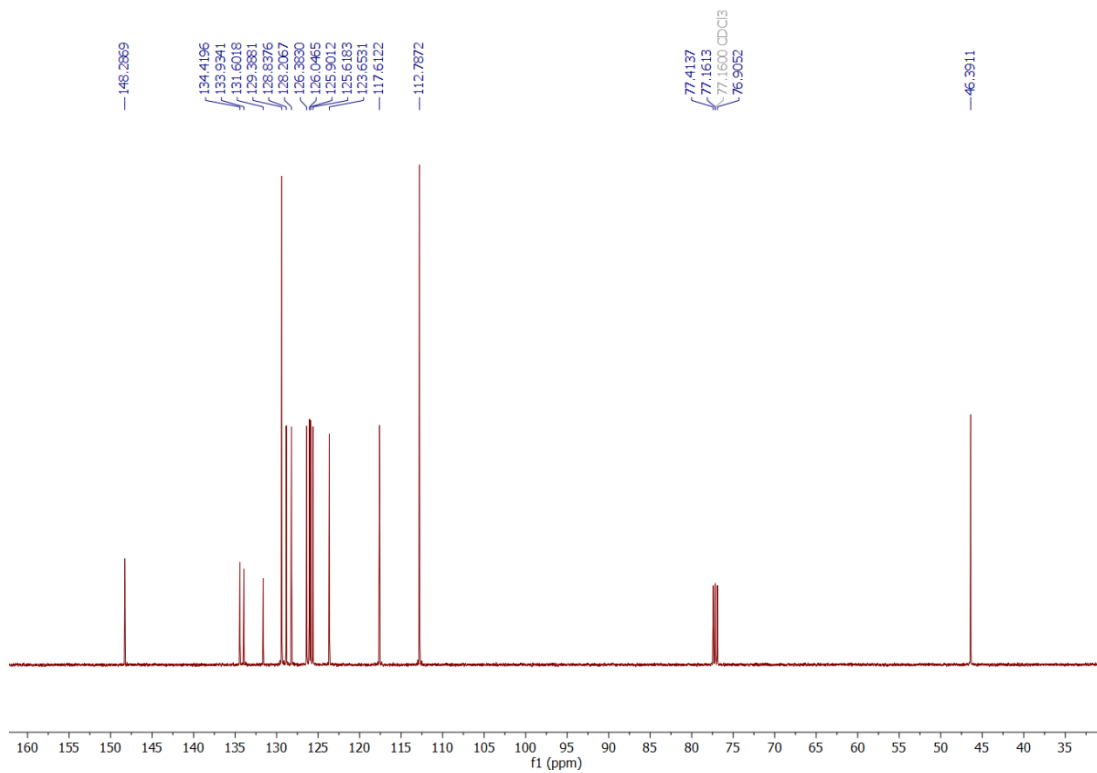


Figure S4 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **4c**

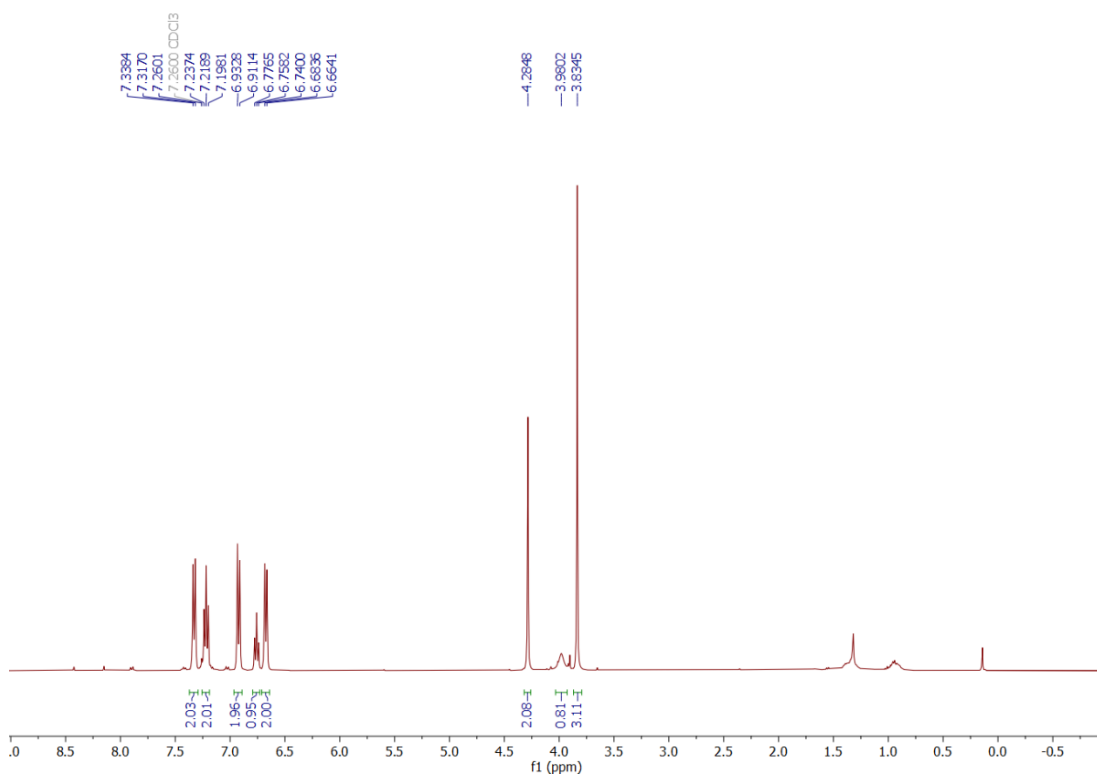


Figure S5 ^1H NMR (400 MHz) in CDCl_3 **4d**

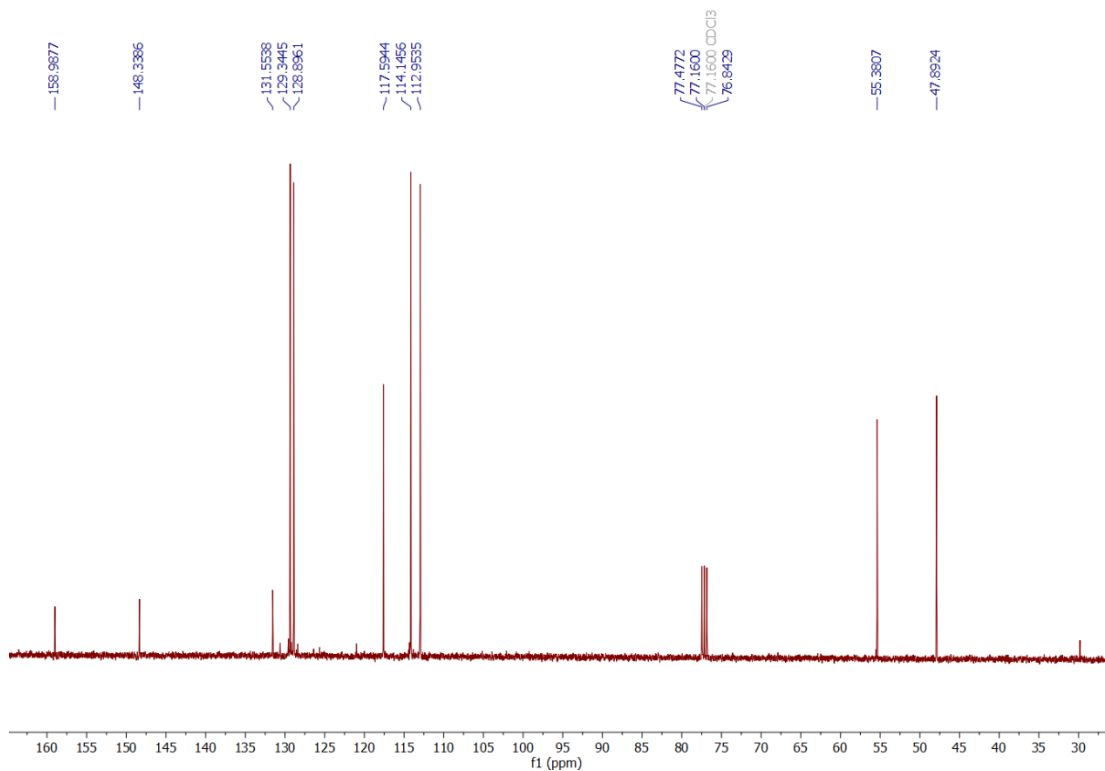


Figure S6 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz) in CDCl_3 **4d**

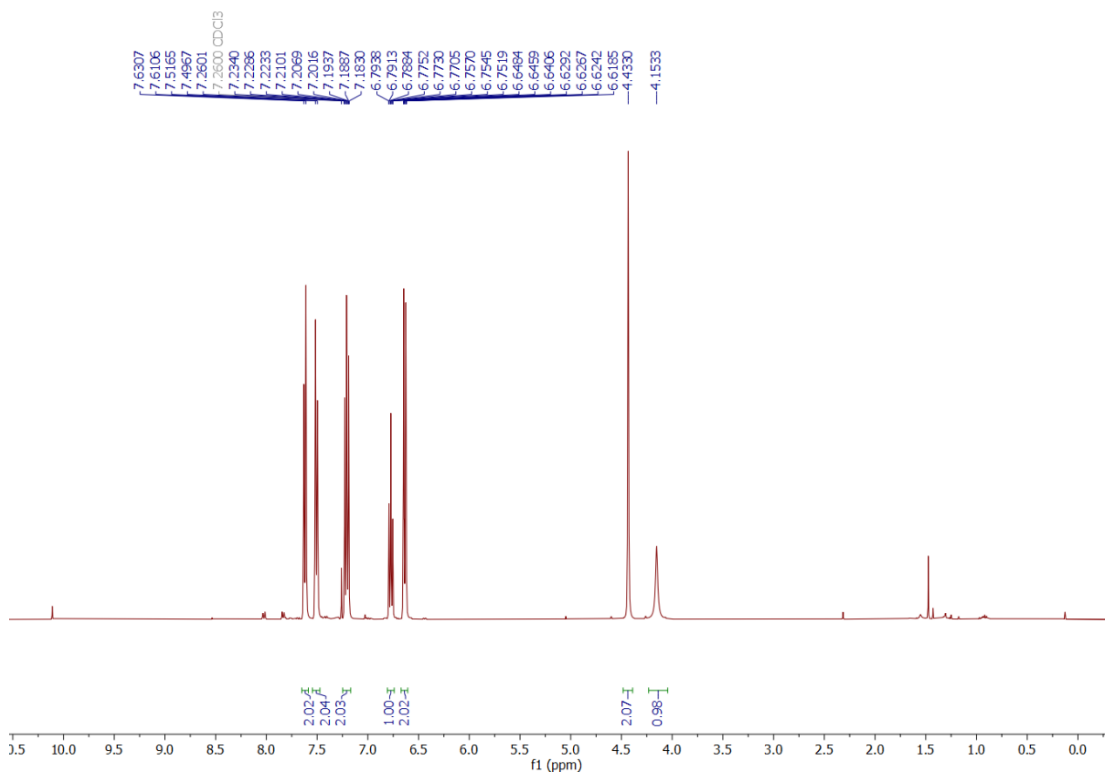


Figure S7 ^1H NMR (400 MHz) in CDCl_3 **4e**

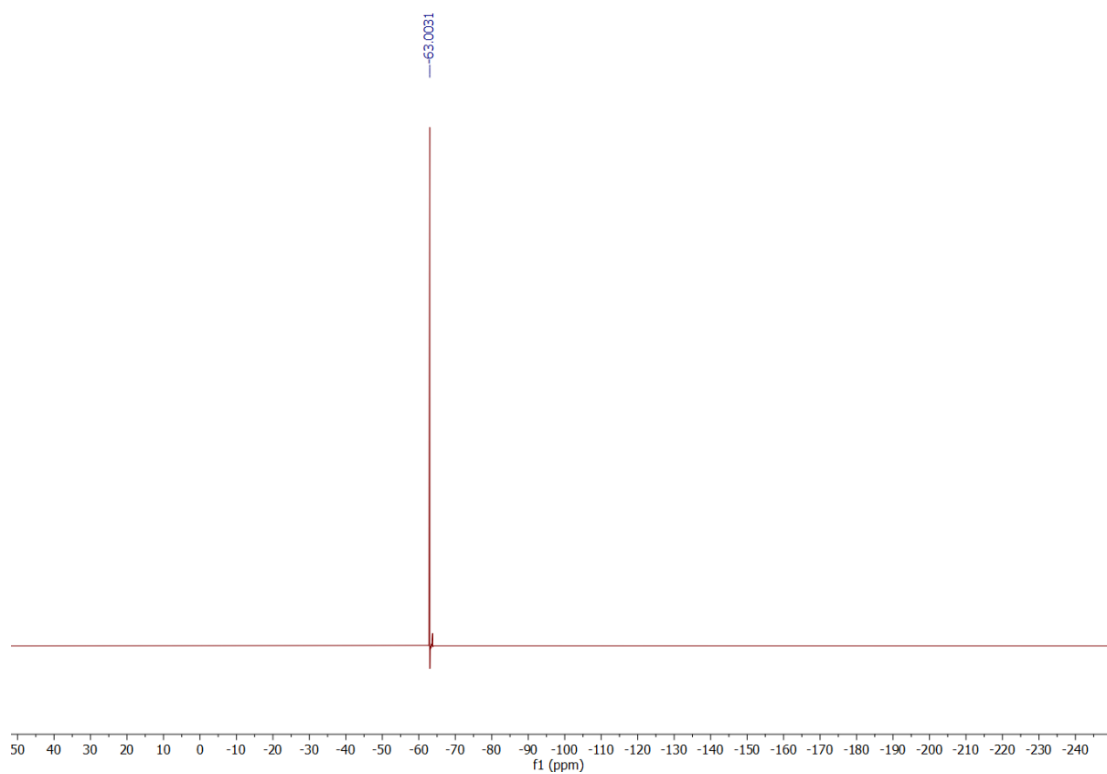


Figure S8 ^{19}F NMR (376 MHz) in CDCl_3 **4e**

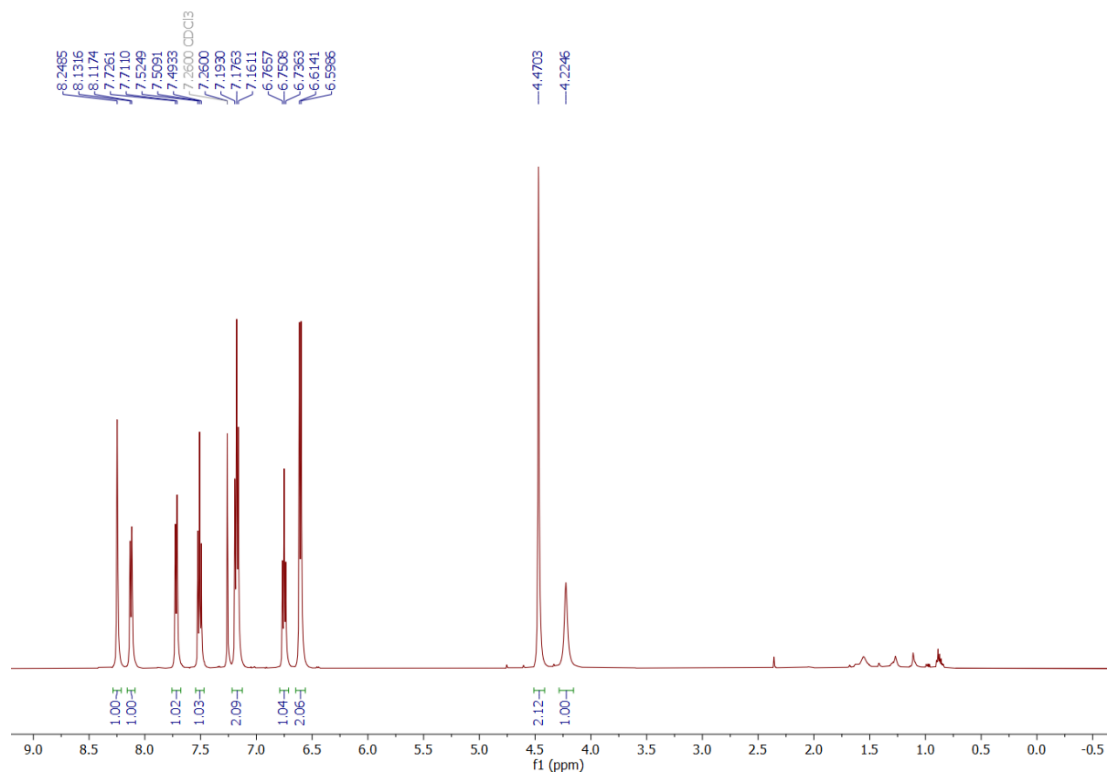


Figure S9 ^1H NMR (500 MHz) in CDCl_3 **4f**

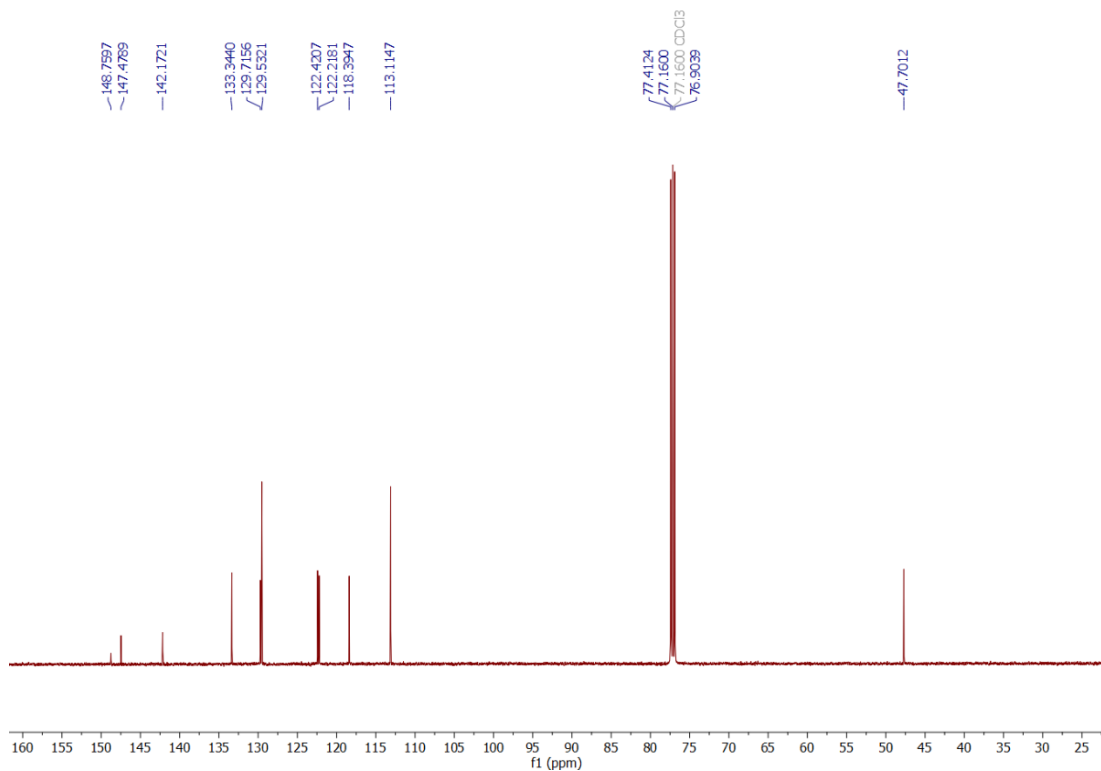


Figure S10 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **4f**

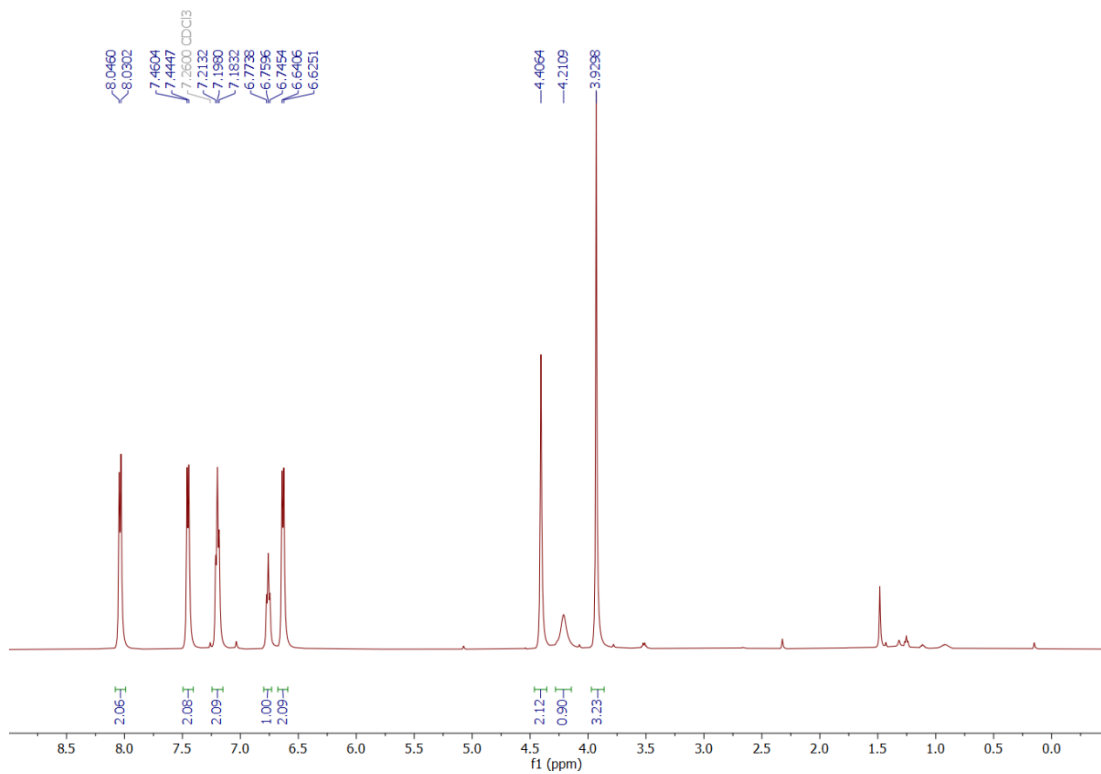


Figure S11 ^1H NMR (500 MHz) in CDCl_3 **4g**

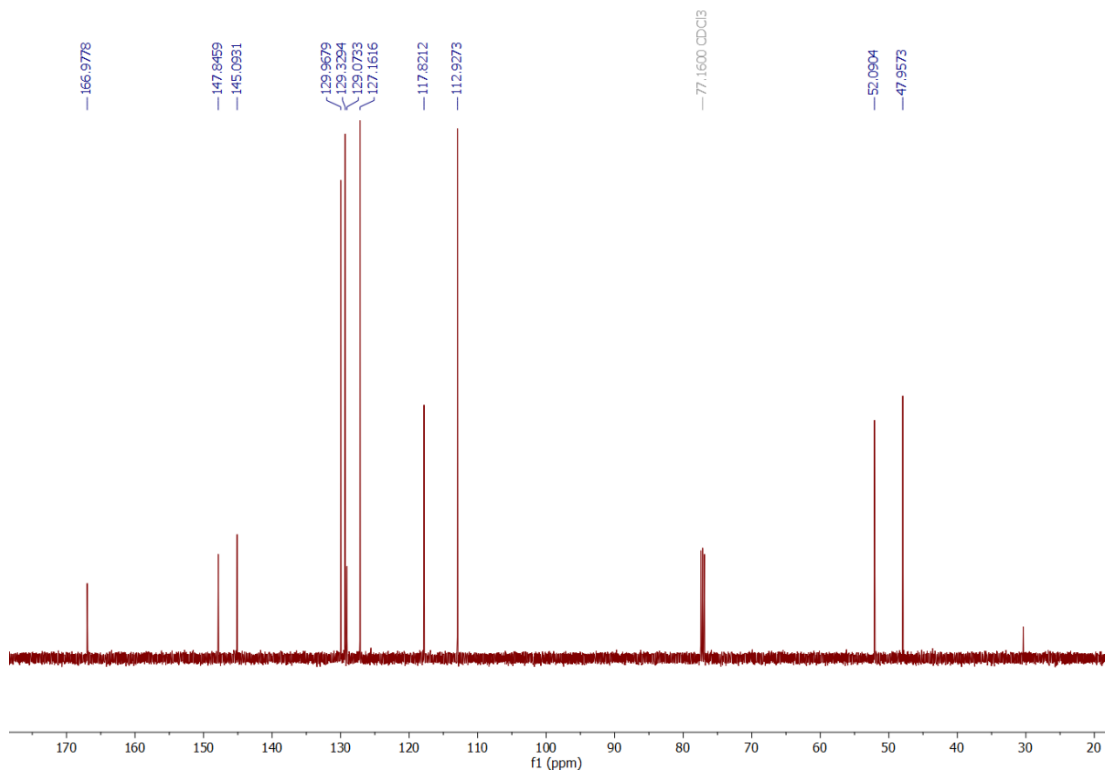


Figure S12 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **4g**

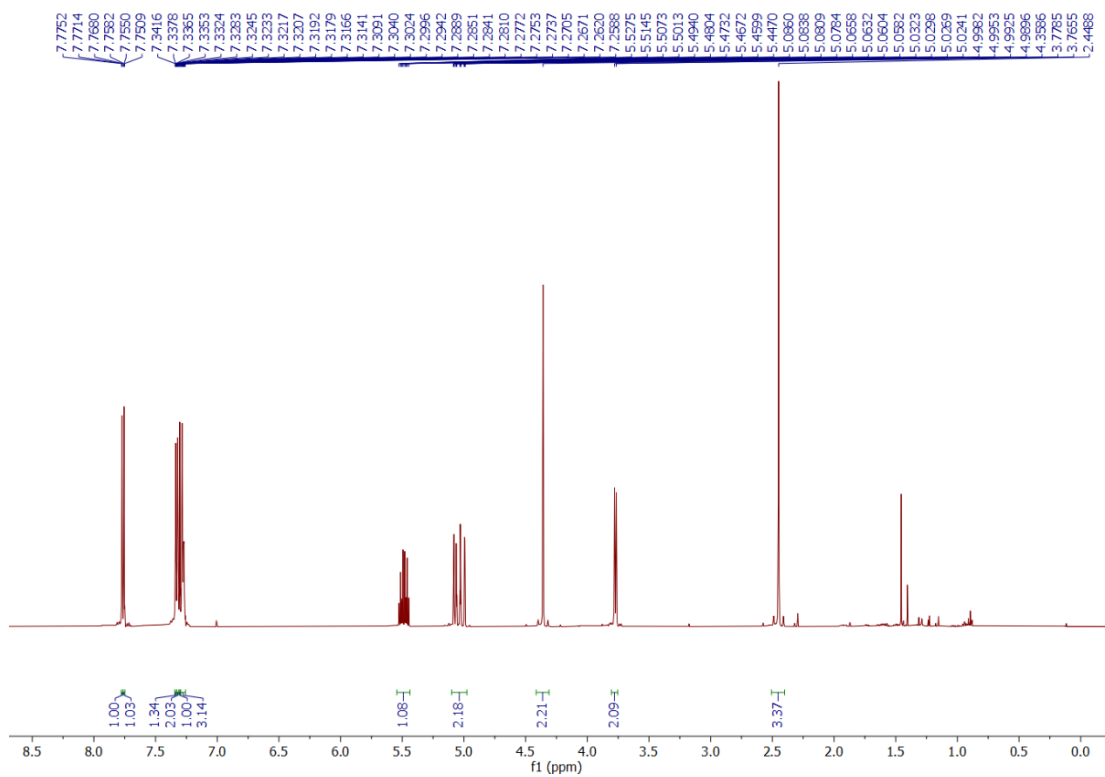


Figure S13 ^1H NMR (500 MHz) in CDCl_3 **4h**

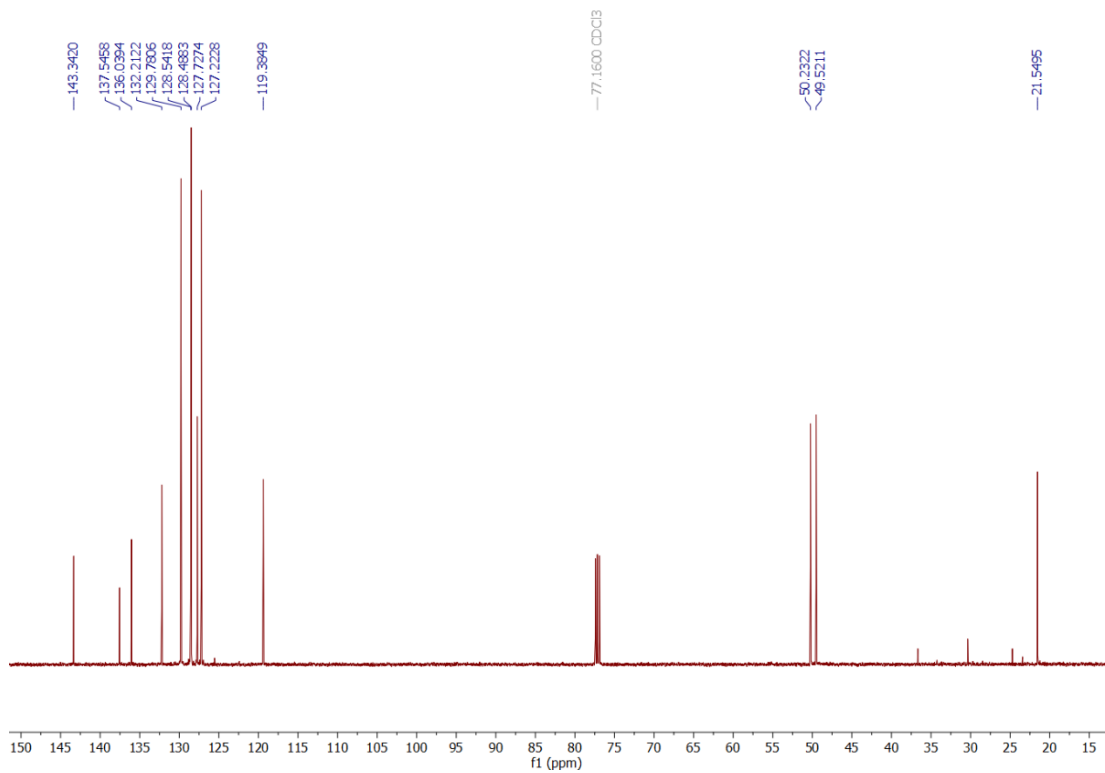


Figure S14 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **4h**

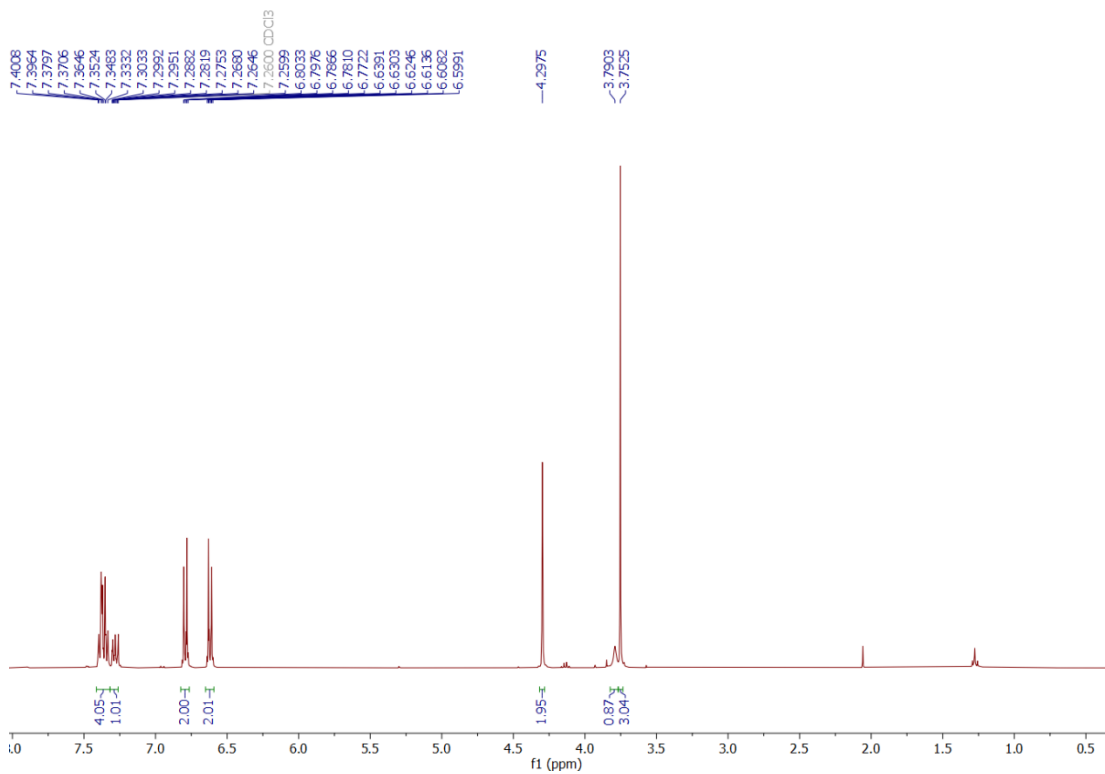


Figure S15 ^1H NMR (400 MHz) in CDCl_3 **4i**

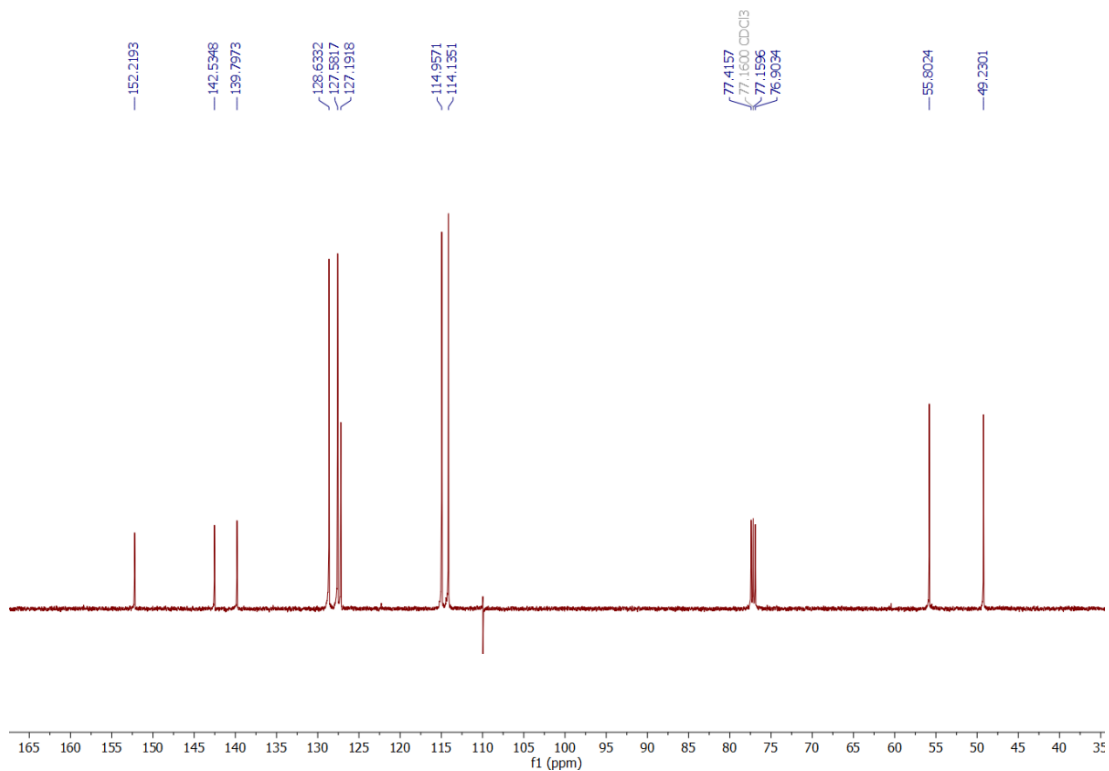


Figure S16 ¹³C{¹H} NMR (126 MHz) in CDCl₃ **4i**

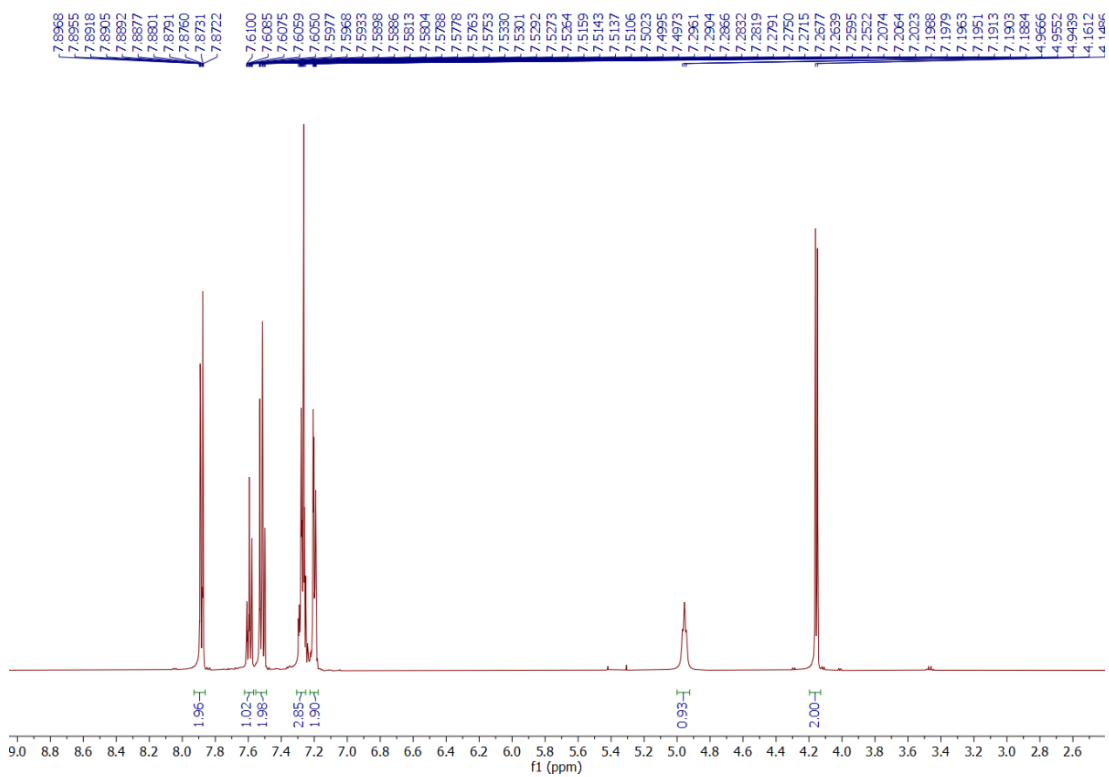


Figure S17 ¹H NMR (500 MHz) in CDCl₃ **4i**

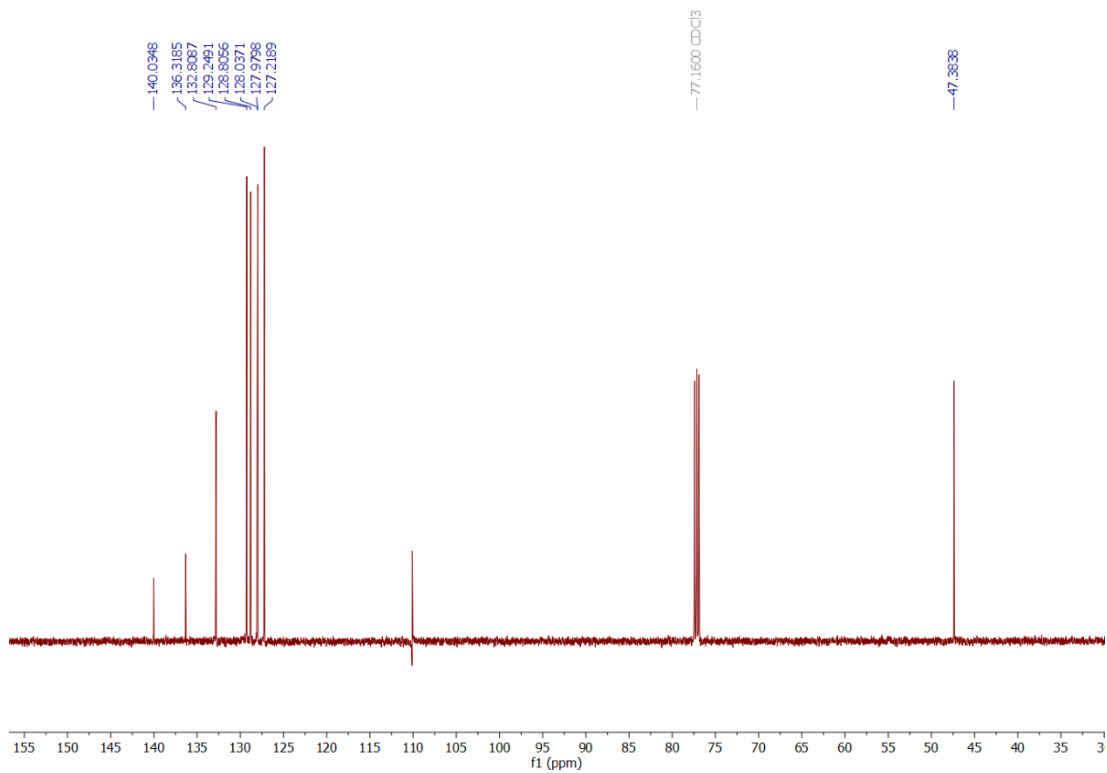


Figure S18 ^{13}C NMR (126 MHz) in CDCl_3 **4j**

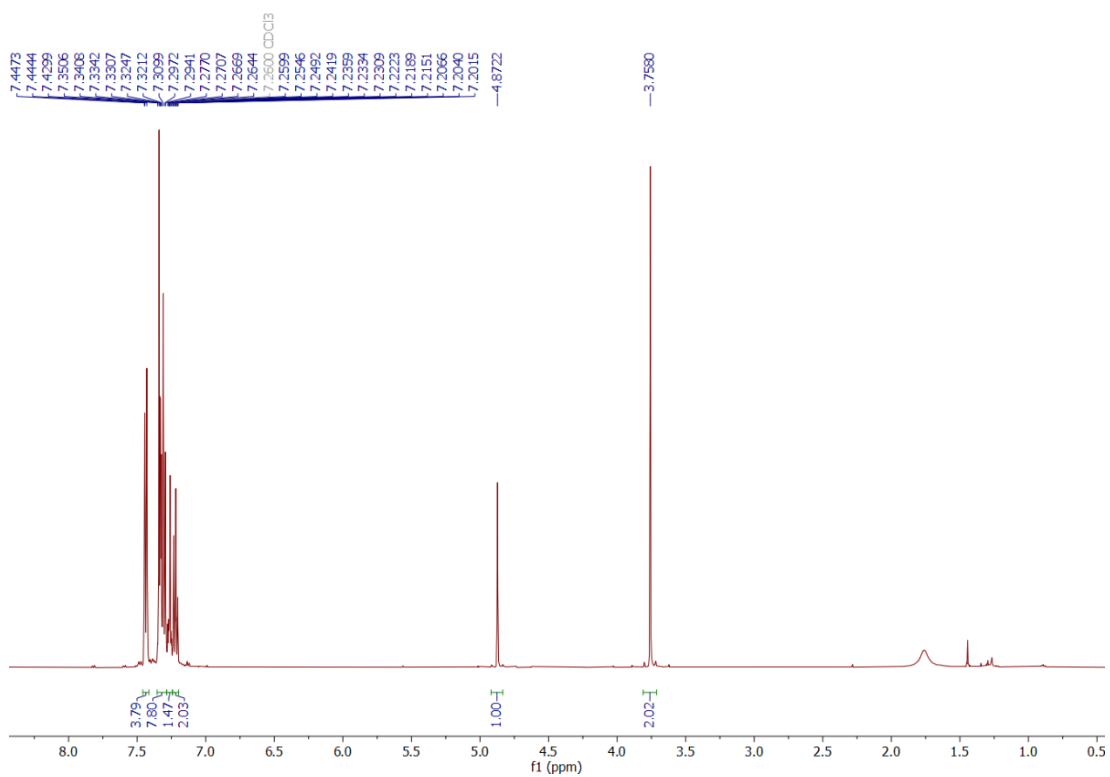


Figure S19 ^1H NMR (500 MHz) in CDCl_3 **4k**

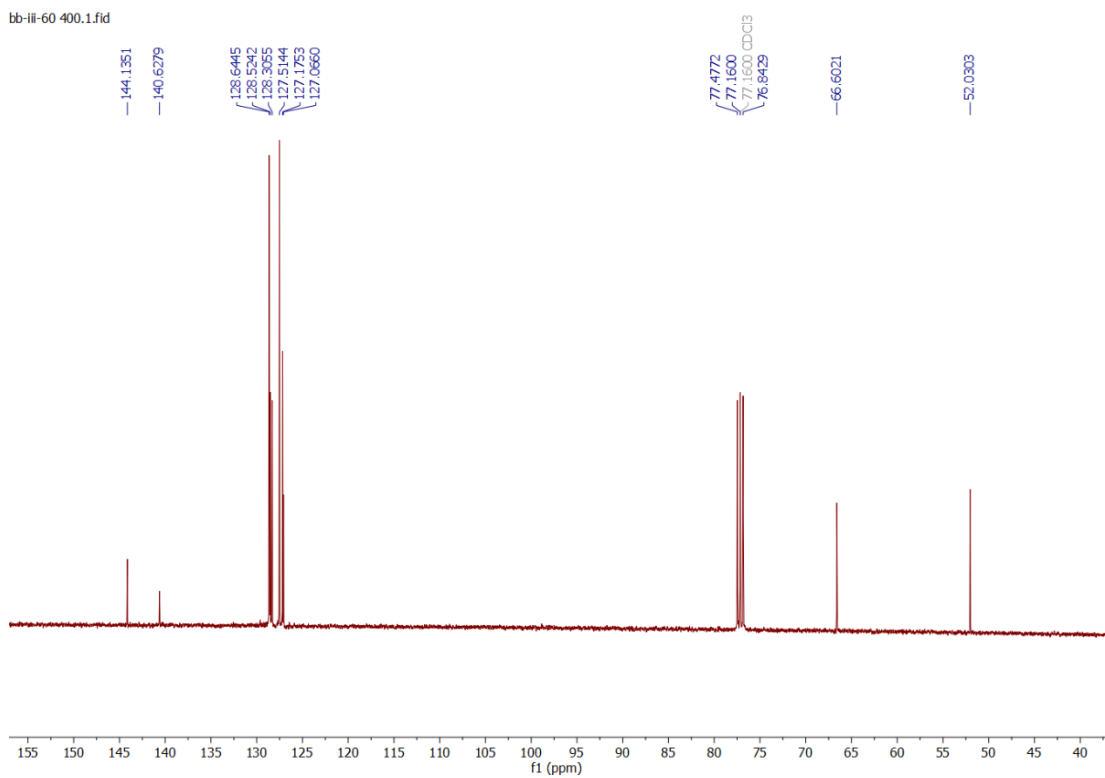


Figure S20 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz) in CDCl_3 **4k**

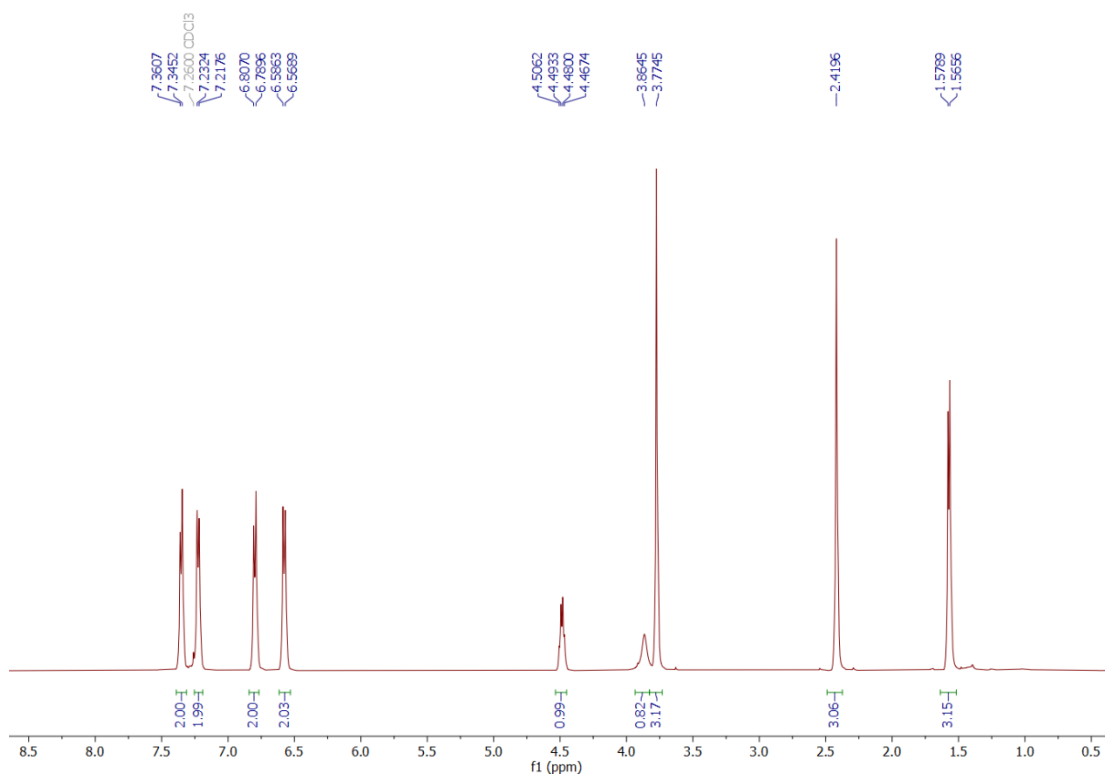


Figure S21 ^1H NMR (500 MHz) in CDCl_3 **4l**

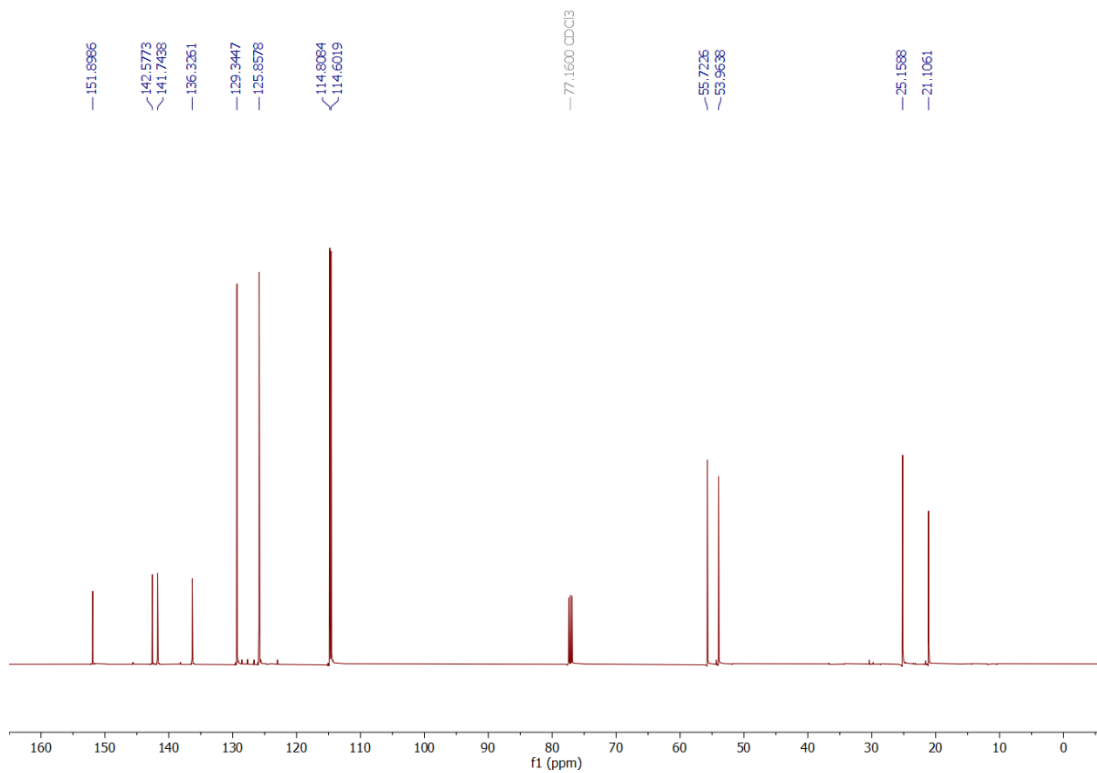


Figure S22 ¹³C{¹H} NMR (126 MHz) in CDCl₃ **4I**

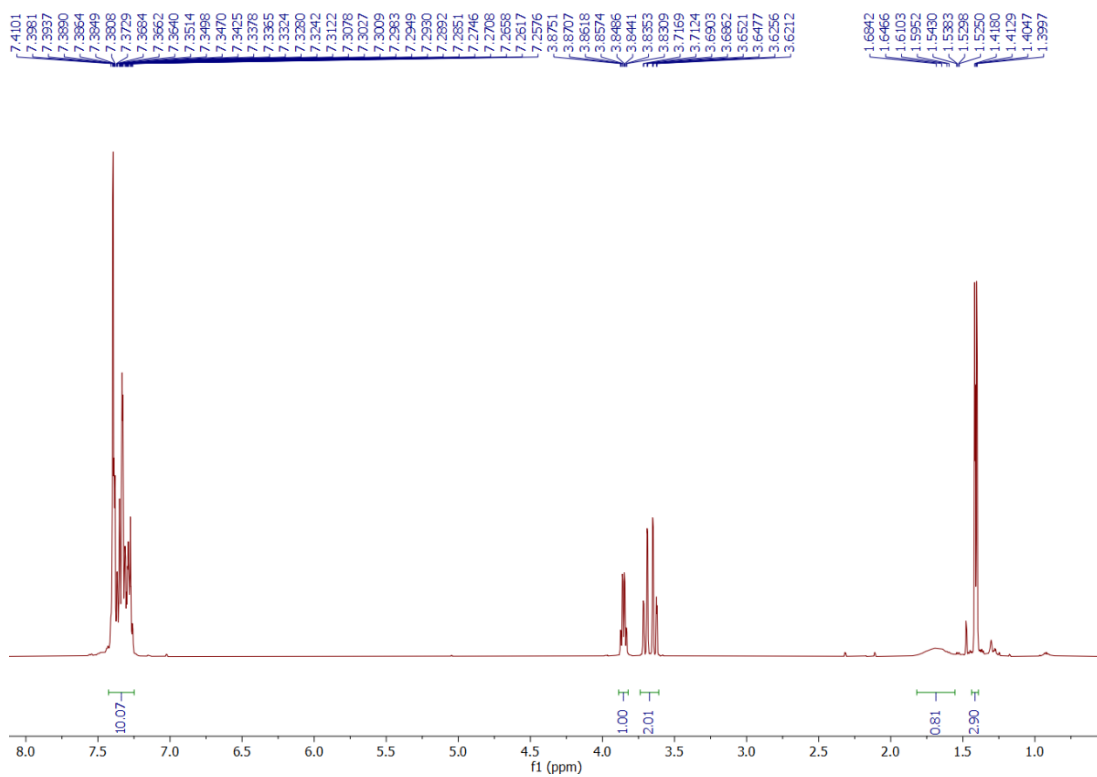


Figure S23 ¹H NMR (500 MHz) in CDCl₃ **4m**

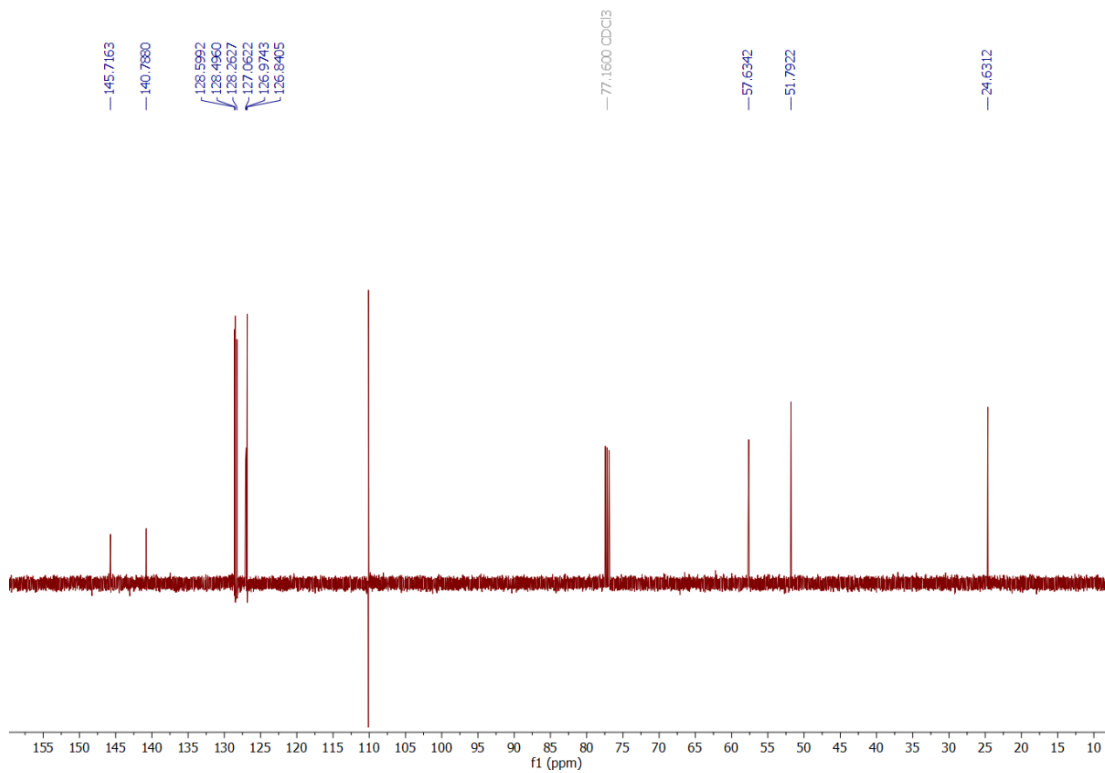


Figure S24 ¹³C{¹H} NMR (126 MHz) in CDCl₃ **4m**

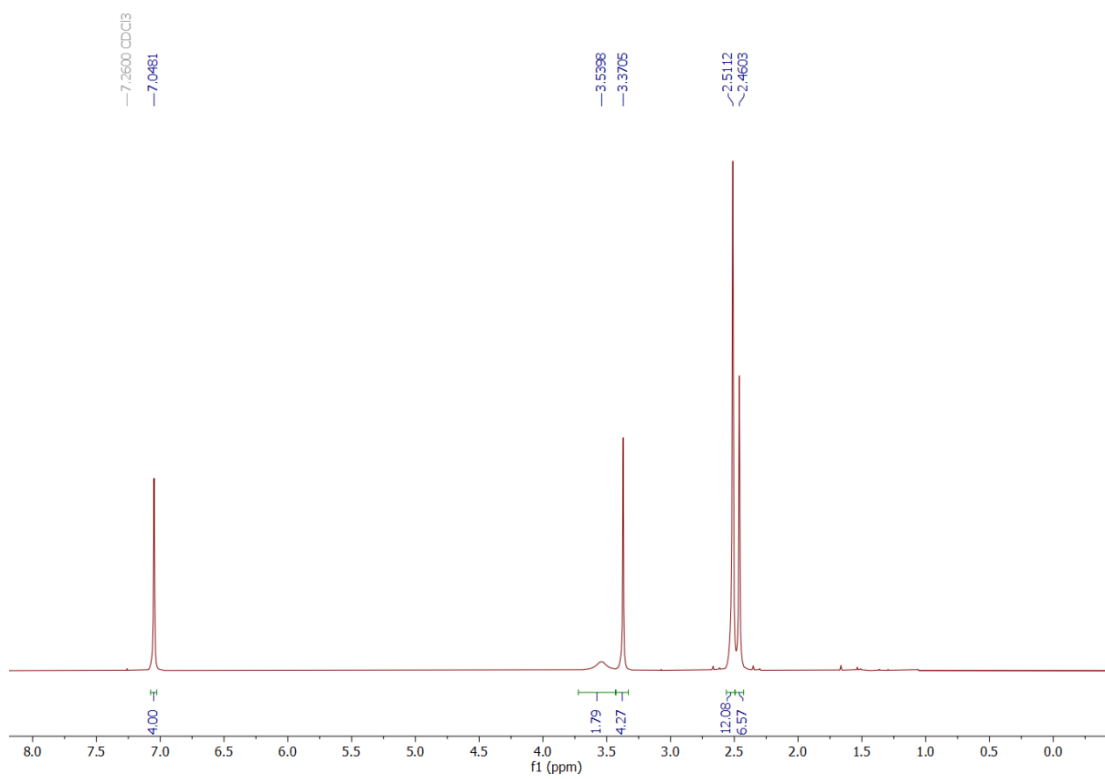


Figure S25 ¹H NMR (400 MHz) in CDCl₃ **4n**

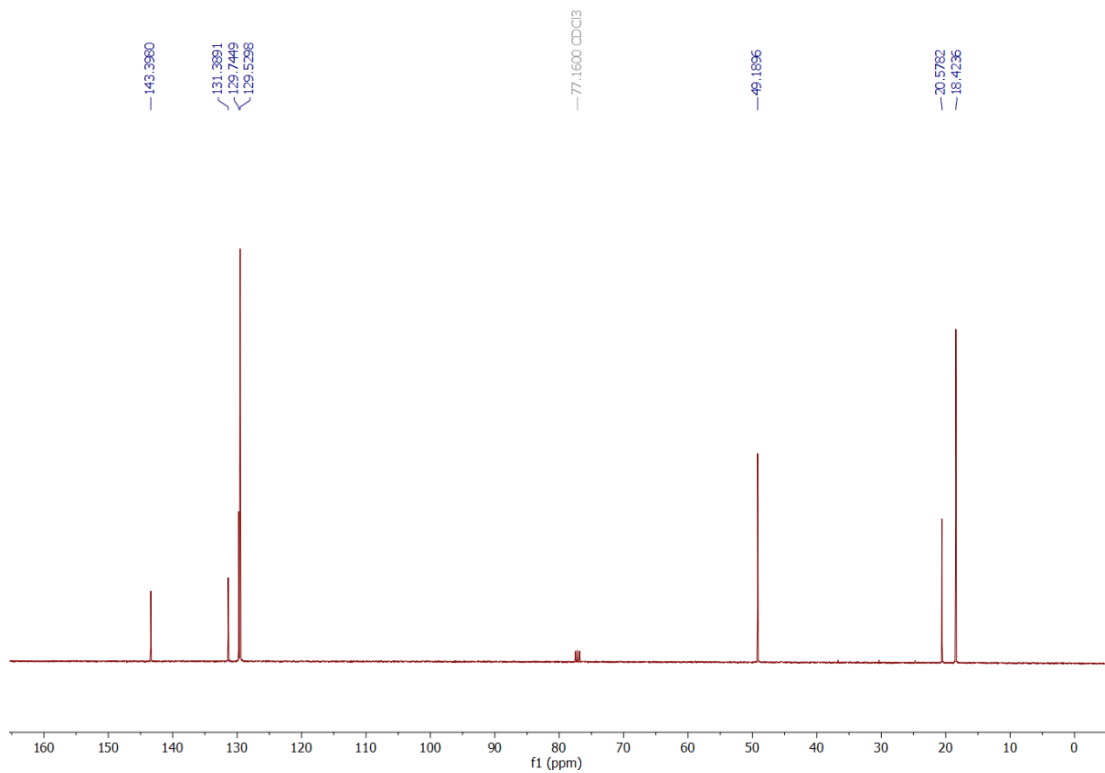


Figure S26 ¹³C{¹H} NMR (101 MHz) in CDCl₃ **4n**

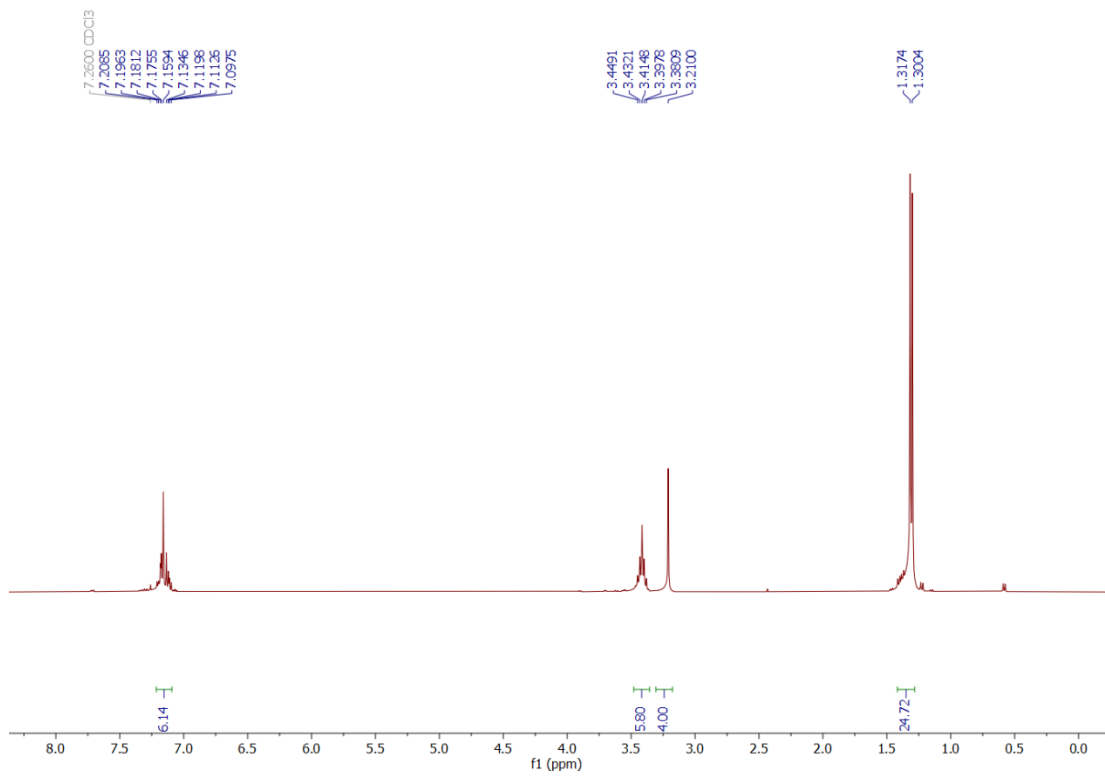


Figure S27 ¹H NMR (400 MHz) in CDCl₃ **4o**

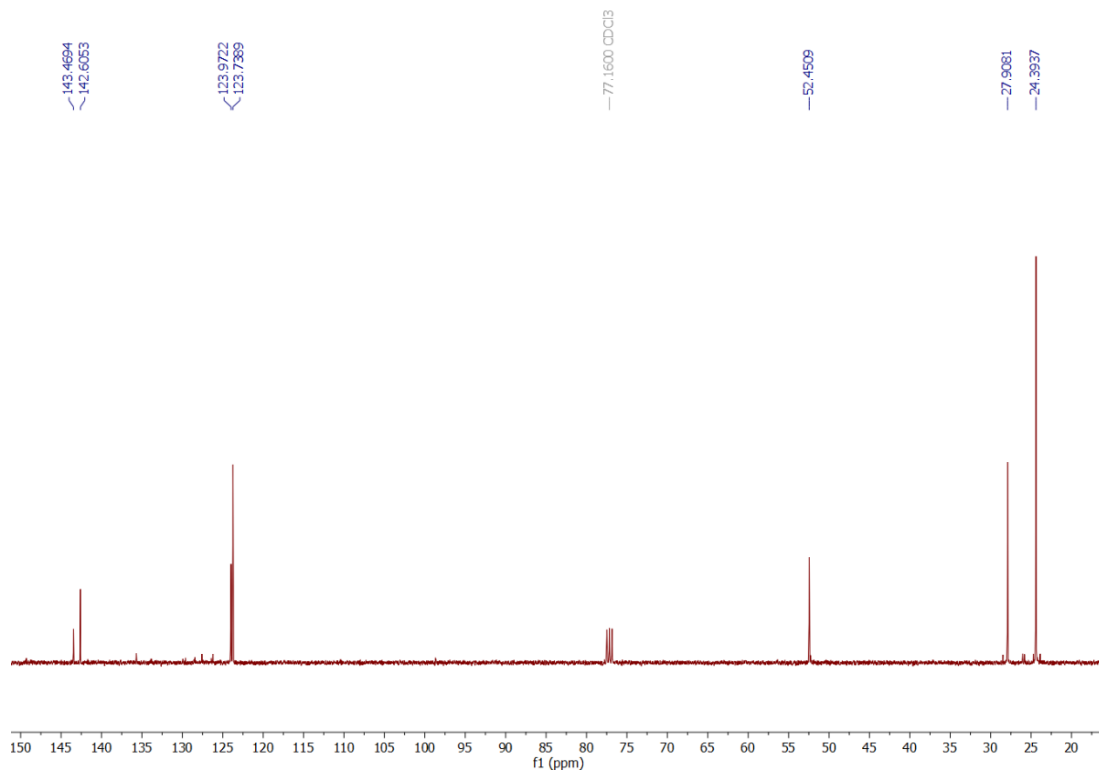


Figure S28 ¹³C{¹H} NMR (101 MHz) in CDCl₃ **4o**

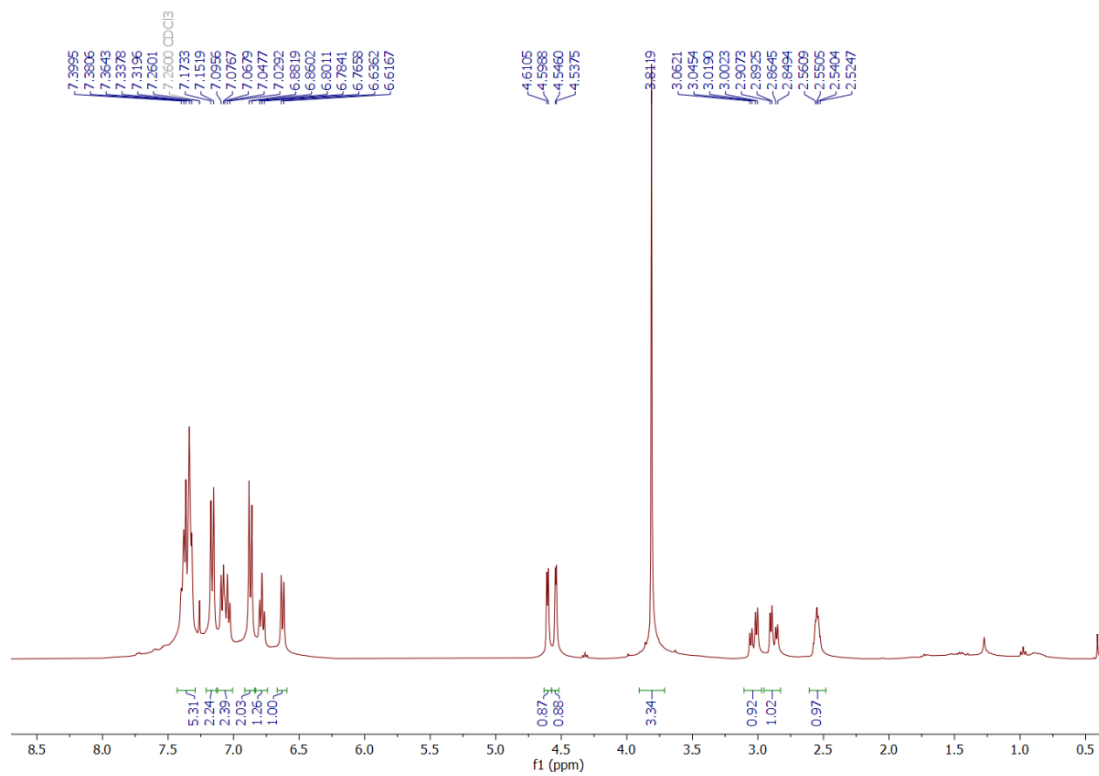


Figure S29 ¹H NMR (400 MHz) in CDCl₃ **8a**

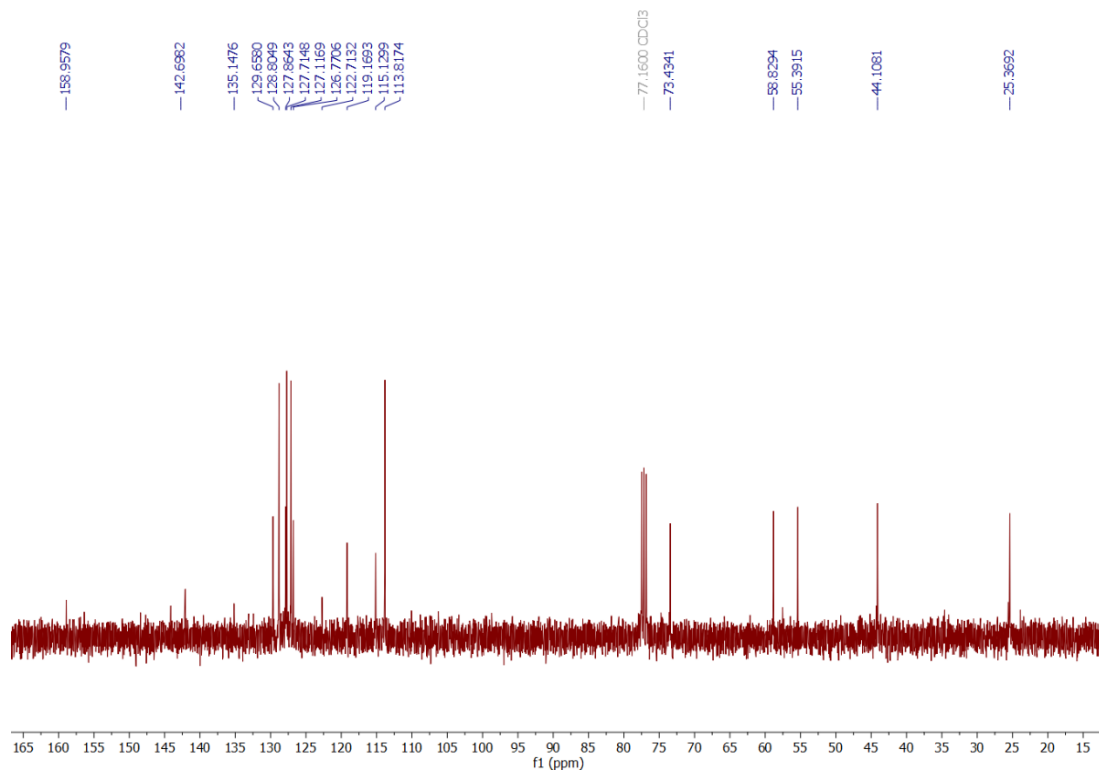


Figure S30 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **8a**

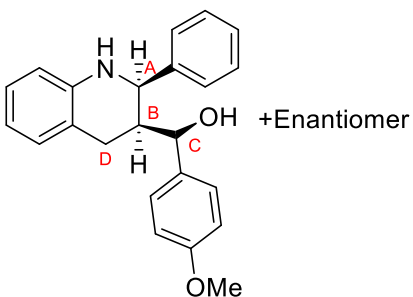


Table S1 NMR assignments for **8a**.

Atom Label	^1H NMR Peak (ppm)	Splitting and J Coupling (Hz)
A	4.54	d, $J = 3.4$ (Confirms <i>syn</i> to B)
B	2.54	m
C	4.60	d, $J = 4.7$ (Confirms <i>syn</i> to B)
D	3.03, 2.88	dd, $J = 17.2, 6.7$; dd, $J = 17.1, 5.9$

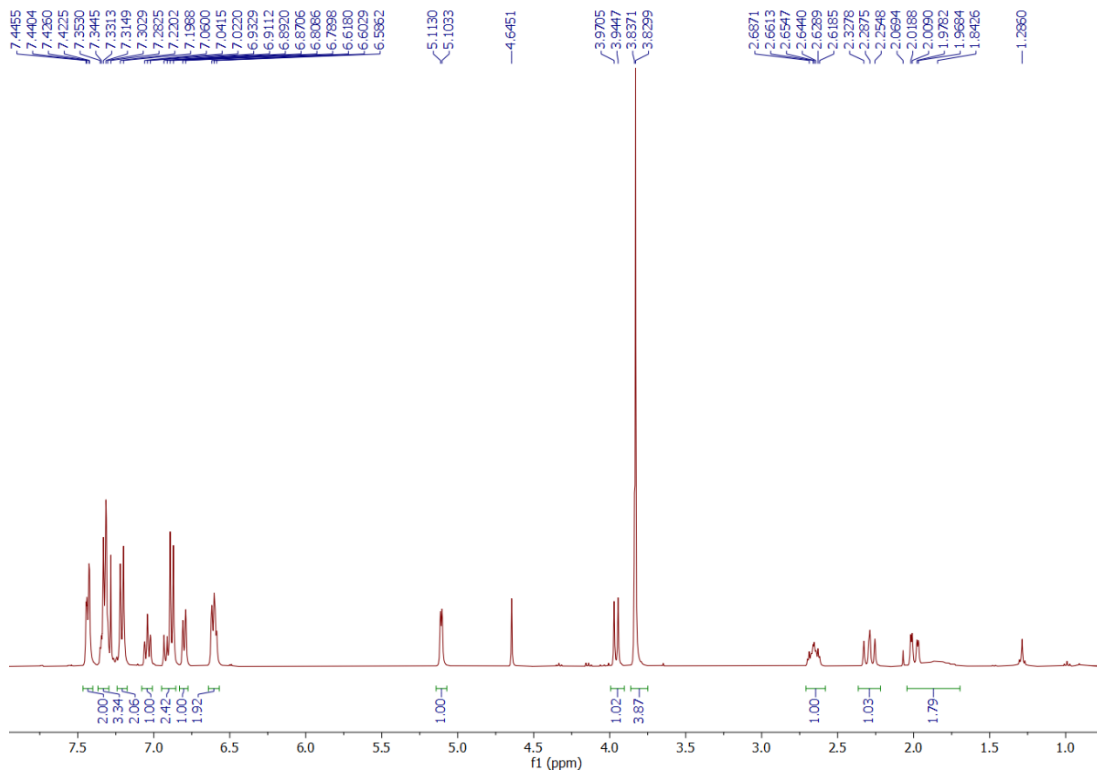


Figure S31 ^1H NMR (400 MHz) in CDCl_3 **8a'**

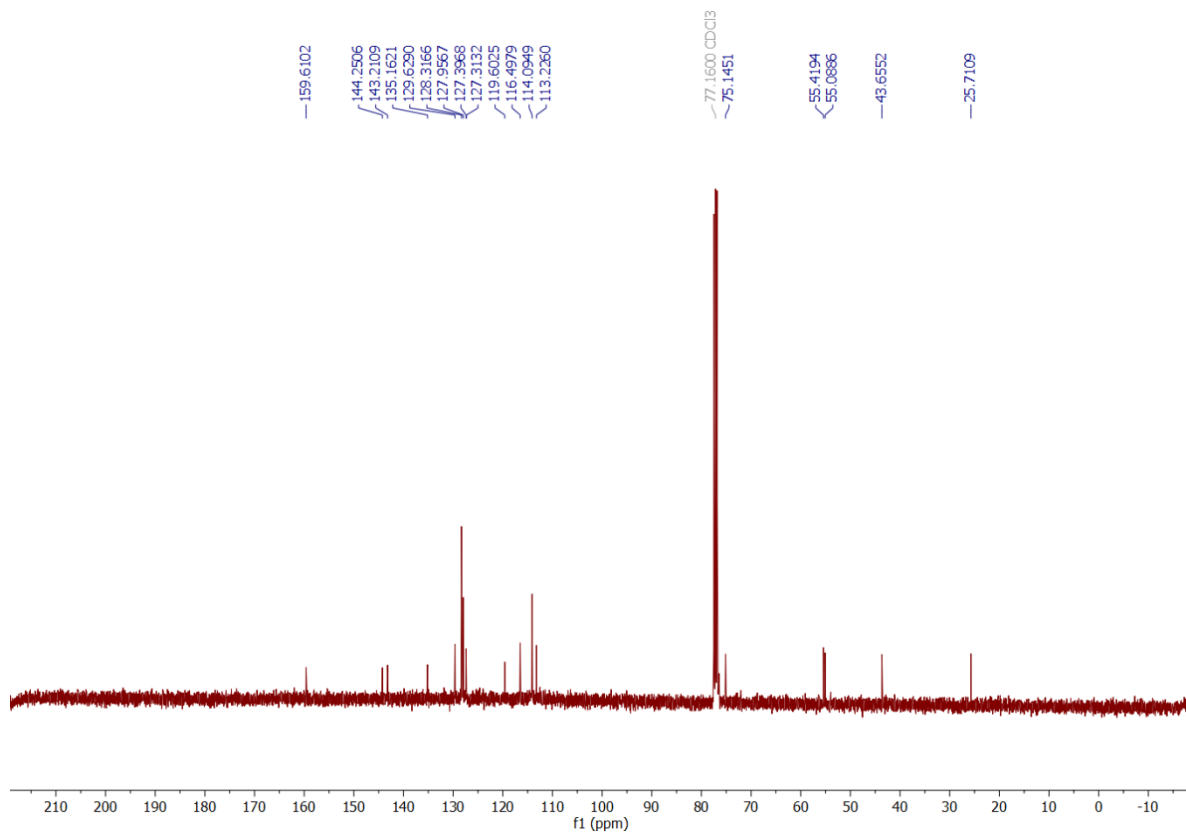


Figure S32 ^{13}C NMR (125 MHz) in CDCl_3 **8a'**

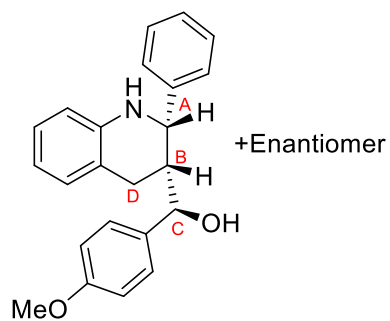


Table S2 NMR assignments for **8a'**.

Atom Label	¹ H NMR Peak (ppm)	Splitting and J Coupling (Hz)
A	5.08	d, J = 3.49 (Confirms <i>syn</i> to B)
B	3.81	m
C	3.94	d, J = 10.4 (Confirms <i>anti</i> to B)
D	2.26, 1.97	dd, J = 16.1, 13.1; dd, J = 16.2, 3.8

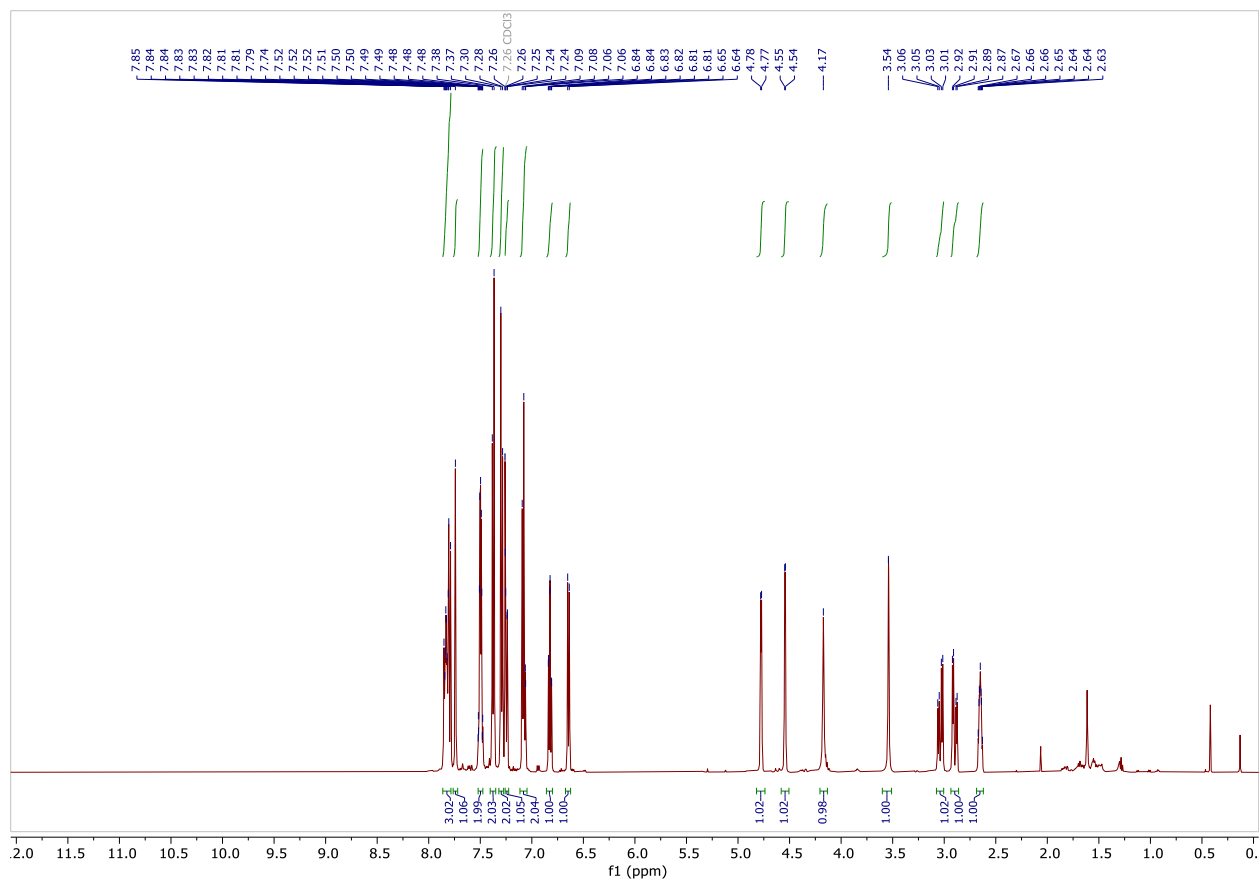


Figure S33 ^1H NMR (400 MHz) in CDCl_3 **8b**

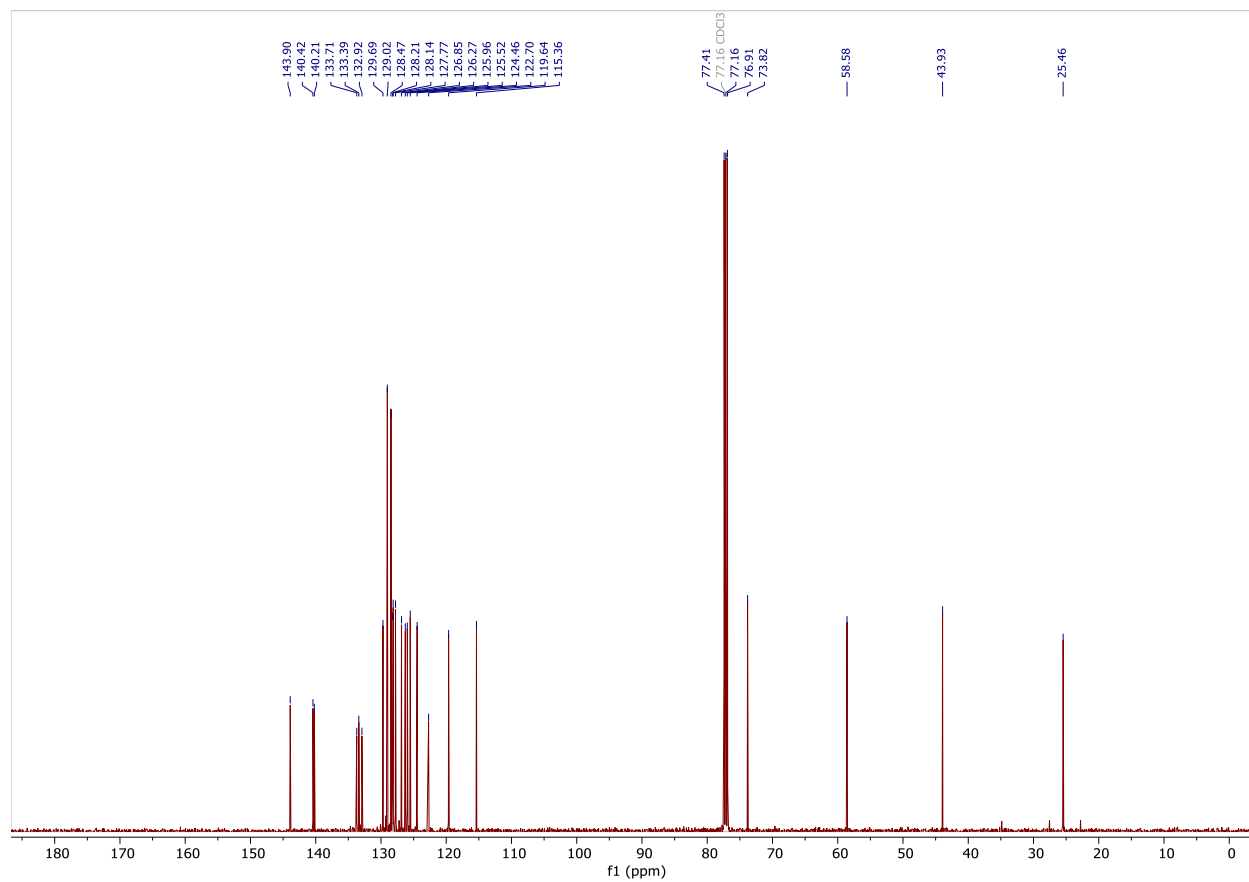


Figure S34 $^{13}\text{C}\{^1\text{H}\}$ NMR (176 MHz) in CDCl_3 **8b**

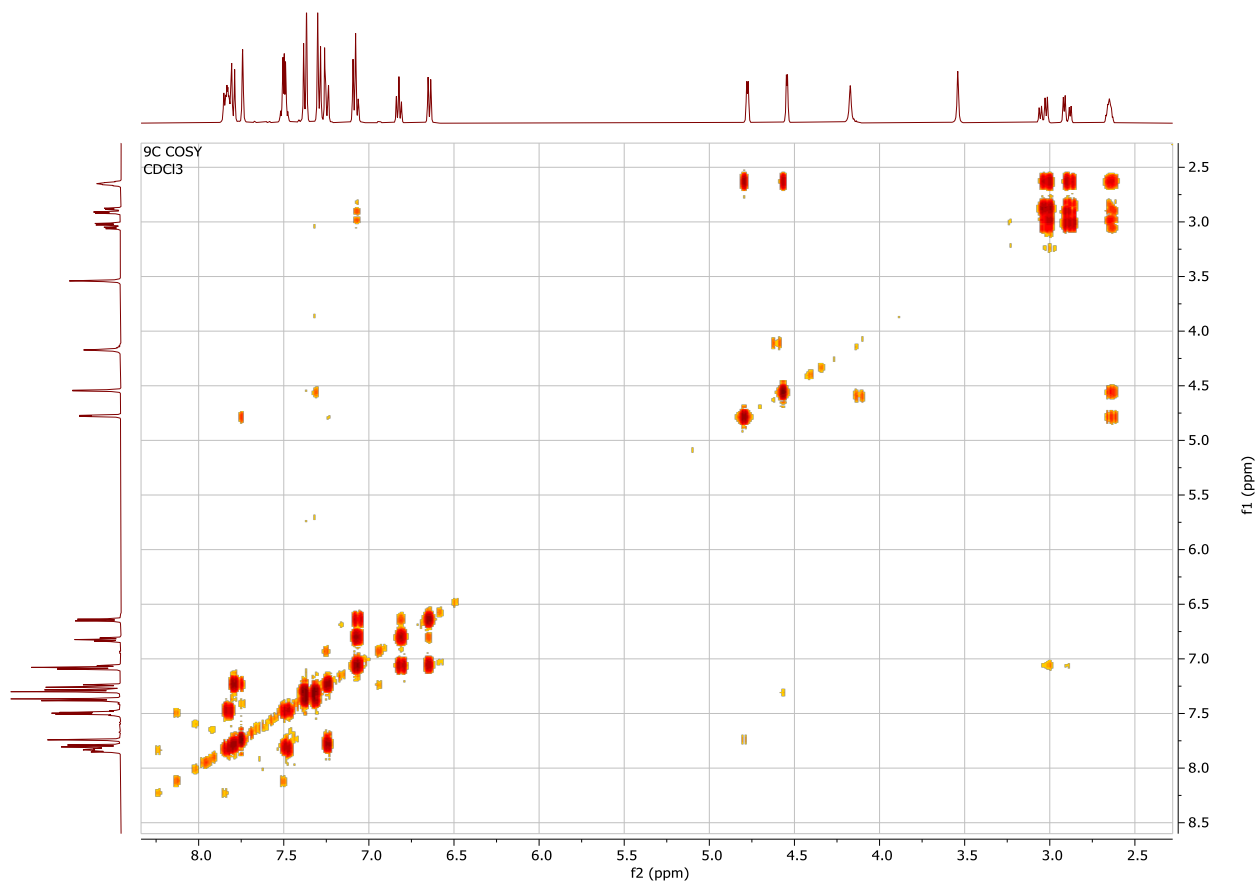


Figure S35 ¹H-¹H COSY NMR in CDCl₃ **8b**

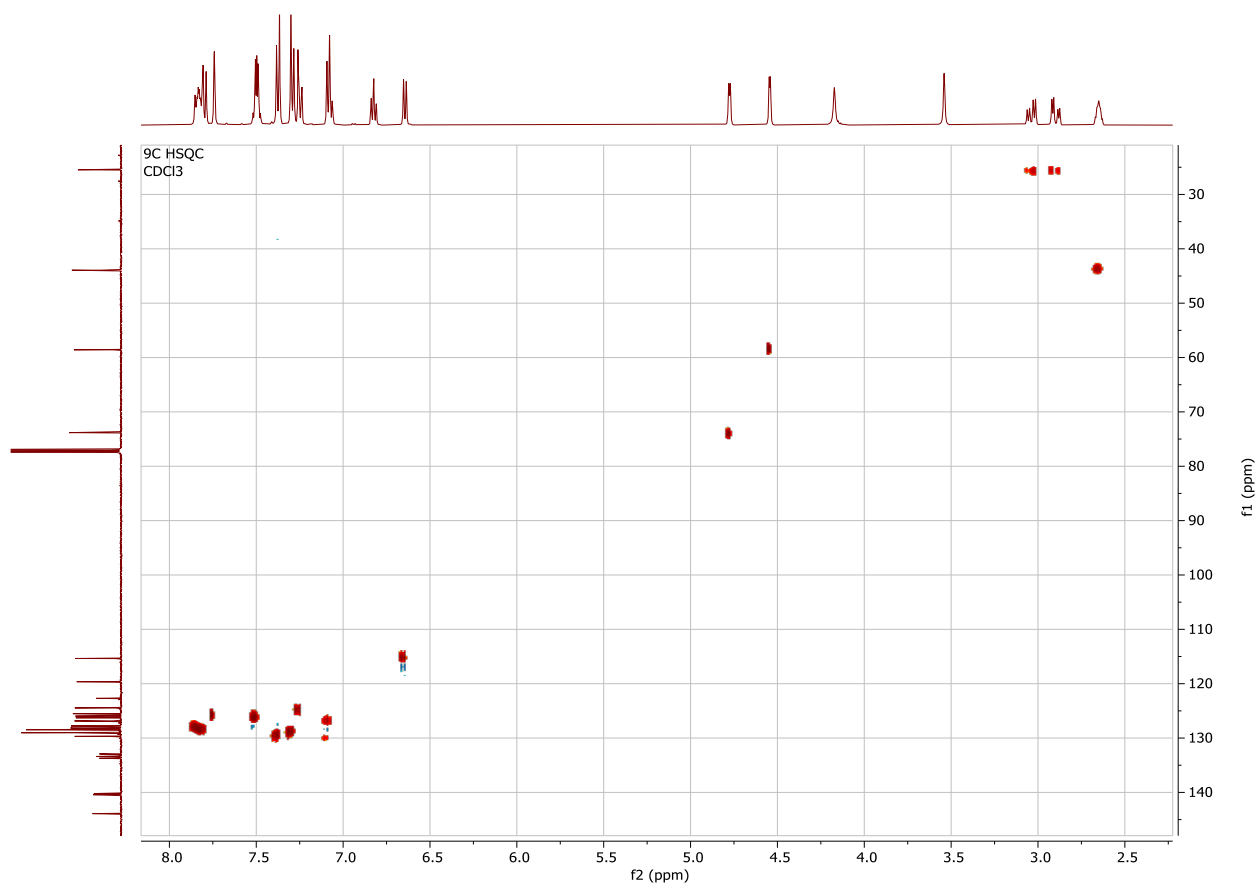


Figure S36 ¹H-¹³C HSQC NMR in CDCl₃ **8b**

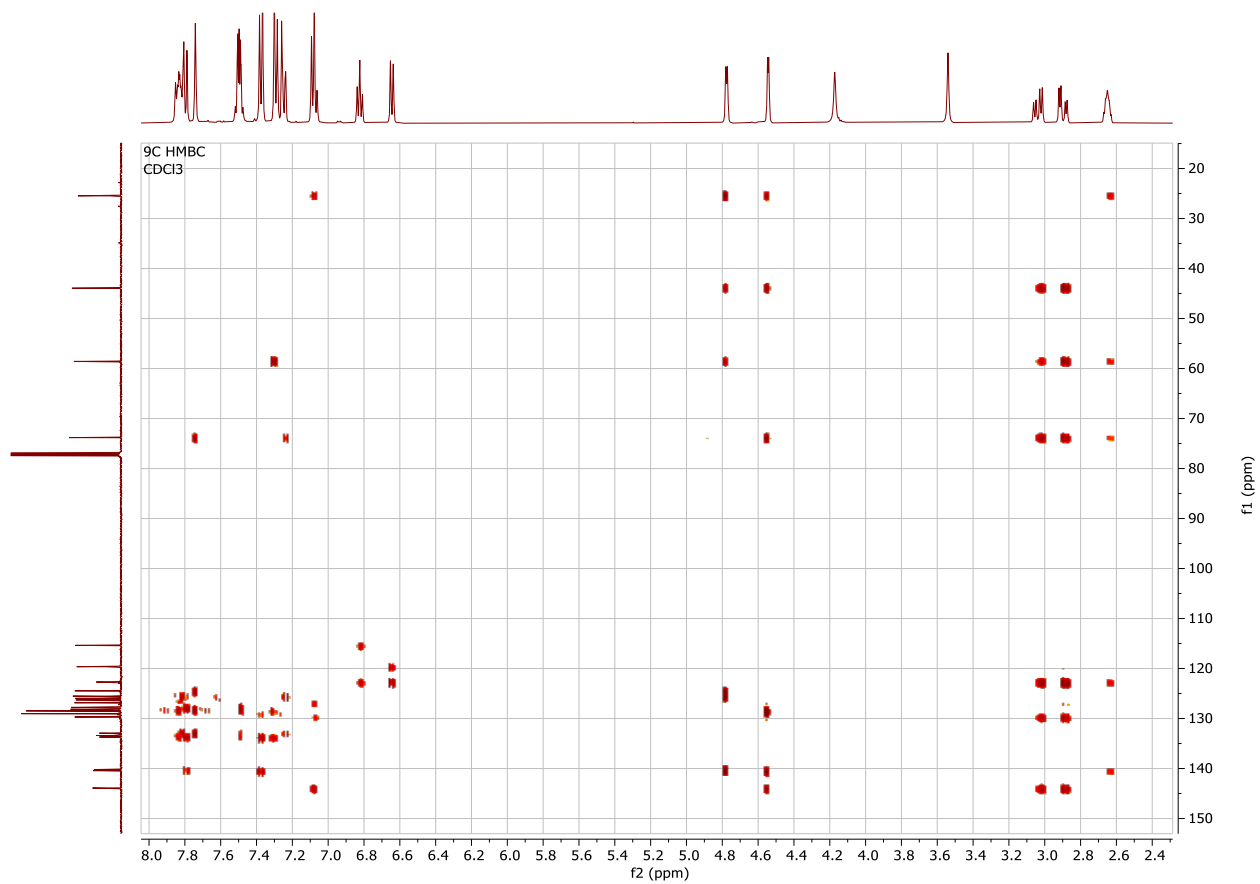


Figure S37 ^1H - ^{13}C HMBC NMR in CDCl_3 **8b**

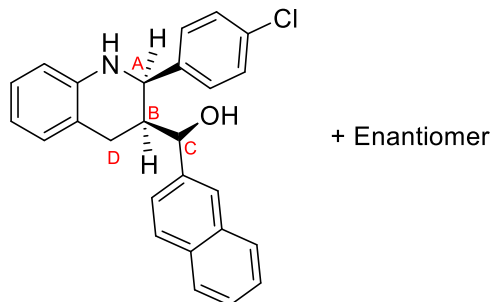


Table S3 NMR assignments for **8b**.

Atom Label	¹ H NMR Peak (ppm)	Splitting and J Coupling (Hz)	¹³ C NMR Peak (ppm)
A	4.54	d, J = 3.5 (Confirms <i>syn</i> to B)	58.58
B	2.67-2.63	m	43.93
C	4.78	d, J = 4.4 (Confirms <i>syn</i> to B)	73.82
D	3.04, 2.90	dd, J = 17.3, 7.0; dd, J = 17.3, 5.4	25.46

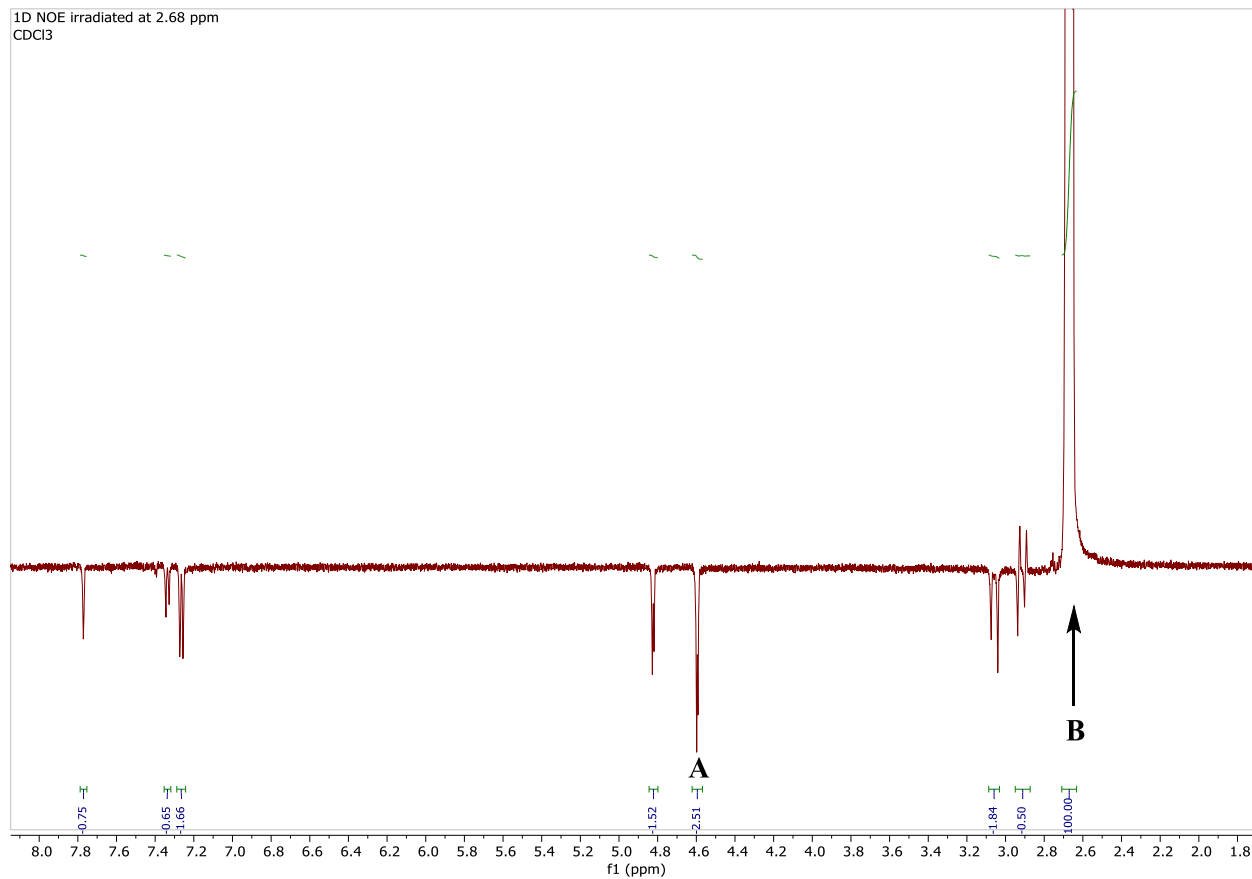


Figure S38 1D NOESY NMR of **8b** irradiated at 2.68 ppm in CDCl₃.

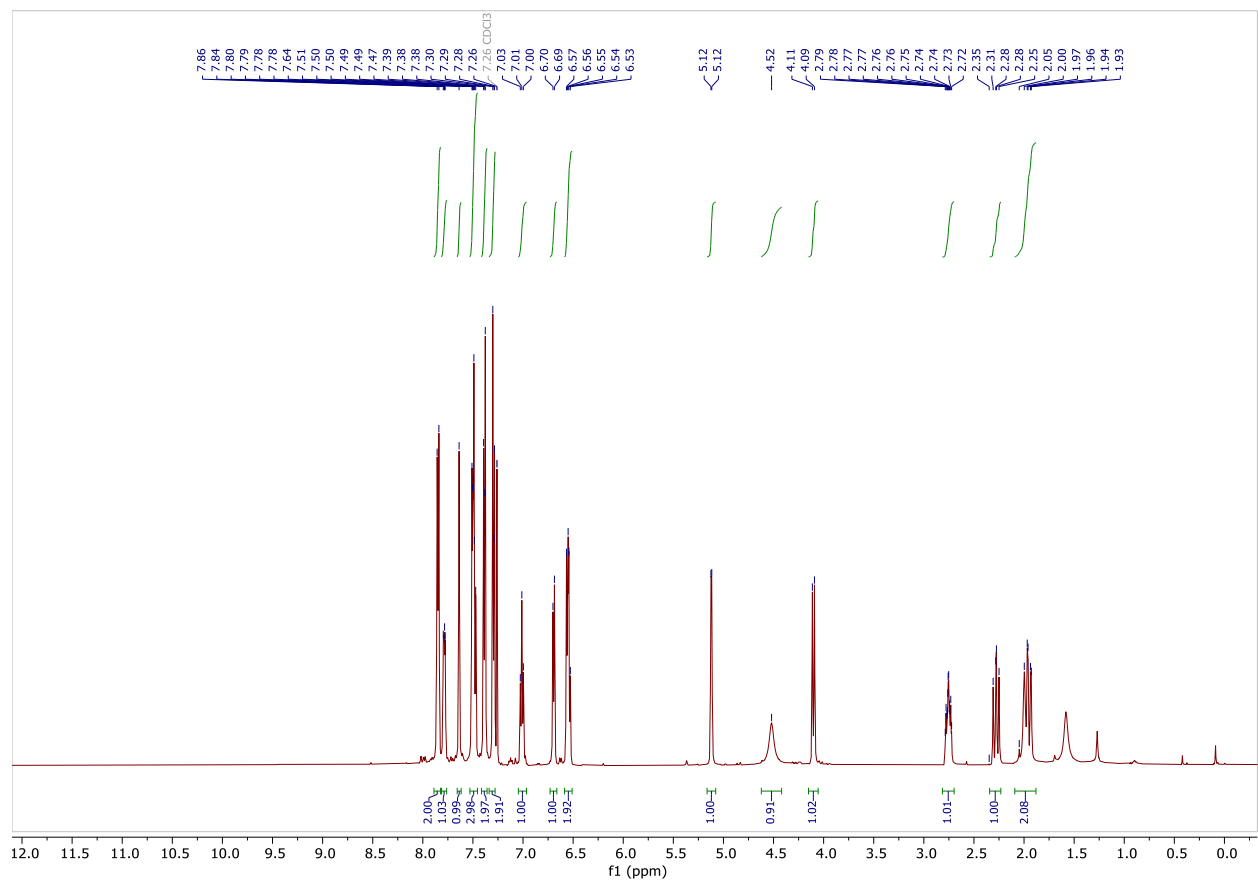


Figure S39 ^1H NMR (500 MHz) in CDCl_3 **8b'**

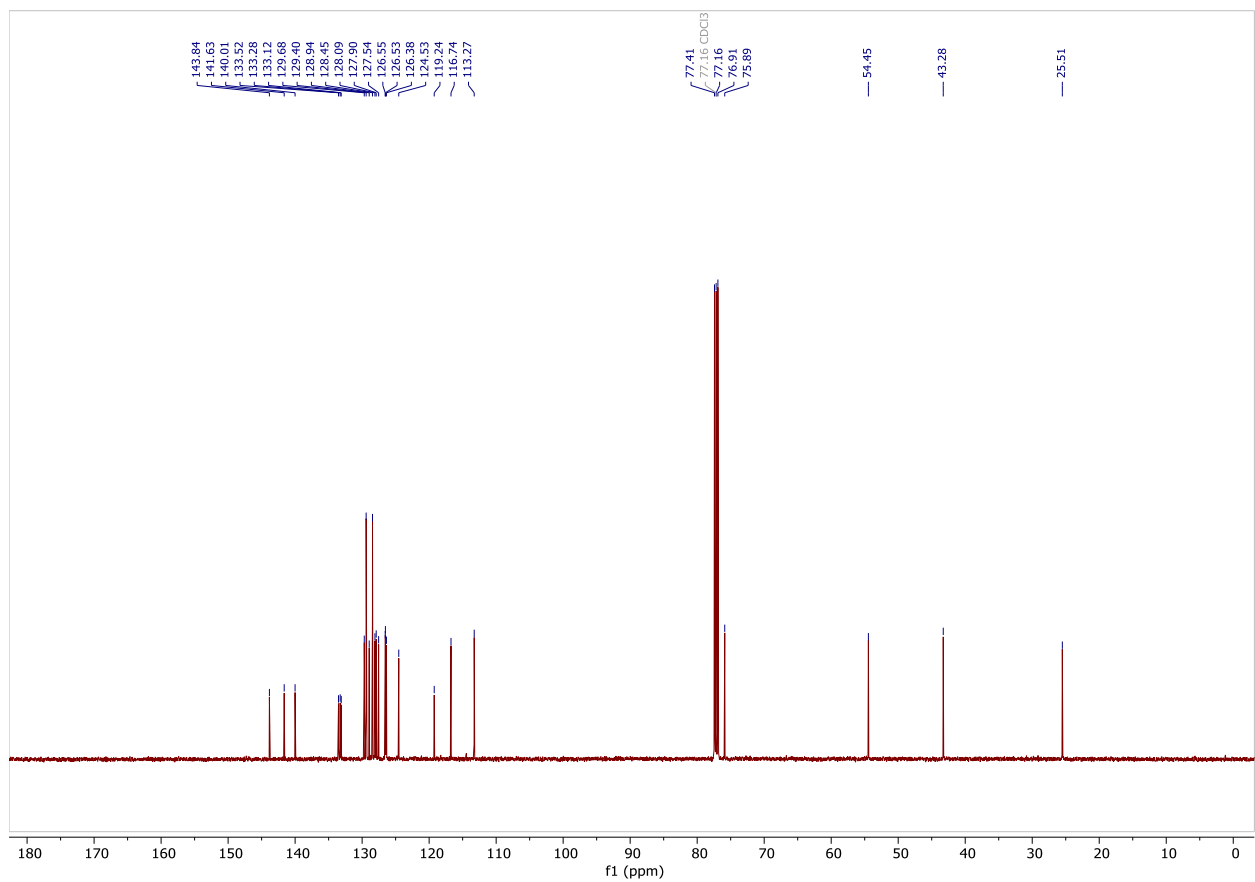


Figure S40 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **8b'**

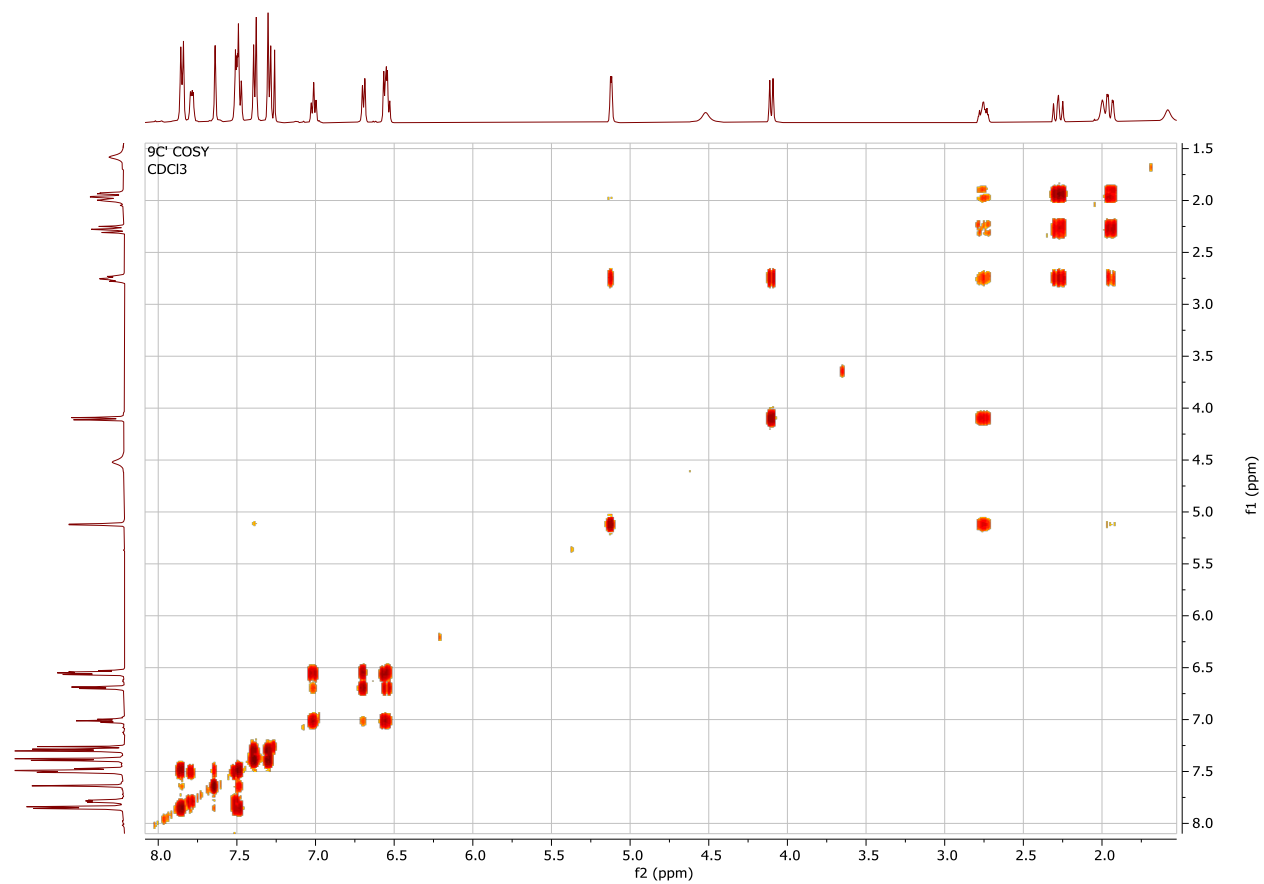


Figure S41 ¹H-¹H COSY NMR in CDCl₃ **8b'**

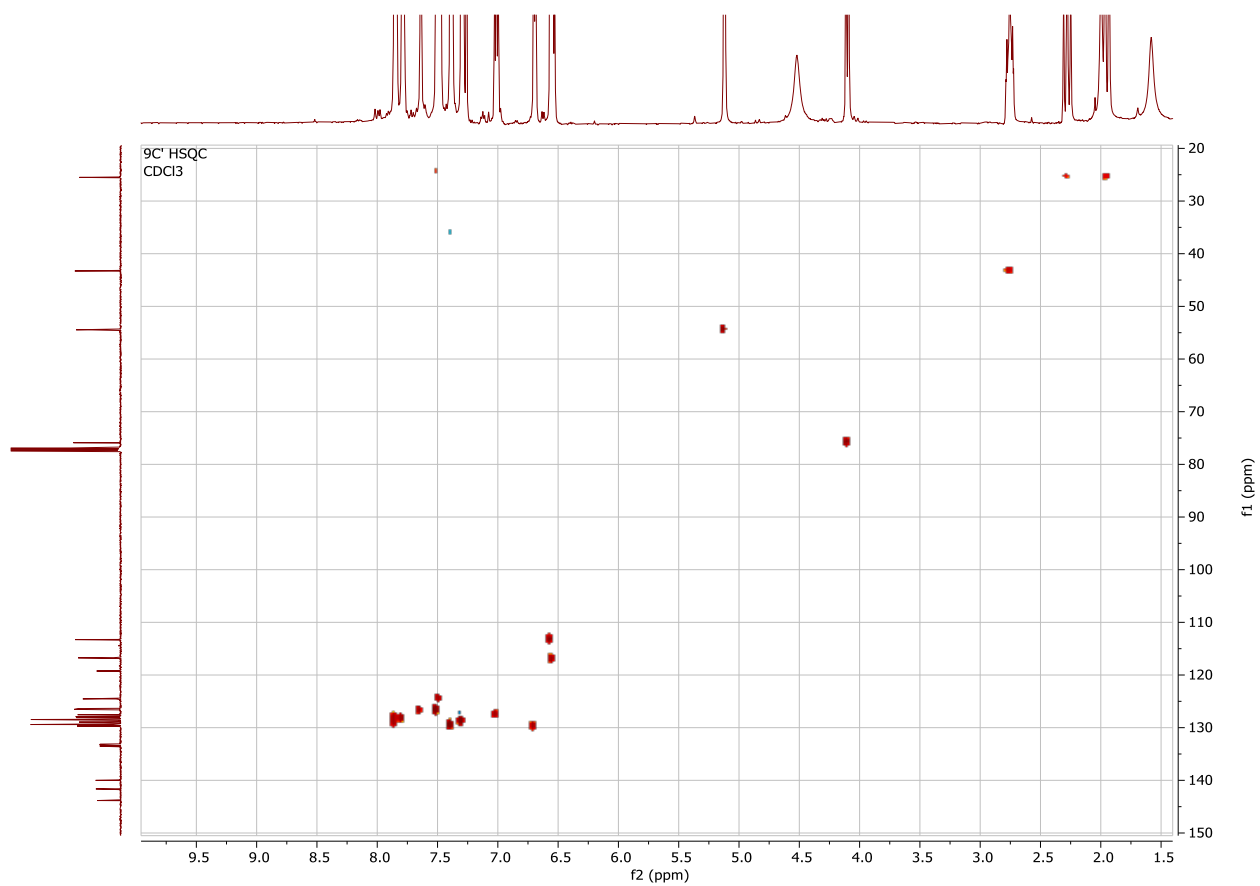


Figure S42 ¹H-¹³C HSQC NMR in CDCl₃ **8b'**

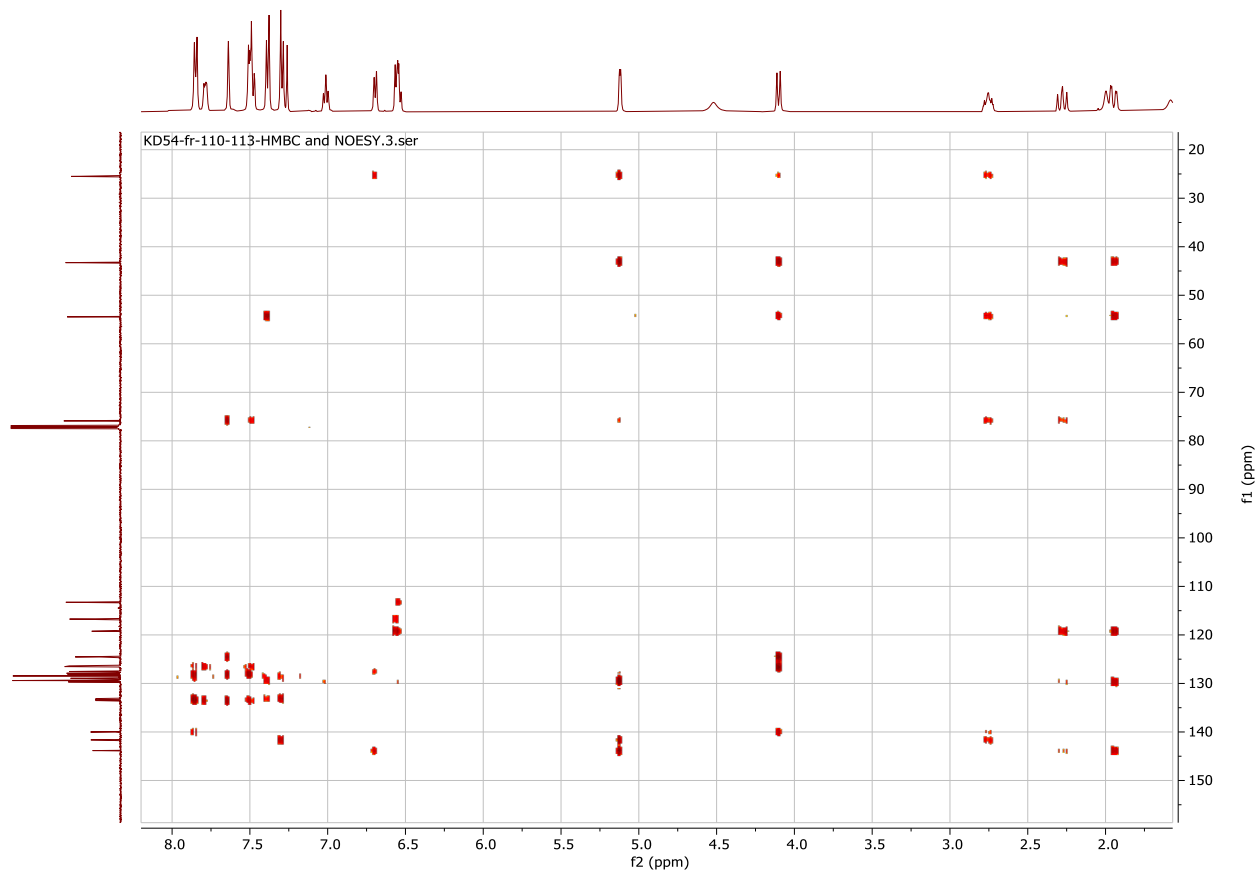


Figure S43 ^1H - ^{13}C HMBC NMR in CDCl_3 **8b'**

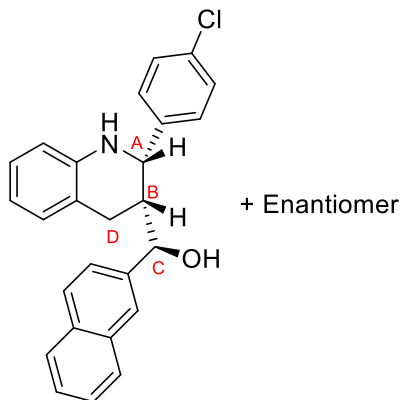


Table S4: NMR assignments for **8b'**.

Atom Label	¹ H NMR Peak (ppm)	Splitting and J Coupling (Hz)	¹³ C NMR Peak (ppm)
A	5.12	d, J = 4.3 (Confirms <i>syn</i> to B)	54.45
B	2.78	m	43.28
C	4.10	d, J = 10.4 (Confirms <i>anti</i> to B)	75.89
D	2.27, 1.95	dd, J = 16.4, 13.4; dd, J = 16.4, 4.1	25.51

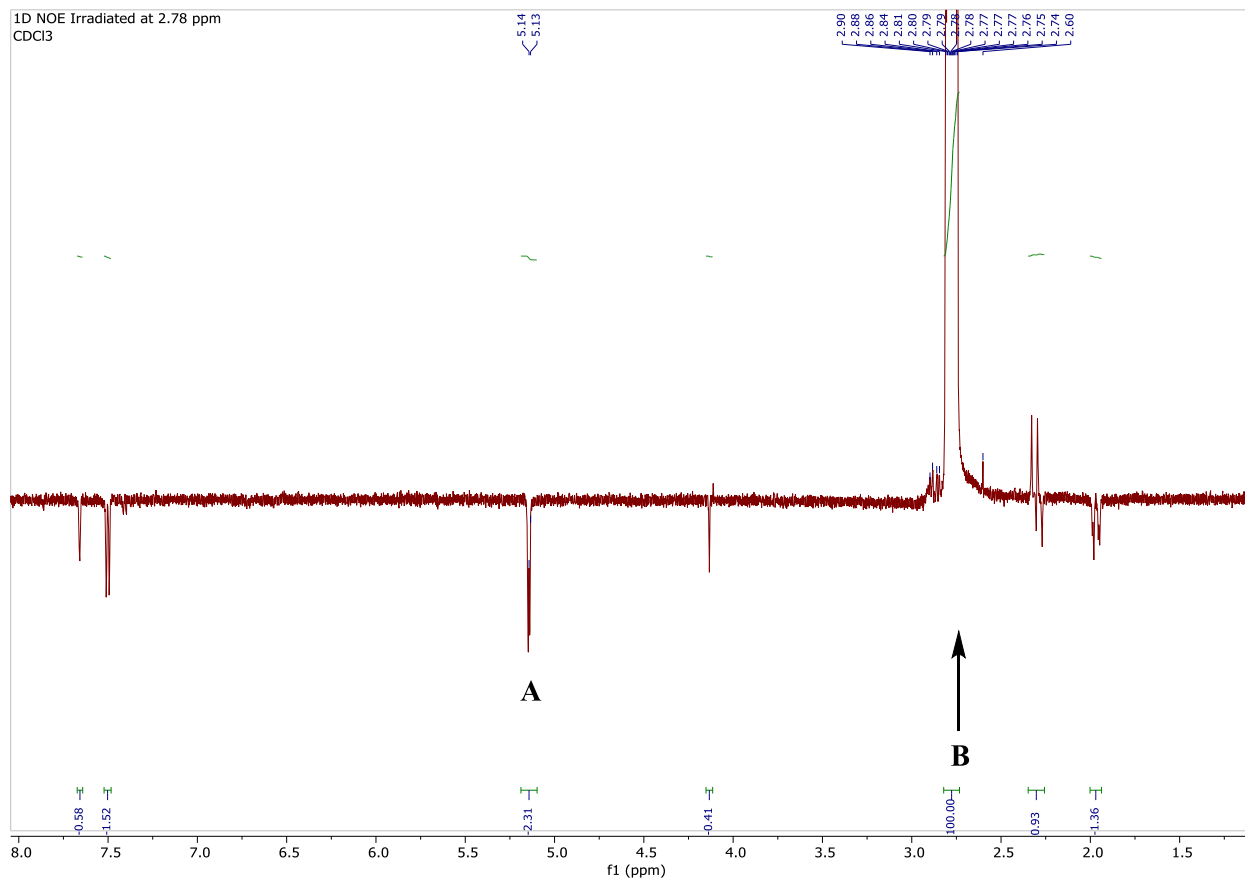


Figure S44 1D NOESY NMR of **8b'** irradiated at 2.78 ppm in CDCl₃.

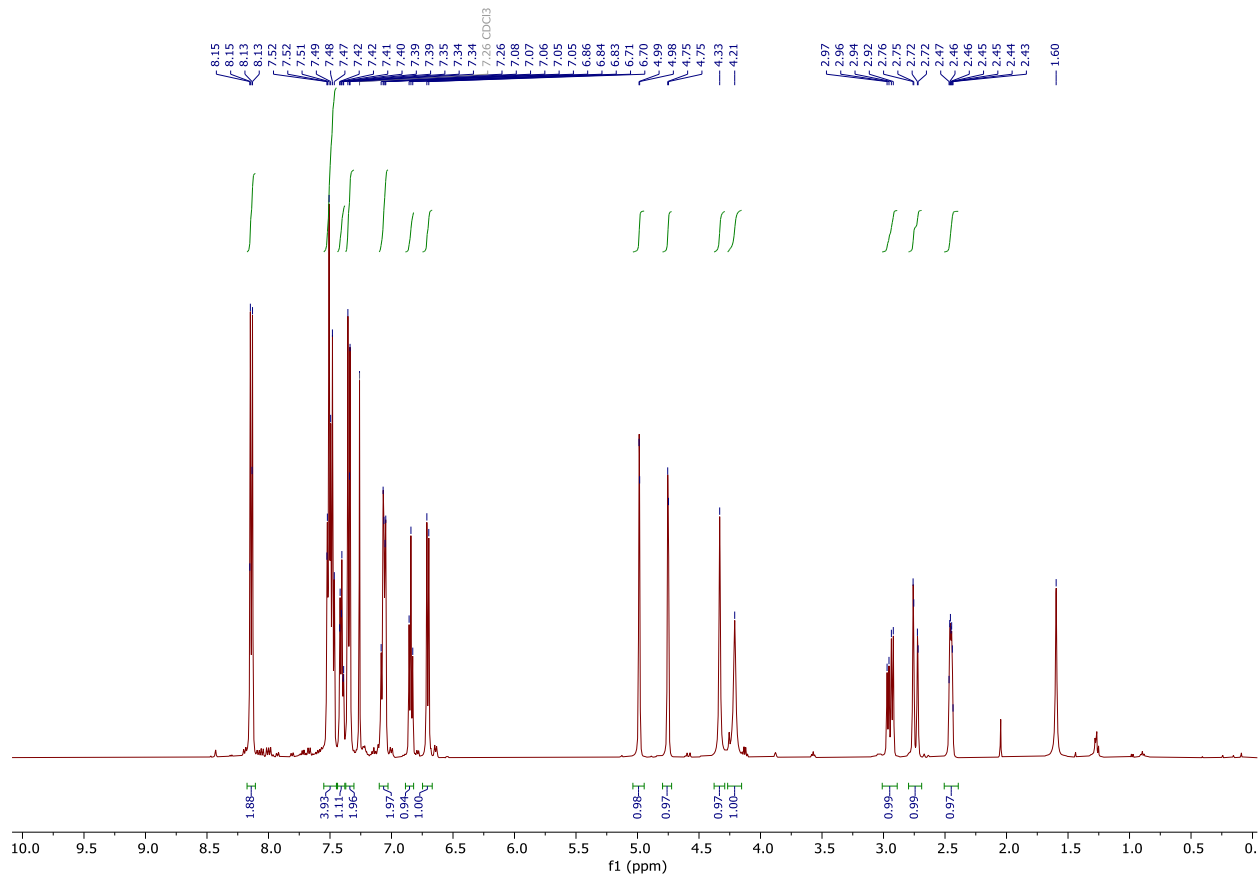


Figure S45 ^1H NMR (400 MHz) in CDCl_3 **8c**

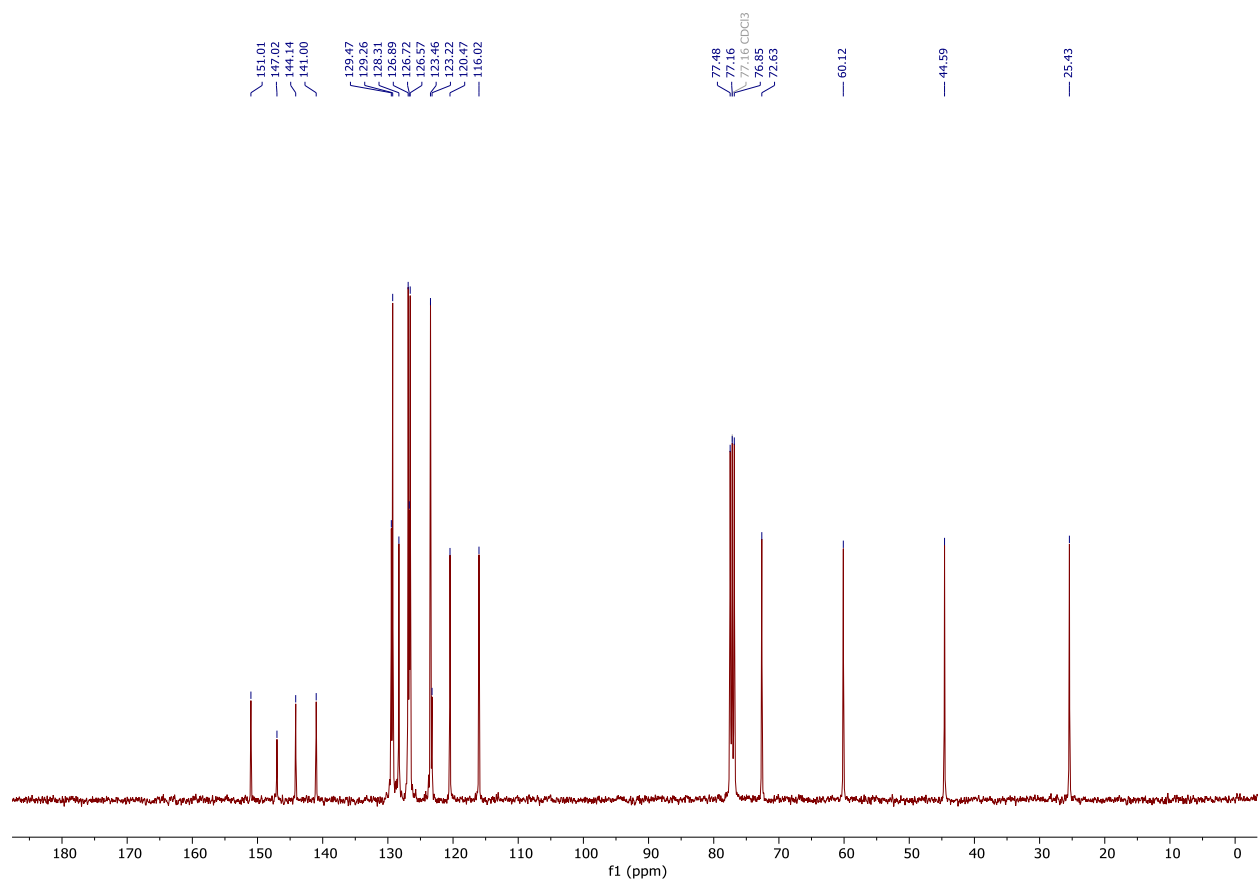


Figure S46 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **8c**

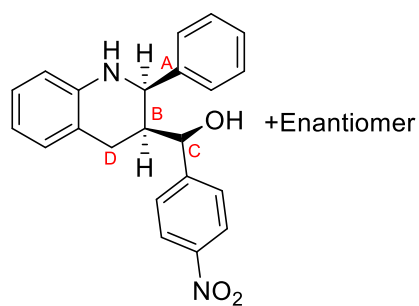


Table S5: NMR assignments for **8c**.

Atom Label	^1H NMR Peak (ppm)	Splitting and J Coupling (Hz)
A	4.75	d, J = 3.3 (Confirms <i>syn</i> to B)
B	2.47	m
C	4.99	d, J = 1.6 (Confirms <i>syn</i> to B)
D	2.95, 2.74	dd, J = 17.5, 7.8; dd, J = 17.5, 2.1

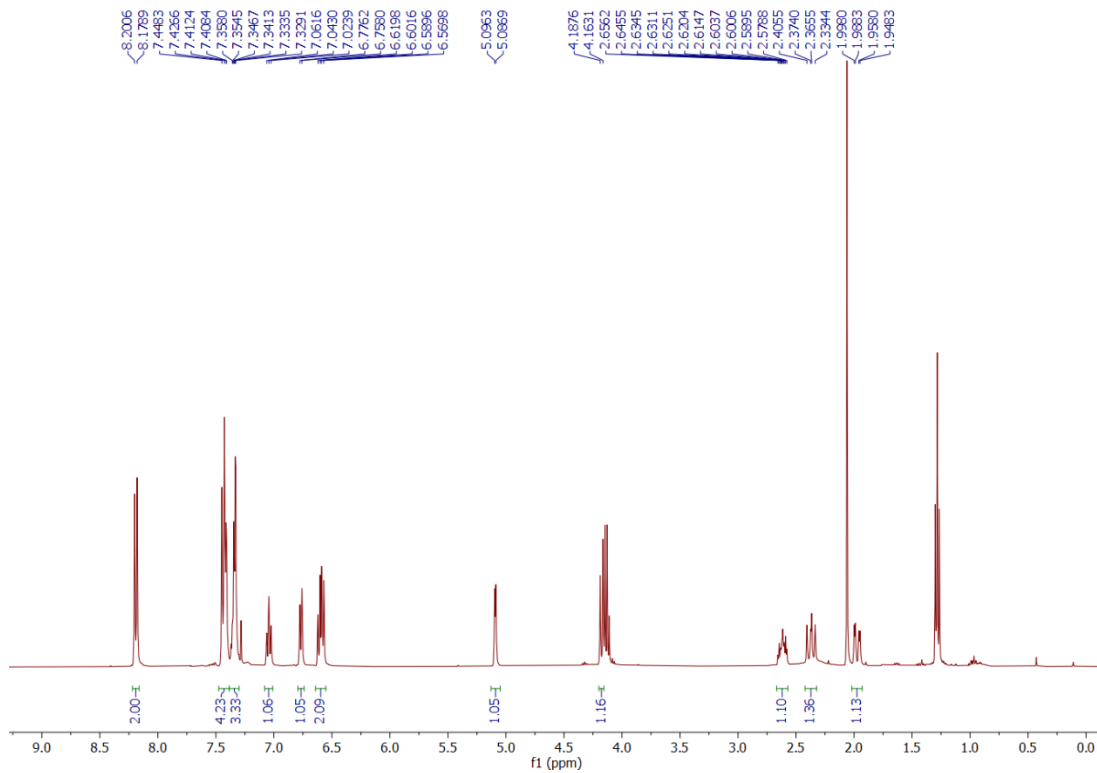


Figure S47 ^1H NMR (400 MHz) in CDCl_3 **8c'**

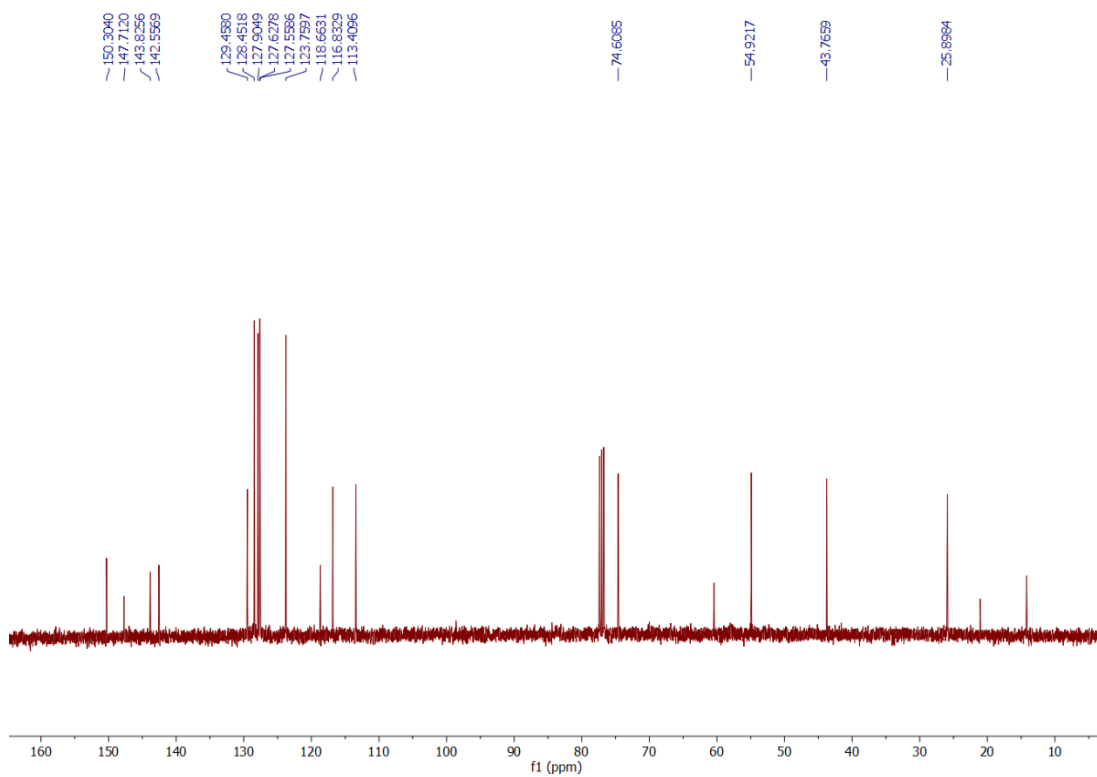


Figure S48 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 **8c'**

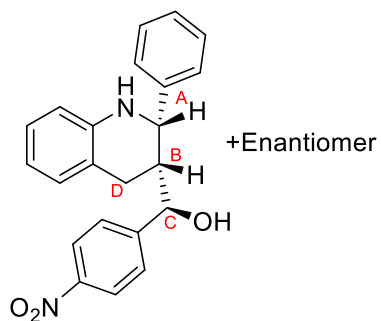


Table S6: NMR assignments for **8c'**.

Atom Label	¹ H NMR Peak (ppm)	Splitting and J Coupling (Hz)
A	5.09	d, J = 3.8 (Confirms <i>syn</i> to B)
B	2.64-2.58	m
C	4.18	d, J = 9.8 (Confirms <i>anti</i> to B)
D	2.37, 1.96	dd, J = 15.9, 12.5; dd, J = 16.0, 3.9

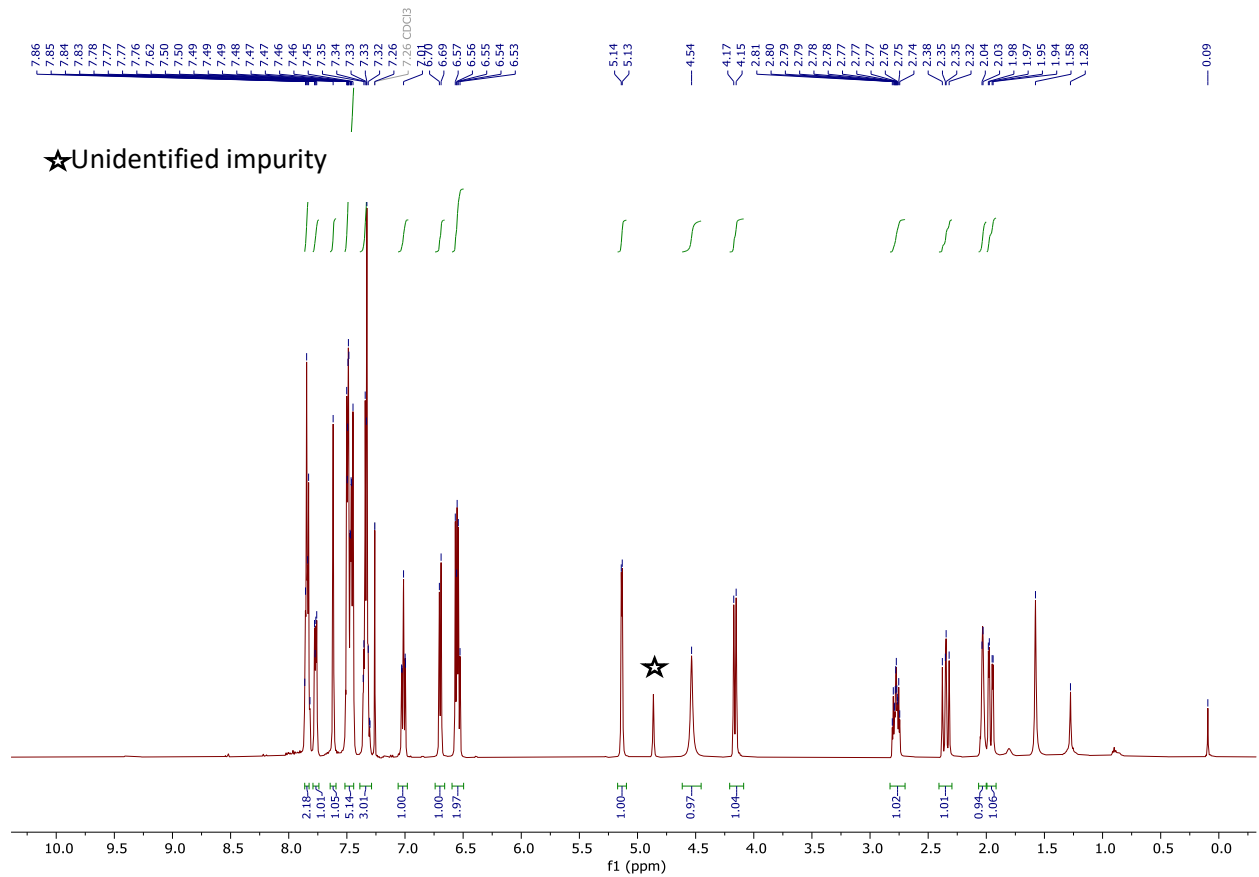


Figure S49 ^1H NMR (500 MHz) in CDCl_3 **8d'**

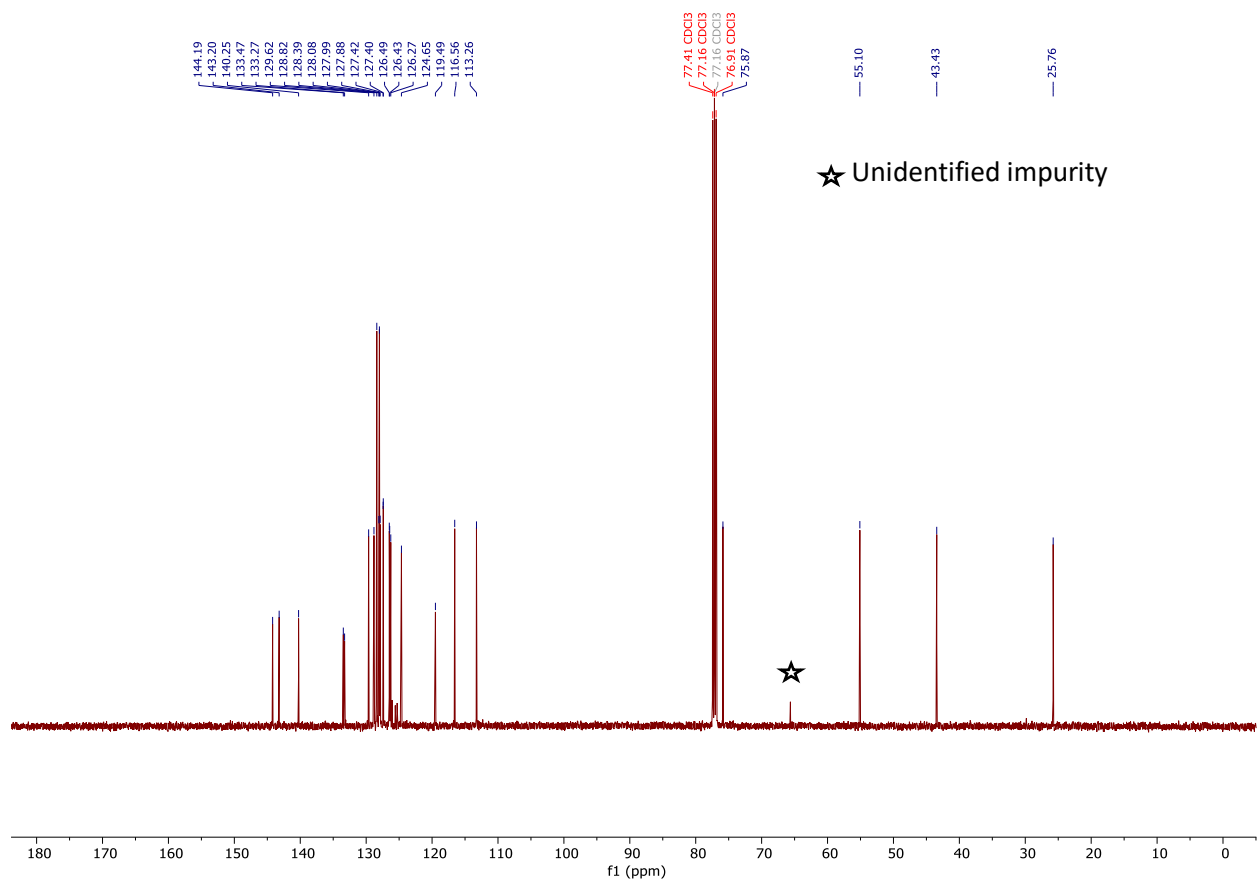


Figure S50 $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz) in CDCl_3 $8d'$

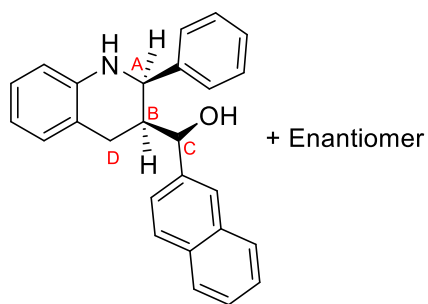


Table S7: NMR assignments for $8d'$.

Atom Label	^1H NMR Peak (ppm)	Splitting and J Coupling (Hz)
A	5.14	d, J = 4.3 (Confirms <i>syn</i> to B)
B	2.81-2.74	m
C	4.16	d, J = 10.3 (Confirms <i>anti</i> to B)
D	2.35, 1.96	dd, J = 16.3, 13.0; dd, J = 16.4, 4.2

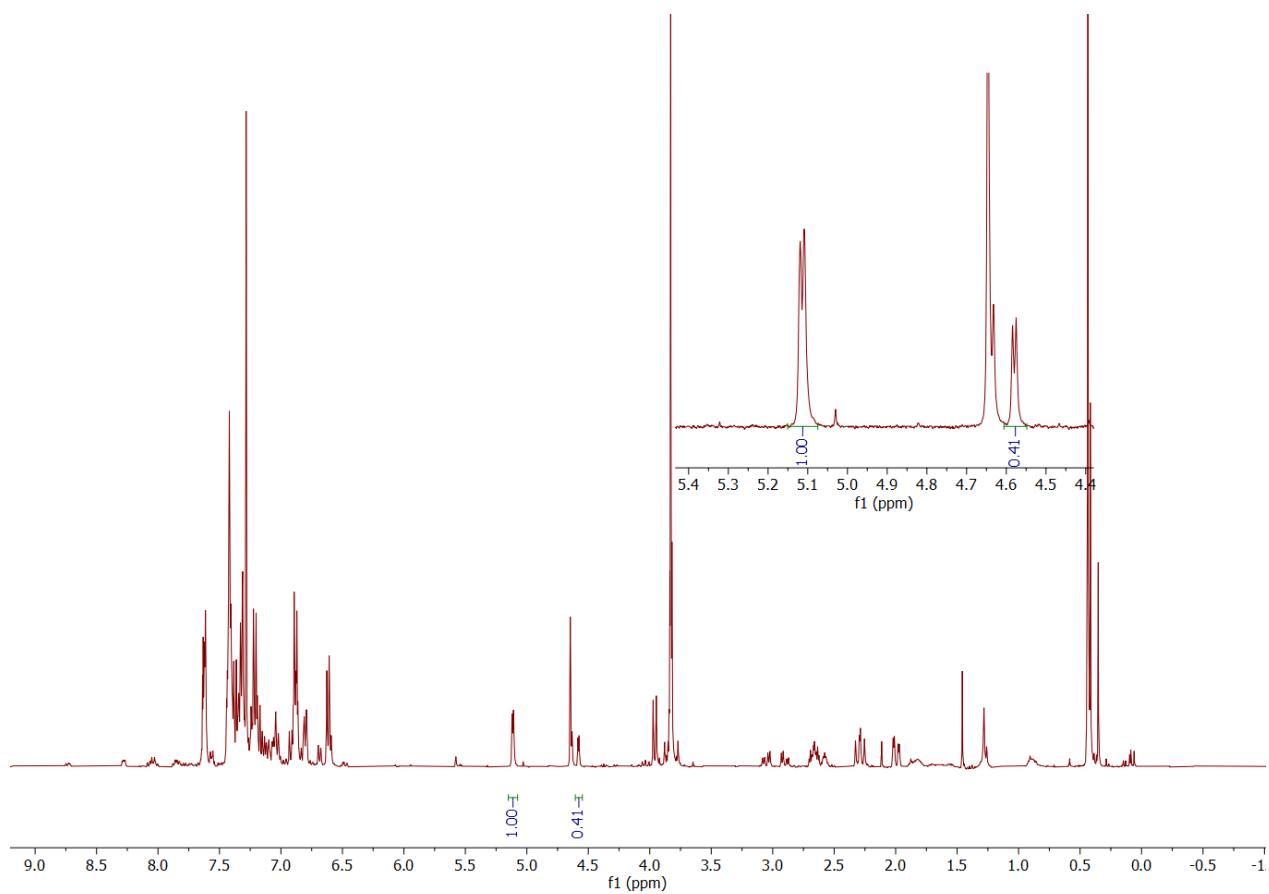


Figure S51 Crude ^1H NMR of **8a** to determine the crude diastereomeric ratio of **8a** (4.60 ppm signal) to **8a'** (5.08 ppm signal)

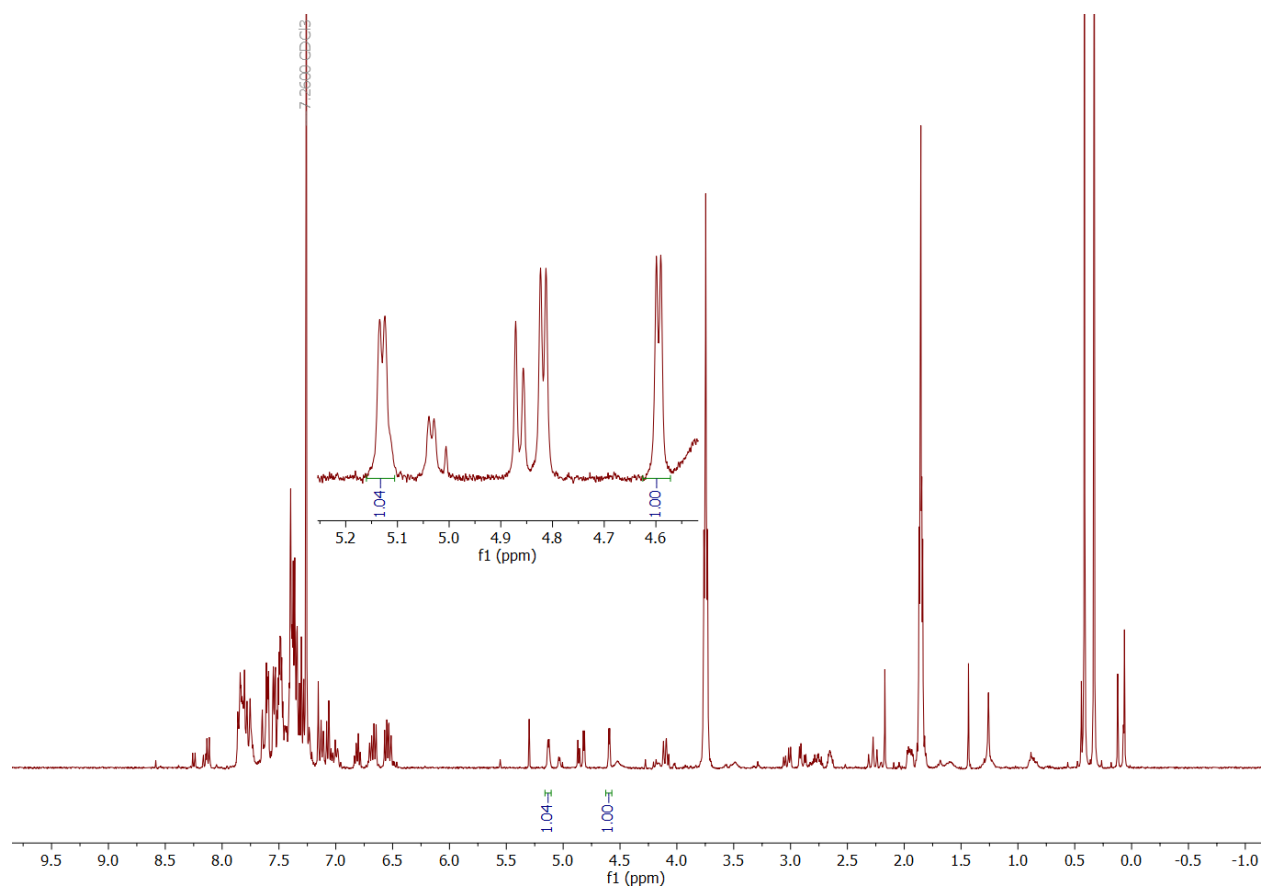


Figure S52 Crude ^1H NMR of **8b** to determine the crude diastereomeric ratio of **8b** (4.54 ppm signal) to **8b'** (5.12 ppm signal)

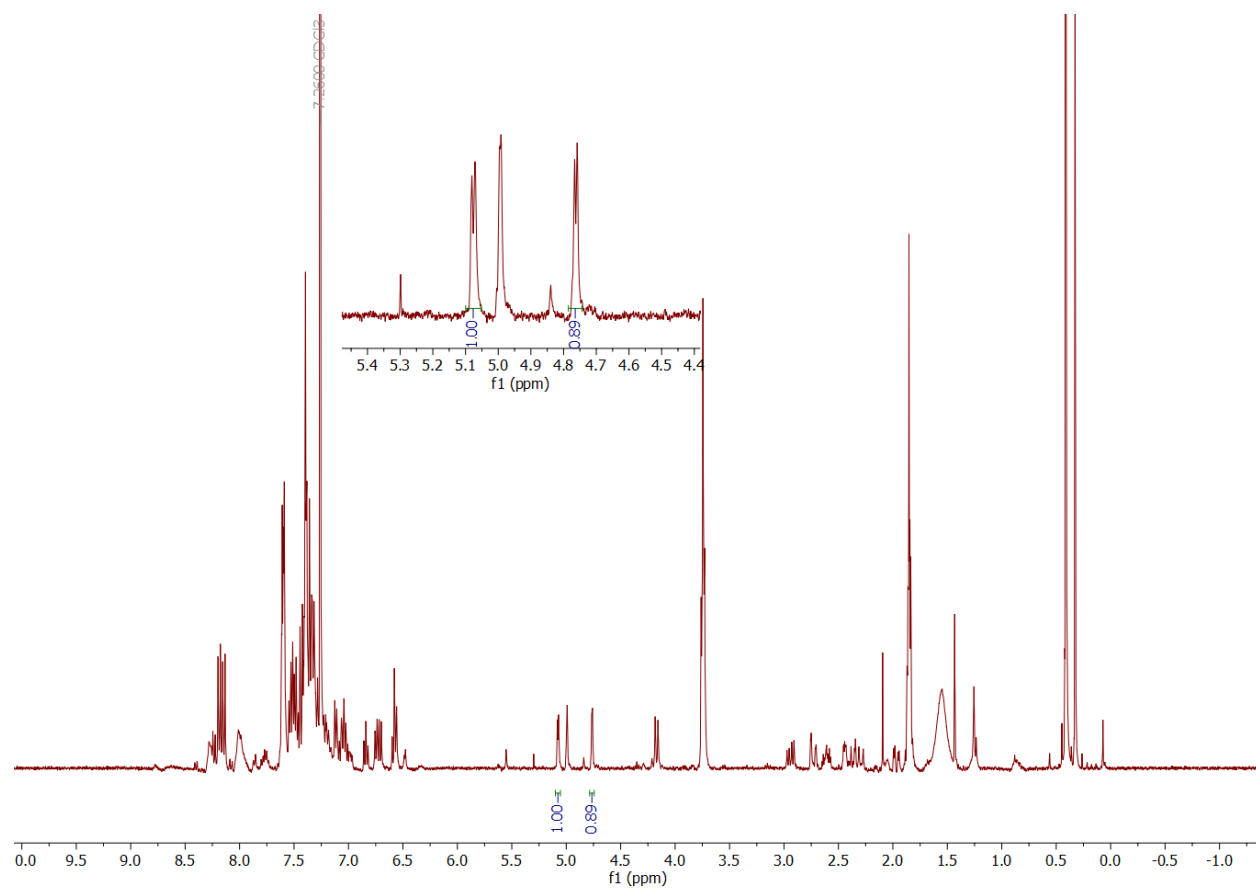


Figure S53 Crude ¹H NMR of **8c** to determine the crude diastereomeric ratio of **8c** (4.75 ppm signal) to **8c'** (5.09 ppm signal)

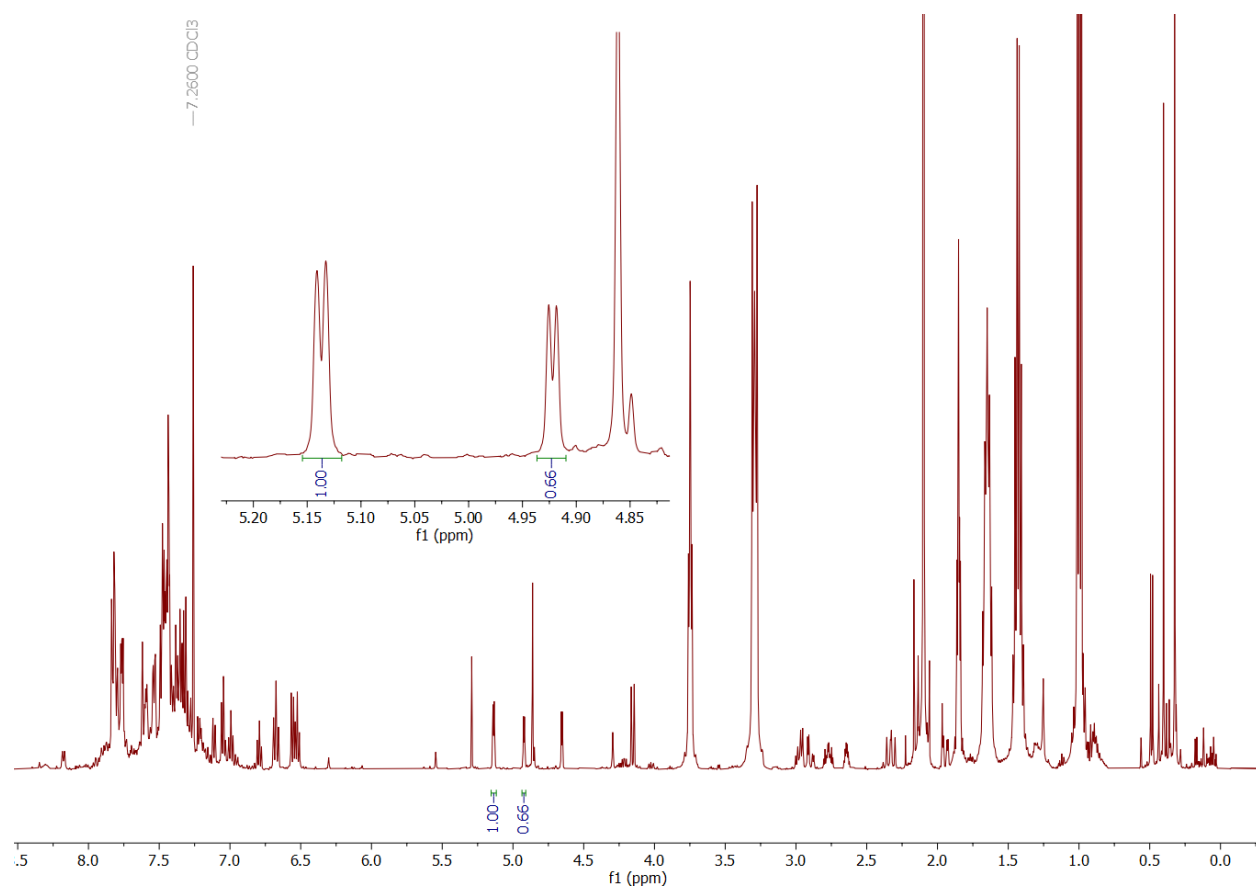


Figure S54 Crude ^1H NMR of **8d** to determine the crude diastereomeric ratio of **8d** (4.92 ppm signal) to **8d'** (5.14 ppm signal)

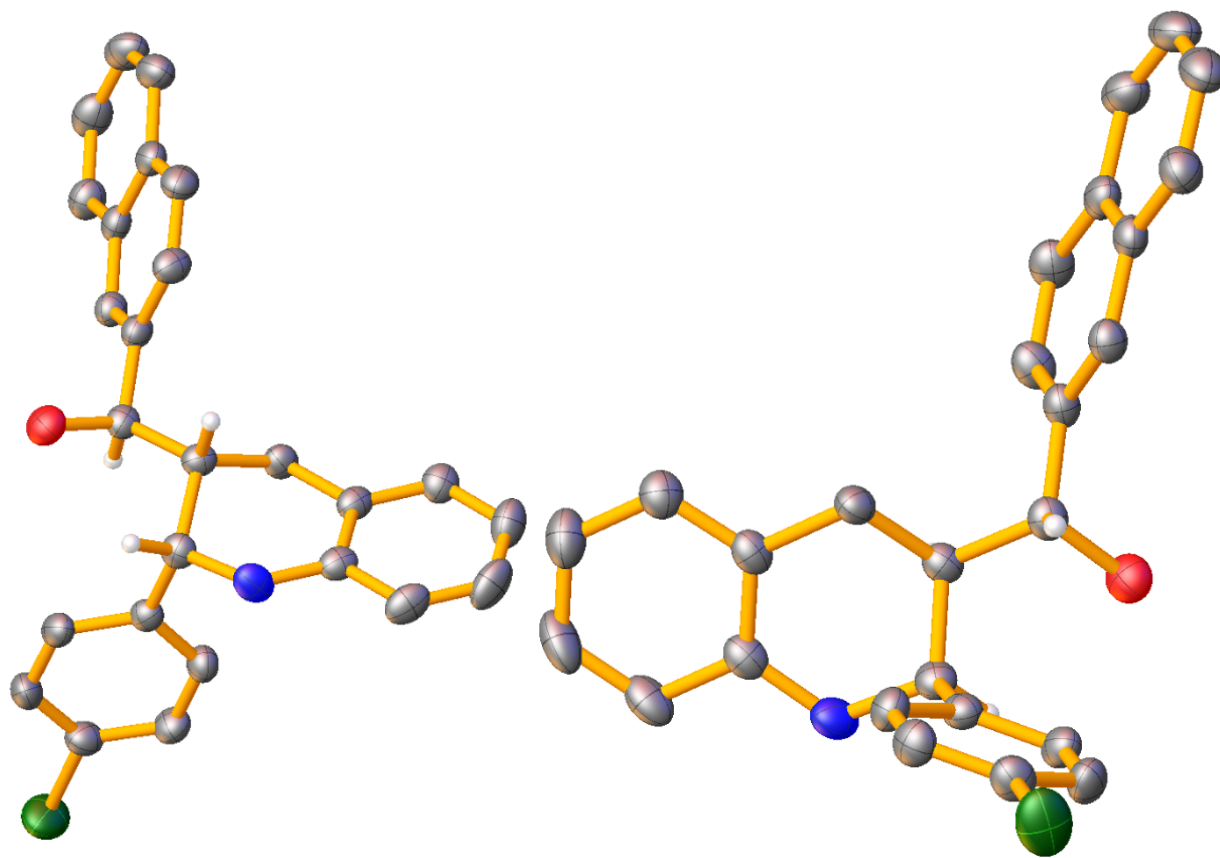


Figure S55 X-ray crystal structure of **8b'** in 2 different viewing orientations. Gray, blue, red, green and white ellipsoids (50% probability) represent carbon, nitrogen, oxygen, chlorine and hydrogen atoms, respectively. Hydrogen atoms on non-stereogenic atoms are omitted for clarity.

Table S3. Crystal data and structure refinement for **8b'**.

Empirical formula	C ₂₆ H ₂₂ ClNO	
Formula weight	399.89	
Temperature	180(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 7.5536(3) Å	a = 90°.
	b = 15.5289(6) Å	b = 98.8470(10)°.
	c = 19.9941(7) Å	g = 90°.
Volume	2317.39(15) Å ³	
Z	4	
Density (calculated)	1.146 Mg/m ³	
Absorption coefficient	0.180 mm ⁻¹	
F(000)	840	
Crystal size	0.223 x 0.197 x 0.134 mm ³	
Theta range for data collection	2.444 to 26.393°.	
Reflections collected	34942	
Independent reflections	4732 [R(int) = 0.0480]	
Completeness to theta = 25.242°	99.7 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4737 / 0 / 267	
Goodness-of-fit on F ²	1.064	
R indices (all data)	R1 = 0.0532, wR2 = 0.1219	
Largest diff. peak and hole	0.343 and -0.423 e.Å ⁻³	

CIF Check for Crystal Structure of 8b'.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) cckd_54dia2_25aug2020_0m_a_sqd

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. [CIF dictionary](#) [Interpreting this report](#)

Datablock: cckd_54dia2_25aug2020_0m_a_sqd

Bond precision: C-C = 0.0022 A Wavelength=0.71073

Cell: a=7.5536 (3) b=15.5289 (6) c=19.9941 (7)
 alpha=90 beta=98.847 (1) gamma=90

Temperature: 180 K

	Calculated	Reported
Volume	2317.39 (15)	2317.39 (15)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C26 H22 Cl N O	C26 H22 Cl N O
Sum formula	C26 H22 Cl N O	C26 H22 Cl N O
Mr	399.90	399.89
Dx,g cm-3	1.146	1.146
Z	4	4
Mu (mm-1)	0.180	0.180
F000	840.0	840.0
F000'	840.89	
h,k,lmax	9,19,24	9,19,24
Nref	4751	4737
Tmin,Tmax	0.961,0.976	0.702,0.745
Tmin'	0.961	

Correction method= # Reported T Limits: Tmin=0.702 Tmax=0.745
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta (max)= 26.393

R(reflections)= 0.0437(3970) wR2(reflections)= 0.1219(4737)

S = 1.064 Npar= 267

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

[PLAT601_ALERT_2_B](#) Unit Cell Contains Solvent Accessible VOIDS of . 190 Ang**3

Author Response: SQUEEZE was used to remove disordered toluene on an inversion center.

Alert level C

PLAT222_ALERT_3_C	NonSolvent Resd 1 H Uiso(max)/Uiso(min) Range	10.0 Ratio
PLAT420_ALERT_2_C	D-H Without Acceptor N1 --H3 .	Please Check
PLAT906_ALERT_3_C	Large K Value in the Analysis of Variance	4.532 Check
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L= 0.600	9 Report
PLAT918_ALERT_3_C	Reflection(s) with I(obs) much Smaller I(calc) .	1 Check

Alert level G

PLAT007_ALERT_5_G	Number of Unrefined Donor-H Atoms	1 Report
PLAT793_ALERT_4_C	Model has Chirality at C1 (Centro SPGR)	S Verify
PLAT793_ALERT_4_C	Model has Chirality at C2 (Centro SPGR)	R Verify
PLAT793_ALERT_4_C	Model has Chirality at C10 (Centro SPGR)	R Verify
PLAT910_ALERT_3_C	Missing # of FCF Reflection(s) Below Theta(Min).	2 Note
PLAT912_ALERT_4_C	Missing # of FCF Reflections Above STh/L= 0.600	4 Note
PLAT913_ALERT_3_C	Missing # of Very Strong Reflections in FCF	3 Note
PLAT933_ALERT_2_C	Number of OMIT Records in Embedded .res File ...	2 Note
PLAT961_ALERT_5_C	Dataset Contains no Negative Intensities	Please Check
PLAT978_ALERT_2_C	Number C-C Bonds with Positive Residual Density.	17 Info
PLAT992_ALERT_5_C	Repd & Actual _reflns_number_gt Values Differ by	2 Check

0 **ALERT level A** = Most likely a serious problem - resolve or explain
1 **ALERT level B** = A potentially serious problem, consider carefully
5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
11 **ALERT level G** = General information/check it is not something unexpected

0 **ALERT type 1** CIF construction/syntax error, inconsistent or missing data
4 **ALERT type 2** Indicator that the structure model may be wrong or deficient
6 **ALERT type 3** Indicator that the structure quality may be low
4 **ALERT type 4** Improvement, methodology, query or suggestion
3 **ALERT type 5** Informative message, check

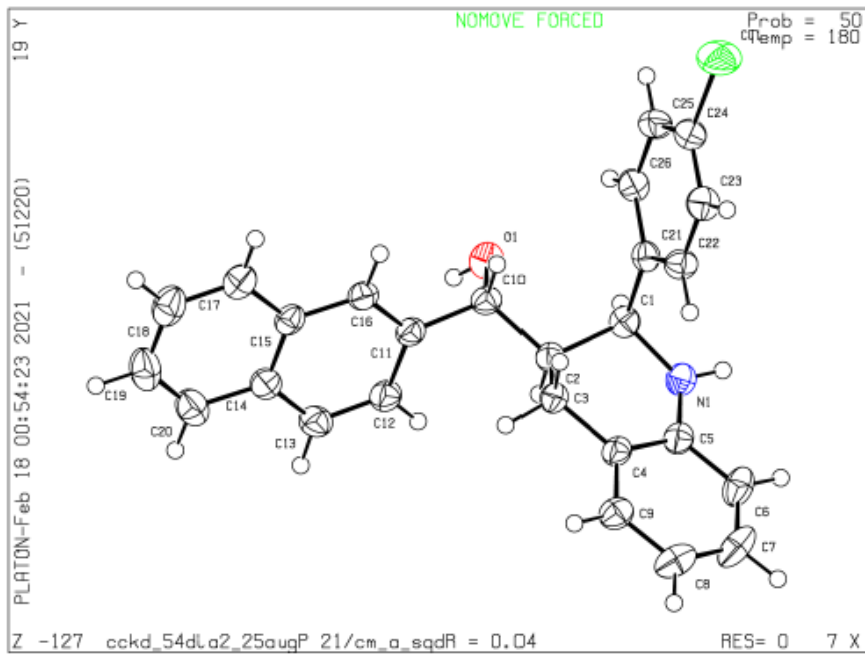
It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

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A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.



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