# Supporting Information

# For

# Copper-Catalyzed Deborodeuteration of Arylboronic acids/Borates Using $D_2O$ as Deuterium Source

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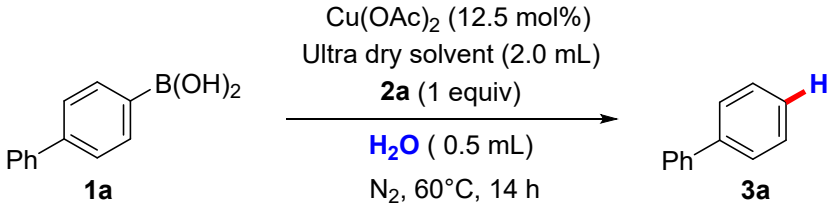
## 1. General Information

Commercially available arylboronic acids, Cu catalysts, bases, $D_2O$ and solvents were purchased from Macklin, TCI Chemicals, Sigma Aldrich, and Bide chemicals without further purification. The borate, including **1u, 1v,** and Pinacol loratadine borate **1w,** Pinacol fenofibrate borate **1x,** Cholesterol pinacol ester derivative **1y,** were prepared according to the corresponding reports.[1, 2]

All reactions were conducted in an inert nitrogen atmosphere and heated in an oil bath. Analytical thin layer chromatography (TLC) was performed using silica gel HSGF254 pre-coated plates. Flash column chromatography was performed using silica gel (200-300 mesh). $^1H$, $^{13}C$ NMR spectra were measured on Brucker Avance IIDMX 400MHz spectrometers (400 MHz for $^1H$ NMR, 101 MHz for $^{13}C$ NMR). Chemical shifts (δ) are measured by internal tetramethylsilane (TMS: 0.00 ppm) or deuterated solvent (chloroform-d: 7.26 ppm, 77.16 ppm). Abbreviations for signal couplings are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet and br, broad. Coupling constants (*J*) were taken from the spectra directly and are uncorrected. Melting points are uncorrected. High resolution mass spectra (HRMS) were recorded on a Waters TOFMS GCT Premier using ESI ionization.

## 2. Reaction Optimization

**Table S1: Screening of solvent**

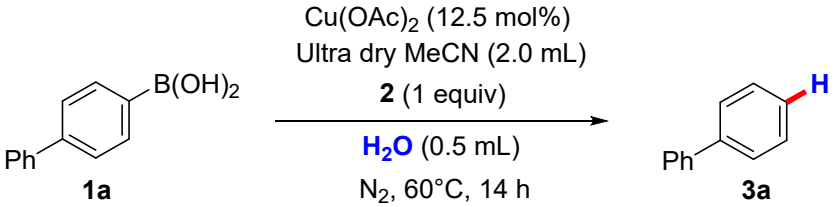



**1a** **3a**

| Entry | Ultra dry Solvent | Yield (%) |
|---|---|---|
| 1 | MeCN | 96 |
| 2 | DMSO | 72 |
| 3 | DMF | 61 |
| 4 | DCE | 48 |
| 5 | EA | 60 |
| 6 | THF | 33 |
| 7 | toluene | 73 |

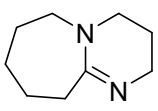
General conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (12.5 mol%), **2a** (0.2 mmol), $H_2O$ (0.5 mL), and Ultra dry solvents (2.0 mL), under a $N_2$ atmosphere with the temperature of 60 ℃. All yields were isolated yields.

**Table S2: Screening of base**

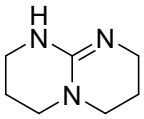



**1a** **3a**

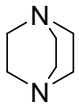
| Entry | **2** | Yield (%) |
|---|---|---|
| 1 | **2a** | 96 |
| 2 | **2b** | 69 |
| 3 | **2c** | 27 |
| 4 | **2d** | 58 |
| 5 | **2e** | 71 |
| 6 | **2f** | 86 |
| 7 | **2g** | 78 |



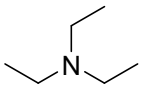
**2a**



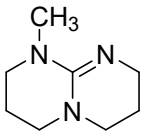
**2b**



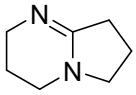
**2c**



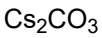
**2d**



**2e**



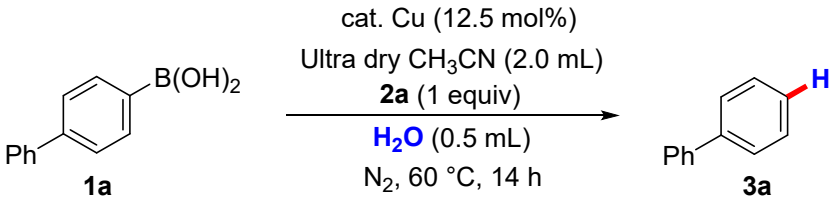
**2f**



**2g**

General conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (12.5 mol%), **2** (0.2 mmol), $H_2O$ (0.5 mL), and Ultra dry MeCN (2.0 mL), under a $N_2$ atmosphere with the temperature of 60°C. All yields were isolated yields.

**Table S3: Screening of Cu catalyst**

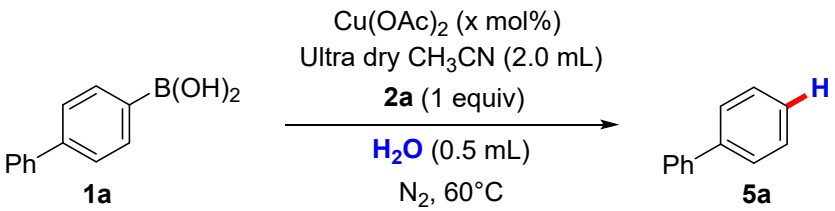



**1a** **3a**

| Entry | Cu | Yield (%) |
|---|---|---|
| 1 | $Cu(OAc)_2$ | 96 |
| 2 | $Cu(OTf)_2$ | 87 |
| 3 | $CuCl_2$ | 81 |
| 4 | CuCl | 82 |
| 5 | $CuSO_4$ | 88 |

General conditions: **1a** (0.2 mmol), Cu catalyst (12.5 mol%), **2a** (0.2 mmol), $H_2O$ (0.5 mL), and Ultra dry MeCN (2.0 mL), under a $N_2$ atmosphere with the temperature of 60 °C. All yields were isolated yields.

**Table S4: Screening of the amount of $Cu(OAc)_2$**



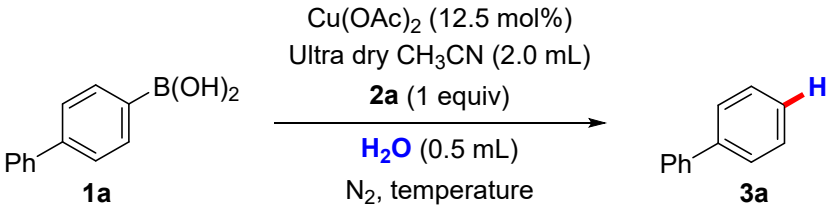

**1a** **5a**

| Entry | x | Yield (%) |
|---|---|---|
| 1 | 5 | 66 |
| 2 | 7.5 | 86 |
| 3 | 10 | 92 |

| | | |
|---|---|---|
| 4 | 12.5 | 96 |
| 5 | 20 | 67 |

General conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (x mol%), **2a** (0.2 mmol), $H_2O$ (0.5 mL), and Ultra dry MeCN (2.0 mL), under a $N_2$ atmosphere with the temperature of 60 °C. All yields were isolated yields.

**Table S5: Screening of the temperature**




| Entry | Temperature (°C) | Yield (%) |
|---|---|---|
| 1 | 20 | 44 |
| 2 | 40 | 69 |
| 3 | 60 | 96 |
| 4 | 80 | 96 |

General conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (12.5 mol%), **2a** (0.2 mmol), $H_2O$ (0.5 mL), and Ultra dry MeCN (2.0 mL), under a $N_2$ atmosphere with the temperature described above. All yields were isolated yields.

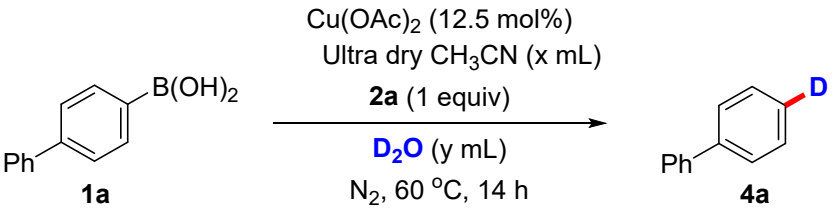
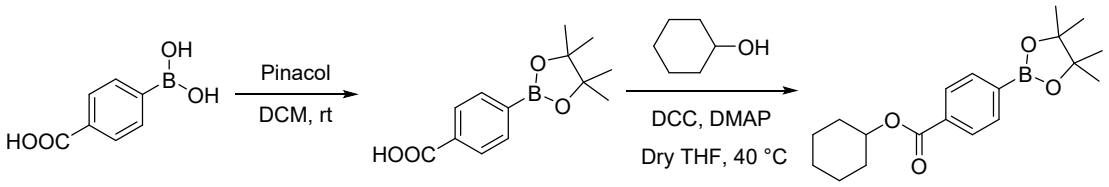



**Table S6: Screening of the amount of $D_2O$**

| Entry | x | y | Deuterium radio(%) |
|---|---|---|---|
| 1[a] | 2.0 | 0.5 | 63 |
| 2[b] | 1.0 | 1 | 94 |

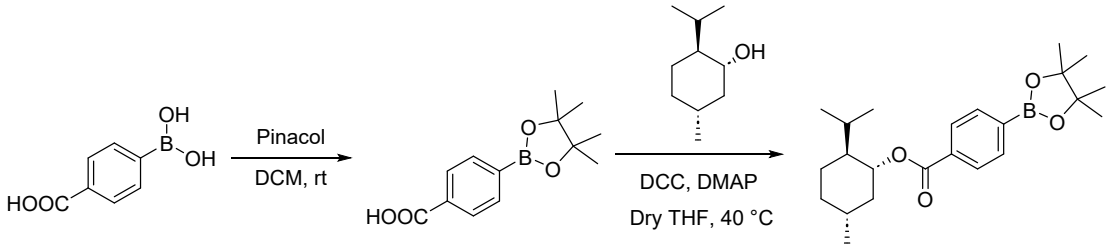
[a]General conditions: **1a** (0.2 mmol), $Cu(OAc)_2$ (12.5 mol%), **2a** (0.2 mmol), $D_2O$ (y mL), and Ultra dry MeCN (x mL), under a $N_2$ atmosphere with the temperature of 60 °C. All yields were isolated yields.

## 3. General procedure for synthesis of arylborates



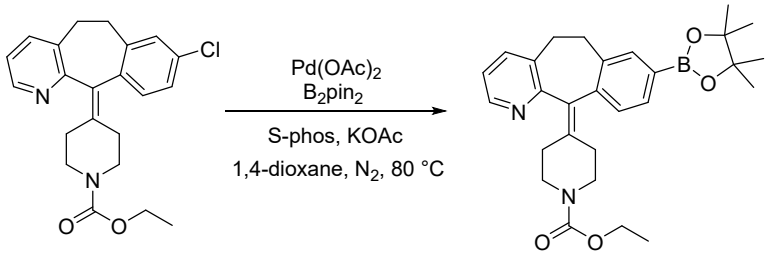

4-boronobenzoic acid (3 mmol), pinacol (3.3 mmol) were dispersed in 20 mL DCM. The reaction mixture was stirred at room temperature for 1.5 h. Then, the reaction solution was concentrated in vacuum to get the product.

Under nitrogen, a 20-mL vial was charged with boric ester (1.0 mmol), cyclohexanol (1.2 mmol), DCC (1.5 mmol), DMAP (0.20 mmol) and dry THF (4 mL). The vial was sealed with a Teflon-lined cap and stirred at 40 °C overnight. The reaction was quenched with water (10 mL) and extracted with ethyl acetate (20 mL × 3). The organic layer was condensed, and the residue was purified by flash column chromatography (hexane/ethyl acetate = 30/1) to afford the **1u** as white solid.[1]

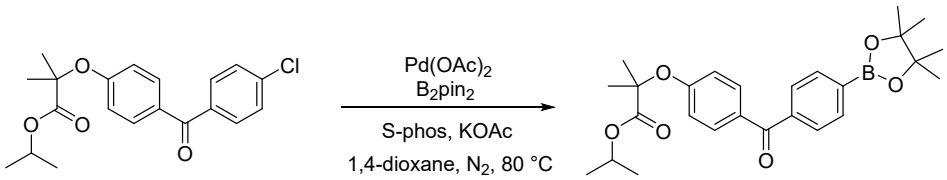



4-boronobenzoic acid (3 mmol), pinacol (3.3 mmol) were dispersed in 40 mL DCM. The reaction mixture was stirred at room temperature for 1.5h. Then, the reaction solution was concentrated in vacuum to get the product.

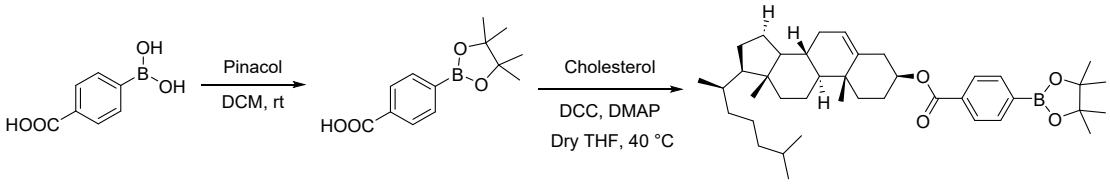
Under nitrogen, a 20-mL vial was charged with Acid (1.0 mmol), piperitol (1.2 mmol), DCC (1.5 mmol), DMAP (0.20 mmol) and dry THF (4 mL). The vial was sealed with a Teflon-lined cap and stirred at 40 °C overnight. The reaction was quenched with water (20 mL) and extracted with ethyl acetate (20 mL × 3). The organic layer was condensed, and the residue was purified by flash column chromatography (hexane/ethyl acetate = 30/1) to afford the **1v** as white solid.[1]



A mixture of Loratadine (1 mmol, 383 mg), bis(pinacolato)diboron ($B_2pin_2$, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under $N_2$ atmosphere at 80 ℃. After the reaction, the reaction mixture was diluted with $H_2O$ and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over $Na_2SO_4$, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate = 1/1 as eluent to get **1w**.[2]

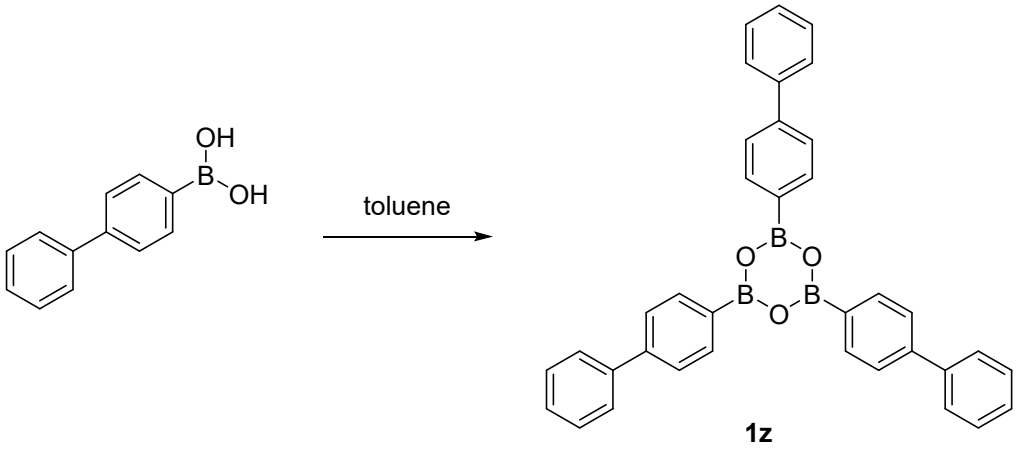



A mixture of Fenofibrate (1 mmol, 361 mg), bis(pinacolato)diboron ($B_2pin_2$, 3 mmol, 762 mg), $Pd(OAc)_2$ (4 mol%, 9 mg), 2- dicyclohexylphosphino-2',6'-dimethoxybiphenyl (S-Phos, 10 mol%, 41 mg), KOAc (3 mol, 295 mg) was stirred in 1,4-dioxane (4 mL) under $N_2$ atmosphere at 80 ℃. After the reaction, the reaction mixture was diluted with $H_2O$ and extracted with ethyl acetate (×3). The combined organics were washed with brine, dried over $Na_2SO_4$, and concentrated under reduced pressure. The resulting crude mixture was chromatographed on silica gel using petroleum ether/ethyl acetate = 10/1 as eluent to get **1x**.[2]




4-boronobenzoic acid (3 mmol), pinacol (3.3 mmol) were dispersed in 40 mL DCM. The reaction mixture was stirred at room temperature for 1.5 h. Then, the reaction solution was concentrated in vacuum to get the product.

Under nitrogen, a 20-mL vial was charged with Acid (1.0 mmol), Cholesterol (1.2 mmol), DCC (1.5 mmol), DMAP (0.20 mmol) and dry THF (4 mL). The vial was sealed with a Teflon-lined cap and stirred at 40 °C for 1.5 d. The reaction was quenched with water (20 mL) and extracted with ethyl acetate (20 mL × 3). The organic layer was condensed, and the residue was purified by flash column chromatography (hexane/ethyl acetate = 40/1) to afford the **1y** as white solid.[1]




**1z**

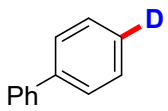
A solution of [1,1'-biphenyl]-4-ylboronic acid (3 mmol) in toluene (30 mL) was refluxed for 2 h with a Dean–Stark trap. The resulting solution was filtered. The solid obtained was the corresponding arylboroxine **1z** as a white solid.

## 4. Experimental procedures and characterization data

### 4.1 General procedure for the arylboronic acid deborylation deuteration

A mixture of boric acid **1** (0.2 mmol, 1.0 equiv), $Cu(OAc)_2$ (4.6 mg, 0.025 mmol, 12.5 mol %) was added to a 10 mL reaction tube, vacuum through nitrogen cycle three times and then added Ultra dry MeCN (1.0 mL), Base **2a** (30 μL, 0.2 mmol, 1.0 equiv), dropwise added $D_2O$ (1.0 mL). The mixture was then stirred rapidly in a 60 ℃ oil bath for 5-24 h. The reaction mixture was quenched with 10 mL 1M $NaHCO_3$ solution, then extracted with DCM (3×20 mL). The combined organic solution was washed with brine (30 mL), dried over anhydrous $Na_2SO_4$, then concentrated in vacuum. The crude mixture was purified by silica gel chromatography to get the product.

### 4.2 Product Characterization




**1,1'-biphenyl-4-*d* (4a).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

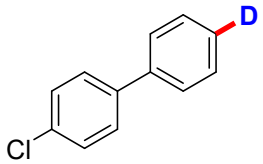
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (29.9 mg, 96% (from boronic acid), 29.1 mg, 94% (from borate) or 84.2mg, 90% (from arylboroxine)).

D incorporation by $^{1}$H NMR: 94% (from boronic acid), >99% (from borate) or 99% (form arylboroxine).

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 7.7 Hz, 4H), 7.43 (m, 4H), 7.33 (t, *J* = 7.4 Hz, 1H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 141.3, 128.8, 128.7, 127.3, 127.0 (t, *J* = 25 Hz).




**4-chloro-1,1'-biphenyl-4'-*d* (4b).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

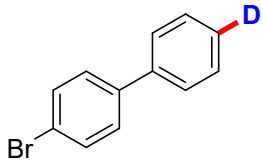
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (33.9 mg, 89%).

D incorporation by $^{1}$H NMR: 99%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.55 (M, *J* = 14.4, 8.4 Hz, 4H), 7.44 (M, *J* = 13.5, 8.2 Hz, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 140.1, 139.8, 133.5, 129.0, 128.9, 128.5, 127.4 (t, *J* = 24.5 Hz), 127.1.




**4-bromo-1,1'-biphenyl-4'-*d* (4c).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

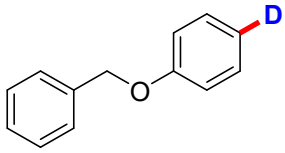
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (45.0 mg, 96%).

D incorporation by $^1$H NMR: 92%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.60 – 7.55 (m, 4H), 7.49 – 7.44 (m, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 140.3, 140.1, 132.0, 129.0, 128.9, 128.9, 127.7, 127.5, 127.2 (t, *J* = 24.7 Hz), 127.1, 121.7.




**1-(benzyloxy)benzene-4-*d* (4d).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

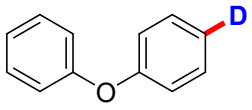
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (25.6 mg, 69%).

D incorporation by $^1$H NMR: 96%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.46 (d, *J* = 6.7 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 2H), 7.33 (m, 7.8 Hz, 3H), 7.00 (d, *J* = 8.6 Hz, 2H), 5.09 (s, 2H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 158.9, 137.2, 129.5, 128.7, 128.1, 127.6, 120.8(t, *J* = 26.3 Hz), 115.0, 70.1.




**1-phenoxybenzene-4-*d* (4e).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[4]

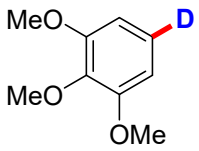
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White thick oil (20.9 mg, 61%).

D incorporation by $^1$H NMR: 97%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.37 – 7.31 (m, 4H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.02 (d, *J* = 8.8 Hz, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 157.4, 129.9, 129.8, 123.1 (t, *J* = 26.3 Hz), 119.0.




**1,2,3-trimethoxybenzene-5-*d* (4f).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

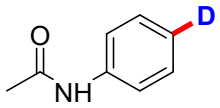
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 30:1).

Yellow liquid(14.6 mg, 43%).

D incorporation by $^{1}$H NMR: 92%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 6.58 (s, 2H), 3.86 (d, *J* = 3.7 Hz, 9H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 153.6, 138.2, 123.7, 123.4 (t, *J* = 25.3 Hz), 105.3, 105.2, 60.9, 56.1.




**N-(phenyl-4-*d*)acetamide (4g).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

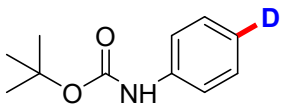
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (23.8 mg, 87%).

D incorporation by $^{1}$H NMR: 91%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.75 (s, 1H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.15 (s, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 168.8, 138.1, 129.0, 128.9, 124.1 (t, *J* = 24.3 Hz), 120.1, 24.6.




**tert-butyl (phenyl-4-*d*)carbamate (4h).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[5]

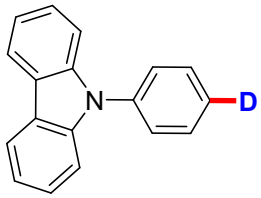
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 15:1).

White solid (27.9 mg, 72%).

D incorporation by $^1$H NMR: 93%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 6.52 (s, 1H), 1.52 (s, 9H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 152.9, 138.4, 129.0, 122.9 (t, *J* = 24.3 Hz), 118.6, 80.6, 28.5.




**9-(phenyl-4-*d*)-9H-carbazole(4i).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[6]

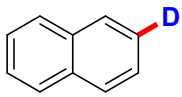
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (44.6 mg, 91%).

D incorporation by $^1$H NMR: 97%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.19 (d, *J* = 7.8 Hz, 2H), 7.67 – 7.57 (m, 4H), 7.45 (dd, *J* = 4.5, 1.3 Hz, 4H), 7.37 – 7.29 (m, 2H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 141.0, 137.9, 130.0, 129.9, 127.6, 127.3 (t, *J* = 24.3 Hz), 126.1, 123.5, 120.4, 120.0, 109.9.




**naphthalene-2-*d* (4j).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

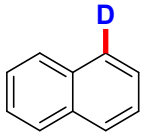
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (19.4 mg, 75%).

D incorporation by $^1$H NMR: 96%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.87 (dd, *J* = 5.8, 3.2 Hz, 4H), 7.52 – 7.48 (m, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 133.6, 128.0, 127.9, 126.0, 125.9, 125.7 (t, *J* = 24 Hz).




**naphthalene-1-*d* (4k).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

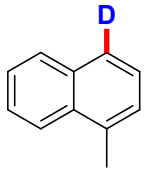
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (20.5 mg, 79%).

D incorporation by $^{1}$H NMR: 98%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.89 – 7.85 (m, 3H), 7.52 – 7.49 (m, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 133.6, 133.5, 128.3, 128.0, 127.7 (t, *J* = 24.3 Hz), 126.0, 125.8.




**1-methylnaphthalene-4-*d* (4l).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

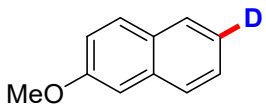
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (26.6 mg, 84%).

D incorporation by $^{1}$H NMR: 92%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.58 – 7.49 (m, 2H), 7.42 – 7.34 (m, 2H), 2.73 (s, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 134.4, 133.6, 132.8, 128.6, 126.7, 126.4 (t, *J* = 25.3 Hz), 125.8, 125.7, 125.6, 124.2, 19.5.




**2-methoxynaphthalene-6-*d* (4m).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

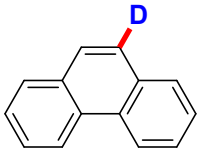
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (18.8 mg, 59%).[3]

D incorporation by $^{1}$H NMR: 94%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.80 – 7.74 (m, 3H), 7.46 (d, *J* = 8.3 Hz, 1H), 7.17 (d, *J* = 8.2 Hz, 2H), 3.94 (s, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 157.7, 134.7, 129.5, 129.1, 127.7, 126.9, 126.4, 123.4 (t, *J* = 24.7 Hz), 118.8, 105.9, 55.4.




**phenanthrene-9-*d* (4n).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

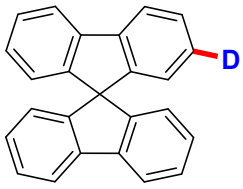
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (33.7 mg, 94%).

D incorporation by $^{1}$H NMR: 95%.

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 8.73 (d, *J* = 7.5 Hz, 2H), 7.93 (dd, *J* = 7.8, 1.6 Hz, 2H), 7.78 (s, 1H), 7.73 – 7.61 (m, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 132.2, 132.1, 130.4, 128.7, 128.6, 127.0, 126.9, 126.7, 122.8.




**9,9'-spirobi[fluorene]-2-*d* (4o).**

According to the *general procedure*, $CDCl_3$ instead of MeCN as the solvent. The spectral Data is consistent with the literature data.[3]

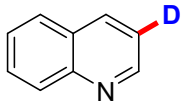
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (60.3 mg, 95%).

D incorporation by $^{1}$H NMR: 98%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 7.6 Hz, 4H), 7.43 – 7.37 (m, 4H), 7.14 (td, *J* = 7.5, 1.2 Hz, 3H), 6.80 – 6.75 (m, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 148.9, 141.9, 128.0, 127.8, 127.7, 124.2, 124.1, 120.1.




**quinoline-3-*d* (4p).**

According tothe *general procedure*. The spectral Data is consistent with the literature data.[7]

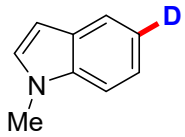
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 5:1).

Yellow liquid (16.0 mg, 61%).

D incorporation by $^1$H NMR: 95%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.94 (s, 1H), 8.20 – 8.08 (m, 2H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.72 (ddd, *J* = 8.5, 6.8, 1.7 Hz, 1H), 7.55 (dd, *J* = 8.3, 6.7 Hz, 1H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 150.5, 148.4, 136.2, 136.1, 129.6, 128.4, 127.9, 126.7, 120.9 (t, *J* = 24.3 Hz).




**1-methyl-1H-indole-5-*d* (4q).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

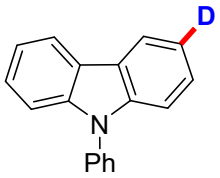
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

Yellow liquid (24.5 mg, 93%).

D incorporation by $^1$H NMR: 95%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.62 (s, 1H), 7.31 (d, *J* = 8.2 Hz, 1H), 7.20 (s, 1H), 7.03 (d, *J* = 3.1 Hz, 1H), 6.48 (d, *J* = 3.1 Hz, 1H), 3.77 (s, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 136.8, 128.9, 128.6, 121.6, 121.5, 121.0, 120.9, 119.1 (t, *J* = 25.3 Hz), 109.3, 101.0, 32.9.




**9-phenyl-9H-carbazole-3-*d* (4r).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

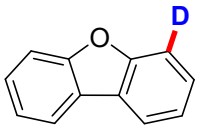
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

White solid (51.1 mg, 95%).

D incorporation by $^1$H NMR: 91%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.23 – 8.19 (m, 2H), 7.68 – 7.59 (m, 4H), 7.54 – 7.43 (m, 5H), 7.35 (ddd, *J* = 8.0, 5.1, 3.1 Hz, 1H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 141.0, 137.9, 130.0, 127.6, 127.3, 126.0, 125.9, 123.5, 120.4, 120.3, 120.0, 119.8 (t, *J* = 24.3 Hz), 109.9.




**dibenzo[b,d]furan-4-*d* (4s).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[8]

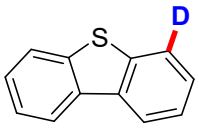
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

White solid (36.8 mg, 93%).

D incorporation by $^1$H NMR: 97%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.95 (dd, *J* = 7.7, 1.3 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 1H), 7.48 – 7.43 (m, 2H), 7.34 (t, *J* = 7.5 Hz, 2H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 127.3, 127.2, 124.4, 122.8, 120.8, 111.8, 111.6 (t, *J* = 25.3 Hz).




**dibenzo[b,d]thiophene-4-*d* (4t).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[3]

Isolated by flash column chromatography (petroleum ether: ethyl acetate = 80:1).

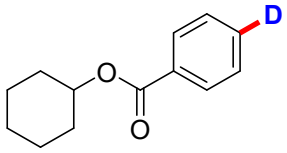
White solid (39.1 mg, >99%).

D incorporation by $^1$H NMR: 98%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.19 – 8.13 (m, 2H), 7.85 (dd, *J* = 5.7, 3.3 Hz, 1H), 7.49 – 7.43 (m, 4H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 139.6, 135.7, 126.8, 126.7, 124.5, 123.0, 121.7.

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 141.4, 128.9, 128.8, 127.4, 127.3, 127.1 (t, *J* = 24.3 Hz).




**cyclohexanol benzoate-4-*d* (4u).**

According to the *general procedure*.

Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

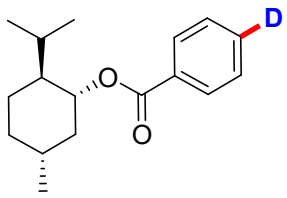
Yellow thick oil (28.3 mg, 69%).

D incorporation by $^1$H NMR: 95%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.09 (d, *J* = 7.8 Hz, 2H), 7.47 (d, *J* = 7.8 Hz, 2H), 5.07 (tt, *J* = 8.6, 3.9 Hz, 1H), 1.99 (dd, *J* = 12.3, 6.3 Hz, 2H), 1.87 – 1.79 (m, 2H), 1.71 – 1.56 (m, 4H), 1.49 – 1.39 (m, 2H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 166.1, 132.5 (t, *J* = 24.3 Hz), 131.2, 130.3, 129.7, 128.3, 127.3, 73.1, 31.8, 25.6, 23.8.

**ESI-HRMS** m/z calcd for $C_{13}H_{16}DO_2^+$ $[M+H]^+$ 206.1286, found 206.1275.




**(1R,2S,5R)-2-isopropyl-5-methylcyclohexyl benzoate-4-*d* (4v).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[9]

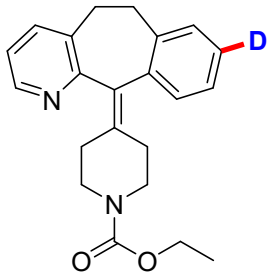
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

Yellow thick oil (27.8 mg, 53%).

D incorporation by $^1$H NMR: 92%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.14 – 8.06 (m, 2H), 7.53 – 7.44 (m, 2H), 4.98 (td, *J* = 10.9, 4.4 Hz, 1H), 2.17 – 2.16 (m, 1H), 2.01 – 1.99 (m, 1H), 1.82 – 1.74 (m, 2H), 1.69 – 1.58 (m, 2H), 1.15 – 1.10 (m, 2H), 0.97 (dd, *J* = 6.8, 4.2 Hz, 7H), 0.84 (d, *J* = 7.0 Hz, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 166.2, 132.5 (t, *J* = 24.3 Hz), 131.0, 129.7, 128.3, 75.0, 47.4, 41.1, 34.5, 31.6, 26.6, 23.8, 22.2, 20.9, 16.7.




**ethyl 4-(5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene-8-*d*)piperidine-1-carboxylate (4w).**

According to the *general procedure*.

Isolated by flash column chromatography (petroleum ether: ethyl acetate = 1:1).

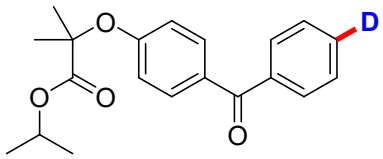
White thick oil (56.9 mg, 81%).

D incorporation by $^1$H NMR: 98%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.38 (dd, *J* = 4.8, 1.7 Hz, 1H), 7.42 (d, *J* = 6.9 Hz, 1H), 7.20 – 7.12 (m, 3H), 7.07 (dd, *J* = 7.7, 4.8 Hz, 1H), 4.12 (d, *J* = 7.4 Hz, 2H), 3.79 (s, 2H), 3.41 (d, *J* = 21.0 Hz, 2H), 3.18 – 3.07 (m, 2H), 2.83 (d, *J* = 21.0 Hz, 2H), 2.54 – 2.26 (m, 4H), 1.24 (d, *J* = 7.1 Hz, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 157.6, 155.7, 146.5, 139.4, 137.7, 137.6, 136.8, 133.79, 129.3, 129.0, 126.0, 122.2, 61.4, 45.0, 44.9, 31.9, 30.8, 30.6, 24.9, 14.8.

**ESI-HRMS** m/z calcd for $C_{22}H_{24}DN_2O_2^+$ $[M+H]^+$ 350.1973, found 350.1974.




6

**isopropyl 2-(4-(benzoyl-4-*d*)phenoxy)-2-methylpropanoate (4x).**

According to the *general procedure*. The spectral Data is consistent with the literature data.[7]

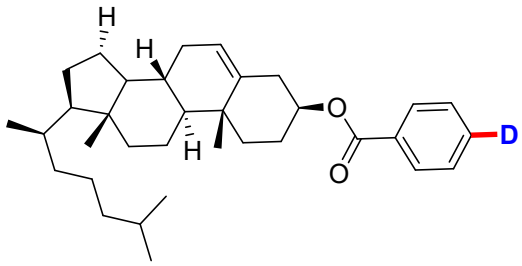
Isolated by flash column chromatography (petroleum ether: ethyl acetate = 15:1).

White solid (62.8 mg, 96%).

D incorporation by $^1$H NMR: 97%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.72 (dd, *J* = 13.1, 8.6 Hz, 4H), 7.45 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 5.09 (p, *J* = 6.2 Hz, 1H), 1.66 (s, 6H), 1.21 (d, *J* = 6.2 Hz, 6H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 194.4, 173.2, 159.9, 138.5, 136.6, 132.1, 131.3, 130.4, 128.7, 117.4, 69.5, 25.5, 21.7.




**(3S,8S,9S,10R,13R,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl benzoate-4-*d* (4y).**

According to the *general procedure*.

Isolated by flash column chromatography (petroleum ether: ethyl acetate = 40:1).

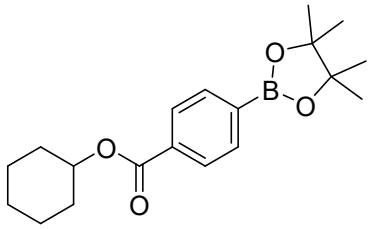
White solid (59.9 mg, 61%). Mp: 137.2 – 137.8 ℃.

D incorporation by $^1$H NMR: 90%.

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 8.1 Hz, 2H), 7.43 (dd, *J* = 8.0, 3.8 Hz, 2H), 5.42 (d, *J* = 4.2 Hz, 1H), 4.86 (dtd, *J* = 12.1, 8.4, 4.5 Hz, 1H), 2.47 (d, *J* = 8.0 Hz, 2H), 2.01 (ddt, *J* = 15.6, 11.8, 3.6 Hz, 3H), 1.94 – 1.89 (m, 1H), 1.83 (tdd, *J* = 14.8, 6.4, 3.7 Hz, 1H), 1.74 (dt, *J* = 12.0, 2.8 Hz, 1H), 1.59 – 1.05 (m, 25H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.87 (dd, *J* = 6.6, 1.8 Hz, 6H), 0.69 (s, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 129.7, 128.4, 128.3, 122.9, 74.7, 56.9, 56.3, 50.2, 42.5, 39.9, 39.7, 38.4, 37.2, 36.4, 32.1, 32.0, 28.4, 28.2, 28.0, 24.4, 24.0, 23.0, 22.7, 21.2, 19.5, 18.9.

**ESI-HRMS** m/z calcd for $C_{34}H_{50}DO_2^+$ $[M+H]^+$ 492.3946, found 492.3629.



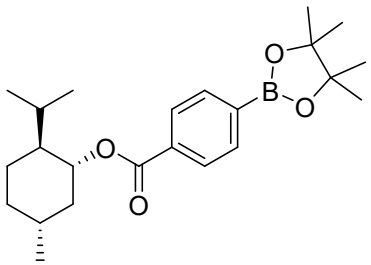
**cyclohexyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (1u).**

Yellow thick oil (201.9 mg, 61%).

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 8.04 – 7.99 (m, 2H), 7.89 – 7.82 (m, 2H), 5.03 (ddt, *J* = 12.8, 8.6, 3.8 Hz, 1H), 1.99 – 1.91 (m, 2H), 1.82 – 1.75 (m, 2H), 1.64 – 1.40 (m, 6H), 1.35 (s, 12H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 166.1, 134.7, 133.3, 128.6, 84.2, 73.3, 68.0, 31.7, 25.7, 25.6, 25.0, 23.8.

**ESI-HRMS** m/z calcd for $C_{19}H_{28}BO_4^+$ $[M+H]^+$ 331.2075, found 331.2079.

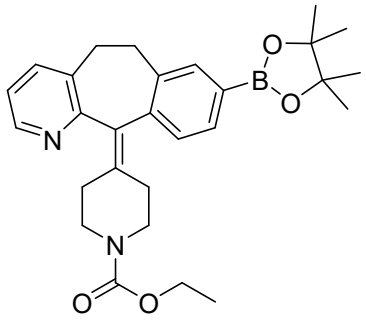


**(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (1v).**

White solid (282.5 mg, 73%).

**$^{1}$H NMR** (400 MHz, Chloroform-*d*) δ 7.98 – 7.94 (m, 2H), 7.83 – 7.79 (m, 2H), 4.88 (td, *J* = 10.9, 4.4 Hz, 1H), 2.15 – 2.11 (m, 1H), 2.10 – 1.99 (m, 1H), 1.85 – 1.83 (m, 2H), 1.63 – 1.55 (m, 2H), 1.29 (s, 12H), 1.19 – 1.16 (m, 2H), 0.96 – 0.86 (m, 7H), 0.74 (d, *J* = 1.8 Hz, 3H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 166.04, 134.8, 134.6, 133.0, 128.5, 84.1, 74.9, 47.2, 40.9, 34.6, 31.7, 26.5, 24.9, 24.8, 23.7, 22.2, 20.7, 16.6.

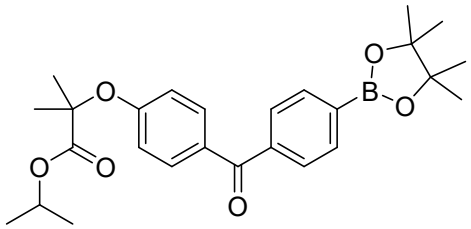


**ethyl 4-(8-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-5,6-dihydro-11H-benzo[5,6]cyclohepta[1,2-b]pyridin-11-ylidene)piperidine-1-carboxylate (1w).**

White solid (343.3 mg, 72.4%).

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.37 (d, *J* = 4.6 Hz, 1H), 7.64 – 7.57 (m, 2H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 7.5 Hz, 1H), 7.05 (dd, *J* = 7.7, 4.7 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.87 – 3.70 (m, 2H), 3.54 – 3.25 (m, 2H), 3.18 – 3.06 (m, 2H), 2.95 – 2.74 (m, 2H), 2.55 – 2.24 (m, 4H), 1.31 (s, 12H), 1.24 (t, *J* = 6.5 Hz, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 157.1, 155.6, 146.6, 142.5, 137.6, 137.1, 136.9, 135.4, 133.7, 132.6, 128.7, 122.2, 83.8, 61.3, 44.9, 31.9, 31.6, 30.8, 30.6, 24.9, 24.9, 14.7.

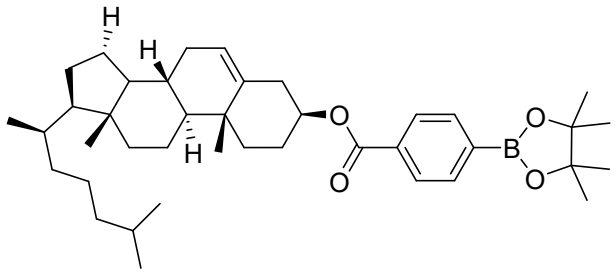


**isopropyl 2-methyl-2-(4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoyl)phenoxy)propanoate (1x).**

White solid (287.6 mg, 64%).

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.1 Hz, 2H), 7.74 (d, *J* = 8.8 Hz, 2H), 7.70 (d, *J* = 8.1 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.07 (p, *J* = 6.3 Hz, 1H), 1.64 (s, 6H), 1.35 (s, 12H), 1.19 (d, *J* = 6.3 Hz, 6H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 195.7, 173.2, 159.7, 140.4, 134.5, 132.1, 130.6, 128.7, 117.2, 84.2, 79.4, 69.4, 25.4, 24.9, 21.6.

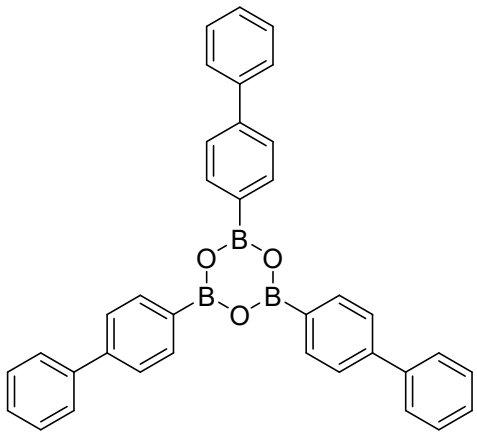


**(3S,8S,9S,10R,13R,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (1y).**

White solid (358.2 mg, 58% yield).

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.1 Hz, 2H), 7.86 (d, *J* = 8.1 Hz, 2H), 5.54 – 5.31 (m, 1H), 5.08 – 4.71 (m, 1H), 2.47 (d, *J* = 8.1 Hz, 2H), 2.10 – 1.67 (m, 6H), 1.63 – 1.43 (m, 8H), 1.36 (s, 12H), 1.24 – 0.96 (m, 14H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.87 (dd, *J* = 6.7, 1.8 Hz, 6H), 0.69 (s, 3H).

**$^{13}$C NMR** (101 MHz, $CDCl_3$) δ 166.1, 139.7, 134.6, 133.1, 128.6, 122.8, 84.2, 74.7, 56.8, 56.2, 50.1, 42.4, 39.8, 39.6, 38.3, 37.1, 36.7, 36.3, 35.9, 32.0, 32.0, 28.3, 28.1, 27.9, 24.9, 24.4, 23.9, 22.9, 22.6, 21.1, 19.5, 18.8, 11.9.



**2,4,6-tri([1,1'-biphenyl]-4-yl)-1,3,5,2,4,6-trioxatriborinane (1z)**

White solid (1442.3 mg, 89% yield);

**$^1$H NMR** (400 MHz, Chloroform-*d*) δ 8.34 (d, *J* = 7.9 Hz, 6H), 7.85 – 7.73 (m, 6H), 7.73 – 7.60 (m, 6H), 7.51 (t, *J* = 7.6 Hz, 3H), 7.42 (t, *J* = 7.3 Hz, 1H).

**$^{13}$C NMR** (101 MHz, Chloroform-*d*) δ 145.4, 141.0, 136.4, 129.0, 128.0, 127.5, 126.9.

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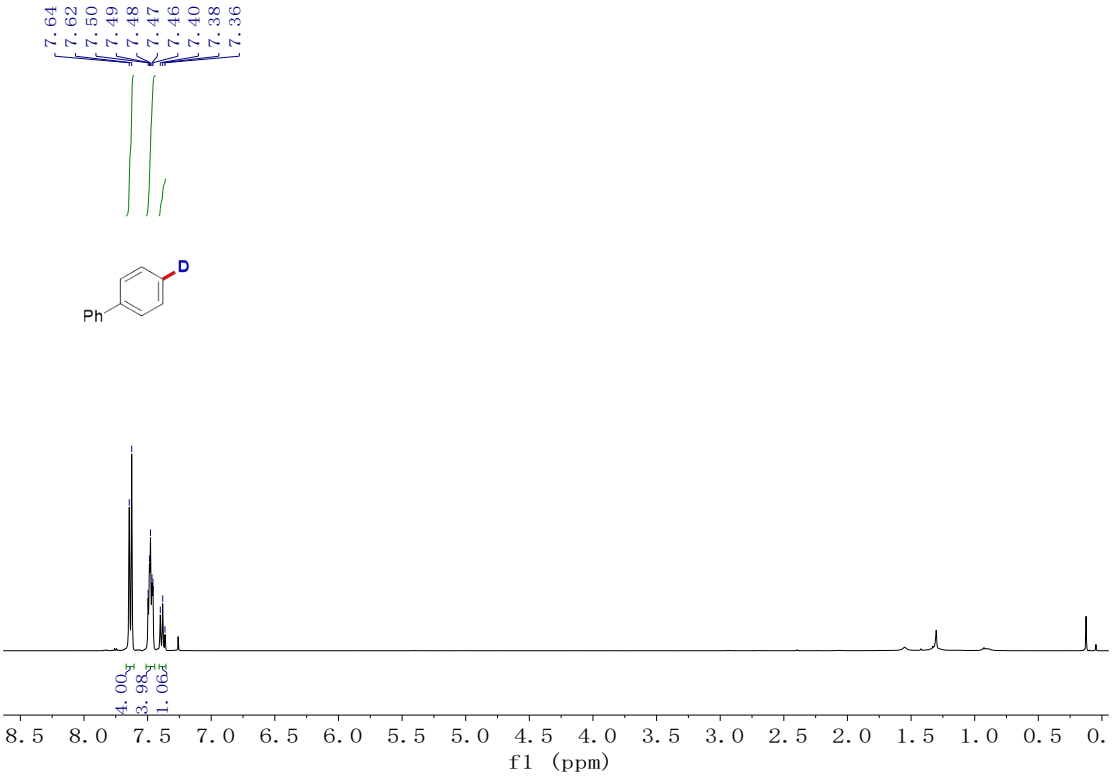
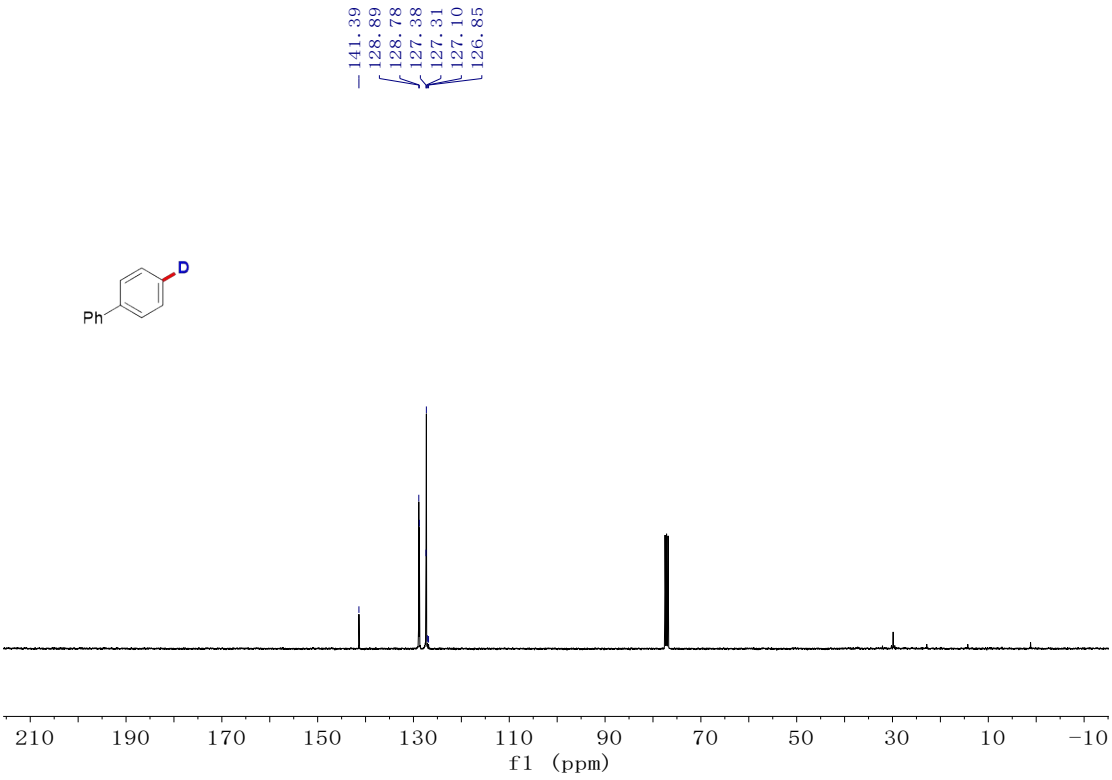
## NMR Spectra

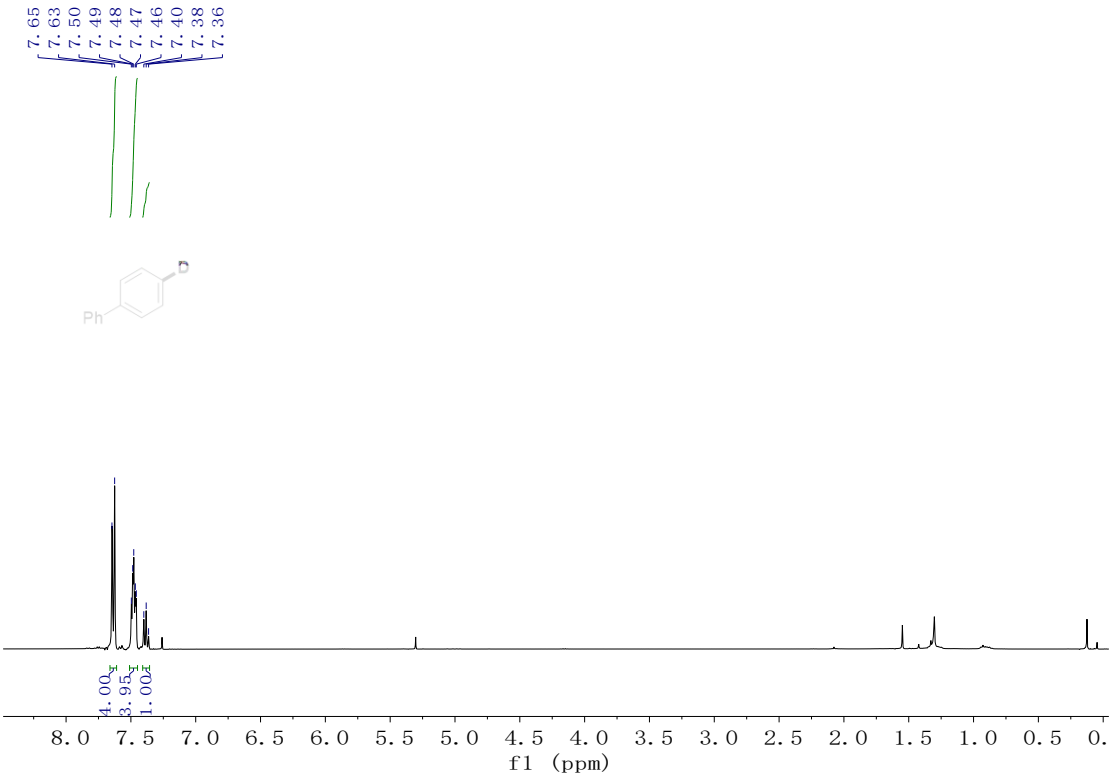
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4a** (from boronic acid)



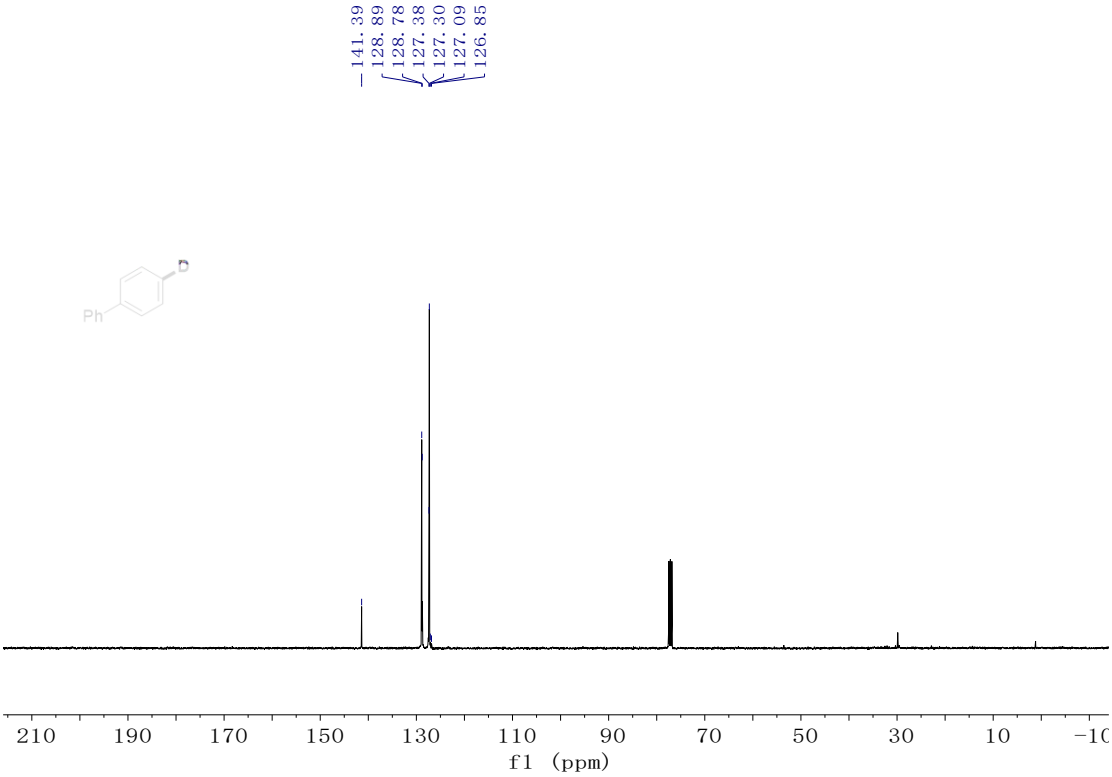

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4a** (from boronic acid)



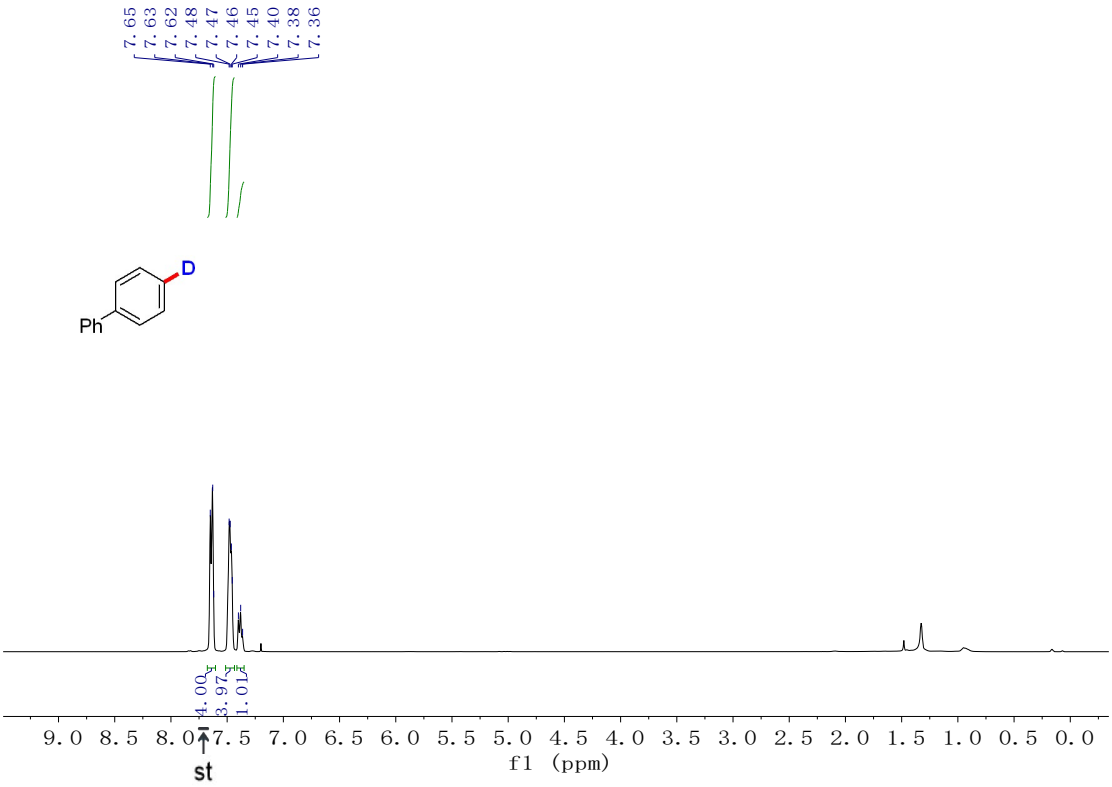
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4a** (from boronic borate)



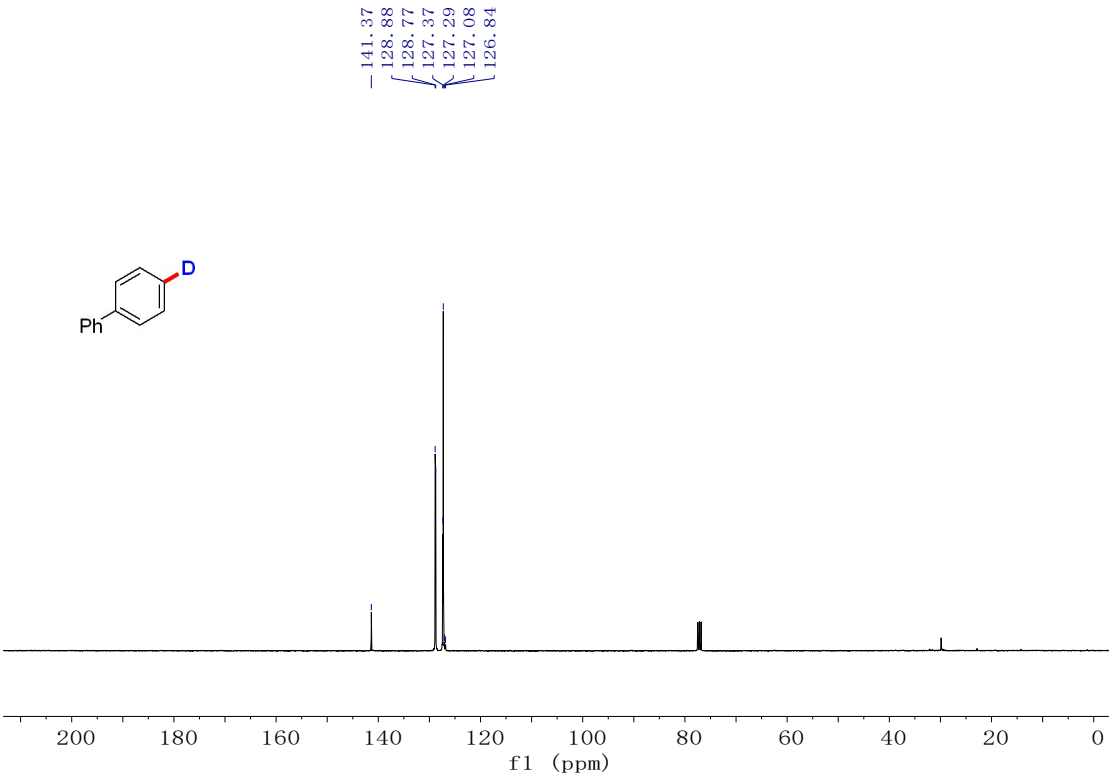

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4a** (from boronic borate)



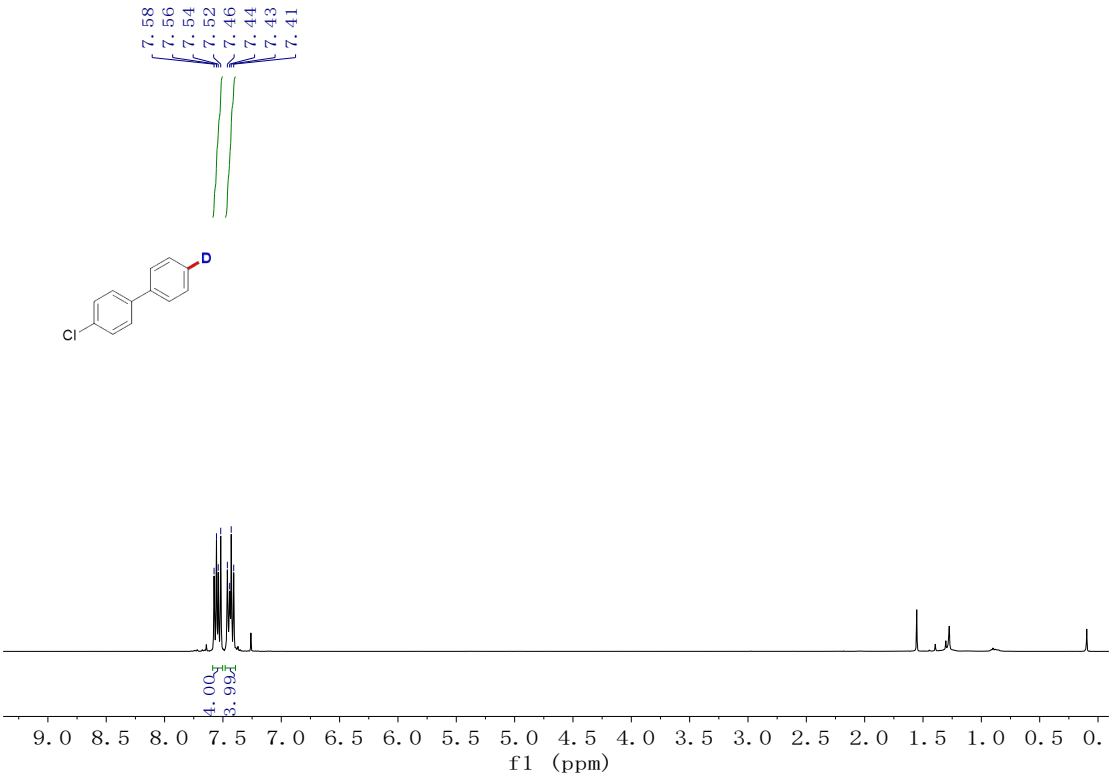
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4a** (from arylboroxine)



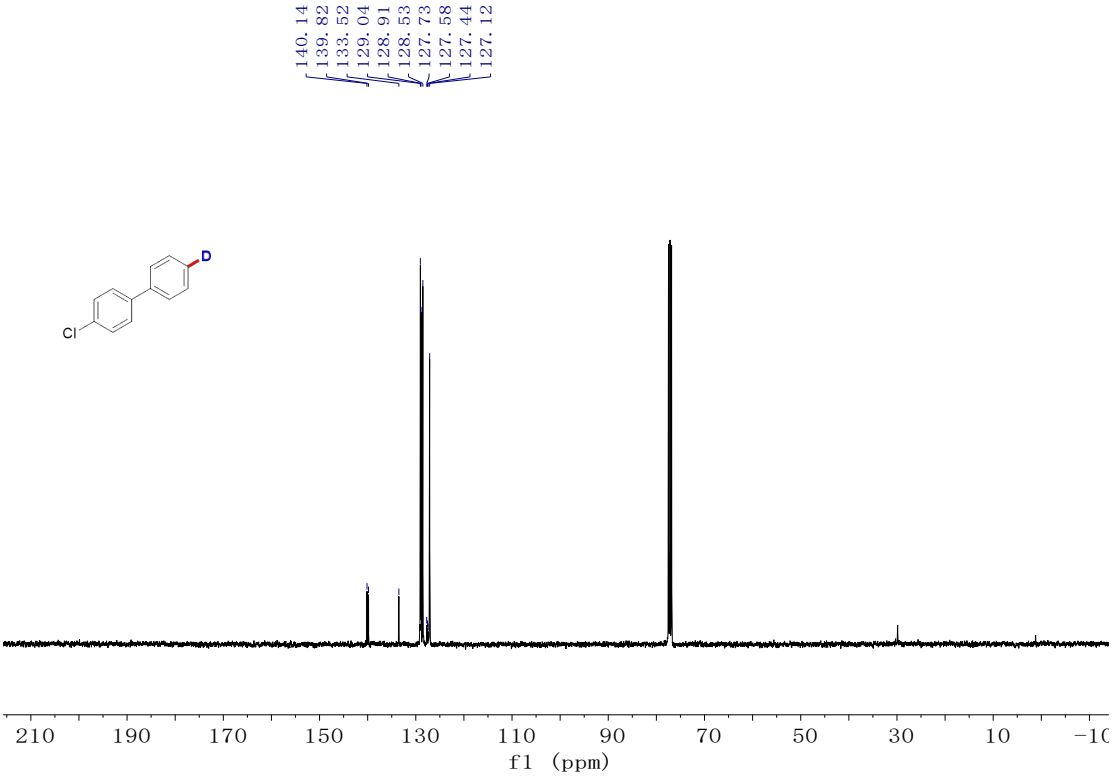

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4a** (from arylboroxine)



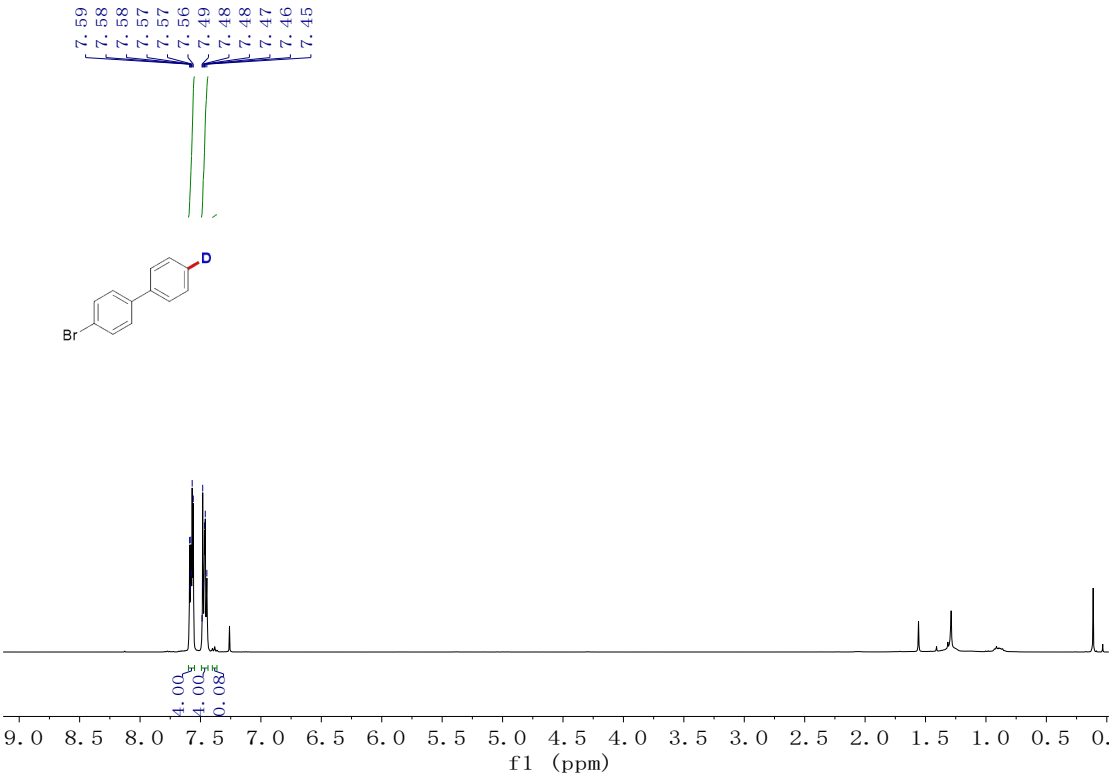
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4b**



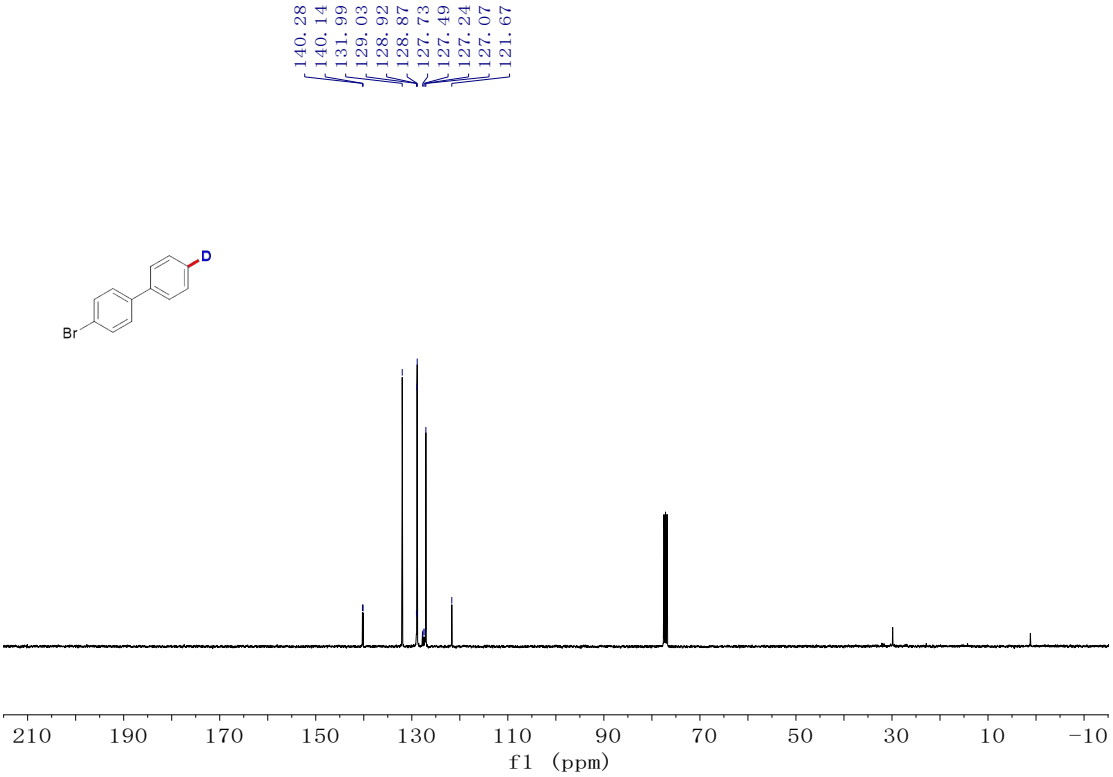

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4b**



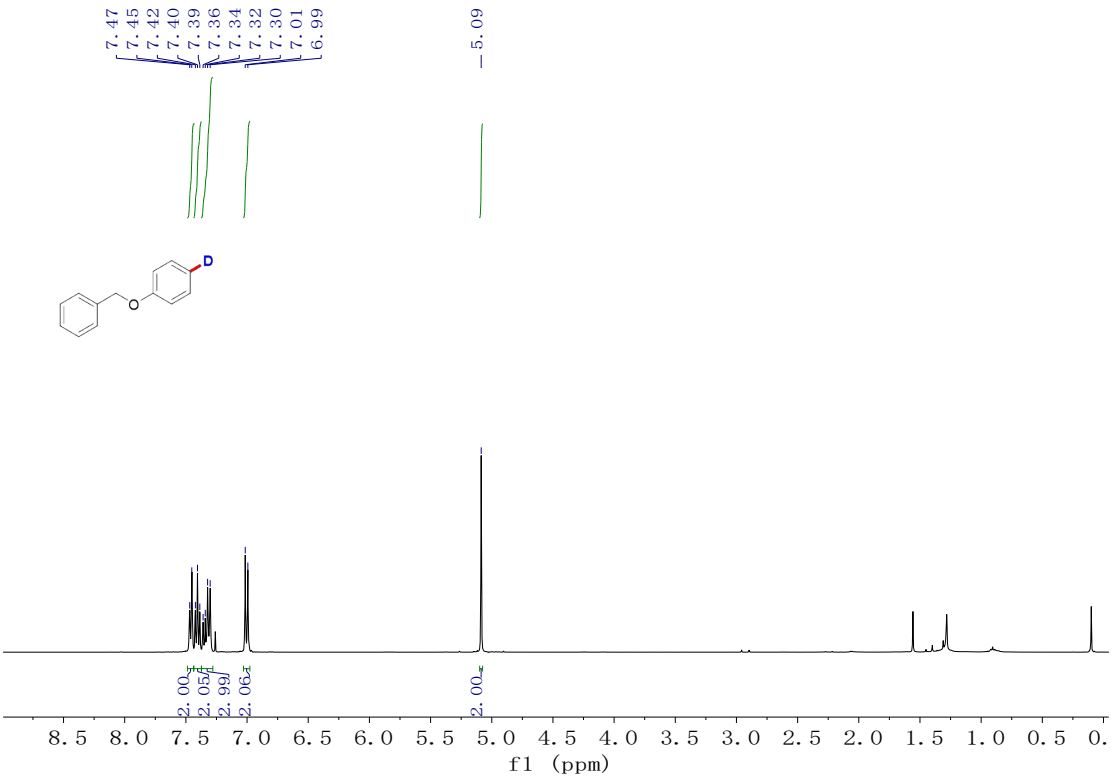
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4c**



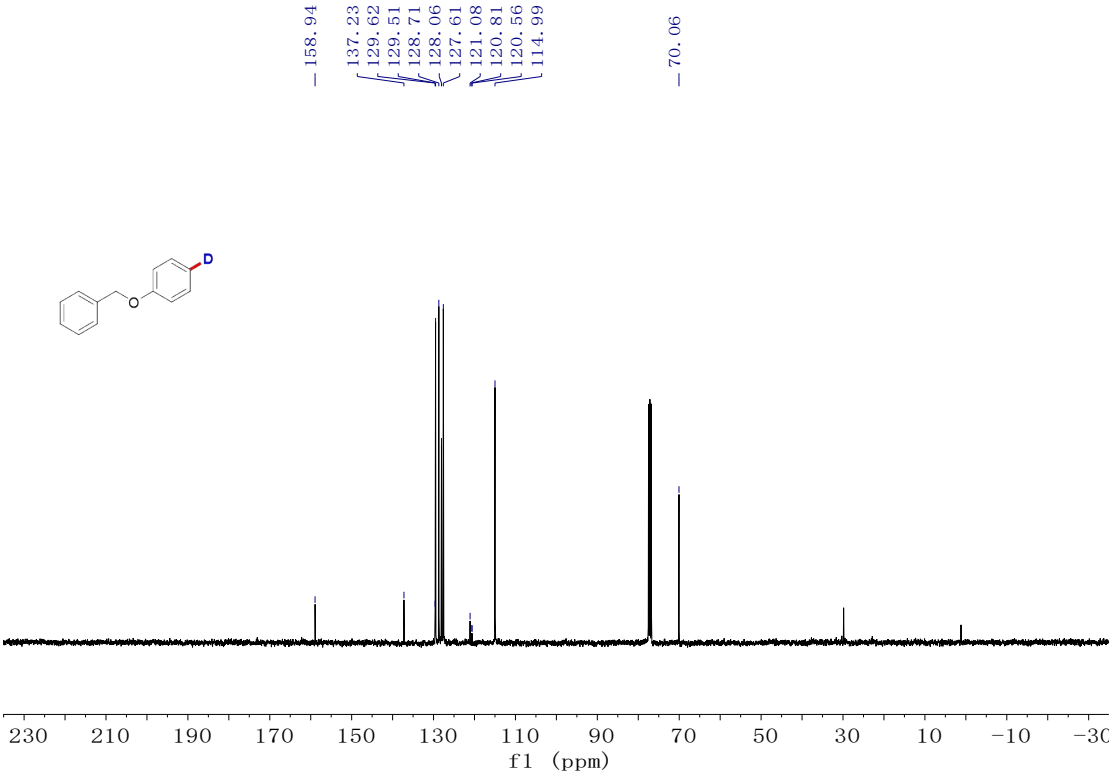

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4c**



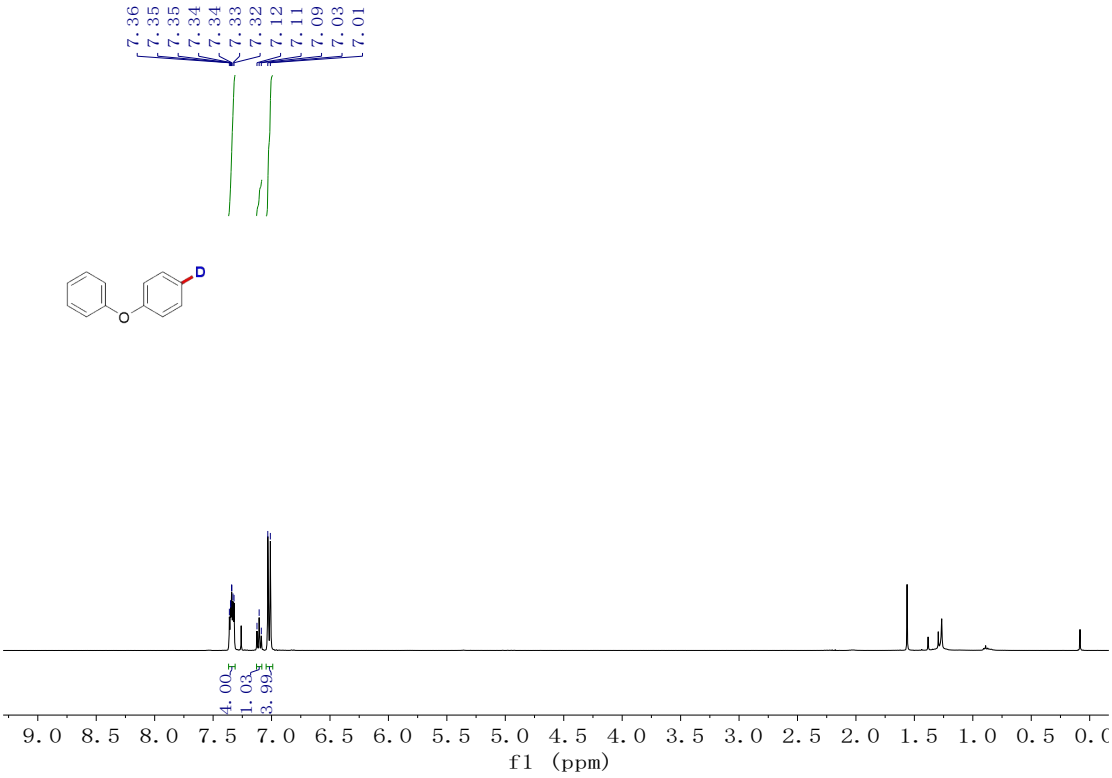
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4d**



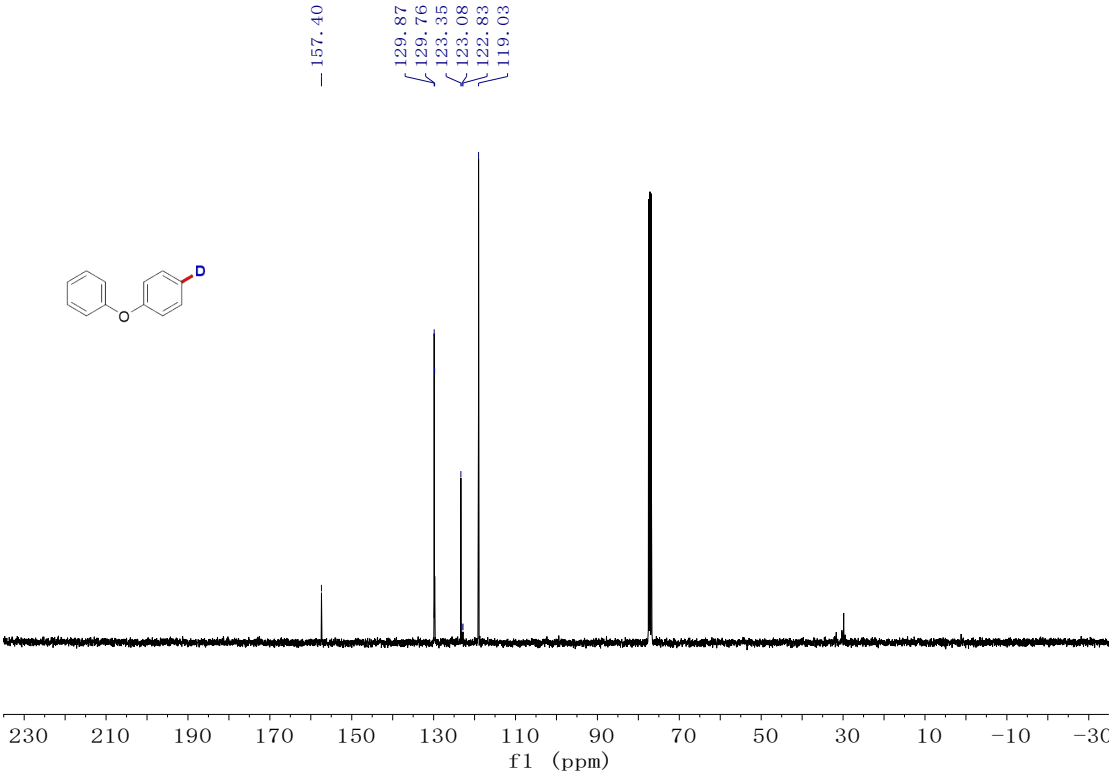

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4d**



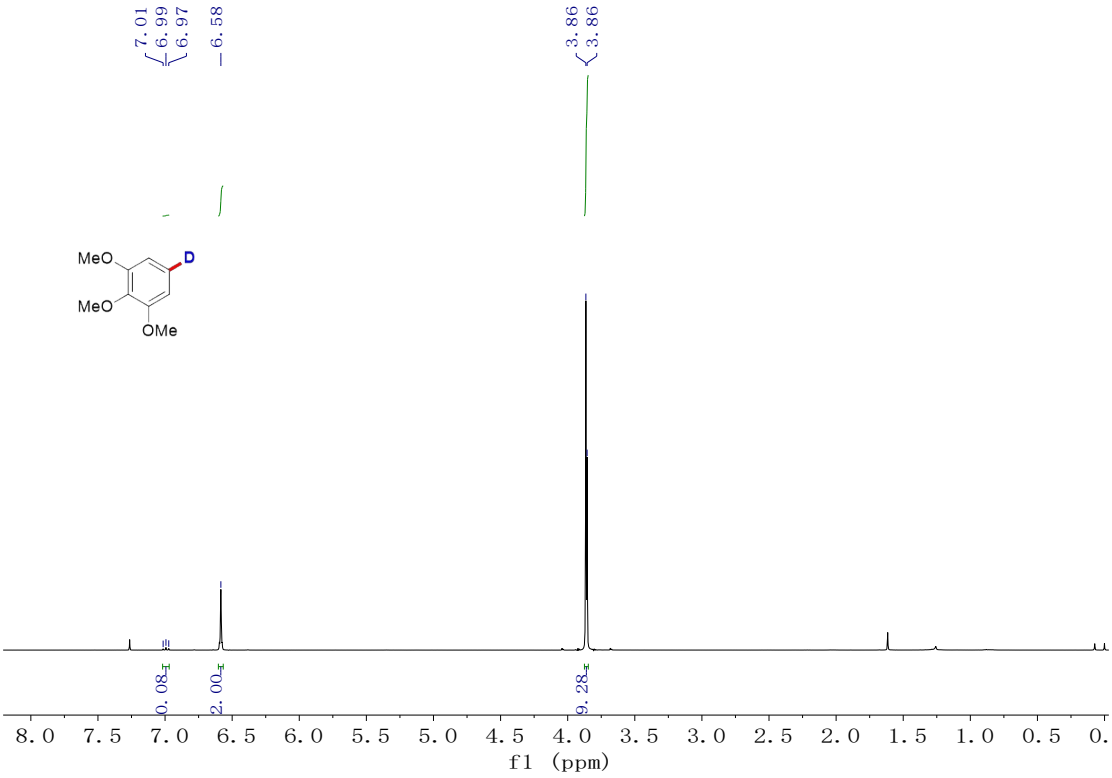
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4e**



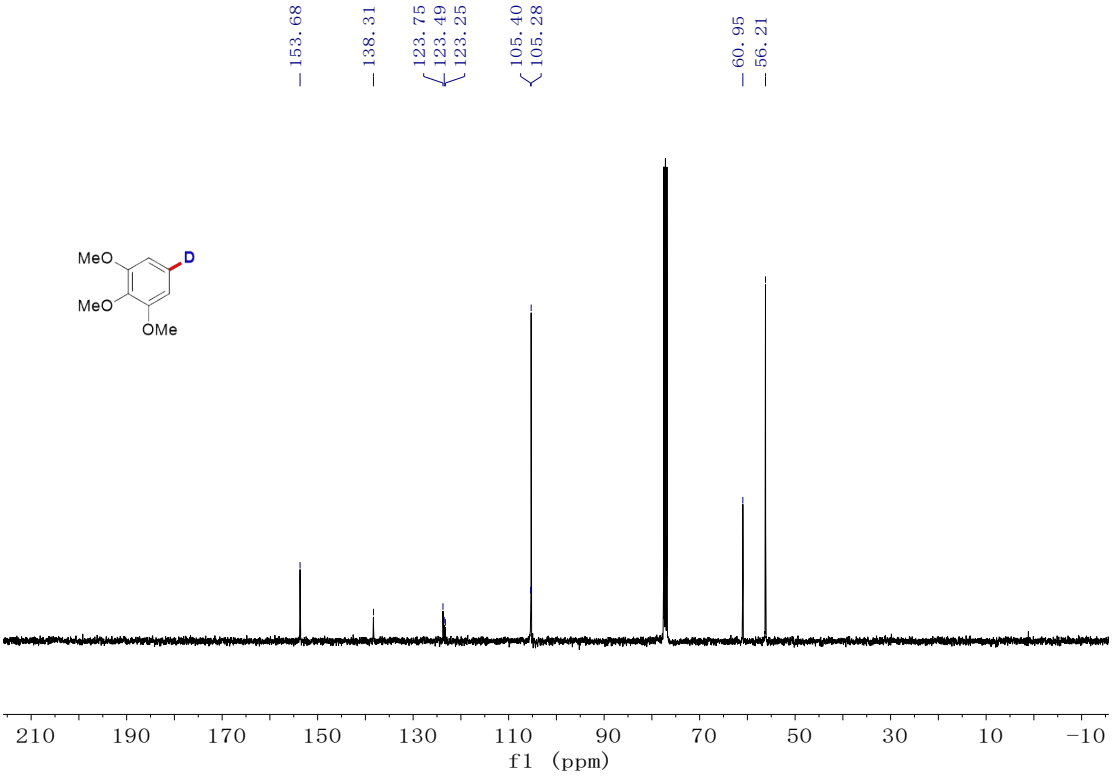

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4e**



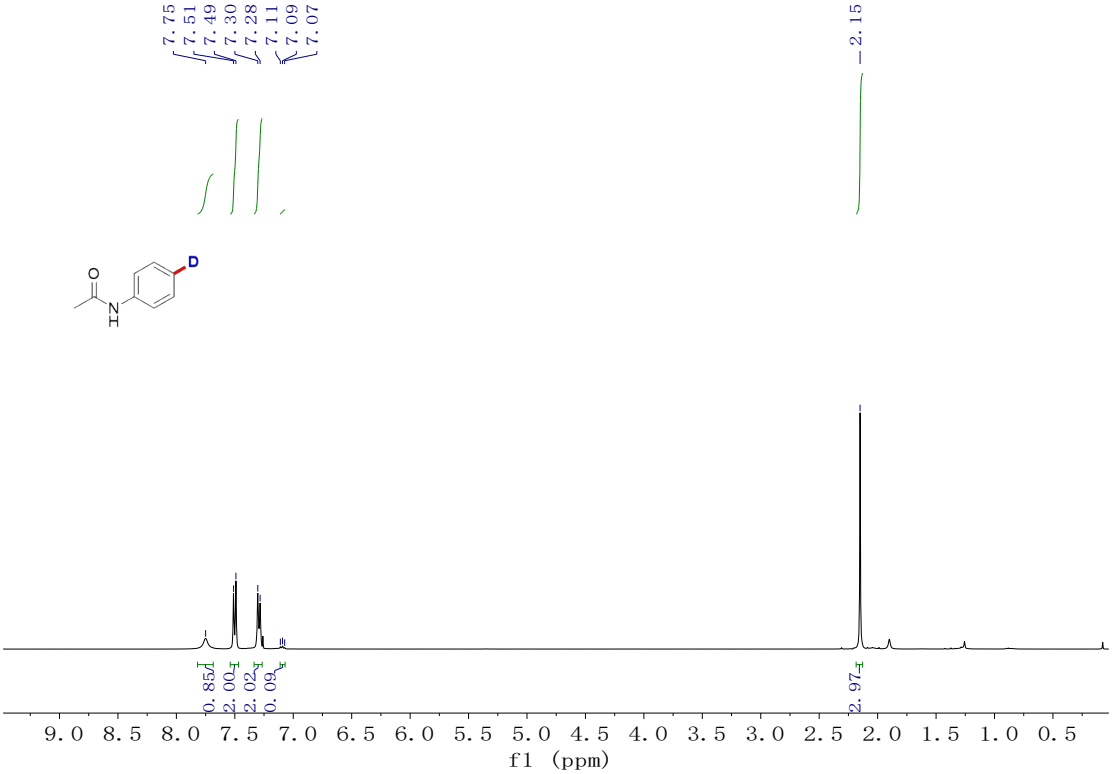
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4f**



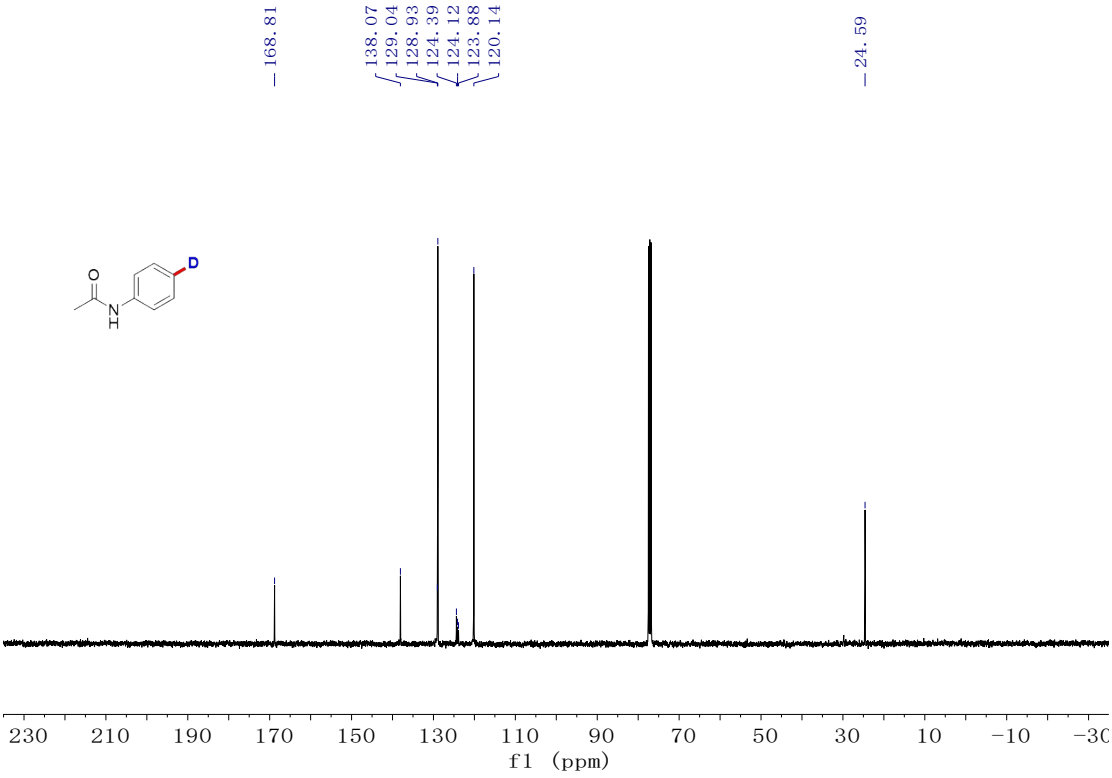

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4f**



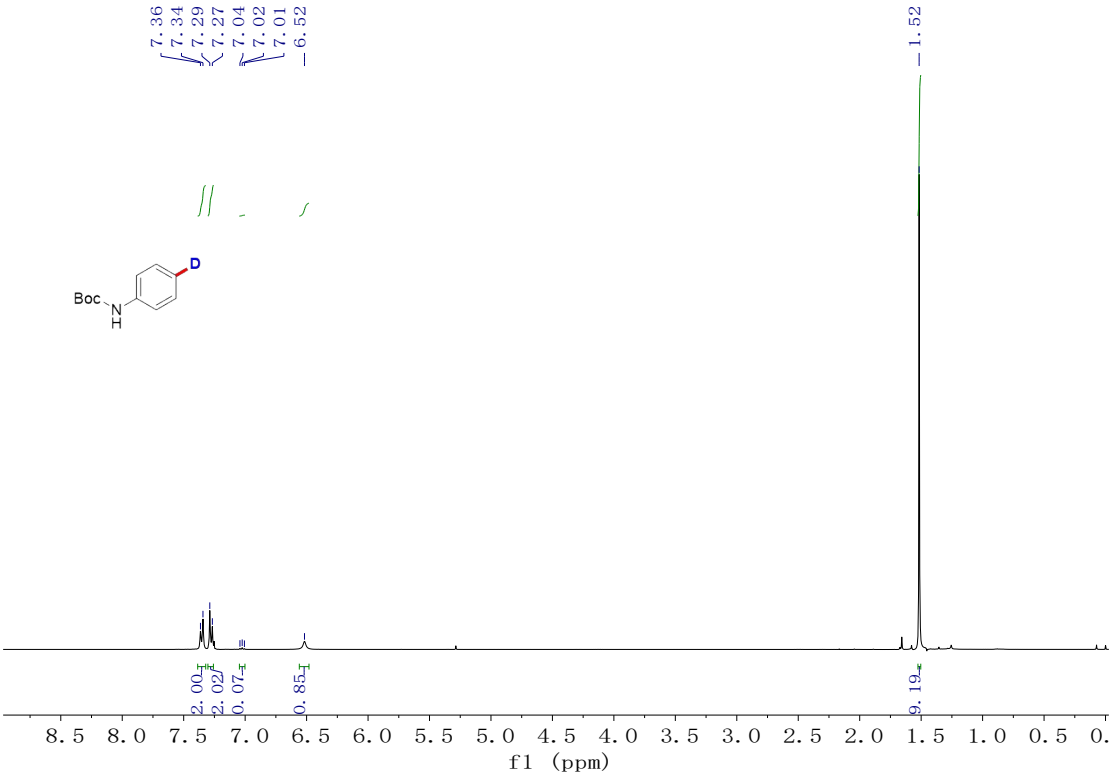
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4g**



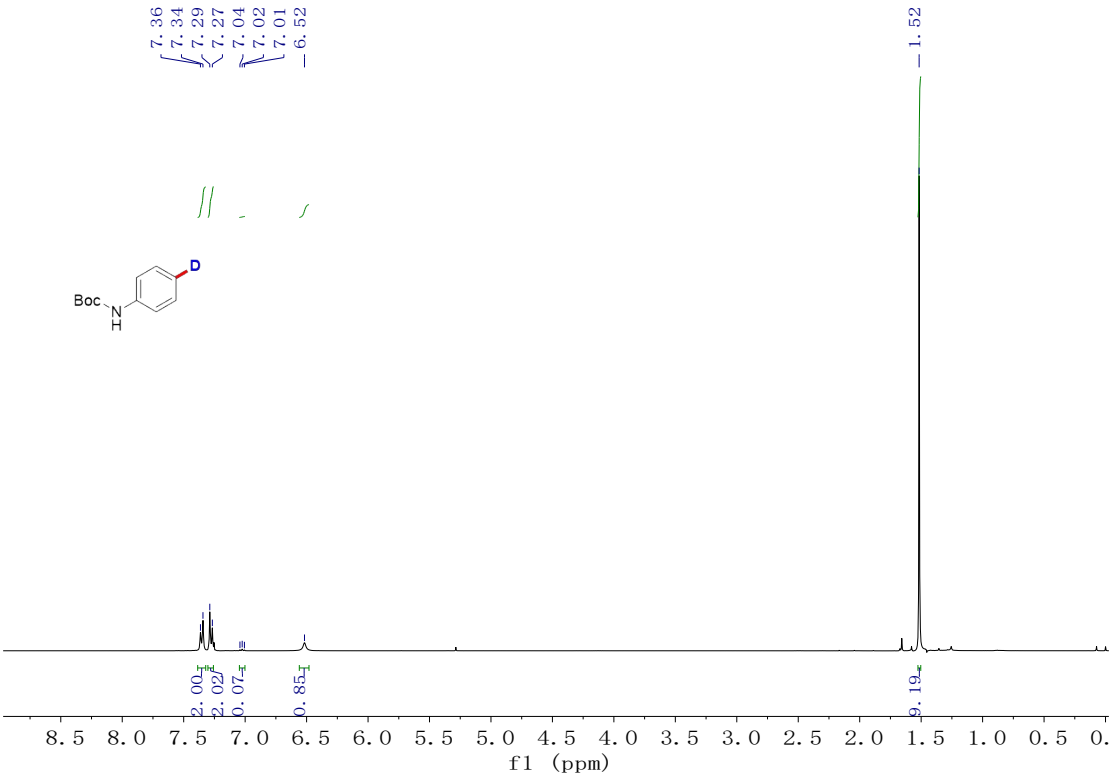

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4g**



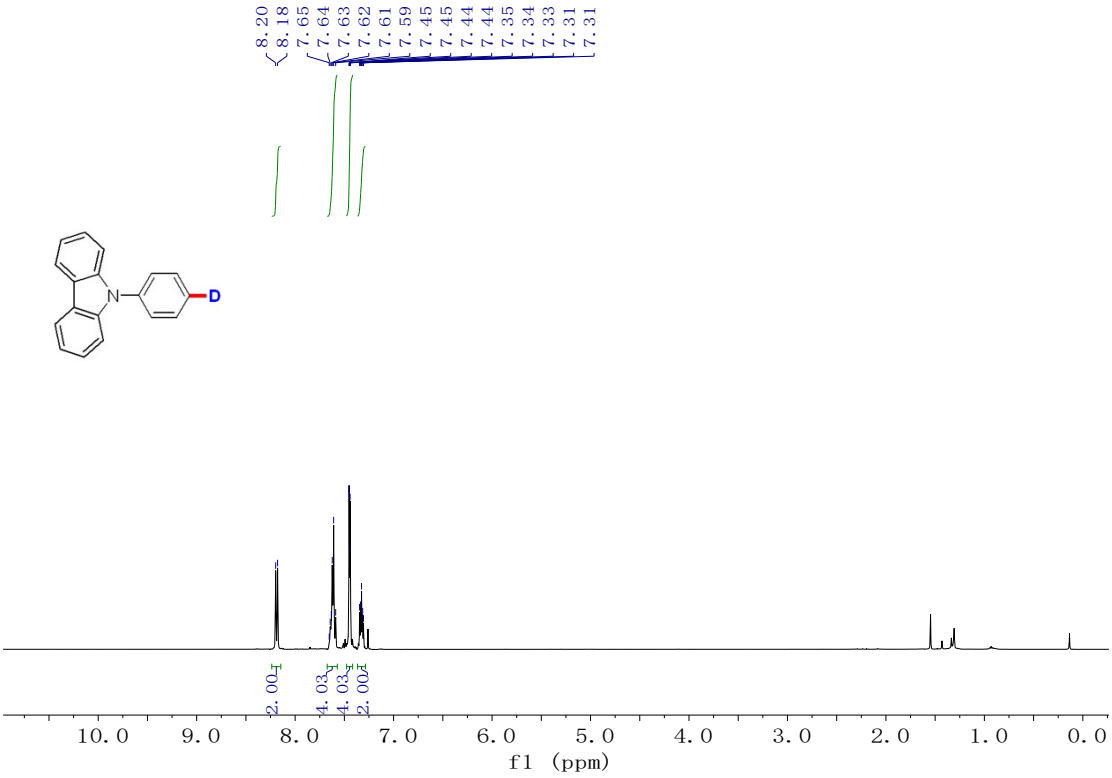
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4h**



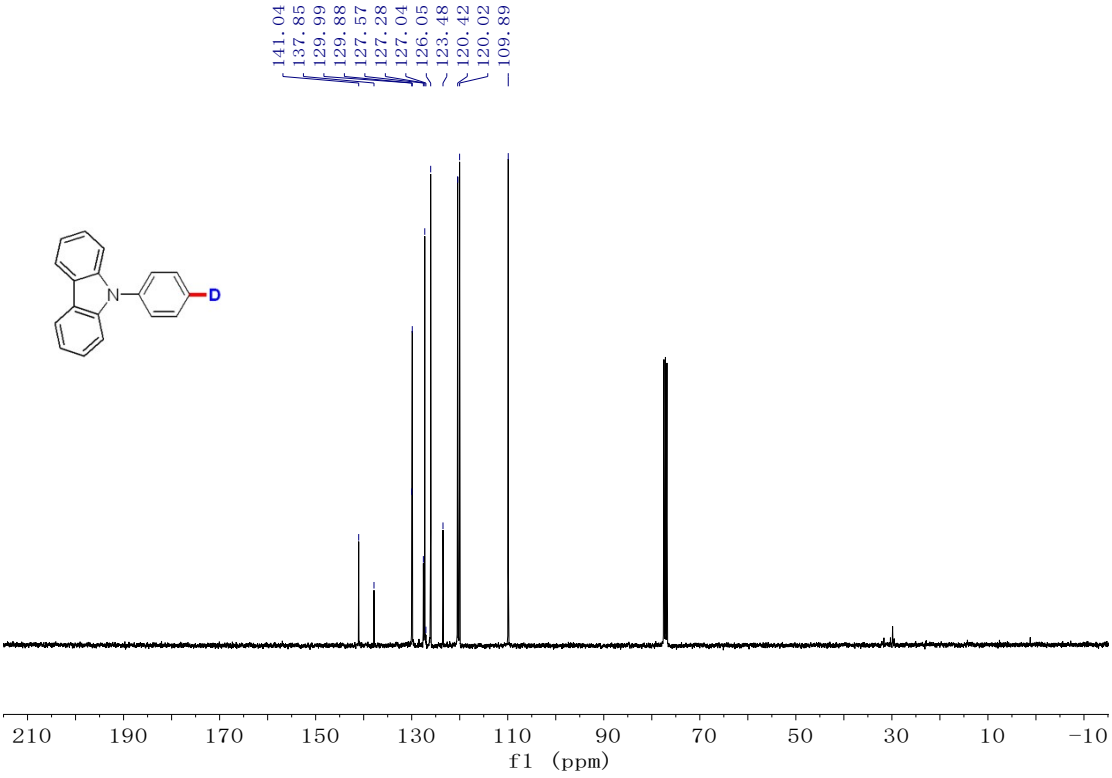

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4h**



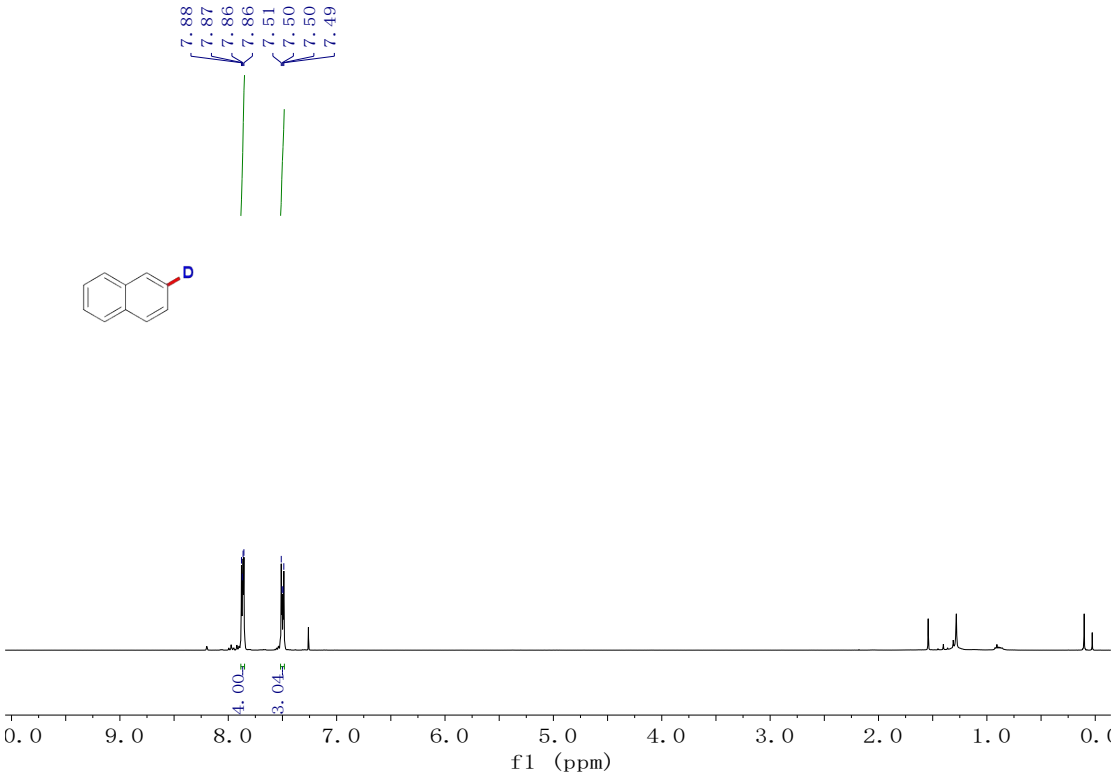
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4i**



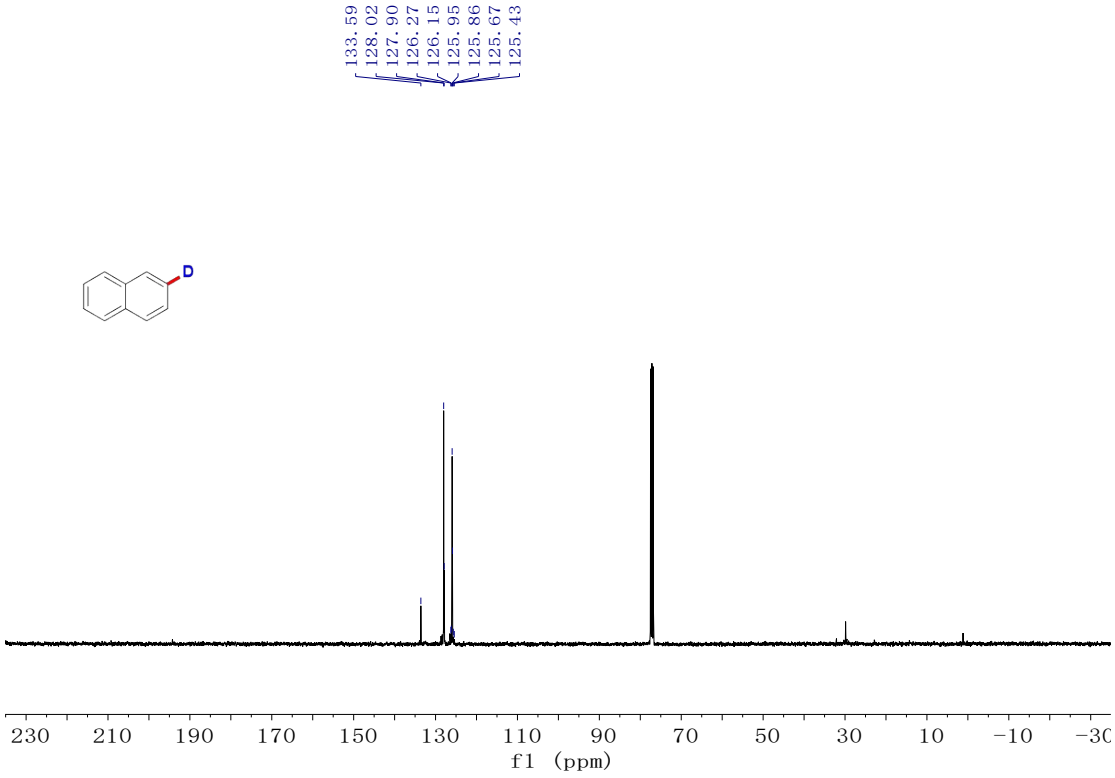

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4i**



$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4j**




$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4j**



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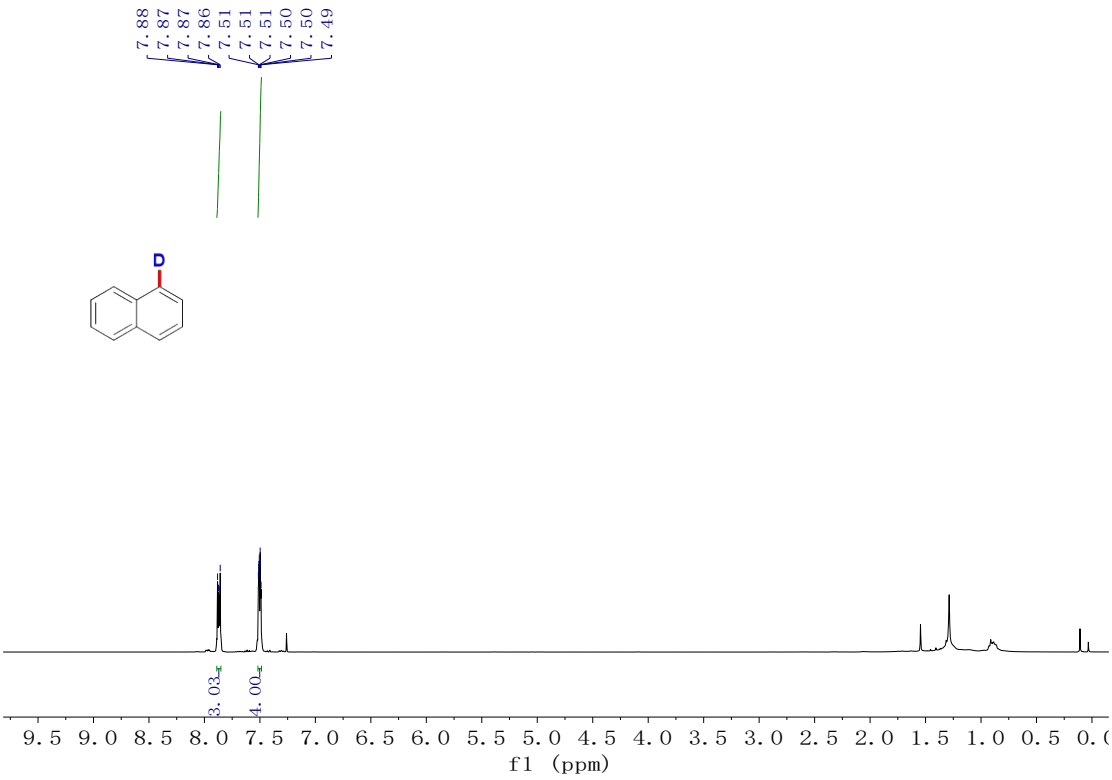
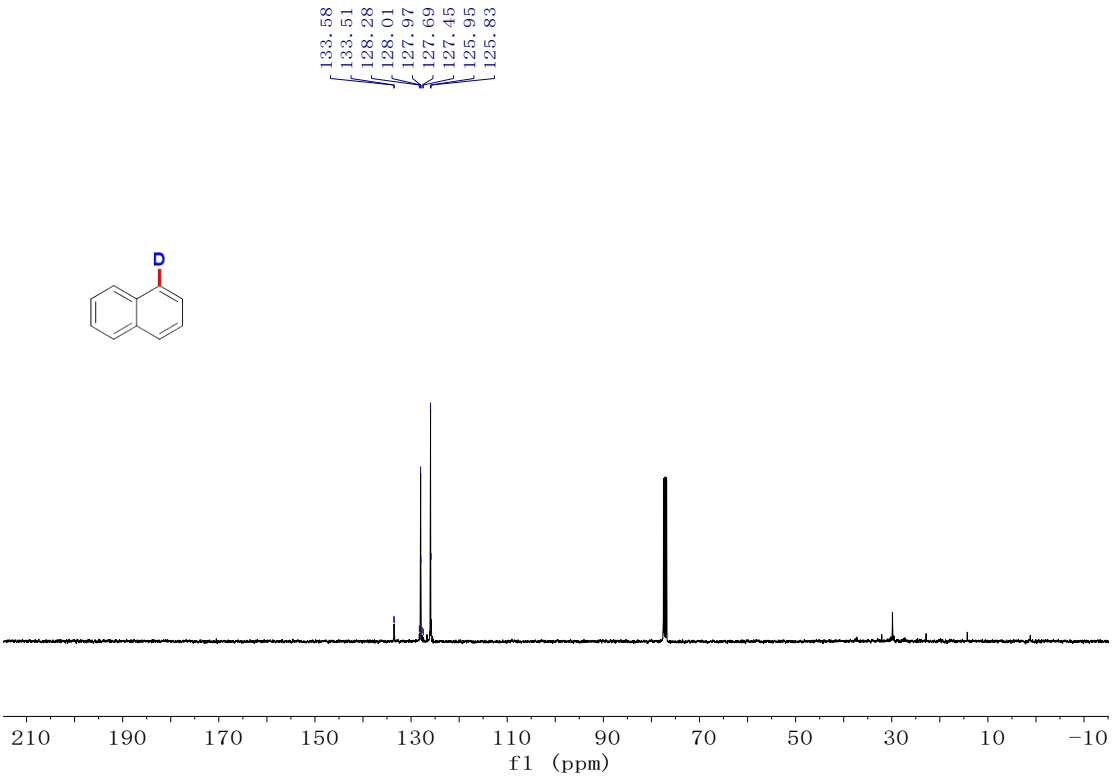
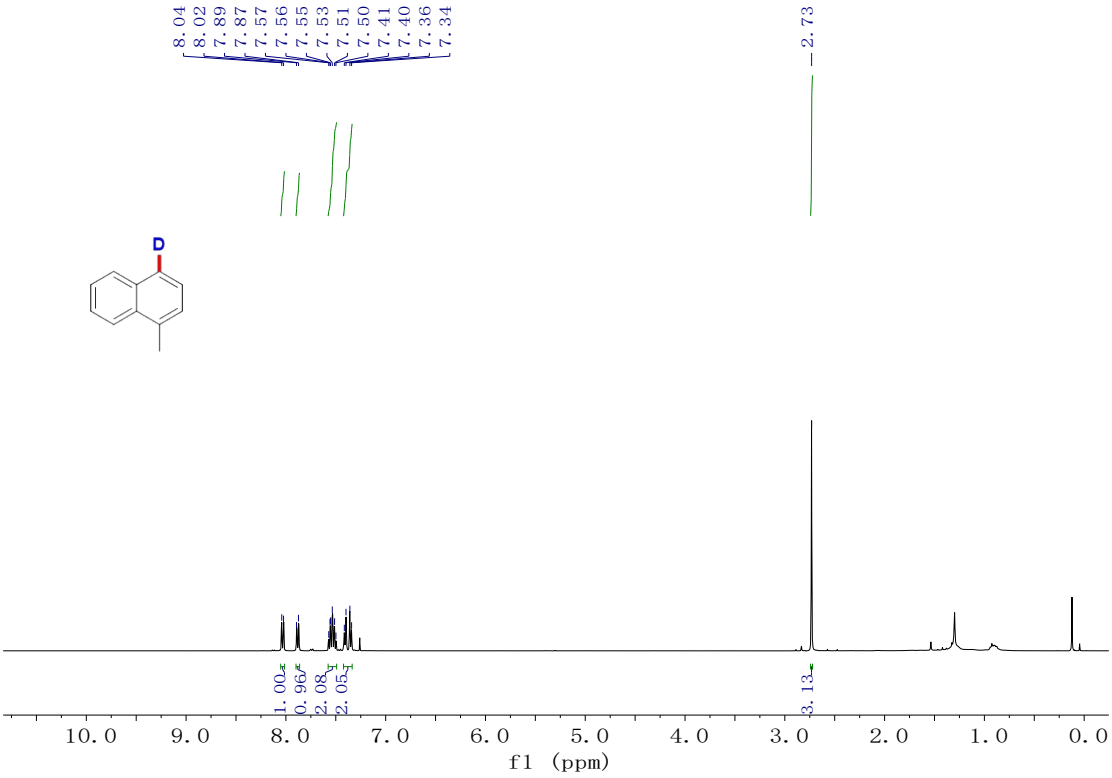


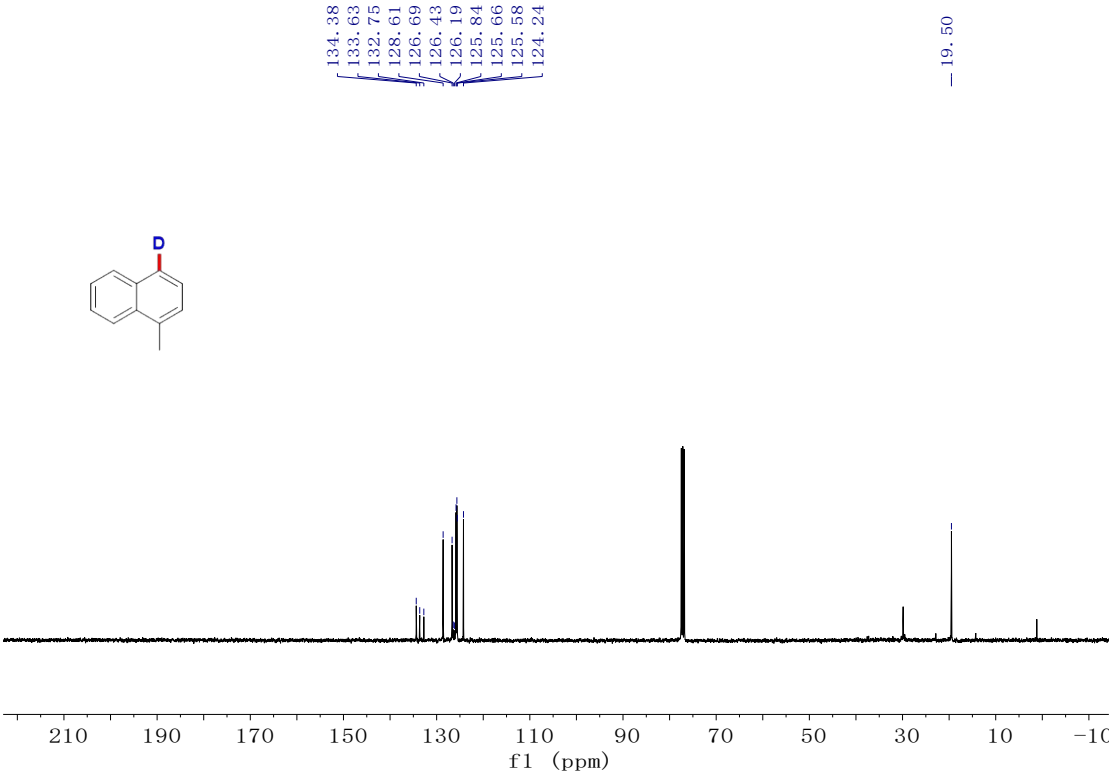

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4k**



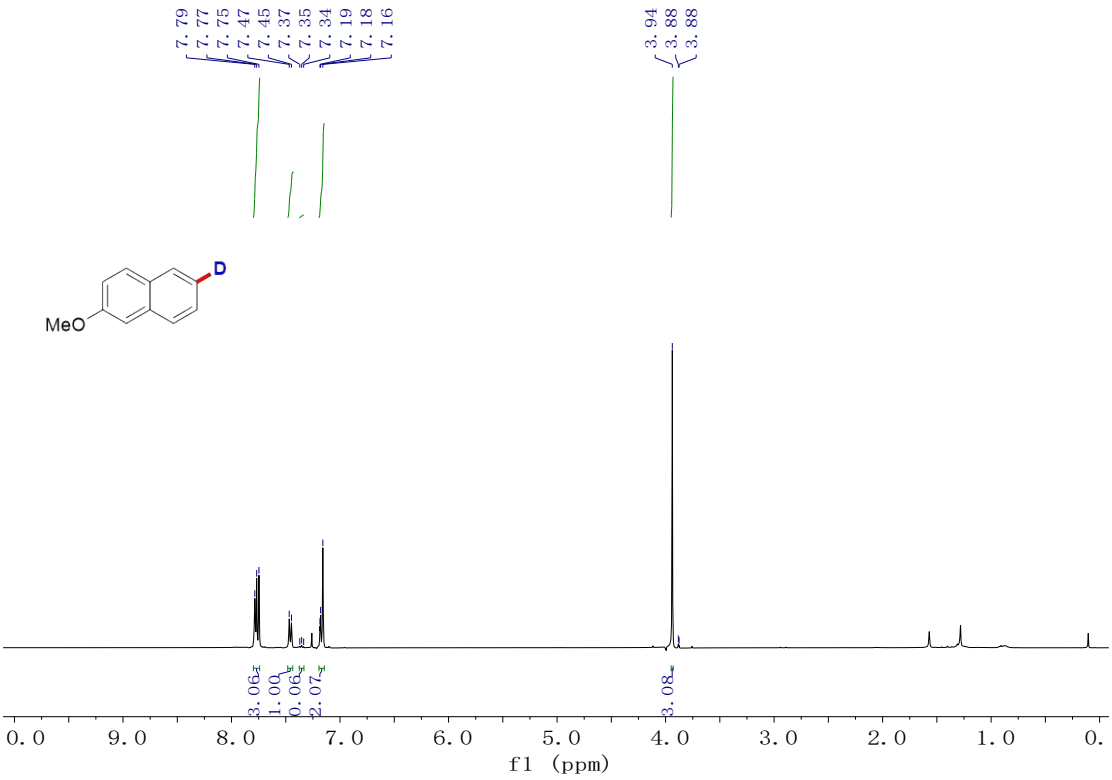
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4l**



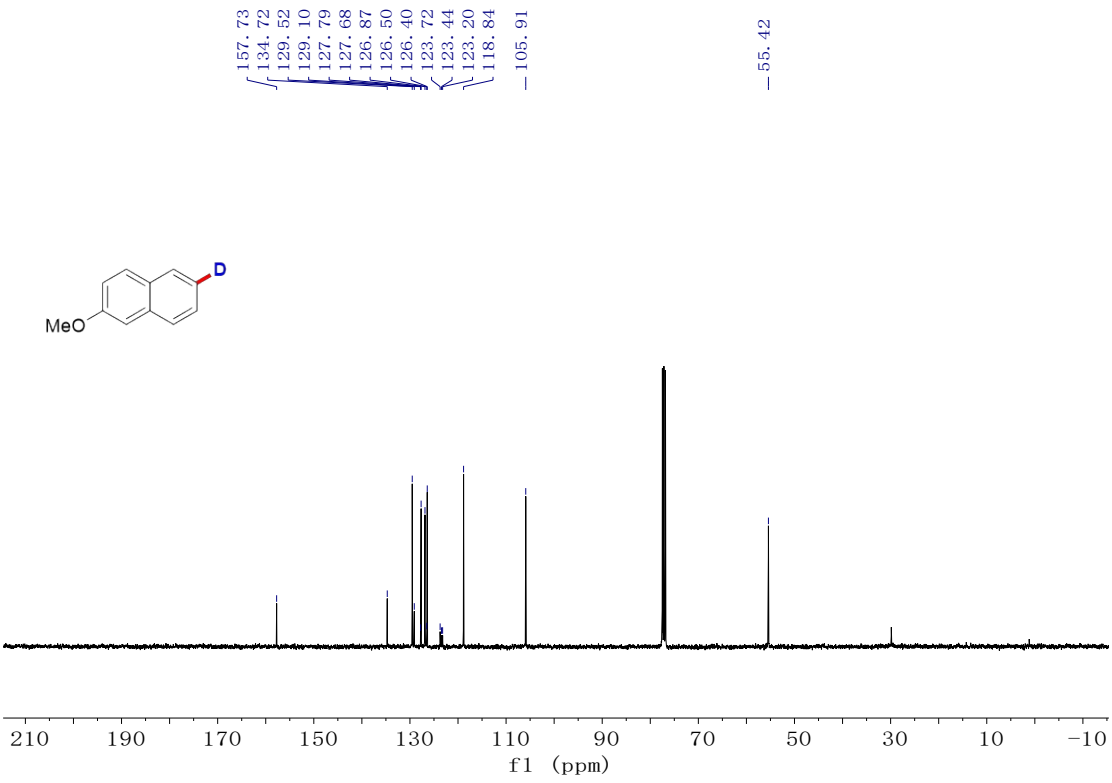

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4l**



$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4m**




$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4m**



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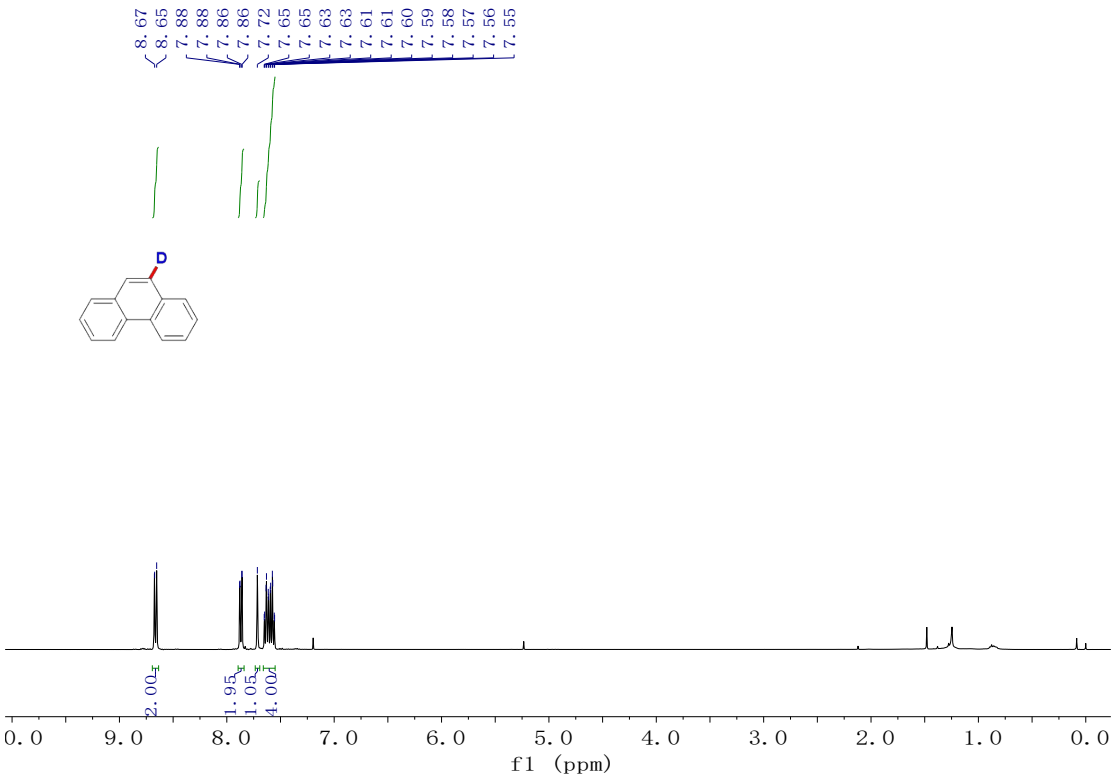
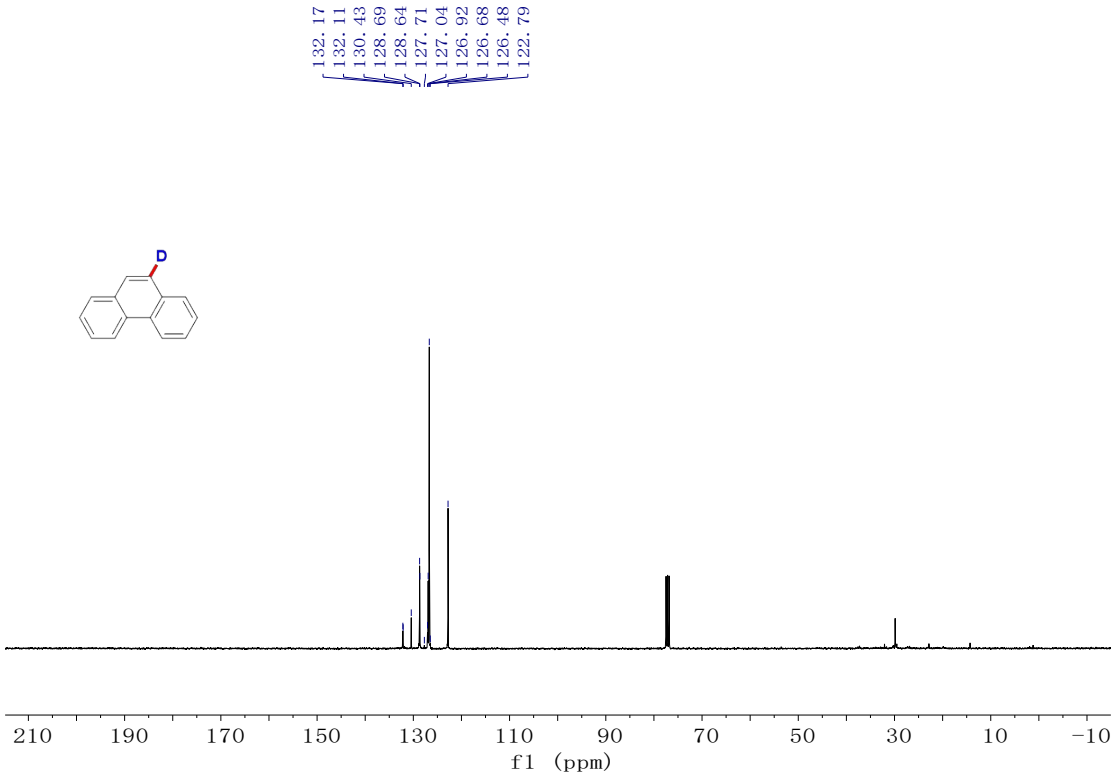
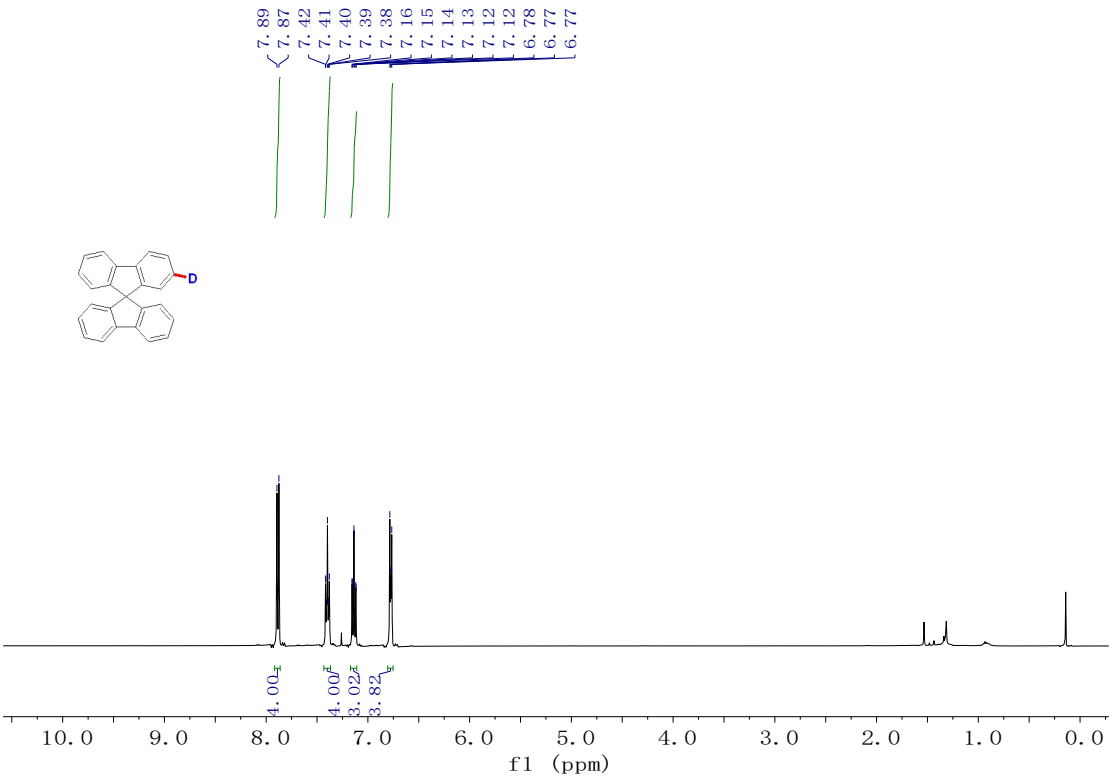


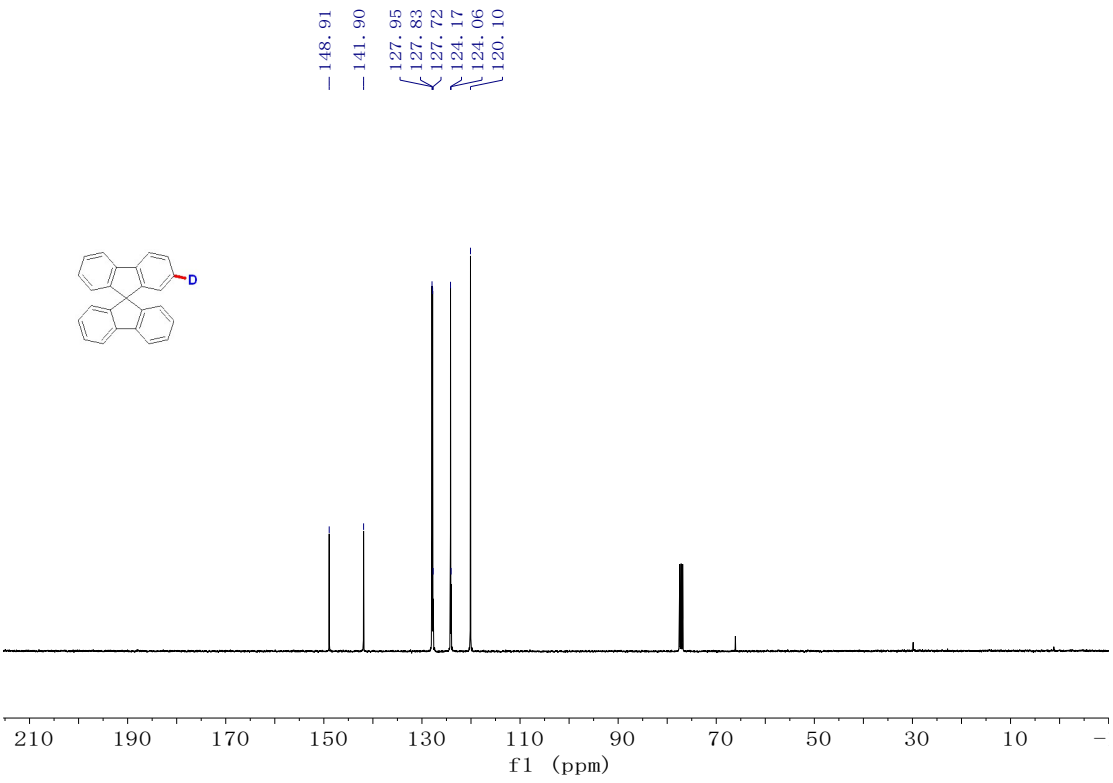

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4n**



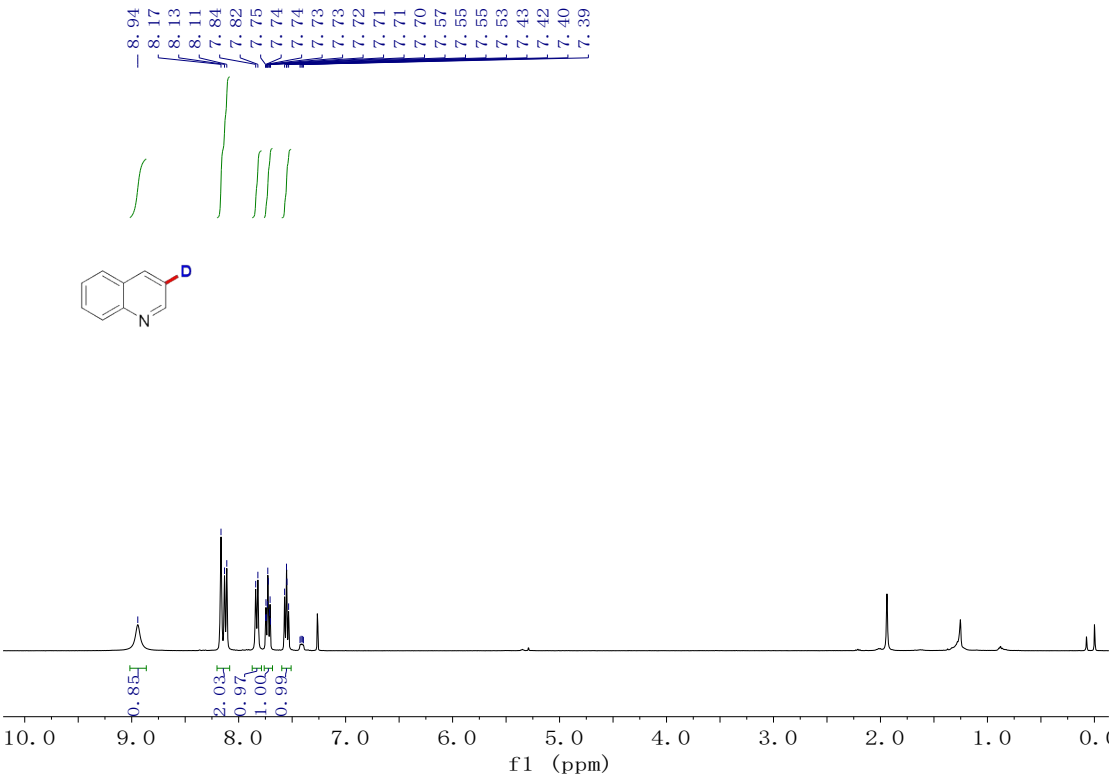
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4o**



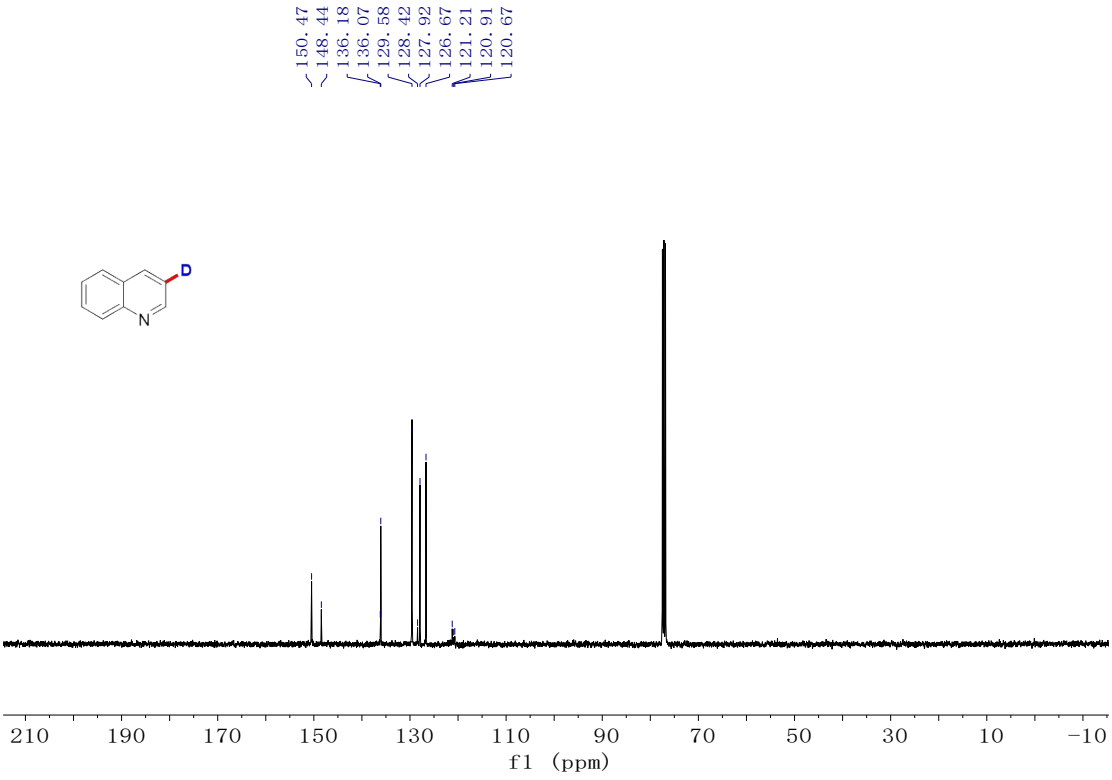

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4o**



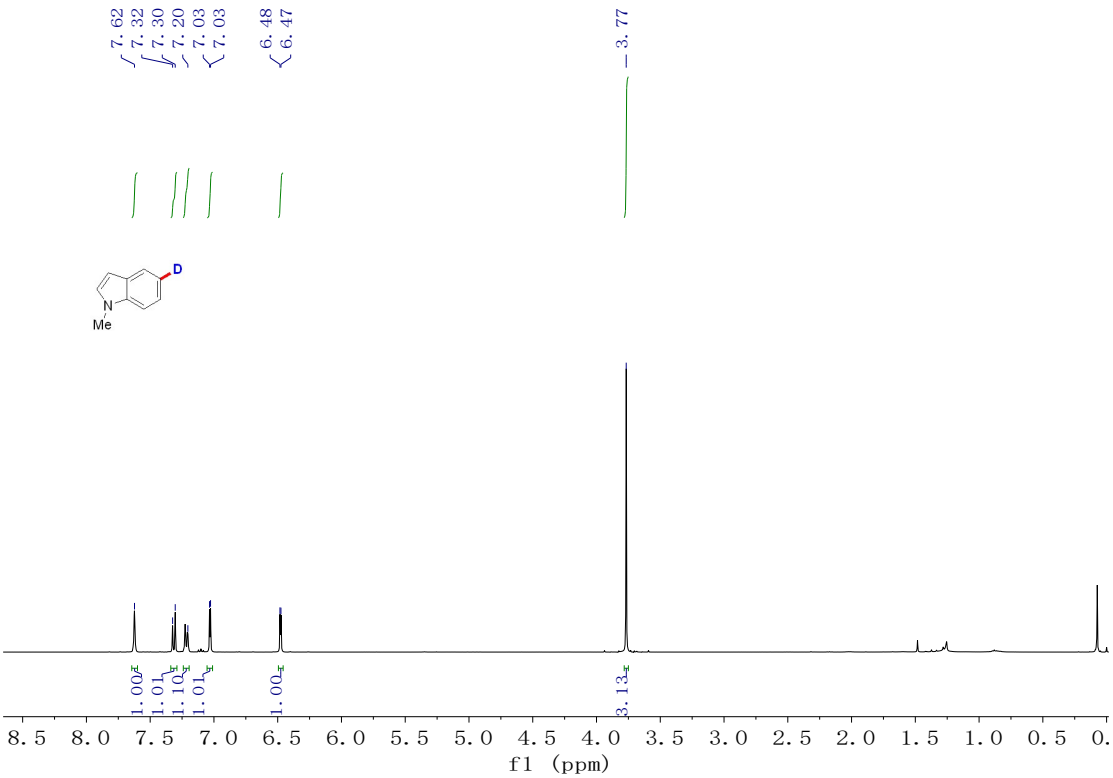
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4p**



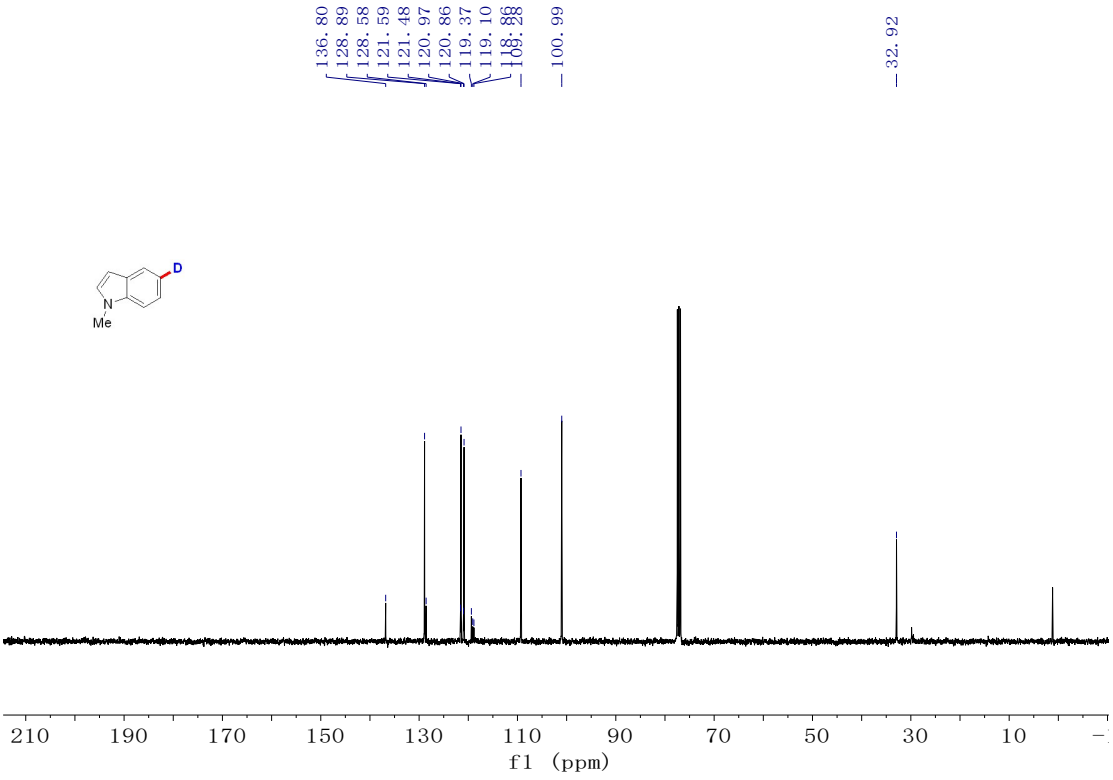

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4p**



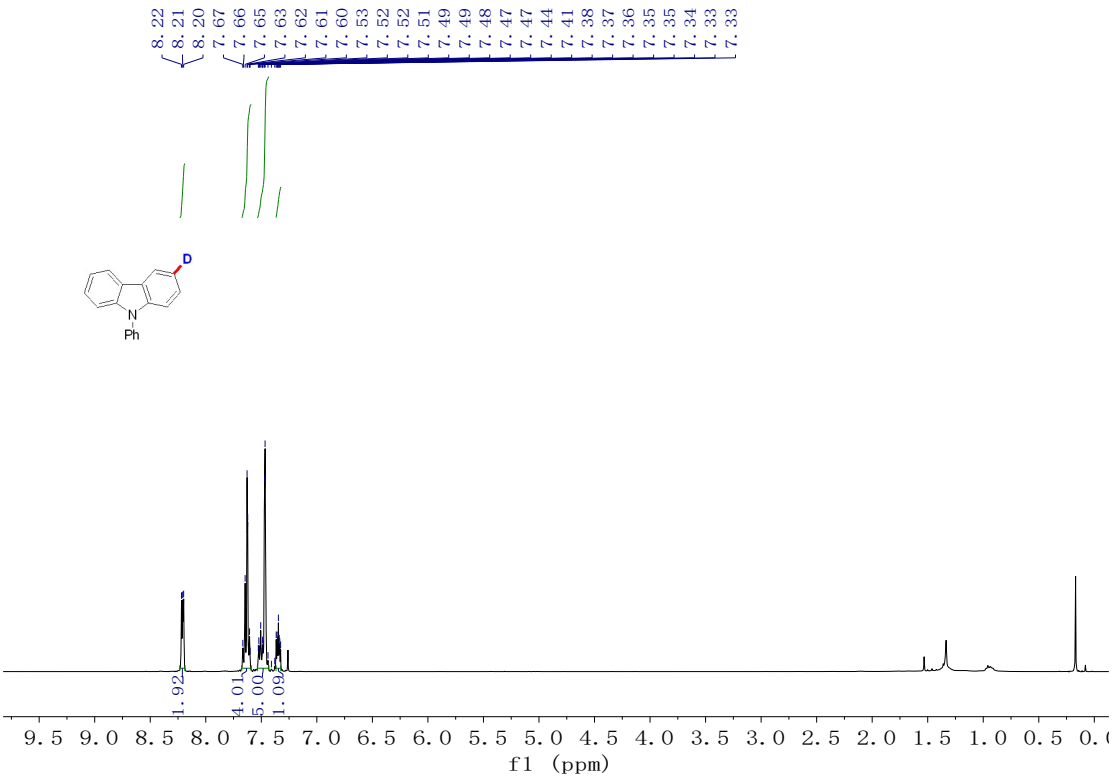
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4q**



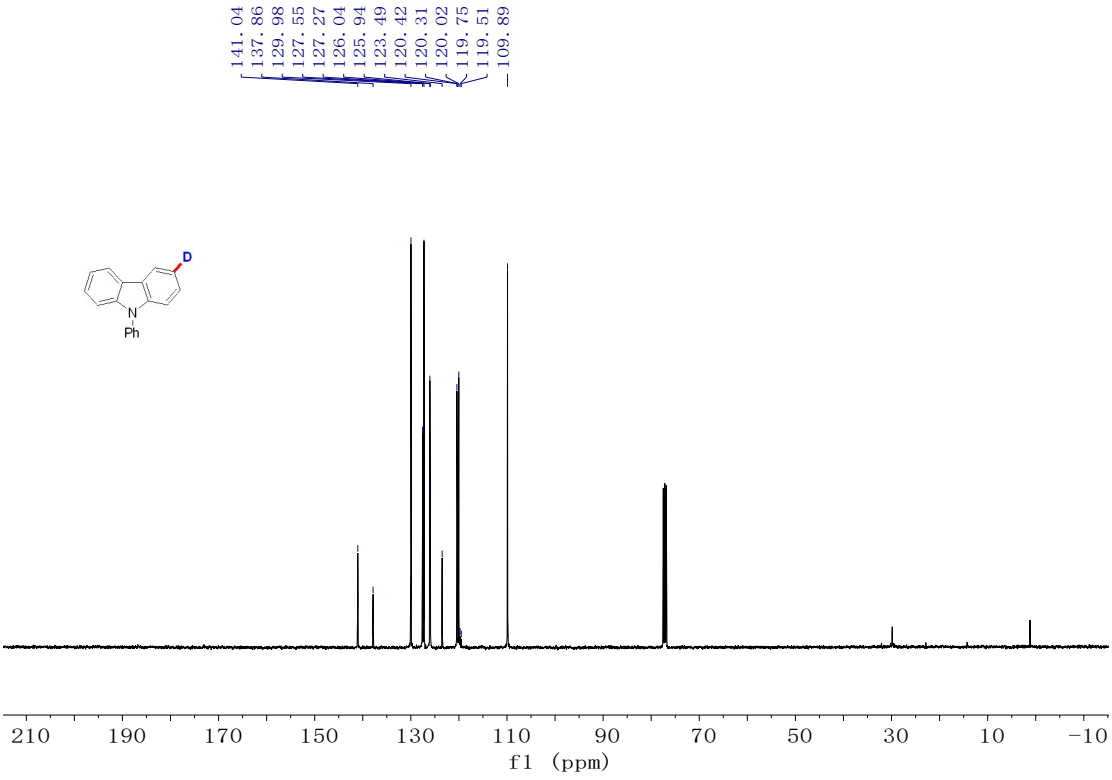

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4q**



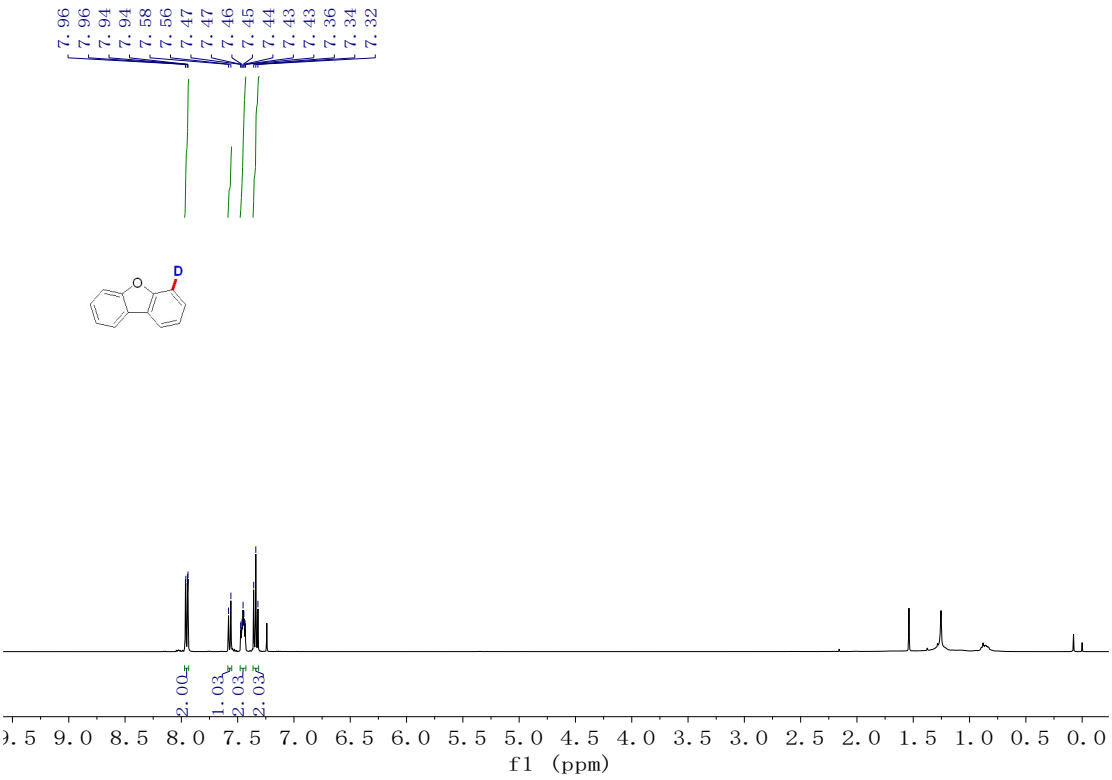
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4r**



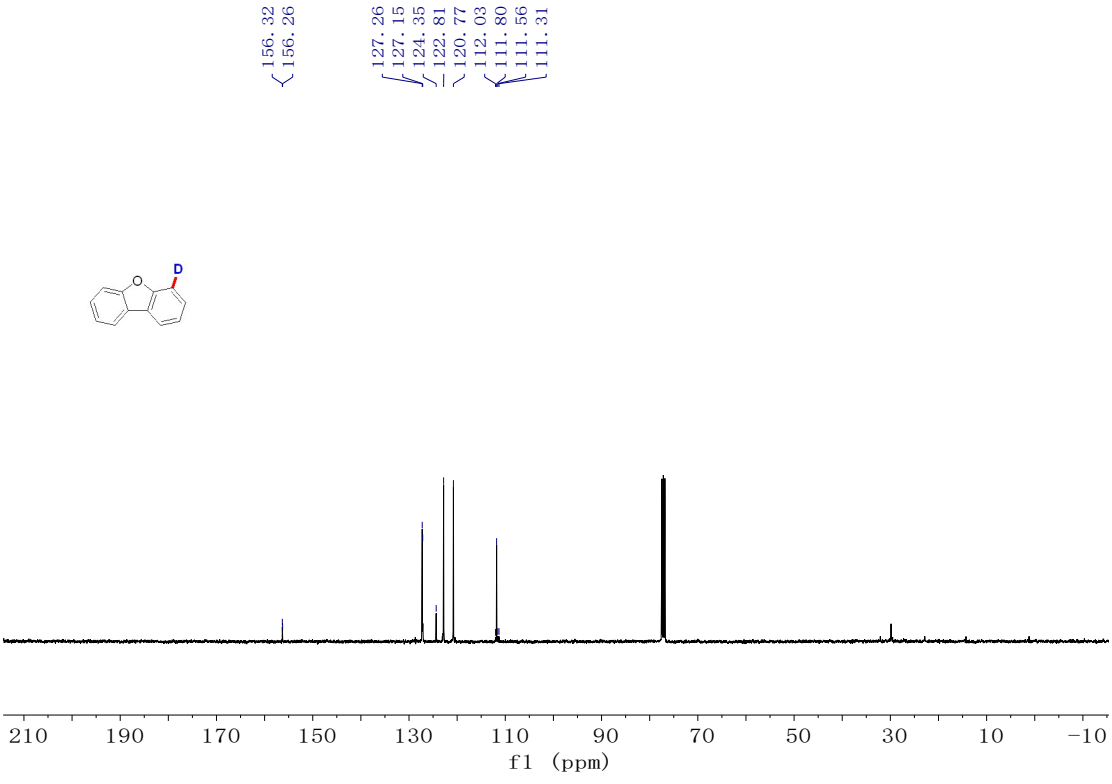

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4r**



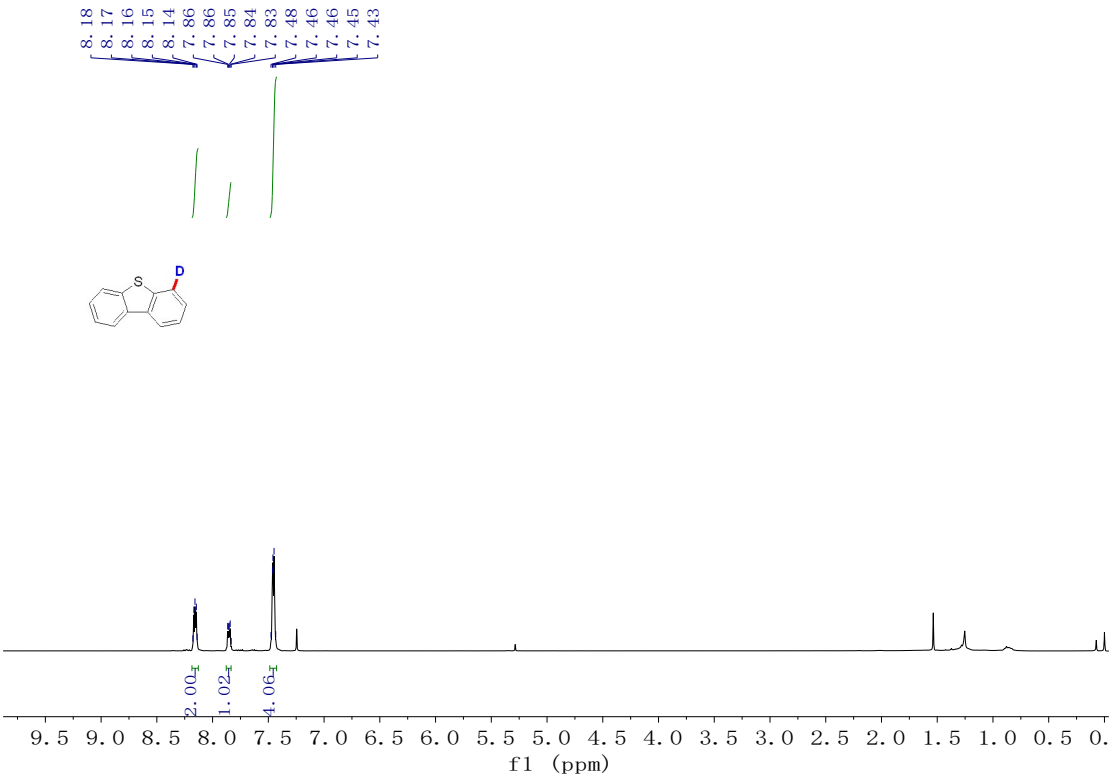
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4s**



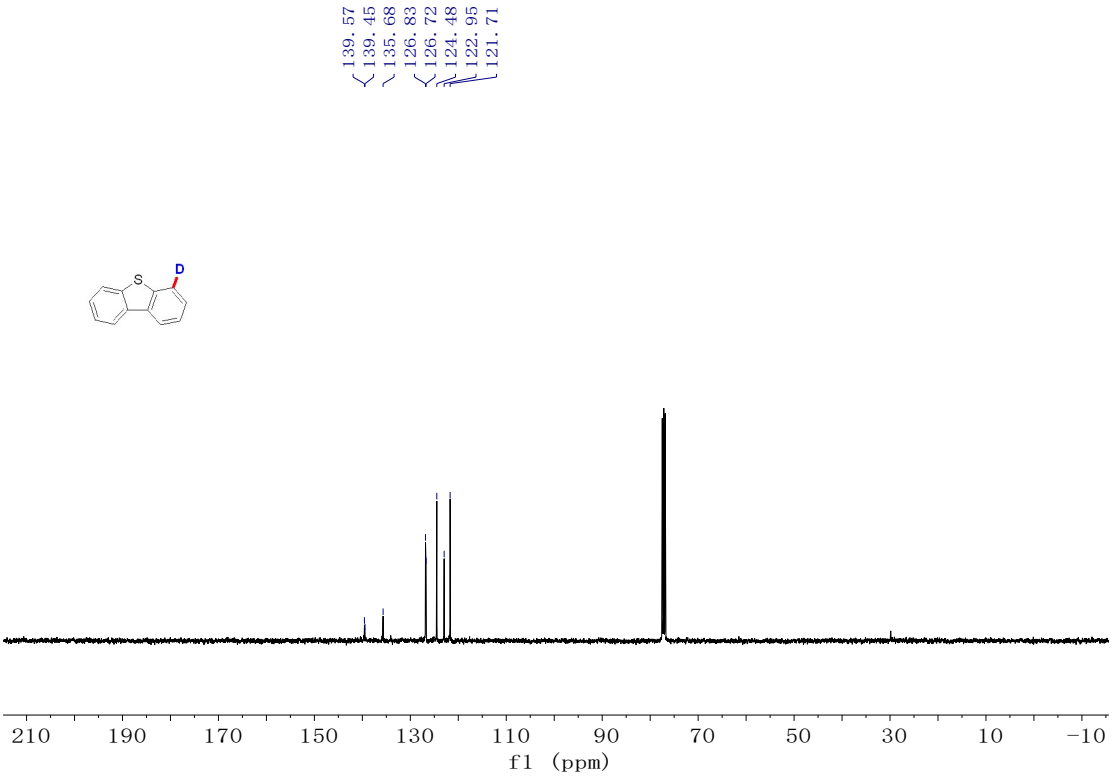

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4s**



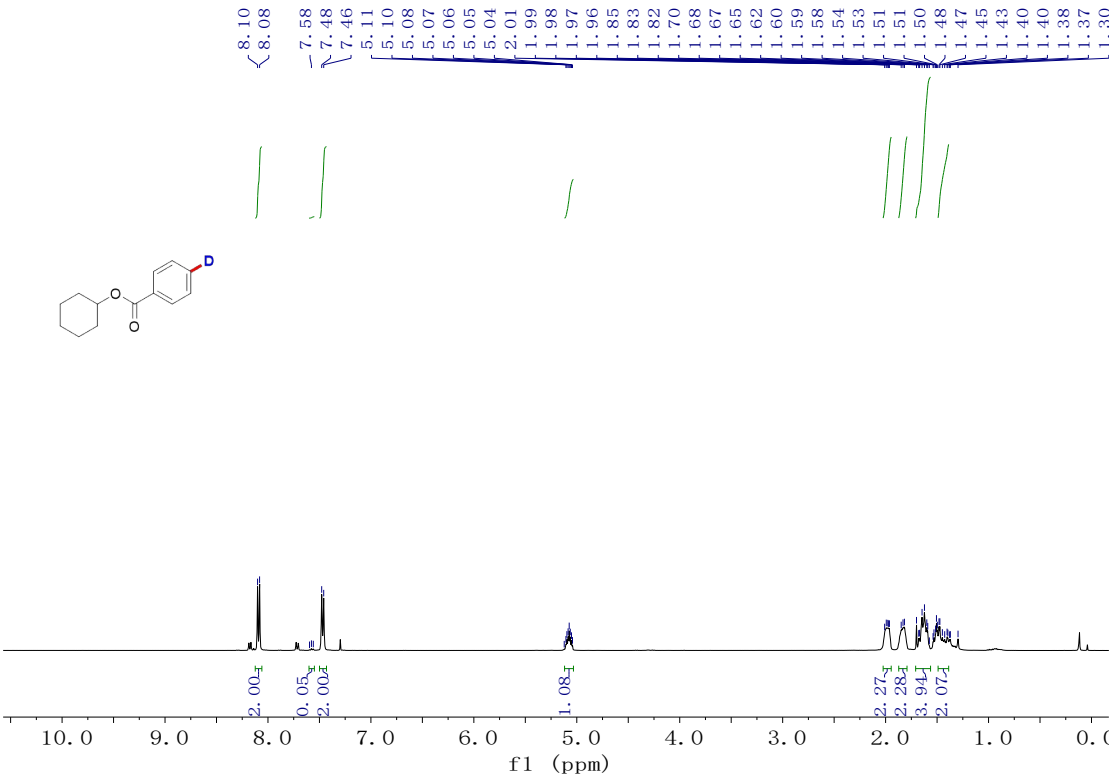
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4t**



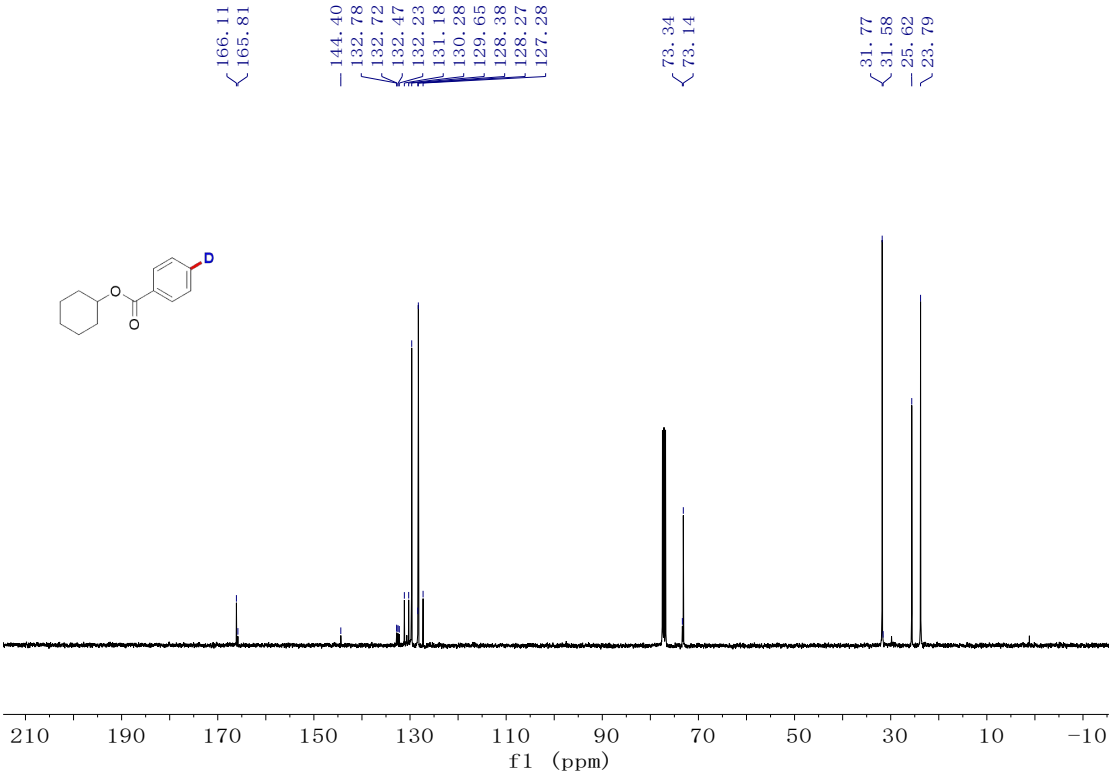

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4t**



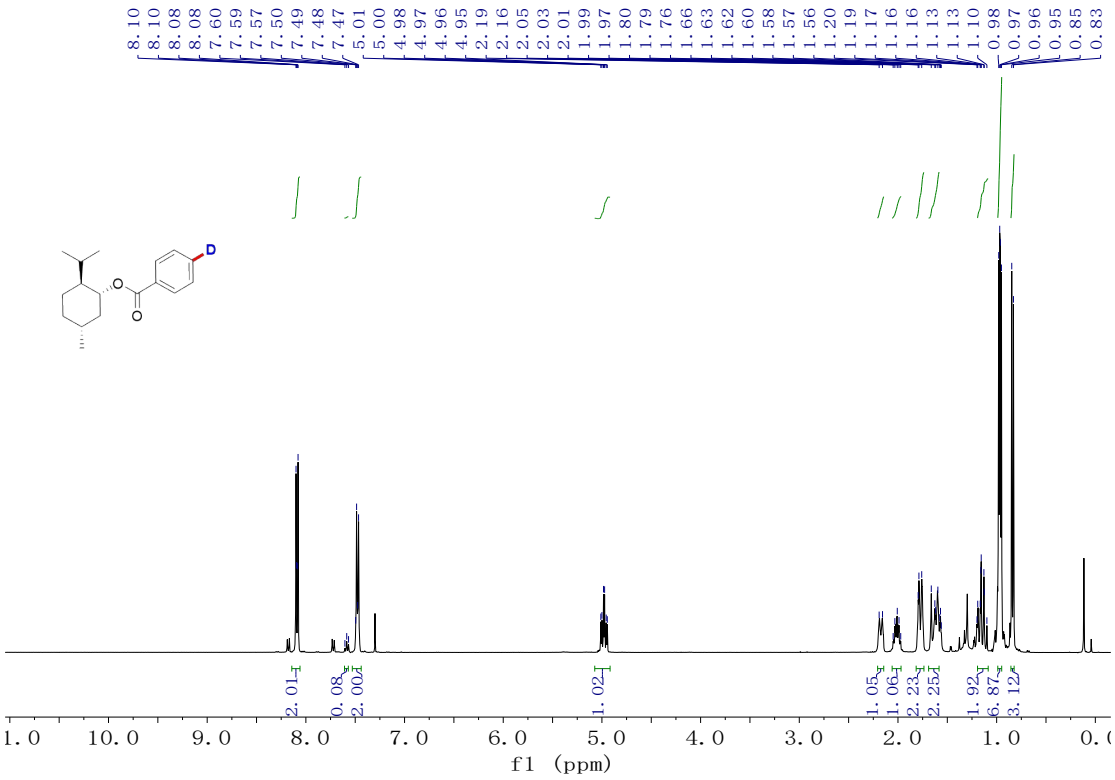
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4u**



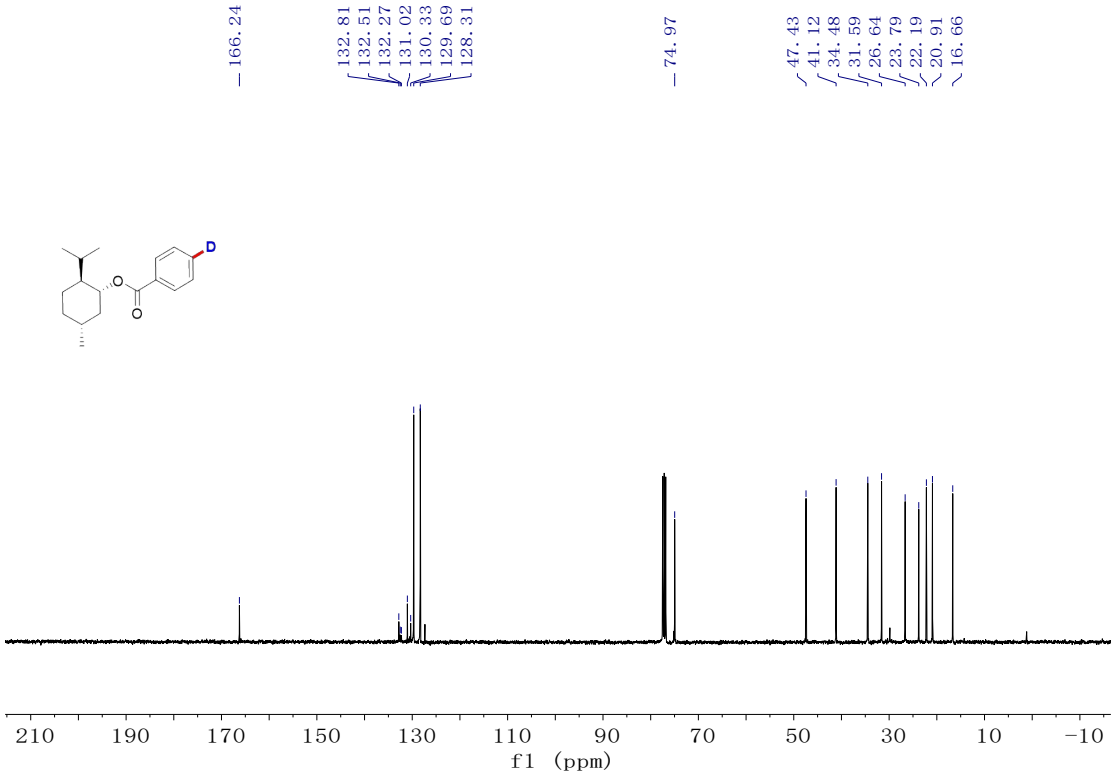

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4u**



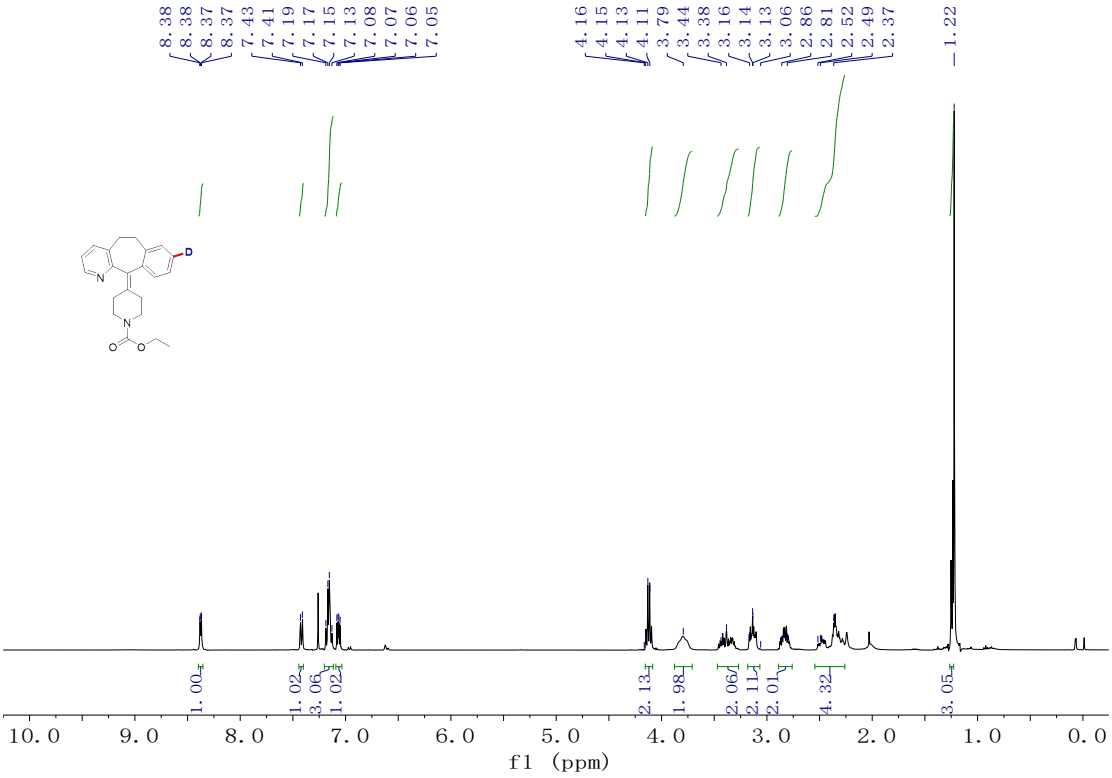
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4v**



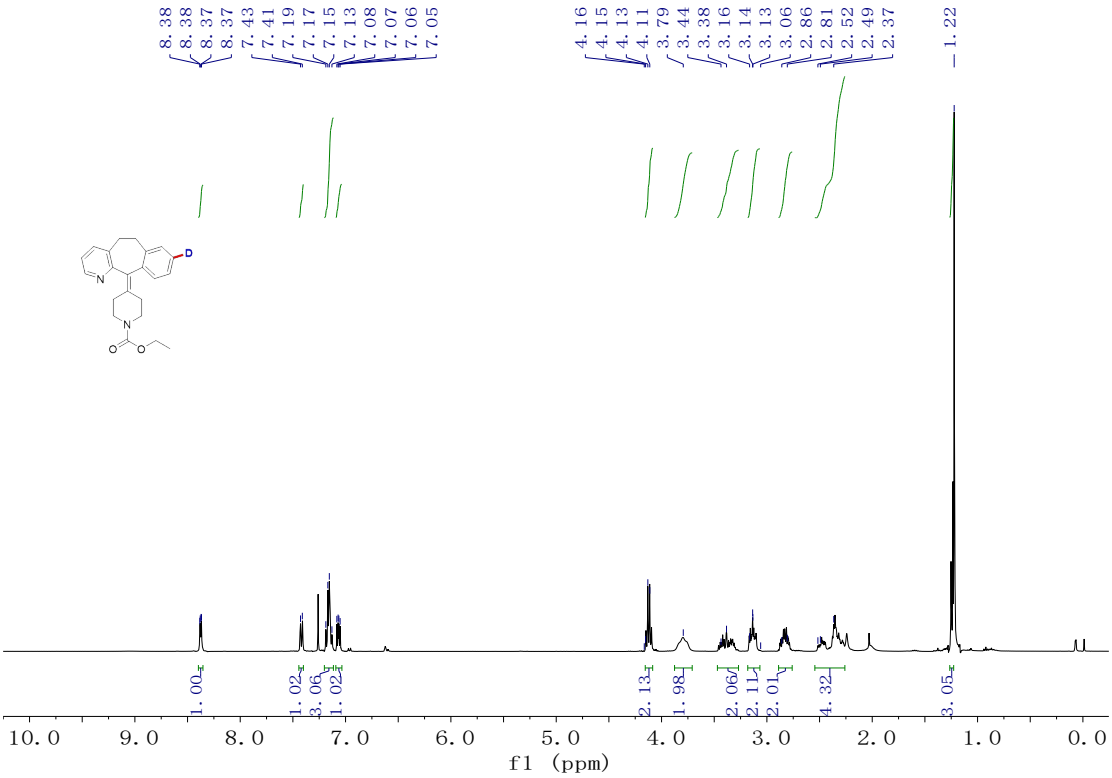

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4v**



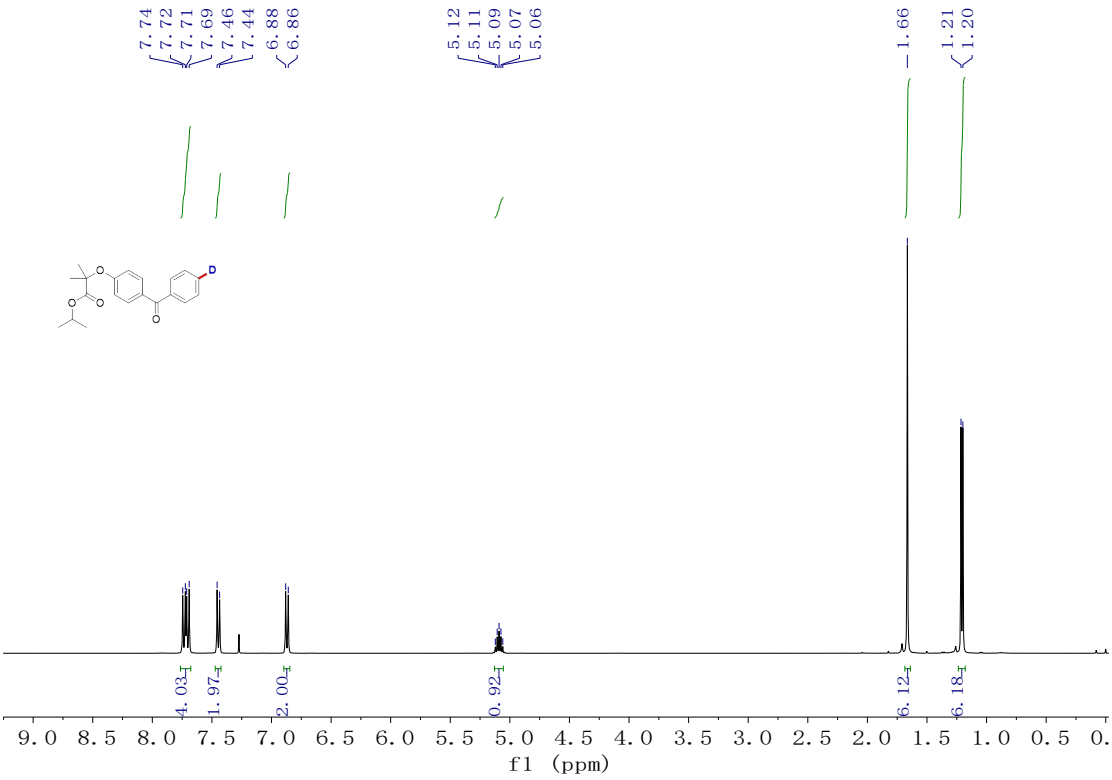
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4w**



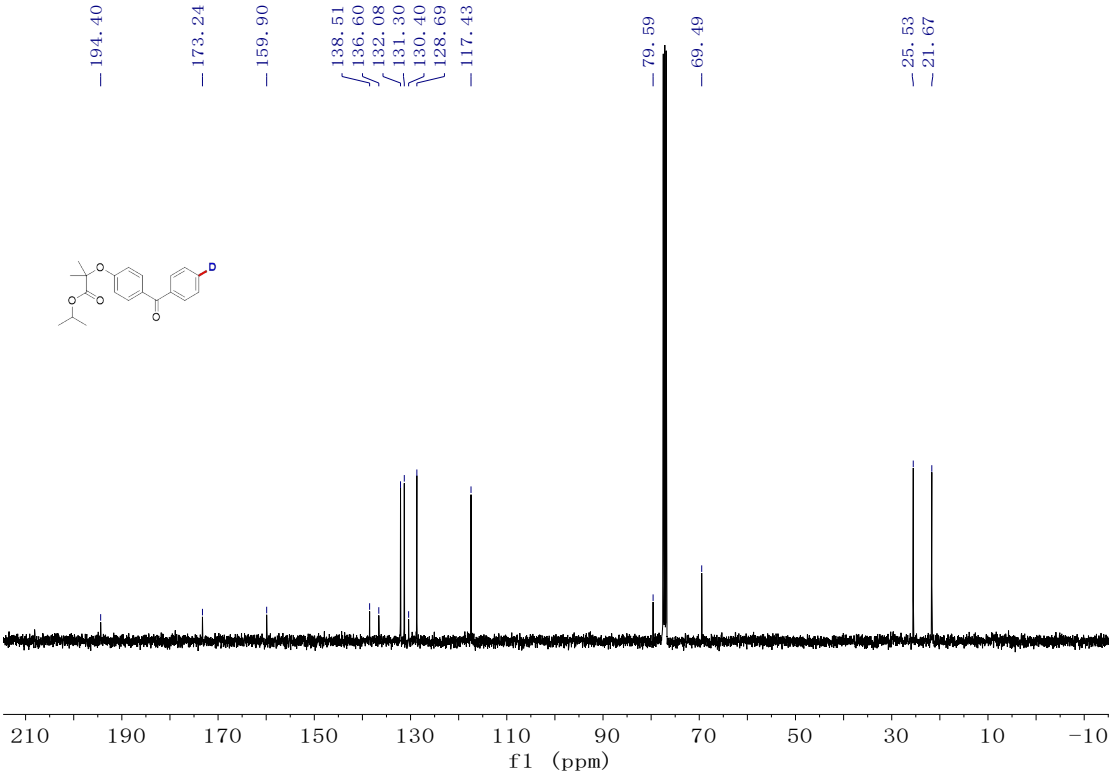

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4w**



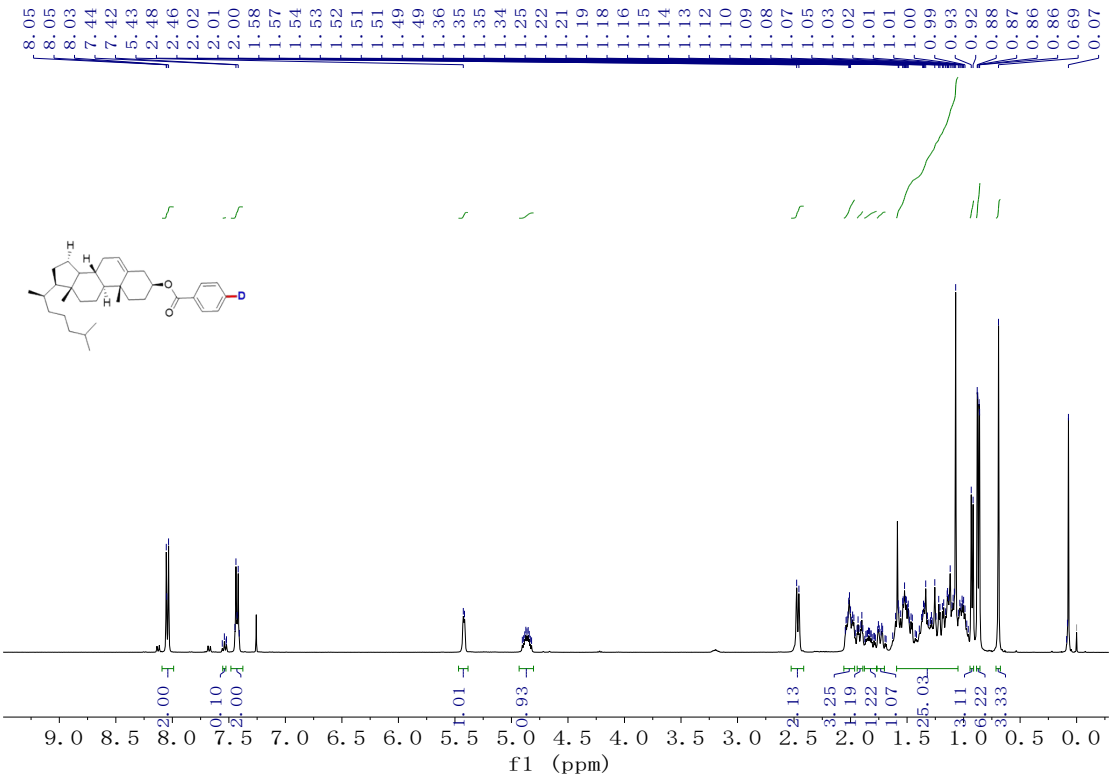
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4x**



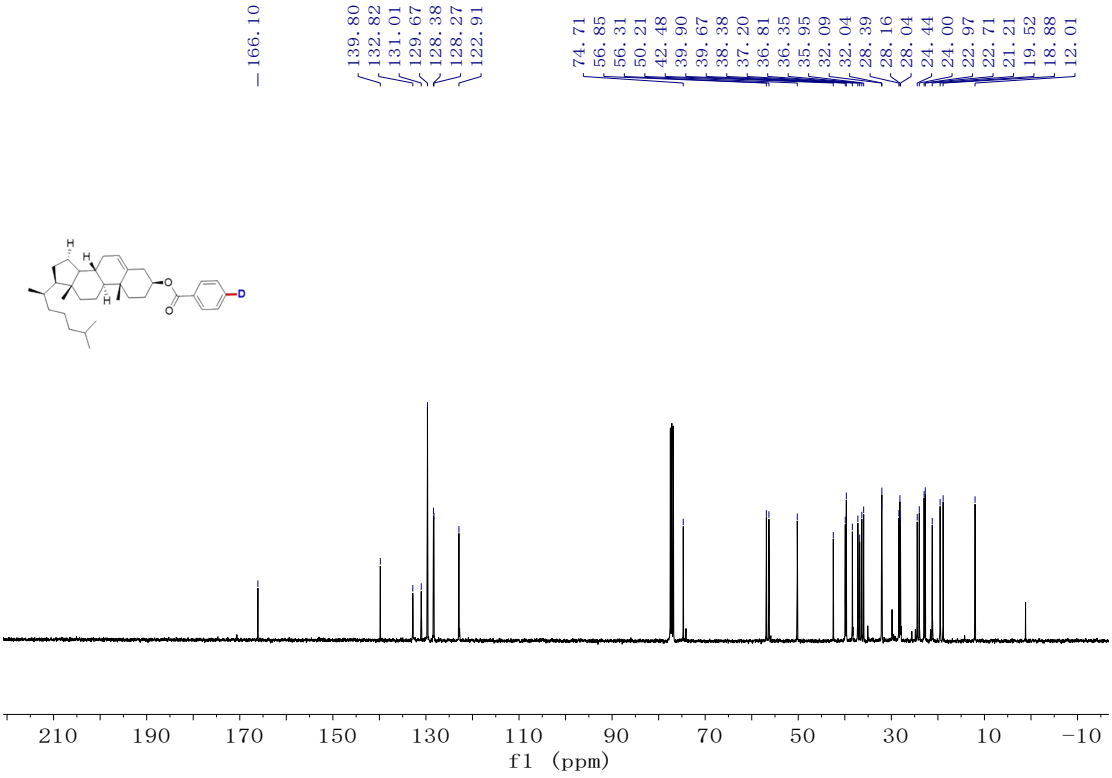

$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4x**



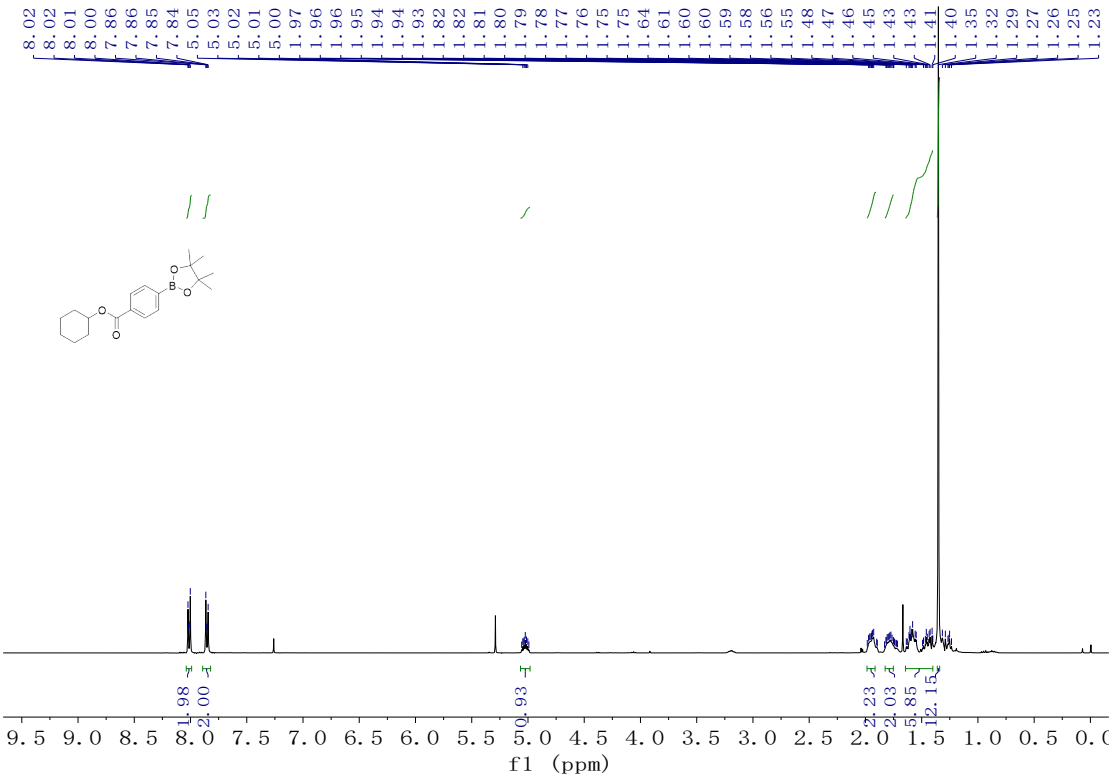
$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **4y**




$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **4y**



$^{1}$H NMR spectrum (400 MHz, $CDCl_3$) of compound **1u**




$^{13}$C NMR spectrum (101 MHz, $CDCl_3$) of compound **1u**