

## Supporting Information

# Acetylation of Alcohols and Amines under Visible Light Irradiation: Diacetyl as an Acylation Reagent and Photosensitizer

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## **General information**

Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The <sup>1</sup>H NMR (400 MHz) and <sup>13</sup>C NMR (100 MHz) and <sup>19</sup>F NMR (376 MHz) data were recorded with CDCl<sub>3</sub> or DMSO-d6 as solvent at room temperature unless specified otherwise. The chemical shifts ( $\delta$ ) are reported in ppm and coupling constants ( $J$ ) in Hz. <sup>1</sup>H NMR spectra was recorded with tetramethylsilane ( $\delta$ = 0.00 ppm) as internal reference; <sup>13</sup>C NMR spectra was recorded with CDCl<sub>3</sub> ( $\delta$  = 77.00 ppm) or DMSO ( $\delta$  = 39.50 ppm) as internal reference. IR and HRMS were performed by the State-authorized Analytical Center in Soochow University.

## **Acylation of Alcohols**

To a 25 mL Schlenk tube, alcohols (0.2 mmol), Cerium (III) trifluoro methanesulfonate (0.01 mmol, 0.05 equiv., 5.9 mg) and 2,3-butanedione (0.8 mmol, 4.0 equiv., 68.9 mg) were dissolved in nitromethane (0.5 mL). Under the irradiation of 40 W blue LEDs, the reaction mixture was stirred at room temperature under oxygen atmosphere for 24 h. After the reaction, the reaction system is quenched with sodium thiosulfate, then poured in brine solution (15 mL) and extracted with ethyl acetate (3 x 20 mL) and then dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (ethyl acetate/ petroleum ether ether) to afford desired products.

## **Acylation of Amines**

To a 25 mL Schlenk tube, amines (0.2 mmol), 2,3-butanedione (1.2 mmol, 6.0 equiv., 103.3 mg) were dissolved in ethanol (95%, 0.5 mL). Under the irradiation of 40 W white LEDs, the reaction mixture was stirred at room temperature under air atmosphere for 6 h. After the reaction, the reaction system is quenched with sodium thiosulfate, then poured in brine solution (15 mL) and extracted with ethyl acetate (3 x 20 mL) and then dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (ethyl acetate/ petroleum ether ether) to afford desired products.

**Table S1 Optimization of reaction conditions<sup>a</sup>**

Entry	Variation from the "standard conditions"	Yield (%) <sup>b</sup>
1	none	90
2	In the dark	< 5
3	MeCN instead of $\text{MeNO}_2$	71
4	Cyclohexane instead of $\text{MeNO}_2$	62
5	EA instead of $\text{MeNO}_2$	56
6	Acetone instead of $\text{MeNO}_2$	56
7	DMF instead of $\text{MeNO}_2$	< 5
8	DMSO instead of $\text{MeNO}_2$	< 5
9	$^t\text{BuOH}$ instead of $\text{MeNO}_2$	< 5
10	DCM instead of $\text{MeNO}_2$	40
11	DCE instead of $\text{MeNO}_2$	35
12	air instead of $\text{O}_2$	66
13	$\text{N}_2$ instead of $\text{O}_2$	< 5
14	$\text{AgOAc}$ instead of $\text{Ce}(\text{OTf})_3$	< 5
15	$\text{MnCl}_2$ instead of $\text{Ce}(\text{OTf})_3$	27
16	$\text{Fe}_2(\text{SO}_4)_3$ instead of $\text{Ce}(\text{OTf})_3$	< 5
17	$\text{CeCl}_3$ instead of $\text{Ce}(\text{OTf})_3$	< 5
18	$\text{Cu}(\text{OTf})_2$ instead of $\text{Ce}(\text{OTf})_3$	50

<sup>a</sup> Reaction conditions: alcohols **1aa** (0.2 mmol), diacetyl **2** (0.8 mmol), catalyst (5.0 mol%) in solvent (0.5 mL) irradiation with 40 W blue LEDs at 28 °C for 24 h. <sup>b</sup> Isolated yield.

**Table S2. Optimization for Acylation of Amines.<sup>a</sup>**

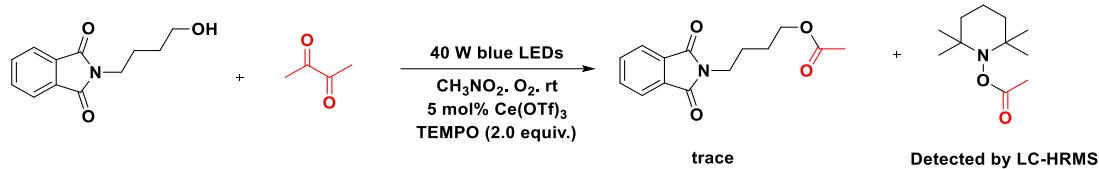
The reaction scheme shows 4-bromoaniline reacting with diacetyl (two acetyl groups) under 40 W white LEDs, Solvent, air, rt conditions to yield N-(4-bromophenyl)-2,2-dimethylpropanamide.

Entry <sup>a</sup>	Solvent	Yield (%) <sup>b</sup>
1	Methanol	57
2	Ethanol (95%)	85
3	Isopropyl alcohol	80
4	EA	54
5	Acetone	45
6	2-Butanone	52
7	DMF	61
8	CHCl <sub>3</sub>	30
9	CCl <sub>4</sub>	34
10	Benzene	71

<sup>a</sup>Reaction conditions: Under air, amine (0.2 mmol), diacetyl (6.0 equiv.) in solvent (0.5 mL) irradiation with 40 W white LEDs at ambient temperature for 6h, the reaction completed monitored by TLC. <sup>b</sup>isolated yield by column chromatography.

## Mechanistic experiments:

## Radical trapping experiments



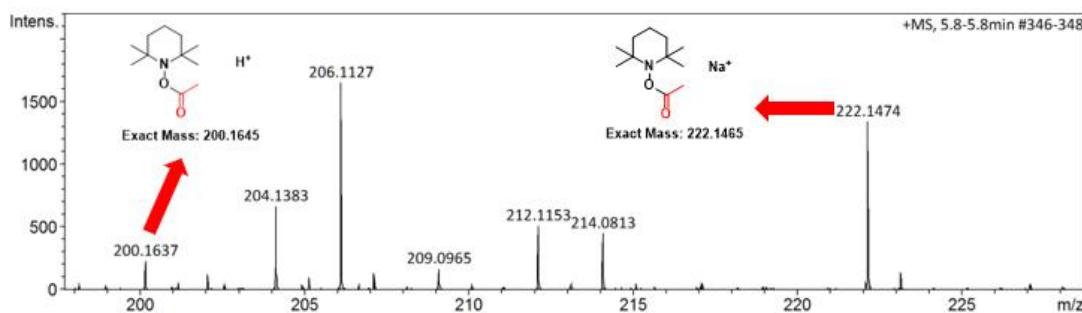
## Display Report

## Analysis Info

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Method 0919-MS-low-METHODS.m  
Sample Name LPC1072-1  
Comment  
Operator bruker  
Instrument micrOTOF-Q III 8228888.20487

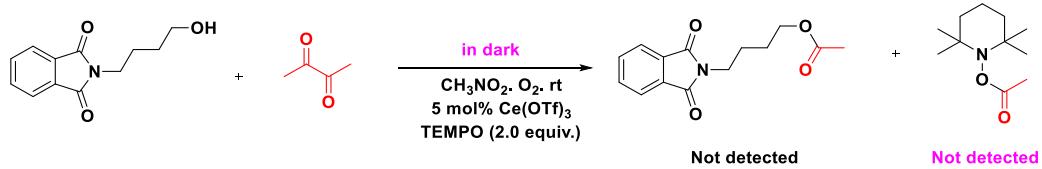
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Scan End	3000 m/z	Set Collision Cell RF	200.0 Vpp	Set Divert Valve	Waste

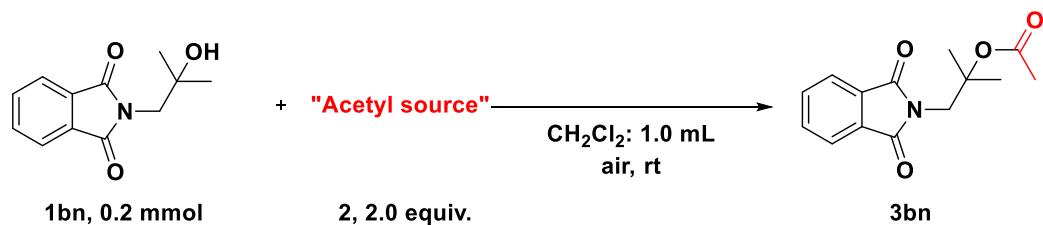


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## Radical trapping experiments **in dark**



### Acetylation of tertiary alcohols using Ac<sub>2</sub>O or AcCl:



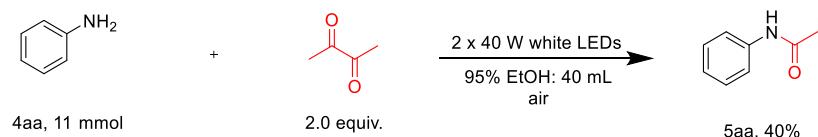
Reference: 1) M. Xie. *J. Chil. Chem. Soc.*, **2011**, *56*, 884-886; 2) S. Ning, L. Zhang, J. Ma, L. Chen, G. Zeng, C. Yang, Y. Zhou, X. Guo, X. Deng, *Org. Biomol. Chem.* **2020**, *18*, 4956-4961. (see Supporting Information)

Acetyl source	(CH <sub>3</sub> CO) <sub>2</sub> O	(CH <sub>3</sub> CO) <sub>2</sub> O + Et <sub>3</sub> N
Yield	Not detected	Not detected

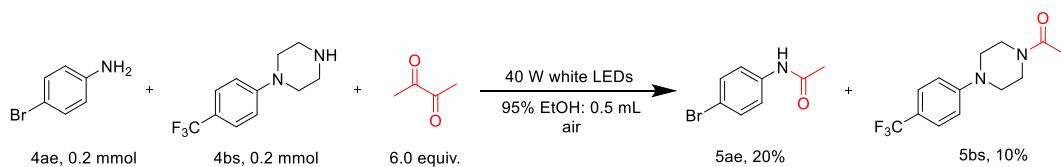
Reference: H. Li, P. Zhang, L. P. Smaga, R. A. Hoffman, J. Chan, *J. Am. Chem. Soc.* **2015**, *137*, 15628-15631. (see Supporting Information)

Acetyl source	CH <sub>3</sub> COCl	CH <sub>3</sub> COCl + Et <sub>3</sub> N
Yield	10%	15%

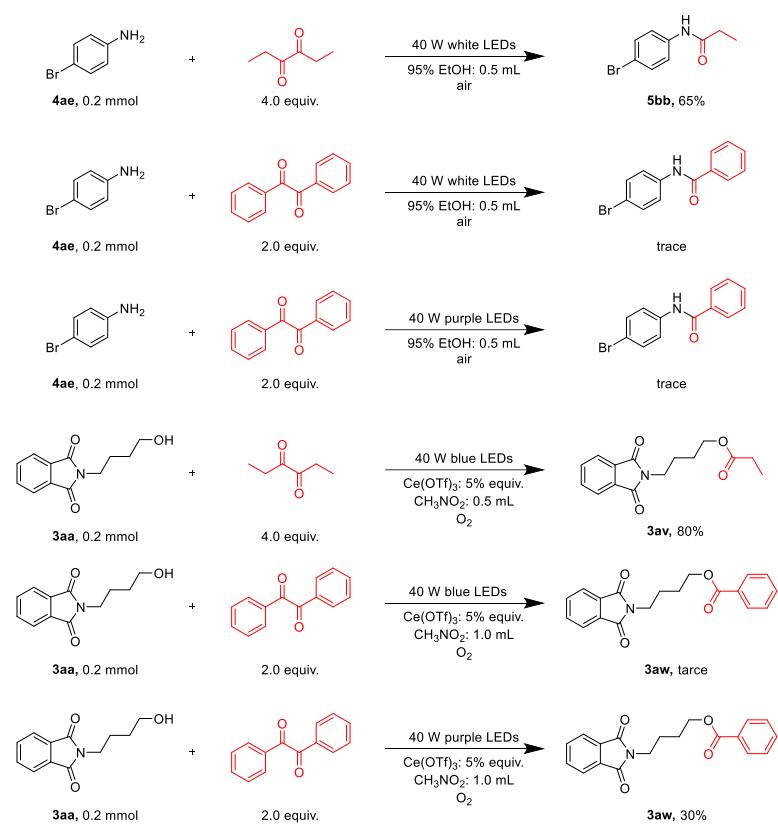
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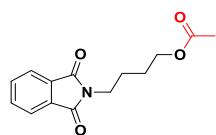
### Compare the reactivity of aliphatic vs. aromatic substrates



## The use of other 1,2-dicarbonyl compounds

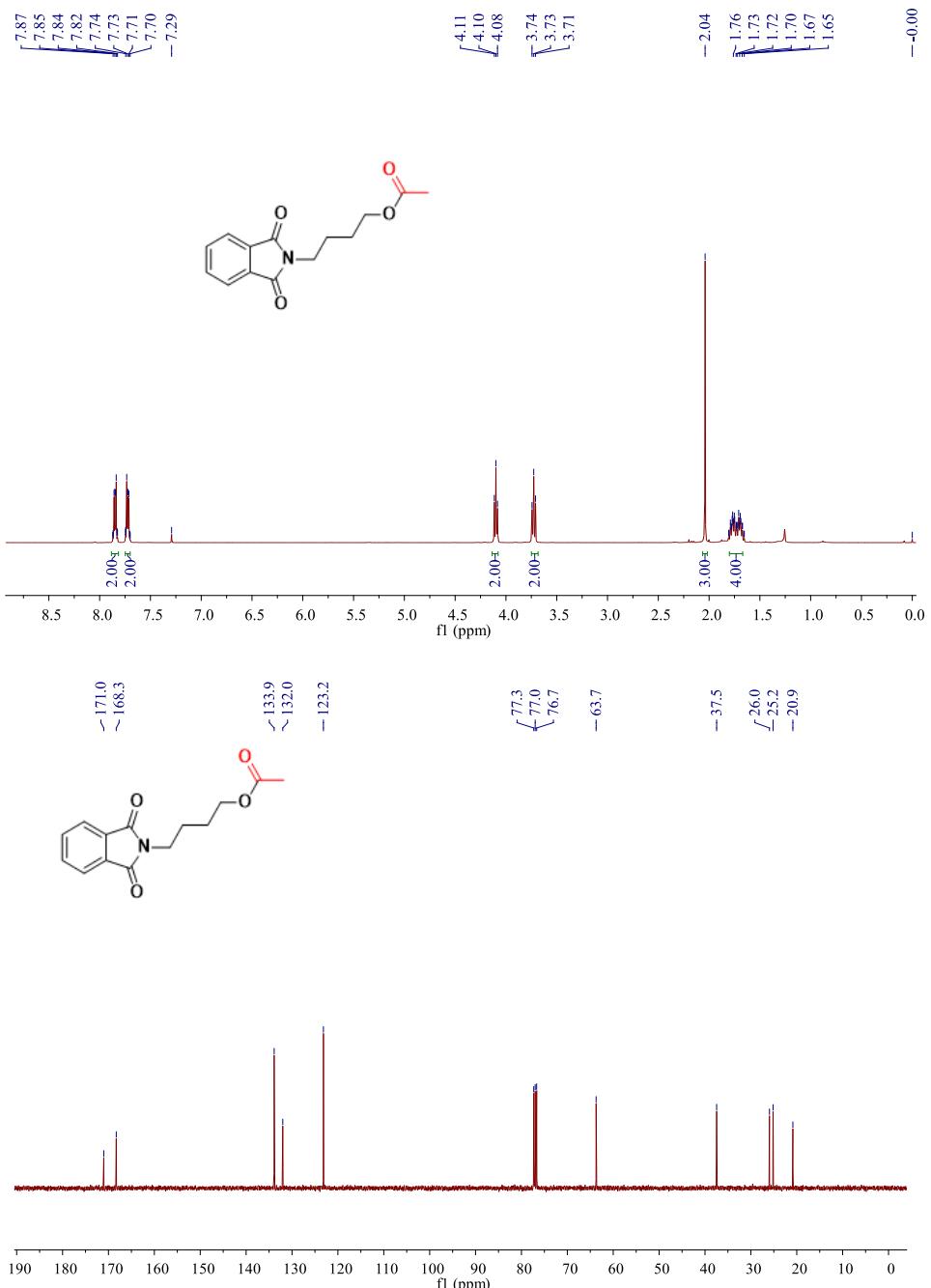


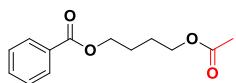
## Spectral Data for Products



### 4-(1,3-Dioxoisindolin-2-yl)butyl acetate (3aa)

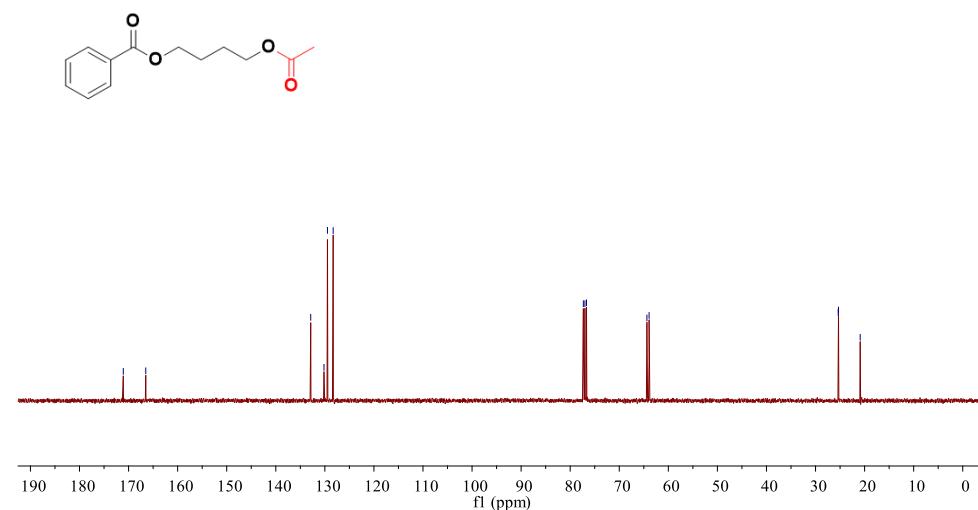
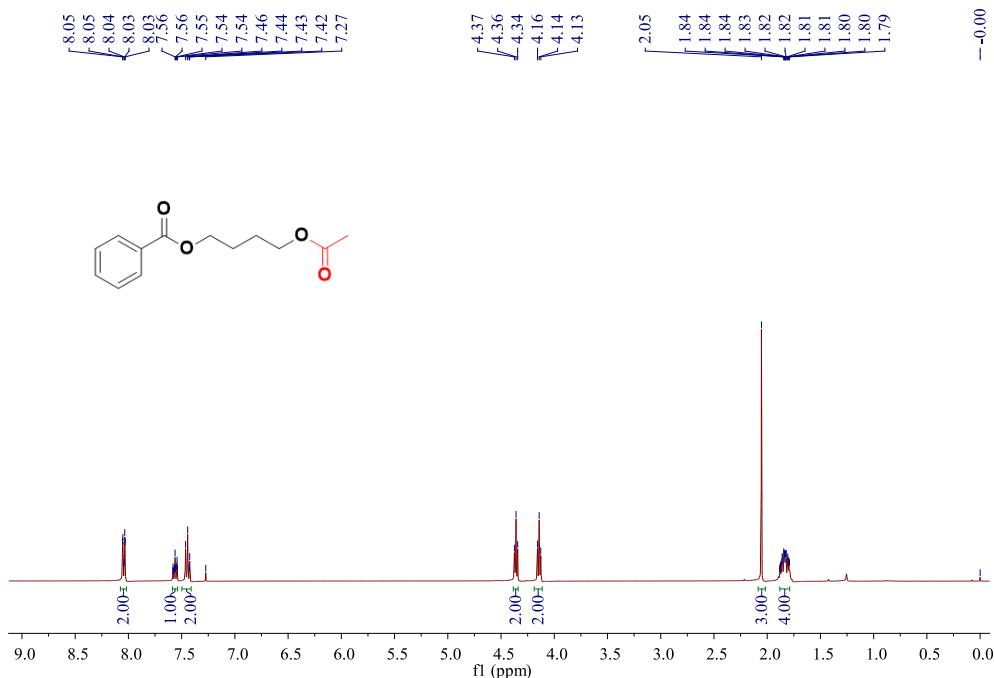
petroleum ether / ethyl acetate = 5:1, white solid, 90% yield (47.0 mg). mp: 63 – 66°C.  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.87 – 7.82 (m, 2H), 7.75 – 7.70 (m, 2H), 4.10 (t,  $J$  = 6.3 Hz, 2H), 3.73 (t,  $J$  = 6.3 Hz, 2H), 2.04 (s, 3H), 1.77 – 1.67 (m, 4H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 168.3, 133.9, 132.0, 123.2, 63.7, 37.5, 26.0, 25.2, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{14}\text{H}_{15}\text{NO}_4+\text{H}^+$ : 276.1230, Found: 276.1227. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  2850, 1751, 1600, 1535, 1460, 1380, 1202, 1008, 719, 620.

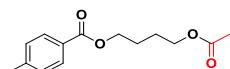




**4-Acetoxybutyl benzoate (3ab)**

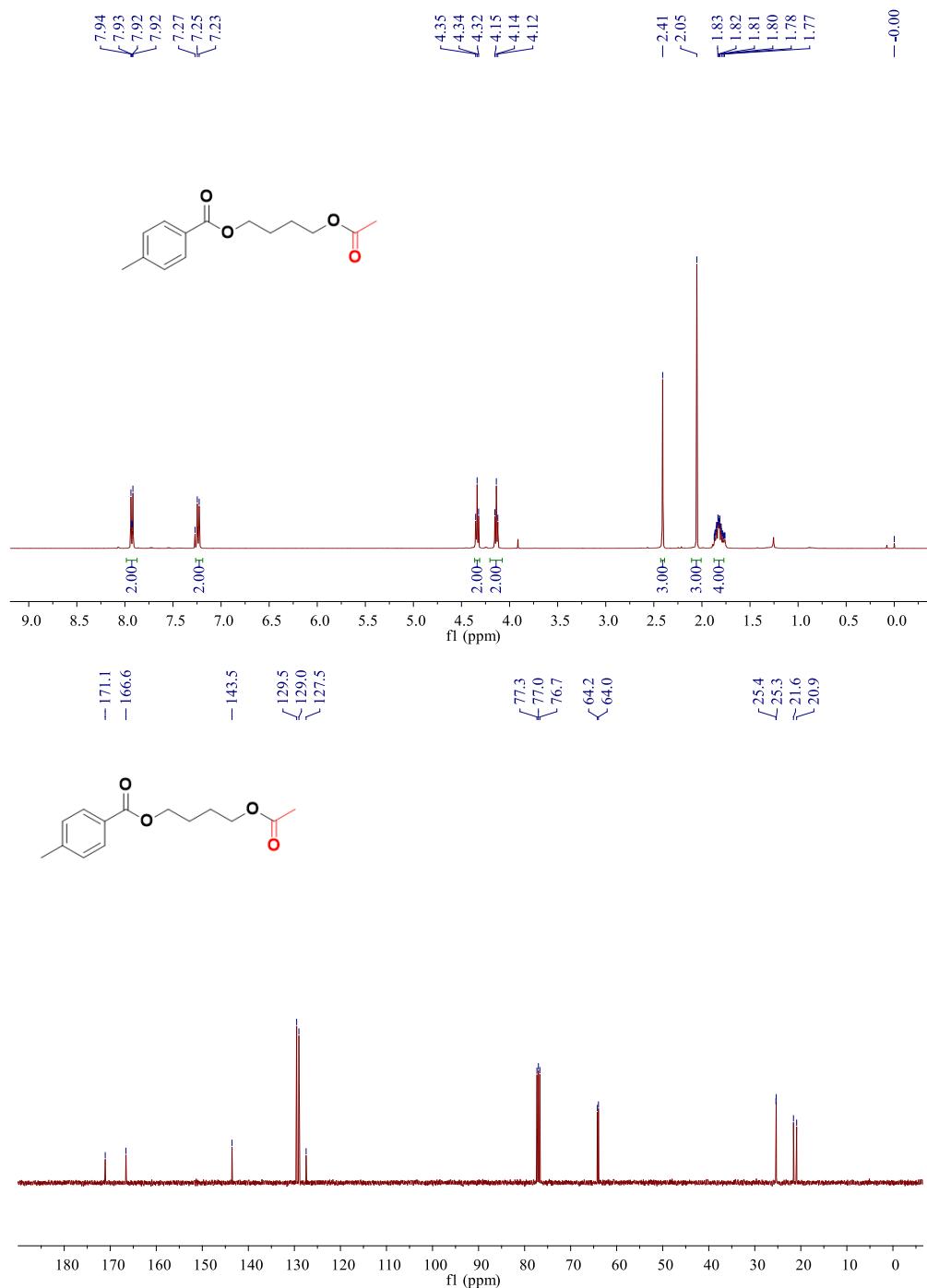
petroleum ether / ethyl acetate = 15:1, colorless oil, 85% yield (40.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.05 – 8.03 (m, 2H), 7.58 – 7.54 (m, 1H), 7.44 – 7.42 (m, 2H), 4.36 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.05 (s, 3H), 1.88 – 1.79 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.1, 166.5, 132.9, 130.2, 129.5, 128.3, 64.4, 63.9, 25.4, 25.3, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>16</sub>O<sub>4</sub>+H<sup>+</sup>: 237.1121, Found: 237.1117. **IR** (neat, cm<sup>-1</sup>): ν 2899, 1736, 1602, 1584, 1451, 1366, 1234, 1176, 1026, 915, 709.

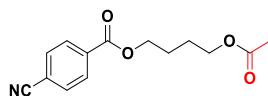




**4-Acetoxybutyl 4-methylbenzoate (3ac)**

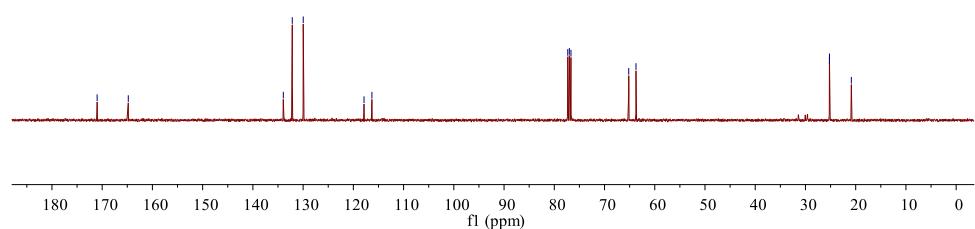
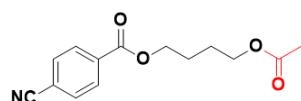
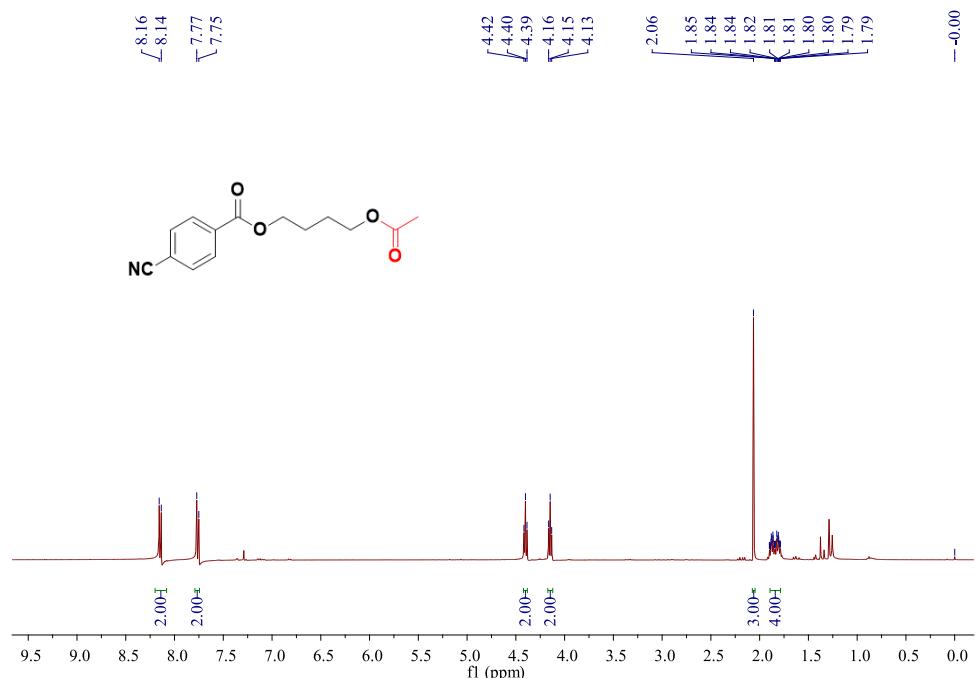
petroleum ether / ethyl acetate = 15:1, colorless oil, 85% yield (40.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.94 – 7.92 (m, 2H), 7.25 – 7.23 (m, 2H), 4.34 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.41 (s, 3H), 2.05 (s, 3H), 1.87 – 1.76 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.1, 166.6, 143.5, 129.5, 129.0, 127.5, 64.2, 64.0, 25.4, 25.3, 21.6, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For.   **IR** (neat, cm<sup>-1</sup>): ν 2924, 1736, 1612, 1577, 1449, 1386, 1271, 1020, 920, 753, 690.

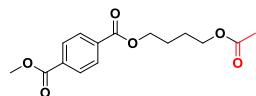




**4-Acetoxylbutyl 4-cyanobenzoate (3ad)**

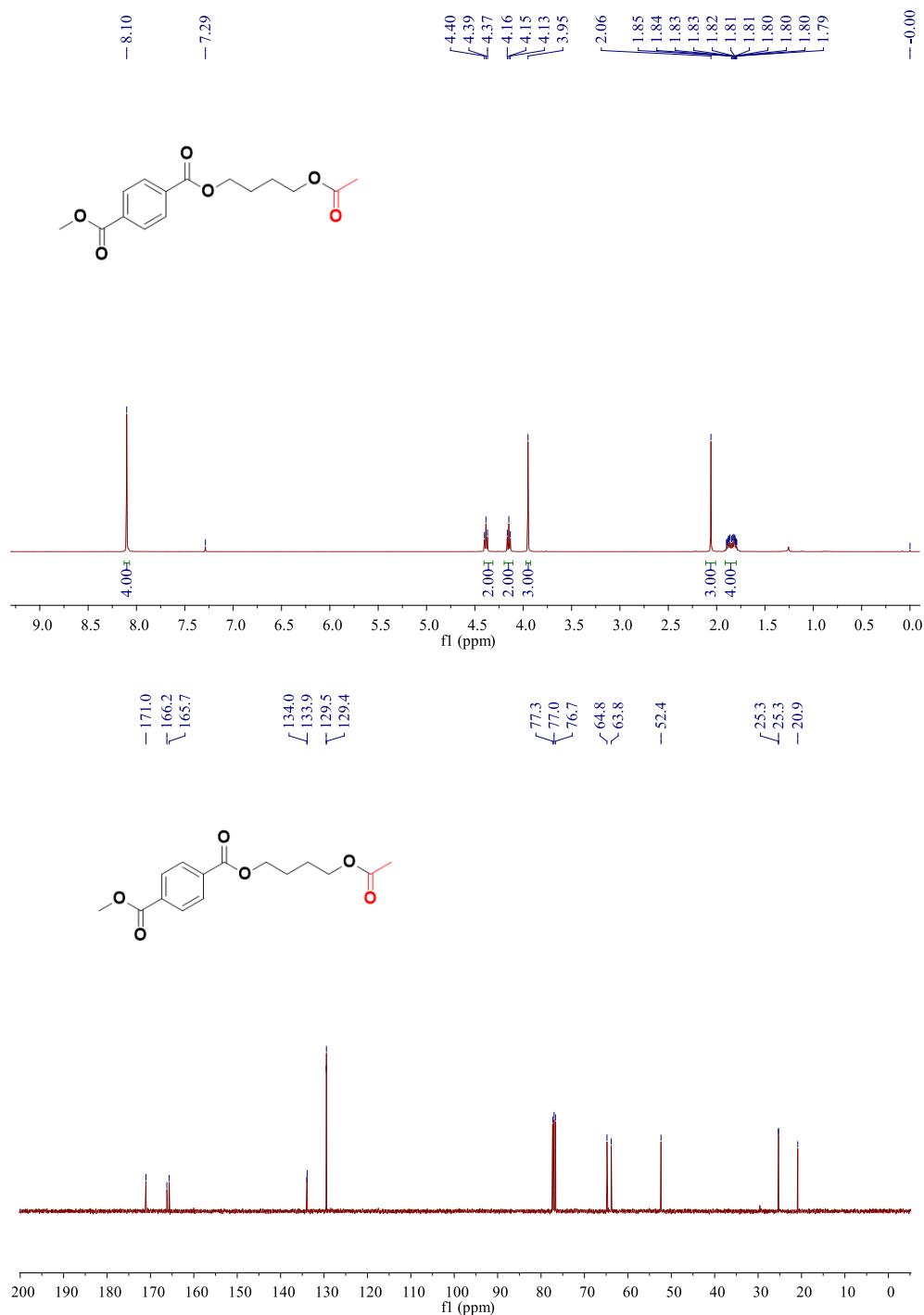
petroleum ether / ethyl acetate = 10:1, colorless oil, 85% yield (44.4 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.15 (d, *J* = 8.6 Hz, 2H), 7.76 (d, *J* = 8.6 Hz, 2H), 4.40 (t, *J* = 6.3 Hz, 2H), 4.15 (t, *J* = 6.3 Hz, 2H), 2.06 (s, 3H), 1.90 – 1.79 (m, 2H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 164.8, 133.9, 132.2, 130.0, 117.9, 116.3, 65.2, 63.7, 25.5, 25.2, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>15</sub>NO<sub>4</sub>+Na<sup>+</sup>: 284.0893, Found: 284.0889. **IR** (neat, cm<sup>-1</sup>): ν 2853, 2232, 1721, 1610, 1569, 1491, 1388, 1237, 1105, 729, 691.

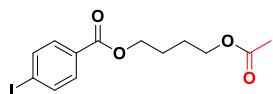




**4-Acetoxybutyl methyl terephthalate (3ae)**

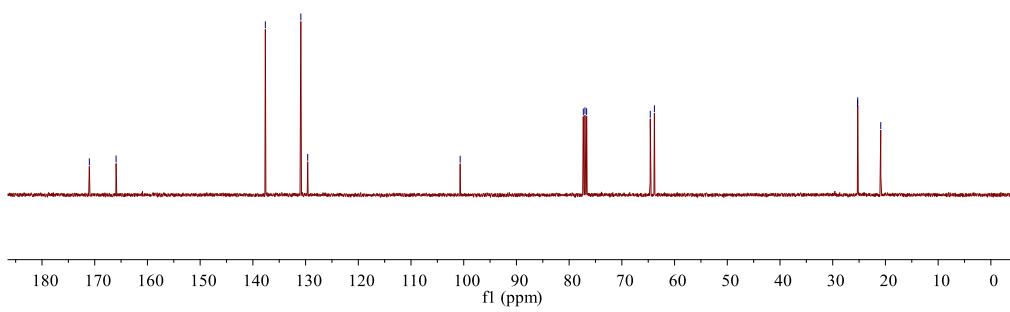
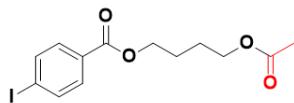
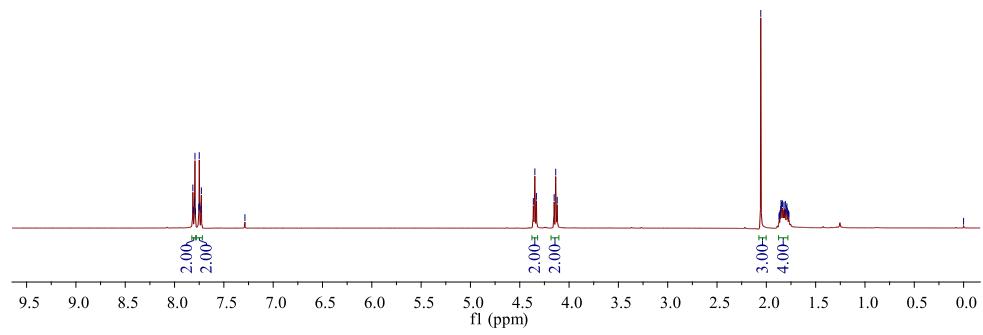
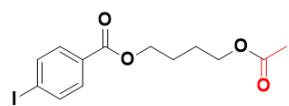
petroleum ether / ethyl acetate = 10:1, yellow oil, 80% yield (46.3 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.10 (s, 4H), 4.39 (t, *J* = 6.3 Hz, 2H), 4.15 (t, *J* = 6.3 Hz, 2H), 3.95 (s, 3H), 2.06 (s, 3H), 1.85 – 1.79 (m, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 171.0, 166.2, 165.7, 134.0, 133.9, 129.5, 129.4, 64.8, 63.8, 52.4, 25.3, 25.3, 20.9. HRMS (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>18</sub>O<sub>6</sub>+H<sup>+</sup>: 295.1176, Found: 295.1173. IR (neat, cm<sup>-1</sup>): ν 2853, 1716, 1615, 1577, 1408, 1268, 1235, 1102, 962, 728.

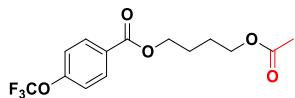




#### **4-Acetoxybutyl 4-iodobenzoate (3af)**

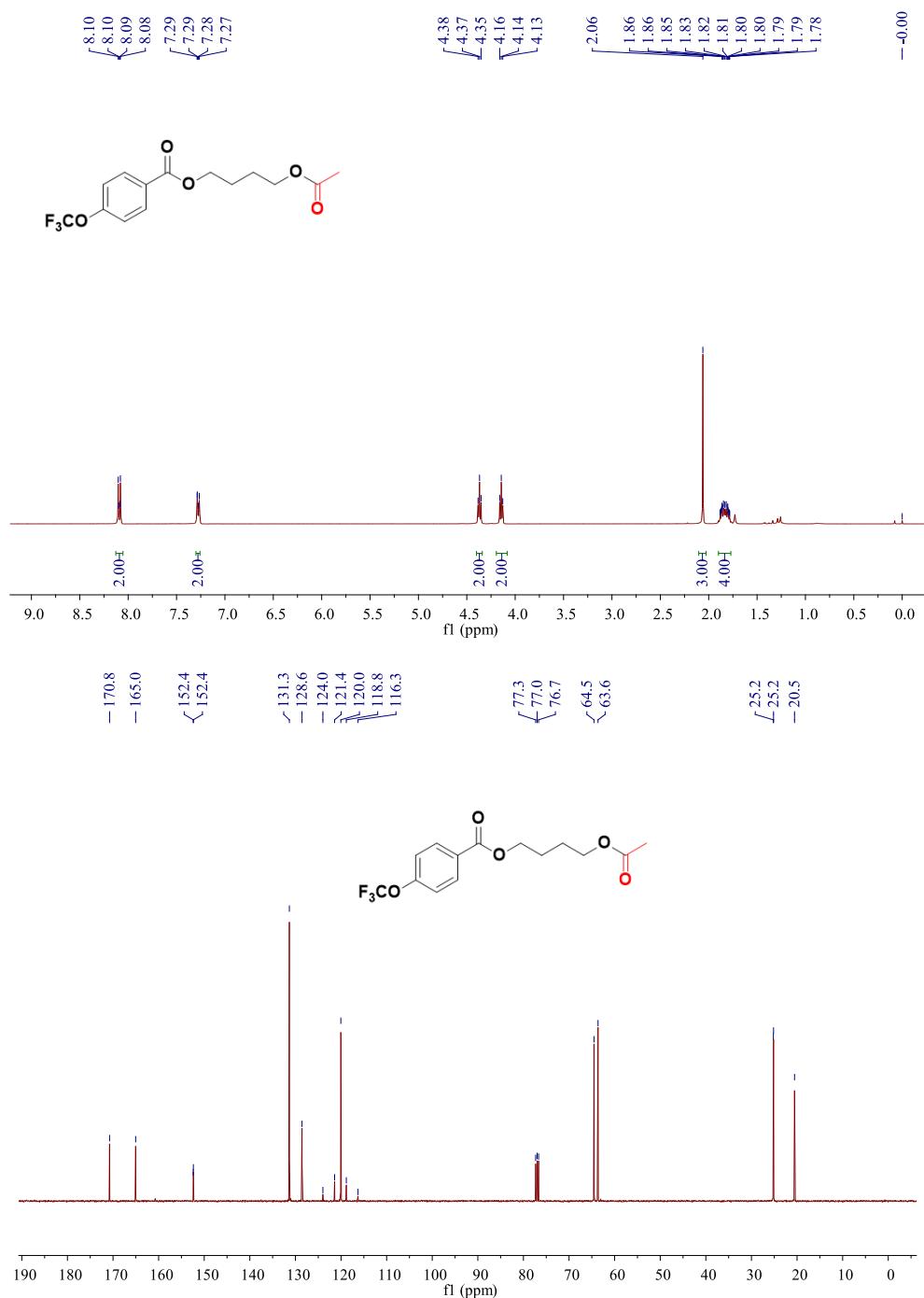
petroleum ether / ethyl acetate = 10:1, yellow oil, 78% yield (60.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.81 – 7.79 (m, 2H), 7.75 – 7.73 (m, 2H), 4.35 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.84 – 1.77 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 165.9, 137.6, 130.9, 129.6, 100.7, 64.6, 63.8, 25.3, 25.3, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>15</sub>IO<sub>4</sub>+H<sup>+</sup>: 363.0088, Found: 363.0081. **IR** (neat, cm<sup>-1</sup>): ν 2853, 1716, 1585, 1470, 1364, 1232, 1006, 920, 752, 682.

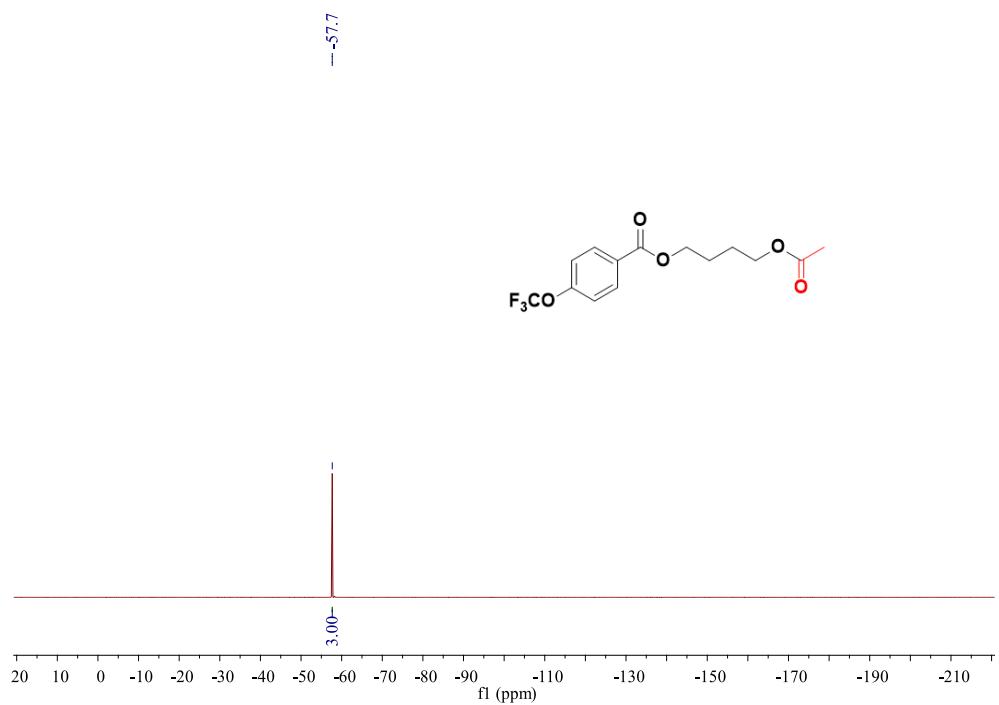


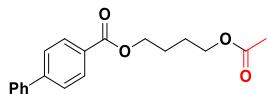


**4-Acetoxylbutyl 4-(trifluoromethoxy)benzoate (3ag)**

petroleum ether / ethyl acetate = 10:1, yellow oil, 74% yield (47.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.10 – 8.08 (m, 2H), 7.29 – 7.27 (m, 2H), 4.37 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.86 – 1.78 (m, 2H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.8, 165.0, 152.4, 131.3, 128.6, 120.1 (q, *J* = 257.0 Hz), 120.0, 64.5, 63.6, 25.2, 25.2, 20.5. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -57.7 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>15</sub>F<sub>3</sub>O<sub>5</sub>+H<sup>+</sup>: 321.0944, Found: 321.0940. **IR** (neat, cm<sup>-1</sup>): ν 2858, 1721, 1607, 1506, 1470, 1388, 1242, 1161, 951, 708, 634.

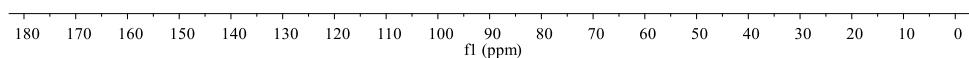
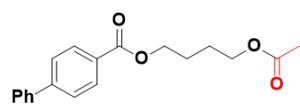
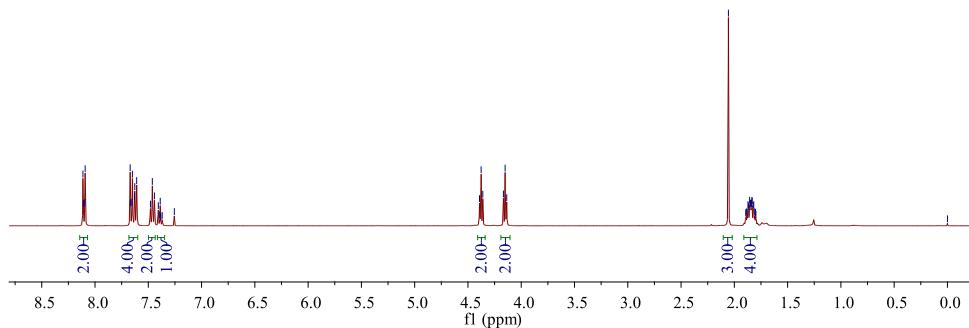
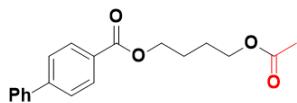


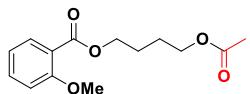




**4-Acetoxylbutyl [1,1'-biphenyl]-4-carboxylate (3ah)**

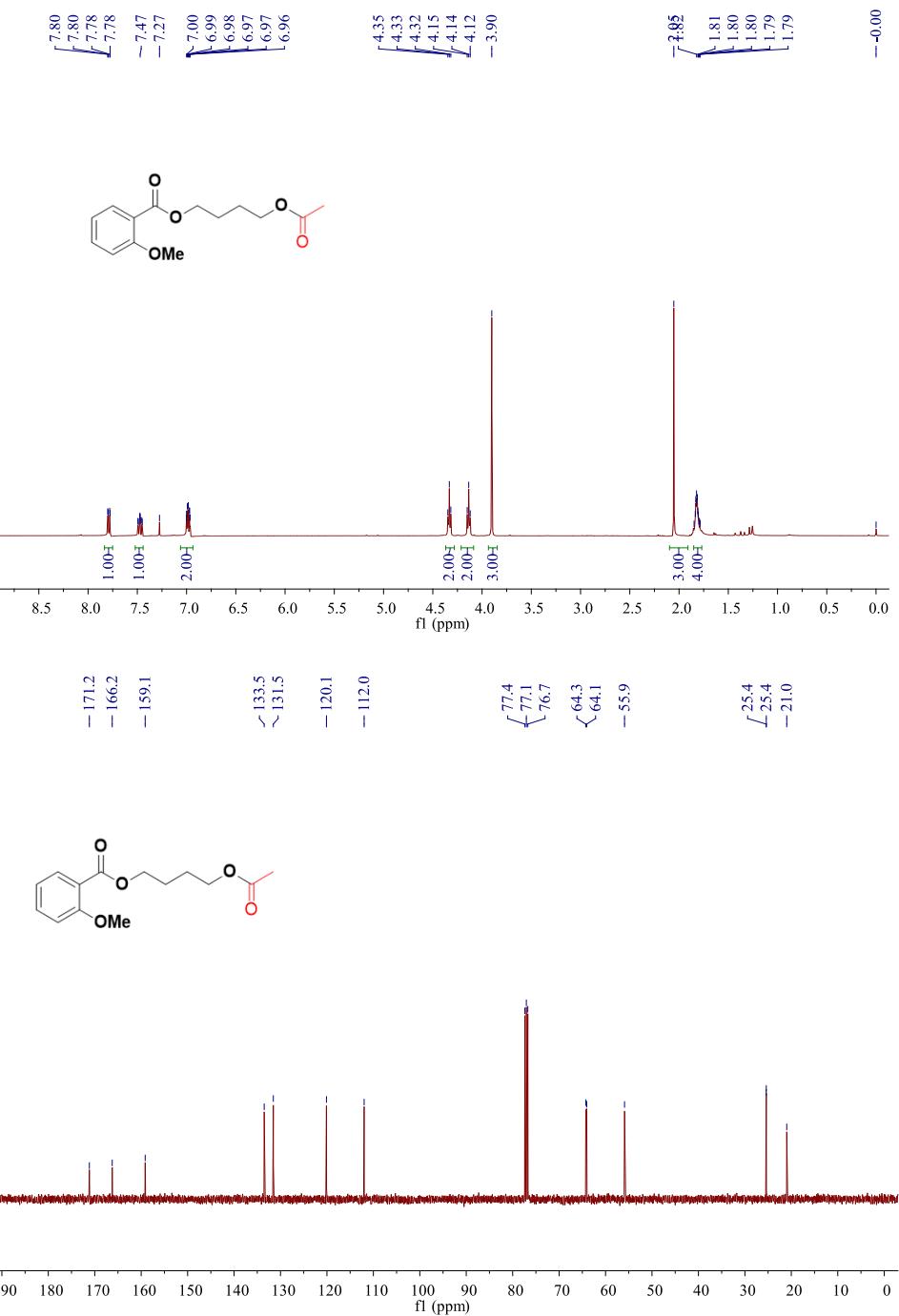
petroleum ether / ethyl acetate = 10:1, colorless oil, 80% yield (49.9 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.11 – 8.09 (m, 2H), 7.67 – 7.61 (m, 4H), 7.48 – 7.44 (m, 2H), 7.41 – 7.37 (m, 1H), 4.38 (t, *J* = 6.2 Hz, 2H), 4.15 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.89 – 1.80 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.1, 166.4, 145.6, 139.9, 130.0, 129.0, 128.9, 128.1, 127.2, 127.0, 64.4, 63.9, 25.4, 25.4, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>19</sub>H<sub>20</sub>O<sub>4</sub>+H<sup>+</sup>: 313.1434, Found: 313.1429. **IR** (neat, cm<sup>-1</sup>):  $\nu$  2854, 1772, 1713, 1609, 1564, 1487, 1188, 907, 727, 647.

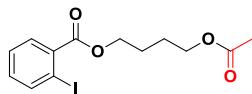




**4-Acetoxybutyl 2-methoxybenzoate (3ai)**

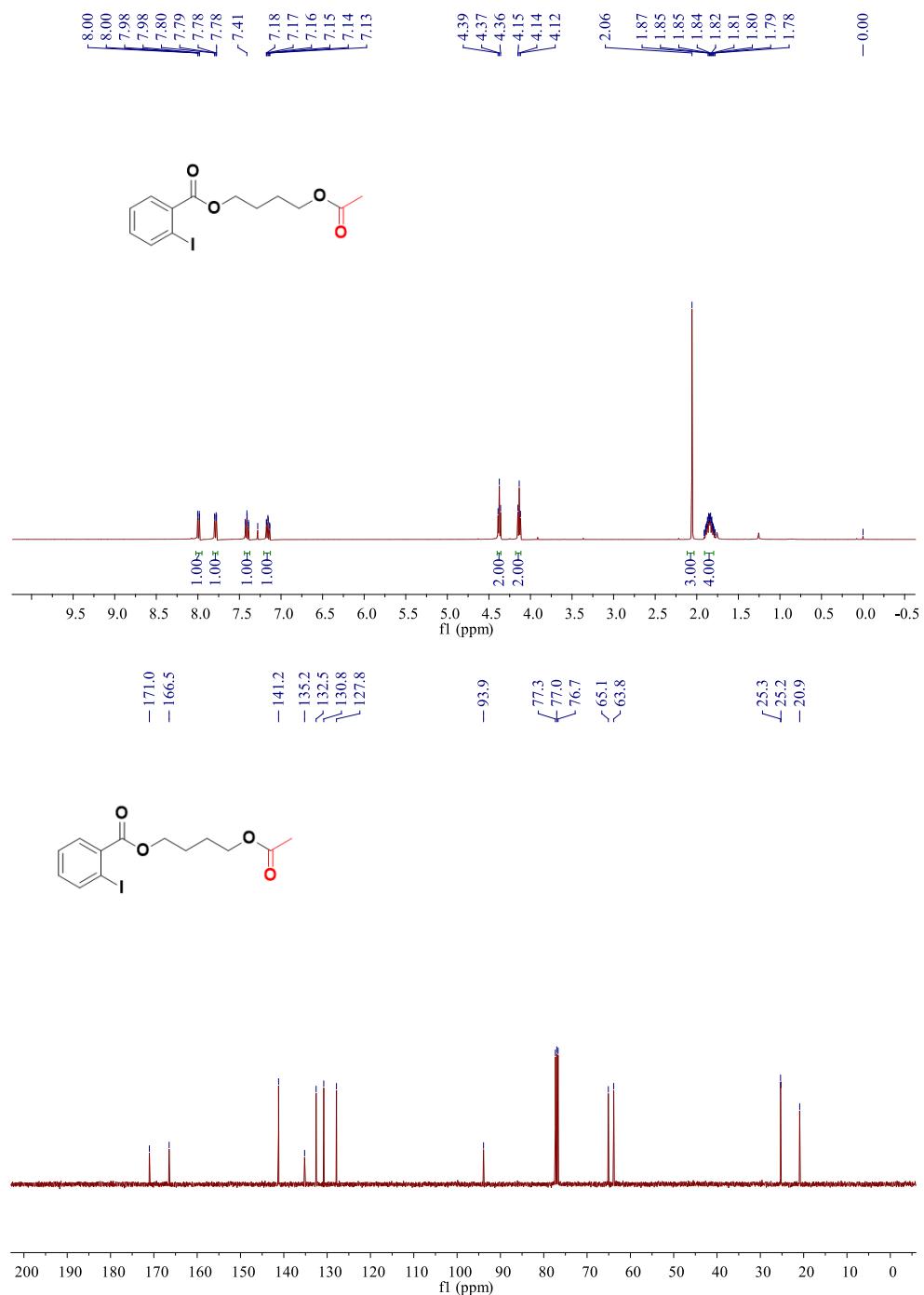
petroleum ether / ethyl acetate = 10:1, colorless oil, 63% yield (33.5 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.80 – 7.78 (m, 1H), 7.49 – 7.45 (m, OH), 7.00 – 6.96 (m, 2H), 4.33 (t, J = 6.1 Hz, 2H), 4.14 (t, J = 6.1 Hz, 2H), 3.90 (s, 3H), 2.05 (s, 3H), 1.85 – 1.79 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.2, 166.2, 159.1, 133.5, 131.5, 120.1, 112, 64.3, 64.1, 55.9, 25.4, 25.4, 21.0. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>18</sub>O<sub>5</sub>+H<sup>+</sup>: 267.1227, Found: 267.1222. **IR** (neat, cm<sup>-1</sup>): ν 2840, 1723, 1601, 1583, 1465, 1240, 1131, 727, 647.

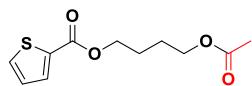




**4-Acetoxymethyl 2-iodobenzoate (3aj)**

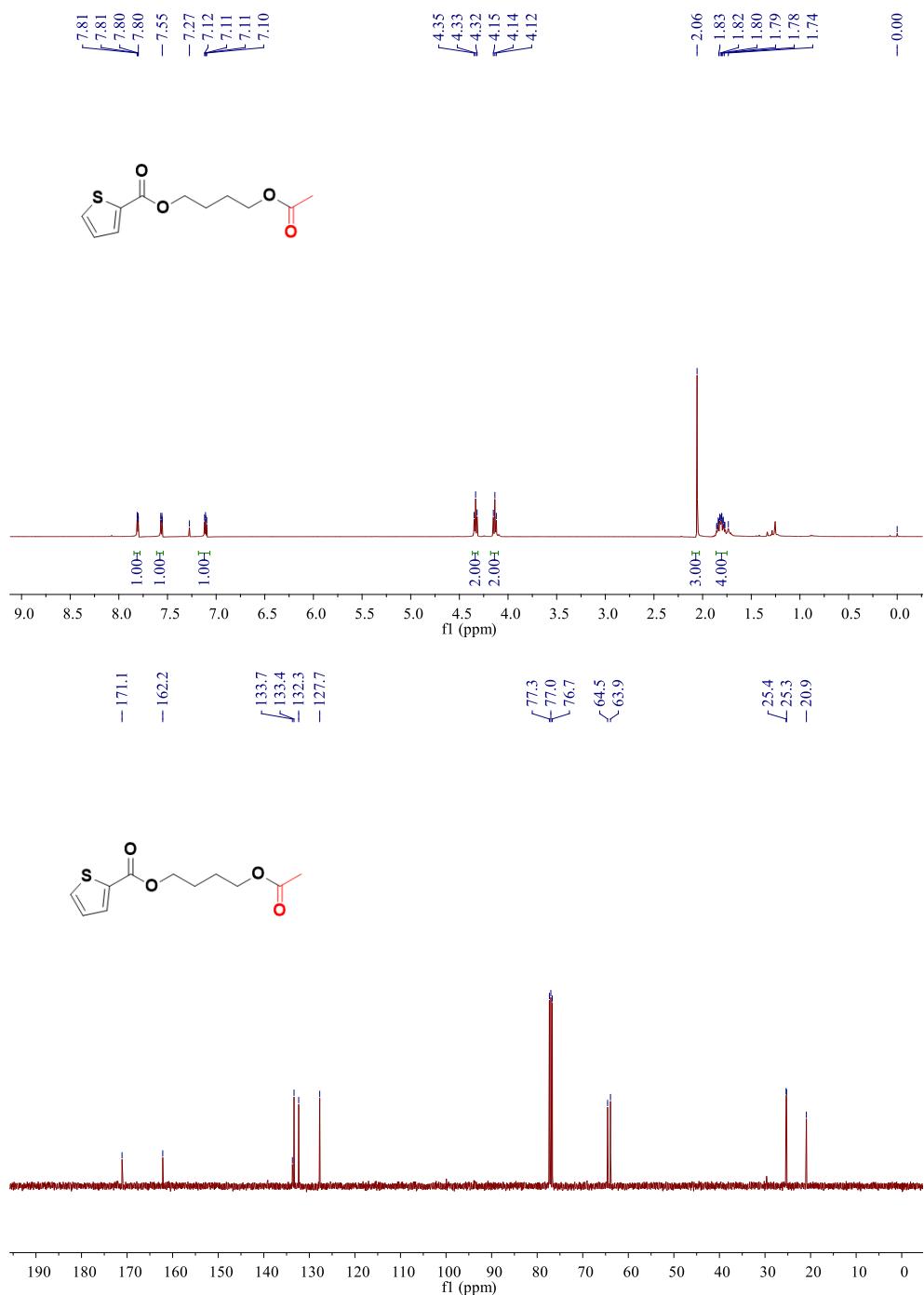
petroleum ether / ethyl acetate = 10:1, colorless oil, 76% yield (55.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.00 – 7.98 (m, 1H), 7.80 – 7.78 (m, 1H), 7.43 – 7.39 (m, 1H), 7.18 – 7.13 (m, 1H), 4.37 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.87 – 1.78 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 166.5, 141.2, 135.2, 132.5, 130.8, 127.8, 93.9, 65.1, 63.8, 25.3, 25.2, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>15</sub>IO<sub>4</sub>+H<sup>+</sup>: 363.0088, Found: 363.0082. **IR** (neat, cm<sup>-1</sup>): ν 2853, 1724, 1583, 1463, 1388, 1234, 1014, 740, 606.

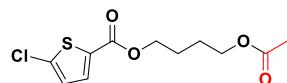




**4-Acetoxymethyl thiophene-2-carboxylate (3ak)**

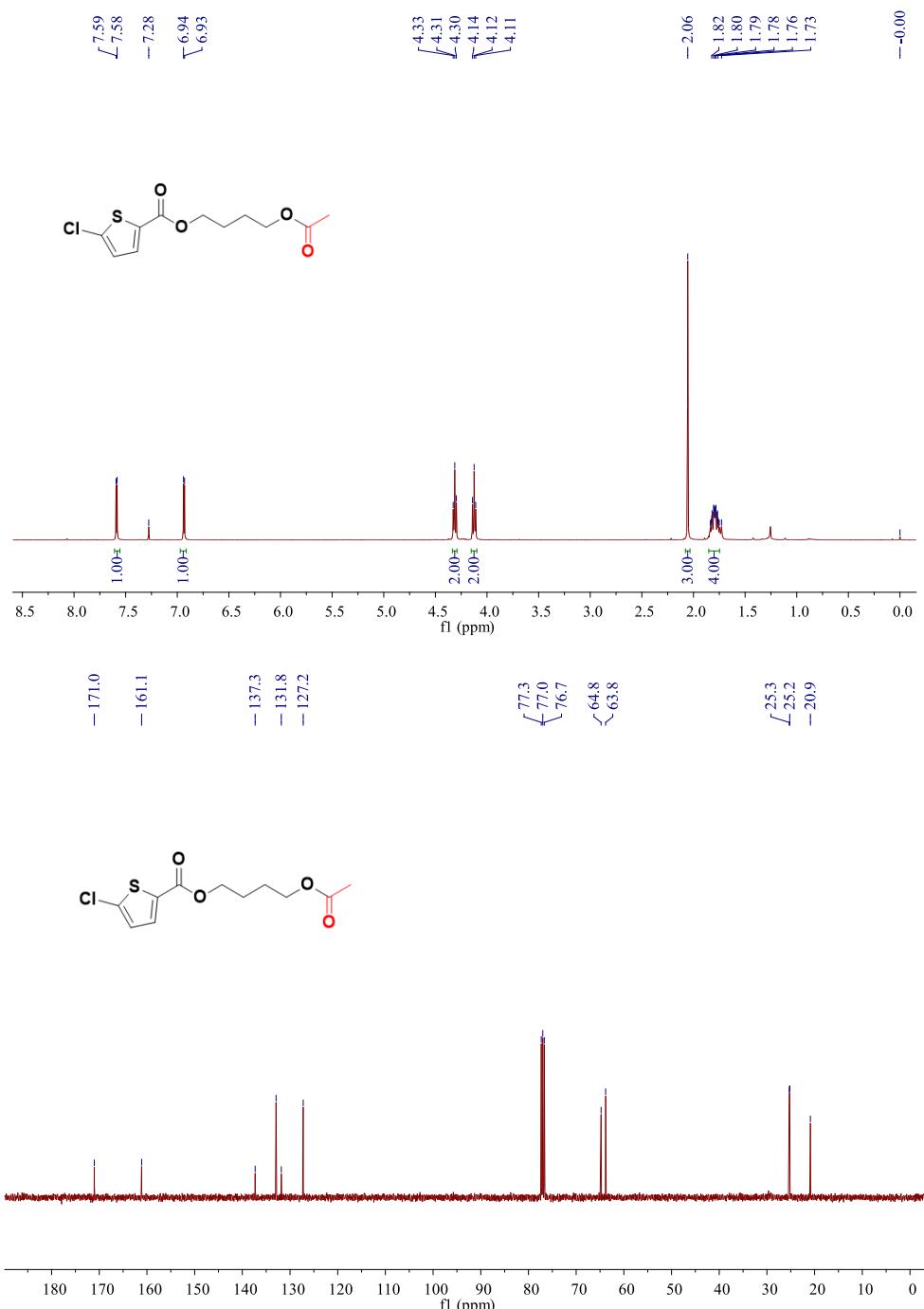
petroleum ether / ethyl acetate = 10:1, yellow oil, 63% yield (30.6 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.81 (dd, *J* = 3.7, 1.2 Hz, 1H), 7.56 (dd, *J* = 5.0, 1.2 Hz, 1H), 7.11 (dd, *J* = 5.0, 3.7 Hz, 1H), 4.33 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.83 – 1.74 (m, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 171.1, 162.2, 133.7, 133.4, 132.3, 127.7, 64.5, 63.9, 25.4, 25.3, 20.9. HRMS (ESI-TOF): Anal Calcd. For. C<sub>11</sub>H<sub>14</sub>O<sub>4</sub>S+H<sup>+</sup>: 243.0686, Found: 243.0684. IR (neat, cm<sup>-1</sup>): ν 2855, 1735, 1525, 1450, 1386, 1257, 1076, 948, 750, 635.

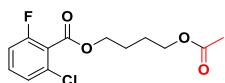




**4-Acetoxylbutyl 5-chlorothiophene-2-carboxylate (3al)**

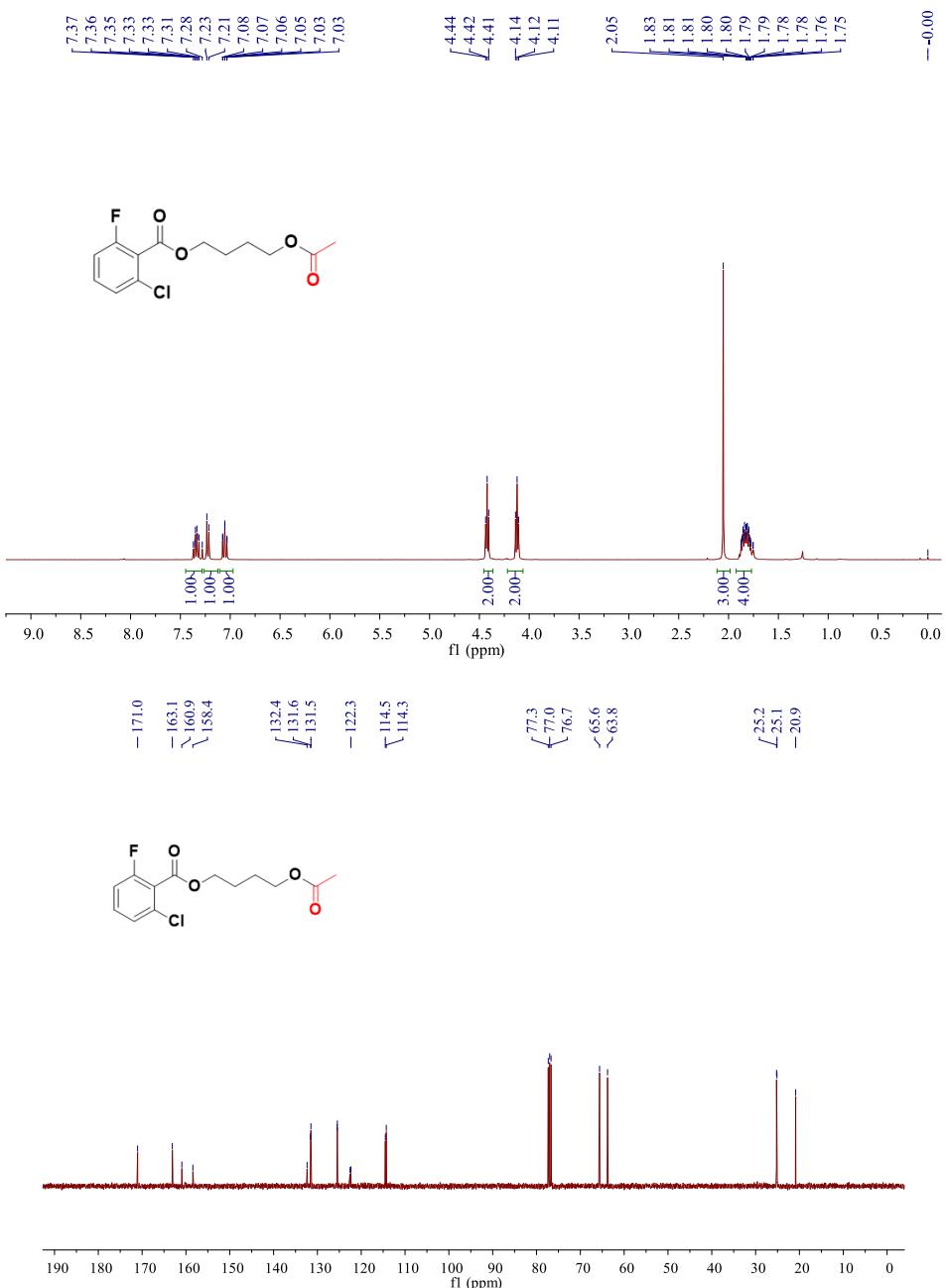
petroleum ether / ethyl acetate = 10:1, yellow oil, 70% yield (38.4 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.59 (d, *J* = 4.0 Hz, 1H), 6.93 (d, *J* = 4.0 Hz, 1H), 4.31 (t, *J* = 6.2 Hz, 2H), 4.12 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.82 – 1.73 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 161.1, 137.3, 132.9, 131.8, 127.2, 64.8, 63.8, 25.3, 25.2, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>11</sub>H<sub>13</sub><sup>35</sup>ClO<sub>4</sub>S+H<sup>+</sup>: 277.0296, Found: 277.0294; Anal Calcd. For. C<sub>11</sub>H<sub>13</sub><sup>37</sup>ClO<sub>4</sub>S+H<sup>+</sup>: 279.0266, Found: 279.0264. **IR** (neat, cm<sup>-1</sup>): ν 3103, 2854, 1736, 1536, 1423, 1233, 1058, 916, 811, 743, 606.



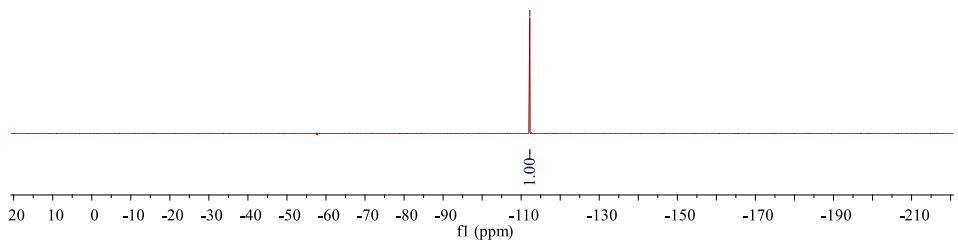
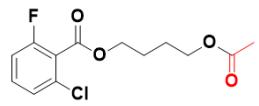


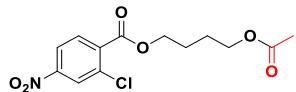
**4-Acetoxybutyl 2-chloro-6-fluorobenzoate (3am)**

petroleum ether / ethyl acetate = 10:1, yellow oil, 92% yield (53.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.31 (m, 1H), 7.23 – 7.2 (m, 1H), 7.08 – 7.03 (m, 1H), 4.42 (t, *J* = 6.2 Hz, 2H), 4.12 (t, *J* = 6.2 Hz, 2H), 2.05 (s, 1H), 1.83 – 1.75 (m, 1H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 163.1, 159.7 (d, *J* = 253.3 Hz), 132.3 (d, *J* = 5.0 Hz), 131.5 (d, *J* = 9.1 Hz), 125.5 (d, *J* = 3.5 Hz), 122.5 (d, *J* = 20.4 Hz), 114.4 (d, *J* = 21.5 Hz), 65.6, 63.8, 25.2, 25.1, 20.9. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -112.2 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>14</sub><sup>35</sup>ClFO<sub>4</sub>+H<sup>+</sup>: 289.0637, Found: 289.0634; Anal Calcd. For. C<sub>13</sub>H<sub>14</sub><sup>37</sup>ClFO<sub>4</sub>+H<sup>+</sup>: 291.0608, Found: 291.0605. **IR** (neat, cm<sup>-1</sup>): ν 2856, 1732, 1602, 1576, 1450, 1387, 1268, 1055, 901, 787, 634.



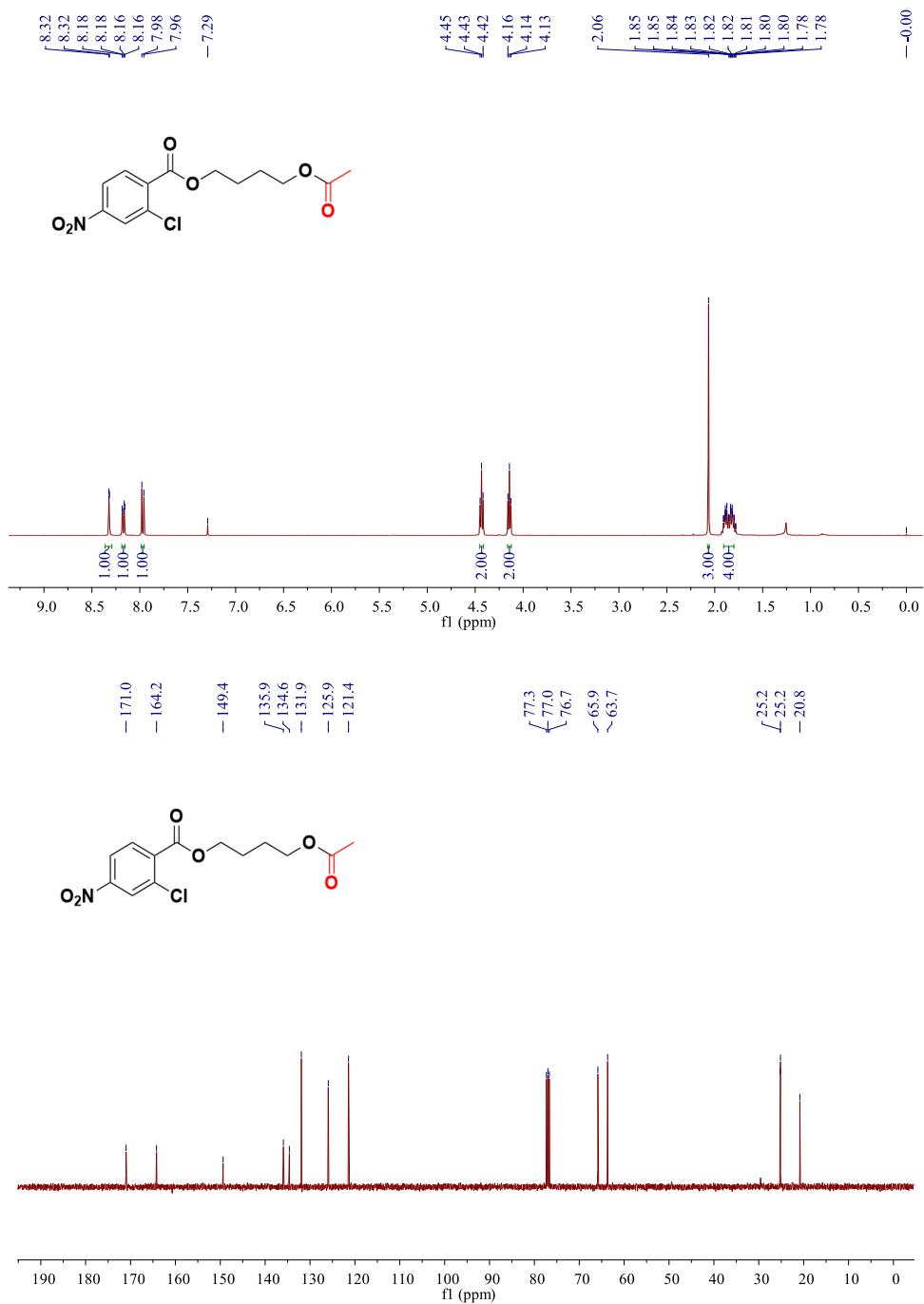
-112.2

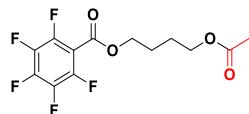




### 3-Acetoxypropyl 2-chloro-4-nitrobenzoate (3an)

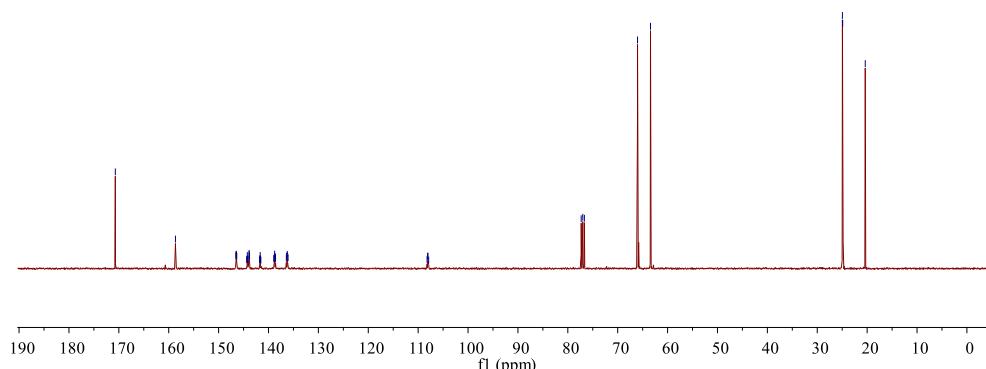
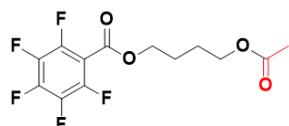
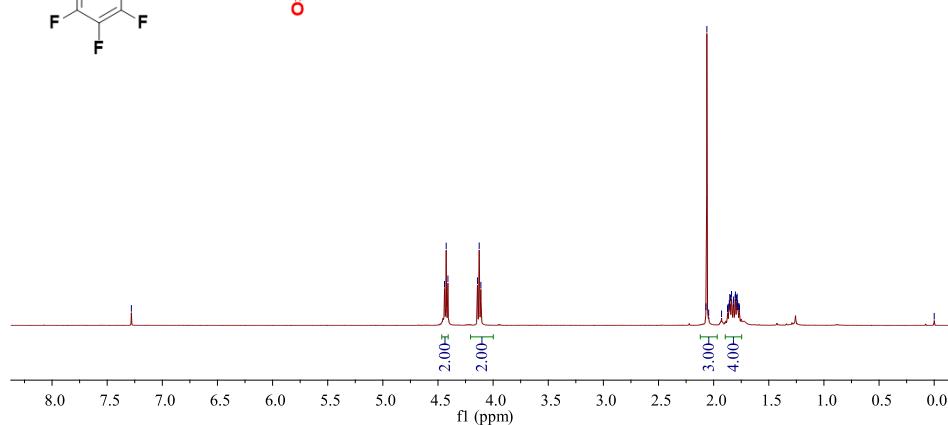
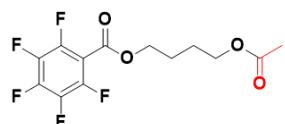
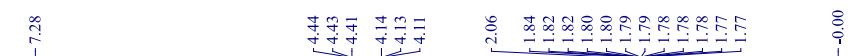
petroleum ether / ethyl acetate = 10:1, yellow oil, 88% yield (52.8 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.32 (d, *J* = 2.1 Hz, 1H), 8.17 (dd, *J* = 8.6, 2.1 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 4.43 (t, *J* = 6.2 Hz, 2H), 4.14 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.85 – 1.78 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.0, 164.2, 149.4, 135.9, 134.6, 131.9, 125.9, 121.4, 65.9, 63.7, 25.2, 25.2, 20.8. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>14</sub><sup>35</sup>ClNO<sub>6</sub>+Na<sup>+</sup>: 338.0402, Found: 338.0400; Anal Calcd. For. C<sub>13</sub>H<sub>14</sub><sup>37</sup>ClNO<sub>6</sub>+Na<sup>+</sup>: 340.0372, Found: 340.0370. **IR** (neat, cm<sup>-1</sup>): ν 2853, 1731, 1600, 1589, 1349, 1236, 1044, 806, 731.

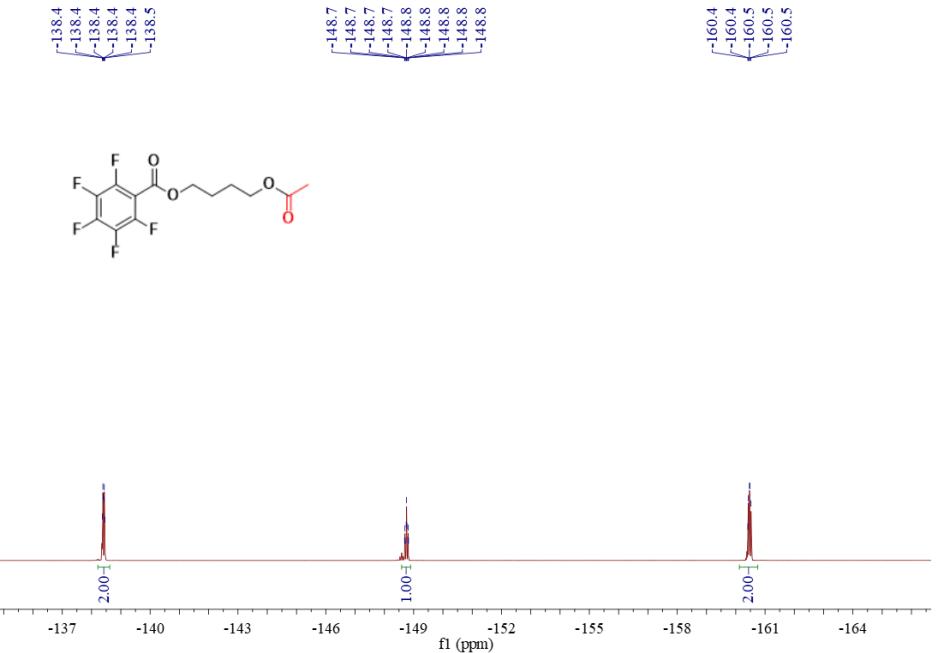


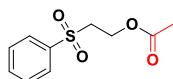


**4-Acetoxybutyl 2,3,4,5,6-pentafluorobenzoate (3ao)**

petroleum ether / ethyl acetate = 10:1, yellow oil, 75% yield (48.9 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 4.43 (t, *J* = 6.2 Hz, 2H), 4.13 (t, *J* = 6.2 Hz, 2H), 2.06 (s, 3H), 1.82 – 1.77 (m, 4H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.7, 158.6, 145.2 (m), 142.9 (m), 137.5 (m), 108.1 (m), 66.0, 63.4, 25.0, 24.9, 20.4. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -138.4 – -138.5 (m, 2F), -148.7 – -148.8 (m, 1F), -160.4 – -160.5 (m, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>11</sub>F<sub>5</sub>O<sub>4</sub>+Na<sup>+</sup>: 349.0470, Found: 349.0466. **IR** (neat, cm<sup>-1</sup>): ν 2859, 1736, 1652, 1524, 1496, 1387, 1222, 1038, 754, 699.

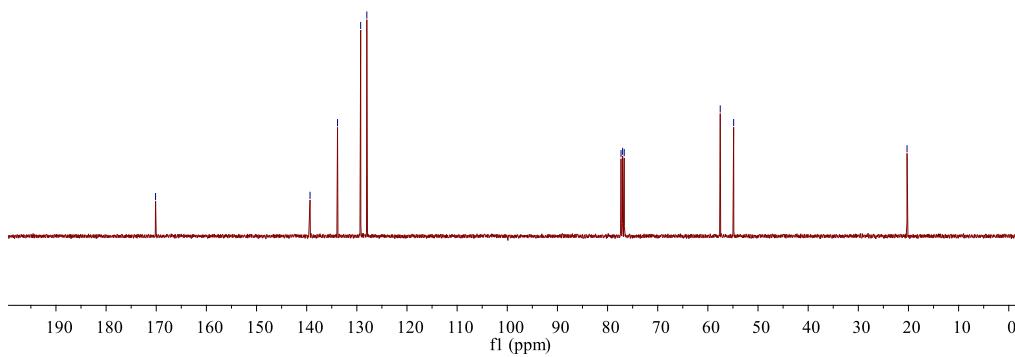
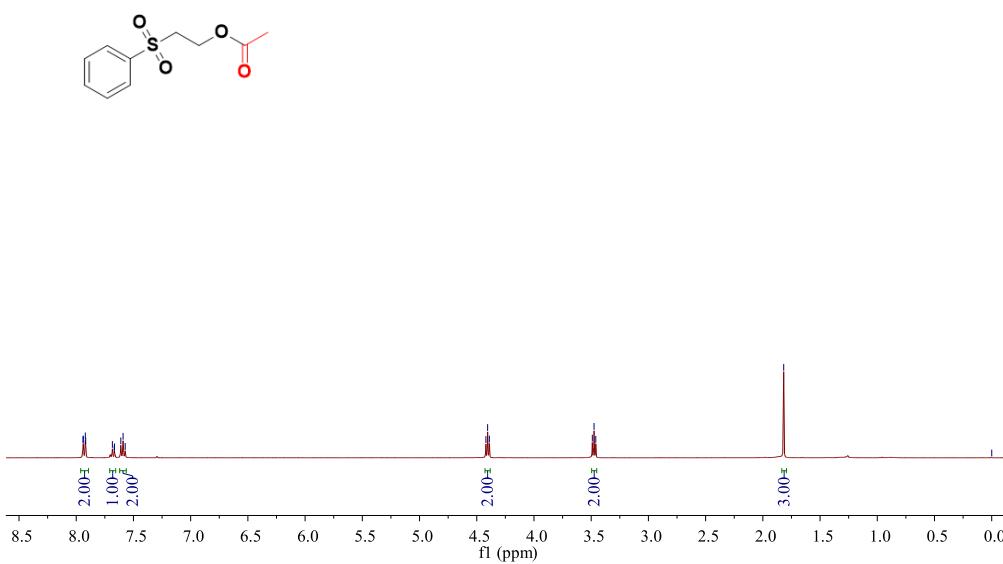


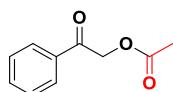




**2-(Phenylsulfonyl)ethyl acetate (3ap)**

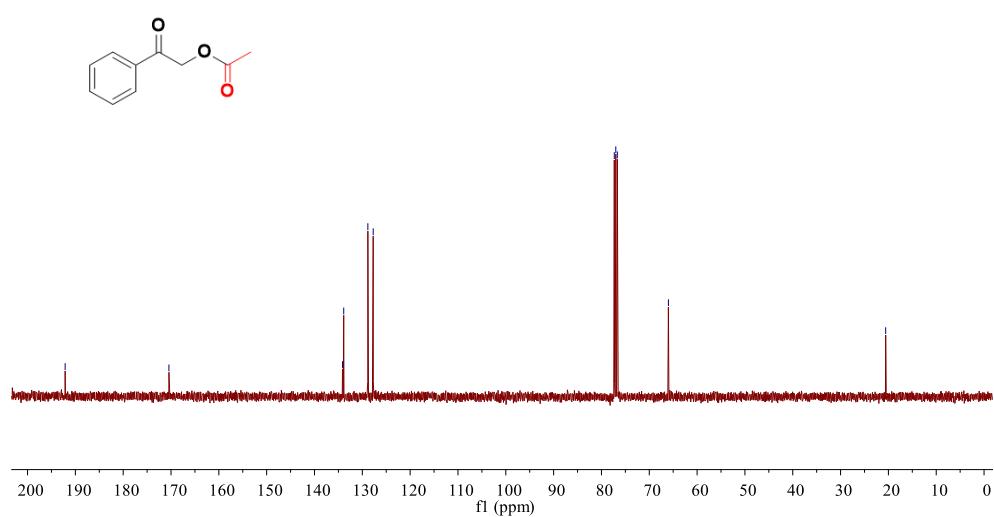
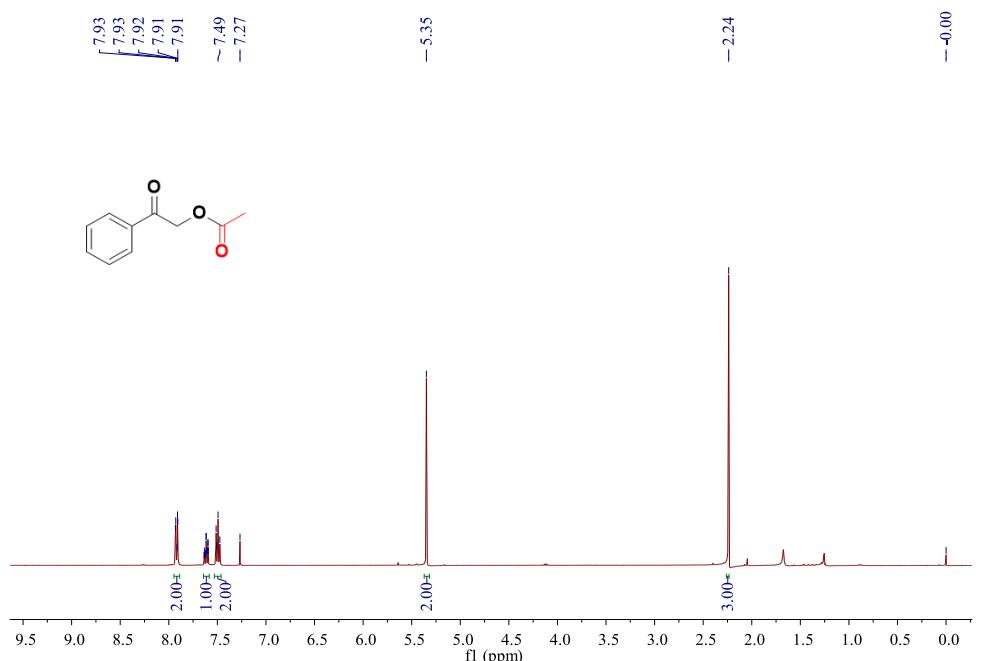
petroleum ether / ethyl acetate = 5:1, colorless oil, 99% yield (45.2 mg).  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.94 – 7.92 (m, 2H), 7.70 – 7.67 (m, 1H), 7.61 – 7.57 (m, 2H), 4.40 (t,  $J$  = 6.1 Hz, 2H), 3.48 (t,  $J$  = 6.1 Hz, 2H), 1.82 (s, 3H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 139.3, 133.9, 129.2, 128.0, 57.5, 54.9, 20.3. **HRMS (ESI-TOF):** Anal Calcd. For.  $\text{C}_{10}\text{H}_{12}\text{O}_4\text{S} + \text{H}^+$ : 229.0529, Found: 229.0525. **IR (neat, cm}^{-1}):**  $\nu$  2900, 1605, 1580, 1350, 1251, 750.

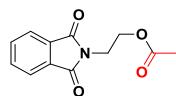




**2-Oxo-2-phenylethyl acetate (3aq)**

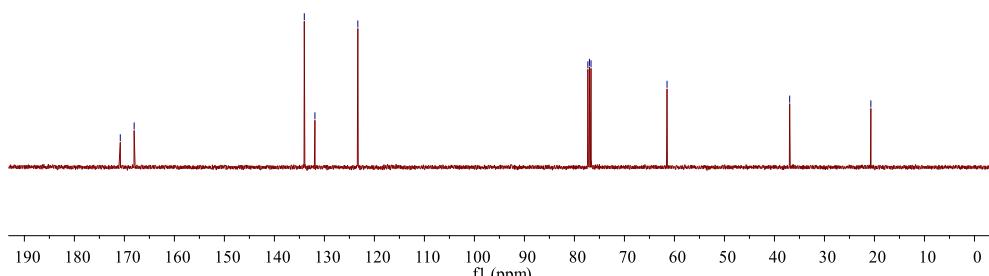
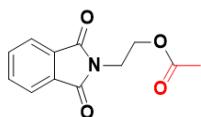
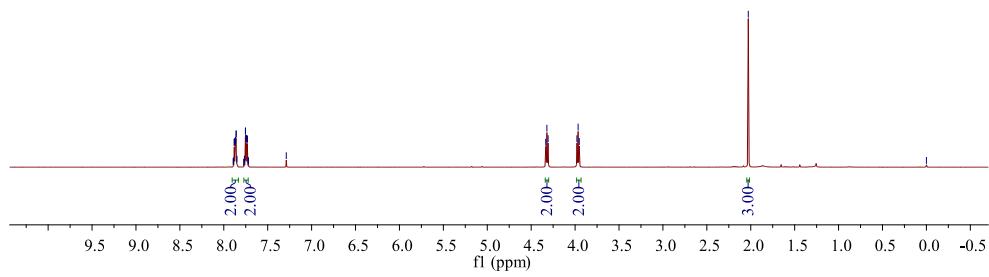
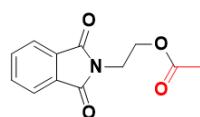
petroleum ether / ethyl acetate = 10:1, yellow oil, 50% yield (33.5 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.93 – 7.91 (m, 1H), 7.63 – 7.59 (m, 1H), 7.51 – 7.47 (m, 2H), 5.35 (s, 2H), 2.24 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 192.1, 170.4, 134.1, 133.9, 128.8, 127.7, 66.0, 20.6. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>10</sub>H<sub>10</sub>O<sub>3</sub>+H<sup>+</sup>: 179.0703, Found: 179.0701. **IR** (neat, cm<sup>-1</sup>): ν 2849, 1748, 1702, 1597, 1450, 1372, 1216, 912, 731, 647.

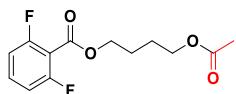




### **2-(1,3-Dioxoisooindolin-2-yl)ethyl acetate (3ar)**

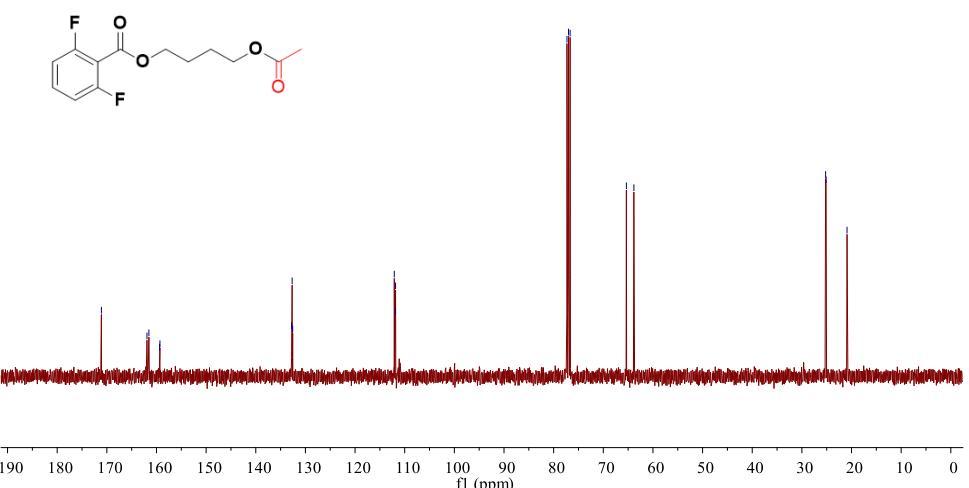
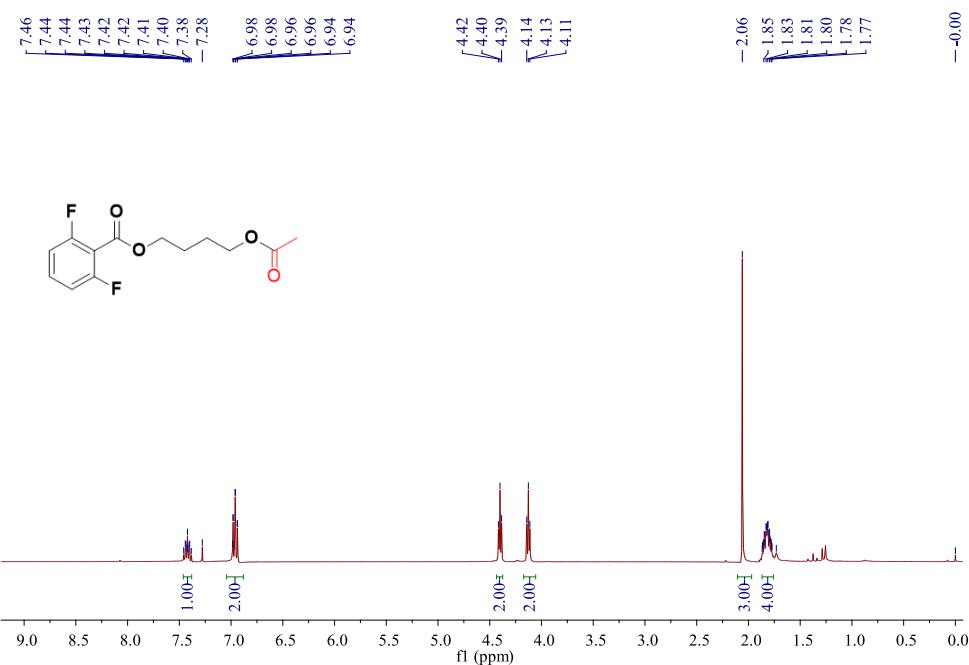
petroleum ether / ethyl acetate = 5:1, white solid, 97% yield (45.1 mg). mp: 77 – 80°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.89 – 7.85 (m, 2H), 7.77 – 7.72 (m, 2H), 4.32 (t, *J* = 5.3 Hz, 2H), 3.97 (t, *J* = 5.3 Hz, 2H), 2.03 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.8, 168.0, 134.0, 131.9, 123.3, 61.5, 37.0, 20.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>12</sub>H<sub>11</sub>NO<sub>4</sub>+H<sup>+</sup>: 234.0761, Found: 234.0758. **IR** (neat, cm<sup>-1</sup>): ν 5825, 1772, 1705, 1615, 1559, 1369, 1273, 1153, 983, 717.

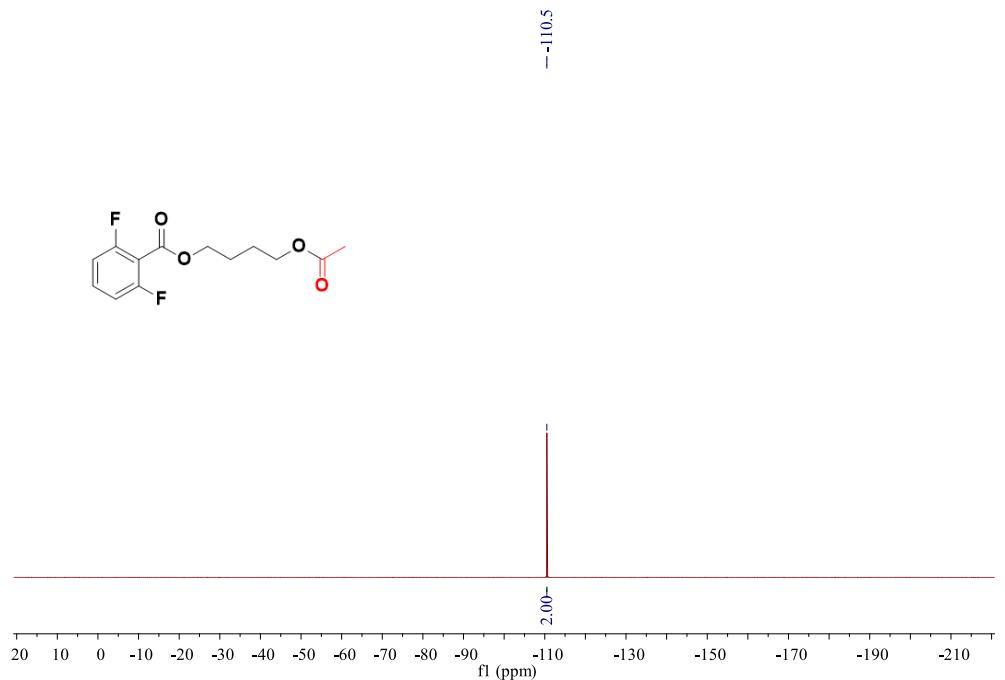


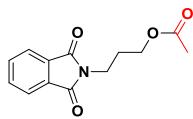


**4-Acetoxybutyl 2,6-difluorobenzoate (3as)**

petroleum ether / ethyl acetate = 10:1, yellow oil, 82% yield (44.5 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.46 – 7.38 (m, 1H), 6.98 – 6.94 (m, 2H), 4.40 (t, *J* = 6.1 Hz, 2H), 4.13 (t, *J* = 6.1 Hz, 2H), 2.06 (s, 3H), 1.85 – 1.73 (m, 7H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 171.1, 161.7 (d, *J* = 39.2 Hz), 159.3 (d, *J* = 6.2 Hz,), 132.7 (t, *J* = 10.5 Hz), 112.0 (dd, *J* = 24.3, 1.3 Hz), 65.4, 63.8, 25.2, 25.1, 20.9. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -110.5 (s, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>14</sub>F<sub>2</sub>O<sub>4</sub>+H<sup>+</sup>: 273.0933, Found: 273.0930. **IR** (neat, cm<sup>-1</sup>): ν 2857, 1730, 1624, 1594, 1469, 1288, 1110, 768, 634.



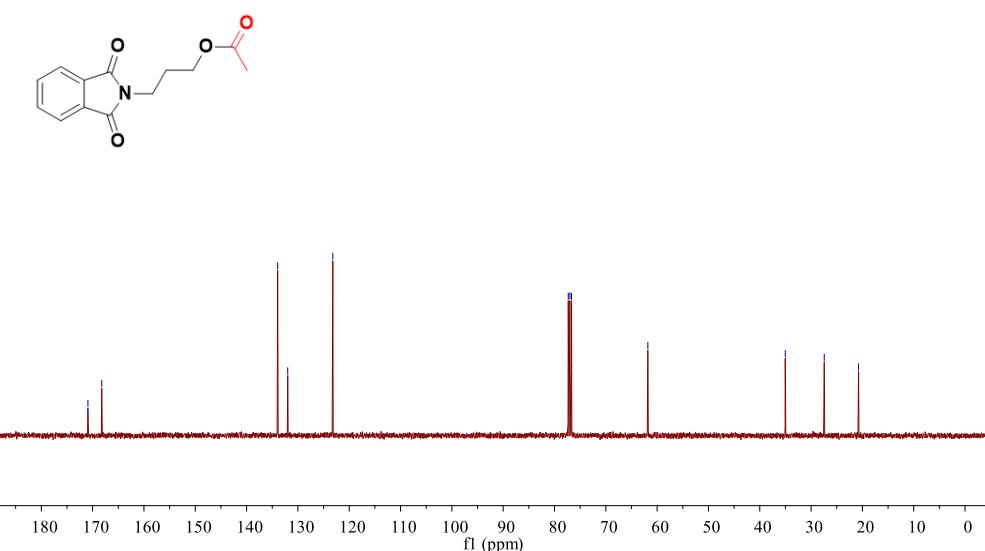
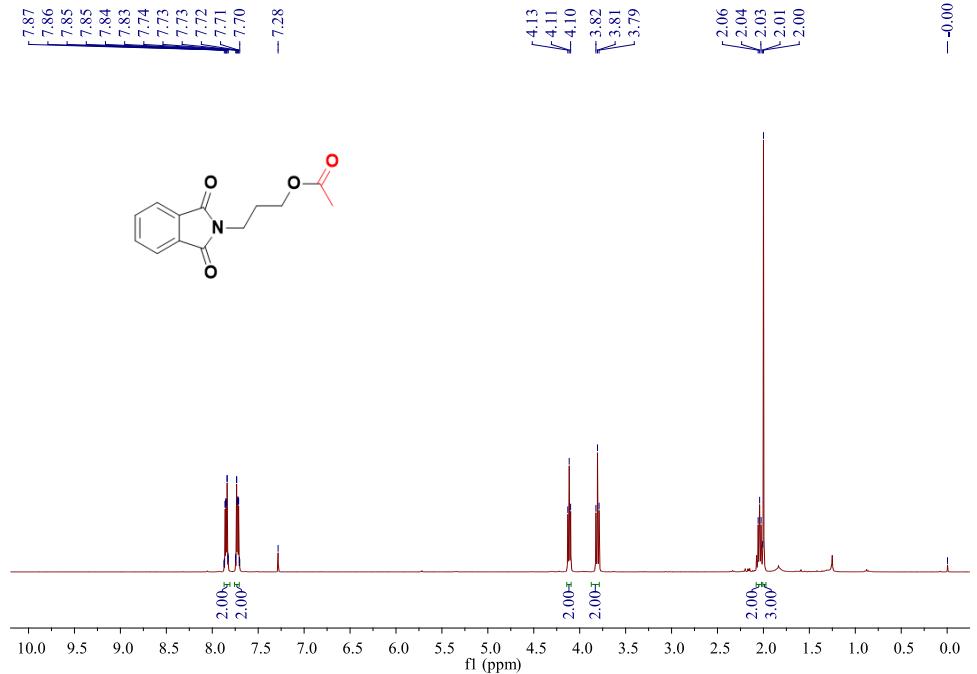


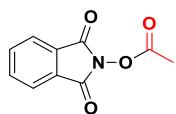


**3-(1,3-Dioxoisindolin-2-yl)propyl acetate (3at)**

petroleum ether / ethyl acetate = 10:1, white solid, 88% yield (46.0 mg). mp: 63 – 66°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.87 – 7.83 (m, 2H), 7.75 – 7.70 (m, 2H), 4.11 (t, *J* = 6.1 Hz, 2H), 3.81 (t, *J* = 6.1 Hz, 2H), 2.07 – 2.01 (m, 2H), 2.00 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.9, 168.2, 133.9, 132.0, 123.2, 61.8, 35.0, 27.4, 20.8. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>13</sub>NO<sub>4</sub>+H<sup>+</sup>: 248.0917, Found: 248.0913.

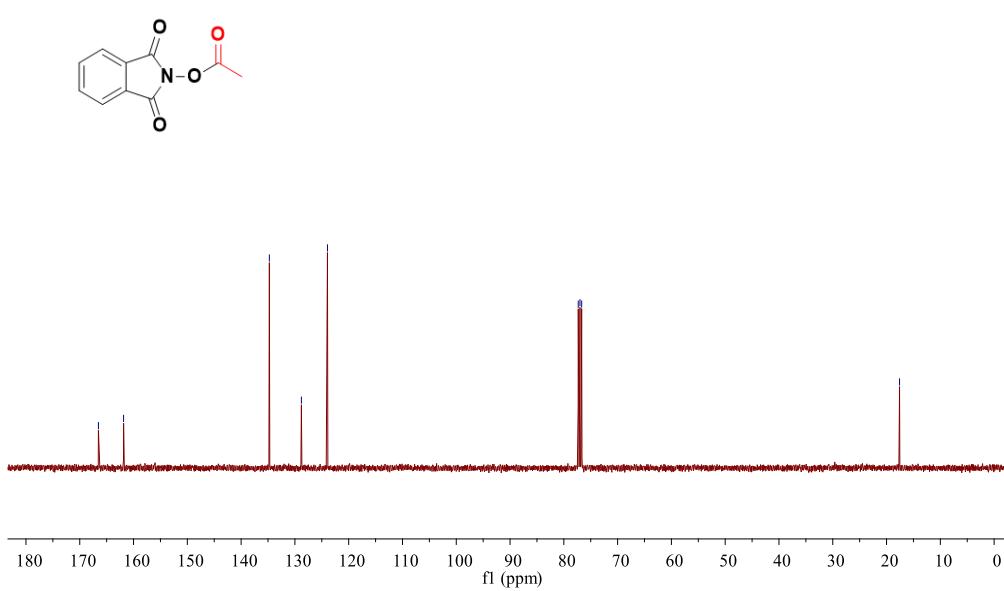
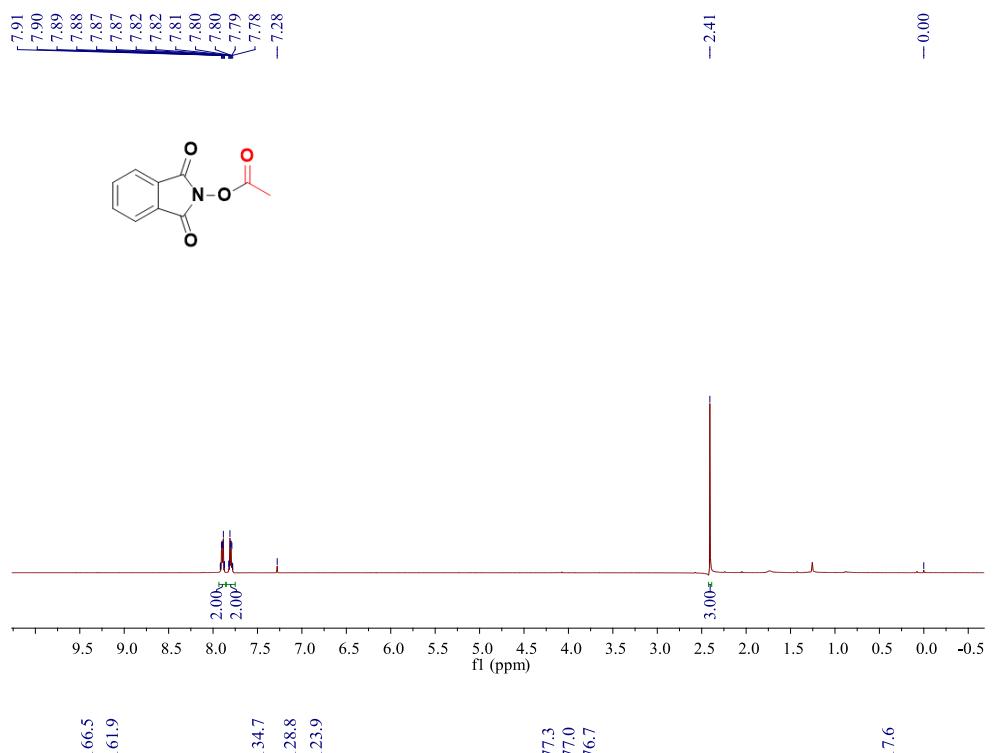
**IR** (neat, cm<sup>-1</sup>): ν 2900, 1771, 1614, 1527, 1467, 1365, 1188, 1001, 717, 605.

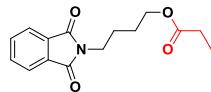




**1,3-Dioxoisindolin-2-yl acetate (3au)**

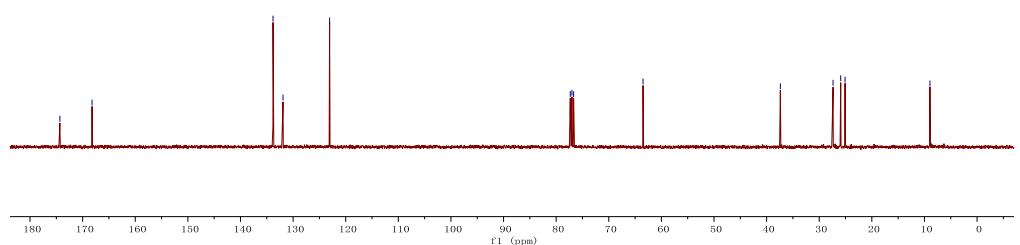
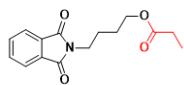
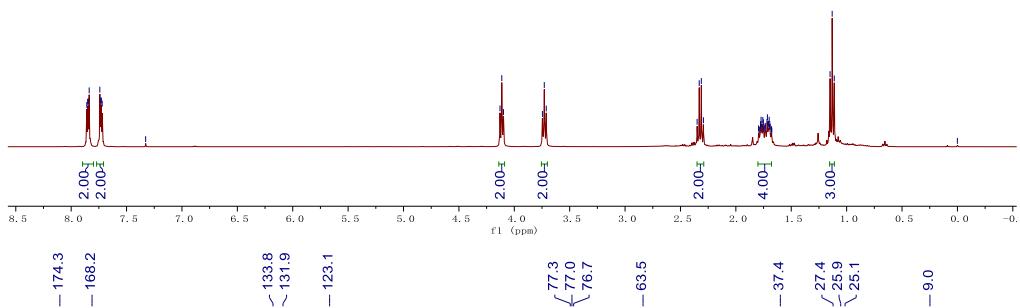
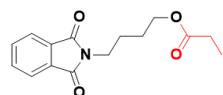
petroleum ether / ethyl acetate = 5:1, white solid, 61% yield (24.9 mg). mp: 183 – 185°C.  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 – 7.87 (m, 2H), 7.82 – 7.78 (m, 2H), 2.41 (s, 1H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.5, 161.9, 134.7, 128.8, 123.9, 17.6. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{10}\text{H}_7\text{NO}_4+\text{H}^+$ : 206.0448, Found: 206.0450. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  2850, 1808, 1785, 1609, 1466, 1236, 967, 720, 694.

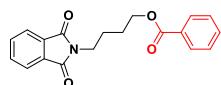




#### 4-(1,3-Dioxoisooindolin-2-yl)butyl propionate (3av)

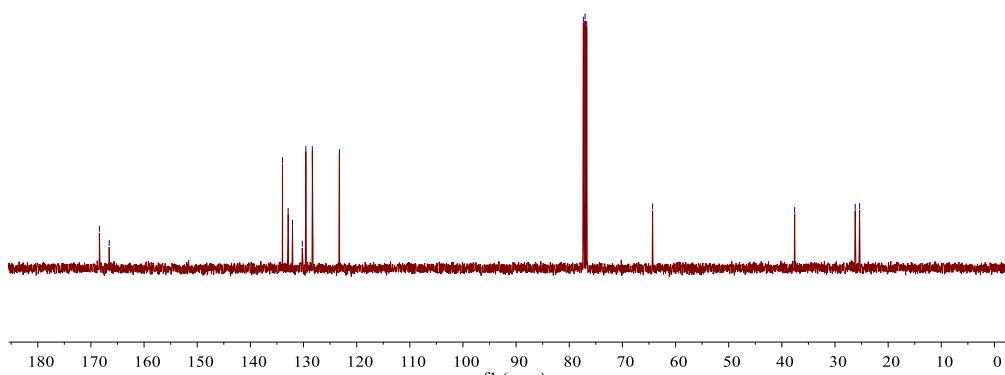
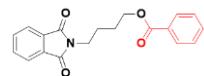
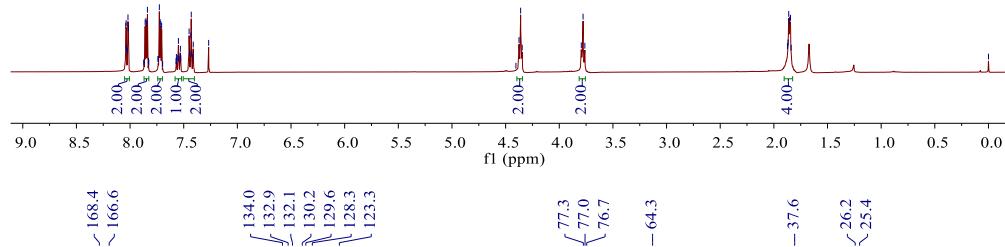
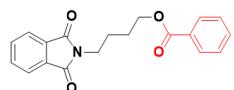
petroleum ether / ethyl acetate = 5:1, yellow liquid, 80% yield (44.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.8 – 7.84 (m, 2H), 7.74 – 7.72 (m, 2H), 4.11 (t, *J* = 6.9 Hz, 2H), 3.73 (t, *J* = 6.9 Hz, 2H), 2.32 (q, *J* = 7.6 Hz, 1H), 1.80 – 1.66 (m, 2H), 1.13 (t, *J* = 7.6 Hz, 2H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 174.3, 168.2, 133.8, 131.9, 123.1, 63.5, 37.4, 27.4, 25.9, 25.1, 9.0. **HRMS** (ESI-TOF): Anal Calcd. For C<sub>15</sub>H<sub>17</sub>NO<sub>4</sub>+Na<sup>+</sup>: 298.1050, Found: 298.1055; **IR** (neat, cm<sup>-1</sup>): ν 2859, 1720, 1610, 1555, 1438, 1190, 1008, 719.

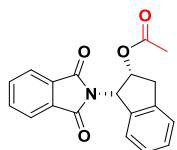




**4-(1,3-dioxoisindolin-2-yl)butyl benzoate (3aw)**

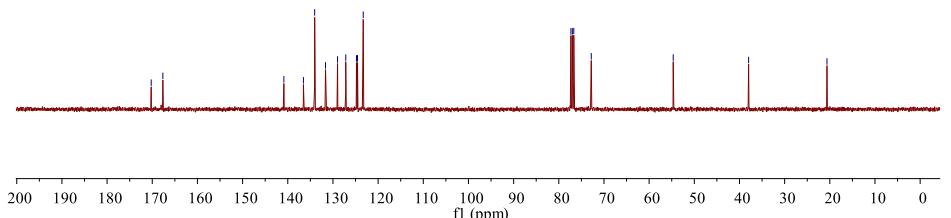
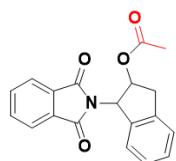
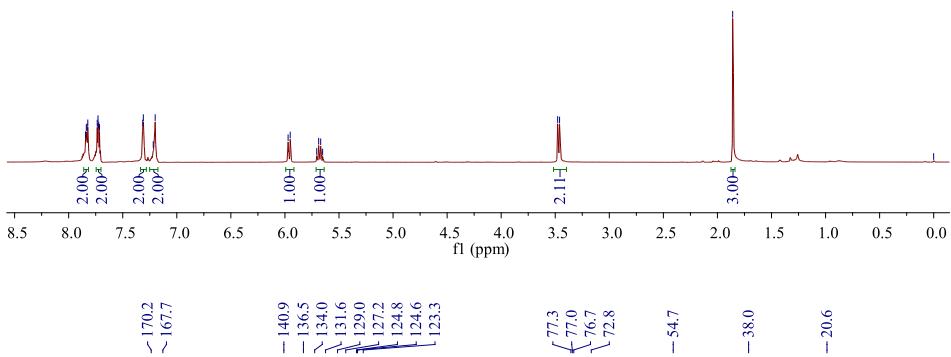
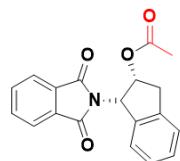
petroleum ether / ethyl acetate = 5:1, yellow liquid, 30% yield (19.4 mg).  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05 – 8.00 (m, 2H), 7.87 – 7.83 (m, 2H), 7.74 – 7.70 (m, 2H), 7.57 – 7.53 (m, 1H), 7.45 – 7.41 (m, 2H), 4.37 (t,  $J=6.6$  Hz, 2H), 3.78 (t,  $J=6.6$  Hz, 2H), 1.87 – 1.84 (m, 4H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 166.6, 134.0, 132.9, 132.1, 130.2, 129.6, 128.3, 123.3, 64.3, 37.6, 26.2, 25.4. **HRMS (ESI-TOF):** Anal Calcd. For.  $\text{C}_{19}\text{H}_{17}\text{NO}_4$ : 346.1050, Found: 346.1054; **IR (neat, cm}^{-1}):**  $\nu$  2790, 1760, 1610, 1499, 1223, 752.

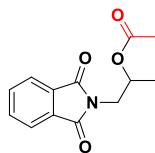




**(1S,2R)-1-(1,3-Dioxoisooindolin-2-yl)-2,3-dihydro-1H-inden-2-yl acetate (3ax)**

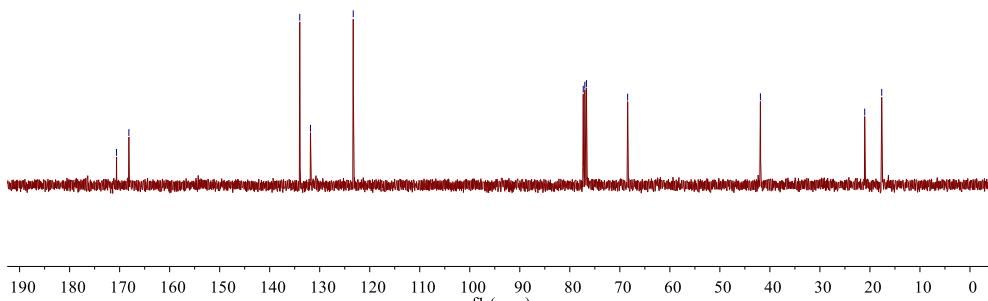
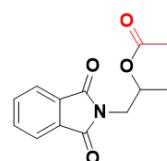
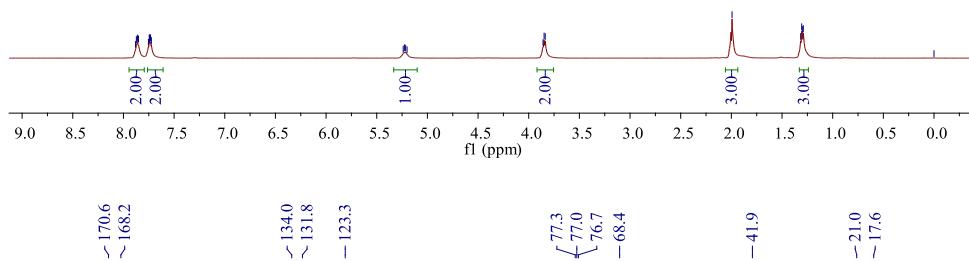
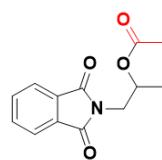
petroleum ether / ethyl acetate = 5:1, white solid, 62% yield (39.6 mg). mp: 135 – 137°C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87 – 7.82 (m, 2H), 7.76 – 7.71 (m, 2H), 7.32 – 7.31 (m, 2H), 7.22 – 7.20 (m, 2H), 5.96 (d, *J* = 7.8 Hz, 1H), 5.68 (q, *J* = 7.3 Hz, 1H), 3.47 (d, *J* = 7.1 Hz, 2H), 1.86 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.2, 167.7, 140.9, 136.5, 134.0, 131.6, 129.0, 127.2, 124.8, 124.6, 123.3, 72.8, 54.7, 38.0, 20.6. HRMS (ESI-TOF): Anal Calcd. For. C<sub>19</sub>H<sub>15</sub>NO<sub>4</sub>+H<sup>+</sup>: 322.1074, Found: 322.1070. IR (neat, cm<sup>-1</sup>): ν 2850, 1705, 1600, 1450, 1205, 905, 710.

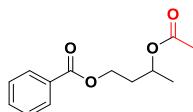




**1-(1,3-Dioxoisindolin-2-yl)propan-2-yl acetate (3ay)**

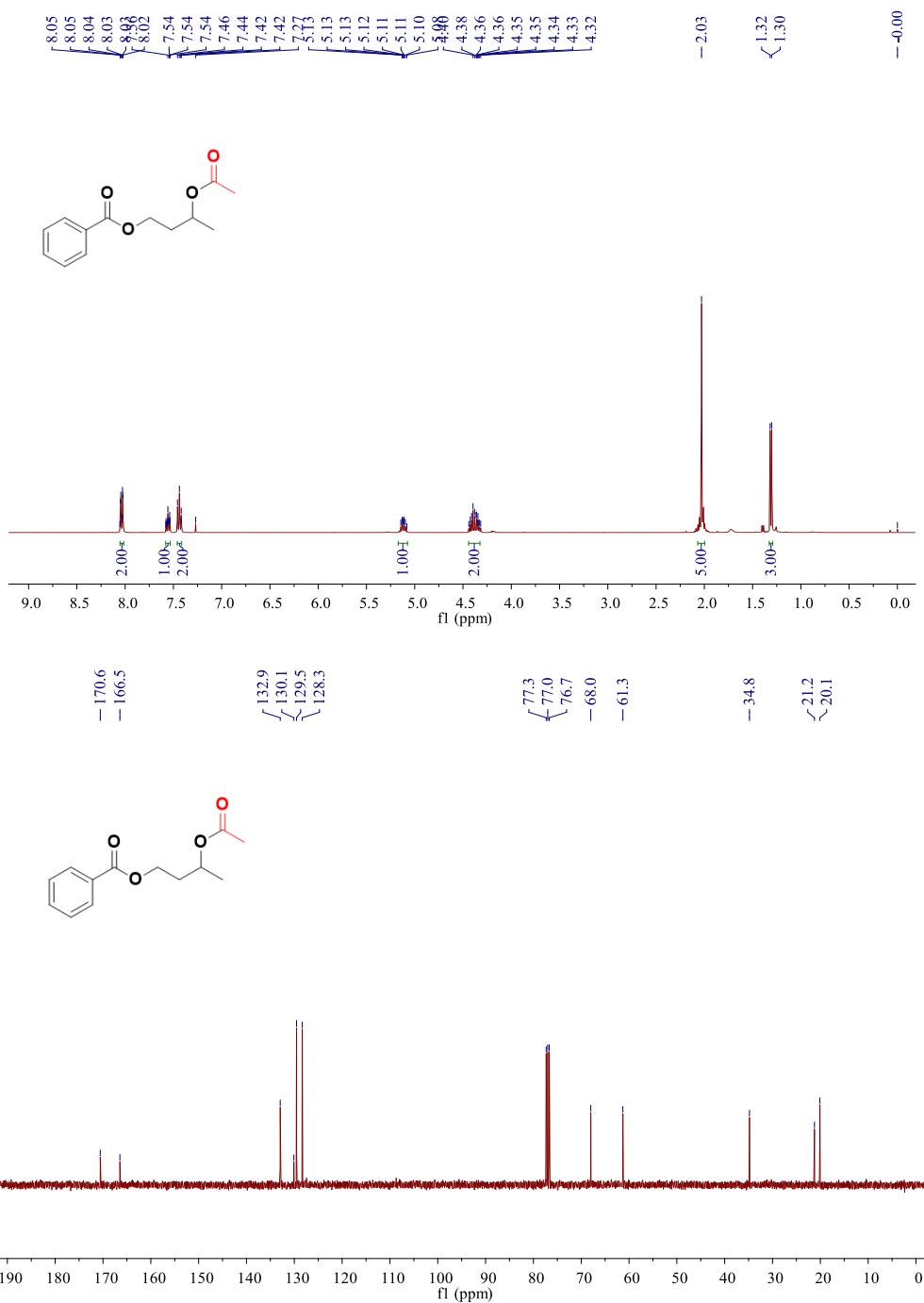
petroleum ether / ethyl acetate = 5:1, white solid, 91% yield (44.7 mg). mp: 80 – 83°C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.88 – 7.85 (m, 2H), 7.75 – 7.72 (m, 2H), 5.22 (dd, *J* = 10.1, 5.2 Hz, 1H), 3.86 – 3.84 (m, 2H), 1.99 (s, 3H), 1.32 – 1.29 (m, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.6, 168.2, 134.0, 131.8, 123.3, 68.4, 41.9, 21.1, 17.6. HRMS (ESI-TOF): Anal Calcd. For. C<sub>17</sub>H<sub>20</sub>Cl<sub>2</sub>O<sub>5</sub> +H<sup>+</sup>: 248.0917, Found: 248.0913. IR (neat, cm<sup>-1</sup>): ν 1775, 1713, 1615, 1468, 1397, 1241, 1036, 906, 723, 648.

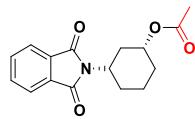




**3-Acetoxybutyl benzoate (3az)**

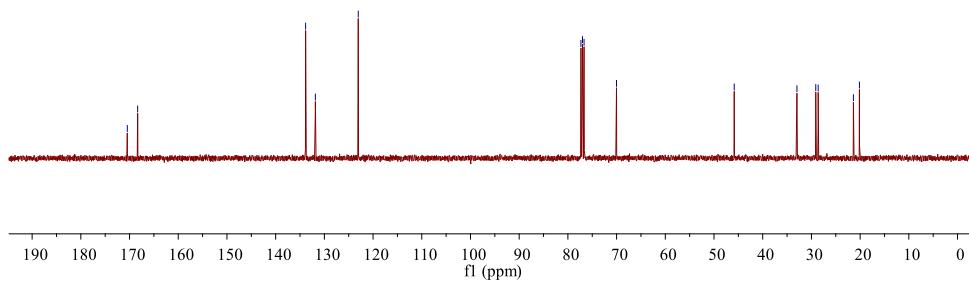
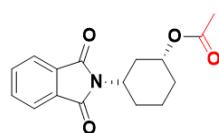
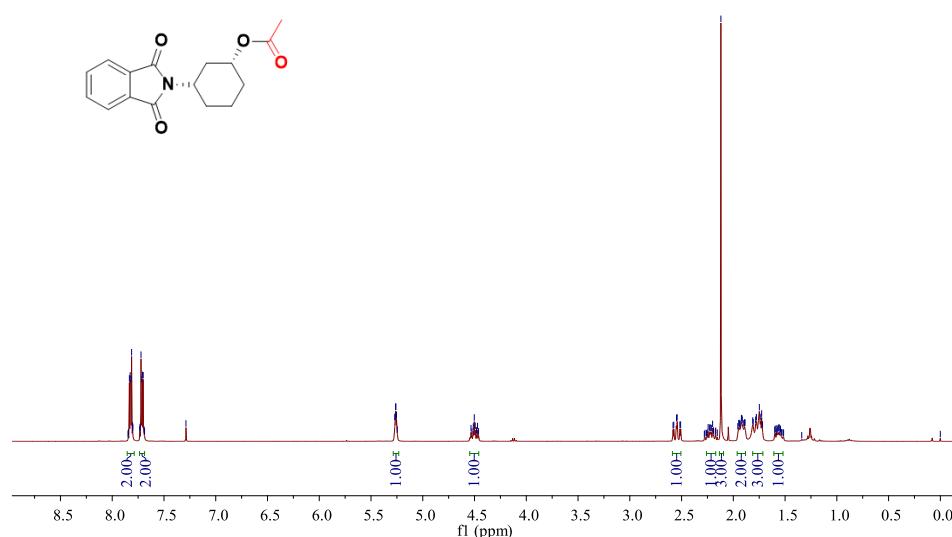
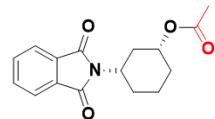
petroleum ether / ethyl acetate = 20:1, colorless oil, 82% yield (38.9 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.05 – 8.02 (m, 2H), 7.58 – 7.54 (m, 1H), 7.44 – 7.42 (m, 1H), 5.14 – 5.08 (m, 1H), 4.44 – 4.32 (m, 2H), 2.09 – 1.97 (m, 5H), 1.31 (d, *J* = 6.3 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.6, 166.5, 132.9, 130.1, 129.5, 128.3, 68.0, 61.3, 34.8, 21.2, 20.1. **HRMS (ESI-TOF)**: Anal Calcd. For. C<sub>13</sub>H<sub>16</sub>O<sub>4</sub>+H<sup>+</sup>: 237.1121, Found: 237.1118. **IR** (neat, cm<sup>-1</sup>): ν 2869, 1709, 1613, 1586, 1396, 1240, 818, 676.

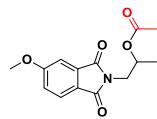




**(1R,3S)-3-(1,3-dioxoisindolin-2-yl) cyclohexyl acetate (3ba)**

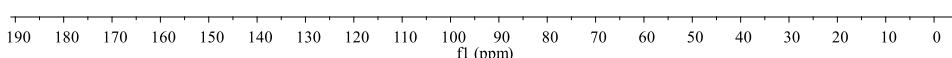
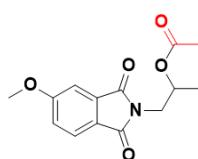
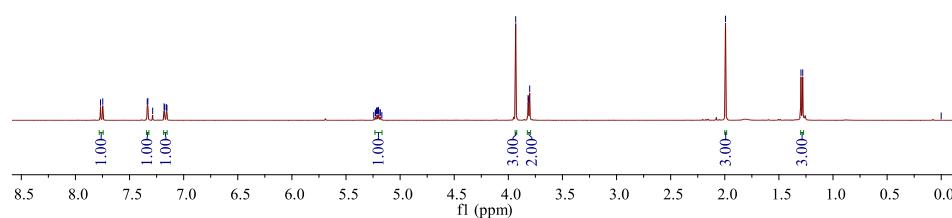
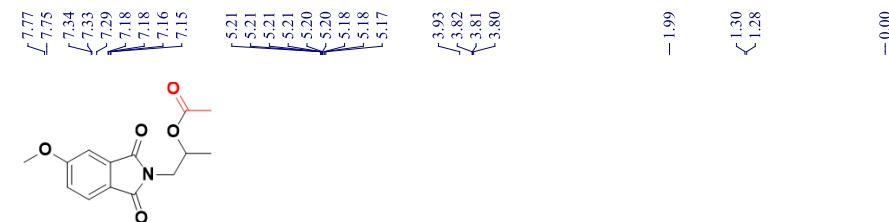
petroleum ether / ethyl acetate = 5:1, white solid, 77% yield (44.1 mg). mp: 100 – 105°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.85 – 7.80 (m, 2H), 7.73 – 7.69 (m, 2H), 5.27 – 5.25 (m, 1H), 4.53 – 4.46 (m, 1H), 2.58 – 2.51 (m, 1H), 2.24 – 2.15 (m, 1H), 2.12 (s, 3H), 1.95 – 1.89 (m, 2H), 1.81 – 1.72 (m, 3H), 1.60 – 1.51 (m, 1H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 168.3, 133.8, 131.8, 123.0, 70.0, 45.8, 33.0, 29.1, 28.6, 21.4, 20.1. **HRMS (ESI-TOF)**: Anal Calcd. For. C<sub>16</sub>H<sub>17</sub>NO<sub>4</sub>+Na<sup>+</sup>: 310.1050, Found: 310.1048. **IR** (neat, cm<sup>-1</sup>): ν 2868, 1760, 1702, 1613, 1455, 1397, 1211, 1075, 896, 714.

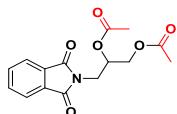




**1-(5-Methoxy-1,3-dioxoisindolin-2-yl)propan-2-yl acetate (3bb)**

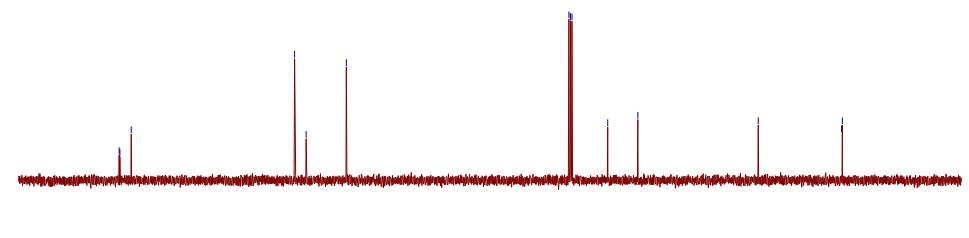
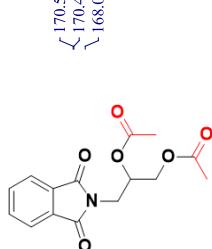
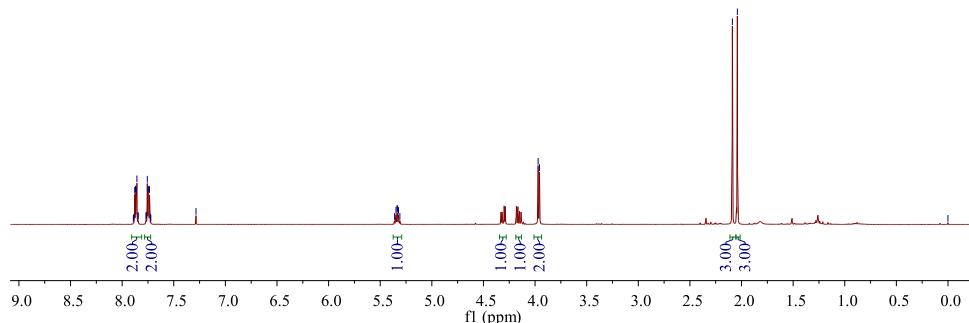
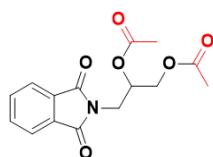
petroleum ether / ethyl acetate = 5:1, white solid, 80% yield (44.3 mg). mp: 80 – 82°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.76 (d, *J* = 8.3 Hz, 1H), 7.33 (d, *J* = 2.2 Hz, 1H), 7.17 (dd, *J* = 8.3, 2.2 Hz, 1H), 5.24 – 5.17 (m, 1H), 3.93 (s, 3H), 3.82 – 3.80 (m, 2H), 1.99 (s, 3H), 1.29 (d, *J* = 6.5 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.6, 168.0, 167.9, 164.7, 134.5, 125.0, 123.7, 119.8, 108.0, 68.5, 56.0, 41.9, 21.1, 17.6. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>15</sub>NO<sub>5</sub>+H<sup>+</sup>: 278.1023, Found: 278.1020. **IR** (neat, cm<sup>-1</sup>): ν 2844, 1770, 1616, 1489, 1286, 1093, 955, 769, 605.

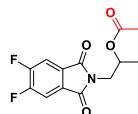




**3-(1,3-Dioxoisindolin-2-yl)propane-1,2-diyI diacetate (3bc)**

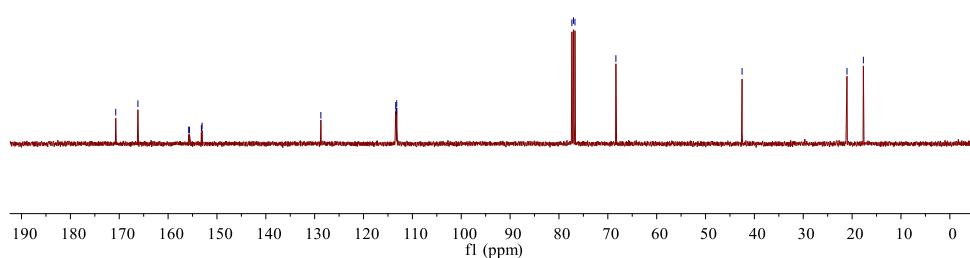
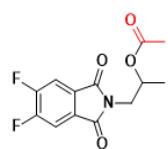
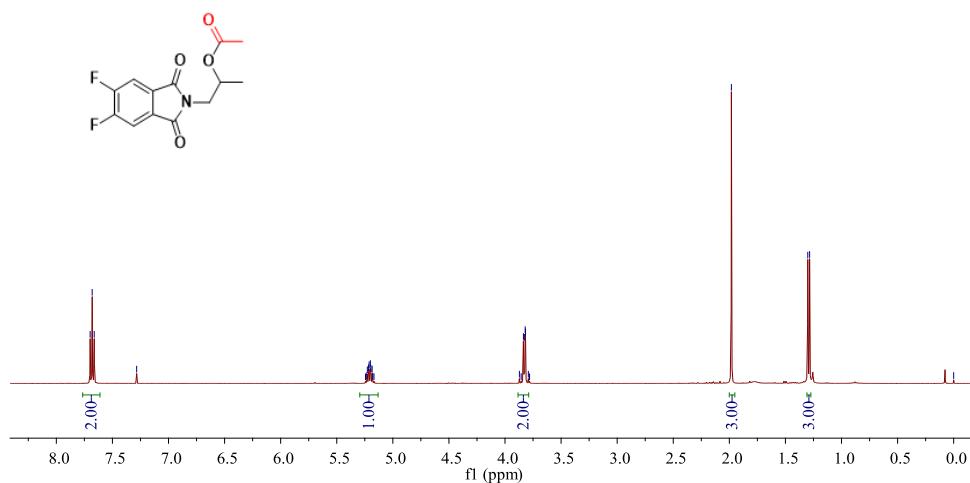
petroleum ether / ethyl acetate = 5:1, colorless oil, 60% yield (36.8 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.89 – 7.84 (m, 2H), 7.77 – 7.72 (m, 2H), 5.36 – 5.31 (m, 1H), 4.31 (dd, *J* = 12.1, 4.1 Hz, 1H), 4.16 (dd, *J* = 12.1, 5.8 Hz, 1H), 3.96 (d, *J* = 5.2 Hz, 2H), 2.09 (s, 3H), 2.04 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 170.4, 168.0, 134.2, 131.8, 123.4, 69.3, 63.0, 38.1, 20.8, 20.7. **HRMS** (ESI-TOF): Anal Calcd. For C<sub>15</sub>H<sub>15</sub>NO<sub>6</sub>+H<sup>+</sup>: 306.0972, Found: 306.0969. **IR** (neat, cm<sup>-1</sup>): ν 2852, 1775, 1713, 1615, 1468, 1390, 1220, 1034, 794, 627.

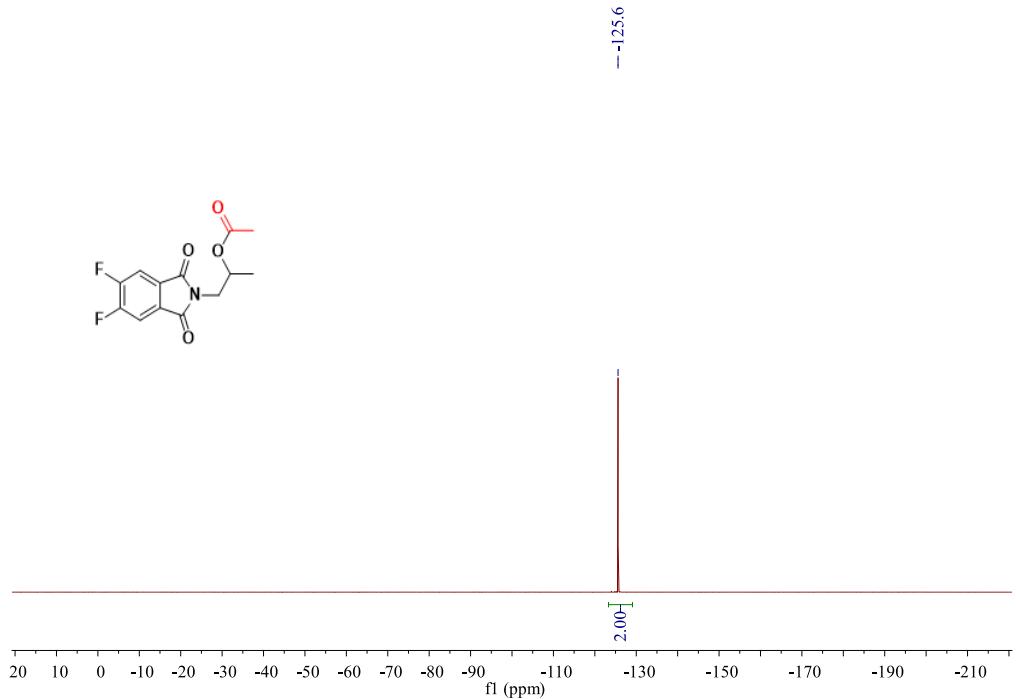


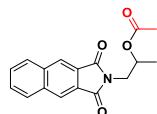


**1-(5,6-Difluoro-1,3-dioxoisindolin-2-yl)propan-2-yl acetate (3bd)**

petroleum ether / ethyl acetate = 5:1, colorless oil, 80% yield (45.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.68 (t, *J* = 7.3 Hz, 2H), 5.24 – 5.17 (m, 1H), 3.87 – 3.78 (m, 2H), 1.98 (s, 3H), 1.29 (d, *J* = 6.5 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.7, 166.2, 155.7 (d, *J* = 15.3 Hz), 153.1 (d, *J* = 15.3 Hz), 128.7 (t, *J* = 6.0 Hz), 113.3 (dd, *J* = 14.6, 7.5 Hz), 68.3, 42.5, 21.0, 17.7. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -125.6 (s, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>11</sub>F<sub>2</sub>NO<sub>4</sub>+H<sup>+</sup>: 284.0729, Found: 284.0725. **IR** (neat, cm<sup>-1</sup>): ν 2850, 1780, 1714, 1622, 1494, 1397, 1295, 1030, 907, 727, 605.

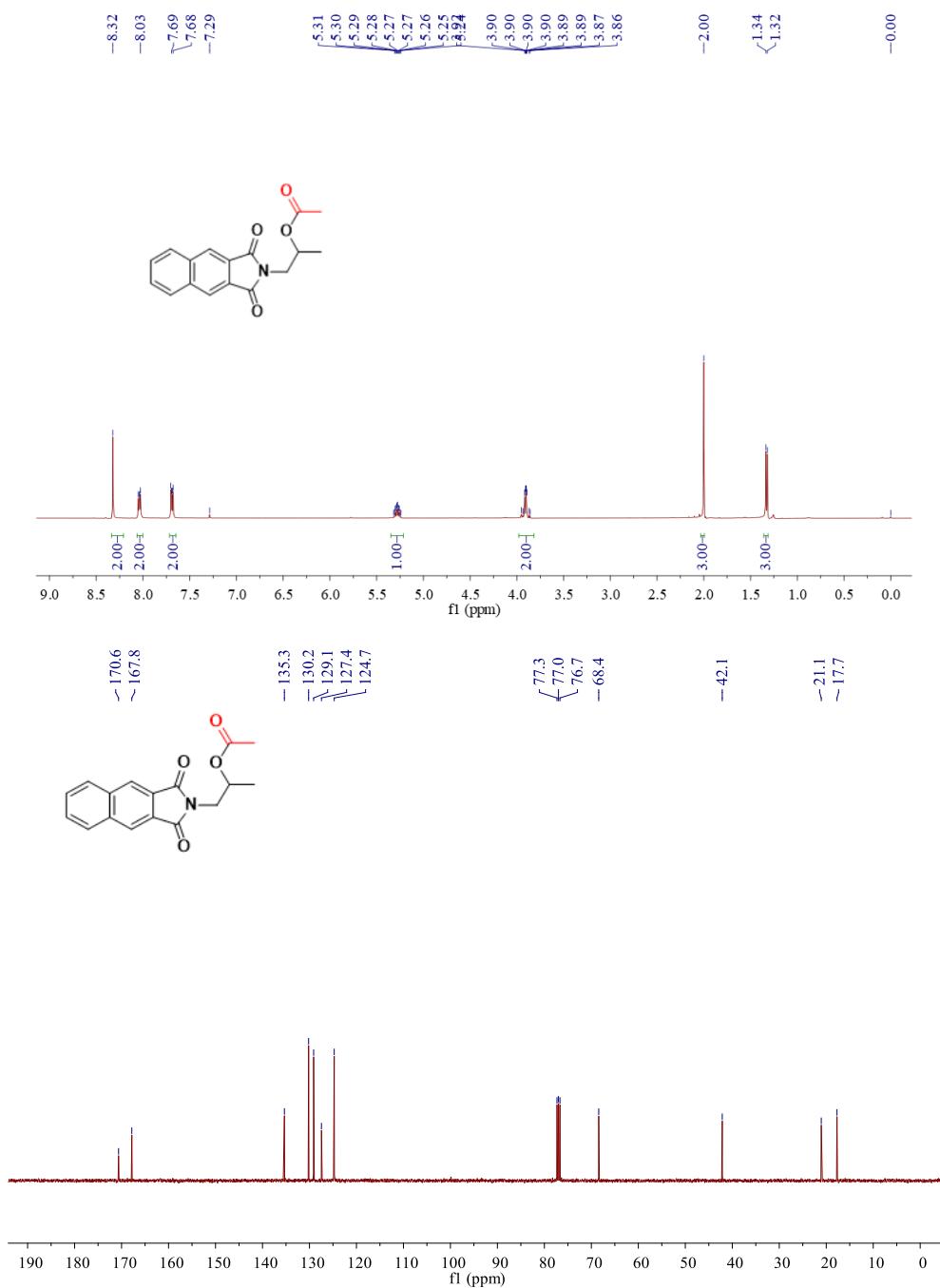


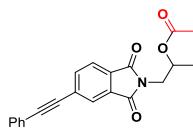




**1-(1,3-Dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl)propan-2-yl acetate (3be)**

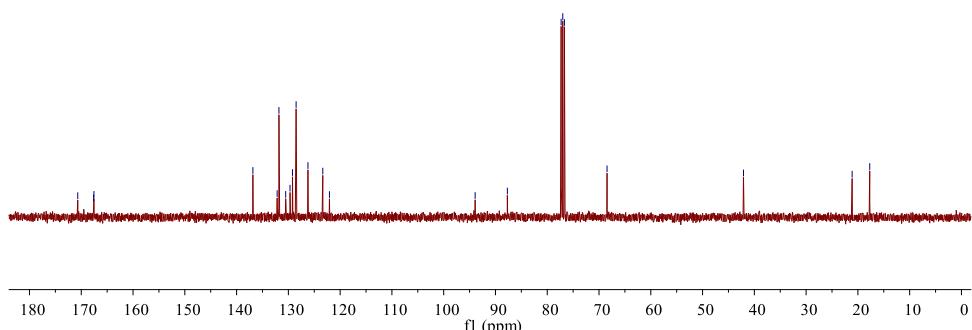
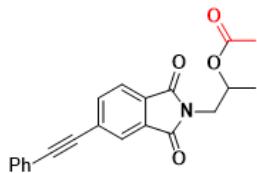
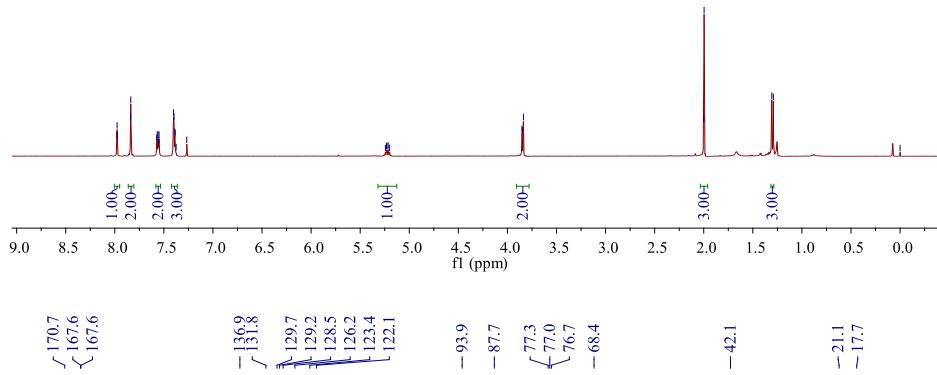
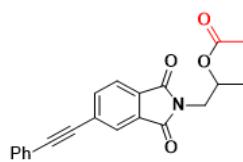
petroleum ether / ethyl acetate = 5:1, white solid, 85% yield (50.3 mg). mp: 116 – 118°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.32 (s, 1H), 8.04 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.69 (dd, *J* = 6.2, 3.3 Hz, 2H), 5.32 – 5.24 (m, 1H), 3.95 – 3.86 (m, 2H), 2.00 (s, 3H), 1.33 (d, *J* = 6.5 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.6, 167.8, 135.3, 130.2, 129.1, 127.4, 124.7, 68.4, 42.1, 21.1, 17.7. **HRMS** (ESI-TOF): Anal Calcd. For C<sub>17</sub>H<sub>15</sub>NO<sub>4</sub>+H<sup>+</sup>: 298.1074, Found: 298.1070. **IR** (neat, cm<sup>-1</sup>): ν 2852, 1766, 1602, 1515, 1376, 1242, 1182, 1032, 764.

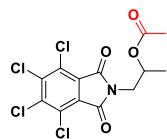




**1-(1,3-Dioxo-5-(phenylethynyl)isoindolin-2-yl)propan-2-yl acetate (3bf)**

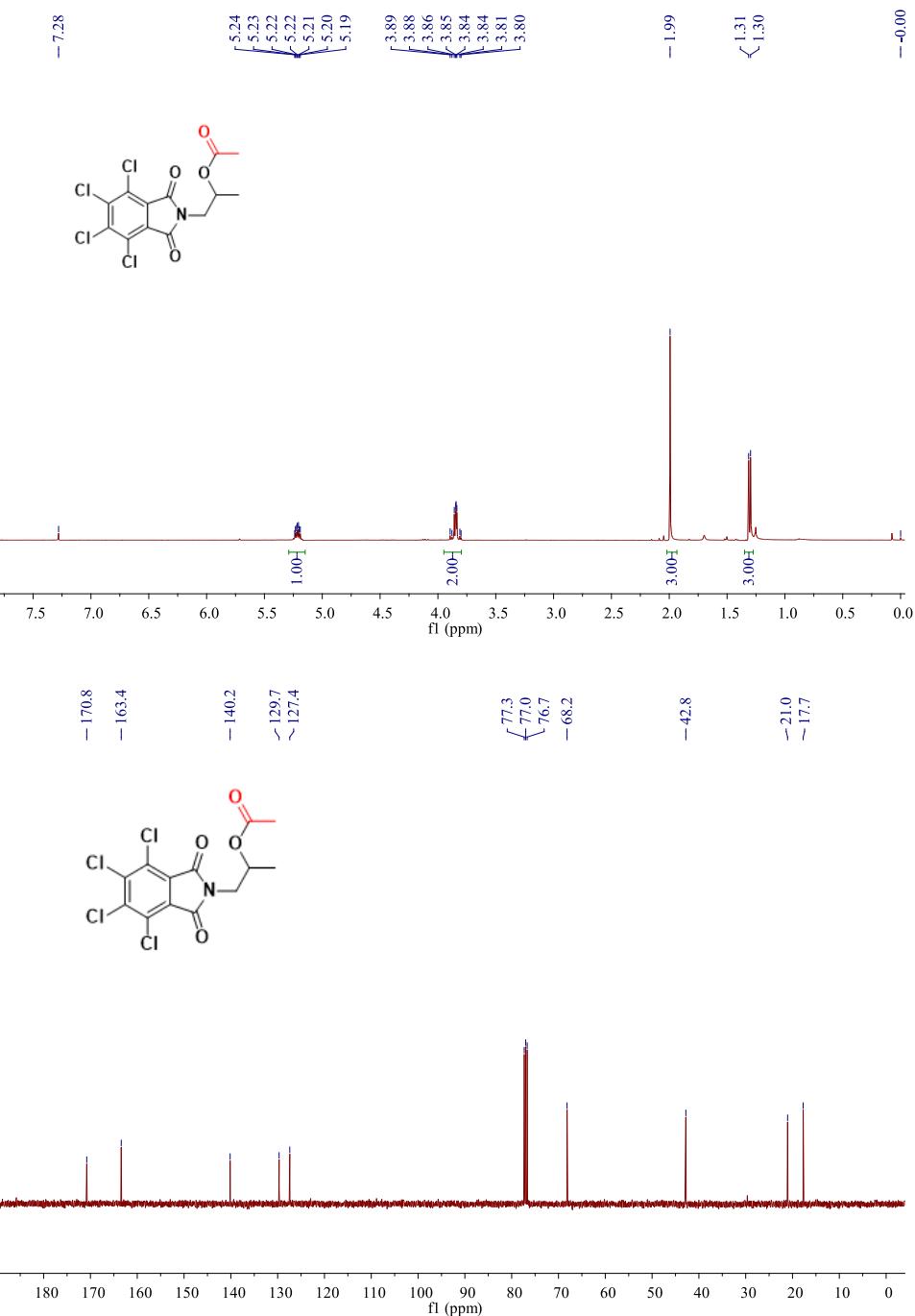
petroleum ether / ethyl acetate = 5:1, white solid, 35% yield (24.2 mg). mp: 78 – 80°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.98 (t, *J* = 1.0 Hz, 1H), 7.84 (d, *J* = 1.0 Hz, 2H), 7.57 – 7.55 (m, 2H), 7.40 – 7.38 (m, 2H), 5.24 – 5.20 (m, 1H), 3.85 – 3.84 (m, 2H), 2.00 (s, 3H), 1.30 (d, *J* = 6.5 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.7, 167.6, 167.6, 136.9, 132.2, 131.8, 130.5, 129.7, 129.2, 128.5, 126.2, 123.4, 122.1, 93.9, 87.7, 68.4, 42.1, 21.1, 17.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>21</sub>H<sub>17</sub>NO<sub>4</sub>+H<sup>+</sup>: 348.1230, Found: 348.1226. **IR** (neat, cm<sup>-1</sup>): ν 2850, 2200, 1770, 1713, 1615, 1515, 1494, 1244, 1033, 906, 725.

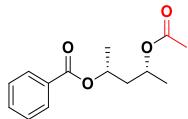




**1-(4,5,6,7-Tetrachloro-1,3-dioxoisindolin-2-yl)propan-2-yl acetate (3bg)**

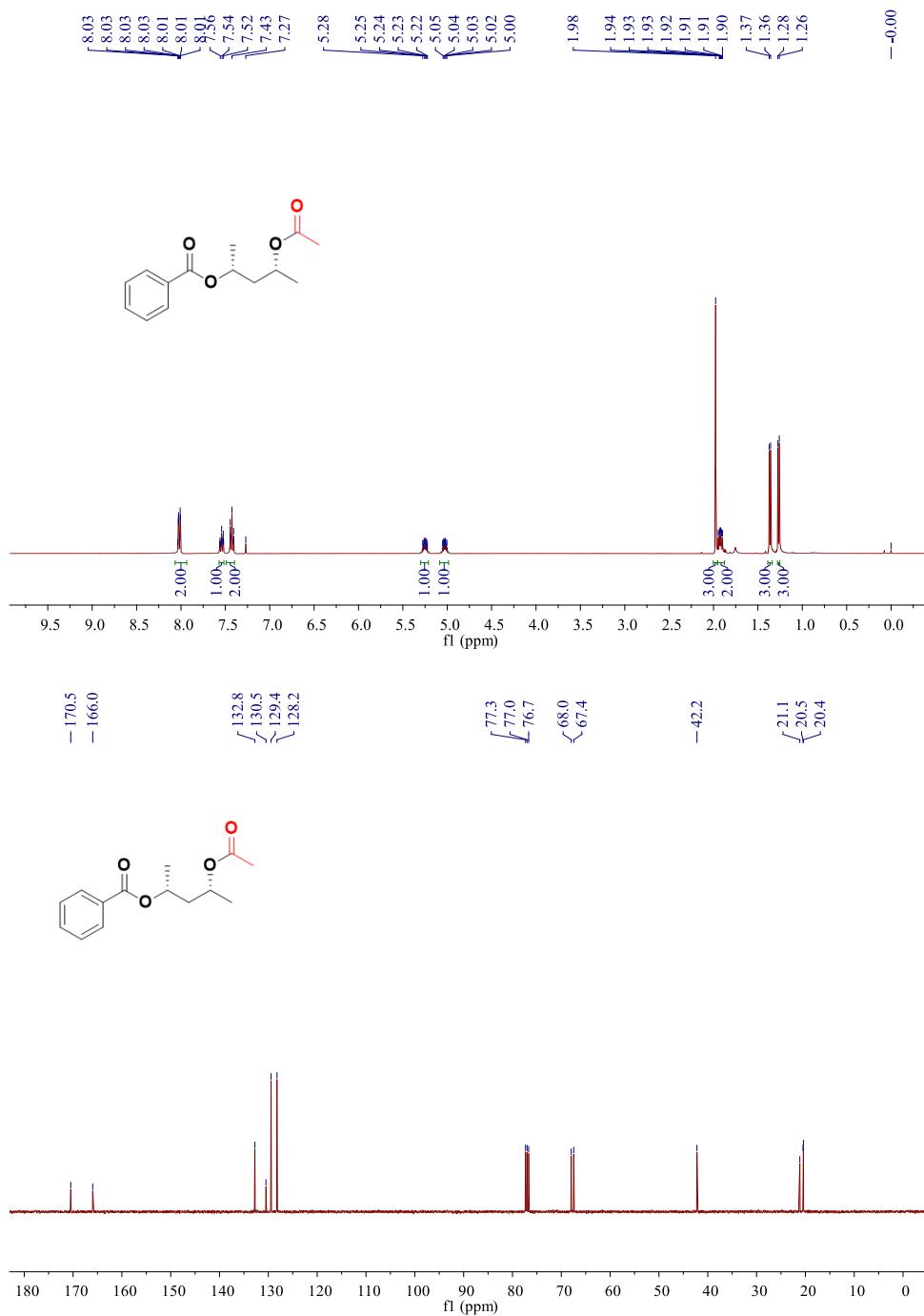
petroleum ether / ethyl acetate = 5:1, white solid, 83% yield (63.8 mg). mp: 151 – 153°C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.24 – 5.19 (m, 1H), 3.89 – 3.80 (m, 2H), 1.99 (s, 3H), 1.31 (d,  $J$  = 6.5 Hz, 3H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 163.4, 140.2, 129.7, 127.4, 68.2, 42.8, 21.0, 17.7. HRMS (ESI-TOF): Anal Calcd. For.  $\text{C}_{13}\text{H}_{9}\text{Cl}_4\text{NO}_4+\text{Na}^+$ : 405.9178, Found: 405.9181. IR (neat,  $\text{cm}^{-1}$ ):  $\nu$  2851, 1774, 1426, 1357, 1301, 1194, 1045, 740, 628.

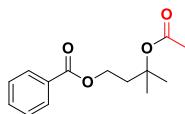




**(2R,4R)-4-Acetoxypentan-2-yl benzoate (3bh)**

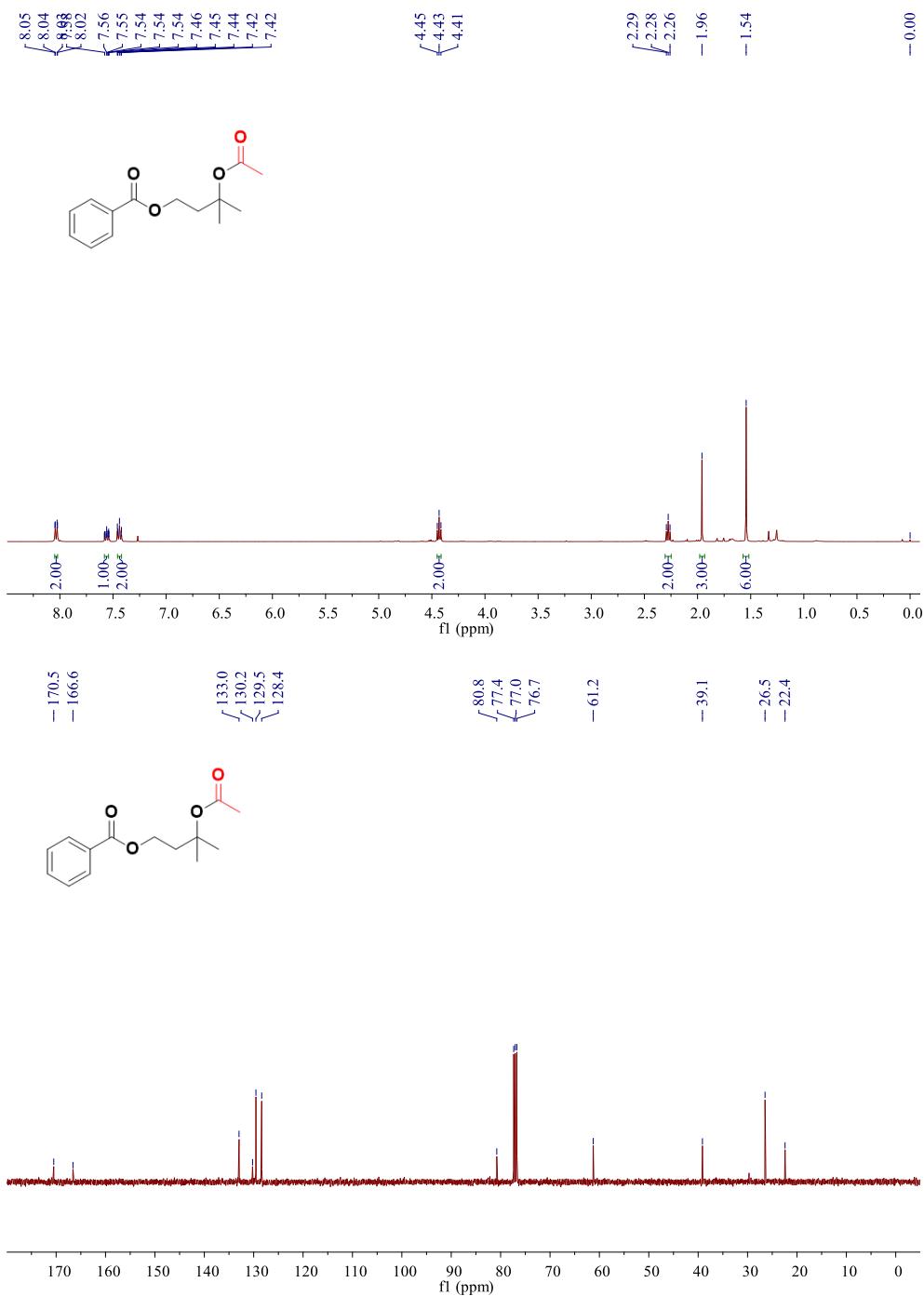
petroleum ether / ethyl acetate = 10:1, colorless oil, 80% yield (40.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.03 – 8.01 (m, 2H), 7.56 – 7.52 (m, 1H), 7.44 – 7.41 (m, 2H), 5.28 – 5.22 (m, 1H), 5.05 – 5.00 (m, 1H), 1.98 (s, 3H), 1.95 – 1.90 (m, 2H), 1.36 (d, *J* = 6.3 Hz, 2H), 1.27 (d, *J* = 6.3 Hz, 2H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 166.0, 132.8, 130.5, 129.4, 128.2, 68.0, 67.4, 42.2, 21.1, 20.5, 20.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>+H<sup>+</sup>: 251.1278, Found: 251.1274. **IR** (neat, cm<sup>-1</sup>):  $\nu$  2853, 1773, 1608, 1507, 1427, 1367, 1234, 986, 728.

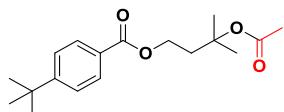




### **3-Acetoxy-3-methylbutyl benzoate (3bi)**

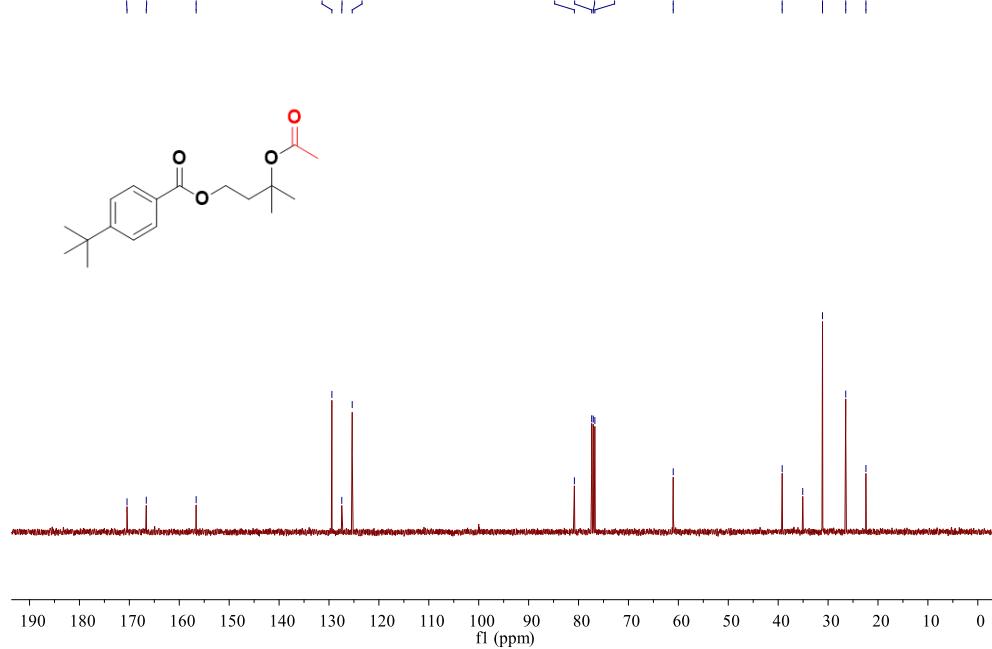
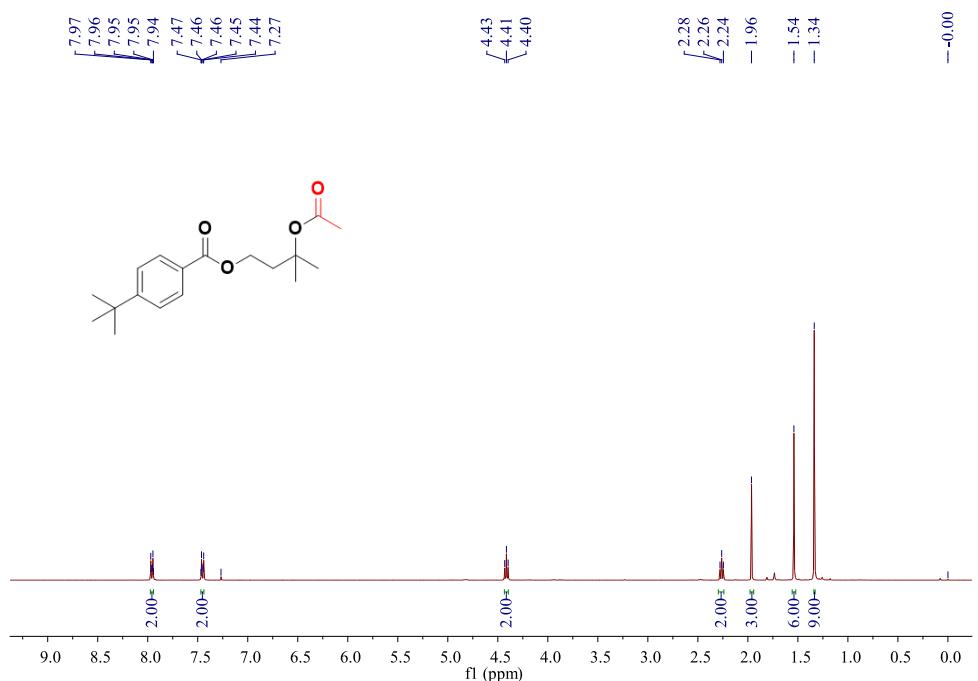
petroleum ether / ethyl acetate = 10:1, colorless oil, 70% yield (35.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.03 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.58 – 7.54 (m, 1H), 7.46 – 7.42 (m, 2H), 4.43 (t, *J* = 6.8 Hz, 2H), 2.28 (t, *J* = 6.8 Hz, 2H), 1.96 (s, 3H), 1.54 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 166.6, 133.0, 130.2, 129.5, 128.4, 80.8, 61.2, 39.1, 26.5, 22.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>+Na<sup>+</sup>: 273.1097, Found: 273.1093. **IR** (neat, cm<sup>-1</sup>): ν 2929, 1717, 1652, 1585, 1471, 1222, 1176, 903, 723, 649.

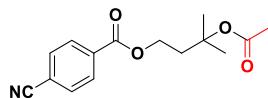




**3-Acetoxy-3-methylbutyl 4-(tert-butyl)benzoate (3bj)**

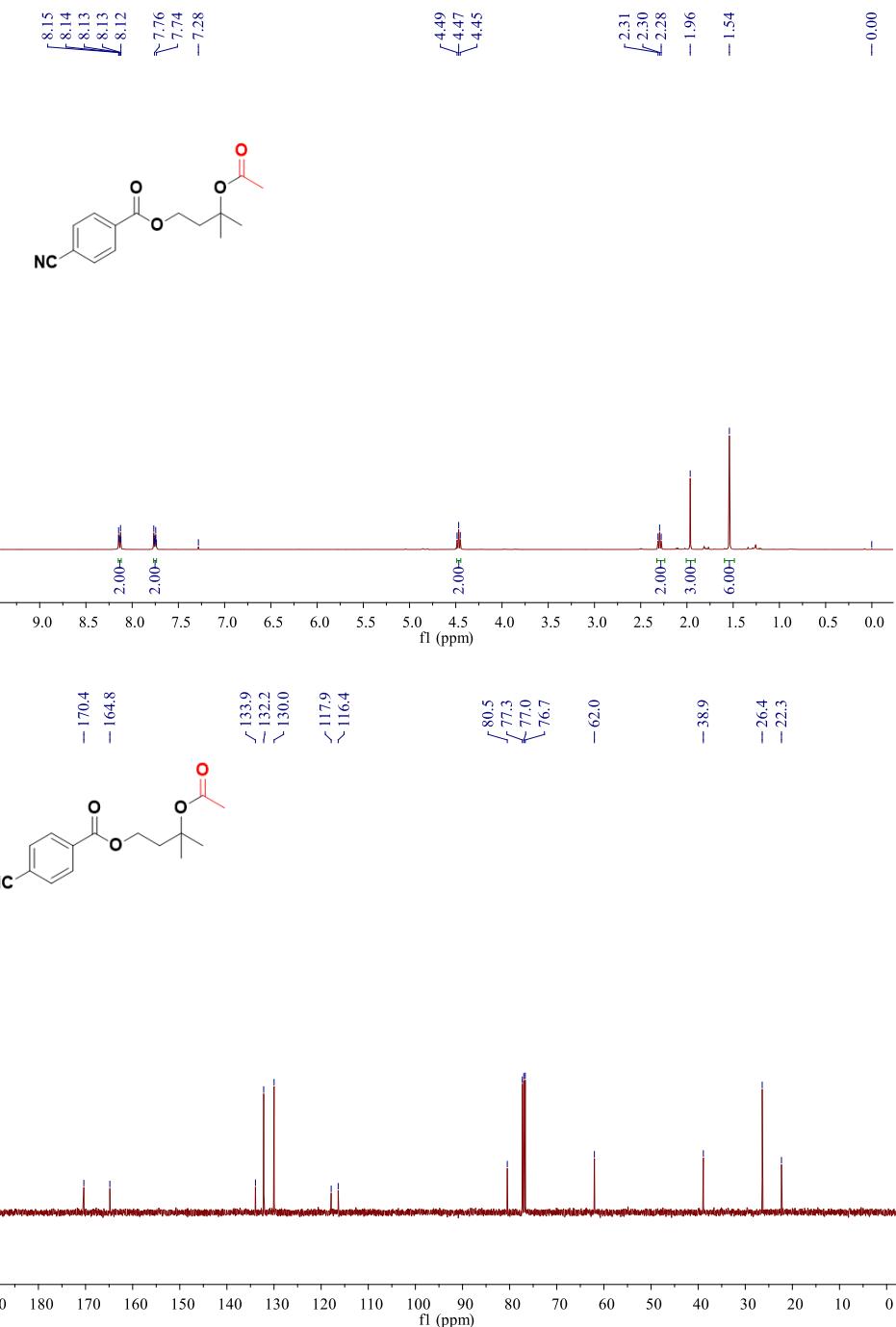
petroleum ether / ethyl acetate = 20:1, colorless oil, 75% yield (45.8 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.97 – 7.94 (m, 2H), 7.47 – 7.44 (m, 2H), 4.41 (t, *J* = 6.8 Hz, 2H), 2.26 (t, *J* = 6.8 Hz, 2H), 1.96 (s, 3H), 1.54 (s, 6H), 1.34 (s, 9H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 166.6, 156.6, 129.4, 127.4, 125.3, 80.8, 61.0, 39.2, 35.1, 31.1, 26.5, 22.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>18</sub>H<sub>26</sub>O<sub>4</sub>+Na<sup>+</sup>: 329.1723, Found: 329.1713. **IR** (neat, cm<sup>-1</sup>): ν 2870, 1716, 1610, 1570, 1472, 1388, 1277, 905, 725.

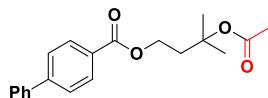




**3-Acetoxy-3-methylbutyl 4-cyanobenzoate (3bk)**

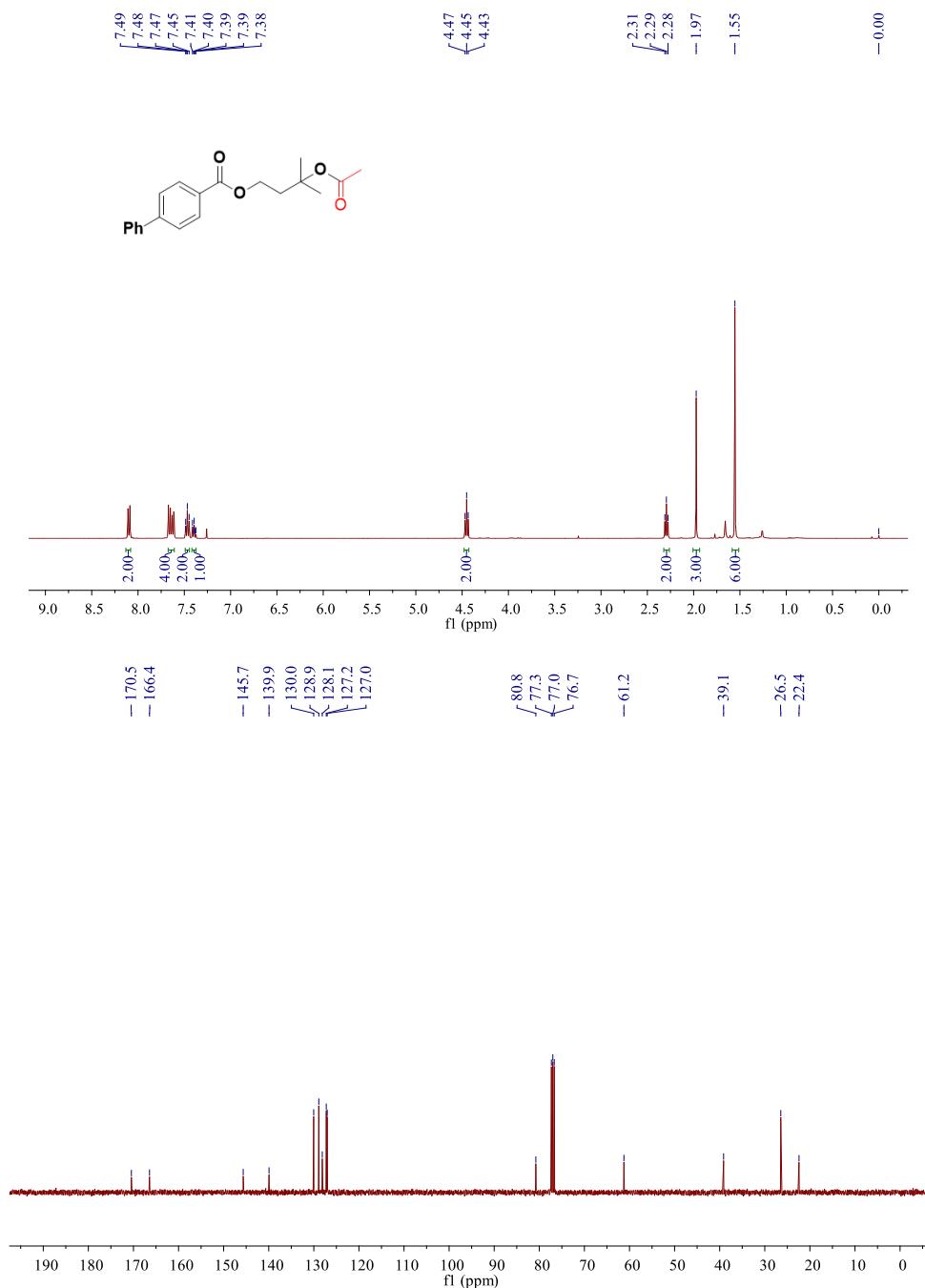
petroleum ether / ethyl acetate = 20:1, yellow oil, 66% yield (36.4 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.15 – 8.12 (m, 2H), 7.77 – 7.74 (m, 2H), 4.47 (t, *J* = 6.9 Hz, 2H), 2.30 (t, *J* = 6.9 Hz, 2H), 1.96 (s, 3H), 1.54 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.4, 164.8, 133.9, 132.2, 130.0, 117.9, 116.4, 80.5, 62.0, 38.9, 26.4, 22.3. HRMS (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>17</sub>NO<sub>4</sub>+Na<sup>+</sup>: 298.1050, Found: 298.1049. IR (neat, cm<sup>-1</sup>): ν 2931, 2205, 1770, 1611, 1514, 1465, 1368, 1249, 1045, 767, 691.

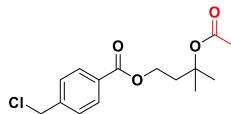




**3-Acetoxy-3-methylbutyl [1,1'-biphenyl]-4-carboxylate (3bl)**

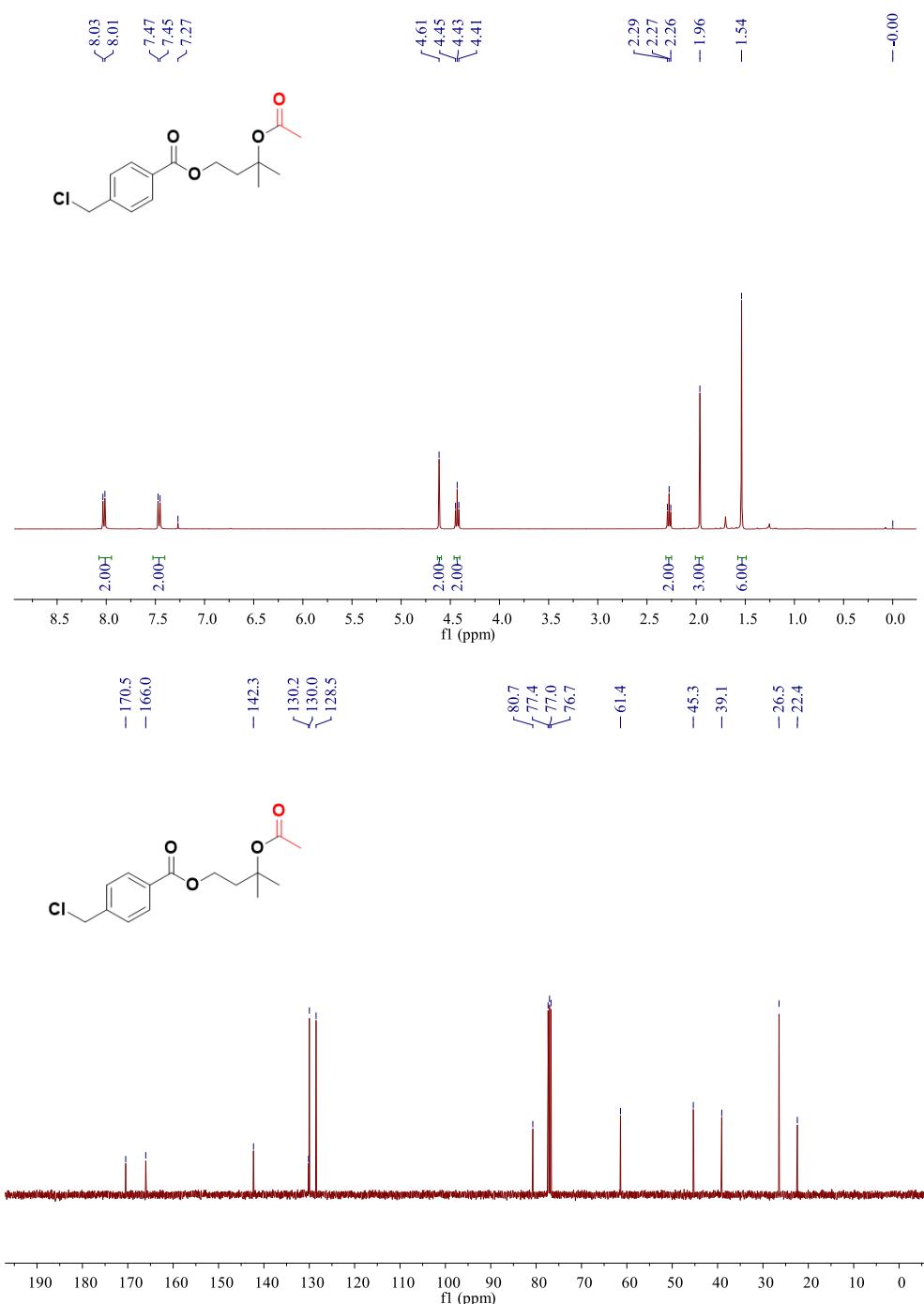
petroleum ether / ethyl acetate = 20:1, yellow oil, 40% yield (25.6 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.11 – 8.09 (m, 2H), 7.67 – 7.61 (m, 4H), 7.49 – 7.45 (m, 2H), 7.41 – 7.38 (m, 1H), 4.45 (t, *J* = 6.8 Hz, 2H), 2.29 (t, *J* = 6.8 Hz, 2H), 1.97 (s, 3H), 1.55 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 166.4, 145.7, 139.9, 130.0, 128.9, 128.1, 127.2, 127.0, 80.8, 61.2, 39.1, 26.5, 22.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>20</sub>H<sub>22</sub>O<sub>4</sub>+Na<sup>+</sup>: 349.1410, Found: 349.1405. **IR** (neat, cm<sup>-1</sup>): ν 2929, 1716, 1609, 1560, 1487, 1368, 1267, 1019, 727, 647.

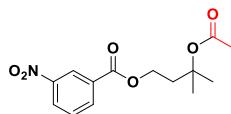




**3-Acetoxy-3-methylbutyl 4-(chloromethyl)benzoate (3bm)**

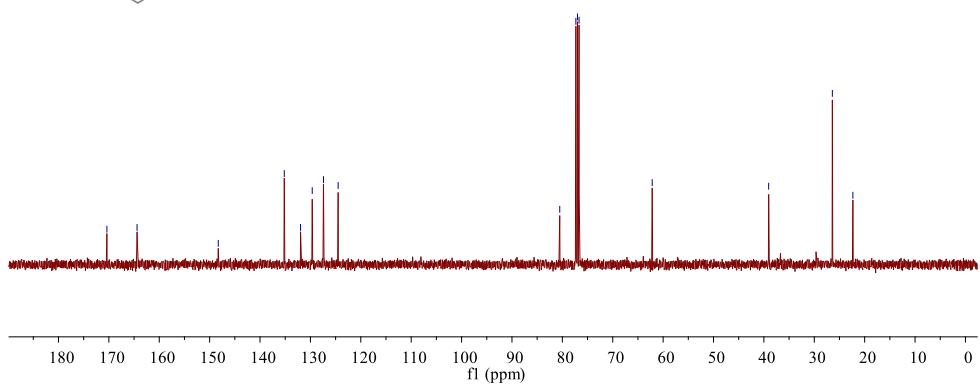
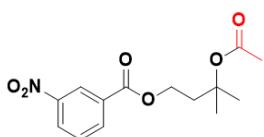
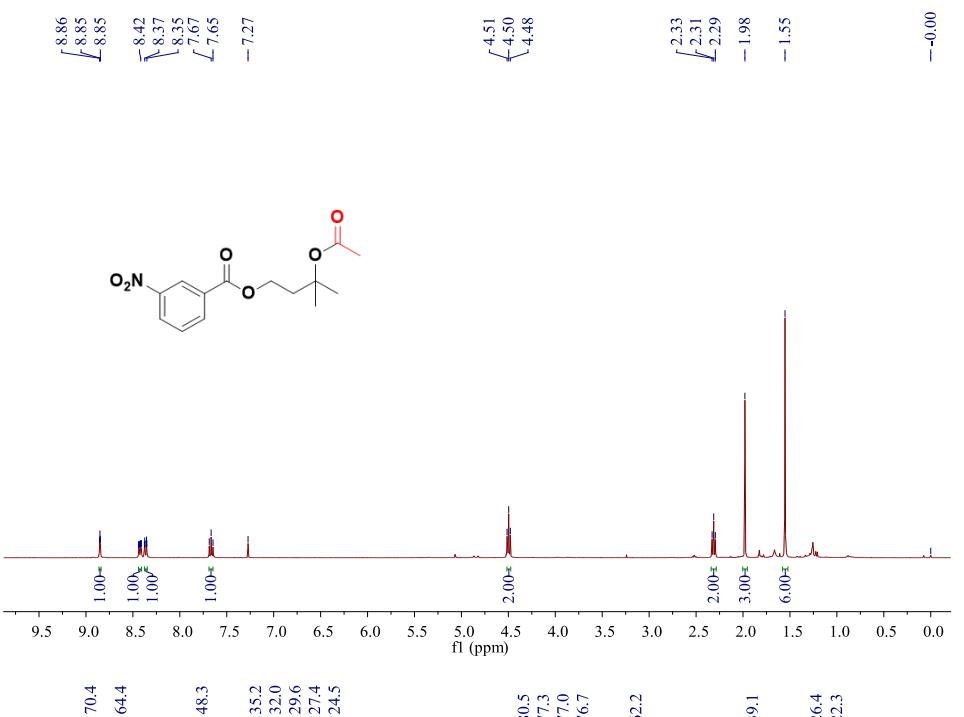
petroleum ether / ethyl acetate = 20:1, colorless oil, 65% yield (38.7 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 4.61 (s, 2H), 4.43 (t, *J* = 6.8 Hz, 2H), 2.27 (t, *J* = 6.8 Hz, 2H), 1.96 (s, 3H), 1.54 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.5, 166.0, 142.3, 130.2, 130.0, 128.5, 80.7, 61.4, 45.3, 39.1, 26.5, 22.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>19</sub><sup>35</sup>ClO<sub>4</sub> +Na<sup>+</sup>: 321.0864, Found: 321.0863; Anal Calcd. For. C<sub>15</sub>H<sub>19</sub><sup>37</sup>ClO<sub>4</sub> +Na<sup>+</sup>: 323.0835, Found: 323.0834. **IR** (neat, cm<sup>-1</sup>): ν 2936, 1713, 1613, 1454, 1369, 1222, 948, 726.

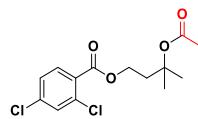




**3-Acetoxy-3-methylbutyl 3-nitrobenzoate (3bn)**

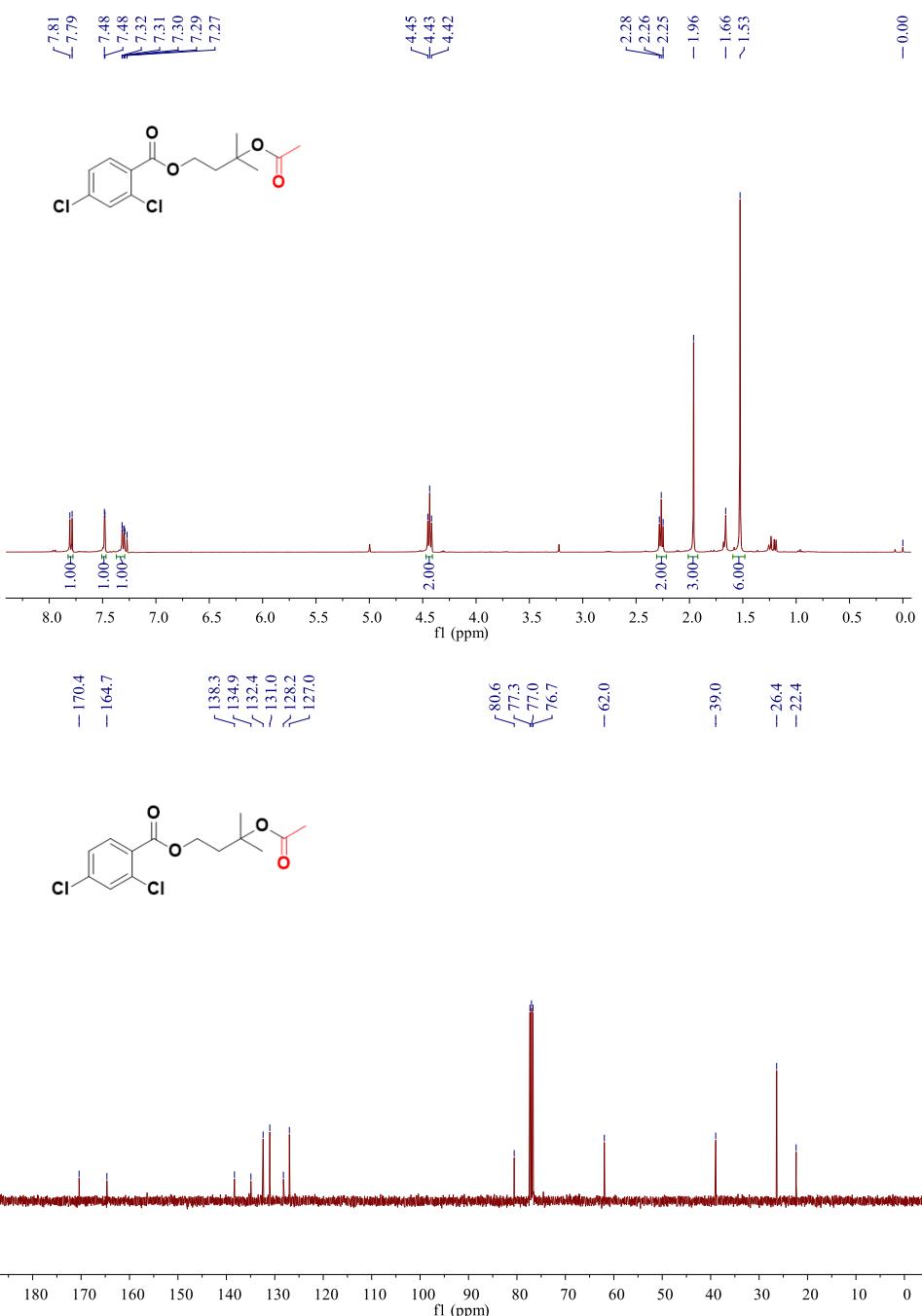
petroleum ether / ethyl acetate = 10:1, yellow oil, 65% yield (38.1 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.86 – 8.85 (m, 1H), 8.44 – 8.41 (m, 1H), 8.38 – 8.35 (m, 1H), 7.69 – 7.65 (m, 1H), 4.50 (t, *J* = 7.0 Hz, 2H), 2.31 (t, *J* = 7.0 Hz, 2H), 1.98 (s, 3H), 1.55 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.4, 164.4, 148.3, 135.2, 132.0, 129.6, 127.4, 124.5, 80.5, 62.2, 39.1, 26.4, 22.3. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>17</sub>NO<sub>6</sub>+Na<sup>+</sup>: 318.0948, Found: 318.0947. **IR** (neat, cm<sup>-1</sup>): ν 2932, 1722, 1617, 1532, 1440, 1387, 1294, 1096, 915, 822, 716, 652.

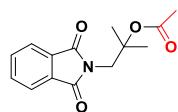




**3-Acetoxy-3-methylbutyl 2,4-dichlorobenzoate (3bo)**

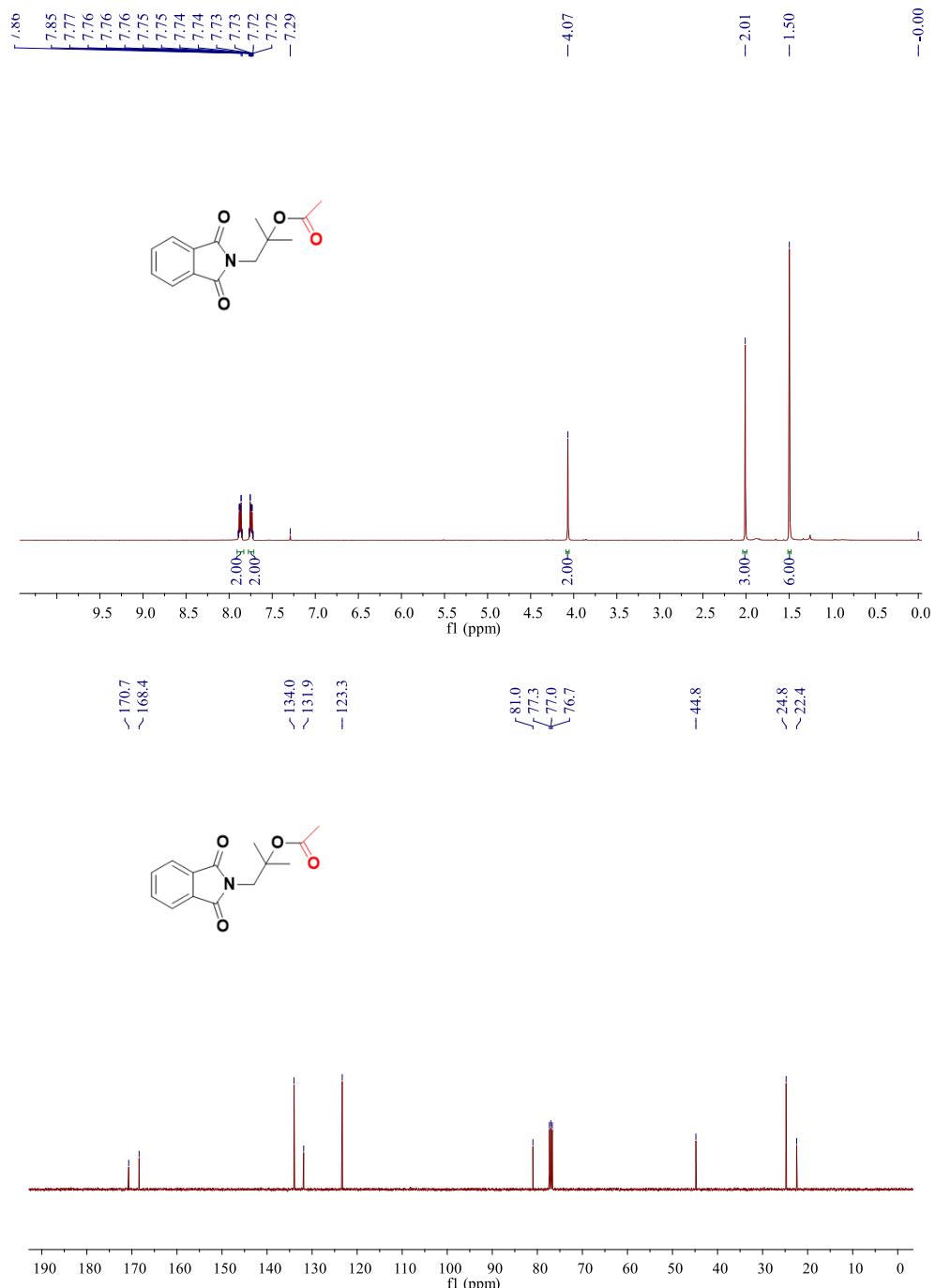
petroleum ether / ethyl acetate = 20:1, colorless oil, 60% yield (37.2 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.80 (d, *J* = 8.4 Hz, 1H), 7.48 (d, *J* = 2.0 Hz, 1H), 7.30 (dd, *J* = 8.4, 2.0 Hz, 1H), 4.43 (t, *J* = 7.0 Hz, 2H), 2.26 (t, *J* = 7.0 Hz, 2H), 1.96 (s, 3H), 1.53 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.4, 164.7, 138.3, 134.9, 132.4, 131.0, 128.2, 127.0, 80.6, 62.0, 39.0, 26.4, 22.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>16</sub>Cl<sub>2</sub>O<sub>4</sub>+Na<sup>+</sup>: 341.0318, Found: 341.0313; Anal Calcd. For. C<sub>14</sub>H<sub>16</sub><sup>35</sup>Cl<sup>37</sup>ClO<sub>4</sub>+Na<sup>+</sup>: 343.0288, Found: 343.0284; Anal Calcd. For. C<sub>14</sub>H<sub>16</sub><sup>37</sup>Cl<sub>2</sub>O<sub>4</sub>+Na<sup>+</sup>: 345.0259, Found: 345.0254. **IR** (neat, cm<sup>-1</sup>): ν 2900, 1736, 1624, 1582, 1418, 1228, 956, 727, 648.

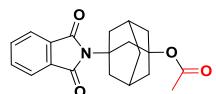




**1-(1,3-Dioxoisindolin-2-yl)-2-methylpropan-2-yl acetate (3bp)**

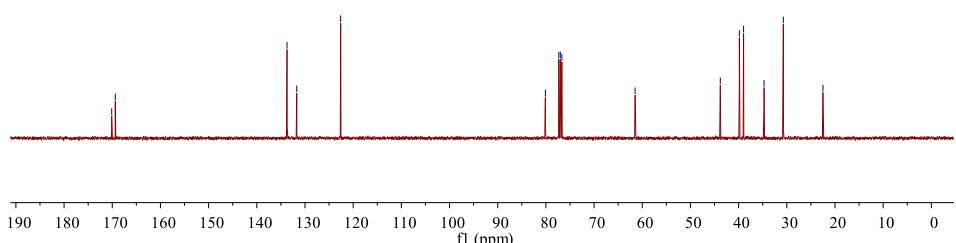
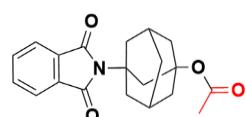
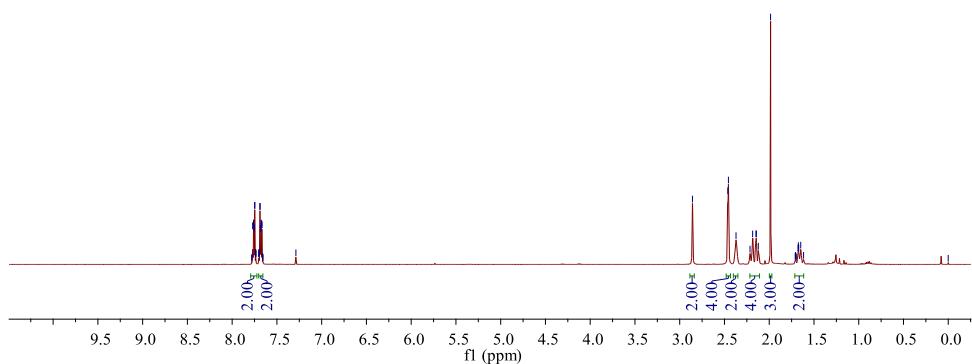
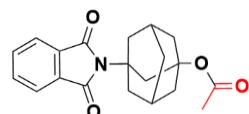
petroleum ether / ethyl acetate = 10:1, white solid, 70% yield (36.0 mg). mp: 90 – 92°C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.90 – 7.85 (m, 2H), 7.77 – 7.72 (m, 2H), 4.07 (s, 2H), 2.01 (s, 3H), 1.50 (s, 6H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 170.7, 168.4, 134.0, 131.9, 123.3, 81.0, 44.8, 24.8, 22.4. HRMS (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>15</sub>NO<sub>4</sub> +Na<sup>+</sup>: 284.0893, Found: 284.0892. IR (neat, cm<sup>-1</sup>): ν 2850, 1769, 1614, 1515, 1464, 1339, 1224, 1076, 727, 606.

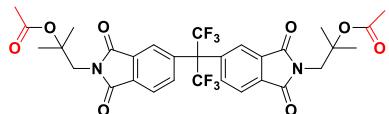




### 3-(1,3-Dioxoisooindolin-2-yl)adamantan-1-yl acetate (3bq)

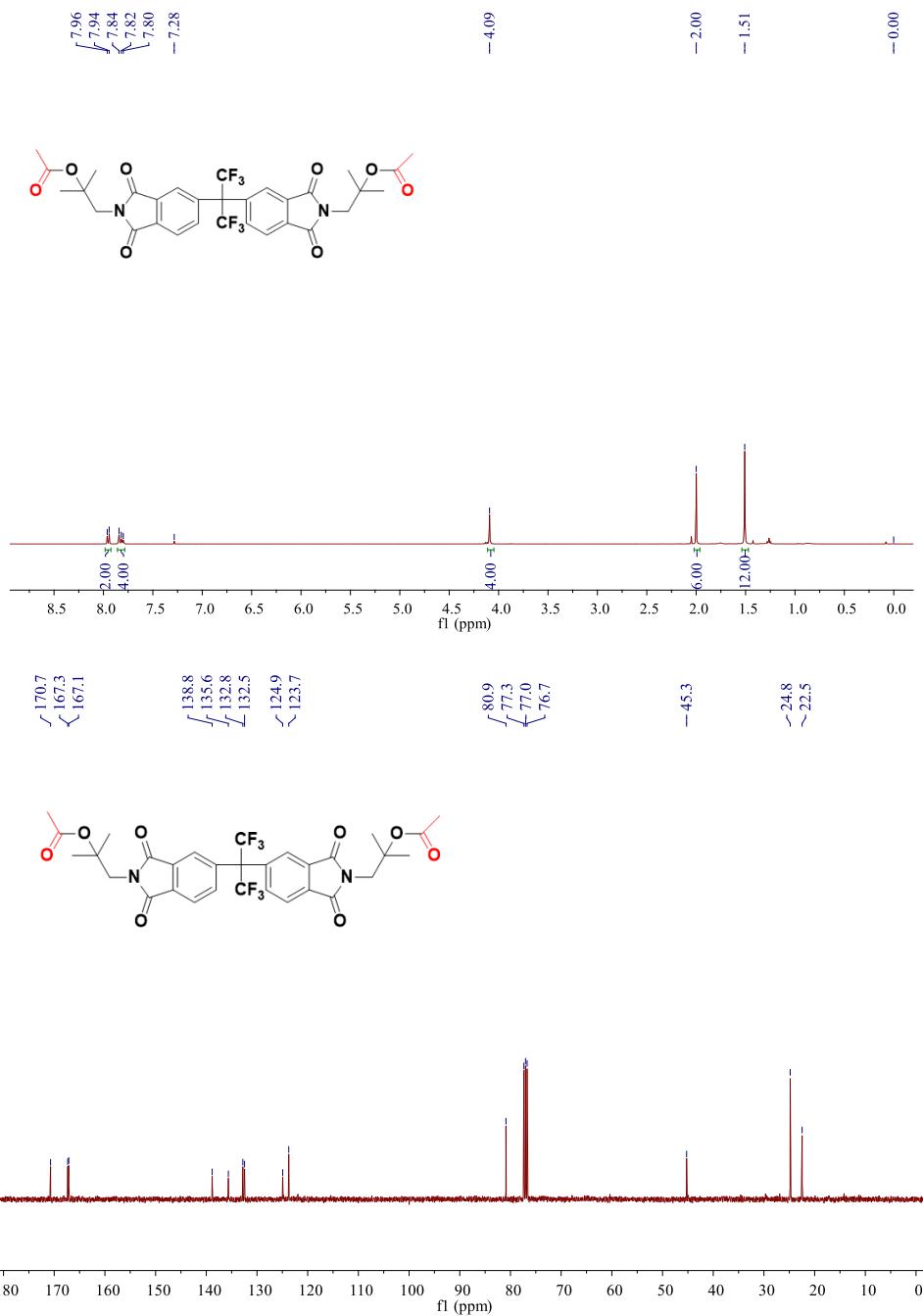
petroleum ether / ethyl acetate = 5:1, white solid, 80% yield (53.5 mg). mp: 103 – 105°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.78 – 7.74 (m, 2H), 7.70 – 7.66 (m, 2H), 2.86 (s, 2H), 2.46 (d, *J* = 2.8 Hz, 4H), 2.37 (s, 2H), 2.21 – 2.12 (m, 4H), 1.98 (s, 3H), 1.71 – 1.62 (m, 2H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.1, 169.4, 133.7, 131.7, 122.6, 80.1, 61.5, 43.8, 39.8, 39.0, 34.7, 30.7, 22.5. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>20</sub>H<sub>21</sub>NO<sub>4</sub> +Na<sup>+</sup>: 362.1363, Found: 362.1361. **IR** (neat, cm<sup>-1</sup>): ν 2917, 1729, 1697, 1558, 1250, 958, 863, 711, 676.

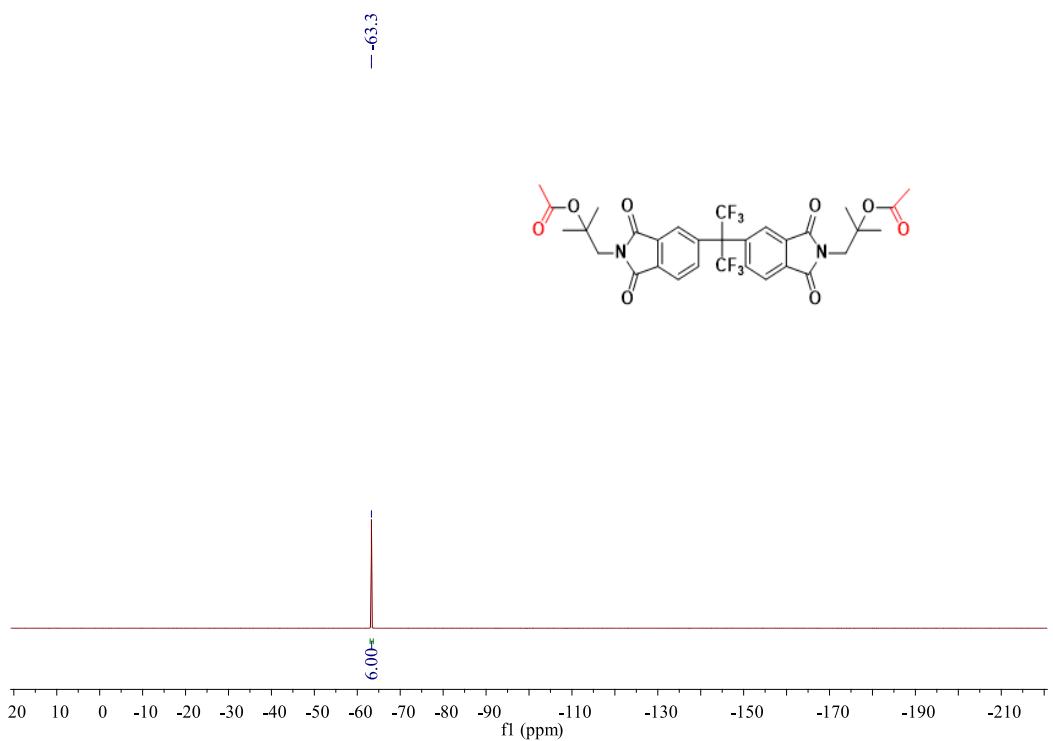


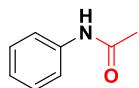


**(Perfluoropropane-2,2-diyl)bis(1,3-dioxoisindoline-5,2-diyl)bis(2-methylpropane-1,2-diyl) diacetate (3br)**

petroleum ether / ethyl acetate = 5:1, white solid, 60% yield (78.4 mg). mp: 145 – 147°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.84 – 7.80 (m, 4H), 4.09 (s, 4H), 2.00 (s, 6H), 1.51 (s, 12H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.7, 167.3, 167.1, 138.8, 135.6, 132.8, 132.5, 124.9, 123.7, 80.9, 45.3, 24.8, 22.5. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -63.3 (s, 6F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>31</sub>H<sub>28</sub>F<sub>6</sub>N<sub>2</sub>O<sub>8</sub>+Na<sup>+</sup>: 693.1642, Found: 693.1632. **IR** (neat, cm<sup>-1</sup>): ν 2942, 1778, 1465, 1426, 1202, 1168, 1016, 727, 606.

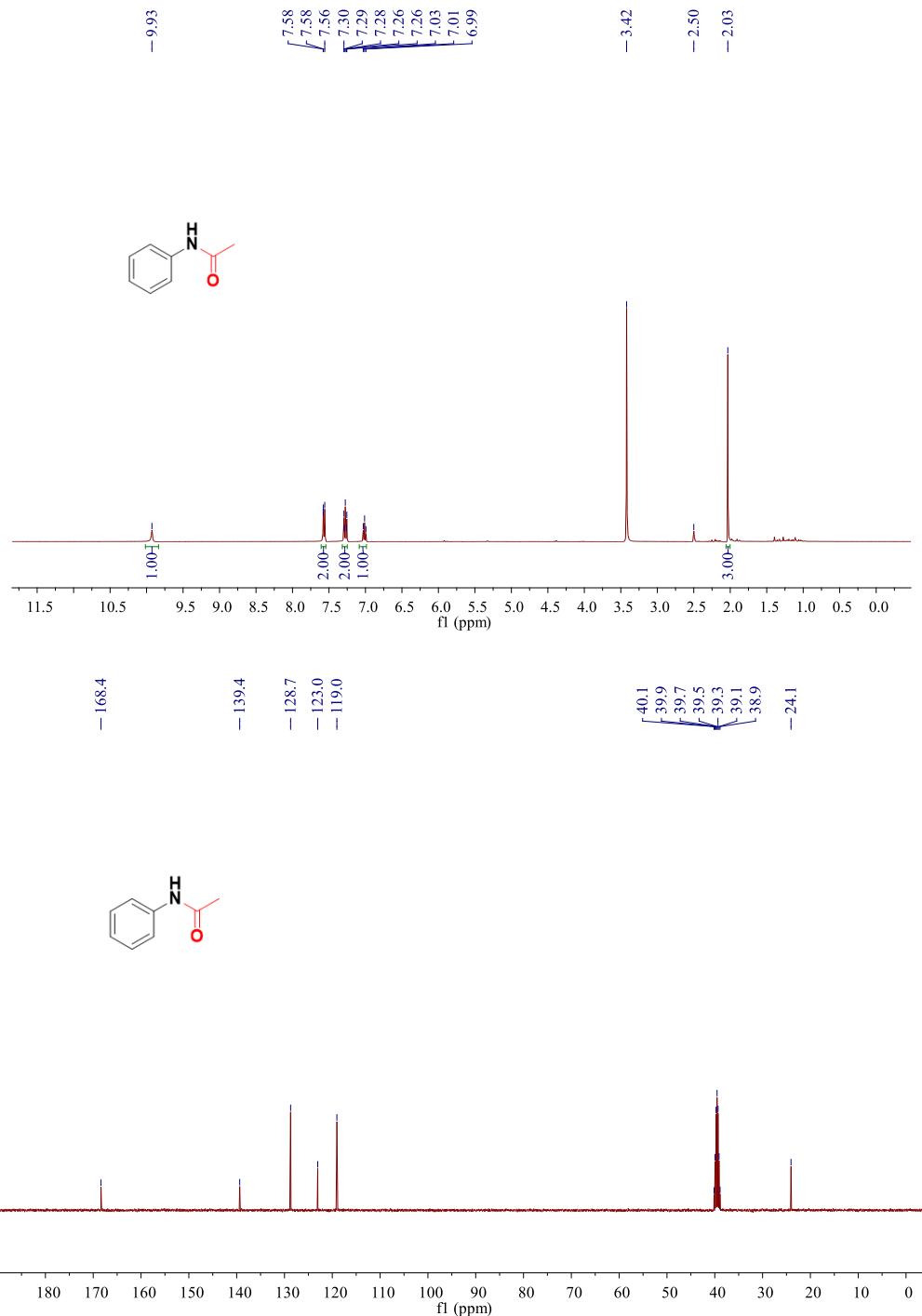


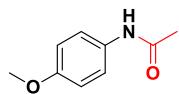




**N-Phenylacetamide (5aa)**

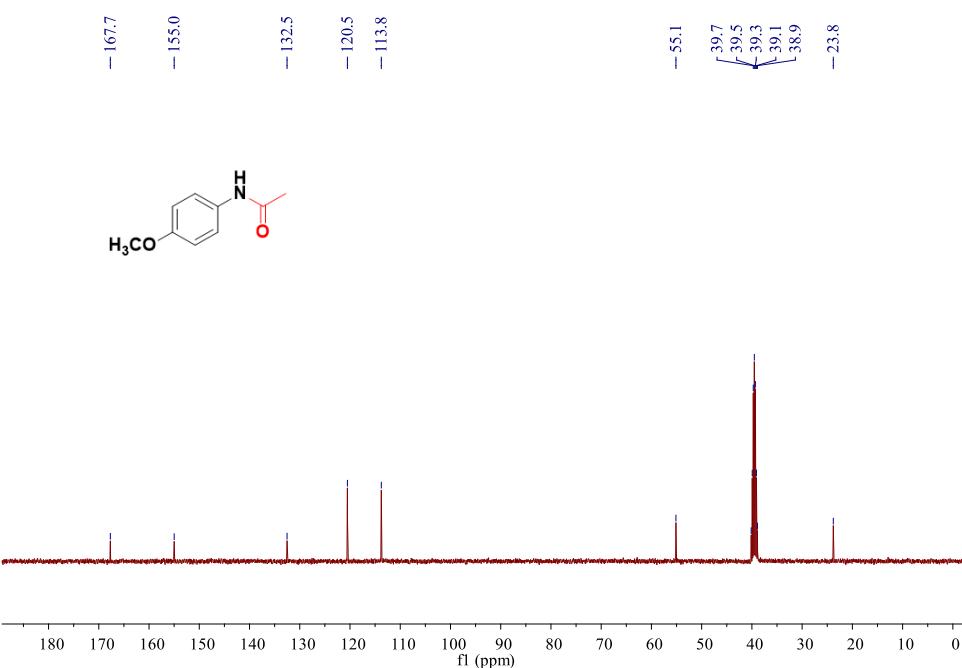
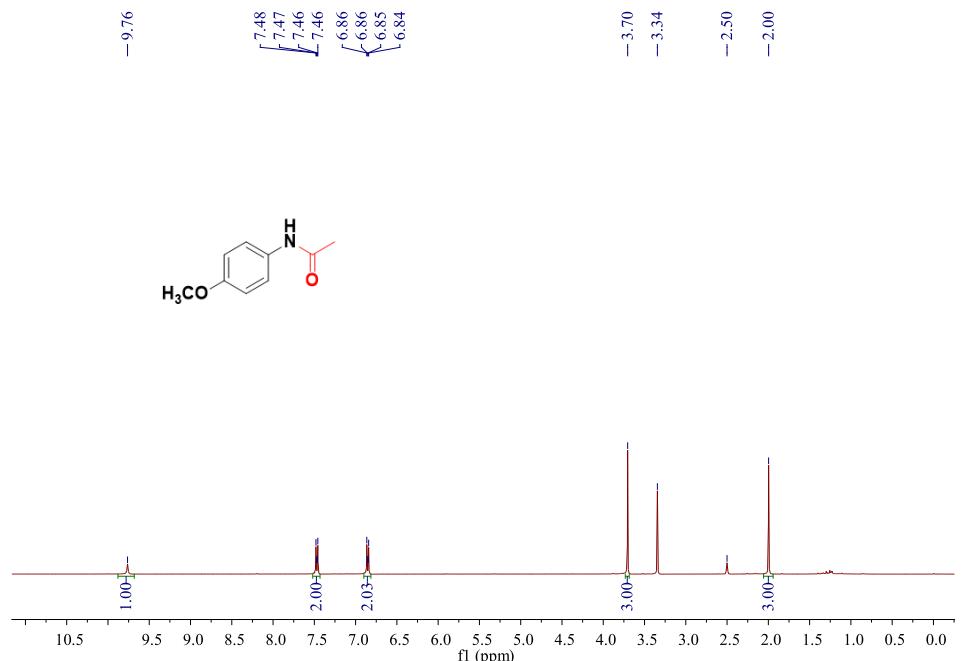
dichloromethane / ethyl acetate = 5:1, white solid, 93% yield (22.0 mg). mp: 113 – 115°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.93 (s, 1H), 7.58 – 7.56 (m, 2H), 7.30 – 7.26 (m, 2H), 7.01 (t, *J* = 7.4 Hz, 1H), 2.03 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.4, 139.4, 128.7, 123.0, 119.0, 24.1. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>9</sub>NO +H<sup>+</sup>: 136.0757, Found: 136.0757. **IR** (neat, cm<sup>-1</sup>): ν 3587, 2900, 1732, 1683, 1598, 1496, 1373, 1023, 822, 759, 696.

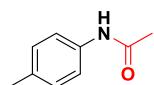




**N-(4-Methoxyphenyl)acetamide (5ab)**

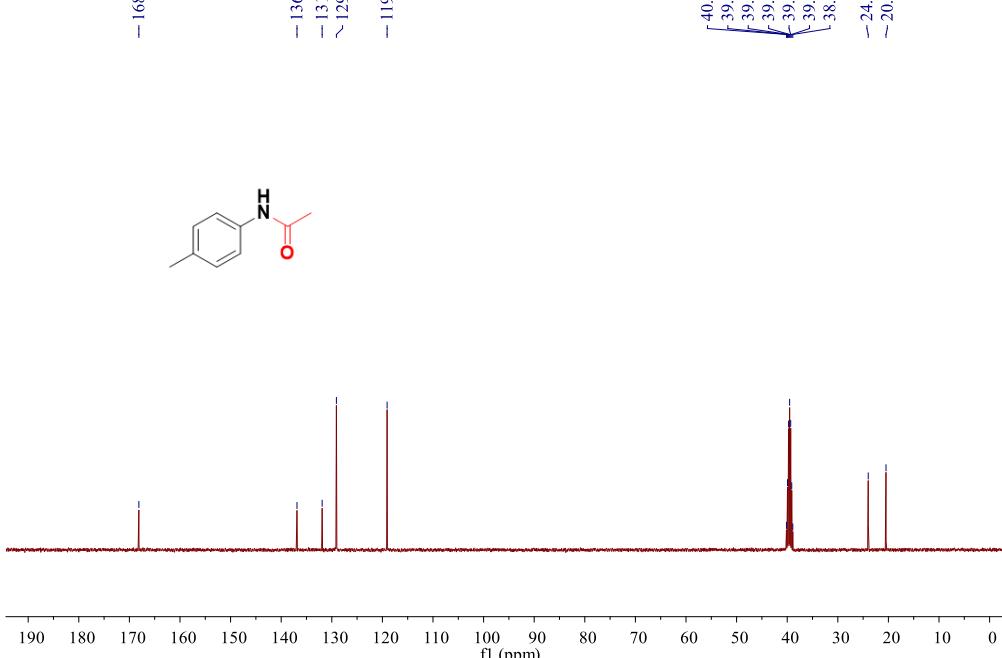
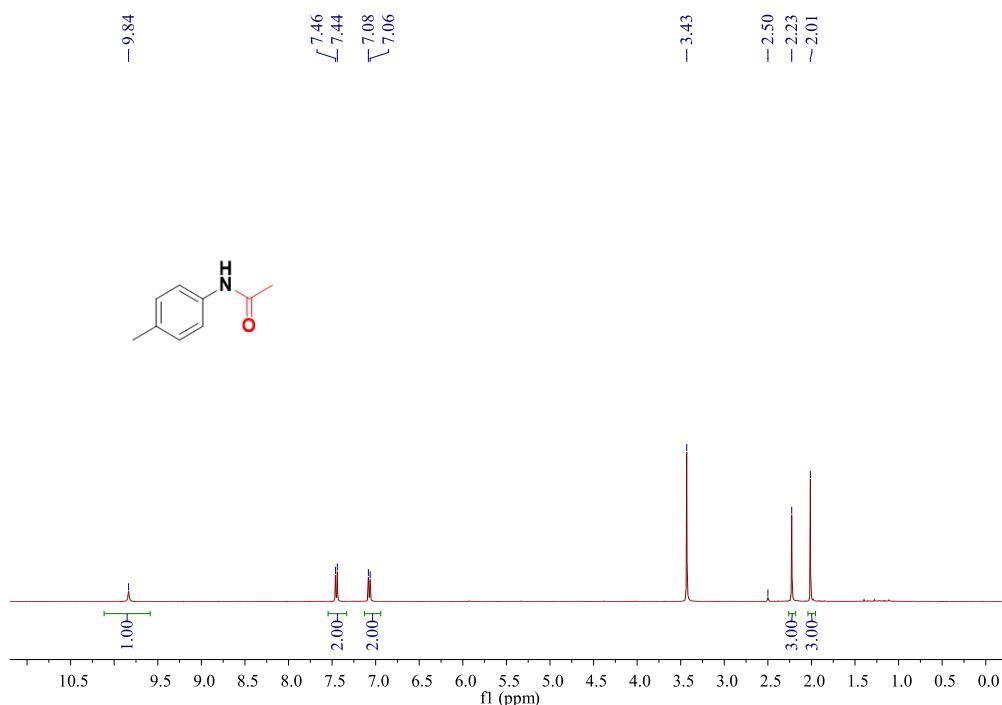
dichloromethane / ethyl acetate = 5:1, yellow solid, 69% yield (20.3 mg). mp: 126 – 128°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.76 (s, 1H), 7.48 – 7.46 (m, 2H), 6.86 – 6.84 (m, 2H), 3.70 (s, 3H), 2.00 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 167.7, 155.0, 132.5, 120.5, 113.8, 55.1, 23.8. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>11</sub>NO<sub>2</sub>+H<sup>+</sup>: 166.0863, Found: 166.0862. **IR** (neat, cm<sup>-1</sup>): ν 3365, 1645, 1553, 1512, 1246, 1024, 990, 826, 763.

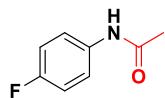




**N-(p-Tolyl)acetamide (5ac)**

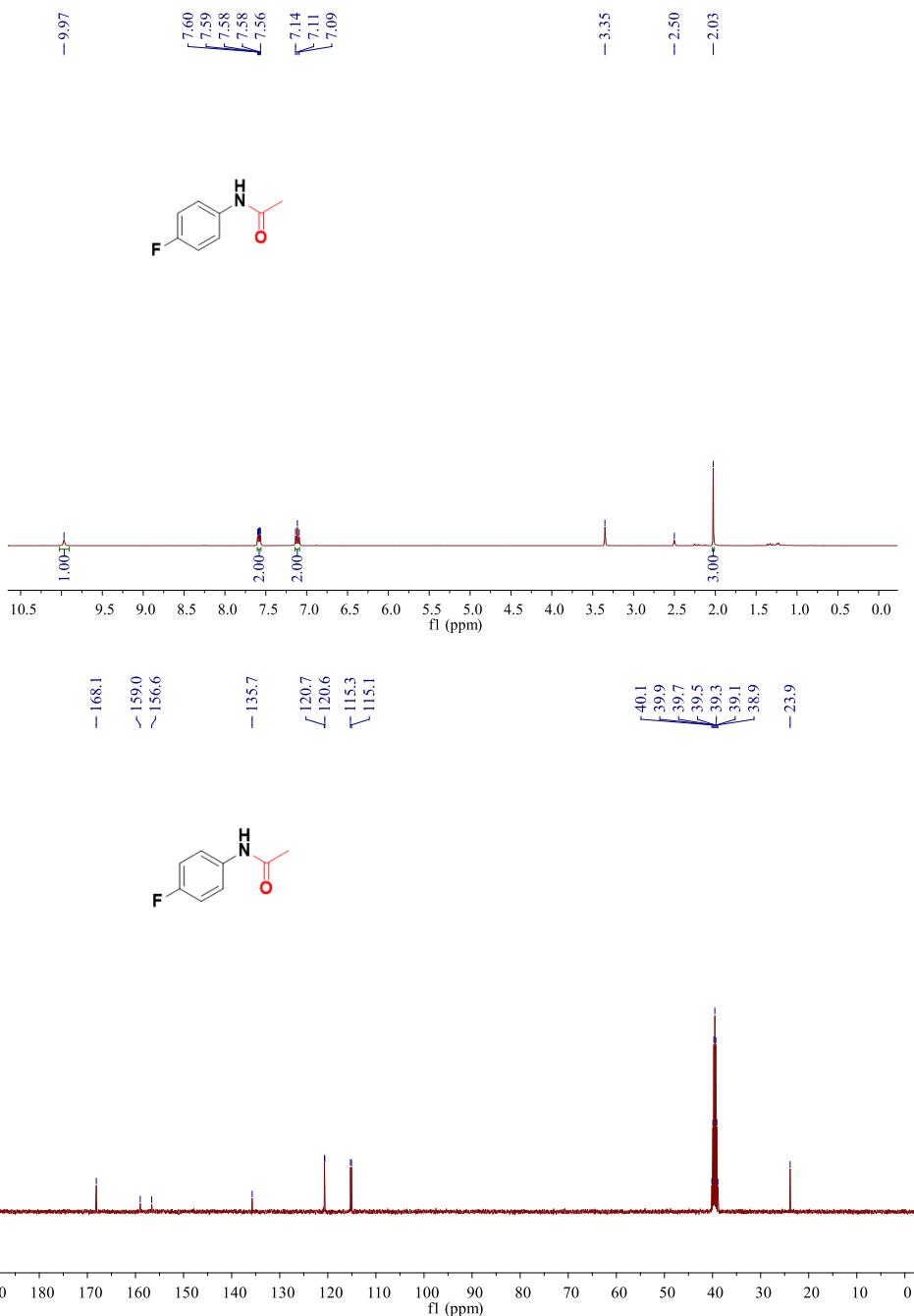
dichloromethane / ethyl acetate = 5:1, yellow solid, 93% yield (23.6 mg). mp: 151 – 153°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.84 (s, 1H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 2.23 (s, 3H), 2.01 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.1, 136.9, 131.9, 129.1, 119.1, 24.0, 20.5. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>11</sub>NO+H<sup>+</sup>: 150.0913, Found: 150.0913. **IR** (neat, cm<sup>-1</sup>): ν 3298, 2976, 1662, 1590, 1488, 922, 816, 729.

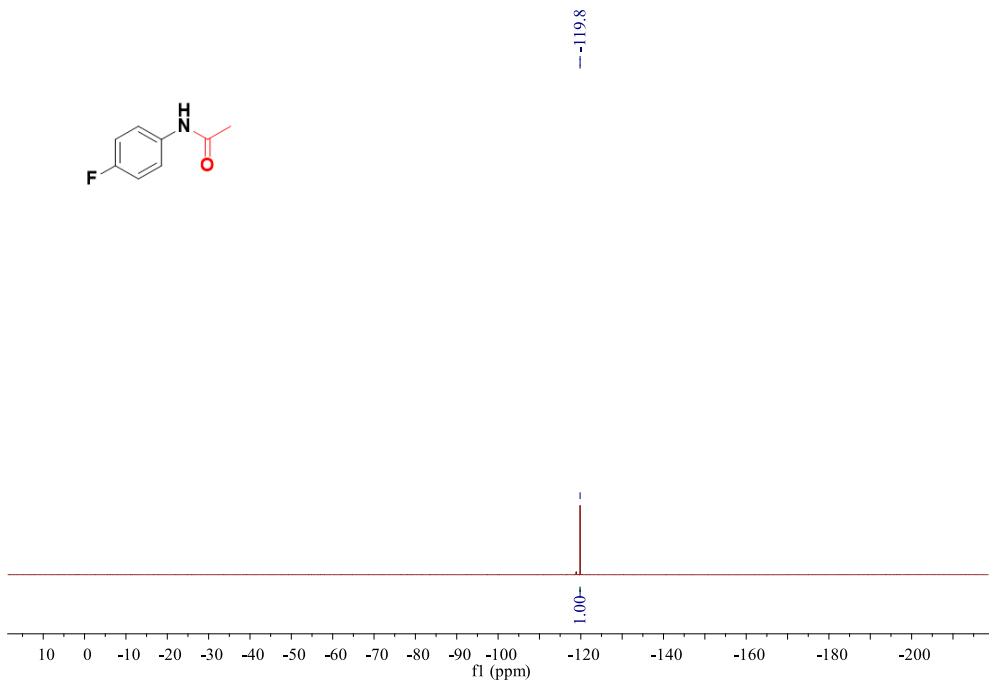


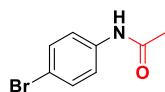


**N-(4-Fluorophenyl)acetamide (5ad)**

dichloromethane / ethyl acetate = 5:1, yellow solid, 83% yield (24.2 mg). mp: 150 – 152°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.97 (s, 1H), 7.60 – 7.56 (m, 2H), 7.14–7.09 (m, 2H), 2.03 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.1, 157.8 (d, *J* = 237.0 Hz), 135.7 (d, *J* = 3.0 Hz), 120.7 (d, *J* = 7.0 Hz), 115.2 (d, *J* = 22 Hz), 23.9. **<sup>19</sup>F NMR** (377 MHz, DMSO) δ -119.8 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub>FNO+H<sup>+</sup>: 154.0663, Found: 154.0661; **IR** (neat, cm<sup>-1</sup>): ν 3360, 1645, 1509, 1406, 1377, 1213, 1024, 989, 826.

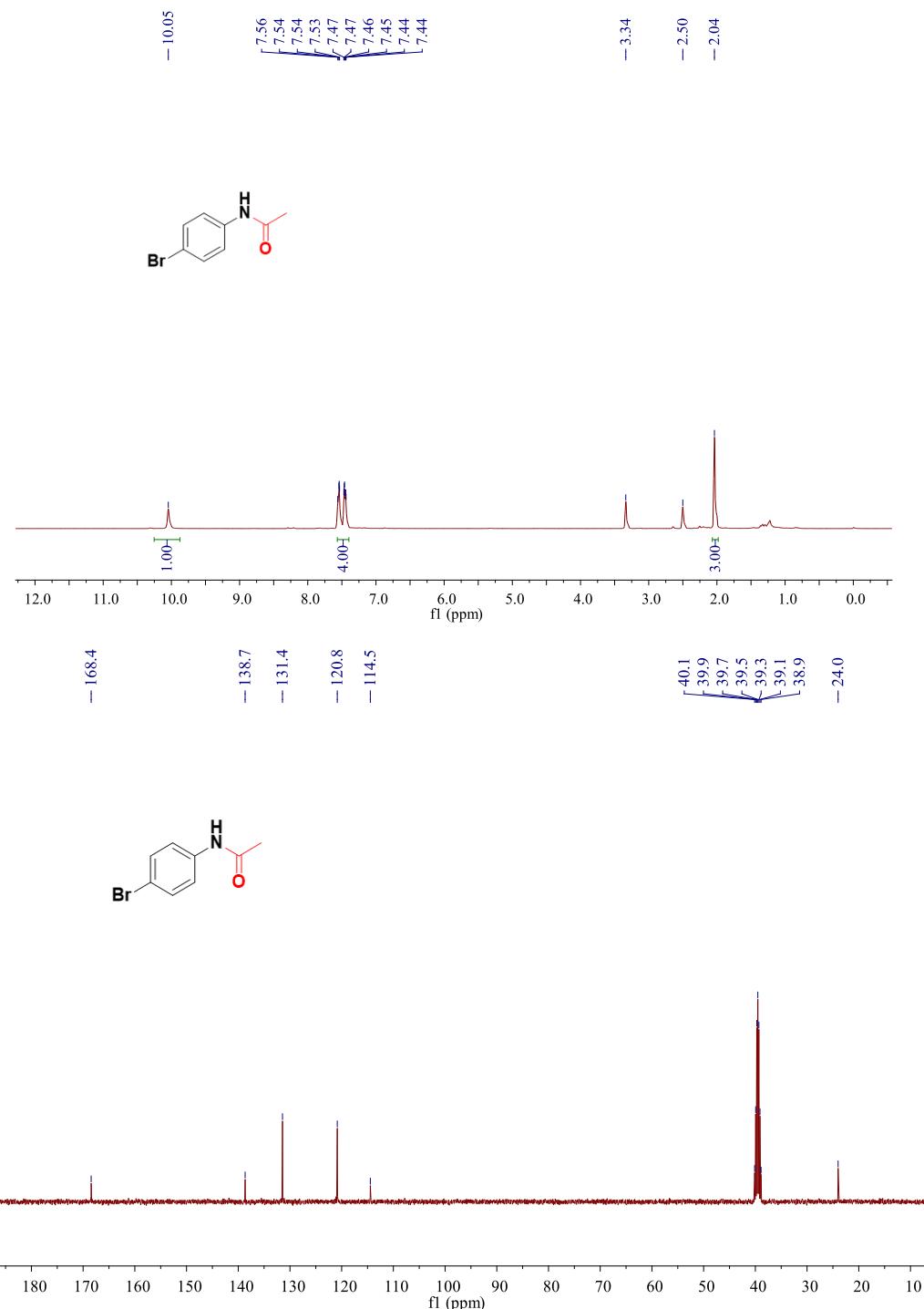


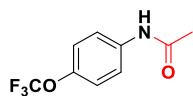




**N-(4-Bromophenyl)acetamide (5ae)**

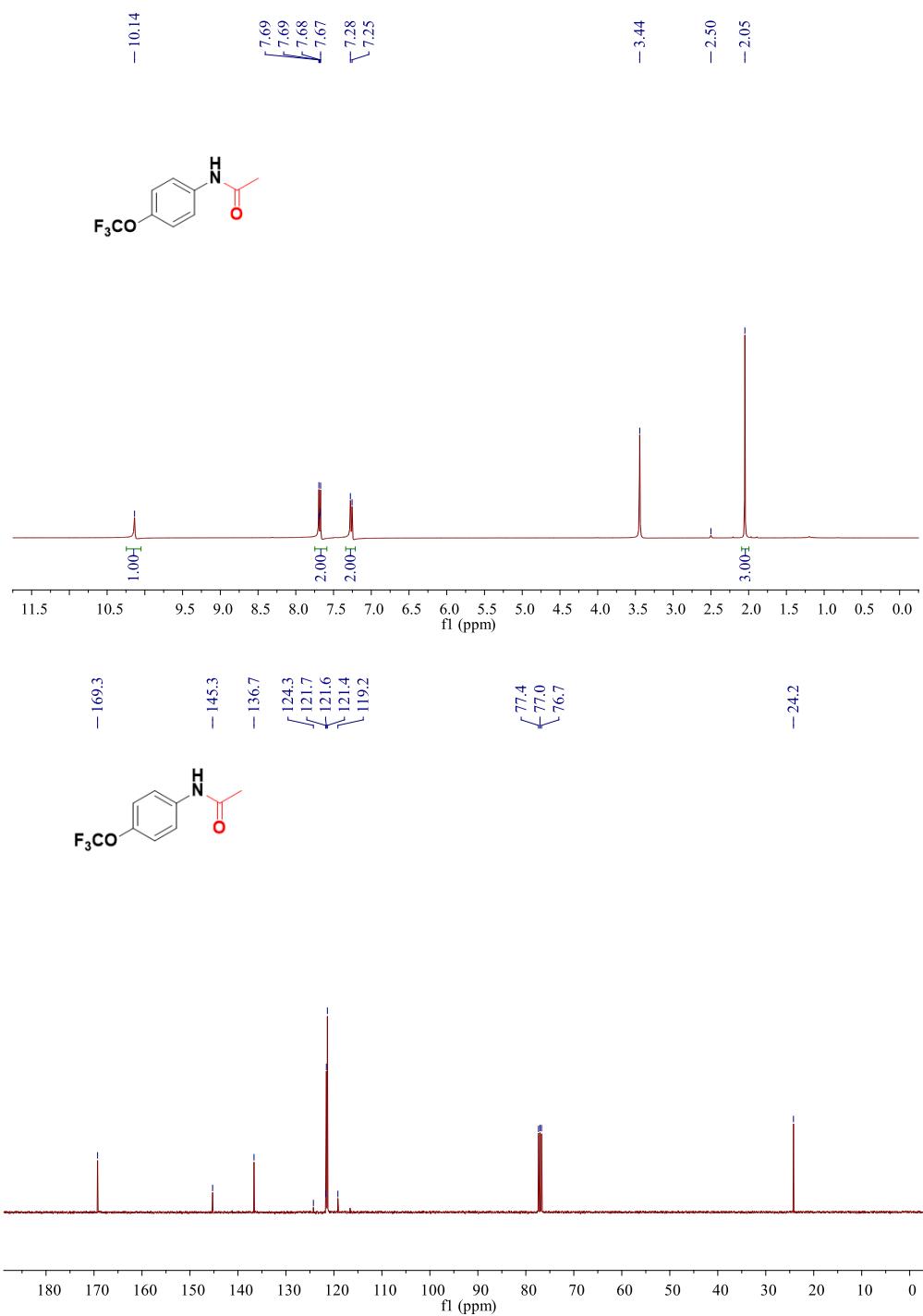
dichloromethane / ethyl acetate = 5:1, yellow solid, 85% yield (36.2 mg). mp: 168 – 170°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.05 (s, 1H), 7.56 – 7.44 (m, 4H), 2.04 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.4, 138.7, 131.4, 120.8, 114.5, 24.0. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub><sup>79</sup>BrNO+H<sup>+</sup>: 213.9862, Found: 213.9860; C<sub>8</sub>H<sub>8</sub><sup>81</sup>BrNO+H<sup>+</sup>: 215.9842, Found: 215.9840. **IR** (neat, cm<sup>-1</sup>): ν 3376, 2851, 1649, 1538, 1491, 1396, 1258, 1046, 991, 825, 763.

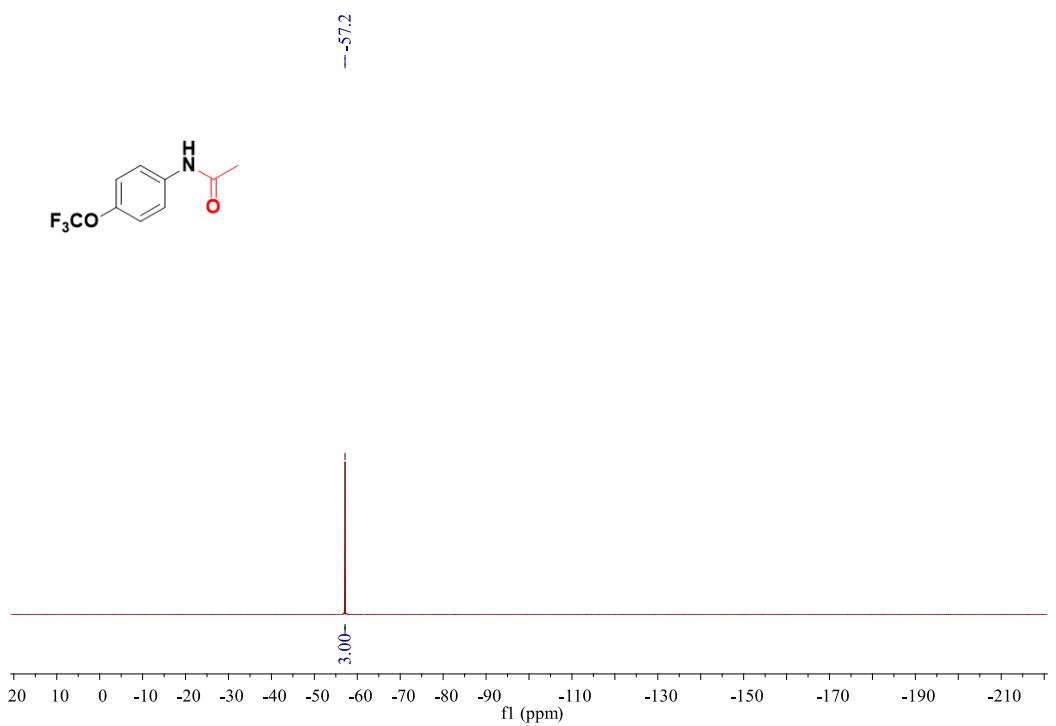


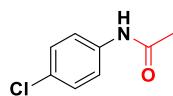


***N*-(4-(Trifluoromethoxy)phenyl)acetamide (5af)**

dichloromethane / ethyl acetate = 5:1, yellow solid, 90% yield (39.6 mg). mp: 113 – 115°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  10.14 (s, 1H), 7.69 – 7.67 (m, 2H), 7.28 – 7.25 (m, 2H), 2.05 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 145.3, 136.7, 121.6, 121.4, 120.5 (q,  $J = 255$  Hz), 24.2.  **$^{19}\text{F}$  NMR** (377 MHz, DMSO)  $\delta$  -57.1 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_8\text{F}_3\text{NO}_2+\text{H}^+$ : 220.0580, Found: 220.0578. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3378, 3270, 1665, 1618, 1556, 1508, 1154, 992, 825, 762, 659.

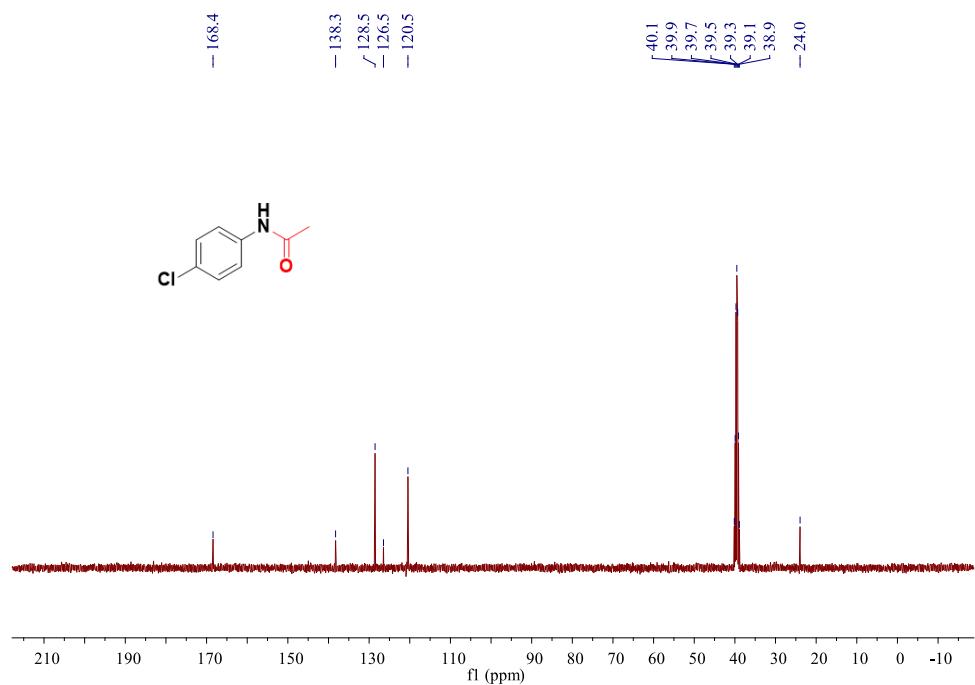
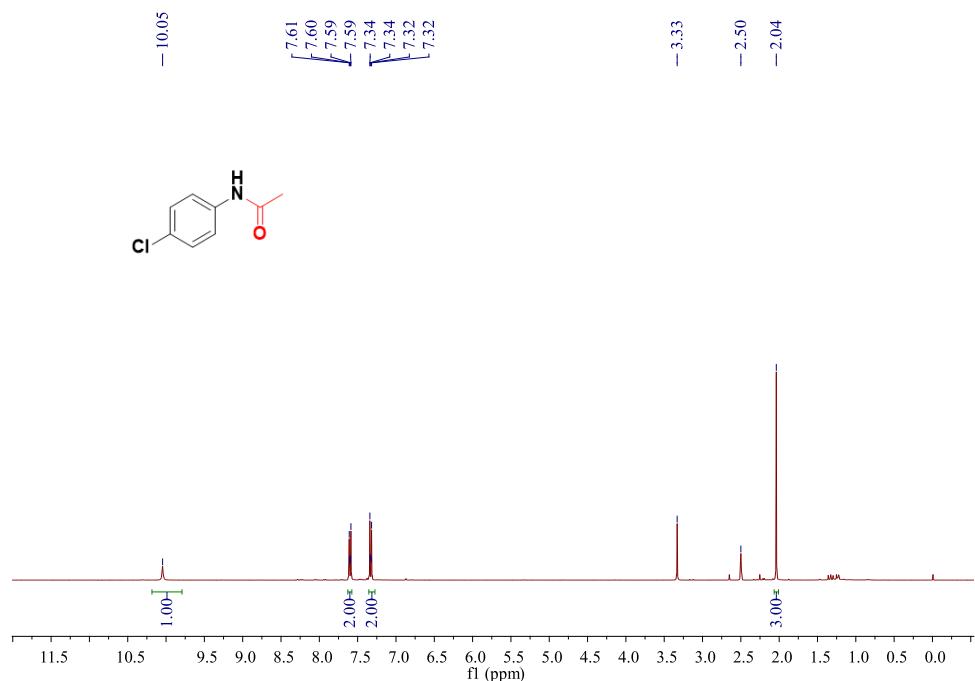


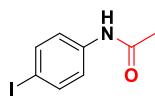




**N-(4-Chlorophenyl)acetamide (5ag)**

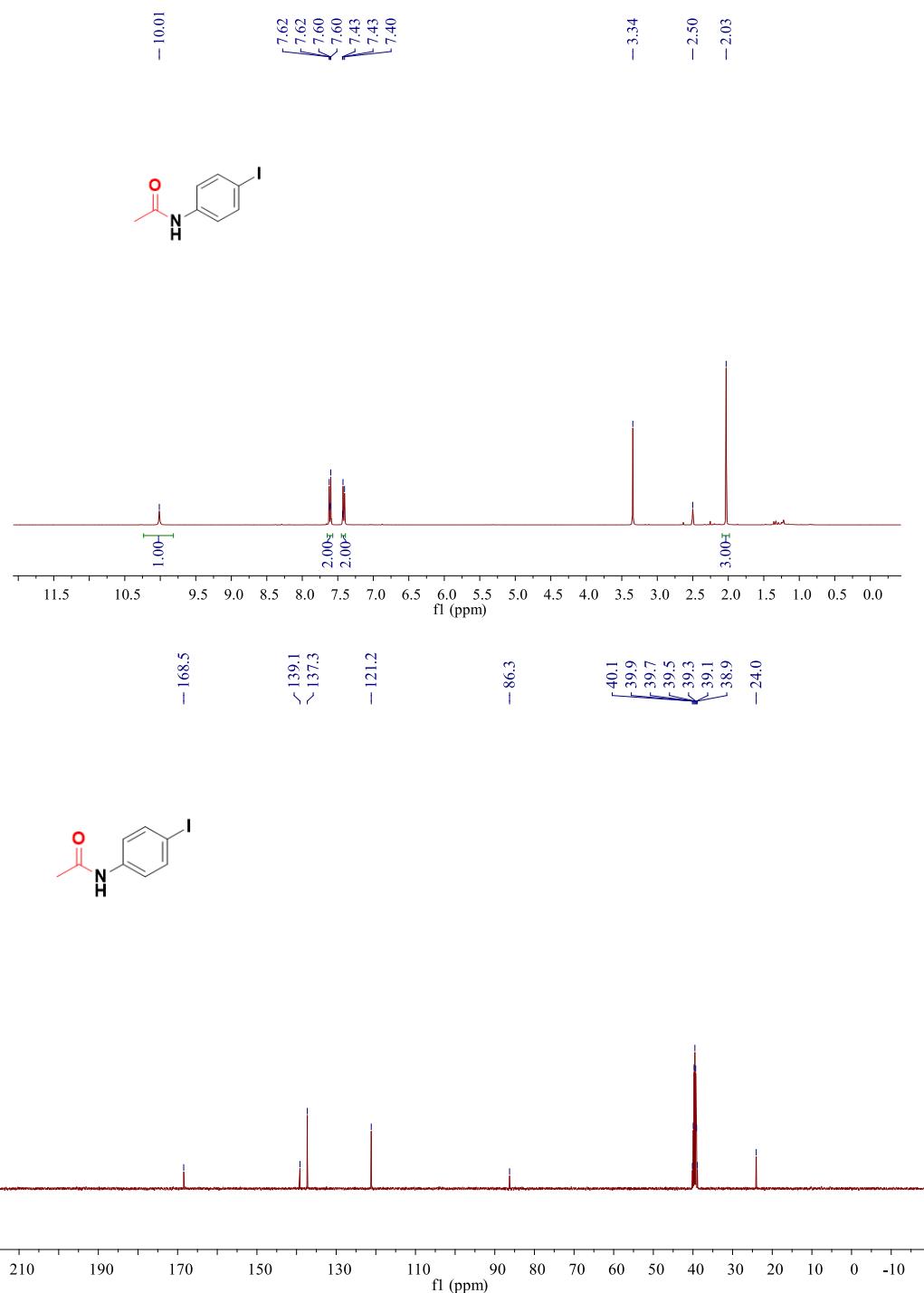
dichloromethane / ethyl acetate = 5:1, yellow solid, 86% yield (29.0 mg). mp: 177 – 179°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.05 (s, 1H), 7.61 – 7.59 (m, 2H), 7.34 – 7.32 (m, 2H), 2.04 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.4, 138.3, 128.5, 126.5, 120.5, 24.0. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub><sup>35</sup>ClNO+H<sup>+</sup>: 170.0367, Found: 170.0366; C<sub>8</sub>H<sub>8</sub><sup>37</sup>ClNO+H<sup>+</sup>: 172.0338, Found: 172.0340. **IR** (neat, cm<sup>-1</sup>): ν 3393, 2920, 1658, 1547, 1487, 1392, 1258, 1092, 996, 821, 709.

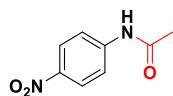




**N-(4-Iodophenyl)acetamide (5ah)**

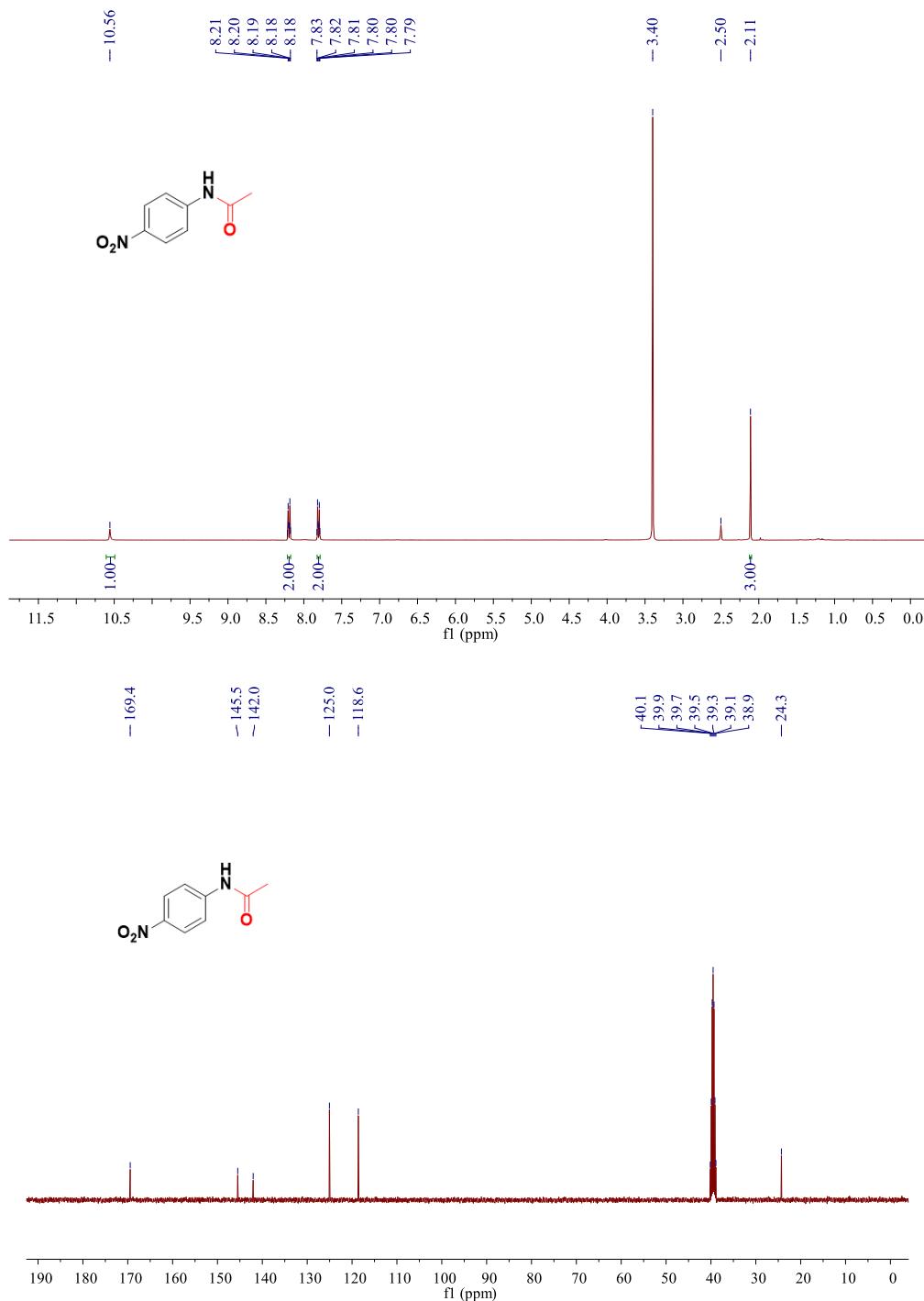
dichloromethane / ethyl acetate = 5:1, yellow solid, 77% yield (40.0 mg). mp: 180 – 182°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.01 (s, 1H), 7.62 – 7.60 (m, 2H), 7.43 – 7.40 (m, 2H), 2.03 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.5, 139.1, 137.3, 121.2, 86.3, 24.0. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub>INO+H<sup>+</sup>: 261.9723, Found: 261.9721. **IR** (neat, cm<sup>-1</sup>): ν 3392, 2958, 2849, 1663, 1596, 1579, 1482, 1253, 1024, 992, 814, 731, 679.

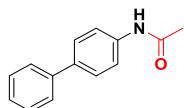




**N-(4-Nitrophenyl)acetamide (5ai)**

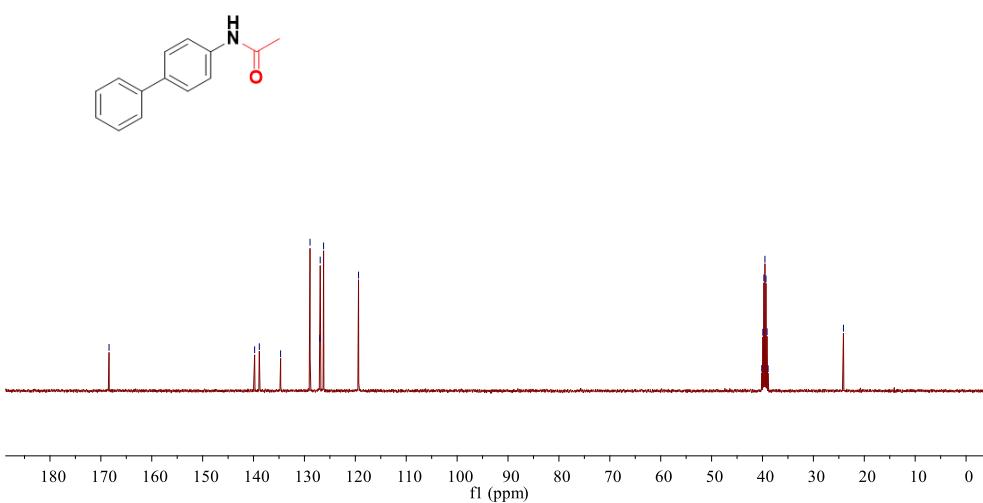
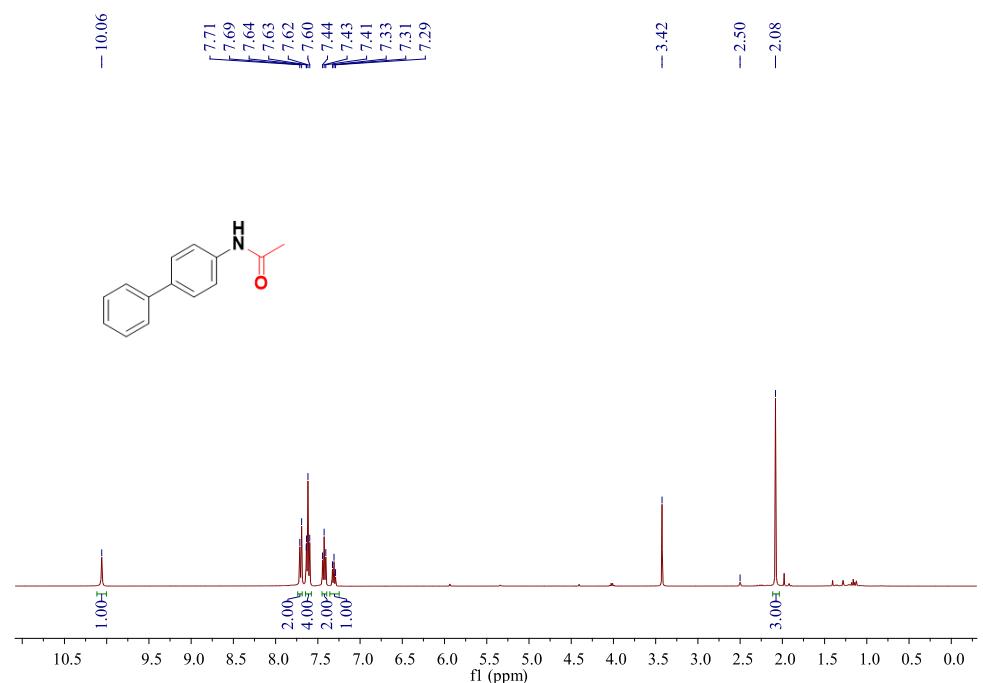
dichloromethane / ethyl acetate = 5:1, yellow solid, 46% yield (16.7 mg). mp: 208 – 210°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.56 (s, 1H), 8.21 – 8.18 (m, 2H), 7.83 – 7.79 (m, 2H), 2.11 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.4, 145.5, 142.0, 125.0, 118.6, 24.3. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub>N<sub>2</sub>O<sub>3</sub>+H<sup>+</sup>: 181.0608, Found: 181.0606. **IR** (neat, cm<sup>-1</sup>): ν 3414, 1654, 1616, 1597, 1264, 1023, 859, 759.

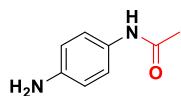




***N*-(**[1,1'-Biphenyl]-4-yl**)acetamide (**5aj**)**

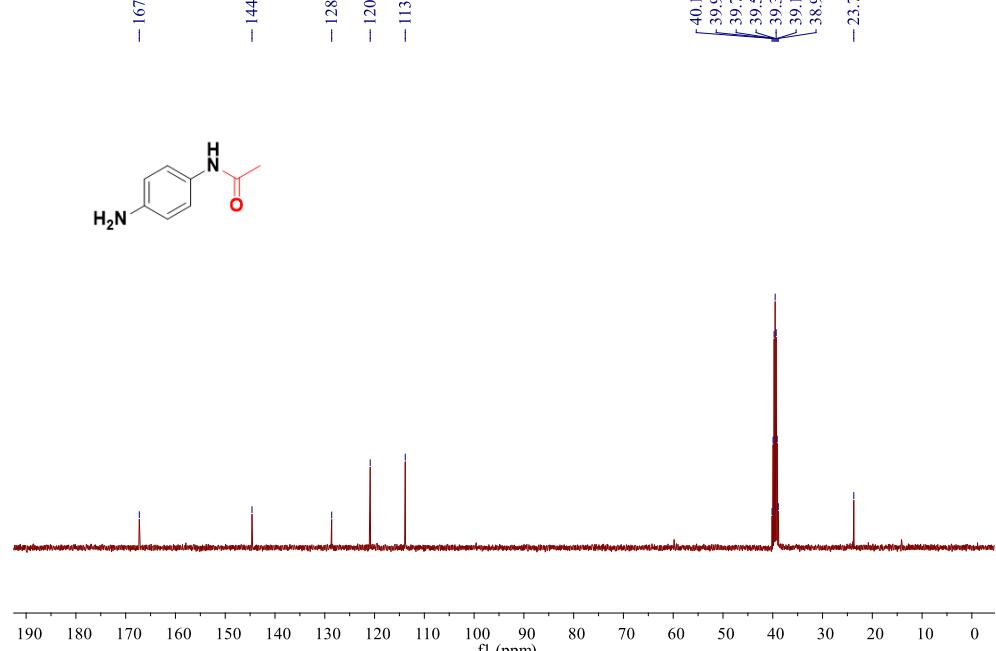
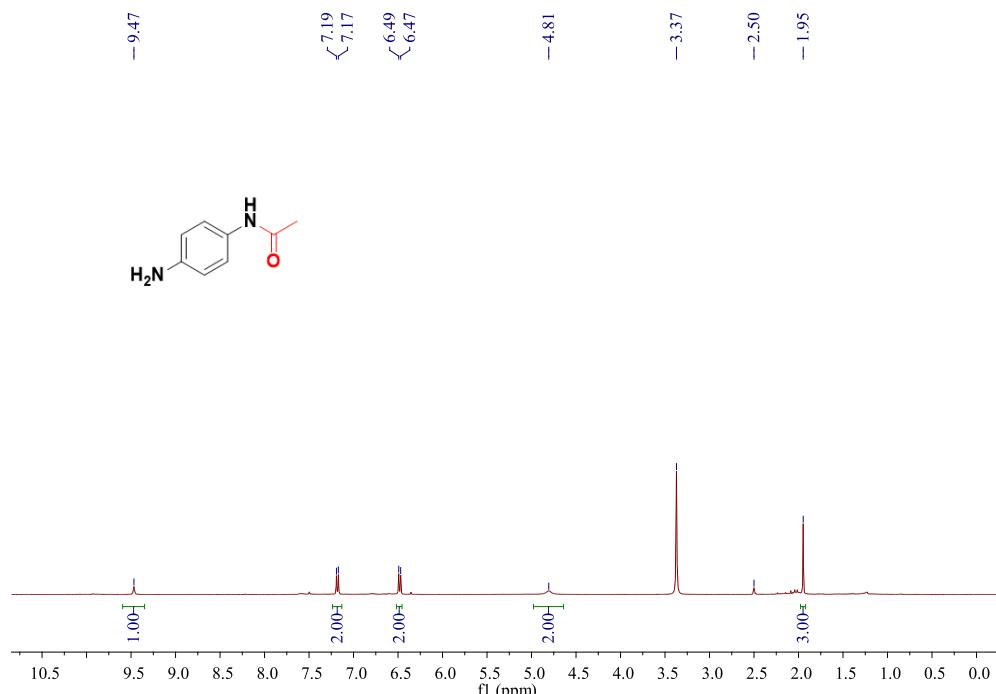
petroleum ether / ethyl acetate = 10:1, yellow solid, 67% yield (28.3 mg). mp: 171 – 173°C. **<sup>1</sup>H NMR** (400 MHz, DMSO)  $\delta$  10.06 (s, 1H), 7.70 (d,  $J$  = 8.7 Hz, 2H), 7.64 – 7.60 (m, 4H), 7.43 (t,  $J$  = 7.7 Hz, 2H), 7.31 (t,  $J$  = 7.7 Hz, 1H), 2.08 (s, 1H). **<sup>13</sup>C NMR** (100 MHz, DMSO)  $\delta$  168.4, 139.8, 139.8, 138.9, 138.9, 134.7, 128.9, 127.0, 126.9, 126.2, 119.4, 24.1. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{14}\text{H}_{13}\text{NO} + \text{H}^+$ : 212.1070, Found: 212.1069. **IR** (neat, cm<sup>-1</sup>):  $\nu$  3354, 2963, 1672, 1600, 1538, 1452, 1199, 1007, 895, 758, 685.

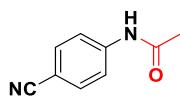




**N-(4-Aminophenyl)acetamide (5ak)**

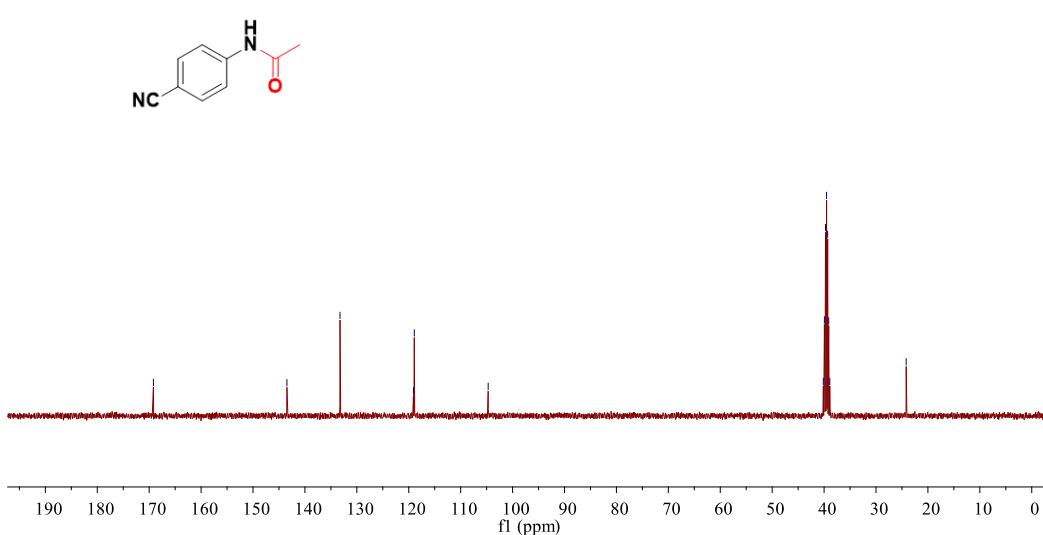
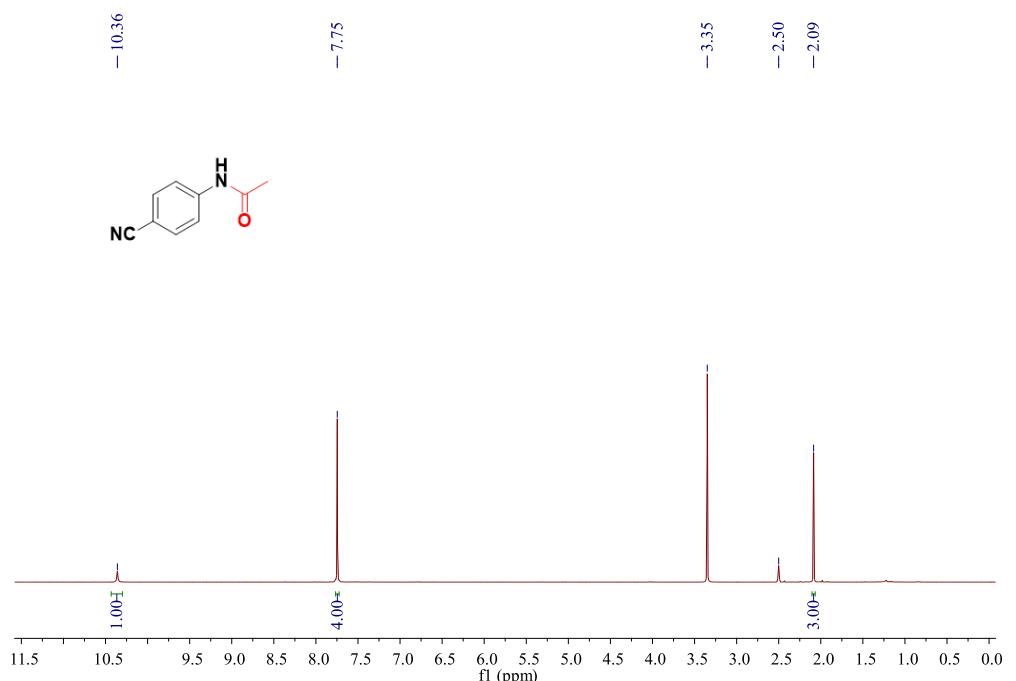
petroleum ether / ethyl acetate = 2:1, yellow solid, 64% yield (19.2 mg). mp: 126 – 128°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.47 (s, 1H), 7.18 (d, *J* = 8.7 Hz, 2H), 6.48 (d, *J* = 8.7 Hz, 2H), 4.81 (s, 2H), 1.95 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 167.3, 144.6, 128.6, 120.9, 113.8, 23.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>O+H<sup>+</sup>: 151.0866, Found: 151.0865. **IR** (neat, cm<sup>-1</sup>): ν 3357, 3305, 1641, 1553, 1429, 1264, 1024, 989, 826.

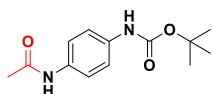




**N-(4-Cyanophenyl)acetamide (5al)**

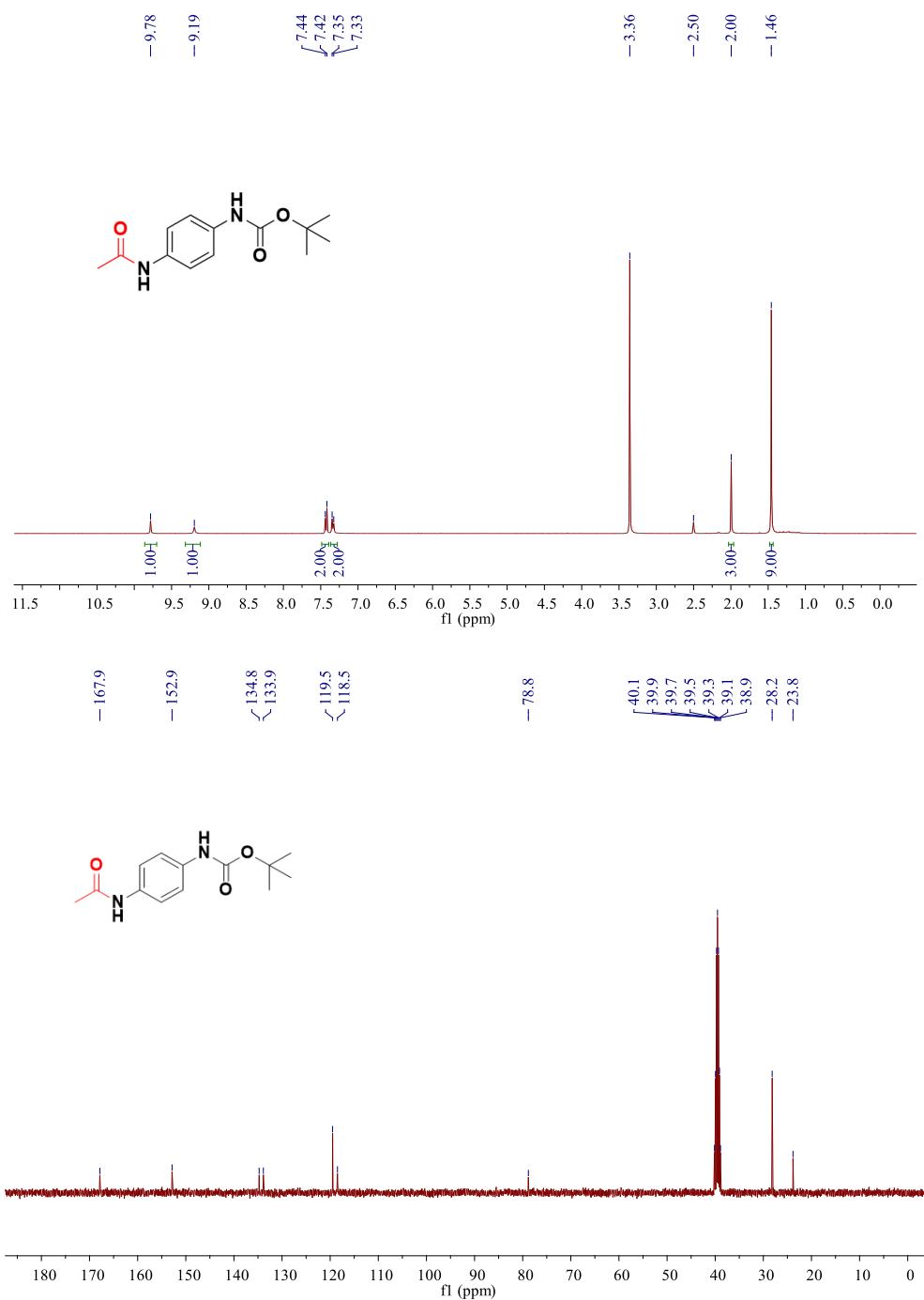
petroleum ether / ethyl acetate = 2:1, yellow solid, 73% yield (23.5 mg). mp: 200 – 203°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.36 (s, 1H), 7.75 (s, 4H), 2.09 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.2, 143.5, 133.2, 119.1, 118.9, 104.7, 24.2. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>8</sub>N<sub>2</sub>O+H<sup>+</sup>: 161.0709, Found: 161.0710. **IR** (neat, cm<sup>-1</sup>): ν 3301, 3257, 2924, 2221, 1666, 1596, 1403, 1319, 1203, 1024, 996, 818, 714, 648.

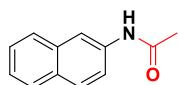




**Tert-butyl (4-acetamidophenyl)carbamate (5am)**

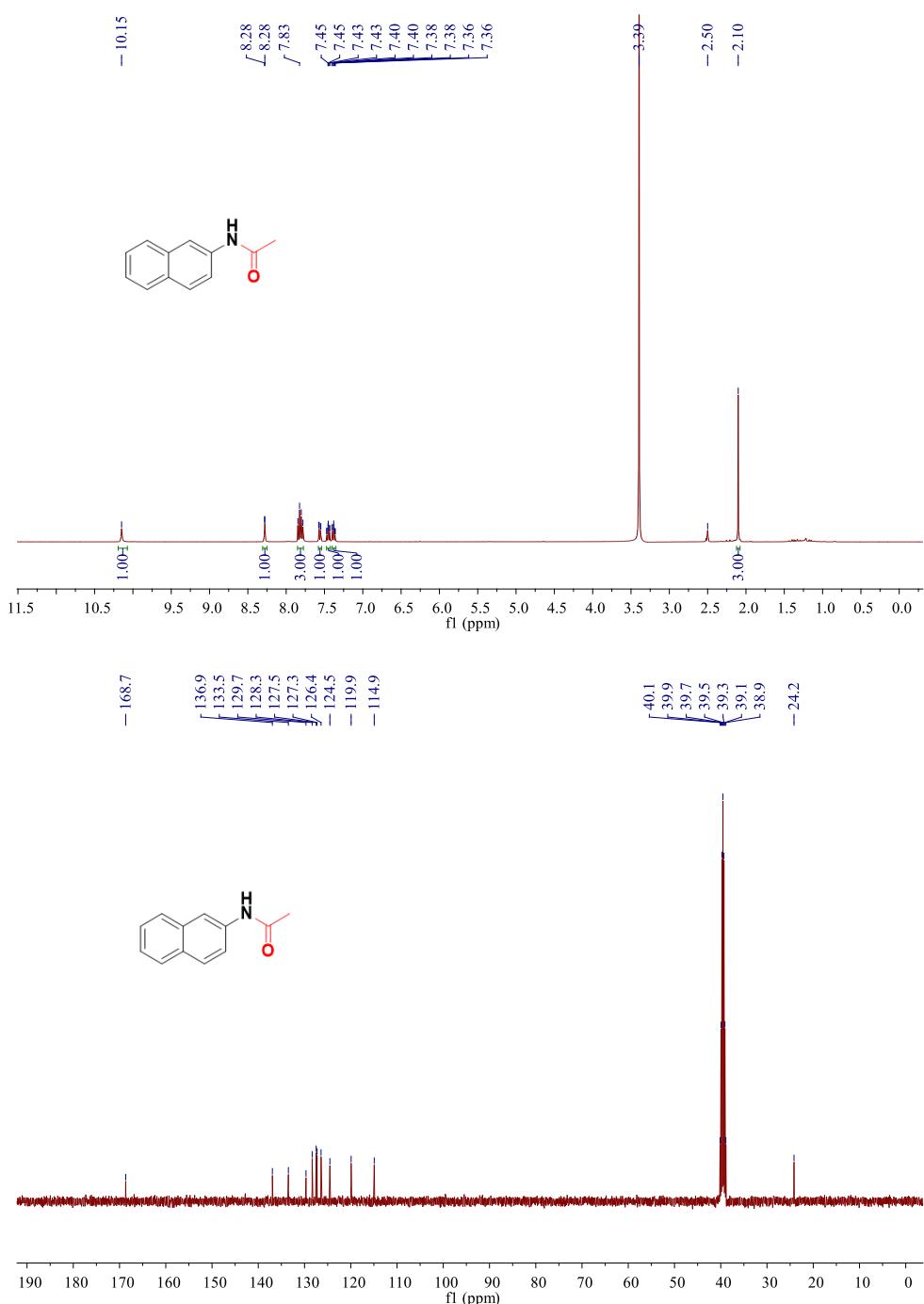
petroleum ether / ethyl acetate = 2:1, yellow solid, 45% yield (22.5 mg). mp: 161 – 163°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  9.78 (s, 1H), 9.19 (s, 1H), 7.43 (d,  $J$  = 9.0 Hz, 2H), 7.34 (d,  $J$  = 9.0 Hz, 2H), 2.00 (s, 3H), 1.46 (s, 9H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  167.9, 152.9, 134.8, 133.9, 119.5, 118.5, 78.8, 28.2, 23.8. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_3+\text{H}^+$ : 251.1390, Found: 251.1388. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3426, 2850, 1716, 1671, 1518, 1404, 1242, 1026, 909, 727, 646.

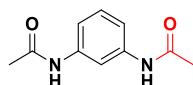




**N-(Naphthalen-2-yl)acetamide (5an)**

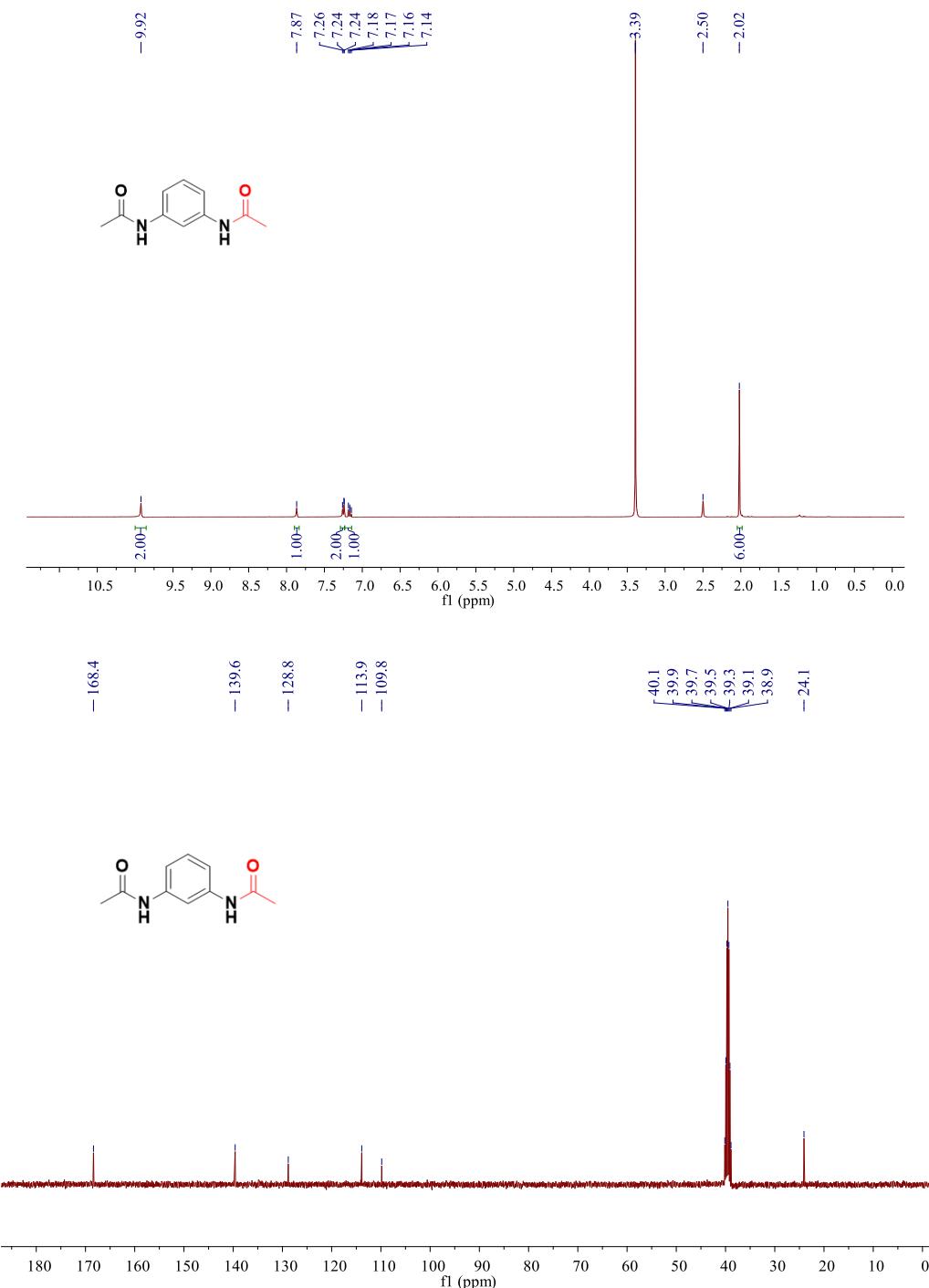
dichloromethane / ethyl acetate = 5:1, yellow solid, 90% yield (33.4 mg). mp: 134 – 136°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.15 (s, 1H), 8.28 (d, *J* = 2.0 Hz, 1H), 7.81 (dd, *J* = 17.7, 8.8 Hz, 3H), 7.56 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.47 – 7.43 (m, 1H), 7.40 – 7.36 (m, 1H), 2.10 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.7, 136.9, 133.5, 129.7, 128.3, 127.5, 127.3, 126.4, 124.5, 119.9, 114.9, 24.2. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>12</sub>H<sub>11</sub>NO+H<sup>+</sup>: 186.0913, Found: 186.0913. **IR** (neat, cm<sup>-1</sup>): ν 3405, 3293, 1670, 1527, 1489, 1393, 1258, 1049, 823, 760.

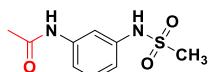




***N,N'*-(1,3-Phenylene) diacetamide (5ao)**

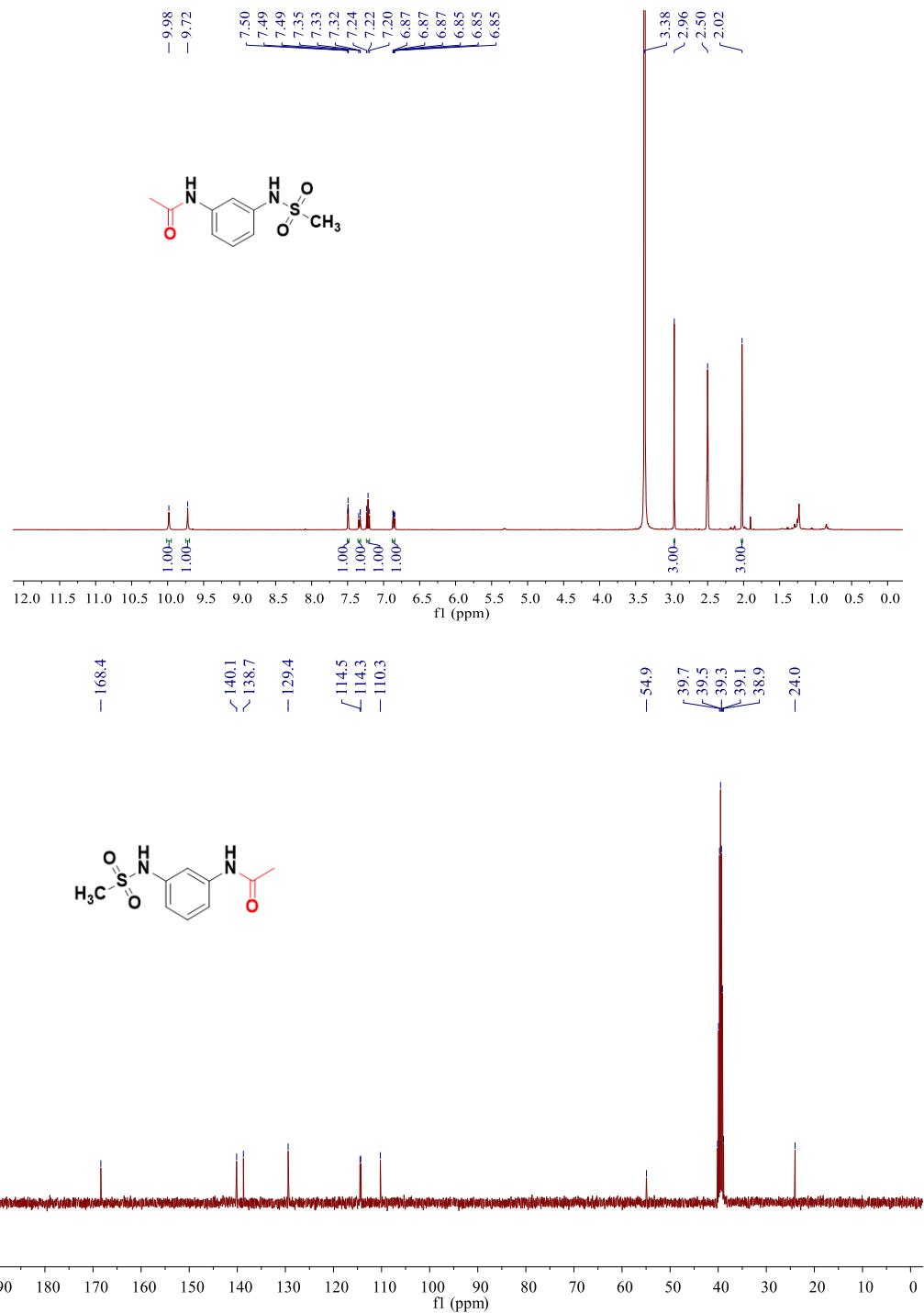
ethyl acetate, yellow solid, 66% yield (25.4 mg). mp: 180 – 182°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.92 (s, 2H), 7.87 (s, 1H), 7.26 – 7.24 (m, 2H), 7.16 (dd, *J* = 8.7, 7.2 Hz, 0H), 2.02 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.4, 139.6, 128.8, 113.9, 109.8, 24.1. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>+H<sup>+</sup>: 193.0972, Found: 193.0971. **IR** (neat, cm<sup>-1</sup>): ν 3404, 1665, 1550, 1485, 1419, 1373, 1049, 823, 761.

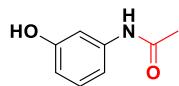




#### *N*-(3-(Methylsulfonamido)phenyl)acetamide (**5ap**)

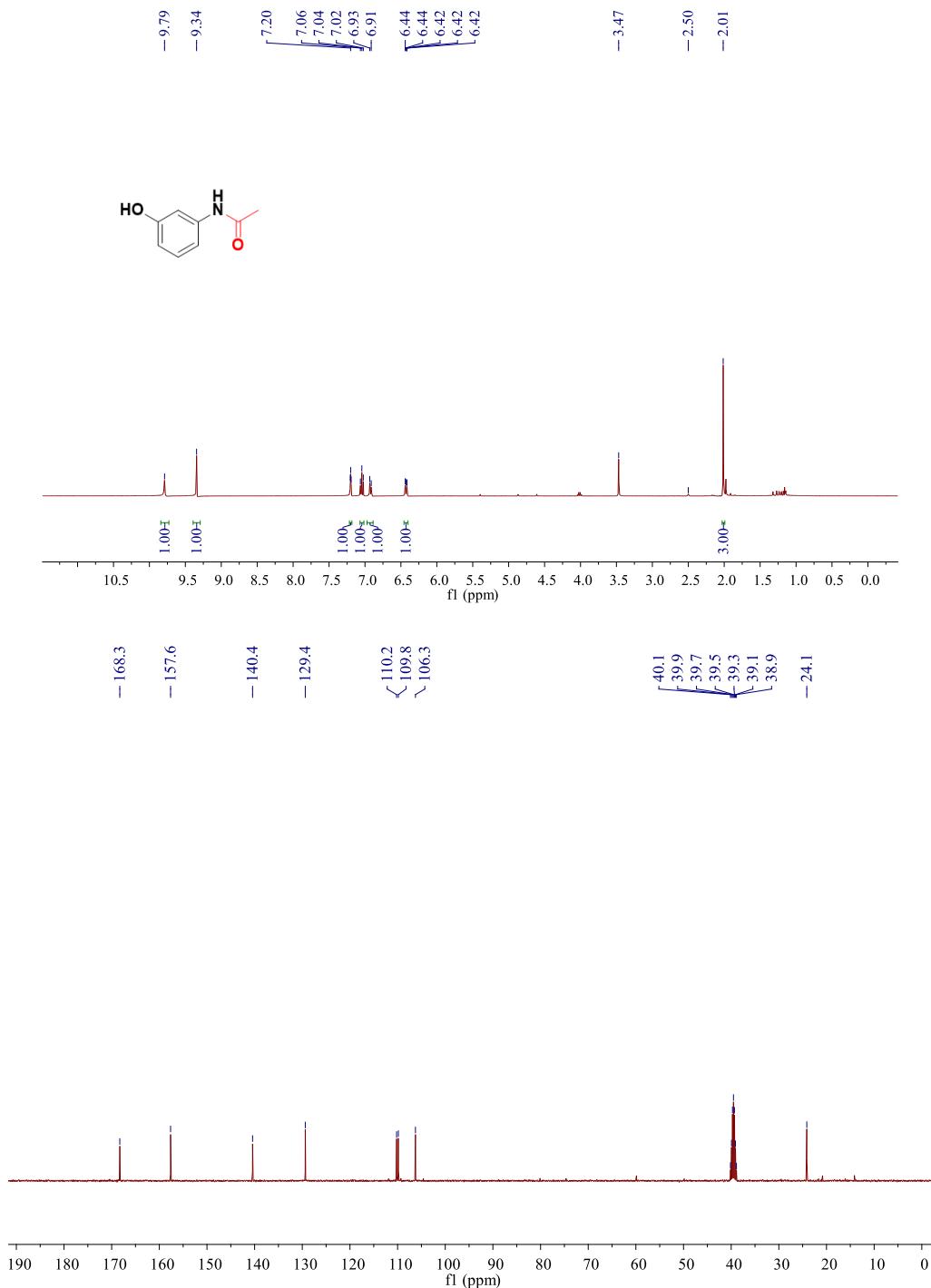
dichloromethane / ethyl acetate = 2:1, yellow oil, 74% yield (33.7 mg). **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.98 (s, 1H), 9.72 (s, 1H), 7.50 – 7.49 (m, 1H), 7.35 – 7.32 (m, 1H), 7.24 – 7.20 (m, 1H), 6.87 – 6.85 (m, 1H), 2.96 (s, 3H), 2.02 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.4, 140.1, 138.7, 129.4, 114.5, 114.3, 110.3, 54.9, 24.0. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>S+H<sup>+</sup>: 229.0641, Found: 229.0640. **IR** (neat, cm<sup>-1</sup>): ν 3398, 3274, 2240, 1674, 1608, 1542, 1471, 1301, 1200, 1053, 904, 820, 724, 648.





**N-(3-Hydroxyphenyl)acetamide (5aq)**

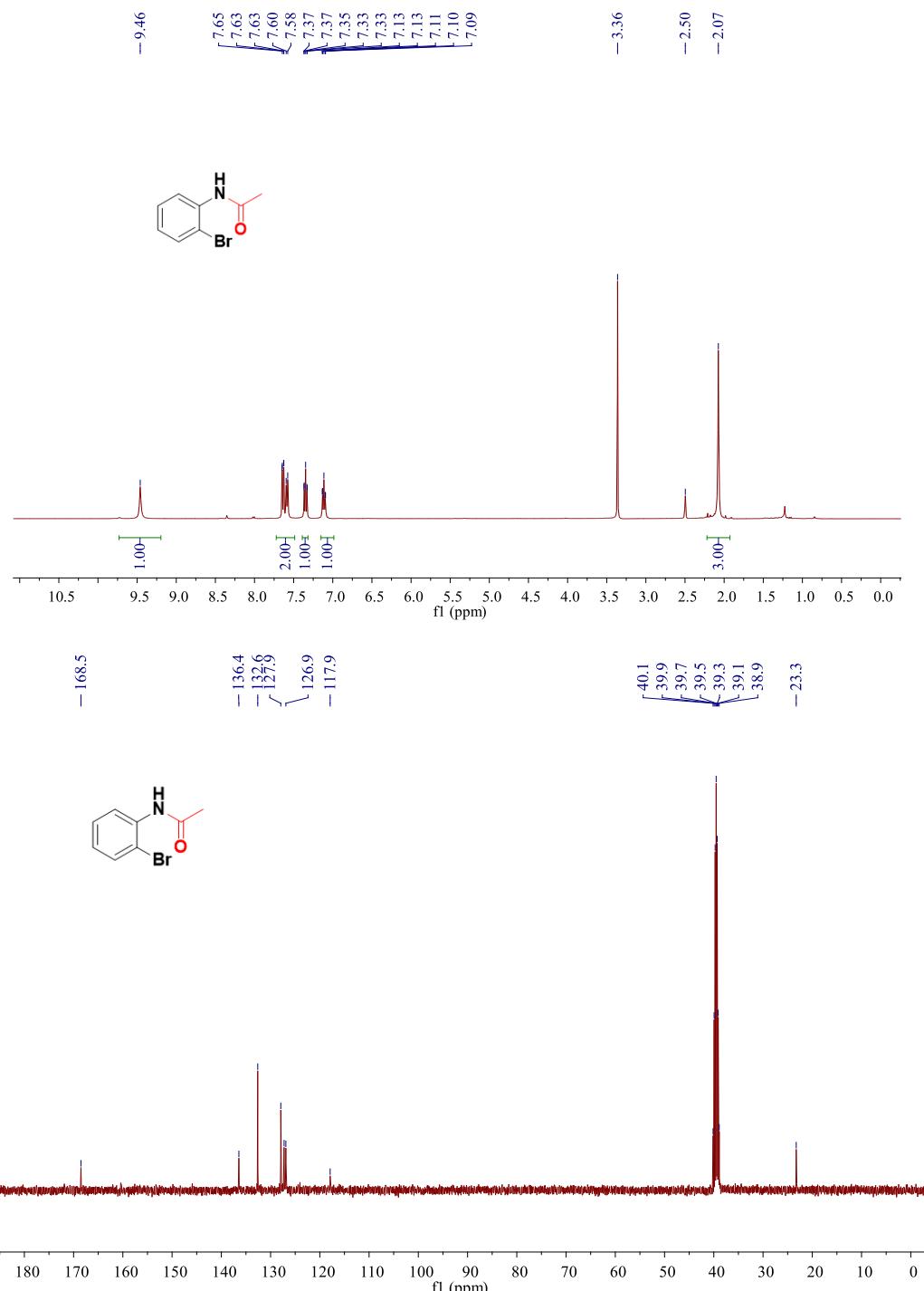
dichloromethane / ethyl acetate = 2:1, yellow solid, 53% yield (16.1 mg). mp: 150 – 153°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.79 (s, 1H), 9.34 (s, 1H), 7.20 (t, *J* = 2.0 Hz, 1H), 7.04 (t, *J* = 8.1 Hz, 1H), 6.92 (d, *J* = 8.1 Hz, 1H), 6.44 – 6.42 (m, 1H), 2.01 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.3, 157.6, 140.4, 129.4, 110.2, 109.8, 106.3, 24.1. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>9</sub>NO<sub>2</sub>+H<sup>+</sup>: 152.0706, Found: 152.0706. **IR** (neat, cm<sup>-1</sup>): ν 3299, 3261, 2926, 1661, 1513, 1453, 1371, 908, 816, 729, 647.

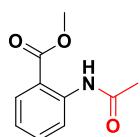




**N-(2-Bromophenyl)acetamide (5ar)**

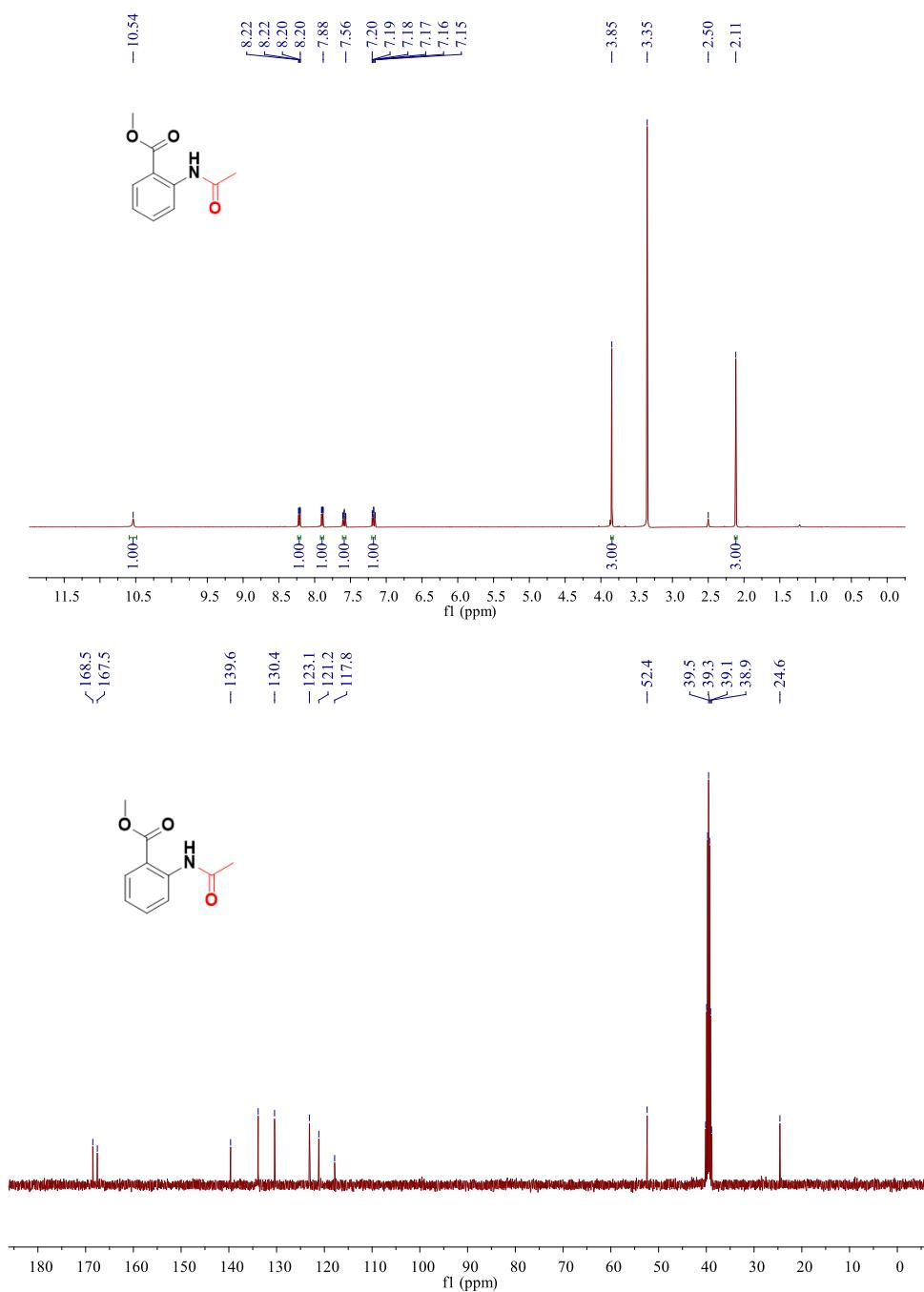
dichloromethane, yellow solid, 64% yield (27.3 mg). mp: 90 – 92°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.46 (s, 1H), 7.67 – 7.58 (m, 2H), 7.27 – 7.33 (m, 1H), 7.13 – 7.09 (m, 1H), 2.07 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.5, 136.4, 132.6, 127.9, 127.3, 126.9, 117.9, 23.3. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>8</sub><sup>79</sup>BrNO+H<sup>+</sup>: 213.9862, Found: 213.9861; C<sub>8</sub>H<sub>8</sub><sup>81</sup>BrNO+H<sup>+</sup>: 215.9842, Found: 215.9841. **IR** (neat, cm<sup>-1</sup>): ν 3410, 1660, 1510, 1430, 1207, 1005, 785, 690.

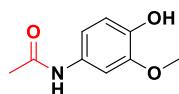




**Methyl 2-acetamidobenzoate (5as)**

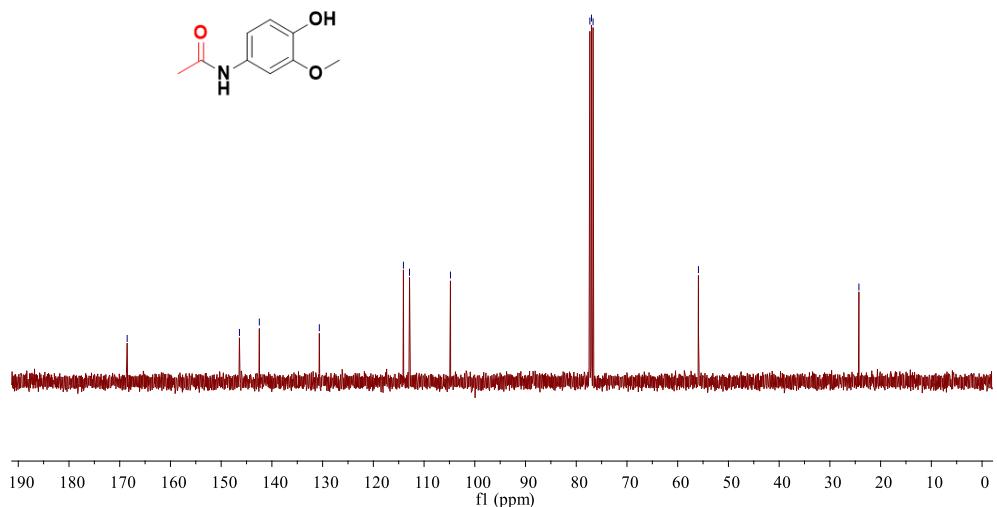
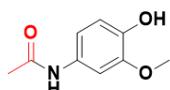
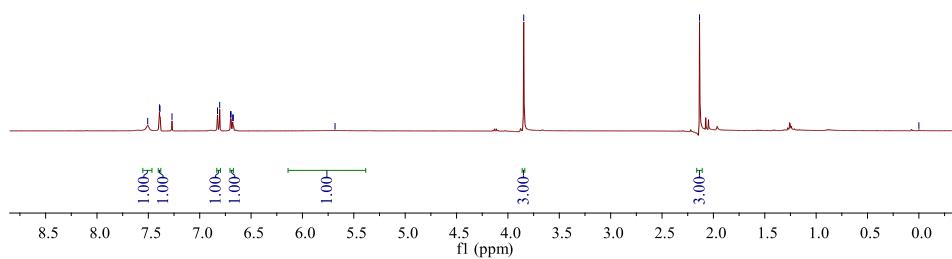
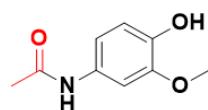
petroleum ether / ethyl acetate = 10:1, yellow solid, 60% yield (23.0 mg). mp: 98 – 100°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.54 (s, 1H), 8.22 – 8.20 (m, 1H), 7.90 – 7.88 (m, 1H), 7.61 – 7.56 (m, 1H), 7.20 – 7.15 (m, 1H), 3.85 (s, 3H), 2.11 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.5, 167.5, 139.6, 133.9, 130.4, 123.1, 121.2, 117.8, 52.4, 24.6. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>10</sub>H<sub>11</sub>NO<sub>3</sub>+H<sup>+</sup>: 194.0812, Found: 194.0812. **IR** (neat, cm<sup>-1</sup>): ν 3385, 2955, 1684, 1588, 1524, 1368, 1296, 993, 824, 760.

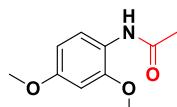




**N-(4-Hydroxy-3-methoxyphenyl)acetamide (5at)**

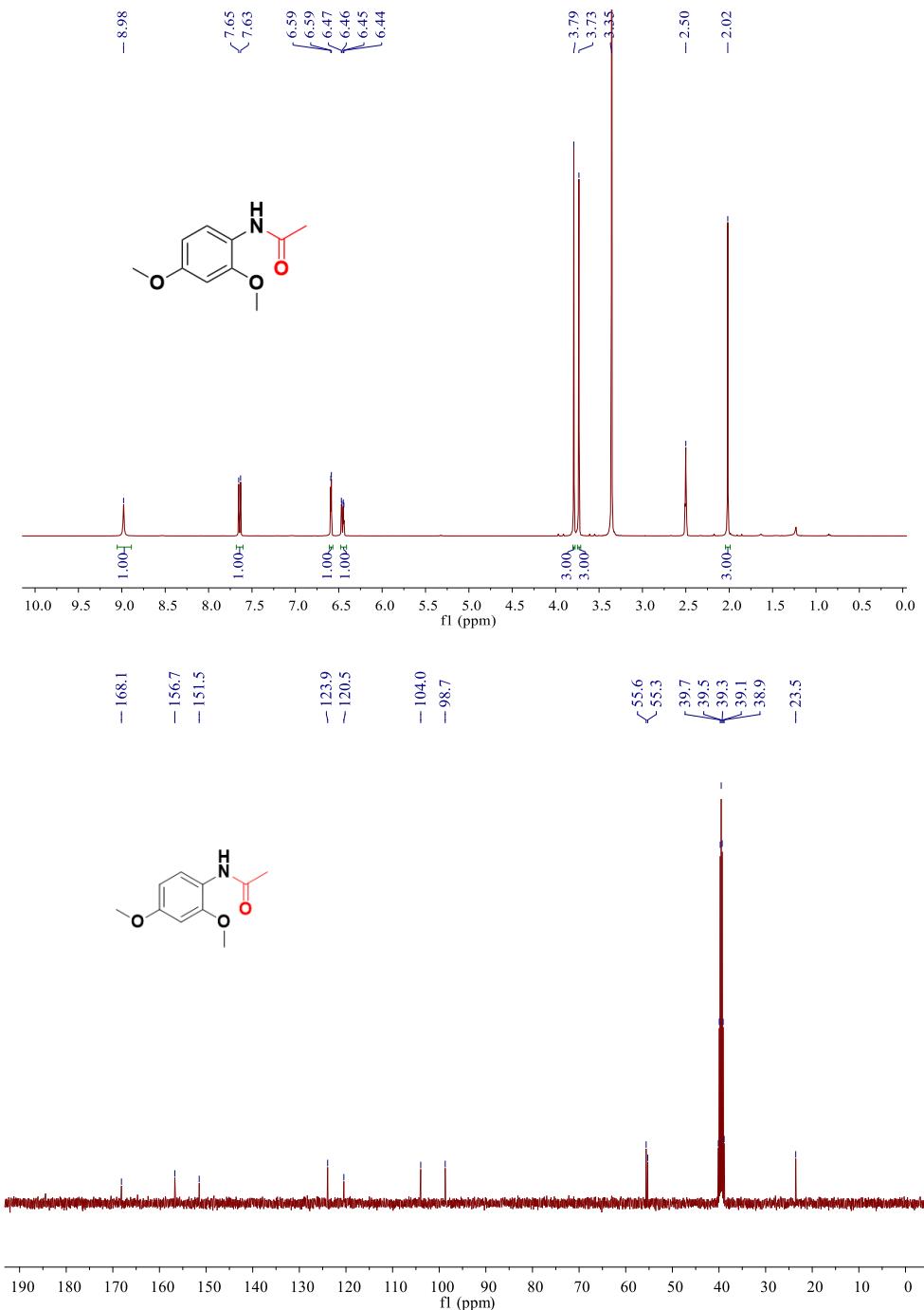
petroleum ether / ethyl acetate = 1:1, yellow solid, 48% yield (17.2 mg). mp: 110 – 112°C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.51 (s, 1H), 7.39 (d, *J* = 2.2 Hz, 1H), 6.82 (d, *J* = 8.5 Hz, 1H), 6.69 (dd, *J* = 8.5, 2.2 Hz, 1H), 5.68 (s, 1H), 3.85 (s, 3H), 2.14 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 168.5, 146.4, 142.5, 130.6, 114.1, 112.9, 104.8, 55.9, 24.3. HRMS (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>11</sub>NO<sub>3</sub>+H<sup>+</sup>: 182.0812, Found: 182.0810. IR (neat, cm<sup>-1</sup>): ν 3298, 3261, 2926, 1661, 1513, 1453, 1371, 1200, 907, 816, 729, 647.

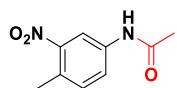




**N-(2,4-Dimethoxyphenyl)acetamide (5au)**

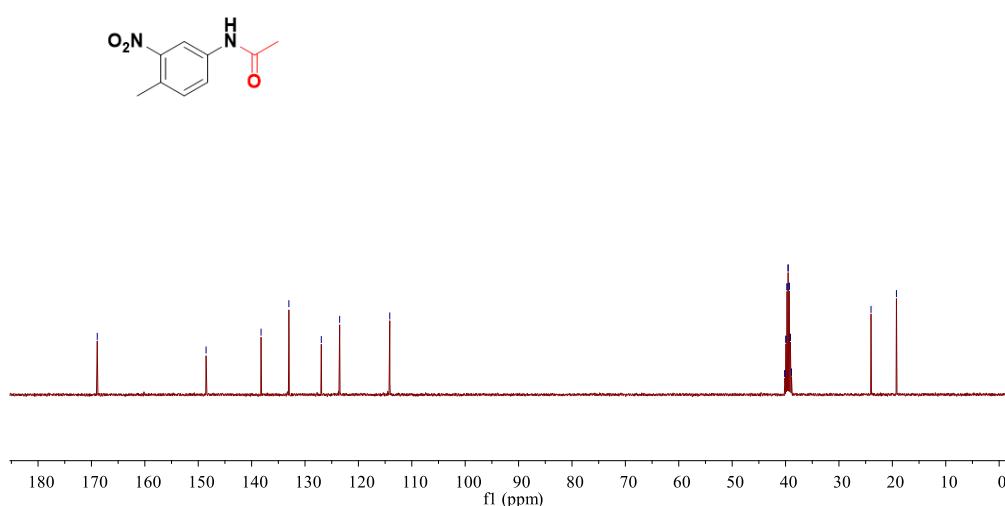
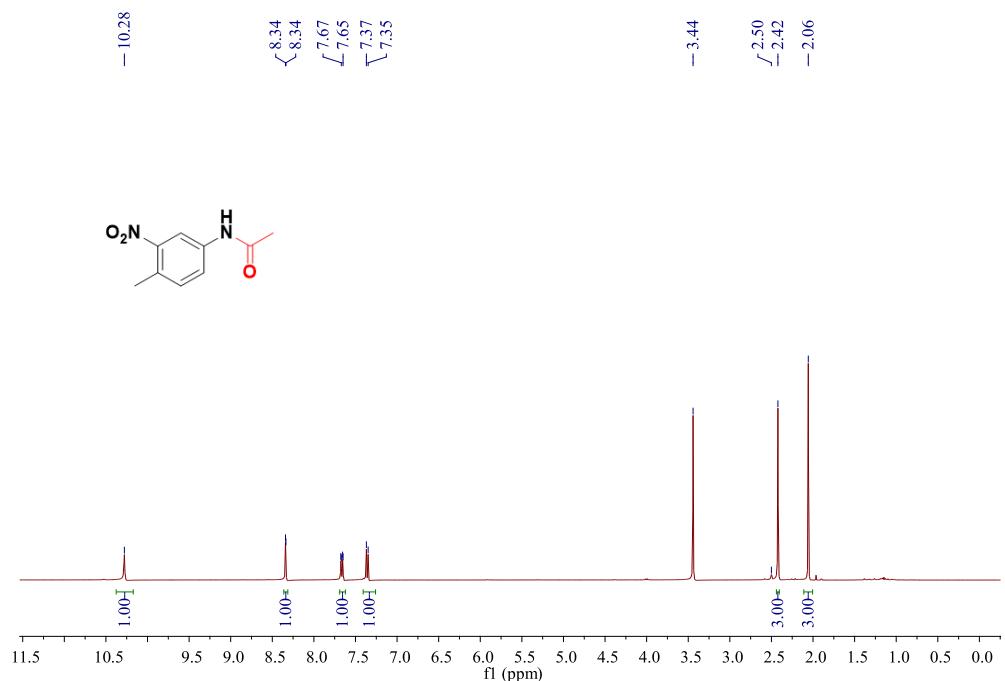
dichloromethane / ethyl acetate = 5:1, yellow solid, 80% yield (31.2 mg). mp: 113 – 115°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 8.98 (s, 1H), 7.64 (d, *J* = 8.7 Hz, 1H), 6.59 (d, *J* = 2.6 Hz, 1H), 6.46 (dd, *J* = 8.7, 2.6 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 2.02 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 168.2, 156.7, 151.5, 123.9, 120.5, 104.0, 98.7, 55.6, 55.3, 23.5. **HRMS** (ESI-TOF): Anal. Calcd. For. C<sub>10</sub>H<sub>13</sub>NO<sub>3</sub>+H<sup>+</sup>: 196.0968, Found: 196.0968. **IR** (neat, cm<sup>-1</sup>): ν 3421, 1731, 1670, 1528, 1455, 137, 1247, 1049, 822, 760.

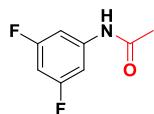




**N-(4-Methyl-3-nitrophenyl)acetamide (5av)**

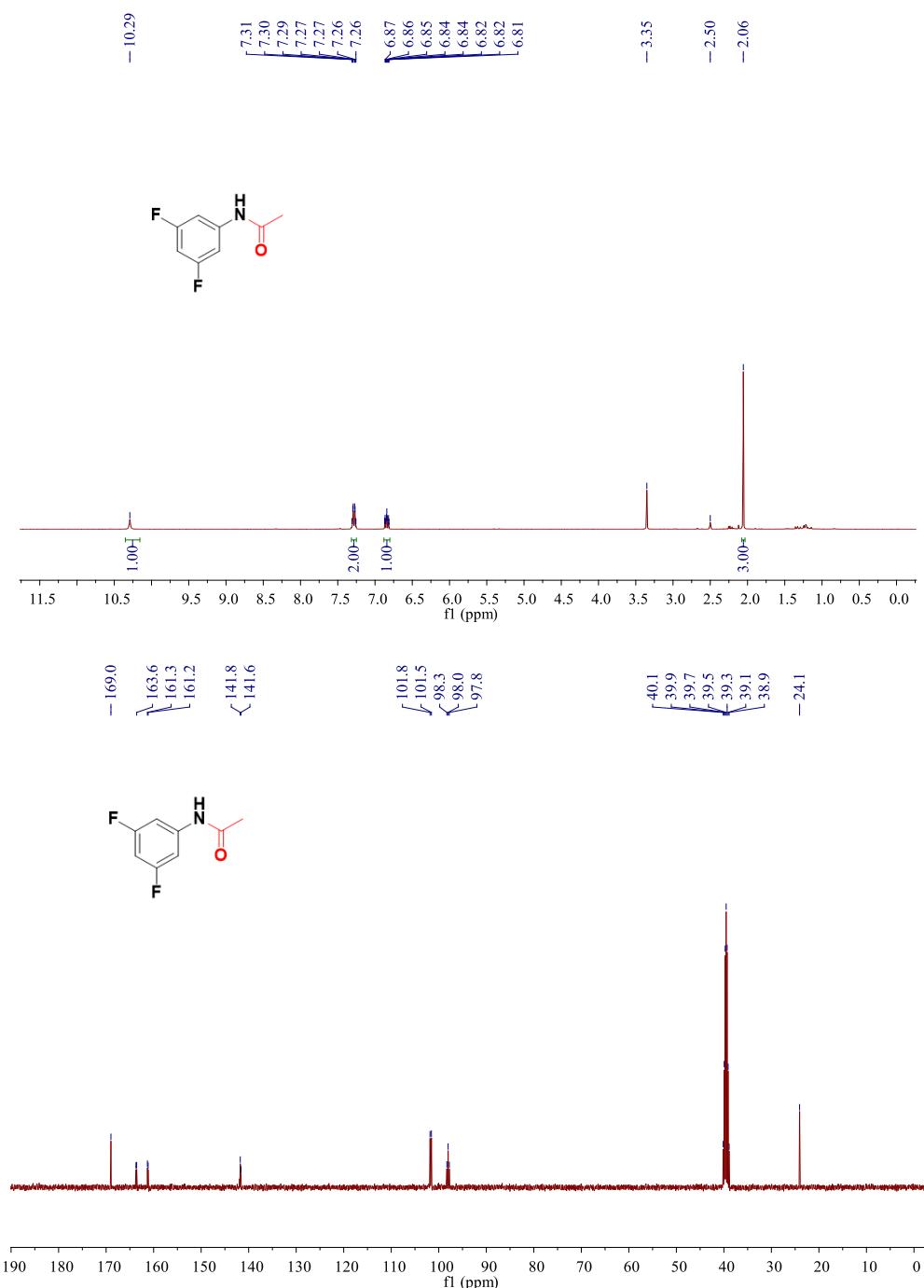
dichloromethane / ethyl acetate = 5:1, yellow solid, 78% yield (30.3 mg). mp: 141 – 143°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  10.28 (s, 1H), 8.34 (d,  $J$  = 2.2 Hz, 1H), 7.66 (dd,  $J$  = 8.4, 2.2 Hz, 1H), 7.36 (d,  $J$  = 8.4 Hz, 1H), 2.42 (s, 3H), 2.06 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  168.9, 148.5, 138.2, 133.0, 126.9, 123.5, 114.1, 24.0, 19.2. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_3+\text{H}^+$ : 195.0764, Found: 195.0764. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3353, 1672, 1537, 1489, 1392, 1198, 990, 894, 758, 684.

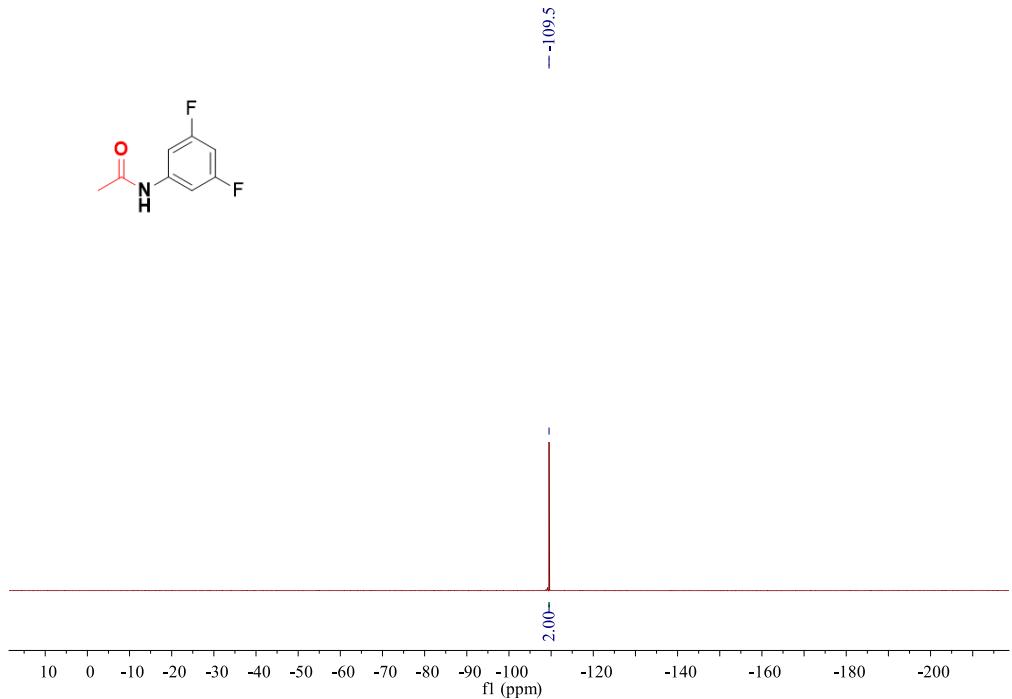


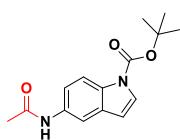


**N-(3,5-Difluorophenyl)acetamide (5aw)**

dichloromethane / ethyl acetate = 7:1, yellow solid, 97% yield (33.2 mg). mp: 125 – 127°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 10.29 (s, 1H), 7.31 – 7.26 (m, 2H), 6.87 – 6.81 (m, 1H), 2.06 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.0, 162.4 (dd, *J* = 242.8, 15.3 Hz), 141.7 (t, *J* = 14.0 Hz), 101.7 (d, *J* = 29.2 Hz), 98.0 (t, *J* = 26.2 Hz), 24.1. **<sup>19</sup>F NMR** (377 MHz, DMSO) δ -109.5 (s, 2F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>8</sub>H<sub>7</sub>F<sub>2</sub>NO+H<sup>+</sup>: 172.0568, Found: 172.0568. **IR** (neat, cm<sup>-1</sup>): ν 3450, 1700, 1653, 1500, 1051, 655.

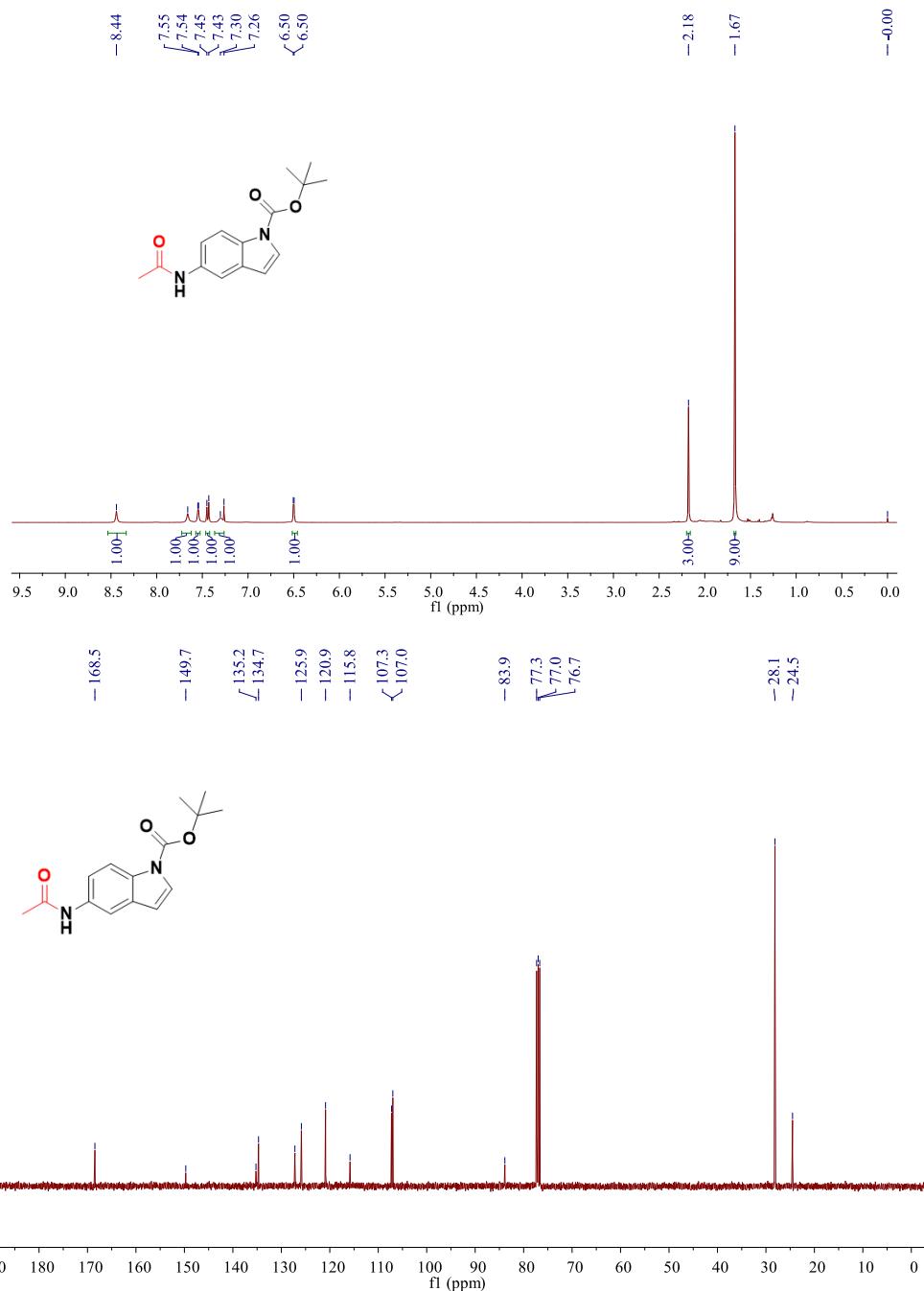






**Tert-butyl 5-acetamido-1H-indole-1-carboxylate (5ax)**

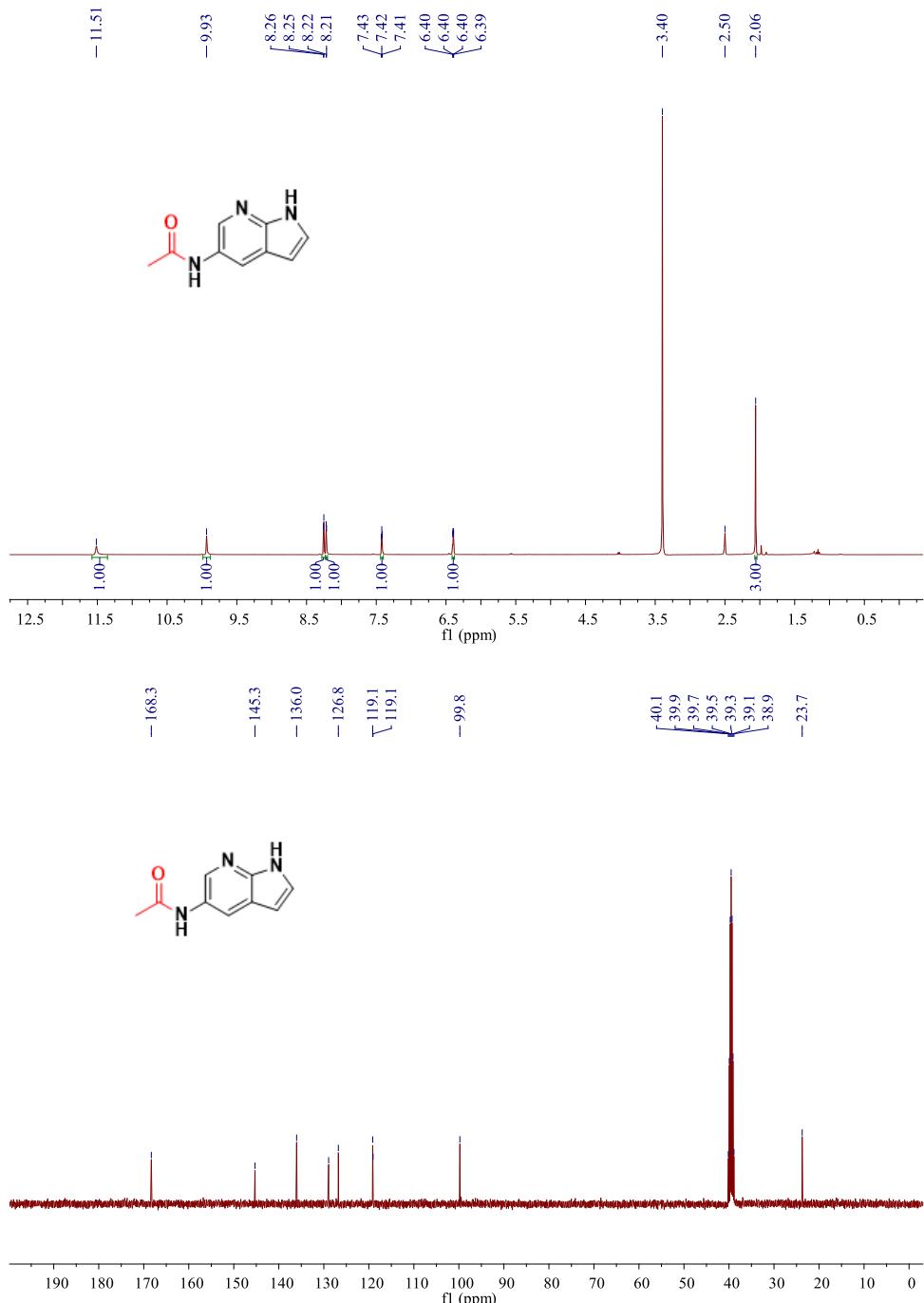
petroleum ether / ethyl acetate = 5:1, yellow oil, 57% yield (31.3 mg).  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.44 (s, 1H), 7.66 (s, 1H), 7.54 (d,  $J$  = 3.5 Hz, 1H), 7.44 (d,  $J$  = 8.4 Hz, 1H), 7.30 (s, 1H), 6.50 (d,  $J$  = 3.5 Hz, 1H), 2.18 (s, 3H), 1.67 (s, 9H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  168.5, 149.7, 135.2, 134.7, 127.2, 125.9, 120.9, 115.8, 107.3, 107.0, 83.9, 28.1, 24.5. **HRMS (ESI-TOF)**: Anal Calcd. For.  $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_3+\text{H}^+$ : 275.1390, Found: 275.1388. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3300, 2933, 1731, 1662, 1524, 1433, 1214, 1024, 906, 726, 647.

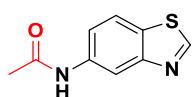




**N-(1H-pyrrolo[2,3-b]pyridin-5-yl)acetamide (5ay)**

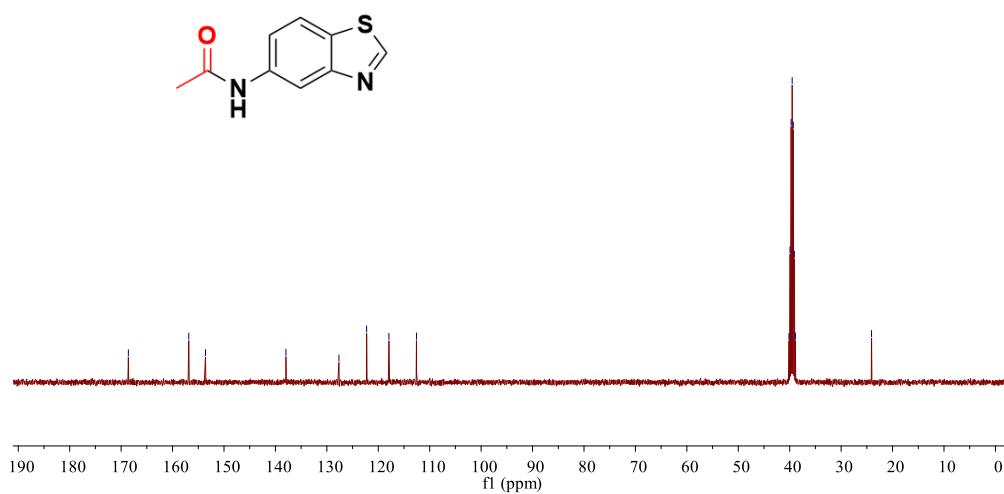
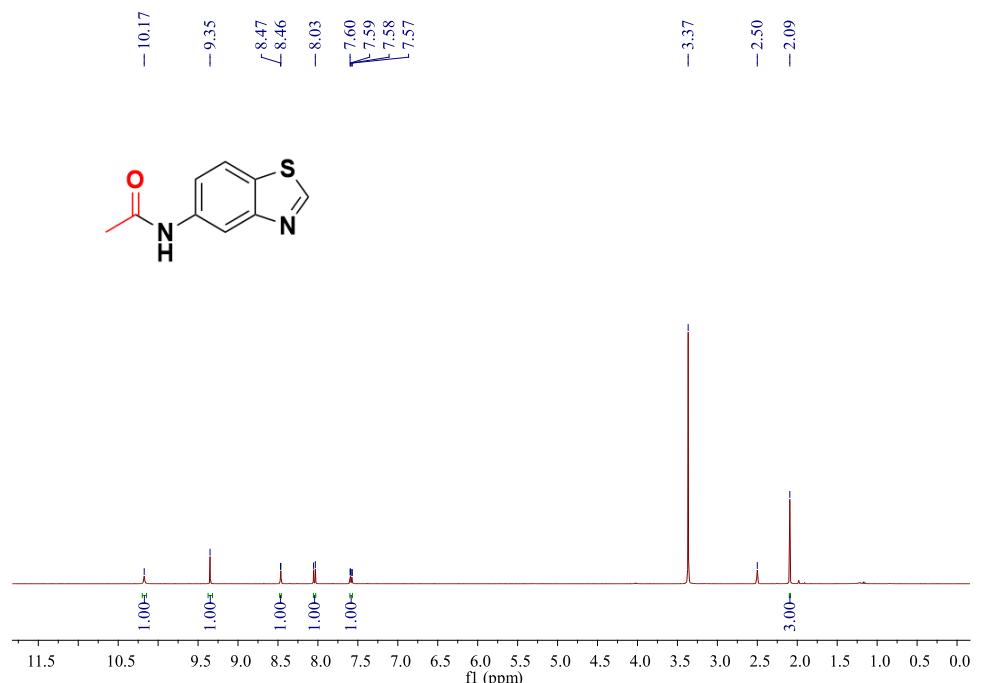
petroleum ether / ethyl acetate = 1:1, yellow oil, 55% yield (19.2 mg).  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  11.51 (s, 1H), 9.93 (s, 1H), 8.25 (d,  $J$  = 2.3 Hz, 1H), 8.22 (d,  $J$  = 2.3 Hz, 1H), 7.43 – 7.31 (m, 1H), 6.40 (dd,  $J$  = 3.4, 1.9 Hz, 1H), 2.06 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  168.3, 145.3, 136.0, 126.8, 119.1, 119.1, 99.8, 23.7. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_9\text{N}_3\text{O}+\text{H}^+$ : 176.0818, Found: 176.0817. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3415, 2922, 1620, 1548, 1210, 1005, 745.

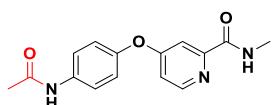




***N*-(Benzo[d]thiazol-5-yl)acetamide (5az)**

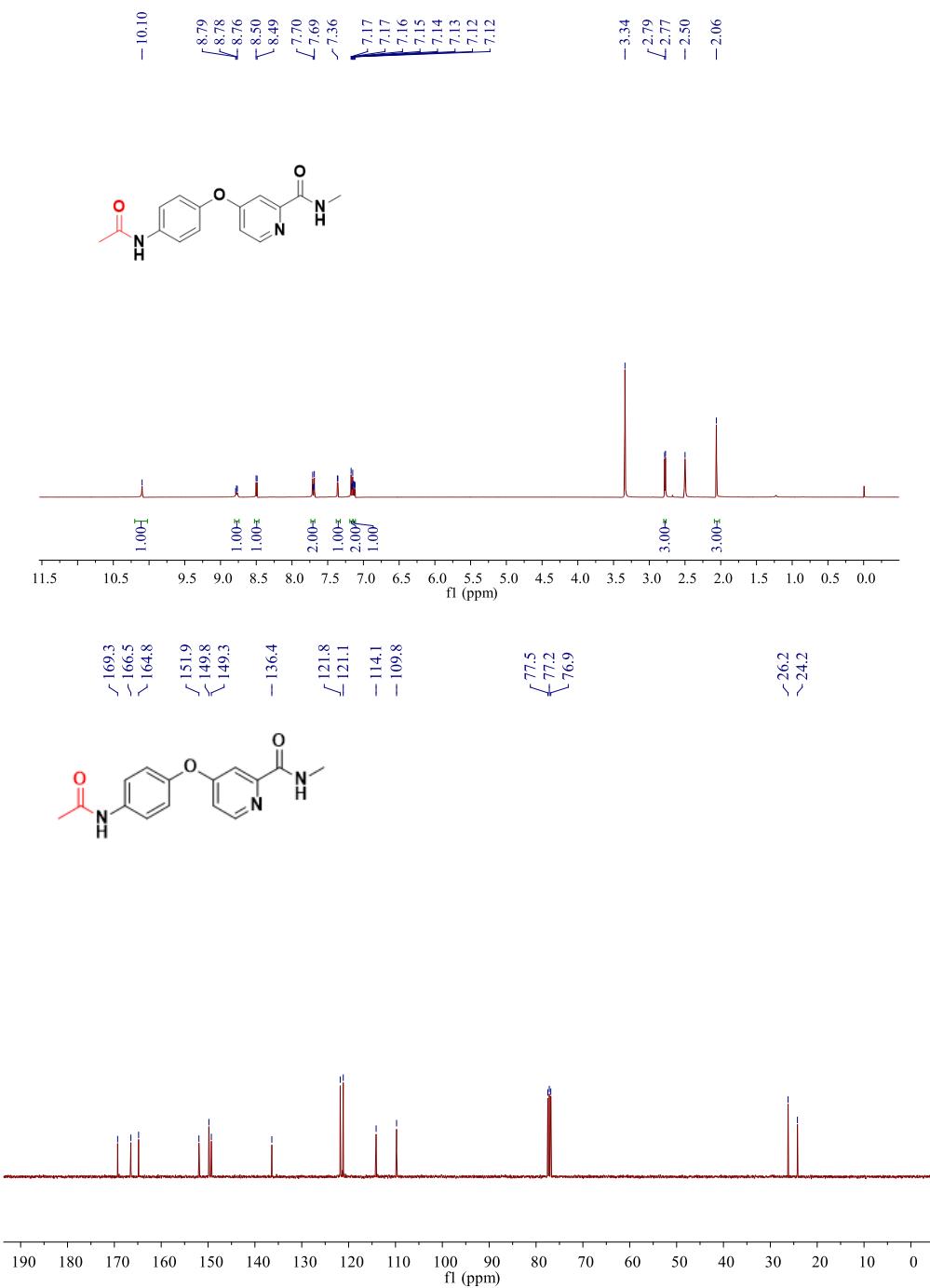
petroleum ether / ethyl acetate = 1:1, yellow solid, 63% yield (24.0 mg). mp: 185 – 187°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  10.17 (s, 1H), 9.35 (s, 1H), 8.47 (d,  $J$  = 1.9 Hz, 1H), 8.03 (d,  $J$  = 8.7 Hz, 1H), 7.58 (dd,  $J$  = 8.7, 1.9 Hz, 1H), 2.09 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  168.6, 156.8, 153.6, 152.3, 127.7, 122.3, 117.9, 112.6, 24.1. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_8\text{N}_2\text{OS}+\text{H}^+$ : 193.0430, Found: 193.0430. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3414, 1731, 1665, 1526, 1444, 1374, 1247, 1023, 823, 760.

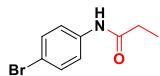




**4-(4-Acetamidobenzyl)-N-methylpicolinamide (5ba)**

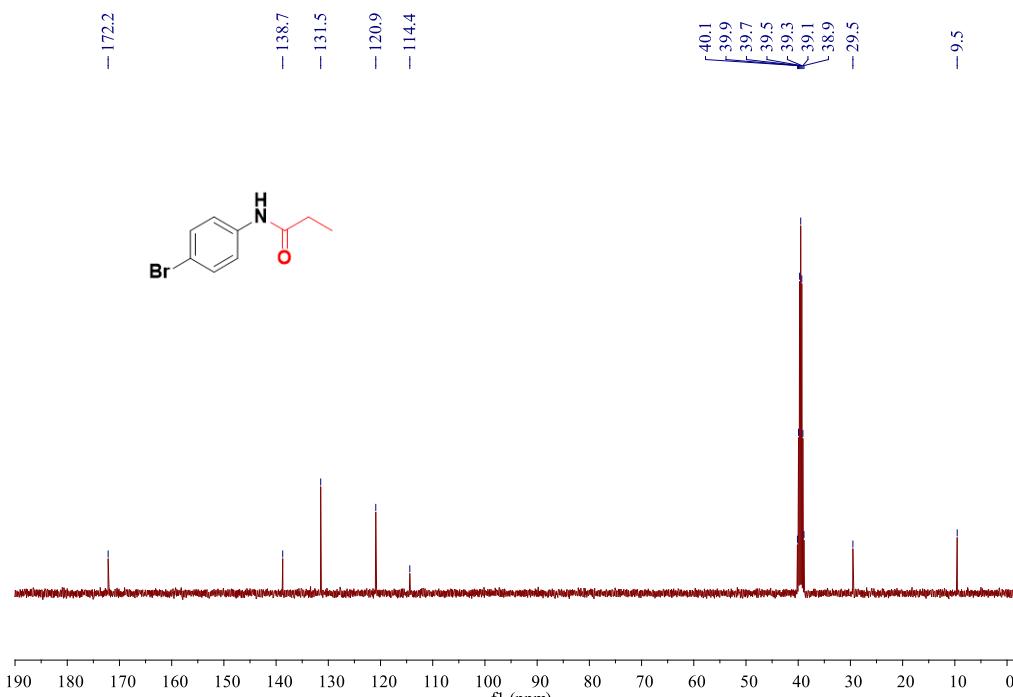
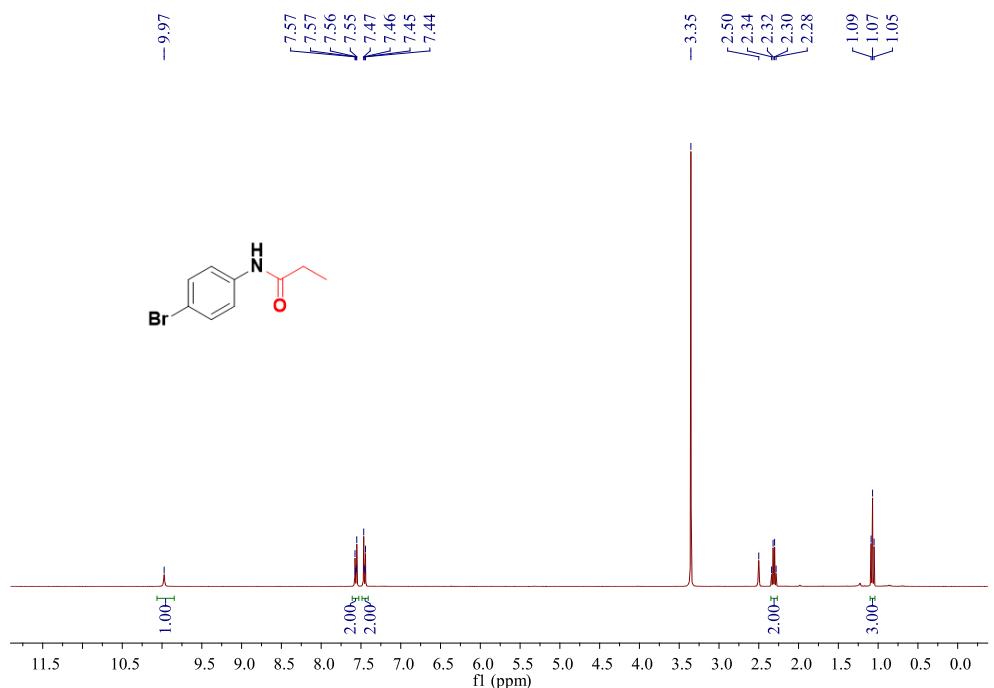
ethyl acetate, yellow oil, 64% yield (36.5 mg). <sup>1</sup>H NMR (400 MHz, DMSO) δ 10.10 (s, 1H), 8.79 – 8.76 (m, 1H), 8.50–8.49 (m, 1H), 7.70 – 7.69 (m, 2H), 7.36 (d, *J* = 0.6 Hz, 1H), 7.17 – 7.15 (m, 2H), 7.14 – 7.12 (m, 1H), 2.78 (d, *J* = 4.9 Hz, 3H), 2.06 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 169.3, 166.5, 164.8, 151.9, 149.8, 149.3, 136.4, 121.8, 121.1, 114.1, 109.8, 26.2, 24.2. HRMS (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>15</sub>N<sub>3</sub>O<sub>3</sub>+H<sup>+</sup>: 286.1186, Found: 286.1184. IR (neat, cm<sup>-1</sup>): ν 3354, 2921, 1672, 1538, 1454, 1369, 1254, 1065, 894, 758, 685.

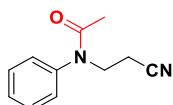




**N-(4-Bromophenyl)-N-methylpropionamide (5bb)**

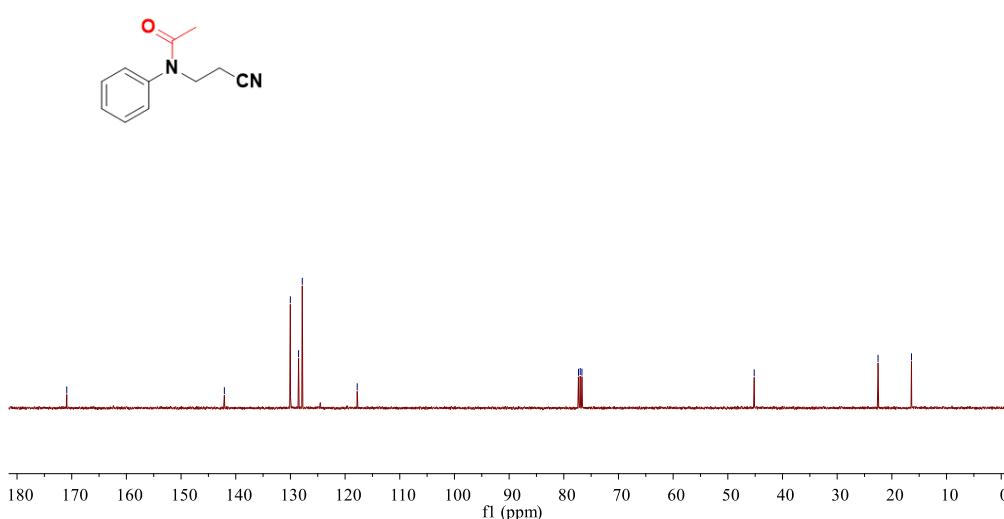
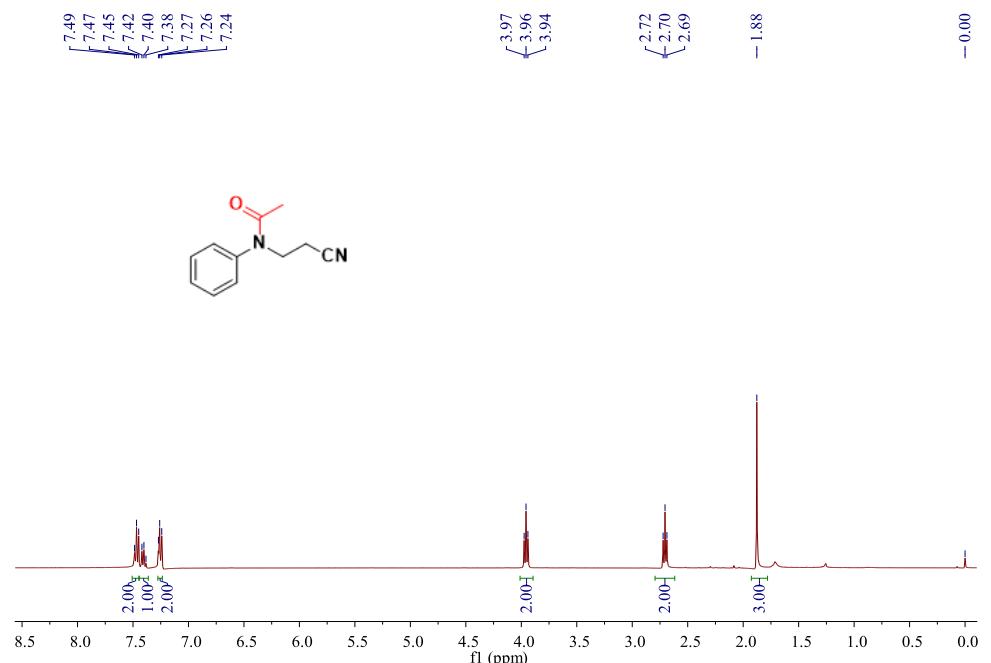
petroleum ether / ethyl acetate = 5:1, yellow liquid, 65% yield (31.5 mg). **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 9.97 (s, 1H), 7.57 – 7.55 (m, 2H), 7.47 – 7.44 (m, 2H), 2.31 (q,  $J = 7.5$  Hz, 2H), 1.07 (t,  $J = 7.5$  Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 172.2, 138.7, 131.5, 120.9, 114.4, 29.5, 9.5. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>9</sub>H<sub>10</sub><sup>79</sup>BrNO+H<sup>+</sup>: 228.0019, Found: 228.0014; C<sub>9</sub>H<sub>10</sub><sup>81</sup>BrNO+Na<sup>+</sup>: 229.9998, Found: 229.9994. **IR** (neat, cm<sup>-1</sup>): ν 3298, 2977, 1661, 1603, 1590, 1487, 921, 726, 682.

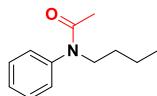




**N-(2-Cyanoethyl)-N-phenylacetamide (5bc)**

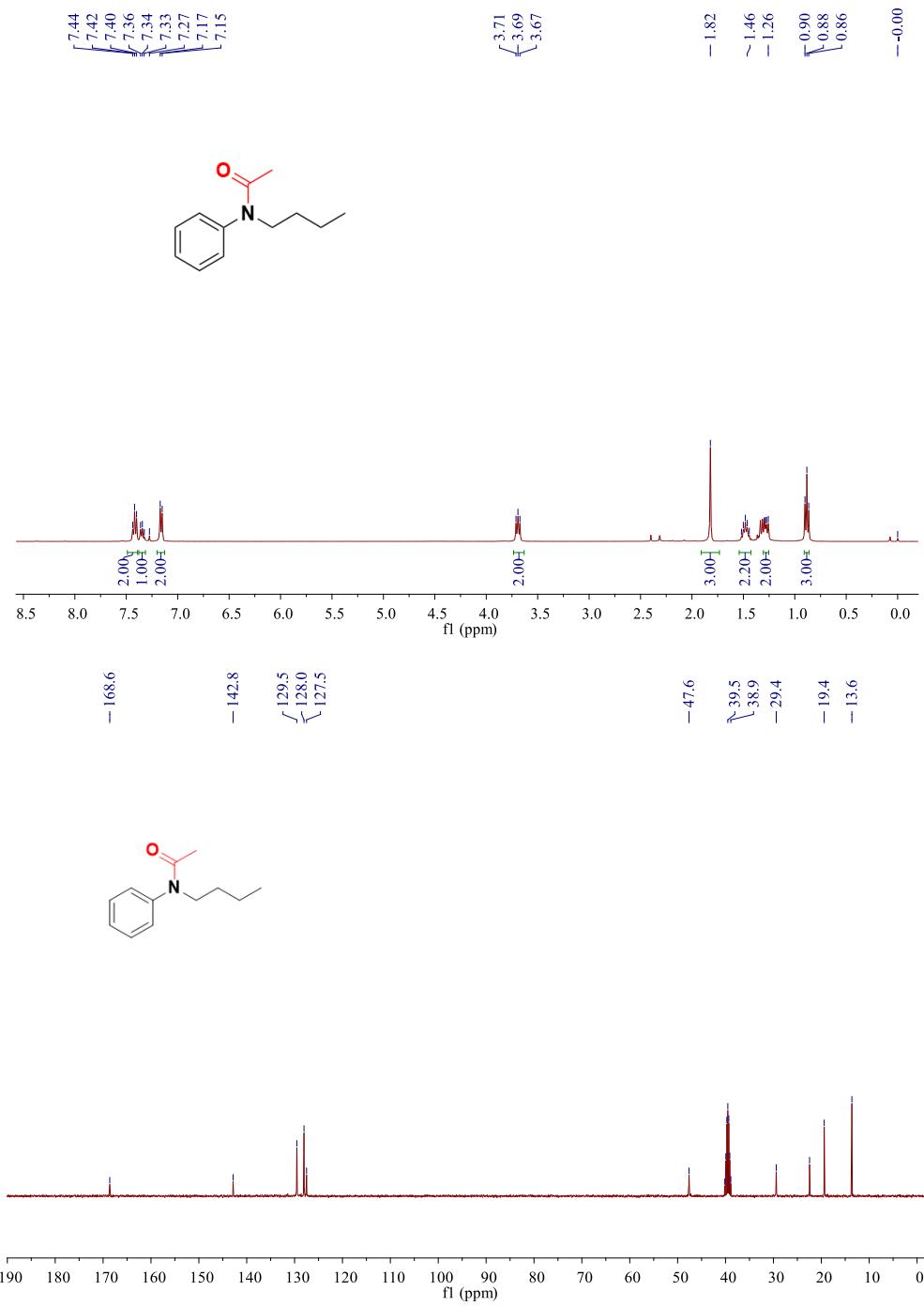
petroleum ether / ethyl acetate =2:1, light yellow oil, 45% yield (16.9 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.47 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.27 – 7.24 (m, 2H), 3.96 (t, *J* = 6.8 Hz, 2H), 2.70 (t, *J* = 6.8 Hz, 2H), 1.88 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.9, 142.1, 130.0, 128.5, 127.8, 117.8, 45.2, 22.5, 16.4. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>O+H<sup>+</sup>: 189.1022, Found: 189.1020. **IR** (neat, cm<sup>-1</sup>): ν 2934, 2251, 1655, 1596, 1494, 1395, 1202, 1025, 908, 726, 646.

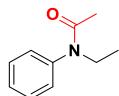




**N-Butyl-N-phenylacetamide (5bd)**

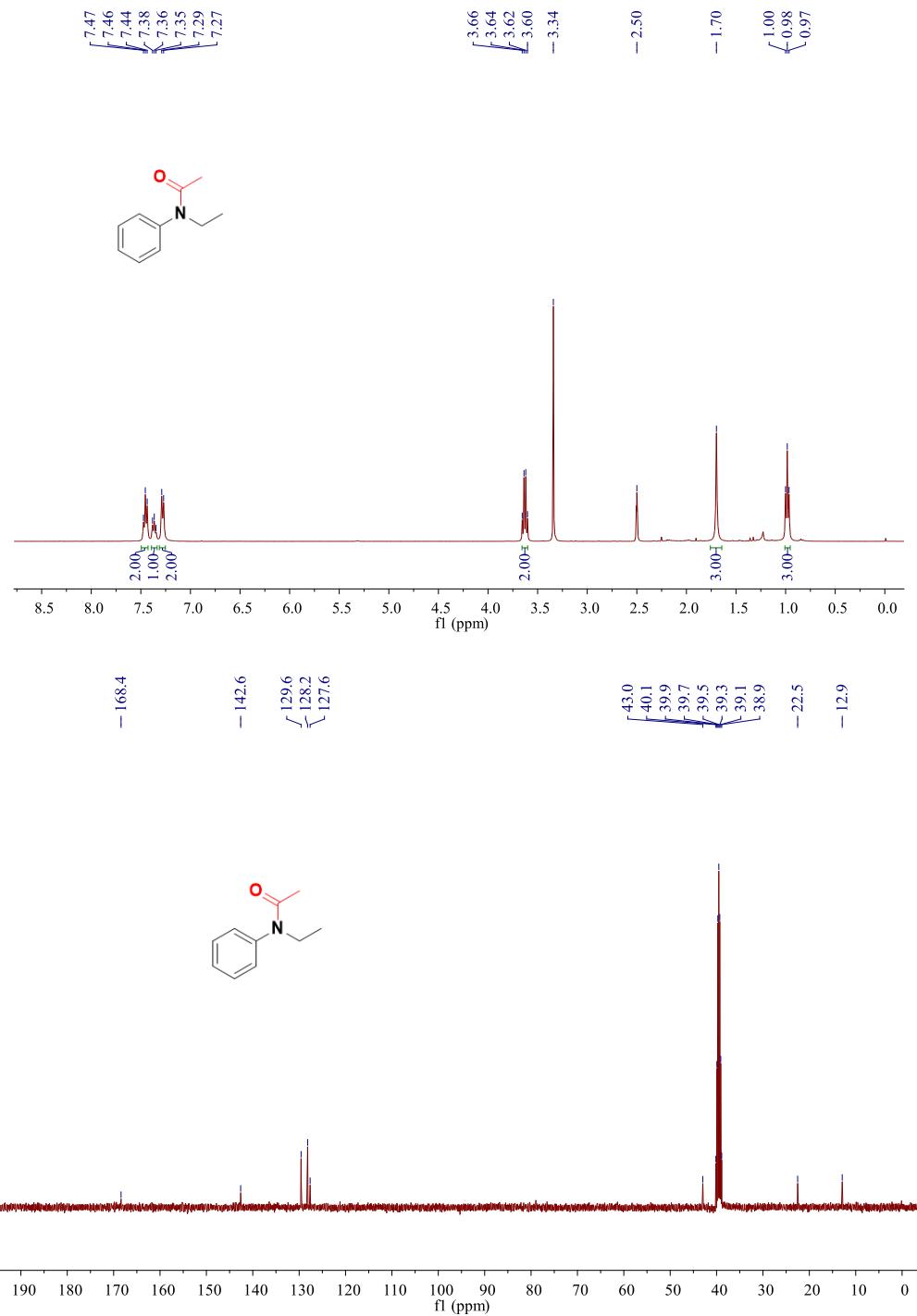
dichloromethane / ethyl acetate = 1:1, yellow oil, 70% yield (26.7 mg). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.44 – 7.40 (m, 2H), 7.36 – 7.33 (m, 1H), 7.17 – 7.15 (m, 1H), 3.71 – 3.67 (m, 2H), 1.82 (s, 3H), 1.52 – 1.44 (m, 2H), 1.29 – 1.26 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 2H). <sup>13</sup>C NMR (100 MHz, DMSO) δ 168.6, 142.8, 129.5, 128.0, 127.5, 47.6, 29.4, 22.5, 19.4, 13.6. HRMS (ESI-TOF): Anal Calcd. For. C<sub>12</sub>H<sub>17</sub>NO+H<sup>+</sup>: 192.1383, Found: 192.1380. IR (neat, cm<sup>-1</sup>): ν 2956, 1649, 1516, 1455, 1383, 1261, 1083, 908, 826, 732.

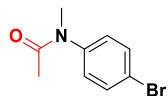




**N-Ethyl-N-phenylacetamide (5be)**

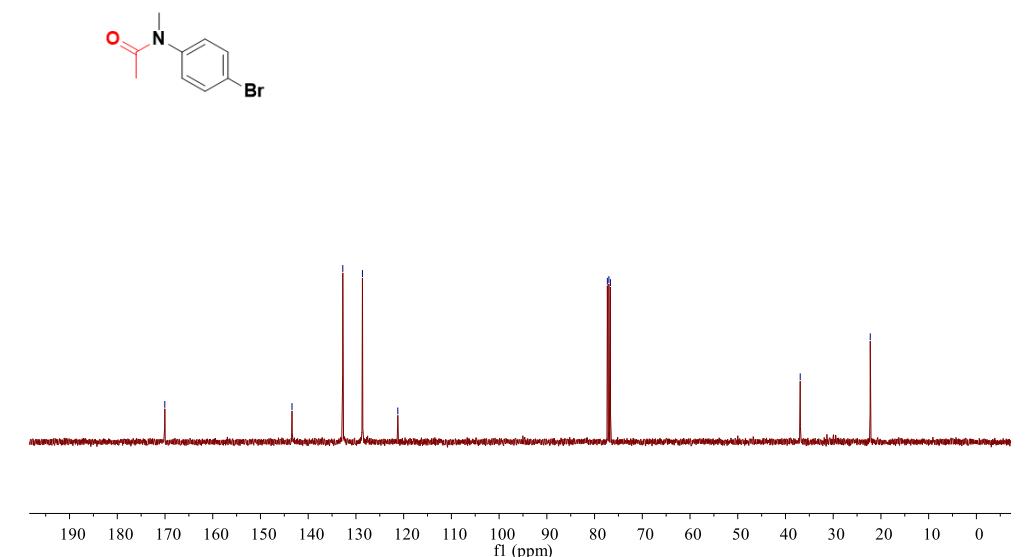
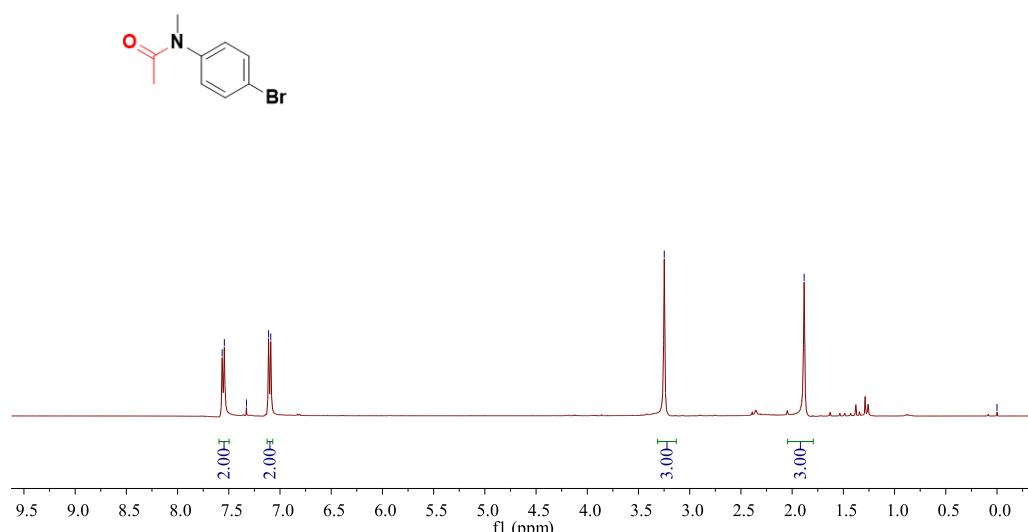
dichloromethane / ethyl acetate = 1:1, yellow solid, 73% yield (23.7 mg). mp: 48 – 50°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  7.47 – 7.44 (m, 2H), 7.38 – 7.35 (m, 1H), 7.29 – 7.27 (m, 1H), 3.63 (q,  $J$  = 7.1 Hz, 2H), 1.70 (s, 3H), 0.98 (t,  $J$  = 7.1 Hz, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  168.4, 142.61, 129.6, 128.2, 127.6, 43.0, 22.5, 12.9. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{10}\text{H}_{13}\text{NO}+\text{H}^+$ : 164.1070, Found: 164.1068. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3368, 2932, 1640, 1594, 1496, 1300, 1259, 1046, 990, 826, 765.

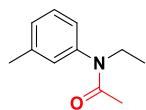




**N-(4-bromophenyl)-N-methylacetamide (5bf)**

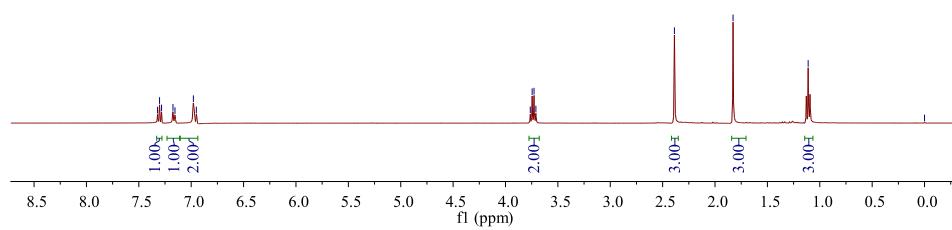
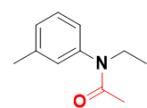
petroleum ether / ethyl acetate = 2:1, yellow solid, 80% yield (36.5 mg). mp: 95 – 97°C.  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 (d,  $J$  = 8.4 Hz, 2H), 7.10 (d,  $J$  = 8.4 Hz, 2H), 3.25 (s, 3H), 1.88 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.0, 143.4, 132.7, 128.6, 121.2, 36.9, 22.2. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_{10}^{79}\text{BrNO} + \text{H}^+$ : 228.0019, Found: 228.0014;  $\text{C}_9\text{H}_{10}^{81}\text{BrNO} + \text{H}^+$ : 229.9998, Found: 229.9994. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3395, 3060, 2932, 1649, 1587, 1484, 1371, 1179, 1084, 837, 722, 643.





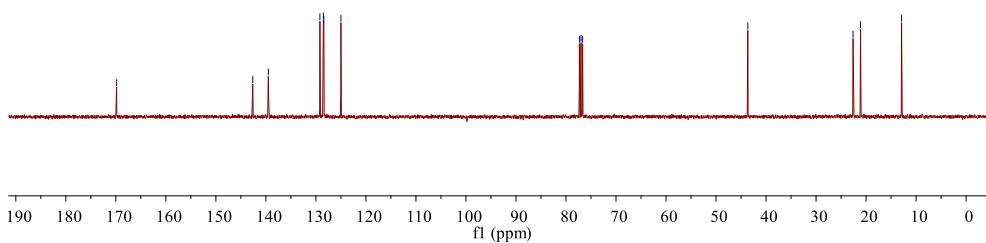
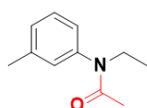
**N-Ethyl-N-(m-tolyl)acetamide (5bg)**

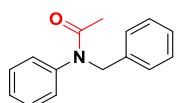
petroleum ether / ethyl acetate = 5:1, yellow oil, 70% yield (24.6 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.30 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 1H), 6.98 – 6.95 (m, 2H), 3.74 (q, *J* = 7.2 Hz, 2H), 2.39 (s, 3H), 1.83 (s, 3H), 1.11 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 169.9, 142.6, 139.5, 129.2, 128.5, 128.4, 125.0, 43.6, 22.6, 21.1, 12.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>11</sub>H<sub>15</sub>NO+H<sup>+</sup>: 178.1226, Found: 178.1223. **IR** (neat, cm<sup>-1</sup>): ν 3481, 2974, 2874, 1653, 1586, 1445, 1395, 1299, 1192, 838, 706, 626.



–169.9  
–142.6  
–139.5  
129.2  
128.5  
128.4  
125.0

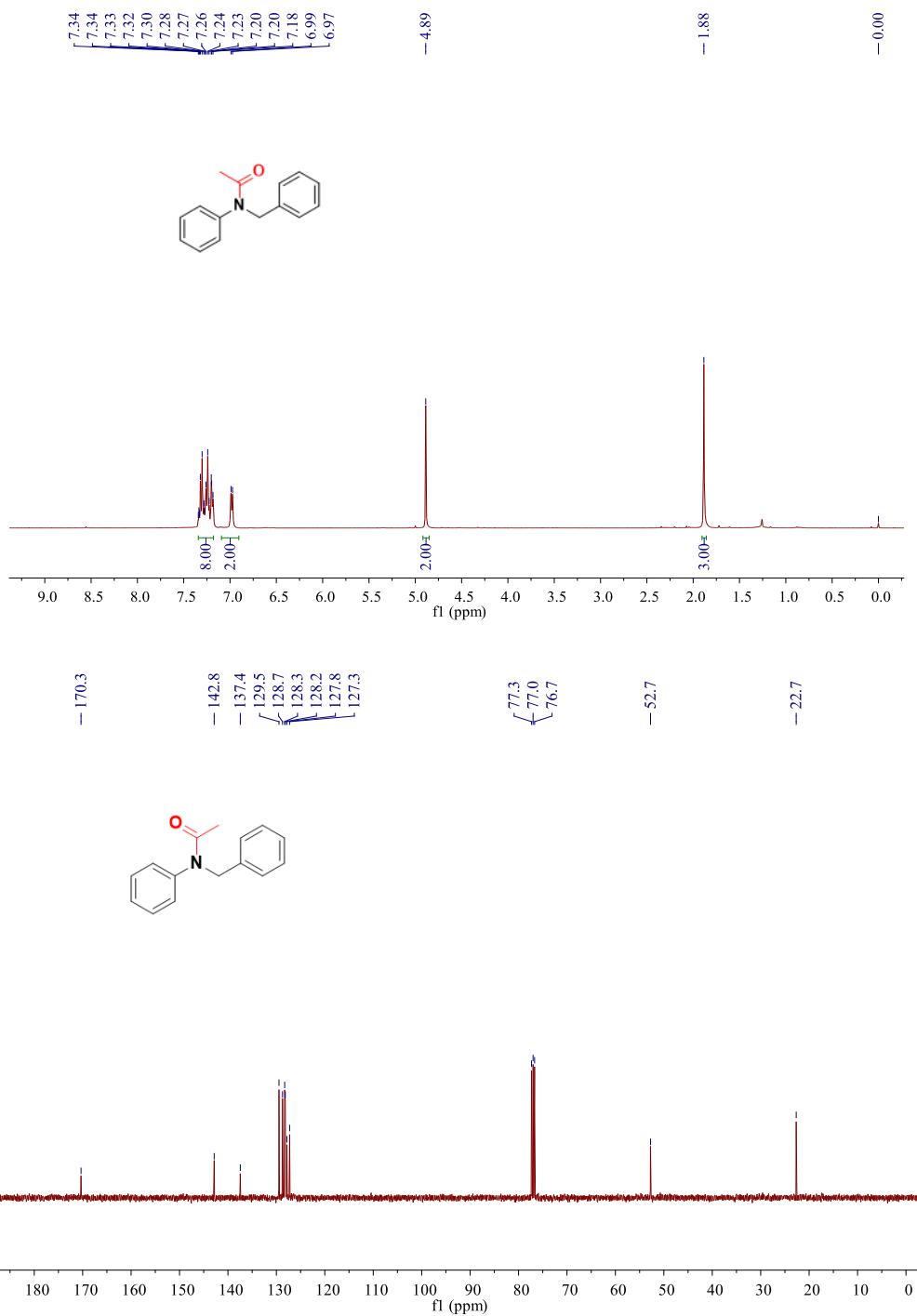
2.00H  
3.00H  
3.00H  
3.00H  
–43.6  
–22.6  
–21.1  
–12.9





**N-Benzyl-N-phenylacetamide (5bh)**

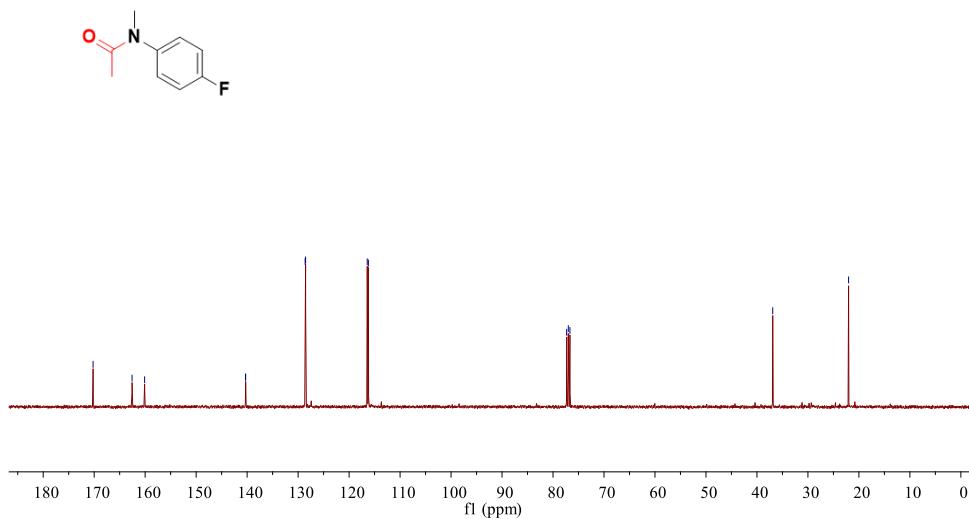
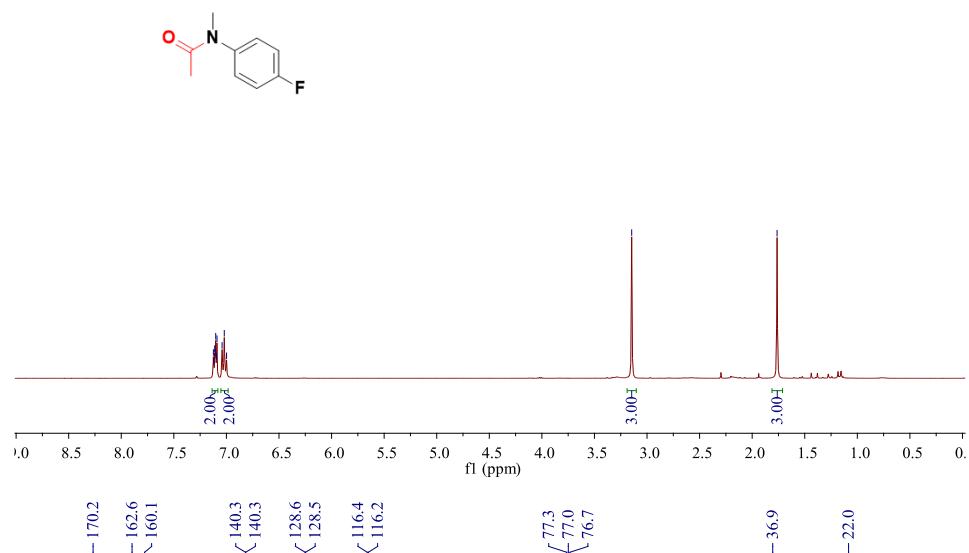
petroleum ether / ethyl acetate = 1:1, yellow solid, 69% yield (31.0 mg). mp: 55 – 57°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.18 (m, 8H), 6.99 – 6.97 (m, 2H), 4.89 (s, 2H), 1.88 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.3, 142.8, 137.4, 129.5, 128.7, 128.3, 128.2, 127.8, 127.3, 52.7, 22.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>15</sub>NO+H<sup>+</sup>: 226.1226, Found: 226.1222. **IR** (neat, cm<sup>-1</sup>): ν 2931, 1650, 1596, 1496, 1397, 1212, 1029, 906, 726, 647.



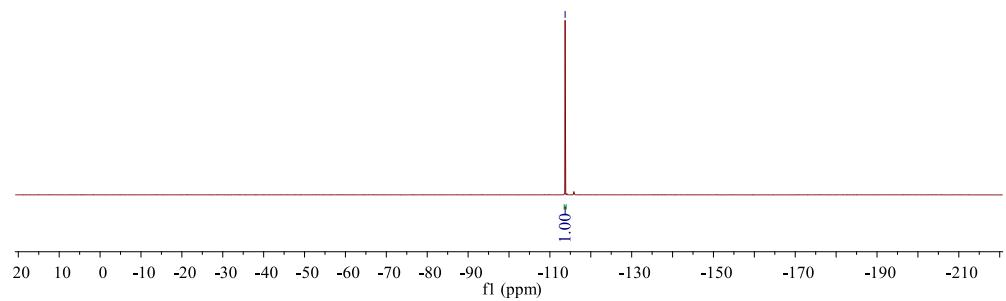
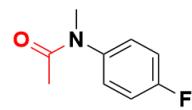


**N-(4-Fluorophenyl)-N-methylacetamide (5bi)**

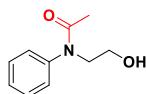
petroleum ether / ethyl acetate = 2:1, yellow solid, 74% yield (25.5 mg). mp: 60 – 63°C.  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.12 – 7.09 (m, 2H), 7.04 – 7.00 (m, 2H), 3.15 (s, 3H), 1.76 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 161.3 (d,  $J$  = 247.7 Hz), 140.3 (d,  $J$  = 3.2 Hz), 128.6 (d,  $J$  = 8.6 Hz), 116.3 (d,  $J$  = 22.7 Hz), 36.9, 22.0.  **$^{19}\text{F}$  NMR** (377 MHz,  $\text{CDCl}_3$ )  $\delta$  -113.7. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_9\text{H}_{10}\text{FNO} + \text{H}^+$ : 168.0819, Found: 168.0817. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  2934, 1767, 1652, 1509, 1423, 1354, 1222, 910, 726, 645.



-113.7

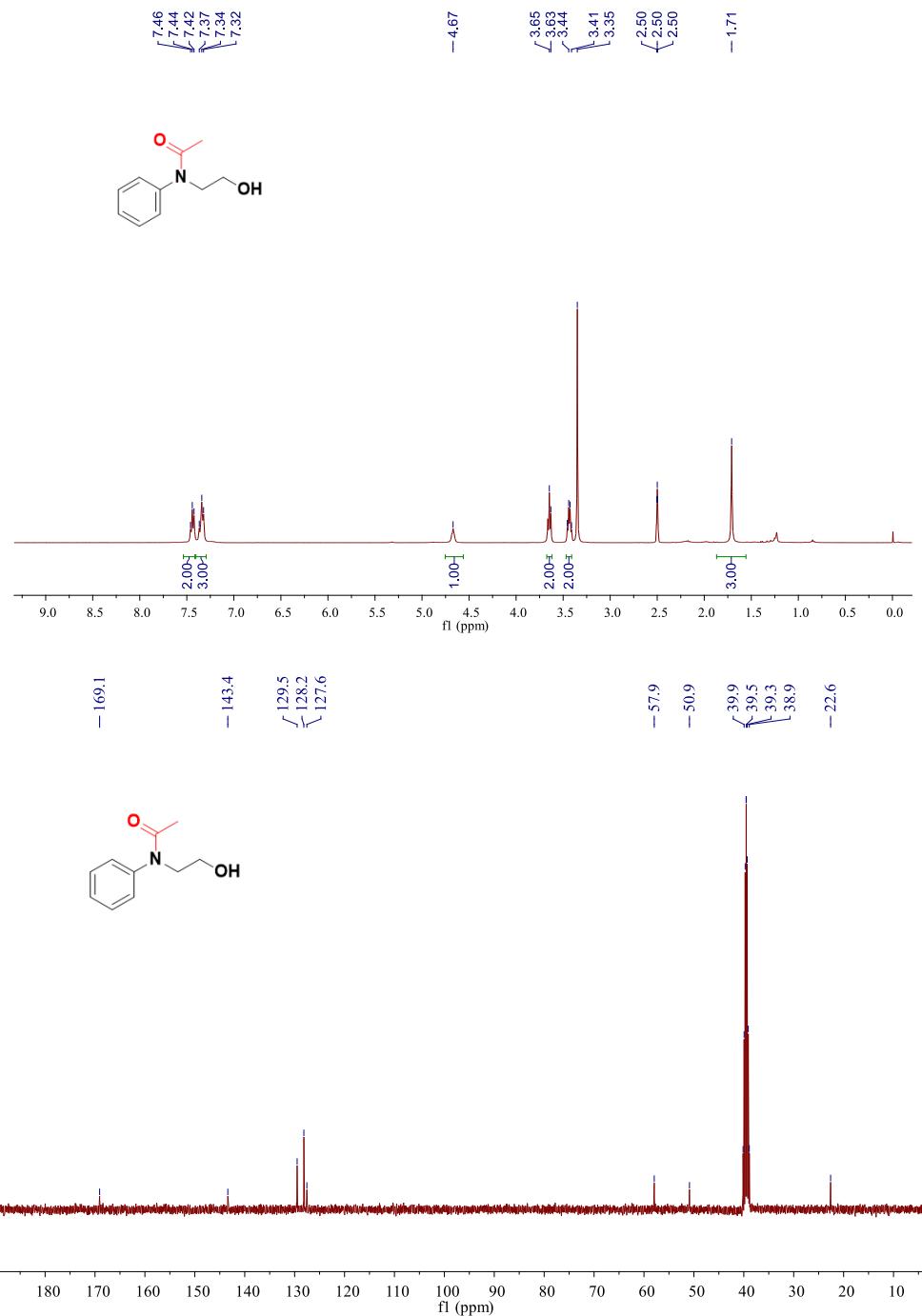


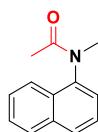
**S96**



**N-(2-Hydroxyethyl)-N-phenylacetamide (5bj)**

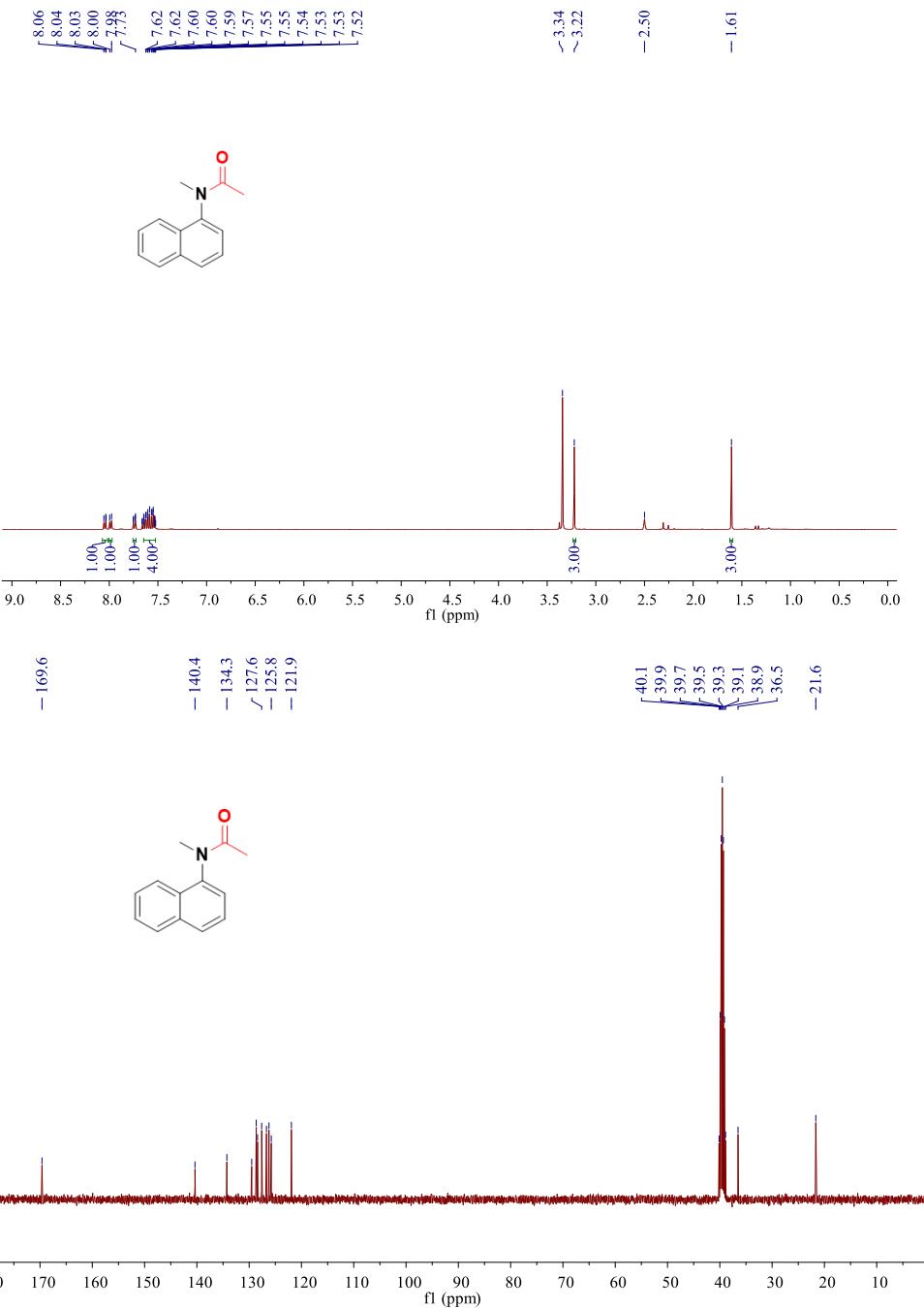
dichloromethane / ethyl acetate = 1:1, yellow solid, 63% yield (22.5 mg). mp: 59 – 61°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 7.46 – 7.42 (m, 2H), 7.37 – 7.32 (m, 3H), 4.67 (s, 1H), 3.65 (t, *J* = 6.5 Hz, 2H), 3.43 (dd, *J* = 11.9, 6.5 Hz, 2H), 1.71 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.1, 143.4, 129.5, 128.2, 127.6, 57.9, 50.9, 22.6. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>10</sub>H<sub>13</sub>NO<sub>2</sub>+H<sup>+</sup>: 180.1019, Found: 180.1016. **IR** (neat, cm<sup>-1</sup>): ν 3384, 3063, 2927, 2878, 1716, 1630, 1593, 1494, 1397, 1279, 996, 852, 733.

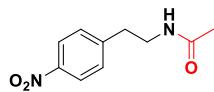




**N-Methyl-N-(naphthalen-1-yl)acetamide (5bk)**

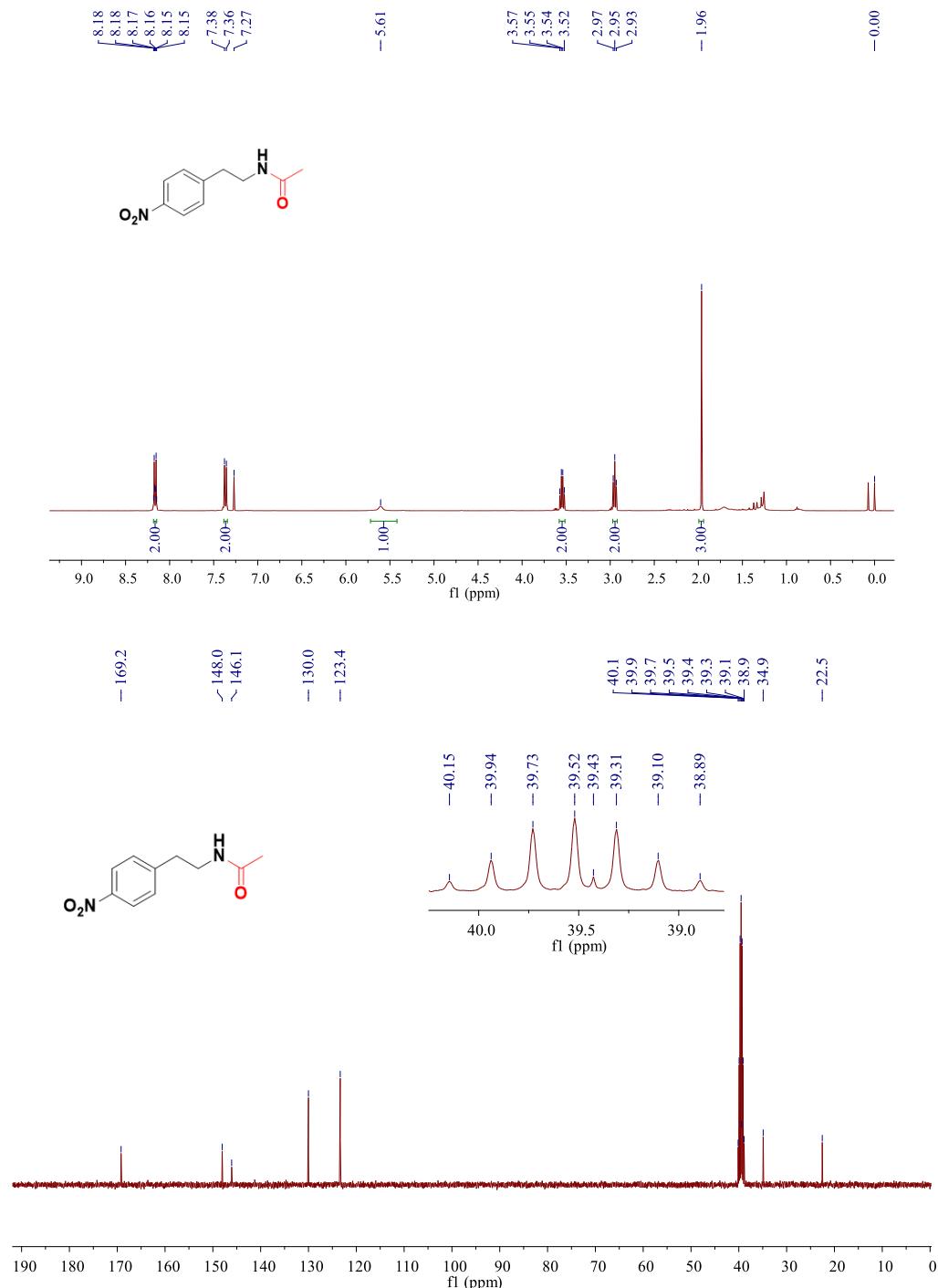
petroleum ether / ethyl acetate = 2:1, yellow solid, 52% yield (20.5 mg). mp: 90 – 93°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 8.06 – 8.03 (m, 1H), 8.00 – 7.98 (m, 1H), 7.75 – 7.73 (m, 1H), 7.66–7.52 (m, 4H), 3.22 (s, 3H), 1.61 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.6, 140.4, 134.3, 129.5, 128.4, 127.6, 126.7, 126.2, 125.8, 121.9, 36.5, 21.6. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>13</sub>NO+H<sup>+</sup>: 200.1070, Found: 200.1067. **IR** (neat, cm<sup>-1</sup>): ν 1652, 1541, 1488, 1379, 823, 761, 626.

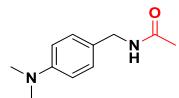




**N-(4-Nitrophenethyl)acetamide (5bl)**

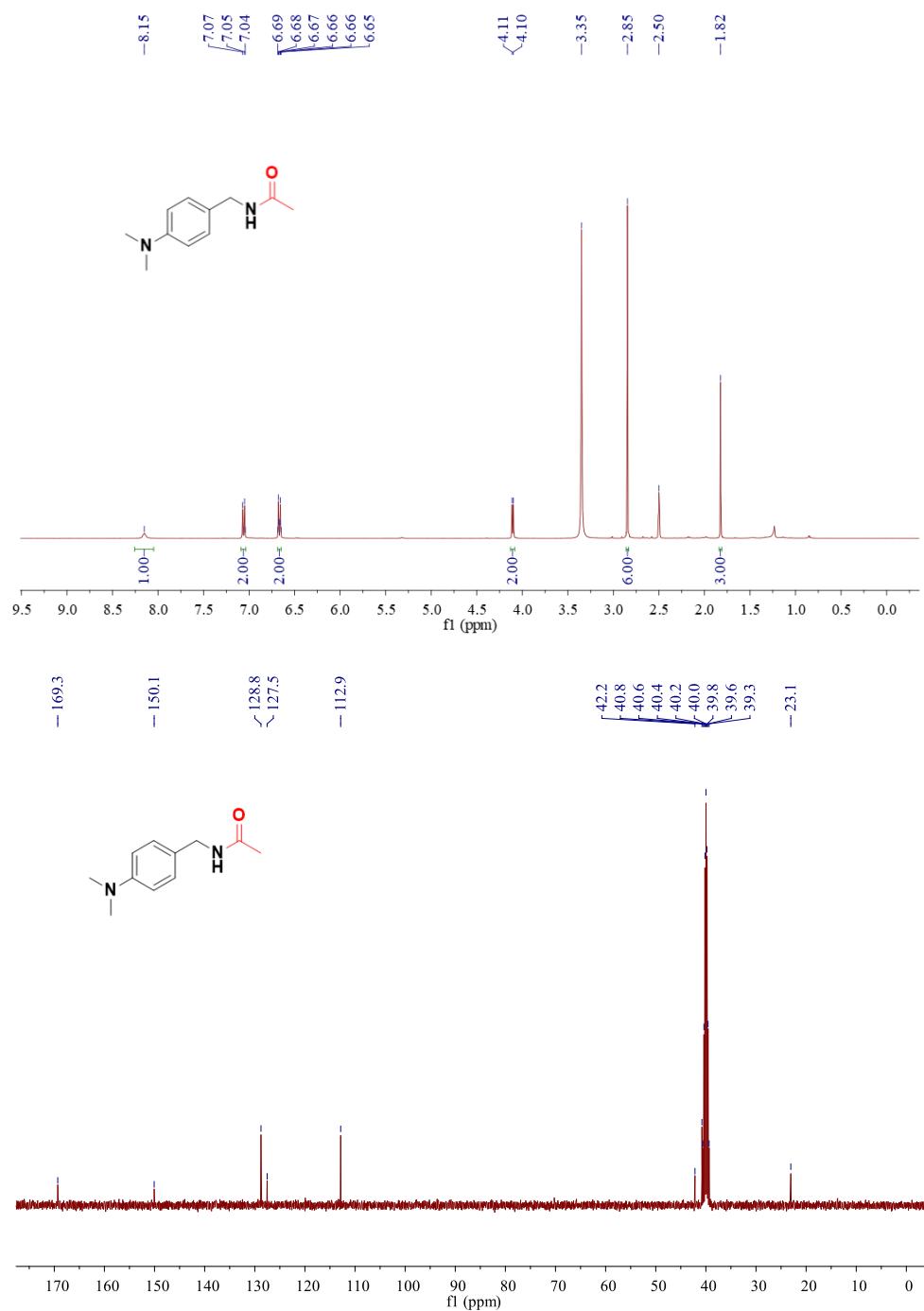
ethyl acetate, dark red oil, 55% yield (33.2 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.18 – 8.15 (m, 2H), 7.38 – 7.27 (m, 2H), 5.61 (s, 1H), 3.55 (dd, *J* = 13.2, 6.9 Hz, 2H), 2.95 (t, *J* = 6.9 Hz, 2H), 1.96 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 169.2, 148.1, 146.1, 130.0, 123.4, 34.9, 22.5. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>+Na<sup>+</sup>: 231.0740, Found: 231.0737. **IR** (neat, cm<sup>-1</sup>): ν 3299, 2854, 1711, 1661, 1516, 1344, 1256, 855, 746, 697.

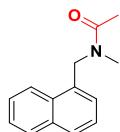




**N-(4-(Dimethylamino)benzyl)acetamide (5bm)**

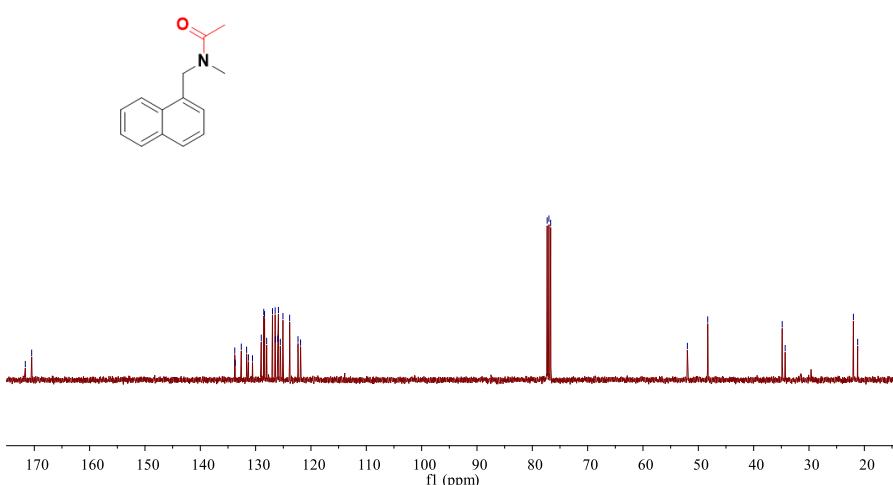
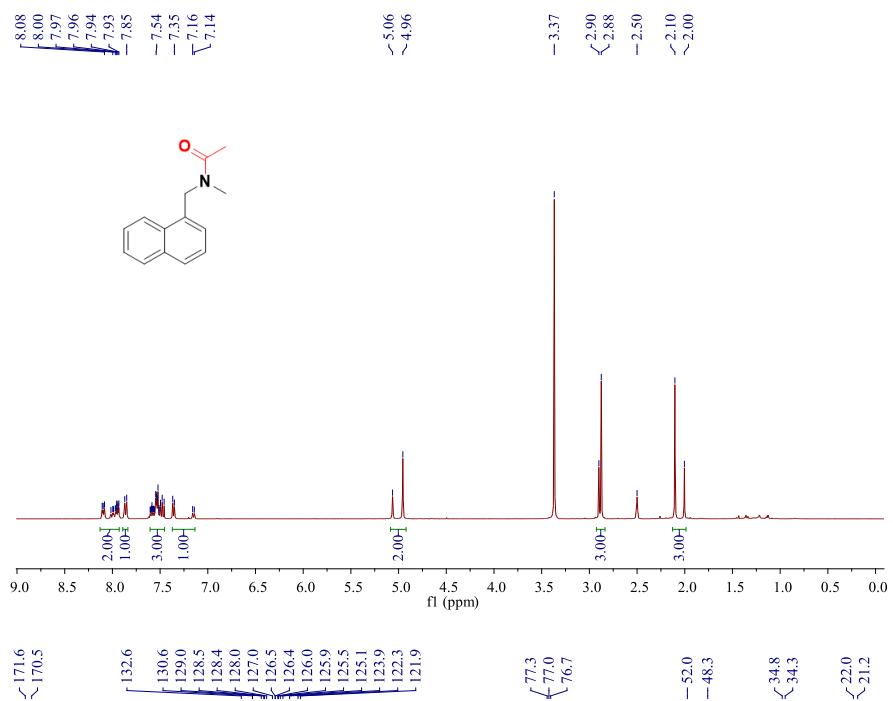
petroleum ether / ethyl acetate = 2:1, yellow oil, 55% yield (21.1 mg).  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  8.15 (s, 1H), 7.07 – 7.04 (m, 2H), 6.69 – 6.65 (m, 2H), 4.11 (d,  $J$  = 5.8 Hz, 2H), 2.85 (s, 6H), 1.82 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  169.3, 150.1, 128.8, 127.5, 112.9, 42.2, 40.8, 23.1. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}+\text{H}^+$ : 193.1335, Found: 193.1333. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  3393, 2958, 1652, 1541, 1457, 1362, 1229, 1048, 993, 824, 762, 628.

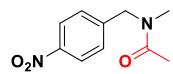




**N-Methyl-N-(naphthalen-1-ylmethyl)acetamide (5bn)**

dichloromethane / ethyl acetate = 10:1, yellow oil, 50% yield (20.9 mg). **<sup>1</sup>H NMR** (400 MHz, DMSO)  $\delta$  8.11 – 7.93 (m, 2H), 7.86 (d,  $J$  = 8.2 Hz, 1H), 7.59 – 7.46 (m, 3H), 7.37 – 7.14 (m, 1H), 5.06 (minor isomer, s, 2H), 4.96 (major isomer, s, 3H), 2.90 (minor isomer, s, 3H), 2.90 (minor isomer, s, 3H), 2.10 (major isomer, s, 3H), 2.00 (minor isomer, s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  171.6, 170.5, 133.8, 133.7, 132.6, 131.6, 131.3, 130.6, 129.0, 128.5, 128.4, 128.0, 127.0, 126.5, 126.4, 126.0, 125.9, 125.5, 125.1, 123.9, 122.3, 121.9, 52.0, 48.3, 34.8, 34.3, 22.0, 21.2. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>14</sub>H<sub>15</sub>NO+H<sup>+</sup>: 214.1226, Found: 214.1222. **IR** (neat, cm<sup>-1</sup>):  $\nu$  2961, 1636, 1510, 1485, 1398, 1260, 906, 725, 645.



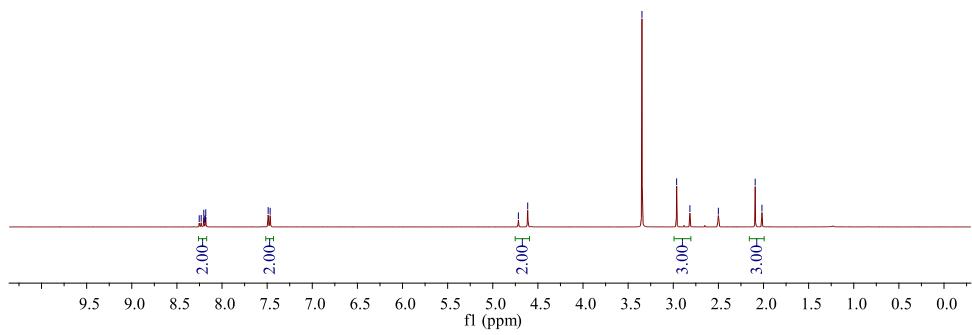
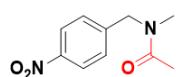


**N-methyl-N-(4-nitrobenzyl)acetamide (5bo)**

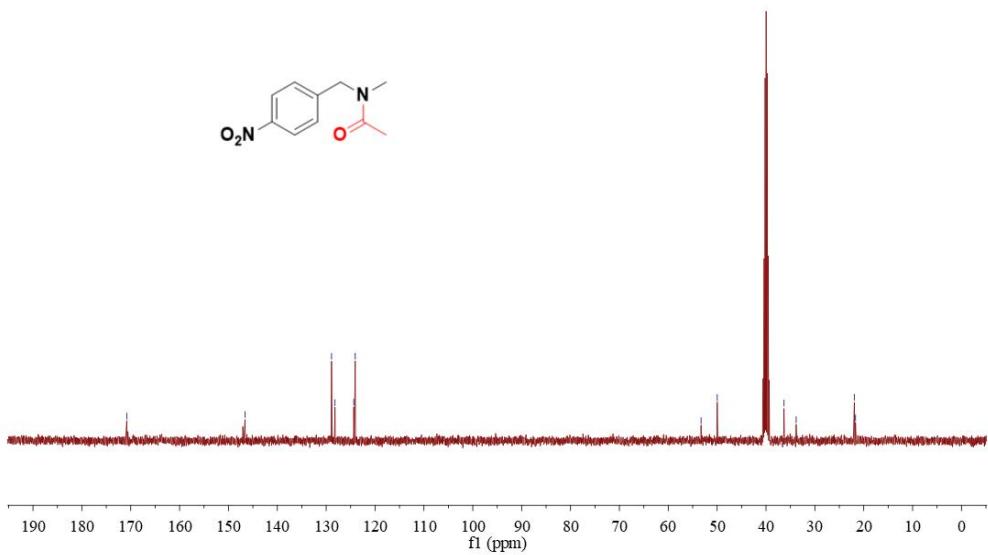
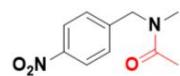
petroleum ether / ethyl acetate = 1:1, yellow solid, 65% yield (26.4 mg). mp: 80 – 83°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  8.25 – 8.18 (m, 2H), 7.49 – 7.47 (m, 2H), 4.72 (minor isomer, s, 2H), 4.61 (major isomer, s, 2H), 2.96 (major isomer, s, 3H), 2.82 (minor isomer, s, 3H), 2.09 (major isomer, s, 3H), 2.02 (minor isomer, s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  170.8, 170.5, 147.1, 146.6, 128.9, 128.2, 124.4, 124.1, 53.3, 50.0, 36.3, 33.8, 21.9, 21.7. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_3+\text{H}^+$ : 209.0921, Found: 209.0918. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  1635, 1519, 1473, 1407, 1346, 1249, 1049, 858, 736.

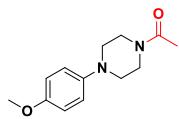
8.25  
8.23  
8.20  
8.20  
8.19  
8.18  
7.49  
7.47

4.72  
4.61  
3.35  
2.82  
2.50  
2.09  
2.02



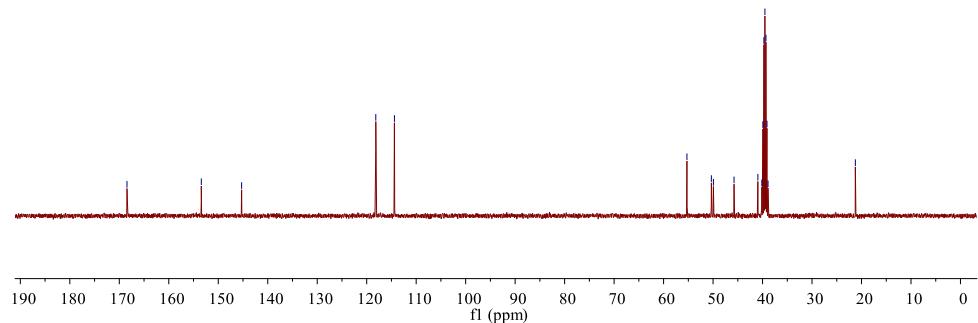
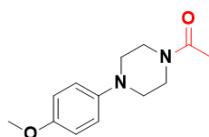
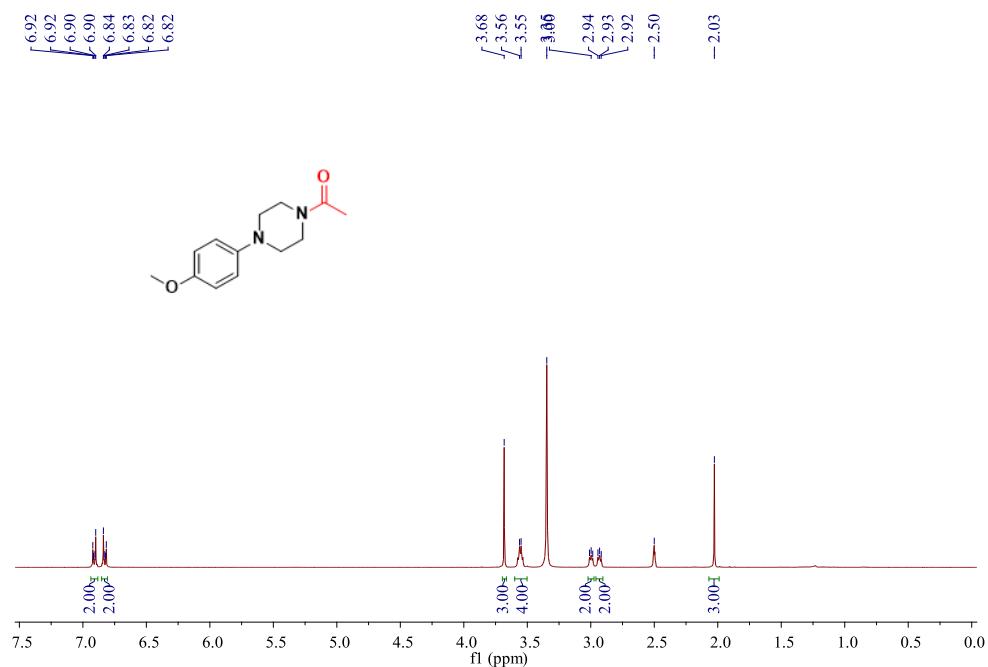
170.8  
146.6  
128.9  
128.2  
124.4  
124.1  
53.3  
50.0  
36.3  
33.8  
21.9  
21.7

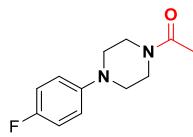




**1-(4-(4-Methoxyphenyl)piperazin-1-yl)ethan-1-one (5bp)**

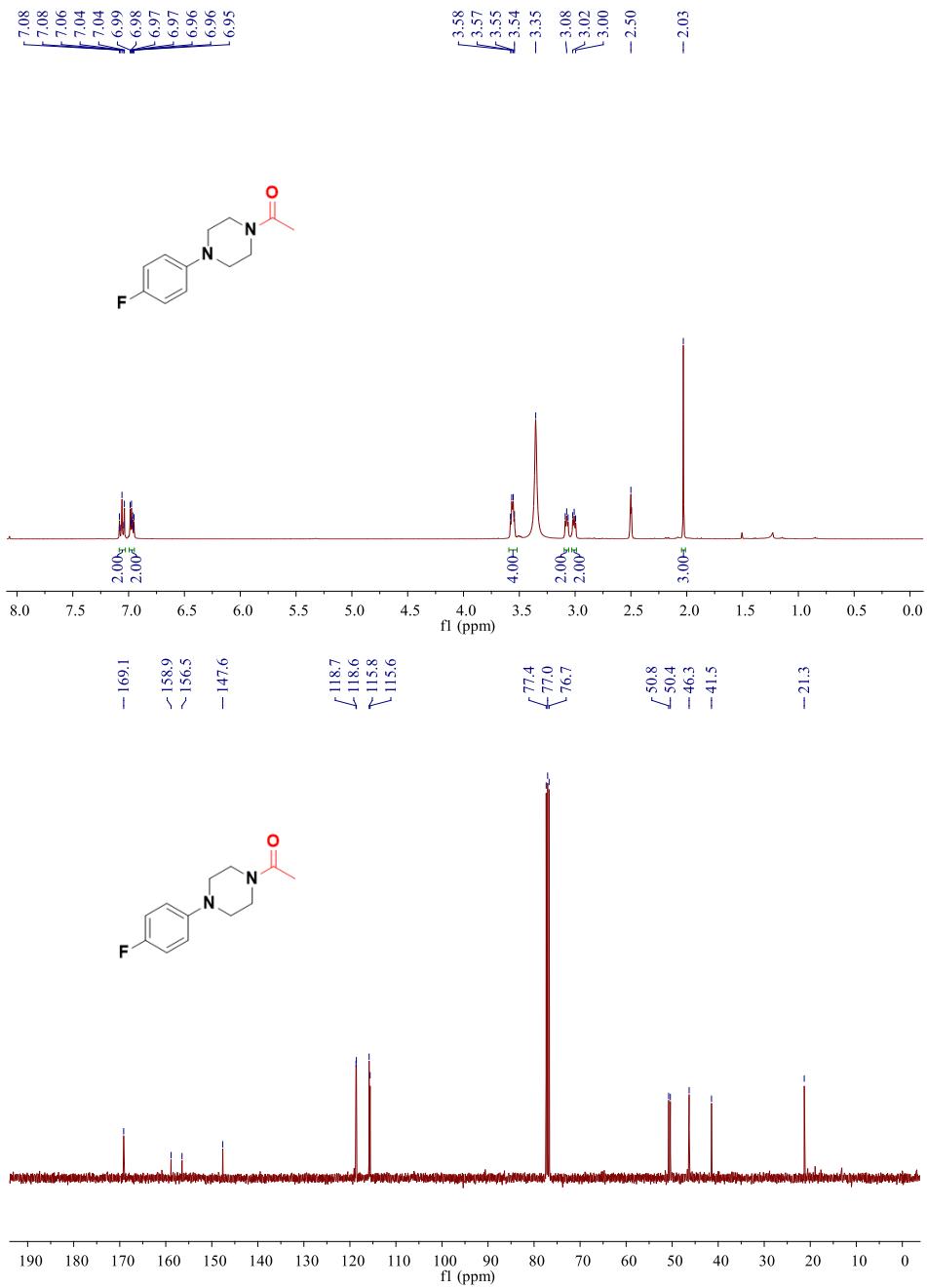
petroleum ether / ethyl acetate = 1:1, yellow solid, 60% yield (27.7 mg). mp: 74 – 76°C.  **$^1\text{H}$  NMR** (400 MHz, DMSO)  $\delta$  6.92 – 6.90 (m, 2H), 6.84 – 6.82 (m, 2H), 3.68 (s, 3H), 3.55 (dd,  $J$  = 10.3, 5.5 Hz, 4H), 3.01 – 2.98 (m, 2H), 2.94 – 2.92 (m, 2H), 2.03 (s, 3H).  **$^{13}\text{C}$  NMR** (100 MHz, DMSO)  $\delta$  168.45, 153.4, 145.3, 118.2, 114.4, 55.3, 50.3, 49.9, 45.8, 40.9, 21.2. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_2+\text{H}^+$ : 235.1441, Found: 235.1438. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  2988, 1626, 1512, 1445, 1331, 1233, 909, 823, 761.

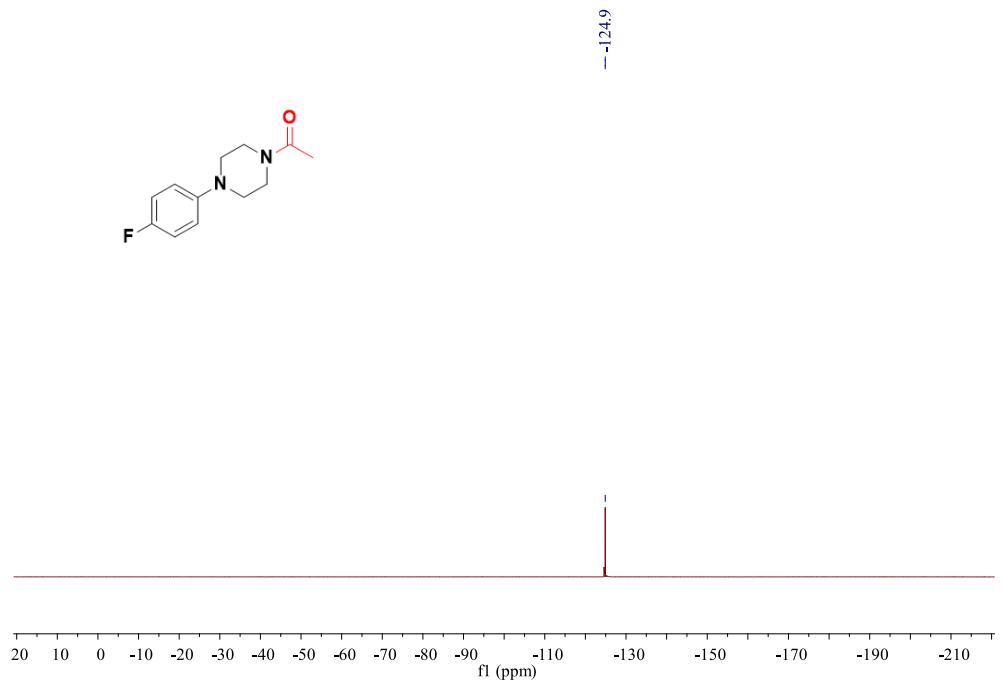


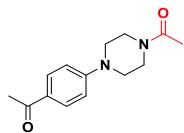


### **1-(4-(4-Fluorophenyl)piperazin-1-yl)ethan-1-one (5bq)**

petroleum ether / ethyl acetate = 1:1, yellow solid, 40% yield (17.8 mg). mp: 80 – 82°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 7.08 – 7.04 (m, 2H), 6.99 – 6.95 (m, 2H), 3.58 – 3.54 (m, 2H), 3.09 – 3.06 (m, 2H), 3.02 – 3.00 (m, 2H), 2.03 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 169.1, 157.8 (d, *J* = 239.8 Hz), 147.6, 118.6 (d, *J* = 7.9 Hz), 115.7 (d, *J* = 22.2 Hz), 50.8, 50.4, 46.3, 41.5, 21.3. **<sup>19</sup>F NMR** (377 MHz, DMSO) δ - 124.9 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>12</sub>H<sub>15</sub>FN<sub>2</sub>O+H<sup>+</sup>: 223.1241, Found: 223.1238. **IR** (neat, cm<sup>-1</sup>): ν 1652, 1541, 1488, 1379, 1314, 1823, 761, 626.

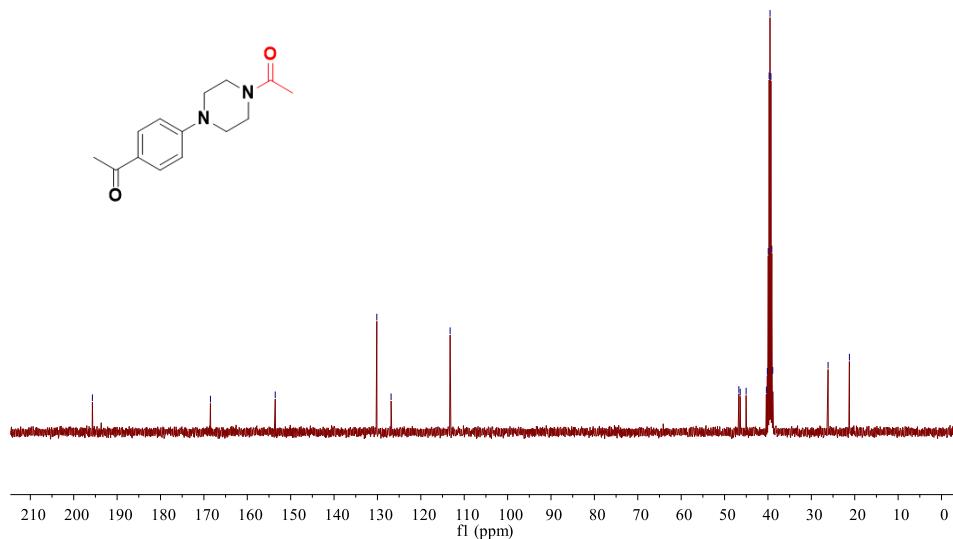
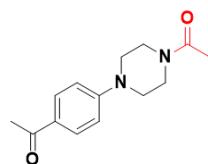
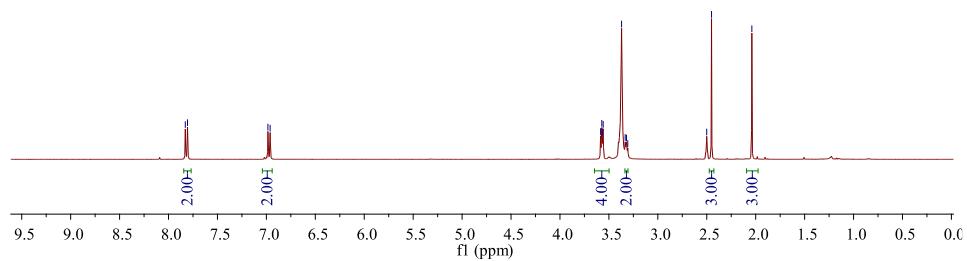
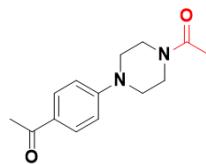


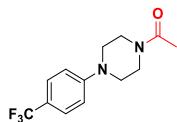




**1-(4-(4-Acetylphenyl)piperazin-1-yl)ethan-1-one (5br)**

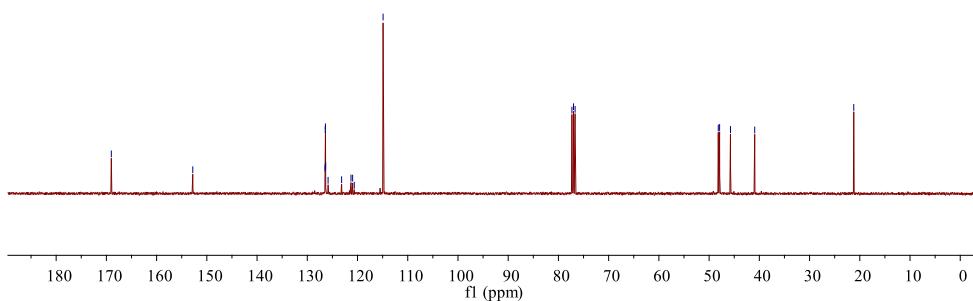
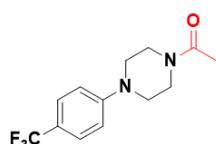
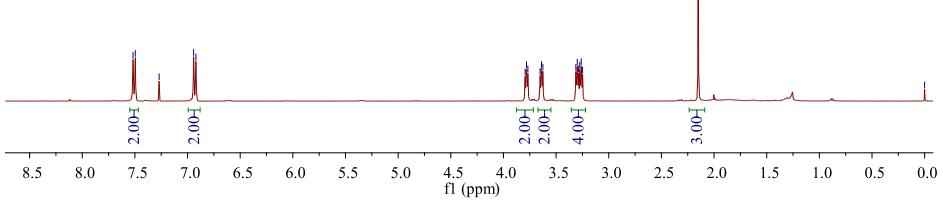
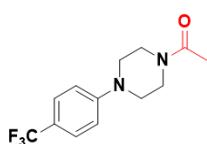
petroleum ether / ethyl acetate = 1:2, yellow oil, 45% yield (22.1 mg).  **$^1\text{H NMR}$**  (400 MHz, DMSO)  $\delta$  7.82 (d,  $J$  = 9.0 Hz, 2H), 6.97 (d,  $J$  = 9.0 Hz, 2H), 3.57 (dd,  $J$  = 6.2, 4.3 Hz, 4H), 3.35 – 3.31 (m, 2H), 2.45 (s, 3H), 2.04 (s, 3H).  **$^{13}\text{C NMR}$**  (100 MHz, DMSO)  $\delta$  195.7, 168.5, 153.6, 130.1, 126.9, 113.2, 46.7, 46.4, 45.0, 26.1, 21.2. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_2+\text{H}^+$ : 247.1441, Found: 247.1437. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  2950, 1596, 1541, 1498, 1395, 1047, 992, 825, 763.

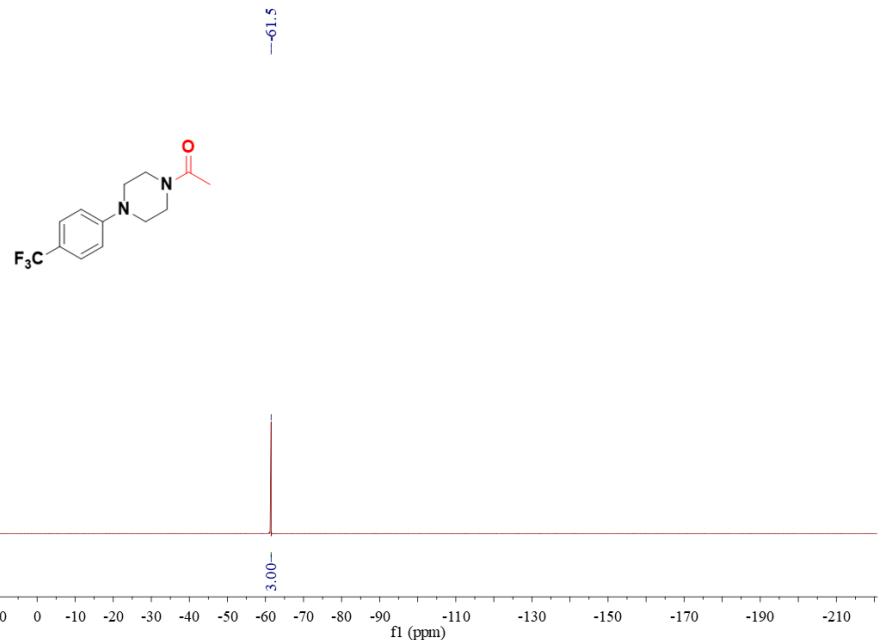


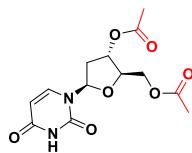


### 1-(4-(4-(Trifluoromethyl)phenyl)piperazin-1-yl)ethan-1-one (5bs)

petroleum ether / ethyl acetate = 2:1, yellow solid, 70% yield (38.1 mg). mp: 75 – 77°C. **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.51 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 3.79 – 3.77 (m, 2H), 3.65 – 3.63 (m, 2H), 3.31 – 3.25 (m, 4H), 2.15 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 169.0, 152.8, 126.4 (q, *J* = 3.8 Hz), 124.5 (q, *J* = 270.0 Hz), 121.1 (q, *J* = 30.6 Hz), 114.9, 48.2, 47.9, 45.7, 40.9, 21.2. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -61.5 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>15</sub>F<sub>3</sub>N<sub>2</sub>O+H<sup>+</sup>: 273.1209, Found: 273.1204. **IR** (neat, cm<sup>-1</sup>): ν 2989, 1614, 1523, 1442, 1329, 1231, 1098, 979, 819, 647.

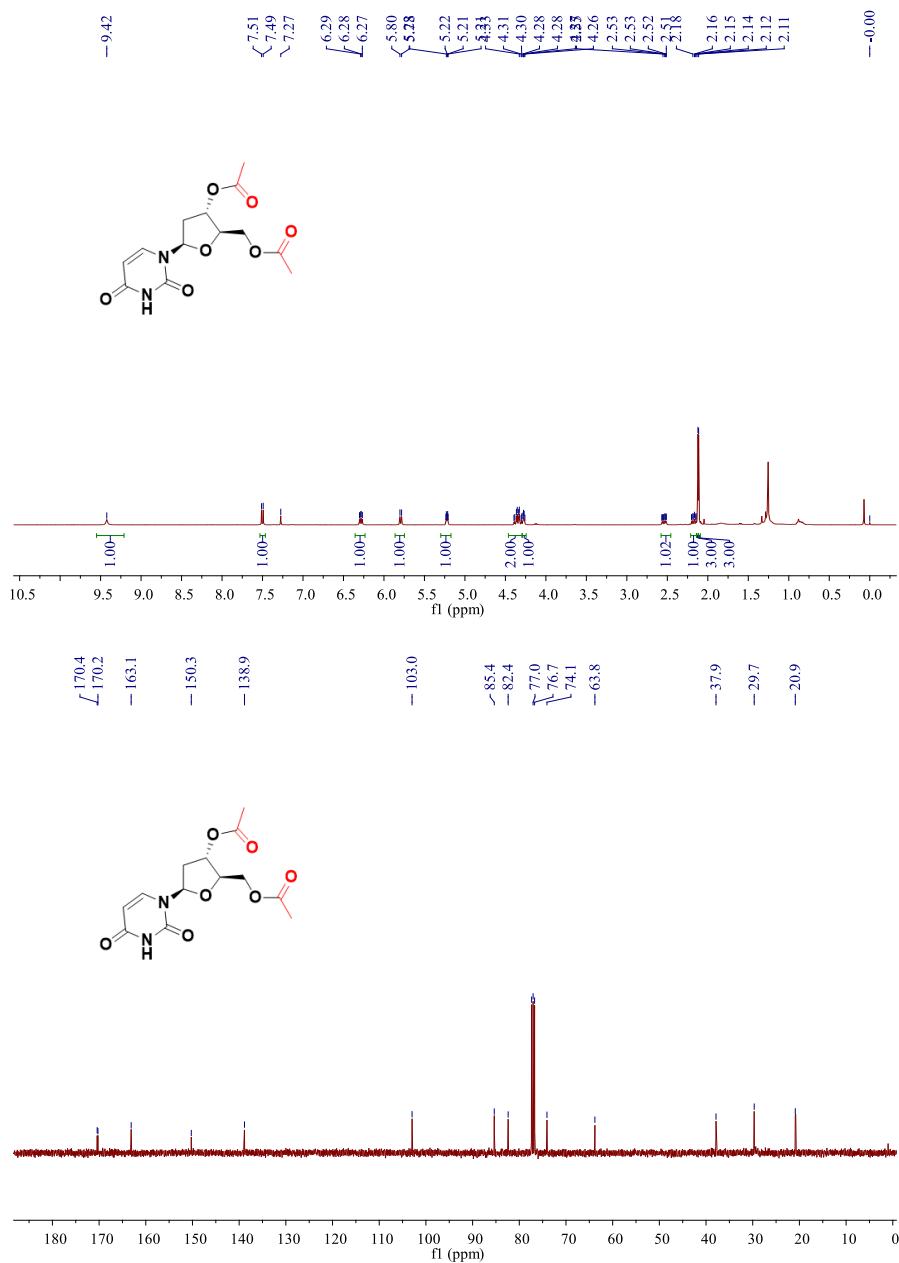


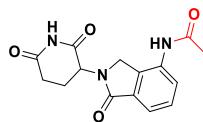




**((2R,3S,5R)-3-Acetoxy-5-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)tetrahydrofuran-2-yl)methyl acetate (6)**

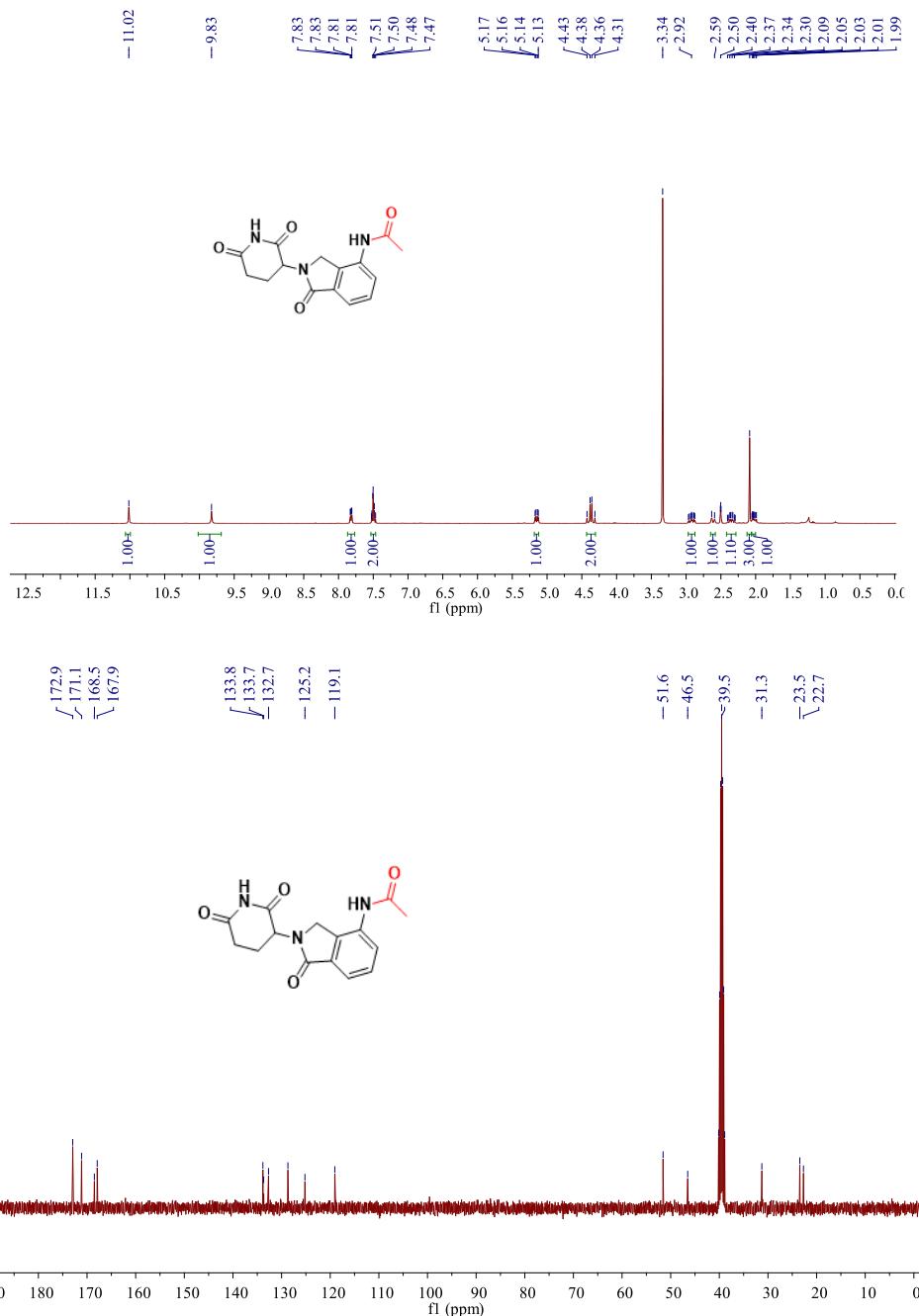
petroleum ether / ethyl acetate = 1:1, yellow oil, 40% yield (24.8 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.42 (s, 1H), 7.50 (d, *J* = 8.2 Hz, 1H), 6.28 (dd, *J* = 8.2, 5.7 Hz, 1H), 5.79 (d, *J* = 8.2 Hz, 1H), 5.23 – 5.21 (m, 1H), 4.39 – 4.30 (m, 2H), 4.28 – 4.26 (m, 1H), 2.57 – 2.51 (m, 1H), 2.20 – 2.14 (m, 1H), 2.12 (s, 3H), 2.11 (s, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 170.4, 170.2, 163.1, 150.3, 138.9, 103.0, 85.4, 82.4, 74.1, 63.8, 37.9, 29.7, 20.9. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>13</sub>H<sub>16</sub>N<sub>2</sub>O<sub>7</sub>+H<sup>+</sup>: 313.1030, Found: 313.1030. **IR** (neat, cm<sup>-1</sup>): ν 2956, 2851, 1740, 1689, 1460, 1379, 1379, 1232, 1197, 905, 726, 648.

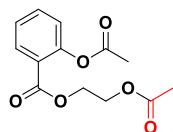




#### *N*-(2-(2,6-Dioxopiperidin-3-yl)-1-oxoisoindolin-4-yl)acetamide (7)

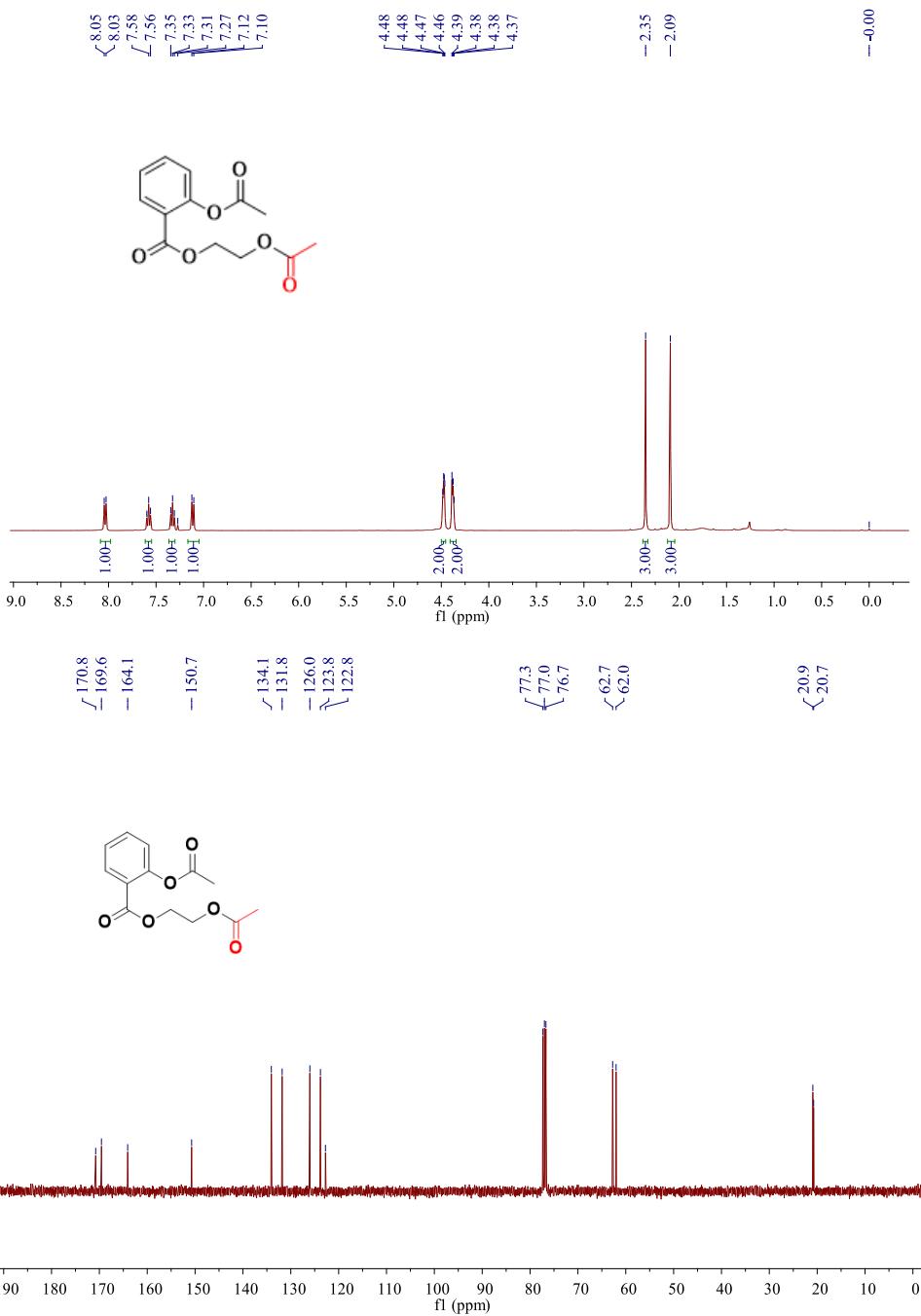
ethyl acetate, yellow solid, 50% yield (30.0 mg). mp: 235 – 237°C. **<sup>1</sup>H NMR** (400 MHz, DMSO) δ 11.02 (s, 1H), 9.83 (s, 1H), 7.83–7.81 (m, 1H), 7.52 – 7.47 (m, 2H), 5.15 (dd, *J* = 13.3, 5.1 Hz, 1H), 4.43 – 4.31 (q, *J* = 17.5 Hz, 2H), 2.97 – 2.88 (m, 1H), 2.63 – 2.59 (m, 1H), 2.40 – 2.29 (m, 1H), 2.09 (s, 3H), 2.05 – 1.99 (m, 1H). **<sup>13</sup>C NMR** (100 MHz, DMSO) δ 172.9, 171.1, 168.5, 167.9, 133.8, 133.7, 132.7, 128.7, 125.20, 119.1, 51.6, 46.5, 31.3, 23.5, 22.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>15</sub>H<sub>15</sub>N<sub>3</sub>O<sub>4</sub>+Na<sup>+</sup>: 324.0955, Found: 324.0953. **IR** (neat, cm<sup>-1</sup>): ν 3379, 2960, 1662, 1545, 1462, 1322, 1267, 1046, 991, 826, 763.

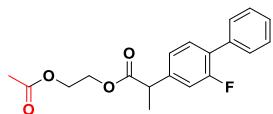




**2-Acetoxyethyl 2-acetoxybenzoate (8)**

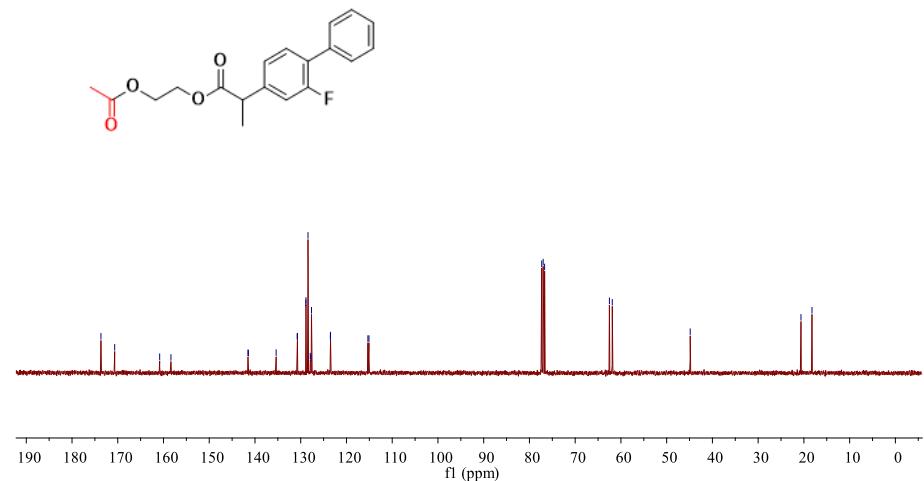
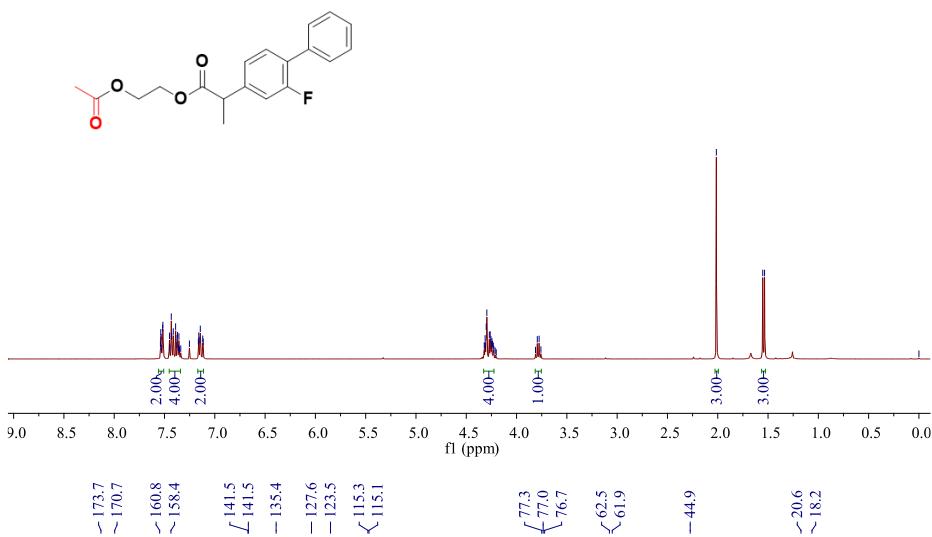
petroleum ether / ethyl acetate = 5:1, colorless oil, 76% yield (40.6 mg).  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.04 (d,  $J = 7.8$  Hz, 1H), 7.58 (t,  $J = 7.7$  Hz, 1H), 7.33 (t,  $J = 7.6$  Hz, 1H), 7.11 (d,  $J = 8.1$  Hz, 1H), 4.47 (dd,  $J = 5.4, 3.5$  Hz, 2H), 4.38 (dd,  $J = 5.4, 3.5$  Hz, 2H), 2.35 (s, 3H), 2.09 (s, 3H).  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 169.6, 164.1, 150.7, 134.1, 131.8, 126.0, 123.8, 122.8, 62.7, 62.0, 20.9, 20.7. **HRMS** (ESI-TOF): Anal Calcd. For.  $\text{C}_{13}\text{H}_{14}\text{O}_6+\text{Na}^+$ : 289.0683, Found: 289.0682. **IR** (neat,  $\text{cm}^{-1}$ ):  $\nu$  1729, 1608, 1485, 1371, 1230, 1161, 905, 726, 648.

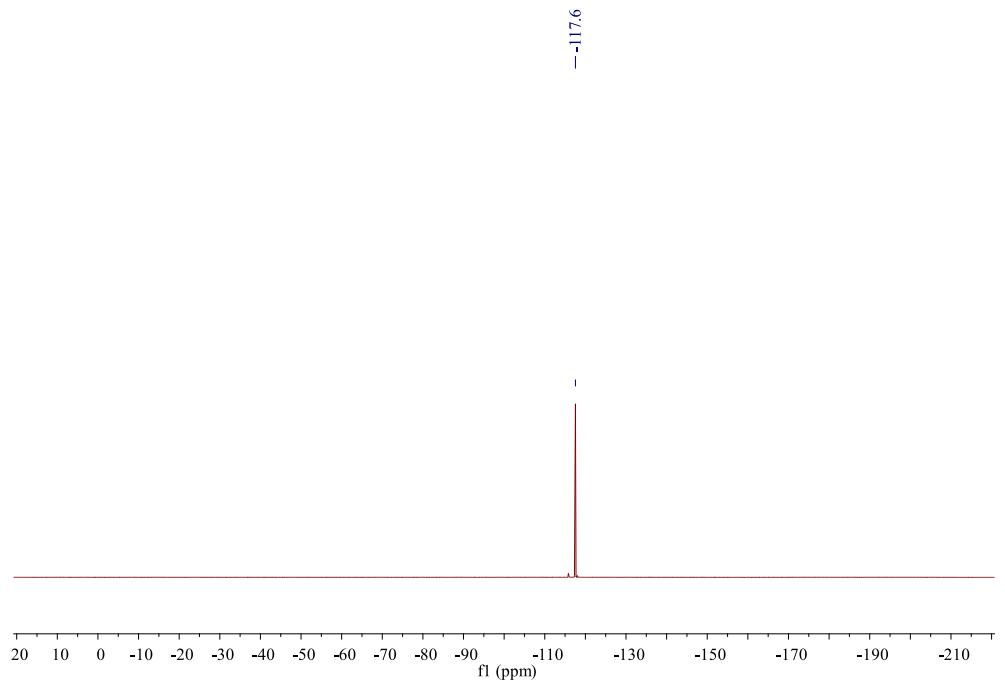


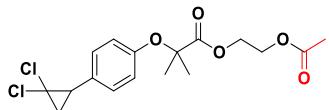


**2-Acetoxyethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (9)**

petroleum ether / ethyl acetate = 5:1, yellow oil, 85% yield (46.0 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.54 – 7.52 (m, 2H), 7.45 – 7.34 (m, 4H), 7.16 – 7.12 (m, 2H), 4.32 – 4.20 (m, 4H), 3.78 (q, *J* = 7.2 Hz, 1H), 2.01 (s, 3H), 1.54 (d, *J* = 7.2 Hz, 3H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 173.7, 170.7, 159.60 (d, *J* = 248.2 Hz), 141.5 (d, *J* = 7.7 Hz), 135.4 (d, *J* = 1.4 Hz), 130.7 (d, *J* = 4.0 Hz), 128.9 (d, *J* = 2.9 Hz), 128.4, 127.8 (d, *J* = 13.7 Hz), 127.6, 123.5 (d, *J* = 3.3 Hz), 115.2 (d, *J* = 23.7 Hz), 62.5, 61.9, 44.9, 20.6, 18.2. **<sup>19</sup>F NMR** (377 MHz, CDCl<sub>3</sub>) δ -117.6 (s, 1F). **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>19</sub>H<sub>19</sub>FO<sub>4</sub> + Na<sup>+</sup>: 353.1160, Found: 353.1157. **IR** (neat, cm<sup>-1</sup>): ν 2855, 1735, 1600 1515, 1402, 1237, 915, 822, 723.







**2-Acetoxyethyl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (10)**

petroleum ether / ethyl acetate = 5:1, yellow oil, 40% yield (29.9 mg). **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.12 – 7.09 (m, 2H), 6.84 – 6.80 (m, 2H), 4.39 – 4.36 (m, 2H), 4.27 – 4.24 (m, 2H), 2.82 (dd, *J* = 10.6, 8.4 Hz, 1H), 1.99 (s, 3H), 1.94 (dd, *J* = 10.6, 7.4 Hz, 1H), 1.77 (dd, *J* = 8.4, 7.4 Hz, 1H), 1.61 (s, 6H). **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 174.1, 170.7, 154.8, 129.6, 128.3, 118.8, 118.7, 79.1, 62.9, 61.8, 34.8, 25.8, 25.4, 25.4, 20.7. **HRMS** (ESI-TOF): Anal Calcd. For. C<sub>17</sub>H<sub>20</sub><sup>35</sup>Cl<sub>2</sub>O<sub>5</sub>+Na<sup>+</sup>:397.0580, Found: 397.0579. Anal Calcd. For. C<sub>17</sub>H<sub>20</sub><sup>35</sup>Cl<sup>37</sup>ClO<sub>5</sub>+Na<sup>+</sup>:399.0551, Found: 399.0548. Anal Calcd. For. C<sub>17</sub>H<sub>20</sub><sup>37</sup>Cl<sub>2</sub>O<sub>5</sub>+Na<sup>+</sup>:401.0521, Found: 401.0518. **IR** (neat, cm<sup>-1</sup>):  $\nu$  2849, 1737, 1611, 1581, 1464, 1374, 1229, 1140, 1048, 754, 760.

