Supporting Information

Pd-Catalyzed *ortho-/meta*-annulation of biphenyl amines with enynes through non-rollover cyclometallation

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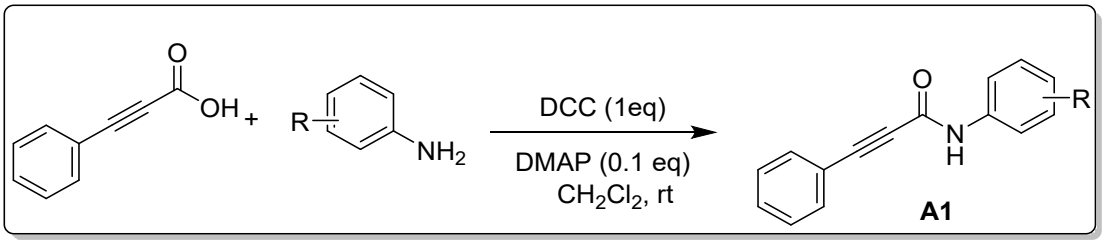
1. General Information and methods.

All reagents and solvents were purchased from commercial sources and used without purification. NMR spectra were recorded with a 300, 400 or 500 MHz spectrometer for ^{1}H NMR, 100 or 125 MHz for ^{13}C NMR spectroscopy. Chemical shifts are reported relative to the residual signals of tetramethyl silane in $CDCl_3$ or deuterated solvent $CDCl_3$ for ^{1}H and ^{13}C NMR spectroscopy. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of triplets (dt), triplet (t), quartet (q), multiplet (m). HRMS were recorded by using QTof mass spectrometer. Column chromatography was performed with silica gel (100–200 mesh) as the stationary phase. All reactions were monitored by using TLC.

Following the known below procedures, the 1,6-enynes **1** and biphenyl amines **2** were prepared.

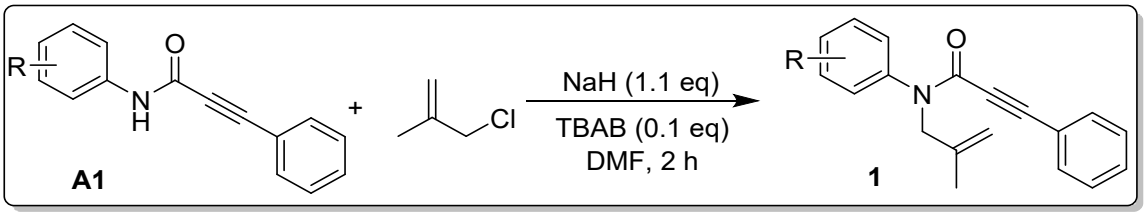
2. Experimental Procedures:

2.1. Preparation of amide A1 from phenyl propiolic acid and anilines: General Procedure (GP-1):[1,2]

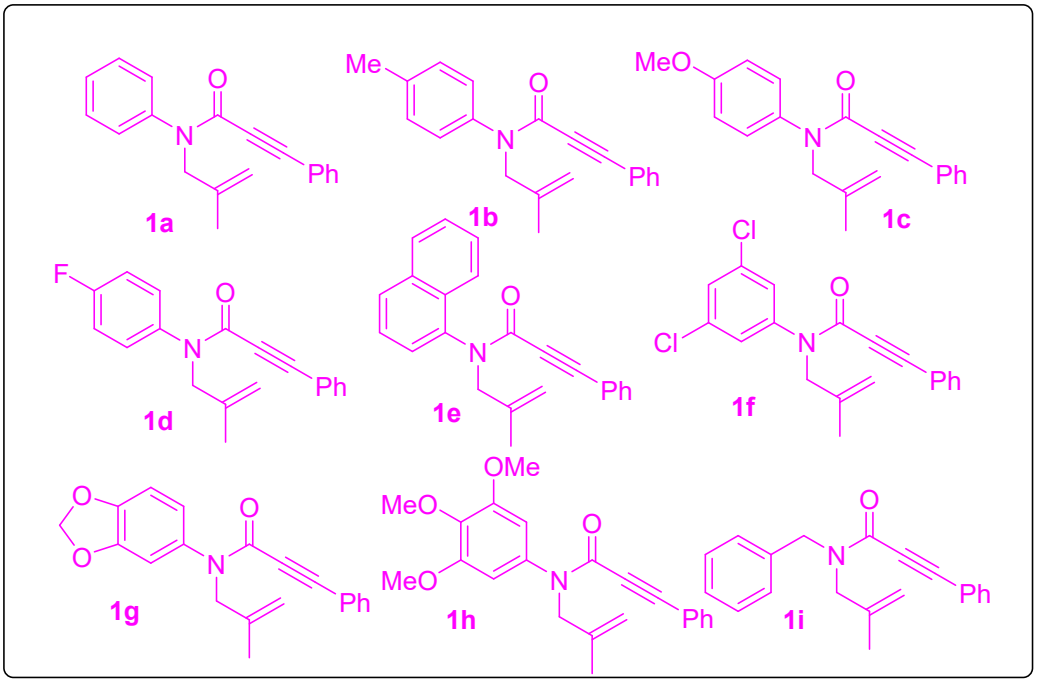


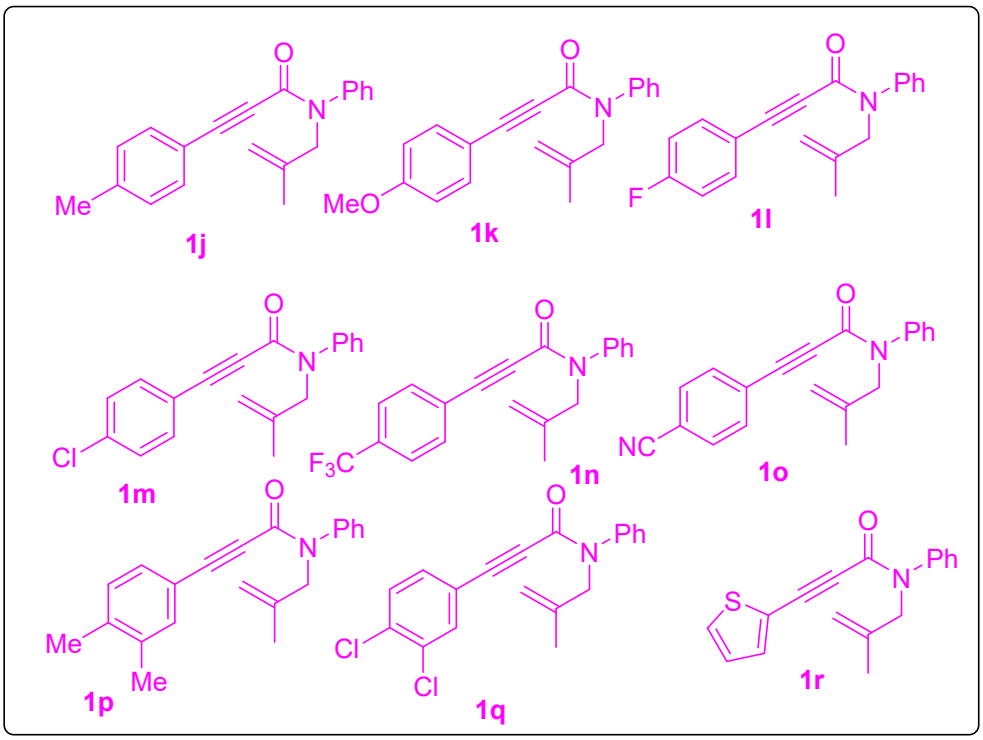
An oven dried round-bottom flask was charged with phenylpropiolic acid (1 equiv) and DMAP (10 mol %), and purged with argon for 10 minutes. The contents were dissolved in CH_2Cl_2 before the amine was added (1.1 equiv). The mixture was cooled to 0 °C and a CH_2Cl_2 solution of DCC (1.0 equiv) was added drop-wise and the reaction was warmed to room temperature and stirred overnight (approximately 12 hours). The contents of the flask were filtered through a plug of celite eluting with CH_2Cl_2. The filtrate was concentrated in vacuo while adsorbing onto silica gel and the crude material was purified by flash column silica gel chromatography using EtOAc:hexane system to get **A1**.

2.2. General Procedure (GP-2):



A 60% dispersion of NaH in mineral oil (1.1 equiv) was added to a stirred solution of **A1** (1 equiv, 0.15 M) in DMF at 0 °C. The corresponding solution was stirred for 5 minutes before warming to room temperature where it was stirred for 30 minutes. The solution of the sodium propiolamide was again cooled to 0 °C and TBAB (10 mol %) and 3-chloro-2-methyl-1-propene (4 equiv, dropwise) were added in that order. The reaction was warmed to room temperature and was stirred for 2 hours after which the completion of the reaction was determined by TLC analysis. The reaction was carefully quenched with a saturated solution of NH_4Cl and then was diluted with water and ethyl acetate. After separating the layers, the aqueous layer was extracted with EtOAc. The combined organic layers were washed sequentially with water and brine, dried over sodium sulfate and concentrated under reduced pressure. The material was purified by flash column silica gel chromatography using EtOAc:hexane system to get **1** with 60-85% yield.



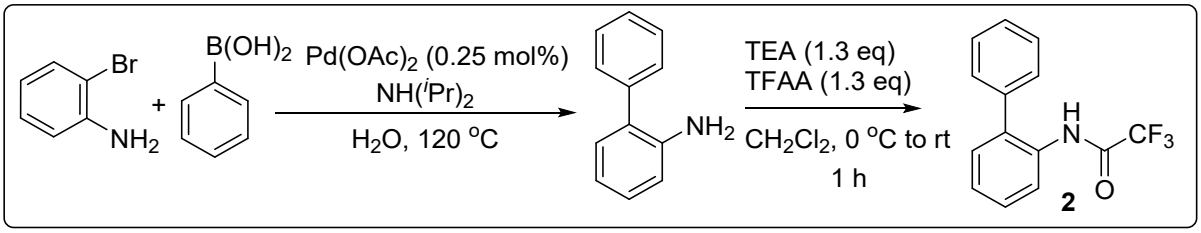


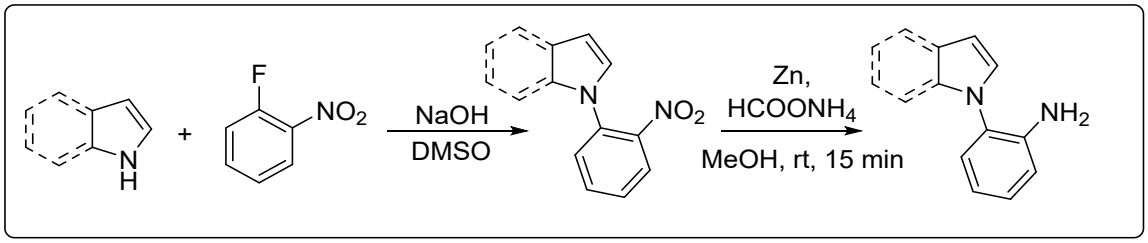
2.3. Preparation of protected biphenyl amine: General Procedure (GP-3):[3,4]

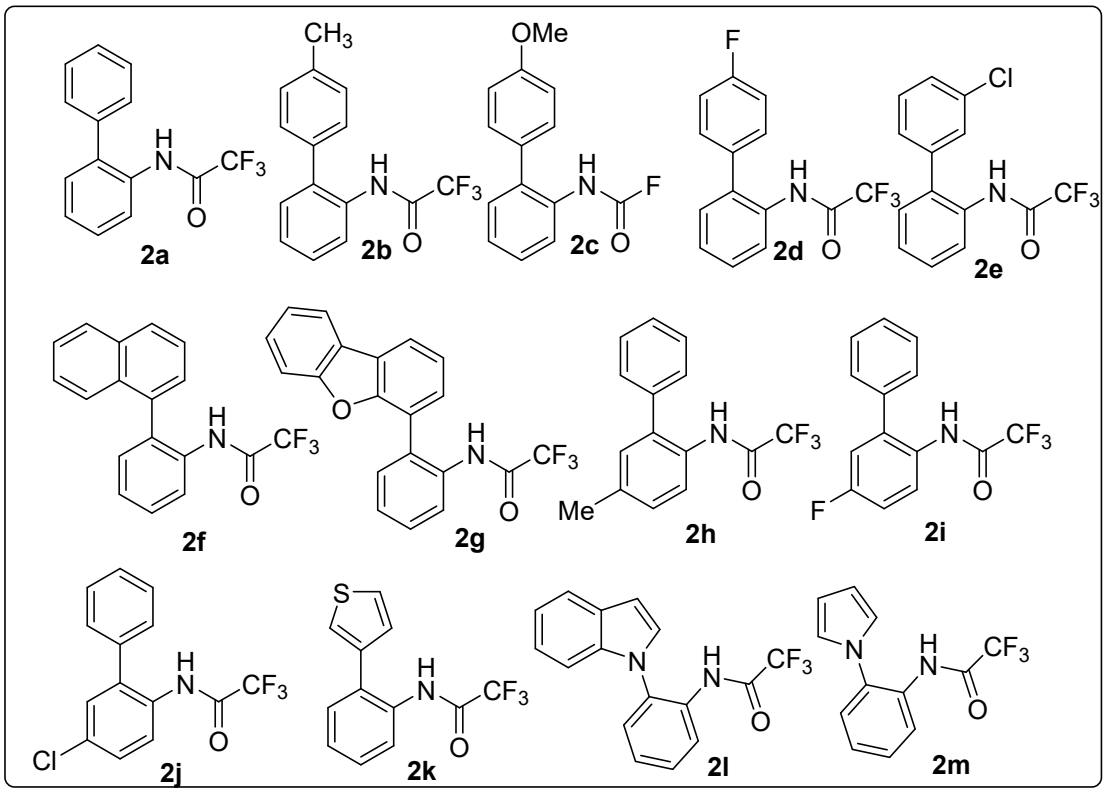
A mixture of *o*-bromoanilines (3.0 mmol), aryl boronic acids (4.5 mmol), base (6.0 mmol), $Pd(OAc)_2$ (0.25 mol%, 1.68 mg) and H_2O (6.0 mL) was stirred at 100 °C for 0.5 h. The reaction mixture was added with brine solution (10 mL) and was extracted with ethyl acetate (3 × 20 mL). The solvent was concentrated under vacuum and the product was purified through a short silica gel column using petroleum ether/EtOAc (20:1) as the eluent.

$(CF_3CO)_2O$ (1.3 equiv) was added to biaryl-2-amines (3mmol, 1 equiv) and triethylamine (1.3 equiv) in dry dichloromethane (6 mL) at 0 °C under nitrogen atmosphere. Stirring was continued for an additional 1 h at room temperature. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ (3 mL), extracted with dichloromethane (10 mL×2). The organic phase was washed with brine (10 mL), dried over anhydrous Na_2SO_4,

filtered and concentrated. The residue was purified by flash column chromatography with ethyl acetate and petroleum ether as eluent to afford the corresponding substrate **2**.

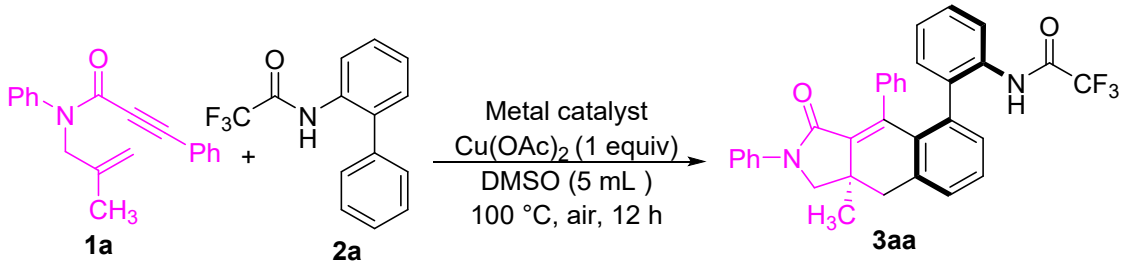






3. Optimization Studies:

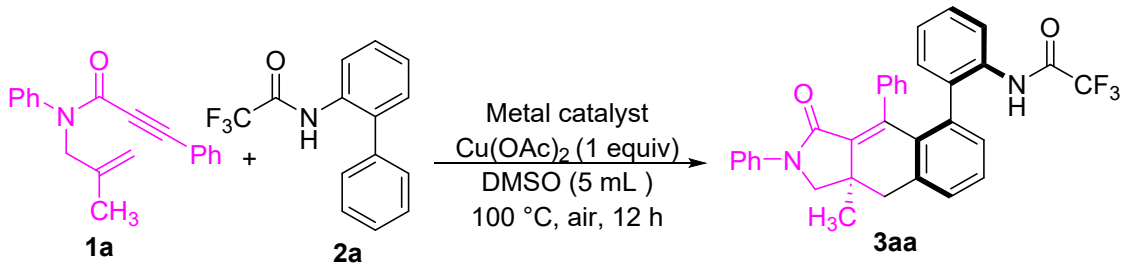
Table S1: Optimization of Metal Catalyst.[a]



Entry	Metal Catalyst	Yield (%) of **3aa**
1	$PdCl_2$ (10 mol%)	24
2	$Pd(PPh_3)_4$ (10 mol%)	--
3	$Pd(TFA)_2$ (10 mol%)	55
4	$Pd(PPh_3)_2Cl_2$ (10 mol%)	10
5	$Pd(OAc)_2$ (5 mol%)	42
6	**$Pd(OAc)_2$ (10 mol%)**	**82**

[a]Reaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol), $Pd(OAc)_2$ (10 mol %), $Cu(OAc)_2$ (1 equiv), DMSO, (5 mL) 100 °C for 12 h, air balloon.

Table S2: Optimization of Solvent.[a]



Entry	Solvent	Yield (%) of **3aa**
1	MeCN	38
2	THF	10
3	MeOH	--
4	MTBE	--
5	**DMSO**	**82**

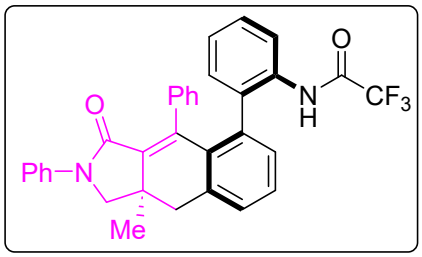
[a]Reaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol), $Pd(OAc)_2$ (10 mol %), $Cu(OAc)_2$ (1 equiv), DMSO, (5 mL) 100 °C for 12 h, air balloon.

4. General Procedure-A for *ortho-/meta*-annulation of biphenyl amines with enynes through non-rollover cyclometallation and Characteristic data:

To a mixture of enyne **1a** (137.5 mg, 0.5 mmol), N-([1,1'-biphenyl]-2-yl)-2,2,2-trifluoroacetamide **2a** (132.5 mg, 0.5 mmol) in DMSO, $Pd(OAc)_2$ (11.2 mg, 10 mol %), $Cu(OAc)_2$ (90 mg, 1 equiv) were introduced and the reaction mixture was stirred at 100 °C (oil bath) for 12 hours under air balloon. After completion of the reaction, the reaction mixture was cooled to room temperature before water was added to it. The aqueous layer was extracted with ethyl acetate (3×10 mL), the combined organic extracts were washed with brine, dried over sodium sulfate and concentrated under reduced pressure. The crude residue was purified by column

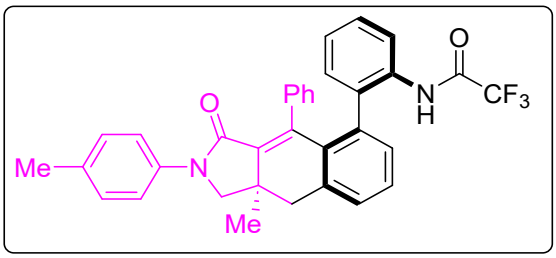
chromatography (R*f* = 0.40) (SiO_2, EtOAc:Hexane, 10:90) to get **3aa** as a white solid (220.58 mg, 82% yield, 7.8:1 dr, mp 194-197 °C). The diastereomeric ratio (dr) was assigned by ^{1}H NMR analysis.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3aa):



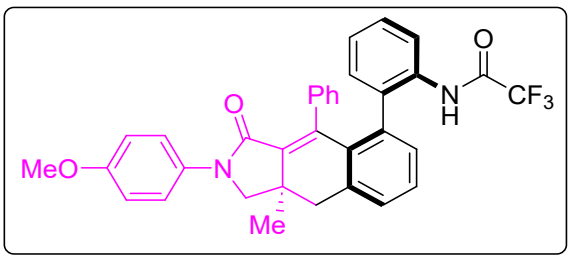
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid, (220.58 mg, 82% yield), 7.8:1 dr, mp 194-197 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.67 (dd, *J* = 8.7, 0.8 Hz, 2H), 7.49 – 7.37 (m, 3H), 7.34 (dd, *J* = 10.8, 5.1 Hz, 3H), 7.13 (dddd, *J* = 8.7, 7.4, 6.3, 1.5 Hz, 3H), 7.04 (d, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 7.0 Hz, 1H), 6.88 (dd, *J* = 7.4, 1.7 Hz, 3H), 6.67 (s, 1H), 3.85 (d, *J* = 9.4 Hz, 1H), 3.80 (d, *J* = 9.3 Hz, 1H), 3.24 (d, *J* = 15.4 Hz, 1H), 3.09 (d, *J* = 15.3 Hz, 1H), 1.31 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.7,(q, 154.1, 153.9, 153.6, 153.3), 141.7, 139.6, 138.0, 137.7, 137.4, 136.8, 136.6, 133.5, 132.0, 131.2, 130.8, 130.1, 128.8, 128.7, 128.5, 126.8, 126.4, 125.4, 124.7, 121.1, 119.8, 116.8, 114.5, 59.6, 42.7, 34.8, 22.4. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{33}H_{26}F_3N_2O_2$ $[M+H]^+$ 539.1946, found 539.1935.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-4-phenyl-2-(p-tolyl)-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ba):



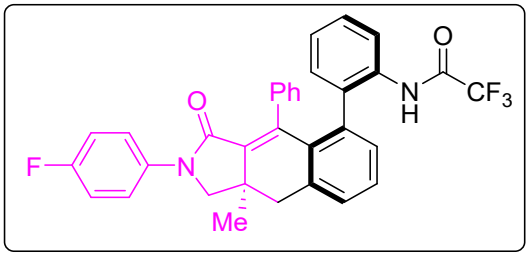
The title compound was prepared from **1b** (137.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure A. Purification using column chromatography (R_f = 0.40 SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (223.56 mg, 81% yield), 7.4:1 dr, mp 210-215 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.61 (dd, *J* = 8.2, 1.0 Hz, 1H), 7.47 – 7.45 (m, 2H), 7.34 (d, *J* = 7.5 Hz, 1H), 7.31 (s, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.08 (td, *J* = 7.9, 1.6 Hz, 1H), 7.05 – 6.99 (m, 3H), 6.94 (d, *J* = 7.3 Hz, 1H), 6.88 (t, *J* = 7.3 Hz, 2H), 6.78 (dd, *J* = 7.5, 1.5 Hz, 3H), 6.54 (s, 1H), 3.73 (d, *J* = 9.4 Hz, 1H), 3.68 (d, *J* = 9.3 Hz, 1H), 3.13 (d, *J* = 15.3 Hz, 1H), 2.98 (d, *J* = 15.3 Hz, 1H), 2.22 (s, 3H), 1.20 (s, 3H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 164.5,(d, 153.9, 153.5), 141.3, 138.0, 137.8, 137.5, 137.2, 136.7, 136.6, 134.3, 133.6, 131.2, 130.8, 130.0, 129.2, 129.2, 128.8, 128.6, 128.5, 126.7, 126.4, 125.4, 121.1, 119.7, 117.1, 59.7, 42.6, 34.8, 22.4, 20.9. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.2. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_2$ $[M+H]^+$ 553.2103, found 553.2098.

2,2,2-trifluoro-N-(2-(2-(4-methoxyphenyl)-9a-methyl-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ca):



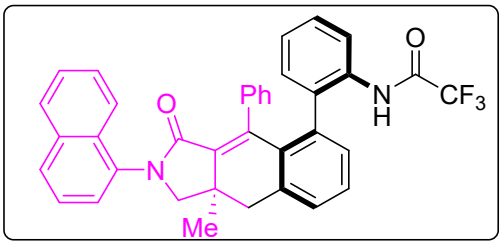
The title compound was prepared from **1c** (152.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.35, SiO_2, EtOAc:hexane, 12:88) gave pure product as an off-white solid (232.88 mg, 82% yield), 7.5:1 dr, mp 212-214 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 (d, *J* = 7.4 Hz, 1H), 7.48 (dd, *J* = 9.4, 2.3 Hz, 2H), 7.34 (d, *J* = 7.5 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.07 (td, *J* = 7.9, 1.5 Hz, 1H), 7.01 (td, *J* = 7.5, 1.2 Hz, 1H), 6.94 (d, *J* = 7.3 Hz, 1H), 6.91 – 6.85 (m, 2H), 6.77 (ddd, *J* = 10.9, 6.4, 4.0 Hz, 5H), 6.55 (s, 1H), 3.72 (d, *J* = 9.4 Hz, 1H), 3.70 (s, 3H), 3.66 (d, *J* = 9.4 Hz, 1H), 3.13 (d, *J* = 15.3 Hz, 1H), 2.98 (d, *J* = 15.3 Hz, 1H), 1.21 (s, 3H)**. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 163.4, 155.6, (q, 153.4, 152.9, 152.4, 151.9), 140.1, 136.9, 136.7, 136.4, 135.6, 135.6, 132.5, 131.9, 130.2, 129.8, 129.0, 127.7, 127.5, 127.4, 125.7, 125.3, 124.3, 120.3, 120.0, 118.8, 116.5, 112.8, 58.9, 54.4, 41.6, 33.8, 21.4. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_3$ $[M+H]^+$ 569.2052, found 569.2052.

2,2,2-trifluoro-N-(2-(2-(4-fluorophenyl)-9a-methyl-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3da):



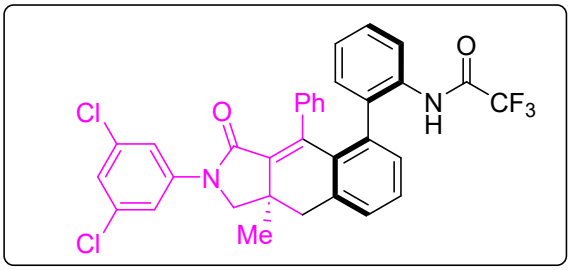
The title compound was prepared from **1d** (146.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid, (225.18 mg, 81% yield), 7:1 dr, mp 217-220 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.1 Hz, 1H), 7.67 – 7.59 (m, 2H), 7.41 (dt, *J* = 14.8, 7.3 Hz, 3H), 7.21 – 7.15 (m, 1H), 7.14 – 7.08 (m, 1H), 7.07 – 6.93 (m, 5H), 6.87 (dd, *J* = 7.4, 1.6 Hz, 3H), 6.66 (s, 1H), 3.82 (d, *J* = 9.3 Hz, 1H), 3.77 (d, *J* = 9.1 Hz, 1H), 3.24 (d, *J* = 15.2 Hz, 1H), 3.13 – 3.05 (d, 1H), 1.32 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.6, 160.8, 158.3, 153.9, 153.5, 141.9, 137.9, 137.6, 137.0, 136.8, 136.5, 135.7, 133.5, 131.2, 130.8, 130.1, 128.8, 128.5, 126.9, 126.4, 125.4, 121.5, 121.4, 121.1, 119.9, 117.1, 115.5, 115.3, 59.8, 42.6, 34.8, 22.4. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.2, -117.4. **HRMS (ESI)** calcd for $C_{33}H_{25}F_4N_2O_2$ $[M+H]^+$ 557.1852, found 557.1841.

2,2,2-trifluoro-N-(2-(9a-methyl-2-(naphthalen-1-yl)-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ea):



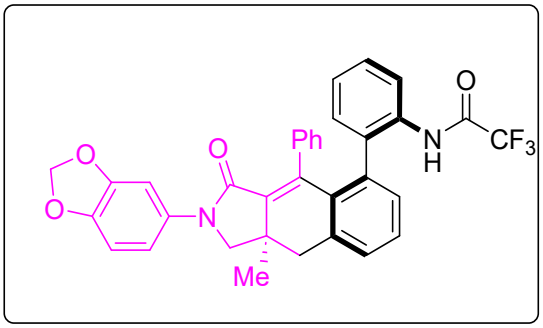
The title compound was prepared from **1e** (162.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (235.2mg, 80% yield), 5.5:1 dr, mp 232-235 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.86 – 7.79 (m, 2H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.54 (dd, *J* = 15.8, 7.5 Hz, 3H), 7.45 (dd, *J* = 13.8, 6.8 Hz, 2H), 7.38 (dd, *J* = 15.3, 8.7 Hz, 2H), 7.17 – 7.03 (m, 3H), 6.92 (d, *J* = 6.1 Hz, 4H), 6.73 (s, 2H), 3.92 (d, *J* = 9.2 Hz, 1H), 3.81 (d, *J* = 9.6 Hz, 1H), 3.34 (d, *J* = 15.1 Hz, 1H), 3.10 (d, *J* = 15.3 Hz, 1H), 1.50 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 165.8, (d, 153.9, 153.6), 141.6, 138.1, 137.8, 136.9, 136.5, 136.4, 135.6, 134.5, 133.5, 131.2, 130.8, 130.0, 129.7, 128.8, 128.7, 128.6, 128.5, 128.3, 126.8, 126.3, 125.5, 125.4, 124.6, 122.8, 121.1, 120.0, 117.1, 114.3, 62.7, 42.6, 36.4, 22.6. **HRMS (ESI)** calcd for $C_{37}H_{28}F_3N_2O_2$ $[M+H]^+$ 589.2103, found 589.2109.

2, N-(2-(2-(3,5-dichlorophenyl)-9a-methyl-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3fa):



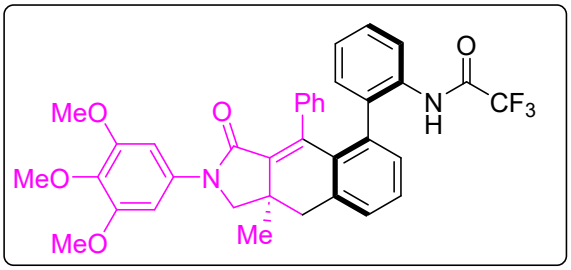
The title compound was prepared from **1g** (172.0 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (218.2 mg, 72% yield), 5.4:1 dr, mp 180-185 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.57 (d, *J* = 1.8 Hz, 2H), 7.35 (d, *J* = 6.9 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.09 (td, *J* = 7.9, 1.6 Hz, 1H), 7.05 – 7.00 (m, 2H), 6.93 (dd, *J* = 18.0, 6.9 Hz, 3H), 6.76 (dd, *J* = 7.5, 1.6 Hz, 3H), 6.54 (s, 1H), 3.68 (s, 2H), 3.14 (d, *J* = 15.3 Hz, 1H), 3.00 (d, *J* = 15.4 Hz, 1H), 1.22 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.9, (q, 154.3, 153.9, 153.5, 153.2, 143.1, 141.4, 137.8, 137.4, 137.0, 136.3, 135.1, 133.3, 132.1, 131.2, 130.8, 130.2, 129.1, 128.9, 128.6, 127.1, 126.5, 125.4, 124.3, 121.1, 120.0, 117.5, 114.2, 59.4, 42.6, 34.6, 22.5. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{33}H_{24}F_3Cl_2N_2O_2$ $[M+H]^+$ 607.1167, found 607.1158.

N-(2-(2-(benzo[d][1,3]dioxol-5-yl)-9a-methyl-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3ga):



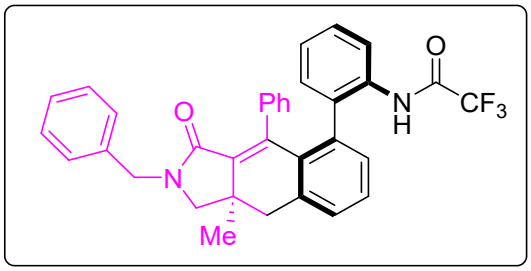
The title compound was prepared from **1f** (159.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.30, SiO_2, EtOAc:hexane, 15:85) gave pure product as an off-white solid (221.2 mg, 76% yield), 6.2:1 dr, mp 235-238 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 – 7.54 (m, 1H), 7.35 – 7.30 (m, 3H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.08 (td, *J* = 7.9, 1.6 Hz, 1H), 7.02 (td, *J* = 7.5, 1.3 Hz, 1H), 6.93 (s, 1H), 6.90 – 6.81 (m, 3H), 6.78 (dd, *J* = 7.5, 1.6 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 1H), 6.54 (s, 1H), 5.85 (s, 2H), 3.69 (d, *J* = 9.4 Hz, 1H), 3.64 (d, *J* = 9.4 Hz, 1H), 3.12 (d, *J* = 15.2 Hz, 1H), 2.98 (d, *J* = 15.3 Hz, 1H), 1.20 (s, 3H). **^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$)** δ 164.47, 153.87, 153.58, 147.75, 144.56, 141.40, 137.95, 137.70, 137.31, 136.74, 136.57, 134.10, 133.53, 131.21, 130.84, 130.04, 128.78, 128.67, 128.48, 126.78, 126.40, 125.39, 121.09, 119.92, 116.81, 112.73, 107.74, 102.70, 101.30, 60.28, 42.61, 34.80, 22.43. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.2. **HRMS (ESI)** calcd for $C_{34}H_{26}F_3N_2O_2$ $[M+H]^+$ 583.1845, found 583.1865.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-4-phenyl-2-(3,4,5-trimethoxyphenyl)-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ha):



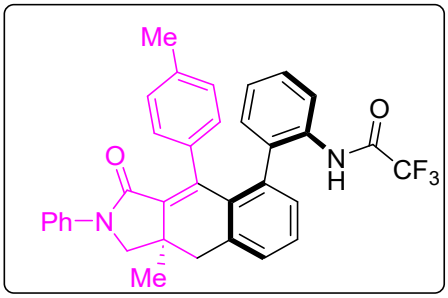
The title compound was prepared from **1h** (182.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.3, SiO_2, EtOAc:hexane, 15:85) gave pure product as an off-white solid (235.5 mg, 75% yield), 5.7:1 dr, mp 210-213 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.35 (d, *J* = 7.4 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.19 (s, 1H), 7.08 (td, *J* = 7.9, 1.6 Hz, 1H), 7.01 (td, *J* = 7.5, 1.2 Hz, 1H), 6.95 (d, *J* = 7.4 Hz, 1H), 6.88 (d, *J* = 7.3 Hz, 1H), 6.84 (s, 2H), 6.81 – 6.76 (m, 3H), 6.61 (s, 1H), 3.77 (s, 6H), 3.72 (s, 3H), 3.68 (d, *J* = 9.2 Hz, 2H), 3.15 (d, *J* = 15.3 Hz, 1H), 2.99 (d, *J* = 15.3 Hz, 1H), 1.21 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 163.6, 152.8, 152.4, 152.0, 140.5, 136.8, 136.7, 136.4, 135.7, 135.5, 134.7, 134.3, 132.4, 130.1, 129.8, 129.0, 127.7, 127.6, 127.4, 125.7, 125.3, 124.3, 120.0, 118.8, 116.0, 113.1, 97.2, 59.9, 59.1, 55.3, 41.5, 33.8, 21.3. **HRMS (ESI)** calcd for $C_{36}H_{32}F_3N_2O_5$ $[M+H]^+$ 629.2263, found 629.2274.

N-(2-(2-benzyl-9a-methyl-3-oxo-4-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3ia):



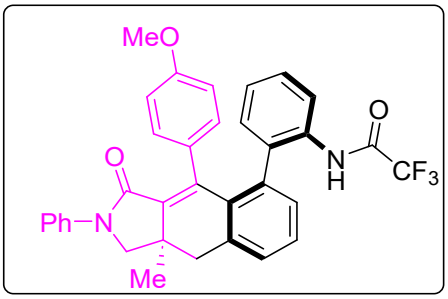
The title compound was prepared from **1i** (144.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.35, SiO_2, EtOAc:hexane, 16:84) gave pure product as an off-white solid (174.85 mg, 65% yield), 5:1 dr, mp 178-182 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.7 Hz, 1H), 7.47 (d, *J* = 8.7 Hz, 1H), 7.40 – 7.30 (m, 5H), 7.24 (dd, *J* = 16.6, 8.8 Hz, 3H), 7.19 – 7.07 (m, 3H), 7.00 (dd, *J* = 15.8, 7.0 Hz, 3H), 6.85 (d, *J* = 7.0 Hz, 1H), 6.65 (s, 1H), 4.81 (d, *J* = 14.8 Hz, 1H), 4.17 (d, *J* = 14.8 Hz, 1H), 3.23 (d, *J* = 9.3 Hz, 1H), 3.18 (d, *J* = 9.4 Hz, 1H), 3.10 (d, *J* = 15.2 Hz, 1H), 2.91 (d, *J* = 15.3 Hz, 1H), 1.14 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 165.6, (d, 153.9, 153.5), 140.3, 138.0, 137.7, 137.1, 136.6, 136.6, 136.4, 133.5, 131.2, 130.8, 129.8, 128.7, 128.5, 128.4, 128.3, 127.9, 127.6, 126.8, 126.4, 125.3, 121.0, 119.9, 117.1, 58.0, 46.8, 42.4, 35.3, 22.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.2. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_2$ $[M+H]^+$ 553.2103, found 553.2093.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-2-phenyl-4-(p-tolyl)-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ja):



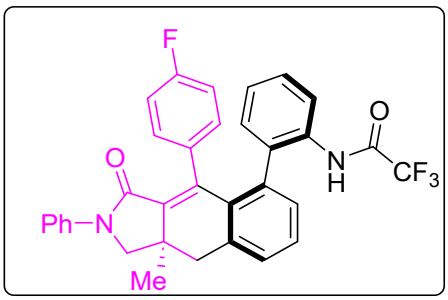
The title compound was prepared from **1j** (144.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (220.8 mg, 80% yield), 6:1 dr, mp 220-225 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.65 (d, *J* = 7.5 Hz, 1H), 7.59 (d, *J* = 7.8 Hz, 2H), 7.33 (d, *J* = 6.3 Hz, 2H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.24 (dd, *J* = 9.8, 6.2 Hz, 2H), 7.12 – 7.04 (m, 1H), 7.00 (dd, *J* = 7.5, 6.4 Hz, 2H), 6.93 (d, *J* = 7.3 Hz, 1H), 6.76 (dd, *J* = 7.5, 1.4 Hz, 1H), 6.59 (d, *J* = 4.3 Hz, 4H), 3.75 (d, *J* = 9.3 Hz, 1H), 3.70 (d, *J* = 6.7 Hz, 1H), 3.14 (d, *J* = 15.2 Hz, 1H), 2.98 (d, *J* = 15.3 Hz, 1H), 2.07 (s, 3H), 1.20 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.8, 153.9, 153.5, 141.9, 139.7, 138.0, 137.1, 136.8, 136.5, 133.6, 131.2, 130.9, 130.0, 128.7, 128.7, 128.6, 128.3, 127.2, 125.3, 124.5, 120.7, 119.7, 117.1, 114.2, 59.6, 42.7, 34.7, 22.4, 21.1. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.20. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_2$ $[M+H]^+$ 553.2103, found 553.2095.

2,2,2-trifluoro-N-(2-(4-(4-methoxyphenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ka):



The title compound was prepared from **1k** (152.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.35, SiO_2, EtOAc:hexane, 12:88) gave pure product as a white solid (230.04 mg, 81% yield), 5.1:1 dr, mp 225-230 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.58 (dd, *J* = 8.7, 1.0 Hz, 2H), 7.44 (s, 1H), 7.34 (d, *J* = 7.4 Hz, 1H), 7.31 – 7.21 (m, 3H), 7.08 (td, *J* = 7.9, 1.6 Hz, 1H), 7.02 (ddd, *J* = 8.7, 7.8, 4.3 Hz, 2H), 6.95 (d, *J* = 7.1 Hz, 1H), 6.76 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.55 (s, 1H), 6.31 (d, *J* = 7.0 Hz, 2H), 3.74 (d, *J* = 9.4 Hz, 1H), 3.70 (d, *J* = 9.2 Hz, 1H), 3.59 (s, 3H), 3.13 (d, *J* = 15.2 Hz, 1H), 2.98 (d, *J* = 15.3 Hz, 1H), 1.20 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.9, 158.2, (d, 153.87, 153.50), 141.6, 139.7, 138.1, 138.0, 137.2, 136.7, 133.6, 132.2, 131.0, 130.8, 129.9, 128.9, 128.8, 128.7, 128.4, 127.5, 125.3, 124.6, 120.8, 119.8, 119.7, 117.1, 114.2, 59.6, 55.1, 42.7, 34.8, 22.4. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.2. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_3$ $[M+H]^+$ 569.2052, found 569.2052.

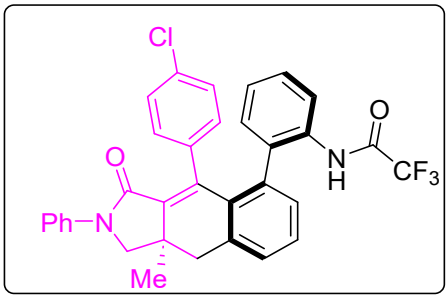
2,2,2-trifluoro-N-(2-(4-(4-fluorophenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3la):



The title compound was prepared from **1l** (144.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.4, SiO_2, EtOAc:hexane, 10:90) gave pure product as a off-white solid (214.06 mg, 77% yield), 3.3:1 dr, mp 210-215 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 8.1 Hz, 1H), 7.56 (d, *J* = 8.1 Hz, 2H), 7.36 (dd, *J* = 9.9, 6.0 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.24 (dd, *J* = 14.8, 6.6 Hz, 2H), 7.12 (t, *J* = 7.3 Hz, 1H), 7.08 – 7.00 (m, 2H), 6.97 (d, *J* = 7.1 Hz, 1H), 6.78 (d, *J* = 6.5 Hz, 2H), 6.46 (d, *J* = 7.5 Hz, 3H), 3.76 (d, *J* = 9.4 Hz, 1H), 3.71 (d, *J* = 8.9 Hz, 1H), 3.14 (d, *J* = 15.2 Hz, 1H), 3.03 (d, *J* = 9.6 Hz, 1H), 1.21 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.7, 162.6, 160.2, 153.8, 153.5, 140.6, 139.5, 138.1, 137.9, 137.4, 136.9, 136.5, 133.2, 132.3, 132.0, 131.1, 130.9, 130.0, 129.0, 128.9,

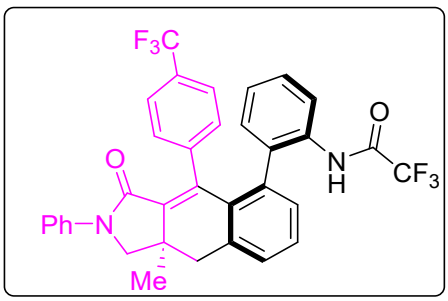
128.8, 128.7, 127.8, 125.3, 124.8, 120.8, 119.8, 117.1, 114.0, 113.8, 113.4, 59.6, 42.5, 34.8, 22.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1, -114.4. **HRMS (ESI)** calcd for $C_{33}H_{25}F_4N_2O_2$ $[M+H]^+$ 557.1852, found 557.1843.

N-(2-(4-(4-chlorophenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3ma):



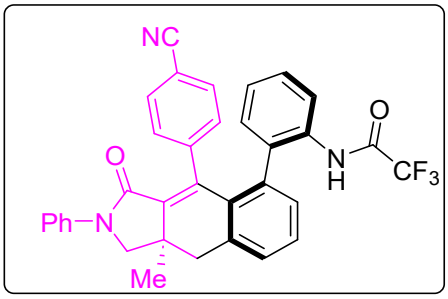
The title compound was prepared from **1m** (154.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40 SiO_2, EtOAc:hexane, 10:90) gave pure product as a brown solid (223.1 mg, 78% yield), 3:1 dr, mp 222-226 °C. **^{1}H NMR (400 MHz, $CDCl_3$) δ** 7.76 (d, *J* = 7.7 Hz, 1H), 7.56 (dd, *J* = 8.7, 0.9 Hz, 2H), 7.35 (d, *J* = 3.9 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.25 (td, *J* = 7.4, 3.7 Hz, 2H), 7.17 – 7.12 (m, 1H), 7.09 – 6.99 (m, 2H), 6.97 (d, *J* = 7.4 Hz, 1H), 6.77 (dd, *J* = 7.6, 1.4 Hz, 4H), 6.51 (s, 1H), 3.76 (d, *J* = 9.5 Hz, 1H), 3.71 (d, *J* = 9.1 Hz, 1H), 3.14 (d, *J* = 15.3 Hz, 1H), 3.03 – 2.97 (m, 1H), 1.21 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 163.5, 153.4, 152.8, 152.4, 152.0, 139.3, 138.4, 137.0, 136.9, 136.2, 135.4, 133.7, 132.0, 131.7, 130.9, 130.1, 129.9, 128.9, 128.0, 127.9, 127.7, 126.7, 125.5, 124.2, 123.8, 119.6, 118.7, 116.0, 113.1, 58.6, 41.5, 33.7, 21.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.08. **HRMS (ESI)** calcd for $C_{33}H_{25}ClF_3N_2O_2$ $[M+H]^+$ 573.1557, found 573.1579.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-2-phenyl-4-(4-(trifluoromethyl)phenyl)-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3na):



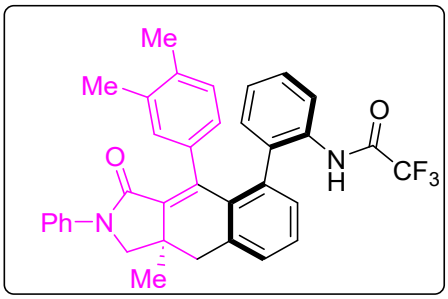
The title compound was prepared from **1n** (171.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a off-white solid (190.9 mg, 63% yield), 2.4:1 dr, mp 216-220 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.68 (d, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 7.9 Hz, 2H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.25 (dd, *J* = 15.1, 6.9 Hz, 3H), 7.17 – 7.07 (m, 2H), 7.03 (dd, *J* = 11.8, 4.2 Hz, 2H), 6.99 (dd, *J* = 12.7, 4.1 Hz, 3H), 6.78 (dd, *J* = 7.5, 1.4 Hz, 1H), 6.61 (t, *J* = 7.5 Hz, 1H), 3.79 (d, *J* = 9.5 Hz, 1H), 3.72 (d, *J* = 9.6 Hz, 1H), 3.16 (d, *J* = 15.4 Hz, 1H), 3.03 (d, *J* = 15.3 Hz, 1H), 1.18 (s, 3H**). ^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.5, (d, 153.7, 153.2), 140.2, 140.0, 139.3, 138.3, 138.1, 137.0, 136.9, 136.3, 134.3, 132.6, 131.9, 131.2, 131.1, 130.6, 130.1, 129.2, 128.8, 128.0, 125.7, 125.3, 125.0, 123.2, 120.6, 119.9, 117.5, 113.7, 59.7, 42.5, 34.8, 22.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -62.9, -76.1. **HRMS (ESI)** calcd for $C_{34}H_{25}F_6N_2O_2$ $[M+H]^+$ 607.1820, found 607.1815.

N-(2-(4-(4-cyanophenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3oa):



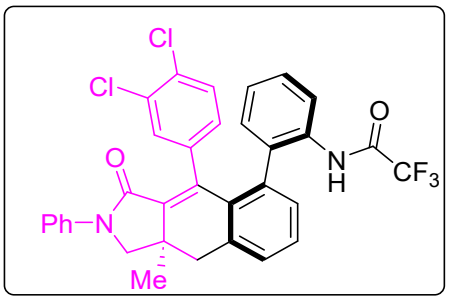
The title compound was prepared from **1o** (150.0 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.38, SiO_2, EtOAc:hexane, 13:83) gave pure product as an off-white solid (154.82 mg, 55% yield), 2.3:1 dr, mp 230-235 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.73 (d, *J* = 8.2 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 2H), 7.39 (dd, *J* = 9.9, 4.7 Hz, 2H), 7.30 – 7.22 (m, 3H), 7.15 – 7.02 (m, 4H), 6.99 (d, *J* = 7.2 Hz, 2H), 6.80 (dd, *J* = 7.6, 1.5 Hz, 1H), 6.69 (t, *J* = 7.5 Hz, 2H), 3.78 (d, *J* = 9.3 Hz, 1H), 3.73 (d, *J* = 6.6 Hz, 1H), 3.15 (d, *J* = 15.5 Hz, 1H), 3.03 (d, *J* = 15.5 Hz, 1H), 1.23 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.2, (d, 153.7, 153.3), 141.1, 139.5, 139.2, 138.7, 138.2, 136.2, 133.9, 132.7, 132.5, 131.9, 131.3, 130.7, 130.1, 129.9, 129.4, 129.3, 129.1, 128.9, 128.3, 125.3, 125.2, 120.5, 119.9, 118.6, 110.4, 59.6, 42.4, 34.8, 22.3. **HRMS (ESI)** calcd for $C_{34}H_{25}F_3N_3O_2$ $[M+H]^+$ 564.1899, found 564.1910.

N-(2-(4-(3,4-dimethylphenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3pa):



The title compound was prepared from **1p** (151.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (212.25 mg, 75% yield), 7.8:1 dr, mp 196-200 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.73 – 7.65 (m, 3H), 7.44 – 7.38 (m, 2H), 7.37 – 7.29 (m, 4H), 7.14 (dt, *J* = 12.8, 7.1 Hz, 3H), 7.01 (d, *J* = 7.3 Hz, 1H), 6.87 (d, *J* = 7.3 Hz, 1H), 6.64 (s, 2H), 6.40 (s, 1H), 3.84 (d, *J* = 9.6 Hz, 1H), 3.80 (d, *J* = 9.5 Hz, 1H), 3.24 (d, *J* = 15.4 Hz, 1H), 3.07 (d, *J* = 15.2 Hz, 1H), 2.07 (s, 3H), 1.96 (s, 3H), 1.30 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.9, (d, 154.0, 153.6), 141.9, 139.7, 137.8, 136.9, 136.8, 134.3, 133.6, 131.3, 130.8, 130.0, 129.4, 128.7, 128.5, 128.1, 127.8, 125.2, 124.5, 120.8, 119.7, 114.2, 59.6, 42.7, 34.7, 22.5, 19.5, 19.4. **HRMS (ESI)** calcd for $C_{35}H_{30}F_3N_2O_2$ $[M+H]^+$ 567.2259, found 567.2252.

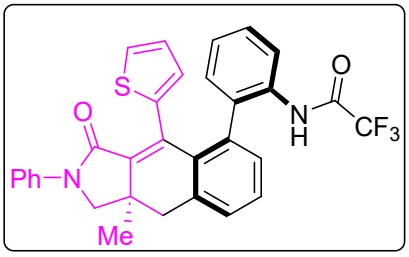
N-(2-(4-(3,4-dichlorophenyl)-9a-methyl-3-oxo-2-phenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3qa):



The title compound was prepared from **1q** (171.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure A. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave

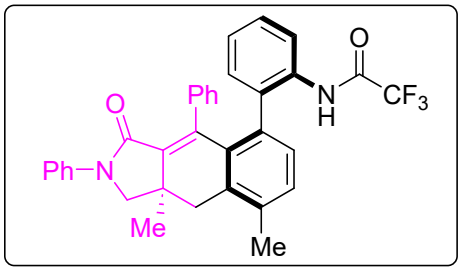
pure product as an off-white solid (218.2 mg, 72% yield), 4.2:1 dr, mp 190-194 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81 (d, *J* = 8.5 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.44 – 7.31 (m, 3H), 7.30 – 7.23 (m, 2H), 7.19 (s, 2H), 7.09 – 7.05 (m, 2H), 7.00 (d, *J* = 7.0 Hz, 1H), 6.84 – 6.77 (m, 2H), 6.66 (s, 1H), 3.77 (d, *J* = 9.3 Hz, 1H), 3.72 (d, *J* = 9.2 Hz, 1H), 3.14 (d, *J* = 15.4 Hz, 1H), 3.01 (d, *J* = 15.4 Hz, 1H), 1.21 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.4, 154.5, 153.8, 139.3, 138.8, 138.5, 138.1, 136.6, 136.3, 132.2, 131.2, 131.1, 130.9, 130.0, 129.8, 129.2, 128.9, 128.2, 127.9, 125.3, 125.0, 120.5, 119.9, 117.0, 114.2, 59.7, 42.4, 34.7, 22.4. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -75.5. **HRMS (ESI)** calcd for $C_{33}H_{24}Cl_2F_3N_2O_2[M+H]^+$ 607.1167, found 607.1176.

2,2,2-trifluoro-N-(2-(9a-methyl-3-oxo-2-phenyl-4-(thiophen-2-yl)-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ra):



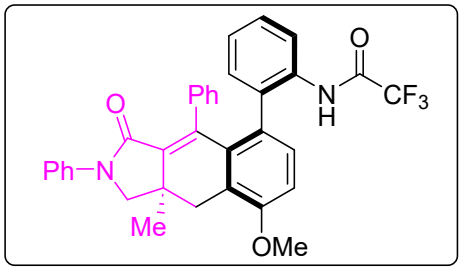
The title compound was prepared from **1r** (140.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a pale yellow solid (160.5 mg, 59% yield), 5.6:1 dr, mp 155-160 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.76 (dd, *J* = 8.2, 0.9 Hz, 1H), 7.66 (s, 1H), 7.61 (dd, *J* = 8.8, 1.0 Hz, 2H), 7.31 (t, *J* = 4.2 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.14 (td, *J* = 7.9, 1.5 Hz, 1H), 7.08 – 7.05 (m, 1H), 7.05 – 7.02 (m, 1H), 7.00 – 6.98 (m, 1H), 6.87 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.83 (dd, *J* = 7.6, 1.5 Hz, 1H), 3.77 (d, *J* = 9.3 Hz, 1H), 3.71 (d, *J* = 9.3 Hz, 1H), 3.13 (d, *J* = 15.2 Hz, 1H), 2.96 (d, *J* = 15.2 Hz, 1H), 1.19 (s, 3H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 164.3, 154.3, 154.0, 139.6, 139.5, 137.7, 137.6, 137.1, 137.0, 134.4, 133.2, 132.4, 131.5, 130.6, 130.1, 129.4, 128.9, 128.8, 128.6, 128.5, 127.5, 126.2, 125.7, 125.5, 125.1, 124.8, 121.7, 119.8, 116.9, 59.6, 42.4, 35.6, 22.1. **HRMS (ESI)** calcd for $C_{31}H_{24}F_3N_2O_3S$ $[M+H]^+$ 545.1511, found 545.1506.

N-(2-(8,9a-dimethyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3ab):



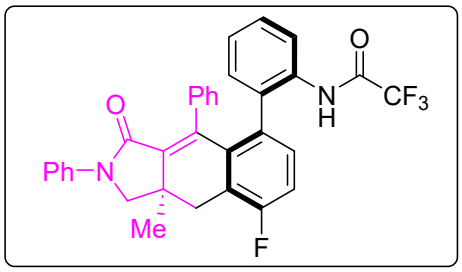
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2b** (139.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as white solid (212.5 mg, 77% yield), 7.5:1 dr, mp 215-220 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.36 (s, 1H), 7.24 (t, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.11 – 6.99 (m, 3H), 6.86 (d, *J* = 7.6 Hz, 2H), 6.78 (dd, *J* = 7.4, 1.5 Hz, 3H), 6.51 (s, 1H), 3.79 (d, *J* = 9.3 Hz, 1H), 3.73 (d, *J* = 9.4 Hz, 1H), 3.16 (d, *J* = 15.7 Hz, 1H), 2.82 (d, *J* = 15.8 Hz, 1H), 2.41 (s, 3H), 1.21 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.8, (d, 153.9, 153.4), 142.1, 139.6, 137.9, 136.8, 136.6, 136.6, 136.1, 134.5, 133.7, 131.4, 130.9, 130.7, 129.1, 128.7, 128.3, 126.7, 126.3, 125.4, 124.6, 121.0, 119.7, 117.6, 113.8, 59.8, 38.8, 34.7, 22.9, 20.1. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_2$ $[M+H]^+$ 553.2103, found 553.2095.

2,2,2-trifluoro-N-(2-(8-methoxy-9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ac):



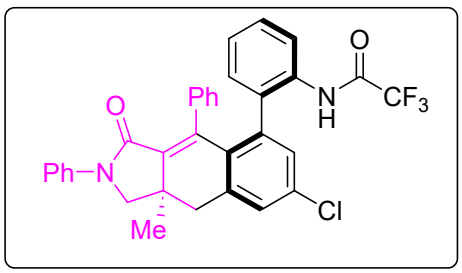
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2c** (147.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.35, SiO_2, EtOAc:hexane, 15:85) gave pure product as an off-white solid (224.4 mg, 79% yield), 6.9:1 dr, mp 224-227 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.62 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.38 (s, 1H), 7.28 – 7.21 (m, 2H), 7.03 (dt, *J* = 15.1, 7.6 Hz, 4H), 6.91 (s, 1H), 6.86 (d, *J* = 6.7 Hz, 2H), 6.77 (d, *J* = 7.2 Hz, 3H), 6.55 (s, 1H), 3.91 (s, 3H), 3.77 (d, *J* = 8.9 Hz, 1H), 3.71 (d, *J* = 9.1 Hz, 1H), 3.47 (d, *J* = 16.2 Hz, 1H), 2.64 (d, *J* = 16.3 Hz, 1H), 1.20 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.8, 156.7, 153.9, 153.4, 141.7, 139.6, 138.8, 137.3, 136.6, 133.4, 131.7, 131.1, 130.1, 128.8, 128.7, 128.2, 126.7, 126.3, 125.3, 124.6, 120.9, 119.8, 117.6, 115.1, 110.7, 59.9, 55.8, 34.8, 34.4, 22.8. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{34}H_{27}F_3N_2O_3$ $[M+H]^+$ 569.2050, found 569.2052.

2,2,2-trifluoro-N-(2-(8-fluoro-9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ad):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2d** (141.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (212.3 mg, 76% yield), 7.5:1 dr, mp 216-218 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.58 (d, *J* = 1.6 Hz, 1H), 7.58 – 7.55 (m, 2H), 7.28 – 7.22 (m, 2H), 7.19 (s, 1H), 7.12 – 7.06 (m, 2H), 7.05 – 7.01 (m, 2H), 6.95 – 6.85 (m, 3H), 6.77 (dd, *J* = 7.5, 1.4 Hz, 3H), 6.53 (s, 1H), 3.80 (d, *J* = 9.5 Hz, 1H), 3.74 (d, *J* = 9.3 Hz, 2H), 3.39 (d, *J* = 15.6 Hz, 1H), 2.79 (d, *J* = 16.0 Hz, 1H), 1.23 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.4, 161.8, 158.5, (d, 154.1, 153.6), 140.9, 139.7, 139.4, 138.1, 136.0, 132.9, 132.7, 132.5, 131.5, 130.9, 130.8, 128.8, 127.0, 126.5, 125.6, 124.8, 121.4, 119.8, 116.1, 115.8, 113.7, 59.6, 34.4, 34.0, 22.8. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1, -117.2. **HRMS (ESI)** calcd for $C_{33}H_{25}F_4N_2O_2$ $[M+H]^+$ 557.1852, found 557.1845.

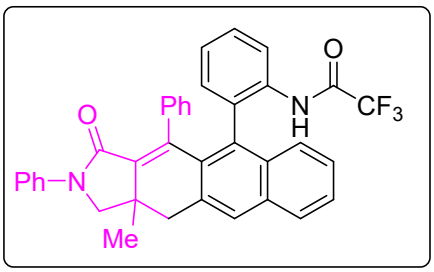
N-(2-(7-chloro-9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3ae):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2e** (149.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 12:88) gave

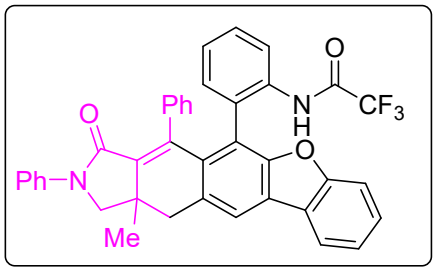
pure product as a pale yellow solid (194.5 mg, 68% yield), 4.7:1 dr, mp 240-244 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.68 – 7.57 (m, 3H), 7.42 (s, 1H), 7.35 – 7.28 (m, 2H), 7.22 (s, 1H), 7.19 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.14 (dd, *J* = 4.0, 1.6 Hz, 1H), 7.10 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.03 (d, *J* = 2.1 Hz, 1H), 6.95 (d, *J* = 7.0 Hz, 2H), 6.84 (dd, *J* = 7.4, 1.6 Hz, 3H), 6.62 (s, 1H), 3.83 (d, *J* = 9.5 Hz, 1H), 3.78 (d, *J* = 6.0 Hz, 1H), 3.20 (d, *J* = 15.5 Hz, 1H), 3.04 (d, *J* = 15.5 Hz, 1H), 1.29 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.4, 154.1, 153.7, 140.7, 139.7, 139.4, 138.4, 137.7, 136.2, 135.0, 134.3, 132.5, 131.0, 130.7, 129.9, 129.0, 128.7, 127.0, 126.6, 125.6, 124.8, 121.6, 119.8, 59.4, 42.4, 34.8, 22.4. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{33}H_{25}F_3ClN_2O_2$ $[M+H]^+$ 573.1557, found 573.1545.

2,2,2-trifluoro-N-(2-(11a-methyl-3-oxo-2,4-diphenyl-2,3,11,11a-tetrahydro-1H-naphtho[2,3-f]isoindol-5-yl)phenyl)acetamide (3af):



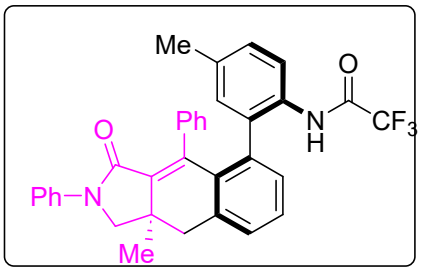
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2f** (157.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a pale yellow solid (241.1 mg, 82% yield), mp 200-205 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93 – 7.83 (m, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.35 – 7.28 (m, 4H), 7.24 – 7.18 (m, 1H), 7.11 (t, *J* = 8.3 Hz, 2H), 6.97 (dd, *J* = 14.3, 8.9 Hz, 4H), 6.84 (s, 2H), 6.60 (s, 1H), 3.90 (d, *J* = 9.2 Hz, 1H), 3.78 (d, *J* = 9.2 Hz, 1H), 3.36 (d, *J* = 14.7 Hz, 1H), 3.23 (d, *J* = 14.4 Hz, 1H), 1.28 (s, 3H).**^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.6, (q, 154.1, 153.8, 153.4, 153.0), 142.4, 139.6, 139.4, 136.9, 136.8, 136.4, 134.2, 133.7, 133.1, 132.5, 131.8, 131.6, 131.5, 131.0, 129.1, 128.7, 128.5, 128.1, 127.8, 127.8, 127.4, 127.1, 126.6, 126.2, 126.2, 125.2, 124.9, 124.7, 120.8, 119.8, 116.8, 113.8, 59.3, 43.5, 35.5, 22.8. **HRMS (ESI)** calcd for $C_{37}H_{28}F_3N_2O_2$ $[M+H]^+$ 589.2103, found 589.2109.

2,2,2-trifluoro-N-(2-(12a-methyl-3-oxo-2,4-diphenyl-2,3,12,12a-tetrahydro-1H-benzo[2,3]benzofuro[5,6-f]isoindol-5-yl)phenyl)acetamide (3ag):



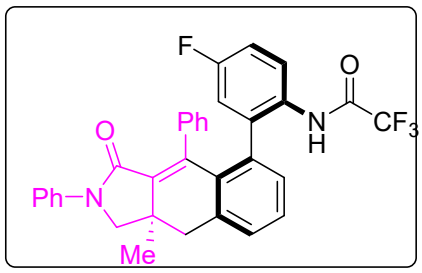
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2g** (177.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (204.1 mg, 65% yield), mp 202-206 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.02 (d, *J* = 7.7 Hz, 1H), 7.97 – 7.91 (m, 2H), 7.56 (dd, *J* = 8.6, 0.9 Hz, 2H), 7.37 (dt, *J* = 8.5, 1.5 Hz, 1H), 7.35 – 7.30 (m, 2H), 7.24 (dd, *J* = 15.2, 7.7 Hz, 3H), 7.15 – 7.09 (m, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.90 (dd, *J* = 12.7, 5.5 Hz, 3H), 6.81 (s, 1H), 6.73 (td, *J* = 7.6, 1.1 Hz, 1H), 6.59 (dd, *J* = 7.7, 1.5 Hz, 2H), 3.78 (d, *J* = 9.3 Hz, 1H), 3.72 (d, *J* = 9.2 Hz, 1H), 3.30 (d, *J* = 14.7 Hz, 1H), 3.18 (d, *J* = 14.9 Hz, 1H), 1.19 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.4, 157.0, 154.7, 154.1, 153.7, 141.9, 139.5, 138.5, 137.8, 135.8, 133.7, 132.9, 132.0, 131.9, 131.7, 129.2, 128.7, 128.7, 128.4, 128.2, 127.3, 126.9, 126.5, 126.0, 125.8, 125.6, 125.0, 124.7, 123.4, 123.3, 121.5, 121.0, 120.5, 120.3, 119.8, 116.9, 114.0, 112.2, 59.6, 43.3, 34.7, 22.0. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -75.4. **HRMS (ESI)** calcd for $C_{39}H_{28}F_3N_2O_3$ $[M+H]^+$ 629.2052, found 629.2039.

2,2,2-trifluoro-N-(4-methyl-2-(9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ah):



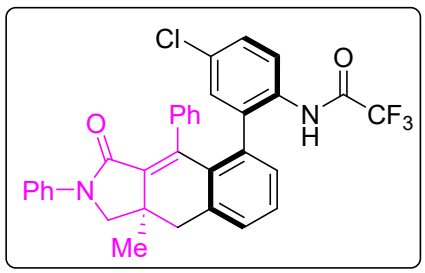
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2h** (139.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (204.24 mg, 74% yield), 4.6:1 dr, mp 193-198 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.58 (d, *J* = 7.9 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.33 (d, *J* = 7.3 Hz, 1H), 7.29 – 7.26 (m, 1H), 7.23 (d, *J* = 8.3 Hz, 2H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.90 (dd, *J* = 22.9, 7.7 Hz, 4H), 6.79 (d, *J* = 8.5 Hz, 2H), 6.58 (s, 2H), 3.76 (d, *J* = 9.3 Hz, 1H), 3.71 (d, *J* = 9.3 Hz, 1H), 3.14 (d, *J* = 15.2 Hz, 1H), 2.99 (d, *J* = 15.3 Hz, 1H), 2.24 (s, 3H), 1.22 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 164.8, 153.9, 153.5, 141.8, 139.6, 137.8, 137.4, 137.3, 137.0, 136.6, 135.2, 133.6, 131.7, 131.2, 130.1, 129.4, 128.9, 128.7, 128.7, 128.4, 128.3, 126.8, 126.7, 126.4, 124.7, 121.3, 119.8, 117.1, 114.3, 59.7, 42.7, 34.8, 22.5, 20.9. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{34}H_{28}F_3N_2O_2$ $[M+H]^+$ 553.2103, found 553.2075.

2,2,2-trifluoro-N-(4-fluoro-2-(9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ai):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2i** (141.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.4, SiO_2, EtOAc:hexane, 10:90) gave pure product as a white solid (200.2 mg, 72% yield), 6.6:1 dr, mp 206-210 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.60 – 7.53 (m, 3H), 7.37 (d, *J* = 7.3 Hz, 1H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.28 – 7.21 (m, 3H), 7.04 (t, *J* = 7.4 Hz, 1H), 6.90 (dd, *J* = 16.9, 9.4 Hz, 5H), 6.82 – 6.74 (m, 1H), 6.53 (dd, *J* = 8.6, 2.9 Hz, 2H), 3.76 (d, *J* = 9.4 Hz, 1H), 3.71 (d, *J* = 9.3 Hz, 1H), 3.15 (d, *J* = 15.4 Hz, 1H), 3.00 (d, *J* = 15.4 Hz, 1H), 1.22 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.6, 161.1, 158.6, 153.7, 141.0, 139.5, 138.1, 137.8, 137.4, 136.6, 135.7, 135.6, 131.5, 129.8, 129.6, 129.2, 128.9, 128.7, 127.0, 126.6, 124.7, 123.2, 123.2, 119.8, 118.1, 117.9, 115.0, 114.8, 59.6, 42.6, 34.8, 22.5. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1, -116.4. **HRMS (ESI)** calcd for $C_{33}H_{25}F_4N_2O_2$ $[M+H]^+$ 557.1852, found 557.1841.

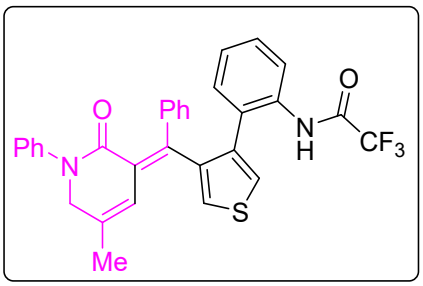
N-(4-chloro-2-(9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)-2,2,2-trifluoroacetamide (3aj):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2j** (149.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.4, SiO_2, EtOAc:hexane, 10:90) gave

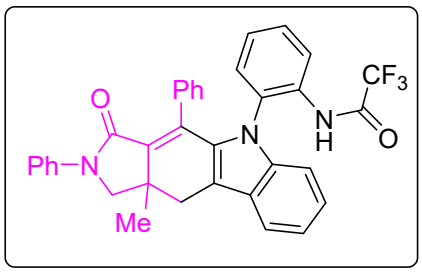
pure product as an off-white solid (69.44 mg, 69% yield), 3.8:1 dr, mp 190-195 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.58 (dd, *J* = 13.7, 4.8 Hz, 3H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.24 (dd, *J* = 11.4, 4.7 Hz, 2H), 7.15 (s, 1H), 7.08 – 7.01 (m, 3H), 6.95 – 6.83 (m, 4H), 6.77 (d, *J* = 2.4 Hz, 1H), 6.52 (s, 1H), 3.76 (d, *J* = 9.4 Hz, 1H), 3.72 (d, *J* = 7.2 Hz, 1H), 3.15 (d, *J* = 15.3 Hz, 1H), 3.01 (d, *J* = 15.4 Hz, 1H), 1.23 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.6, (d, 153.9, 153.5), 140.9, 139.5, 138.2, 138.0, 137.4, 136.5, 135.2, 134.9, 134.5, 131.5, 130.8, 130.7, 129.8, 129.6, 129.4, 128.9, 128.7, 128.2, 126.9, 126.6, 124.7, 122.2, 121.0, 119.8, 117.0, 59.6, 42.6, 34.7, 22.5. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -76.1. **HRMS (ESI)** calcd for $C_{33}H_{25}ClF_3N_2O_2$ $[M+H]^+$ 573.1557, found 573.1549.

(E)-2,2,2-trifluoro-N-(2-(4-((5-methyl-2-oxo-1-phenyl-1,6-dihydropyridin-3(2H)-ylidene)(phenyl)methyl)thiophen-3-yl)phenyl)acetamide (3ak):



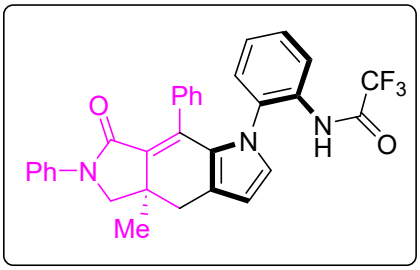
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2k** (135.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a yellow solid (122.4 mg, 45% yield), m160-163 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.58 (s, 1H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.35 – 7.25 (m, 8H), 7.21 (d, *J* = 5.2 Hz, 2H), 7.08 (td, *J* = 7.5, 1.1 Hz, 1H), 7.02 (t, *J* = 7.3 Hz, 3H), 6.68 (d, *J* = 5.1 Hz, 1H), 6.23 (d, *J* = 1.4 Hz, 1H), 3.86 (s, 2H), 1.61 (d, *J* = 1.0 Hz, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 163.1, 155.7, 155.2, 151.7, 143.0, 141.6, 141.2, 138.2, 136.4, 133.8, 132.3, 130.8, 130.6, 129.8, 129.7, 128.9, 128.6, 128.5, 128.4, 127.9, 127.4, 127.3, 126.3, 126.1, 125.9, 124.6, 124.0, 122.8, 117.5, 113.6, 56.1, 20.2. **HRMS (ESI)** calcd for $C_{31}H_{24}F_3N_2O_2S$ $[M+H]^+$ 545.1511, found 545.1497.

2,2,2-trifluoro-N-(2-(10a-methyl-3-oxo-2,4-diphenyl-2,3,10,10a-tetrahydropyrrolo[3,4-b]carbazol-5(1H)-yl)phenyl)acetamide (3al):



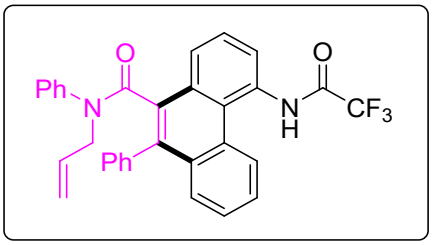
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2l** (152.0 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.50, SiO_2, EtOAc:hexane, 10:90) gave pure product as a yellow solid (150.02 mg, 52% yield), mp 190-194 °C. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.92 (dd, *J* = 8.3, 1.2 Hz, 1H), 7.72 (s, 1H), 7.62 (dd, *J* = 9.6, 8.3 Hz, 3H), 7.28 – 7.20 (m, 3H), 7.19 – 7.12 (m, 3H), 7.07 – 7.00 (m, 2H), 6.96 (d, *J* = 6.0 Hz, 2H), 6.82 (d, *J* = 7.2 Hz, 2H), 6.71 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.67 (d, *J* = 8.0 Hz, 1H), 3.78 (s, 2H), 3.25 (d, *J* = 16.2 Hz, 1H), 3.07 (d, *J* = 16.3 Hz, 1H), 1.34 (s, 3H). **^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)** δ 164.8, (d, 154.3, 153.9), 141.5, 139.8, 138.1, 134.9, 134.8, 133.7, 132.0, 129.4, 128.9, 128.7, 127.7, 127.4, 127.3, 126.0, 124.9, 124.5, 122.2, 120.6, 119.9, 119.7, 117.4, 110.8, 59.9, 36.0, 33.0, 24.0. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -75.7. **HRMS (ESI)** calcd for $C_{35}H_{27}F_3N_3O_2$ $[M+H]^+$ 578.2055, found 578.2053.

2,2,2-trifluoro-N-(2-(4a-methyl-7-oxo-6,8-diphenyl-4a,5,6,7-tetrahydropyrrolo[3,4-f]indol-1(4H)-yl)phenyl)acetamide (3am):



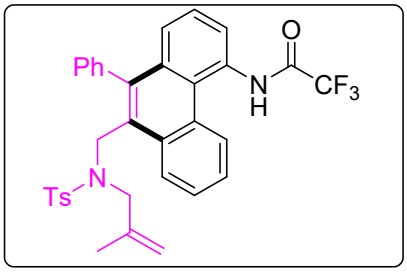
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2m** (127.0 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.50, SiO_2, EtOAc:hexane, 10:90) gave pure product as yellow solid (202.9 mg, 77% yield), 1.8:1 dr, mp 172-175°C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.86 (d, *J* = 8.2 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.21 (dd, *J* = 13.2, 4.6 Hz, 3H), 7.08 – 6.95 (m, 3H), 6.91 (d, *J* = 12.1 Hz, 2H), 6.87 – 6.78 (m, 3H), 6.74 (dd, *J* = 27.5, 5.8 Hz, 1H), 6.60 (d, *J* = 2.6 Hz, 1H), 6.35 (d, *J* = 2.5 Hz, 1H), 3.66 (d, *J* = 7.9 Hz, 2H), 2.91 (d, *J* = 6.1 Hz, 2H), 1.25 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 165.2, (d, 154.4, 154.1), 140.0, 134.7, 133.6, 131.9, 131.0, 130.3, 130.0, 129.3, 129.0, 128.6, 128.2, 127.9, 127.2, 126.8, 126.4, 125.6, 124.2, 123.7, 120.4, 119.6, 116.8, 110.3, 59.9, 35.8, 34.9, 23.4. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -75.6. **HRMS (ESI)** calcd for $C_{31}H_{25}F_3N_3O_2$ $[M+H]^+$ 528.1899, found 528.1893.

N-allyl-N,10-diphenyl-5-(2,2,2-trifluoroacetamido)phenanthrene-9-carboxamide (3sa):



The title compound was prepared from **1s** (130.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 10:90) gave pure product as a yellow solid (157.2 mg, 60% yield), mp 178-182 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 8.2 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 3H), 7.22 (d, *J* = 6.6 Hz, 2H), 7.12 (t, *J* = 7.3 Hz, 2H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.62 (d, *J* = 7.5 Hz, 2H), 6.42 (s, 1H), 5.19 (s, 1H), 4.79 (s, 1H), 4.54 (d, *J* = 13.7 Hz, 1H), 4.45 (d, *J* = 13.7 Hz, 1H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 165.6, (d, 154.4, 153.9, 150.2, 142.7, 139.0, 138.5, 137.2, 135.7, 132.6, 132.3, 131.1, 129.6, 129.4, 129.2, 129.1, 129.0, 128.9, 128.6, 128.2, 127.0, 126.1, 124.7, 121.6, 120.3, 119.7, 111.5, 51.3. **HRMS (ESI)** calcd for $C_{32}H_{24}F_3N_2O_2$ $[M+H]^+$ 525.1790, found 525.1773.

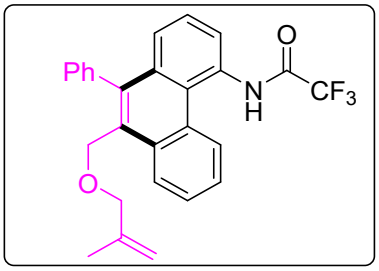
2,2,2-trifluoro-N-(10-(((4-methyl-N-(2-methylallyl)phenyl)sulfonamido)methyl)-9-phenylphenanthren-4-yl)acetamide (3ta):



The title compound was prepared from **1t** (169.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.40, SiO_2, EtOAc:hexane, 12:88) gave pure product as white solid (222.74 mg, 74% yield), mp 210-215 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.72 (t, *J* = 7.5 Hz, 2H), 8.60 (s, 1H), 8.11 (d, *J* = 7.6 Hz, 1H), 7.71 (dt, *J* = 14.3, 6.9 Hz, 2H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.51 – 7.38 (m, 4H), 7.30 (d, *J* = 10.2 Hz, 1H), 7.24 (d, *J* = 7.9 Hz, 2H), 7.13 (d, *J* = 4.6 Hz, 2H), 4.61 (s, 2H), 4.24 (s, 1H), 4.18 (s, 1H), 3.24 (s, 2H), 2.45 (s, 3H), 1.24 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ (d, 155.4,

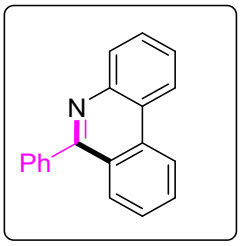
154.9), 143.4, 141.1, 139.9, 138.3, 134.0, 130.6, 130.4, 129.5, 128.5, 127.9, 127.7, 127.7, 127.6, 127.1, 126.6, 126.2, 124.7, 124.4, 118.1, 114.3, 112.4, 53.6, 49.3, 21.5, 19.8. **HRMS (ESI)** calcd for $C_{34}H_{30}F_3N_2O_3S$ $[M+H]^+$ 603.1929, found 603.1896.

2,2,2-trifluoro-N-(10-(((2-methylallyl)oxy)methyl)-9-phenylphenanthren-4-yl)acetamide (3ua):



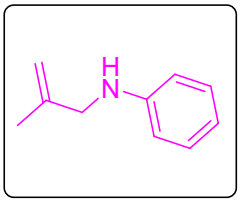
The title compound was prepared from **1u** (137.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.50, SiO_2, EtOAc:hexane, 10:90) gave pure product as an off-white solid (179.6 mg, 80% yield), mp 158-160 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.66 (d, *J* = 7.8 Hz, 1H), 8.50 (s, 1H), 8.33 (dd, *J* = 8.3, 1.1 Hz, 1H), 8.02 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.65 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 7.58 (ddd, *J* = 8.4, 7.0, 1.4 Hz, 1H), 7.44 (ddd, *J* = 6.8, 4.1, 2.4 Hz, 2H), 7.39 (dd, *J* = 13.1, 5.0 Hz, 1H), 7.32 (dd, *J* = 8.3, 1.3 Hz, 1H), 7.28 – 7.23 (m, 2H), 7.20 – 7.17 (m, 1H), 4.80 (d, *J* = 1.0 Hz, 1H), 4.74 (s, 1H), 4.55 (s, 2H), 3.76 (s, 2H), 1.60 (s, 3H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 155.4, 154.9, 142.0, 139.3, 138.7, 134.0, 132.5, 130.7, 130.5, 128.3, 127.7, 127.7, 127.5, 126.7, 126.3, 126.2, 124.9, 124.2, 124.0, 118.1, 114.3, 112.7, 74.8, 67.5, 19.6. **HRMS (ESI)** calcd for $C_{27}H_{21}F_3NO_2$ $[M-H]^+$ 448.1525 found 448.1511.

6-phenylphenanthridine (3an):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2n** (84.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.60, SiO_2, EtOAc:hexane, 8:92) gave pure product as a pale brown solid (71.4 mg, 56% yield), mp 110-115 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.65 (d, *J* = 8.3 Hz, 1H), 8.56 (d, *J* = 7.9 Hz, 1H), 8.24 (d, *J* = 7.6 Hz, 1H), 8.05 (d, *J* = 8.2 Hz, 1H), 7.81 (t, *J* = 7.3 Hz, 1H), 7.74 – 7.61 (m, 4H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.45 (m, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 161.2, 143.1, 139.0, 133.6, 131.0, 129.9, 129.2, 129.1, 129.1, 128.5, 127.3, 127.2, 125.2, 123.8, 122.29, 122.0. **HRMS (ESI)** calcd for $C_{19}H_{14}N$ $[M+H]^+$ 256.1126, found 256.1099.

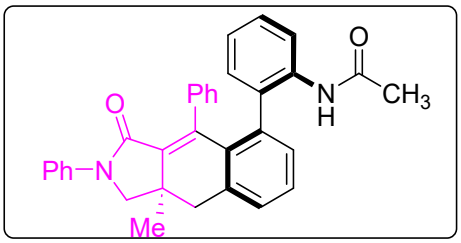
N-(2-methylallyl)aniline (3an'):



The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2n** (84.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.45, SiO_2, EtOAc:hexane, 12:88) gave pure product as an orange solid (11.2 mg, 16% yield). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55 (d, *J* = 7.1 Hz, 1H), 7.49 (td, *J* = 7.8, 1.1 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 7.9 Hz, 1H), 4.93 (s, 1H), 4.89 (s, 1H), 4.23

(s, 2H), 1.70 (s, 3H). **$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)** δ 183.2, 158.1, 151.1, 138.3, 125.3, 123.8, 113.5, 111.1, 46.1, 19.9.

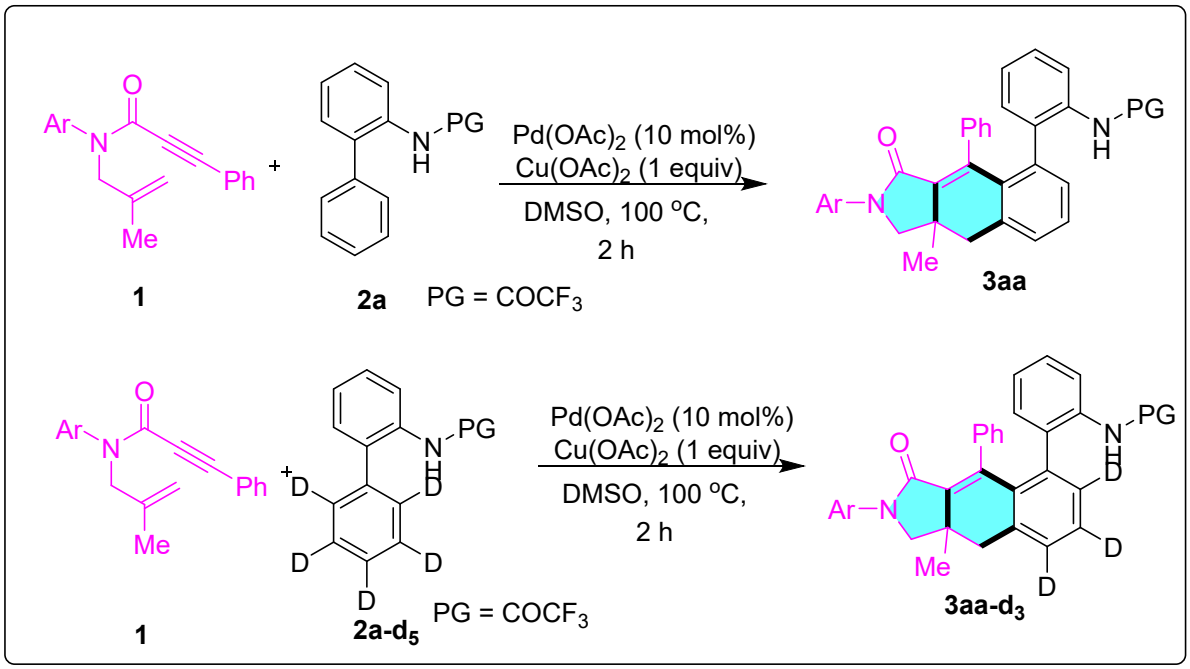
N-(2-(9a-methyl-3-oxo-2,4-diphenyl-2,3,9,9a-tetrahydro-1H-benzo[f]isoindol-5-yl)phenyl)acetamide (3ao):



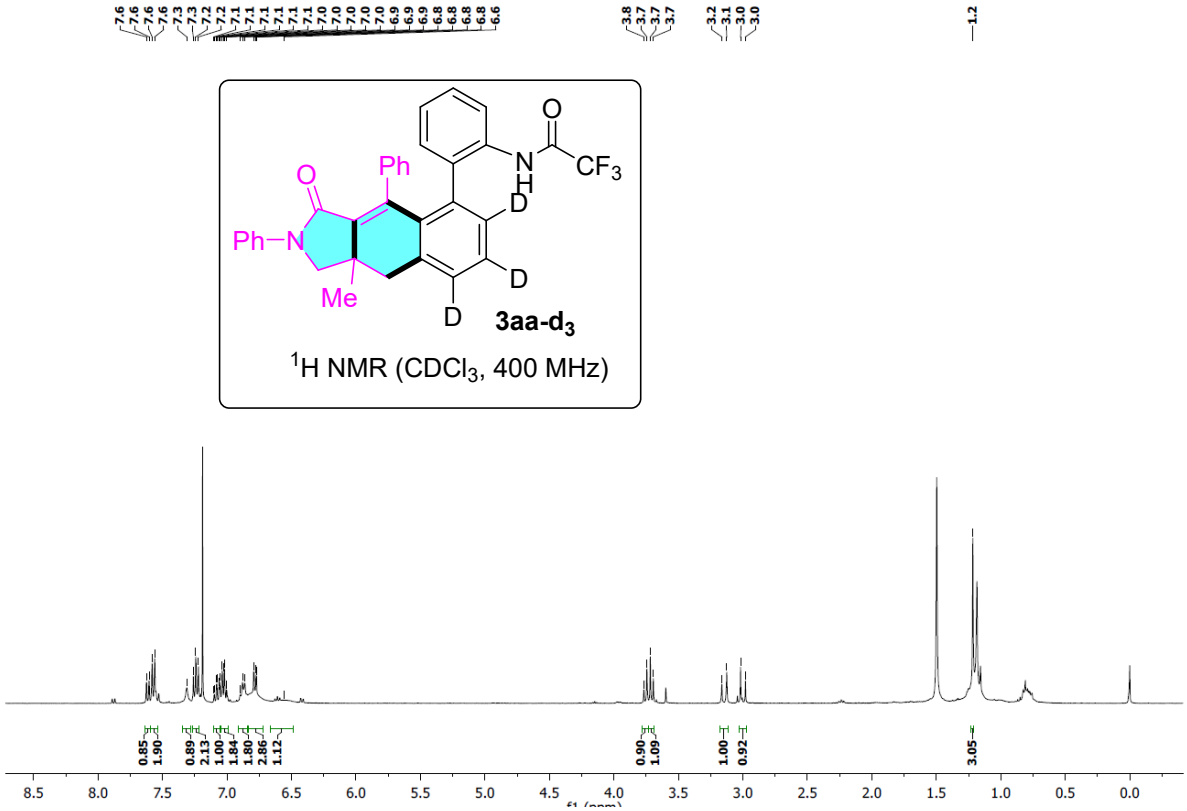
The title compound was prepared from **1a** (137.5 mg, 0.5 mmol) and **2o** (105.5 mg, 0.5 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.3, SiO_2, EtOAc:hexane, 20:80) gave pure product as an off-white solid (72.6 mg, 30% yield), 3:1 dr, mp 280-284 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 – 7.54 (m, 3H), 7.36 – 7.27 (m, 2H), 7.24 (dd, *J* = 11.4, 4.7 Hz, 3H), 7.01 (dt, *J* = 14.1, 7.1 Hz, 3H), 6.93 – 6.77 (m, 5H), 6.69 (dd, *J* = 7.5, 1.3 Hz, 1H), 6.39 (s, 1H), 3.75 (d, *J* = 9.3 Hz, 1H), 3.70 (d, *J* = 9.3 Hz, 1H), 3.13 (d, *J* = 15.0 Hz, 1H), 2.98 (d, *J* = 15.2 Hz, 1H), 1.91 (s, 1H), 1.22 (s, 1H). **$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)** δ 167.5, 164.8, 142.2, 139.7, 138.4, 137.7, 137.6, 137.0, 136.5, 133.4, 132.5, 131.0, 130.6, 128.7, 128.4, 128.2, 128.1, 127.1, 126.7, 126.6, 126.2, 124.6, 123.6, 121.5, 119.7, 59.6, 42.8, 34.8, 24.7, 22.4f. **HRMS (ESI)** calcd for $C_{33}H_{29}N_2O_2$ $[M+H]^+$ 485.2229, found 485.2245.

5. KIE studies:

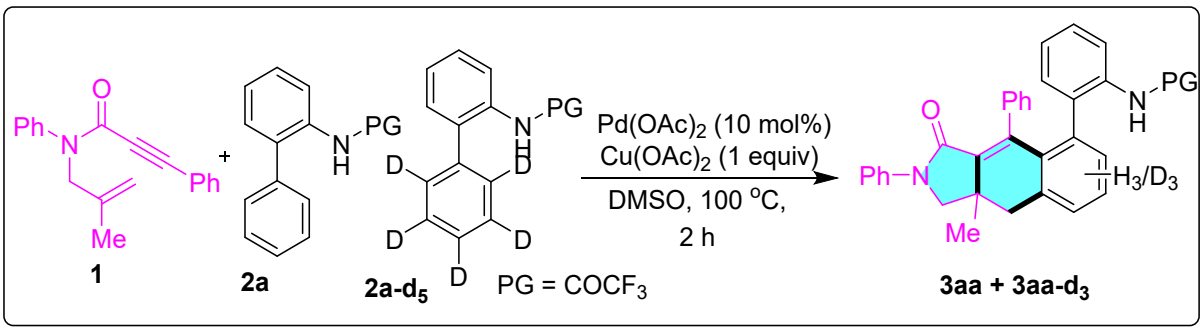
KIE determined from two parallel reactions:



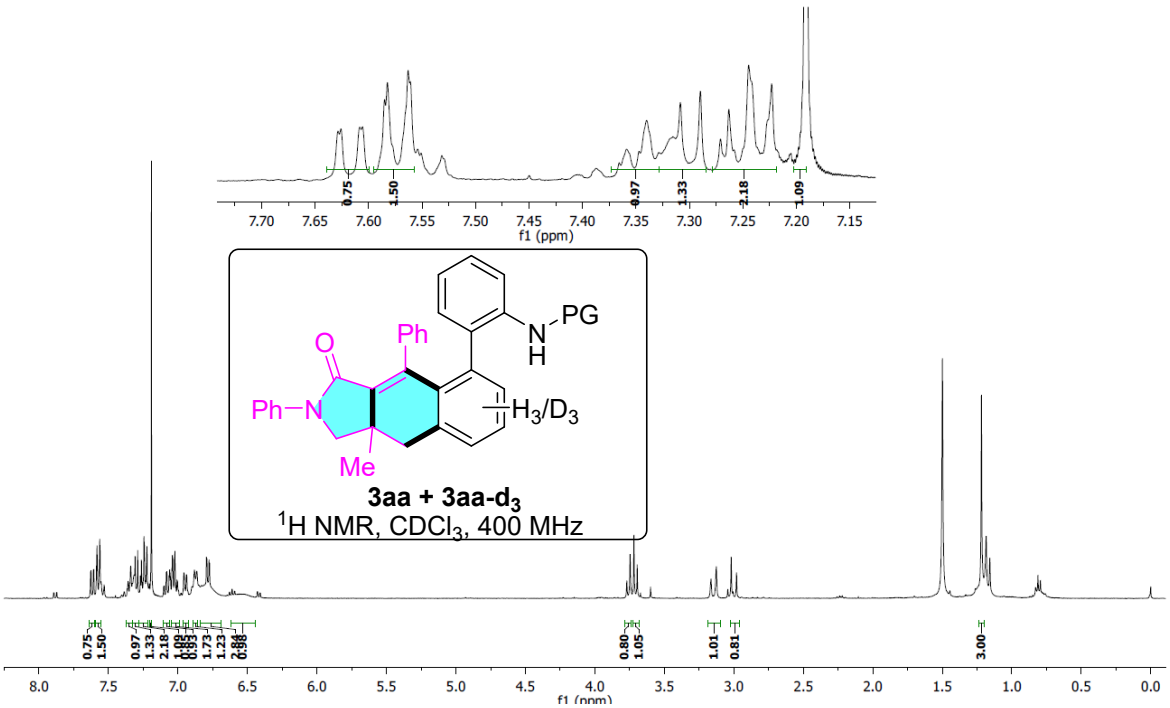
To a mixture of enyne **1a** (137.5 mg, 0.5 mmol) and **2a** (132.5. mg, 0.5 mmol) or **2a-d5** (135 mg, 0.5 mmol) in DMSO, $Pd(OAc)_2$ (11.2 mg, 10 mol%), $Cu(OAc)_2$ (90 mg, 1 equiv) were introduced and the reaction mixture was stirred at 100 °C (oil bath) for 2 hour under air balloon. The reaction mixture was cooled to room temperature before water was added to it. The aqueous layer was extracted with ethyl acetate (3×10 mL), the combined organic extracts were washed with brine, dried over sodium sulfate and concentrated under reduced pressure. The crude residue was purified by column chromatography (Rf = 0.40) (SiO2, EtOAc:Hexane, 10:90) to get **3aa** (123.74 mg, 46%) or **3aa-*d*4** (45.98 mg, 17%) as a white solids. The K_H/K_D value was determined to be 2.7 by the isolated yield.



KIE determined from an intramolecular competition:

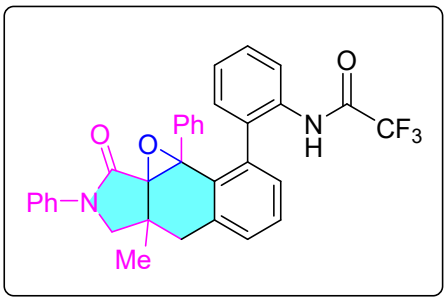


To a mixture of enyne **1a** (137.5 mg, 0.5 mmol) and **2a** (132.5 mg, 0.5 mmol) **2a-d$_4$** (135.0 mg, 0.5 mmol) in DMSO, $Pd(OAc)_2$ (11.2 mg, 10 mol %), $Cu(OAc)_2$ (90 mg, 1 equiv)) were introduced and the reaction mixture was stirred at 100 °C (oil bath) for 2 hour under air balloon. The reaction mixture was cooled to room temperature before water was added to it. The aqueous layer was extracted with ethyl acetate (3×10 mL), the combined organic extracts were washed with brine, dried over sodium sulfate and concentrated under reduced pressure. The crude residue was purified by column chromatography (R_f = 0.50) (SiO_2, EtOAc:Hexane, 10:90) to get **3aa+3aa-*d*$_4$**. The k_H/k_D value of **3aa+3aa-*d*$_4$** was determined to be 3.0 by ^{1}H NMR.



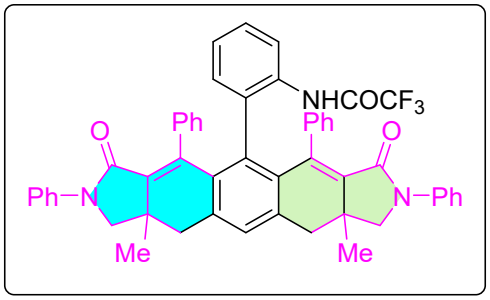
6. General Procedure and Characteristic Data of Synthetic Transformations.

2,2,2-trifluoro-N-(2-(4a-methyl-2-oxo-3,9b-diphenyl-2,3,4,4a,5,9b-hexahydrobenzo[f]oxireno[2,3-d]isoindol-9-yl)phenyl)acetamide (4):



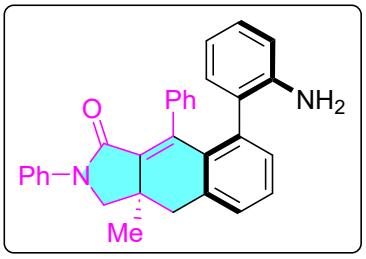
The compound **3aa** (100 mg, 0.18 mmol) and m-chloroperbenzoic acid (96 mg, 0.54 mmol) in DCE (4 ml) were refluxed for 10 h and the solution was cooled and washed with 3 N NaOH. The solvent was dried (Na_2SO_4) and evaporated and the residue was purified by column chromatography (R_f = 0.45, SiO_2, EtOAc:hexane, 12:88) to get **4** as a white solid (64%, 65.23 mg), mp = 225-228 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.63 (d, *J* = 8.1 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.32 (dt, *J* = 17.1, 7.6 Hz, 3H), 7.26 – 7.21 (m, 3H), 7.16 (t, *J* = 7.8 Hz, 1H), 7.06 (t, *J* = 7.3 Hz, 1H), 7.01 (t, *J* = 5.8 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 2H), 6.78 (dd, *J* = 16.6, 10.0 Hz, 2H), 6.65 (t, *J* = 7.0 Hz, 2H), 3.90 (d, *J* = 9.9 Hz, 1H), 3.84 (s, 1H), 3.42 (d, *J* = 14.6 Hz, 1H), 2.80 (d, *J* = 14.6 Hz, 2H), 1.06 (s, 3H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 165.3, 155.7, 155.4, 139.6, 138.8, 137.7, 136.3, 135.4, 134.8, 134.4, 133.3, 132.1, 130.6, 130.1, 129.9, 128.9, 128.7, 128.1, 127.3, 127.0, 126.9, 126.8, 126.4, 125.6, 125.3, 124.3, 121.2, 119.9, 71.7, 64.4, 58.7, 43.1, 33.5, 20.9. **HRMS (ESI)** calcd for $C_{33}H_{26}F_3N_2O_3$ $[M+H]^+$ 555.1896 found 555.1833.

2,2,2-trifluoro-N-(2-(2,4,6,8-tetraargio-9a,12a-dimethyl-3,7-dioxo-1,2,3,7,8,9,9a,10,12,12a-decahydrobenzo[1,2-f:4,5-f']diisoindol-5-yl)phenyl)acetamide (5):



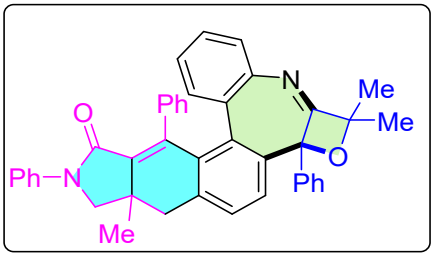
The title compound was prepared from **1a** (51 mg, 0.18 mmol) and **3aa** (100 mg, 0.18 mmol) according to General procedure **A**. Purification using column chromatography (R_f = 0.30, SiO_2, EtOAc:hexane, 15:85) gave pure product as an off-white solid (64.0 mg, 52% yield), mp 325-328 °C. **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.53 (d, *J* = 8.0 Hz, 4H), 7.48 (s, 1H), 7.28 – 7.24 (m, 4H), 7.10 – 7.03 (m, 4H), 6.94 (t, *J* = 7.4 Hz, 2H), 6.86 (dd, *J* = 14.1, 6.9 Hz, 3H), 6.73 (s, 1H), 6.68 (d, *J* = 7.5 Hz, 1H), 6.64 (t, *J* = 7.2 Hz, 2H), 6.57 (d, *J* = 7.4 Hz, 2H), 6.29 (d, *J* = 7.8 Hz, 2H), 3.77 (d, *J* = 9.2 Hz, 2H), 3.67 (d, *J* = 9.1 Hz, 2H), 3.25 (d, *J* = 14.3 Hz, 2H), 3.02 (d, *J* = 14.3 Hz, 2H), 1.12 (s, 6H). **^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)** δ 164.3, 151.8, 151.4, 142.4, 139.5, 139.2, 137.8, 136.9, 136.7, 135.5, 134.5, 133.6, 131.5, 130.5, 130.3, 130.2, 129.1, 128.6, 128.5, 127.8, 126.6, 126.5, 126.2, 125.6, 125.1, 124.6, 119.8, 118.3. 113.8, 58.9, 43.7, 34.6, 21.6. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -74.3. **HRMS (ESI)** calcd for $C_{52}H_{41}F_3N_2O_2$ $[M+H]^+$ 812.3100, found 812.3115.

8-(2-aminophenyl)-2,9-diargio-3a-methyl-2,3,3a,4-tetrahydro-1H-benzo[f]isoindol-1-one (6):



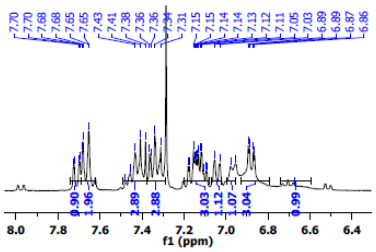
To a 50 mL RB flask, **3aa** (1 mmol), K_2CO_3 (4.0 equiv), methanol (20 mL) and H_2O (20 mL) were added. The reaction mixture was heated at 70 °C under N_2 atmosphere for 12 hours. After cooling to room temperature, the reaction was diluted with dichloromethane (30 mL), washed by H_2O (30 mL), brine (30 mL), and dried over anhydrous Na_2SO_4. After concentration, The residue was purified by flash column chromatography with ethyl acetate and petroleum ether (4 : 1) as eluent to afford the desired product **6** (88% yield), 4:1 dr, mp 220-225 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.49 (s, 1H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.34 – 7.29 (m, 1H), 7.24 (t, *J* = 7.7 Hz, 5H), 7.11 – 7.00 (m, 3H), 6.96 – 6.85 (m, 4H), 6.82 (d, *J* = 7.2 Hz, 2H), 6.56 (dd, 1H), 3.73 (d, *J* = 9.5 Hz, 1H), 3.69 (t, *J* = 7.0 Hz, 1H), 3.68 (d, *J* = 9.4 Hz, 1H), 3.15 (d, *J* = 15.0 Hz, 1H), 2.92 (d, *J* = 14.9 Hz, 1H), 1.19 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 165.1, 143.4, 143.0, 137.4, 137.1, 136.7, 136.2, 135.9, 135.6, 135.3, 132.1, 131.5, 130.8, 129.9, 128.7, 128.2, 127.6, 127.6, 127.5, 127.0, 126.3, 126.2, 126.0, 124.4, 119.8, 118.4, 114.4, 59.6, 42.9, 34.7, 22.5. **HRMS (ESI)** calcd for $C_{31}H_{27}N_2O_2$ $[M+H]^+$ 443.2123, found 443.2103.

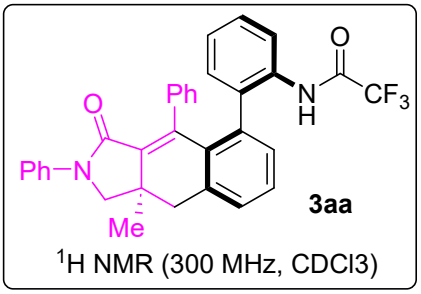
2,4-diargio-10,10,14a-trimethyl-11a-phenyl-1,2,10,11a,14,14a-hexahydro-3H-benzo[2',3']oxeto[2'',3'':6',7']azepino[4',5':3,4]benzo[1,2-f]isoindol-3-one (8):



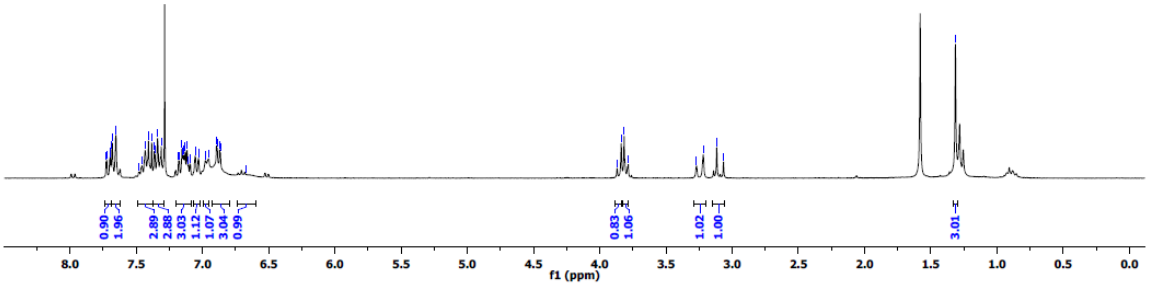
To an oven-dried 25 mL round bottom flask, a mixture of compound **6 (**100 mg, 0.22 mmol) and propargyl alcohol **7** (75 mg, 0.44 mmol) in anhydrous DMF (4 mL), $Pd(OAc)_2$ (5.1 mg, 10 mol%) was added $Cu(OAc)_2$ (162 mg, 4 eq) and 4 Å molecular sieves, and the reaction mixture was stirred at 100 °C (oil bath) for 12 hours under nitrogen atmosphere. After completion of the reaction, reaction mixture was cooled to rt before ice water was added to it. The aqueous layer was extracted with ethyl acetate (2×10 mL). The organic layer was evaporated and the residue was purified by column chromatography (R_f = 0.30) (SiO_2, EtOAc:Hexane, 25:75) to get **8** (89.3 mg, 66% yield). mp = 281-285 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.62 (dd, *J* = 7.7, 5.3 Hz, 3H), 7.40 (d, *J* = 7.7 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 2H), 7.04 (t, *J* = 7.4 Hz, 2H), 6.95 (q, *J* = 5.5 Hz, 1H), 6.89 (dd, *J* = 7.6, 1.3 Hz, 3H), 6.85 (d, *J* = 7.2 Hz, 1H), 6.82 (d, *J* = 7.8 Hz, 1H), 6.78 (dd, *J* = 7.9, 1.7 Hz, 2H), 6.74 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.71 (d, *J* = 7.8 Hz, 1H), 6.65 (s, 1H), 3.75 (s, 2H), 3.10 (d, *J* = 15.8 Hz, 1H), 3.02 (d, *J* = 15.8 Hz, 1H), 1.63 (s, 3H), 1.48 (s, 3H), 1.35 (s, 3H). **^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)** δ 179.4, 165.0, 147.6, 143.9, 143.5, 139.8, 138.5, 137.3, 137.1, 136.7, 135.4, 134.7, 133.3, 132.0, 128.7, 128.1, 127.9, 127.3, 126.7, 126.4, 125.9, 124.9, 124.5, 124.2, 123.8, 119.8, 94.3, 93.8, 59.8, 42.9, 35.2, 27.8, 24.6, 22.6. **HRMS (ESI)** calcd for $C_{42}H_{35}N_2O_2$ $[M+H]^+$ 599.2699, found 599.2719.

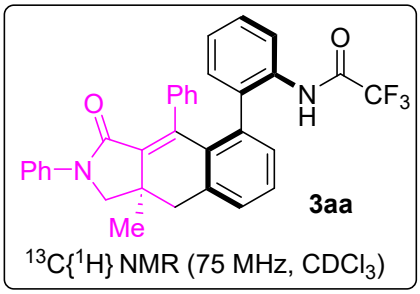
7. Copies of ^{1}H, ^{13}C and ^{19}F spectra for all final compounds



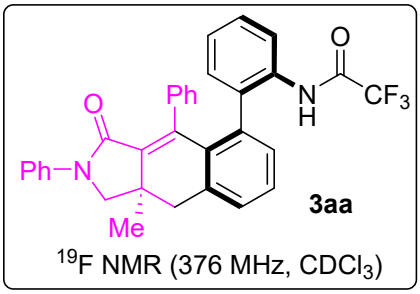


^{1}H NMR (300 MHz, CDCl3)

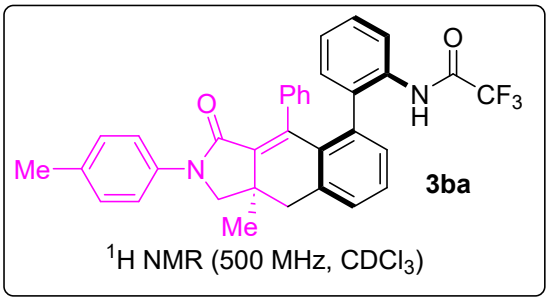




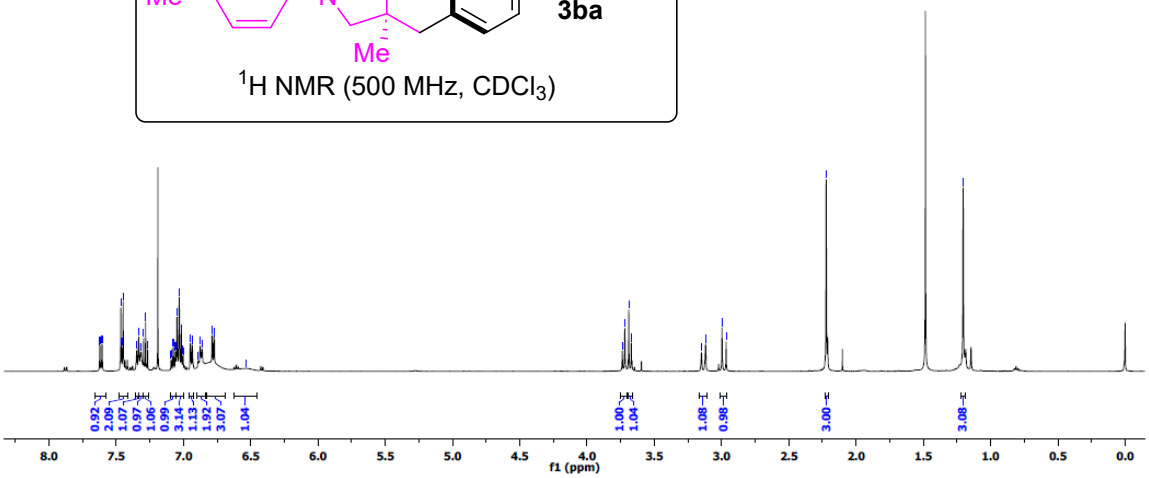
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)

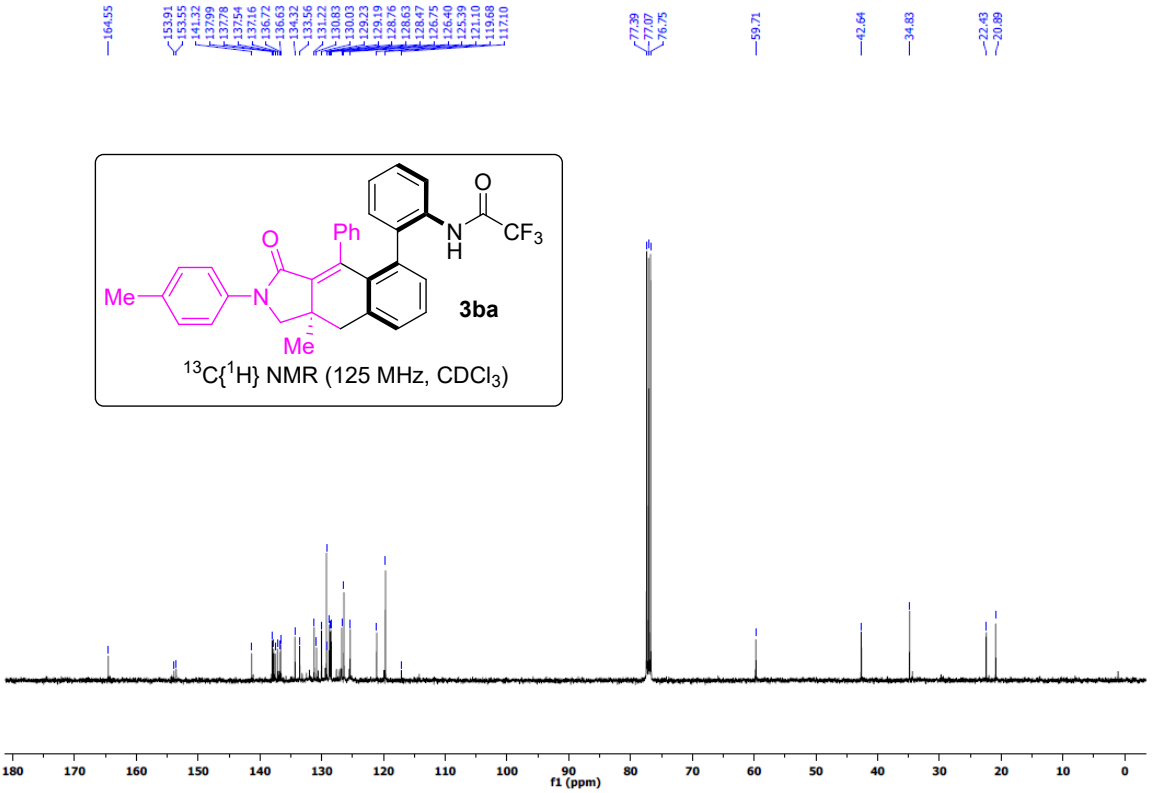


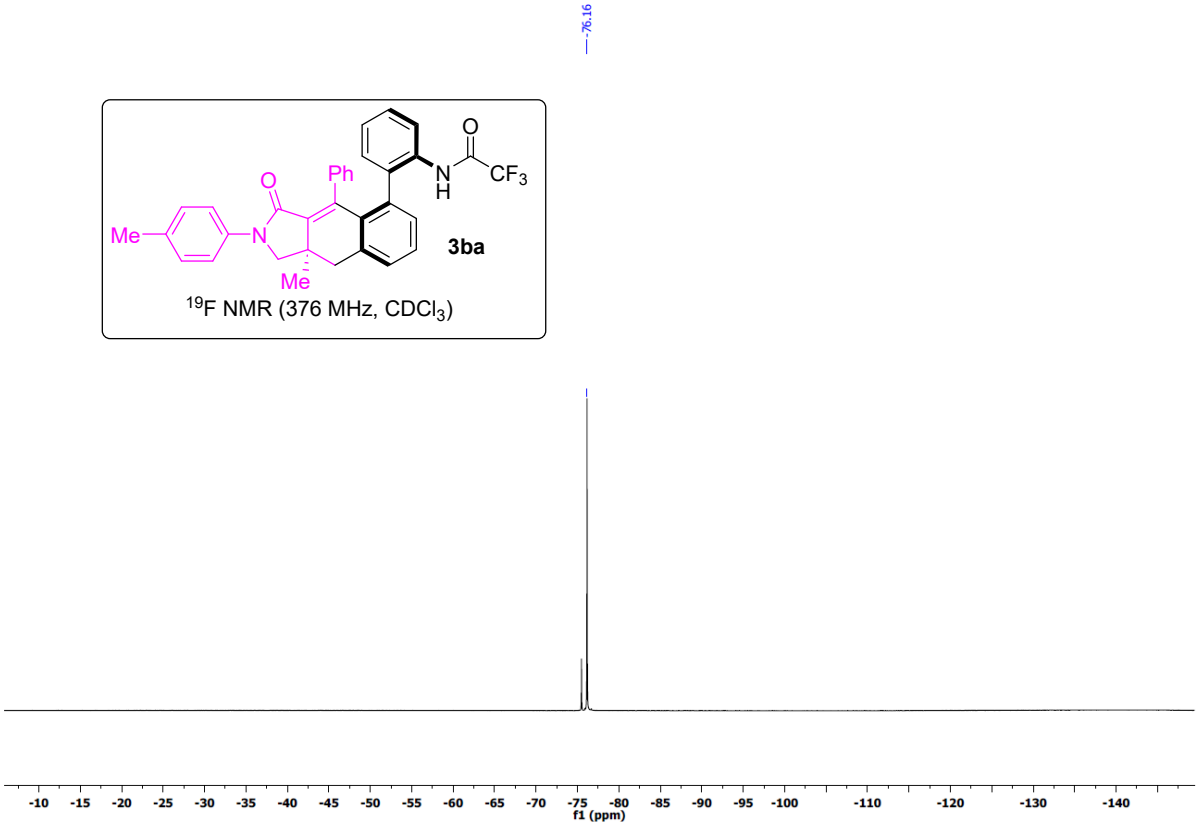
^{19}F NMR (376 MHz, $CDCl_3$)

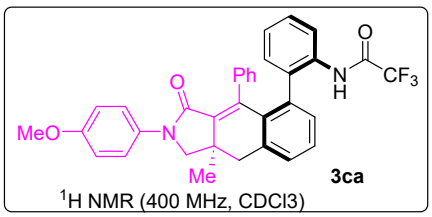


^{1}H NMR (500 MHz, $CDCl_3$)

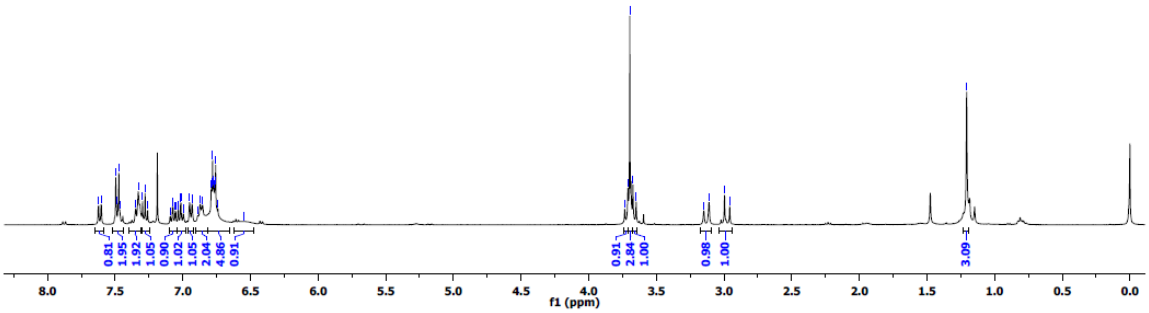


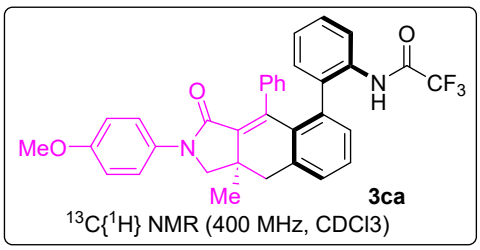

164.55
153.91
153.55
141.32
137.99
137.78
137.54
137.16
136.72
136.63
134.32
133.56
131.22
130.83
130.03
129.23
129.19
128.76
128.63
128.47
126.75
126.40
125.39
121.10
119.68
117.10
77.39
77.07
76.75
59.71
42.64
34.83
22.43
20.89
O
N
H
CF3
Ph
O
Me
N
Me
3ba
13C{1H} NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


76.16
O
N
H
CF3
Ph
O
Me
N
Me
3ba
19F NMR (376 MHz, CDCl3)
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-110
-120
-130
-140
f1 (ppm)

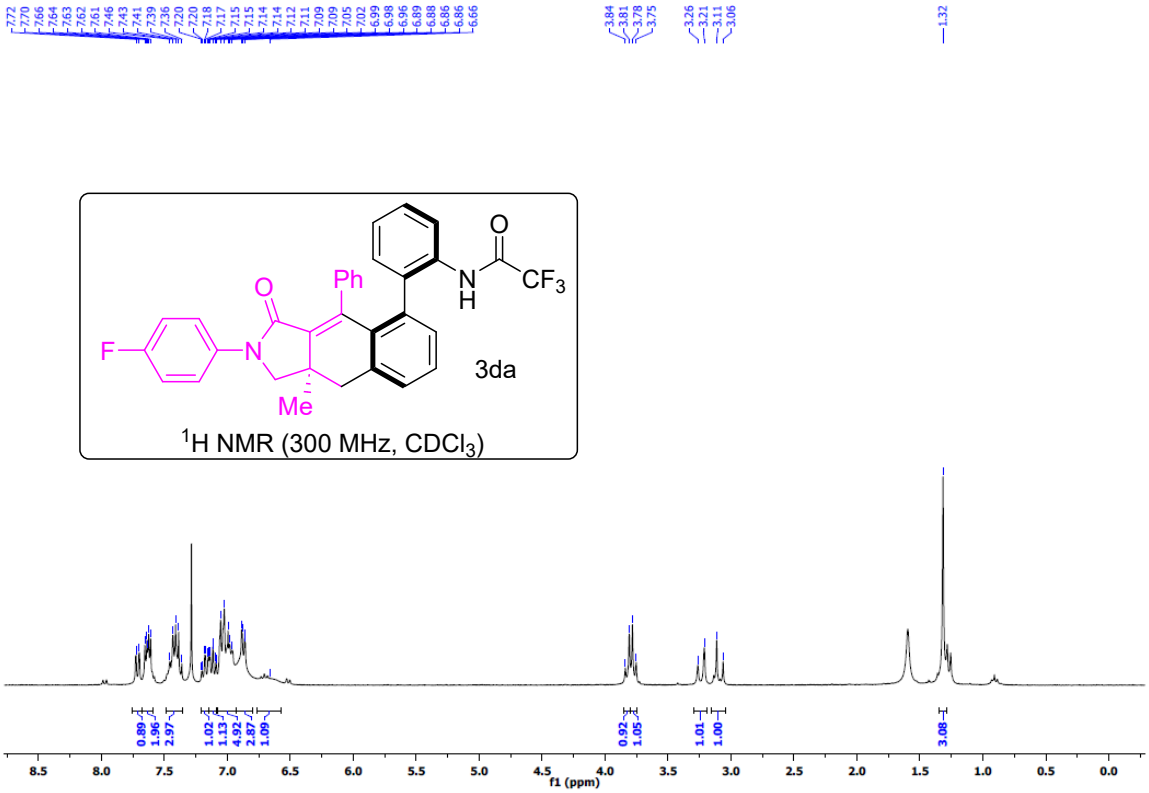


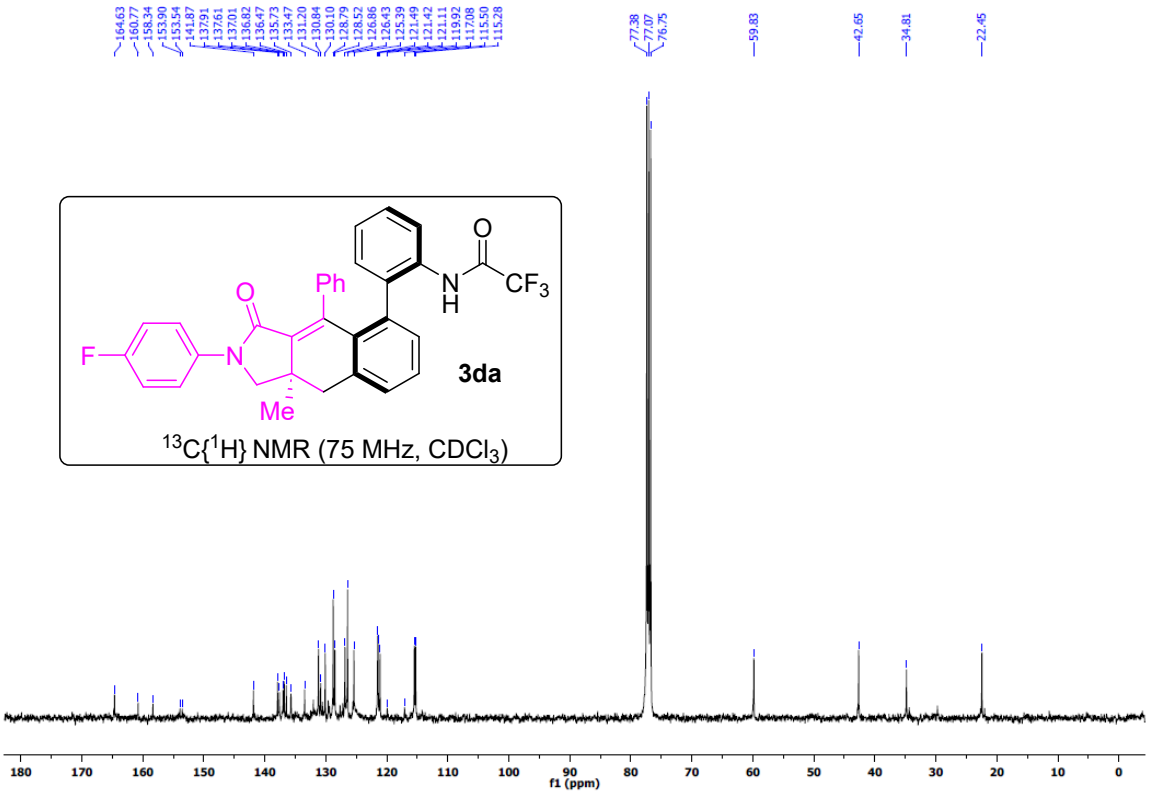
^{1}H NMR (400 MHz, CDCl3)



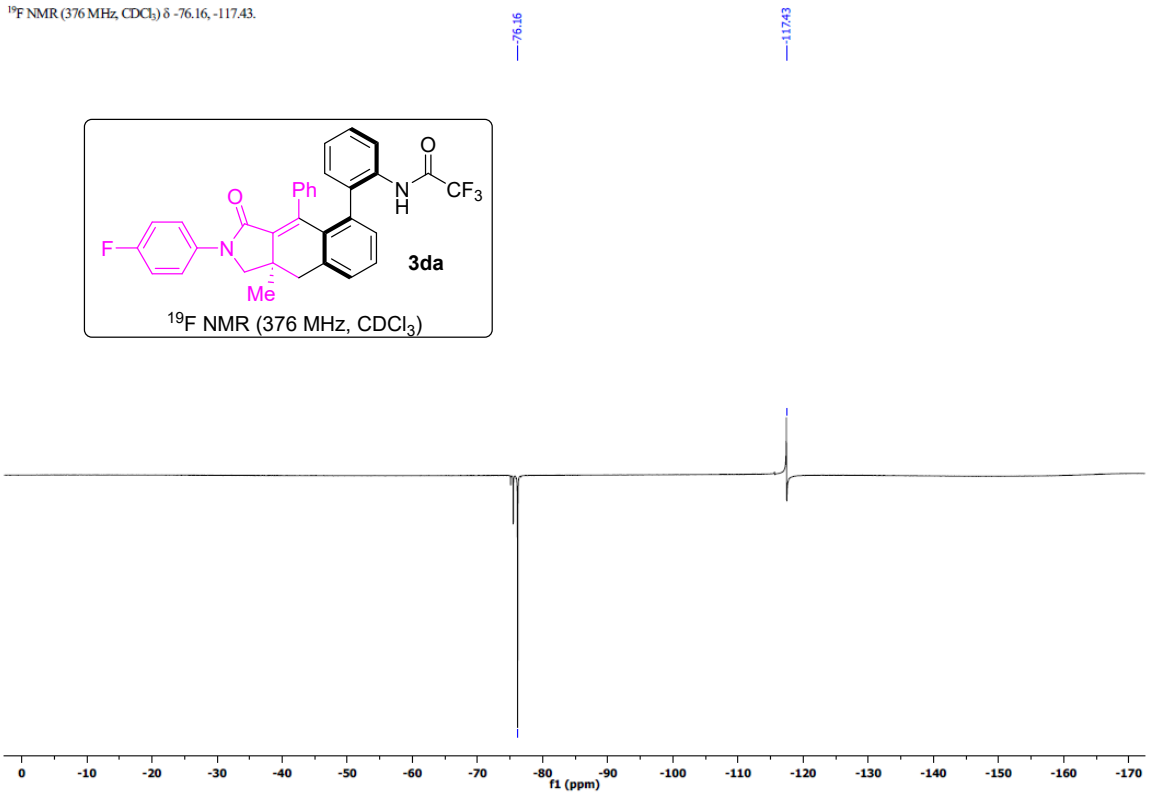


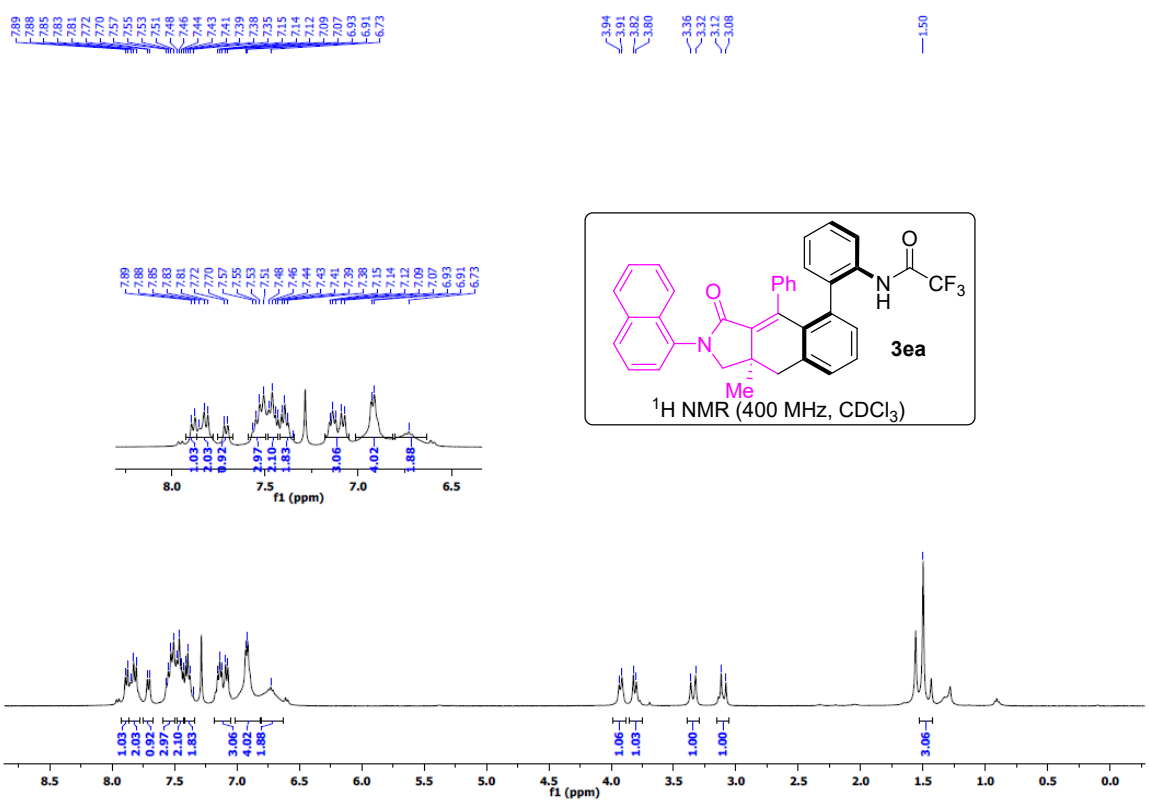
^{13}C{^{1}H} NMR (400 MHz, CDCl3)

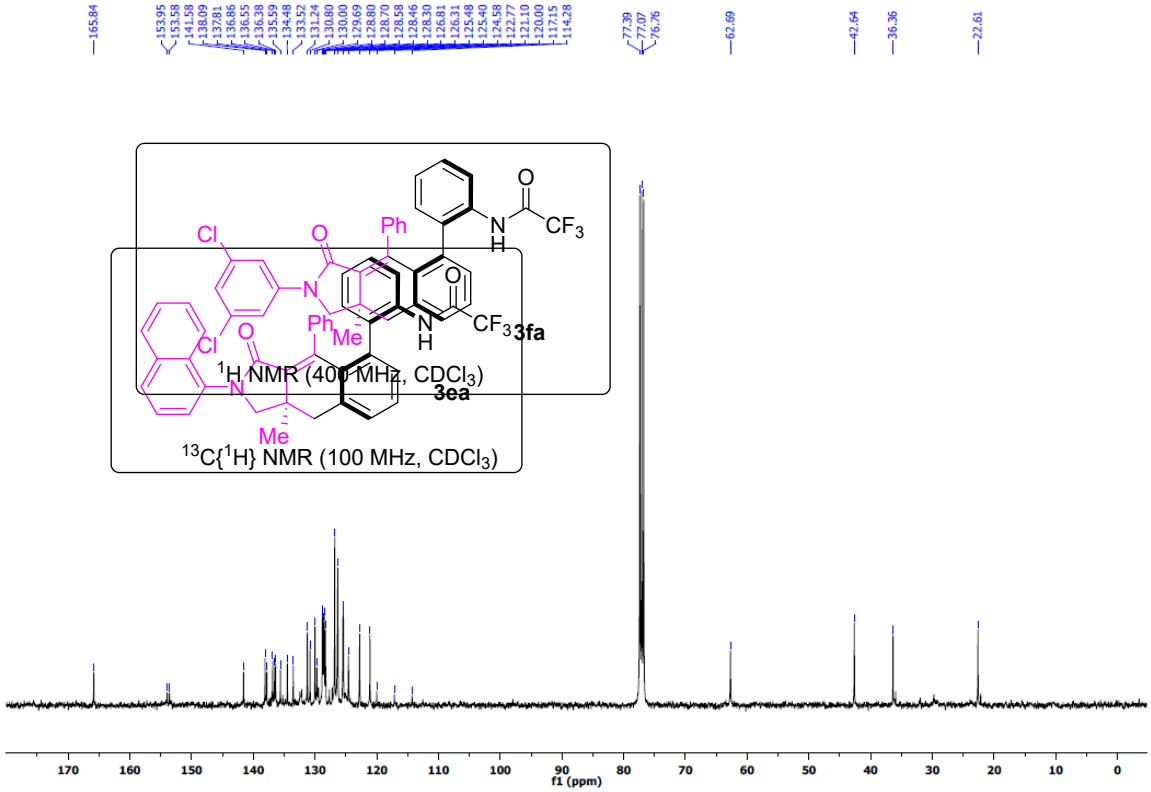

7.72
7.70
7.66
7.64
7.63
7.62
7.61
7.46
7.43
7.41
7.39
7.36
7.20
7.20
7.18
7.17
7.15
7.15
7.14
7.14
7.12
7.11
7.09
7.09
7.05
7.02
6.99
6.98
6.96
6.89
6.88
6.86
6.86
6.66
3.84
3.81
3.78
3.75
3.26
3.21
3.11
3.06
1.32
Ph
O
N
H
O
CF3
F
N
Me
3da
1H NMR (300 MHz, CDCl3)
0.89
1.96
2.97
1.02
1.13
4.92
2.87
1.09
0.92
1.05
1.01
1.00
3.08
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
f1 (ppm)

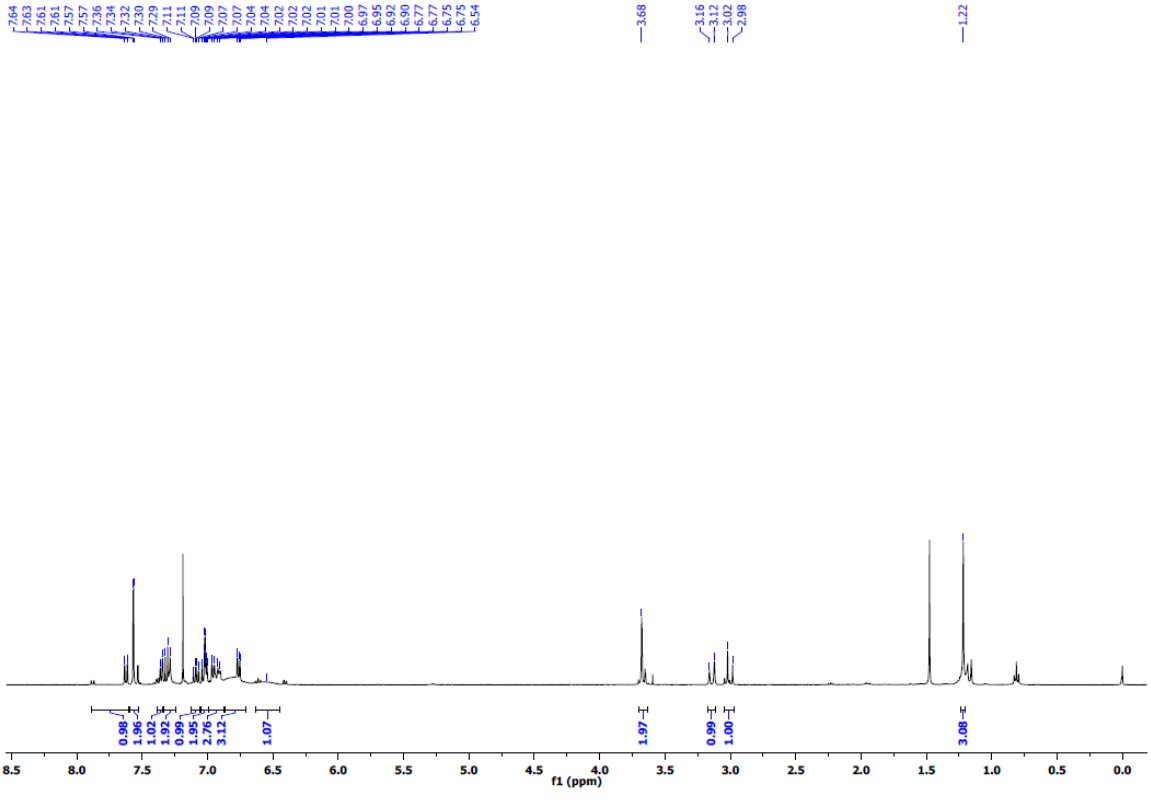

164.63
160.77
158.34
153.90
153.54
141.87
137.91
137.61
137.01
136.82
136.47
135.73
133.47
131.20
130.84
130.10
128.79
128.52
126.86
126.43
125.39
121.49
121.42
121.11
119.92
117.08
115.50
115.28
77.38
77.07
76.75
59.83
42.65
34.81
22.45
Ph
O
N
H
O
CF3
F
N
Me
3da
13C{1H} NMR (75 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

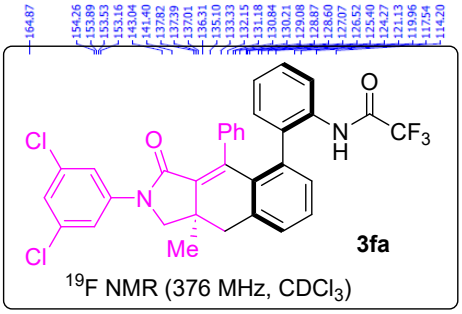
^{19}F NMR (376 MHz, $CDCl_3$) δ -76.16, -117.43.



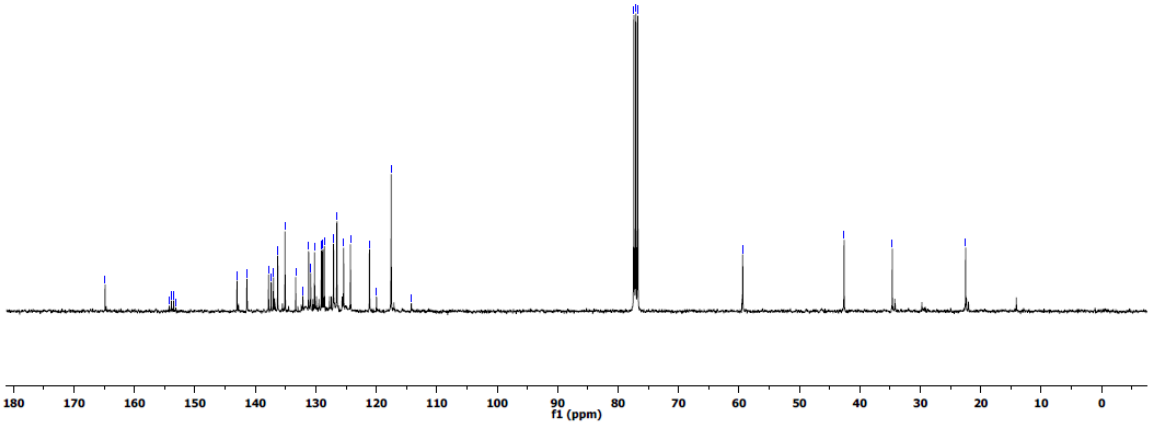


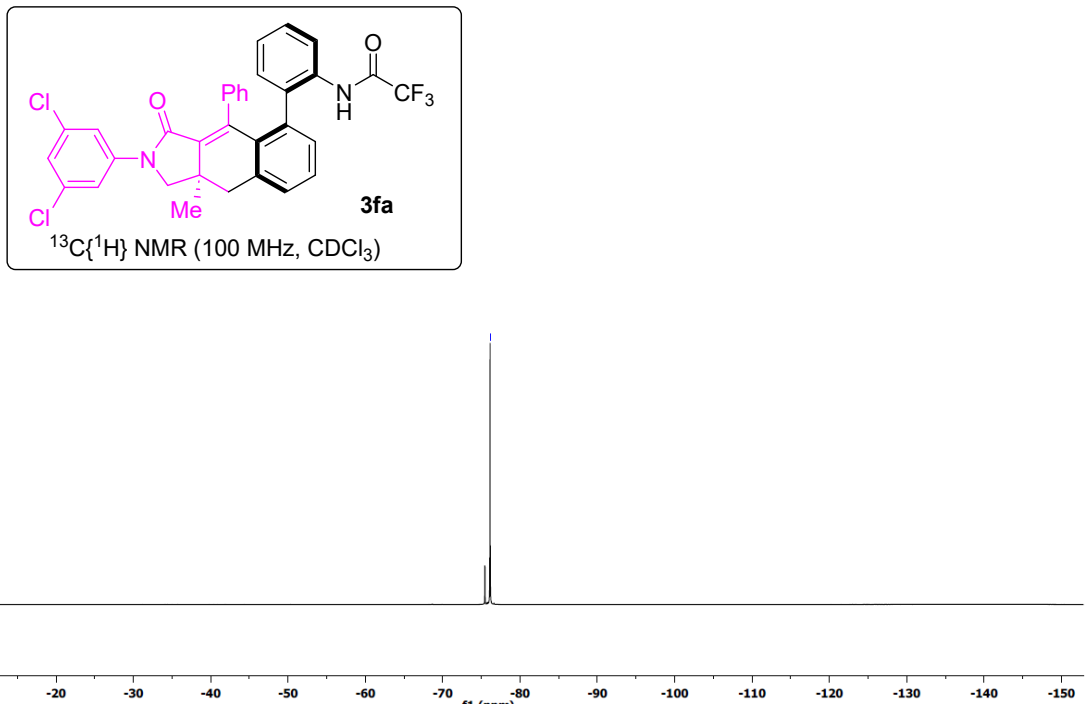

3fa
3ea
^{1}H NMR (400 MHz, CDCl$_3$)
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$)
f1 (ppm)


f1 (ppm)

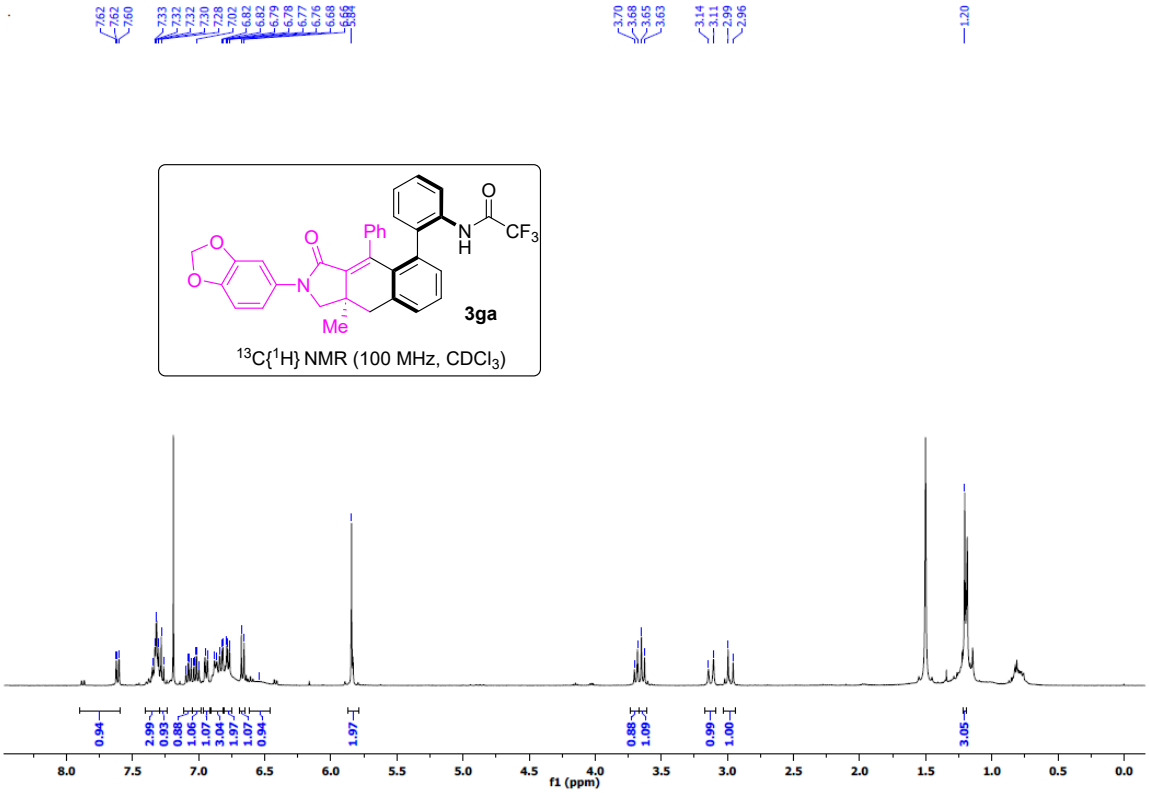


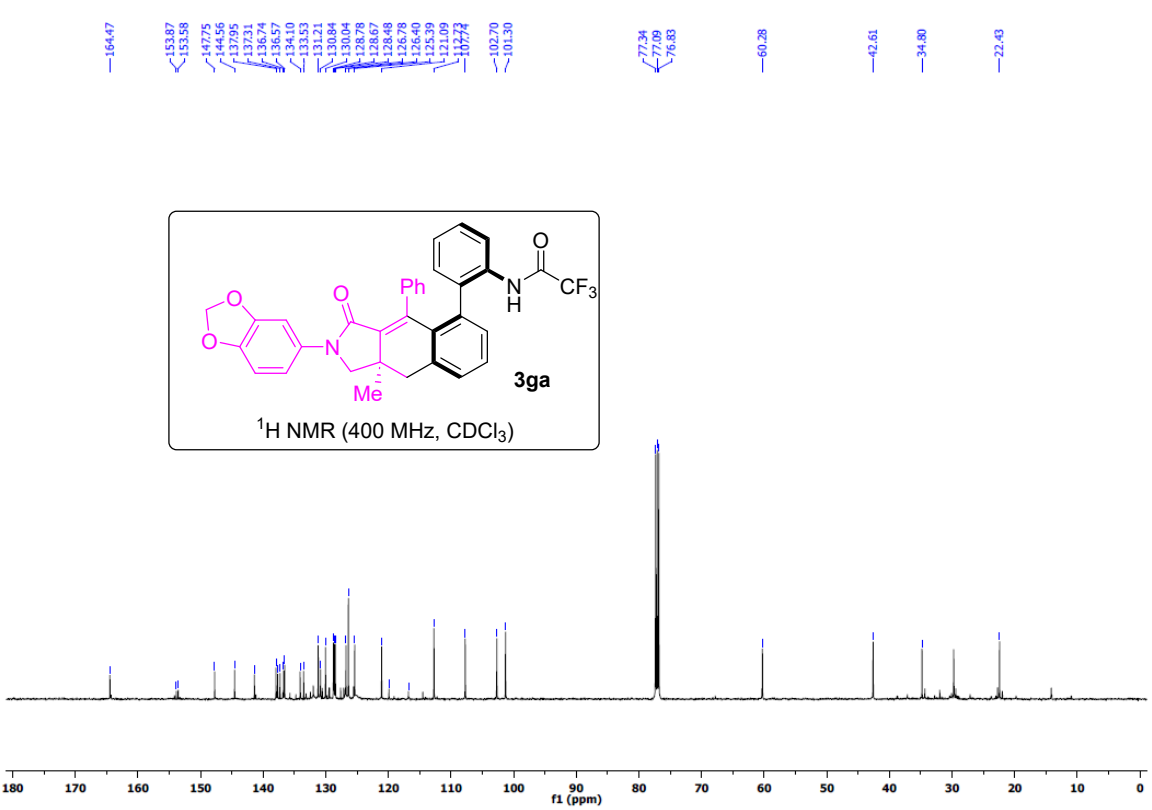
^{19}F NMR (376 MHz, $CDCl_3$)

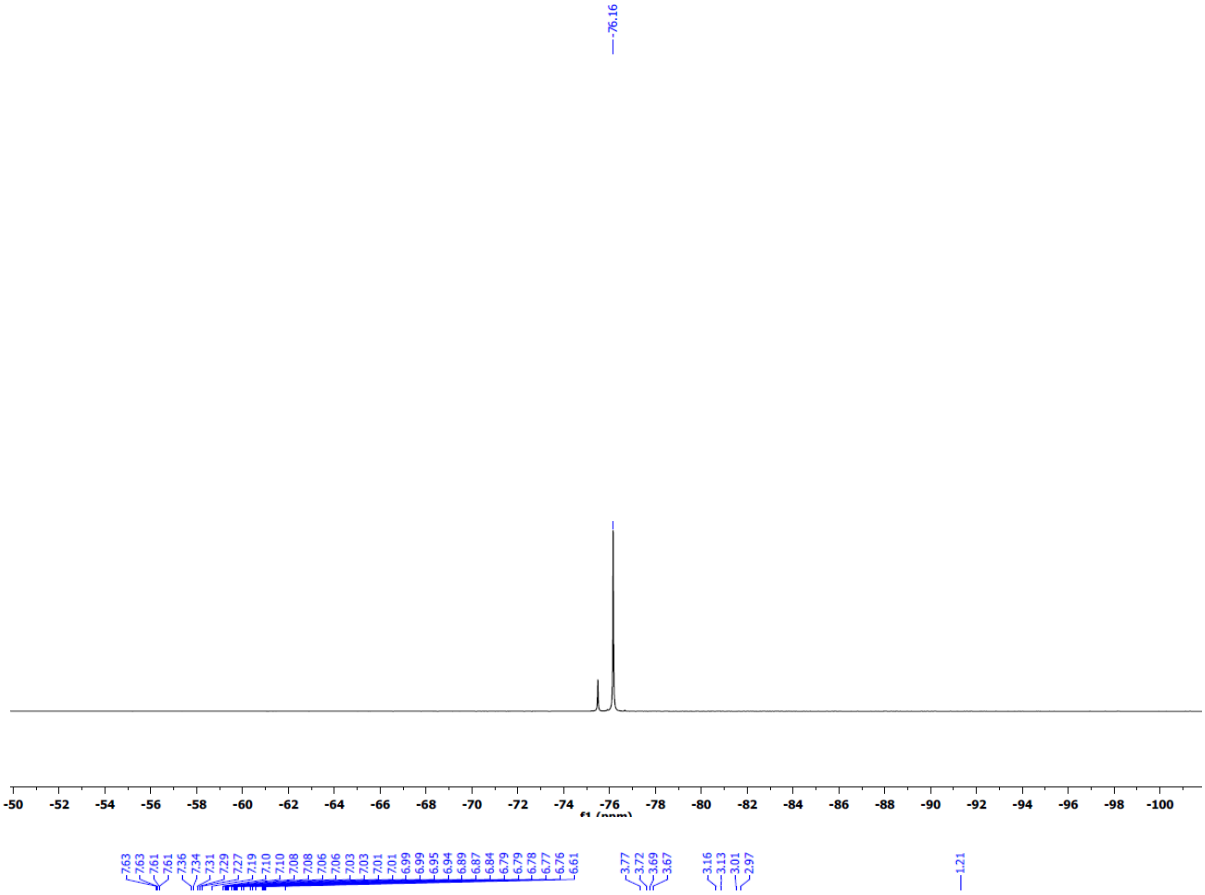


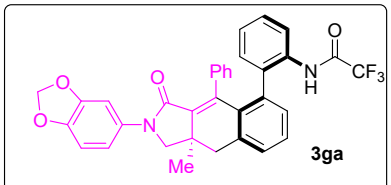


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)

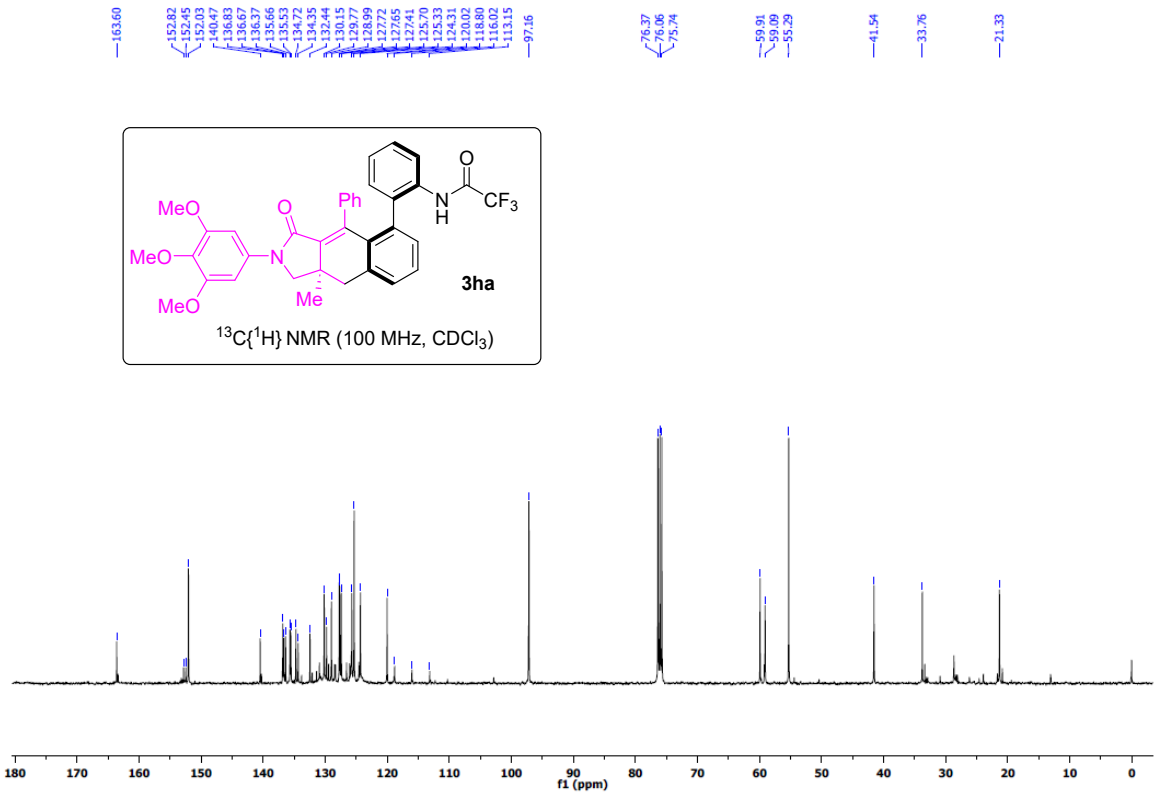

3ga
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)
f1 (ppm)

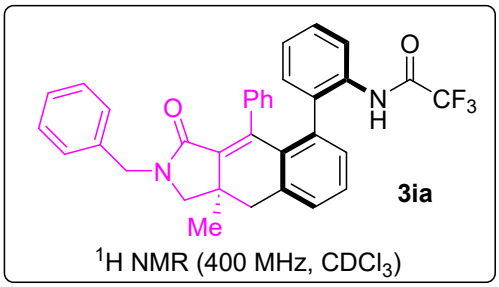

3ga
^{1}H NMR (400 MHz, $CDCl_3$)
f1 (ppm)



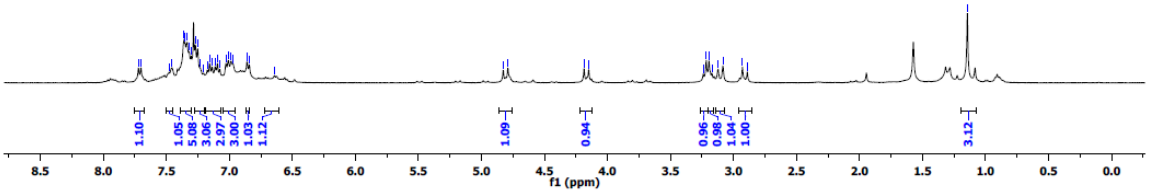


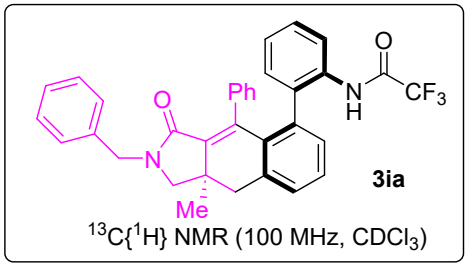
^{19}F NMR (376 MHz, $CDCl_3$)


163.60
152.82
152.45
152.03
140.47
136.83
136.67
136.37
135.66
135.53
134.72
134.35
132.44
130.15
129.77
128.99
127.72
127.65
127.41
125.70
125.33
124.31
120.02
118.80
116.02
113.15
97.16
76.37
76.06
75.74
59.91
59.09
55.29
41.54
33.76
21.33
MeO
MeO
MeO
O
N
Ph
Me
O
N
H
CF3
3ha
13C{1H} NMR (100 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

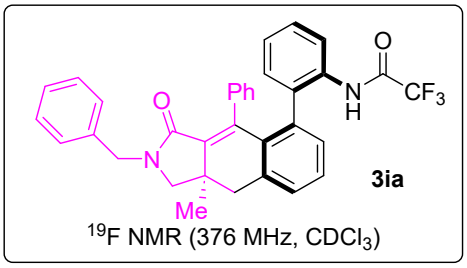


^{1}H NMR (400 MHz, $CDCl_3$)

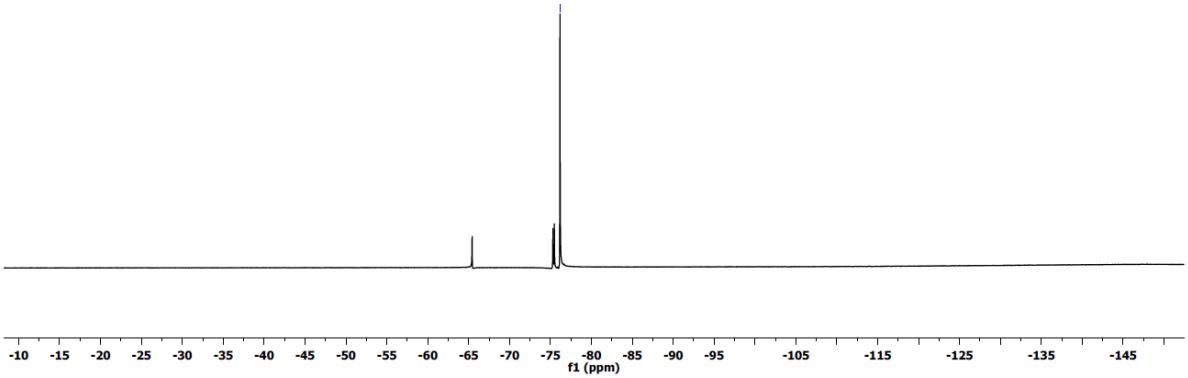


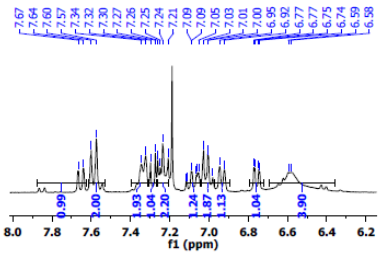


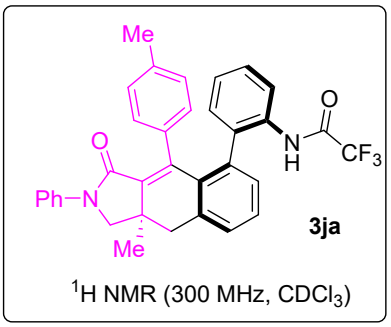
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$)



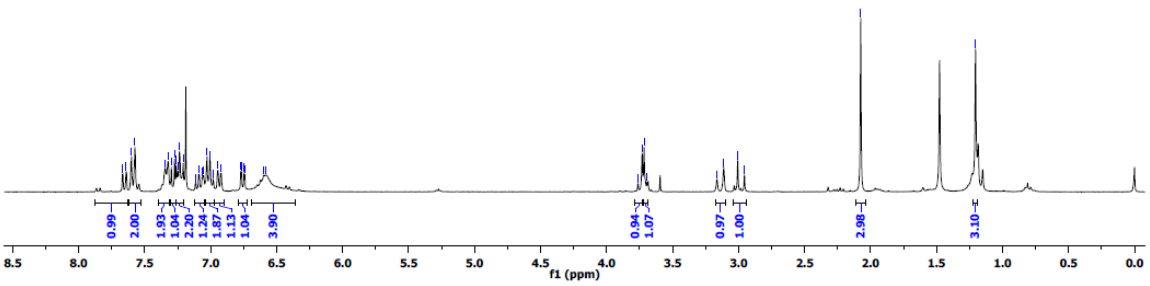
^{19}F NMR (376 MHz, $CDCl_3$)

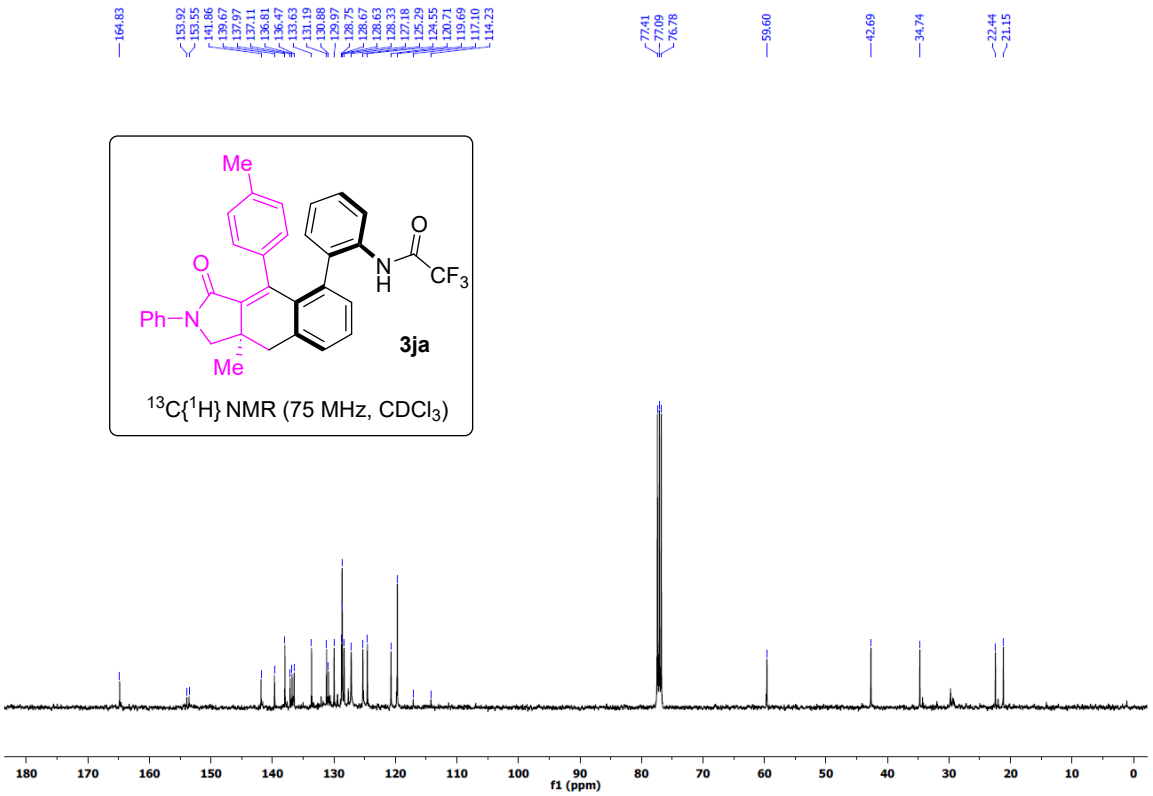


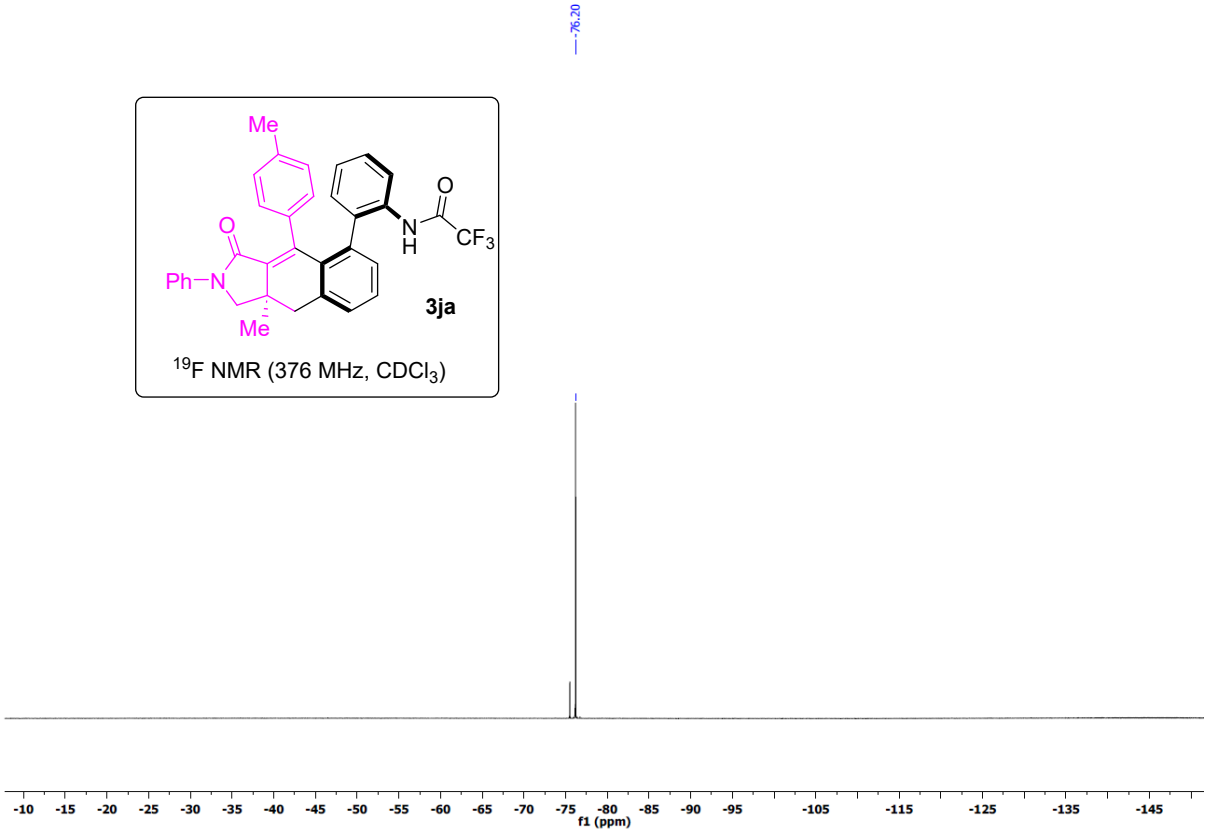


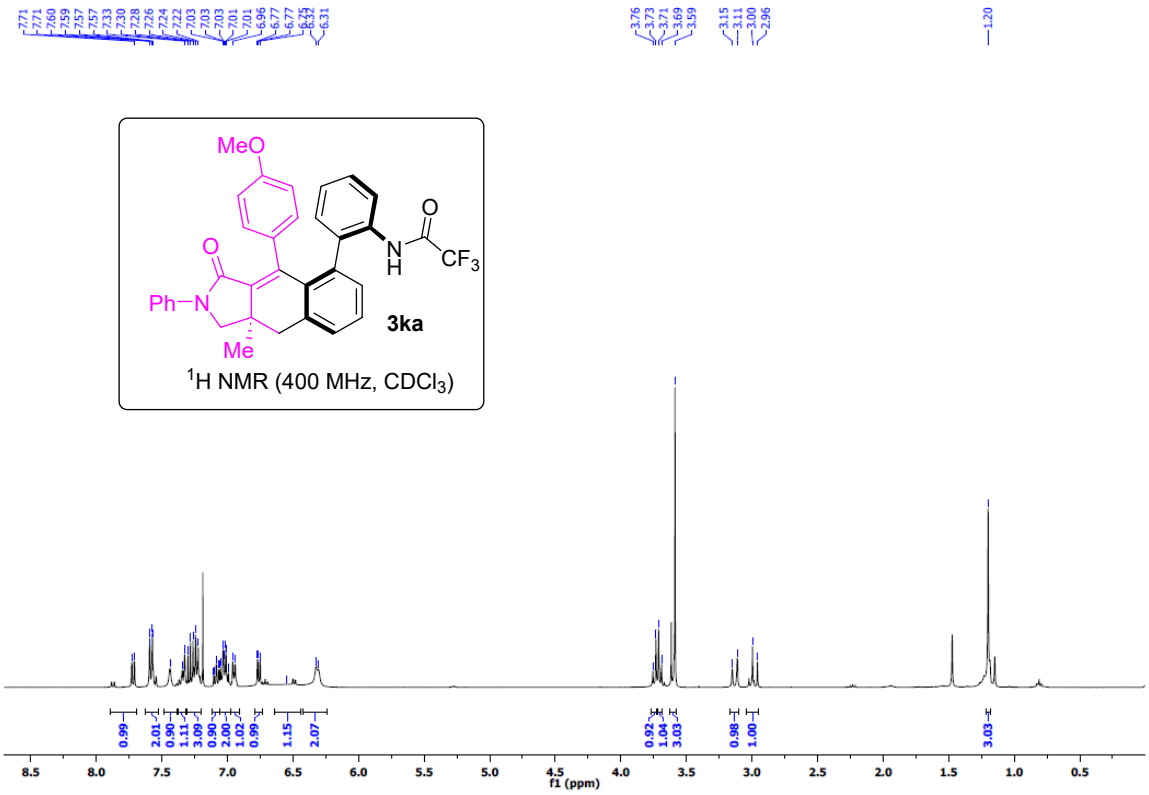


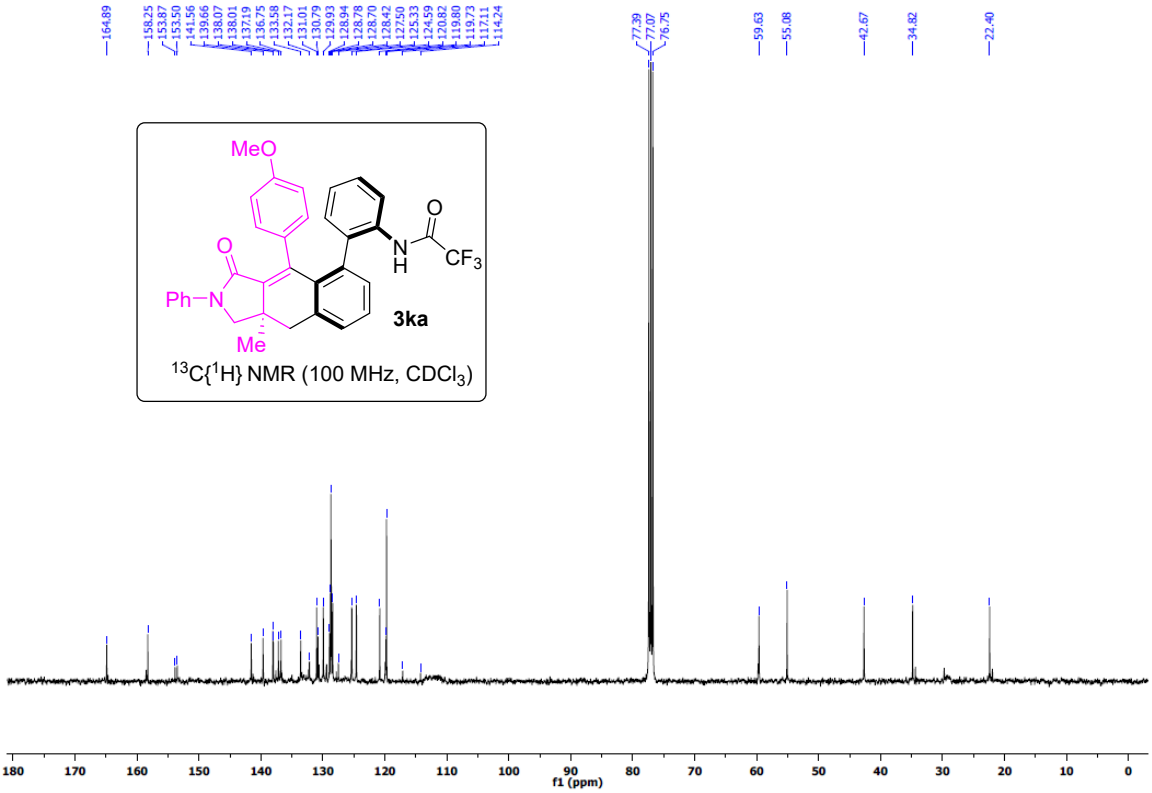
^{1}H NMR (300 MHz, $CDCl_3$)

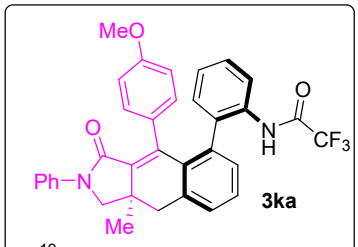



164.83
153.92
153.55
141.86
139.67
137.97
137.11
136.81
136.47
133.63
131.19
130.88
129.97
128.75
128.67
128.63
128.33
127.18
125.29
124.55
120.71
119.69
117.10
114.23
77.41
77.09
76.78
59.60
42.69
34.74
22.44
21.15
Me
O
N
H
CF3
Ph–N
O
Me
3ja
13C{1H} NMR (75 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-76.20
Me
O
N
H
CF3
Ph–N
O
Me
3ja
19F NMR (376 MHz, CDCl3)
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
-145
f1 (ppm)

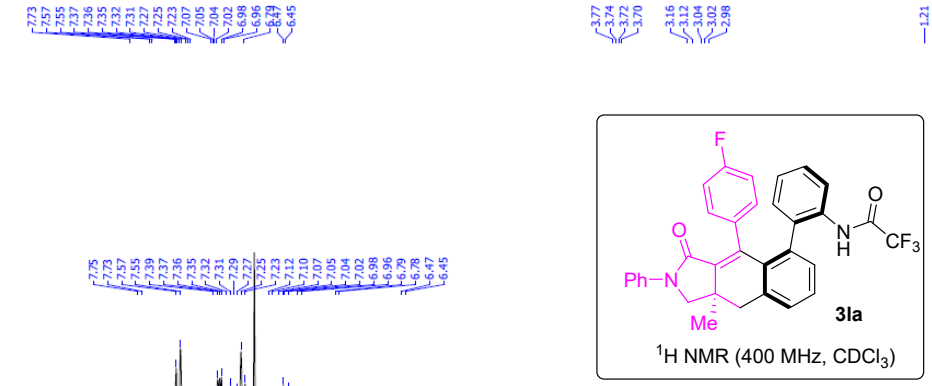

MeO
O
N
H
CF3
Ph–N
O
Me
3ka
1H NMR (400 MHz, CDCl3)
f1 (ppm)


MeO
O
N
H
CF3
Ph–N
O
Me
3ka
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)

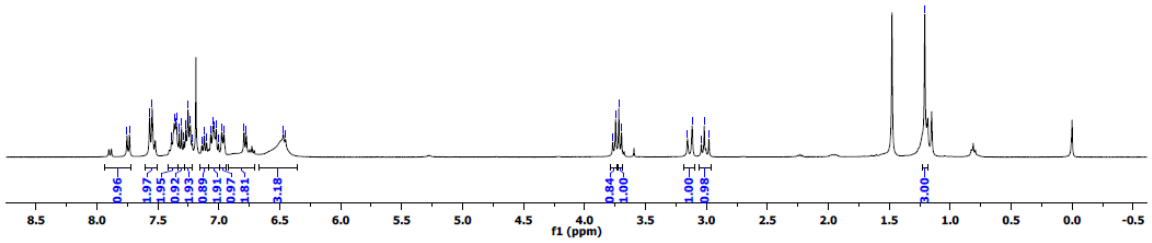


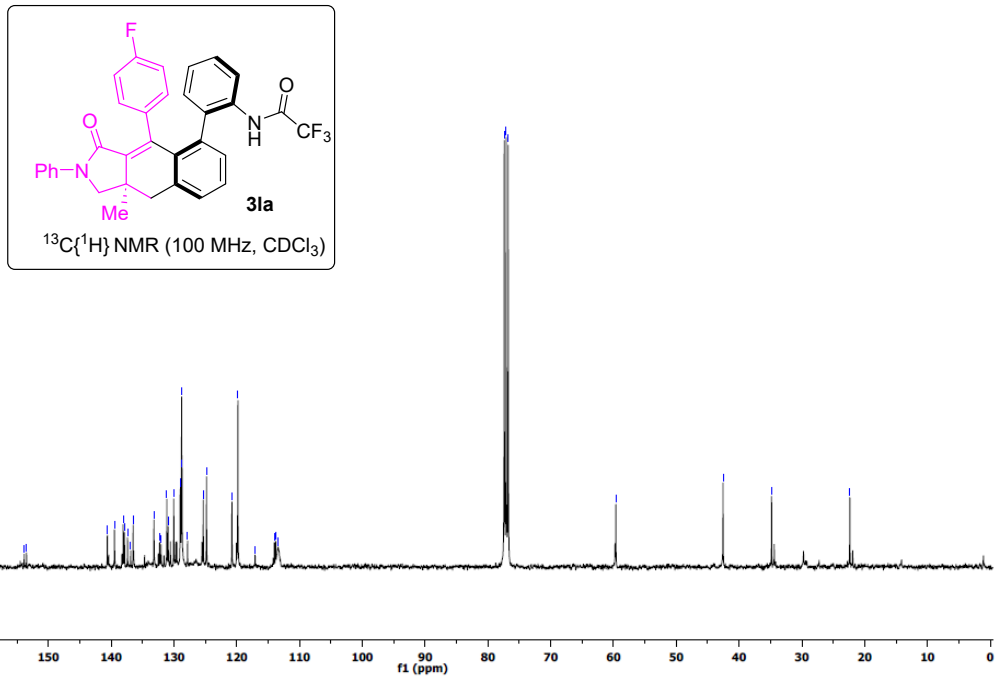
3ka

^{19}F NMR (376 MHz, $CDCl_3$)

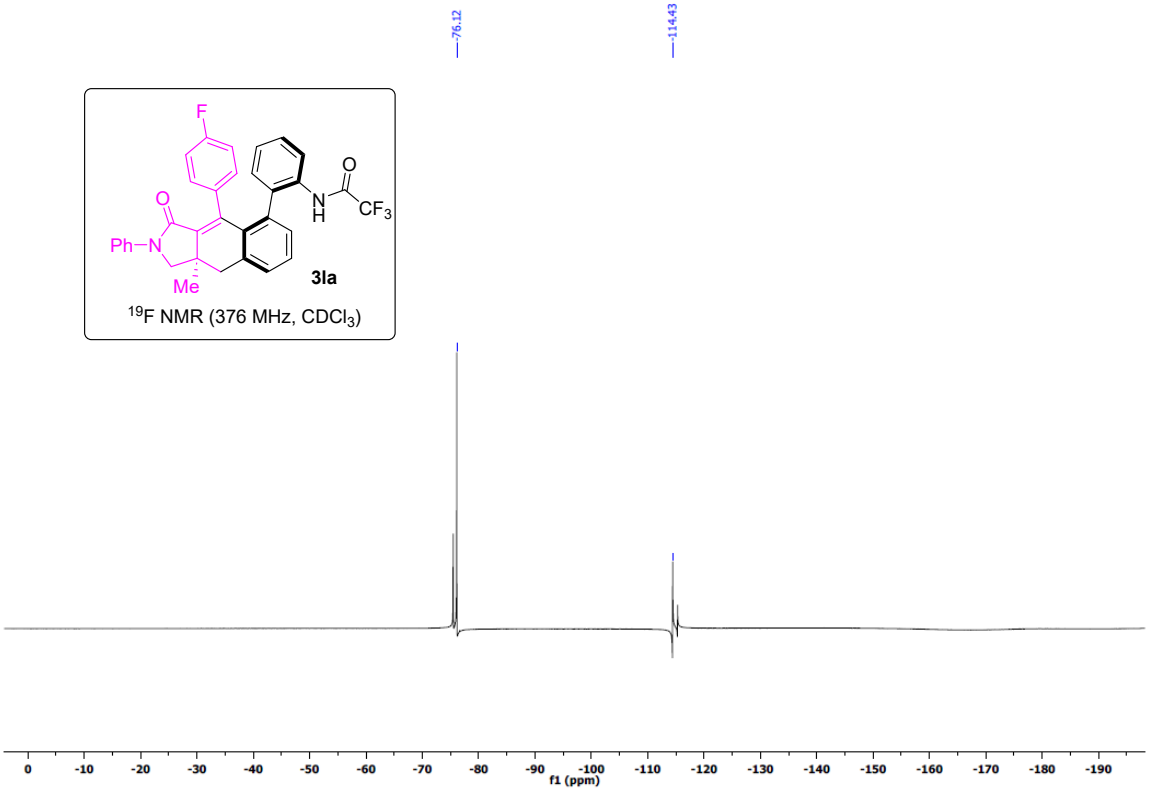


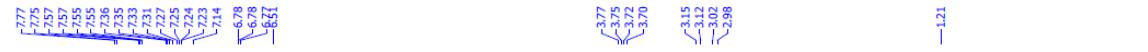
^{1}H NMR (400 MHz, $CDCl_3$)

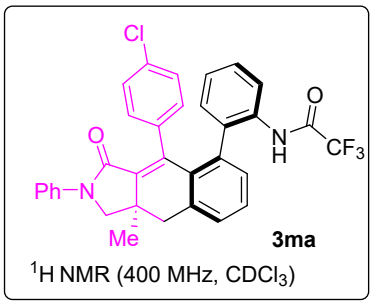


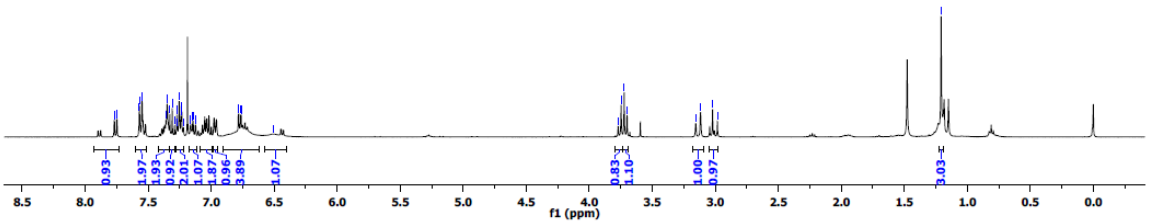


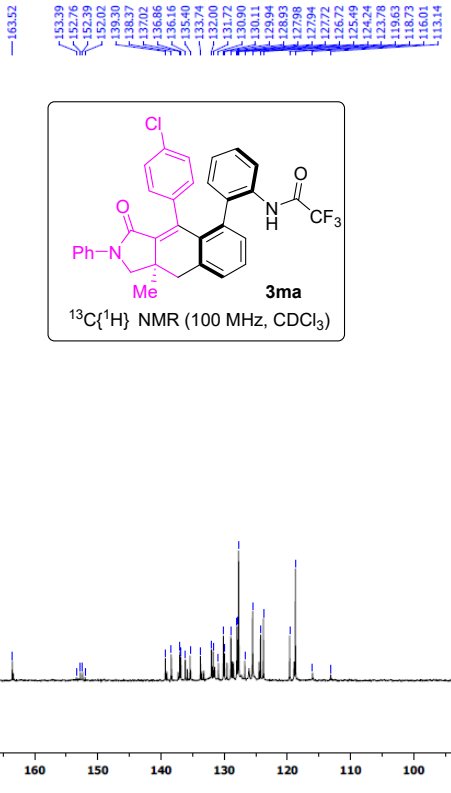
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

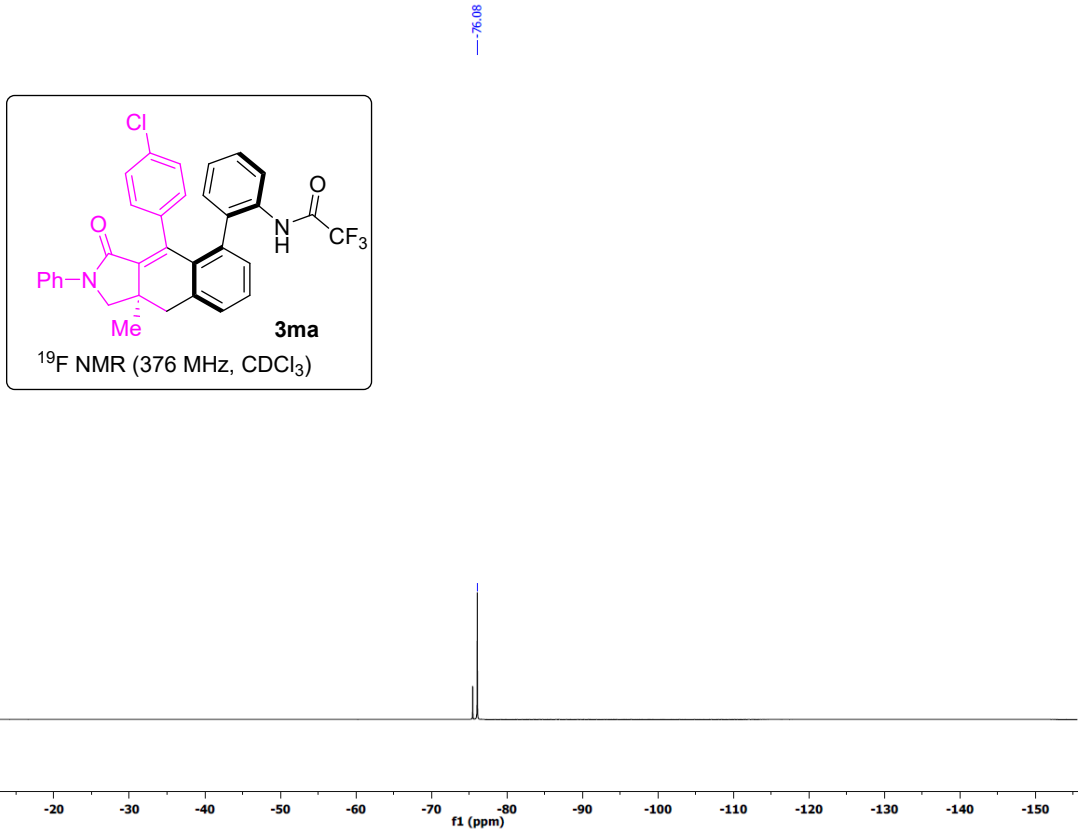

-76.12
-114.43
F
O
N
H
O
CF3
Ph–N
Me
3la
19F NMR (376 MHz, CDCl3)
f1 (ppm)

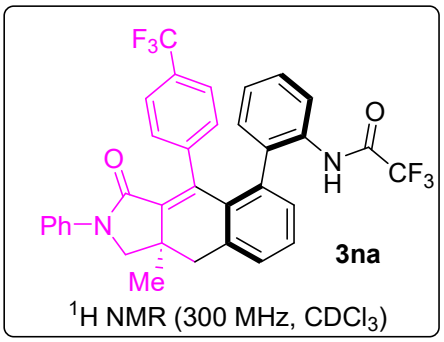

7.77
7.75
7.57
7.57
7.55
7.55
7.36
7.35
7.33
7.31
7.27
7.25
7.24
7.23
7.14
6.78
6.78
6.71
6.51
3.77
3.75
3.72
3.70
3.15
3.12
3.02
2.98
1.21


Cl
O
N
H
O
CF3
Ph–N
Me
3ma
1H NMR (400 MHz, CDCl3)

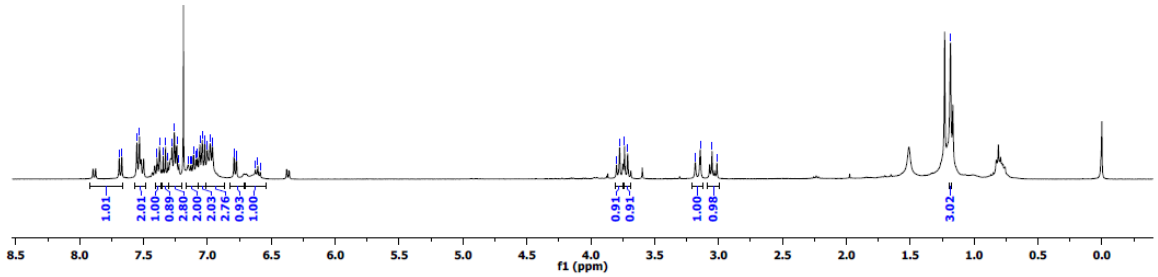

0.93
1.97
1.93
0.92
2.01
1.07
1.87
0.96
3.89
1.07
0.83
1.10
1.00
0.97
3.03
f1 (ppm)

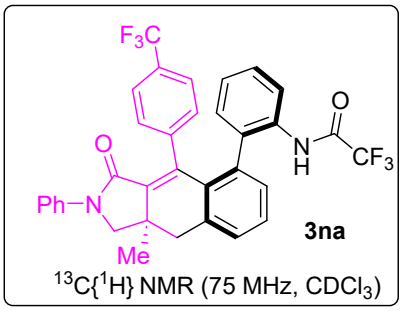

163.52
153.39
152.76
152.39
152.02
139.30
138.37
137.02
136.86
136.16
135.40
133.74
132.00
131.72
130.90
130.11
129.94
128.93
127.98
127.94
127.72
126.72
125.49
124.24
123.78
119.63
118.73
116.01
113.14
76.34
76.02
75.71
58.56
41.46
33.73
21.29
Cl
O
N
H
O
CF3
Ph—N
Me
3ma
13C{1H} NMR (100 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-76.08
Cl
O
N
H
O
CF3
Ph—N
Me
3ma
19F NMR (376 MHz, CDCl3)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)

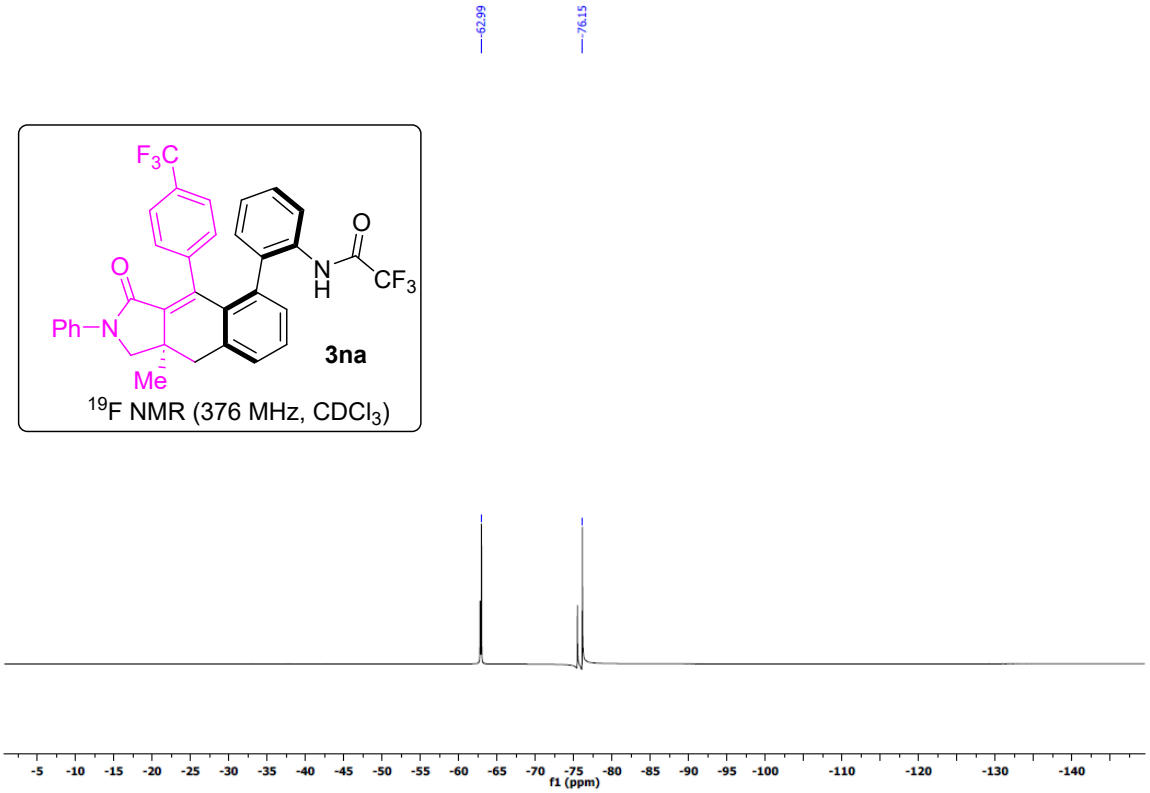


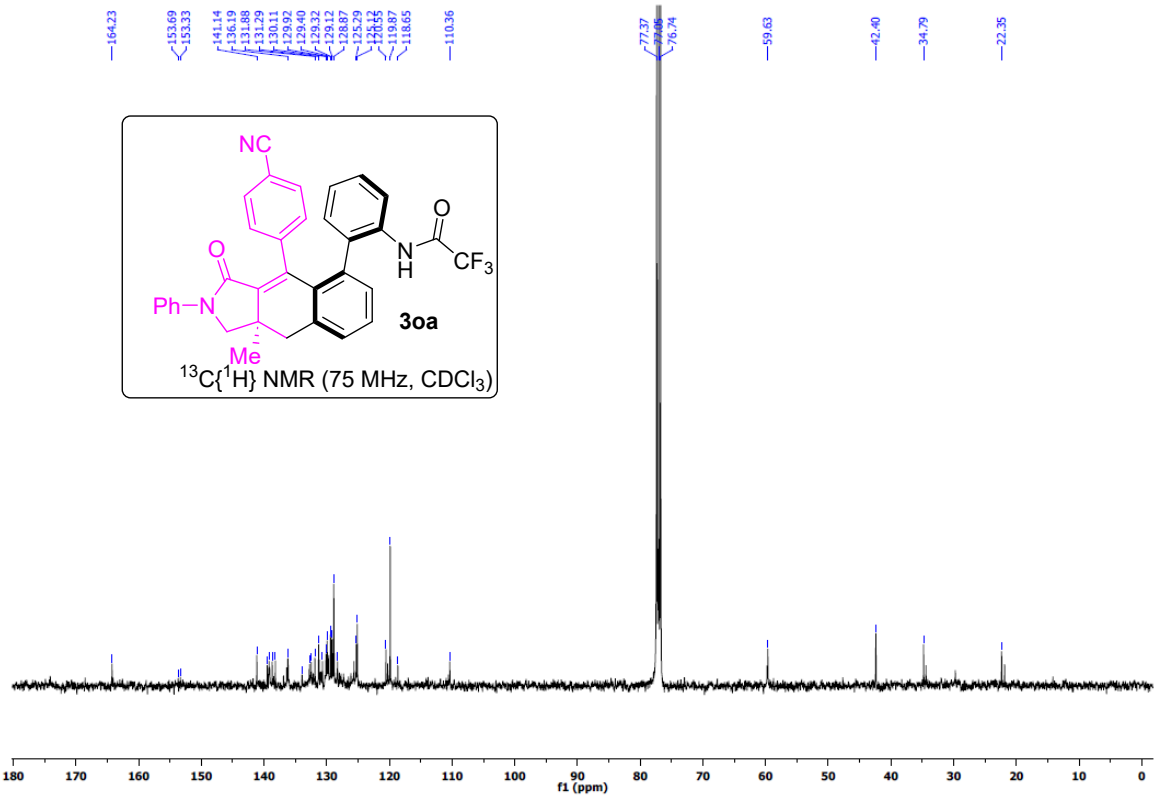
^{1}H NMR (300 MHz, $CDCl_3$)

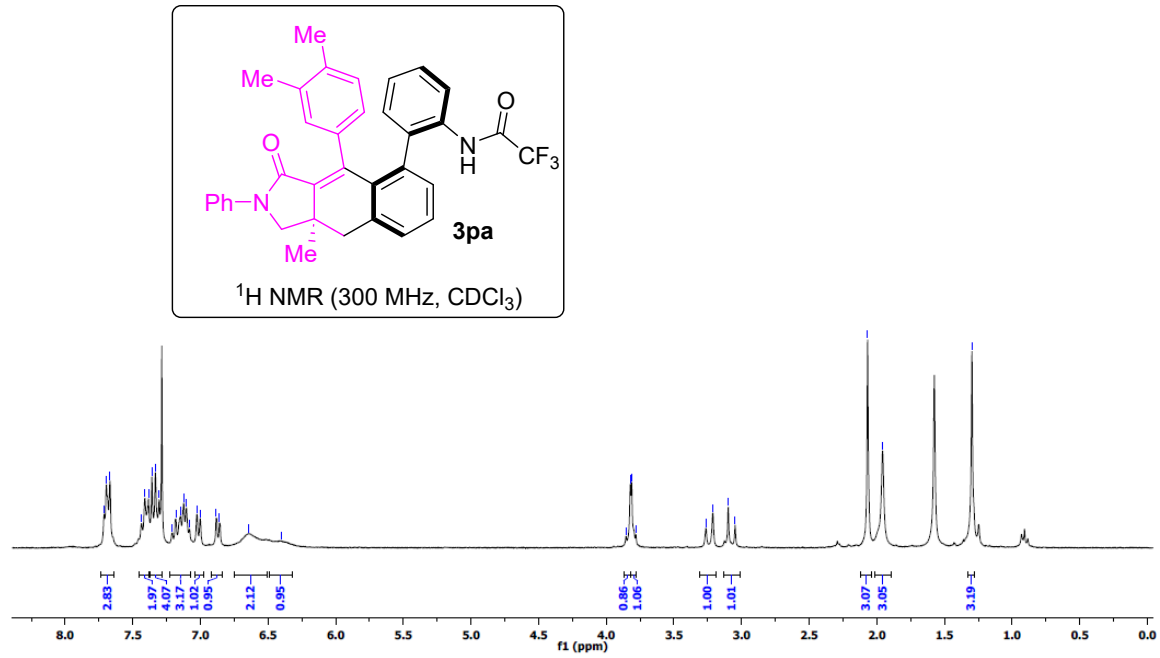


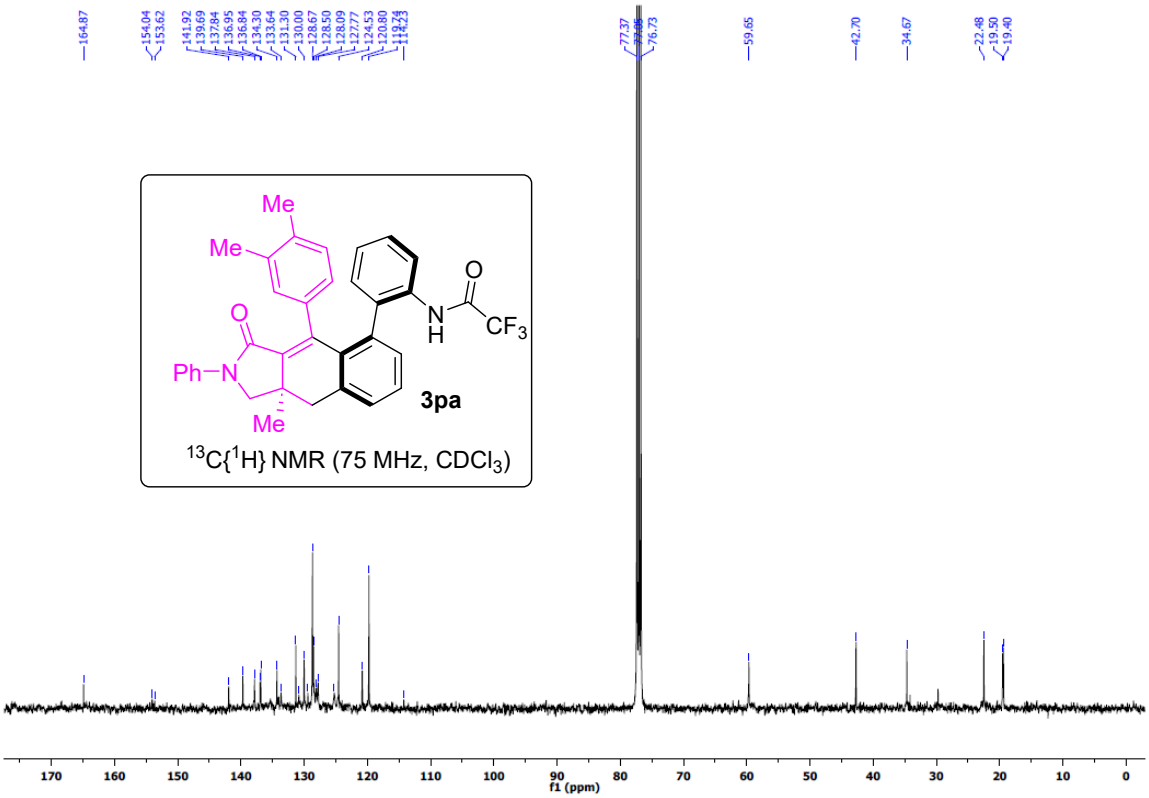


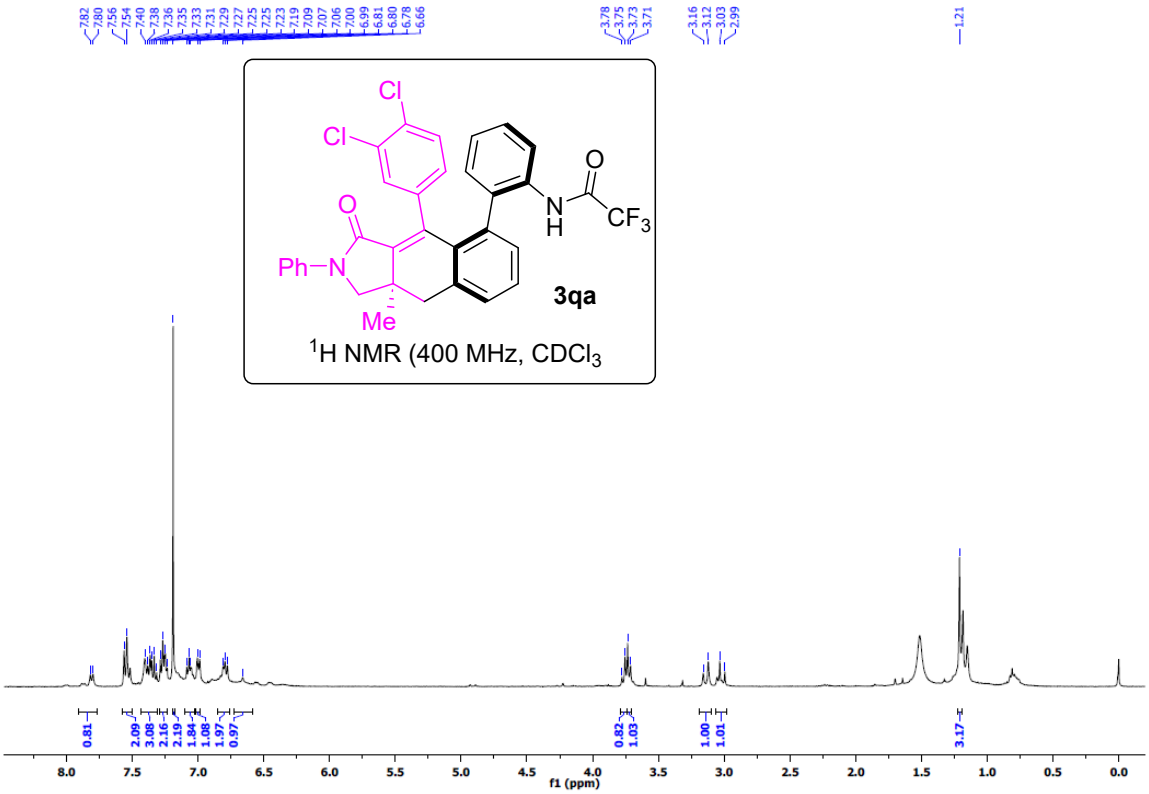
^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$)

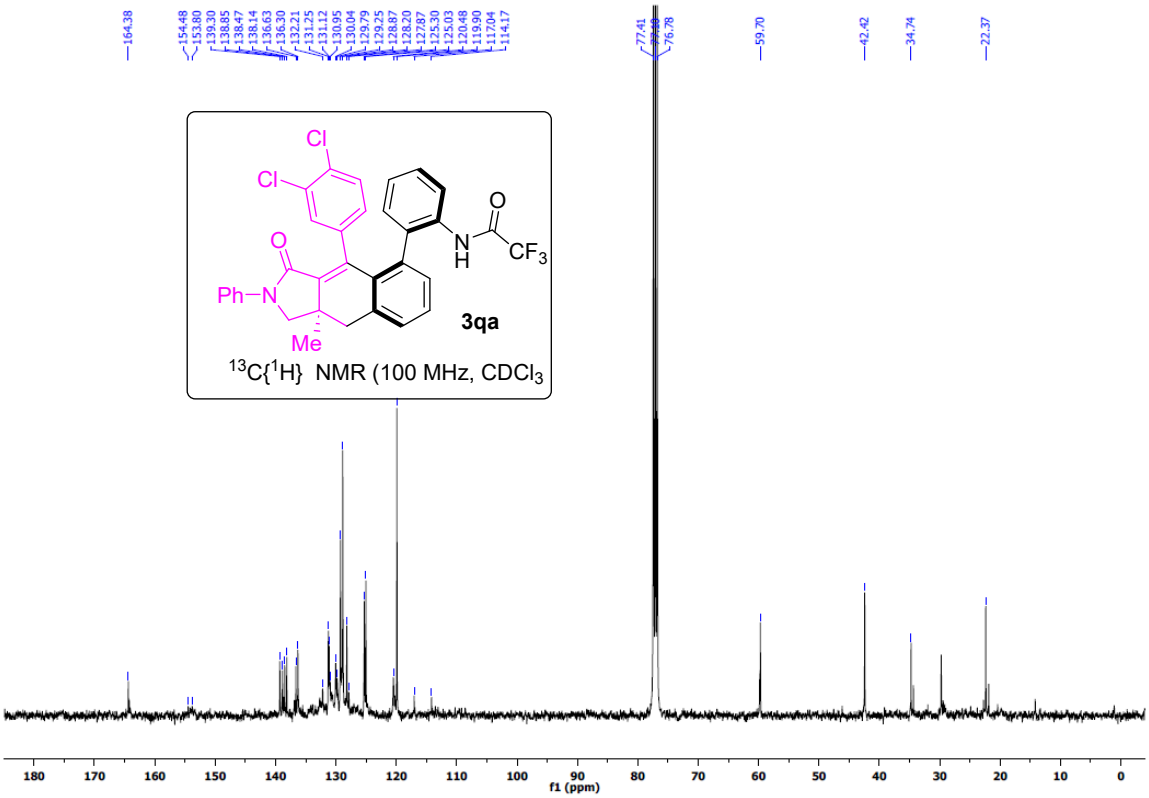

62.99
76.15
F3C
O
N
H
CF3
O
Ph–N
Me
3na
19F NMR (376 MHz, CDCl3)
f1 (ppm)
7.74
7.71
7.54
7.52
7.42
7.39
7.37
7.37
7.35
7.29
7.27
7.26
7.25
7.23
7.23
7.22
7.14
7.11
7.09
7.07
7.05
7.03
7.00
6.98
6.81
6.81
6.79
6.78
6.71
6.69
6.66
3.80
3.76
3.74
3.72
3.18
3.13
3.06
3.01
1.23
NC
O
N
H
CF3
O
Ph–N
Me
3oa
1H NMR (300 MHz, CDCl3)
1.07
2.05
2.08
2.90
4.18
1.85
0.79
2.12
0.79
1.09
0.96
1.00
2.96
f1 (ppm)

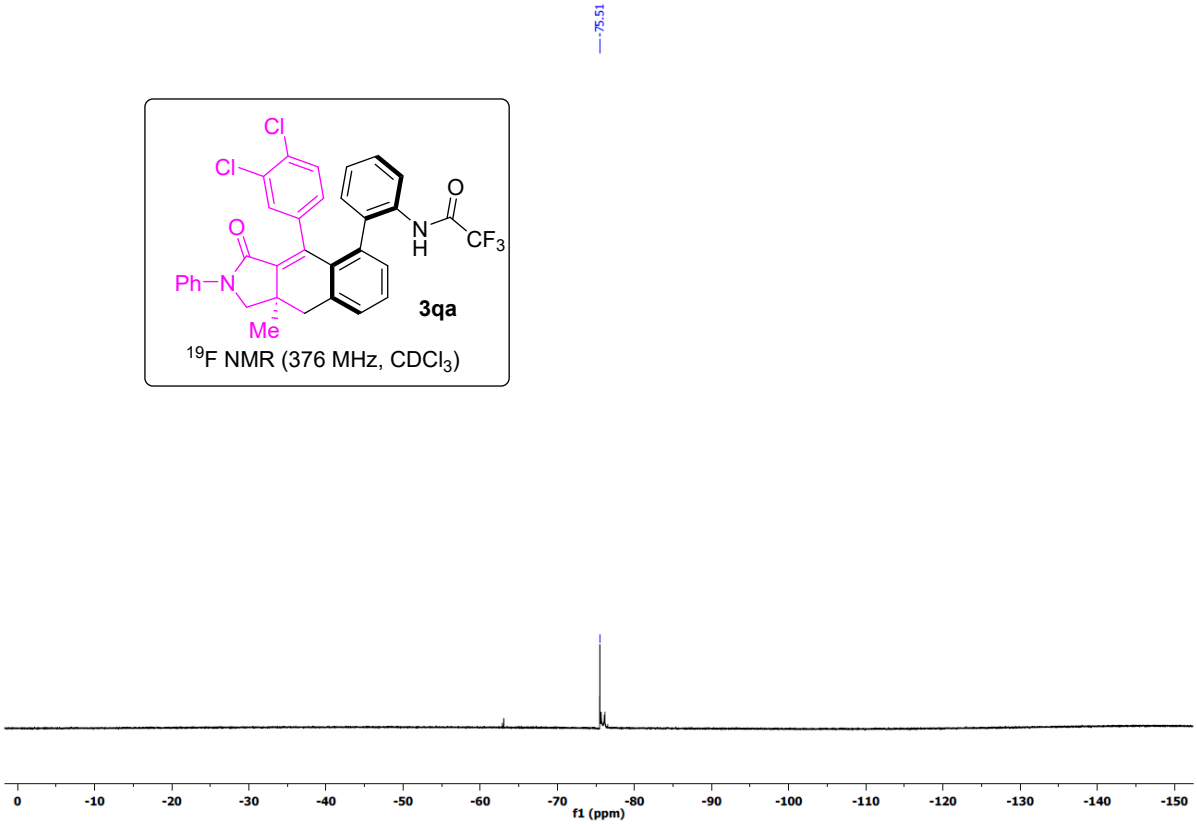

NC
O
N
H
CF3
O
Ph–N
Me
3oa
13C{1H} NMR (75 MHz, CDCl3)
f1 (ppm)

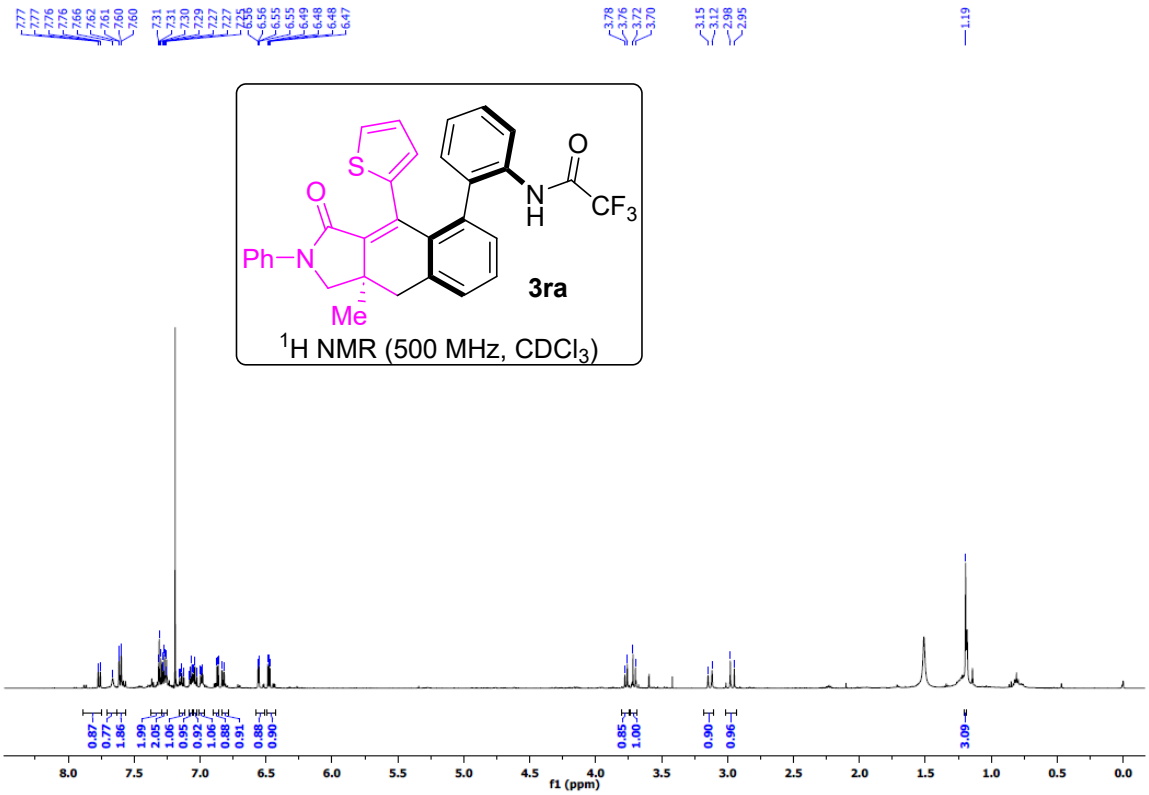

Me
Me
O
N
H
CF3
Ph–N
Me
3pa
1H NMR (300 MHz, CDCl3)
f1 (ppm)

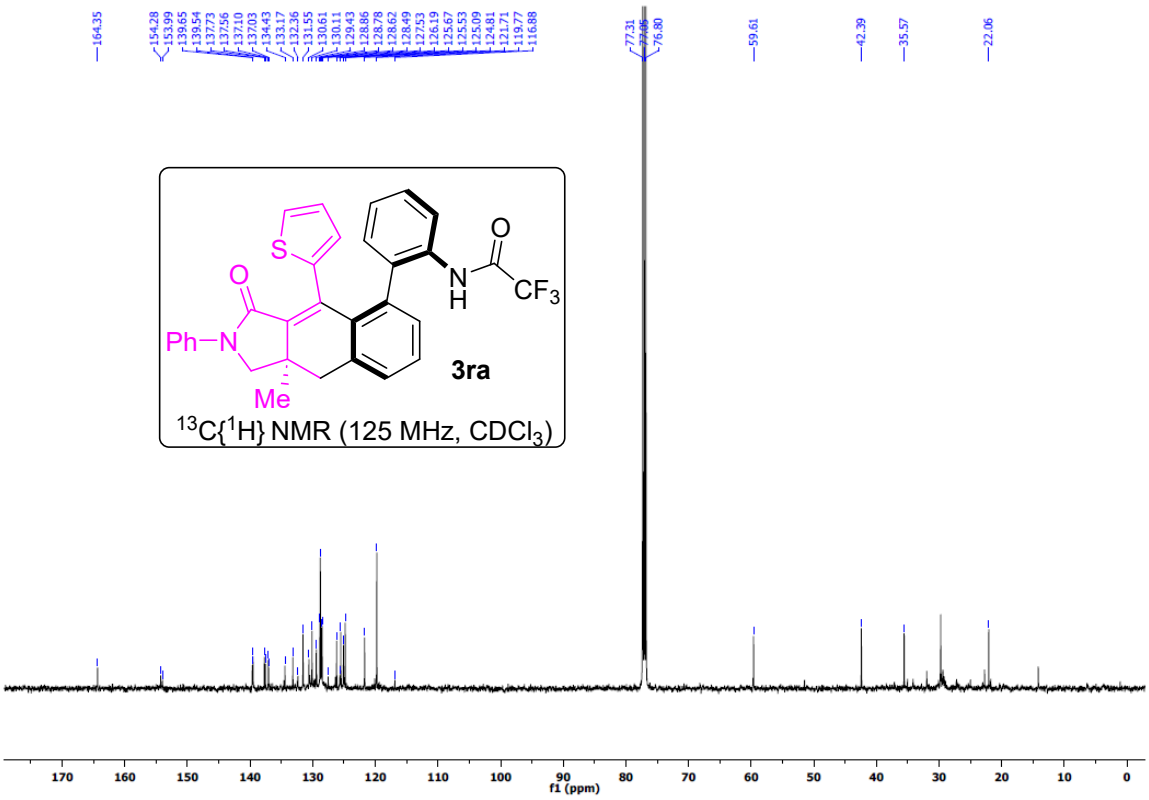

Me
Me
O
Ph–N
Me
N
H
O
CF3
3pa
13C{1H} NMR (75 MHz, CDCl3)
f1 (ppm)

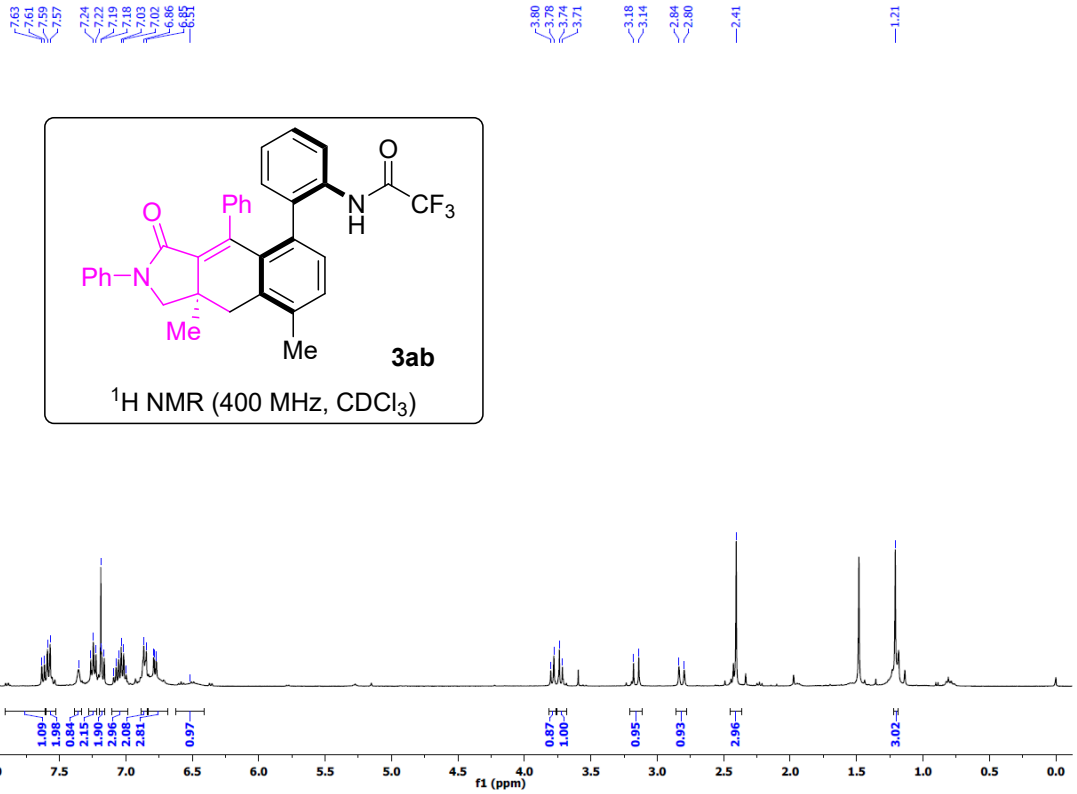

Cl
Cl
O
Ph–N
Me
N
H
O
CF3
3qa
1H NMR (400 MHz, CDCl3
f1 (ppm)

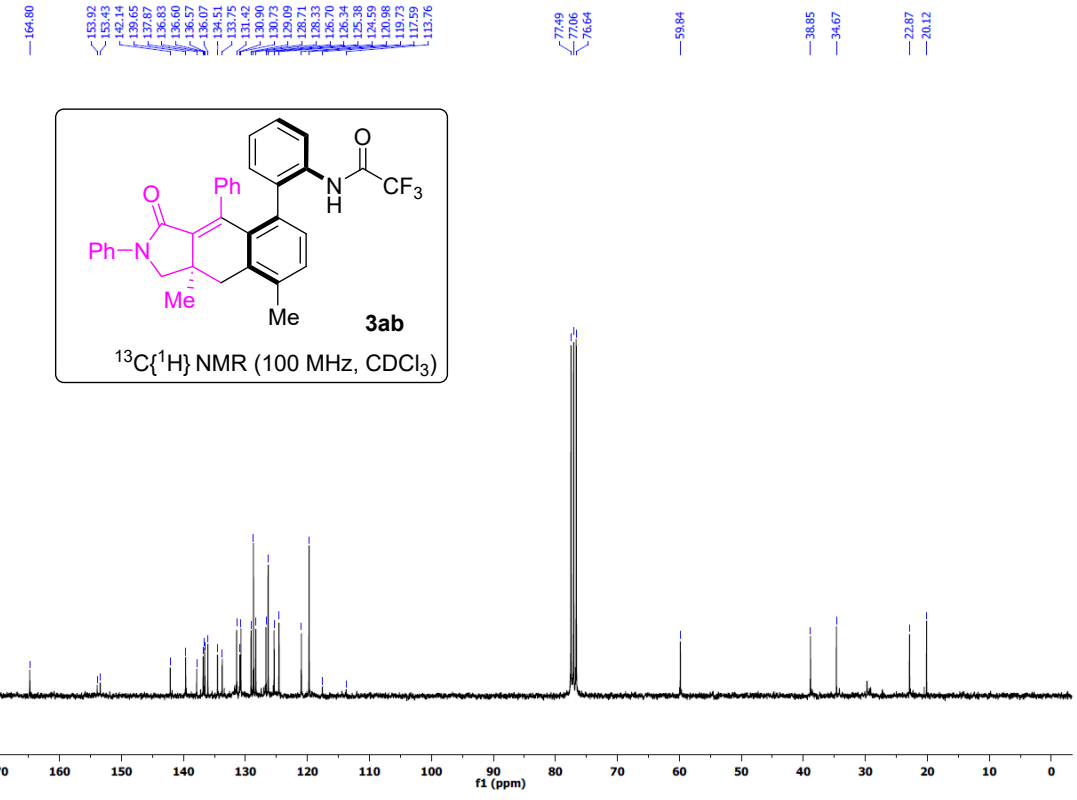

164.38
154.48
153.80
139.30
138.85
138.47
138.14
136.63
136.30
132.21
131.25
131.12
130.95
130.04
129.79
129.25
128.87
128.20
127.87
125.30
125.03
120.48
119.90
117.04
114.17
77.41
77.16
76.78
59.70
42.42
34.74
22.37
Cl
Cl
O
Ph–N
Me
N
H
O
CF3
3qa
13C{1H} NMR (100 MHz, CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

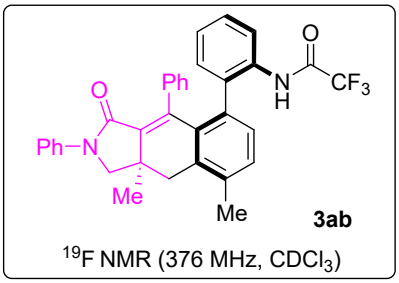

-75.51
Cl
Cl
O
Ph–N
Me
N
H
O
CF3
3qa
19F NMR (376 MHz, CDCl3)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
f1 (ppm)


S
O
Ph–N
Me
N
H
O
CF3
3ra
1H NMR (500 MHz, CDCl3)
f1 (ppm)


S
O
Ph–N
Me
N
H
O
CF3
3ra
13C{1H} NMR (125 MHz, CDCl3)
f1 (ppm)

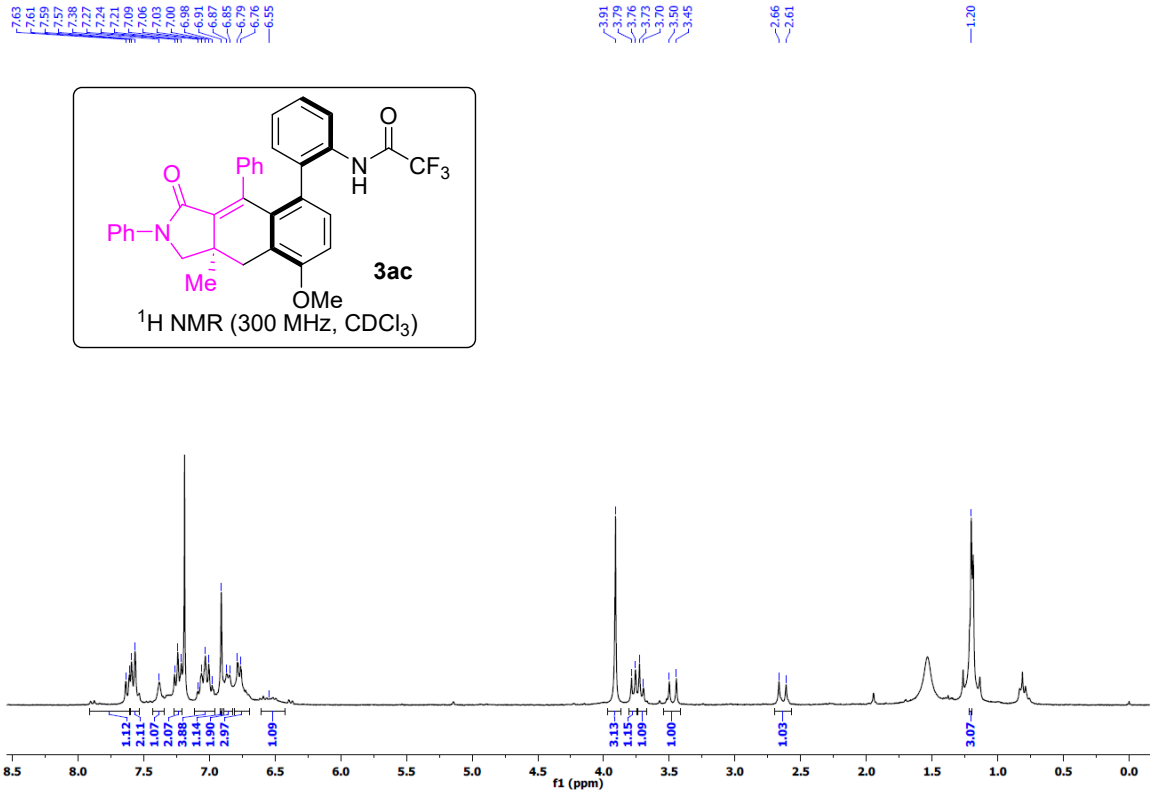

7.63
7.61
7.59
7.57
7.24
7.22
7.19
7.18
7.03
7.02
6.86
6.85
6.81
3.80
3.78
3.74
3.71
3.18
3.14
2.84
2.80
2.41
1.21
O
N
H
CF3
Ph
O
Ph—N
Me
Me
3ab
1H NMR (400 MHz, CDCl3)
1.09
1.98
0.84
2.15
1.90
2.96
2.08
2.81
0.97
0.87
1.00
0.95
0.93
2.96
3.02
f1 (ppm)

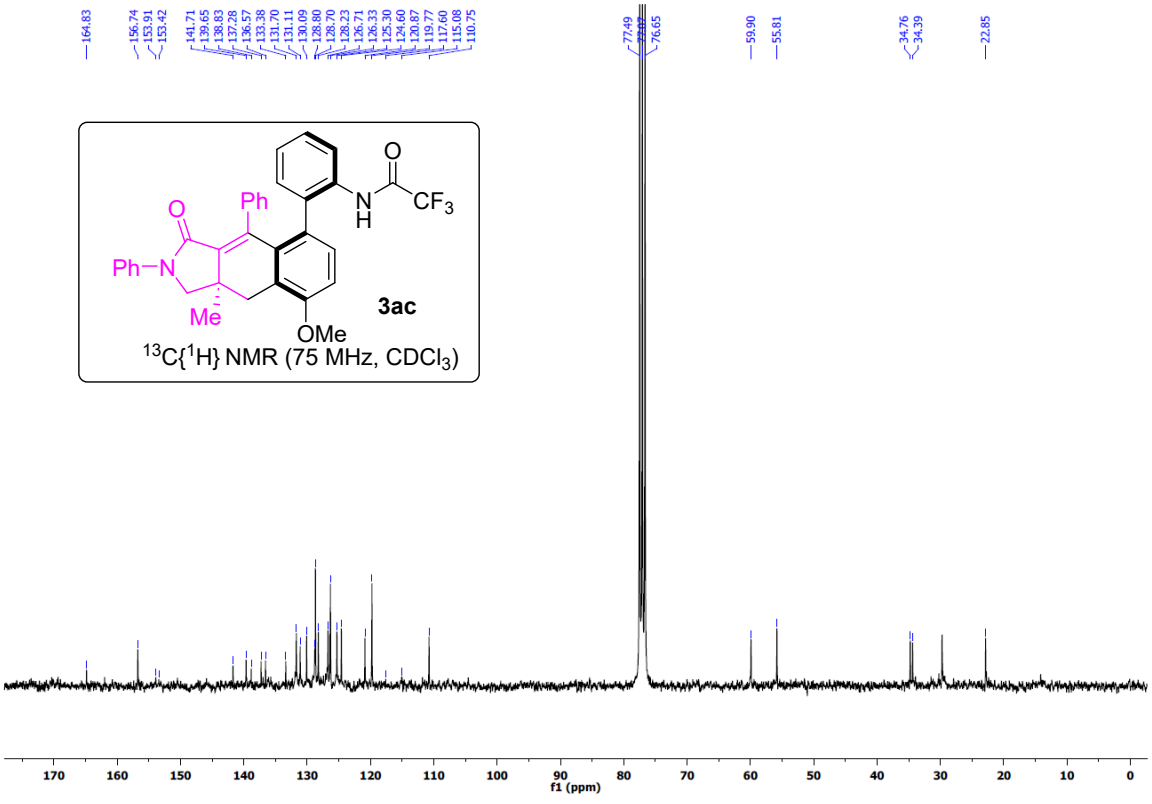

164.80
153.92
153.43
142.14
139.65
137.87
136.83
136.60
136.57
136.07
134.51
133.75
131.42
130.90
130.73
129.09
128.71
128.33
126.70
126.34
125.38
124.59
120.98
119.73
117.59
113.76
77.49
77.06
76.64
59.84
38.85
34.67
22.87
20.12
O
N
H
CF3
Ph
O
Ph—N
Me
Me
3ab
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)

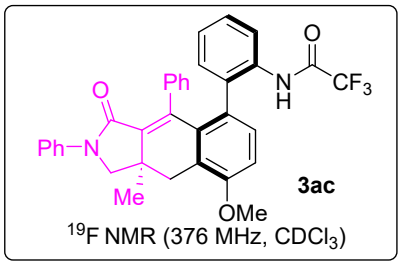


3ab

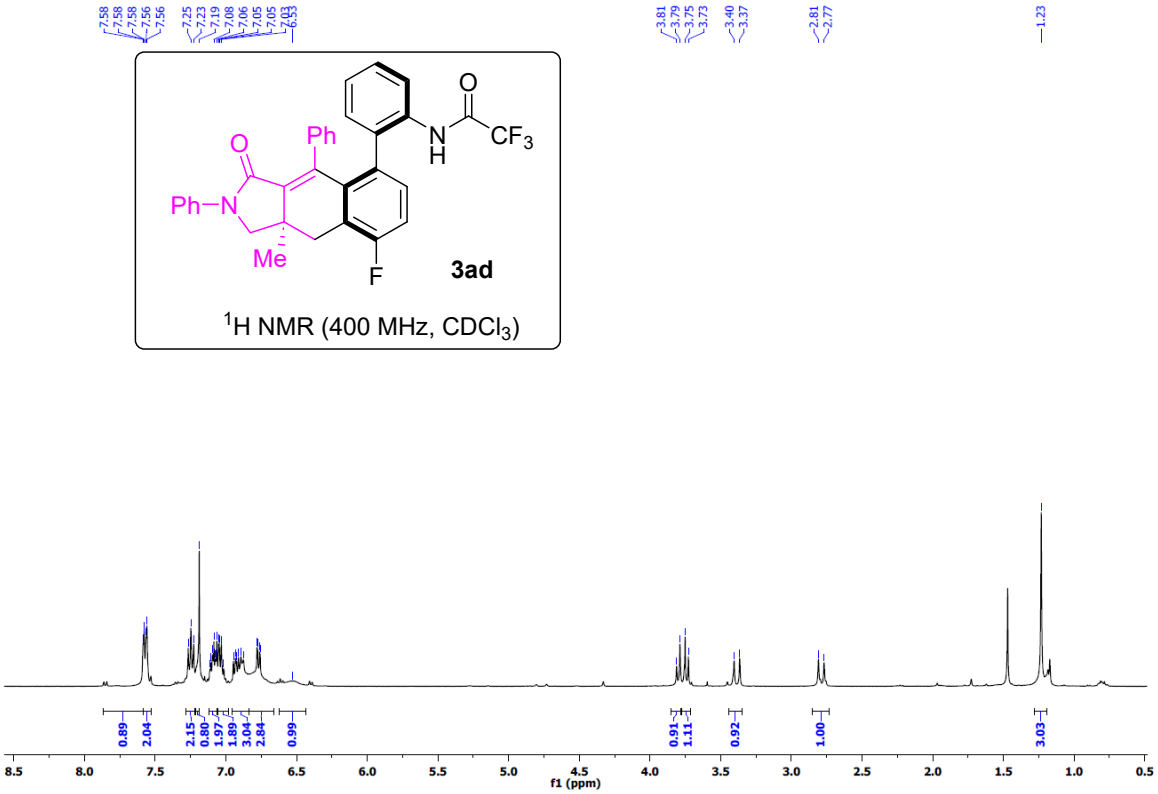
^{19}F NMR (376 MHz, $CDCl_3$)

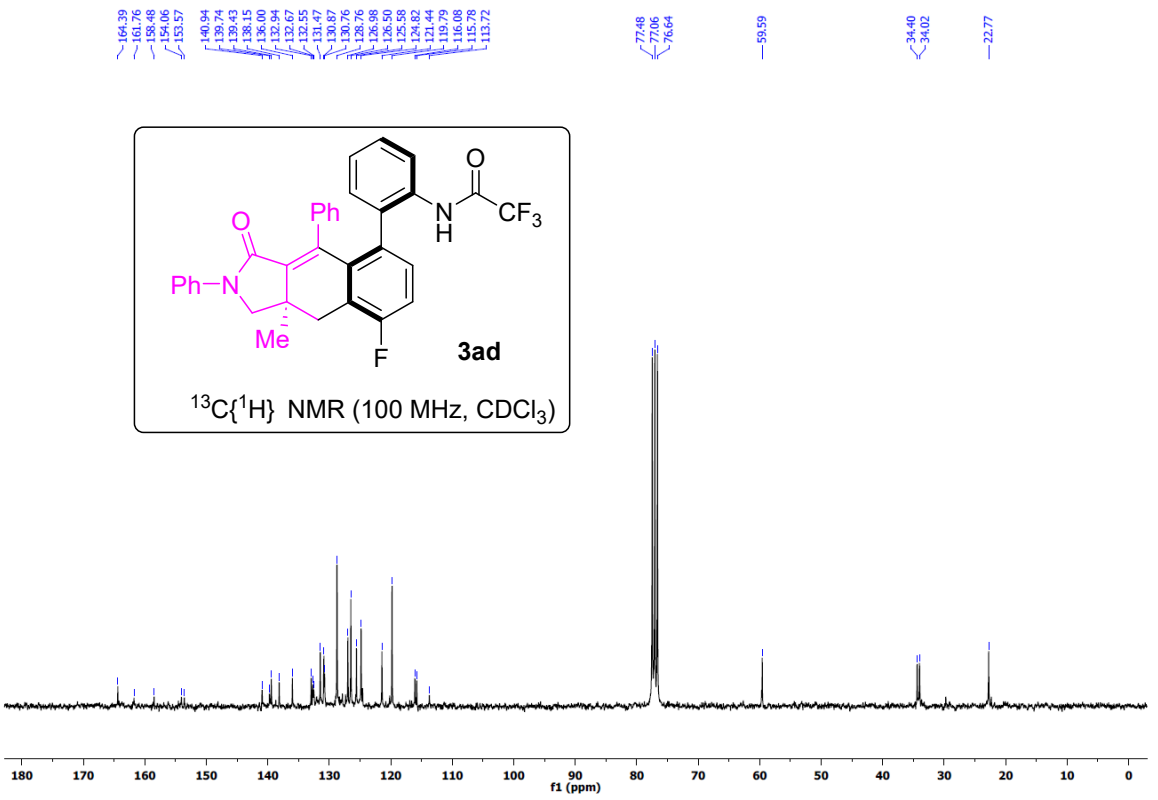

7.63
7.61
7.59
7.57
7.38
7.27
7.24
7.21
7.09
7.06
7.03
7.00
6.98
6.91
6.87
6.85
6.79
6.76
6.55
3.91
3.79
3.76
3.73
3.70
3.50
3.45
2.66
2.61
1.20
O
Ph
N
H
CF3
O
Ph—N
Me
OMe
3ac
1H NMR (300 MHz, CDCl3)
1.12
2.11
1.07
2.07
3.88
1.14
1.90
2.97
1.09
3.13
1.15
1.09
1.00
1.03
3.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

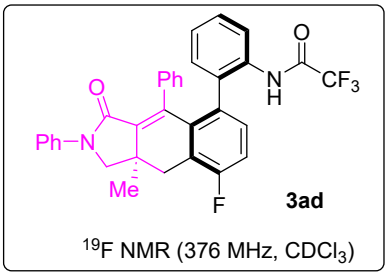

164.83
156.74
153.91
153.42
141.71
139.65
138.83
137.28
136.57
133.38
131.70
131.11
130.09
128.80
128.70
128.23
126.71
126.33
125.30
124.60
120.87
119.77
117.60
115.08
110.75
77.49
77.16
76.65
59.90
55.81
34.76
34.39
22.85
O
Ph
N
H
CF3
O
Ph—N
Me
OMe
3ac
13C{1H} NMR (75 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{19}F NMR (376 MHz, $CDCl_3$)

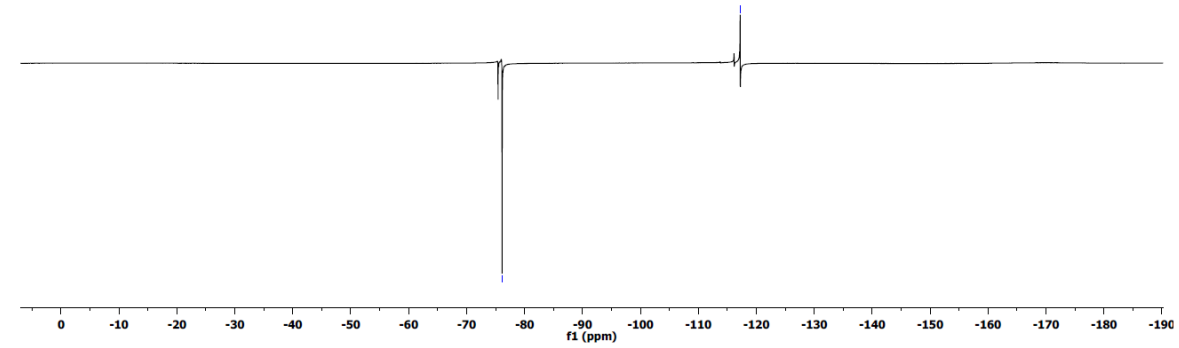

Ph
O
N
H
CF3
Ph–N
Me
F
3ad
1H NMR (400 MHz, CDCl3)
f1 (ppm)

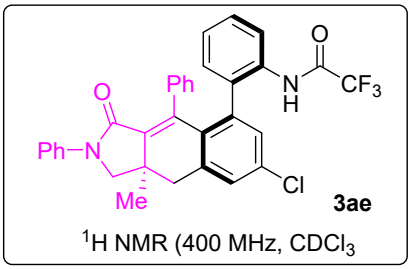

Ph
O
N
H
CF3
Ph–N
Me
F
3ad
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)



3ad

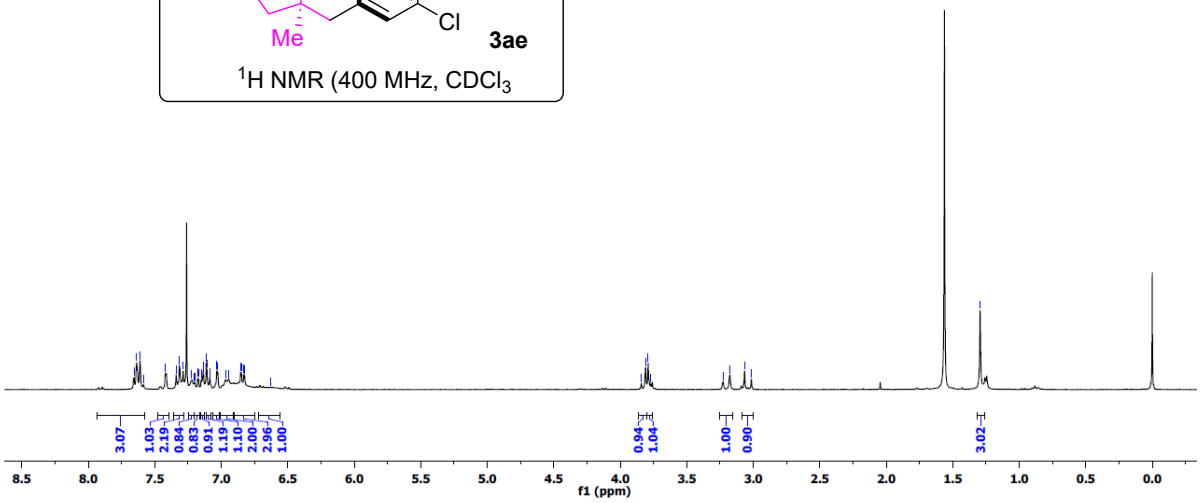
^{19}F NMR (376 MHz, $CDCl_3$)

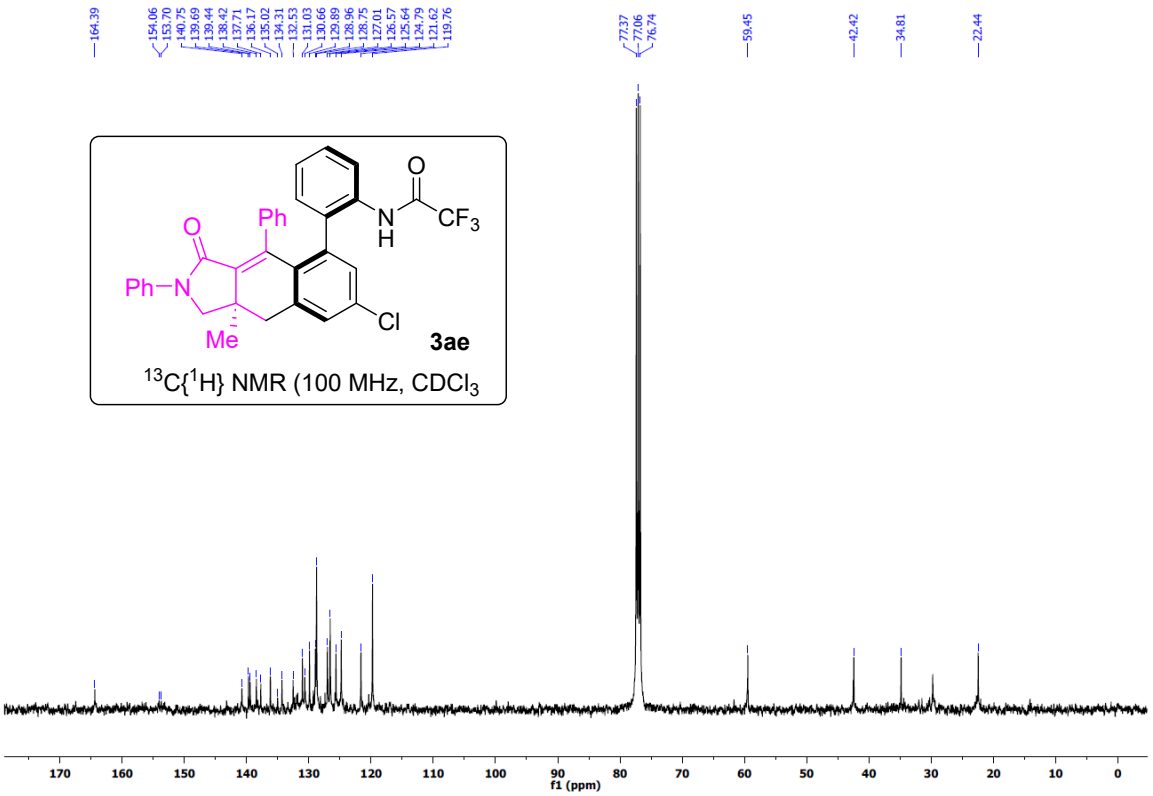


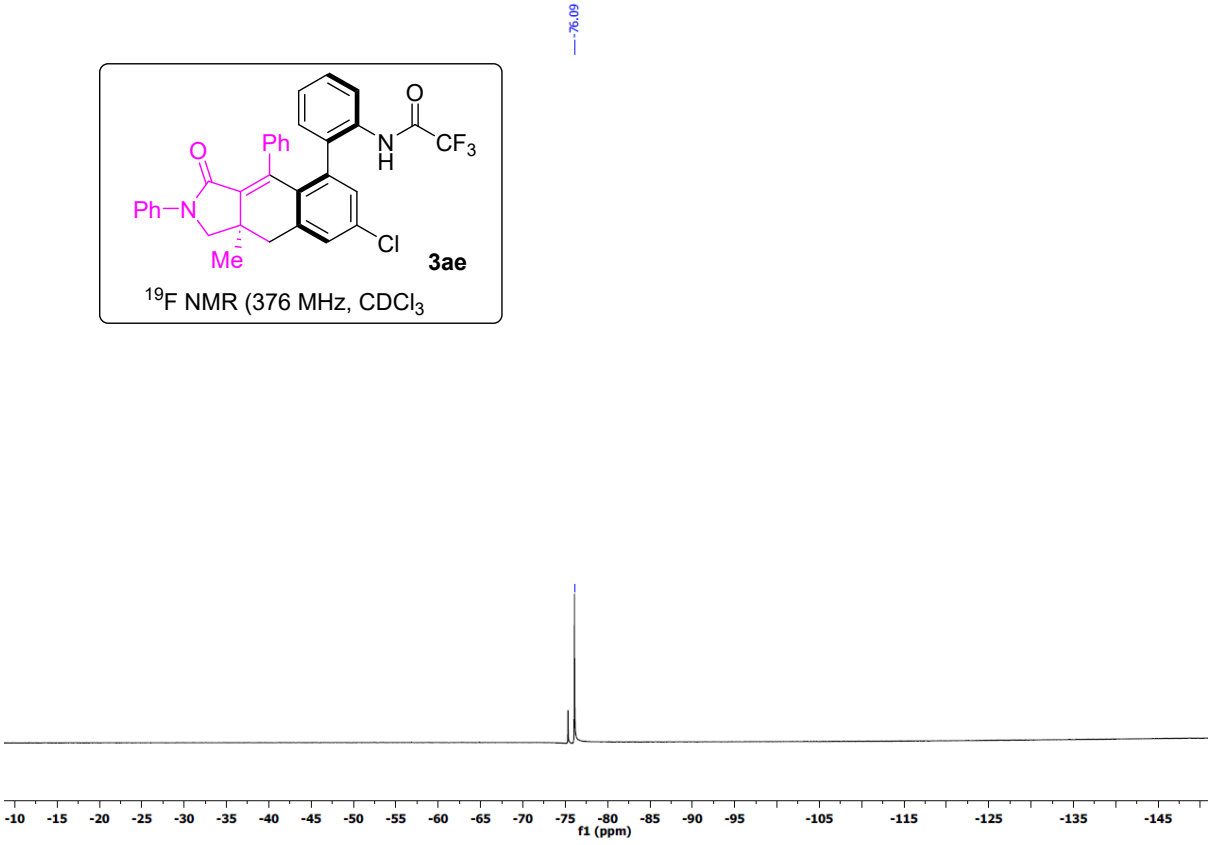


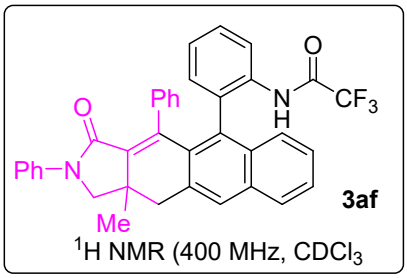
3ae

^{1}H NMR (400 MHz, $CDCl_3$

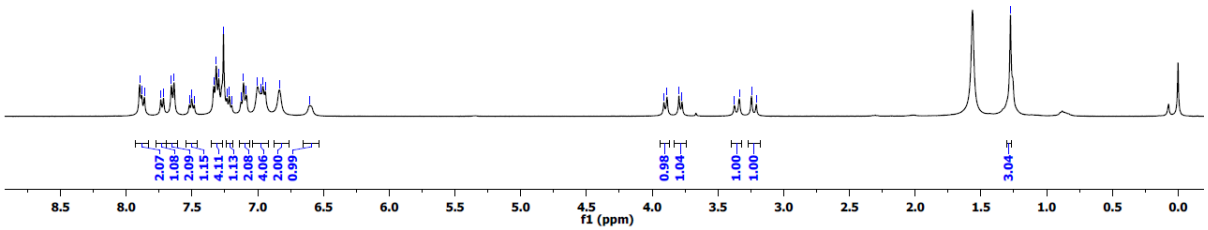


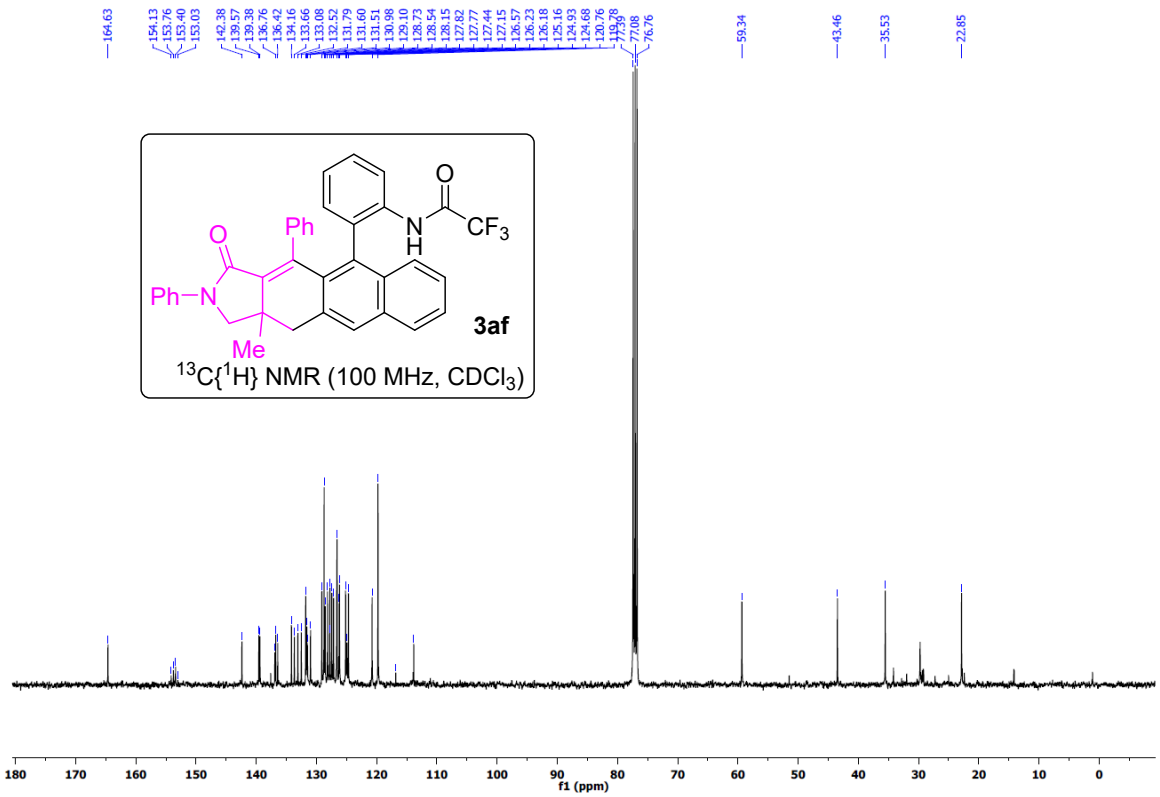

164.39
154.06
153.70
140.75
139.69
139.44
138.42
137.71
136.17
135.02
134.31
132.53
131.03
130.66
129.89
128.96
128.75
127.01
126.57
125.64
124.79
121.62
119.76
77.37
77.06
76.74
59.45
42.42
34.81
22.44
O
Ph
N
H
CF3
O
Ph—N
Cl
Me
3ae
13C{1H} NMR (100 MHz, CDCl3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-76.09
O
Ph
N
H
CF3
O
Ph—N
Cl
Me
3ae
19F NMR (376 MHz, CDCl3
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-105
-115
-125
-135
-145
f1 (ppm)

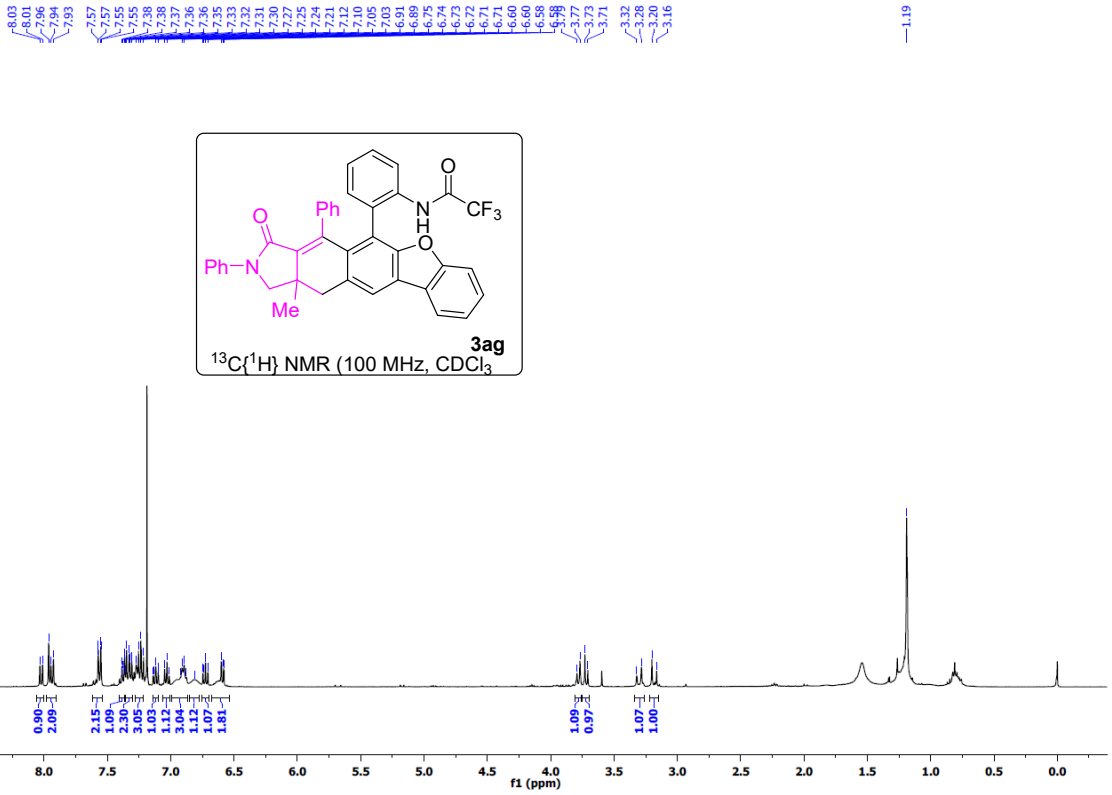


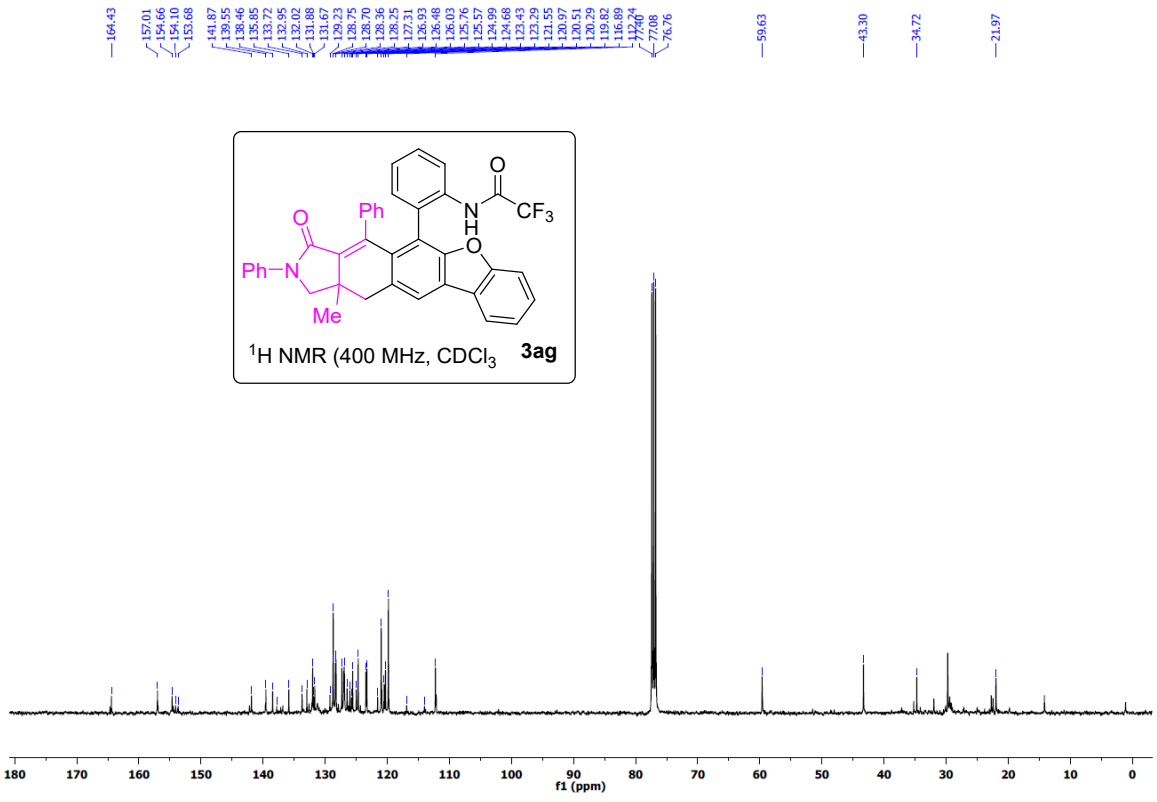
^{1}H NMR (400 MHz, $CDCl_3$)

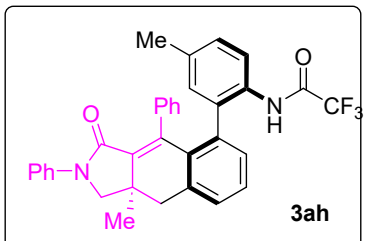




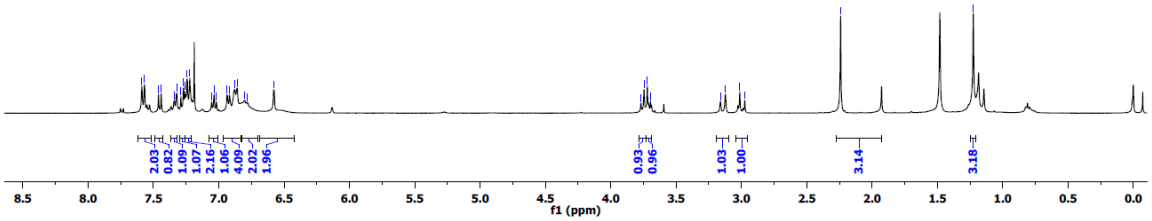
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

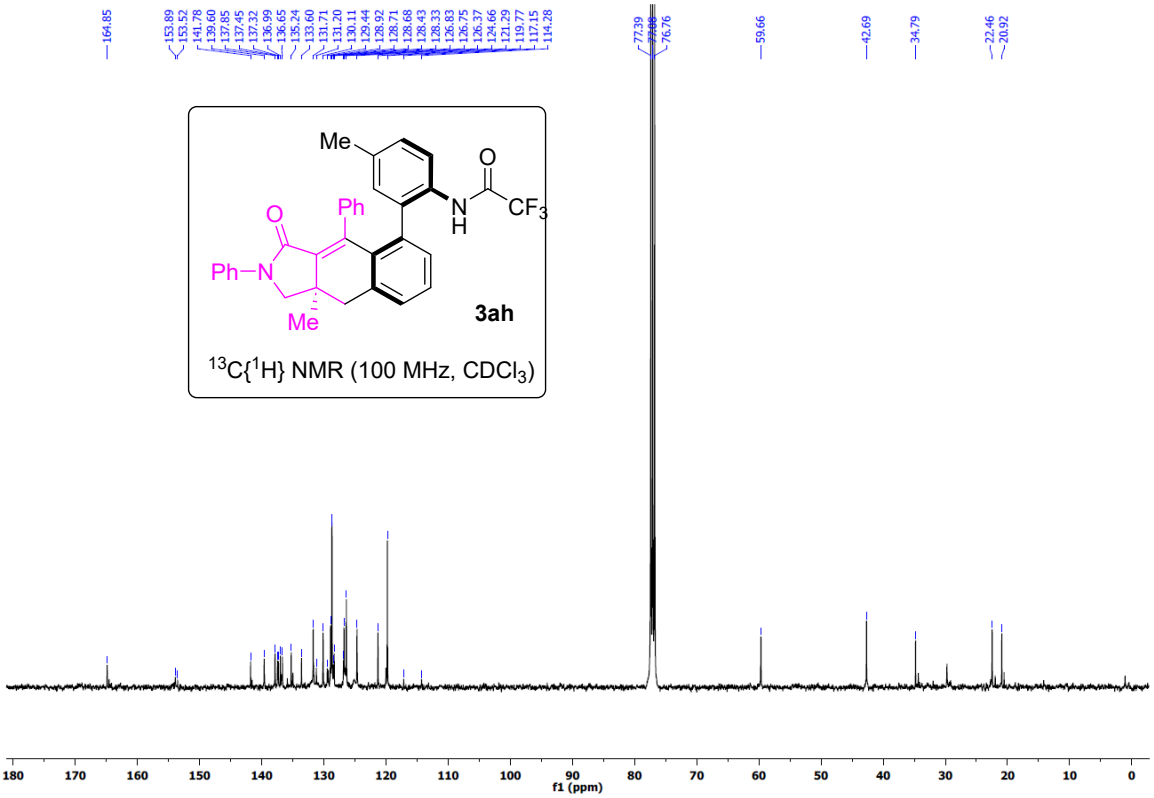

Ph
O
N
H
CF3
Ph–N
Me
3ag
13C{1H} NMR (100 MHz, CDCl3
f1 (ppm)


Ph
O
N
H
CF3
Ph–N
Me
1H NMR (400 MHz, CDCl3
3ag
f1 (ppm)

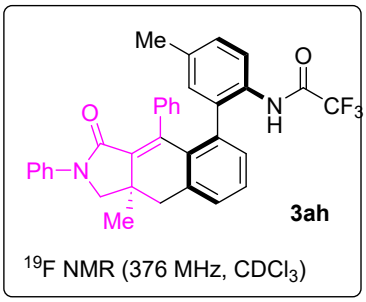


^{1}H NMR (400 MHz, $CDCl_3$)

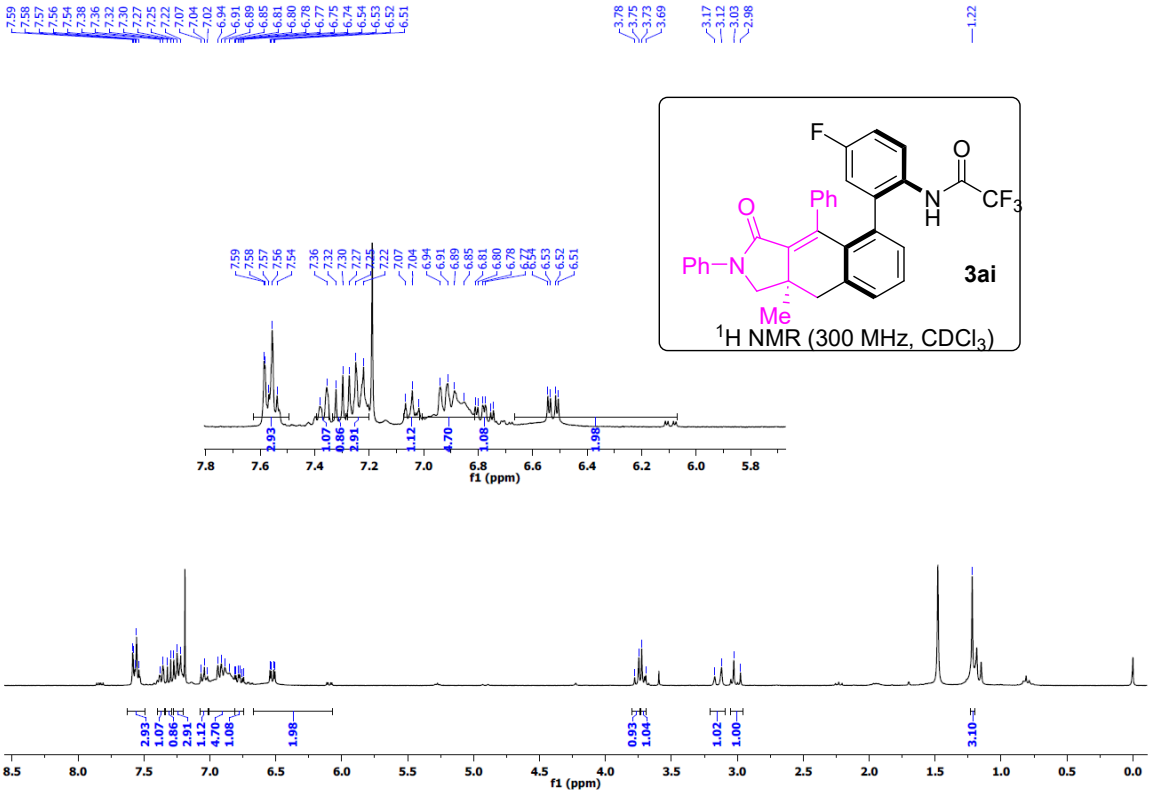




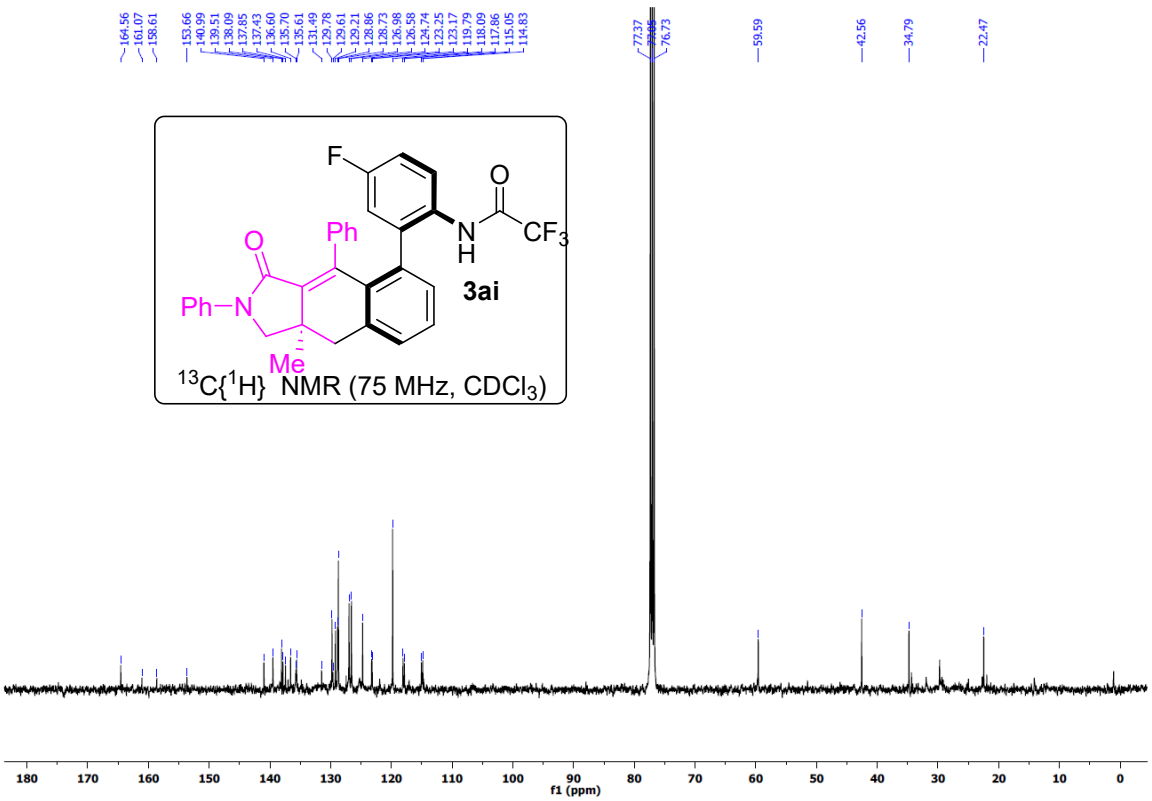
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

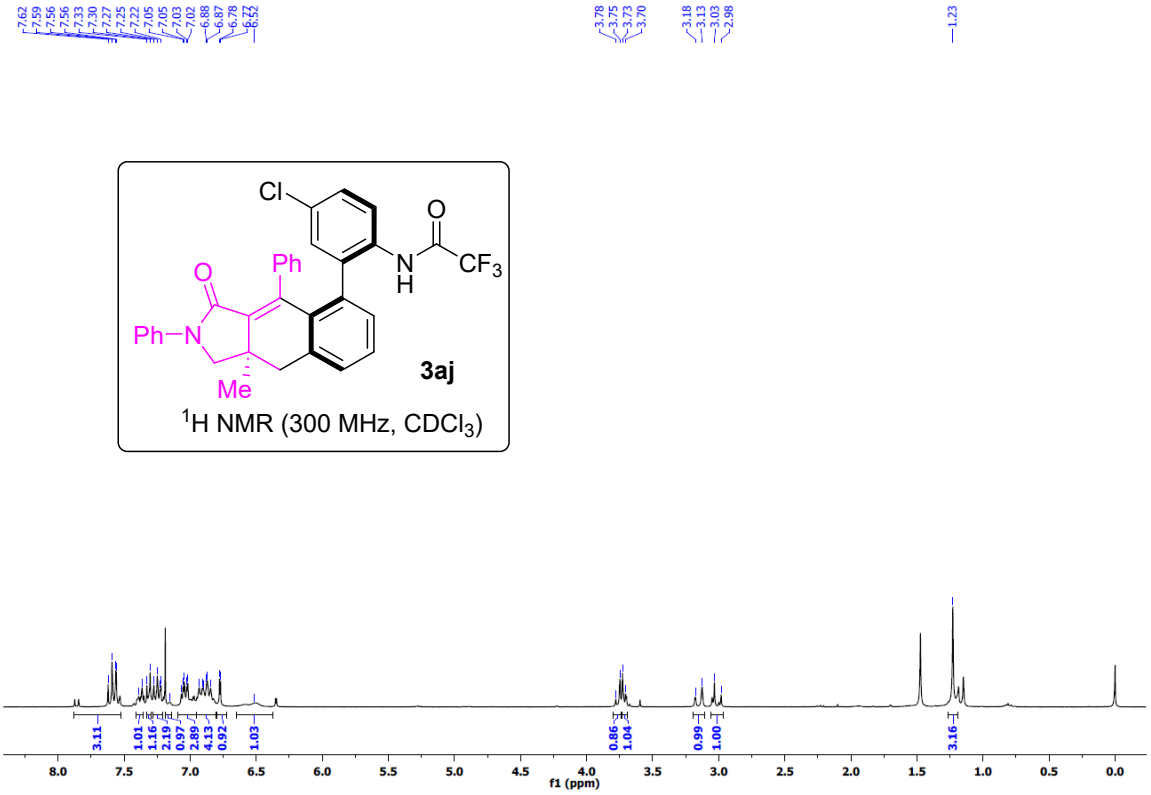


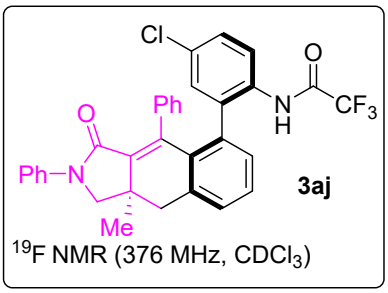
^{19}F NMR (376 MHz, $CDCl_3$)


F
O
N
H
CF3
Ph
O
Ph—N
Me
3ai
1H NMR (300 MHz, CDCl3)
f1 (ppm)

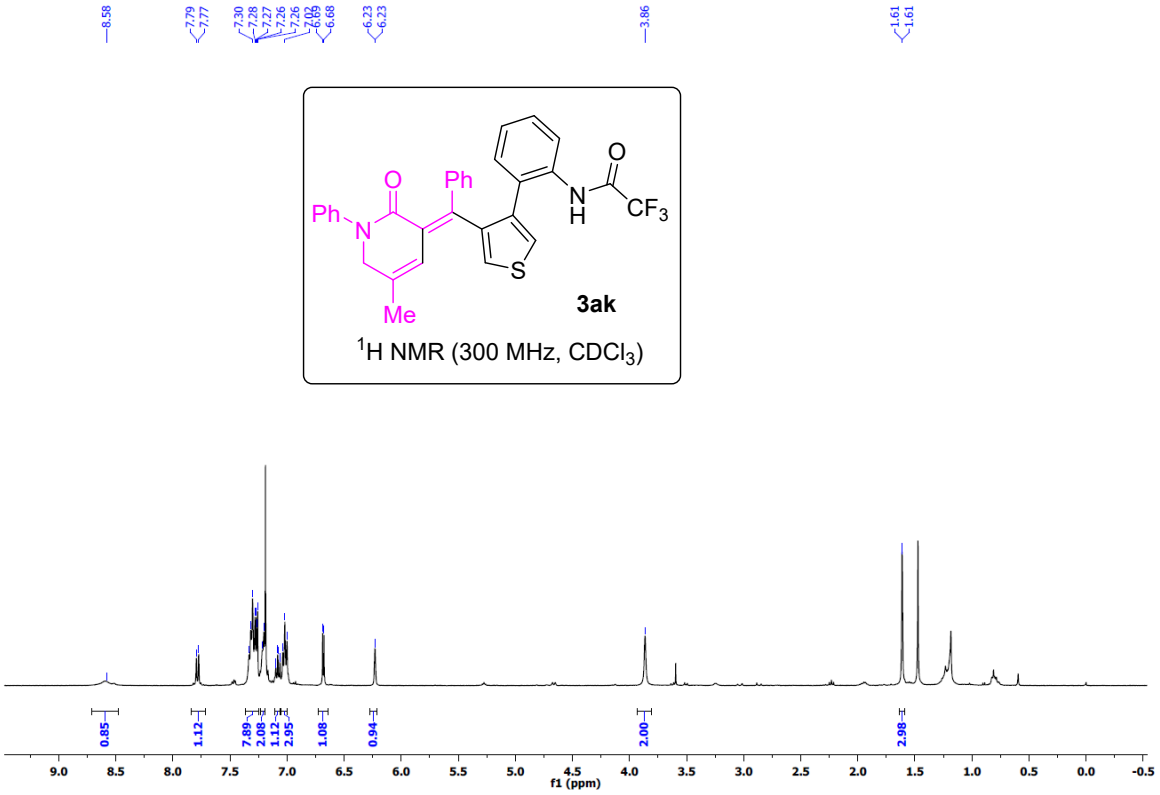
f1 (ppm)

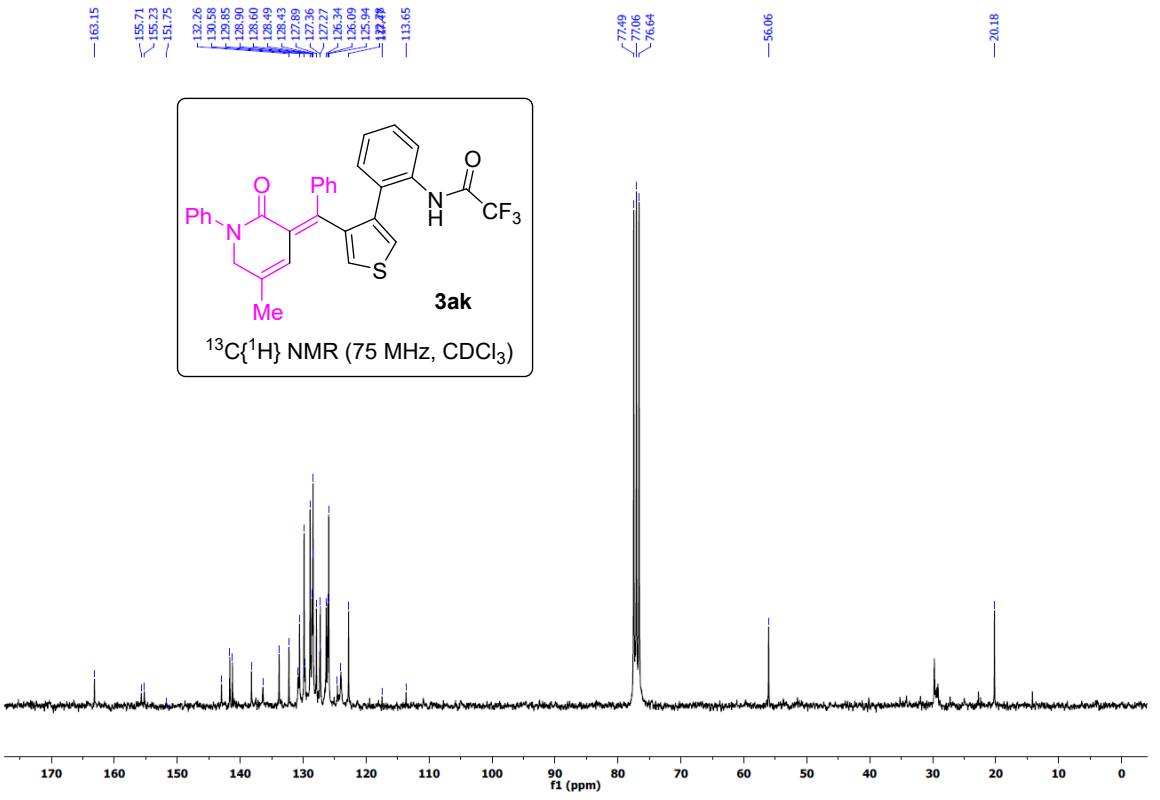

F
O
N
H
CF3
Ph
O
Ph—N
Me
3ai
13C{1H} NMR (75 MHz, CDCl3)
f1 (ppm)

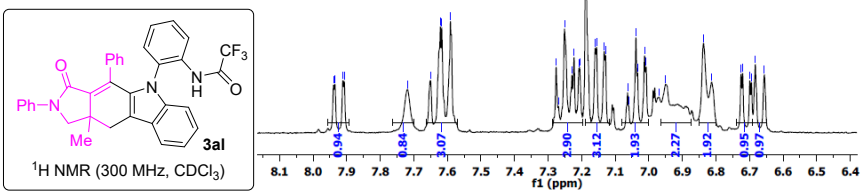

Cl
O
N
H
CF3
Ph
O
Ph–N
Me
3aj
1H NMR (300 MHz, CDCl3)
f1 (ppm)
13C{1H} NMR (75 MHz, CDCl3)

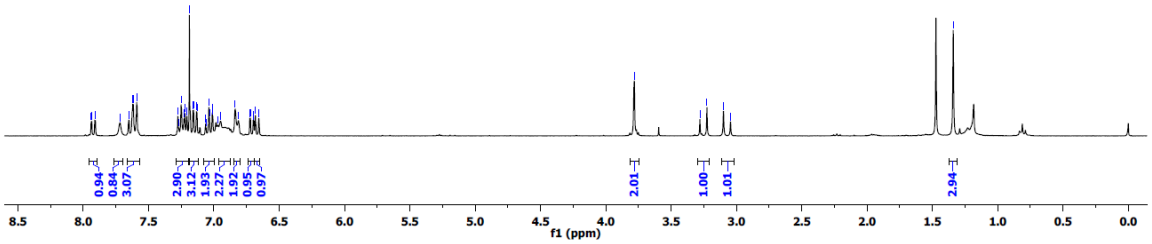


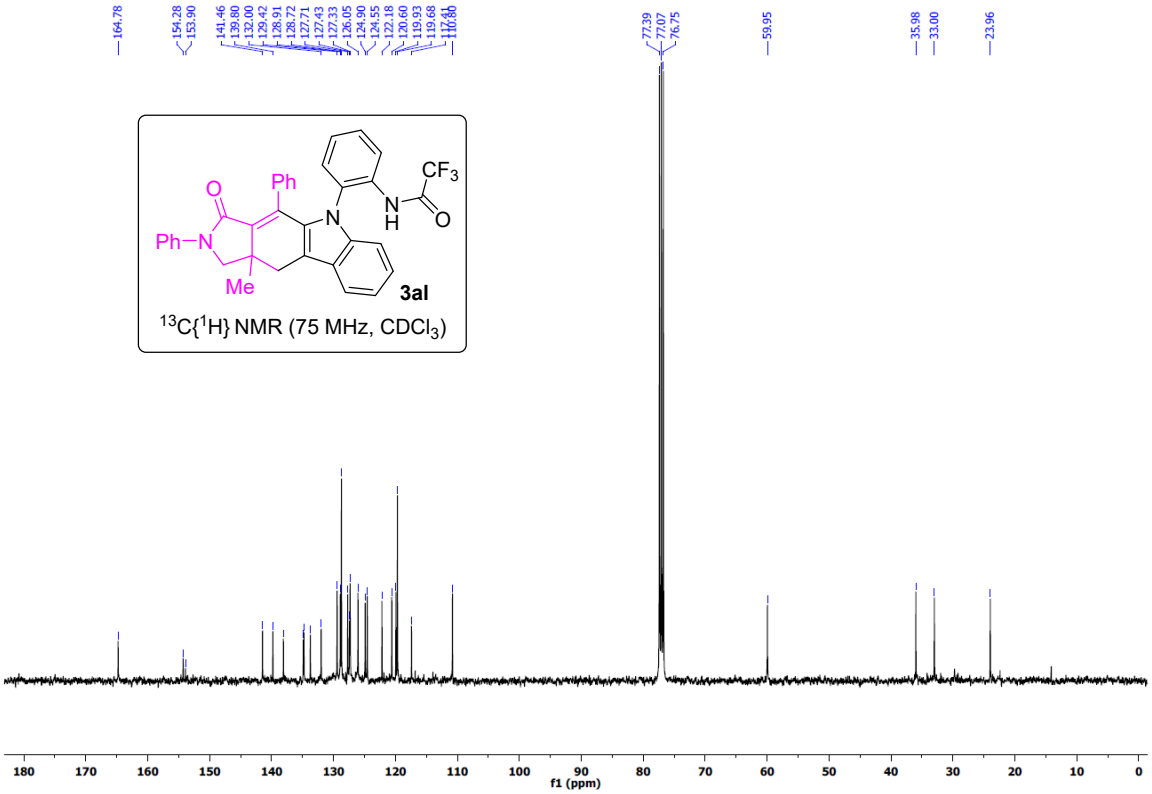
^{19}F NMR (376 MHz, $CDCl_3$)

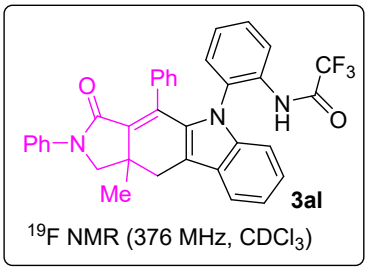

8.58
7.79
7.77
7.30
7.28
7.27
7.26
7.26
7.03
6.69
6.68
6.23
6.23
3.86
1.61
1.61
Ph
O
Ph
N
O
N
H
CF3
S
Me
3ak
1H NMR (300 MHz, CDCl3)
0.85
1.12
7.89
2.08
1.12
2.95
1.08
0.94
2.00
2.98
f1 (ppm)


163.15
155.71
155.23
151.75
132.26
130.58
129.85
128.90
128.60
128.49
128.43
127.89
127.36
127.27
126.34
126.09
125.94
113.65
77.49
77.06
76.64
56.06
20.18
Ph
O
Ph
N
O
N
H
CF3
S
Me
3ak
13C{1H} NMR (75 MHz, CDCl3)
f1 (ppm)


3al
^{1}H NMR (300 MHz, CDCl$_3$)
f1 (ppm)

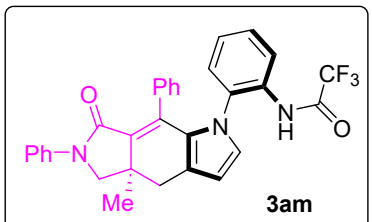

f1 (ppm)


3al
^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$)
f1 (ppm)



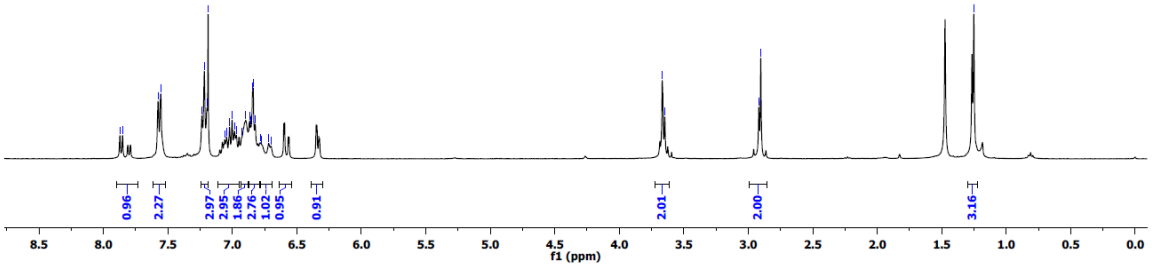
3al

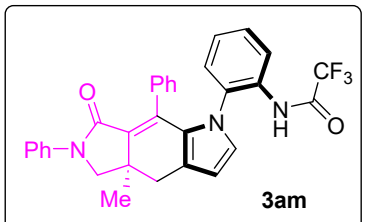
^{19}F NMR (376 MHz, $CDCl_3$)



3am

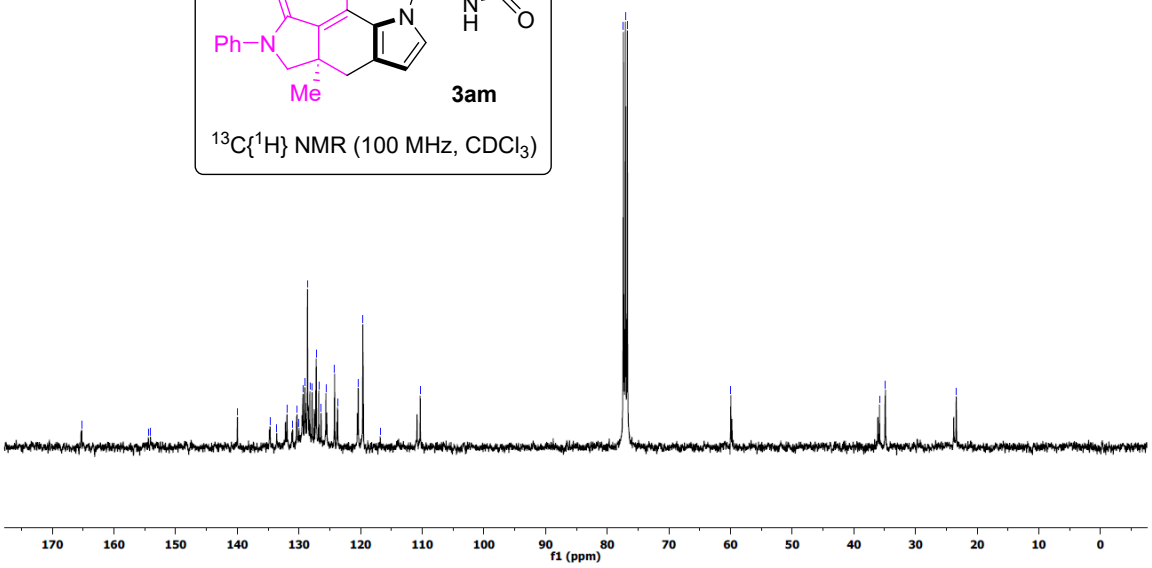
^{1}H NMR (400 MHz, $CDCl_3$)

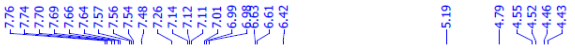


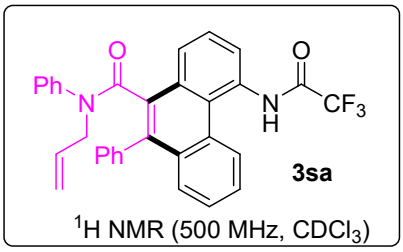


3am

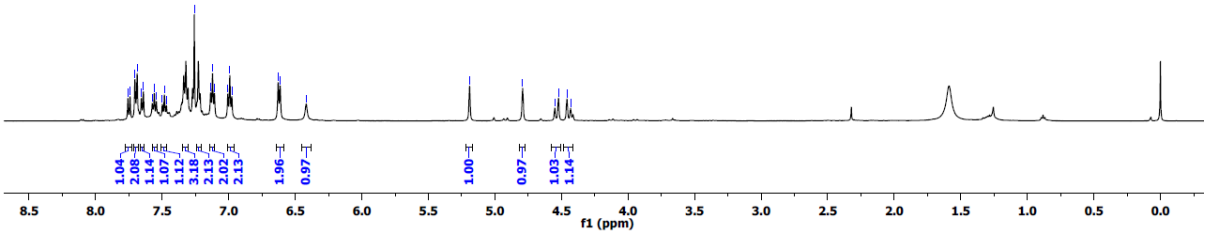
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

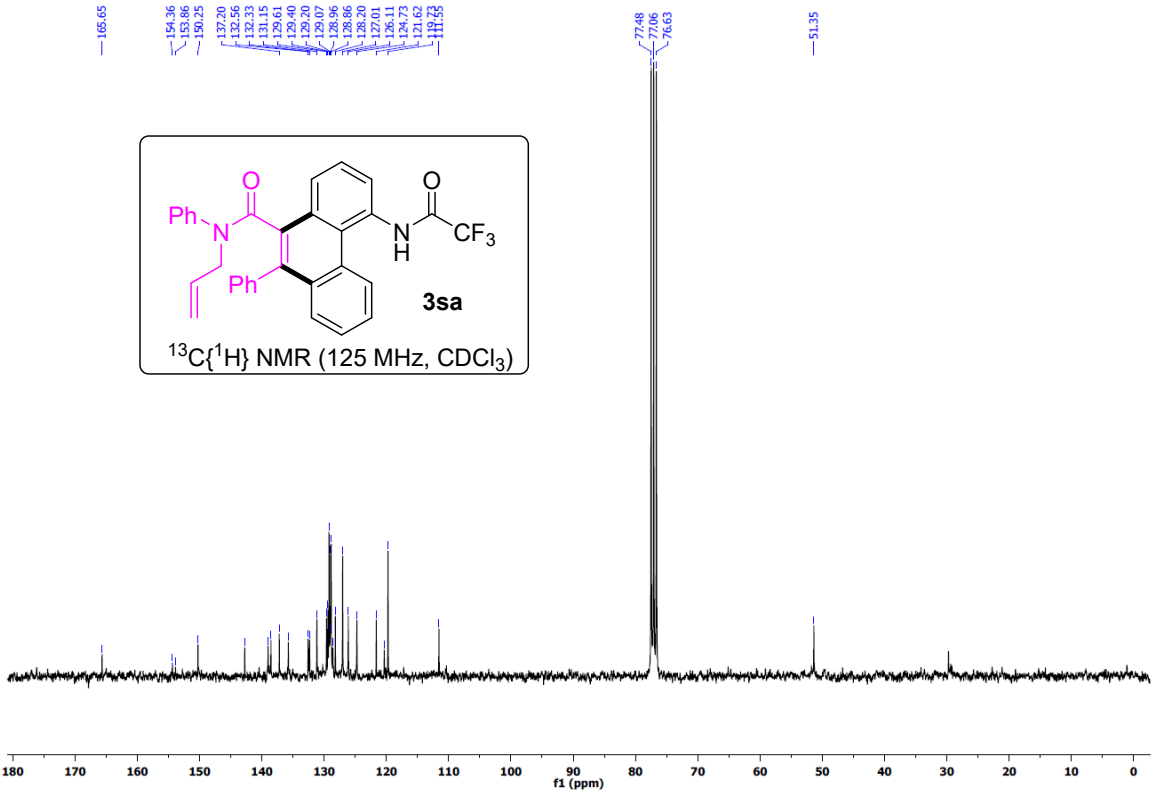




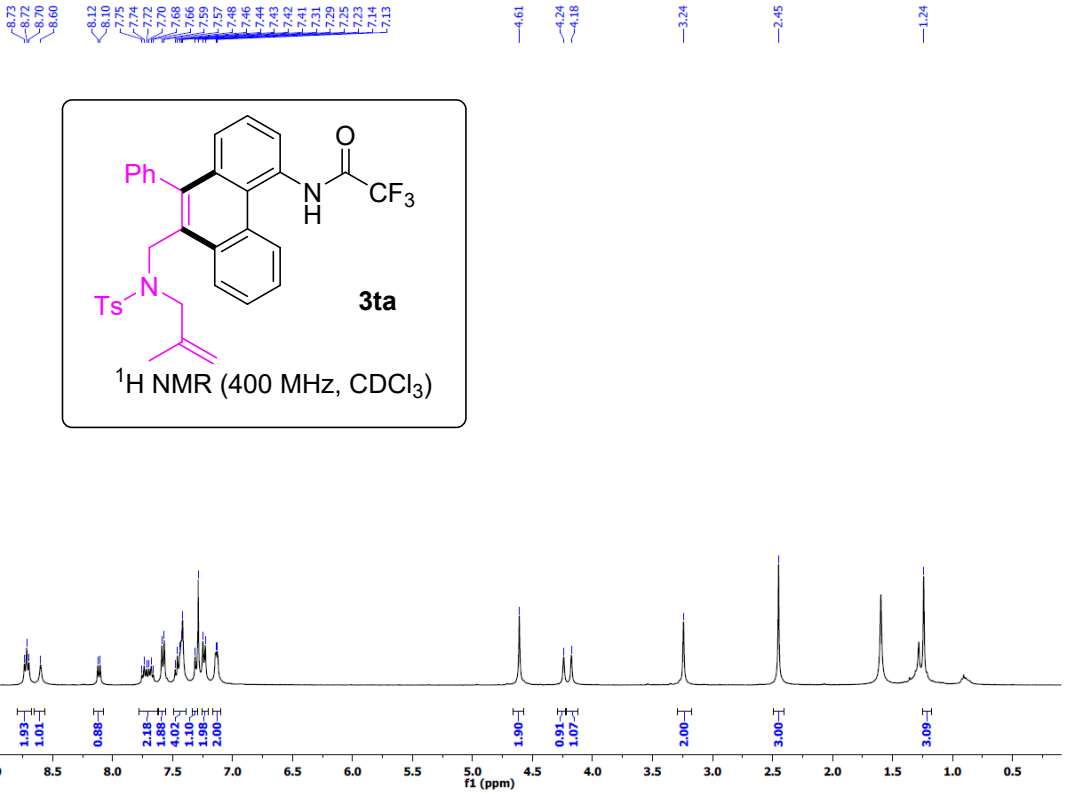


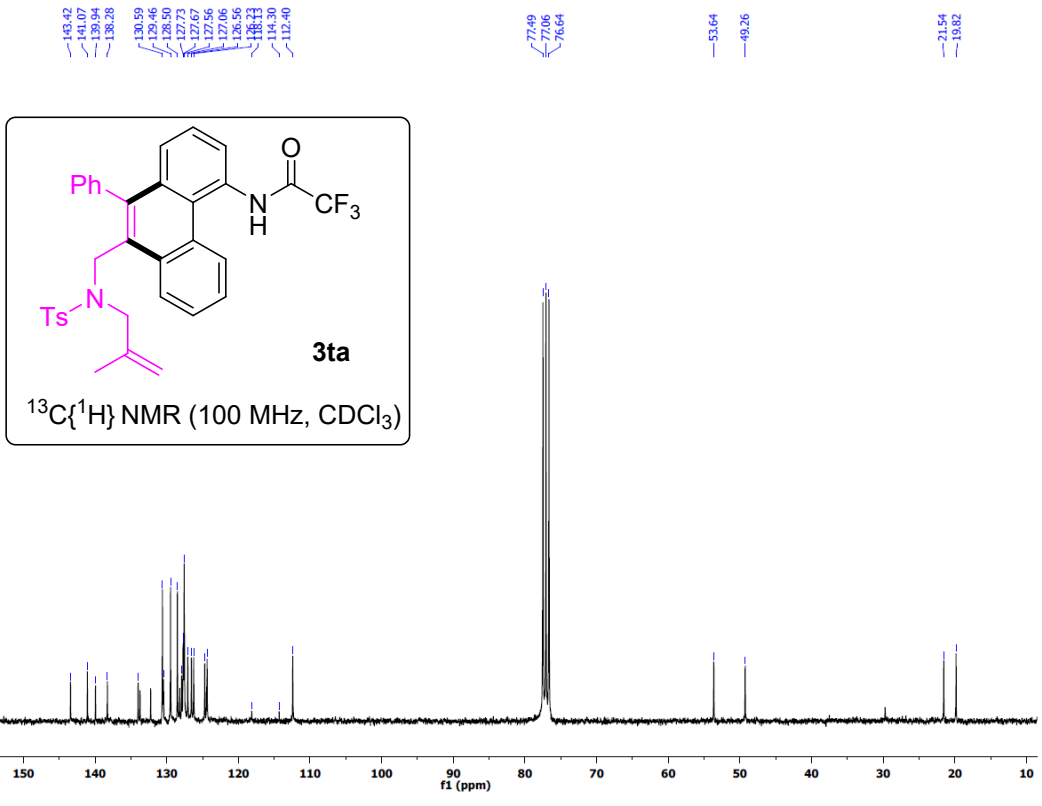
^{1}H NMR (500 MHz, $CDCl_3$)

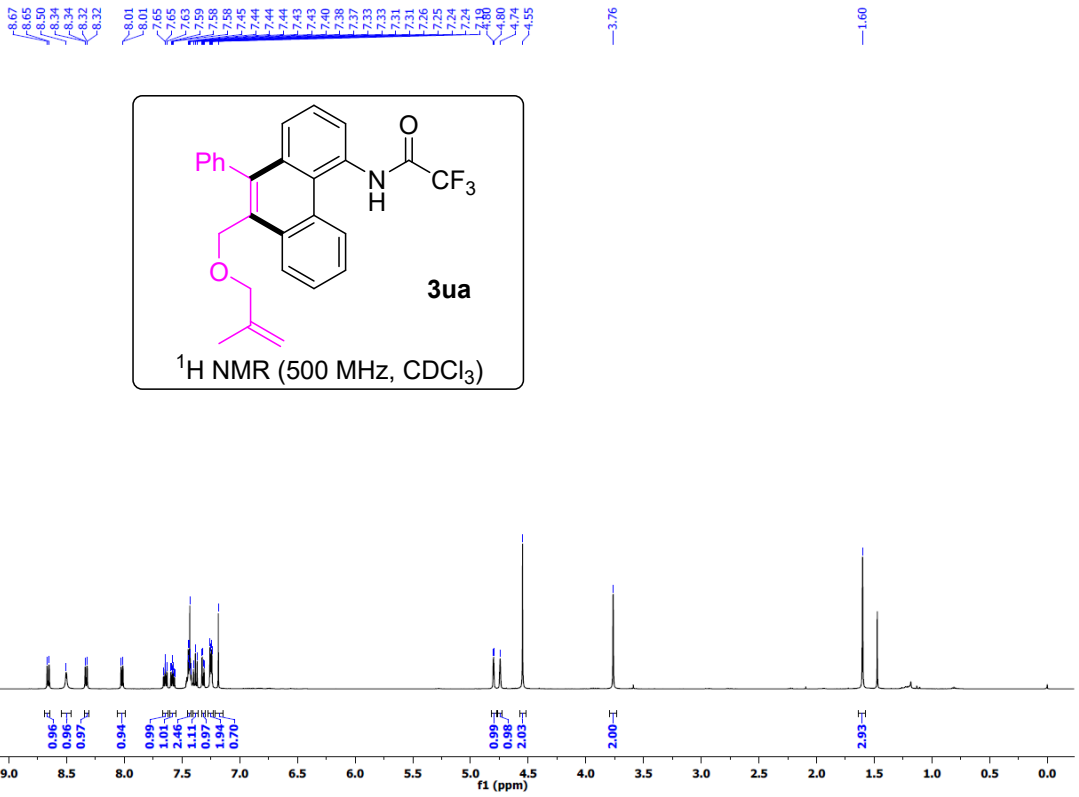


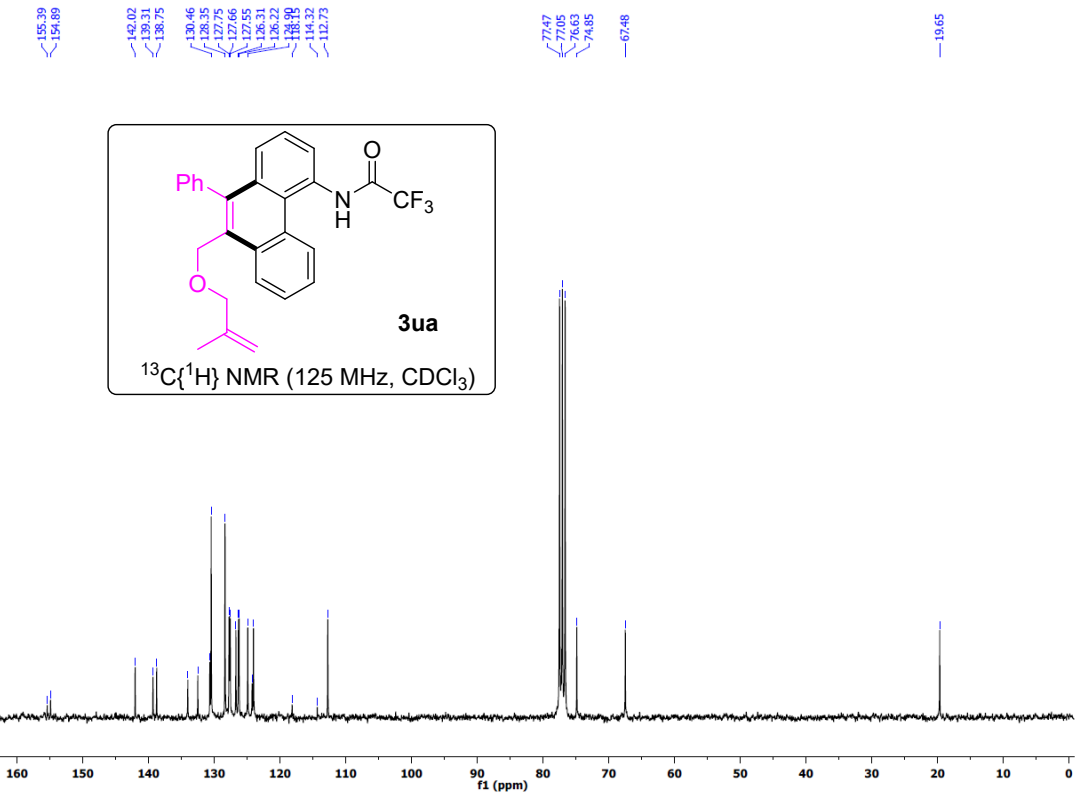


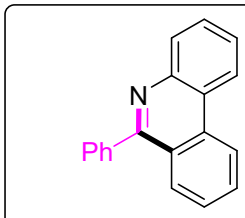
^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$)


Ph
N
H
O
CF3
Ts
N
3ta
1H NMR (400 MHz, CDCl3)
f1 (ppm)


Ph
N
H
O
CF3
Ts
N
3ta
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)

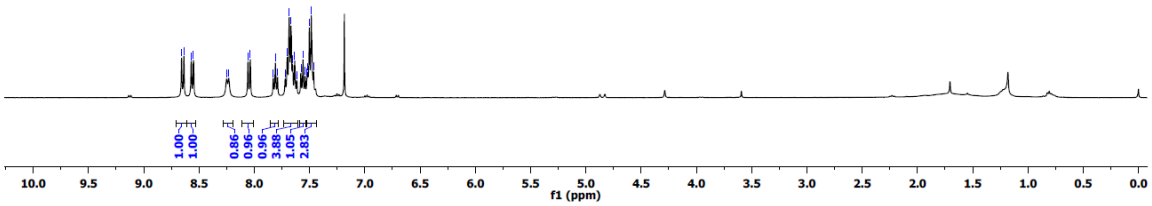

Ph
O
N
H
CF3
O
3ua
1H NMR (500 MHz, CDCl3)
f1 (ppm)

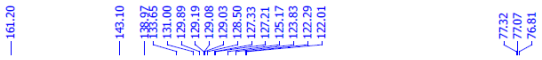

Ph
O
N
H
CF3
O
3ua
13C{1H} NMR (125 MHz, CDCl3)
f1 (ppm)

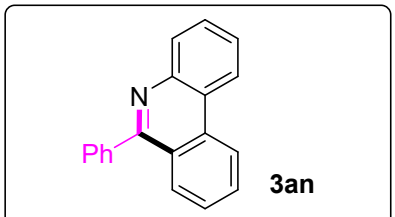


3an

^{1}H NMR (400 MHz, $CDCl_3$)

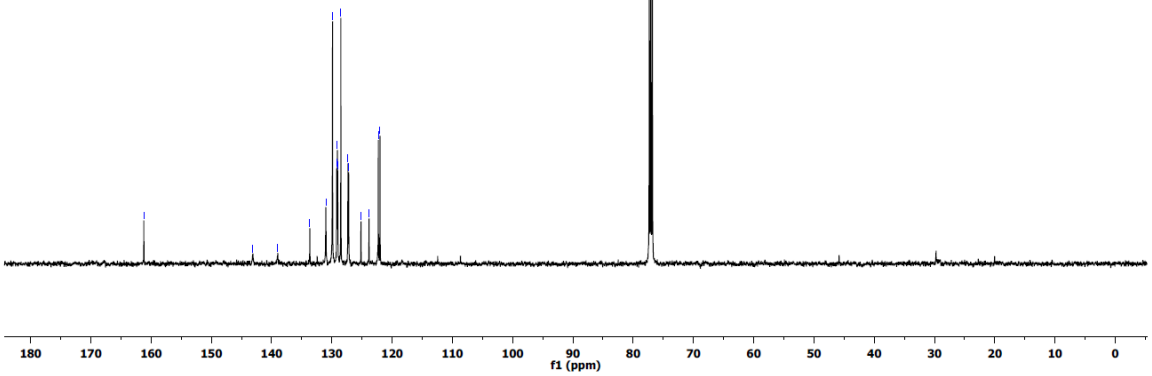


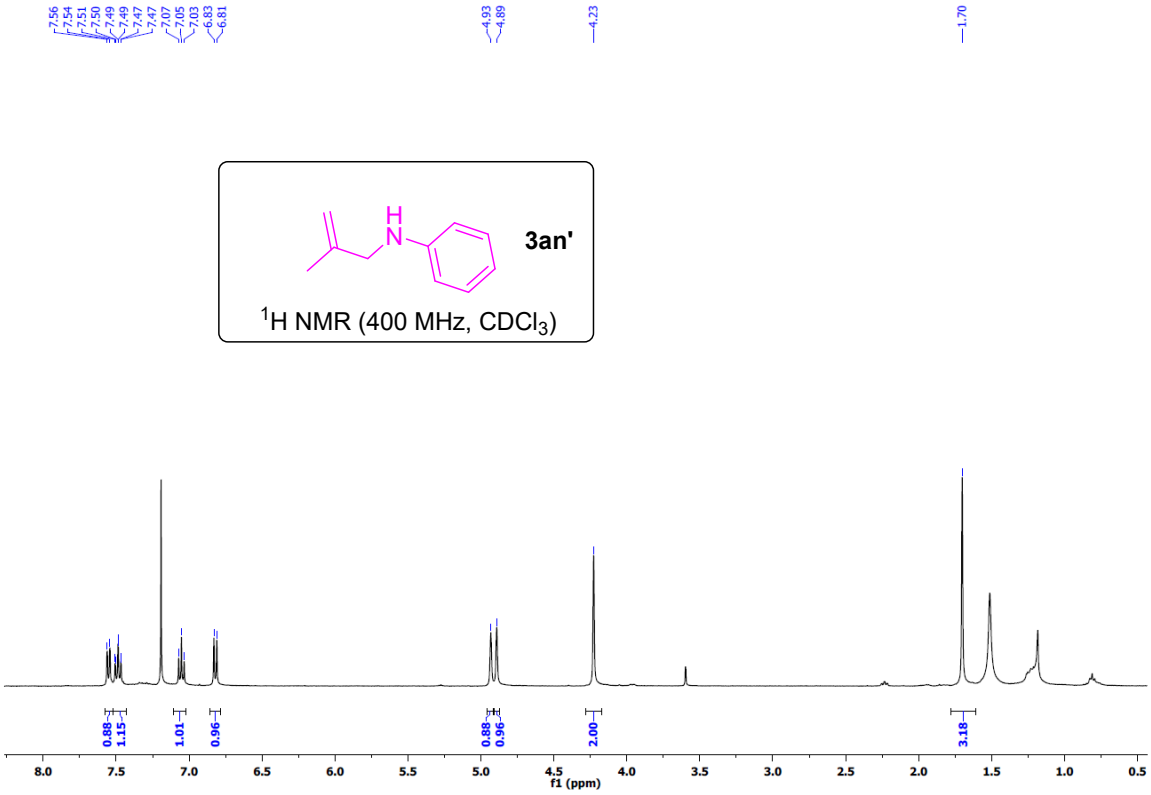


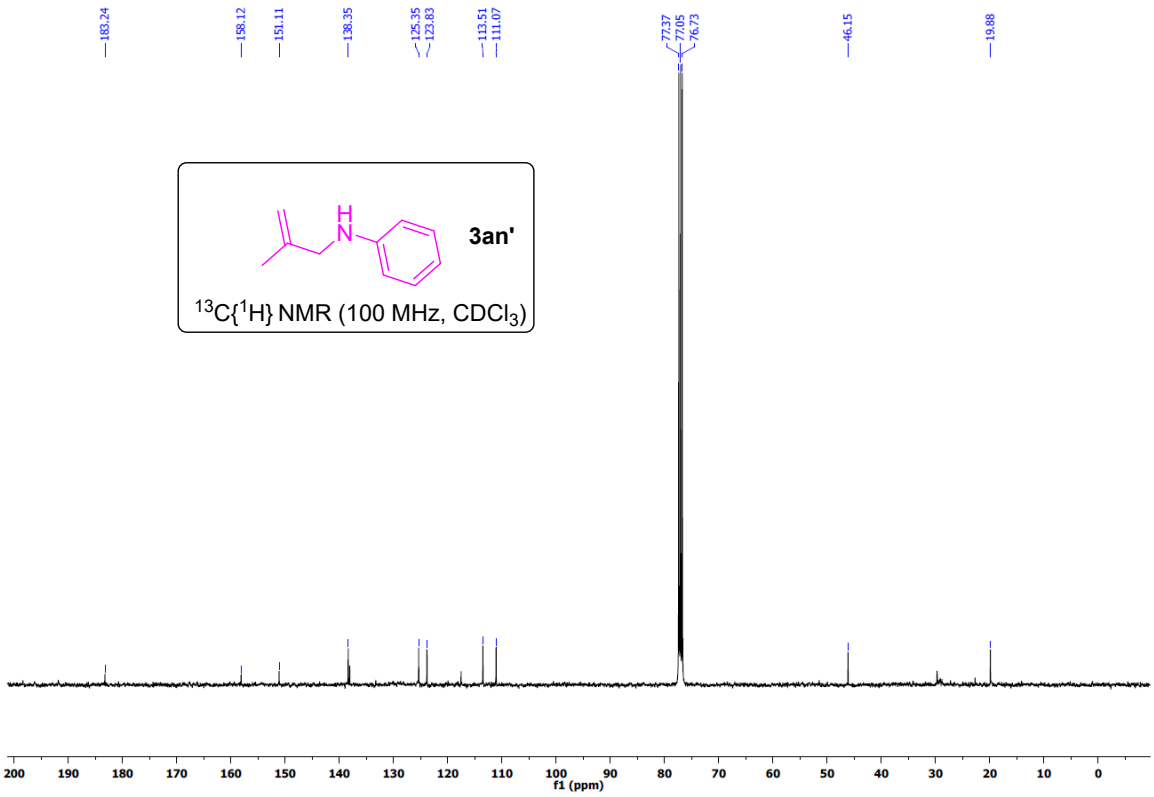


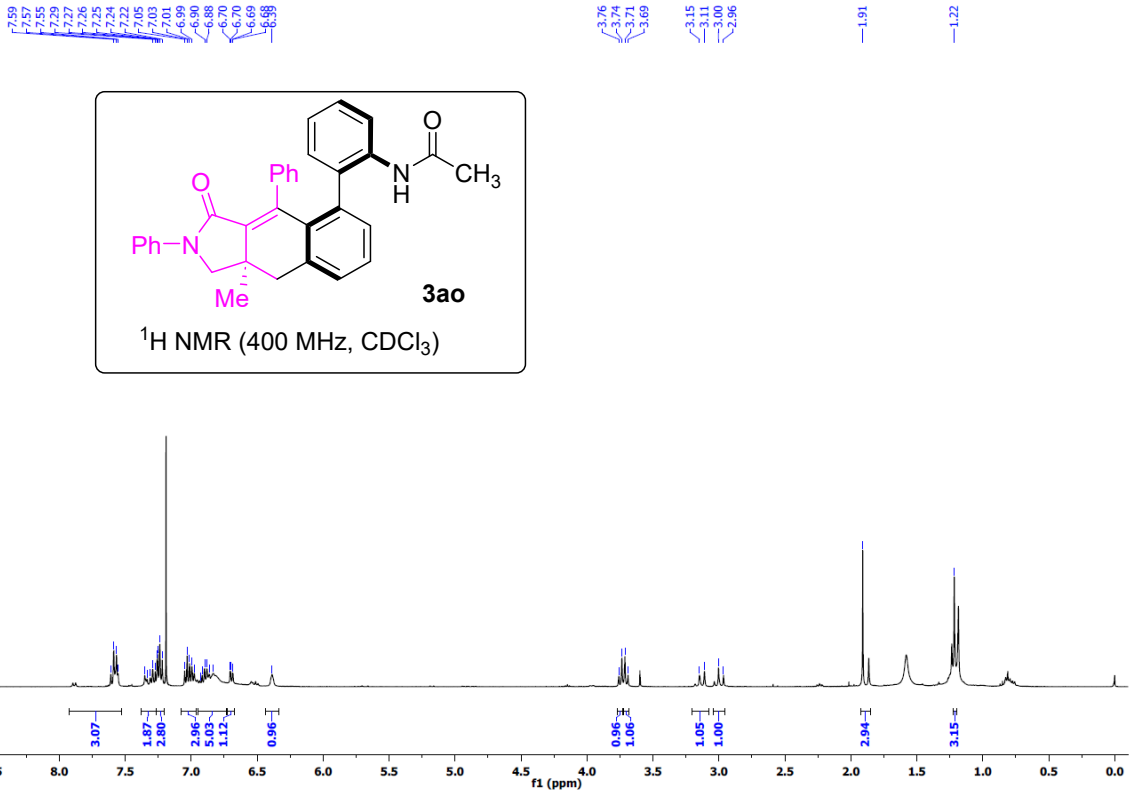
3an

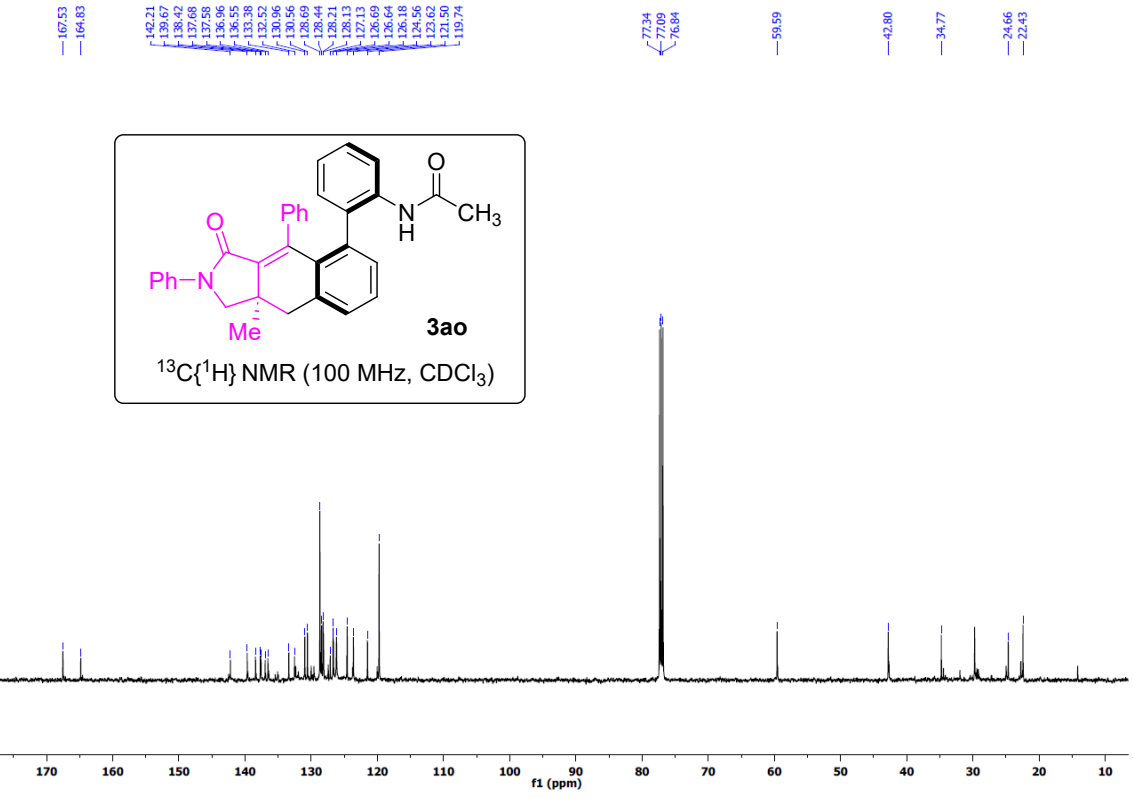
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$)

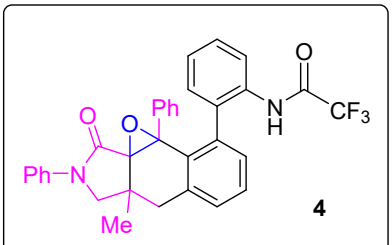



7.56
7.54
7.51
7.50
7.49
7.49
7.47
7.47
7.07
7.05
7.03
6.83
6.81
4.93
4.89
4.23
1.70
3an'
^{1}H NMR (400 MHz, $CDCl_3$)
0.88
1.15
1.01
0.96
0.88
0.96
2.00
3.18
f1 (ppm)


183.24
158.12
151.11
138.35
125.35
123.83
113.51
111.07
77.37
77.05
76.73
46.15
19.88
3an'
$^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$)
f1 (ppm)

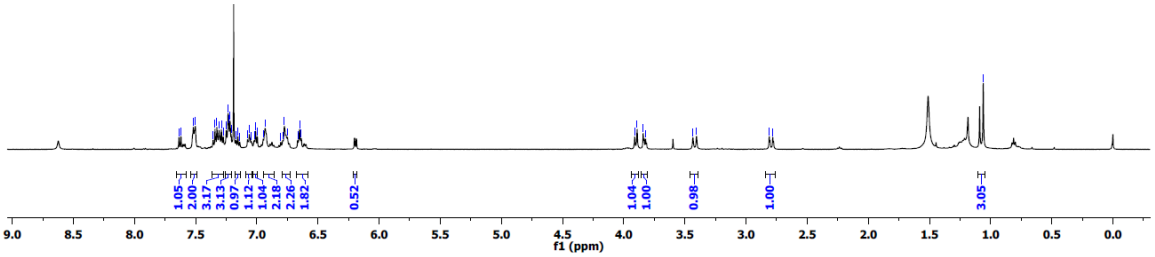

Ph
O
N
H
CH3
Ph–N
Me
3ao
1H NMR (400 MHz, CDCl3)
f1 (ppm)

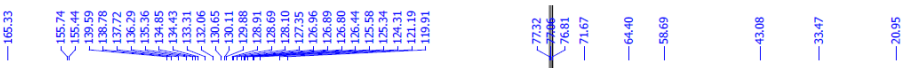

Ph
O
N
H
CH3
Ph–N
Me
3ao
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)

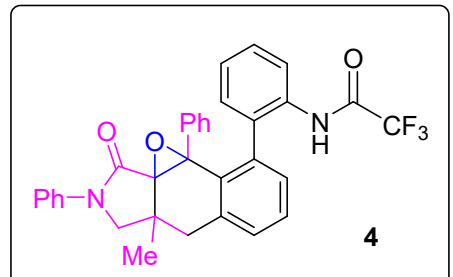


4

^{1}H NMR (500 MHz, $CDCl_3$)

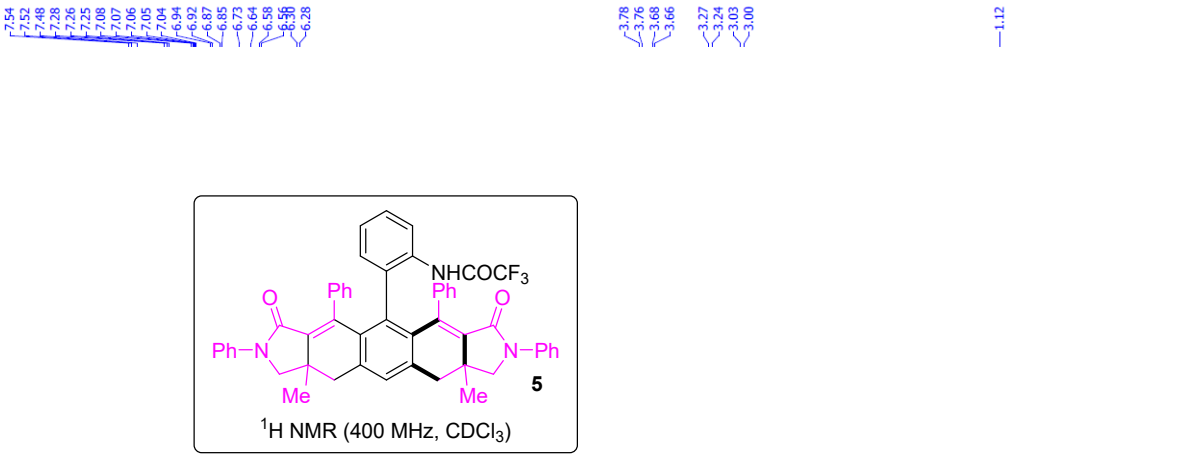


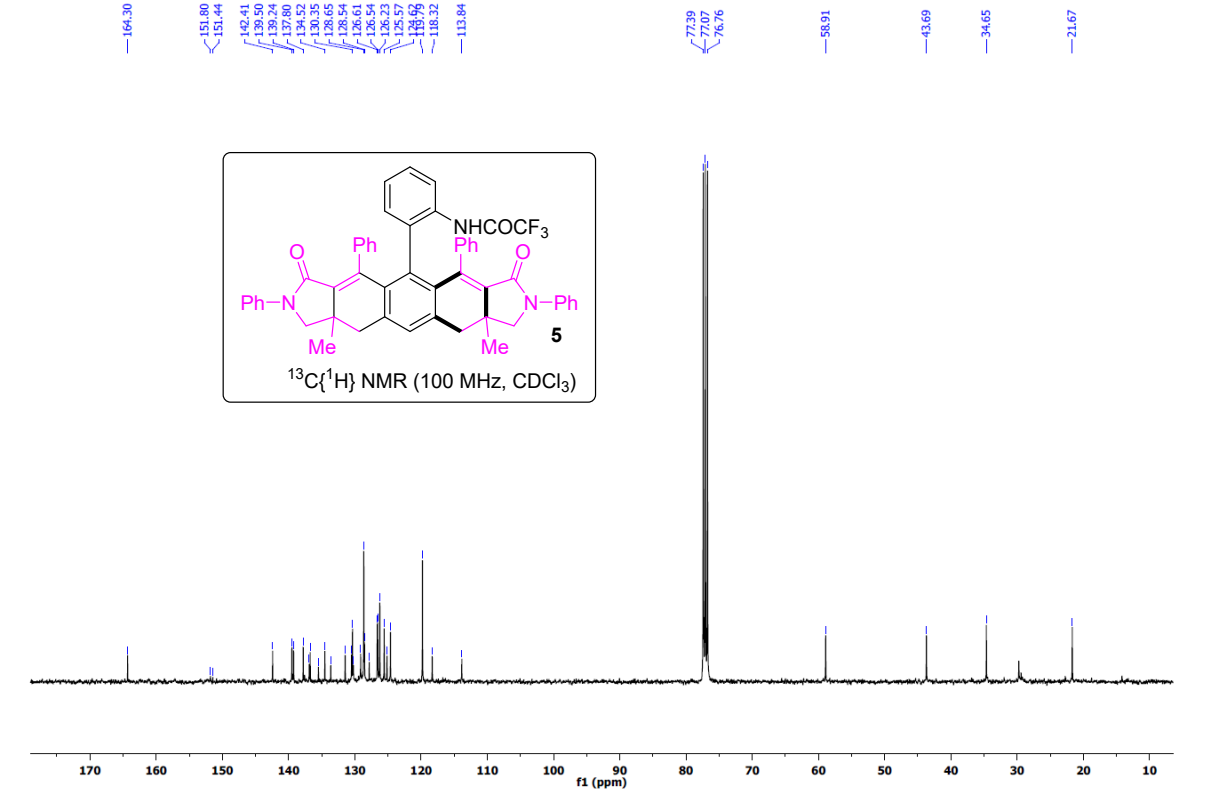


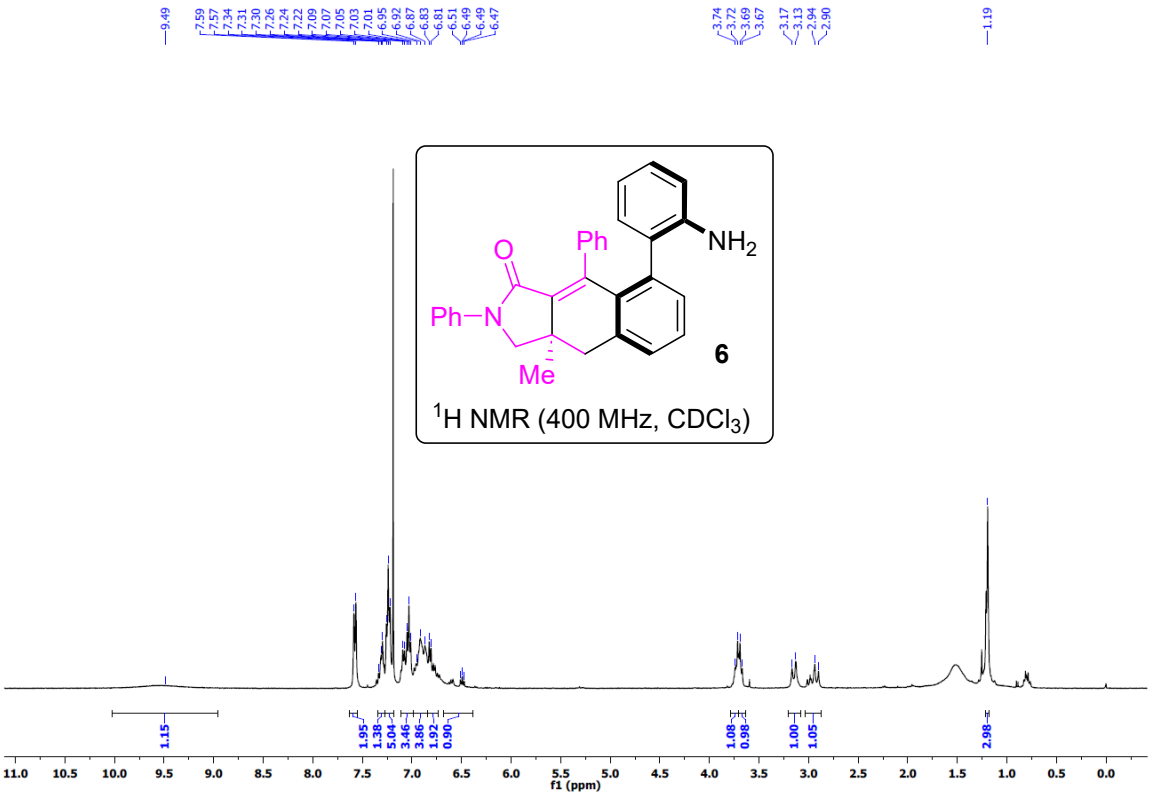


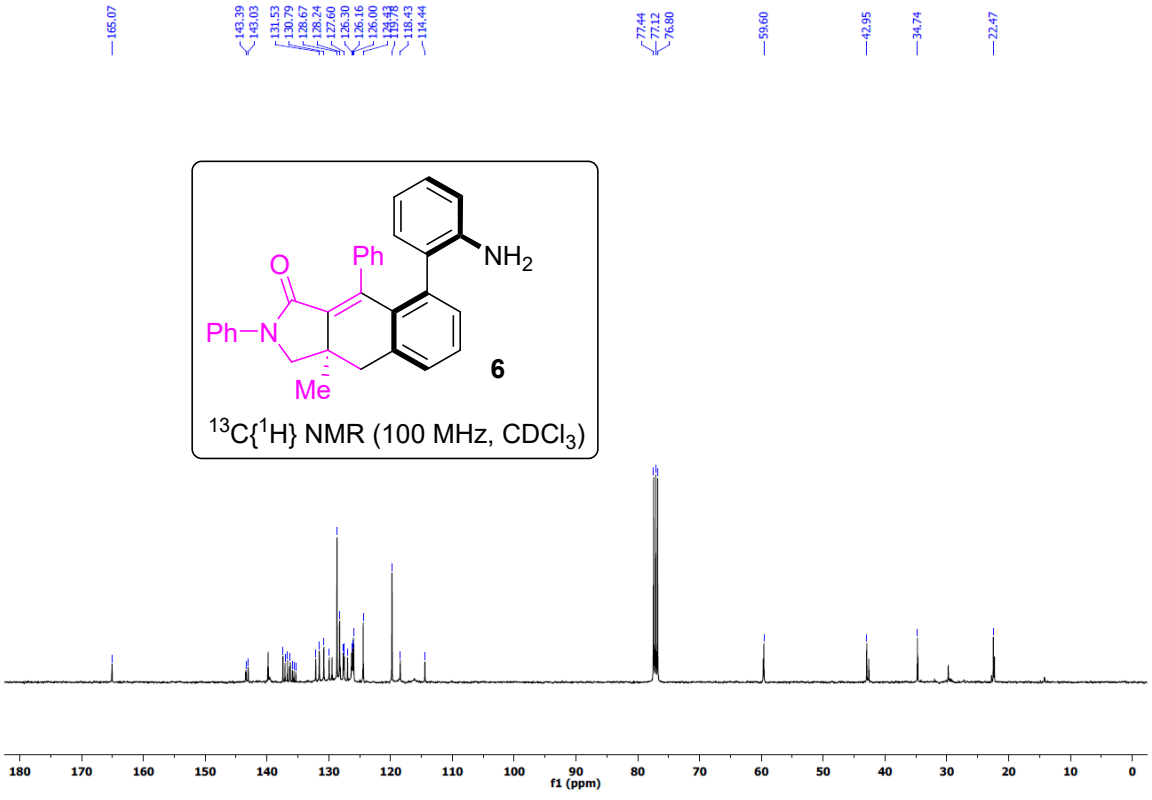
4

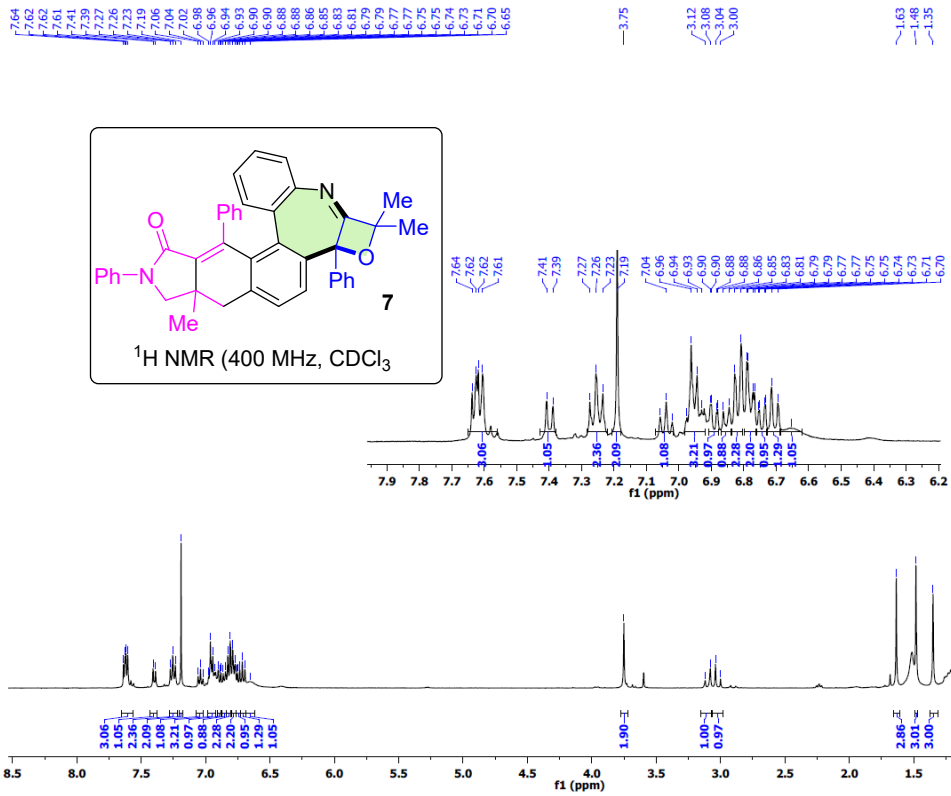
$^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$)


7.54
7.52
7.48
7.28
7.26
7.25
7.08
7.07
7.06
7.05
7.04
6.94
6.92
6.87
6.85
6.73
6.64
6.58
6.56
6.30
6.28
3.78
3.76
3.68
3.66
3.27
3.24
3.03
3.00
1.12
NHCOCF3
Ph
Ph
O
O
Ph–N
N–Ph
Me
Me
5
1H NMR (400 MHz, CDCl3)
4.13
1.01
4.30
4.05
2.08
3.13
1.06
1.05
2.08
1.98
1.91
2.08
2.04
1.99
2.00
6.02
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
f1 (ppm)

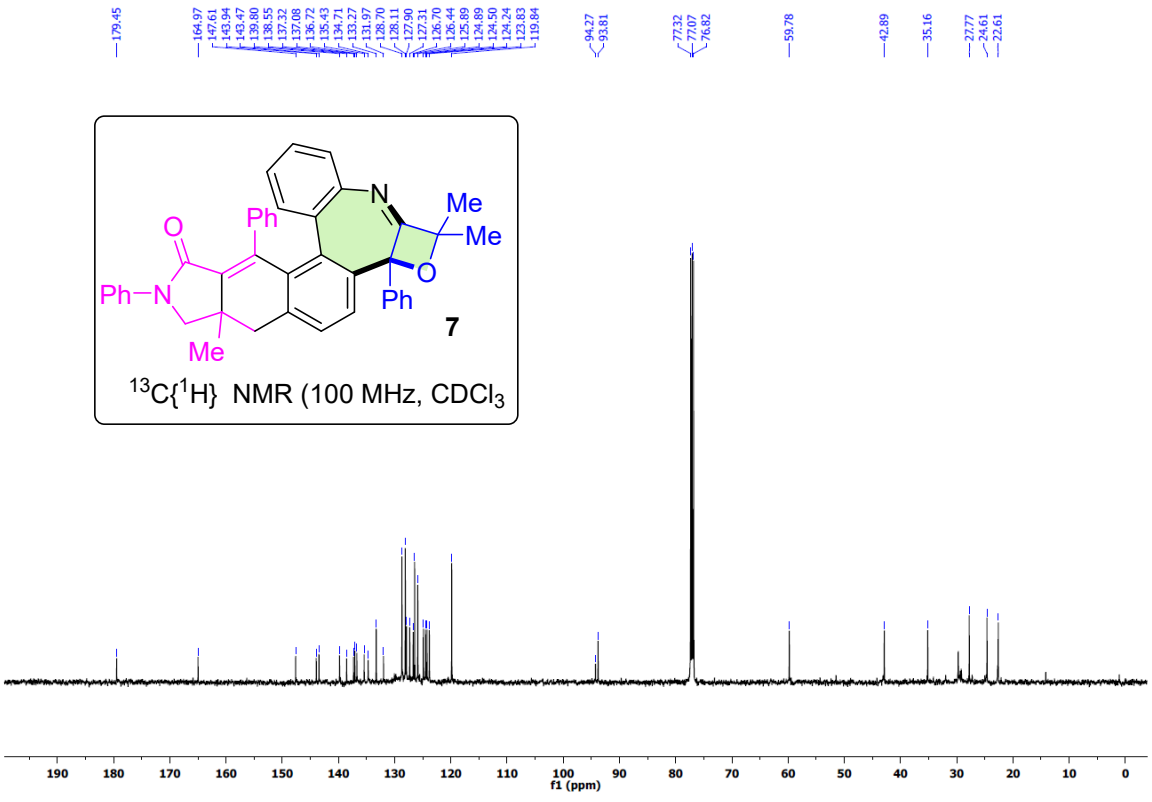

164.30
151.80
151.44
142.41
139.50
139.24
137.80
134.52
130.35
128.65
128.54
126.61
126.54
126.23
125.57
118.32
113.84
77.39
77.07
76.76
58.91
43.69
34.65
21.67
NHCOCF3
Ph
Ph
O
O
Ph–N
N–Ph
Me
Me
5
13C{1H} NMR (100 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


9.49
7.59
7.57
7.34
7.31
7.30
7.26
7.24
7.22
7.09
7.07
7.05
7.03
7.01
6.95
6.92
6.87
6.83
6.81
6.51
6.49
6.49
6.47
3.74
3.72
3.69
3.67
3.17
3.13
2.94
2.90
1.19
O
Ph
NH2
Ph—N
Me
6
1H NMR (400 MHz, CDCl3)
1.15
1.95
1.38
5.04
3.46
3.86
1.92
0.90
1.08
0.98
1.00
1.05
2.98
f1 (ppm)


165.07
143.39
143.03
131.53
130.79
128.67
128.24
127.60
126.30
126.16
126.00
118.43
114.44
77.44
77.12
76.80
59.60
42.95
34.74
22.47
O
Ph
NH2
Ph—N
Me
6
13C{1H} NMR (100 MHz, CDCl3)
f1 (ppm)


Ph
N
Me
Me
O
Ph–N
O
Ph
Me
7
1H NMR (400 MHz, CDCl3
f1 (ppm)

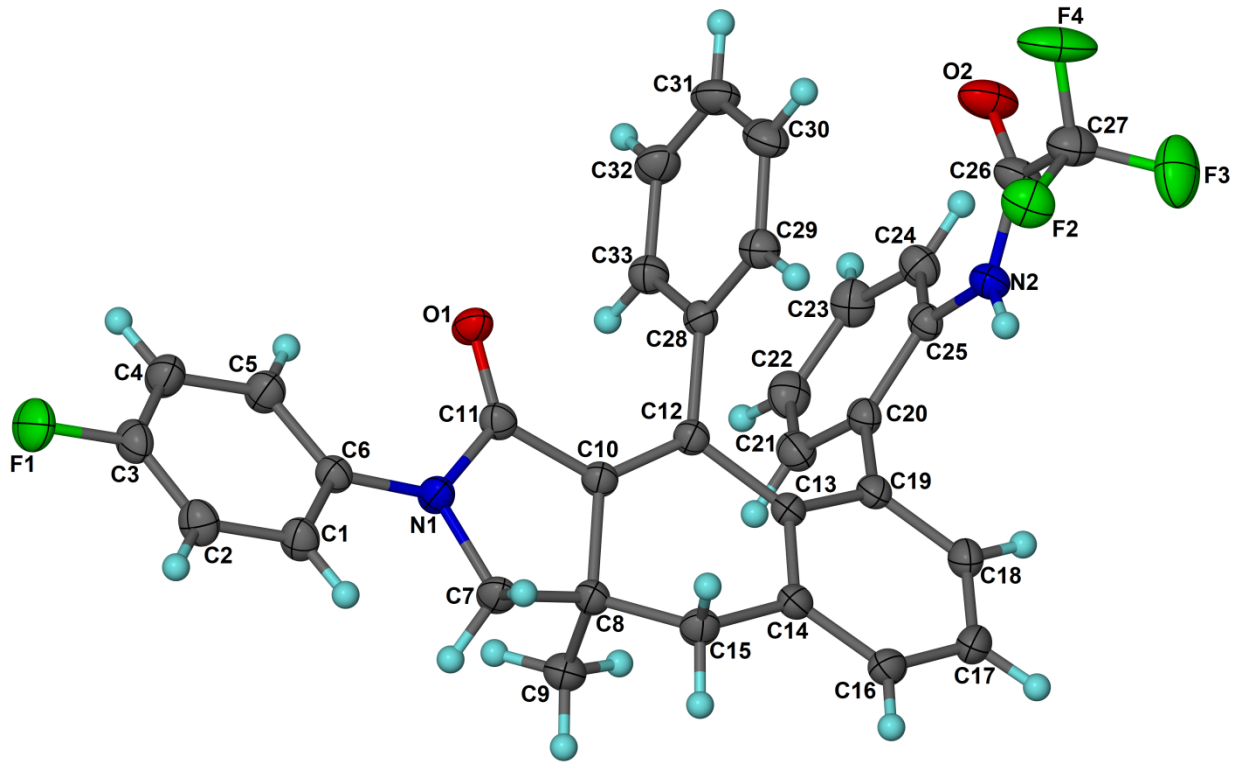
f1 (ppm)


Ph
N
Me
Me
O
Ph–N
O
Ph
Me
7
13C{1H} NMR (100 MHz, CDCl3
f1 (ppm)

8. X-ray crystallography data:

X-ray data for 3da:

CRYSTAL DATA - KB1371



Sample Preparation for Crystal Growth: The compound **3da** was dissolved in Acetone in beaker and kept for slow evaporation at room temperature. Formation of needle shape crystals was observed after five days. The single crystals were then subjected to X-ray diffraction analysis.

Figure caption: ORTEP of **KB1371** compound with the atom-numbering. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

Crystal Data for 3da: $C_{33}H_{24}F_4N_2O_2$, M = 556.54, Monoclinic, space group $P2_1/c$ (No. 14), a = 10.940(5) Å, b = 16.898(8) Å, c = 14.590(8) Å, α = 90º, β = 104.697(14)º, γ = 90º, V = 2609(2) Å^3, Z = 4, Dc = 1.417 g/cm^3, F_{000} = 1152, Bruker D8 QUEST PHOTON III C7 HPAD detector, Mo-Kα radiation, λ = 0.71073 Å, T = 294(2) K, $2\theta_{max}$ = 52º, μ = 0.108 mm^{-1}, 41526 reflections measured, 5950 unique (R_{int} = 0.0799), 412 parameters, $R1$ = 0.0514, $wR2$ = 0.1058, R indicated based on 2929 reflections with I > 2σ(I) (refinement on F^2), Final

GooF = 1.019, largest difference hole and peak = -0.199 and 0.171 e.Å^{-3}. **CCDC deposition number 2366769** contains the supplementary crystallographic data for this paper which can be obtained free of charge at https://www.ccdc.cam.ac.uk/structures/

Data collection and Structure solution details for 3da

X-ray data for the compound were collected at room temperature on a Bruker D8 QUEST instrument with an IμS Mo microsource (λ = 0.7107 Å) and a PHOTON-III C7 HPAD detector. The raw data frames were reduced and corrected for absorption effects using the Bruker Apex 3 software suite programs [1]. The structure was solved using intrinsic phasing method [2] and further refined with the SHELXL [2-4] program and expanded using Fourier techniques. Anisotropic displacement parameters were included for all non-hydrogen atoms. All C bound H atoms were positioned geometrically and treated as riding on their parent C atoms [C-H = 0.93-0.97 Å, and Uiso(H) = 1.5Ueq(C) for methyl H or 1.2Ueq(C) for other H atoms]. The fluorine atoms of $–CF_3$ group and O atom of C=O group were disordered over two sites, with site occupancy factor of 0.738(7) for the major component of the disordered atoms (F1/F2/F3 and O2) and 0.262(7) for the minor component of the disordered atoms (F1D/F2D/F3D and O2D). PART, FVAR, DELU, and SIMU instructions were utilized for modelling the structural disorder and structural refinement [4]. **CCDC deposition number 2366769** contains the supplementary crystallographic data for this paper which can be obtained free of charge at https://www.ccdc.cam.ac.uk/structures/

1. Bruker (2016). APEX3, SAINT and SADABS. Bruker AXS, Inc., Madison, Wisconsin, USA.
2. G. M. Sheldrick, ActaCrystallogr., 2015, C71: 3-8.
3. C. B. Hübschle, G. M. Sheldrick and B. Dittrich, ShelXle: aQt graphical user interface for SHELXL, *J. Appl. Cryst.*, 2011, 44, 1281-1284.
4. Muller, P, Herbst-Imer, R, Spek, A. L, Schneider, T. R, and Sawaya, M. R. Crystal Structure Refinement: A Crystallographer's Guide to SHELXL. Muller, P. Ed. 2006 Oxford University Press: Oxford, New York, pp. 57–91.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) KB1371_0m_a

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

No syntax errors found. CIF dictionary Interpreting this report

Datablock: KB1371_0m_a

```
Bond precision:   C-C = 0.0033 A          Wavelength=0.71073

Cell:             a=10.940(5)     b=16.898(8)          c=14.590(8)
                  alpha=90        beta=104.697(14)     gamma=90
Temperature:      294 K

                  Calculated                Reported
Volume            2609(2)                   2609(2)
Space group       P 21/c                    P 21/c
Hall group        -P 2ybc                   -P 2ybc
Moiety formula    C33 H24 F4 N2 O2          C33 H24 F4 N2 O2
Sum formula       C33 H24 F4 N2 O2          C33 H24 F4 N2 O2
Mr                556.54                    556.54
Dx,g cm-3         1.417                     1.417
Z                 4                         4
Mu (mm-1)         0.108                     0.108
F000              1152.0                    1152.0
F000'             1152.67
h,k,lmax          14,21,18                  14,21,18
Nref              5969                      5950
Tmin,Tmax         0.977,0.983               0.659,0.746
Tmin'             0.977

Correction method= # Reported T Limits: Tmin=0.659 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 27.498

R(reflections)= 0.0514( 2929)                wR2(reflections)=
                                             0.1412( 5950)
S = 1.004                 Npar= 412
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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

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PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C1       -C6       .      1.37 Ang.
PLAT767_ALERT_4_C INS Embedded LIST 6 Instruction Should be LIST 4    Please Check
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......     6.072 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600       15 Report
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                  -1  6  1,    0 10  1,   -1 11  1,   -1  3  2,   -2  0  4,    1  0  4,
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Alert level G

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PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite         2 Note
PLAT003_ALERT_2_G Number of Uiso or U(i,j) Restrained non-H Atoms         10 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records         1 Report
PLAT177_ALERT_4_G The CIF-Embedded .res File Contains DELU Records         1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records         1 Report
PLAT188_ALERT_3_G A Non-default SIMU Restraint Value has been used    0.0100 Report
PLAT242_ALERT_2_G Low     'MainMol' Ueq as Compared to Neighbors of      C27 Check
PLAT301_ALERT_3_G Main Residue  Disorder ..............(Resd    1)       10% Note
PLAT793_ALERT_4_G Model has Chirality at C8          (Centro SpGr)        R Verify
PLAT860_ALERT_3_G Number of Least-Squares Restraints .............      120 Note
PLAT883_ALERT_1_G No Info/Value for _atom_sites_solution_primary .    Please Do !
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).         4 Note
                   1  0  0,    1  1  0,    0  2  0,    0  1  1,
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         1 Note
PLAT913_ALERT_3_G Missing # of Very Strong Reflections in FCF ....         1 Note
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PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File        11 Note
                   0  4  0,    0  5  1,   -1  5  1,   -1  2  1,    0  2  1,   -2  0  4,
                  -1  3  2,    1  0  0,    0  2  0,    1  0  4,    1  1  0,
PLAT967_ALERT_5_G Note: Two-Theta Cutoff Value in Embedded .res ..      55.0 Degree
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........     3.285 Note
                  Predicted wR2: Based on SigI**2  4.30 or SHELX Weight 14.06
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         3 Info
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   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
  18 ALERT level G = General information/check it is not something unexpected

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

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

Publication of your CIF in IUCr journals

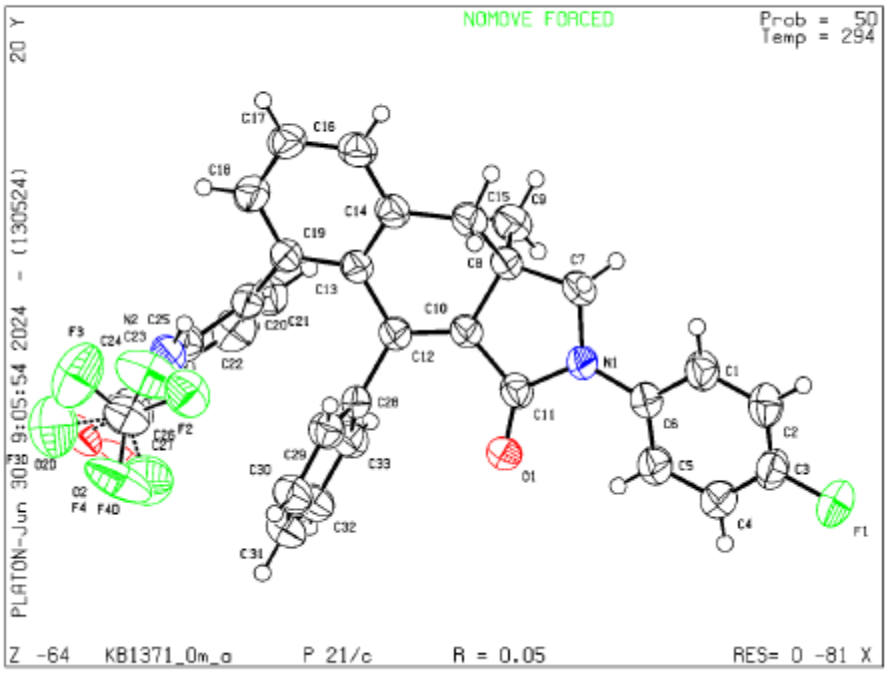
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section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

Publication of your CIF in other journals

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

PLATON version of 13/05/2024; check.def file version of 04/05/2024

Datablock KB1371_0m_a - ellipsoid plot



9. References:

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