Sulfonyl Radical Triggered Selective Iodosulfonylation and Bicyclizations of 1,6-Dienes

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Supporting Information

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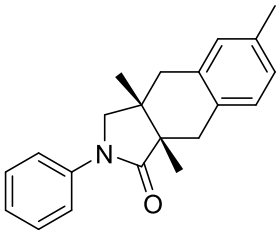
(A) Typical experimental procedure for the selective cyclization

To a Schlenk tube were added 1,6-dienes **1** (0.2 mmol), sulfonyl hydrazides **2** (0.4 mmol), CuI (1.2 equiv), TBHP (2.0 equiv), and MeCN (2.0 mL). Then the tube was stirred at 90 °C sealed in air for the indicated time until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the mixture was extracted three times with EtOAc. The organic layer was dried over Na_2SO_4, filtration and evaporation of the solvent. The mixture was purified by flash column chromatography over silica gel (hexane/ethyl acetate = 3:1) to afford the desired products **4**.

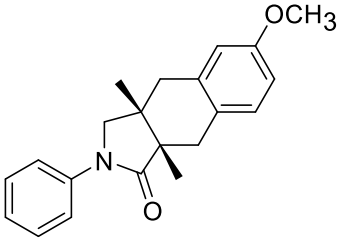
To a Schlenk tube were added 1,6-dienes **1** (0.2 mmol), sulfonyl hydrazides **2** (0.4 mmol), $Cu(OAc)_2$ (20 mol%), TBHP (2.0 equiv), and MeCN (2.0 mL). Then the tube was stirred at 90 °C sealed in air for the indicated time until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the mixture was extracted three times with EtOAc. The organic layer was dried over Na_2SO_4, filtration and evaporation of the solvent. The mixture was purified by flash column chromatography over silica gel (hexane/ethyl acetate = 6:1) to afford the desired products **3**.

(B) Analytical data

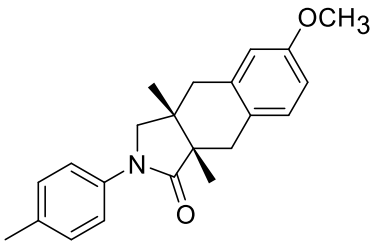


3a,6,9a-Trimethyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3a), white solid (0.0439 g, 72% yield,

d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.45 (d, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 3.5 Hz, 2H), 6.92 (t, *J* = 9.5 Hz, 1H), 3.54 (t, *J* = 4.5 Hz, 1H), 3.42 (d, *J* = 9.5 Hz, 1H), 3.10 (d, *J* = 15.5 Hz, 1H), 2.71 (d, *J* = 14.5 Hz, 1H), 2.64 (d, *J* = 14.5 Hz, 1H), 2.58 (d, *J* = 15.5 Hz, 1H), 2.28 (s, 3H), 1.27 (s, 3H), 1.21 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 178.1, 139.4, 136.1 (2), 133.3, 128.7, 128.6, 127.4, 127.0, 124.5, 120.1, 59.5, 49.2, 41.8, 38.9, 38.2, 24.0, 21.1, 21.0; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO$ ($[M+H]^+$) 306.1852, found 306.1856.

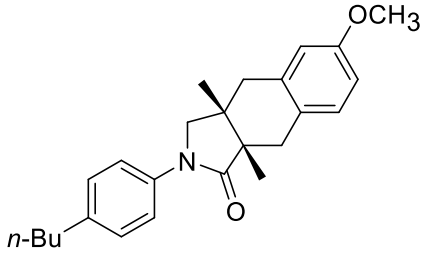


6-Methoxy-3a,9a-dimethyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3b), white solid (0.0475 g, 74% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.46 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 6.72 (d, *J* = 2.5 Hz, 1H), 6.68-6.66 (m, 1H), 3.75 (s, 3H), 3.54 (d, *J* = 9.5 Hz, 1H), 3.42 (d, *J* = 9.5 Hz, 1H), 3.12 (d, *J* = 15.5 Hz, 1H), 2.69 (t, *J* = 7.5 Hz, 1H), 2.62-2.57 (m, 2H), 1.27 (s, 3H), 1.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 178.1, 158.4, 139.4, 137.5, 128.8, 128.5, 128.4, 124.5, 120.2, 113.2, 112.1, 59.5, 55.3, 49.2, 41.4, 39.2, 38.2, 23.9, 21.0; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO_2$ ($[M+H]^+$) 322.1802, found 322.1808.

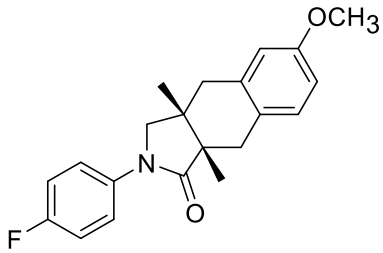


6-Methoxy-3a,9a-dimethyl-2-(*p*-tolyl)-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3c), white solid (0.0503 g, 75% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.31 (d, *J* = 8.5 Hz, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 1H), 6.72 (d, *J* = 2.0 Hz, 1H), 6.68-6.66 (m, 1H),

3.75 (s, 3H), 3.52 (d, *J* = 9.5 Hz, 1H), 3.38 (d, *J* = 10.0 Hz, 1H), 3.11 (d, *J* = 15.5 Hz, 1H), 2.68 (t, *J* = 7.5 Hz, 1H), 2.59 (t, *J* = 14.0 Hz, 2H), 2.28 (s, 3H), 1.27 (s, 3H), 1.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 177.9, 158.4, 137.6, 136.8, 134.3, 129.3, 128.5, 128.3, 120.3, 113.2, 112.1, 59.8, 55.3, 49.2, 41.5, 39.3, 38.3, 24.0, 21.0, 20.8; HRMS *m/z* (ESI) calcd for $C_{22}H_{26}NO_2$ ($[M+H]^+$) 336.1958, found 336.1950.

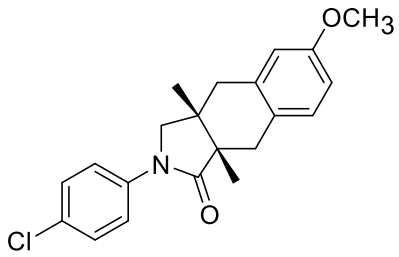


2-(4-Butylphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3d), white solid (0.0543 g, 72% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.36 (d, *J* = 9.0 Hz, 2H), 7.11 (d, *J* = 9.5 Hz, 2H), 6.99 (d, *J* = 10.0 Hz, 1H), 6.72 (s, 1H), 6.67 (d, *J* = 9.5 Hz, 1H), 3.76 (s, 3H), 3.52 (d, *J* = 12.0 Hz, 1H), 3.39 (d, *J* = 11.5 Hz, 1H), 3.11 (d, *J* = 18.5 Hz, 1H), 2.66 (s, 1H), 2.61 (d, *J* = 12.5 Hz, 1H), 2.57-2.51 (m, 3H), 1.54 (t, *J* = 9.0 Hz, 2H), 1.32 (t, *J* = 8.5 Hz , 2H), 1.27 (s, 3H), 1.20 (s, 3H), 0.90 (t, *J* = 8.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 177.8, 158.3, 139.3, 137.6, 137.0, 128.7, 128.6, 128.4, 120.1, 113.1, 112.0, 59.6, 55.3, 49.1, 41.4, 39.3, 38.2, 35.0, 33.7, 24.1, 22.3, 21.1, 14.0; HRMS *m/z* (ESI) calcd for $C_{25}H_{32}NO_2$ ($[M+H]^+$) 378.2428, found 378.2432.

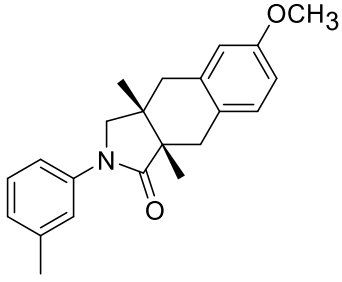


2-(4-Fluorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3e), yellow oil (0.0461 g, 68% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.39-7.36 (m, 2H), 7.00-6.95 (m, 3H), 6.76 (s, 1H), 6.72 (d, *J* = 2.5 Hz, 1H), 3.75 (s, 3H), 3.51 (d, *J* = 9.5

Hz, 1H), 3.37 (d, *J* = 9.5 Hz, 1H), 3.10 (d, *J* = 15.0 Hz, 1H), 2.69-2.66 (m, 1H), 2.60 (t, *J* = 14.5 Hz, 2H), 1.28 (s, 3H), 1.22 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 177.9, 159.0 (d, J_{C-F} = 242.8 Hz), 158.4, 137.6, 135.3 (d, J_{C-F} = 2.9 Hz), 128.4, 128.3,122.0 (d, J_{C-F} = 7.9 Hz), 115.4 (d, J_{C-F} = 22.3 Hz), 113.0, 112.1, 59.9, 55.3, 49.1, 41.5, 39.3, 38.3, 24.0, 21.0; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -117.6; HRMS *m/z* (ESI) calcd for $C_{21}H_{23}FNO_2$ ($[M+H]^+$) 340.1707, found 340.1701.

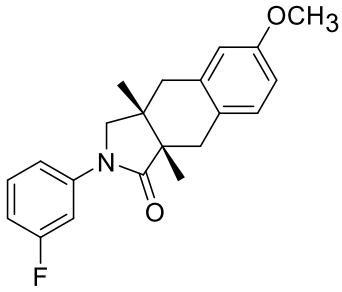


2-(4-Chlorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3f), colorless oil (0.0469 g, 66% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (d, *J* = 9.0 Hz, 2H), 7.25 (d, *J* = 10.5 Hz, 2H), 6.99 (d, *J* = 9.5 Hz, 1H), 6.72 (s, 1H), 6.68 (d, *J* = 10.0 Hz, 1H), 3.76 (s, 3H), 3.51 (d, *J* = 11.5 Hz, 1H), 3.38 (d, *J* = 11.5 Hz, 1H), 3.10 (d, *J* = 18.0 Hz, 1H), 2.68 (t, *J* = 9.0 Hz, 1H), 2.61 (t, *J* = 16.0 Hz, 2H), 1.28 (s, 3H), 1.22 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 178.1, 158.4, 137.8, 137.5, 129.5, 128.7, 128.6, 128.4, 121.1, 113.1, 112.0, 59.4, 55.3, 49.2, 41.4, 39.3, 38.2, 24.0, 21.1; HRMS *m/z* (ESI) calcd for $C_{21}H_{23}ClNO_2$ ($[M+H]^+$) 356.1412, found 356.1414.

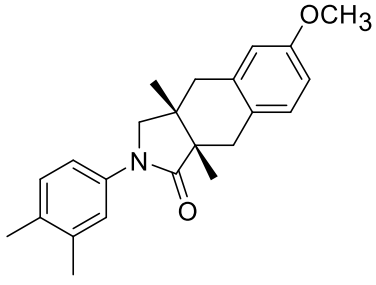


6-Methoxy-3a,9a-dimethyl-2-(*m*-tolyl)-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3g), white solid (0.0489 g, 73% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.32 (s, 1H), 7.21-7.16 (m, 2H), 6.99 (d, *J* = 8.0 Hz, 1H), 6.91 (d, *J* = 7.0 Hz, 1H), 6.76 (s, 1H), 6.72 (d, *J* = 2.5 Hz, 1H), 3.75 (s, 3H), 3.53 (d, *J* = 10.0 Hz, 1H), 3.41 (d, *J* = 10.0 Hz, 1H), 3.11 (d, *J* = 15.5 Hz, 1H),

2.68 (d, *J* = 15.0 Hz, 1H), 2.62-2.57 (m, 2H), 2.30 (s, 3H), 1.27 (s, 3H), 1.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 178.2, 158.4, 139.2, 138.7, 137.5, 128.6, 128.5, 128.4, 125.5, 121.1, 117.3, 113.2, 112.1, 59.8, 55.3, 49.2, 41.4, 39.2, 38.3, 24.0, 21.5, 21.0; HRMS *m/z* (ESI) calcd for $C_{22}H_{26}NO_2$ ($[M+H]^+$) 336.1958, found 336.1962.

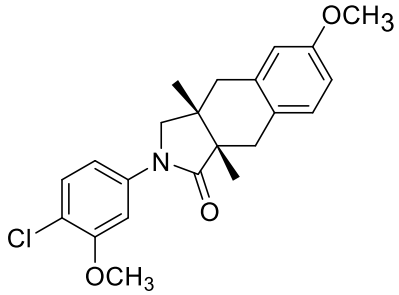


2-(3-Fluorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3h), yellow oil (0.0454 g, 67% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.38 (d, *J* = 14.0 Hz, 1H), 7.25-7.18 (m, 2H), 7.00 (d, *J* = 9.5 Hz, 1H), 6.83-6.78 (m, 2H), 6.69 (t, *J* = 12.0 Hz, 1H), 3.75 (s, 3H), 3.52 (d, *J* = 11.5 Hz, 1H), 3.40 (d, *J* = 12.0 Hz, 1H), 3.09 (t, *J* = 17.5 Hz, 1H), 2.70-2.53 (m, 3H), 1.28 (s, 3H), 1.22 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 178.6, 162.5 (d, J_{C-F} = 303.9 Hz), 158.4, 140.6 (d, J_{C-F} = 13.3 Hz), 137.4, 129.9 (d, J_{C-F} = 11.5 Hz), 128.4 (d, J_{C-F} = 12.5 Hz), 116.1, 115.1 (d, J_{C-F} = 3.5 Hz), 114.8, 112.5, 112.2, 59.5, 55.3, 49.5, 41.4, 39.2, 38.2, 24.0, 21.1; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -111.5; HRMS *m/z* (ESI) calcd for $C_{21}H_{23}FNO_2$ ($[M+H]^+$) 340.1707, found 340.1703.

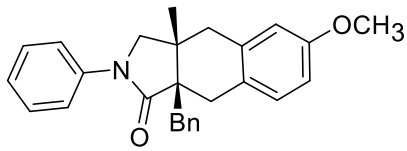


2-(3,4-Dimethylphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3i), white solid (0.0531 g, 76% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.27 (d, *J* = 9.5 Hz, 1H), 7.12 (d, *J* = 7.0 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.99 (d, *J* = 7.5 Hz, 1H), 6.74 (d, *J* = 22.5 Hz, 1H), 6.67 (d, *J* = 7.5 Hz, 1H), 3.76 (s, 3H), 3.51 (d, *J* = 9.5 Hz, 1H), 3.38

(d, *J* = 9.5 Hz, 1H), 3.11 (d, *J* = 15.5 Hz, 1H), 2.69-2.56 (m, 3H), 2.20 (d, *J* = 10.5 Hz, 6H), 1.26 (s, 3H), 1.19 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 177.8, 158.4, 137.6, 137.2, 137.0, 133.0, 129.7, 128.6, 128.3, 121.6, 117.7, 113.1, 112.0, 59.7, 55.3, 49.1, 41.4, 39.3, 38.2, 24.0, 21.0, 20.0, 19.2; HRMS m/z (ESI) calcd for $C_{23}H_{28}NO_2$ ($[M+H]^+$) 350.2115, found 350.2117.

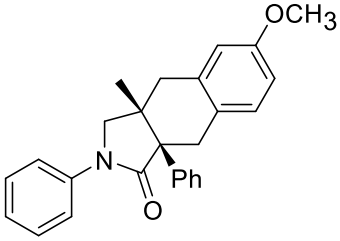


2-(4-Chloro-3-methoxyphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3j), white solid (0.0547 g, 71% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.88-7.85 (m, 2H), 7.68 (d, *J* = 2.5 Hz, 1H), 7.31 (d, *J* = 8.5 Hz, 1H), 7.02-6.99 (m, 2H), 3.93-3.92 (m, 1H), 3.88 (d, *J* = 2.5 Hz, 6H), 3.85-3.75 (m, 1H), 3.66 (d, *J* = 10.0 Hz, 1H), 3.60 (d, *J* = 15.0 Hz, 1H), 3.39 (d, *J* = 9.5 Hz, 1H), 3.27 (d, *J* = 15.0 Hz, 1H), 1.47 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 175.8, 163.7, 155.1, 139.0, 133.2, 129.8 (2), 129.1, 118.2, 114.7, 114.5, 111.3, 104.5, 59.5, 59.2, 56.1, 55.7, 52.6, 38.7, 25.2, 21.5, 18.1; HRMS *m/z* (ESI) calcd for $C_{22}H_{25}ClNO_3$ ($[M+H]^+$) 386.1517, found 386.1511.

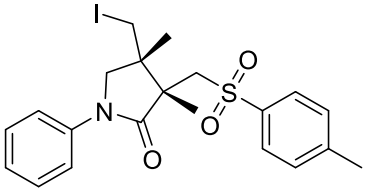


9a-Benzyl-6-methoxy-3a-methyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3k), colorless oil (0.0564 g, 71% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.77 (d, *J* = 9.5 Hz, 2H), 7.66 (d, *J* = 10.0 Hz, 2H), 7.38 (t, *J* = 9.0 Hz, 2H), 7.21-7.10 (m, 5H), 6.97 (d, *J* = 9.5 Hz, 2H), 4.29 (d, *J* = 13.0 Hz, 1H), 3.92 (d, *J* = 13.5 Hz, 1H), 3.87 (s, 3H), 3.40 (d, *J* = 21.0 Hz, 1H), 3.24 (d, *J* = 17.5 Hz, 1H), 3.11 (t, *J* = 10.0 Hz,

3H), 2.99 (d, *J* = 18.0 Hz, 1H), 1.51 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 175.7, 163.9, 140.9, 139.4, 139.3, 132.6, 129.7, 128.9, 127.1, 127.0, 124.6, 124.2, 123.9, 119.7, 114.6, 61.8, 61.0, 55.8, 41.6, 37.5, 35.5, 21.2; HRMS *m/z* (ESI) calcd for $C_{27}H_{28}NO_2$([M+H]$^+$) 398.2115, found 398.2119.

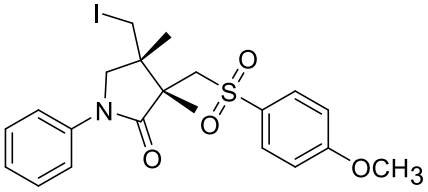


6-Methoxy-3a-methyl-2,9a-diphenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3l), colorless oil (0.0475 g, 62% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.54-7.52 (m, 2H), 7.36 (t, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.0 Hz, 1H), 7.18-7.15 (m, 3H), 7.05 (d, *J* = 8.5 Hz, 1H), 6.90 (d, *J* = 2.5 Hz, 1H), 6.74-6.72 (m, 1H), 3.82 (s, 3H), 3.70 (d, *J* = 9.5 Hz, 1H), 3.59 (d, *J* = 9.5 Hz, 1H), 3.44-3.35 (m, 2H), 2.82 (d, *J* = 14.0 Hz, 1H), 2.67 (d, *J* = 14.5 Hz, 1H), 0.66 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 176.7, 158.7, 142.1, 139.0, 138.0, 128.9, 128.7, 128.4, 128.3, 127.4, 126.8, 125.0, 120.6, 113.4, 111.9, 59.8, 58.9, 55.4, 42.3, 40.8, 38.1, 25.5; HRMS *m/z* (ESI) calcd for $C_{26}H_{26}NO_2$ ([M+H]$^+$) 384.1985, found 384.1989.

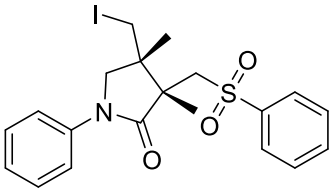


4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-(tosylmethyl)pyrrolidin-2-one (4a), colorless oil (0.0805 g, 81% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.86-7.81 (m, 2H), 7.60-7.54 (m, 2H), 7.42-7.34 (m, 4H), 7.18 (t, *J* = 9.5 Hz, 1H), 4.04-3.90 (m, 2H), 3.65-3.61 (m, 2H), 3.59-3.51 (m, 1H), 3.40-3.37 (m, 1H), 2.46 (s, 3H), 1.66 (s, 3H), 1.59 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.4, 145.0, 138.6,

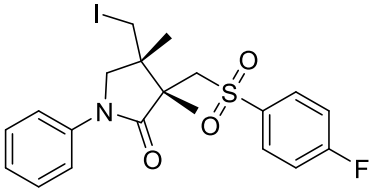
138.2, 130.0, 129.0, 127.6, 125.3, 120.3, 59.2, 57.8, 51.5, 41.7, 21.7, 20.8, 20.2, 16.1; HRMS *m/z* (ESI) calcd for $C_{21}H_{25}INO_3S$ ($[M+H]^+$) 498.0594, found 498.0598.



4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl) methyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4b), colorless oil (0.0872 g, 85% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.89-7.84 (m, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.38-7.33 (m, 2H), 7.18-7.14 (m, 1H), 7.01 (d, *J* = 8.5 Hz, 2H), 3.92 (d, *J* = 10.5 Hz, 2H), 3.88 (s, 3H), 3.70-3.60 (m, 2H), 3.58 (d, *J* = 10.0 Hz, 1H), 3.39 (t, *J* = 7.5 Hz, 1H), 1.64 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.4, 163.9, 138.6, 132.8, 129.8, 129.0, 125.2, 120.3, 114.6, 59.5, 57.8, 55.8, 51.5, 41.7, 20.9, 20.1, 16.1; HRMS *m/z* (ESI) calcd for $C_{21}H_{25}INO_4S$ ($[M+H]^+$) 514.0543, found 514.0547.

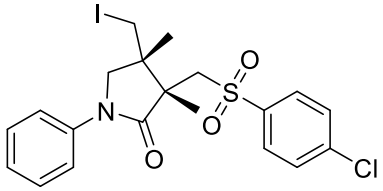


4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((phenylsulfo nyl)methyl)pyrrolidin-2-one (4c), colorless oil (0.0773 g, 80% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.96-7.90 (m, 2H), 7.68-7.65 (m, 1H), 7.60-7.56 (m, 2H), 7.56-7.54 (m, 2H), 7.39-7.35 (m, 2H), 7.19-7.16 (m, 1H), 3.94-3.90 (m, 2H), 3.66-3.63 (m, 2H), 3.58 (d, *J* = 10.0 Hz, 1H), 3.41 (d, *J* = 15.5 Hz, 1H), 1.66 (s, 3H), 1.59 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.3, 141.0, 138.6, 134.0, 129.5, 129.0, 127.6, 125.3, 120.3, 59.2, 57.8, 51.6, 41.7, 20.8, 20.2, 16.0; HRMS *m/z* (ESI) calcd for $C_{20}H_{23}INO_3S$ ($[M+H]^+$) 484.0438, found 484.0434.

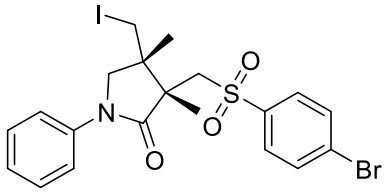


3-(((4-Fluorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4d),

colorless oil (0.0782 g, 78% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.98-7.95 (m, 2H), 7.55-7.53 (m, 2H), 7.39-7.36 (m, 2H), 7.27-7.24 (m, 2H), 7.20-7.17 (m, 1H), 3.92-3.89 (m, 2H), 3.65-3.62 (m, 2H), 3.58 (d, *J* = 10.5 Hz, 1H), 3.41 (d, *J* = 15.0 Hz, 1H), 1.66 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.2, 165.9 (d, J_{C-F} = 255.5 Hz), 138.5, 137.1, 130.5 (d, J_{C-F} = 9.5 Hz), 129.1, 125.3, 120.3, 116.8 (d, J_{C-F} =22.5 Hz), 59.4, 57.8, 51.6, 41.7, 20.8, 20.2, 15.8; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -102.8; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}FINO_3S$ ($[M+H]^+$) 502.0344, found 502.0340.

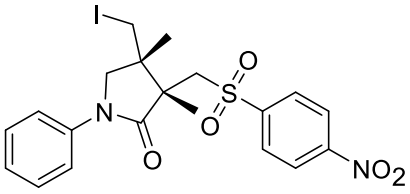


3-(((4-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4e), colorless oil (0.0796 g, 77% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.92-7.87 (m, 2H), 7.59-7.51 (m, 4H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 3.97-3.87 (m, 2H), 3.65-3.62 (m, 2H), 3.57 (d, *J* = 10.5 Hz, 1H), 3.40 (d, *J* = 15.0 Hz, 1H), 1.65 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.1, 140.8, 139.4, 138.5, 129.8, 129.1 (2), 125.4, 120.3, 59.3, 57.8, 51.6, 41.7, 20.9, 20.2, 15.7; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}ClINO_3S$ ($[M+H]^+$) 518.0048, found 518.0054.

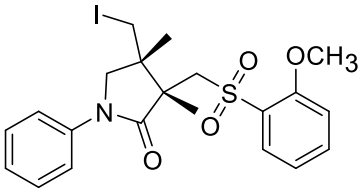


3-(((4-Bromophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4f), colorless oil (0.0853 g, 76% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.84-7.78 (m, 2H), 7.73-7.69 (m, 2H), 7.53 (t, *J* = 4.0 Hz, 2H), 7.40-7.36 (m, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 3.92-3.87 (m, 2H), 3.67-3.61 (m,

2H), 3.57 (d, *J* = 10.0 Hz, 1H), 3.40 (d, *J* = 15.0 Hz, 1H), 1.65 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.1, 139.9, 138.5, 132.8, 129.4, 129.2, 129.1, 125.4, 120.3, 59.2, 57.8, 51.6, 41.7, 20.9, 20.2, 15.7; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}BrINO_3S$ ($[M+H]^+$) 561.9543, found 561.9547.

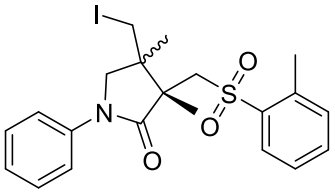


4-(Iodomethyl)-3,4-dimethyl-3-(((4-nitrophenyl)sulfonyl)methyl)-1-phenylpyrrolidin-2-one (4g), colorless oil (0.0718 g, 68% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.37-8.35 (m, 2H), 8.08-8.05 (m, 2H), 7.47-7.44 (m, 2H), 7.41-7.38 (m, 1H), 7.31 (d, *J* = 7.5 Hz, 2H), 4.78 (d, *J* = 52.0 Hz, 2H), 4.31-4.22 (m, 2H), 4.03-3.99 (m, 1H), 2.98-2.95 (m, 1H), 1.78 (s, 3H), 1.12 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 172.9, 150.8, 145.6, 141.6, 140.3, 129.7, 129.3, 128.4, 124.4, 59.5, 56.7, 55.8, 41.3, 32.0, 20.3, 18.7; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}IN_2O_5S$ ($[M+H]^+$) 529.0289, found 529.0293.

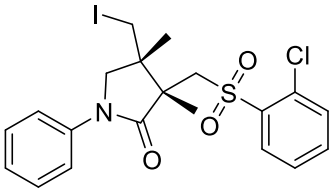


4-(Iodomethyl)-3-(((2-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4h), colorless oil (0.0862 g, 84% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.97-7.95 (m, 1H), 7.61-7.56 (m, 3H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 8.0 Hz, 1H), 7.04 (d, *J* = 8.5 Hz, 1H), 4.01 (s, 3H), 3.95 (d, *J* = 10.0 Hz, 1H), 3.90 (t, *J* = 10.0 Hz, 1H), 3.85 (d, *J* = 14.5 Hz, 2H), 3.74-3.64 (m, 2H), 1.65 (s, 3H), 1.59 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.6, 157.2, 138.6, 135.8, 130.1, 129.0, 128.7, 125.2, 120.9, 120.3, 112.4, 57.9, 57.0, 56.6,

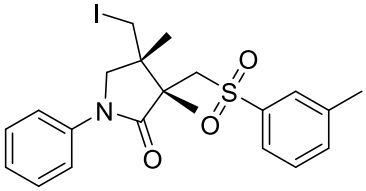
51.4, 41.6, 21.1, 20.3, 16.2; HRMS *m/z* (ESI) calcd for $C_{21}H_{25}INO_4S$ ($[M+H]^+$) 514.0543, found 514.0547.



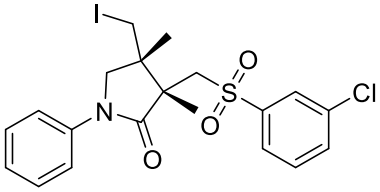
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((*o*-tolylsulfonyl)methyl)pyrrolidin-2-one (4i), colorless oil (0.0805 g, 81% yield, d.r. = 1.5:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 7.5 Hz, 0.4H), 7.81 (d, *J* = 8.5 Hz, 0.6H), 7.56-7.53 (m, 3H), 7.39-7.34 (m, 4H), 7.18 (t, *J* = 7.0 Hz, 1H), 3.95-3.89 (m, 2H), 3.66-3.57(m, 3H), 3.45-3.37 (m, 1H), 2.75 (s, 1.8H), 2.45 (s, 1.2H), 1.66 (s, 1.8H), 1.65 (s, 1.2H), 1.59 (s, 1.8H), 1.59 (s, 1.2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.4 (2), 145.0, 139.2, 138.6 (2), 137.8, 134.0, 133.0, 130.0, 129.5, 128.9, 127.6, 126.9, 125.3, 120.3, 59.3, 58.0, 57.8, 51.6, 51.5, 41.7 (2), 21.7, 20.8, 20.5, 20.3, 16.1, 16.0; HRMS *m/z* (ESI) calcd for $C_{21}H_{25}INO_3S$ ($[M+H]^+$) 498.0594, found 498.0590.



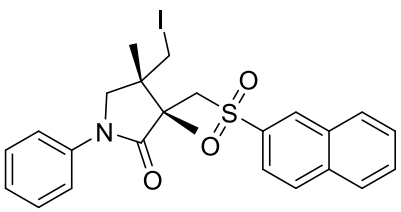
3-(((2-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4j), colorless oil (0.0786 g, 76% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16-8.14 (m, 1H), 7.60-7.54 (m, 4H), 7.50-7.47 (m, 1H), 7.39-7.36 (m, 2H), 7.18 (t, *J* = 7.0 Hz, 1H), 3.97-3.87 (m, 3H), 3.73 (d, *J* = 15.0 Hz, 1H), 3.64 (t, *J* = 11.0 Hz, 2H), 1.68 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.1, 138.5, 138.2, 135.0, 132.8, 132.1, 131.2, 129.0, 127.6, 125.2, 120.2, 57.8, 56.9, 51.5, 41.8, 20.9, 20.2, 15.8; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}ClINO_3S$ ($[M+H]^+$) 518.0048, found 581.0054.



4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((*m*-tolylsulfonyl)methyl)pyrrolidin-2-one (4k), colorless oil (0.0796 g, 80% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.74-7.70 (m, 2H), 7.56-7.53 (m, 2H), 7.47-7.45 (m, 2H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 7.0 Hz, 1H), 3.97-3.90 (m, 2H), 3.62 (t, *J* = 7.5 Hz, 2H), 3.58 (d, *J* = 10.0 Hz, 1H), 3.39 (t, *J* = 7.5 Hz, 1H), 2.45 (s, 3H), 1.67 (s, 3H), 1.59 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.4, 140.9, 139.8, 138.6, 134.7, 129.3, 129.0, 127.9, 125.3, 124.6, 120.3, 59.1, 57.8, 51.6, 41.8, 21.4, 20.8, 20.2, 16.2; HRMS *m/z* (ESI) calcd for $C_{21}H_{25}INO_3S$ ($[M+H]^+$) 498.0594, found 498.0590.

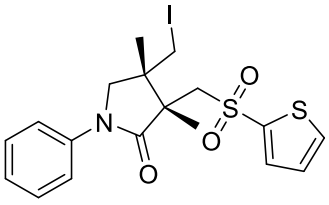


3-(((3-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4l), yellow oil (0.0775 g, 75% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.93 (t, *J* = 2.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 9.0 Hz, 1H), 7.55-7.50 (m, 3H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.18 (t, *J* = 7.5 Hz, 1H), 3.90 (t, *J* = 10.0 Hz, 2H), 3.65 (t, *J* = 7.5 Hz, 2H), 3.56 (d, *J* = 10.0 Hz, 1H), 3.41 (d, *J* = 15.0 Hz, 1H), 1.66 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.1, 142.6, 138.5, 135.8, 134.2, 130.8, 129.1, 127.7, 125.7, 125.3, 120.3, 59.2, 57.8, 51.6, 41.8, 20.8, 20.2, 15.8; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}ClINO_3S$ ($[M+H]^+$) 518.0048, found 518.0052.

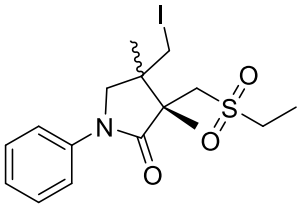


4-(Iodomethyl)-3,4-dimethyl-3-((naphthalen-2-ylsulfonyl)methyl)-1-phenylpyrrolidin-2-one (4m), colorless oil (0.0757 g, 71% yield, d.r. > 20:1); ^{1}H

NMR (500 MHz, $CDCl_3$) δ: 8.50 (d, *J* = 1.0 Hz, 1H), 8.00-7.97 (m, 2H), 7.92-7.87 (m, 2H), 7.66-7.62 (m, 2H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.37-7.33 (m, 2H), 7.15 (t, *J* = 7.0 Hz, 1H), 3.98-3.91 (m, 2H), 3.73 (d, *J* = 15.0 Hz, 1H), 3.62 (t, *J* = 10.0 Hz, 2H), 3.48 (d, *J* = 15.0 Hz, 1H), 1.68 (s, 3H), 1.61 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.3, 138.6, 137.8, 135.4, 132.2, 129.9, 129.5 (2), 129.4, 129.0, 128.1, 127.9, 125.3, 122.2, 120.3, 59.2, 57.8, 51.6, 41.8, 20.9, 20.2, 16.2; HRMS *m/z* (ESI) calcd for $C_{24}H_{25}INO_3S$ ($[M+H]^+$) 534.0594, found 534.0590.

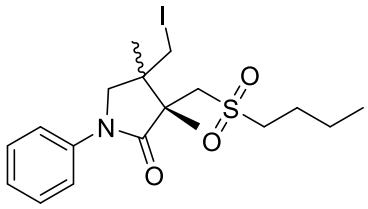


4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((thiophen-2-ylsulfonyl)methyl)pyrrolidin-2-one (4n), colorless oil (0.0665 g, 68% yield, d.r. > 20:1); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.74-7.72 (m, 2H), 7.56-7.54 (m, 2H), 7.40-7.37 (m, 2H), 7.19-7.15 (m, 2H), 3.92-3.88 (m, 2H), 3.82 (d, *J* = 15.0 Hz, 1H), 3.65-3.63 (m, 1H), 3.58 (t, *J* = 12.0 Hz, 2H), 1.66 (s, 3H), 1.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.2, 142.3, 138.5, 134.1, 133.9, 129.1, 128.0, 125.3, 120.3, 60.9, 57.8, 51.6, 41.8, 20.7, 20.1, 15.9; HRMS *m/z* (ESI) calcd for $C_{18}H_{21}INO_3S_2$ ($[M+H]^+$) 490.0002, found 490.0008.

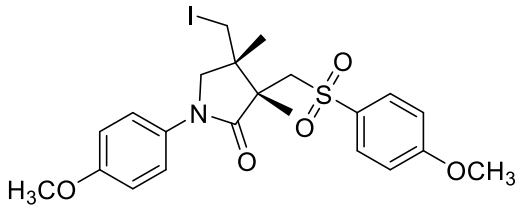


3-((Ethylsulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4o), colorless oil (0.0548 g, 63% yield, d.r.= 1:1); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.59-7.57 (m, 2H), 7.41-7.36 (m, 2H), 7.21-7,16 (m, 1H), 3.89 (d, *J* = 10.5 Hz, 0.5H), 3.80-3.78 (m, 0.5Hz), 3.69 (d, *J* = 10.0 Hz, 0.5H), 3.64-3.62 (m, 0.5H), 3.56-3.52 (m, 1H), 3.39 (d, *J* = 10.0 Hz, 0.5H), 3.33 (d, *J* = 15.0 Hz, 0.5H), 3.13-3.05 (m, 2H), 1.62 (t, *J* = 8.0 Hz, 2H), 1.51 (s, 3H), 1.45-1.42 (m, 3H), 1.38 (s, 1.5H), 1.29 (s, 1.5H); ^{13}C NMR

(125 MHz, $CDCl_3$) δ: 175.6, 174.5, 139.2, 138.5, 129.1, 129.0, 125.4, 125.0, 120.4, 119.9, 59.3, 57.8, 54.3, 54.1, 51.9, 50.9, 50.5 (2), 41.6, 38.9, 25.1, 21.2, 20.8, 20.1, 17.9, 15.7, 6.9, 6.8; HRMS *m/z* (ESI) calcd for $C_{16}H_{23}INO_3S$ ($[M+H]^+$) 436.0438, found 436.0442.

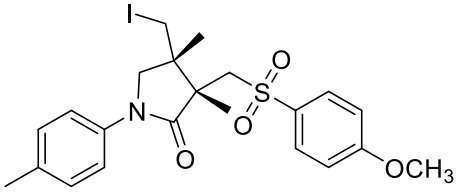


3-((Butylsulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4p), colorless oil (0.0574 g, 62% yield, d.r. = 1:1); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.59-7.57 (m, 2H), 7.41-7.36 (m, 2H), 7.21-7.16 (m, 1H), 3.89 (d, *J* = 10.5 Hz, 0.5H), 3.79 (d, *J* = 10.0 Hz, 0.5H), 3.68 (d, *J* = 9.5 Hz, 0.5H), 3.62 (d, *J* = 10.5 Hz, 0.5H), 3.58-3.52 (m, 1.5H), 3.39 (d, *J* = 10.0 Hz, 1H), 3.33 (d, *J* = 14.5 Hz, 0.5H), 3.19 (d, *J* = 14.5 Hz, 0.5H), 3.09-3.05 (m, 1.5H), 3.04-3.01 (m, 1H), 1.88-1.83 (m, 2H), 1.50 (s, 3H), 1.48-1.41 (m, 2H), 1.38 (s, 1.5H), 1.29 (s, 1.5H), 0.99-0.95 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 175.7, 174.5, 139.2, 138.5, 129.1, 129.0, 125.3, 125.0, 120.3, 119.9, 59.2, 57.8, 55.9 (2), 54.9, 54.8, 52.0, 51.0, 41.6, 38.9, 25.1, 24.3, 24.2, 21.7 (2), 21.2, 20.8, 20.1, 17.9, 15.7, 13.6, 13.5; HRMS *m/z* (ESI) calcd for $C_{18}H_{27}INO_3S$ ($[M+H]^+$) 464.0751, found 464.0754.

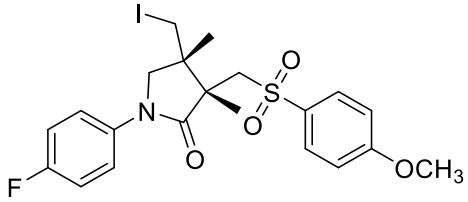


4-(Iodomethyl)-1-(4-methoxyphenyl)-3-((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4q), colorless oil (0.0945 g, 87% yield, d.r. > 20:1); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.87-7.84 (m, 2H), 7.45-7.42 (m, 2H), 7.02-7.00 (m, 2H), 6.90-6.88 (m, 2H), 3.92 (d, *J* = 9.5 Hz, 1H), 3.87 (s, 3H), 3.85 (d, *J* = 10.5 Hz, 1H), 3.78 (s, 3H), 3.62-3.59 (m, 2H), 3.58 (d,

J = 2.5 Hz, 1H), 3.38 (d, *J* = 15.0 Hz, 1H), 1.63 (s, 3H), 1.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.0, 163.9, 157.1, 132.7, 131.7, 129.8, 122.1, 114.6, 114.2, 59.5, 58.2, 55.8, 55.5, 51.3, 41.8, 20.8, 20.1, 16.3; HRMS *m/z* (ESI) calcd for $C_{22}H_{27}INO_5S$ ($[M+H]^+$) 544.0649, found 544.0655.

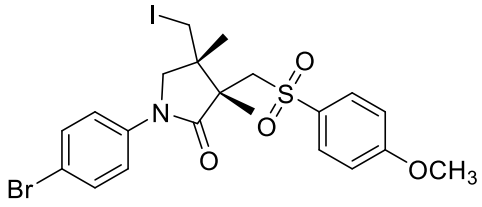


4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-(*p*-tolyl)pyrrolidin-2-one (4r), colorless oil (0.0885 g, 84% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.85 (d, *J* = 8.5 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 8.5 Hz, 2H), 3.92 (d, *J* = 10.0 Hz, 1H), 3.87 (s, 3H), 3.84 (d, *J* = 20.5 Hz, 1H), 3.69-3.52 (m, 3H), 3.38 (d, *J* = 15.0 Hz, 1H), 2.32 (s, 3H), 1.63 (s, 3H), 1.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.2, 163.9, 136.1, 135.0, 132.8, 129.8, 129.5, 120.4, 114.6, 59.5, 57.9, 55.8, 51.4, 41.8, 20.9, 20.8, 20.1, 16.3; HRMS *m/z* (ESI) calcd for $C_{22}H_{27}INO_4S$ ($[M+H]^+$) 528.0700, found 528.0704.

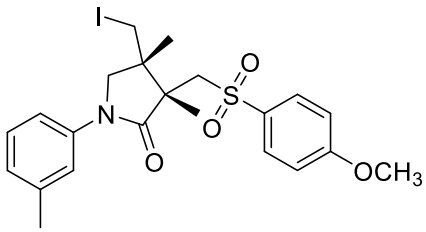


1-(4-Fluorophenyl)-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4s), yellow oil (0.0839 g, 79% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.87-7.84 (m, 2H), 7.52-7.49 (m, 2H), 7.07-7.01 (m, 4H), 3.91 (d, *J* = 7.5 Hz, 1H), 3.88 (s, 3H), 3.86 (d, *J* = 10.5 Hz, 1H), 3.62-3.59 (m, 3H), 3.39 (d, *J* = 15.0 Hz, 1H), 1.63 (s, 3H), 1.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.3, 163.9, 159.9 (d, J_{C-F} = 243.6 Hz), 134.7 (d, J_{C-F} = 2.9 Hz), 132.6, 129.8, 122.1 (d, J_{C-F} = 7.9 Hz), 115.7 (d, J_{C-F} = 22.4 Hz), 114.6, 59.5,

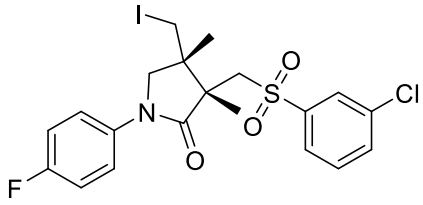
58.1, 55.8, 51.4, 41.8, 21.0, 20.1, 15.9; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -116.5; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}FINO_4S$ ($[M+H]^+$) 532.0449, found 532.0453.



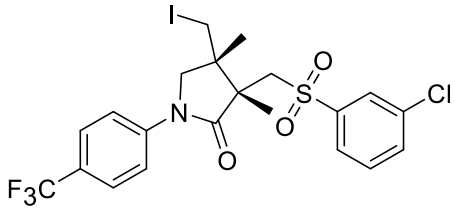
1-(4-Bromophenyl)-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4t), colorless oil (0.0886 g, 75% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.86-7.83 (m, 2H), 7.49-7.44 (m, 4H), 7.02-7.00 (m, 2H), 3.89 (d, *J* = 3.0 Hz, 1H), 3.88 (s, 3H), 3.85 (d, *J* = 10.5 Hz, 1H), 3.61-3.57 (m, 3H), 3.39 (d, *J* = 15.0 Hz, 1H), 1.62 (s, 3H), 1.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.5, 163.9, 137.7, 132.6, 132.0, 129.8, 121.6, 118.1, 114.6, 59.4, 57.7, 55.8, 51.5, 41.6, 21.0, 20.2, 15.8; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}BrINO_4S$ ($[M+H]^+$) 591.9649, found 591.9653.



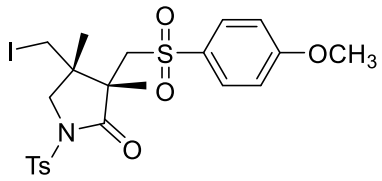
4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-(*m*-tolyl)pyrrolidin-2-one (4u), colorless oil (0.0864 g, 82% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.87-7.83 (m, 2H), 7.37 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 8.0 Hz, 1H), 7.01-6.97 (m, 3H), 3.92 (d, *J* = 10.0 Hz, 1H), 3.88 (d, *J* = 2.5 Hz, 1H), 3.86 (s, 3H), 3.63-3.56 (m, 3H), 3.39 (d, *J* = 15.0 Hz, 1H), 2.35 (s, 3H), 1.63 (s, 3H), 1.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.4, 163.9, 138.9, 138.6, 132.8, 129.8, 128.8, 126.1, 121.0, 117.4, 114.6, 59.5, 57.9, 55.8, 51.5, 41.7, 21.6, 20.9, 20.1, 16.2; HRMS *m/z* (ESI) calcd for $C_{22}H_{27}INO_4S$ ($[M+H]^+$) 528.0700, found 528.0704.



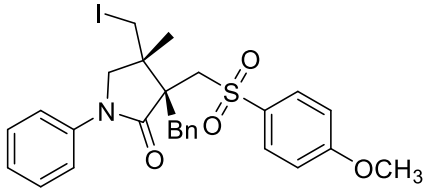
3-(((3-Chlorophenyl)sulfonyl)methyl)-1-(4-fluorophenyl)-4-(iodomethyl)-3,4-dimethylpyrrolidin-2-one (4v), yellow oil (0.0760 g, 71% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.93 (t, *J* = 2.0 Hz, 1H), 7.84-7,82 (m, 1H), 7.65-7.63 (m, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.52-7.49 (m, 2H), 7.08-7.05 (m, 2H), 3.87 (d, *J* = 10.5 Hz, 2H), 3.64-3.57 (m, 3H), 3.41 (d, *J* = 15.0 Hz, 1H), 1.66 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.0, 160.0 (d, J_{C-F} =243.9 Hz), 142.5, 135.8, 134.5 (d, J_{C-F} = 2.9 Hz), 134.2, 130.8, 127.7, 125.7, 122.2 (d, J_{C-F} = 8.0 Hz), 115.8 (d, J_{C-F} = 22.4 Hz), 59.2, 58.1, 51.5, 41.8, 20.9, 20.2, 15.5; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -116.3; HRMS *m/z* (ESI) calcd for $C_{20}H_{21}ClFINO_3S$ ($[M+H]^+$) 535.9954, found 535.9950.



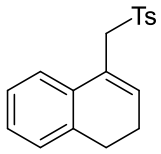
3-(((3-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-(4-(trifluoromethyl)phenyl)pyrrolidin-2-one (4w), yellow oil (0.0725 g, 62% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.94 (t, *J* = 2.0 Hz, 1H), 7.85-7.83 (m, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.66-7.63 (m, 3H), 7.54 (t, *J* = 8.0 Hz, 1H), 3.94 (d, *J* = 10.5 Hz, 1H), 3.86 (d, *J* = 10.0 Hz, 1H), 3.67 (d, *J* = 10.5 Hz, 1H), 3.63 (d, *J* = 15.0 Hz, 1H), 3.58 (d, *J* = 10.0 Hz, 1H), 3.42 (d, *J* = 15.0 Hz, 1H), 1.67 (s, 3H), 1.60 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.6, 142.4, 141.4, 135.8, 134.3, 130.9, 127.7, 126.3 (*q*, J_{C-F} = 2.7 Hz), 125.7, 125.0, 122.9, 119.7, 59.1, 57.5, 51.7, 41.6, 21.0, 20.3, 15.2; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -62.2; HRMS *m/z* (ESI) calcd for $C_{21}H_{21}ClF_3INO_3S$ ($[M+H]^+$) 585.9922, found 585,9926.



4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-tosylpyrrolidin-2-one (4x), colorless oil (0.0733 g, 62% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.5 Hz, 2H), 7.81-7.79 (m, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.04-7.02 (m, 2H), 4.47 (d, *J* = 11.0 Hz, 1H), 3.89 (s, 3H), 3.73 (d, *J* = 11.0 Hz, 1H), 3.21-3.17 (m, 2H), 3.11 (d, *J* = 14.0 Hz, 1H), 3.01 (d, *J* = 11.0 Hz, 1H), 2.45 (s, 3H), 1.38 (s, 3H), 1.13 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 172.7, 164.1, 145.6, 134.6, 133.8, 132.4, 129.8, 128.3, 114.8, 59.1, 55.8, 53.9, 51.3, 42.5, 21.8, 21.0, 18.8, 6.5; HRMS *m/z* (ESI) calcd for $C_{22}H_{27}INO_6S_2$ ($[M+H]^+$) 592.0319, found 592.0323.



3-Benzyl-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-4-methyl-1-phenylpyrrolidin-2-one (4y), colorless oil (0.0931 g, 79% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.88 (d, *J* = 8.0 Hz, 2H), 7.51-7.45 (m, 2H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.29 (s, 2H), 7.23 (t, *J* = 10.0 Hz, 4H), 7.03 (d, *J* = 7.5 Hz, 2H), 4.28 (t, *J* = 13.0 Hz, 1H), 3.89 (s, 3H), 3.82-3.77 (m, 1H), 3.68 (t, *J* = 22.0 Hz, 1H), 3.59-3.52 (m, 3H), 3.27-3.16 (m, 2H), 1.59 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 172.7, 164.0, 135.9, 132.7, 130.9, 130.6, 129.7, 129.1, 128.2, 127.3, 125.1, 120.0, 114.6, 57.4, 56.8, 56.3, 55.8, 43.2, 37.7, 19.2, 17.2; HRMS *m/z* (ESI) calcd for $C_{27}H_{29}INO_4S$ ($[M+H]^+$) 590.0856, found 590.0852.



4-(Tosylmethyl)-1,2-dihydronaphthalene (5a),[1] (0.0423 g, 71% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.78 (d, *J* = 8.5 Hz, 2H), 7.29 (t,

J = 4.0 Hz, 5H), 7.25-7.22 (m, 1H), 5.58-5.54 (m, 1H), 4.11 (t, *J* = 7.0 Hz, 2H), 2.58-2.54 (m, 2H), 2.42 (s, 3H), 1.98 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.7, 143.1, 138.5, 133.2, 129.8, 128.2, 127.9, 127.0, 125.6, 121.3, 69.6, 28.5, 21.6, 16.0.

(C) Reference

[1] Chen, P.; Zhou, Q.; Chen, Z.; Liu, Y.; Liang, Y.; Tang, K.; Liu, Y. Silver-promoted oxidative sulfonylation and ring-expansion of vinylcyclopropanes with sodium sulfinates leading to dihydronaphthalene derivatives. *Org. Biomol. Chem*. **2020**, *18*, 7345-7354.

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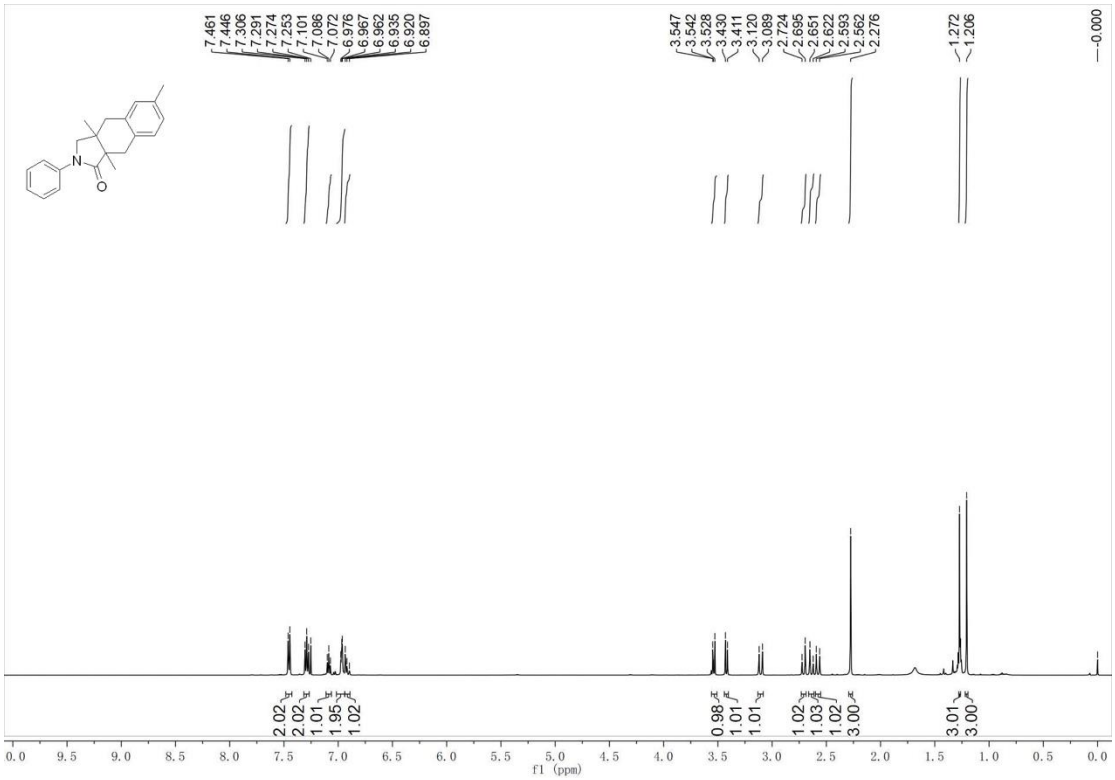
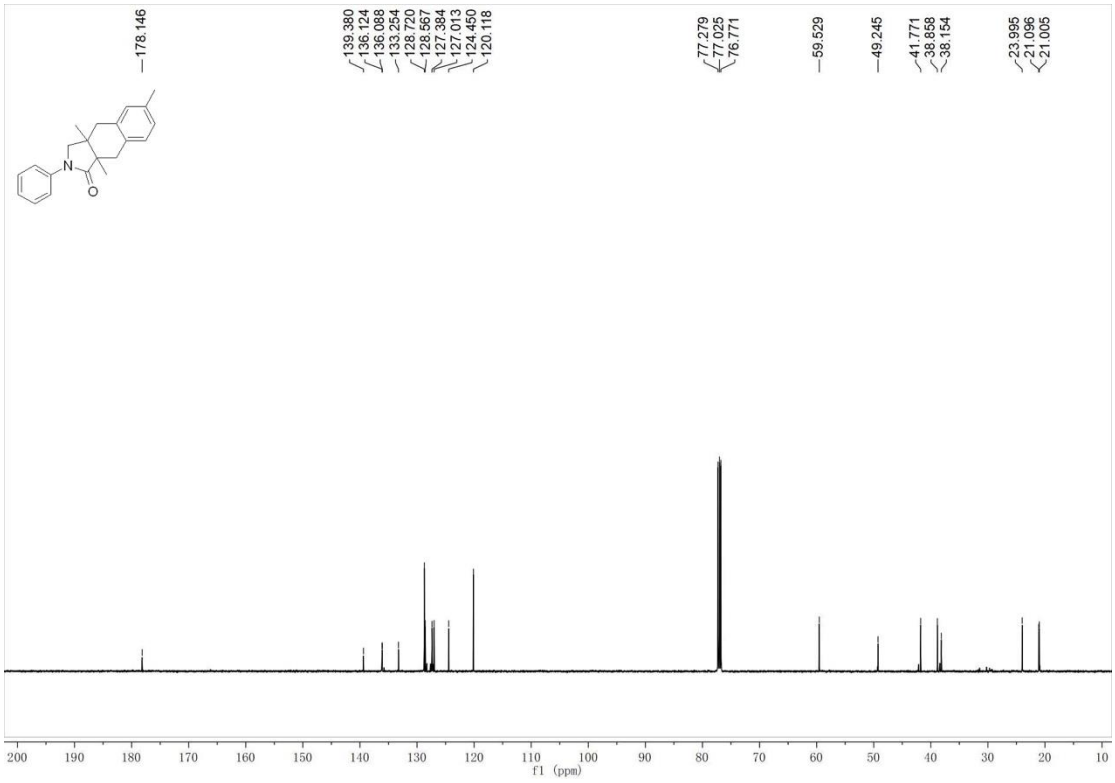
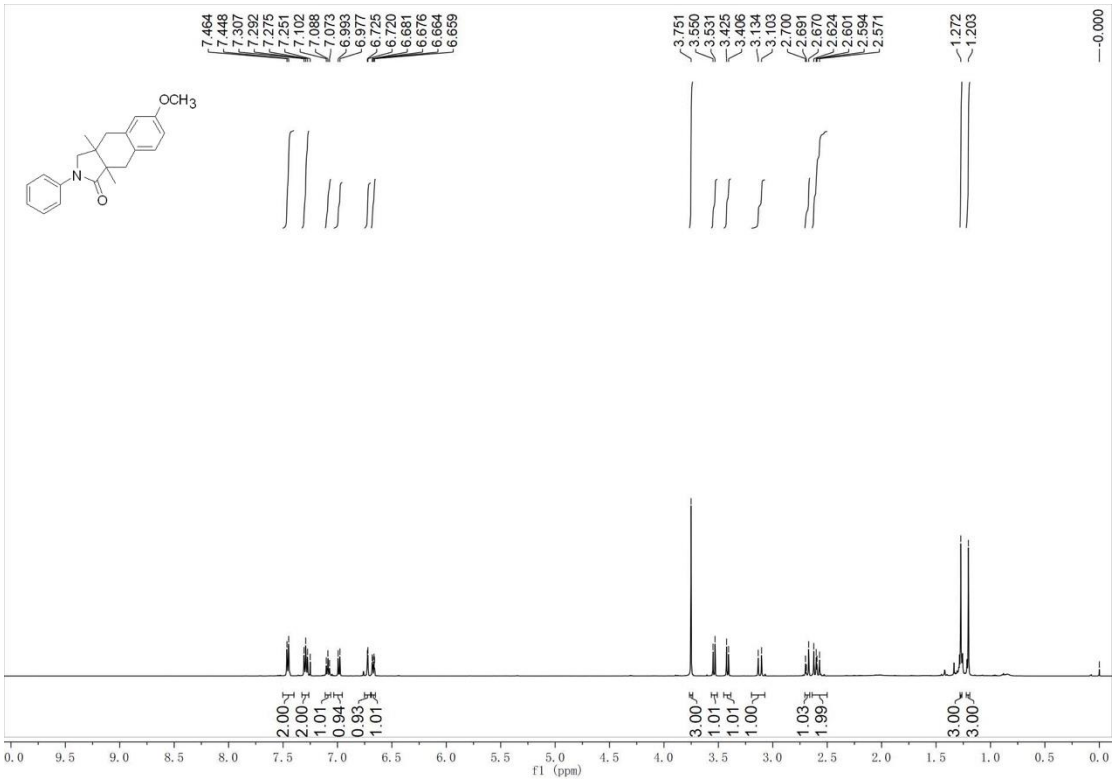
(D) Spectra

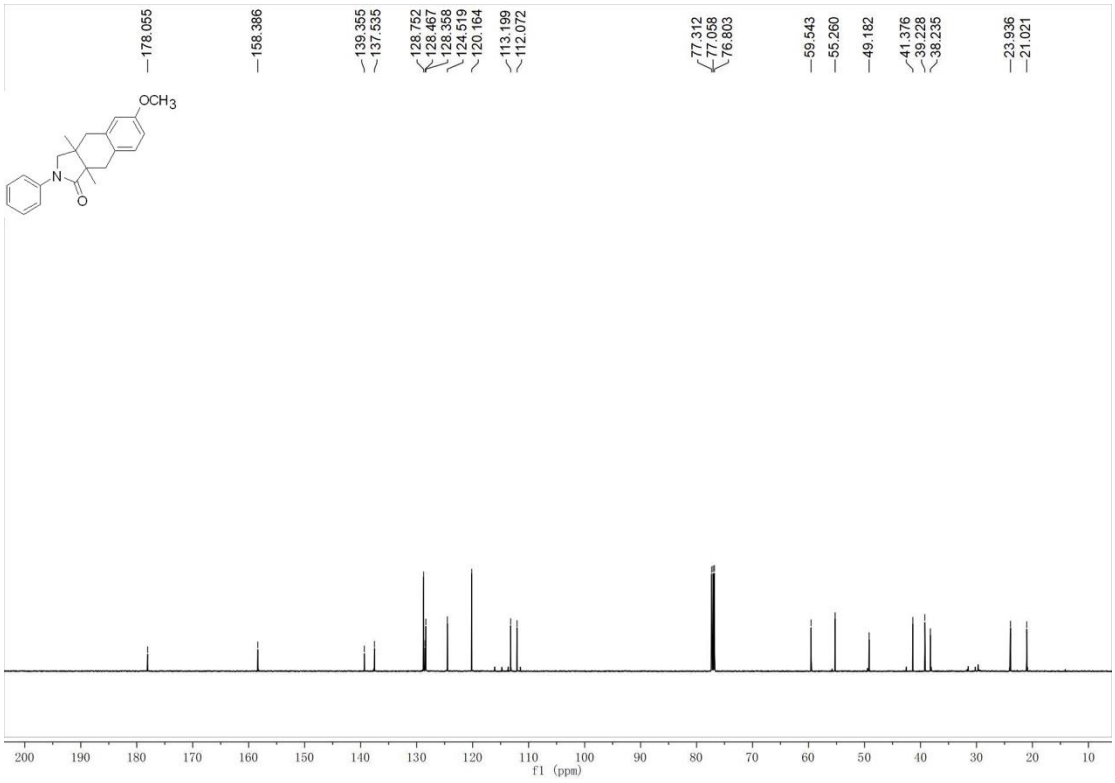
3a,6,9a-Trimethyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3a)



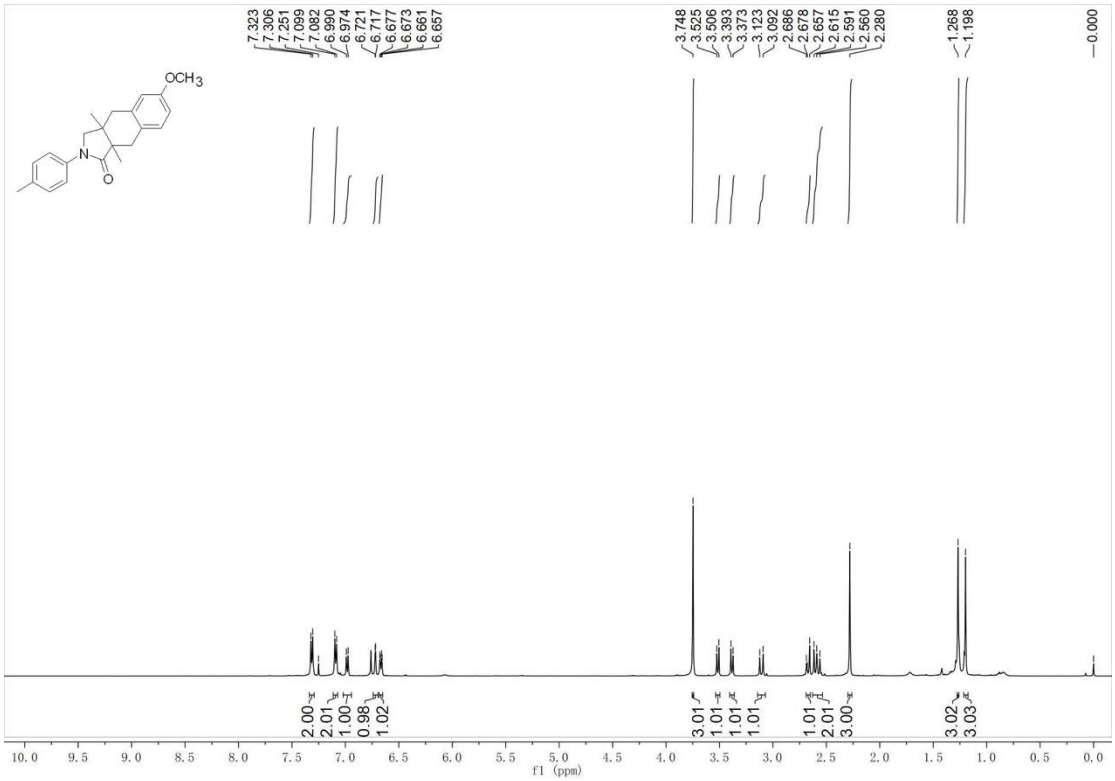


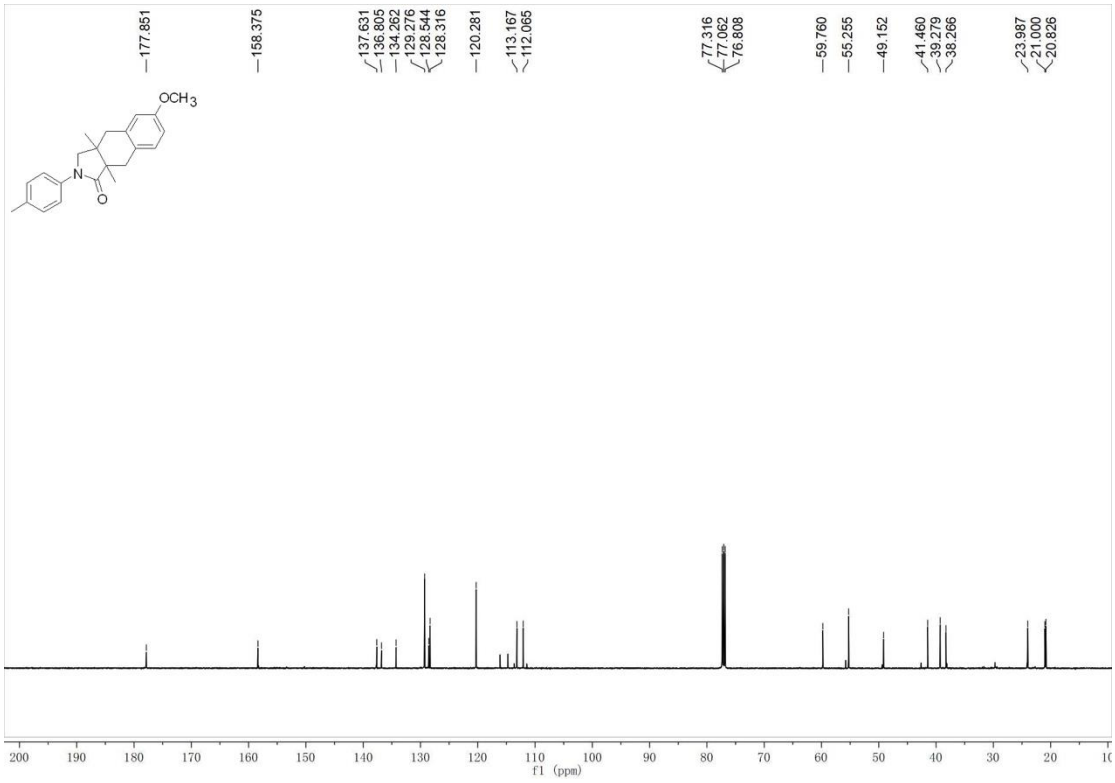
6-Methoxy-3a,9a-dimethyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3b)



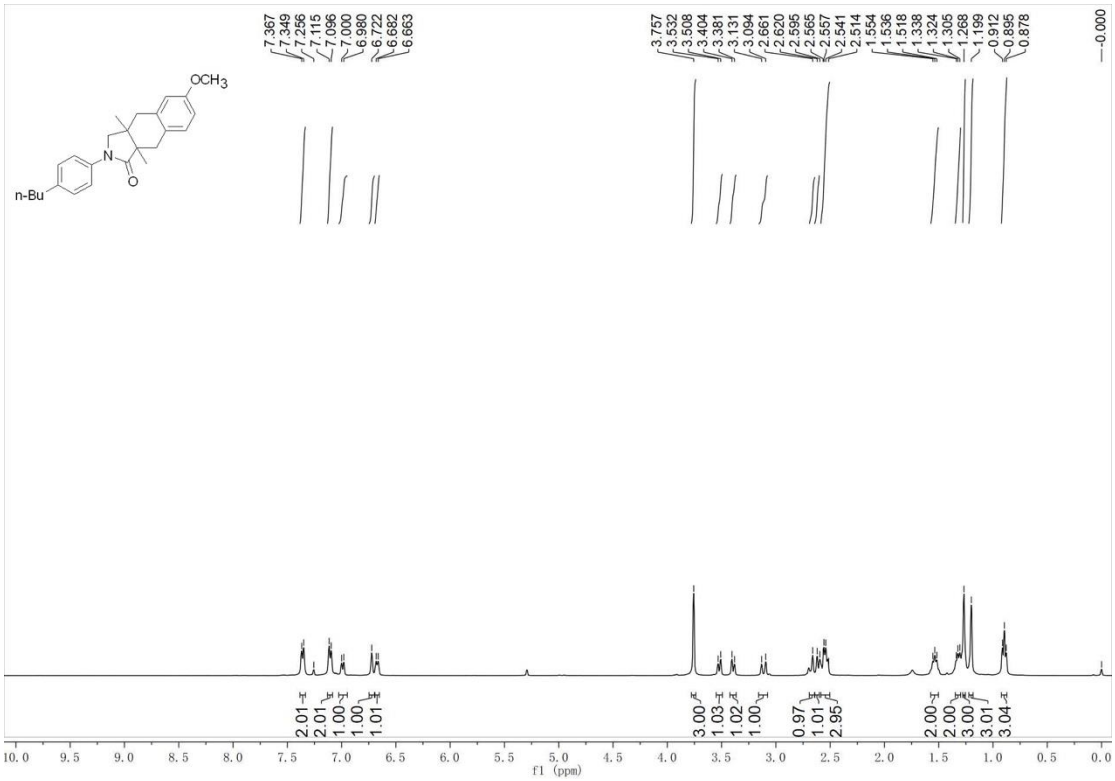


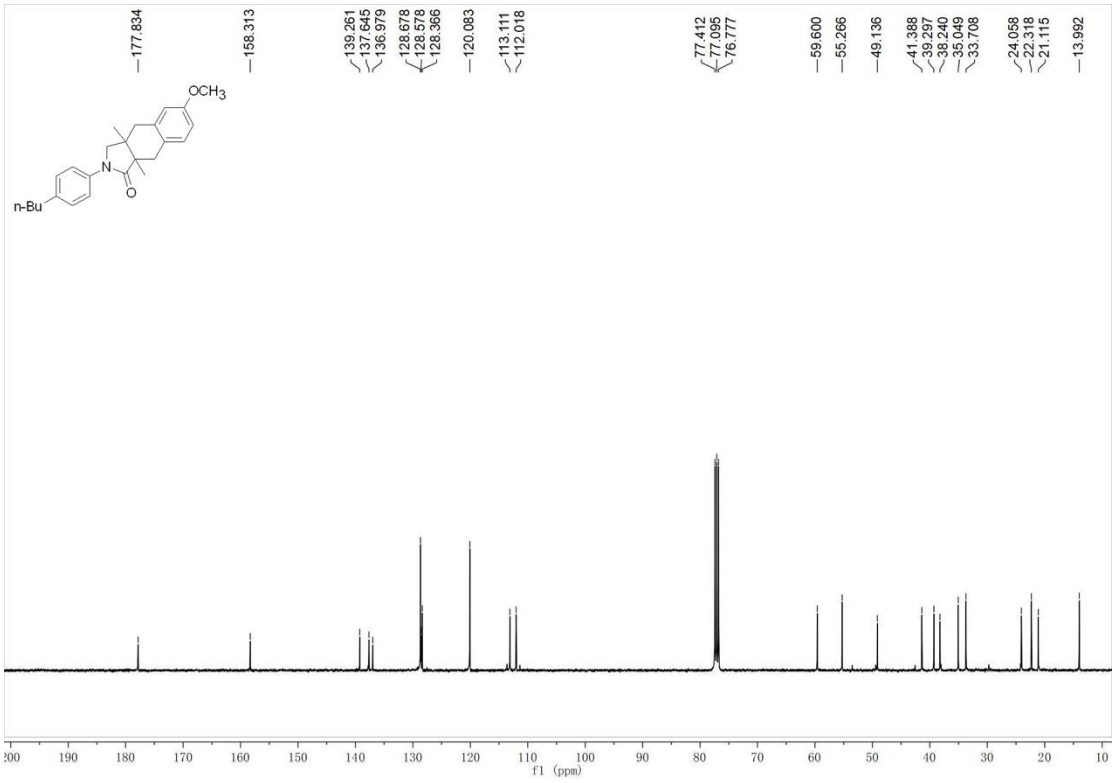
6-Methoxy-3a,9a-dimethyl-2-(*p*-tolyl)-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3c)



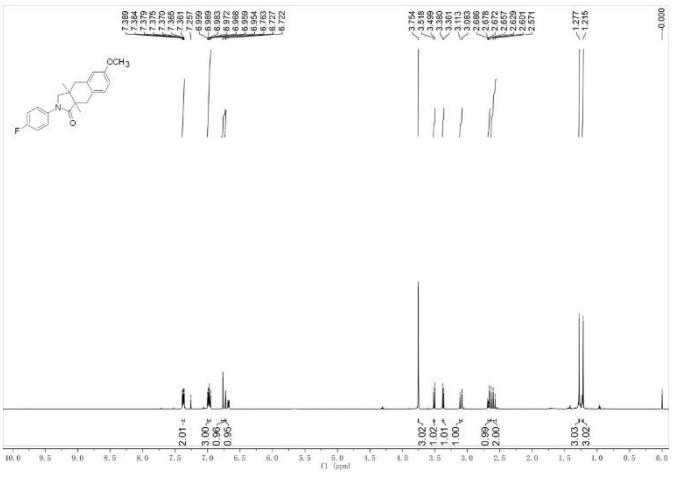


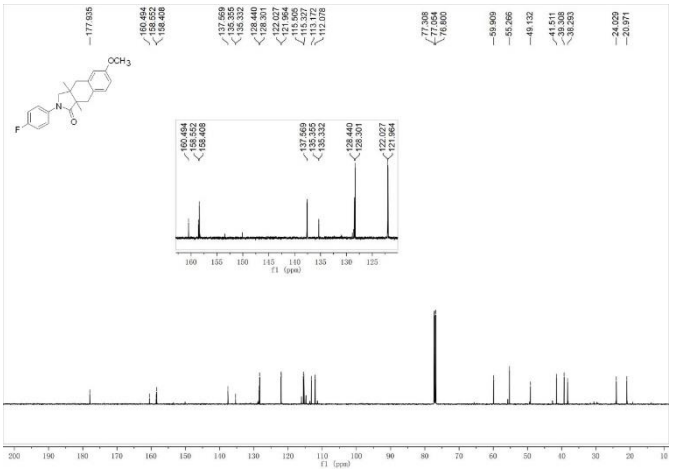
2-(4-Butylphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3d)

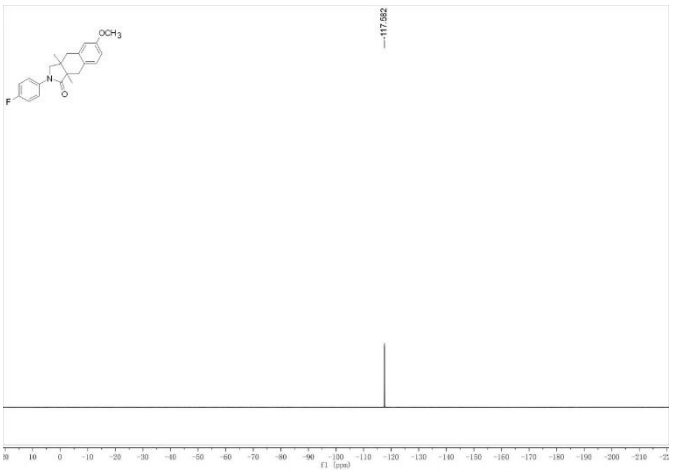




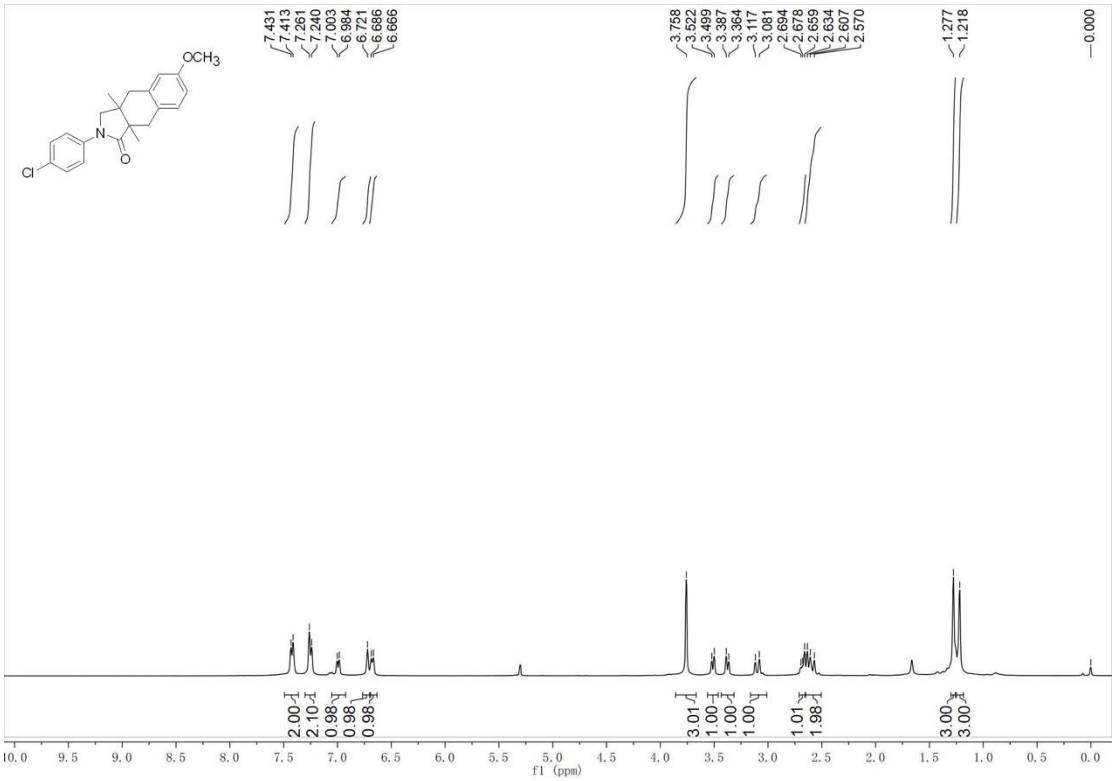
2-(4-Fluorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo [*f*]isoindol-1-one (3e)

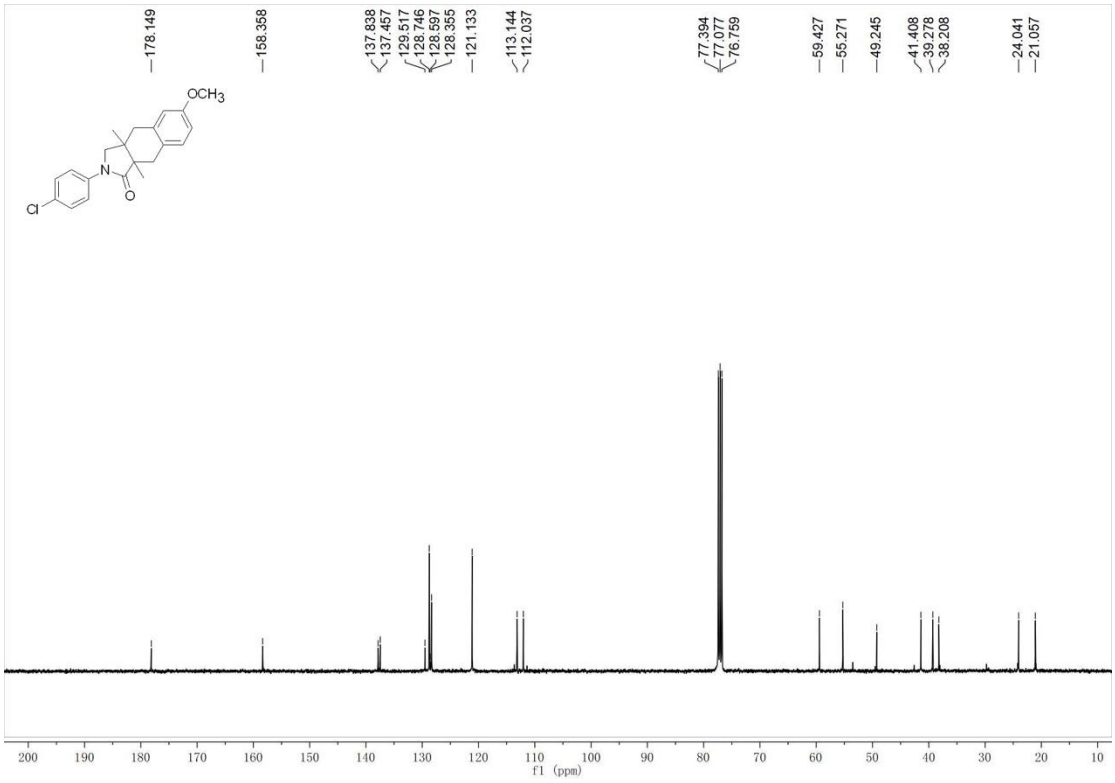




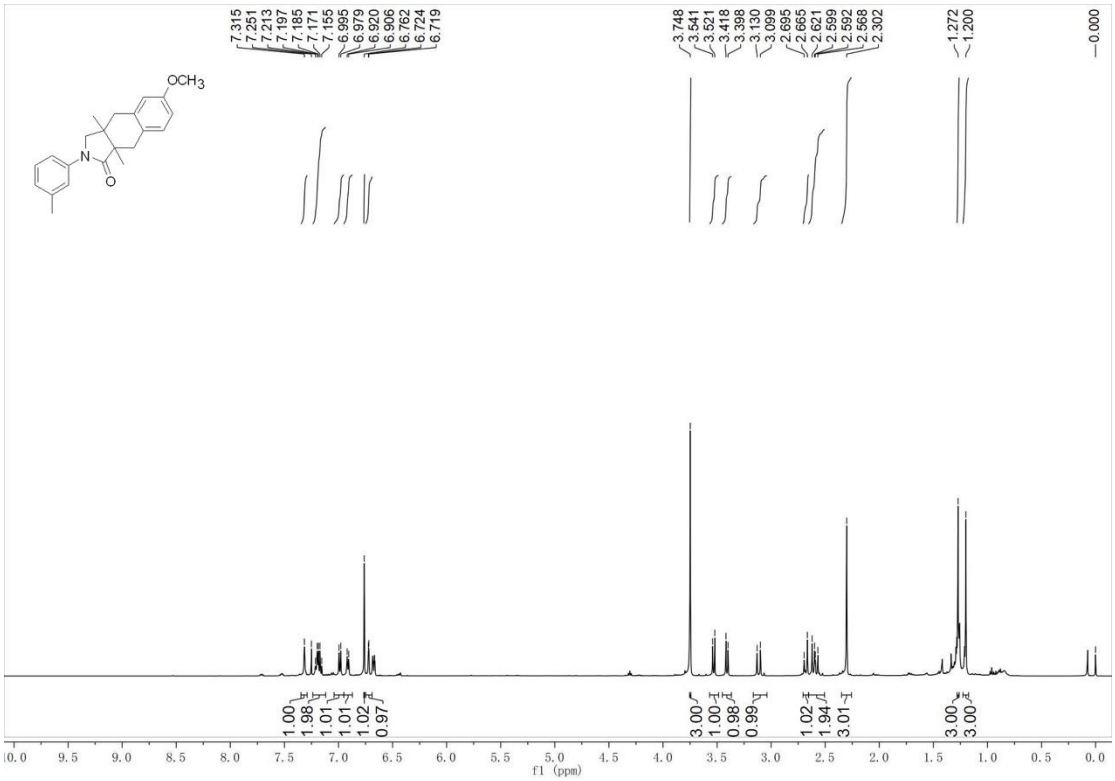


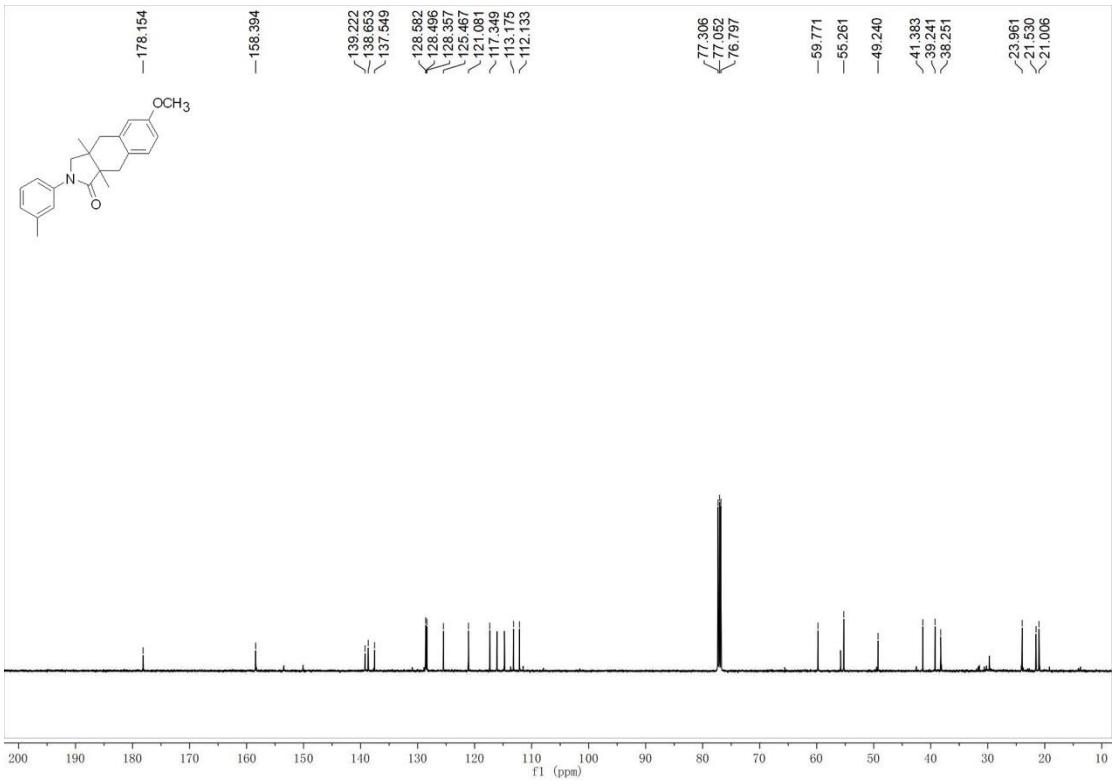
2-(4-Chlorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3f)



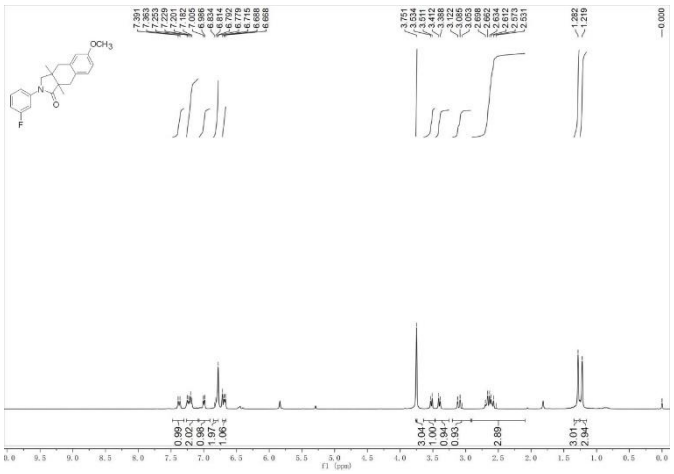


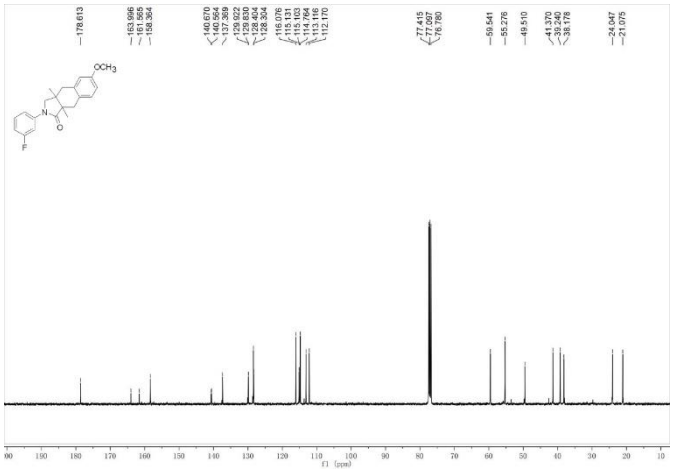
6-Methoxy-3a,9a-dimethyl-2-(*m*-tolyl)-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3g)

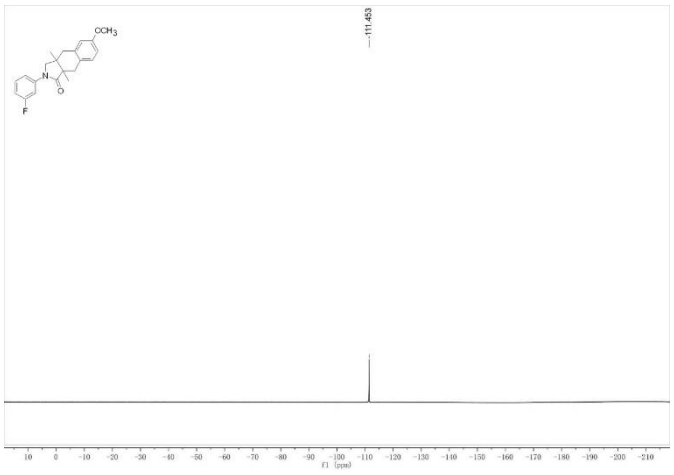




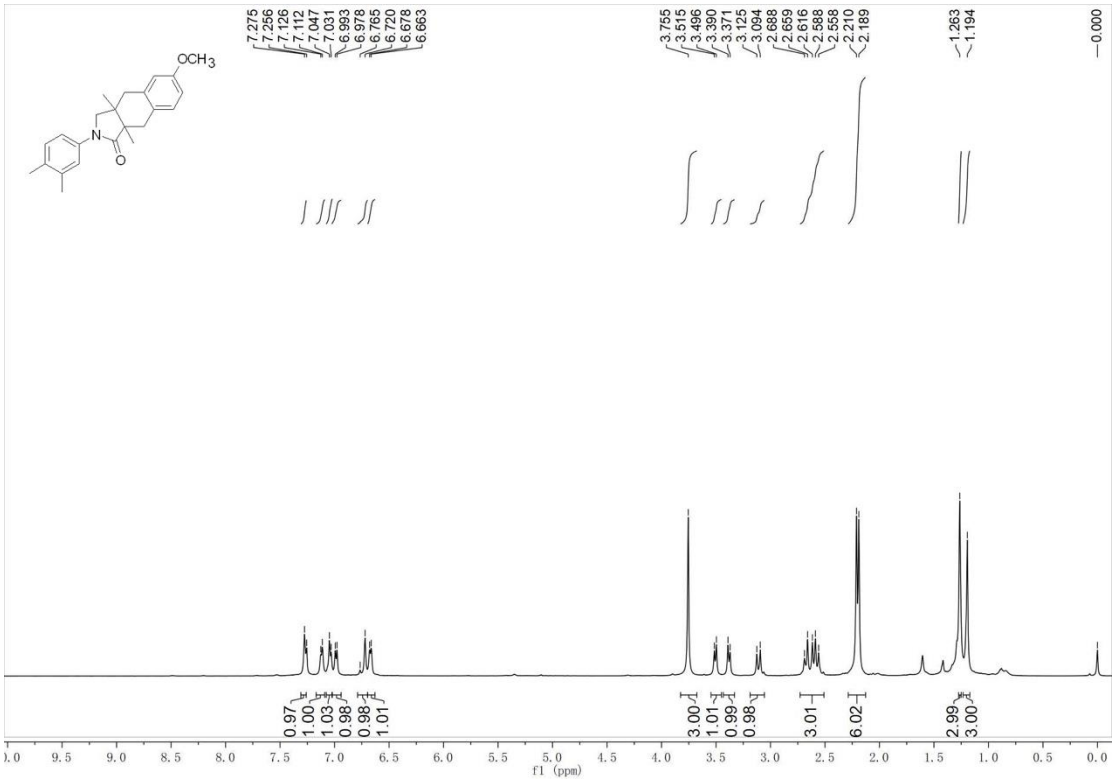
2-(3-Fluorophenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3h)

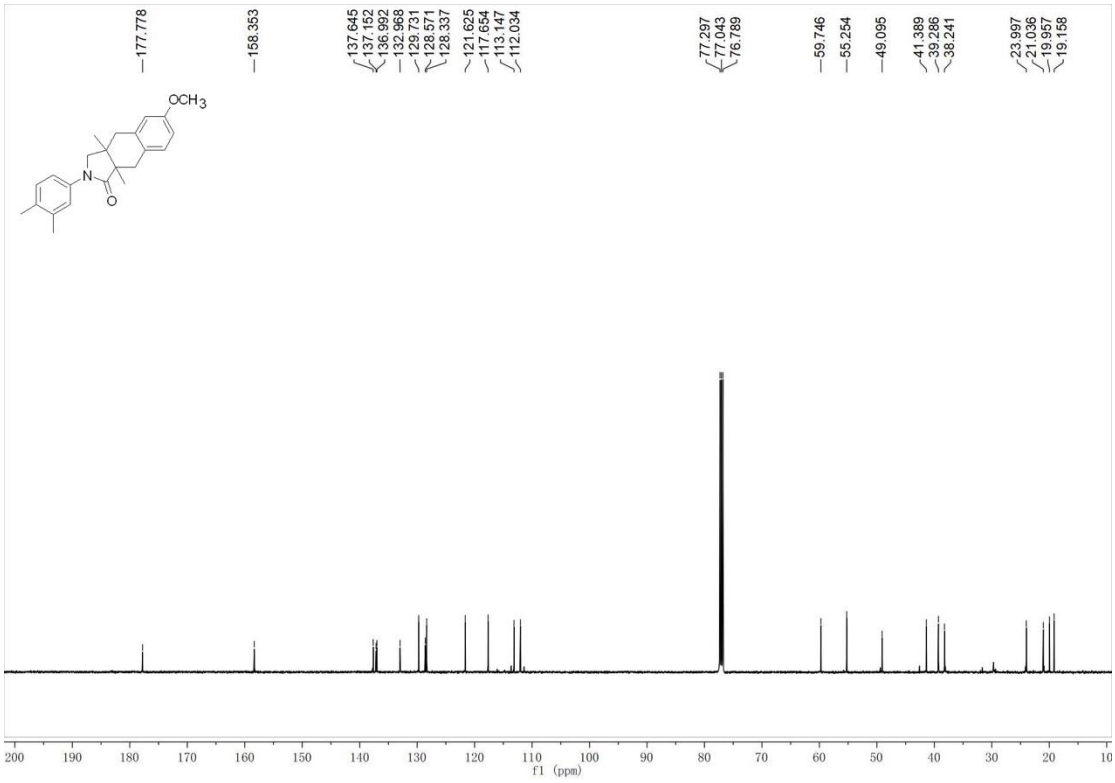




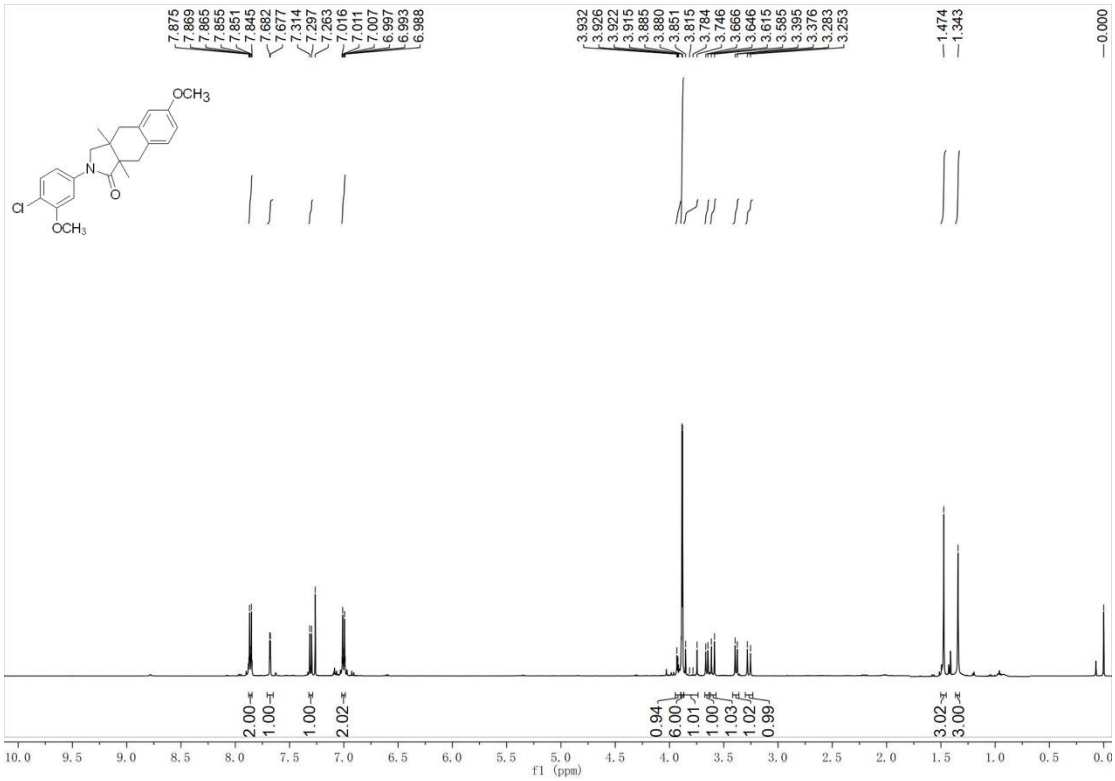


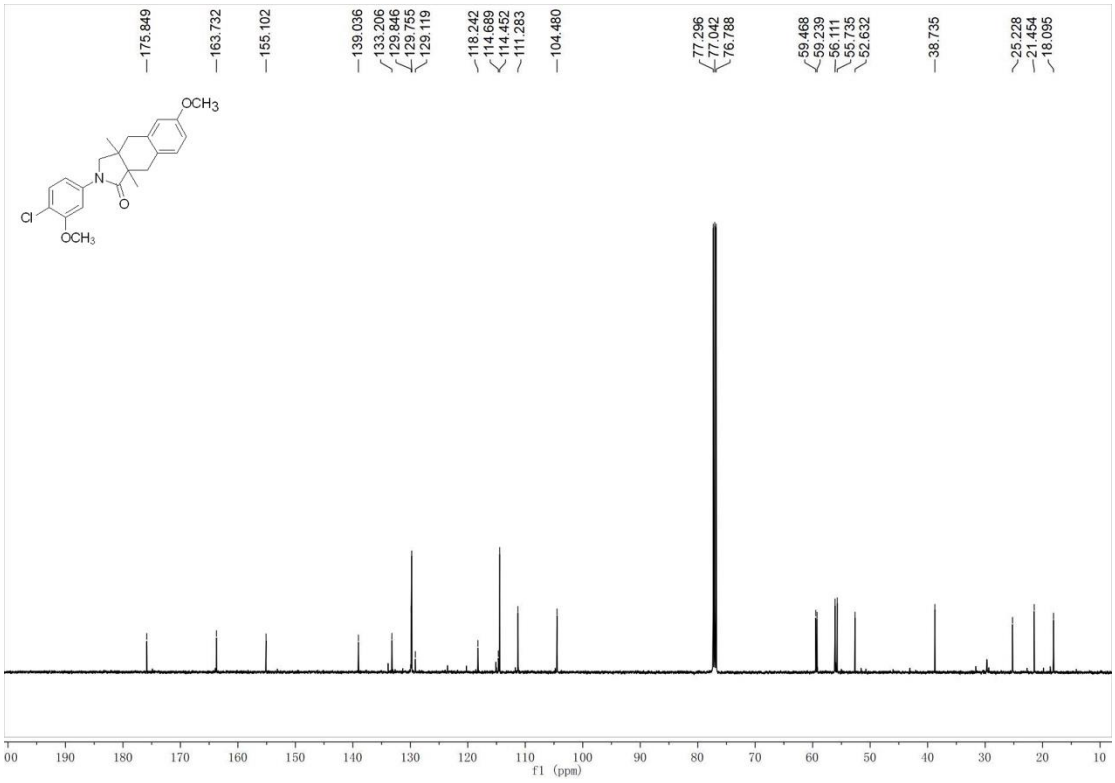
2-(3,4-Dimethylphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3i)



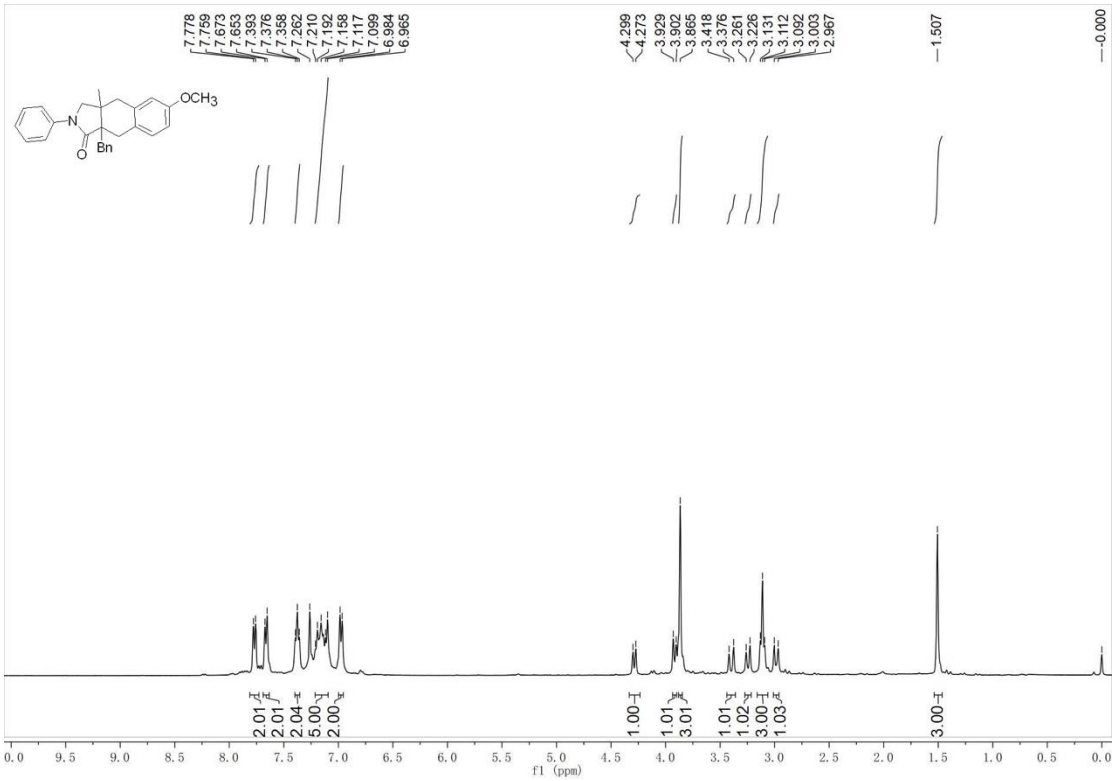


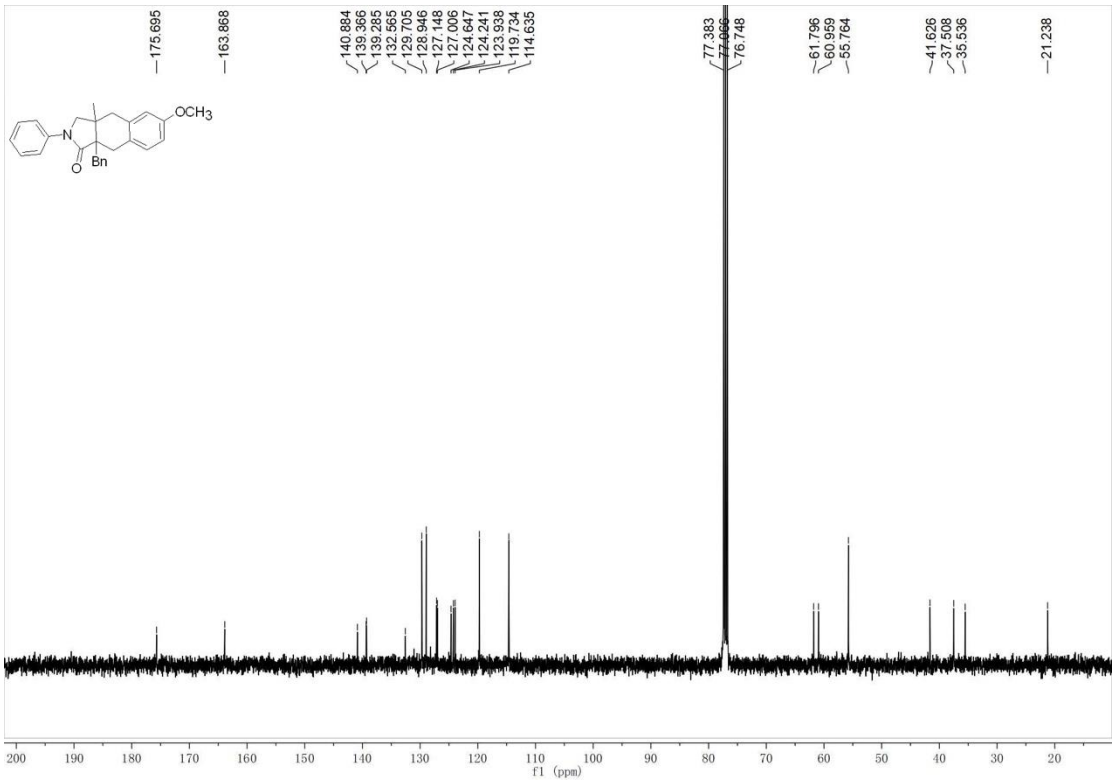
2-(4-Chloro-3-methoxyphenyl)-6-methoxy-3a,9a-dimethyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3j)



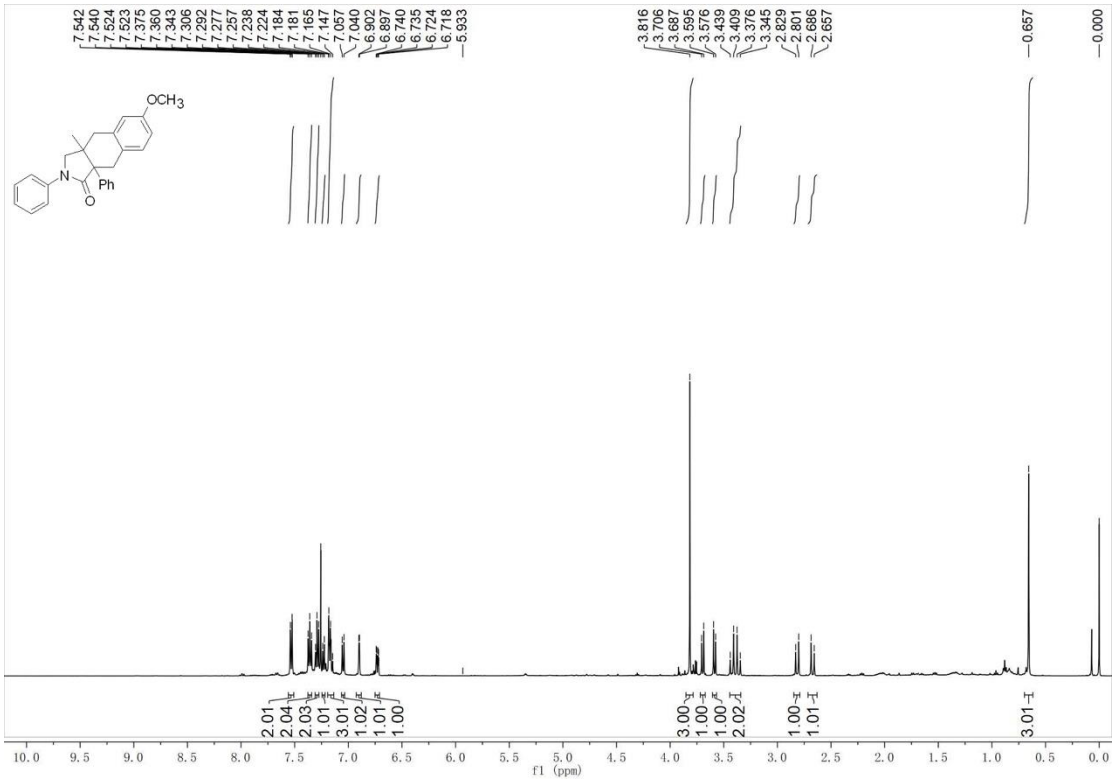


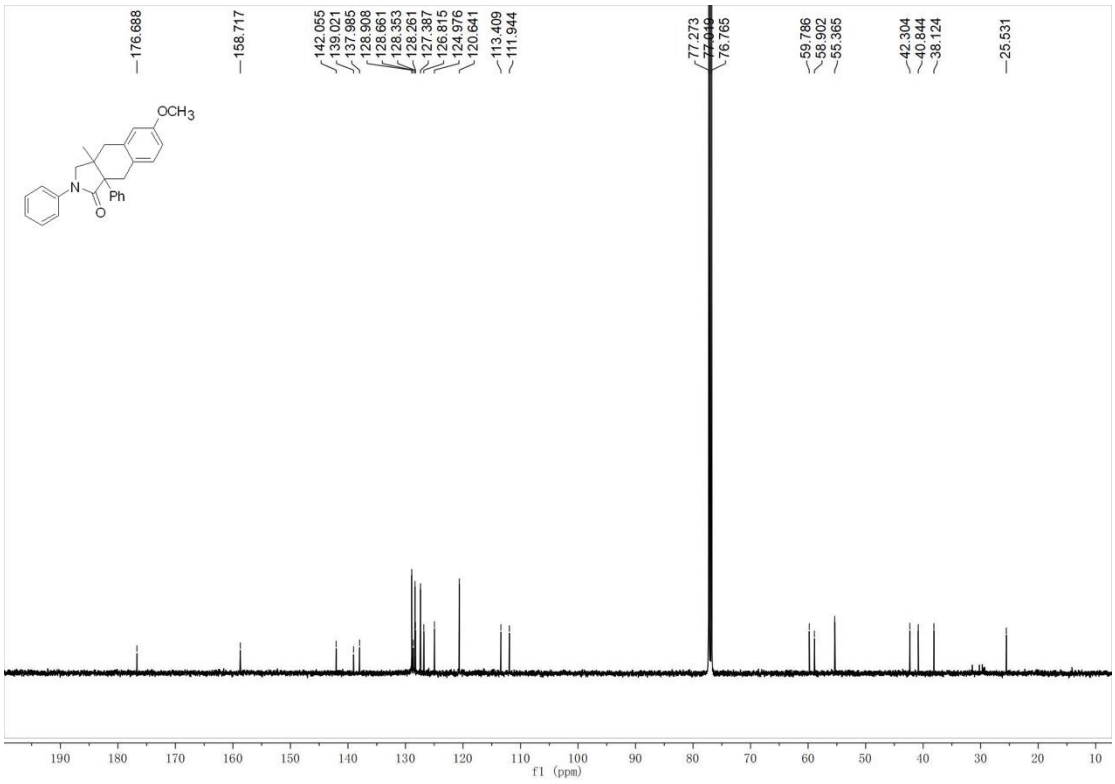
9a-Benzyl-6-methoxy-3a-methyl-2-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3k)



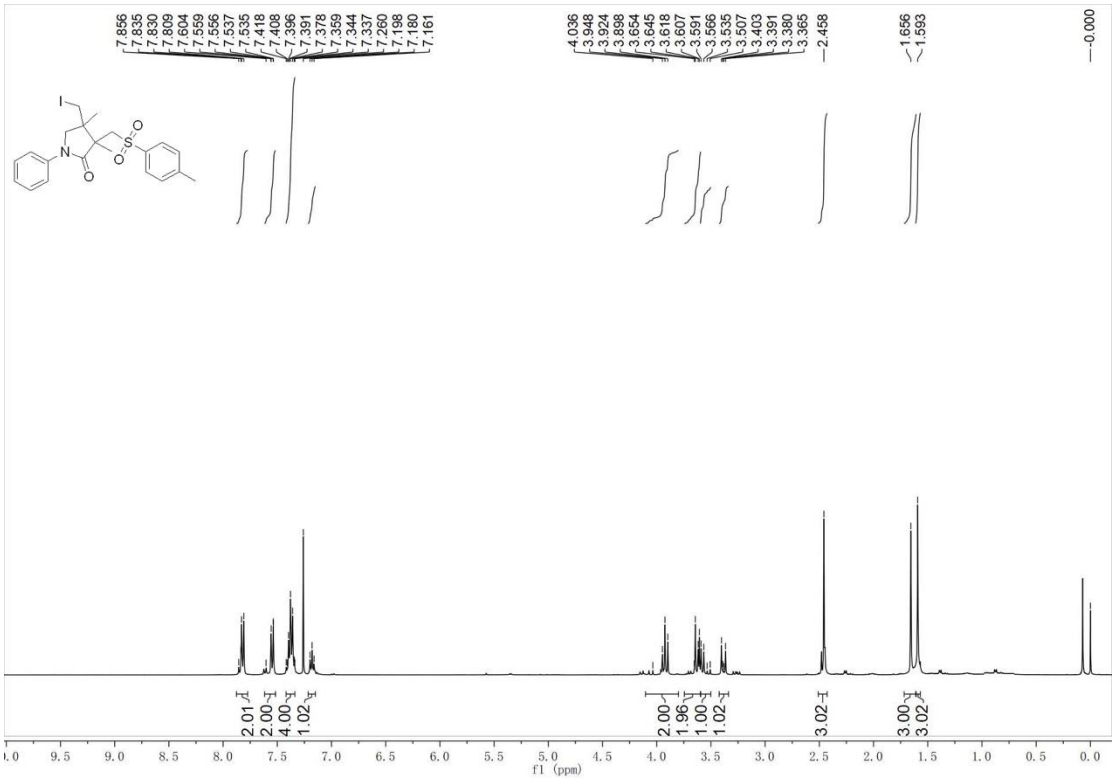


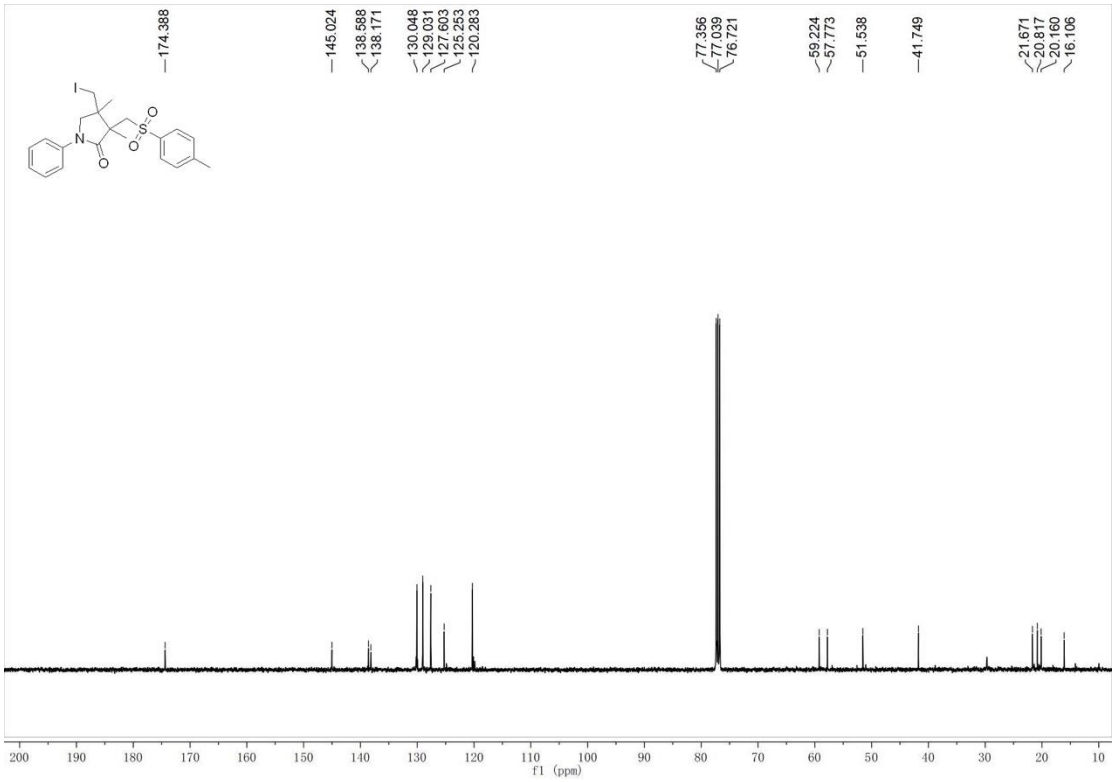
6-Methoxy-3a-methyl-2,9a-diphenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindol-1-one (3l)



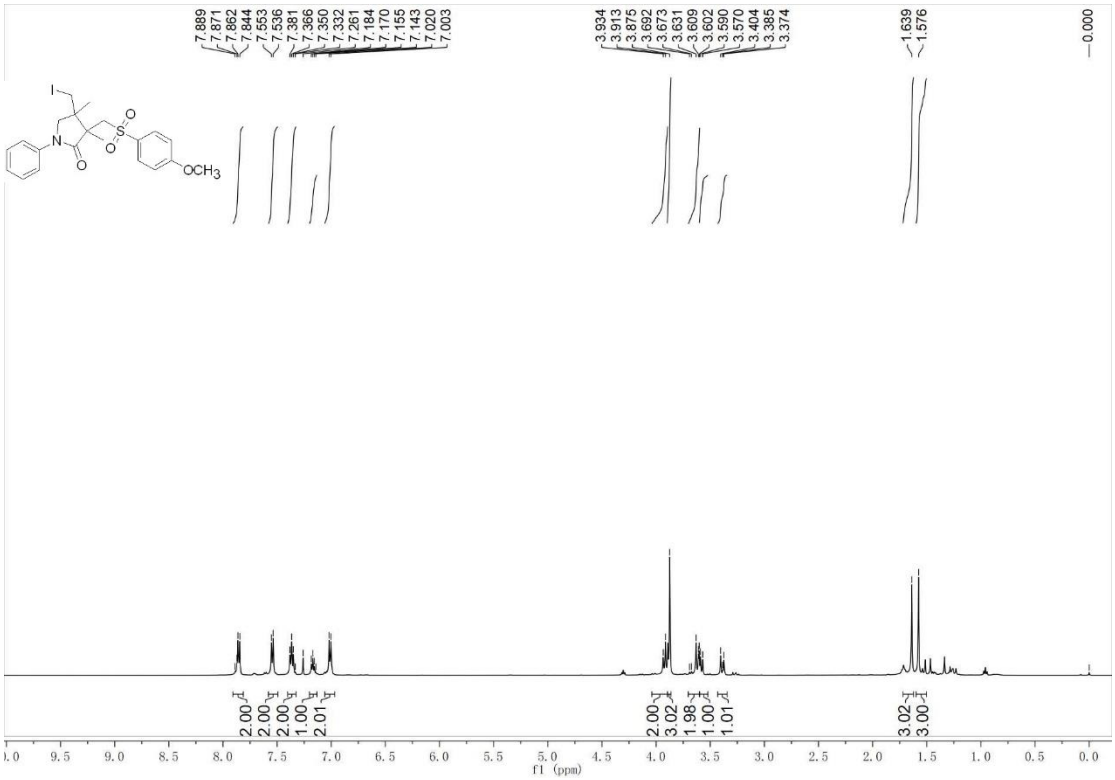


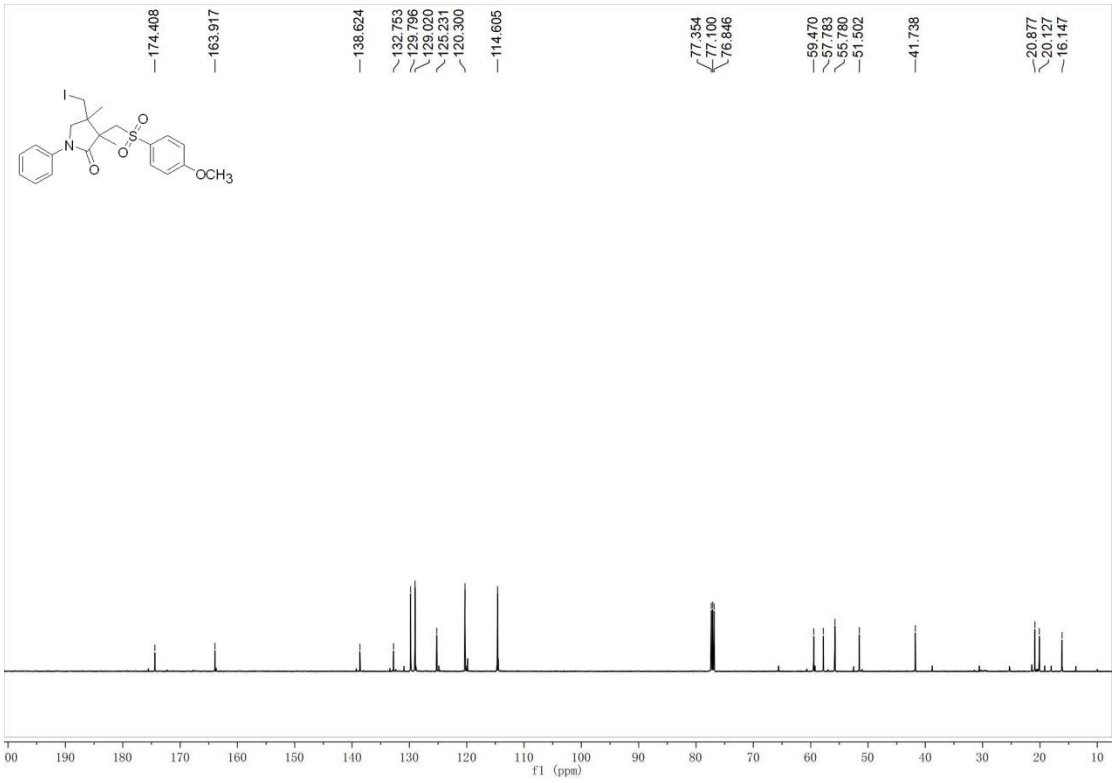
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-(tosylmethyl)pyrrolidin-2-one (4a)



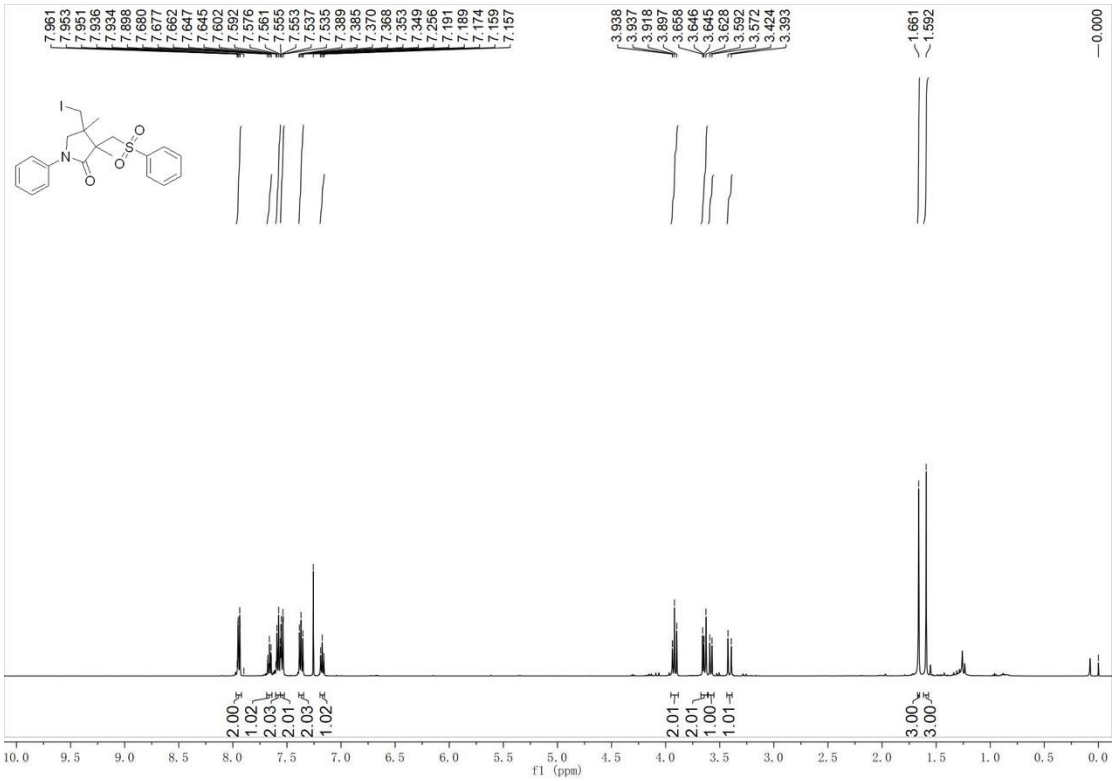


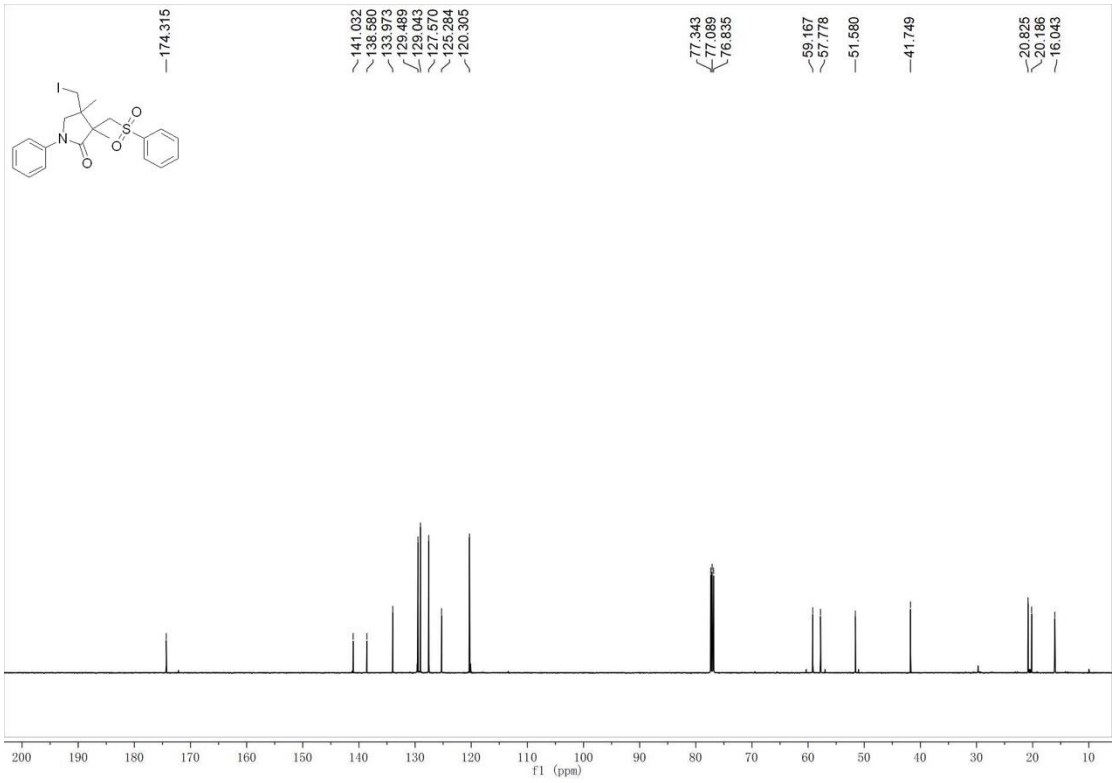
4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4b)



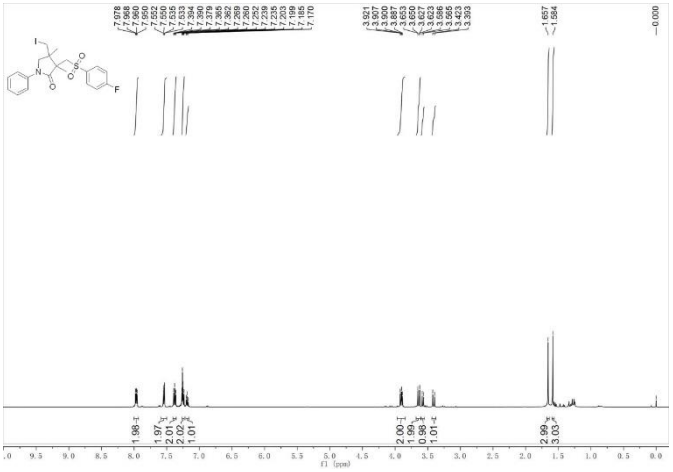


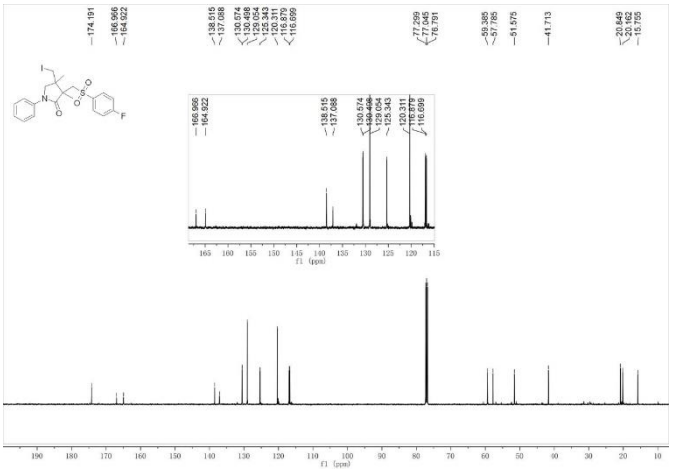
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((phenylsulfonyl)methyl)pyrrolidin-2-one (4c)

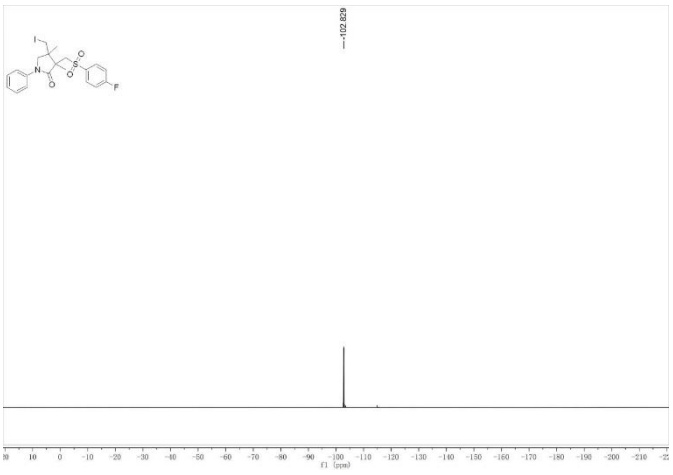




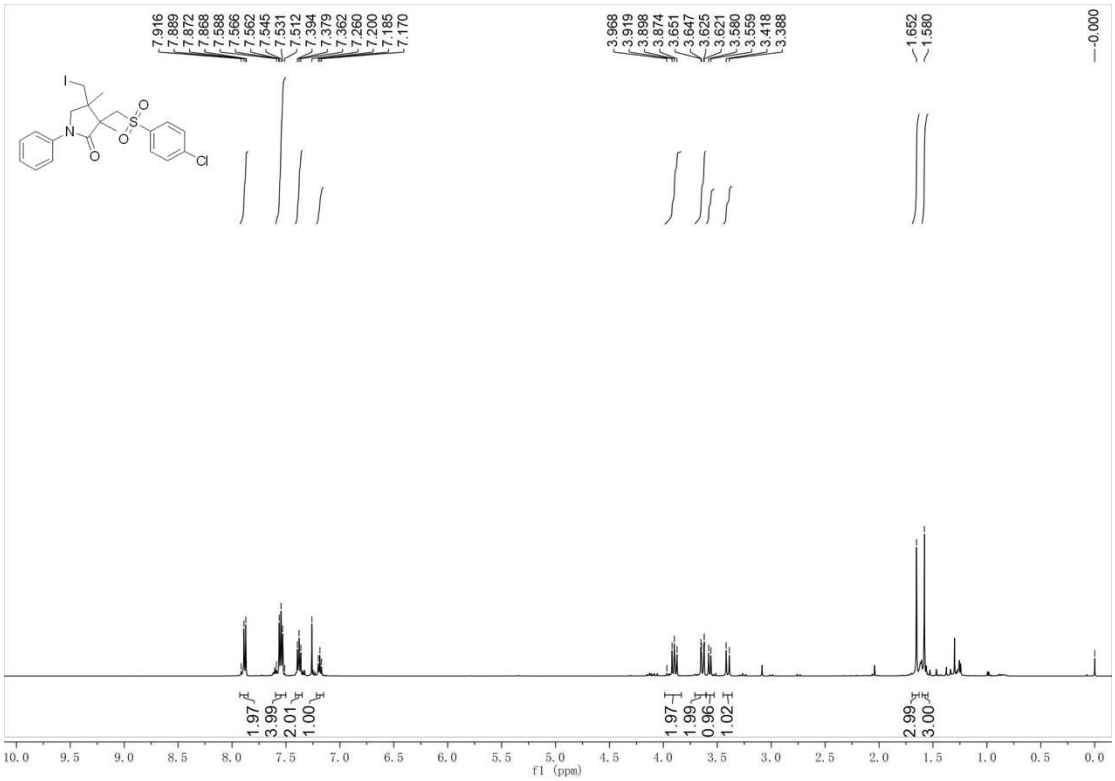
3-(((4-Fluorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4d)

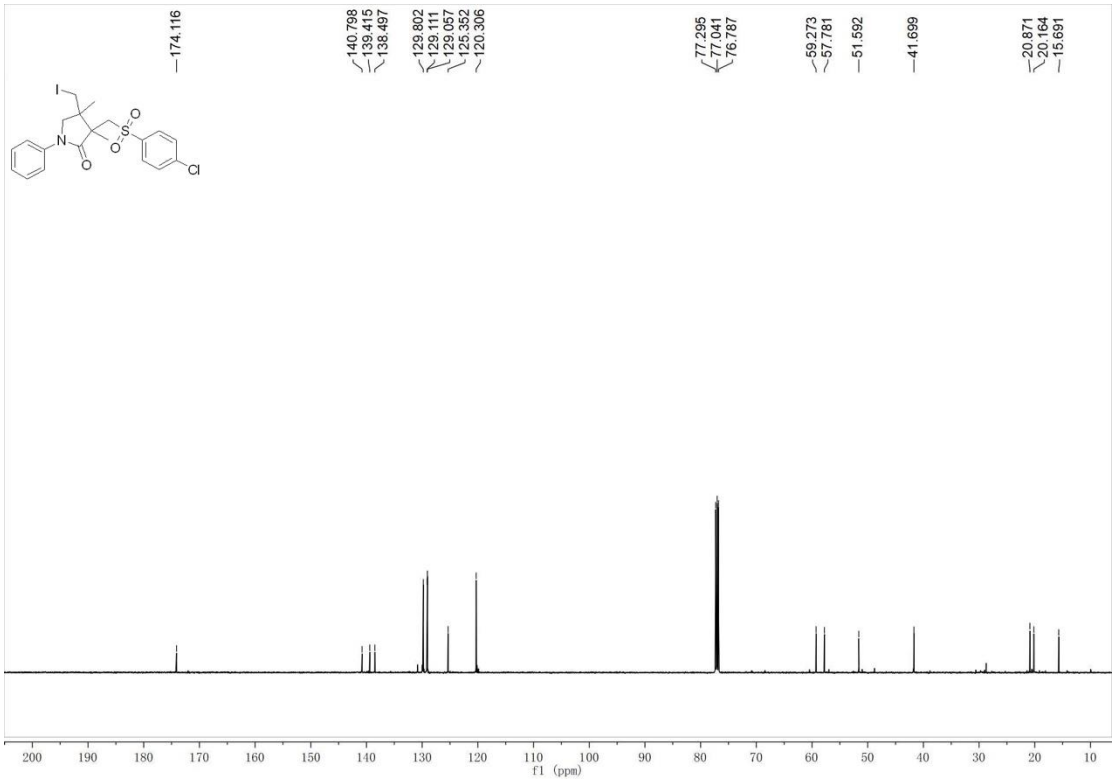




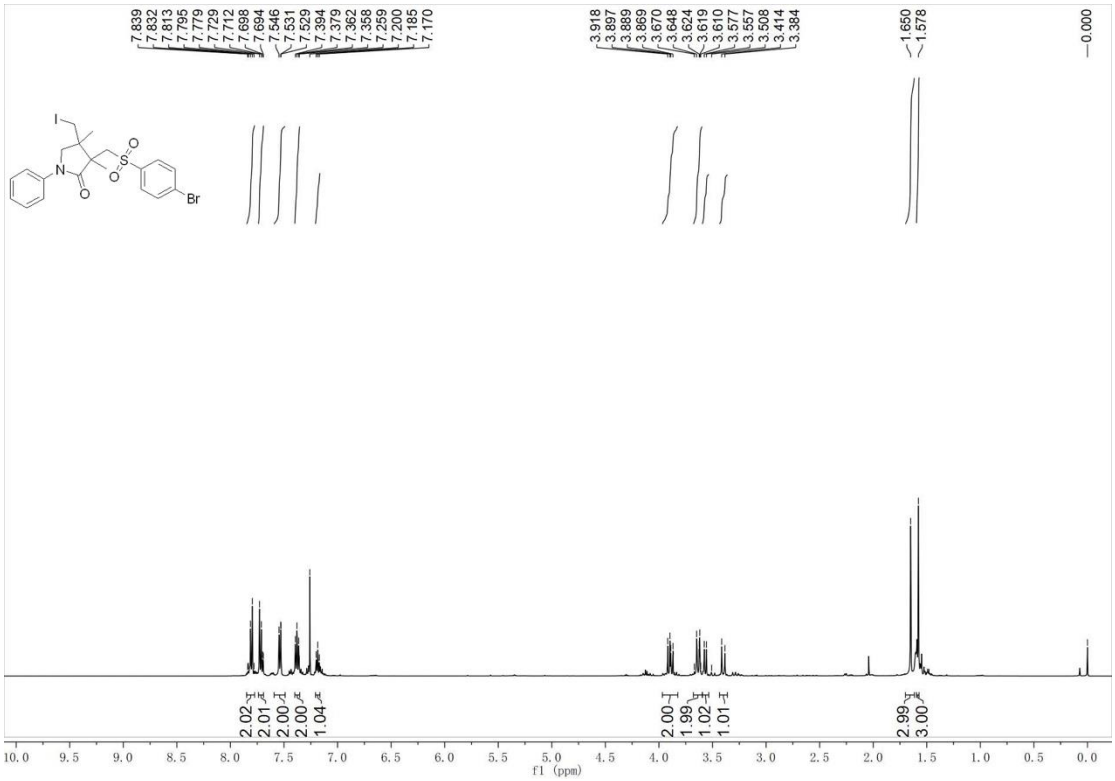


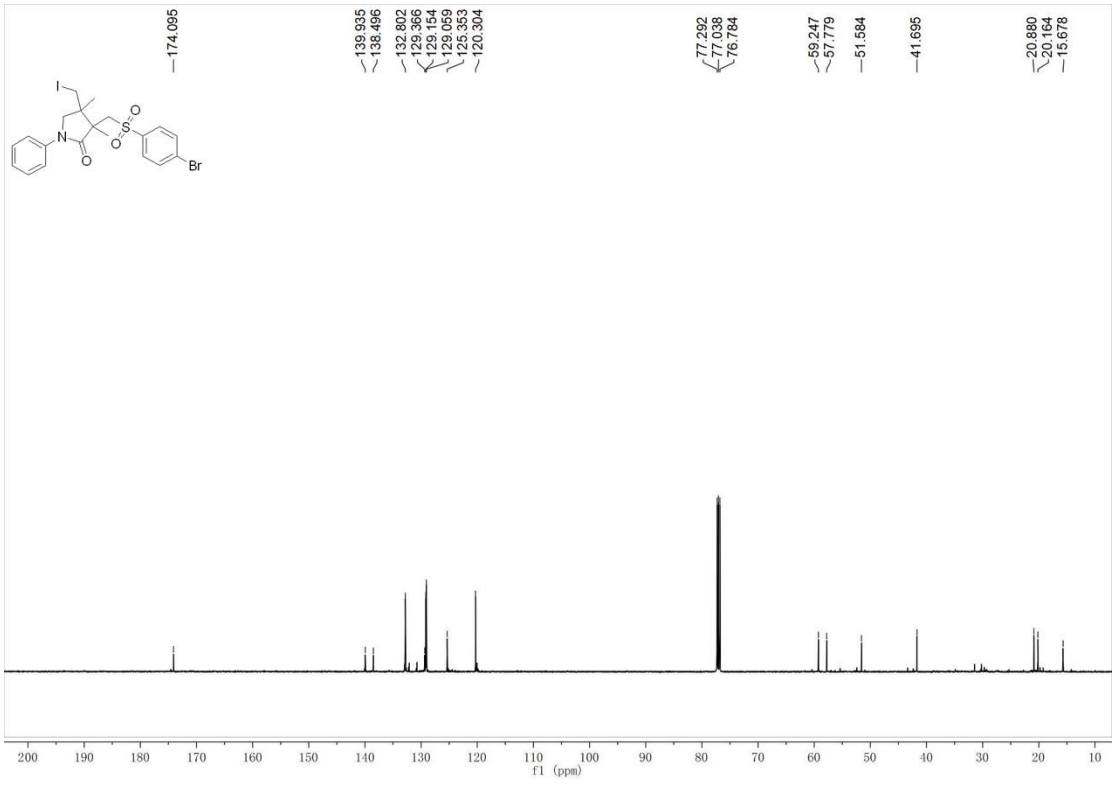
3-(((4-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4e)



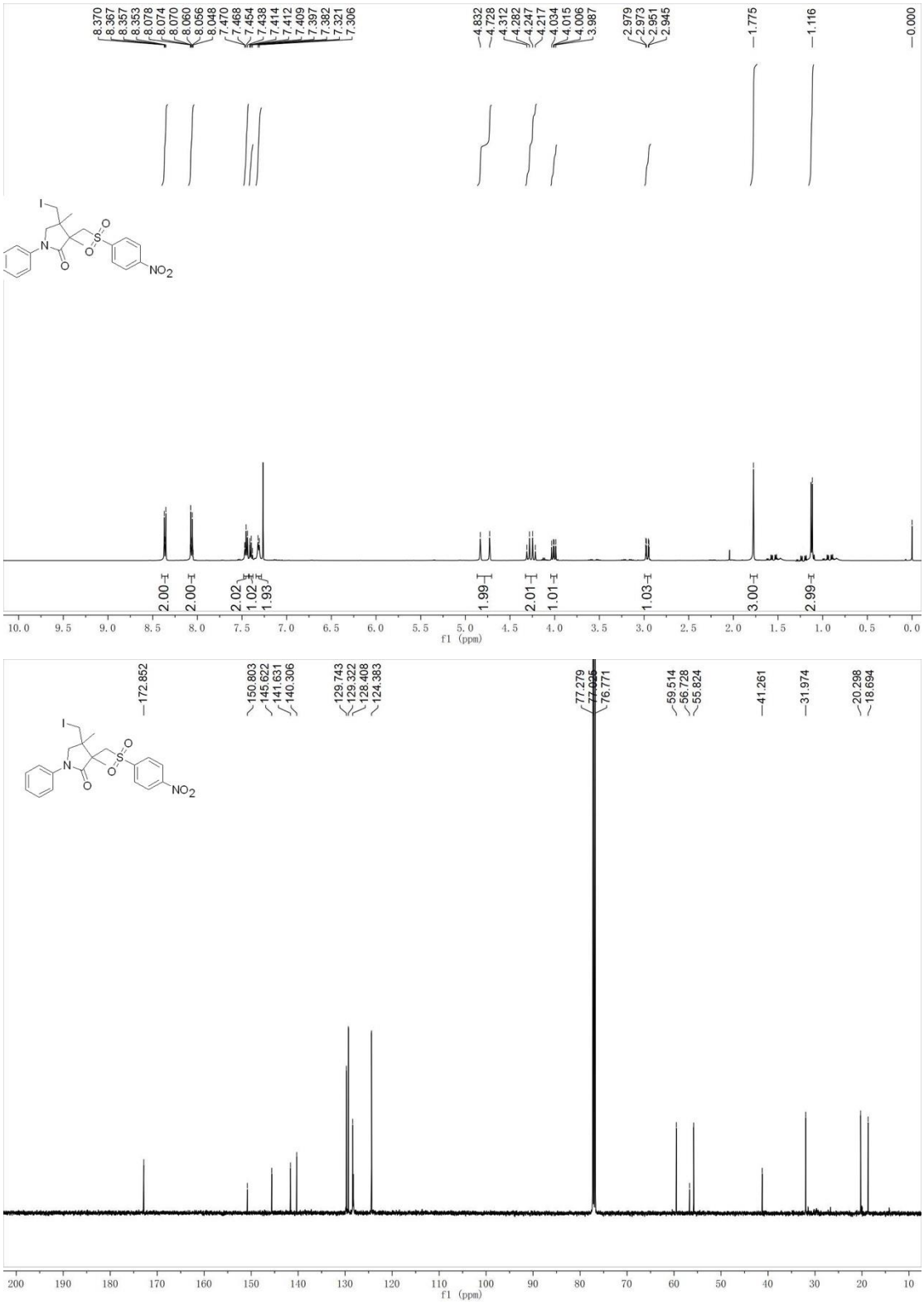


3-(((4-Bromophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4f)

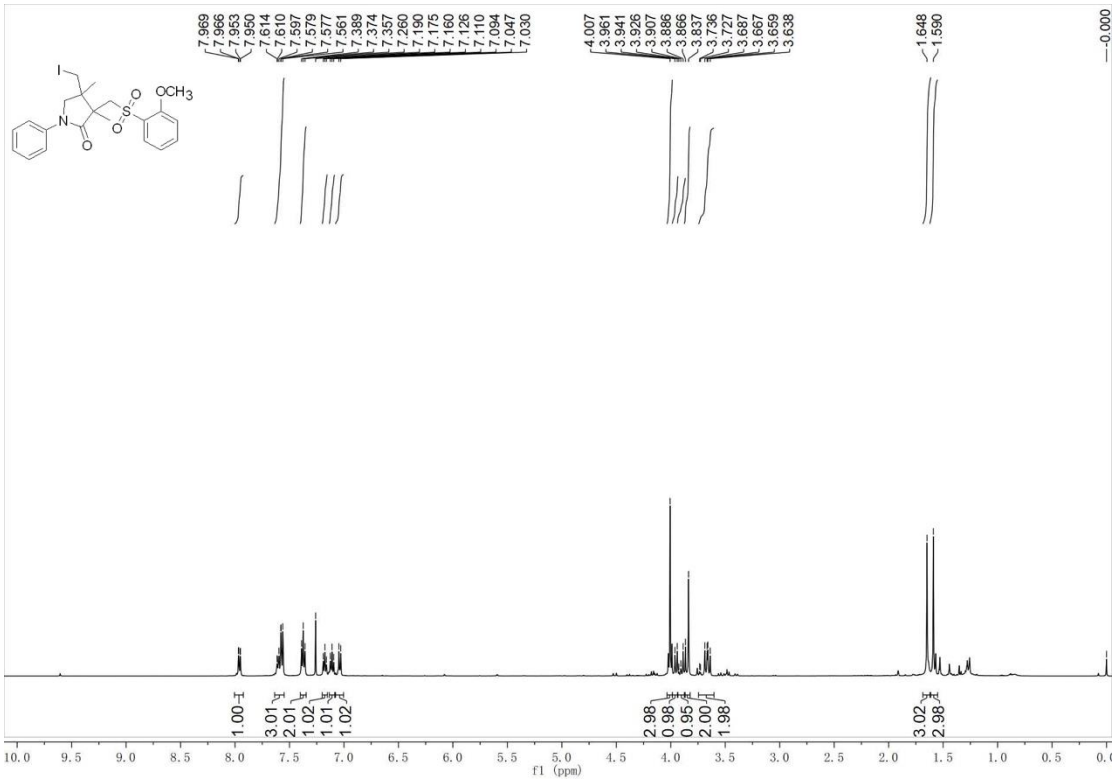


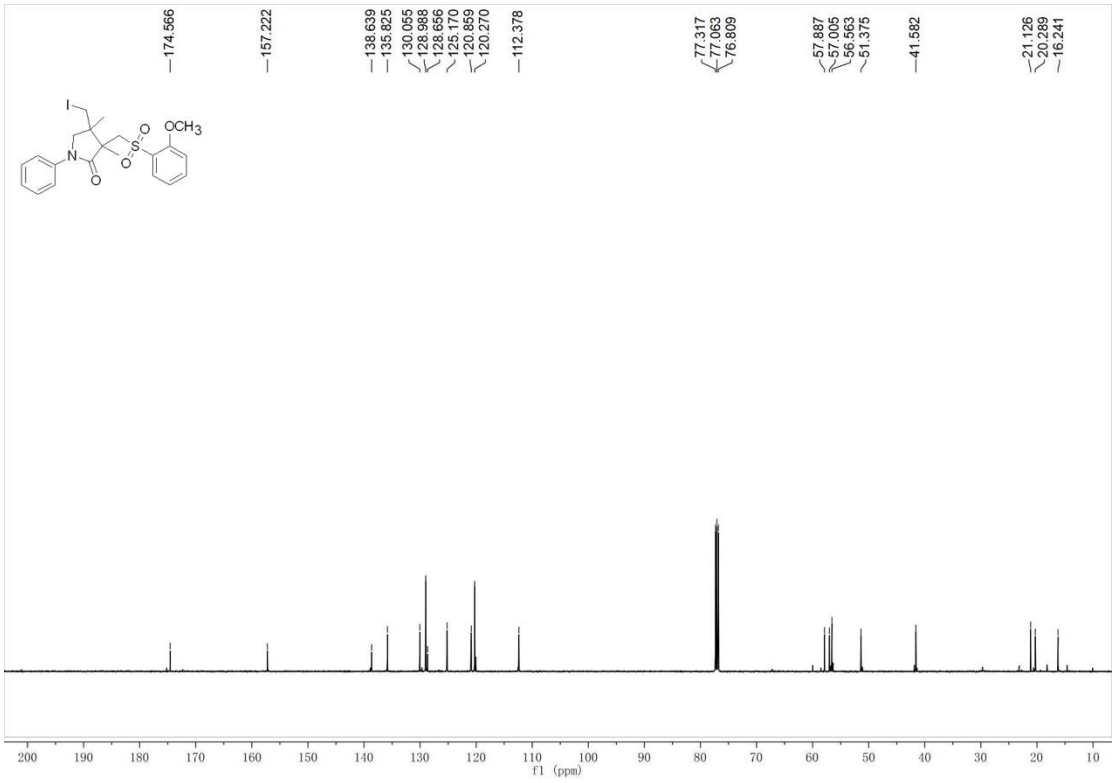


4-(Iodomethyl)-3,4-dimethyl-3-(((4-nitrophenyl)sulfonyl)methyl)-1-phenylpyrrolidin-2-one (4g)

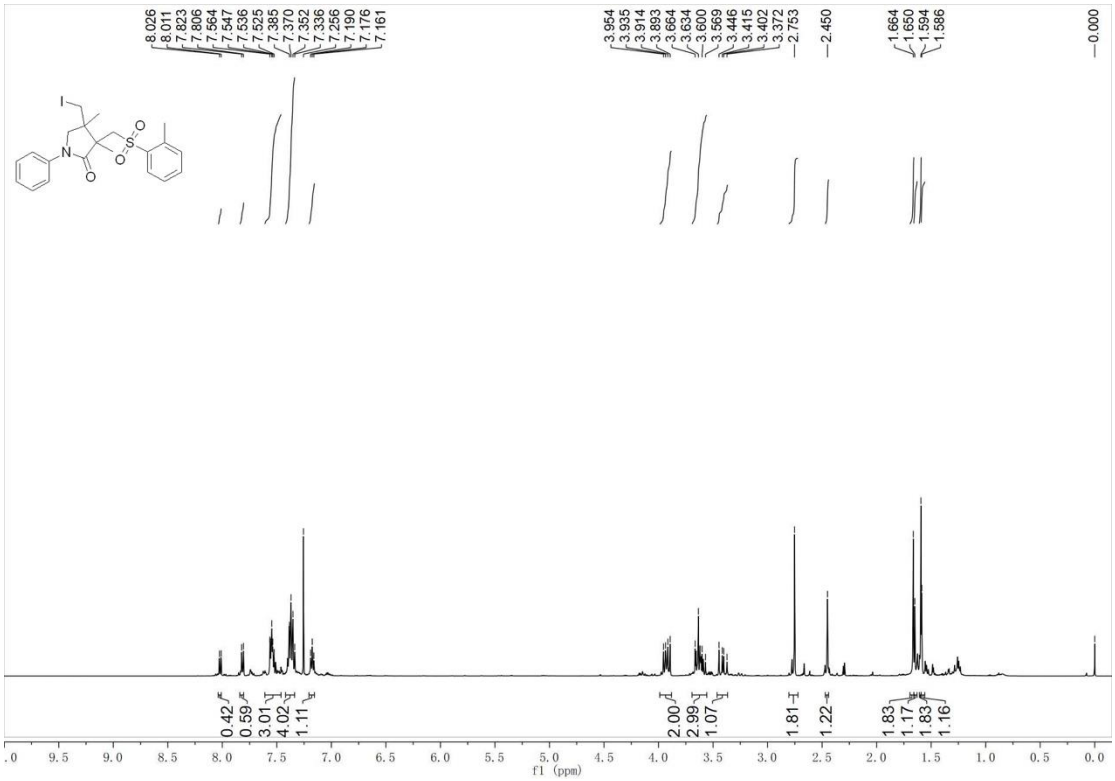


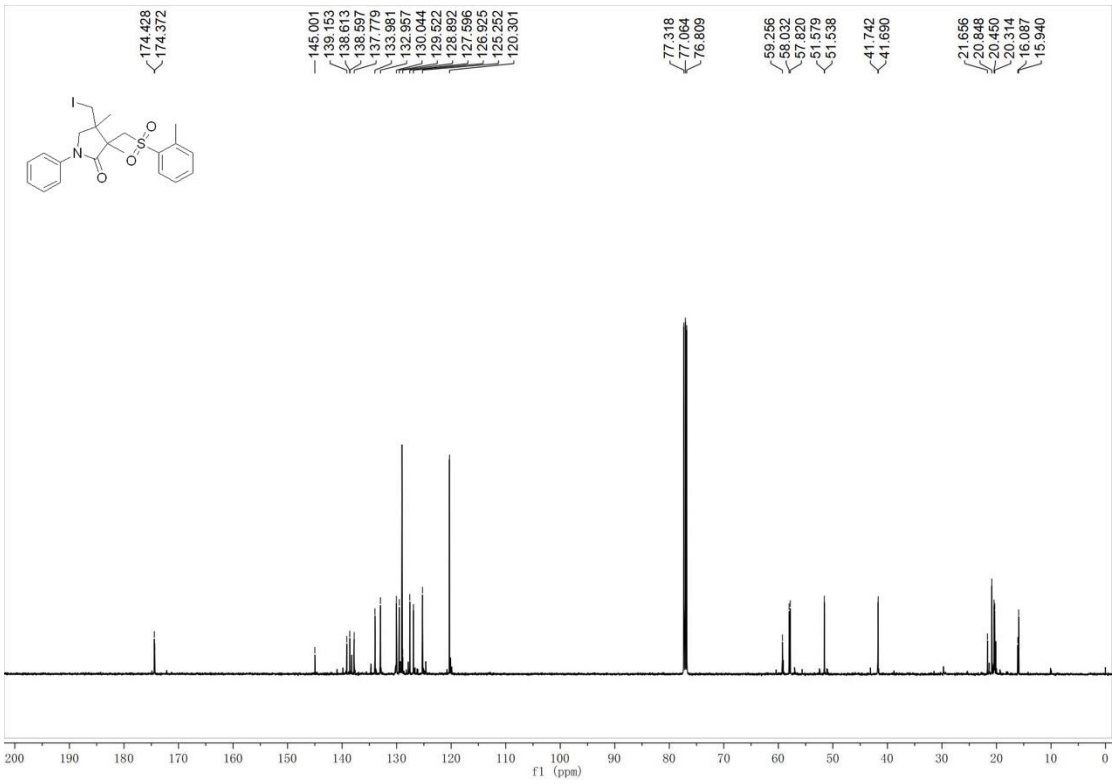
4-(Iodomethyl)-3-(((2-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4h)



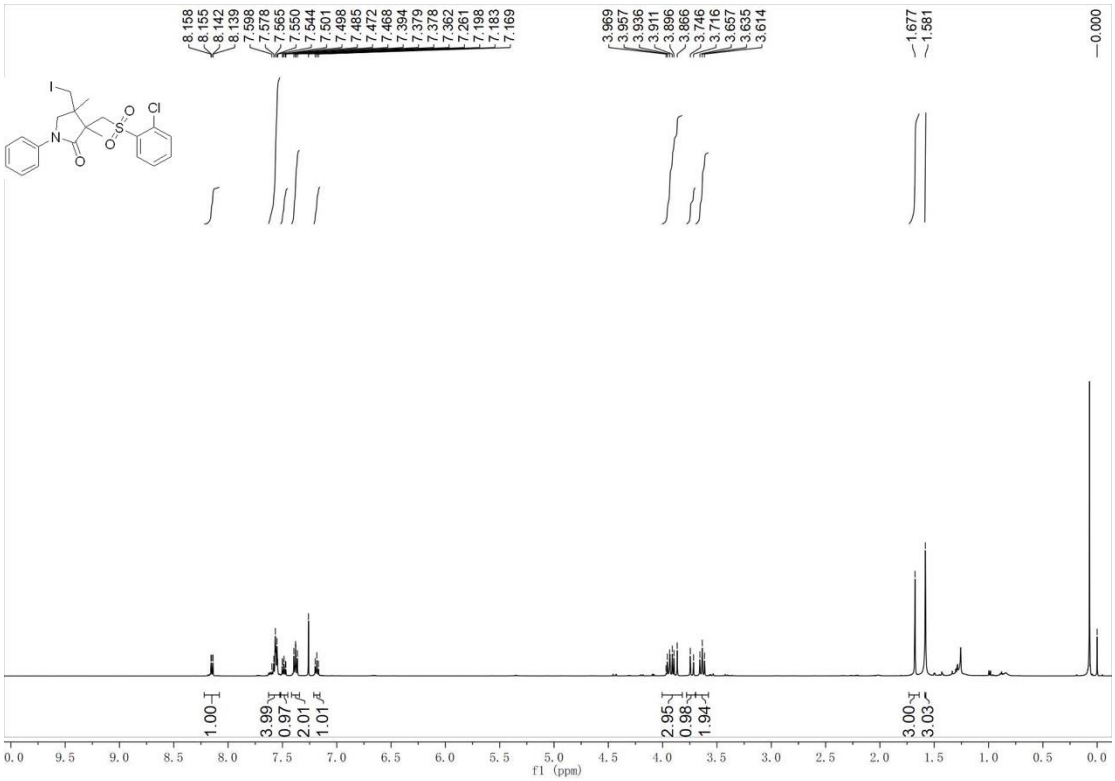


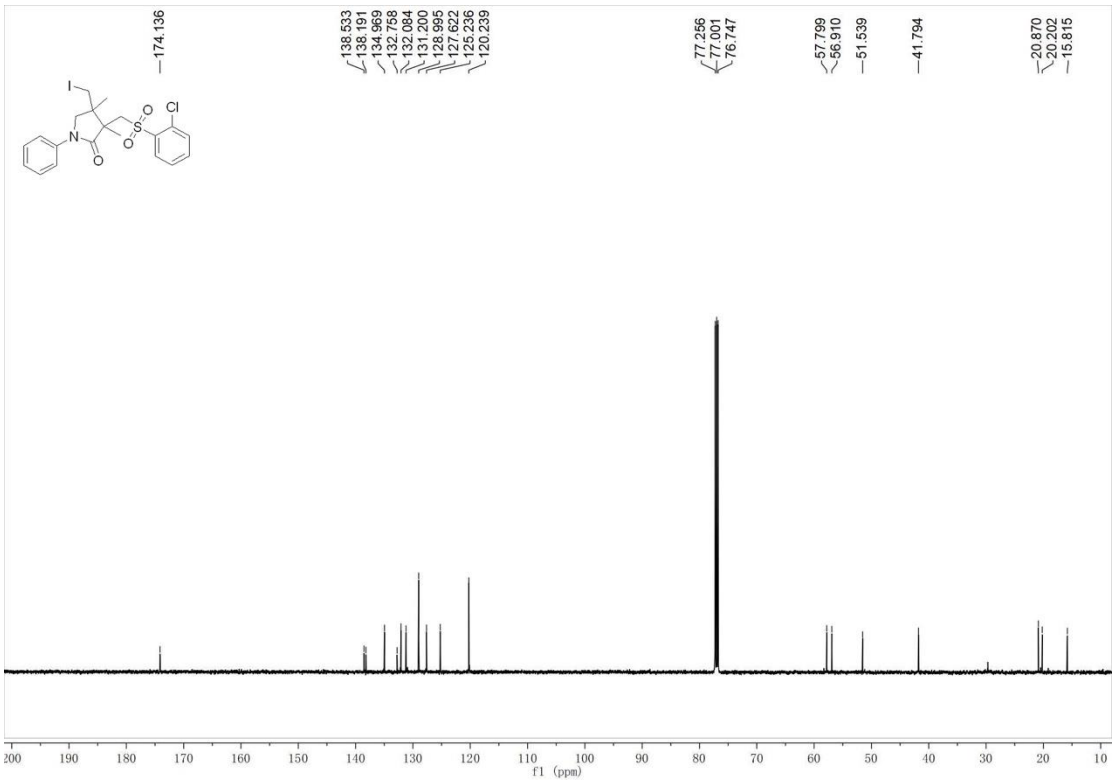
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((*o*-tolylsulfonyl)methyl)pyrrolidin-2-one (4i)



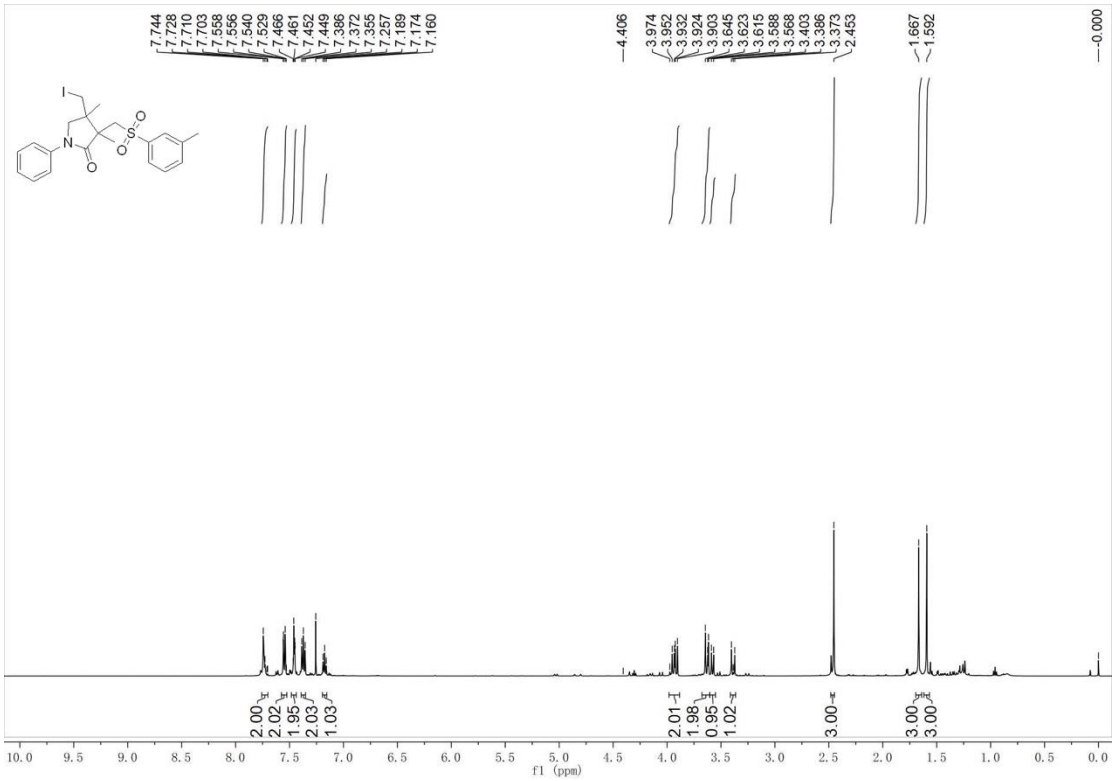


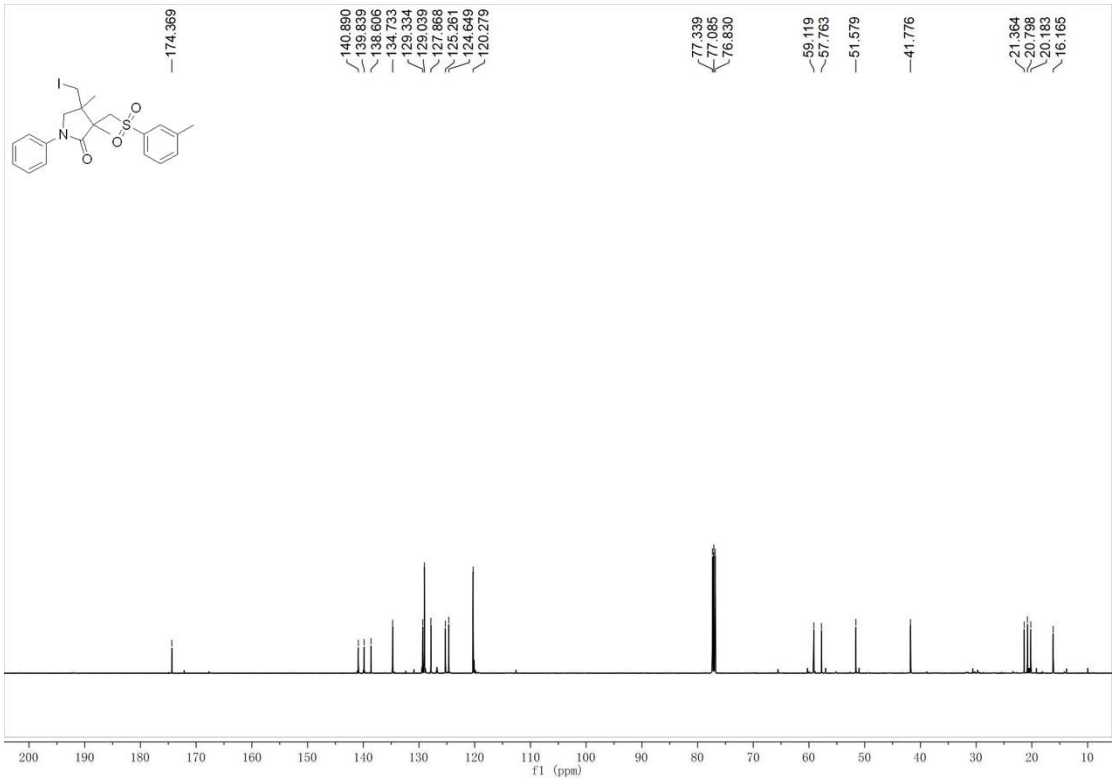
3-(((2-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4j)



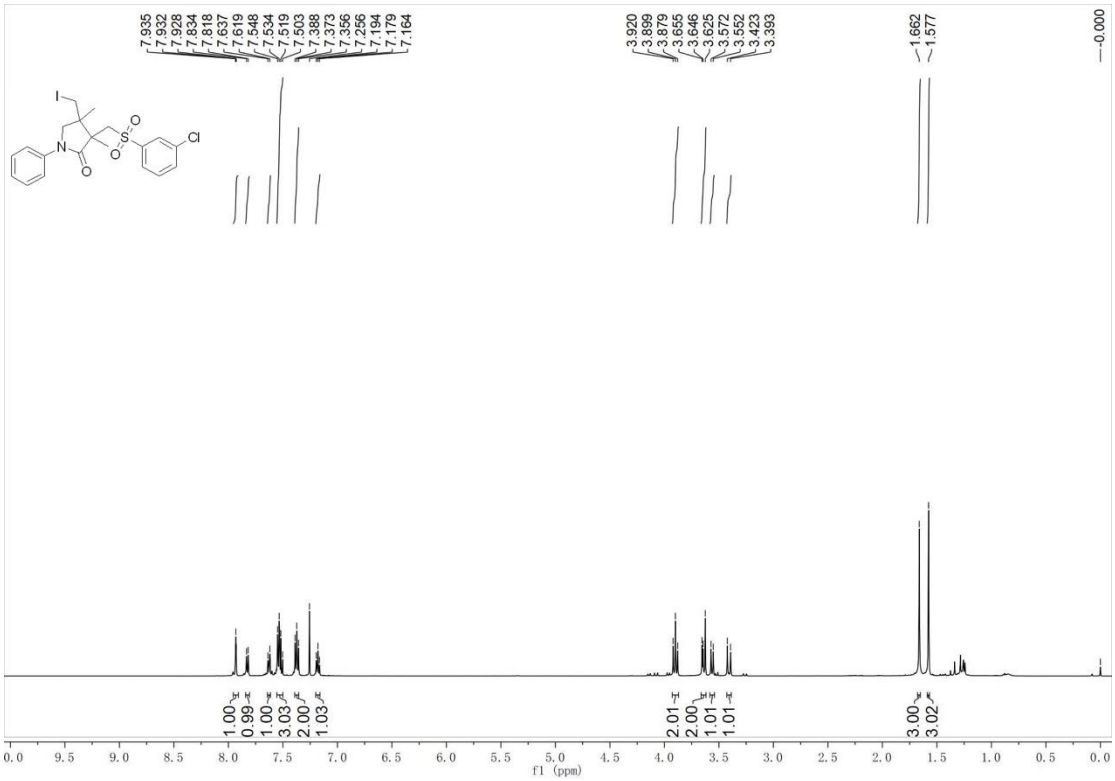


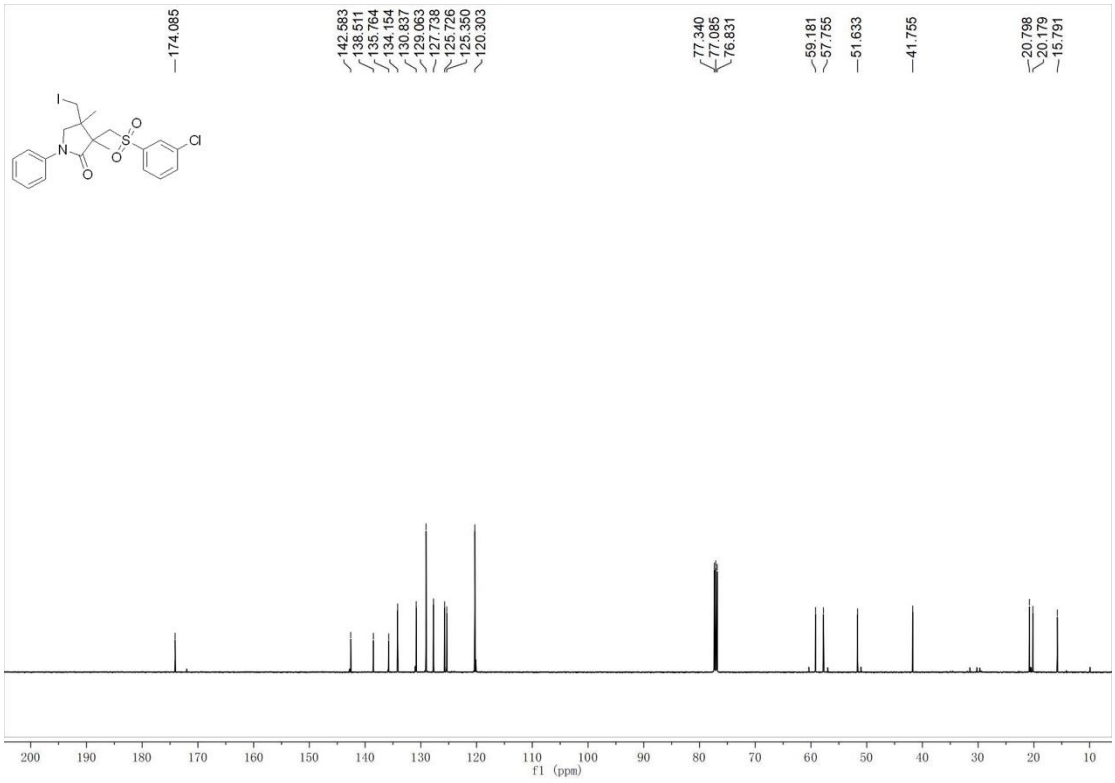
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((*m*-tolylsulfonyl)methyl)pyrrolidin-2-one (4k)



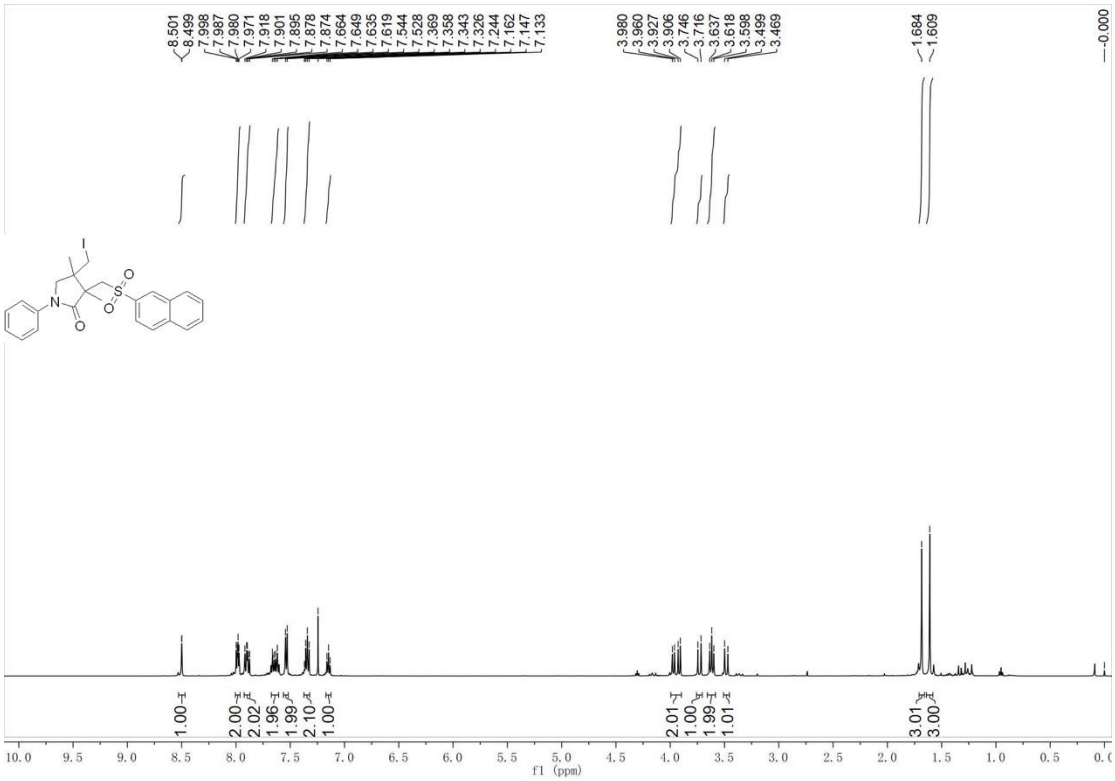


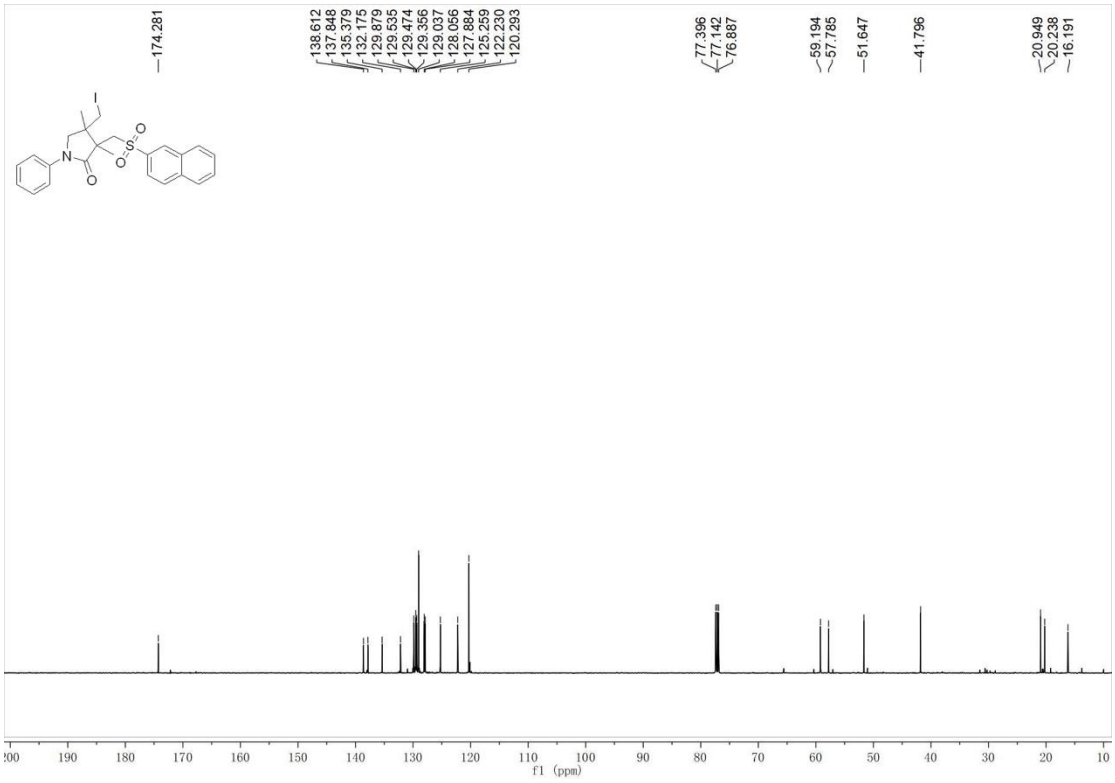
3-(((3-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4l)



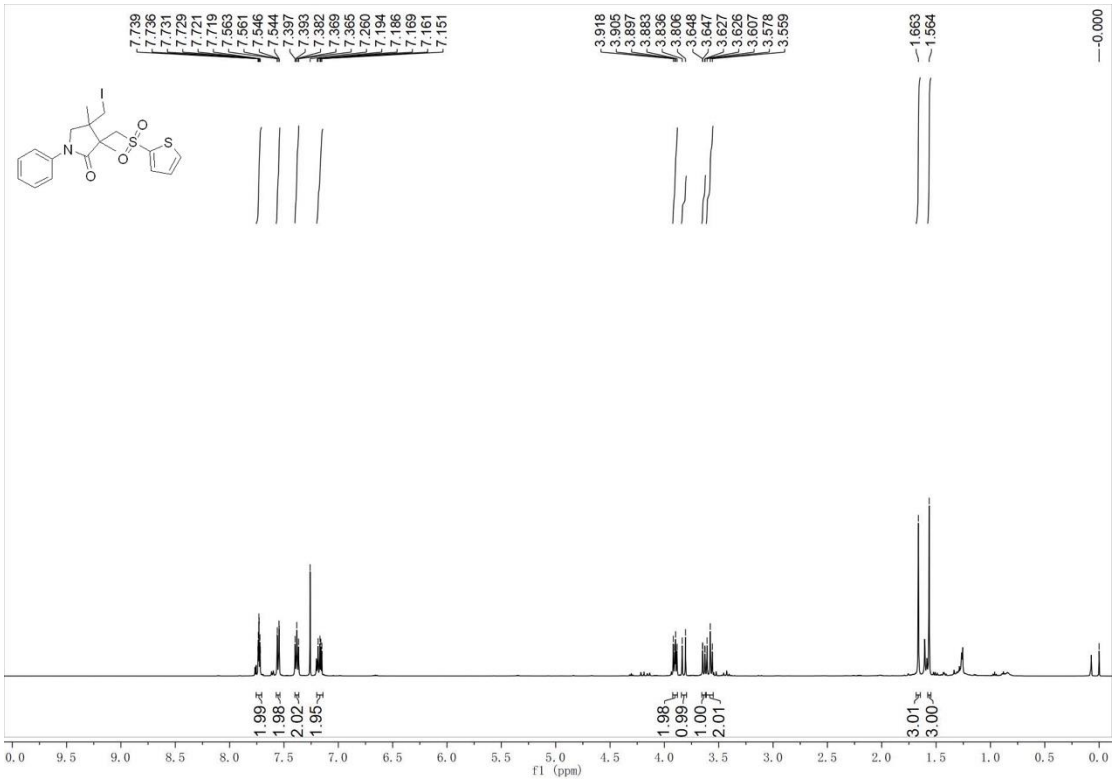


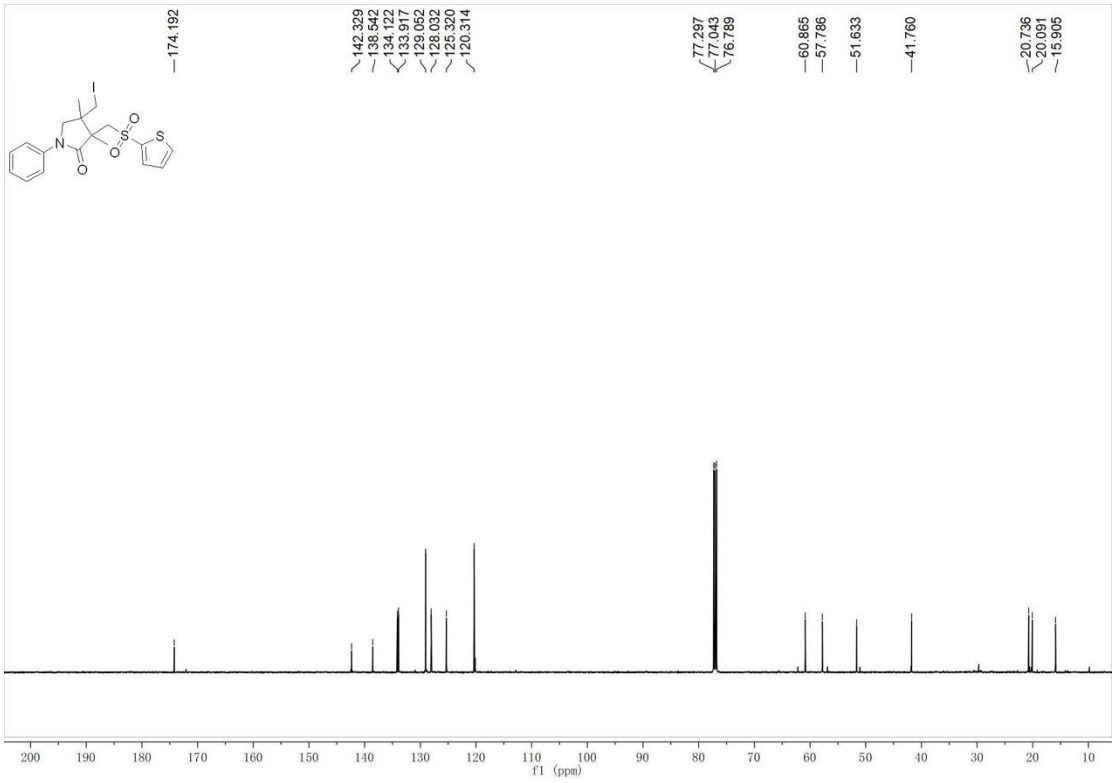
4-(Iodomethyl)-3,4-dimethyl-3-((naphthalen-2-ylsulfonyl)methyl)-1-phenylpyrrolidin-2-one (4m)



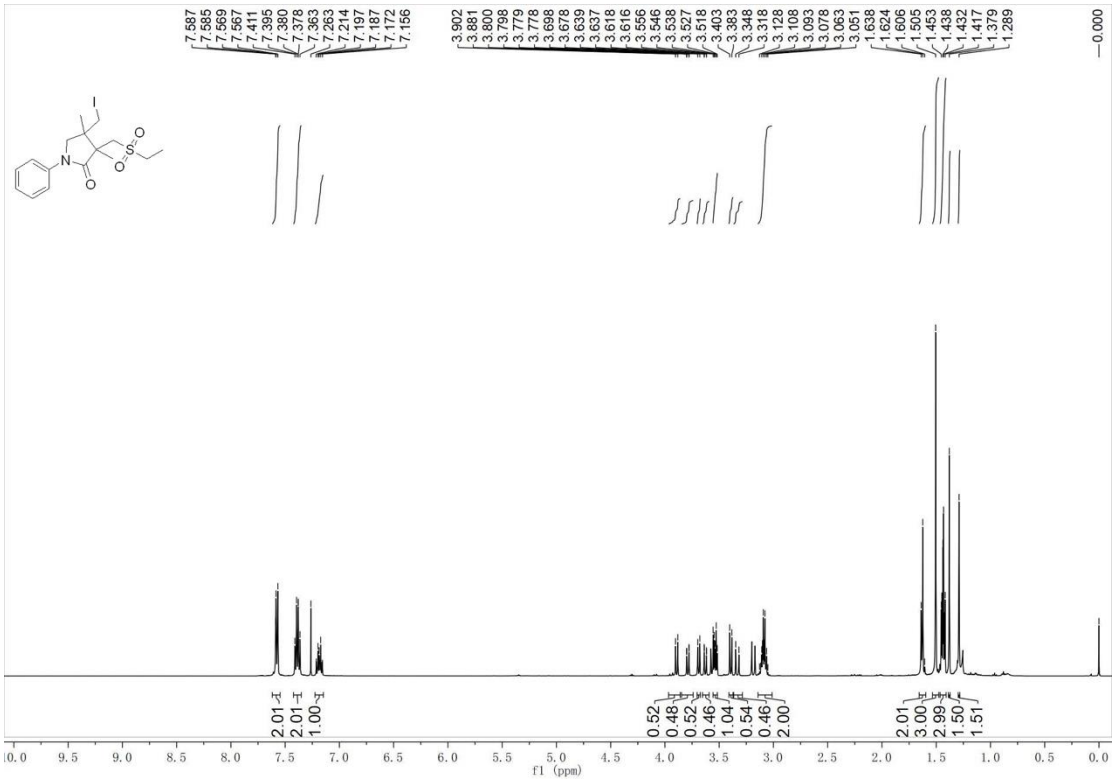


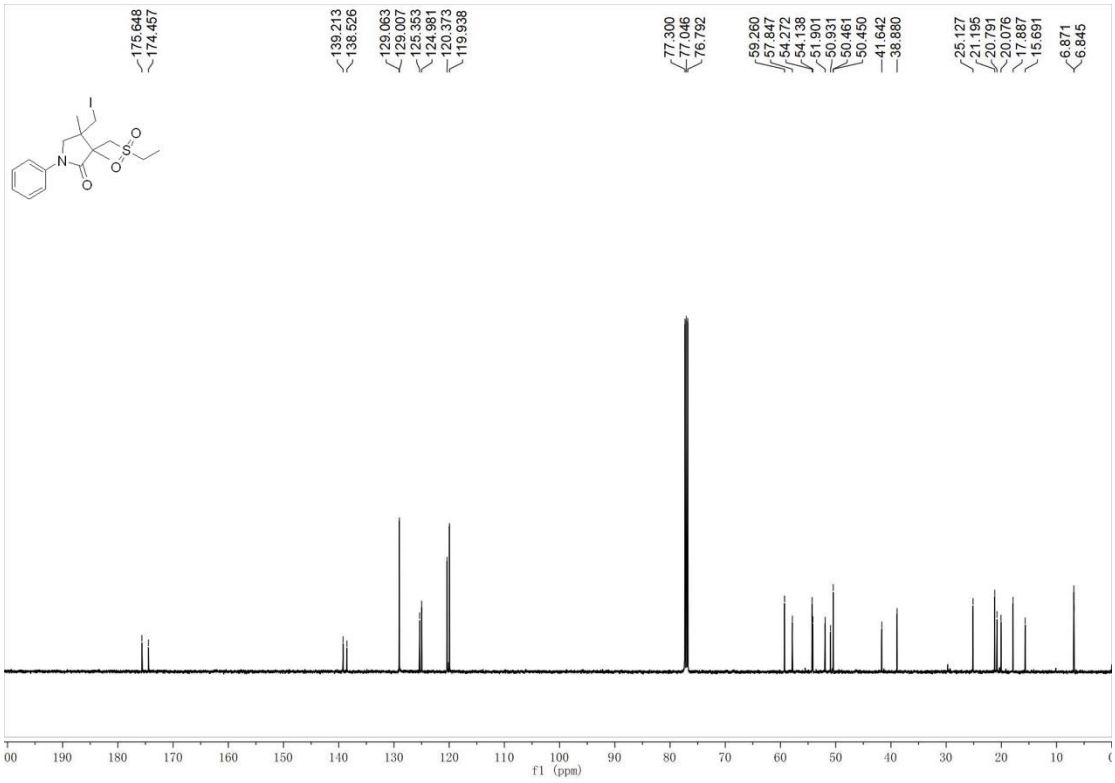
4-(Iodomethyl)-3,4-dimethyl-1-phenyl-3-((thiophen-2-ylsulfonyl)methyl)pyrrolidin-2-one (4n)



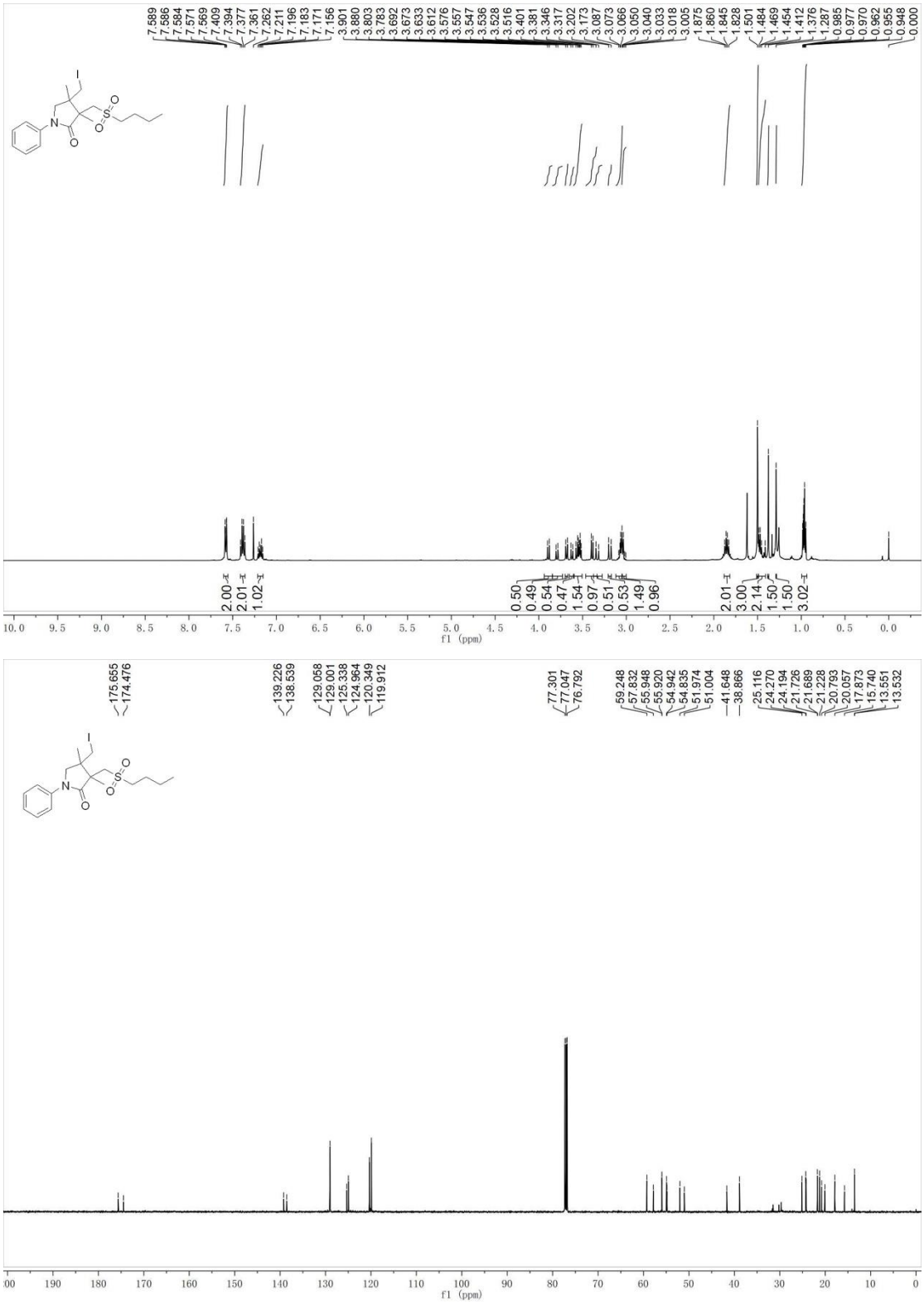


3-((Ethylsulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4o)

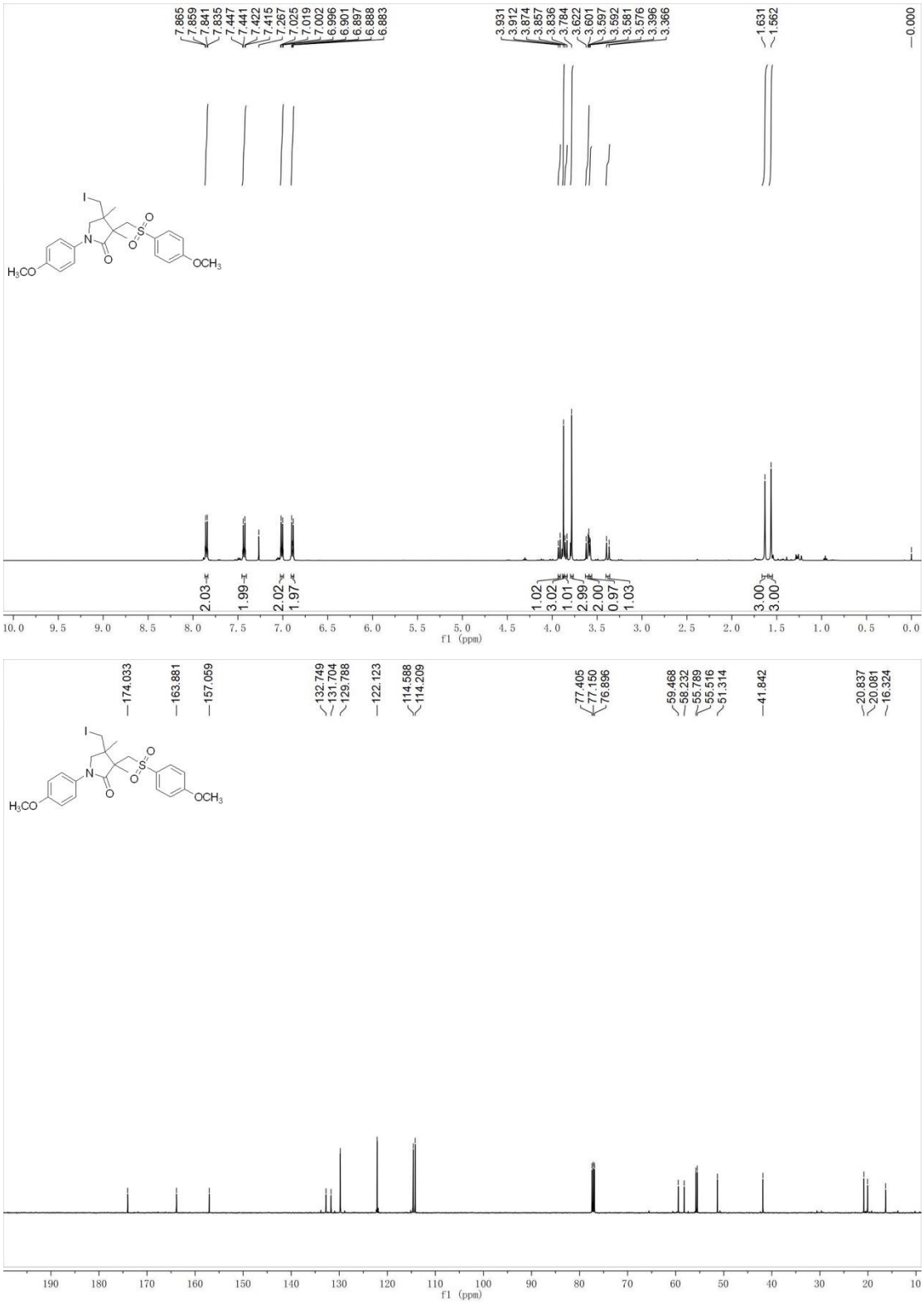




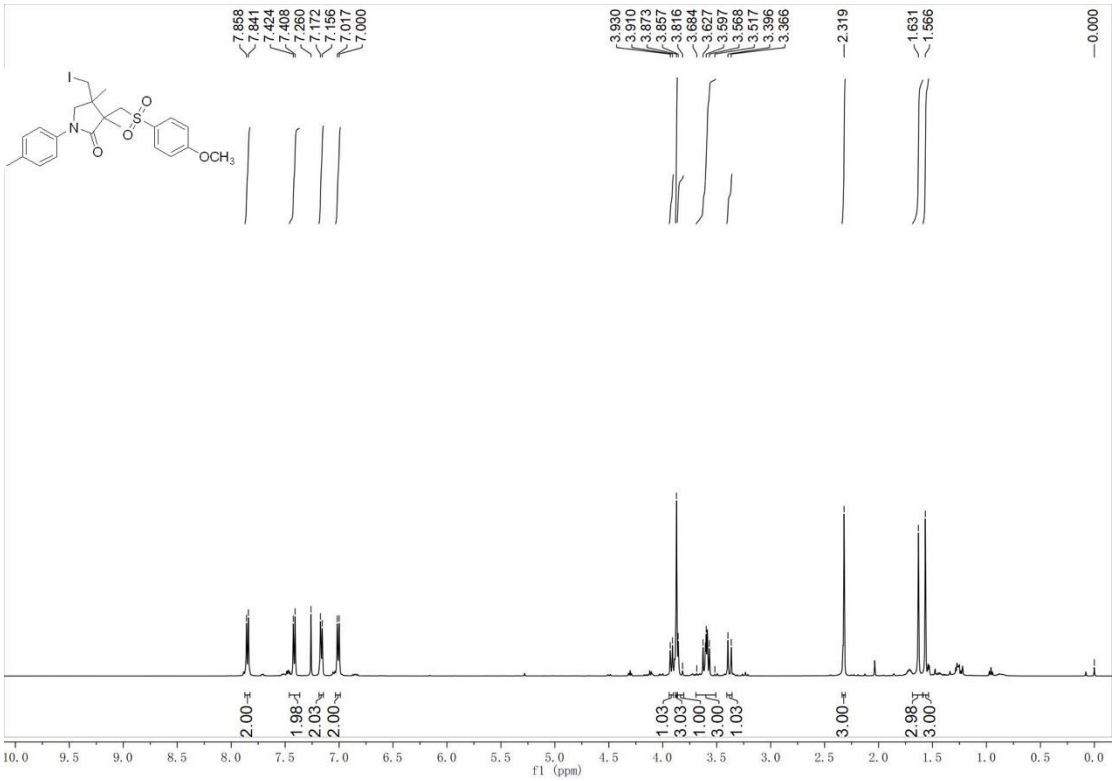
3-((Butylsulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-phenylpyrrolidin-2-one (4p)

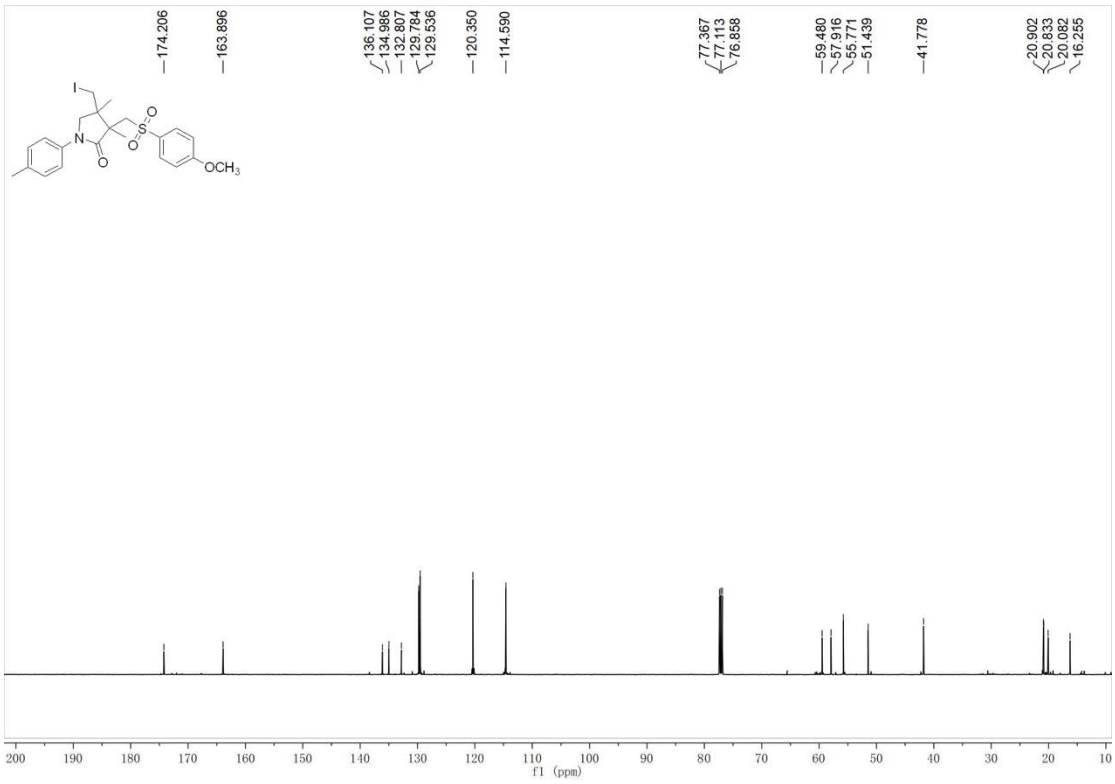


4-(Iodomethyl)-1-(4-methoxyphenyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4q)

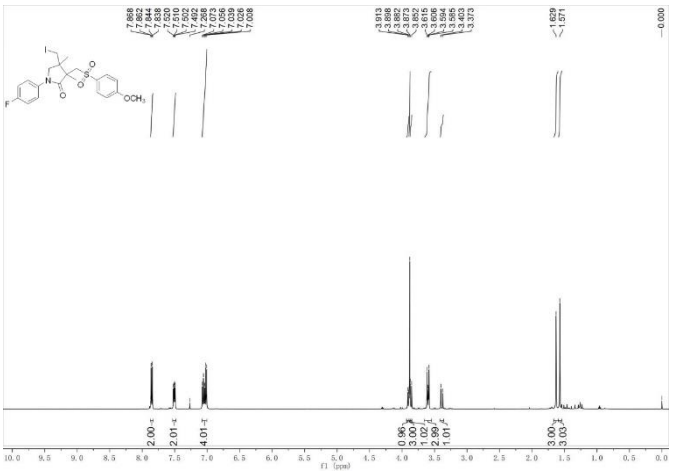


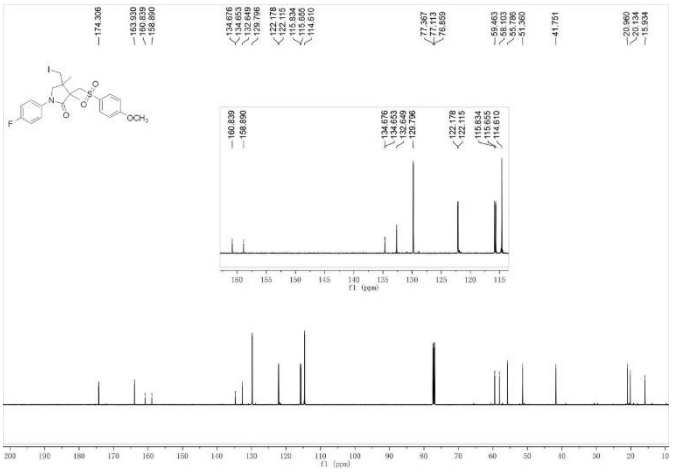
4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-(*p*-tolyl)pyrrolidin-2-one (4r)

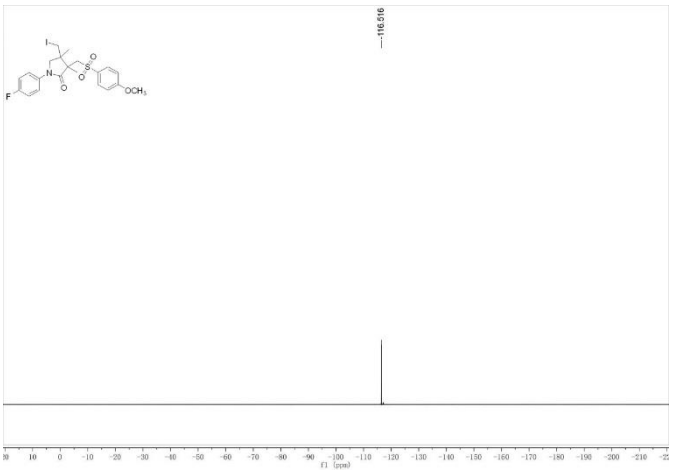




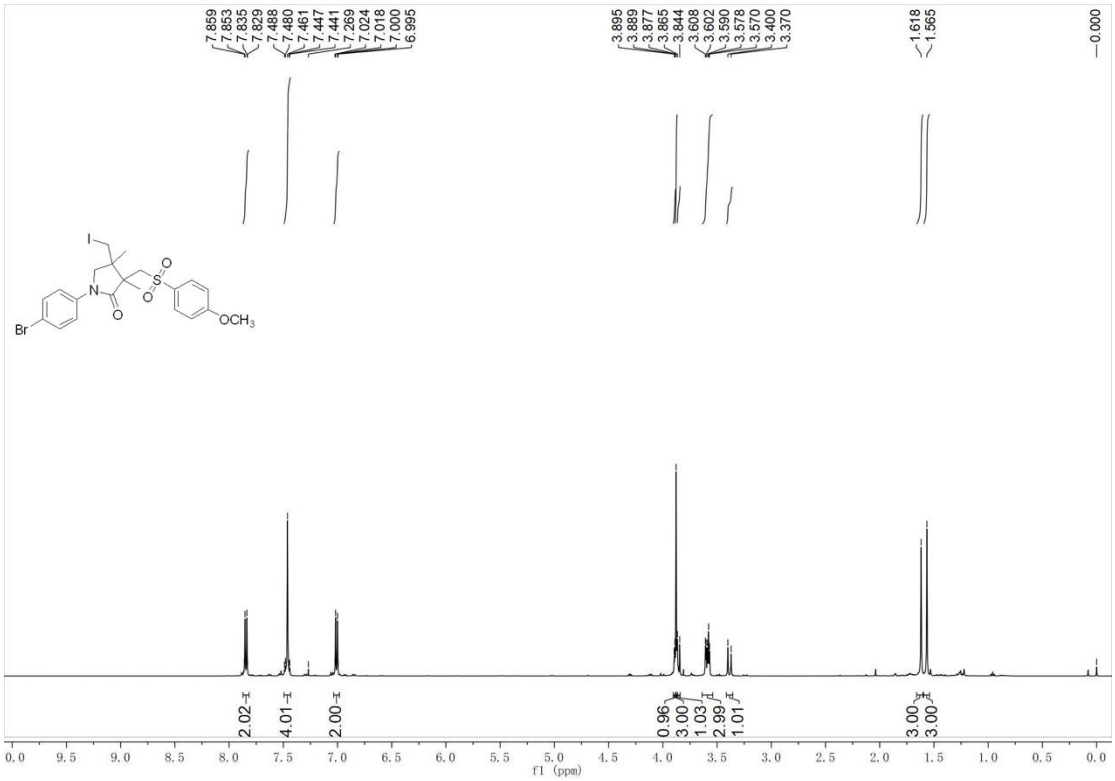
1-(4-Fluorophenyl)-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4s)

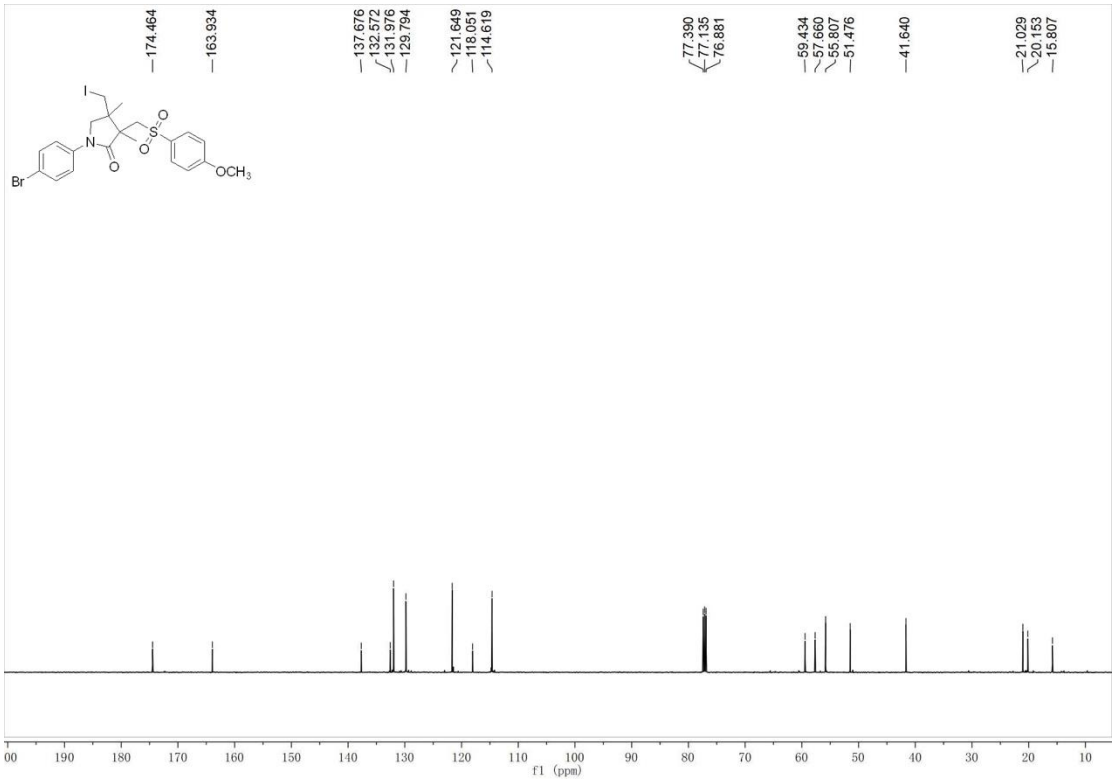




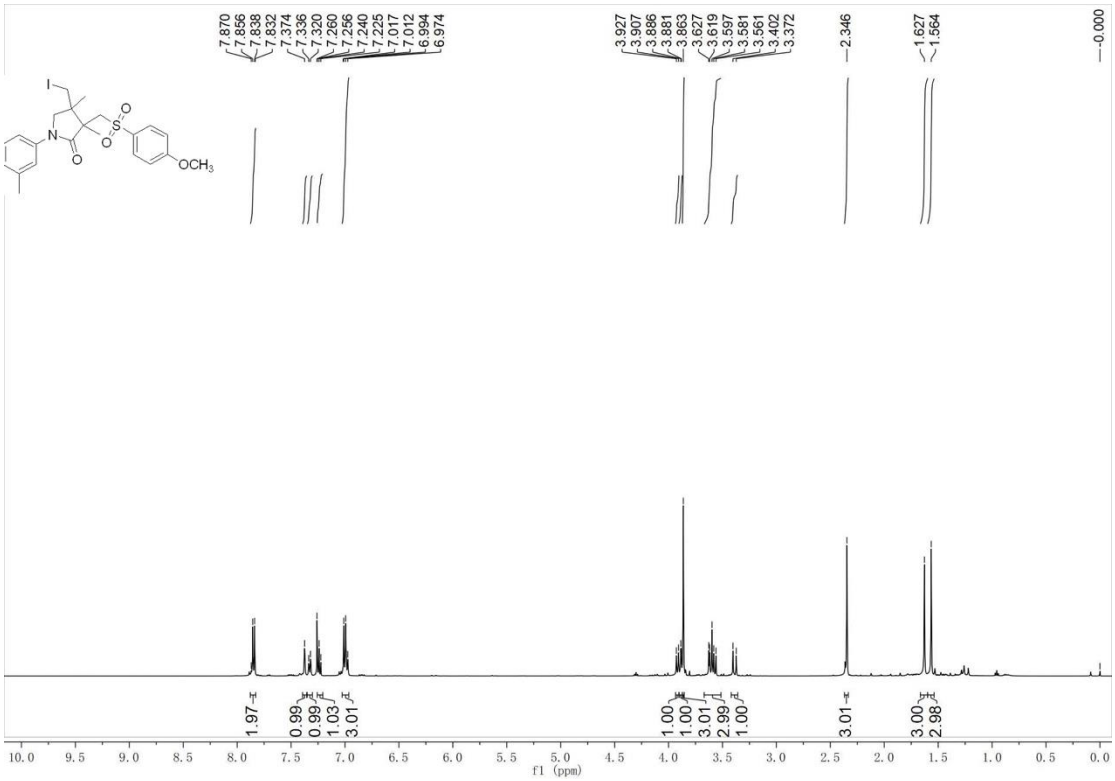


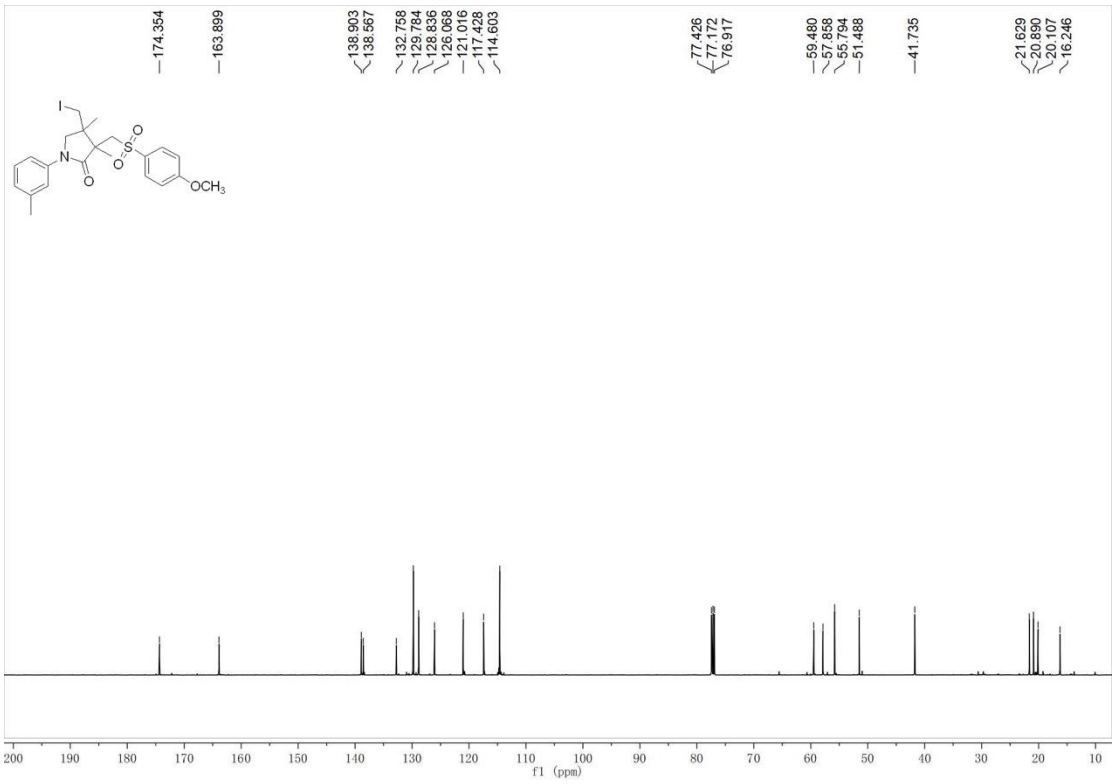
1-(4-Bromophenyl)-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethylpyrrolidin-2-one (4t)



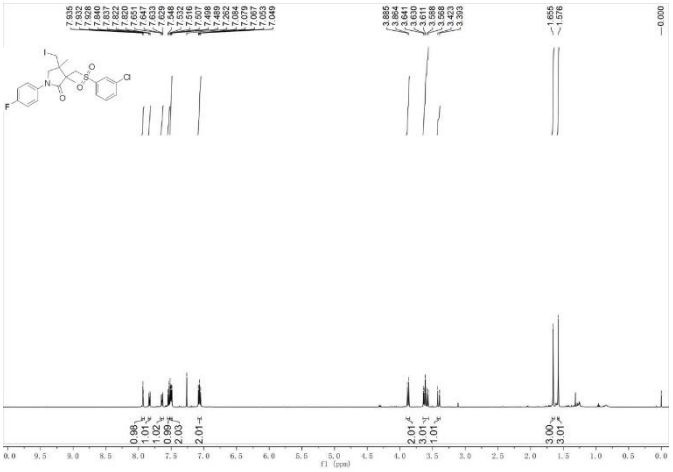


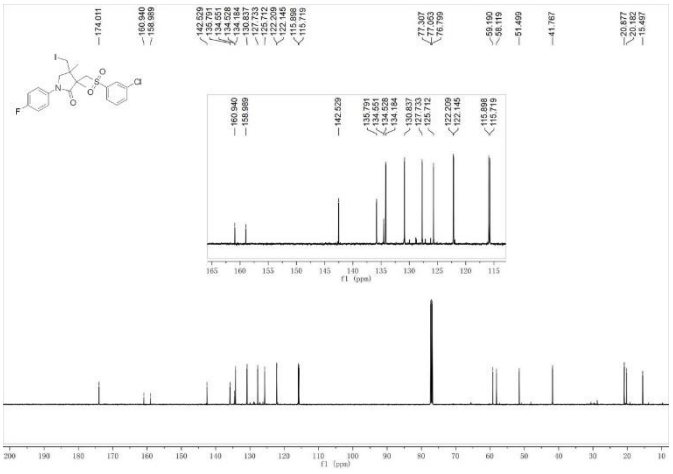
4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-(*m*-tolyl)pyrrolidin-2-one (4u)

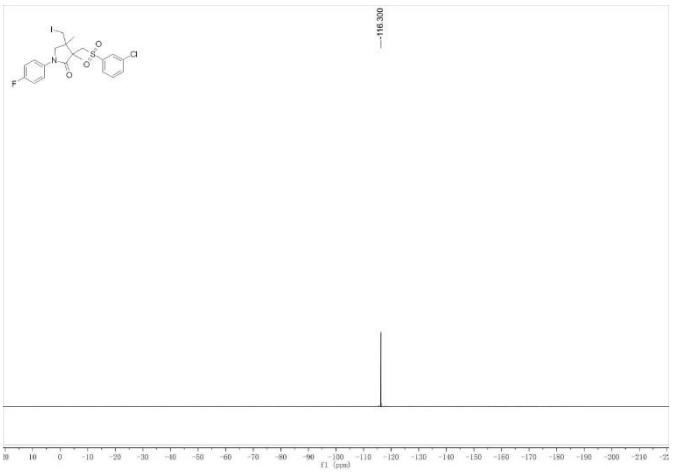




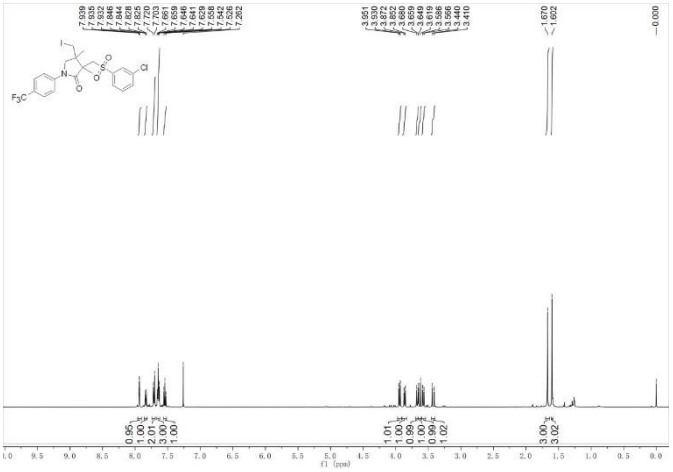
3-(((3-Chlorophenyl)sulfonyl)methyl)-1-(4-fluorophenyl)-4-(iodomethyl)-3,4-dimethylpyrrolidin-2-one (4v)

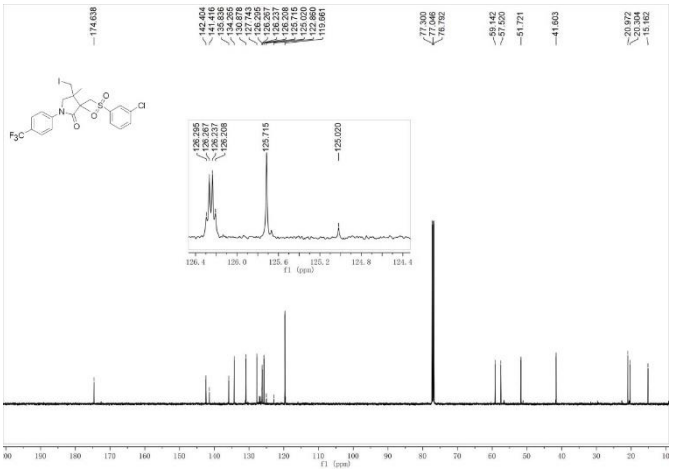


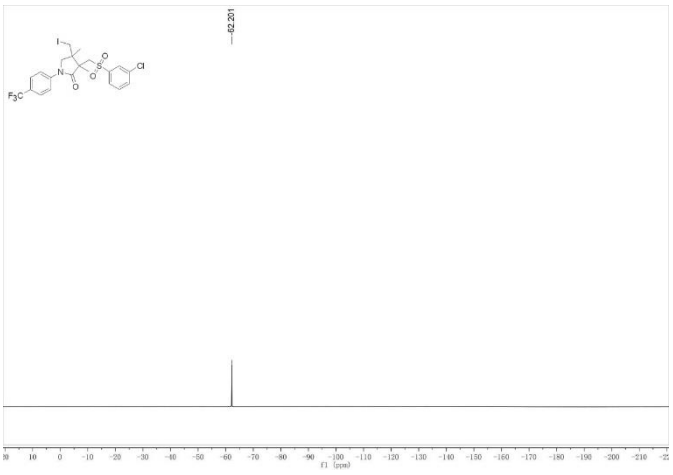




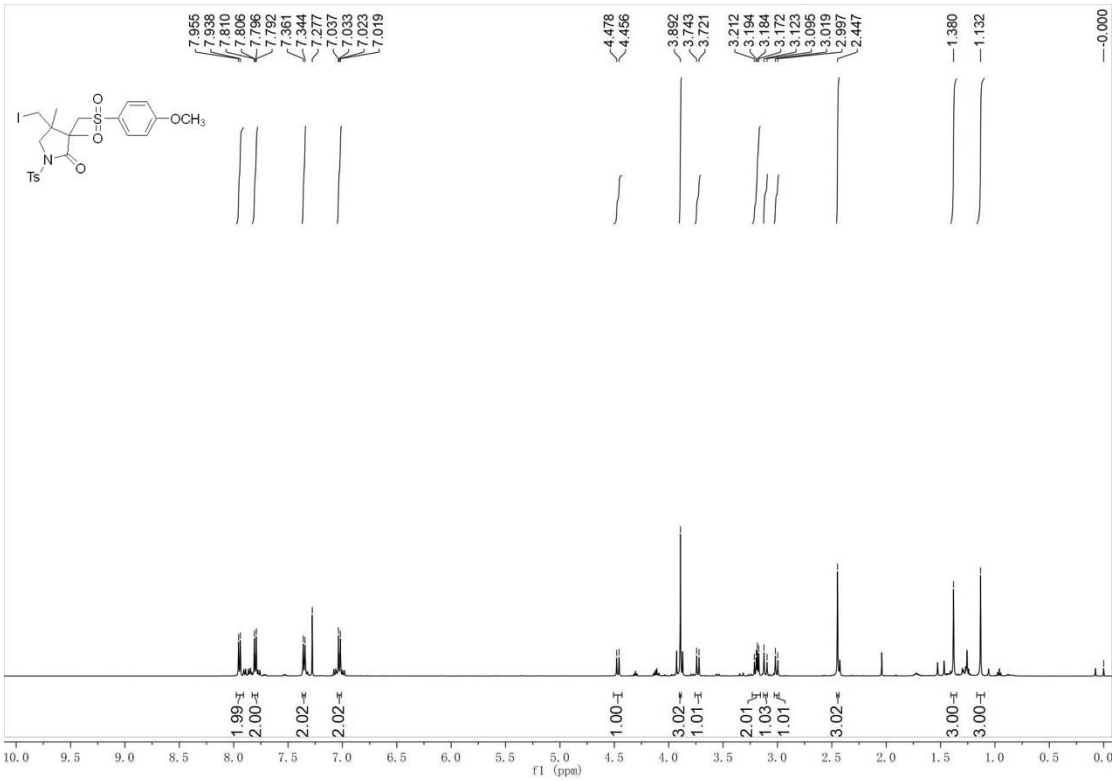
3-(((3-Chlorophenyl)sulfonyl)methyl)-4-(iodomethyl)-3,4-dimethyl-1-(4-(trifluoro methyl)phenyl)pyrrolidin-2-one (4w)

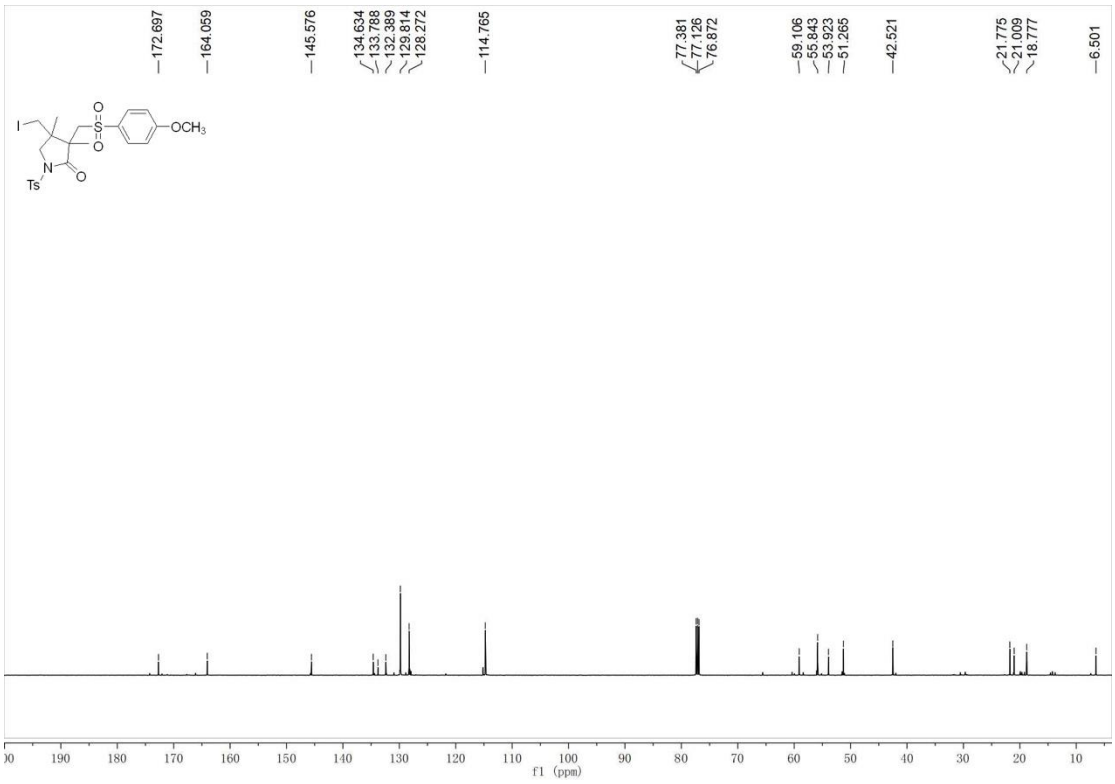




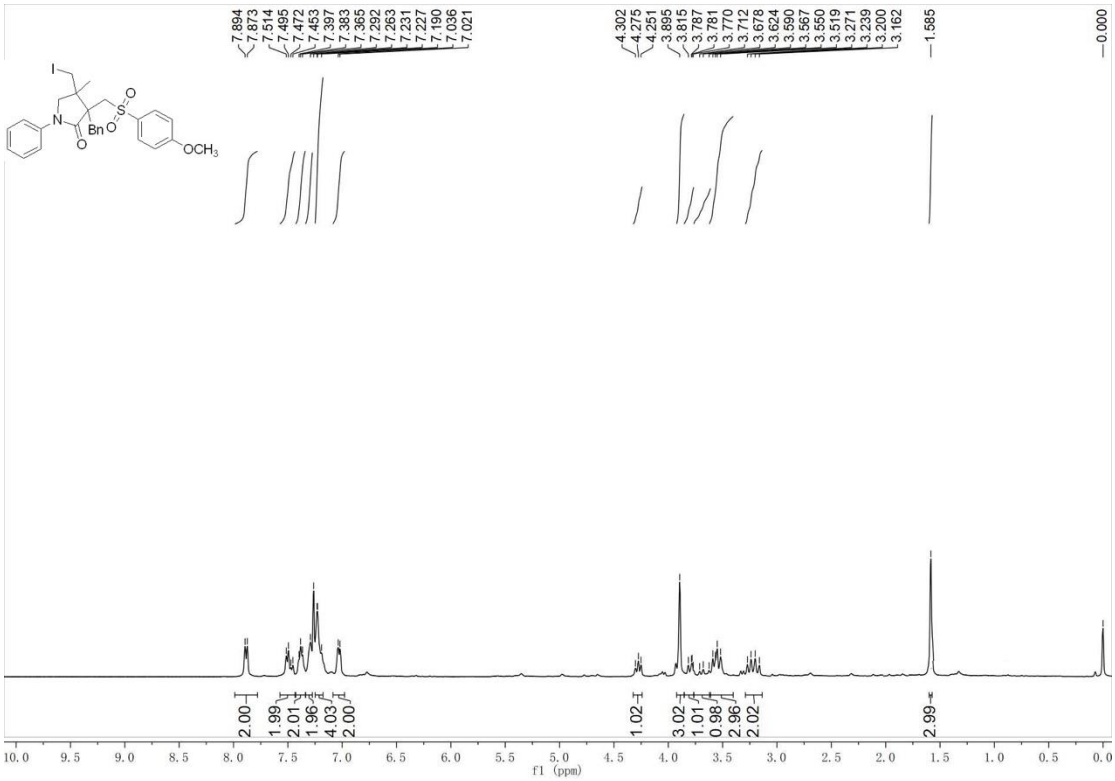


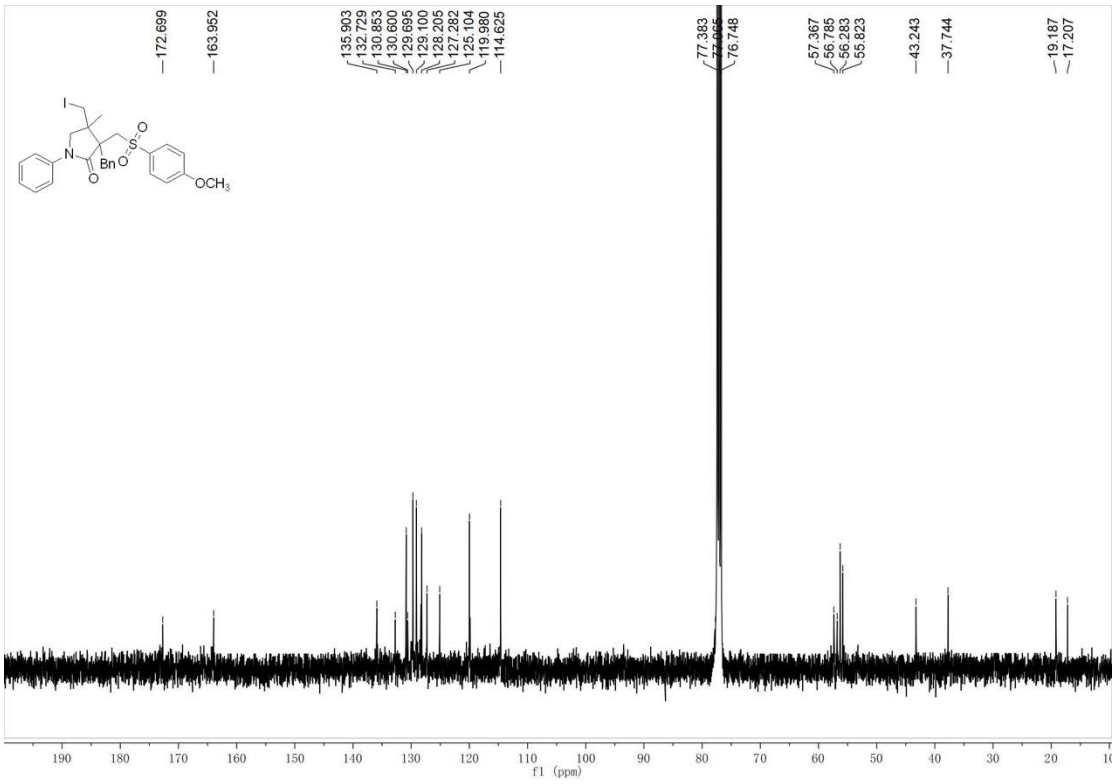
4-(Iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-3,4-dimethyl-1-tosylpyrrolidin-2-one (4x)



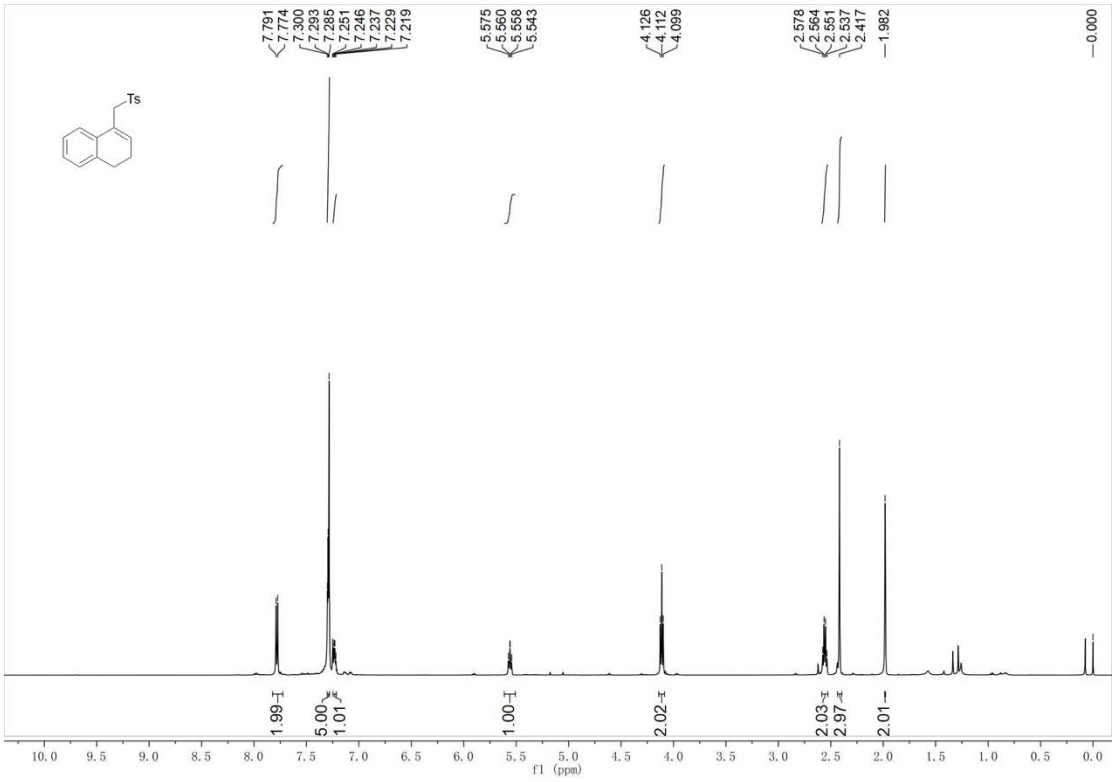


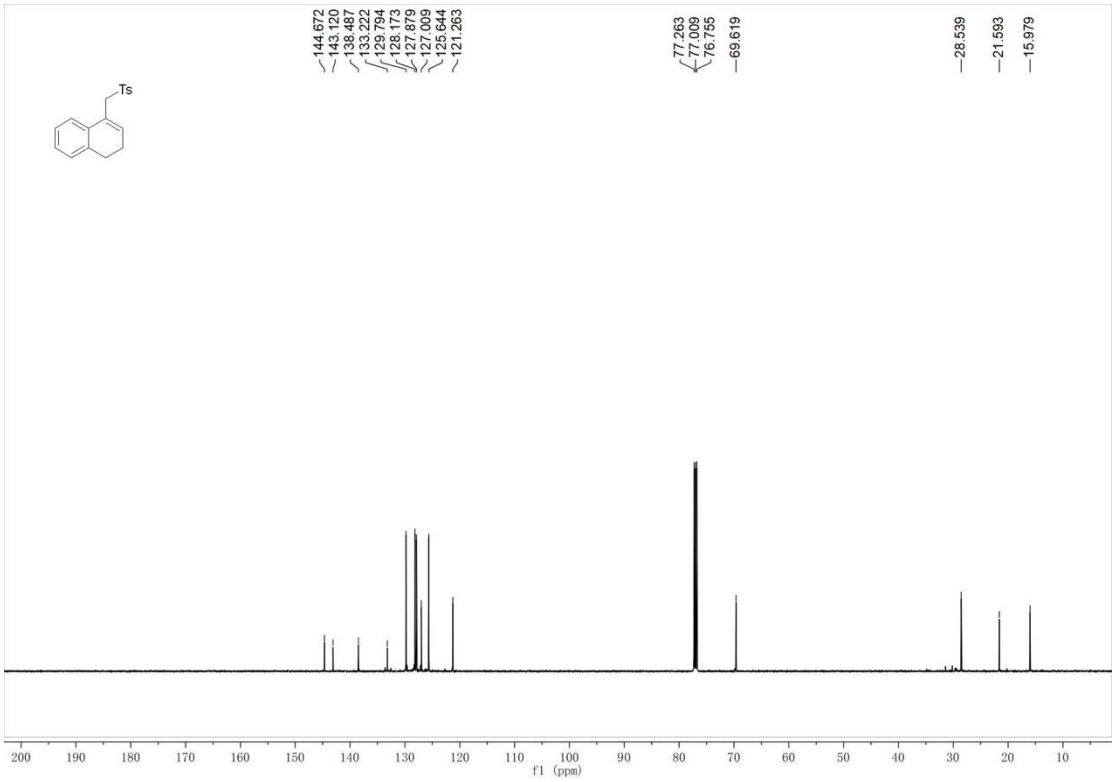
3-Benzyl-4-(iodomethyl)-3-(((4-methoxyphenyl)sulfonyl)methyl)-4-methyl-1-phenylpyrrolidin-2-one (4y)





4-(Tosylmethyl)-1,2-dihydronaphthalene (5a)





(E) The X-ray single-crystal diffraction analysis of product 3a

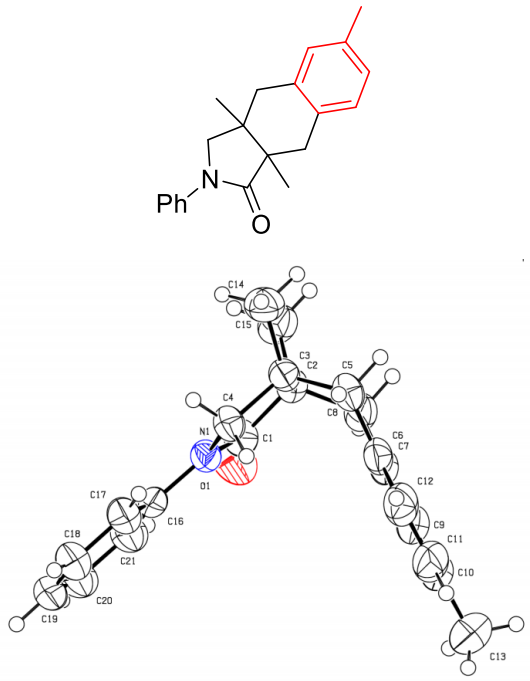


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Table 1. Crystal data and structure refinement for 3a.

Identification code	**3a**	
Empirical formula	$C_{21}H_{23}NO$	
Formula weight	305.40	
Temperature	297.0 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 6.747(2) Å	a= 90°.
	b = 9.863(3) Å	b= 95.475(9)°.
	c = 25.692(7) Å	g = 90°.
Volume	1702.0(9) $Å^3$	
Z	4	
Density (calculated)	1.192 Mg/m^3	
Absorption coefficient	0.072 mm^{-1}	
F(000)	656	
Crystal size	0.22 x 0.21 x 0.16 mm^3	
Theta range for data collection	2.608 to 25.389°.	
Index ranges	-8<=h<=8, -11<=k<=11, -30<=l<=30	
Reflections collected	17903	

Independent reflections	3124 [R(int) = 0.0273]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6922
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3124 / 0 / 212
Goodness-of-fit on F^2	1.027
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1323
R indices (all data)	R1 = 0.0573, wR2 = 0.1424
Extinction coefficient	0.021(3)
Largest diff. peak and hole	0.358 and -0.181 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	4782(2)	7272(2)	6729(1)	87(1)
N(1)	8074(2)	7129(1)	6569(1)	44(1)
C(1)	6259(2)	7736(2)	6554(1)	52(1)
C(2)	6302(2)	9119(2)	6294(1)	47(1)
C(3)	8460(2)	9260(2)	6128(1)	45(1)
C(4)	9496(2)	7920(2)	6304(1)	48(1)
C(5)	8464(2)	9408(2)	5531(1)	53(1)
C(6)	7150(3)	8406(2)	5229(1)	53(1)
C(7)	5239(2)	8252(2)	5380(1)	52(1)
C(8)	4702(2)	9121(2)	5823(1)	55(1)
C(9)	3924(3)	7362(2)	5108(1)	66(1)
C(10)	4457(4)	6633(2)	4681(1)	77(1)
C(11)	6367(4)	6788(2)	4538(1)	82(1)
C(12)	7714(3)	7656(2)	4810(1)	70(1)
C(13)	3002(5)	5712(3)	4376(1)	118(1)
C(14)	9624(3)	10453(2)	6385(1)	64(1)
C(15)	5703(3)	10179(2)	6688(1)	67(1)
C(16)	8629(2)	5851(2)	6793(1)	48(1)
C(17)	10548(3)	5376(2)	6759(1)	62(1)
C(18)	11122(4)	4126(2)	6966(1)	77(1)
C(19)	9803(4)	3345(2)	7214(1)	83(1)
C(20)	7931(4)	3821(2)	7257(1)	82(1)
C(21)	7315(3)	5063(2)	7050(1)	65(1)

Table 3. Bond lengths [Å] and angles [deg] for 3a.

O(1)-C(1)	1.220(2)
N(1)-C(1)	1.361(2)
N(1)-C(4)	1.4549(19)
N(1)-C(16)	1.421(2)
C(1)-C(2)	1.521(2)
C(2)-C(3)	1.562(2)
C(2)-C(8)	1.543(2)
C(2)-C(15)	1.536(2)
C(3)-C(4)	1.543(2)
C(3)-C(5)	1.541(2)
C(3)-C(14)	1.529(2)
C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700
C(5)-H(5A)	0.9700
C(5)-H(5B)	0.9700
C(5)-C(6)	1.495(2)
C(6)-C(7)	1.389(2)
C(6)-C(12)	1.389(2)
C(7)-C(8)	1.496(2)
C(7)-C(9)	1.388(2)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-H(9)	0.9300
C(9)-C(10)	1.387(3)
C(10)-C(11)	1.382(3)
C(10)-C(13)	1.502(3)
C(11)-H(11)	0.9300
C(11)-C(12)	1.388(3)
C(12)-H(12)	0.9300
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-H(15A)	0.9600

C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-C(17)	1.387(3)
C(16)-C(21)	1.392(2)
C(17)-H(17)	0.9300
C(17)-C(18)	1.384(3)
C(18)-H(18)	0.9300
C(18)-C(19)	1.377(3)
C(19)-H(19)	0.9300
C(19)-C(20)	1.362(3)
C(20)-H(20)	0.9300
C(20)-C(21)	1.383(3)
C(21)-H(21)	0.9300
C(1)-N(1)-C(4)	112.52(13)
C(1)-N(1)-C(16)	127.13(13)
C(16)-N(1)-C(4)	120.32(13)
O(1)-C(1)-N(1)	126.06(16)
O(1)-C(1)-C(2)	123.32(15)
N(1)-C(1)-C(2)	110.61(13)
C(1)-C(2)-C(3)	104.91(12)
C(1)-C(2)-C(8)	107.74(13)
C(1)-C(2)-C(15)	107.63(14)
C(8)-C(2)-C(3)	112.76(13)
C(15)-C(2)-C(3)	115.46(14)
C(15)-C(2)-C(8)	107.95(13)
C(4)-C(3)-C(2)	104.62(12)
C(5)-C(3)-C(2)	111.70(12)
C(5)-C(3)-C(4)	109.22(13)
C(14)-C(3)-C(2)	113.89(13)
C(14)-C(3)-C(4)	109.48(13)
C(14)-C(3)-C(5)	107.84(13)
N(1)-C(4)-C(3)	107.20(12)
N(1)-C(4)-H(4A)	110.3
N(1)-C(4)-H(4B)	110.3
C(3)-C(4)-H(4A)	110.3
C(3)-C(4)-H(4B)	110.3
H(4A)-C(4)-H(4B)	108.5
C(3)-C(5)-H(5A)	108.9

C(3)-C(5)-H(5B)	108.9
H(5A)-C(5)-H(5B)	107.7
C(6)-C(5)-C(3)	113.39(13)
C(6)-C(5)-H(5A)	108.9
C(6)-C(5)-H(5B)	108.9
C(7)-C(6)-C(5)	116.60(14)
C(7)-C(6)-C(12)	119.24(17)
C(12)-C(6)-C(5)	124.14(17)
C(6)-C(7)-C(8)	116.60(15)
C(9)-C(7)-C(6)	119.63(17)
C(9)-C(7)-C(8)	123.73(17)
C(2)-C(8)-H(8A)	108.9
C(2)-C(8)-H(8B)	108.9
C(7)-C(8)-C(2)	113.21(13)
C(7)-C(8)-H(8A)	108.9
C(7)-C(8)-H(8B)	108.9
H(8A)-C(8)-H(8B)	107.8
C(7)-C(9)-H(9)	119.2
C(10)-C(9)-C(7)	121.6(2)
C(10)-C(9)-H(9)	119.2
C(9)-C(10)-C(13)	121.3(3)
C(11)-C(10)-C(9)	118.12(19)
C(11)-C(10)-C(13)	120.5(2)
C(10)-C(11)-H(11)	119.4
C(10)-C(11)-C(12)	121.16(19)
C(12)-C(11)-H(11)	119.4
C(6)-C(12)-H(12)	119.9
C(11)-C(12)-C(6)	120.2(2)
C(11)-C(12)-H(12)	119.9
C(10)-C(13)-H(13A)	109.5
C(10)-C(13)-H(13B)	109.5
C(10)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(3)-C(14)-H(14A)	109.5
C(3)-C(14)-H(14B)	109.5
C(3)-C(14)-H(14C)	109.5

H(14A)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(2)-C(15)-H(15A)	109.5
C(2)-C(15)-H(15B)	109.5
C(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(17)-C(16)-N(1)	119.01(14)
C(17)-C(16)-C(21)	118.83(16)
C(21)-C(16)-N(1)	122.16(16)
C(16)-C(17)-H(17)	119.8
C(18)-C(17)-C(16)	120.31(19)
C(18)-C(17)-H(17)	119.8
C(17)-C(18)-H(18)	119.8
C(19)-C(18)-C(17)	120.4(2)
C(19)-C(18)-H(18)	119.8
C(18)-C(19)-H(19)	120.3
C(20)-C(19)-C(18)	119.32(19)
C(20)-C(19)-H(19)	120.3
C(19)-C(20)-H(20)	119.3
C(19)-C(20)-C(21)	121.4(2)
C(21)-C(20)-H(20)	119.3
C(16)-C(21)-H(21)	120.2
C(20)-C(21)-C(16)	119.6(2)
C(20)-C(21)-H(21)	120.2

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 3a.

The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ \ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	55(1)	92(1)	118(1)	33(1)	25(1)	-4(1)
N(1)	46(1)	43(1)	43(1)	5(1)	1(1)	-4(1)
C(1)	45(1)	59(1)	52(1)	5(1)	5(1)	-4(1)
C(2)	45(1)	51(1)	44(1)	1(1)	2(1)	4(1)
C(3)	46(1)	43(1)	47(1)	5(1)	2(1)	0(1)
C(4)	45(1)	49(1)	50(1)	9(1)	5(1)	0(1)
C(5)	57(1)	53(1)	51(1)	14(1)	6(1)	1(1)
C(6)	65(1)	53(1)	40(1)	10(1)	1(1)	8(1)
C(7)	57(1)	53(1)	45(1)	5(1)	-9(1)	8(1)
C(8)	48(1)	63(1)	54(1)	0(1)	-3(1)	9(1)
C(9)	67(1)	65(1)	62(1)	2(1)	-18(1)	4(1)
C(10)	106(2)	60(1)	59(1)	-4(1)	-28(1)	10(1)
C(11)	120(2)	73(1)	50(1)	-8(1)	-4(1)	25(1)
C(12)	89(1)	76(1)	46(1)	6(1)	9(1)	17(1)
C(13)	160(3)	87(2)	95(2)	-19(2)	-50(2)	1(2)
C(14)	61(1)	54(1)	75(1)	-3(1)	0(1)	-7(1)
C(15)	66(1)	74(1)	60(1)	-12(1)	6(1)	8(1)
C(16)	64(1)	42(1)	36(1)	1(1)	-6(1)	-6(1)
C(17)	66(1)	52(1)	65(1)	7(1)	-8(1)	2(1)
C(18)	95(2)	55(1)	77(1)	5(1)	-19(1)	12(1)
C(19)	147(2)	45(1)	52(1)	4(1)	-16(1)	3(1)
C(20)	135(2)	55(1)	55(1)	11(1)	11(1)	-18(1)
C(21)	88(1)	54(1)	53(1)	7(1)	10(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 3a.

	x	y	z	U(eq)
H(4A)	9891	7427	6004	57
H(4B)	10677	8102	6540	57
H(5A)	8031	10316	5431	64
H(5B)	9816	9298	5439	64
H(8A)	3455	8802	5937	66
H(8B)	4500	10044	5699	66
H(9)	2654	7253	5214	79
H(11)	6757	6301	4255	98
H(12)	8999	7737	4711	84
H(13A)	2181	5272	4610	177
H(13B)	3715	5041	4197	177
H(13C)	2179	6235	4125	177
H(14A)	8926	11282	6296	96
H(14B)	10923	10488	6262	96
H(14C)	9754	10338	6757	96
H(15A)	4397	9974	6785	100
H(15B)	5698	11064	6532	100
H(15C)	6640	10163	6994	100
H(17)	11453	5901	6596	75
H(18)	12407	3811	6939	93
H(19)	10187	2501	7350	99
H(20)	7048	3300	7429	98
H(21)	6030	5370	7083	78

Table 6. Torsion angles [°] for 3a.

O(1)-C(1)-C(2)-C(3)	-179.05(17)
O(1)-C(1)-C(2)-C(8)	-58.7(2)
O(1)-C(1)-C(2)-C(15)	57.5(2)
N(1)-C(1)-C(2)-C(3)	1.86(17)
N(1)-C(1)-C(2)-C(8)	122.22(14)
N(1)-C(1)-C(2)-C(15)	-121.60(15)
N(1)-C(16)-C(17)-C(18)	-179.02(15)
N(1)-C(16)-C(21)-C(20)	179.41(15)
C(1)-N(1)-C(4)-C(3)	4.06(17)
C(1)-N(1)-C(16)-C(17)	178.36(15)
C(1)-N(1)-C(16)-C(21)	-2.3(2)
C(1)-C(2)-C(3)-C(4)	0.56(15)
C(1)-C(2)-C(3)-C(5)	118.60(14)
C(1)-C(2)-C(3)-C(14)	-118.93(14)
C(1)-C(2)-C(8)-C(7)	-71.15(18)
C(2)-C(3)-C(4)-N(1)	-2.62(15)
C(2)-C(3)-C(5)-C(6)	-46.56(18)
C(3)-C(2)-C(8)-C(7)	44.12(19)
C(3)-C(5)-C(6)-C(7)	47.45(19)
C(3)-C(5)-C(6)-C(12)	-133.75(17)
C(4)-N(1)-C(1)-O(1)	177.17(17)
C(4)-N(1)-C(1)-C(2)	-3.77(18)
C(4)-N(1)-C(16)-C(17)	0.5(2)
C(4)-N(1)-C(16)-C(21)	179.88(14)
C(4)-C(3)-C(5)-C(6)	68.68(17)
C(5)-C(3)-C(4)-N(1)	-122.33(13)
C(5)-C(6)-C(7)-C(8)	0.8(2)
C(5)-C(6)-C(7)-C(9)	178.45(14)
C(5)-C(6)-C(12)-C(11)	-177.39(16)
C(6)-C(7)-C(8)-C(2)	-47.4(2)
C(6)-C(7)-C(9)-C(10)	-1.1(3)
C(7)-C(6)-C(12)-C(11)	1.4(3)
C(7)-C(9)-C(10)-C(11)	1.5(3)
C(7)-C(9)-C(10)-C(13)	-177.63(19)
C(8)-C(2)-C(3)-C(4)	-116.41(14)
C(8)-C(2)-C(3)-C(5)	1.63(18)

C(8)-C(2)-C(3)-C(14)	124.10(15)
C(8)-C(7)-C(9)-C(10)	176.40(16)
C(9)-C(7)-C(8)-C(2)	135.06(17)
C(9)-C(10)-C(11)-C(12)	-0.5(3)
C(10)-C(11)-C(12)-C(6)	-0.9(3)
C(12)-C(6)-C(7)-C(8)	-178.06(15)
C(12)-C(6)-C(7)-C(9)	-0.4(2)
C(13)-C(10)-C(11)-C(12)	178.6(2)
C(14)-C(3)-C(4)-N(1)	119.80(14)
C(14)-C(3)-C(5)-C(6)	-172.43(13)
C(15)-C(2)-C(3)-C(4)	118.85(15)
C(15)-C(2)-C(3)-C(5)	-123.12(15)
C(15)-C(2)-C(3)-C(14)	-0.64(19)
C(15)-C(2)-C(8)-C(7)	172.88(15)
C(16)-N(1)-C(1)-O(1)	-0.8(3)
C(16)-N(1)-C(1)-C(2)	178.23(13)
C(16)-N(1)-C(4)-C(3)	-177.79(12)
C(16)-C(17)-C(18)-C(19)	-0.7(3)
C(17)-C(16)-C(21)-C(20)	-1.2(3)
C(17)-C(18)-C(19)-C(20)	-0.6(3)
C(18)-C(19)-C(20)-C(21)	1.0(3)
C(19)-C(20)-C(21)-C(16)	-0.1(3)
C(21)-C(16)-C(17)-C(18)	1.6(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for 3a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)