Redox-neutral rhodium(III)-catalyzed chemo- and regiospecific [4+1] annulation between benzamides and alkenes for the synthesis of functionalized isoindolinones

Supporting Information

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General Information

If not otherwise specified, the reagents were obtained from commercial sources and used directly without purification. Heating source: all the reactions that require heating were carried out in an oil bath. Analytical thin-layer chromatography (TLC): HSGF 254 (0.15-0.2 mm thickness). Detection under UV light at 254 nm. Column chromatography: separations were carried out on silica gel FCP 200-300. Yields refer to isolated compounds. Melting point apparatus: a micro melting point apparatus, values are uncorrected. Nuclear magnetic resonance (NMR) apparatus: a Brucker 400, 500 or 600 MHz instrument. Chemical shifts (δ) are given in ppm. Proton coupling patterns were recorded as singlet (s), doublet (d), triplet (t), quartet (q), and multiplet (m). HRMS (high-resolution mass) were measured on a Thermo Scientific LTQ Orbitrap Discovery (Bremen, Germany). The linear ion trap (LTQ) part of the hybrid MS system was equipped with electrospray ionization (ESI) probe and operated in both positive and negative ion modes.

Preparation of the Starting Materials

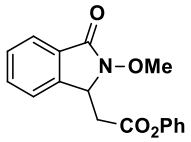
All the aryl amides were prepared according to the literature procedure and their characterization data were in accordance with the published ones.[1]

All the alkenes were prepared according to the literature procedure and their characterization data were in accordance with the published ones.[2]

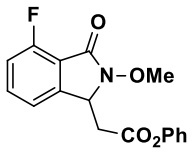
General Procedure for the Rhodium-Catalyzed Chemo- and Regiospecific [4+1] Annulation between Aryl Amides and Alkenes

To a mixture of aryl amides **1** (0.25 mmol), $[Cp^*RhCl_2]_2$ (5 mol%) and NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of alkenes **2** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at the temperature for the time indicated in Table 2 and 3. After removal of the solvent, the residue was purified by flash chromatography on silica gel to give the desired products.

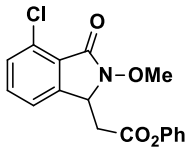
Characterization Data of Products 3, 4 and 5



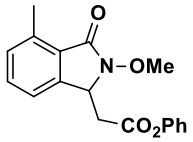
phenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (3aa): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (70.3 mg, 95%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.5 Hz, 1H), 7.63-7.58 (m, 1H), 7.55-7.49 (m, 2H), 7.43-7.37 (m, 2H), 7.28-7.25 (m, 1H), 7.10-7.04 (m, 2H), 5.34-5.28 (m, 1H), 3.99 (s, 3H), 3.13 (dd, *J* = 16.3, 7.0 Hz, 1H), 3.03 (dd, *J* = 16.3, 5.9 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.36, 164.70, 150.48, 141.32, 132.69, 129.85, 129.73, 129.25, 126.37, 124.29, 122.80, 121.48, 64.09, 56.25, 37.67; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{16}NO_4$ 298.1074; Found 298.1072.



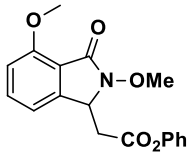
phenyl 2-(4-fluoro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ab): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow oil (45.6 mg, yield 58%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61-7.55 (m, 1H), 7.42-7.37 (m, 2H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.27-7.24 (m, 1H), 7.17-7.12 (m, 1H), 7.06 (d, *J* = 7.7 Hz, 2H), 5.32-5.23 (m, 1H), 3.96 (s, 3H), 3.13 (dd, *J* = 16.4, 6.9 Hz, 1H), 3.03 (dd, *J* = 16.4, 5.9 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.04, 162.02 (d, $J_{C\text{-}F}$ = 1.9 Hz), 158.93 (d, $J_{C\text{-}F}$ = 261.7 Hz), 150.35, 143.67, 134.67 (d, $J_{C\text{-}F}$ = 7.7 Hz), 129.69, 126.36, 121.38, 118.85 (d, $J_{C\text{-}F}$ = 4.1 Hz), 117.11 (d, $J_{C\text{-}F}$ = 13.5 Hz), 116.62 (d, $J_{C\text{-}F}$ = 19.2 Hz), 64.13, 56.09, 37.52; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}FNO_4$ 316.0980; Found 316.0975.



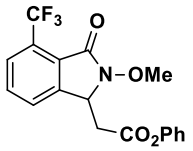
phenyl 2-(4-chloro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ac): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (42.7 mg, yield 51%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53-7.48 (m, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.42-7.35 (m, 3H), 7.27-7.22 (m, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 5.27-5.19 (m, 1H), 3.97 (s, 3H), 3.11 (dd, *J* = 16.3, 6.9 Hz, 1H), 3.02 (dd, *J* = 16.3, 5.8 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.06, 162.87, 150.33, 143.67, 133.33, 132.01, 130.81, 129.67, 126.34, 126.05, 121.36, 121.30, 64.07, 55.47, 37.48; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}ClNO_4$ 332.0684; Found 332.0680.



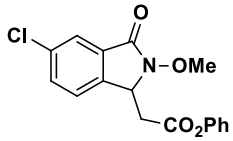
phenyl 2-(2-methoxy-4-methyl-3-oxoisoindolin-1-yl)acetate (3ad): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (53.4 mg, yield 69%), mp 78-79 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48-7.44 (m, 1H), 7.42-7.37 (m, 2H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.26-7.23 (m, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 5.27-5.20 (m, 1H), 3.97 (s, 3H), 3.07 (dd, *J* = 16.1, 7.2 Hz, 1H), 3.01 (dd, *J* = 16.1, 5.7 Hz, 1H), 2.72 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.45, 166.13, 150.52, 141.94, 138.54, 132.16, 131.13, 129.69, 126.80, 126.30, 121.50, 120.04, 63.98, 55.93, 37.99, 17.46; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1222.



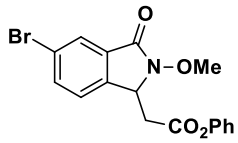
phenyl 2-(2,4-dimethoxy-3-oxoisoindolin-1-yl)acetate (3ae): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (42.4 mg, yield 52%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56-7.52 (m, 1H), 7.42-7.37 (m, 2H), 7.27-7.24 (m, 1H), 7.09-7.06 (m, 2H), 7.04 (d, J = 7.6 Hz, 1H), 6.94 (d, J = 8.4 Hz, 1H), 5.26-5.18 (m, 1H), 3.97 (s, 3H), 3.94 (s, 3H), 3.07 (dd, J = 16.1, 7.2 Hz, 1H), 3.00 (dd, J = 16.1, 5.7 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.38, 164.59, 157.75, 150.51, 143.97, 134.45, 132.69, 129.68, 126.30, 121.49, 114.66, 111.36, 63.99, 56.11, 56.07, 37.94; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_5$ 328.1179; Found 328.1175.



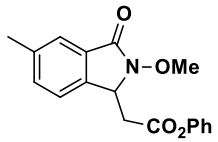
phenyl 2-(2-methoxy-3-oxo-4-(trifluoromethyl)isoindolin-1-yl)acetate (3af): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (54.6 mg, yield 60%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, J = 7.6 Hz, 1H), 7.76-7.73 (m, 1H), 7.73-7.68 (m, 1H), 7.42-7.36 (m, 2H), 7.27-7.23 (m, 1H), 7.08-7.03 (m, 2H), 5.33-5.25 (m, 1H), 3.99 (s, 3H), 3.15 (dd, J = 16.4, 6.8 Hz, 1H), 3.07 (dd, J = 16.4, 5.9 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.01, 161.87, 150.32, 143.49, 132.41, 129.69, 127.59 (q, J_{C-F} = 34.9 Hz), 127.41, 126.72 (q, J_{C-F} = 5.5 Hz), 126.58, 126.38, 122.55 (q, J_{C-F} = 273.6 Hz), 121.34, 64.10, 55.90, 37.25; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{15}F_3NO_4$ 366.0948; Found 366.0945.



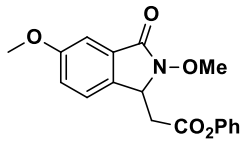
phenyl 2-(5-chloro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ag): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (75.8 mg, yield 91%), mp 87-88 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (d, J = 1.9 Hz, 1H), 7.57 (dd, J = 8.1, 2.0 Hz, 1H), 7.46 (d, J = 8.1 Hz, 1H), 7.43-7.38 (m, 2H), 7.29-7.26 (m, 1H), 7.10-7.05 (m, 2H), 5.30-5.25 (m, 1H), 3.98 (s, 3H), 3.16 (dd, J = 16.4, 6.7 Hz, 1H), 2.99 (dd, J = 16.4, 6.2 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.12, 163.30, 150.38, 139.41, 135.61, 132.81, 131.63, 129.75, 126.43, 124.41, 124.32, 121.41, 64.19, 56.01, 37.35; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}ClNO_4$ 332.0684; Found 332.0681.



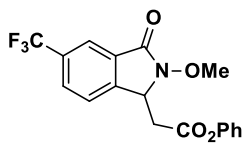
phenyl 2-(5-bromo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ah): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (84.9 mg, yield 90%), mp 85-86 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 1.8 Hz, 1H), 7.72 (dd, *J* = 8.1, 1.9 Hz, 1H), 7.44-7.37 (m, 3H), 7.28-7.26 (m, 1H), 7.09-7.04 (m, 2H), 5.28-5.21 (m, 1H), 3.98 (s, 3H), 3.16 (dd, *J* = 16.4, 6.7 Hz, 1H), 2.99 (dd, *J* = 16.4, 6.2 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.10, 163.14, 150.38, 139.91, 135.62, 131.85, 129.75, 127.40, 126.43, 124.59, 123.38, 121.40, 64.19, 56.05, 37.28; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}BrNO_4$ 376.0179; Found 376.0174.



phenyl 2-(2-methoxy-5-methyl-3-oxoisoindolin-1-yl)acetate (3ai): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (68.5 mg, yield 88%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (s, 1H), 7.44-7.34 (m, 4H), 7.28-7.25 (m, 1H), 7.07 (d, *J* = 8.2 Hz, 2H), 5.30-5.22 (m, 1H), 3.97 (s, 3H), 3.10 (dd, *J* = 16.2, 7.0 Hz, 1H), 3.00 (dd, *J* = 16.2, 5.9 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.40, 164.94, 150.49, 139.43, 138.52, 133.58, 129.81, 129.69, 126.32, 124.47, 122.54, 121.48, 64.04, 56.15, 37.79, 21.55; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1225.

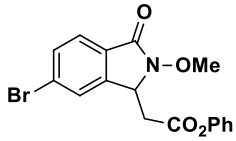


phenyl 2-(2,5-dimethoxy-3-oxoisoindolin-1-yl)acetate (3aj): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (70.6 mg, yield 86%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42-7.37 (m, 3H), 7.36 (d, *J* = 2.4 Hz, 1H), 7.27-7.25 (m, 1H), 7.14 (dd, *J* = 8.4, 2.5 Hz, 1H), 7.10-7.06 (m, 2H), 5.27-5.20 (m, 1H), 3.97 (s, 3H), 3.86 (s, 3H), 3.10 (dd, *J* = 16.2, 7.0 Hz, 1H), 2.98 (dd, *J* = 16.2, 6.0 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.39, 164.80, 160.67, 150.48, 133.36, 131.12, 129.70, 126.32, 123.89, 121.47, 120.74, 107.15, 64.08, 55.98, 55.87, 37.80; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_5$ 328.1179; Found 328.1176.

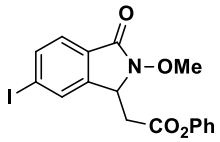


phenyl 2-(2-methoxy-3-oxo-5-(trifluoromethyl)isoindolin-1-yl)acetate (3ak): The reaction

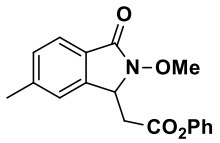
mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (67.6 mg, yield 74%), mp 73-74 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (s, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.44-7.38 (m, 2H), 7.29-7.26 (m, 1H), 7.07 (d, *J* = 8.4 Hz, 2H), 5.39-5.32 (m, 1H), 4.01 (s, 3H), 3.22 (dd, *J* = 16.6, 6.6 Hz, 1H), 3.04 (dd, *J* = 16.5, 6.3 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.99, 163.08, 150.35, 144.62, 132.04 (q, J_{C-F} = 33.3 Hz), 130.87, 129.78, 129.46 (q, J_{C-F} = 3.5 Hz), 126.50, 123.74, 123.62 (q, J_{C-F} = 272.6 Hz), 121.56 (q, J_{C-F} = 3.7 Hz), 121.38, 64.27, 56.21, 37.10; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{15}F_3NO_4$ 366.0948; Found 366.0945.



phenyl 2-(6-bromo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3al): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (84.7 mg, yield 90%), mp 103-104 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.1 Hz, 1H), 7.69 (s, 1H), 7.65 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.44-7.37 (m, 2H), 7.28-7.25 (m, 1H), 7.08 (d, *J* = 7.8 Hz, 2H), 5.29-5.22 (m, 1H), 3.97 (s, 3H), 3.16 (dd, *J* = 16.5, 6.7 Hz, 1H), 3.00 (dd, *J* = 16.5, 6.2 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.04, 163.80, 150.36, 143.01, 132.71, 129.73, 128.73, 127.33, 126.41, 126.36, 125.66, 121.40, 64.16, 55.85, 37.27; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}BrNO_4$ 376.0179; Found 376.0177.

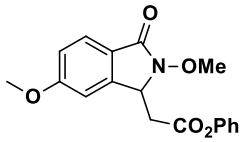


phenyl 2-(6-iodo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3am): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (97.8 mg, yield 92%), mp 108-109 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 0.6 Hz, 1H), 7.88 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.44-7.38 (m, 2H), 7.29-7.26 (m, 1H), 7.11-7.05 (m, 2H), 5.28-5.23 (m, 1H), 3.97 (s, 3H), 3.15 (dd, *J* = 16.5, 6.8 Hz, 1H), 3.00 (dd, *J* = 16.4, 6.1 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.11, 163.98, 150.38, 142.95, 138.59, 132.15, 129.77, 129.32, 126.45, 125.65, 121.44, 99.48, 64.18, 55.64, 37.32; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}INO_4$ 424.0040; Found 424.0037.

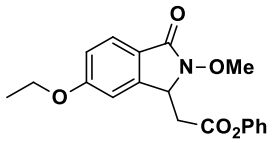


phenyl 2-(2-methoxy-6-methyl-3-oxoisoindolin-1-yl)acetate (3an): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on

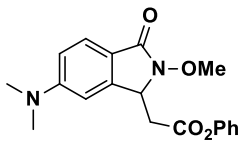
silica gel to provide the product as a colorless viscous oil (71.0 mg, yield 91%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.7 Hz, 1H), 7.43-7.36 (m, 2H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.30 (s, 1H), 7.28-7.25 (m, 1H), 7.11-7.04 (m, 2H), 5.29-5.22 (m, 1H), 3.96 (s, 3H), 3.09 (dd, *J* = 16.2, 7.1 Hz, 1H), 3.02 (dd, *J* = 16.2, 5.8 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.43, 165.06, 150.46, 143.56, 141.63, 130.16, 129.69, 127.02, 126.31, 124.07, 123.23, 121.45, 64.03, 56.17, 37.72, 22.22; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1227.



phenyl 2-(2,6-dimethoxy-3-oxoisoindolin-1-yl)acetate (3ao): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a colorless viscous oil (72.7 mg, yield 89%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 1H), 7.41-7.36 (m, 2H), 7.26-7.23 (m, 1H), 7.09-7.05 (m, 2H), 7.01 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.98 (d, *J* = 2.2 Hz, 1H), 5.25-5.20 (m, 1H), 3.95 (s, 3H), 3.85 (s, 3H), 3.10 (dd, *J* = 16.3, 7.1 Hz, 1H), 3.00 (dd, *J* = 16.3, 5.9 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.36, 165.37, 163.62, 150.45, 143.69, 129.66, 126.29, 125.82, 121.86, 121.42, 115.46, 108.00, 64.08, 56.33, 55.80, 37.76; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_5$ 328.1179; Found 328.1176.

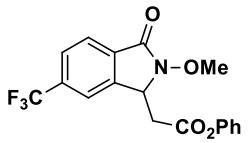


phenyl 2-(6-ethoxy-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ap): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (74.3 mg, yield 87%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 1H), 7.41-7.35 (m, 2H), 7.26-7.21 (m, 1H), 7.09-7.04 (m, 2H), 6.98 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.97-6.95 (m, 1H), 5.25-5.16 (m, 1H), 4.06 (q, *J* = 7.0 Hz, 2H), 3.94 (s, 3H), 3.09 (dd, *J* = 16.2, 7.1 Hz, 1H), 2.99 (dd, *J* = 16.3, 5.8 Hz, 1H), 1.42 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.34, 165.43, 162.99, 150.43, 143.64, 129.62, 126.24, 125.74, 121.60, 121.40, 115.87, 108.40, 64.12, 64.03, 56.31, 37.74, 14.69; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}NO_5$ 342.1336; Found 342.1334.

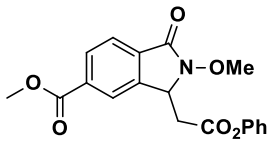


phenyl 2-(6-(dimethylamino)-2-methoxy-3-oxoisoindolin-1-yl)acetate (3aq): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a pale yellow viscous oil (65.5 mg, yield 77%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.6 Hz, 1H), 7.41-7.35 (m, 2H), 7.26-7.22 (m, 1H), 7.09-7.04 (m, 2H), 6.74 (dd, *J* = 8.6, 2.3 Hz, 1H), 6.65 (d, *J* = 1.8 Hz, 1H),

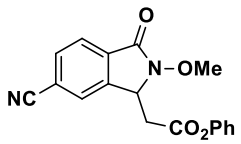
5.22-5.16 (m, 1H), 3.93 (s, 3H), 3.08-2.98 (m, 8H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.65, 167.01, 153.64, 150.52, 143.98, 129.61, 126.18, 125.36, 121.45, 116.26, 112.61, 104.43, 64.00, 56.75, 40.50, 38.28; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{21}N_2O_4$ 341.1496; Found 341.1492.



phenyl 2-(2-methoxy-3-oxo-6-(trifluoromethyl)isoindolin-1-yl)acetate (3ar): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (88.5 mg, yield 97%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.9 Hz, 1H), 7.81 (s, 1H), 7.78 (d, J = 8.0 Hz, 1H), 7.42-7.36 (m, 2H), 7.27-7.24 (m, 1H), 7.06 (d, J = 7.7 Hz, 2H), 5.38-5.32 (m, 1H), 3.99 (s, 3H), 3.19 (dd, J = 16.5, 6.7 Hz, 1H), 3.06 (dd, J = 16.5, 6.1 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.94, 162.88, 150.31, 141.70, 134.41 (q, J_{C-F} = 32.8 Hz), 133.28, 129.72, 126.43, 126.42 (q, J_{C-F} = 3.8 Hz), 124.80, 123.59 (q, J_{C-F} = 272.9 Hz), 121.33, 120.25 (q, J_{C-F} = 3.9 Hz), 64.17, 56.11, 37.09; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{15}F_3NO_4$ 366.0948; Found 366.0939.

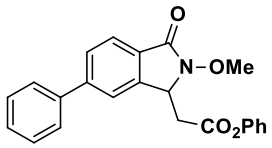


methyl 2-methoxy-1-oxo-3-(2-oxo-2-phenoxyethyl)isoindoline-5-carboxylate (3as): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a white amorphous solid (79.1 mg, yield 89%), mp 135-136 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.23-8.17 (m, 2H), 7.95 (d, J = 7.8 Hz, 1H), 7.44-7.37 (m, 2H), 7.28-7.26 (m, 1H), 7.08 (dd, J = 8.5, 0.9 Hz, 2H), 5.39-5.32 (m, 1H), 4.00 (s, 3H), 3.96 (s, 3H), 3.16-3.07 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.00, 166.10, 163.38, 150.42, 141.16, 134.05, 133.90, 130.64, 129.74, 126.42, 124.31, 124.07, 121.46, 64.18, 56.27, 52.78, 37.37; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{18}NO_6$ 356.1129; Found 356.1128.

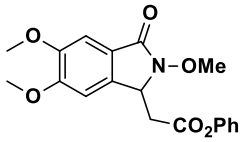


phenyl 2-(6-cyano-2-methoxy-3-oxoisoindolin-1-yl)acetate (3at): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a white amorphous solid (44.6 mg, yield 55%), mp 183-184 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.8 Hz, 1H), 7.87 (s, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.46-7.38 (m, 2H), 7.30-7.26 (m, 1H), 7.12-7.04 (m, 2H), 5.36-5.29 (m, 1H), 4.01 (s, 3H), 3.26 (dd, J = 16.8, 6.1 Hz, 1H), 3.02 (dd, J = 16.8, 6.7 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.84, 162.38, 150.28, 141.89, 134.04, 133.22, 129.82, 127.06, 126.58, 125.08, 121.35, 117.91, 116.17, 64.34, 55.92, 36.84; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{15}N_2O_4$ 323.1026; Found

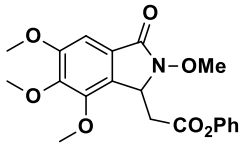
323.1024.



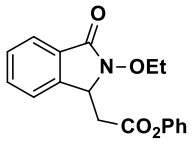
phenyl 2-(2-methoxy-3-oxo-6-phenylisoindolin-1-yl)acetate (3au): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (88.9 mg, yield 95%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.9 Hz, 1H), 7.73 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.71 (s, 1H), 7.62-7.56 (m, 2H), 7.50-7.45 (m, 2H), 7.43-7.36 (m, 3H), 7.27-7.23 (m, 1H), 7.07 (dd, *J* = 8.4, 0.9 Hz, 2H), 5.39-5.31 (m, 1H), 4.00 (s, 3H), 3.15 (dd, *J* = 16.3, 7.1 Hz, 1H), 3.09 (dd, *J* = 16.3, 5.8 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.29, 164.64, 150.39, 145.95, 141.93, 140.00, 129.64, 129.10, 128.46, 128.40, 128.31, 127.44, 126.28, 124.55, 121.42, 121.39, 64.04, 56.32, 37.62; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{20}NO_4$ 374.1387; Found 374.1382.



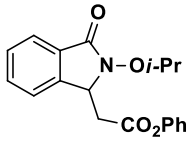
phenyl 2-(2,5,6-trimethoxy-3-oxoisoindolin-1-yl)acetate (3av): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a colorless viscous oil (66.7 mg, yield 75%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42-7.36 (m, 2H), 7.31 (s, 1H), 7.26-7.22 (m, 1H), 7.06 (d, *J* = 8.2 Hz, 2H), 6.96 (s, 1H), 5.24-5.14 (m, 1H), 3.95 (s, 3H), 3.91 (s, 3H), 3.91 (s, 3H), 3.12 (dd, *J* = 16.3, 6.9 Hz, 1H), 2.97 (dd, *J* = 16.3, 6.1 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.49, 165.98, 153.47, 150.42, 135.23, 129.68, 126.30, 121.73, 121.39, 105.76, 105.10, 64.18, 56.42, 56.38, 56.25, 37.85; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}NO_6$ 358.1285; Found 358.1280.



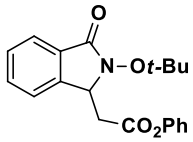
phenyl 2-(2,5,6,7-tetramethoxy-3-oxoisoindolin-1-yl)acetate (3aw): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (86.0 mg, yield 89%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39-7.34 (m, 2H), 7.25-7.20 (m, 1H), 7.15 (s, 1H), 7.04 (d, *J* = 7.8 Hz, 2H), 5.29 (dd, *J* = 8.1, 3.6 Hz, 1H), 4.01 (s, 3H), 3.92 (s, 3H), 3.90 (s, 6H), 3.36 (dd, *J* = 15.7, 3.6 Hz, 1H), 2.83 (dd, *J* = 15.7, 8.2 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.37, 164.87, 155.63, 150.58, 148.58, 145.41, 129.57, 126.12, 125.65, 125.05, 121.49, 102.12, 64.03, 61.20, 61.02, 56.51, 54.90, 36.43; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_7$ 388.1391; Found 388.1385.



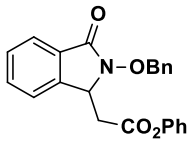
phenyl 2-(2-ethoxy-3-oxoisoindolin-1-yl)acetate (3ax): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (72.0 mg, yield 93%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.5 Hz, 1H), 7.64-7.58 (m, 1H), 7.55-7.49 (m, 2H), 7.44-7.37 (m, 2H), 7.28 (d, *J* = 6.2 Hz, 1H), 7.10 (d, *J* = 7.7 Hz, 2H), 5.35-5.25 (m, 1H), 4.23 (q, *J* = 7.0 Hz, 2H), 3.15 (dd, *J* = 16.4, 7.1 Hz, 1H), 3.02 (dd, *J* = 16.4, 5.9 Hz, 1H), 1.37 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.35, 164.78, 150.46, 141.43, 132.50, 129.93, 129.62, 129.13, 126.27, 124.19, 122.72, 121.46, 72.11, 56.65, 37.70, 13.80; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1223.



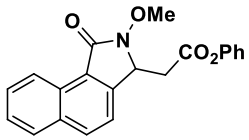
phenyl 2-(2-isopropoxy-3-oxoisoindolin-1-yl)acetate (3ay): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (69.7 mg, yield 86%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.5 Hz, 1H), 7.64-7.58 (m, 1H), 7.56-7.50 (m, 2H), 7.45-7.38 (m, 2H), 7.31-7.27 (m, 1H), 7.11 (d, *J* = 8.2 Hz, 2H), 5.32-5.24 (m, 1H), 4.55-4.45 (m, 1H), 3.20 (dd, *J* = 16.6, 6.8 Hz, 1H), 2.95 (dd, *J* = 16.6, 6.2 Hz, 1H), 1.38 (d, *J* = 6.2 Hz, 3H), 1.33 (d, *J* = 6.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.48, 165.51, 150.47, 141.88, 132.46, 129.93, 129.63, 129.13, 126.26, 124.25, 122.76, 121.48, 78.59, 57.49, 37.84, 21.12, 20.96; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}NO_4$ 326.1387; Found 326.1384.



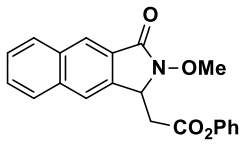
phenyl 2-(2-(*tert*-butoxy)-3-oxoisoindolin-1-yl)acetate (3az): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (18.0 mg, yield 21%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.8 Hz, 1H), 7.61-7.57 (m, 1H), 7.54-7.49 (m, 2H), 7.42-7.37 (m, 2H), 7.26-7.24 (m, 1H), 7.09 (d, *J* = 7.7 Hz, 2H), 5.24 (dd, *J* = 7.5, 5.6 Hz, 1H), 3.36 (dd, *J* = 16.7, 5.5 Hz, 1H), 2.75 (dd, *J* = 16.7, 7.7 Hz, 1H), 1.43 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.63, 167.64, 150.49, 142.97, 132.59, 129.69, 129.65, 129.14, 126.26, 124.43, 122.98, 121.53, 85.05, 59.80, 37.88, 27.58; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_4$ 340.1543; Found 340.1536.



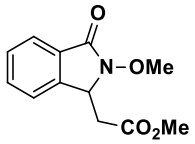
phenyl 2-(2-(benzyloxy)-3-oxoisoindolin-1-yl)acetate (3ba): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (89.6 mg, yield 96%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.5 Hz, 1H), 7.61-7.55 (m, 1H), 7.54-7.47 (m, 3H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.41-7.34 (m, 3H), 7.34-7.29 (m, 2H), 7.25-7.19 (m, 1H), 6.92 (d, *J* = 8.2 Hz, 2H), 5.18 (dd, *J* = 22.3, 10.2 Hz, 2H), 5.08-5.01 (m, 1H), 3.05 (dd, *J* = 16.3, 6.8 Hz, 1H), 2.92 (dd, *J* = 16.3, 6.0 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.21, 164.80, 150.32, 141.49, 134.71, 132.51, 129.97, 129.78, 129.50, 129.12, 129.07, 128.66, 126.13, 124.10, 122.68, 121.46, 78.42, 57.06, 37.35; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{20}NO_4$ 374.1387; Found 374.1380.



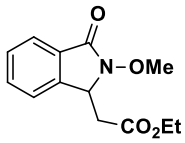
phenyl 2-(2-methoxy-1-oxo-2,3-dihydro-1*H*-benzo[*e*]isoindol-3-yl)acetate (3bb): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (45.8 mg, yield 53%), mp 119-120 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.15 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.72-7.66 (m, 1H), 7.63-7.58 (m, 1H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.44-7.37 (m, 2H), 7.28-7.26 (m, 1H), 7.09 (d, *J* = 7.6 Hz, 2H), 5.37 (t, *J* = 6.5 Hz, 1H), 4.04 (s, 3H), 3.13 (d, *J* = 6.5 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.46, 166.82, 150.52, 141.83, 133.72, 133.45, 129.71, 129.38, 128.58, 128.41, 127.27, 126.34, 124.12, 123.97, 121.49, 119.43, 64.33, 56.56, 37.64; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{21}H_{18}NO_4$ 348.1230; Found 348.1225.



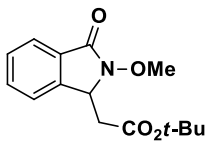
phenyl 2-(2-methoxy-3-oxo-2,3-dihydro-1*H*-benzo[*f*]isoindol-1-yl)acetate (3bc): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a yellow amorphous solid (79.7 mg, yield 92%), mp 136-137 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.42 (s, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.95 (s, 1H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.64-7.55 (m, 2H), 7.44-7.38 (m, 2H), 7.29-7.26 (m, 1H), 7.13-7.08 (m, 2H), 5.50-5.43 (m, 1H), 4.03 (s, 3H), 3.24 (dd, *J* = 16.4, 6.8 Hz, 1H), 3.10 (dd, *J* = 16.4, 6.1 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.46, 164.21, 150.49, 136.28, 135.46, 133.26, 129.73, 129.67, 128.37, 128.24, 127.25, 127.09, 126.36, 124.82, 122.16, 121.49, 63.96, 56.01, 38.21; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{21}H_{18}NO_4$ 348.1230; Found 348.1225.



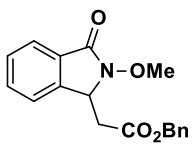
methyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4aa): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (50.2 mg, yield 85%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 1H), 7.56-7.51 (m, 1H), 7.47-7.43 (m, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 5.20-5.13 (m, 1H), 3.90 (s, 3H), 3.72 (s, 3H), 2.90 (dd, *J* = 16.2, 6.5 Hz, 1H), 2.69 (dd, *J* = 16.2, 6.6 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.98, 164.53, 141.54, 132.48, 129.65, 128.96, 123.98, 122.68, 63.86, 56.13, 52.16, 37.04; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}NO_4$ 236.0917; Found 236.0913.



ethyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ab): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (56.4 mg, yield 91%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.6 Hz, 1H), 7.57-7.52 (m, 1H), 7.49-7.45 (m, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 5.21-5.13 (m 1H), 4.25-4.14 (m, 2H), 3.92 (s, 3H), 2.90 (dd, *J* = 16.2, 6.5 Hz, 1H), 2.70 (dd, *J* = 16.2, 6.4 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.57, 164.60, 141.65, 132.48, 129.76, 128.98, 124.05, 122.73, 63.91, 61.24, 56.24, 37.34, 14.22; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{13}H_{16}NO_4$ 250.1074; Found 250.1071.

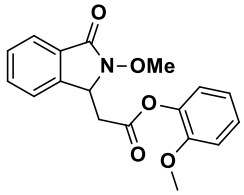


***tert*-butyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ac)**: The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (39.3 mg, yield 57%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.5 Hz, 1H), 7.60-7.52 (m, 1H), 7.50-7.45 (m, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 5.19-5.09 (m, 1H), 3.94 (s, 3H), 2.78 (dd, *J* = 16.0, 6.7 Hz, 1H), 2.69 (dd, *J* = 15.9, 5.9 Hz, 1H), 1.43 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.75, 164.61, 141.77, 132.40, 129.89, 128.88, 124.01, 122.69, 81.76, 63.92, 56.49, 38.49, 28.04; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{15}H_{20}NO_4$ 278.1387; Found 278.1381.

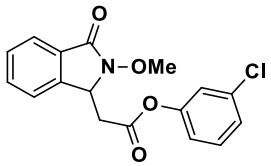


benzyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ad): The reaction mixture was subjected

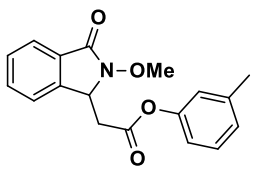
directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (64.9 mg, yield 83%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.5 Hz, 1H), 7.54-7.49 (m, 1H), 7.48-7.43 (m, 1H), 7.40-7.28 (m, 6H), 5.25-5.11 (m, 3H), 3.85 (s, 3H), 2.95 (dd, *J* = 16.2, 6.7 Hz, 1H), 2.76 (dd, *J* = 16.2, 6.3 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.41, 164.60, 141.49, 135.41, 132.48, 129.70, 128.98, 128.70, 128.59, 128.57, 124.05, 122.68, 67.04, 63.87, 56.25, 37.39; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1226.



2-methoxyphenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ae): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (68.0 mg, yield 83%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.63-7.55 (m, 2H), 7.54-7.48 (m, 1H), 7.26-7.21 (m, 1H), 7.04-6.98 (m, 2H), 6.98-6.94 (m, 1H), 5.33-5.25 (m, 1H), 3.99 (s, 3H), 3.84 (s, 3H), 3.27 (dd, *J* = 16.4, 6.0 Hz, 1H), 2.97 (dd, *J* = 16.4, 7.0 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.80, 164.71, 151.06, 141.59, 139.47, 132.55, 129.79, 129.12, 127.42, 124.12, 123.01, 122.69, 120.99, 112.57, 64.11, 56.27, 55.88, 36.97; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_5$ 328.1179; Found 328.1174.

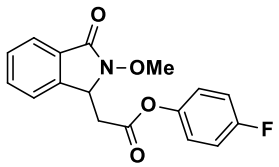


3-chlorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4af): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (80.5 mg, yield 97%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.6 Hz, 1H), 7.63-7.57 (m, 1H), 7.53-7.46 (m, 2H), 7.34-7.28 (m, 1H), 7.22 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.12-7.06 (m, 1H), 6.96 (dd, *J* = 8.1, 1.5 Hz, 1H), 5.31-5.23 (m, 1H), 3.96 (s, 3H), 3.12-3.00 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.83, 164.62, 150.84, 141.08, 134.87, 132.68, 130.38, 129.77, 129.25, 126.56, 124.22, 122.70, 122.12, 119.83, 63.99, 56.13, 37.50; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}ClNO_4$ 332.0684; Found 332.0678.

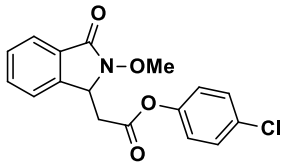


***m*-tolyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ag)**: The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to

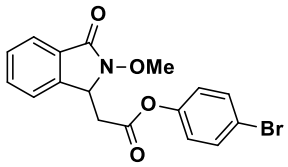
provide the product as a colorless viscous oil (73.3 mg, yield 94%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.63-7.56 (m, 1H), 7.55-7.46 (m, 2H), 7.28-7.24 (m, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.90-6.81 (m, 2H), 5.34-5.22 (m, 1H), 3.97 (s, 3H), 3.11 (dd, *J* = 16.2, 7.0 Hz, 1H), 3.01 (dd, *J* = 16.2, 5.9 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.39, 164.63, 150.38, 141.30, 139.91, 132.64, 129.79, 129.37, 129.18, 127.11, 124.20, 122.79, 121.99, 118.36, 64.04, 56.21, 37.60, 21.43; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1229.



4-fluorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ah): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (75.6 mg, yield 96%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.62-7.57 (m, 1H), 7.53-7.46 (m, 2H), 7.08-7.03 (m, 2H), 7.03-6.97 (m, 2H), 5.31-5.23 (m, 1H), 3.96 (s, 3H), 3.12-3.00 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.25, 164.63, 160.45 (d, J_{C-F} = 244.9 Hz), 146.23 (d, J_{C-F} = 2.6 Hz), 141.15, 132.65, 129.78, 129.23, 124.21, 122.85 (d, J_{C-F} = 8.5 Hz), 122.71, 116.31 (d, J_{C-F} = 23.6 Hz), 63.99, 56.17, 37.48; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}FNO_4$ 316.0980; Found 316.0973.

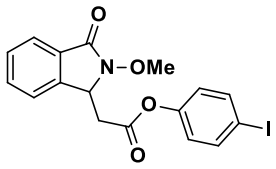


4-chlorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ai): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (78.9 mg, yield 95%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.6 Hz, 1H), 7.62-7.57 (m, 1H), 7.53-7.45 (m, 2H), 7.36-7.31 (m, 2H), 7.02-6.96 (m, 2H), 5.30-5.24 (m, 1H), 3.95 (s, 3H), 3.12-3.00 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.99, 164.63, 148.88, 141.12, 132.66, 131.67, 129.79, 129.69, 129.24, 124.22, 122.80, 122.69, 63.99, 56.16, 37.52; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}ClNO_4$ 332.0684; Found 332.0682.

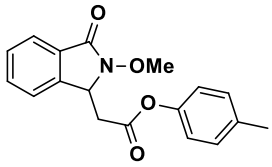


4-bromophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4aj): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a white amorphous solid (91.9 mg, yield 98%), mp 92-93 °C.

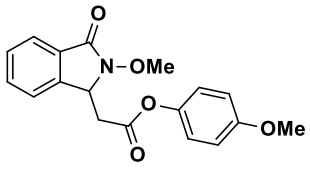
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.64-7.57 (m, 1H), 7.55-7.45 (m, 4H), 6.99-6.92 (m, 2H), 5.32-5.24 (m, 1H), 3.96 (s, 3H), 3.13-3.00 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.98, 164.67, 149.47, 141.15, 132.75, 132.70, 129.85, 129.30, 124.31, 123.26, 122.72, 119.46, 64.05, 56.19, 37.62; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}BrNO_4$ 376.0179; Found 376.0174.



4-iodophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ak): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (105.1 mg, yield 99%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.6 Hz, 1H), 7.67 (d, *J* = 8.7 Hz, 2H), 7.61-7.55 (m, 1H), 7.52-7.44 (m, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 5.29-5.22 (m, 1H), 3.94 (s, 3H), 3.10-2.99 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.81, 164.57, 150.21, 141.05, 138.64, 132.63, 129.72, 129.20, 124.16, 123.56, 122.67, 90.33, 63.96, 56.10, 37.50; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{17}H_{15}INO_4$ 424.0040; Found 424.0034.

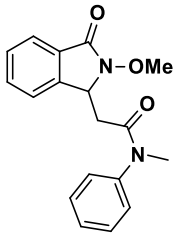


***p*-tolyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4al)**: The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (68.4 mg, yield 88%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.3 Hz, 1H), 7.62-7.56 (m, 1H), 7.53-7.46 (m, 2H), 7.17 (d, *J* = 8.3 Hz, 2H), 6.94 (d, *J* = 8.4 Hz, 2H), 5.32-5.25 (m, 1H), 3.96 (s, 3H), 3.10 (dd, *J* = 16.2, 7.0 Hz, 1H), 3.00 (dd, *J* = 16.2, 5.9 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.41, 164.59, 148.19, 141.29, 135.94, 132.58, 130.11, 129.75, 129.11, 124.12, 122.74, 121.04, 63.97, 56.21, 37.53, 20.92; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1223.

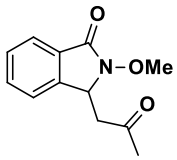


4-methoxyphenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4am): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a colorless viscous oil (75.7 mg, yield 93%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.62-7.56 (m, 1H), 7.52-7.46 (m, 2H), 7.01-6.93 (m, 2H), 6.91-6.85 (m, 2H), 5.31-5.23 (m, 1H), 3.96 (s, 3H), 3.77 (s, 3H), 3.08 (dd, *J* = 16.2, 7.0 Hz, 1H),

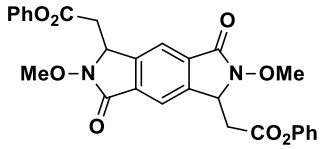
3.00 (dd, *J* = 16.2, 5.9 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.53, 164.61, 157.57, 143.93, 141.32, 132.58, 129.80, 129.12, 124.13, 122.74, 122.14, 114.63, 63.96, 56.27, 55.65, 37.49; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_5$ 328.1179; Found 328.1172.



2-(2-methoxy-3-oxoisoindolin-1-yl)-*N*-methyl-*N*-phenylacetamide (4an): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a colorless viscous oil (54.8 mg, yield 71%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.5 Hz, 1H), 7.57-7.51 (m, 1H), 7.48-7.40 (m, 2H), 7.37-7.31 (m, 2H), 7.30-7.26 (m, 1H), 7.10 (d, *J* = 7.6 Hz, 2H), 5.48-5.36 (m, 1H), 3.89 (s, 3H), 3.35 (s, 3H), 2.68 (dd, *J* = 16.0, 6.8 Hz, 1H), 2.32 (dd, *J* = 16.0, 6.5 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.71, 163.95, 143.33, 142.39, 132.29, 130.13, 129.73, 128.74, 128.29, 127.31, 123.95, 123.00, 63.65, 56.19, 37.63, 37.34; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{18}H_{19}N_2O_3$ 311.1390; Found 311.1386.

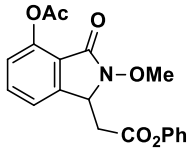


2-methoxy-3-(2-oxopropyl)isoindolin-1-one (4ao): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a yellow viscous oil (41.0 mg, yield 75%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.6 Hz, 1H), 7.57-7.51 (m, 1H), 7.49-7.44 (m, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 5.37-5.28 (m, 1H), 3.87 (s, 3H), 3.06 (dd, *J* = 17.4, 6.6 Hz, 1H), 2.72 (dd, *J* = 17.4, 6.2 Hz, 1H), 2.25 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.79, 164.42, 142.28, 132.50, 129.72, 128.89, 124.09, 122.91, 63.65, 55.28, 45.95, 30.88; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}NO_3$ 220.0968; Found 220.0966.

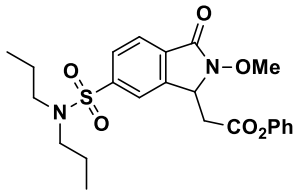


diphenyl 2,2'-(2,6-dimethoxy-3,7-dioxo-1,2,3,5,6,7-hexahydropyrrolo[3,4-*f*]isoindole-1,5-diyl)diacetate (5a): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 2:1→Petroleum/EtOAc: 1/1) on silica gel to provide the product as a white amorphous solid (64.7 mg, yield 50%), mp 225-226 ºC. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 (s, 2H), 7.44-7.36 (m, 4H), 7.29-7.26 (m, 2H), 7.08 (d, *J* = 8.0 Hz, 4H), 5.42-5.37 (m, 2H), 4.01 (s, 6H), 3.17-3.06 (m, 4H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.79, 163.10, 150.36, 142.04, 134.09, 129.76, 126.45, 121.45,

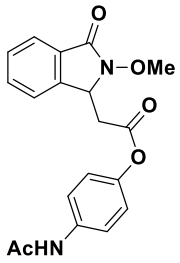
118.97, 64.29, 56.39, 37.15; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{28}H_{25}N_2O_8$ 517.1605; Found 517.1600.



phenyl 2-(4-acetoxy-2-methoxy-3-oxoisoindolin-1-yl)acetate (5b): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the product as a pale yellow viscous oil (57.9 mg, yield 65%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62-7.57 (m, 1H), 7.42-7.35 (m, 3H), 7.27-7.24 (m, 1H), 7.14 (d, *J* = 8.1 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 2H), 5.29-5.22 (m, 1H), 3.94 (s, 3H), 3.12 (dd, *J* = 16.3, 7.0 Hz, 1H), 3.02 (dd, *J* = 16.3, 5.9 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 169.35, 169.17, 162.57, 150.39, 147.79, 143.05, 133.92, 129.66, 126.31, 122.88, 121.48, 121.42, 120.50, 64.10, 55.97, 37.49, 20.77; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{18}NO_6$ 356.1129; Found 356.1122.



phenyl 2-(6-(*N*,*N*-dipropylsulfamoyl)-2-methoxy-3-oxoisoindolin-1-yl)acetate (5c): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 8:1→Petroleum/EtOAc: 4/1) on silica gel to provide the product as a colorless viscous oil (76.2 mg, yield 66%). ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.25 (s, 1H), 7.98-7.89 (m, 2H), 7.43-7.36 (m, 2H), 7.29-7.22 (m, 1H), 6.97-6.89 (m, 2H), 5.48 (dd, *J* = 6.2, 4.8 Hz, 1H), 3.90 (s, 3H), 3.53 (dd, *J* = 16.2, 4.5 Hz, 1H), 3.29 (dd, *J* = 16.2, 6.5 Hz, 1H), 3.07-2.93 (m, 4H), 1.49-1.38 (m, 4H), 0.78 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR (151 MHz, DMSO-d_6) δ 168.50, 161.52, 150.04, 142.92, 142.23, 133.28, 129.59, 127.28, 126.01, 123.94, 122.15, 121.37, 63.40, 55.73, 49.68, 35.32, 21.61, 10.94; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}N_2O_6S$ 461.1741; Found 461.1736.

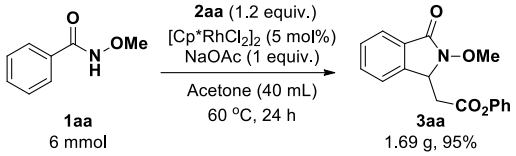


4-acetamidophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (5d): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 1/2→Petroleum/EtOAc: 1/4) on silica gel to provide the product as a white amorphous solid (67.0 mg, yield 76%), mp 169-170 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.01 (s, 1H), 7.78-7.64 (m, 3H), 7.61-7.57 (m, 2H), 7.57-7.53 (m, 1H), 7.00-6.85 (m, 2H), 5.36 (dd, *J* = 7.1, 4.7 Hz, 1H), 3.87 (s, 3H), 3.39 (dd, *J* = 16.0, 4.6 Hz, 1H), 3.04 (dd, *J* = 16.0, 7.2 Hz, 1H), 2.04 (s, 3H); ^{13}C NMR (151 MHz, DMSO-d_6) δ 168.98,

168.26, 163.21, 145.29, 141.42, 137.10, 132.45, 129.47, 128.81, 123.32, 122.95, 121.62, 119.89, 63.23, 55.66, 36.25, 23.92; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}N_2O_5$ 355.1288; Found 355.1285.

phenyl (*E*)-3-(2-carbamoylphenyl)acrylate (3aa''): The reaction mixture was subjected directly to flash chromatography (Petroleum/EtOAc: 8/1→Petroleum/EtOAc: 4/1) on silica gel to provide the product as a pale yellow viscous oil (38.3 mg, yield 57%). ^{1}H NMR (600 MHz, DMSO-d_6) δ 8.20 (d, J = 16.0 Hz, 1H), 8.05 (s, 1H), 8.02-7.97 (m, 1H), 7.68 (s, 1H), 7.58-7.50 (m, 3H), 7.49-7.42 (m, 2H), 7.33-7.27 (m, 1H), 7.22 (d, J = 8.2 Hz, 2H), 6.87 (d, J = 16.0 Hz, 1H); ^{13}C NMR (151 MHz, DMSO-d_6) δ 170.08, 164.94, 150.48, 144.17, 138.21, 131.39, 130.36, 129.96, 129.61, 127.76, 127.12, 125.94, 121.87, 118.39; HRMS (ESI) m/z: $[M + H]^+$ Calcd for $C_{16}H_{14}NO_3$ 268.0968; Found 268.0966.

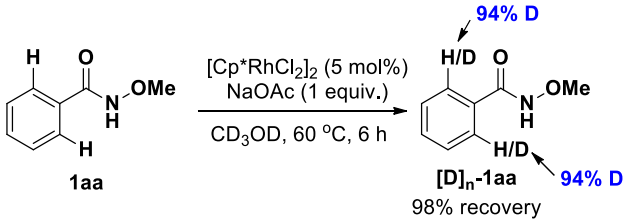
Gram-Scale Experiments



To a mixture of **1aa** (6 mmol), $[Cp^*RhCl_2]_2$ (5 mol%) and NaOAc (6 mmol) in a 100 mL round-bottom flask was added a solution of **2aa** (7.2 mmol) in acetone (40.0 mL). Then the flask was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 24 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to provide the desired product **3aa** as a pale yellow viscous oil (1.69 g, 95% yield).

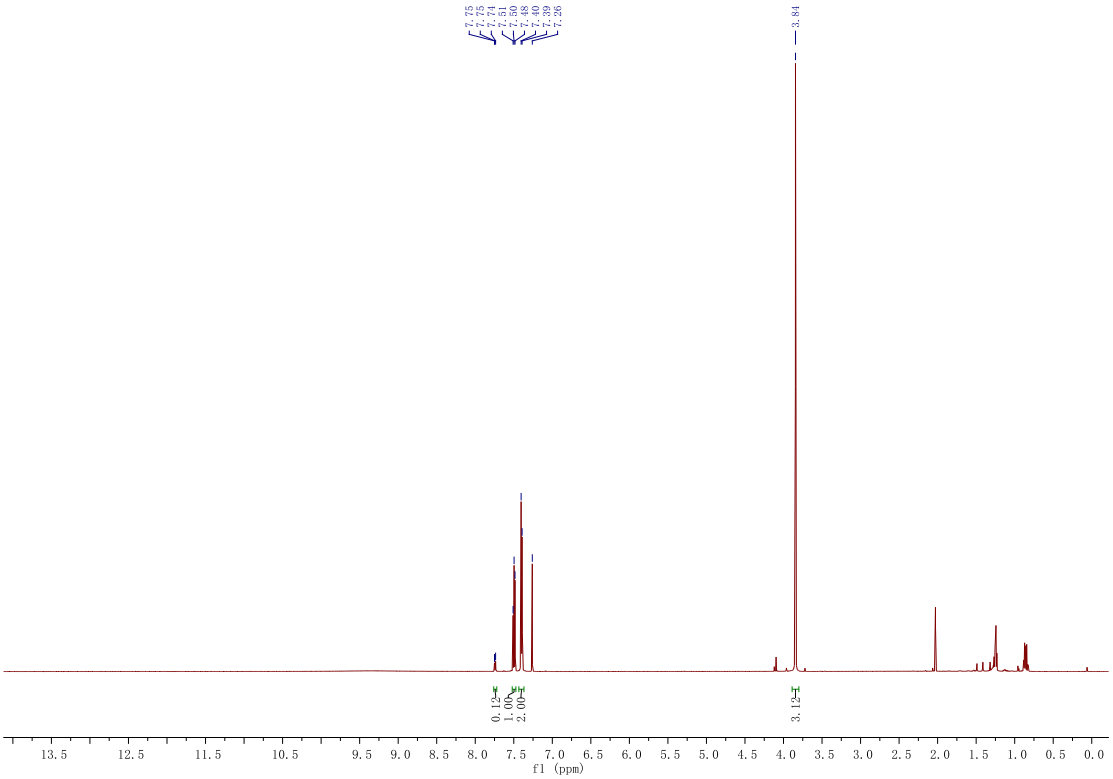
Mechanistic Experiments

Deuterium Incorporation Experiments A

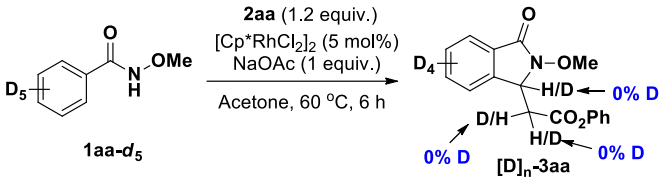


To a mixture of **1aa** (0.25 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added CD_3OD (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 6 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to give the product. *Found H/D exchange occurred at the ortho-position of the N-methoxybenzamide (94% D).*

^{1}H NMR of the product **[D]$_n$-1aa** of this reaction

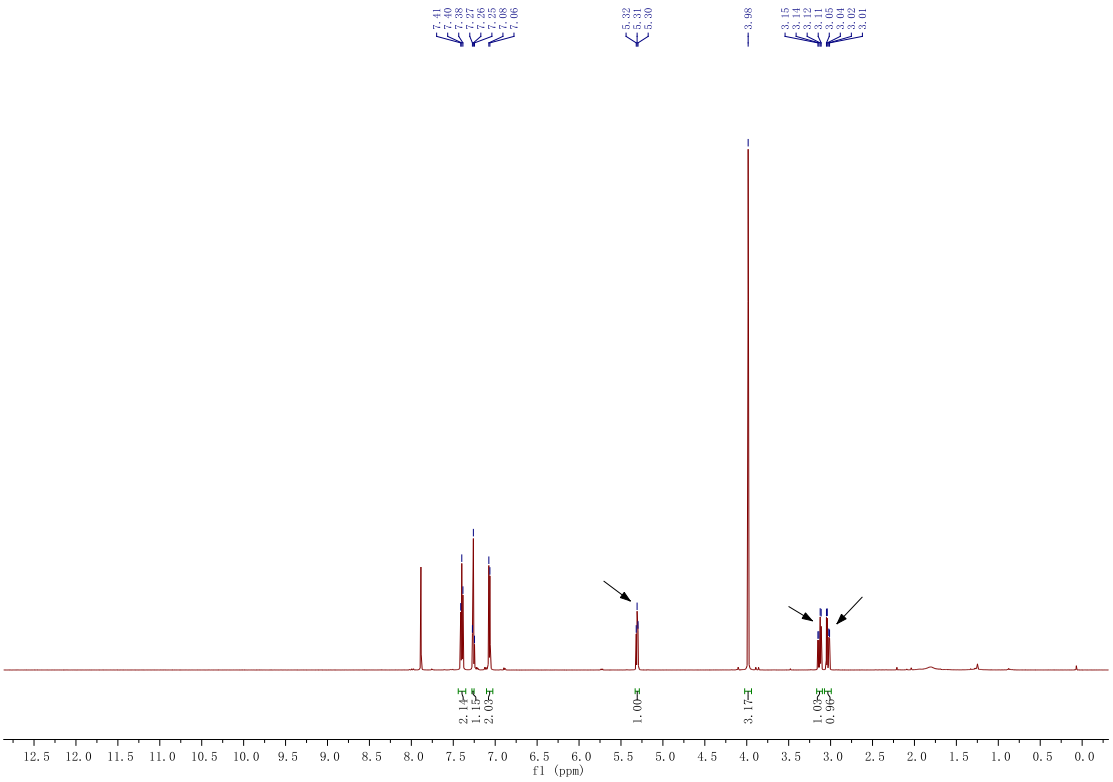


Deuterium Incorporation Experiments B



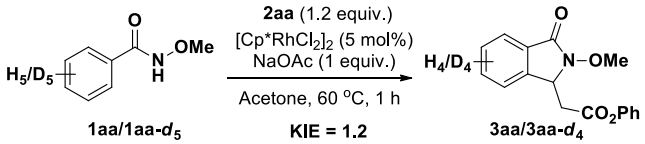
To a mixture of **1aa-*d*$_5$** (0.25 mmol, 99% D), [Cp*RhCl$_2$]$_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of **2aa** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 6 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to give the product. *Found no H/D exchange occurred at the α and β positions of the ester group.*

^{1}H NMR of the product **[D]$_n$-3aa** of this reaction

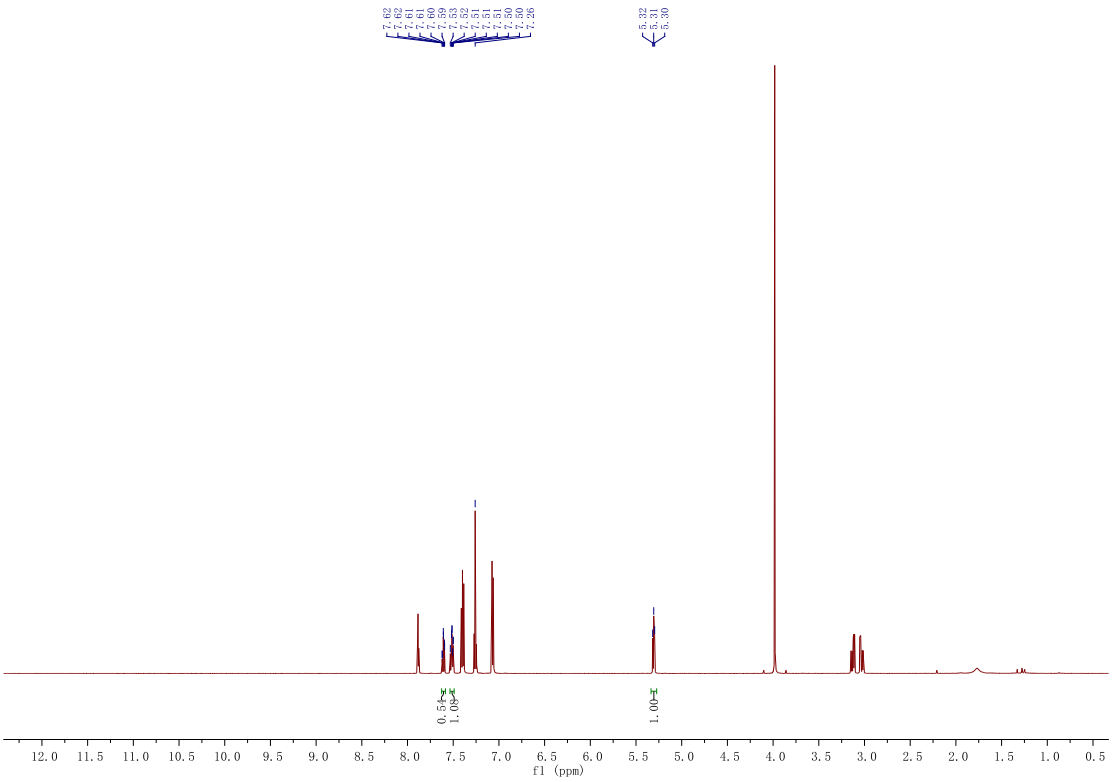


Determination of the KIE

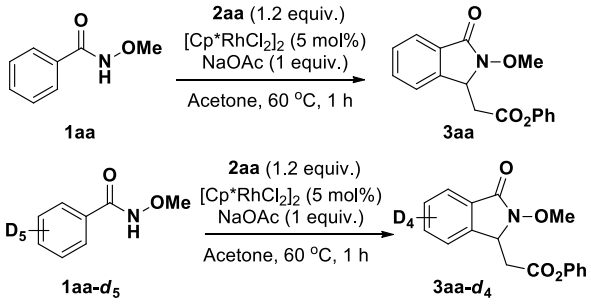
(1) The intermolecular competition experiments for KIE value measurement



To a mixture of **1aa** (0.25 mmol), **1aa-*d*$_5$** (0.25 mmol, 99% D), $[Cp^*RhCl_2]_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of **2aa** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 1 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to give a mixture of **3aa** and **3aa-*d*$_4$**, which was analyzed by ^{1}H NMR. *A kinetic isotopic effect of this reaction was determined to be* k_H/k_D *= 1.2 (0.54/0.46).*



(2) Two parallel reactions for KIE value measurement

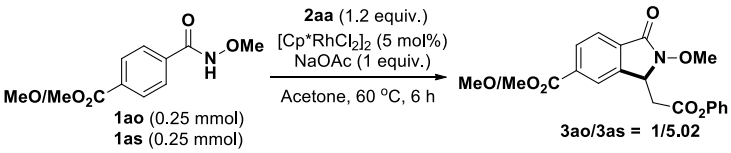


To a mixture of **1aa** (0.25 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of **2aa** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 1 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to give product **3aa** (137.57 μmol, 40.9 mg, 55% yield).

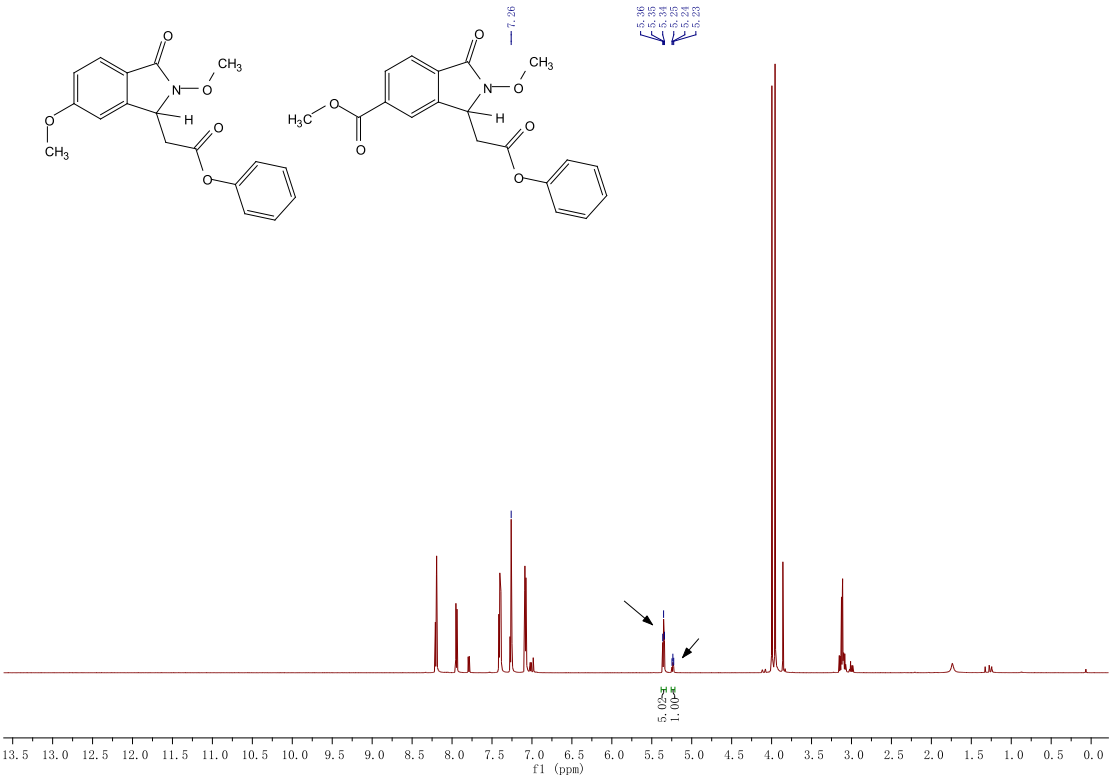
To a mixture of **1aa-*d*$_5$** (0.25 mmol, 99% D), $[Cp^*RhCl_2]_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of **2aa** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 1 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 4:1→Petroleum/EtOAc: 2/1) on silica gel to give product **3aa-*d*$_4$** (131.42 μmol, 39.6 mg, 53% yield).

A kinetic isotopic effect of these two reactions was determined to be $k_H/k_D = 1.0$

Intermolecular Competition Experiments



To a mixture of **1ao** (0.25 mmol), **1as** (0.25 mmol), $[Cp^*RhCl_2]_2$ (5 mol%), NaOAc (0.25 mmol) in a 25 mL Schlenk tube was added a solution of **2aa** (0.3 mmol) in acetone (4.0 mL). Then the tube was capped with septa, and the resulting mixture was stirred at 60 °C in an oil bath for 6 h. After removal of the solvent, the residue was purified by flash chromatography (Petroleum/EtOAc: 8:1→Petroleum/EtOAc: 4/1) on silica gel to give a crude mixture of **3ao** and **3as**. The ratio of **3ao**/**3as** was determined to be **1/5.02** by ^{1}HNMR integration (see below).



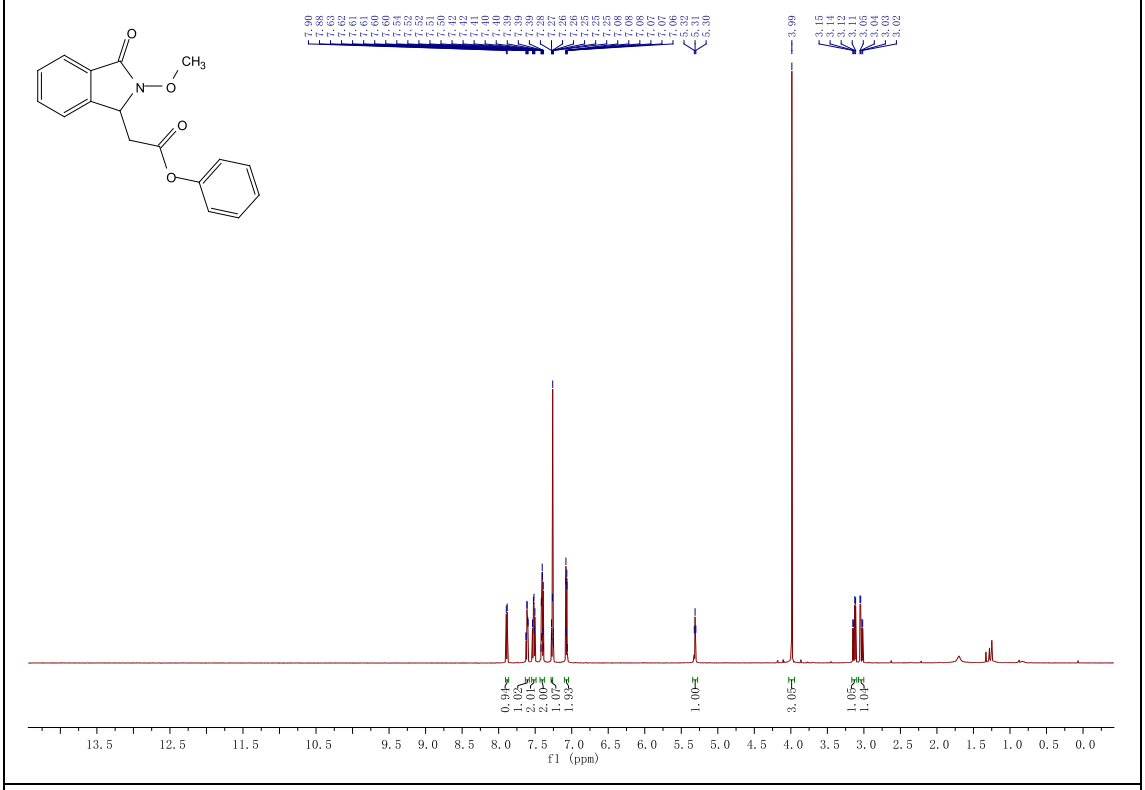
References

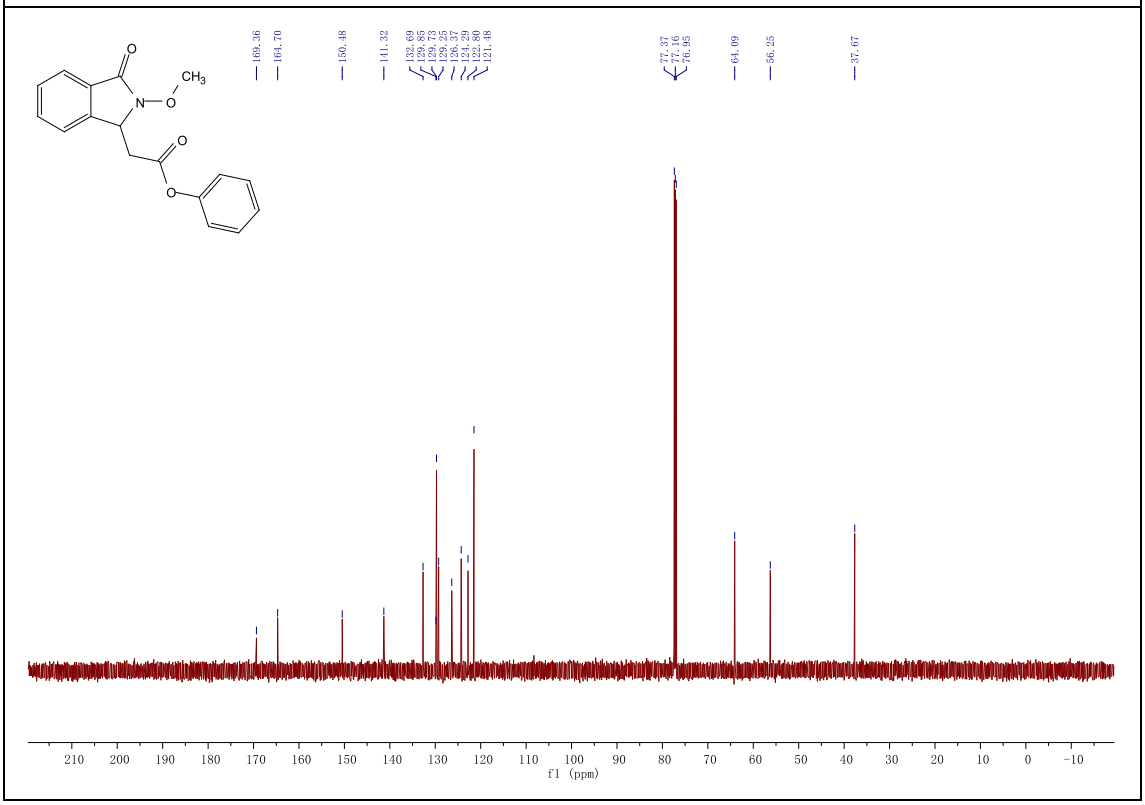
1. (*a*) N. Zhang, Q. Yu, R. Chen, J. Huang, Y. Xia and K. Zhao, *Chem. Commun*., 2013, **49**, 9464–9466; (*b*) X. Wu, B. Wang, S. Zhou, Y. Zhou and H. Liu, *ACS Catal*., 2017, **7**, 2494−2499; (*c*) C.-Q. Wang, L. Ye, C. Feng and T.-P. Loh, *J. Am. Chem. Soc*., 2017, **139**, 1762–1765; (*d*) X. Kou and K. G. M. Kou, *ACS Catal*., 2020, **10**, 3103–3109.

2. (*a*) F. Zhao, J. Qiao, Y. Lu, X. Zhang, L. Dai, S. Liu, H. Ni, X. Jia, X. Wu and S. Lu, *J. Org. Chem*., 2021, **86**, 10591–10607; (*b*) I. Shigeyuki and A. Tomohiro, Patent, WO2009064031A1, 2009; (*c*) H. Nakatsuji, K. Ueno, T. Misaki and Y. Tanabe, *Org. Lett.*, 2008, **10**, 2131–2134; (*d*) H. Nakatsuji, H. Nishikado, K. Ueno and Y. Tanabe, *Org. Lett.*, 2009, **11**, 4258–4261.

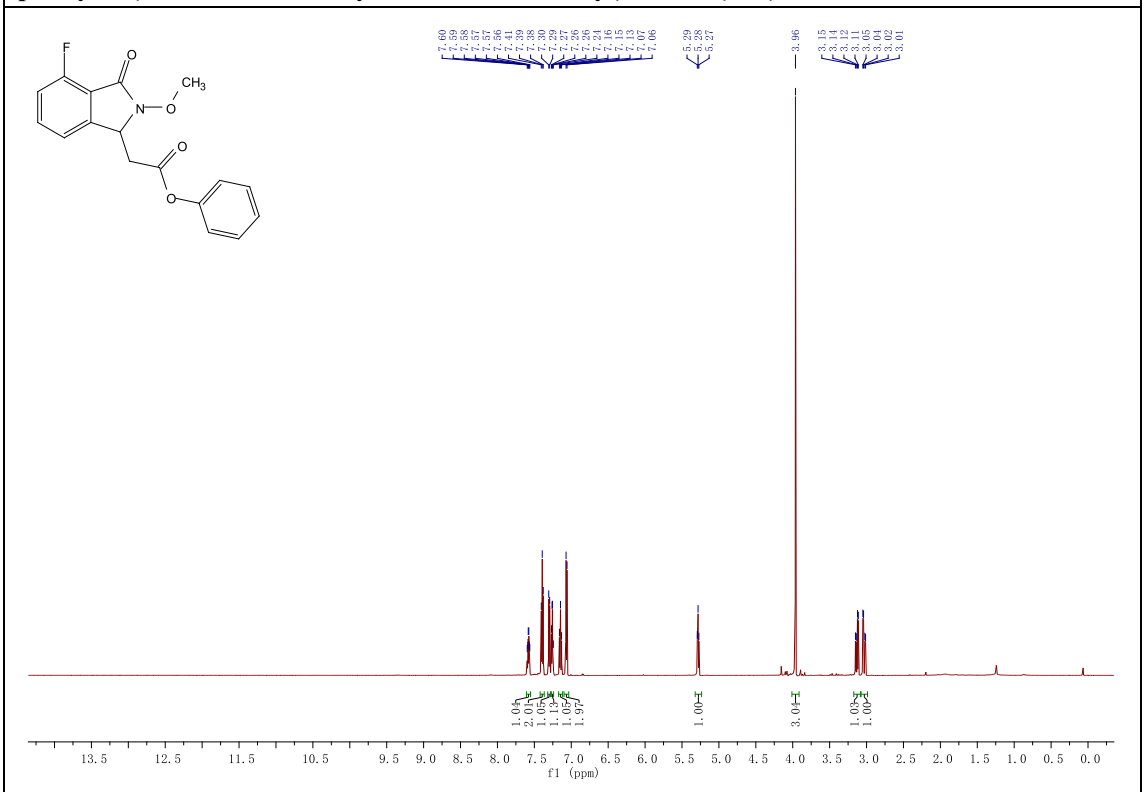
Copies of ^{1}H and ^{13}C NMR Spectra

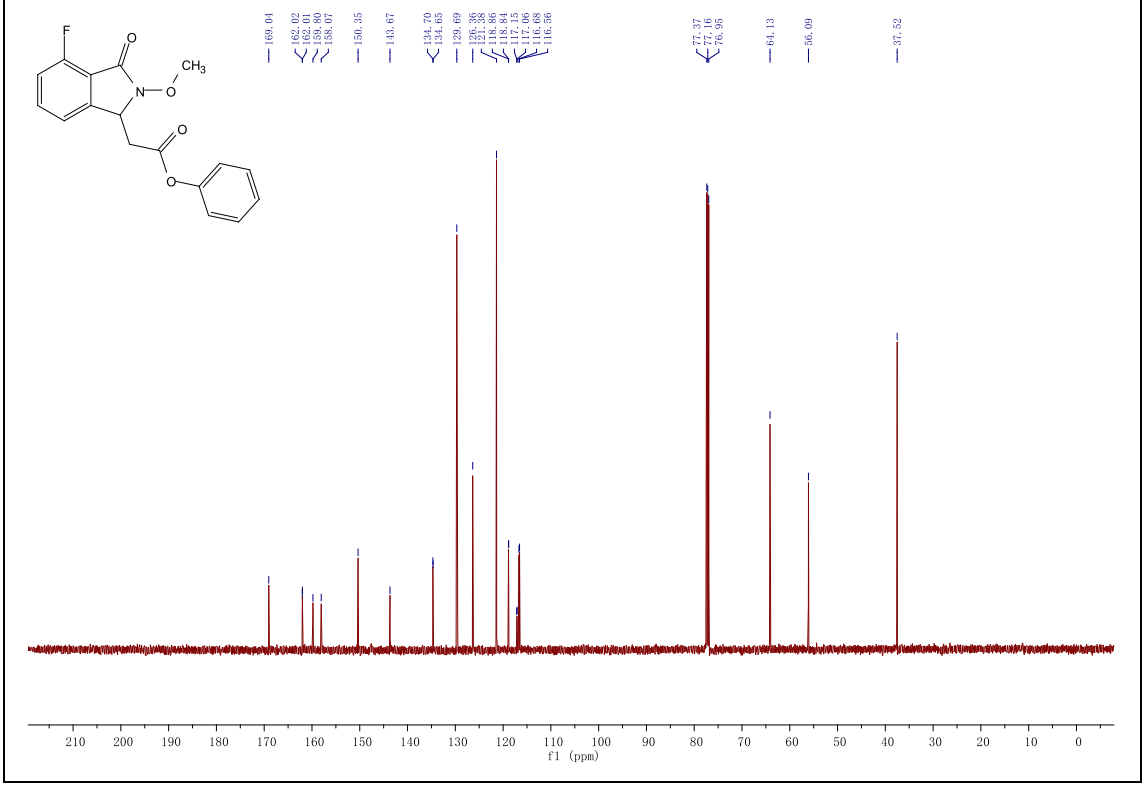
phenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (3aa)



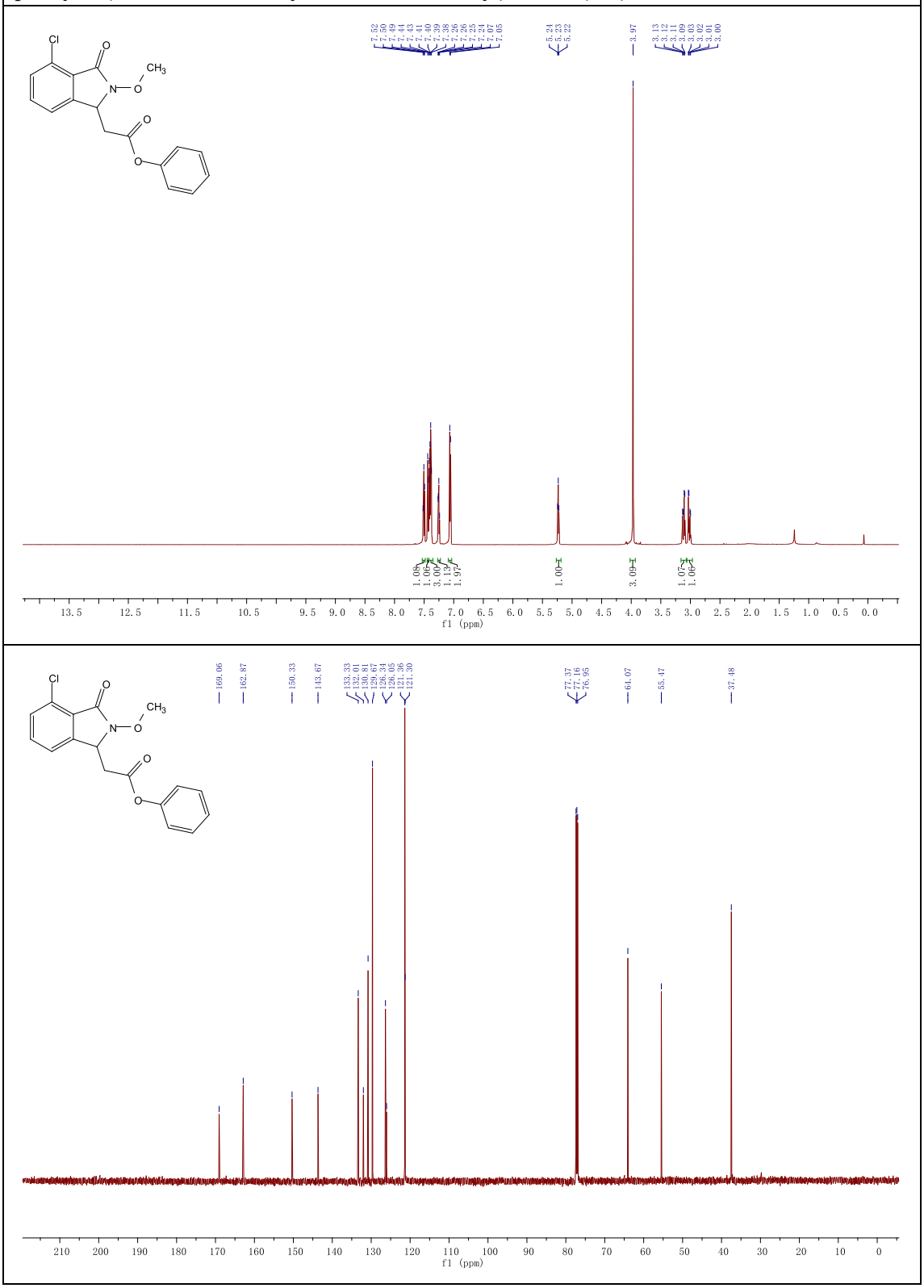


phenyl 2-(4-fluoro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ab)

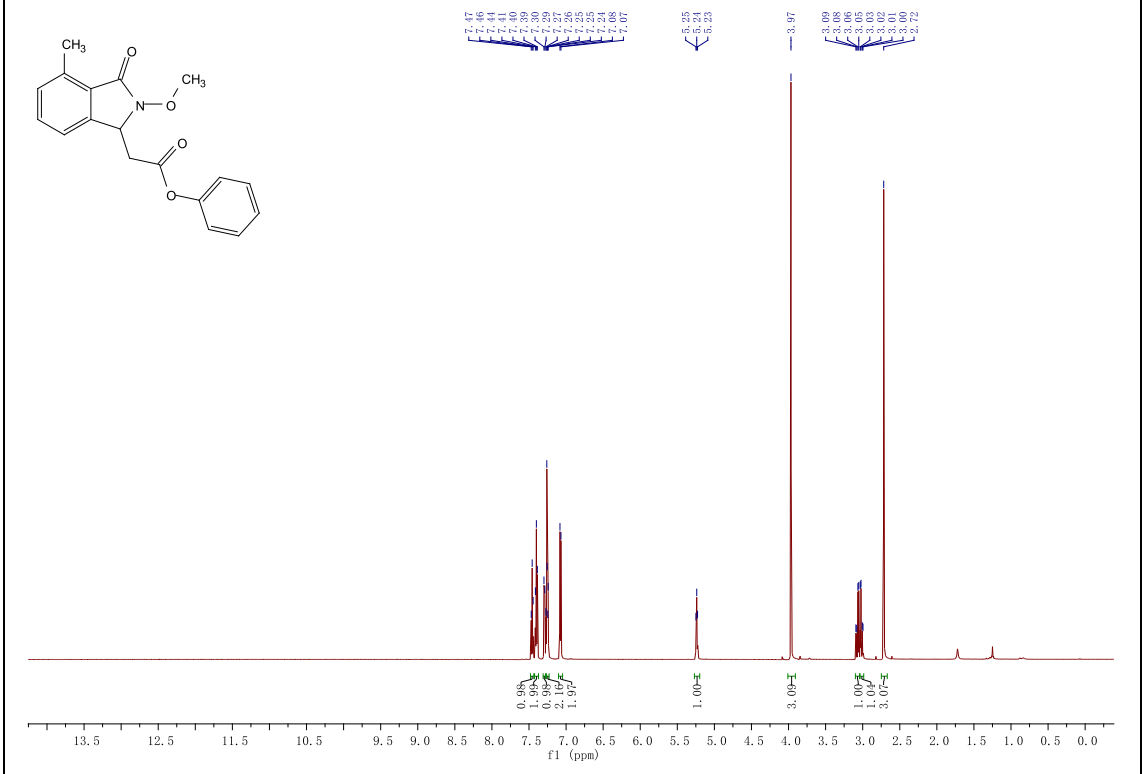


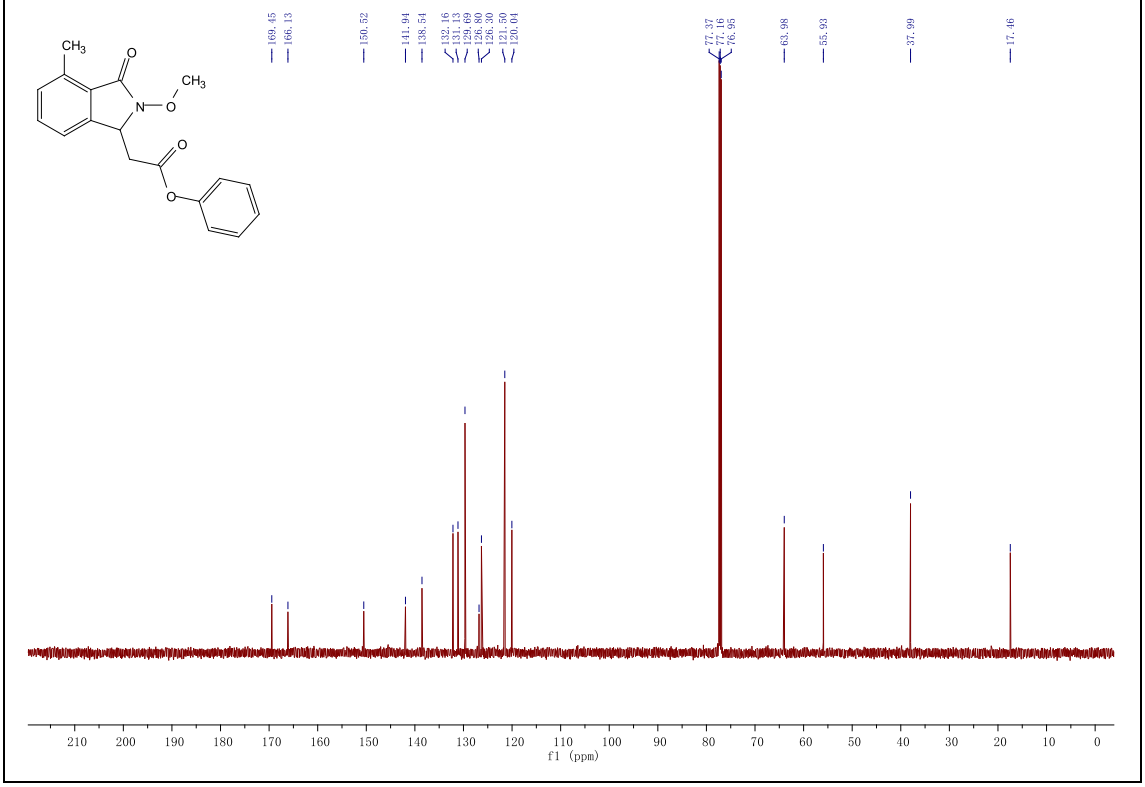


phenyl 2-(4-chloro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ac)

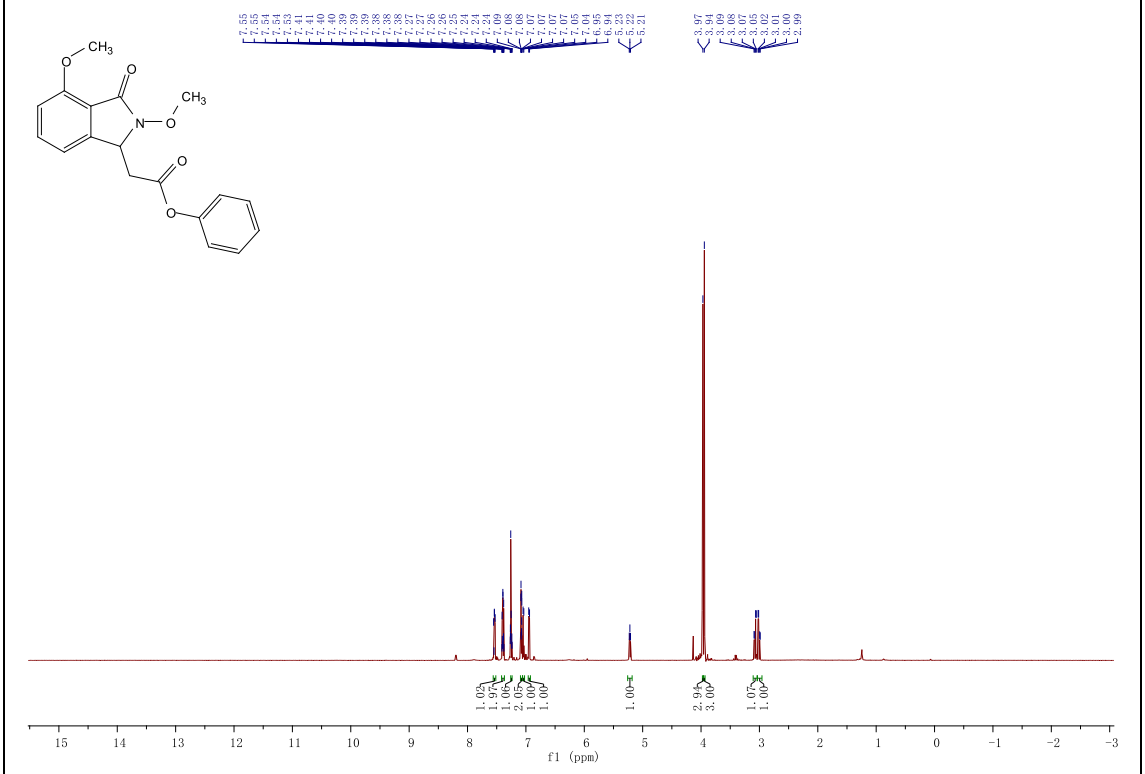


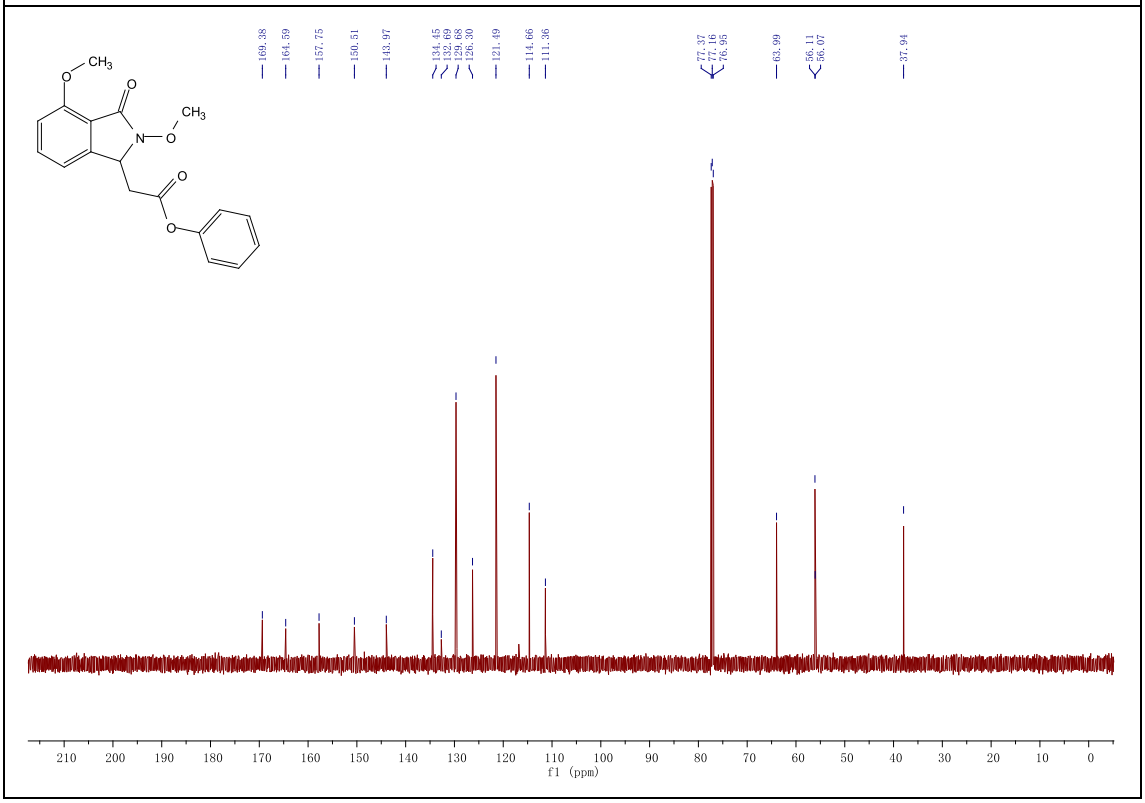
phenyl 2-(2-methoxy-4-methyl-3-oxoisoindolin-1-yl)acetate (3ad)



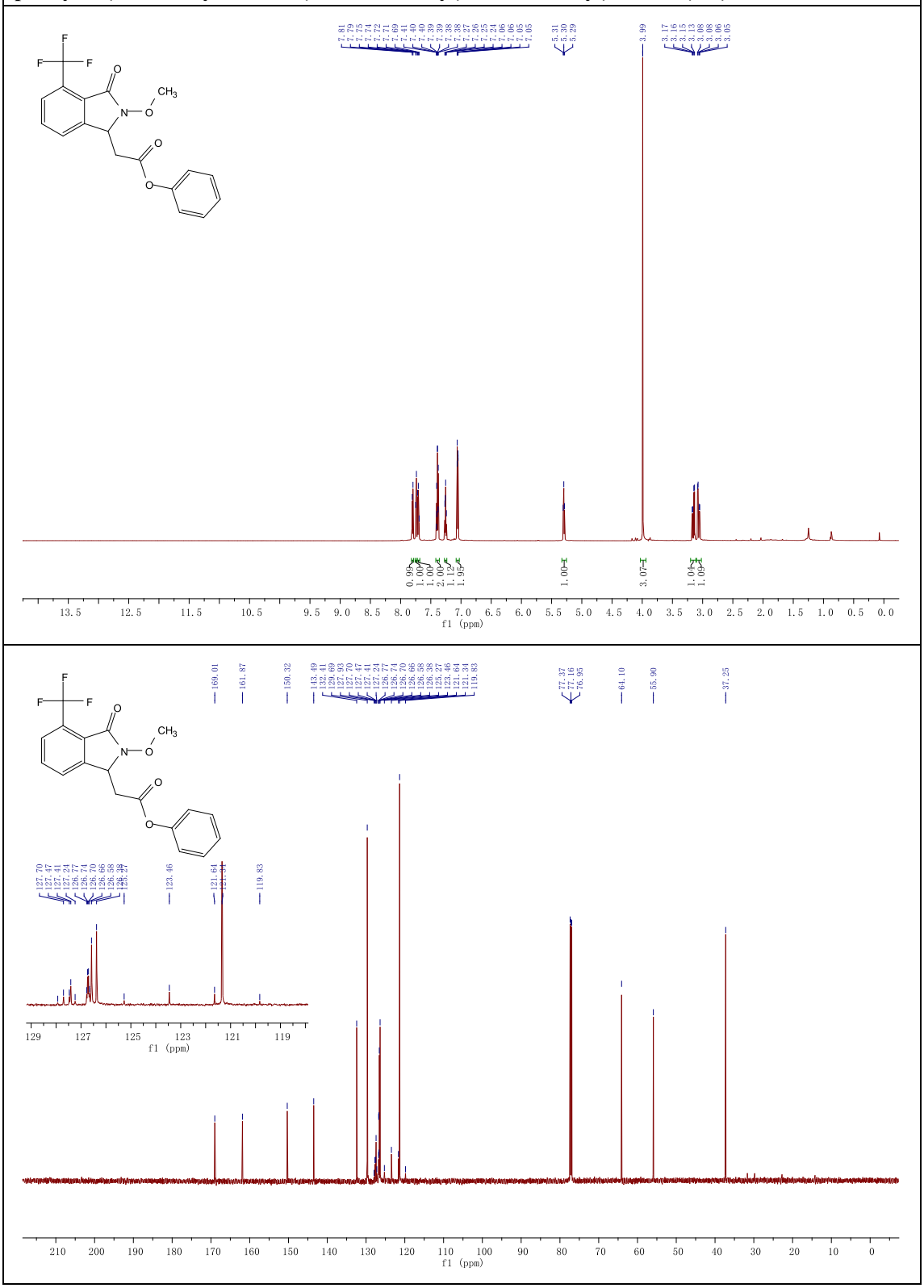


phenyl 2-(2,4-dimethoxy-3-oxoisoindolin-1-yl)acetate (3ae)

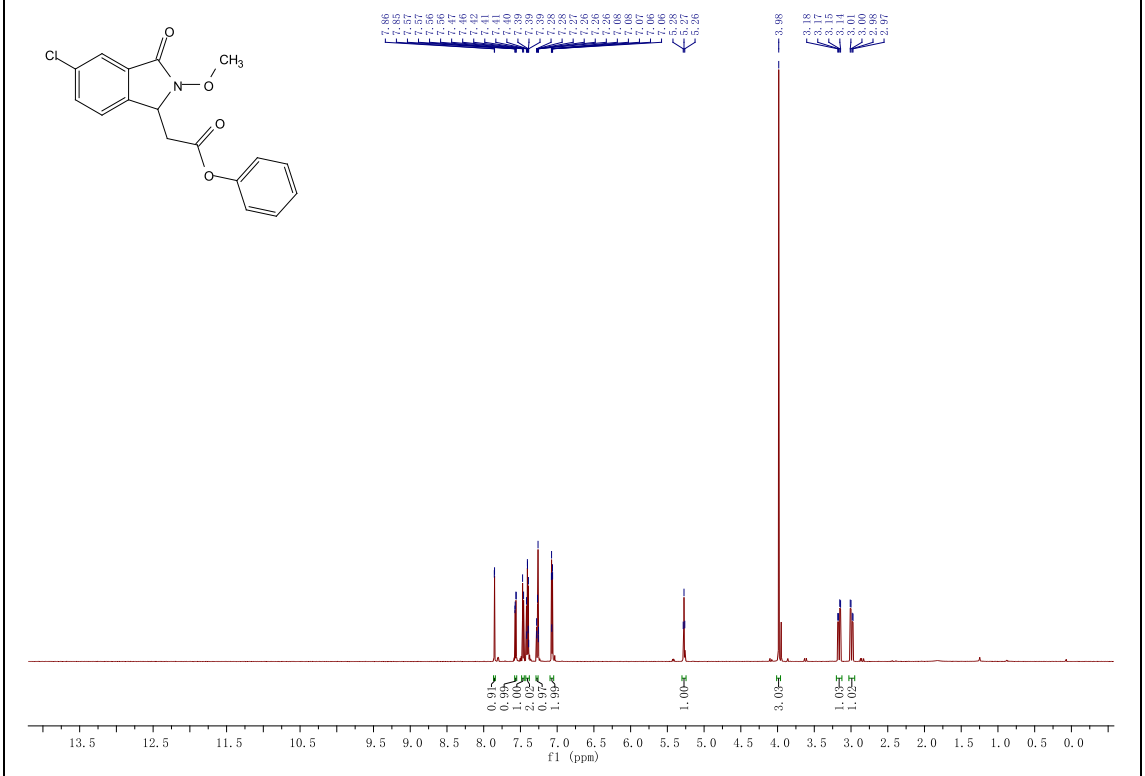


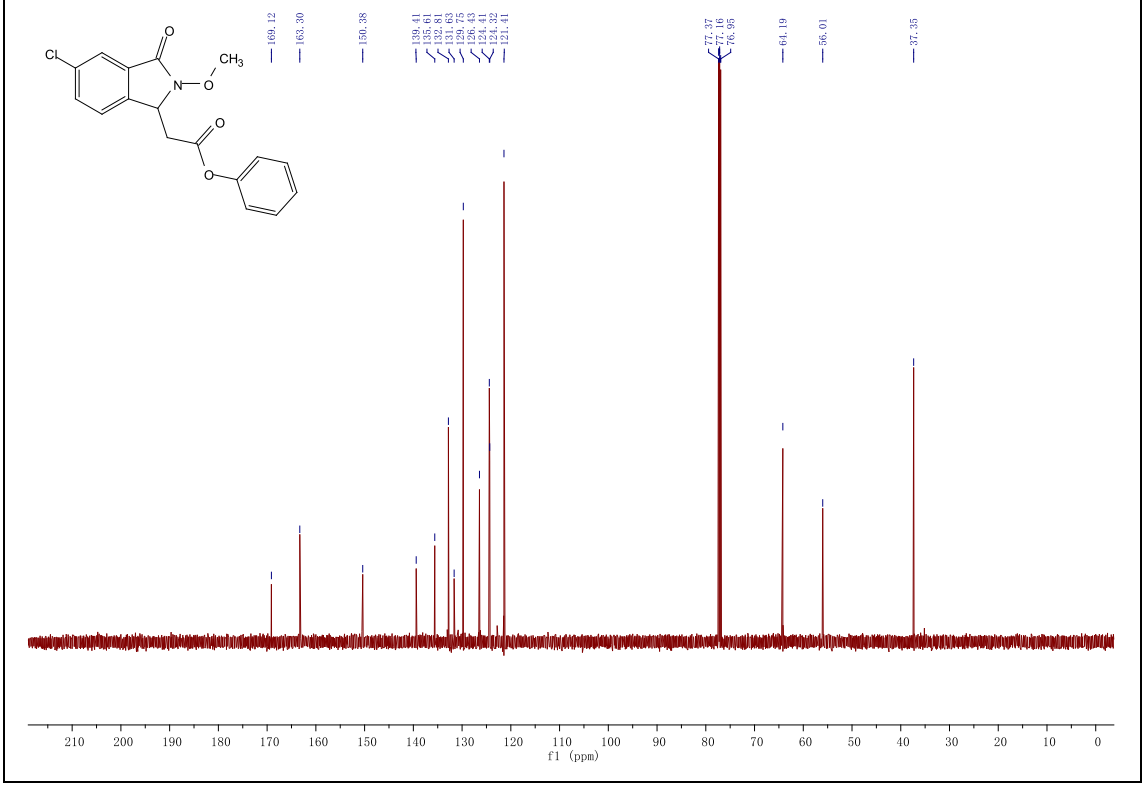


phenyl 2-(2-methoxy-3-oxo-4-(trifluoromethyl)isoindolin-1-yl)acetate (3af)

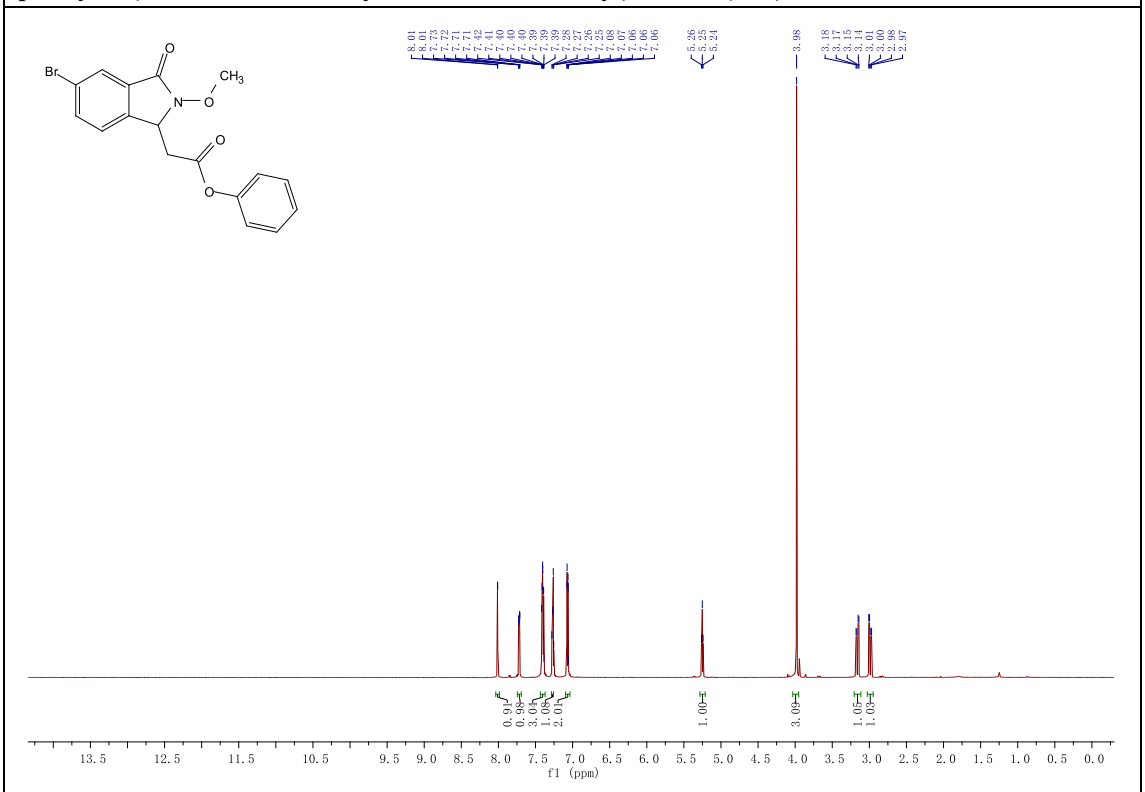


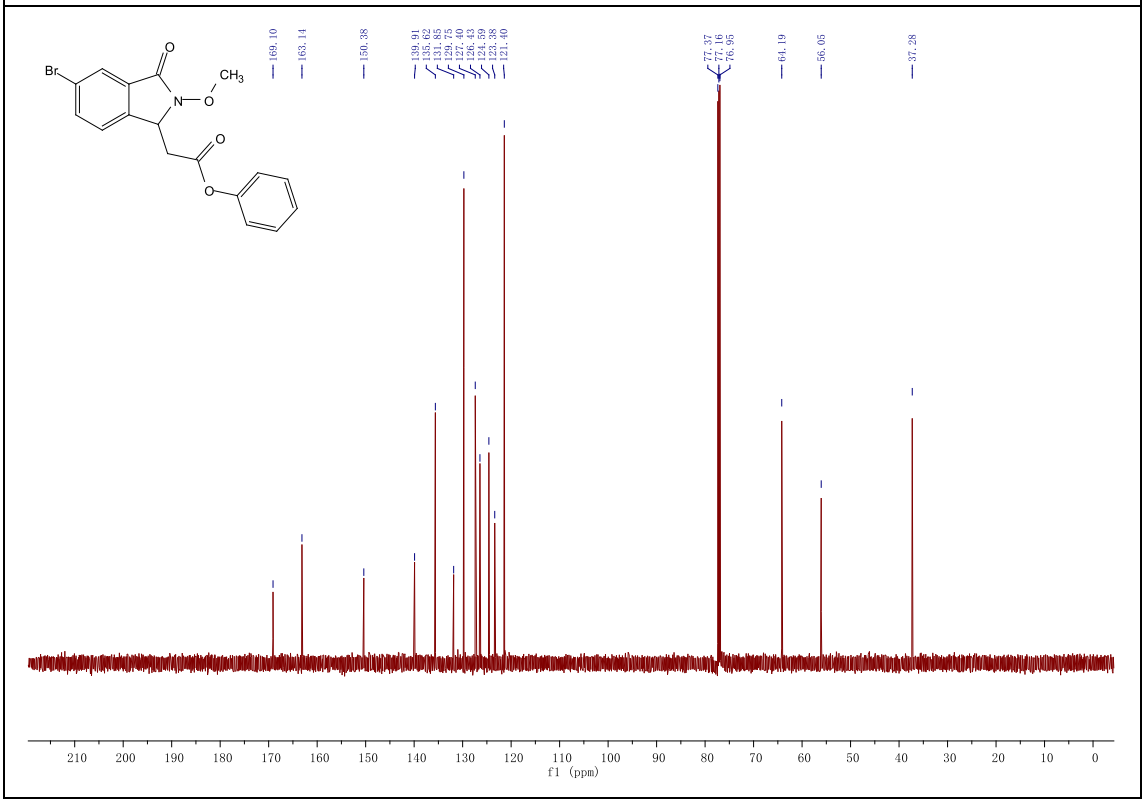
phenyl 2-(5-chloro-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ag)



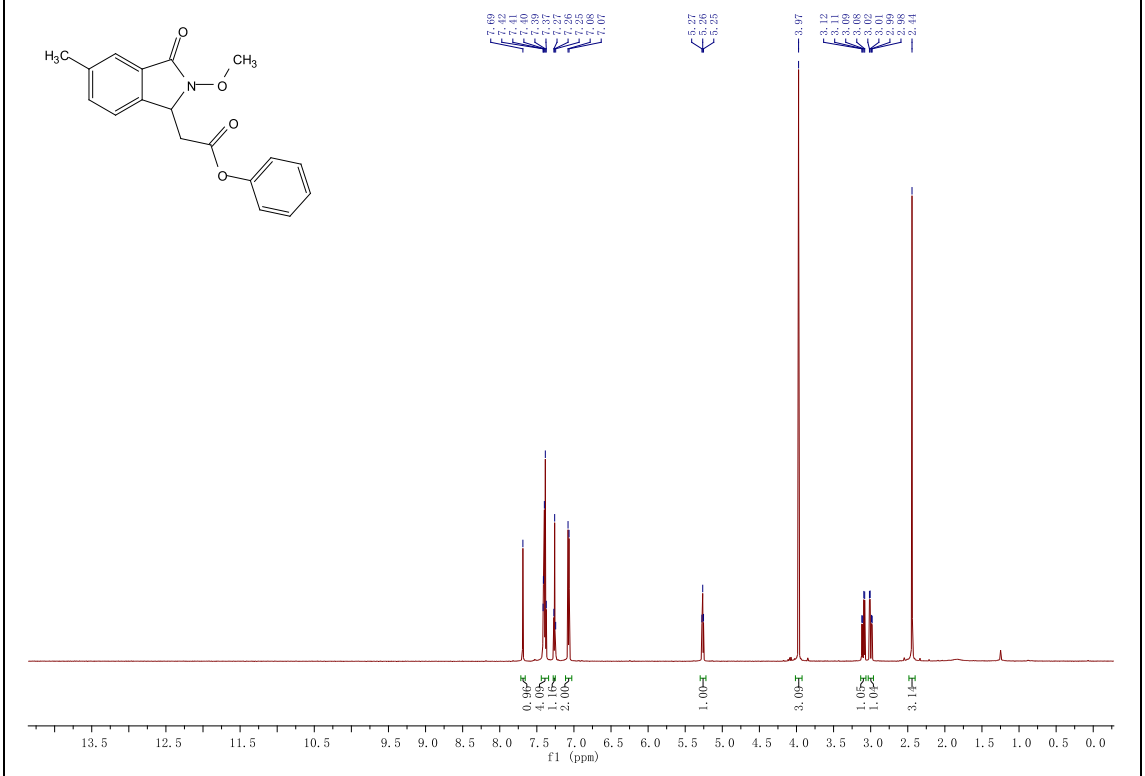


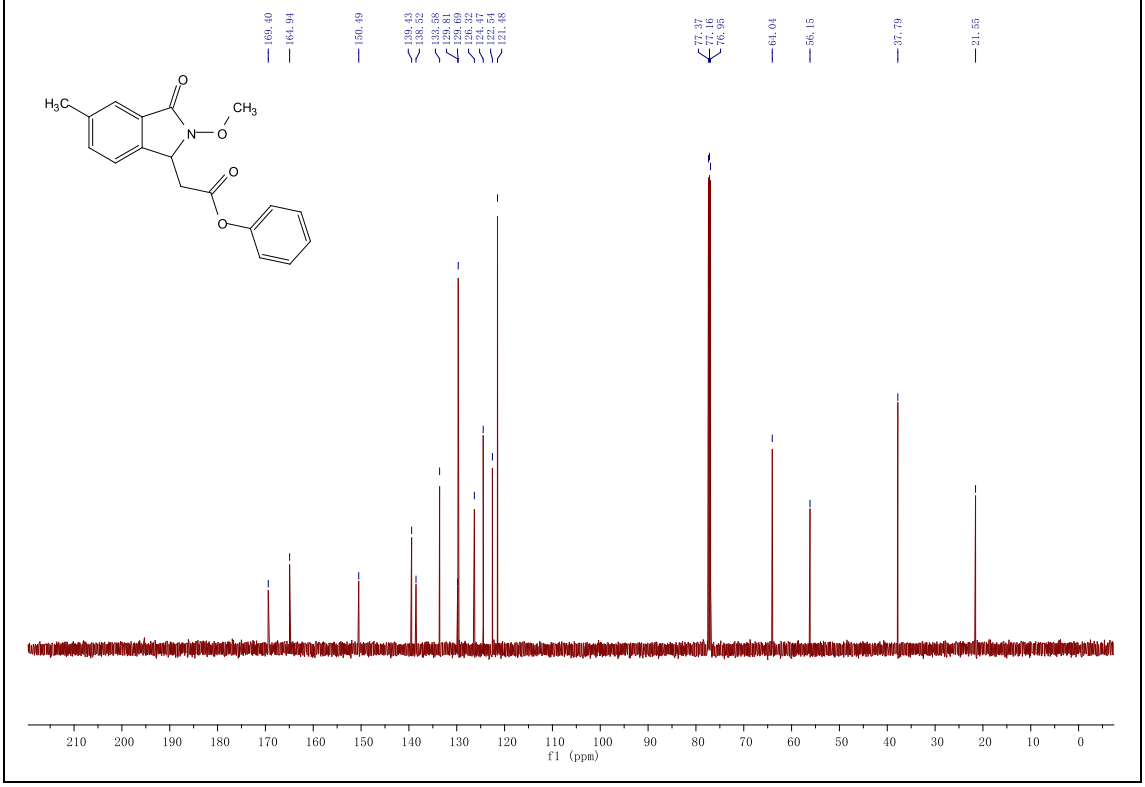
phenyl 2-(5-bromo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ah)



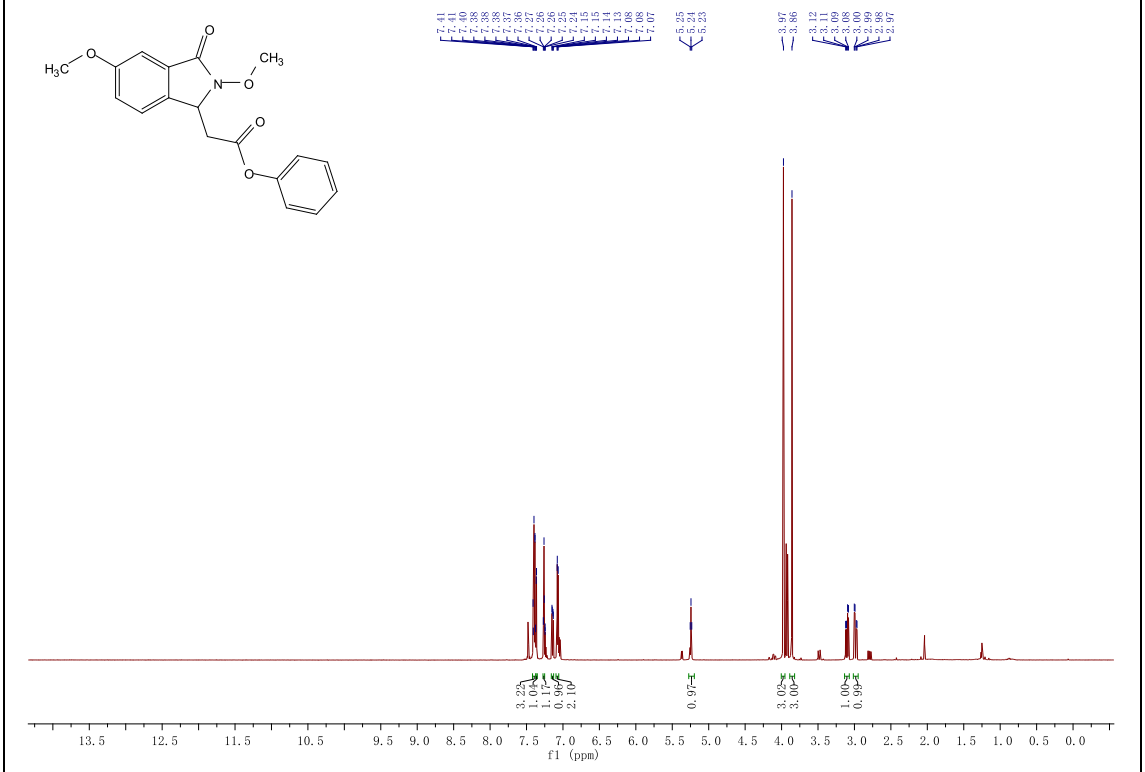


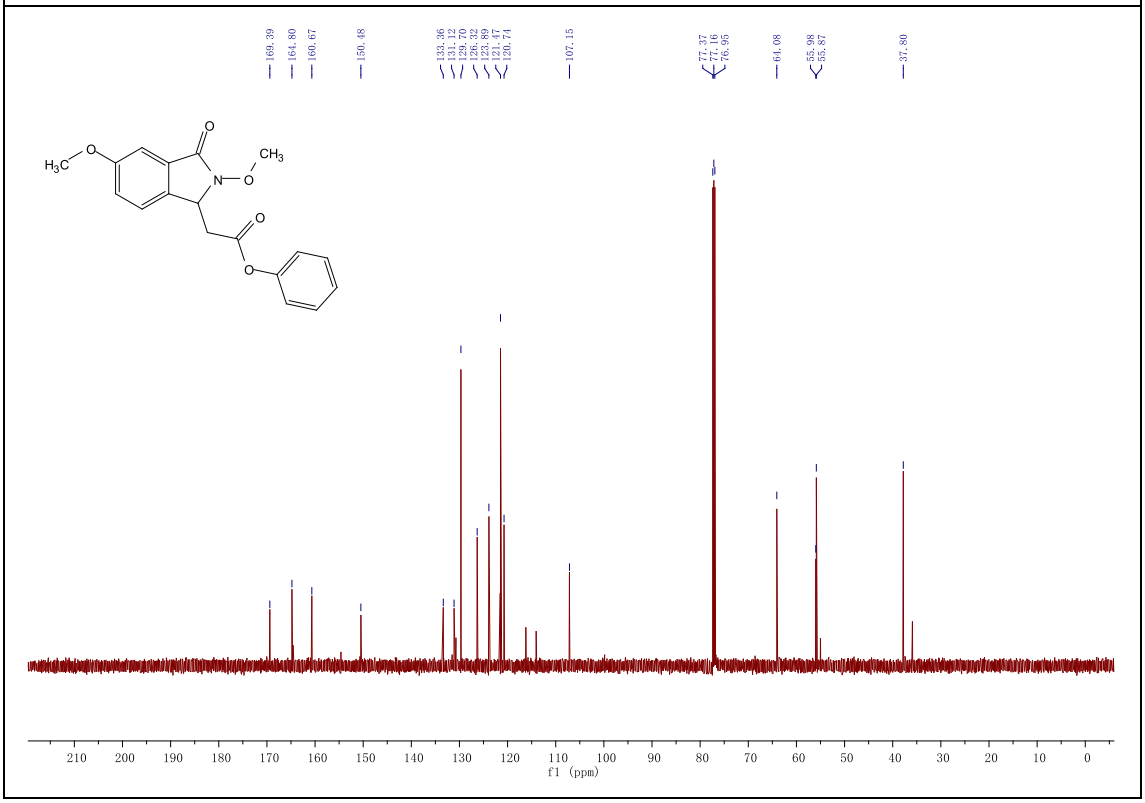
phenyl 2-(2-methoxy-5-methyl-3-oxoisoindolin-1-yl)acetate (3ai)



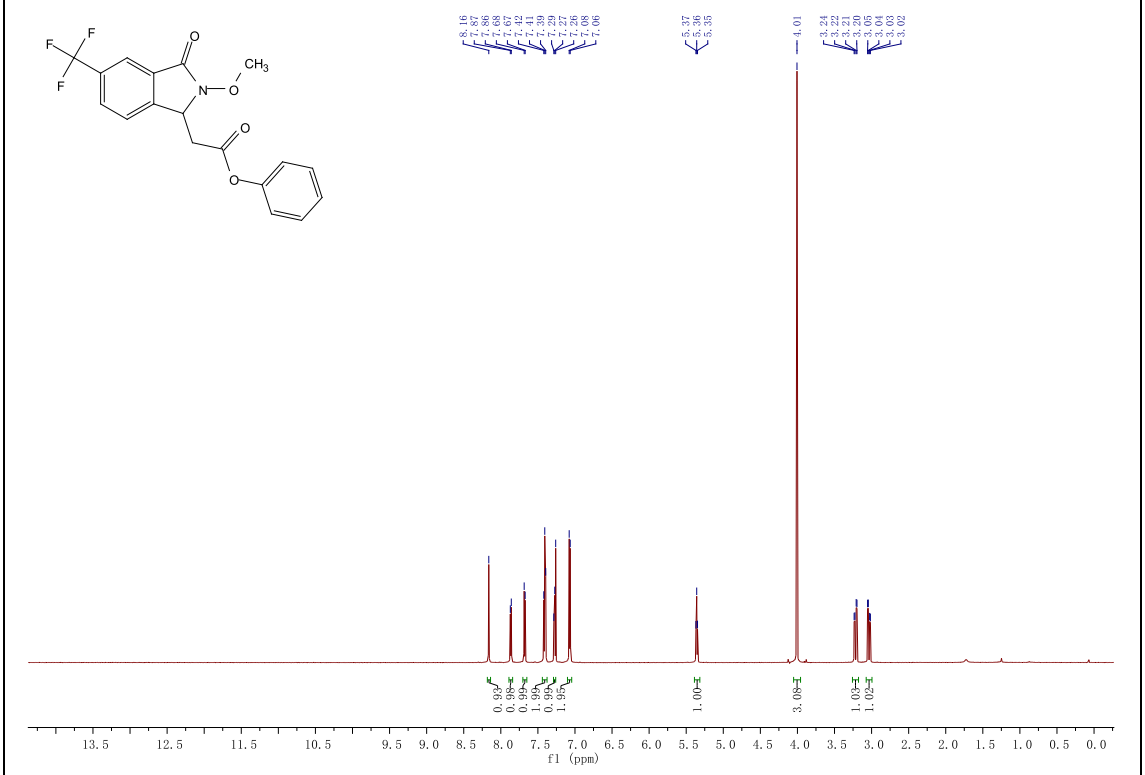


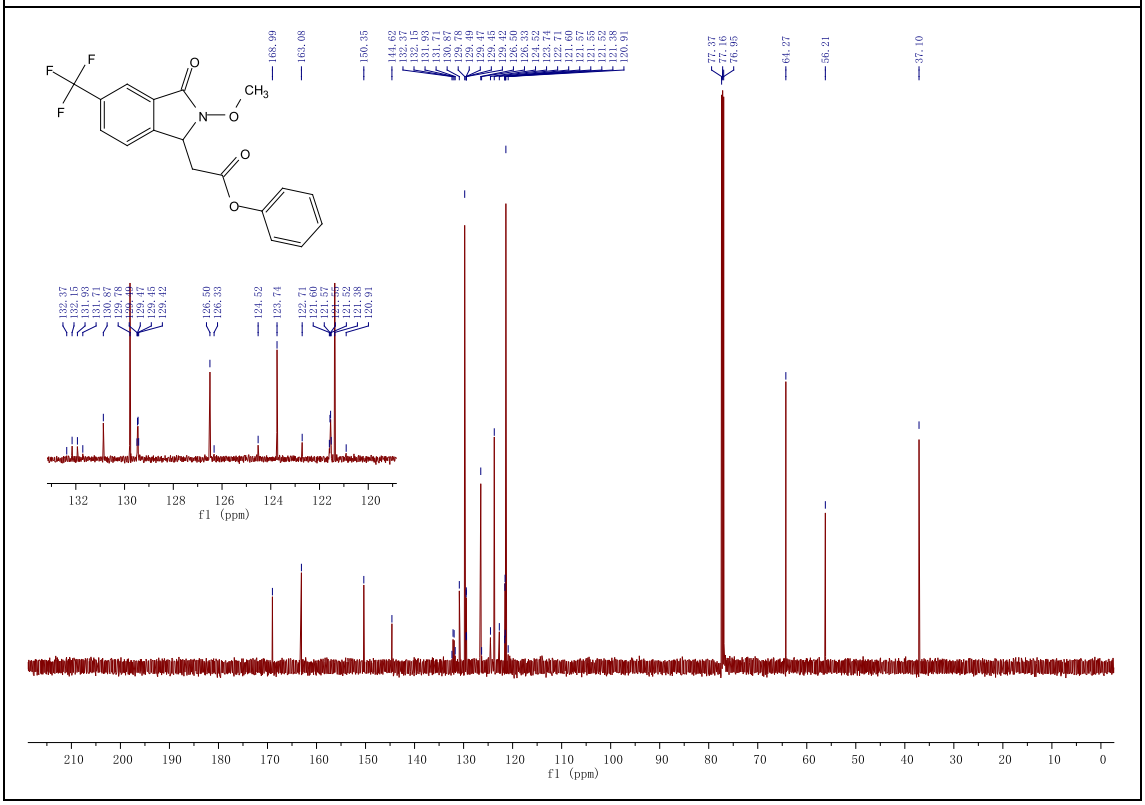
phenyl 2-(2,5-dimethoxy-3-oxoisoindolin-1-yl)acetate (3aj)



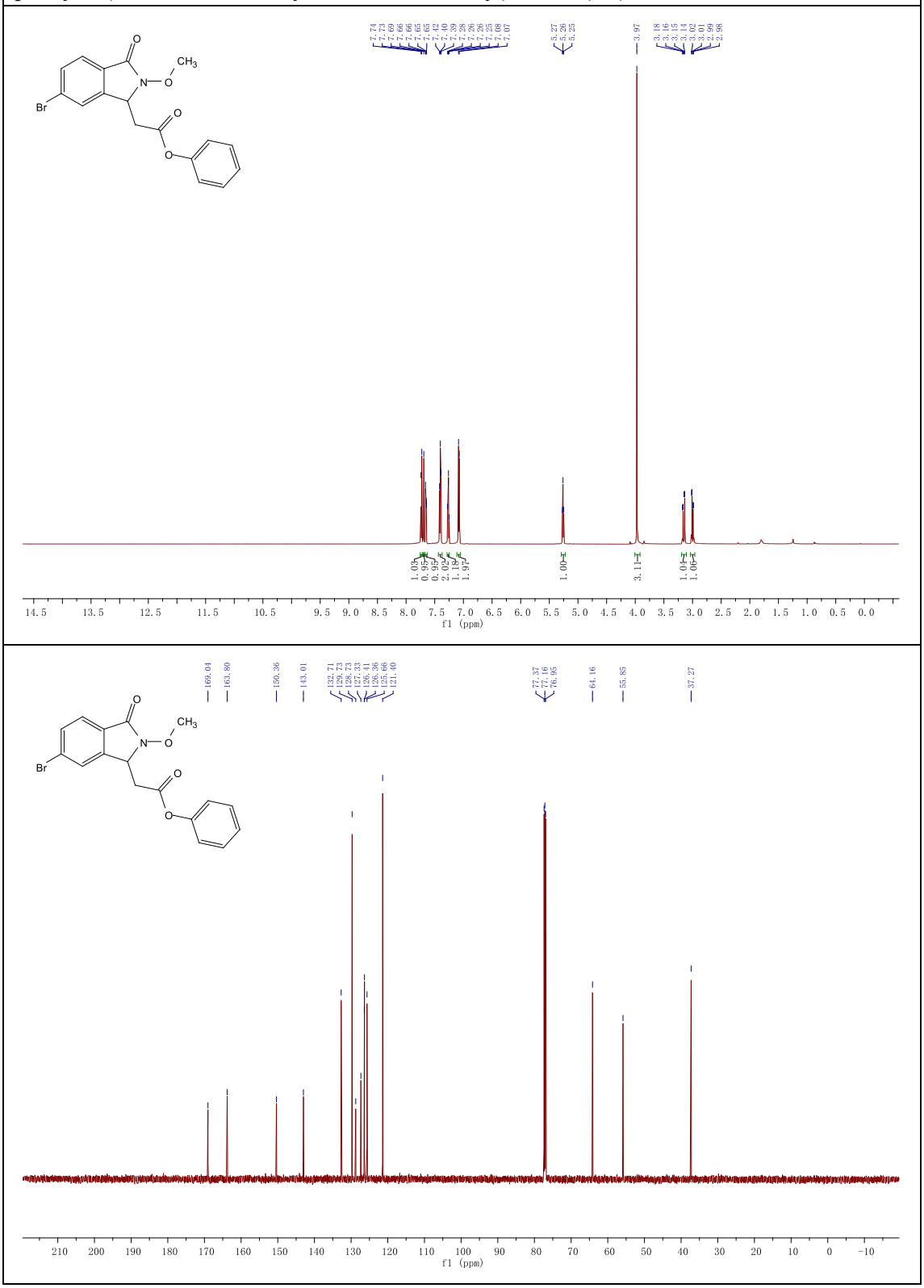


phenyl 2-(2-methoxy-3-oxo-5-(trifluoromethyl)isoindolin-1-yl)acetate (3ak)

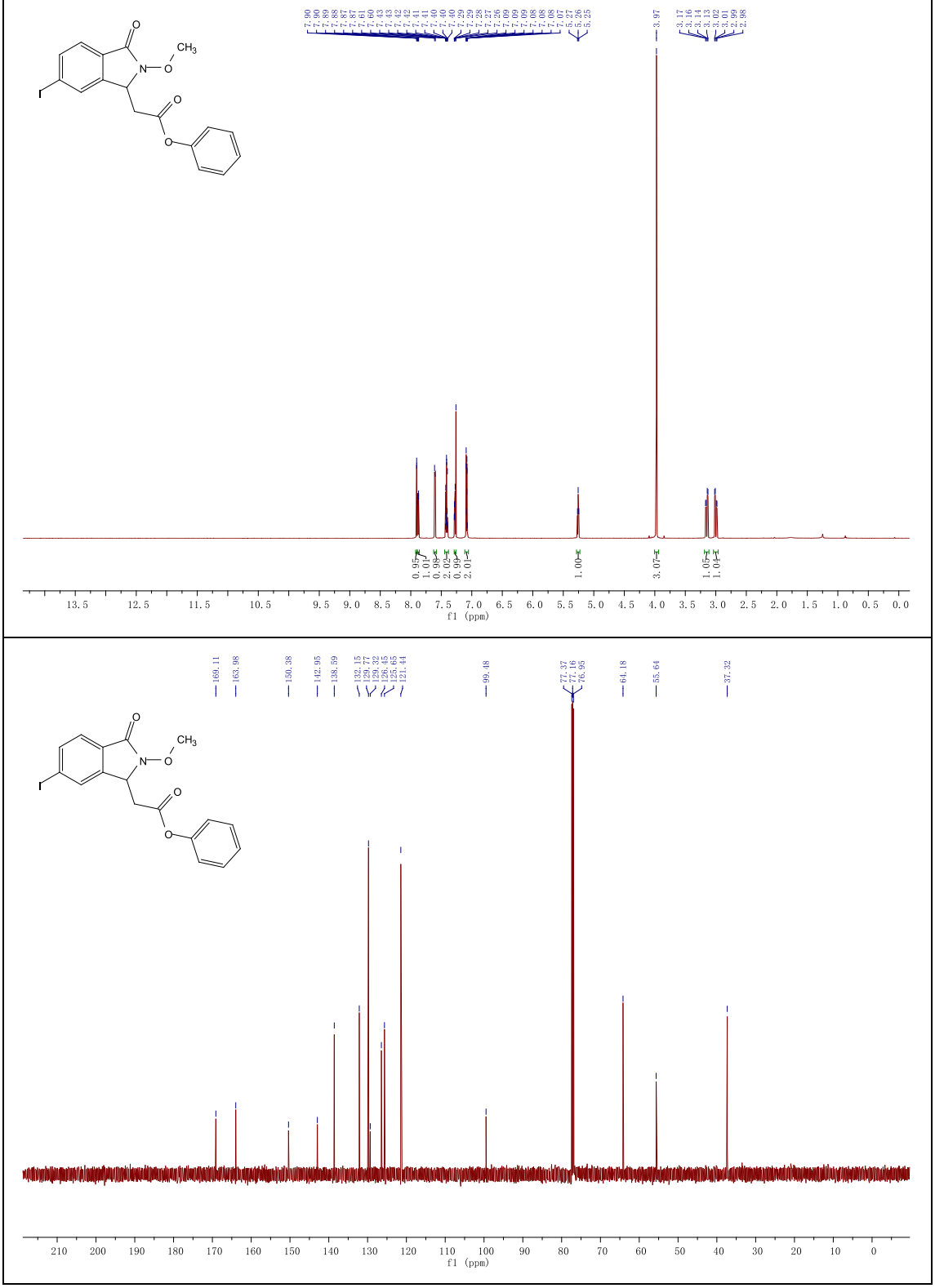




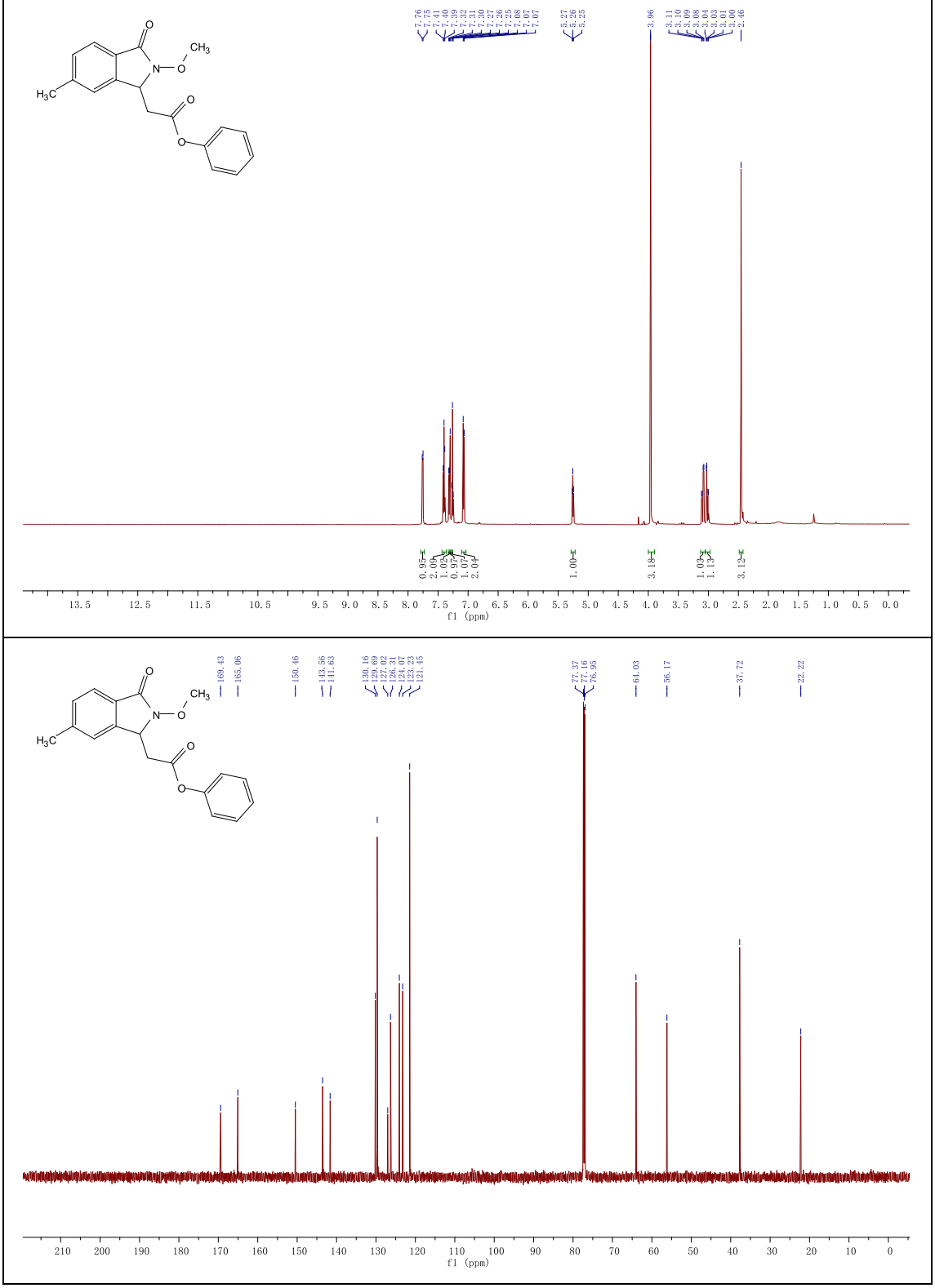
phenyl 2-(6-bromo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3al)



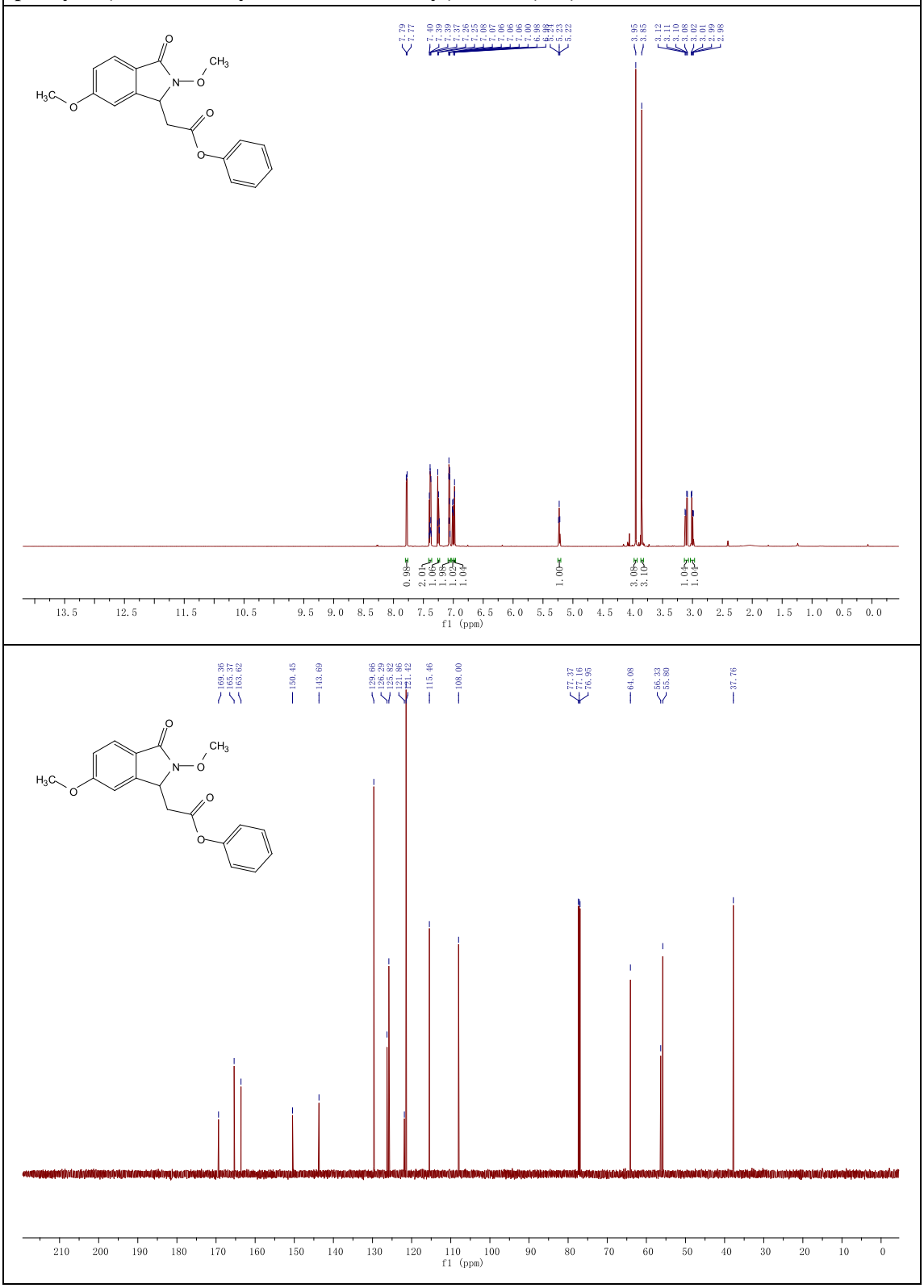
phenyl 2-(6-iodo-2-methoxy-3-oxoisoindolin-1-yl)acetate (3am)



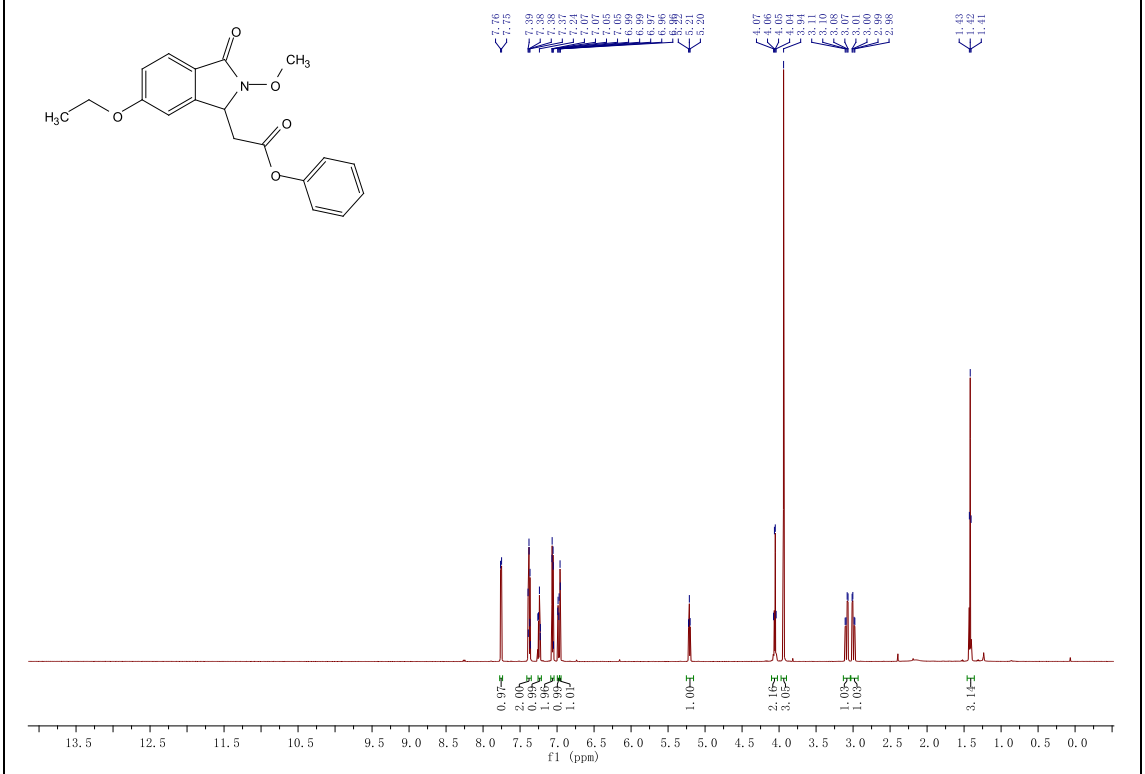
phenyl 2-(2-methoxy-6-methyl-3-oxoisoindolin-1-yl)acetate (3an)

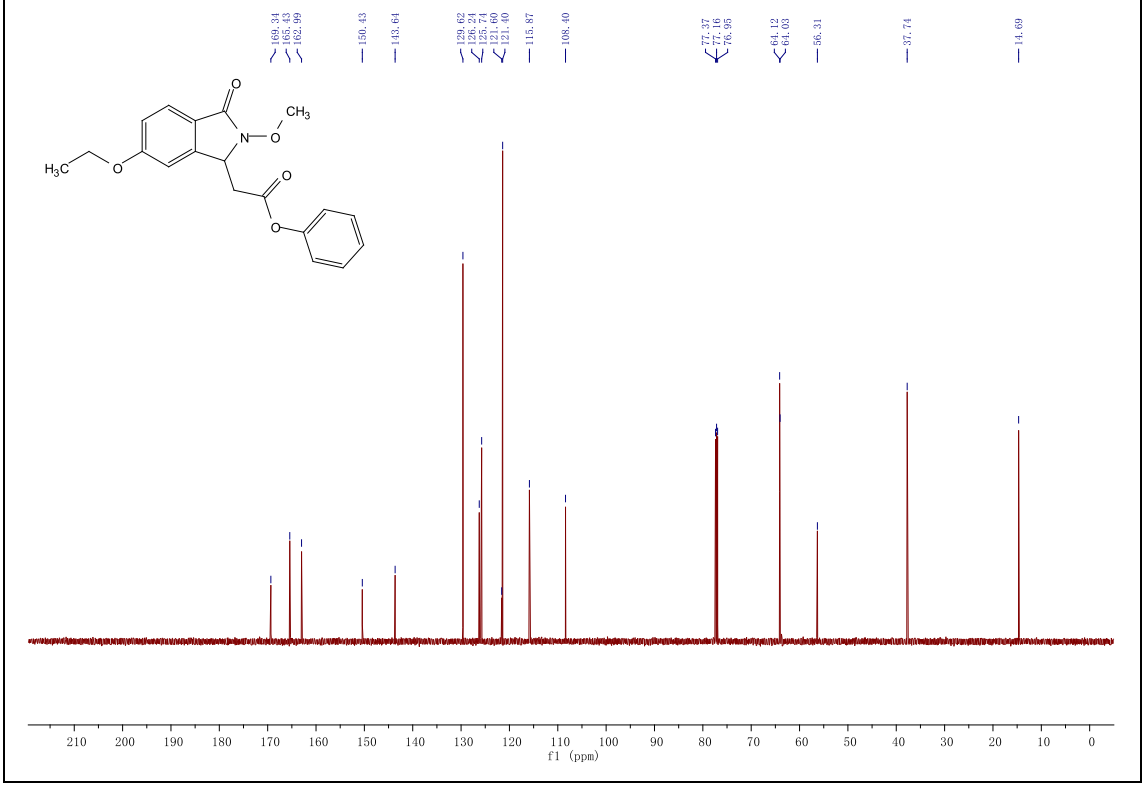


phenyl 2-(2,6-dimethoxy-3-oxoisoindolin-1-yl)acetate (3ao)

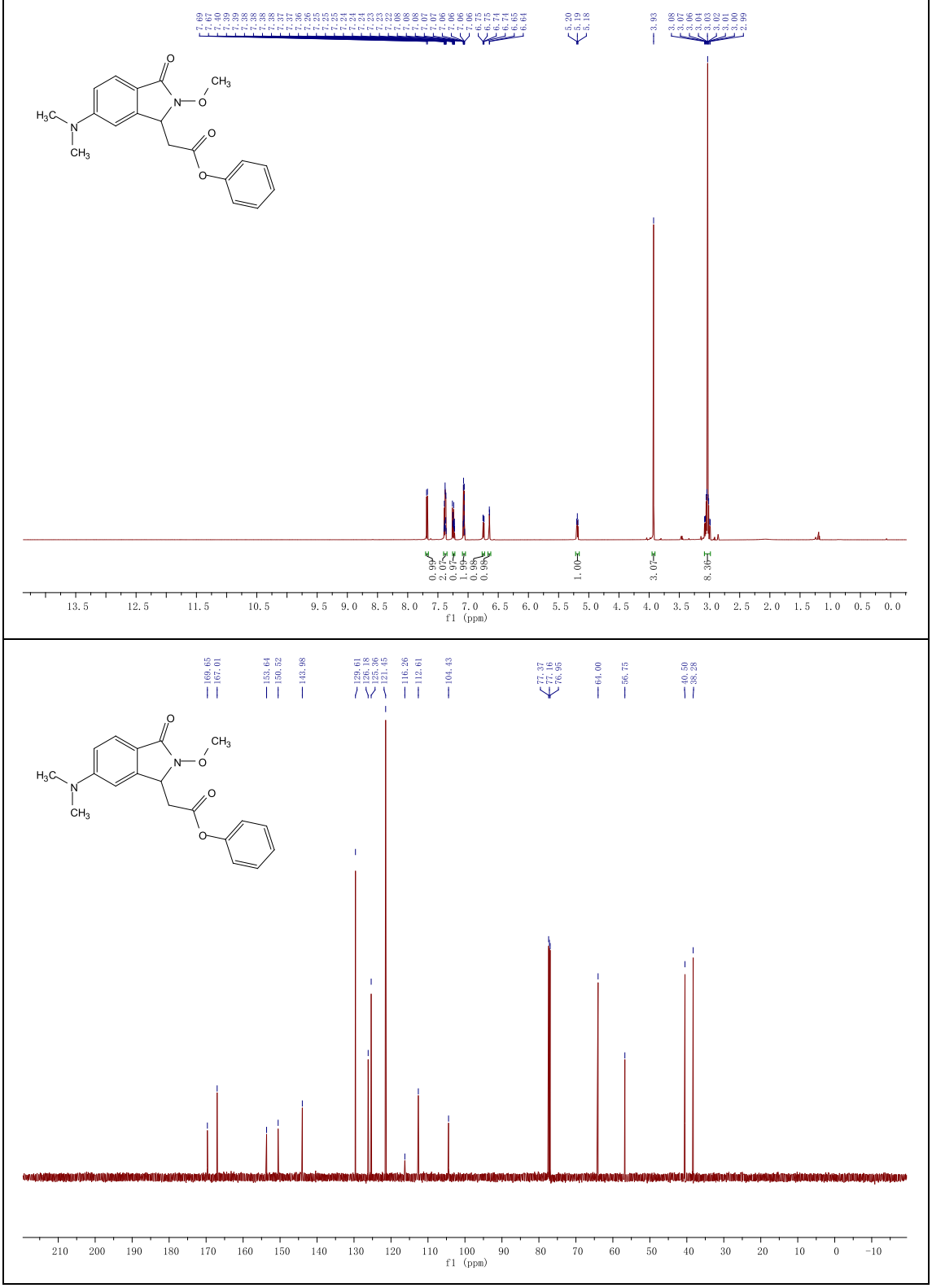


phenyl 2-(6-ethoxy-2-methoxy-3-oxoisoindolin-1-yl)acetate (3ap)

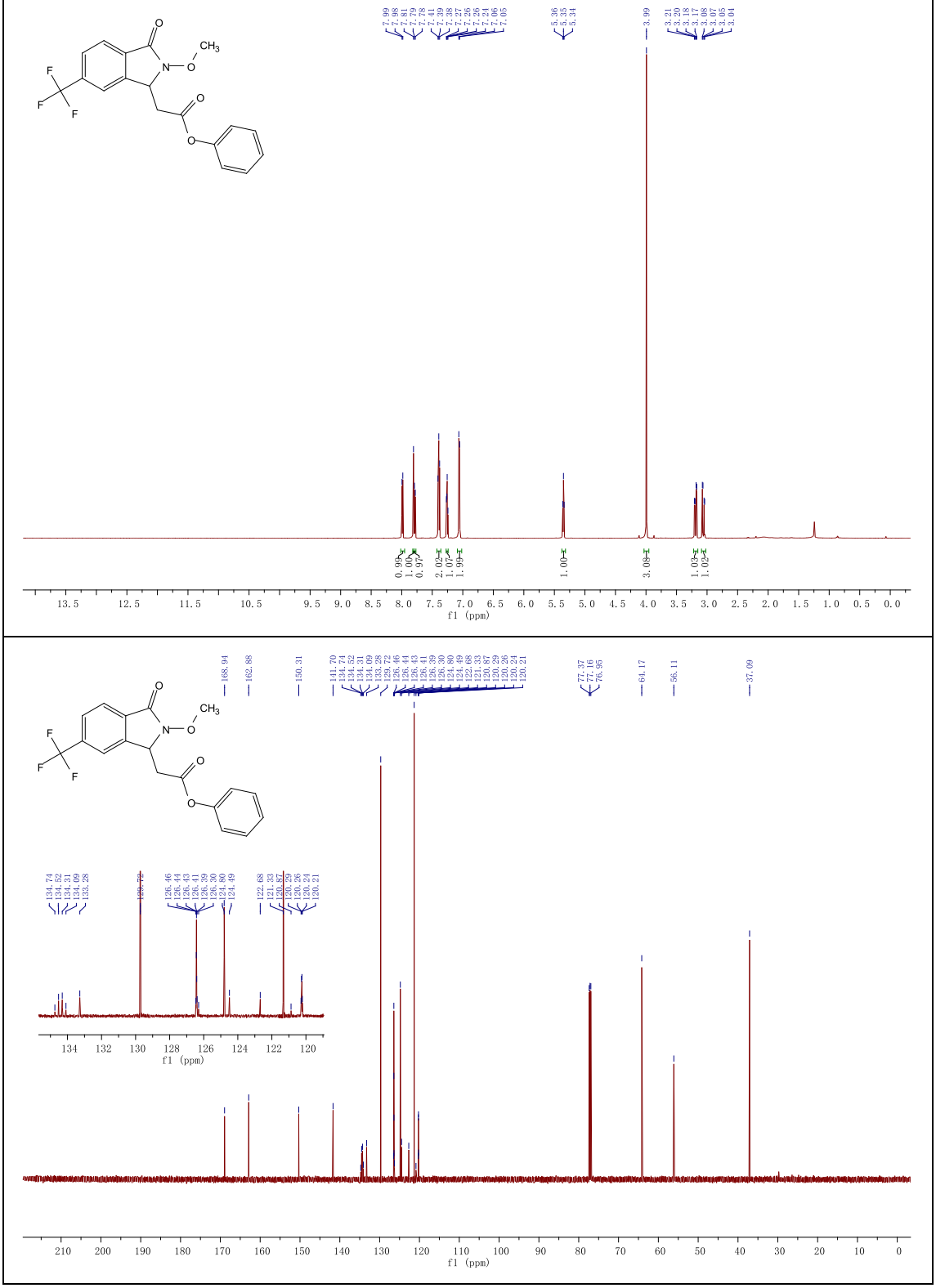




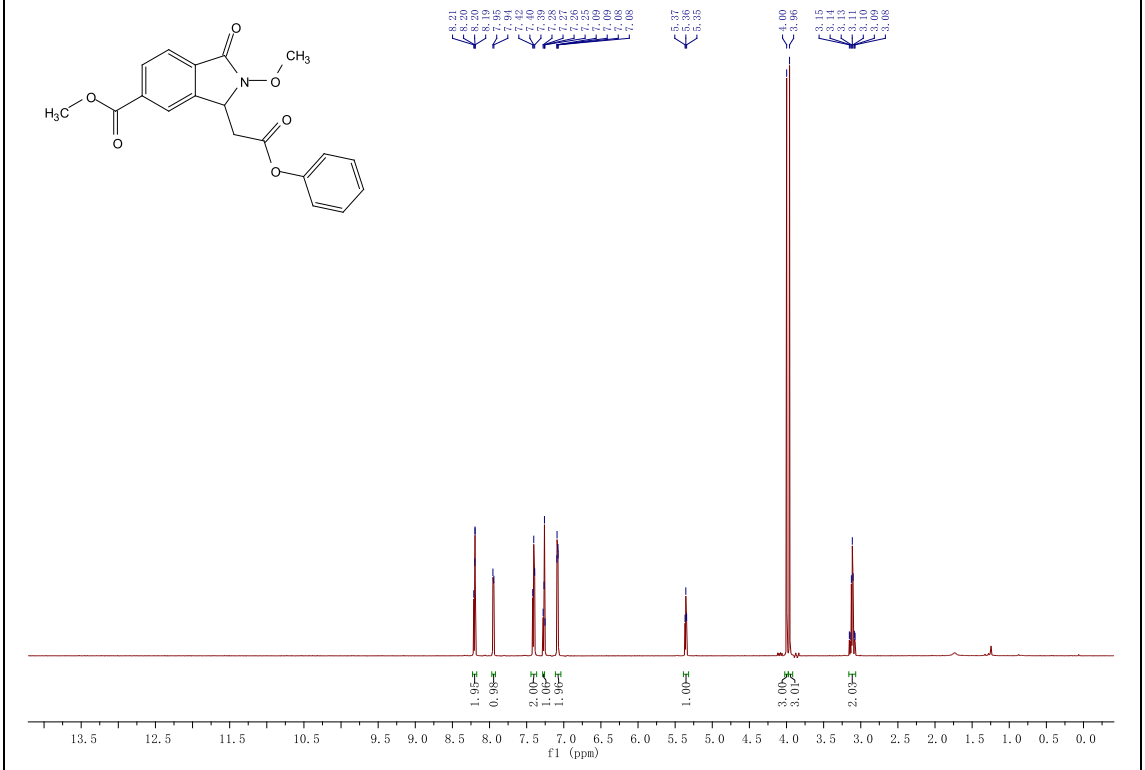
phenyl 2-(6-(dimethylamino)-2-methoxy-3-oxoisoindolin-1-yl)acetate (3aq)

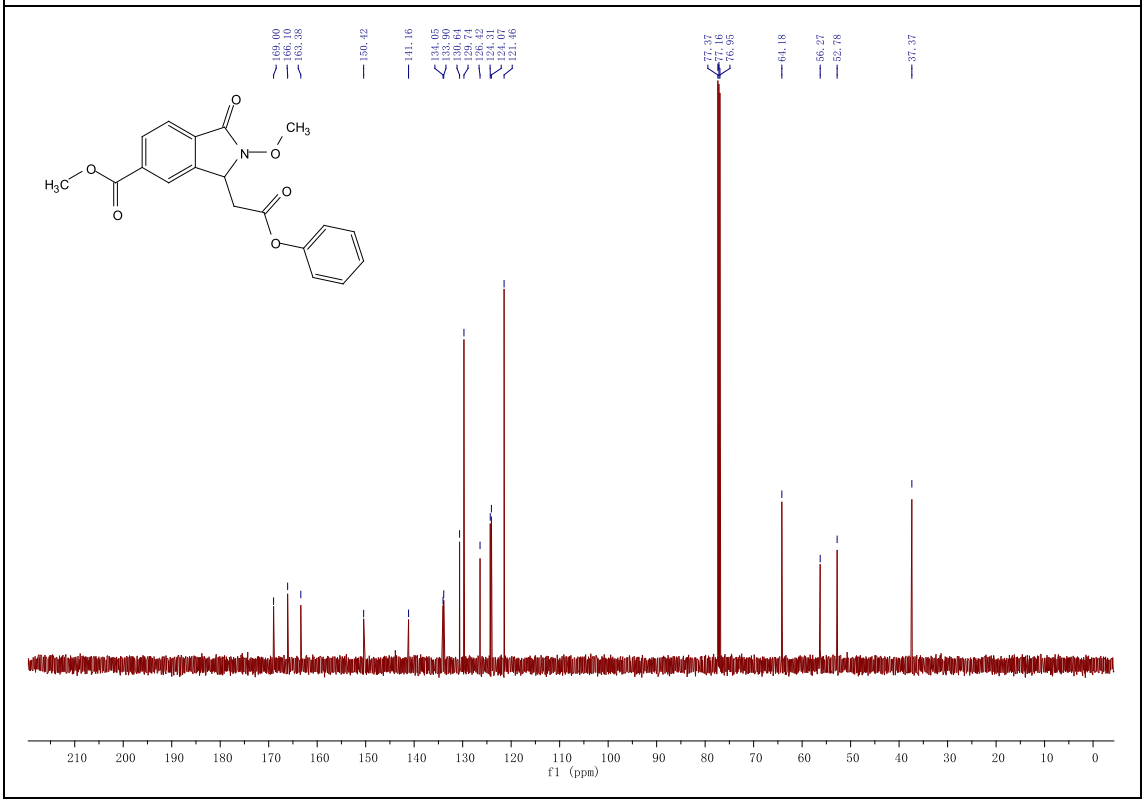


phenyl 2-(2-methoxy-3-oxo-6-(trifluoromethyl)isoindolin-1-yl)acetate (3ar)

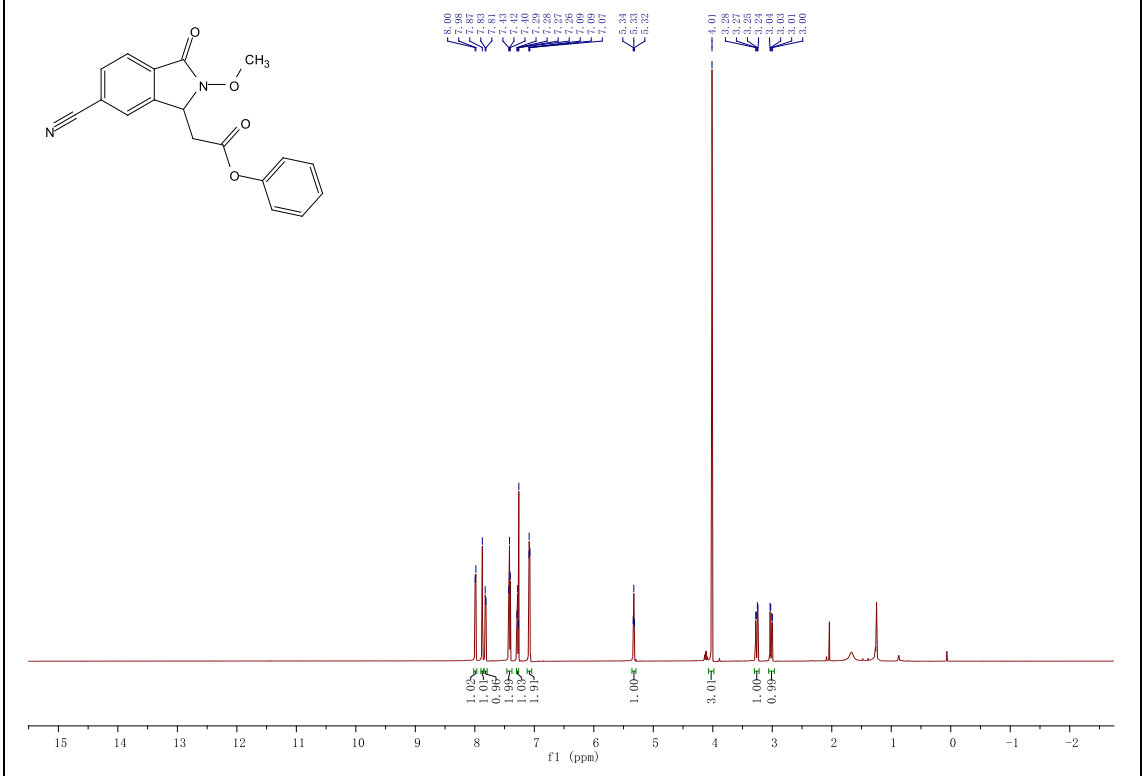


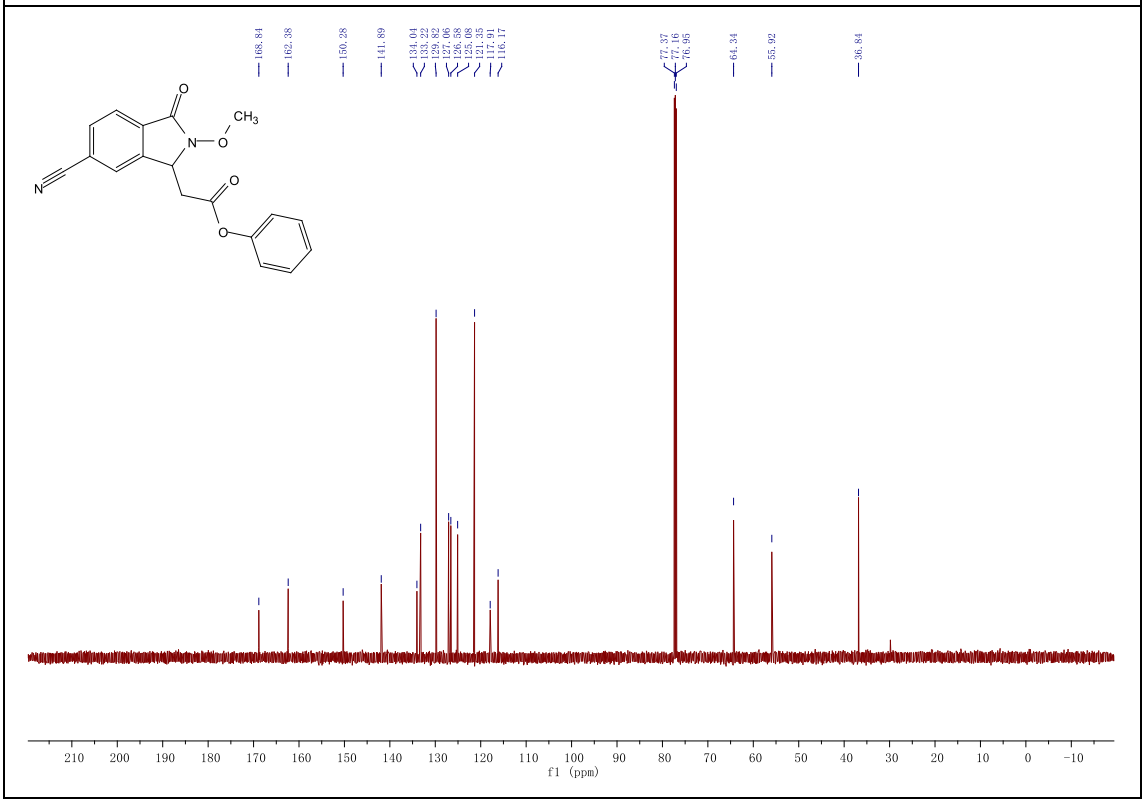
methyl 2-methoxy-1-oxo-3-(2-oxo-2-phenoxyethyl)isoindoline-5-carboxylate (3as)



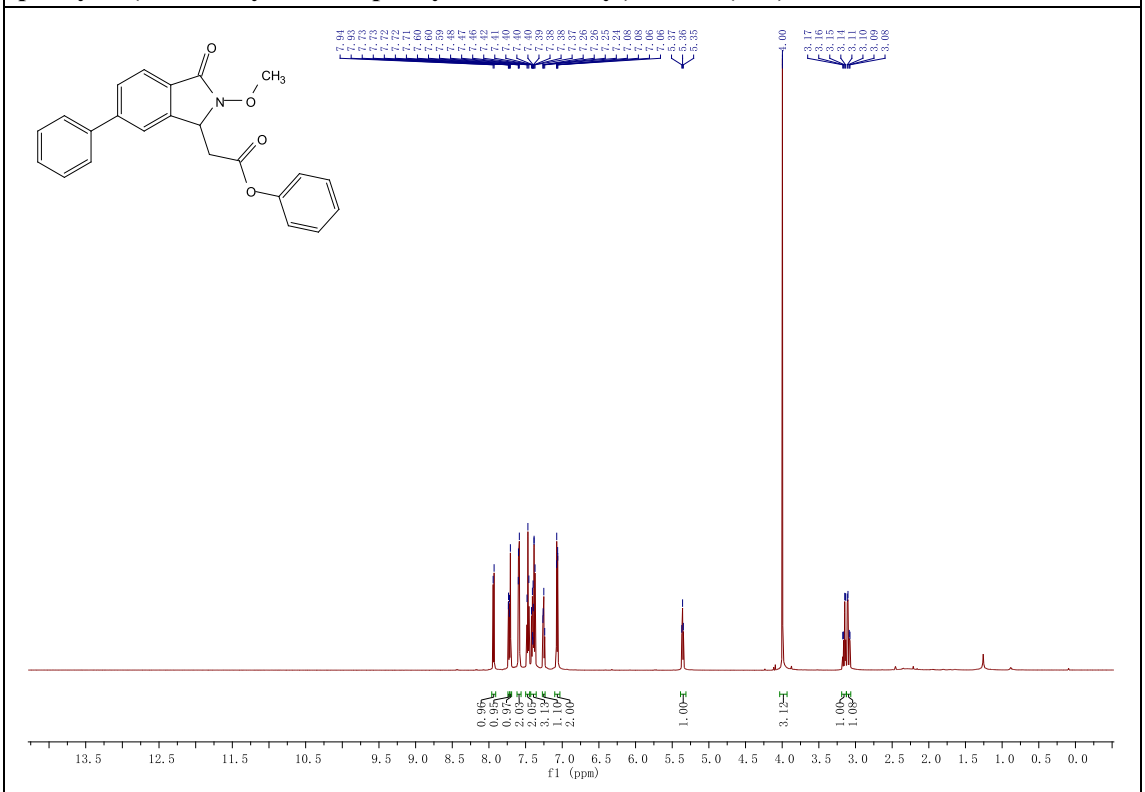


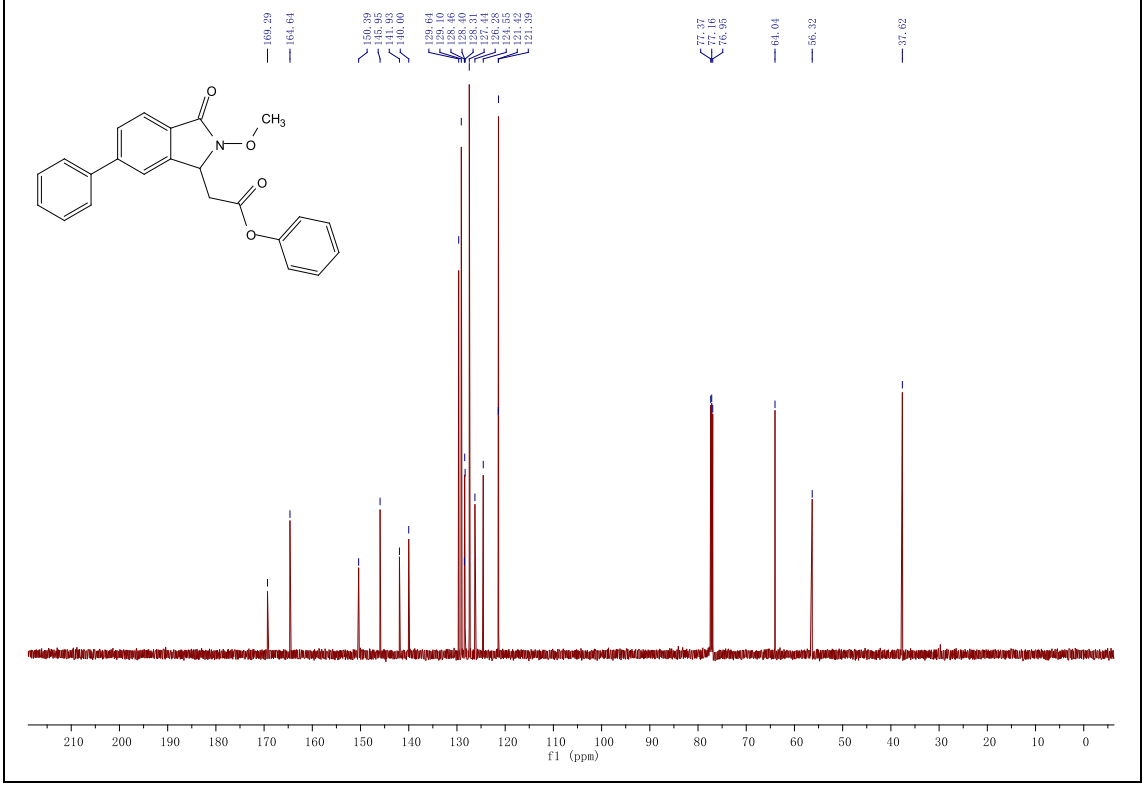
phenyl 2-(6-cyano-2-methoxy-3-oxoisoindolin-1-yl)acetate (3at)



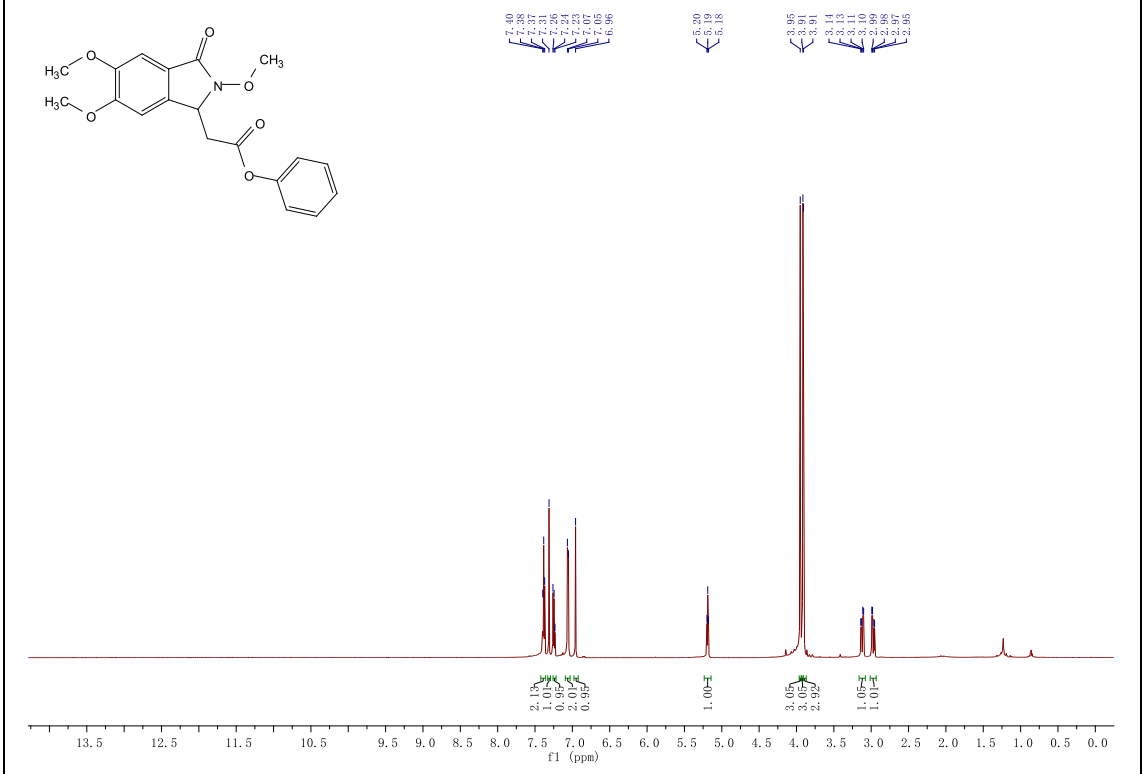


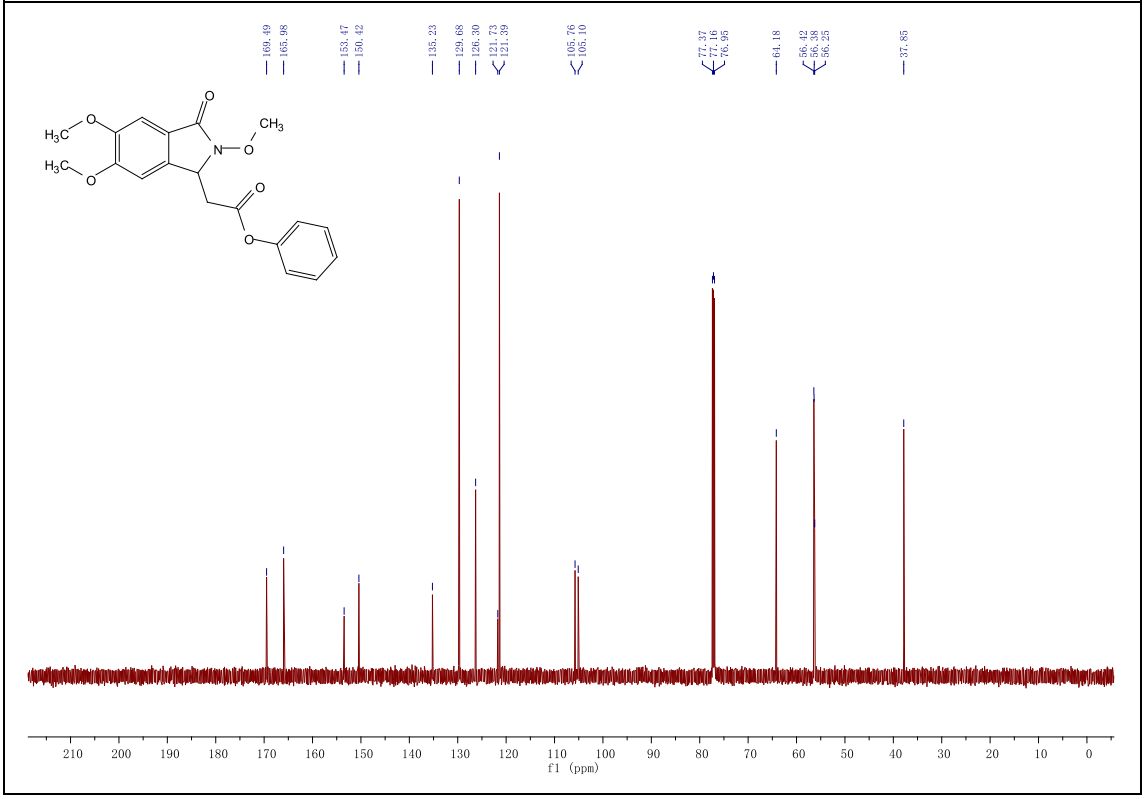
phenyl 2-(2-methoxy-3-oxo-6-phenylisoindolin-1-yl)acetate (3au)



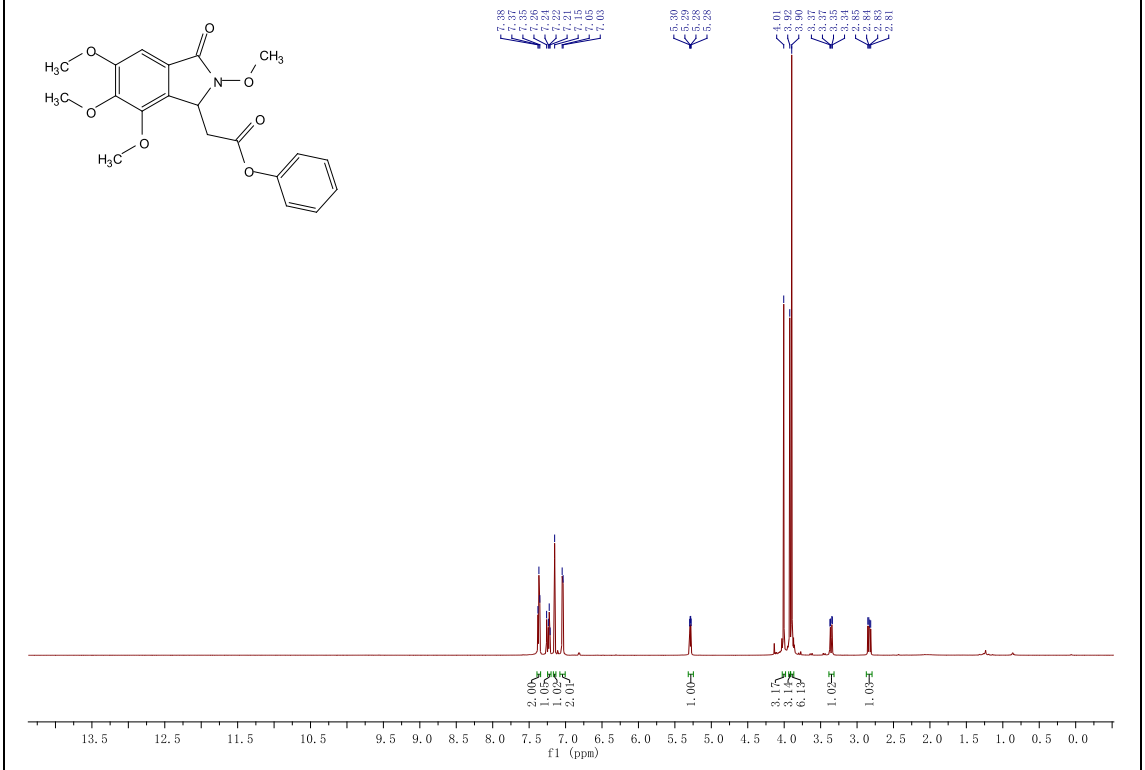


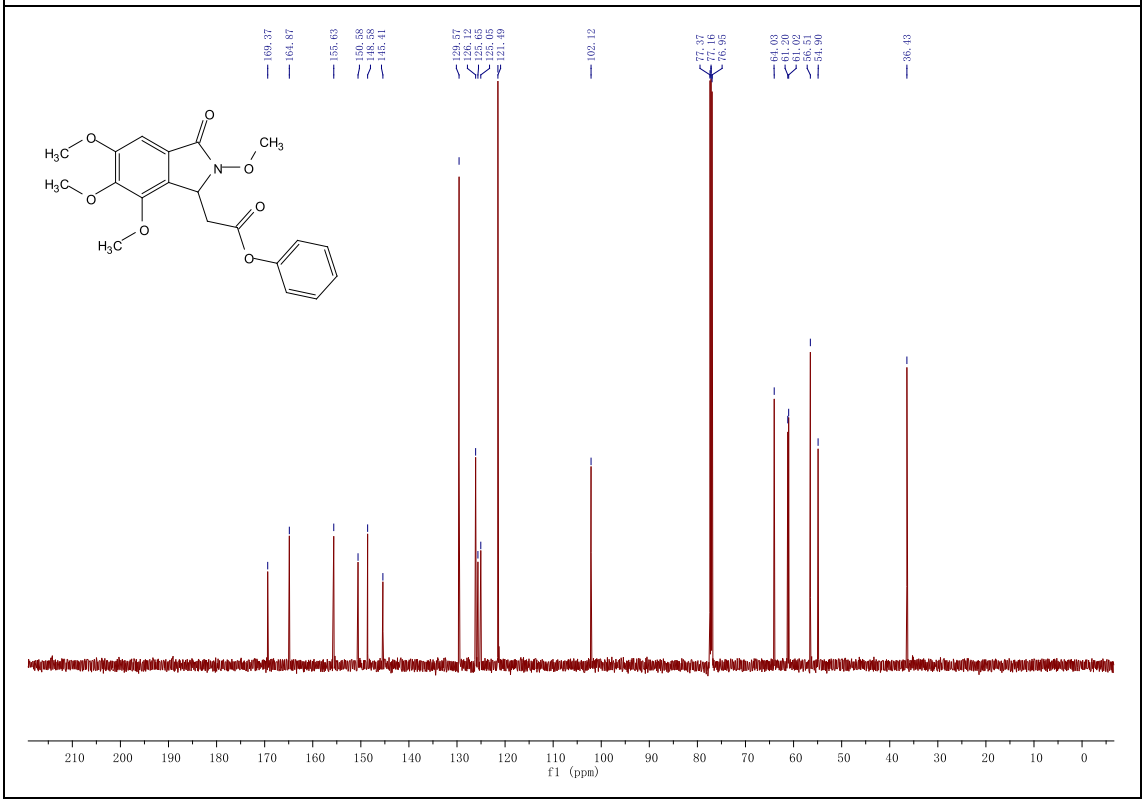
phenyl 2-(2,5,6-trimethoxy-3-oxoisoindolin-1-yl)acetate (3av)



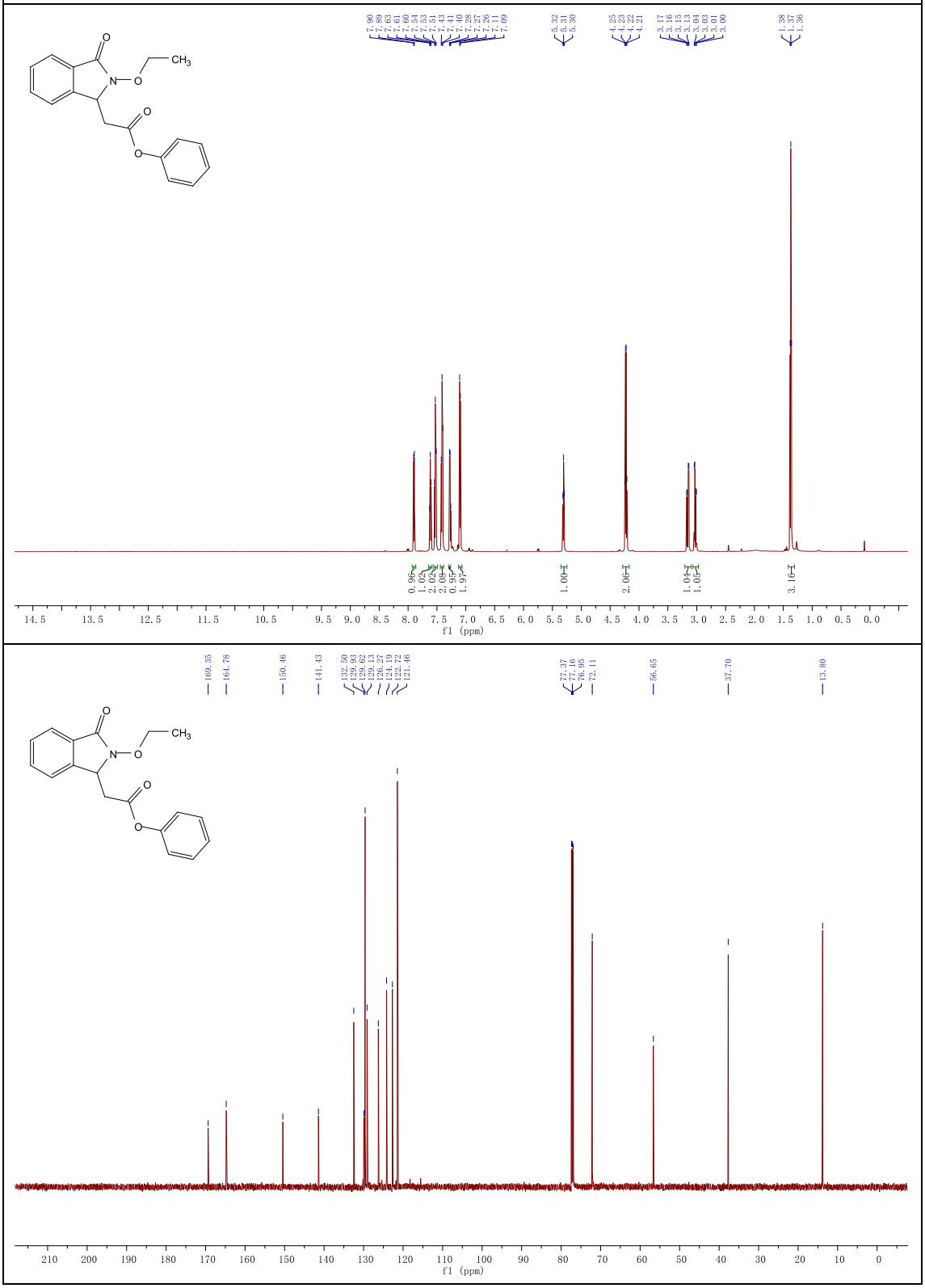


phenyl 2-(2,5,6,7-tetramethoxy-3-oxoisoindolin-1-yl)acetate (3aw)

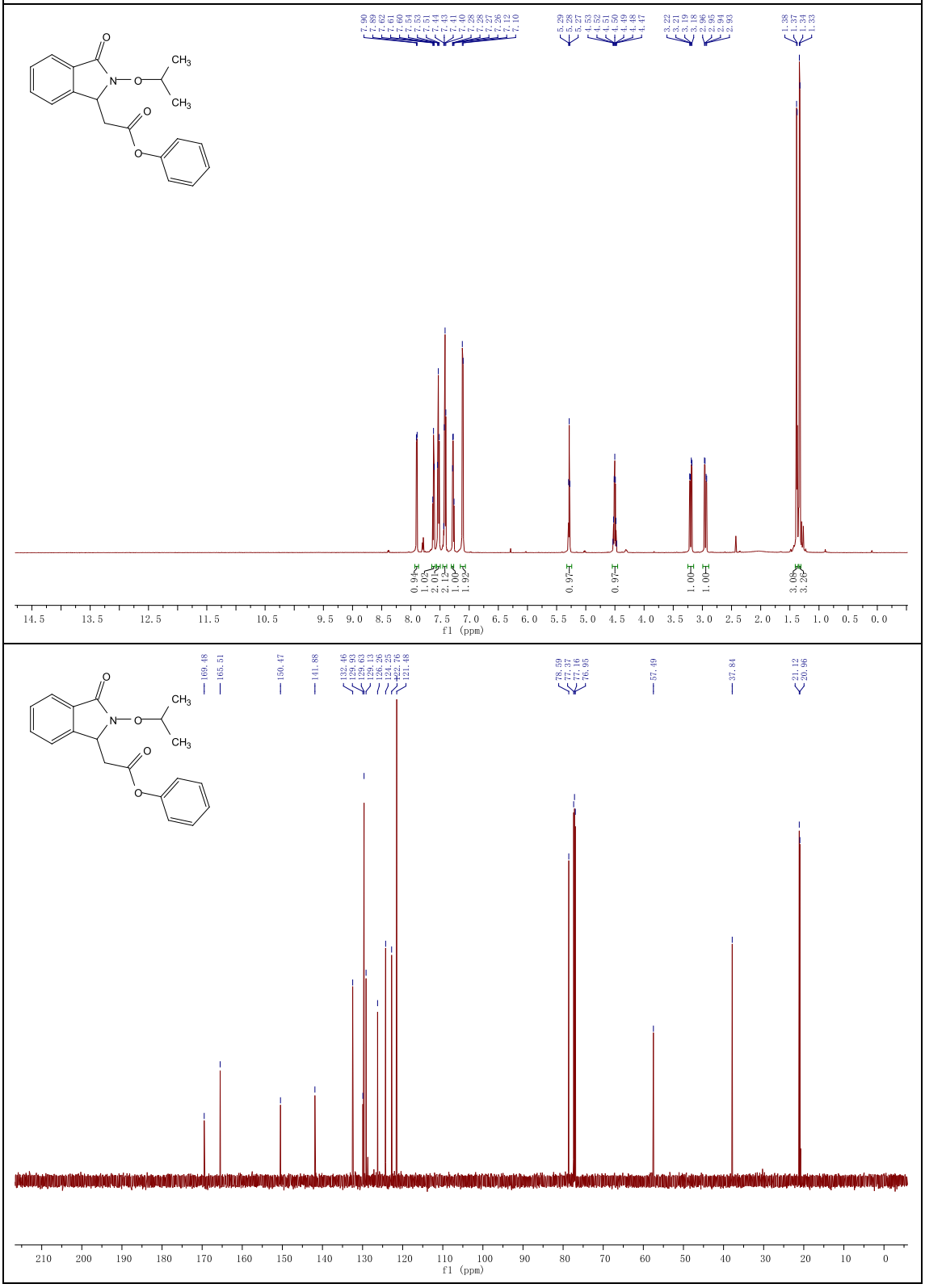




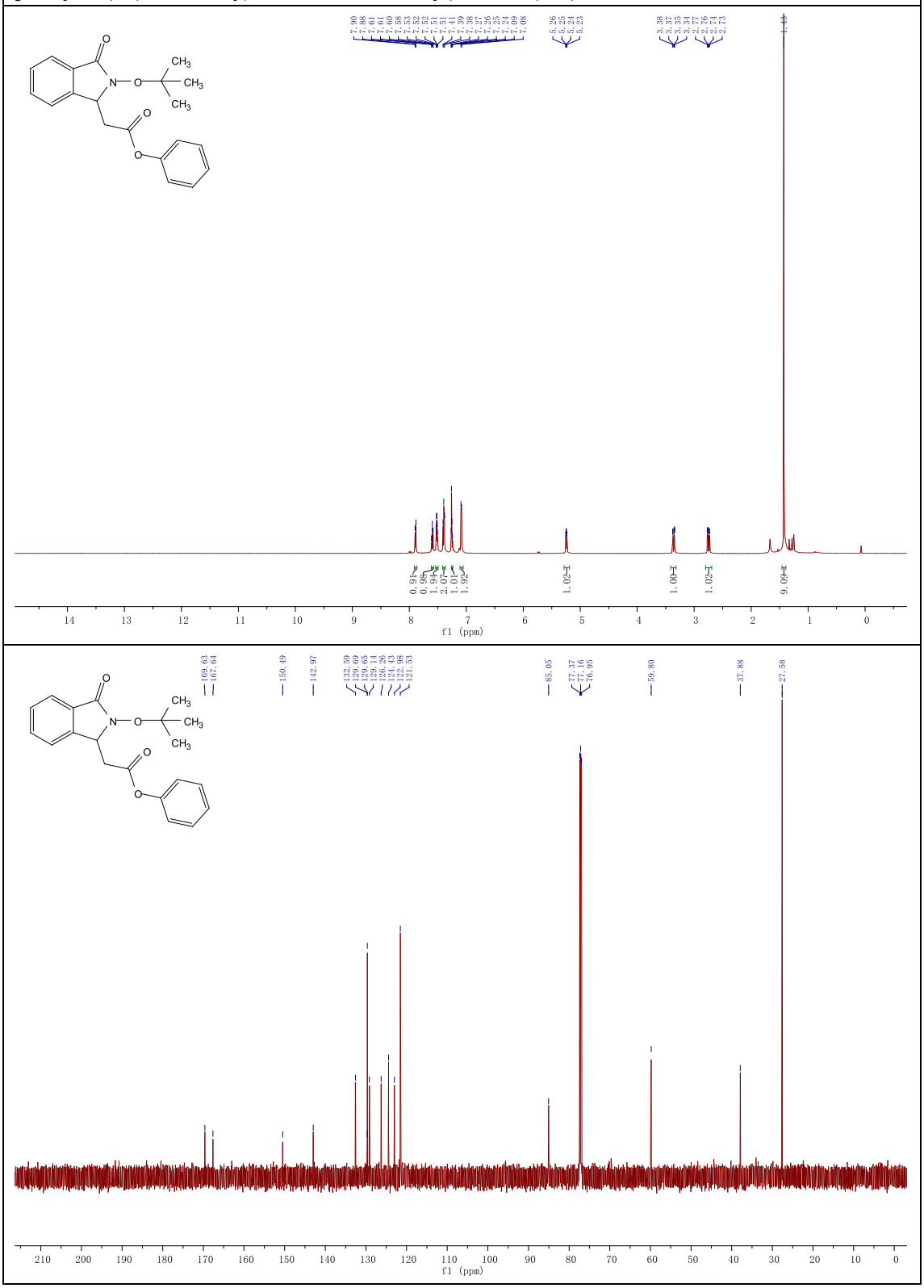
phenyl 2-(2-ethoxy-3-oxoisoindolin-1-yl)acetate (3ax)



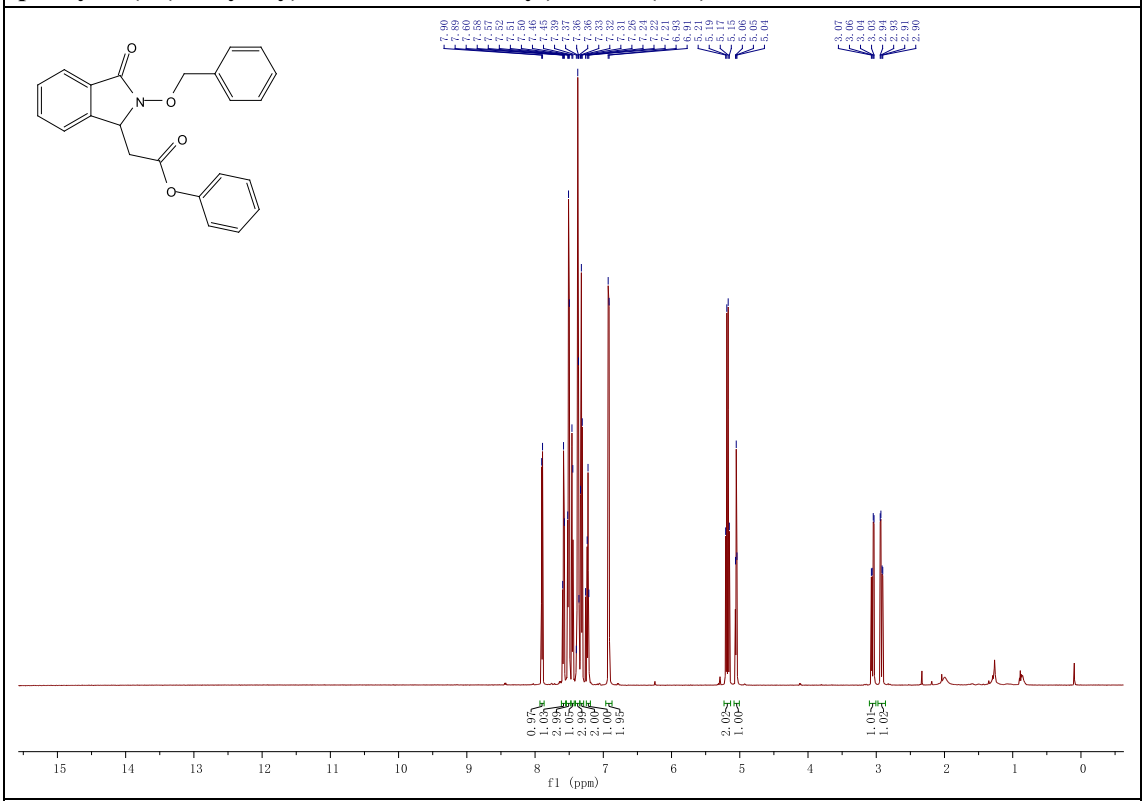
phenyl 2-(2-isopropoxy-3-oxoisoindolin-1-yl)acetate (3ay)

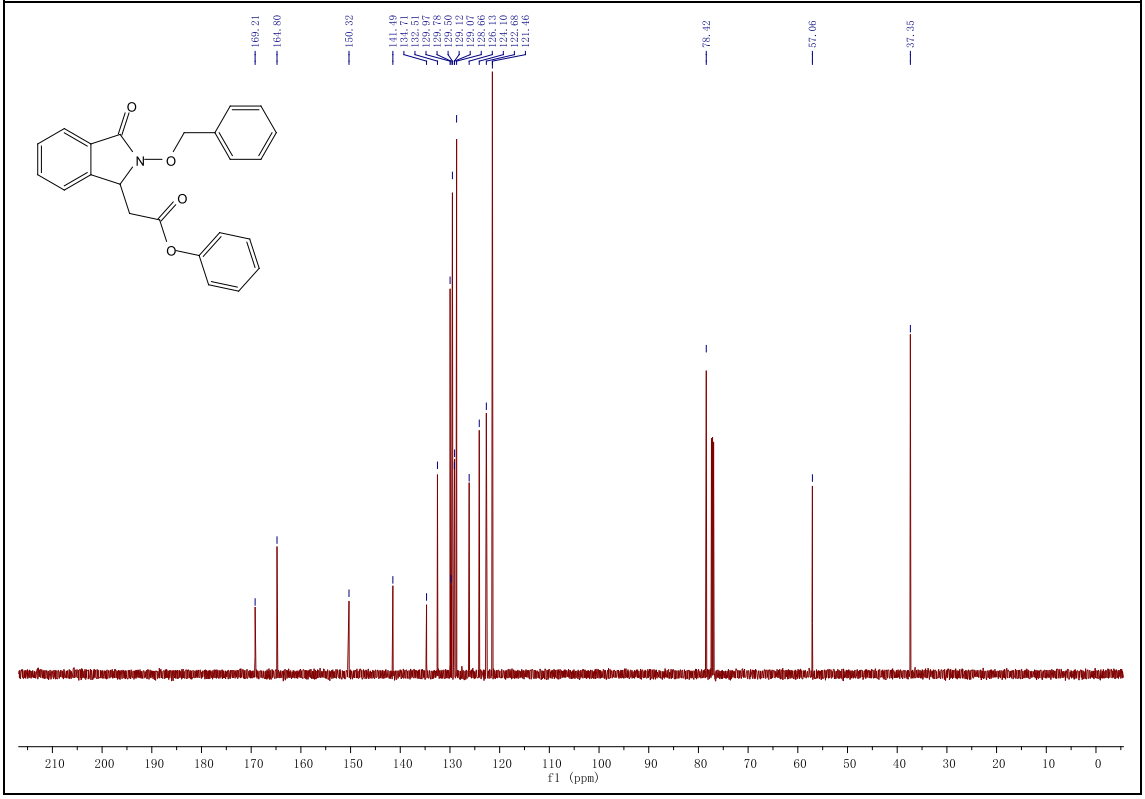


phenyl 2-(2-(*tert*-butoxy)-3-oxoisoindolin-1-yl)acetate (3az)

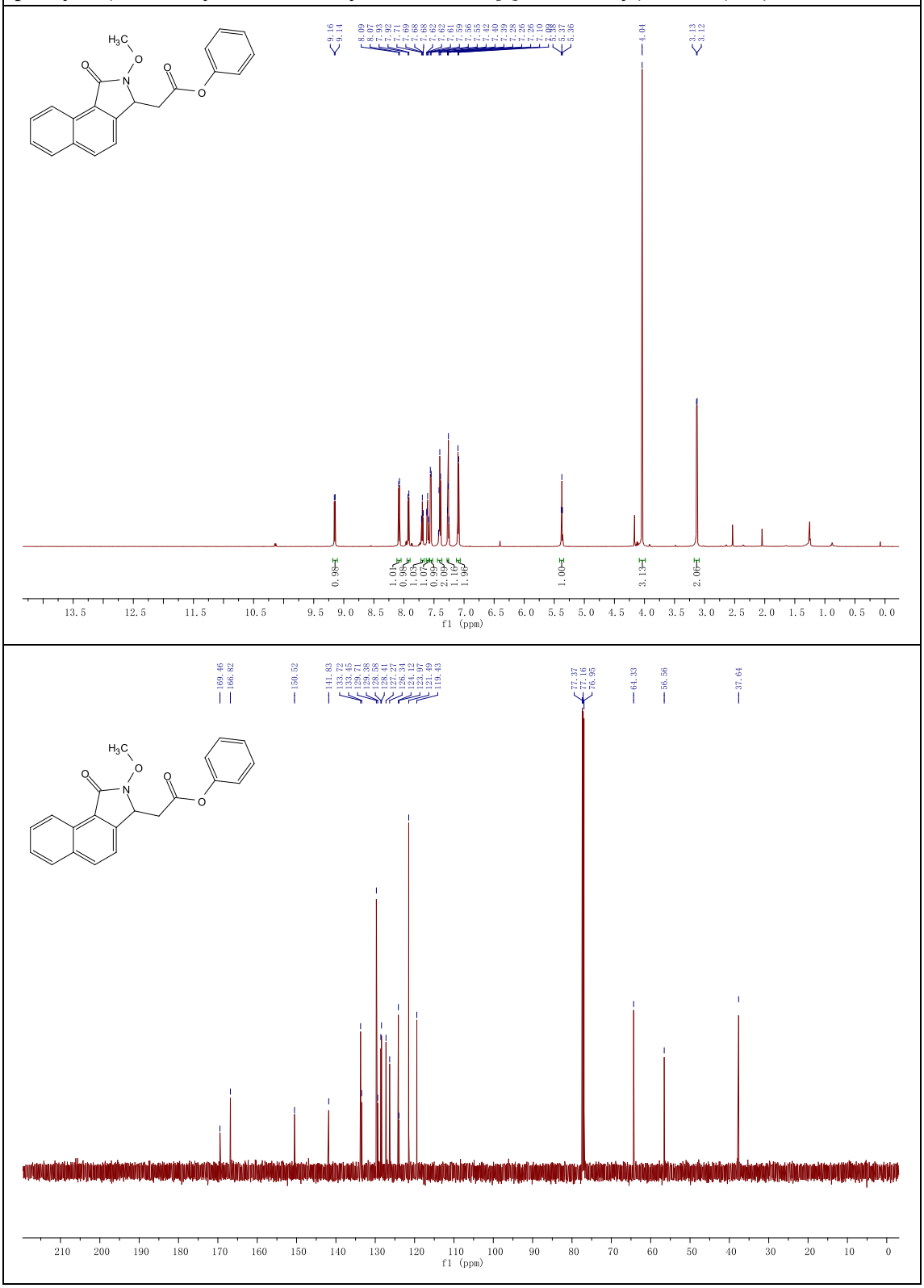


phenyl 2-(2-(benzyloxy)-3-oxoisoindolin-1-yl)acetate (3ba)

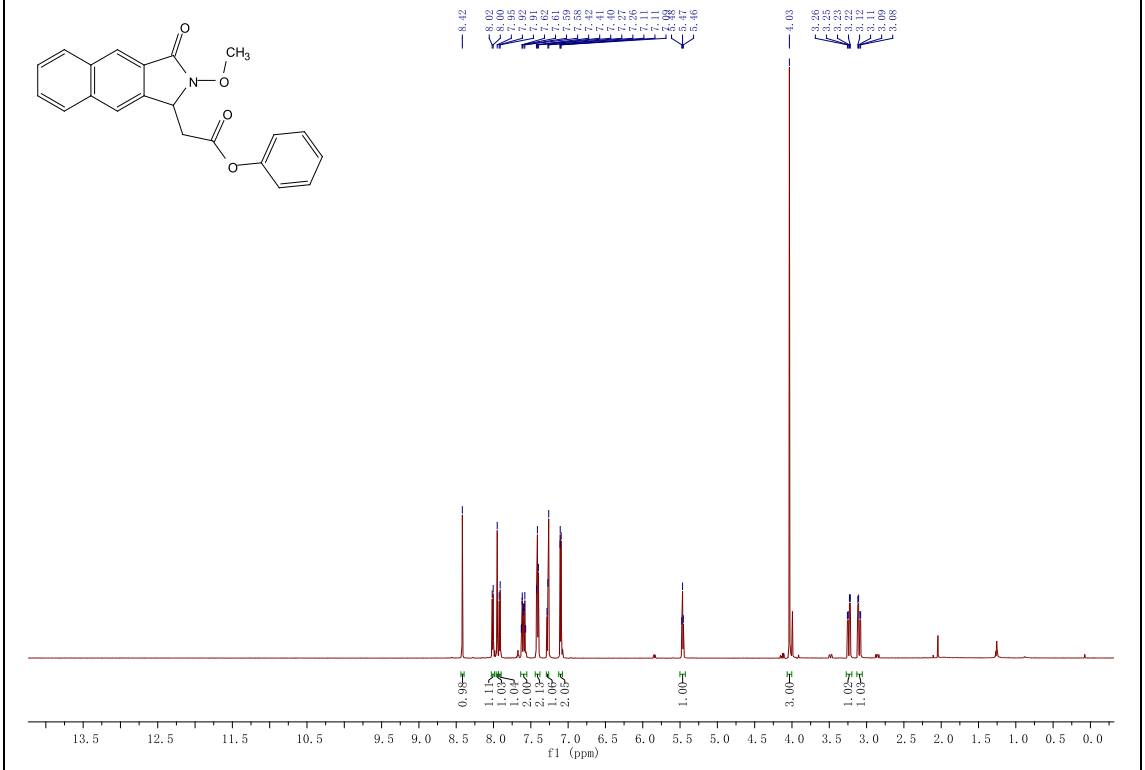


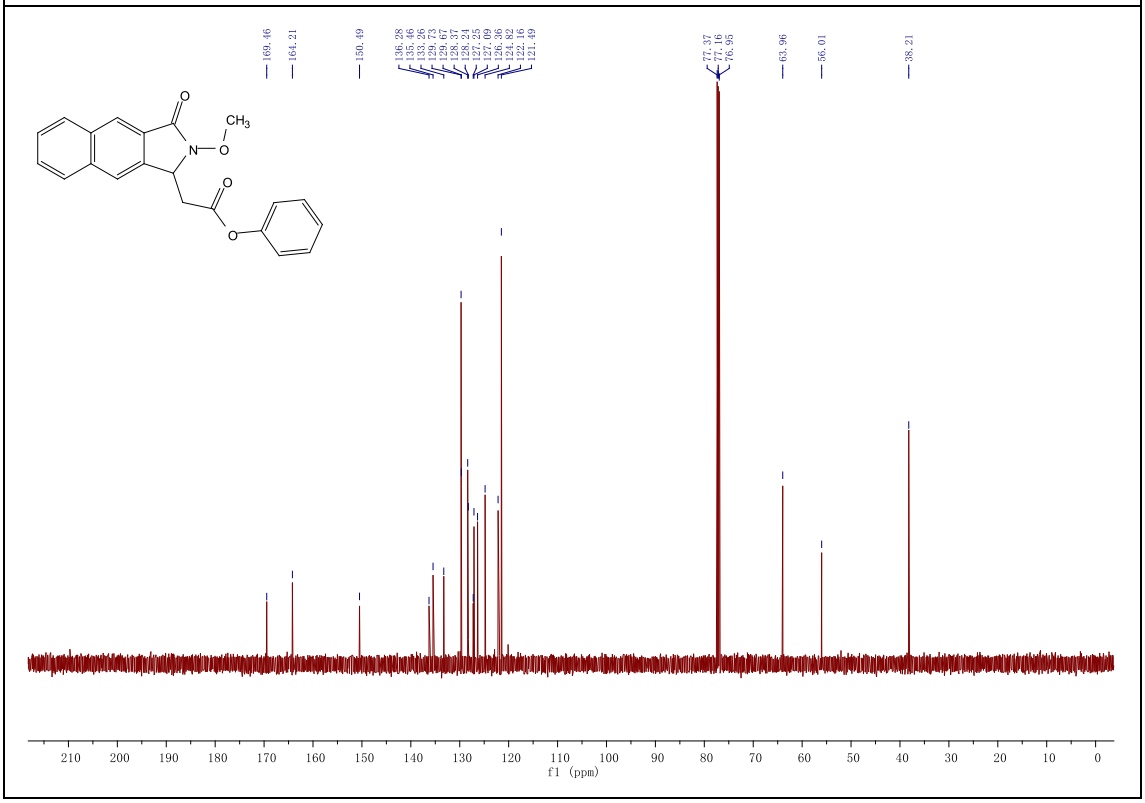


phenyl 2-(2-methoxy-1-oxo-2,3-dihydro-1*H*-benzo[*e*]isoindol-3-yl)acetate (3bb)

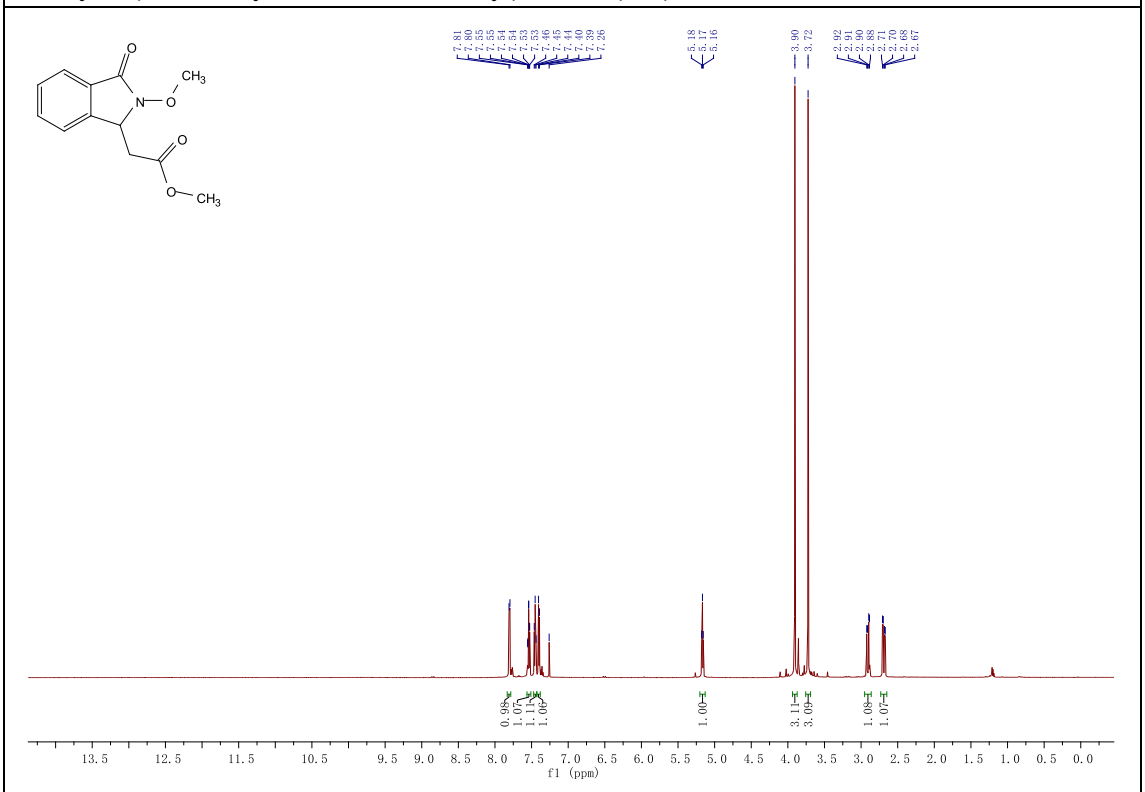


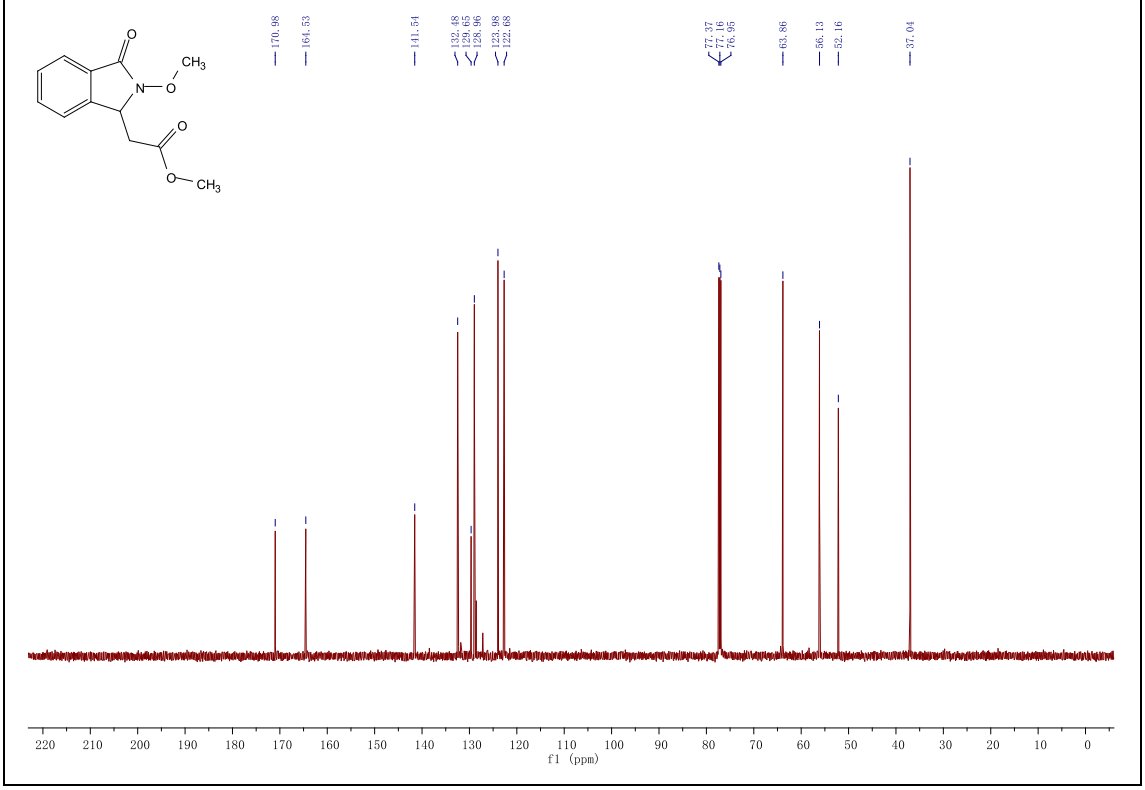
phenyl 2-(2-methoxy-3-oxo-2,3-dihydro-1*H*-benzo[*f*]isoindol-1-yl)acetate (3bc)



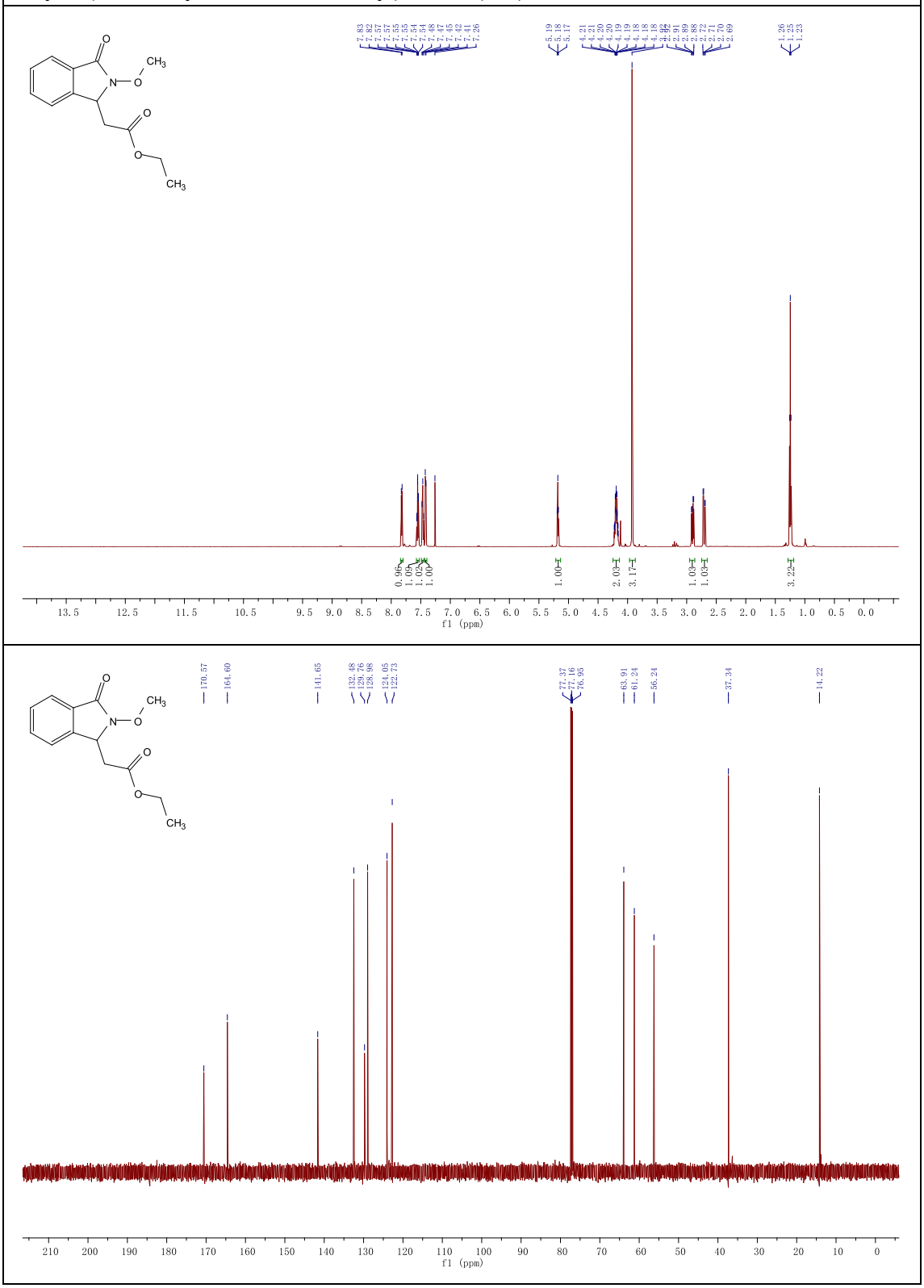


methyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4aa)

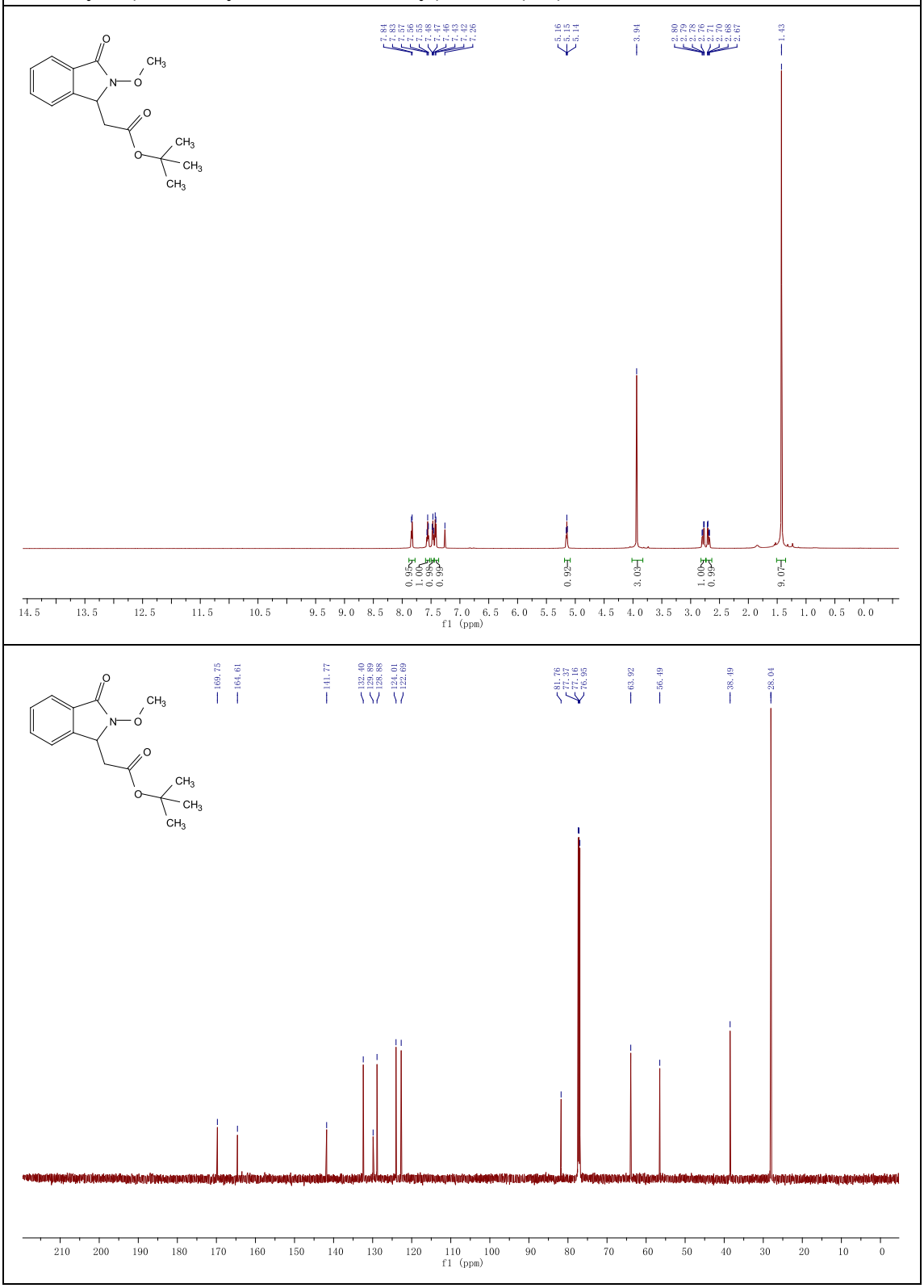




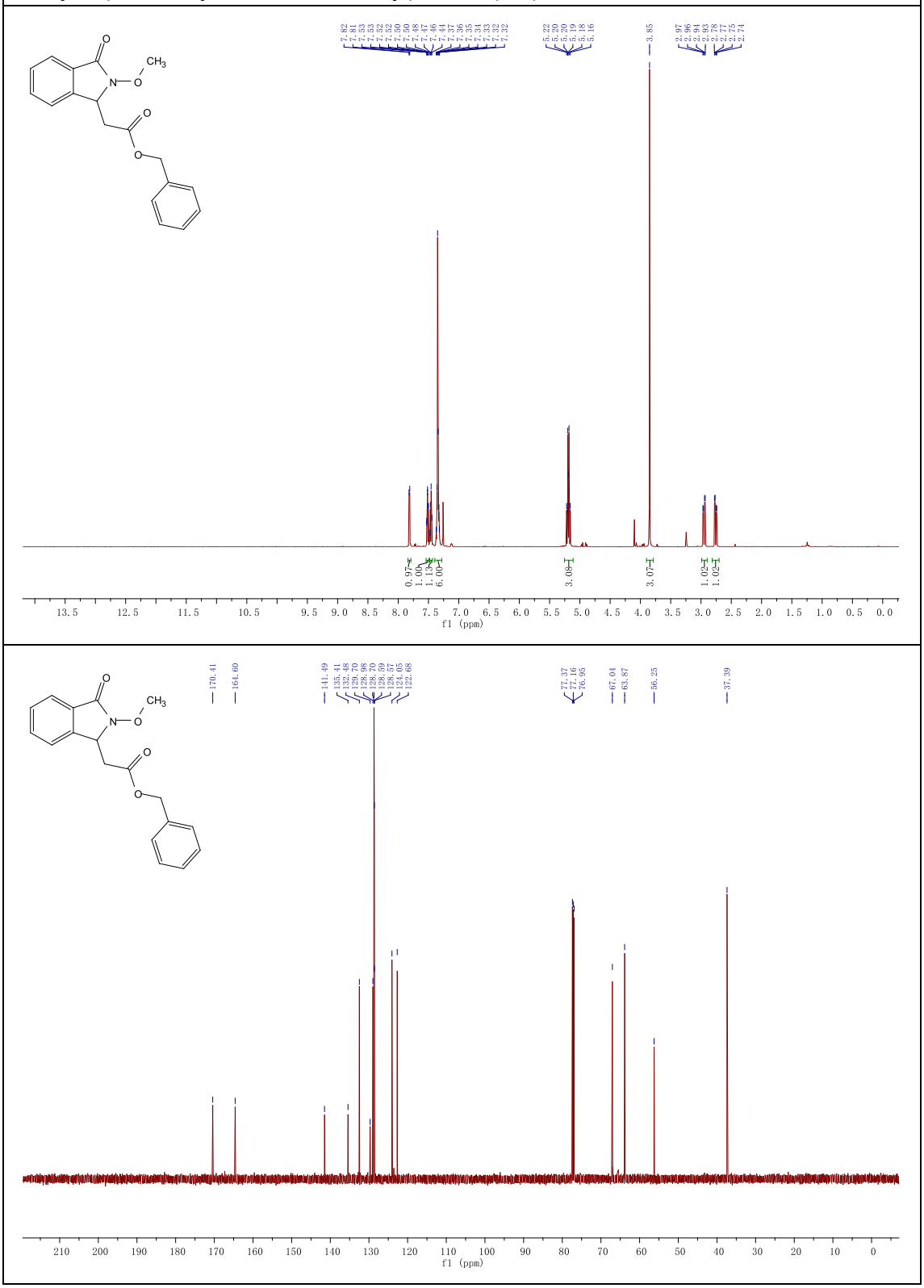
ethyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ab)



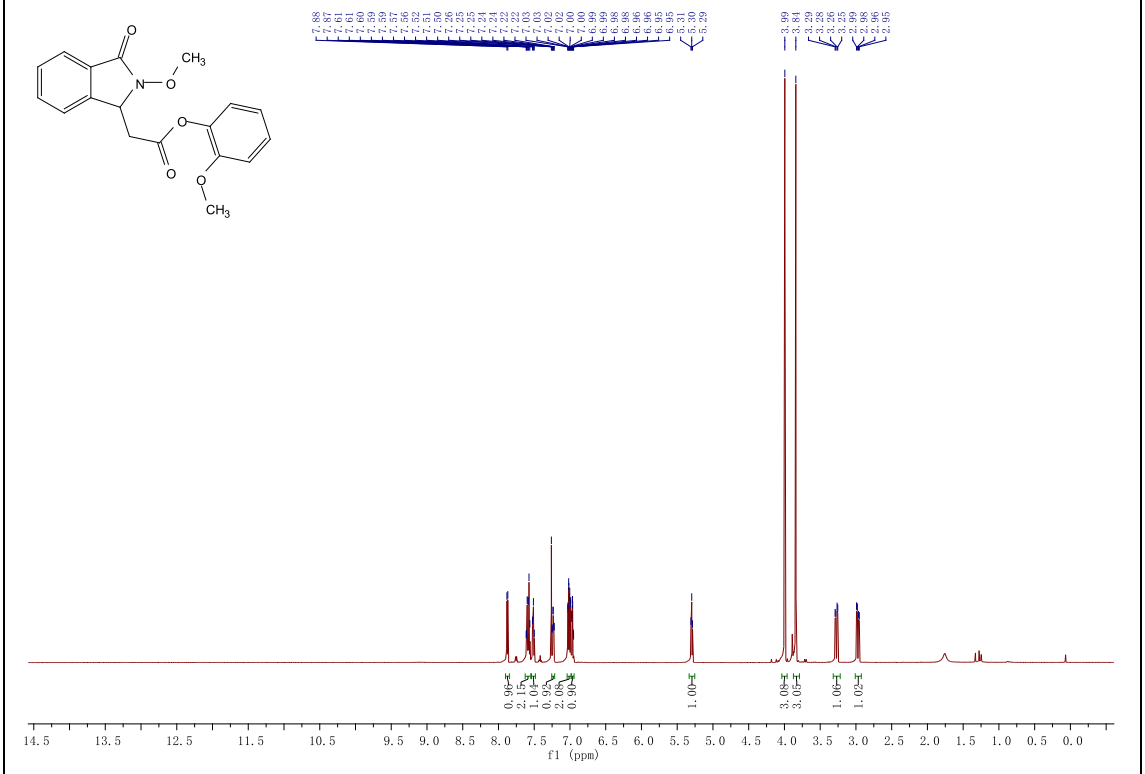
tert-butyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ac)

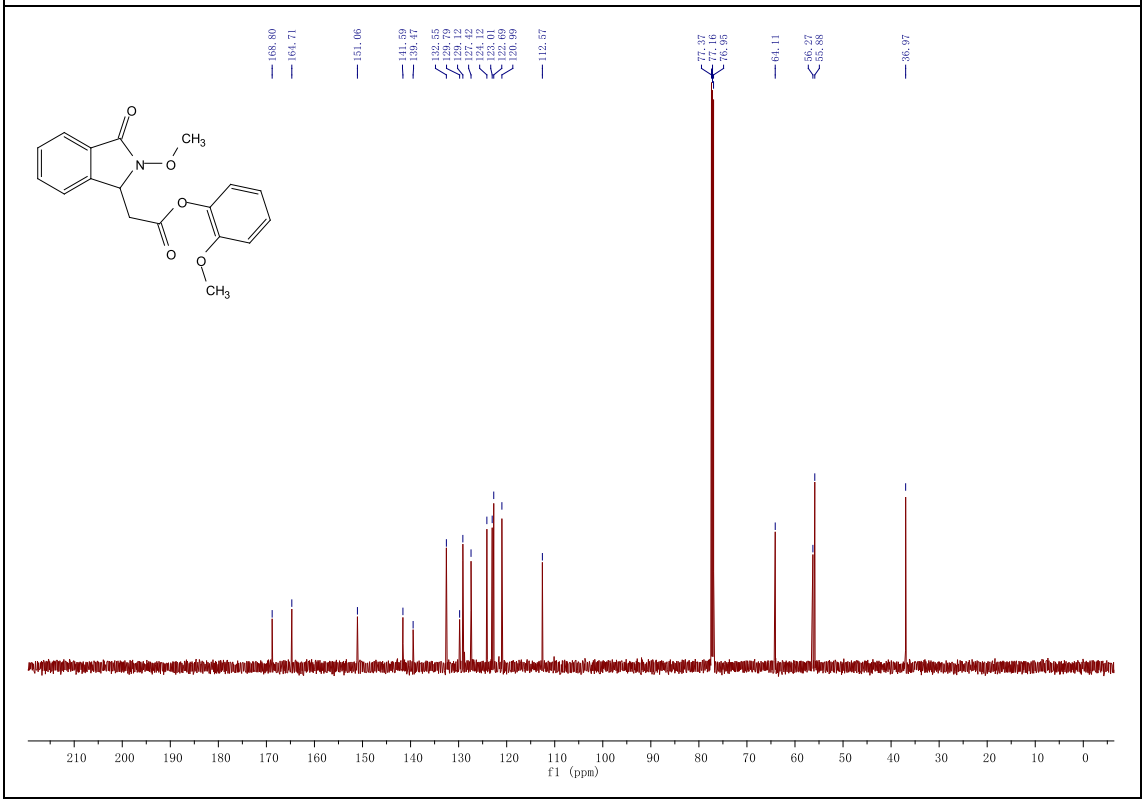


benzyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ad)

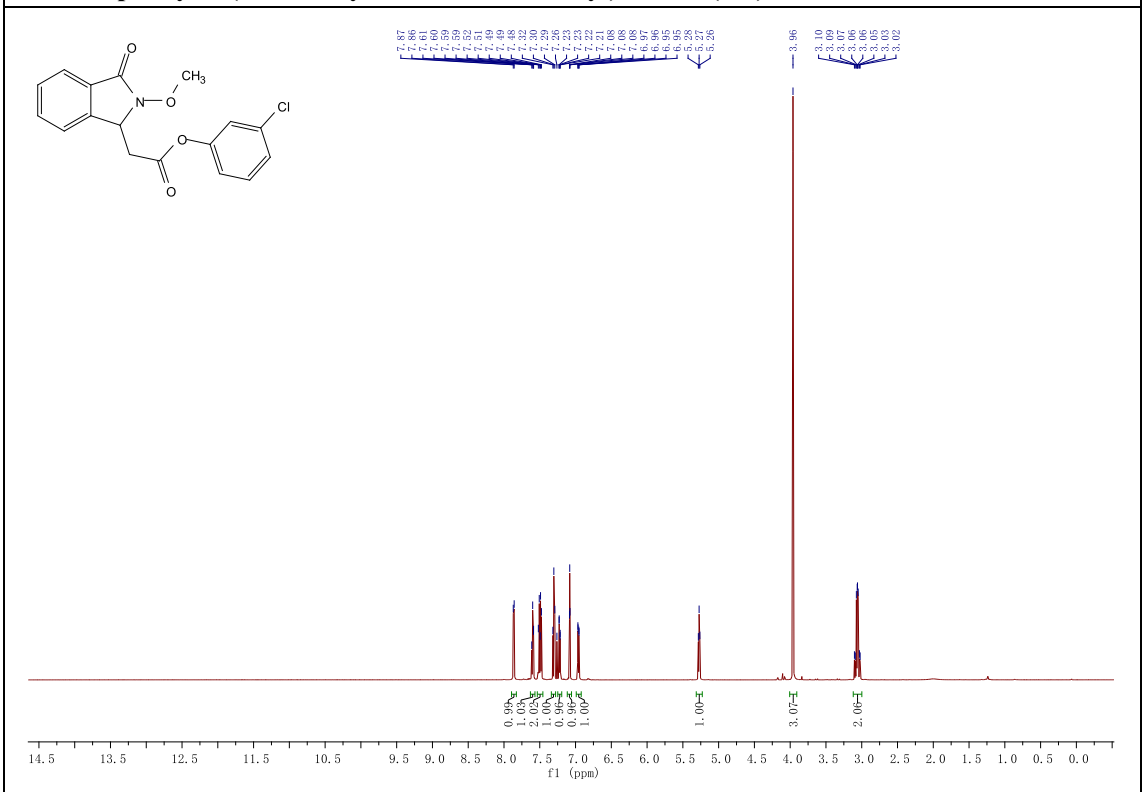


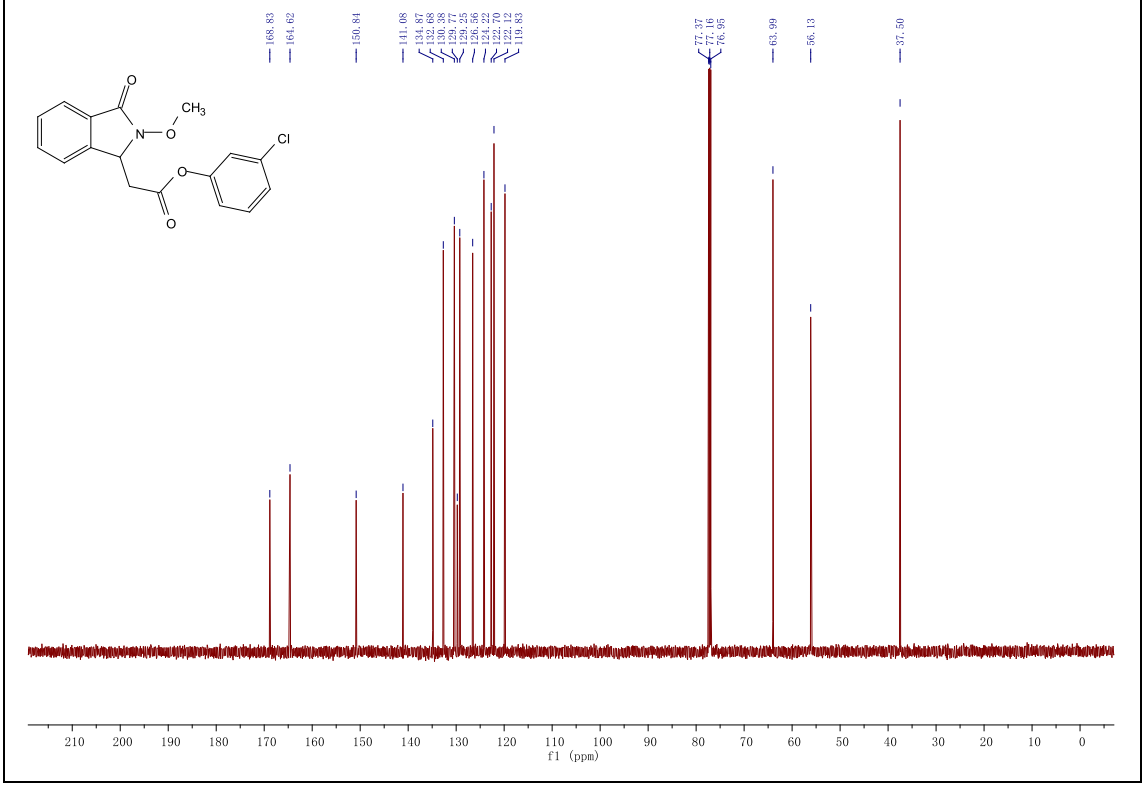
2-methoxyphenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ae)



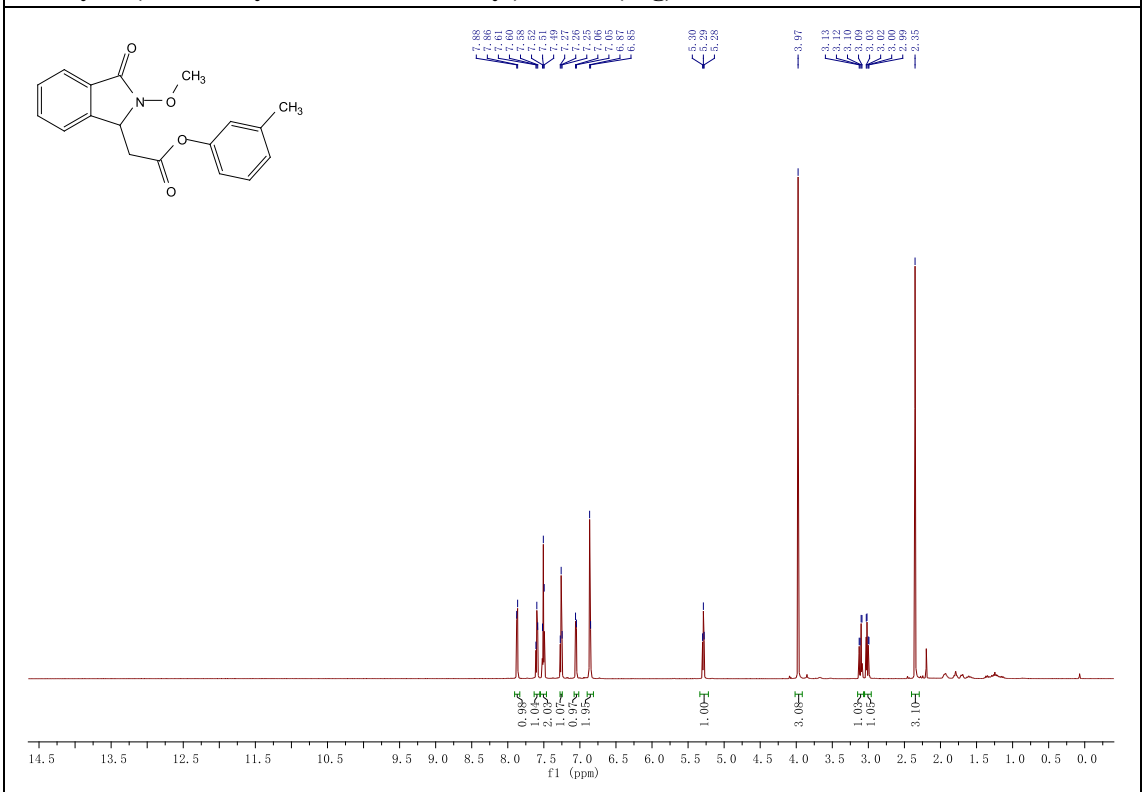


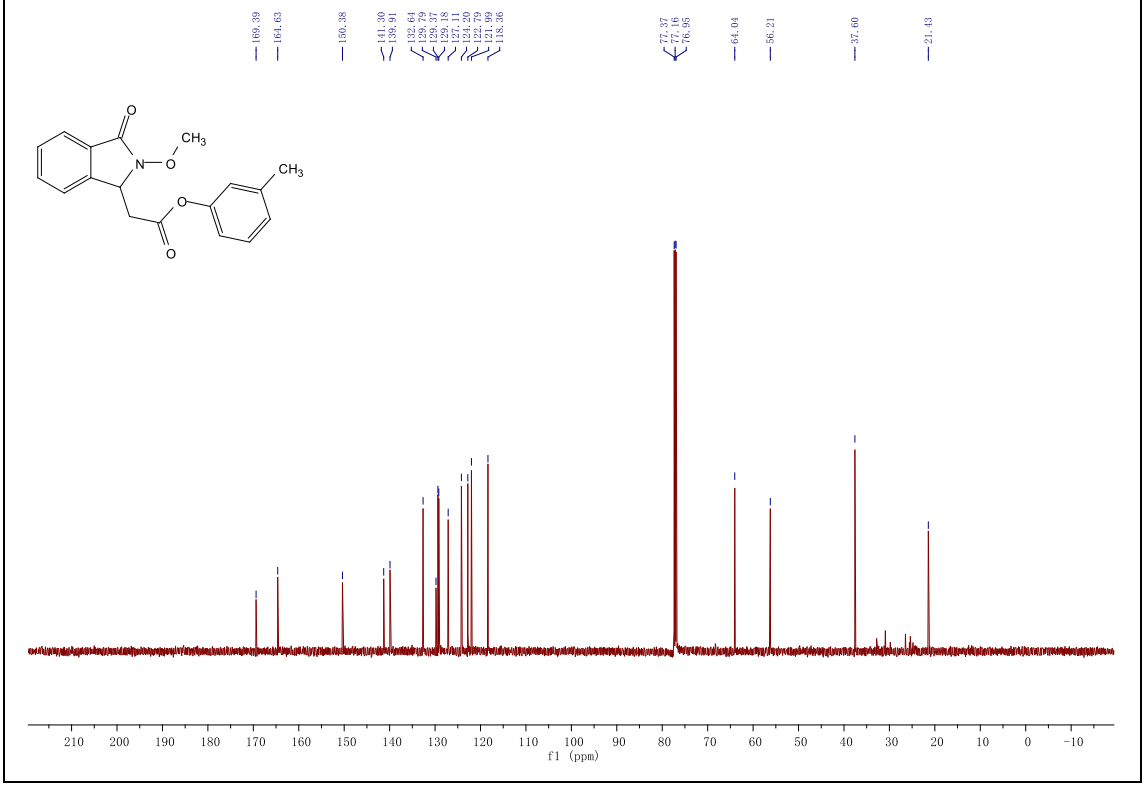
3-chlorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4af)



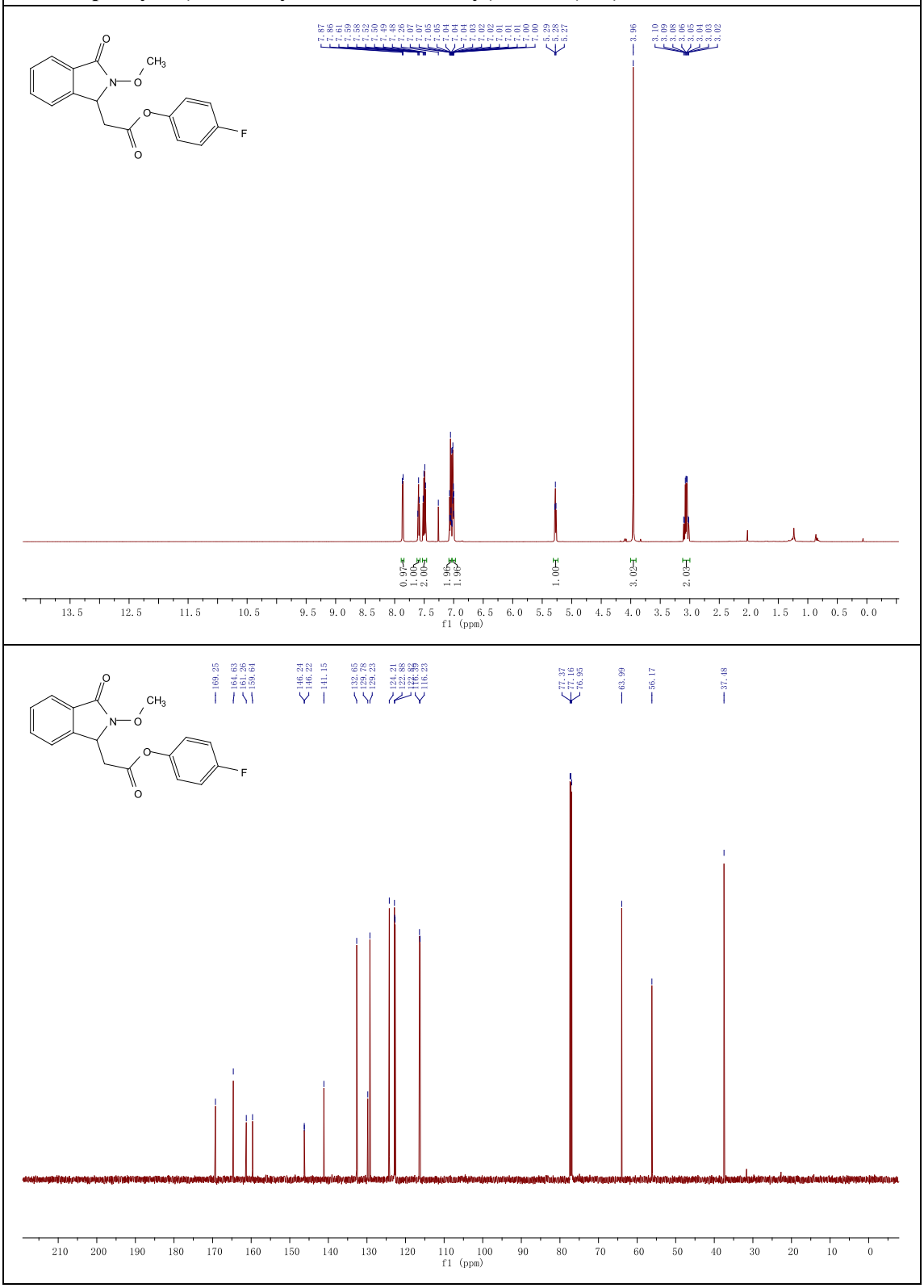


_m_-tolyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ag)

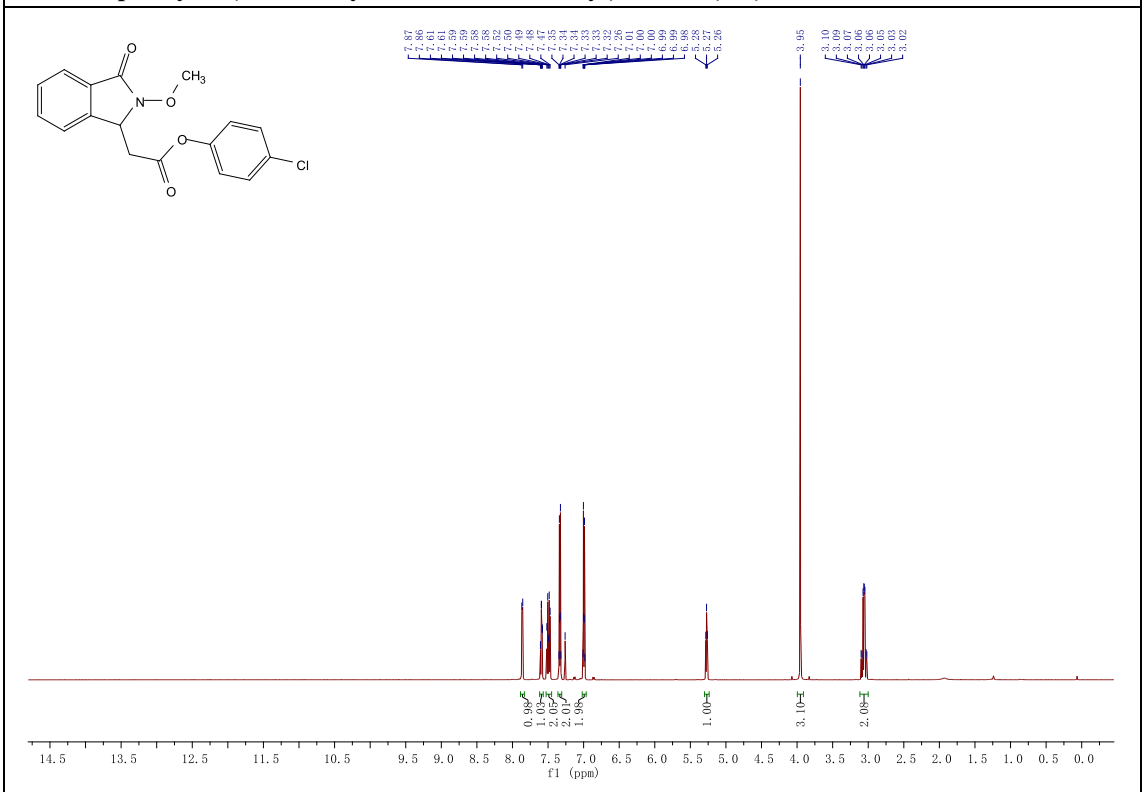


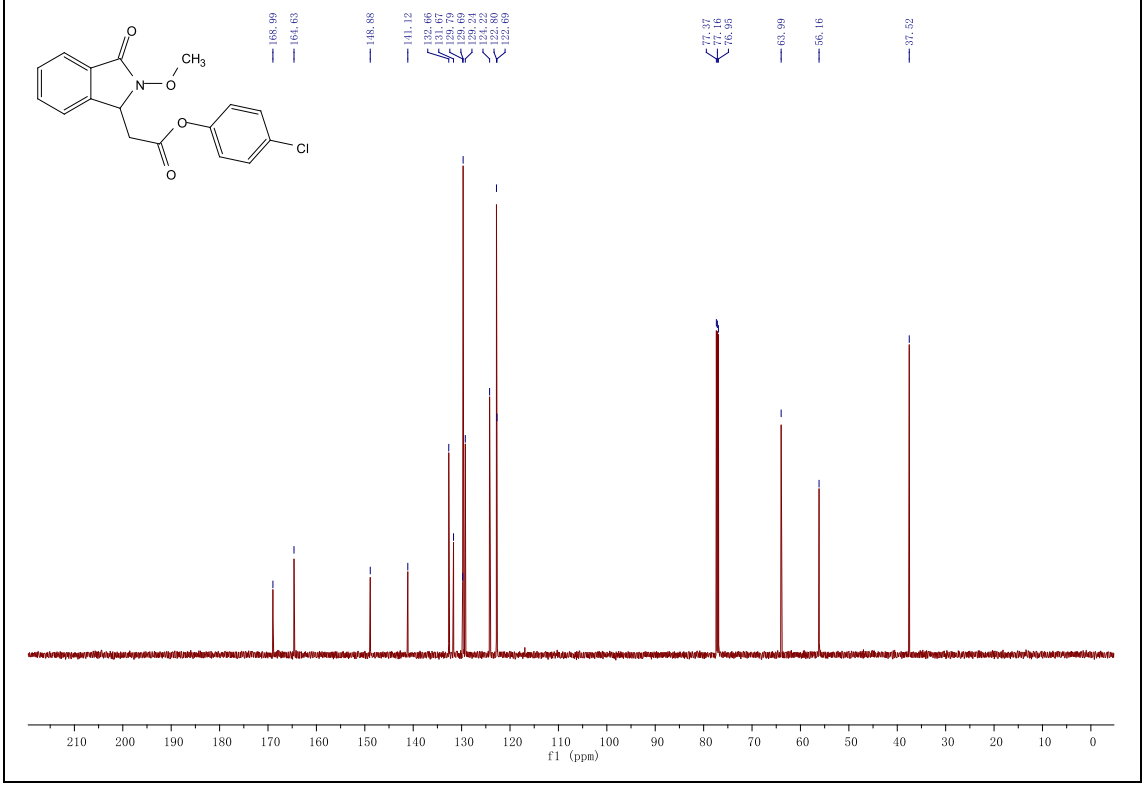


4-fluorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ah)

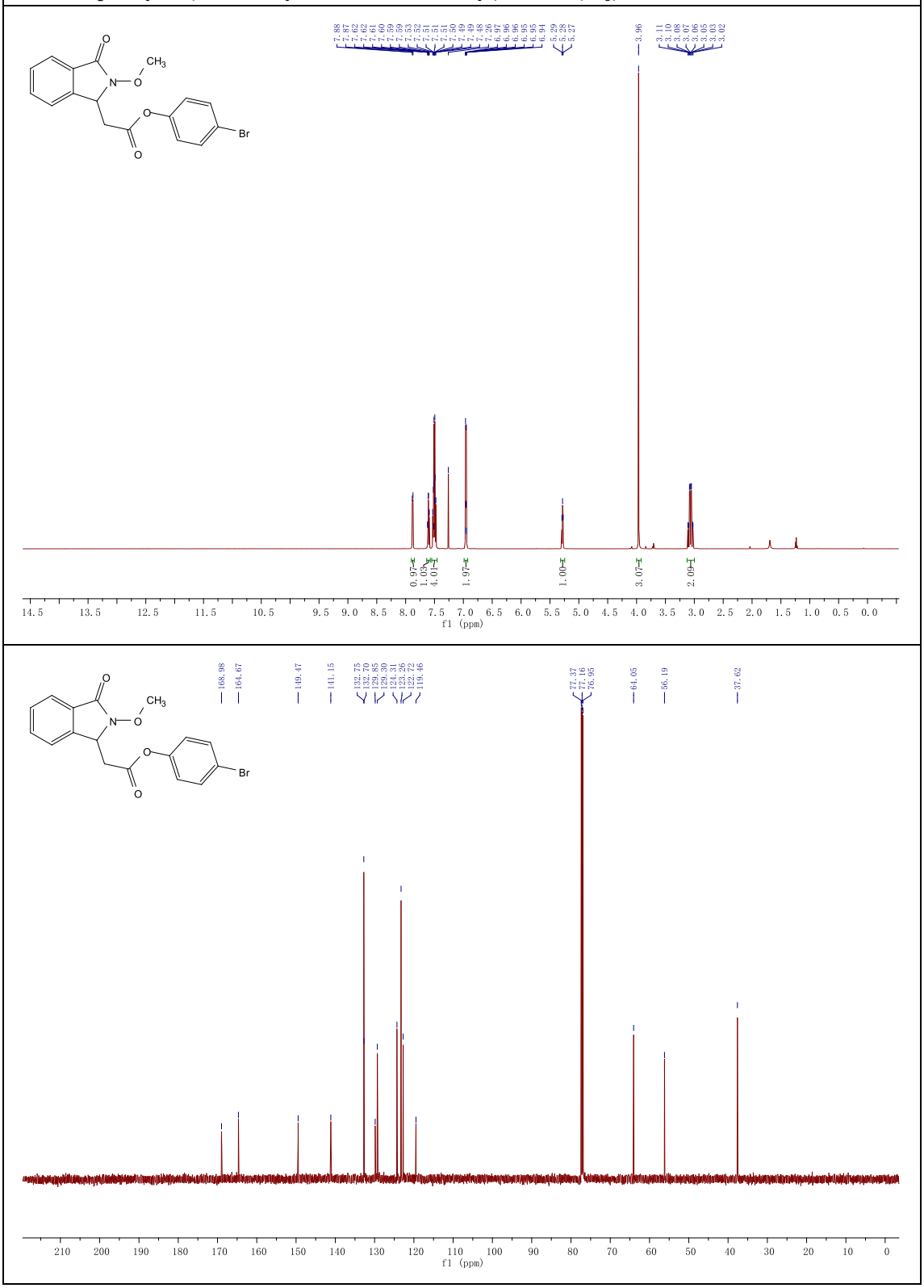


4-chlorophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ai)

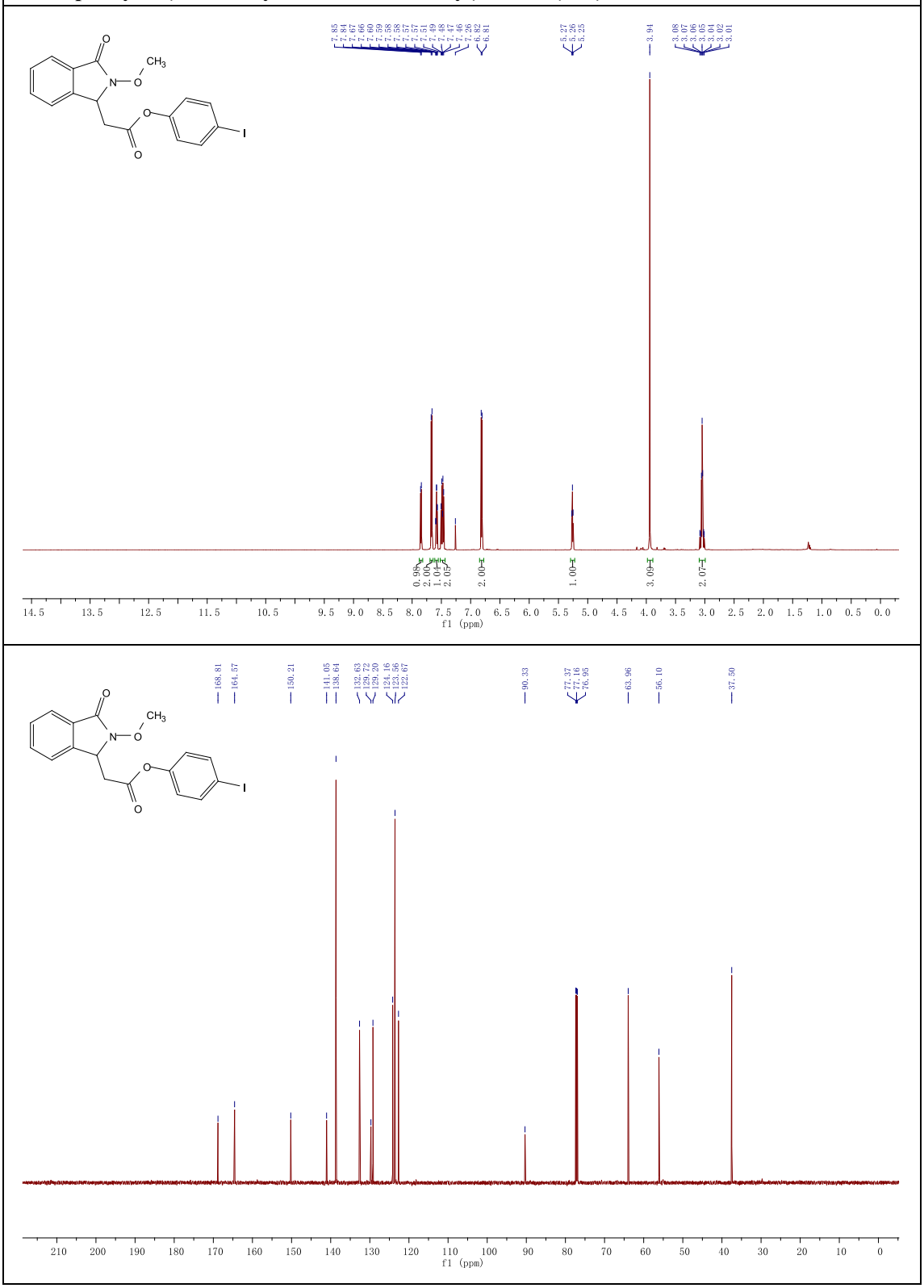




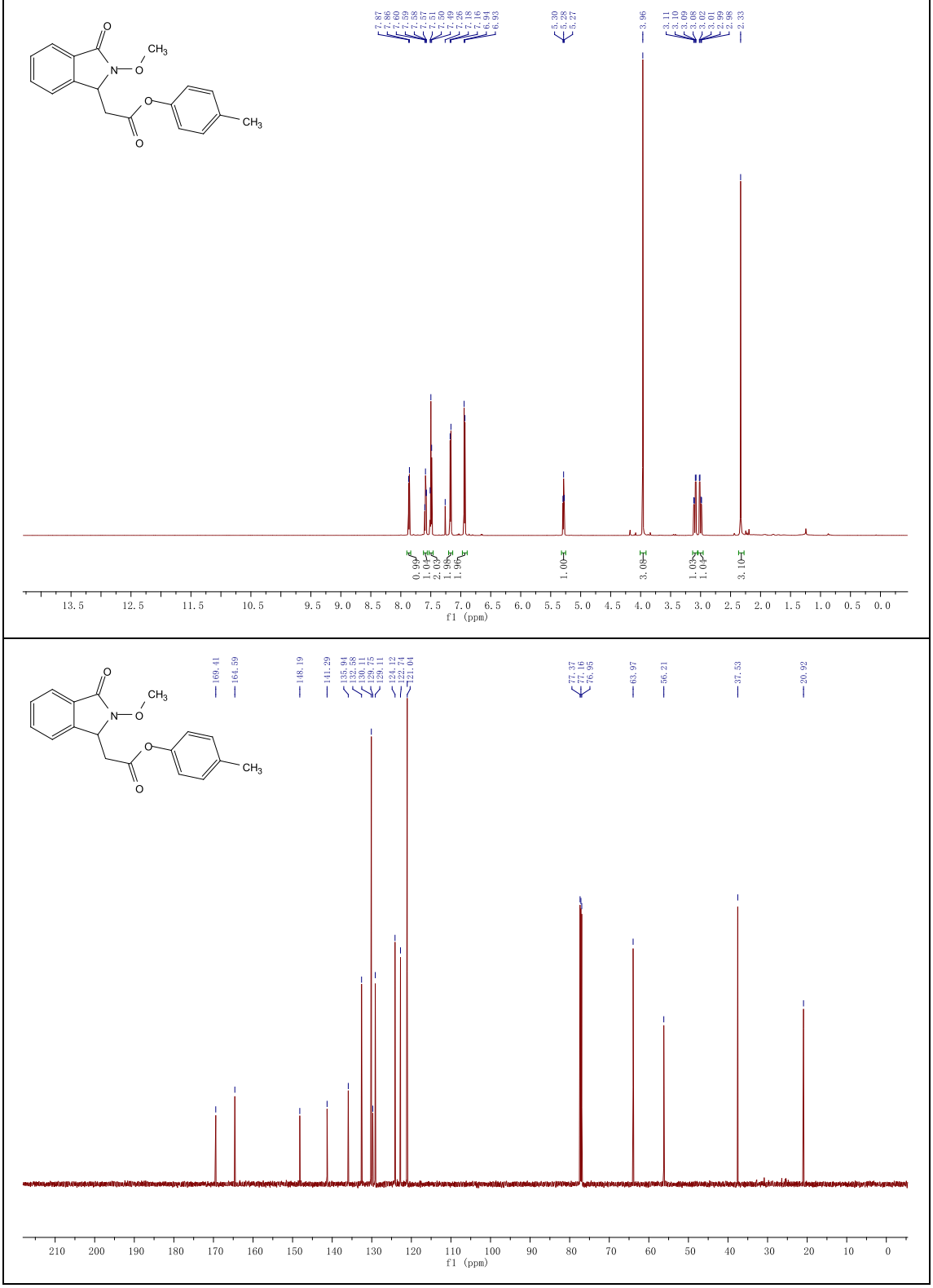
4-bromophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4aj)



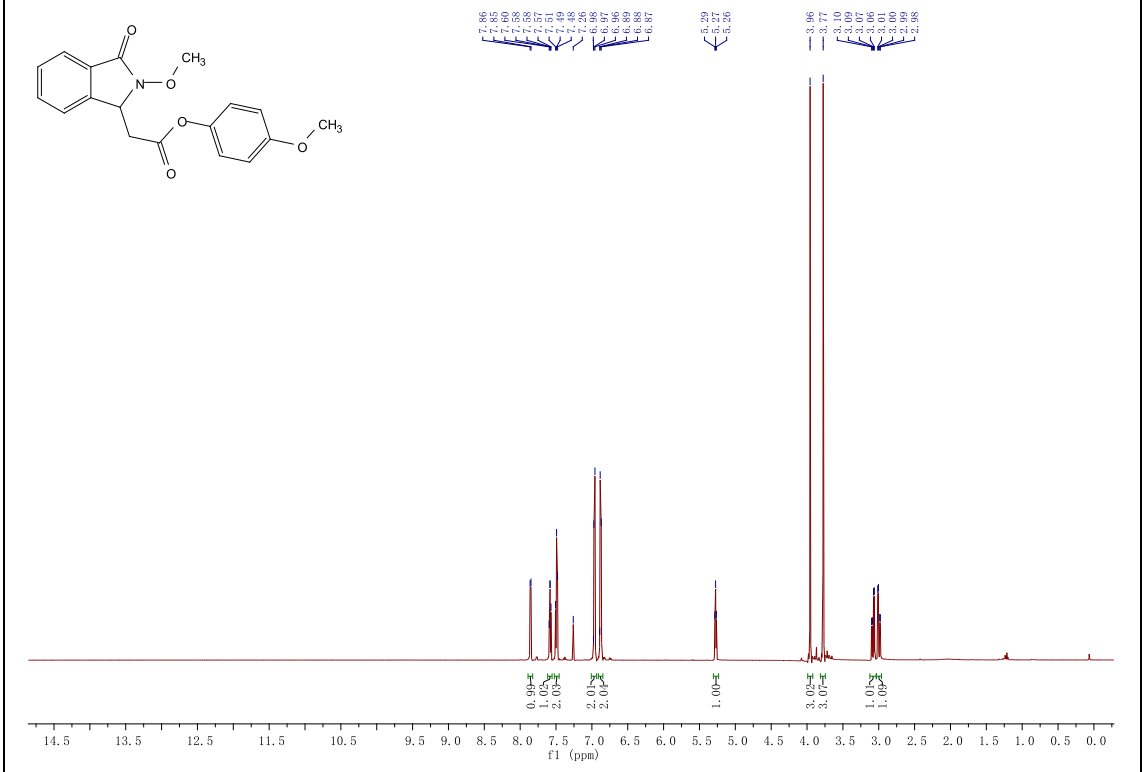
4-iodophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4ak)

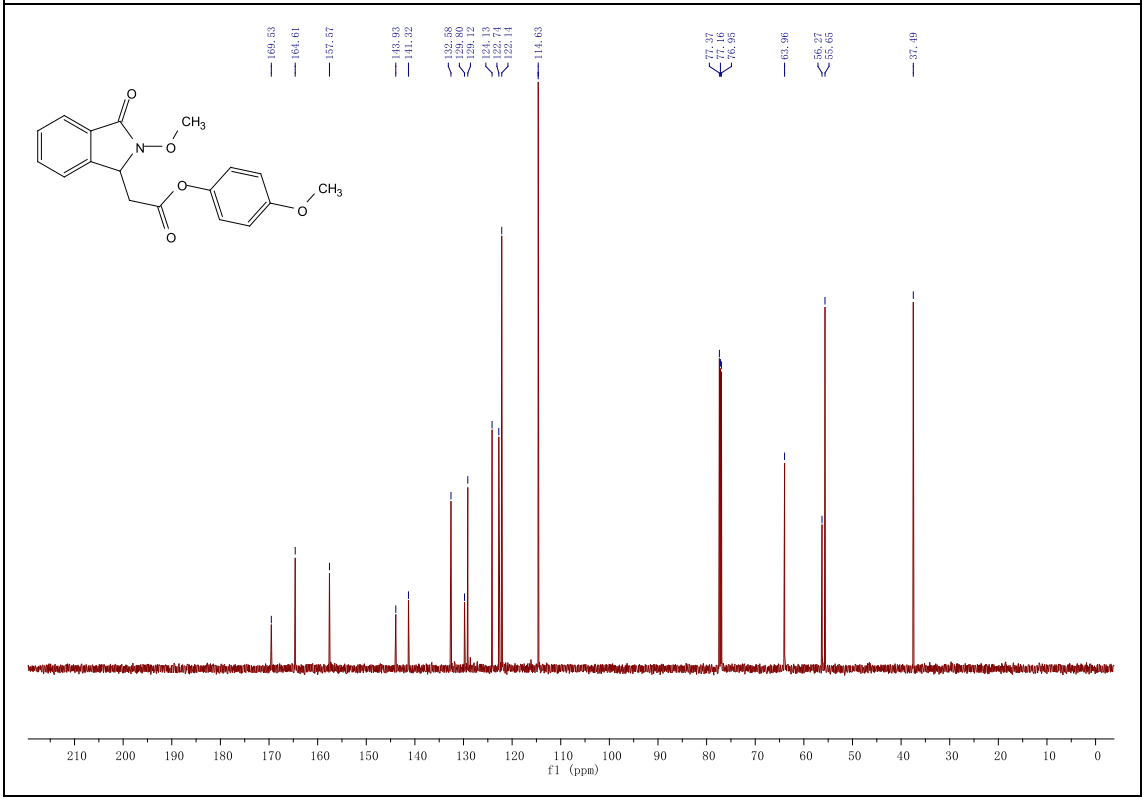


_p_-tolyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4al)

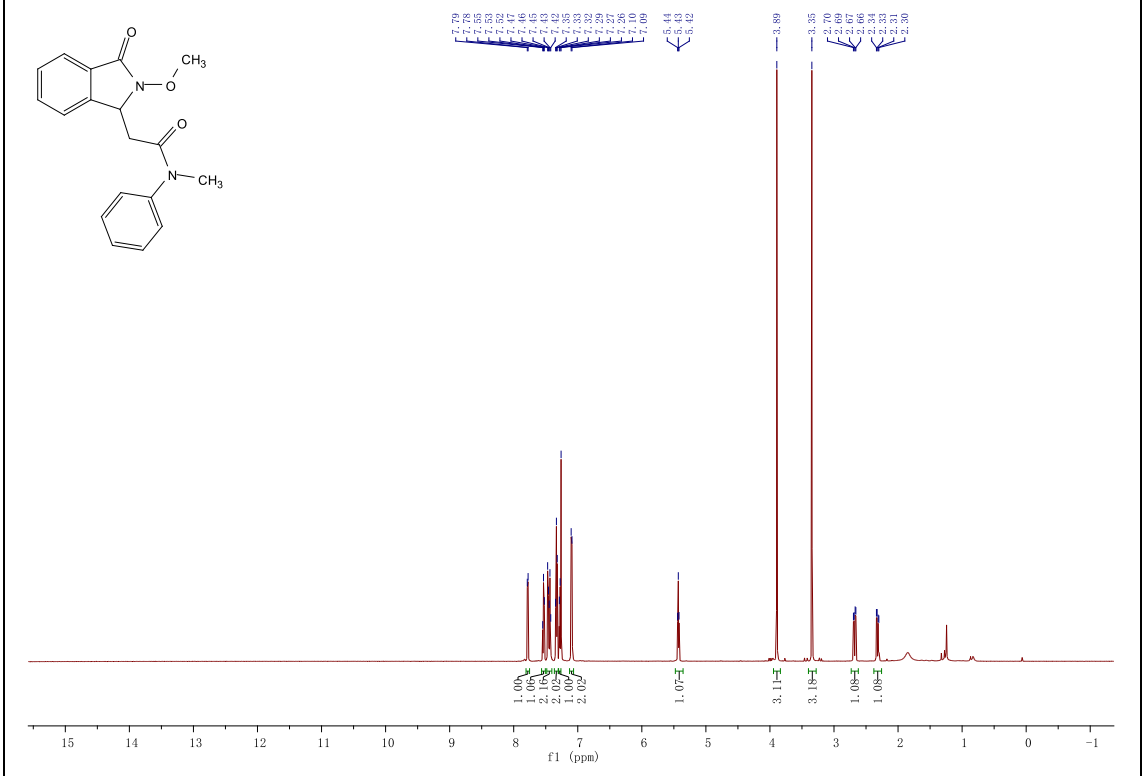


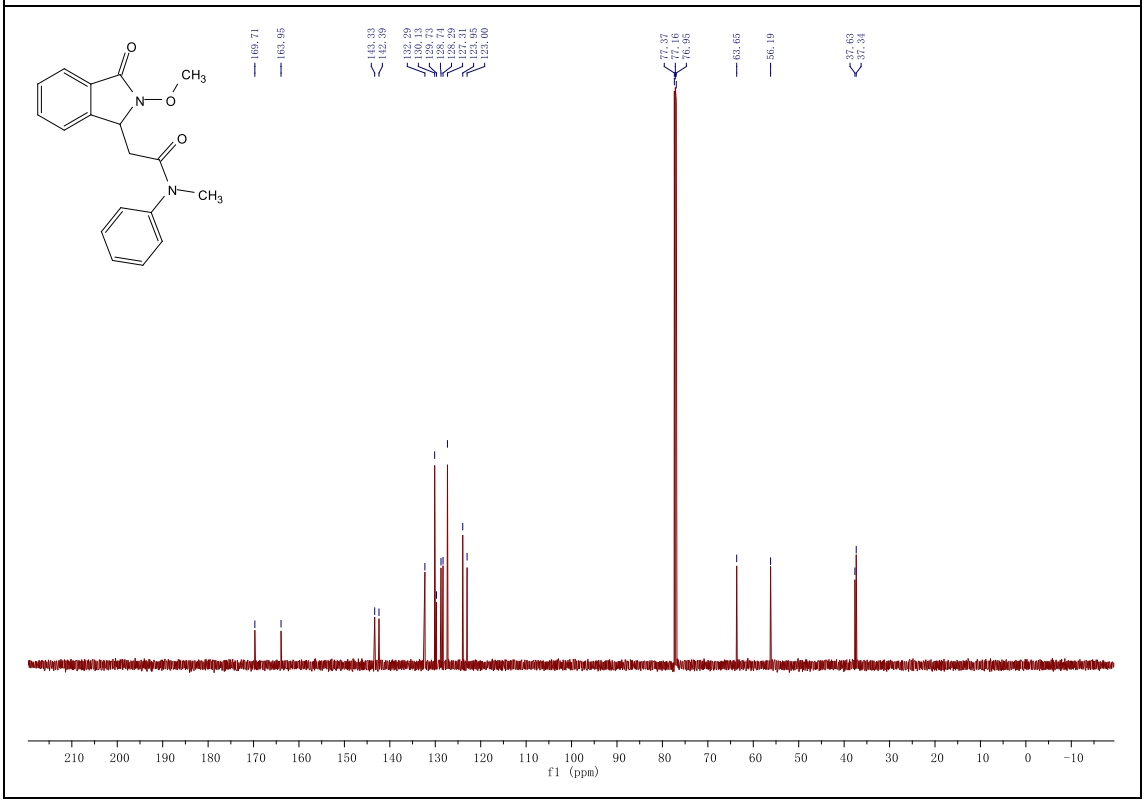
4-methoxyphenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (4am)



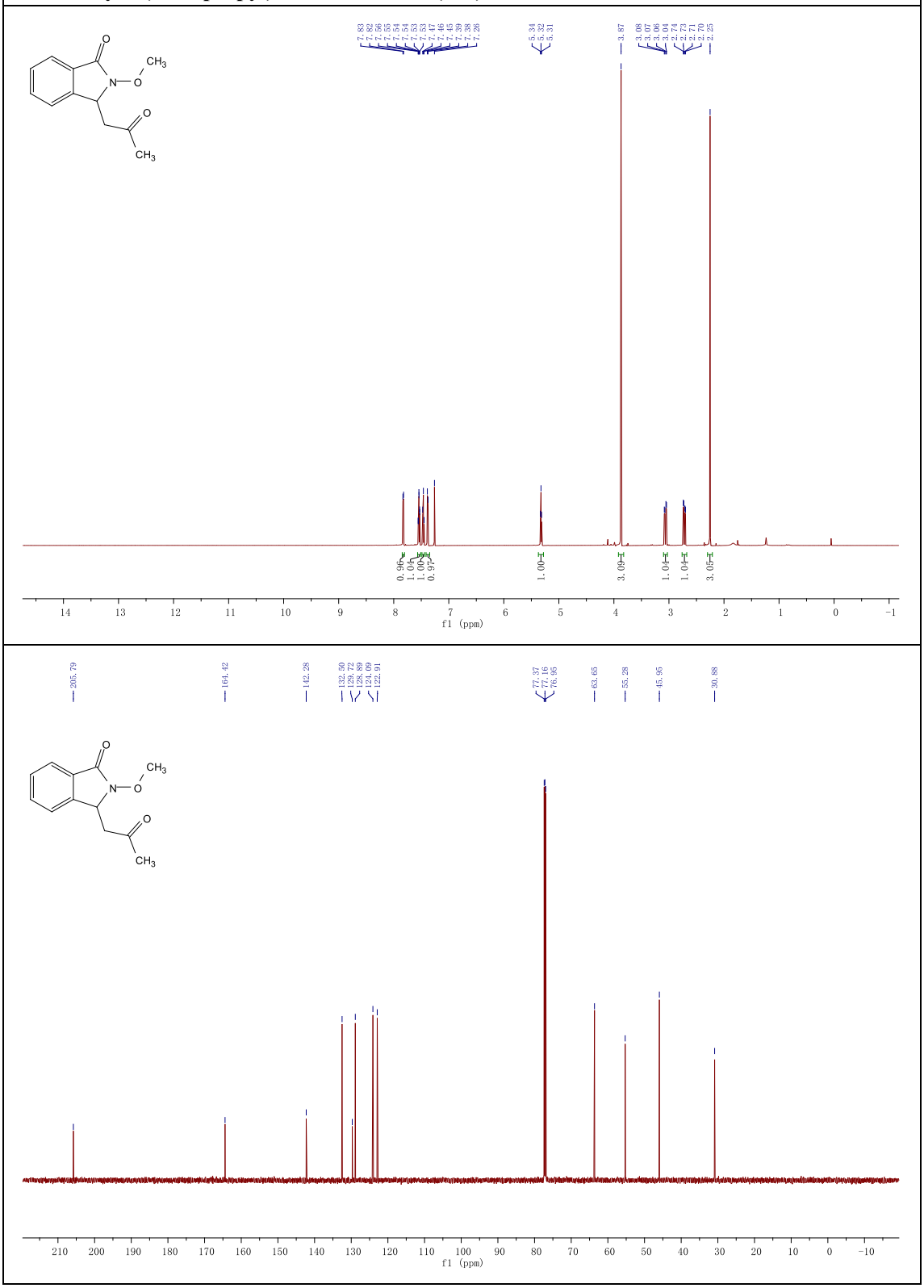


2-(2-methoxy-3-oxoisoindolin-1-yl)-*N*-methyl-*N*-phenylacetamide (4an)

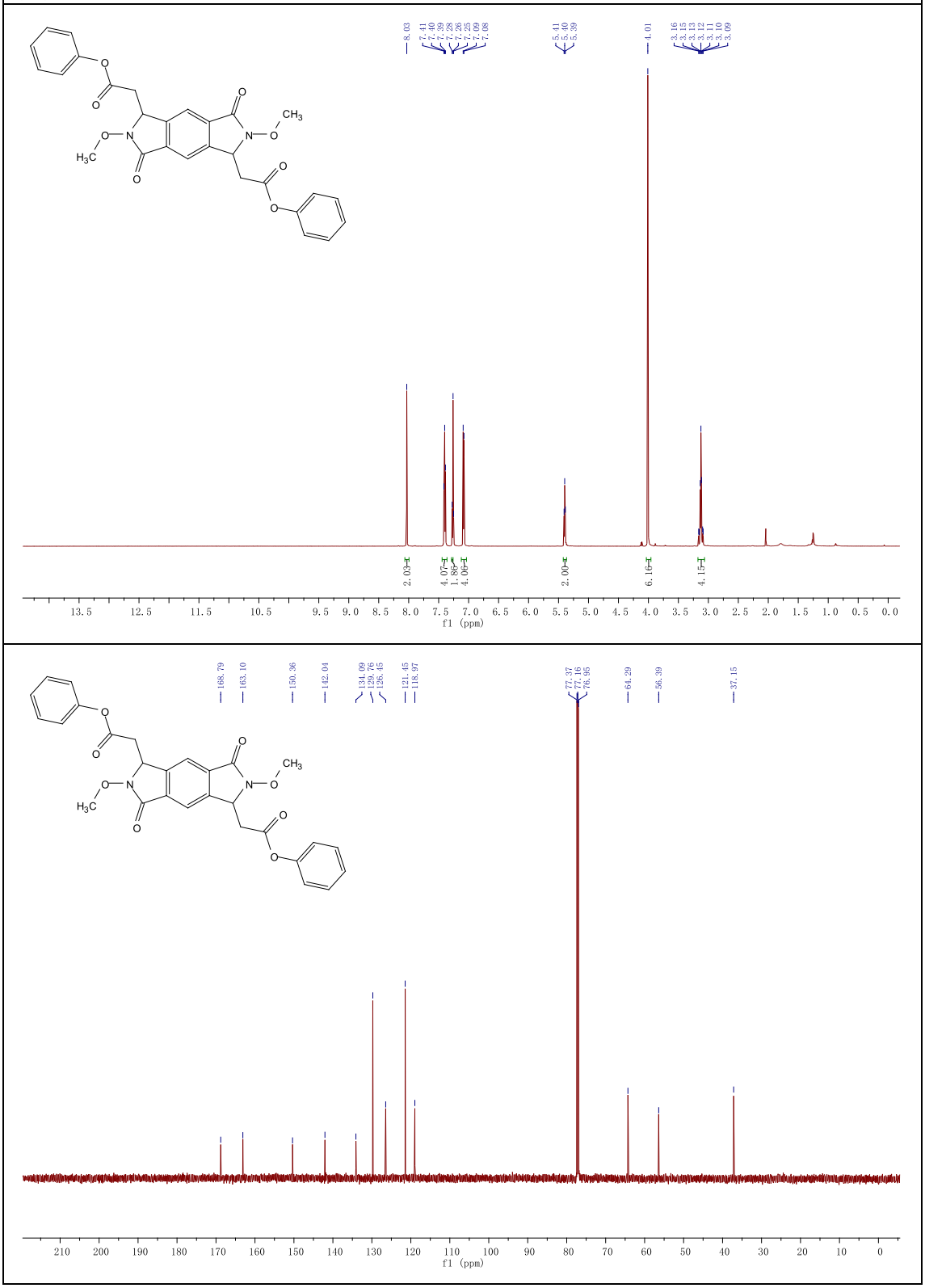




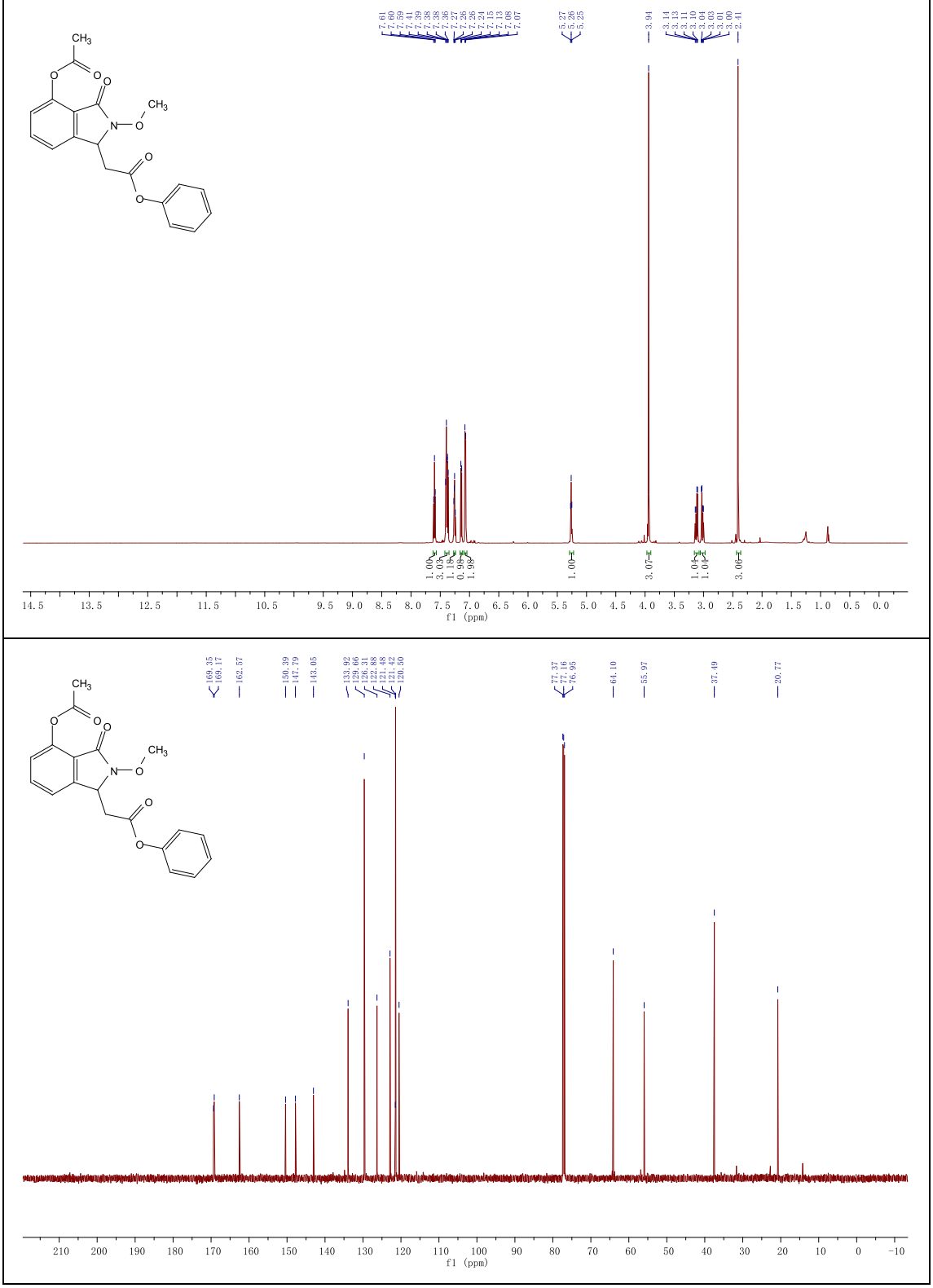
2-methoxy-3-(2-oxopropyl)isoindolin-1-one (4ao)



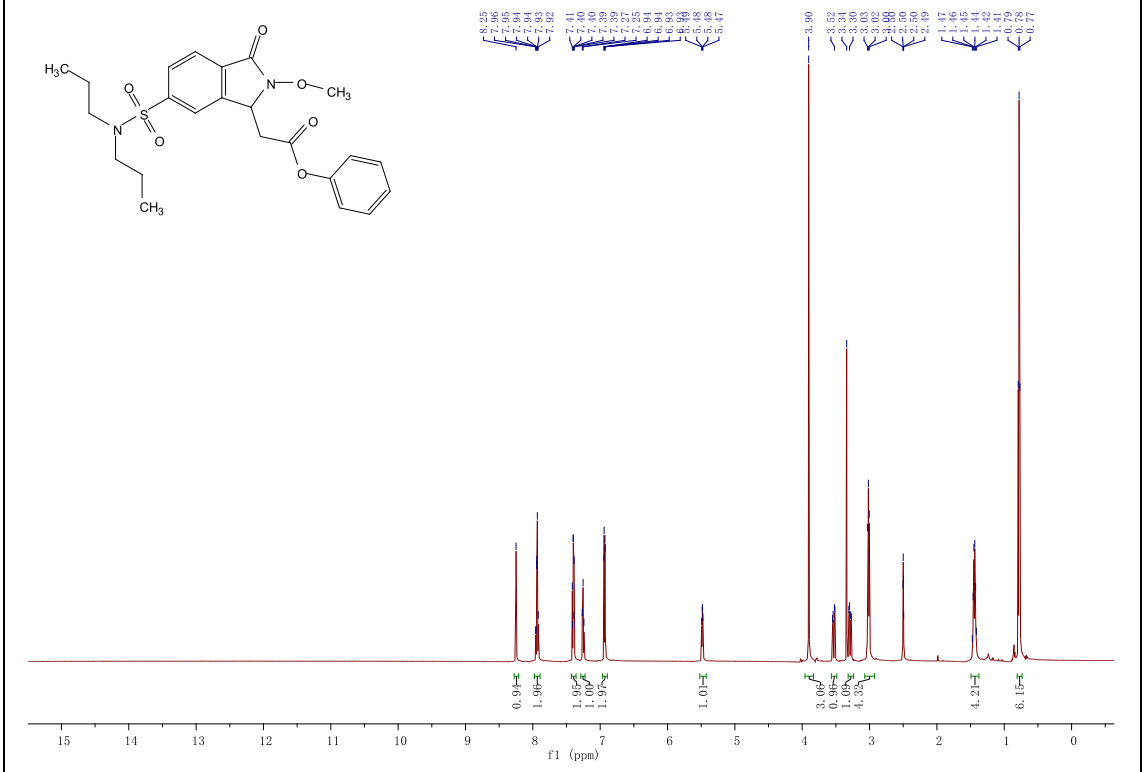
diphenyl 2,2'-(2,6-dimethoxy-3,7-dioxo-1,2,3,5,6,7-hexahydropyrrolo[3,4-*f*]isoindole-1,5-diyl)diacetate (5a)

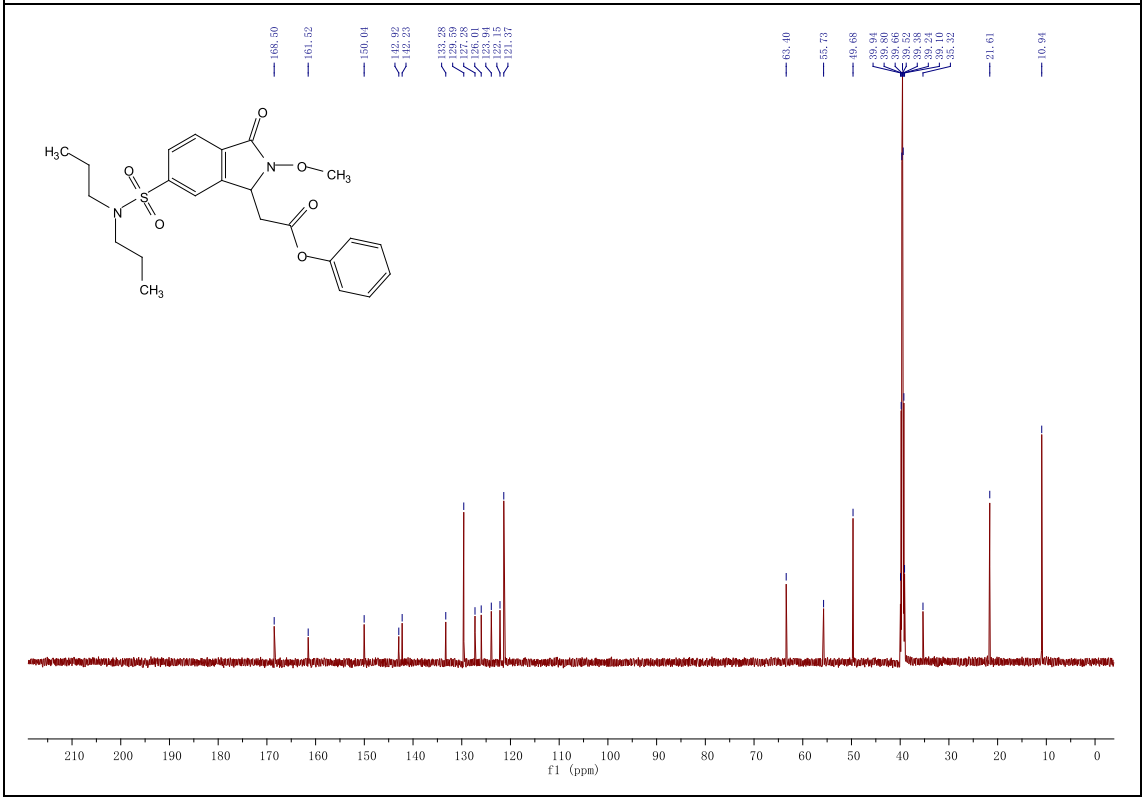


phenyl 2-(4-acetoxy-2-methoxy-3-oxoisoindolin-1-yl)acetate (5b)

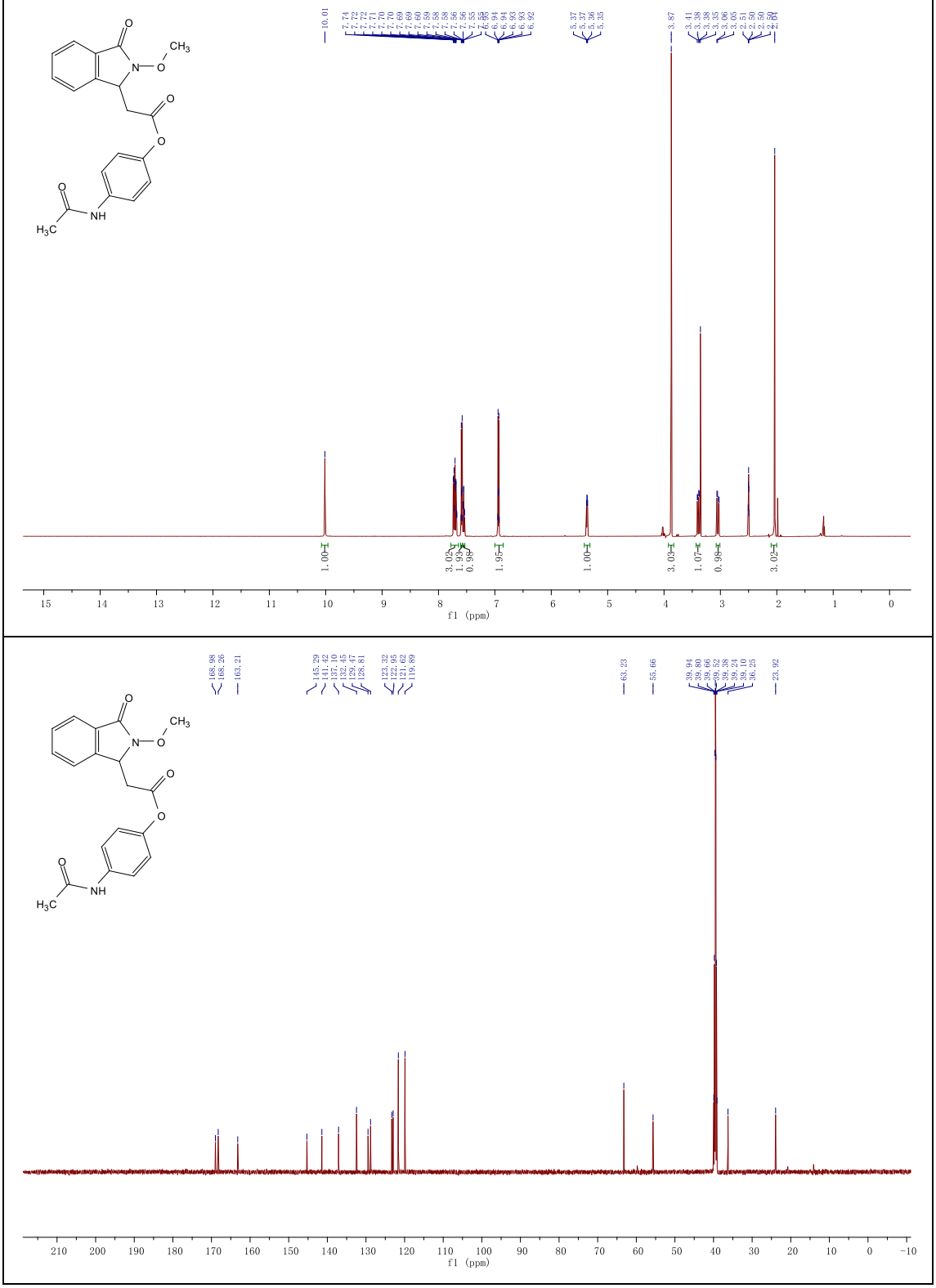


phenyl 2-(6-(*N*,*N*-dipropylsulfamoyl)-2-methoxy-3-oxoisoindolin-1-yl)acetate (5c)





4-acetamidophenyl 2-(2-methoxy-3-oxoisoindolin-1-yl)acetate (5d)



phenyl (*E*)-3-(2-carbamoylphenyl)acrylate (3aa'')