Supplementary Information

Ortho-Methylation of Pyridine *via* Intramolecular *N*-methyl Migration

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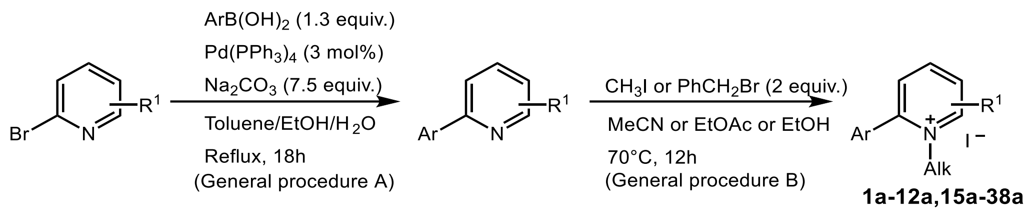
1. General Information

^{1}H NMR spectra were recorded on Bruker 400 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. The peak patterns were indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, *J*, were reported in Hertz (Hz). ^{13}C NMR spectra were obtained at Bruker 101 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in $CDCl_3$). ^{19}F NMR were obtained at Bruker 376 MHz ($CFCl_3$ as outside standard and low field is positive). GC-MS analysis was recorded on GCMS-QP2020 of SHIMADZU. High-resolution mass spectra (HRMS) were obtained on an Agilent 6520 Q-TOF LC/MS. X-ray photoelectron spectroscopy (XPS) were obtained at AXIS Supra (Kratos). All reagents were weighed and handled in air at room temperature. All solvents, reagents, and deuterated solvents were purchased from Aladdin, Aldrich, Adamas, and TCI without further purification.

2. Experimental Procedure

2.1 General procedure for synthesis of *N*-alkylpyridinium salts 1a-12a, 15a-38a



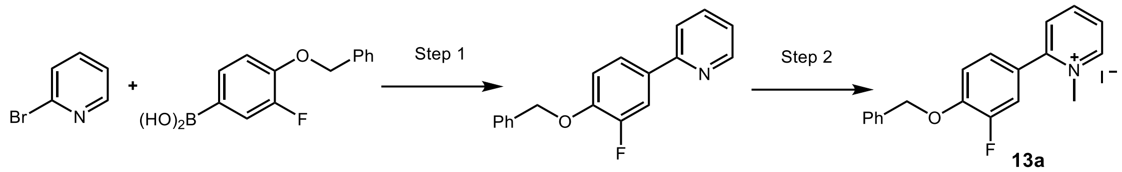
1a-12a,15a-38a

General Procedure A: To an oven-dried 100 mL flask, equipped with a magnetic stirrer, was charged with 2-bromopyridine (0.95 mL, 10.0 mmol), substituted phenylboronic acid (13.0 mmol, 1.3 equiv.), Pd $(PPh_3)_4$ (0.35 g, 0.3 mmol, 3 mol%), and Na_2CO_3 (7.95 g, 75.0 mmol, 7.5 equiv.). The system was then purged with nitrogen through three cycles of evacuation and backfilling using a balloon. Subsequently, toluene (35.0 mL), ethanol (7.5 mL), and water (35.0 mL) were added to the flask. The reaction mixture was heated at 110 °C for 18 hours. Upon completion of the reaction, the mixture was allowed to cool, and added aqueous NH_4Cl (30 mL), followed by filtration and extraction with ethyl acetate (3 x 30 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate, 20:1 to 5:1), to yield the desired 2-arylpyridine product[1].

General Procedure B: To an oven-dried 25 mL flask, equipped with a magnetic stirrer, was charged with 2-arylpyridine (5.0 mmol), methyl iodide or benzyl bromide (10.0 mmol, 2 equiv.), and acetonitrile or ethyl acetate or ethyl alcohol (5.0 mL). The reaction mixture was heated at 70 °C for 12 hours. Then it was cooled to room temperature, and the solvent was removed under reduced pressure to obtain the crude product, which was recrystallized in MeCN/EtOAc co-solvent to afford a pure product[2].

2.2 Synthesis of *N*-alkylpyridinium salts 13a - 14a, 39a - 43a

Synthesis of compound 13a

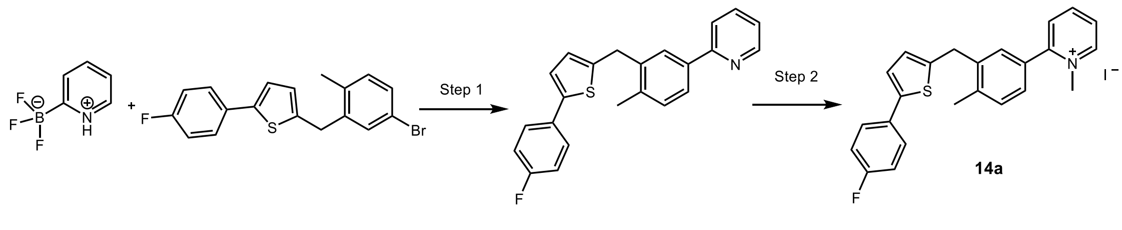


13a

Step 1: According to **General procedure A,** to an oven-dried 100 mL flask with a magnetic stirring bar was charged with 2-brompiridine (0.31 mL, 3.2 mmol, 1.0 equiv.), (4-(benzyloxy)-3-fluorophenyl) boronic acid (1.00 g, 4.1 mmol, 1.3 equiv.), Pd $(PPh_3)_4$ (0.19 g, 0.16 mmol, 5 mol%) and Na_2CO_3 (2.50 g, 24.0 mmol, 7.5 equiv.), and then subjected to three cycles of vacuum/nitrogen backfill. Subsequently, toluene (12.0 mL), ethanol (2.5 mL), and water (12.0 mL) were added to the flask, and the mixture was heated at 110 °C for 18 hours. Upon completion of the reaction, the mixture was allowed to cool, and added aqueous NH_4Cl (15 mL), followed by filtration and extraction with ethyl acetate (3 x 15 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography[1]. (589 mg, 66% yield)

Step 2: The crude product obtained from **step 1** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **13a**.

Synthesis of compound 14a



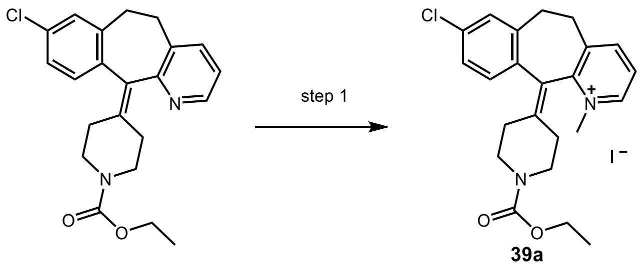
14a

Step 1: To an oven-dried 100 mL flask, equipped with a magnetic stirrer, was charged with Pd(Amphos)Cl_2 (0.20 g, 7.5 mol%) and ZnO (0.62 g, 7.6 mmol, 2.0 equiv.). Subsequently, a mixture of 2-pyridylboronic acid (0.93 g, 7.6 mmol, 2.0 equiv.) and HBF_4·OEt_2 (1.00 mL, 7.6 mmol, 2.0 equiv.) in MeOH (19 mL, c = 0.4 M) was added into the flask, followed by 2-(5-bromo-2-methylbenzyl)-5 (4-fluorophenyl)thiophene (1.37 g, 3.8 mmol, 1.0 equiv.). The reaction mixture was stirred vigorously at 70 °C for

3 h in an oil bath. After that, the reaction vessel was opened to air, and the residue was purified by silica gel column chromatography[3]. (482 mg, 35% yield)

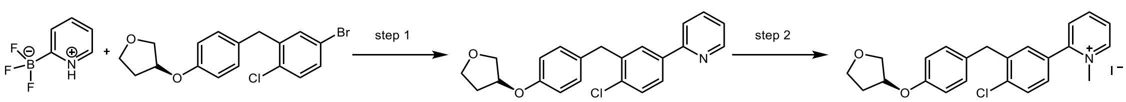
Step 2: The crude product obtained from **step 1** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **14a**.

Synthesis of compound 39a



According to **General procedure B,** loratadine (1.91 g, 5.0 mmol) and methyliodide (0.62 mL, 10.0 mmol, 2.0 equiv.) were reacted in ethyl alcohol (5.0 mL) at 70 °C for 12 hours. The mixture was allowed to cool to room temperature, and the solvent was removed under reduced pressure to obtain the crude product, which was recrystallized in MeCN/EtOAc co-solvent to afford a pure product **39a**.

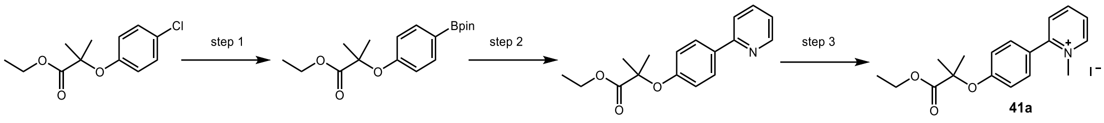
Synthesis of compound 40a



Step 1: To an oven-dried 100 mL flask, equipped with a magnetic stirrer, was charged with Pd(Amphos)Cl_2 (0.27 g, 7.5 mol%) and ZnO (1.23 g, 10.0 mmol, 2.0 equiv.). Subsequently, a mixture of 2-pyridylboronic acid (1.23 g, 10.0 mmol, 2.0 equiv.) and HBF_4·OEt_2 (1.36 mL, 10.0 mmol, 2.0 equiv.) in MeOH (25 mL, c = 0.4 M) was added into the flask, followed by (1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 3-bromo-benzoate (1.84 g, 5.0 mmol, 1.0 equiv.). The reaction mixture was stirred vigorously at 70 °C for 3 h in an oil bath. After that, the reaction vessel was opened to air, and the residue was purified by silica gel column chromatography[3]. (1.23 g, 68% yield)

Step 2: The crude product obtained from **step 1** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **40a**.

Synthesis of compound 41a[5]

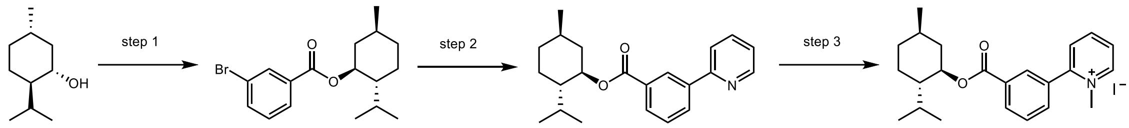


Step 1: To an oven-dried 100 mL flask with a magnetic stirring bar was charged with clofibrate (2.40 g, 10.0 mmol, 1.0 equiv.), Pd_2dba_3 (0.092 g, 1 mol%), Xphos (0.19 g, 4 mol%), B_2Pin_2 (2.92 g, 11.5 mmol, 1.15 equiv.) and KOAc (2.90 g, 30.0 mmol, 3.0 equiv.) and then subjected to three cycles of vacuum/nitrogen backfill. Subsequently, dry dioxane (47 mL) was added to the flask, and the mixture was heated at 110 °C for 12 hours. Upon completion of the reaction, the mixture was allowed to cool, and added brine (30 mL), followed by filtration and extraction with ethyl acetate (3 x 15 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. (3.13 g, 94% yield)

Step 2: To an oven-dried 100 mL flask with a magnetic stirring bar was charged with pinacol borate (3.13 g, 9.4 mmol, 1.0 equiv.), $Pd(PPh_3)_4$ (0.33 g, 3 mol%), 2-brompiridine (1.10 mL, 11.3 mmol, 1.2 equiv.) and Na_2CO_3 (3.00 g, 28.0 mmol, 3.0 equiv.) and then subjected to three cycles of vacuum/nitrogen backfill. Subsequently, toluene (50 mL) and water (50 mL) were added to the flask, and the mixture was heated at 110 °C for 12 hours. Upon completion of the reaction, the mixture was allowed to cool, and added aqueous NH_4Cl (30 mL), followed by filtration and extraction with ethyl acetate (3 x 30 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. (1.25 g, 47% yield)

Step 3: The crude product obtained from **step 2** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **41a**.

Synthesis of compound 42a



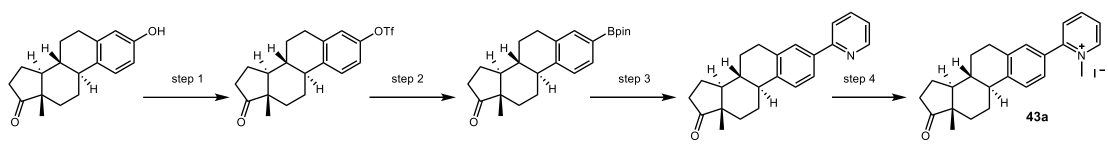
42a

Step 1: To an oven-dried 50 mL flask, equipped with a magnetic stirrer, was charged with 3-bromobenzoic acid (2.00 g, 10.0 mmol, 1.0 equiv.) and (+)-menthol (1.56 g, 10.0 mmol, 1.0 equiv.) in CH_2Cl_2 (0.25 M). Subsequently, DCC (2.06 g, 10.0 mmol, 1.0 equiv.), and DMAP (1.22 g, 10.0 mmol, 1.0 equiv.) were added to the flask. The reaction mixture was stirred vigorously at room temperature for 12h. After that, the solution was concentrated under reduced pressure, and the residue was purified by silica gel column chromatography[4]. (2.80 g, 83% yield)

Step 2: To an oven-dried 100 mL flask, equipped with a magnetic stirrer, was charged with Pd(Amphos)Cl_2 (0.27 g, 7.5 mol%) and ZnO (1.23 g, 10.0 mmol, 2.0 equiv.). Subsequently, a mixture of 2-pyridylboronic acid (1.23 g, 10.0 mmol, 2.0 equiv.) and $HBF_4{\cdot}OEt_2$ (1.36 mL, 10.0 mmol, 2.0 equiv.) in MeOH (25 mL, c = 0.4 M) was added into the flask, followed by (1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 3-bromo-benzoate (1.69 g, 5.0 mmol, 1.0 equiv.). The reaction mixture was stirred vigorously at 70 °C for 3 h in an oil bath. After that, the reaction vessel was opened to air, and the residue was purified by silica gel column chromatography[3]. (733 mg, 44% yield)

Step 3: The crude product obtained from **step 2** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **42a**.

Synthesis of compound 43a[5]



43a

Step 1: To an oven-dried 50 mL flask, equipped with a magnetic stirrer, was charged with estrone (2.70 g, 10.0 mmol, 1.0 equiv.) and DIPEA (1.90 mL, 10.0 mmol, 1.0 equiv.) in CH_2Cl_2 (40 mL) under N_2 atmosphere. The reaction mixture was stirred at

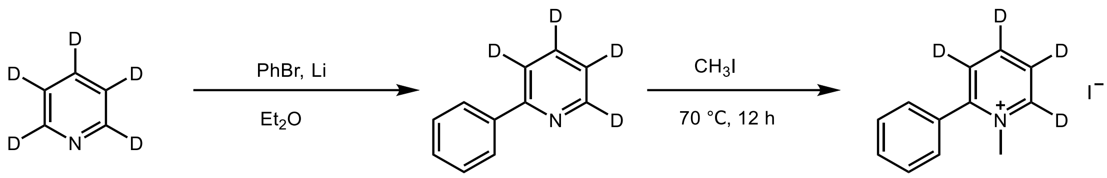
0 °C , and Tf_2O was dropwise added into reaction system over 5 min. The reaction mixture was then allowed to warm to room temperature and stirred 30 min. Upon completion of the reaction, water (50 mL) was added to quench the reaction, followed by filtration and extraction with DCM (3 x 30 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. (2.82 g, 70% yield)

Step 2: To an oven-dried 100 mL flask with a magnetic stirring bar was charged with trifluoromethanesulfonic (2.82 g, 7.0 mmol, 1.0 equiv.), Pd(dppf)Cl_2 (0.51 g, 10 mol%), B_2Pin_2 (3.56 g, 14.0 mmol, 2.0 equiv.) and KOAc (2.06 g, 21.0 mmol, 3.0 equiv.) and then subjected to three cycles of vacuum/nitrogen backfill. Subsequently, dry dioxane (28 mL) was added to the flask, and the mixture was heated at 110 °C for 12 hours. Upon completion of the reaction, the mixture was allowed to cool, and added brine (30 mL), followed by filtration and extraction with ethyl acetate (3 x 15 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. (2.43 g, 91% yield)

Step 3: To an oven-dried 100 mL flask with a magnetic stirring bar was charged with pinacol borate (2.43 g, 6.4 mmol, 1.0 equiv.), $Pd(PPh_3)_4$ (0.22 g, 3 mol%), 2-brompiridine (0.74 mL, 7.7 mmol, 1.2 equiv.) and Na_2CO_3 (2.03 g, 19.2 mmol, 3.0 equiv.) and then subjected to three cycles of vacuum/nitrogen backfill. Subsequently, toluene (50 mL) and water (50 mL) were added to the flask, and the mixture was heated at 110 °C for 12 hours. Upon completion of the reaction, the mixture was allowed to cool, and added aqueous NH_4Cl (30 mL), followed by filtration and extraction with ethyl acetate (3 x 30 mL). The combined organic extracts were then dried over anhydrous magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. (369 mg, 17% yield)

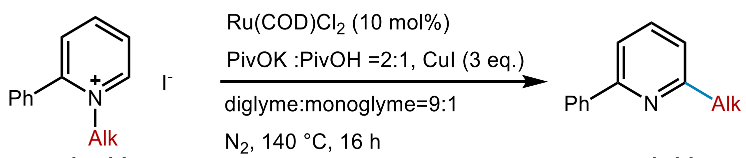
Step 4: The crude product obtained from **step 3** was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **43a**.

2.3 Procedure for synthesis of D*4*-1-Methyl-2-phenyl-pyridin-1-ium iodide (1a-D*4*)



General procedure C: The mixture of bromobenzene (2.25 g, 14.3 mmol) and 2 mL anhydrous ether was added gradually to a three-neck flask containing Li (210.0 mg, 30.0 mmol) and 4 mL anhydrous ether. And then the reaction mixture was refluxed for 1.5 h. The mixture of 2 mL anhydrous toluene and pyridine-d5 (1.34 g, 16.0 mmol) was then added dropwise. The mixture was then stirred for about 50 min at room temperature and refluxed for another 4 h. The reaction mixture was poured cautiously into ice-water, and then acidified with concentrated hydrochloric acid, which was transferred to 250 mL separating funnel and extracted with 50 mL water, the aqueous solution was saturated with sodium hydroxide and extracted with ether. The organic phase was concentrated to give oil crude product, then purified by column chromatography to give pure product as a colorless oil (420.0 mg, 18%). The crude product was reacted according to **General procedure B** to obtain the *N*-alkylpyridinium salt **1a-D*4*** (445 mg, 57%).

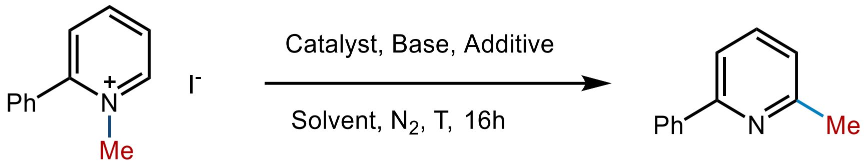
2.4 General procedure for the synthesis of 1 - 44



General procedure D: An oven-dried 10 mL Schlenk tube equipped with magnetic stirring bar were charged with corresponding *N*-alkylpyridinium salts (0.25 mmol, 1.0 equiv.), Ru(COD)Cl_2 (10 mol%), PivOK (0.5 mmol, 2.0 equiv.), PivOH (0.25 mmol,1.0

equiv.), CuI (0.75 mmol, 3.0 equiv.), diglyme (0.9 mL) and monoglyme (0.1 mL), and then subjected to three cycles of vacuum/nitrogen backfill. The resulting solution was stirred at 140 °C (heated by heating plate magnetic stirrer) for 16 h. After cooling to room temperature, water (2 mL) was added to quench the reaction, followed by filtration and extraction with EA (3 x 2 mL) and filtered through a short pad of celite. The volatiles were removed under vacuum and the residue was purified by preparative thin layer chromatography (silica gel, petroleum ether/ethyl acetate 30:1 to 5:1) to give pure product.

2.5 Optimization results of the reaction conditions



1a **1**

Table S1. Optimization of Catalyst

Entry	Catalyst	Base	Additive	Solvent	Yield
1	$Pd(OAc)_2$	PivOK	Cu_2O	DMA	n.d.
2	$Pd(COD)Cl_2$	PivOK	Cu_2O	DMA	n.d.
3	$(PCy)_2NiCl_2$	PivOK	Cu_2O	DMA	n.d.
4	$Ni(acac)_2$	PivOK	Cu_2O	DMA	n.d.
5	$Rh(CO)_2Cl_2$	PivOK	Cu_2O	DMA	n.d.
6	$[(p\text{-cymene})RuCl_2]_2$	PivOK	Cu_2O	DMA	14
7	Tris(2,2- bipyridine)$RuCl_2$	PivOK	Cu_2O	DMA	n.d.
8	$Ru(COD)Cl_2$	PivOK	Cu_2O	DMA	15
9	$RuCl_2(C_6H_6)_2$	PivOK	Cu_2O	DMA	14

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), Catalyst (10 mol%), PivOK (0.5 mmol), Cu_2O (0.5 mmol), DMA (1 mL), 130 °C, 16 h. [b] Isolated yields.

Table S2. Optimization of Additive A

Entry	Additive A	Solvent	Base	Yield (%)
1	Cu_2O	DMA	PivOK	15
2	CuI	DMA	PivOK	27
3	CuBr	DMA	PivOK	17
4	CuCl	DMA	PivOK	23
5	CuCN	DMA	PivOK	n.d.
6	CuSCN	DMA	PivOK	n.d.
7	Cu_2S	DMA	PivOK	n.d.
9	CH_3COOCu	DMA	PivOK	trace
10	CF_3O_3SCu	DMA	PivOK	trace
11	-	DMA	PivOK	n.d.

Table S3. Optimization of Base

Entry	Base	Solvent	Yield (%)
1	PivOK	DMA	27
2	KOH	DMA	trace
3	K_2CO_3	DMA	n.d.
4	Li_2CO_3	DMA	trace
5	t-BuOK	DMA	trace
6	K_3PO_4	DMA	trace
7	NaF	DMA	n.d.
9	CH_3COONa	DMA	trace
10	NEt_3	DMA	trace
11	DBU	DMA	trace

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), Ru(COD)Cl_2 (10 mol%), Base (0.5 mmol), CuI (0.5 mmol), DMA (1 mL), 130 °C, 16 h. [b] Isolated yields.

Table S4. Optimization of solvent and additive A

Entry	Solvent	CuI (x equiv.)	Yield (%)
1	DMA	2.0	27
2	DMF	2.0	trace
3	DMSO	2.0	trace
4	Monoglyme	2.0	trace
5	Trimethyl orthoacetate	2.0	24
6	N-Methyl-2-pyrrolidone	2.0	24
7	Diglyme	0.5	n.d.
8	Diglyme	1.0	n.d.
9	Diglyme	1.5	11
10	Diglyme	2.0	13
11	Diglyme	3.0	28

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), Ru(COD)Cl_2 (10 mol%), PivOK (0.5 mmol), CuI (x equiv.), Solvent (1 mL), 130 °C, 16 h. [b] Isolated yields.

Table S5. Optimization of Solvent and temperature.

Entry	Solvent A	Solvent B	Ratio	Temperature (°C)	Yield (%)
1	Diglyme	-	-	130	28
2	Diglyme	DMA	5:5	130	32
3	Diglyme	DMA	7:3	130	37
4	Diglyme	Monoglyme	5:5	130	44
5	Diglyme	Monoglyme	6:4	130	31
6	Diglyme	Monoglyme	7:3	130	46
7	Diglyme	Monoglyme	8:2	130	40
8	Diglyme	Monoglyme	9:1	130	51
9	Diglyme	Monoglyme	9:1	140	53

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), Ru(COD)Cl_2 (10 mol%), PivOK (0.5 mmol), CuI (3 equiv.), Co-solvent (1 mL), T °C, 16 h, N_2. [b] Isolated yields.

Table S6. Optimization of additive B

Entry	Base	x_1 equiv.	Additive B	x_2 equiv.	Yield (%)
1	PivOK	2.0	-	-	53
2	PivOK	2.0	PivOH	0.5	63
3	PivOK	2.0	PivOH	1.0	87
4	PivOK	2.0	PivOH	1.6	73
5	PivOK	2.0	PivOH	2.0	68
6	PivOK	1.0	PivOH	1.0	50

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), Ru(COD)Cl_2 (10 mol%), PivOK (x_1 equiv.), CuI (0.75 mmol), Additive B (x_2 equiv.), Co-solvent (1 mL), N_2, 140 °C, 16 h. [b] Isolated yields.

Table S7. Optimization of the loading of catalyst

Entry	$Ru(COD)Cl_2$	PivOK (x_1 eq.)	PivOH (x_2 eq.)	CuI (y eq.)	Solvent (mL)	Yield (%)
1	5	2.0	1.0	3.0	1	63
2	5	2.0	1.0	2.0	1	34
3	5	2.0	1.0	1.0	1	trace
4	5	1.0	1.0	3.0	1	60
5	5	1.0	0.5	3.0	1	50
6	5	2.0	1.0	3.0	0.5	60

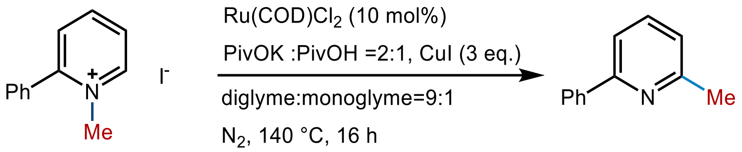
[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), $Ru(COD)Cl_2$ (5 mol%), PivOK (x_1 equiv.), CuI (y equiv.), PivOH (x_2 equiv.), Co-solvent (1 mL), N_2, 140 °C, 24 h. [b] Isolated yields.

Table S8. The other first-row transition metal salts as potential additives.

Entry	Additive	1
1	$FeSO_4 \cdot 7H_2O$	n.d.
2	$Ni(CH_3COO)_2 \cdot 4H_2O$	n.d.
3	$Zn(CH_3COO)_2 \cdot 2H_2O$	n.d.
4	$ZnSO_4 \cdot 7H_2O$	trace

[a] Reaction conditions: *N*-alkylpyridinium salt (0.25 mmol), $Ru(COD)Cl_2$ (5 mol%), PivOK (2 equiv.), Additive (3 equiv.), PivOH (1 equiv.), Co-solvent (1 mL), N_2, 140 °C, 24 h. [b] Isolated yields.

2.6 Scale-up Reaction



1a **1**

Experimental step: An oven-dried 10 mL Schlenk tube equipped with magnetic stirring bar were charged with *N*-methylpyridinium salt (1.5 g, 5.0 mmol, 1.0 equiv.), Ru(COD)Cl_2 (0.14 g, 10 mol%), PivOK (1.4 g, 10.0 mmol, 2.0 equiv.), PivOH (0.57 mL, 5.0 mmol,1.0 equiv.), CuI (2.86 g, 15.0 mmol, 3.0 equiv.), diglyme (18 mL) and monoglyme (2 mL), and then subjected to three cycles of vacuum/nitrogen backfill. The resulting solution was stirred at 140 °C (heated by heating plate magnetic stirrer) for 16 h. After cooling to room temperature, water (40 mL) was added to quench the reaction, followed by filtration and extraction with EA (3 x 40 mL) and filtered through a short pad of celite. The volatiles were removed under vacuum and the residue was purified by silica gel column chromatography to give pure product (A colorless oil, 658 mg, 78%).

2.7 Mechanistic Study

2.7.1 Mercury poisoning test

Experimental step 1: According to **General procedure D,** 200 μL of mercury was added under the standard conditions. The target product was obtained (78% yield). The result is shown in **Figure S1**. The result clearly shows that the addition of Hg did not inhibit the reaction.

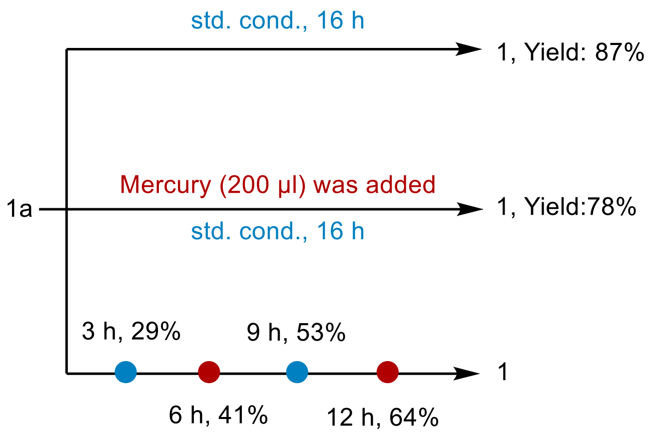


Figure S1. Mercury poisoning test.

Experimental step 2: According to **General procedure D,** the resulting solution was stirred at 140 °C for 3 h, 6 h, 9 h, and 12 h, with yields of 29%, 41%, 53%, and 64%, respectively. The variation in yield over time is shown in **Figure S2**.

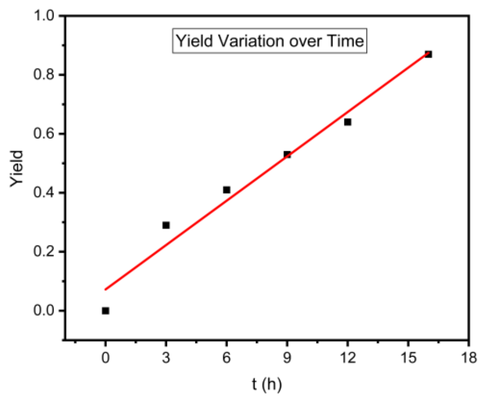


Figure S2. The variation in yield over time

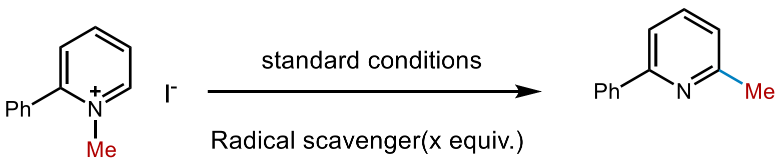
These results clearly show that the reaction proceeded via homogeneous catalysis.

2.7.2 Radical verification experiment

1. Radical trapping experiment

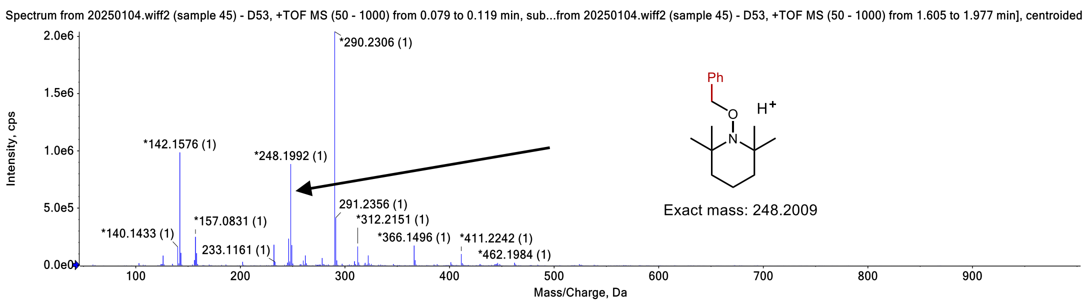
Experimental step 1: According to **General procedure D,** radical scavenger (x equiv.) was added under the standard conditions. The result is shown in **Table S9**.

Table S9. Radical trapping experiment



Entry	Radical scavenger	x equiv.	Yield (%)
1	TEMPO	3	17
2	TEMPO	6	trace
3	DMPO	2	trace

Experimental step 2: According to **General procedure D,** TEMPO (6 equiv.) was added. The resulting solution was stirred at 140 °C for 8 h. After the reaction was completed, the volatiles were measured by HRMS. The phenyl-TEMPO adduct was detected. The result is shown in **Figure S3**.



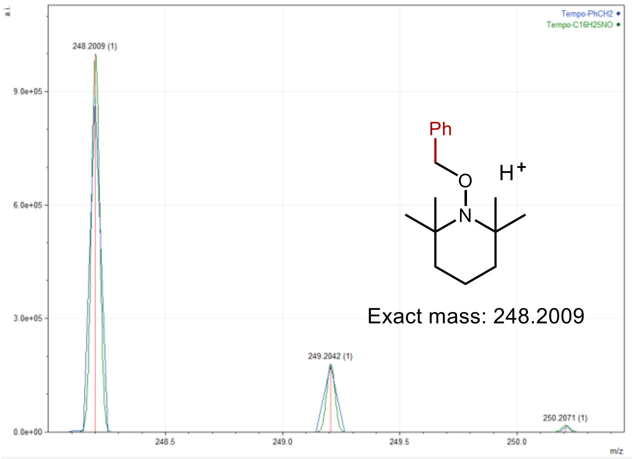
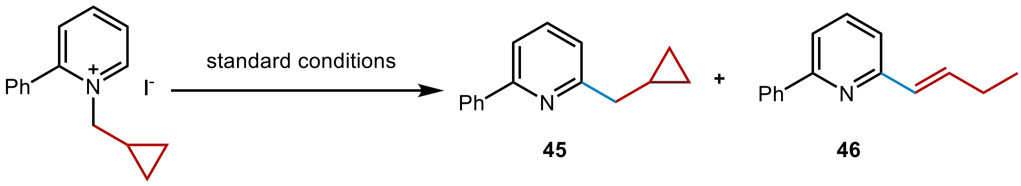


Figure S3. Phenyl-TEMPO adduct was detected by HRMS

2. Radical clock experiment

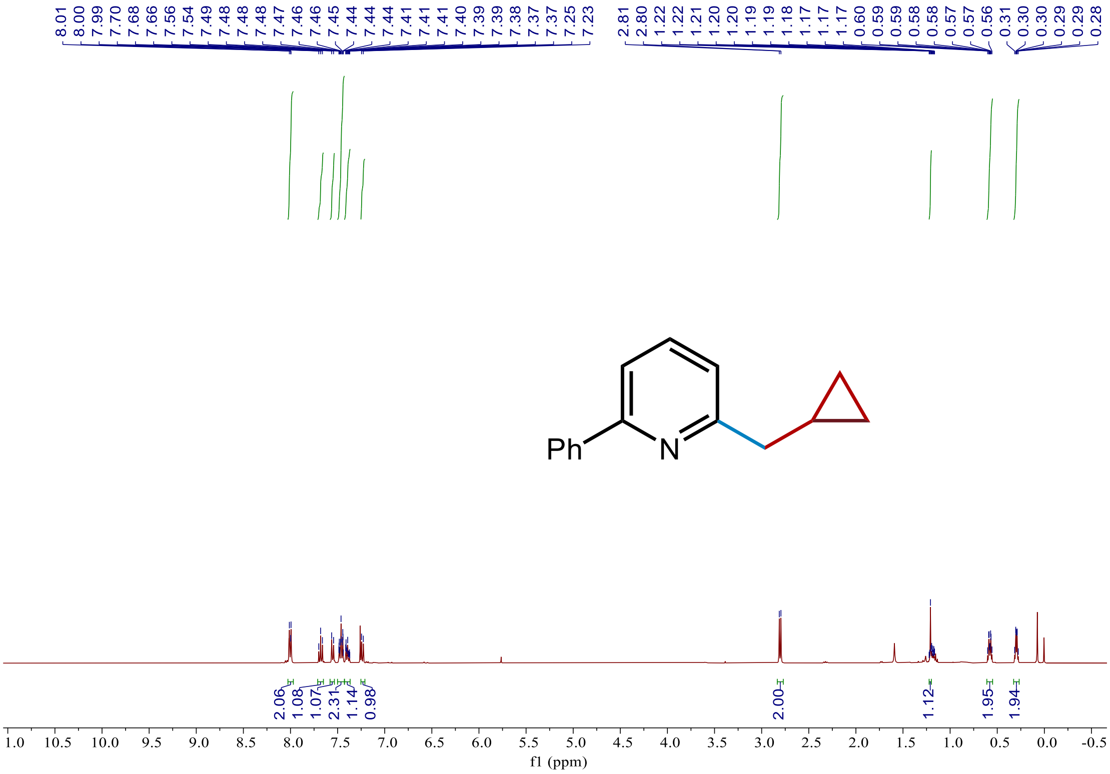


Experimental step: According to **General procedure D**, the volatiles were removed under vacuum and the residue was purified by preparative thin layer chromatography (silica gel, petroleum ether/ethyl acetate 30:1) to yield **45** (3 mg, 6%) as a colorless oil and a ring-opening alkylation product **46** (2 mg, 4%).

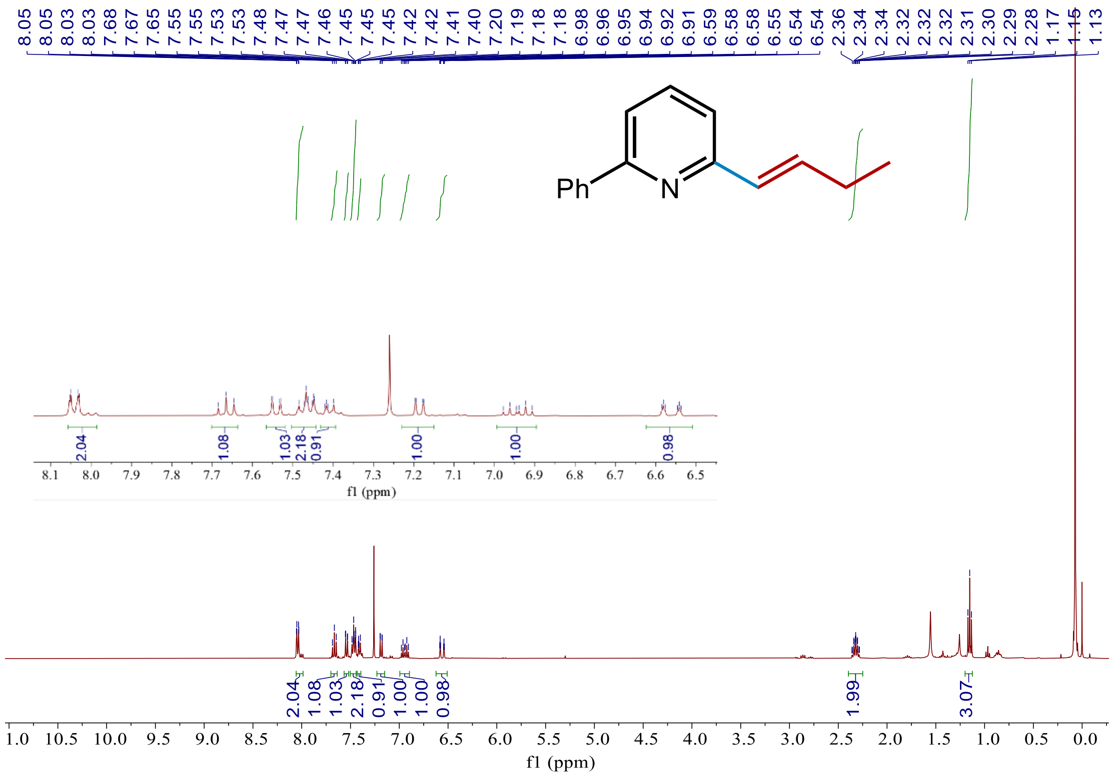
2-(cyclopropylmethyl)-6-phenylpyridine (45): ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 8.03 – 7.97 (m, 2H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.50 – 7.43 (m, 2H), 7.43 – 7.37 (m, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 2.80 (d, *J* = 7.0 Hz, 2H), 1.24 – 1.17 (m, 1H), 0.61 – 0.55 (m, 2H), 0.33 – 0.27 (m, 2H).

(E)-2-(but-1-en-1-yl)-6-phenylpyridine (46): ^{1}**H NMR** (400 MHz, Chloroform-*d*) 8.08 – 7.98 (m, 2H), 7.67 (t, *J* = 7.8 Hz, 1H), 7.54 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.44 – 7.38 (m, 1H), 7.19 (dd, *J* = 7.7, 0.9 Hz, 1H), 6.94 (dt, *J* = 15.6, 6.5 Hz, 1H), 6.56 (dt, *J* = 15.6, 1.7 Hz, 1H), 2.38 – 2.26 (m, 2H), 1.15 (t, *J* = 7.5 Hz, 3H).

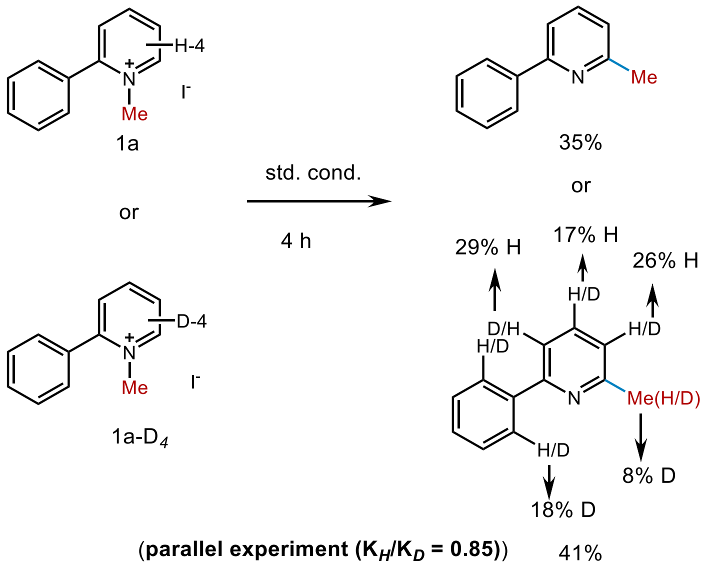
2-(cyclopropylmethyl)-6-phenylpyridine (45):



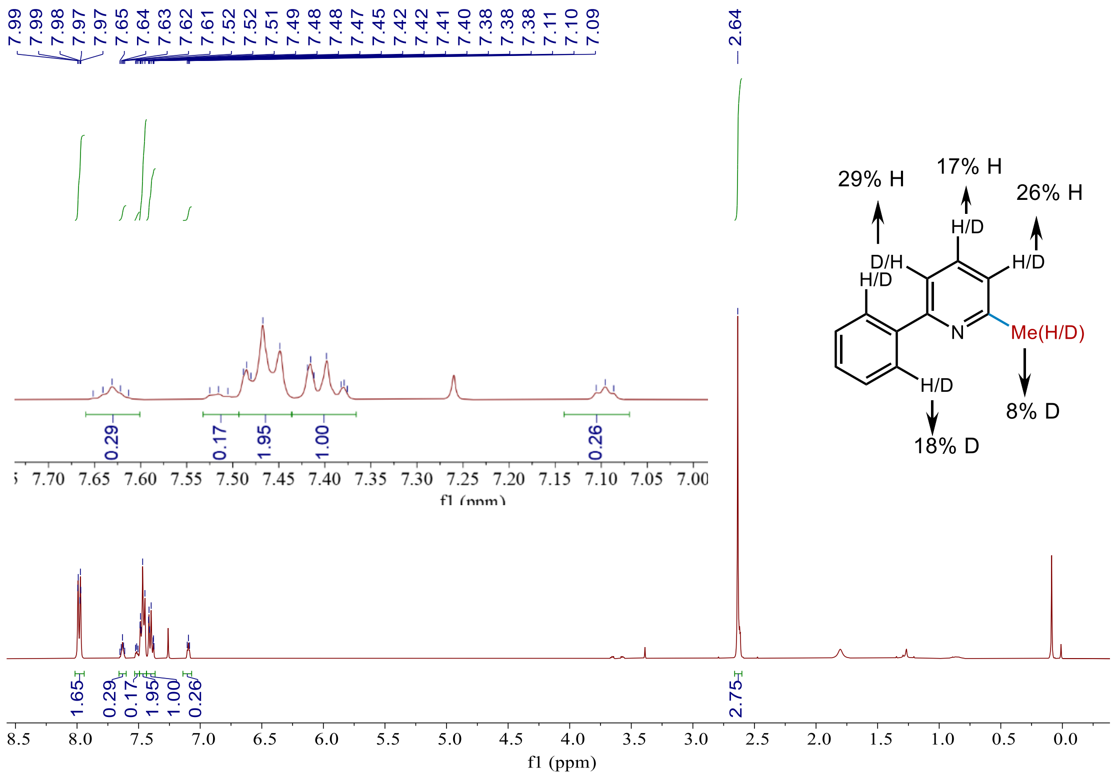
(E)-2-(but-1-en-1-yl)-6-phenylpyridine (46):



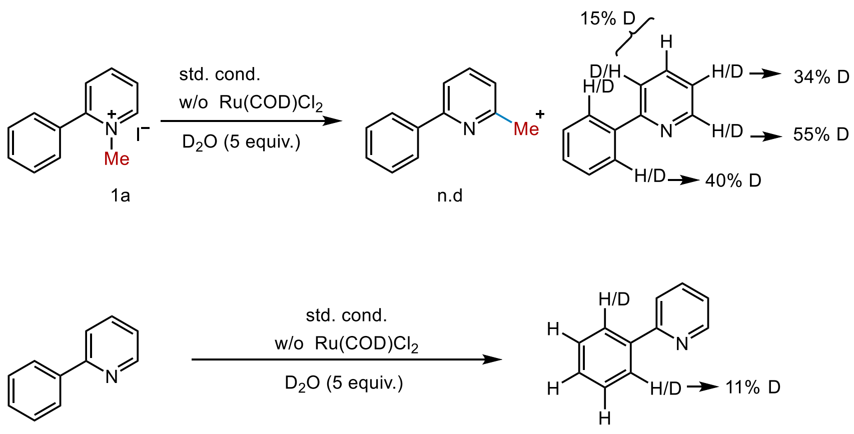
2.7.3 KIE experiment



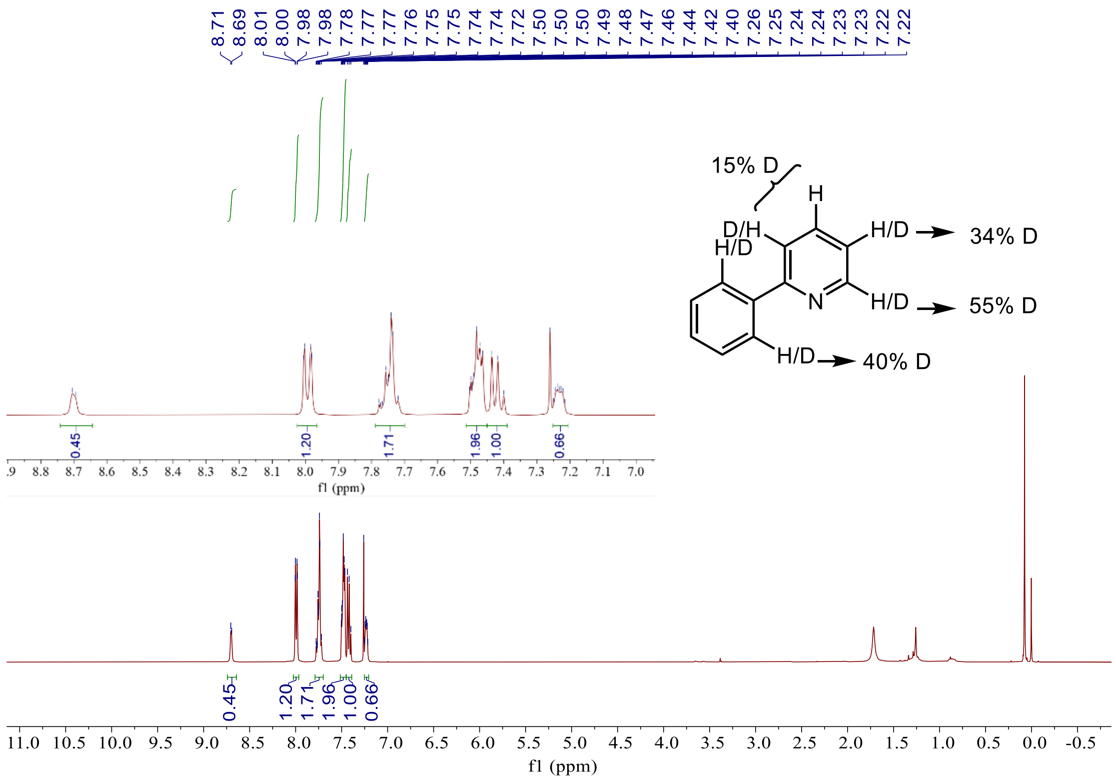
Experimental step: According to **General procedure D,** the resulting solution was stirred at 140 °C for 4 h. The isolated yields of **1a**/**1a-D4** were 35% and 41%, respectively. The KIE value was determined to be K_H: K_D = 35%/41% = 0.85. The results were analysis by ^{1}H NMR.



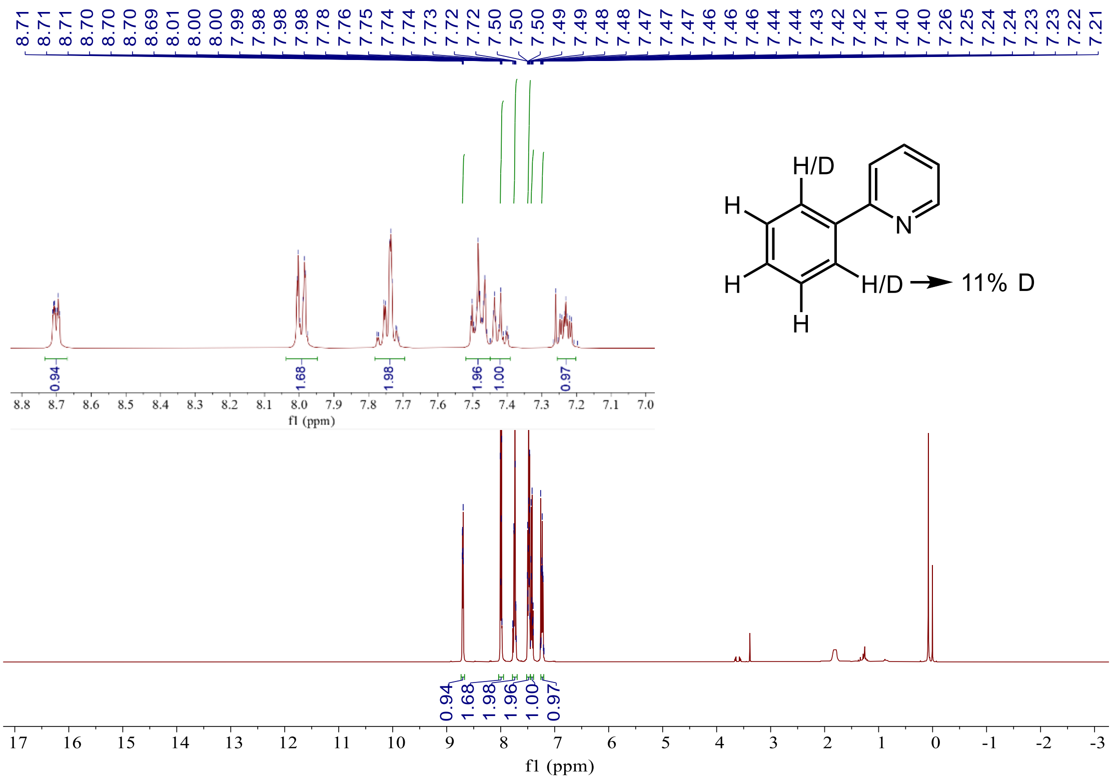
2.7.4 Deuterium experiment



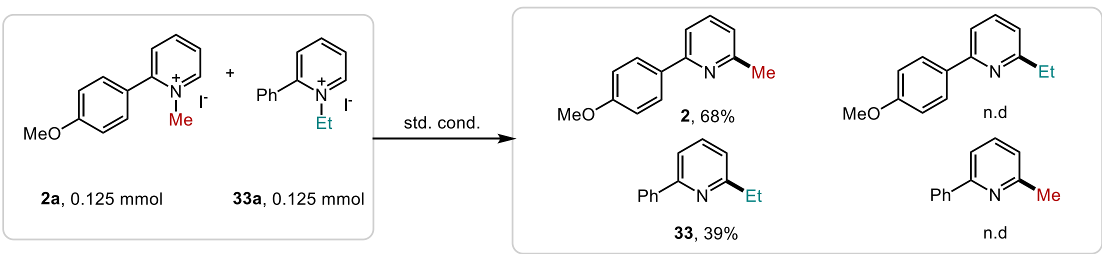
Experimental step 1: According to **General procedure D,** the reaction was carried out under standard conditions in the absence of Ru(COD)Cl_2 and with the addition of D_2O (1.1 mL, 1.25mmol, 5 equiv.). The results were analysis by ^{1}H NMR.

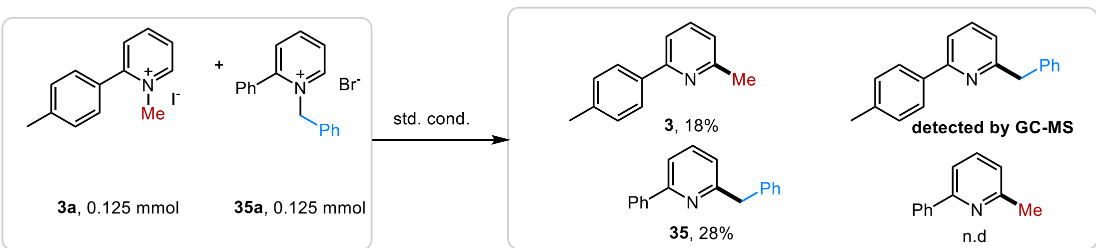


Experimental step 2: According to **General procedure D,** 2-phenylpyridine (35.7 μL, 0.25 mmol, 1.0 equiv.) was used to replace the *N*-methylpyridinium salt (1a). The reaction was carried out under standard conditions in the absence of Ru(COD)Cl_2 and with the addition of D_2O (1.13 mL, 1.25mmol, 5 equiv.).



2.7.5 Crossover experiment





Experimental step 1: According to **General procedure D**, an oven-dried 10 mL Schlenk tube equipped with magnetic stirring bar were charged with **2a** (0.125 mmol, 1.0 equiv.) and **33a** (0.125 mmol, 1.0 equiv.), Ru(COD)Cl_2 (7.0 mg, 10 mol%), PivOK (70.0 mg, 0.5 mmol, 2.0 equiv.), PivOH (28.7 uL, 0.25 mmol,1.0 equiv.), CuI (142.8 mg, 0.75 mmol, 3.0 equiv.), diglyme (0.9 mL) and monoglyme (0.1 mL), and then subjected to three cycles of vacuum/nitrogen backfill. The resulting solution was stirred at 140 °C (heated by heating plate magnetic stirrer) for 16 h. After cooling to room temperature, water (2 mL) was added to quench the reaction, followed by filtration and extraction with EA (3 x 2 mL). No product was detected in the crossover experiment.

Experimental step 2: According to **General procedure D**, an oven-dried 10 mL Schlenk tube equipped with magnetic stirring bar were charged with **3a** (0.125 mmol, 1.0 equiv.) and **35a** (0.125 mmol, 1.0 equiv.), Ru(COD)Cl_2 (7.0 mg, 10 mol%), PivOK (70.0 mg, 0.5 mmol, 2.0 equiv.), PivOH (28.7 μL, 0.25 mmol,1.0 equiv.), CuI (142.8 mg, 0.75 mmol, 3.0 equiv.), diglyme (0.9 mL) and monoglyme (0.1 mL), and then subjected to three cycles of vacuum/nitrogen backfill. The resulting solution was stirred at 140 °C (heated by heating plate magnetic stirrer) for 16 h. After cooling to room temperature, water (2 mL) was added to quench the reaction, followed by filtration and extraction with EA (3 x 2 mL). Cross-products were detected in the crossover experiment by GC-MS.

2.7.6 Proposed mechanism

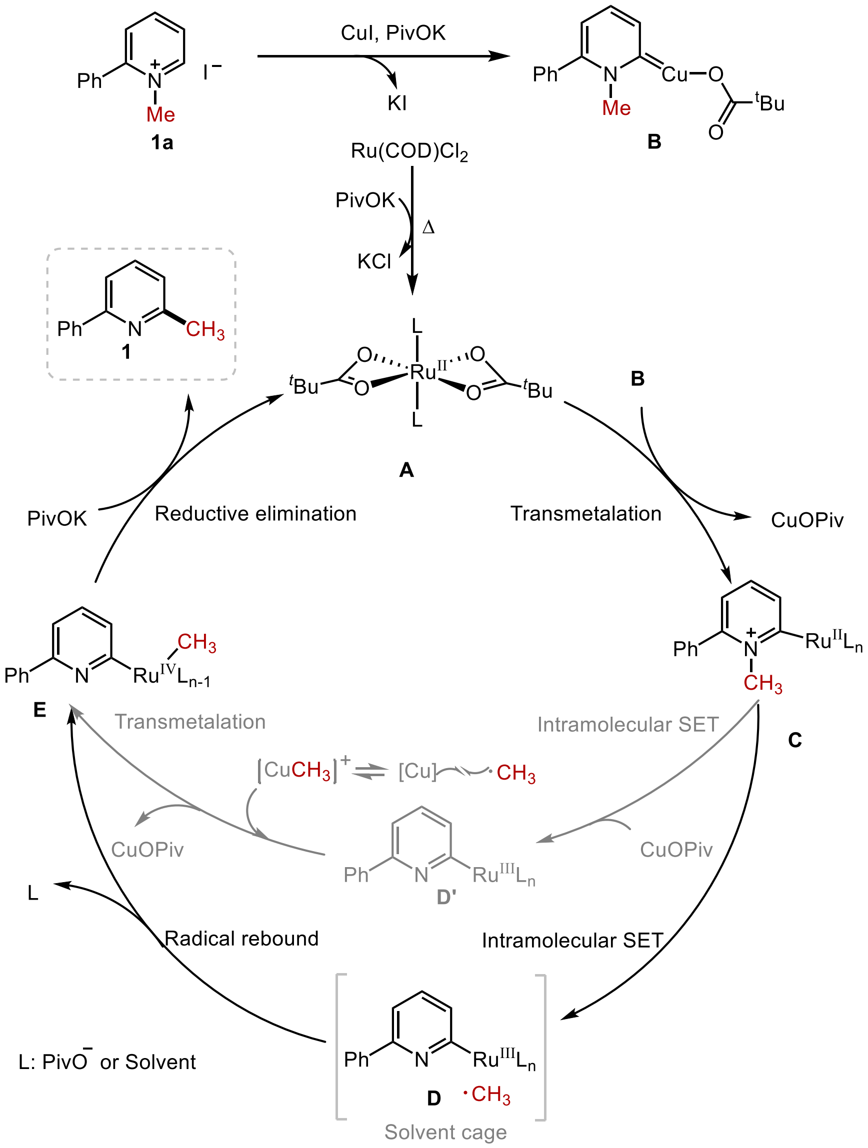
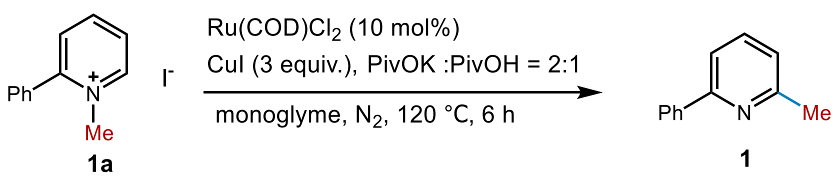


Figure S4. Proposed mechanism

2.7.7 X-ray experiment



Experimental step: According to **General procedure D**, an oven-dried 10 mL Schlenk tube equipped with magnetic stirring bar were charged with corresponding *N*-

alkylpyridinium salts (0.25 mmol, 1.0 equiv.), Ru(COD)Cl_2 (10 mol%), PivOK (0.5 mmol, 2.0 equiv.), PivOH (0.25 mmol, 1.0 equiv.), CuI (0.75 mmol, 3.0 equiv.), monoglyme (0.5 mL), and then subjected to three cycles of vacuum/nitrogen backfill. The resulting solution was stirred at 120 °C (heated by heating plate magnetic stirrer) for 6 h. The reaction was concentrated, then the resulting powder was analyzed by X-ray photoelectron spectroscopy (XPS). The result was shown in **Figure S4.**

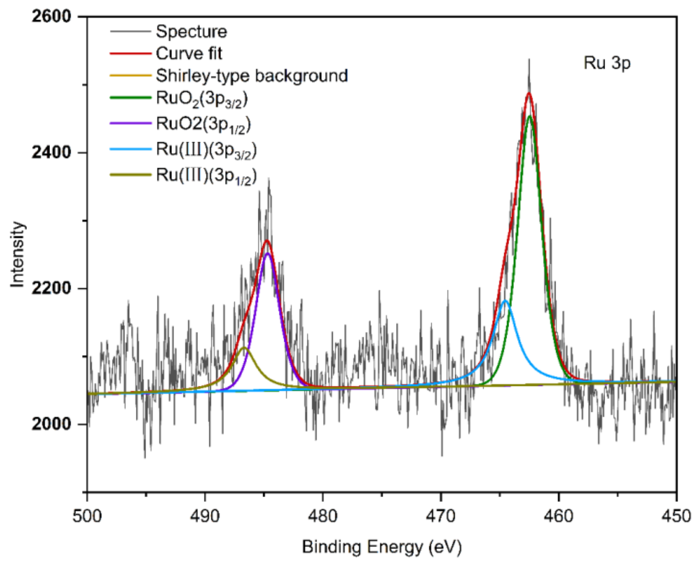


Figure S5. Two distinct oxidation states of Ru(III) and Ru(IV)

The Ru 3p spectra indicate the presence of two distinct Ru oxidation states, namely Ru(III) and Ru(IV).

3. Characterization Data

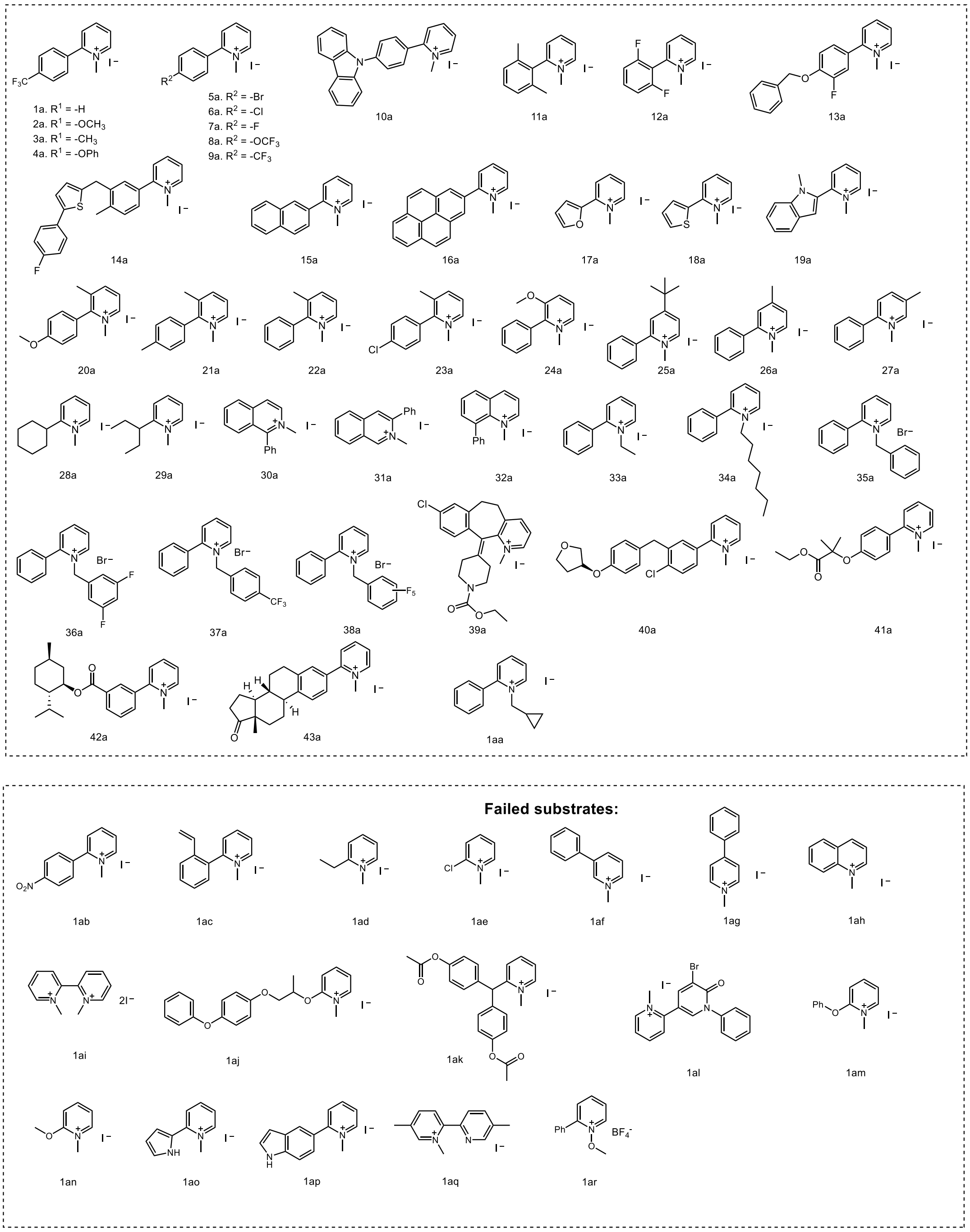
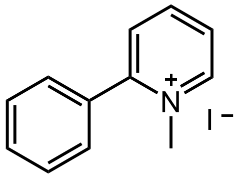
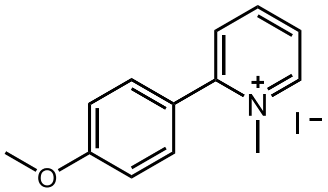


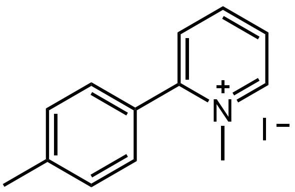
Figure S6: Characterization data for raw materials



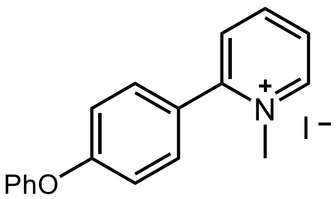
1-methyl-2-phenylpyridin-1-ium iodide (1a): The substance was prepared using **General Procedure B.** Yellow solid (1.36 g, 92% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.17 (d, J = 6.9 Hz, 1H), 8.65 (td, J = 7.9, 1.5 Hz, 1H), 8.18 (ddd, J = 7.7, 6.2, 1.6 Hz, 1H), 8.09 (dd, J = 7.9, 1.6 Hz, 1H), 7.73 – 7.64 (m, 5H), 4.13 (s, 3H). Spectra matched literature values.[6]



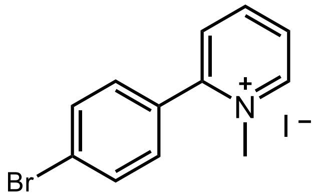
2-(4-methoxyphenyl)-1-methylpyridin-1-ium iodide (2a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.31 g, 80% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.11 (d, J = 6.3 Hz, 1H), 8.60 (td, J = 7.8, 1.5 Hz, 1H), 8.11 (ddd, J = 7.7, 6.2, 1.6 Hz, 1H), 8.05 (dd, J = 8.0, 1.5 Hz, 1H), 7.71 – 7.59 (m, 2H), 7.24 – 7.16 (m, 2H), 4.15 (s, 3H), 3.87 (s, 3H). Spectra matched literature values.[6]



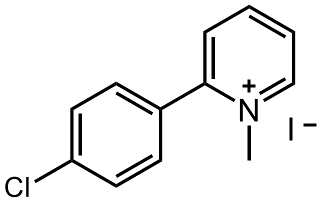
1-methyl-2-(p-tolyl)pyridin-1-ium iodide (3a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.23 g, 79% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.14 (d, J = 6.3 Hz, 1H), 8.62 (td, J = 7.9, 1.5 Hz, 1H), 8.14 (ddd, J = 7.8, 6.1, 1.5 Hz, 1H), 8.05 (dd, J = 7.9, 1.5 Hz, 1H), 7.59 (d, J = 4.6 Hz, 2H), 7.47 (d, J = 8.0 Hz, 2H), 4.13 (s, 3H), 2.43 (s, 3H). Spectra matched literature values.[6]



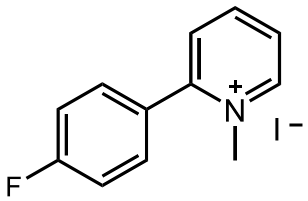
1-methyl-2-(4-phenoxyphenyl)pyridin-1-ium iodide (4a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.43 g, 74% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.13 (d, J = 6.3 Hz, 1H), 8.63 (td, J = 7.9, 1.4 Hz, 1H), 8.14 (ddd, J = 7.7, 6.2, 1.6 Hz, 1H), 8.08 (dd, J = 8.0, 1.5 Hz, 1H), 7.74 – 7.68 (m, 2H), 7.53 – 7.45 (m, 2H), 7.30 – 7.23 (m, 1H), 7.23 – 7.13 (m, 4H), 4.16 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 159.8, 155.6, 155.1, 147.2, 145.8, 132.0, 130.9, 130.4, 126.9, 126.6, 125.2, 120.4, 118.4, 47.6. **HRMS (ESI)**: call for $C_{18}H_{16}NO[M-I]^+$: 262.1226; Found: 262.1217.



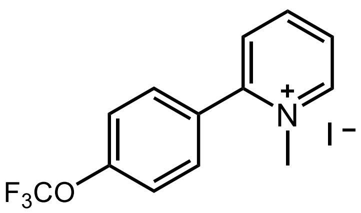
2-(4-bromophenyl)-1-methylpyridin-1-ium iodide (5a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.42 g, 76% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.16 (d, J = 6.1 Hz, 1H), 8.65 (td, J = 7.9, 1.4 Hz, 1H), 8.18 (ddd, J = 7.8, 6.2, 1.6 Hz, 1H), 8.09 (dd, J = 7.9, 1.6 Hz, 1H), 7.92 – 7.85 (m, 2H), 7.67 – 7.63 (m, 2H), 4.12 (s, 3H). Spectra matched literature values.[7]



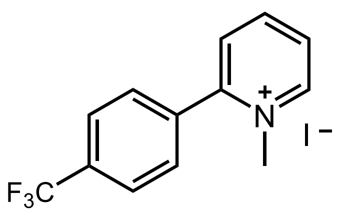
2-(4-chlorophenyl)-1-methylpyridin-1-ium iodide (6a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.24 g, 75% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.17 (d, J = 6.3 Hz, 1H), 8.66 (td, J = 7.8, 1.5 Hz, 1H), 8.19 (ddd, J = 7.8, 6.1, 1.6 Hz, 1H), 8.10 (d, J = 7.9 Hz, 1H), 7.80 – 7.70 (m, 4H), 4.12 (s, 3H). Spectra matched literature values.[6]



2-(4-fluorophenyl)-1-methylpyridin-1-ium iodide (7a): The substance was prepared using **General Procedure A and B.** White solid (1.18 g, 75% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.16 (d, J = 6.9 Hz, 1H), 8.64 (td, J = 7.8, 1.5 Hz, 1H), 8.17 (ddd, J = 7.7, 6.2, 1.6 Hz, 1H), 8.08 (dd, J = 8.1, 1.6 Hz, 1H), 7.81 – 7.75 (m, 2H), 7.55 – 7.49 (m, 2H), 4.12 (s, 3H). Spectra matched literature values.[6]

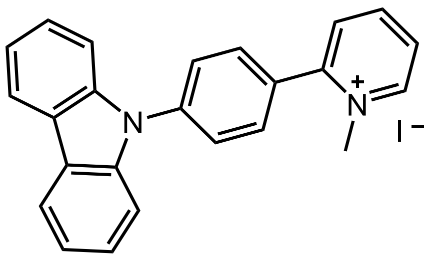


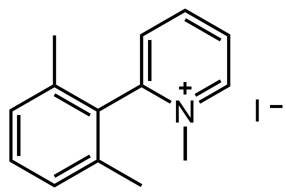
1-methyl-2-(4-(trifluoromethoxy)phenyl)pyridin-1-ium iodide (8a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.56 g, 82% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.17 (d, J = 6.1 Hz, 1H), 8.66 (td, J = 7.9, 1.4 Hz, 1H), 8.19 (ddd, J = 7.8, 6.2, 1.6 Hz, 1H), 8.12 (dd, J = 8.0, 1.6 Hz, 1H), 7.88 – 7.83 (m, 2H), 7.69 – 7.67 (m, 2H), 4.12 (s, 3H). **^{19}F NMR** (376 MHz, DMSO-d_6) δ -56.65. **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 154.2, 150.5, 147.3, 146.0, 132.3, 131.4, 130.4, 127.5, 121.9, 121.7 (q, J_{CF} = 252.5 Hz), 47.7. **HRMS (ESI)**: call for $C_{13}H_{11}F_3NO$ [M-I]$^+$: 254.0787. Found: 254.0776.



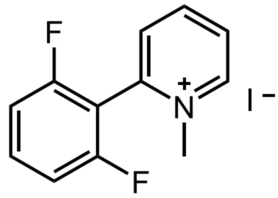
1-methyl-2-(4-(trifluoromethyl)phenyl)pyridin-1-ium iodide (9a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.54 g, 84% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.20 (d, *J* = 5.5 Hz, 1H), 8.69 (td, *J* = 7.9, 1.4 Hz, 1H), 8.23 (ddd, *J* = 7.8, 6.1, 1.6 Hz, 1H), 8.14 (dd, *J* = 8.0, 1.6 Hz, 1H), 8.08 – 8.03 (m, 2H), 7.97 – 7.92 (m, 2H), 4.12 (s, 3H). Spectra matched literature values.[6]

2-(4-(9H-carbazol-9-yl)phenyl)-1-methylpyridin-1-ium iodide (10a): The substance was prepared using **General Procedure A and B.** Brown solid (1.61 g, 70% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.20 (d, *J* = 6.3 Hz, 1H), 8.71 (t, *J* = 7.9 Hz, 1H), 8.29 (d, *J* = 7.7 Hz, 2H), 8.27 – 8.19 (m, 2H), 8.03 – 7.93 (m, 4H), 7.56 (d, *J* = 8.2 Hz, 2H), 7.50 (ddd, *J* = 8.2, 7.0, 1.3 Hz, 2H), 7.35 (td, J = 7.4, 1.1 Hz, 2H), 4.27 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 154.9, 147.3, 146.0, 140.2, 139.8, 131.8, 130.9, 130.4, 127.4, 127.3, 126.9, 123.6, 121.2, 121.1, 110.3, 47.8. **HRMS (ESI)**: call for $C_{24}H_{19}N_2$ [M-I]$^+$: 335.1543. Found: 335.1535.



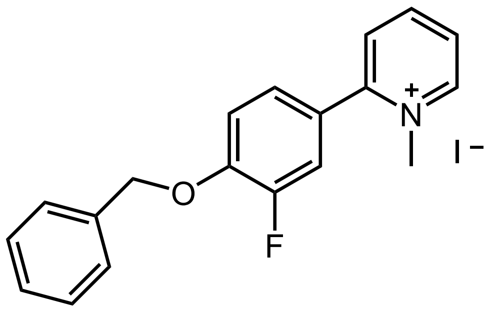


2-(2,6-dimethylphenyl)-1-methylpyridin-1-ium iodide (11a): The substance was prepared using **General Procedure A and B.** White solid (1.18 g, 73% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.25 (d, *J* = 6.3 Hz, 1H), 8.71 (td, *J* = 7.9, 1.4 Hz, 1H), 8.23 (ddd, *J* = 7.8, 6.2, 1.6 Hz, 1H), 8.09 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.48 – 7.44 (m, 1H), 7.32 (d, *J* = 7.6 Hz, 2H), 3.93 (s, 3H), 1.99 (s, 6H). Spectra matched literature values.[8]



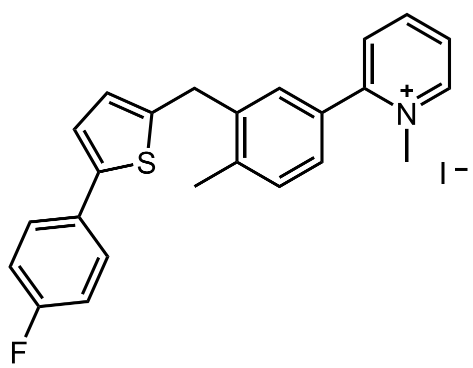
2-(2,6-difluorophenyl)-1-methylpyridin-1-ium iodide (12a): The substance was prepared using **General Procedure A and B.** White solid (1.27 g, 76% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.20 (d, *J* = 6.7 Hz, 1H), 8.69 (td, *J* = 7.9, 1.4 Hz, 1H), 8.23 (ddd, *J* = 7.8, 6.1, 1.5 Hz, 1H), 8.14 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.64 (tt, *J* =

9.5, 2.4 Hz, 1H), 7.59 – 7.49 (m, 2H), 4.15 (s, 3H). **^{19}F NMR** (376 MHz, DMSO-*d*$_6$) δ -107.70. **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*$_6$) δ 162.7 (dd, J_{CF} = 252.5 Hz, 10.1 Hz), 152.8, 147.3, 146.2, 134.9 (t, J_{CF} = 10.8 Hz), 130.4, 128.0, 114.2 – 113.6 (m), 107.2 (t, J_{CF} = 25.7 Hz), 47.7. **HRMS (ESI)**: call for $C_{12}H_{10}F_2N$ [M-I]$^+$: 206.0776. Found: 206.0771.

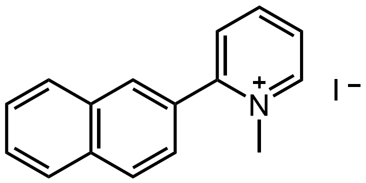


2-(4-(benzyloxy)-3-fluorophenyl)-1-methylpyridin-1-ium iodide (13a): White solid (1.72 g, 82% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 9.18 (d, *J* = 6.2 Hz, 1H), 8.67 (t, *J* = 7.8 Hz, 1H), 8.24 – 8.16 (m, 1H), 8.10 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.56 (dd, *J* = 10.1, 8.4 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.44 (dd, *J* = 8.3, 6.8 Hz, 2H), 7.41 – 7.34 (m, 1H), 7.30 (ddt, *J* = 8.1, 3.8, 1.7 Hz, 1H), 5.26 (s, 2H), 4.12 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*$_6$) δ 154.5, 153.6 (d, J_{CF} = 252.5 Hz), 147.1, 146.7 (d, J_{CF} = 11.0 Hz), 145.9, 136.4, 130.4, 129.1, 128.8 (d, J_{CF} = 3.3 Hz), 128.5, 127.3, , 123.2 (d, J_{CF} = 7.8 Hz), 117.4, 117.2, 117.2 (d, J_{CF} = 2.8 Hz), 71.1, 47.6. **HRMS (ESI)**: call for $C_{19}H_{17}FNO$ [M-I]$^+$: 294.1289. Found: 294.1281.

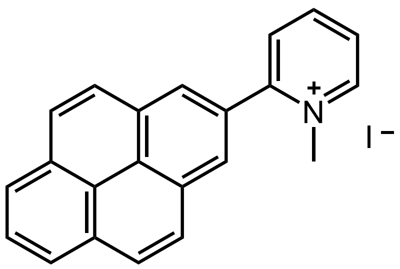
2-(3-((5-(4-fluorophenyl)thiophen-2-yl) methyl)-4-methylphenyl)-1-methylpridin-1-ium iodide (14a): Light yellow solid (2.03 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 9.13 (d, *J* = 6.2 Hz, 1H), 8.63 (t, *J* = 7.9 Hz, 1H), 8.14 (t, *J* = 7.0 Hz, 1H), 8.07 (d, *J* = 7.9 Hz, 1H), 7.69 – 7.56 (m, 3H), 7.56 – 7.45 (m, 2H), 7.32 (d, *J* = 3.7 Hz, 1H), 7.22 (t, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 3.6 Hz, 1H), 4.27 (s, 2H), 4.16 (s, 3H), 2.42 (s, 3H). **^{19}F NMR** (376 MHz, DMSO-*d*$_6$) δ -114.85. **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*$_6$) δ 161.9 (d, J_{CF} = 242.4 Hz), 155.4, 147.2, 145.9, 142.8, 141.1, 140.2, 139.9, 131.4, 130.9 (d, J_{CF} = 3.2 Hz), 130.3 (d, J_{CF} = 7.8 Hz), 130.1, 128.2, 127.5, 127.4,



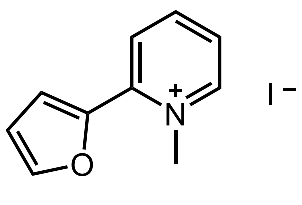
126.9, 124.1, 116.5, 116.3, 47.7, 33.5, 19.5. **HRMS (ESI)**: call for $C_{24}H_{21}FNS$ [M-I]$^+$: 374.1373. **Found**: 374.1356.



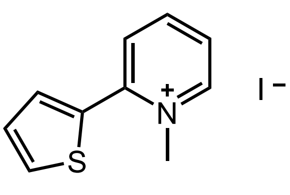
1-methyl-2-(naphthalen-2-yl)pyridin-1-ium iodide (15a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.40 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.29 (d, *J* = 6.8 Hz, 1H), 8.73 (td, *J* = 7.9, 1.5 Hz, 1H), 8.29 (ddd, *J* = 13.4, 7.6, 1.8 Hz, 2H), 8.22 – 8.12 (m, 2H), 7.82 – 7.74 (m, 2H), 7.68 (ddd, *J* = 8.2, 6.8, 1.2 Hz, 1H), 7.60 (ddd, *J* = 8.3, 6.8, 1.4 Hz, 1H), 7.43 (d, *J* = 8.4 Hz, 1H), 3.95 (s, 3H). Spectra matched literature values.[6]



1-methyl-2-(pyren-2-yl)pyridin-1-ium iodide (16a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (1.5 g, 71% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.35 (d, *J* = 6.3 Hz, 1H), 8.79 (td, *J* = 7.9, 1.4 Hz, 1H), 8.55 (d, *J* = 7.9 Hz, 1H), 8.47 (t, *J* = 8.0 Hz, 2H), 8.41 (d, *J* = 9.0 Hz, 1H), 8.37 – 8.25 (m, 5H), 8.20 (t, *J* = 7.6 Hz, 1H), 7.74 (d, *J* = 9.2 Hz, 1H), 4.01 (s, 3H). Spectra matched literature values.[6]

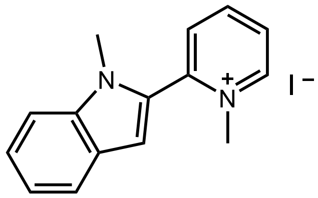


2-(furan-2-yl)-1-methylpyridin-1-ium iodide (17a): The substance was prepared using **General Procedure A and B.** Light yellow solid (1.11 g, 77% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.01 (d, *J* = 6.2 Hz, 1H), 8.57 (td, *J* = 8.0, 1.5 Hz, 1H), 8.44 (dd, *J* = 8.4, 1.5 Hz, 1H), 8.29 (d, *J* = 1.8 Hz, 1H), 7.98 (ddd, *J* = 7.7, 6.2, 1.5 Hz, 1H), 7.75 (d, *J* = 3.7 Hz, 1H), 6.98 (dd, *J* = 3.8, 1.7 Hz, 1H), 4.44 (s, 3H). Spectra matched literature values.[6]

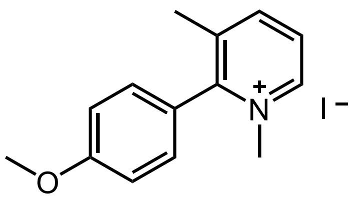


1-methyl-2-(thiophen-2-yl)pyridin-1-ium iodide (18a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (1.09 g, 60% yield). **^{1}H NMR** (400 MHz, DMSO-

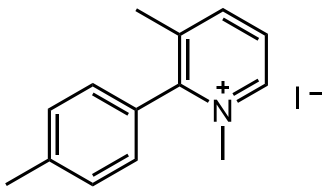
*d*6) δ 9.13 (d, *J* = 6.0 Hz, 1H), 8.58 (td, *J* = 7.9, 1.5 Hz, 1H), 8.22 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.15 – 8.07 (m, 2H), 7.82 (dd, *J* = 3.7, 1.2 Hz, 1H), 7.38 (dd, *J* = 5.1, 3.7 Hz, 1H), 4.32 (s, 3H). Spectra matched literature values.[6]



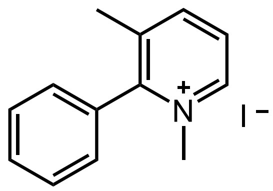
1-methyl-2-(1-methyl-1H-indol-2-yl)pyridin-1-ium iodide (19a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (0.43 g, 72% yield). **^{1}H NMR** (400 MHz, DMSO-*d*6) δ 9.27 (d, J = 6.2 Hz, 1H), 8.70 (t, J = 7.9 Hz, 1H), 8.32 – 8.27 (m, 1H), 8.27 – 8.20 (m, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 8.4 Hz, 1H), 7.42 – 7.34 (m, 1H), 7.21 (t, J = 7.5 Hz, 1H), 7.12 (s, 1H), 4.27 (s, 3H), 3.73 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*6) δ 148.3, 146.9, 145.4, 138.7, 131.6, 129.3, 127.7, 127.0, 124.4, 122.0, 121.1, 111.4, 107.8, 47.7, 31.8. **HRMS (ESI)**: call for $C_{15}H_{15}N_2$ [M-I]$^+$: 223.1230. Found:223.1234.



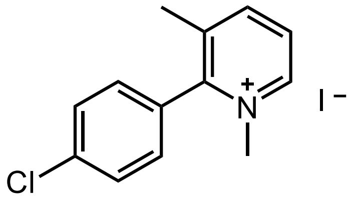
2-(4-methoxyphenyl)-1,3-dimethylpyridin-1-ium iodide (20a): The substance was prepared using **General Procedure A and B.** Brown solid (1.39 g, 82% yield). **^{1}H NMR** (400 MHz, DMSO-*d*6) δ 9.02 (d, *J* = 7.5 Hz, 1H), 8.56 (d, *J* = 7.9 Hz, 1H), 8.06 (dd, *J* = 8.0, 6.1 Hz, 1H), 7.53 – 7.44 (m, 2H), 7.25 – 7.17 (m, 2H), 3.95 (s, 3H), 3.86 (s, 3H), 2.17 (s, 3H). Spectra matched literature values.[9]



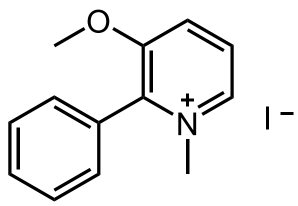
1,3-dimethyl-2-(p-tolyl)pyridin-1-ium iodide (21a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (1.46 g, 90% yield). **^{1}H NMR** (400 MHz, DMSO-*d*6) δ 9.03 (d, *J* = 6.1 Hz, 1H), 8.58 (d, *J* = 7.3 Hz, 1H), 8.08 (dd, *J* = 8.0, 6.1 Hz, 1H), 7.54 – 7.38 (m, 4H), 3.93 (s, 3H), 2.43 (s, 3H), 2.16 (s, 3H). Spectra matched literature values.[9]



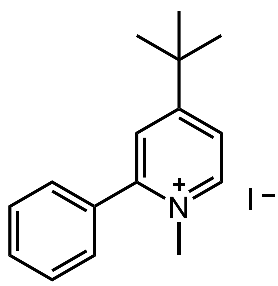
1,3-dimethyl-2-phenylpyridin-1-ium iodide (22a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.14 g, 73% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 9.05 (d, *J* = 6.2 Hz, 1H), 8.59 (d, *J* = 8.0 Hz, 1H), 8.10 (t, *J* = 7.1 Hz, 1H), 7.72 – 7.61 (m, 3H), 7.62 – 7.53 (m, 2H), 3.94 (s, 3H), 2.15 (s, 3H). Spectra matched literature values.[9]



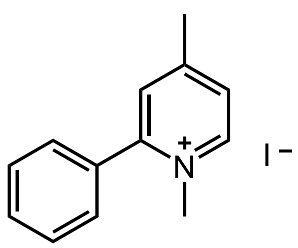
2-(4-chlorophenyl)-1,3-dimethylpyridin-1-ium iodide (23a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.28 g, 74% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 9.05 (d, *J* = 6.2 Hz, 1H), 8.60 (d, *J* = 8.0 Hz, 1H), 8.11 (dd, *J* = 8.0, 6.1 Hz, 1H), 7.81 – 7.72 (m, 2H), 7.66 – 7.58 (m, 2H), 3.94 (s, 3H), 2.16 (s, 3H). Spectra matched literature values.[9]



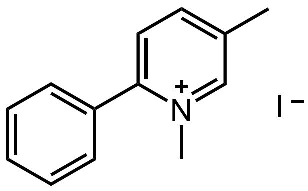
3-methoxy-1-methyl-2-phenylpyridin-1-ium iodide (24a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.21 g, 74% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 8.78 (d, *J* = 5.5 Hz, 1H), 8.39 (d, *J* = 7.9 Hz, 1H), 8.15 (dd, *J* = 8.8, 6.1 Hz, 1H), 7.69 – 7.59 (m, 3H), 7.60 – 7.52 (m, 2H), 3.97 (s, 3H), 3.90 (s, 3H). Spectra matched literature values.[9]



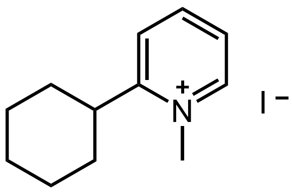
4-(tert-butyl)-1-methyl-2-phenylpyridin-1-ium iodide (25a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.43 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 9.05 (d, *J* = 6.7 Hz, 1H), 8.21 (dd, *J* = 6.6, 2.4 Hz, 1H), 8.00 (d, *J* = 2.3 Hz, 1H), 7.75 – 7.67 (m, 3H), 7.70 – 7.61 (m, 4H), 4.07 (s, 3H), 1.40 (s, 9H). Spectra matched literature values.[9]



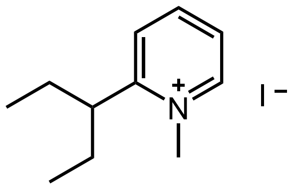
1,4-dimethyl-2-phenylpyridin-1-ium iodide (26a): The sub-stance was prepared using **General Procedure A and B.** White solid (1.03 g, 66% yield). **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ 8.98 (d, *J* = 6.4 Hz, 1H), 8.00 (dd, *J* = 6.4, 2.0 Hz, 1H), 7.96 (d, *J* = 2.1 Hz, 1H), 7.71 – 7.61 (m, 5H), 4.05 (s, 3H), 3.34 (s, 3H). Spectra matched literature values.[9]



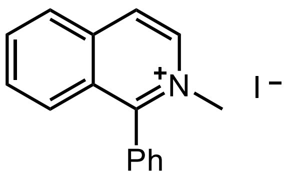
1,5-dimethyl-2-phenylpyridin-1-ium iodide (27a): The sub-stance was prepared using **General Procedure A and B.** Light yellow solid (1.07 g, 69% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.11 (s, 1H), 8.50 (d, *J* = 8.2 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.70 – 7.60 (m, 5H), 4.09 (s, 3H), 2.55 (s, 3H). Spectra matched literature values.[9]



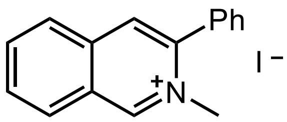
2-cyclohexyl-1-methylpyridin-1-ium iodide (28a): The substance was prepared using **General Procedure B.** White solid (1.12 g, 74% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.94 (d, *J* = 6.3 Hz, 1H), 8.54 – 8.49 (m, 1H), 8.10 (d, *J* = 8.2 Hz, 1H), 7.93 (ddd, *J* = 7.8, 6.2, 1.5 Hz, 1H), 4.34 (s, 3H), 3.22 – 3.15 (m, 1H), 1.94 (d, *J* = 11.5 Hz, 2H), 1.83 (dd, *J* = 12.3, 3.1 Hz, 2H), 1.79 – 1.69 (m, 1H), 1.61 – 1.38 (m, 4H), 1.38 – 1.23 (m, 1H). Spectra matched literature values.[6]



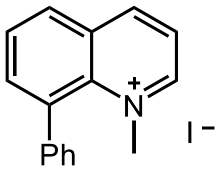
1-methyl-2-(pentan-3-yl)pyridin-1-ium iodide (29a): The substance was prepared using **General Procedure B.** White solid (1.28 g, 88% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.99 (d, *J* = 6.3 Hz, 1H), 8.54 (t, *J* = 7.9 Hz, 1H), 8.11 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.97 (ddd, *J* = 7.7, 6.2, 1.5 Hz, 1H), 4.34 (s, 3H), 3.31 – 3.24 (m, 1H), 1.86 – 1.68 (m, 4H), 0.81 (t, *J* = 7.4 Hz, 6H). Spectra matched literature values.[6]



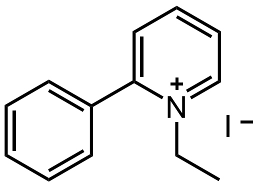
2-methyl-1-phenylisoquinolin-2-ium iodide (30a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (1.56 g, 90% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.94 (d, *J* = 6.9 Hz, 1H), 8.69 (d, *J* = 6.8 Hz, 1H), 8.43 (d, *J* = 8.2 Hz, 1H), 8.24 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 7.94 (ddd, *J* = 8.4, 7.0, 1.2 Hz, 1H), 7.82 – 7.70 (m, 5H), 7.62 (d, *J* = 8.6 Hz, 1H), 4.11 (s, 3H). Spectra matched literature values.[10]



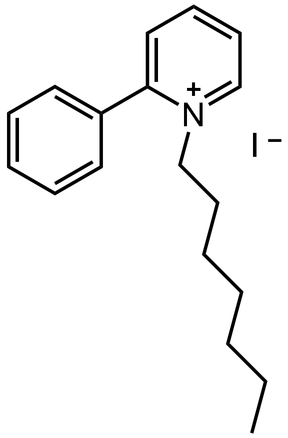
2-methyl-3-phenylisoquinolin-2-ium iodide (31a): The sub-stance was prepared using **General Procedure A and B.** Yellow solid (1.38 g, 80% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.24 (s, 1H), 8.57 (s, 1H), 8.54 (d, *J* = 8.4 Hz, 1H), 8.35 (d, *J* = 8.3 Hz, 1H), 8.33 – 8.24 (m, 1H), 8.10 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.78 – 7.69 (m, 2H), 7.70 – 7.65 (m, 3H), 4.22 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 152.3, 146.2, 137.9, 137.5, 132.7, 131.7, 131.0, 130.4, 130.3, 129.5, 127.6, 127.2, 127.0, 47.9. **HRMS (ESI)**: call for $C_{16}H_{14}N$ [M-I]$^+$: 220.1121. Found: 220.1116.



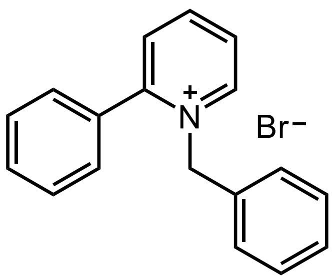
1-methyl-8-phenylquinolin-1-ium iodide (32a): The substance was prepared using **General Procedure A and B.** Yellow solid (1.41 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.44 – 9.36 (m, 2H), 8.54 (dd, *J* = 7.4, 2.4 Hz, 1H), 8.22 (dd, *J* = 8.2, 5.9 Hz, 1H), 8.12 – 7.99 (m, 2H), 7.57 – 7.53(m, 5H), 3.95 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 153.3, 148.6, 140.4, 140.0, 137.4, 134.4, 131.3, 130.0, 129.3, 129.1, 129.0, 122.5, 51.8. **HRMS (ESI)**: call for $C_{16}H_{14}N$ [M-I]$^+$: 220.1121. Found: 220.1113.



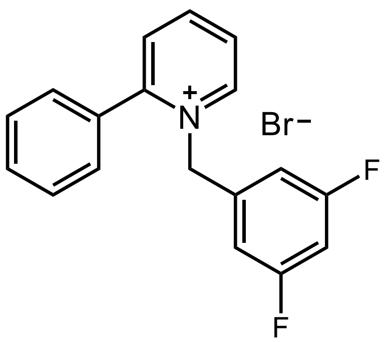
1-ethyl-2-phenylpyridin-1-ium iodide (33a): The substance was prepared using **General Procedure B.** Brown solid (1.35 g, 87% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.25 (dd, *J* = 6.3, 1.4 Hz, 1H), 8.65 (td, *J* = 7.8, 1.4 Hz, 1H), 8.22 (ddd, *J* = 7.8, 6.2, 1.5 Hz, 1H), 8.06 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.72 – 7.64 (m, 5H), 4.46 (q, *J* = 7.3 Hz, 2H), 1.35 (t, *J* = 7.3 Hz, 3H). Spectra matched literature values.[2]



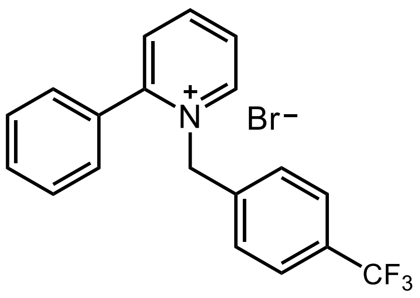
1-heptyl-2-phenylpyridin-1-ium iodide (34a): The substance was prepared using **General Procedure B.** Yellow solid (0.98 g, 51% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.25 (dd, J = 6.3, 1.4 Hz, 1H), 8.66 (td, J = 7.9, 1.4 Hz, 1H), 8.22 (ddd, J = 7.8, 6.2, 1.6 Hz, 1H), 8.08 (dd, J = 7.9, 1.5 Hz, 1H), 7.74 – 7.61 (m, 5H), 4.51 – 4.38 (m, 2H), 1.82 – 1.58 (m, 2H), 1.19 – 0.99 (m, 8H), 0.79 (t, J = 7.1 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 155.2, 146.2, 146.0, 132.2, 131.5, 130.9, 129.6, 129.5, 127.7, 58.4, 31.2, 30.3, 28.1, 25.7, 22.3, 14.3. **HRMS (ESI)**: call for $C_{18}H_{24}N$ [M-I]$^+$: 254.1903. Found: 254.1902.



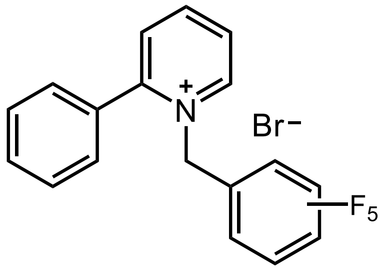
1-benzyl-2-phenylpyridin-1-ium bromide (35a): The sub-stance was prepared using **General Procedure B.** White solid (1.38 g, 85% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.37 (dd, J = 6.4, 1.4 Hz, 1H), 8.75 (td, J = 7.8, 1.4 Hz, 1H), 8.28 (ddd, J = 7.8, 6.2, 1.6 Hz, 1H), 8.12 (dd, J = 8.0, 1.5 Hz, 1H), 7.63 (ddt, J = 7.8, 6.0, 1.9 Hz, 1H), 7.59 – 7.50 (m, 4H), 7.34 – 7.22 (m, 3H), 6.96 – 6.84 (m, 2H), 5.83 (s, 2H). Spectra matched literature values.[2]



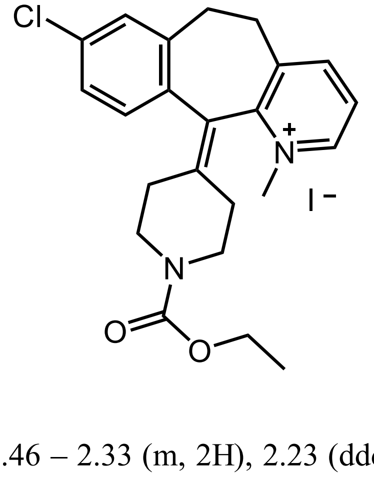
1-(3,5-difluorobenzyl)-2-phenylpyridin-1-ium bromide (36a): The substance was prepared using **General Procedure B.** White solid (1.35g, 75% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.39 – 9.36 (m, 1H), 8.76 (td, J = 7.9, 1.4 Hz, 1H), 8.28 (ddd, J = 7.7, 6.1, 1.5 Hz, 1H), 8.12 (dd, J = 8.0, 1.5 Hz, 1H), 7.62 (ddt, J = 8.2, 6.2, 1.7 Hz, 1H), 7.58 – 7.50 (m, 4H), 7.20 (tt, J = 9.3, 2.4 Hz, 1H), 6.68 (dt, J = 6.6, 2.1 Hz, 2H), 5.87 (s, 2H). **^{19}F NMR** (376 MHz, DMSO-d_6) δ -108.95. **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 162.8 (dd, J_{CF} = 242.4 Hz, 10.1 Hz), 155.4, 147.4, 147.1, 138.2 (t, J_{CF} = 9.9 Hz), 132.1, 131.4, 131.3, 129.4, 128.1, 111.9 (d, J_{CF} = 10.1 Hz), 111.8 (d, J_{CF} = 10.1 Hz), 104.6 (t, J_{CF} = 25.7 Hz), 60.8. **HRMS (ESI)**: call for $C_{18}H_{14}F_2N$ [M-Br]$^+$: 282.1089. Found: 282.1083.



2-phenyl-1-(4-(trifluoromethyl)benzyl)pyridin-1-ium bromide (37a): The substance was prepared using **General Procedure B.** White solid (1.41 g, 72% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.39 (d, *J* = 6.3 Hz, 1H), 8.78 (td, *J* = 7.9, 1.4 Hz, 1H), 8.30 (ddd, *J* = 7.8, 6.2, 1.6 Hz, 1H), 8.15 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.66 – 7.59 (m, 3H), 7.56 – 7.49 (m, 4H), 7.13 (d, *J* = 8.1 Hz, 2H), 5.95 (s, 2H). **^{19}F NMR** (376 MHz, DMSO-d_6) δ -61.18. **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 155.6, 147.3, 147.2, 138.9, 132.0, 131.5, 131.4, 129.4, 129.3 (q, J_{CF} = 30.3 Hz), 128.9, 128.1, 126.0 (q, J_{CF} = 3.7 Hz), 124.4 ((q, J_{CF} = 272.7 Hz)), 61.1. **HRMS (ESI)**: call for $C_{19}H_{15}F_3N$ $[M\text{-}Br]^+$: 314.1151. Found: 314.1140.

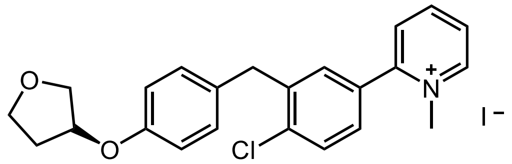


1-((perfluorophenyl)methyl)-2-phenylpyridin-1-ium bromide (38a): The substance was prepared using **General Procedure B.** Light yellow solid (1.31 g, 63% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.41 (d, *J* = 6.2 Hz, 1H), 8.77 (td, *J* = 7.9, 1.4 Hz, 1H), 8.29 (ddd, *J* = 7.8, 6.2, 1.6 Hz, 1H), 8.11 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.68 – 7.63 (m, 1H), 7.59 (d, *J* = 4.1 Hz, 4H). **^{19}F NMR** (376 MHz, DMSO-d_6) δ -140.18 – -140.32 (m), -152.33 (t, *J* = 22.4 Hz), -161.89 – -162.14 (m). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 155.6, 147.7, 147.6, 131.7, 131.6, 131.4, 129.5, 129.2, 127.8, 51.4. **HRMS (ESI)**: call for $C_{18}H_{11}F_5N^+$ $[M\text{-}Br]^+$: 336.0806. Found: 336.0799.



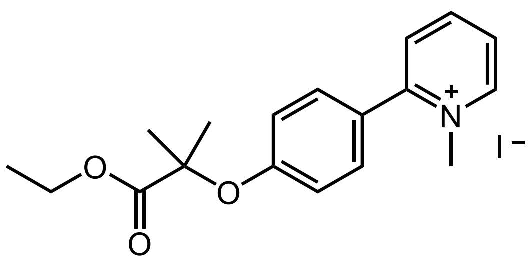
dihydro-5H-benzo[5,6]cyclohepta[1,2-b]pyridin-1-ium iodide (39a): Yellow solid (2.32 g, 89% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.85 (d, J = 6.2 Hz, 1H), 8.57 (d, J = 7.8 Hz, 1H), 8.01 (dd, J = 7.9, 6.2 Hz, 1H), 7.45 – 7.26 (m, 3H), 4.19 (s, 3H), 4.05 (q, J = 7.0 Hz, 2H), 3.83 – 3.78 (m, 1H), 3.72 – 3.68 (m, 1H), 3.49 – 3.38 (m, 2H), 3.19 – 3.08 (m, 3H), 2.99 – 2.82 (m, 1H), 2.46 – 2.33 (m, 2H), 2.23 (ddd, J = 14.2, 9.5, 4.8 Hz, 1H), 1.89 (dt, J = 14.2, 4.4 Hz, 1H), 1.18 (t, J = 7.1 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*6) δ 155.0, 154.3, 145.2, 144.8, 144.3, 140.3, 139.5, 133.7, 132.9, 131.7, 131.2, 126.8, 126.7, 123.7, 61.4, 46.0, 43.9, 43.6, 31.5, 29.3, 15.1. **HRMS (ESI)**: call for $C_{23}H_{26}ClN_2O_2$ [M-I]$^+$: 397.1677. Found: 397.1669.

(R)-2-(4-chloro-3-(4-((tetrahydrofuran-3-yl)oxy)benzyl)phenyl)-1-



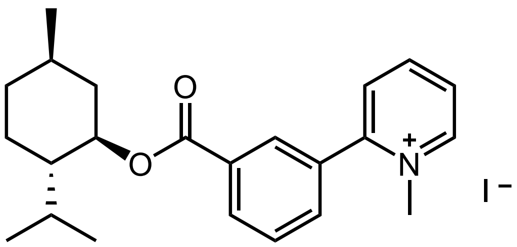
methylpyridin-1-ium iodide (40a): Gray solid (1.54 g, 64% yield). **^{1}H NMR** (400 MHz, DMSO-*d*6) δ 9.14 (d, *J* = 6.4 Hz, 1H), 8.64 (t, *J* = 7.9 Hz, 1H), 8.17 (ddd, *J* = 7.8, 6.1, 1.6 Hz, 1H), 8.09 (, 1 dd, *J* = 7.9, 1.6 Hz H), 7.81 – 7.70 (m, 2H), 7.60 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.18 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.96 (ddt, *J* = 6.3, 4.1, 1.8 Hz, 1H), 4.11 (d, *J* = 12.7 Hz, 5H), 3.89 – 3.69 (m, 4H), 2.30 – 2.13 (m, 1H), 1.98 – 1.86 (m, 1H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*6) δ 156.1, 154.3, 147.3, 146.0, 140.2, 136.6, 132.5, 131.3, 131.2, 130.6, 130.4, 130.3, 129.5, 127.4, 115.8, 77.4, 72.7, 66.9, 47.7, 38.0, 32.9. **HRMS (ESI)**: call for $C_{23}H_{23}ClNO_2$ [M-I]$^+$: 380.1412. **Found**: 380.1380.

2-(4-((1-ethoxy-2-methyl-1-oxopropan-2-yl)oxy)phenyl)-1-methylpyridin-1-ium



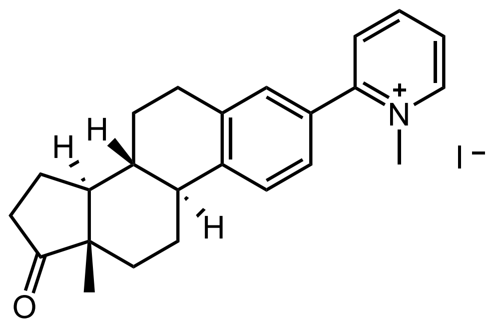
iodide (41a): Yellow solid (1.92 g, 80% yield). **^{1}H NMR** (400 MHz, DMSO-*d*6) δ 9.11 (d, J = 4.8 Hz, 1H), 8.60 (td, J = 7.8, 1.5 Hz, 1H), 8.12 (ddd, J = 7.8, 6.2, 1.6 Hz, 1H), 8.06 (d, J = 6.5 Hz, 1H), 7.70 – 7.56 (m, 2H), 7.15 – 6.96 (m, 2H), 4.20 (q, J = 7.1 Hz, 2H), 4.14 (s, 3H), 1.62 (s, 6H), 1.17 (t, J = 7.1 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*6) δ 173.2, 157.7, 155.3, 147.1, 145.7, 131.4, 130.4, 126.7, 125.1, 118.5, 79.5, 61.8, 47.6, 25.6, 14.4. **HRMS (ESI)**: call for $C_{18}H_{22}NO_3$ [M-I]$^+$: 300.1594. **Found**: 300.1591.

2-(3-((((1R,2S,5R)-2-isopropyl-5-methyl-cyclohexyl)oxy)carbonyl)phenyl)-1-methyl-pyridin-1-ium iodide (42a):

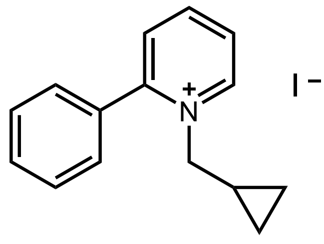


Yellow solid (1.92 g, 80% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.17 (d, *J* = 5.2 Hz, 1H), 8.67 (td, *J* = 7.9, 1.4 Hz, 1H), 8.26 – 8.23 (m, 1H), 8.23 – 8.17 (m, 2H), 8.14 (d, *J* = 8.0 Hz, 1H), 8.00 – 7.98 (m, 1H), 7.82 (t, *J* = 7.8 Hz, 1H), 4.90 (td, *J* = 10.8, 4.3 Hz, 1H), 4.10 (s, 3H), 2.07 – 1.95 (m, 1H), 1.89 (td, *J* = 6.9, 2.7 Hz, 1H), 1.74 – 1.61 (m, 2H), 1.58 – 1.52 (m, 2H), 1.17 – 1.08 (m, 2H), 0.89 (dd, *J* = 8.6, 6.7 Hz, 7H), 0.76 (d, *J* = 6.9 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 164.9, 154.4, 147.2, 146.0, 134.3, 132.9, 131.9, 131.2, 130.4, 130.4, 130.2, 127.5, 75.2, 47.7, 47.0, 41.0, 34.2, 31.3, 26.6, 23.6, 22.4, 21.0, 16.9. **HRMS (ESI)**: call for $C_{23}H_{30}NO_2$ [M-I]$^+$: 352.2271. **Found**: 352.2262.

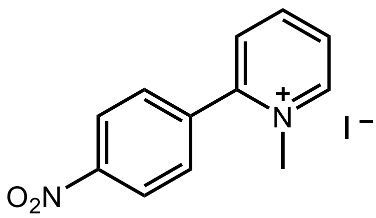
1-methyl-2-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)pyridin-1-ium iodide (43a):



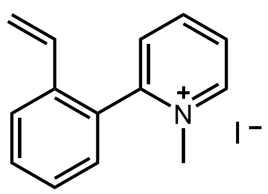
Yellow solid (1.68 g, 71% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.13 (d, J = 5.3 Hz, 1H), 8.63 (t, J = 7.9 Hz, 1H), 8.15 (t, J = 7.0 Hz, 1H), 8.05 (d, J = 9.6 Hz, 1H), 7.57 (d, J = 8.2 Hz, 1H), 7.46 (dd, J = 8.2, 2.0 Hz, 1H), 7.42 (s, 1H), 4.16 (s, 3H), 2.97 (dd, J = 9.1, 4.2 Hz, 2H), 2.50 – 2.35 (m, 3H), 2.15 – 1.96 (m, 3H), 1.86 – 1.77 (m, 1H), 1.68 – 1.38 (m, 6H), 0.87 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 155.6, 147.1, 145.8, 143.4, 137.8, 130.3, 130.0, 129.7, 126.9, 126.5, 50.1, 47.7, 47.6, 44.4, 37.7, 35.9, 31.8, 29.3, 26.1, 25.6, 21.6, 14.0. **HRMS (ESI)**: call for $C_{24}H_{28}NO$ [M-I]$^+$: 346.2165. **Found**: 346.2161.



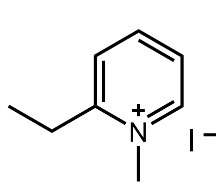
1-(cyclopropylmethyl)-2-phenylpyridin-1-ium iodide (1aa): Yellow solid (1.03 g, 61% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.29 (dd, *J* = 6.4, 1.4 Hz, 1H), 8.69 (td, *J* = 7.8, 1.5 Hz, 1H), 8.24 (ddd, *J* = 7.8, 6.2, 1.6 Hz, 1H), 8.09 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.73 – 7.71 (m, 2H), 7.69 – 7.64 (m, 3H), 4.37 (d, *J* = 7.3 Hz, 2H), 1.13 – 1.07 (m, 1H), 0.59 – 0.49 (m, 2H), 0.39 (dt, *J* = 6.5, 4.5 Hz, 2H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 155.1, 146.1, 145.8, 132.3, 131.5, 130.9, 129.6, 129.6, 127.7, 62.8, 11.7, 4.7. **HRMS (ESI)**: call for $C_{15}H_{16}N^+$ [M-Br]$^+$: 210.1277. Found: 210.1275.



1-methyl-2-(4-nitrophenyl)pyridin-1-ium iodide (1ab): The substance was prepared using **General Procedure A and B.** Yellow solid (1.39 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.21 (dd, *J* = 6.3, 1.4 Hz, 1H), 8.71 (td, *J* = 7.9, 1.5 Hz, 1H), 8.63 (t, *J* = 2.0 Hz, 1H), 8.52 (ddd, *J* = 8.3, 2.3, 1.1 Hz, 1H), 8.24 (ddd, *J* = 7.8, 6.2, 1.6 Hz, 1H), 8.21 – 8.12 (m, 2H), 7.97 (t, *J* = 8.0 Hz, 1H), 4.13 (s, 3H). Spectra matched literature values.[11]

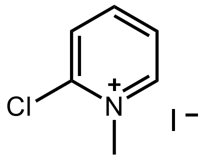


1-methyl-2-(2-vinylphenyl)pyridin-1-ium iodide (1ac): The substance was prepared using **General Procedure A and B.** Yellow solid (1.21 g, 75% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.21 (d, *J* = 6.1 Hz, 1H), 8.67 (t, *J* = 7.6 Hz, 1H), 8.23 (t, *J* = 6.7 Hz, 1H), 8.06 (d, *J* = 7.8 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H), 7.81 – 7.64 (m, 1H), 7.62 – 7.50 (m, 2H), 6.35 (dd, *J* = 17.2, 11.0 Hz, 1H), 5.89 (d, *J* = 17.2 Hz, 1H), 5.36 (d, *J* = 11.0 Hz, 1H), 3.96 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 154.3, 147.7, 146.0, 136.2, 133.0, 131.9, 130.7, 130.4, 129.9, 129.0, 127.6, 126.5, 119.5, 47.1. **HRMS (ESI)**: call for $C_{14}H_{14}N$ [M-I]$^+$: 196.1121. Found: 196.1106.

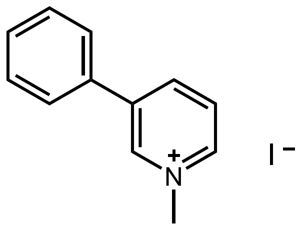


2-ethyl-1-methylpyridin-1-ium iodide (1ad): The substance was prepared using **General Procedure B.** Brown solid (0.78 g, 63% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.97 (d, *J* = 6.2 Hz, 1H), 8.61 (t, *J* = 7.9 Hz, 1H), 8.22 (d, *J* = 8.1 Hz, 1H), 8.10

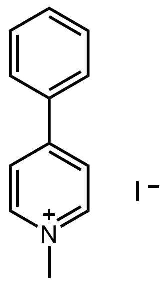
– 7.88 (m, 1H), 6.17 (d, *J* = 5.0 Hz, 1H), 5.41 – 5.08 (m, 1H), 4.35 (s, 3H), 1.49 (d, *J* = 6.4 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 161.7, 147.0, 146.3, 126.5, 125.7, 64.2, 45.7, 22.5. **HRMS (ESI)**: call for $C_8H_{12}N$ [M-I]$^+$: 122.0964. Found: 122.0961.



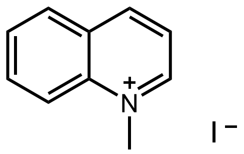
2-chloro-1-methylpyridin-1-ium iodide (1ae): The substance was prepared using **General Procedure B.** Yellow solid (0.76 g, 60% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.24 (dd, *J* = 6.2, 1.7 Hz, 1H), 8.65 (dd, *J* = 8.0, 1.6 Hz, 1H), 8.15 (td, *J* = 7.8, 1.7 Hz, 1H), 8.07 (ddd, *J* = 7.7, 6.1, 1.6 Hz, 1H), 4.41 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 148.6, 144.5, 140.4, 127.3, 55.2. **HRMS (ESI)**: call for C_6H_7ClN [M-I]$^+$: 128.0262. Found: 128.0262.



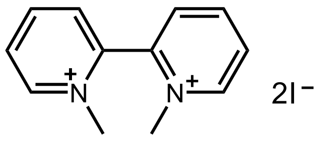
1-methyl-3-phenylpyridin-1-ium iodide (1af): The substance was prepared using **General Procedure B.** Yellow solid (1.27 g, 86% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.43 (s, 1H), 8.98 (d, *J* = 6.0 Hz, 1H), 8.91 (d, *J* = 8.3 Hz, 1H), 8.22 (dd, *J* = 8.2, 5.9 Hz, 1H), 7.97 – 7.84 (m, 2H), 7.68 – 7.49 (m, 3H), 4.44 (s, 3H). Spectra matched literature values.[6]



1-methyl-4-phenylpyridin-1-ium iodide (1ag): The substance was prepared using **General Procedure B.** Brown solid (1.32 g, 89% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.04 (d, *J* = 6.7 Hz, 2H), 8.53 (d, *J* = 6.8 Hz, 2H), 8.12 – 8.02 (m, 2H), 7.73 – 7.60 (m, 3H), 4.36 (s, 3H). Spectra matched literature values.[6]

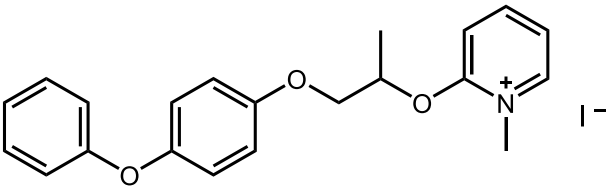


1-methylquinolin-1-ium iodide (1ah): The substance was prepared using **General Procedure B.** Green solid (1.14 g, 84% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.53 (d, *J* = 5.7 Hz, 1H), 9.30 (d, *J* = 8.4 Hz, 1H), 8.52 (dd, *J* = 12.2, 8.6 Hz, 2H), 8.31 (ddd, *J* = 8.8, 7.0, 1.5 Hz, 1H), 8.19 (dd, *J* = 8.4, 5.7 Hz, 1H), 8.11 – 8.03 (m, 1H), 4.65 (s, 3H). Spectra matched literature values.[6]

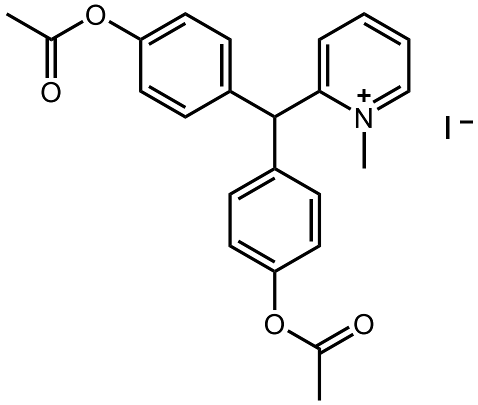


1,1'-dimethyl-[2,2'-bipyridine]-1,1'-diium (1ai): The substance was prepared using **General Procedure B.** Yellow solid (1.41 g, 64% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.44 (d, J = 6.1 Hz, 2H), 8.91 (t, J = 7.9 Hz, 2H), 8.55 (d, J = 7.9 Hz, 2H), 8.49 (t, J = 7.1 Hz, 2H), 4.17 (s, 6H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 149.6, 147.1, 143.5, 131.1, 130.6, 48.2. **HRMS (ESI)**: call for $C_{12}H_{14}N_2$ $[M-2I]^{2+}$: 186.1146. Found: 186.1148.

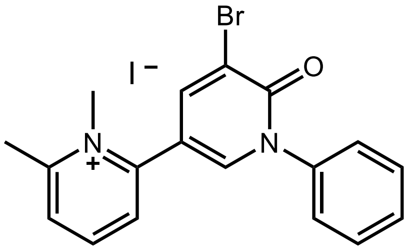
1-methyl-2-((1-(4-phenoxyphenoxy)-propan-2-yl)oxy)pyridin-1-ium iodide (1aj):



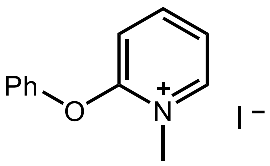
The substance was prepared using **General Procedure B.** Yellow solid (1.81 g, 78% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.70 (d, J = 6.3 Hz, 1H), 8.52 (t, J = 8.3 Hz, 1H), 7.96 (d, J = 8.9 Hz, 1H), 7.58 (t, J = 6.8 Hz, 1H), 7.36 (t, J = 7.7 Hz, 2H), 7.11 – 6.87 (m, 7H), 5.60 – 5.48 (m, 1H), 4.44 – 4.25 (m, 2H), 3.96 (s, 3H), 1.55 (d, J = 6.2 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 159.5, 158.3, 154.7, 150.4, 148.0, 144.4, 130.4, 123.2, 121.2, 119.0, 117.9, 116.5, 113.1, 79.3, 70.9, 42.0, 16.4. **HRMS (ESI)**: call for $C_{21}H_{22}NO_3$ $[M-I]^+$: 336.1594. **Found**: 336.1598.



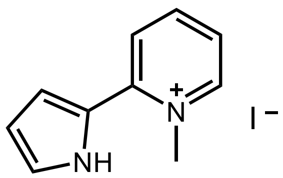
2-(bis(4-acetoxyphenyl)methyl)-1-methyl-pyridin-1-ium iodide (1ak): The substance was prepared using **General Procedure B.** Yellow solid (2.04 g, 81% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.08 (d, J = 6.2 Hz, 1H), 8.55 (t, J = 7.9 Hz, 1H), 8.08 (t, J = 7.0 Hz, 1H), 7.51 (d, J = 8.0 Hz, 1H), 7.32 – 7.18 (m, 8H), 6.44 (s, 1H), 4.25 (s, 3H), 2.28 (s, 6H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 169.5, 158.5, 150.5, 148.5, 146.3, 135.8, 130.9, 129.3, 126.6, 123.1, 51.0, 46.5, 21.4. **HRMS (ESI)**: call for $C_{23}H_{22}NO_4$ $[M-I]^+$: 376.1543. Found: 376.1548.



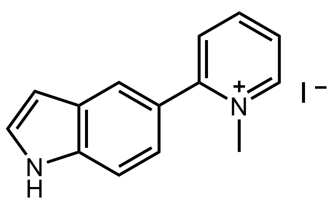
5'-bromo-1-methyl-6'-oxo-1'-phenyl-1',6'-dihydro-[2,3'-bipyridin]-1-ium iodide (1al): The substance was prepared using **General Procedure B.** Yellow solid (1.36 g, 58% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.13 (d, *J* = 6.1 Hz, 1H), 8.64 (t, *J* = 8.6 Hz, 1H), 8.42 (d, *J* = 2.4 Hz, 1H), 8.27 (d, *J* = 2.4 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 1H), 8.15 (t, *J* = 7.0 Hz, 1H), 7.61 – 7.51 (m, 5H), 4.33 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 157.6, 150.8, 147.2, 145.8, 142.3, 142.1, 140.6, 131.0, 129.7, 129.5, 127.4, 127.3, 116.0, 110.9, 47.4. **HRMS (ESI)**: call for $C_{17}H_{14}BrN_2O$ [M-I]$^+$: 341.0284. Found: 341.0285.



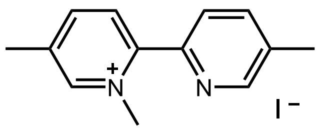
1-methyl-2-phenoxypyridin-1-ium iodide (1am): The substance was prepared using **General Procedure B.** White solid (1.25g, 80%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.86 (d, J = 6.3 Hz, 1H), 8.41 (t, J = 8.1 Hz, 1H), 7.67 (dt, J = 23.9, 7.3 Hz, 6H), 7.56 – 7.41 (m, 5H), 7.14 (d, J = 8.8 Hz, 2H), 4.23 (s, 5H). Spectra matched literature values.[6]



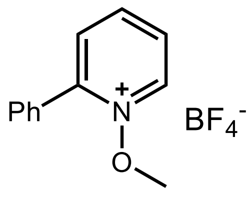
1-methyl-2-(1H-pyrrol-2-yl)pyridin-1-ium iodide (1ao): The substance was prepared using **General Procedure B.** Yellow solid (0.12 g, 84%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 12.21 (s, 1H), 8.90 (dd, *J* = 6.4, 1.5 Hz, 1H), 8.47 (ddd, *J* = 8.8, 7.5, 1.5 Hz, 1H), 8.14 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.81 (ddd, *J* = 7.6, 6.3, 1.5 Hz, 1H), 7.42 (dd, *J* = 2.5, 1.4 Hz, 1H), 7.13 (dd, *J* = 3.9, 1.4 Hz, 1H), 6.48 (dd, *J* = 3.9, 2.6 Hz, 1H), 4.37 (s, 3H). Spectra matched literature values.[12]



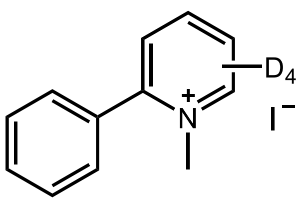
2-(1H-indol-5-yl)-1-methylpyridin-1-ium iodide (1ap): The substance was prepared using **General Procedure B.** Yellow solid (1.2 g, 89%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 11.57 (s, 1H), 9.12 (d, J = 6.1 Hz, 1H), 8.60 (t, J = 7.8 Hz, 1H), 8.11 (t, J = 6.9 Hz, 2H), 7.90 (d, J = 1.7 Hz, 1H), 7.65 (d, J = 8.5 Hz, 1H), 7.57 (t, J = 2.8 Hz, 1H), 7.39 (dd, J = 8.5, 1.8 Hz, 1H), 6.62 (t, J = 2.5 Hz, 1H), 4.19 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 157.4, 146.9, 145.3, 137.2, 130.7, 128.1, 127.9, 126.2, 122.8, 122.5, 122.1, 112.5, 102.6, 47.6. **HRMS (ESI)**: call for $C_{14}H_{13}N_2$ $[M-I]^+$: 209.1073. Found: 209.1079.



1,5,5'-trimethyl-[2,2'-bipyridin]-1-ium iodide (1aq): The substance was prepared using **General Procedure B.** Yellow solid (1.23 g, 73%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.11 (s, 1H), 8.70 (s, 1H), 8.58 – 8.51 (m, 1H), 8.16 (d, J = 8.1 Hz, 1H), 7.98 (dd, J = 8.1, 2.2 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 4.24 (s, 3H), 2.56 (s, 3H), 2.45 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 150.6, 150.4, 147.5, 147.4, 146.5, 138.6, 138.3, 136.2, 129.5, 126.1, 47.5, 18.4, 18.1. **HRMS (ESI)**: call for $C_{13}H_{15}N_2$ $[M-I]^+$: 199.1230. Found: 199.1231.



1-methoxy-2-phenylpyridin-1-ium tetrafluoroborate (1ar): White solid (0.68g, 50%). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 9.58 (dd, J = 6.7, 1.3 Hz, 1H), 8.68 (td, J = 7.8, 1.3 Hz, 1H), 8.32 (dd, J = 8.0, 1.8 Hz, 1H), 8.27 (ddd, J = 7.7, 6.6, 1.9 Hz, 1H), 7.87 – 7.83 (m, 2H), 7.75 – 7.65 (m, 3H), 4.09 (s, 3H). Spectra matched literature values.[12]



1-methyl-2-phenylpyridin-1-ium-3,4,5,6-d4 iodide (1a - D*4*): The substance was prepared using **General Procedure C.** Light yellow solid (1.21 g, 80% yield). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.71 - 7.63 (m, 5H), 4.13 (s, 3H). Spectra matched literature values. [11]

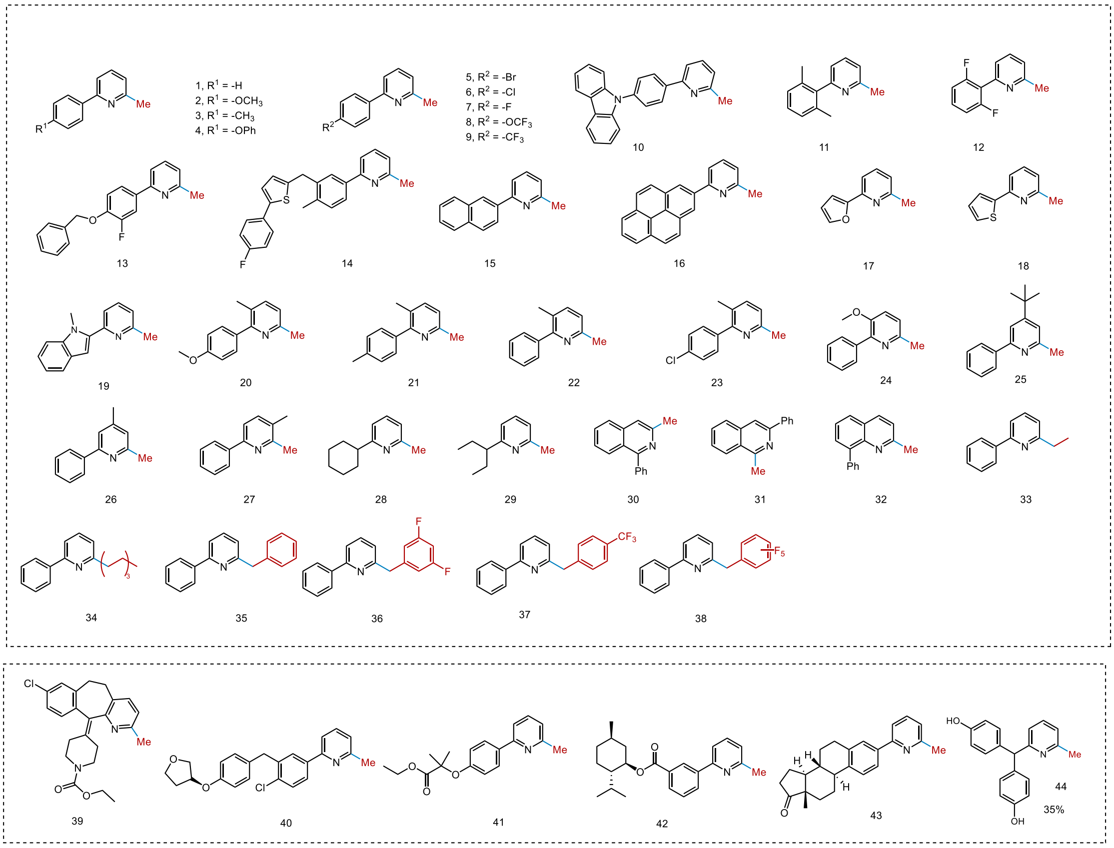
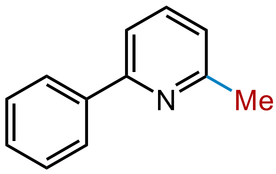
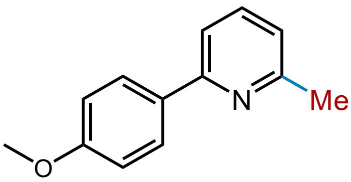


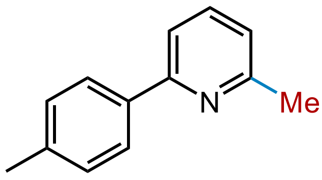
Figure S7. Characterization data of the products



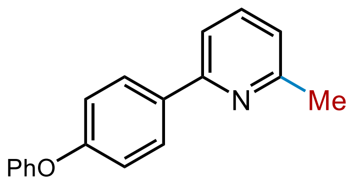
2-methyl-6-phenylpyridine (1): A colorless oil (36.9 mg, 87% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.03 – 7.93 (m, 2H), 7.63 (t, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.43 – 7.37 (m, 1H), 7.10 (d, *J* = 7.6 Hz, 1H), 2.64 (s, 3H). Spectra matched literature values.[13]



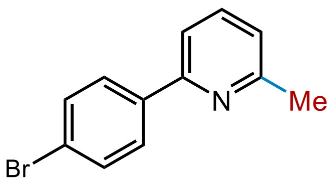
2-(4-methoxyphenyl)-6-methylpyridine (2): A pale yellow solid (36.9 mg, 74% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.98 – 7.89 (m, 2H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.45 (d, *J* = 7.8 Hz, 1H), 7.03 (d, *J* = 7.5 Hz, 1H), 7.01 – 6.96 (m, 2H), 3.86 (s, 3H), 2.61 (s, 3H). Spectra matched literature values.[14]



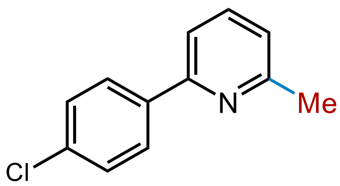
2-methyl-6-(p-tolyl)pyridine (3): A colorless oil (24.1 mg, 54% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.92 – 7.85 (m, 2H), 7.61 (t, *J* = 7.7 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.06 (d, *J* = 7.6 Hz, 1H), 2.62 (s, 3H), 2.41 (s, 3H). Spectra matched literature values.[14]



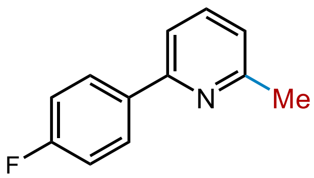
2-methyl-6-(4-phenoxyphenyl)pyridine (4): A colorless oil (29.1 mg, 46% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 8.00 – 7.94 (m, 2H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.41 – 7.31 (m, 2H), 7.16 – 7.02 (m, 6H), 2.63 (s, 3H). Spectra matched literature values.[14]



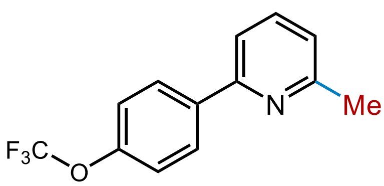
2-(4-bromophenyl)-6-methylpyridine (5): A white solid (25.0 mg, 43% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.90 – 7.84 (m, 2H), 7.66 – 7.56 (m, 3H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 2.62 (s, 3H). Spectra matched literature values.[14]



2-(4-chlorophenyl)-6-methylpyridine (6): A white solid (20.7 mg, 44% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.97 – 7.90 (m, 2H), 7.63 (t, *J* = 7.7 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.44 – 7.40 (m, 2H), 7.10 (d, *J* = 7.6 Hz, 1H), 2.62 (s, 3H). Spectra matched literature values.[14]

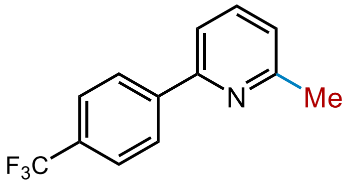


2-(4-fluorophenyl)-6-methylpyridine (7): A white solid (23.2 mg, 54% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 8.01 – 7.93 (m, 2H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.17 – 7.11 (m, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 2.62 (s, 3H). Spectra matched literature values.[14]

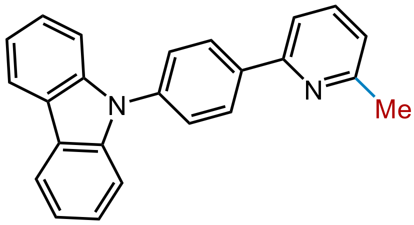


2-methyl-6-(4-(trifluoromethoxy)phenyl)pyridine (8): A white solid (20.0 mg, 32% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 8.06 – 7.96 (m, 2H), 7.64 (t, *J*

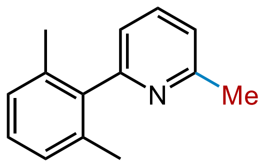
= 7.7 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 1H), 2.62 (s, 3H). Spectra matched literature values.[14]



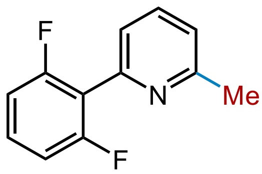
2-methyl-6-(4-(trifluoromethyl)phenyl)pyridine (9): A white solid (17.5 mg, 30% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 6.8 Hz, 2H), 7.73 – 7.67 (m, 3H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 1H), 2.64 (s, 3H). Spectra matched literature values.[13]



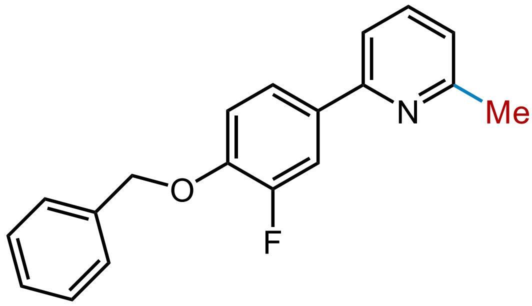
9-(4-(6-methylpyridin-2-yl)phenyl)-9H-carbazole (10): A colorless oil (21.2 mg, 46% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.22 (d, *J* = 8.7 Hz, 2H), 8.17 (d, *J* = 7.8 Hz, 2H), 7.75 – 7.65 (m, 3H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.51 – 7.38 (m, 4H), 7.31 (t, *J* = 7.3 Hz, 2H), 7.17 (d, *J* = 7.6 Hz, 1H), 2.68 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, DMSO-*d*$_6$) δ 157.6, 155.1, 139.8, 137.9, 137.1, 136.1, 127.5, 126.2, 125.0, 122.4, 120.9, 119.3, 119.0, 116.6, 108.8, 23.8. **HRMS (ESI)**: call for $C_{24}H_{18}N_2$ $[M+H]^+$: 335.1543. Found: 335.1519.



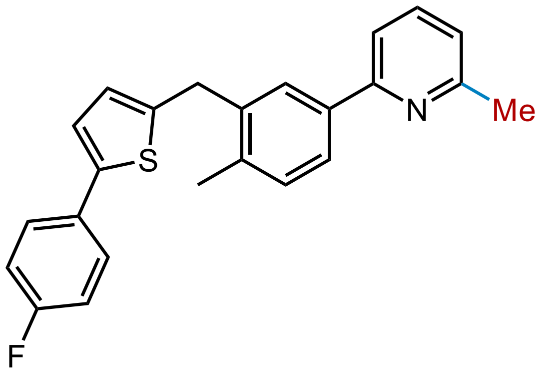
2-(2,6-dimethylphenyl)-6-methylpyridine (11): A colorless oil (23.1 mg, 47% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.64 (t, *J* = 7.7 Hz, 1H), 7.17 (dd, *J* = 8.4, 6.6 Hz, 1H), 7.12 – 7.08 (m, 3H), 7.02 (d, *J* = 7.6 Hz, 1H), 2.61 (s, 3H), 2.05 (s, 6H). Spectra matched literature values.[15]



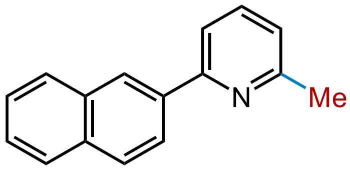
2-(2,6-difluorophenyl)-6-methylpyridine (12): A white solid (20.4 mg, 40% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.66 (t, *J* = 7.7 Hz, 1H), 7.58 – 7.49 (m, 2H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 1H), 6.83 (tt, *J* = 8.7, 2.4 Hz, 1H), 2.62 (s, 3H). **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -109.79. **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 163.4 (dd, J_{CF} = 242.4 Hz), 158.7, 154.2, 143.1 (t, *J* = 9.3 Hz), 137.2, 122.8, 117.6, 110.0 – 109.5 (m), 103.9 (t, J_{CF} = 25.6 Hz), 24.7. **HRMS (ESI)**: call for $C_{12}H_9F_2N$ $[M+H]^+$: 206.0776. Found: 206.0761.



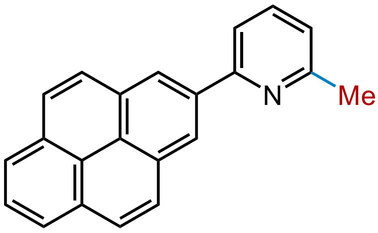
2-(4-(benzyloxy)-3-fluorophenyl)-6-methyl-pyridine (13): A white solid (42.8 mg, 58% yield). ^{1}**H NMR** (400 MHz, Chloroform-*d*) δ 7.77 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.51 – 7.47 (m, 2H), 7.41 (dd, *J* = 14.1, 7.3 Hz, 3H), 7.37 – 7.31 (m, 1H), 7.17 (dd, *J* = 10.9, 8.4 Hz, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 5.24 (s, 2H), 2.63 (s, 3H). ^{13}**C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 157.3, 154.8, 152.5(d, J_{CF} = 252.5 Hz), 145.9 (d, J_{CF} = 11.0 Hz), 135.9,135.5, 135.3 (d, J_{CF} = 3.6 Hz), 127.6, 127.1, 126.7, 120.6, 119.0 (d, J_{CF} = 7.1 Hz), 116.3, 115.3 (d, J_{CF} = 18.8 Hz), 113.3 (d, J_{CF} = 2.2 Hz), 70.3, 23.7. **HRMS (ESI)**: call for $C_{19}H_{16}FNO$ $[M+H]^+$: 294.1289. Found: 294.1287.



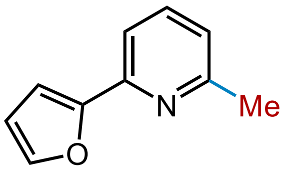
2-(3-((5-(4-fluorophenyl)thiophen-2-yl)methyl)-4-methylphenyl)-6-methylpyridine (14): A white solid (55.9 mg, 60% yield). ^{1}**H NMR** (400 MHz, DMSO-d_6) δ 8.00 (d, *J* = 2.0 Hz, 1H), 7.88 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.77 – 7.67 (m, 2H), 7.63 – 7.52 (m, 2H), 7.33 – 7.24 (m, 2H), 7.23 – 7.12 (m, 3H), 6.84 (d, *J* = 3.6 Hz, 1H), 4.23 (s, 2H), 2.52 (s, 3H), 2.33 (s, 3H). ^{19}**F NMR** (376 MHz, DMSO-d_6) δ -115.02. ^{13}**C {^{1}H} NMR** (101 MHz, DMSO-d_6) δ 161.9(d, J_{CF} = 252.5 Hz), 158.1, 155.7, 144.1,140.8, 139.1, 137.8, 137.4 (d, J_{CF} = 15.5 Hz), 131.2, 131.0 (d, J_{CF} = 3.2 Hz), 128.0, 127.4 (d, J_{CF} = 8.1 Hz), 126.9,125.4, 123.9, 122.1,117.4, 116.4, 116n.2, 34.0, 24.8, 19.3. **HRMS (ESI)**: call for $C_{24}H_{20}FNS$ $[M+H]^+$: 374.1373. Found: 374.1375.



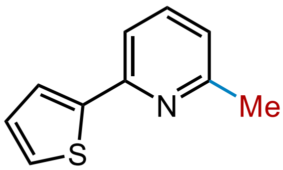
2-methyl-6-(naphthalen-2-yl)pyridine (15): A white solid (44.3 mg, 81% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.10 – 8.02 (m, 1H), 7.96 – 7.85 (m, 2H), 7.71 (t, *J* = 7.7 Hz, 1H), 7.63 – 7.51 (m, 2H), 7.52 – 7.44 (m, 2H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 7.7 Hz, 1H), 2.68 (s, 3H). Spectra matched literature values. [14]



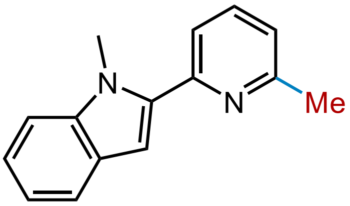
2-methyl-6-(pyren-2-yl)pyridine (16): A white solid (50.1 mg, 69% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.37 (d, *J* = 9.3 Hz, 1H), 8.26 (d, *J* = 7.9 Hz, 1H), 8.24 – 8.13 (m, 3H), 8.11 (s, 2H), 8.07 (d, *J* = 9.3 Hz, 1H), 8.02 (t, *J* = 7.6 Hz, 1H), 7.77 (t, *J* = 7.7 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.25 (d, *J* = 7.9 Hz, 1H), 2.74 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 158.9, 158.5, 136.5, 136.1, 131.4, 131.3, 131.0, 128.7, 127.9, 127.8, 127.6, 127.5, 126.0, 125.3, 125.1, 125.0, 125.0, 124.9, 124.8, 122.8, 121.5, 24.9. **HRMS (ESI)**: call for $C_{22}H_{15}N$ $[M+H]^+$: 294.1277. Found: 294.1266.



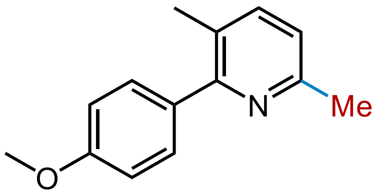
2-(furan-2-yl)-6-methylpyridine (17): A colorless oil (18.9 mg, 48% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.59 (t, *J* = 7.7 Hz, 1H), 7.52 (d, *J* = 1.0 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.05 – 6.95 (m, 2H), 6.51 (dd, *J* = 3.4, 1.8 Hz, 1H), 2.59 (s, 3H). Spectra matched literature values.[16]



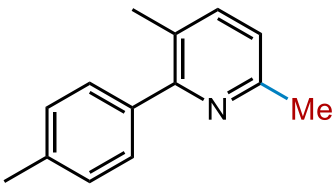
2-methyl-6-(thiophen-2-yl)pyridine (18): A colorless oil (27.2 mg, 62% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 – 7.52 (m, 2H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.37 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.10 (dd, *J* = 5.1, 3.6 Hz, 1H), 7.00 (d, *J* = 7.6 Hz, 1H), 2.58 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 157.4, 150.9, 144.2, 135.7, 126.9, 126.1, 123.3, 120.4, 114.8, 23.5. **HRMS (ESI)**: call for $C_{10}H_9NS$ $[M+H]^+$: 176.0528. Found: 176.0515.



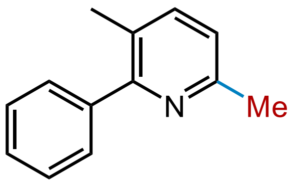
1-methyl-2-(6-methylpyridin-2-yl)-1H-indole (19): A yellow solid (27.6 mg, 50% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.52 – 7.44 (m, 2H), 7.35 (d, J = 7.8 Hz, 1H), 7.25 (d, J = 8.3 Hz, 1H), 7.15 – 7.08 (m, 1H), 7.01 – 6.91 (m, 2H), 6.68 (s, 1H), 3.92 (s, 3H), 2.48 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 157.8, 151.9, 139.3, 139.3, 136.8, 127.6, 122.4, 121.3, 120.9, 120.6, 119.8, 109.9, 103.3, 31.9, 24.7. **HRMS (ESI)**: call for $C_{15}H_{14}N_2$ $[M+H]^+$: 222.1157. Found: 222.1162.



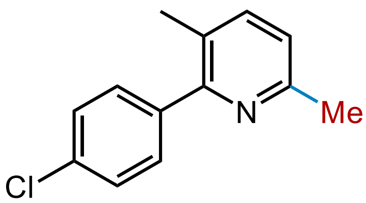
2-(4-methoxyphenyl)-3,6-dimethylpyridine (20): A colorless oil (44.3 mg, 84% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.51 – 7.39 (m, 3H), 7.05 – 6.91 (m, 3H), 3.84 (s, 3H), 2.55 (s, 3H), 2.29 (s, 3H). Spectra matched literature values.[16]



3,6-dimethyl-2-(p-tolyl)pyridine (21): A colorless oil (44.3 mg, 63% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 7.7 Hz, 1H), 7.42 – 7.35 (m, 2H), 7.29 – 7.21 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 2.56 (s, 3H), 2.40 (s, 3H), 2.29 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 158.0, 155.3, 138.7, 138.0, 137.4, 128.9, 128.8, 127.4, 121.5, 24.2, 21.3, 19.7. **HRMS (ESI)**: call for $C_{14}H_{15}N$ $[M+H]^+$: 198.1283. Found: 198.1267.

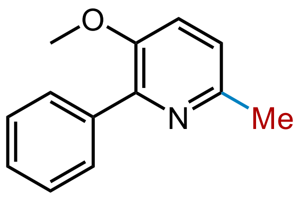


3,6-dimethyl-2-phenylpyridine (22): A colorless oil (24.0 mg, 52% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.52 – 7.40 (m, 5H), 7.40 – 7.34 (m, 1H), 7.04 (d, *J* = 7.8 Hz, 1H), 2.57 (s, 3H), 2.28 (s, 3H). Spectra matched literature values.[17]

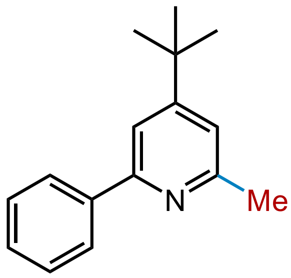


2-(4-chlorophenyl)-3,6-dimethylpyridine (23): A white solid (23.5 mg, 43% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.48 – 7.42 (m, 3H), 7.42 – 7.38 (m, 2H), 7.05 (d, *J* = 7.8 Hz, 1H), 2.56 (s, 3H), 2.27 (s, 3H).

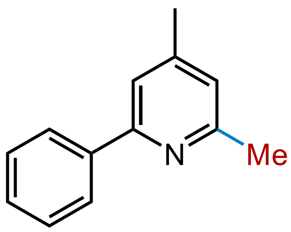
^{13}C {^{1}H} NMR (101 MHz, Chloroform-*d*) δ 156.7, 155.6, 139.3, 139.0, 133.8, 130.4, 128.4, 127.5, 122.0, 24.2, 19.5. **HRMS (ESI)**: call for $C_{13}H_{12}ClN$ $[M+H]^+$: 218.0731. Found: 218.0718.



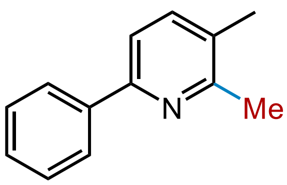
3-methoxy-6-methyl-2-phenylpyridine (24): A colorless oil (44.2 mg, 89% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.91 – 7.84 (m, 2H), 7.47 – 7.40 (m, 2H), 7.40 – 7.32 (m, 1H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.07 (d, *J* = 8.4 Hz, 1H), 3.82 (s, 3H), 2.56 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 151.5, 149.7, 147.2, 137.9, 129.4, 128.1, 128.0, 122.3, 119.3, 55.7, 23.6. **HRMS (ESI)**: call for $C_{13}H_{13}NO$ $[M+H]^+$: 200.1070. Found: 200.1059.



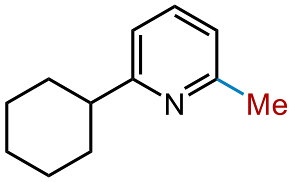
4-(tert-butyl)-2-methyl-6-phenylpyridine (25): A colorless oil (34.4 mg, 61% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 – 7.94 (m, 2H), 7.52 – 7.43 (m, 3H), 7.43 – 7.36 (m, 1H), 7.11 (d, *J* = 1.6 Hz, 1H), 2.63 (s, 3H), 1.36 (s, 9H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 159.8, 157.1, 156.1, 139.4, 127.6, 127.4, 126.1, 117.7, 114.1, 33.7, 29.6, 23.9. **HRMS (ESI)**: call for $C_{16}H_{19}N$ $[M+H]^+$: 226.1590. Found: 226.1583.



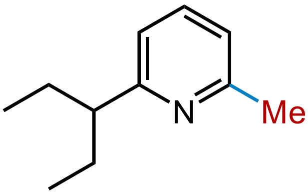
2,4-dimethyl-6-phenylpyridine (26): A colorless oil (24.0 mg, 34% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.00 – 7.91 (m, 2H), 7.50 – 7.42 (m, 2H), 7.41 – 7.36 (m, 1H), 7.34 (s, 1H), 6.94 (s, 1H), 2.59 (s, 3H), 2.37 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 157.1, 155.9, 146.7, 138.9, 127.6, 127.5, 126.0, 121.6, 117.8, 23.5, 20.1. **HRMS (ESI)**: call for $C_{13}H_{13}N$ $[M+H]^+$: 184.1121. Found: 184.1109.



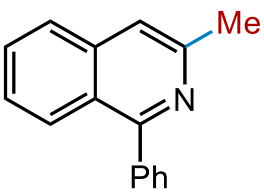
2,3-dimethyl-6-phenylpyridine (27): A colorless oil (9.5 mg, 21% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 – 7.94 (m, 2H), 7.48 – 7.42 (m, 4H), 7.40 – 7.34 (m, 1H), 2.58 (s, 3H), 2.32 (s, 3H). Spectra matched literature values.[18]



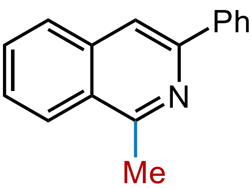
2-cyclohexyl-6-methylpyridine (28): A colorless oil (23.4 mg, 53% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.48 (t, *J* = 7.7 Hz, 1H), 6.94 (d, *J* = 7.1 Hz, 2H), 2.70 – 2.63 (m, 1H), 2.52 (s, 3H), 1.99 – 1.92 (m, 2H), 1.87 – 1.80 (m, 2H), 1.77 – 1.71 (m, 1H), 1.51 – 1.36 (m, 4H), 1.32 – 1.24 (m, 1H). Spectra matched literature values.[18]



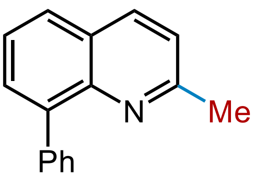
2-methyl-6-(pentan-3-yl)pyridine (29): A colorless oil (14.0 mg, 34% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.46 (t, *J* = 7.7 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 2.52 (s, 4H), 1.72 – 1.63 (m, 4H), 0.78 (t, *J* = 7.4 Hz, 6H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 163.7, 156.5, 135.1, 119.4, 117.8, 50.4, 27.1, 23.7, 11.0. **HRMS (ESI)**: call for $C_{11}H_{17}N$ $[M+H]^+$: 164.1434. Found: 164.1421.



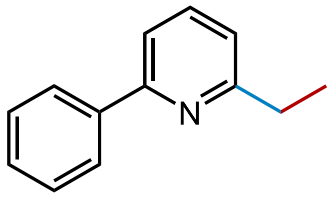
3-methyl-1-phenylisoquinoline (30): A white solid (38.6 mg, 71% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.5 Hz, 1H), 7.78 (d, *J* = 8.3 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.65 – 7.58 (m, 1H), 7.56 – 7.47 (m, 4H), 7.46 – 7.40 (m, 1H), 2.76 (s, 3H). Spectra matched literature values.[19]



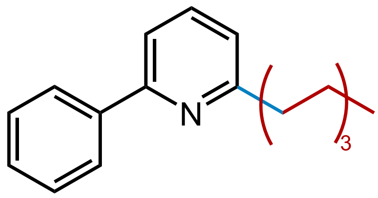
1-methyl-3-phenylisoquinoline (31): A white solid (10.5 mg, 19% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 – 8.10 (m, 3H), 7.93 (s, 1H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.67 (t, *J* = 7.0 Hz, 1H), 7.57 (t, *J* = 7.0 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 3.05 (s, 3H). Spectra matched literature values.[20]



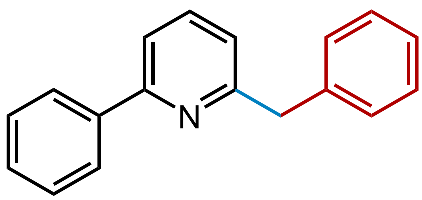
2-methyl-8-phenylquinoline (32): A colorless oil (9.5 mg, 17% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.4 Hz, 1H), 7.82 – 7.75 (m, 3H), 7.72 (dd, *J* = 7.2, 1.5 Hz, 1H), 7.58 – 7.44 (m, 3H), 7.43 – 7.38 (m, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 2.69 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 157.7, 144.4, 138.8, 138.5, 135.2, 130.0, 129.2, 126.7, 126.2, 126.1, 125.9, 124.3, 120.8, 24.7. **HRMS (ESI)**: call for $C_{16}H_{13}N$ $[M+H]^+$: 220.1121. Found: 220.1120.



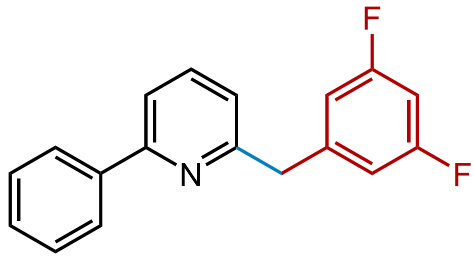
2-ethyl-6-phenylpyridine (33): A colorless oil (24.1 mg, 53% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.06 – 7.98 (m, 2H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 7.52 – 7.43 (m, 2H), 7.44 – 7.36 (m, 1H), 7.11 (d, *J* = 7.7 Hz, 1H), 2.91 (q, *J* = 7.6 Hz, 2H), 1.38 (t, *J* = 7.6 Hz, 3H). Spectra matched literature values.[21]



2-heptyl-6-phenylpyridine (34): A colorless oil (10.6 mg, 17% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.00 (dd, *J* = 7.2, 1.7 Hz, 1H), 7.64 (t, *J* = 7.7 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.42 – 7.37 (m, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 2.88 – 2.83 (m, 1H), 1.84 – 1.76 (m, 1H), 1.41 – 1.27 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 2H).**^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 161.4, 155.8, 138.9, 135.8, 127.6, 127.6, 126.0, 119.9, 116.7, 37.5, 30.8, 28.8, 28.4, 28.2, 21.7, 13.1. **HRMS (ESI)**: call for $C_{18}H_{23}N$ $[M+H]^+$: 254.1903. Found: 254.1858.

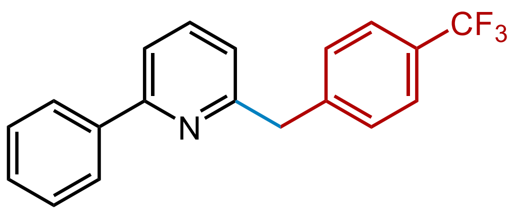


2-benzyl-6-phenylpyridine (35): A colorless oil (19.5 mg, 33% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.02 (d, J = 8.5 Hz, 2H), 7.63 (t, J = 7.7 Hz, 1H), 7.55 (d, J = 7.7 Hz, 1H), 7.50 – 7.45 (m, 2H), 7.43 – 7.39 (m, 1H), 7.36 – 7.30 (m, 4H), 7.26 – 7.21 (m, 1H), 7.02 (d, J = 7.5 Hz, 1H), 4.24 (s, 2H). Spectra matched literature values.[22]

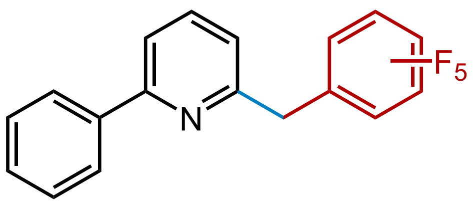


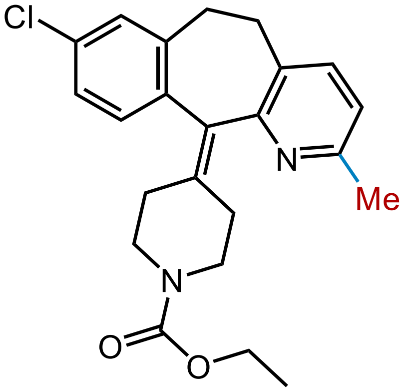
2-(3,5-difluorobenzyl)-6-phenylpyridine (36): A white solid (31.4 mg, 45% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.07 – 7.98 (m, 2H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.60 (d, *J* = 6.9 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.46 – 7.39 (m, 1H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.92 – 6.82 (m, 2H), 6.68 (tt, *J* = 9.0, 2.3 Hz, 1H), 4.20 (s, 2H). **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -110.29 – -110.33 (m). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 163.1 (dd, J_{CF} = 242.4 Hz, 10.1 Hz), 159.2, 157.2, 143.6 (t, J_{CF} = 9.4 Hz), 139.3, 137.4, 129.0, 128.8, 127.0, 121.4, 118.4, 112.5 – 111.6 (m), 101.8 (t, *J* = 25.3 Hz), 44.5 (t, J_{CF} = 2.0 Hz). **HRMS (ESI)**: call for $C_{18}H_{13}F_2N$ [M+H]$^+$: 282.1089. Found: 282.1088.

2-phenyl-6-(4-(trifluoromethyl)benzyl)pyridine (37): A white solid (28.1 mg, 36% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.05 – 7.98 (m, 2H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.62 – 7.54 (m, 3H), 7.52 – 7.39 (m, 5H), 7.04 (d, *J* = 7.6 Hz, 1H), 4.28 (s, 2H). **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -62.33. **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) 159.7, 157.1, 143.8, 139.4, 137.4, 129.5, 129.0, 128.8, 128.7 (q, J_{CF} = 30.3 Hz), 127.0, 125.4 (q, J_{CF} = 3.9 Hz), 124.4 (q, J_{CF} = 272.2 Hz),121.4, 118.3, 44.6. **HRMS (ESI)**: call for $C_{19}H_{14}F_3N$ [M+H]$^+$: 314.1151. Found: 314.1126.



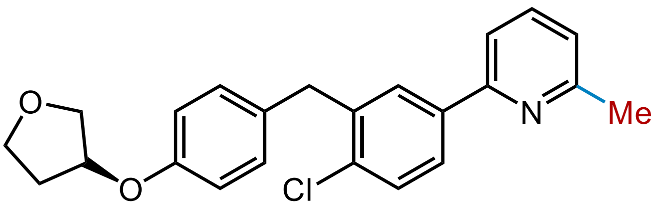
2-((perfluorophenyl)methyl)-6-phenylpyridine (38): A white solid (20.9 mg, 25% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.98 – 7.93 (m, 2H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.61 (d, *J* = 7.0 Hz, 1H), 7.49 – 7.38 (m, 3H), 7.11 (d, *J* = 7.5 Hz, 1H), 4.28 (s, 2H). **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -142.10 – -142.49 (m), -156.88 (td, J = 20.8, 2.7 Hz), -162.41 – -164.03 (m). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 156.98, 156.61, 138.94, 137.49, 129.09, 128.73, 126.81, 120.60, 118.41, 30.93. **HRMS (ESI)**: call for $C_{18}H_{10}F_5N$ [M+H]$^+$: 336.0806. Found: 336.0802.



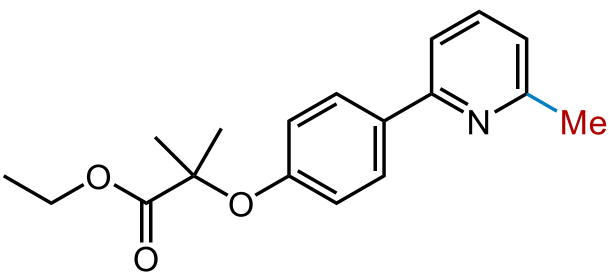


ethyl 4-(8-chloro-2-methyl-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene) piperidine-1-carboxylate (39): A white solid (27.9 mg, 31% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.32 (d, *J* = 7.8 Hz, 1H), 7.18 – 7.11 (m, 3H), 6.95 (d, *J* = 7.8 Hz, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.87 (s, 2H), 3.40 – 3.25 (m, 2H), 3.06 – 2.98 (m, 2H), 2.83 – 2.73 (m, 2H), 2.49 (s, 3H), 2.43 (dd, *J* = 9.9, 4.7 Hz, 1H), 2.34 (t, *J* = 6.6 Hz, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 155.5, 155.3, 139.6, 137.8, 137.6, 137.2, 132.8, 130.6, 130.0, 129.1, 126.1, 121.9, 61.3, 44.8, 44.7, 31.9, 31.1, 30.8, 30.5, 24.1, 14.7. **HRMS (ESI)**: call for $C_{23}H_{25}ClN_2O_2$ [M+H]$^+$: 397.1677. Found: 397.1675.

(R)-2-(4-chloro-3-(4-((tetrahydrofuran-3-yl)oxy)benzyl)phenyl)-6-methylpyridine (40): A colorless oil (42.9 mg, 45% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.86 – 7.74 (m, 2H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 11.0, 8.0 Hz, 2H), 7.14 (d, *J* = 8.7 Hz, 2H), 7.09 (d, *J* = 7.6 Hz, 1H), 6.84 – 6.69 (m, 2H), 4.90. – 4.86 (m, 1H), 412 (s, 2H), 4.02 – 3.93 (m, 3H), 3.88 (td, *J* = 8.0, 4.8 Hz, 1H), 2.60 (s, 3H), 2.21 – 2.09 (m, 2H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 158.5, 155.8, 155.8, 139.0, 138.6, 137.0, 134.9, 132.0, 129.9, 129.9, 129.6, 126.3, 121.9, 117.5, 115.4, 77.3, 73.2, 67.2, 38.5, 33.0, 24.7. **HRMS (ESI)**: call for $C_{23}H_{22}ClNO_2$ [M+H]$^+$: 380.1412. Found: 380.1410.

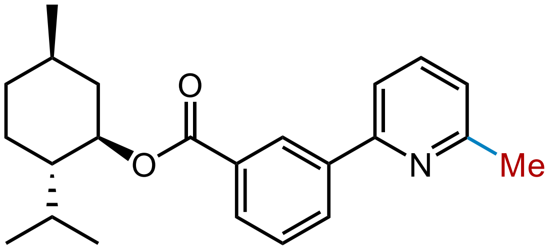


ethyl 2-methyl-2-(4-(6-methylpyridin-2-yl)phenoxy)propanoate (41): A colorless oil (30.5 mg, 41% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.90 – 7.84 (m, 2H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 1H), 6.96 – 6.86 (m, 2H),

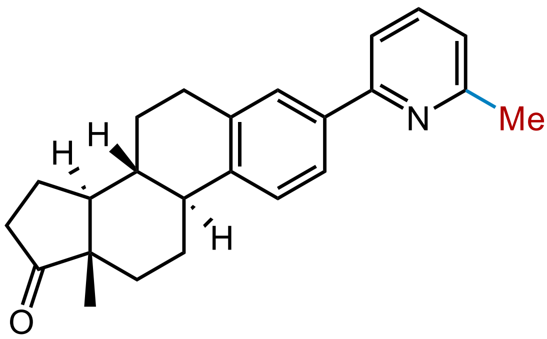


4.24 (q, *J* = 7.1 Hz, 2H), 2.59 (s, 3H), 1.62 (s, 6H), 1.25 (t, *J* = 7.1 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 174.3, 158.2, 156.5, 156.2, 136.8, 133.7, 127.9, 121.1, 119.0, 117.1, 79.2, 61.5, 25.4, 24.8, 14.1. **HRMS (ESI)**: call for $C_{18}H_{21}NO_3$ $[M+H]^+$: 300.1594. Found: 300.1591.

(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl 3-(6-methylpyridin-2-yl)benzoate (42):

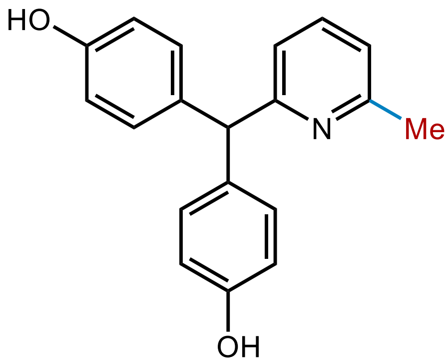


A colorless oil (35.1 mg, 40% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.60 – 8.60 (m, 1H), 8.25 – 8.18 (m, 1H), 8.07 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.59 – 7.50 (m, 2H), 7.13 (d, *J* = 7.5 Hz, 1H), 4.98 (td, *J* = 10.9, 4.4 Hz, 1H), 2.64 (s, 3H), 2.19 – 2.11 (m, 1H), 1.77 – 1.70 (m, 2H), 1.63 – 1.52 (m, 2H), 1.20 – 1.09 (m, 2H), 0.93 (dd, *J* = 6.8, 4.9 Hz, 7H), 0.81 (d, *J* = 7.0 Hz, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 166.1, 158.6, 156.0, 140.1, 137.0, 131.3, 131.3, 129.7, 128.8, 128.1, 122.0, 117.7, 75.0, 47.3, 41.0, 34.4, 31.5, 26.5, 24.7, 23.7, 22.1, 20.8, 16.6. **HRMS (ESI)**: call for $C_{23}H_{29}NO_2$ $[M+H]^+$: 352.2271. Found: 352.2270.



(8R,9S,13S,14S)-13-methyl-3-(6-methylpyridin-2-yl)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (43): A white solid (73.0 mg, 85% yield). **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.72 (dd, *J* = 10.5, 2.6 Hz, 2H), 7.61 (t, *J* = 7.7 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 3.05 – 2.98 (m, 2H), 2.62 (s, 3H), 2.55 – 2.45 (m, 3H), 2.36 (td, *J* = 10.8, 4.2 Hz, 1H), 2.18 – 1.95 (m, 5H), 1.67 – 1.46 (m, 9H), 0.93 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Chloroform-*d*) δ 158.3, 156.9, 140.5, 137.3, 136.9, 136.8, 127.5, 125.7, 124.3, 121.4, 117.5, 50.6, 48.0, 44.5, 38.2, 38.1, 35.9, 31.6, 29.5, 26.6, 25.8, 24.8, 21.6, 13.9. **HRMS (ESI)**: call for $C_{24}H_{27}NO$ $[M+H]^+$: 346.2165. Found: 346.2165.

4,4'-((6-methylpyridin-2-yl)methylene)diphenol (44): ^{1}H NMR (400 MHz,



Methanol-*d*$_4$) δ 7.64 (t, J = 7.8 Hz, 1H), 7.14 (d, J = 7.7 Hz, 1H), 6.90 (d, J = 8.6 Hz, 5H), 6.73 (d, J = 8.6 Hz, 4H), 5.51 (s, 1H), 2.52 (s, 3H). **^{13}C {^{1}H} NMR** (101 MHz, Methanol-*d*$_4$) δ 163.4, 157.4, 155.7, 137.5, 133.8, 129.9, 121.2, 121.0, 114.7, 57.1, 22.1. **HRMS (ESI)**: call for $C_{19}H_{17}NO_2$ [M+H]$^+$: 291.1259. Found: 291.1250.

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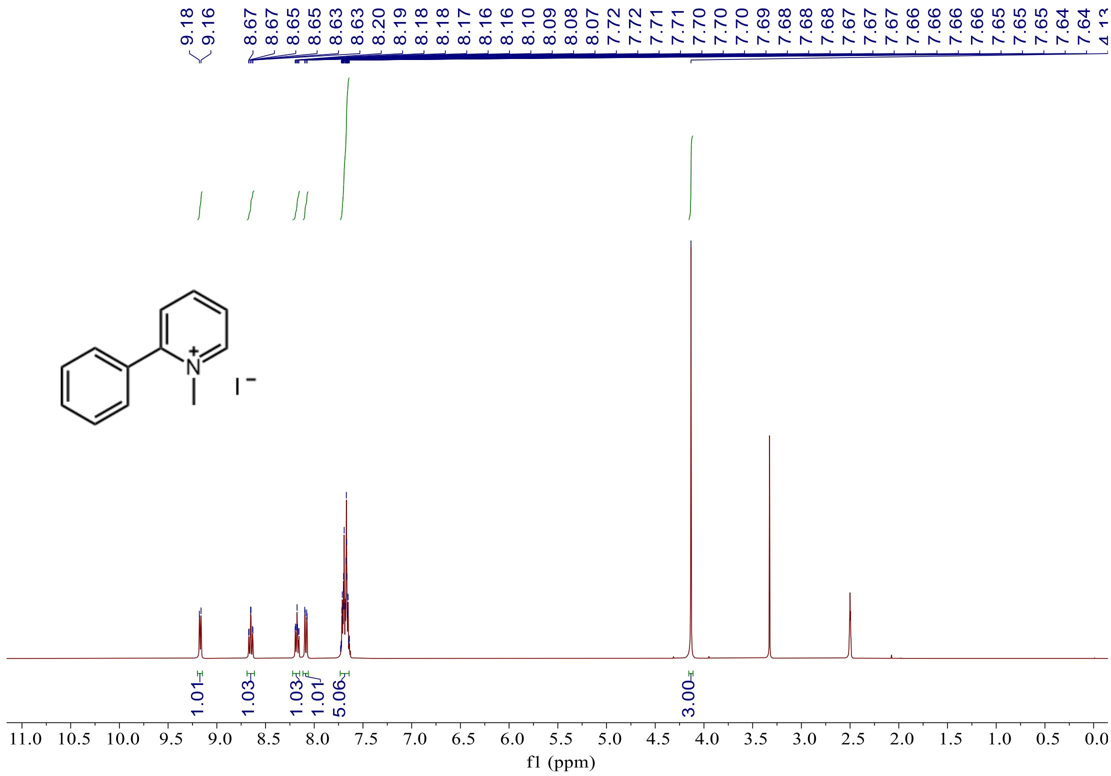
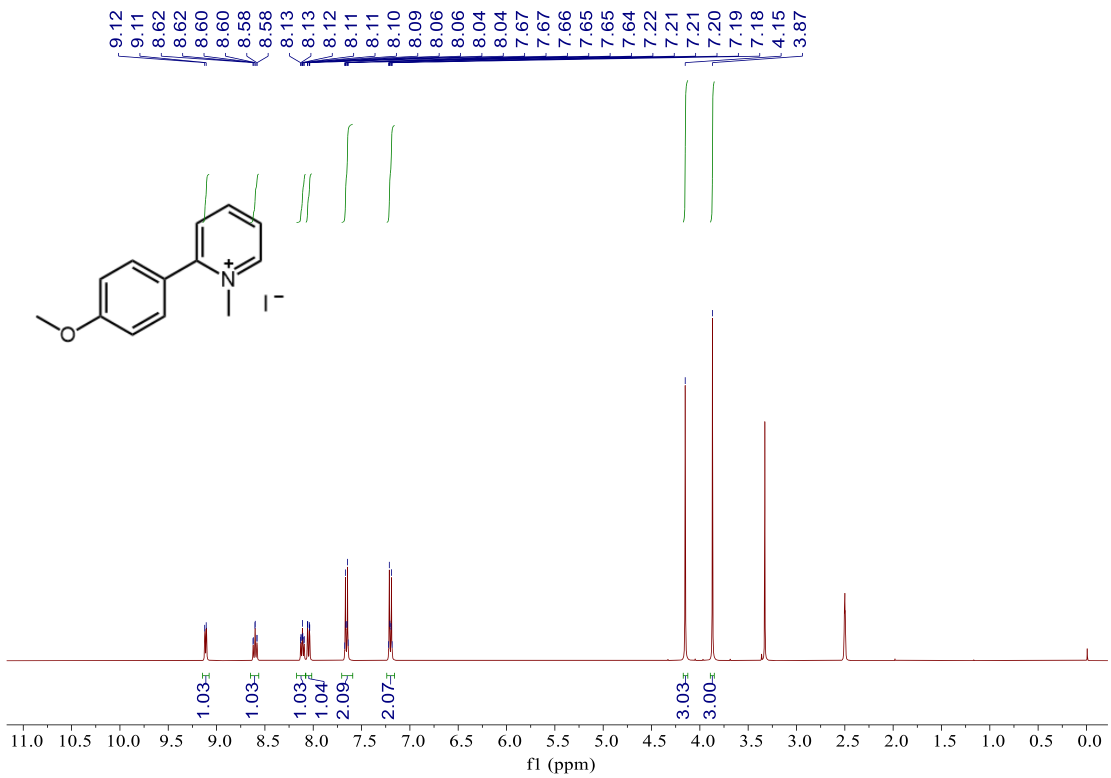
5. NMR spectra

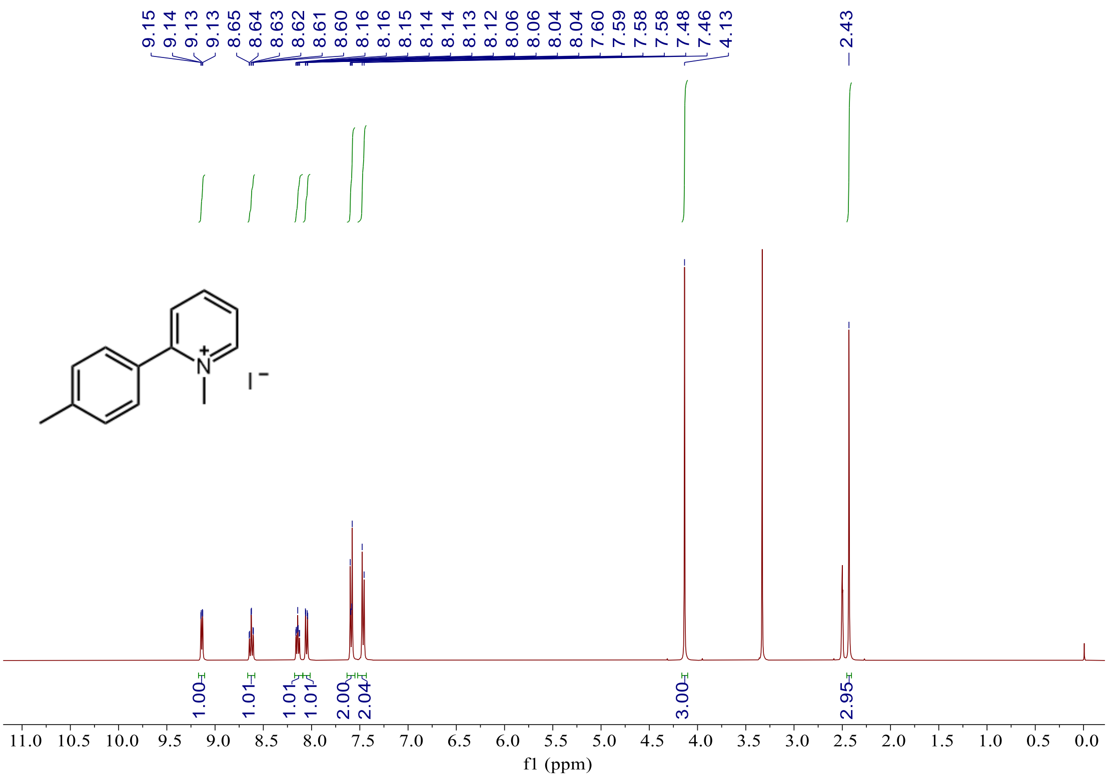
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1a**



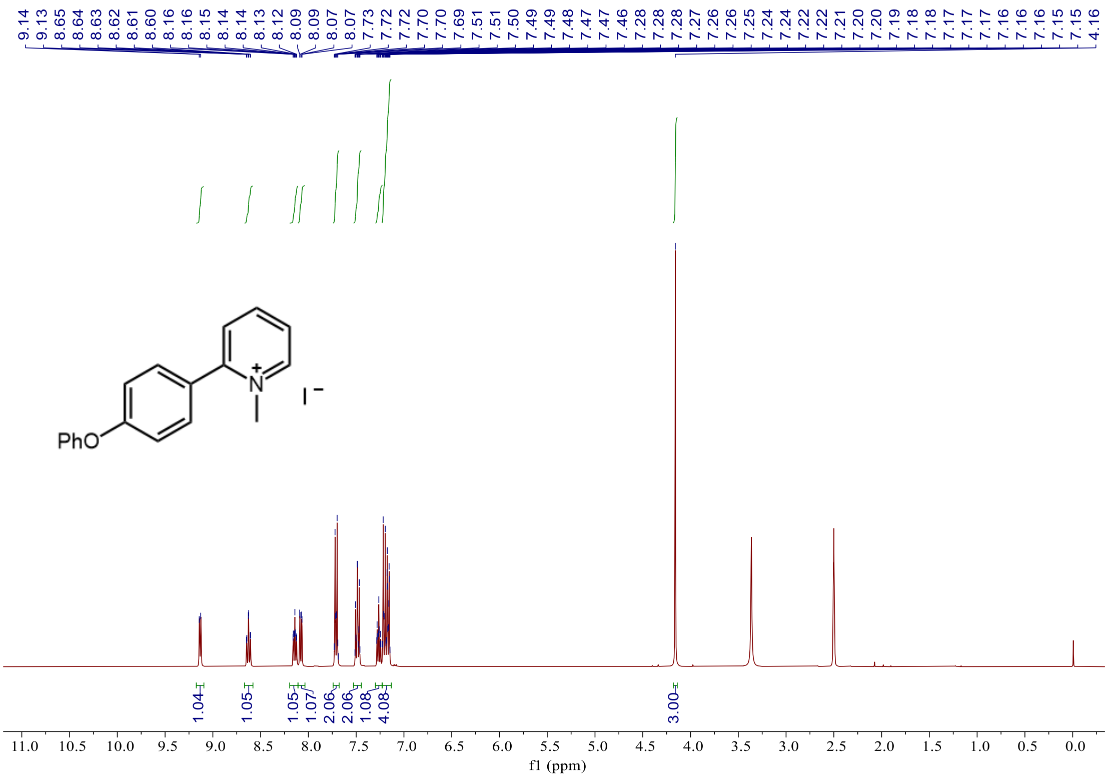
^{1}H NMR (400 MHz, DMSO-d_6) of compound **2a**



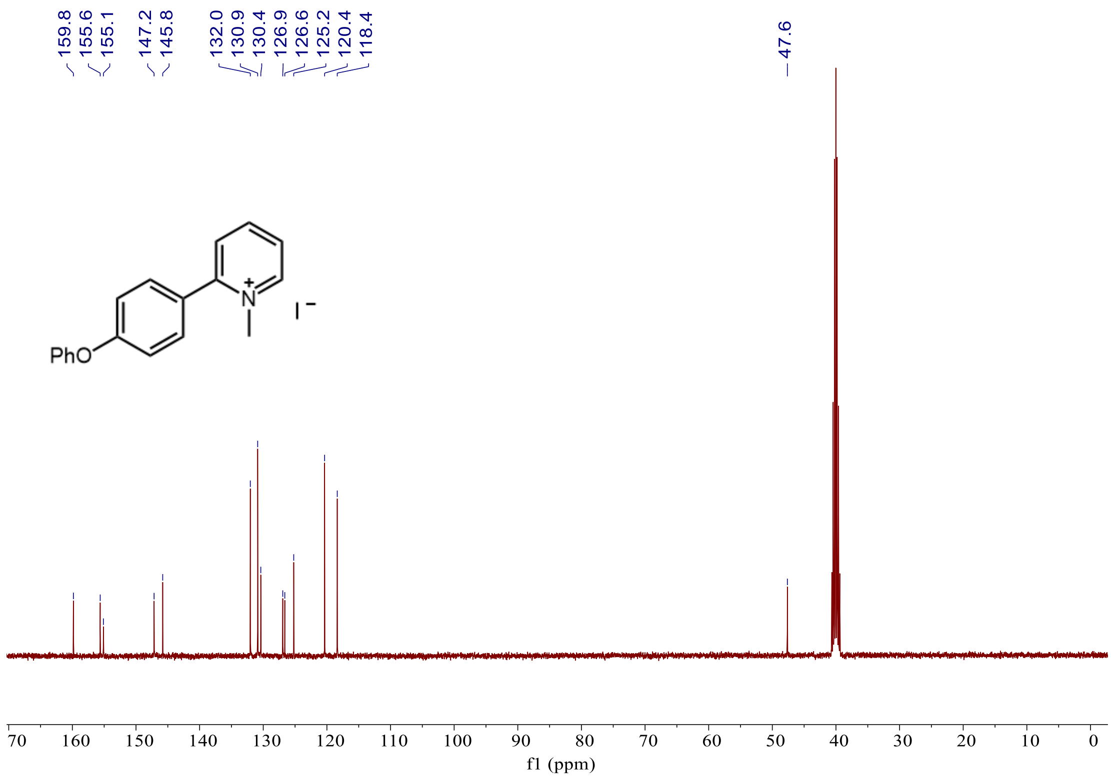
^{1}H NMR (400 MHz, DMSO-d_6) of compound **3a**



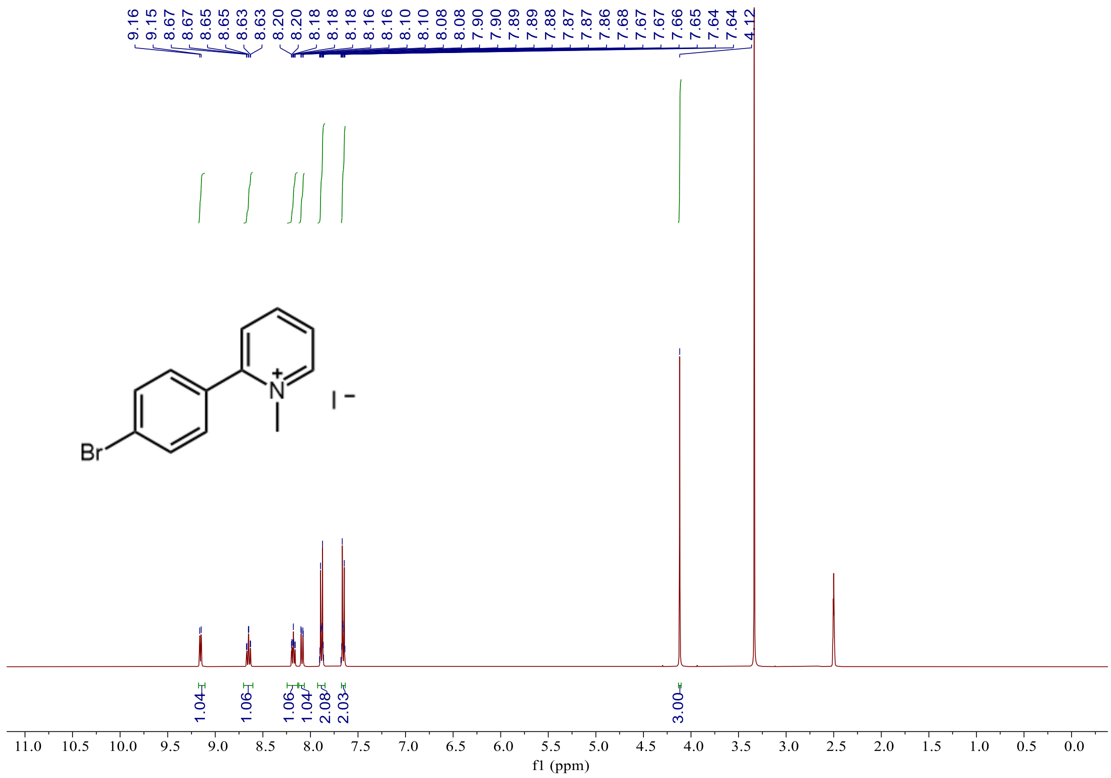
^{1}H NMR (400 MHz, DMSO-d_6) of compound **4a**



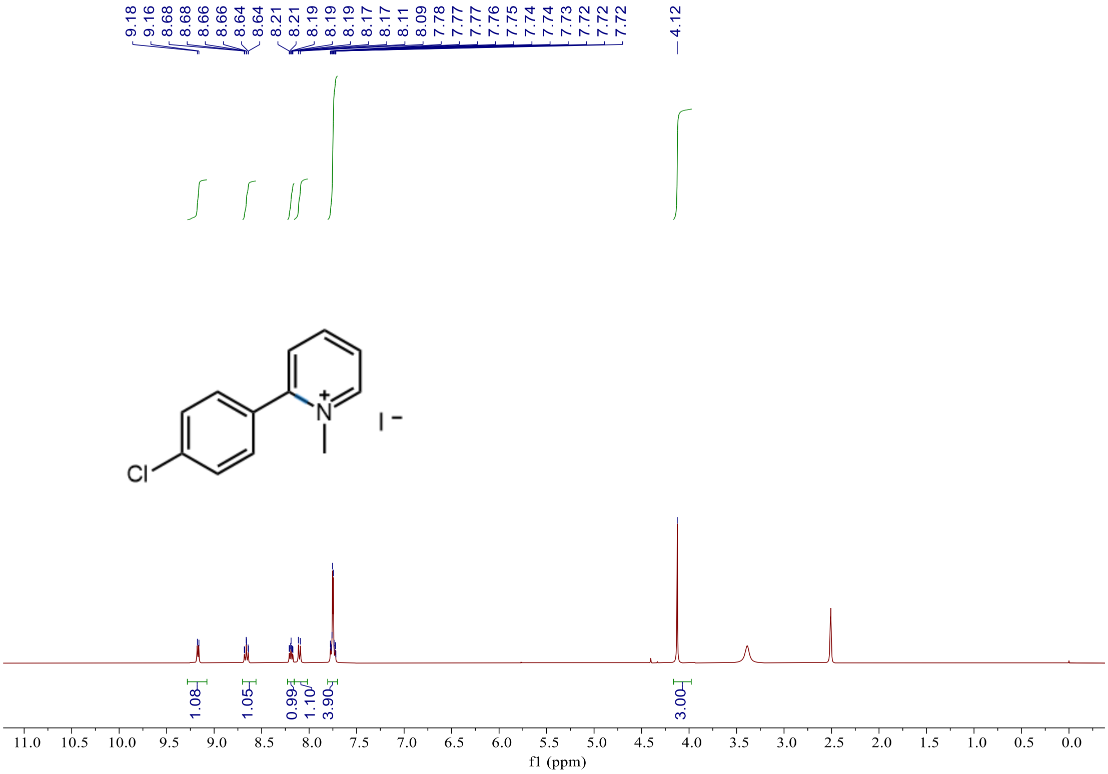
^{13}C NMR (101 MHz, DMSO-d_6) of compound **4a**



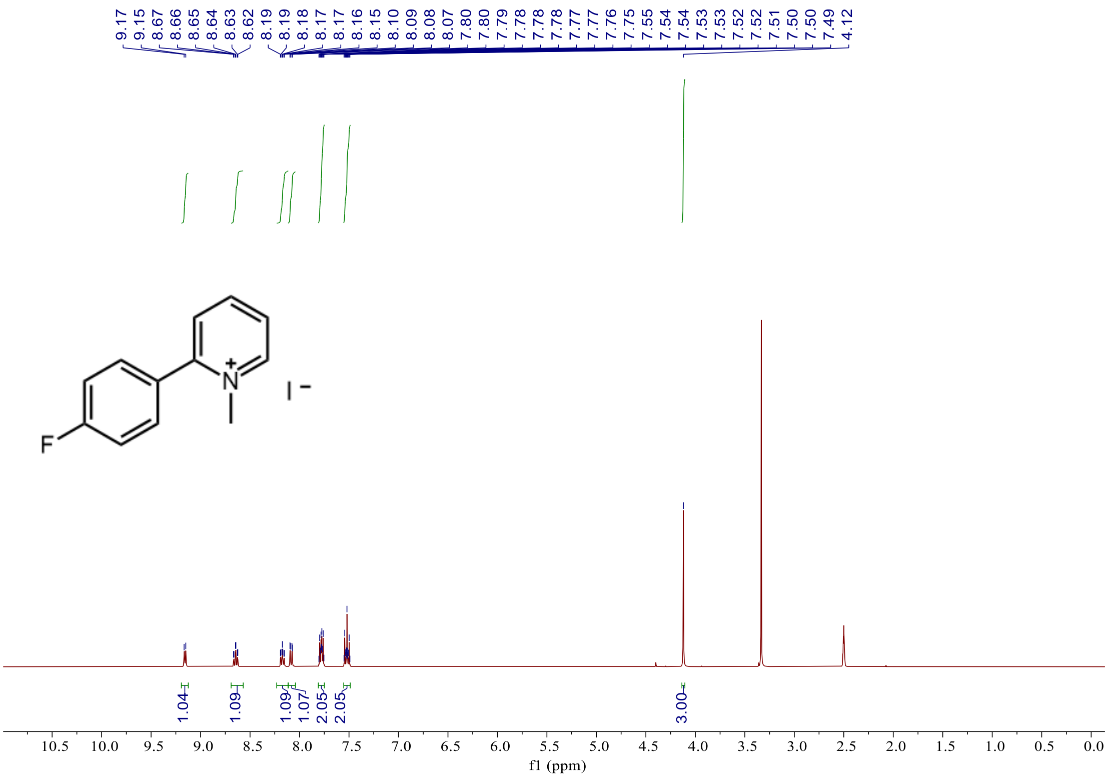
^{1}H NMR (400 MHz, DMSO-d_6) of compound **5a**



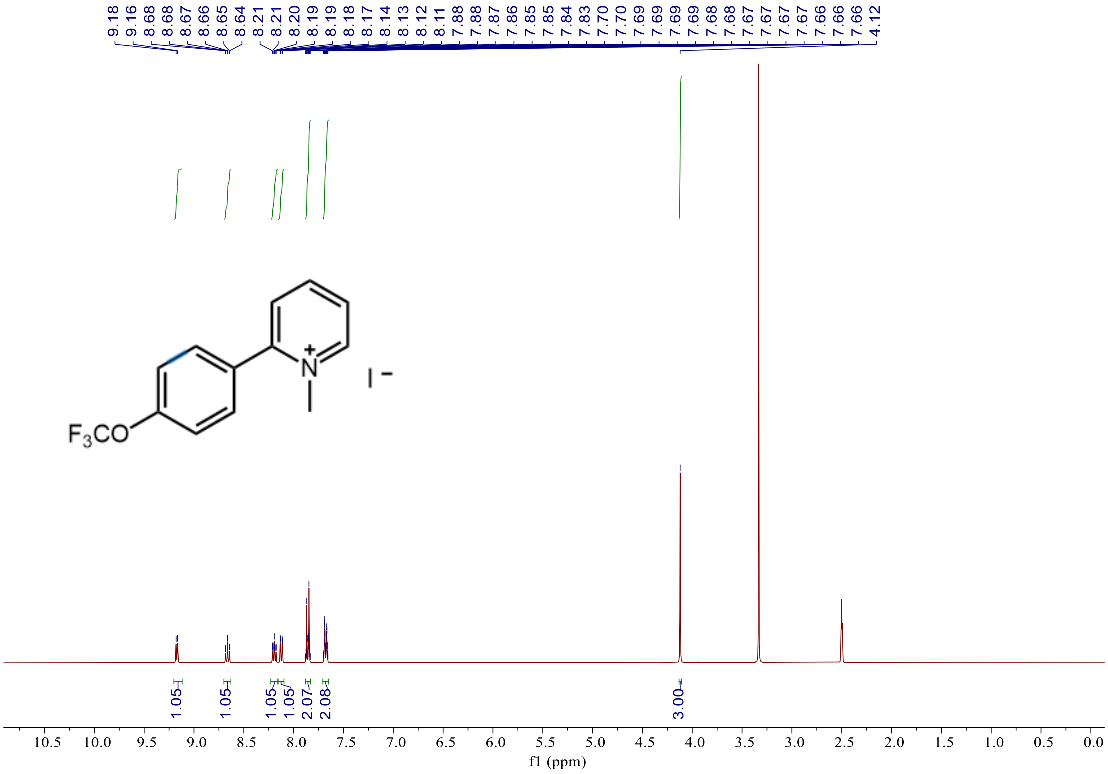
[1]H NMR (400 MHz, DMSO-d_6) of compound **6a**



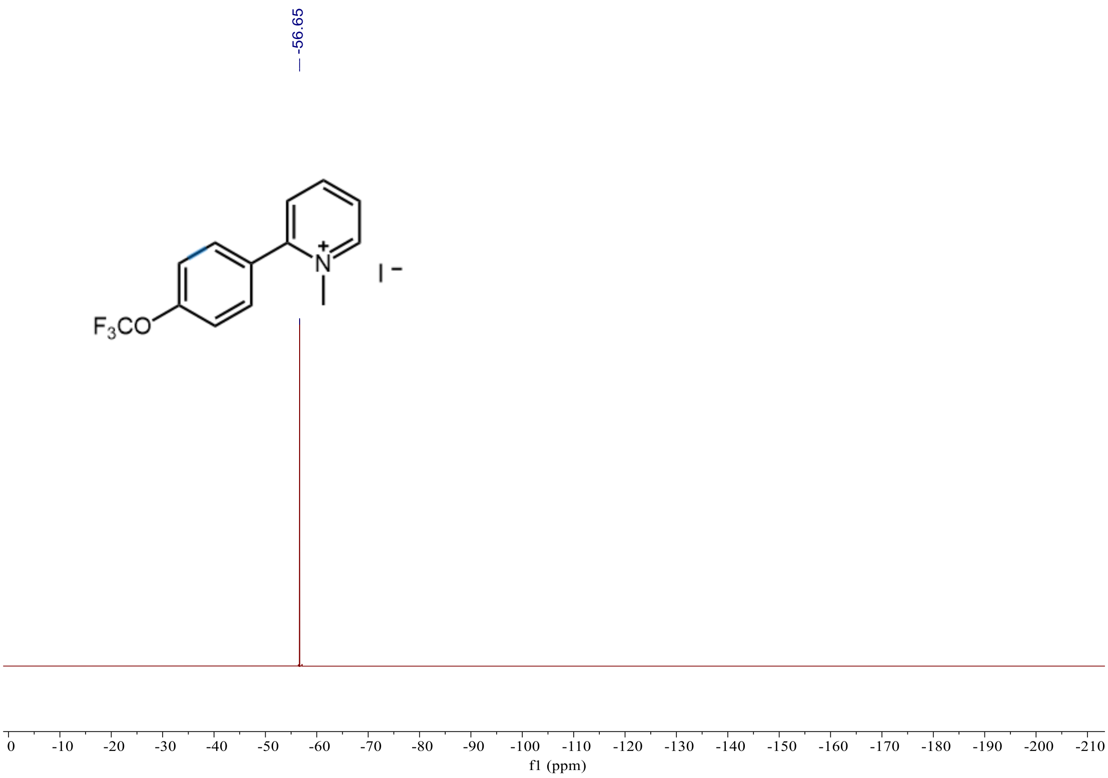
[1]H NMR (400 MHz, DMSO-d_6) of compound **7a**



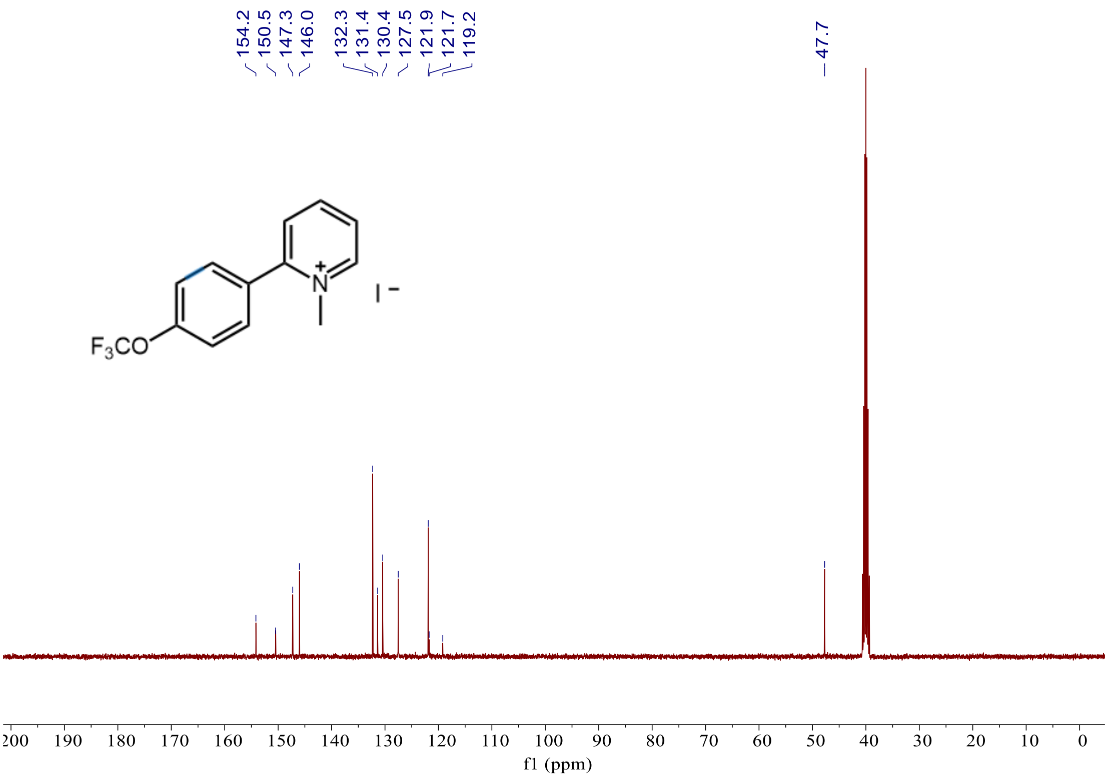
^{1}H NMR (400 MHz, DMSO-d_6) of compound **8a**



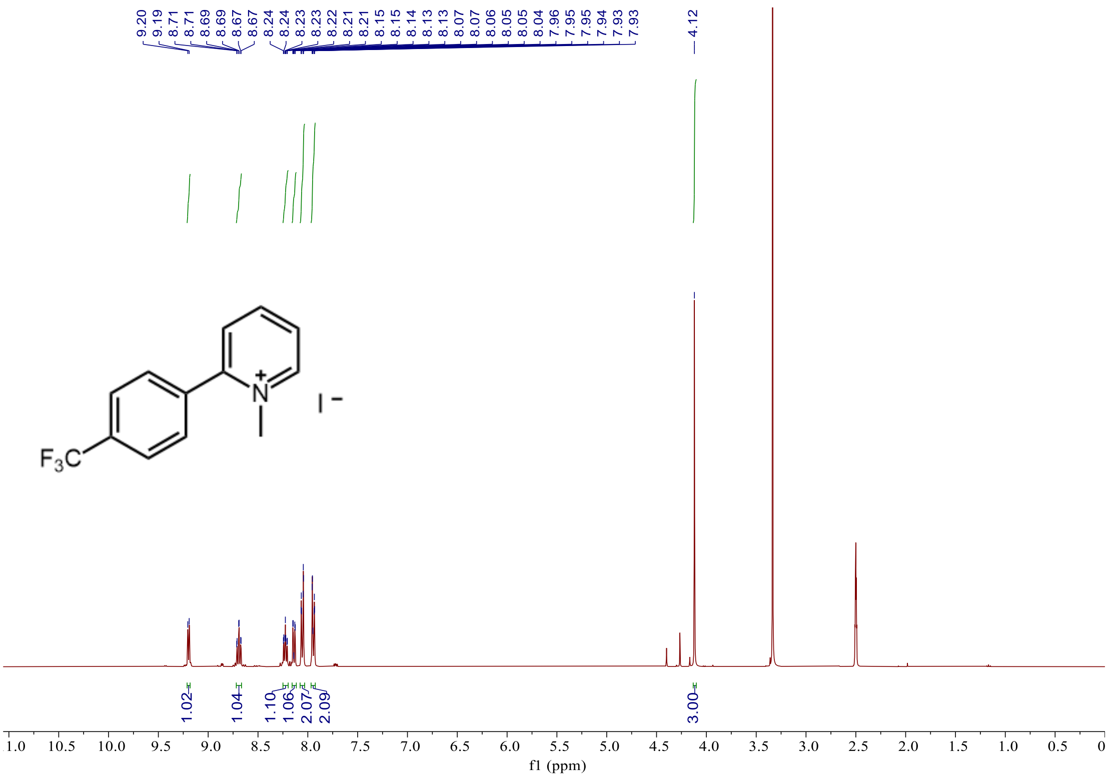
^{19}F NMR (400 MHz, DMSO-d_6) of compound **8a**



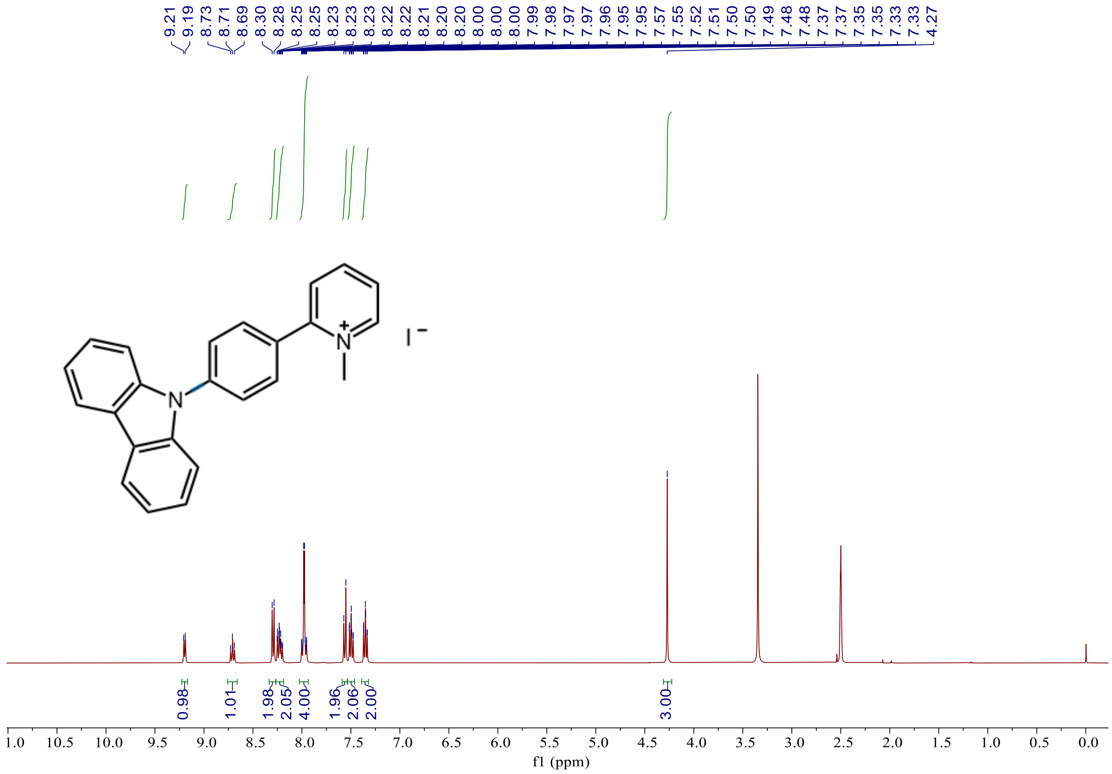
^{13}C NMR (101 MHz, DMSO-d_6) of compound **8a**



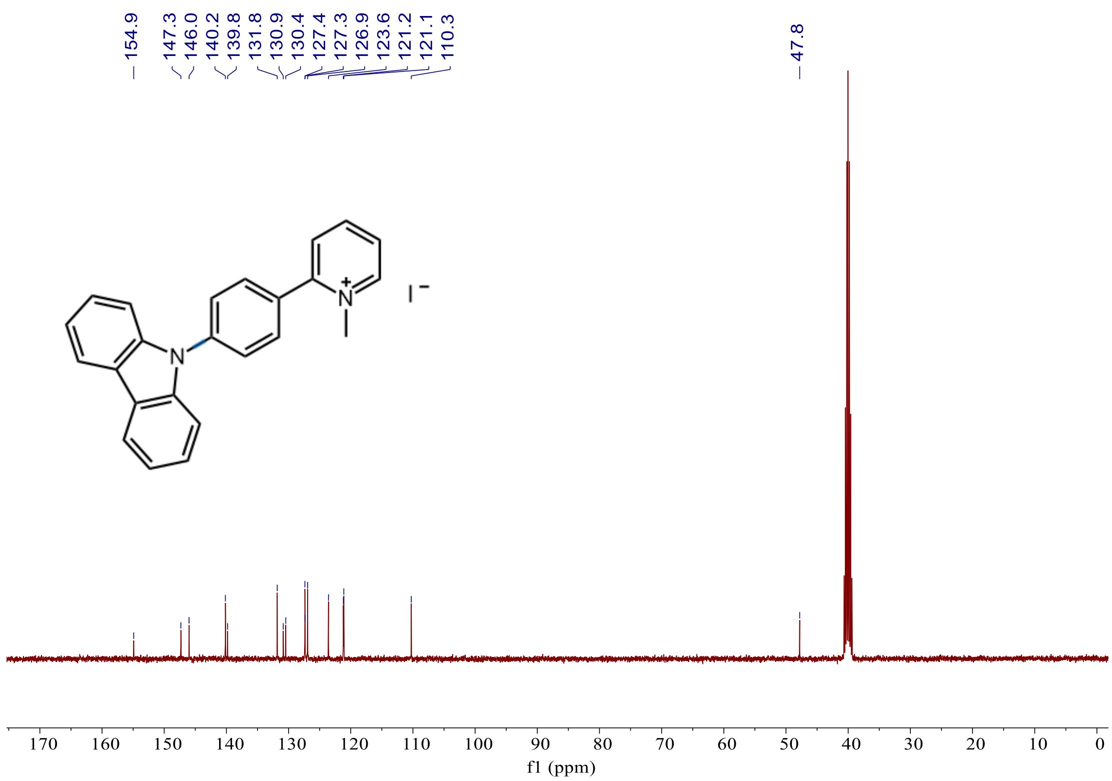
^{1}H NMR (400 MHz, DMSO-d_6) of compound **9a**



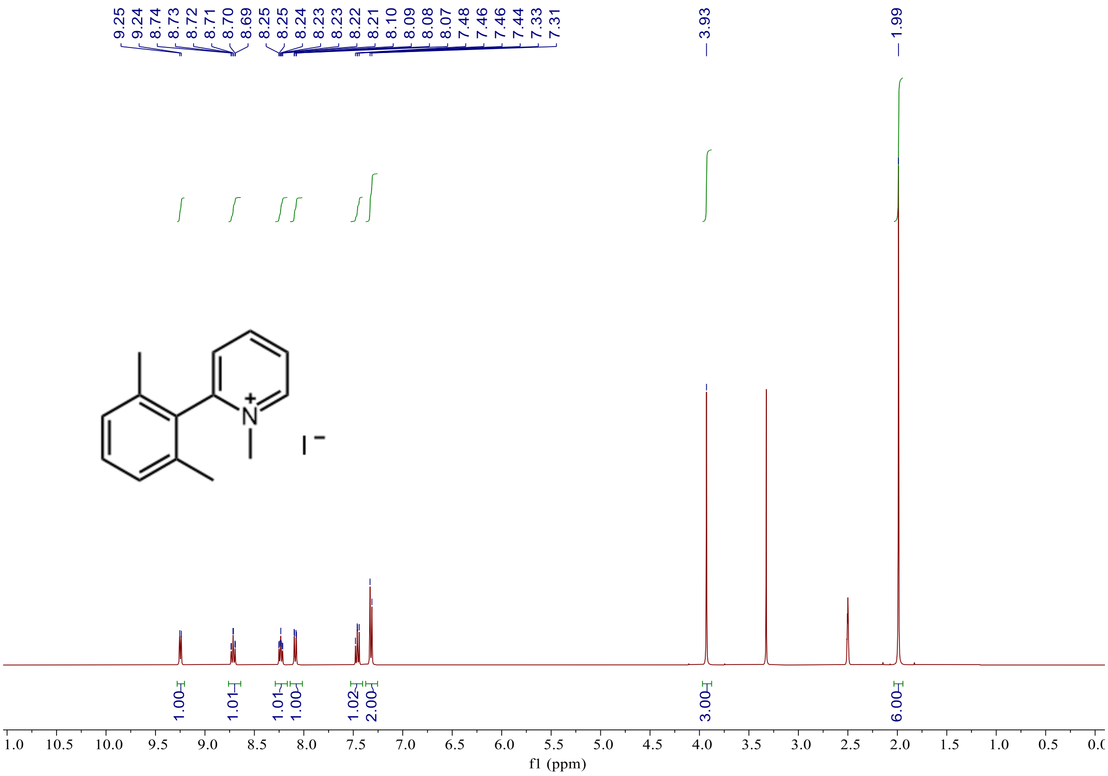
[1]H NMR (400 MHz, DMSO-d_6) of compound **10a**



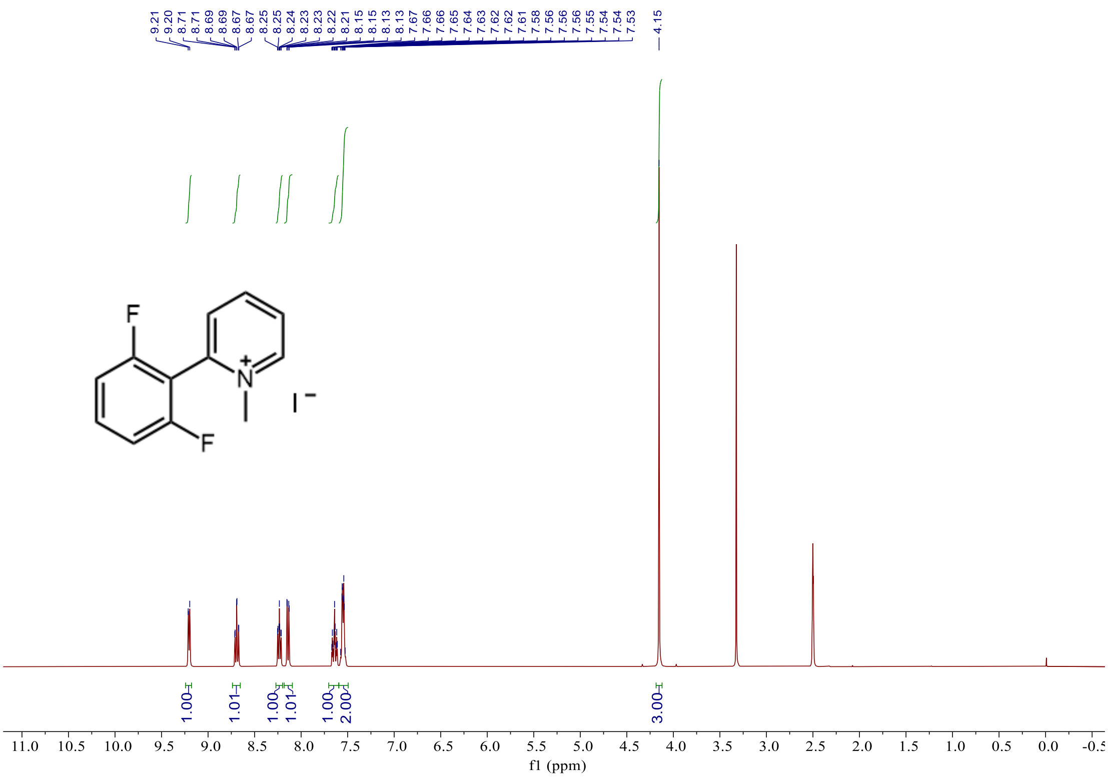
[13]C NMR (101 MHz, DMSO-d_6) of compound **10a**



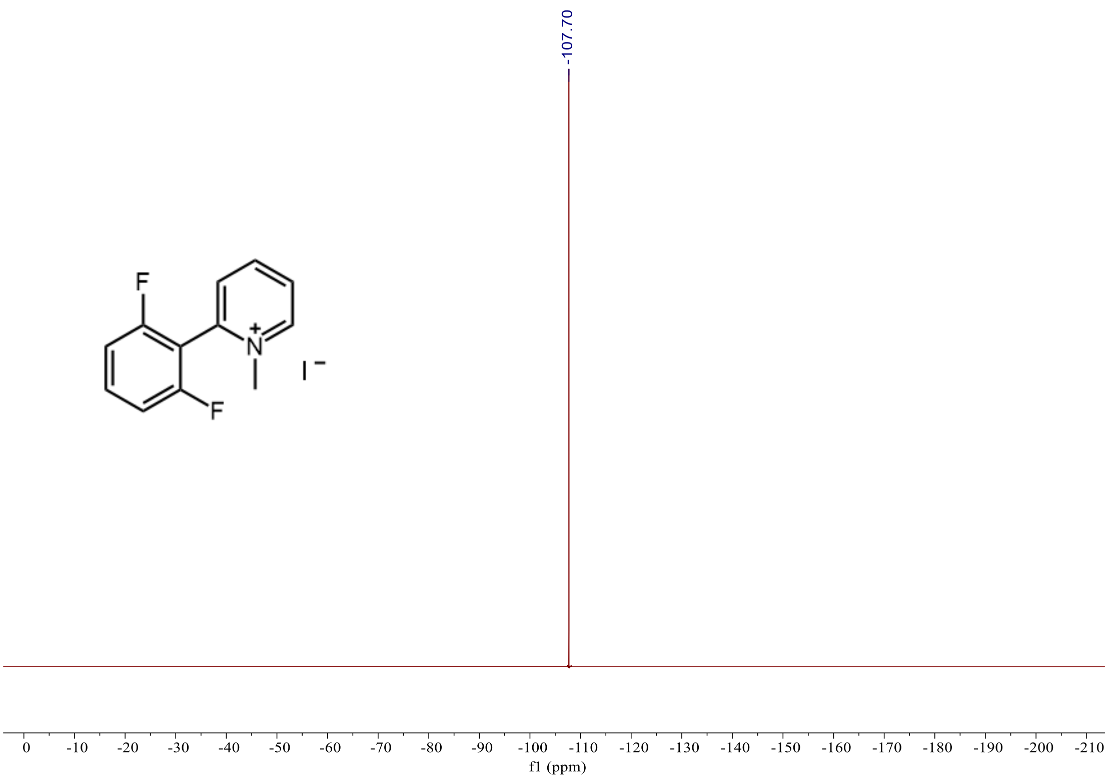
[1]H NMR (400 MHz, DMSO-d_6) of compound **11a**



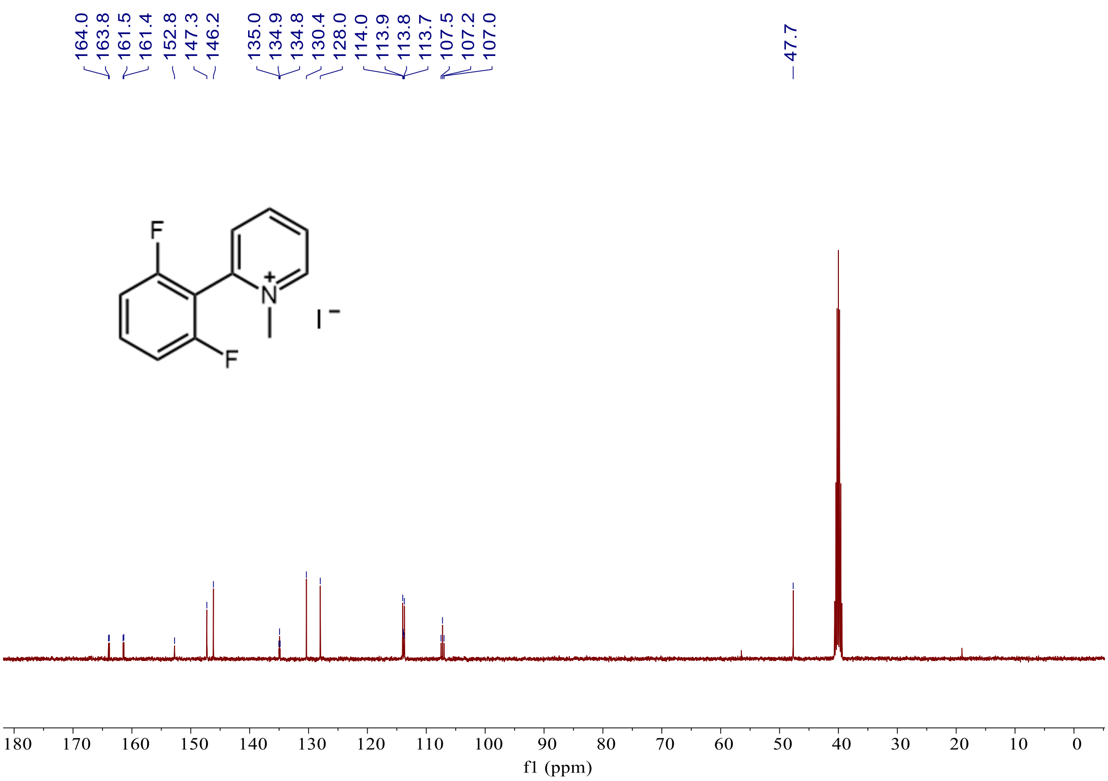
[1]H NMR (400 MHz, DMSO-d_6) of compound **12a**



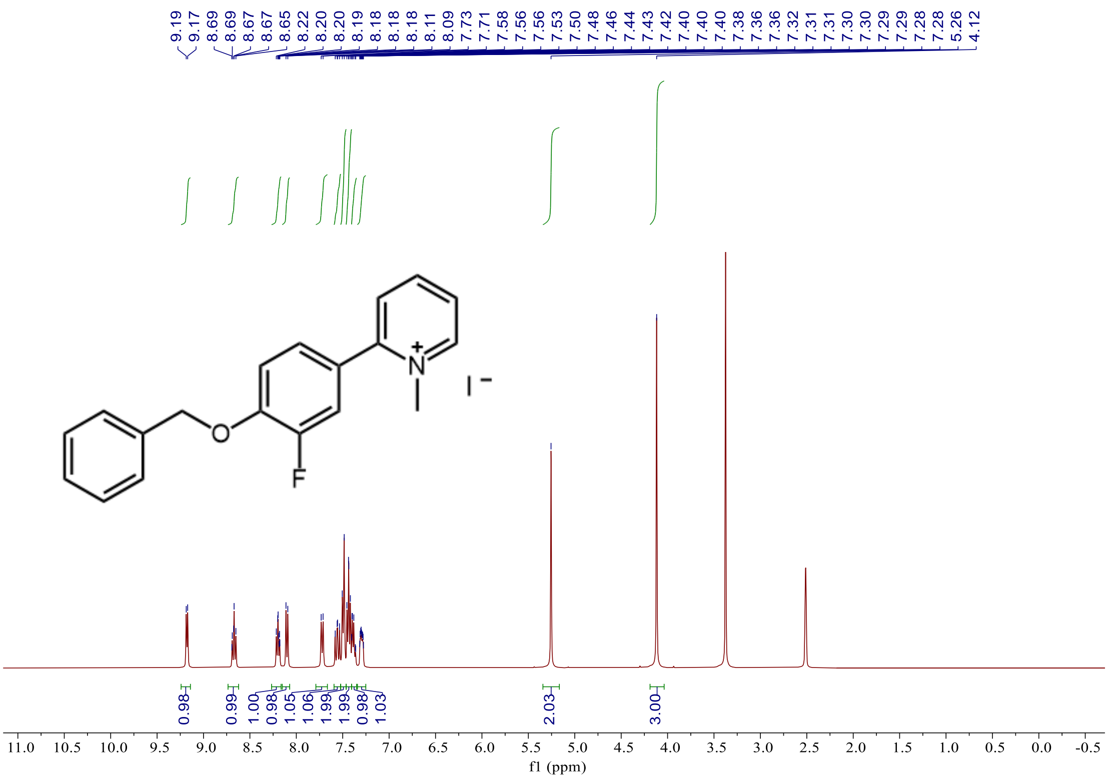
^{19}F NMR (400 MHz, DMSO-d_6) of compound **12a**



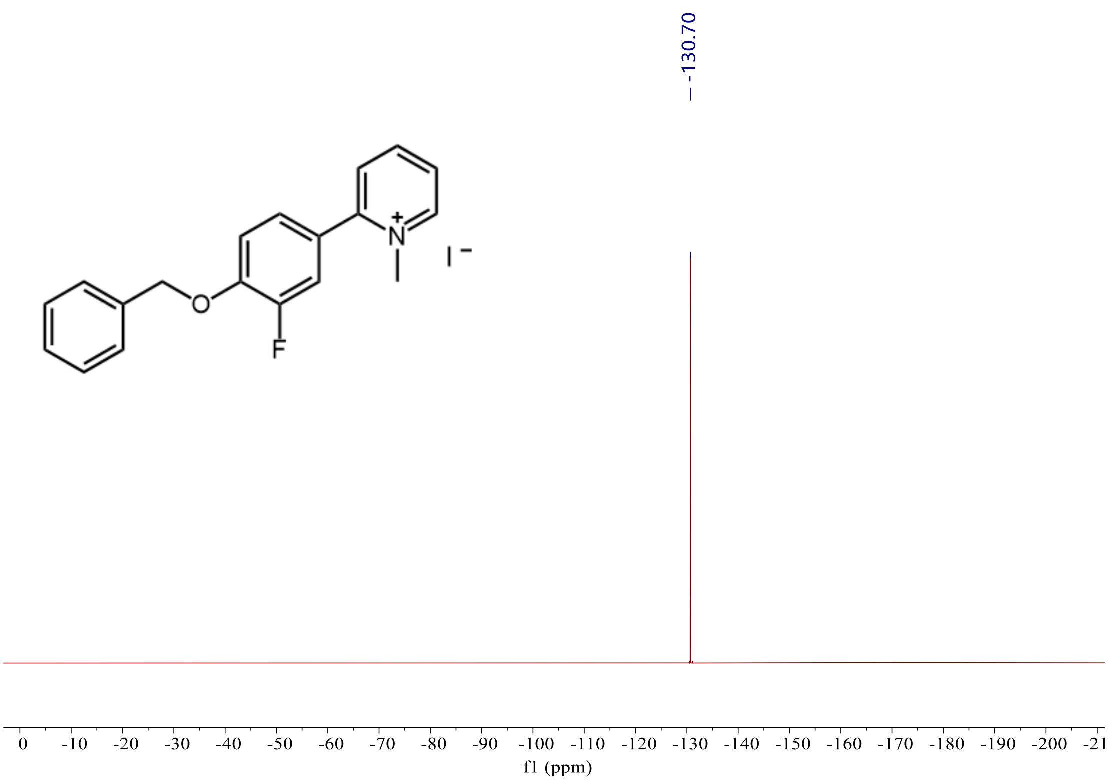
^{13}C NMR (101 MHz, DMSO-d_6) of compound **12a**



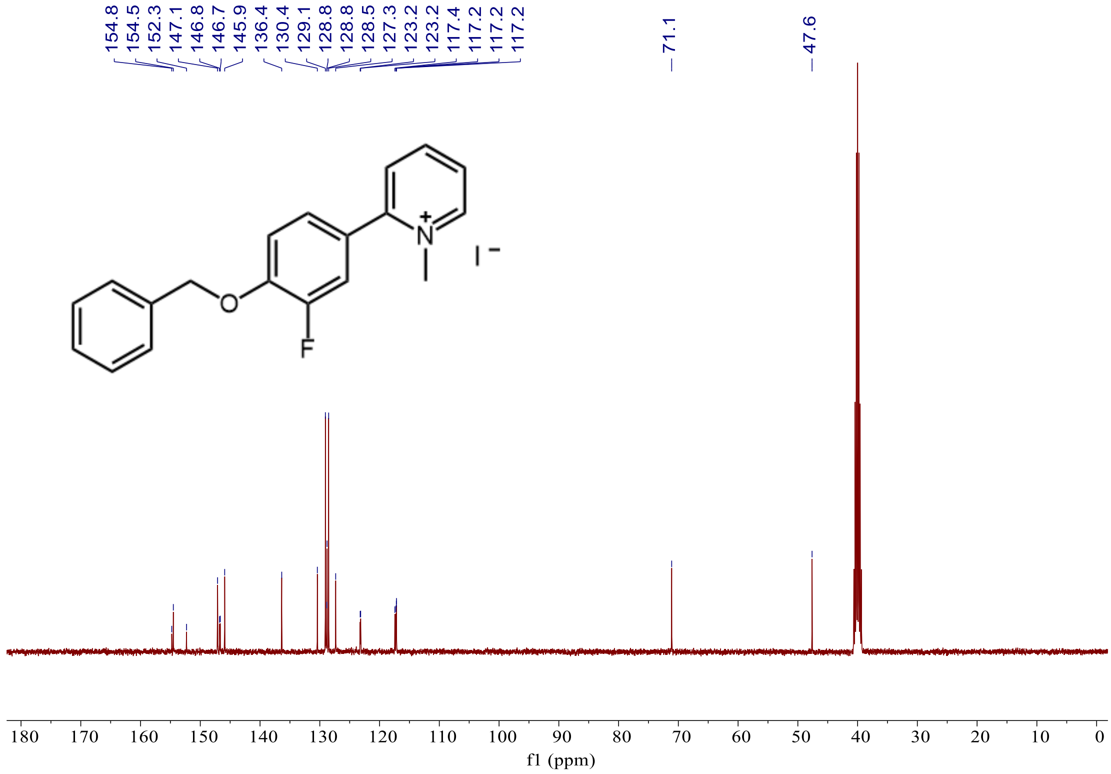
^{1}H NMR (400 MHz, DMSO-d_6) of compound **13a**



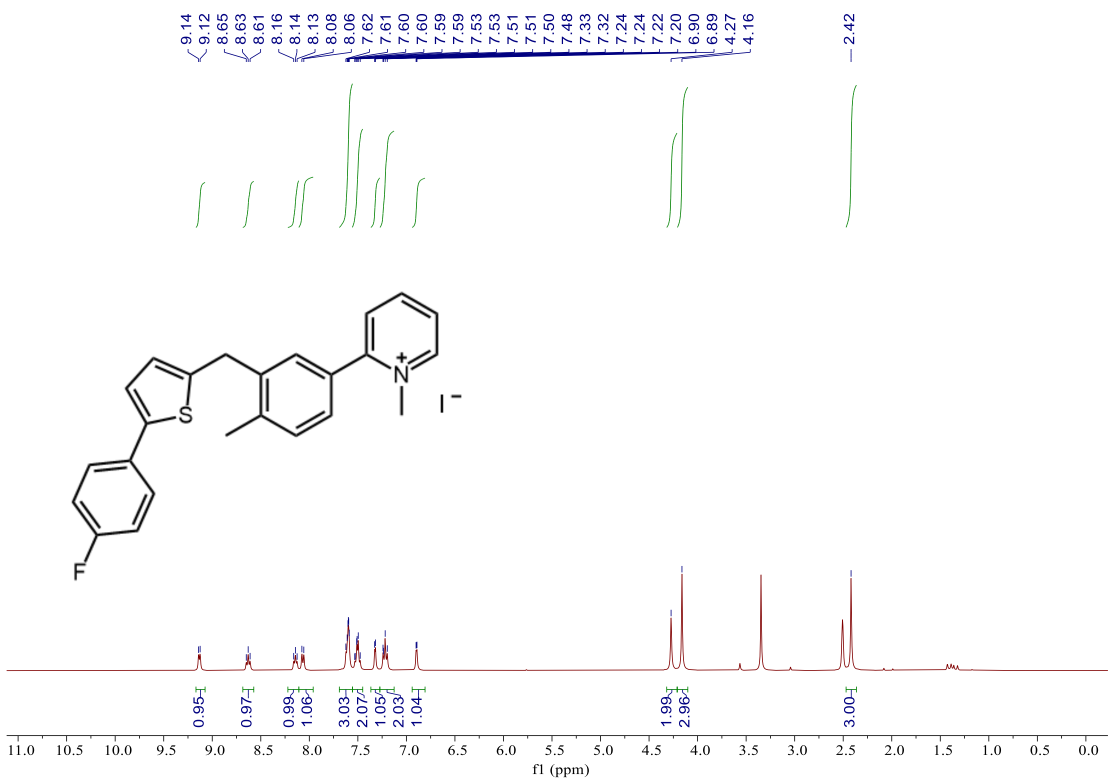
^{19}F NMR (400 MHz, DMSO-d_6) of compound **13a**



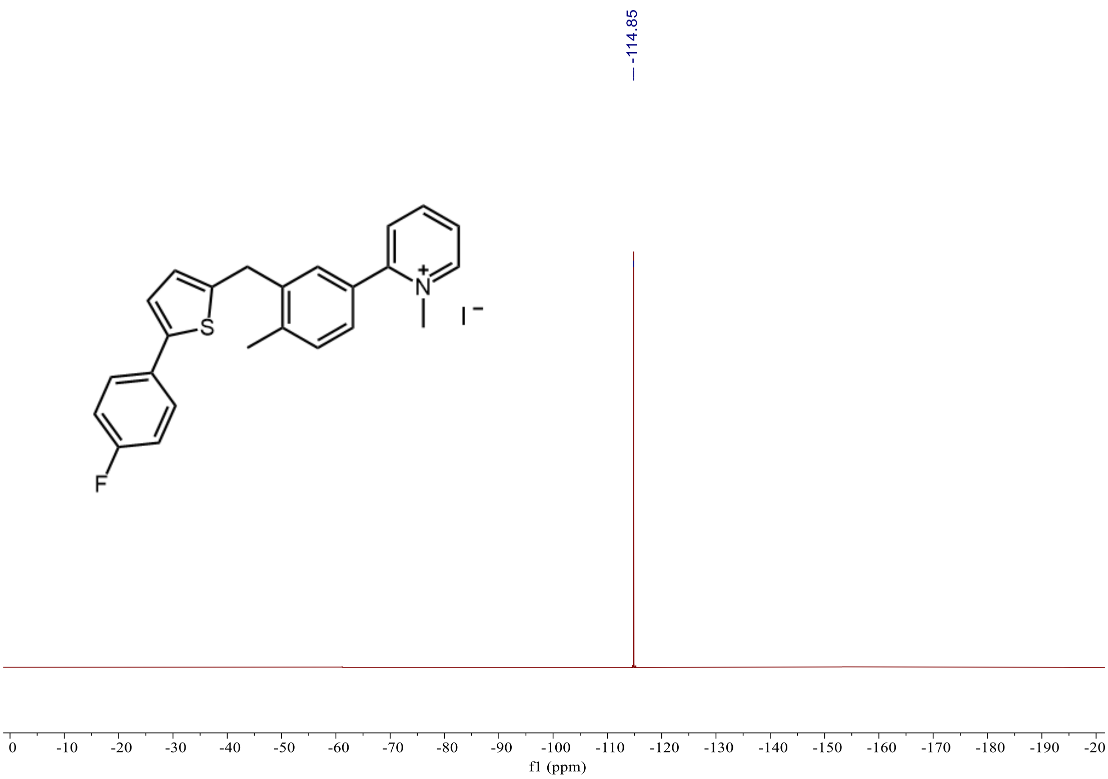
^{13}C NMR (101 MHz, DMSO-d_6) of compound **13a**



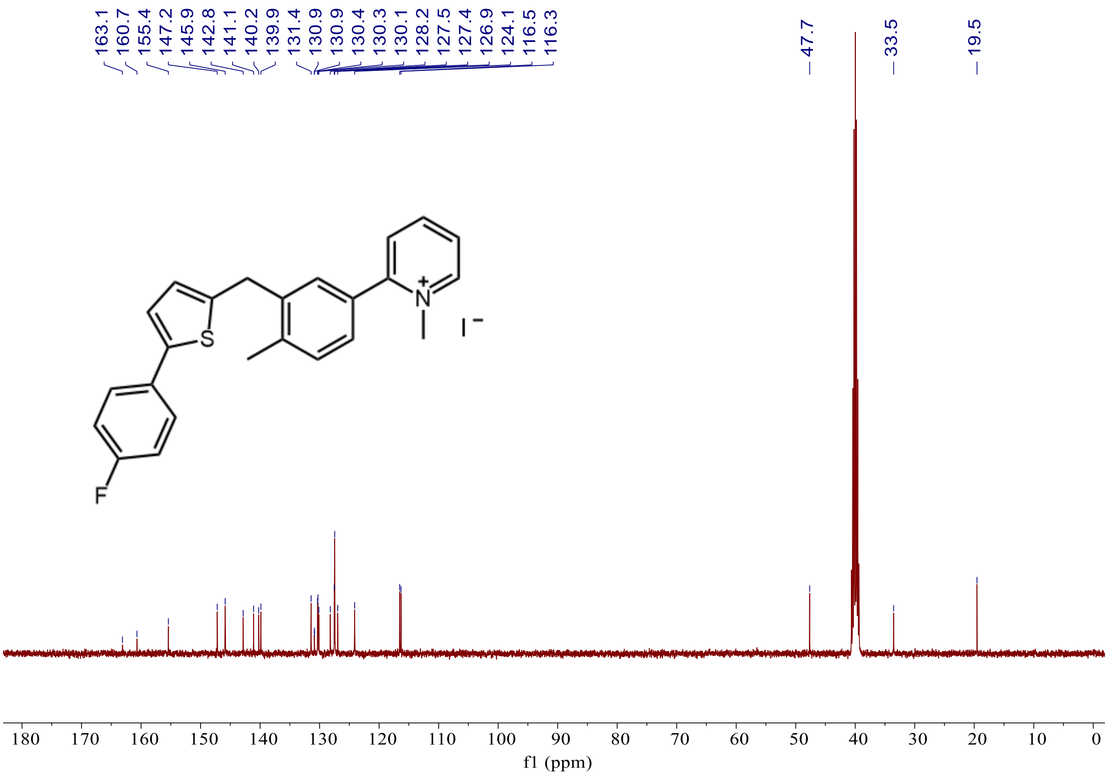
^{1}H NMR (400 MHz, DMSO-d_6) of compound **14a**



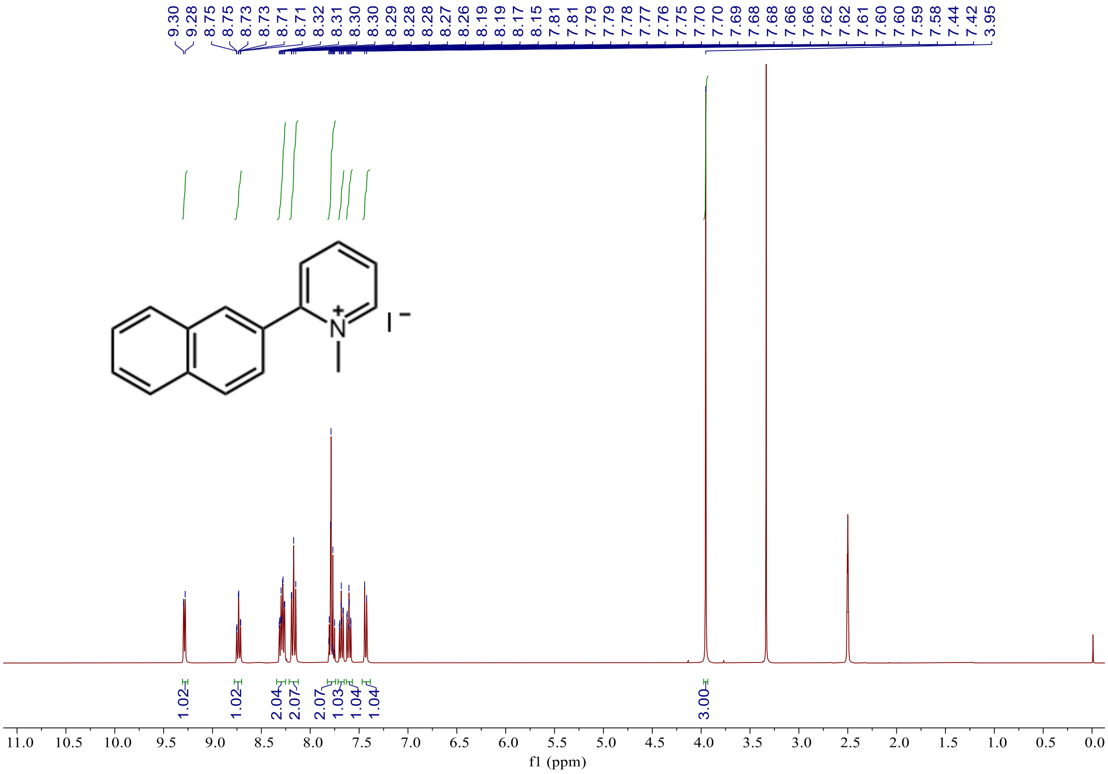
^{19}F NMR (400 MHz, DMSO-d_6) of compound **14a**



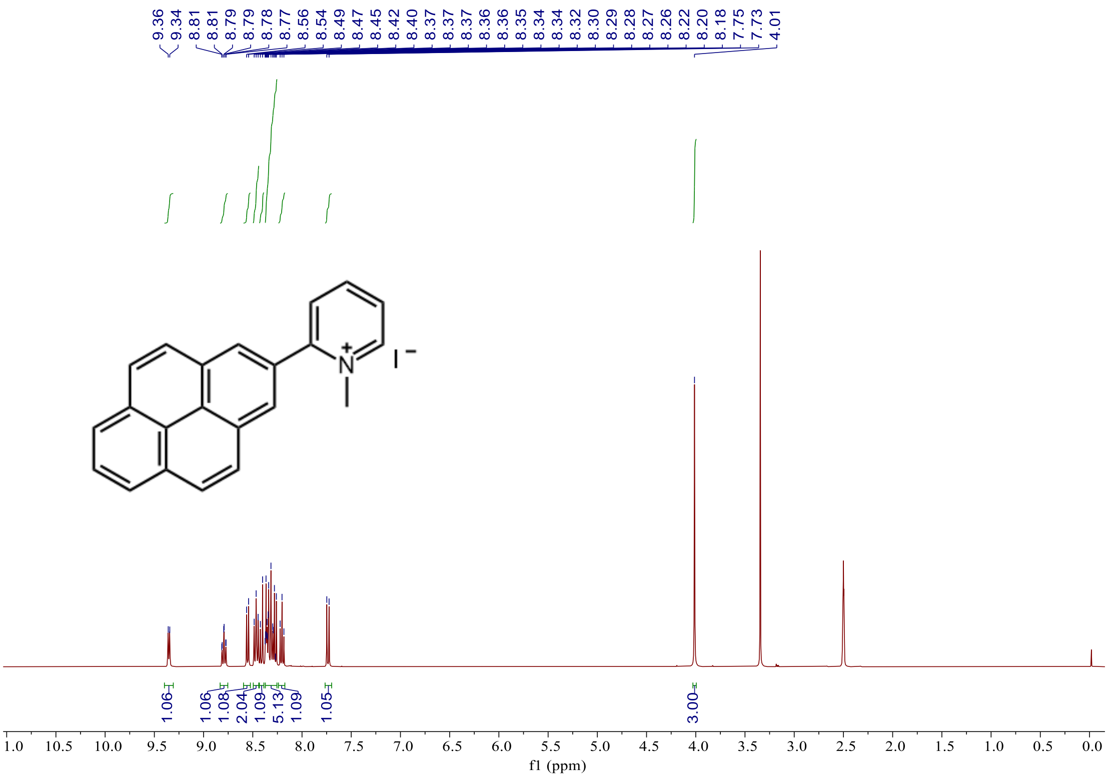
^{13}C NMR (101 MHz, DMSO-d_6) of compound **14a**



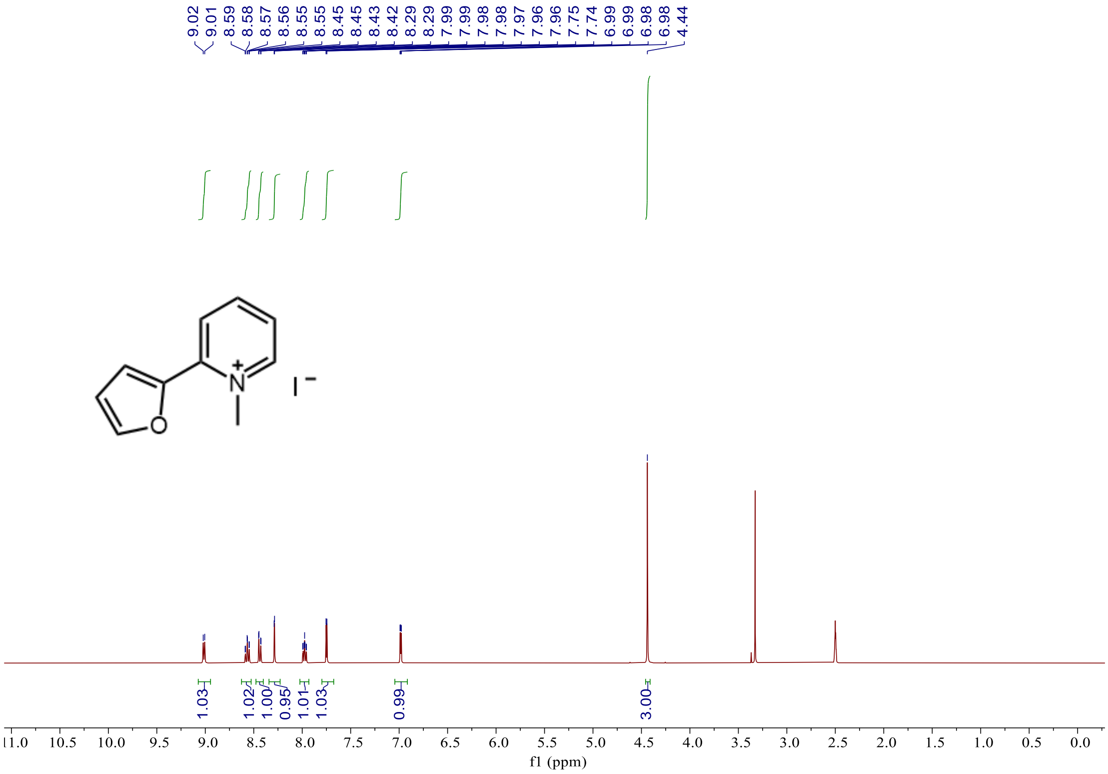
^{1}H NMR (400 MHz, DMSO-d_6) of compound **15a**



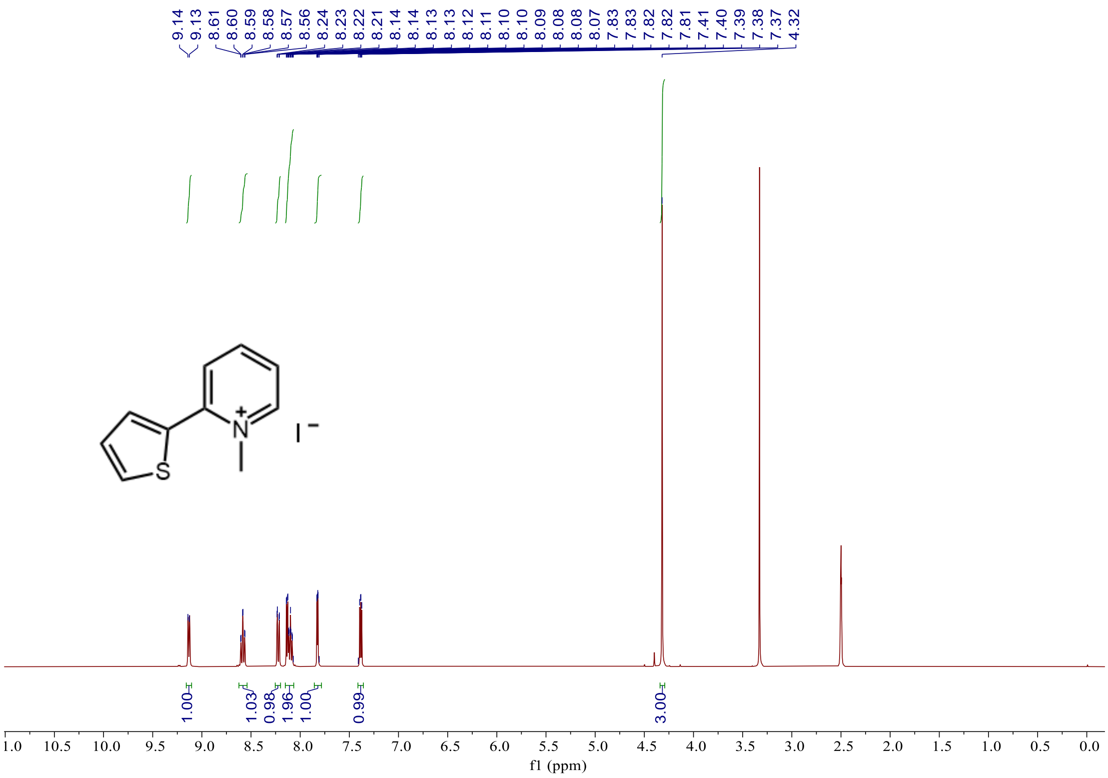
^{1}H NMR (400 MHz, DMSO-d_6) of compound **16a**



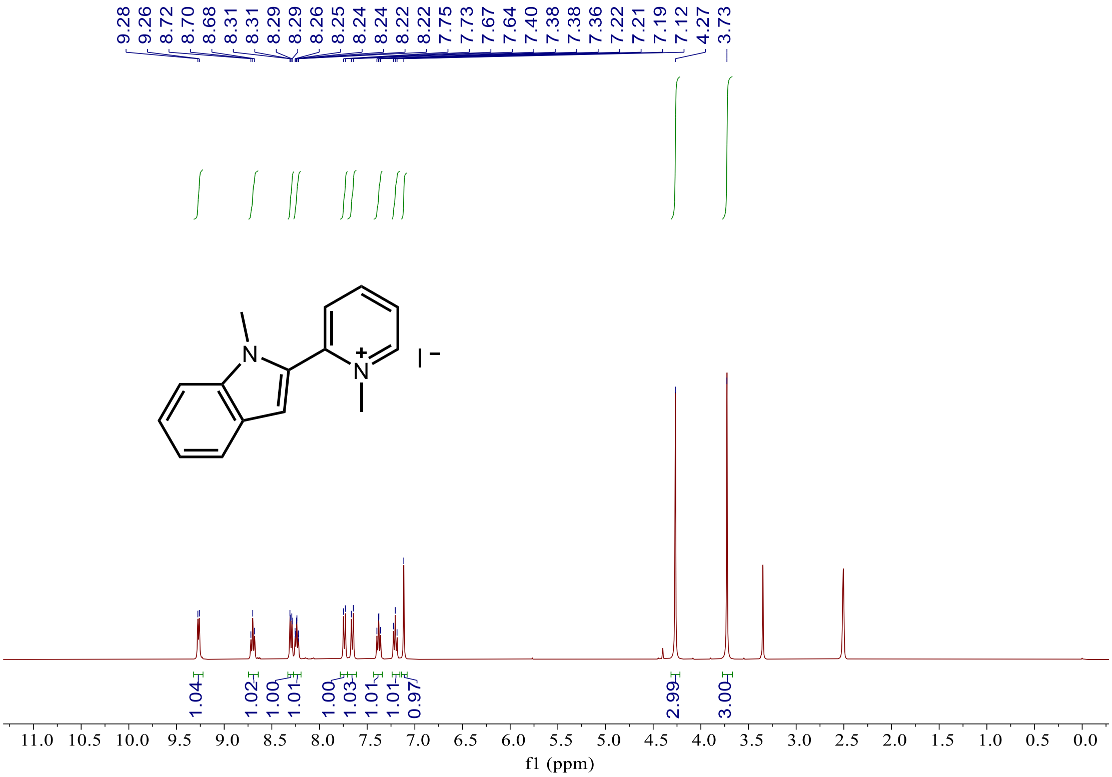
[1]H NMR (400 MHz, DMSO-d_6) of compound **17a**



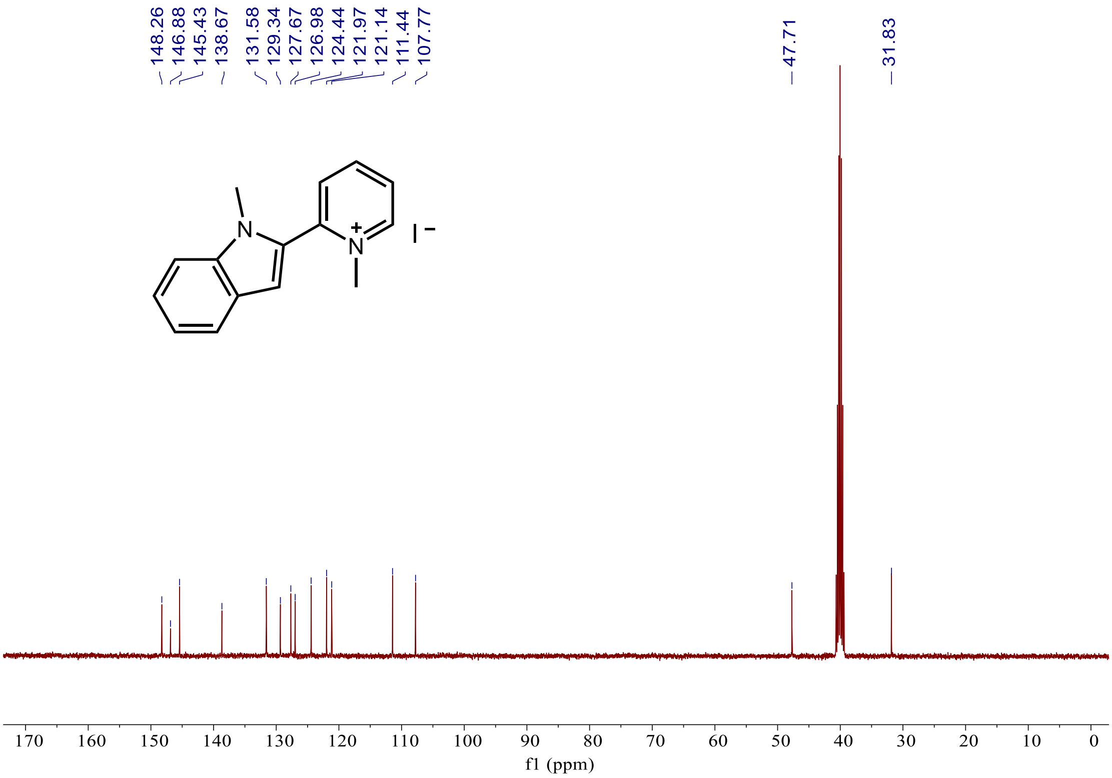
[1]H NMR (400 MHz, DMSO-d_6) of compound **18a**



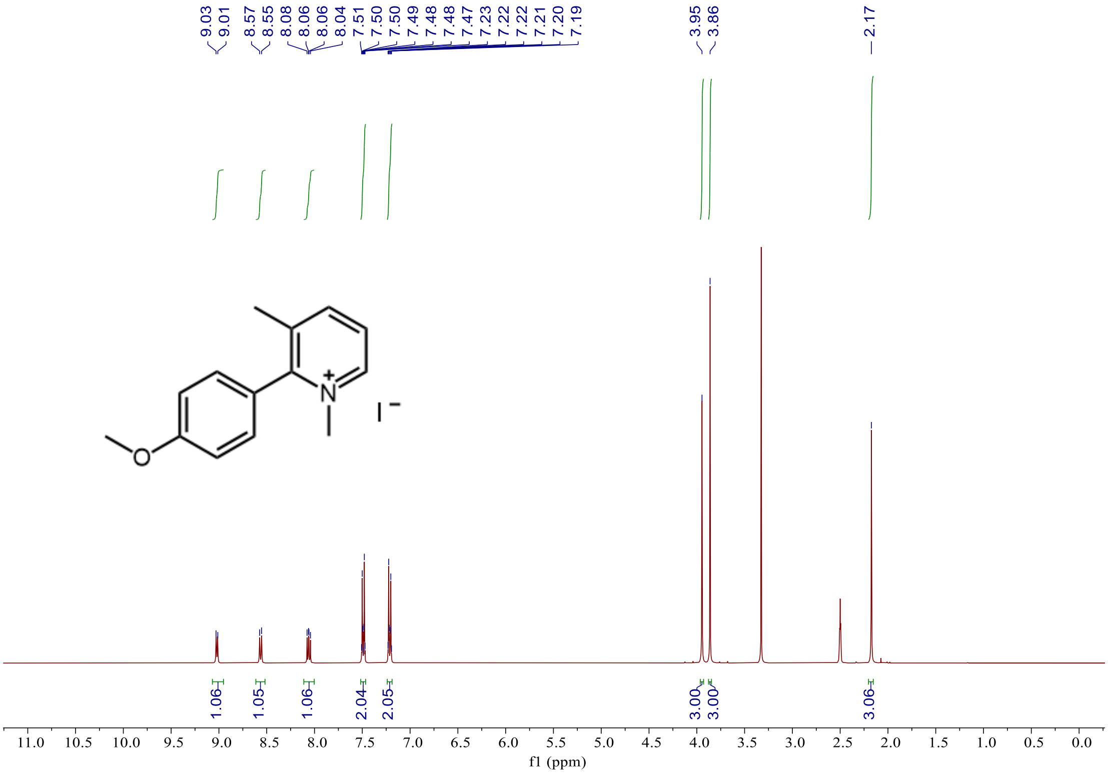
[1]H NMR (400 MHz, DMSO-d_6) of compound **19a**



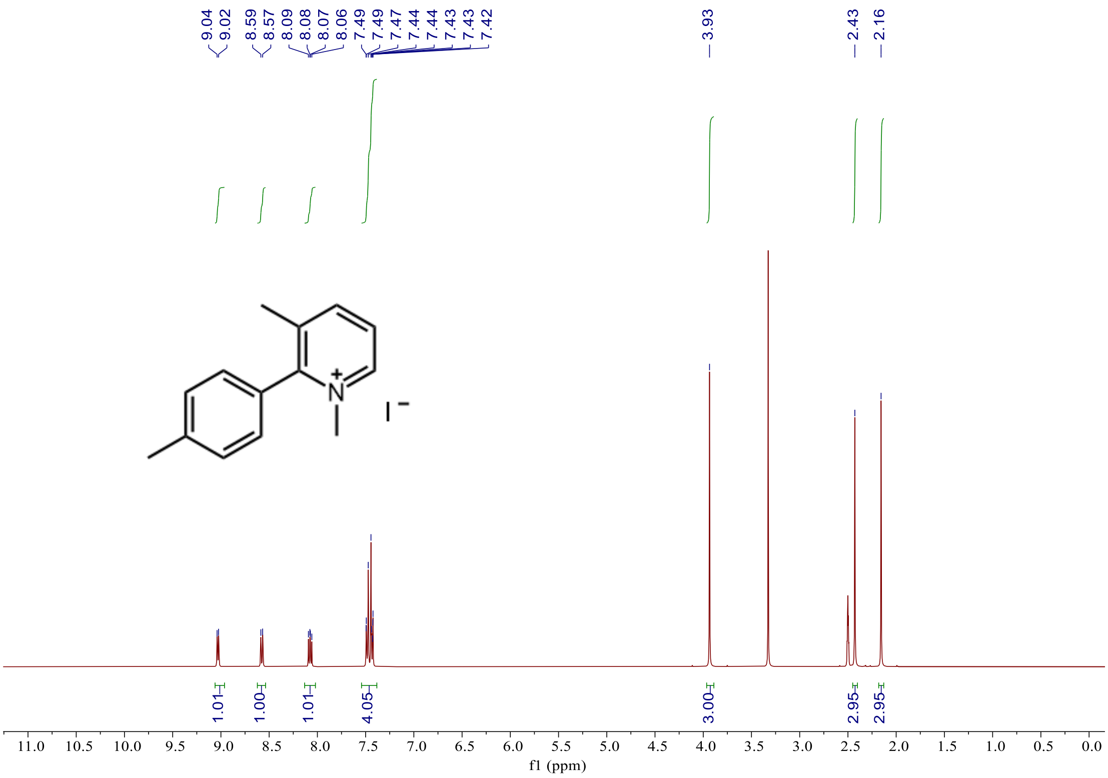
[13]C NMR (101 MHz, DMSO-d_6) of compound **19a**



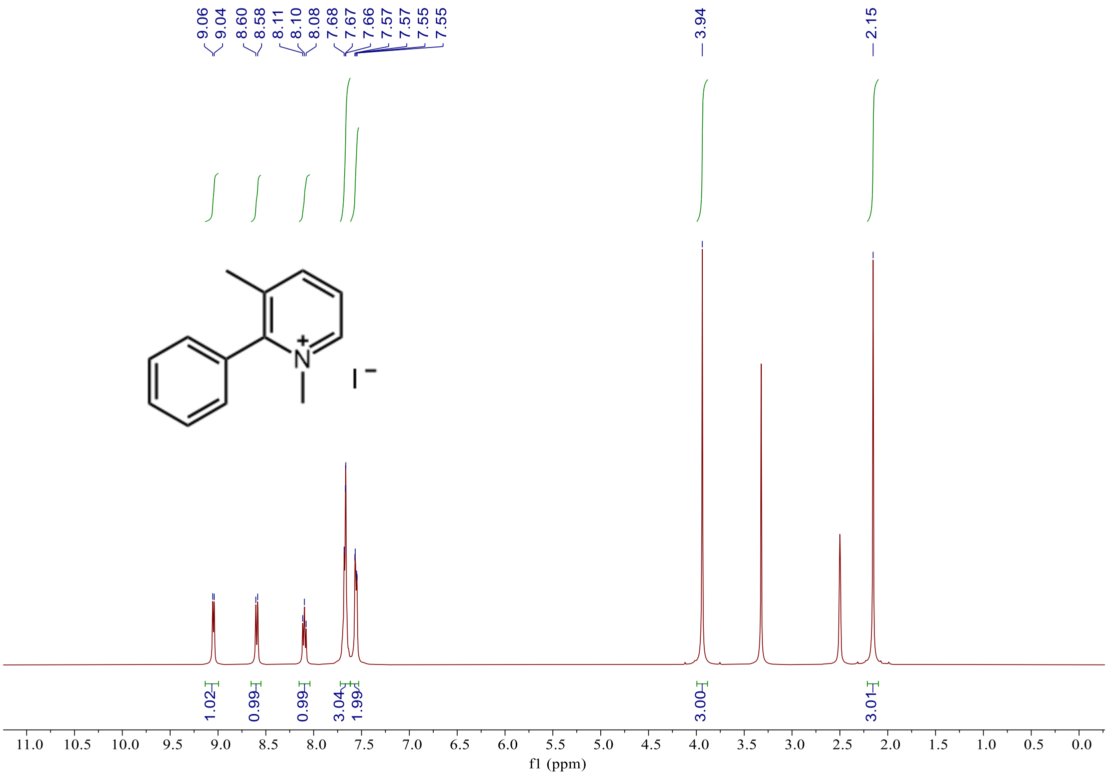
[1]H NMR (400 MHz, DMSO-d_6) of compound **20a**



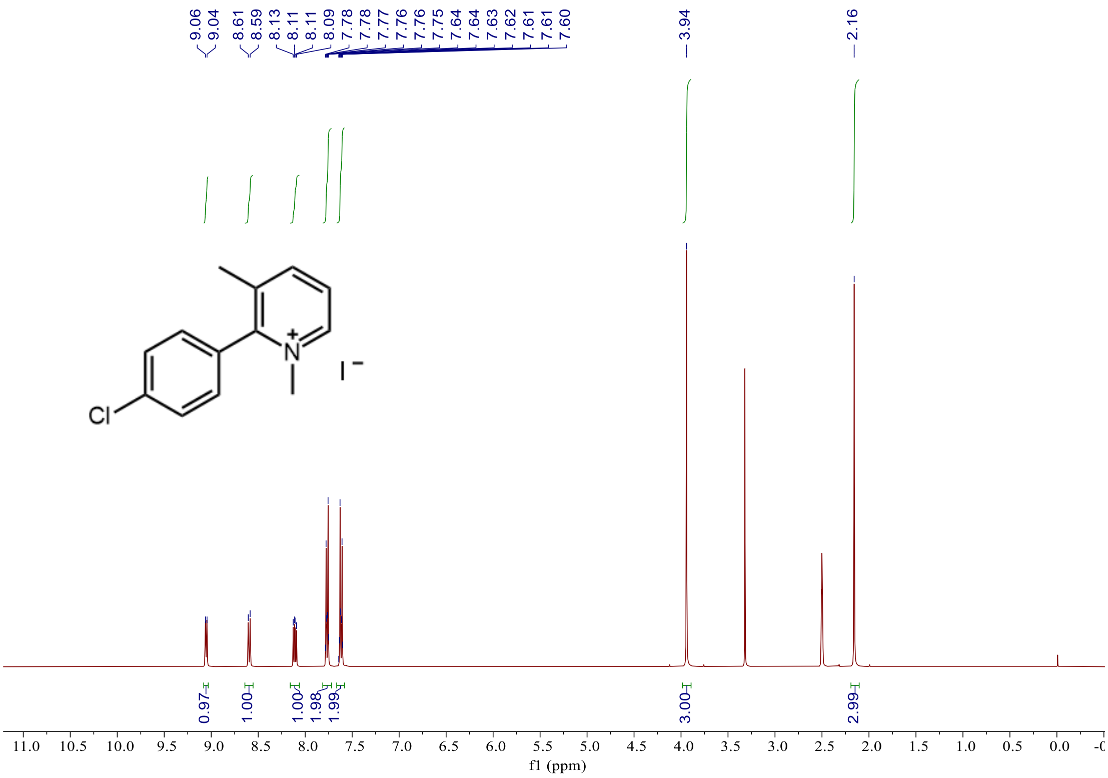
[1]H NMR (400 MHz, DMSO-d_6) of compound **21a**



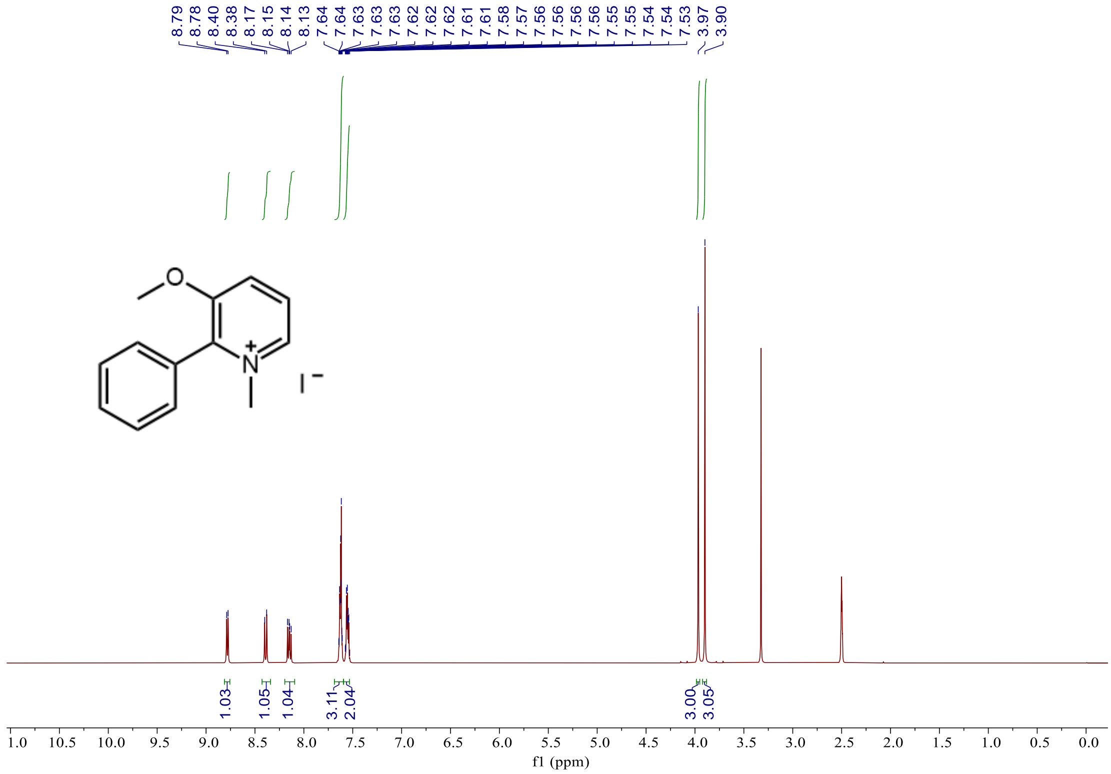
^{1}H NMR (400 MHz, DMSO-d_6) of compound **22a**



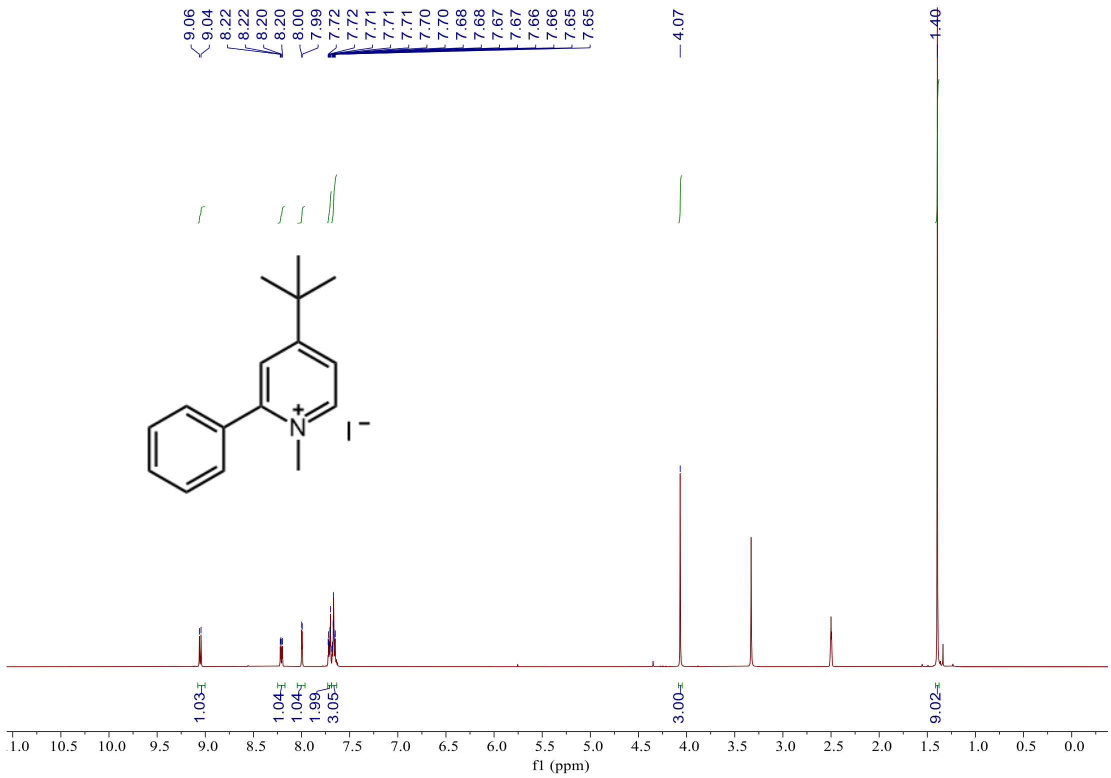
^{1}H NMR (400 MHz, DMSO-d_6) of compound **23a**



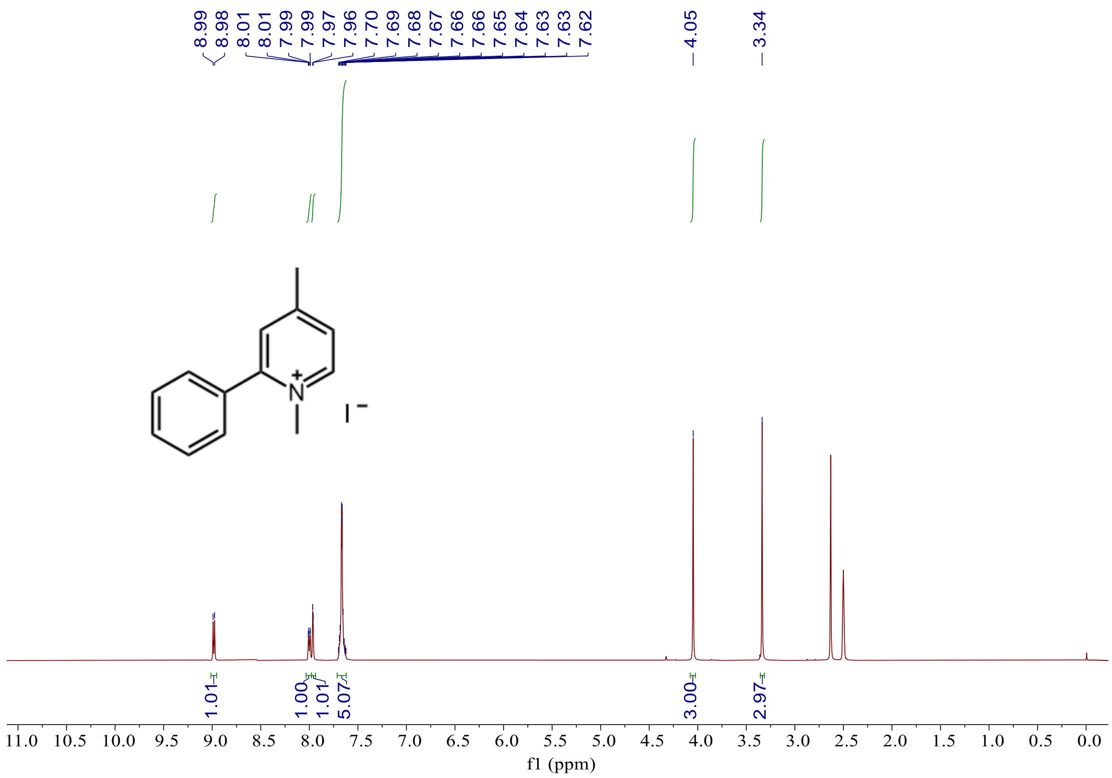
^{1}H NMR (400 MHz, DMSO-d_6) of compound **24a**



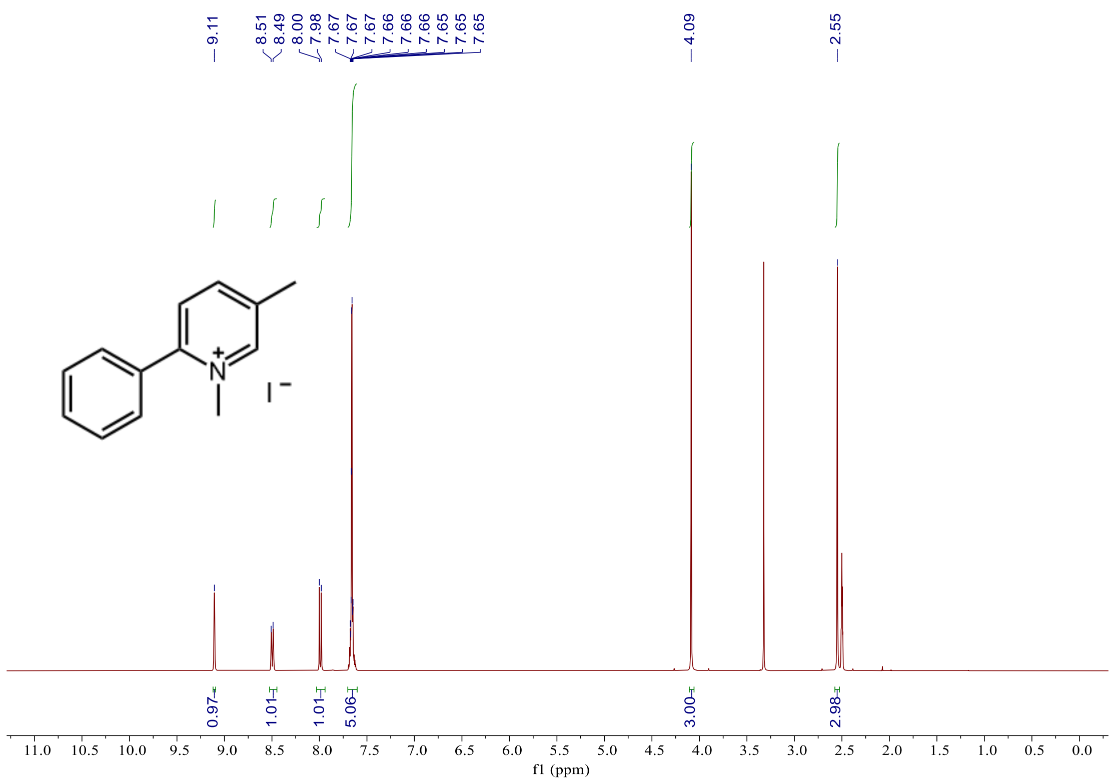
^{1}H NMR (400 MHz, DMSO-d_6) of compound **25a**



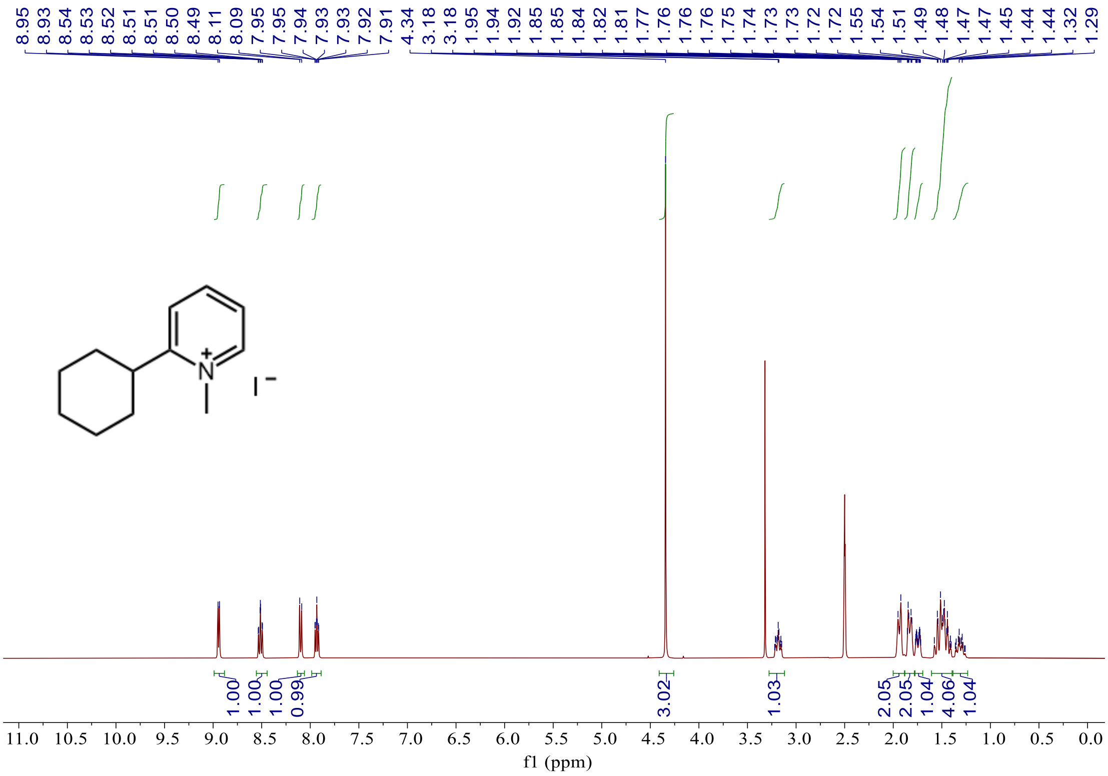
^{1}H NMR (400 MHz, DMSO-d_6) of compound **26a**



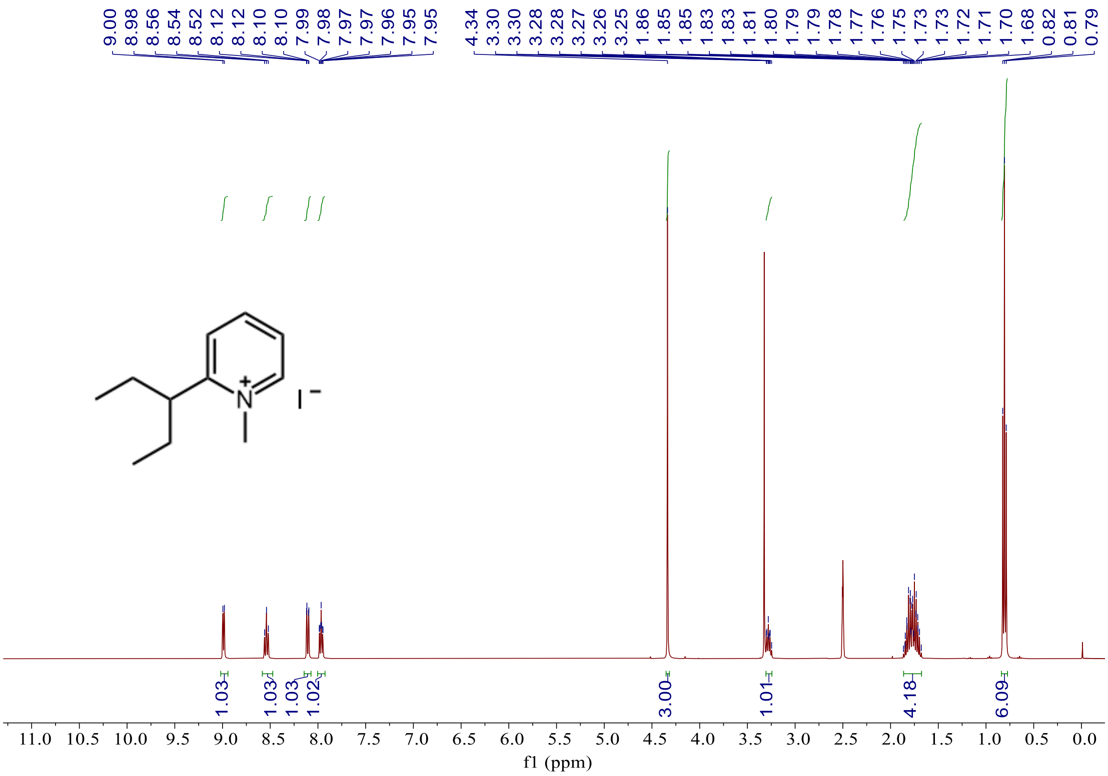
^{1}H NMR (400 MHz, DMSO-d_6) of compound **27a**



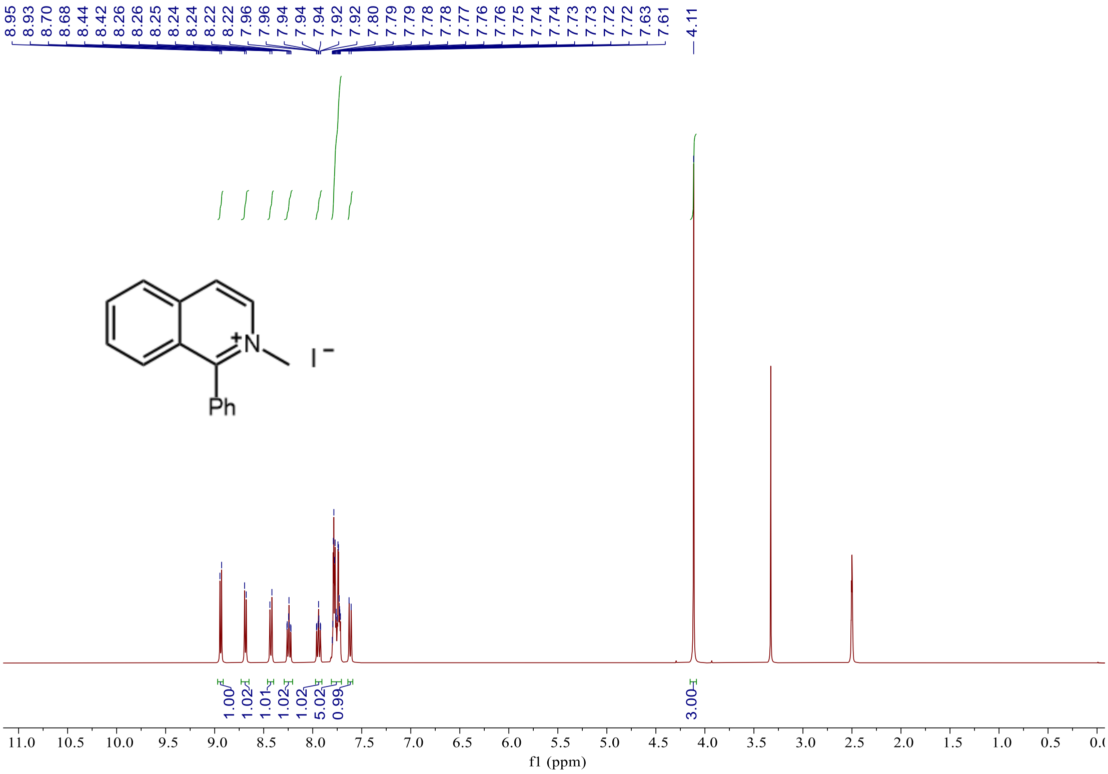
^{1}H NMR (400 MHz, DMSO-d_6) of compound **28a**



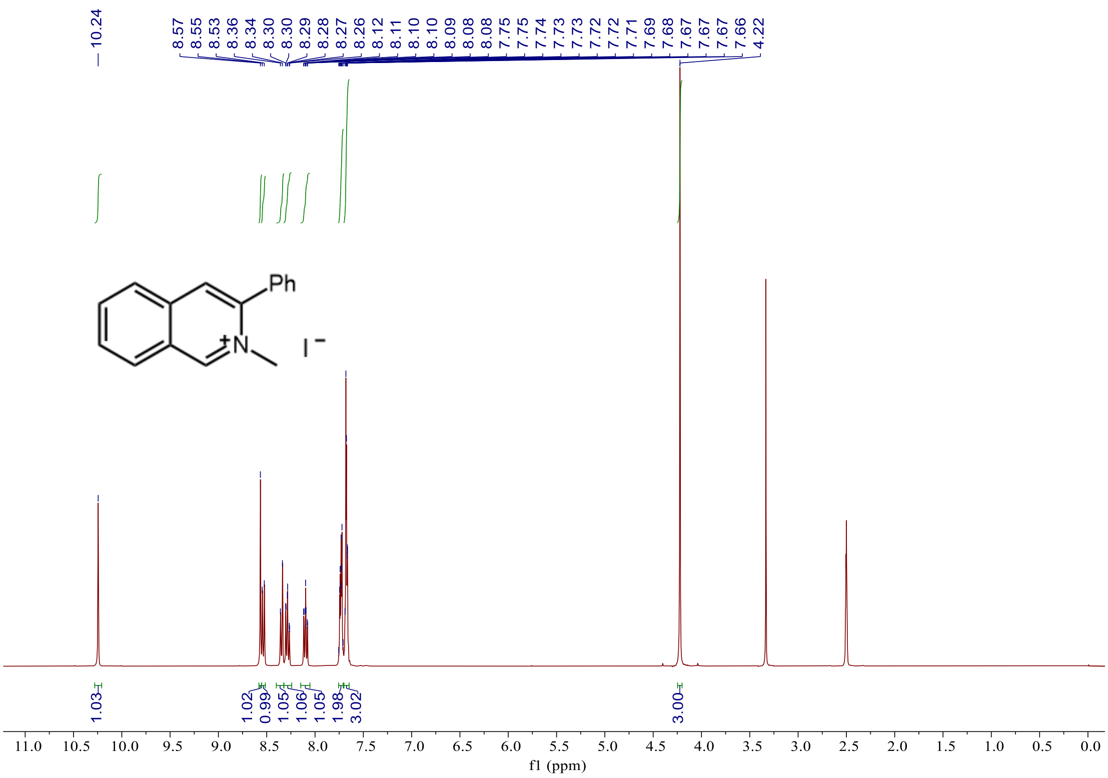
^{1}H NMR (400 MHz, DMSO-d_6) of compound **29a**



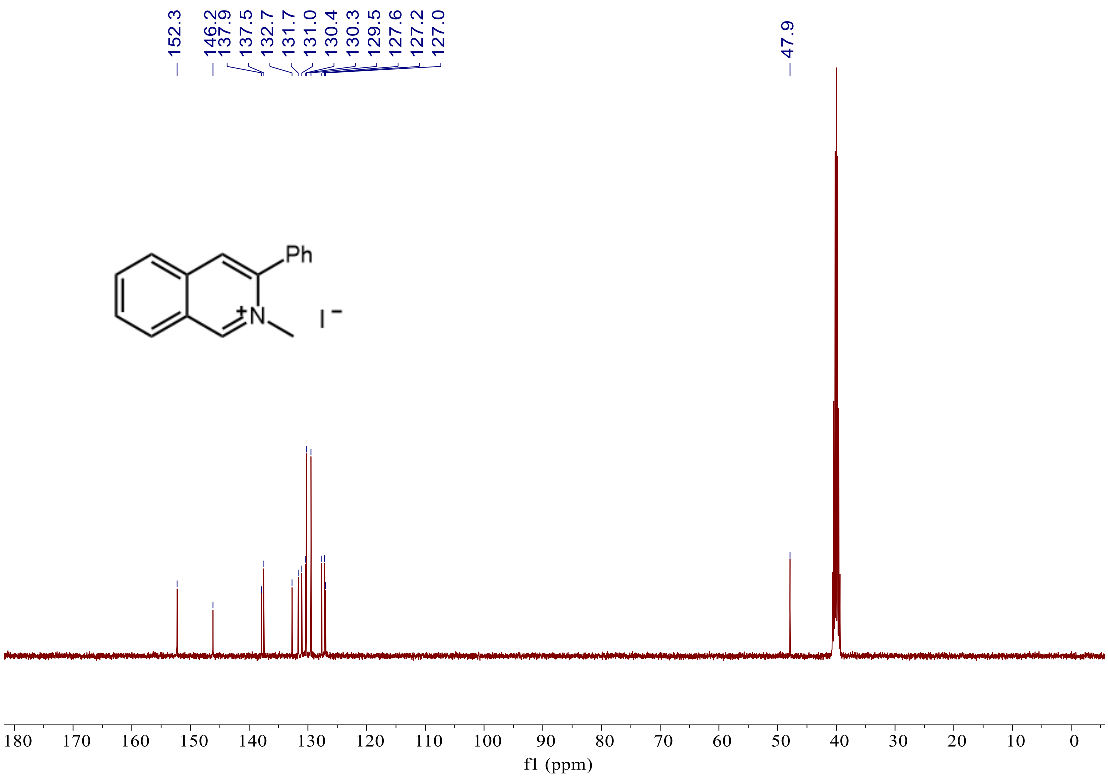
^{1}H NMR (400 MHz, DMSO-d_6) of compound **30a**



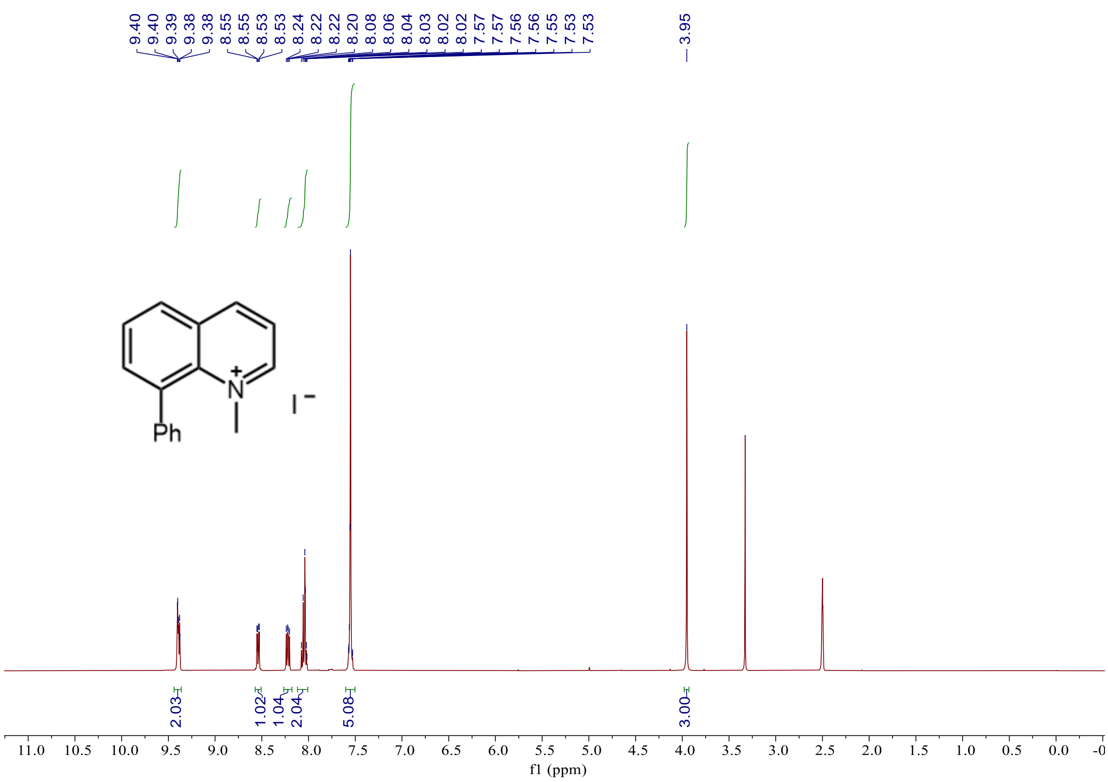
^{1}H NMR (400 MHz, DMSO-d_6) of compound **31a**



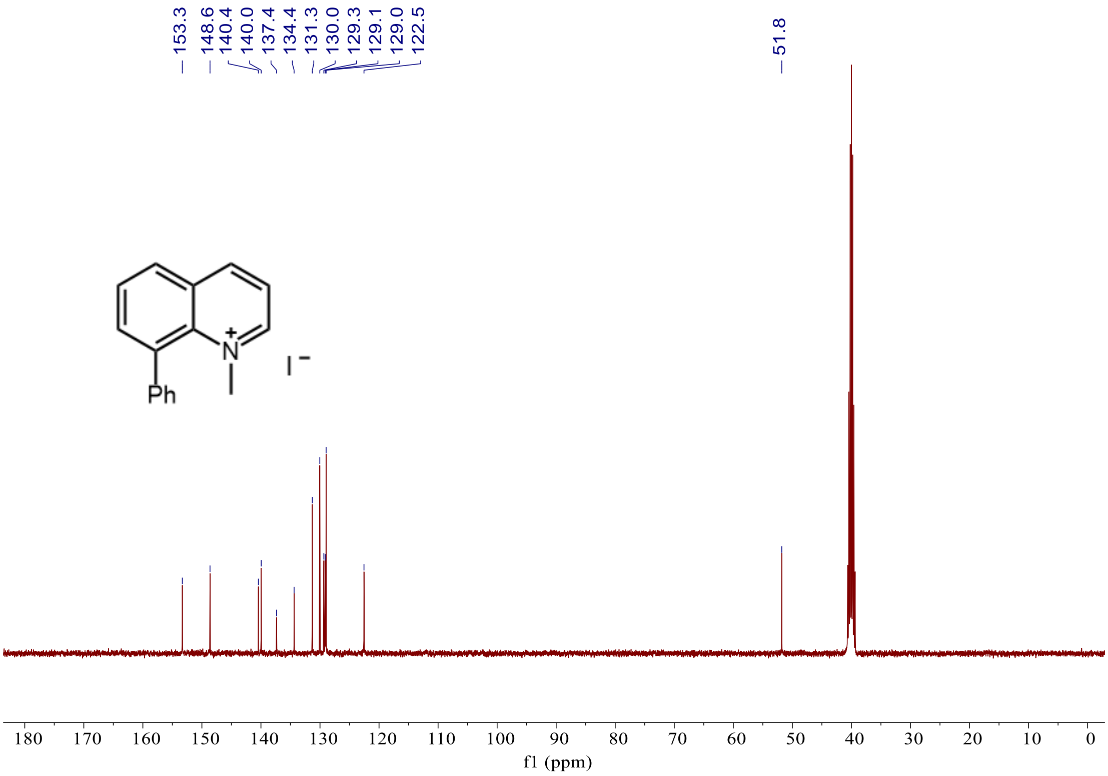
^{13}C NMR (101 MHz, DMSO-d_6) of compound **31a**



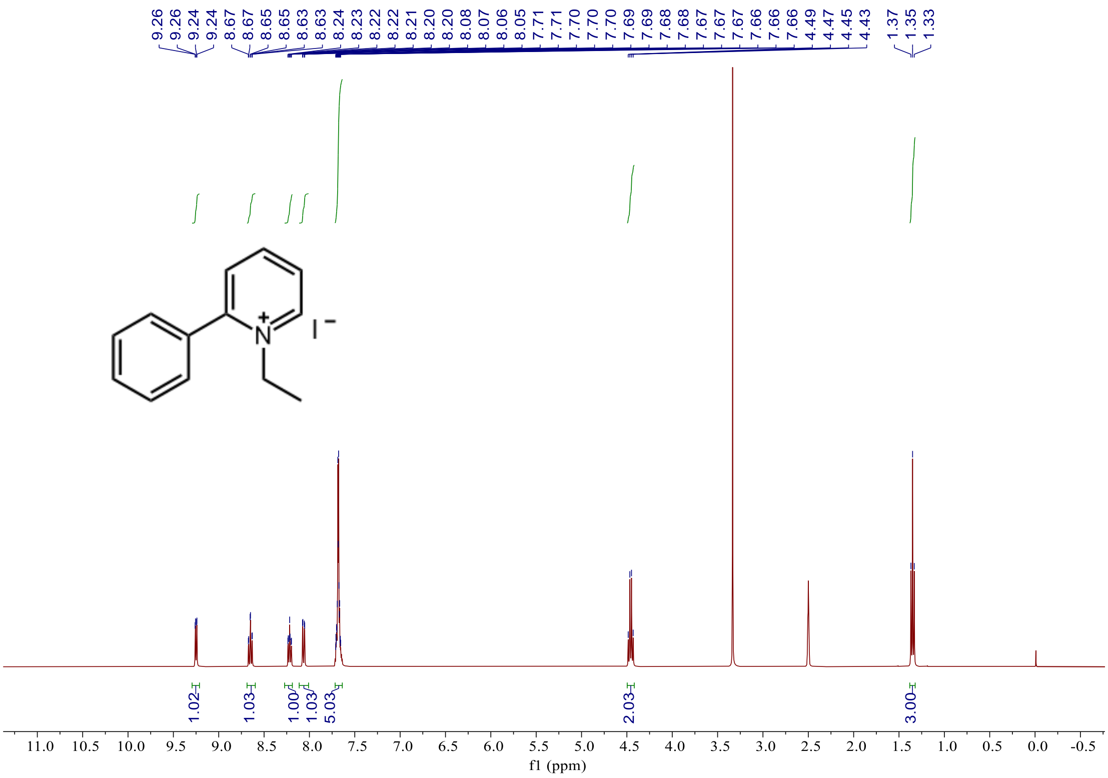
^{1}H NMR (400 MHz, DMSO-d_6) of compound **32a**



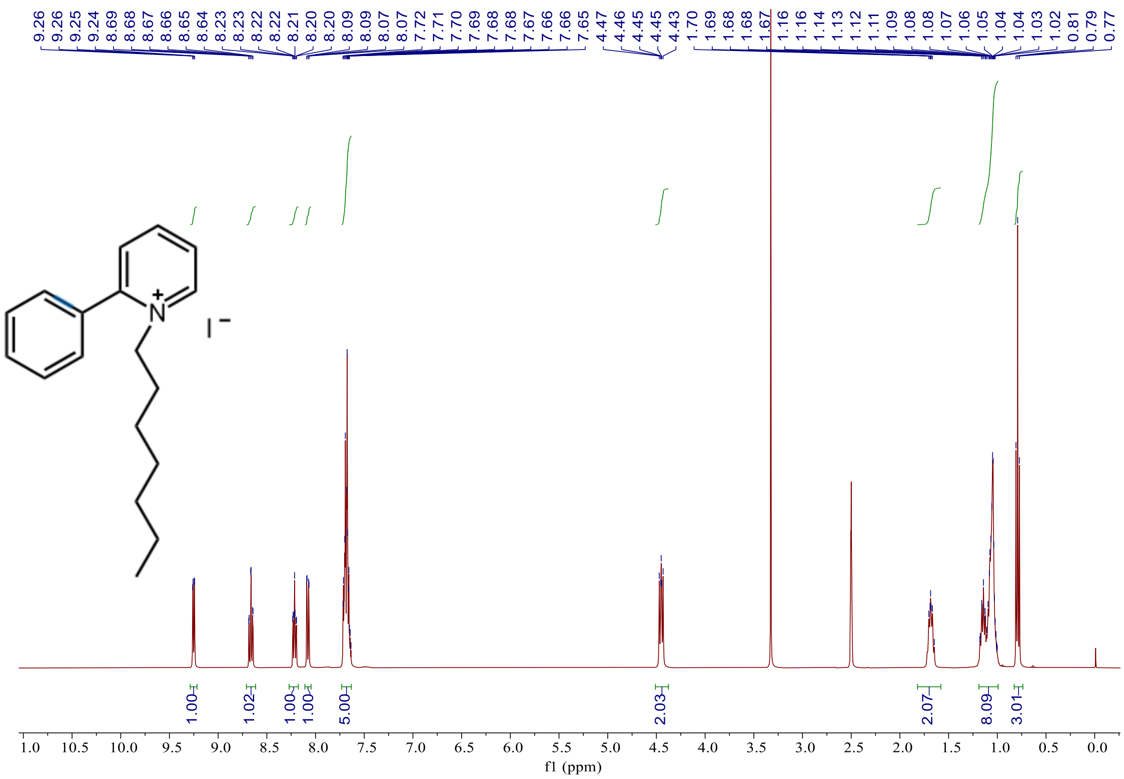
^{13}C NMR (101 MHz, DMSO-d_6) of compound **32a**



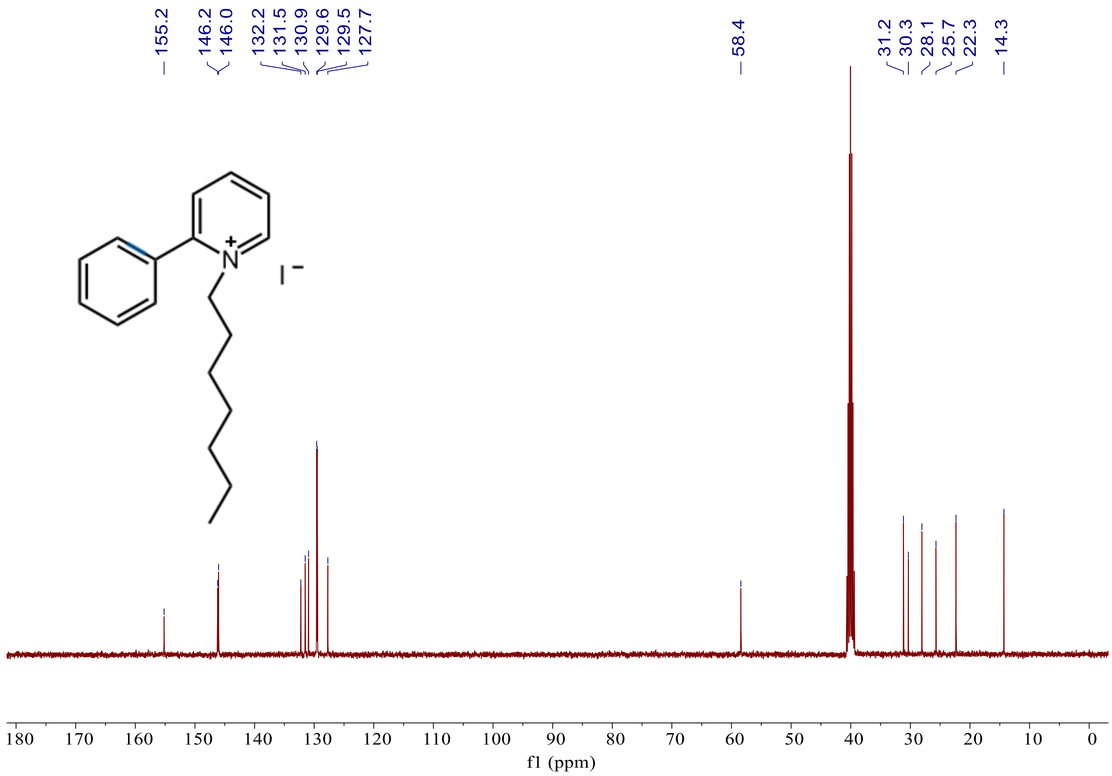
^{1}H NMR (400 MHz, DMSO-d_6) of compound **33a**



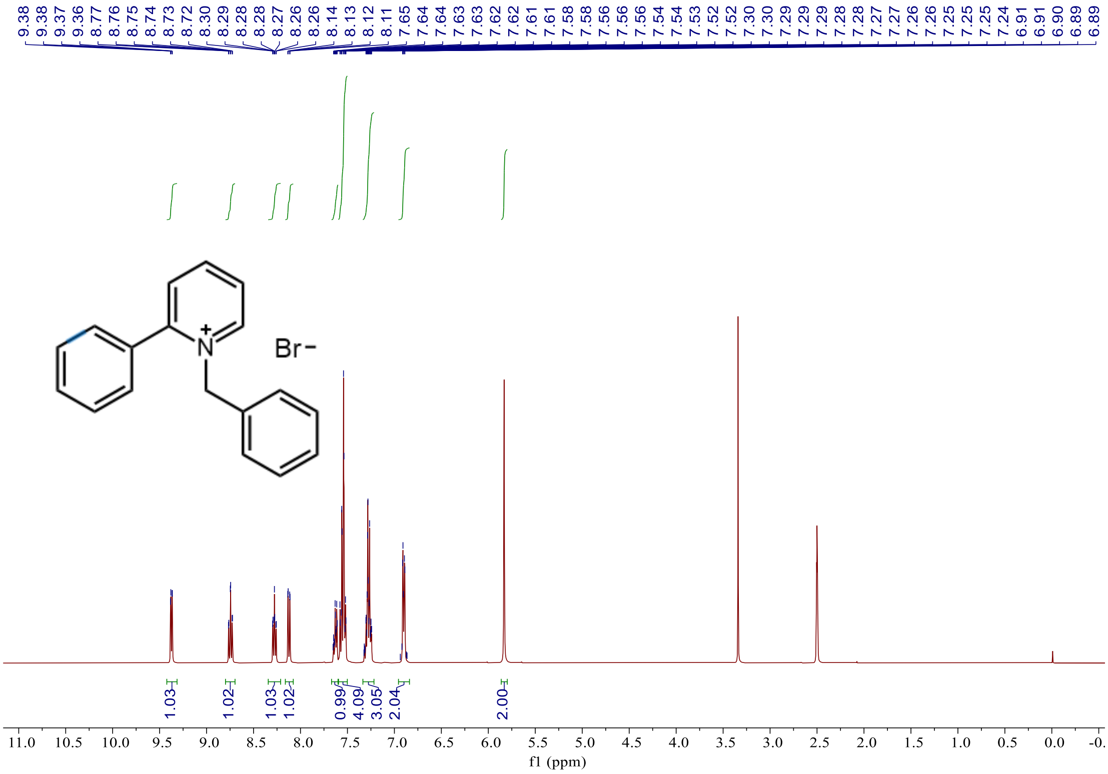
[1]H NMR (400 MHz, DMSO-d_6) of compound **34a**



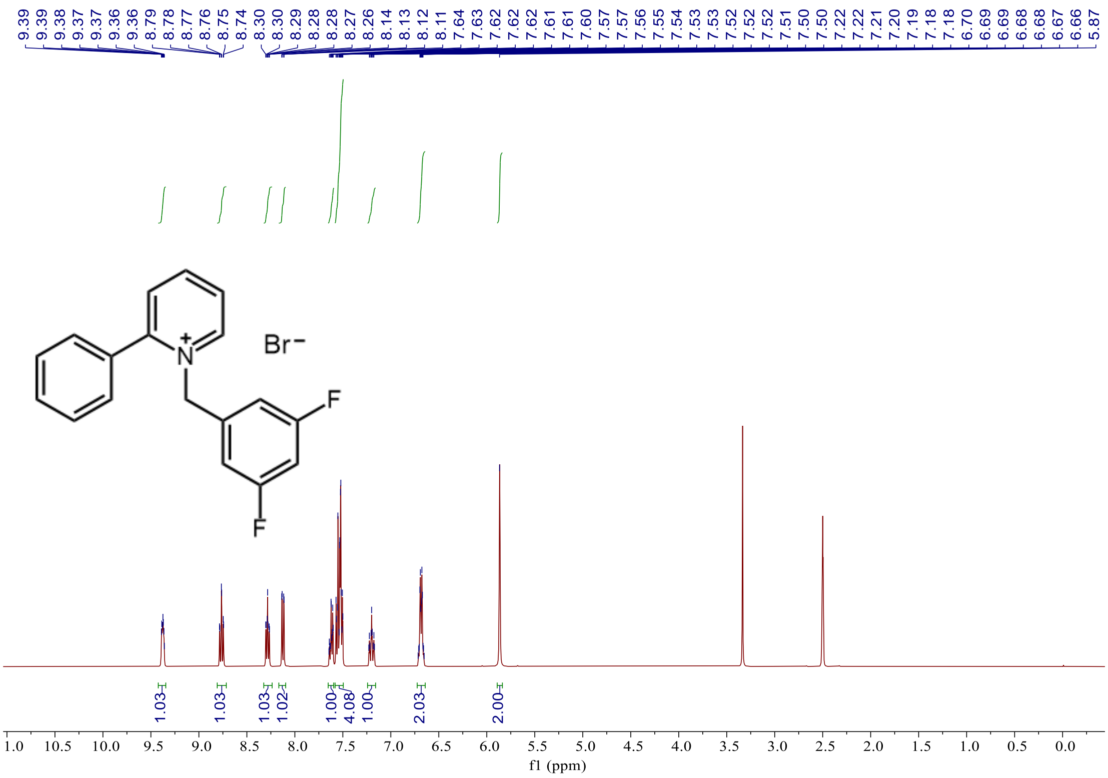
[13]C NMR (101 MHz, DMSO-d_6) of compound **34a**



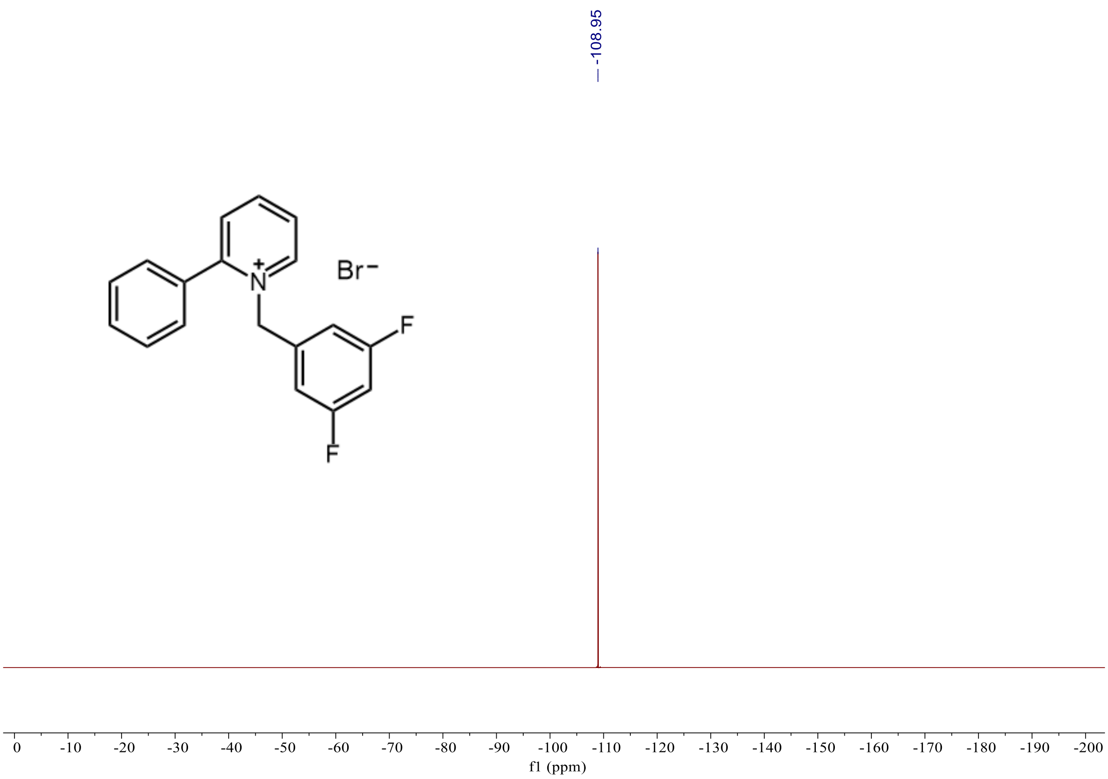
[1]H NMR (400 MHz, DMSO-d_6) of compound **35a**



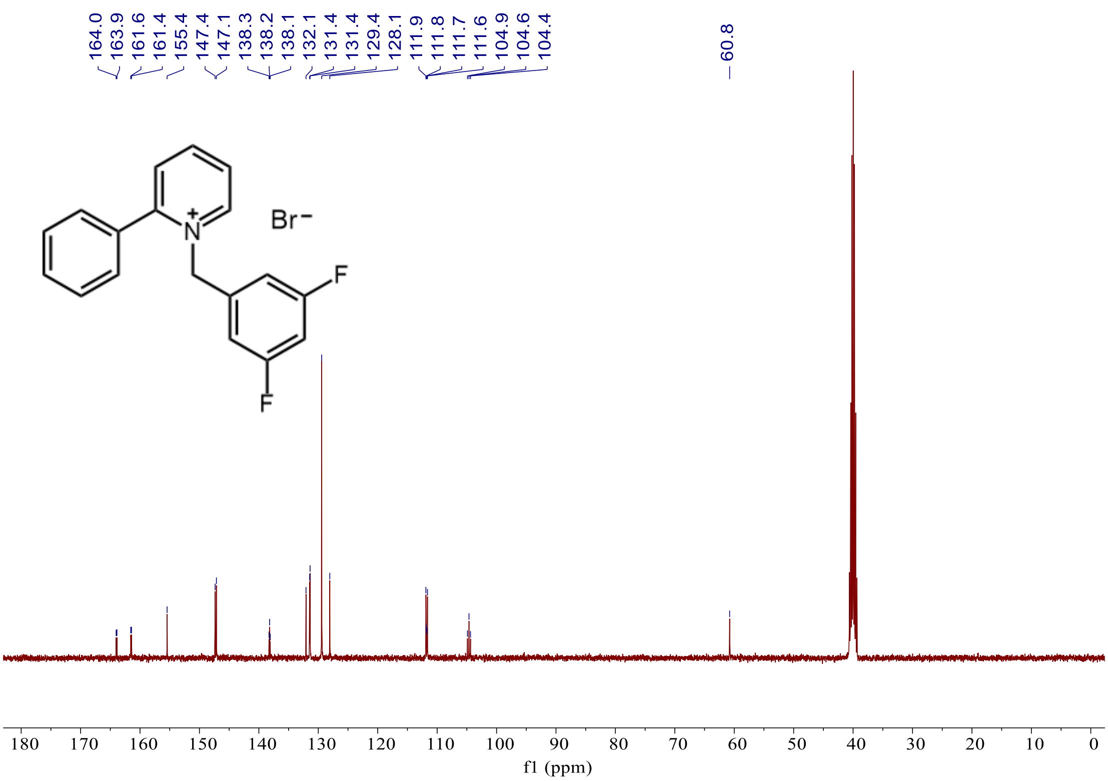
[1]H NMR (400 MHz, DMSO-d_6) of compound **36a**



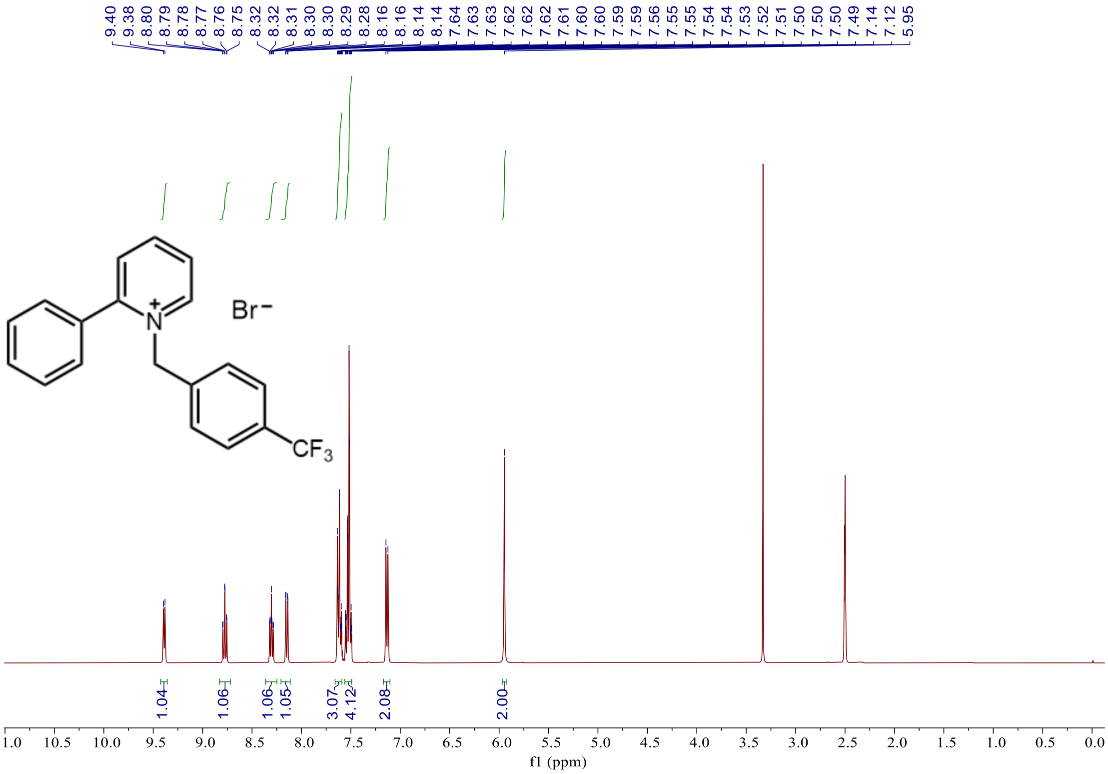
^{19}F NMR (400 MHz, DMSO-d_6) of compound **36a**



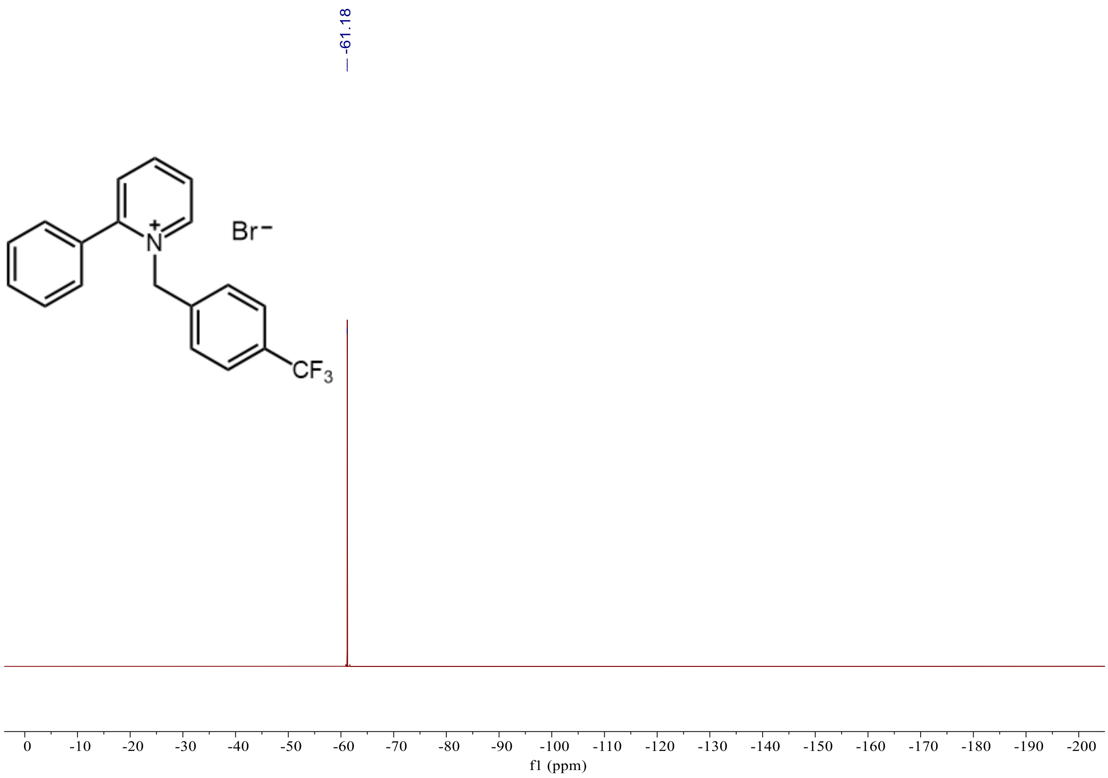
^{13}C NMR (101 MHz, DMSO-d_6) of compound **36a**



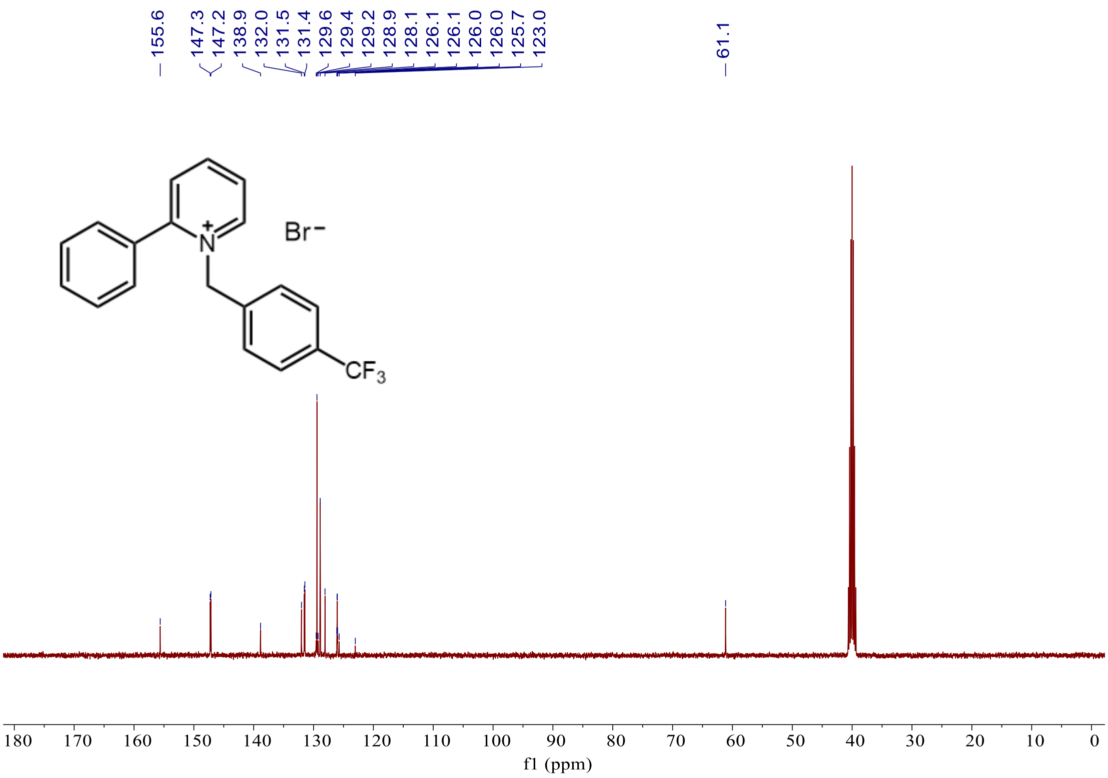
^{1}H NMR (400 MHz, DMSO-d_6) of compound **37a**



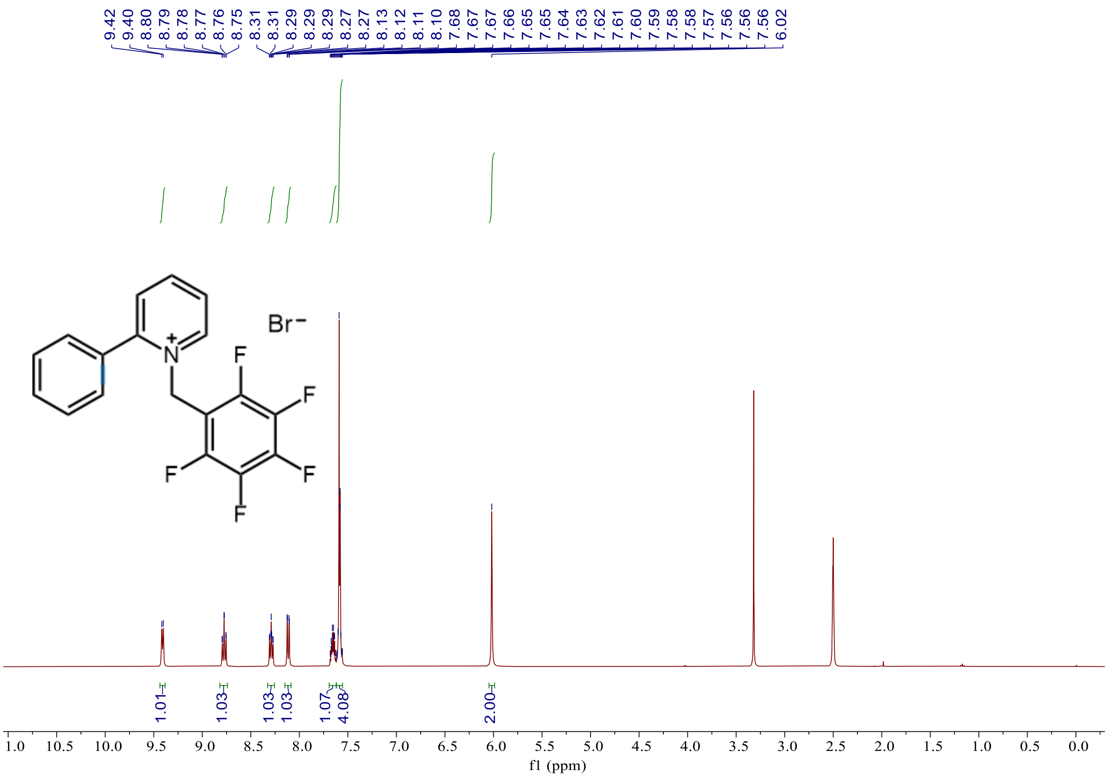
^{19}F NMR (400 MHz, DMSO-d_6) of compound **37a**



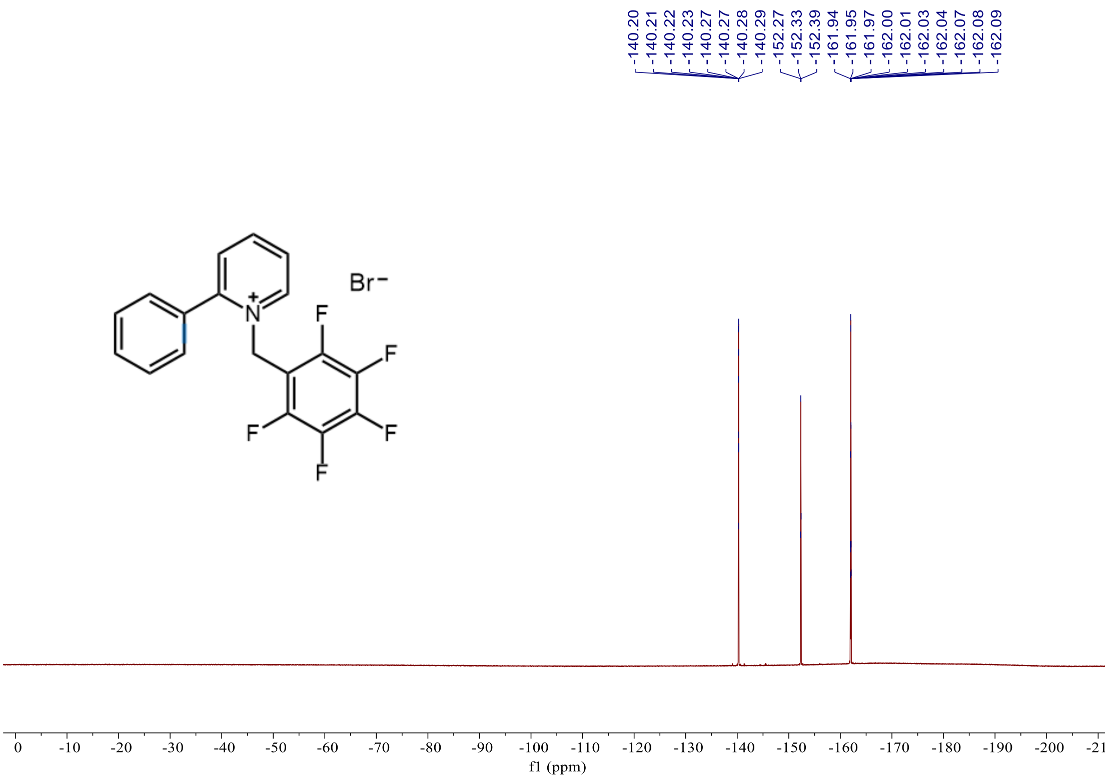
^{13}C NMR (101 MHz, DMSO-d_6) of compound **37a**



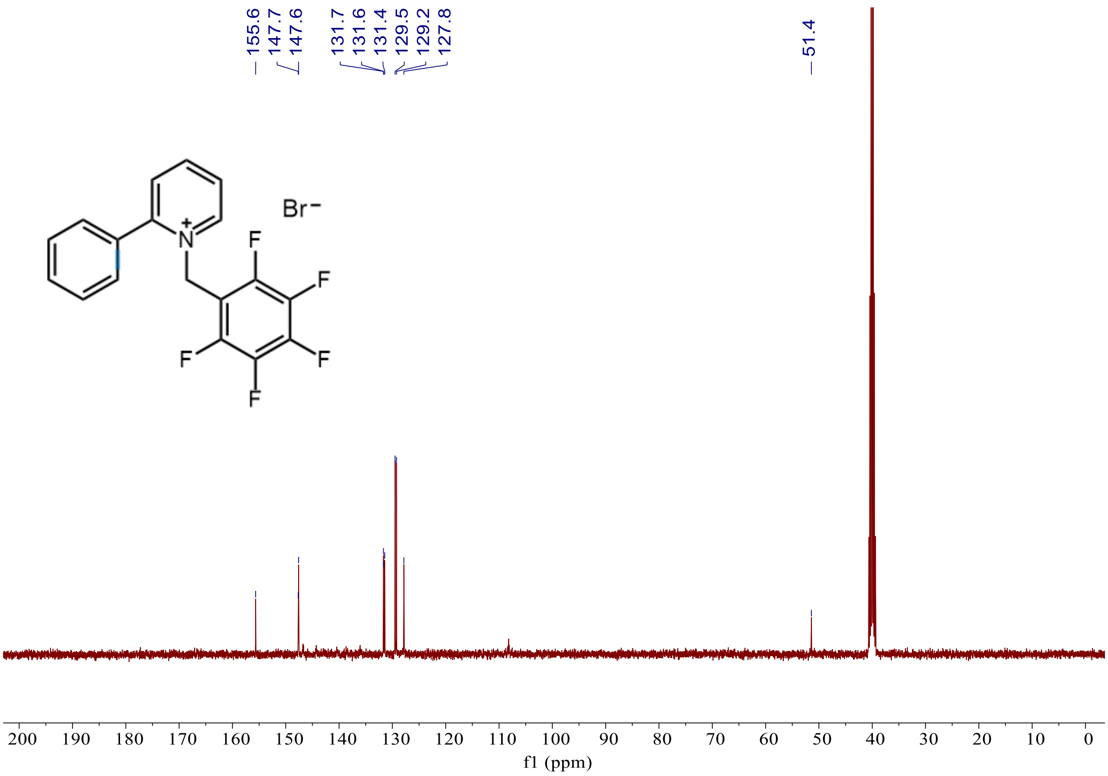
^{1}H NMR (400 MHz, DMSO-d_6) of compound **38a**



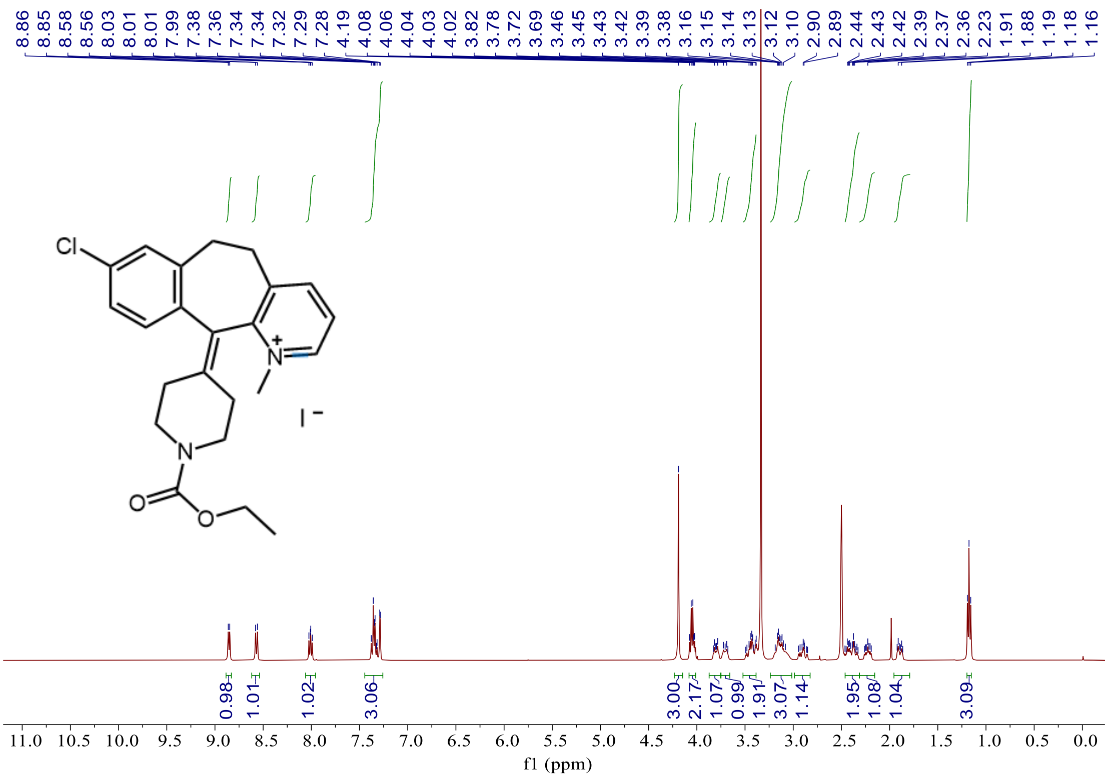
^{19}F NMR (400 MHz, DMSO-d_6) of compound **38a**



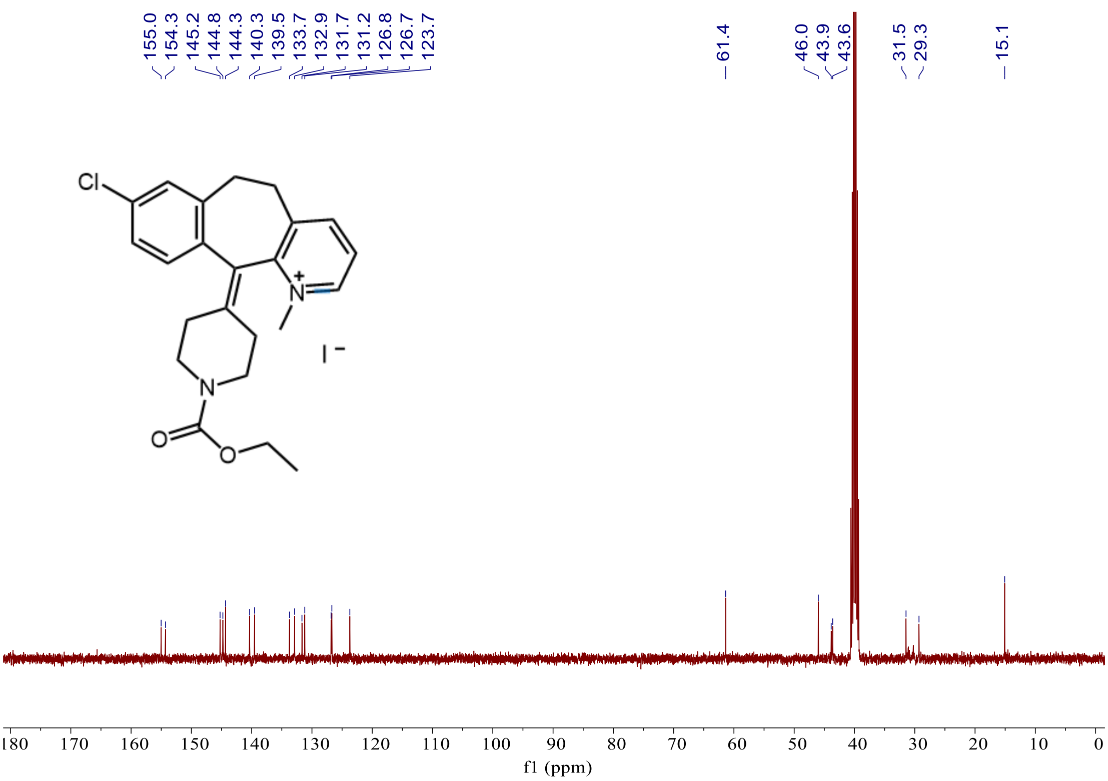
^{13}C NMR (101 MHz, DMSO-d_6) of compound **38a**



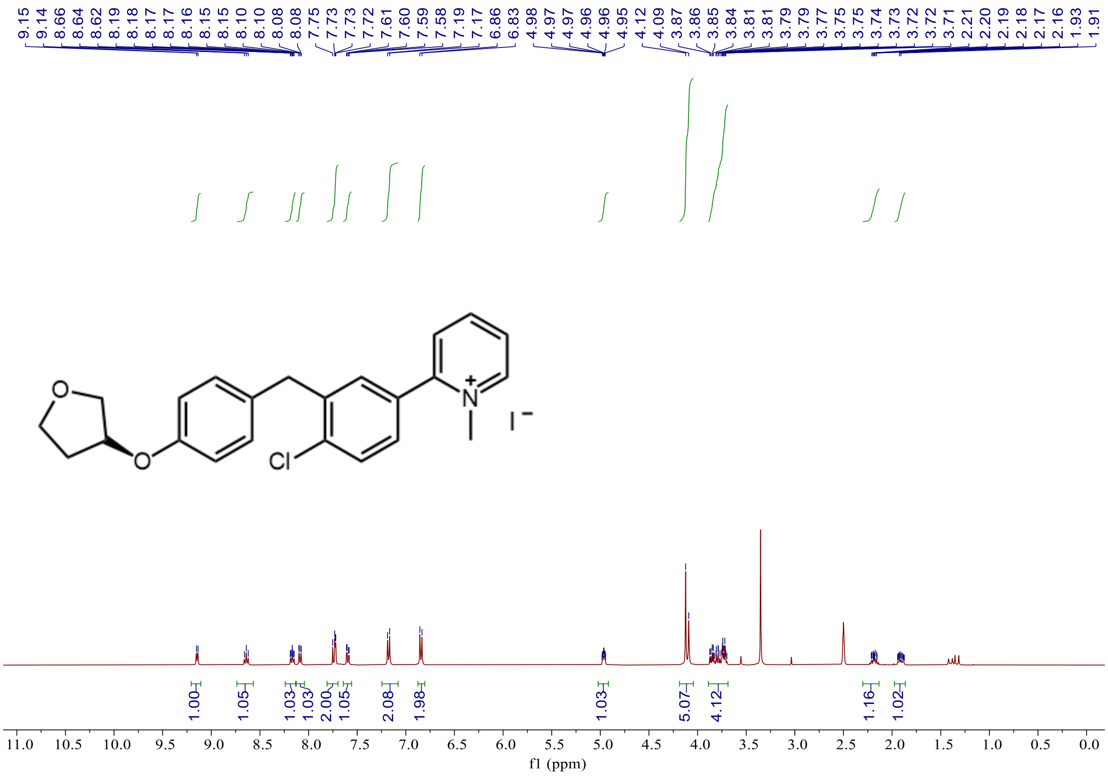
[1]H NMR (400 MHz, DMSO-d_6) of compound **39a**



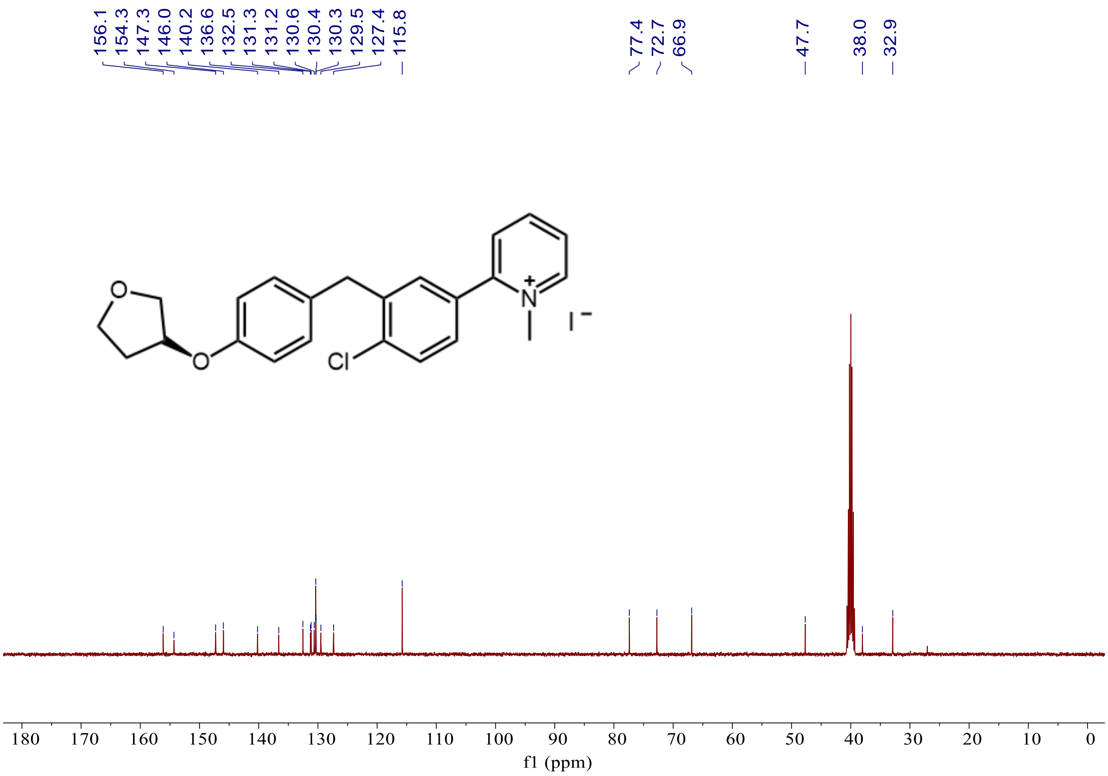
[13]C NMR (101 MHz, DMSO-d_6) of compound **39a**



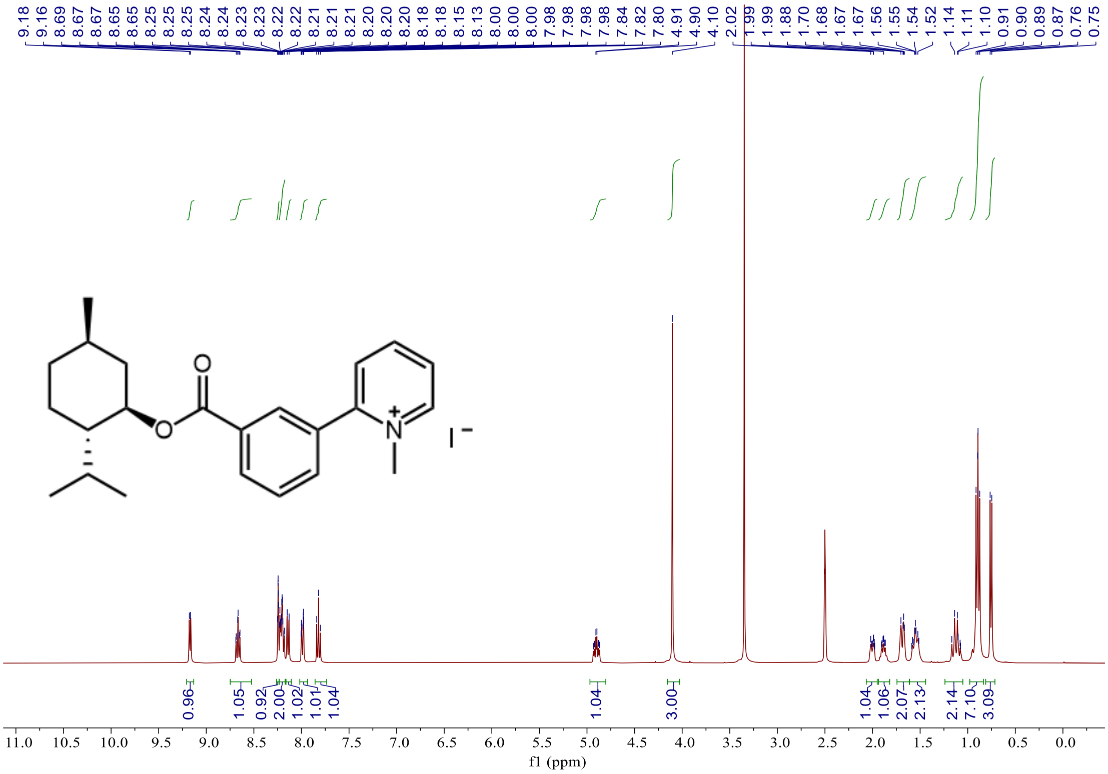
[1]H NMR (400 MHz, DMSO-d_6) of compound **40a**



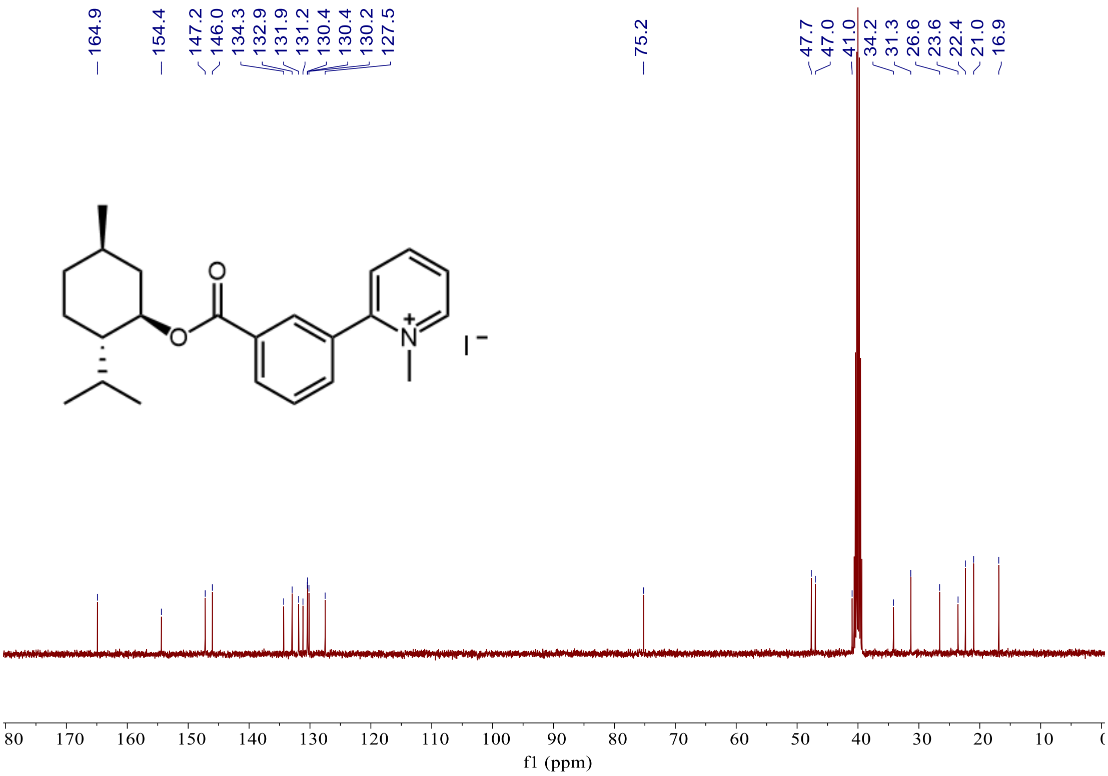
[13]C NMR (101 MHz, DMSO-d_6) of compound **40a**



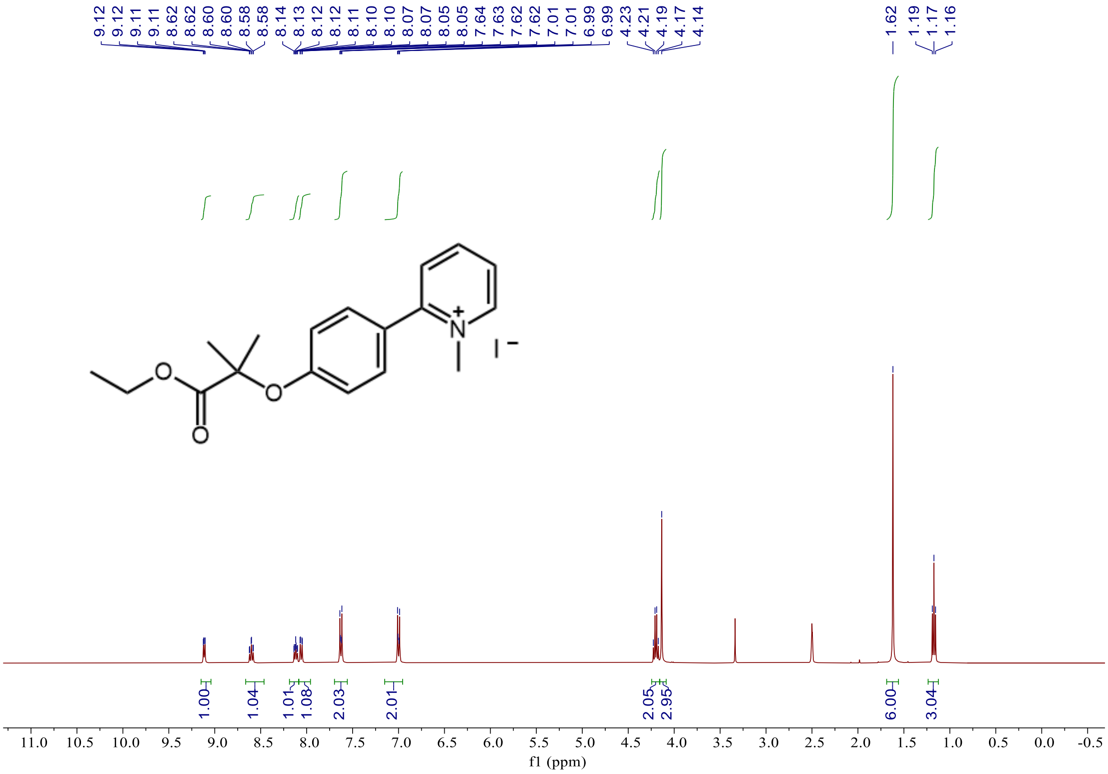
^{1}H NMR (400 MHz, DMSO-d_6) of compound **41a**



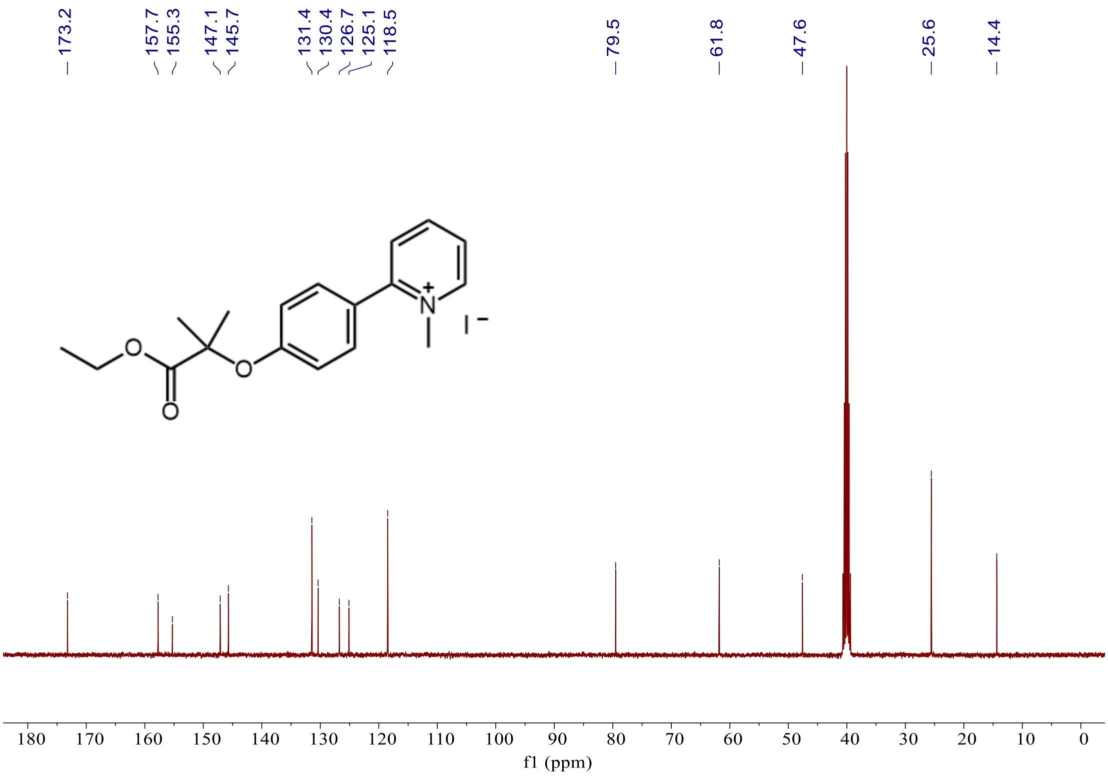
^{13}C NMR (101 MHz, DMSO-d_6) of compound **41a**



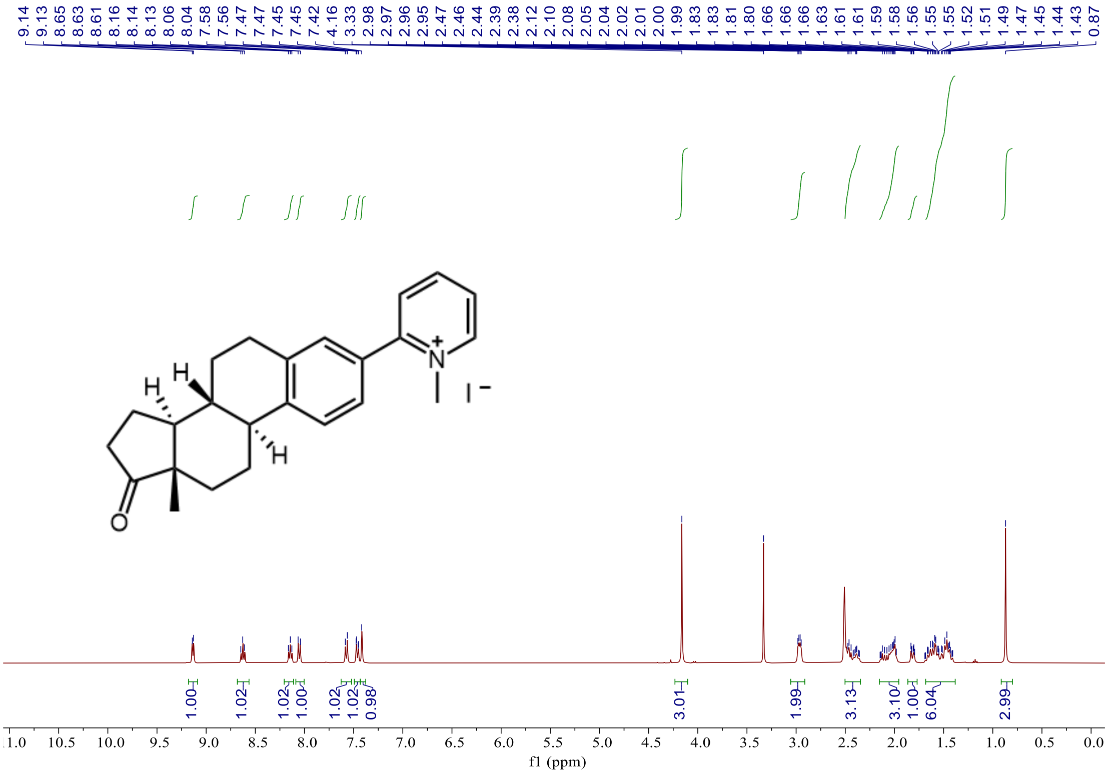
^{1}H NMR (400 MHz, DMSO-d_6) of compound **42a**



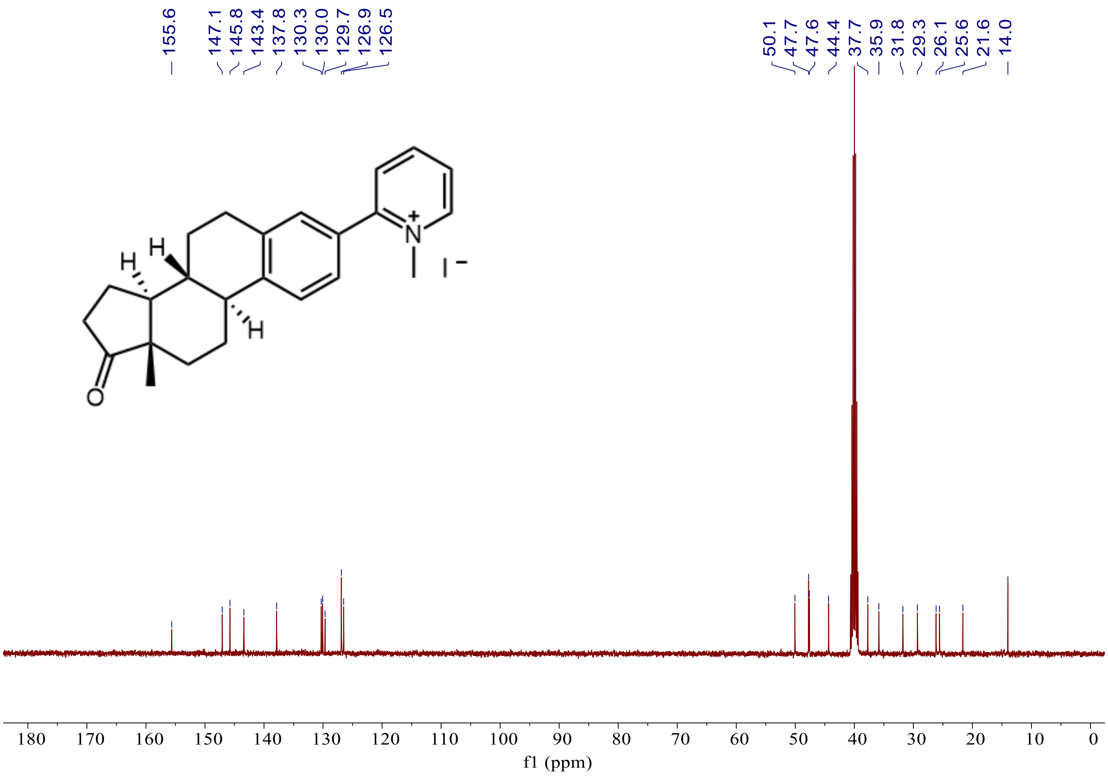
^{13}C NMR (101 MHz, DMSO-d_6) of compound **42a**



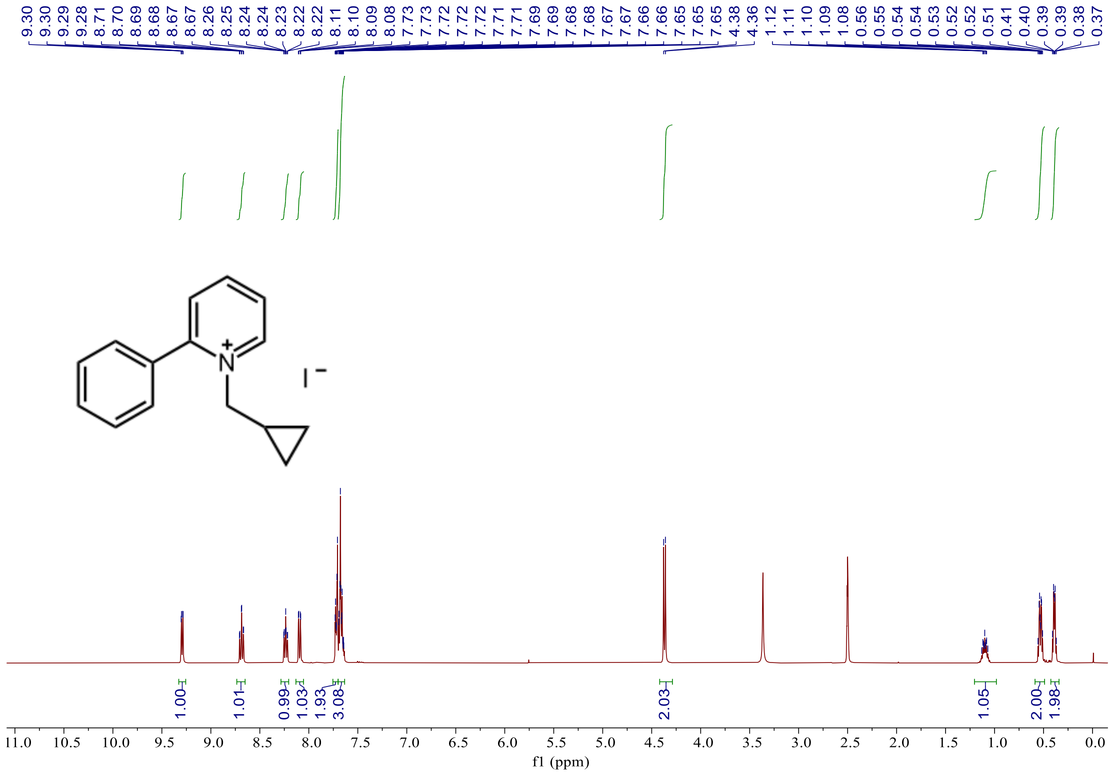
^{1}H NMR (400 MHz, DMSO-d_6) of compound **43a**



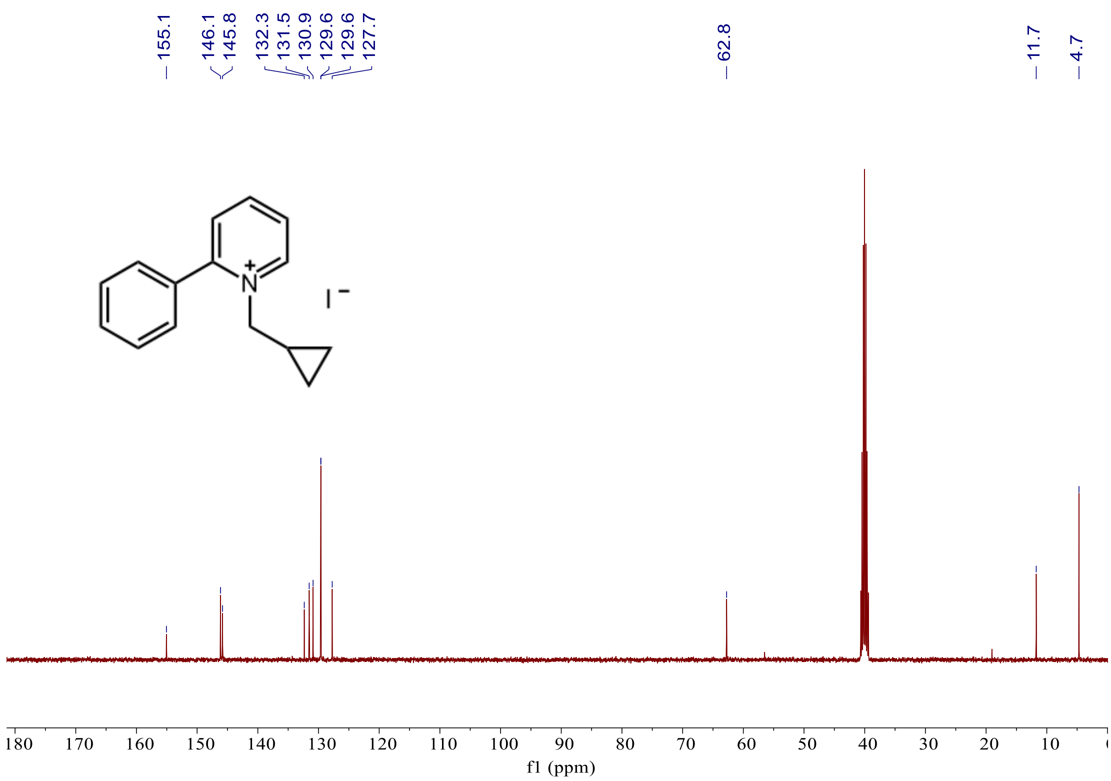
^{13}C NMR (101 MHz, DMSO-d_6) of compound **43a**



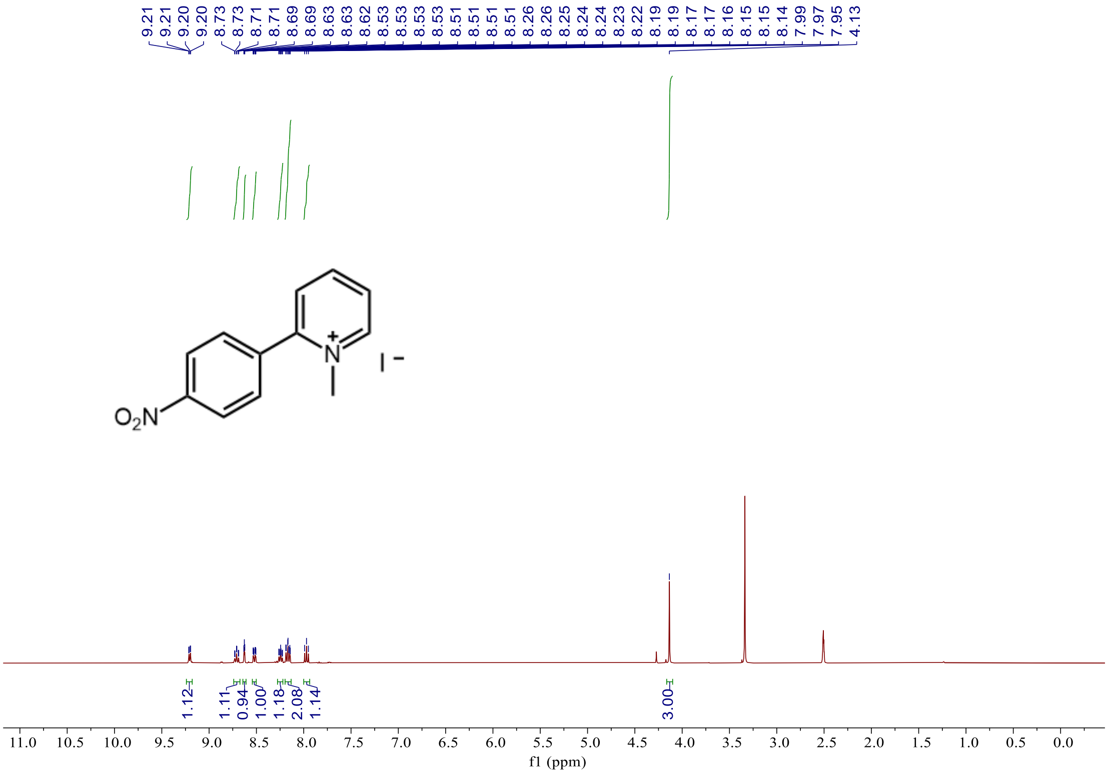
[1]H NMR (400 MHz, DMSO-d_6) of compound **1aa**



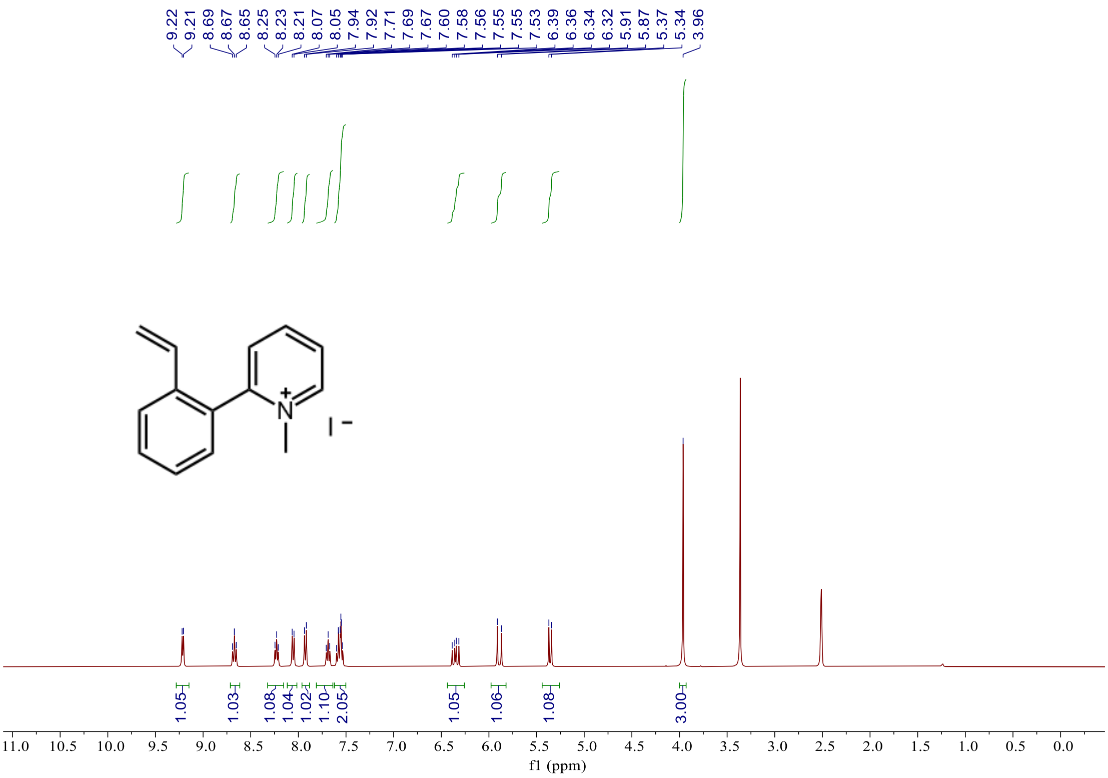
[13]C NMR (101 MHz, DMSO-d_6) of compound **1aa**



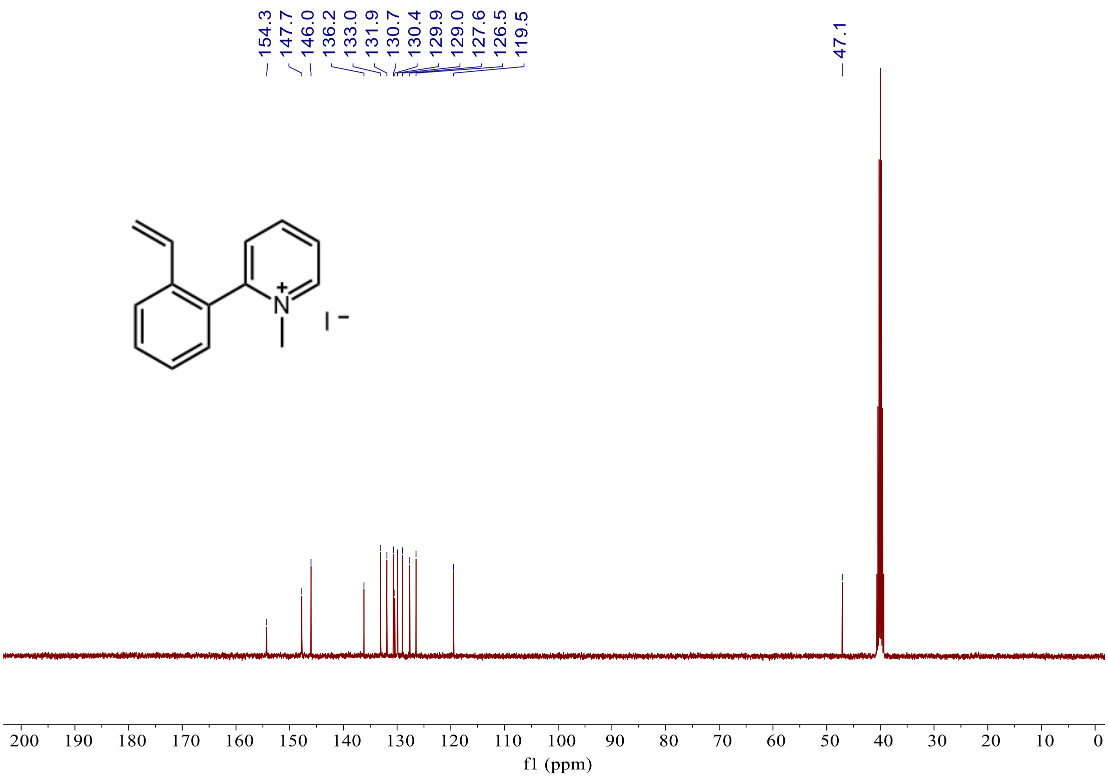
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ab**



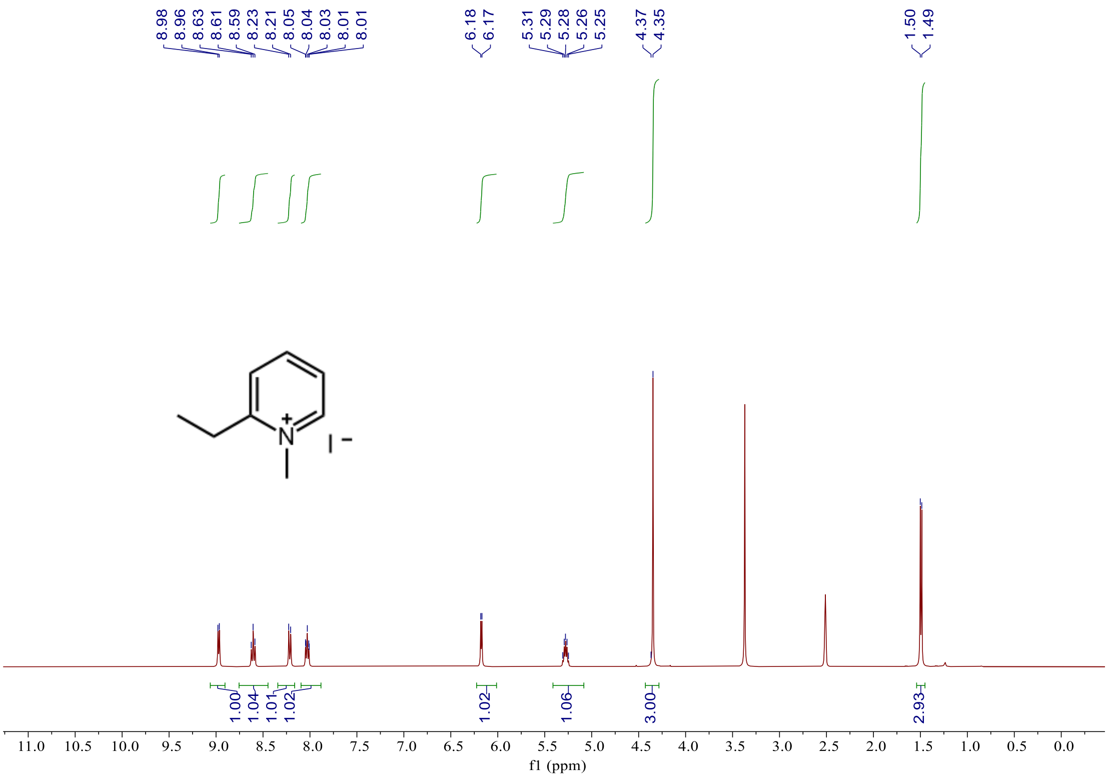
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ac**



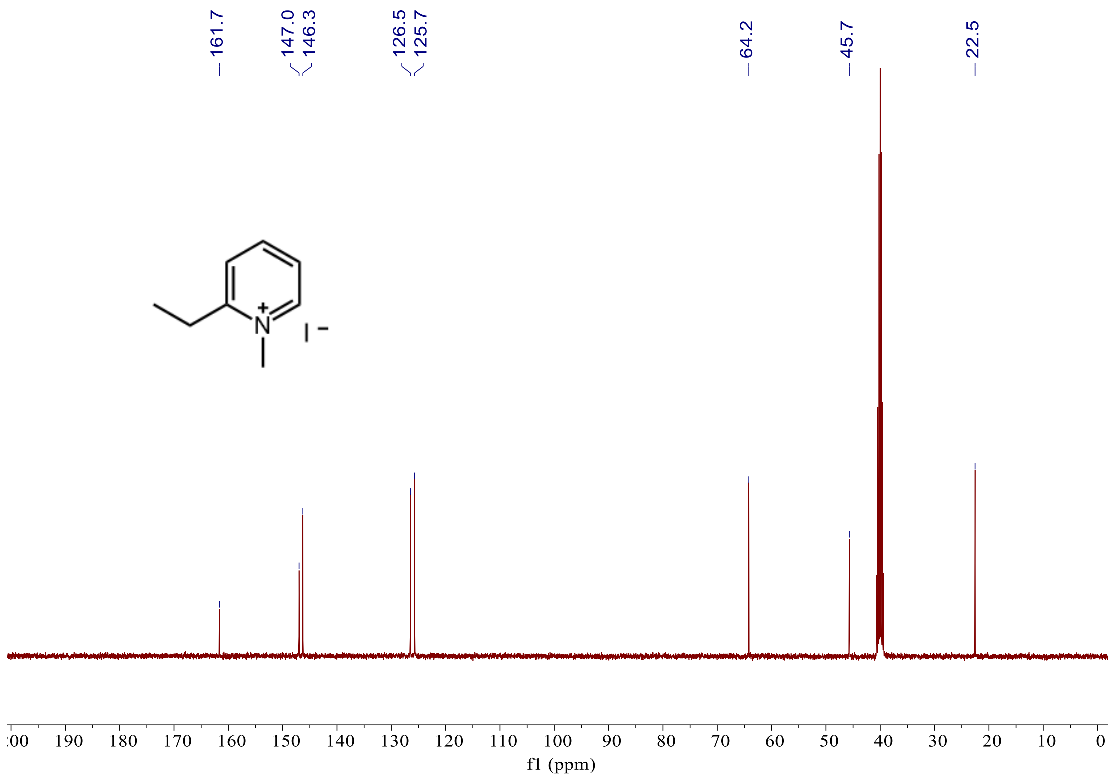
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1ac**



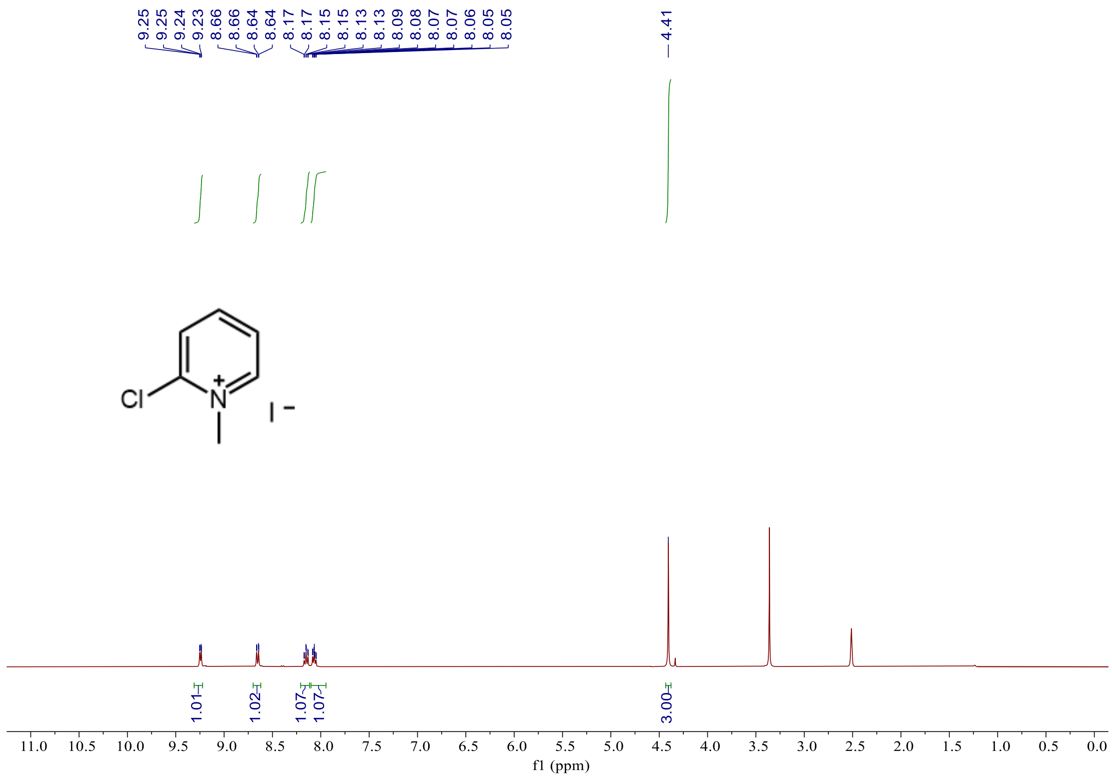
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ad**



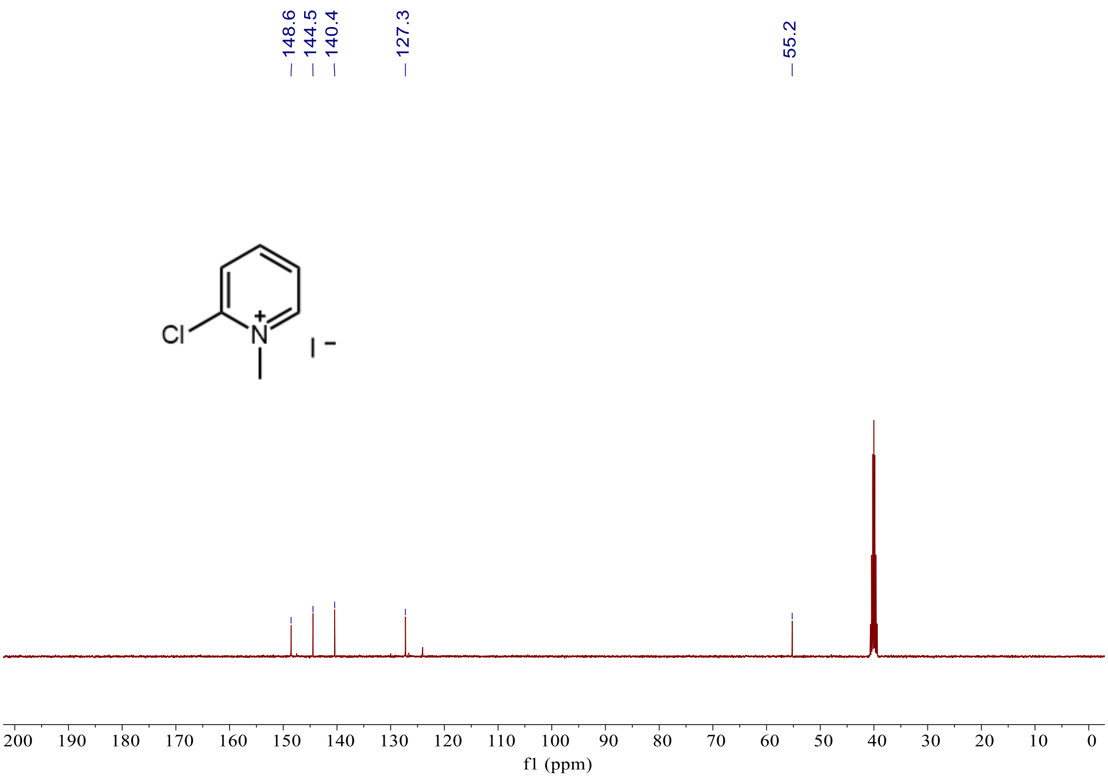
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1ad**



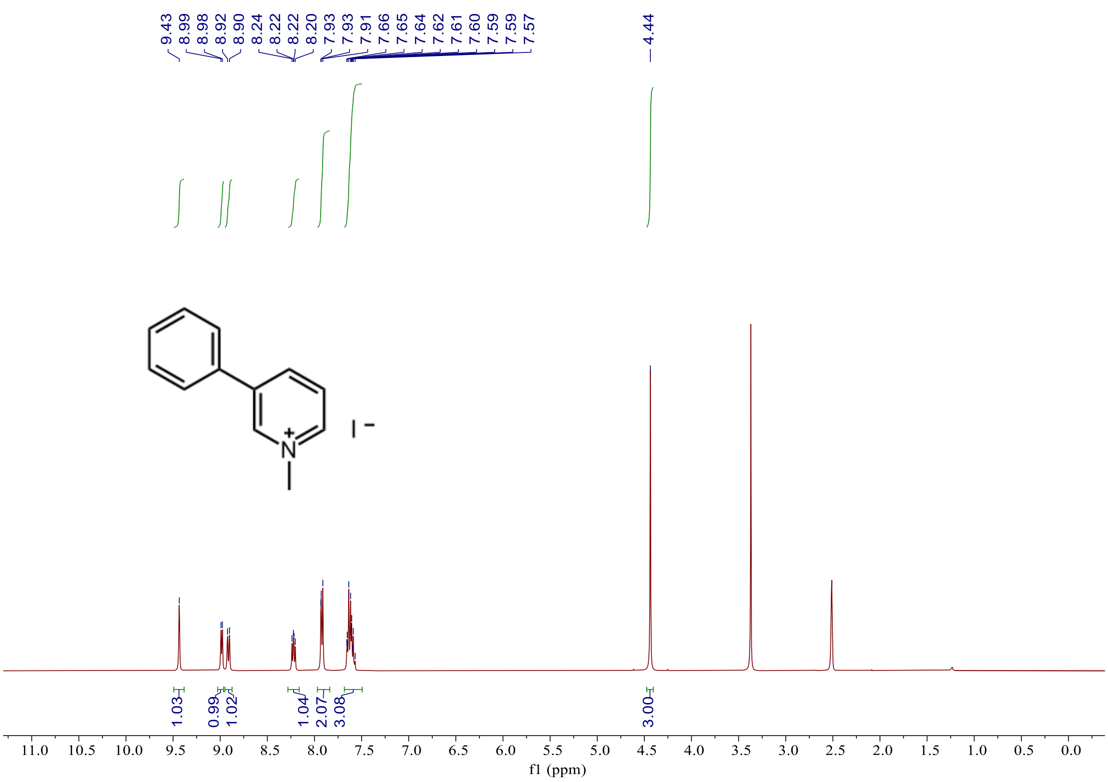
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ae**



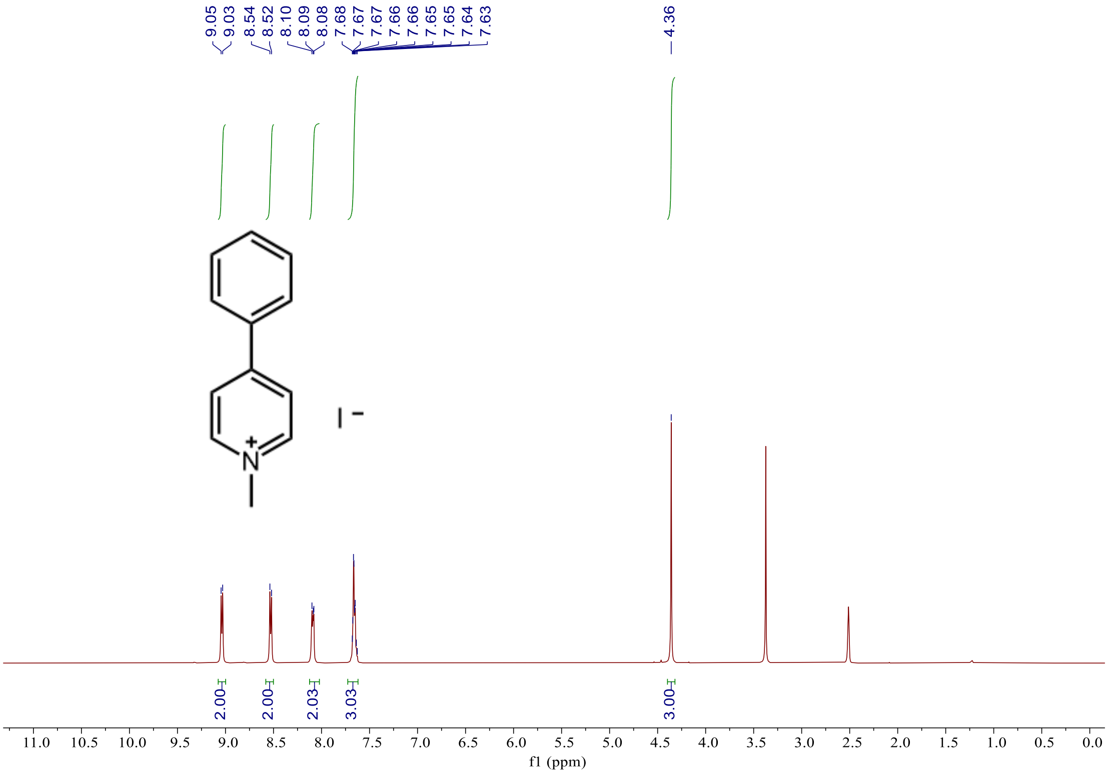
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1ae**



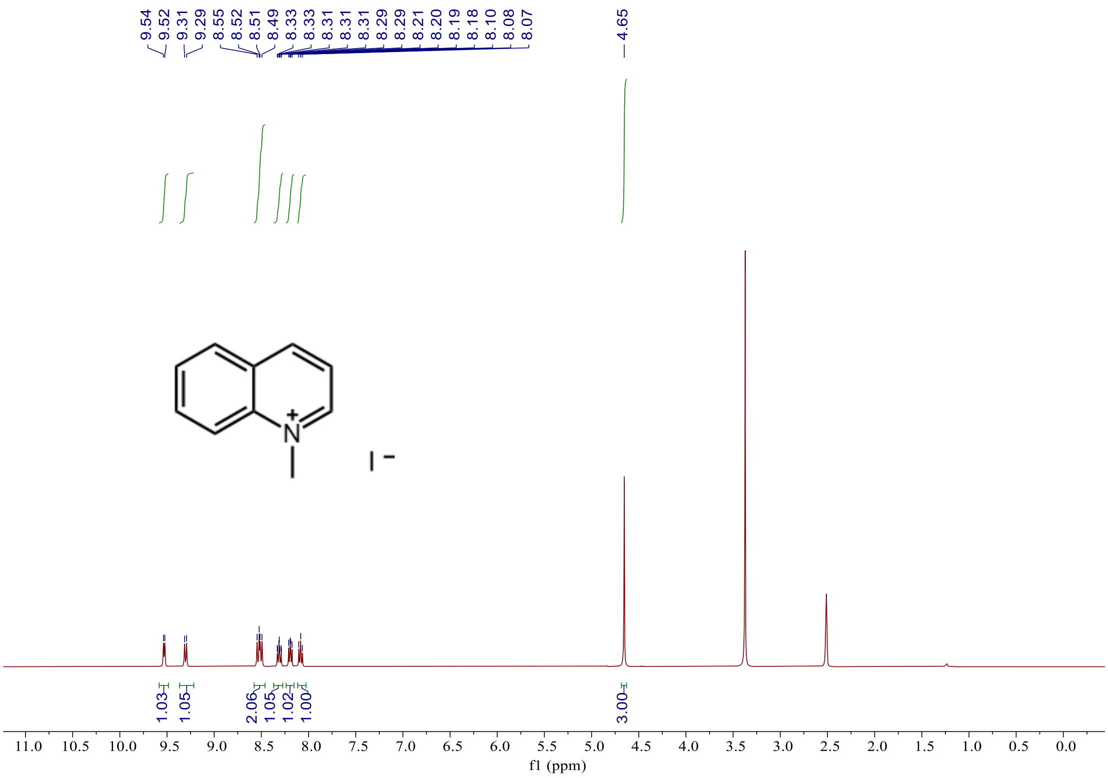
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1af**



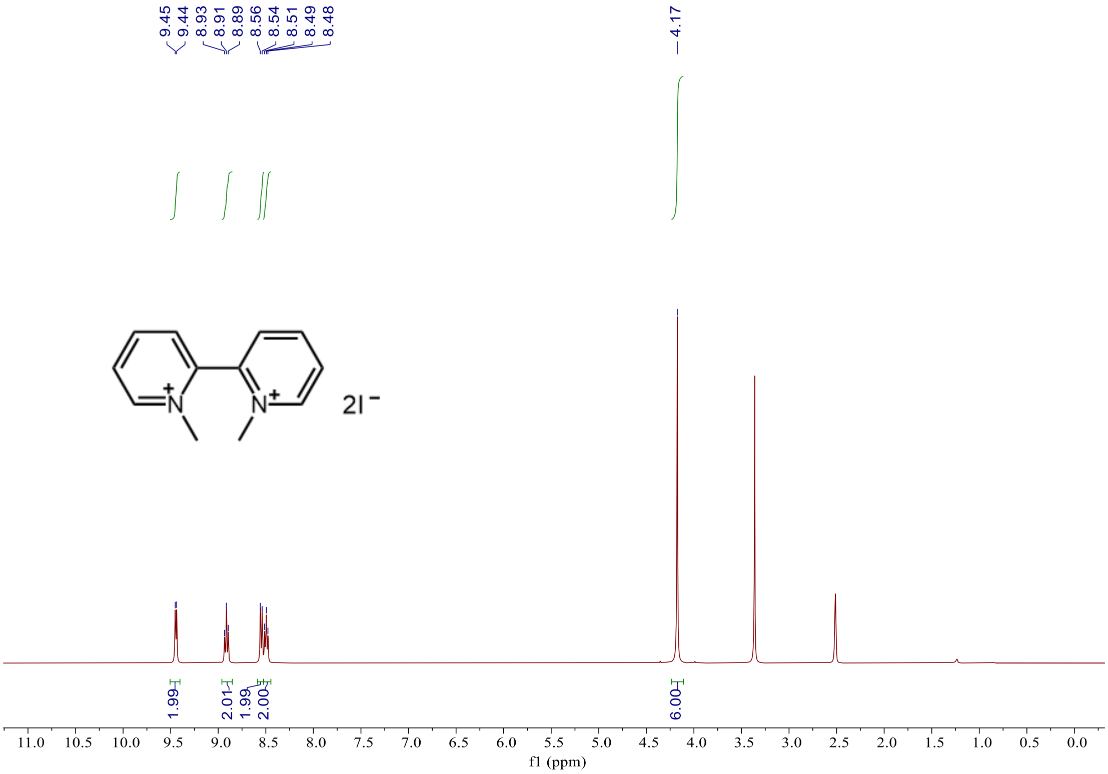
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ag**



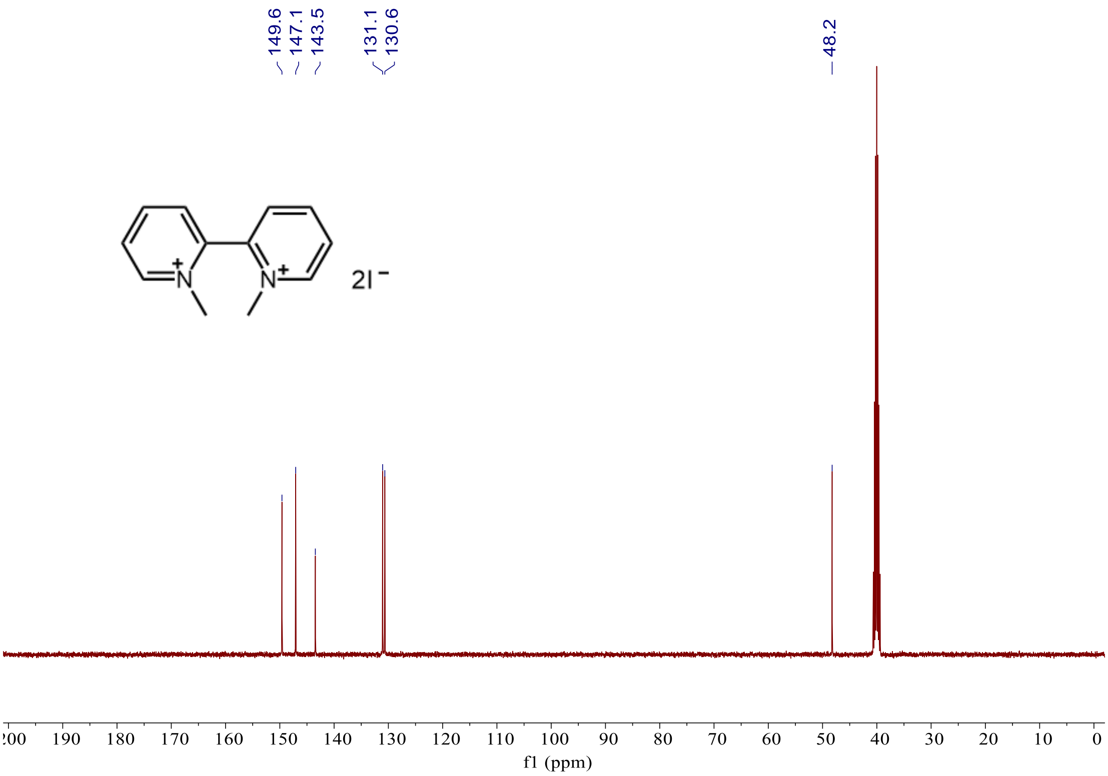
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ah**



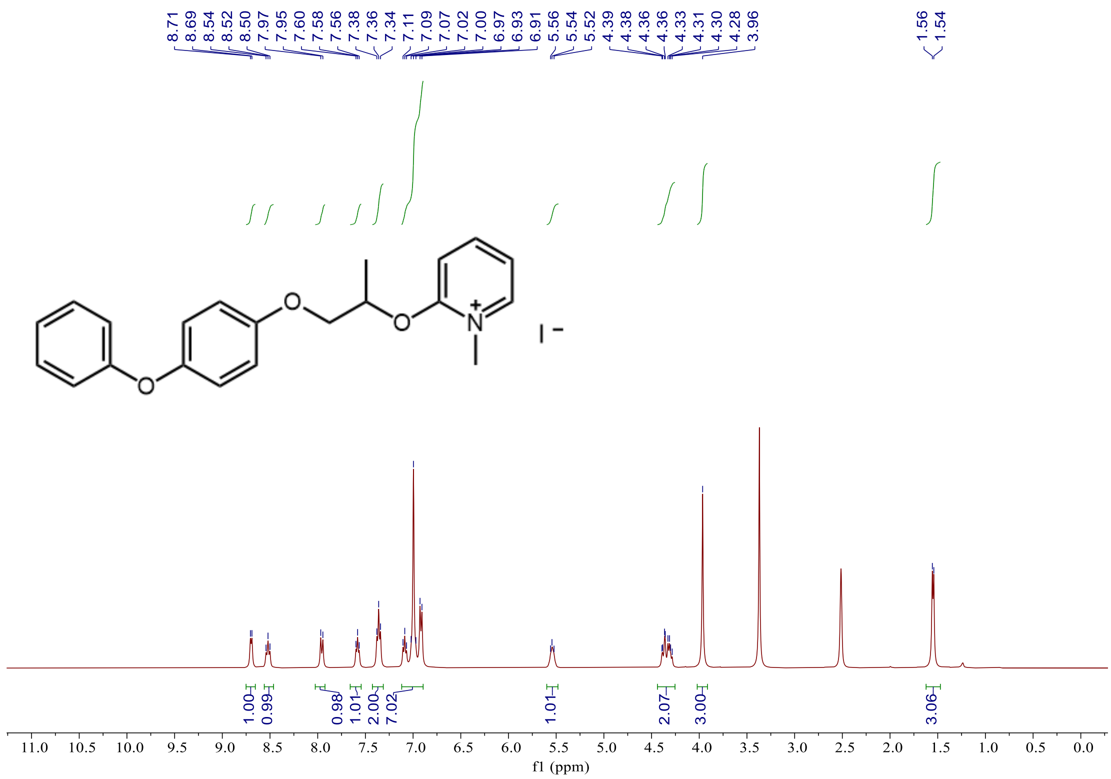
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ai**



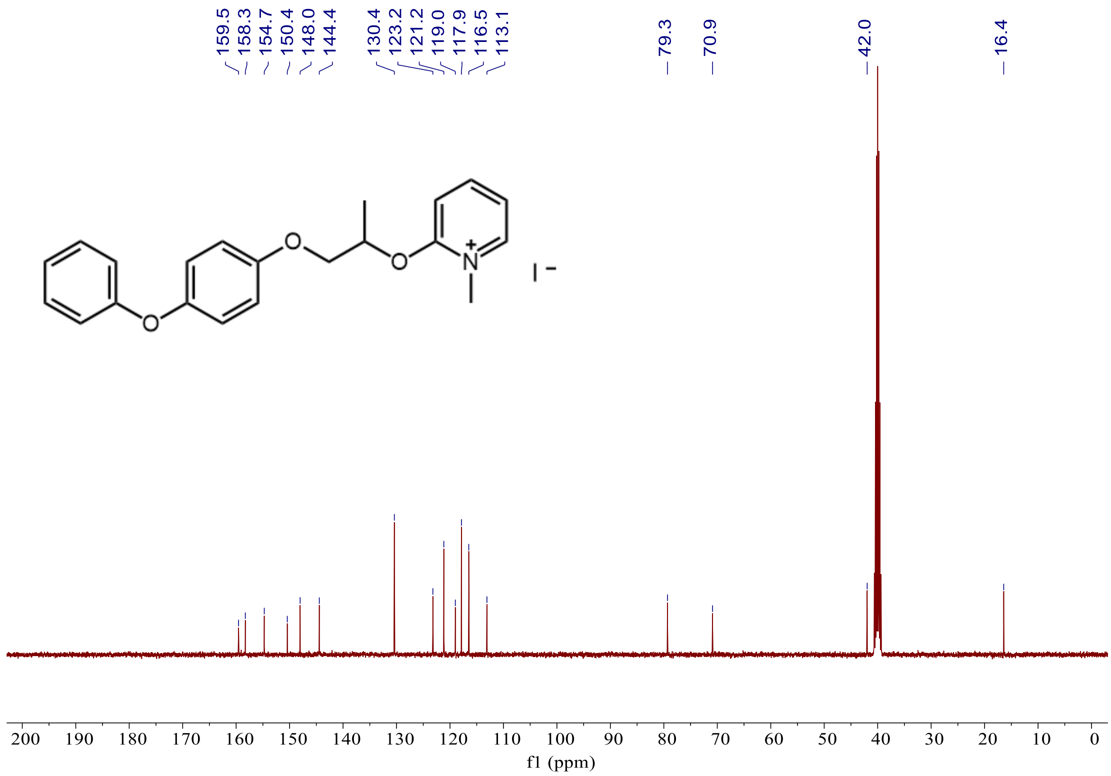
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1ai**



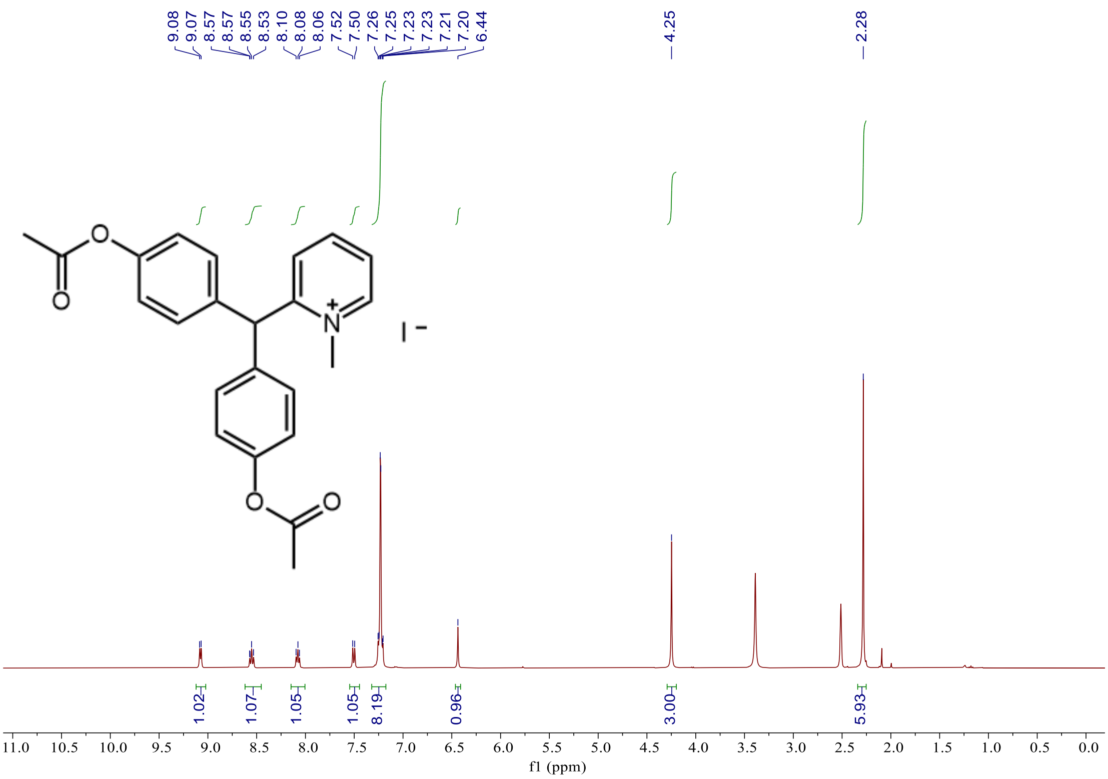
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1aj**



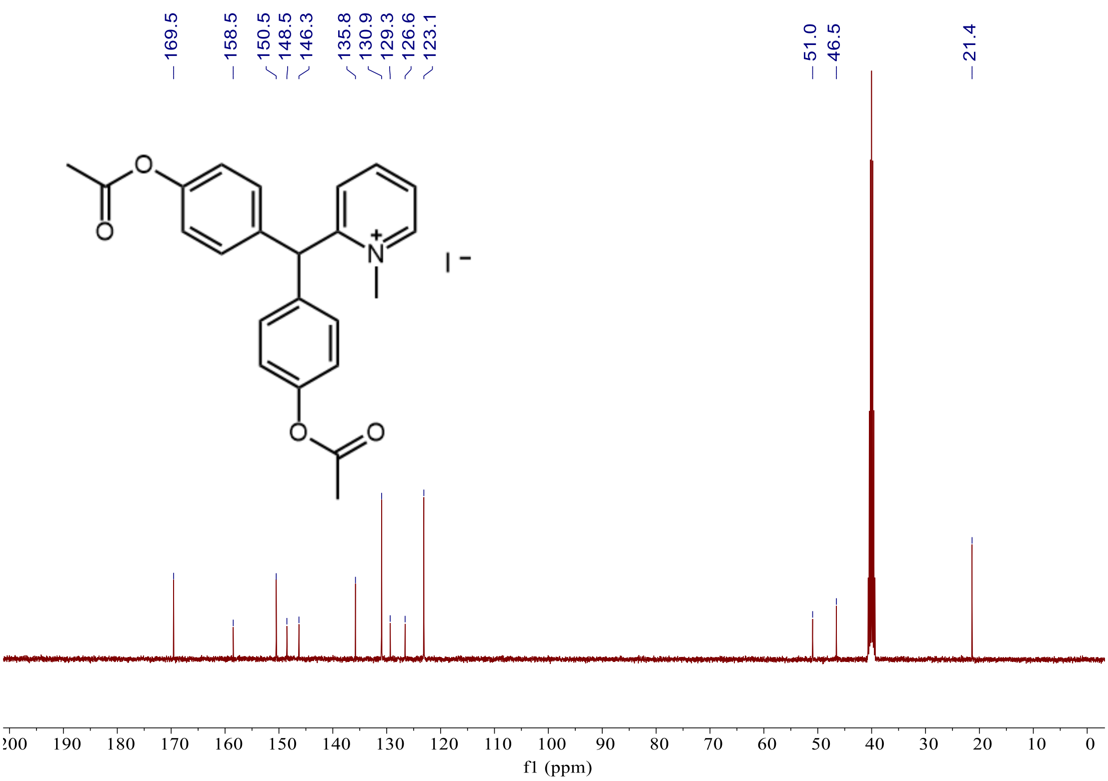
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1aj**



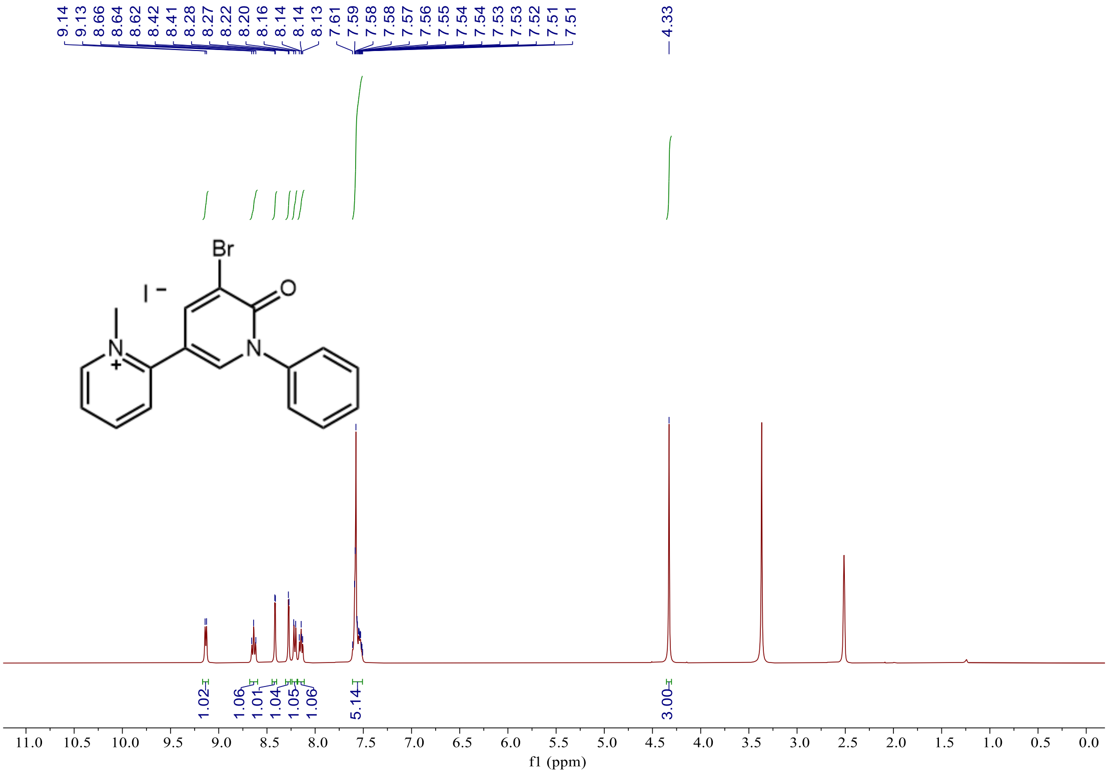
[1]H NMR (400 MHz, DMSO-d_6) of compound **1ak**



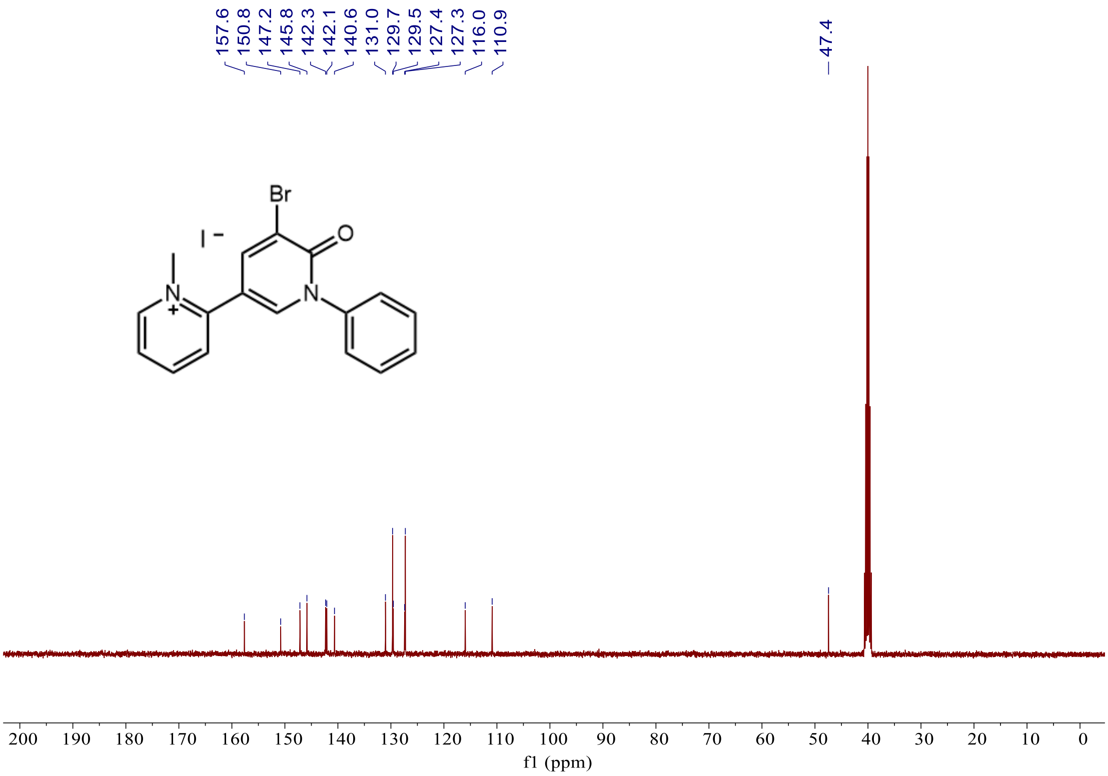
[13]C NMR (101 MHz, DMSO-d_6) of compound **1aj**



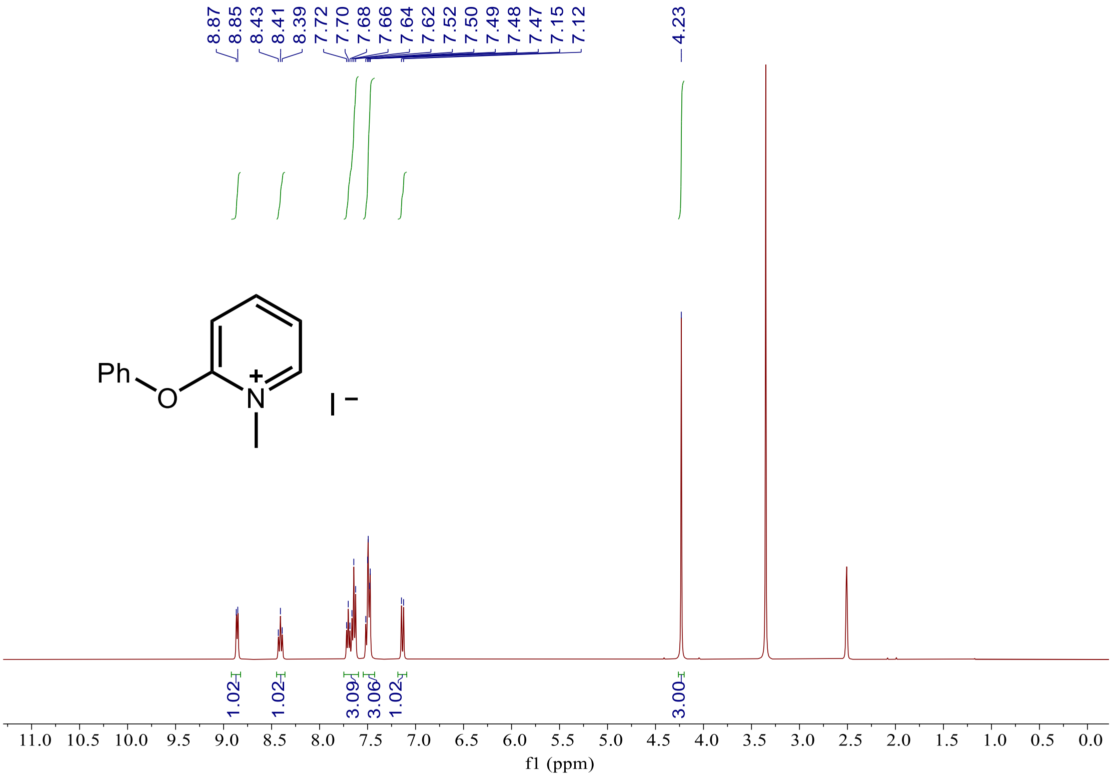
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1al**



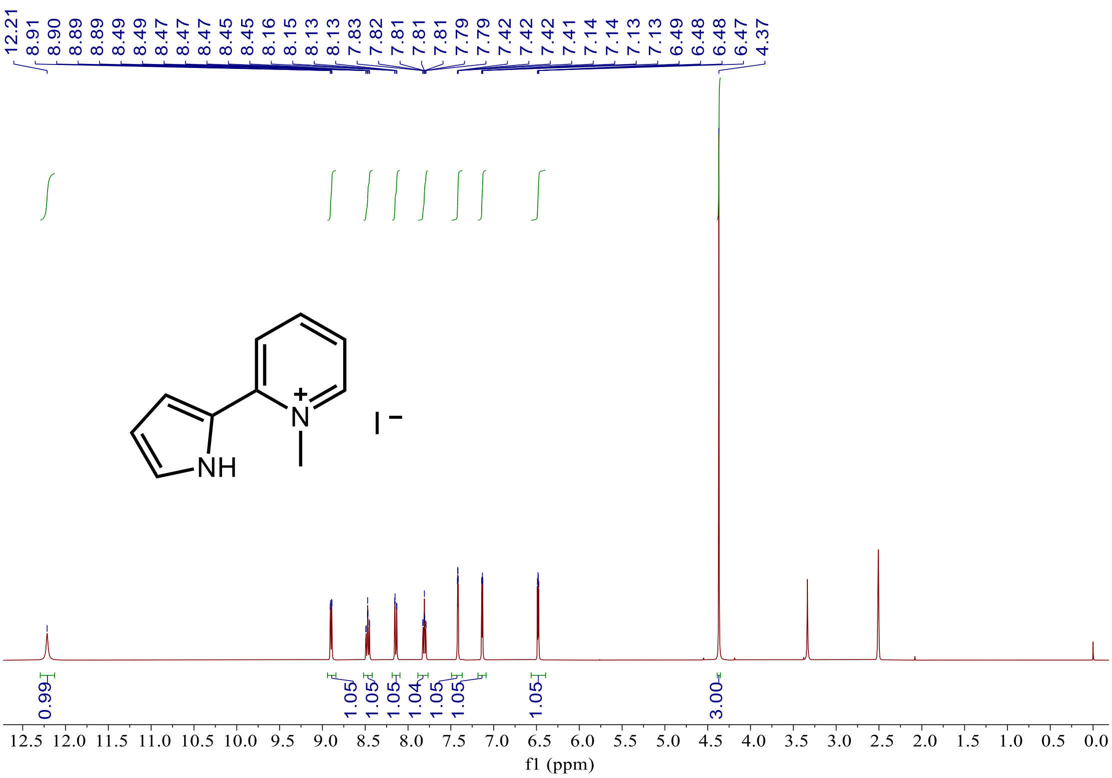
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1aj**



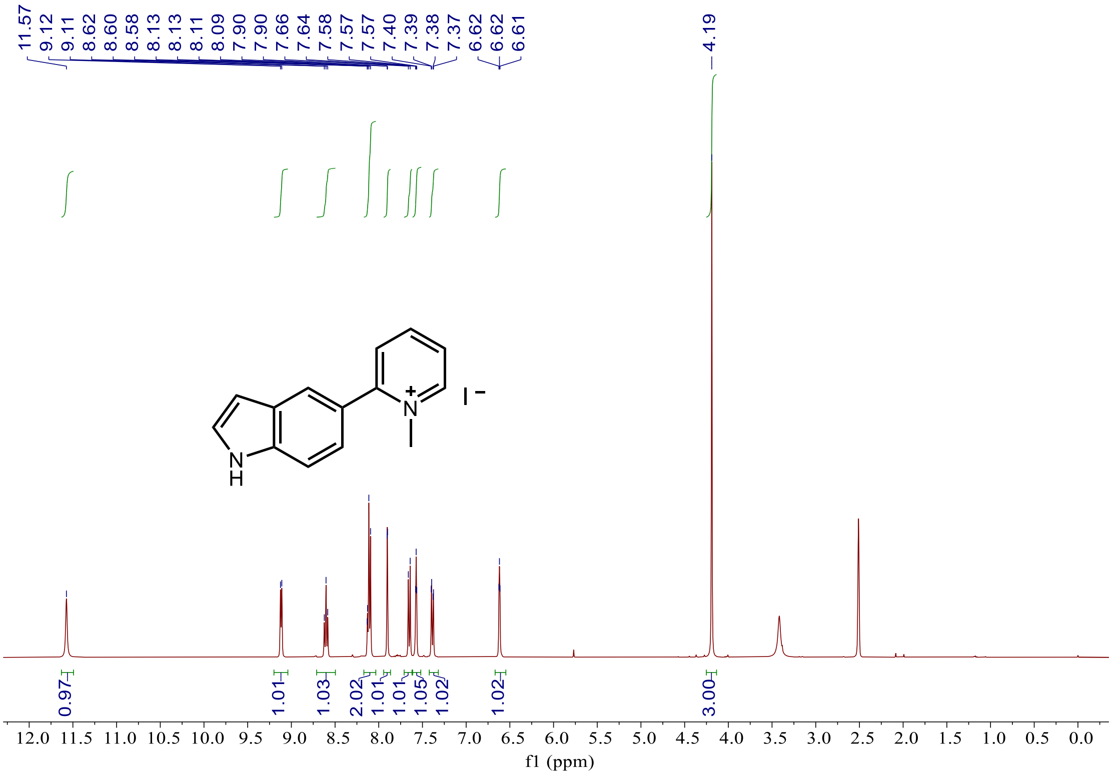
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1am**



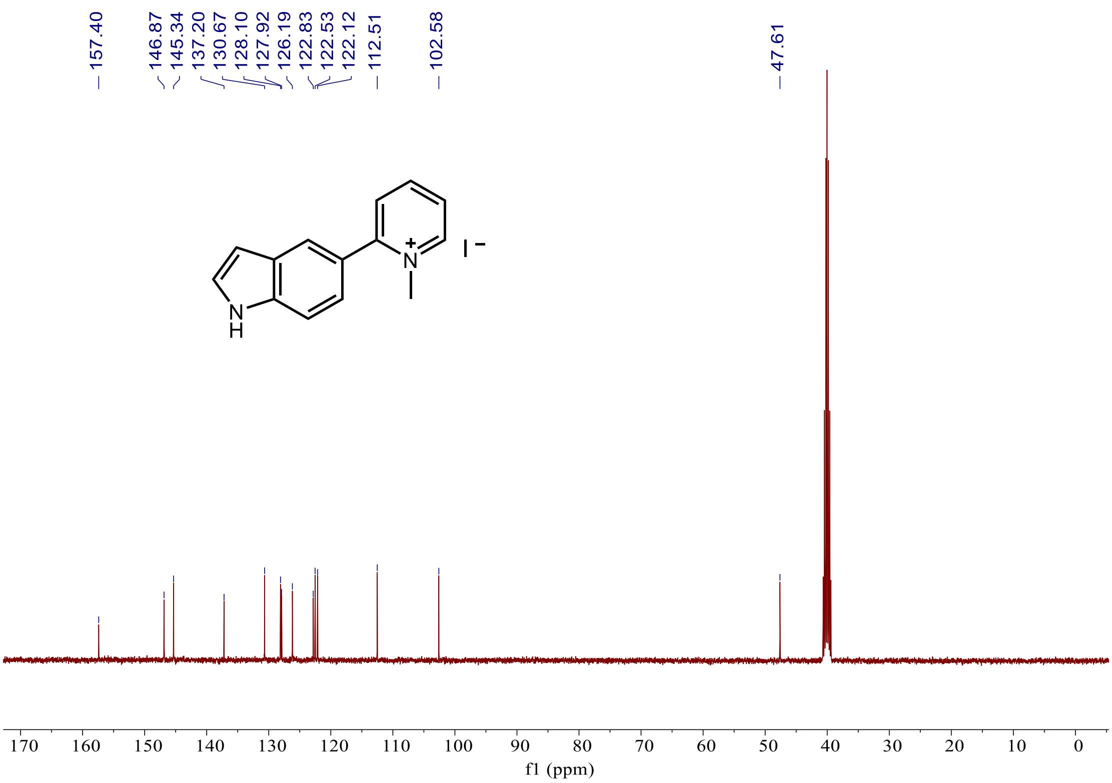
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ao**



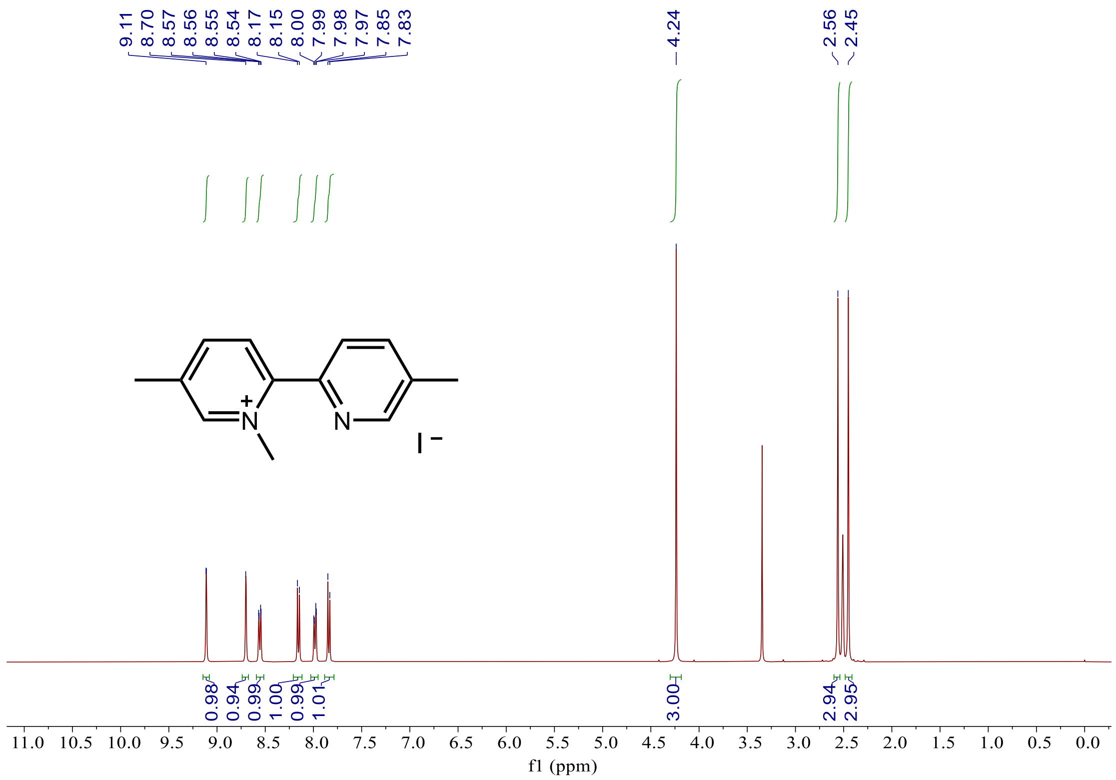
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ap**



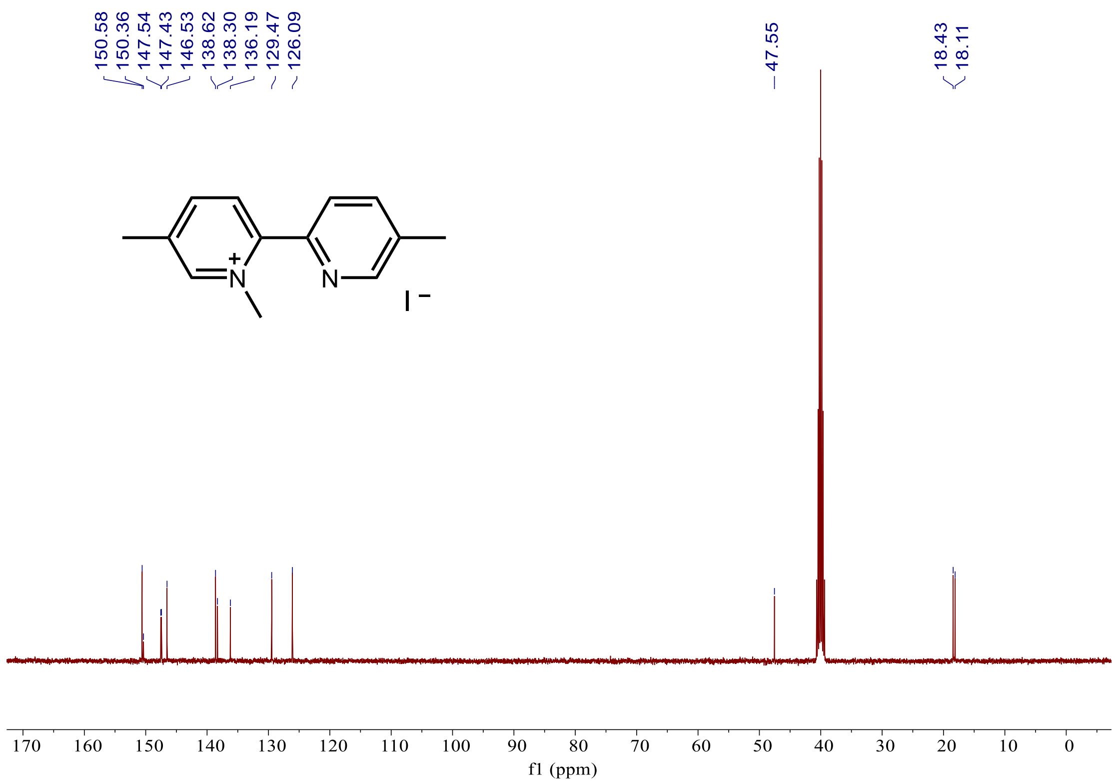
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1ap**



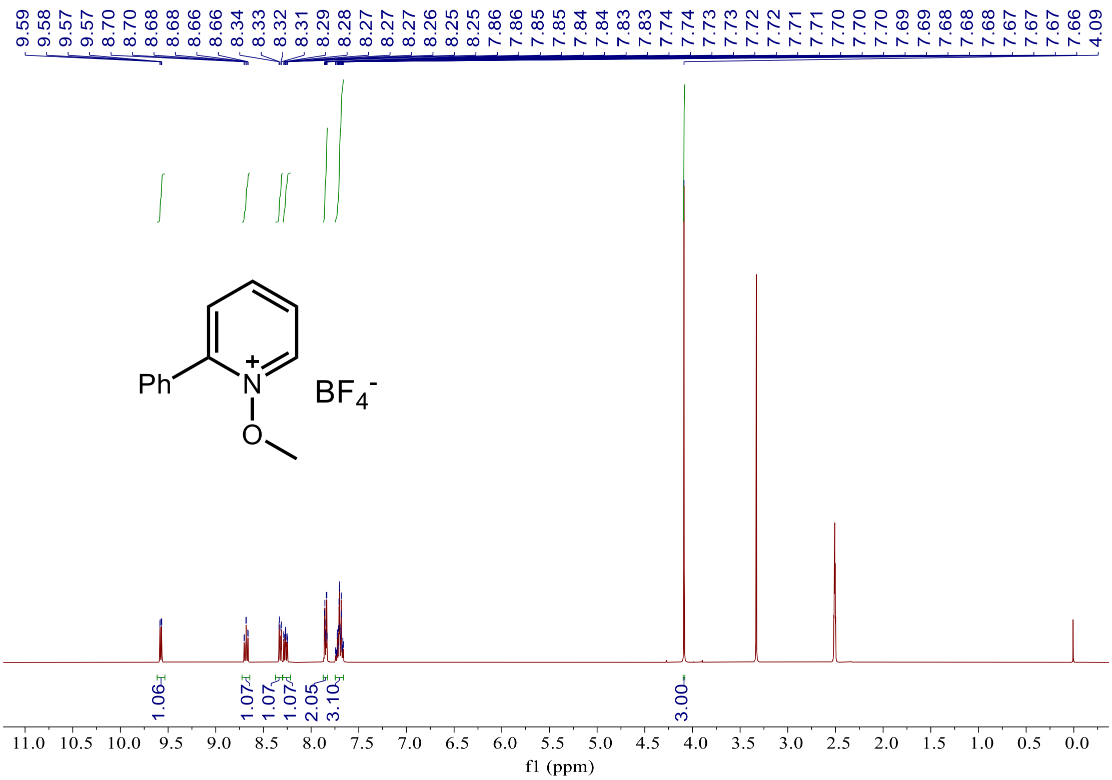
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1aq**



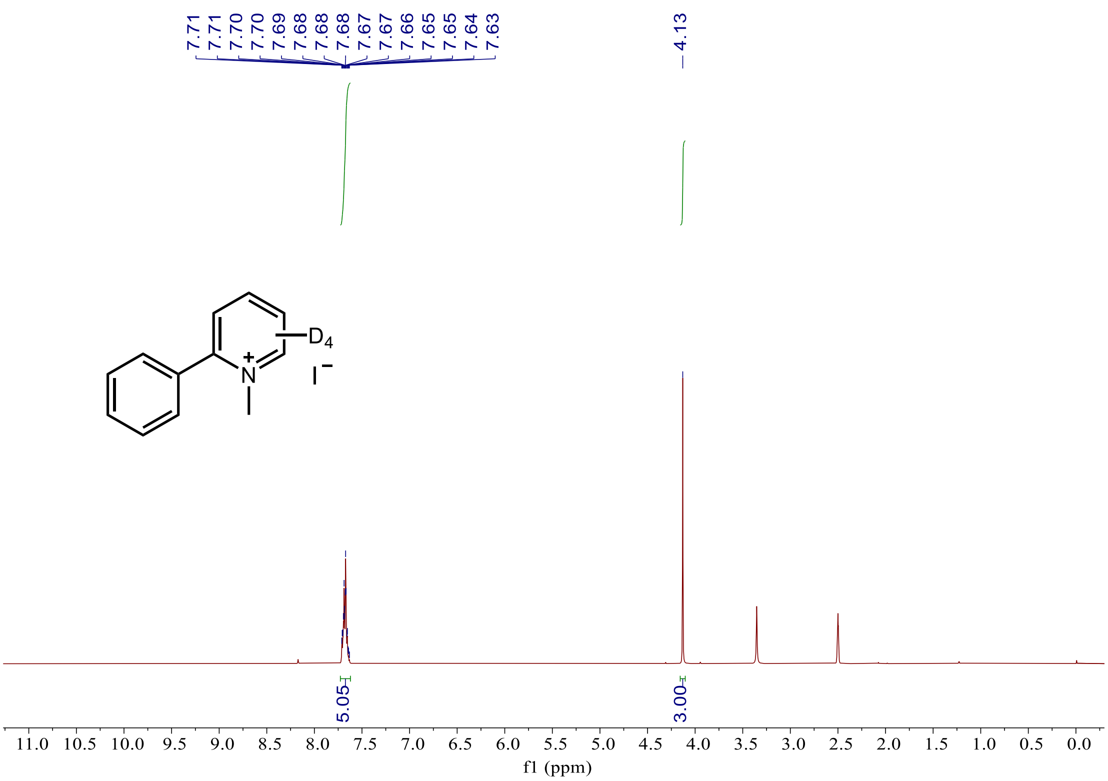
^{13}C NMR (101 MHz, DMSO-d_6) of compound **1aq**



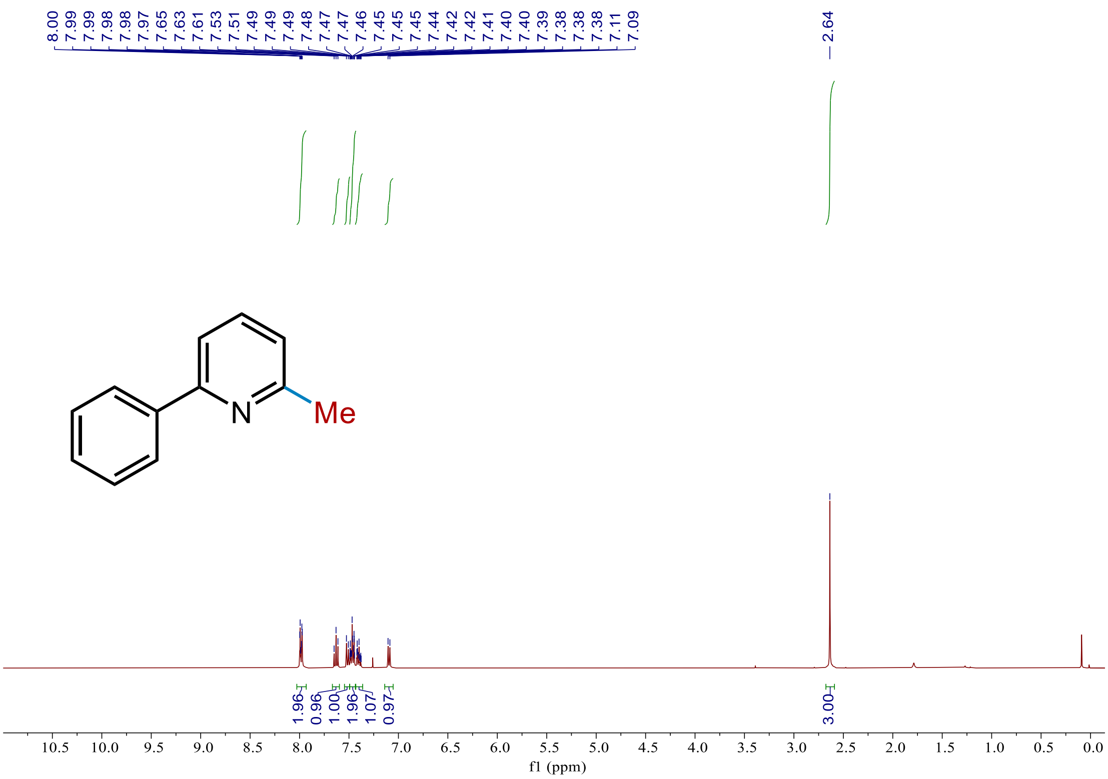
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1ar**



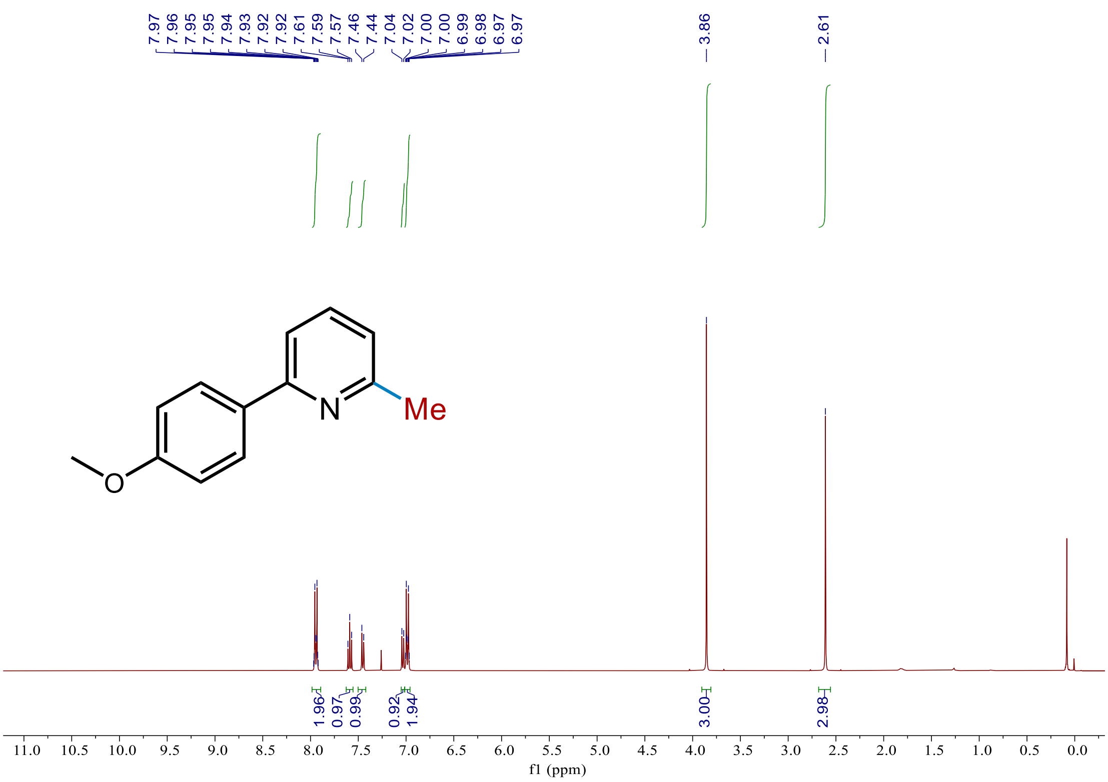
^{1}H NMR (400 MHz, DMSO-d_6) of compound **1a-D*4***



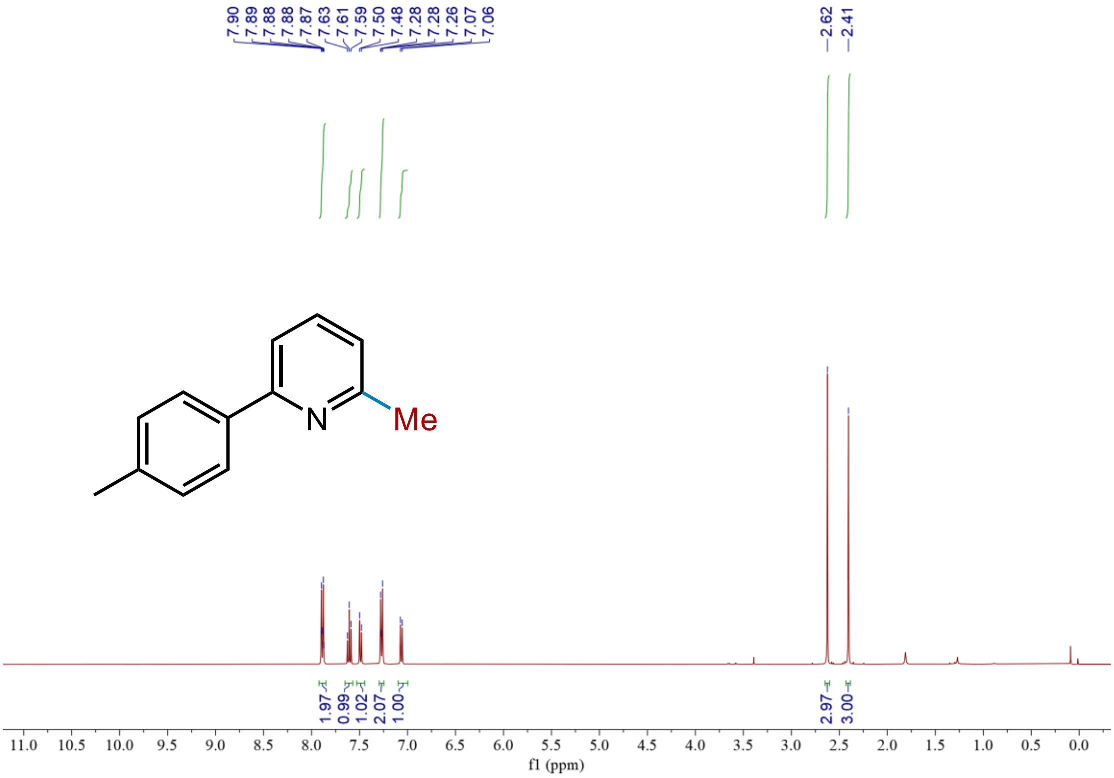
[1]H NMR (400 MHz, Chloroform-*d*) of compound **1**



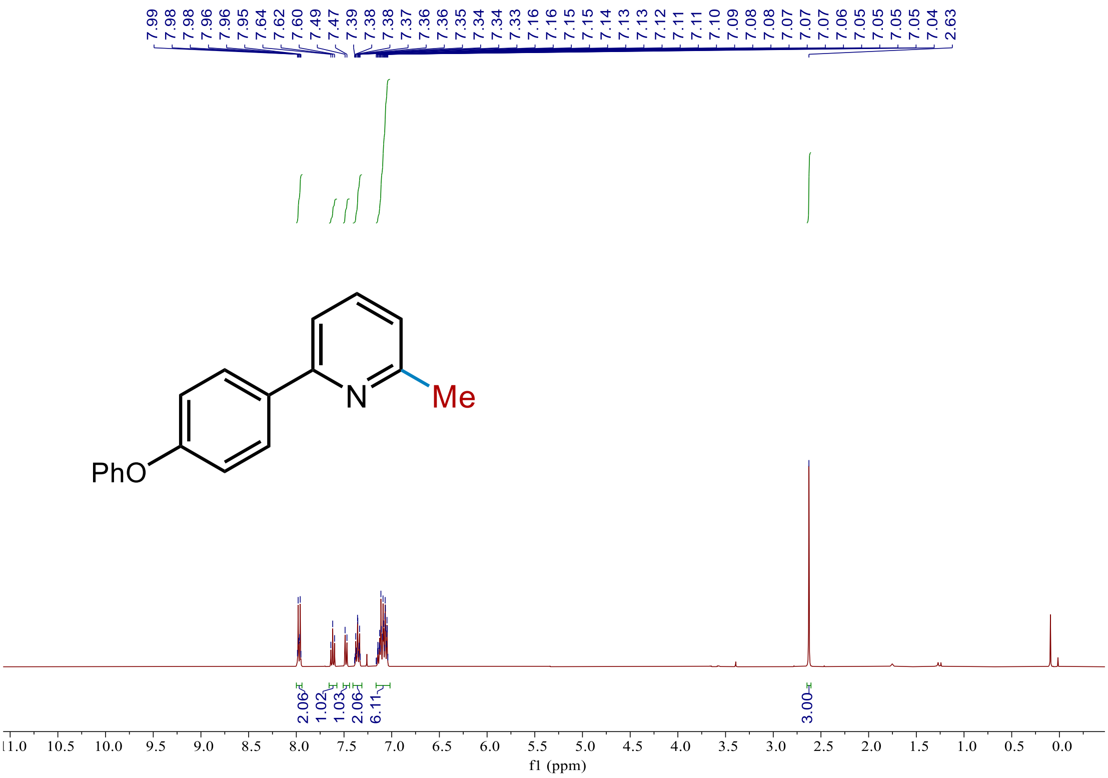
[1]H NMR (400 MHz, Chloroform-*d*) of compound **2**



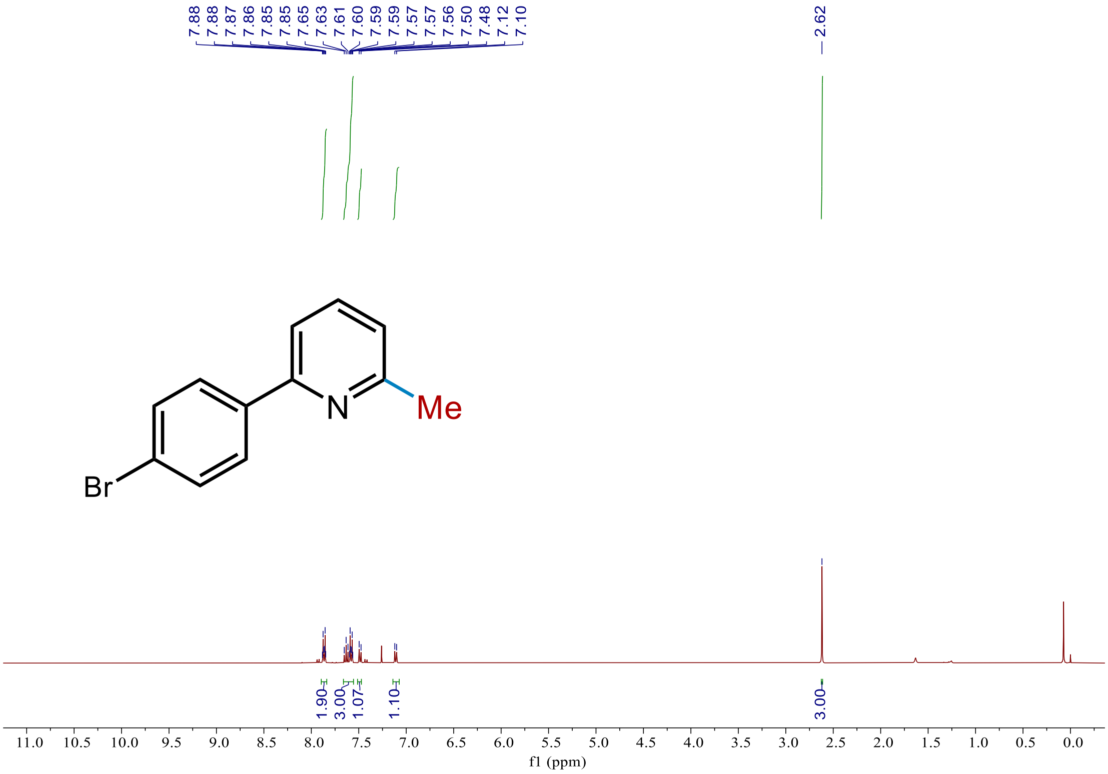
[1]H NMR (400 MHz, Chloroform-*d*) of compound **3**



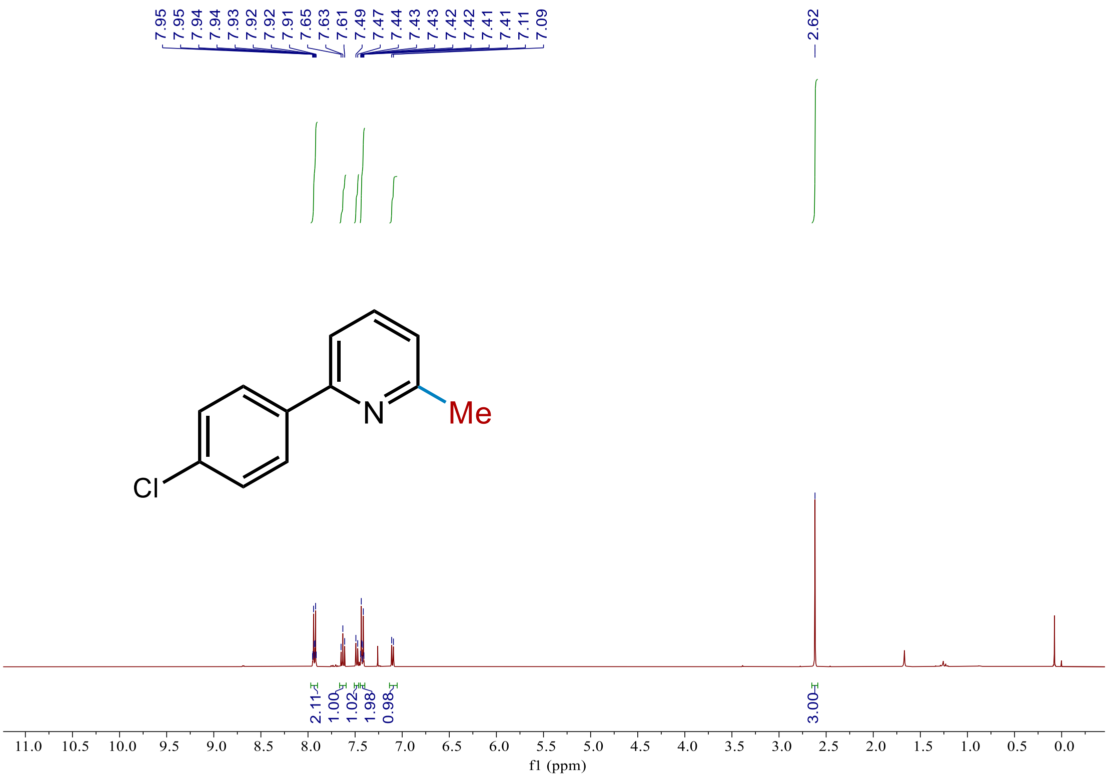
[1]H NMR (400 MHz, Chloroform-*d*) of compound **4**



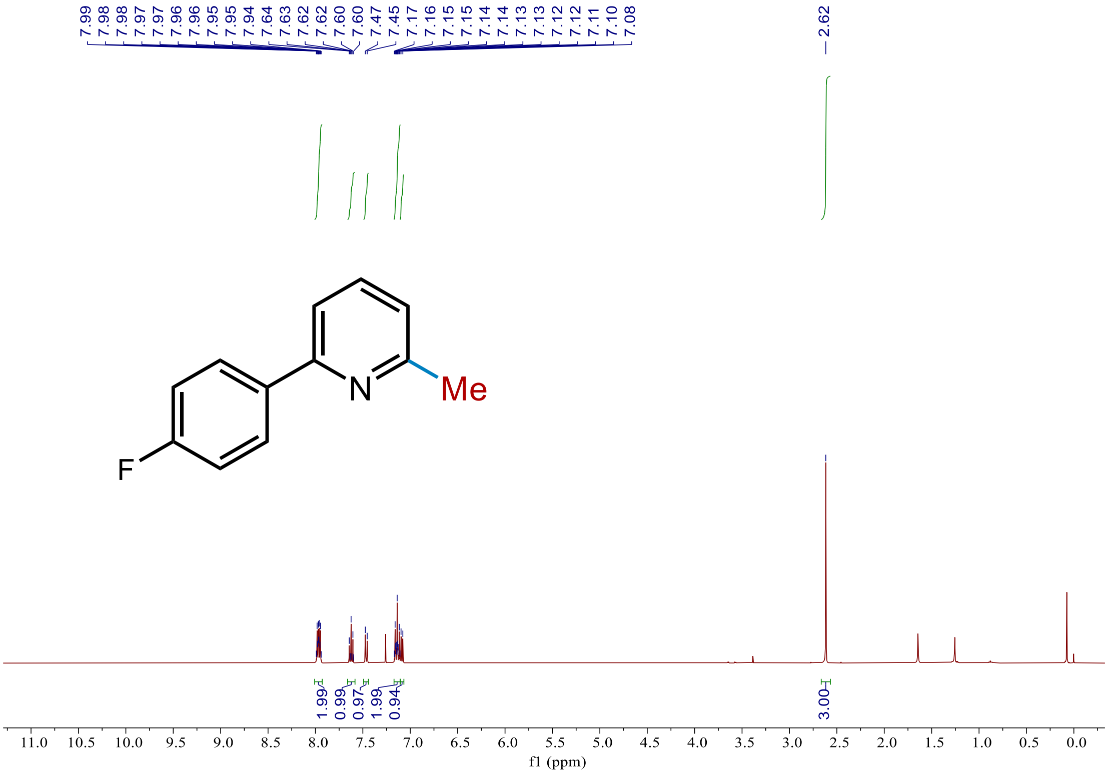
[1]H NMR (400 MHz, Chloroform-*d*) of compound **5**



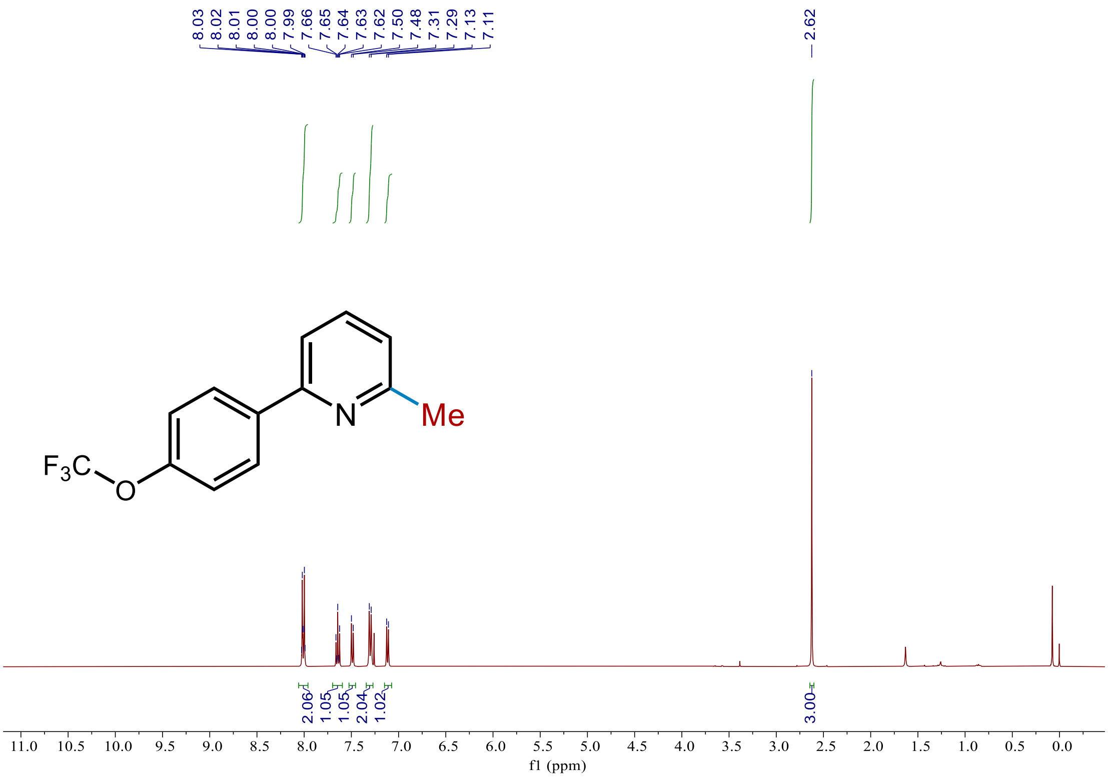
[1]H NMR (400 MHz, Chloroform-*d*) of compound **6**



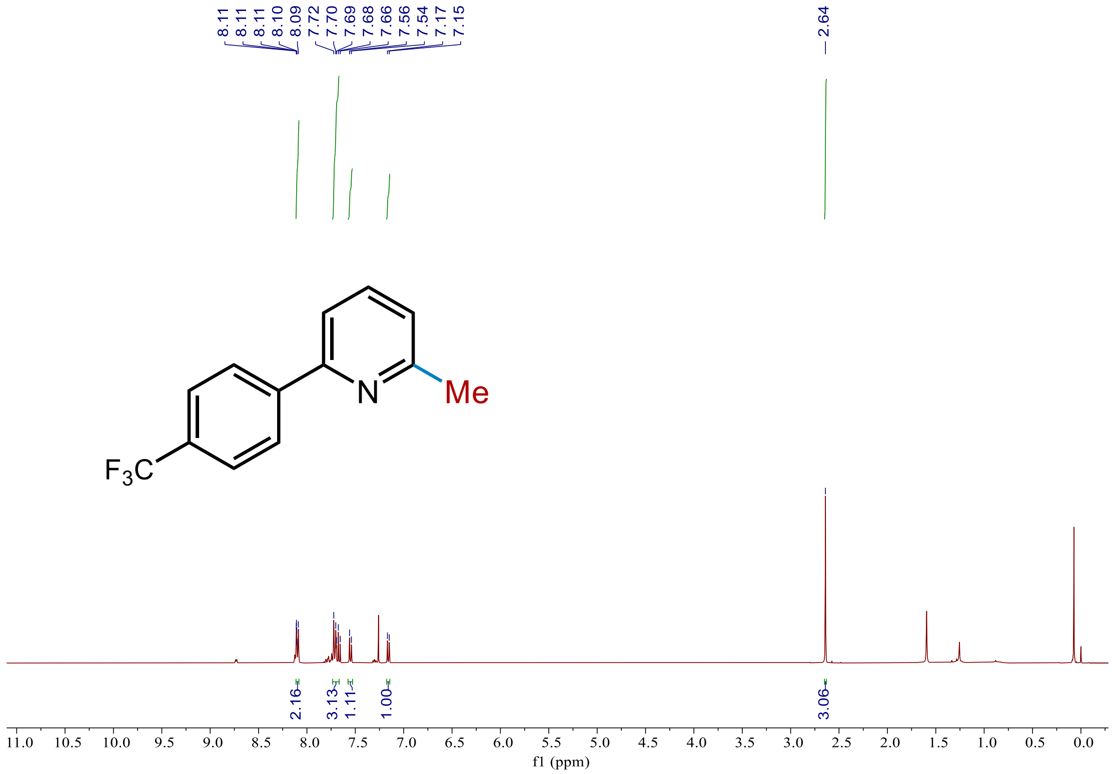
[1]H NMR (400 MHz, Chloroform-*d*) of compound **7**



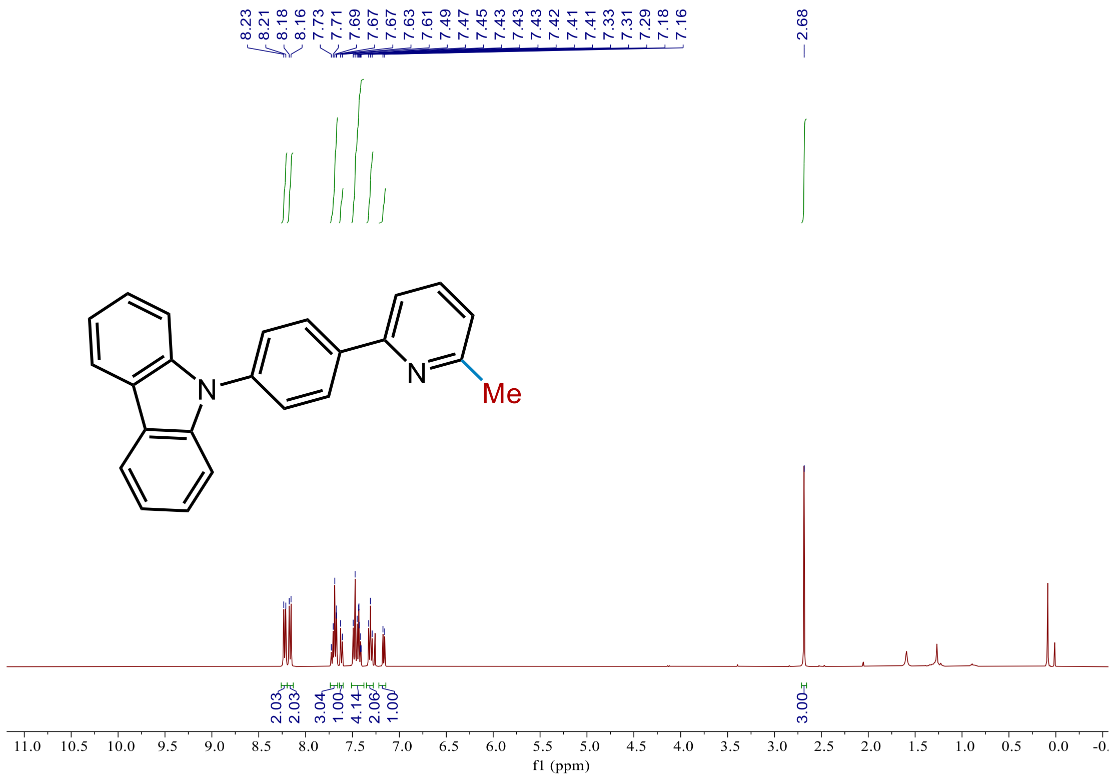
[1]H NMR (400 MHz, Chloroform-*d*) of compound **8**



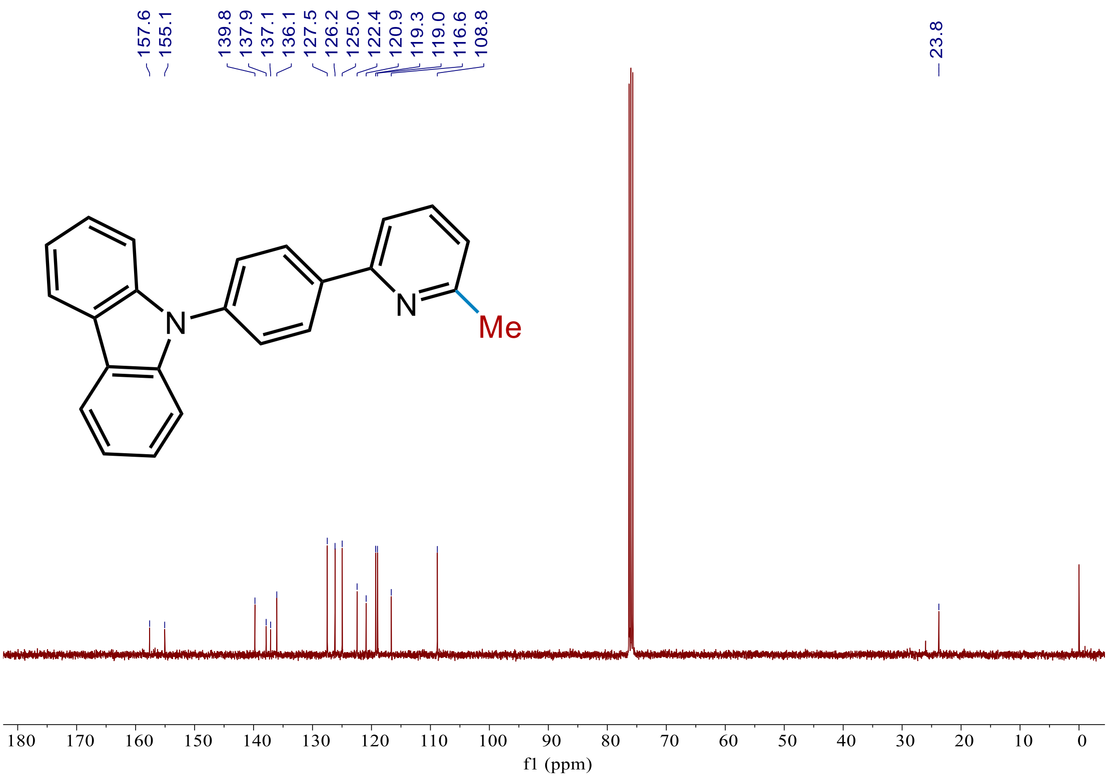
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **9**



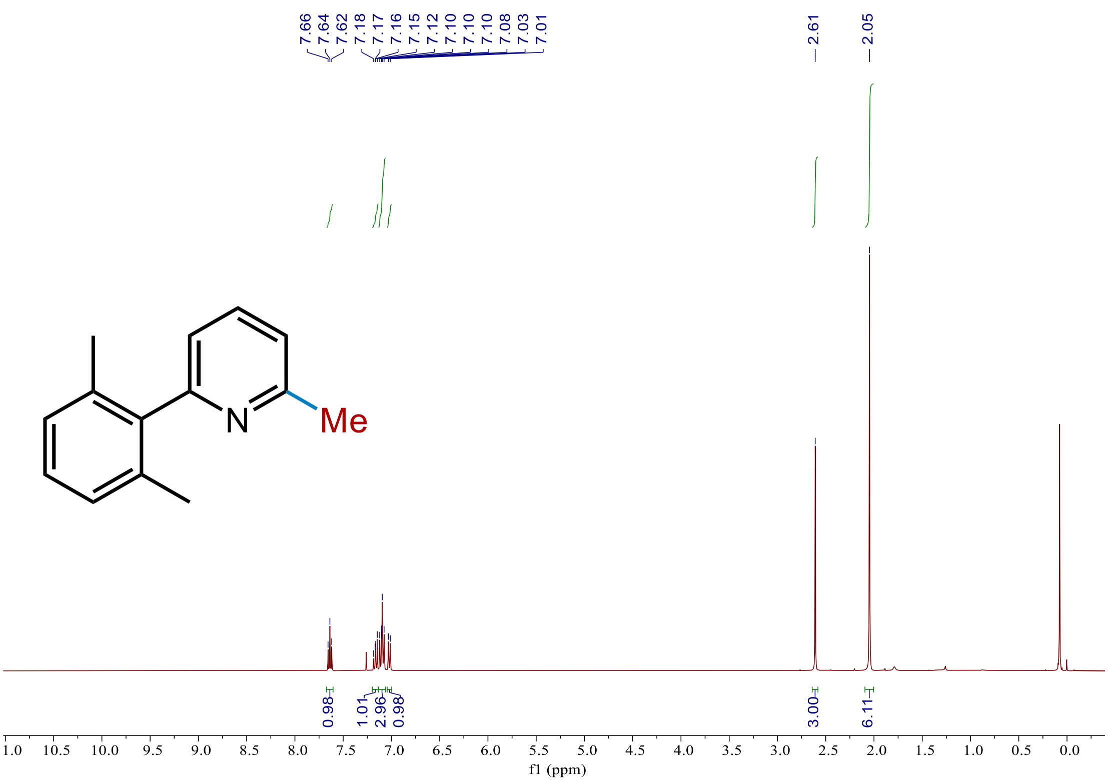
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **10**



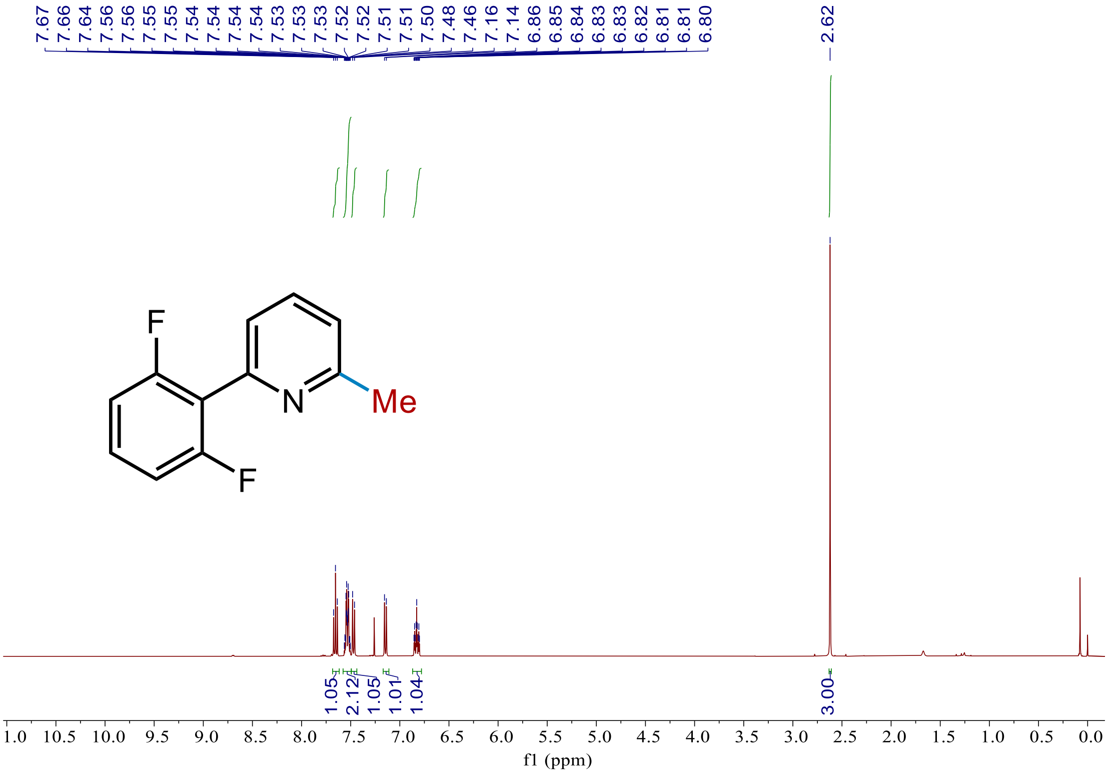
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **10**



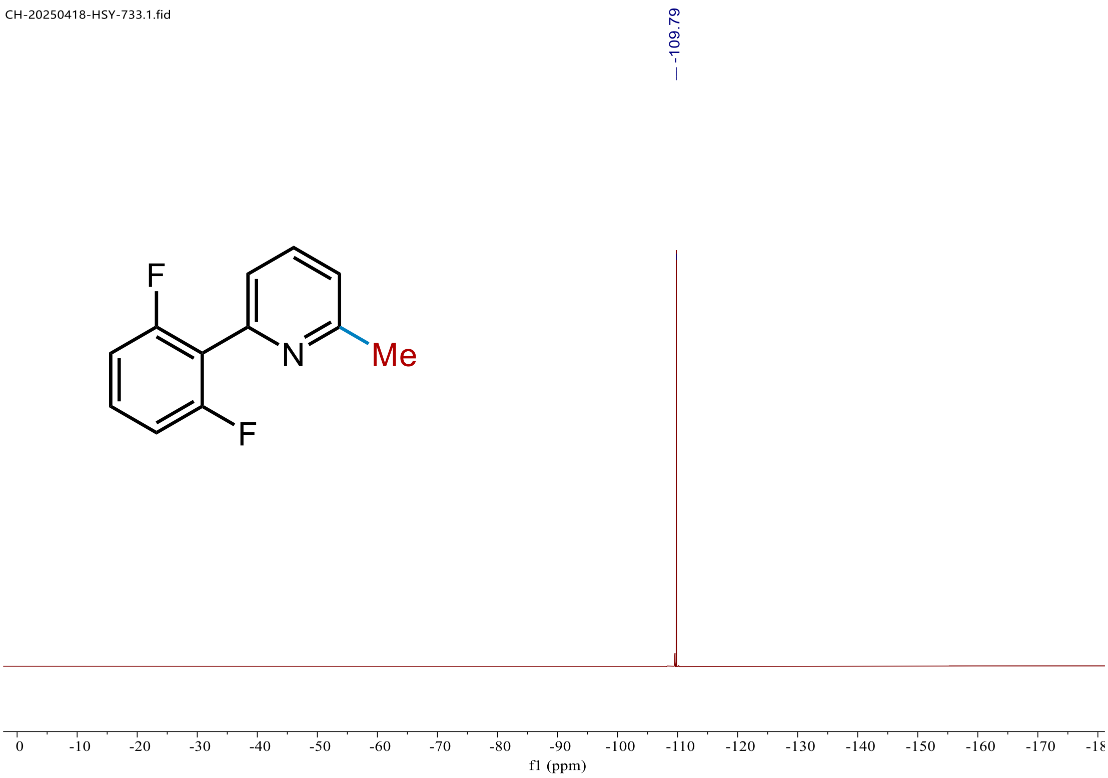
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **11**



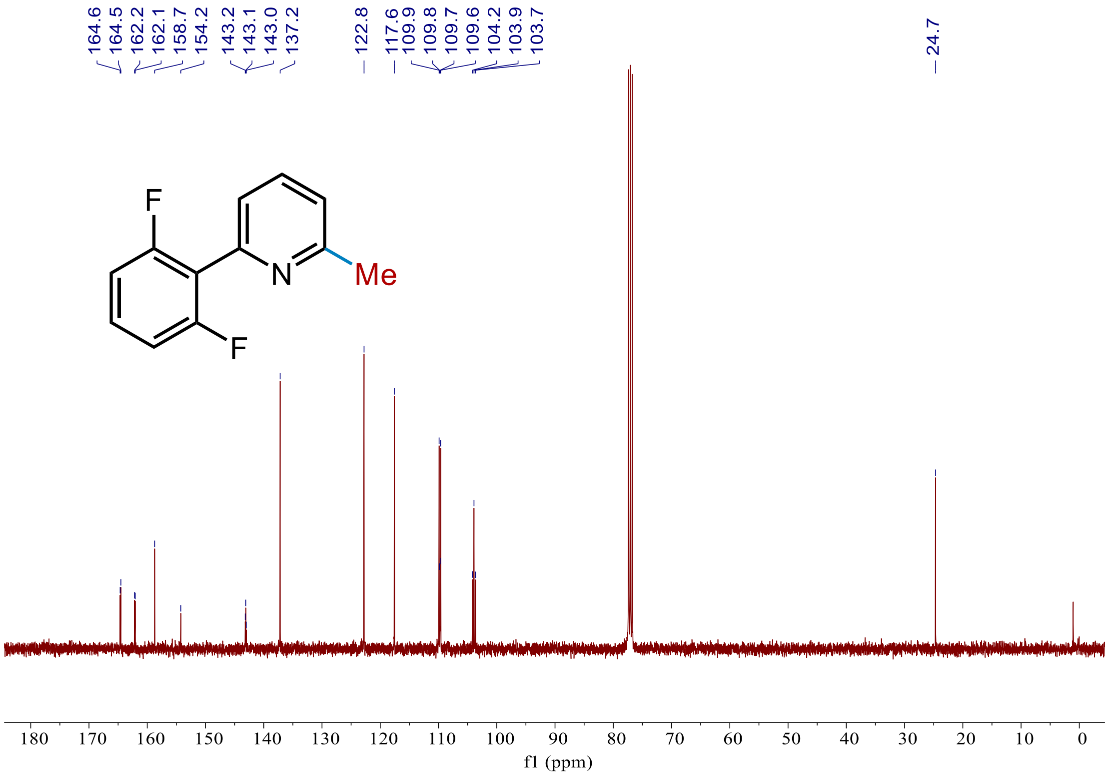
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **12**



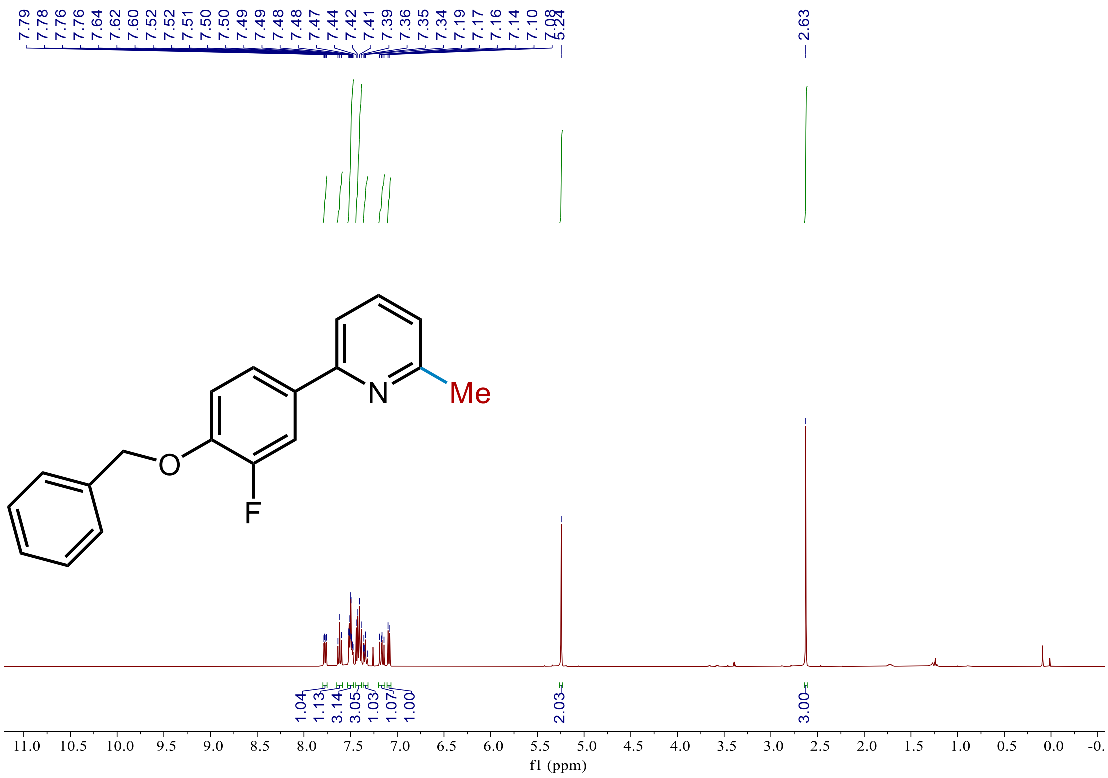
^{19}F NMR (400 MHz, Chloroform-*d*) of compound **12**



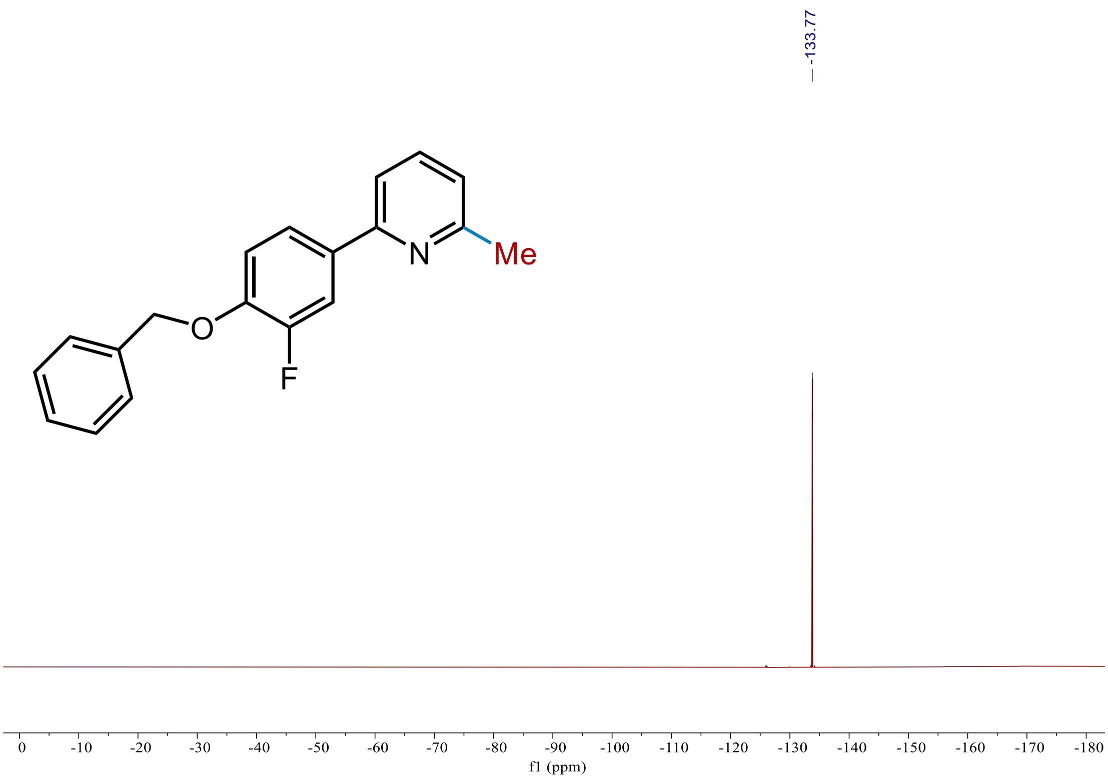
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **12**



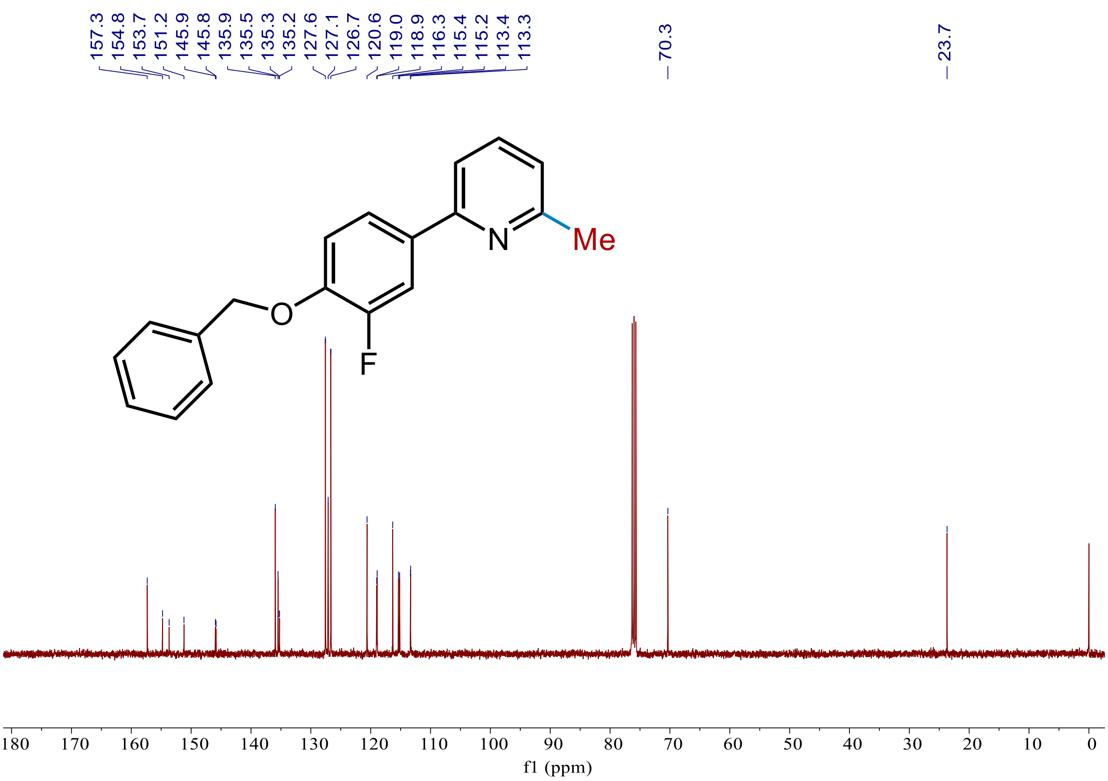
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **13**



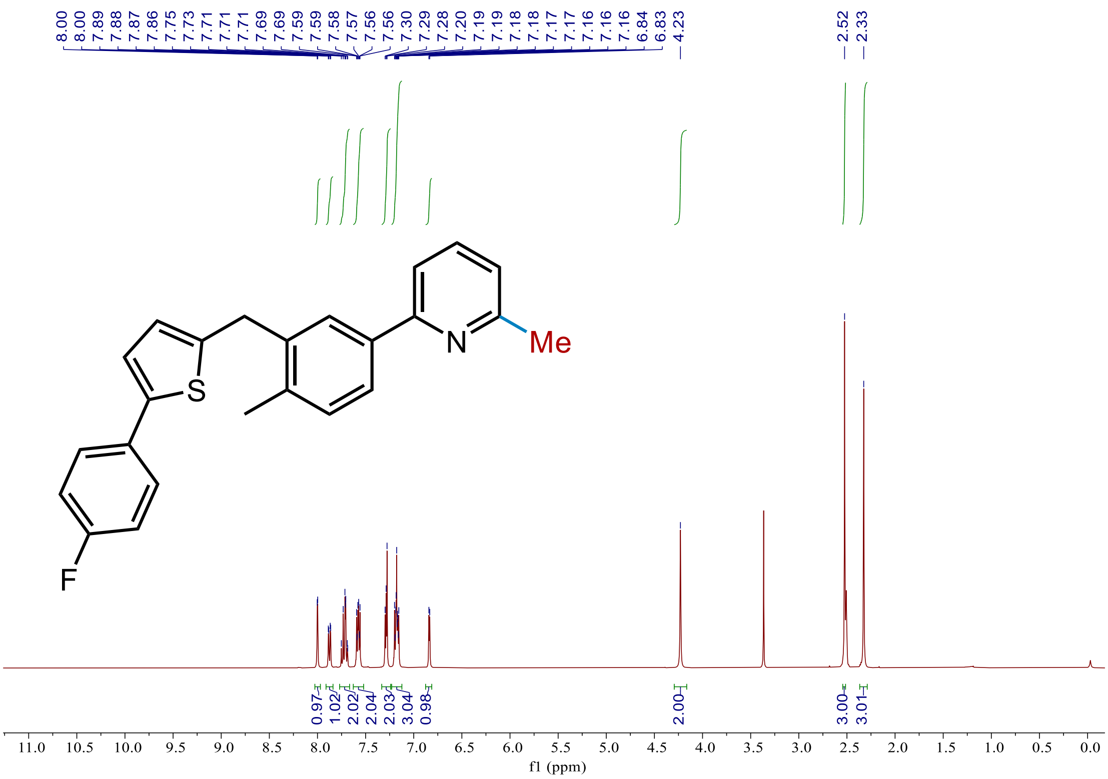
^{19}F NMR (400 MHz, Chloroform-*d*) of compound **13**



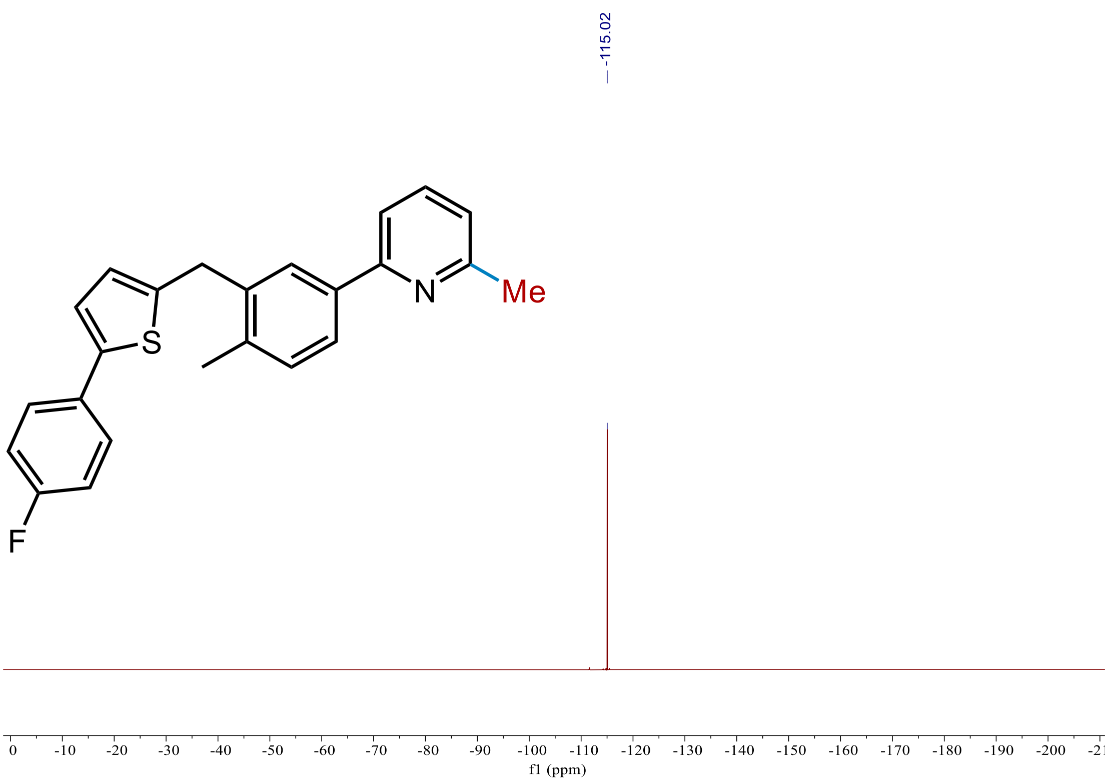
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **13**



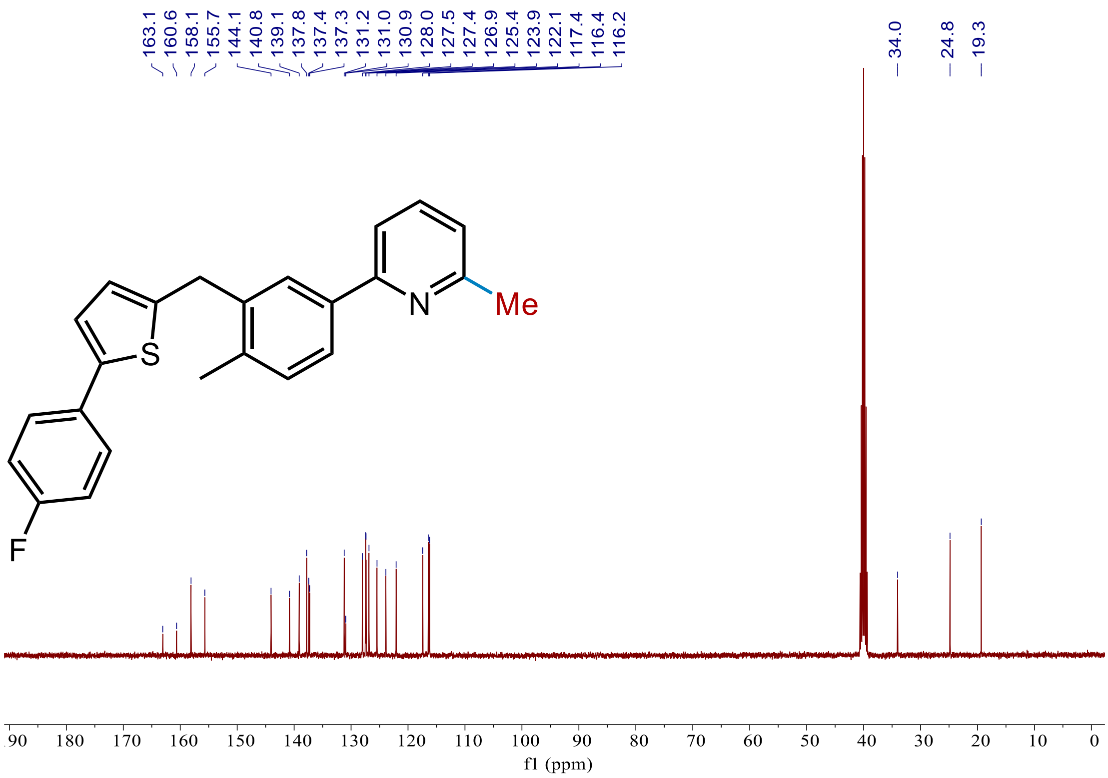
^{1}H NMR (400 MHz, DMSO-d_6) of compound **14**



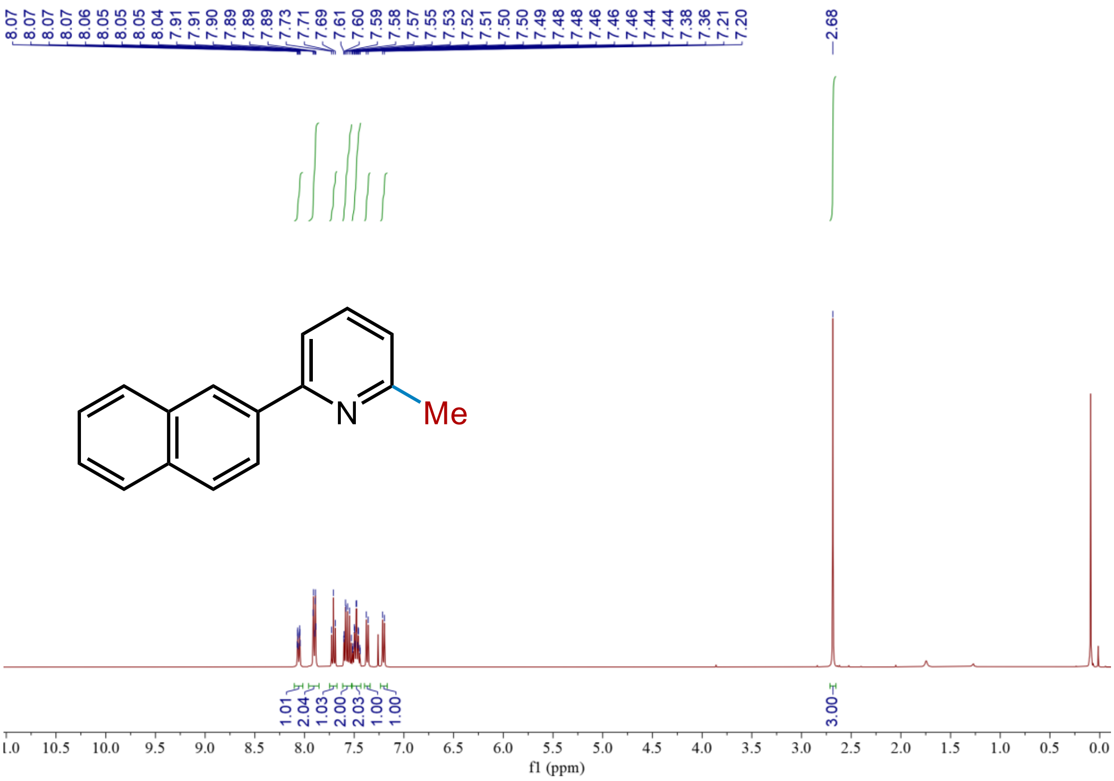
^{19}F NMR (400 MHz, DMSO-d_6) of compound **14**



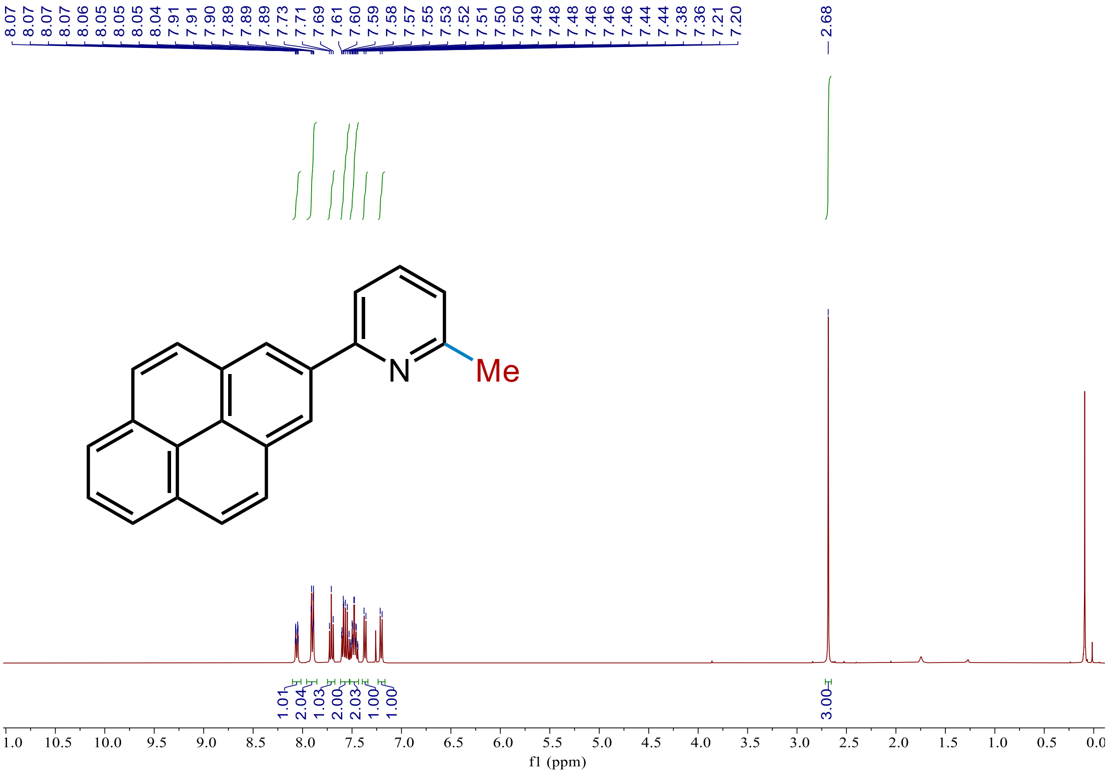
^{13}C NMR (101 MHz, DMSO-d_6) of compound **14**



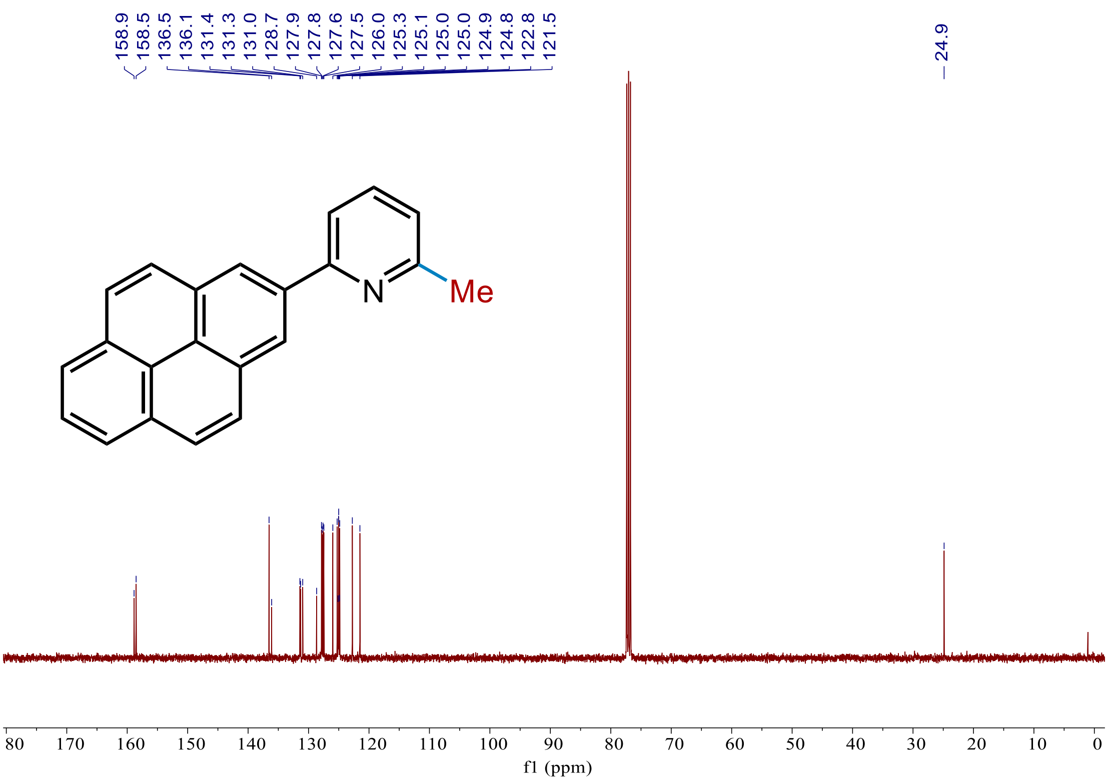
^{1}H NMR (400 MHz, Chloroform-d) of compound **15**



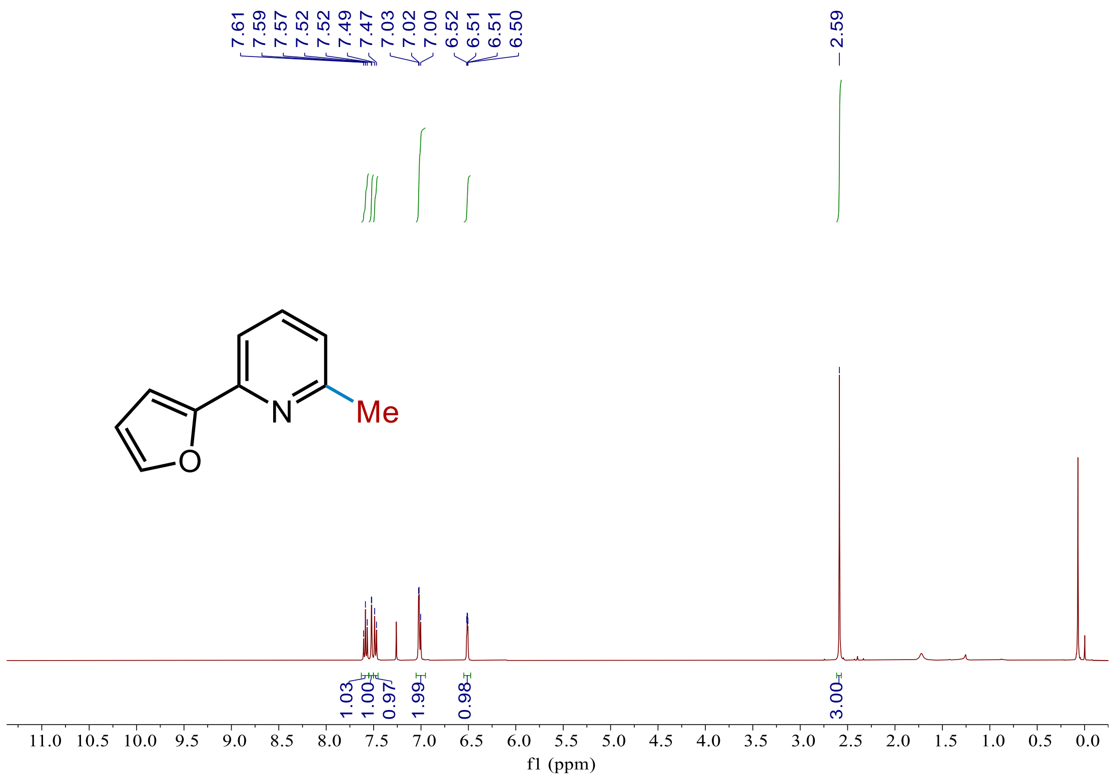
[1]H NMR (400 MHz, Chloroform-*d*) of compound **16**



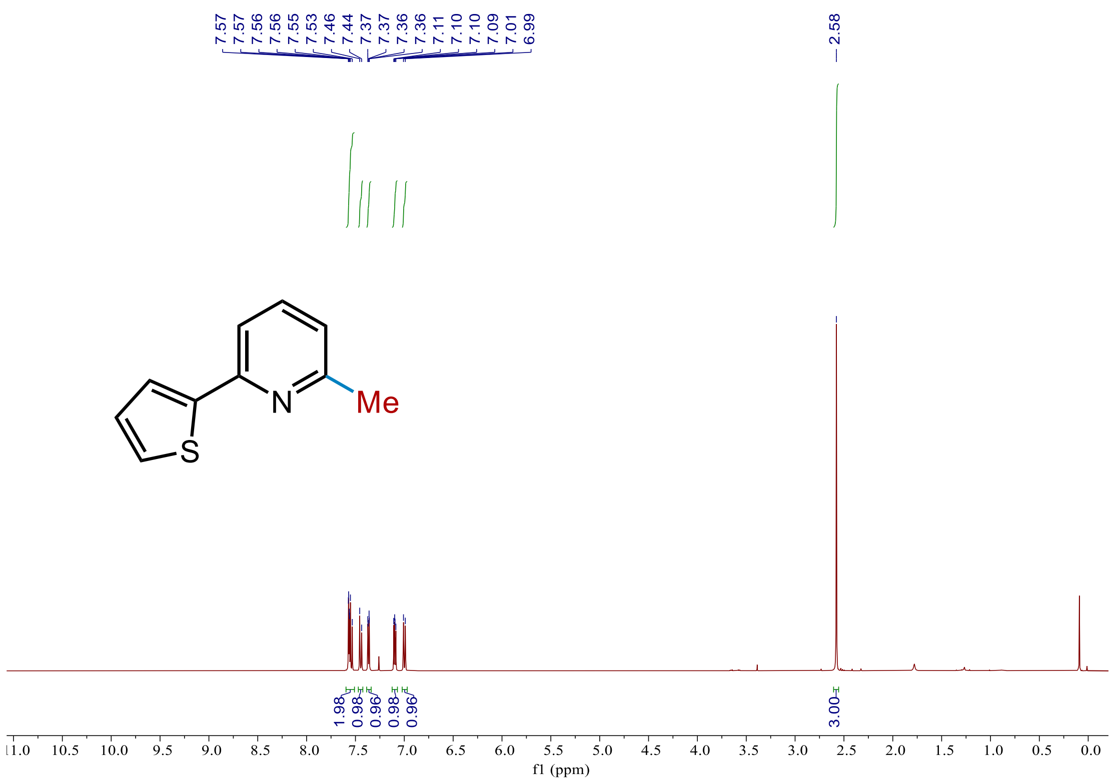
[13]C NMR (101 MHz, Chloroform-*d*) of compound **16**



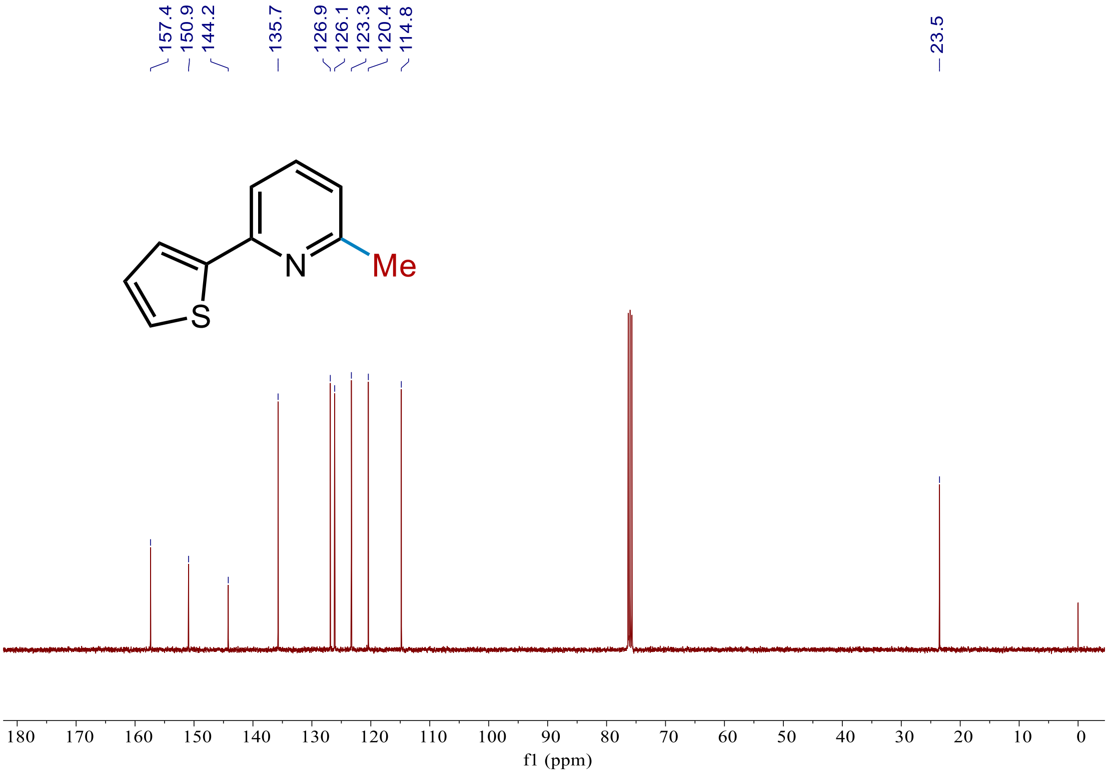
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **17**



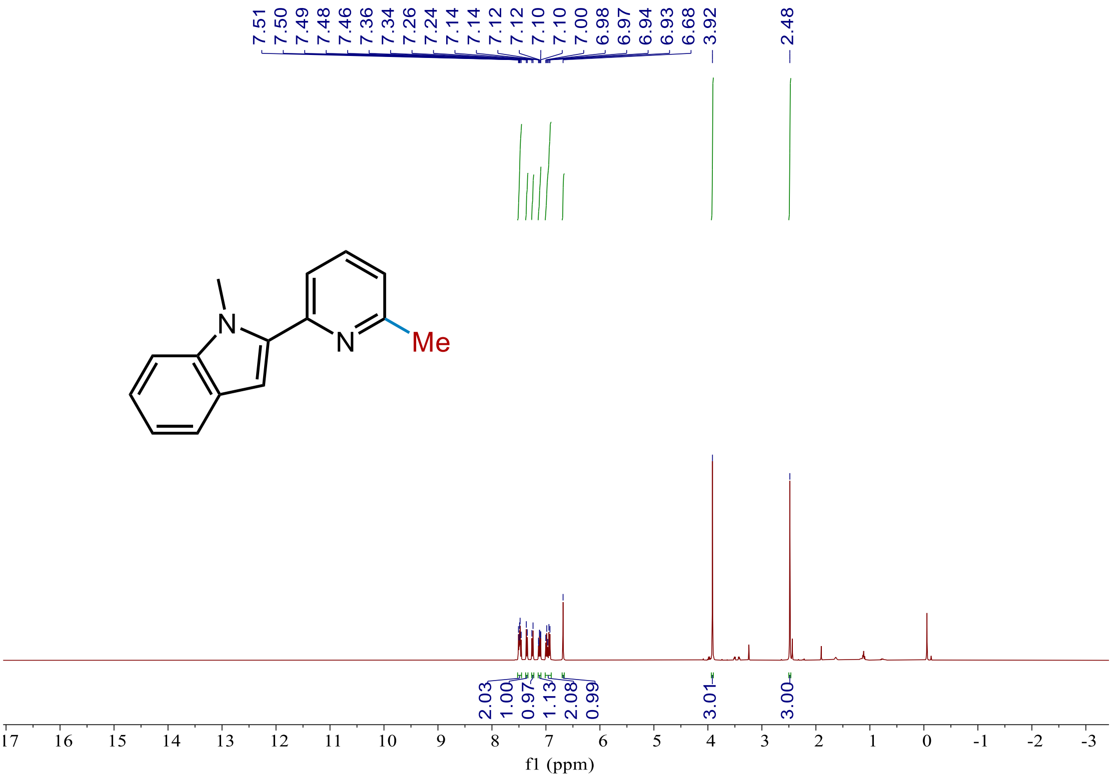
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **18**



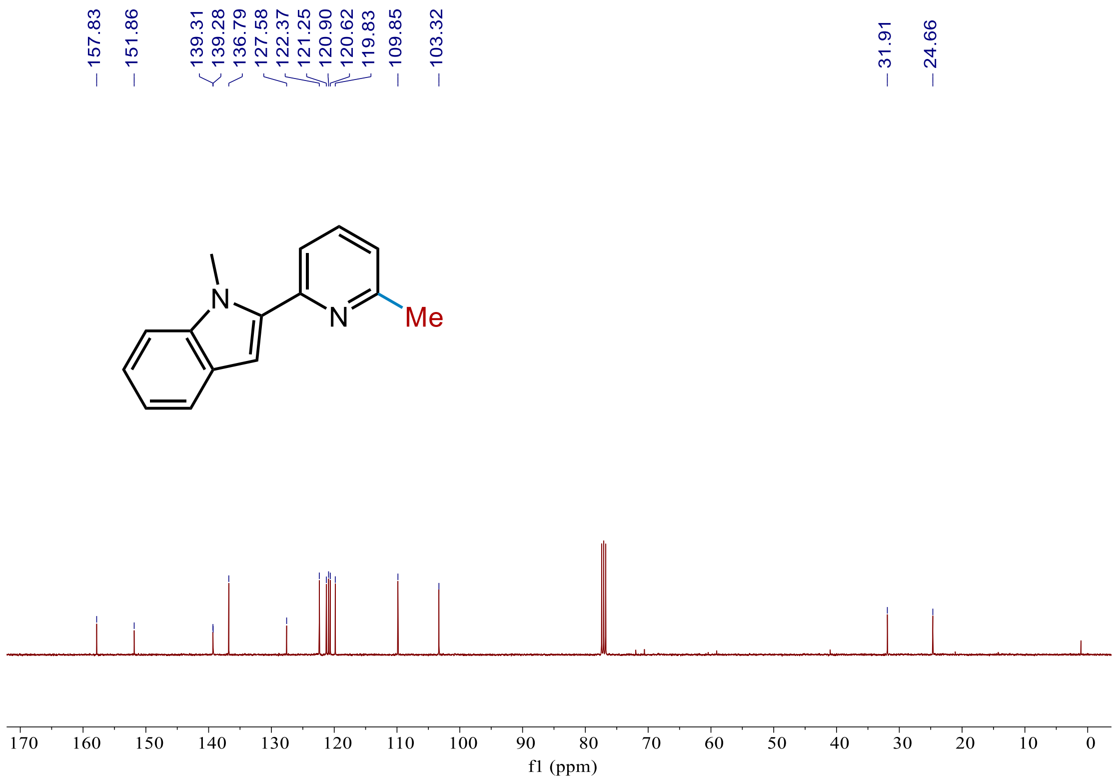
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **18**



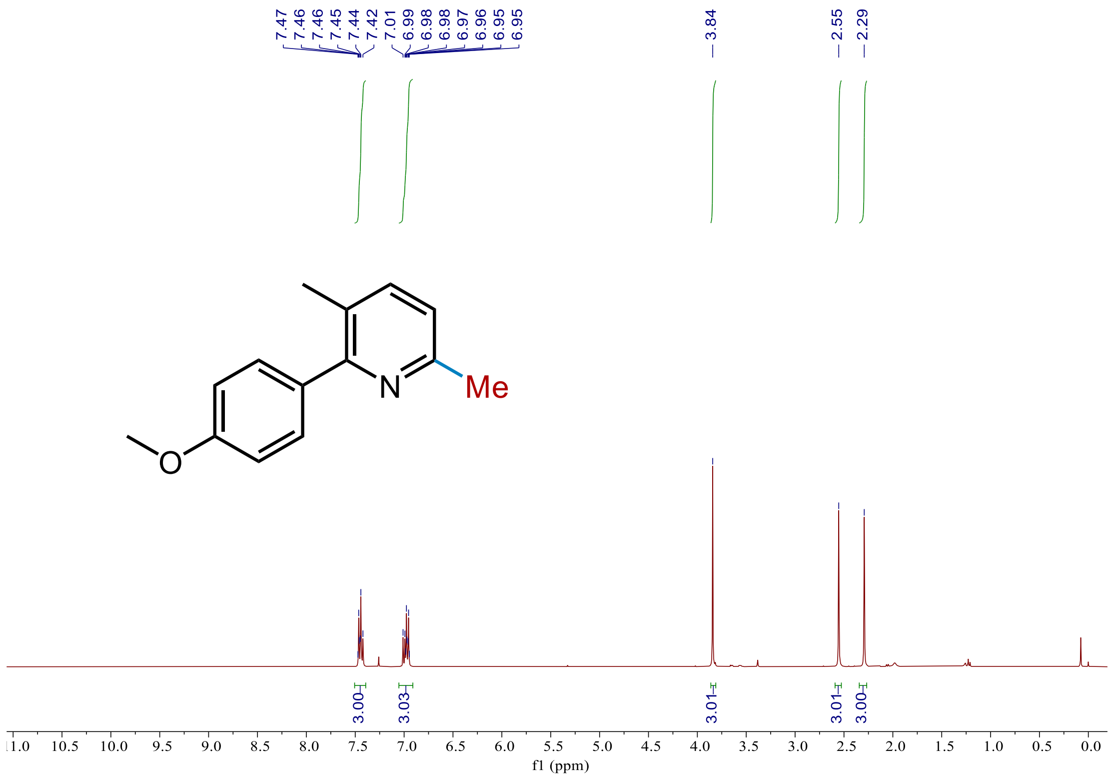
^{1}H NMR (101 MHz, Chloroform-*d*) of compound **19**



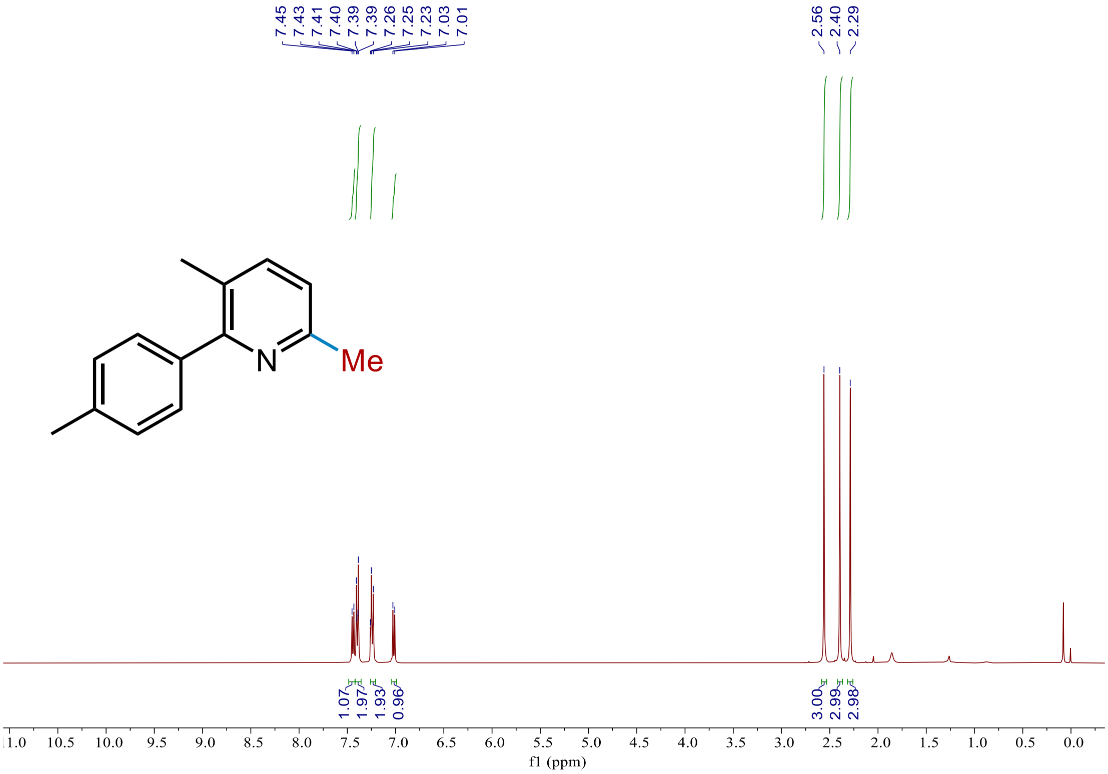
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **19**



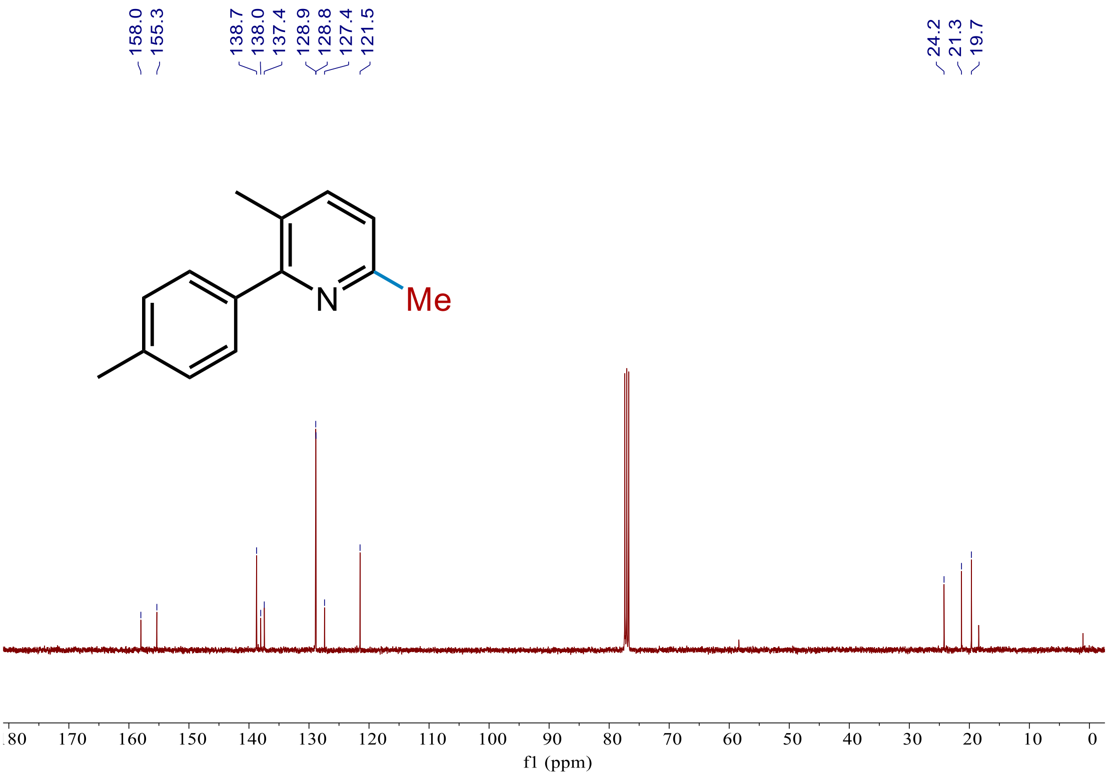
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **20**



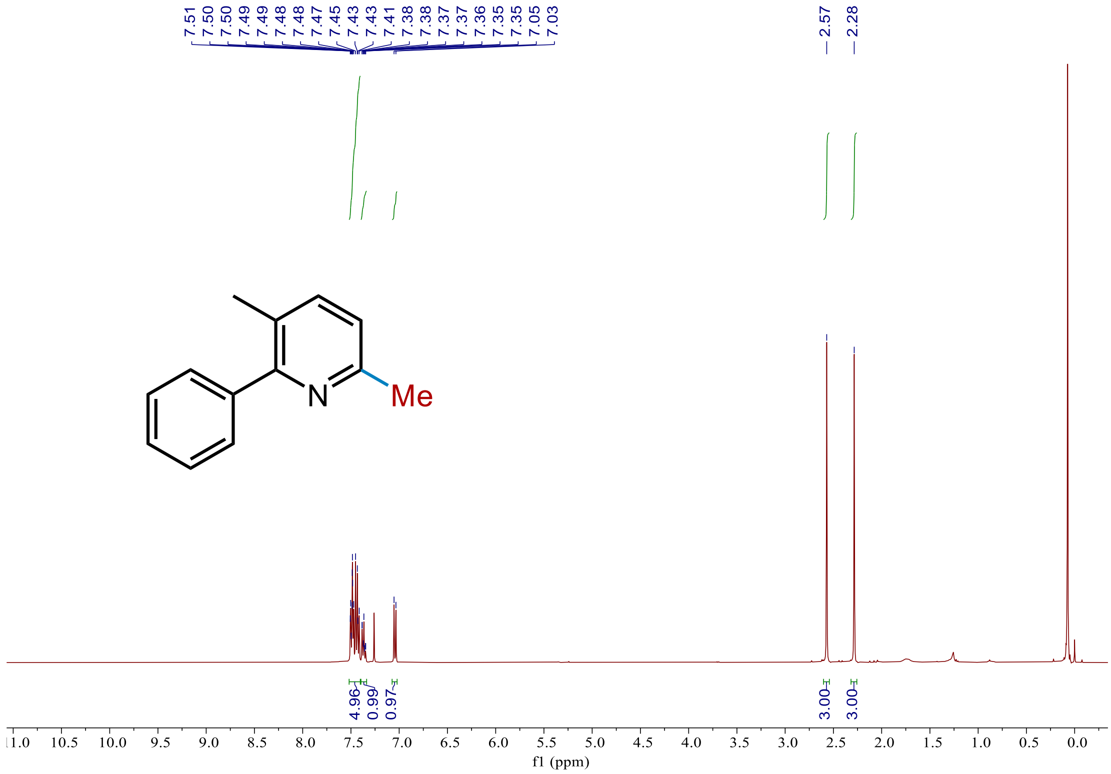
[1]H NMR (400 MHz, Chloroform-*d*) of compound **21**



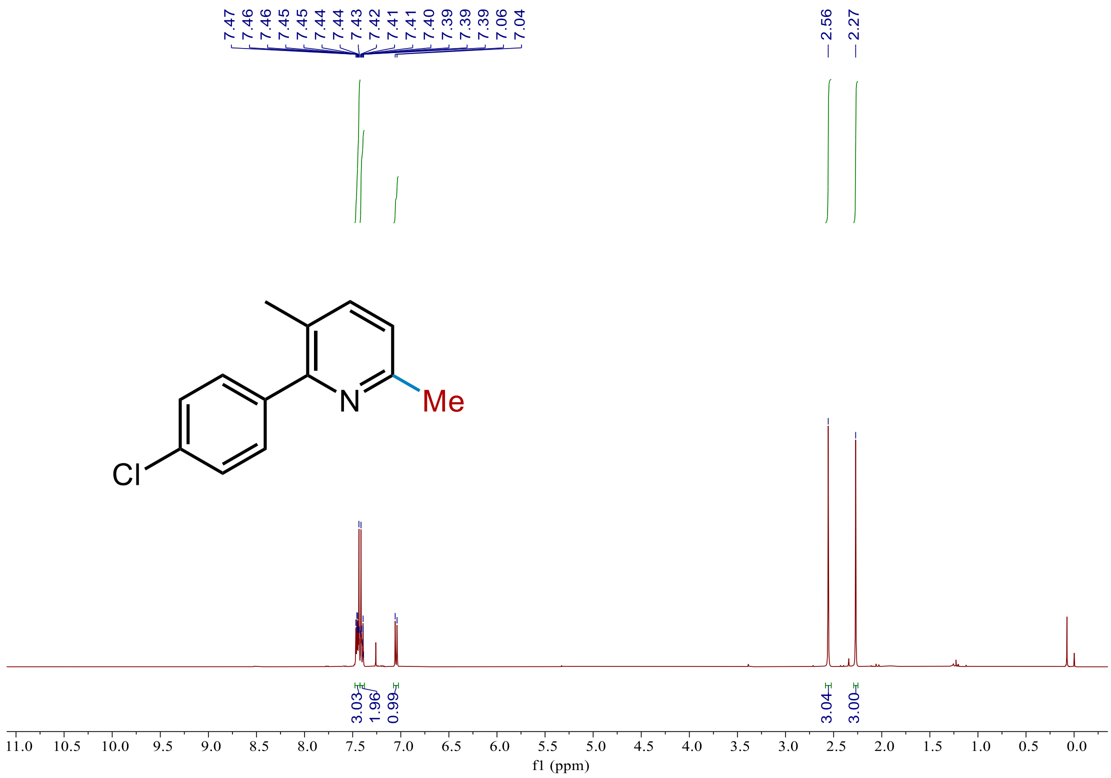
[13]C NMR (101 MHz, Chloroform-*d*) of compound **21**



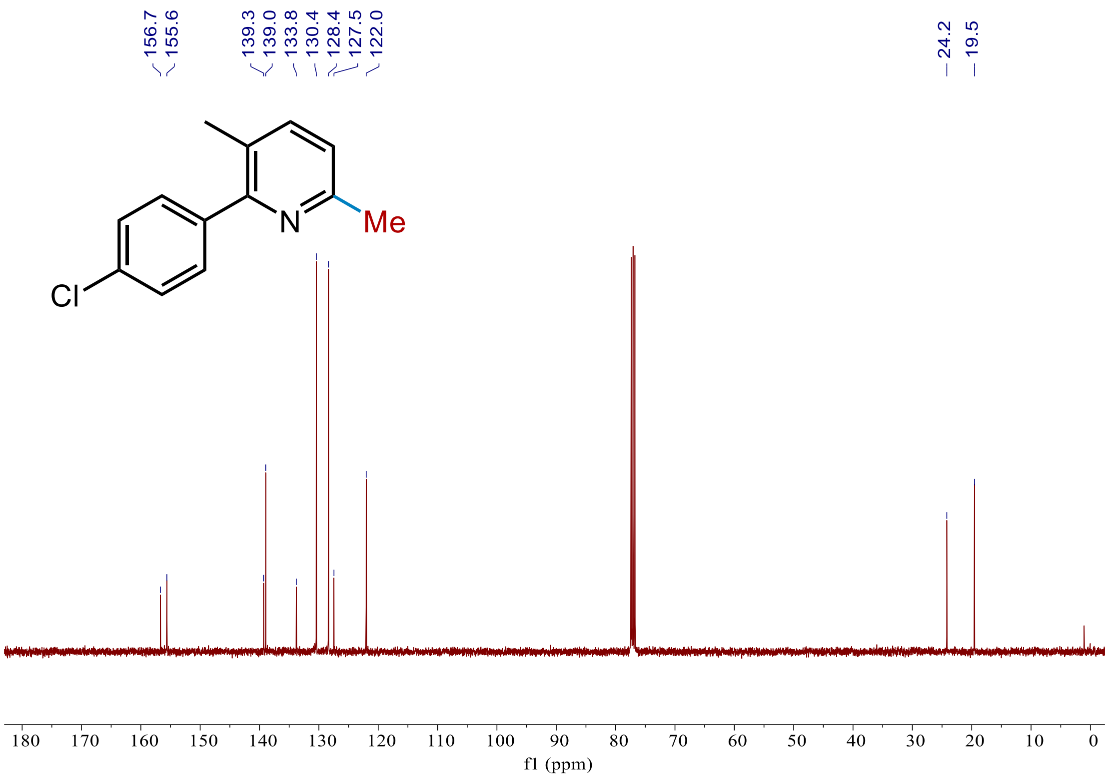
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **22**



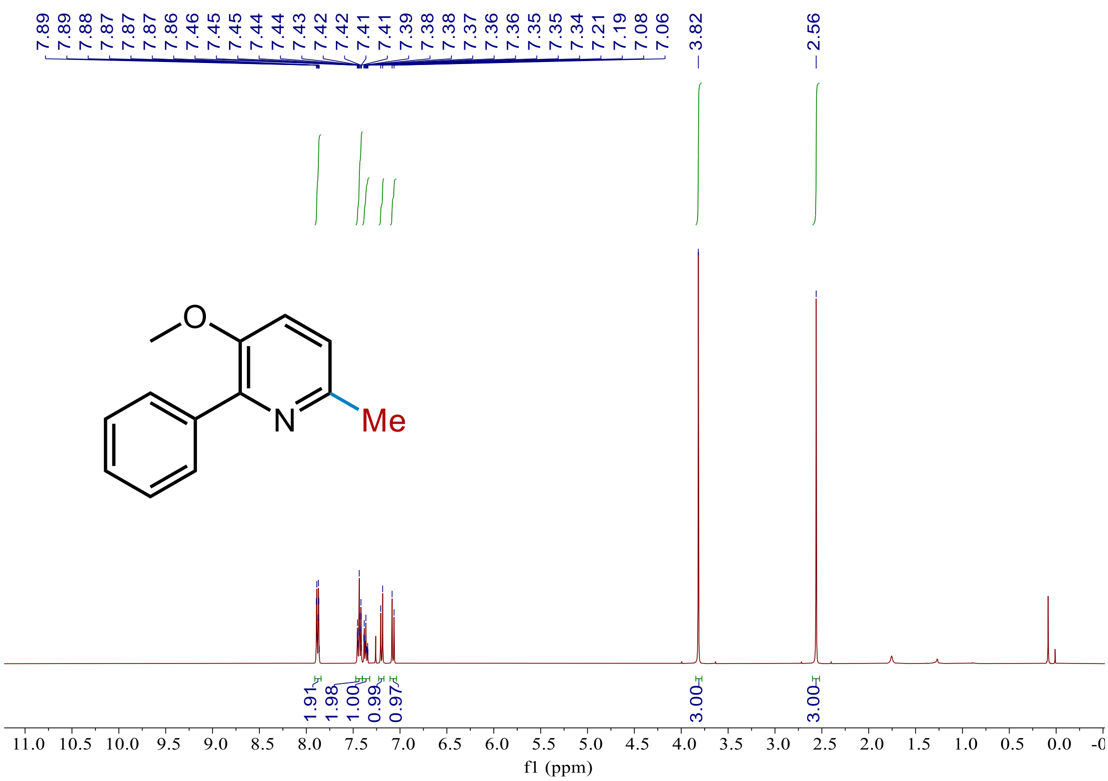
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **23**



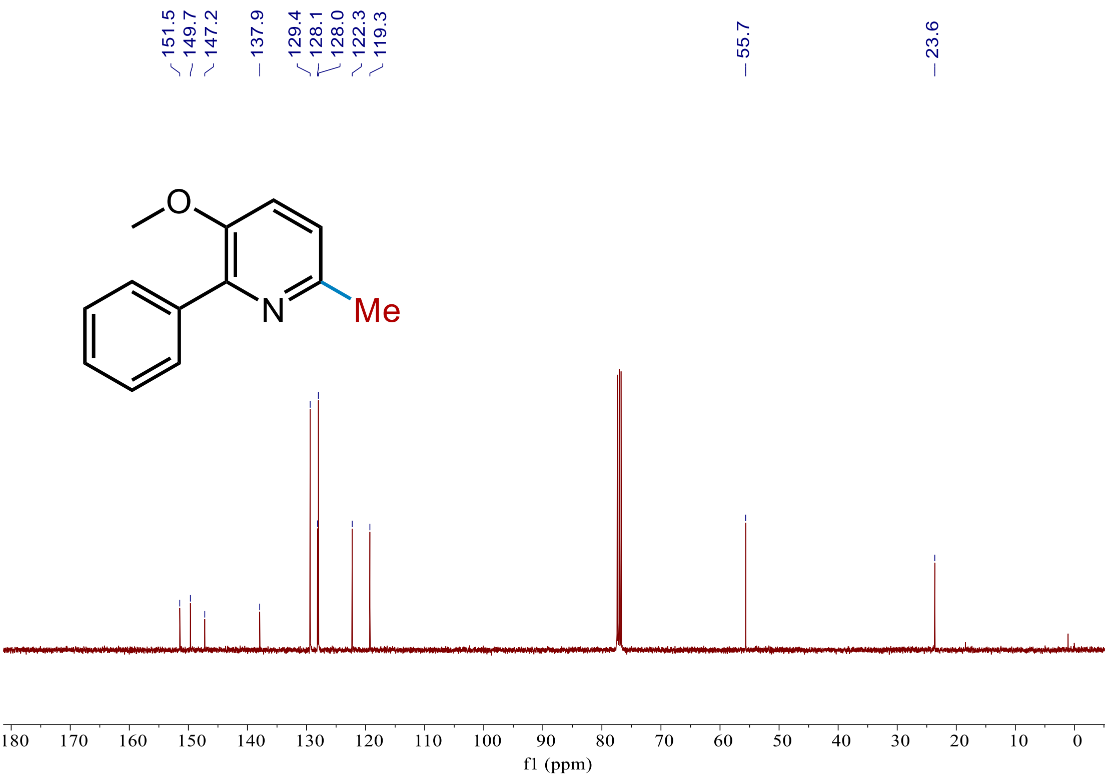
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **23**



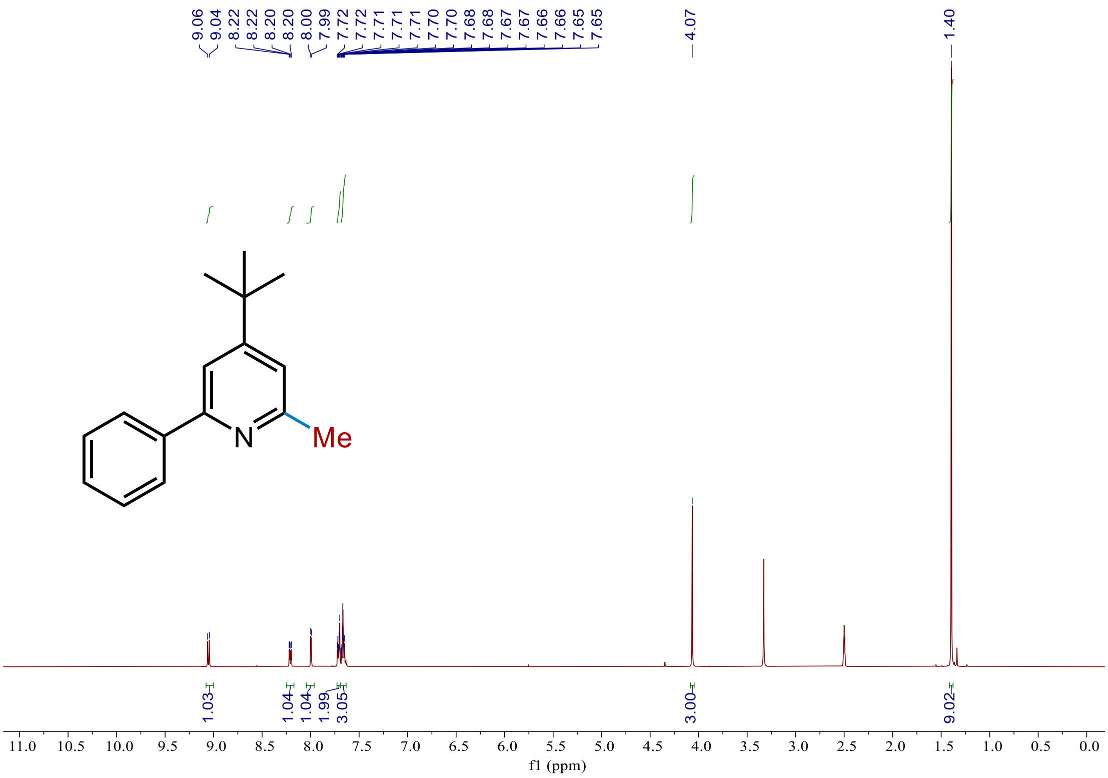
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **24**



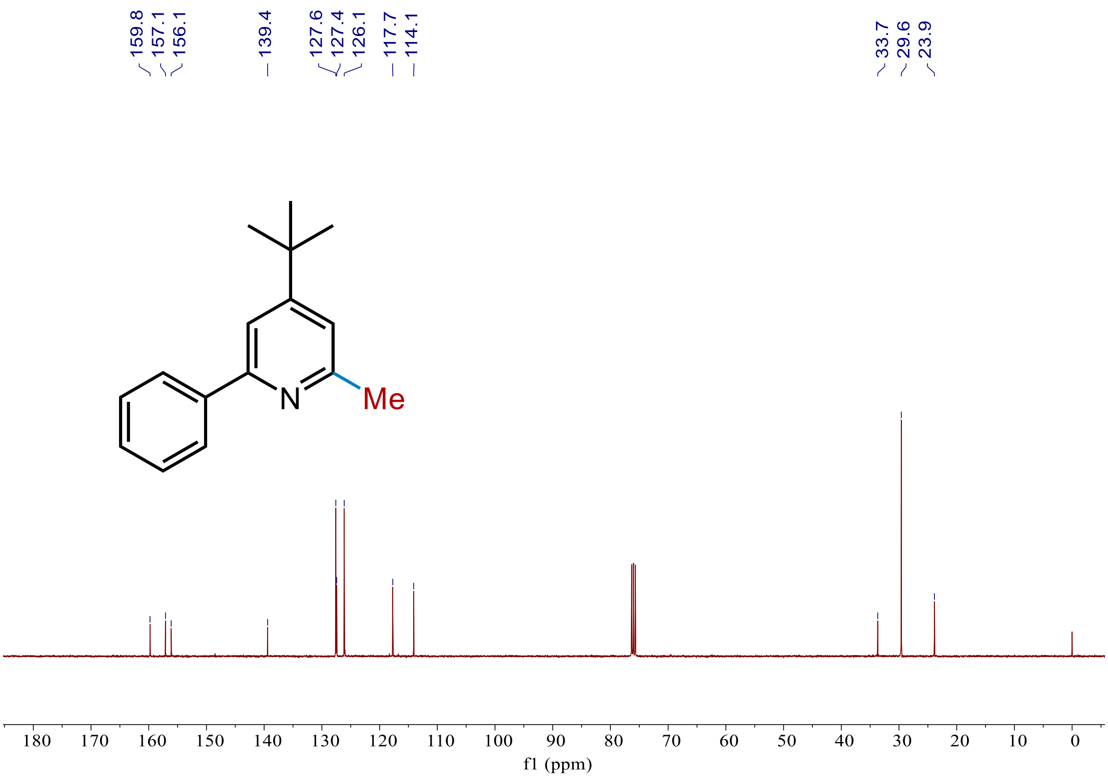
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **24**



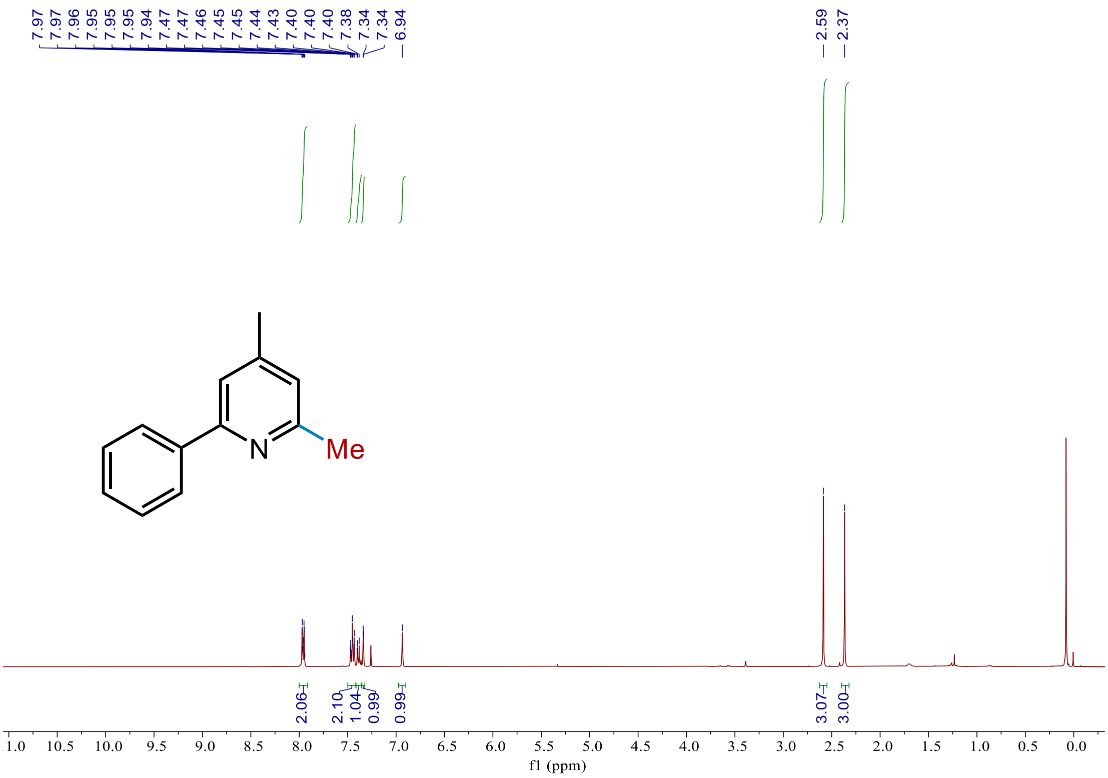
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **25**



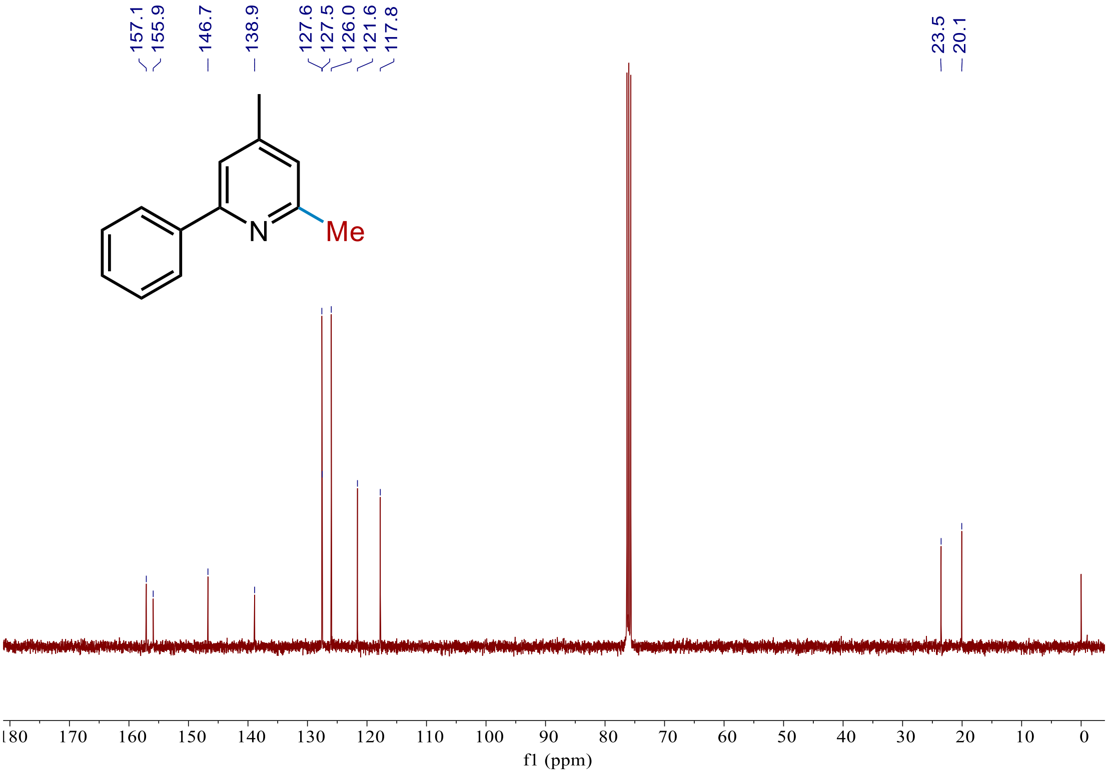
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **25**



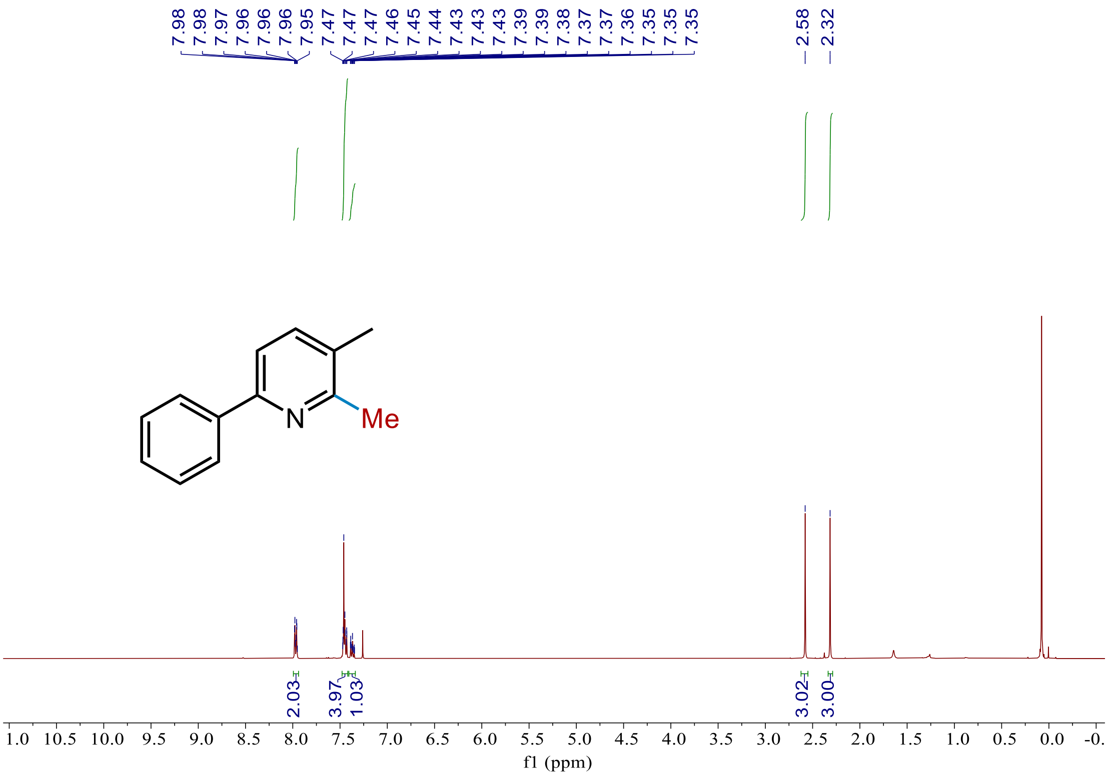
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **26**



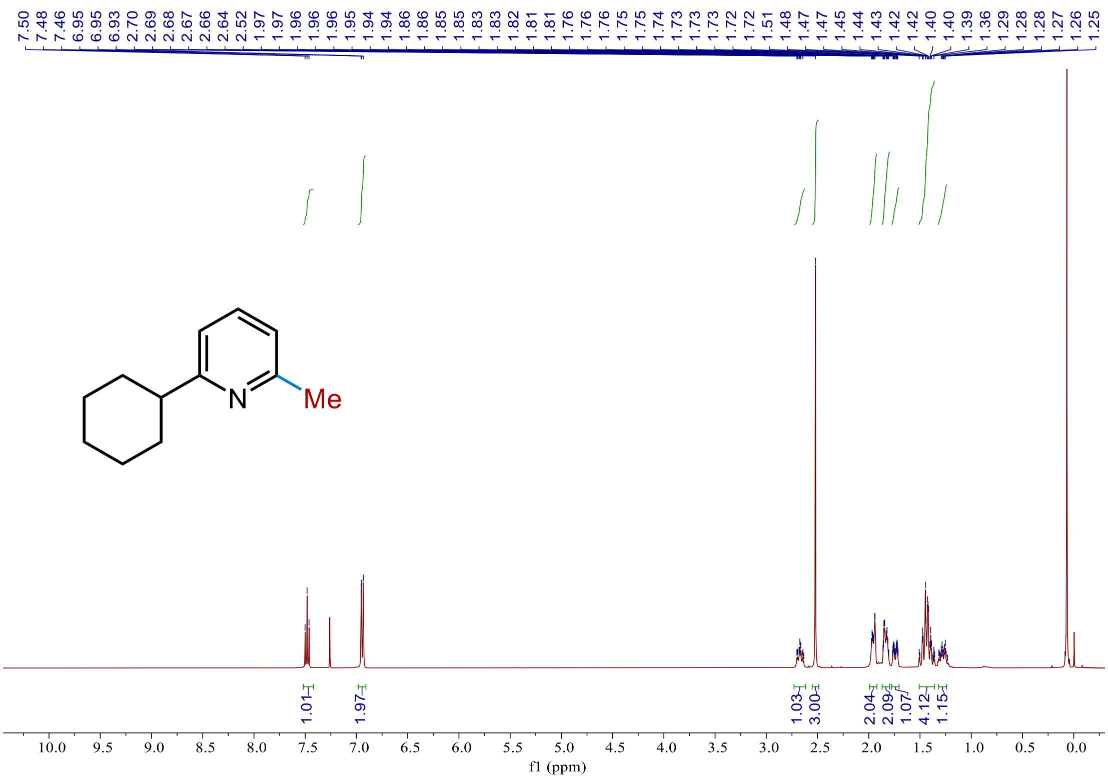
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **26**



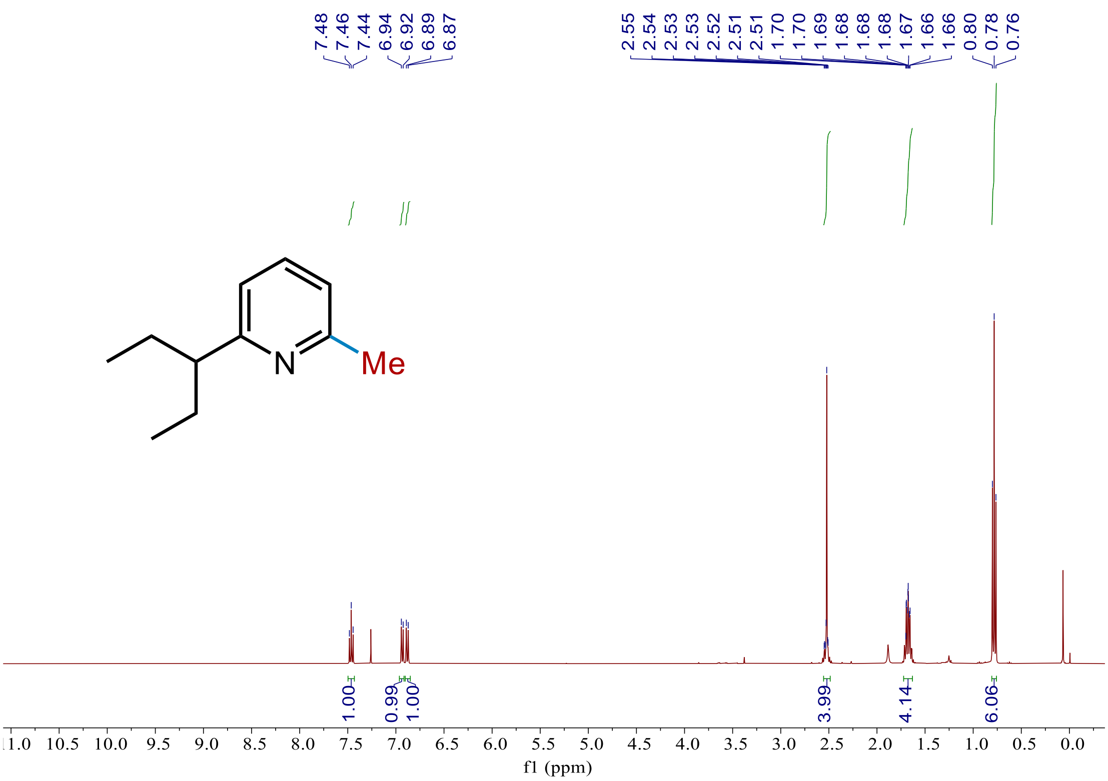
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **27**



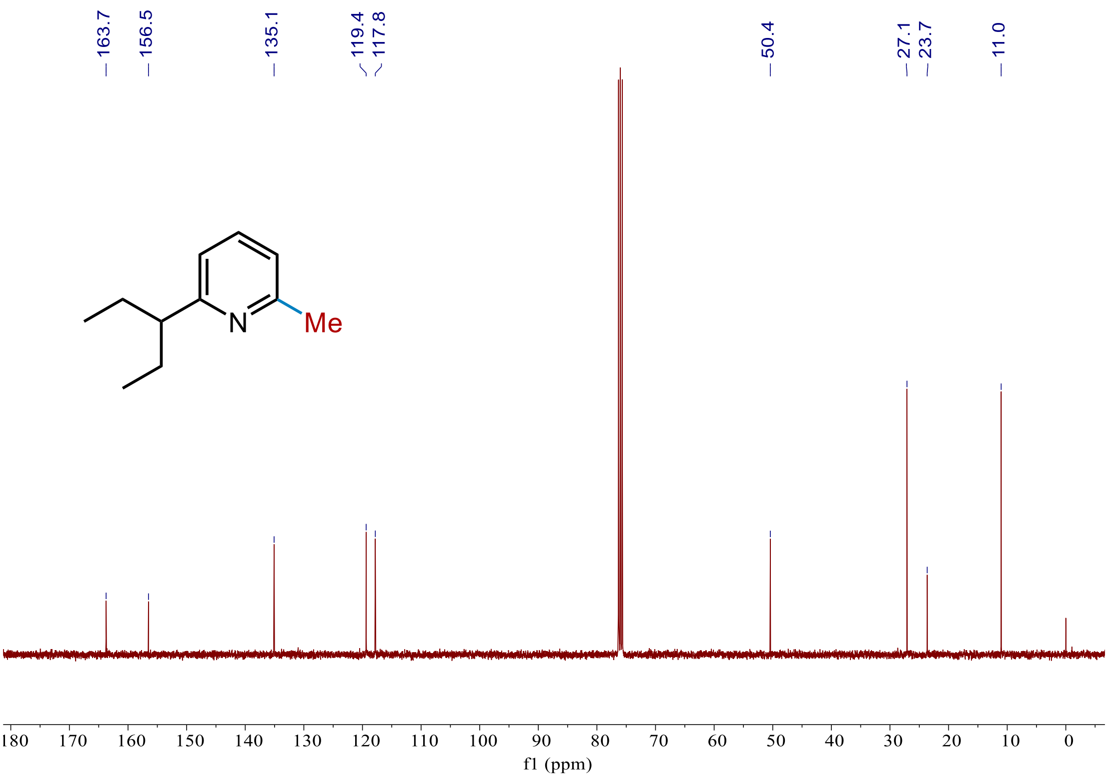
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **28**



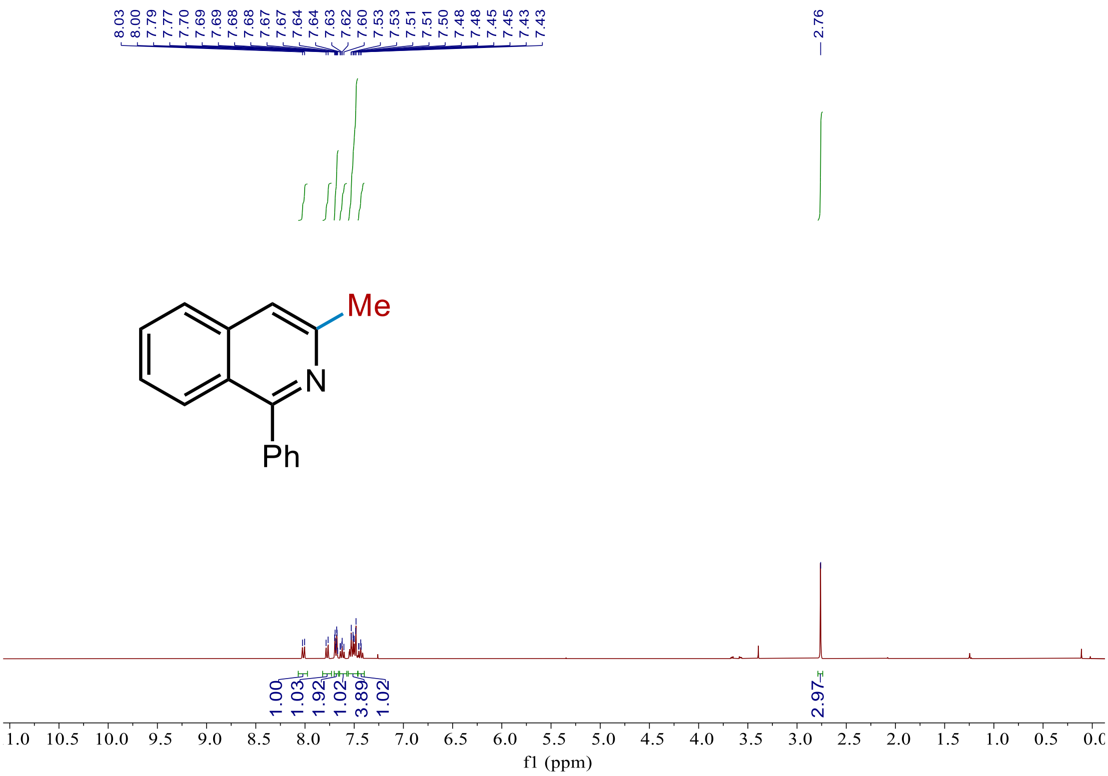
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **29**



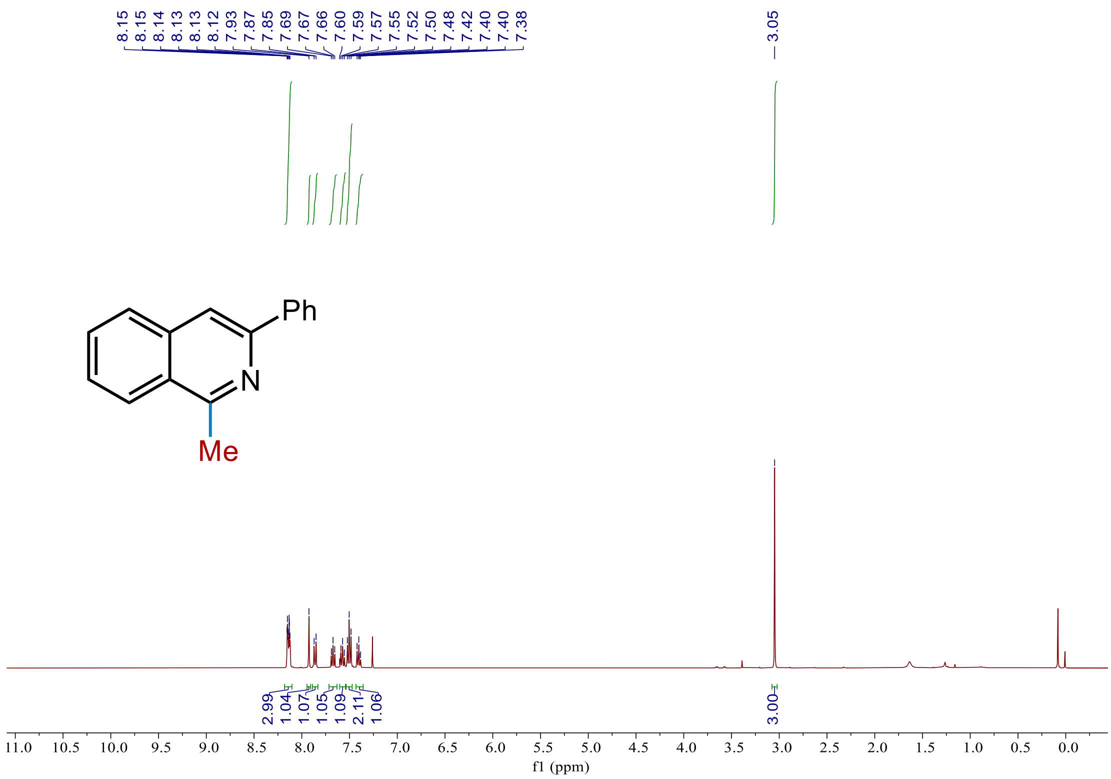
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **29**



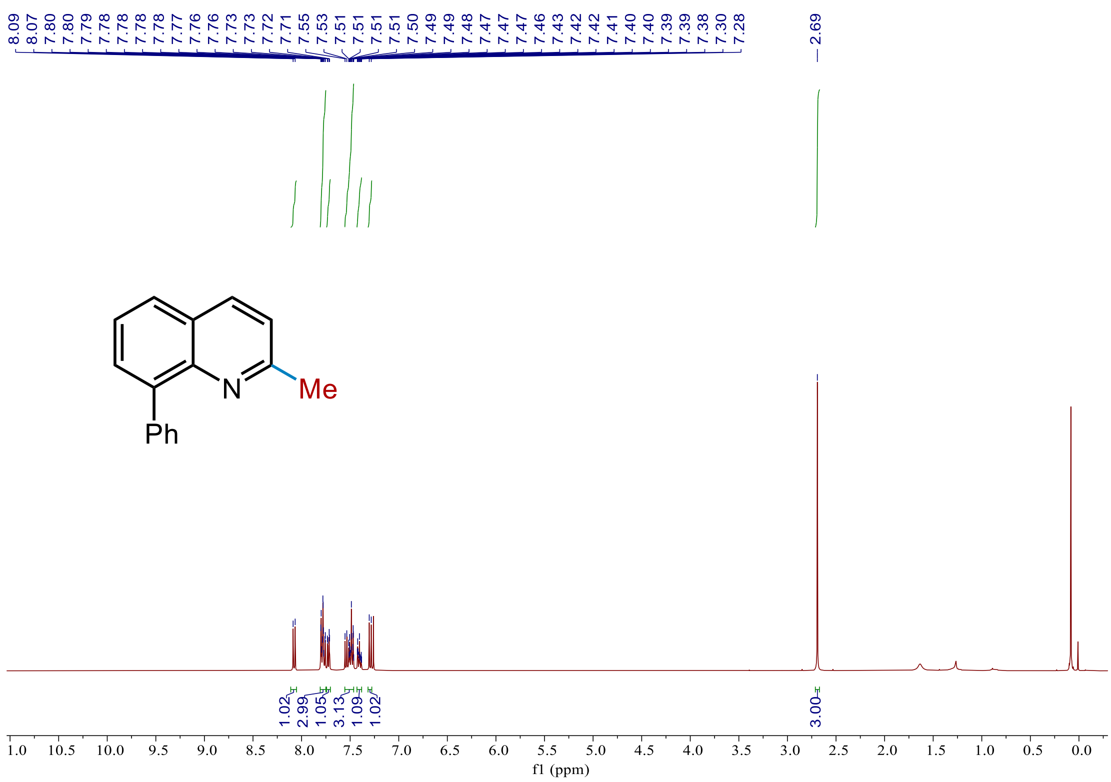
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **30**



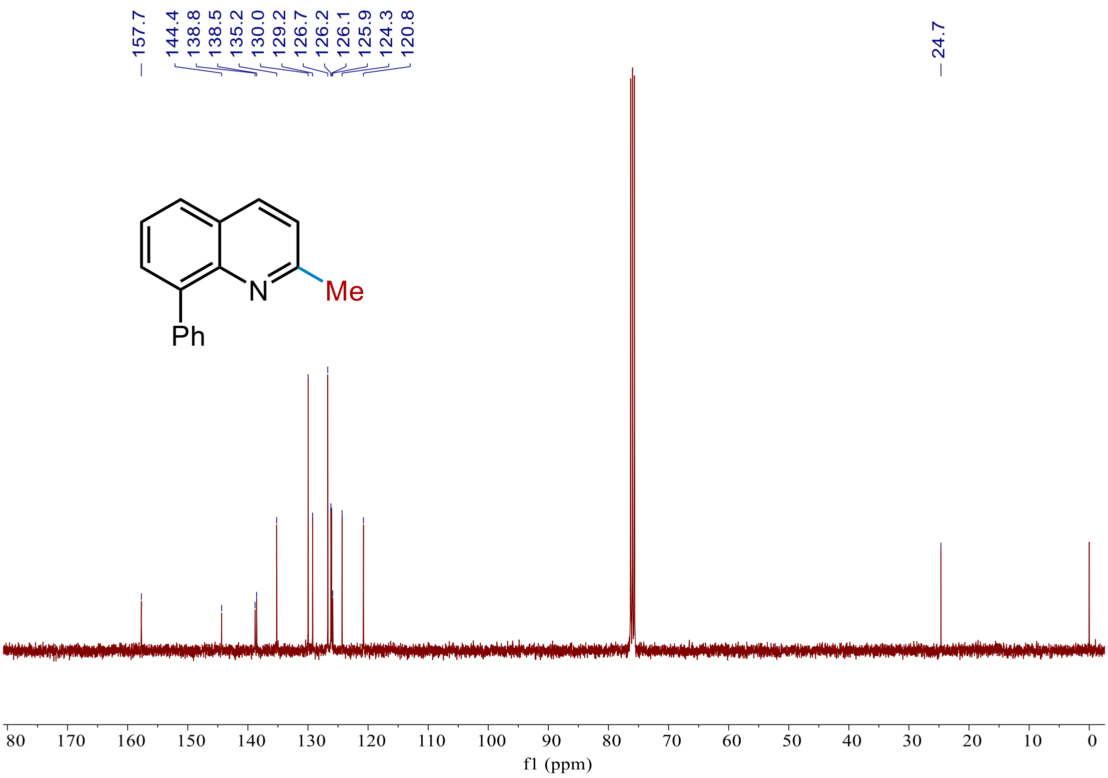
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **31**



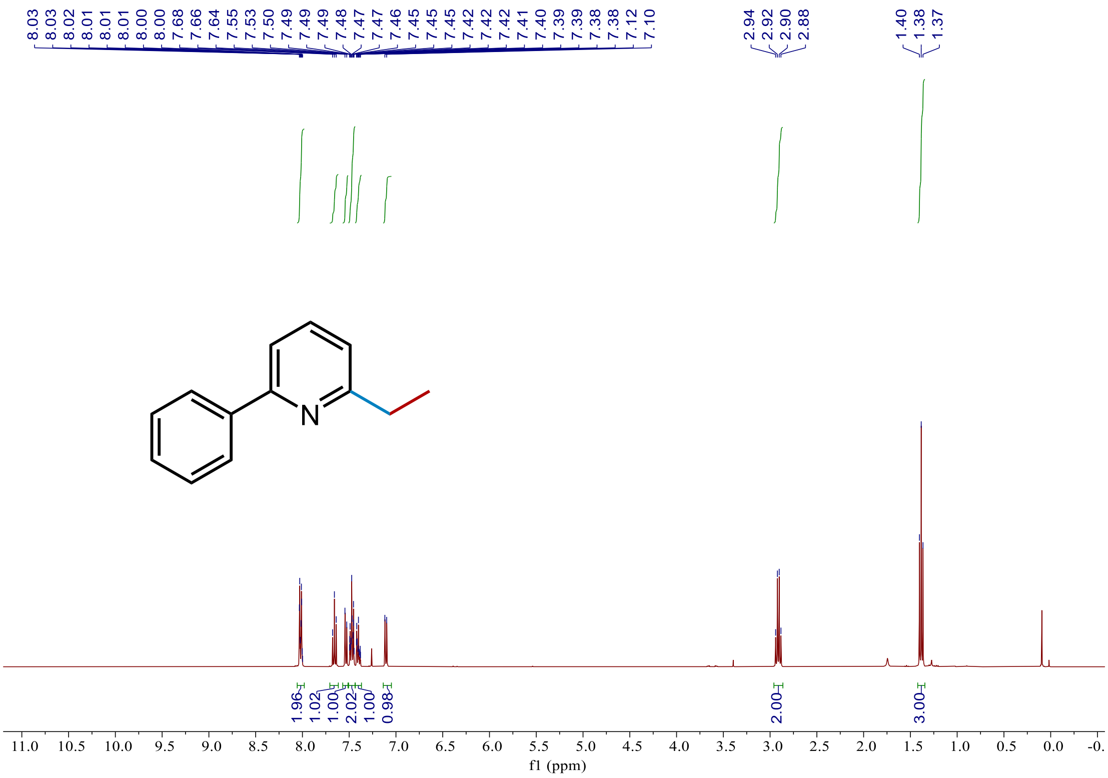
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **32**



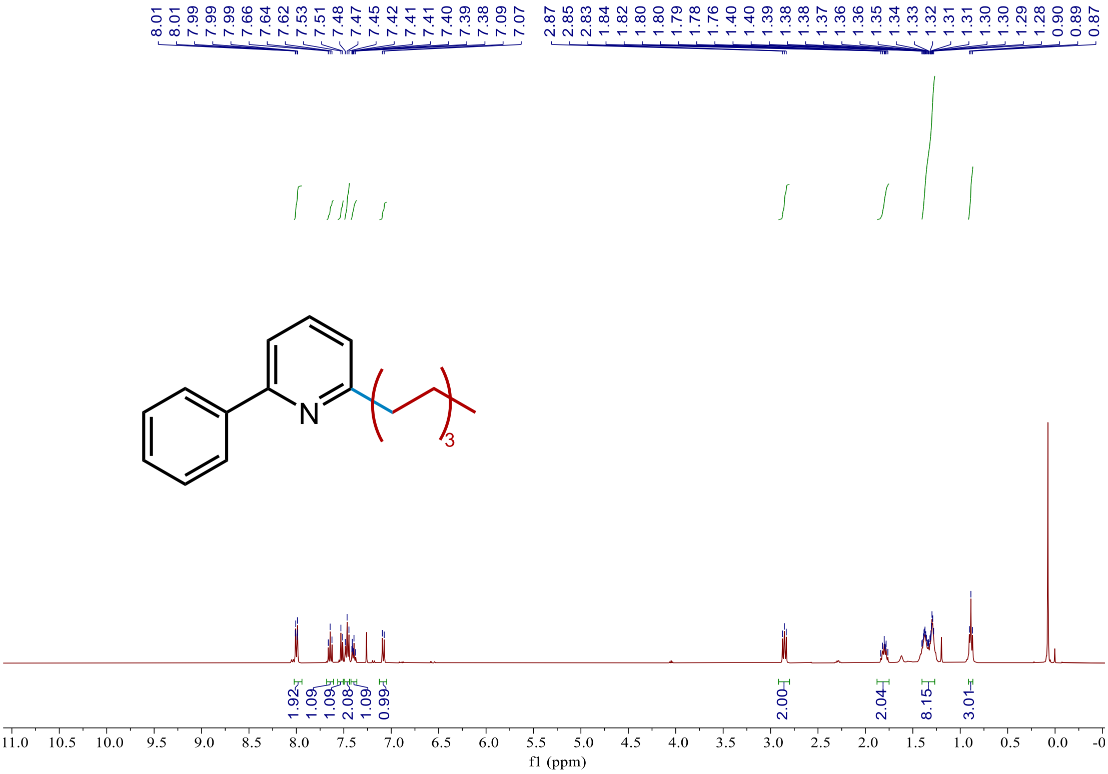
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **32**



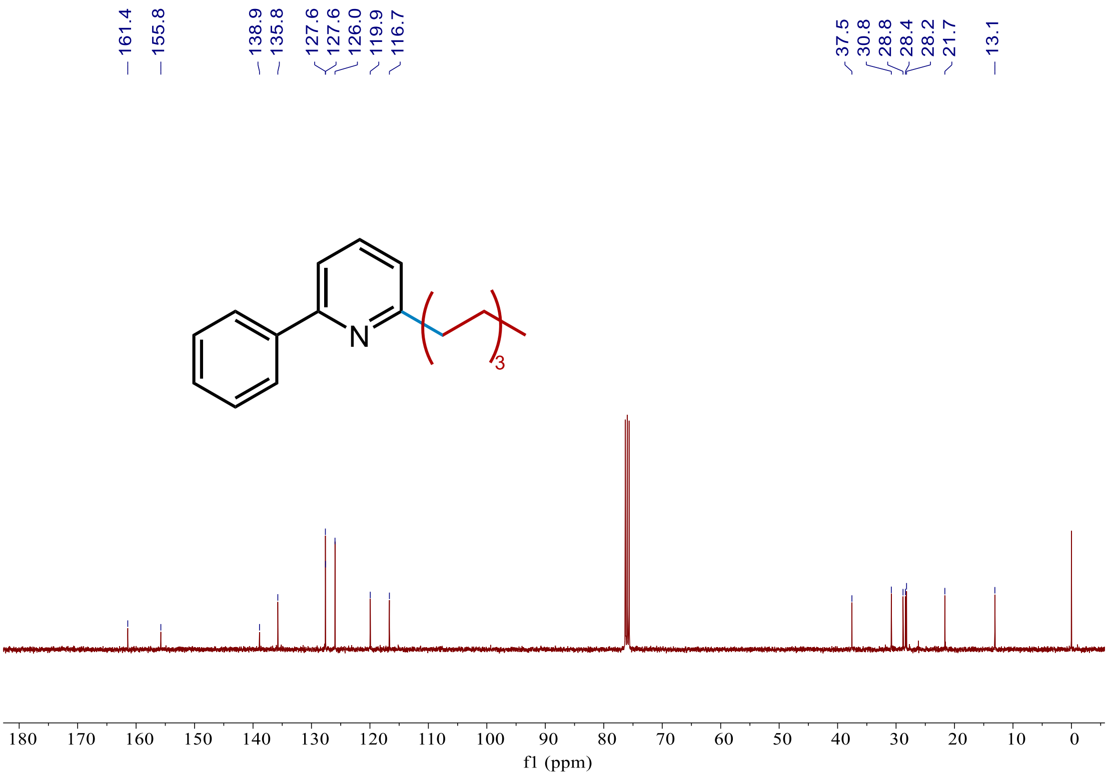
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **33**



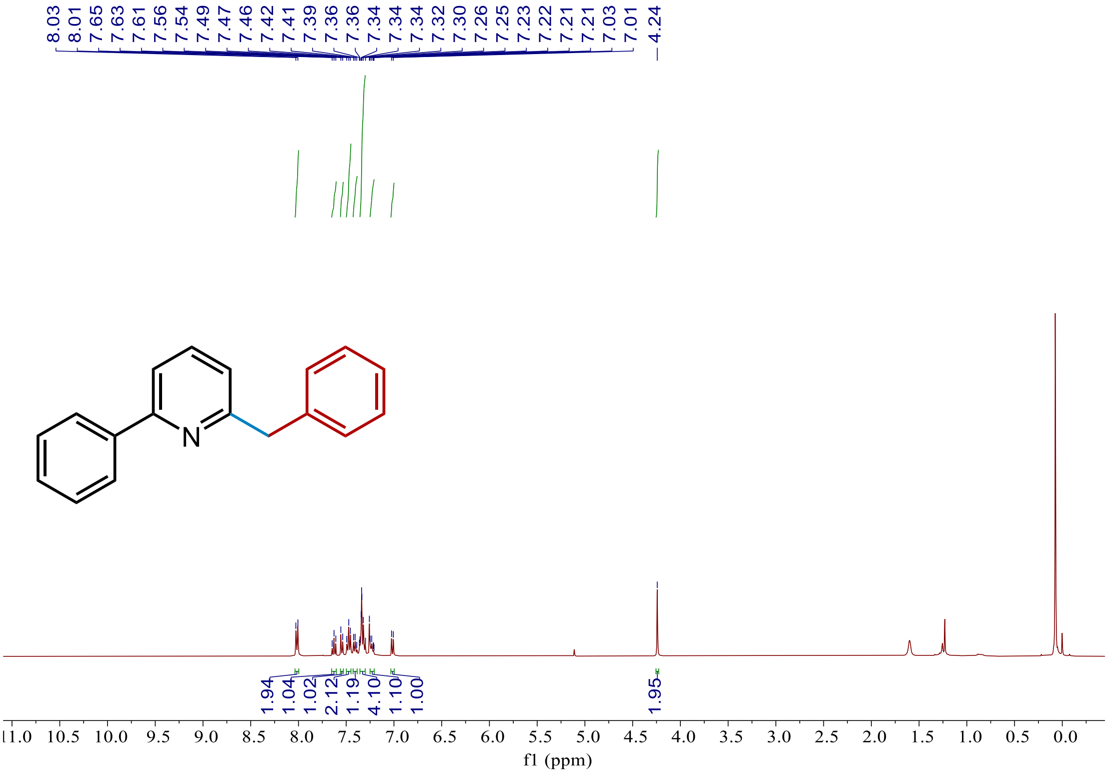
[1]H NMR (400 MHz, Chloroform-*d*) of compound **34**



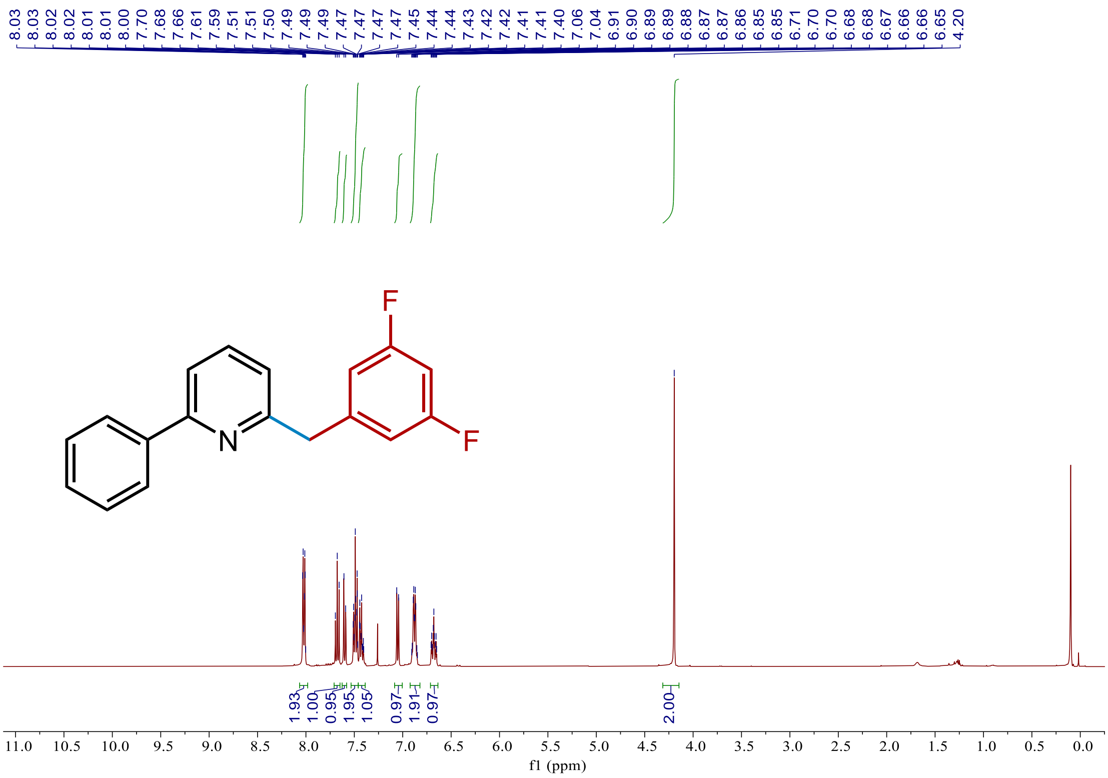
[13]C NMR (101 MHz, Chloroform-*d*) of compound **34**



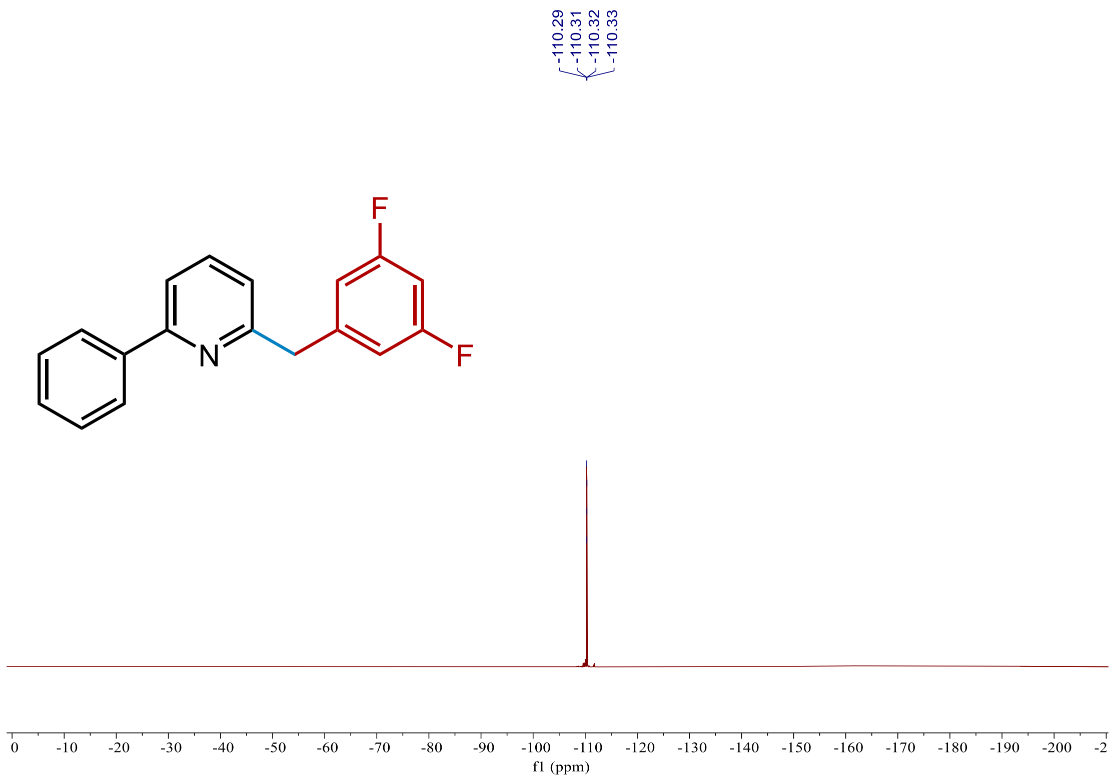
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **35**



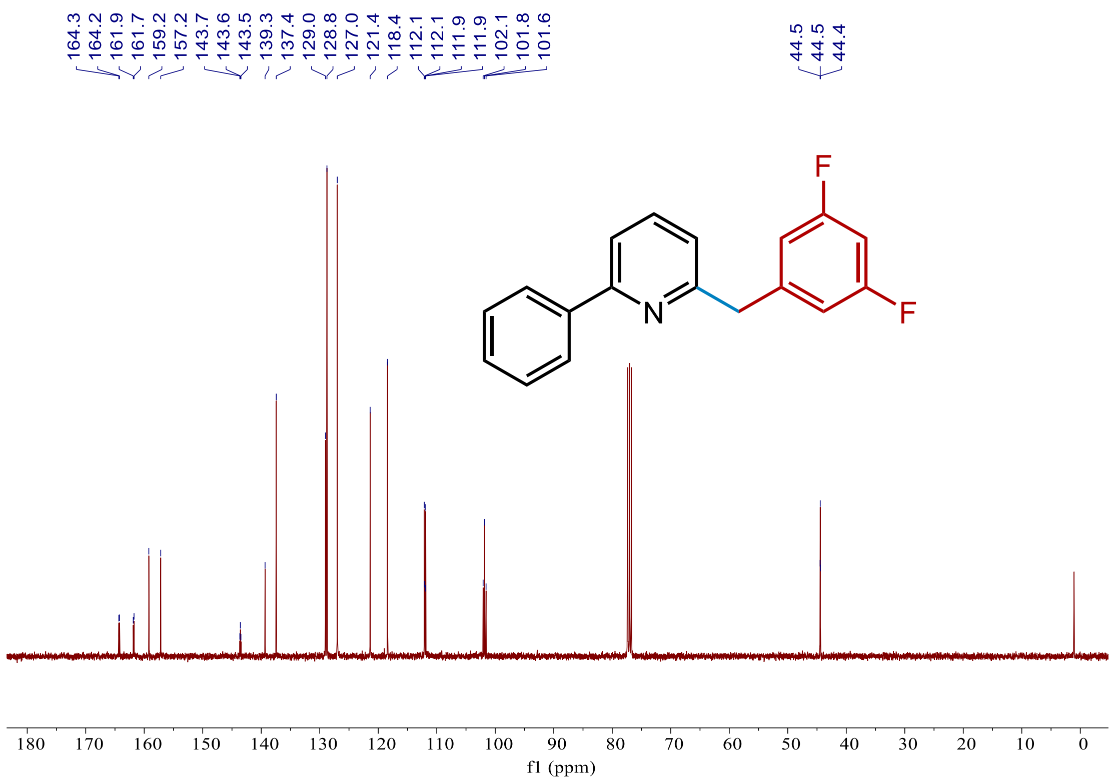
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **36**



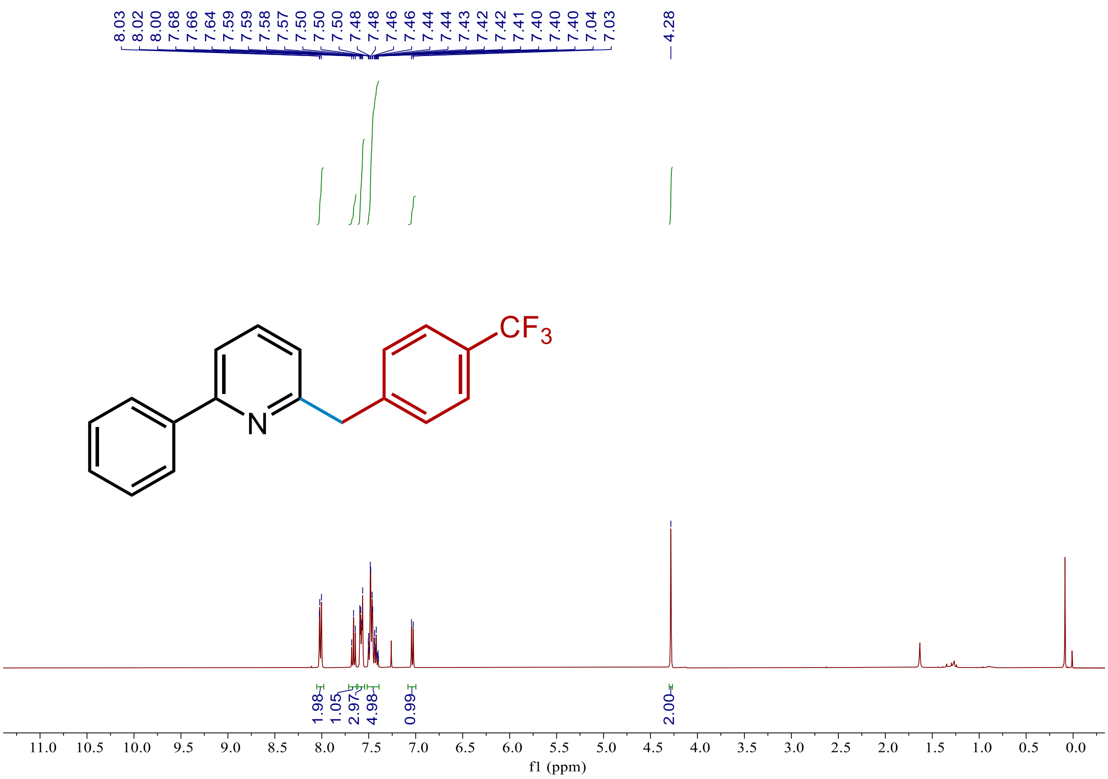
^{19}F NMR (400 MHz, Chloroform-*d*) of compound **36**



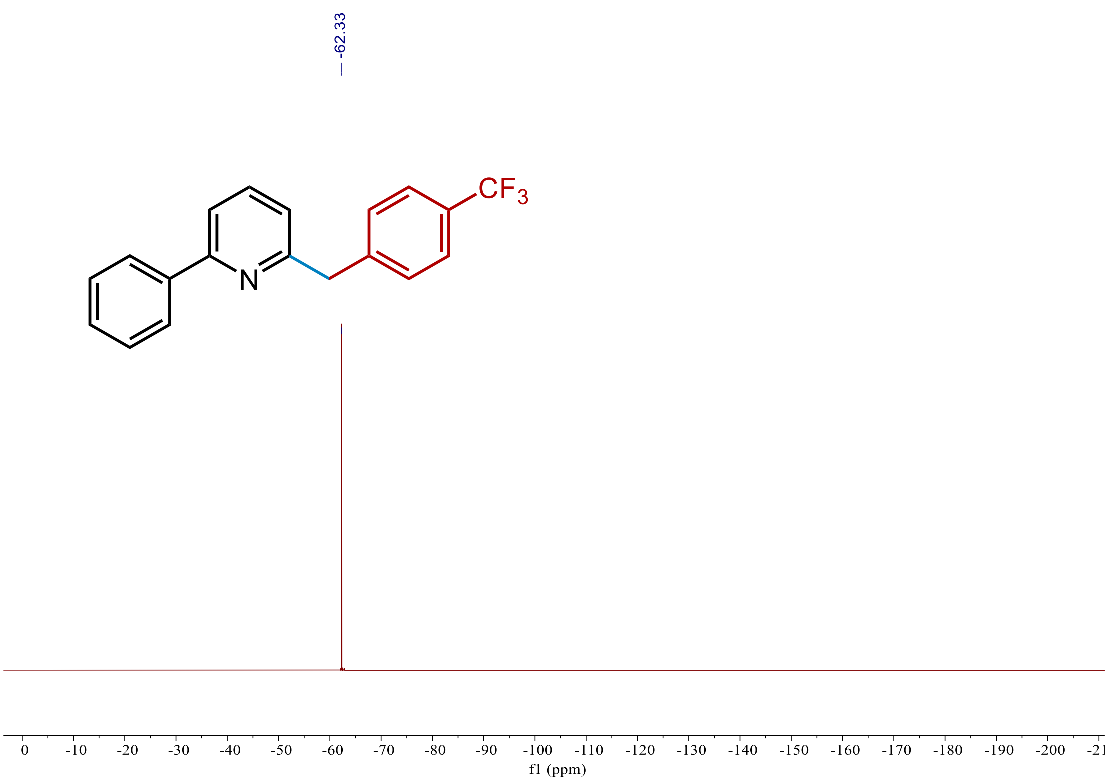
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **36**



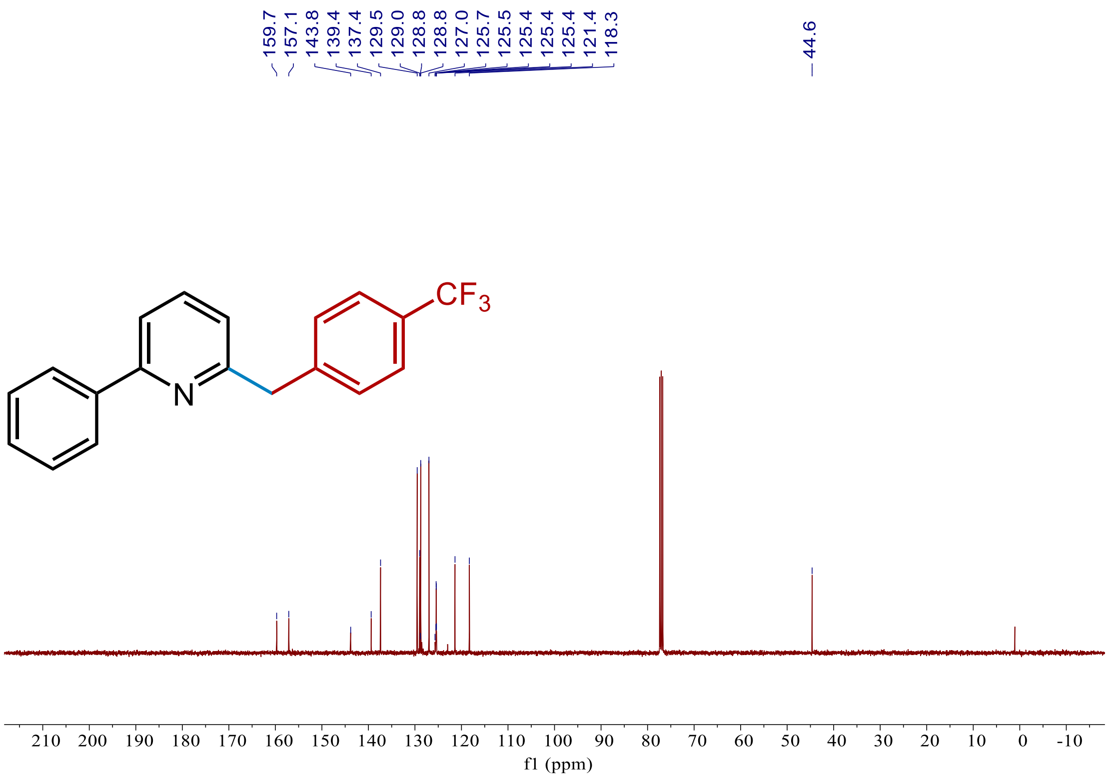
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **37**



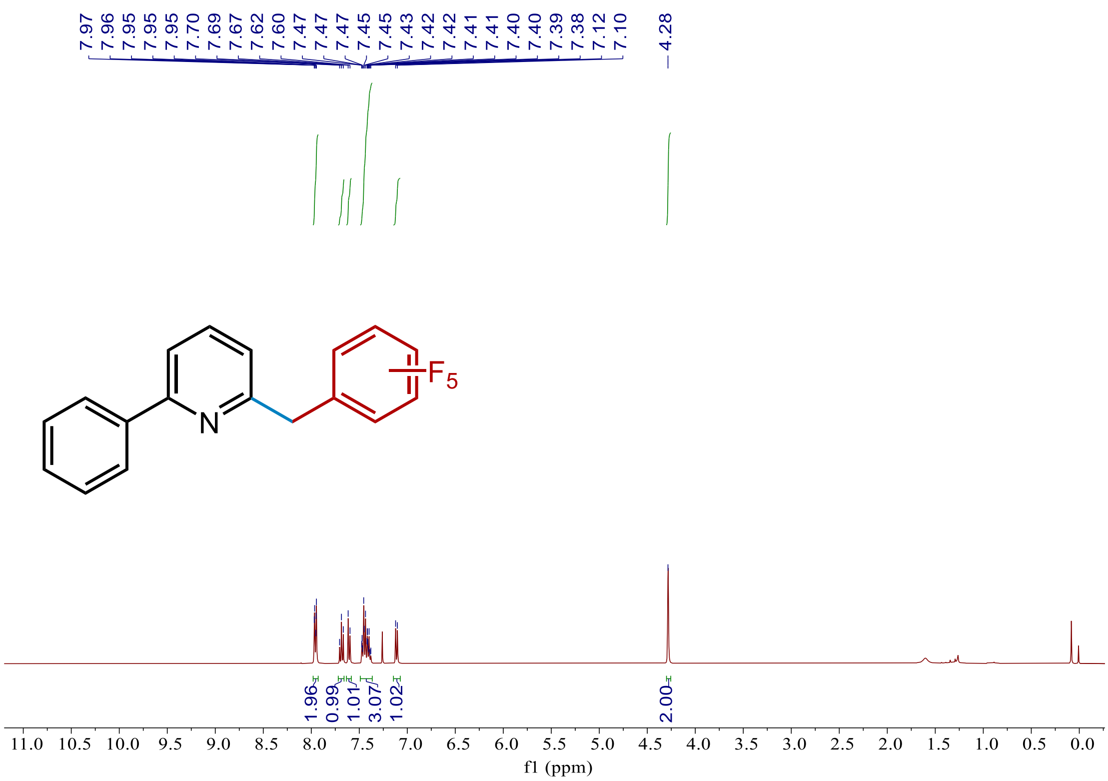
^{19}F NMR (400 MHz, Chloroform-*d*) of compound **37**



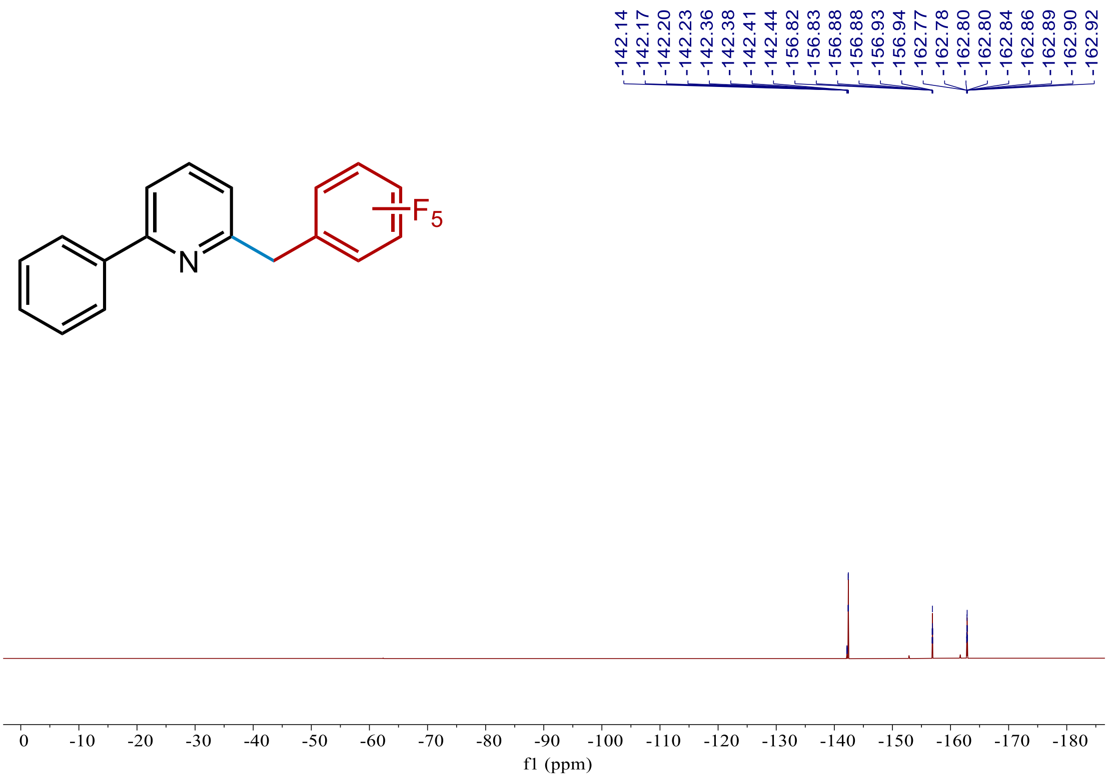
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **37**



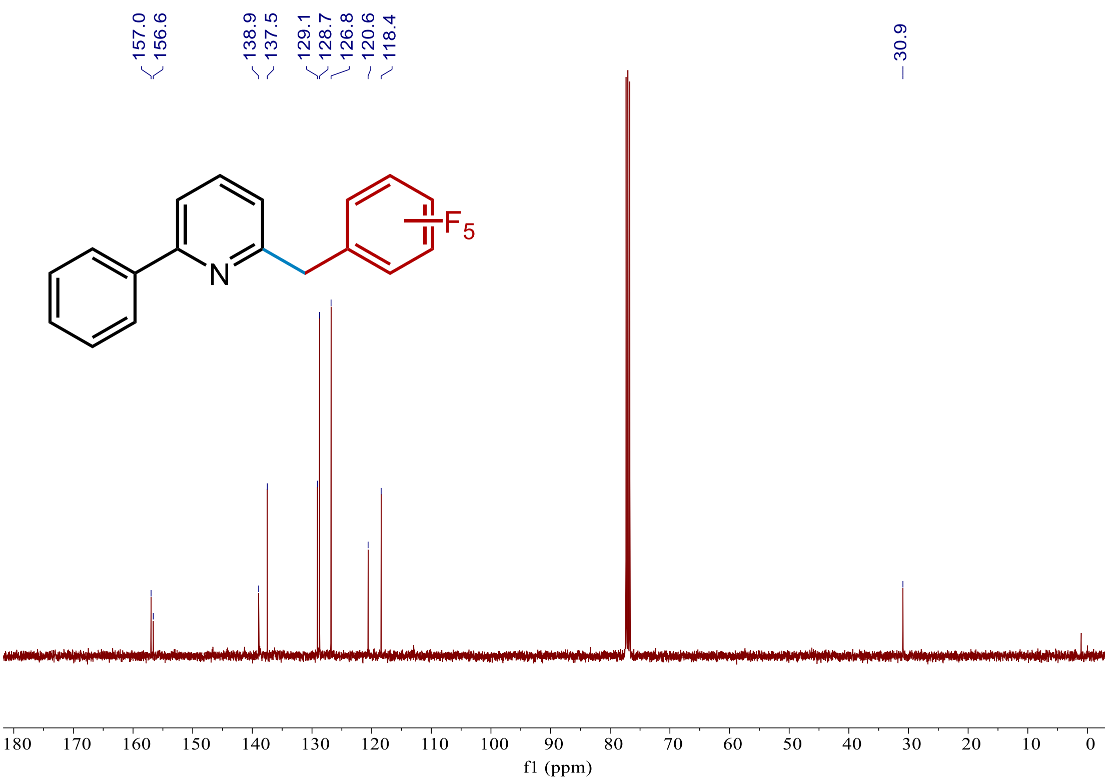
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **38**



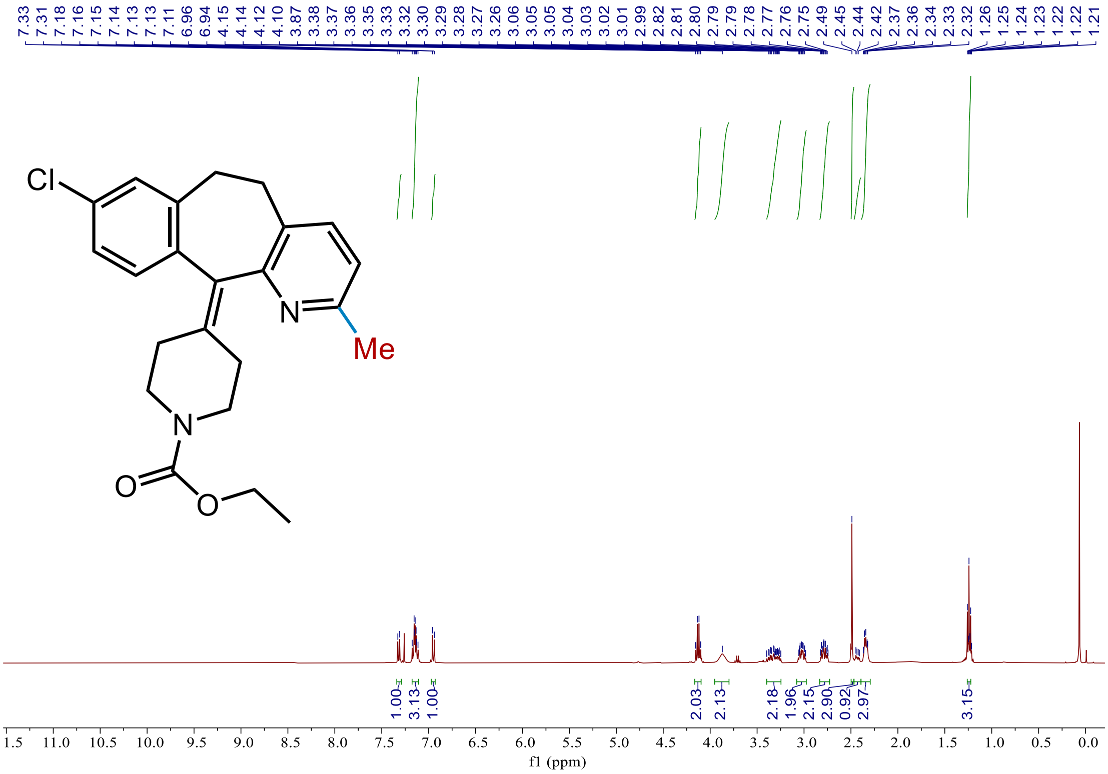
[19]F NMR (400 MHz, Chloroform-*d*) of compound **38**



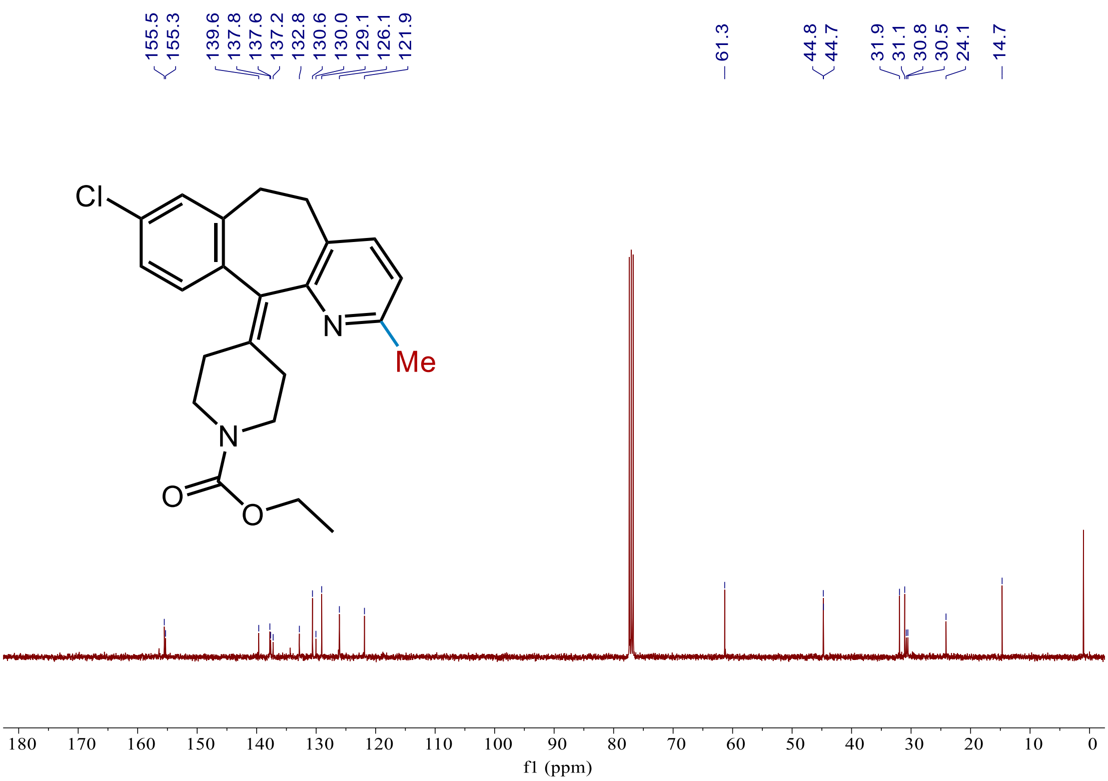
[13]C NMR (101 MHz, Chloroform-*d*) of compound **38**



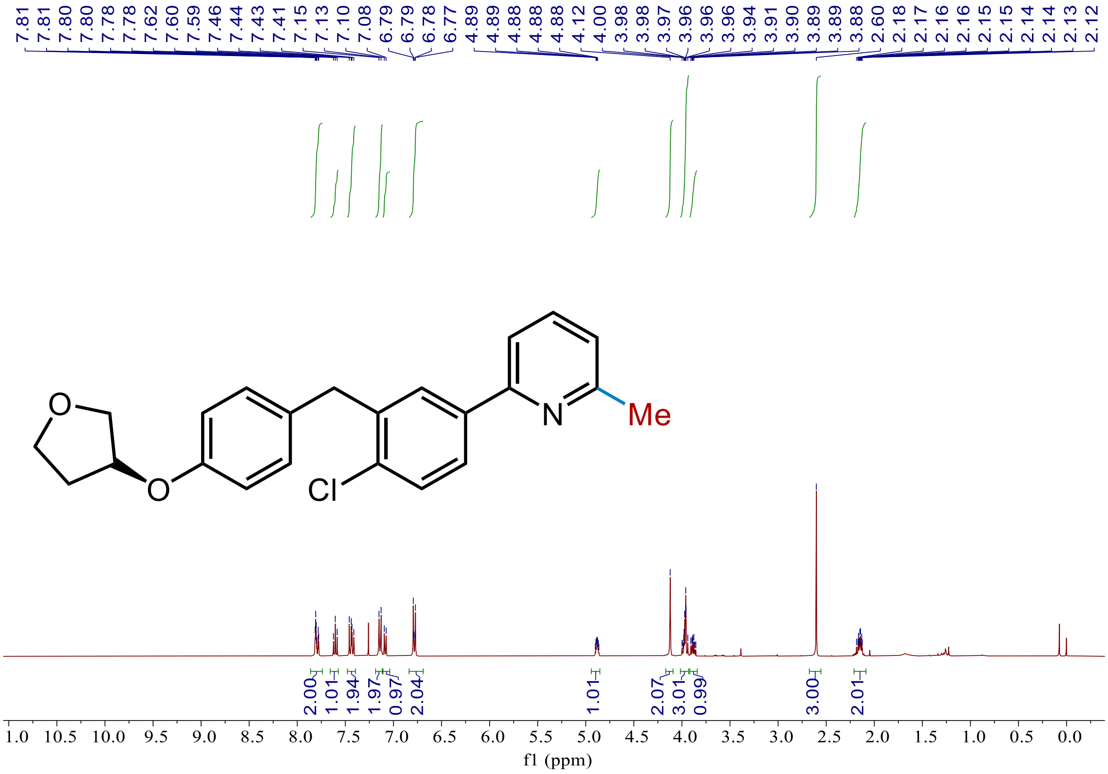
[1]H NMR (400 MHz, Chloroform-*d*) of compound **39**



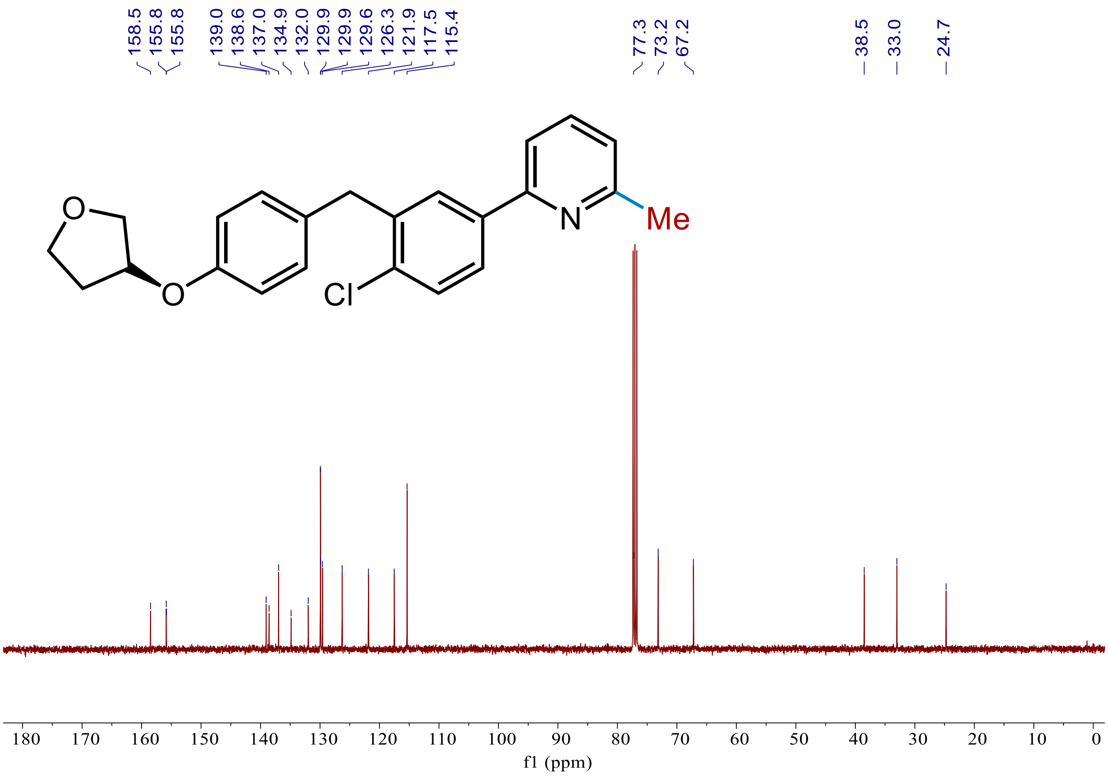
[13]C NMR (101 MHz, Chloroform-*d*) of compound **39**



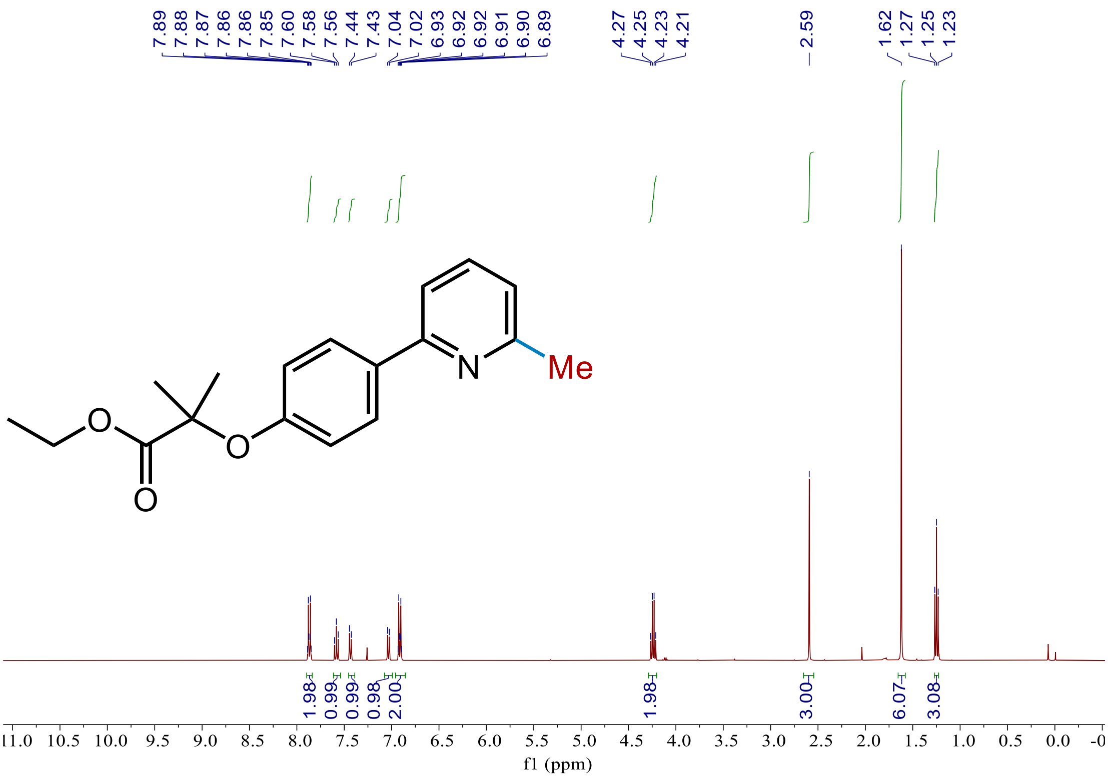
^{1}H NMR (400 MHz, Chloroform-*d*) of compound **40**



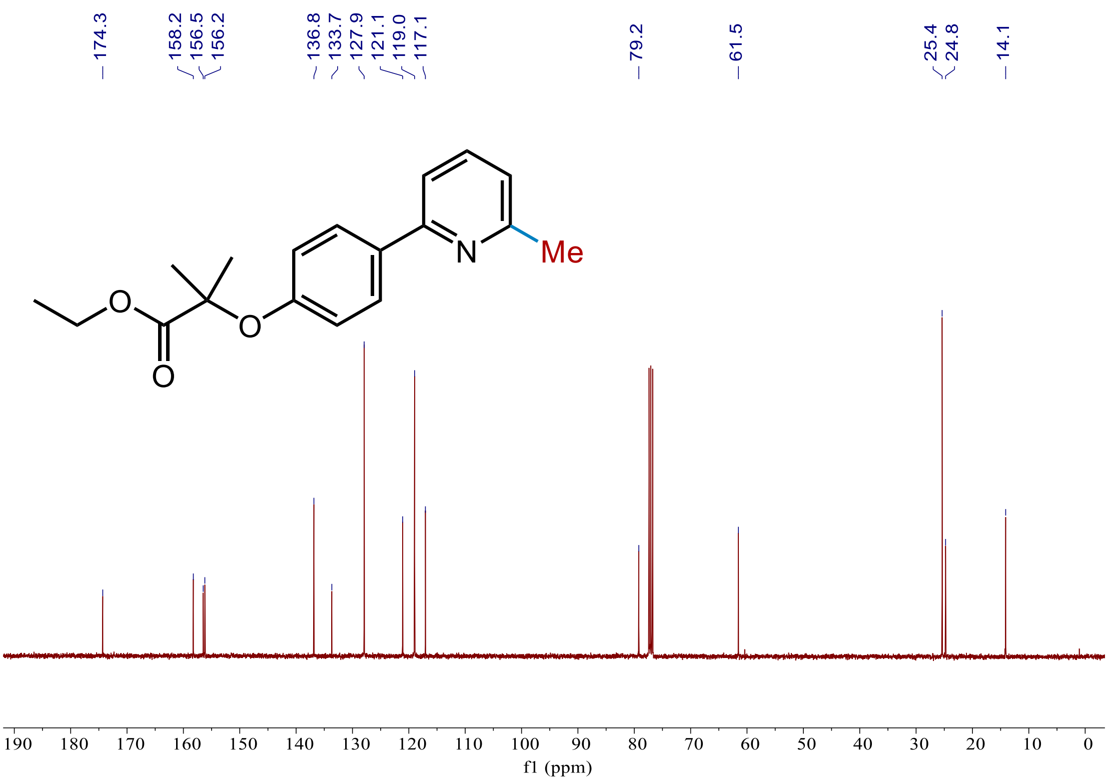
^{13}C NMR (101 MHz, Chloroform-*d*) of compound **40**



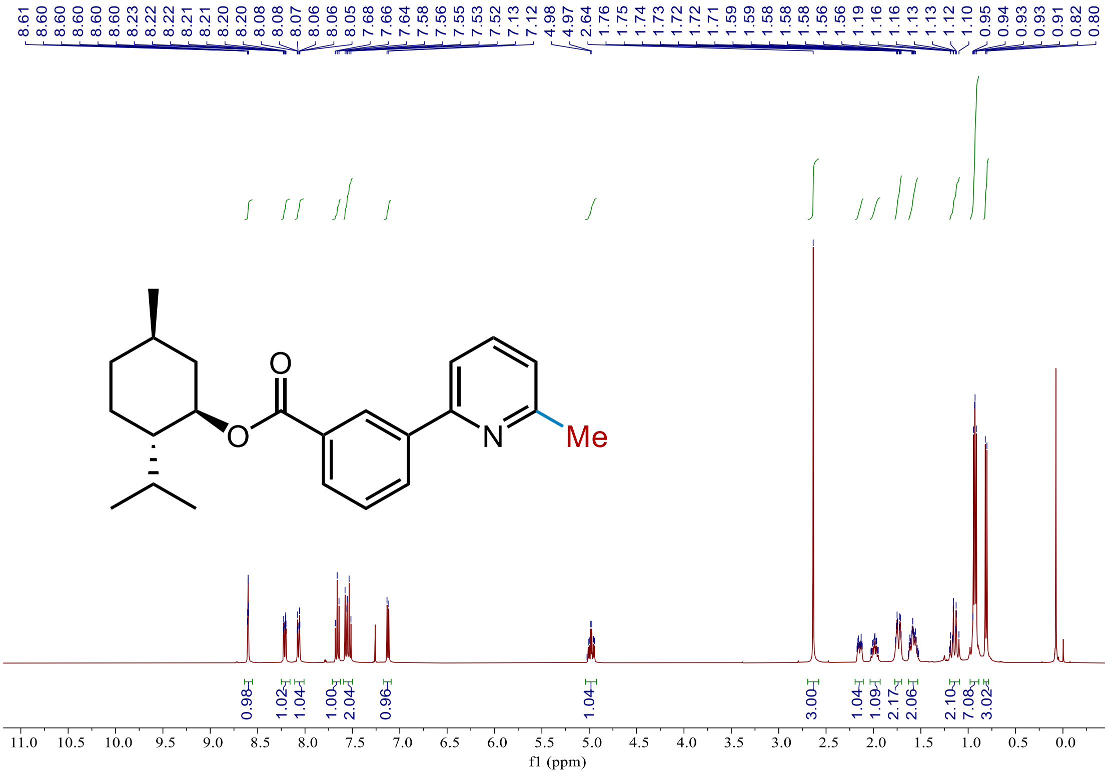
[1]H NMR (400 MHz, Chloroform-*d*) of compound **41**



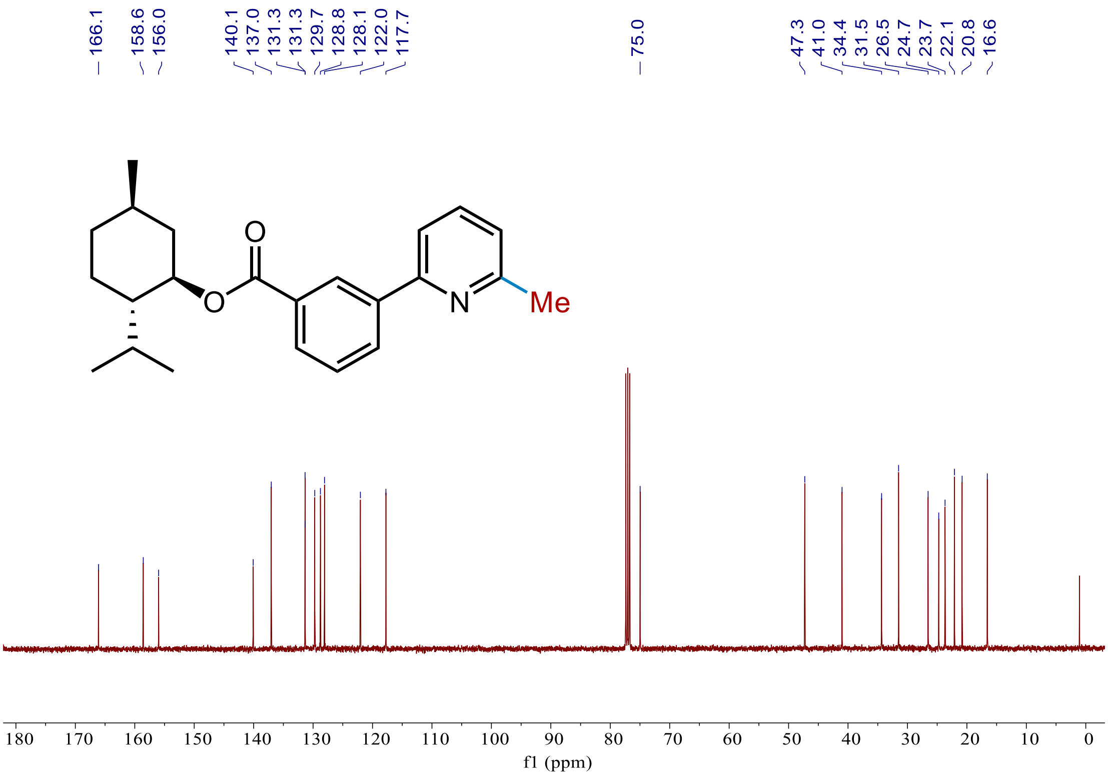
[13]C NMR (101 MHz, Chloroform-*d*) of compound **41**



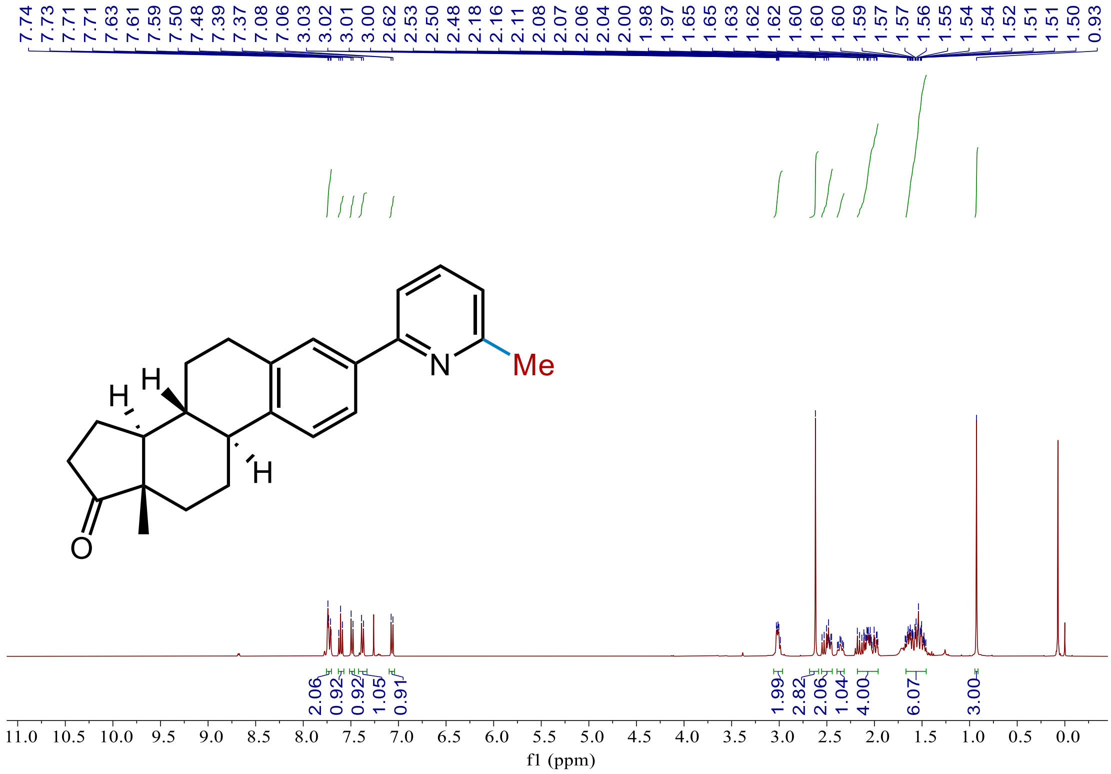
[1]H NMR (400 MHz, Chloroform-*d*) of compound **42**



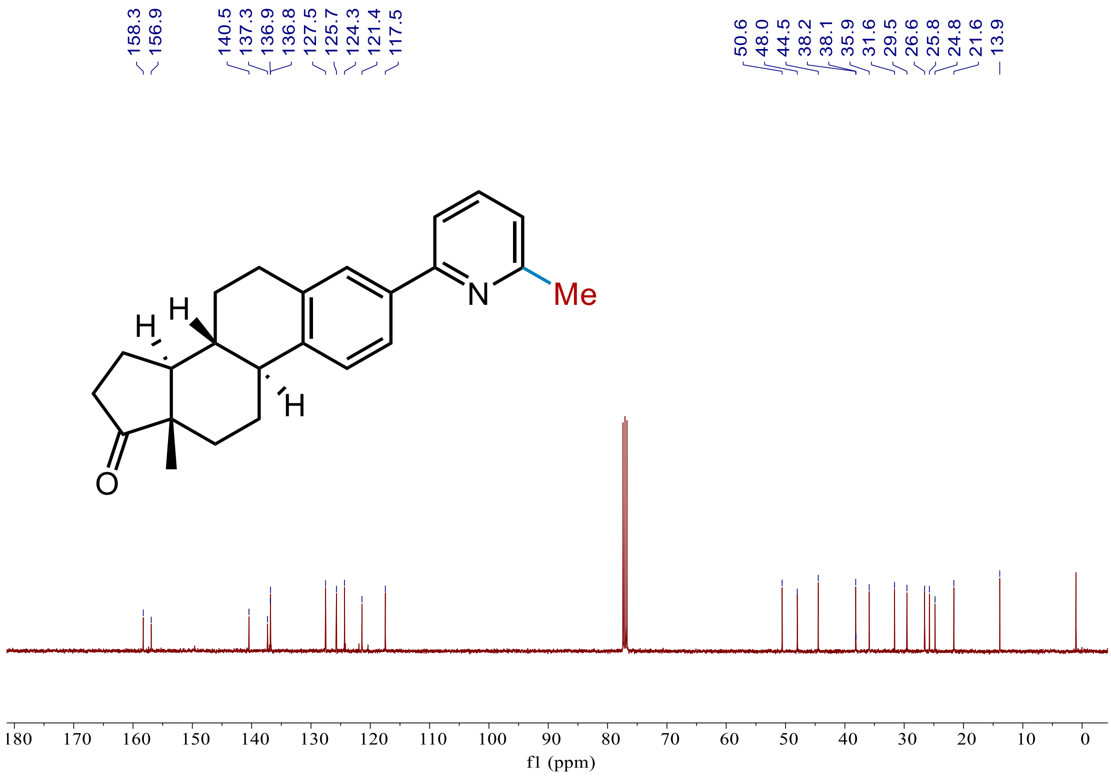
[13]C NMR (101 MHz, Chloroform-*d*) of compound **42**



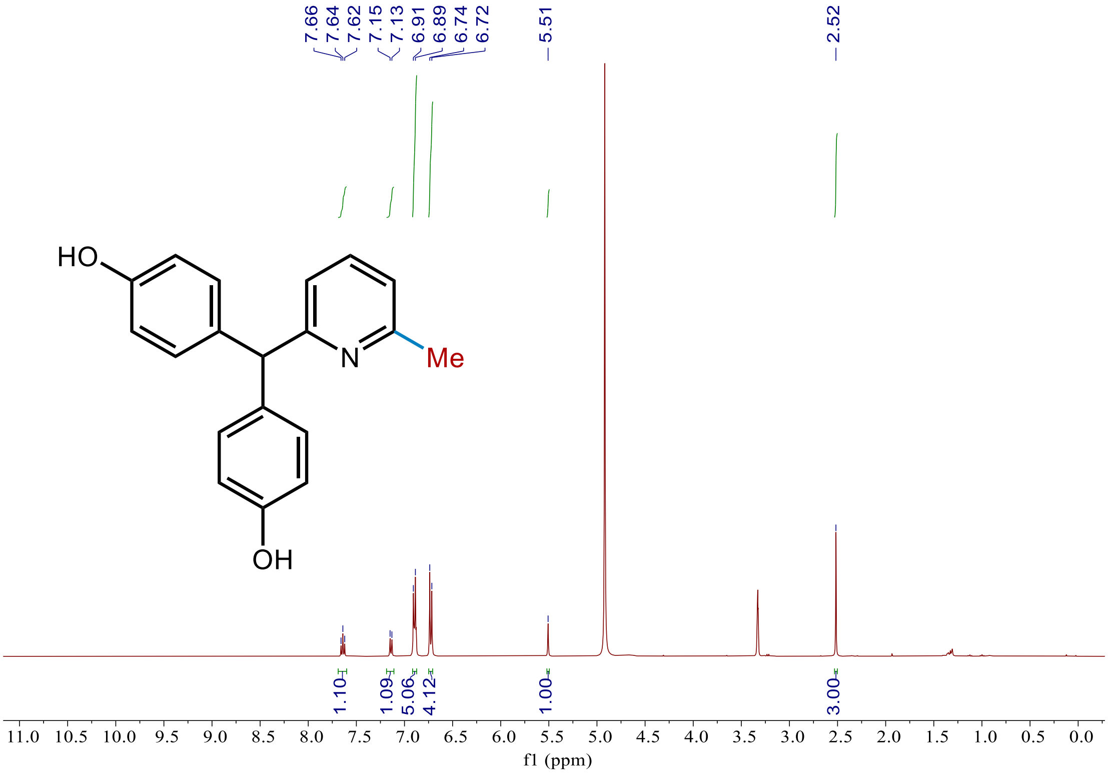
[1]H NMR (400 MHz, Chloroform-*d*) of compound **43**



[13]C NMR (101 MHz, Chloroform-*d*) of compound **43**



^{1}H NMR (400 MHz, Methanol-d_4) of compound **44**



^{13}C NMR (101 MHz, Methanol-d_4) of compound **44**