

Supporting Information

Synthesis of *N*-Acetoxy-*N*-arylamides via Diacetoxyiodobenzene Promoted Double Acylation Reaction of Hydroxylamines with Aldehydes

Huaiyuan Zhang,^a Yingpeng Su,^a Ke-Hu Wang,^a Danfeng Huang,^a Jun Li,^a Yulai Hu^{*a,b}

^aCollege of Chemistry and Chemical Engineering, Northwest Normal University, 967 Anning East Road, Lanzhou 730070, China

^bState Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 730000, China.

E-mail: huyl@nwnu.edu.cn

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Experimental Section

1 General methods.

All of the new compounds were characterized on the basis of ^1H NMR, ^{13}C NMR and HRMS data. Chemical shifts of ^1H NMR and ^{13}C NMR are reported in ppm using tetramethylsilane (TMS) as an internal standard.

2 General Procedure for the Synthesis of Compounds 4 from 1, 2 and 3a.

The aldehyde **1** (0.47 mmol, 1 equiv) and hydroxylamine **2** (0.47 mmol, 1 equiv) was put in a dried round-bottom flask (50 mL) fitted with a magnetic bar. Then dichloromethane (5 mL) was added. The mixture was stirred at room temperature and the reaction was monitored by TLC. After complete generation of nitrones, the reaction mixture was concentrated under reduced pressure, and diacetoxyiodobenzene **3a** (0.94 mmol, 2 equiv) in acetic acid (4 mL) were added into the flask. The resulting mixture was stirred at room temperature under nitrogen atmosphere for 1 h, and then the saturated Na_2CO_3 solution (10 mL) was poured into the flask. After stirring for 10 min, the mixture was extracted with EtOAc (3×10 mL). The organic layer were combined, dried over anhydrous MgSO_4 and then concentrated under reduced pressure. The residue was purified by silica gel column chromatography using PE : EA (10:1) as eluent to afford the product **4**.

3 General Procedure for the Synthesis of Compounds 4 from 7 and 3a.

The nitrone **7** (0.25 mmol, 1 equiv) and diacetoxyiodobenzene **3a** (0.50 mmol, 2 equiv) were put in a dried round-bottom flask (50 mL) fitted with a magnetic bar. Then acetic acid (4 mL) was added under nitrogen atmosphere. The mixture was stirred at room temperature for 1 h, and then the saturated Na_2CO_3 solution was poured into the flask. After stirring for 10 min, the mixture was extracted with EtOAc (3×10 mL). The organic layer were combined, dried over anhydrous MgSO_4 and then concentrated under reduced pressure. The residue was purified by silica gel column chromatography using PE : EA (10:1) as eluent to afford the product **4**.

***N*-acetoxy-*N*-phenylbenzamide (4a)**. White solid, 90.0 mg, yield 75%. m.p. 47–48 °C; ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.53–7.52 (m, 2H), 7.38–7.35 (m, 1H), 7.31–7.25 (m, 7H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) 167.9, 166.8,

140.6, 133.2, 131.0, 129.2, 128.8, 128.4, 128.1, 126.9, 18.4; HRMS (ESI) M/Z calcd for C₁₅H₁₄NO₃ [M+H⁺] 256.0968, found: 256.0970.

***N*-acetoxy-2-methyl-*N*-phenylbenzamide (4b).** White solid, 78.5 mg, yield 62%. m.p. 61–62 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.30–7.19 (m, 7H), 7.13 (d, *J* = 7.8 Hz, 1H), 7.06 (t, *J* = 7.2 Hz, 1H), 2.44 (s, 3H), 2.13 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 139.4, 135.9, 133.9, 130.5, 129.8, 129.0, 128.3, 127.5, 126.1, 125.3, 19.3, 18.2; HRMS (ESI) M/Z calcd for C₁₆H₁₆NO₃ [M+H⁺] 270.1125, found: 270.1123.

***N*-acetoxy-3-methyl-*N*-phenylbenzamide (4c).** White solid, 81.0 mg, yield 64%. m.p. 90–91 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.40 (s, 1H), 7.31–7.29 (m, 4H), 7.27–7.25 (m, 2H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.2 Hz, 1H), 2.28 (s, 3H), 2.18 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.8, 167.0, 140.6, 138.0, 133.1, 131.8, 129.3, 129.1, 128.3, 127.8, 126.8, 125.8, 21.2, 18.4; HRMS (ESI) M/Z calcd for C₁₆H₁₆NO₃ [M+H⁺] 270.1125, found: 270.1122.

***N*-acetoxy-4-methyl-*N*-phenylbenzamide (4d).** White solid, 75.9 mg, yield 60%. m.p. 88–89 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.42 (d, *J* = 7.8 Hz, 2H), 7.30–7.25 (m, 5H), 7.06 (d, *J* = 7.8 Hz, 2H), 2.30 (s, 3H), 2.19 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 166.8, 141.5, 140.8, 130.2, 129.1, 128.9, 128.7, 128.3, 126.9, 21.4, 18.4; HRMS (ESI) M/Z calcd for C₁₆H₁₆NO₃ [M+H⁺] 270.1125, found: 270.1123.

***N*-acetoxy-3-methoxy-*N*-phenylbenzamide (4e).** White solid, 100.6 mg, yield 75%. m.p. 68–69 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.31–7.28 (m, 4H), 7.27–7.24 (m, 1H), 7.14 (t, *J* = 7.8 Hz, 1H), 7.07–7.05 (m, 2H), 6.89–6.88 (m, 1H), 3.69 (s, 3H), 2.17 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.8, 166.4, 159.1, 140.5, 134.3, 129.1, 129.0, 128.4, 126.8, 121.0, 117.4, 113.5, 55.2, 18.3; HRMS (ESI) M/Z calcd for C₁₆H₁₅NO₄Na [M+Na⁺] 308.0893, found: 308.0889.

***N*-acetoxy-4-methoxy-*N*-phenylbenzamide (4f).** White solid, 83.1 mg, yield 62%. m.p. 74–75 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.51 (d, *J* = 9.0 Hz, 2H), 7.33–7.27 (m, 5H), 6.76 (d, *J* = 9.0 Hz, 2H), 3.78 (s, 3H), 2.20 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 168.0, 166.5, 161.8, 141.2, 131.0, 129.2, 128.3, 127.0, 125.1,

113.4, 55.3, 18.5; HRMS (ESI) M/Z calcd for $C_{16}H_{15}NO_4Na$ $[M+Na^+]$ 308.0893, found: 308.0889.

***N*-acetoxy-2-chloro-*N*-phenylbenzamide (4g).** White solid, 87.1 mg, yield 64%. m.p. 88–89 °C; 1H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.38–7.26 (m, 9H), 2.25 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 167.6, 162.8, 138.8, 134.0, 131.1, 130.9, 129.7, 129.0, 127.6, 126.5, 110.0, 18.1; HRMS (ESI) M/Z calcd for $C_{15}H_{12}ClNO_3Na$ $[M+Na^+]$ 312.0398, found: 312.0399.

***N*-acetoxy-3-chloro-*N*-phenylbenzamide (4h).** White solid, 104.3 mg, yield 76%. m.p. 102–103 °C; 1H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.55 (s, 1H), 7.37–7.28 (m, 7H), 7.20 (t, $J = 7.8$ Hz, 1H), 2.19 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 167.7, 165.3, 140.1, 135.0, 134.2, 131.1, 129.4, 129.3, 128.9, 128.8, 127.0, 126.8, 18.3; HRMS (ESI) M/Z calcd for $C_{15}H_{12}ClNO_3Na$ $[M+Na^+]$ 312.0398, found: 312.0401.

***N*-acetoxy-4-chloro-*N*-phenylbenzamide (4i).** White solid, 81.7 mg, yield 60%. m.p. 105–107 °C; 1H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.47 (d, $J = 7.8$ Hz, 2H), 7.34–7.27 (m, 5H), 7.25 (d, $J = 8.4$ Hz, 2H), 2.19 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 167.7, 165.6, 140.2, 137.2, 131.6, 130.2, 129.3, 128.6, 128.4, 126.9, 18.3; HRMS (ESI) M/Z calcd for $C_{15}H_{13}ClNO_3$ $[M+H^+]$ 290.0578, found: 290.0576.

***N*-acetoxy-3-nitro-*N*-phenylbenzamide (4k).** Yellow oil, 119.9 mg, yield 85%. 1H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.38 (s, 1H), 8.23 (d, $J = 8.4$ Hz, 1H), 7.86 (d, $J = 7.8$ Hz, 1H), 7.49 (t, $J = 6.0$ Hz, 1H), 7.36–7.32 (m, 5H), 2.21 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 167.6, 147.7, 139.6, 134.9, 134.5, 129.6, 129.4, 129.3, 127.3, 125.6, 123.9, 18.3; HRMS (ESI) M/Z calcd for $C_{15}H_{12}N_2O_5Na$ $[M+Na^+]$ 323.0638, found: 323.0635.

***N*-acetoxy-4-nitro-*N*-phenylbenzamide (4l).** White solid, 103.0 mg, yield 73%. m.p. 157–158 °C; 1H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.14 (d, $J = 9.0$ Hz, 2H), 7.69 (d, $J = 8.4$ Hz, 2H), 7.35–7.32 (m, 5H), 2.21 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 167.6, 148.9, 139.4, 129.7, 129.5, 129.3, 127.1, 123.4, 110.0, 18.3; HRMS (ESI) M/Z calcd for $C_{15}H_{12}N_2O_5Na$ $[M+Na^+]$ 323.0638, found: 323.0634.

***N*-acetoxy-4-cyano-*N*-phenylbenzamide (4m).** White solid, 93.5 mg, yield 71%. m.p.

105–106 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.61 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.35–7.29 (m, 5H), 2.19 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.6, 164.6, 139.6, 137.5, 131.9, 129.5, 129.2, 129.1, 127.0, 117.8, 114.6, 18.2; HRMS (ESI) *M/Z* calcd for C₁₆H₁₂N₂O₃Na [M+Na⁺] 303.0740, found: 303.0737.

***N*-acetoxy-*N*-phenylfuran-2-carboxamide (4n).** White solid, 59.9 mg, yield 52%. m.p. 70–71 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.50–7.48 (m, 2H), 7.44–7.40 (m, 4H), 6.66 (d, *J* = 3.0 Hz, 1H), 6.38 (dd, *J* = 3.6, 1.8 Hz, 1H), 2.25 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 164.6, 151.7, 148.5, 140.1, 136.6, 128.9, 128.2, 125.2, 124.4, 18.3; HRMS (ESI) *M/Z* calcd for C₁₃H₁₂NO₄ [M+H⁺] 246.0761, found: 246.0758.

***N*-acetoxy-*N*-phenylpicolinamide (4o).** White solid, 92.7 mg, yield 77%. m.p. 75–76 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 8.45 (s, 1H), 7.79–7.72 (m, 2H), 7.39–7.25 (m, 6H), 2.19 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 156.2, 145.4, 145.3, 140.0, 129.3, 129.2, 127.4, 118.2, 111.5, 18.4; HRMS (ESI) *M/Z* calcd for C₁₄H₁₃N₂O₃ [M+H⁺] 257.0921, found: 257.0918.

***N*-acetoxy-*N*-(*o*-tolyl)benzamide (4r).** White solid, 64.6 mg, yield 51%. m.p. 65–66 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.49 (d, *J* = 7.2 Hz, 2H), 7.35–7.32 (m, 2H), 7.25–7.22 (m, 4H), 7.12 (d, *J* = 4.8 Hz, 1H), 2.38 (s, 3H), 2.16 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.5, 139.0, 137.0, 133.0, 131.1, 131.0, 129.9, 129.7, 128.4, 127.9, 126.7, 18.3, 17.9; HRMS (ESI) *M/Z* calcd for C₁₆H₁₅NO₃Na [M+Na⁺] 292.0944, found: 292.0950.

***N*-acetoxy-*N*-(*m*-tolyl)benzamide (4s).** White solid, 72.1 mg, yield 57%. m.p. 70–71 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.53 (d, *J* = 7.8 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 1H), 7.26 (t, *J* = 7.8 Hz, 2H), 7.16 (t, *J* = 8.4 Hz, 2H), 7.07 (t, *J* = 10.2 Hz, 2H), 2.28 (s, 3H), 2.16 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.8, 166.8, 140.4, 139.2, 133.3, 130.9, 129.2, 128.8, 128.6, 128.0, 127.3, 124.0, 21.1, 18.3; HRMS (ESI) *M/Z* calcd for C₁₆H₁₅NO₃Na [M+Na⁺] 292.0944, found: 292.0952.

***N*-acetoxy-*N*-(*p*-tolyl)benzamide (4t).** White solid, 92.4 mg, yield 73%. m.p. 88–89 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.52 (d, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.25 (t, *J* = 7.8 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 7.8 Hz, 2H), 2.30

(s, 3H), 2.17 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 167.8, 166.7, 138.8, 138.0, 133.3, 130.9, 129.8, 128.7, 128.0, 127.2, 21.1, 18.3; HRMS (ESI) M/Z calcd for $\text{C}_{16}\text{H}_{16}\text{NO}_3$ $[\text{M}+\text{H}^+]$ 270.1125, found: 270.1122.

***N*-acetoxy-*N*-(2-chlorophenyl)benzamide (4u).** White solid, 88.5 mg, yield 65%. m.p. 80–81 °C; ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.56 (d, $J = 7.8$ Hz, 2H), 7.51 (d, $J = 7.2$ Hz, 1H), 7.42 (d, $J = 7.8$ Hz, 1H), 7.36 (t, $J = 7.2$ Hz, 1H), 7.29–7.26 (m, 3H), 7.22 (t, $J = 7.8$ Hz, 1H), 2.19 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 167.7, 137.9, 133.8, 132.9, 132.2, 131.2, 131.0, 130.6, 128.5, 128.1, 127.6, 18.4; HRMS (ESI) M/Z calcd for $\text{C}_{15}\text{H}_{13}\text{ClNO}_3$ $[\text{M}+\text{H}^+]$ 290.0578, found: 290.0580.

***N*-acetoxy-*N*-(3-chlorophenyl)benzamide (4v).** Yellow liquid, 102.1 mg, yield 75%. ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.55–7.53 (m, 2H), 7.42–7.39 (m, 2H), 7.32 (t, $J = 7.8$ Hz, 2H), 7.24–7.19 (m, 3H), 2.16 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 167.8, 166.9, 141.5, 134.6, 132.9, 131.3, 129.9, 128.5, 128.2, 128.1, 125.9, 124.3, 18.3; HRMS (ESI) M/Z calcd for $\text{C}_{15}\text{H}_{13}\text{ClNO}_3$ $[\text{M}+\text{H}^+]$ 290.0578, found: 290.0577.

***N*-acetoxy-*N*-(4-chlorophenyl)benzamide (4w).** White solid, 61.3 mg, yield 45%. m.p. 108–109 °C; ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.53–7.52 (m, 2H), 7.41–7.38 (m, 1H), 7.32–7.26 (m, 6H), 2.16 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 167.8, 166.8, 139.0, 134.0, 133.0, 131.2, 129.3, 128.6, 128.2, 127.8, 18.3; HRMS (ESI) M/Z calcd for $\text{C}_{15}\text{H}_{12}\text{ClNO}_3\text{Na}$ $[\text{M}+\text{Na}^+]$ 312.0398, found: 312.0403.

***N*-acetoxy-*N*-(4-bromophenyl)benzamide (4x).** White solid, 108.4 mg, yield 69%. m.p. 66–67 °C; ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.52 (d, $J = 7.2$ Hz, 2H), 7.44–7.39 (m, 3H), 7.31 (t, $J = 7.8$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 2.16 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 167.8, 166.8, 139.5, 132.9, 132.3, 131.3, 128.6, 128.2, 128.0, 122.0, 18.3; HRMS (ESI) M/Z calcd for $\text{C}_{15}\text{H}_{12}\text{BrNO}_3\text{Na}$ $[\text{M}+\text{Na}^+]$ 355.9893, found: 355.9905.

***N*-acetoxy-*N*-(4-acetylphenyl)benzamide (4y).** White solid, 86.6 mg, yield 62%. m.p. 116–117 °C; ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.90 (d, $J = 8.4$ Hz, 2H), 7.56 (d, $J = 7.2$ Hz, 2H), 7.45–7.39 (m, 3H), 7.34 (t, $J = 7.2$ Hz, 2H), 2.57 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (150 MHz, CDCl_3) δ (ppm) 196.6, 167.8, 166.9, 144.1, 135.4, 133.0, 131.5,

129.2, 128.5, 128.3, 124.2, 26.5, 18.2; HRMS (ESI) M/Z calcd for C₁₇H₁₅NO₄Na [M+Na⁺] 320.0893, found: 320.0901.

Ethyl 4-(*N*-acetoxybenzamido)benzoate (4z). White solid, 93.8 mg, yield 61%. m.p. 72–73 °C; ¹H NMR (600 MHz, CDCl₃) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 7.8 Hz, 2H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 2H), 4.35 (q, *J* = 7.2 Hz, 2H), 2.17 (s, 3H), 1.37 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 167.8, 166.8, 165.5, 144.1, 133.1, 131.4, 130.4, 129.2, 128.6, 128.3, 124.4, 61.2, 18.2, 14.2; HRMS (ESI) M/Z calcd for C₁₈H₁₈NO₅ [M+H⁺] 328.1179, found: 328.1176.

***N*-acetoxy-*N*-(*p*-tolyl)-4-(trifluoromethyl)benzamide (4ac).** White solid, 152.2 mg, yield 96%. m.p. 67–68 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.63 (d, *J* = 7.8 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 2.32 (s, 3H), 2.19 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.7, 165.3, 139.4, 137.3, 136.9, 132.5 (q, *J*_{C-F} = 33.0 Hz), 130.0, 129.1, 127.3, 125.1 (q, *J*_{C-F} = 3.0 Hz), 123.5 (q, *J*_{C-F} = 271.5 Hz), 21.1, 18.3; HRMS (ESI) M/Z calcd for C₁₇H₁₅F₃NO₃ [M+H⁺] 338.0999, found: 338.0998.

***N*-acetoxy-*N*-(4-chlorophenyl)-4-(trifluoromethyl)benzamide (4ad).** Yellow oil, 139.3 mg, yield 83%. ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.65 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 7.8 Hz, 2H), 7.33–7.27 (m, 4H), 2.17 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.7, 165.5, 138.3, 136.5, 134.6, 132.9 (q, *J*_{C-F} = 33.0 Hz), 129.6, 129.0, 127.9, 125.3 (q, *J*_{C-F} = 4.5 Hz), 123.4 (q, *J*_{C-F} = 271.5 Hz), 18.2; HRMS (ESI) M/Z calcd for C₁₆H₁₁ClF₃NO₃Na [M+Na⁺] 380.0272, found: 380.0278.

***N*-acetoxy-*N*-(4-chlorophenyl)-2-fluorobenzamide (4ae).** White solid, 112.8 mg, yield 78%. m.p. 98–99 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.48 (t, *J* = 7.2 Hz, 1H), 7.37–7.27 (m, 5H), 7.14 (t, *J* = 7.2 Hz, 1H), 6.98 (s, 1H), 2.12 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.5, 158.4 (d, *J*_{C-F} = 250.5 Hz), 137.5, 134.3, 132.5 (d, *J*_{C-F} = 7.5 Hz), 129.6, 129.1, 127.9, 124.3 (d, *J*_{C-F} = 3.0 Hz), 122.3 (d, *J*_{C-F} = 15.0 Hz), 115.8 (d, *J*_{C-F} = 21.0 Hz), 18.0; HRMS (ESI) M/Z calcd for C₁₅H₁₁ClFNO₃Na [M+Na⁺] 330.0304, found: 330.0303.

***N*-acetoxy-2-chloro-*N*-(4-chlorophenyl)benzamide (4af).** White solid, 106.6 mg,

yield 70%. m.p. 72–73 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.38–7.27 (m, 8H), 2.25 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.6, 137.3, 133.8, 131.1, 129.7, 129.3, 126.7, 18.1; HRMS (ESI) M/Z calcd for C₁₅H₁₅Cl₂N₂O₃ [M+NH₄⁺] 341.0454, found: 341.0453.

***N*-acetoxy-4-chloro-*N*-(4-chlorophenyl)benzamide (4ag).** White solid, 114.27 mg, yield 75%. m.p. 75–77 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.47 (d, *J* = 7.8 Hz, 2H), 7.31–7.25 (m, 6H), 2.17 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 166.8, 140.6, 133.2, 131.0, 129.2, 128.8, 128.4, 128.1, 126.9, 18.4; HRMS (ESI) M/Z calcd for C₁₅H₁₁Cl₂NO₃Na [M+Na⁺] 346.0008, found: 346.0012.

***N*-acetoxy-4-bromo-*N*-(4-chlorophenyl)benzamide (4ah).** White solid, 128.2 mg, yield 74%. m.p. 70–71 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.45 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 9.0 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 2.17 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.7, 165.9, 138.7, 134.4, 131.8, 131.5, 130.2, 129.5, 127.9, 126.0, 18.3; HRMS (ESI) M/Z calcd for C₁₅H₁₁BrClNO₃Na [M+Na⁺] 389.9503, found: 389.9499.

***N*-acetoxy-*N*-(4-chlorophenyl)-4-nitrobenzamide (4ai).** White solid, 114.8 mg, yield 73%. m.p. 166–167 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 8.18 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.34–7.27 (m, 4H), 2.18 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.6, 149.1, 139.0, 137.9, 135.0, 129.7, 128.0, 123.5, 109.9, 18.2; HRMS (ESI) M/Z calcd for C₁₅H₁₁ClN₂O₅Na [M+Na⁺] 357.0249, found: 357.0246.

***N*-acetoxy-*N*-(*p*-tolyl)furan-2-carboxamide (4aj).** White solid, 81.6 mg, yield 67%. m.p. 107–108 °C; ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.42 (d, *J* = 1.2 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 6.55 (s, 1H), 6.36 (dd, *J* = 3.6, 1.2 Hz, 1H), 2.40 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) 167.9, 156.2, 145.4, 145.2, 139.8, 137.3, 130.0, 127.8, 118.0, 111.4, 21.3, 18.5; HRMS (ESI) M/Z calcd for C₁₄H₁₄NO₄ [M+H⁺] 260.0917, found: 260.0914.

***N*-acetoxy-*N*-(4-chlorophenyl)-4-methoxybenzamide (4ak).** Yellow oil, 37.5 mg, yield 25%. ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.50 (d, *J* = 9.0 Hz, 2H), 7.30–7.28 (m, 2H), 7.25–7.24 (m, 2H), 6.79 (d, *J* = 9.0 Hz, 2H), 3.80 (s, 3H), 2.19 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 162.1, 134.0, 131.0, 129.4, 128.0, 124.7,

113.5, 110.0, 55.3, 18.4; HRMS (ESI) M/Z calcd for C₁₆H₁₄ClNO₄Na [M+Na⁺] 342.0604, found: 342.0608.

***N*-acetoxy-*N*-(4-chlorophenyl)-4-methylbenzamide (4a1).** yellow oil, 44.1 mg, yield 31%. ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.42 (d, *J* = 8.4 Hz, 2H), 7.29–7.24 (m, 4H), 7.10 (d, *J* = 7.8 Hz, 2H), 2.33 (s, 3H), 2.18 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 167.9, 166.9, 141.9, 139.4, 134.0, 130.0, 129.4, 128.9, 128.8, 128.0, 21.5, 18.4; HRMS (ESI) M/Z calcd for C₁₆H₁₄ClNO₃Na [M+Na⁺] 326.0554, found: 326.0558.

General Procedure for the Synthesis of Compounds 5.

Hydrated barium hydroxide (1.47 mmol, 7 equiv) was added to a solution of the *N*-acetoxy-2-chloro-*N*-phenylbenzamide **4g** in ethanol (7 ml) under nitrogen atmosphere and the mixture was stirred at room temperature for 1.5 h. Then, hydrochloric acid was added to acidify the mixture to pH 1. The resulting mixture was extracted with EtOAc (3 × 10 mL). The organic layer were combined, dried over anhydrous MgSO₄ and then concentrated under reduced pressure. The residue was purified by silica gel column chromatography using PE : EA (5:3) as eluent to afford the product **5**.

2-chloro-*N*-hydroxy-*N*-phenylbenzamide (5). White solid, 38.0 mg, yield 73%. m.p. 103–104 °C; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 9.46 (s, 1H), 7.29–7.21 (m, 9H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 162.5, 137.8, 133.1, 131.6, 131.2, 129.9, 129.4, 128.8, 128.2, 126.7, 125.1.

General Procedure for the Synthesis of Compounds 6.

To a stirring solution of the *N*-acetoxy-2-chloro-*N*-phenylbenzamide **4g** (0.21 mmol, 1 equiv) in dry THF (5 mL), maintained under nitrogen atmosphere at room temperature, was added a freshly prepared solution of SmI₂ in THF dropwise. After TLC analysis indicated complete reaction, the mixture was diluted with CH₂Cl₂ (10 mL), then quenched with 10 ml of 10% aqueous sodium thiosulfate solution. The resulting mixture was extracted with CH₂Cl₂ (3 × 10 mL). The organic layer were combined, dried over anhydrous MgSO₄ and then concentrated under reduced pressure. The residue was purified by silica gel column chromatography using PE :

EA (100:1) as eluent to afford the product **6**.

2-chloro-N-phenylbenzamide (6). White solid, 46.2 mg, yield 95%. m.p. 116–118 °C; ¹H NMR (400 MHz, CDCl₃) δ (ppm) 8.02 (s, 1H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.44–7.32 (m, 5H), 7.17 (t, *J* = 7.6 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ (ppm) 164.5, 137.5, 135.2, 131.6, 130.6, 130.3, 130.2, 129.1, 127.2, 124.8, 120.1.

Figure S1. Diffraction Structure of 4l; thermal ellipsoids are set at a 50% probability level

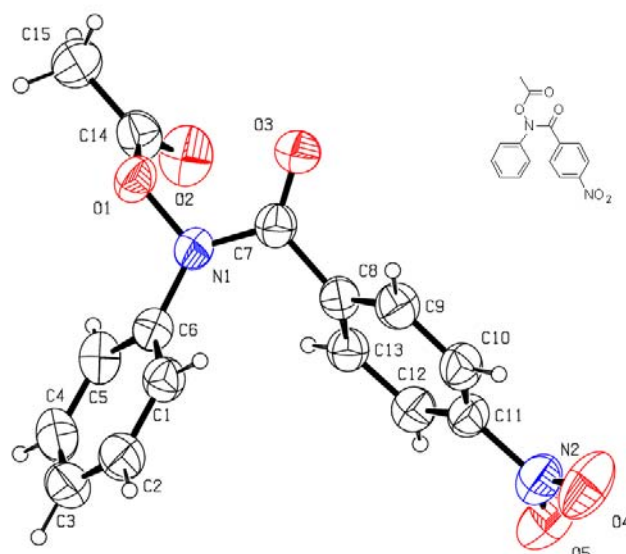


Table S1 Crystal data and structure refinement for 4l

Identification code	Xray_0827
Empirical formula	C ₁₅ H ₁₂ N ₂ O ₅
Formula weight	300.27
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	Pna2 ₁
a/Å	10.558(2)
b/Å	16.971(4)
c/Å	8.182(3)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	1466.1(7)
Z	4
ρ _{calc} /cm ³	1.360
μ/mm ⁻¹	0.879
F(000)	624.0
Crystal size/mm ³	0.33 × 0.27 × 0.25
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	10.424 to 139.77
Index ranges	-12 ≤ h ≤ 8, -20 ≤ k ≤ 19, -9 ≤ l ≤ 9
Reflections collected	4142
Independent reflections	2257 [R _{int} = 0.0359, R _{sigma} = 0.0462]

Data/restraints/parameters	2257/1/200
Goodness-of-fit on F^2	0.976
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0564$, $wR_2 = 0.1397$
Final R indexes [all data]	$R_1 = 0.1024$, $wR_2 = 0.1872$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.12/-0.21
Flack parameter	-0.1(4)

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 4l. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{ij} tensor.

Atom	x	y	z	U(eq)
O1	6689(4)	4839(3)	1867(7)	85.0(13)
O2	6347(6)	4929(4)	4579(9)	120(2)
O3	8860(4)	4287(2)	2933(8)	92.6(14)
O4	14029(6)	6694(5)	4029(14)	157(3)
O5	12833(6)	7393(5)	5509(9)	130(2)
N1	7731(4)	5352(3)	2119(8)	81.6(16)
N2	13006(7)	6870(4)	4560(10)	102(2)
C1	8408(6)	6308(4)	94(9)	79.9(17)
C2	8234(8)	7011(5)	-694(11)	98(2)
C3	7241(9)	7503(5)	-280(14)	107(3)
C4	6442(8)	7279(5)	930(12)	99(2)
C5	6605(6)	6580(4)	1766(10)	87.1(18)
C6	7590(5)	6086(4)	1324(9)	75.1(16)
C7	8795(6)	4987(4)	2672(10)	77.4(16)
C8	9869(5)	5535(3)	3069(9)	73.6(15)
C9	11078(6)	5343(4)	2500(10)	85.2(19)
C10	12092(6)	5794(4)	2967(11)	88.1(19)
C11	11902(6)	6400(4)	4018(10)	79.2(17)
C12	10734(6)	6605(4)	4618(10)	82.0(17)
C13	9715(6)	6161(3)	4093(9)	75.6(16)
C14	6051(7)	4663(5)	3306(11)	91(2)
C15	4999(7)	4118(5)	2903(14)	115(3)

Table S3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 4l. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
O1	70(2)	106(3)	79(3)	8(2)	-2(2)	-17(2)
O2	118(4)	159(5)	82(4)	-6(4)	13(3)	1(4)
O3	89(3)	75(2)	113(4)	5(3)	-6(3)	-1(2)
O4	75(3)	207(7)	188(9)	-38(7)	16(5)	-30(4)
O5	127(5)	162(6)	100(5)	-21(5)	6(4)	-54(4)
N1	65(3)	86(3)	94(4)	14(3)	-11(3)	-12(3)
N2	91(5)	127(5)	87(5)	8(4)	-6(4)	-25(4)
C1	79(4)	89(4)	72(4)	2(3)	-3(3)	-2(3)
C2	103(5)	106(5)	87(6)	17(4)	-10(5)	-9(4)
C3	124(6)	86(4)	110(7)	11(5)	-34(6)	1(5)
C4	98(5)	103(5)	96(6)	-15(5)	-23(5)	24(4)
C5	75(4)	111(5)	76(4)	-10(4)	-4(4)	11(4)
C6	69(3)	79(3)	77(4)	-3(3)	-4(3)	-2(3)
C7	70(4)	84(4)	78(4)	0(3)	1(3)	1(3)
C8	69(3)	79(3)	73(4)	4(3)	-4(3)	3(3)
C9	76(4)	85(4)	95(5)	-5(3)	5(4)	10(3)
C10	66(4)	101(4)	97(5)	9(4)	4(4)	1(3)
C11	73(4)	86(4)	80(4)	9(4)	-5(3)	-11(3)
C12	83(4)	88(4)	75(4)	-1(3)	1(3)	-3(3)
C13	72(3)	80(3)	75(4)	0(3)	1(3)	1(3)
C14	82(4)	107(5)	83(5)	20(4)	10(4)	8(4)
C15	81(4)	127(6)	136(8)	36(6)	15(5)	-17(4)

Table S4 Bond Lengths for 4l

Atom	Atom	Length/ \AA	Atom	Atom	Length/ \AA
O1	N1	1.418(6)	C3	C4	1.355(13)
O1	C14	1.389(10)	C4	C5	1.379(12)
O2	C14	1.177(11)	C5	C6	1.383(9)
O3	C7	1.209(7)	C7	C8	1.502(9)
O4	N2	1.202(9)	C8	C9	1.398(9)
O5	N2	1.194(10)	C8	C13	1.363(8)
N1	C6	1.413(8)	C9	C10	1.371(9)
N1	C7	1.360(8)	C10	C11	1.356(11)
N2	C11	1.480(9)	C11	C12	1.372(9)
C1	C2	1.369(10)	C12	C13	1.382(9)
C1	C6	1.379(10)	C14	C15	1.482(11)
C2	C3	1.382(12)			

Table S5 Bond Angles for 4l

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C14	O1	N1	112.7(6)	O3	C7	C8	121.8(6)
C6	N1	O1	113.1(5)	N1	C7	C8	114.4(5)
C7	N1	O1	114.2(5)	C9	C8	C7	118.2(6)
C7	N1	C6	129.9(5)	C13	C8	C7	121.7(6)
O4	N2	C11	117.7(8)	C13	C8	C9	119.7(6)
O5	N2	O4	123.9(8)	C10	C9	C8	119.4(7)
O5	N2	C11	118.3(8)	C11	C10	C9	119.0(6)
C2	C1	C6	119.8(7)	C10	C11	N2	118.8(7)
C1	C2	C3	120.8(8)	C10	C11	C12	123.5(6)
C4	C3	C2	118.8(8)	C12	C11	N2	117.7(7)
C3	C4	C5	121.7(7)	C11	C12	C13	116.8(7)
C4	C5	C6	119.0(8)	C8	C13	C12	121.6(6)
C1	C6	N1	120.7(6)	O1	C14	C15	108.0(8)
C1	C6	C5	119.8(7)	O2	C14	O1	122.6(7)
C5	C6	N1	119.5(6)	O2	C14	C15	129.4(8)
O3	C7	N1	123.6(6)				

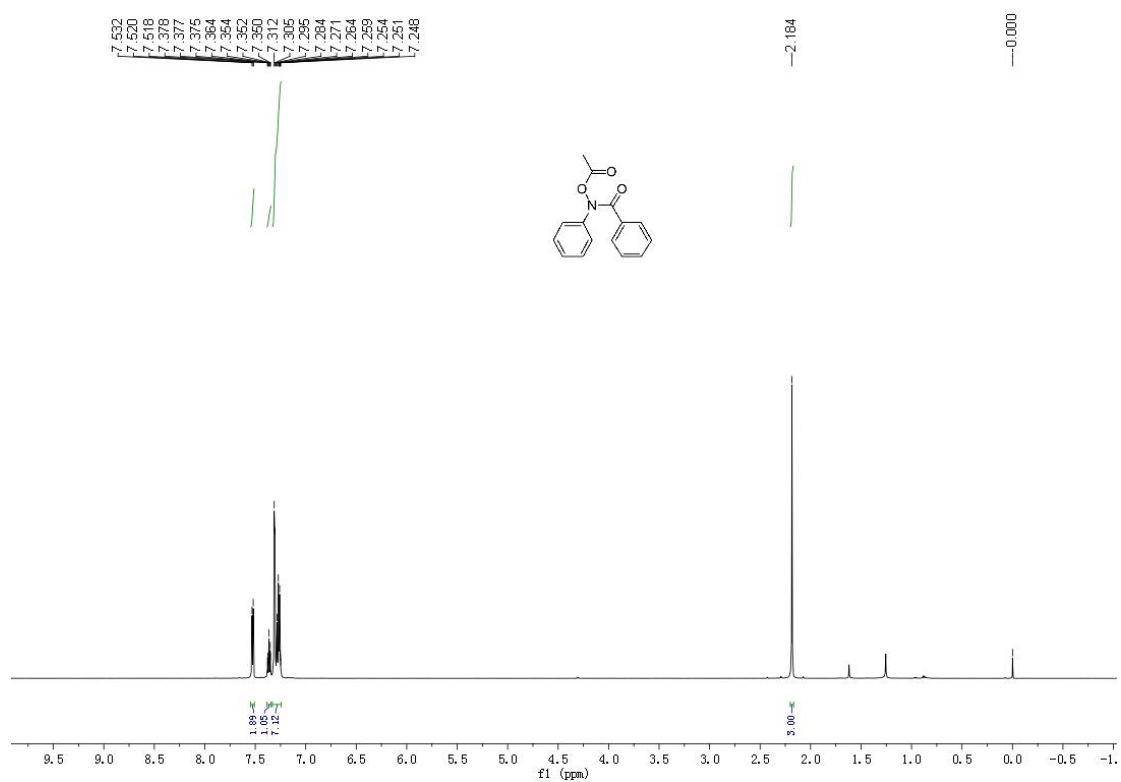
Table S6 Torsion Angles for 4l

A	B	C	D	Angle/°	A	B	C	D	Angle/°
O1	N1	C6	C1	-116.0(7)	C3	C4	C5	C6	-1.7(12)
O1	N1	C6	C5	62.6(8)	C4	C5	C6	N1	-177.1(7)
O1	N1	C7	O3	-0.1(11)	C4	C5	C6	C1	1.6(11)
O1	N1	C7	C8	-175.3(6)	C6	N1	C7	O3	-159.5(7)
O3	C7	C8	C9	50.9(11)	C6	N1	C7	C8	25.2(11)
O3	C7	C8	C13	-122.4(8)	C6	C1	C2	C3	-0.4(12)
O4	N2	C11	C10	0.5(11)	C7	N1	C6	C1	43.6(11)
O4	N2	C11	C12	-179.6(9)	C7	N1	C6	C5	-137.7(8)
O5	N2	C11	C10	-178.6(8)	C7	C8	C9	C10	-174.2(7)
O5	N2	C11	C12	1.2(11)	C7	C8	C13	C12	171.8(7)
N1	O1	C14	O2	1.4(10)	C8	C9	C10	C11	2.3(11)
N1	O1	C14	C15	-179.2(5)	C9	C8	C13	C12	-1.4(10)
N1	C7	C8	C9	-133.7(7)	C9	C10	C11	N2	178.1(7)
N1	C7	C8	C13	53.0(9)	C9	C10	C11	C12	-1.8(11)
N2	C11	C12	C13	179.8(6)	C10	C11	C12	C13	-0.4(11)
C1	C2	C3	C4	0.3(13)	C11	C12	C13	C8	2.0(10)
C2	C1	C6	N1	178.1(7)	C13	C8	C9	C10	-0.8(11)
C2	C1	C6	C5	-0.6(11)	C14	O1	N1	C6	-116.4(6)
C2	C3	C4	C5	0.7(13)	C14	O1	N1	C7	80.6(7)

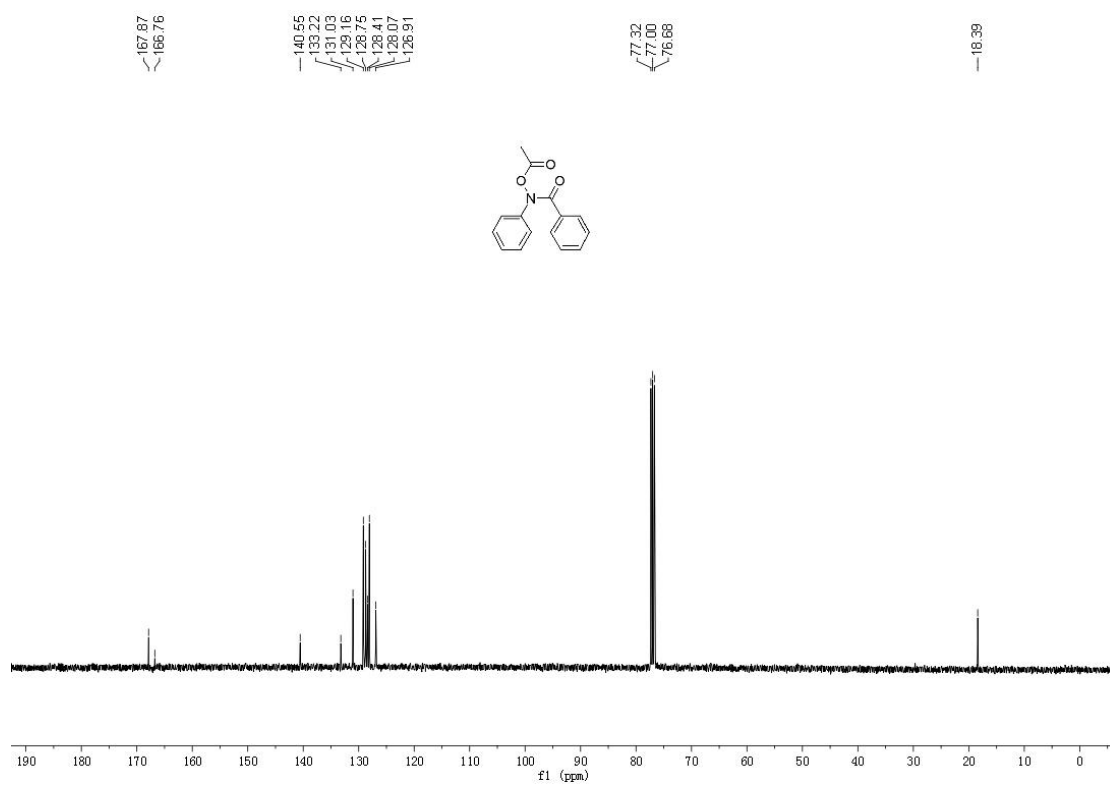
Table S7 Hydrogen Atom Coordinates ($\text{\AA}\times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2\times 10^3$) for 4l

Atom	x	y	z	U(eq)
H1	9077	5981	-200	96
H2	8791	7160	-1519	118
H3	7123	7979	-822	128
H4	5767	7604	1205	119
H5	6061	6442	2612	105
H9	11194	4913	1811	102
H10	12899	5686	2569	106
H12	10633	7023	5343	98
H13	8905	6293	4447	91
H15A	4490	4343	2051	172
H15B	4487	4035	3857	172
H15C	5340	3623	2541	172

¹H NMR copies of compound **4a**:



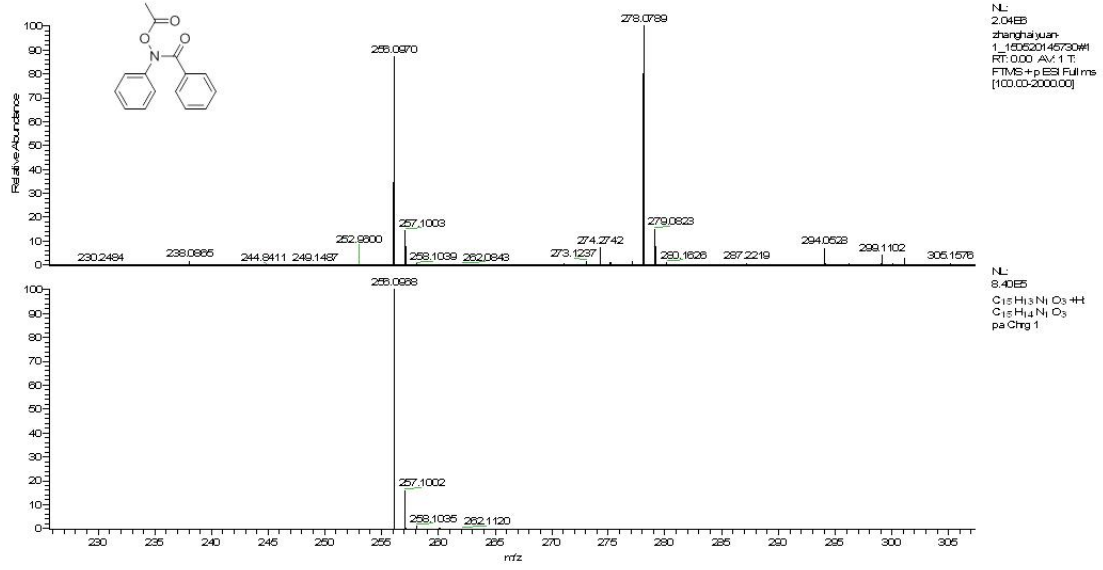
¹³C NMR copies of compound **4a**:



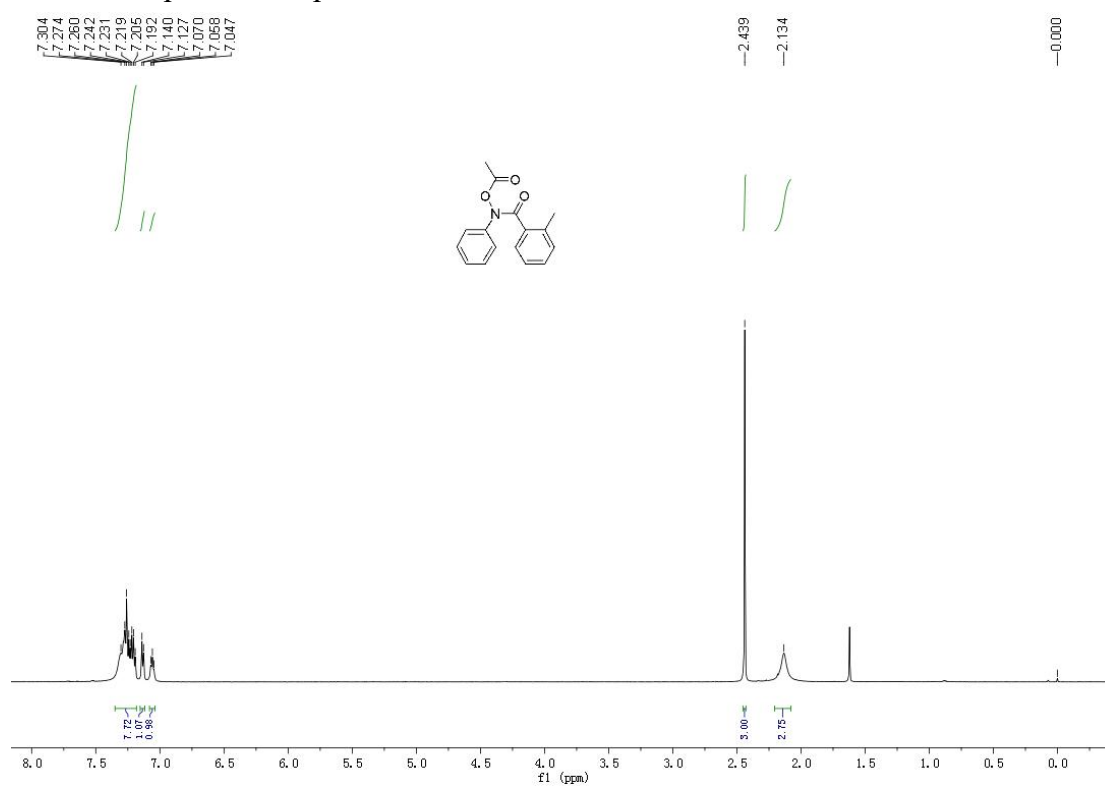
HRMS (ESI) of compound **4a**:

F:\User\zhanghaijun_1\0520145730

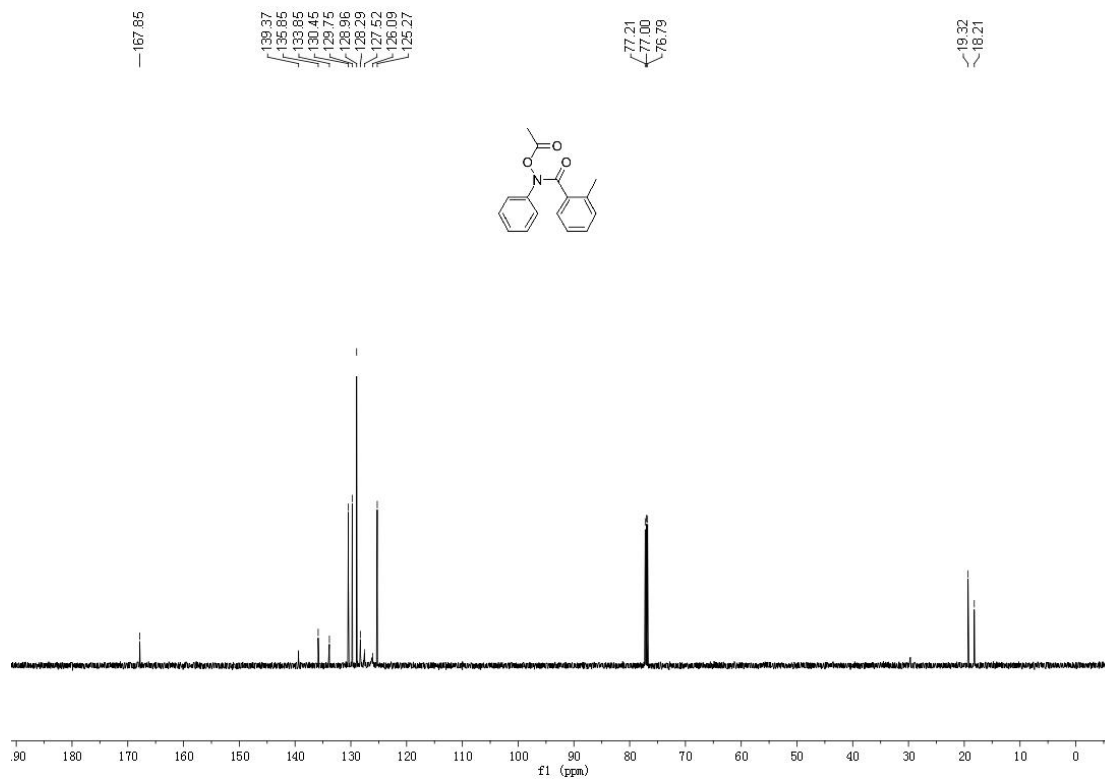
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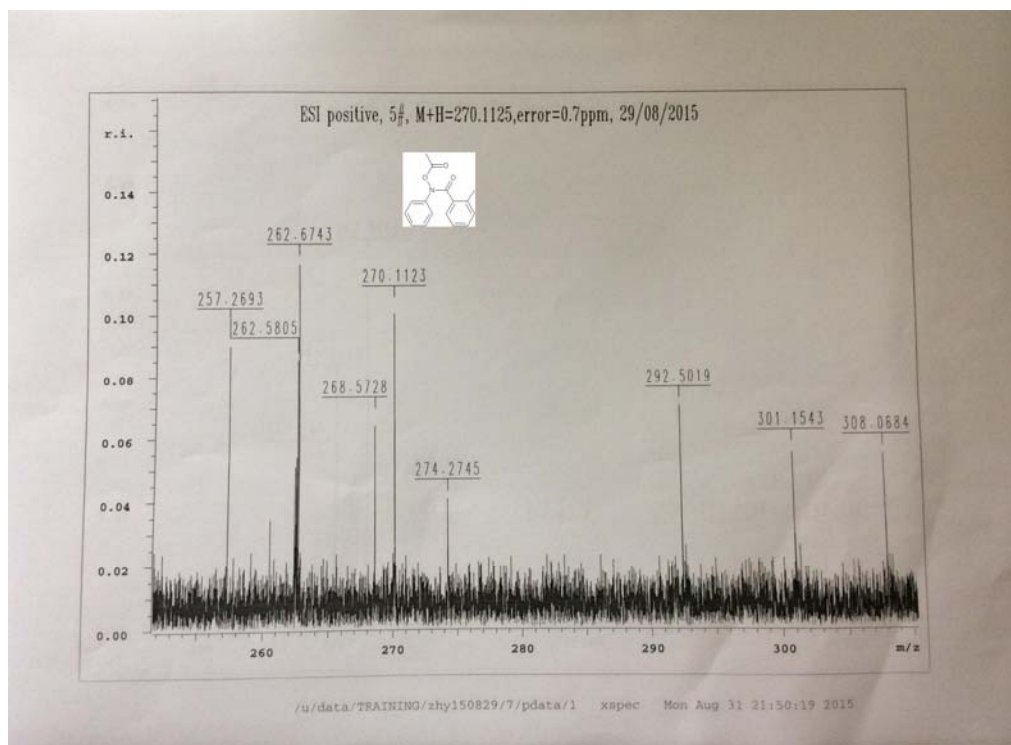
¹H NMR copies of compound **4b**:



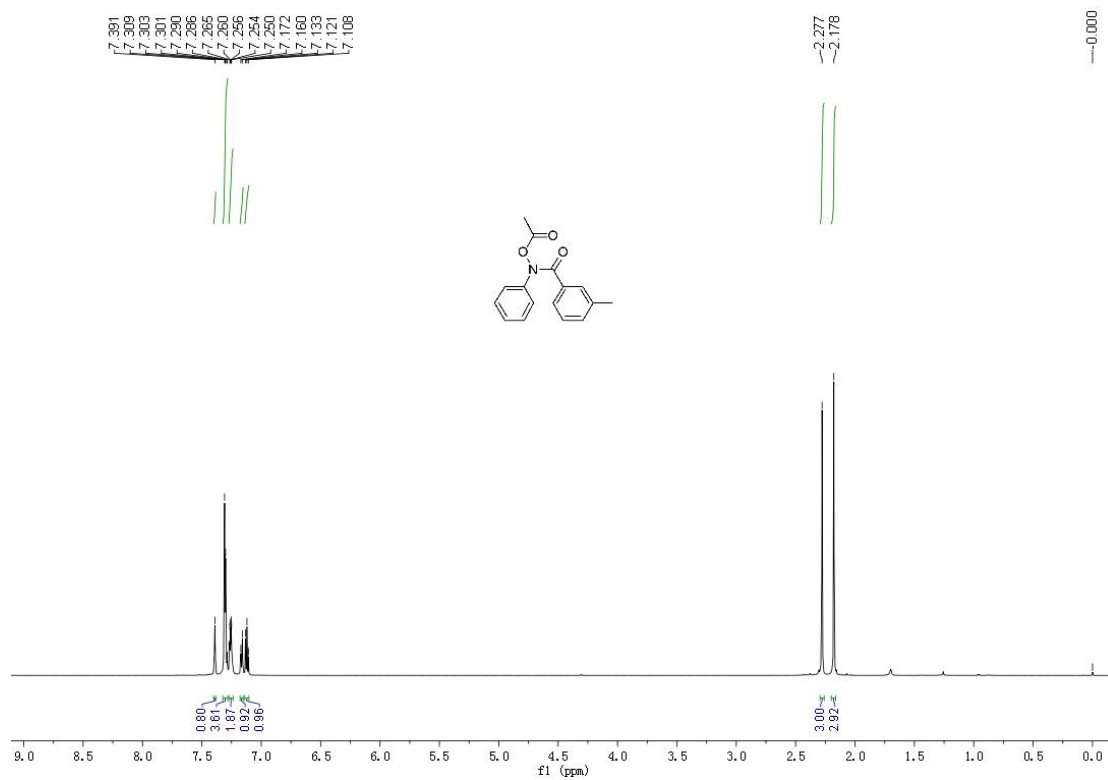
¹³C NMR copies of compound **4b**:



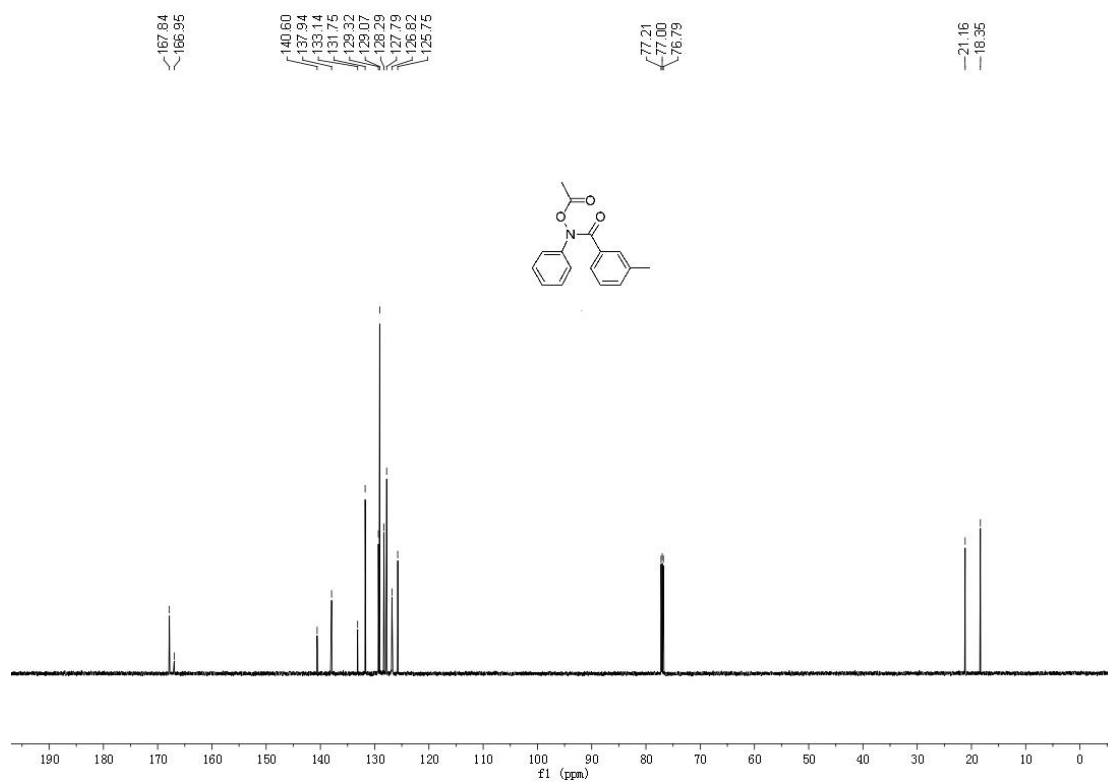
HRMS (ESI) of compound **4b**:



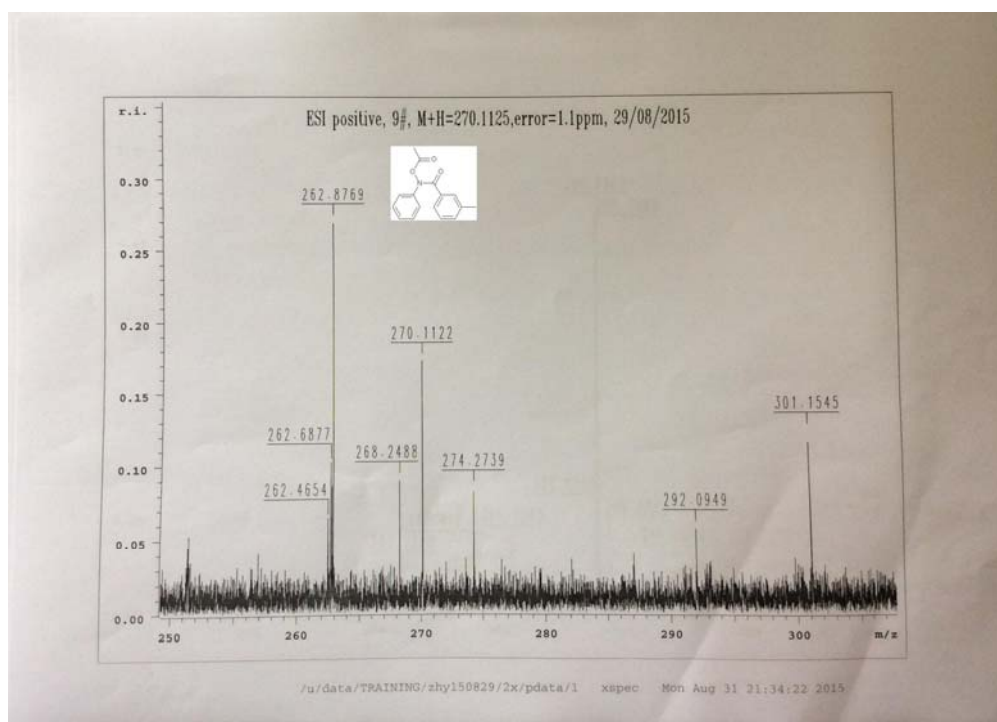
¹H NMR copies of compound 4c:



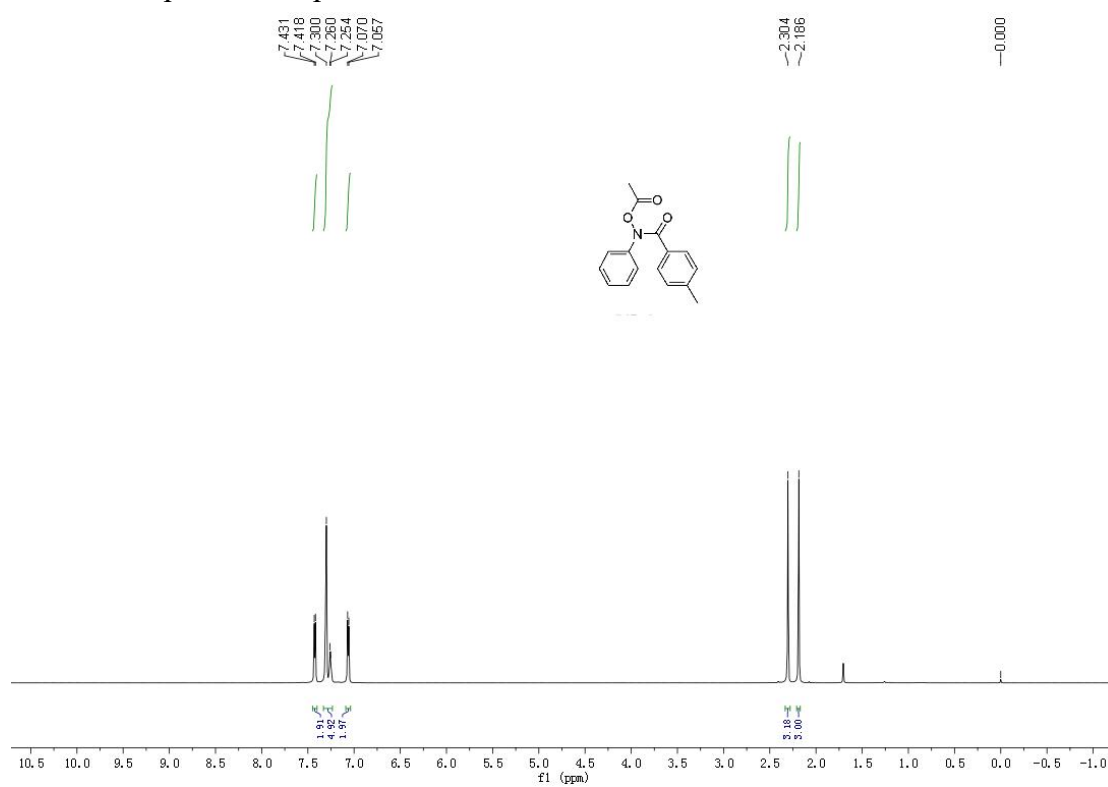
¹³C NMR copies of compound 4c:



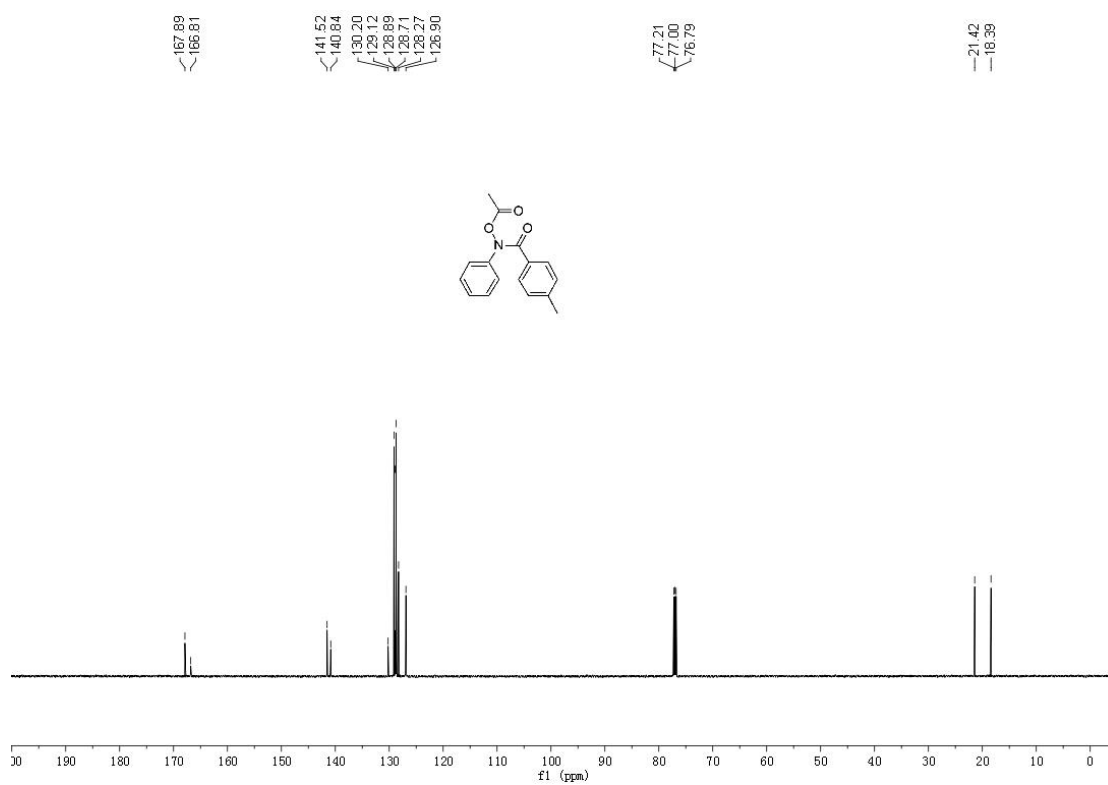
HRMS (ESI) of compound **4c**:



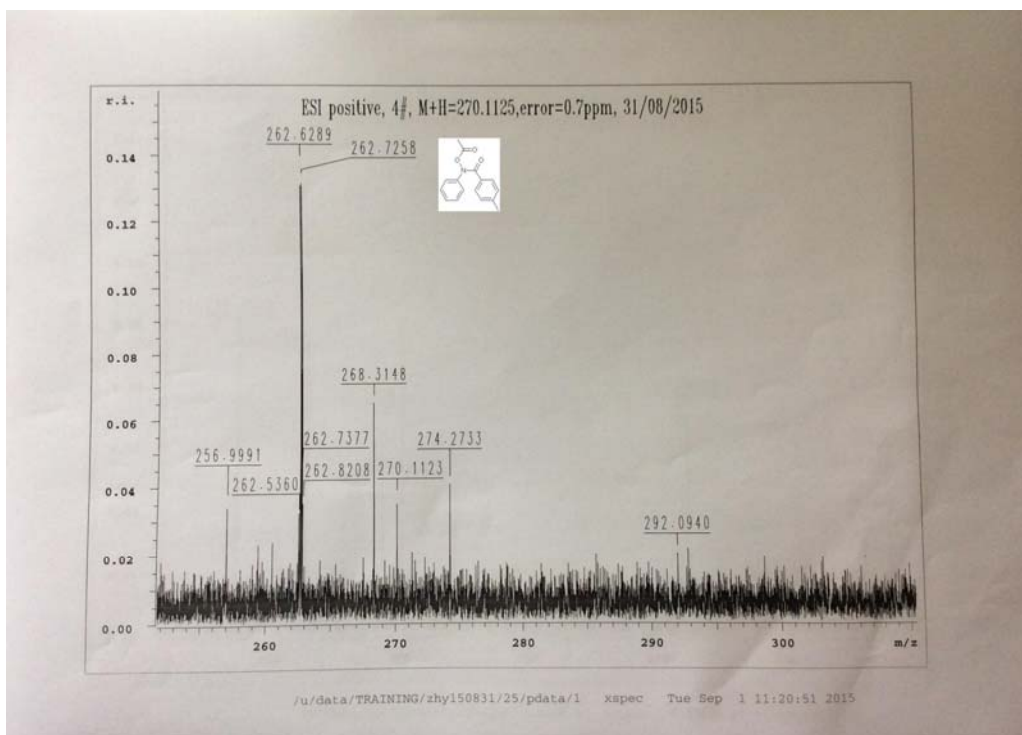
¹H NMR copies of compound **4d**:



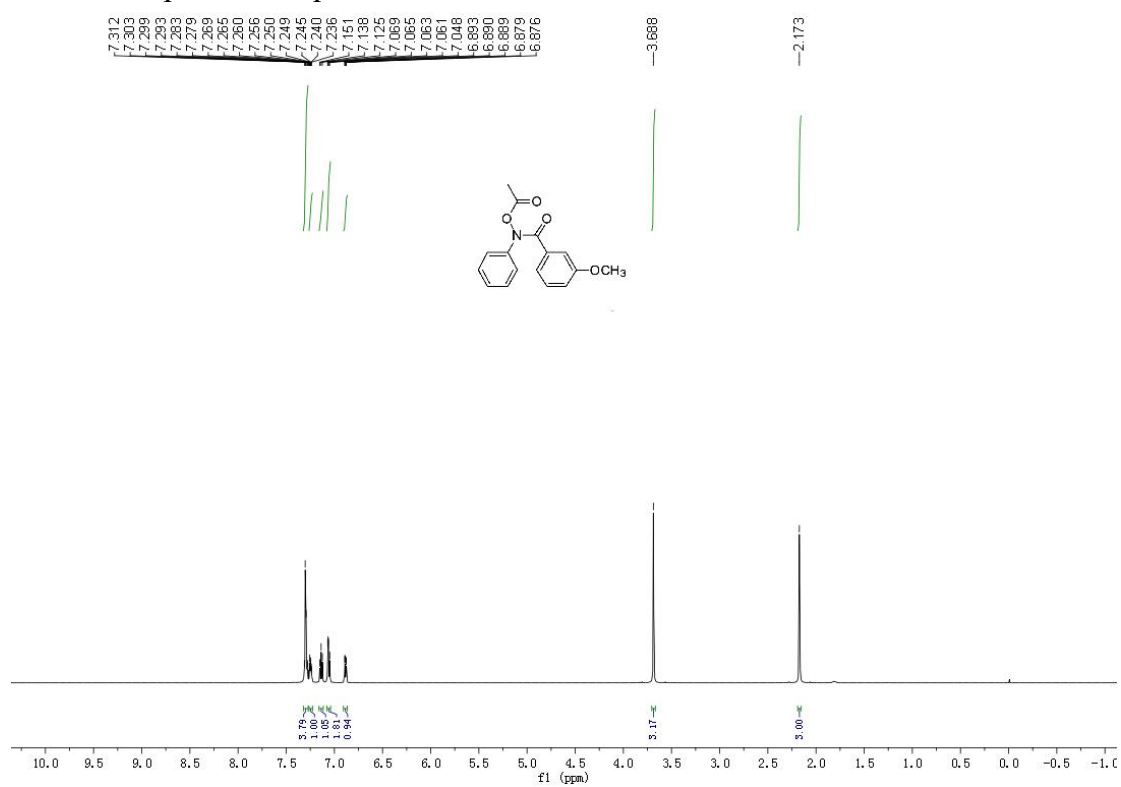
¹³C NMR copies of compound **4d**:



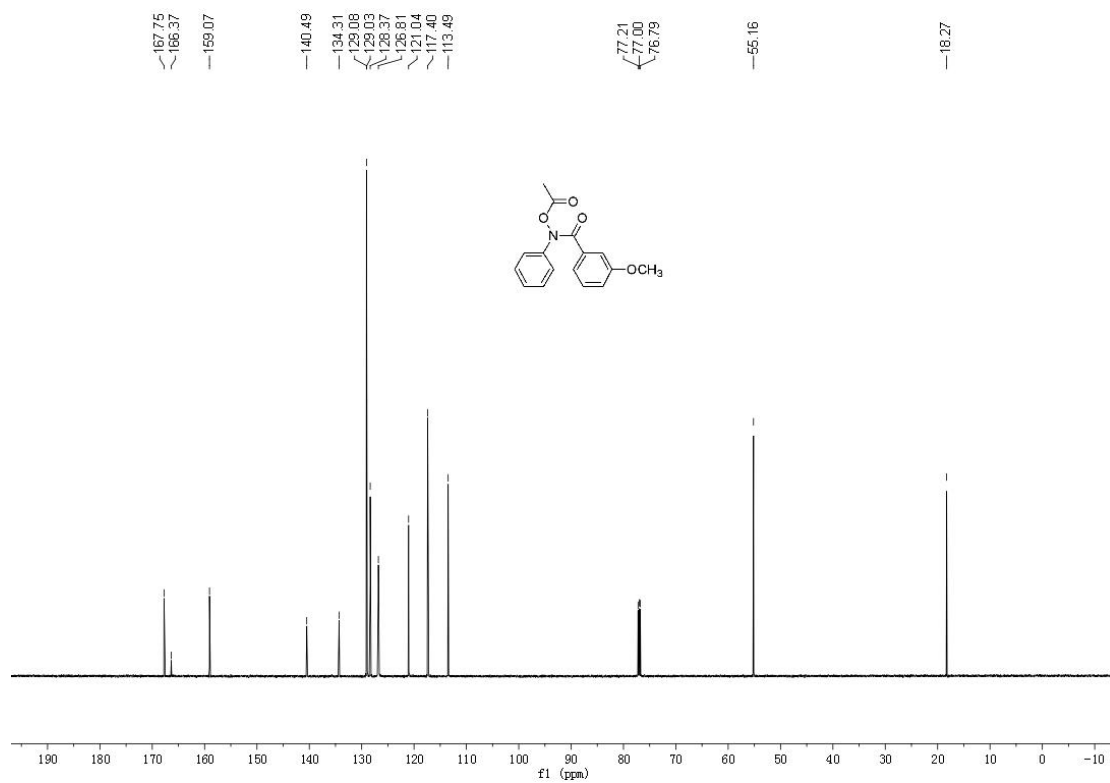
HRMS (ESI) of compound **4d**:

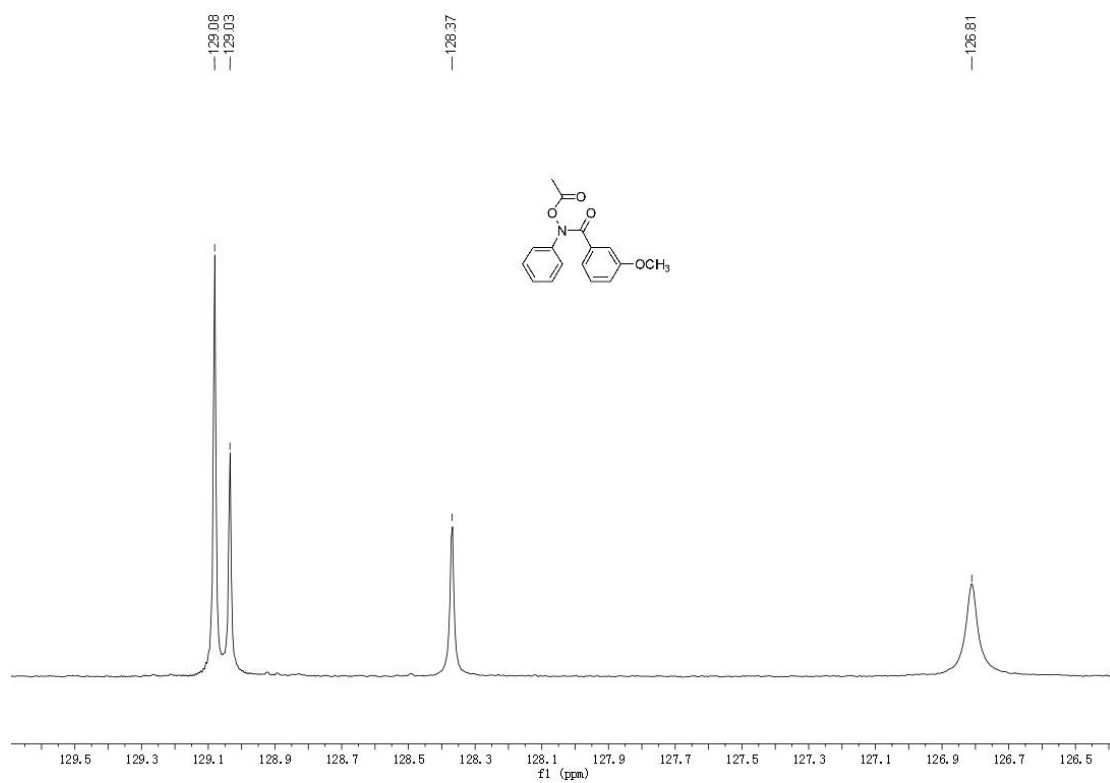


¹H NMR copies of compound 4e:

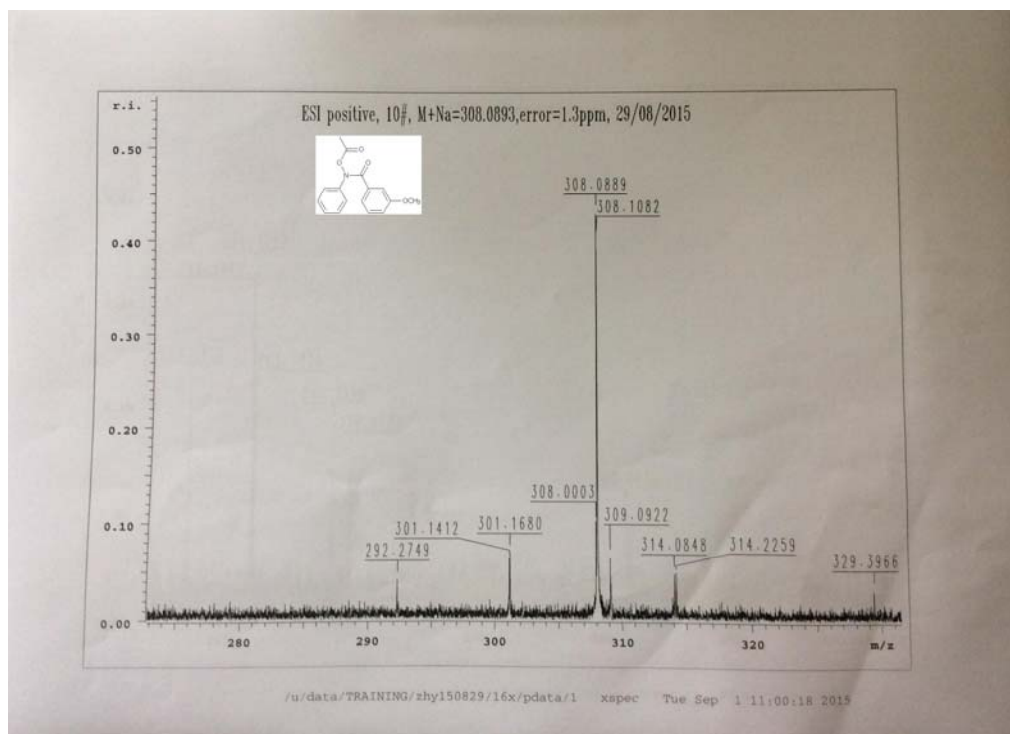


¹³C NMR copies of compound 4e:

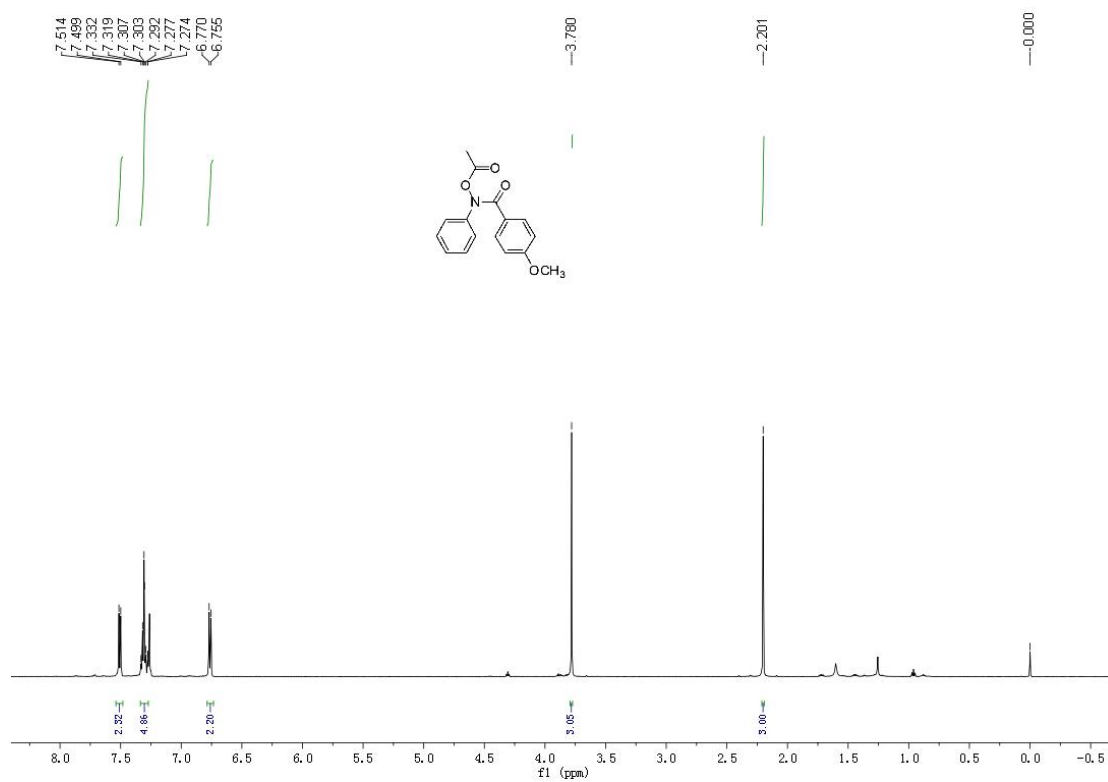




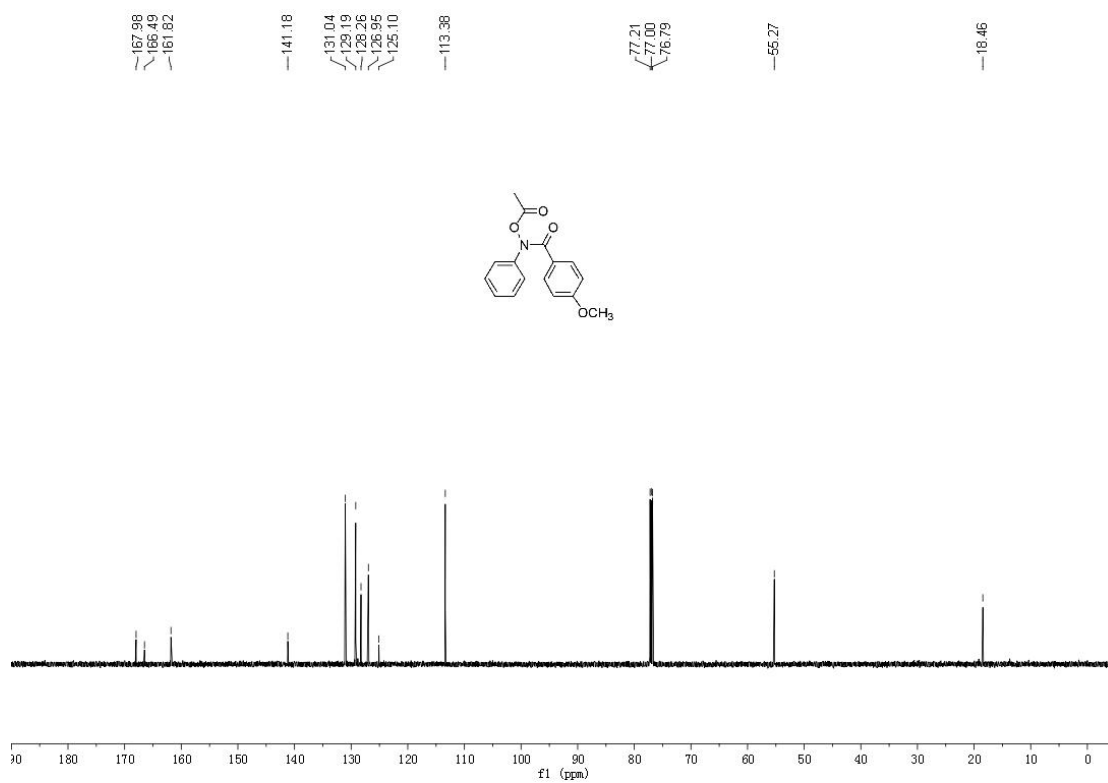
HRMS (ESI) of compound 4e:



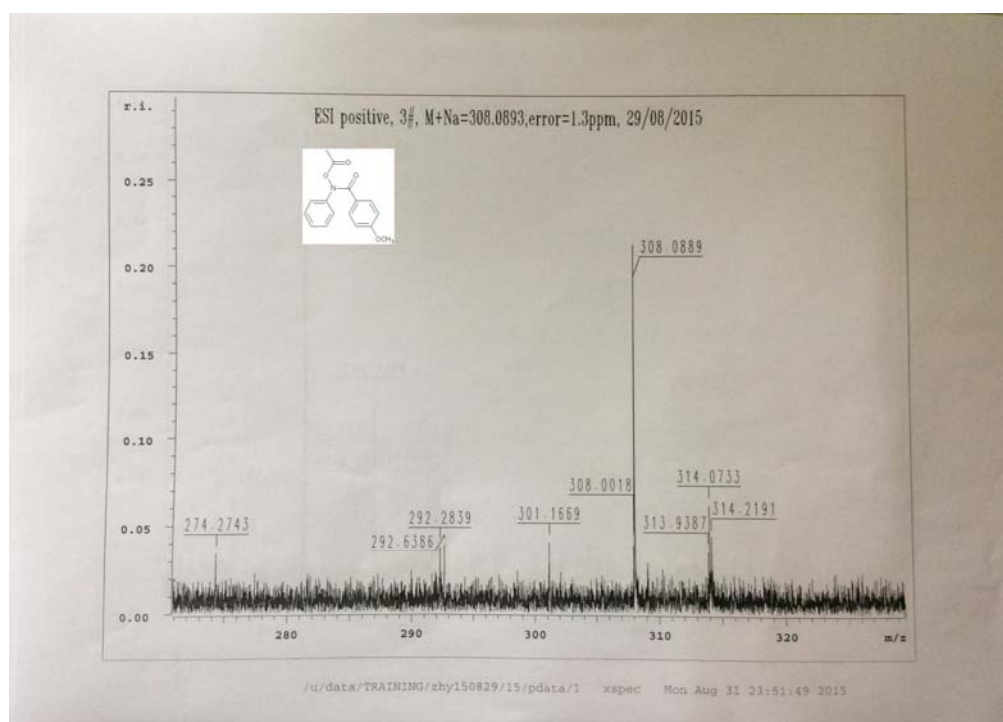
¹H NMR copies of compound **4f**:



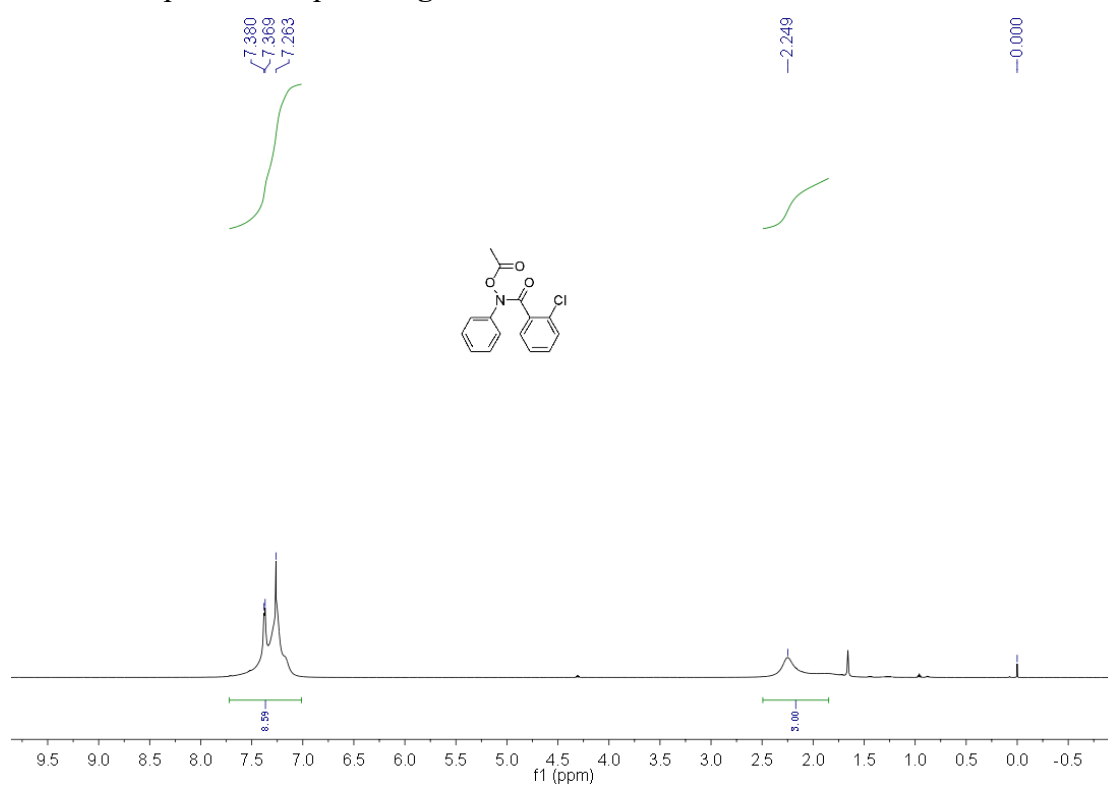
¹³C NMR copies of compound **4f**:



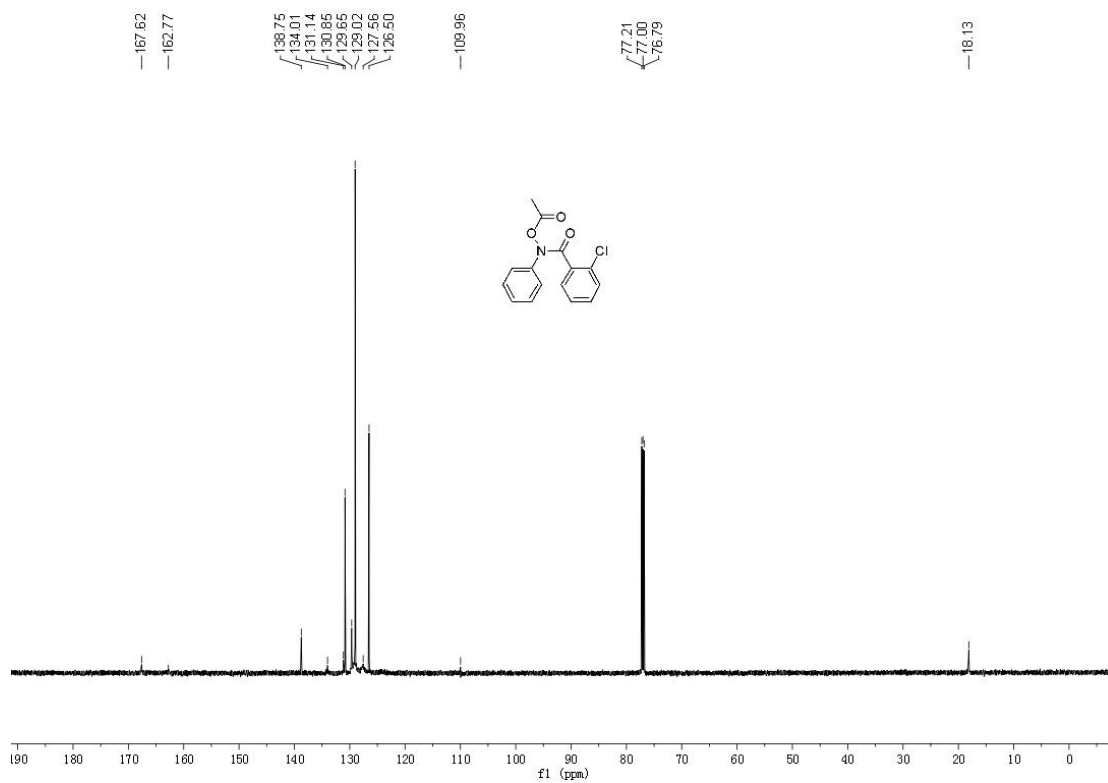
HRMS (ESI) of compound **4f**:



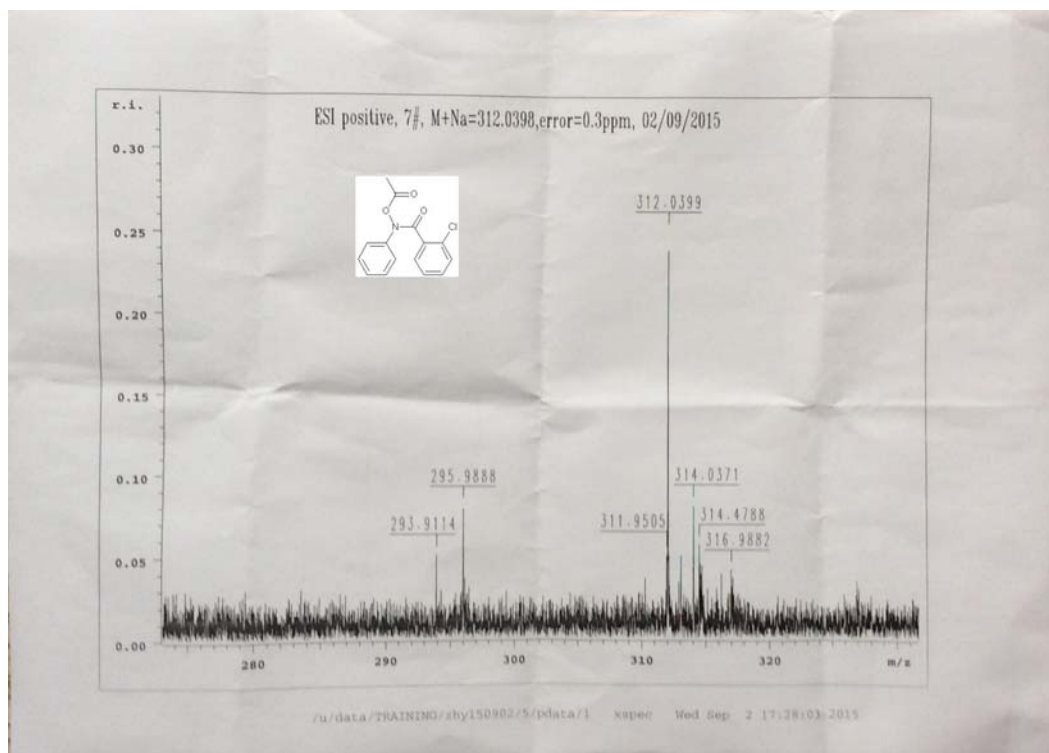
¹H NMR copies of compound **4g**:



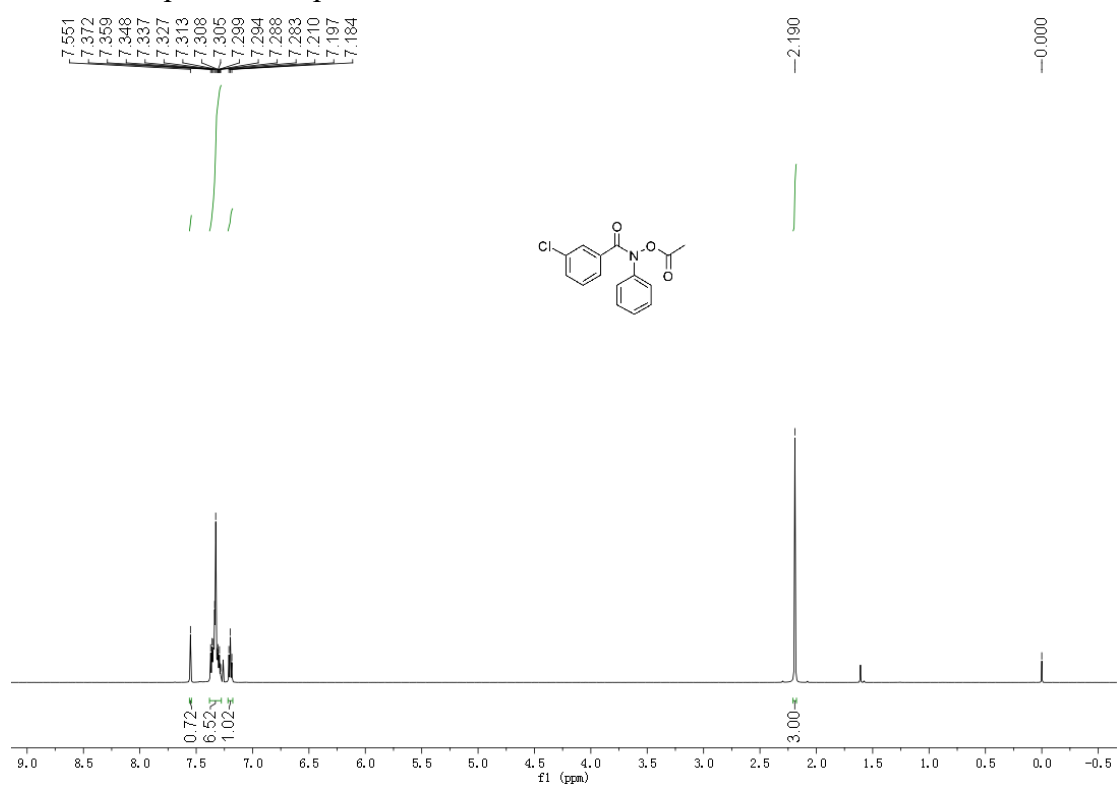
¹³C NMR copies of compound **4g**:



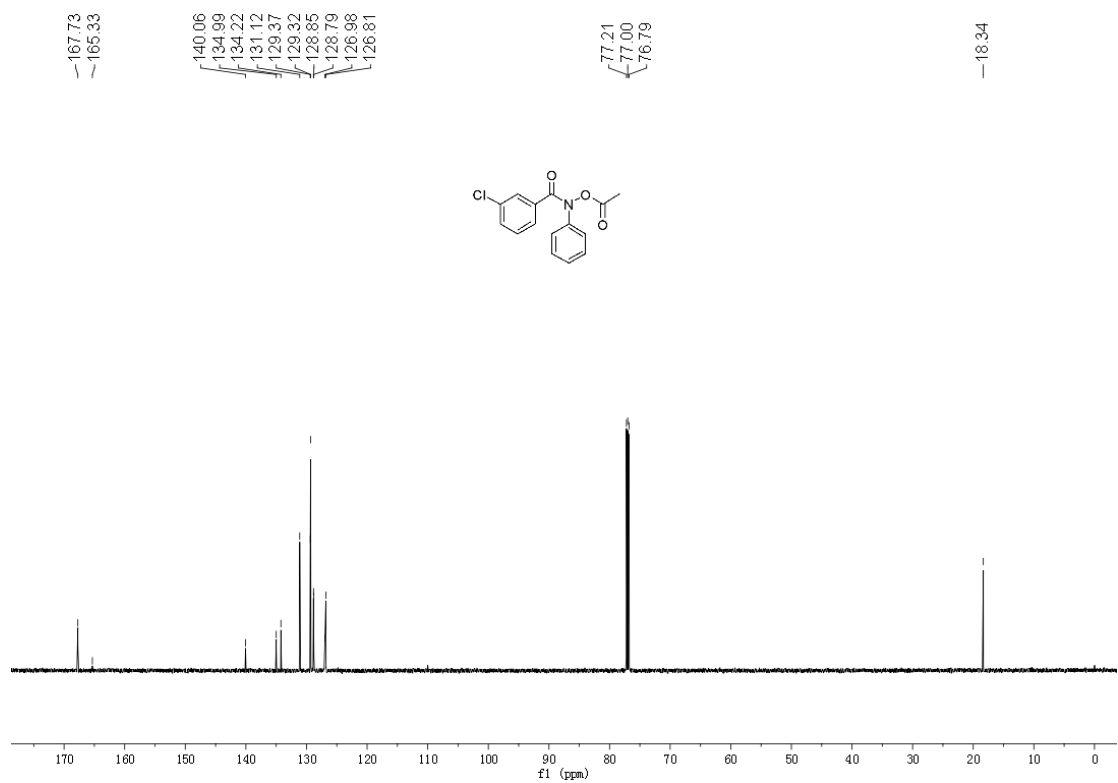
HRMS (ESI) of compound **4g**:



¹H NMR copies of compound 4h:



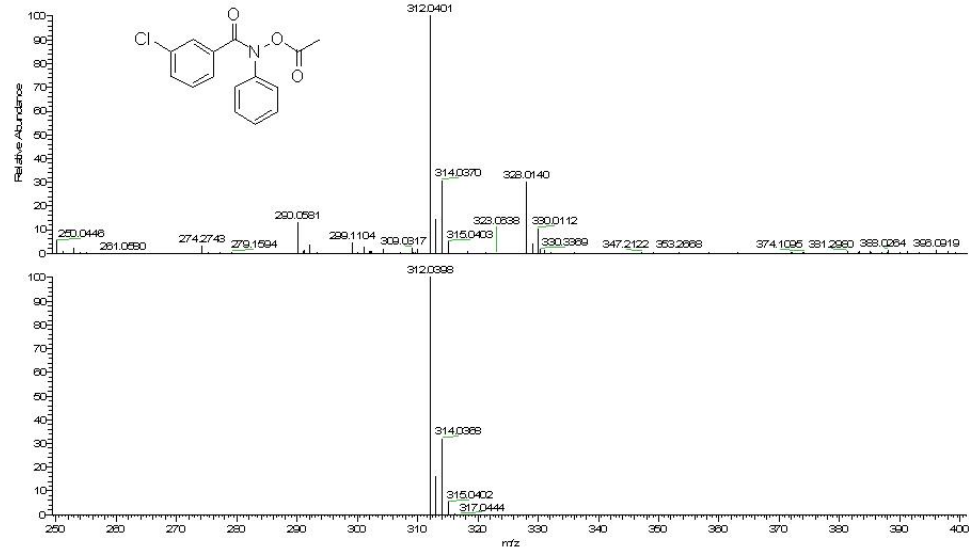
¹³C NMR copies of compound 4h:



HRMS (ESI) of compound 4h:

F:\User\zhanghuiyuan_2_170307155558

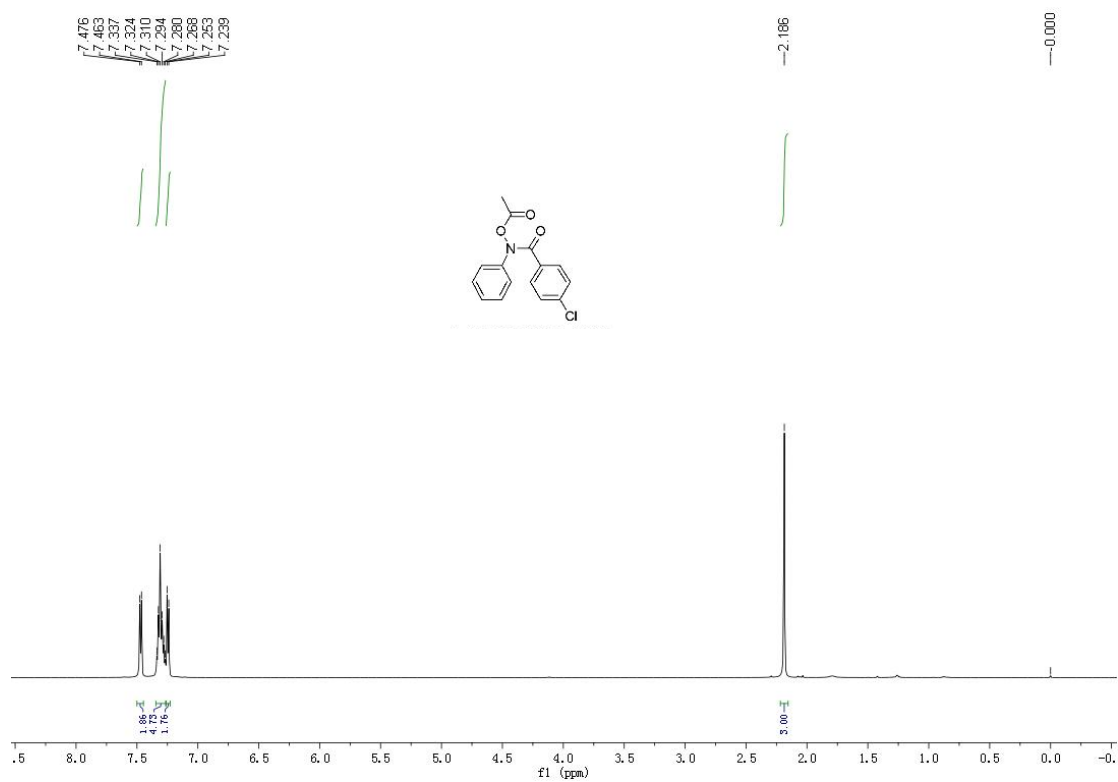
3/7/2017 5:02:20 PM
Error!0 ppm



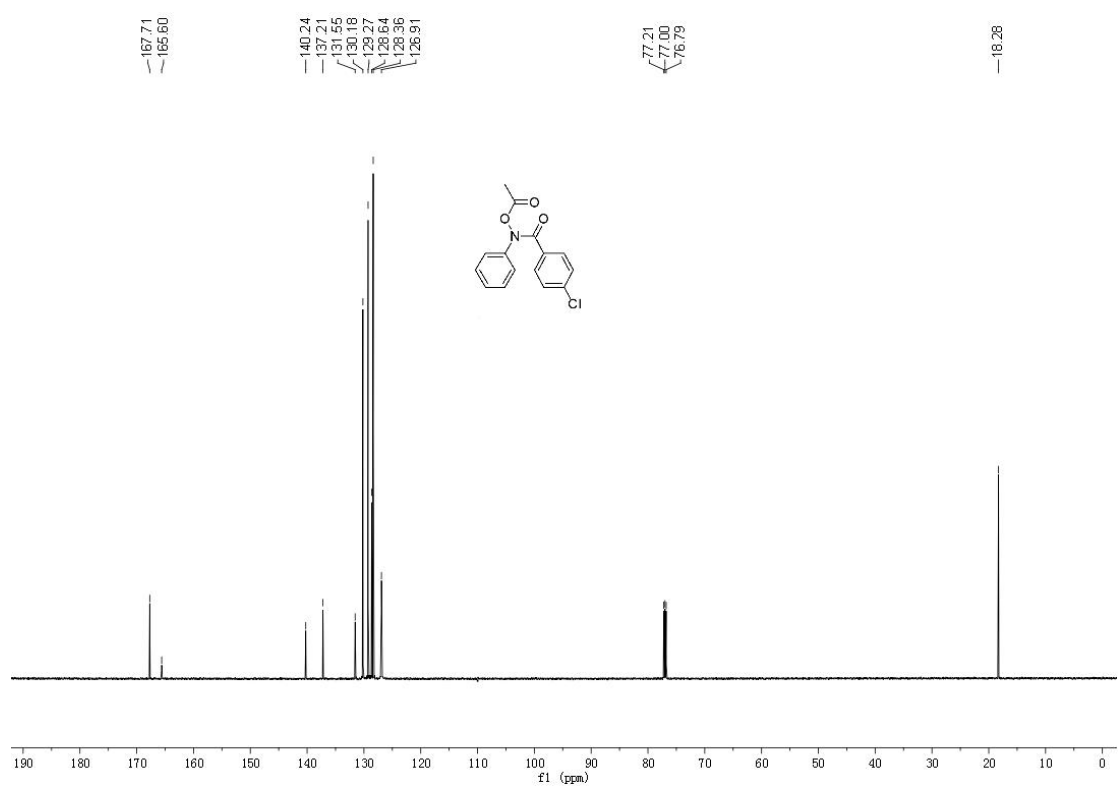
NL:
3.98EB
zhanghuiyuan
2_170307155558W18
RT: 0.19 AV: 1 T: FTMS
+p ESI Full MS
(100.00-2000.00)

NL:
6.37EB
C₁₅H₁₂Cl₁N₁O₃+Na⁺
C₁₅H₁₂Cl₁N₁O₃Na₁
pa Chrg 1

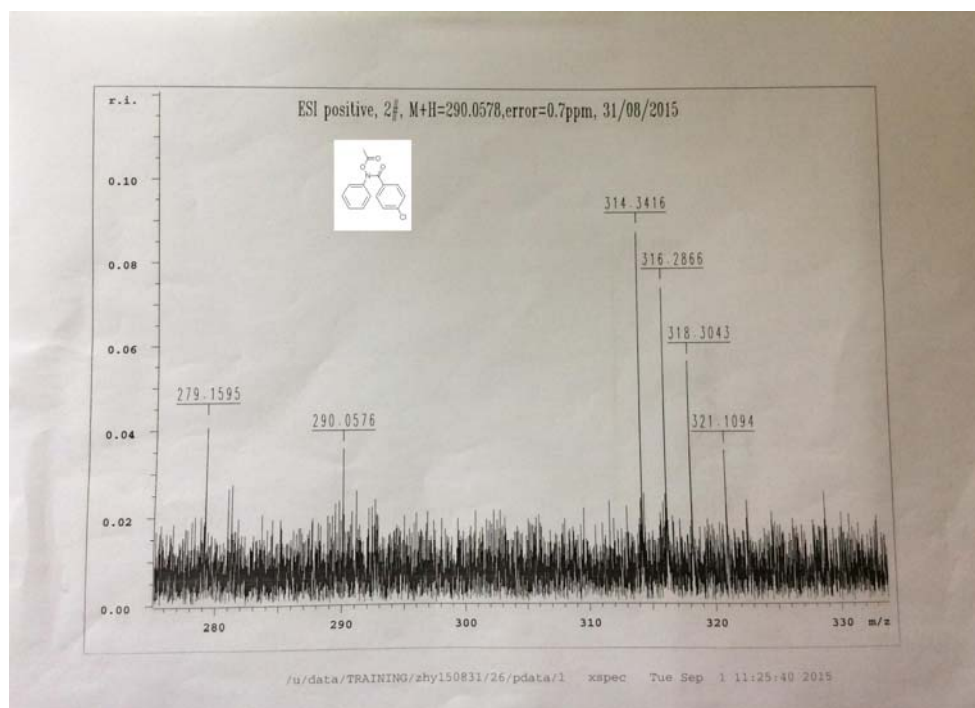
¹H NMR copies of compound **4i**:



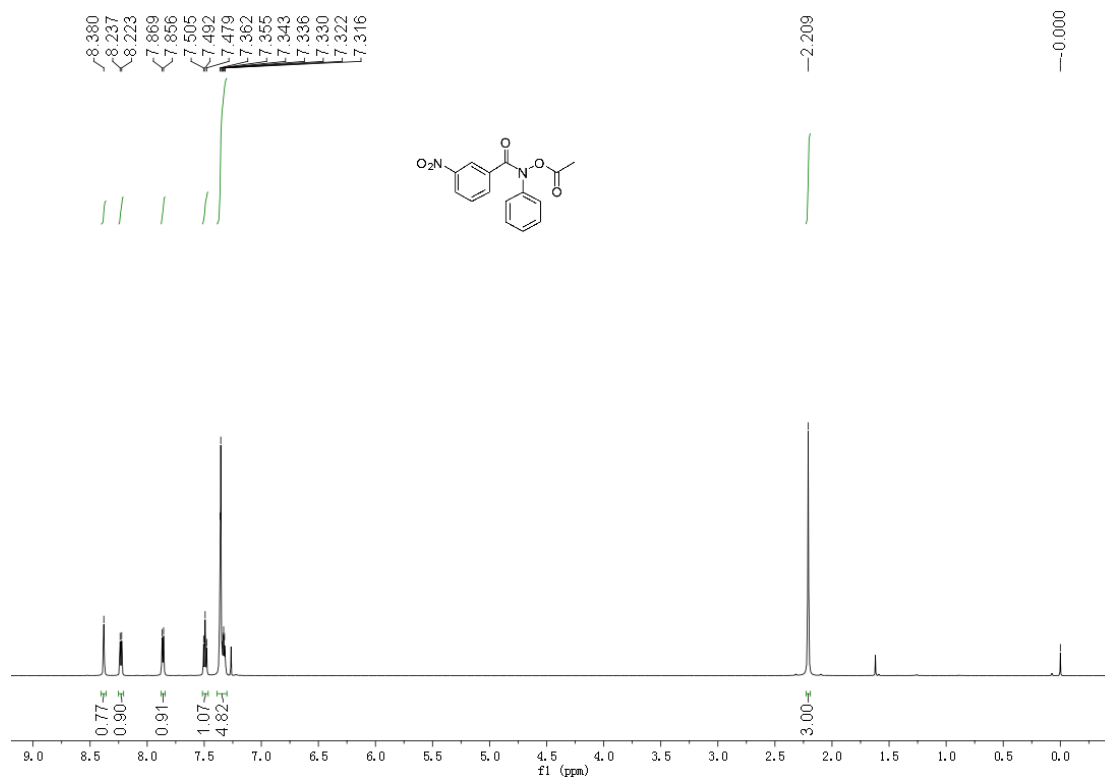
¹³C NMR copies of compound **4i**:



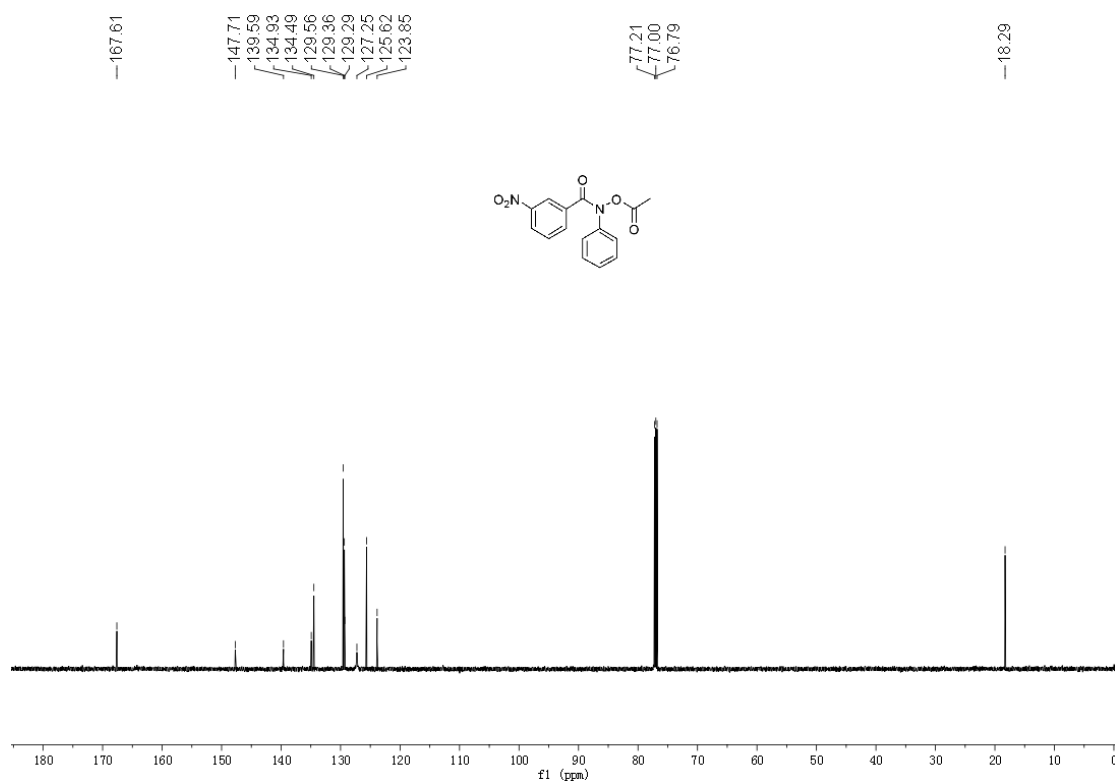
HRMS (ESI) of compound **4i**:



¹H NMR copies of compound **4k**:



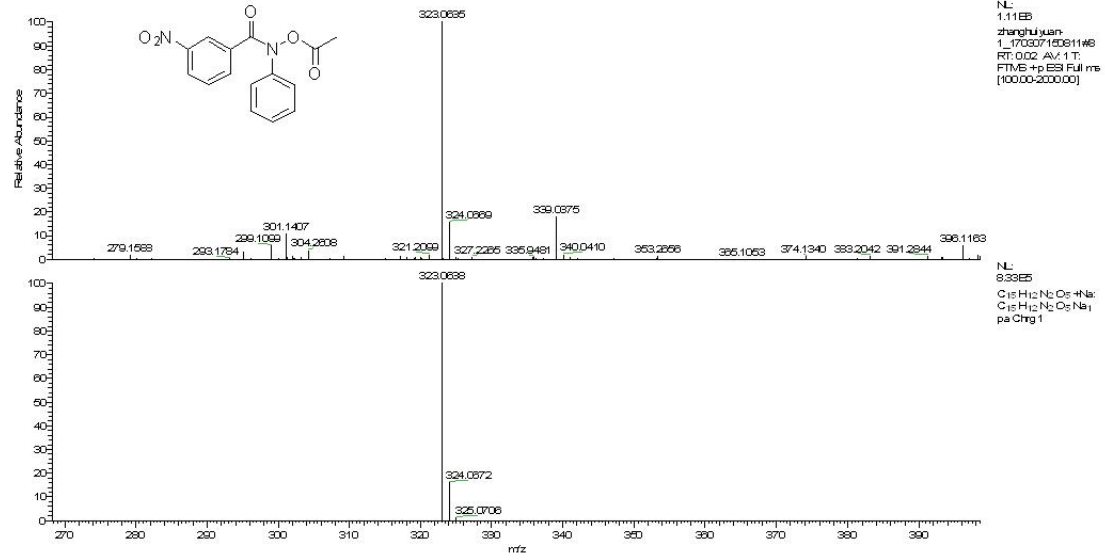
¹³C NMR copies of compound **4k**:



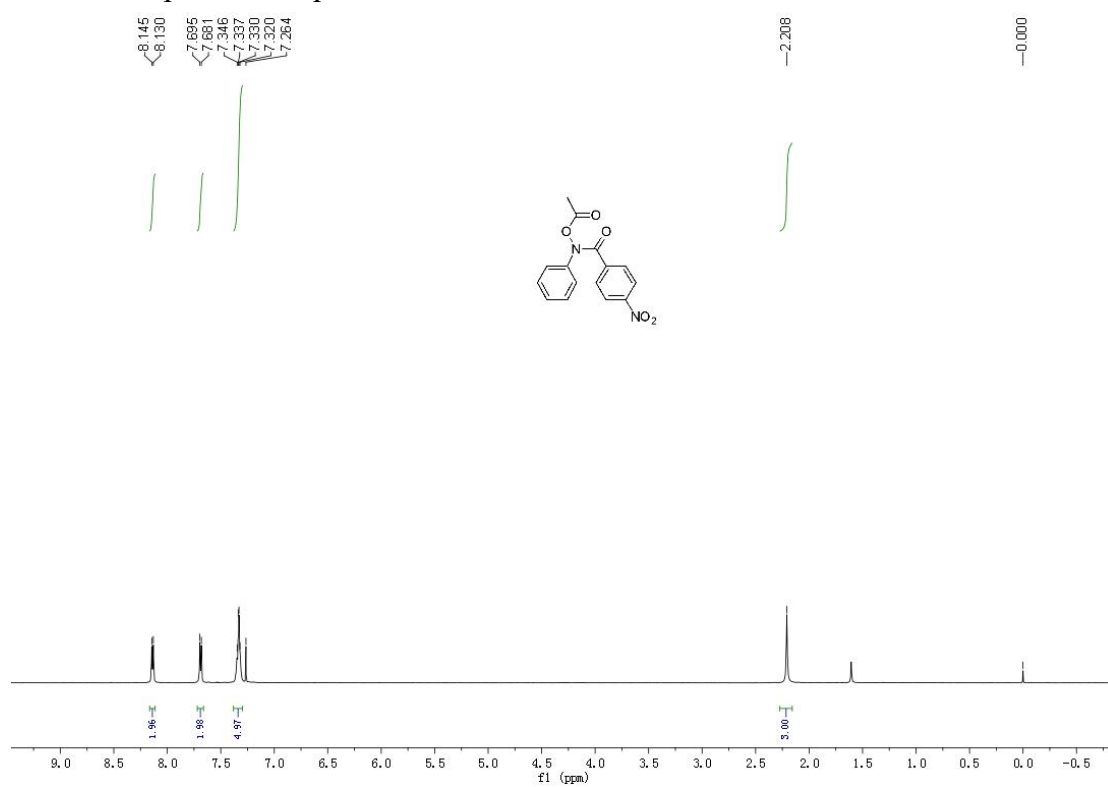
HRMS (ESI) of compound 4k:

F:\User\zhanghuiyuan_1_170307160811

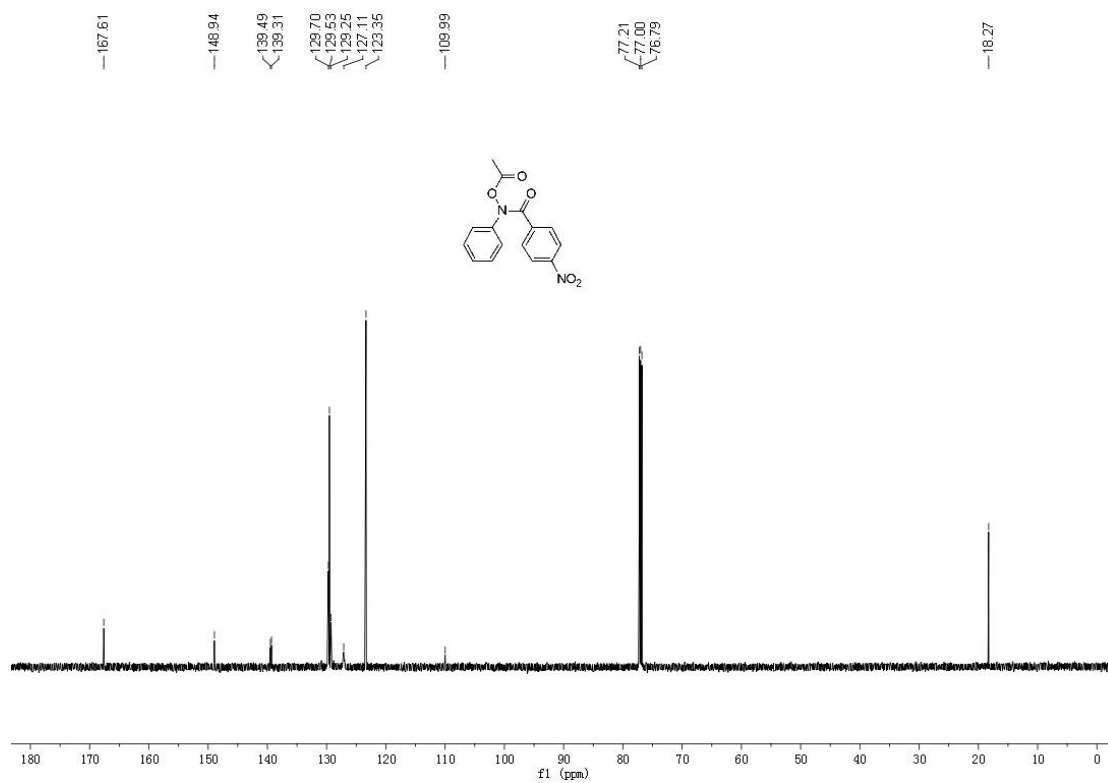
3/7/2017 3:28:22 PM
Error=0.9 ppm



¹H NMR copies of compound 4I:



¹³C NMR copies of compound 4I:

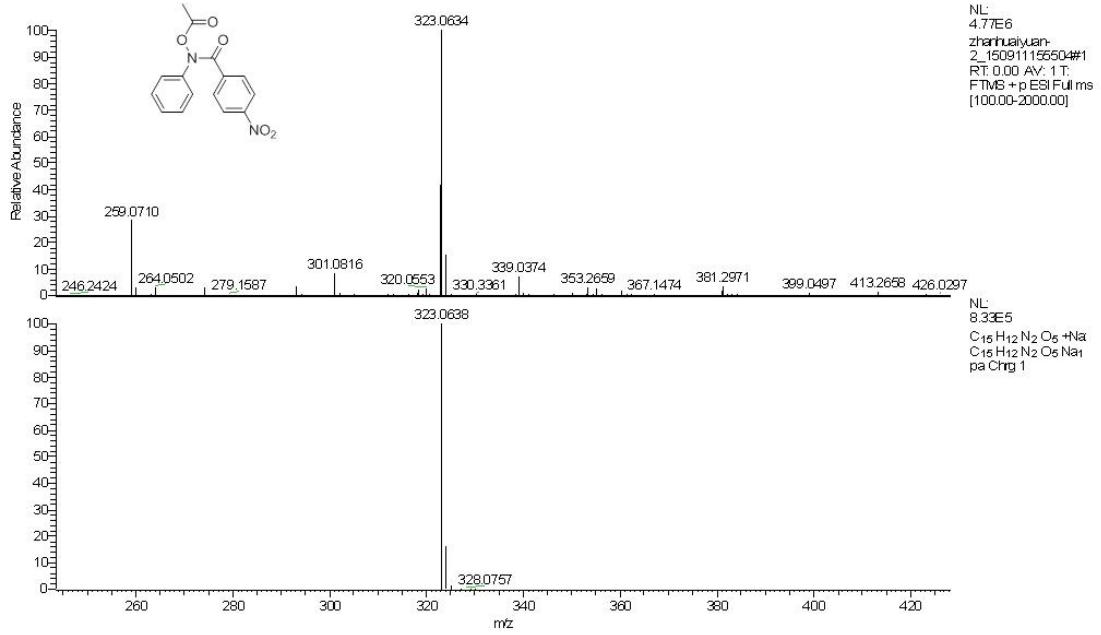


HRMS (ESI) of compound 4I:

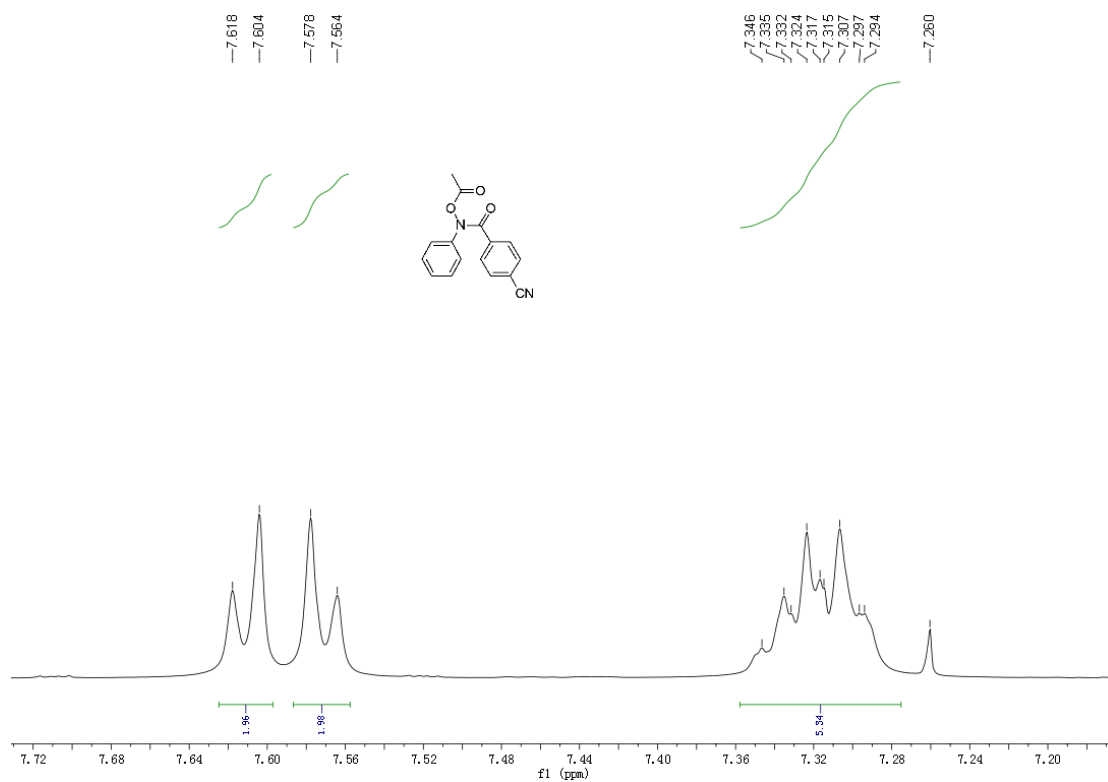
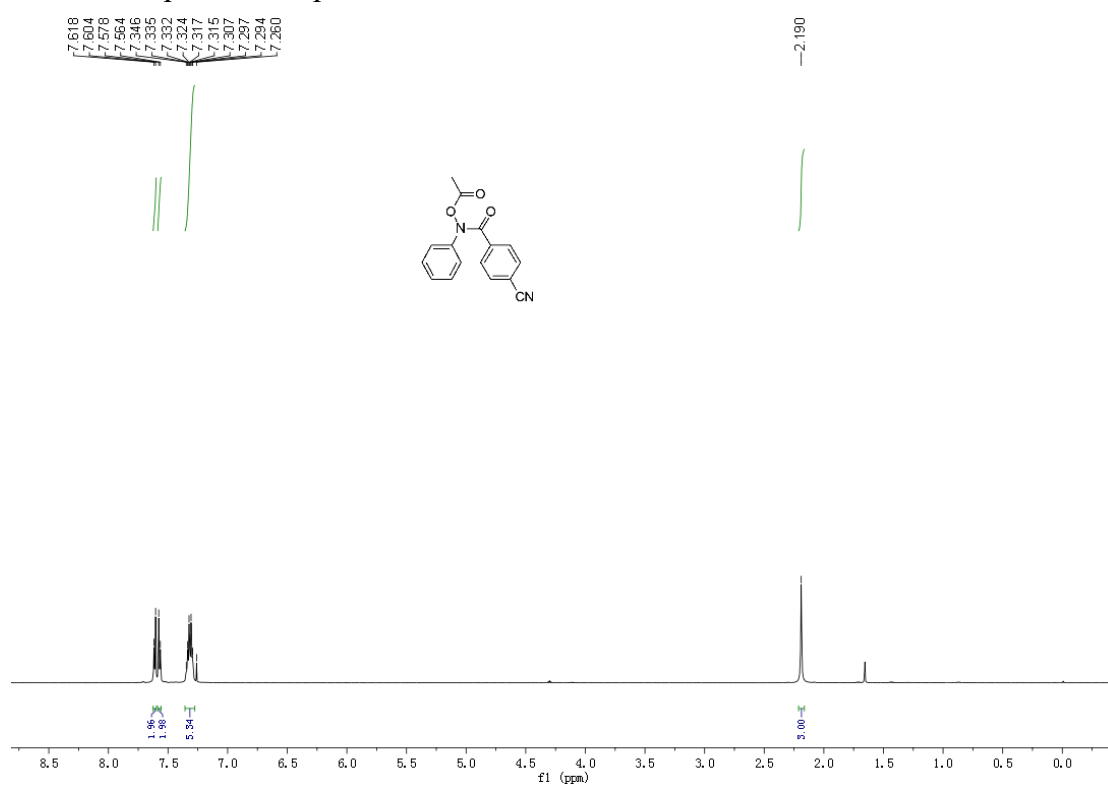
D:\Users\...zhanhuaiyuan-2_150911155504

9/11/2015 4:01:09 PM
Error=1.2 ppm

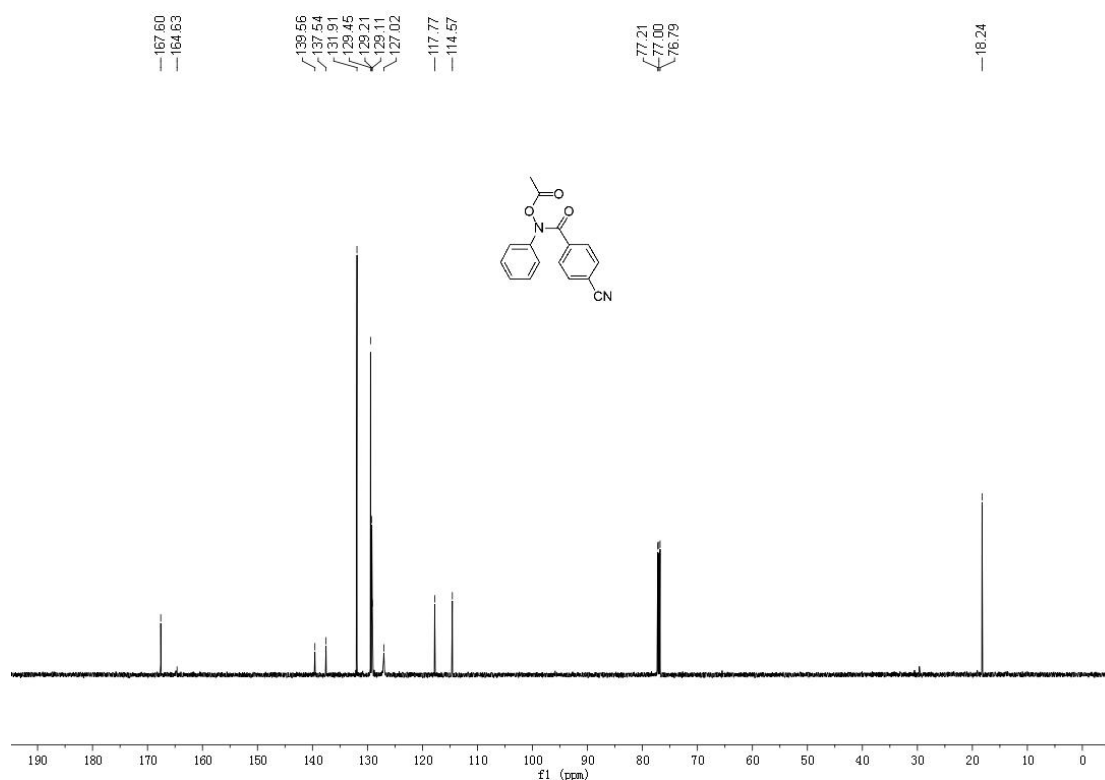
2#



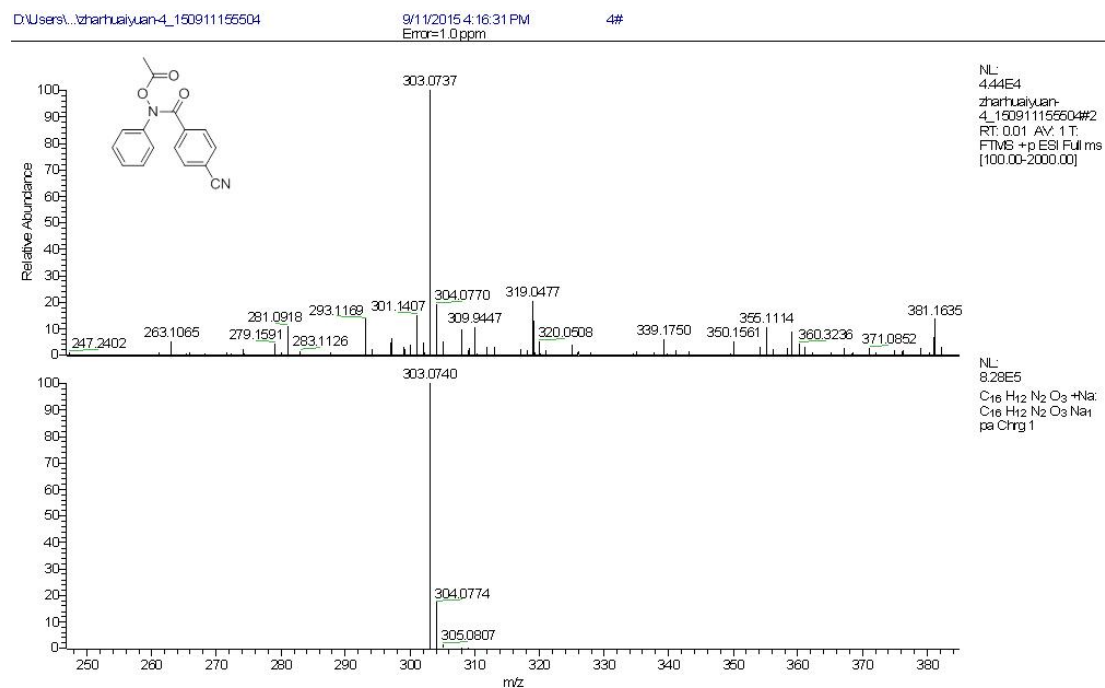
¹H NMR copies of compound 4m:



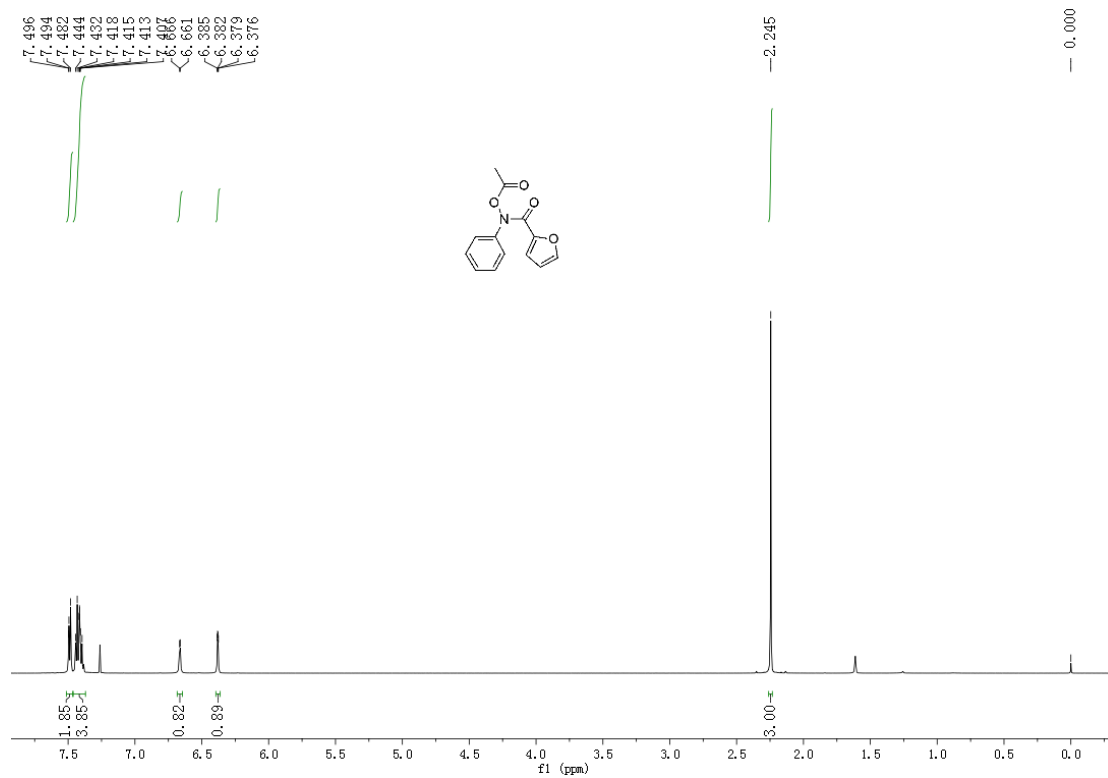
¹³C NMR copies of compound **4m**:



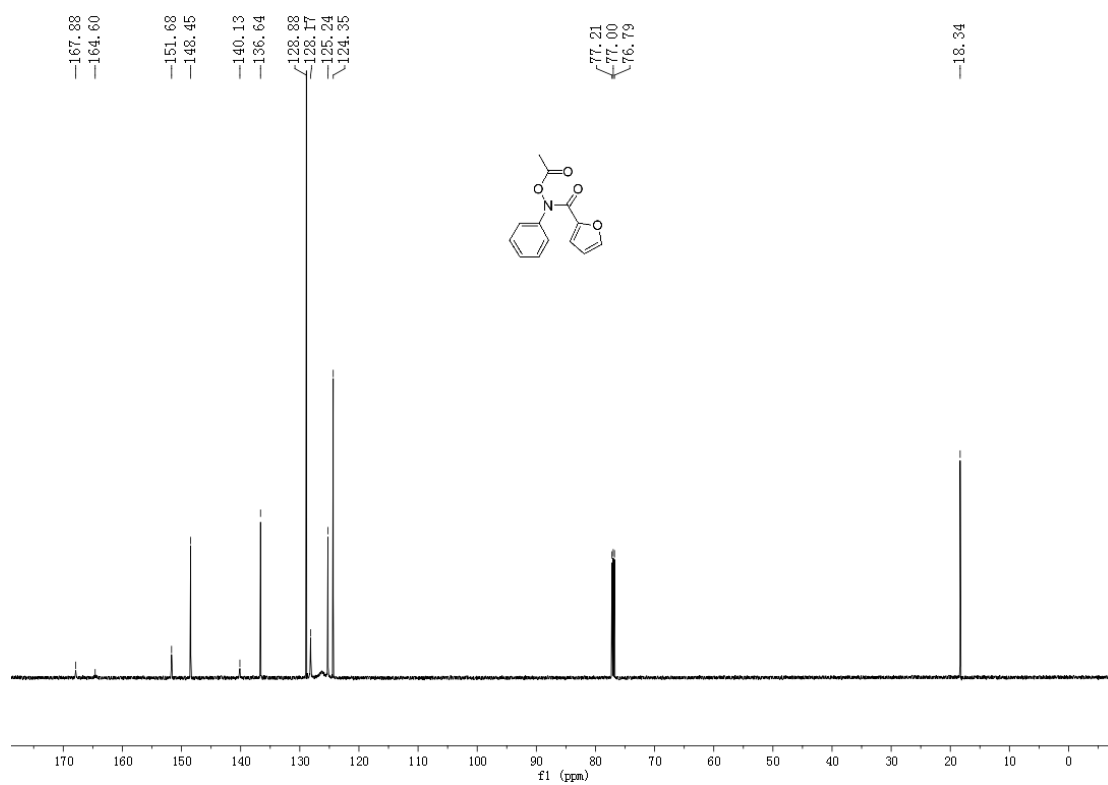
HRMS (ESI) of compound **4m**:



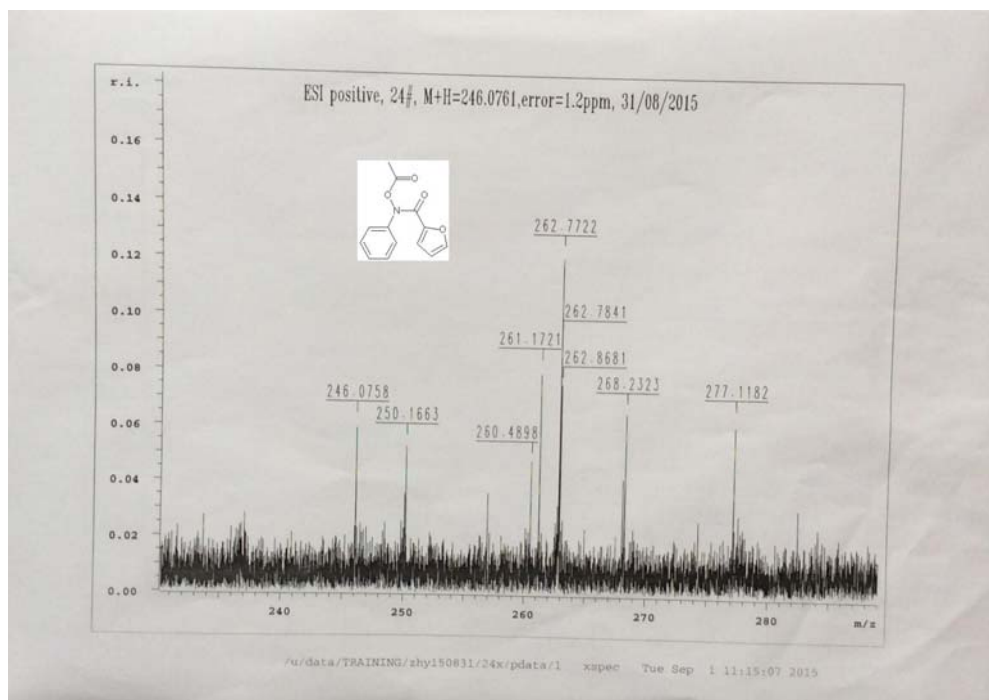
¹H NMR copies of compound **4n**:



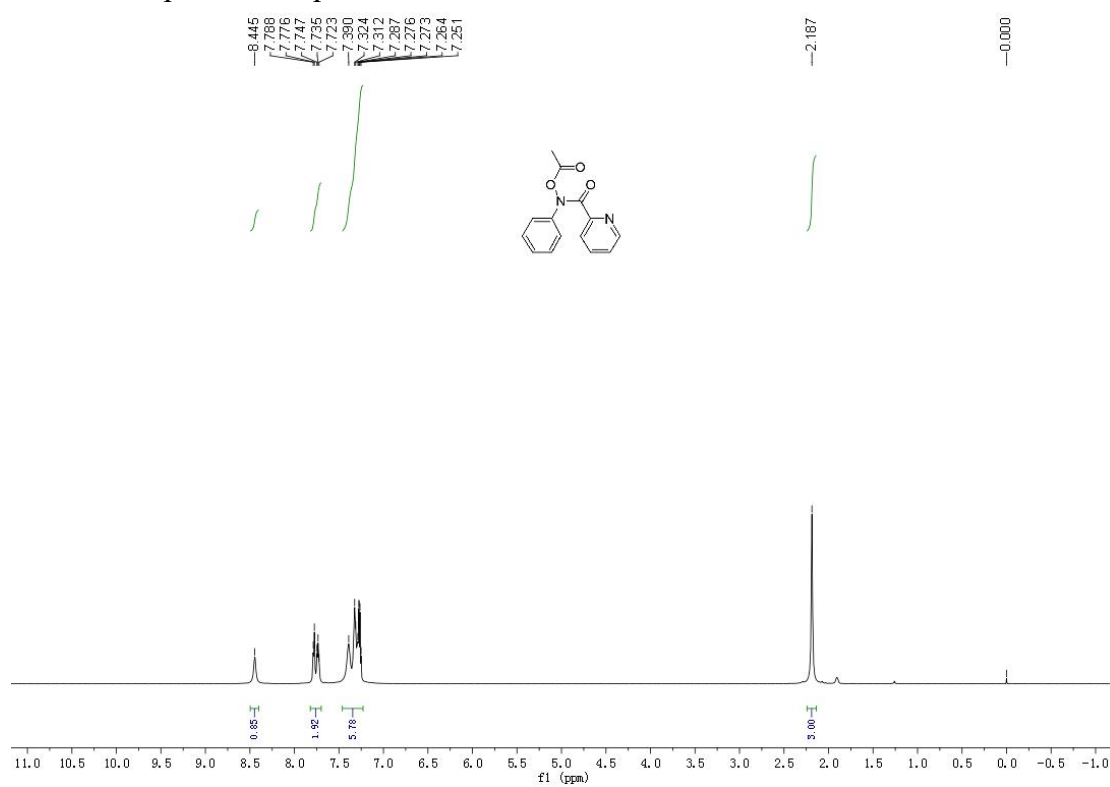
¹³C NMR copies of compound **4n**:



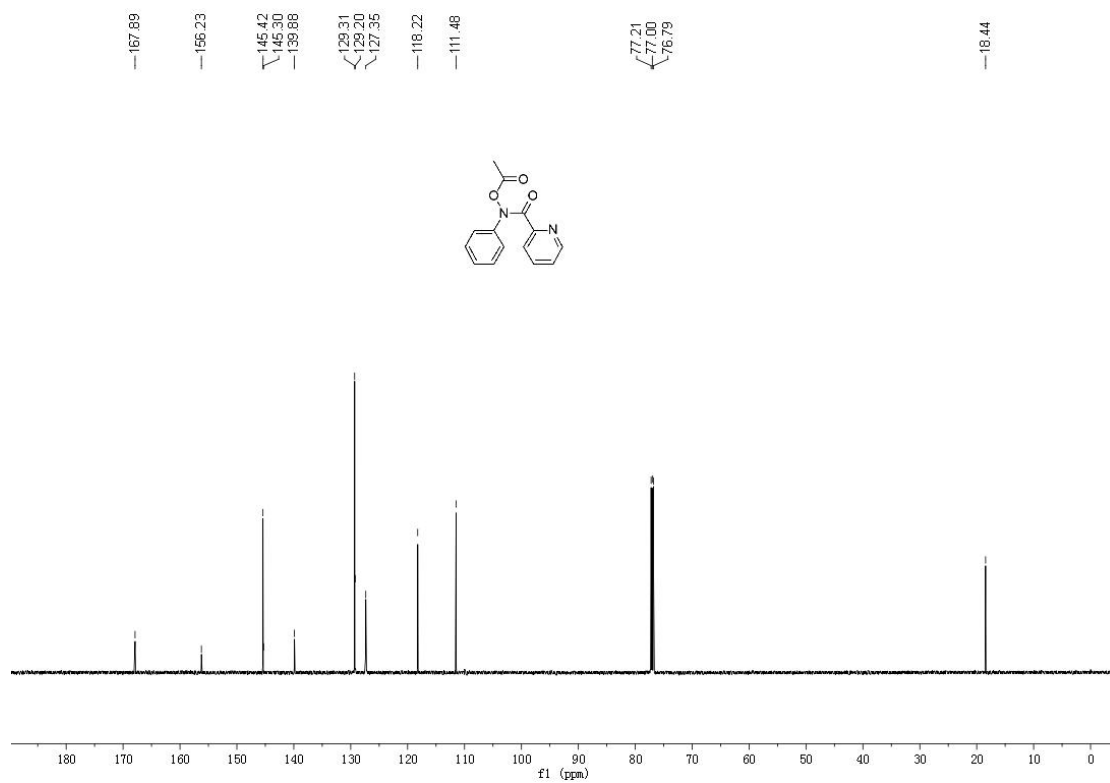
HRMS (ESI) of compound **4n**:



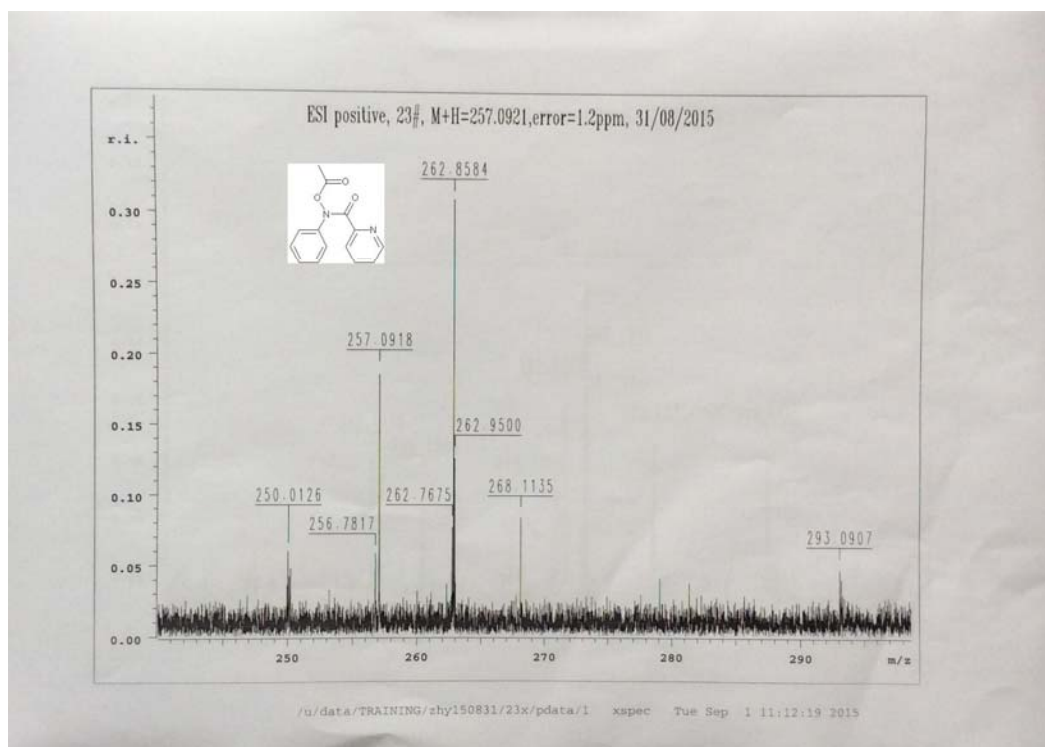
¹H NMR copies of compound **4o**:



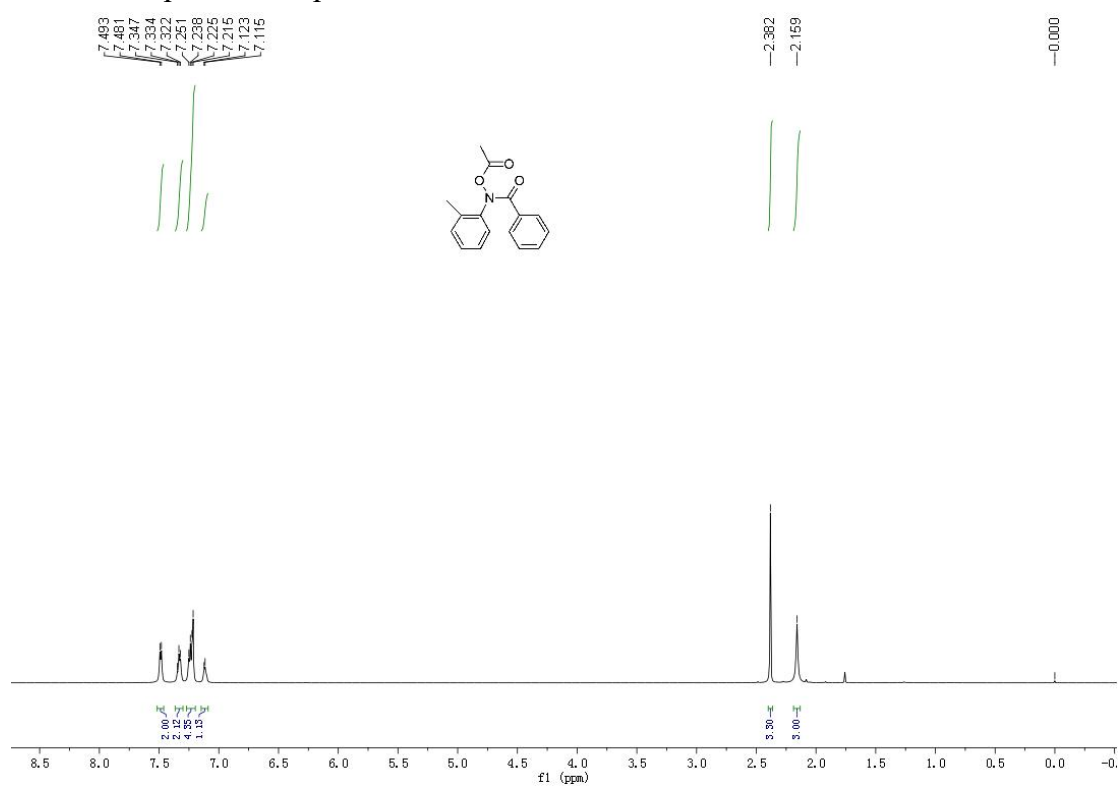
¹³C NMR copies of compound **4o**:



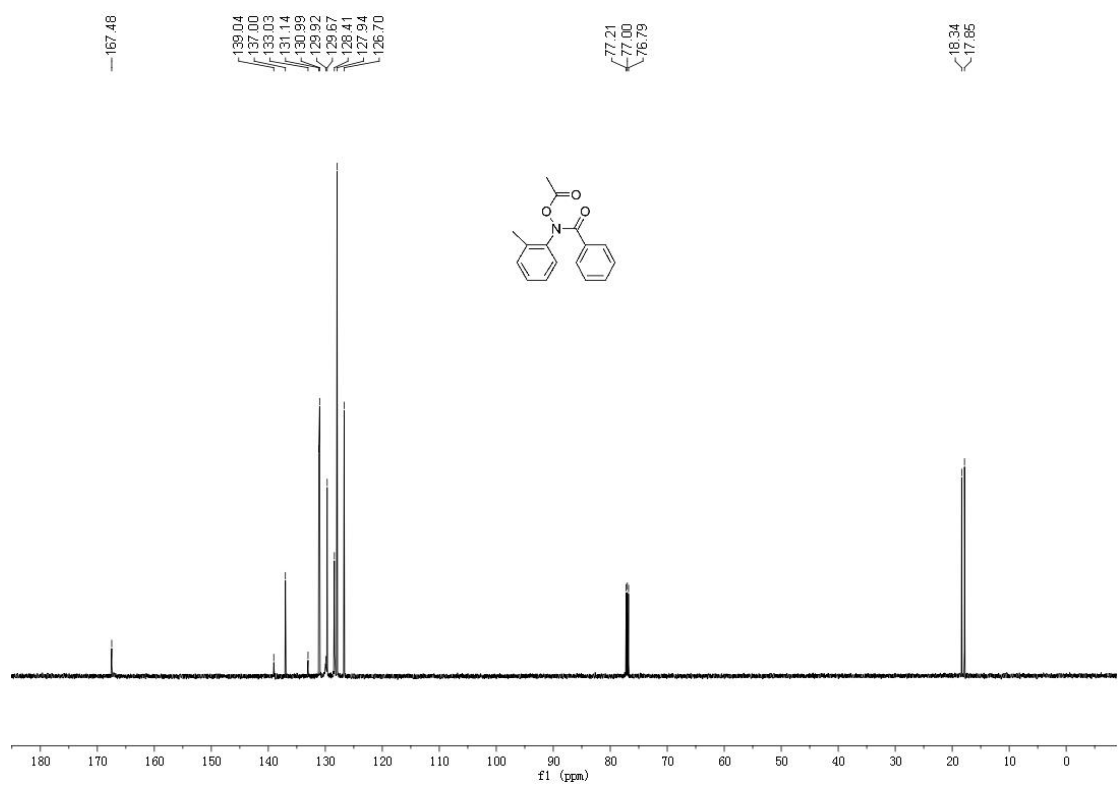
HRMS (ESI) of compound **4o**:



¹H NMR copies of compound 4r:



¹³C NMR copies of compound 4r:

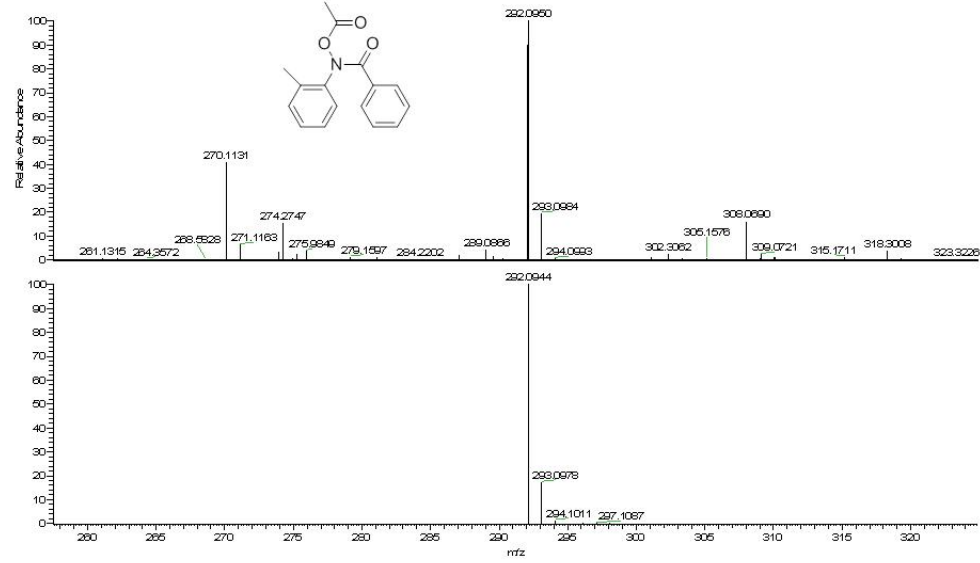


HRMS (ESI) of compound **4r**:

F:\UserA\zhanghujuan_3_150930143550

9/30/2015 2:52:05 PM
Err=2.1 ppm

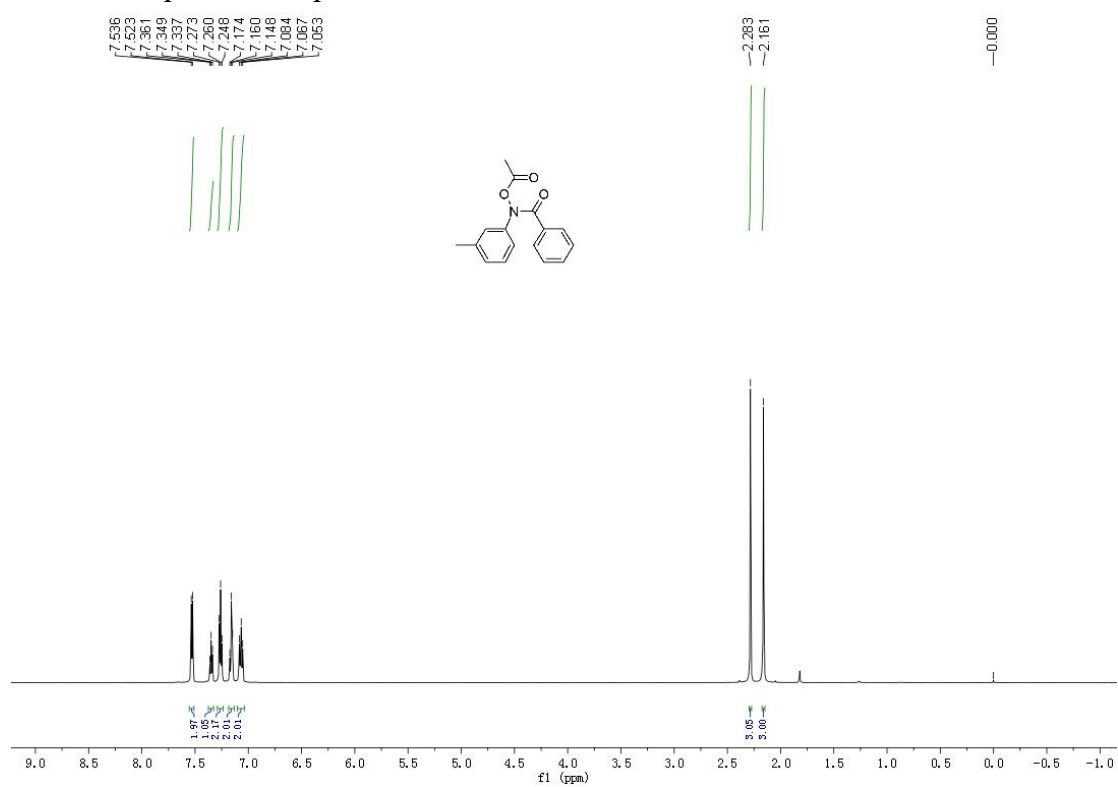
3#



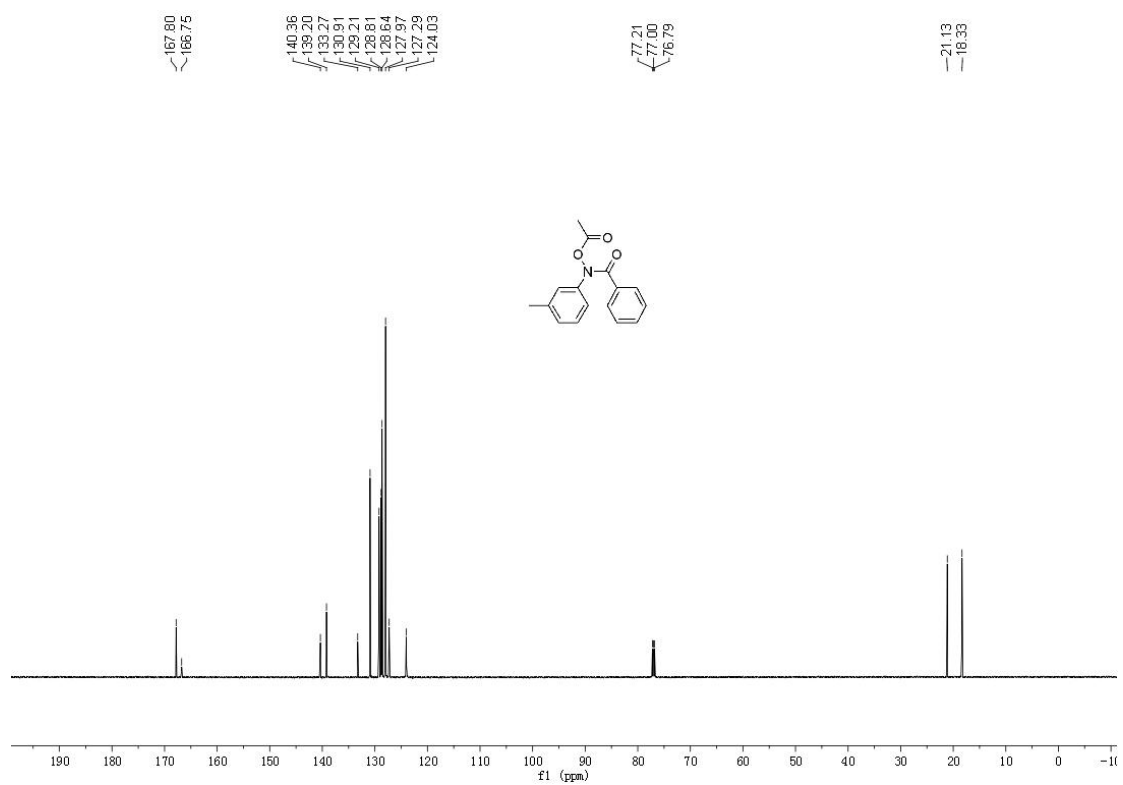
NL:
1.62E5
zhanghujuan-
3_150930143550#9
RT: 0.19, AV: 1 T:
FTMS +p, ES Full ms
[100.00-300.00]

NL:
8.31E5
C₁₈H₁₅N₁O₃#Na:
C₁₈H₁₅N₁O₃Na₁
pa-Chrg 1

¹H NMR copies of compound **4s**:



¹³C NMR copies of compound **4s**:

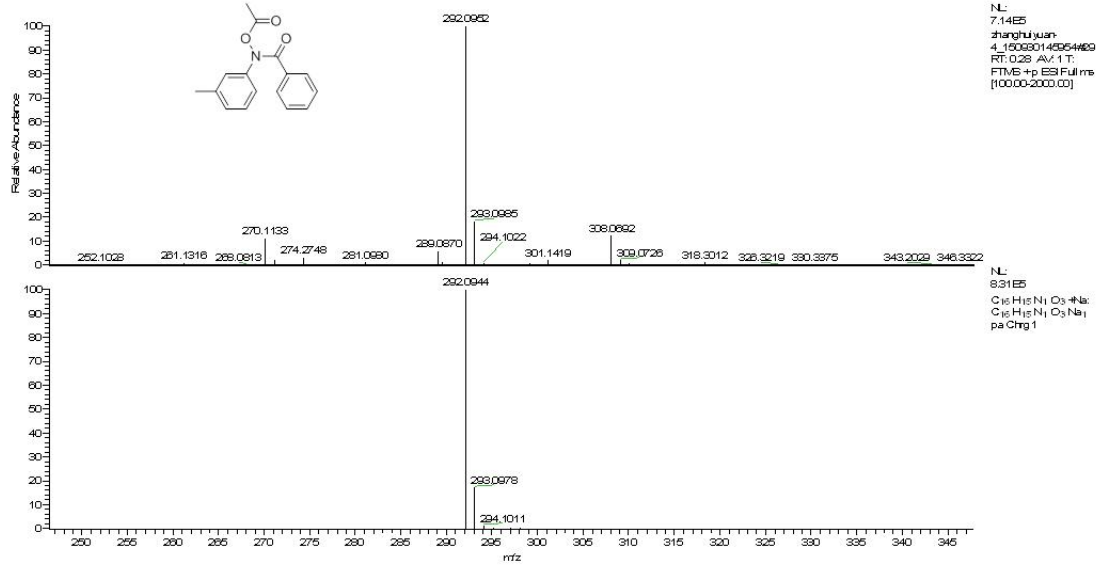


HRMS (ESI) of compound 4s:

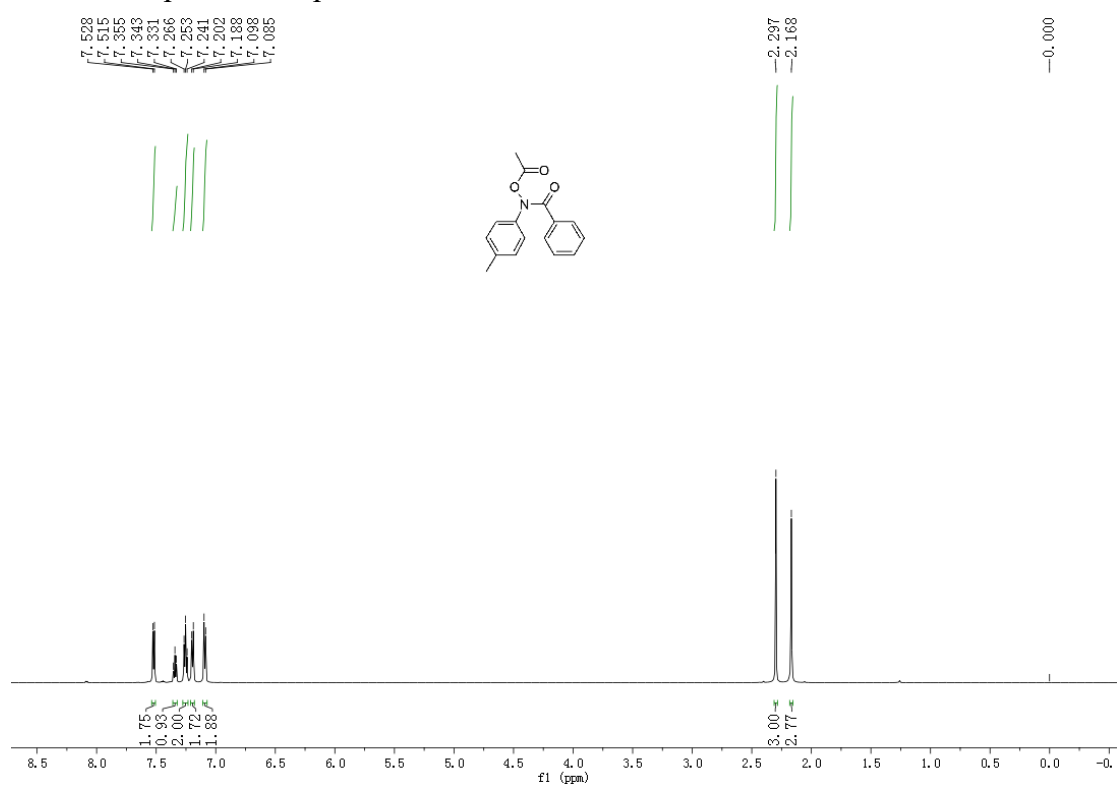
F:\UserA\zhanghujuan-4_150930145954

9/30/2015 2:59:55 PM
Err=2.7 ppm

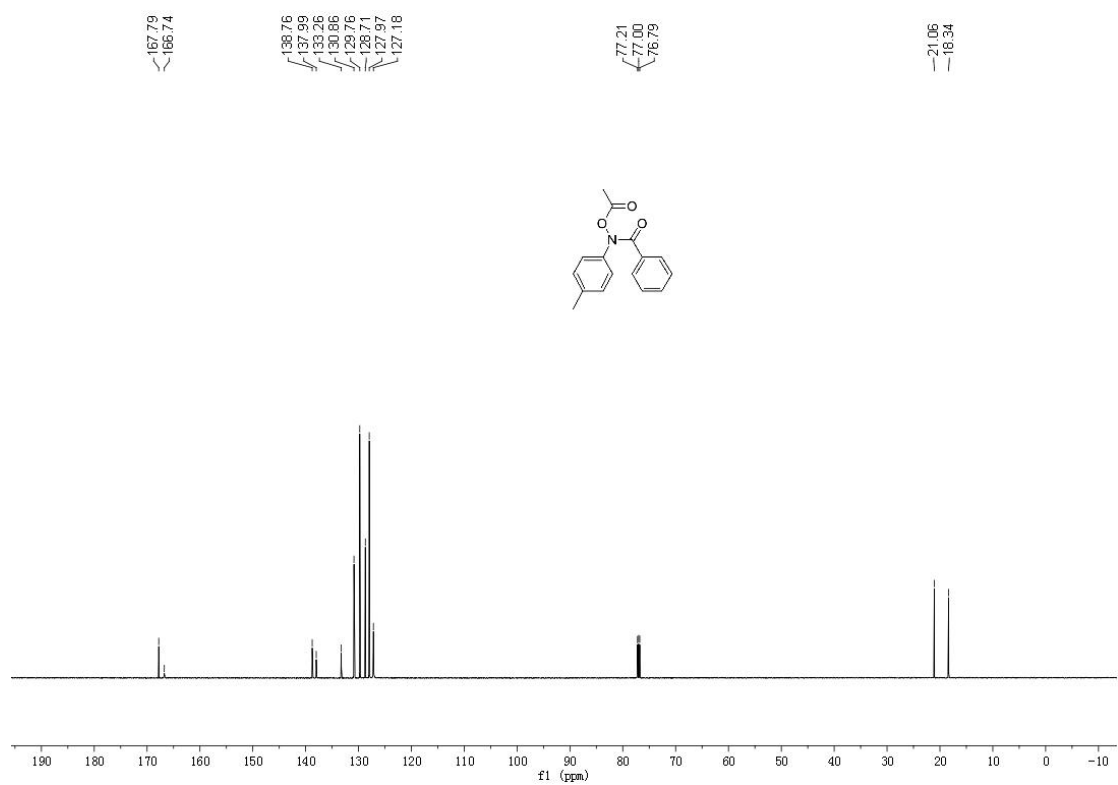
4#



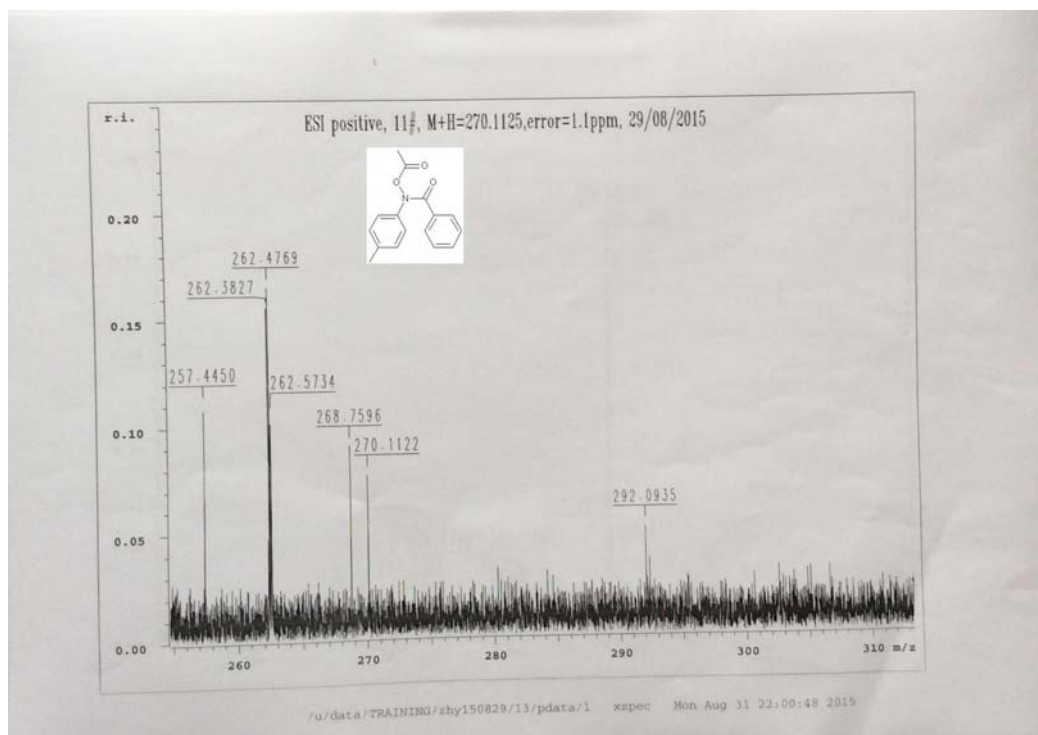
¹H NMR copies of compound 4t:



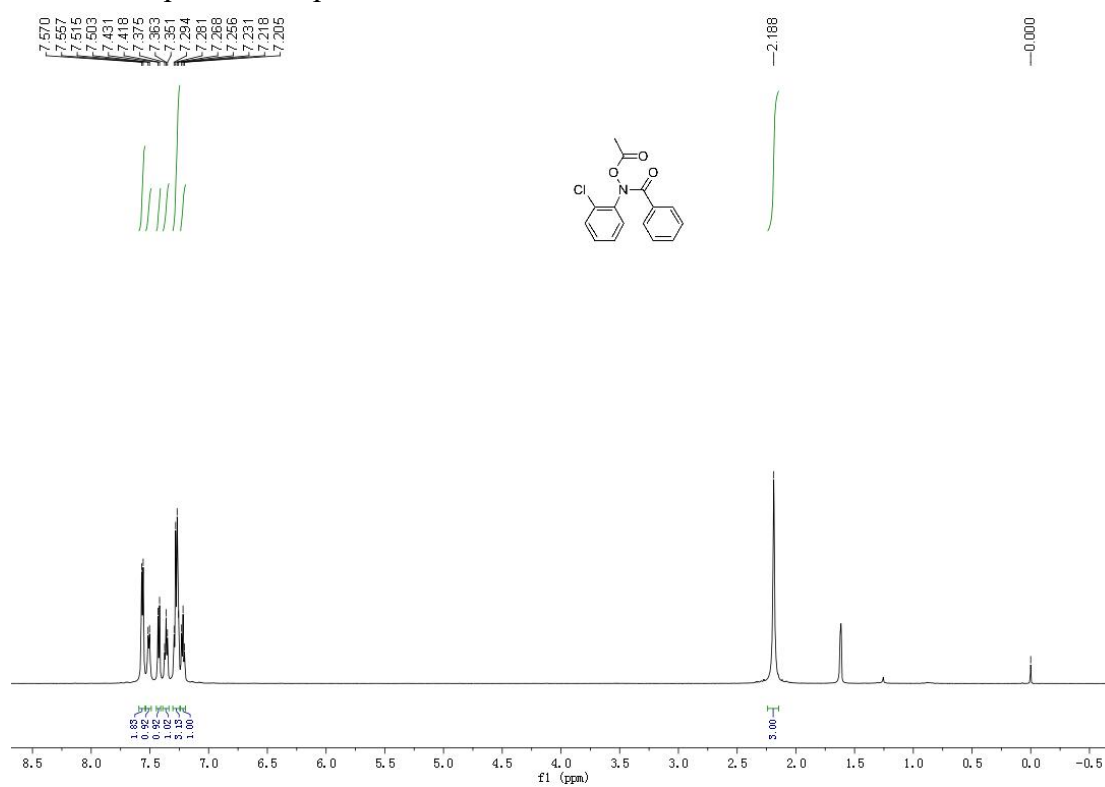
¹³C NMR copies of compound 4t:



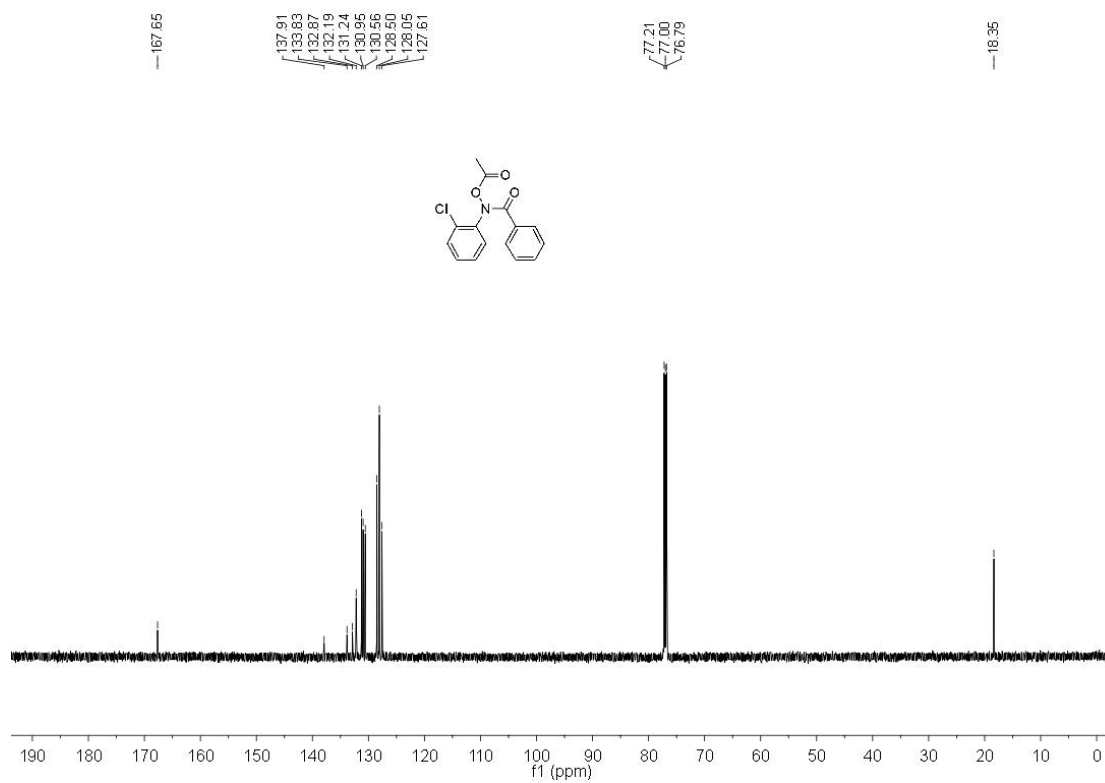
HRMS (ESI) of compound **4t**:



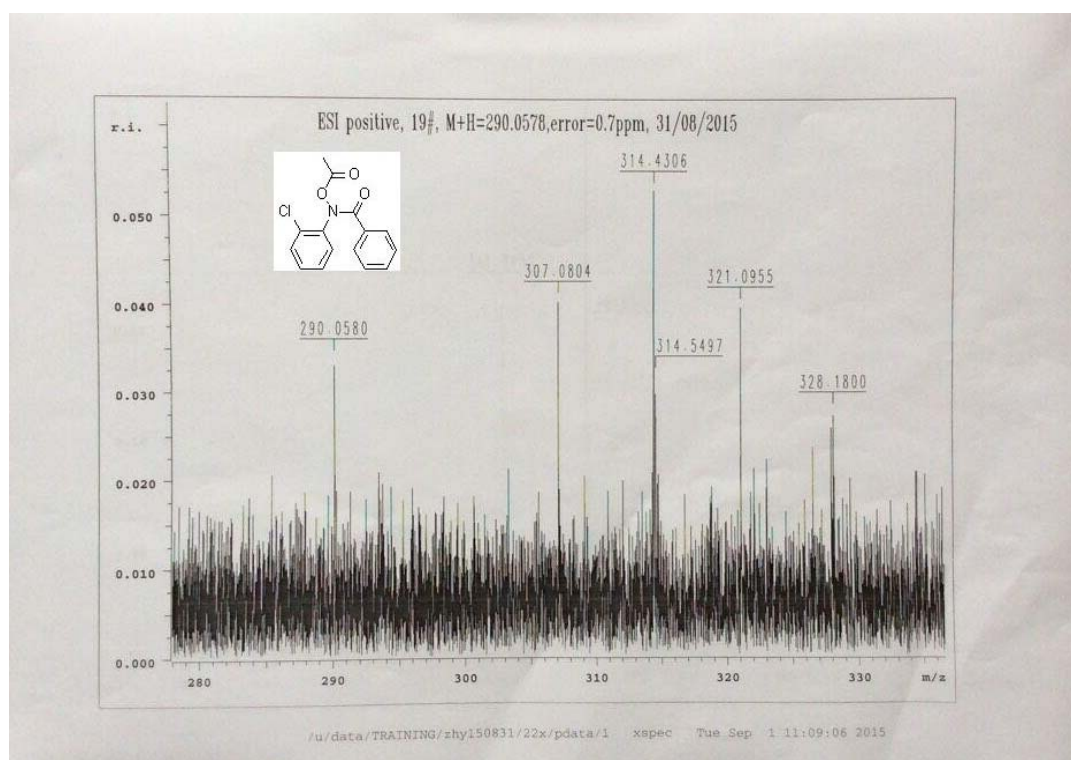
¹H NMR copies of compound **4u**:



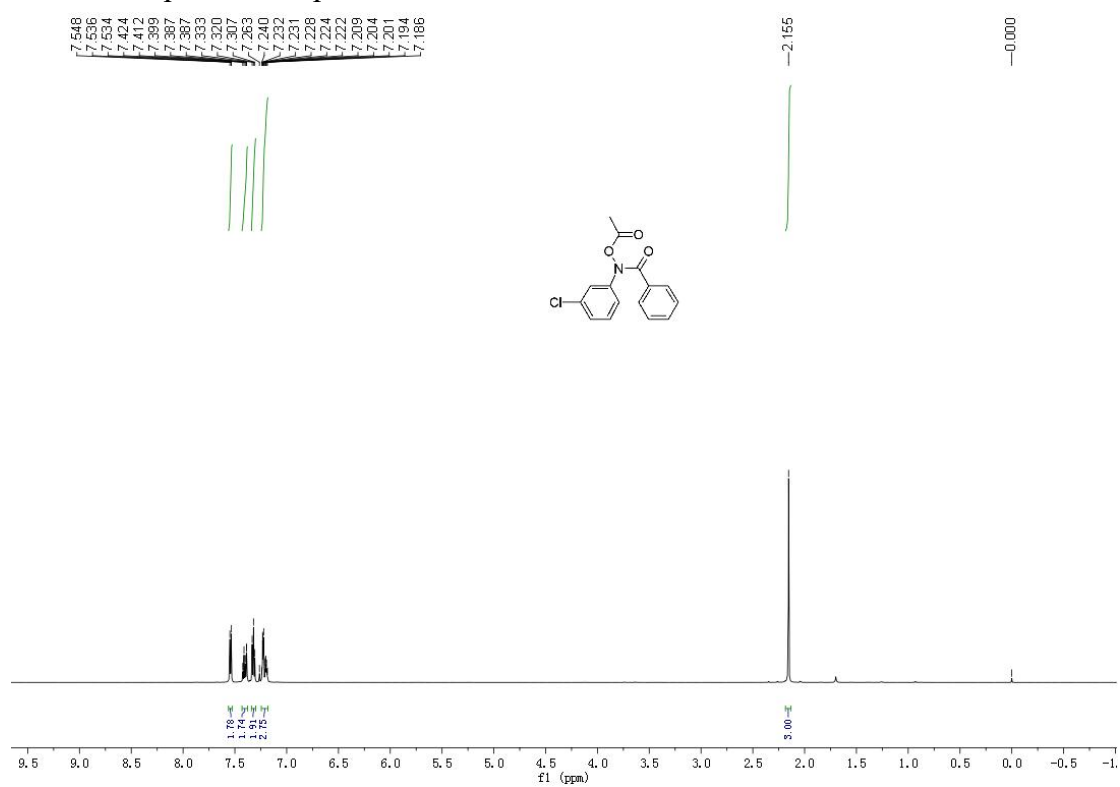
¹³C NMR copies of compound **4u**:



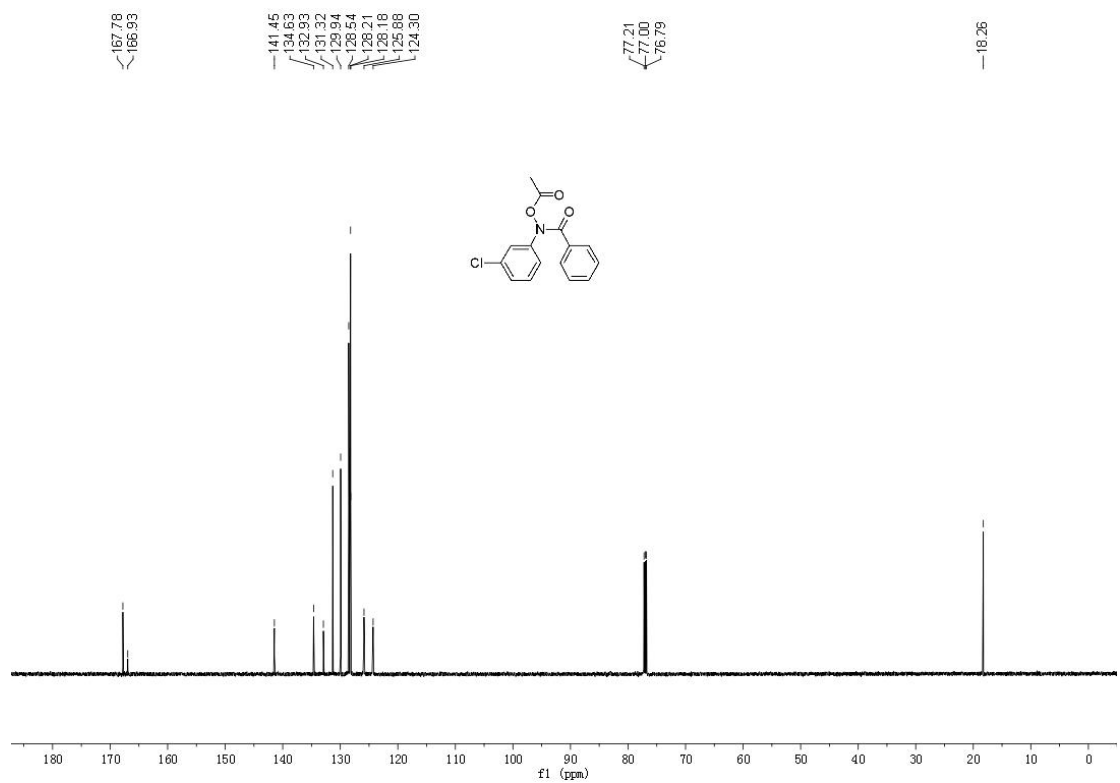
HRMS (ESI) of compound **4u**:



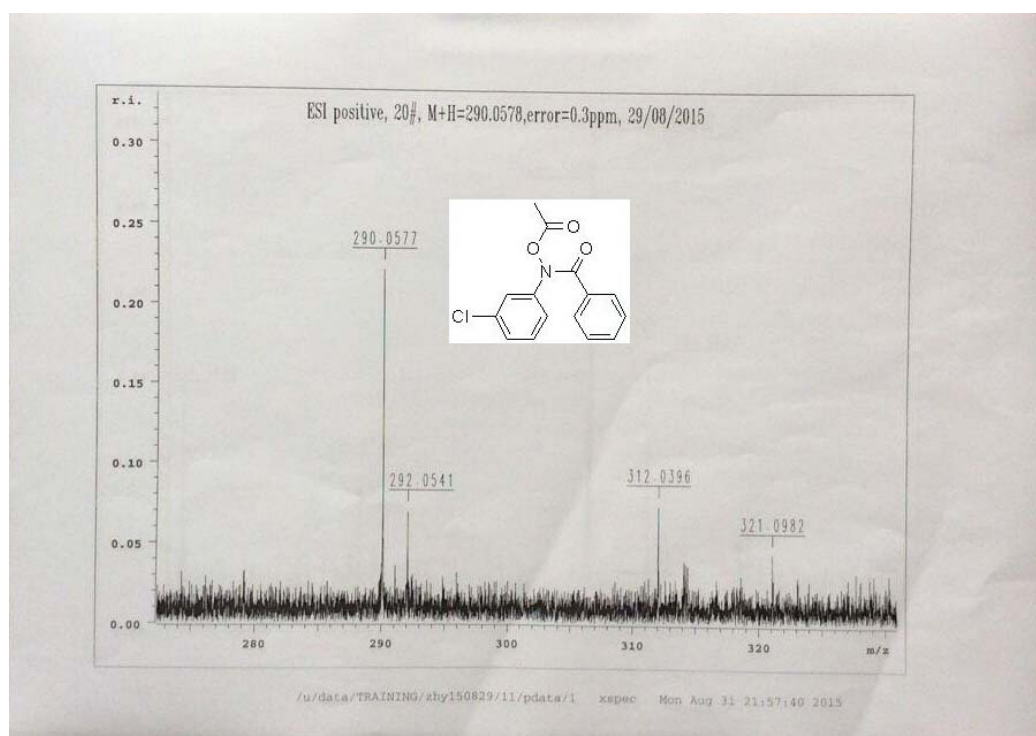
¹H NMR copies of compound 4v:



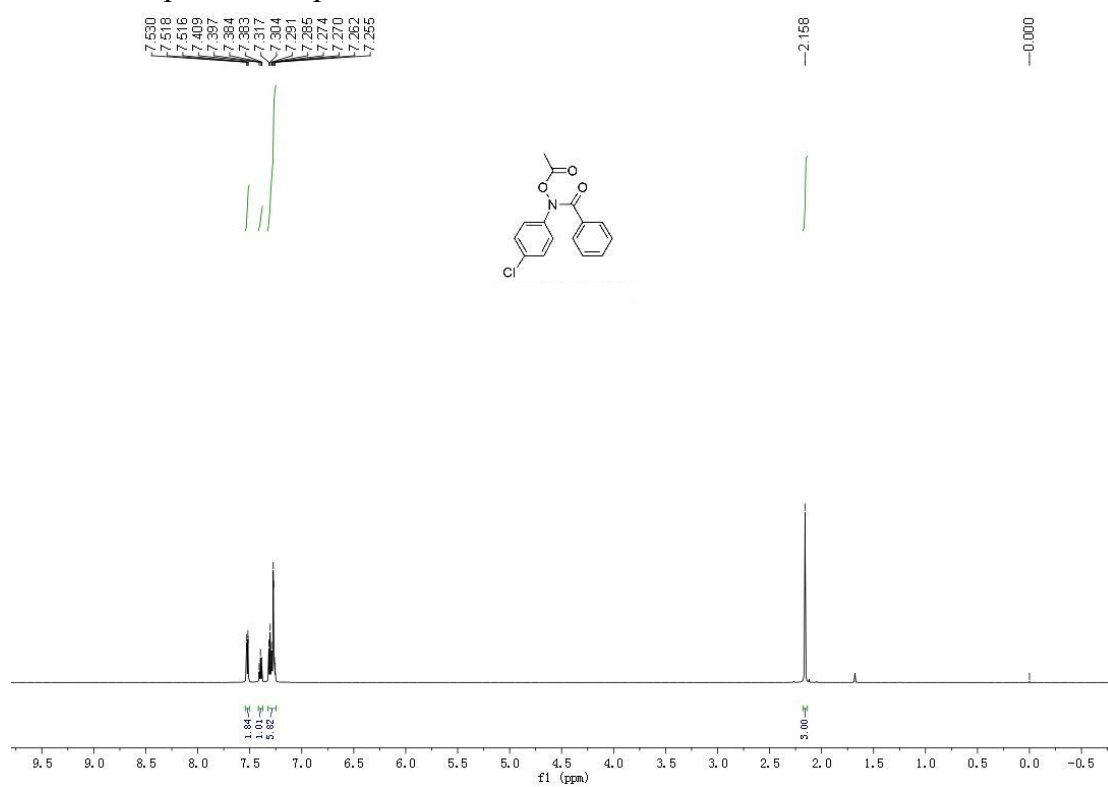
¹³C NMR copies of compound 4v:



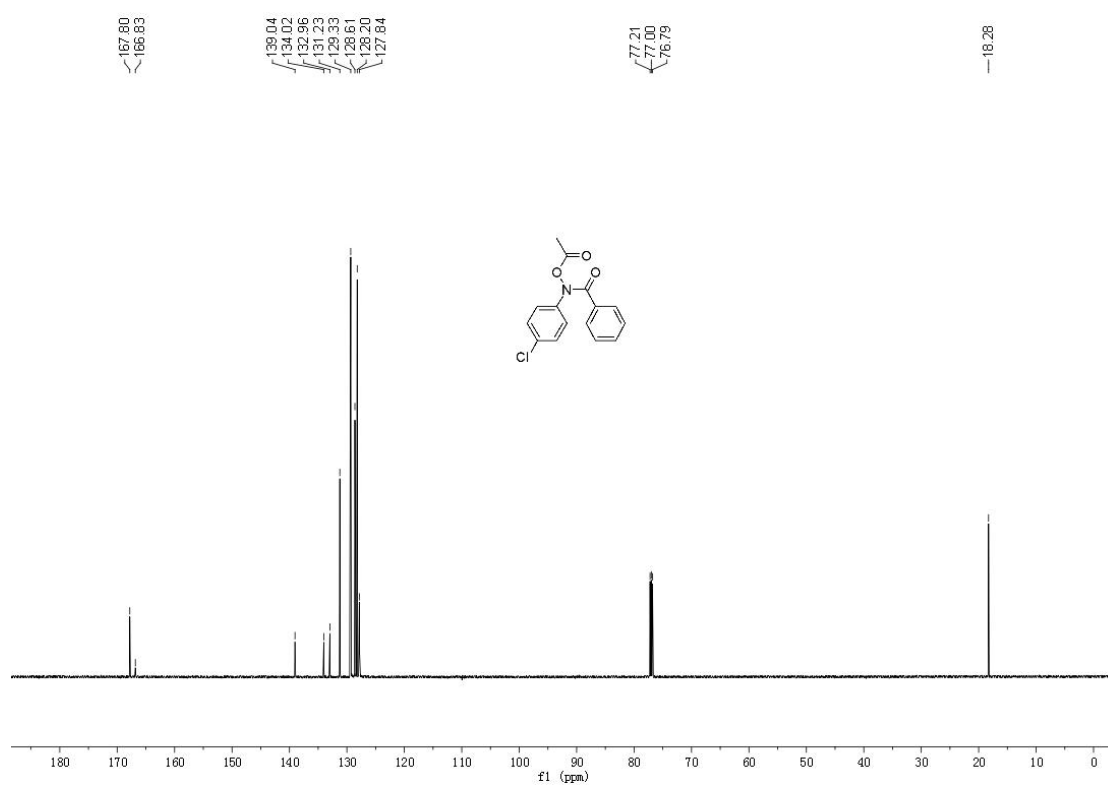
HRMS (ESI) of compound **4v**:



¹H NMR copies of compound **4w**:



¹³C NMR copies of compound **4w**:

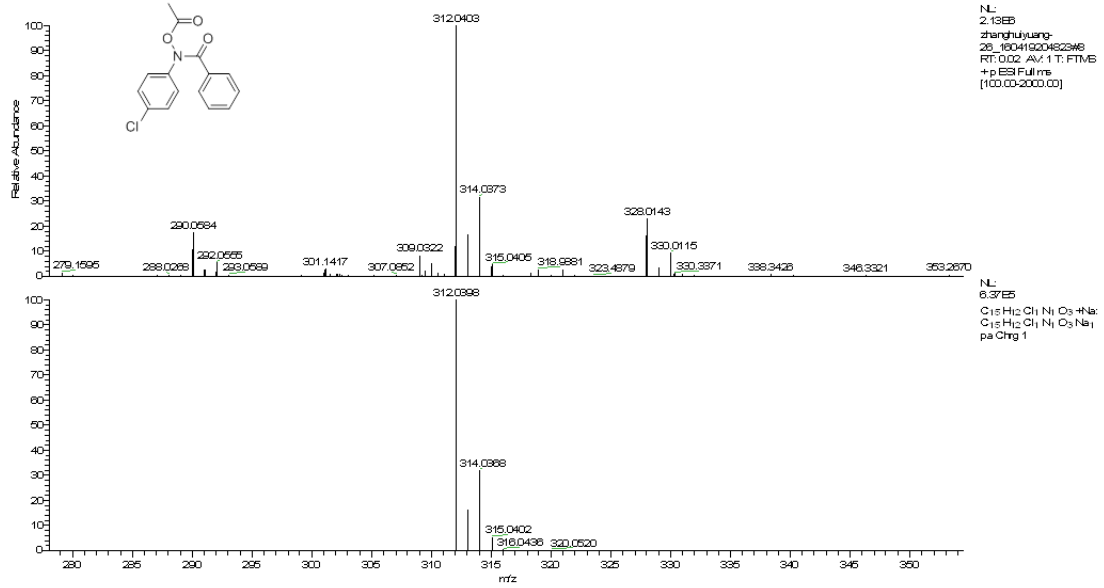


HRMS (ESI) of compound **4w**:

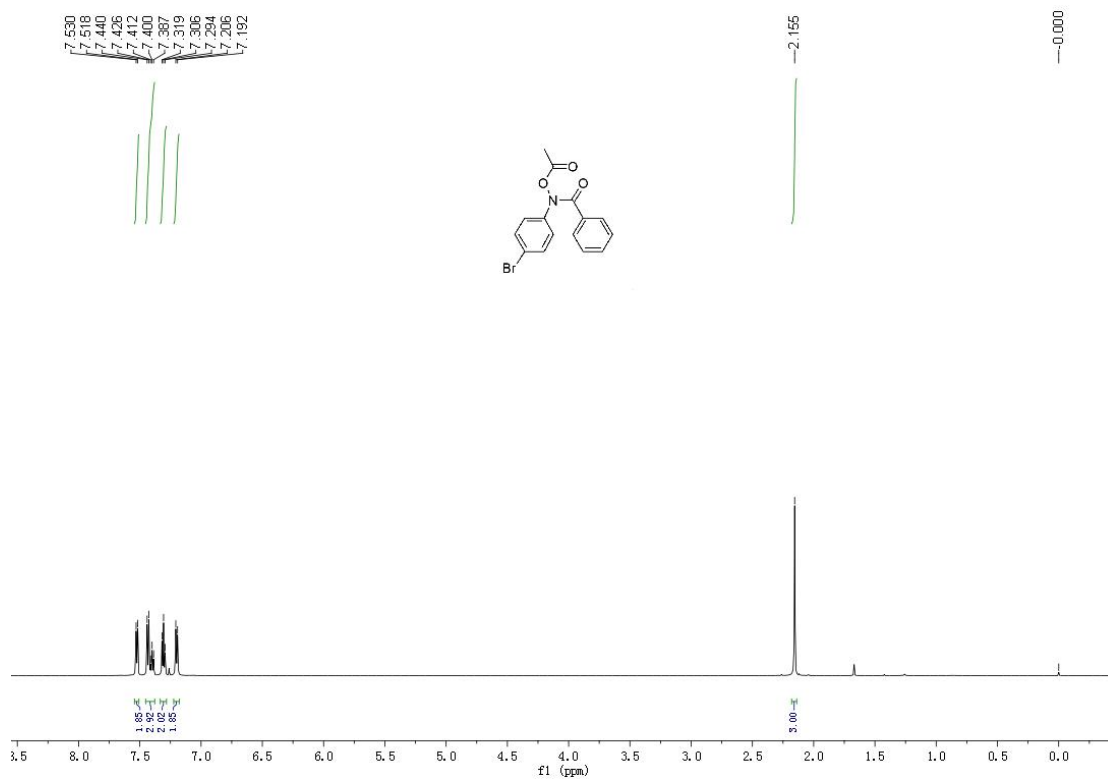
zhenghuyuang_28_180418204823

4/20/2018 9:45:17 AM
Error: 1.3 ppm

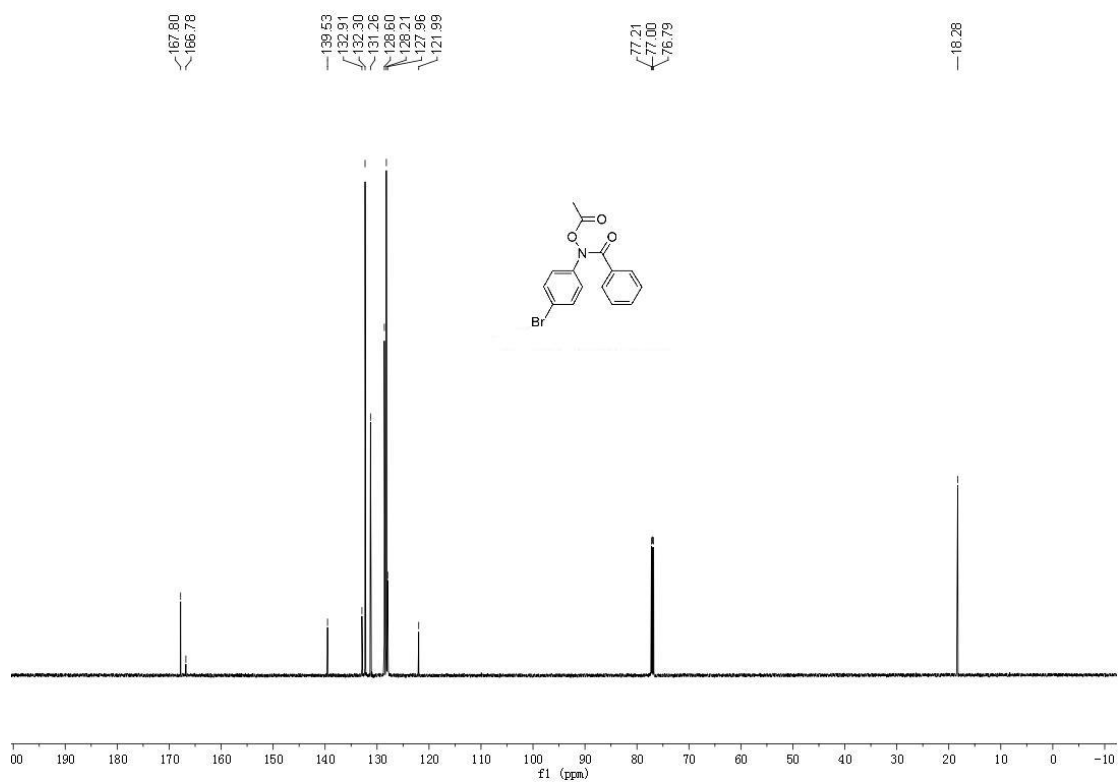
4r



¹H NMR copies of compound **4x**:



¹³C NMR copies of compound **4x**:

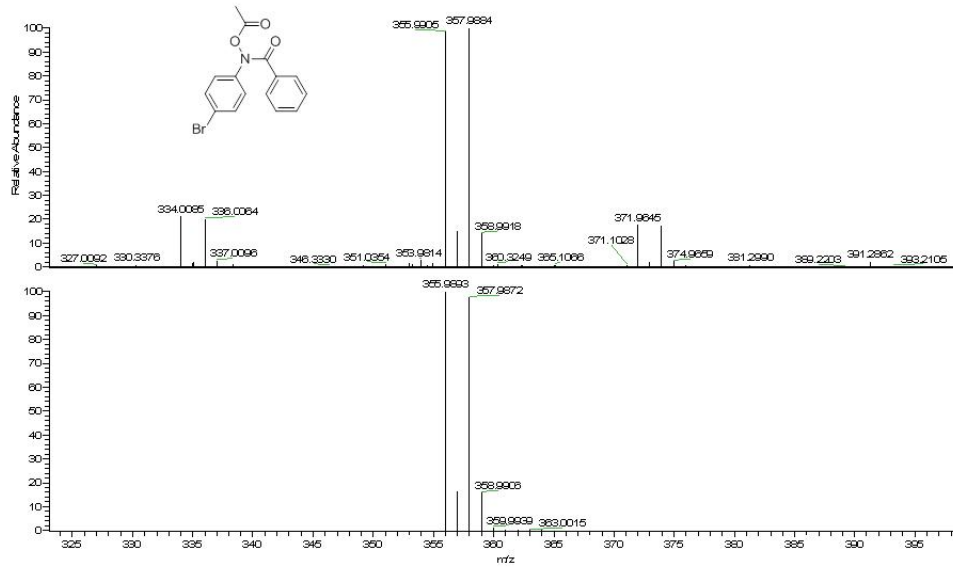


HRMS (ESI) of compound **4x**:

F:\User\zhanghujuan_2_15030143550

9/30/2015 2:43:51 PM
Error=8.4 ppm

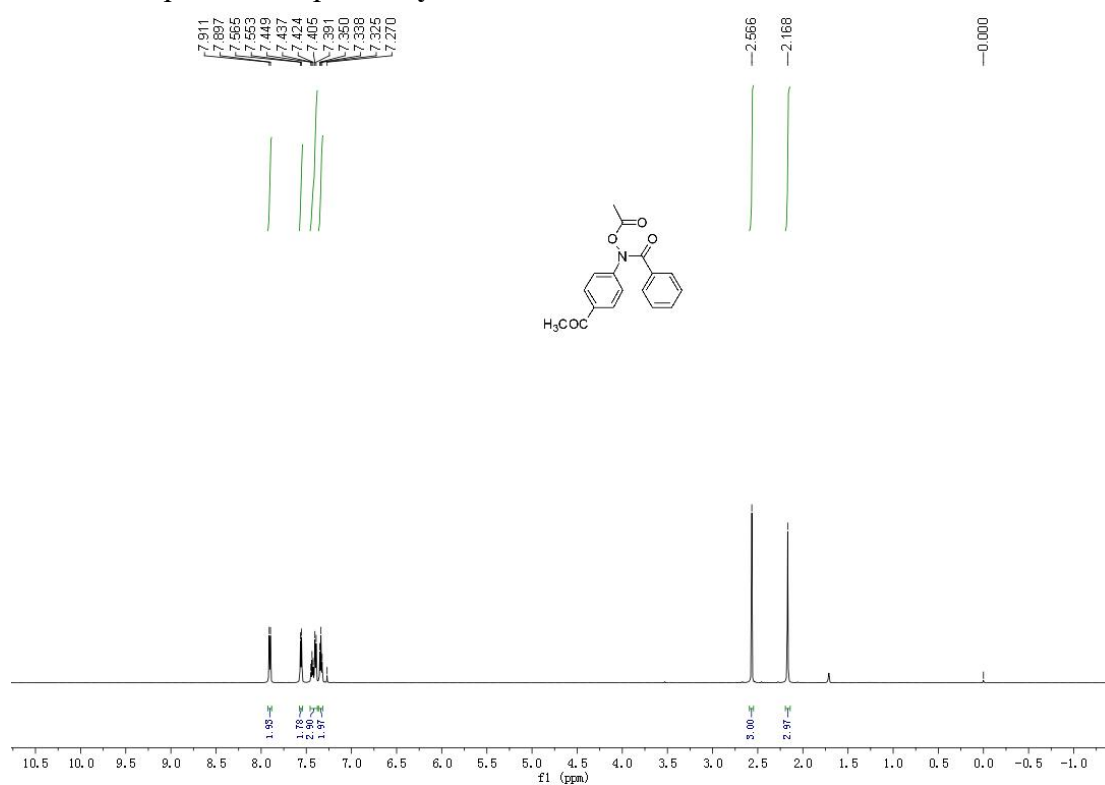
2#



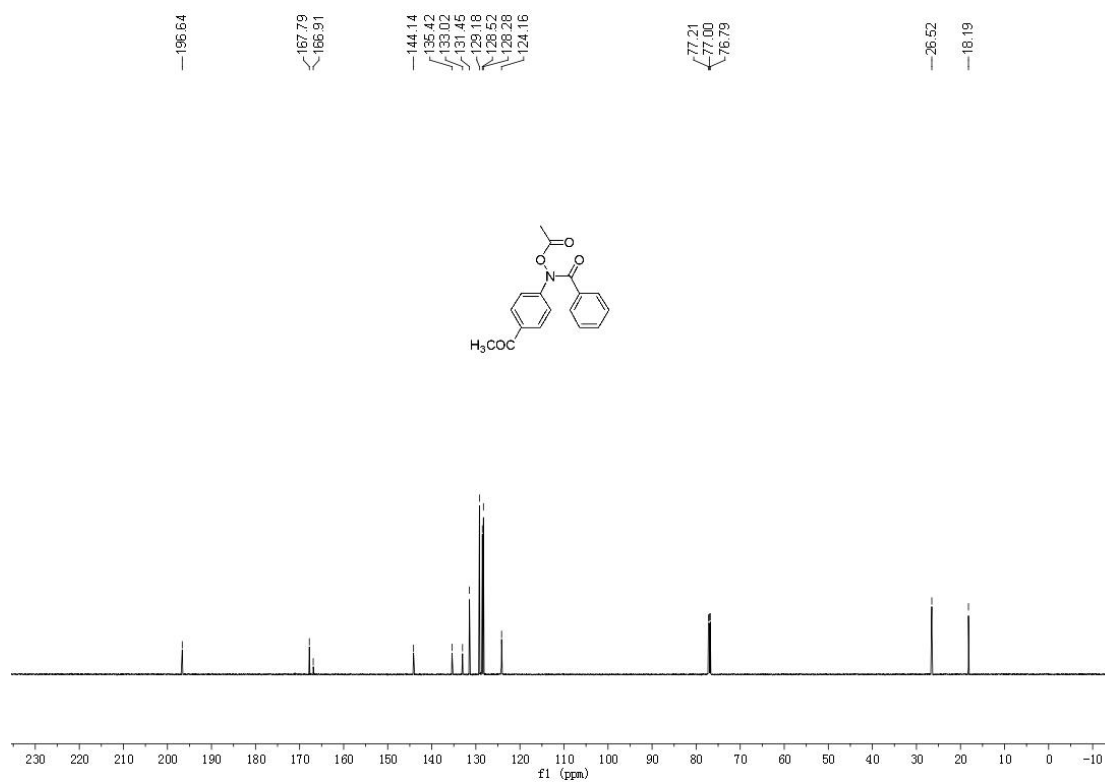
NL:
4.26E5
zhanghujuan
2_15030143550W21
RT: 0.18 AV: 1 T: FTMS
+p ESI Full ms
(100.00-300.00)

NL:
4.26E5
C₁₅H₁₂Br₁N₁O₃+Na₁
C₁₅H₁₂Br₁N₁O₃N₁
pa Chg 1

¹H NMR copies of compound **4y**:



¹³C NMR copies of compound **4y**:

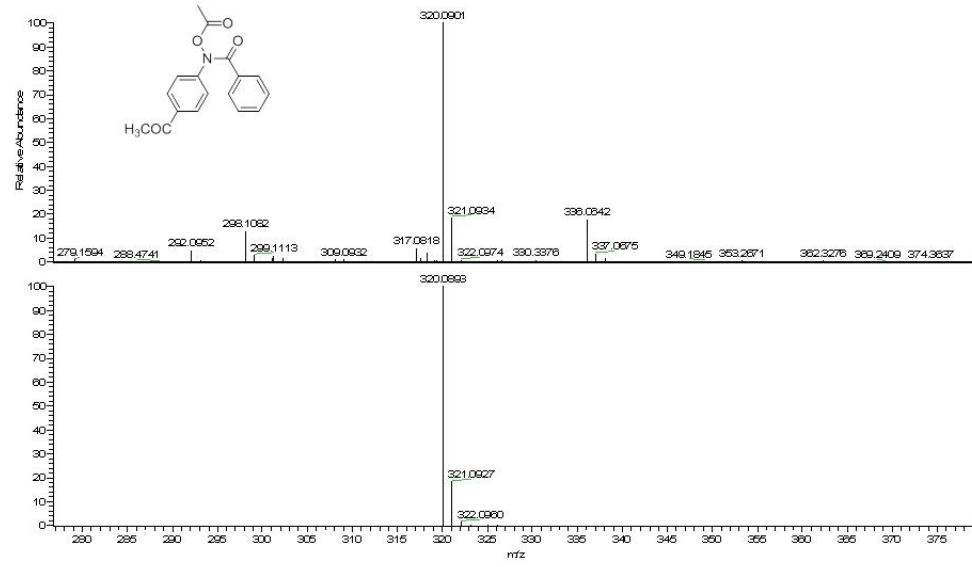


HRMS (ESI) of compound **4y**:

F:\UserA\zhanghujuan_5_150930145954

9/30/2015 3:09:50 PM
Err=2.5 ppm

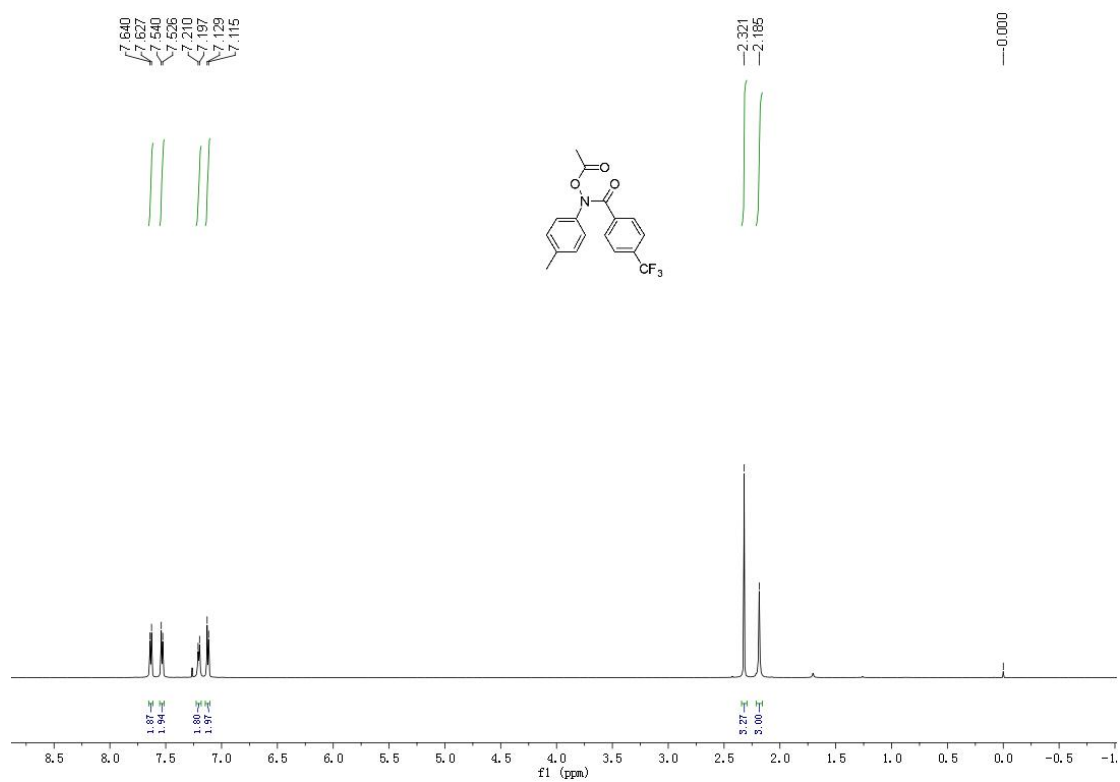
5#



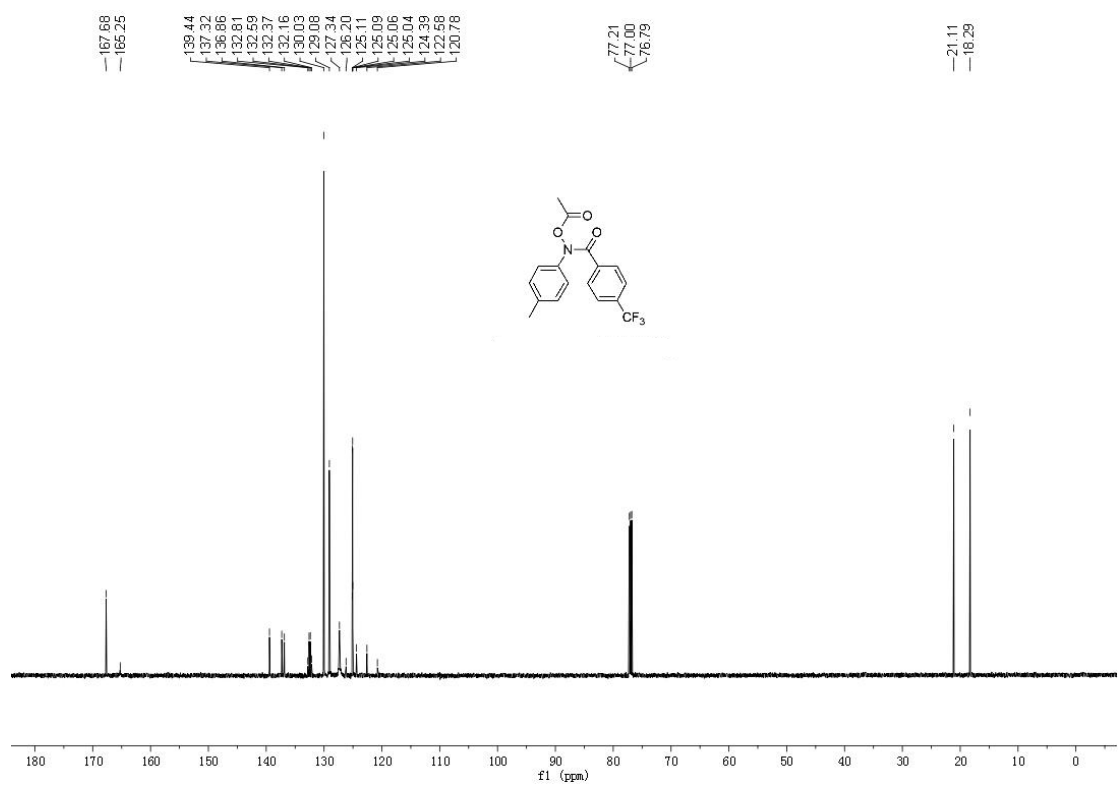
NL:
3.14E5
zhanghujuan-
5_150930145954#09
RT: 0.28 AV: 1 T:
FTMS +p ES Full ms
[100.00-300.00]

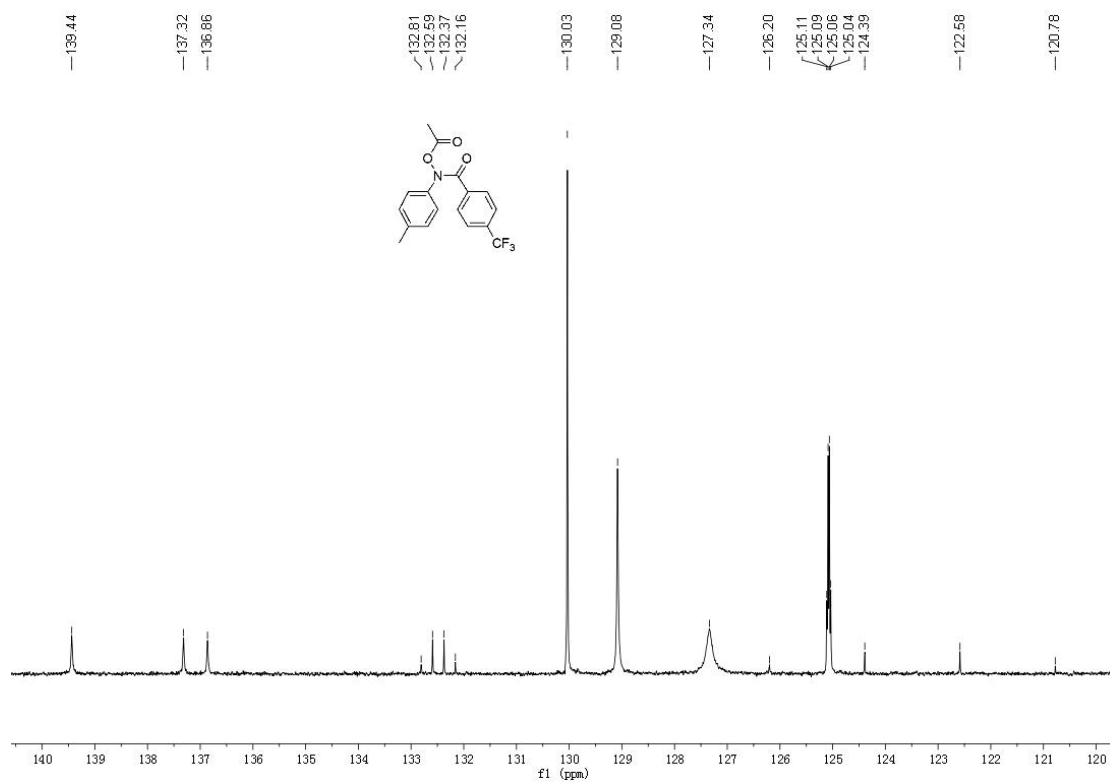
NL:
8.20E5
C₁₇H₁₅N₁O₁#Na:
C₁₇H₁₅N₁O₁Na₁
ps-Chrg 1

¹H NMR copies of compound **4ac**:

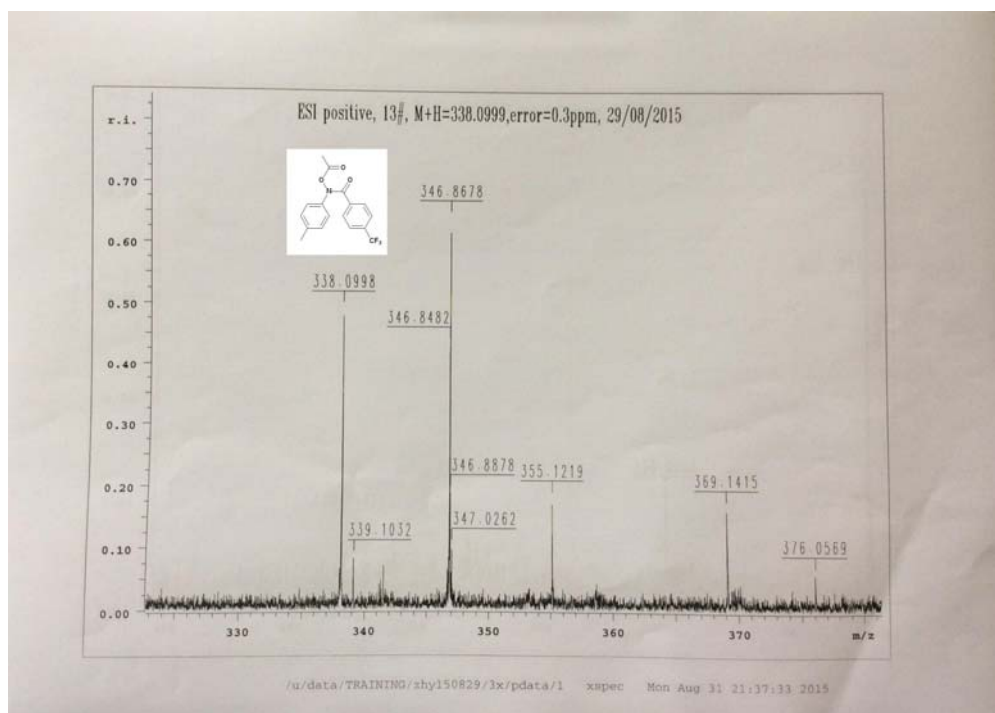


¹³C NMR copies of compound **4ac**:

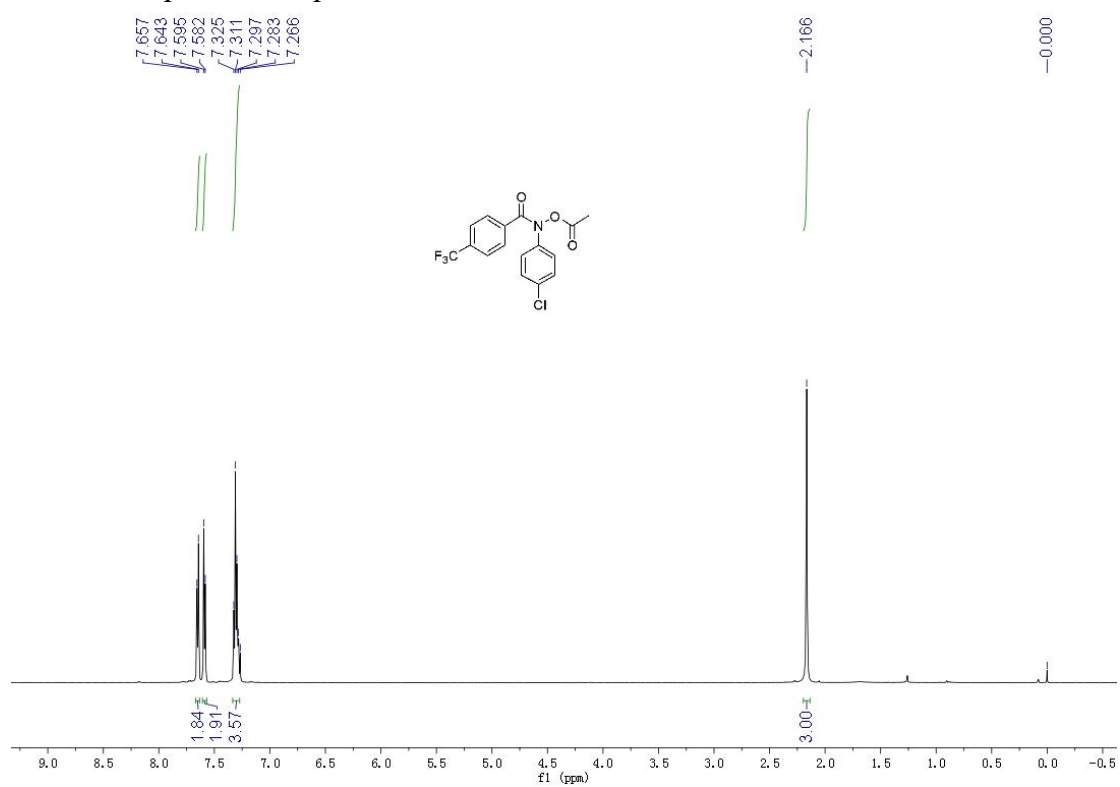




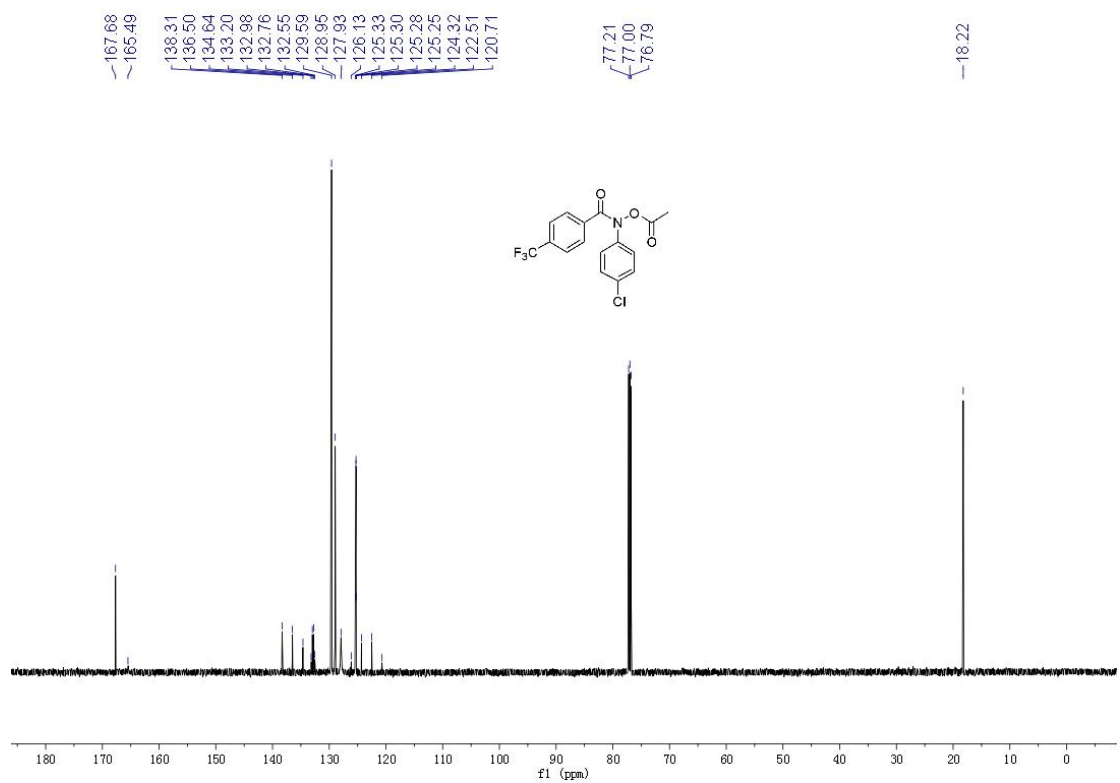
HRMS (ESI) of compound **4ac**:

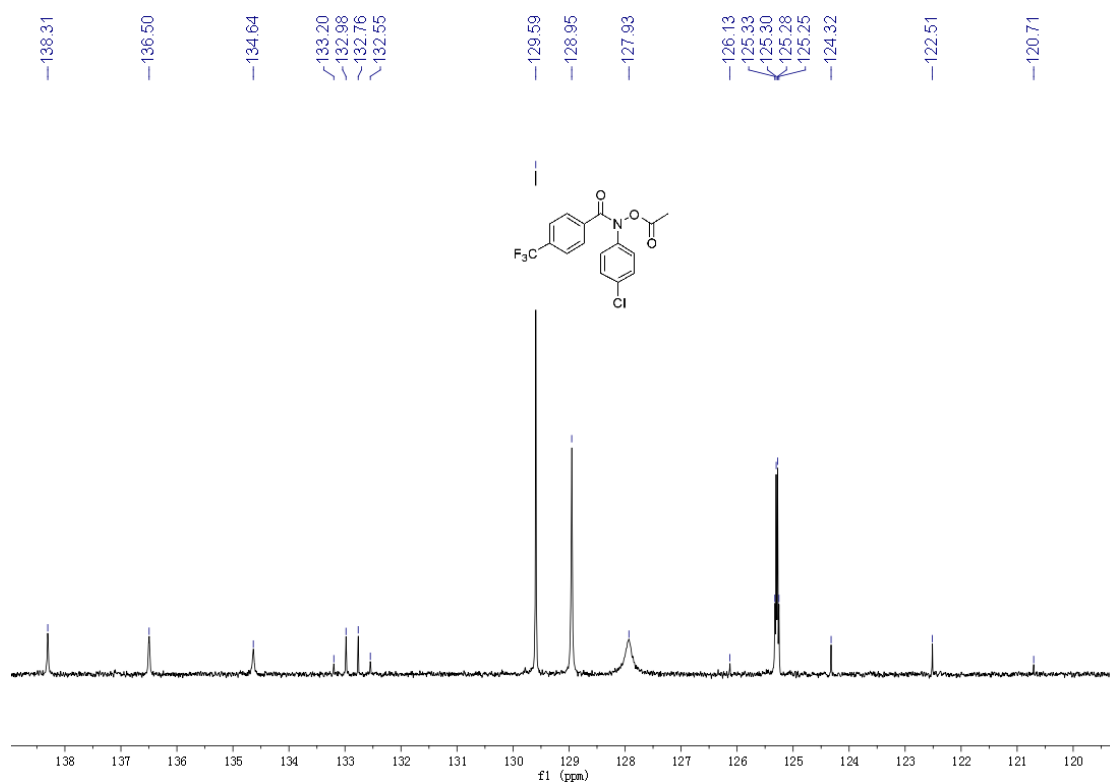


¹H NMR copies of compound **4ad**:

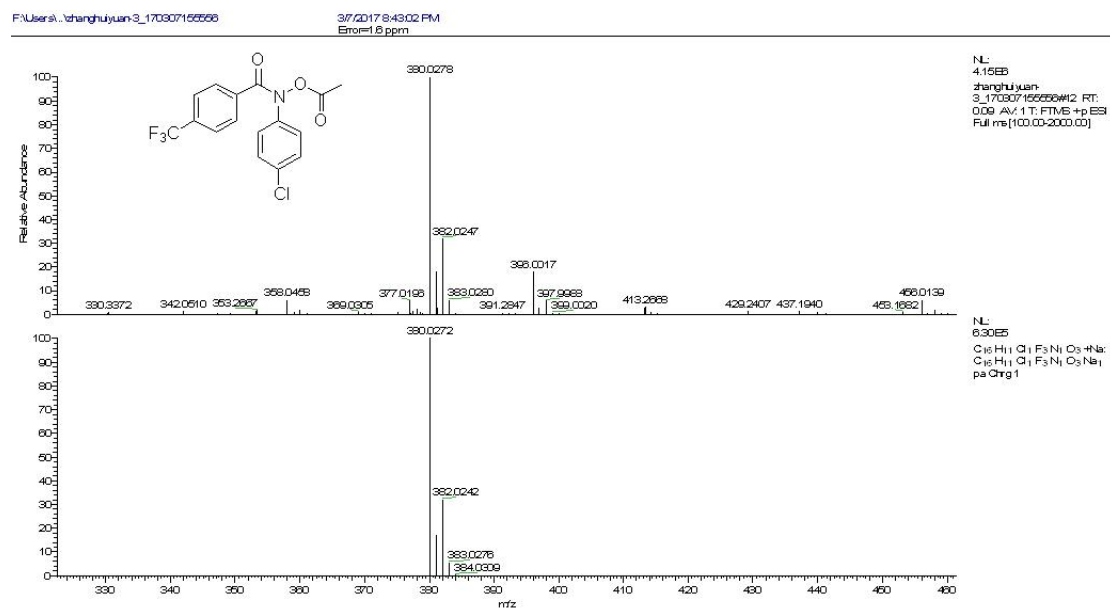


¹³C NMR copies of compound **4ad**:

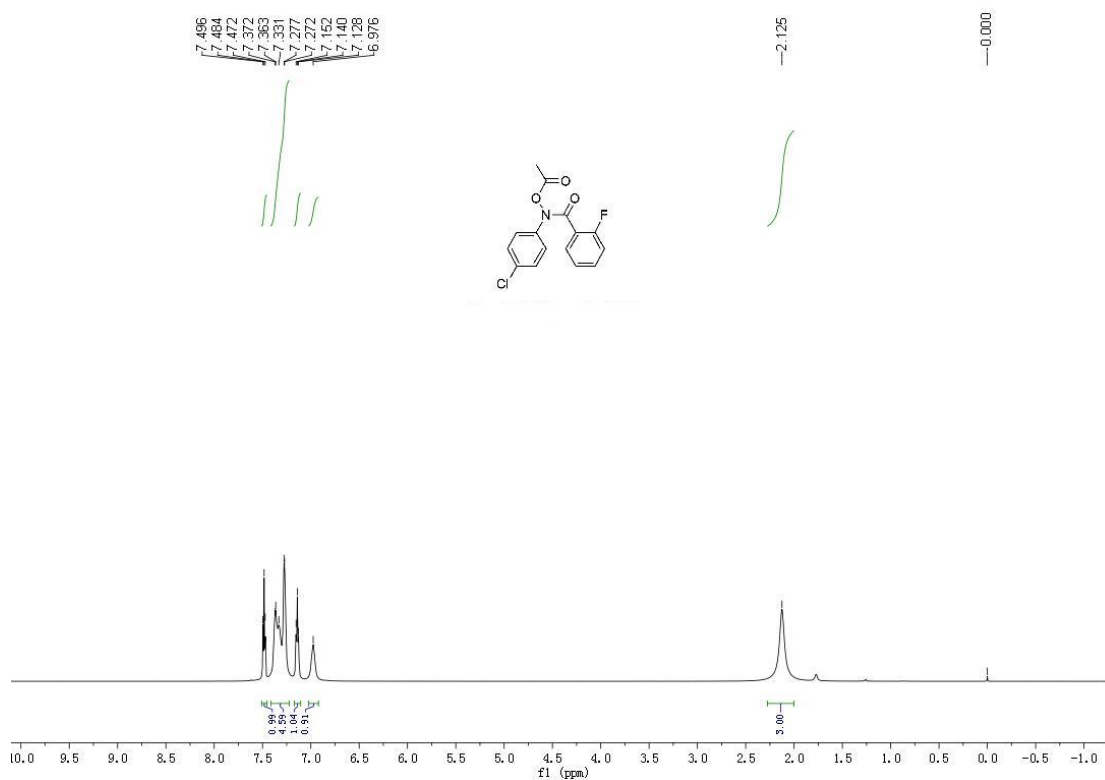




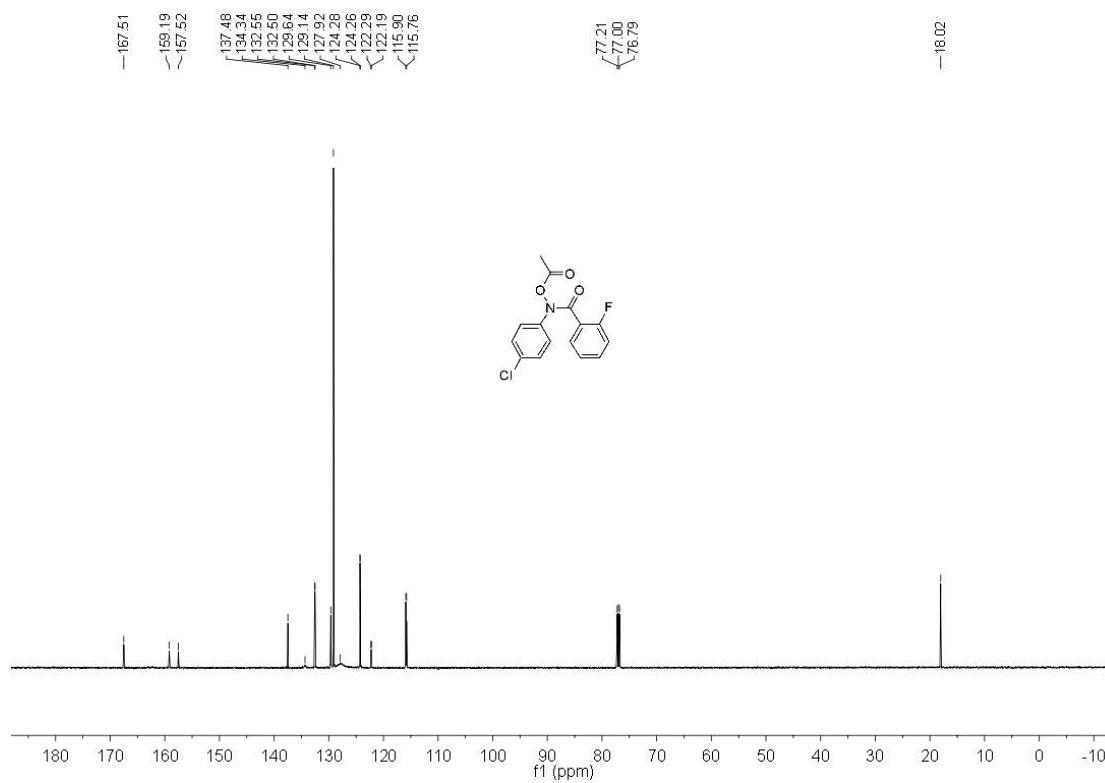
HRMS (ESI) of compound 4ad:

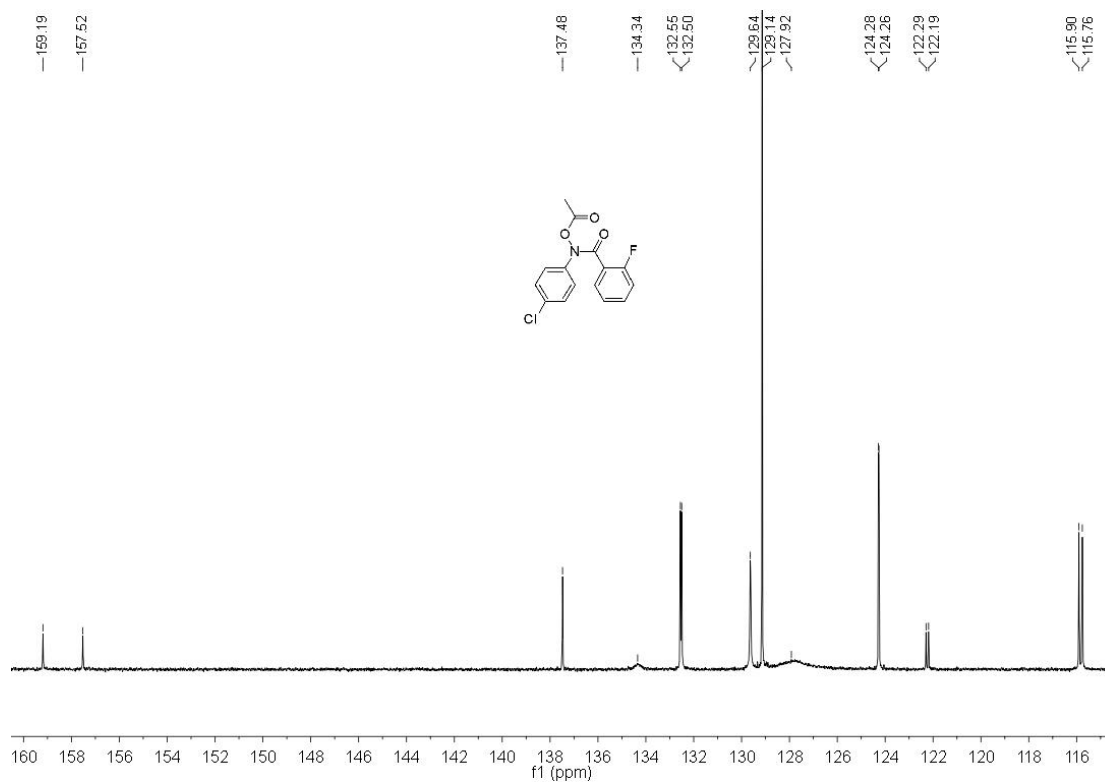


¹H NMR copies of compound **4ae**:

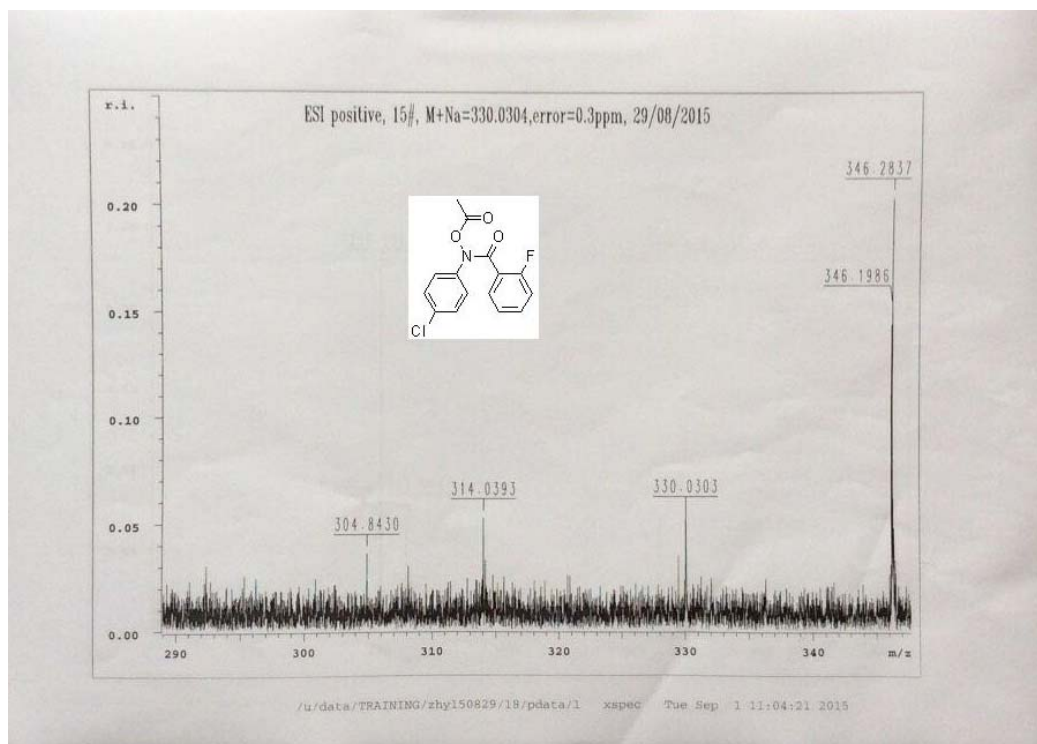


¹³C NMR copies of compound **4ae**:

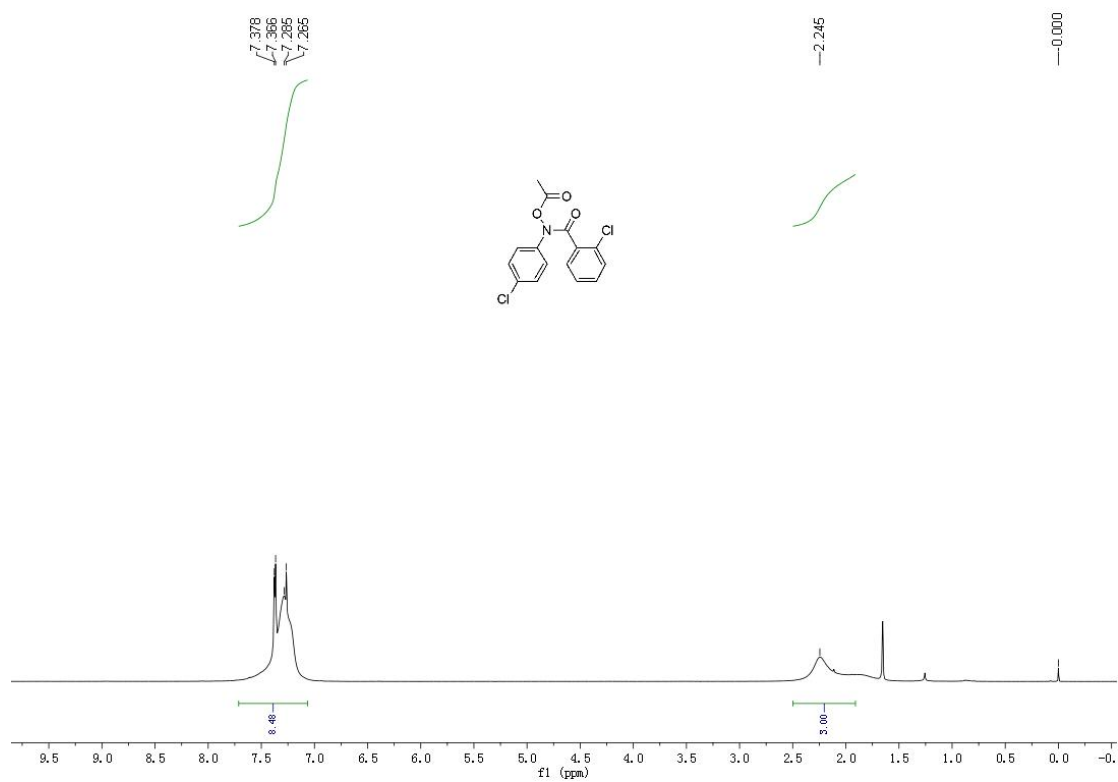




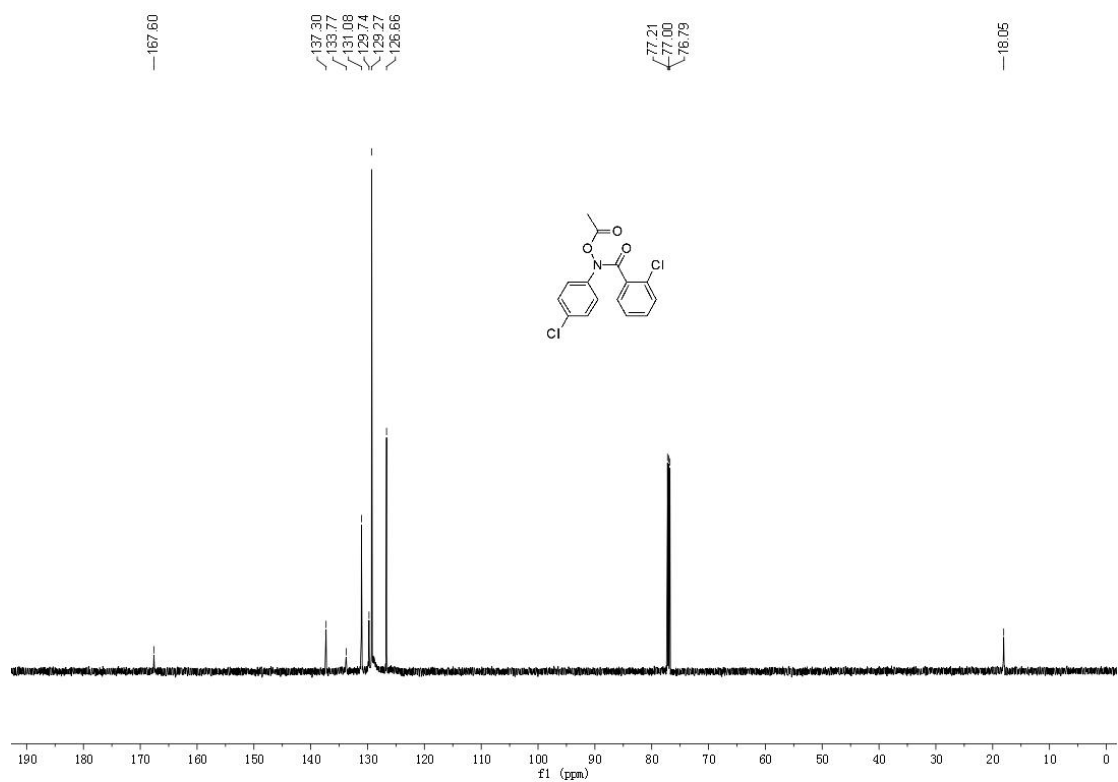
HRMS (ESI) of compound **4ae**:



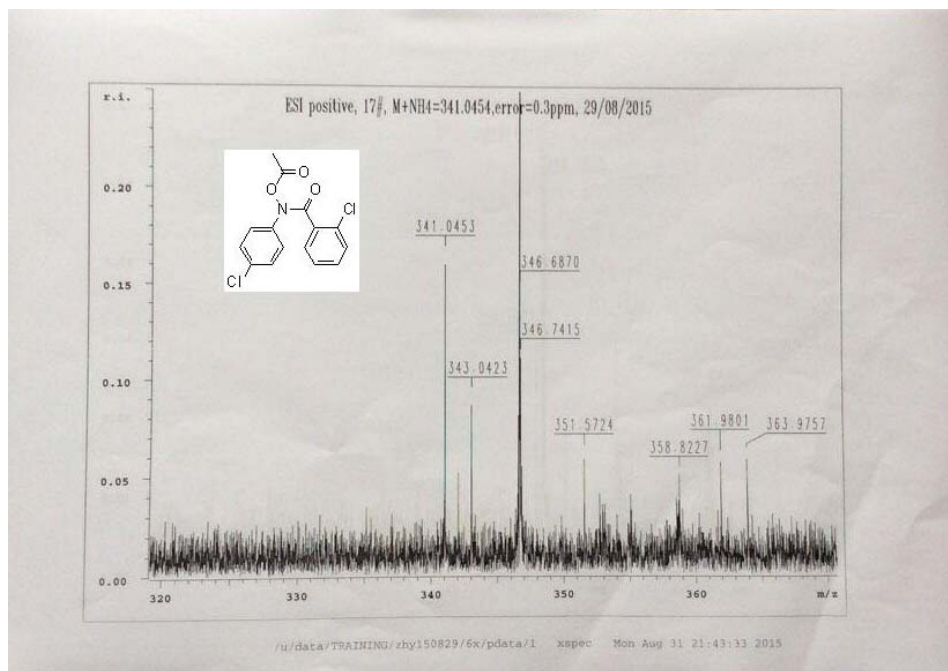
¹H NMR copies of compound **4af**:



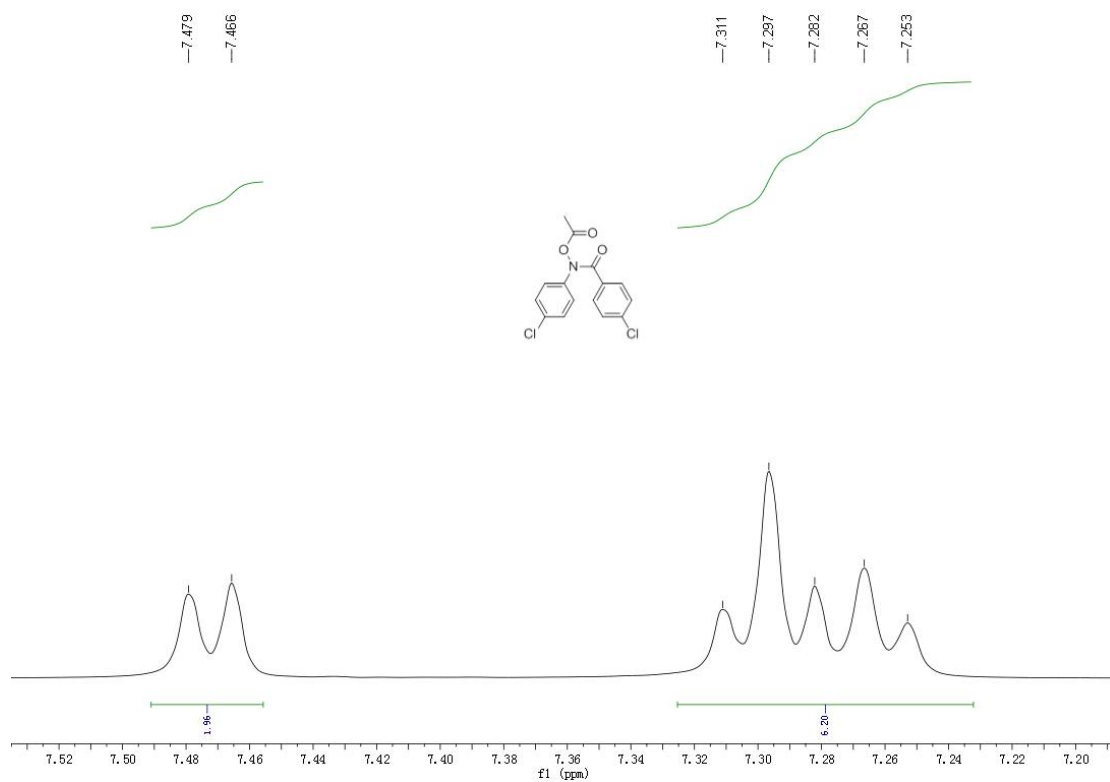
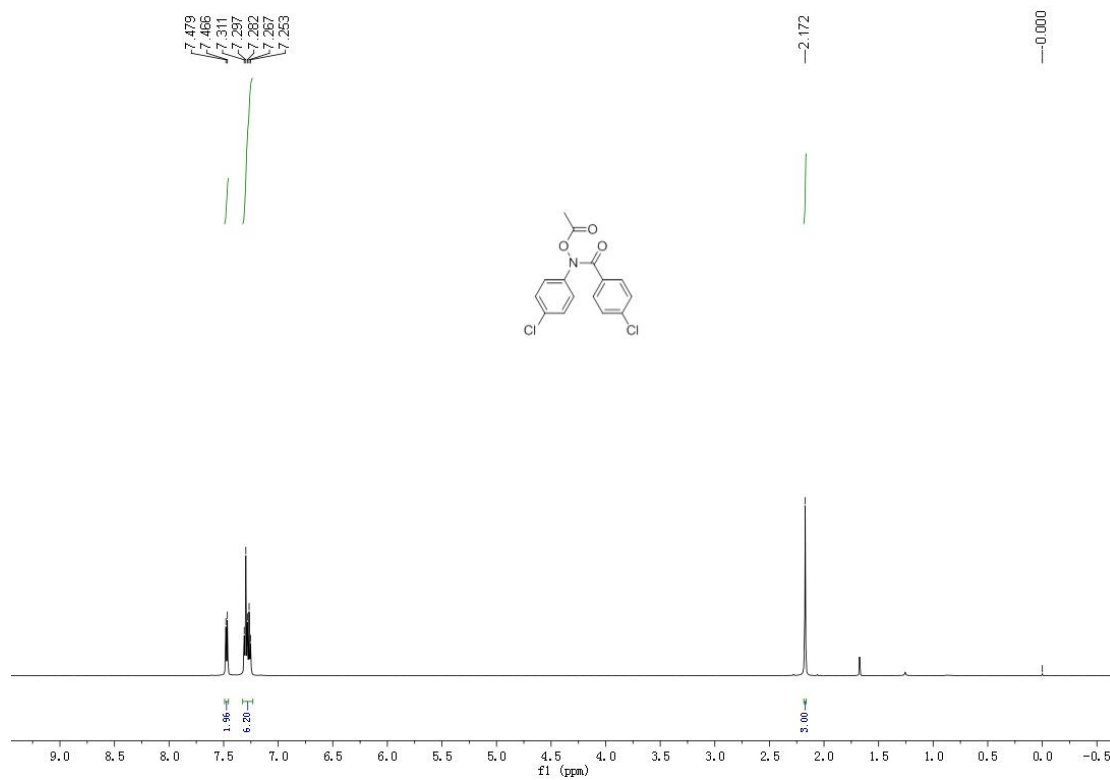
¹³C NMR copies of compound **4af**:



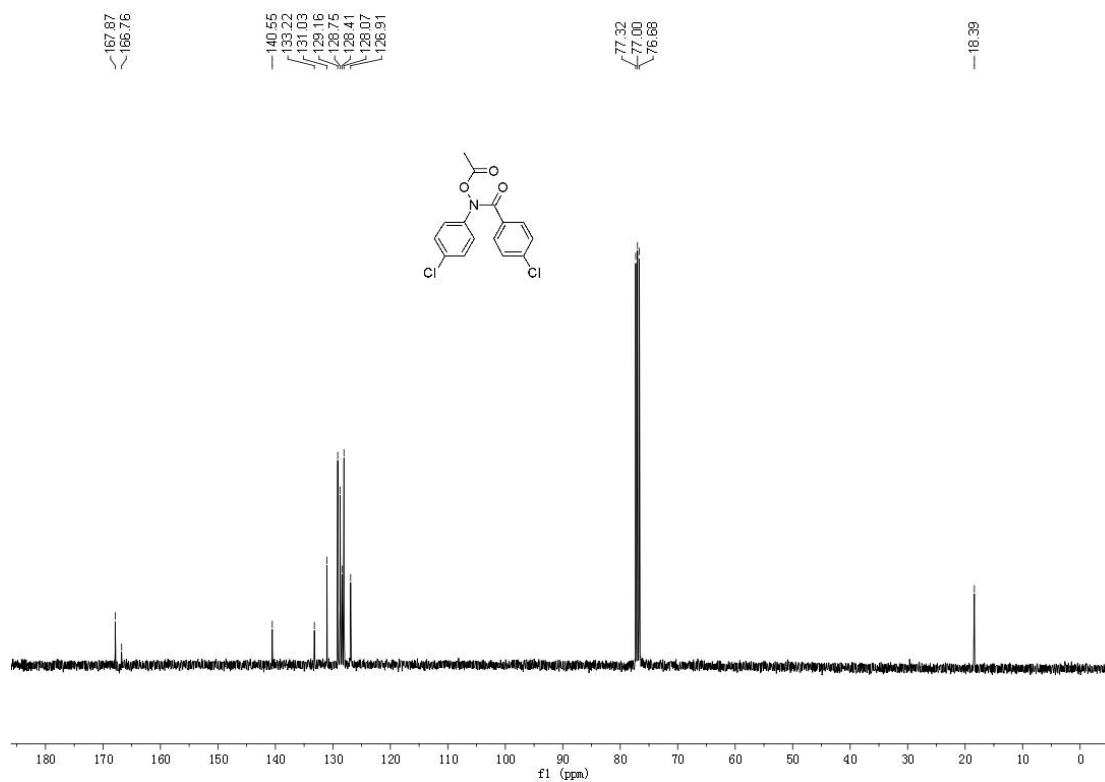
HRMS (ESI) of compound **4af**:



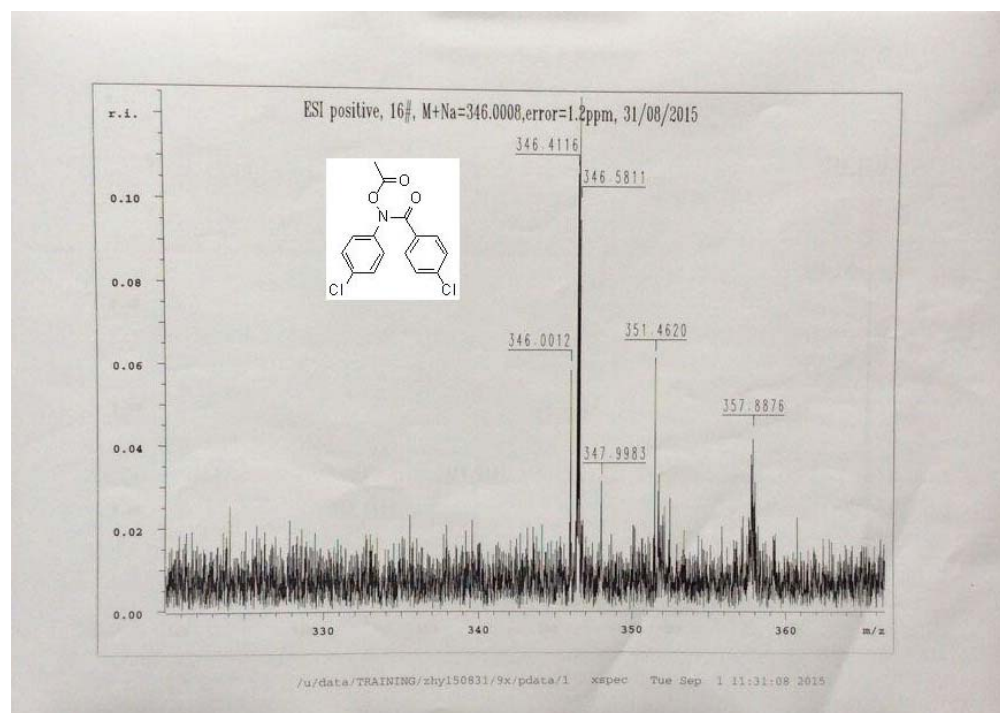
¹H NMR copies of compound **4ag**:



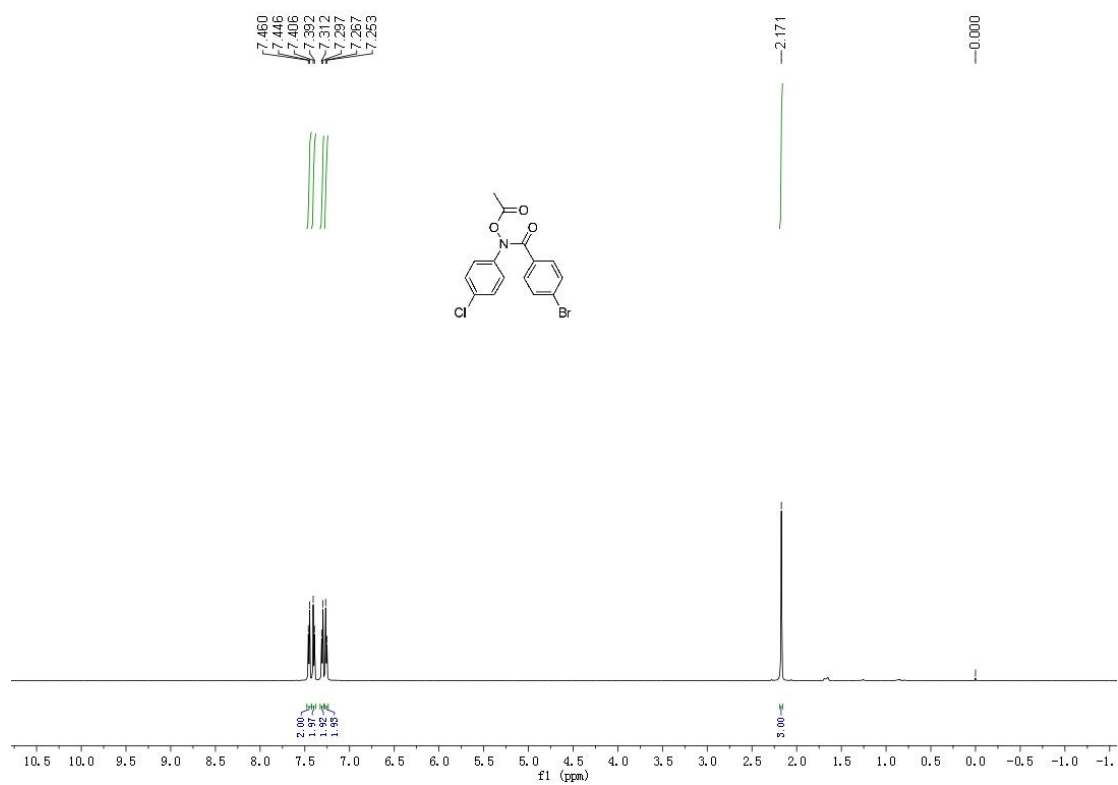
¹³C NMR copies of compound **4ag**:



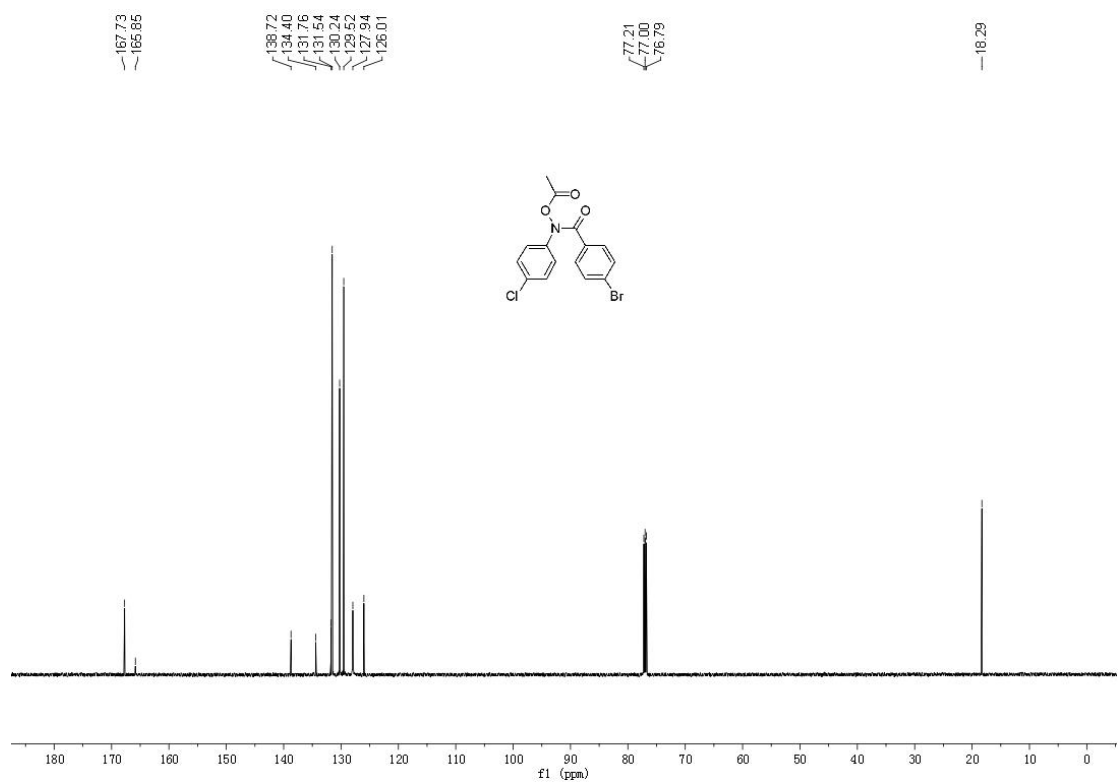
HRMS (ESI) of compound **4ag**:



¹H NMR copies of compound **4ah**:



¹³C NMR copies of compound **4ah**:

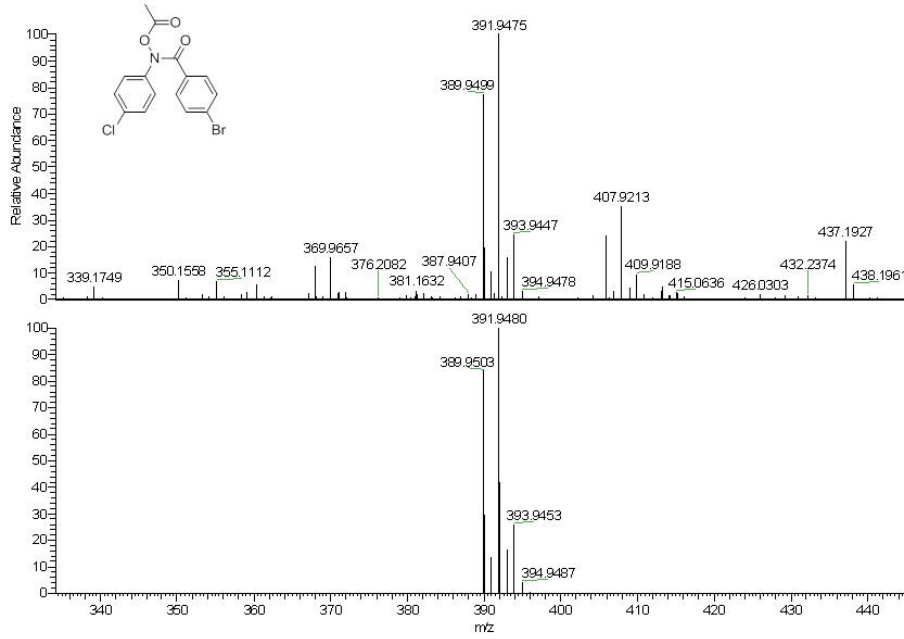


HRMS (ESI) of compound **4ah**:

D:\Users\...zhanhuaiyuan-3_150911155504

9/11/2015 4:08:43 PM
Error=1.0ppm

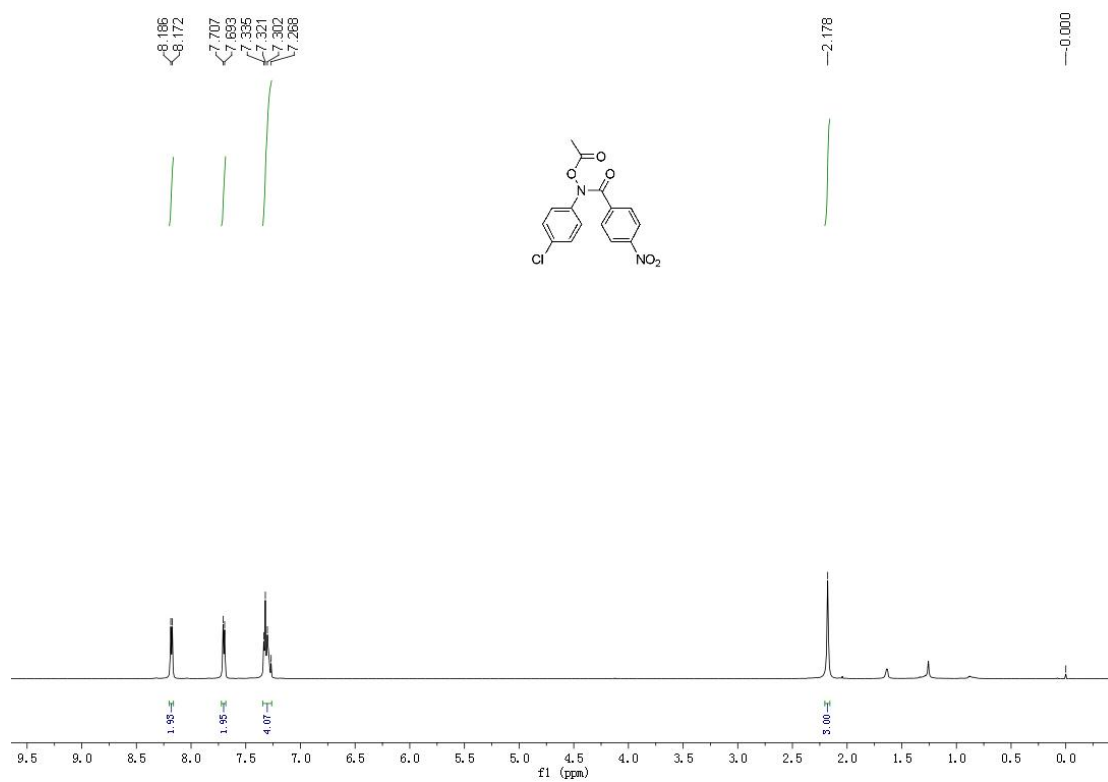
3#



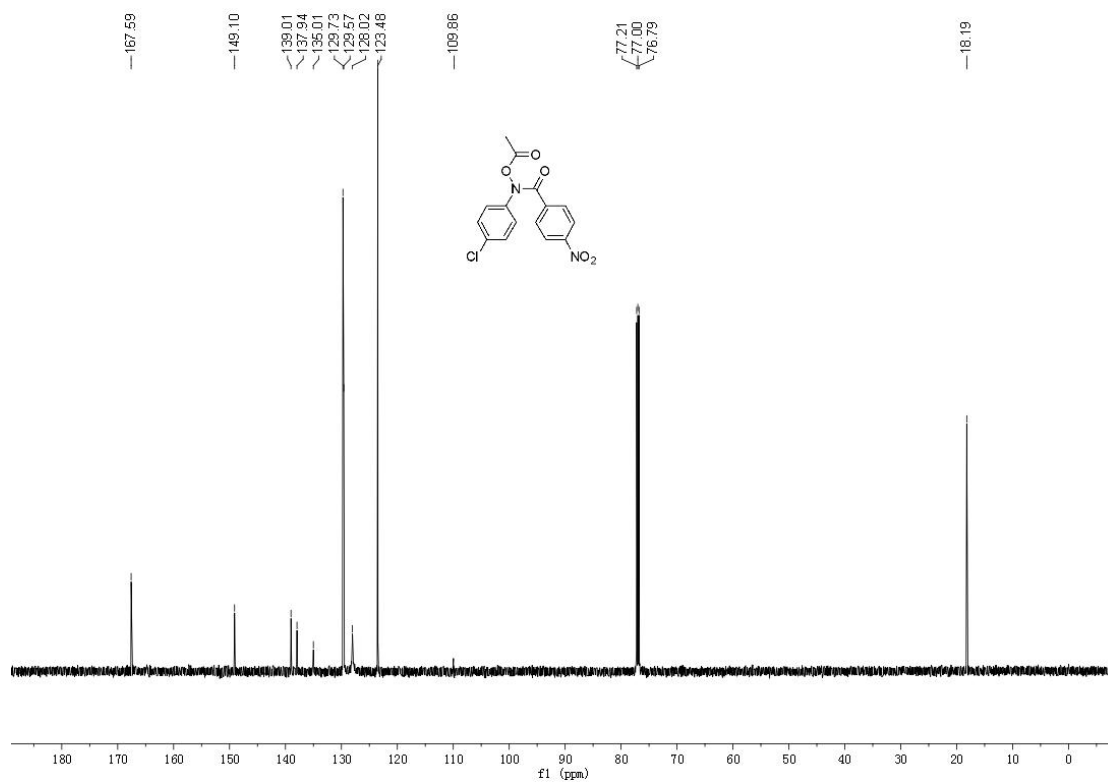
NL
1.83E5
zhanhuaiyuan-
3_150911155504#5 RT:
0.04 AV: 1.T: FTMS + p ESI
Full ms [100.00-2000.00]

NL
9.61E3
C₁₅H₁₁Br₁Cl₁N₁O₃+Na
C₁₅H₁₁Br₁Cl₁N₁O₃Na
p (b1 s/p40) Chrg 1
R: 0.003 Da @FWHM

¹H NMR copies of compound **4ai**:



¹³C NMR copies of compound **4ai**:

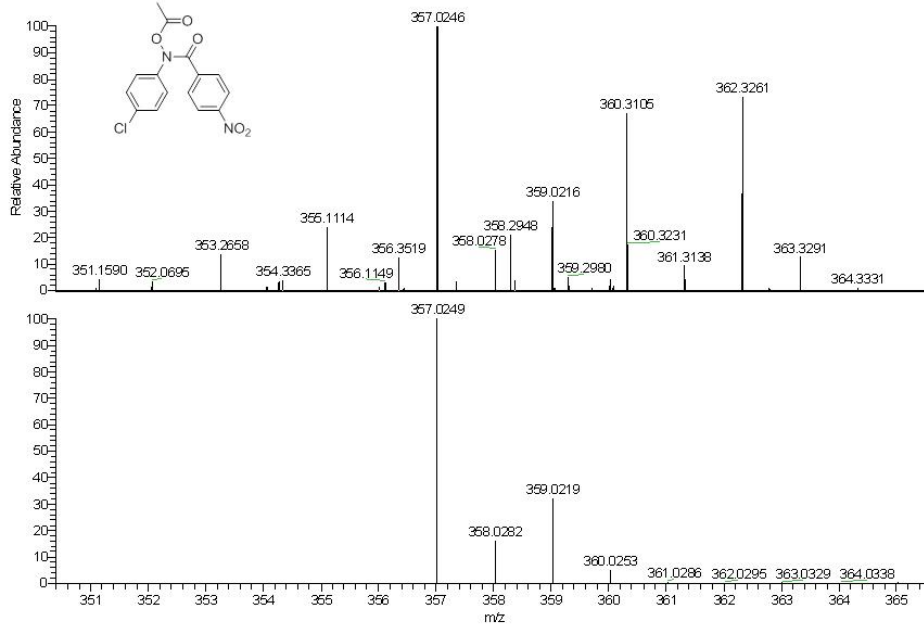


HRMS (ESI) of compound **4ai**:

D:\Users\...zhanhuaiyuan-1_150911155504

9/11/2015 3:55:05 PM
Error=0.8 ppm

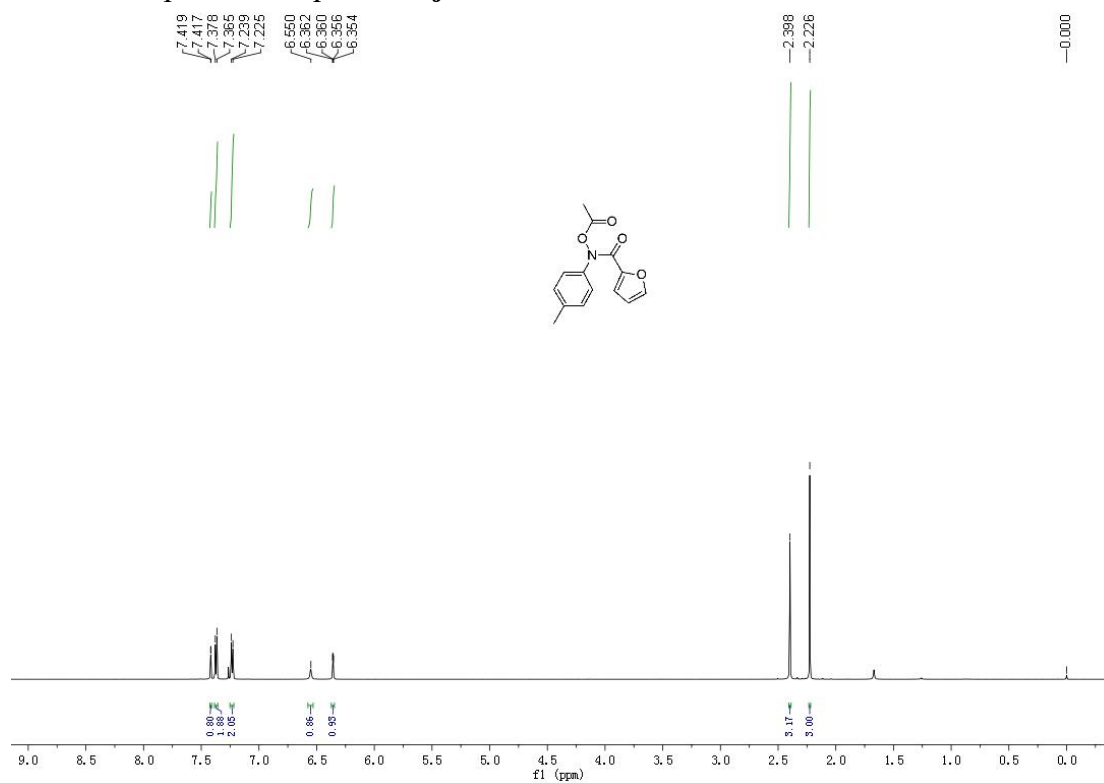
1#



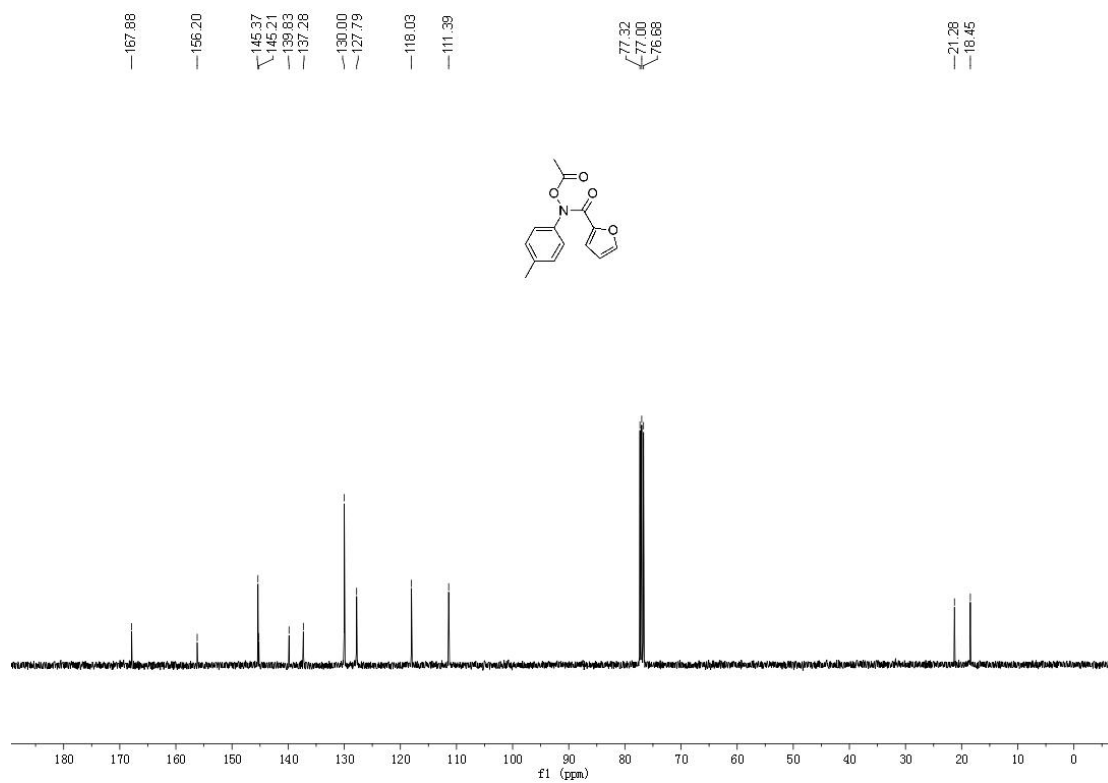
NL:
1.09E6
zhanhuaiyuan
1_150911155504#6 RT:
0.03 AV: 1 T: FTMS +p
ESI Full ms
[100.00-2000.00]

NL:
6.32E5
C15 H11 Cl1 N2 O5 +Na
C15 H11 Cl1 N2 O5 Na1
pa Chrg 1

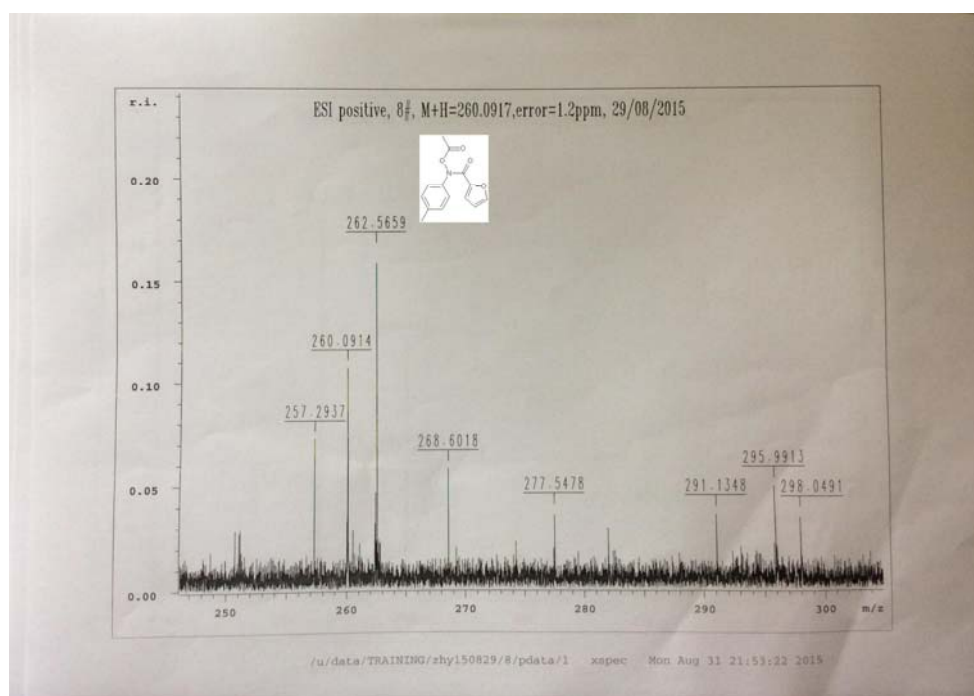
¹H NMR copies of compound **4aj**:



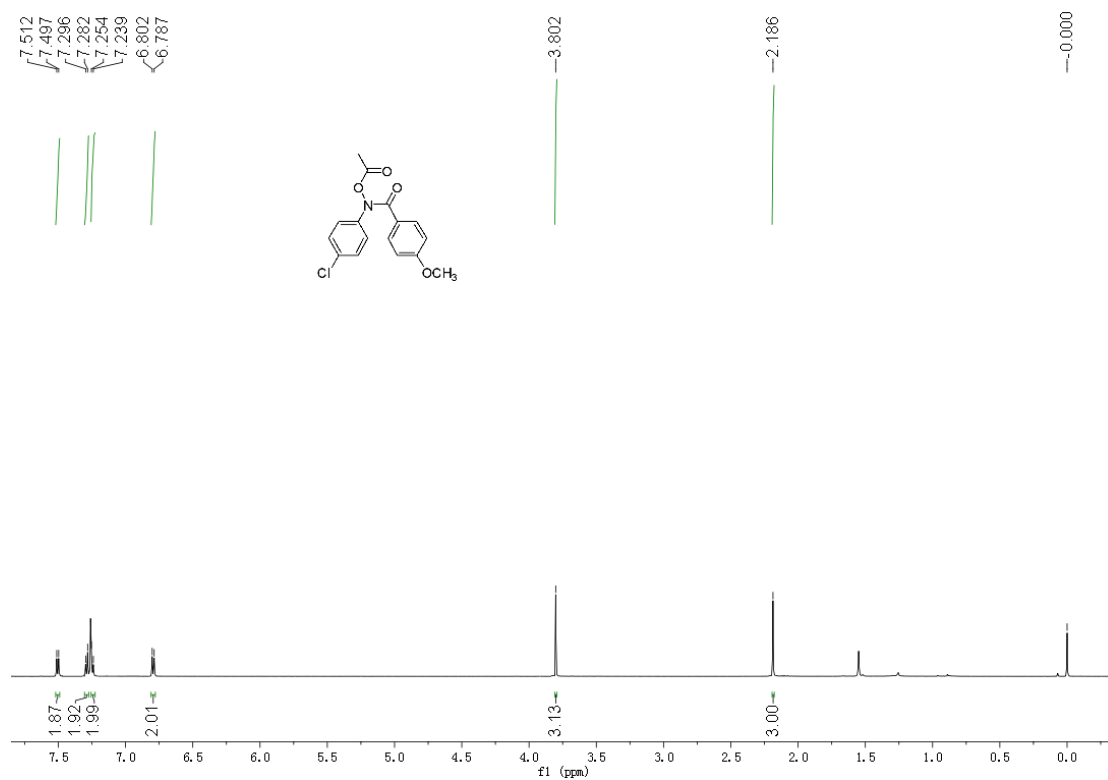
¹³C NMR copies of compound **4aj**:



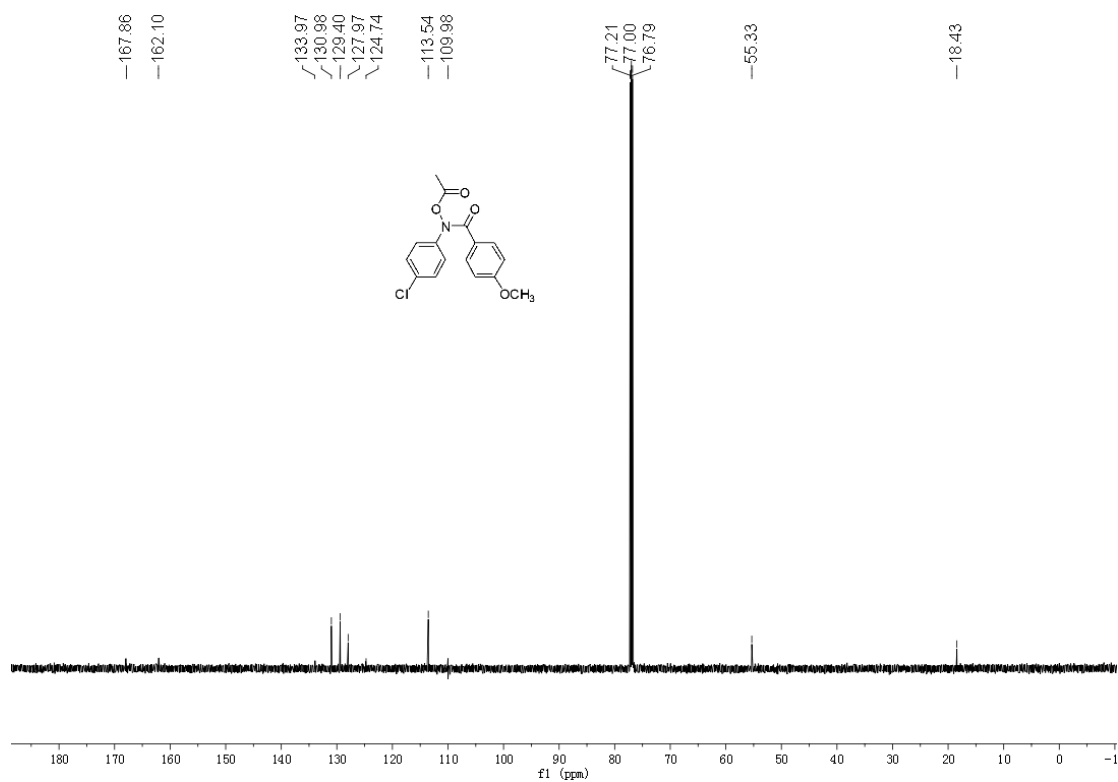
HRMS (ESI) of compound **4aj**:



¹H NMR copies of compound **4ak**:



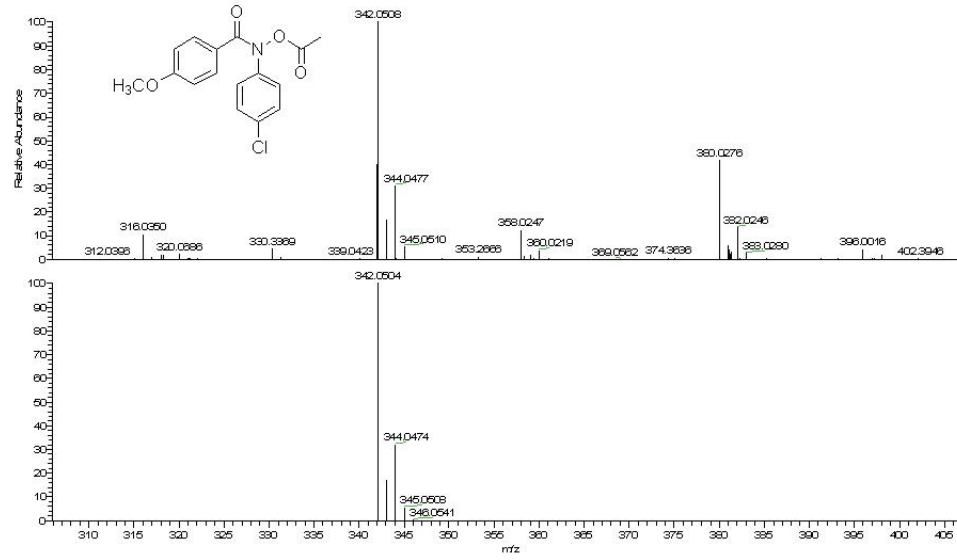
¹³C NMR copies of compound **4ak**:



HRMS (ESI) of compound **4ak**:

F:\User\zhanghuijun_4_170307155558

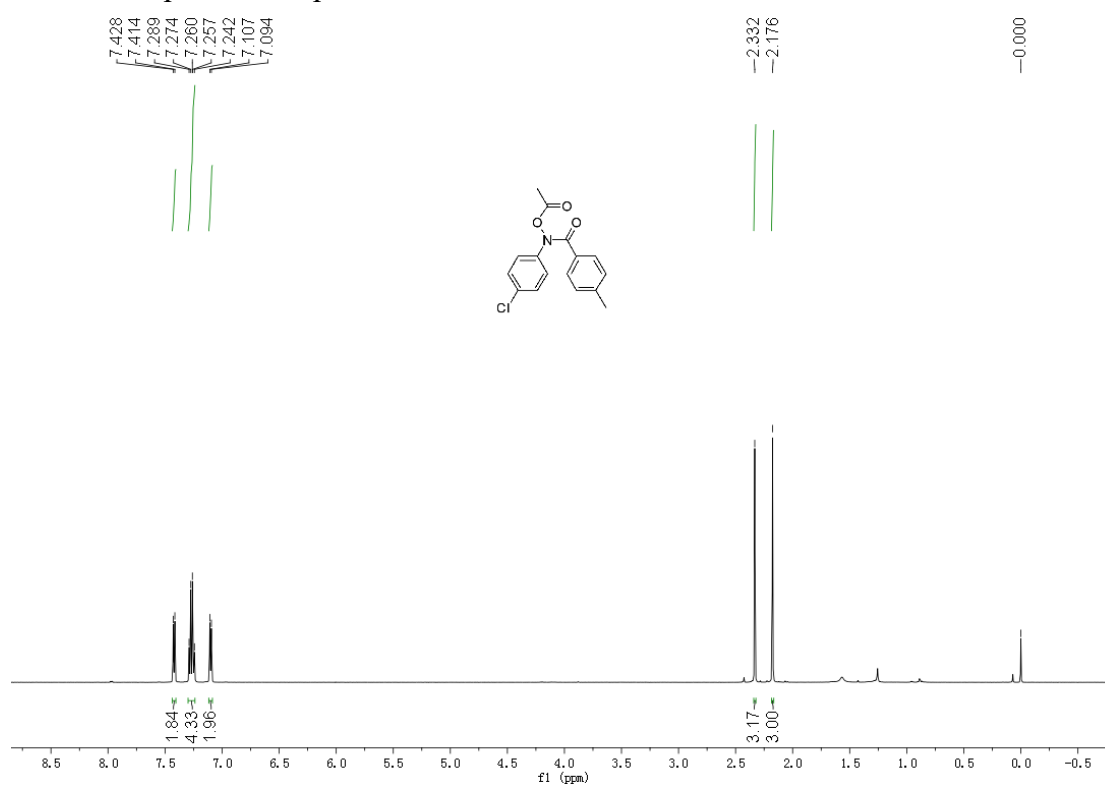
3/7/2017 5:17:02 PM
Error! 2 ppm



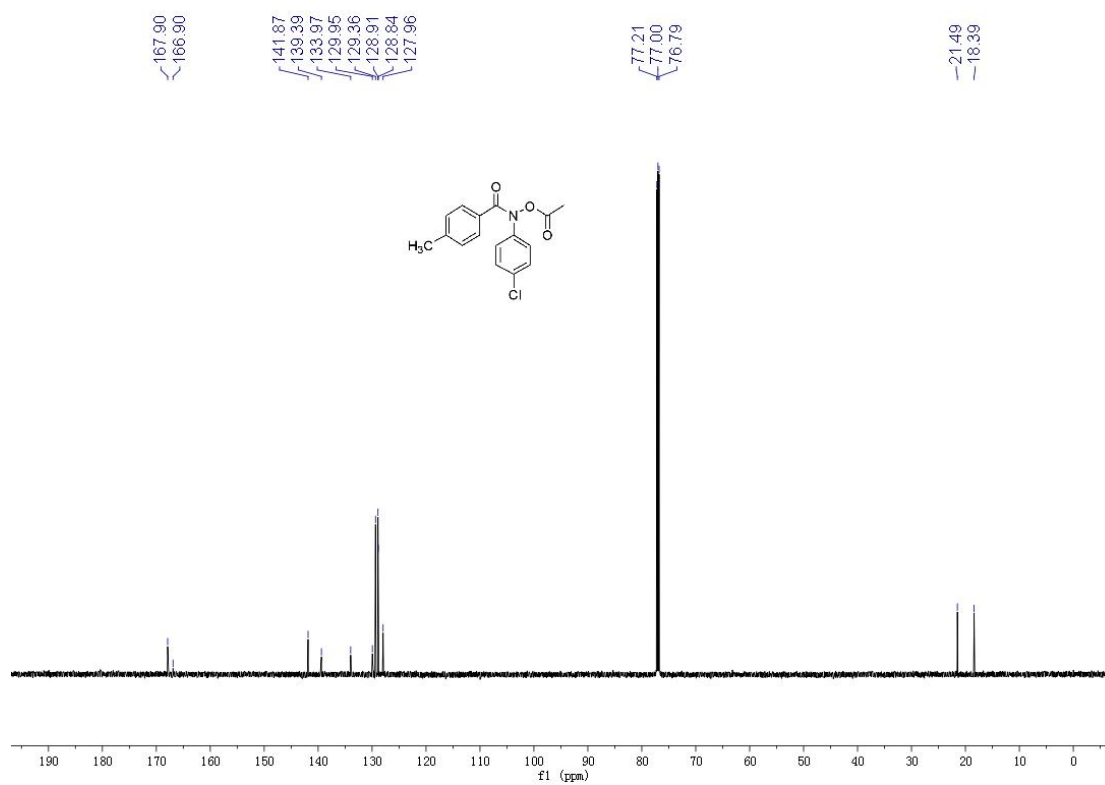
NL:
6.05E5
zhanghuijun
4_170307155558W17
RT: 0.12, AV: 1 T: FTMS
+p ESI Full MS
(100.00-2000.00)

NL:
6.28E5
C16 H14 Cl1 N1 O2 +Na+
C16 H14 Cl1 N1 O2 Na+
pa C1rg 1

¹H NMR copies of compound 4al:



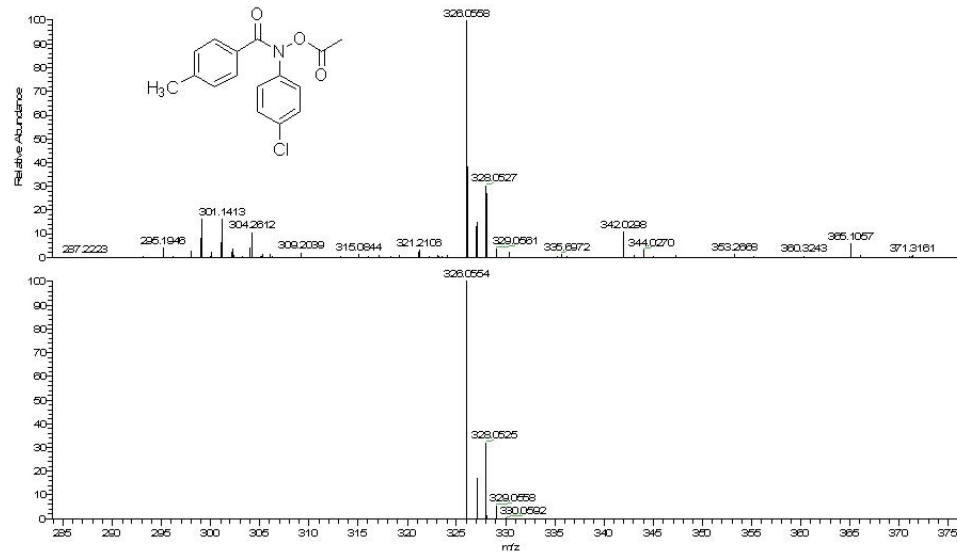
¹³C NMR copies of compound 4al:



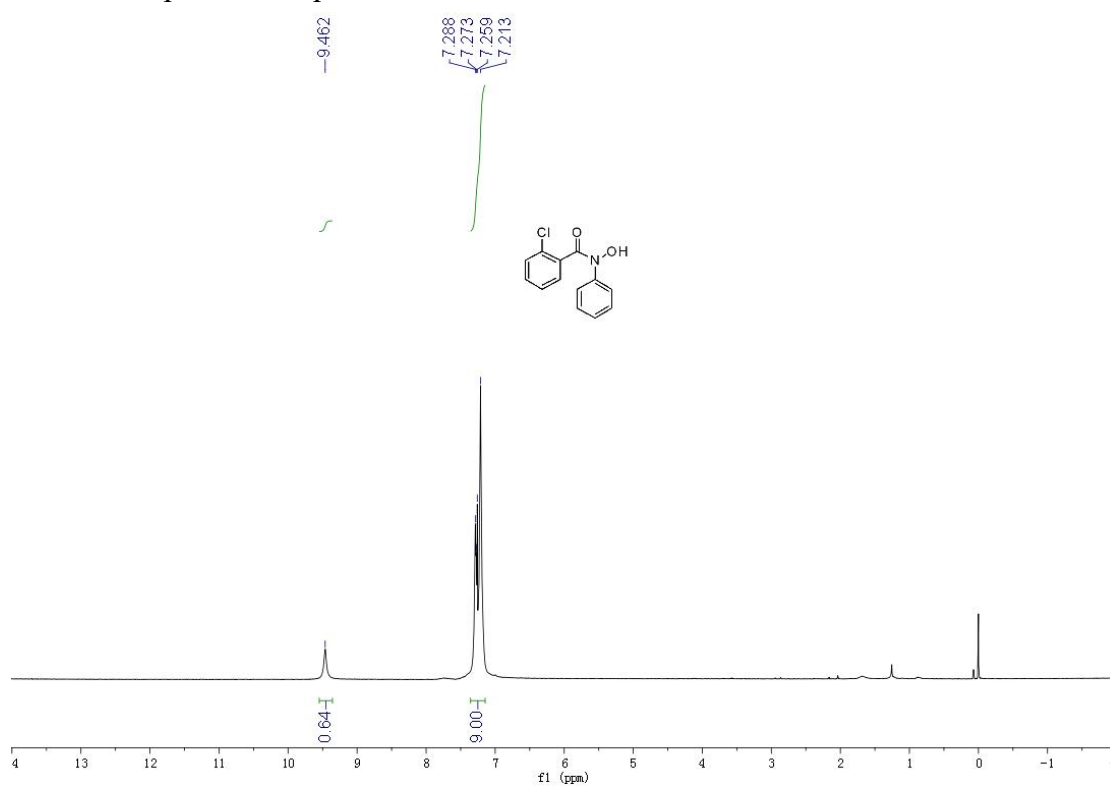
HRMS (ESI) of compound **4al**:

F:\User\zhanghuiyuan_5_170307155558

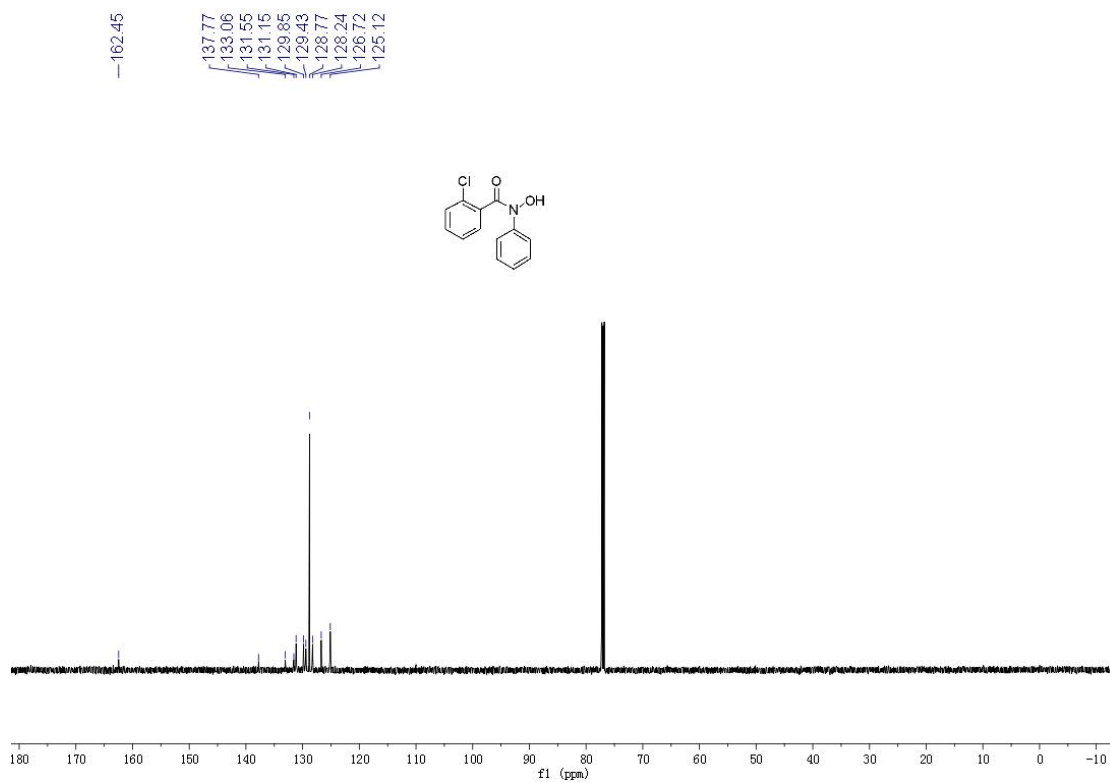
3/7/2017 8:48:02 PM
Error! 2 ppm

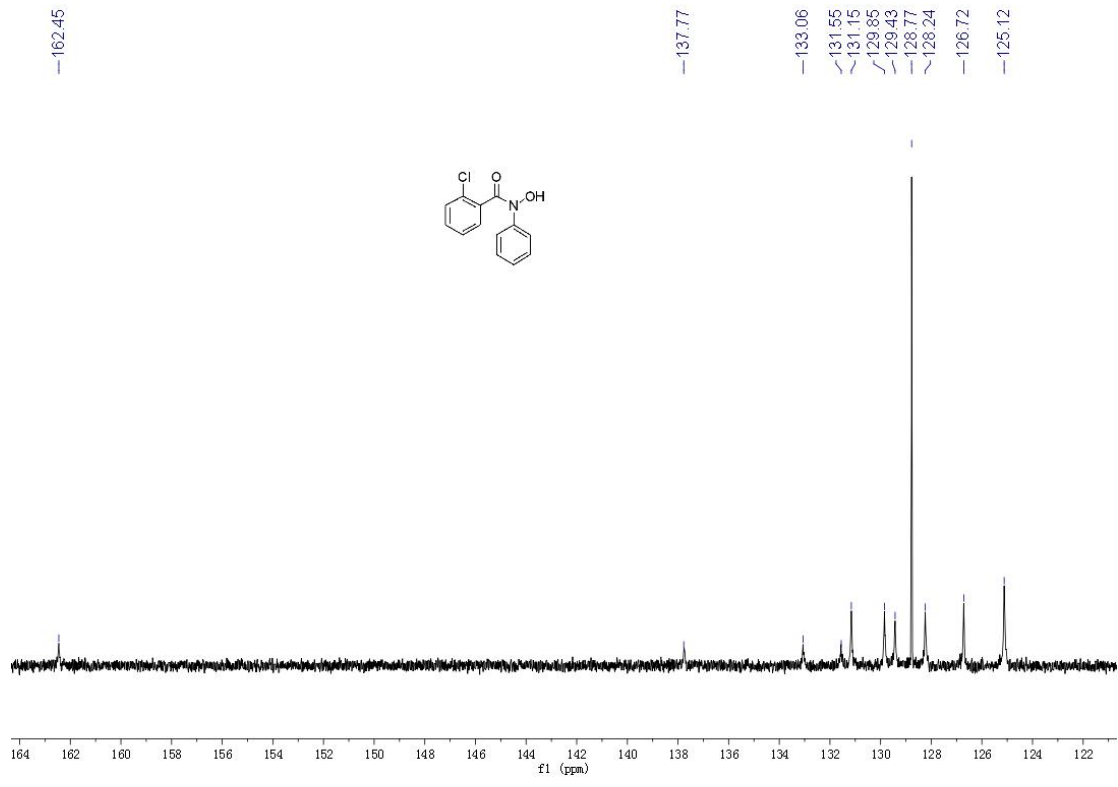


^1H NMR copies of compound **5**:

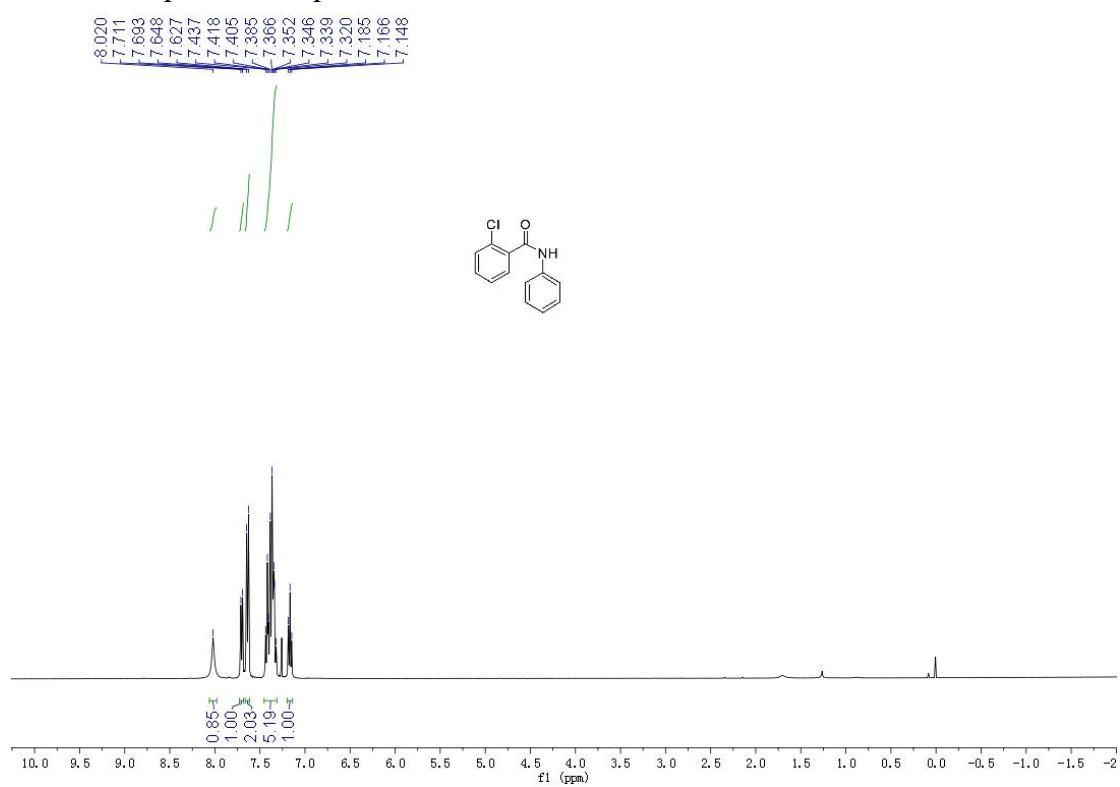


^{13}C NMR copies of compound **5**:





¹H NMR copies of compound 6:



¹³C NMR copies of compound 6:

