Cyclization/hydrolysis of 1,5-enenitriles initiated by sulfonyl radicals in aqueous phase under I_2/TBHP system

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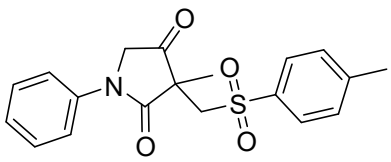
Supporting Information

List of Contents

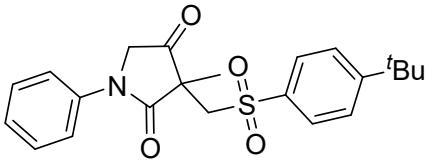
(A) Typical experimental procedure for the cyclization/hydrolysis

To a Schlenk tube were added 1,5-enenitrile **1** (0.2 mmol), sulfonyl hydrazide **2** (2.0 equiv.), I_2 (10 mol%) and TBHP (2.0 equiv.). Then the tube was stirred at 85 ℃ sealed in N_2 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 = 2:1) to afford the desired product **3**. This reaction can not be directly precipitated and filtered to obtain the produces because of the following reason: There are a bit byproducts produced in the transformation, so the precipitation and filtration produce a mixture that cannot be purified.

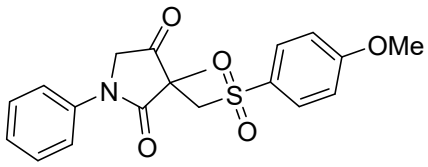
(B) Analytical data



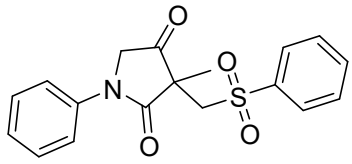
3-Methyl-1-phenyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3a), white solid (0.0659 g, 92% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.71-7.67 (m, 4H), 7.44 (t, J = 7.5 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 7.5 Hz, 1H), 4.63 (d, J = 17.0 Hz, 1H), 4.40 (d, J = 17.0 Hz, 1H), 3.87 (d, J = 14.5 Hz, 1H), 3.73 (d, J = 14.5 Hz, 1H), 2.42 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.8, 171.2, 145.1, 137.9, 136.9, 129.9, 129.2, 127.8, 125.7, 120.9, 61.7, 56.3, 50.0, 21.6 (2); HRMS *m/z* (ESI) calcd for $C_{19}H_{20}NO_4S([M+H]^+)$ 358.1108, found 358.1112.



3-(((4-(*tert*-Butyl)phenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3b), white solid (0.0728 g, 91% yield); ^{1}H NMR (500 MHz, $CDCl_3$) *δ:* 7.75-7.74 (m, 2H), 7.67-7.65 (m, 2H), 7.50-7.49 (m, 2H), 7.44 (t, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.5 Hz, 1H), 4.64 (d, *J* = 17.0 Hz, 1H), 4.40 (d, *J* = 17.5 Hz, 1H), 3.88 (d, *J* = 14.5 Hz, 1H), 3.73 (d, *J* = 14.5 Hz, 1H), 1.37 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 205.8, 171.2, 158.0, 137.9, 136.7, 129.2, 127.6, 126.3, 125.7, 120.9, 61.7, 56.3, 49.9, 35.3, 31.0, 21.6; HRMS *m/z* (ESI) calcd for $C_{22}H_{26}NO_4S$ ($[M+H]^+$) 400.1577, found 400.1581.

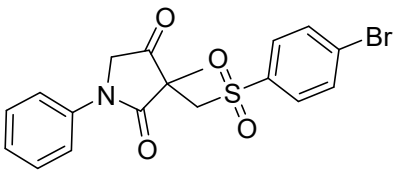


3-(((4-Methoxyphenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3c), white solid (0.0694 g, 93% yield,); ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7,75 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.44 (t, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 7.0 Hz, 1H), 6.94 (d, *J* = 8.5 Hz, 2H), 4.63 (d, *J* = 17.5 Hz, 1H), 4.39 (d, *J* = 17.0 Hz, 1H), 3.88 (d, *J* = 14.0 Hz, 1H), 3.85 (s, 3H), 3.73 (d, *J* = 14.0 Hz, 1H), 1.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 205.8, 171.3, 164.0, 137.9, 131.3, 130.0, 129.2, 125.7, 120.9, 114.5, 62.0, 56.3, 55.7, 50.0, 21.6; HRMS *m/z* (ESI) calcd for $C_{19}H_{20}NO_5S$ ($[M+H]^+$) 374.1057, found 374.1051.

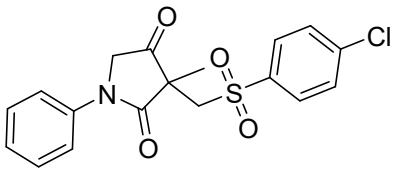


3-Methyl-1-phenyl-3-((phenylsulfonyl)methyl)pyrrolidine-2,4-dione (3d), white solid (0.0597 g, 87% yield,); ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7.84 (d, *J* = 7.5 Hz, 2H), 7.68-7.62 (m, 3H), 7.51 (t, *J* = 8.0 Hz, 2H), 7.44 (t, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 3.0 Hz, 1H), 4.63 (d, *J* = 17.5 Hz, 1H), 4.41 (d, *J* =

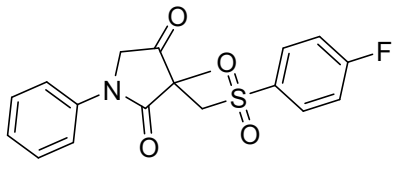
17.0 Hz, 1H), 3.89 (d, *J* = 14.5 Hz, 1H), 3.75 (d, *J* = 14.5 Hz, 1H), 1.37 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 205.8, 171.2, 139.8, 137.8, 134.0, 129.3, 129.1, 127.7, 125.8, 120.9, 61.6, 56.3, 50.0, 21.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{18}NO_4S$ ($[M+H]^+$) 344.0951, found 344.0955.



3-(((4-Bromophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3e), white solid (0.0717 g, 85% yield); ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7.69-7.66 (m, 3H), 7.65-7.62 (m, 3H), 7.45 (t, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 3.5 Hz, 1H), 4.60 (d, *J* = 17.5 Hz, 1H), 4.41 (d, *J* = 17.5 Hz, 1H), 3.89 (d, *J* = 14.5 Hz, 1H), 3.76 (d, *J* = 14.5 Hz, 1H), 1.37 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 205.5, 171.1, 138.8, 137.7, 132.6, 129.3, 129.2, 128.0, 125.9, 120.8, 61.5, 56.2, 50.1, 21.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{17}BrNO_4S$ ($[M+H]^+$) 422.0056, found 422.0050.

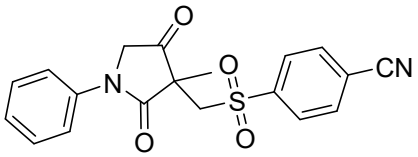


3-(((4-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3f), white solid (0.0605 g, 80% yield,); ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7.77-7.75 (m, 2H), 7.68-7.65 (m, 2H), 7.48-7.44 (m, 4H), 7.27 (t, *J* = 4.5 Hz, 1H), 4.61 (d, *J* = 17.0 Hz, 1H), 4.41 (d, *J* = 17.0 Hz, 1H), 3.89 (d, *J* = 14.5 Hz, 1H), 3.76 (d, *J* = 14.5 Hz, 1H), 1.38 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 205.6, 171.1, 140.9, 138.3, 137.8, 129.6, 129.3 (2), 125.9, 120.8, 61.6, 56.2, 50,2, 21.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{17}ClNO_4S$ ($[M+H]^+$) 378.0561, found 378.0567.

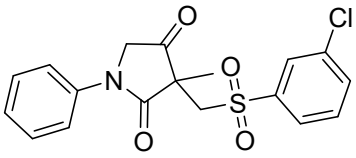


3-(((4-Fluorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3g), white solid

(0.0543 g, 75% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.86-7.83 (m, 2H), 7.66 (d, J = 8.5 Hz, 2H), 7.44 (t, J = 8.0 Hz, 2H), 7.27 (t, J = 4.0 Hz, 1H), 7.17 (t, J = 8.5 Hz, 2H), 4.62 (d, J = 17.5 Hz, 1H), 4.42 (d, J = 17.5 Hz, 1H), 3.90 (d, J = 14.5 Hz, 1H), 3.76 (d, J = 14.5 Hz, 1H), 1.37 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.6, 171.2, 165.9 (d, $J_{C\text{-}F}$ = 255.5 Hz), 137.7, 135.8 (d, $J_{C\text{-}F}$ = 3.1 Hz), 130.7 (d, $J_{C\text{-}F}$ = 9.8 Hz), 129.2, 125.9, 120.9, 116.6 (d, $J_{C\text{-}F}$ = 22.6 Hz), 61.6, 56.2, 50.1, 21.5; ^{19}F NMR (471 MHz, $CDCl_3$) δ: -102.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{17}FNO_4S$ ($[M+H]^+$) 362.0857, found 362.0853.

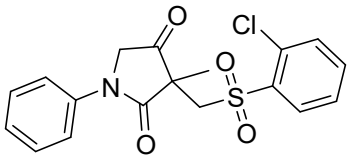


4-(((3-Methyl-2,4-dioxo-1-phenylpyrrolidin-3-yl)methyl)sulfonyl)benzonitrile (3h), white solid (0.0517 g, 70 % yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95-7.94 (m, 2H), 7.80-7.78 (m, 2H), 7.66-7.64 (m, 2H), 7.47 (t, J = 8.0 Hz, 2H), 7.29 (t, J = 7.5 Hz, 1H), 4.62 (d, J = 17.0 Hz, 1H), 4.43 (d, J = 17.5 Hz, 1H), 3.92 (d, J = 15.0 Hz, 1H), 3.82 (d, J = 15.0 Hz, 1H), 1.39 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.2, 171.0, 143.9, 137.7, 133.0, 129.4, 128.5, 126.0, 120.7, 117.8, 117.0, 61.2, 56.2, 50.3, 21.6; HRMS *m/z* (ESI) calcd for $C_{19}H_{17}N_2O_4S$ ($[M+H]^+$) 369.0904, found 369.0900.

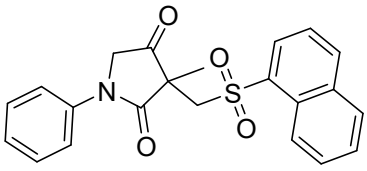


3-(((3-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3i), white solid (0.0597 g, 79% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.82 (t, J = 2.0 Hz, 2H), 7.73-7.71 (m, 1H), 7.68-7.66 (m, 2H), 7.62-7.60 (m, 1H), 7.48-7.43 (m, 3H), 7.27 (d, J = 6.5 Hz, 1H), 4.62 (d, J = 17.5 Hz, 1H), 4.43 (d, J = 17.5 Hz, 1H), 3.90 (d, J = 14.5 Hz, 1H), 3.77 (d, J = 14.5 Hz, 1H), 1.38 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.5, 171.0,

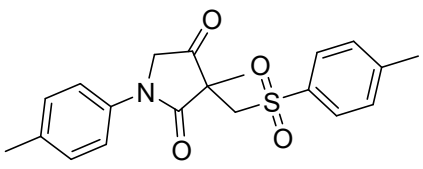
141.5, 137.7, 135.5, 134.2, 130.6, 129.3, 127.9, 125.9 (2), 120.9, 61.4, 56.3, 50.1, 21.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{17}ClNO_4S$ ([M+H]$^+$) 378.0561, found 378.0567.



3-(((2-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3j), white solid (0.0590 g, 78% yield,); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.82-7.80 (m, 1H), 7.59-7.57 (m, 2H), 7.55-7.51 (m, 2H), 7.41 (t, J = 8.0 Hz, 1H), 7.26-7.22 (m, 2H), 4.57 (d, J = 17.5 Hz, 1H), 4.37 (d, J = 17.0 Hz, 1H), 4.26 (d, J = 15.0 Hz, 1H), 3.96 (d, J = 15.0 Hz, 1H), 1.38 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.3, 170.7, 137.7, 137.4, 135.0, 132.5, 131.8, 130.7, 129.1, 127.2, 125.6, 120.5, 59.6, 56.0, 49.9, 21.6; HRMS *m/z* (ESI) calcd for $C_{18}H_{17}ClNO_4S$ ([M+H]$^+$) 378.0561, found 378.0557.

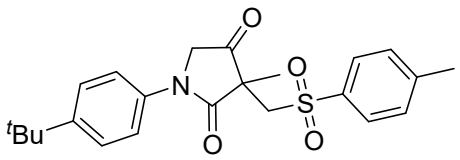


3-Methyl-3-((naphthalen-1-ylsulfonyl)methyl)-1-phenylpyrrolidine-2,4-dione (3k), white solid (0.0638 g, 81% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.36 (s, 1H), 7.98 (d, J = 8.5 Hz, 1H), 7.91 (d, J = 8.5 Hz, 1H), 7.82-7.80 (m, 2H), 7.65 (t, J = 7.0 Hz, 3H), 7.58 (t, J = 15.0 Hz, 1H), 7.41 (t, J = 8.0 Hz, 2H), 7.24 (d, J = 7.0 Hz, 1H), 4.68 (d, J = 17.0 Hz, 1H), 4.42 (d, J = 17.5 Hz, 1H), 3.96 (d, J = 14.5 Hz, 1H), 3.82 (d, J = 14.5 Hz, 1H), 1.37 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.8, 171.1, 137.9, 136.5, 135.4, 131.9, 129.8 (2), 129.5, 129.4, 129.2, 128.0, 127.7, 125.7, 122.1, 120.8, 61.6, 56.3, 50.0, 21.7; HRMS *m/z* (ESI) calcd for $C_{22}H_{20}NO_4S$ ([M+H]$^+$) 394.1108, found 394.1114.

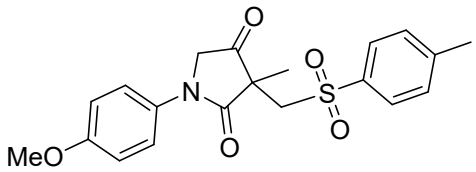


3-Methyl-1-(*p*-tolyl)-3-(tosylmethyl)pyrrolidine-2,4-dione (3m), white solid (0.0670 g, 90% yield,

d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.70 (d, J = 8.0 Hz, 2H), 7.53 (d, J = 8.5 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 4.58 (d, J = 17.5 Hz, 1H), 4.37 (d, J = 17.0 Hz, 1H), 3.85 (d, J = 14.5 Hz, 1H), 3.71 (d, J = 14.5 Hz, 1H), 2.42 (s, 3H), 2.37 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 206.0, 171.0, 145.0, 136.9, 135.5, 135.3, 129.8, 129.6, 127.7, 120.9, 61.6, 56.4, 49.8, 21.6 (2), 20.9; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}NO_4S$ ([M+H]$^+$) 372.1264, found 372.1260.

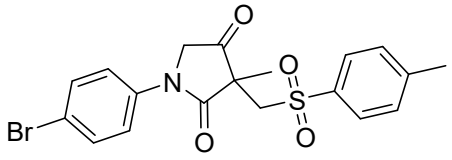


1-(4-(*tert*-Butyl)phenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3n), white solid (0.0712 g, 86% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.4 Hz, 2H), 7.60 (d, J = 8.8 Hz, 2H), 7.45 (d, J = 8.8 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 4.61 (d, J = 17.6 Hz, 1H), 4.39 (d, J = 17.2 Hz, 1H), 3.86 (d, J = 14.4 Hz, 1H), 3.72 (d, J = 14.4 Hz, 1H), 2.42 (s, 3H), 1.35 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 206.0, 171.0, 148.7, 145.1, 136.8, 135.2, 129.9, 127.7, 126.0, 120.5, 61.6, 56.3, 49.9, 34.4, 31.2, 21.6 (2); HRMS *m/z* (ESI) calcd for $C_{23}H_{28}NO_4S$ ([M+H]$^+$) 414.1734, found 414.1730.

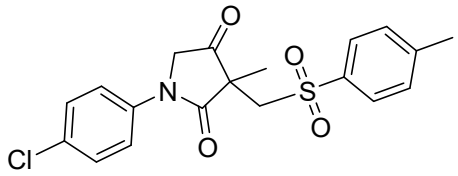


1-(4-Methoxyphenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3o), white solid (0.0730 g, 94% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.5 Hz, 2H), 7.57-7.55 (m, 2H), 7.29 (d, J = 8.0 Hz, 2H), 6.97-6.95 (m, 2H), 4.57 (d, J = 17.0 Hz, 1H), 4.36 (d, J = 17.5 Hz, 1H), 3.84 (t, J = 7.5 Hz, 4H), 3.71 (d, J = 14.5 Hz, 1H), 2.42 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 206.1, 170.9, 157.4, 145.1, 136.9, 130.9, 129.9, 127.7, 122.9, 114.3,

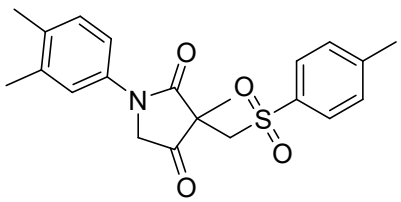
61.7, 56.8, 55.5, 49.6, 21.6, 21.5; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}NO_5S$ ($[M+H]^+$) 388.1213, found 388.1219.



1-(4-Bromophenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3p), white solid (0.0698 g, 80% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.4 Hz, 2H), 7.60-7.54 (m, 4H), 7.31 (d, J = 8.0 Hz, 2H), 4.60 (d, J = 17.2 Hz, 1H), 4.37 (d, J = 17.2 Hz, 1H), 3.85 (d, J = 14.4 Hz, 1H), 3.73 (d, J = 14.4 Hz, 1H), 2.44 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 205.2, 171.4, 145.3, 137.0, 136.7, 132.2, 130.0, 127.7, 122.3, 118.8, 61.8, 56.1, 49.9, 21.7, 21.5; HRMS *m/z* (ESI) calcd for $C_{19}H_{19}BrNO_4S$ ($[M+H]^+$) 436.0213, found 436.0209.

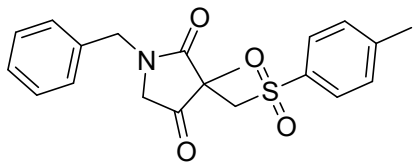


1-(4-Chlorophenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3q), white solid (0.0620 g, 79% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.0 Hz, 2H), 7.64 (d, J = 9.0 Hz, 2H), 7.41-7.39 (m, 2H), 7.30 (d, J = 8.0 Hz, 2H), 4.60 (d, J = 17.0 Hz, 1H), 4.37 (d, J = 17.0 Hz, 1H), 3.85 (d, J = 14.5 Hz, 1H), 3.72 (d, J = 14.0 Hz, 1H), 2.43 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 205.3, 171.4, 145.3, 136.8, 136.5, 131.0, 129.9, 129.2, 127.7, 122.1, 61.8, 56.2, 49.9, 21.7, 21.5; HRMS *m/z* (ESI) calcd for $C_{19}H_{19}ClNO_4S$ ($[M+H]^+$) 392.0718, found 392.0726.

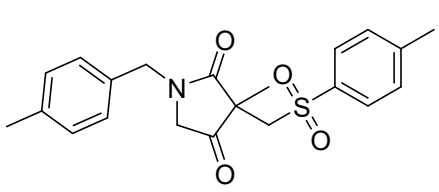


1-(3,4-Dimethylphenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3r), white solid (0.0687 g, 89 % yield); ^{1}H NMR (500 MHz,

$CDCl_3$) *δ:* 7.71 (d, *J* = 8.5 Hz, 2H), 7.46 (s, 1H), 7.34-7.32 (m, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 1H), 4.59 (d, *J* = 17.5 Hz, 1H), 4.36 (d, *J* = 17.5 Hz, 1H), 3.85 (d, *J* = 14.0 Hz, 1H), 3.71 (d, *J* = 14.0 Hz, 1H), 2.42 (s, 3H), 2.30 (s, 3H), 2.27 (s, 3H), 1.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*: 206.2, 171.0, 145.0, 137.5, 136.9, 135.6, 134.4, 130.1, 129.9, 127.8, 122.4, 118.5, 61.7, 56.6, 49.8, 21.6 (2), 20.0, 19.3; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO_4S$ ($[M+H]^+$) 386.1421, found 386.1427.

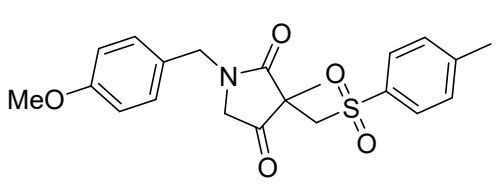


1-Benzyl-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3s), white solid (0.0625 g, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) *δ*: 7.74 (d, *J* = 8.4 Hz, 2H), 7.36 (t, *J* = 7.2 Hz, 7H), 4.81 (d, *J* = 14.8 Hz, 1H), 4.64 (d, *J* = 14.8 Hz, 1H), 4.04 (d, *J* = 17.6 Hz, 1H), 3.81 (d, *J* = 11.6 Hz, 1H), 3.77 (d, *J* = 18.0 Hz, 1H), 3.64 (d, *J* = 14.4 Hz, 1H), 2.45 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) *δ*: 206.7, 172.1, 145.1, 137.1, 134.8, 129.9, 128.9, 128.3, 128.0, 127.7, 61.2, 54.3, 48.6, 46.3, 21.7, 21.5; HRMS *m/z* (ESI) calcd for $C_{20}H_{22}NO_4S$ ($[M+H]^+$) 372.1264, found 372.1268.

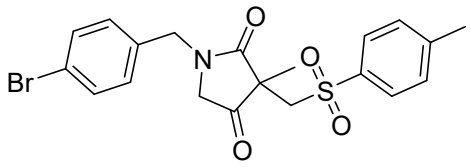


3-Methyl-1-(4-methylbenzyl)-3-(tosylmethyl)pyrrolidine-2,4-dione (3t), white solid (0.0626 g, 81% yield); ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7.73 (d, *J* = 8.0 Hz, 2H), 7.37-7.31 (m, 3H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 2.0 Hz, 1H), 4.77 (d, *J* = 14.5 Hz, 1H), 4.57 (d, *J* = 14.5 Hz, 1H), 4.01 (d, *J* = 17.5 Hz, 1H), 3.80 (d, *J* = 14.5 Hz, 1H), 3.74 (d, *J* = 17.5 Hz, 1H), 3.63 (d, *J* = 14.5 Hz, 1H), 2.44 (s, 3H), 2.34 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) *δ*:

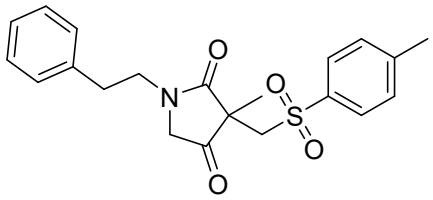
206.7, 172.0, 145.1, 137.8, 137.2, 131.8, 129.9, 129.6, 128.4, 127.7, 61.3, 54.3, 48.7, 46.1, 21.7, 21.5, 21.1; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO_4S$ ($[M+H]^+$) 386.1421, found 386.1427.



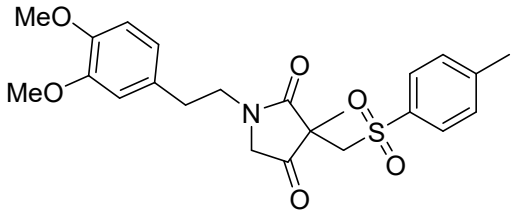
1-(4-Methoxybenzyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3u), white solid (0.0611 g, 76% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 7.27 (t, J = 4.0 Hz, 2H), 6.89 (d, J = 8.4 Hz, 2H), 4.70 (d, J = 14.8 Hz, 1H), 4.60 (d, J = 14.8 Hz, 1H), 4.01 (d, J = 18.0 Hz, 1H), 3.79 (s, 3H), 3.77 (d, J = 1.6 Hz, 1H), 3.74 (d, J = 3.6 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 2.44 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 206.8, 172.0, 159.3, 145.1, 137.0, 129.9, 129.7, 127.7, 126.7, 114.2, 61.1, 55.2, 54.2, 48.7, 45.7, 21.6, 21.5; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO_5S$ ($[M+H]^+$) 402.1370, found 402.1374.



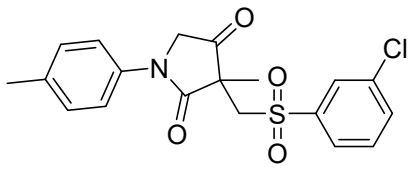
1-(4-Bromobenzyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3v), white solid (0.0630 g, 70% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73 (d, J = 8.0 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 7.27 (s, 1H), 7.24 (d, J = 3.6, 1H), 4.74 (d, J = 14.8 Hz, 1H), 4.62 (d, J = 15.2 Hz, 1H), 4.03 (d, J = 17.6 Hz, 1H), 3.82-3.75 (m, 2H), 3.64 (d, J = 14.4 Hz, 1H), 2.45 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 206.4, 172.2, 145.2, 137.0 133.9, 132.0, 130.1, 129.9, 127.7, 122.1, 61.3, 54.3, 48.5, 45.7, 21.7, 21.5; HRMS *m/z* (ESI) calcd for $C_{20}H_{21}BrNO_4S$ ($[M+H]^+$) 450.0369, found 450.0373.



3-Methyl-1-phenethyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3w), white solid (0.0664 g, 86% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73-7.67 (m, 2H), 7.32-7.27 (m, 7H), 4.09-3.94 (m, 2H), 3.74-3.57 (m, 4H), 3.03-2.93 (m, 2H), 2.43 (s, 3H), 1.11 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 207.0, 172.0, 145.0, 138.1, 137.0, 129.8, 128.6 (2), 127.6, 126.6, 61.2, 55.3, 48.5, 43.5, 33.2, 21.6, 21.2; HRMS *m/z* (ESI) calcd for $C_{21}H_{24}NO_4S$ ($[M+H]^+$) 386.1421, found 386.1425.

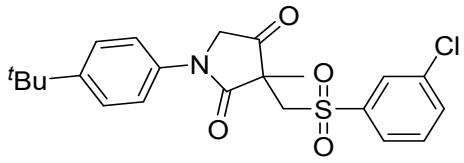


1-(3,4-Dimethoxyphenethyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3x), yellow oil (0.0741 g, 83% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 8.4 Hz, 2H), 6.81 (s, 1H), 6.80-6.76 (m, 2H), 4.08 (d, J = 17.6 Hz, 1H), 3.86 (s, 3H), 3.84 (s, 3H), 3.79-3.73 (m, 3H), 3.69-3.64 (m, 1H), 3.59 (d, J = 14.4 Hz, 1H), 3.00-2.87 (m, 2H), 2.43 (s, 3H), 1.13 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 207.0, 172.0, 148.9, 147.6, 145.0, 137.0, 130.6, 129.8, 127.5, 120.5, 111.7, 111.1, 61.1, 55.8, 55.7 (2), 55.3, 48.4, 43.5, 32.8, 21.5, 21.2; HRMS *m/z* (ESI) calcd for $C_{23}H_{28}NO_6S$ ($[M+H]^+$) 446.1632, found 446.1638.

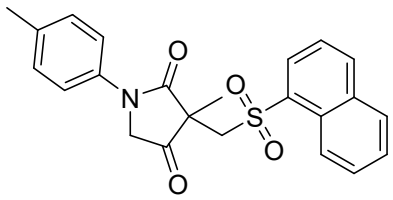


3-(((3-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-(*p*-tolyl)pyrrolidine-2,4-dione (3z), white solid (0.0659 g, 84% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (t, J =2.0 Hz, 1H), 7.73-7.71 (m, 1H), 7.61-7.59 (m, 1H), 7.53 (t, J = 4.4 Hz,

2H), 7.46 (t, J = 4.0 Hz, 1H), 7.24 (d, J = 8.4 Hz, 2H), 4.58 (d, J = 17.6 Hz, 1H), 4.39 (d, J = 17.6 Hz, 1H), 3.89 (d, J = 12.8 Hz, 1H), 3.76 (d, J = 14.4 Hz, 1H), 2.37 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 205.8, 170.9, 141.4, 135.8, 135.5, 135.1, 134.2, 130.6, 129.8, 127.8, 125.9, 120.9, 61.3, 56.4, 50.0, 21.6, 20.9; HRMS *m/z* (ESI) calcd for $C_{19}H_{19}ClNO_4S$ ($[M+H]^+$) 392.0718, found 392.0724.

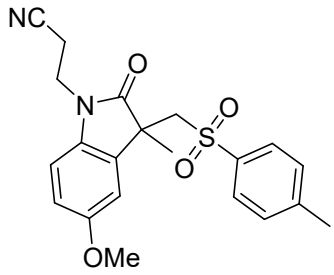


1-(4-(*tert*-Butyl)phenyl)-3-(((3-chlorophenyl)sulfonyl)methyl)-3-methylpyrrolidine-2,4-dione (3aa), white solid (0.0695 g, 80% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (t, J = 2.0 Hz, 1H), 7.72 (d, J = 2.8 Hz, 1H), 7.60-7.58 (m, 3H), 7.46 (t, J = 4.4 Hz, 3H), 4.60 (d, J = 17.2 Hz, 1H), 4.41 (d, J = 17.6 Hz, 1H), 3.91 (s, 1H), 3.76 (d, J = 14.8 Hz, 1H), 1.37 (s, 3H), 1.34 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 205.8, 170.9, 149.0, 141.6, 135.2, 134.3, 130.7, 128.9, 127.9, 126.2, 126.0, 120.6, 61.4, 56.3, 50.1, 34.5, 31.3, 21.6; HRMS *m/z* (ESI) calcd for $C_{22}H_{25}ClNO_4S$ ($[M+H]^+$) 434.1187, found 434.1183.

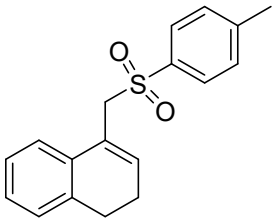


3-Methyl-3-((naphthalen-1-ylsulfonyl)methyl)-1-(*p*-tolyl)pyrrolidine-2,4-dione (3ab), white solid (0.0677 g, 83% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.35 (s, 1H), 7.98-7.89 (m, 3H), 7.82-7.79 (m, 2H), 7.66 (t, J = 7.6 Hz, 1H), 7.51 (d, J = 8.4 Hz, 2H), 7.19 (d, J = 8.4 Hz, 2H), 4.65 (d, J = 17.2 Hz, 1H), 4.40 (d, J = 17.6 Hz, 1H), 3.95 (d, J = 14.4 Hz, 1H), 3.80 (d, J = 14.4 Hz, 1H), 2.37 (s, 3H), 1.36 (s, 3H); ^{13}C (101 MHz, $CDCl_3$) δ: 206.0, 170.8, 136.4, 135.5, 135.3 (2), 131.1, 129.8 (2),

129.7, 129.5, 129.4, 127.9, 127.6, 122.1, 120.7, 61.5, 56.4, 49.8, 21.6, 20.9; HRMS *m/z* (ESI) calcd for $C_{23}H_{22}NO_4S$ ($[M+H]^+$) 408.1264, found 408.1260.



3-(5-Methoxy-3-methyl-2-oxo-3-(tosylmethyl)indolin-1-yl)propanenitrile (4a), yellow oil (0.0510 g, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.29 (t, J = 4.0 Hz, 2H), 7.11 (d, J = 8.0 Hz, 2H), 6.91 (d, J = 8.4 Hz, 1H), 6.77-6.74 (m, 1H), 6.21 (d, J = 2.4 Hz, 1H), 4.22-4.15 (m, 1H), 3.92-3.88 (m, 2H), 3.71 (d, J = 14.8 Hz, 1H), 3.56 (s, 3H), 2.89-2.84 (m, 2H), 2.37 (s, 3H), 1.34 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 177.4, 155.8, 144.1, 136.8, 134.6, 130.0, 129.4, 127.4, 118.0, 113.1, 110.4, 108.9, 61.9, 55.0, 45.5, 36.0, 25.1, 21.3, 15.7; HRMS *m/z* (ESI) calcd for $C_{21}H_{23}N_2O_4S$ ($[M+H]^+$) 399.1373, found 399.1377.



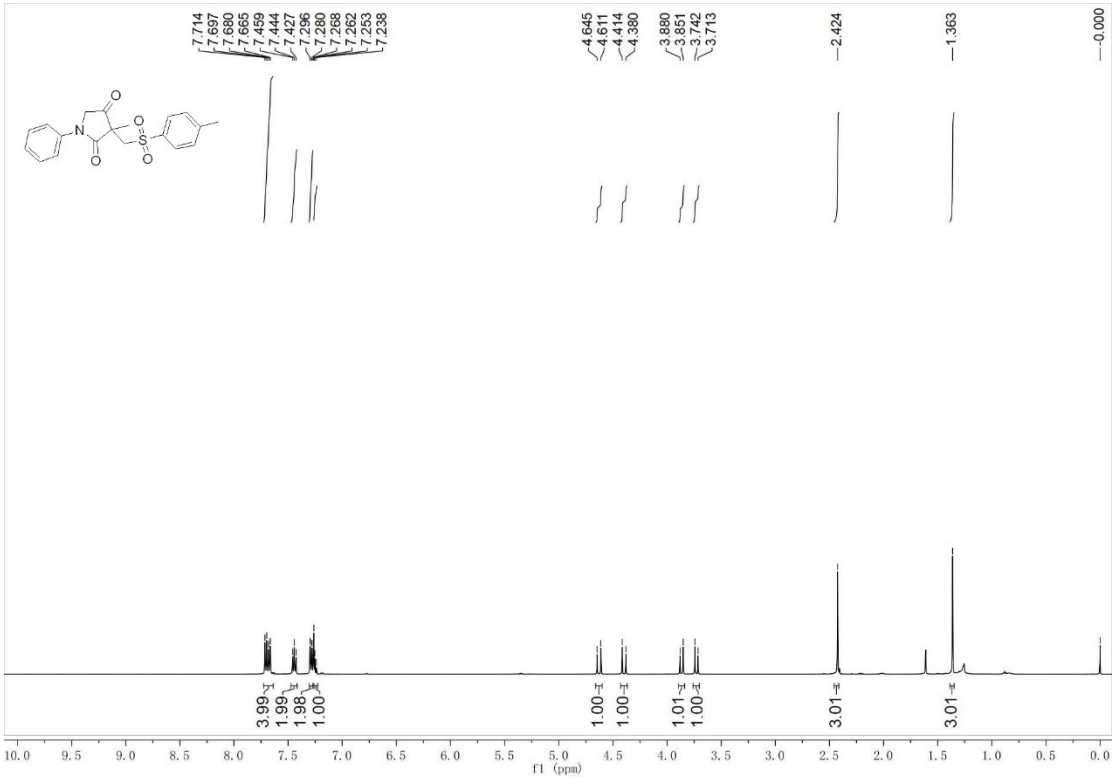
4-(Tosylmethyl)-1,2-dihydronaphthalene (5a),[1] (0.0533 g, 89% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.78 (d, J = 8.5 Hz, 2H), 7.29 (t, J = 4.0 Hz, 5H), 7.25-7.22 (m, 1H), 5.58-5.54 (m, 1H), 4.11 (t, J = 7.0 Hz, 2H), 2.58-2.54 (m, 2H), 2.42 (s, 3H), 1.98 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 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.

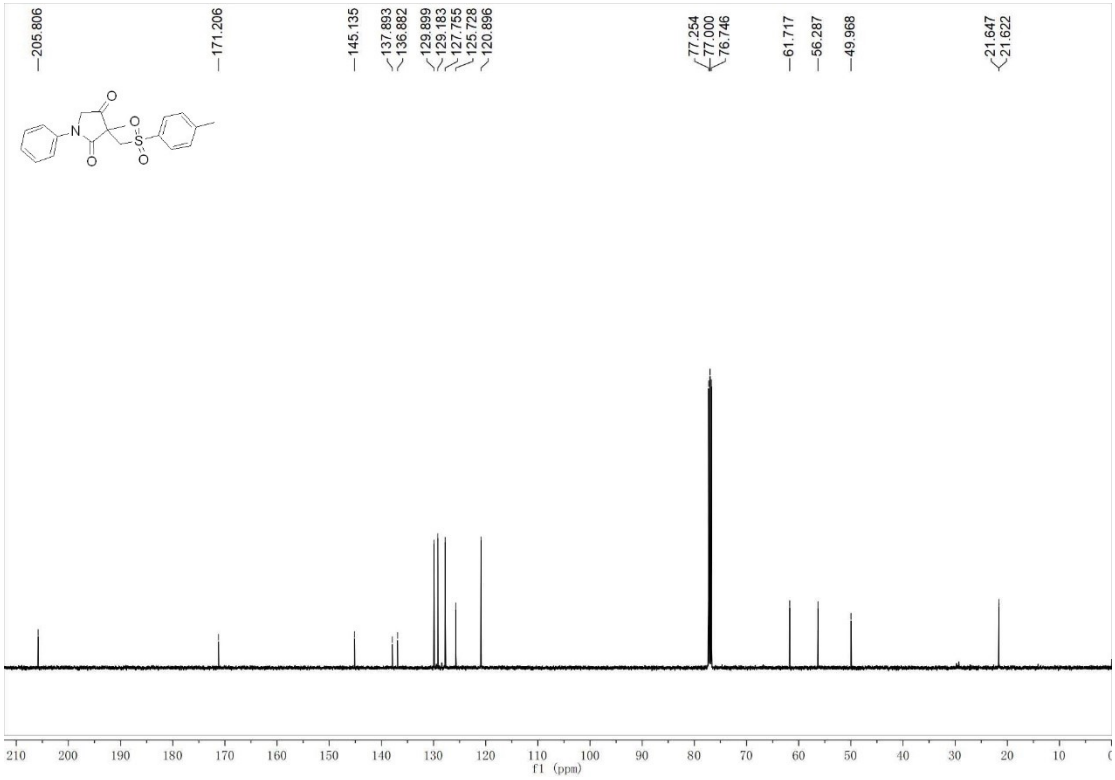
(C) Reference

[1] P. Chen, Q. Zhou, Z. Chen, Y.-K. Liu, K.-W. Tang and Y. Liu, *Org. Biomol. Chem.*, 2020, **18**, 7345.

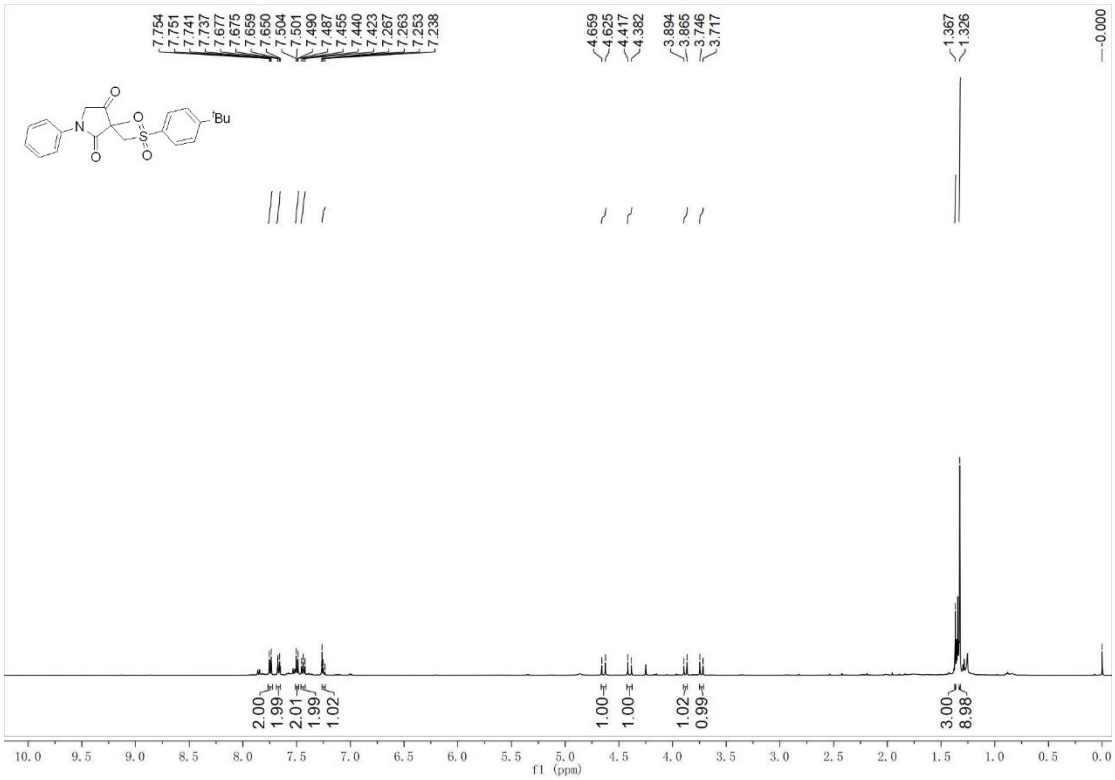
(D) Spectra

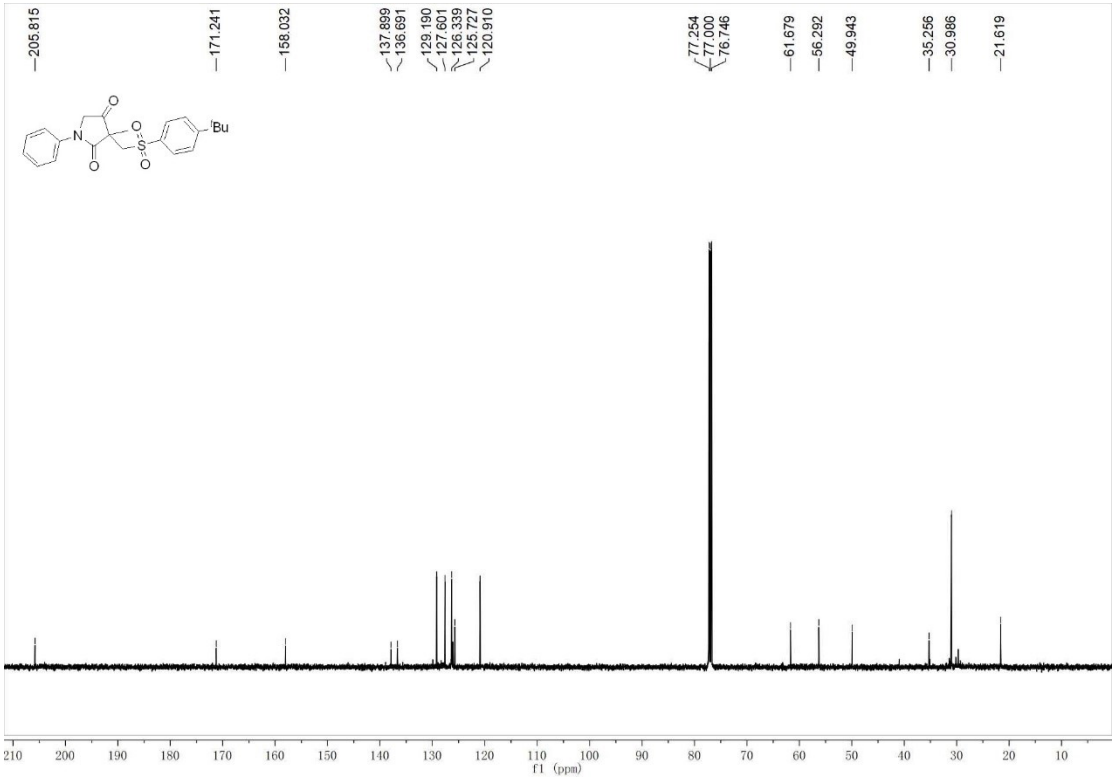
3-Methyl-1-phenyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3a)



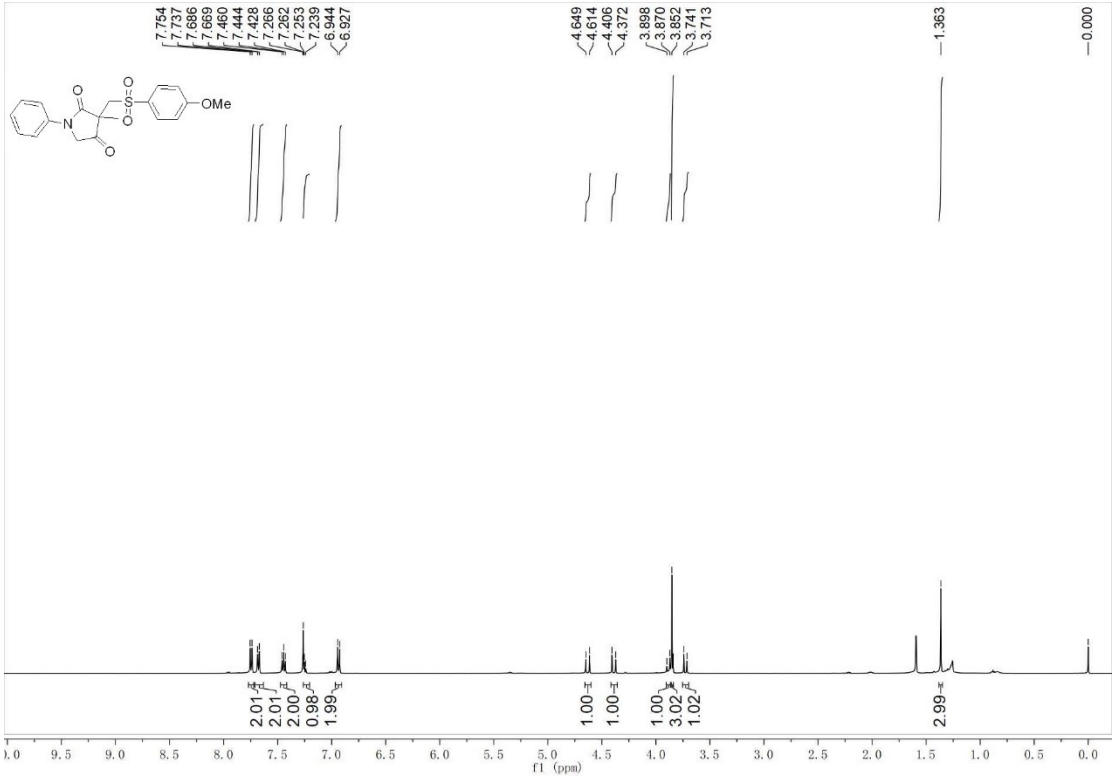


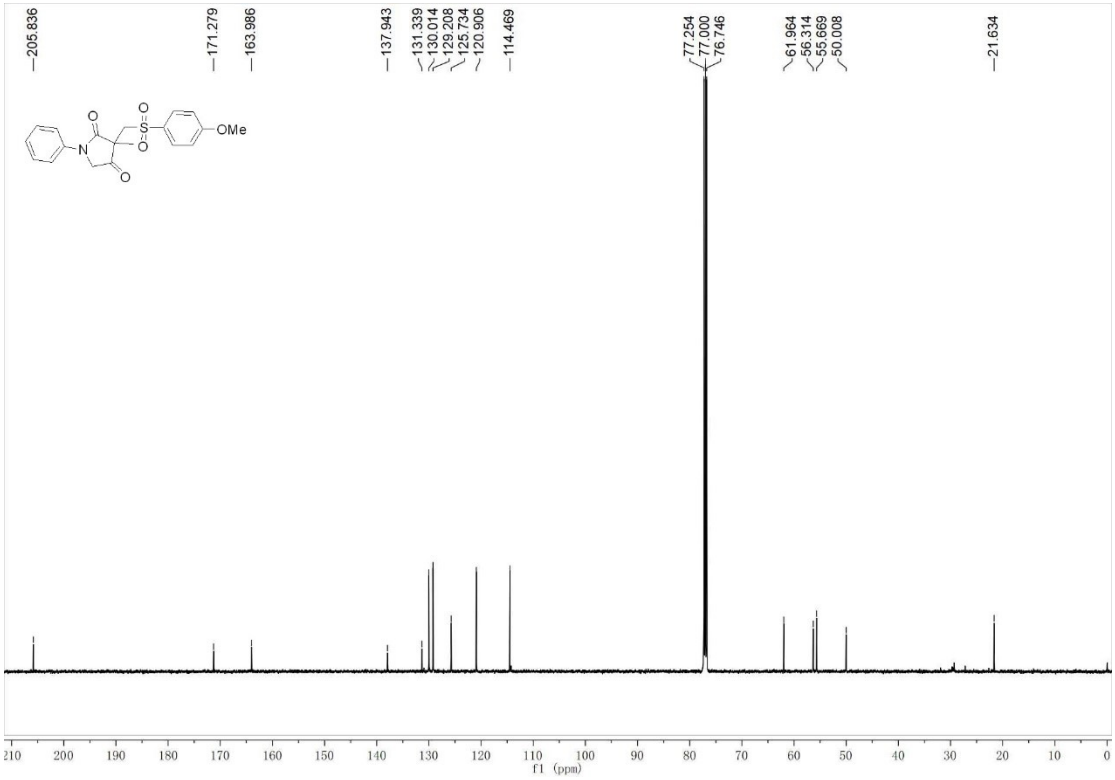
3-(((4-(*tert*-Butyl)phenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3b)



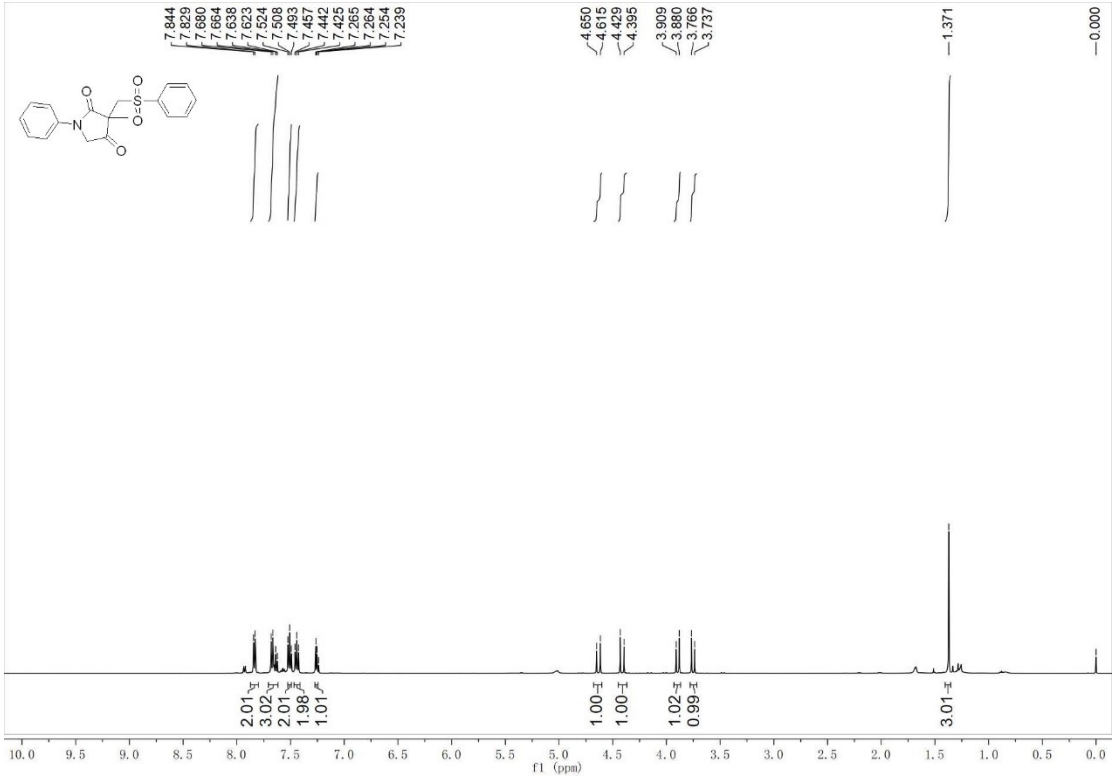


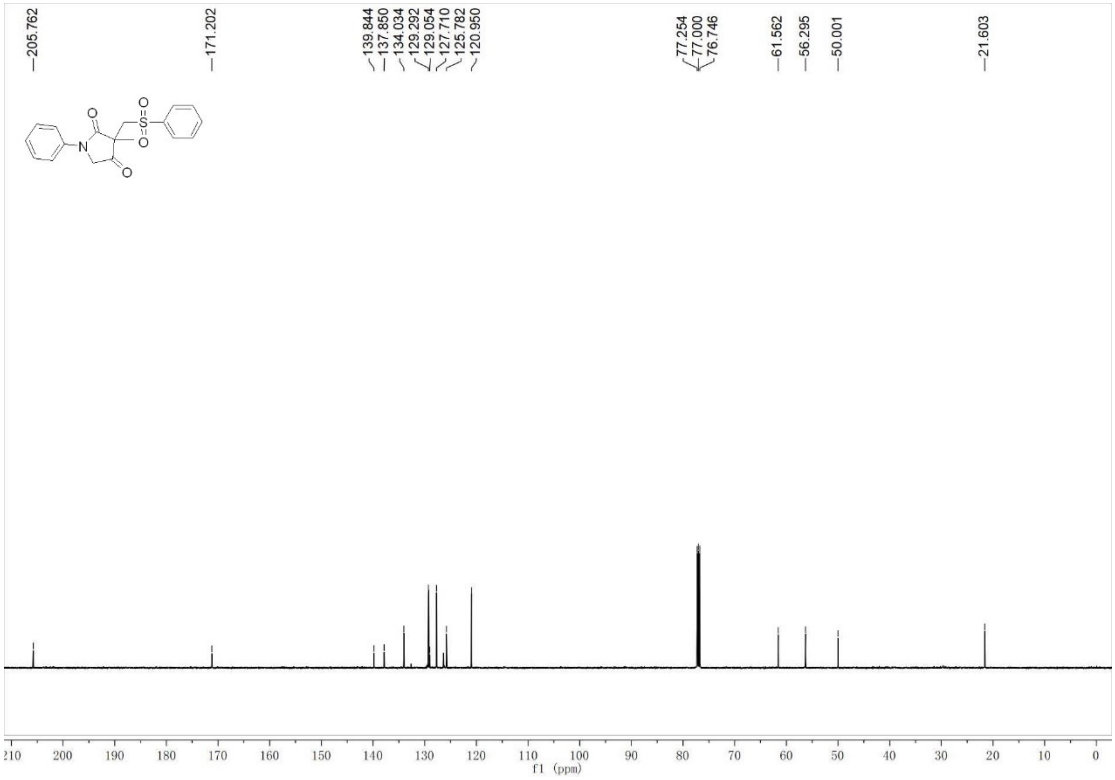
3-(((4-Methoxyphenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3c)



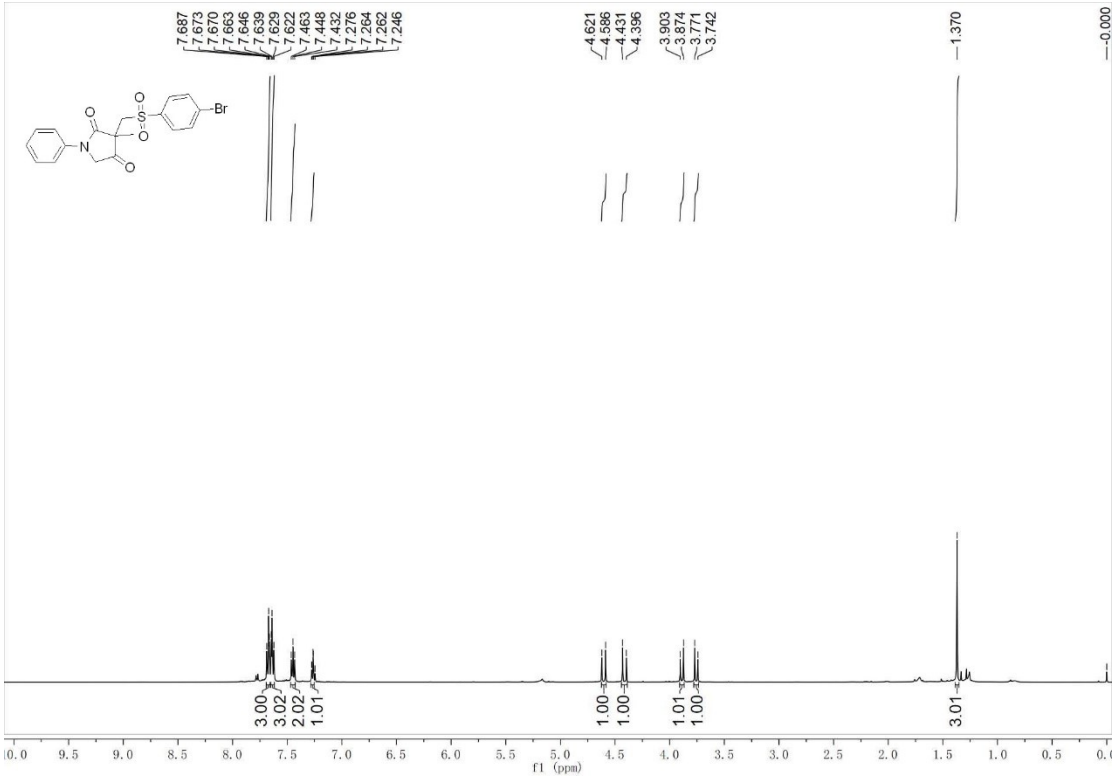


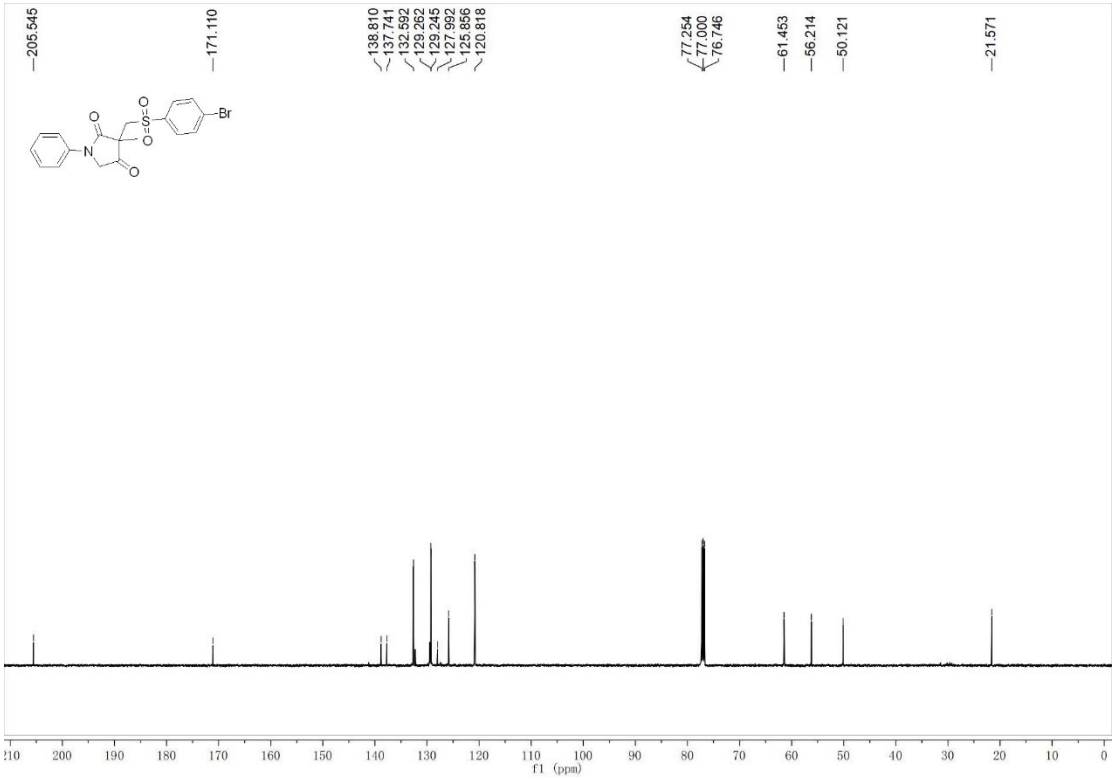
3-Methyl-1-phenyl-3-((phenylsulfonyl)methyl)pyrrolidine-2,4-dione (3d)



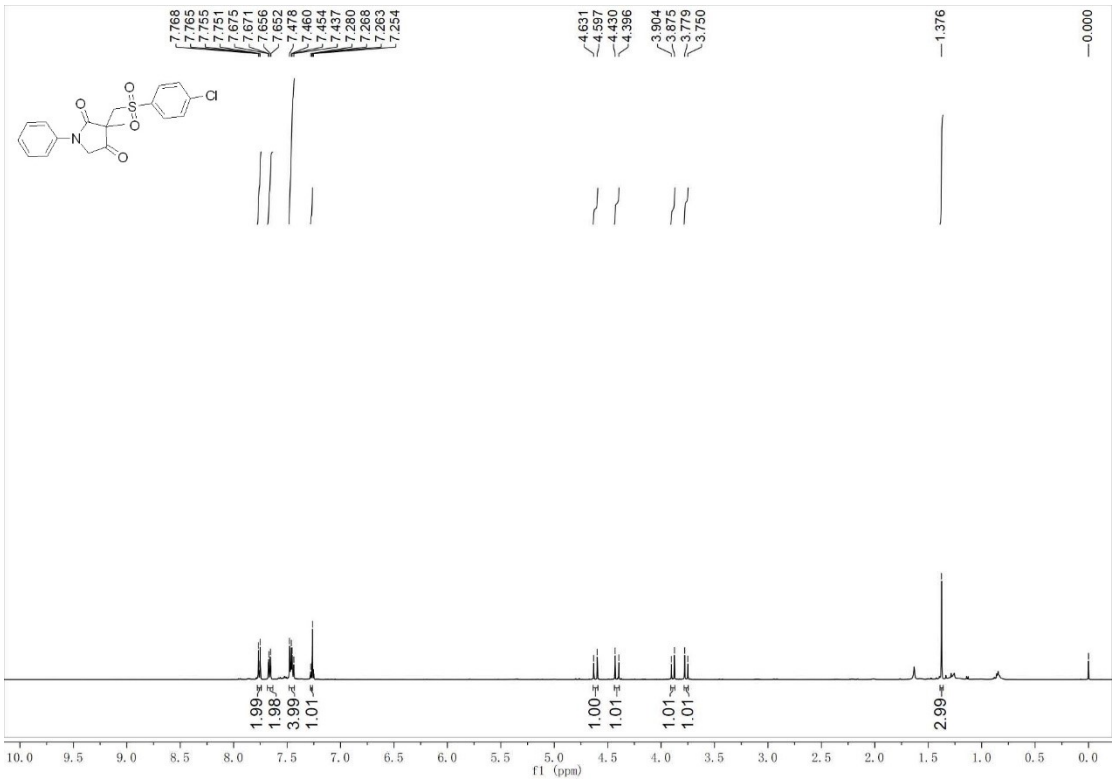


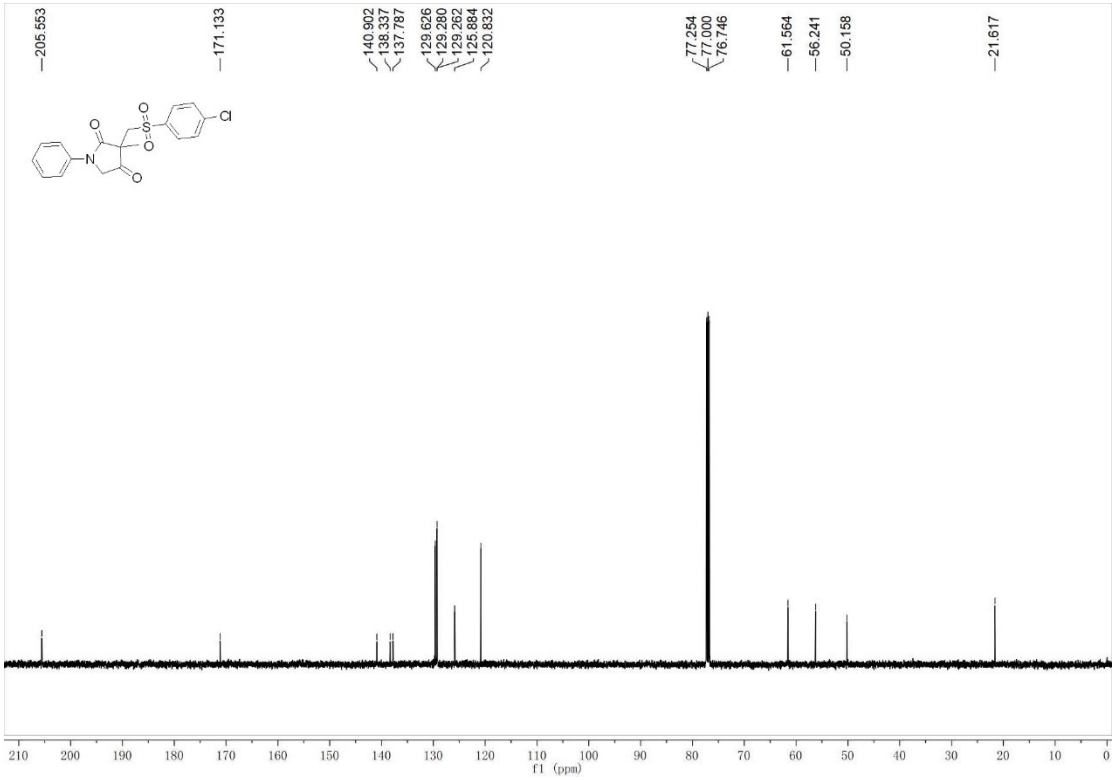
3-(((4-Bromophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3e)



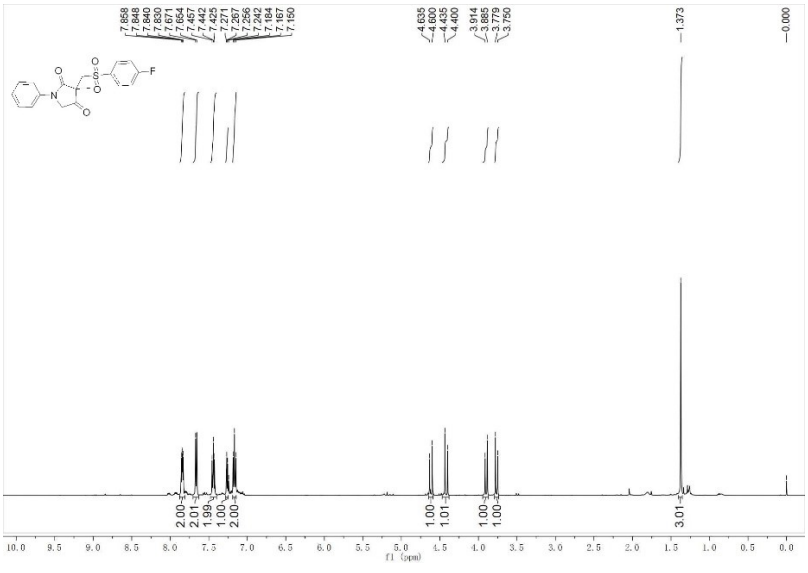


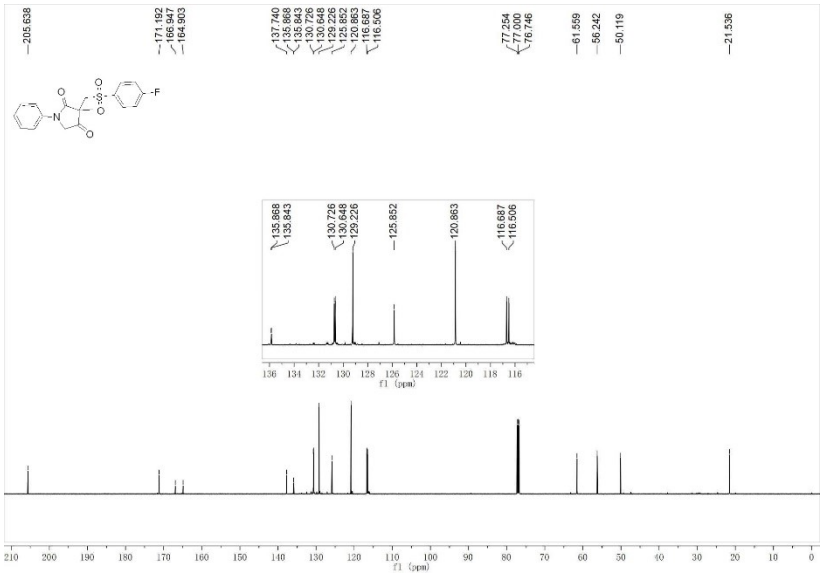
3-(((4-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3f)





3-(((4-Fluorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3g)

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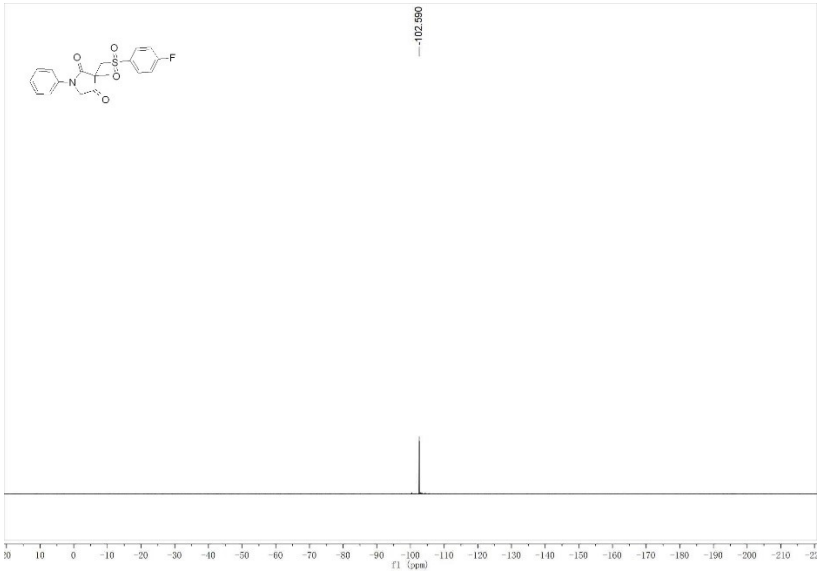
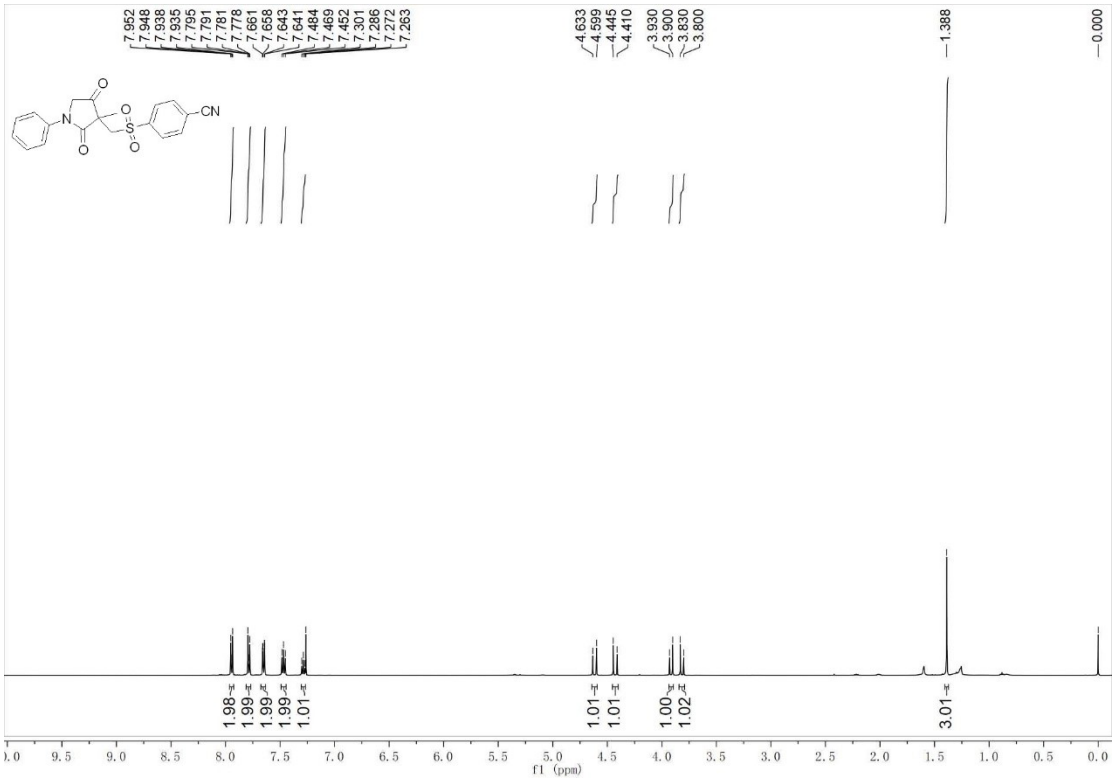
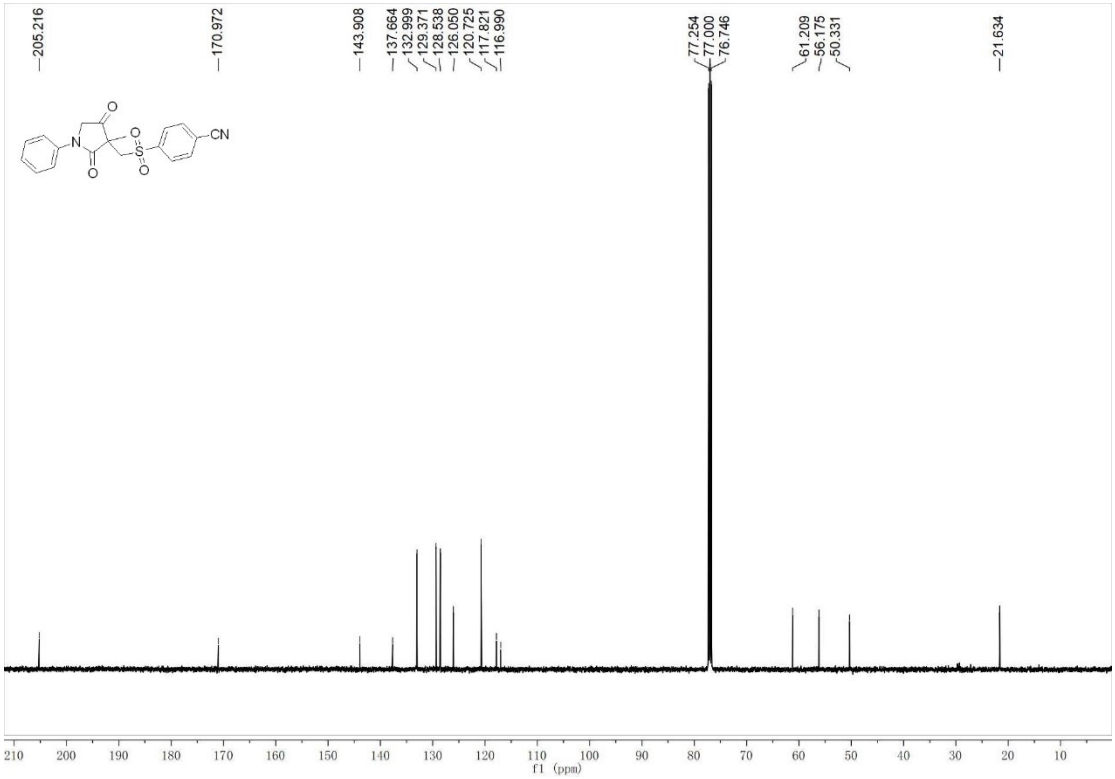
171.192
166.947
164.903
137.740
135.868
135.843
130.726
130.648
129.226
125.852
120.863
116.687
116.506
77.254
77.000
76.746
61.559
56.242
50.119
21.536
F
O
O
O
O
O
N
O
135.868
135.843
130.726
130.648
129.226
125.852
120.863
116.687
116.506
136 134 132 130 128 126 124 122 120 118 116
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

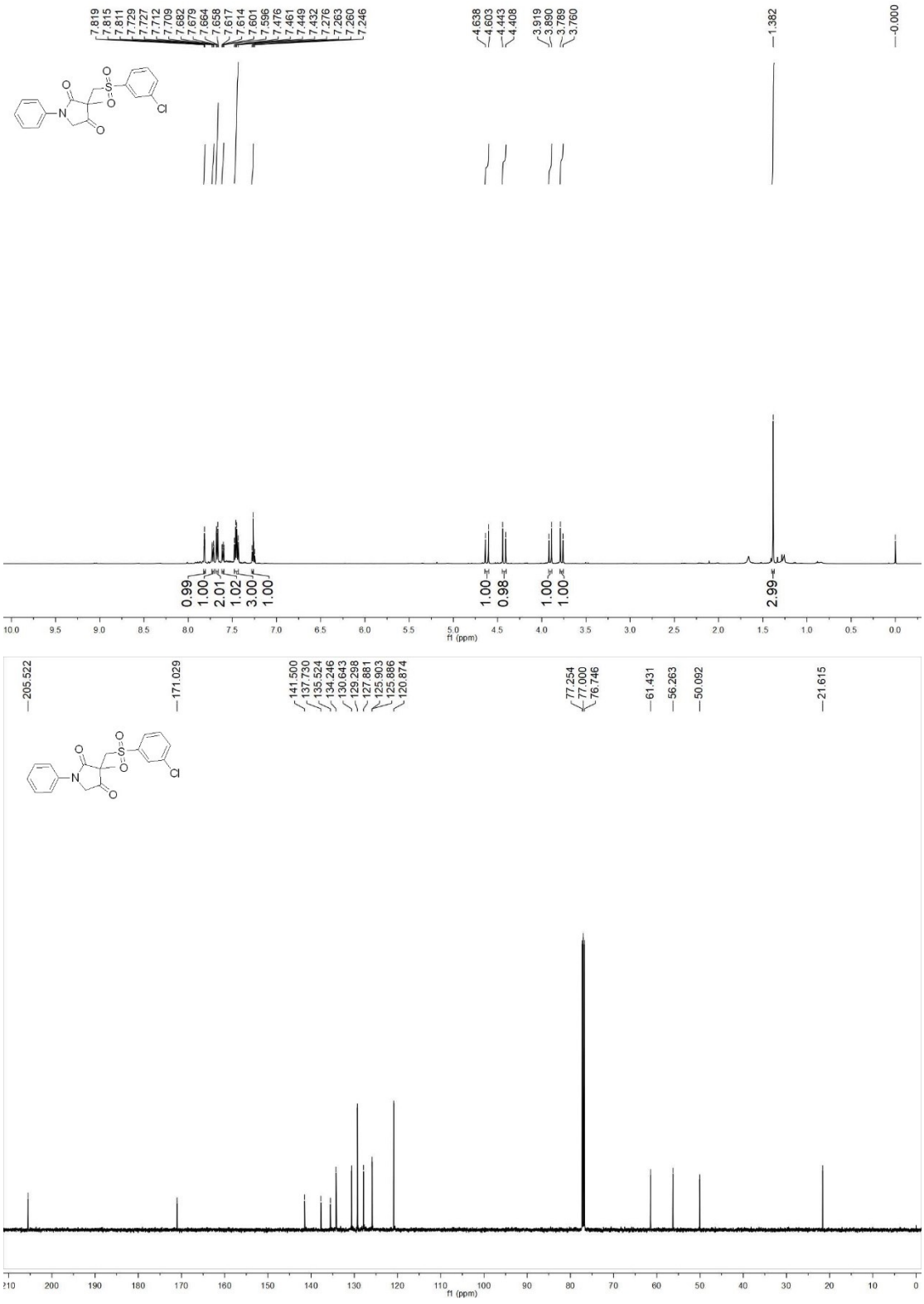

-102.590
F
O
O
O
O
O
N
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

4-(((3-Methyl-2,4-dioxo-1-phenylpyrrolidin-3-yl)methyl)sulfonyl)benzonitrile (3h)

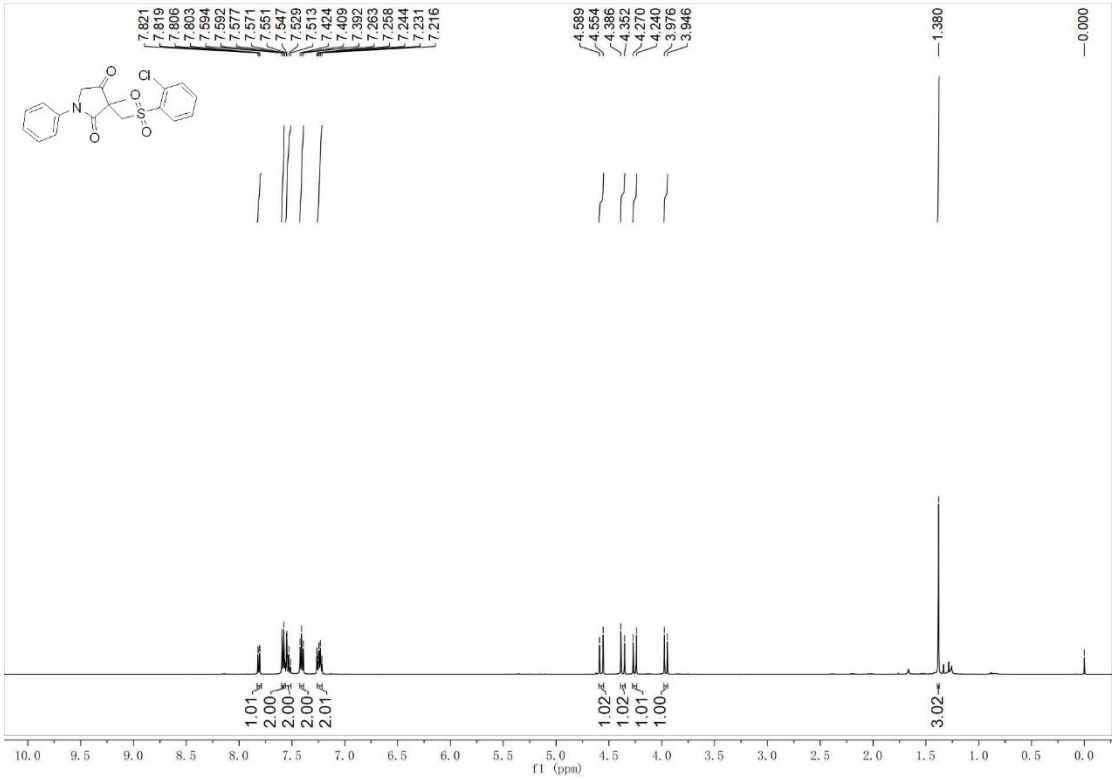


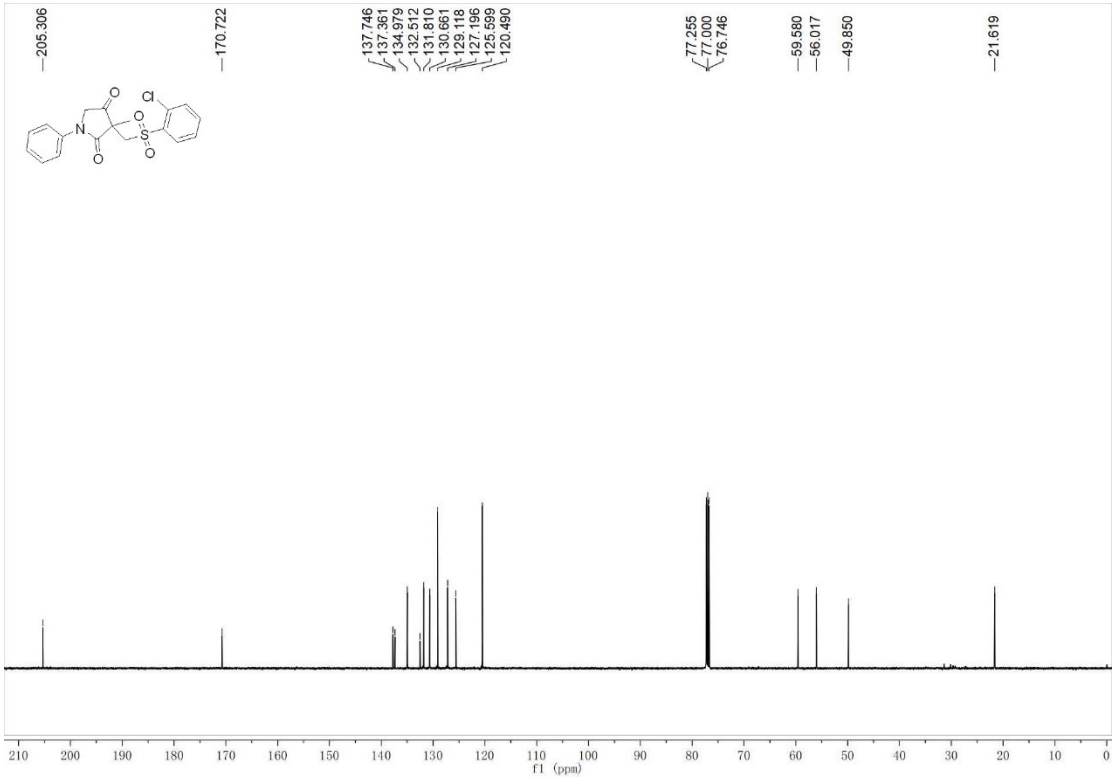


3-(((3-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3i)

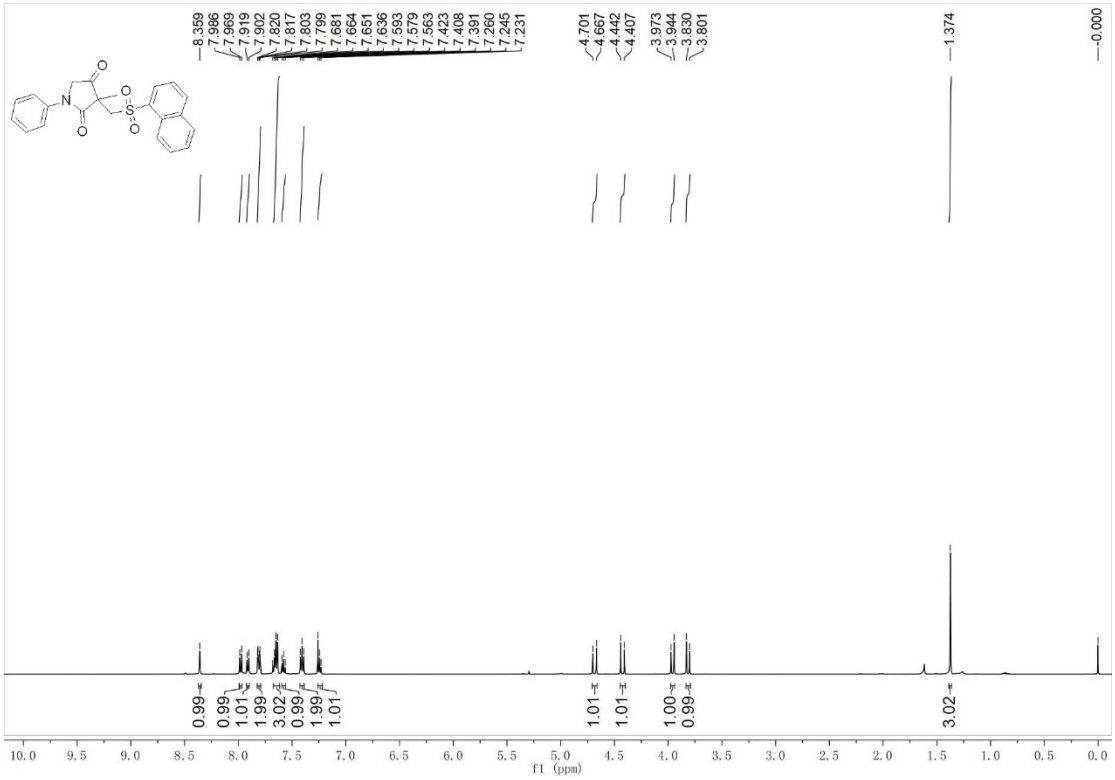


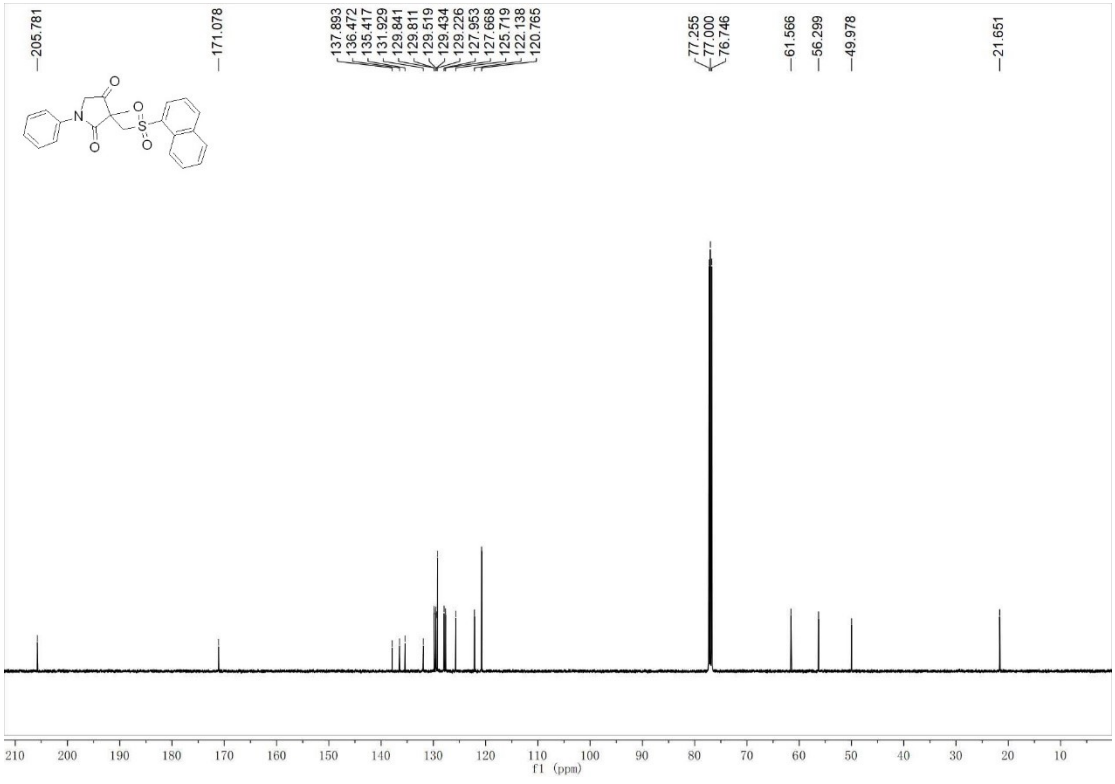
3-(((2-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-phenylpyrrolidine-2,4-dione (3j)



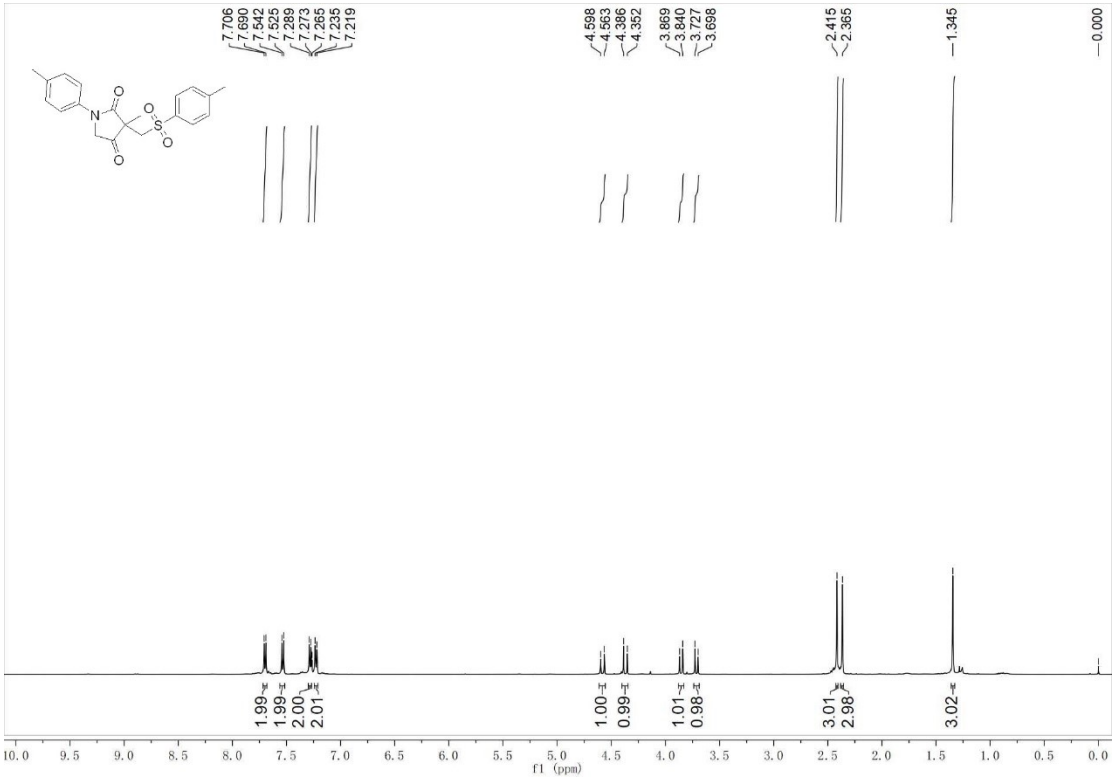


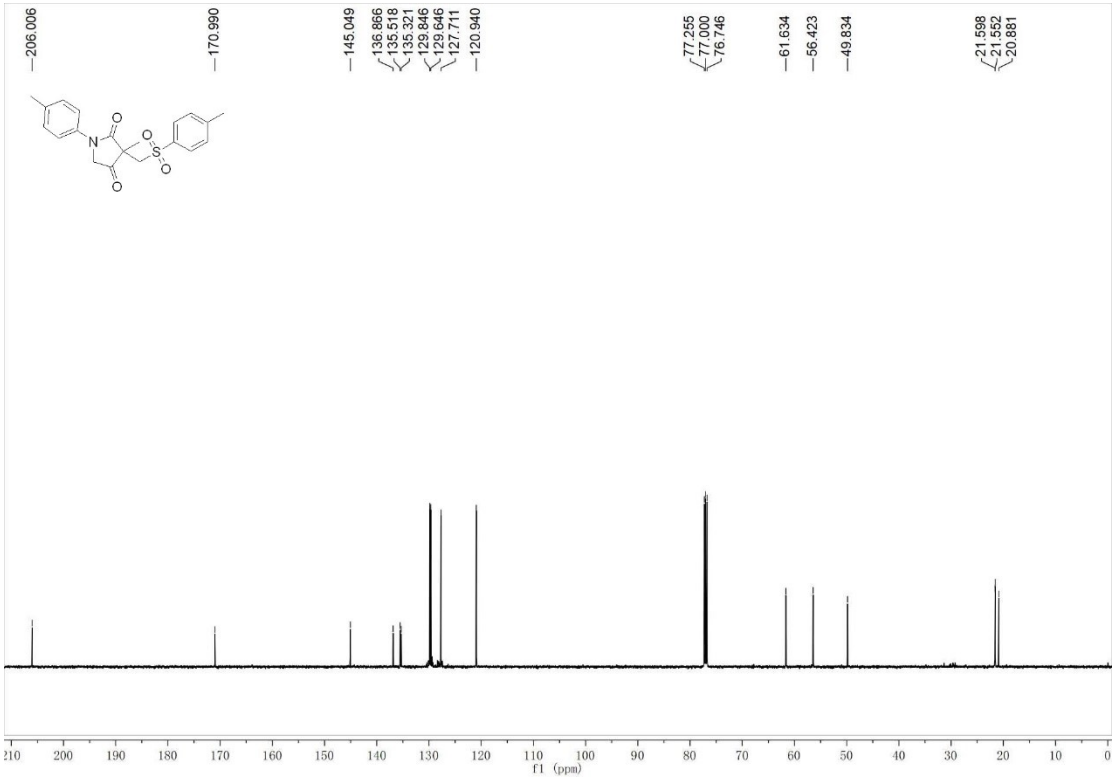
3-Methyl-3-((naphthalen-1-ylsulfonyl)methyl)-1-phenylpyrrolidine-2,4-dione (3k)



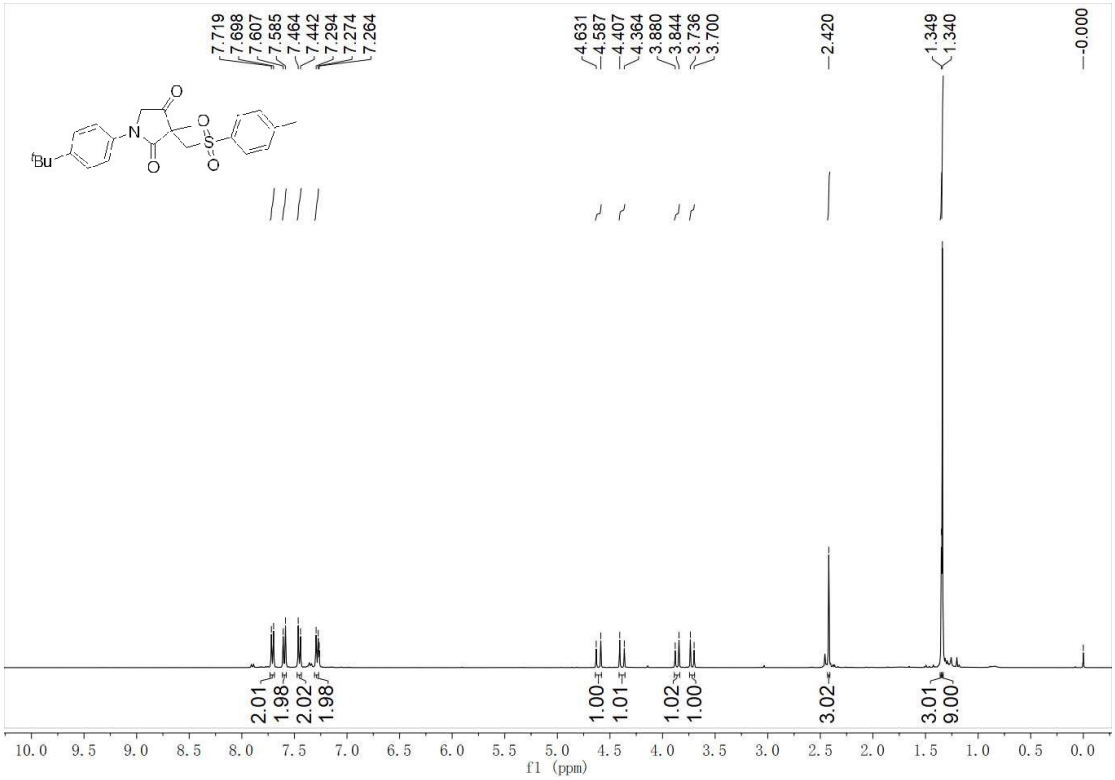


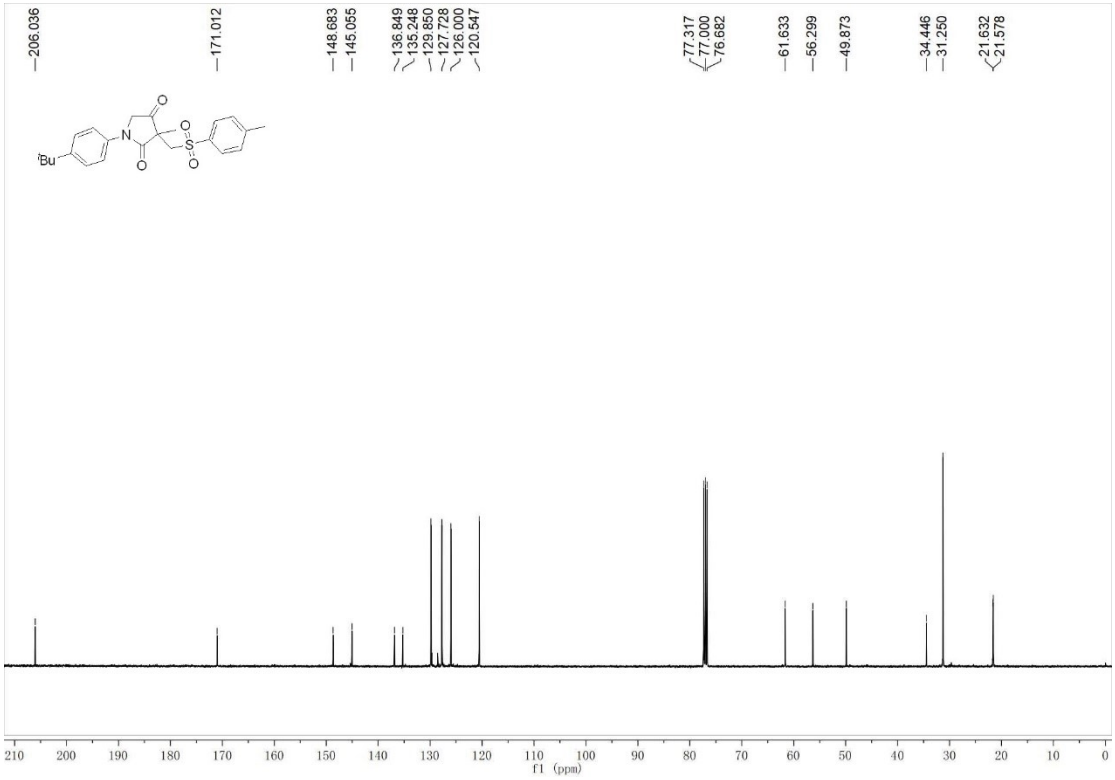
3-Methyl-1-(*p*-tolyl)-3-(tosylmethyl)pyrrolidine-2,4-dione (3m)



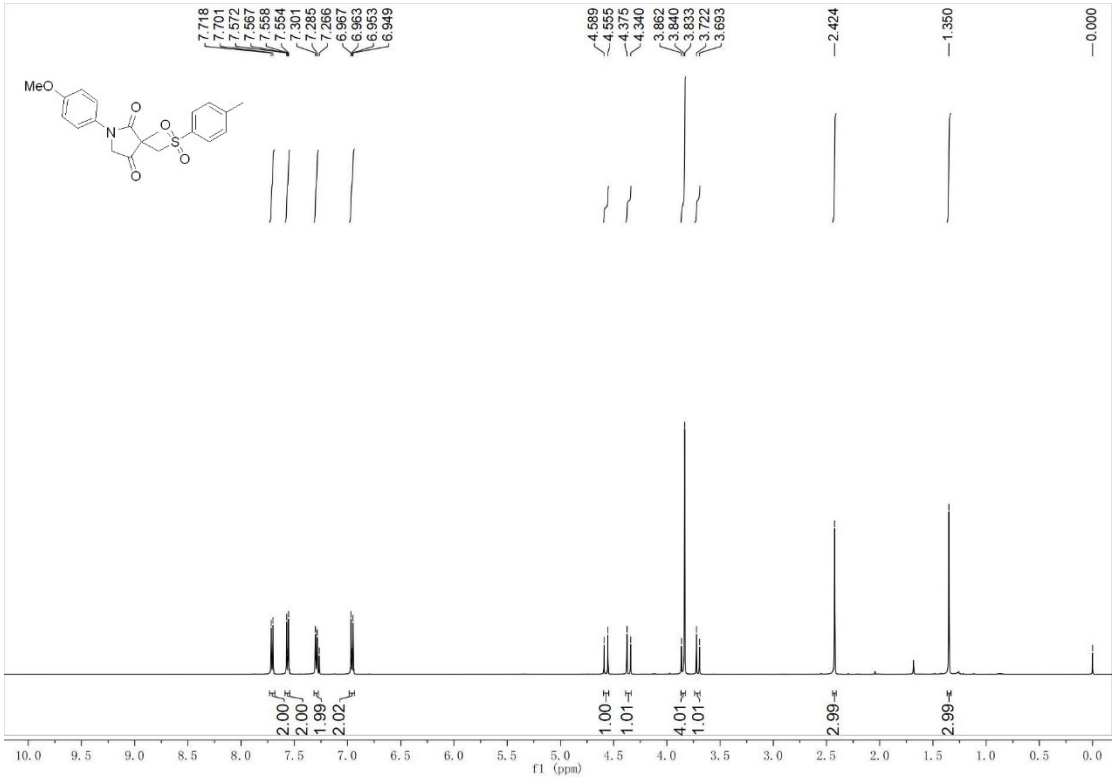


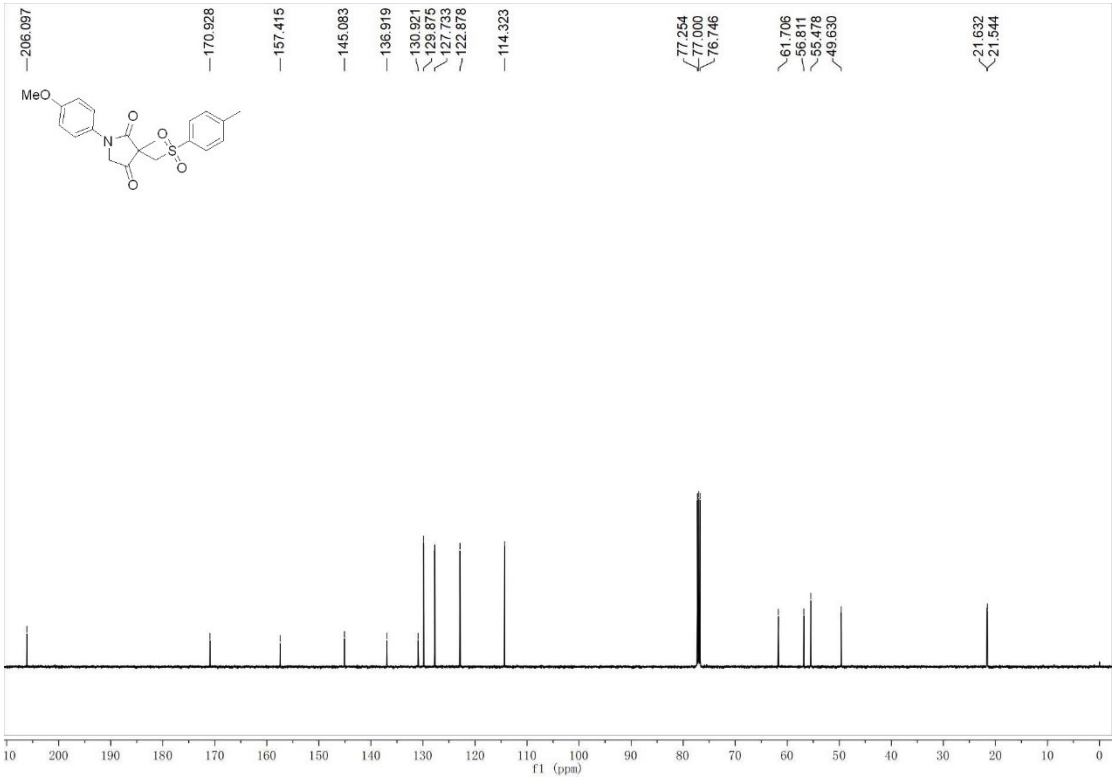
1-(4-(*tert*-Butyl)phenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3n)



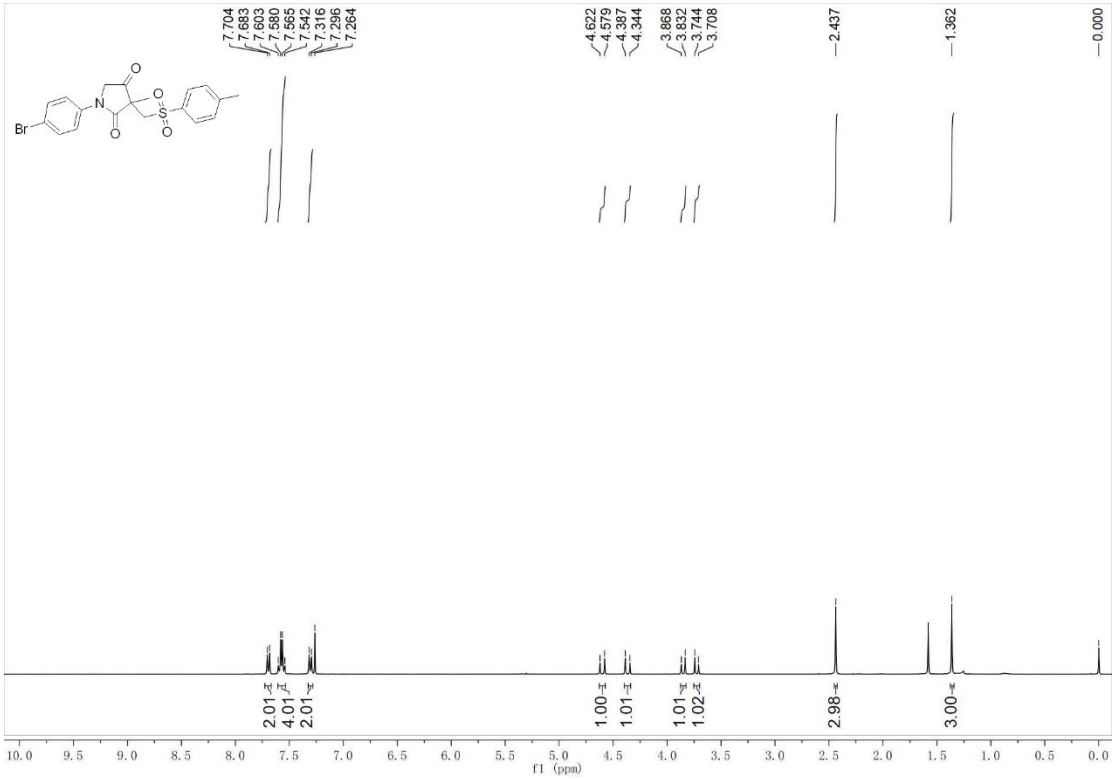


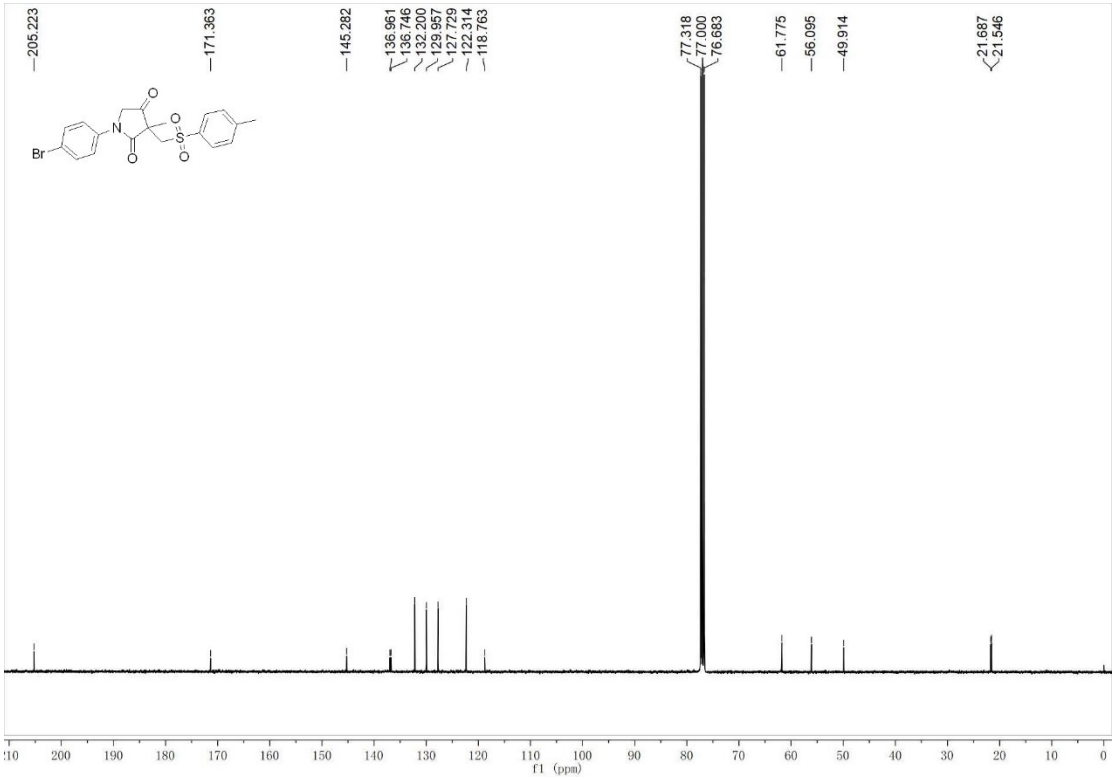
1-(4-Methoxyphenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3o)



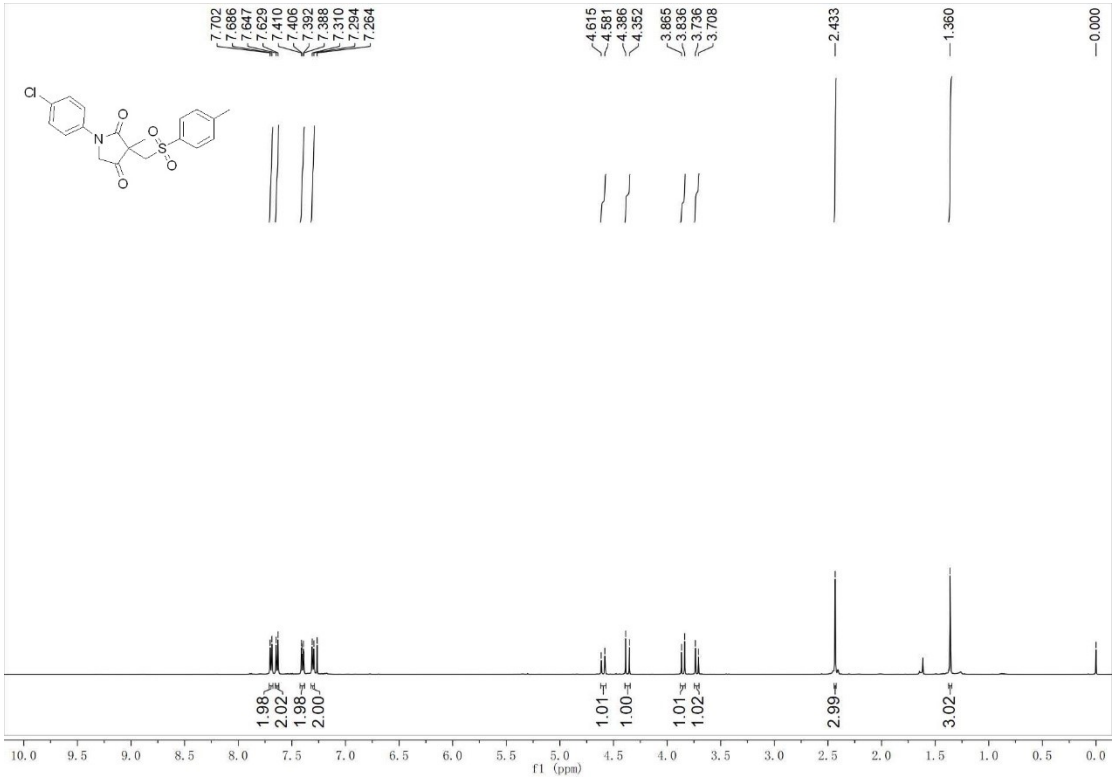


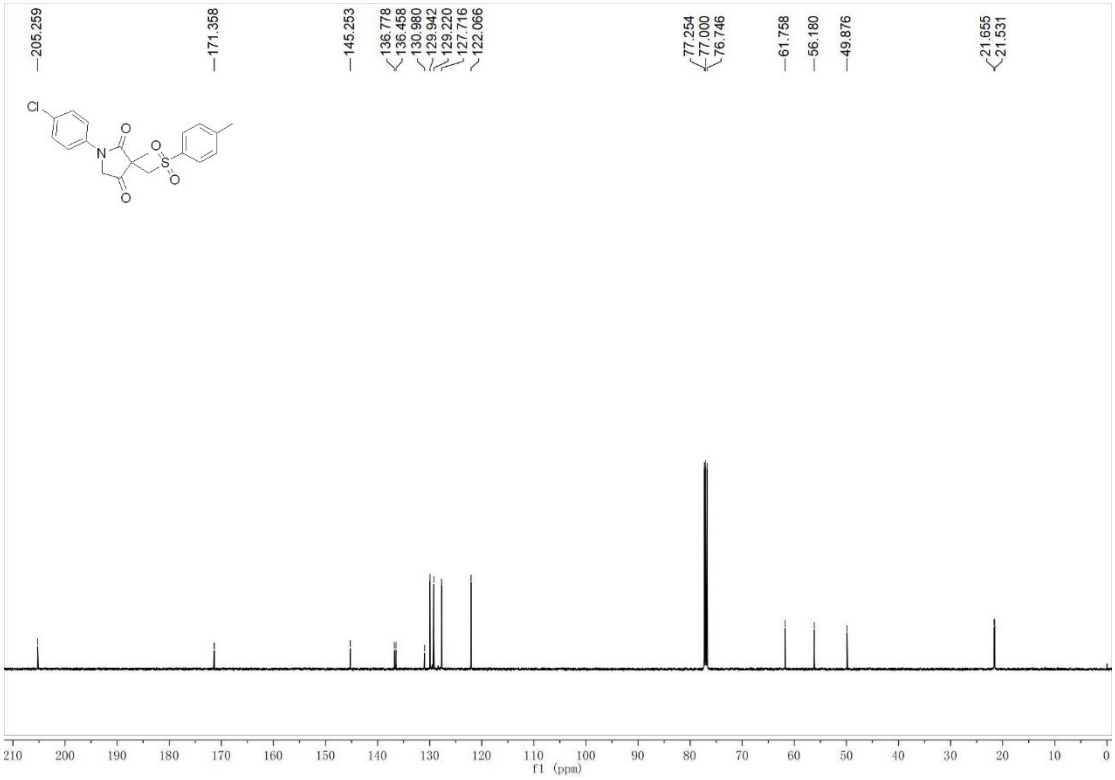
1-(4-Bromophenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3p)



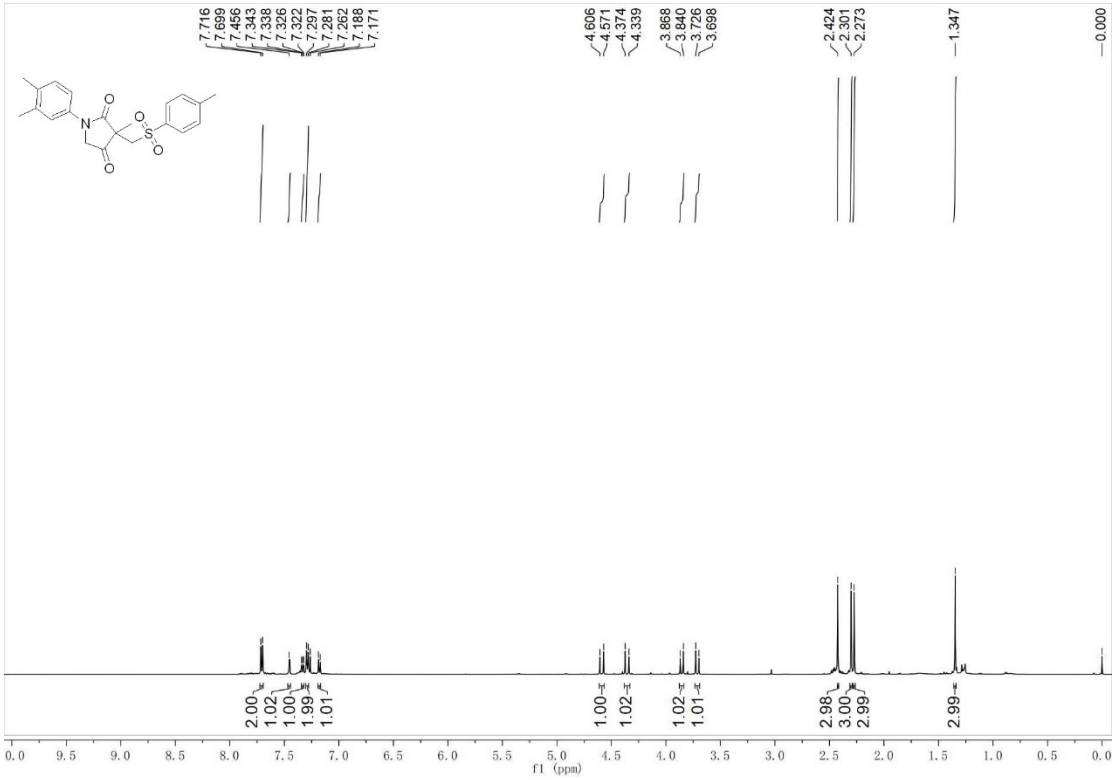


1-(4-Chlorophenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3q)





1-(3,4-Dimethylphenyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3r)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7.326
7.322
7.297
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7.262
7.188
7.171
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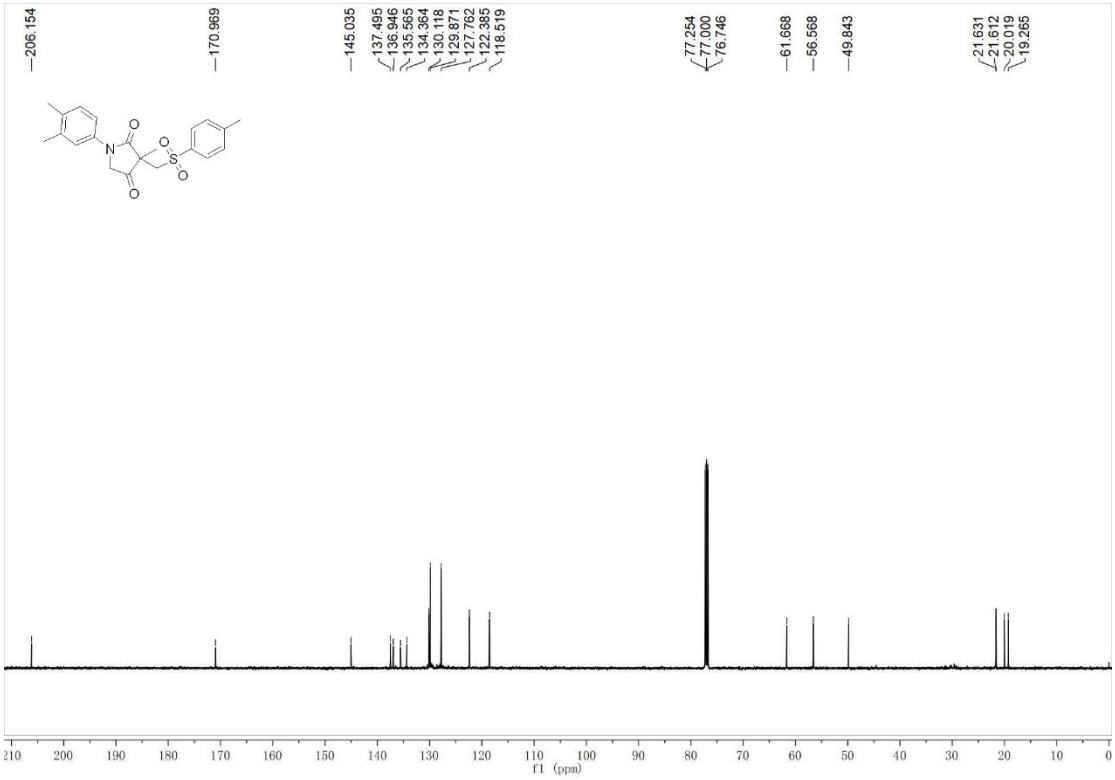
4.571
4.374
4.339
3.868
3.840
3.726
3.698
2.424
2.301
2.273
1.347
0.000
2.00
1.02
1.00
1.99
1.01
1.00
1.02
1.02
1.01
2.98
3.00
2.99
2.99
f1 (ppm)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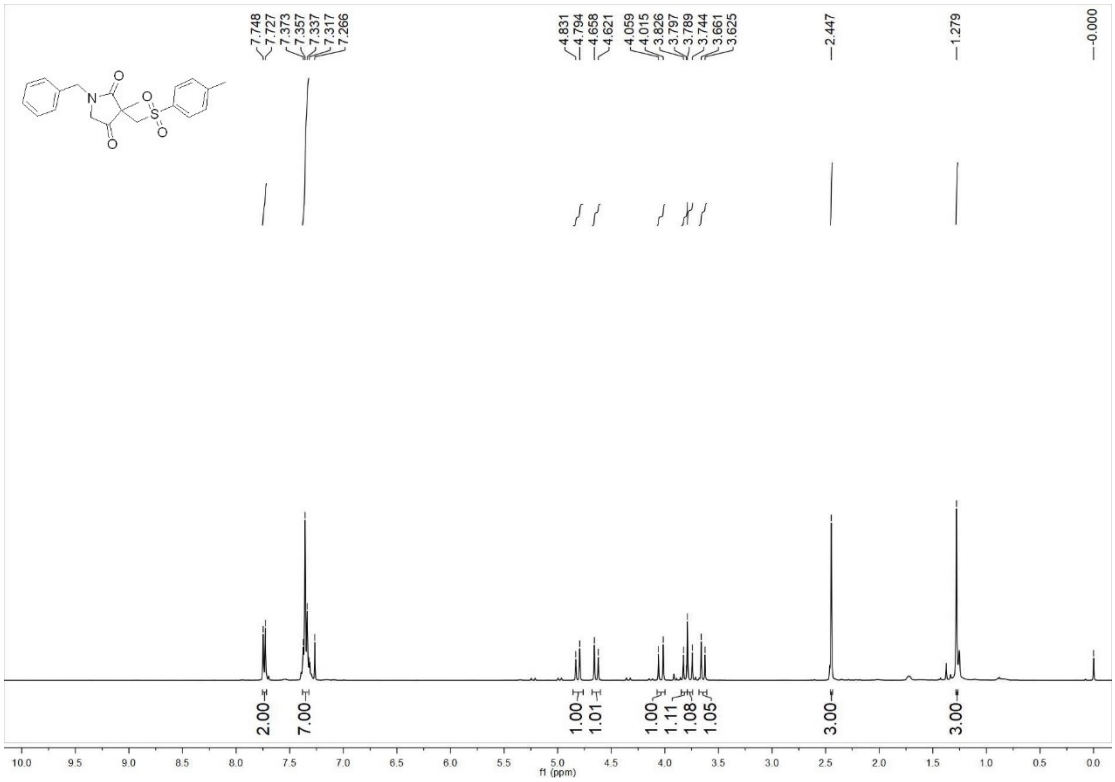
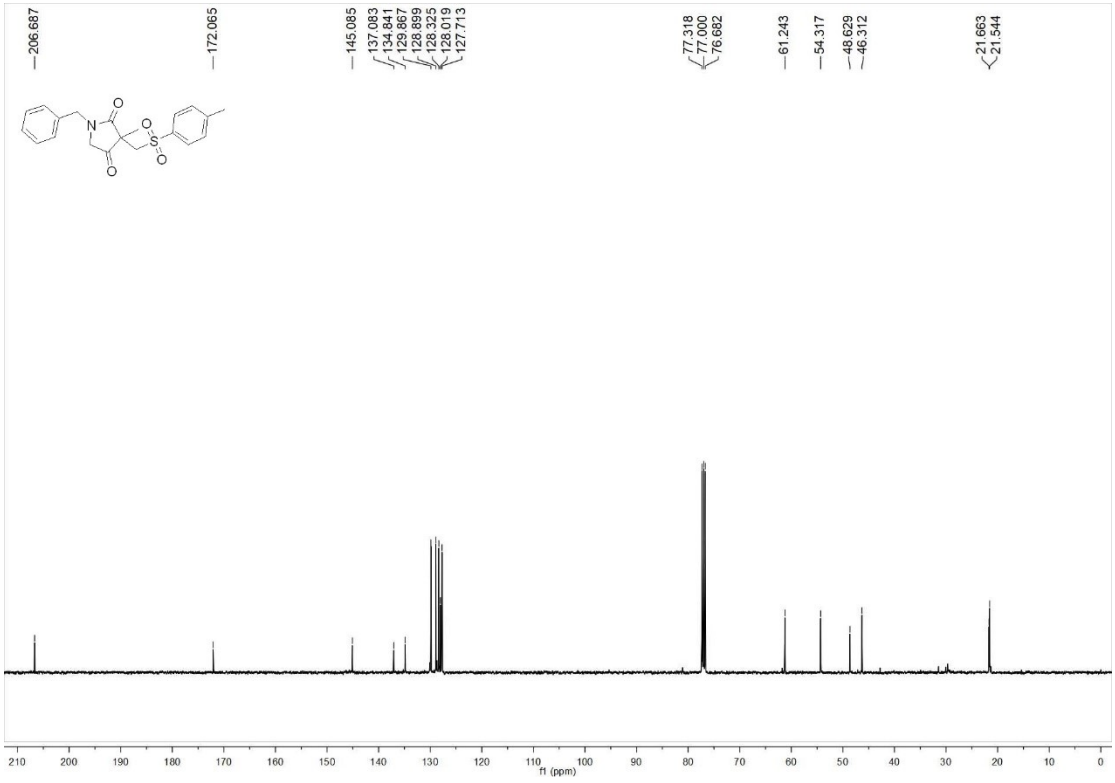
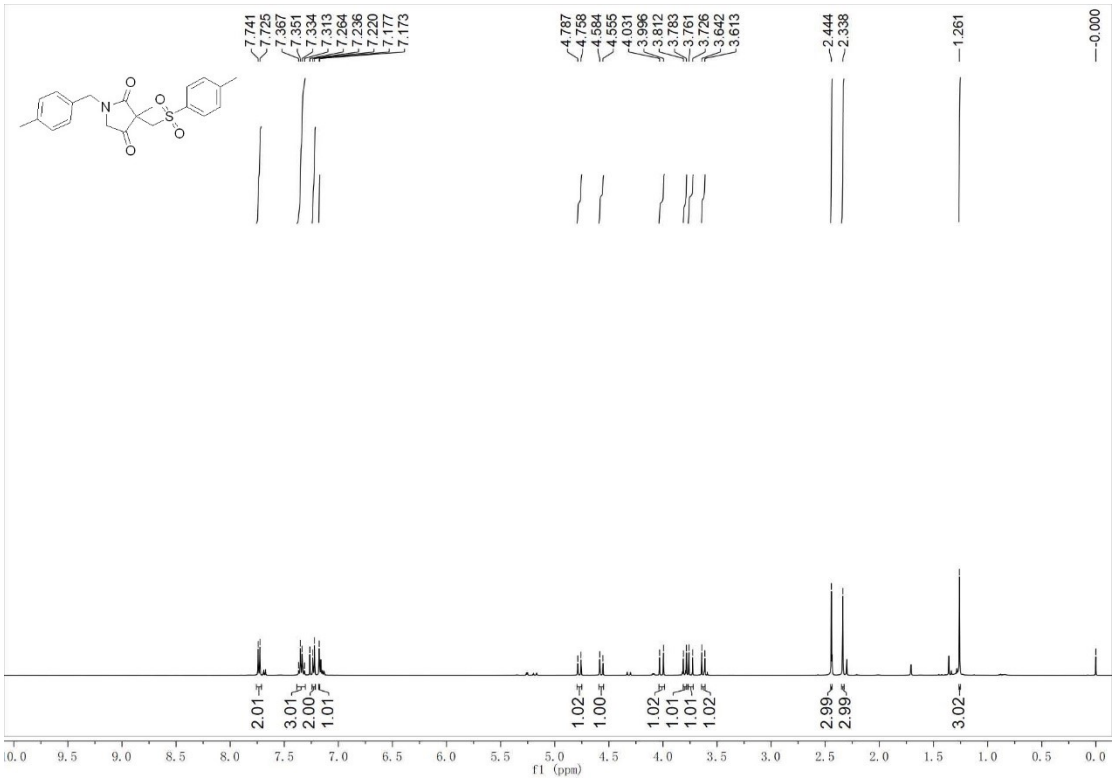
145.035
137.495
136.946
135.565
134.364
130.118
129.871
127.762
122.385
118.519
77.254
77.000
76.746
61.668
56.568
49.843
21.631
21.612
20.019
19.265
f1 (ppm)

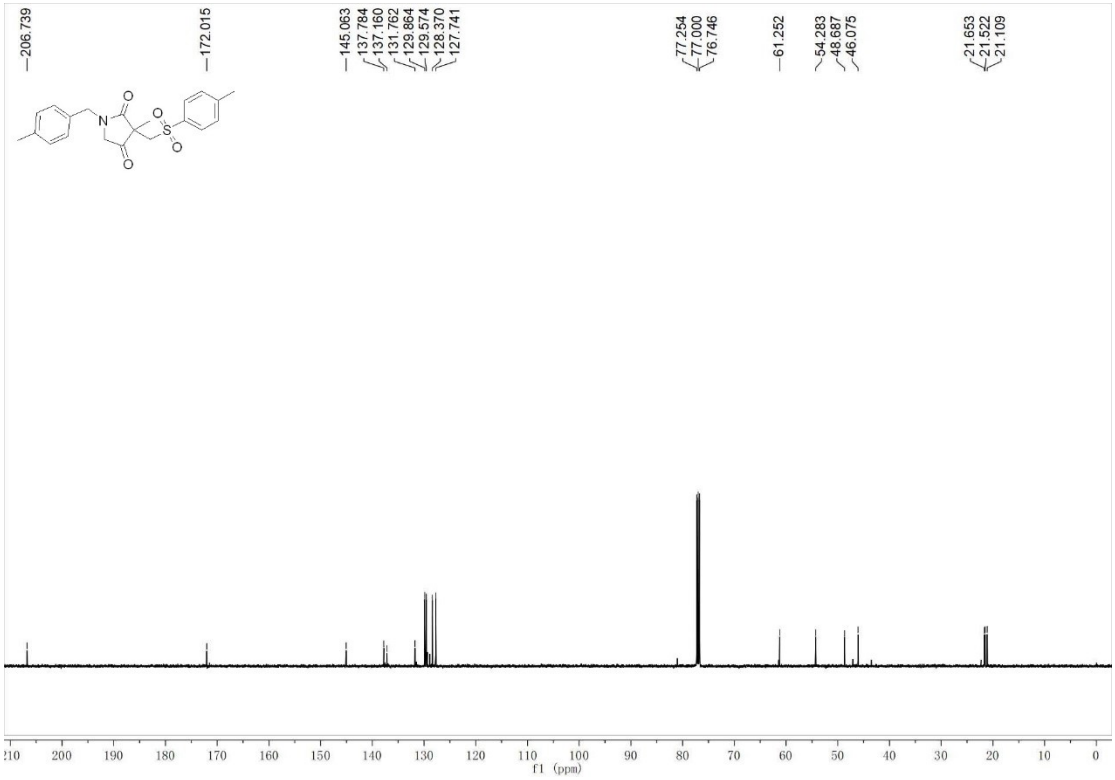
1-Benzyl-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3s)



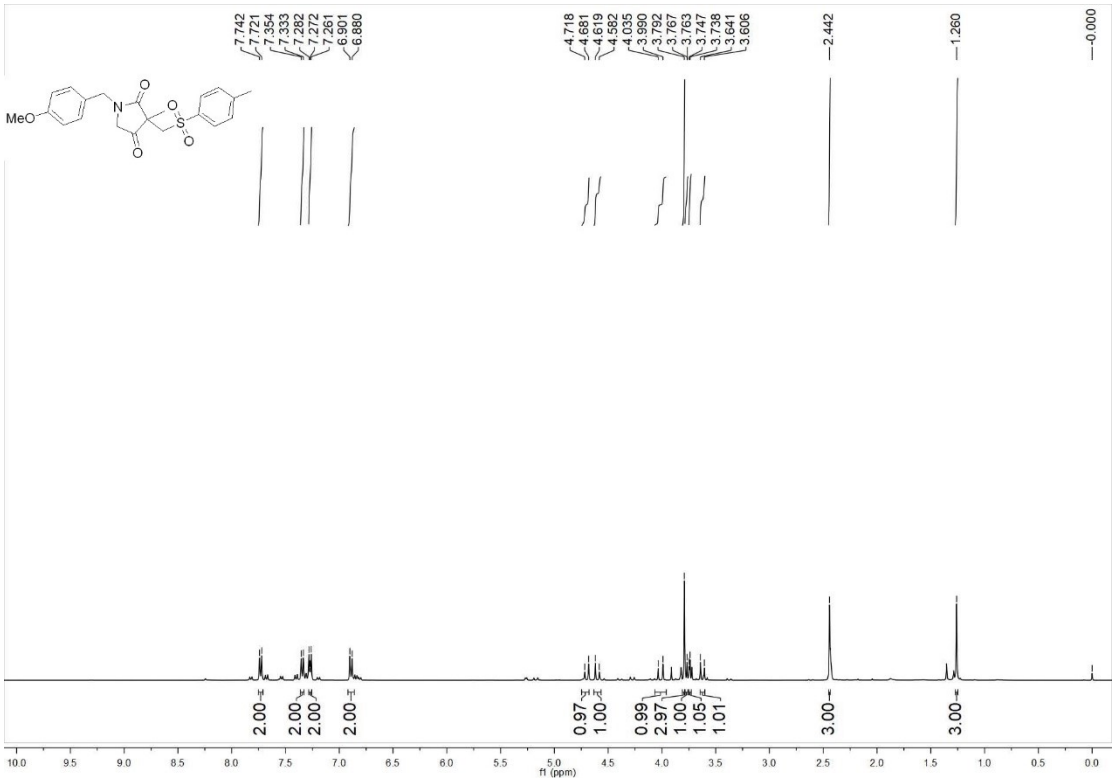


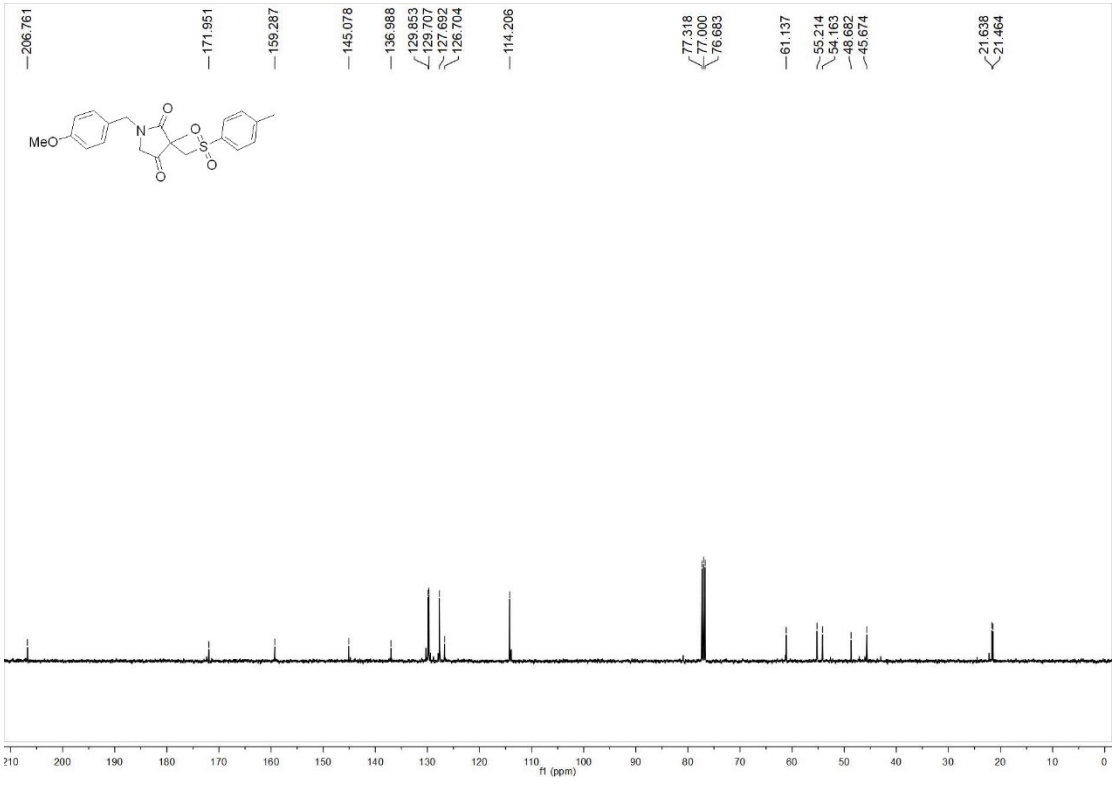
3-Methyl-1-(4-methylbenzyl)-3-(tosylmethyl)pyrrolidine-2,4-dione (3t)



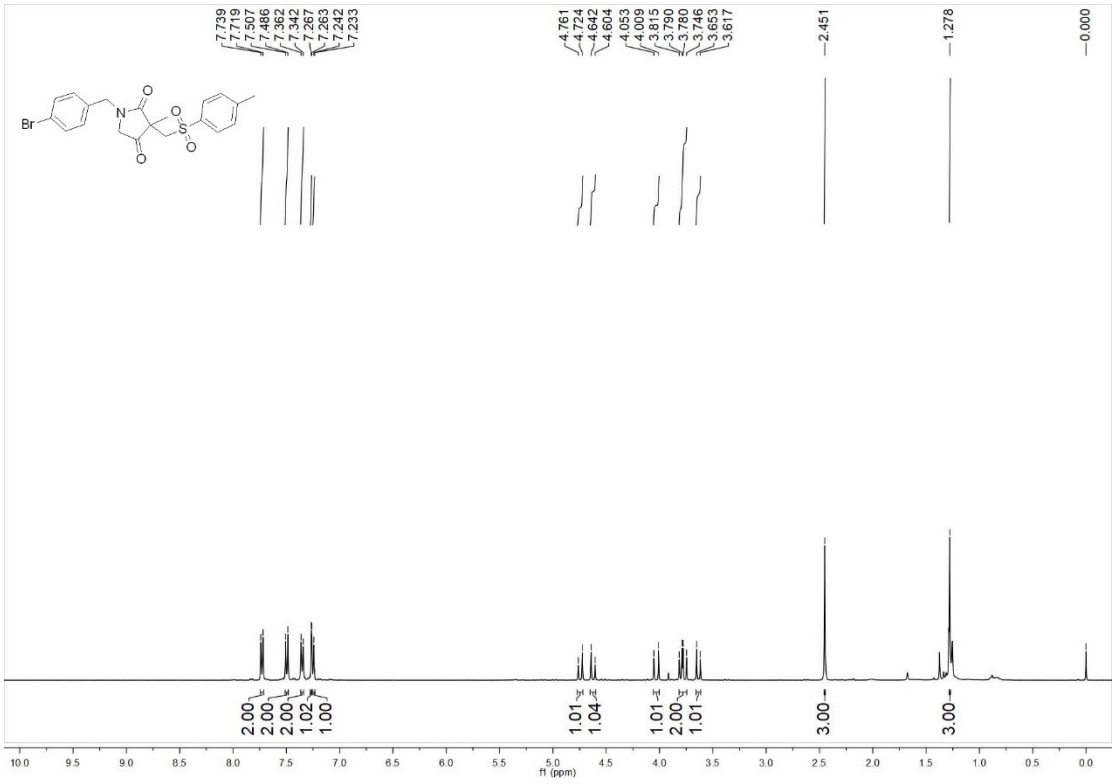


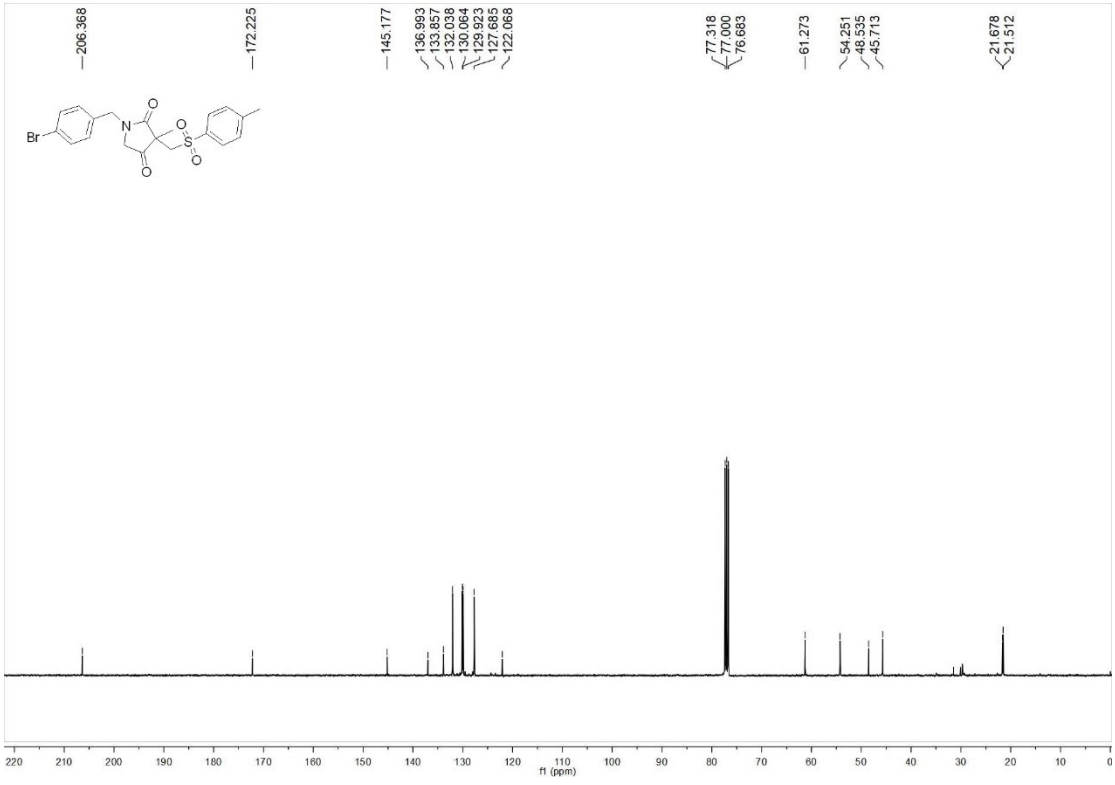
1-(4-Methoxybenzyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3u)



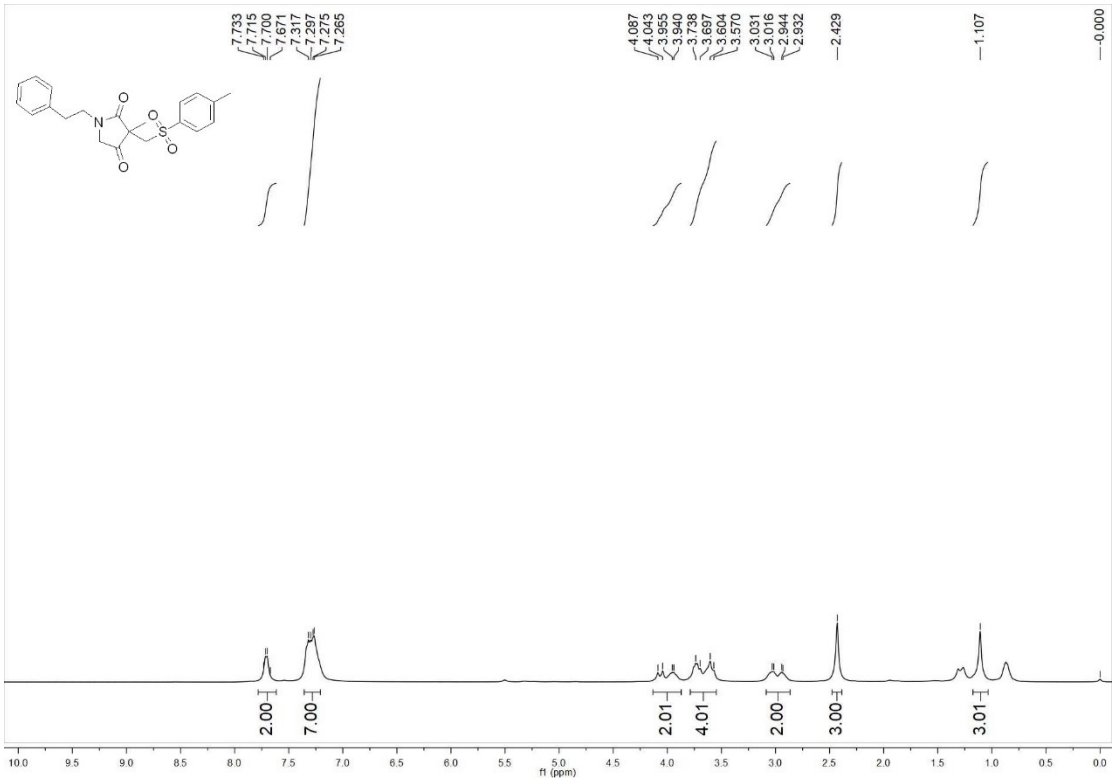


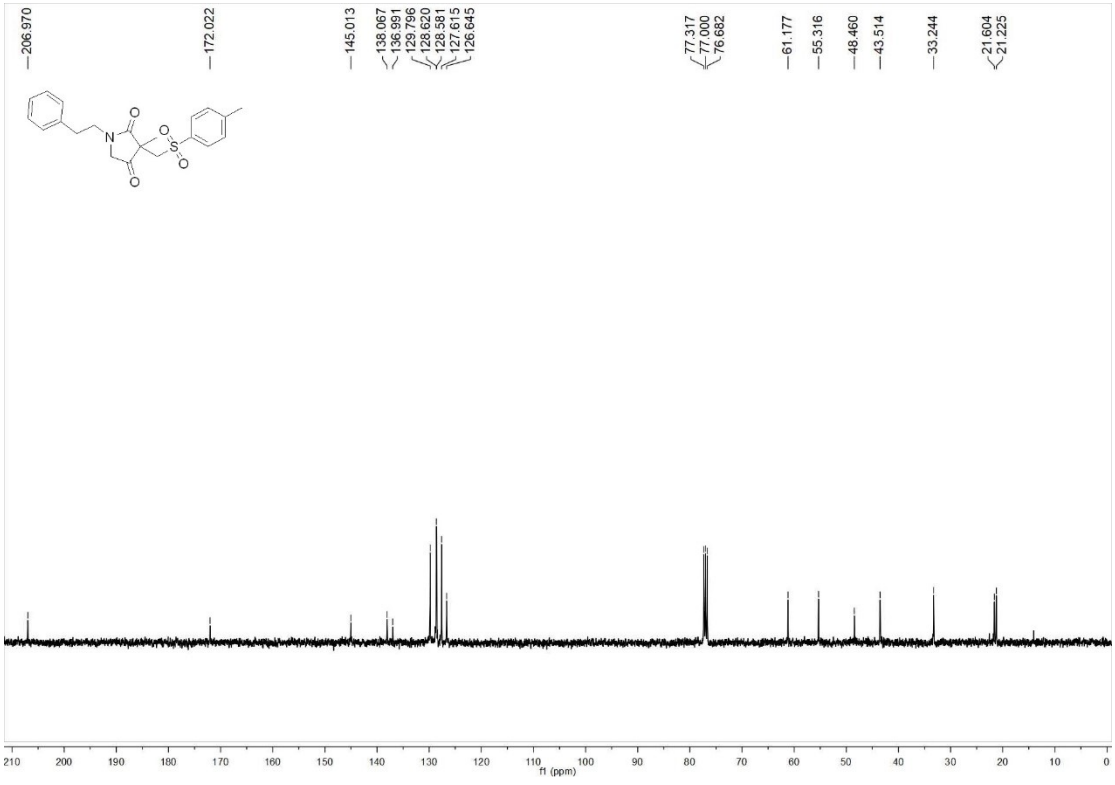
1-(4-Bromobenzyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3v)



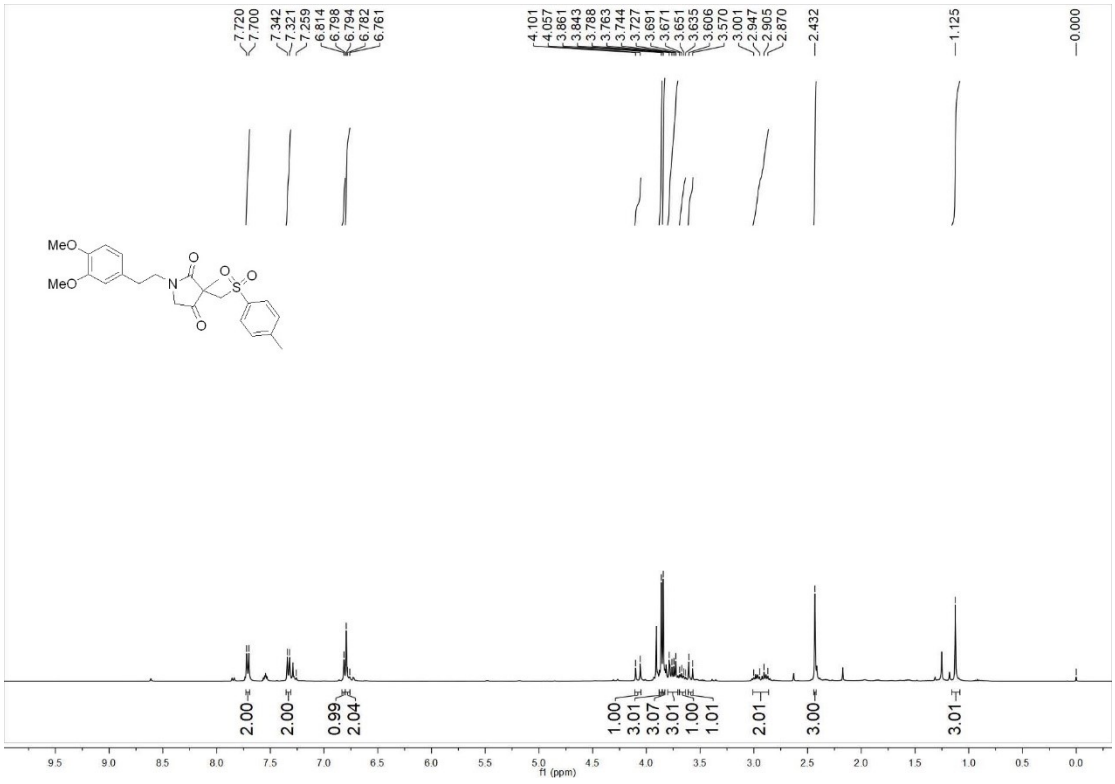


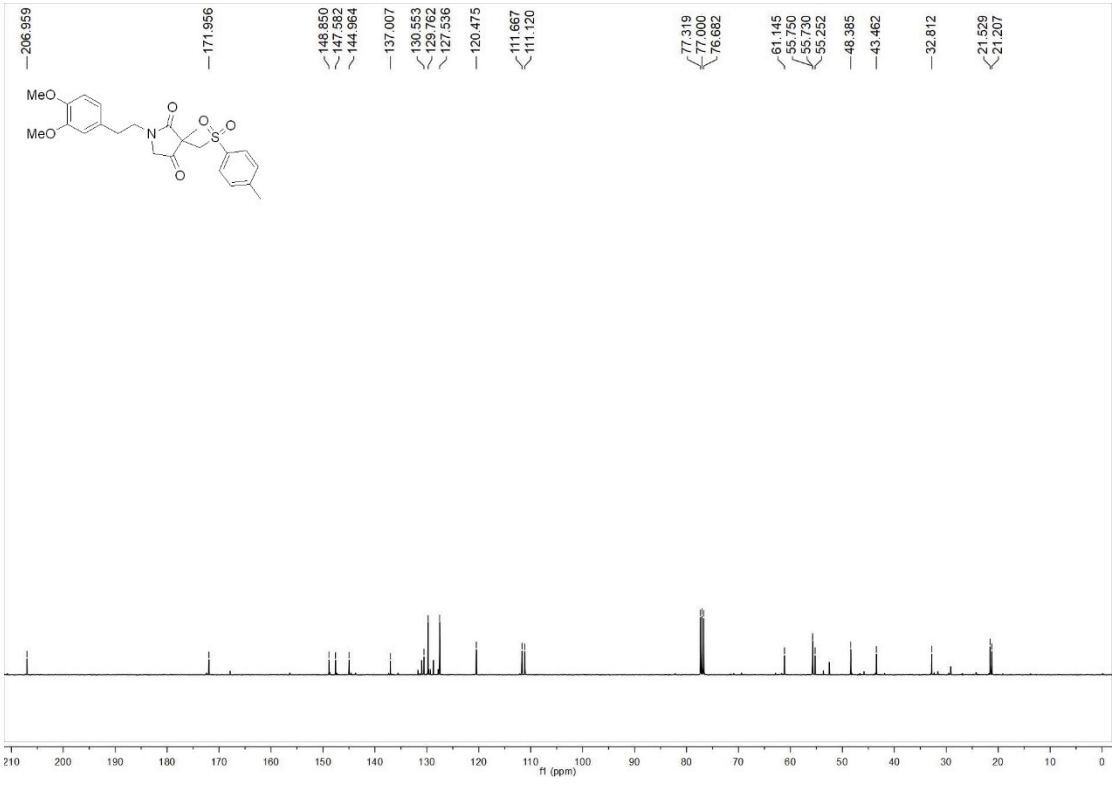
3-Methyl-1-phenethyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3w)



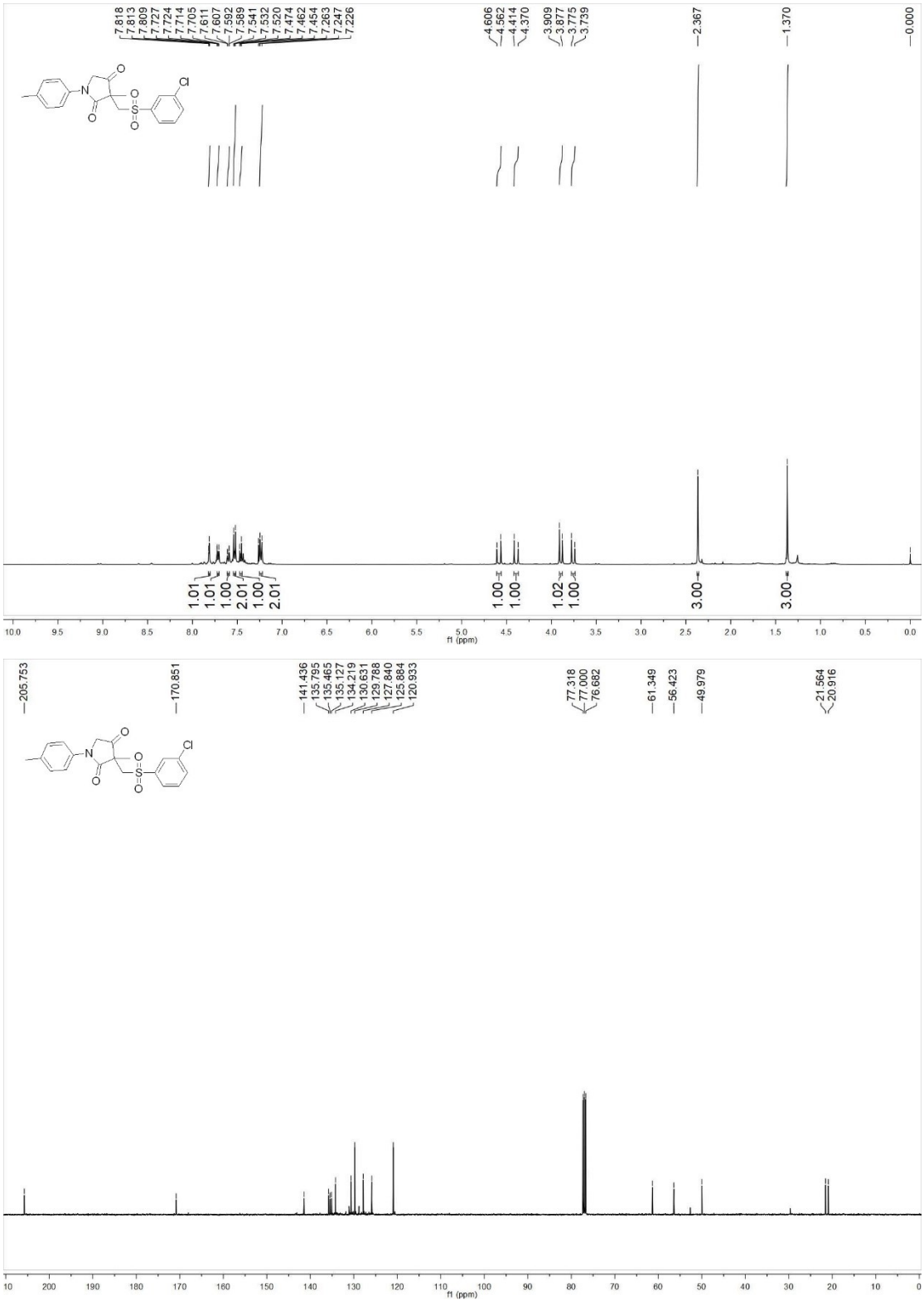


1-(3,4-Dimethoxyphenethyl)-3-methyl-3-(tosylmethyl)pyrrolidine-2,4-dione (3x)

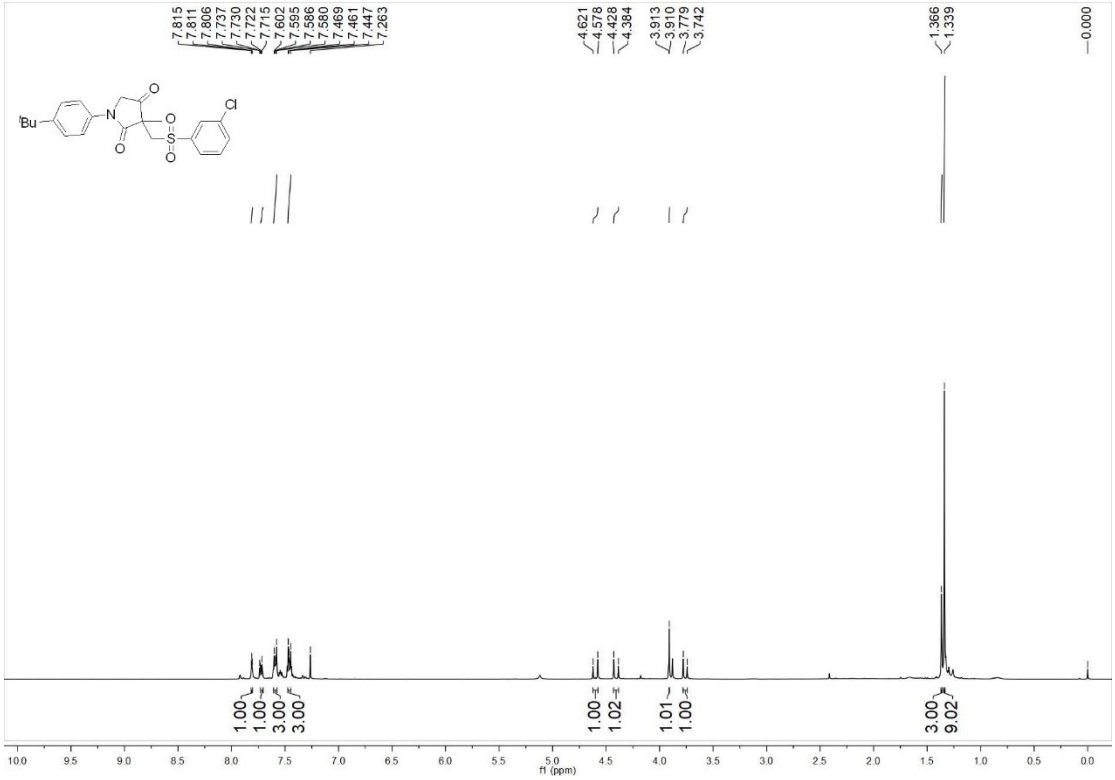


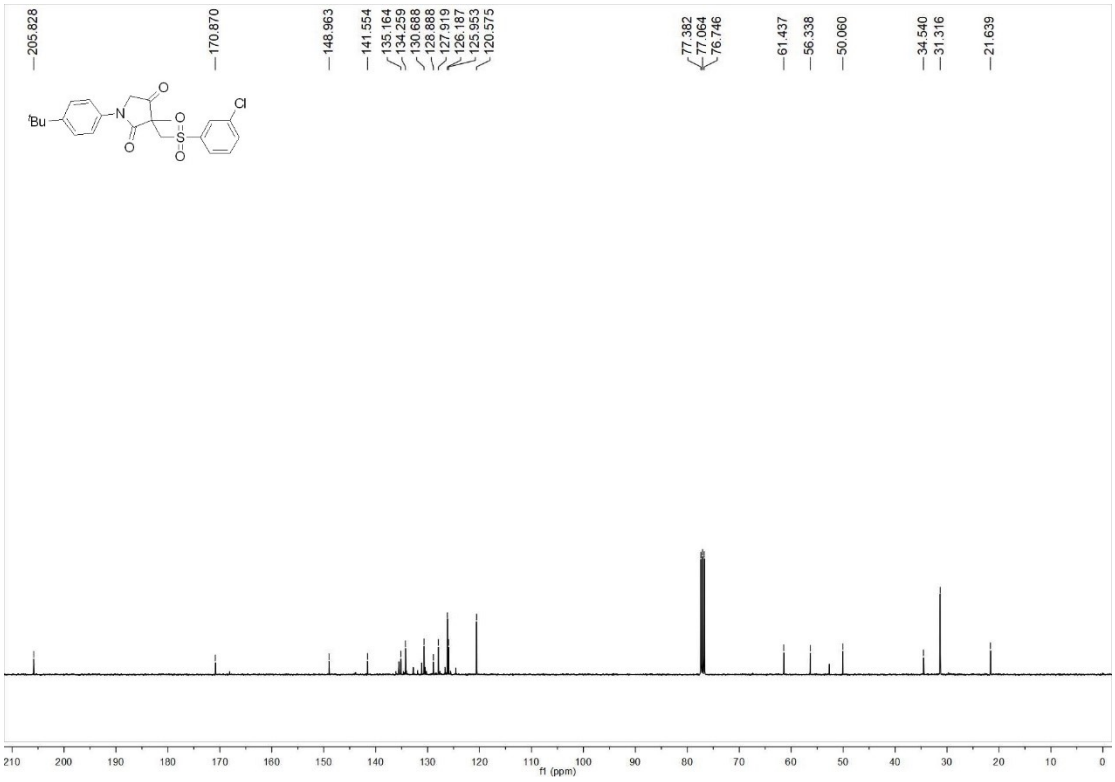


3-(((3-Chlorophenyl)sulfonyl)methyl)-3-methyl-1-(*p*-tolyl)pyrrolidine-2,4-dione (3z)

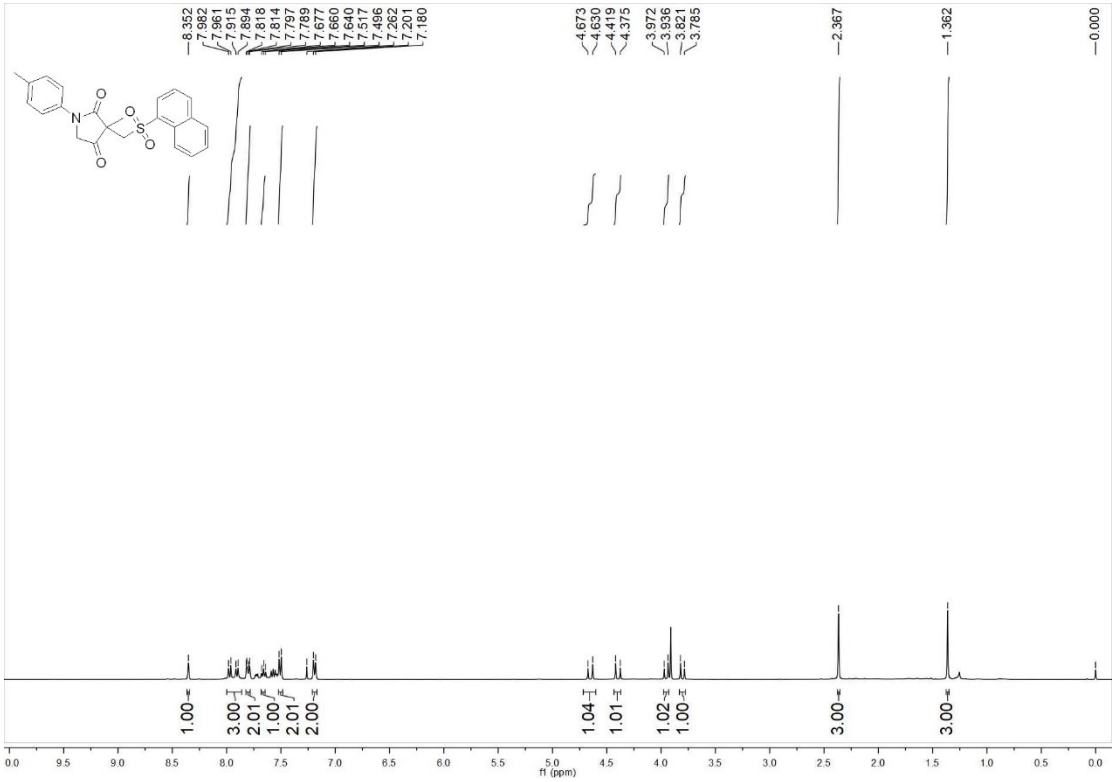


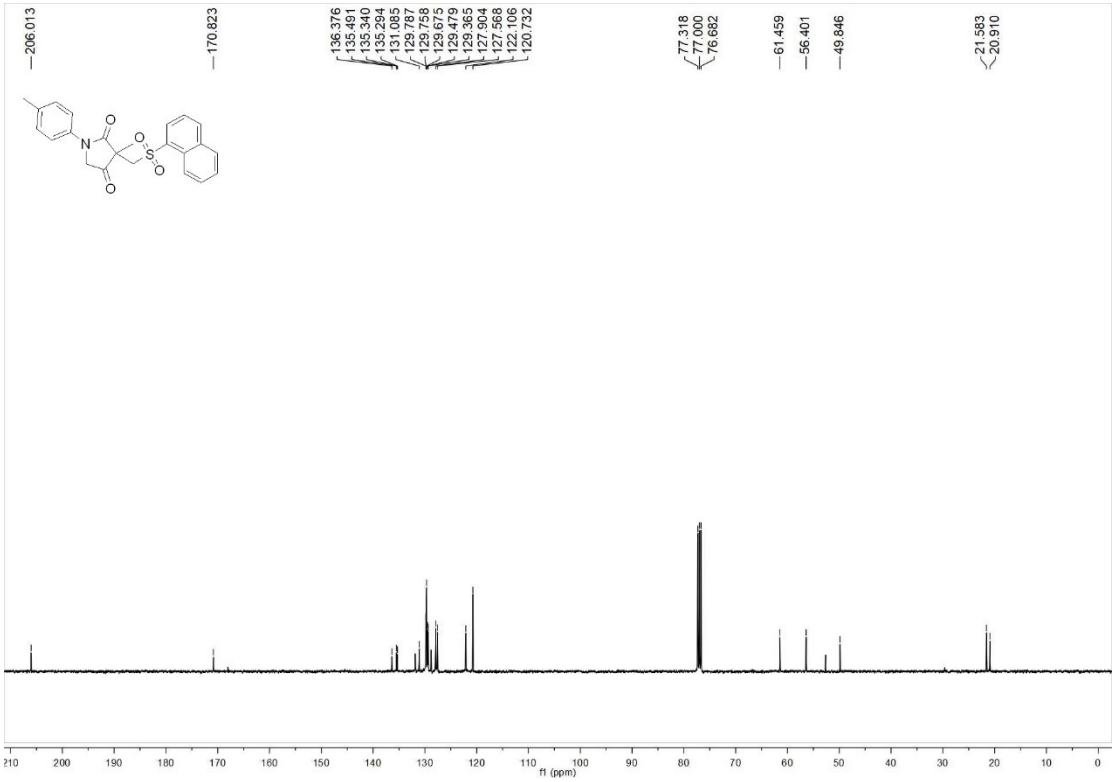
1-(4-(*tert*-Butyl)phenyl)-3-(((3-chlorophenyl)sulfonyl)methyl)-3-methylpyrrolidine-2,4-dione (3aa)



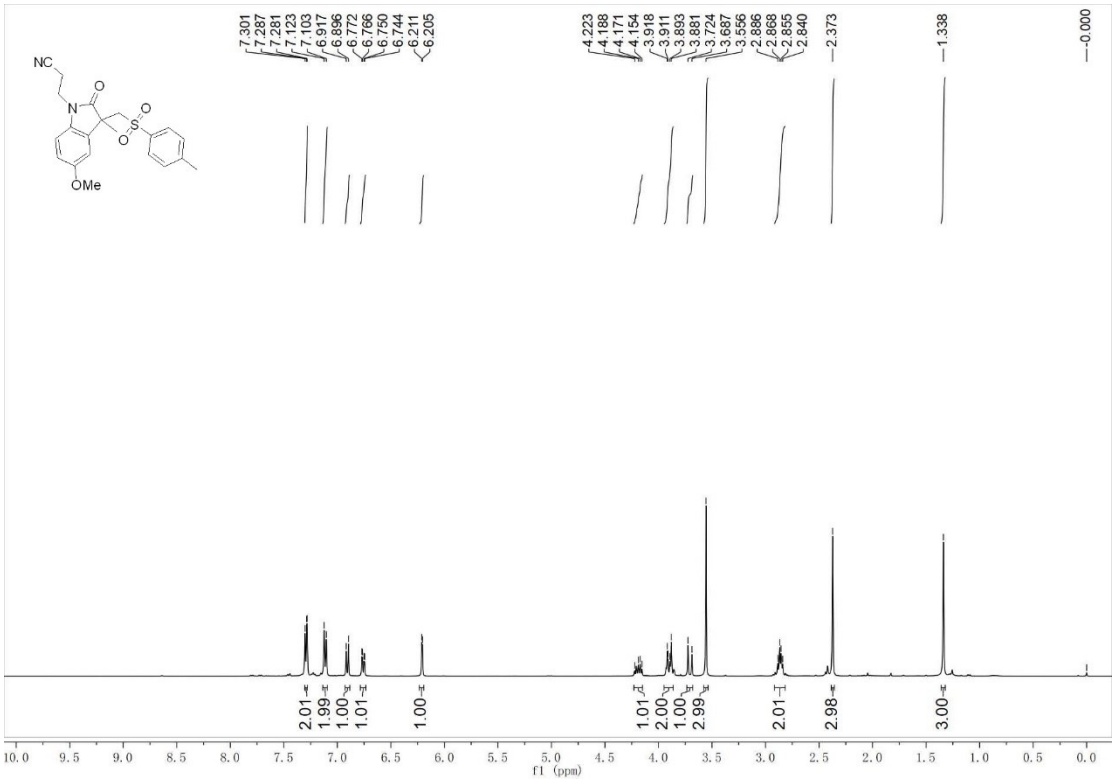


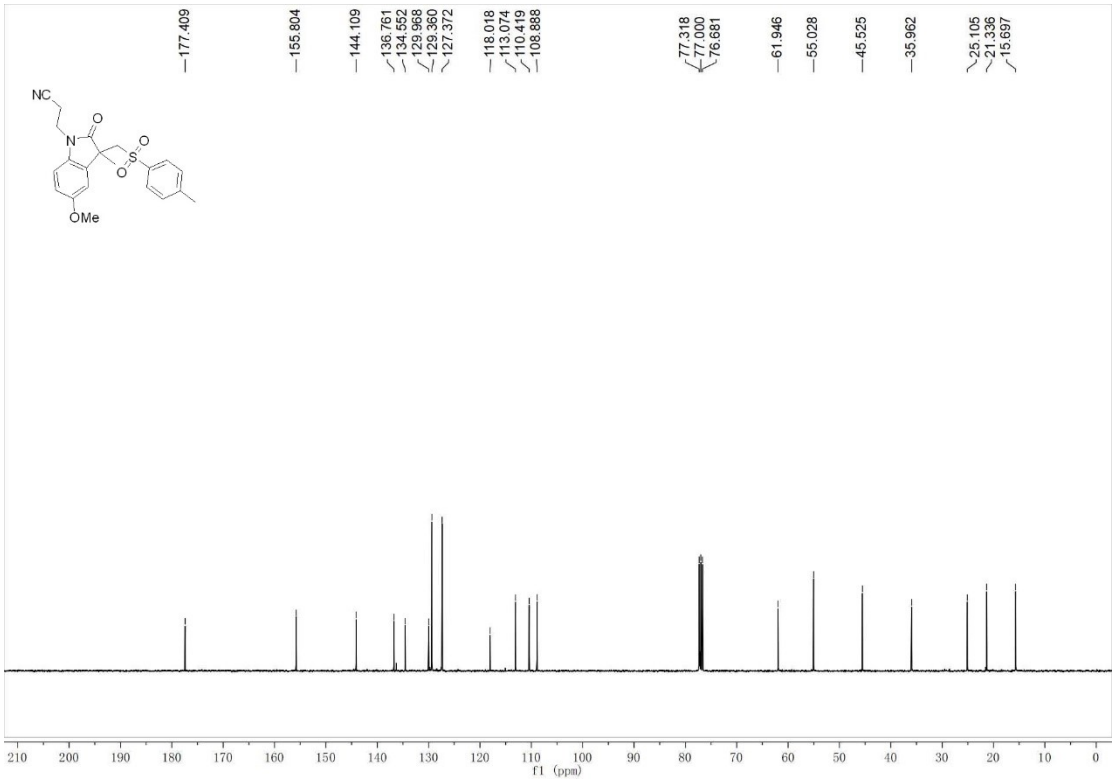
3-Methyl-3-((naphthalen-1-ylsulfonyl)methyl)-1-(*p*-tolyl)pyrrolidine-2,4-dione (3ab)



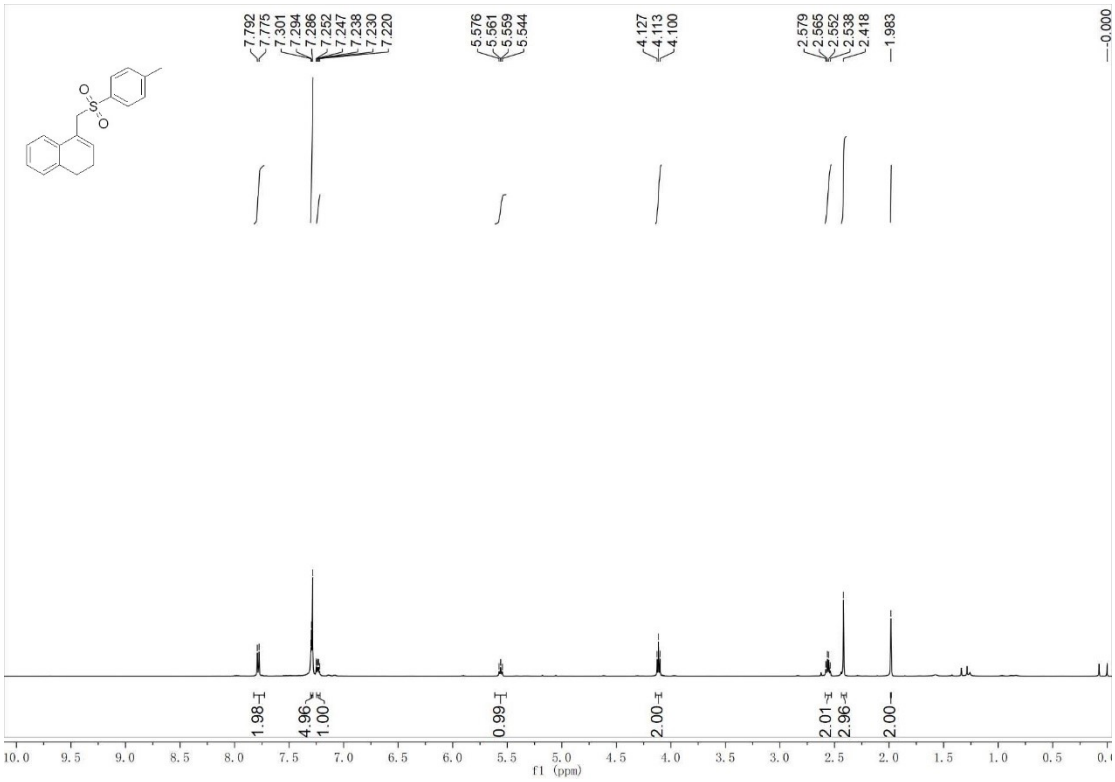


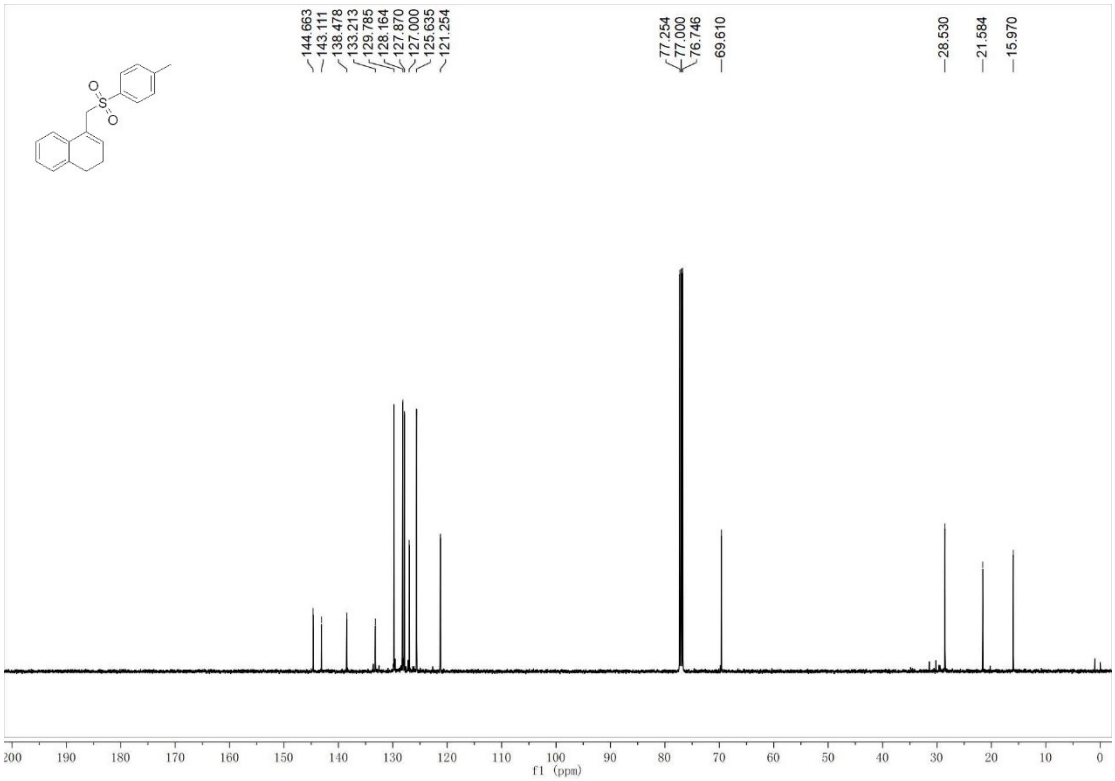
3-(5-Methoxy-3-methyl-2-oxo-3-(tosylmethyl)indolin-1-yl)propanenitrile (4a)





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





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

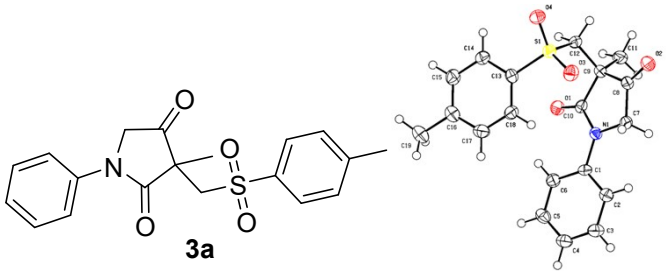


Table 1 Crystal data and structure refinement for 3a.

Identification code	**3a**
Empirical formula	$C_{19}H_{19}NO_4S$
Formula weight	357.41
Temperature/K	150.0
Crystal system	monoclinic
Space group	Cc
a/Å	14.0585(9)
b/Å	6.5274(3)
c/Å	19.2202(11)
α/°	90
β/°	106.051(2)
γ/°	90
Volume/Å^3	1694.99(17)
Z	4
ρ_{calc}g/cm^3	1.401
μ/mm^{-1}	0.215
F(000)	752.0
Crystal size/mm^3	0.15 × 0.08 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.03 to 52.87
Index ranges	-17 ≤ h ≤ 16, -8 ≤ k ≤ 7, -24 ≤ l ≤ 24
Reflections collected	7146
Independent reflections	2980 [R_{int} = 0.0399, R_{sigma} = 0.0488]
Data/restraints/parameters	2980/2/228
Goodness-of-fit on F^2	1.079
Final R indexes [I>=2σ (I)]	R_1 = 0.0406, wR_2 = 0.0846
Final R indexes [all data]	R_1 = 0.0532, wR_2 = 0.0951
Largest diff. peak/hole / e Å^{-3}	0.33/-0.34
Flack parameter	0.05(5)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 3a.

U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	6532.6(7)	3984.5(15)	6186.1(5)	26.9(3)
O4	7446(2)	3220(5)	6652.9(15)	33.5(7)
O1	3835(2)	2618(4)	5336.9(14)	29.2(7)
O2	5198(2)	7923(5)	6972.9(15)	39.0(8)
O3	6457(3)	6138(4)	5998.7(15)	33.6(8)
C1	3435(3)	6484(6)	4489(2)	23.2(9)
C10	4081(3)	4306(6)	5587(2)	22.6(9)
C13	6241(3)	2553(6)	5376(2)	25.7(9)
N1	3909(2)	6148(5)	5240.7(16)	22.8(8)
C7	4254(3)	7920(6)	5721(2)	25.0(9)
C8	4775(3)	6967(6)	6437(2)	27.1(9)
C6	3249(3)	4871(6)	3995(2)	27.7(10)
C12	5603(3)	3463(6)	6638(2)	24.9(9)
C18	5745(3)	3454(7)	4723(2)	29.7(10)
C11	3969(3)	3979(7)	6862(2)	32.6(11)
C14	6517(3)	506(6)	5412(2)	29.0(10)
C2	3176(3)	8459(7)	4252(2)	28.6(10)
C4	2561(3)	7250(7)	3027(2)	32.3(11)
C9	4638(3)	4655(6)	6391(2)	24.2(9)
C17	5513(4)	2265(7)	4100(2)	34.3(11)
C5	2815(3)	5275(7)	3266(2)	32.0(11)
C3	2747(4)	8831(7)	3518(2)	36.2(11)
C15	6281(4)	-643(7)	4779(2)	32.9(10)
C16	5765(3)	215(7)	4122(2)	31.0(10)
C19	5465(4)	-1116(8)	3450(2)	40.3(12)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for 3a.

The Anisotropic displacement factor exponent takes the form: -$2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	26.0(6)	26.5(5)	27.2(5)	-1.0(4)	5.8(4)	1.0(5)
O4	22.8(16)	40.2(18)	33.7(16)	-0.7(13)	1.6(13)	3.8(14)
O1	37.5(19)	18.2(14)	30.2(14)	-1.4(12)	6.8(14)	-3.0(13)
O2	44(2)	35.3(18)	29.8(15)	-11.2(14)	-2.6(15)	4.4(16)
O3	38.9(19)	22.9(15)	39.6(18)	0.8(13)	11.6(16)	-2.6(15)
C1	20(2)	26(2)	20.6(19)	3.7(15)	1.8(17)	1.2(17)
C10	24(2)	19(2)	27(2)	3.9(16)	12.2(18)	2.4(17)
C13	27(2)	27(2)	25.0(19)	-2.2(16)	10.3(18)	-2.4(18)
N1	26(2)	21.0(17)	20.2(15)	-0.8(13)	4.8(14)	-0.7(15)
C7	26(2)	21(2)	25.2(19)	-3.7(16)	3.1(17)	-0.4(18)
C8	27(2)	32(2)	22.2(19)	-2.8(18)	6.2(17)	5(2)
C6	30(3)	25(2)	28(2)	1.5(17)	8.6(18)	0.0(19)
C12	28(2)	23(2)	24.0(19)	3.8(15)	7.1(18)	4.0(17)
C18	32(3)	29(2)	30(2)	4.3(18)	10.3(19)	6(2)
C11	33(3)	41(3)	25(2)	6.0(18)	10.4(19)	8(2)
C14	26(2)	30(2)	30(2)	1.9(17)	6.3(19)	2(2)
C2	30(3)	29(2)	26(2)	-0.7(17)	5.8(19)	0.7(19)
C4	28(3)	43(3)	25(2)	5.1(19)	5.1(19)	2(2)
C9	27(2)	25.1(19)	20.1(18)	-1.7(16)	5.6(18)	1.0(18)
C17	32(3)	42(3)	27(2)	5.5(19)	6(2)	2(2)
C5	29(3)	39(3)	26(2)	-5.5(18)	5(2)	-5(2)
C3	35(3)	42(3)	28(2)	5(2)	3(2)	6(2)
C15	41(3)	23(2)	36(2)	-0.9(19)	12(2)	-2(2)
C16	27(3)	37(3)	31(2)	-4.3(19)	12(2)	-8(2)
C19	43(3)	47(3)	31(2)	-9(2)	11(2)	-9(2)

Table 4 Bond Lengths for 3a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O4	1.438(3)	C7	C8	1.503(5)
S1	O3	1.448(3)	C8	C9	1.521(6)
S1	C13	1.764(4)	C6	C5	1.390(6)
S1	C12	1.790(4)	C12	C9	1.521(6)
O1	C10	1.213(5)	C18	C17	1.389(6)
O2	C8	1.210(5)	C11	C9	1.538(6)
C1	N1	1.430(5)	C14	C15	1.389(6)
C1	C6	1.394(6)	C2	C3	1.394(6)
C1	C2	1.382(6)	C4	C5	1.381(6)
C10	N1	1.363(5)	C4	C3	1.373(6)
C10	C9	1.545(5)	C17	C16	1.382(7)
C13	C18	1.387(6)	C15	C16	1.389(6)
C13	C14	1.388(6)	C16	C19	1.516(6)
N1	C7	1.476(5)			

Table 5 Bond Angles for 3a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O4	S1	O3	118.93(19)	C7	C8	C9	109.6(3)
O4	S1	C13	108.43(19)	C5	C6	C1	119.4(4)
O4	S1	C12	106.12(18)	C9	C12	S1	117.4(3)
O3	S1	C13	108.16(18)	C13	C18	C17	118.9(4)
O3	S1	C12	106.98(19)	C13	C14	C15	118.8(4)
C13	S1	C12	107.74(19)	C1	C2	C3	119.9(4)
C6	C1	N1	121.3(3)	C3	C4	C5	119.4(4)
C2	C1	N1	118.9(3)	C8	C9	C10	103.0(3)
C2	C1	C6	119.8(3)	C8	C9	C12	113.6(4)
O1	C10	N1	128.0(4)	C8	C9	C11	109.8(3)
O1	C10	C9	122.7(3)	C12	C9	C10	113.4(3)
N1	C10	C9	109.3(3)	C12	C9	C11	107.9(3)
C18	C13	S1	120.7(3)	C11	C9	C10	108.9(3)
C18	C13	C14	121.1(4)	C16	C17	C18	121.2(4)
C14	C13	S1	118.3(3)	C4	C5	C6	120.8(4)
C1	N1	C7	119.5(3)	C4	C3	C2	120.7(4)
C10	N1	C1	126.8(3)	C16	C15	C14	121.1(4)
C10	N1	C7	113.7(3)	C17	C16	C15	118.9(4)
N1	C7	C8	104.0(3)	C17	C16	C19	121.4(4)
O2	C8	C7	124.5(4)	C15	C16	C19	119.7(4)
O2	C8	C9	125.9(4)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 3a.

Atom	*x*	*y*	*z*	U(eq)
H7A	3689.64	8771.62	5762.28	30
H7B	4713.65	8780.62	5539.27	30
H6	3416.7	3507.31	4154.6	33
H12A	5443.13	1984.89	6582.58	30
H12B	5893.21	3727.96	7160.64	30
H18	5567.88	4861.36	4702.81	36
H11A	3364.55	4807.87	6741.03	49
H11B	4321.74	4167.67	7374.26	49
H11C	3795.71	2530.69	6770.4	49
H14	6860.99	-98.4	5860.41	35
H2	3291.43	9560.42	4587.78	34
H4	2260.39	7511.71	2528.64	39
H17	5174.16	2871.38	3650.29	41
H5	2691.98	4179.64	2927.05	38
H3	2580.28	10193.04	3355.15	43
H15	6476.41	-2039.5	4795.44	39
H19A	4792.92	-1636.54	3388.79	60
H19B	5925.41	-2270.15	3502.71	60
H19C	5483.19	-302.6	3025.78	60