Supporting Information

Visible-Light-Driven Eosin Y Catalyzed Arylation of Alkenes with Formate Salt

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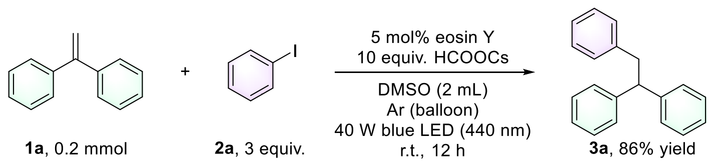
Content

1. General Information

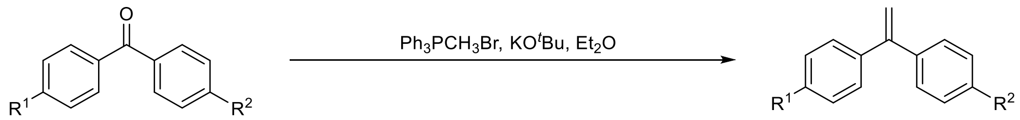
Chemicals and solvents were purchased from commercial suppliers and used as received. ^1H NMR, ^{13}C NMR spectra were recorded on a Bruker AVANCE III 500 MHz spectrometer. Chemical shifts were calibrated using residual undeuterated solvent as an internal reference ($CDCl_3$: 7.26 ppm ^1H NMR, 77.16 ppm ^{13}C NMR). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), brs (broad singlet). All gas chromatography-mass spectrometry (GC-MS) spectra were obtained on an Agilent (7890A/5975C) spectrometer. Cyclic voltammetry experiments were performed on a CH Instruments Electrochemical Analyzer. Reactions were monitored using thin layer chromatography (TLC) on glass backed plates and visualised by UV radiation at a wavelength of 254 nm.

Fig. S1 Blue LED photoreactor

2. General procedures



General Procedure for the Alkene Arylation Products. An oven-dried Schlenk tube (10 mL) equipped with a magnetic stir bar was charged with HCOOCs (cesium formate, 2 mmol, 355.8 mg, 10 equiv.) and eosin Y (0.01 mmol, 6.8 mg, 5 mol%). The Schlenk tube was sealed and degassed *via* vacuum evacuation and subsequent backfilled with argon for three times. Subsequently, the **1a** (0.2 mmol, 35 μL), **2a** (0.6 mmol, 63 μL, 3 equiv.) and anhydrous DMSO (2 mL) were added through injection port under argon atmosphere. Then the reaction was placed under a blue LED (wavelength 440 nm, 40 W) with an argon balloon and irradiated at room temperature for 12 h. Upon completion of the reaction, the yield was determined by GC-MS employing 1,3,5-trimethoxybenzene as an internal standard for calibration. The crude product was purified by column chromatography isolation on silica gel (eluent: PE/EA = 500/1 to 200/1 v/v) to give the pure desired product as white solid (**3a**, 44.3 mg, 86% yield).



General Procedure for the Alkene [1] [2]. A round flask equipped with a magnetic stirring bar was charged with Ph_3PCH_3Br (12 mmol, 1.2 equiv.), and evacuated, backfilled with argon three times, followed by addition of anhydrous Et_2O (50 mL, 0.2 M). ^{t}BuOK (1.2 equiv) was added to the vigorously stirring solution at room temperature. The resulting solution was cooled to 0 °C when a bright yellow heterogeneous mixture was achieved. Then aryl ketone (10 mmol, 1.0 equiv.) was slowly added to the mixture. Upon complete addition, the reaction was allowed to stir at room temperature overnight. Then, the solution was quenched by brine and diluted with ethyl acetate. The organic phase was separated and the aqueous layer was extracted with EA. The combined organic layer was dried over sodium sulfate, and concentrated under vacuum. The product **1** was purified by column chromatography on silica gel using PE/EA (100/1 v/v) as the eluent.

3. Investigation of the key reaction parameters

Table S1 Screening of photocatalysts

1a, 0.2 mmol　**2a**, 3 equiv.　**3a**, 86% yield　**3a'**, 3% yield

Entry	Reaction condition^a	**3a** yield (%) ^{b}	**3a'** yield (%) ^{b}
1	"standard reaction condition"	92 (86 ^{c})	3
2	5 mol% rose bengal instead of eosin Y	64	3
3	5 mol% eosin B instead of eosin Y	13	2
4	5 mol% riboflavin instead of eosin Y	59	3
5	10 mol% eosin Y instead of 5 mol%	47	7
6	without riboflavin	N.D.	N.D.

^{a} standard reaction condition: 0.2 mmol **1a**, 5 mol% eosin Y, 3 equiv. **2a**,10 equiv. HCOOCs, DMSO (2 mL), Ar (balloon), 40 W blue LED (440 nm), r.t., 12 h. ^{b} Yield determined by GC-MS analysis using 1,3,5-trimethoxybenzene as an internal standard. ^{c} Isolated yield.

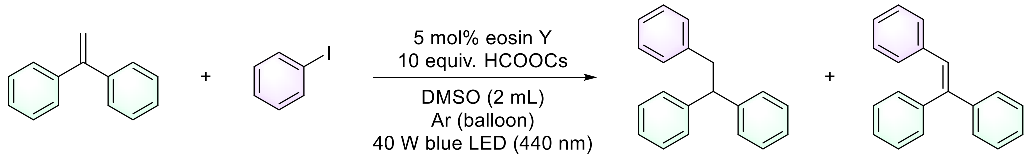
Table S2 Screening of solvents

1a, 0.2 mmol **2a**, 3 equiv. **3a**, 86% yield **3a'**, 3% yield

Entry	Reaction condition[a]	**3a** yield (%) [b]	**3a'** yield (%) [b]
1	"standard reaction condition"	92 (86 [c])	3
2	DMF instead of DMSO	4	2
3	MeOH instead of DMSO	2	1
4	1,4-dioxane instead of DMSO	N.D.	N.D.
5	H_2O instead of DMSO	1	1
6	3 mL instead of 2 mL DMSO	86	2

[a] standard reaction condition: 0.2 mmol **1a**, 5 mol% eosin Y, 3 equiv. **2a**,10 equiv. HCOOCs, solvent (2 mL), Ar (balloon), 40 W blue LED (440 nm), r.t., 12 h. [b] Yield determined by GC-MS analysis using 1,3,5-trimethoxybenzene as an internal standard. [c] Isolated yield.

Table S3 Screening of formate salt

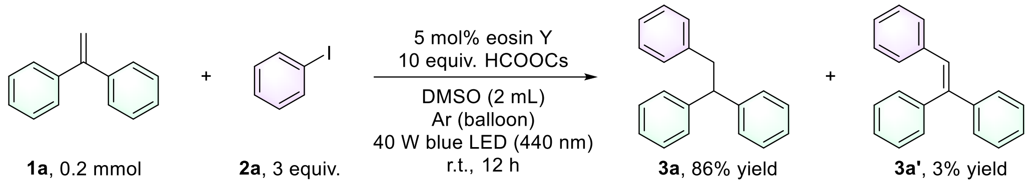


1a, 0.2 mmol **2a**, 3 equiv. **3a**, 86% yield **3a'**, 3% yield

Entry	Reaction condition[a]	**3a** yield (%) [b]	**3a'** yield (%) [b]
1	"standard reaction condition"	92 (86 [c])	3
2	10 equiv. HCOONa instead of HCOOCs	5	6
3	10 equiv. HCOOK instead of HCOOCs	17	9
4	8 equiv. HCOOCs instead of 10 equiv	81	5
5	10 equiv. HCOOH instead of HCOOCs	N.D.	N.D.
6	10 equiv. Cs_2CO_3 instead of HCOOCs	N.D.	N.D.
7	without HCOOCs	N.D.	N.D.

[a] standard reaction condition: 0.2 mmol **1a**, 5 mol% eosin Y, 3 equiv. **2a**,10 equiv. formate salt, DMSO (2 mL), Ar (balloon), 40 W blue LED (440 nm), r.t., 12 h. [b] Yield determined by GC-MS analysis using 1,3,5-trimethoxybenzene as an internal standard. [c] Isolated yield.

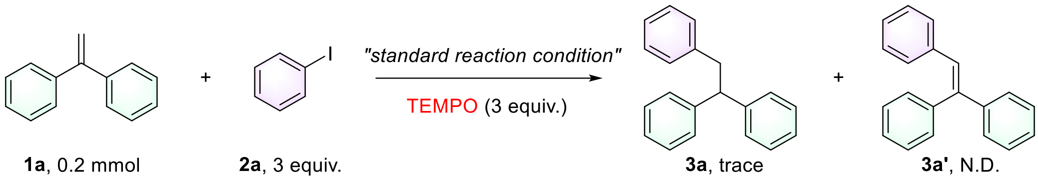
Table S4 Screening of concentration



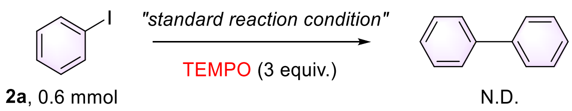
Entry	Reaction condition[a]	**3a** yield (%) [b]	**3a'** yield (%) [b]
1	"standard reaction condition"	92 (86 [c])	3
2	1.5 equiv. iodobenzene instead of 3 equiv	55	trace
3	5 equiv. iodobenzene instead of 3 equiv	34	5
4	0.1 mmol **1a** instead of 0.2 mmol	8	trace
5	0.3 mmol **1a** instead of 0.2 mmol	28	8

[a] standard reaction condition: 0.2 mmol **1a**, 5 mol% eosin Y, 3 equiv. **2a**,10 equiv. HCOOCs, DMSO (2 mL), Ar (balloon), 40 W blue LED (440 nm), r.t., 12 h. [b] Yield determined by GC-MS analysis using 1,3,5-trimethoxybenzene as an internal standard. [c] Isolated yield.

4. Radical trapping experiment



An oven-dried Schlenk tube (10 mL) equipped with a magnetic stir bar was charged with HCOOCs (cesium formate, 2 mmol, 355.8 mg, 10 equiv.), TEMPO (0.6 mmol, 93.8 mg, 3 equiv.) and eosin Y (0.01 mmol, 6.8 mg, 5 mol%). The Schlenk tube was sealed and degassed *via* vacuum evacuation and subsequent backfilled with argon for three times. Subsequently, the **1a** (0.2 mmol, 35 μL), **2a** (0.6 mmol, 63 μL, 3 equiv.) and anhydrous DMSO (2 mL) were added through injection port under argon atmosphere. Then the reaction was placed under a blue LED (wavelength 440 nm, 40 W) with an argon balloon and irradiated at room temperature for 12 h. Upon completion of the reaction, the yield was determined by GC-MS employing 1,3,5-trimethoxybenzene as an internal standard for calibration.

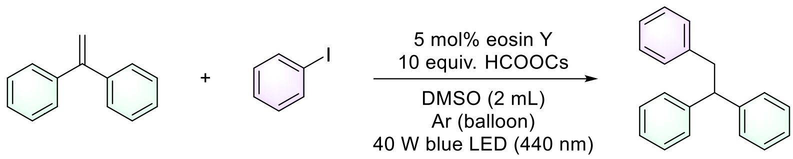


An oven-dried Schlenk tube (10 mL) equipped with a magnetic stir bar was charged with HCOOCs (cesium formate, 2 mmol, 355.8 mg, 10 equiv.), TEMPO (0.6 mmol, 93.8 mg, 3 equiv.) and eosin Y (0.01 mmol,6.8 mg, 5 mol%). The Schlenk tube was sealed and degassed *via* vacuum evacuation and subsequent backfilled with argon for three times. Subsequently, the **2a** (0.6 mmol, 63 μL) and anhydrous DMSO (2 mL) were added through injection port under argon atmosphere. Then the reaction was placed under a blue LED (wavelength 440 nm, 40 W) with an argon balloon and irradiated at room temperature for 12 h. Upon completion of the reaction, the yield was determined by GC-MS employing 1,3,5-trimethoxybenzene as an internal standard for calibration.

5. Determination of photochemical quantum yields by standard ferrioxalate actinometry

The photon flux at 440 nm was calculated to be 4.67 × 10^{-9} einstein s^{-1} [3].

Determination of quantum yield:



1a, 0.2 mmol　**2a**, 3 equiv.　**3a**, 27% yield

An oven-dried Schlenk tube (10 mL) equipped with a magnetic stir bar was charged with HCOOCs (cesium formate, 2 mmol, 355.8 mg, 10 equiv.) and eosin Y (0.01 mmol, 6.8 mg, 5 mol%). The Schlenk tube was sealed and degassed *via* vacuum evacuation and subsequent backfilled with argon for three times. Subsequently, the **1a** (0.2 mmol, 35 μL), **2a** (0.6 mmol, 63 μL, 3 equiv.) and anhydrous DMSO (2 mL) were added through injection port under argon atmosphere. Then the reaction was placed under a blue LED (wavelength 440 nm, 40 W) with an argon balloon and irradiated at room temperature for 7200 s (2 h). Upon completion of the reaction, the yield of product formed was determined as 27% yield (0.054 mmol) by GC-MS employing 1,3,5-trimethoxybenzene as an internal standard for calibration. The quantum yield was determined using eq (1). Essentially all incident light (f > 0.999, vide infra) is absorbed by the Eosin Y at the reaction conditions described above.

$$\varphi = \frac{\text{mol product}}{\text{flux} \cdot \text{t} \cdot \text{f}} \quad (1)$$

$$\varphi = \frac{5.4 \times 10^{-5}\ \text{mol}}{4.67 \times 10^{-9}\ \text{einstein s}^{-1} \times 7200\ \text{s} \times 1.00} = 1.6$$

6. UV-Vis study

Table S5 Detailed information for preparing UV-Vis samples

Entry	HCOOCs	Eosin Y	DMSO	Irradiation time
Sample-01	--	1 mg	3 mL	--
Sample-02	--	1 mg	3 mL	5 min
Sample-03	10 mg	1 mg	3 mL	--
Sample-04	10 mg	1 mg	3 mL	5 min

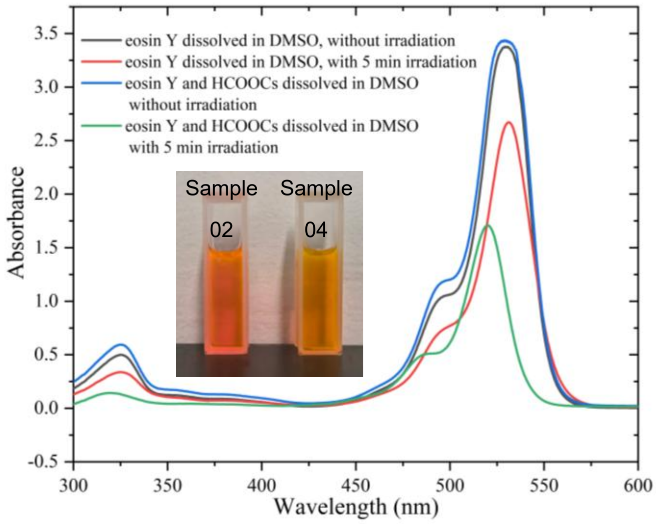


Fig. S2 UV-Vis absorption spectrum of a mixture of HCOOCs and Eosin Y.

7. Cyclic voltammetry studies

Sample 5 mM and tetrabutylammonium tetrafluoroborate 0.1 M in DMSO were used for tests. Measurements were run using glassy carbon working electrode, platinum wire counter electrode, and Hg-$HgCl_2$ reference electrode in a scan rate of 0.1 V/s.

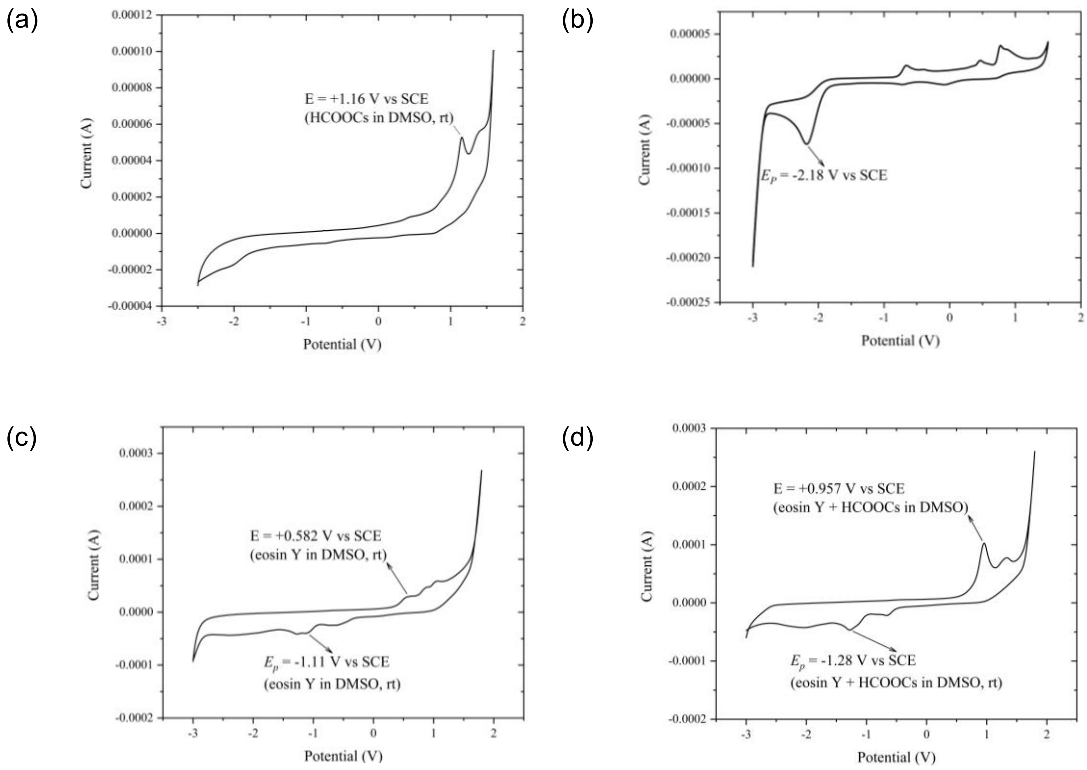


Fig. S3 (a) Cyclic voltammogram of HCOOCs in DMSO (room temperature). Scanned from -2.5 V to +1.5 V to -2.5 V at 0.1 V/s. (b) Cyclic voltammogram of PhI in DMSO at room temperature. Scanned from -3 V to +1.5 V to -3 V at 0.1 V/s. (c) Cyclic voltammogram of eosin

Y in DMSO at room temperature. Scanned from -3 V to +1.8 V to -3 V at 0.1 V/s. (d) Cyclic voltammogram of eosin Y + HCOOCs in DMSO at room temperature. Scanned from -3 V to +1.8 V to -3 V at 0.1 V/s. (The supporting electrolyte was a 0.1 M solution of tetrabutylammonium tetrafluoroborate in DMSO)

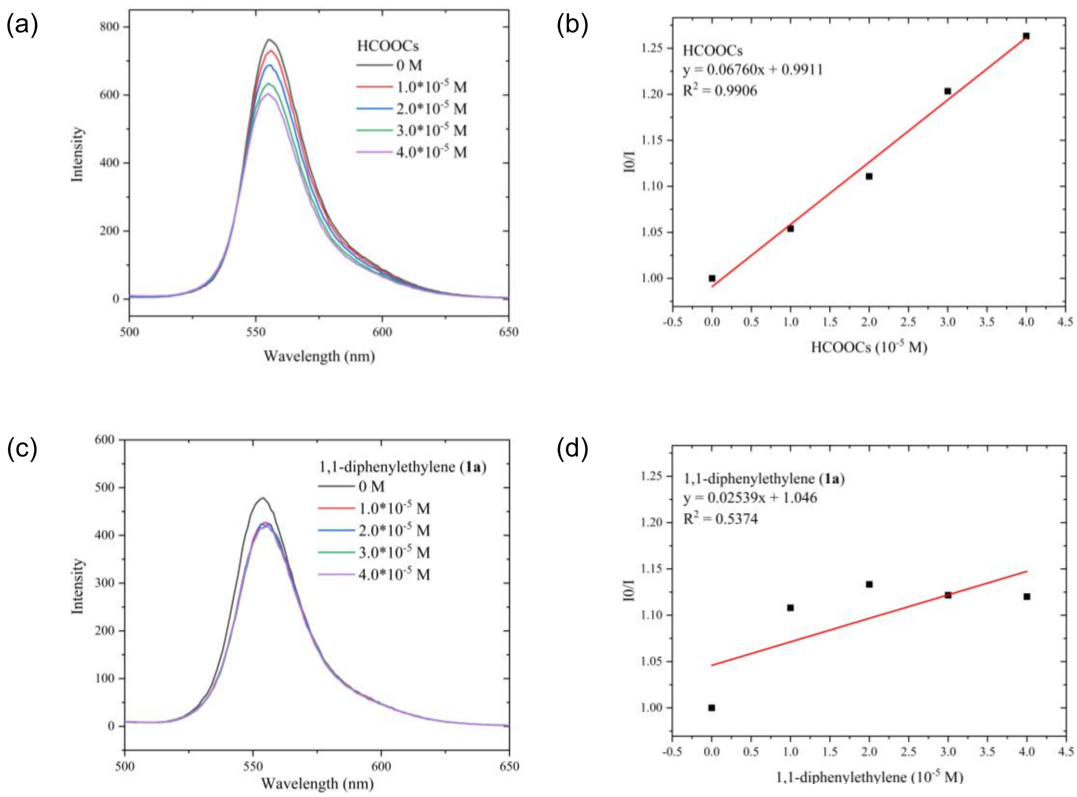
8. Stern-Volmer emission quenching

From Stern-Volmer quenching studies, we found that luminescence of Eosin Y was efficiently quenched by HCOOCs but not the PhI and olefin (1,1-diphenylethylene).

HCOOCs: A stock solution of HCOOCs (10^{-3} M) was prepared. Then, 20 μL of this stock solution were added to 2.0 mL of Eosin Y in DMSO (10^{-5} M).

1,1-diphenylethylene (**1a**): A stock solution of PhI (10^{-3} M) was prepared. Then, 20 μL of this stock solution were added to 2.0 mL of Eosin Y in DMSO (10^{-5} M).

PhI (**2a**): A stock solution of PhI (10^{-3} M) was prepared. Then, 20 μL of this stock solution were added to 2.0 mL of Eosin Y in DMSO (10^{-5} M).



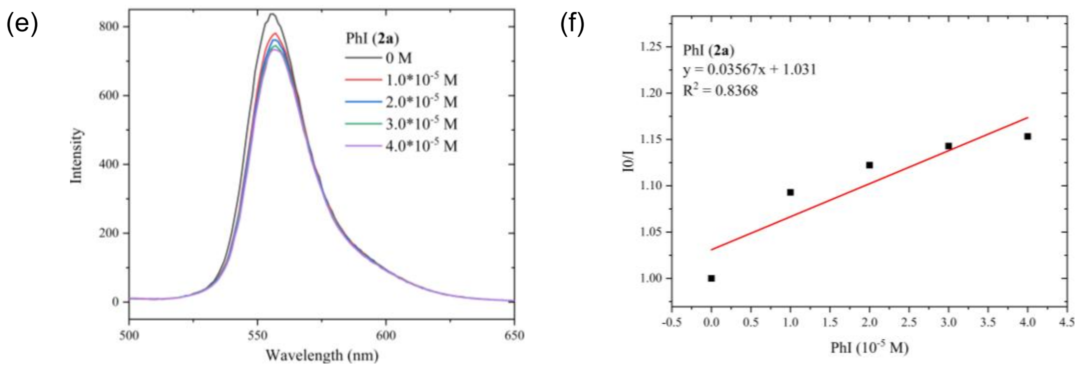


Fig. S4 Fluorescence quenching experiments

9. The proposed mechanism of the oxygen tolerance

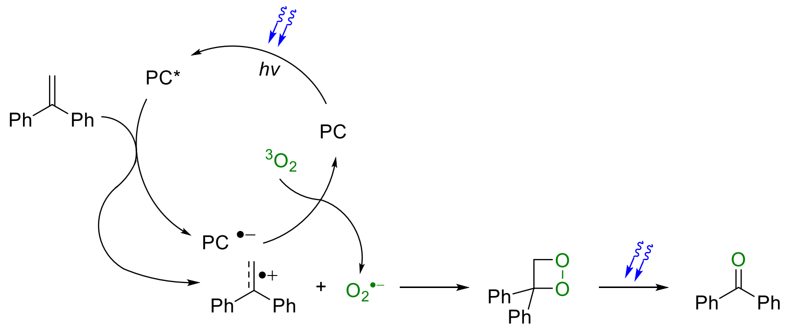
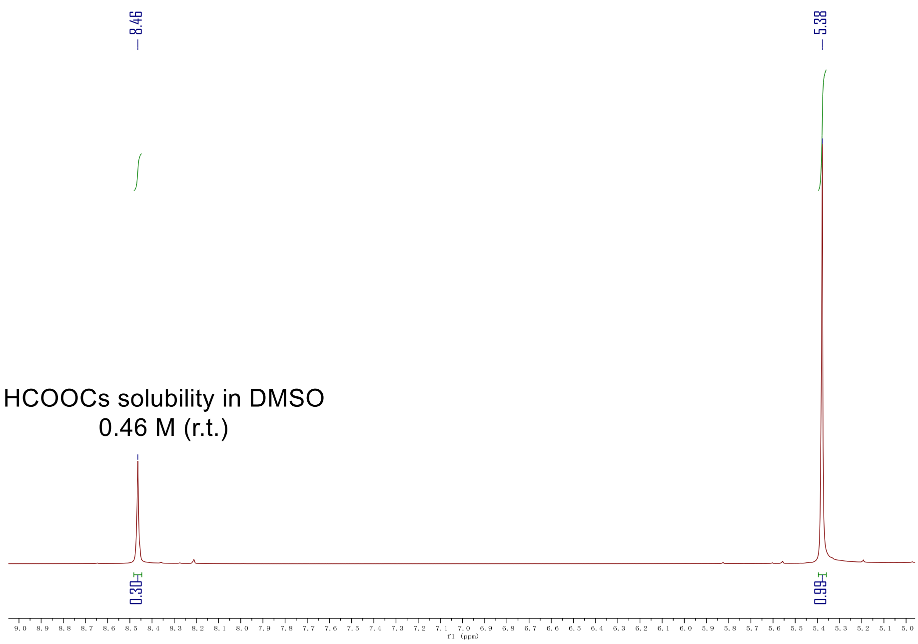


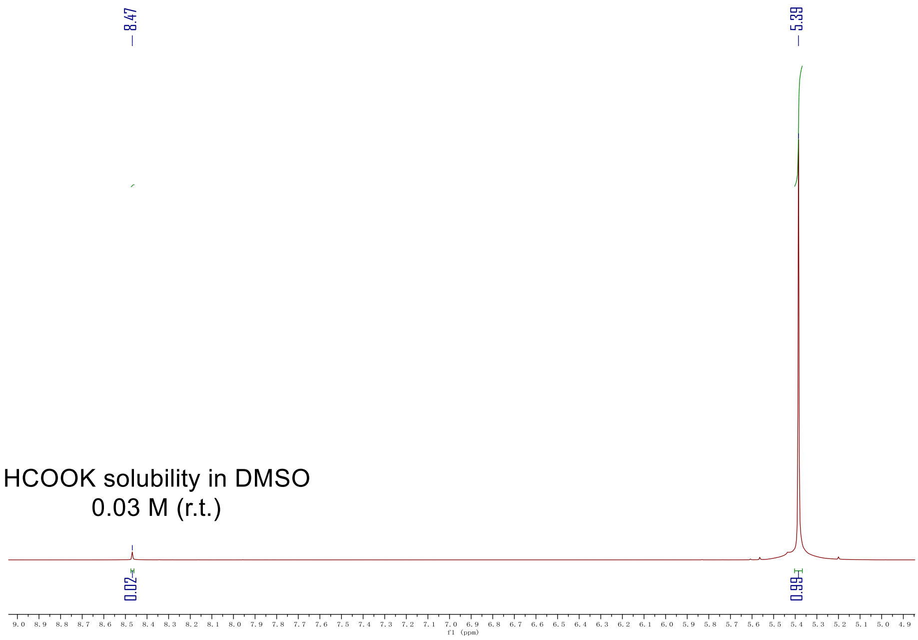
Fig. S5 The proposed mechanism of the oxygen tolerance

10. Formate salt solubility test

Details of HCOOCs solubility test experiment: HCOOCs (138.5 mg, 779 μmol) and CH_2Br_2 (54.0 μL, 769 μmol, 0.988 equiv.) were added to a 2 dram vial. DMSO-d_6 (1 mL) was then added and the mixture was violently shaken for 30 s and 2×10 s. Solubility was determined via ^1H NMR.



Details of HCOOK solubility test experiment: HCOOK (65.5 mg, 779 μmol) and CH_2Br_2 (54.0 μL, 769 μmol, 0.988 equiv.) were added to a 2 dram vial. DMSO-d_6 (1 mL) was then added and the mixture was violently shaken for 30 s and 2×10 s. Solubility was determined via ^{1}H NMR.

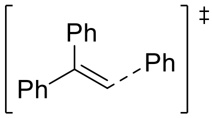


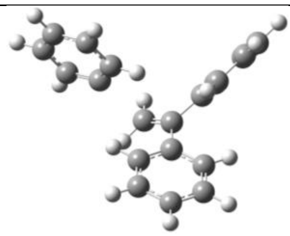
11. DFT calculation

Table S6 Detailed information for DFT calculation

Entry	Chemical structure	Geometric optimization structure	G [a.u.] [a]

Ts-1

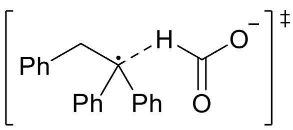


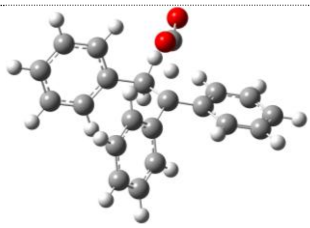


-771.962871

C	0.69918300	0.00610600	-0.93985800
C	1.29347000	1.23239300	-0.35872600
C	1.62598700	1.29264500	0.99730000
C	1.50422600	2.36985100	-1.14101800
C	2.12929300	2.45692400	1.55597100
H	1.47962200	0.41702500	1.61849600
C	2.01298400	3.53267600	-0.58442200
H	1.28635000	2.33086900	-2.20095100
C	2.32390100	3.58276900	0.76750500
H	2.36959400	2.48491700	2.61117800
H	2.17668500	4.40040200	-1.21074400
H	2.72192600	4.49062600	1.20178700
C	1.14840100	-1.31279200	-0.43989500
C	2.48905500	-1.53507100	-0.11251500
C	0.24745400	-2.37185800	-0.30505100
C	2.91730200	-2.78106100	0.31586600
H	3.19960900	-0.72348300	-0.20968800
C	0.67595900	-3.61706100	0.12981500
H	-0.80143600	-2.20599400	-0.51928000
C	2.01236900	-3.82769100	0.43908600
H	3.96214400	-2.93716600	0.55253200
H	-0.03972200	-4.42216500	0.23716700
H	2.34697100	-4.79896900	0.77961000
C	-0.28020800	0.09291800	-1.86422700
H	-0.60359800	1.05251600	-2.24430000
H	-0.64905700	-0.78928200	-2.37027300
C	-2.33349000	0.12064300	-0.55135700
C	-3.55870100	0.16435300	-1.16808700
C	-2.14047200	0.10181000	0.80644500
C	-4.68521500	0.19049200	-0.34572500
H	-3.65799200	0.17908100	-2.24699700
C	-3.27668600	0.12798900	1.61303300
H	-1.14419400	0.06570600	1.23339900
C	-4.54043700	0.17201100	1.03575600
H	-5.67378000	0.22650700	-0.78728900
H	-3.17278400	0.11430300	2.69112900
H	-5.41899700	0.19129100	1.66752000

Ts-2

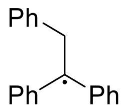
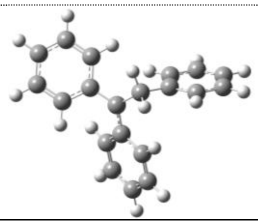




-961.304292

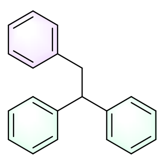
C	-2.07753400	-0.15029500	-0.41787400
C	-2.72560700	-1.02788000	-1.30624700
C	-2.90679500	0.68565500	0.36041200
C	-4.10665300	-1.04497700	-1.42827400
H	-2.14181100	-1.71036400	-1.90833200
C	-4.28171500	0.66410100	0.23460100
H	-2.44631600	1.35036800	1.08081300
C	-4.90122700	-0.19745600	-0.66777300
H	-4.56624700	-1.73489700	-2.12681800

H	-4.88250900	1.32009600	0.85414500
H	-5.97938000	-0.21570000	-0.76392200
C	-0.00237400	1.15953800	0.14868800
C	0.99697800	1.22878400	1.12308000
C	-0.35215400	2.33833100	-0.51713600
C	1.63193700	2.43078200	1.40394800
H	1.25876900	0.33257200	1.67395900
C	0.28187700	3.53990800	-0.23654700
H	-1.13530100	2.30235200	-1.26637700
C	1.28375200	3.59048900	0.72471700
H	2.40399600	2.45853700	2.16348300
H	-0.00399600	4.43741700	-0.77270100
H	1.78286300	4.52616500	0.94585600
C	0.17232300	-1.01576000	-1.19282500
H	-0.16274400	-2.04839000	-1.06357200
C	1.66517100	-0.96551000	-1.00021000
C	2.42634300	0.03611000	-1.60020500
C	2.31227700	-1.89918000	-0.19439000
C	3.79618900	0.11140400	-1.39949900
H	1.92736500	0.78090800	-2.21095700
C	3.68415700	-1.82453000	0.01153000
H	1.72676900	-2.67607500	0.28442400
C	4.43119800	-0.82051600	-0.58822600
H	4.36621900	0.90485000	-1.86776700
H	4.16747400	-2.55283900	0.65106700
H	5.50004000	-0.76112500	-0.42228500
H	-0.05934200	-0.71570400	-2.22438200
C	-0.63191800	-0.14294300	-0.22535200
C	-0.20331800	-1.93604400	1.85713200
H	-0.44278000	-0.97621500	0.98645700
O	-0.47154300	-3.02512000	1.35991300
O	0.25153400	-1.52728900	2.91667500

Int-1			-772.033123

[a] calculated at (u)m062x-gd3/def2tzvp/SMD(DMSO) level

12. Characterization data



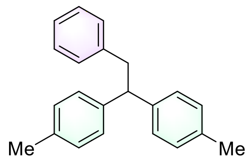
1,1,2-triphenylethane (3a)

44.3 mg, white solid, 86% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.20 (m, 8H), 7.19 – 7.10 (m, 5H), 7.00 (d, *J* = 6.99 Hz, 2H), 4.23 (t, *J* = 7.82 Hz, 1H), 3.37 (d, *J* = 7.86 Hz, 2H).

The spectral data were in accordance with those reported in the literature.[4] [5] [6]



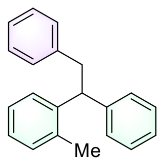
4,4'-(2-phenylethane-1,1-diyl)bis(methyl-benzene) (3b)

30.0 mg, white solid, 52% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (t, *J* = 7.28 Hz, 2H), 7.11 – 7.00 (m, 11H), 4.17 (t, *J* = 7.79 Hz, 1H), 3.32 (d, *J* = 7.78 Hz, 2H).

The spectral data were in accordance with those reported in the literature.[6]



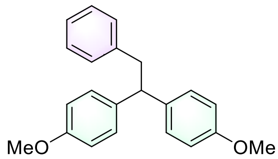
(1-(*o*-tolyl)ethane-1,2-diyl)dibenzene (3c)

19.3 mg, white solid, 35% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22 – 7.06 (m, 12H), 6.97 (d, *J* = 7.33 Hz, 2H), 4.41 (t, *J* = 7.67 Hz, 1H), 3.33 (d, *J* = 7.28 Hz, 2H), 2.12 (s, 3H).

The spectral data were in accordance with those reported in the literature. [4]



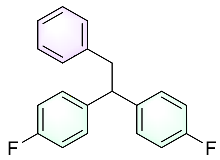
4,4'-(2-phenylethane-1,1-diyl)bis(methoxybenzene) (3d)

21.9 mg, white solid, 34% yield.

R_f (PE/EA 200/1 v/v): 0.1

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (t, *J* = 7.27 Hz, 2H), 7.13 – 7.08 (m, 5H), 7.00 (t, *J* = 6.46 Hz, 2H), 6.79 (d, *J* = 8.67 Hz, 4H), 4.14 (t, *J* = 7.80 Hz, 1H), 3.76 (s, 6H), 3.29 (d, *J* = 7.81 Hz, 2H).

The spectral data were in accordance with those reported in the literature. [6]



4,4'-(2-phenylethane-1,1-diyl)bis(fluorobenzene) (3e)

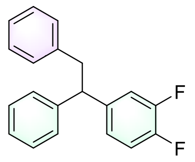
41.9 mg, white solid, 71% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 – 7.09 (m, 7H), 7.00 – 6.89 (m, 6H), 4.19 (t, *J* = 7.85 Hz, 1H), 3.29 (d, *J* = 7.86 Hz, 2H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -116.8.

The spectral data were in accordance with those reported in the literature.[7]



(1-(3,4-difluorophenyl)ethane-1,2-diyl)dibenzene (3f)

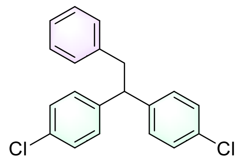
49.1 mg, white solid, 83% yield.

R_f (PE/EA 200/1 v/v): 0.26

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.57 Hz, 2H), 7.22 – 7.13 (m, 6H), 7.04 – 6.94 (m, 4H), 6.90 – 6.84 (m, 1H), 4.20 (t, *J* = 7.85 Hz, 1H), 3.38 – 3.25 (m, 2H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -137.9 (d, *J* = 21.16 Hz), -141.5 (d, *J* = 21.32 Hz).

The spectral data were in accordance with those reported in the literature.[8]



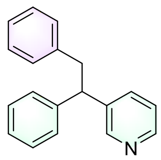
4,4'-(2-phenylethane-1,1-diyl)bis(chlorobenzene) (3g)

40.8 mg, white solid, 62% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.41 Hz, 4H), 7.19 – 7.12 (m, 3H), 7.08 (d, *J* = 8.48 Hz, 4H), 7.00 – 6.95 (m, 2H), 4.18 (t, *J* = 7.82 Hz, 1H), 3.29 (d, *J* = 7.86 Hz, 2H).

The spectral data were in accordance with those reported in the literature.[9]



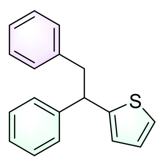
3-(1,2-diphenylethyl)pyridine (3h)

42.7 mg, white solid, 82% yield.

R_f (PE/EA 4/1 v/v): 0.55

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45 (d, *J* = 5.39 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.23 – 7.14 (m, 6H), 7.09 (d, *J* = 6.15 Hz, 2H), 6.99 (d, *J* = 6.97 Hz, 2H), 4.20 (t, *J* = 7.85 Hz, 1H), 3.35 (qd, *J* = 13.68, 7.88 Hz, 2H).

The spectral data were in accordance with those reported in the literature. [6]



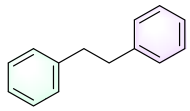
2-(1,2-diphenylethyl)thiophene (3i)

18.2 mg, white solid, 34% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 – 7.22 (m, 2H), 7.21 – 7.15 (m, 5H), 7.14 – 7.09 (m, 2H), 7.01 (d, *J* = 7.14 Hz, 2H), 6.90 – 6.85 (m, 1H), 6.79 (d, *J* = 3.26 Hz, 1H), 4.43 (t, *J* = 7.75 Hz, 1H), 3.43 (dd, *J* = 13.60, 7.25 Hz, 1H), 3.30 (dd, *J* = 13.57, 8.25 Hz, 1H).

The spectral data were in accordance with those reported in the literature. [4] [6]



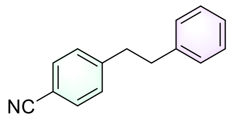
1,2-diphenylethane (3j)

14.0 mg, white solid, 38% yield.

R_f (PE/EA 200/1 v/v): 0.67

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.62 Hz, 4H), 7.24 – 7.16 (m, 6H), 2.93 (s, 4H).

The spectral data were in accordance with those reported in the literature.[5]



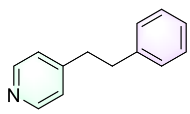
4-phenethylbenzonitrile (3k)

12.2 mg, white solid, 29% yield.

R_f (PE/EA 200/1 v/v): 0.20

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, J = 8.26 Hz, 2H), 7.28 – 7.20 (m, 5H), 7.12 (d, J = 7.06 Hz, 2H), 3.09 – 2.81 (m, 4H).

The spectral data were in accordance with those reported in the literature. [5]



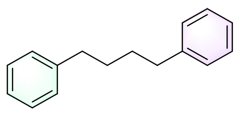
4-phenethylpyridine (3l)

4.9 mg, white solid, 13% yield.

R_f (PE/EA 4/1 v/v): 0.45

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, *J* = 5.06 Hz, 2H), 7.28 (t, *J* = 7.47 Hz, 2H), 7.21 (t, *J* = 7.40 Hz, 1H), 7.15 (d, *J* = 7.06 Hz, 2H), 7.08 (d, *J* = 5.67 Hz, 2H), 2.93 (s, 4H).

The spectral data were in accordance with those reported in the literature.[10]



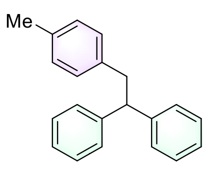
1,4-diphenylbutane (3m)

24.6 mg, white solid, 58% yield.

R_f (PE/EA 200/1 v/v): 0.60

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27 – 7.25 (m, 5H), 7.18 – 7.15 (m, 5H), 2.63 (t, *J* = 6.90 Hz, 4H), 1.68 – 1.65 (m, 4H).

The spectral data were in accordance with those reported in the literature.[11]



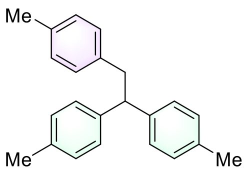
(2-(*p*-tolyl)ethane-1,1-diyl)dibenzene (3n)

48.6 mg, white solid, 89% yield.

R_f (PE/EA 200/1 v/v): 0.35

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 8H), 7.18 – 7.13 (m, 2H), 6.97 (d, *J* = 7.86 Hz, 2H), 6.89 (d, *J* = 7.94 Hz, 2H), 4.21 (t, *J* = 7.79 Hz, 1H), 3.33 (d, *J* = 7.77 Hz, 2H), 2.26 (s, 3H).

The spectral data were in accordance with those reported in the literature. [4]



4,4',4''-(ethane-1,1,2-triyl)tris(methylbenzene) (3o)

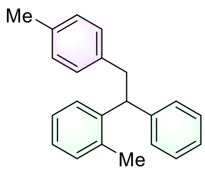
22.4 mg, yellow solid, 37% yield.

R_f (PE/EA 200/1 v/v): 0.30

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.09 (d, *J* = 8.08 Hz, 4H), 7.04 (d, *J* = 5.76 Hz, 4H), 6.97 (d, *J* = 7.75 Hz, 2H), 6.91 (d, *J* = 7.71 Hz, 2H), 4.15 (t, *J* = 7.75 Hz, 1H), 3.29 (d, *J* = 7.70 Hz, 2H), 2.28 (s, 6H), 2.26 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 142.0, 135.6, 129.1, 129.0, 128.9, 128.0, 52.4, 41.8, 21.1.

The spectral data were in accordance with those reported in the literature.[12]



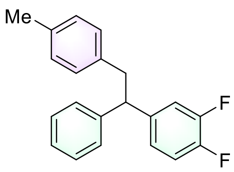
1-methyl-2-(1-phenyl-2-(p-tolyl)ethyl)benzene (3p)

34.4 mg, yellow solid, 60% yield.

R_f (PE/EA 200/1 v/v): 0.38

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.70 Hz, 1H), 7.21 (t, *J* = 7.39 Hz, 3H), 7.15 – 7.06 (m, 5H), 6.97 (d, *J* = 7.80 Hz, 2H), 6.87 (d, *J* = 7.89 Hz, 2H), 4.40 (t, *J* = 7.68 Hz, 1H), 3.29 (dd, *J* = 7.63, 2.40 Hz, 2H), 2.27 (s, 3H), 2.13 (s, 3H).

The spectral data were in accordance with those reported in the literature.[13]



1,2-difluoro-4-(1-phenyl-2-(p-tolyl)ethyl)benzene (3q)

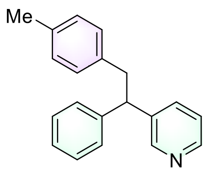
51.2 mg, white solid, 83% yield.

R_f (PE/EA 200/1 v/v): 0.45

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.66 Hz, 2H), 7.18 (d, *J* = 7.51 Hz, 3H), 7.03 – 6.93 (m, 4H), 6.87 (d, *J* = 7.57 Hz, 3H), 4.16 (t, *J* = 7.87 Hz, 1H), 3.33 – 3.19 (m, 2H), 2.27 (s, 3H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -138.0 (d, *J* = 21.1 Hz), -141.6 (d, *J* = 20.9 Hz).

The spectral data were in accordance with those reported in the literature.[8]



3-(1-phenyl-2-(*p*-tolyl)ethyl)pyridine (3r)

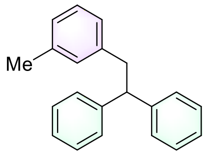
47.0 mg, yellow solid, 86% yield.

R_f (PE/EA 4/1 v/v): 0.73

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45 (d, *J* = 6.13 Hz, 2H), 7.28 (t, *J* = 7.63 Hz, 2H), 7.20 (d, *J* = 8.49 Hz, 3H), 7.10 (d, *J* = 6.12 Hz, 2H), 6.99 (d, *J* = 7.73 Hz, 2H), 6.89 (d, *J* = 7.81 Hz, 2H), 4.19 (t, *J* = 7.83 Hz, 1H), 3.31 (qd, *J* = 13.72, 7.79 Hz, 2H), 2.27 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.5, 149.8, 142.9, 136.3, 135.8, 129.1, 128.9, 128.7, 128.1, 126.9, 123.6, 52.7, 41.1, 21.1.

The spectral data were in accordance with those reported in the literature.[14]



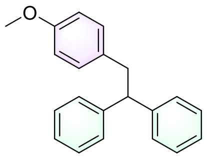
(2-(*m*-tolyl)ethane-1,1-diyl)dibenzene (3s)

33.8 mg, white solid, 62% yield.

R_f (PE/EA 200/1 v/v): 0.55

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 8H), 7.19 – 7.15 (m, 2H), 7.06 (t, *J* = 7.55 Hz, 1H), 6.94 (d, *J* = 7.50 Hz, 1H), 6.84 (s, 1H), 6.80 (d, *J* = 7.56 Hz, 1H), 4.24 (t, *J* = 7.76 Hz, 1H), 3.33 (d, *J* = 7.70 Hz, 2H), 2.25 (s, 3H).

The spectral data were in accordance with those reported in the literature.[4]



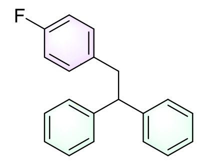
(2-(4-methoxyphenyl)ethane-1,1-diyl)dibenzene (3t)

45.8 mg, yellow solid, 79% yield.

R_f (PE/EA 200/1 v/v): 0.26

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 – 7.14 (m, 8H), 7.16 (t, *J* = 7.12 Hz, 2H), 6.90 (d, *J* = 8.59 Hz, 2H), 6.71 (d, *J* = 8.62 Hz, 2H), 4.18 (t, *J* = 7.76 Hz, 1H), 3.74 (s, 3H), 3.30 (d, *J* = 7.78 Hz, 2H).

The spectral data were in accordance with those reported in the literature.[4]



(2-(4-fluorophenyl)ethane-1,1-diyl)dibenzene (3u)

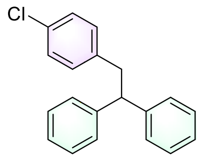
46.9 mg, white solid, 85% yield.

R_f (PE/EA 200/1 v/v): 0.45

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 – 7.23 (m, 4H), 7.19 – 7.14 (m, 6H), 6.94 – 6.91 (m, 2H), 6.84 (t, *J* = 8.73 Hz, 2H), 4.16 (t, *J* = 7.85 Hz, 1H), 3.32 (d, *J* = 7.83 Hz, 2H).

^{19}**F NMR** (471 MHz, $CDCl_3$) δ -117.5.

The spectral data were in accordance with those reported in the literature. [4]



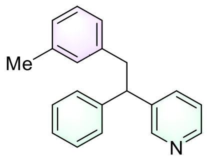
(2-(4-chlorophenyl)ethane-1,1-diyl)dibenzene (3v)

48.7 mg, white solid, 83% yield.

R_f (PE/EA 200/1 v/v): 0.37

^{1}**H NMR** (500 MHz, $CDCl_3$) δ 7.27 – 7.24 (m, 4H), 7.19 – 7.15 (m, 6H), 7.12 (d, *J* = 8.35 Hz, 2H), 6.91 (d, *J* = 8.37 Hz, 2H), 4.17 (t, *J* = 7.87 Hz, 1H), 3.32 (d, *J* = 7.85 Hz, 2H).

The spectral data were in accordance with those reported in the literature. [4]



3-(1-phenyl-2-(*m*-tolyl)ethyl)pyridine (3w)

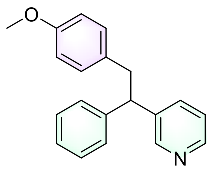
31.2 mg, orange solid, 57% yield.

R_f (PE/EA 4/1 v/v): 0.65

^{1}**H NMR** (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 6.17 Hz, 2H), 7.29 (t, *J* = 7.50 Hz, 2H), 7.23 – 7.19 (m, 3H), 7.11 (d, *J* = 6.17 Hz, 2H), 7.07 (t, *J* = 7.55 Hz, 1H), 6.96 (d, *J* = 7.65 Hz, 1H), 6.83 (s, 1H), 6.79 (d, *J* = 7.55 Hz, 1H), 4.22 (t, *J* = 7.81 Hz, 1H), 3.37 – 3.27 (m, 2H), 2.25 (s, 3H).

^{13}**C NMR** (126 MHz, $CDCl_3$) δ 153.4, 149.8, 142.9, 139.3, 137.9, 129.9, 128.7, 128.2, 128.1, 127.1, 126.9, 126.1, 123.6, 52.6, 41.4, 21.5.

The spectral data were in accordance with those reported in the literature.[14] [16]



3-(2-(4-methoxyphenyl)-1-phenylethyl)pyridine (3x)

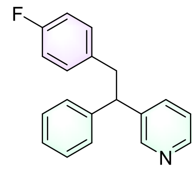
27.4 mg, orange solid, 47% yield.

R_f (PE/EA 4/1 v/v): 0.63

^{1}**H NMR** (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 5.24 Hz, 2H), 7.32 – 7.27 (m, 2H), 7.21 (t, *J* = 6.05 Hz, 3H), 7.10 (d, *J* = 6.12 Hz, 2H), 6.91 (d, *J* = 8.38 Hz, 2H), 6.73 (d, *J* = 8.49 Hz, 2H), 4.17 (t, *J* = 7.82 Hz, 1H), 3.75 (s, 3H), 3.30 (qd, *J* = 13.78, 7.82 Hz, 2H).

^{13}**C NMR** (126 MHz, $CDCl_3$) δ 158.1, 153.4, 149.8, 142.8, 131.4, 130.0, 128.7, 128.1, 126.9, 123.6, 113.8, 55.3, 52.9, 40.6.

The spectral data were in accordance with those reported in the literature.[15] [16]



3-(2-(4-fluorophenyl)-1-phenylethyl)pyridine (3y)

31.6 mg, orange solid, 57% yield.

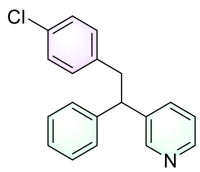
R_f (PE/EA 4/1 v/v): 0.63

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.42 (d, *J* = 6.16 Hz, 2H), 7.26 – 7.21 (m, 2H), 7.19 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 7.05 (d, *J* = 6.13 Hz, 2H), 6.90 – 6.88 (m, 2H), 6.82 (t, *J* = 8.68 Hz, 2H), 4.11 (t, *J* = 7.87 Hz, 1H), 3.33 – 3.24 (m, 2H)

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.5 (d, *J* = 244.38 Hz), 153.1, 149.9, 142.4, 135.0 (d, *J* = 2.50 Hz), 130.5 (d, *J* = 8.75 Hz), 128.8, 128.1, 127.0, 123.5, 115.3, 115.1, 52.8, 40.6.

^{19}F NMR (471 MHz, $CDCl_3$) δ -116.7.

The spectral data were in accordance with those reported in the literature.[16]



3-(2-(4-chlorophenyl)-1-phenylethyl)pyridine (3z)

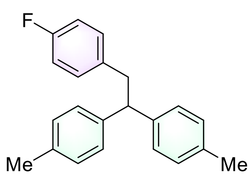
32.3 mg, yellow solid, 55% yield.

R_f (PE/EA 4/1 v/v): 0.57

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (d, *J* = 5.40 Hz, 2H), 7.25 – 7.22 (m, 2H), 7.18 (t, *J* = 7.40 Hz, 1H), 7.13 – 7.10 (m, 4H), 7.06 (d, *J* = 6.12 Hz, 2H), 6.87 (d, *J* = 8.43 Hz, 2H), 4.11 (t, *J* = 7.90 Hz, 1H), 3.32 – 3.23 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 153.0, 149.9, 142.3, 137.8, 132.2, 130.4, 128.8, 128.5, 128.1, 127.1, 123.5, 52.6, 40.8.

The spectral data were in accordance with those reported in the literature.[16]



4,4’-(2-(4-fluorophenyl)ethane-1,1-diyl)bis(methyl-benzene) (3aa)

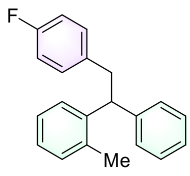
33.7 mg, white solid, 55% yield.

R_f (PE/EA 200/1 v/v): 0.43

^{1}H NMR (500 MHz, $CDCl_3$) δ7.08 – 7.03 (m, 8H), 6.95 – 6.93 (m, 2H), 6.84 (t, *J* = 8.75 Hz, 2H), 4.10 (t, *J* = 7.82 Hz, 1H), 3.29 (d, *J* = 7.85 Hz, 2H), 2.28 (s, 6H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -117.6.

The spectral data were in accordance with those reported in the literature.[13]



1-(2-(4-fluorophenyl)-1-phenylethyl)-2-methylbenzene (3ab)

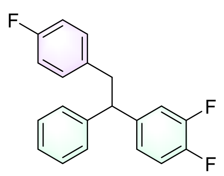
36.1 mg, white solid, 62% yield.

R_f (PE/EA 200/1 v/v): 0.45

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.73 Hz, 1H), 7.21 (t, *J* = 7.72 Hz, 3H), 7.16 – 7.06 (m, 5H), 6.92 – 6.81 (m, 4H), 4.34 (t, *J* = 7.73 Hz, 1H), 3.29 (d, *J* = 8.85 Hz, 2H), 2.11 (s, 3H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -117.5.

The spectral data were in accordance with those reported in the literature.[13]



1,2-difluoro-4-(2-(4-fluorophenyl)-1-phenylethyl)-benzene (3ac)

57.4 mg, white solid, 92% yield.

R_f (PE/EA 200/1 v/v): 0.51

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.20 Hz, 2H), 7.22 – 7.14 (m, 3H), 7.04 – 6.90 (m, 4H), 6.86 (t, *J* = 8.70 Hz, 3H), 4.12 (t, *J* = 7.90 Hz, 1H), 3.33 – 3.21 (m, 2H).

^{19}F NMR (471 MHz, $CDCl_3$) δ -117.0, -137.7 (d, *J* = 21.3 Hz), -141.3 (d, *J* = 21.2 Hz).

The spectral data were in accordance with those reported in the literature.[8]

13. References

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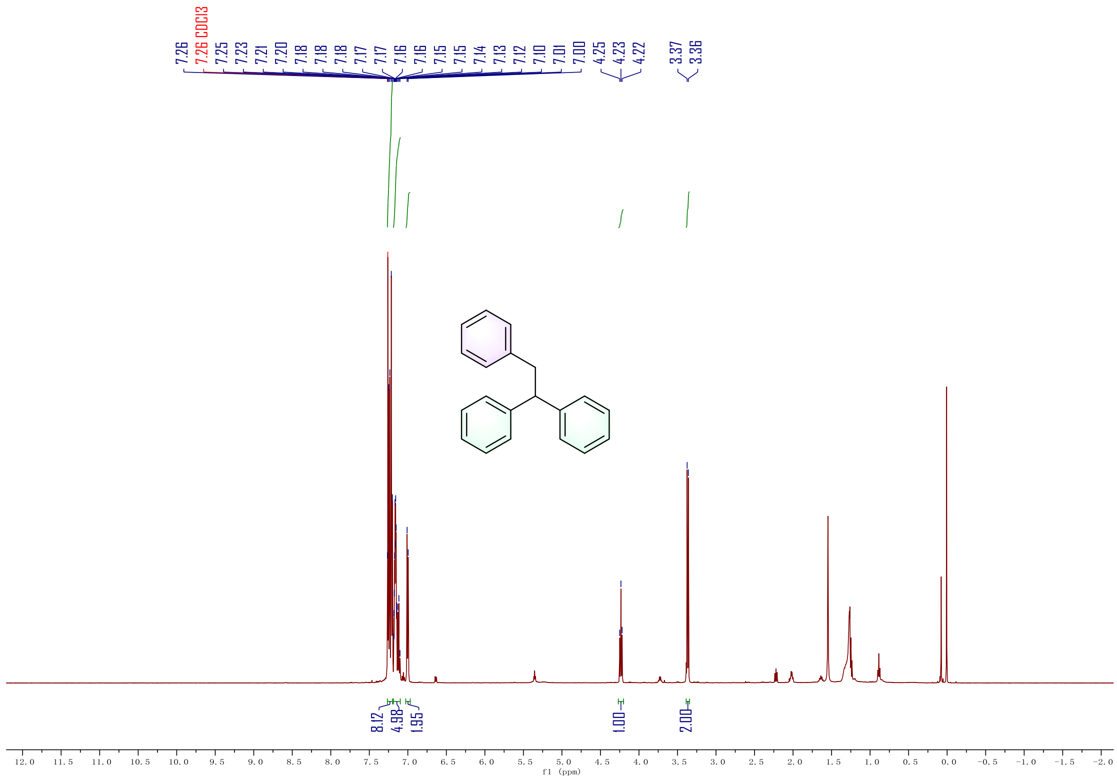
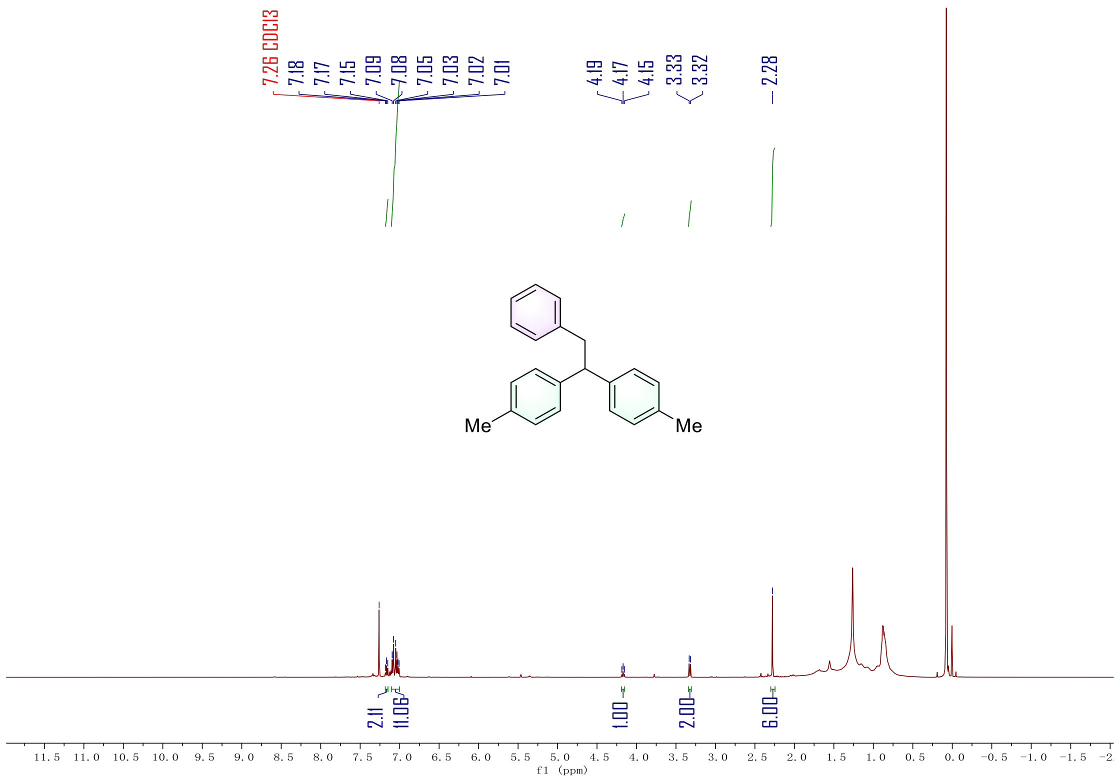
14. ^{1}H and ^{13}C NMR spectra

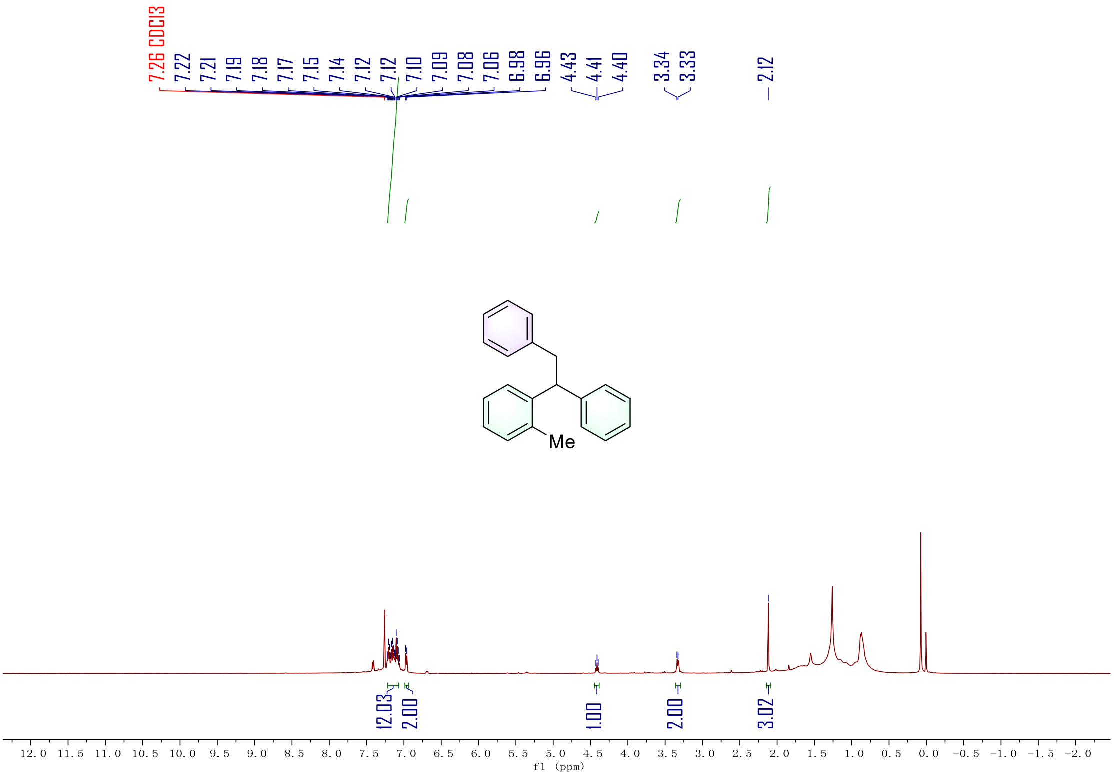
^{1}H NMR of **3a** (500 MHz, $CDCl_3$)



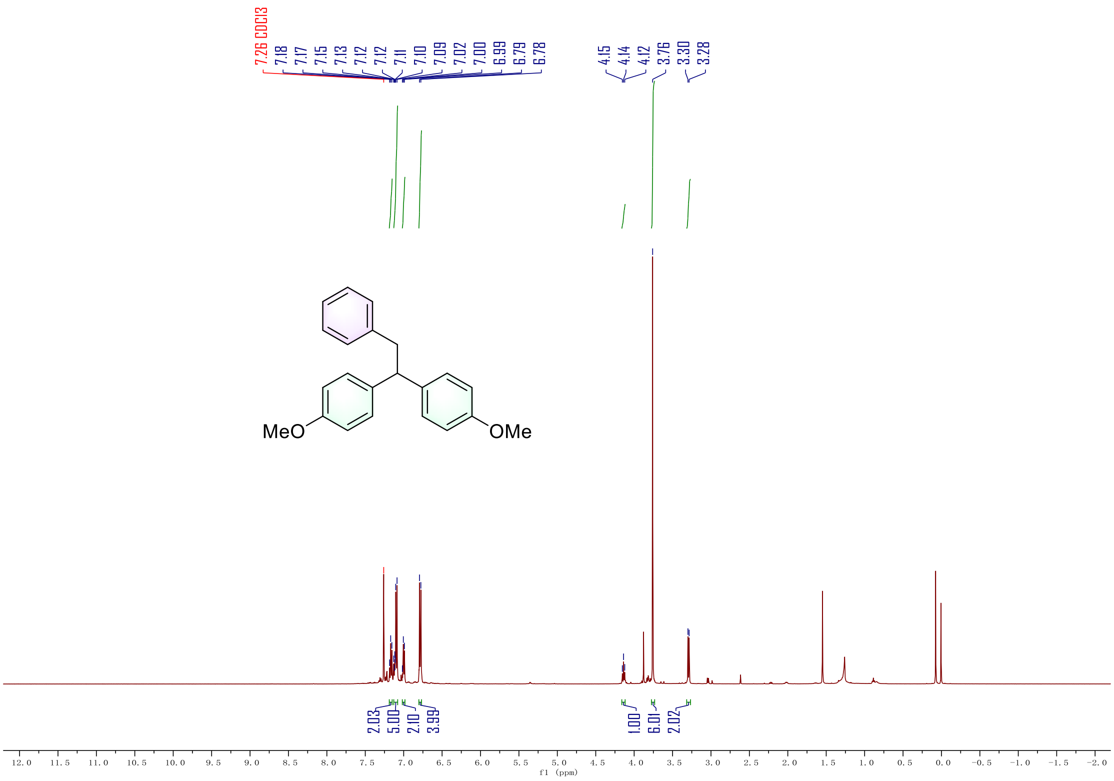
^{1}H NMR of **3b** (500 MHz, $CDCl_3$)



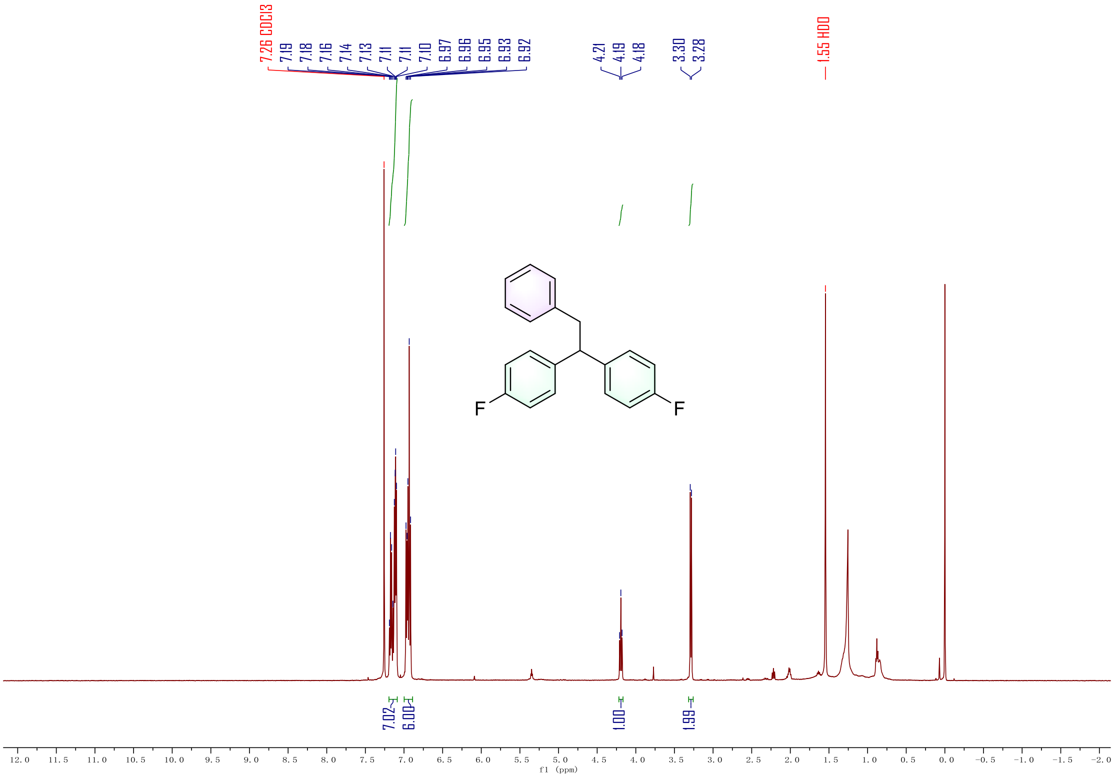
^{1}H NMR of **3c** (500 MHz, $CDCl_3$)



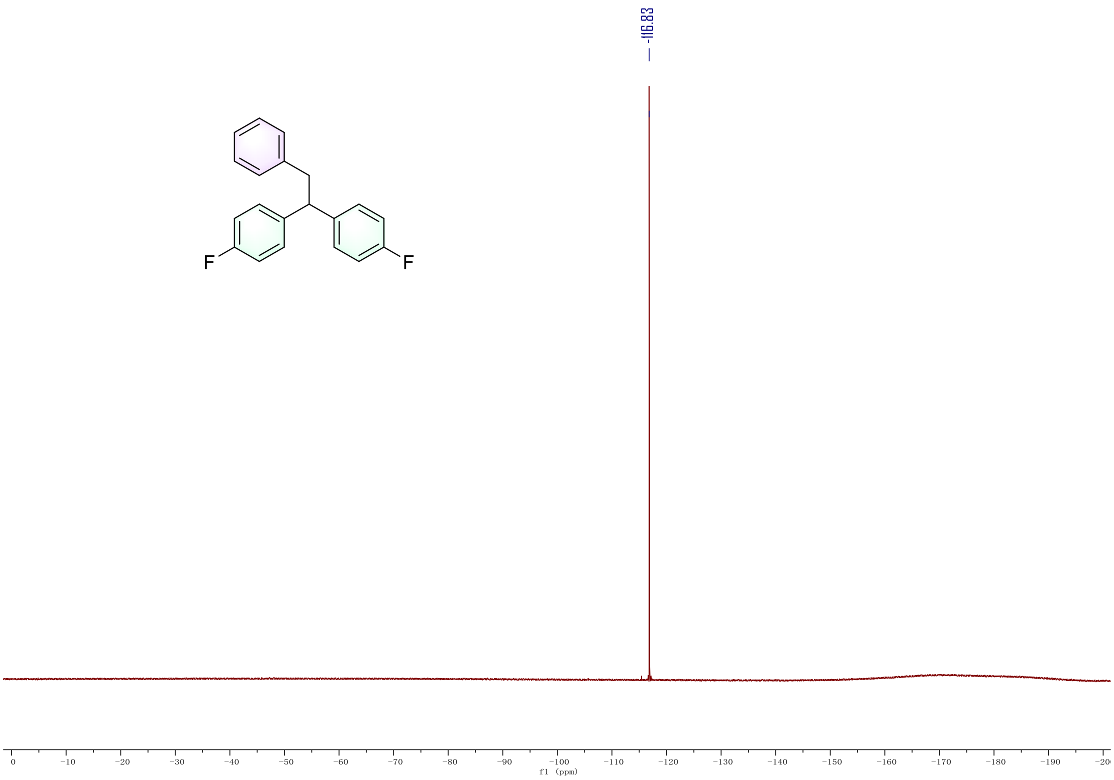
^{1}H NMR of **3d** (500 MHz, $CDCl_3$)



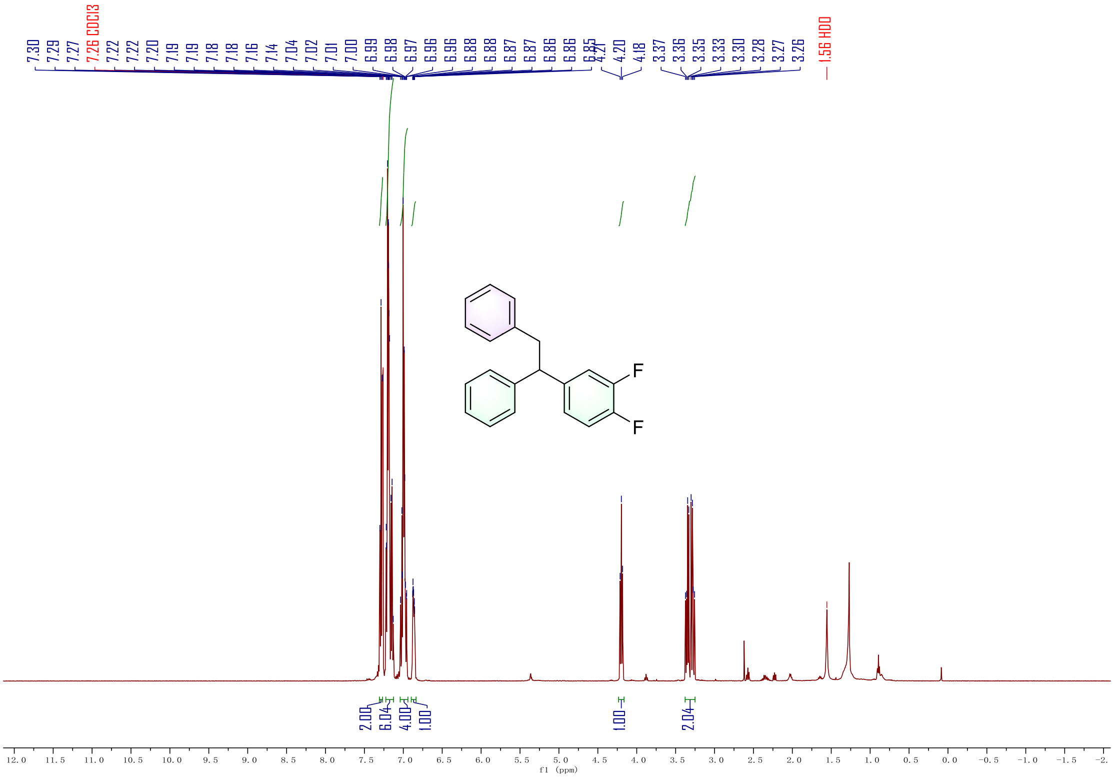
^{1}H NMR of **3e** (500 MHz, $CDCl_3$)



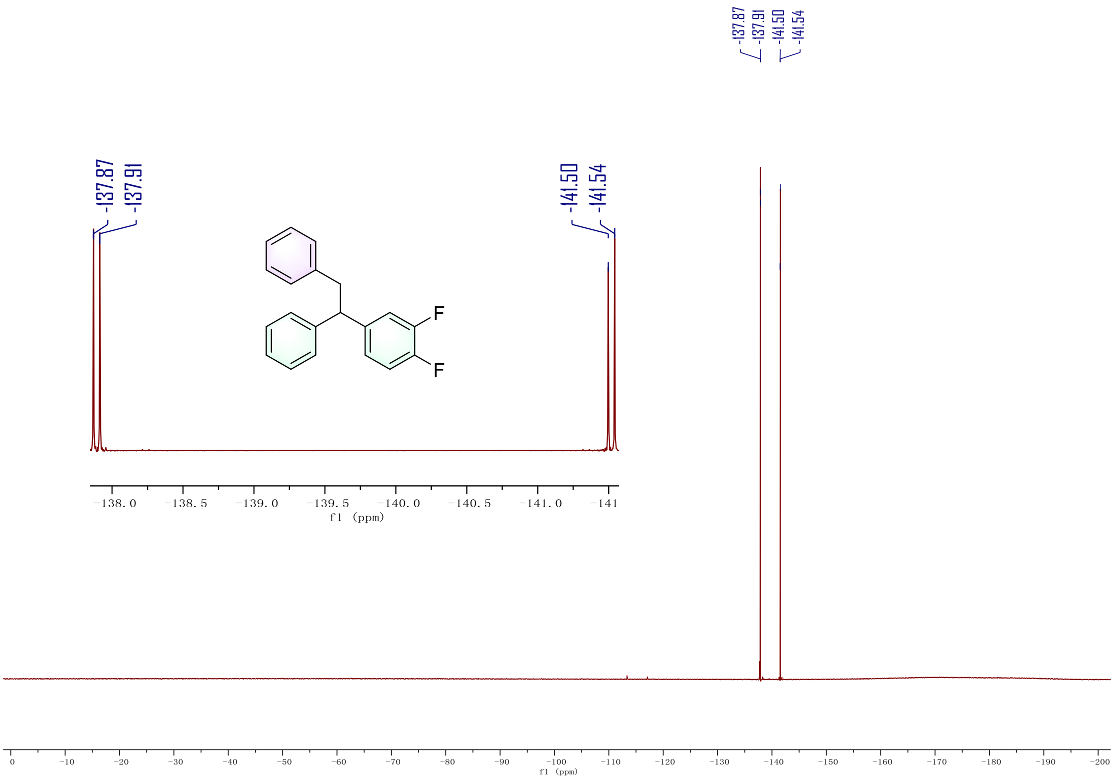
^{19}F NMR of **3e** (471 MHz, $CDCl_3$)



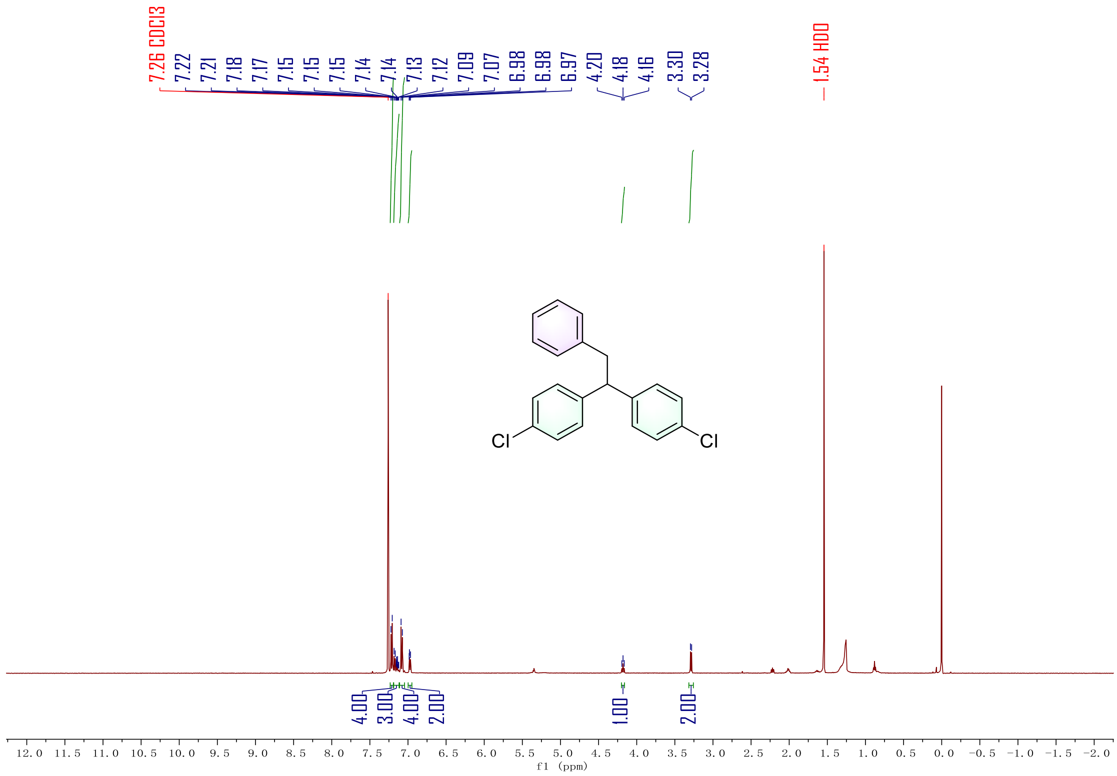
[1]H NMR of **3f** (500 MHz, $CDCl_3$)



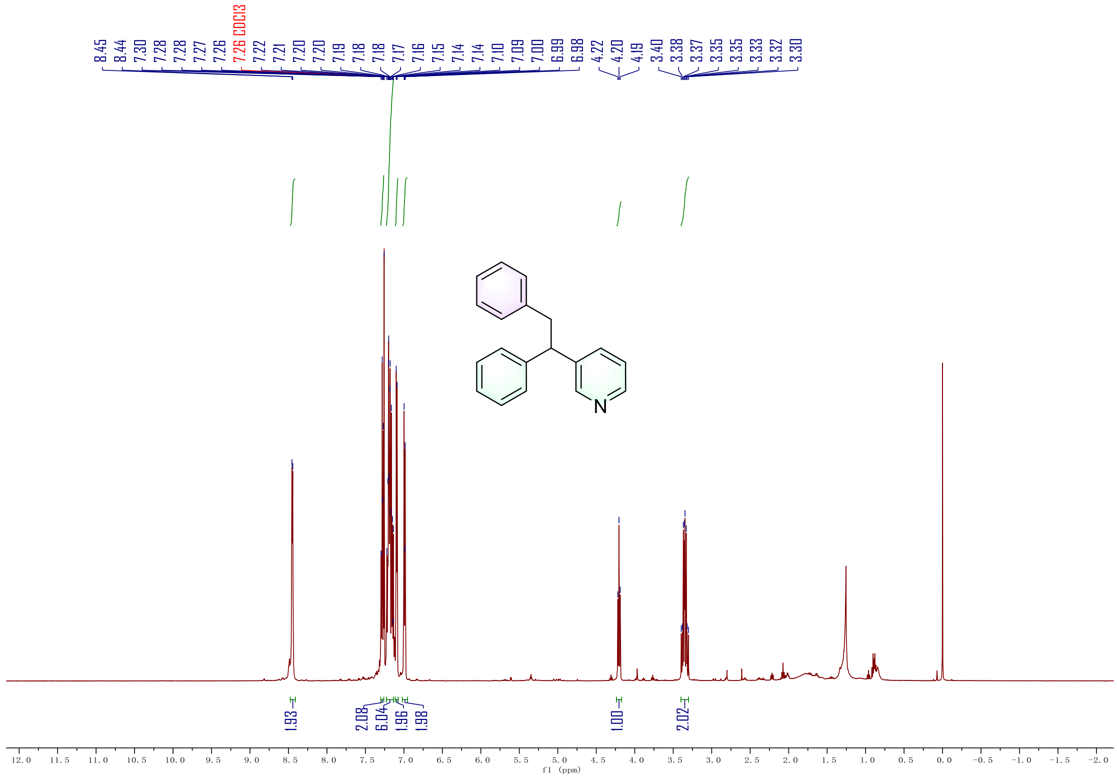
[19]F NMR of **3f** (471 MHz, $CDCl_3$)



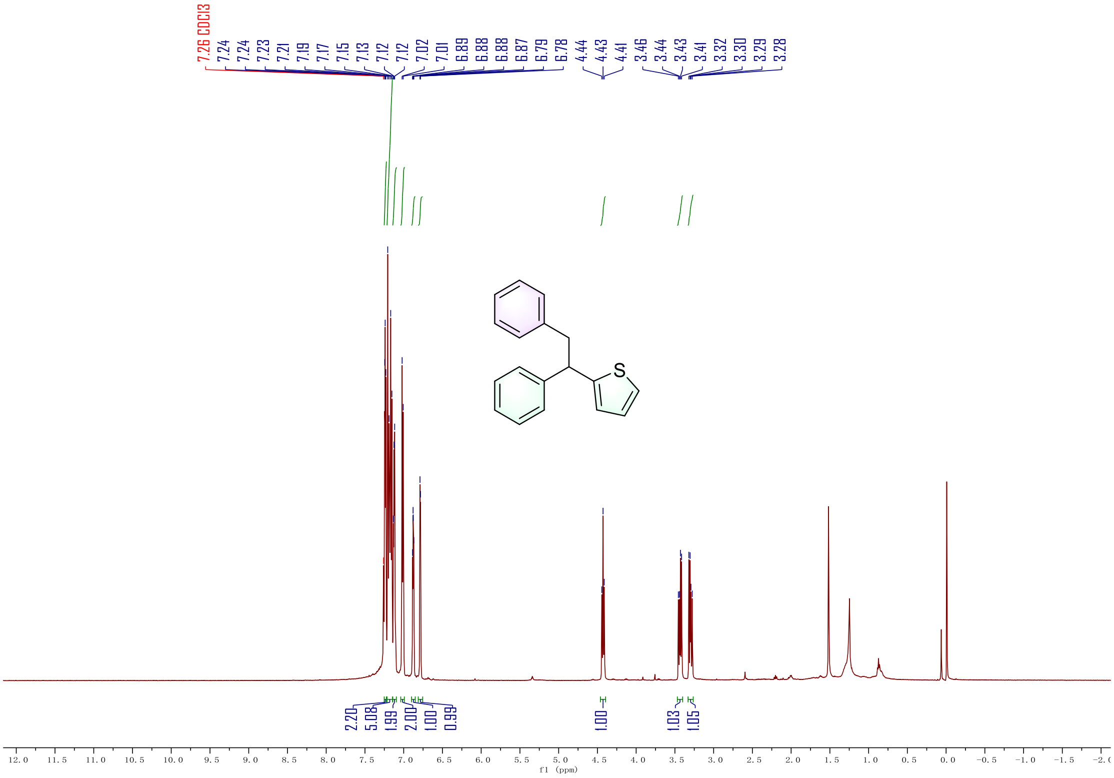
^{1}H NMR of **3g** (500 MHz, $CDCl_3$)



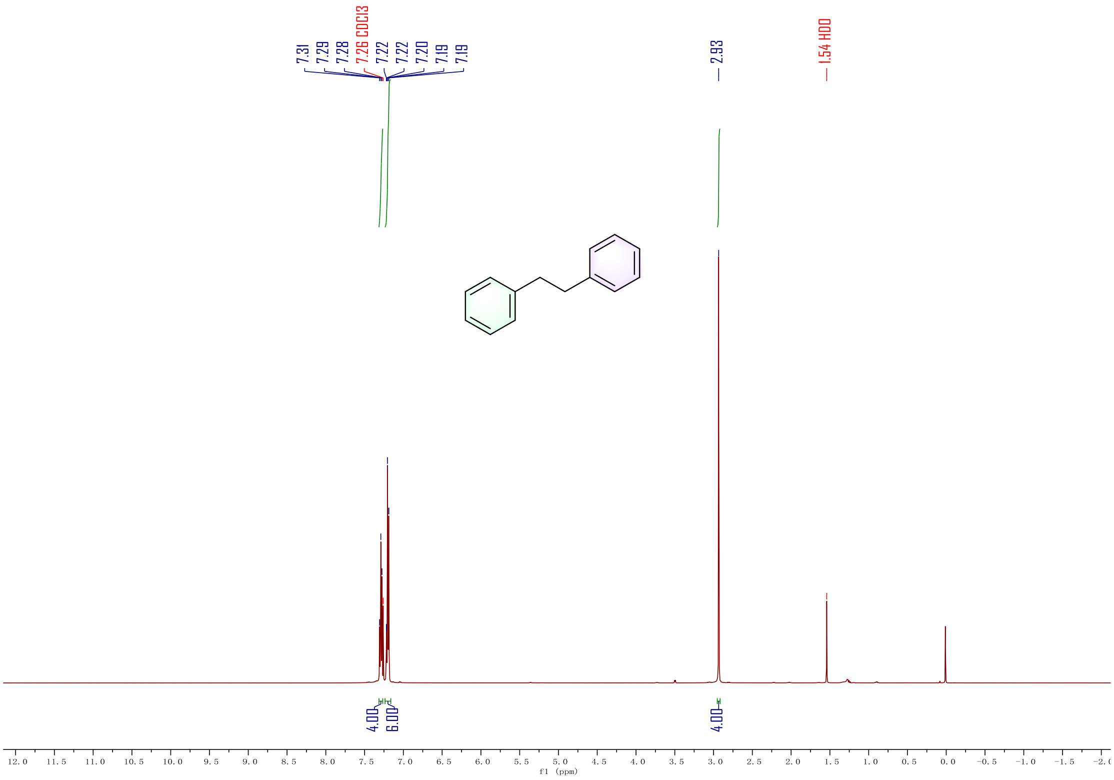
^{1}H NMR of **3h** (500 MHz, $CDCl_3$)



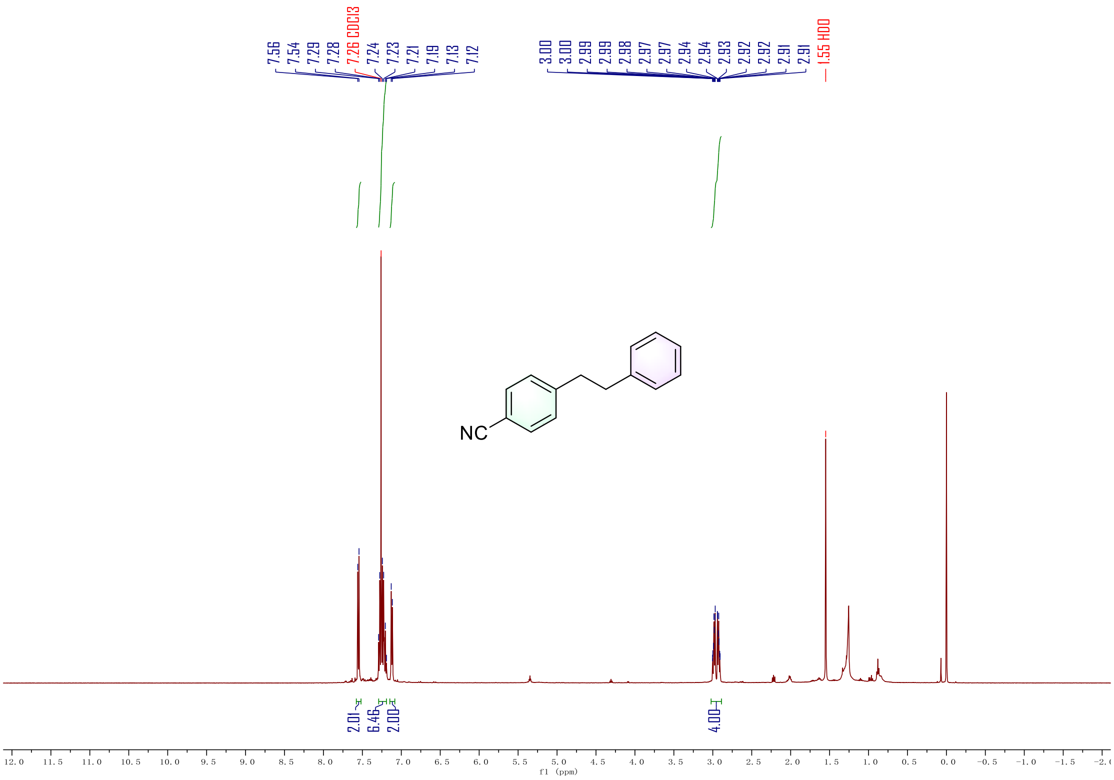
^{1}H NMR of **3i** (500 MHz, $CDCl_3$)



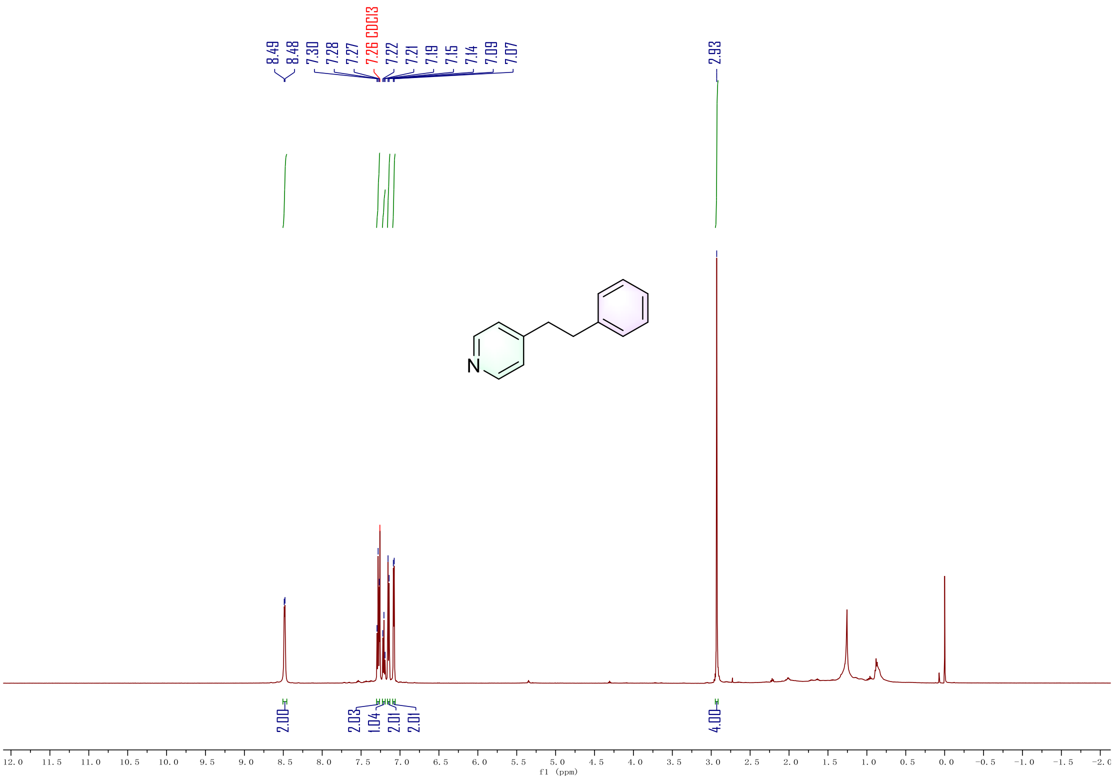
^{1}H NMR of **3j** (500 MHz, $CDCl_3$)



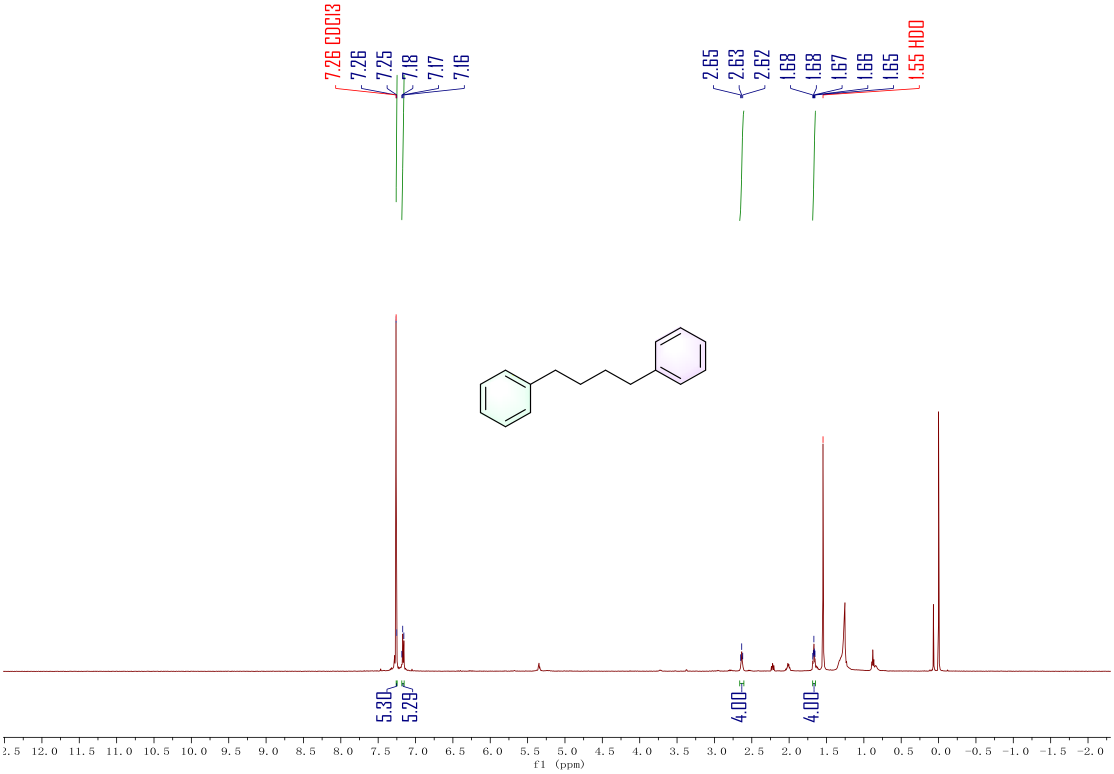
[1]H NMR of **3k** (500 MHz, $CDCl_3$)



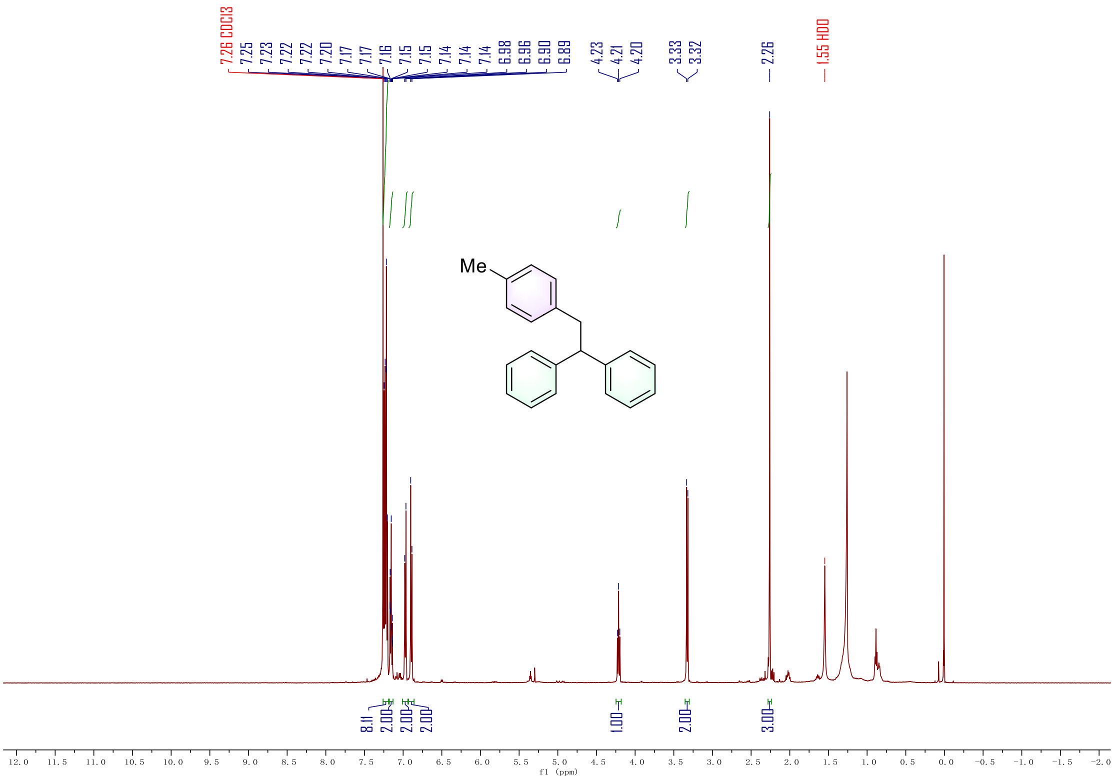
[1]H NMR of **3l** (500 MHz, $CDCl_3$)



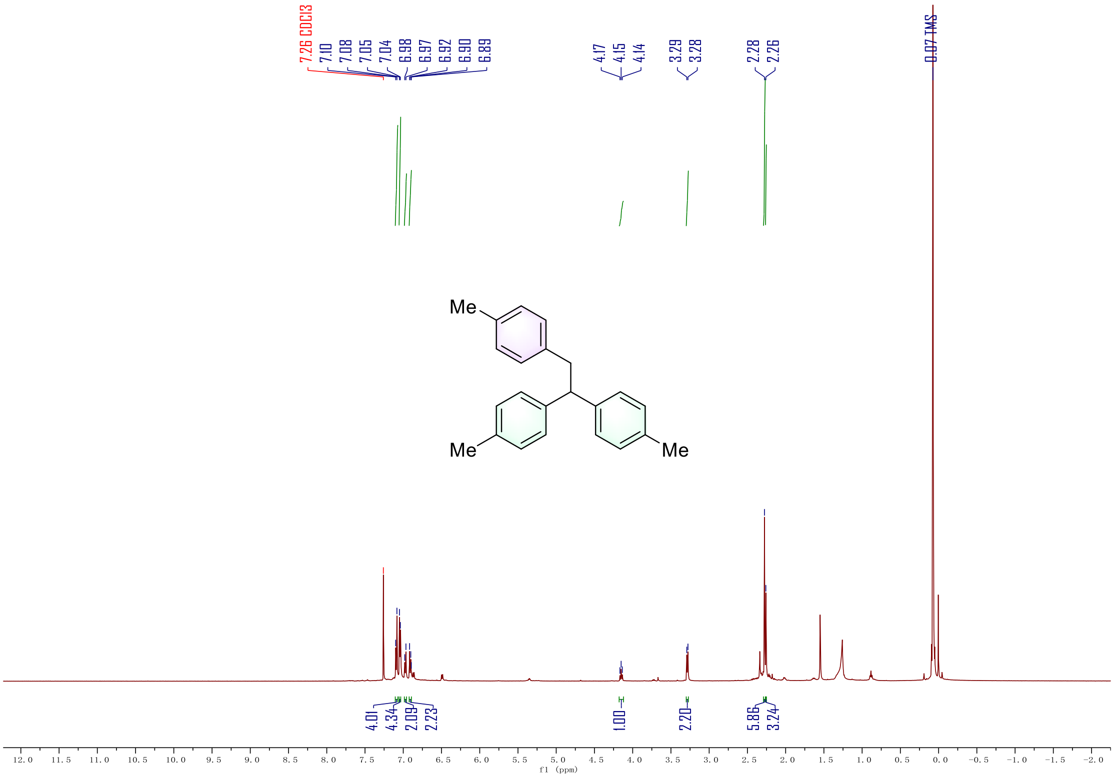
^{1}H NMR of **3m** (500 MHz, $CDCl_3$)



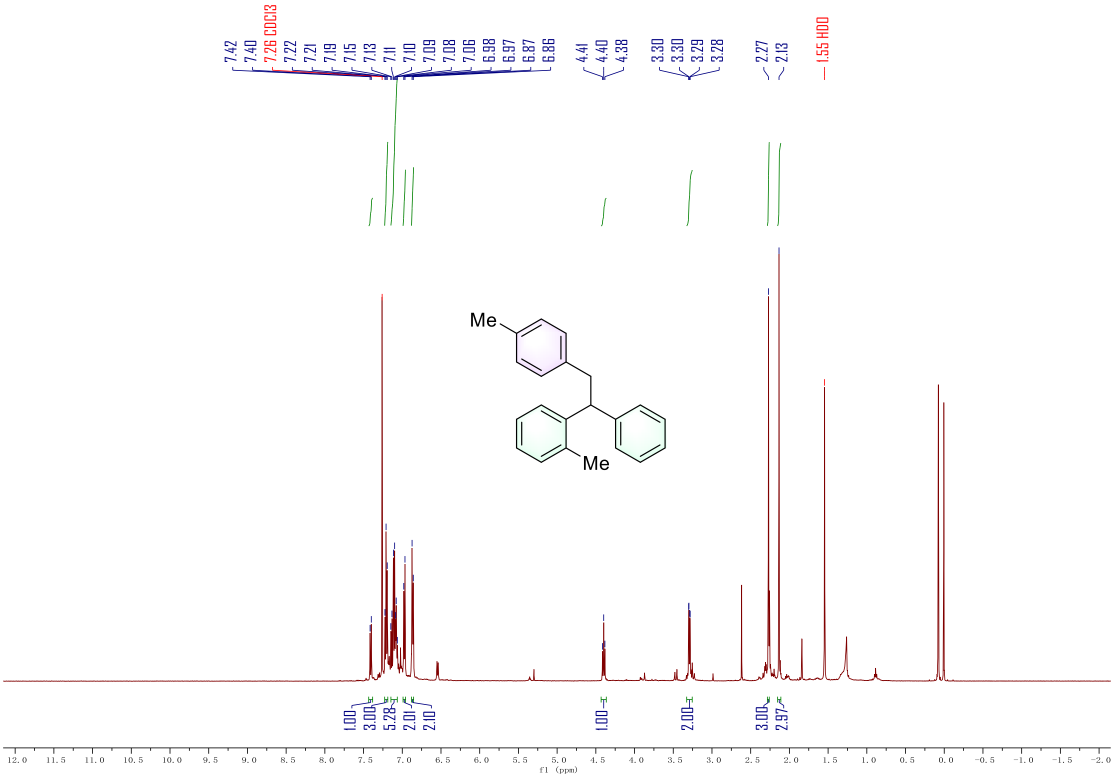
^{1}H NMR of **3n** (500 MHz, $CDCl_3$)



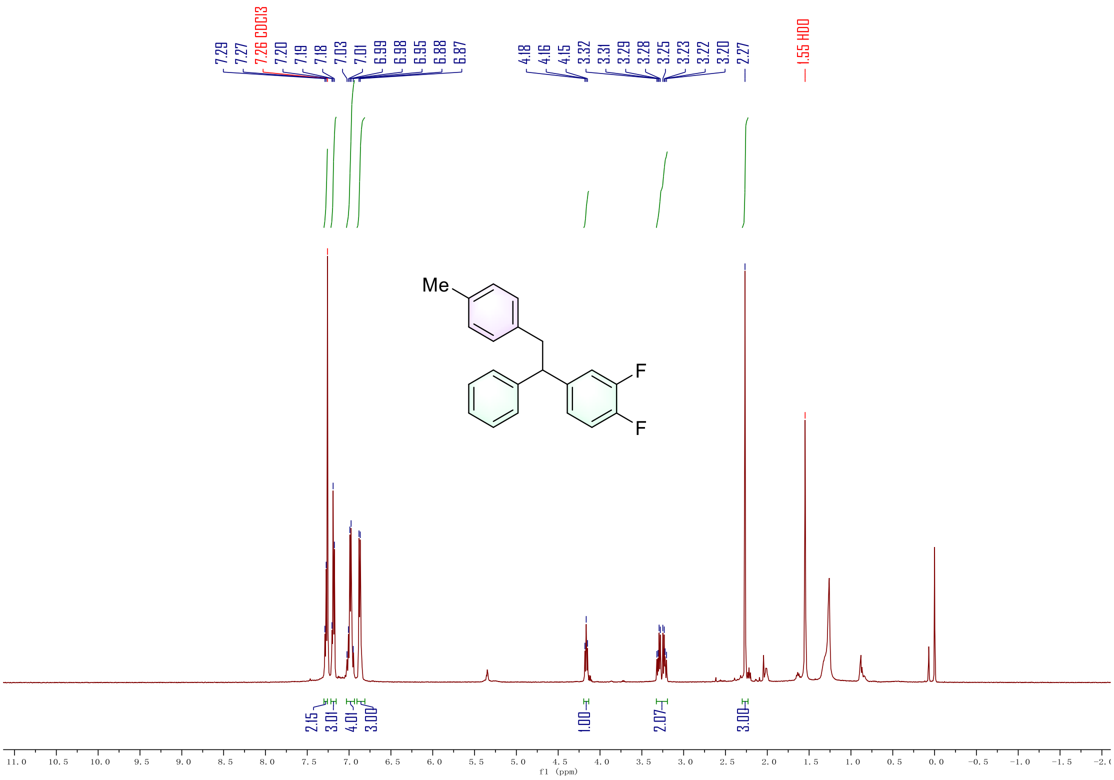
^{1}H NMR of **3o** (500 MHz, $CDCl_3$)



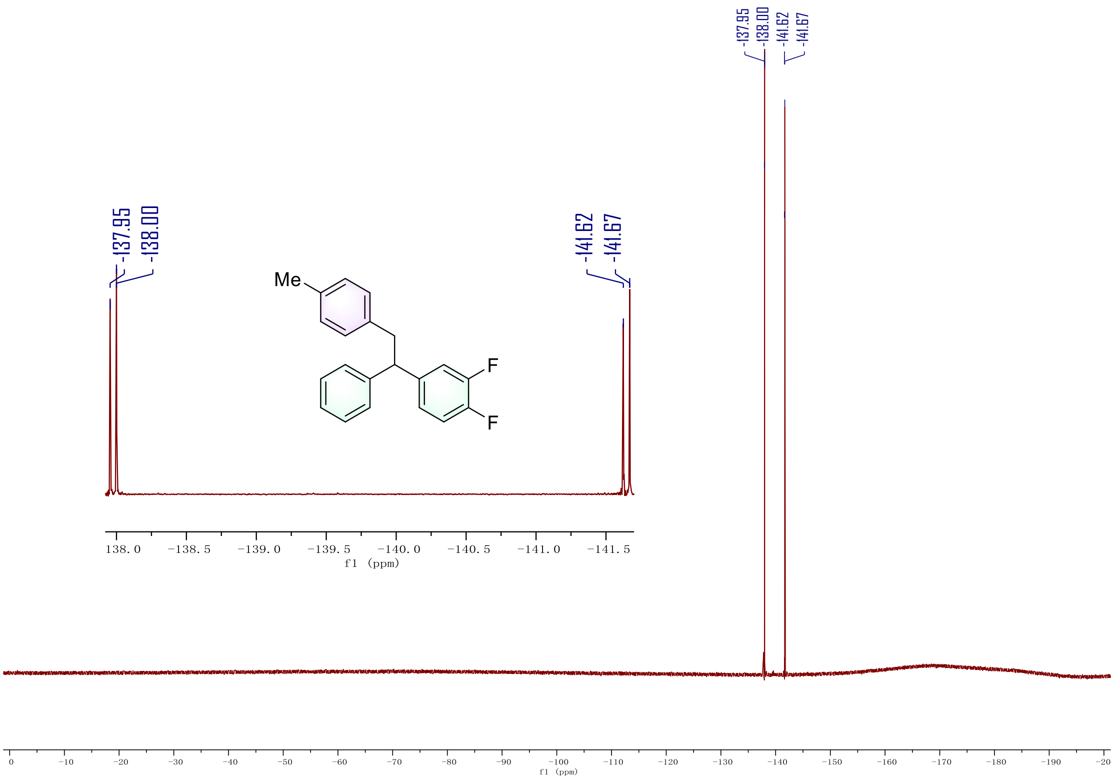
^{1}H NMR of **3p** (500 MHz, $CDCl_3$)



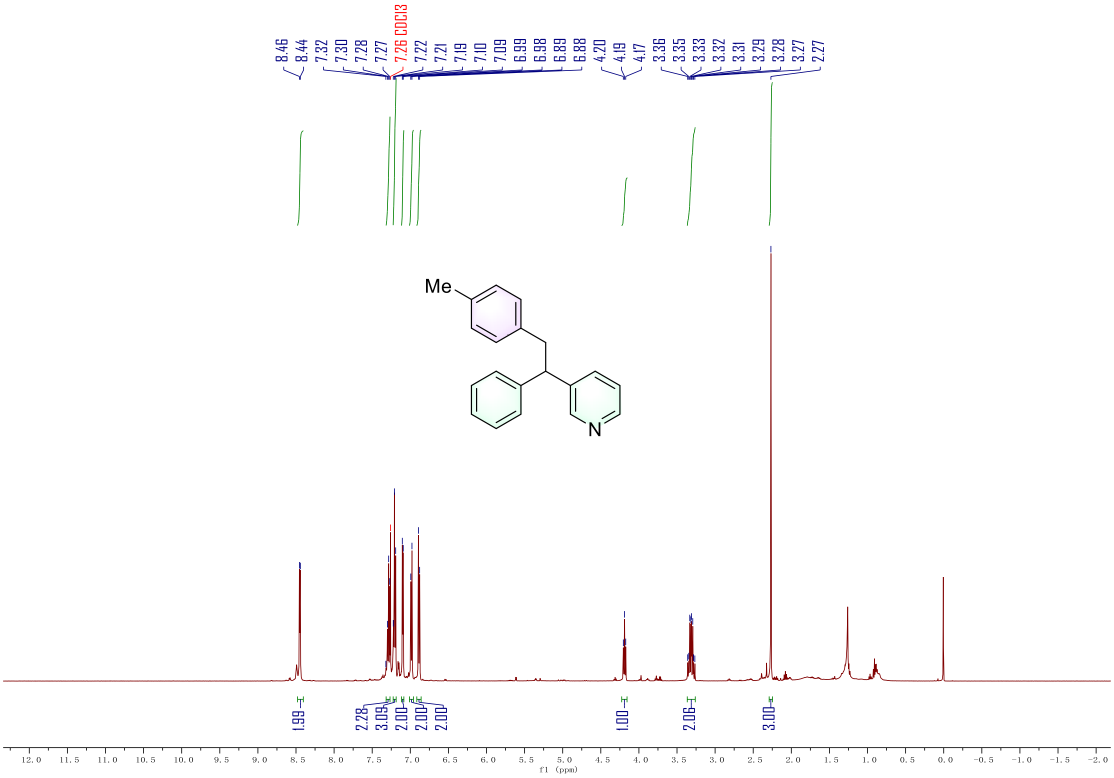
^{1}H NMR of **3q** (500 MHz, $CDCl_3$)



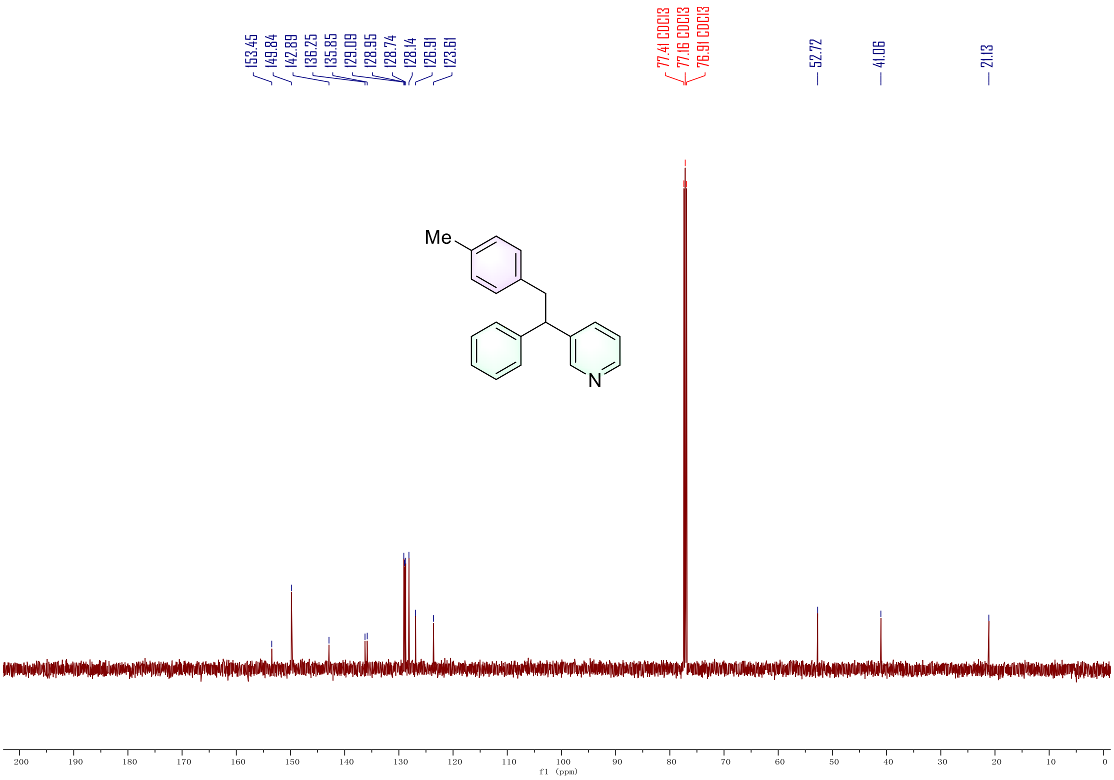
^{19}F NMR of **3q** (471 MHz, $CDCl_3$)



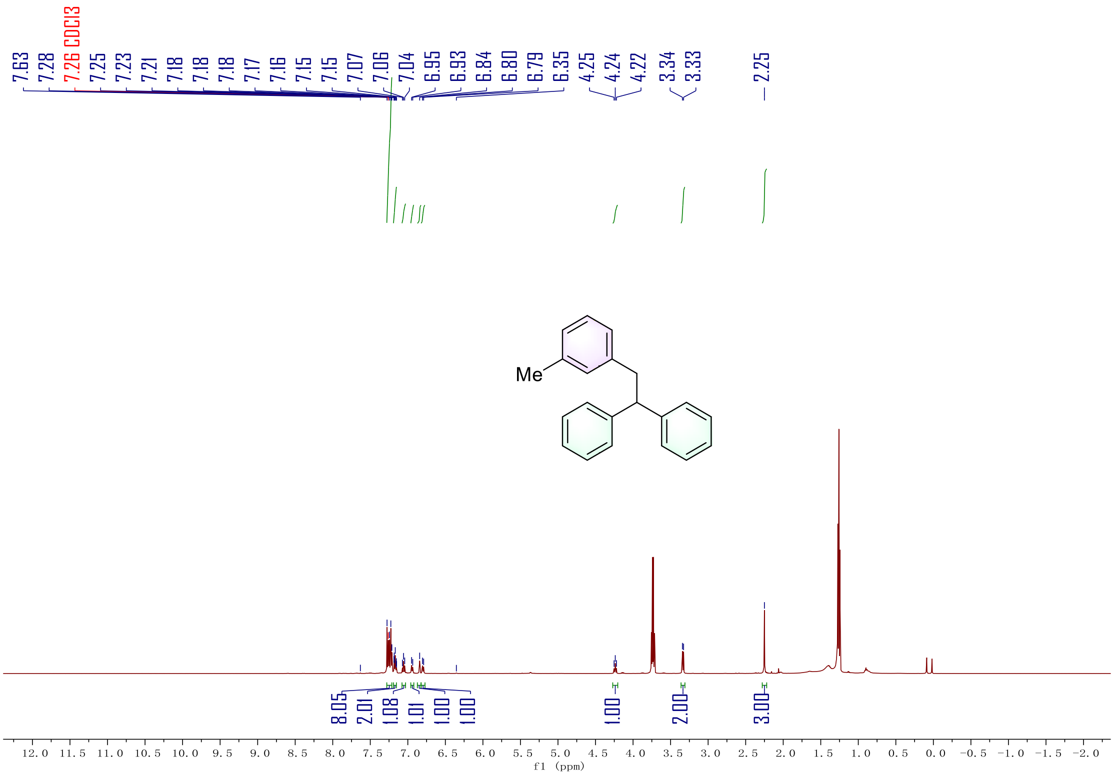
[1]H NMR of **3r** (500 MHz, $CDCl_3$)



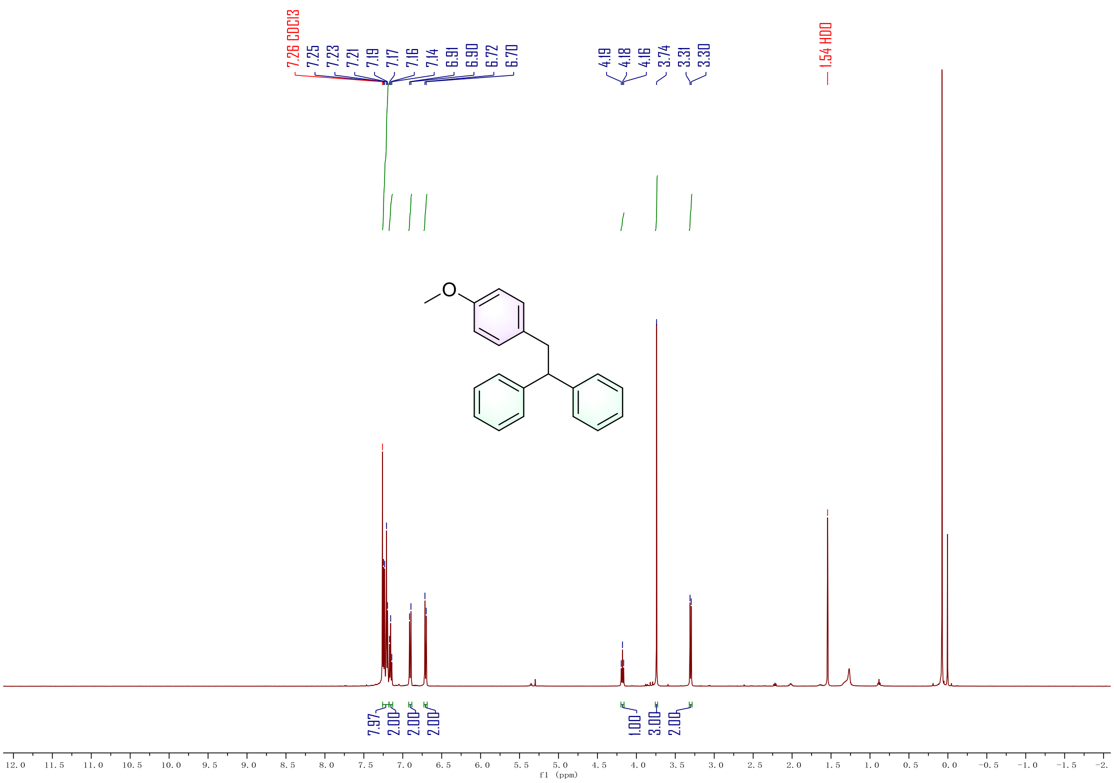
[13]C NMR of **3r** (126 MHz, $CDCl_3$)



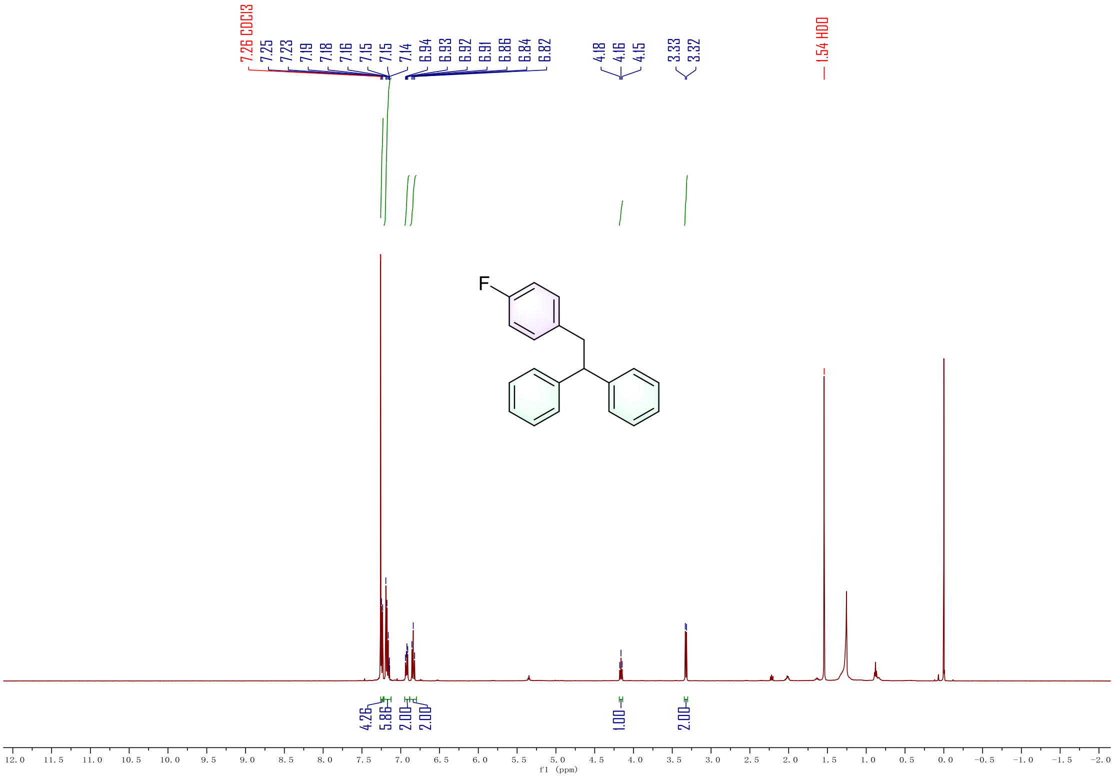
^1H NMR of **3s** (500 MHz, $CDCl_3$)



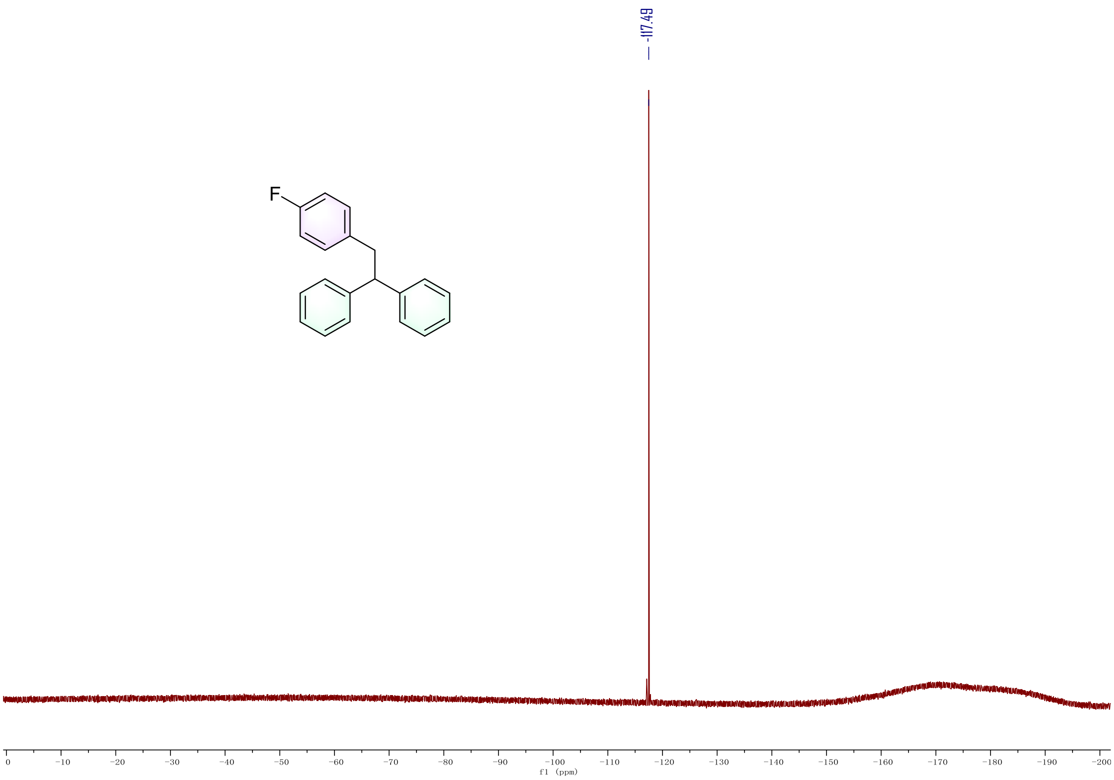
^1H NMR of **3t** (500 MHz, $CDCl_3$)



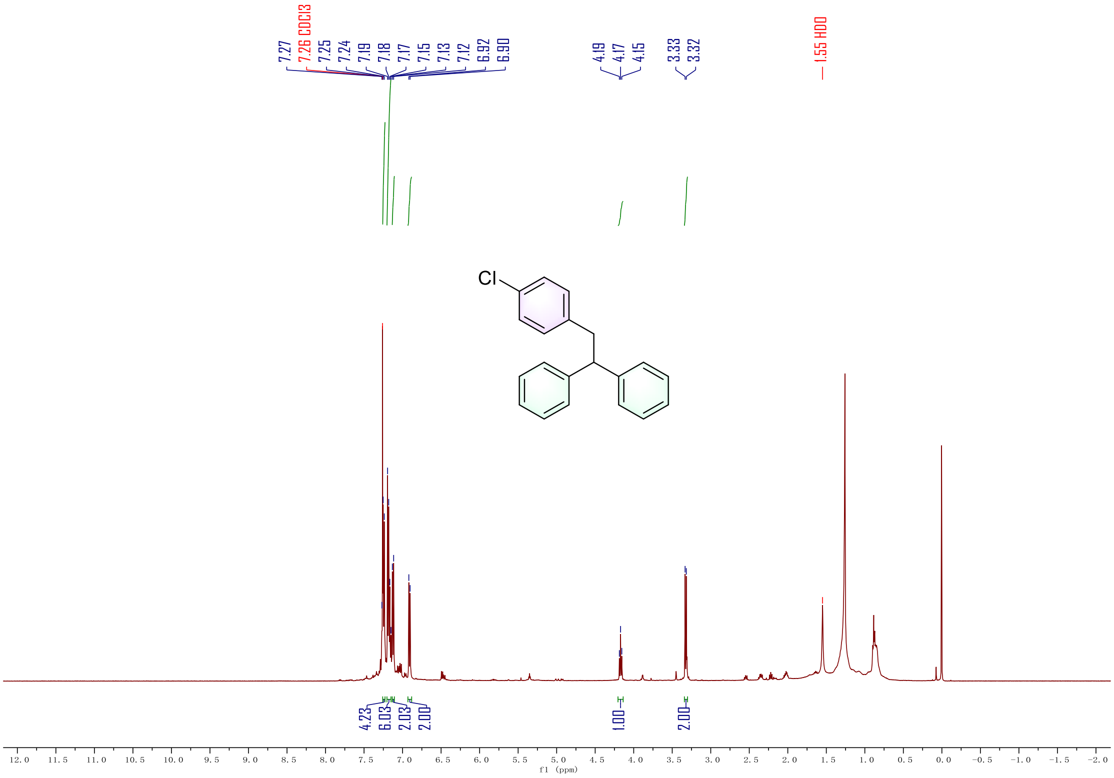
^{1}H NMR of **3u** (500 MHz, $CDCl_3$)



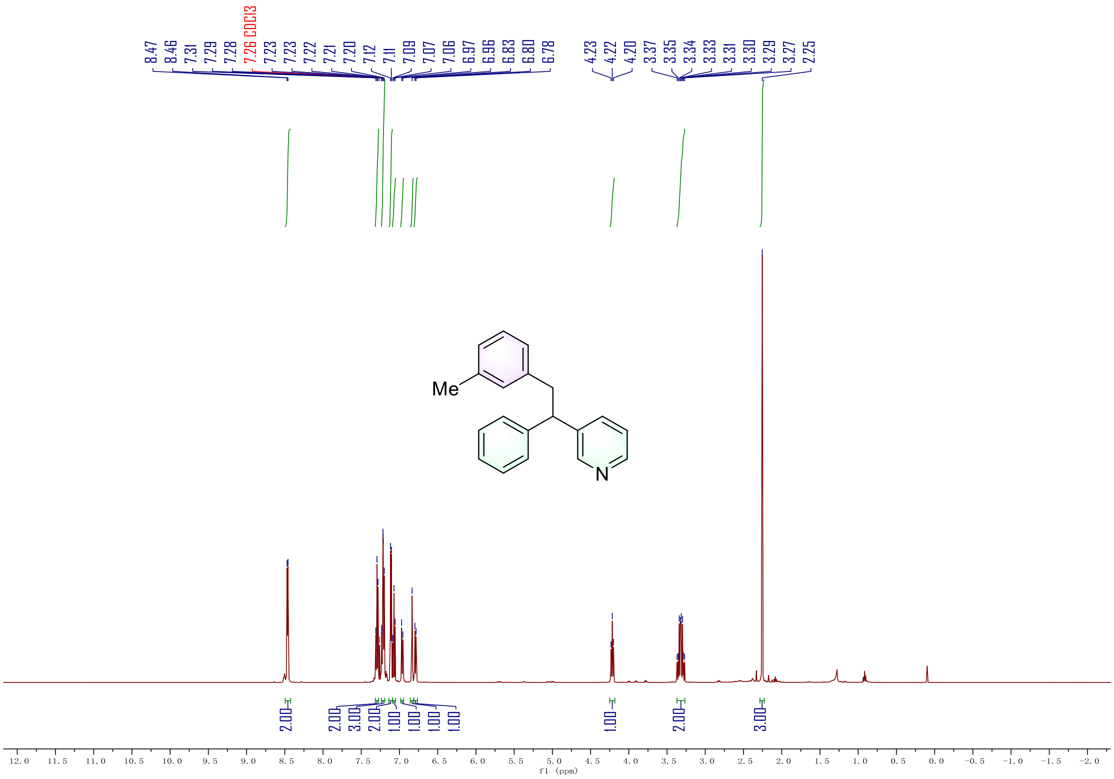
^{19}F NMR of **3u** (471 MHz, $CDCl_3$)



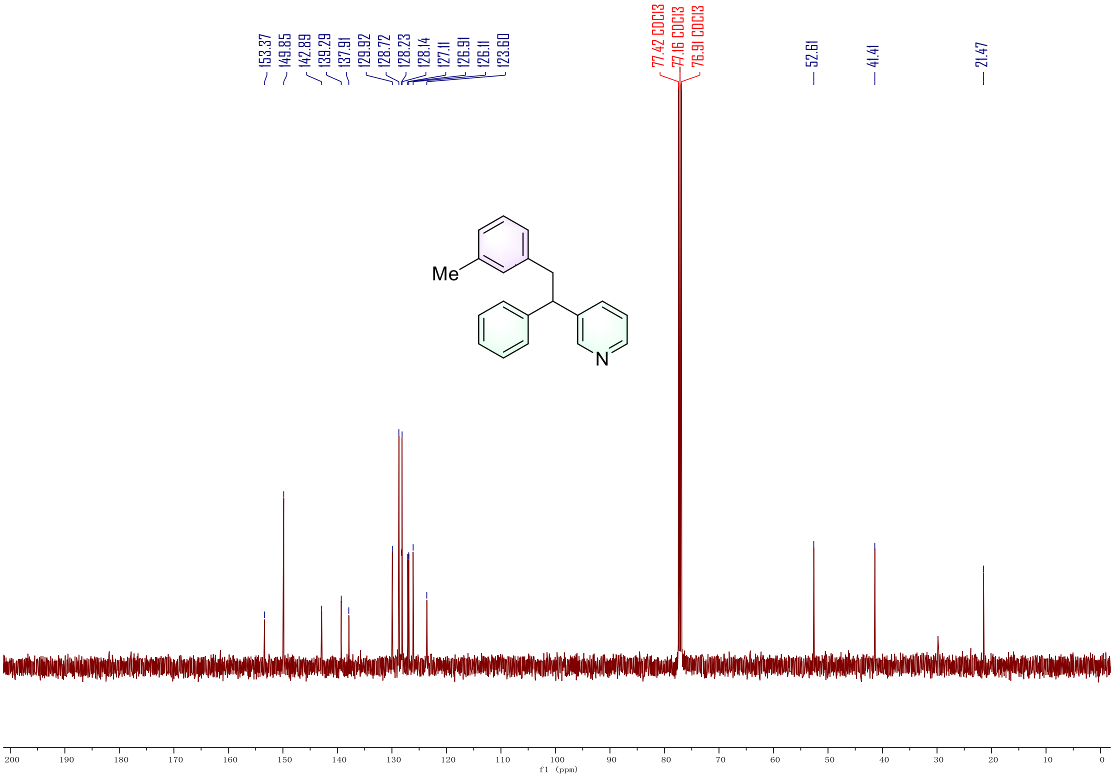
^{1}H NMR of **3v** (500 MHz, $CDCl_3$)



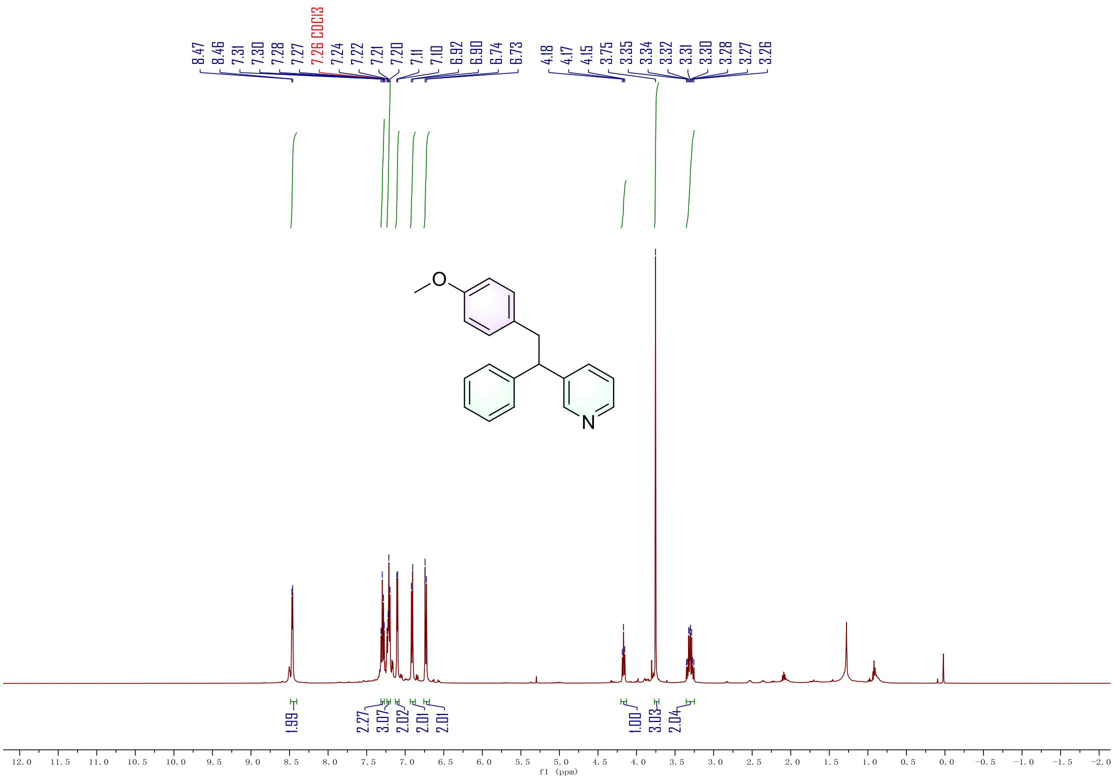
^{1}H NMR of **3w** (500 MHz, $CDCl_3$)



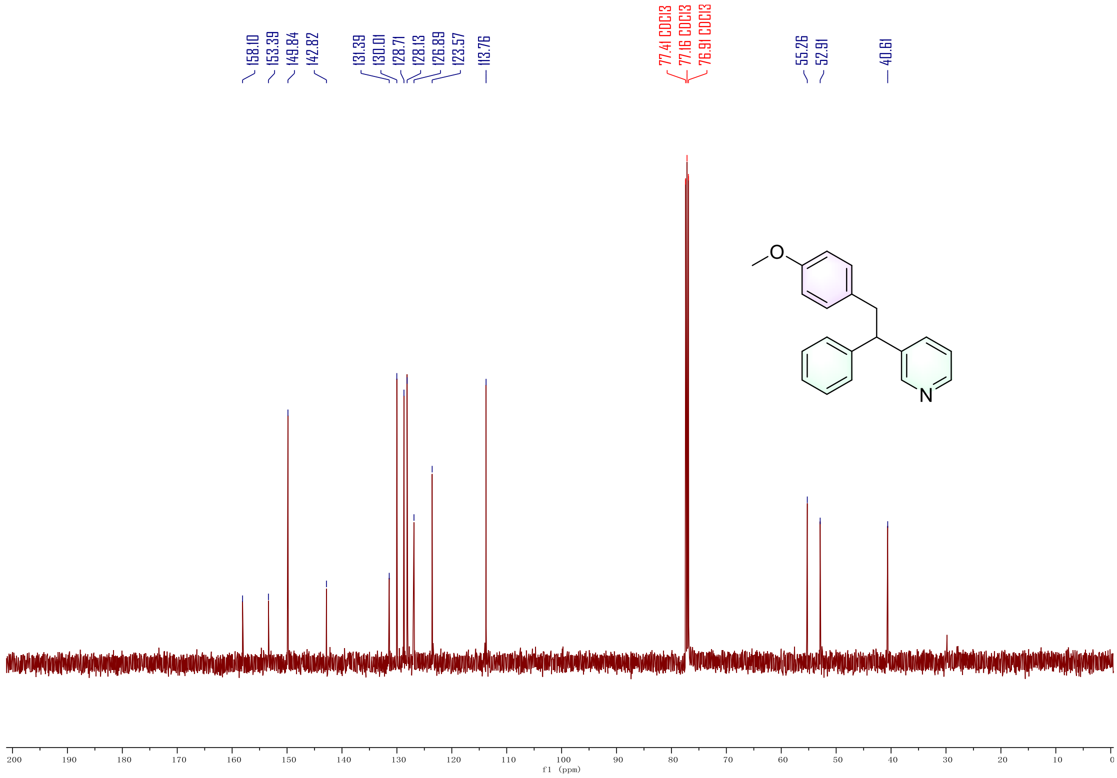
[13]C NMR of **3w** (126 MHz, $CDCl_3$)



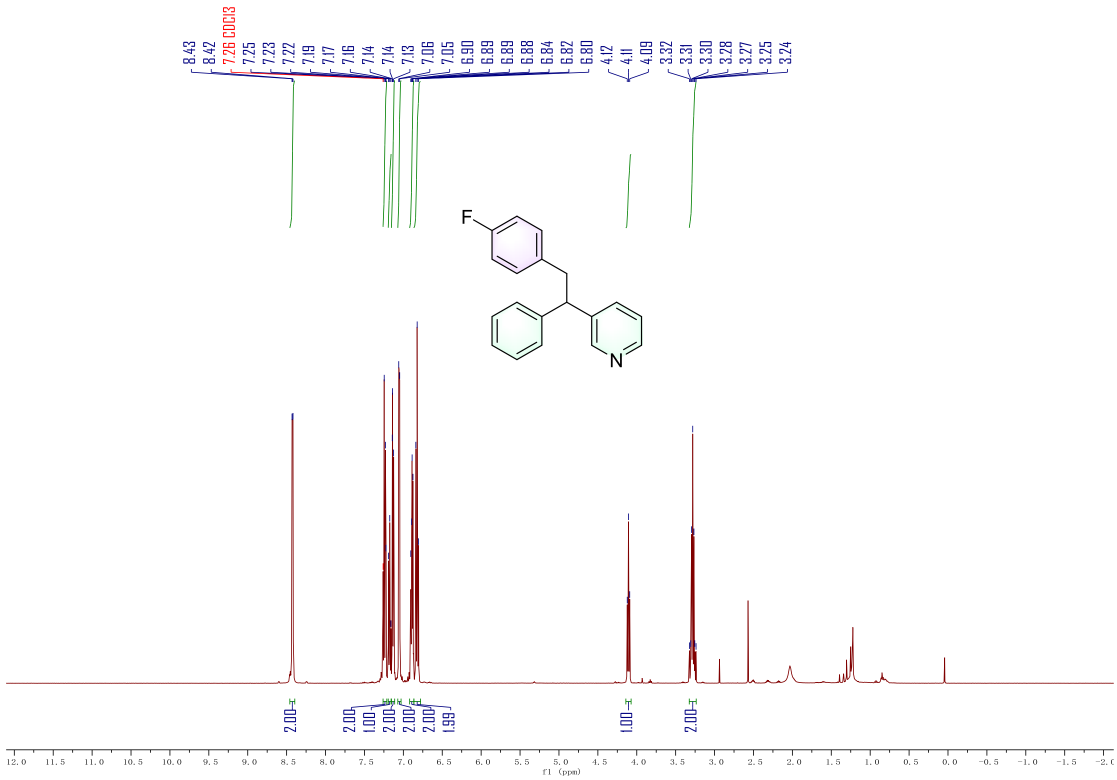
[1]H NMR of **3x** (500 MHz, $CDCl_3$)



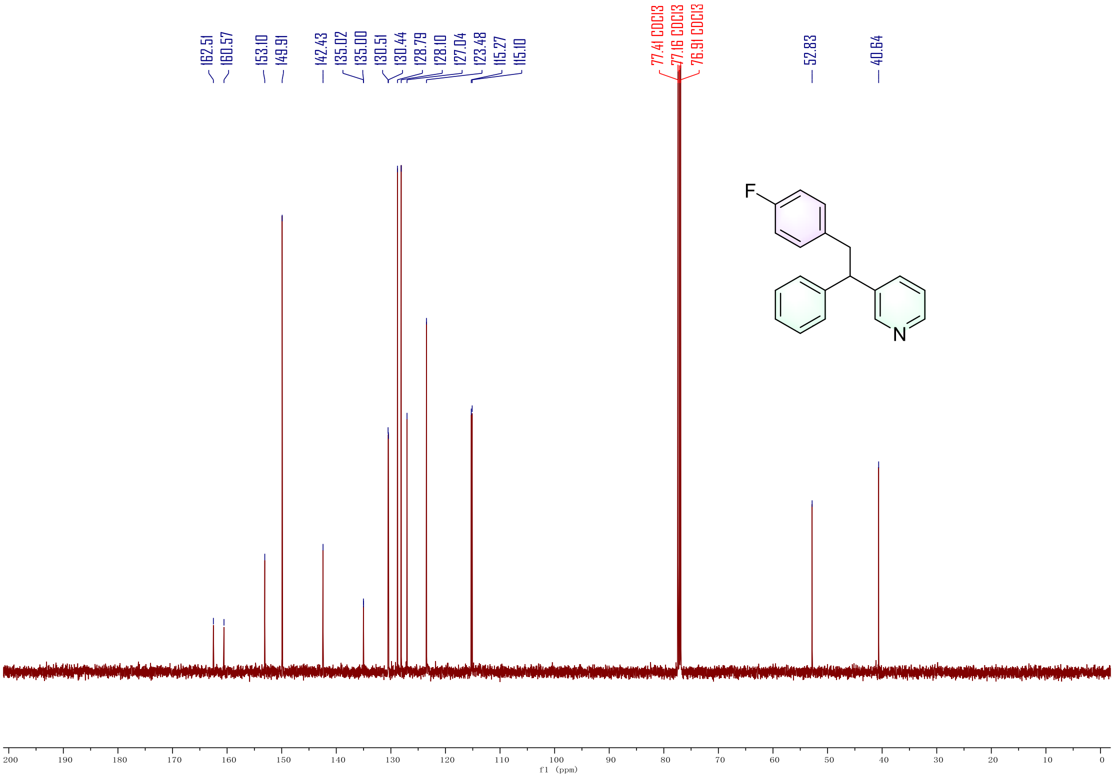
^{13}C NMR of **3x** (126 MHz, $CDCl_3$)



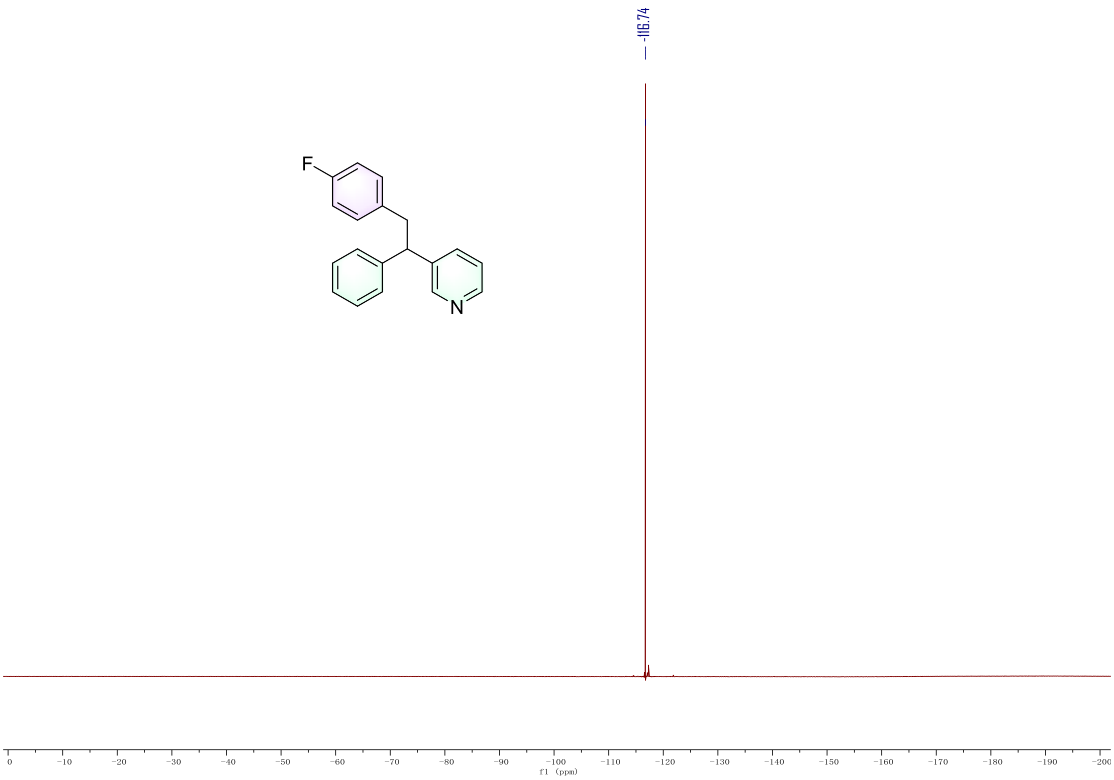
^{1}H NMR of **3y** (500 MHz, $CDCl_3$)



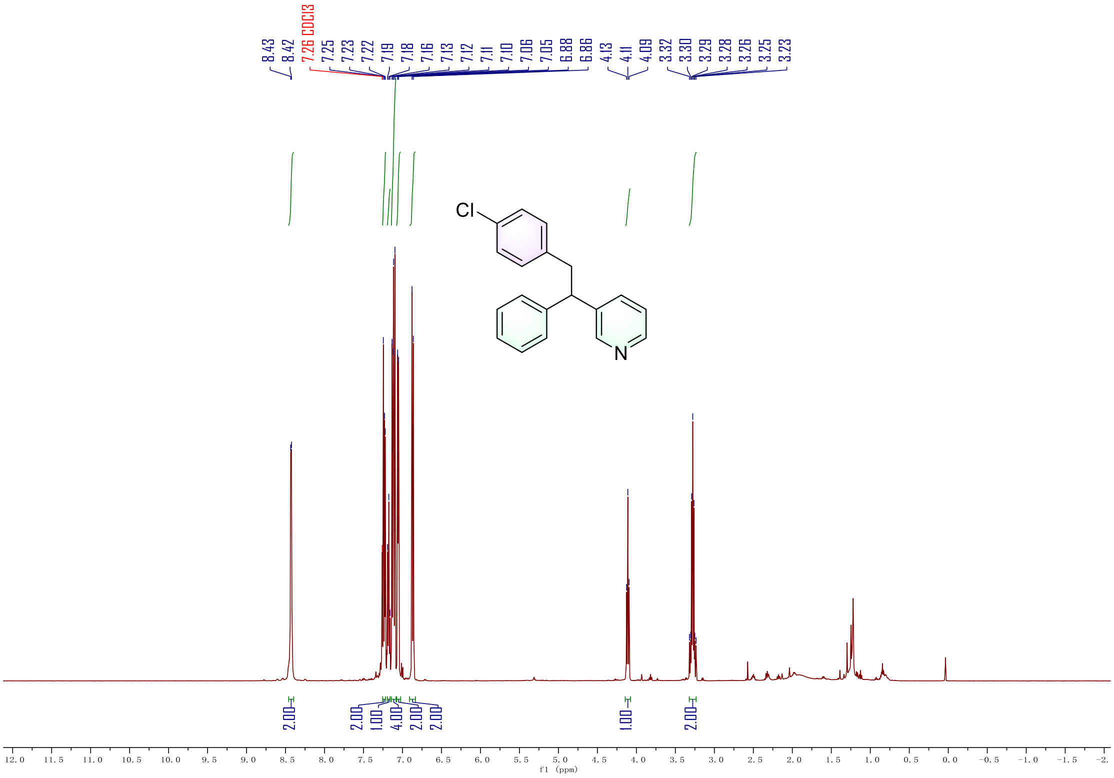
^{13}C NMR of **3y** (126 MHz, $CDCl_3$)



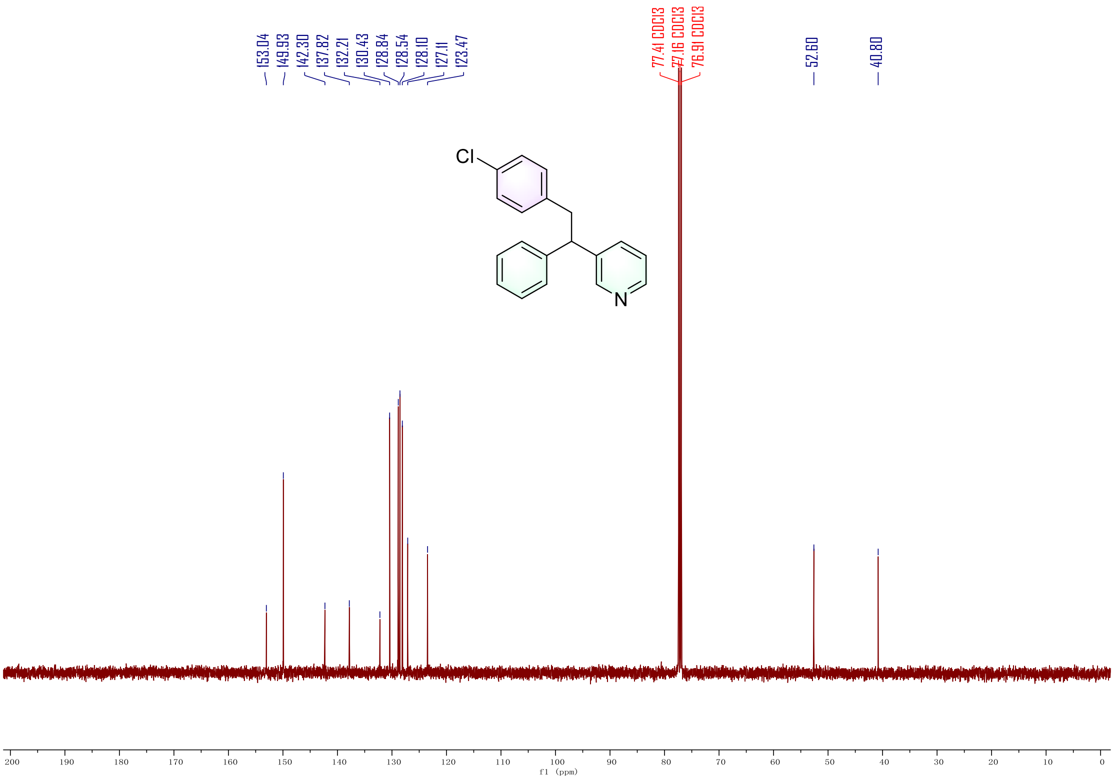
^{19}F NMR of **3y** (471 MHz, $CDCl_3$)



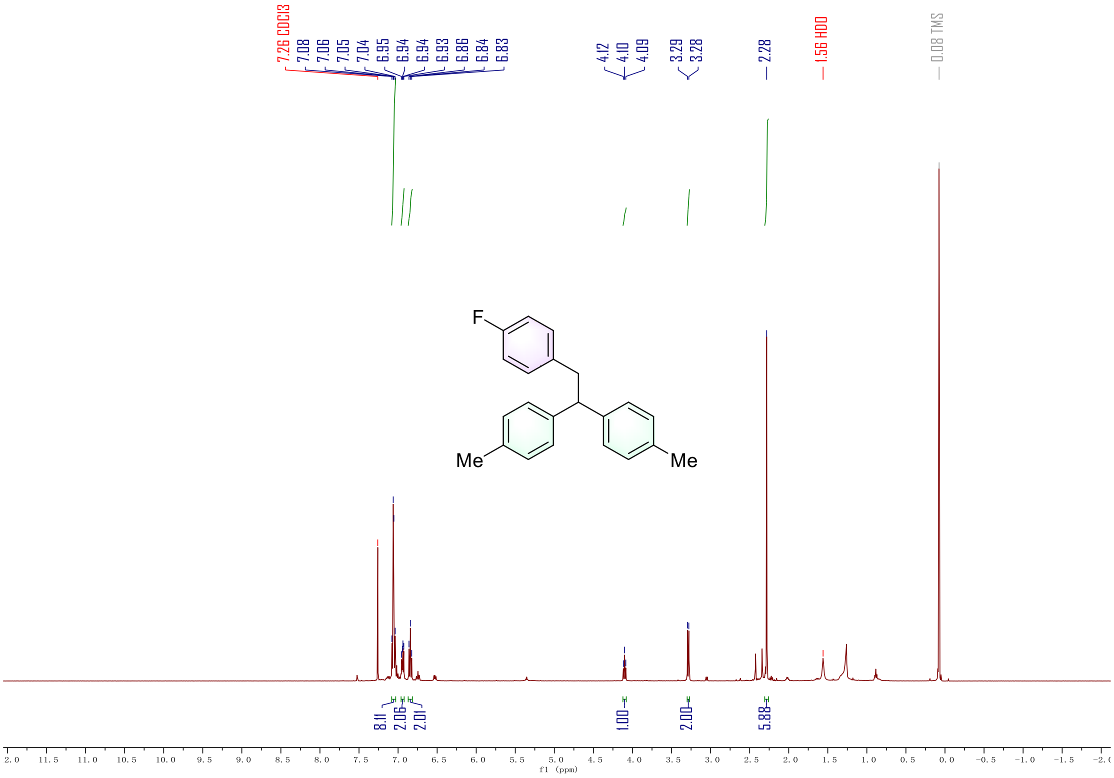
[1]H NMR of **3z** (500 MHz, $CDCl_3$)



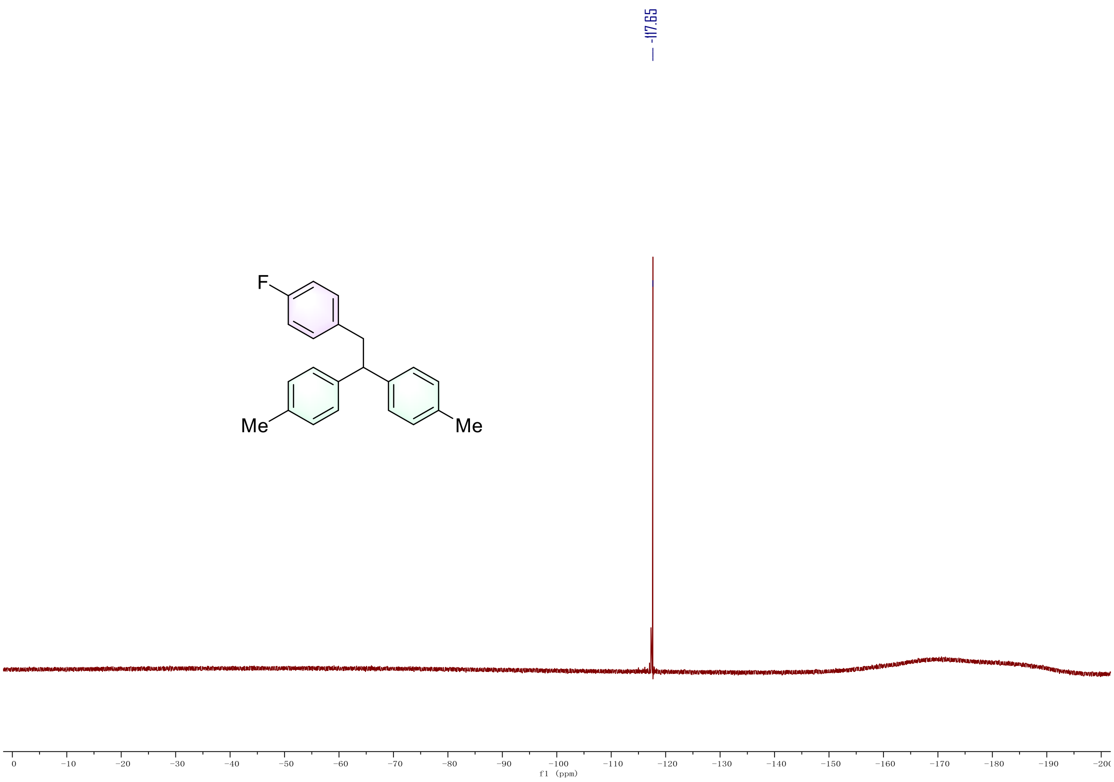
[13]C NMR of **3z** (126 MHz, $CDCl_3$)



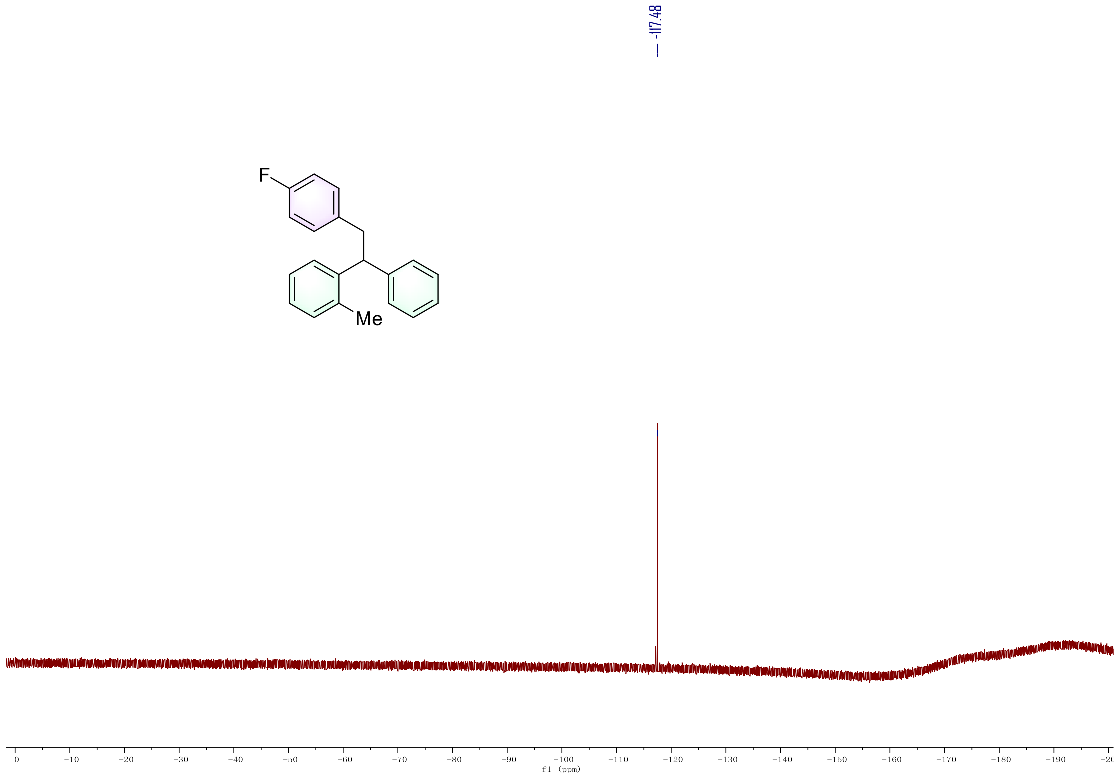
^1H NMR of **3aa** (500 MHz, $CDCl_3$)



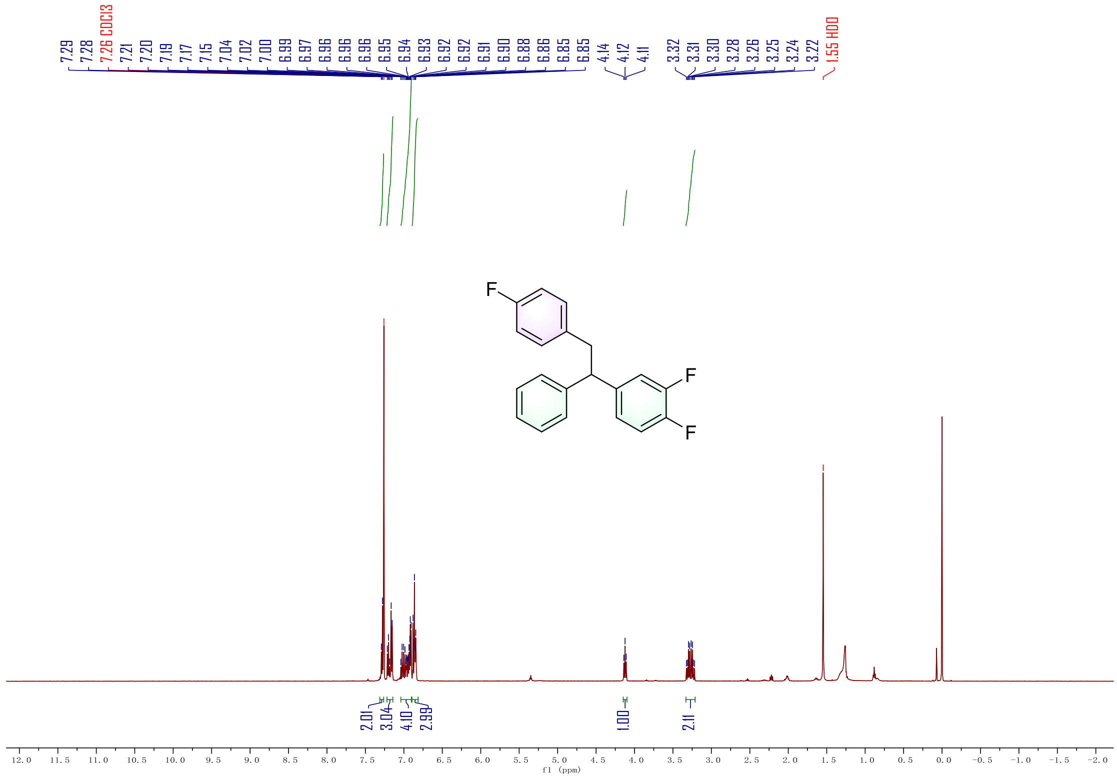
^{19}F NMR of **3aa** (471 MHz, $CDCl_3$)



^{19}F NMR of **3ab** (471 MHz, $CDCl_3$)



^{1}H NMR of **3ac** (500 MHz, $CDCl_3$)



^{19}F NMR of **3ac** (471 MHz, $CDCl_3$)