Supporting Information

NHC-catalyzed base assisted C-C bond cleavage of cyclopropenone: an approach towards synthesis of azetidinone and benzoxazepines

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1. General Information:[1]

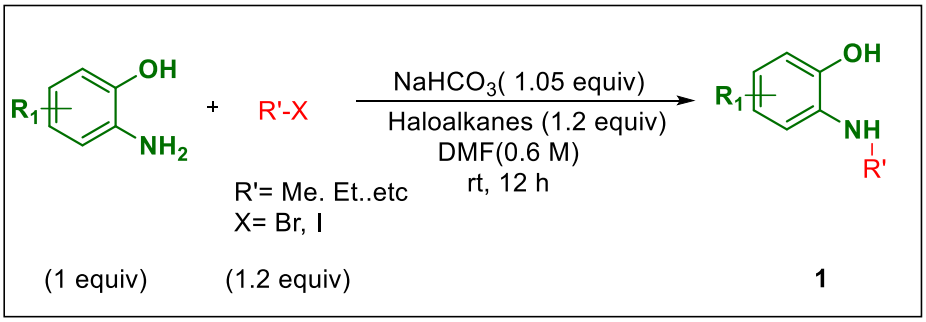
Reactions were performed using borosil schlenk tube vial under N_2 atmosphere. Column chromatography was done by using 100-200 & 230-400 mesh size silica gel of Acme Chemicals. A gradient elution was performed by using distilled petroleum ether and ethyl acetate. TLC plates detected under UV light at 254 nm. ^{1}H NMR and ^{13}C NMR were recorded on Bruker AV 400, 700 MHz spectrometer using $CDCl_3$ and DMSO-d_6 as NMR solvents. The residual $CHCl_3$ and DMSO-H_6 for ^{1}H NMR (δ = 7.26 ppm and 2.54 ppm respectively) were used as reference. The deuterated solvent signal for ^{13}C NMR (δ = 77.36 ppm 40.45 ppm) is used as reference.[2] Multiplicity (s = single, d = doublet, t = triplet, q = quartet, m = multiplet, dd = double doublet), integration, and coupling constants (*J*) in hertz (Hz). HRMS signal analysis was performed using micro TOF Q-II mass spectrometer. X-ray analysis was conducted using Rigaku Smartlab X-ray diffractometer at SCS, NISER. Reagents and starting materials were purchased from Sigma Aldrich, Alfa Aesar, TCI, Avra, Spectrochem and other commercially available sources and used without further purification unless otherwise noted.

Abbreviations:

TEMPO = 2,2,6,6-Tetramethylpiperidine 1-oxyl, BHT = Butylated hydroxytoluene, TLC = Thin layer chromatography, EtOAc = Ethyl acetate, DCM = Dichloromethane, MeOH = Methanol, IPr·HCl = 1,3-Bis(2,6-diisopropylphenyl) imidazole-2-ylidene, $AlCl_3$ = Aluminium chloride, $NaHCO_3$ = Sodium bicarbonate, Na_2SO_4 = Sodium sulphate, DMF = Dimethyl formamide.

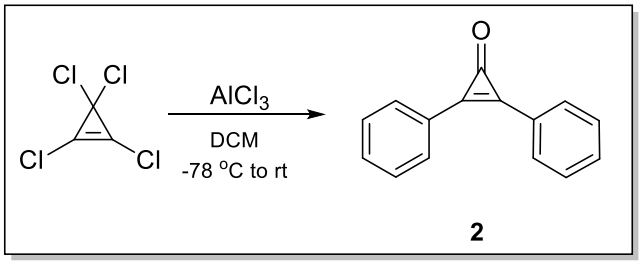
2. General procedures:

(a) General Procedure for *N*-Protection of Aminophenol 1:[3]



To an oven dried seal tube charged with a stir bar, sodium bicarbonate (0.95 mmol, 1.05 equiv) was added under nitrogen atmosphere and vaccum was applied. *Ortho*-aminophenol (0.91 mmol, 1 equiv) followed by dimethylformamide (1.51 ml, 0.6 M) were added under nitrogen atmosphere. The resulting solution was kept under ice bath followed by dropwise addition of halo alkanes (1.09 mmol, 1.2 equiv) where R'= (Methyl, ethyl, etc) and X = (I, and Br). The mixture was allowed to stir for 12 h at room temperature. After completion of the reaction (monitored by TLC), the organic phase was washed thrice with DCM, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel using EtOAc /hexane as eluent to provide the desired product. The product **1** was directly used for the next step.

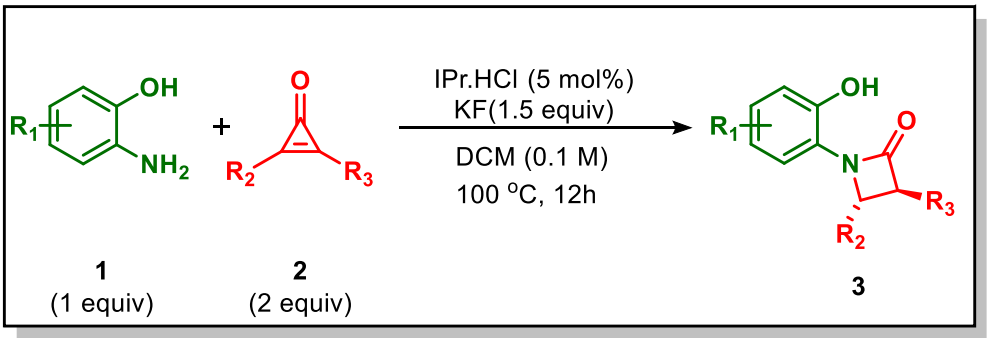
(b) General Procedure for the Synthesis of Cyclopropenones 2:[4]



To a suspension of tetra chloro-cyclopropene **1,** (0.64 mmol, 1 equiv) and anhydrous $AlCl_3$ (1.35 mmol, 1.05 equiv) in CH_2Cl_2 (0.06M, 10 mL) was added dropwise a solution of benzene (1.28 mmol, 2equiv) in CH_2Cl_2 (1.2 M, 1 mL) at −78 °C. The mixture was stirred for 2 h, warmed to room temperature, and stirred for another 2 h. After completion of the reaction as monitored by TLC analysis, the resulting mixture was quenched with water, diluted with CH_2Cl_2, and washed with water (2 × 50 mL) and brine (2 × 50 mL). The organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to yield the

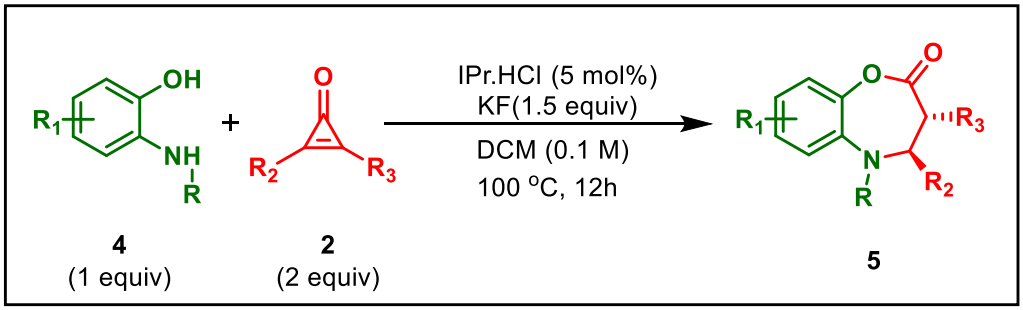
crude residue as an orange oil. The crude residue was then purified by flash column chromatography on silica gel (20% EtOAc in hexanes) to afford diaryl cyclopropenone **2,** (175 mg, 85% yield) as a white solid.

(c) General Procedure for the synthesis of four membered 2-azetidinone derivatives 3:



To an oven dried seal tube charged with a stir bar, *o*-aminophenol **1** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2** (0.2 mmol, 2.00 equiv), IPr.HCl (0.005 mmol, 0.05 equiv), were added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), solvent was evaporated under reduced pressure, and the crude was purified by column chromatography to get a (**3,4-diphenylazetidin-2-one**) **3** as a product.

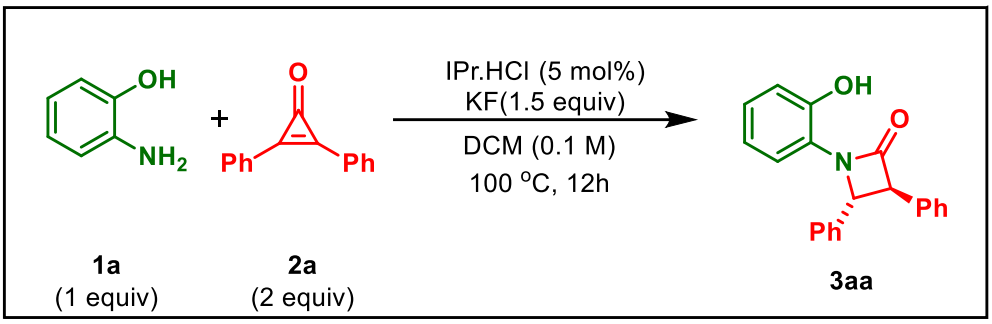
(d) General Procedure for synthesis of seven membered benzoxazepine derivative 5:



To an oven dried seal tube charged with a stir bar, *o*-aminophenol **4** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2** (0.2 mmol, 2.00 equiv), IPr.HCl (0.005 mmol, 0.05 equiv), were added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction

(monitored by TLC), the solvent was evaporated under reduced pressure, and the crude was purified by column chromatography using EtOAc/hexane as eluent to get corresponding seven membered benzoxazepine derivative (**4,5-dihydrobenzo[b][1,4] oxazepin-2(3H)-one**) **5** as a product.

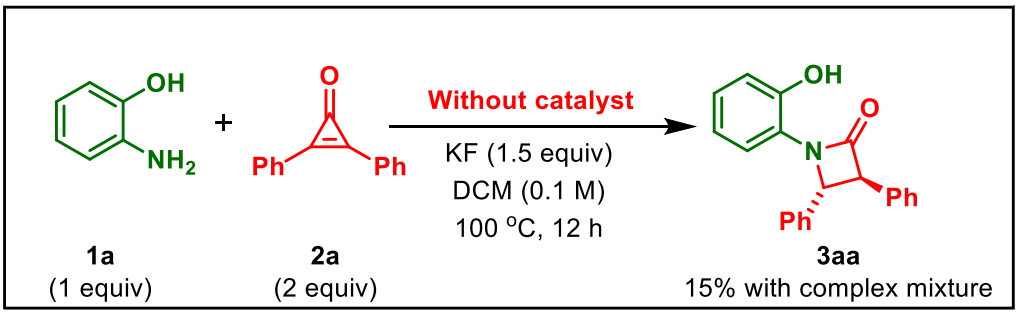
(e) General Procedure for 1 mmol scale synthesis of 2-azetidinone derivatives 3aa:



To an oven dried seal tube charged with a stir bar, *o*-aminophenol **1a** (1 mmol, 1equiv), diphenylcyclopropenone **2a** (2 mmol, 2.0 equiv), ligand (0.05 mmol, 0.05 equiv), were added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 ℃ for 12 h**. After completion of the reaction (monitored by TLC), with the addition of the silica gel in EtOAc to the residue, the solvent was evaporated under reduced pressure, and the crude was purified by column chromatography to get a **3aa** (160 mg, 51%) as a product**.**

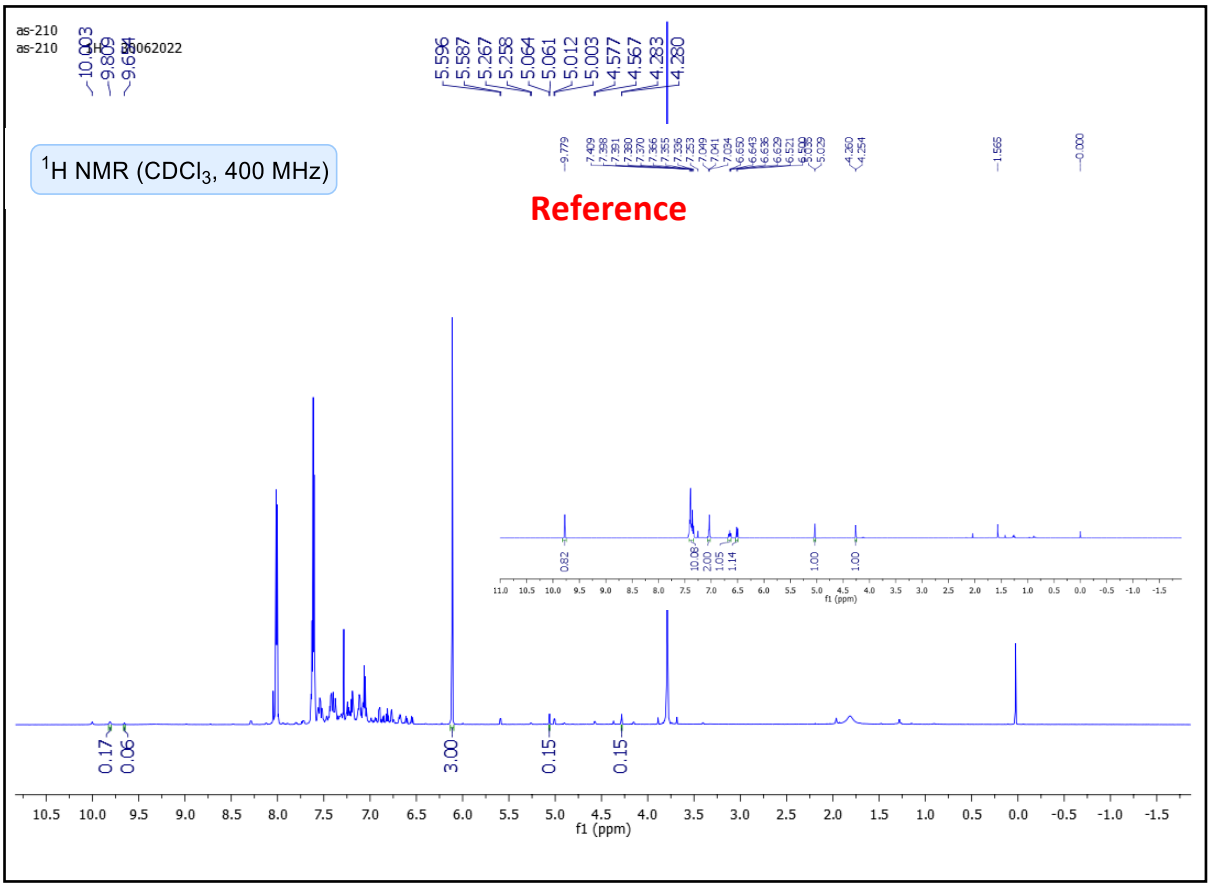
3. Control and mechanistic experiments:

(a) Procedure for reaction without catalyst:

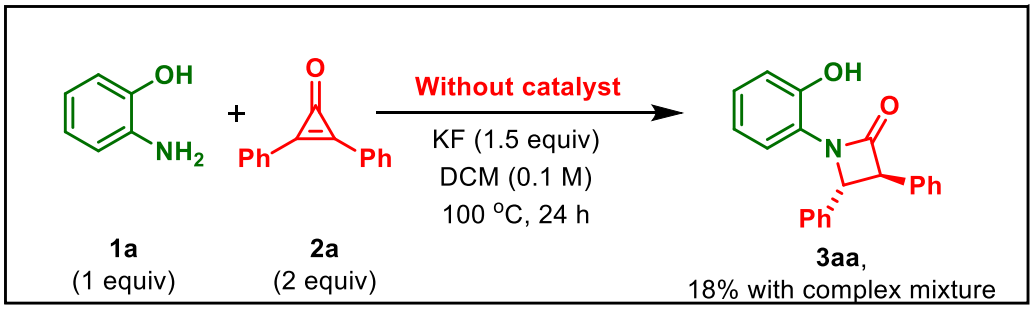


To an oven dried seal tube charged with a stir bar, *o*-aminophenol **1a** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), were added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added

inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), the crude was filtered through short silica bed and washed with EtOAc. The residue was evaporated under reduced pressure. The crude NMR was recorded and 15% of yield of the **3aa** along with complex reaction mixture was observed.

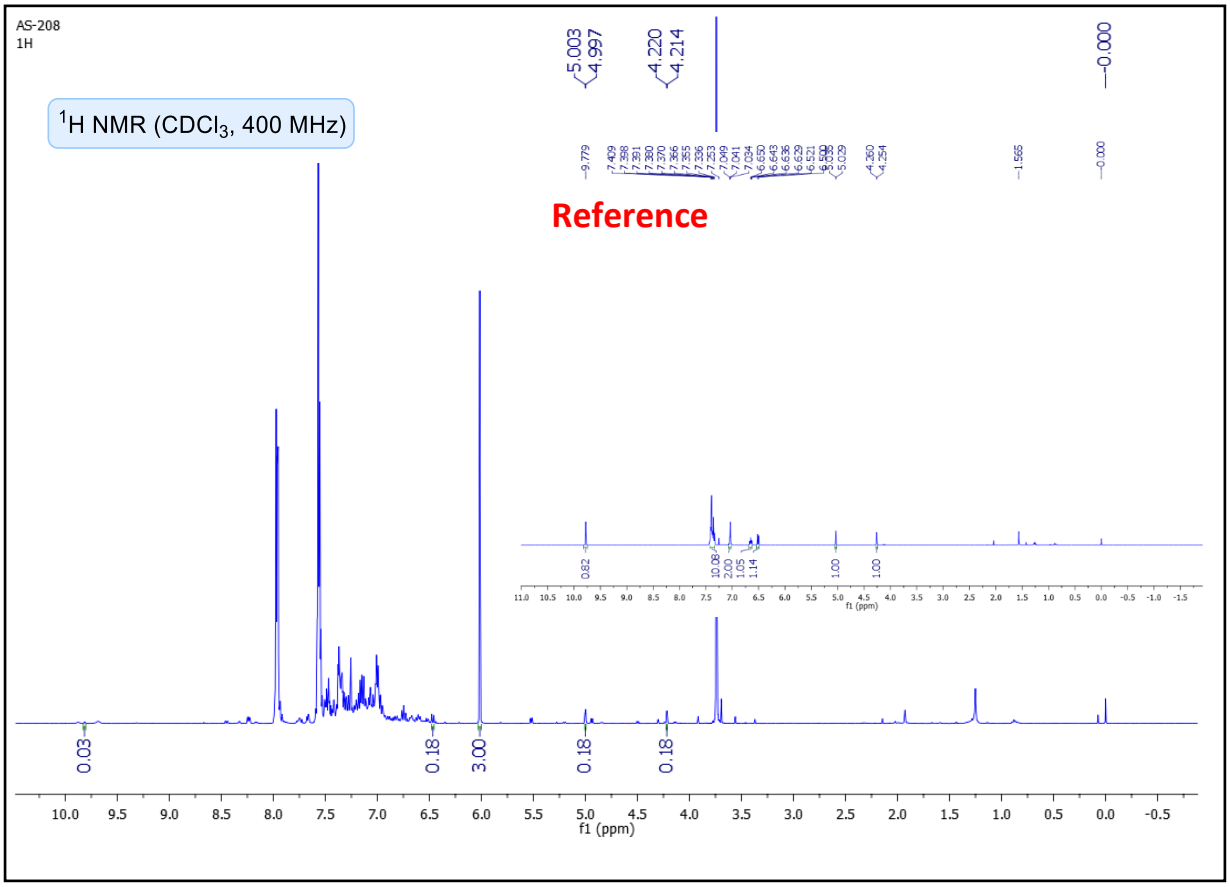


(b) Procedure for reaction with base and without catalyst for prolonged time:

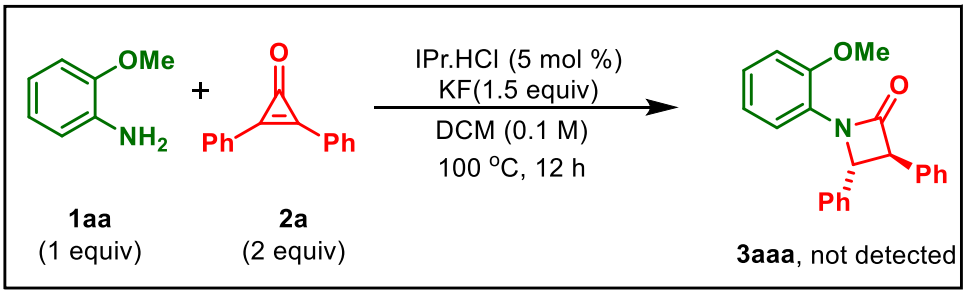


To an oven dried seal tube charged with a stir bar, *o*-aminophenol **1a** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), ligand (5 mol%, 0.05 equiv), were added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a

preheated aluminium block at **100 °C for 24 h**. After completion of the reaction (monitored by TLC) the crude was filtered through short silica bed and washed with EtOAc. The residue was evaporated under reduced pressure. The crude NMR was recorded. The desired product **3aa** was not observed.

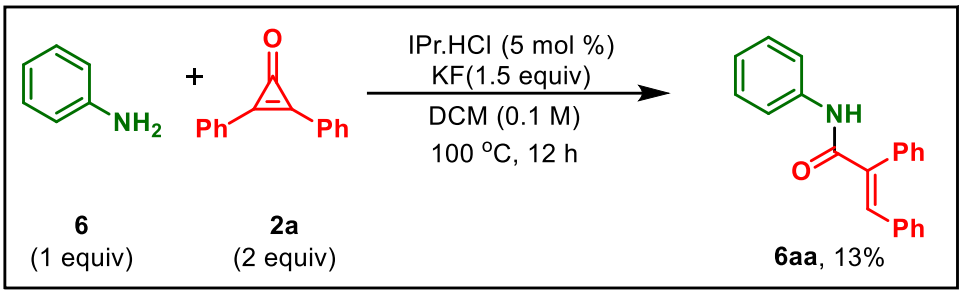


(c) The role of the hydroxy group:

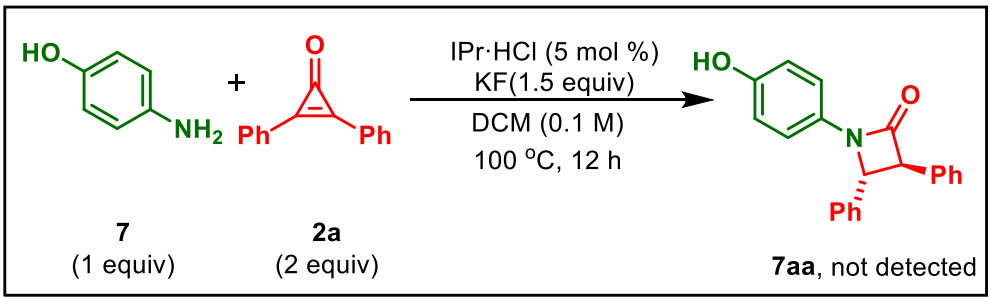


(a) To an oven dried seal tube charged with a stir bar, *o*-amino anisole **1aa** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), IPr.HCl (5 mol %, 0.05 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored

by TLC), the crude was filtered and residue was evaporated under reduced pressure. The crude NMR was recorded and the product **3aaa** was not detected.

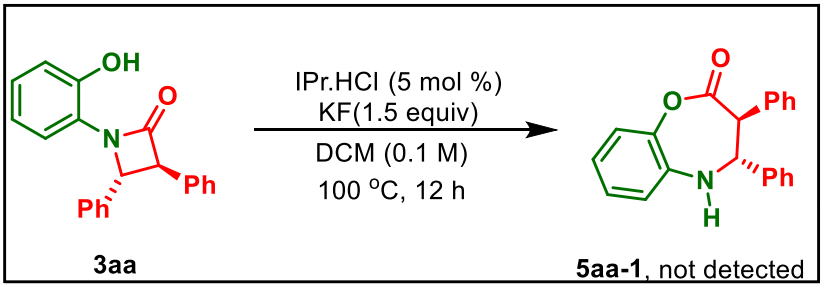


(b) To an oven dried seal tube charged with a stir bar, aniline **6** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), IPr.HCl (5 mol%, 0.05 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), the crude was filtered and residue was evaporated under reduced pressure. The crude NMR was recorded and the product **6aa** was detected.



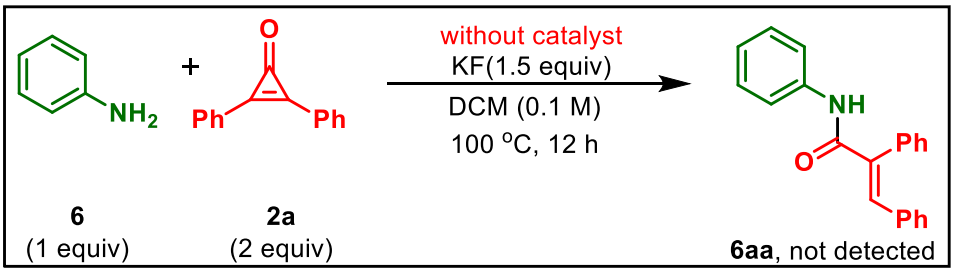
(c) To an oven dried seal tube charged with a stir bar, *p*-hydroxy aniline **7** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), IPr.HCl (5 mol%, 0.05 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), the crude was filtered and residue was evaporated under reduced pressure. The crude NMR was recorded and the product **7aa** was not detected.

(d) Reaction to ascertain the intermediacy of β-lactam:

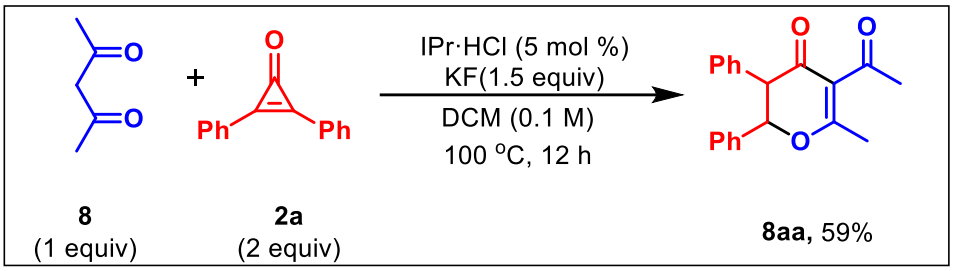


To an oven dried seal tube charged with a stir bar, (3S,4R)-1-(2-hydroxyphenyl)-3,4-diphenylazetidin-2-one **3aa** (0.1 mmol, 1 equiv), IPr.HCl (5 mol%, 0.05 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1mL, 0.1 M) was added under nitrogen atmosphere. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), the crude was filtered and residue was evaporated under reduced pressure. The crude NMR was recorded and the product **5aa-1** was not detected.

(e) Reaction to ascertain the intermediacy Acyl azolium:

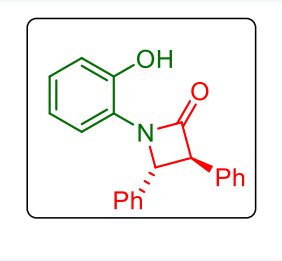


(a) To an oven dried seal tube charged with a stir bar, aniline **6** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), the crude was filtered and residue was evaporated under reduced pressure. The crude NMR was recorded and the product **6aa** was not detected.



(b) To an oven dried seal tube charged with a stir bar, acetyl acetone **8** (0.1 mmol, 1 equiv), diphenylcyclopropenone **2a** (0.2 mmol, 2.00 equiv), IPr.HCl (5 mol%, 0.05 equiv), was added under nitrogen atmosphere and vacuum was applied. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere. Dichloromethane (1 mL, 0.1 M) was added under nitrogen atmosphere and seal tube was sealed. Potassium fluoride (0.15 mmol, 1.5 equiv) was added inside the glove box and the reaction mixture was stirred (700 rpm) in a preheated aluminium block at **100 °C for 12 h**. After completion of the reaction (monitored by TLC), with the addition of the silica gel in EtOAc to the residue, the solvent was evaporated under reduced pressure, and the crude was purified by column chromatography to get a **8aa** (18 mg, 59%) as a product**.**

4. Experimental characterization data for products:



(3S,4R)-1-(2-hydroxyphenyl)-3,4-diphenylazetidin-2-one (3aa): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (24 mg) 75% yield.

Physical State: colorless liquid.

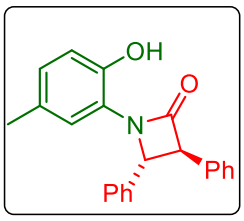
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 7.43 – 7.33 (m, 10H), 7.04 – 7.03 (m, 2H), 6.67-6.62 (m, 1H), 6.51 (d, J = 8.4 Hz, 1H), 5.03 (d, J = 2.4 Hz, 1H), 4.25 (d, J = 2.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 148.2, 136.5, 134.1, 129.8, 129.5, 129.4, 128.6, 127.7, 127.2, 126.2, 125.4, 120.0, 119.5, 118.2, 64.0, 62.1.

IR (KBr, cm^{-1}): 3417, 2961, 1712, 1496, 1146.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{18}NO_2$: 316.1338; Found: 316.1353.



(3S,4R)-1-(2-hydroxy-5-methylphenyl)-3,4-diphenylazetidin-2-one (3ba): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (17 mg) 52% yield.

Physical State: yellow solid.

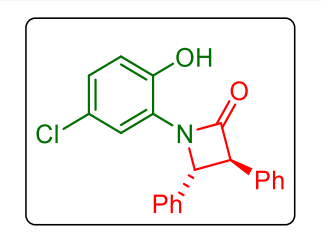
R_f-value: 0.4 (10% EtOAc/hexane), **mp:** 163 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.44 (s, 1H), 7.41 – 7.33 (m, 10 H), 6.93 (d, J = 8.4 Hz, 1H), 6.85 (d, J = 8.4 Hz, 1H), 6.32 (s, 1H), 5.03 (d, J = 1.6 Hz, 1H), 4.24 (d, J = 1.6 Hz, 1H), 2.07 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.5, 146.0, 136.7, 134.2, 129.7, 129.5, 129.3, 128.6 (2C), 127.9, 127.7, 126.2, 125.1, 119.3, 118.6, 63.9, 62.2, 20.7.

IR (KBr, cm^{-1}): 3439, 1710, 1640.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{20}NO_2$: 330.1494; Found: 330.1509.



(3S,4R)-1-(5-chloro-2-hydroxyphenyl)-3,4-diphenylazetidin-2-one (3ca): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (19 mg) 54% yield.

Physical State: brown solid.

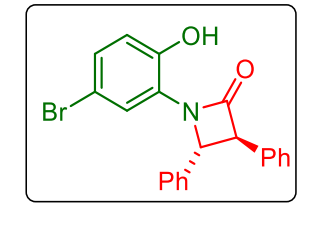
R_f-value: 0.4 (10% EtOAc/hexane), **mp:** 120 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.70 (s, 1H), 7.44-7.36 (m, 8H), 7.33 (d, J = 7.0 Hz, 2H), 6.99 (dd, J = 7.0 Hz, 2.1Hz, 1H), 6.95 (d, J = 9.1 Hz, 1H), 6.47 (d, J = 2.1 Hz, 1H), 5.01 (d, J = 2.1 Hz, 1H), 4.27 (d, J = 2.1 Hz, 1H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.8, 147.0, 135.9, 133.8, 129.9 (2C), 129.6, 128.7, 127.6, 127.0, 126.4, 126.2, 124.8, 120.6, 118.0, 64.1, 62.3.

IR (KBr, cm^{-1}): 3443, 2920, 1712, 1692, 1494.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{17}NO_2Cl$: 350.0948; Found: 350.0963.



(3S,4R)-1-(5-bromo-2-hydroxyphenyl)-3,4-diphenylazetidin-2-one (3da): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (26 mg) 66% yield.

Physical State: Orange solid

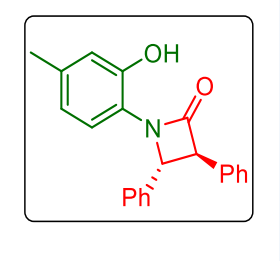
R_f-value: 0.4 (10% EtOAc/hexane), **mp:** 140 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ 9.72 (s, 1H), 7.45-7.36 (m, 8H), 7.33 (d, J = 7.0 Hz, 2H), 7.26 (dd, J = 8.4 Hz, 2.1 Hz,1H), 6.90 (d, J = 9.1 Hz, 1H), 6.61 (d, J = 2.1 Hz, 1H), 5.02 (d, J = 2.1 Hz, 1H), 4.28 (d, J = 2.1 Hz, 1H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.9, 147.6, 135.9, 133.7, 130.0, 129.9, 129.6 (2C), 128.7, 127.6, 126.8, 126.2, 121.1, 120.9, 111.7, 64.1, 62.3.

IR (KBr, cm^{-1}): 3442, 1712, 1632, 1493, 1146.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{21}H_{17}NO_2Br$: 394.0443; Found: 394.0456.



(3S,4R)-1-(2-hydroxy-4-methylphenyl)-3,4-diphenylazetidin-2-one (3ea): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (18 mg) 55% yield.

Physical State: yellow solid.

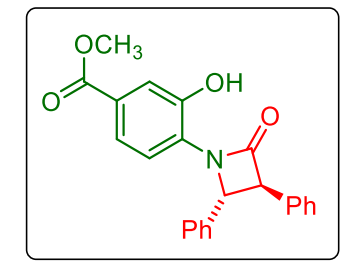
R_f-value: 0.3 (10% EtOAc/hexane), **mp:** 215 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.75 (s, 1H), 7.42 – 7.33 (m, 10 H), 6.85 (s, 1H), 6.45 (d, J = 8.0 Hz, 1H), 6.38 (d, J = 8.0 Hz, 1H), 5.00 (d, J = 1.6 Hz, 1H), 4.23 (d, J = 1.2 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.1, 148.0, 137.5, 136.7, 134.3, 129.7, 129.5, 129.3, 128.6, 127.7, 126.2, 123.0, 120.7, 119.9, 64.0, 62.1, 21.1.

IR (KBr, cm^{-1}): 3450, 1703, 1657.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{20}NO_2$: 330.1494; Found: 330.1524.



(Methyl 3-hydroxy-4-((3S,4R)-2-oxo-3,4-diphenylazetidin-1-yl benzoate) (3fa): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (14 mg) 38% yield.

Physical State: colorless liquid

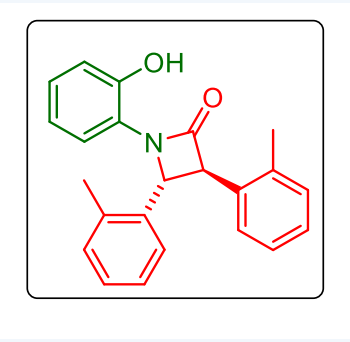
R_f-value: 0.3 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 700 MHz): δ 9.90 (s, 1H), 7.68 (d, J = 1.6 Hz, 1H), 7.42 – 7.32 (m, 11H), 6.52 (d, J = 8.0 Hz, 1H), 5.07 (d, J = 2.4 Hz, 1H), 4.30 (d, J = 2.4 Hz, 1H), 3.85 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.0, 166.6, 148.0, 136.1, 133.7, 129.9 (2C), 129.6, 129.2, 128.8 (2C), 127.6, 126.2, 121.7, 120.7, 117.9, 64.2, 62.2, 52.4.

IR (KBr, cm^{-1}): 3448, 2923, 1711, 1653, 1456, 1223.

HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{23}H_{19}NO_4Na$: 396.1212; Found: 396.1190.



(3S,4R)-1-(2-hydroxyphenyl)-3,4-di-o-tolylazetidin-2-one (3ac): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (19 mg) 55% yield.

Physical State: yellow solid

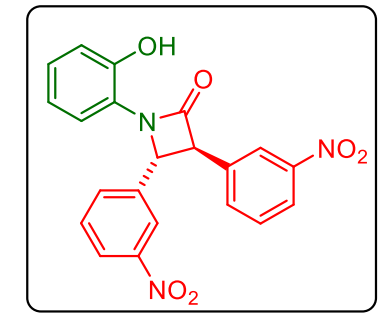
R_f-value: 0.3 (10% EtOAc/hexane), **mp:** 141 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ. 9.84 (s, 1H), 7.41 – 7.39 (m, 1H), 7.30 (d, J = 7.7 Hz, 2H), 7.25 – 7.23 (m, 2H), 7.20 (d, J = 7.7 Hz, 3H), 7.04 – 7.01 (m, 2H), 6.64 – 6.62 (m, 1H), 6.49 (d, J = 7.7 Hz, 1H), 4.87 (d, J = 2.8 Hz, 1H), 4.44 (d, J = 2.1 Hz, 1H), 2.35 (s, 3H), 2.12 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.0, 148.4, 139.4, 137.0, 133.8, 133.1, 131.0, 130.4, 128.5, 127.2, 127.0 (2C), 126.9, 126.4, 125.5, 120.0, 119.4 (2C), 118.2, 63.9, 60.1, 21.5, 20.2.

IR (KBr, cm^{-1}): 3443, 1634.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1666.



(3S,4R)-1-(2-hydroxyphenyl)-3,4-bis(3-nitrophenyl)azetidin-2-one (3af): was prepared according to general procedure (2c). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (20 mg) 50% yield.

Physical State: yellow solid

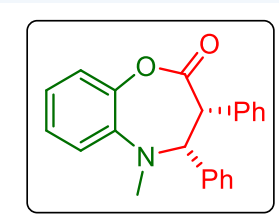
R_f-value: 0.6 (30% EtOAc/hexane), **mp:** 90 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ. 9.18 (s, 1H), 8.28 – 8.26 (m, 3H), 8.22 (t, J = 2.1 Hz, 1H), 7.76 – 7.72 (m, 2H), 7.67-7.64 (m, 2H), 7.11 (dt, J = 8.4 Hz, 1.4 Hz, 1H), 7.07 (dd, J = 8.4 Hz, 1.4 Hz, 1H), 6.72-6.70 (m, 1H), 6.51 (dd, J = 7.7 Hz, 1.4 Hz, 1H), 5.23 (d, J = 2.1 Hz, 1H), 4.41 (d, J = 2.8 Hz, 1H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 165.3, 149.3, 148.3, 138.2, 135.2, 133.7, 131.8, 131.4, 130.9, 128.1 (2C), 124.8, 124.5, 124.1, 122.9, 121.8, 120.5, 120.0, 118.1, 62.6, 61.5.

IR (KBr, cm^{-1}): 3087, 1717.

HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{15}N_3O_6Na$: 428.0858; Found: 428.0878.



(3R,4R)-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ea'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (13 mg) 40% yield.

Physical State: brown solid

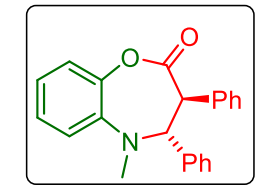
R_f-value: 0.6 (10% EtOAc/hexane), **mp:** 202 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34 – 7.26 (m, 4H), 7.25 – 7.16 (m, 3H), 7.14 – 7.06 (m, 5H), 6.85 (d, J = 8.0 Hz, 2H), 4.49 (d, J = 6.0 Hz, 1H), 4.20 (d, J = 6.0 Hz, 1H), 2.62 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.4, 146.5, 141.2, 137.3, 133.7, 131.0, 129.3, 128.9, 128.3, 127.9, 127.8, 126.9, 123.6, 120.4, 120.3, 78.8, 50.7, 39.1.

IR (KBr, cm^{-1}): 3447, 2962, 1764, 1494, 1127

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{20}NO_2$: 330.1494; Found: 330.1483.



(3S,4R)-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ea): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (15 mg) 46% yield.

Physical State: colorless liquid

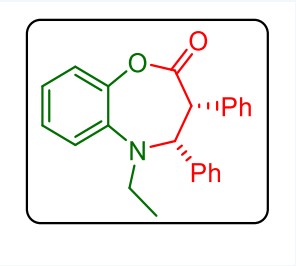
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.22-7.11 (m, 11H), 6.90 (d, J = 8.0 Hz, 1H), 6.80 (d, J = 7.2 Hz, 2H), 4.80 (d, J = 11.6 Hz, 1H), 4.26 (d, J = 11.6 Hz, 1H), 2.60 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.3, 147.0, 138.1, 136.4, 134.5, 130.1, 128.5. 128.4 (2C), 127.9,127.8, 126.1, 124.6, 123.2, 119.8, 77.1, 54.1, 39.2.

IR (KBr, cm^{-1}): 3438, 1762, 1492, 1133.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{20}NO_2$: 330.1494; Found: 330.1505.



(3R,4R)-5-ethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one) (5fa'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (13 mg) 38% yield.

Physical State: yellow solid

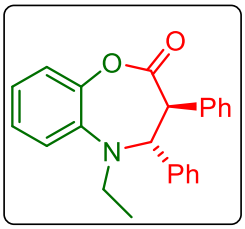
R_f-value: 0.5 (10% EtOAc/hexane), **mp:** 132 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.35-7.38 (m, 3H), 7.20-7.06 (m, 9H), 6.85 (d, J = 7.6 Hz, 2H), 4.47 (d, J = 5.6 Hz, 1H), 4.38 (d, J = 6.0 Hz, 1H), 3.20-3.11 (m, 1H), 2.98-2.91 (m, 1H), 0.97 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.6, 147.5, 138.5, 137.6, 133.8, 131.1, 129.4, 128.8, 128.3, 127.9, 127.7, 126.6, 123.5, 121.2, 120.7, 77.5, 50.6, 43.4, 12.7.

IR (KBr, cm^{-1}): 3447, 2923, 1766, 1494, 1124.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1635.



(3S,4R)-5-ethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5fa): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (17 mg) 50% yield.

Physical State: colorless liquid

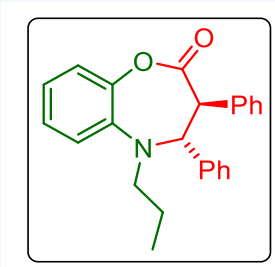
R_f-value: 0.6 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.25-7.24 (m, 1H), 7.22-7.19 (m, 4H), 7.16-7.09 (m, 6H), 6.89-6.87 (m, 1H), 6.80 (d, J = 7.0 Hz, 2H), 4.91 (d, J = 11.9 Hz, 1H), 4.25 (d, J = 11.2 Hz, 1H), 2.95-2.88 (m, 1H), 2.87-2.82 (m, 1H), 1.11 (t, J = 7.0 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.5, 148.0 136.8, 136.5, 134.6, 130.1, 128.4 (2C), 128.3, 127.9 (2C), 125.8, 124.8, 124.6, 119.9, 75.2, 54.1, 45.1, 13.1.

IR (KBr, cm^{-1}): 3439, 1765, 1491, 1158.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1673.



(3S,4R)-3,4-diphenyl-5-propyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ga): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (15 mg) 42% yield.

Physical State: yellow liquid

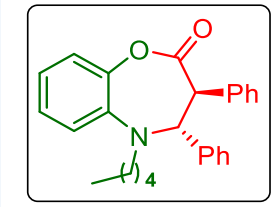
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.24 (d, J = 2.1 Hz, 1H), 7.21-7.18 (m, 4H), 7.16 -7.10 (m, 6H), 6.90 (d, J = 7.7 Hz, 1H), 6.80 (d, J = 7.0 Hz, 2H) 4.86 (d, J = 11.2 Hz, 1H), 4.26 (d, J = 11.2 Hz, 1H), 2.88-2.84 (m, 1H), 2.77-2.73(m, 1H), 1.54-1.48 (m, 2H), 0.87 (t, J = 7.0 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.5, 148.0,136.8, 136.6, 134.6, 130.1, 128.4 (2C), 128.3, 127.9 (2C), 125.8, 124.8, 124.6, 119.9, 75.4, 54.1, 52.5, 20.5, 11.7.

IR (KBr, cm^{-1}): 3447, 2961, 1767, 1492, 1123.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{24}NO_2$: 358.1807; Found: 358.1801.



(3S,4R)-5-pentyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ha): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (28 mg) 73% yield.

Physical State: brown liquid

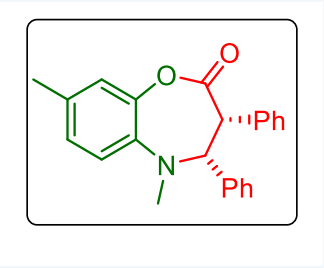
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.24-7.23 (m, 1H), 7.21 – 7.09 (m, 10H), 6.91-6.88 (m, 1H), 6.80 (d, J = 6.4 Hz, 2H), 4.86 (d, J = 11.6 Hz, 1H), 4.25 (d, J = 11.6 Hz, 1H), 2.90-2.83 (m, 1H), 2.80-2.73 (m, 1H), 1.52-1.46 (m, 2H), 1.27-1.25 (m, 4H), 0.85 (t, J = 6.8 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.4, 148.0, 136.9, 136.5, 134.6, 130.1, 128.4 (2C), 128.3, 127.9 (2C), 125.8, 124.8, 124.6, 119.9, 75.4, 54.1, 50.7, 29.3, 26.9, 22.6, 14.3.

IR (KBr, cm^{-1}): 3437, 2953, 1765, 1491, 1126.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{26}H_{28}NO_2$: 386.2120; Found: 386.2092.



(3R,4R)-5,8-dimethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ia'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (26 mg) 76% yield.

Physical State: white solid

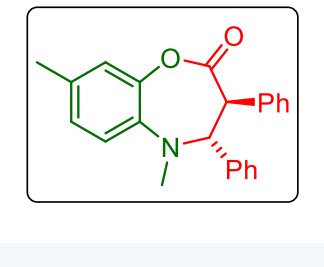
R_f-value: 0.4 (10% EtOAc/hexane), **mp:** 160 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.32 (t, J = 7.0 Hz, 1H), 7.26-7.24 (m, 2H), 7.14 (t, J = 7.0 Hz, 1H), 7.11-7.05 (m, 6H), 6.99 (s, 1H), 6.84 (d, J = 7.7 Hz, 2H), 4.46 (d, J = 6.3 Hz, 1H), 4.15 (d, J = 6.3 Hz, 1H), 2.58 (s, 3H), 2.37 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.7, 146.3, 138.6, 137.4, 133.8 (2C), 131.0, 129.3, 128.8, 128.3, 127.8, 127.7, 127.2, 120.7, 120.1, 79.1, 50.8, 39.2, 20.9.

IR (KBr, cm^{-1}): 3448, 2960, 1764, 1509, 1124.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1639.



(3S,4R)-5,8-dimethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ia): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (4 mg) 12% yield.

Physical State: orange liquid.

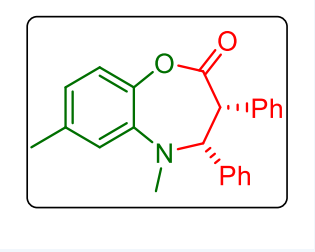
R_f-value: 0.3 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.20 (d, J = 7.7 Hz, 2H), 7.16-7.10 (m, 6H), 7.04 (s, 1H), 7.01 (d, J = 7.7 Hz, 1H), 6.81 (d, J = 7.7 Hz, 2H), 6.76 (d, J = 7.7 Hz, 1H), 4.76 (d, J =11.2 Hz, 1H), 4.25 (d, J = 11.2 Hz, 1H), 2.57 (s, 3H), 2.40 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.6, 146.7, 136.4, 135.3, 134.7 (2C), 130.1, 128.4 (3C), 127.9 (2C), 126.6, 122.9, 120.2, 77.2, 54.2, 39.2, 21.1.

IR (KBr, cm^{-1}): 3438, 1761, 1633.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1662.



(3R,4R)-5,7-dimethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ja'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (21 mg) 61% yield.

Physical State: white solid

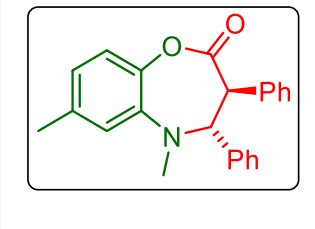
R_f-value: 0.5 (10% EtOAc/hexane), **mp:** 208 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.32 (t, J = 7.0 Hz, 1H), 7.27-7.24 (m, 2H), 7.15 (t, J = 7.7 Hz, 1H), 7.09-7.05 (m, 5H), 7.01 (s, 1H), 6.90 (d, J = 7.7 Hz, 1H), 6.85 (d, J = 7.7 Hz, 2H), 4.49 (d, J = 5.6 Hz, 1H), 4.17 (d, J = 5.6 Hz, 1H), 2.60 (s, 3H), 2.41 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.7, 144.2, 140.8, 137.4, 136.6, 133.8, 131.0, 129.3, 128.9, 128.3, 127.9, 127.7, 123.9, 120.9, 120.0, 78.8, 50.6, 39.1, 21.6.

IR (KBr, cm^{-1}): 3438, 2959, 1764, 1502, 1127.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1656.



(3S,4R)-5,7-dimethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ja): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (7 mg) 20% yield.

Physical State: colorless liquid

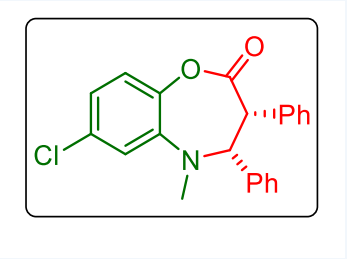
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.18 (d, J = 7.7 Hz, 2H), 7.16-7.13 (m, 7H), 6.97 (d, J = 8.4 Hz, 1H), 6.81 (d, J = 7.0 Hz, 2H), 6.67 (s, 1H), 4.78 (d, J = 11.9 Hz, 1H), 4.26 (d, J = 11.2 Hz, 1H), 2.58 (s, 3H), 2.35 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.6, 144.7, 137.8, 136.6, 135.9, 134.6, 130.1, 128.4 (2C), 128.3, 127.9, 127.8, 125.0, 123.6, 119.4, 77.0, 54.1, 39.2, 21.5, 6.3.

IR (KBr, cm^{-1}): 3443, 2923, 1763, 1498, 1132.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{23}H_{22}NO_2$: 344.1651; Found: 344.1649.



(3R,4R)-7-chloro-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ka'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (8 mg) 22% yield.

Physical State: orange liquid

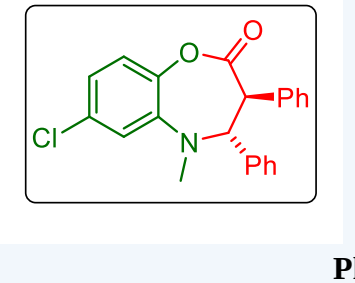
R_f-value: 0.6 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.33 (m, 1H), 7.28 (d, J = 8.0 Hz, 1H), 7.18-7.16 (m, 2H), 7.12-7.06 (m, 7H), 6.87-6.85 (m, 2H), 4.47 (d, J = 5.6 Hz, 1H), 4.21 (d, J = 6.0 Hz, 1H), 2.61 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.8, 144.8, 142.2, 136.8, 133.3, 132.0, 131.0, 129.3, 129.1, 128.4, 128.1, 127.9, 123.1, 121.4, 120.6, 78.4, 50.5, 39.1.

IR (KBr, cm^{-1}): 3438, 1770, 1633, 1491, 1122.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{19}NO_2Cl$: 364.1104; Found: 364.1114.



(3S,4R)-7-chloro-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ka): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (10 mg) 27% yield.

Physical State: colorless liquid

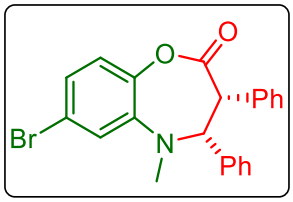
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.19-7.13 (m, 10H), 6.85 (d, J = 2.1 Hz, 1H), 6.81 (d, J = 6.3 Hz, 2H), 4.79 (d, J = 11.9 Hz, 1H), 4.24 (d, J=11.9Hz, 1H), 2.58 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 169.7, 145.5, 139.4, 136.1, 134.1, 131.3, 130.0, 128.7 (2C), 128.5, 128.1, 127.6, 124.4, 123.2, 120.8, 77.0, 53.9, 39.3.

IR (KBr, cm^{-1}): 3438, 2922, 1767, 1635, 1488, 1128.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{19}NO_2Cl$: 364.1104; Found: 364.1120.



(3R,4R)-7-bromo-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5la'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (14 mg) 34% yield.

Physical State: white solid.

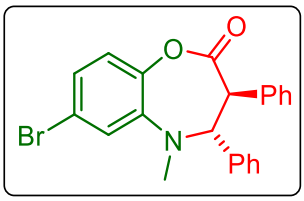
R_f-value: 0.6 (10% EtOAc/hexane), **mp:** 235 ℃

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.31 (m, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.24 – 7.21 (m, 1H), 7.19-7.16 (m, 1H), 7.11-7.04 (m, 5H), 6.87-6.84 (m, 2H), 4.47 (d, J = 5.6 Hz, 1H), 4.21 (d, J = 5.6 Hz, 1H), 2.61 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 167.7, 145.4, 142.5, 136.8, 133.3, 131.0, 129.3, 129.1, 128.4, 128.1, 127.9, 126.2, 123.5, 121.8, 119.6, 78.4, 50.5, 39.1.

IR (KBr, cm^{-1}): 3439, 1767, 1633, 1489, 1122.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{19}NO_2Br$: 408.0599; Found: 408.0616.



(3S,4R)-7-bromo-5-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5la): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (7 mg) 17% yield.

Physical State: orange liquid.

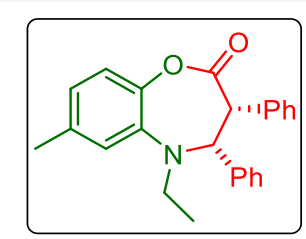
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.30 (d, J = 8.4 Hz, 1H), 7.18-7.13 (m, 8H), 7.10 (d, J = 8.4 Hz, 1H), 6.99 (s, 1H), 6.81 (d, J = 7.0 Hz, 2H), 4.79 (d, J = 11.2 Hz, 1H), 4.23 (d, J = 11.2 Hz, 1H), 2.58 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 169.7, 146.0, 139.6, 136.0, 134.1, 130.0, 128.7 (2C), 128.5, 128.1, 127.6, 127.4, 126.1, 121.2, 118.8, 77.0, 54.0, 39.3.

IR (KBr, cm^{-1}): 3438, 1766, 1633, 1485, 1129.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{22}H_{19}NO_2Br$: 408.0599; Found: 408.0576.



(3R,4R)-5-ethyl-7-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ma'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (20 mg) 56% yield.

Physical State: yellow solid

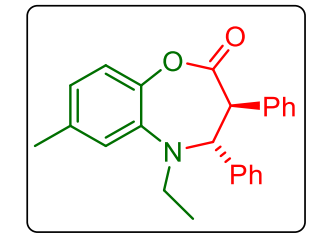
R_f-value: 0.5 (10% EtOAc/hexane), **mp:** 172 °C

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34-7.30 (m, 1H), 7.26-7.23 (m, 2H), 7.17-7.04 (m, 7H), 7.00 (d, J = 2.0 Hz, 1H), 6.85-6.83 (m, 2H), 4.43 (d, J = 6.0 Hz, 1H), 4.33 (d, J = 6.0 Hz, 1H), 3.15-3.06 (m, 1H), 2.93-2.84 (m, 1H), 2.37 (s, 3H), 0.95 (t, J = 6.8 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.8, 147.4, 137.7, 135.8, 133.9, 133.7, 131.0, 129.4, 128.7, 128.2, 127.8, 127.7, 127.0, 121.1, 121.0, 77.7, 50.7, 43.3, 20.9, 12.8.

IR (KBr, cm^{-1}): 3438, 2971, 1765, 1501, 1124.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{24}NO_2$: 358.1807; Found: 358.1803.



(3S,4R)-5-ethyl-7-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ma): was prepared according to general procedure (2.2). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (4 mg) 11% yield.

Physical State: colorless liquid

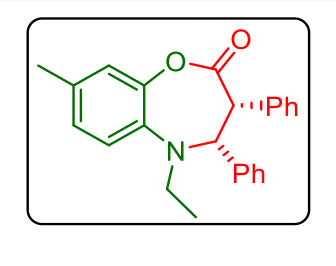
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.21-7.05 (m, 9H), 7.02-6.99 (m, 1H), 6.81 (dd, J = 8.0 Hz, 1.6 HZ, 2H), 6.75 (d, J = 8.0 Hz, 1H), 4.87 (d, J = 11.6 Hz, 1H), 4.24 (d, J = 11.6 Hz, 1H), 2.91-2.77 (m, 2H), 2.41 (s, 3H), 1.08 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.8, 147.7, 136.5, 134.9, 134.8, 133.8, 131.0, 130.1, 128.4 (2C), 128.3, 127.9 (2C), 126.4, 124.1, 120.3, 75.3, 54.2, 45.0, 21.2, 13.1.

IR (KBr, cm^{-1}): 3439, 1761, 1634, 1498, 1128.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{24}NO_2$: 358.1807; Found: 358.1808.



(3R,4R)-5-ethyl-8-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5na'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (16 mg) 45% yield.

Physical State: orange liquid.

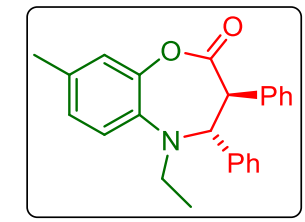
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ. 7.34-7.30 (m, 1H), 7.27-7.23 (m, 2H), 7.18-7.13 (m, 1H), 7.09-7.02 (m, 6H), 6.89 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 6.86-6.84 (m, 2H), 4.47 (d, J = 5.6 Hz, 1H), 4.36 (d, J = 5.6 Hz, 1H), 3.18-3.09 (m, 1H), 2.96-2.87 (m, 1H), 2.42 (s, 3H), 0.97 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.9, 145.2, 138.1, 137.7, 136.3, 133.9, 131.1, 129.4, 128.8, 128.2, 127.8, 128.7, 123.9, 121.7, 120.3, 77.3, 50.5, 43.3, 21.7, 12.7.

IR (KBr, cm^{-1}): 3450, 2972, 1764, 1508, 1123.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{24}NO_2$: 358.1807; Found: 358.1818.



(3S,4R)-5-ethyl-8-methyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5na): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (4 mg) 11% yield.

Physical State: orange liquid.

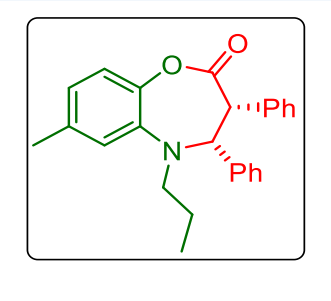
R_f-value: 0.3 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.18-7.11 (m, 9H), 6.99-6.97 (m, 1H), 6.82-6.80 (m, 2H), 6.68 (d, J = 1.6 Hz, 1H), 4.89 (d, J = 11.6 Hz, 1H), 4.25 (d, J = 11.6 Hz, 1H), 2.92-2.77 (m, 2H), 2.35 (s, 3H), 1.11 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.8, 145.6, 136.6, 136.4, 135.6, 134.7, 130.1, 128.4 (2C), 128.3, 127.9(2C), 125.2, 124.8, 119.5, 75.0, 54.0, 45.0, 21.5, 13.1.

IR (KBr, cm^{-1}): 3451, 2923, 1763, 1506, 1126.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{24}H_{24}NO_2$: 358.1807; Found: 358.1818.



(3R,4R)-7-methyl-3,4-diphenyl-5-propyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5oa'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (9 mg) 24% yield.

Physical State: yellow liquid

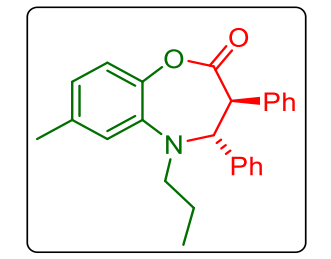
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.34-7.30 (m, 1H), 7.27-7.23 (m, 1H), 7.15 (t, J = 7.6 Hz, 1H), 7.09-7.04 (m, 6H), 7.00 (s, 1H), 6.90 (d, J = 8.4 Hz, 1H), 6.83 (d, J = 7.6 Hz, 2H), 4.45 (d, J = 5.6 Hz, 1H), 4.30 (d, J = 5.6 Hz, 1H), 3.11-3.05 (m, 1H), 2.75-2.68 (m, 1H), 2.42 (s, 3H), 1.44-1.39 (m, 2H), 0.71 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 169.0, 145.3, 138.1, 137.7, 136.2, 134.0, 131.1, 129.6, 128.8, 128.2, 127.8, 127.7, 124.0, 121.9, 120.3, 77.7, 50.6, 50.5, 21.7, 20.2, 11.7.

IR (KBr, cm^{-1}): 3442, 2961, 1766, 1499, 1123.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{26}NO_2$: 372.1964; Found: 372.1951.



(3S,4R)-7-methyl-3,4-diphenyl-5-propyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5oa): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (17 mg) 46% yield.

Physical State: yellow liquid

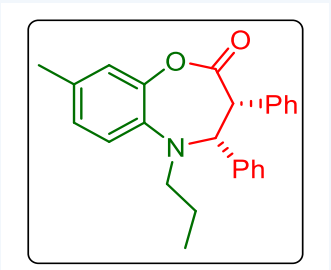
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.20-7.18 (m, 2H), 7.15-7.11 (m, 7H), 6.98 (d, J = 8.4 Hz, 1H), 6.82 (d, J = 6.8 Hz, 2H), 6.69 (s, 1H), 4.84 (d, J = 11.6 Hz, 1H), 4.25 (d, J = 11.6 Hz, 1H), 2.88-2.81 (m, 1H), 2.75-2.69 (m, 1H), 2.34 (s, 3H), 1.53-1.48 (m, 2H), 0.87 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.7, 145.7, 136.7, 136.5, 135.6, 134.7, 130.1, 128.4, 128.2 (2C), 127.9 (2C), 125.2, 125.0, 119.5, 75.1, 54.0, 52.4, 21.5, 20.6, 11.7.

IR (KBr, cm^{-1}): 3443, 2960, 1763, 1498, 1129.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{26}NO_2$: 372.1964; Found: 372.1951.



(3R,4R)-8-methyl-3,4-diphenyl-5-propyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5pa'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (10 mg) 27% yield.

Physical State: yellow liquid

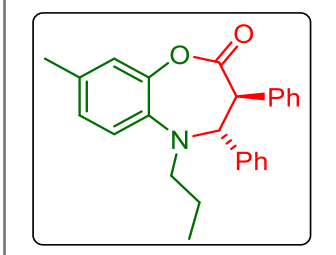
R_f-value: 0.5 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32 (t, J = 7.2 Hz, 1H), 7.25-7.22 (m, 2H), 7.14-7.03 (m, 7H), 7.01 (s, 1H), 6.82 (d, J = 8.0 Hz, 2H), 4.42 (d, J = 6.0 Hz, 1H) , 4.28 (d, J = 6.0 Hz, 1H), 3.09-3.03 (m, 1H), 2.74-2.66 (m, 1H), 2.37 (s, 3H), 1.42-1.37 (m, 2H), 0.70 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 169.0, 1475, 137.7, 135.8, 133.9 (2C), 131.0, 129.6, 128.7, 128.1, 127.8, 127.7, 127.0, 121.2, 121.1, 78.1, 50.7, 50.5, 21.0, 20.3, 11.6.

IR (KBr, cm^{-1}): 3451, 2962, 1765, 1507, 1122.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{26}NO_2$: 372.1964; Found: 372.1977.



(3S,4R)-8-methyl-3,4-diphenyl-5-propyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5pa): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (20 mg) 54% yield.

Physical State: orange liquid

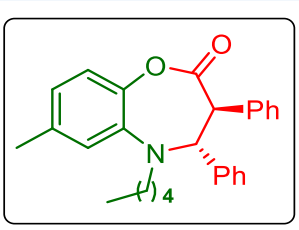
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.21-7.10 (m, 8H), 7.05 (s, 1H), 7.00 (d, J = 8.0 Hz, 1H), 6.81 (d, J = 6.0 Hz, 2H), 6.77 (d, J = 8.4 Hz, 1H), 4.83 (d, J = 11.6 Hz, 1H), 4.25 (d, J = 11.6 Hz, 1H), 2.85-2.79 (m, 1H), 2.75-2.68 (m, 1H), 2.41 (s, 3H), 1.52-1.47 (m, 2H), 0.86 (t, J = 7.2 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.7, 147.8, 136.6, 134.9, 134.8, 133.9, 130.1, 128.4 (2C), 128.2, 127.9 (2C), 126.4, 124.3, 120.3, 75.5, 54.2, 52.4, 21.2, 20.5, 11.7.

IR (KBr, cm^{-1}): 3451, 2961, 1765, 1505, 1127.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{25}H_{26}NO_2$: 372.1964; Found: 372.1949.



(3S,4R)-7-methyl-5-pentyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5qa): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (16 mg) 40% yield.

Physical State: yellow liquid

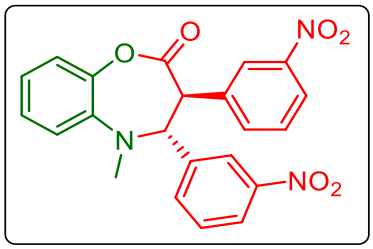
R_f-value: 0.4 (10% EtOAc/hexane)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.20-7.11 (m, 9H), 6.98 (d, J = 8.4 Hz, 1H), 6.81 (d, J = 7.6 Hz, 2H), 6.69 (s, 1H), 4.84 (d, J = 11.6 Hz, 1H), 4.25 (d, J = 11.6 Hz, 1H), 2.89-2.82 (m, 1H), 2.77-2.70 (m, 1H), 2.34 (s, 3H), 1.52-1.49 (m, 2H), 1.27 (m, 4H), 0.85 (t, J = 6.8 Hz, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 170.7, 145.7, 136.7, 135.6, 134.8, 130.1, 128.4 (2C), 128.2, 127.9 (2C), 125.2, 124.9, 119.5, 75.1, 54.0, 50.6, 29.3, 26.9, 22.6, 21.5, 14.3.

IR (KBr, cm^{-1}): 3444, 2954, 1764, 1498, 1128.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{27}H_{30}NO_2$: 400.2277; Found: 400.2287.



(3S,4R)-5-methyl-3,4-bis(3-nitrophenyl)-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5ef+5ef'): was prepared according to general procedure (2d). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (22 mg) 52% yield.

Physical State: yellow solid

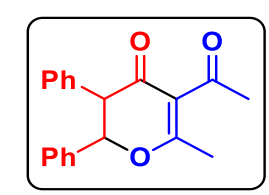
R_f-value: 0.5 (30% EtOAc/hexane); **mp:** 160 °C

^{1}H NMR ($CDCl_3$, 700 MHz): δ 8.09-8.05 (m, 3H), 7.73-7.72 (m, 1H), 7.62 (d, J = 7.7 Hz, 1H), 7.41 (t, J = 8.4 Hz, 1H), 7.38 (t, J = 7.7 Hz, 1H), 7.31-7.28 (m, 3H), 7.16 (d, J = 7.7 Hz, 1H), 6.93-6.92 (m, 1H), 4.93 (d, J = 11.9 Hz, 1H), 4.39 (d, J = 11.9 Hz, 1H), 2.66 (s, 3H).

^{13}C NMR ($CDCl_3$, 175 MHz): δ 168.4, 148.5, 148.4, 146.6, 138.0, 137.0, 136.2, 135.9, 133.5, 130.1, 129.8, 129.1, 126.9, 125.8, 125.1, 124.0, 123.6, 123.2, 122.3, 120.3, 77.9, 76.0, 53.6, 49.9, 39.4, 34.3.

IR (KBr, cm^{-1}): 2937, 1760, 1526, 1348.

HRMS (ESI) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{17}N_3O_6Na$: 442.1015; Found: 442.1014.



5-acetyl-6-methyl-2,3-diphenyl-2,3-dihydro-4H-pyran-4-one (8aa): was prepared according to general procedure (3eb). The crude reaction mixture was purified by column chromatography using silica gel (100-200 mesh size) giving (18 mg) 59% yield.

Physical State: white solid

R_f-value: 0.5 (10% EtOAc/hexane); **mp:** 118 °C

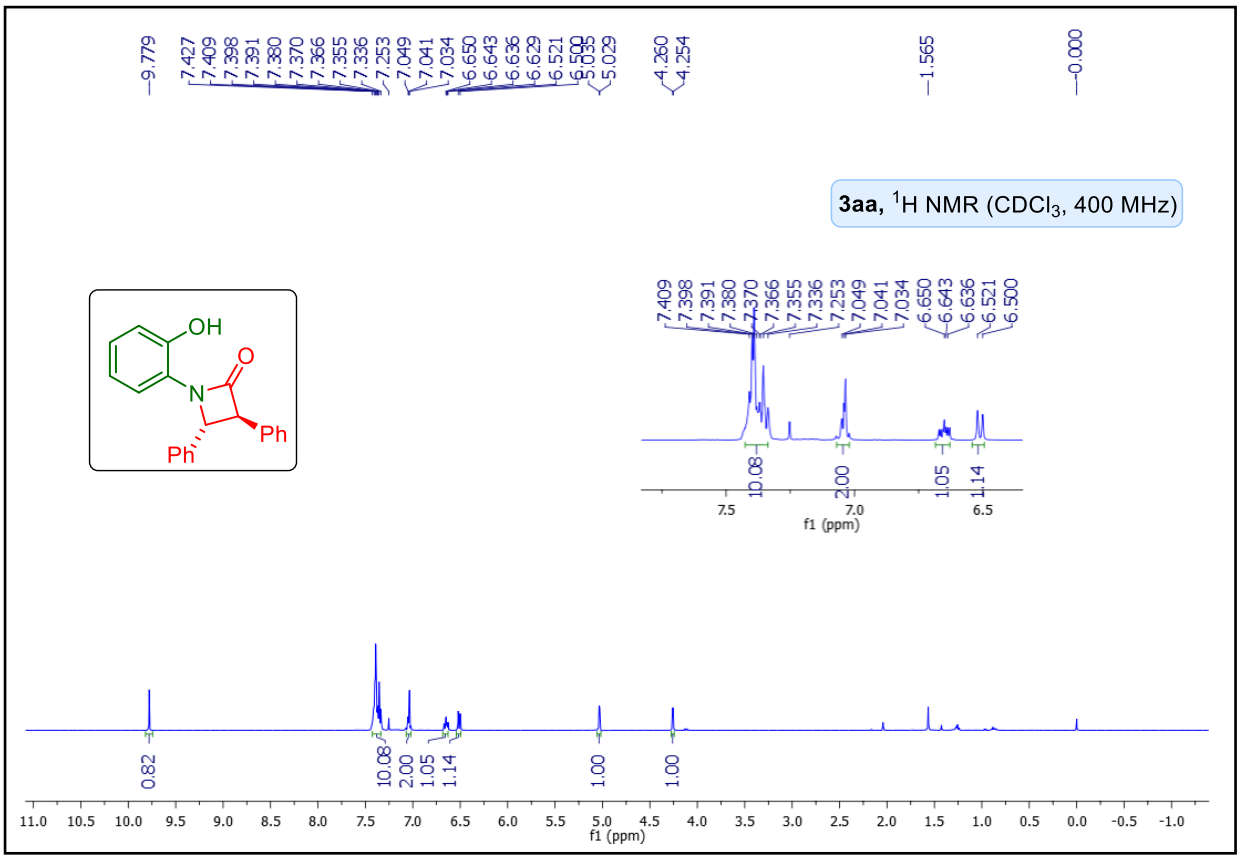
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.36 (m, 5H), 7.26-7.22 (m, 5H), 3.03 (d, J = 15.2 Hz, 1H), 2.81 (d, J = 14.8 Hz, 1H), 2.23 (s, 3H), 1.63 (s, 3H).

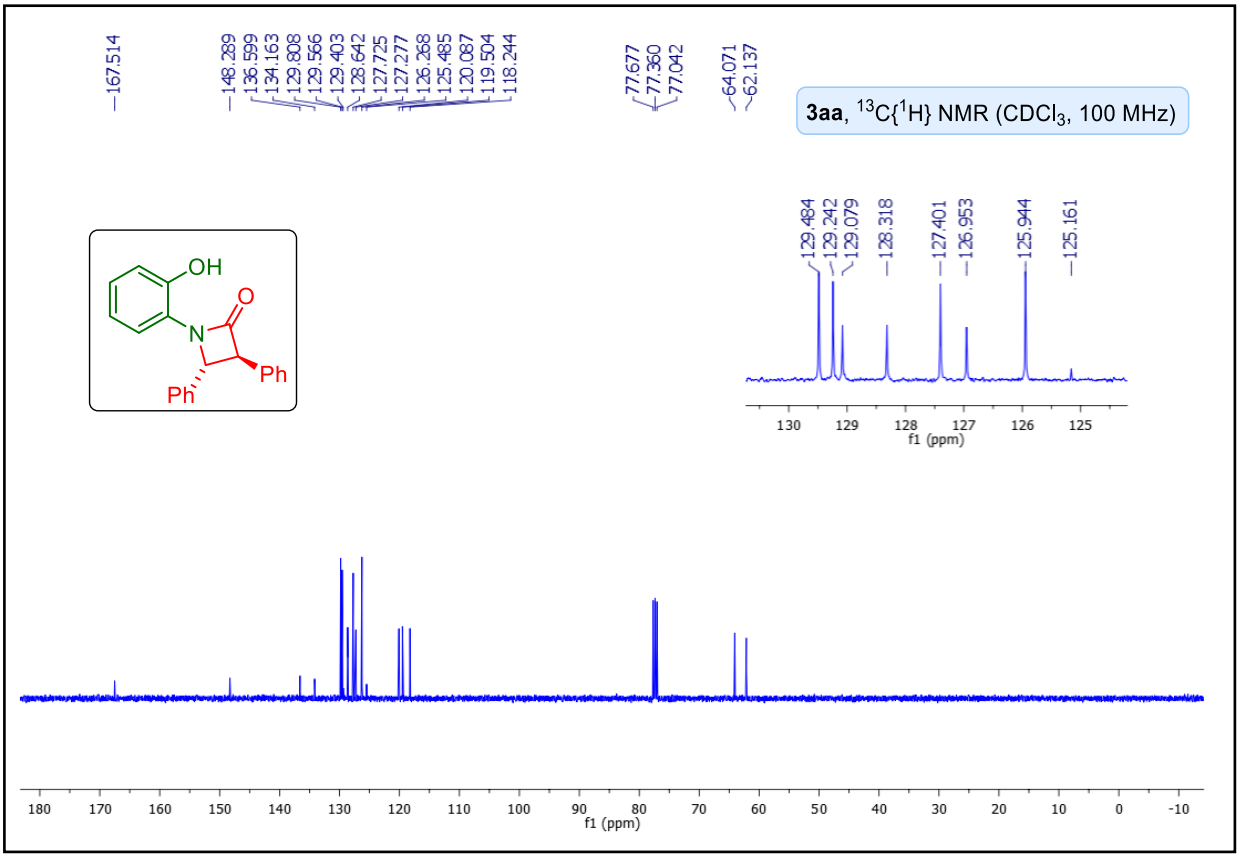
^{13}C NMR ($CDCl_3$, 100 MHz): δ 204.8, 171.3, 164.4, 132.4, 129.8, 129.6, 129.5, 129.3, 128.8, 128.5, 128.4, 127.7, 85.7, 49.2, 32.4, 25.1.

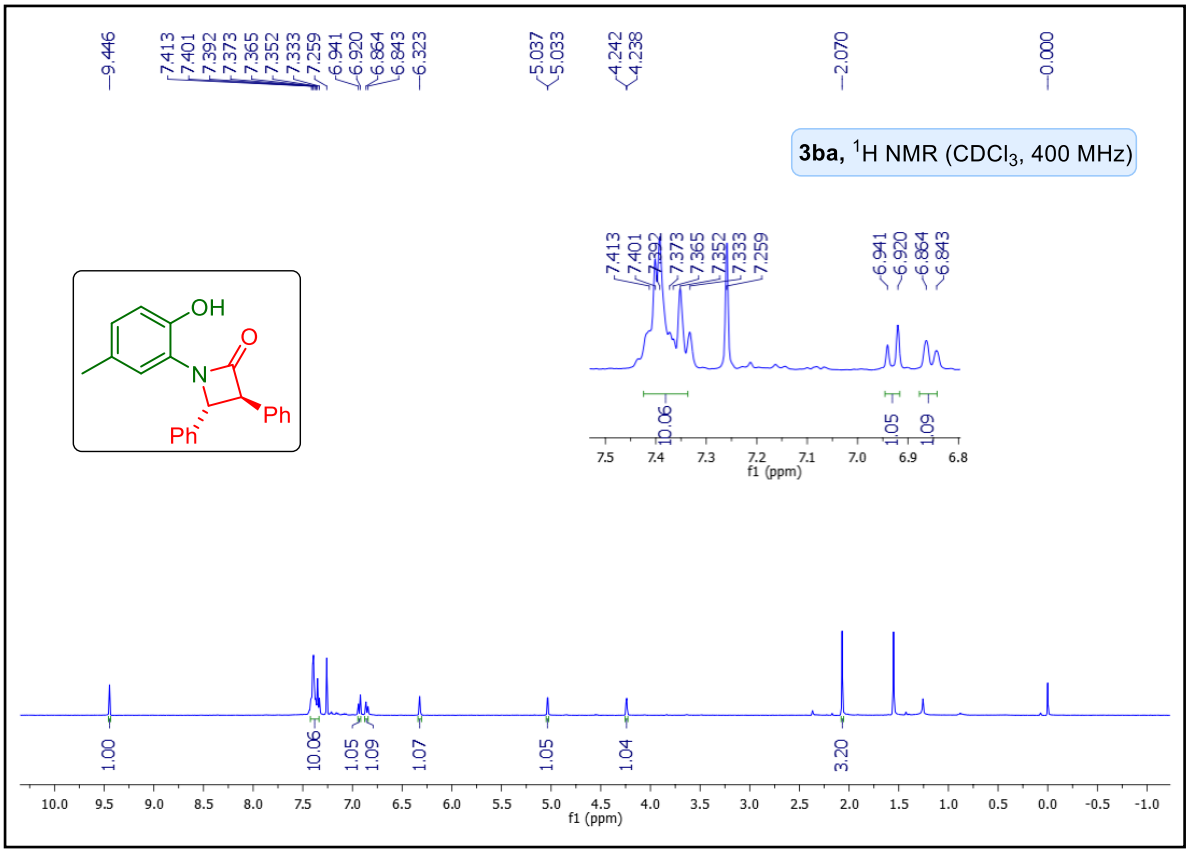
IR (KBr, cm^{-1}): 2918, 1742.

HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{20}H_{19}O_3$: 307.1334; Found: 307.1315.

5. Copies of ^{1}H NMR and ^{13}C NMR spectra:



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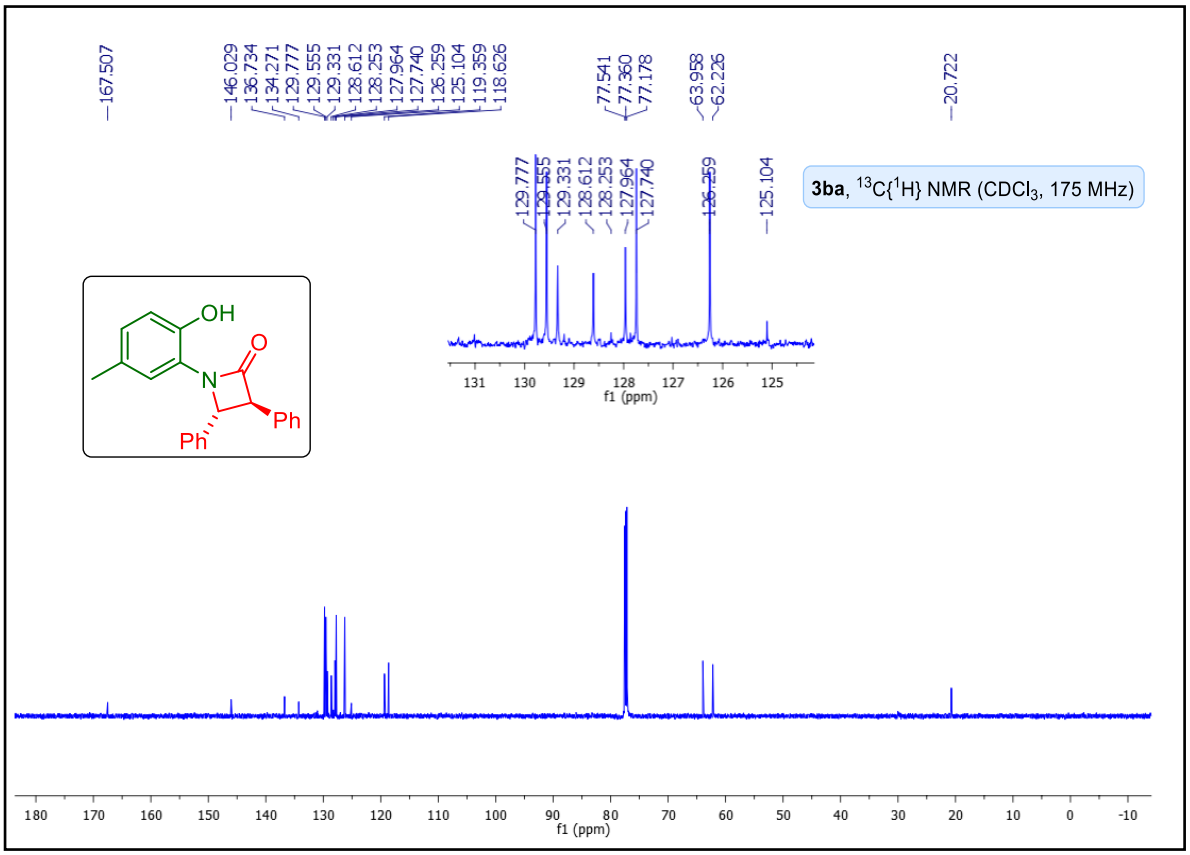
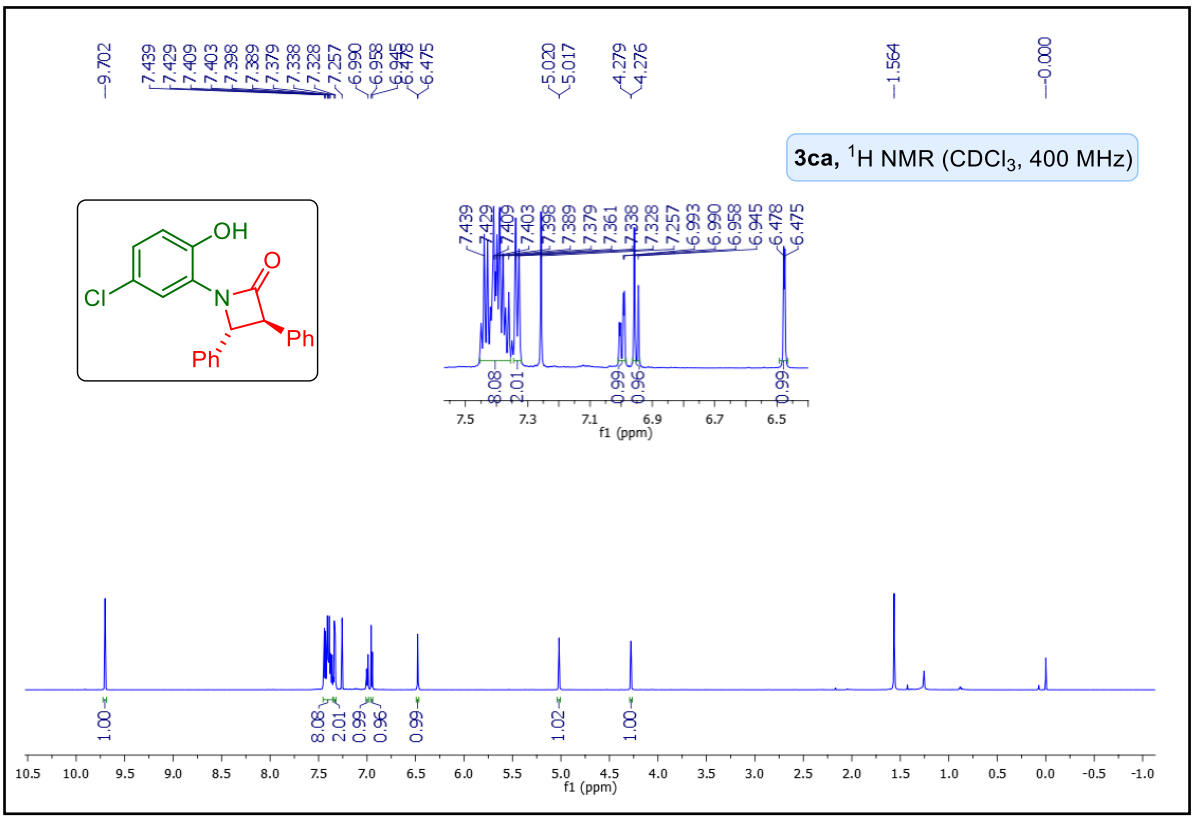
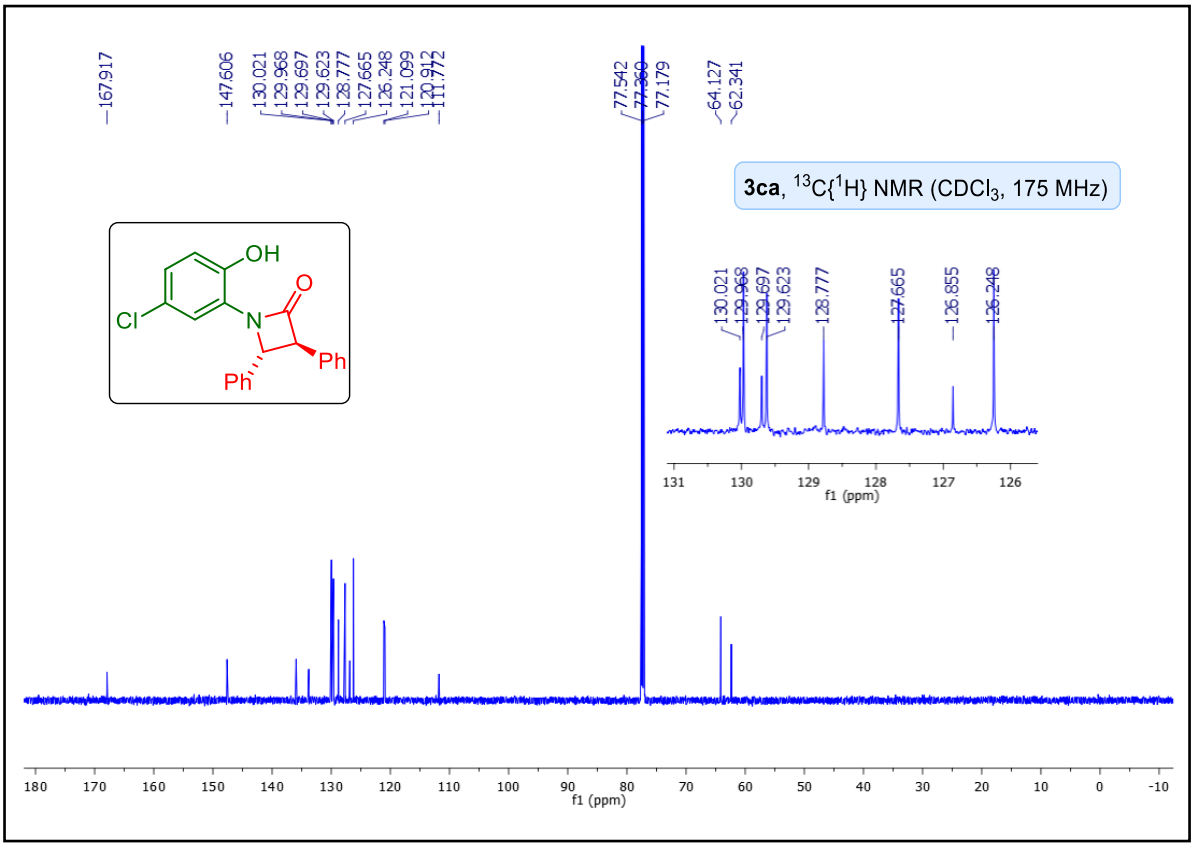
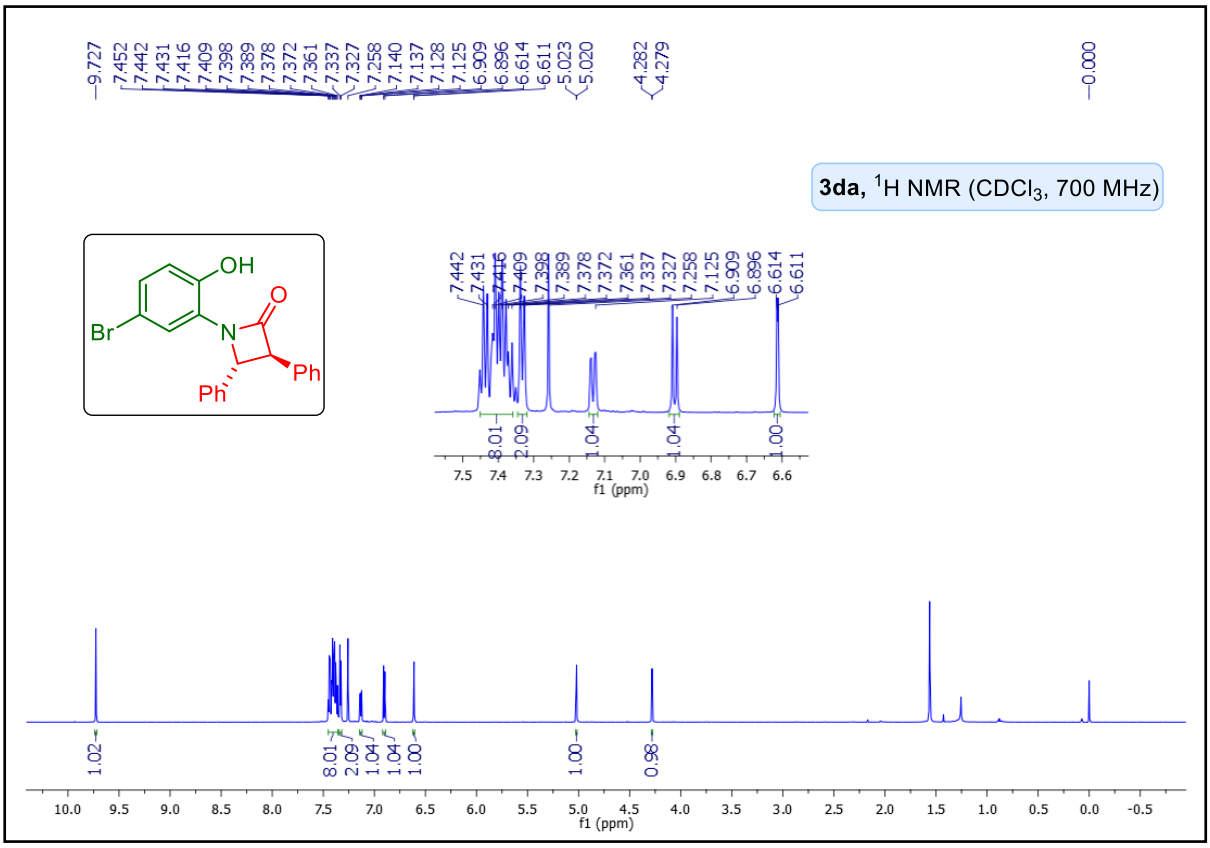
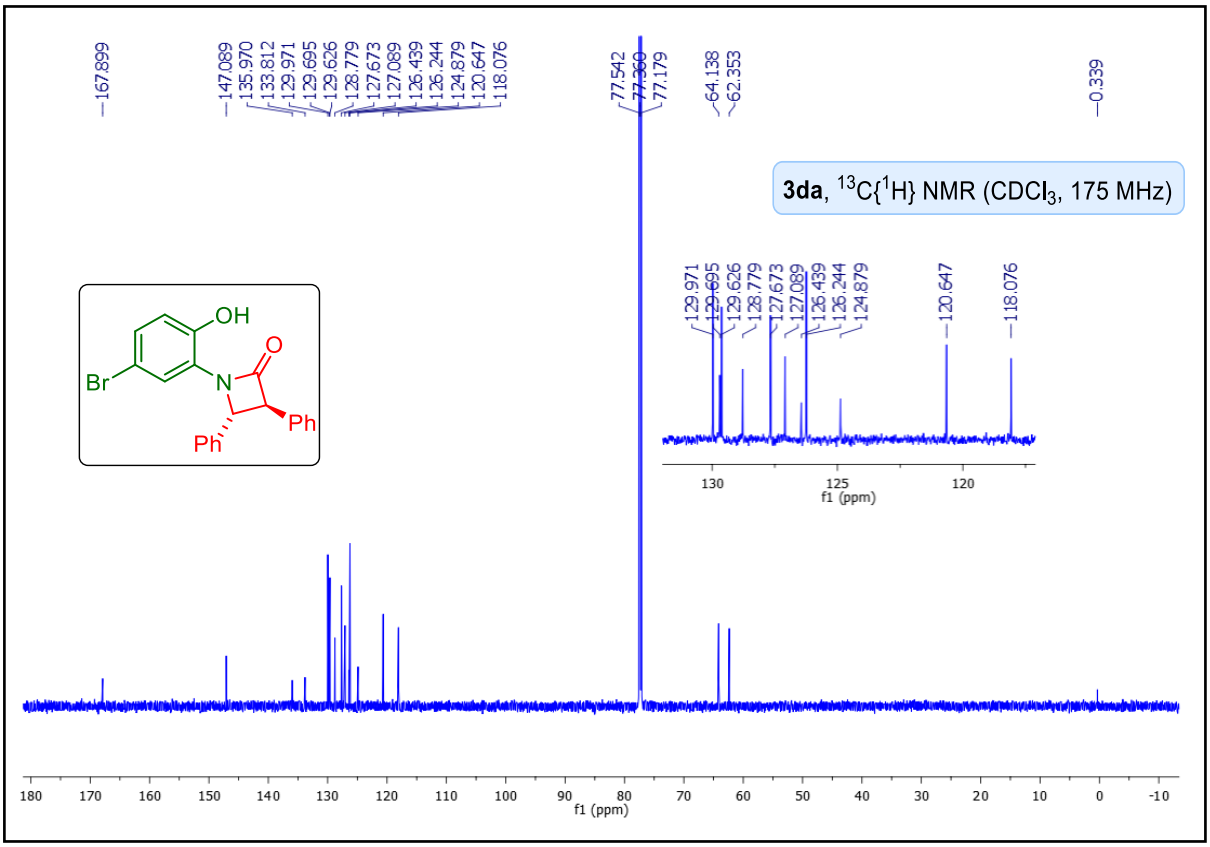
7.413
7.401
7.392
7.373
7.365
7.352
7.333
7.259
6.941
6.920
6.864
6.843
6.323
5.037
5.033
4.242
4.238
2.070
0.000
3ba, 1H NMR (CDCl3, 400 MHz)
OH
O
N
Ph
Ph
10.06
1.05
1.09
f1 (ppm)
1.00
10.06
1.05
1.09
1.07
1.05
1.04
3.20
f1 (ppm)

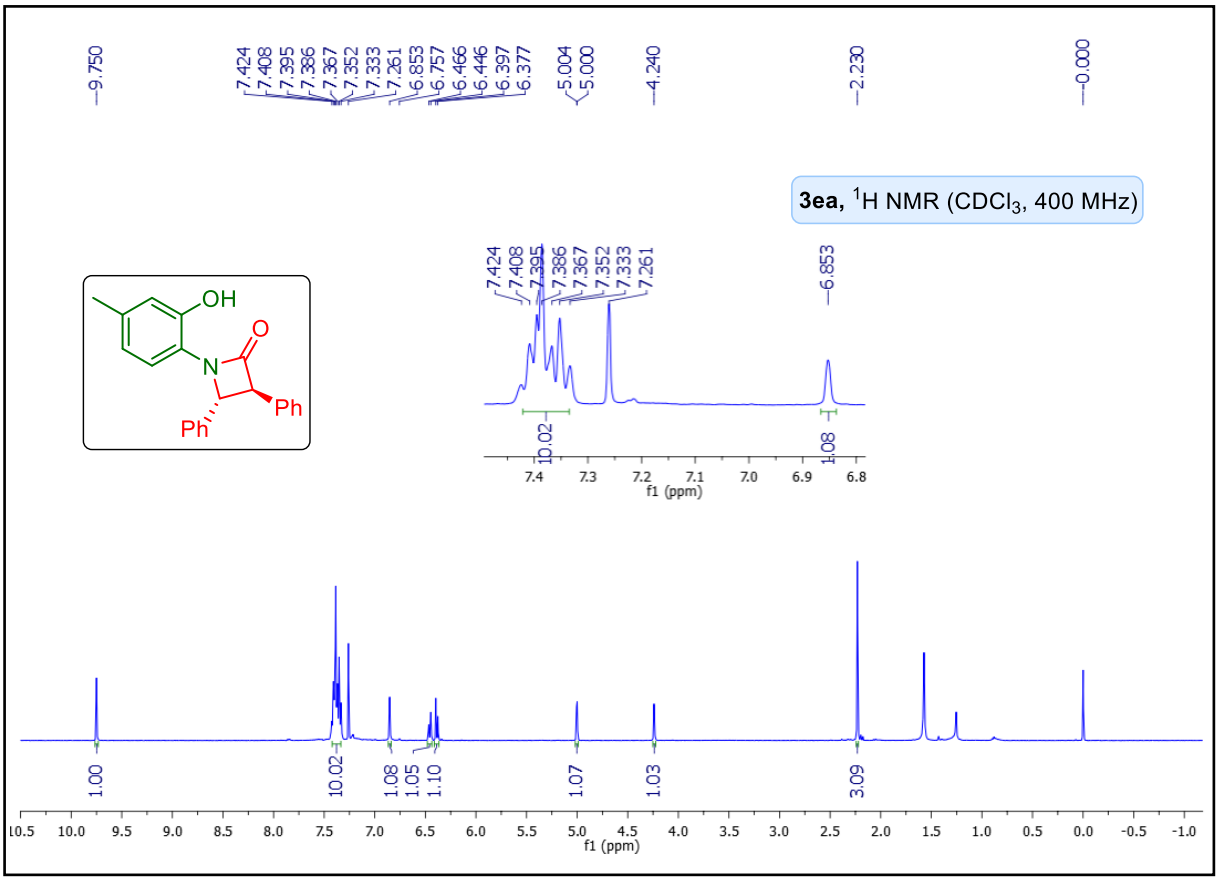

167.507
146.029
136.734
134.271
129.777
129.555
129.331
128.612
128.253
127.964
127.740
126.259
125.104
119.359
118.626
77.541
77.360
77.178
63.958
62.226
20.722
3ba, 13C{1H} NMR (CDCl3, 175 MHz)
OH
O
N
Ph
Ph
f1 (ppm)
f1 (ppm)

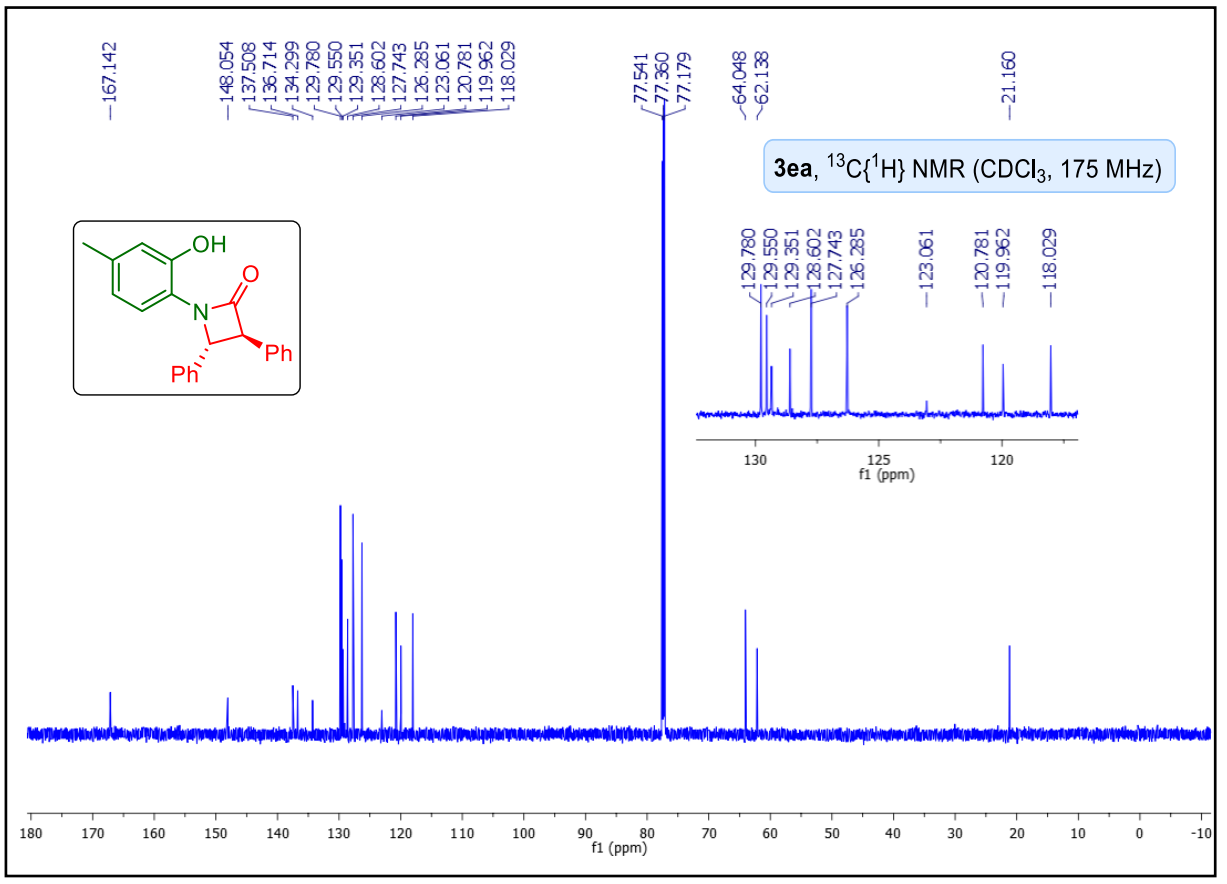

3ca, ^{1}H NMR ($CDCl_3$, 400 MHz)
OH
Cl
N
O
Ph
Ph
f1 (ppm)

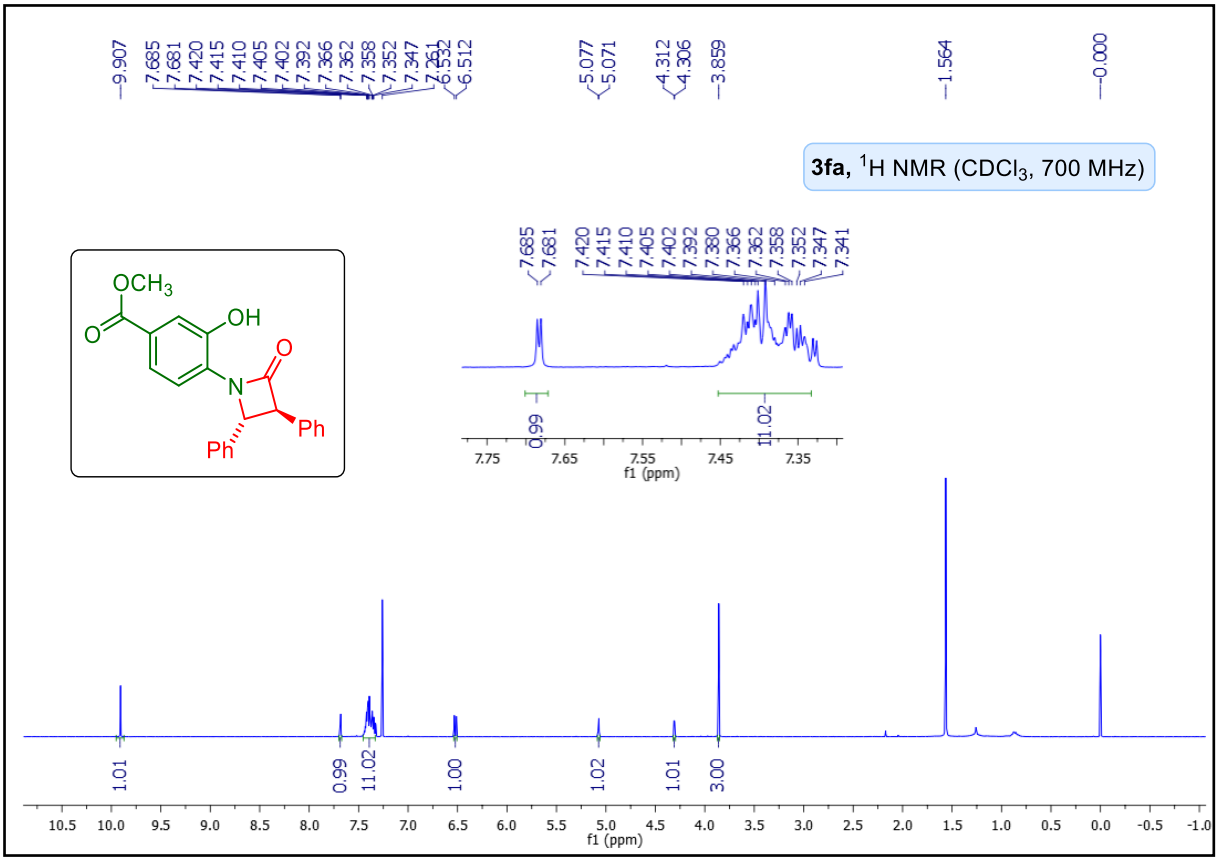

3ca, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
OH
Cl
N
O
Ph
Ph
f1 (ppm)

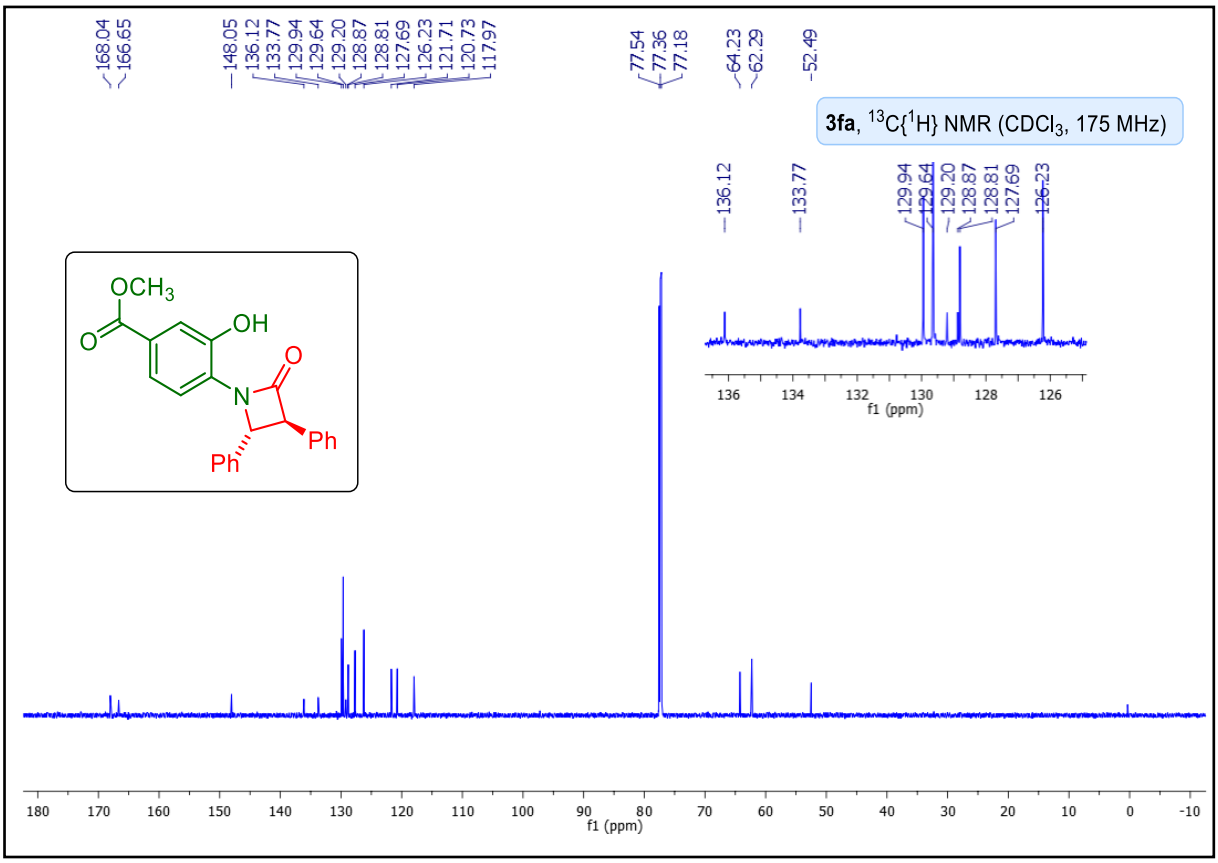

3da, ^{1}H NMR ($CDCl_3$, 700 MHz)
OH
Br
N
O
Ph
Ph
f1 (ppm)

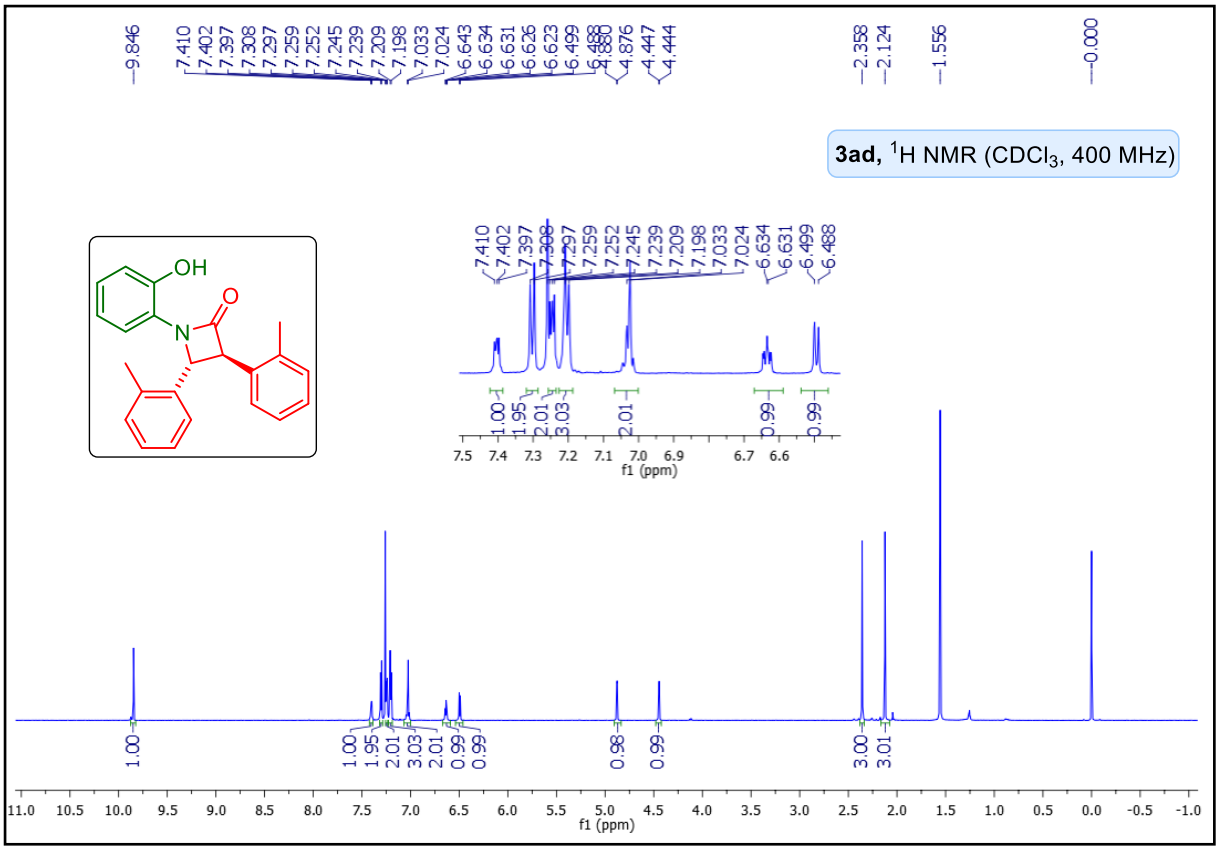

3da, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
OH
Br
N
O
Ph
Ph
f1 (ppm)

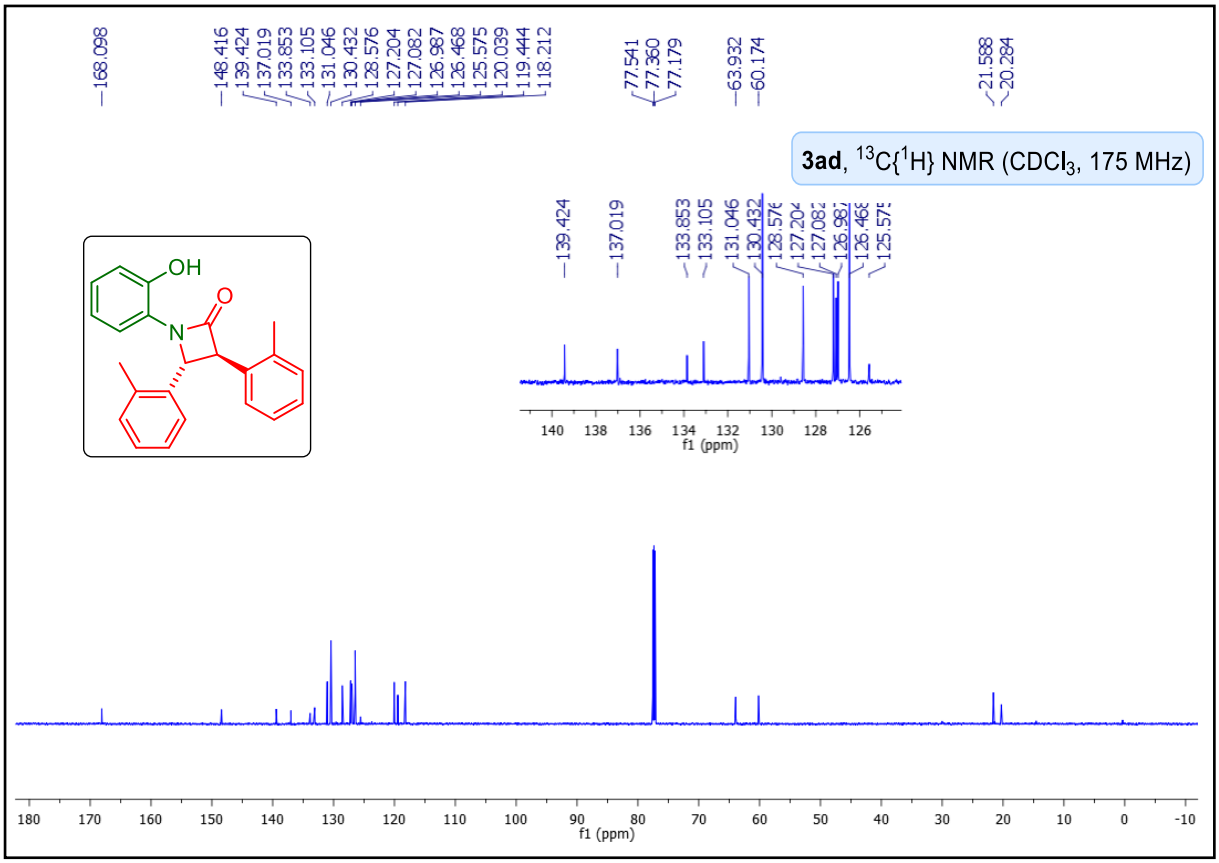

3ea, ^{1}H NMR ($CDCl_3$, 400 MHz)
OH
O
N
Ph
Ph
f1 (ppm)

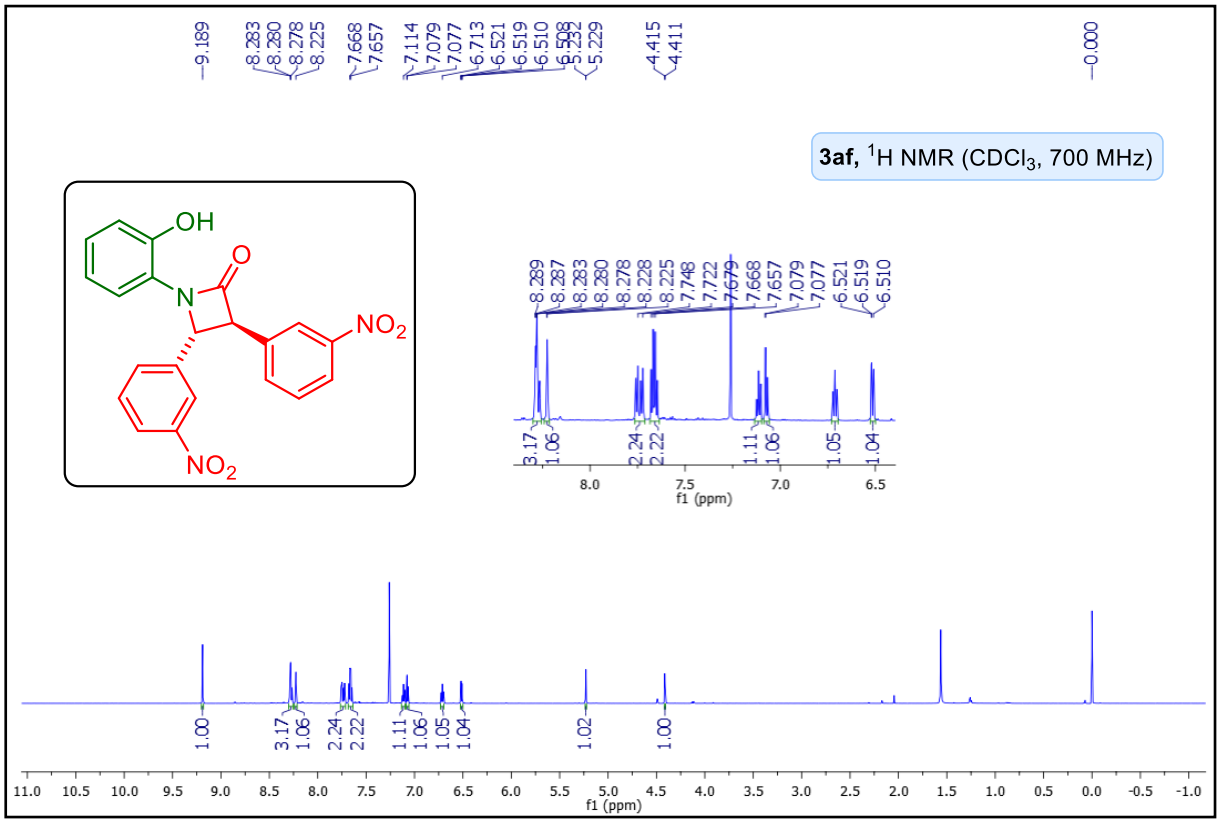

3ea, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
OH
O
N
Ph
Ph
f1 (ppm)

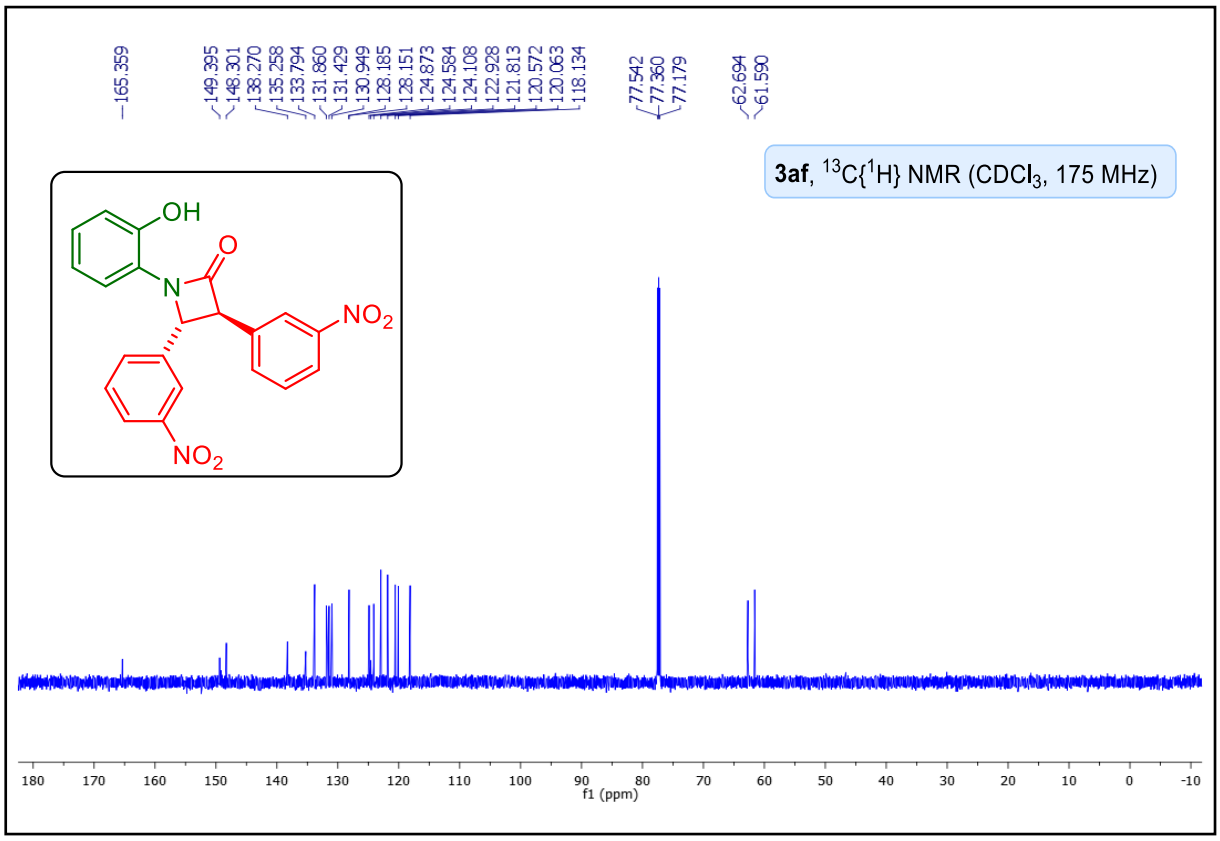

3fa, ^{1}H NMR ($CDCl_3$, 700 MHz)
OCH3
OH
O
N
O
Ph
Ph
f1 (ppm)

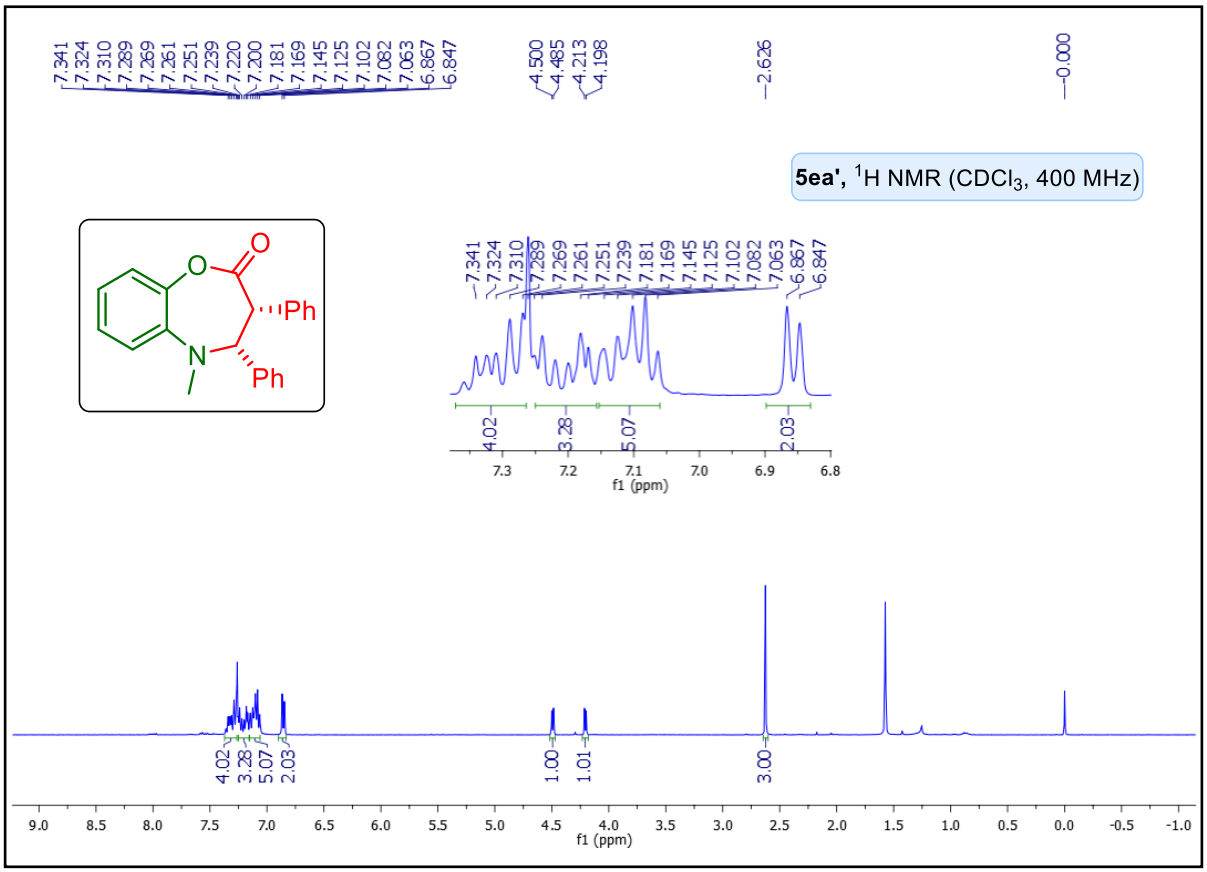

3fa, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
OCH3
OH
O
N
O
Ph
Ph
f1 (ppm)

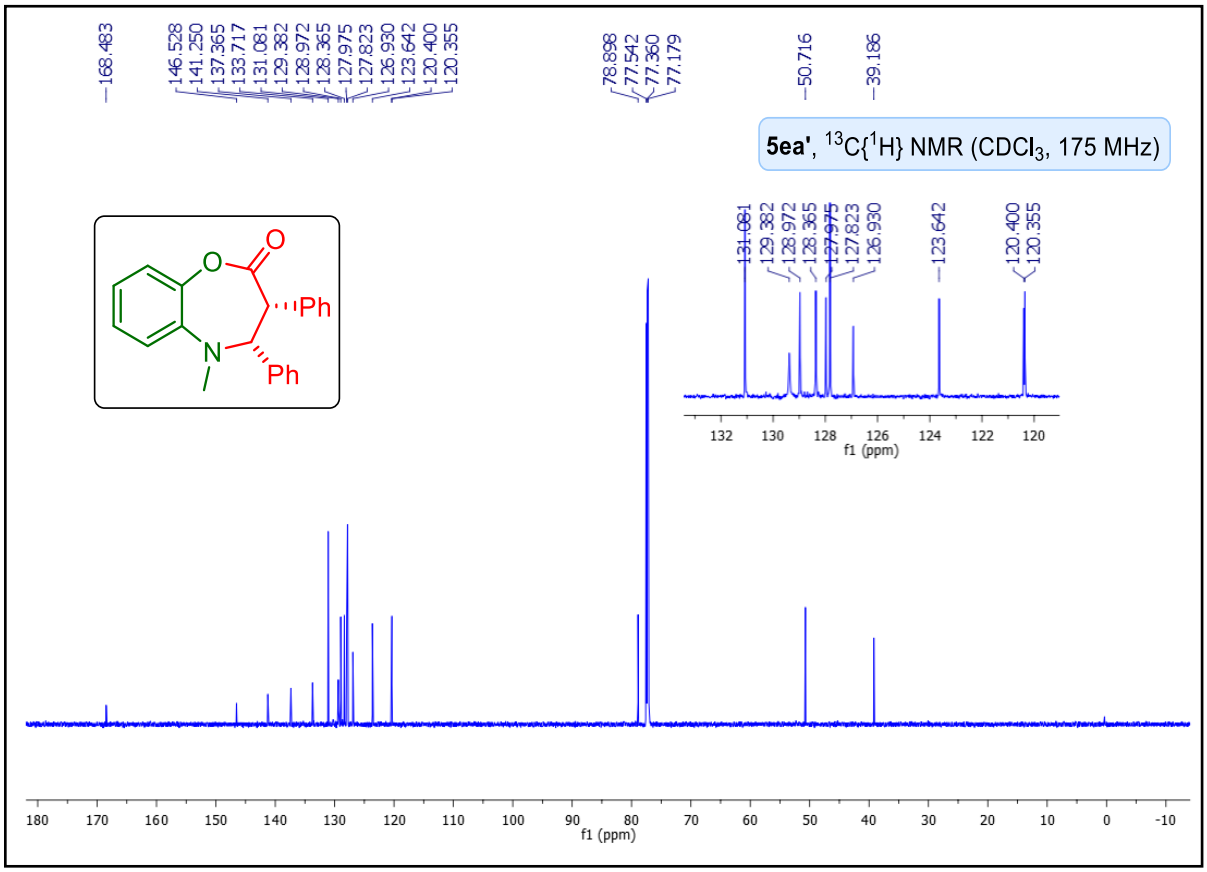

3ad, ^{1}H NMR ($CDCl_3$, 400 MHz)
OH
N
O
f1 (ppm)

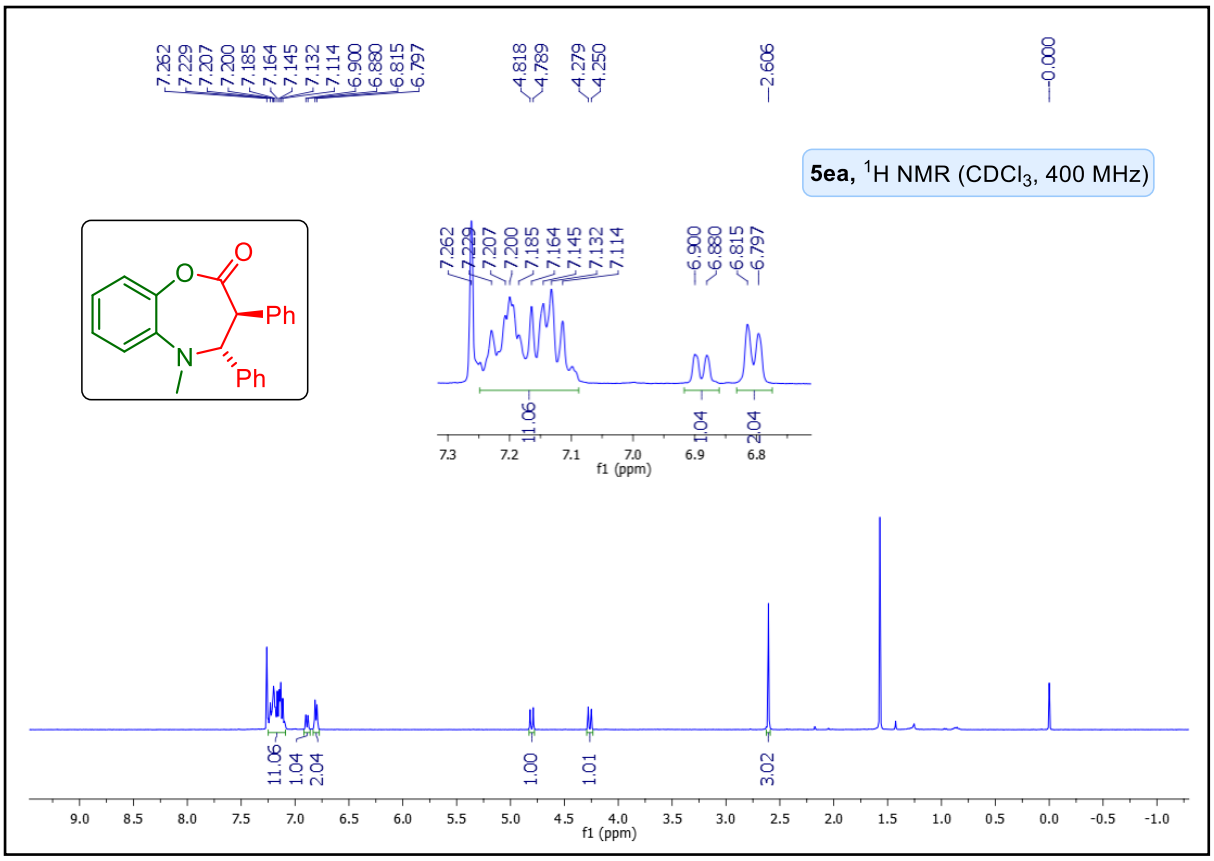

3ad, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
OH
N
O
f1 (ppm)

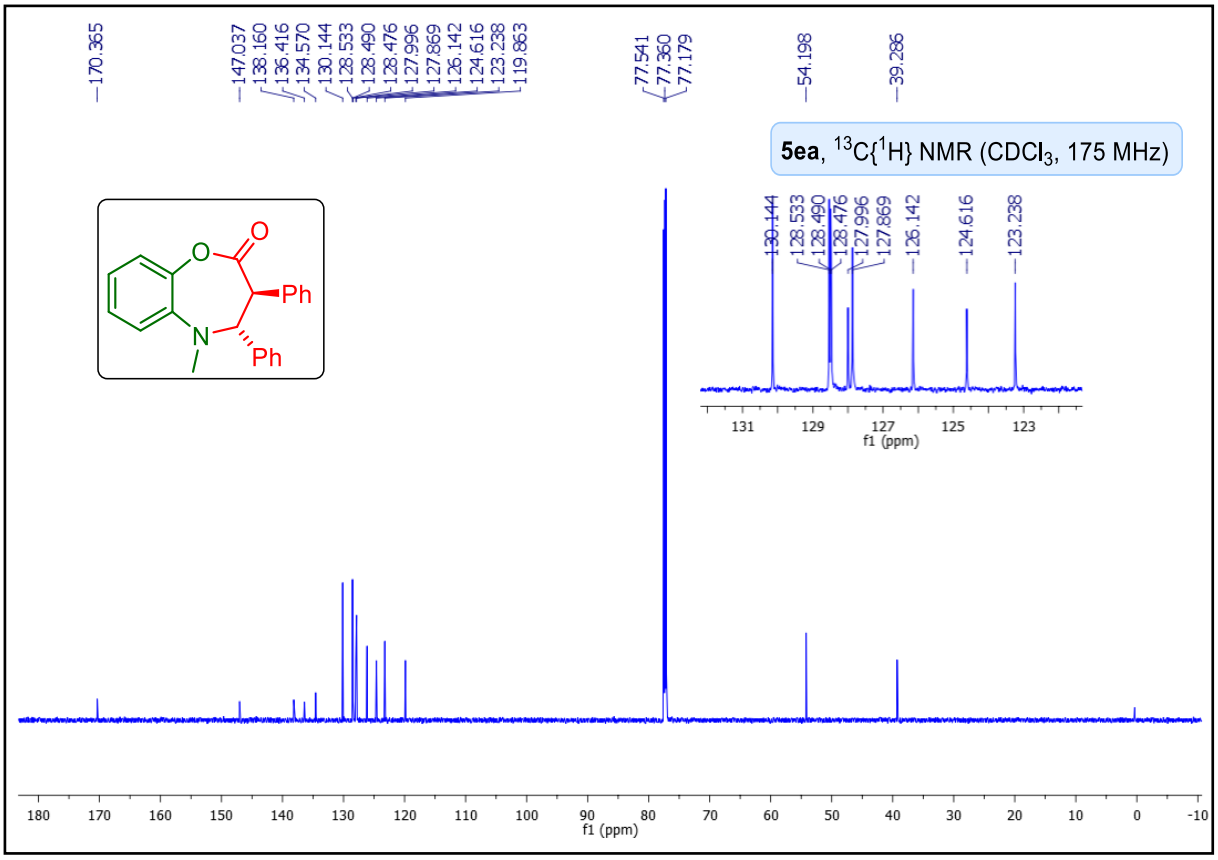

9.189
8.283
8.280
8.278
8.225
7.668
7.657
7.114
7.079
7.077
6.713
6.521
6.519
6.510
6.508
5.232
5.229
4.415
4.411
0.000
3af, 1H NMR (CDCl3, 700 MHz)
OH
O
N
NO2
NO2
f1 (ppm)


165.359
149.395
148.301
138.270
135.258
133.794
131.860
131.429
130.949
128.185
128.151
124.873
124.584
124.108
122.928
121.813
120.572
120.063
118.134
77.542
77.360
77.179
62.694
61.590
3af, 13C{1H} NMR (CDCl3, 175 MHz)
OH
O
N
NO2
NO2
f1 (ppm)


7.341
7.324
7.310
7.289
7.269
7.261
7.251
7.239
7.220
7.200
7.181
7.169
7.145
7.125
7.102
7.082
7.063
6.867
6.847
4.500
4.485
4.213
4.198
2.626
0.000
5ea', ^{1}H NMR ($CDCl_3$, 400 MHz)
O
O
Ph
N
Ph
4.02
3.28
5.07
2.03
f1 (ppm)
1.00
1.01
3.00


168.483
146.528
141.250
137.365
133.717
131.081
129.382
128.972
128.365
127.975
127.823
126.930
123.642
120.400
120.355
78.898
77.542
77.360
77.179
50.716
39.186
5ea', ^{13}C{^{1}H} NMR ($CDCl_3$, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)


7.262
7.229
7.207
7.200
7.185
7.164
7.145
7.132
7.114
6.900
6.880
6.815
6.797
4.818
4.789
4.279
4.250
2.606
0.000
5ea, 1H NMR (CDCl3, 400 MHz)
O
O
Ph
N
Ph
11.06
1.04
2.04
1.00
1.01
3.02
f1 (ppm)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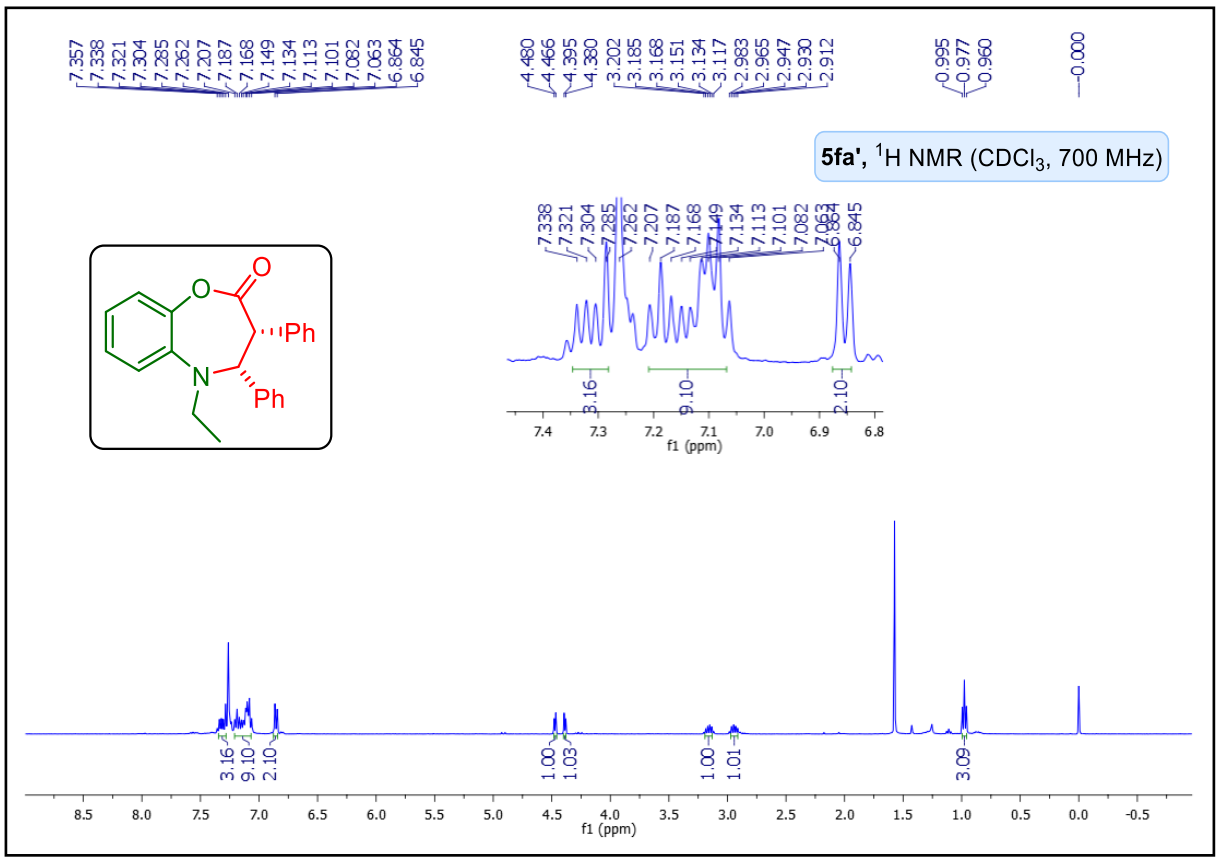
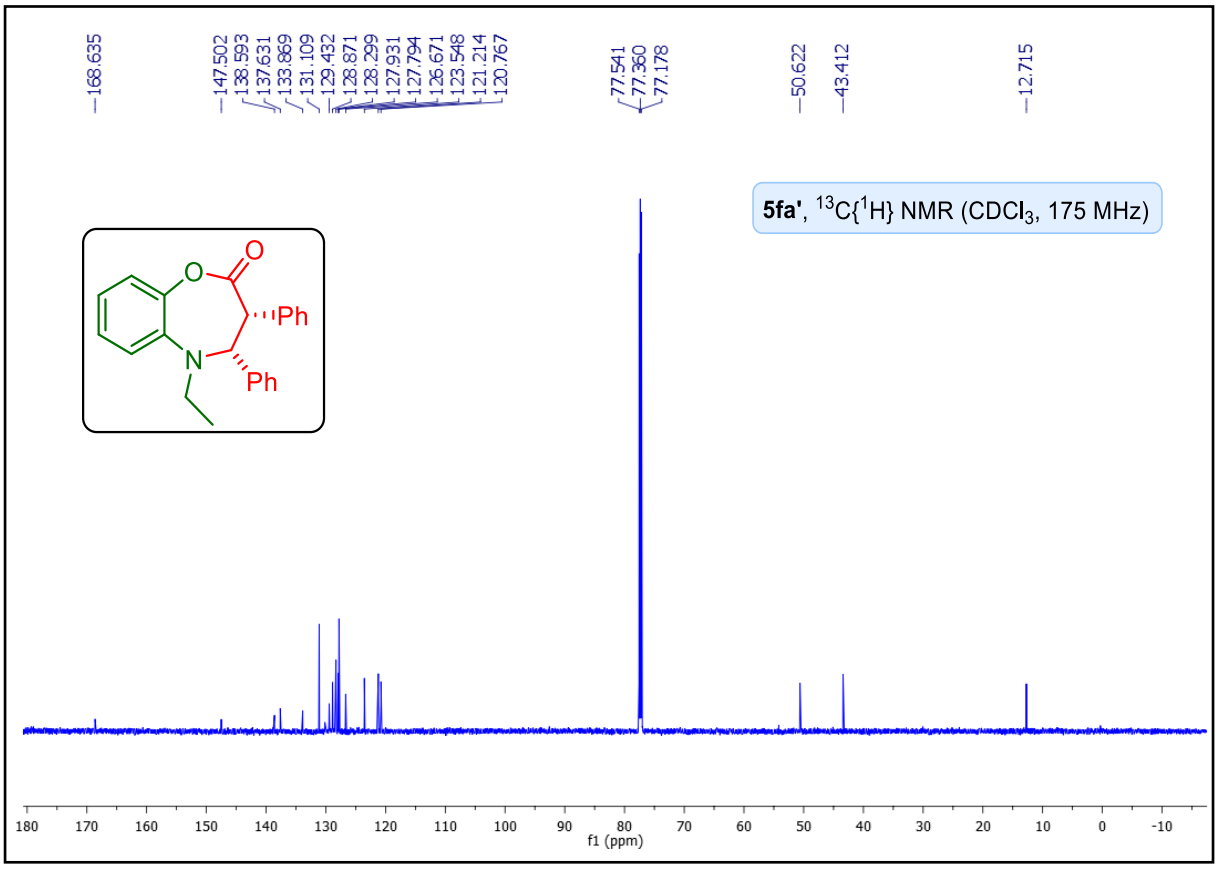
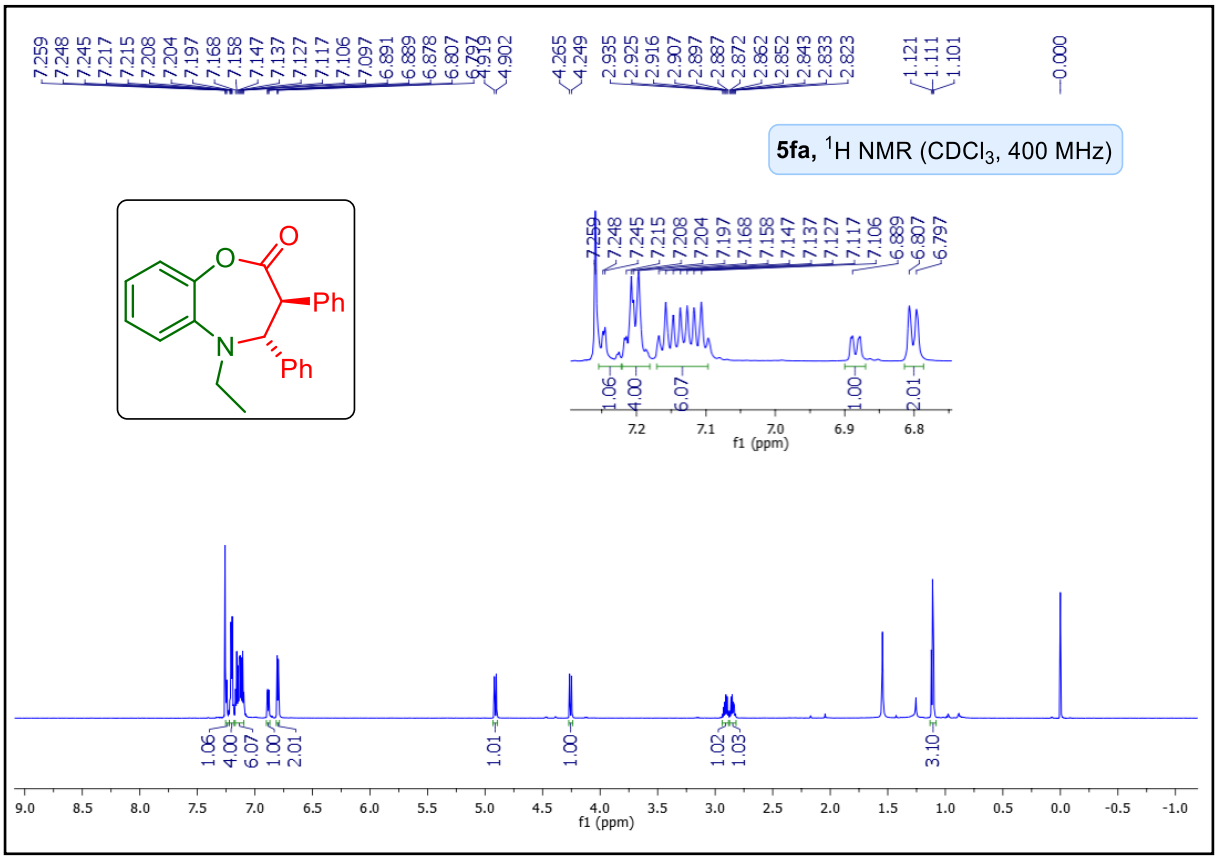
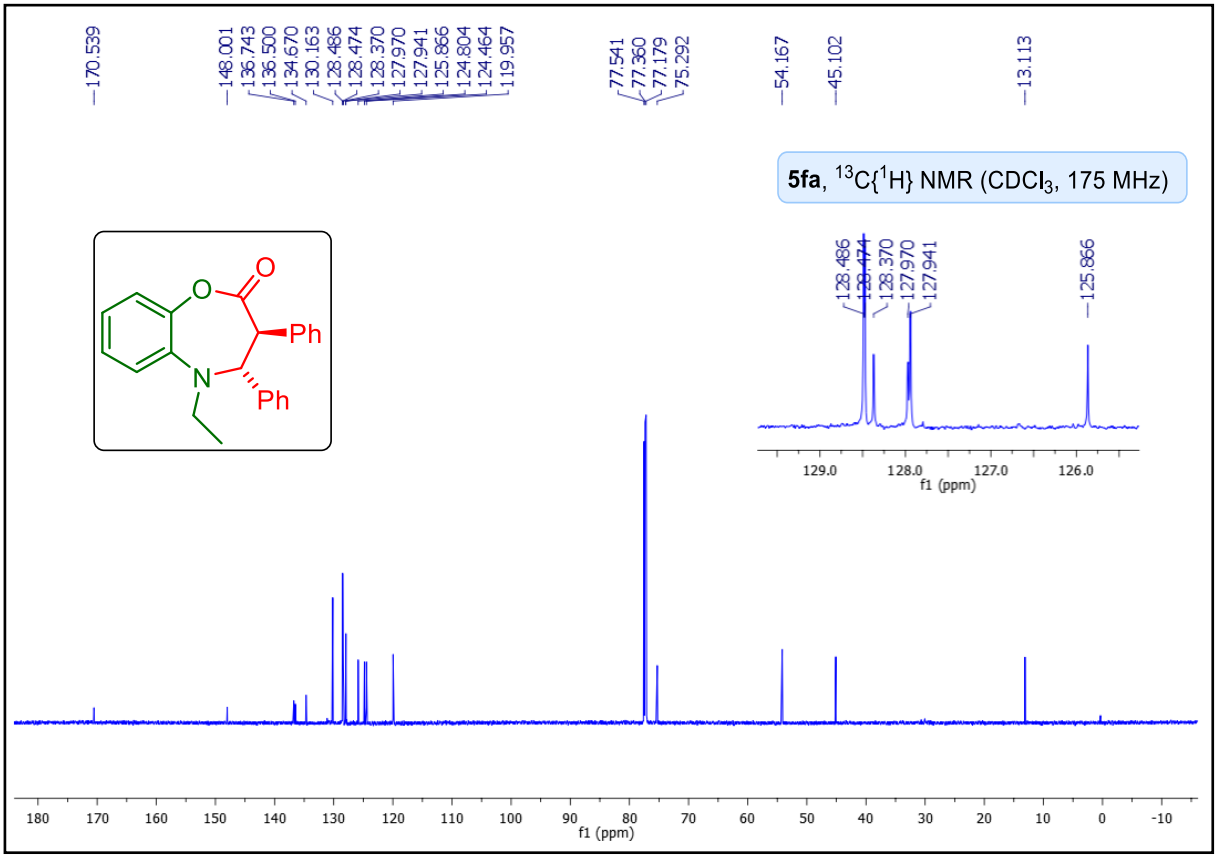
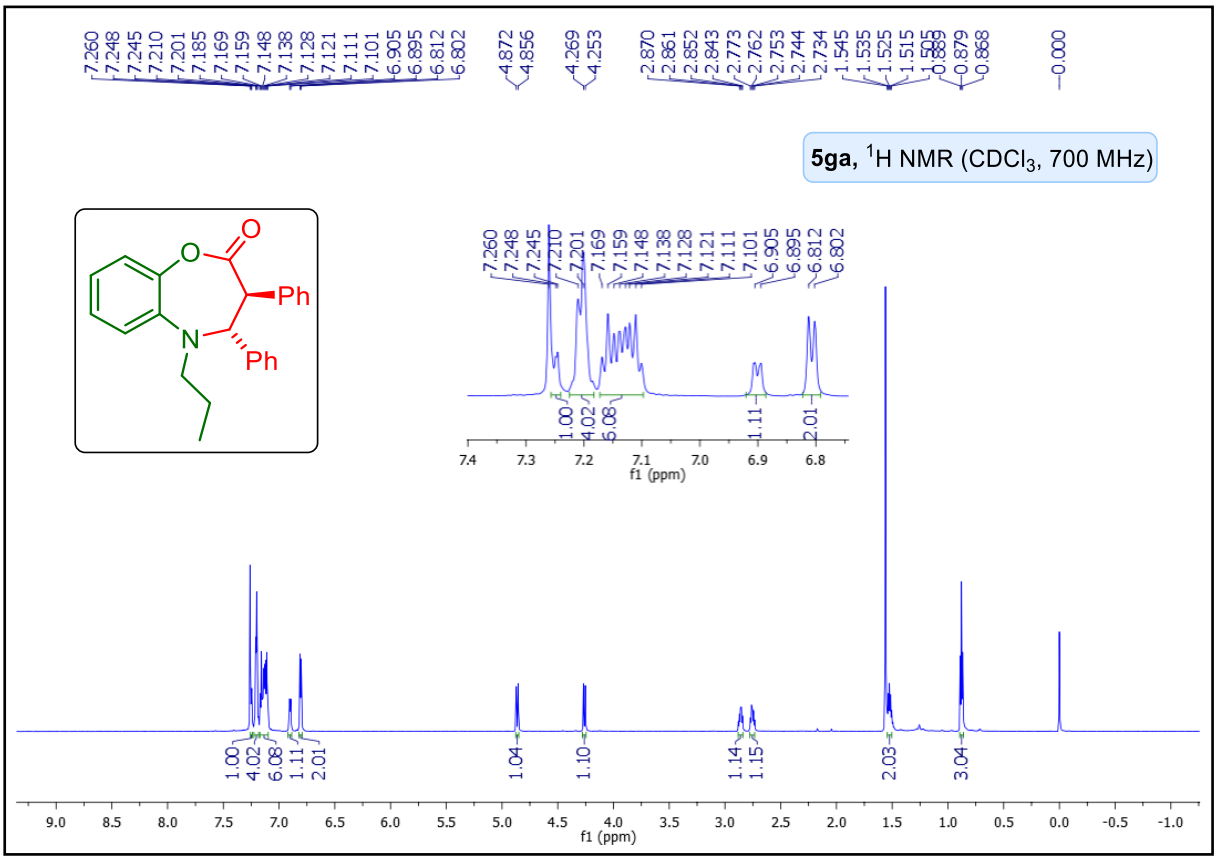
147.037
138.160
136.416
134.570
130.144
128.533
128.490
128.476
127.996
127.869
126.142
124.616
123.238
119.863
77.541
77.360
77.179
54.198
39.286
5ea, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

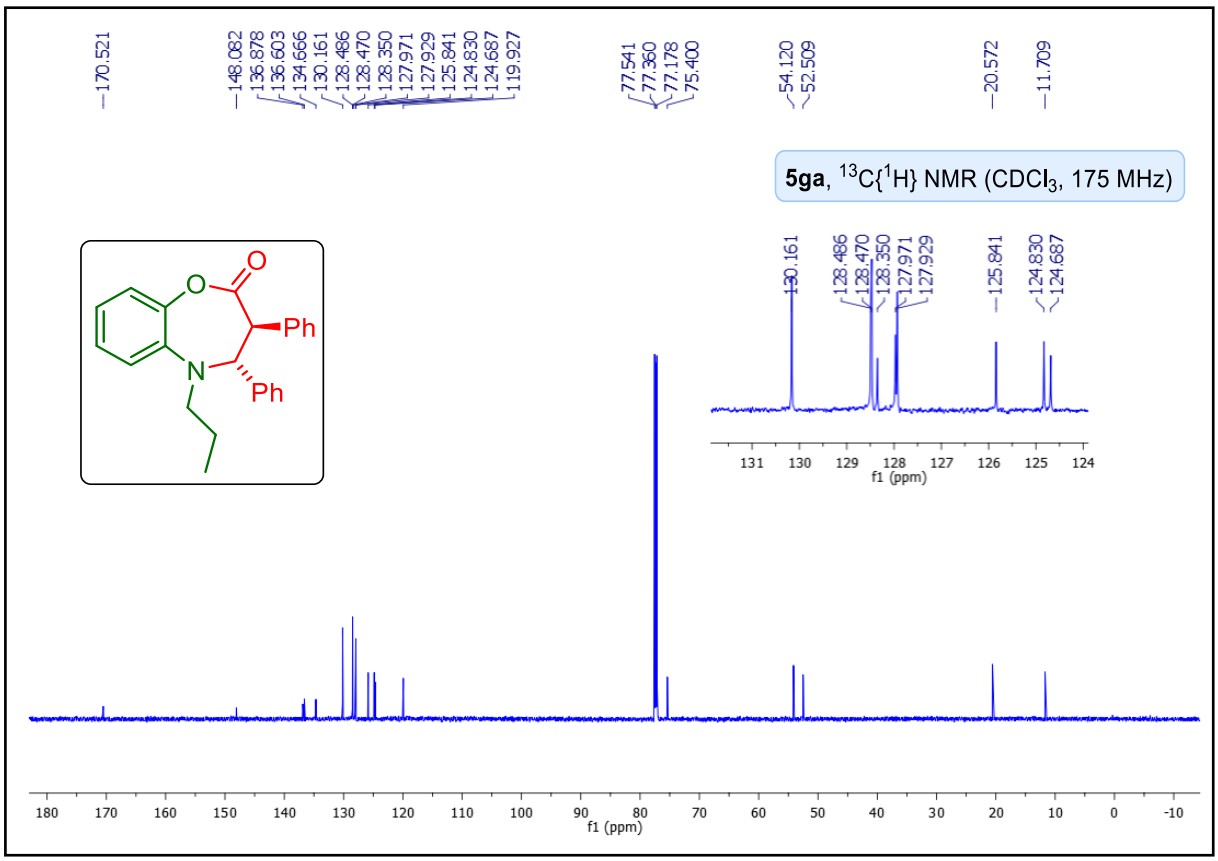

7.357
7.338
7.321
7.304
7.285
7.262
7.207
7.187
7.168
7.149
7.134
7.113
7.101
7.082
7.063
6.864
6.845
4.480
4.466
4.395
4.380
3.202
3.185
3.168
3.151
3.134
3.117
2.983
2.965
2.947
2.930
2.912
0.995
0.977
0.960
0.000
5fa', 1H NMR (CDCl3, 700 MHz)
O
O
Ph
N
Ph
3.16
9.10
2.10
f1 (ppm)
3.16
9.10
2.10
1.00
1.03
1.00
1.01
3.09
f1 (ppm)

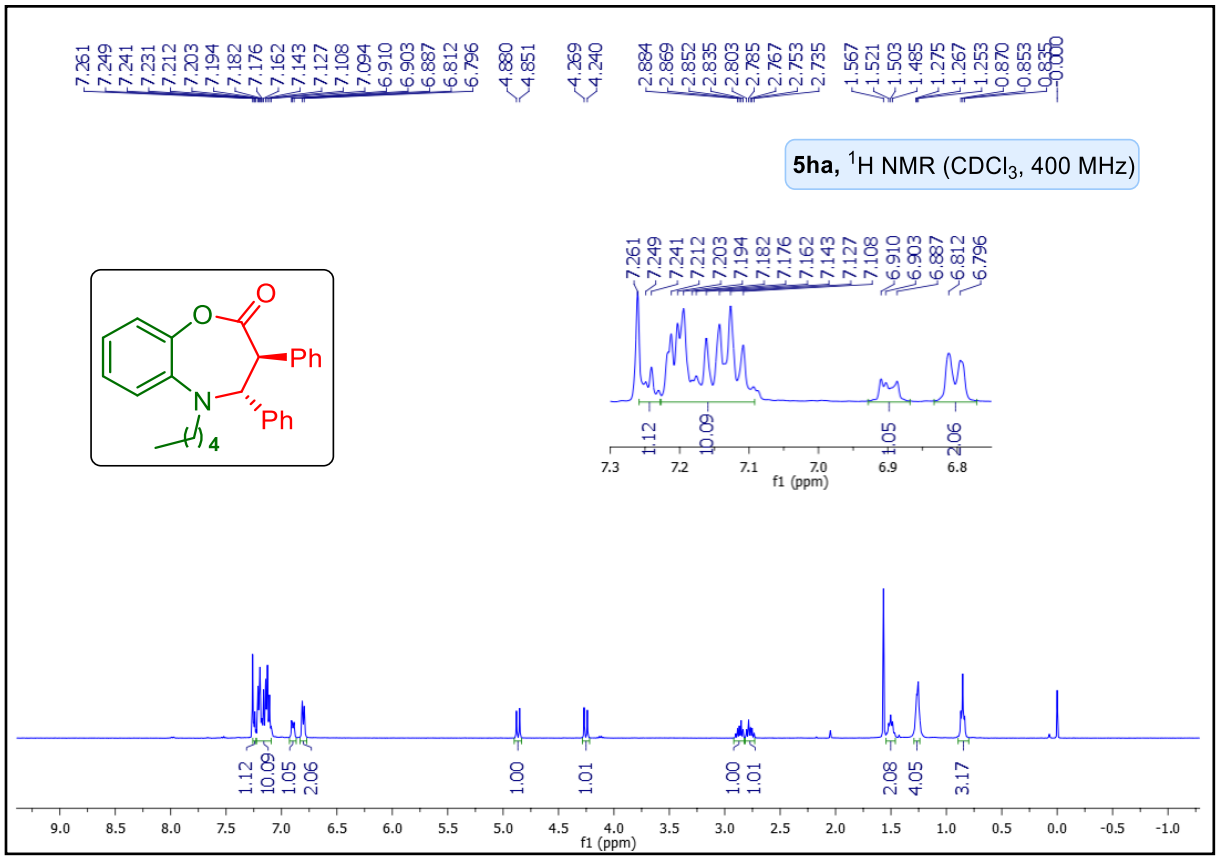

168.635
147.502
138.593
137.631
133.869
131.109
129.432
128.871
128.299
127.931
127.794
126.671
123.548
121.214
120.767
77.541
77.360
77.178
50.622
43.412
12.715
5fa', 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

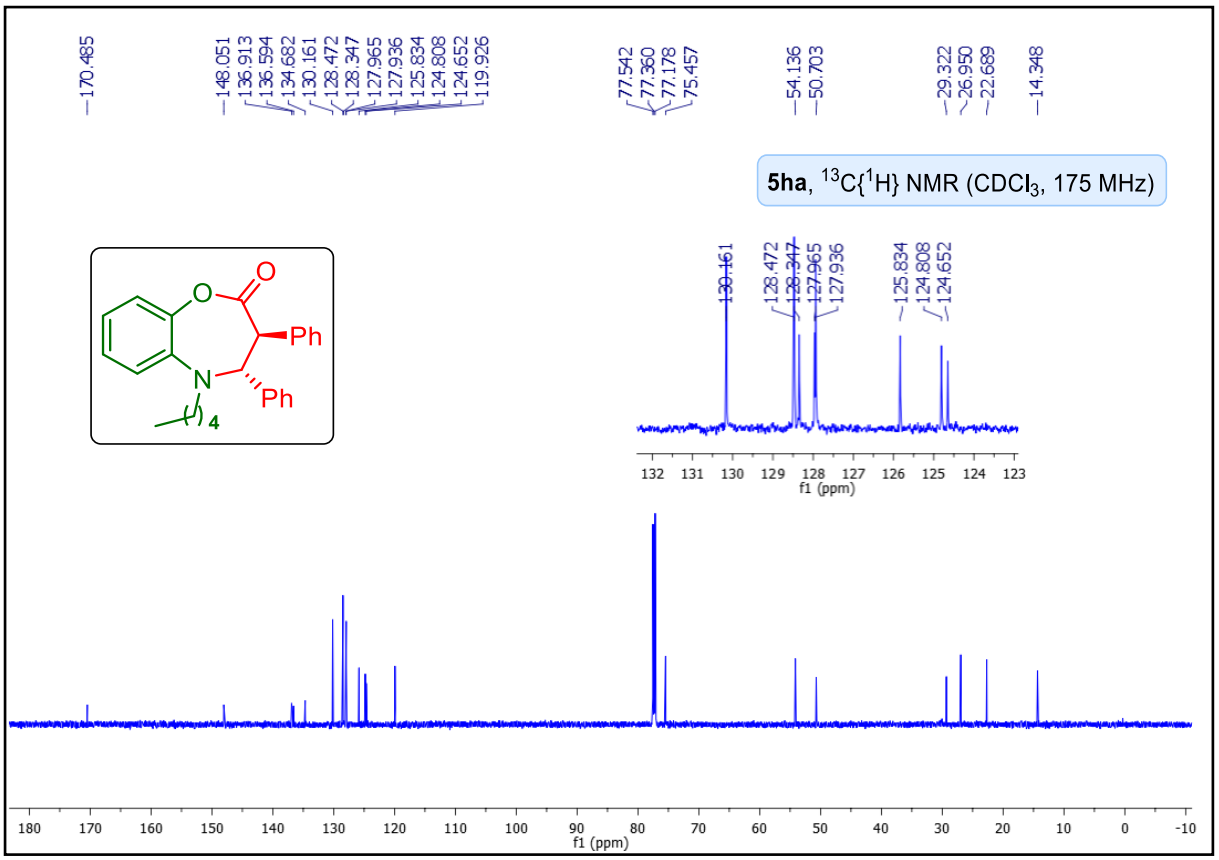

7.259
7.248
7.245
7.217
7.215
7.208
7.204
7.197
7.168
7.158
7.147
7.137
7.127
7.117
7.106
7.097
6.891
6.889
6.878
6.807
6.797
6.919
4.902
4.265
4.249
2.935
2.925
2.916
2.907
2.897
2.887
2.872
2.862
2.852
2.843
2.833
2.823
1.121
1.111
1.101
0.000
5fa, 1H NMR (CDCl3, 400 MHz)
O
O
Ph
N
Ph
1.06
4.00
6.07
1.00
2.01
1.01
1.00
1.02
1.03
3.10
f1 (ppm)

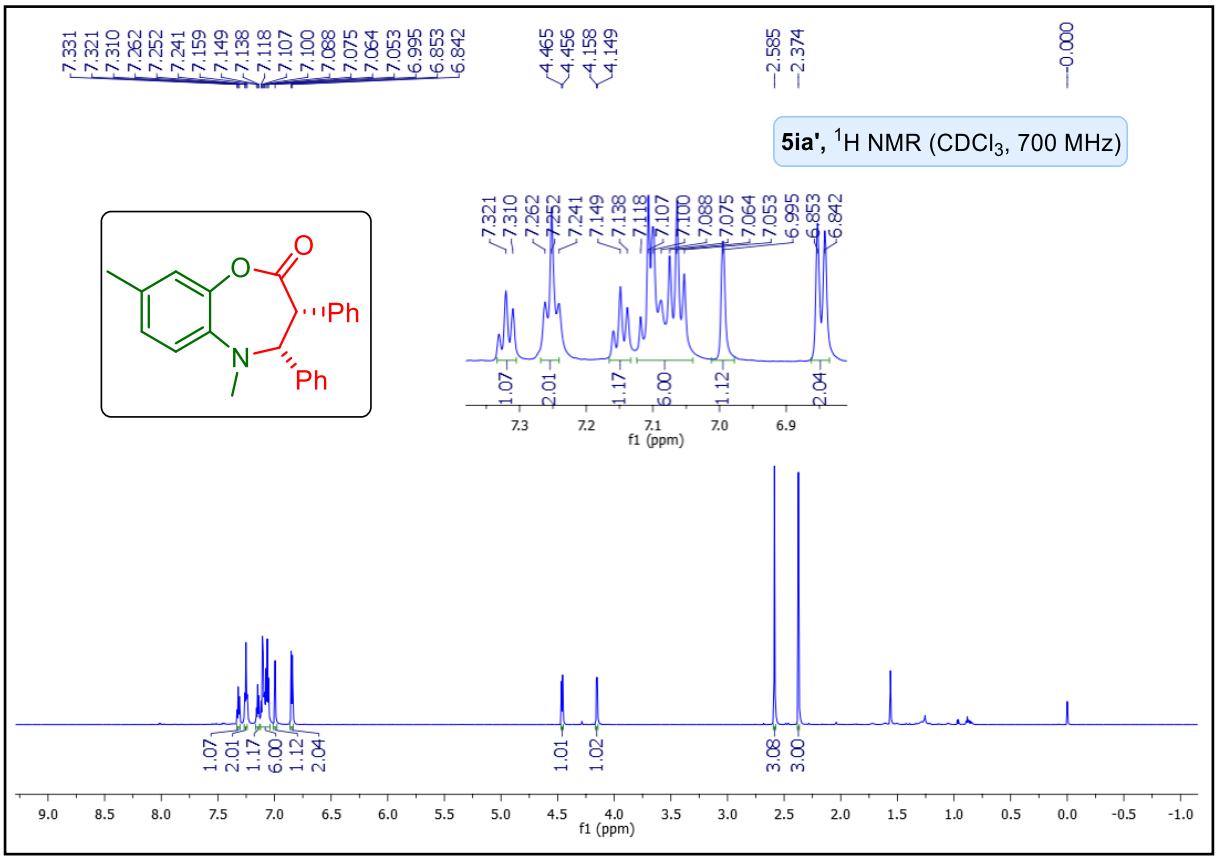

170.539
148.001
136.743
136.500
134.670
130.163
128.486
128.474
128.370
127.970
127.941
125.866
124.804
124.464
119.957
77.541
77.360
77.179
75.292
54.167
45.102
13.113
5fa, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

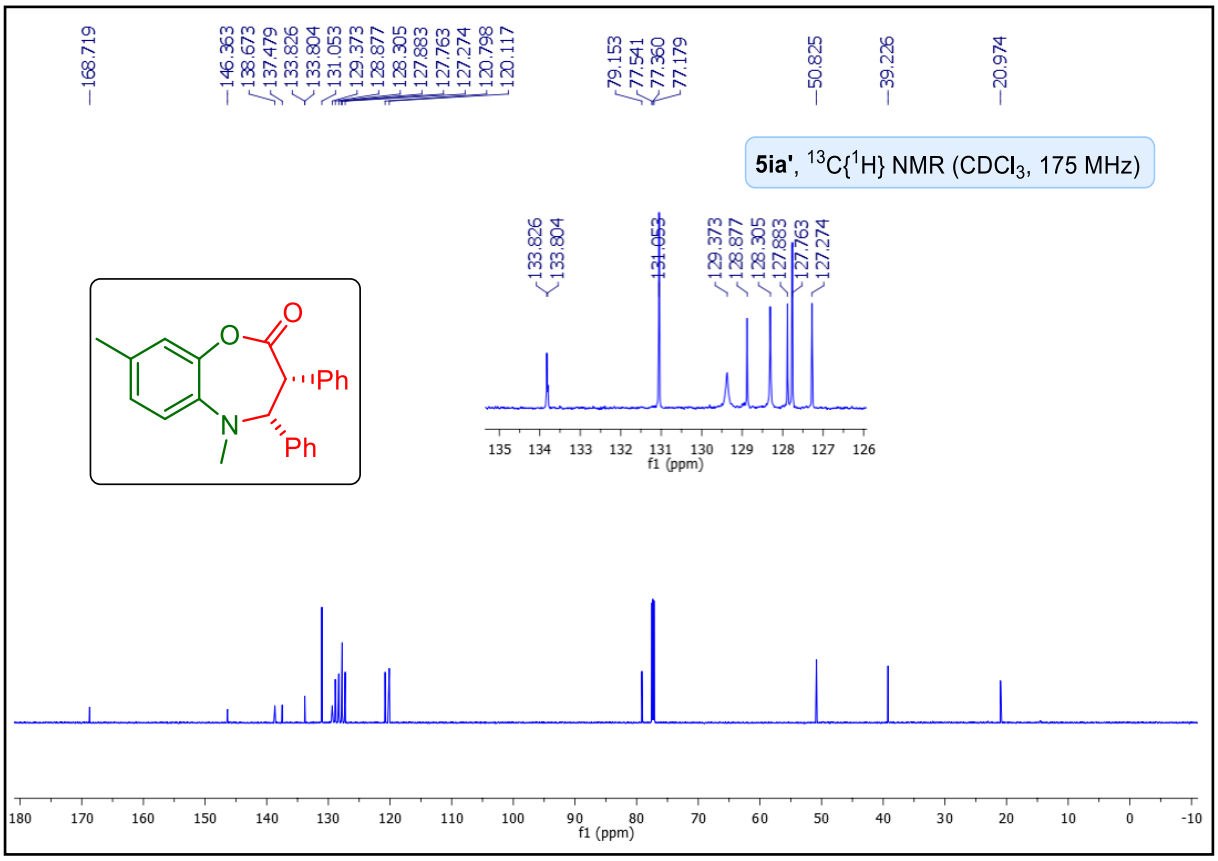

7.260
7.248
7.245
7.210
7.201
7.185
7.169
7.159
7.148
7.138
7.128
7.121
7.111
7.101
6.905
6.895
6.812
6.802
4.872
4.856
4.269
4.253
2.870
2.861
2.852
2.843
2.773
2.762
2.753
2.744
2.734
1.545
1.535
1.525
1.515
0.879
0.868
0.000
5ga, 1H NMR (CDCl3, 700 MHz)
O
Ph
N
Ph
1.00
4.02
6.08
1.11
2.01
1.04
1.10
1.14
1.15
2.03
3.04
f1 (ppm)

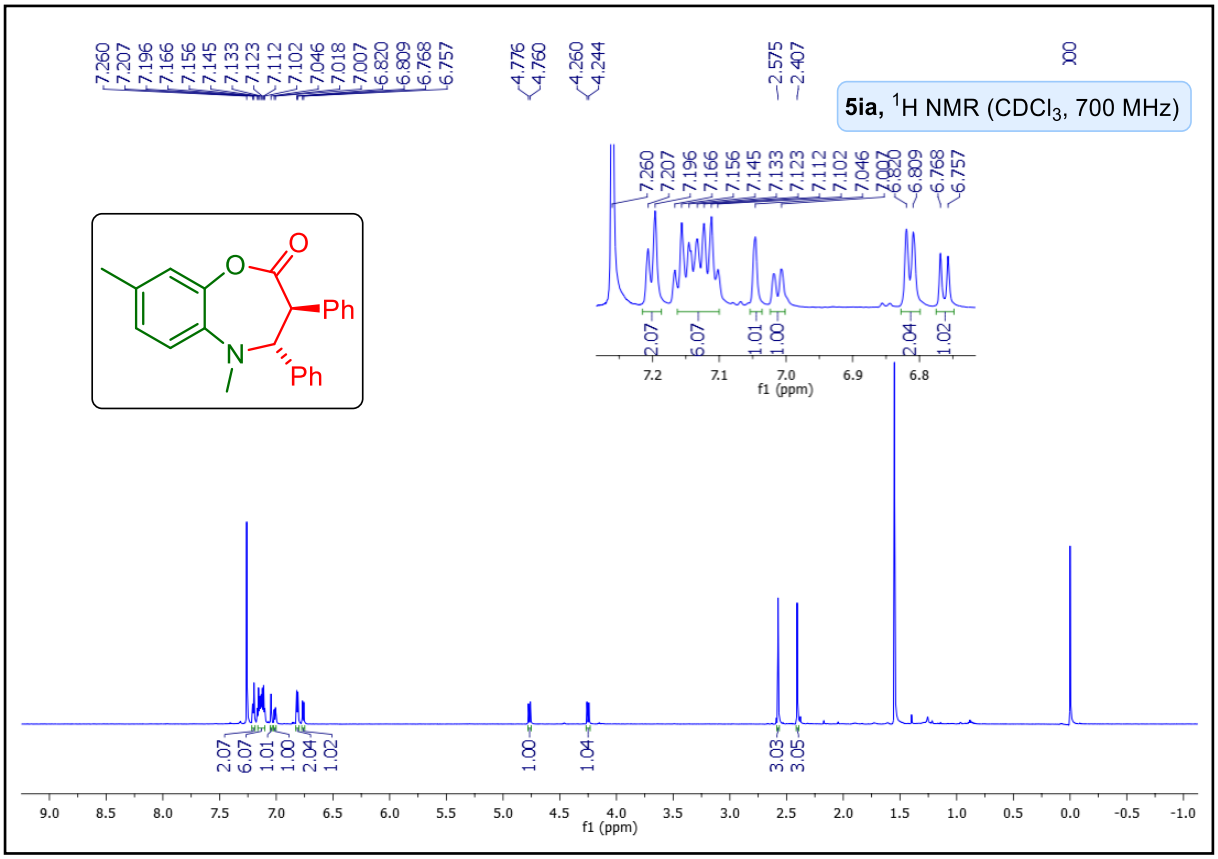

170.521
148.082
136.878
136.603
134.666
130.161
128.486
128.470
128.350
127.971
127.929
125.841
124.830
124.687
119.927
77.541
77.360
77.178
75.400
54.120
52.509
20.572
11.709
5ga, 13C{1H} NMR (CDCl3, 175 MHz)
O
Ph
N
Ph
f1 (ppm)

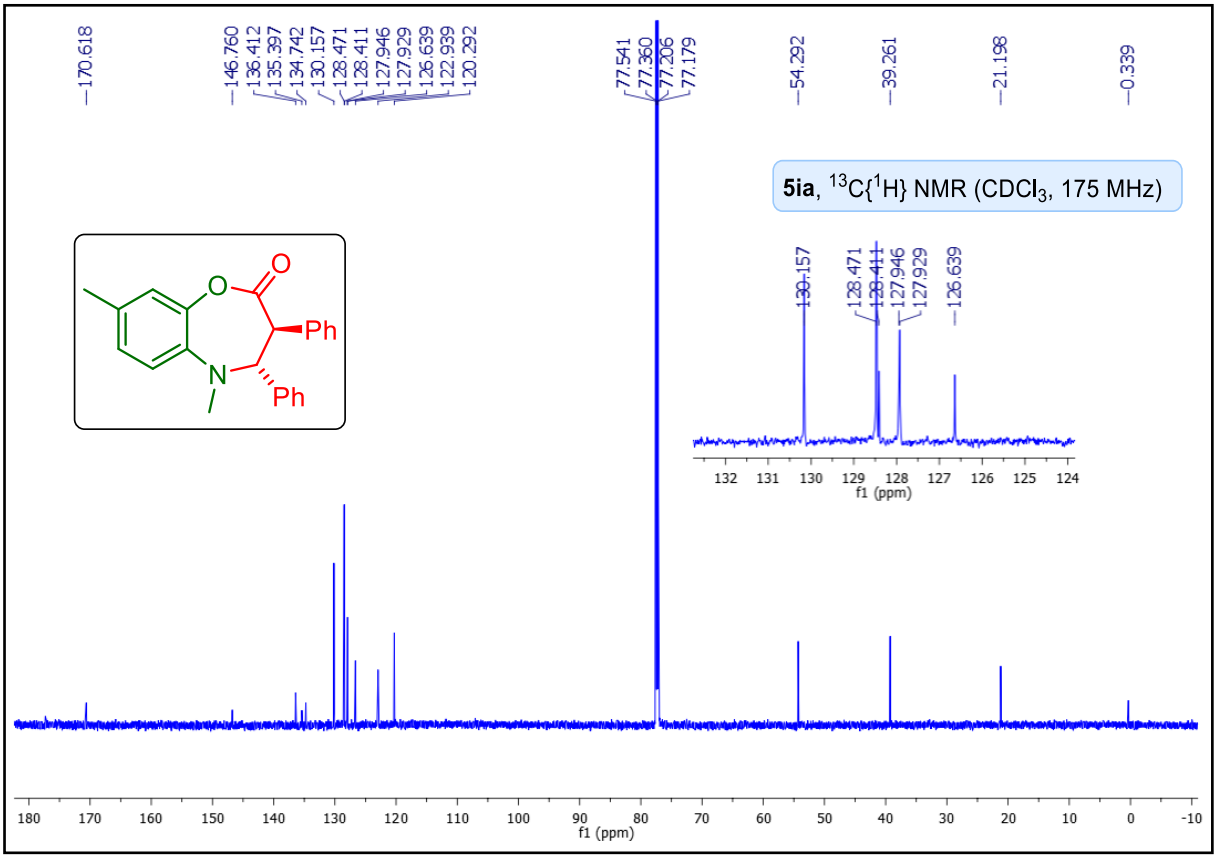

7.261
7.249
7.241
7.231
7.212
7.203
7.194
7.182
7.176
7.162
7.143
7.127
7.108
7.094
6.910
6.903
6.887
6.812
6.796
4.880
4.851
4.269
4.240
2.884
2.869
2.852
2.835
2.803
2.785
2.767
2.753
2.735
1.567
1.521
1.503
1.485
1.275
1.267
1.253
0.870
0.853
0.835
0.000
5ha, 1H NMR (CDCl3, 400 MHz)
O
O
Ph
N
Ph
4
1.12
10.09
1.05
2.06
7.3
7.2
7.1
7.0
6.9
6.8
f1 (ppm)
1.12
10.09
1.05
2.06
1.00
1.01
1.00
1.01
2.08
4.05
3.17
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

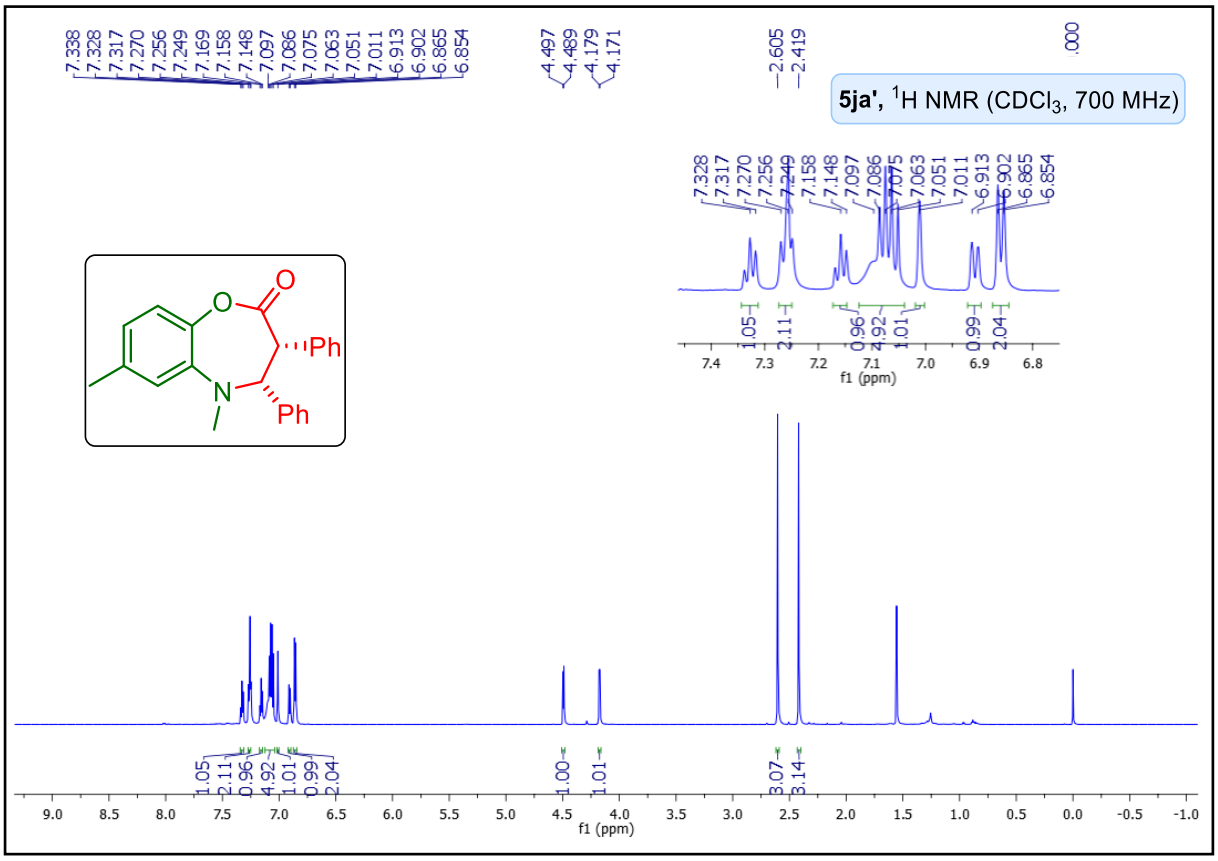

170.485
148.051
136.913
136.594
134.682
130.161
128.472
128.347
127.965
127.936
125.834
124.808
124.652
119.926
77.542
77.360
77.178
75.457
54.136
50.703
29.322
26.950
22.689
14.348
5ha, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
4
130.161
128.472
128.347
127.965
127.936
125.834
124.808
124.652
132
131
130
129
128
127
126
125
124
123
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

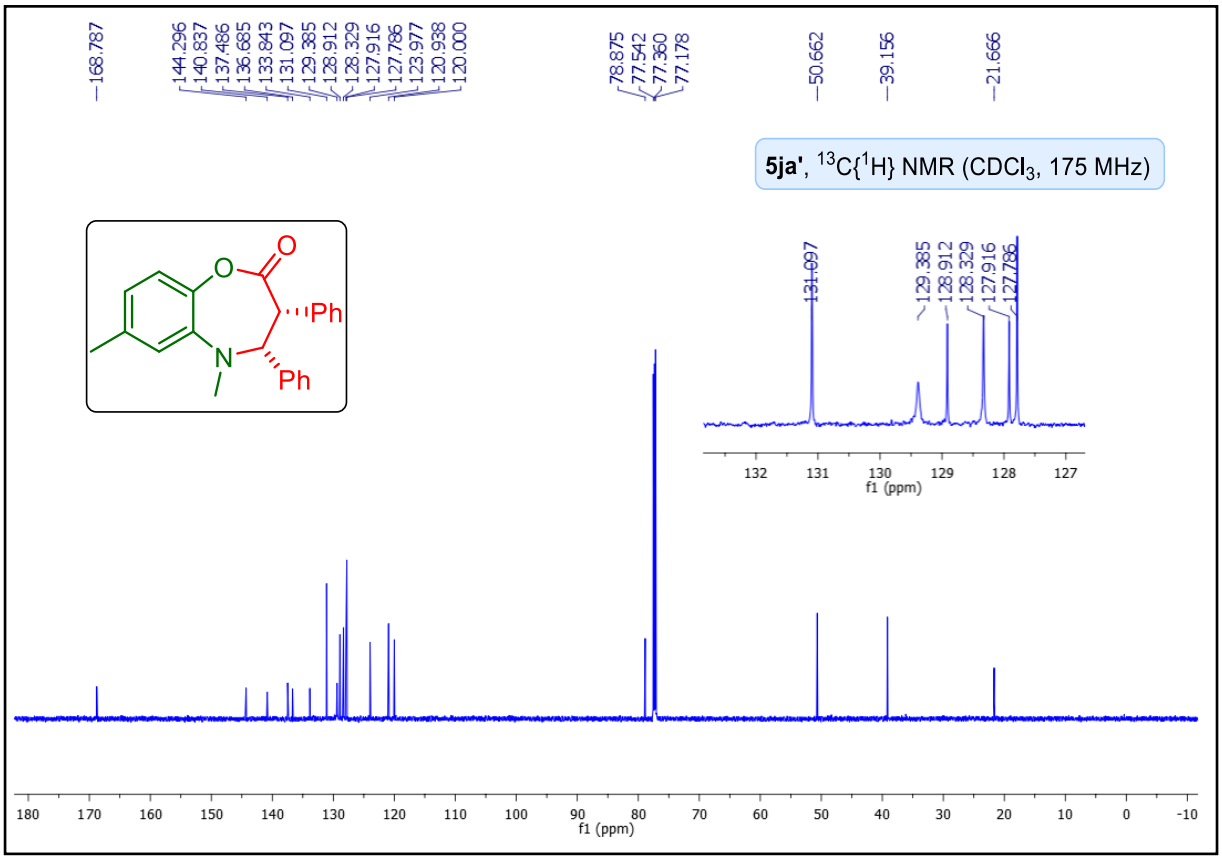

7.331
7.321
7.310
7.262
7.252
7.241
7.159
7.149
7.138
7.118
7.107
7.100
7.088
7.075
7.064
7.053
6.995
6.853
6.842
4.465
4.456
4.158
4.149
2.585
2.374
0.000
5ia', 1H NMR (CDCl3, 700 MHz)
O
O
Ph
N
Ph
1.07
2.01
1.17
6.00
1.12
2.04
1.01
1.02
3.08
3.00
f1 (ppm)

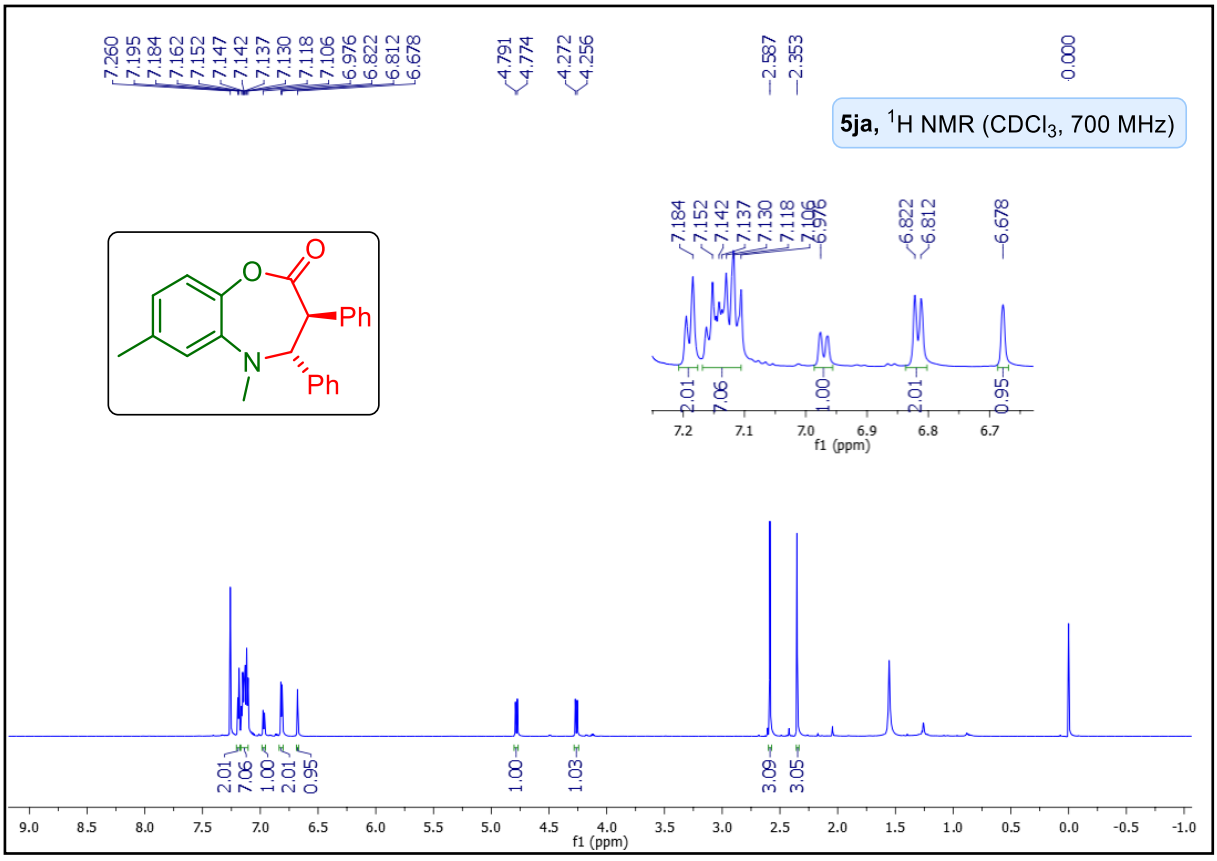

168.719
146.363
138.673
137.479
133.826
133.804
131.053
129.373
128.877
128.305
127.883
127.763
127.274
120.798
120.117
79.153
77.541
77.360
77.179
50.825
39.226
20.974
5ia', 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

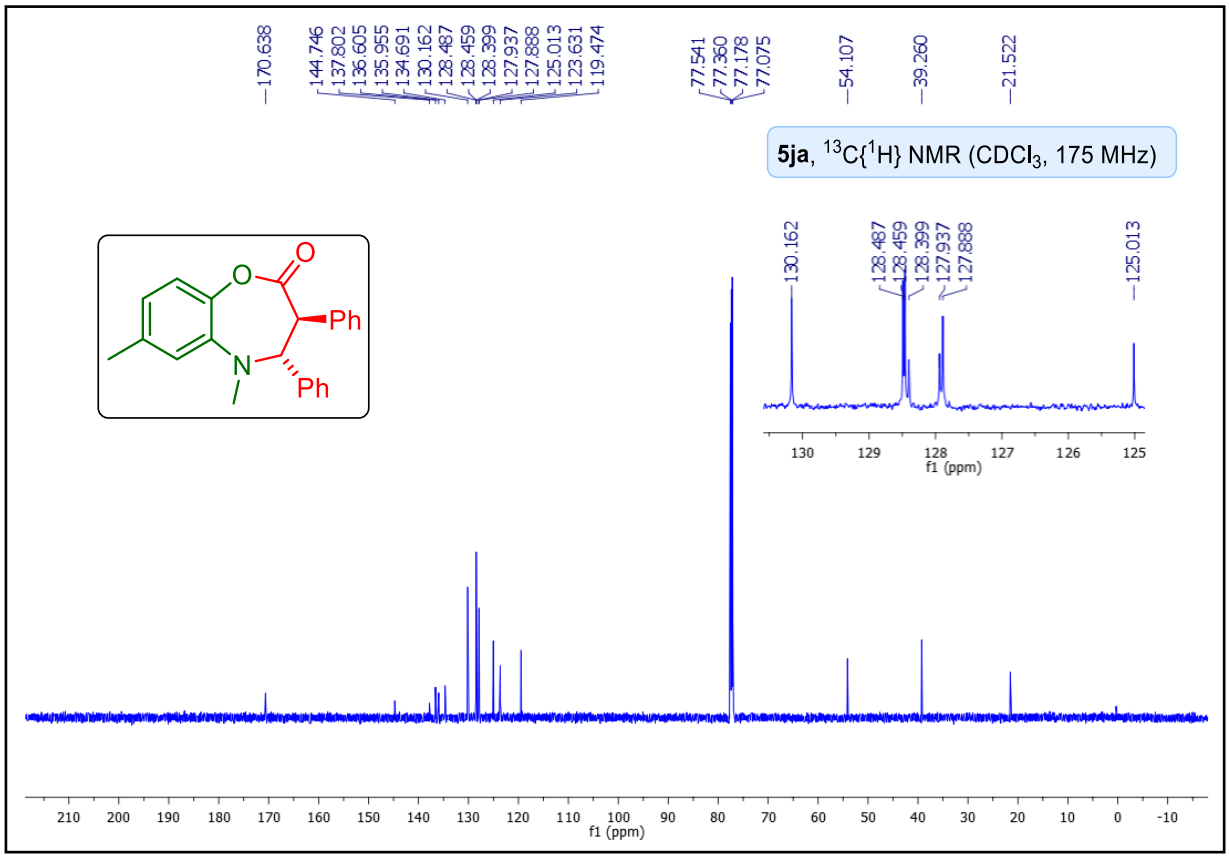

5ia, ^{1}H NMR ($CDCl_3$, 700 MHz)
Ph
Ph
f1 (ppm)

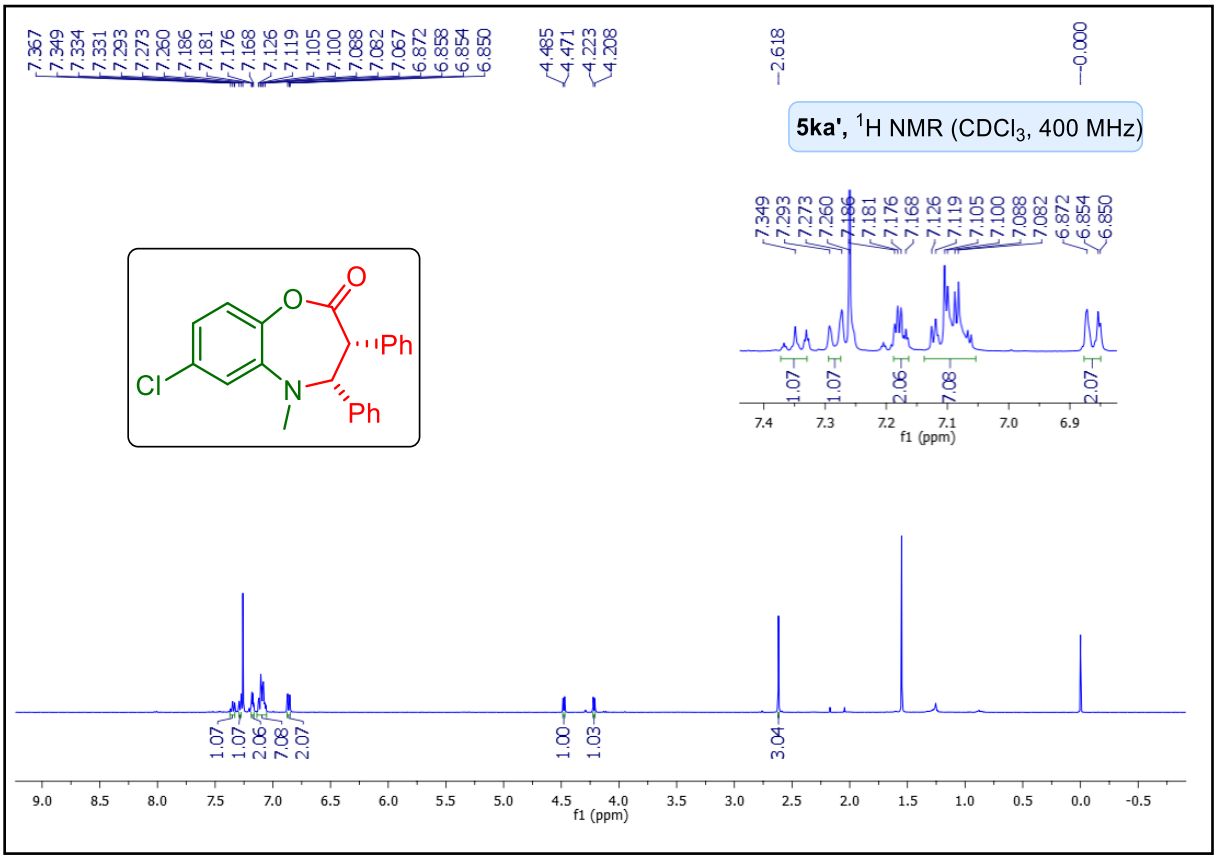

5ia, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
Ph
Ph
f1 (ppm)

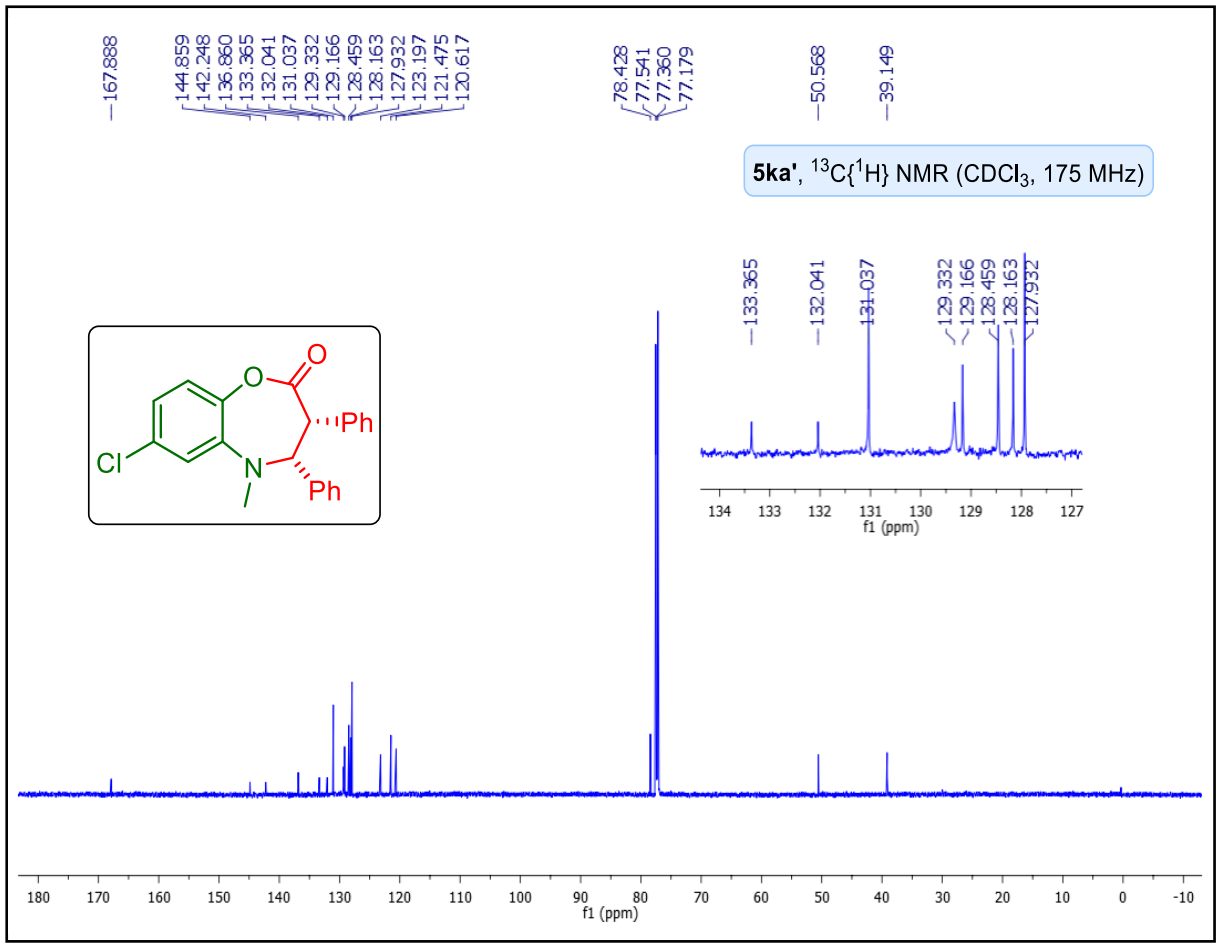

5ja', ^{1}H NMR ($CDCl_3$, 700 MHz)
f1 (ppm)

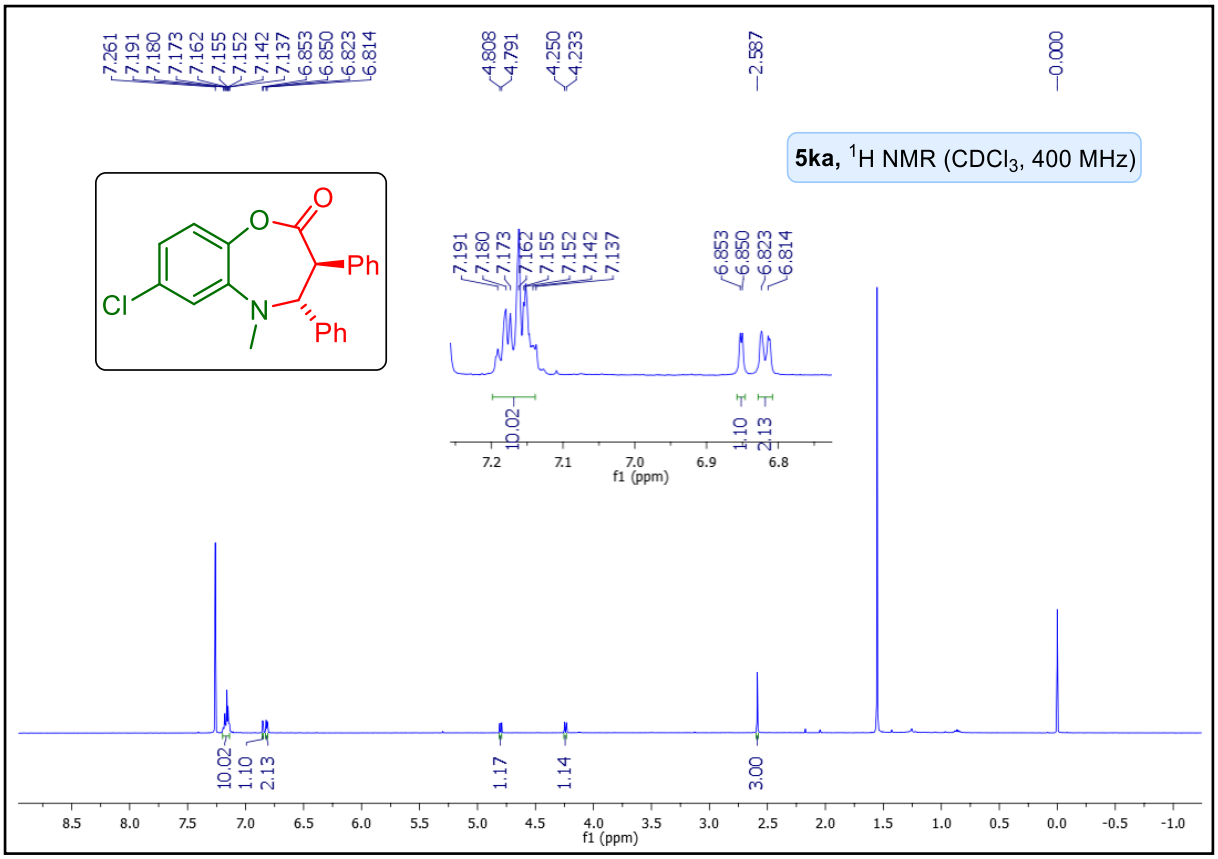

5ja', $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
f1 (ppm)

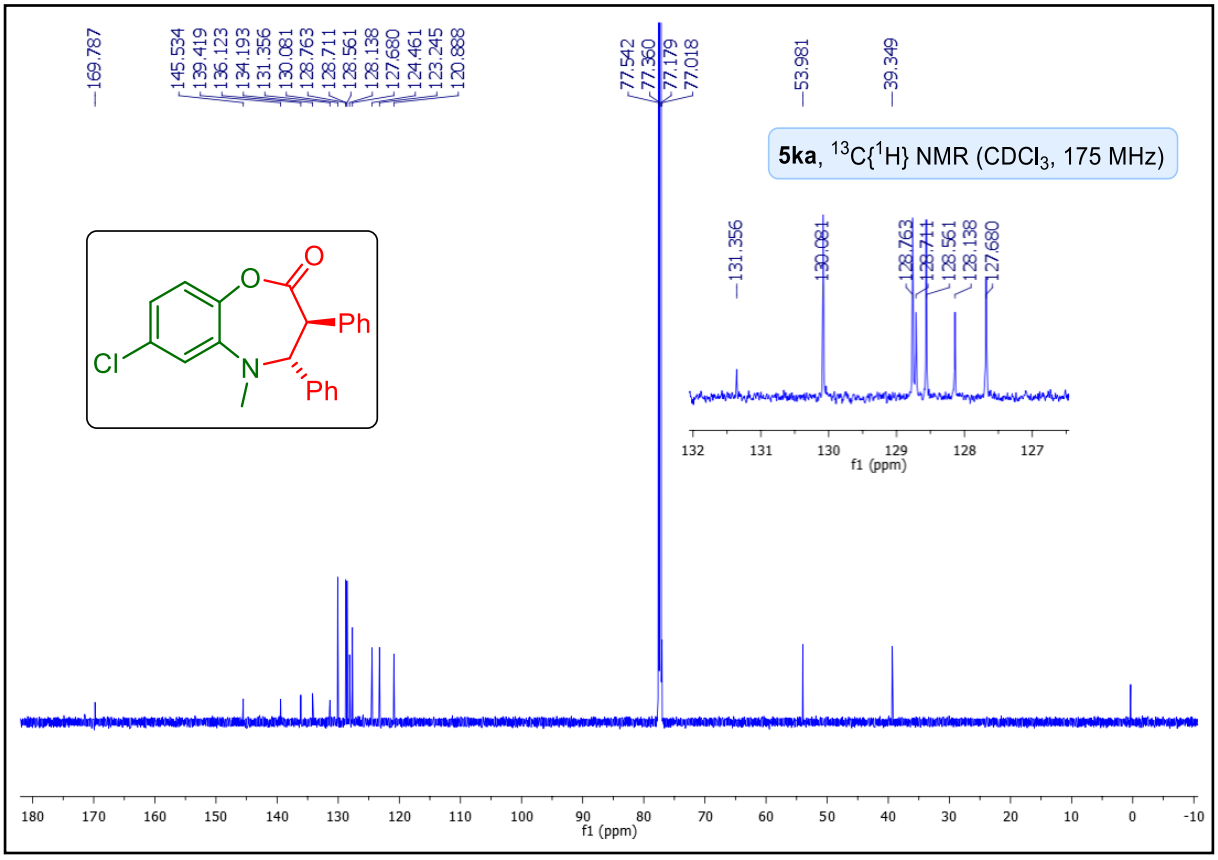

7.260
7.195
7.184
7.162
7.152
7.147
7.142
7.137
7.130
7.118
7.106
6.976
6.822
6.812
6.678
4.791
4.774
4.272
4.256
2.587
2.353
0.000
5ja, 1H NMR (CDCl3, 700 MHz)
O
O
Ph
N
Ph
f1 (ppm)
f1 (ppm)

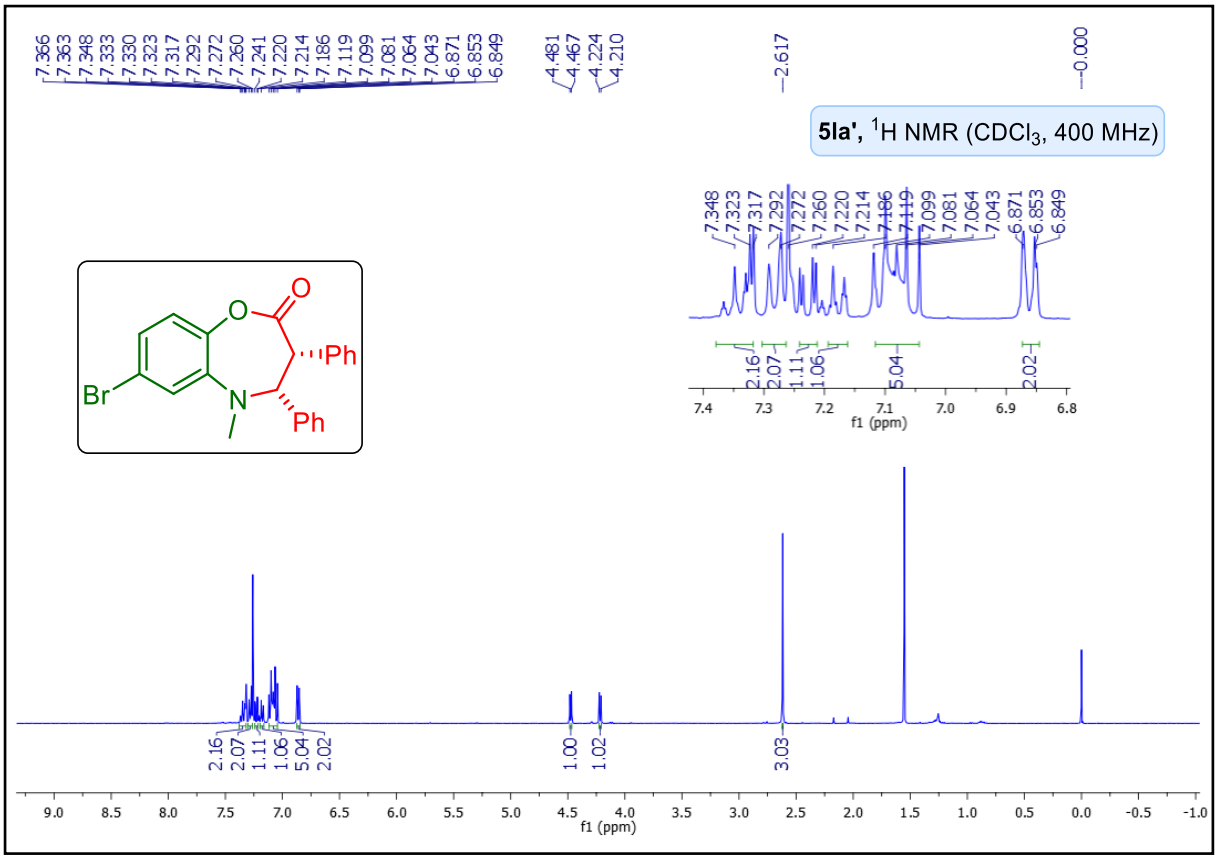

170.638
144.746
137.802
136.605
135.955
134.691
130.162
128.487
128.459
128.399
127.937
127.888
125.013
123.631
119.474
77.541
77.360
77.178
77.075
54.107
39.260
21.522
5ja, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)
f1 (ppm)

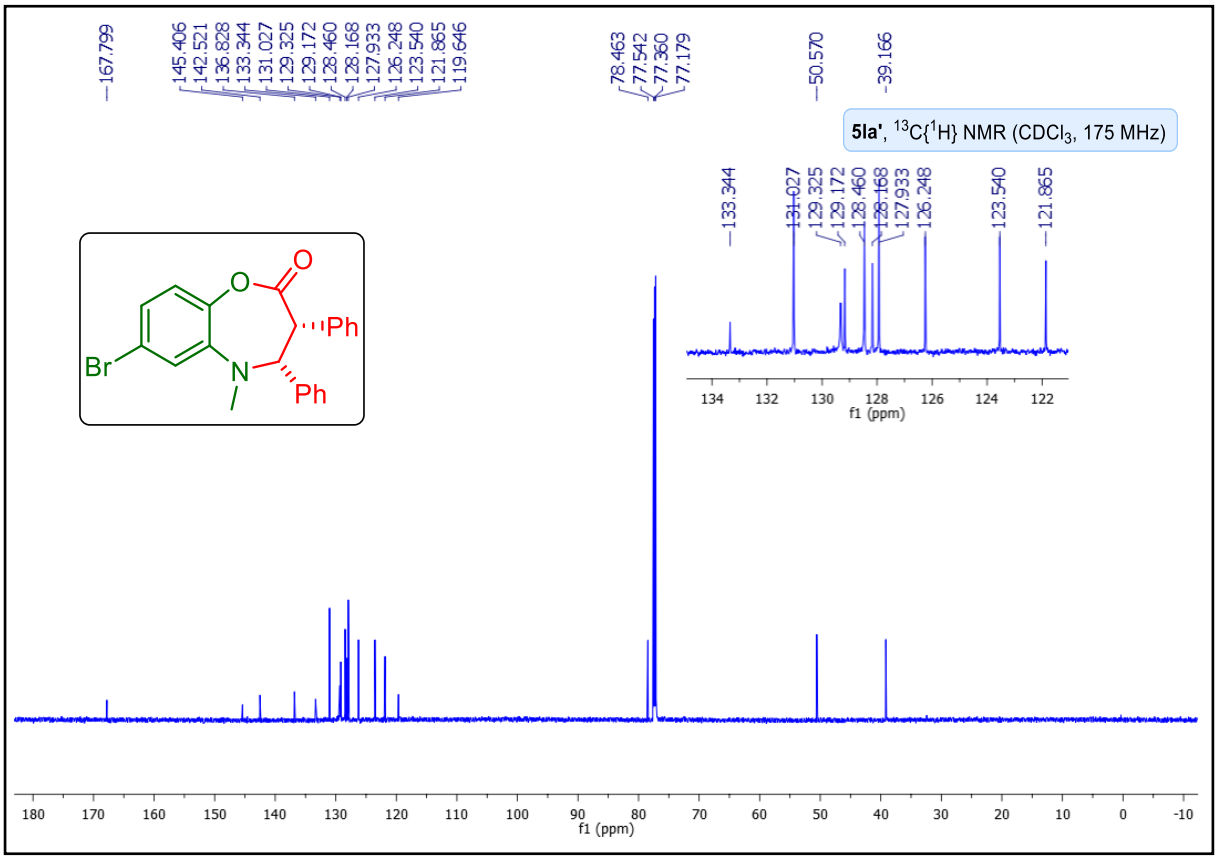

7.367
7.349
7.334
7.331
7.293
7.273
7.260
7.186
7.181
7.176
7.168
7.126
7.119
7.105
7.100
7.088
7.082
7.067
6.872
6.858
6.854
6.850
4.485
4.471
4.223
4.208
2.618
0.000
5ka', 1H NMR (CDCl3, 400 MHz)
Cl
N
O
O
Ph
Ph
1.07
1.07
2.06
7.08
2.07
1.00
1.03
3.04
f1 (ppm)

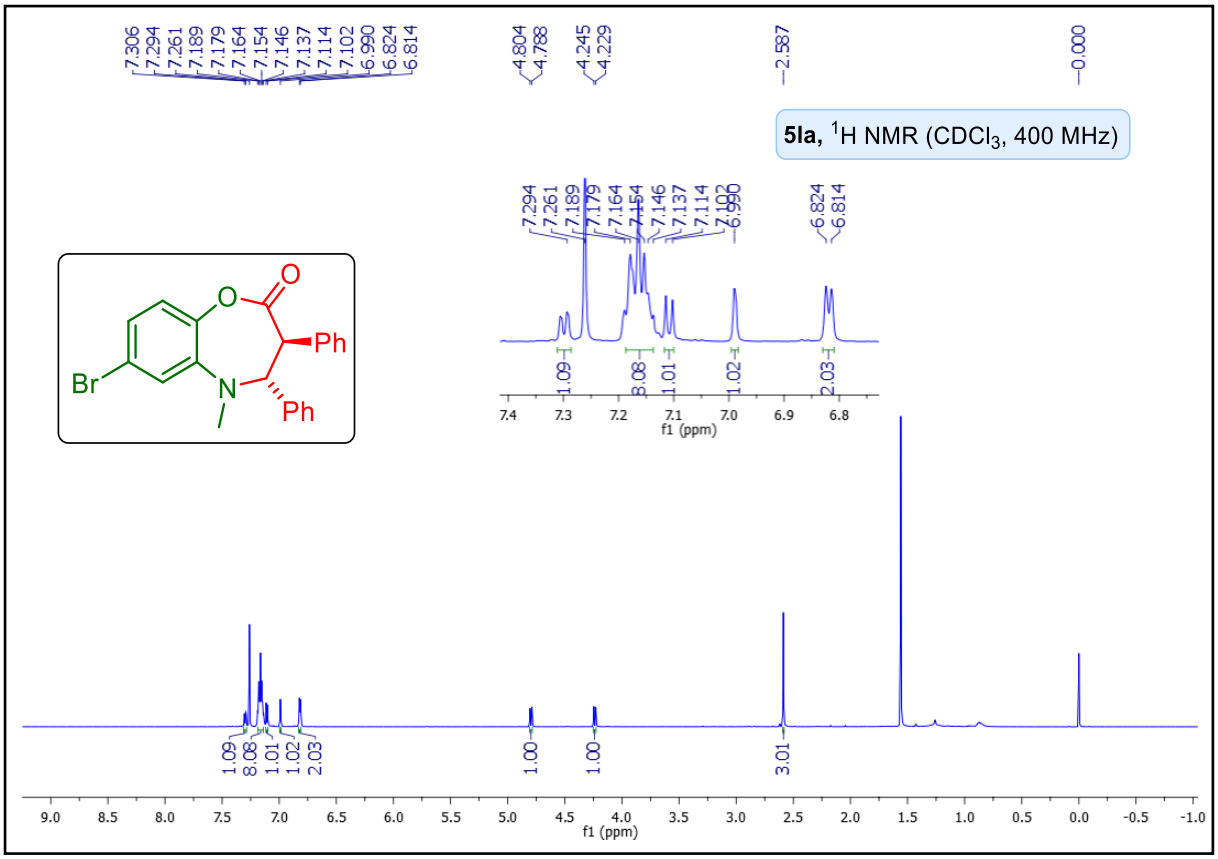

167.888
144.859
142.248
136.860
133.365
132.041
131.037
129.332
129.166
128.459
128.163
127.932
123.197
121.475
120.617
78.428
77.541
77.360
77.179
50.568
39.149
5ka', 13C{1H} NMR (CDCl3, 175 MHz)
Cl
N
O
O
Ph
Ph
f1 (ppm)

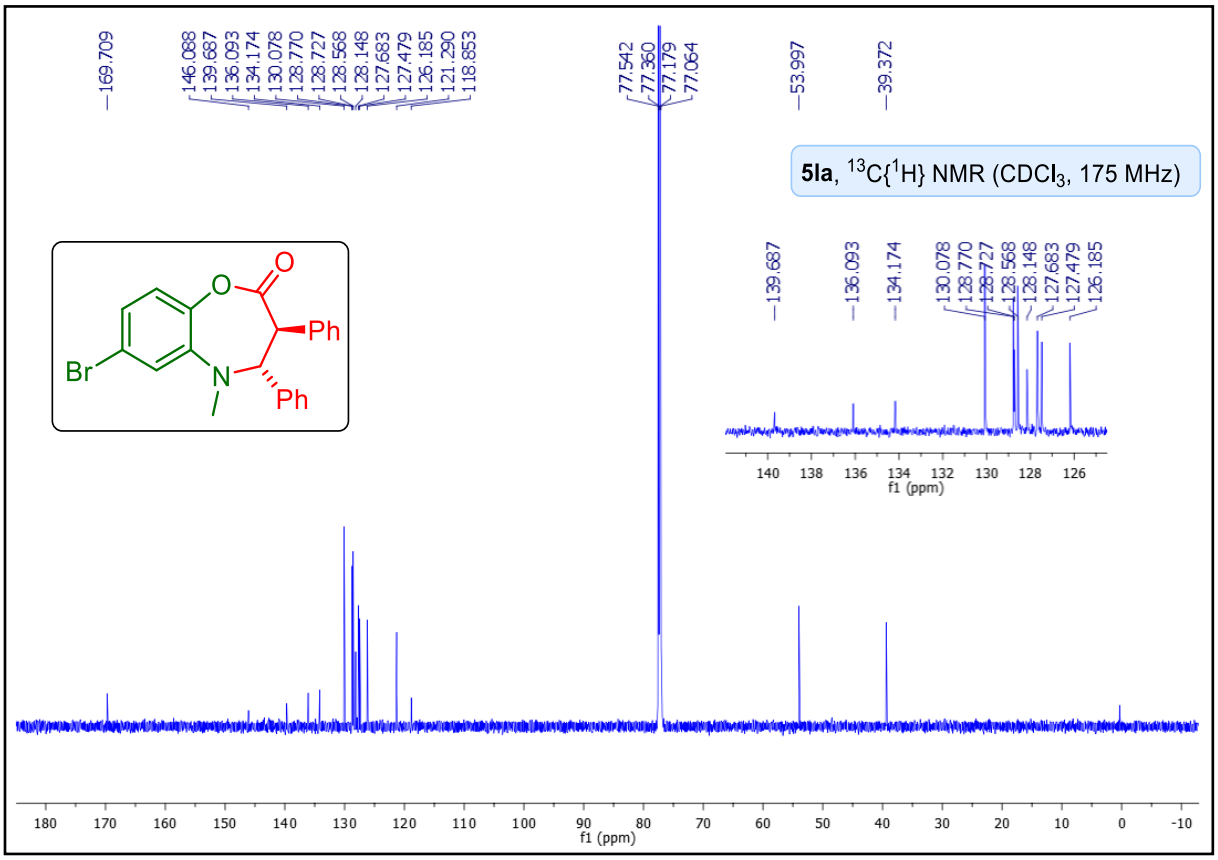

7.261
7.191
7.180
7.173
7.162
7.155
7.152
7.142
7.137
6.853
6.850
6.823
6.814
4.808
4.791
4.250
4.233
2.587
0.000
5ka, 1H NMR (CDCl3, 400 MHz)
Cl
O
O
N
Ph
Ph
10.02
1.10
2.13
1.17
1.14
3.00
f1 (ppm)

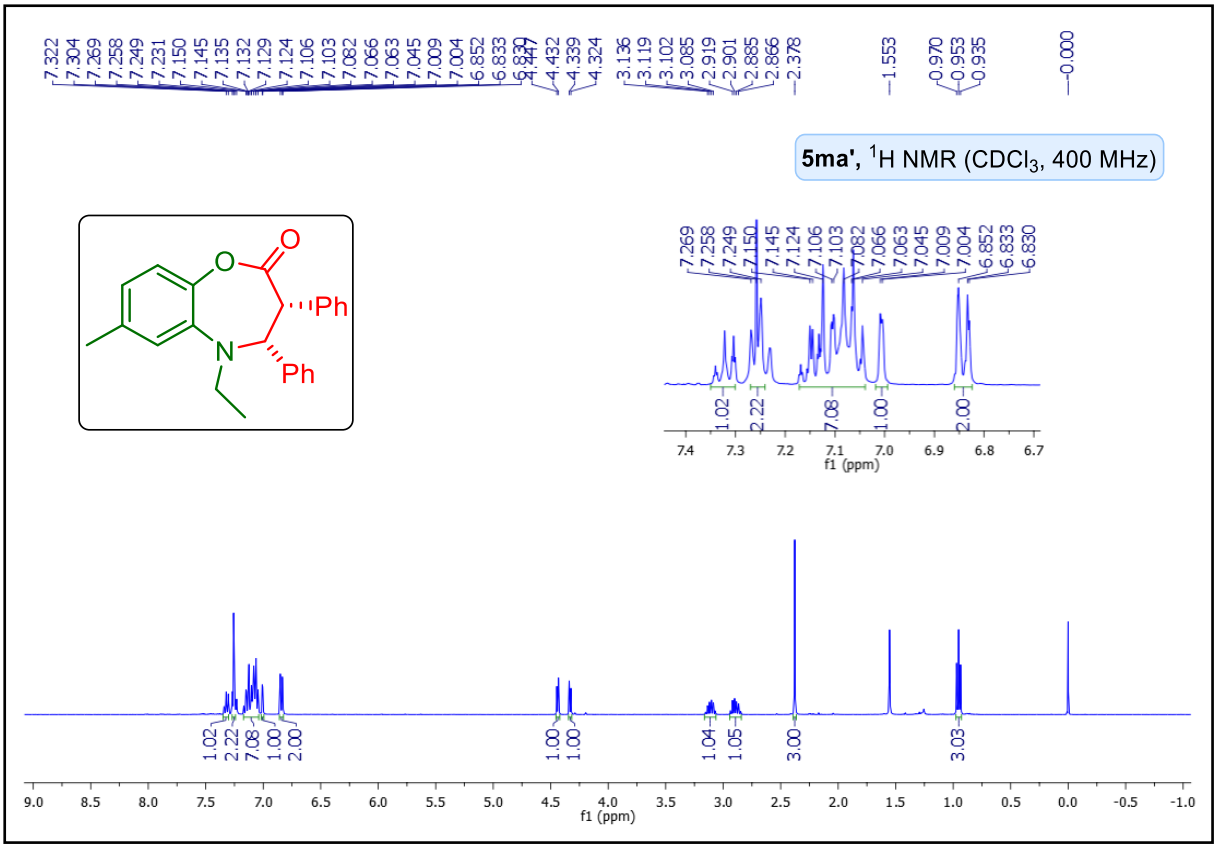

169.787
145.534
139.419
136.123
134.193
131.356
130.081
128.763
128.711
128.561
128.138
127.680
124.461
123.245
120.888
77.542
77.360
77.179
77.018
53.981
39.349
5ka, 13C{1H} NMR (CDCl3, 175 MHz)
Cl
O
O
N
Ph
Ph
f1 (ppm)

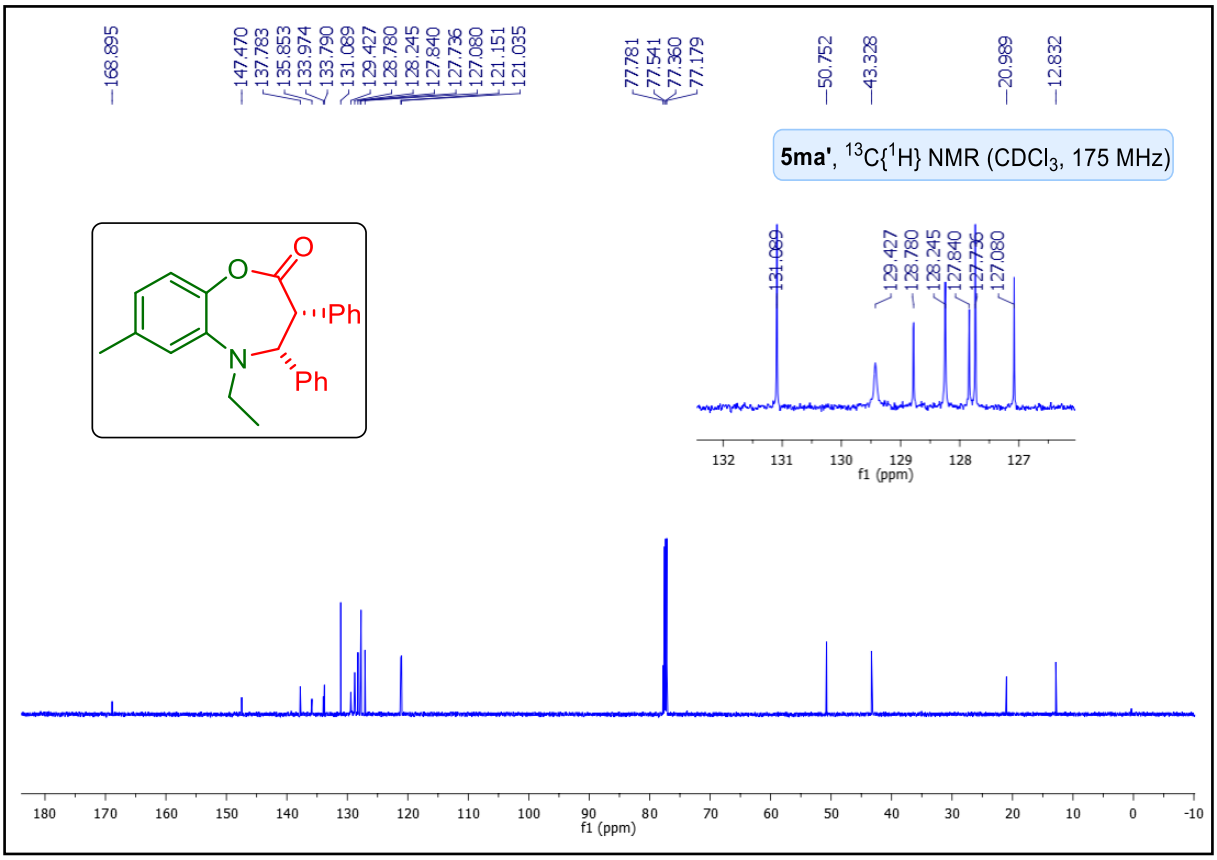

5la', ^{1}H NMR ($CDCl_3$, 400 MHz)
Br
N
O
Ph
Ph
f1 (ppm)

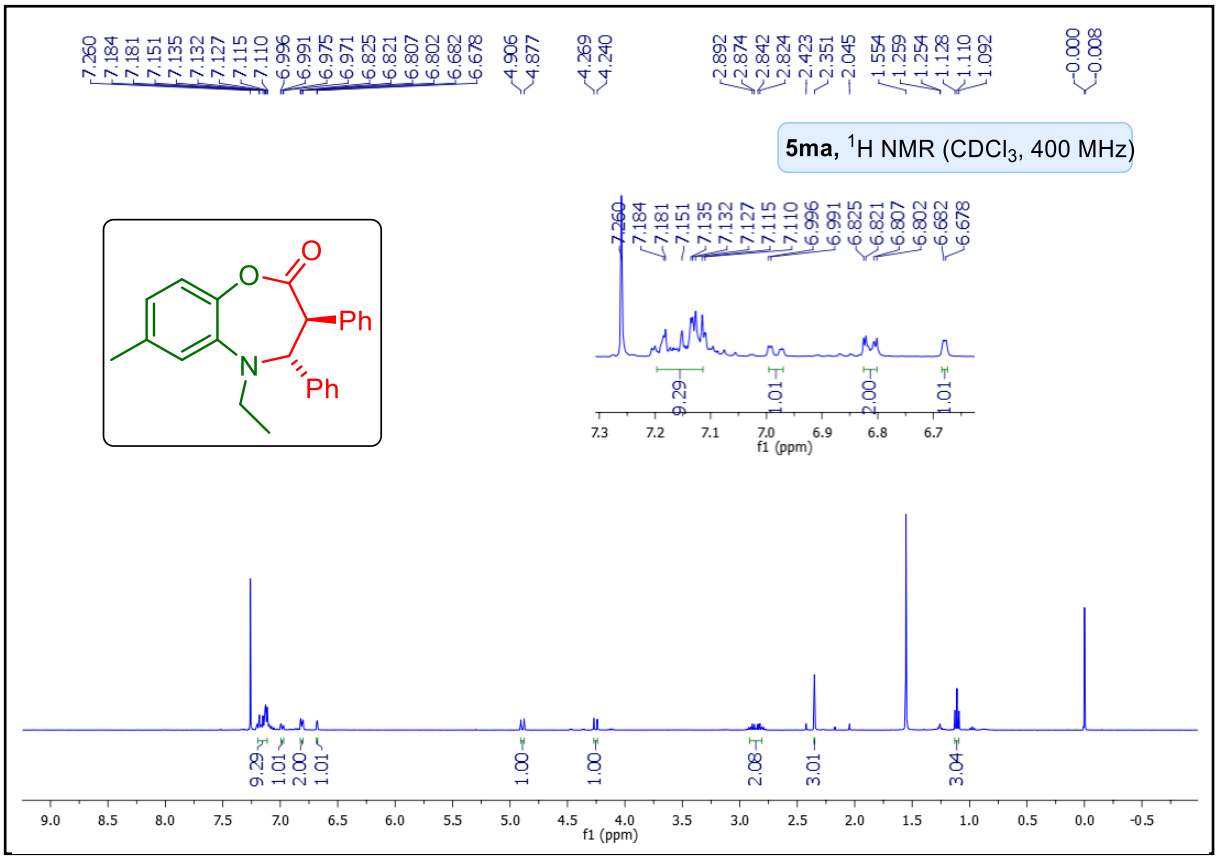

5la', $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
Br
N
O
Ph
Ph
f1 (ppm)

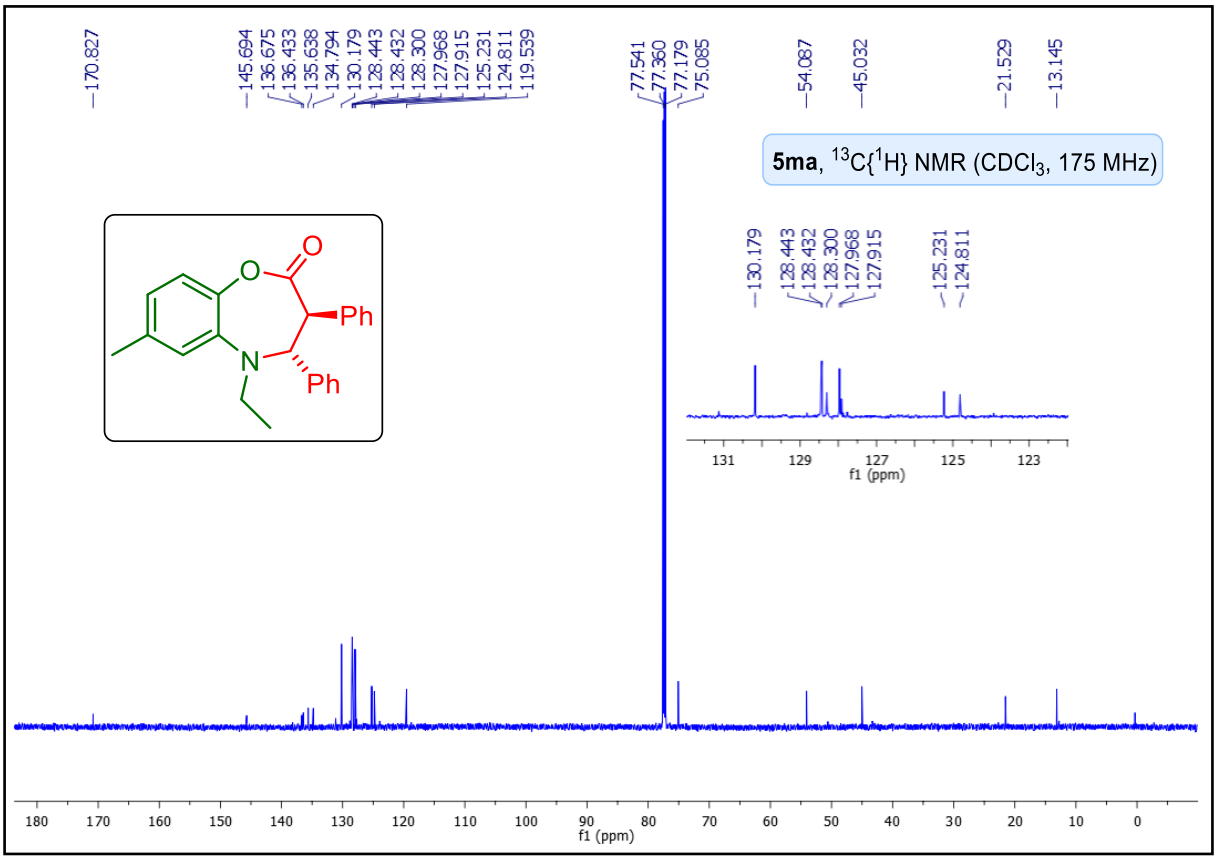

7.306
7.294
7.261
7.189
7.179
7.164
7.154
7.146
7.137
7.114
7.102
6.990
6.824
6.814
4.804
4.788
4.245
4.229
2.587
0.000
5la, 1H NMR (CDCl3, 400 MHz)
Br
N
O
O
Ph
Ph
1.09
8.08
1.01
1.02
2.03
1.00
1.00
3.01
f1 (ppm)

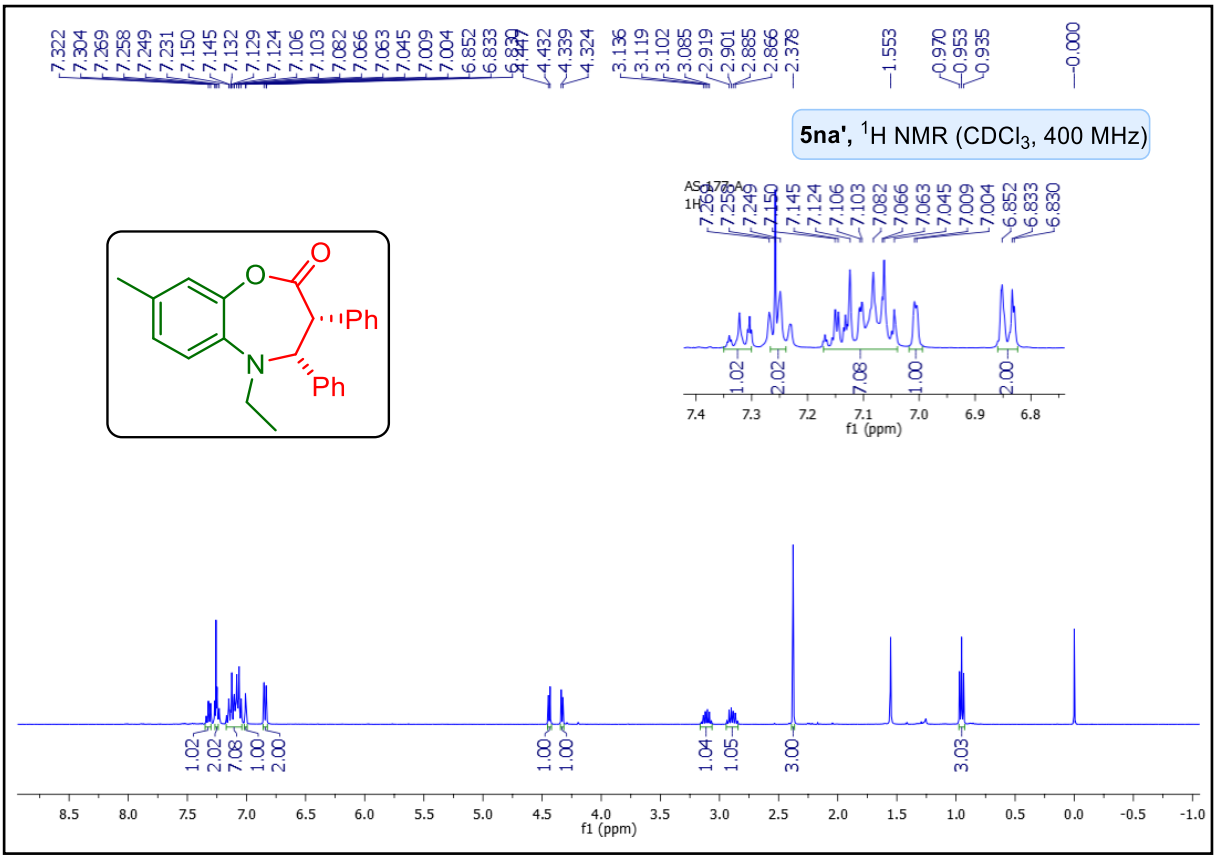

169.709
146.088
139.687
136.093
134.174
130.078
128.770
128.727
128.568
128.148
127.683
127.479
126.185
121.290
118.853
77.542
77.360
77.179
77.064
53.997
39.372
5la, 13C{1H} NMR (CDCl3, 175 MHz)
Br
N
O
O
Ph
Ph
f1 (ppm)

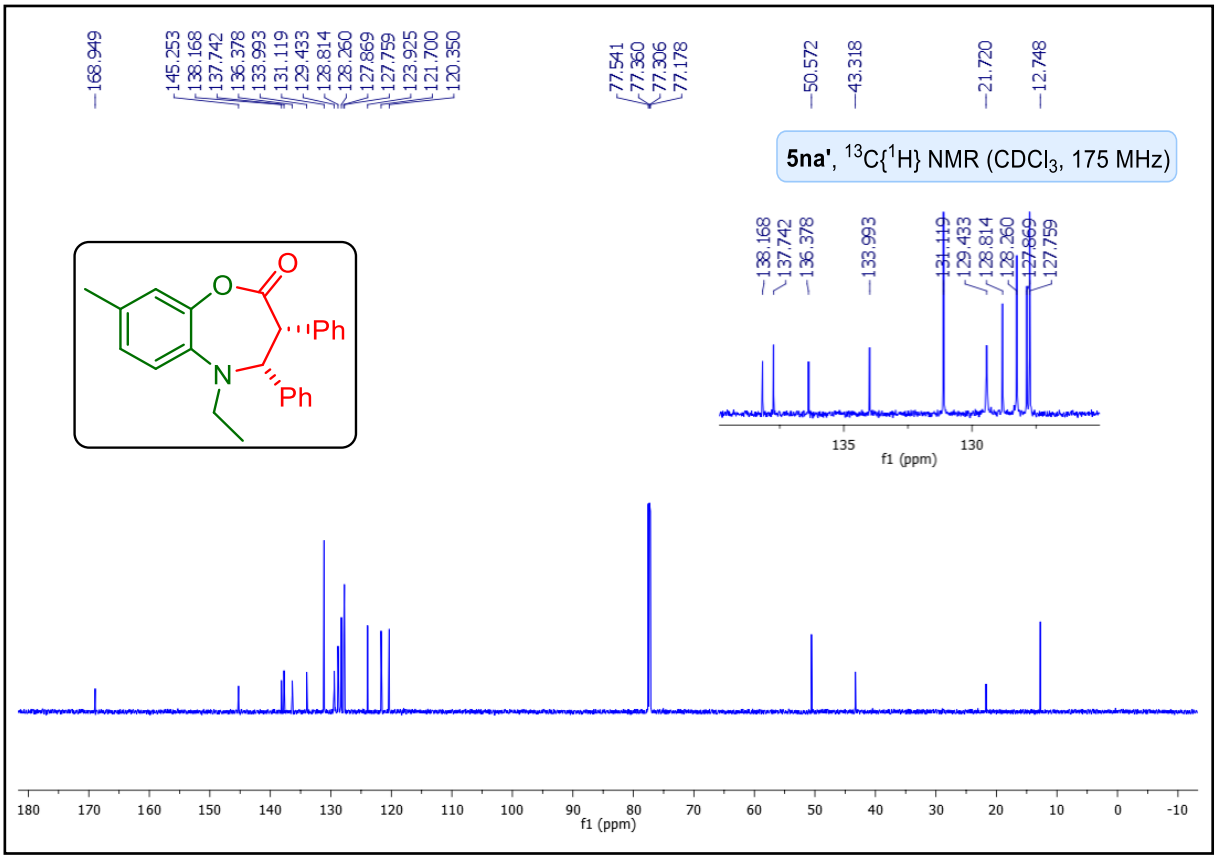

7.322
7.304
7.269
7.258
7.249
7.231
7.150
7.145
7.135
7.132
7.129
7.124
7.106
7.103
7.082
7.066
7.063
7.045
7.009
7.004
6.852
6.833
6.827
4.447
4.432
4.339
4.324
3.136
3.119
3.102
3.085
2.919
2.901
2.885
2.866
2.378
1.553
0.970
0.953
0.935
0.000
5ma', ^{1}H NMR ($CDCl_3$, 400 MHz)
O
O
Ph
N
Ph
7.269
7.258
7.249
7.150
7.145
7.124
7.106
7.103
7.082
7.066
7.063
7.045
7.009
7.004
6.852
6.833
6.830
1.02
2.22
7.08
1.00
2.00
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
f1 (ppm)
1.02
2.22
7.08
1.00
2.00
1.00
1.00
1.04
1.05
3.00
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

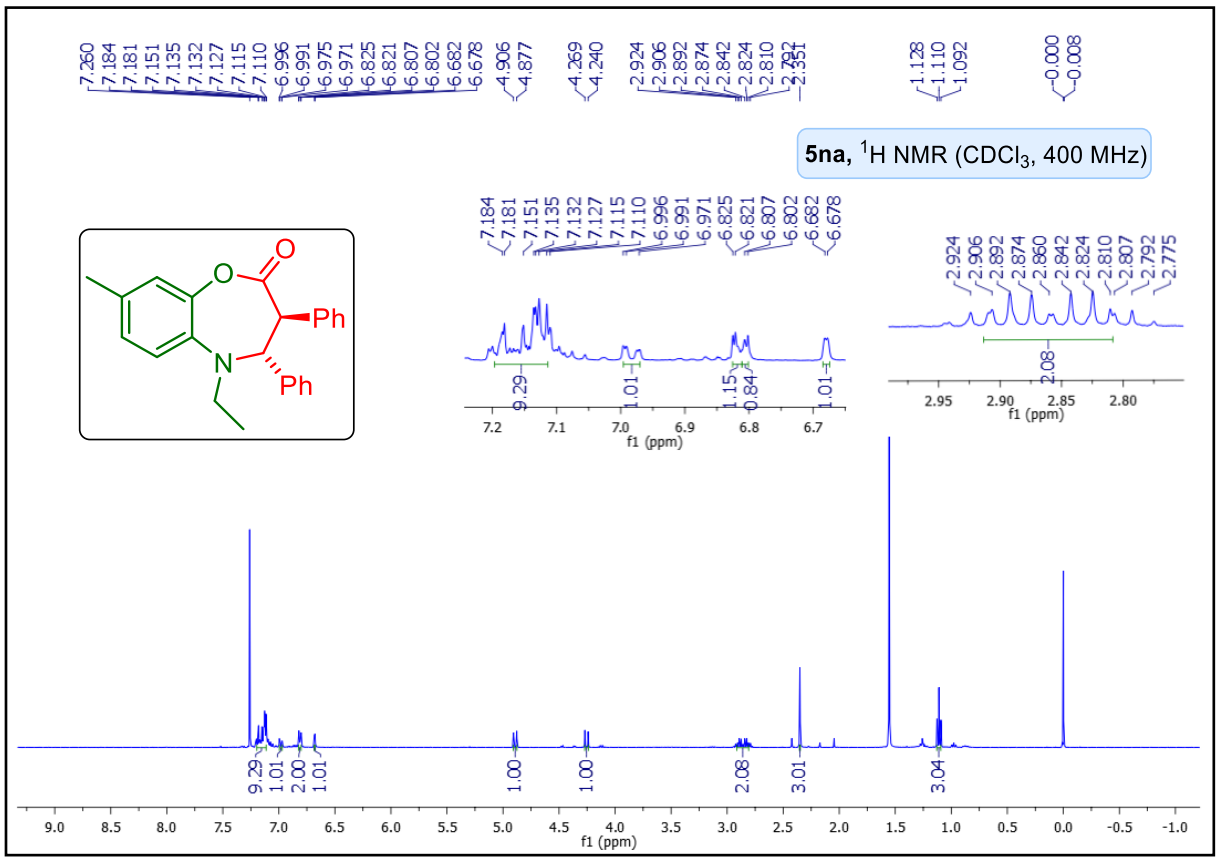

168.895
147.470
137.783
135.853
133.974
133.790
131.089
129.427
128.780
128.245
127.840
127.736
127.080
121.151
121.035
77.781
77.541
77.360
77.179
50.752
43.328
20.989
12.832
5ma', ^{13}C{^{1}H} NMR ($CDCl_3$, 175 MHz)
O
O
Ph
N
Ph
131.089
129.427
128.780
128.245
127.840
127.736
127.080
132
131
130
129
128
127
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

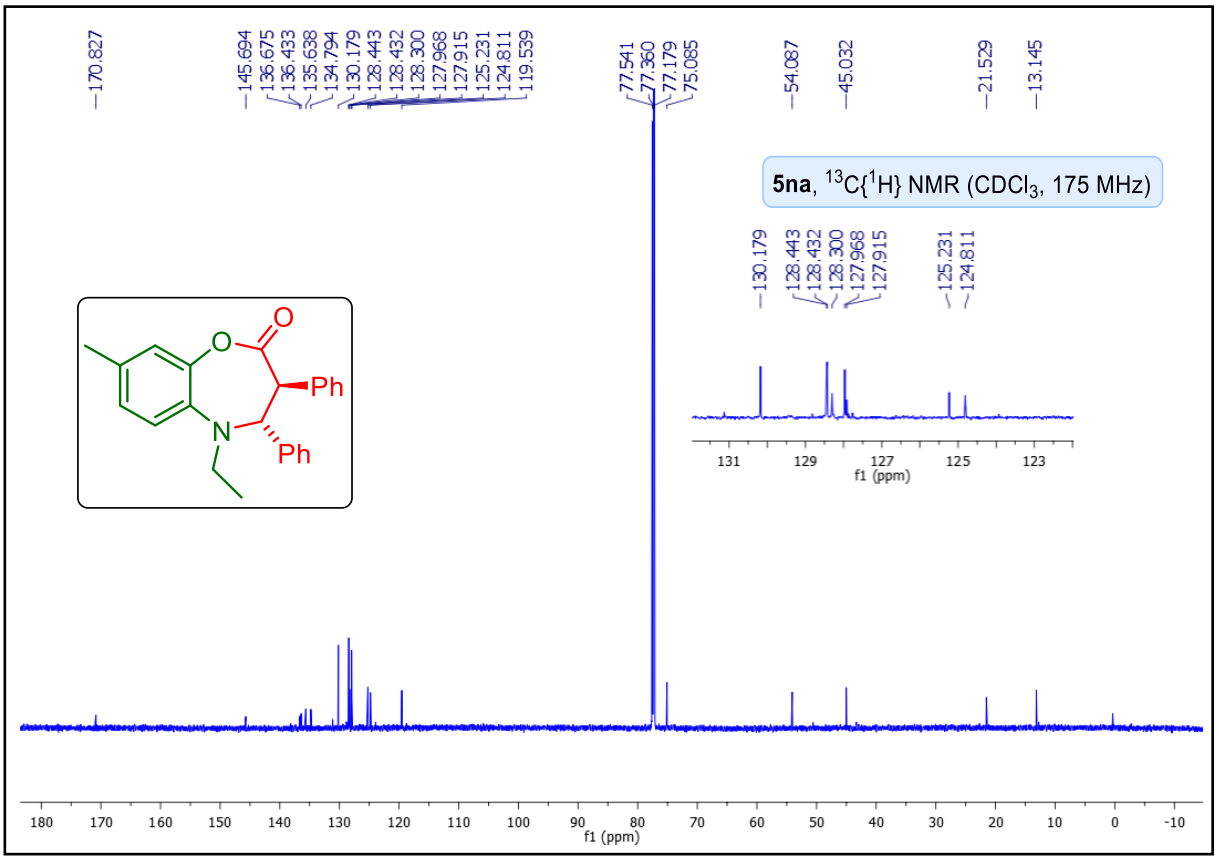

7.260
7.184
7.181
7.151
7.135
7.132
7.127
7.115
7.110
6.996
6.991
6.975
6.971
6.825
6.821
6.807
6.802
6.682
6.678
4.906
4.877
4.269
4.240
2.892
2.874
2.842
2.824
2.423
2.351
2.045
1.554
1.259
1.254
1.128
1.110
1.092
0.000
0.008
5ma, 1H NMR (CDCl3, 400 MHz)
O
O
Ph
N
Ph
9.29
1.01
2.00
1.01
1.00
1.00
2.08
3.01
3.04
f1 (ppm)

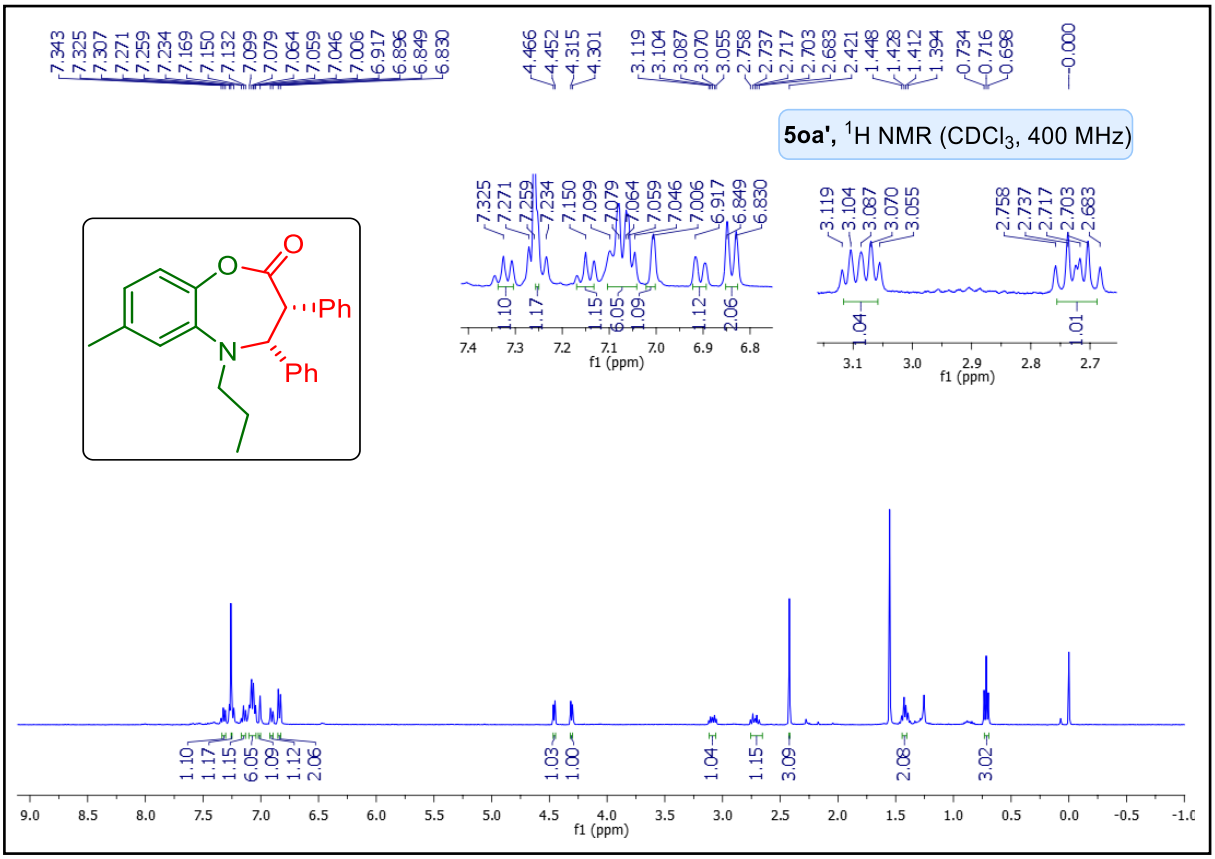

170.827
145.694
136.675
136.433
135.638
134.794
130.179
128.443
128.432
128.300
127.968
127.915
125.231
124.811
119.539
77.541
77.360
77.179
75.085
54.087
45.032
21.529
13.145
5ma, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

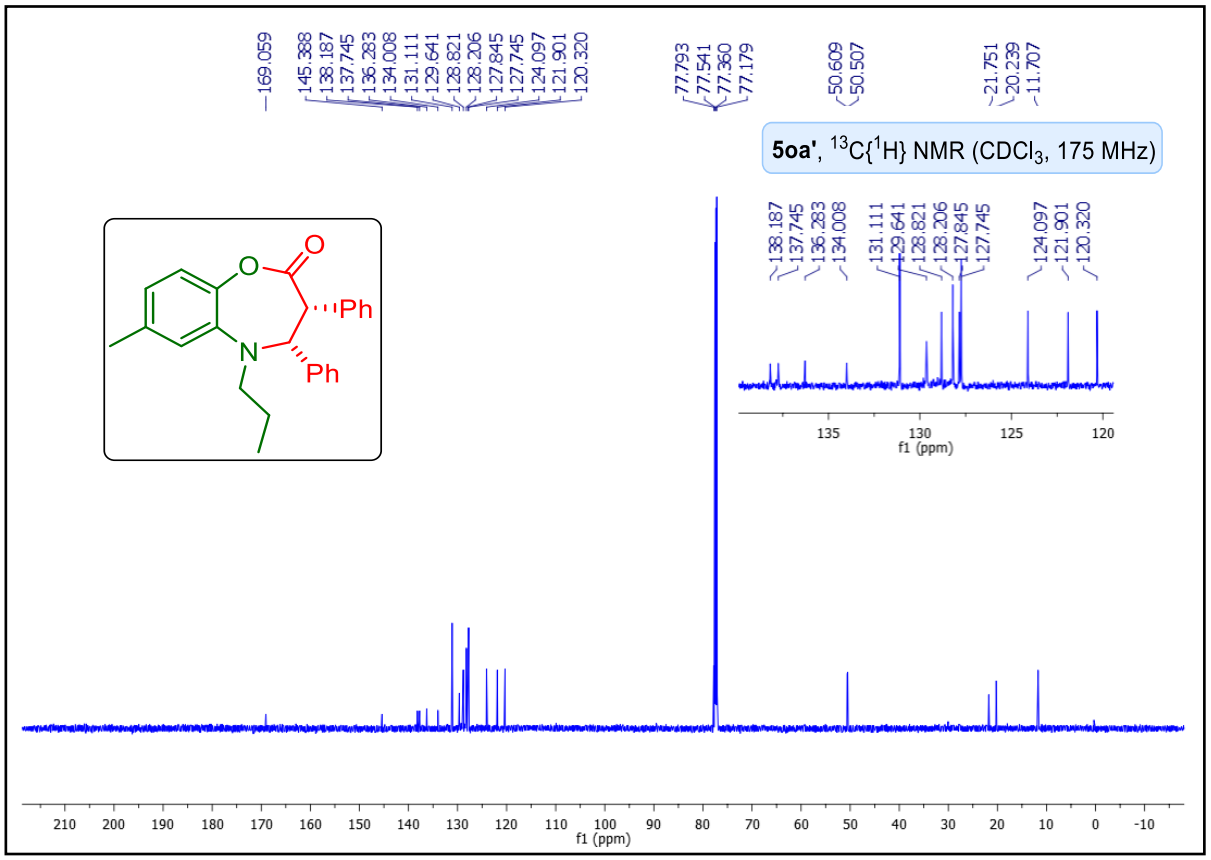

7.322
7.304
7.269
7.258
7.249
7.231
7.150
7.145
7.132
7.129
7.124
7.106
7.103
7.082
7.066
7.063
7.045
7.009
7.004
6.852
6.833
6.830
4.447
4.432
4.339
4.324
3.136
3.119
3.102
3.085
2.919
2.901
2.885
2.866
2.378
1.553
0.970
0.953
0.935
0.000
5na', 1H NMR (CDCl3, 400 MHz)
O
O
Ph
N
Ph
1.02
2.02
7.08
1.00
2.00
1.00
1.00
1.04
1.05
3.00
3.03
f1 (ppm)

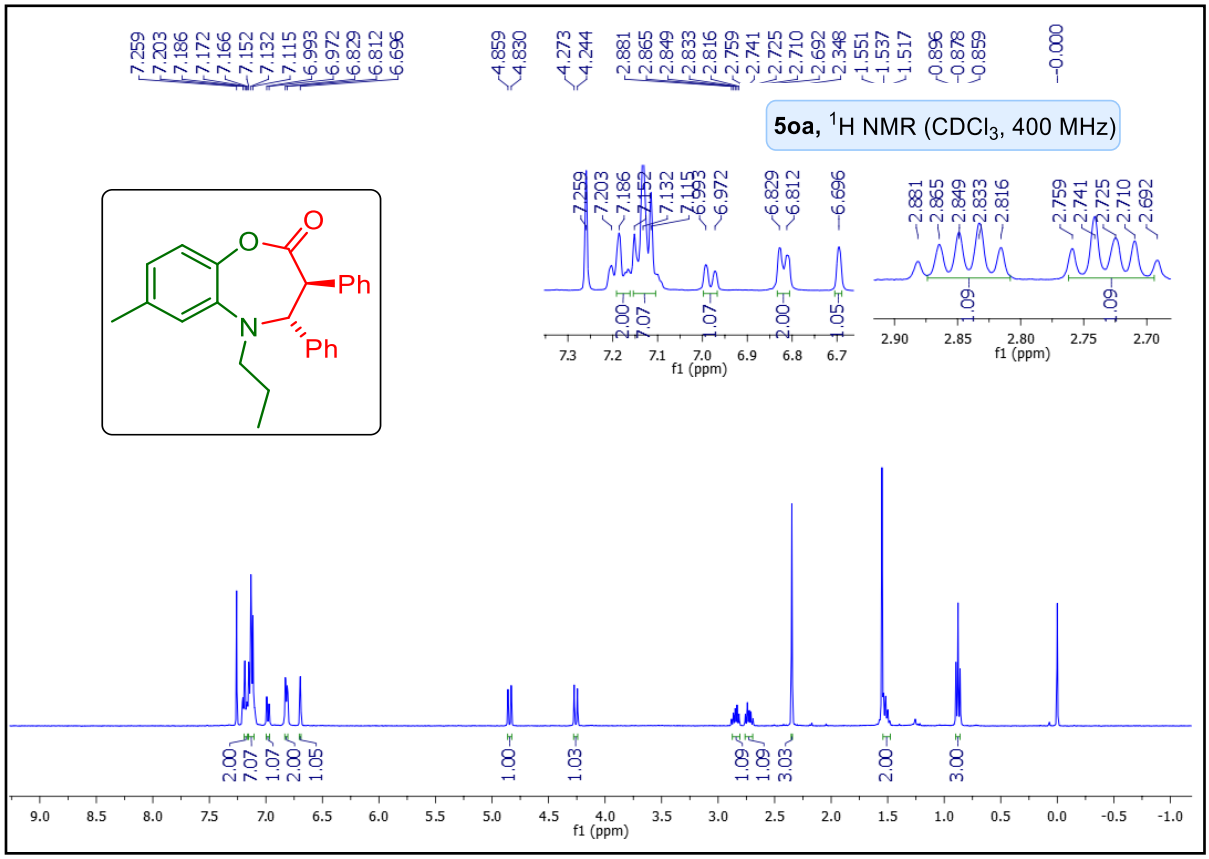

168.949
145.253
138.168
137.742
136.378
133.993
131.119
129.433
128.814
128.260
127.869
127.759
123.925
121.700
120.350
77.541
77.360
77.306
77.178
50.572
43.318
21.720
12.748
5na', 13C{1H} NMR (CDCl3, 175 MHz)
O
O
Ph
N
Ph
f1 (ppm)

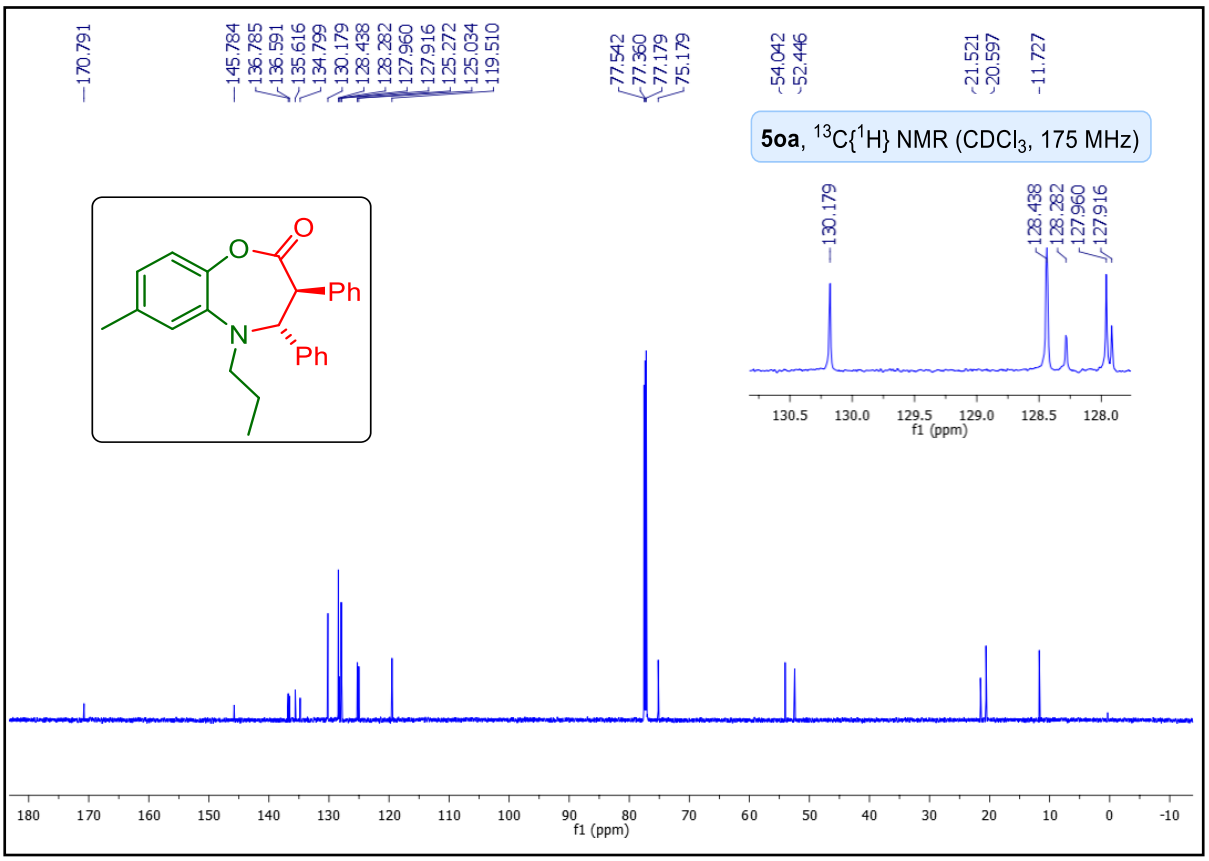

5na, ^{1}H NMR (CDCl$_3$, 400 MHz)
Ph
Ph
f1 (ppm)

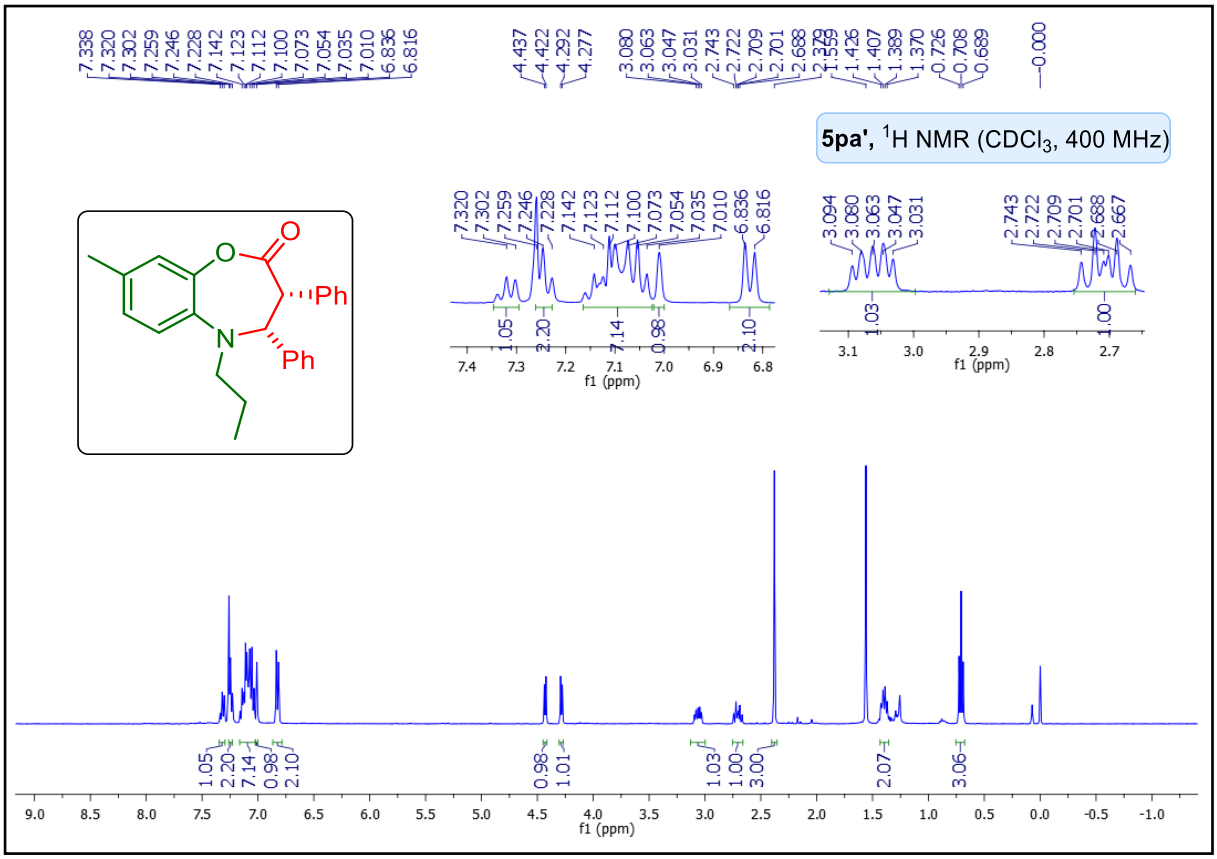

5na, ^{13}C{^{1}H} NMR (CDCl$_3$, 175 MHz)
Ph
Ph
f1 (ppm)

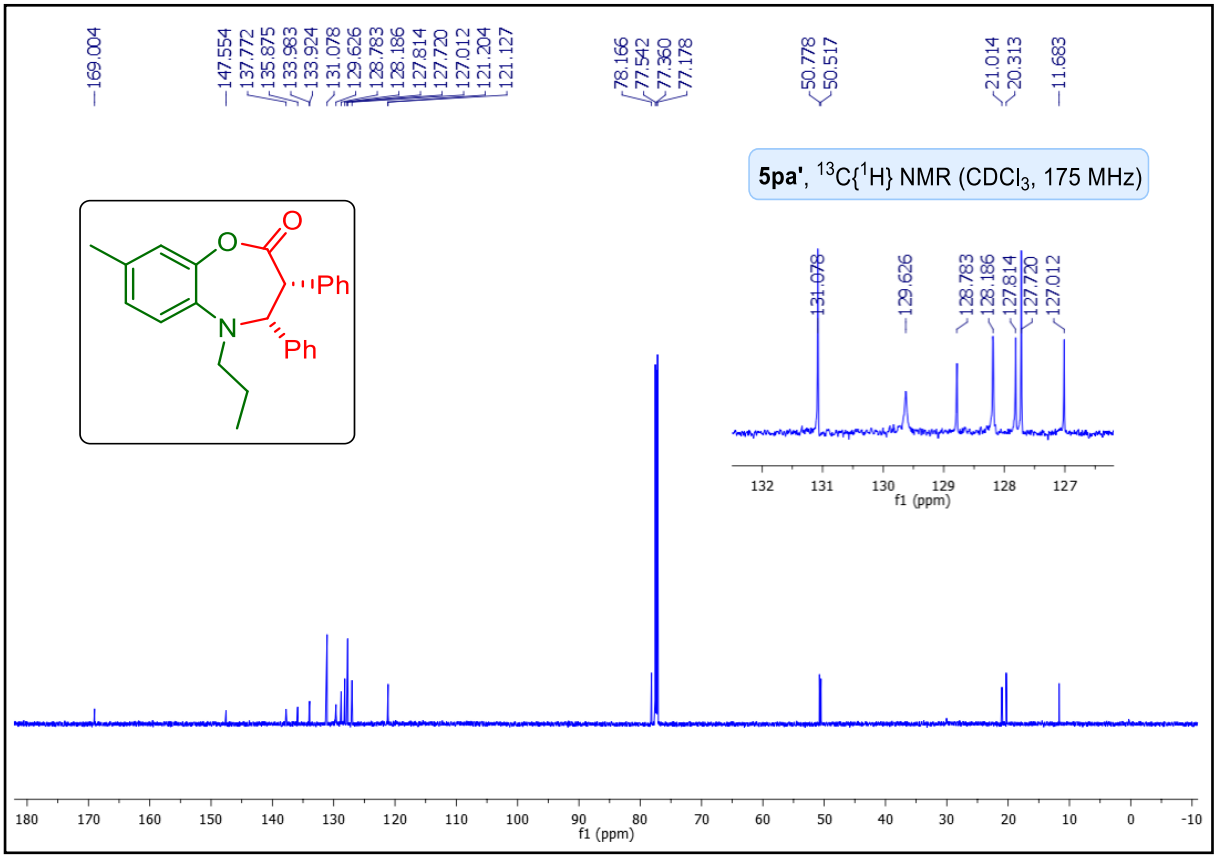

5oa', ^{1}H NMR ($CDCl_3$, 400 MHz)
Ph
Ph
f1 (ppm)

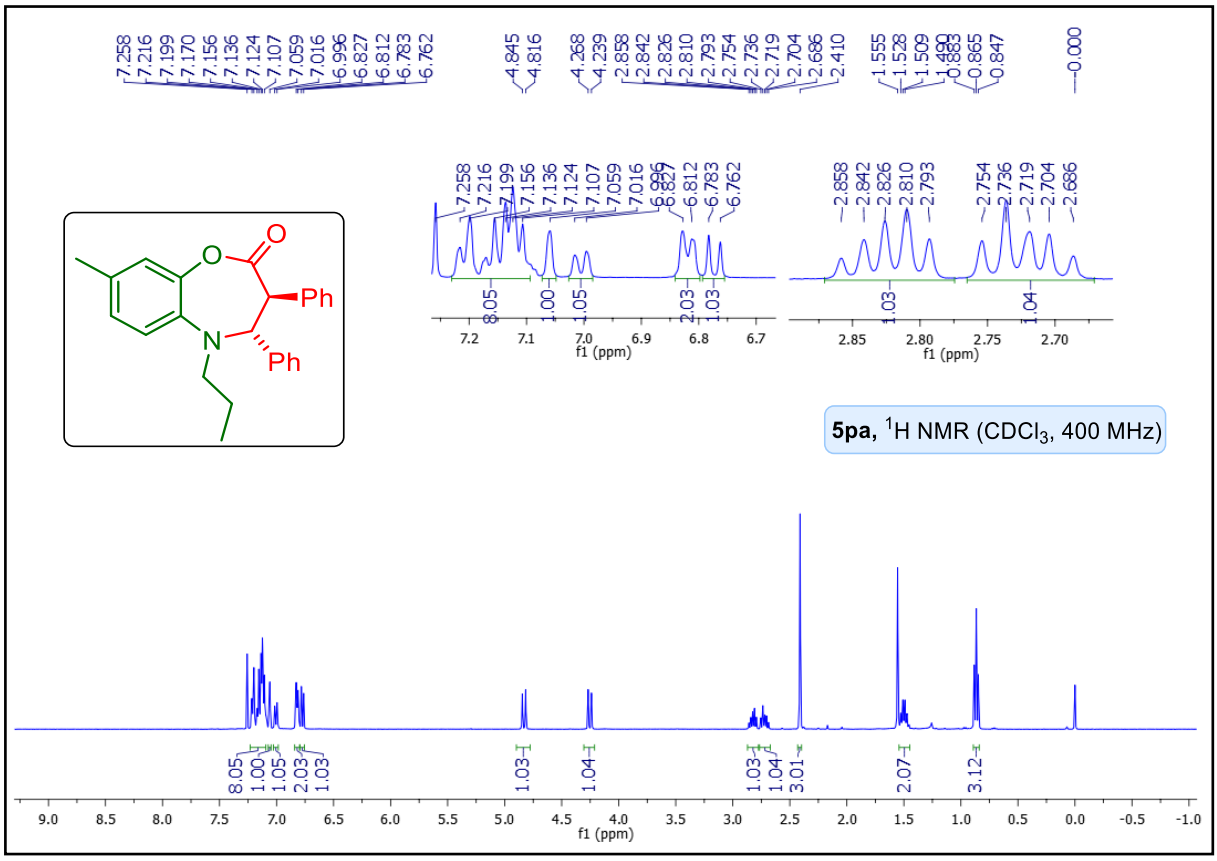

5oa', $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
Ph
Ph
f1 (ppm)

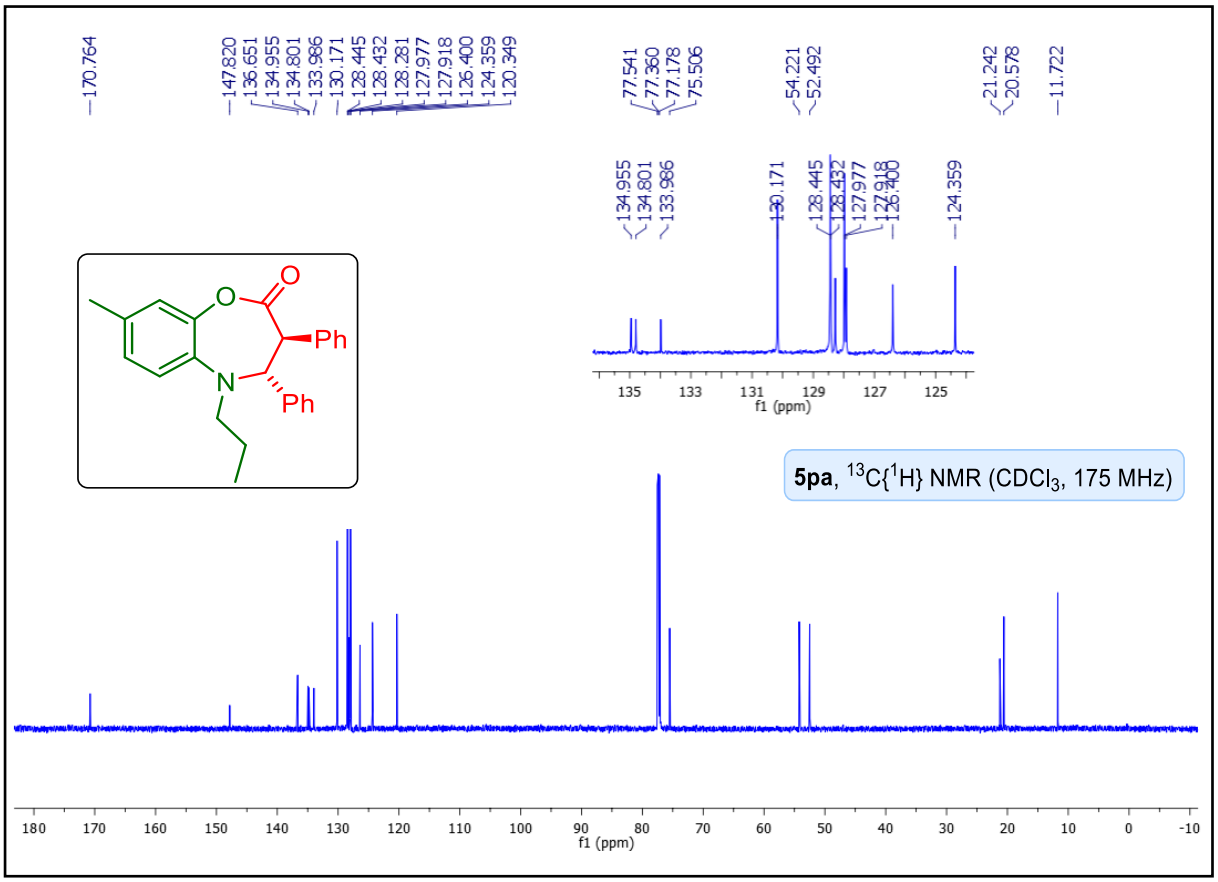

5oa, ^{1}H NMR ($CDCl_3$, 400 MHz)
Ph
Ph
f1 (ppm)

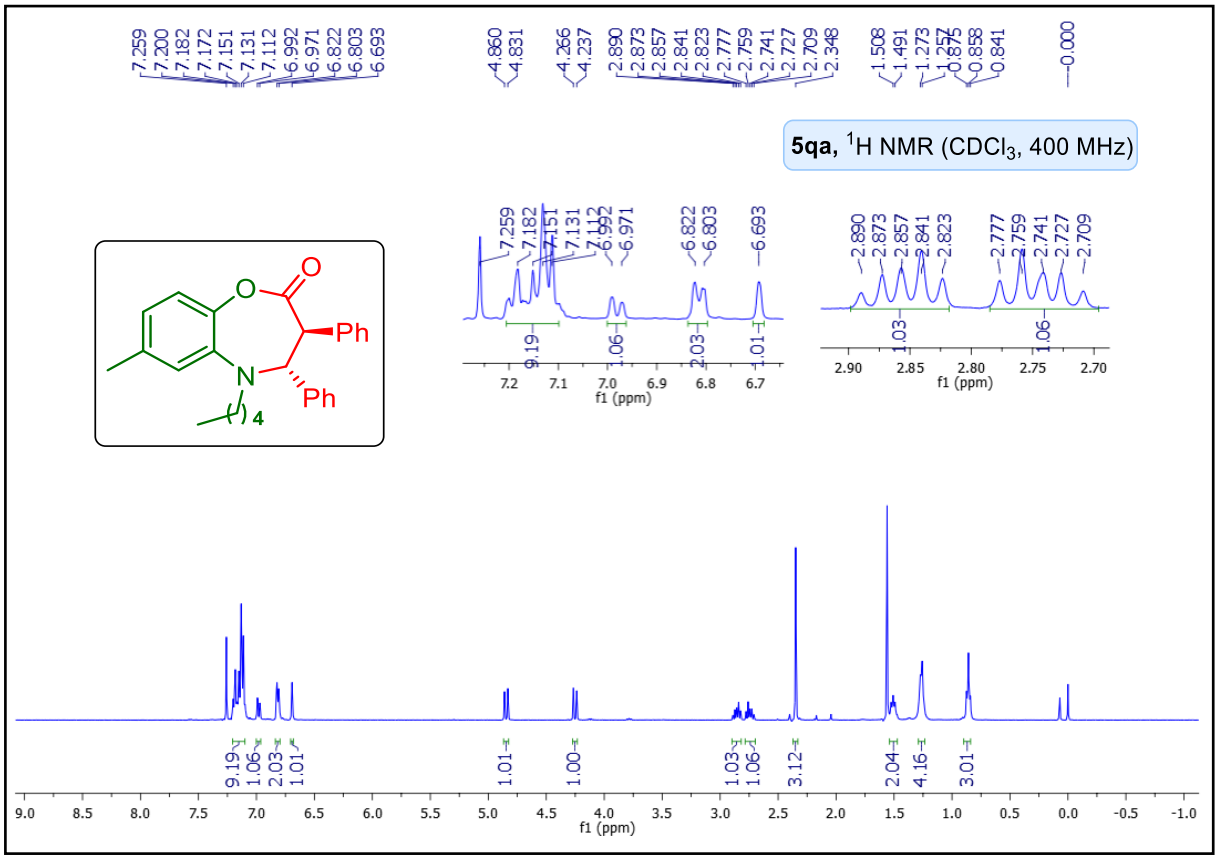

5oa, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
Ph
Ph
f1 (ppm)

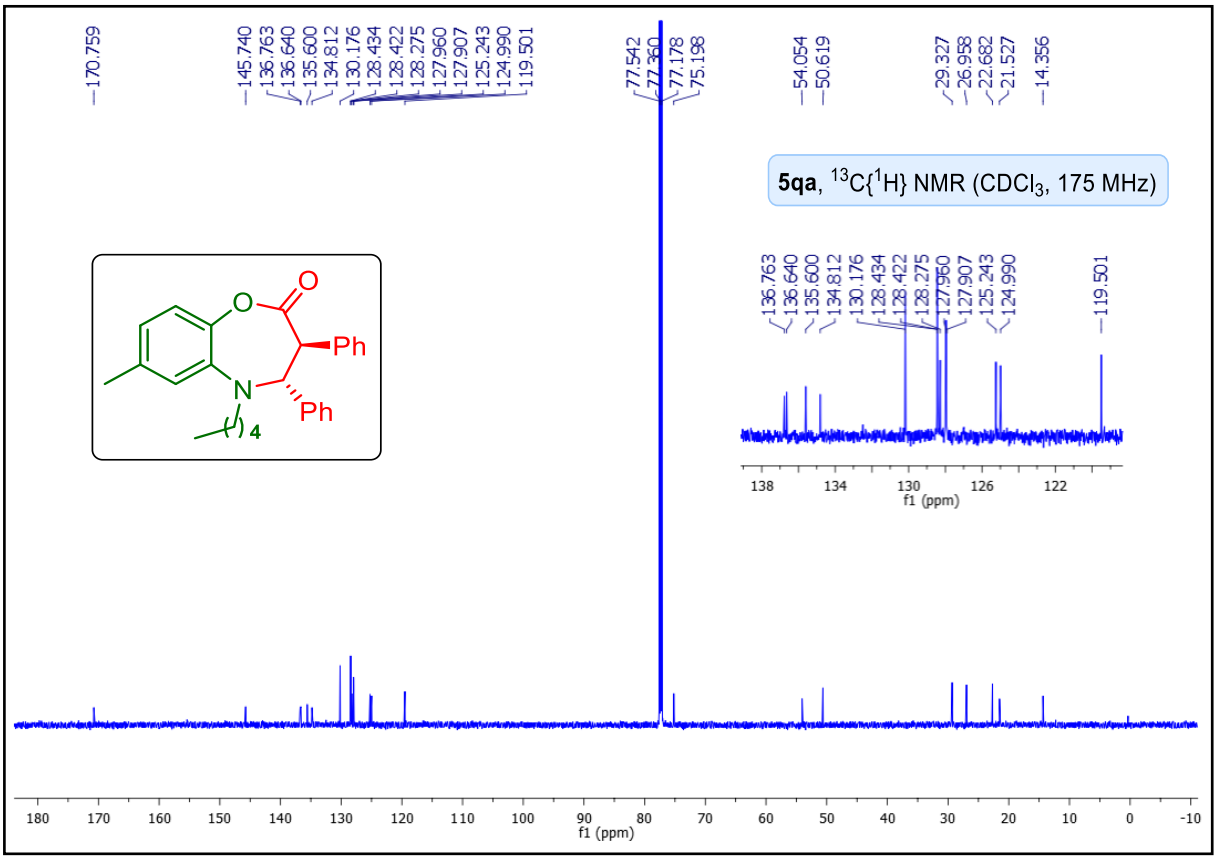

5pa', ^{1}H NMR ($CDCl_3$, 400 MHz)
Ph
Ph
f1 (ppm)

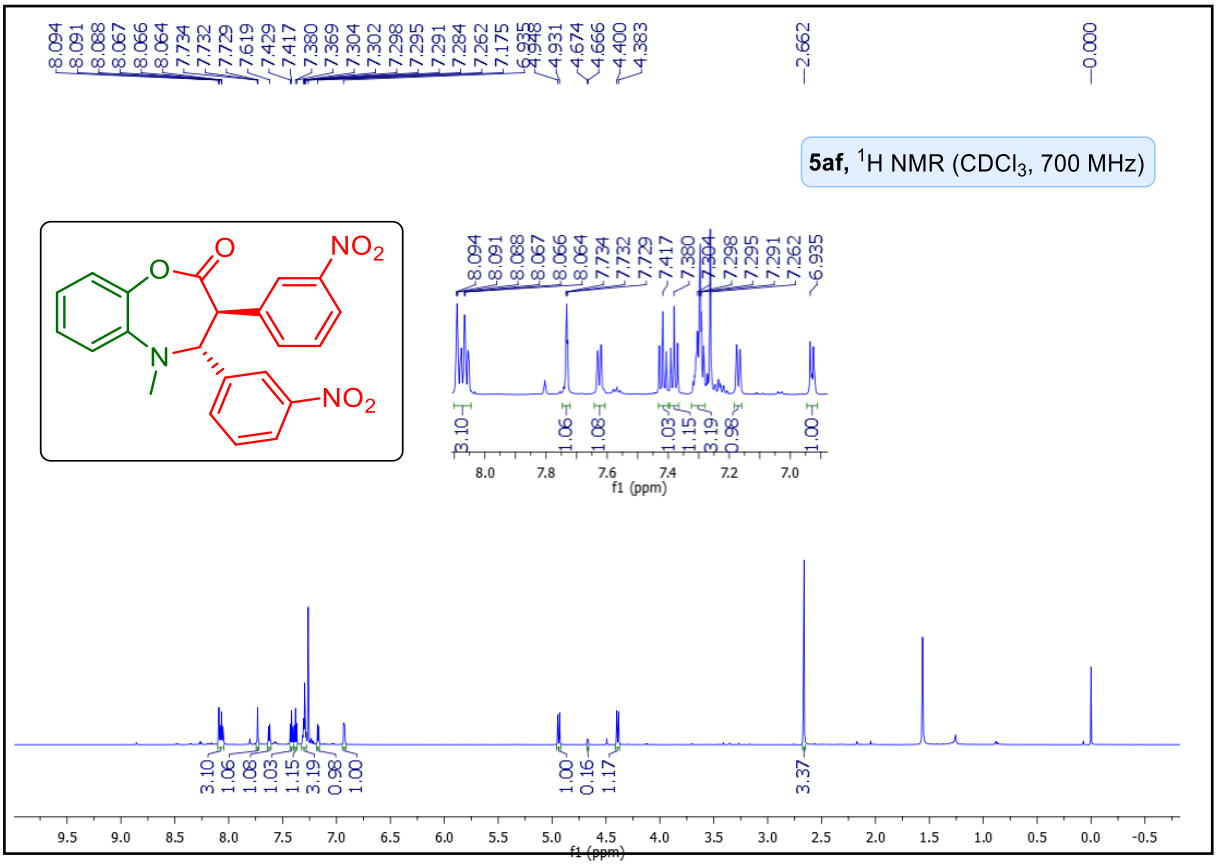

5pa', $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
Ph
Ph
f1 (ppm)

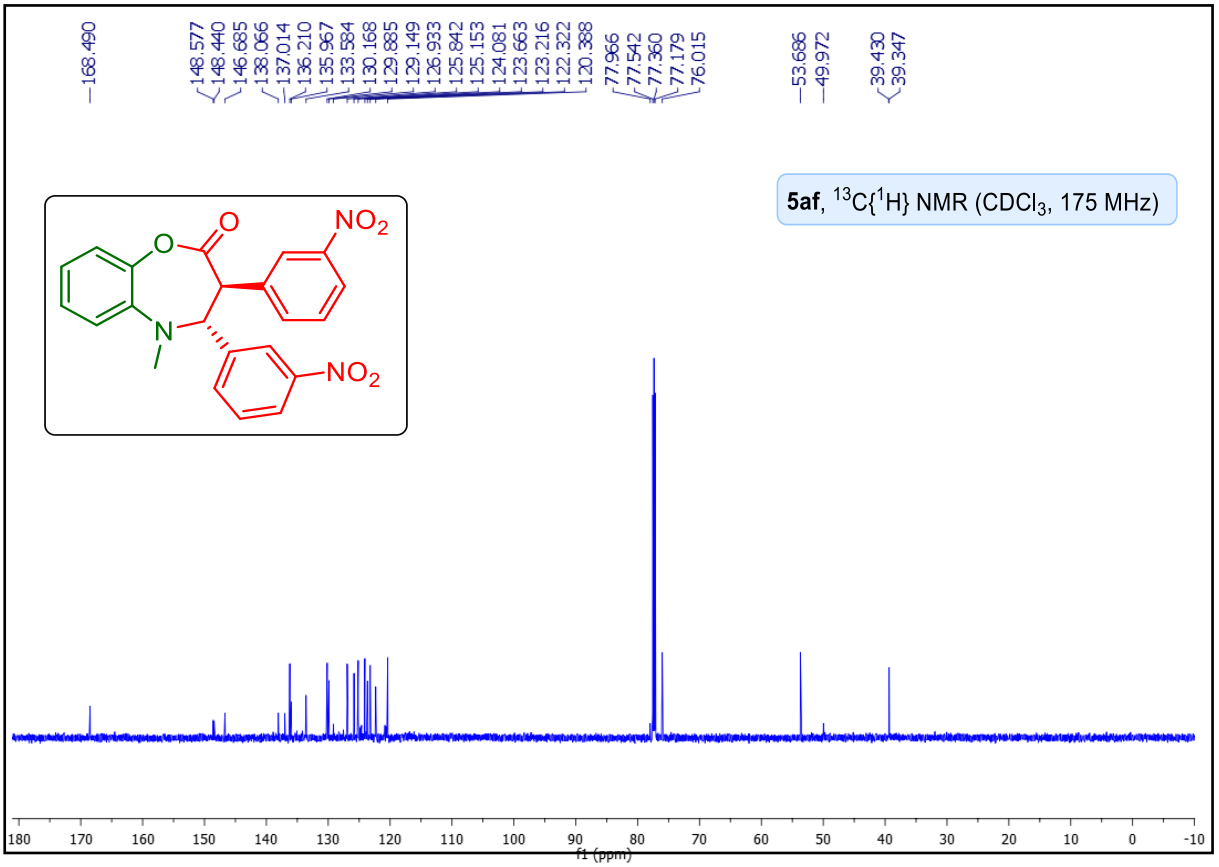

Ph
Ph
5pa, ^{1}H NMR ($CDCl_3$, 400 MHz)
f1 (ppm)

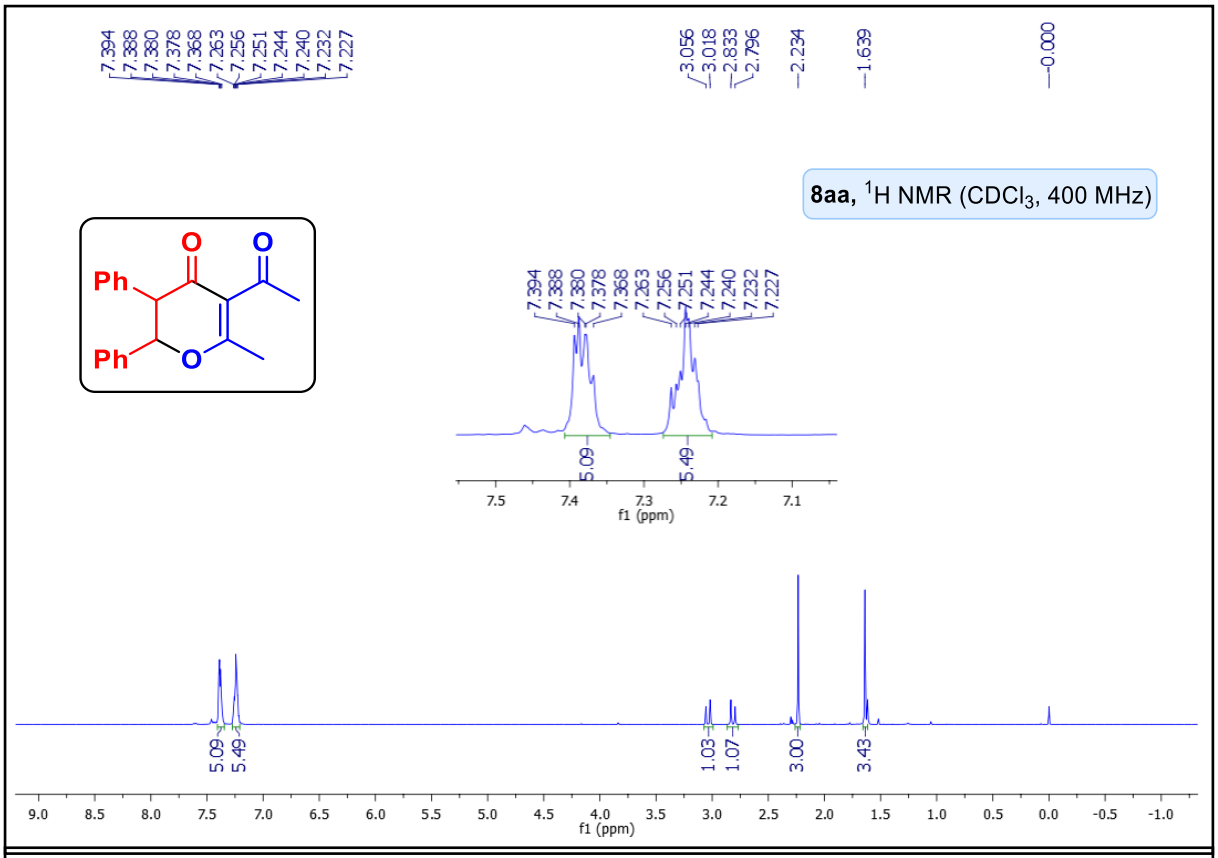

Ph
Ph
5pa, $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 175 MHz)
f1 (ppm)

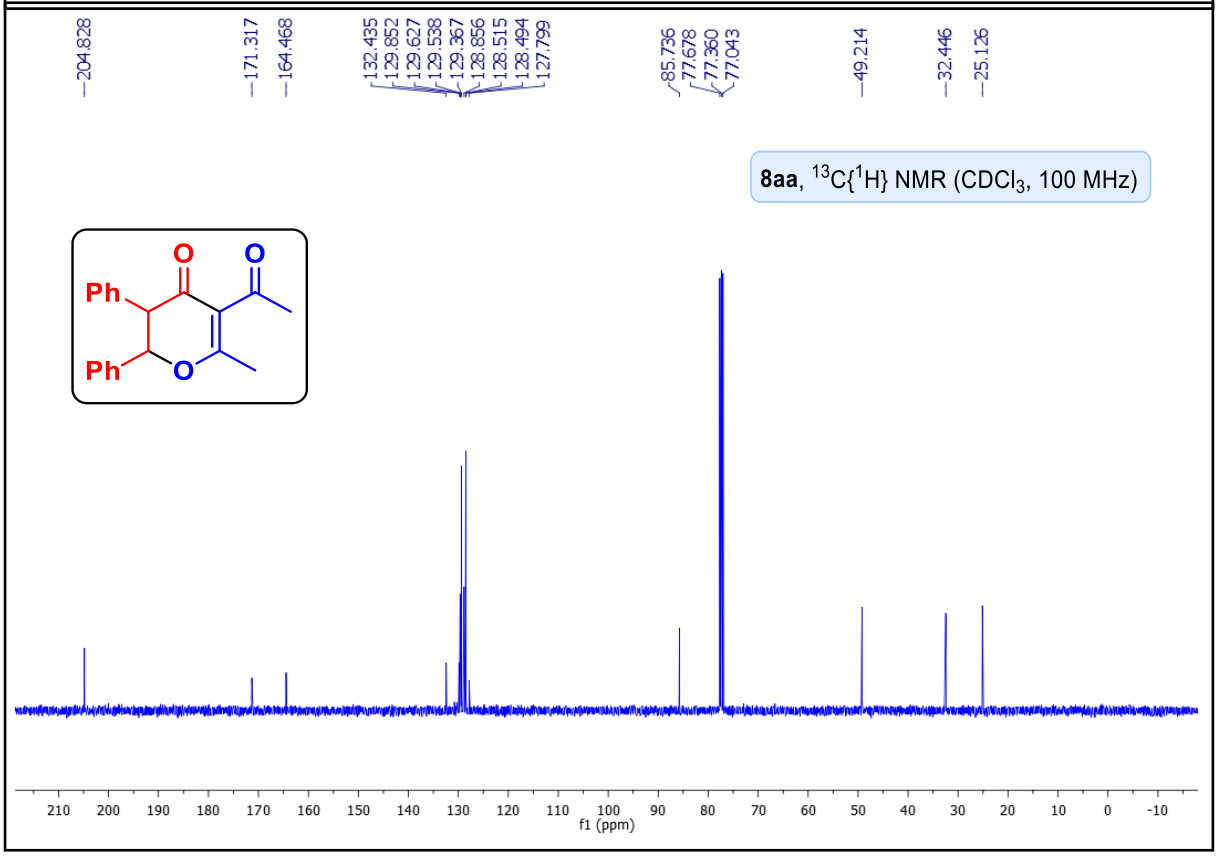

7.259
7.200
7.182
7.172
7.151
7.131
7.112
6.992
6.971
6.822
6.803
6.693
4.860
4.831
4.266
4.237
2.890
2.873
2.857
2.841
2.823
2.777
2.759
2.741
2.727
2.709
2.348
1.508
1.491
1.273
1.257
0.875
0.858
0.841
-0.000
5qa, ^{1}H NMR ($CDCl_3$, 400 MHz)
O
O
Ph
N
Ph
4
9.19
1.06
2.03
1.01
1.01
1.00
1.03
1.06
3.12
2.04
4.16
3.01
f1 (ppm)


170.759
145.740
136.763
136.640
135.600
134.812
130.176
128.434
128.422
128.275
127.960
127.907
125.243
124.990
119.501
77.542
77.360
77.178
75.198
54.054
50.619
29.327
26.958
22.682
21.527
14.356
5qa, ^{13}C{^{1}H} NMR ($CDCl_3$, 175 MHz)
O
O
Ph
N
Ph
4
f1 (ppm)


8.094
8.091
8.088
8.067
8.066
8.064
7.734
7.732
7.729
7.619
7.429
7.417
7.380
7.369
7.304
7.302
7.298
7.295
7.291
7.284
7.262
7.175
6.935
4.931
4.674
4.666
4.400
4.383
2.662
0.000
5af, 1H NMR (CDCl3, 700 MHz)
O
O
N
NO2
NO2
3.10
1.06
1.08
1.03
1.15
3.19
0.98
1.00
f1 (ppm)
3.10
1.06
1.03
1.03
1.15
3.19
0.98
1.00
1.00
0.16
1.17
3.37
f1 (ppm)

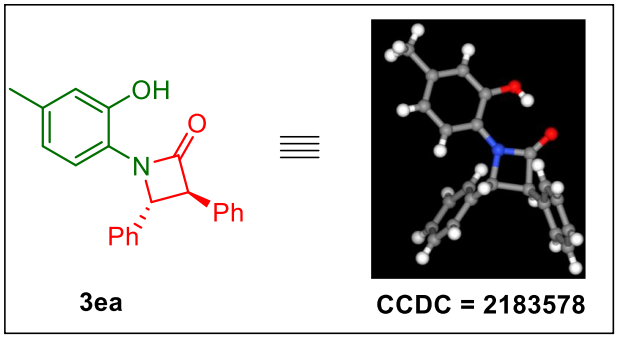

168.490
148.577
148.440
146.685
138.066
137.014
136.210
135.967
133.584
130.168
129.885
129.149
126.933
125.842
125.153
124.081
123.663
123.216
122.322
120.388
77.966
77.542
77.360
77.179
76.015
53.686
49.972
39.430
39.347
5af, 13C{1H} NMR (CDCl3, 175 MHz)
O
O
N
NO2
NO2
f1 (ppm)


7.394
7.388
7.380
7.378
7.368
7.263
7.256
7.251
7.244
7.240
7.232
7.227
3.056
3.018
2.833
2.796
2.234
1.639
0.000
8aa, 1H NMR (CDCl3, 400 MHz)
Ph
Ph
O
O
O
5.09
5.49
7.5
7.4
7.3
7.2
7.1
f1 (ppm)
5.09
5.49
1.03
1.07
3.00
3.43
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


204.828
171.317
164.468
132.435
129.852
129.627
129.538
129.367
128.856
128.515
128.494
127.799
85.736
77.678
77.360
77.043
49.214
32.446
25.126
8aa, 13C{1H} NMR (CDCl3, 100 MHz)
Ph
Ph
O
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

6. Crystallographic data:

X-ray data of (3S,4R)-1-(2-hydroxy-4-methylphenyl)-3,4-diphenylazetidin-2-one (3ea): Crystals of the compound (3S,4R)-1-(2-hydroxy-4-methylphenyl)-3,4-diphenylazetidin-2-one **(3ea**) were obtained after slow evaporation of EtOAc. Crystals suited for single crystal X-Ray diffraction measurements were mounted on a glass fiber. Geometry and intensity data were collected with a Rigaku Smartlab X-ray diffractometer equipped with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å, multilayer optics). Temperature was controlled using an Oxford Cryostream 700 instrument. Intensities were integrated with SAINT and SMART software packages and corrected for absorption with SADABS. The structure was solved by direct methods and refined on F2 with SHELXL-97 using Olex-2 software.



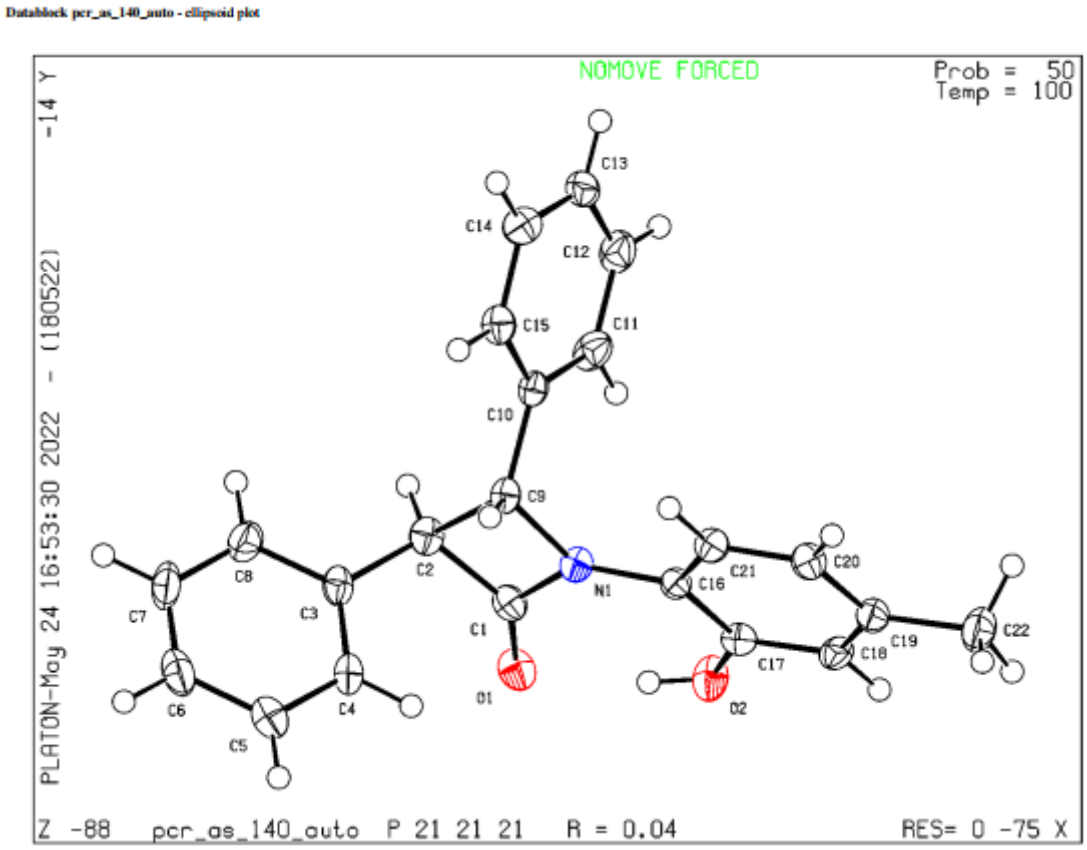


Figure S1. ORTEP diagram of **3ea** with 50% ellipsoid probability

Table S1. Crystal data and structure refinement for **(3S,4R)-1-(2-hydroxy-4-methylphenyl)-3,4-diphenylazetidin-2-one (3ea):**

Identification code	PCR_AS_140_auto
Empirical formula	$C_{22}H_{19}NO_2$
Formula weight	329.38
Temperature/K	100.01(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.0510(3)
b/Å	12.5184(6)
c/Å	16.9921(7)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1712.56(13)
Z	4
ρ_{calc}g/cm^3	1.278
μ/mm^{-1}	0.082
F(000)	696.0
Crystal size/mm^3	0.2 × 0.1 × 0.1
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	6.938 to 60.936
Index ranges	$-11 \leq h \leq 10$, $-17 \leq k \leq 16$, $-18 \leq l \leq 24$
Reflections collected	11357
Independent reflections	3736 [R_{int} = 0.0393, R_{sigma} = 0.0417]
Data/restraints/parameters	3736/0/228
Goodness-of-fit on F^2	1.035
Final R indexes [I>=2σ (I)]	R_1 = 0.0406, wR_2 = 0.1026
Final R indexes [all data]	R_1 = 0.0478, wR_2 = 0.1056
Largest diff. peak/hole / e Å^{-3}	0.30/-0.18
Flack parameter	-0.1(7)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) pcr_as_140_auto

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: pcr_as_140_auto

```
Bond precision:   C-C = 0.0030 A          Wavelength=0.71073

Cell:              a=8.0510(3)      b=12.5184(6)      c=16.9921(7)
                   alpha=90         beta=90           gamma=90
Temperature:       100 K

                  Calculated                Reported
Volume            1712.56(13)               1712.56(13)
Space group       P 21 21 21                P 21 21 21
Hall group        P 2ac 2ab                 P 2ac 2ab
Moiety formula    C22 H19 N O2              C22 H19 N O2
Sum formula       C22 H19 N O2              C22 H19 N O2
Mr                329.38                    329.38
Dx,g cm-3         1.278                     1.278
Z                 4                         4
Mu (mm-1)         0.082                     0.082
F000              696.0                     696.0
F000'             696.30
h,k,lmax          11,17,24                  11,17,24
Nref              5205[ 2949]               3736
Tmin,Tmax         0.990,0.992               0.929,1.000
Tmin'             0.984

Correction method= # Reported T Limits: Tmin=0.929 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.27/0.72        Theta(max)= 30.468

R(reflections)= 0.0406( 3322)                wR2(reflections)=
                                             0.1056( 3736)
S = 1.035                  Npar= 228
```

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 C

```
STRVA01_ALERT_4_C           Flack test results are meaningless.
          From the CIF: _refine_ls_abs_structure_Flack    -0.100
          From the CIF: _refine_ls_abs_structure_Flack_su    0.700
PLAT790_ALERT_4_C Centre of Gravity not Within Unit Cell: Resd.  #          1 Note
            C22 H19 N O2
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min).          7 Note
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          2 Report
PLAT915_ALERT_3_C No Flack x Check Done: Low Friedel Pair Coverage         59 %
```

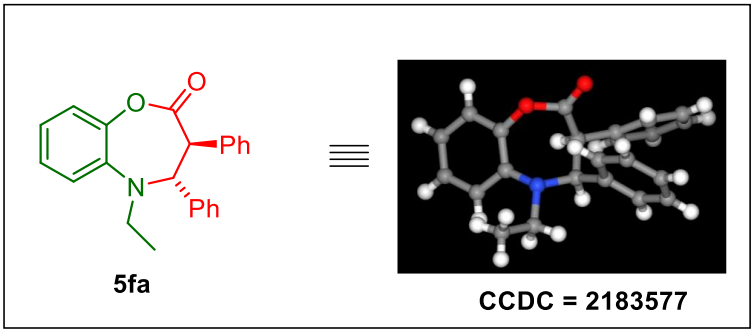
Alert level G

```
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          1 Report
PLAT032_ALERT_4_G Std. Uncertainty on Flack Parameter Value High .      0.700 Report
PLAT791_ALERT_4_G Model has Chirality at C2          (Sohnke SpGr)          S Verify
PLAT791_ALERT_4_G Model has Chirality at C9          (Sohnke SpGr)          R Verify
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        530 Note
PLAT916_ALERT_2_G Hooft y and Flack x Parameter Values Differ by .       0.20 Check
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        4.7 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         10 Info
```

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

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

X-ray data of (3S,4R)-5-ethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5fa): Crystals of the compound (3S,4R)-5-ethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (**5fa**) were obtained after slow evaporation of EtOAc. Crystals suited for single crystal X-Ray diffraction measurements were mounted on a glass fiber. Geometry and intensity data were collected with a Rigaku Smartlab X-ray diffractometer equipped with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å, multilayer optics). Temperature was controlled using an Oxford Cryostream 700 instrument. Intensities were integrated with SAINT and SMART software packages and corrected for absorption with SADABS. The structure was solved by direct methods and refined on F2 with SHELXL-97 using Olex-2 software.



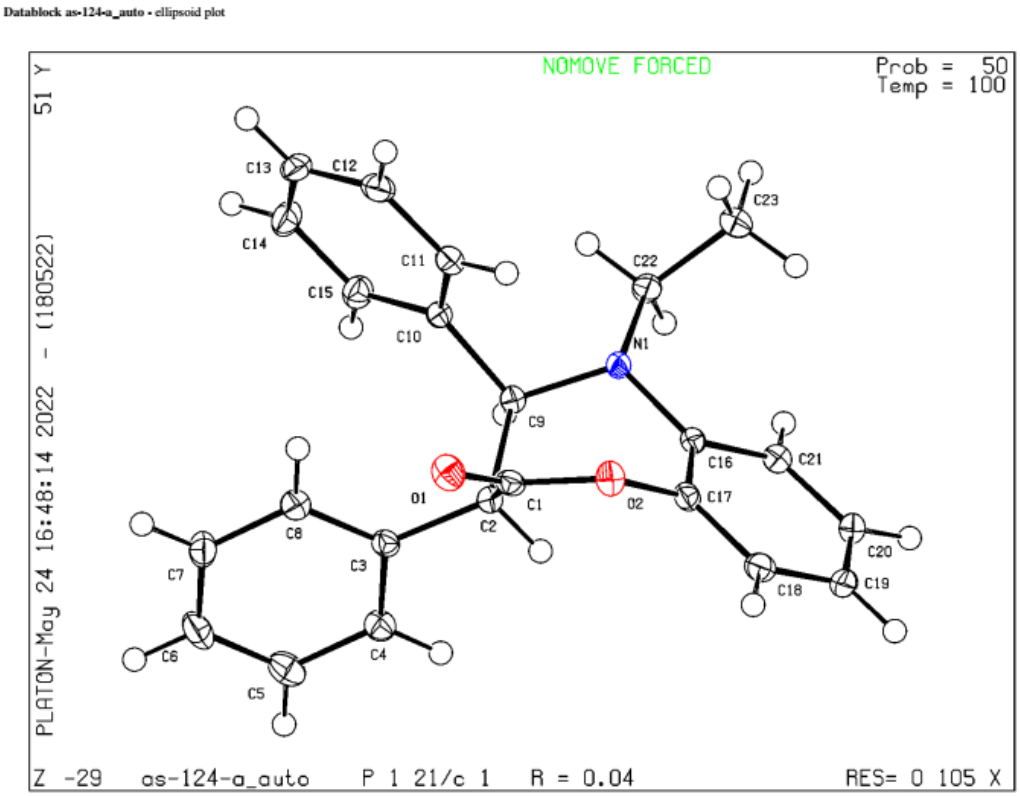


Figure S2. ORTEP diagram of **5fa** with 50% ellipsoid probability

Table S2. Crystal data and structure refinement for **(3S,4R)-5-ethyl-3,4-diphenyl-4,5-dihydrobenzo[b][1,4]oxazepin-2(3H)-one (5fa):**

Identification code	AS-124-A_auto
Empirical formula	$C_{23}H_{21}NO_2$
Formula weight	343.41
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.1816(4)
b/Å	8.6211(3)
c/Å	17.1645(6)
α/°	90
β/°	96.736(3)
γ/°	90
Volume/Å^3	1790.15(11)
Z	4
ρ_{calc}g/cm^3	1.274
μ/mm^{-1}	0.081
F(000)	728.0
Crystal size/mm^3	0.2 × 0.1 × 0.1
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	6.736 to 61.014
Index ranges	$-16 \leq h \leq 14$, $-11 \leq k \leq 11$, $-22 \leq l \leq 23$
Reflections collected	20789
Independent reflections	4460 [R_{int} = 0.0379, R_{sigma} = 0.0287]
Data/restraints/parameters	4460/0/236
Goodness-of-fit on F^2	1.071
Final R indexes [I>=2σ (I)]	R_1 = 0.0377, wR_2 = 0.0938
Final R indexes [all data]	R_1 = 0.0436, wR_2 = 0.0969
Largest diff. peak/hole / e Å^{-3}	0.35/-0.20

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) as-124-a_auto

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: as-124-a_auto

```
Bond precision:   C-C = 0.0015 A          Wavelength=0.71073

Cell:             a=12.1816(4)     b=8.6211(3)         c=17.1645(6)
                  alpha=90         beta=96.736(3)      gamma=90
Temperature:      100 K
```

	Calculated	Reported
Volume	1790.15(11)	1790.15(11)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C23 H21 N O2	C23 H21 N O2
Sum formula	C23 H21 N O2	C23 H21 N O2
Mr	343.41	343.41
Dx,g cm-3	1.274	1.274
Z	4	4
Mu (mm-1)	0.081	0.081
F000	728.0	728.0
F000'	728.31	
h,k,lmax	17,12,24	16,11,23
Nref	5477	4460
Tmin,Tmax	0.990,0.992	0.786,1.000
Tmin'	0.984	

```
Correction method= # Reported T Limits: Tmin=0.786 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.814            Theta(max)= 30.507

R(reflections)= 0.0377( 3880)                    wR2(reflections)=
                                                 0.0969( 4460)
S = 1.071                  Npar= 236
```

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 C

PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min). 9 Note

Alert level G

PLAT793_ALERT_4_G Model has Chirality at C2 (Centro SPGR) S Verify
PLAT793_ALERT_4_G Model has Chirality at C9 (Centro SPGR) S Verify
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 931 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ..... 4.7 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 21 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by 3 Check

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

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

7. Reference:

(1) (a) Similar information was reported in Banjare, S. K.; Nanda, T.; Ravikumar, P. C. Cobalt-Catalyzed Regioselective Direct C-4 Alkenylation of 3-Acetylindole with Michael Acceptors Using a Weakly Coordinating Functional Group. *Org. Lett.* **2019**, *21*, 8138−8143.

(2) Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. NMR chemical shifts of common laboratory solvents as trace impurities. *J. Org. Chem.* **1997**, *62*, 7512.

(3) Matralis, A. N.; Katselou, M. G.; Nikitakis, A.; Kourounakis, A. P. Novel Benzoxazine and Benzothiazine Derivatives as Multifunctional Antihyperlipidemic Agents. *J. Med. Chem.* **2011**, *54*, 5583−5591.

(4) Nanda, T.; Biswal, P.; Pati, B. V.; Banjare, S. K.; Ravikumar, P. C. PalladiumCatalyzed C-C Bond Activation of Cyclopropenone: Modular Access to Trisubstituted α,β-Unsaturated Esters and Amides. *J. Org. Chem.* **2021**, *86*, 2682−2695.