Supporting Information

An Efficient Synthesis of 4,5-Diaryl-3,4-Dihydropyrimidin-2(1*H*)-one via a Cesium Carbonate-Promoted Direct Condensation of 1-Aryl-2-propanone with 1,1'-(Arylmethylene)diurea

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General information

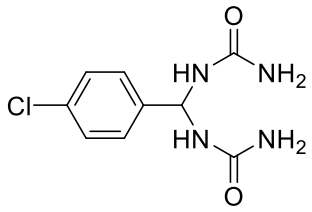
1-Aryl-2-propanone, cesium carbonate, and ethanol were purchased from chemical coM.p.anies and used directly without further purification (without the need of precautions to exclude air and moisture unless otherwise noted). Starting materials **1a-j** were prepared according to reported methods.[1] Analytical thin layer chromatography (TLC) was performed using silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm). Flash chromatography was performed using Merck silica gel (200-300 mesh) for column chromatography with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H, ^{19}F, and ^{13}C NMR spectra were recorded in d^{6}-DMSO on Jeol 400 MHz spectrometer. Tetramethylsilane (TMS) served as internal standard for ^{1}H, ^{19}F, and ^{13}C NMR analysis.

Experimental procedure

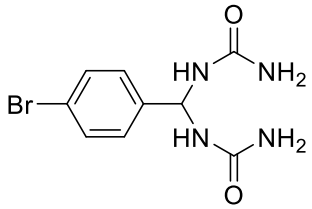
General procedure for the preparation of 1,1'-(arylmethylene)diurea.[1] To a 250 mL round-bottomed flask was sequentially added aryl aldehyde (30 mmol), urea (90 mmol), a catalytic amount of *p*-toluenesulfonic acid (3 mmol) and toluene (100 mL). The reaction mixture was refluxed for overnight. After reaction, the precipitate was collected by filtration, washed with saturated $NaHCO_3$ (10 mL), pure water (20 mL), and Et_2O (20 mL). It was further dried in oven at 120 ºC for half an hour to give the desired product of diurea.

Typical procedure for the condensation of 1,1'-(arylmethylene)diurea with 1-aryl-2-propanone: To a mixture of diurea (0.5 mmol) and 1-aryl-2-propanone (0.6 mmol) was added absolute ethanol (3 mL), and it was stirred at room teM.p.erature for 5 minutes. Then Cs_2CO_3 (1 mmol) was added and the reaction mixture was stirred vigorously at 70 ºC for 24 h. After the coM.p.letion of the reaction, solvent was removed under vacuum. The residue was purified by silica gel column chromatography by using EtOAc/petroleum ether or CH_2Cl_2/MeOH as eluant to afford the desired product of 4,5-biaryl-3,4-dihydropyrimidin-2(1*H*)-one.

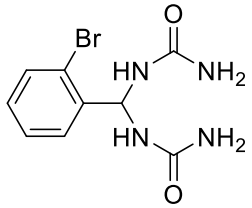
Characterization data of products



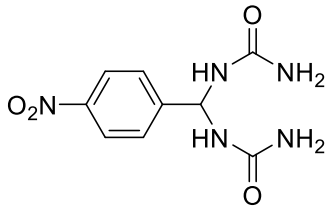
1,1'-((4-Chlorophenyl)methylene)diurea (1a): 6.6 g. Yield = 91%. White solid. **M.p.:** 190.5-191.2 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.40 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 6.79 (d, *J* = 8.3 Hz, 2H), 6.08 (t, *J* = 8.3 Hz, 1H), 5.72 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.8, 141.9, 131.6, 128.1, 128.0, 58.6 ppm. **FTIR (KBr, neat):** ν 3453, 3312, 1667, 1608, 866, 817 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_9H_{12}ClN_4O_2$: 243.0643, found: 243.0643.



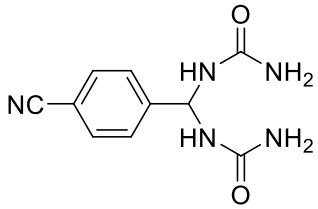
1,1'-((4-Bromophenyl)methylene)diurea (1b): 4.4 g. Yield = 51%. White solid. **M.p.:** 206.6-207.5 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.3 Hz, 2H), 6.06 (t, *J* = 8.3 Hz, 1H), 5.72 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.2, 142.8, 131.4, 128.8, 120.5, 59.1 ppm. **FTIR (KBr, neat):** ν 3453, 3312, 1667, 1608, 866, 813 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_9H_{12}BrN_4O_2$: 287.0138, found: 287.0137.



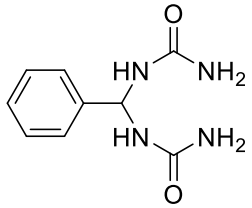
1,1'-((2-Bromophenyl)methylene)diurea (1c): 6.3 g. Yield = 73%. White solid. **M.p.:** 199.2-199.7 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.60 (d, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 6.7 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.23 (t, *J* = 7.0 Hz, 1H), 6.71 (d, *J* = 6.9 Hz, 2H), 6.25 (t, *J* = 7.5 Hz, 1H), 5.63 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.4, 141.2, 132.7, 129.4, 128.0, 127.5, 122.4, 59.6 ppm. **FTIR (KBr, neat):** ν 3430, 3331, 1668, 1612, 750, 719 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_9H_{12}BrN_4O_2$: 287.0138, found: 287.0140.



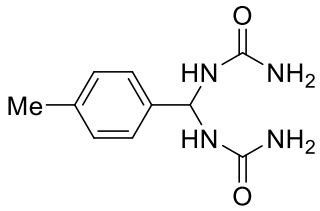
1,1'-((4-Nitrophenyl)methylene)diurea (1d): 6.7 g. Yield = 88%. White solid. **M.p.:** 209.8-210.4 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 8.6 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 6.17 (t, *J* = 8.2 Hz, 1H), 5.82 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.8, 150.8, 146.5, 127.4, 123.4, 58.8 ppm. **FTIR (KBr, neat):** ν 3459, 3312, 1682, 1516, 1351, 856, 835 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_9H_{12}N_5O_4$: 254.0884, found: 254.0881.



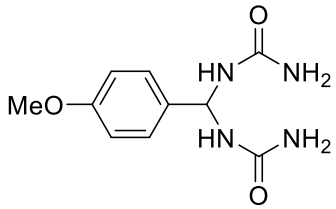
1,1'-((4-Cyanophenyl)methylene)diurea (1e): 6.3 g. Yield = 90%. White solid. **M.p.:** 222.6-222.9 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.82 (d, *J* = 8.1 Hz, 2H), 7.49 (d, *J* = 8.3 Hz, 2H), 6.90 (d, *J* = 8.2 Hz, 2H), 6.12 (t, *J* = 8.1 Hz, 1H), 5.76 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.2, 149.1, 132.6, 127.6, 119.5, 110.1, 59.3 ppm. **FTIR (KBr, neat):** ν 3352, 3300, 2234, 1656, 862, 825 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{10}H_{12}N_5O_2$: 234.0986, found: 234.0987.



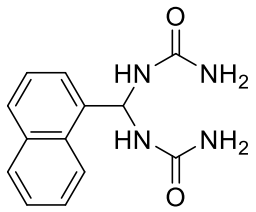
1,1'-(Phenylmethylene)diurea (1f): 4.0 g. Yield = 64%. White solid. **M.p.:** 190.6-191.2 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.34-7.31 (m, 4H), 7.27-7.23 (m, 1H), 6.72 (d, *J* = 8.3 Hz, 2H), 6.12 (t, *J* = 8.3 Hz, 1H), 5.68 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.2, 143.2, 128.6, 127.6, 126.5, 59.6 ppm. **FTIR (KBr, neat):** ν 3420, 3312, 1667, 1535, 747, 699 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_9H_{13}N_4O_2$: 209.1033, found: 209.1033.



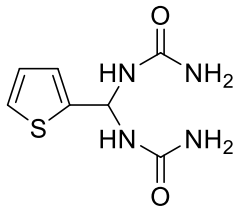
1,1'-(*p*-Tolylmethylene)diurea (1g): 3.7 g. Yield = 55%. White solid. **M.p.:** 196.2-196.8 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.20 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.1 Hz, 2H), 6.67 (d, *J* = 8.1 Hz, 2H), 6.08 (t, *J* = 8.3 Hz, 1H), 5.67 (s, 4H), 2.28 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.2, 140.2, 136.6, 129.1, 126.4, 59.4, 21.2 ppm. **FTIR (KBr, neat):** ν 3458, 3312, 1678, 1532, 864, 814 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{10}H_{15}N_4O_2$: 223.1190, found: 223.1191.



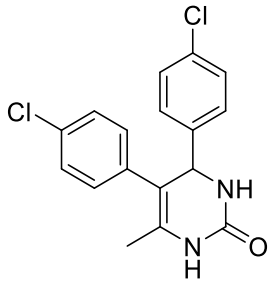
1,1'-((4-Methoxyphenyl)methylene)diurea (1h): 5.2 g. Yield = 73%. White solid. **M.p.:** 186.6-187.3 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.24 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.70 (d, *J* = 8.0 Hz, 2H), 6.09 (t, *J* = 8.1 Hz, 1H), 5.70 (s, 4H), 3.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.9, 158.3, 135.1, 127.7, 114.0, 59.2, 55.6 ppm. **FTIR (KBr, neat):** ν 3428, 3308, 1671, 1515, 1261, 1176, 865, 836 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{10}H_{15}N_4O_3$: 239.1139, found: 239.1134.



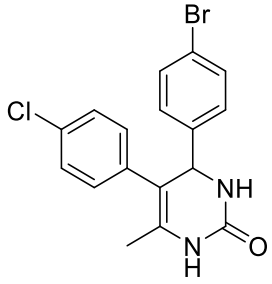
1,1'-(Naphthalen-1-ylmethylene)diurea (1i): 6.8 g. Yield = 88%. White solid. **M.p.:** 221.5-222.7 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.02 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 7.4 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 1H), 7.64-7.47 (m, 4H), 6.91 (t, *J* = 7.8 Hz, 1H), 6.82 (d, *J* = 7.7 Hz, 2H), 5.66 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.4, 138.1, 133.5, 130.3, 128.6, 128.0, 126.3, 125.8, 125.2, 123.4, 122.6, 56.5 ppm. **FTIR (KBr, neat):** ν 3418, 3297, 1667, 1608, 882, 770 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{13}H_{15}N_4O_2$: 259.1190, found: 259.1189.



1,1'-(Thiophen-2-ylmethylene)diurea (1j): 4.2 g. Yield = 65%. White solid. **M.p.:** 197.4-198.9 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 7.41-7.35 (m, 1H), 6.96 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.91-6.88 (m, 1H), 6.86 (d, *J* = 8.5 Hz, 2H), 6.32 (t, *J* = 8.4 Hz, 1H), 5.75 (s, 4H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.5, 147.7, 126.8, 124.9, 123.7, 56.2 ppm. **FTIR (KBr, neat):** ν 3467, 3284, 1671, 1589, 1282, 1123 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_7H_{11}N_4O_2S$: 215.0597, found: 215.0602.

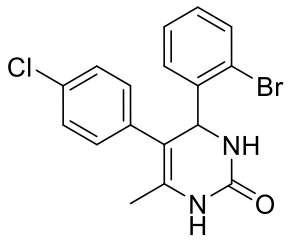


4,5-*bis*(4-Chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (3a): 124.6 mg. Yield = 75%. Yellow solid. **M.p.:** 214.7-215.8 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.50 (s, 1H), 7.38 (s, 1H), 7.35-7.31 (m, 2H), 7.30-7.26 (m, 2H), 7.18-7.14 (m, 2H), 7.11-7.06 (m, 2H), 5.13 (s, 1H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.8, 143.0, 136.7, 131.8, 131.2, 130.9, 130.8, 128.7, 128.4, 128.1, 106.8, 58.5, 16.2 ppm. **FTIR (KBr, neat):** ν 3265, 1693, 1491, 1241, 1091, 758 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}Cl_2N_2O$: 333.0556, found: 333.0558.

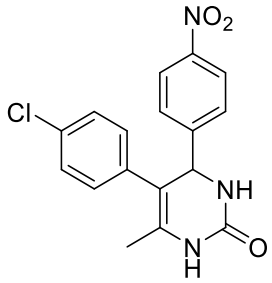


4-(4-Bromophenyl)-5-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (3b): 161.6 mg. Yield = 86%. Brown solid. **M.p.:** 206.8-208.2°C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.51 (s, 1H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.39 (s, 1H), 7.30-7.24 (m, 2H), 7.14-7.05 (m, 4H), 5.12 (s, 1H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$)**: δ 152.8, 143.4, 136.7, 131.3, 131.2, 130.9, 130.8, 129.1, 128.1, 120.4, 106.7, 58.6,

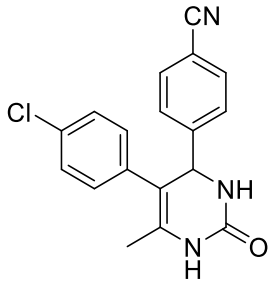
16.2 ppm. **FTIR (KBr, neat):** ν 3268, 1693, 1489, 1241, 757 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}BrClN_2O$: 377.0051, found: 377.0051.



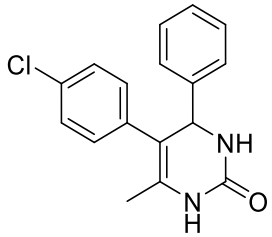
4-(2-Bromophenyl)-5-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (3c): 126.8 mg. Yield = 67%. Yellow solid. **M.p.:** 189.8-191.3 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.56 (s, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.40-7.33 (m, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 2H), 5.58 (s, 1H), 1.69 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.5, 142.6, 136.2, 132.6, 131.3, 131.1, 131.0, 129.9, 129.5, 128.5, 128.1, 121.9, 106.3, 58.6, 16.1 ppm. **FTIR (KBr, neat):** ν 3220, 1694, 1493, 1253, 1095, 737 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}BrClN_2O$: 377.0051, found: 377.0050.



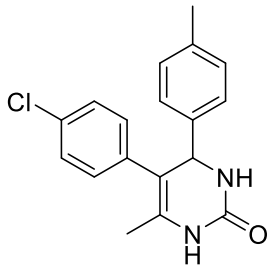
5-(4-Chlorophenyl)-6-methyl-4-(4-nitrophenyl)-3,4-dihydropyrimidin-2(1*H*)-one (3d): 88.8 mg. Yield = 50%. Brown solid. **M.p.:** 200.2-201.7 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$)**: δ 8.51 (s, 1H), 7.44-7.37 (m, 3H), 7.35-7.30 (m, 2H), 7.16 (d, *J* = 8.3 Hz, 2H), 7.02 (d, *J* = 8.3 Hz, 2H), 5.13 (s, 1H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.8, 148.5, 136.6, 131.1, 131.0, 130.8, 128.1, 126.6, 125.3, 124.4, 107.4, 54.2, 16.3 ppm. **FTIR (KBr, neat):** ν 2926, 1721, 1491, 1262, 1093, 1014, 831 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}ClN_3O_3$: 344.0796, found: 344.0799.



4-(5-(4-Chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)benzonitrile (3e): 80.3 mg. Yield = 50%. Yellow solid. **M.p.:** 249.3-250.6 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.57 (s, 1H), 7.78-7.72 (m, 2H), 7.47 (s, 1H), 7.33-7.30 (m, 2H), 7.30-7.26 (m, 2H), 7.11-7.06 (m, 2H), 5.24 (s, 1H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.7, 149.3, 136.4, 132.6, 131.3, 131.2, 130.9, 128.2, 127.8, 118.8, 110.1, 106.2, 58.8, 16.2 ppm. **FTIR (KBr, neat):** ν 3397, 2226, 1679, 1475, 1242, 759 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{15}ClN_3O$: 324.0898, found: 324.0904.

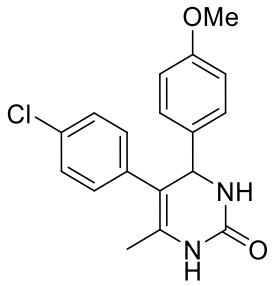


5-(4-Chlorophenyl)-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1*H*)-one (3f): 97.4 mg. Yield = 65%. Light yellow solid. **M.p.:** 179.6-180.1 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.45 (s, 1H), 7.34 (s, 1H), 7.29-7.23 (m, 4H), 7.22-7.18 (m, 1H), 7.16 (d, *J* = 7.9 Hz, 2H), 7.10-7.05 (m, 2H), 5.08 (s, 1H), 1.74 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 153.0, 144.0, 137.0, 131.1, 130.7, 130.7, 128.5, 128.1, 127.3, 126.8, 107.1, 59.2, 16.3 ppm. **FTIR (KBr, neat):** ν 3055, 1690, 1493, 1014, 744, 698 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{16}ClN_2O$: 299.0946, found: 299.0947.

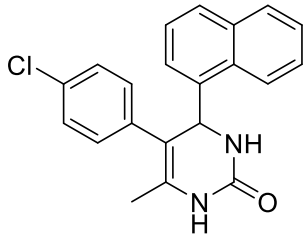


5-(4-Chlorophenyl)-6-methyl-4-(*p*-tolyl)-3,4-dihydropyrimidin-2(1*H*)-one (3g): 118.2 mg. Yield = 76%. Light yellow solid. **M.p.:** 215.6-216.8 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.44 (s, 1H), 7.30-7.24 (m, 3H), 7.10-7.03 (m, 6H), 5.04 (s, 1H), 2.23 (s, 3H), 1.74 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.9, 141.1, 137.1, 136.4,

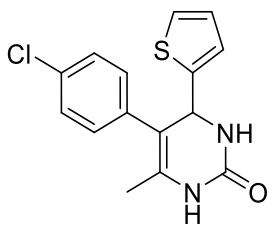
131.1, 130.6, 130.5, 129.0, 128.1, 126.8, 107.2, 58.9, 20.7, 16.3 ppm. **FTIR (KBr, neat):** ν 3271, 1694, 1491, 1241, 832, cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{18}ClN_2O$: 313.1102, found: 313.1102.



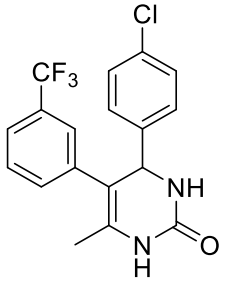
5-(4-Chlorophenyl)-4-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (3h): 120.0 mg. Yield = 73%. Yellow solid. **M.p.:** 161.8-163.2 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.42 (s, 1H), 7.29-7.24 (m, 3H), 7.10-7.06 (m, 4H), 6.85-6.80 (m, 2H), 5.02 (s, 1H), 3.69 (s, 3H), 1.74 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 158.5, 152.9, 137.1, 136.2, 131.1, 130.6, 130.5, 128.1, 128.0, 113.8, 107.3, 58.6, 55.0, 16.3 ppm. **FTIR (KBr, neat):** ν 3238, 1679, 1510, 1248, 1174, 834 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{18}ClN_2O_2$: 329.1051, found: 329.1054.



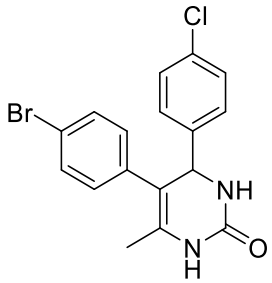
5-(4-Chlorophenyl)-6-methyl-4-(naphthalen-1-yl)-3,4-dihydropyrimidin-2(1*H*)-one (3i): 112.3 mg. Yield = 64%. Light yellow solid. **M.p.:** 250.4-251.6 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.58 (s, 1H), 8.29-8.19 (m, 1H), 7.92-7.86 (m, 1H), 7.82-7.75 (m, 1H), 7.53-7.40 (m, 4H), 7.36 (s, 1H), 7.15 (d, *J* = 7.8 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 5.99 (s, 1H), 1.81 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 152.7, 148.0, 144.0, 138.7, 136.9, 133.7, 131.1, 131.0, 130.6, 130.4, 128.6, 128.1, 127.9, 126.0, 125.9, 125.6, 123.6, 106.8, 16.3 ppm. **FTIR (KBr, neat):** ν 3230, 1703, 1493, 1253, 776 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{21}H_{18}ClN_2O$: 349.1102, found: 349.1103.



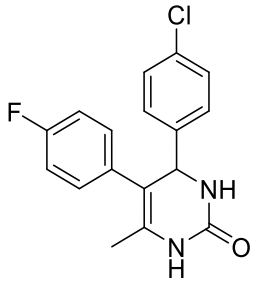
5-(4-Chlorophenyl)-6-methyl-4-(thiophen-2-yl)-3,4-dihydropyrimidin-2(1*H*)-one (3j): 128.8 mg. Yield = 85%. Light yellow solid. **M.p.:** 193.2-194.4 °C. **^{1}H NMR (400 MHz, DMSO-d_6):** δ 8.63 (s, 1H), 7.55 (s, 1H), 7.35 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.33-7.28 (m, 2H), 7.21-7.14 (m, 2H), 6.87 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.82-6.77 (m, 1H), 5.39 (s, 1H), 1.76 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-d_6):** δ 152.9, 148.5, 136.6, 131.1, 131.1, 130.8, 128.1, 126.6, 125.3, 124.4, 107.5, 54.2, 16.3 ppm. **FTIR (KBr, neat):** ν 3244, 1686, 1490, 1389, 1247, 1091, 839, 698 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{15}H_{14}ClN_2OS$: 305.0510, found: 305.0509.



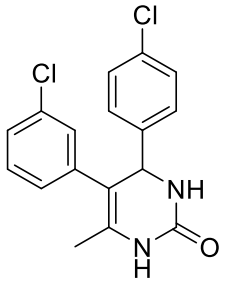
4-(4-Chlorophenyl)-6-methyl-5-(3-(trifluoromethyl)phenyl)-3,4-dihydropyrimidin-2(1H)-one (4b): 132.6 mg. Yield = 72%. Yellow solid. **M.p.:** 109.8-110.4 °C. **^{1}H NMR (400 MHz, DMSO-d_6):** δ 8.60 (s, 1H), 7.51-7.42 (m, 3H), 7.40-7.31 (m, 4H), 7.23 – 7.14 (m, 2H), 5.22 (s, 1H), 1.74 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-d_6):** δ 152.7, 142.9, 139.0, 133.6, 131.9, 131.7, 129.2, 129.0 (q, *J* = 31.3 Hz), 128.8, 128.5, 125.7 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 271.0 Hz), 123.0 (q, *J* = 4.1 Hz), 106.6, 58.4, 16.2 ppm. **^{19}F NMR (376 MHz, DMSO-D_6):** δ -60.98 ppm. **FTIR (KBr, neat):** ν 3238, 1683, 1490, 1339, 1126, 804, 704 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{15}ClF_3N_2O$: 367.0820, found: 367.0822.



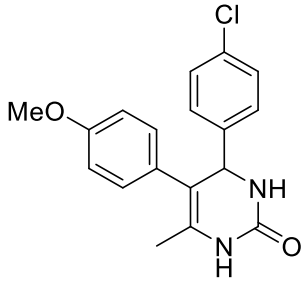
5-(4-Bromophenyl)-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4c): 149.6 mg. Yield = 79%. Light yellow solid. **M.p.:** 209.5-209.8 °C. **^{1}H NMR (400 MHz, DMSO-d_6):** δ 8.51 (s, 1H), 7.42-7.37 (m, 3H), 7.35-7.30 (m, 2H), 7.18-7.14 (m, 2H), 7.04-7.00 (m, 2H), 5.12 (s, 1H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-d_6):** δ 152.8, 143.0, 137.1, 131.8, 131.5, 131.0, 130.9, 128.7, 128.4, 119.3, 106.8, 58.4, 16.2 ppm. **FTIR (KBr, neat):** ν 3269, 1692, 1490, 1240, 1099, 844, 778 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}BrClN_2O$: 377.0051, found: 377.0050.



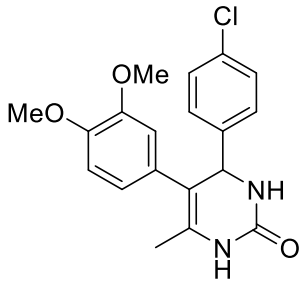
4-(4-Chlorophenyl)-5-(4-fluorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4d): 139.5 mg. Yield = 88%. Yellow solid. **M.p.:** 189.0-190.2 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.46 (s, 1H), 7.35 (s, 1H), 7.34-7.31 (m, 2H), 7.18-7.14 (m, 2H), 7.11-7.02 (m, 4H), 5.10 (s, 1H), 1.71 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 160.6 (d, *J* = 241.7 Hz), 152.8, 143.0, 134.1 (d, *J* = 3.3 Hz), 131.7, 131.3 (d, *J* = 8.0 Hz), 130.4, 128.7, 128.4, 115.0 (d, *J* = 21.1 Hz), 107.0, 58.8, 16.2 ppm. **^{19}F NMR (376 MHz, DMSO-*d*$_6$):** δ -115.91 ppm. **FTIR (KBr, neat):** ν 3259, 1694, 1491, 1242, 1094, 1014, 845, 780 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}ClFN_2O$: 317.0851, found: 317.0851.



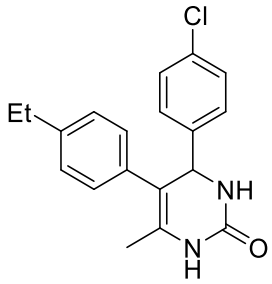
5-(3-Chlorophenyl)-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4e): 155.9 mg. Yield = 94%. Yellow solid. **M.p.:** 93.8-95.3 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.59 (s, 1H), 7.45 (s, 1H), 7.32-7.29 (m, 2H), 7.22-7.15 (m, 4H), 7.11 (s, 1H), 7.04-6.98 (m, 1H), 5.17 (s, 1H), 1.75 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 153.1, 142.9, 140.2, 133.0, 132.1, 131.5, 130.1, 129.1, 128.9, 128.6, 128.3, 126.4, 107.0, 58.6, 16.4 ppm. **FTIR (KBr, neat):** ν 3236, 1682, 1489, 1239, 1091, 785 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{17}H_{15}Cl_2N_2O$: 333.0556, found: 333.0556.



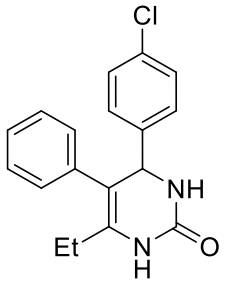
4-(4-Chlorophenyl)-5-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4f): 140.5 mg. Yield = 85%. Yellow solid. **M.p.:** 184.6-186.2 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.42 (s, 1H), 7.35-7.29 (m, 3H), 7.19-7.14 (m, 2H), 6.99-6.94 (m, 2H), 6.82-6.76 (m, 2H), 5.07 (s, 1H), 3.68 (s, 3H), 1.71 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 157.6, 153.1, 143.3, 131.7, 130.5, 129.8, 129.6, 128.7, 128.3, 113.6, 107.7, 58.9, 54.9, 16.2 ppm. **FTIR (KBr, neat):** ν 3259, 2924, 1693, 1489, 1249, 842 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{18}ClN_2O_2$: 329.1051, found: 329.1051.



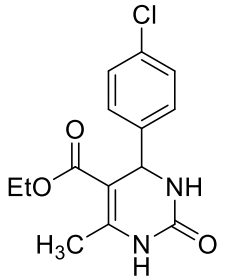
4-(4-Chlorophenyl)-5-(3,4-dimethoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4g): 160.4 mg. Yield = 89%. Yellow solid. **M.p.:** 151.6-152.7 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.36 (s, 1H), 7.34-7.29 (m, 3H), 7.19-7.15 (m, 2H), 6.78 (d, *J* = 8.3 Hz, 1H), 6.63 (d, *J* = 2.0 Hz, 1H), 6.50 (dd, *J* = 8.2, 2.0 Hz, 1H), 5.10 (s, 1H), 3.67 (s, 3H), 3.64 (s, 3H), 1.73 (s, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 153.0, 148.2, 147.2, 143.4, 131.6, 130.2, 129.6, 128.8, 128.3, 121.9, 113.0, 111.4, 108.0, 58.9, 55.4, 55.3, 16.3 ppm. **FTIR (KBr, neat):** ν 3343, 1698, 1515, 1457, 1251, 1135, 1011, 766 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{19}H_{20}ClN_2O_3$: 359.1157, found: 359.1159.



4-(4-Chlorophenyl)-5-(4-ethylphenyl)-6-methyl-3,4-dihydropyrimidin-2(1*H*)-one (4h): 146.5 mg. Yield = 90%. Yellow solid. **M.p.:** 229.8-231.2 ºC. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.40 (s, 1H), 7.35-7.29 (m, 3H), 7.19-7.14 (m, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 6.97 (d, *J* = 8.2 Hz, 2H), 5.07 (s, 1H), 2.52 (q, *J* = 7.6 Hz, 2H), 1.73 (s, 3H), 1.12 (t, *J* = 7.6 Hz, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 153.0, 143.2, 141.6, 135.1, 131.7, 130.1, 129.2, 128.8, 128.4, 127.5, 107.8, 58.7, 27.7, 16.3, 15.3 ppm. **FTIR (KBr, neat):** ν 3223, 2928, 1686, 1241, 1088, 842 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{19}H_{20}ClN_2O$: 327.1259, found: 327.1260.



4-(4-Chlorophenyl)-6-ethyl-5-phenyl-3,4-dihydropyrimidin-2(1*H*)-one (4i): 101.8 mg. Yield = 65%. Yellow solid. **M.p.:** 184.8-185.7 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 8.41 (s, 1H), 7.34-7.30 (m, 3H), 7.23 (t, *J* = 7.4 Hz, 2H), 7.17-7.12 (m, 3H), 7.03-6.98 (m, 2H), 5.05 (d, *J* = 2.5 Hz, 1H), 2.04-1.94 (m, 2H), 1.03 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 153.2, 143.1, 137.9, 135.5, 131.7, 129.3, 128.7, 128.4, 128.2, 126.5, 107.7, 59.0, 22.7, 13.0 ppm. **FTIR (KBr, neat):** ν 3235, 1679, 1489, 1227, 1091, 1014, 765, 701 cm^{-1}. **HRMS (ESI, m/z):** $[M+H]^+$, calcd. for $C_{18}H_{18}ClN_2O$: 313.1102, found: 313.1102.

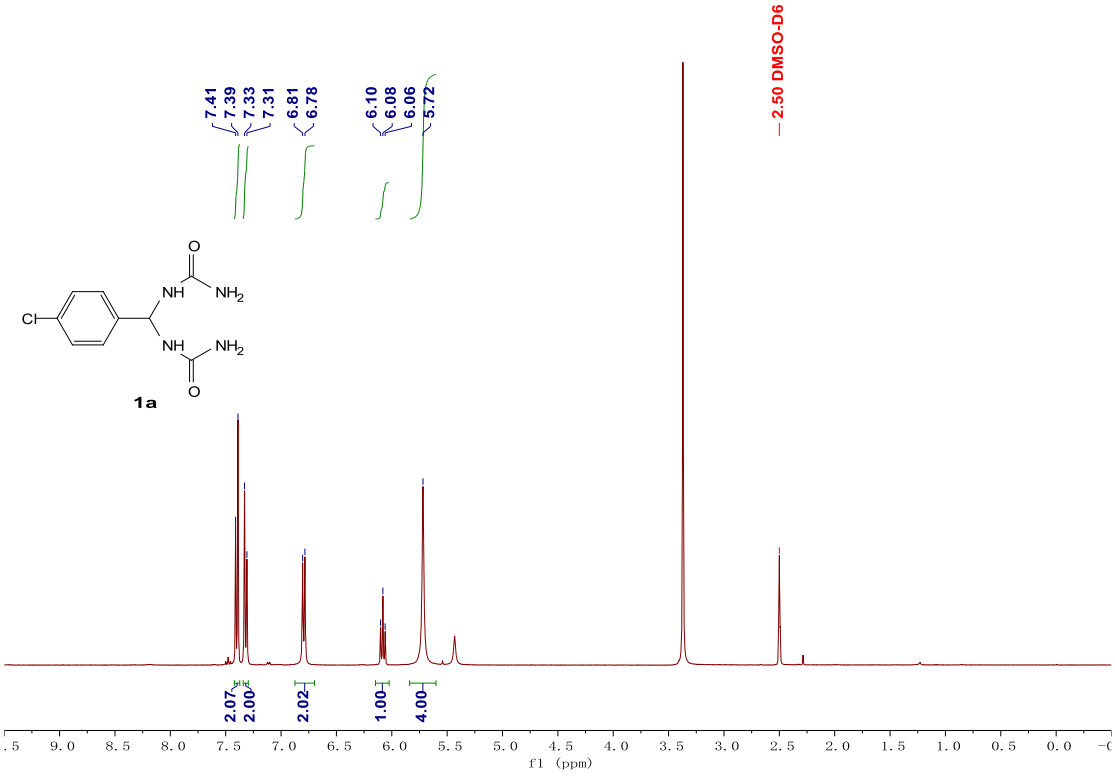


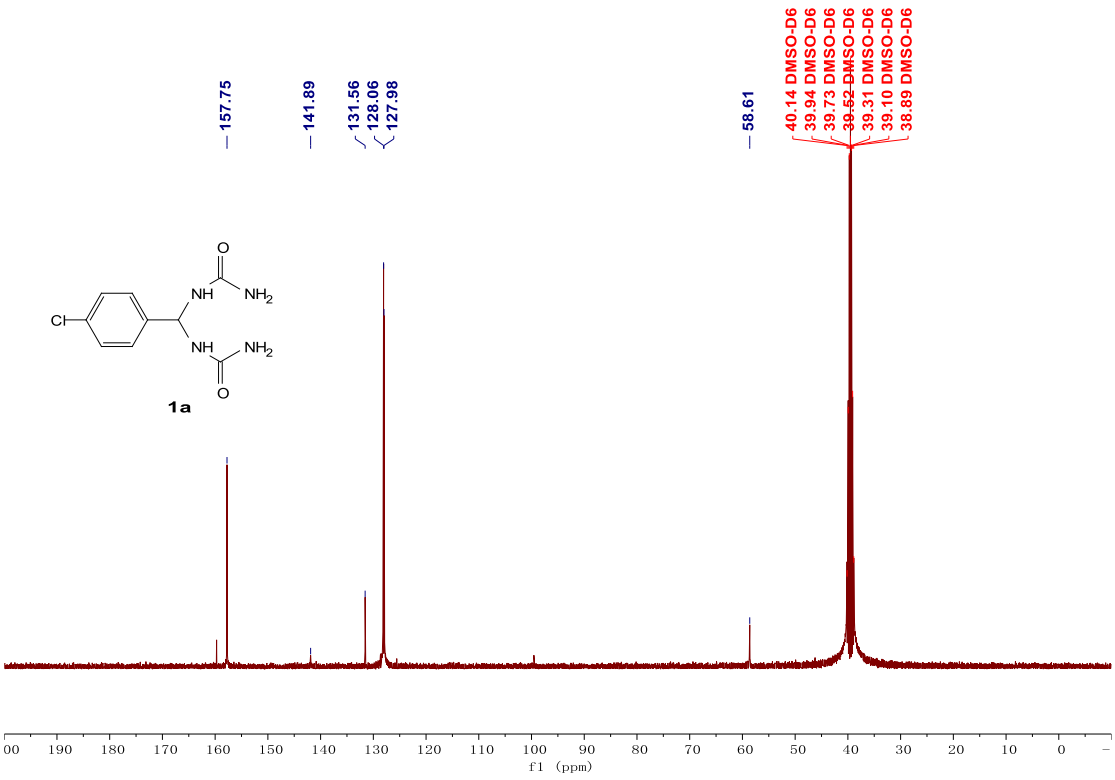
Ethyl 4-(4-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (4j).[2] 38.2 mg. Yield = 26%. White solid. **M.p.:** 212.3-213.6 °C. **^{1}H NMR (400 MHz, DMSO-*d*$_6$):** δ 9.25 (s, 1H), 7.78 (s, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 5.14 (d, *J* = 3.2 Hz, 1H), 3.98 (q, *J* = 7.0 Hz, 2H), 2.24 (s, 3H), 1.09 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (100 MHz, DMSO-*d*$_6$):** δ 165.3, 152.0, 148.8, 143.8, 131.8, 128.5, 128.3, 98.9, 59.3, 53.5, 17.9, 14.1 ppm. The characterization data of this product is in accordance with the reported ones.[2]

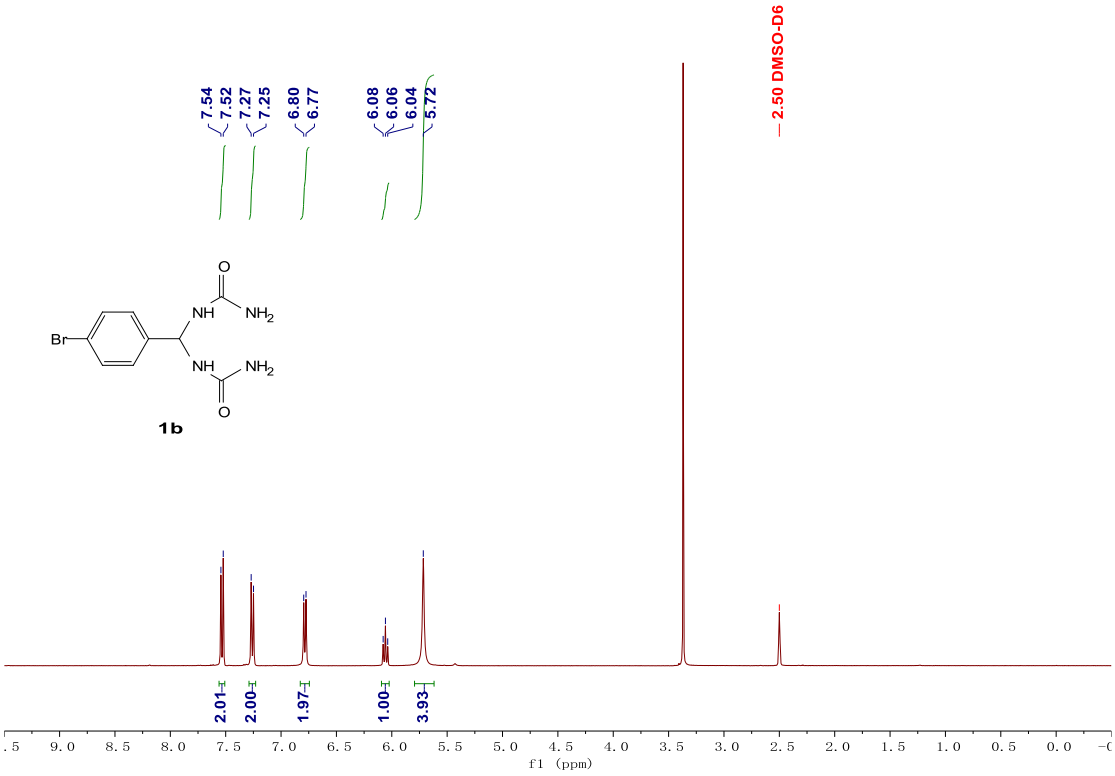
References

1. Kappe, C. O. *J. Org. Chem.* **1997**, *62*, 7201.
2. (a) Ma, J.; Zhong, L.; Peng, X.; Sun, R. *Green Chem.* **2016,** *18***,** 1738. (b) Pasunooti, K. K.; Chai, H.; Jensen, C. N.; Gorityala, B. K.; Wang, S.; Liu, X.-W. *Tetrahedron Lett.* **2011**, *52*, 80. (c) Li, N.; Wang, Y.; Liu, F.; Zhao, X.; Xu, X.; An, Q.; Yun, K. *Appl. Organomet. Chem*. **2020**, *34*, e5454. (d) Bentahar, S.; Ait Taleb, M.; Sabour, A.; Dbik, A.; El Khomri, M.; El Messaoudi, N.; Lacherai, A.; Mamouni, R. *Russ. J. Org. Chem.* **2019**, *55*, 1423.

^{1}H, ^{13}C, and ^{19}F NMR spectra of products



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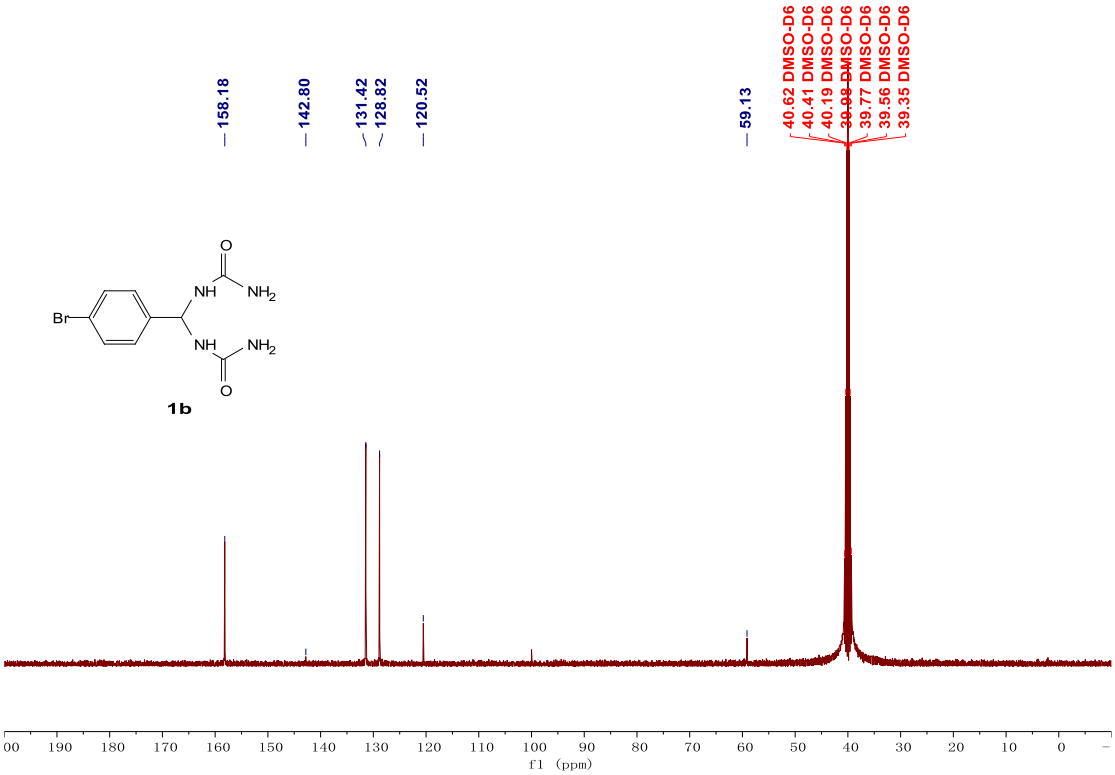
7.52
7.27
7.25
6.80
6.77
6.08
6.06
6.04
5.72
2.50 DMSO-D6
Br
NH
NH2
NH
NH2
O
O
1b
2.01
2.00
1.97
1.00
3.93
f1 (ppm)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142.80
131.42
128.82
120.52
59.13
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40.19 DMSO-D6
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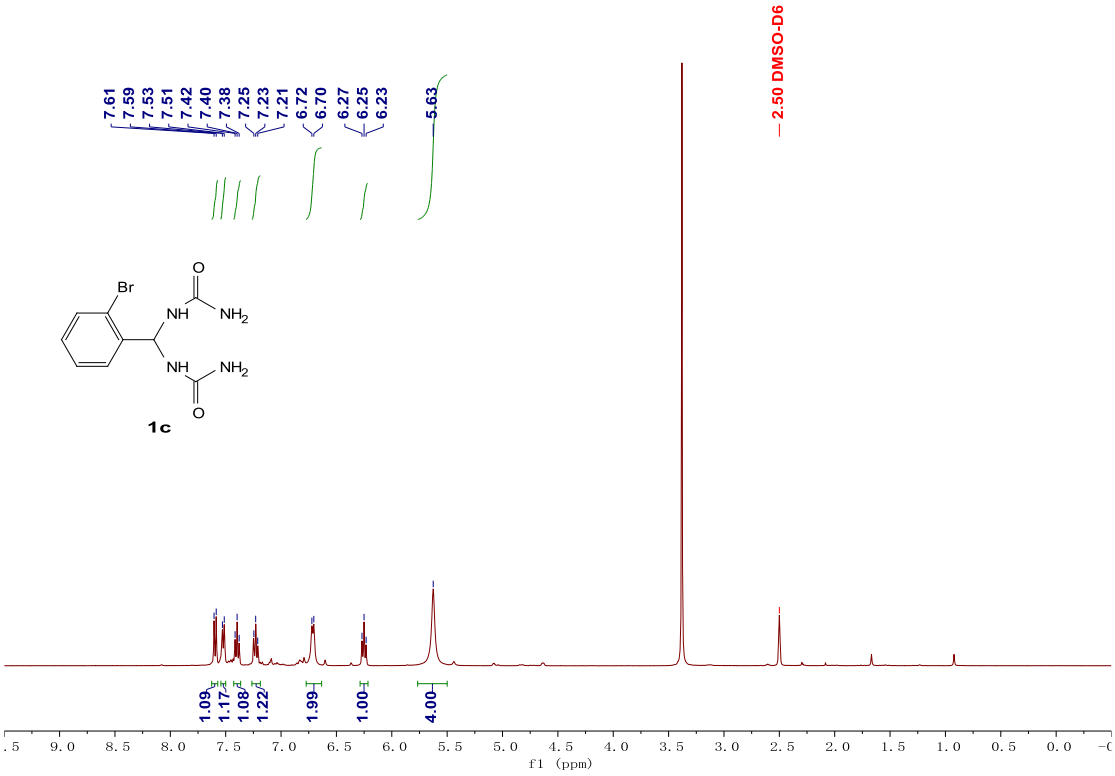
NH2
NH
NH2
O
O
1b
f1 (ppm)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7.59
7.53
7.51
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7.40
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7.25
7.23
7.21
6.72
6.70
6.27
6.25
6.23
5.63
2.50 DMSO-D6
Br
NH
NH2
NH
NH2
O
O
1c
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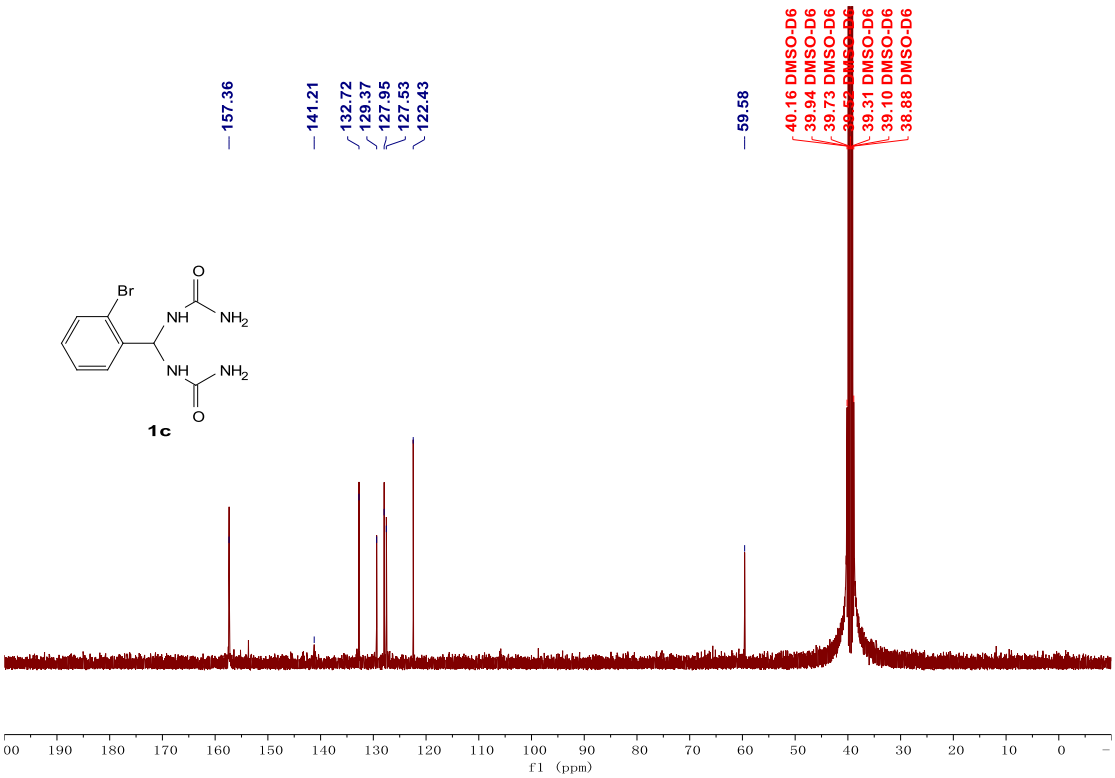
1.17
1.08
1.22
1.99
1.00
4.00
f1 (ppm)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132.72
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127.95
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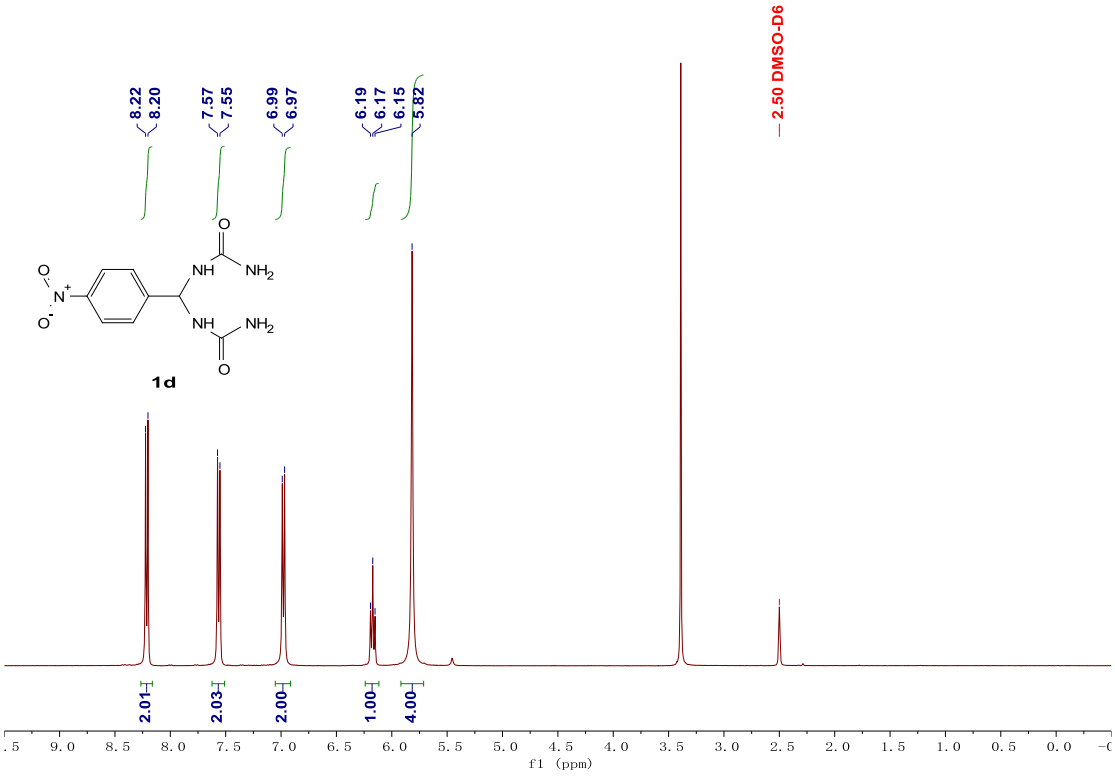
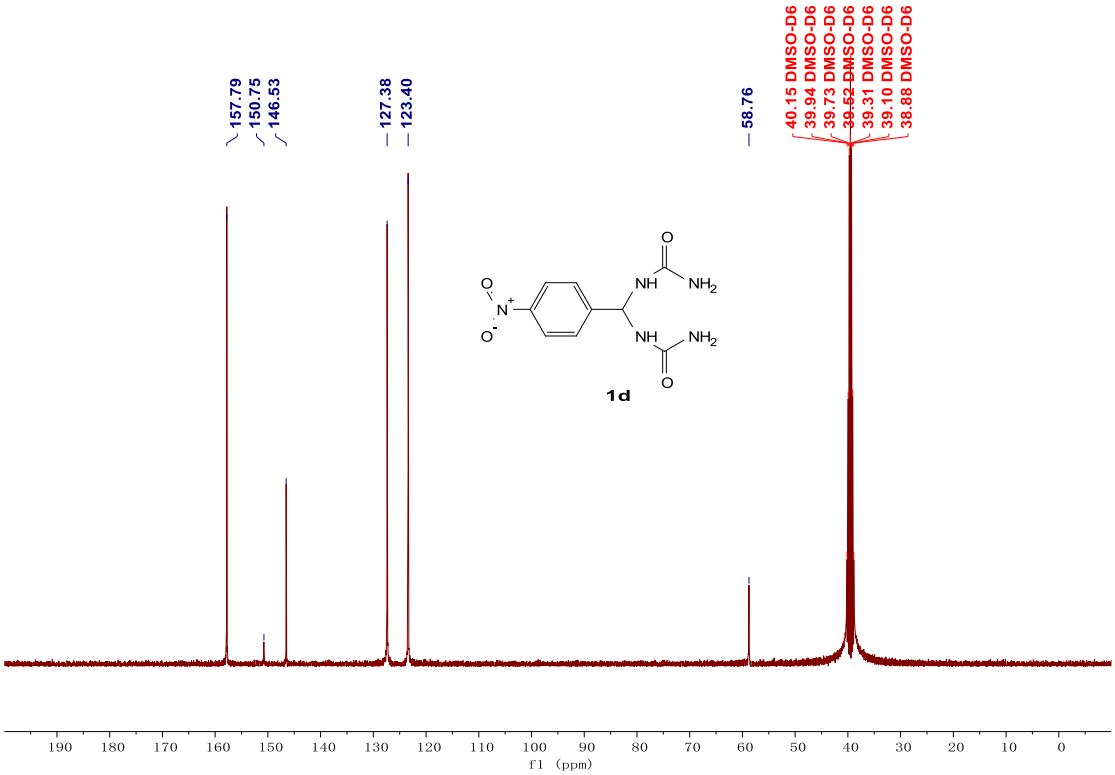
122.43
59.58
40.16 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.88 DMSO-D6
Br
NH
NH2
NH
NH2
O
O
1c
f1 (ppm)


8.22
8.20
7.57
7.55
6.99
6.97
6.19
6.17
6.15
5.82
2.50 DMSO-D6
1d
2.01
2.03
2.00
1.00
4.00
f1 (ppm)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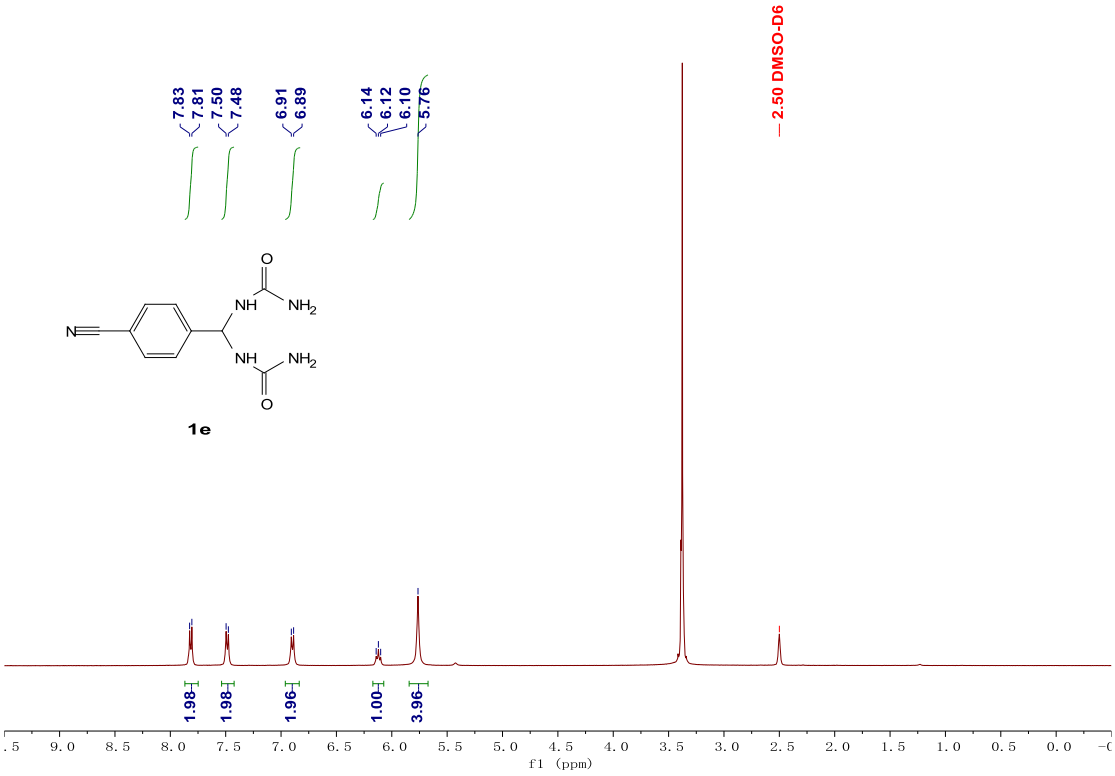
150.75
146.53
127.38
123.40
58.76
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.88 DMSO-D6
1d
f1 (ppm)


7.83
7.81
7.50
7.48
6.91
6.89
6.14
6.12
6.10
5.76
2.50 DMSO-D6
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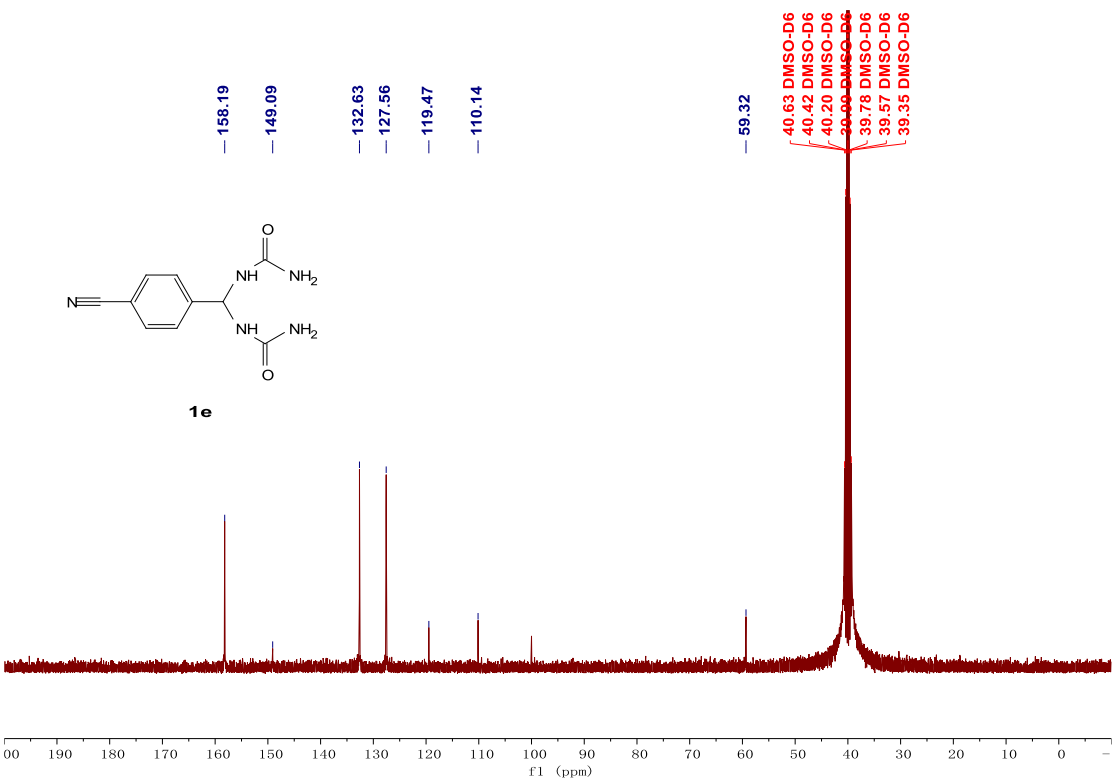
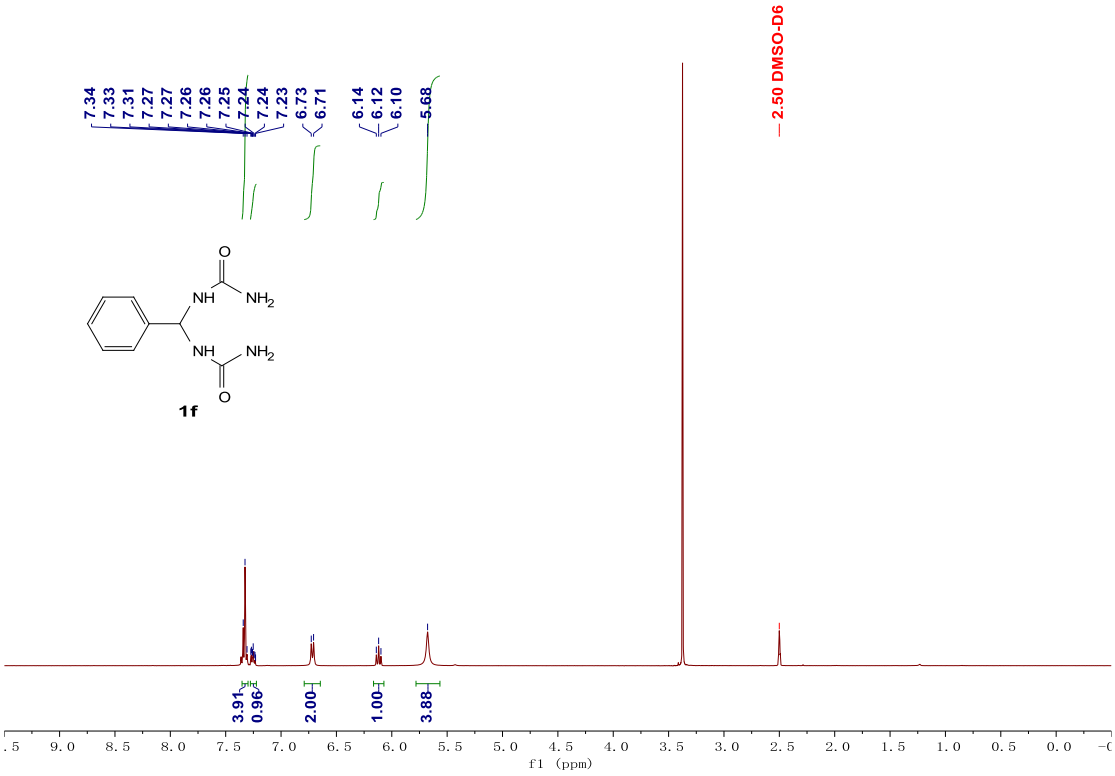
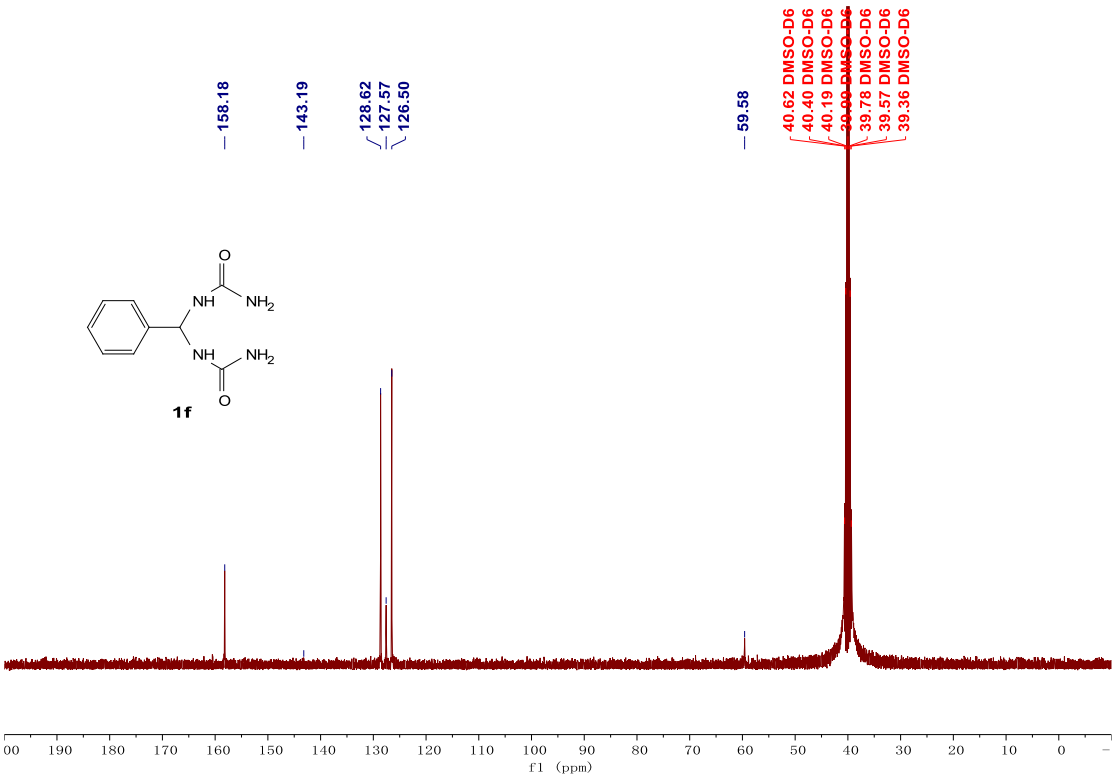
1.96
1.00
3.96
f1 (ppm)

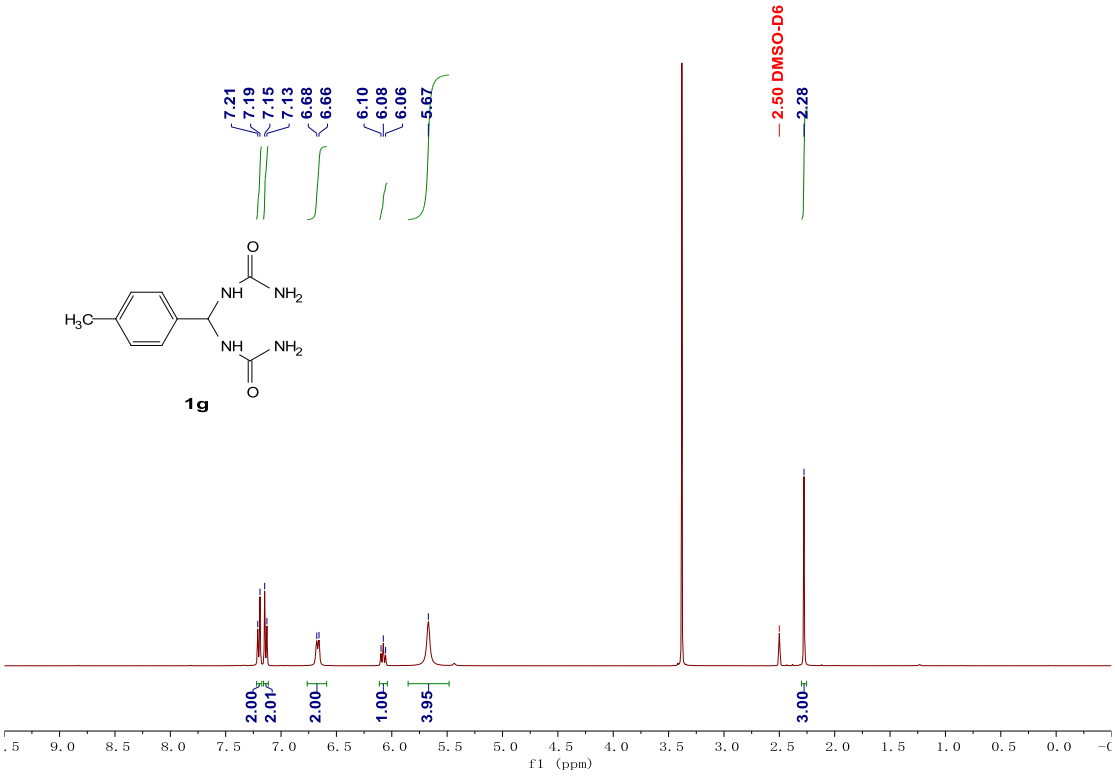
1e

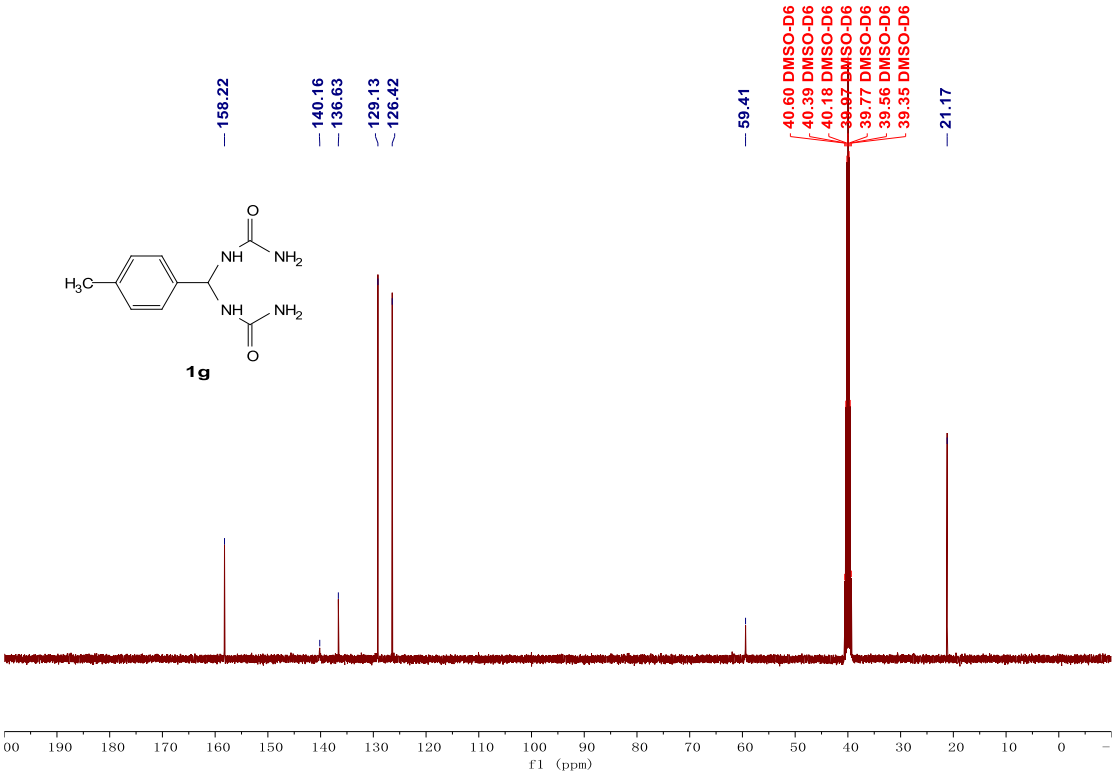

158.19
149.09
132.63
127.56
119.47
110.14
59.32
40.63 DMSO-D6
40.42 DMSO-D6
40.20 DMSO-D6
39.78 DMSO-D6
39.57 DMSO-D6
39.35 DMSO-D6
f1 (ppm)

1e


7.34
7.33
7.31
7.27
7.27
7.26
7.26
7.25
7.24
7.24
7.23
6.73
6.71
6.14
6.12
6.10
5.68
2.50 DMSO-D6
NH
NH2
NH
NH2
O
O
1f
3.91
0.96
2.00
1.00
3.88
f1 (ppm)


158.18
143.19
128.62
127.57
126.50
59.58
40.62 DMSO-D6
40.40 DMSO-D6
40.19 DMSO-D6
39.78 DMSO-D6
39.57 DMSO-D6
39.36 DMSO-D6
NH
NH2
NH
NH2
O
O
1f
f1 (ppm)


7.21
7.19
7.15
7.13
6.68
6.66
6.10
6.08
6.06
5.67
2.50 DMSO-D6
2.28
NH
NH2
H3C
NH
NH2
O
O
1g
2.00
2.01
2.00
1.00
3.95
3.00
f1 (ppm)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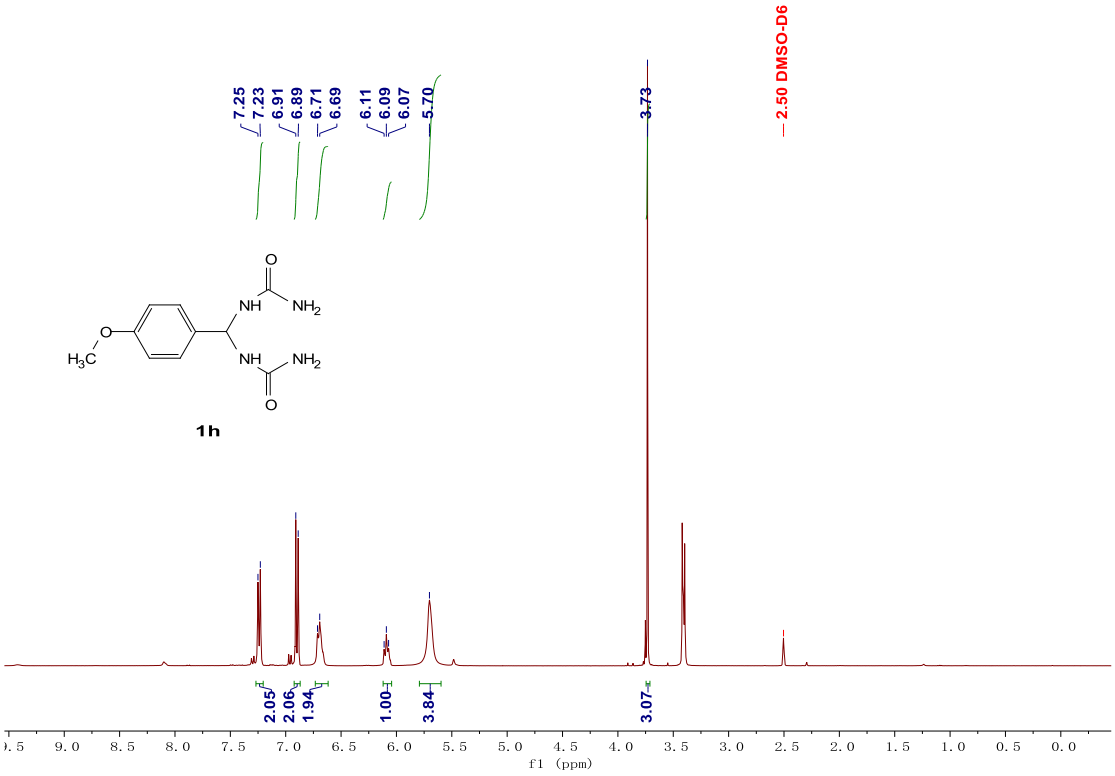
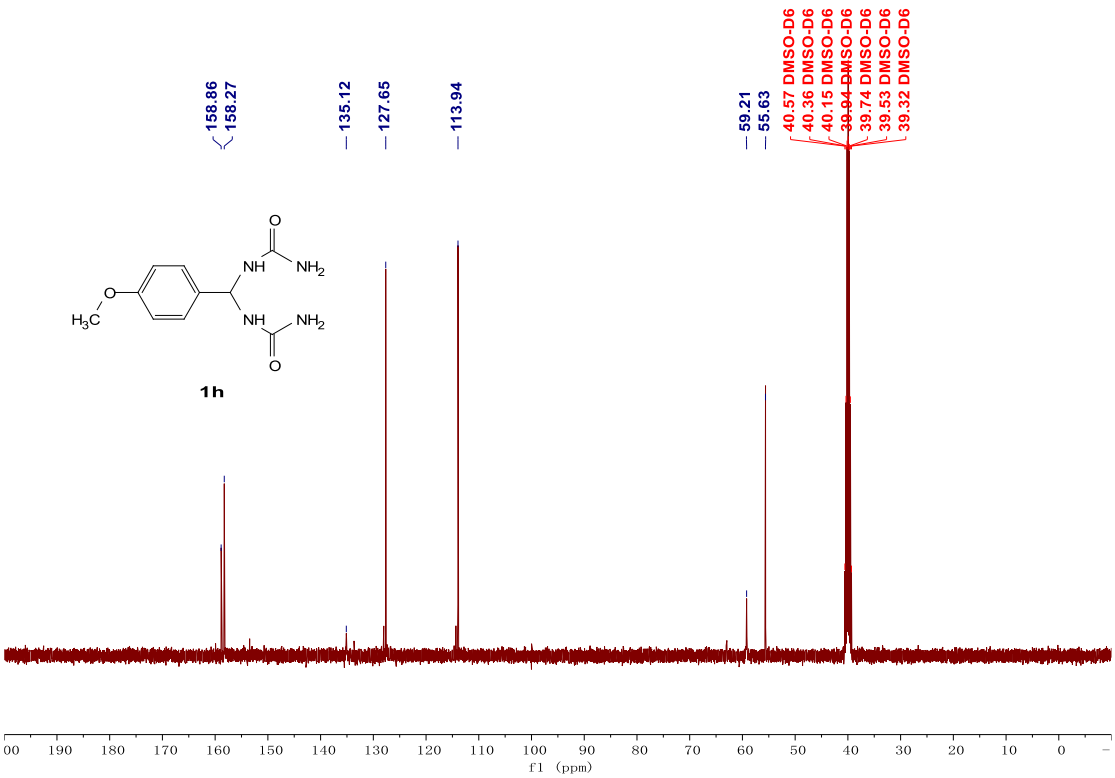
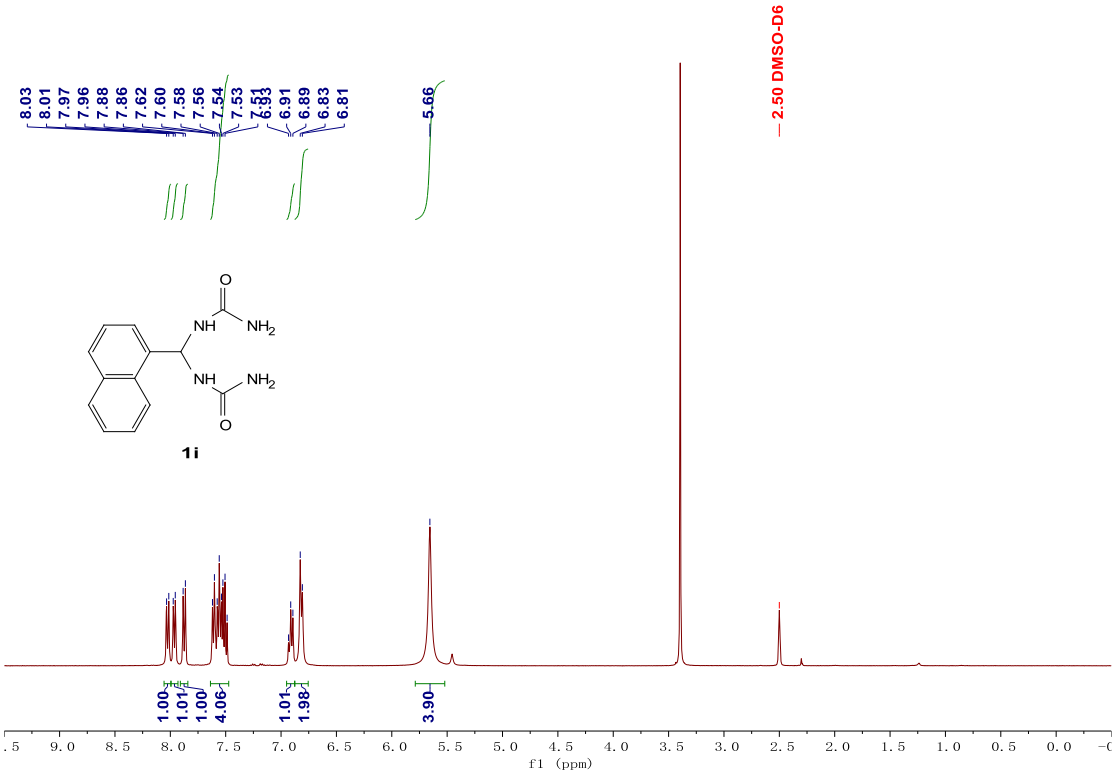
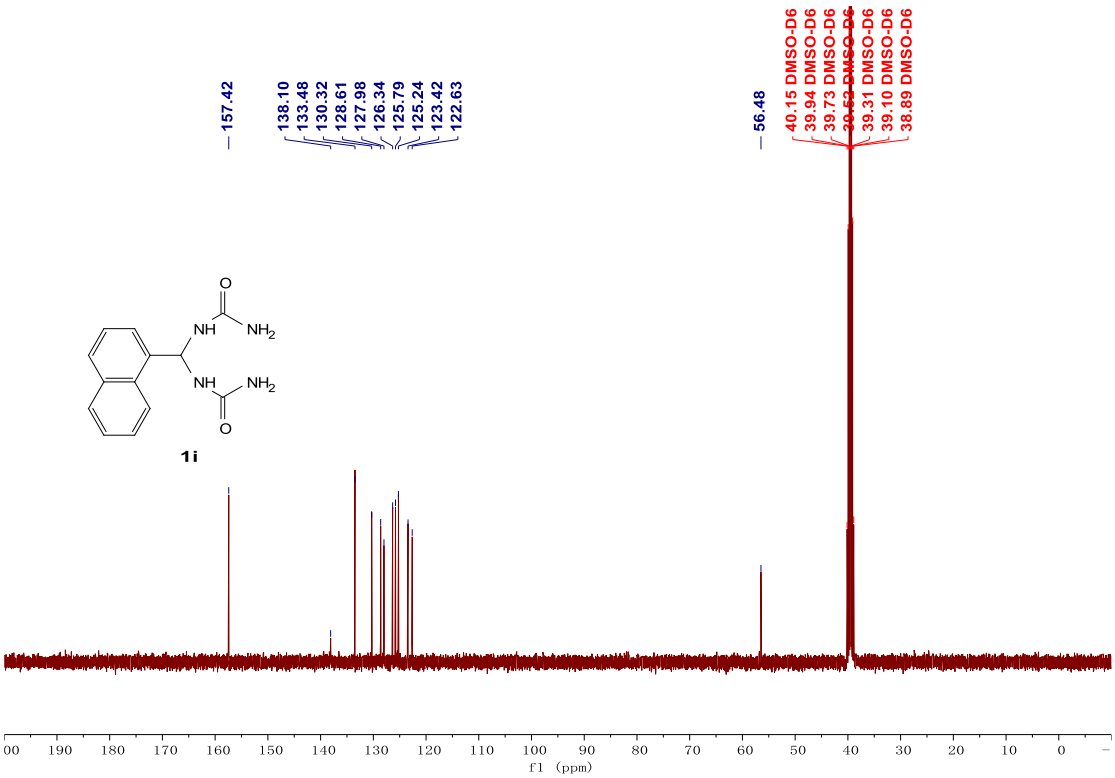
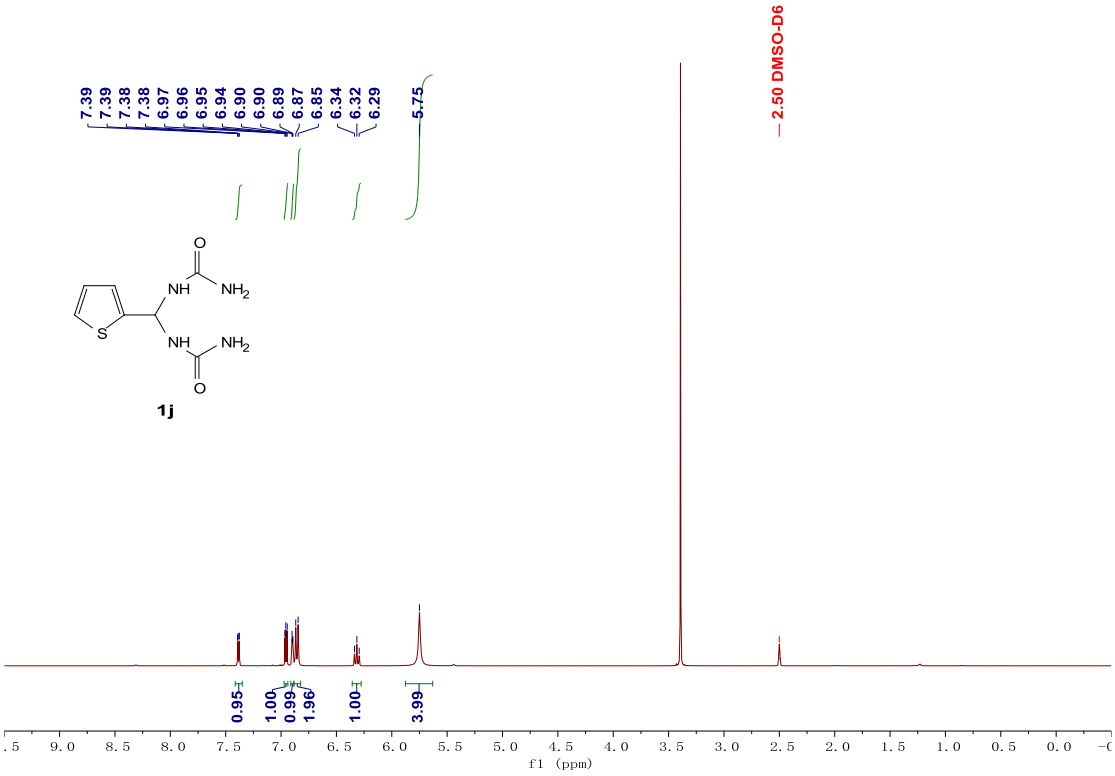
140.16
136.63
129.13
126.42
59.41
40.60 DMSO-D6
40.39 DMSO-D6
40.18 DMSO-D6
39.97 DMSO-D6
39.77 DMSO-D6
39.56 DMSO-D6
39.35 DMSO-D6
21.17
NH
NH2
H3C
NH
NH2
O
O
1g
f1 (ppm)

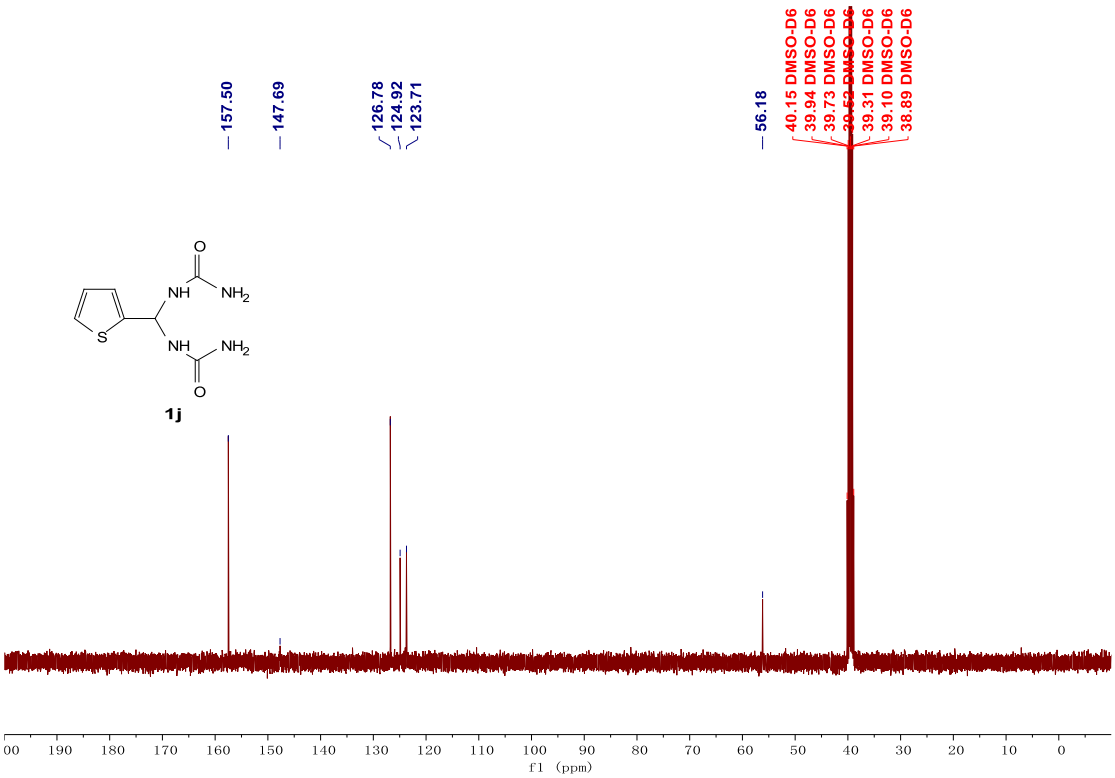

7.25
7.23
6.91
6.89
6.71
6.69
6.11
6.09
6.07
5.70
3.73
2.50 DMSO-D6
NH
NH2
NH
NH2
O
O
H3C
1h
2.05
2.06
1.94
1.00
3.84
3.07
f1 (ppm)


158.86
158.27
135.12
127.65
113.94
59.21
55.63
40.57 DMSO-D6
40.36 DMSO-D6
40.15 DMSO-D6
39.94 DMSO-D6
39.74 DMSO-D6
39.53 DMSO-D6
39.32 DMSO-D6
NH
NH2
NH
NH2
O
O
H3C
1h
f1 (ppm)


8.03
8.01
7.97
7.96
7.88
7.86
7.62
7.60
7.58
7.56
7.54
7.53
6.91
6.89
6.83
6.81
5.66
2.50 DMSO-D6
NH
NH2
NH
NH2
O
O
1i
1.00
1.01
1.00
4.06
1.01
1.98
3.90
f1 (ppm)


157.42
138.10
133.48
130.32
128.61
127.98
126.34
125.79
125.24
123.42
122.63
56.48
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
NH
NH2
NH
NH2
O
O
1i
f1 (ppm)


7.39
7.39
7.38
7.38
6.97
6.96
6.95
6.94
6.90
6.90
6.89
6.87
6.85
6.34
6.32
6.29
5.75
2.50 DMSO-D6
NH
NH2
S
NH
NH2
O
O
1j
0.95
1.00
0.99
1.96
1.00
3.99
f1 (ppm)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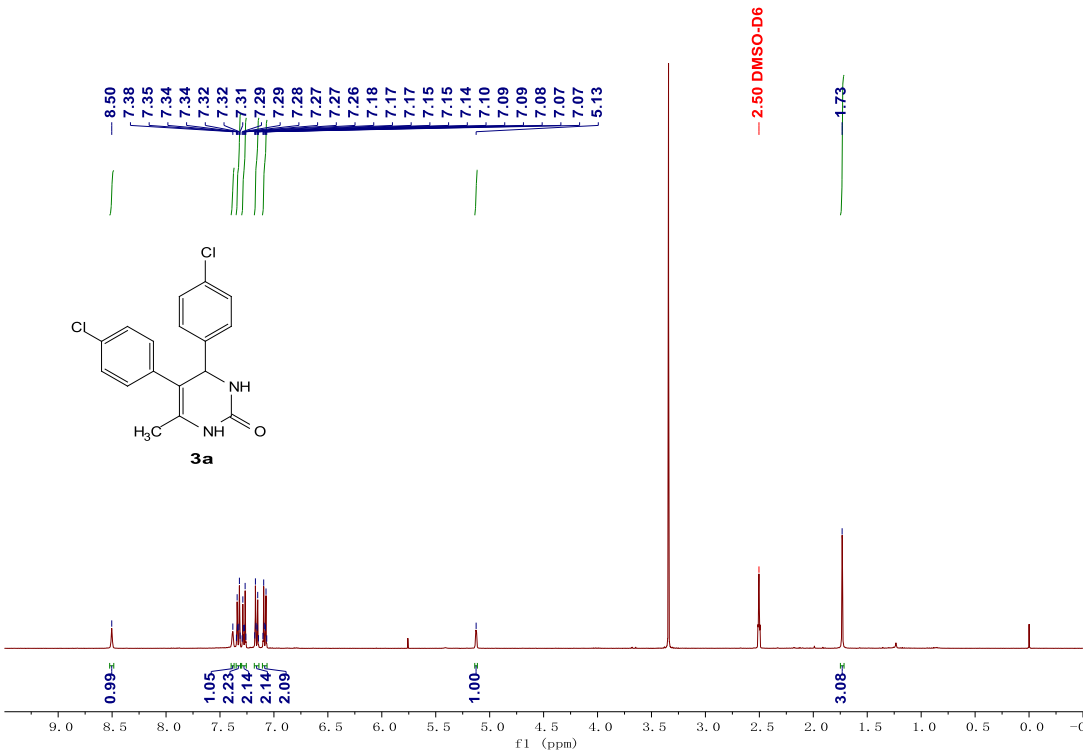
147.69
126.78
124.92
123.71
56.18
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
NH
NH2
S
NH
NH2
O
O
1j
f1 (ppm)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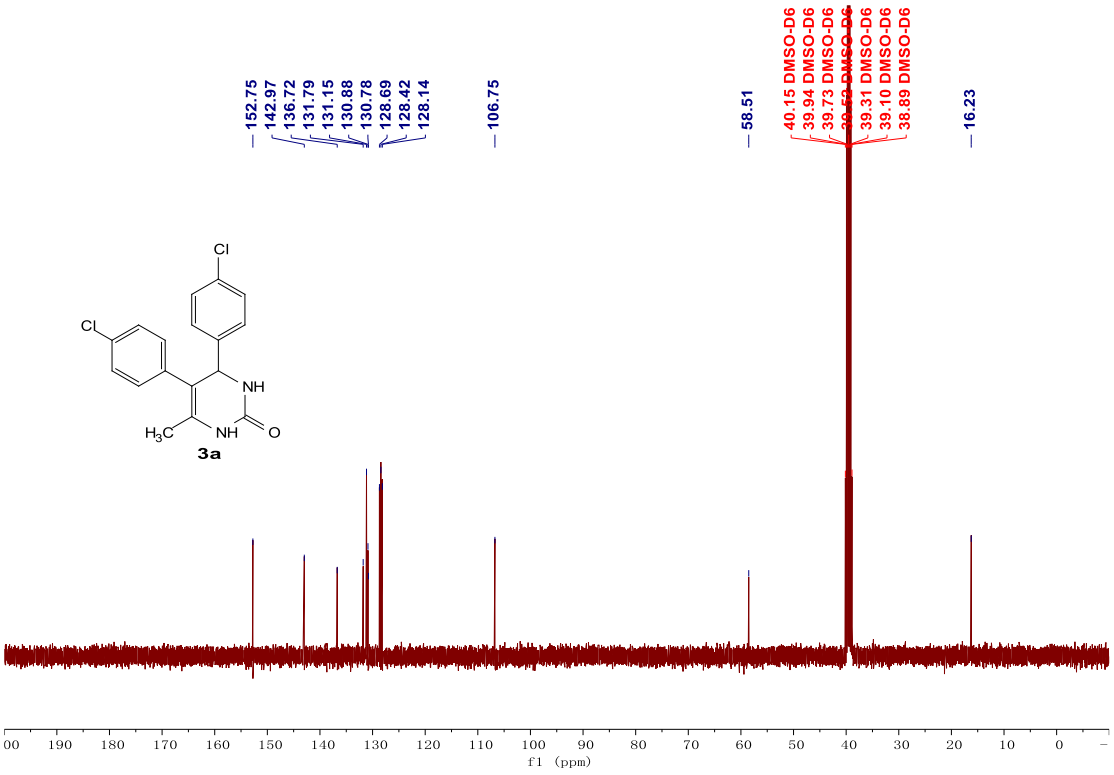
7.38
7.35
7.34
7.34
7.32
7.32
7.31
7.29
7.29
7.28
7.27
7.27
7.26
7.18
7.17
7.17
7.15
7.15
7.14
7.10
7.09
7.09
7.08
7.07
7.07
5.13
2.50 DMSO-D6
1.73
Cl
Cl
NH
H3C
NH
O
3a
0.99
1.05
2.23
2.14
2.14
2.09
1.00
3.08
f1 (ppm)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142.97
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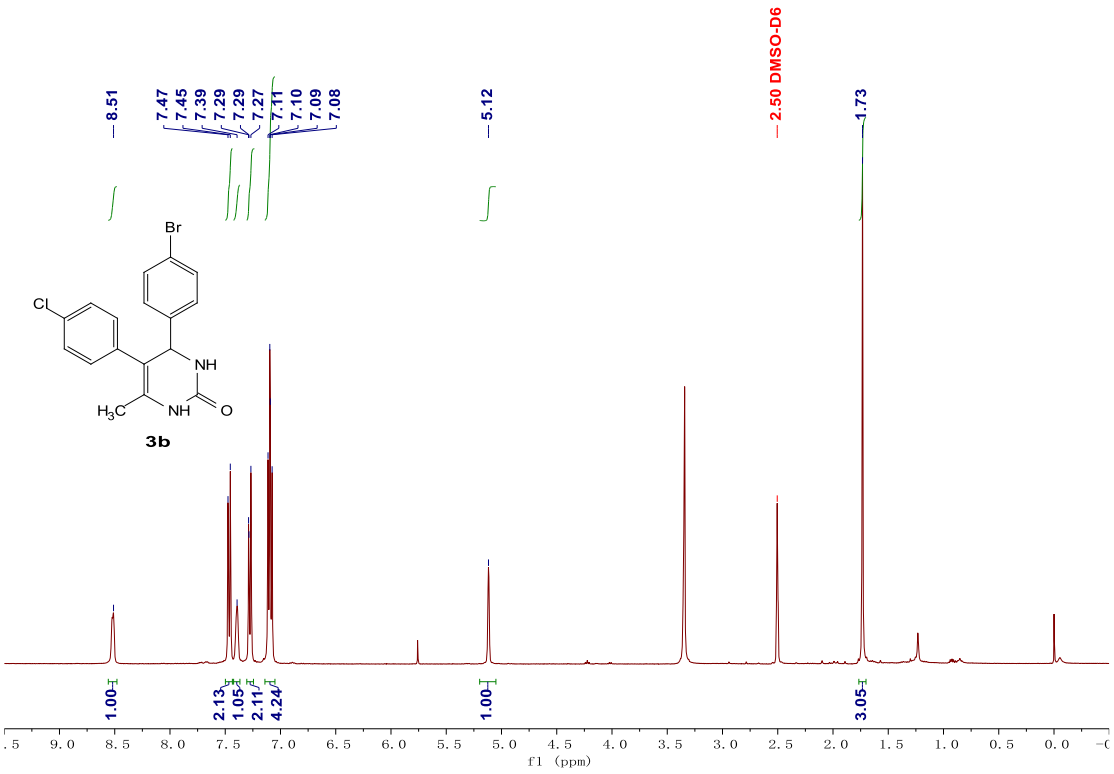
131.79
131.15
130.88
130.78
128.69
128.42
128.14
106.75
58.51
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.23
Cl
Cl
NH
H3C
NH
O
3a
f1 (ppm)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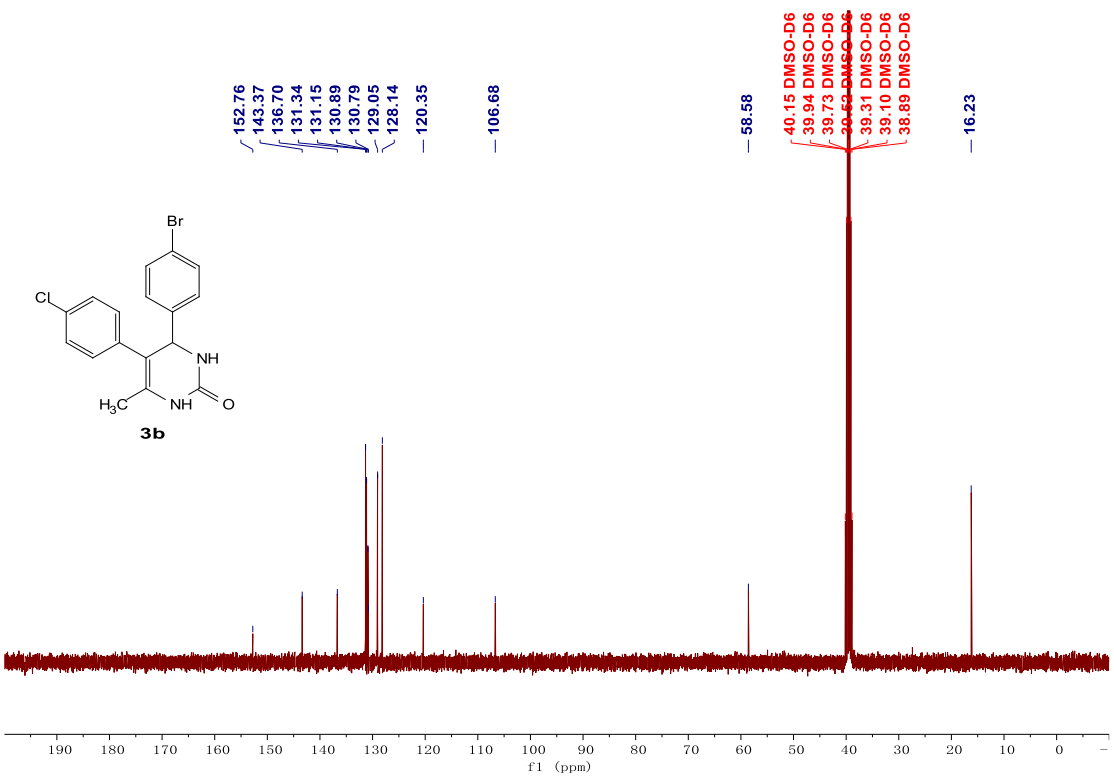
7.47
7.45
7.39
7.29
7.29
7.27
7.11
7.10
7.09
7.08
5.12
2.50 DMSO-D6
1.73
Br
Cl
NH
H3C
NH
O
3b
1.00
2.13
1.05
2.11
4.24
1.00
3.05
f1 (ppm)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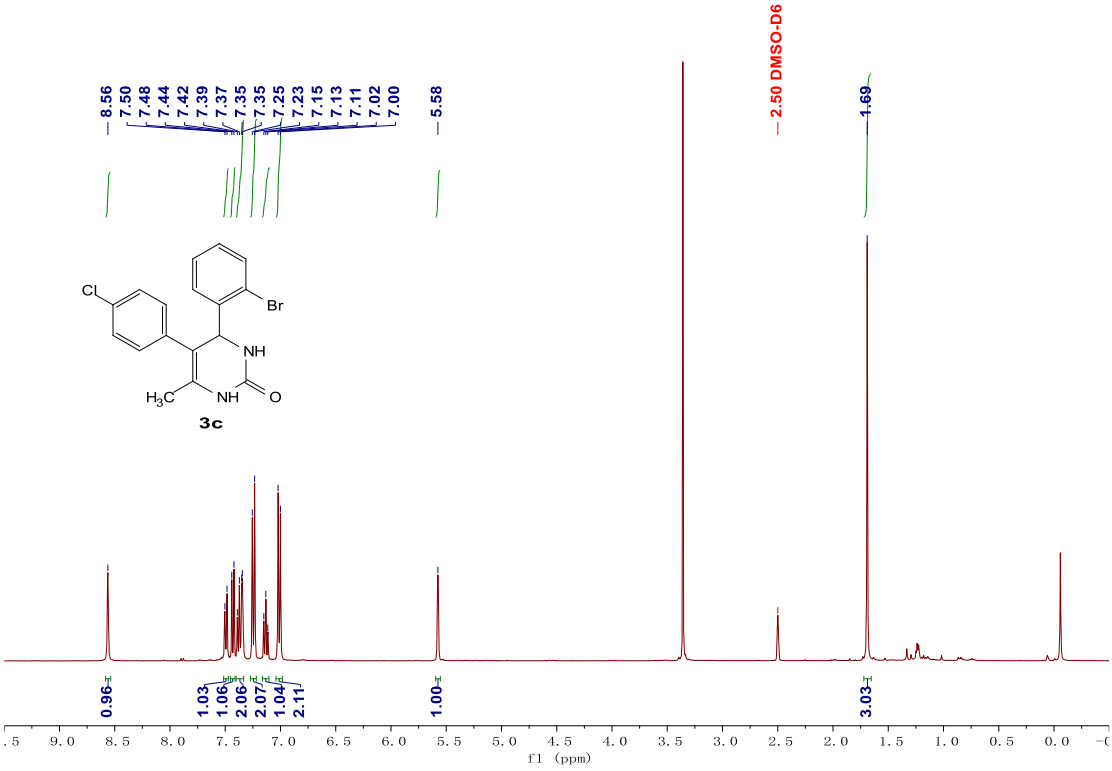
131.15
130.89
130.79
129.05
128.14
120.35
106.68
58.58
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.23
Br
Cl
NH
H3C
NH
O
3b
f1 (ppm)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7.48
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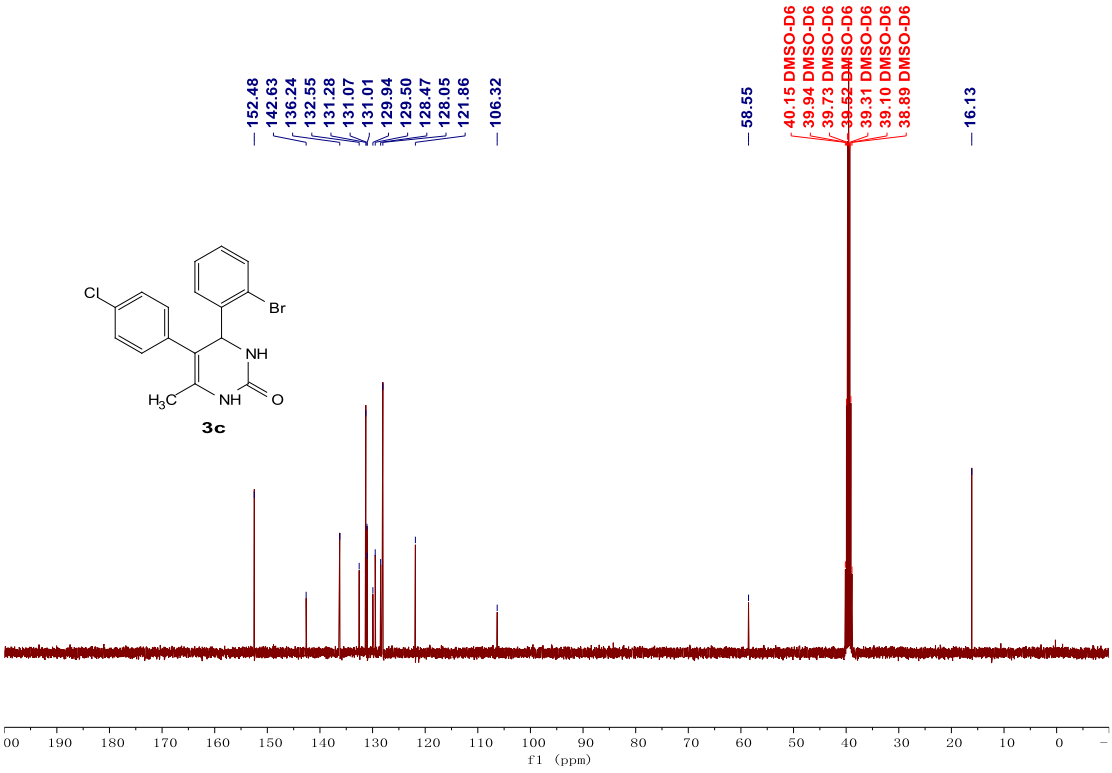
7.42
7.39
7.37
7.35
7.35
7.25
7.23
7.15
7.13
7.11
7.02
7.00
5.58
2.50 DMSO-D6
1.69
Cl
Br
NH
H3C
NH
O
3c
0.96
1.03
1.06
2.06
2.07
1.04
2.11
1.00
3.03
f1 (ppm)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128.47
128.05
121.86
106.32
58.55
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
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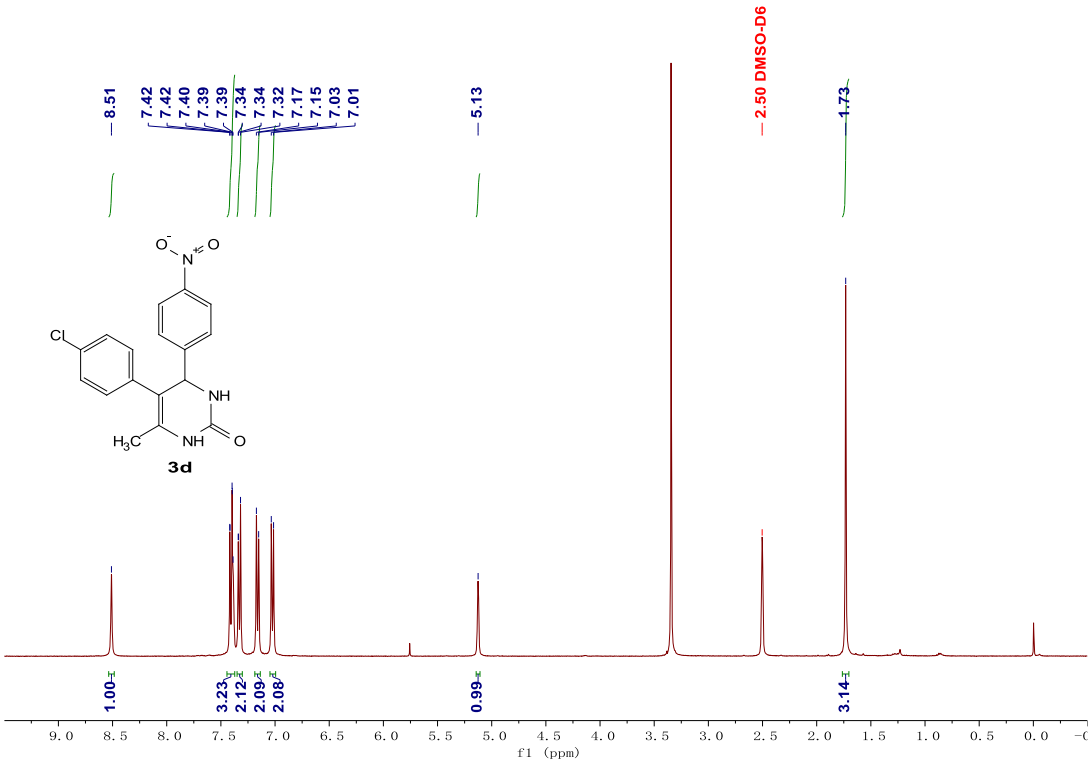
38.89 DMSO-D6
16.13
Cl
Br
NH
H3C
NH
O
3c
f1 (ppm)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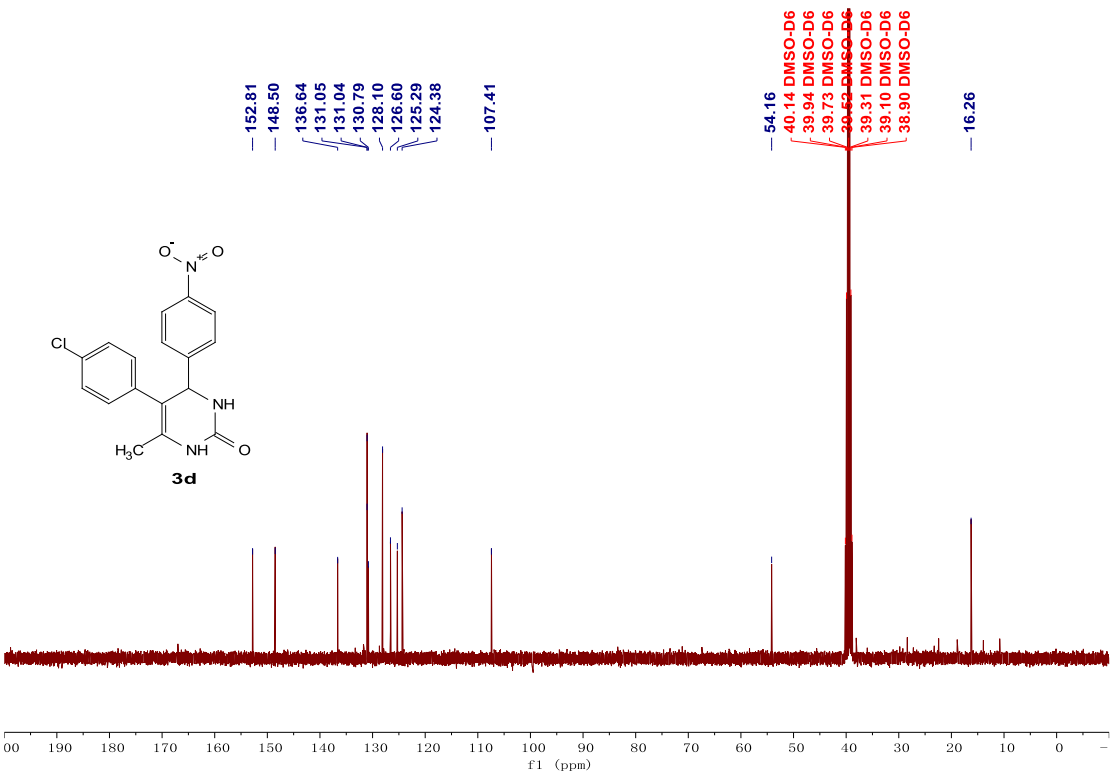
7.42
7.42
7.40
7.39
7.39
7.34
7.34
7.32
7.17
7.15
7.03
7.01
5.13
2.50 DMSO-D6
1.73
Cl
NH
NH
O
H_3C
3d
1.00
3.23
2.12
2.09
2.08
0.99
3.14
f1 (ppm)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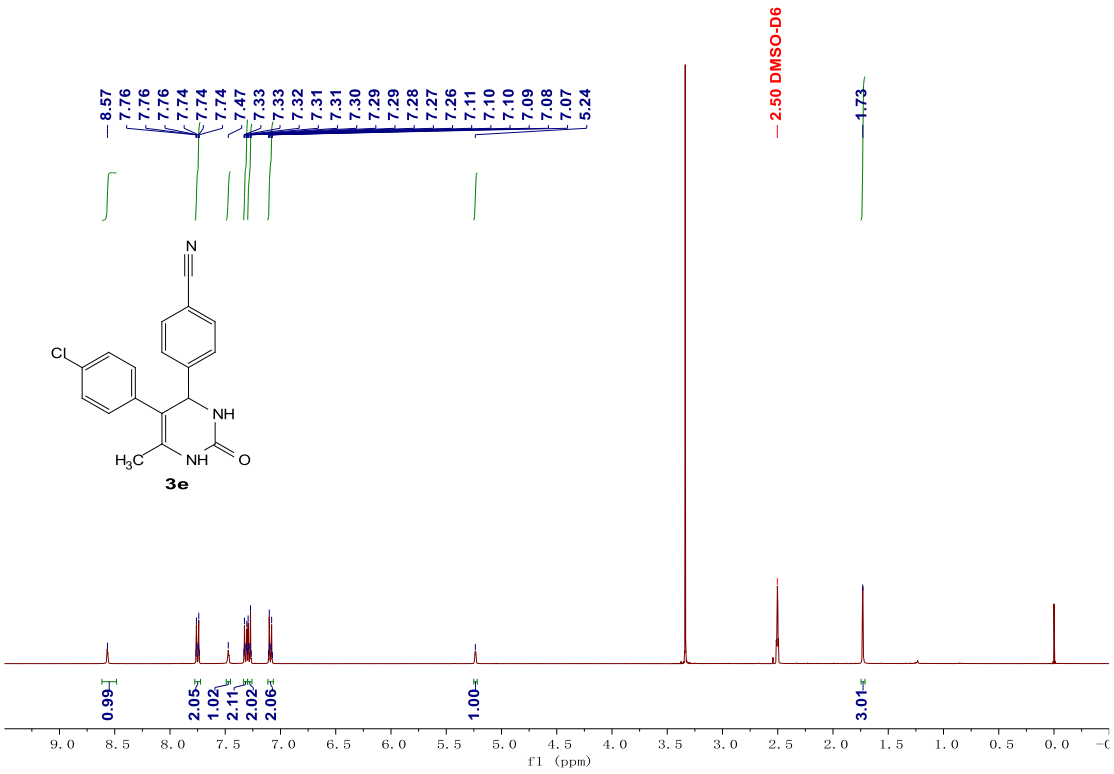
128.10
126.60
125.29
124.38
107.41
54.16
40.14 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.90 DMSO-D6
16.26
Cl
NH
NH
O
H_3C
3d
f1 (ppm)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7.74
7.74
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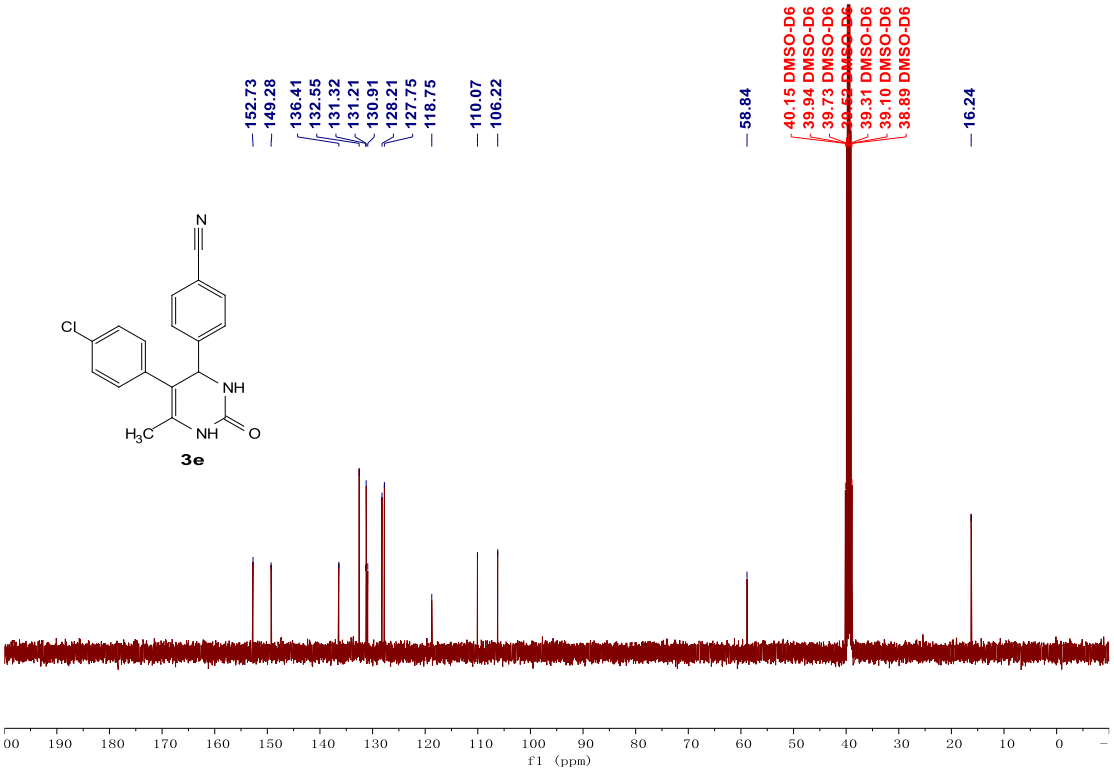
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.27
7.26
7.11
7.10
7.10
7.09
7.08
7.07
5.24
2.50 DMSO-D6
1.73
N
Cl
NH
H3C
NH
O
3e
0.99
2.05
1.02
2.11
2.02
2.06
1.00
3.01
f1 (ppm)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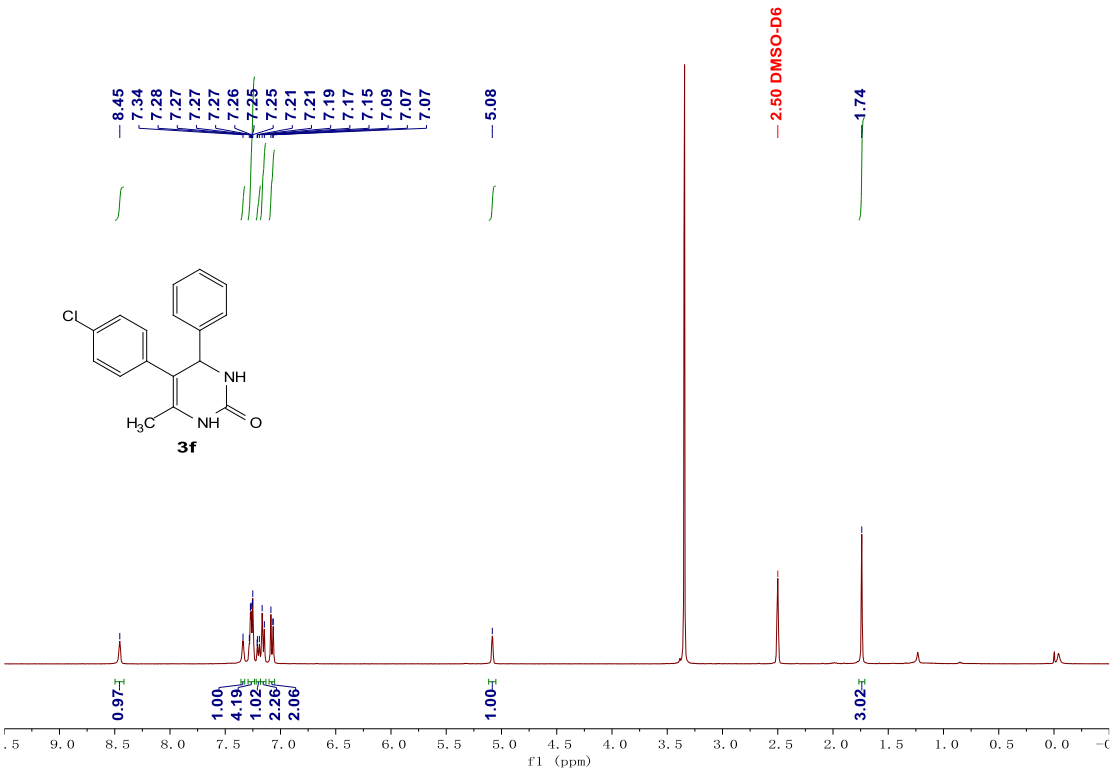
128.21
127.75
118.75
110.07
106.22
58.84
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.24
N
Cl
NH
H3C
NH
O
3e
f1 (ppm)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7.21
7.21
7.19
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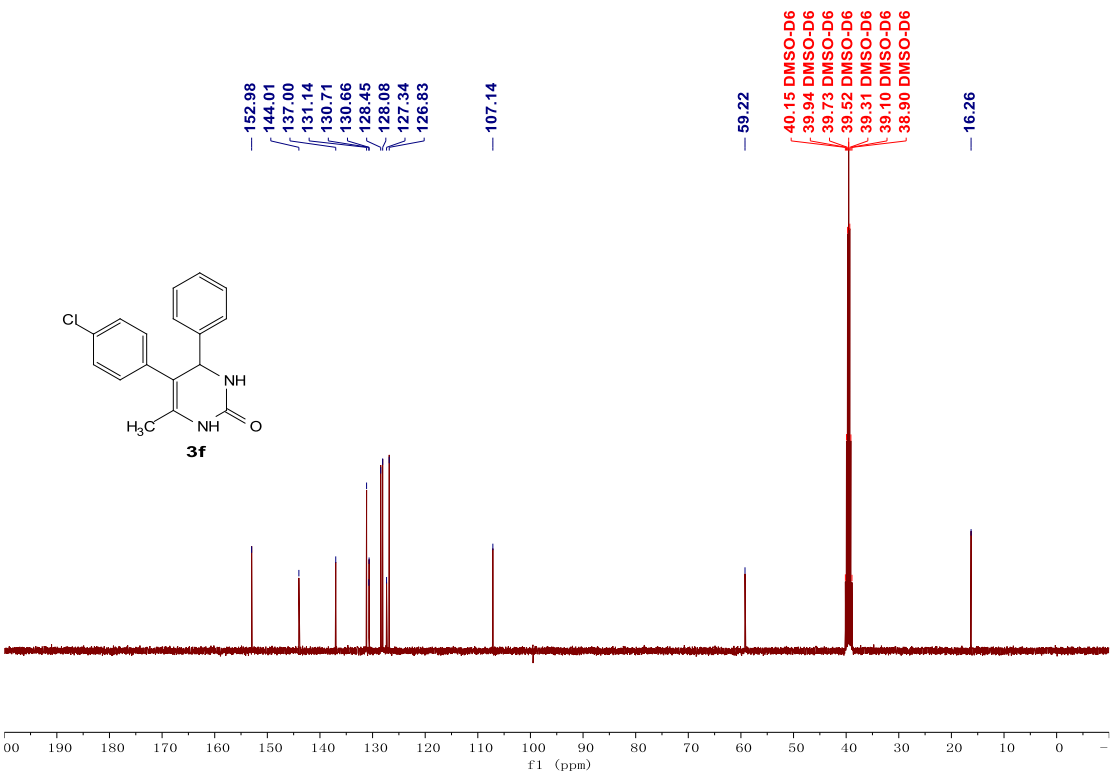
7.15
7.09
7.07
7.07
5.08
2.50 DMSO-D6
1.74
Cl
NH
H3C
NH
O
3f
0.97
1.00
4.19
1.02
2.26
2.06
1.00
3.02
f1 (ppm)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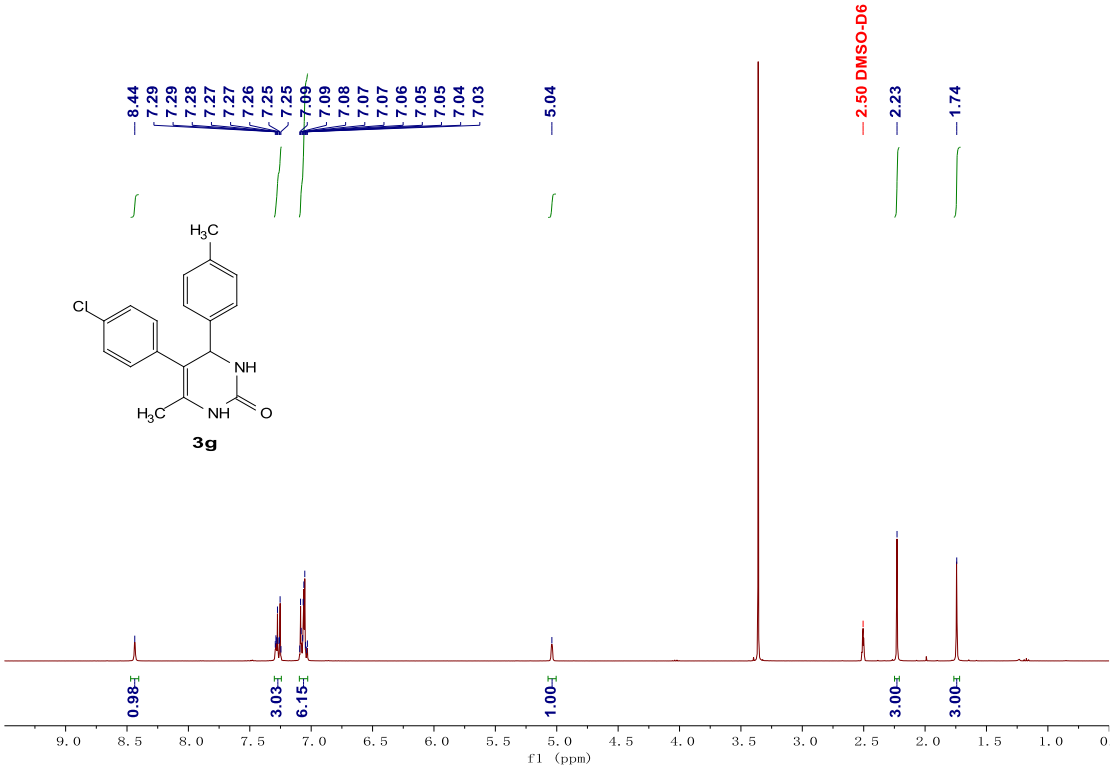
130.66
128.45
128.08
127.34
126.83
107.14
59.22
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.90 DMSO-D6
16.26
Cl
NH
H3C
NH
O
3f
f1 (ppm)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7.08
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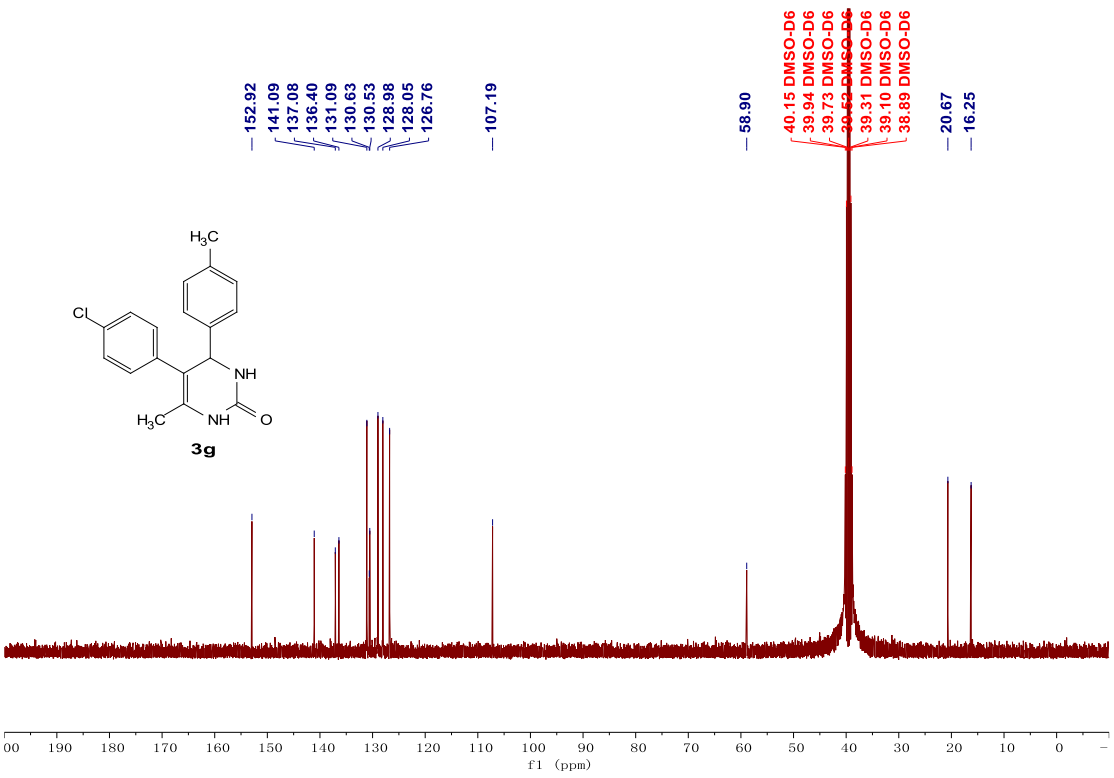
7.07
7.06
7.05
7.05
7.04
7.03
5.04
2.50 DMSO-D6
2.23
1.74
H3C
Cl
NH
H3C
NH
O
3g
0.98
3.03
6.15
1.00
3.00
3.00
f1 (ppm)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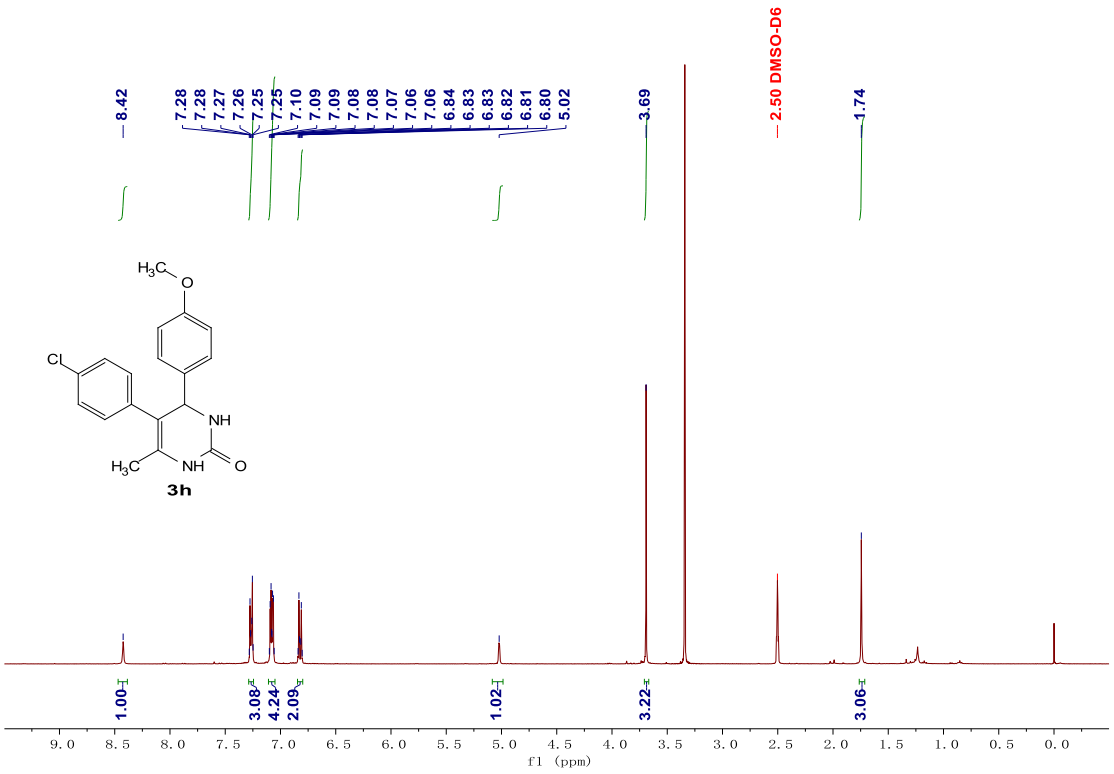
131.09
130.63
130.53
128.98
128.05
126.76
107.19
58.90
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
20.67
16.25
H3C
Cl
NH
H3C
NH
O
3g
f1 (ppm)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7.28
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7.08
7.08
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7.06
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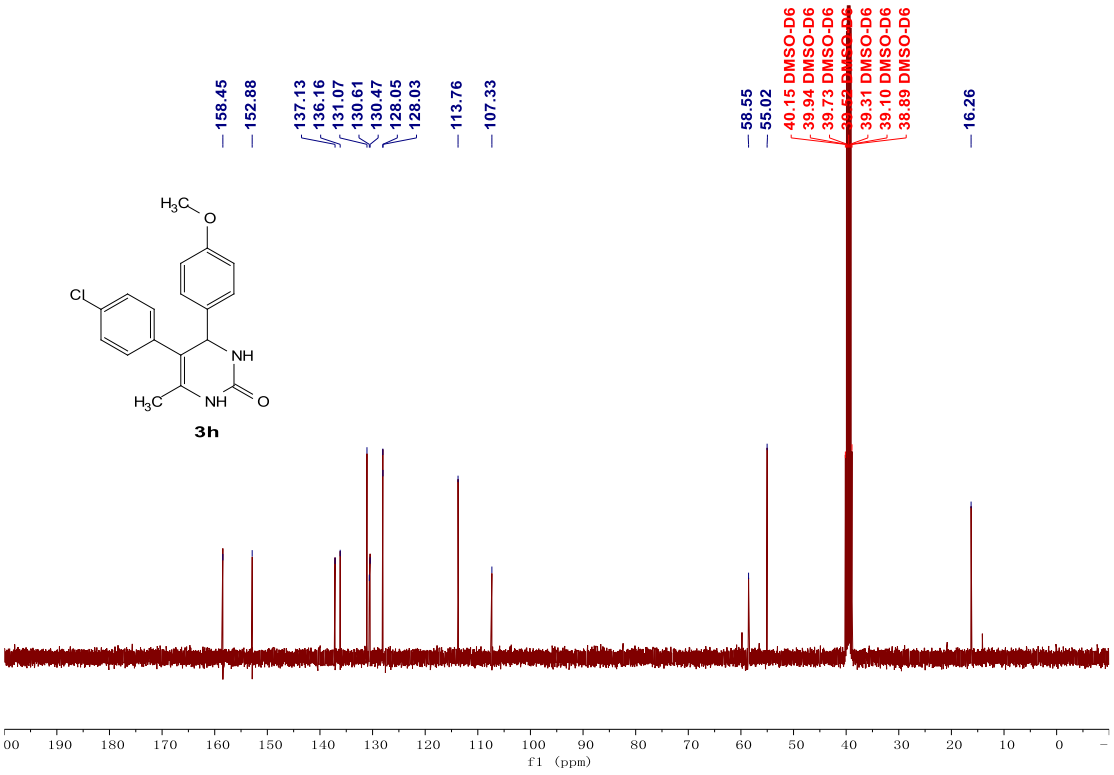
6.83
6.83
6.82
6.81
6.80
5.02
3.69
2.50 DMSO-D6
1.74
1.00
3.08
4.24
2.09
1.02
3.22
3.06
f1 (ppm)

3h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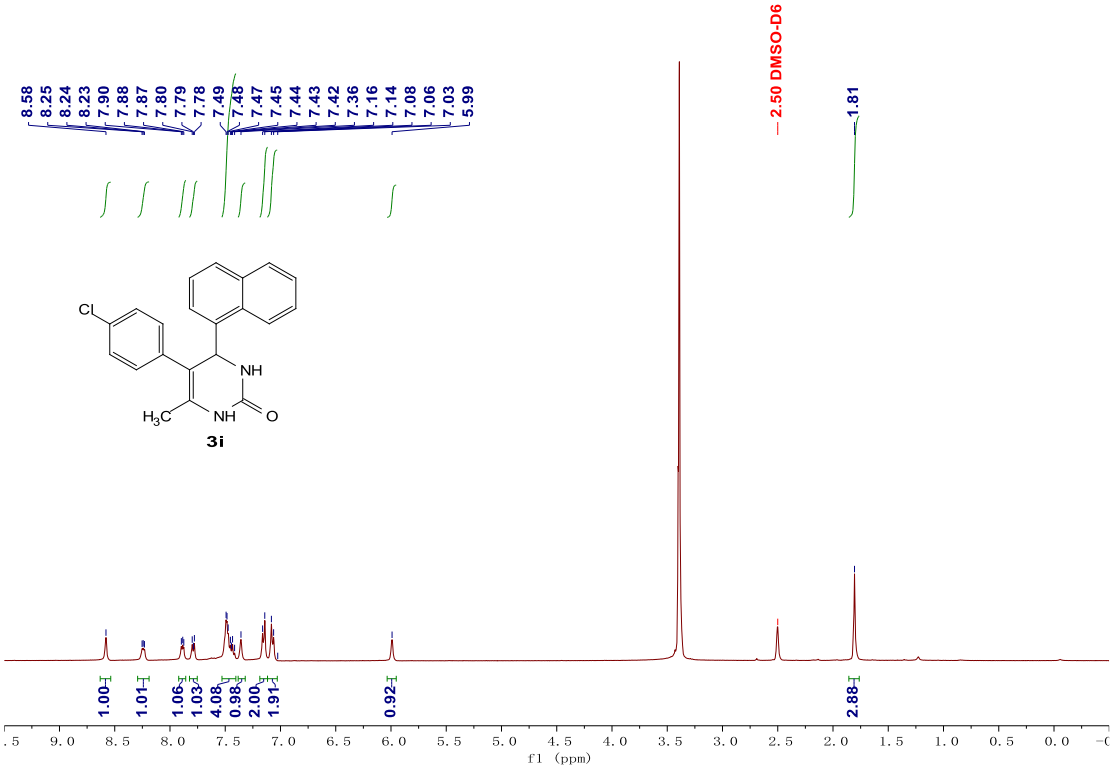
130.47
128.05
128.03
113.76
107.33
58.55
55.02
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.26
f1 (ppm)

3h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7.80
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7.78
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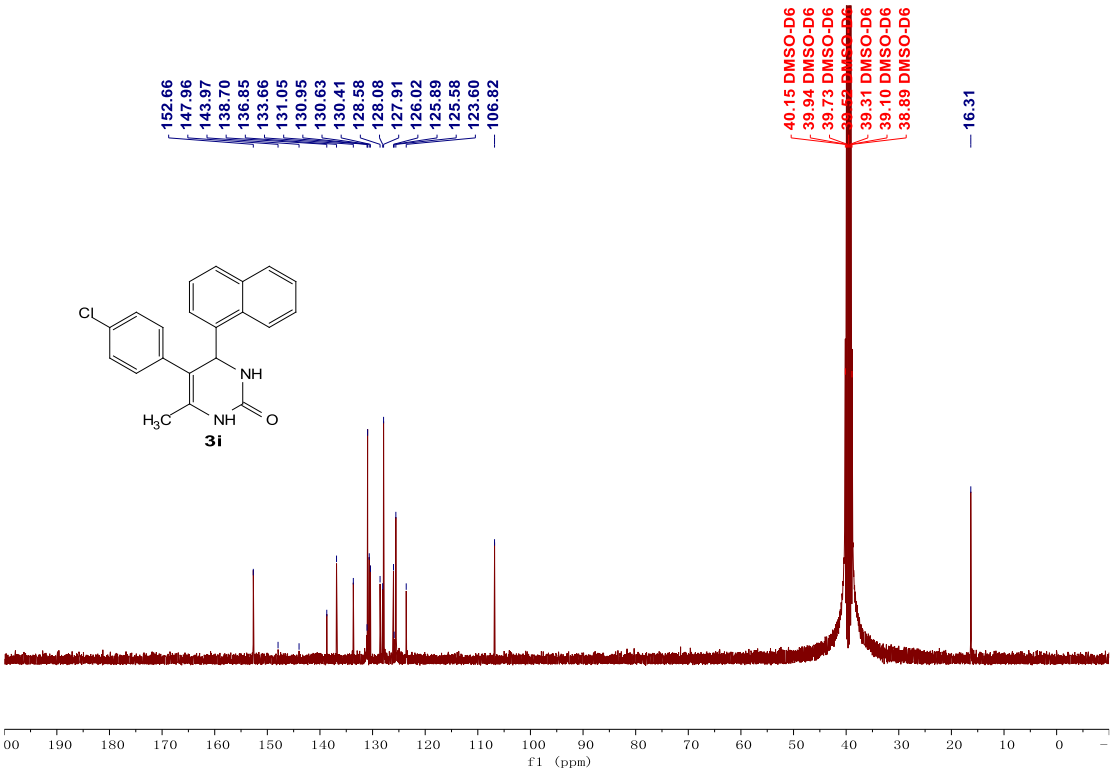
7.48
7.47
7.45
7.44
7.43
7.42
7.36
7.16
7.14
7.08
7.06
7.03
5.99
2.50 DMSO-D6
1.81
Cl
NH
H3C
NH
O
3i
1.00
1.01
1.06
1.03
4.08
0.98
2.00
1.91
0.92
2.88
f1 (ppm)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130.95
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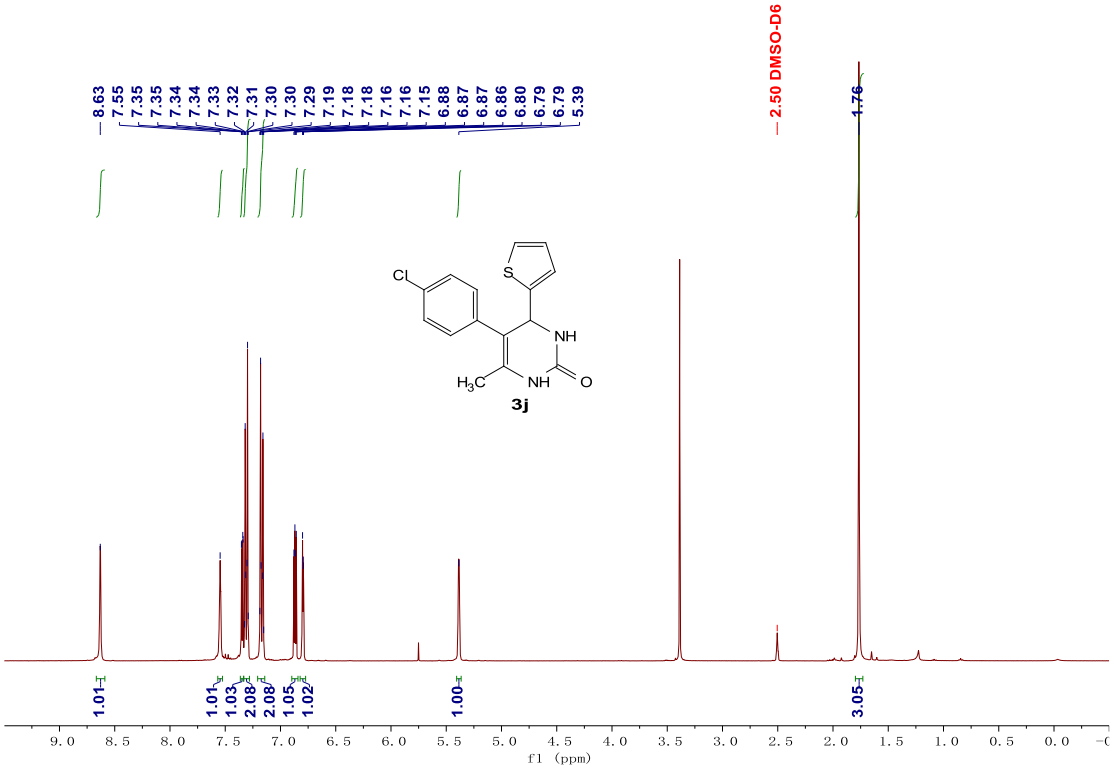
130.41
128.58
128.08
127.91
126.02
125.89
125.58
123.60
106.82
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.31
Cl
NH
H3C
NH
O
3i
f1 (ppm)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7.30
7.29
7.19
7.18
7.18
7.16
7.16
7.15
6.88
6.87
6.87
6.86
6.80
6.79
6.79
5.39
2.50 DMSO-D6
1.76
Cl
S
NH
H3C
NH
O
3j
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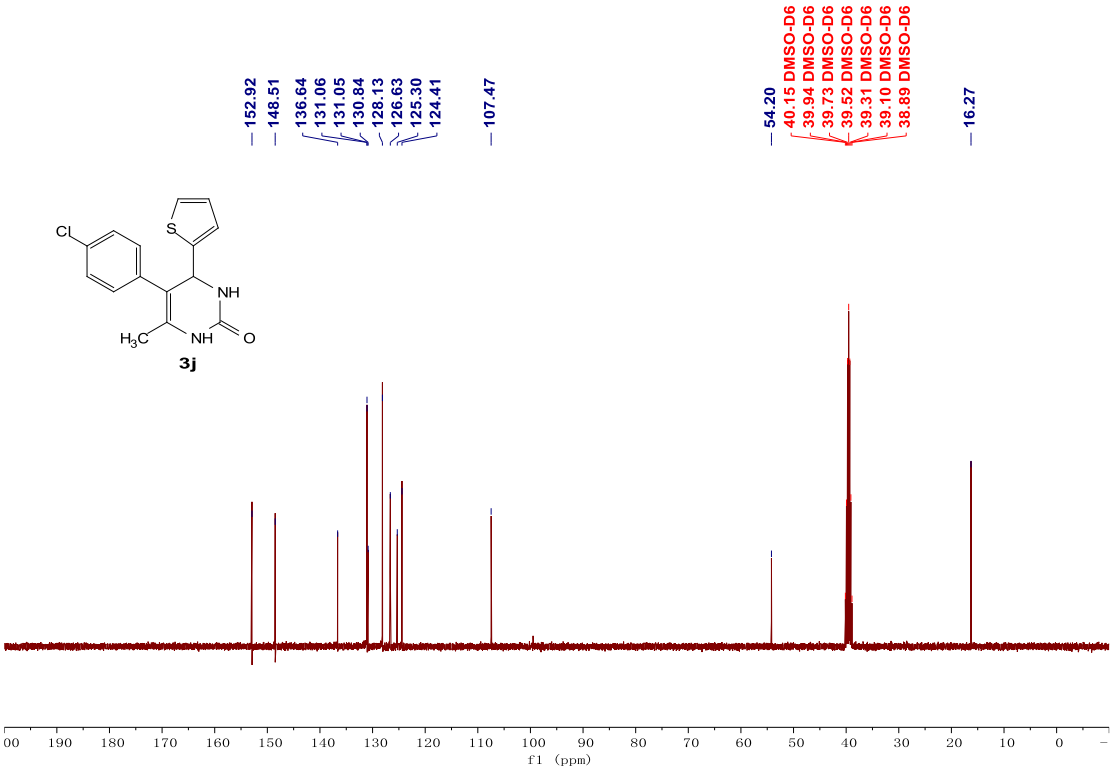
1.01
1.03
2.08
2.08
1.05
1.02
1.00
3.05
f1 (ppm)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126.63
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124.41
107.47
54.20
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39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
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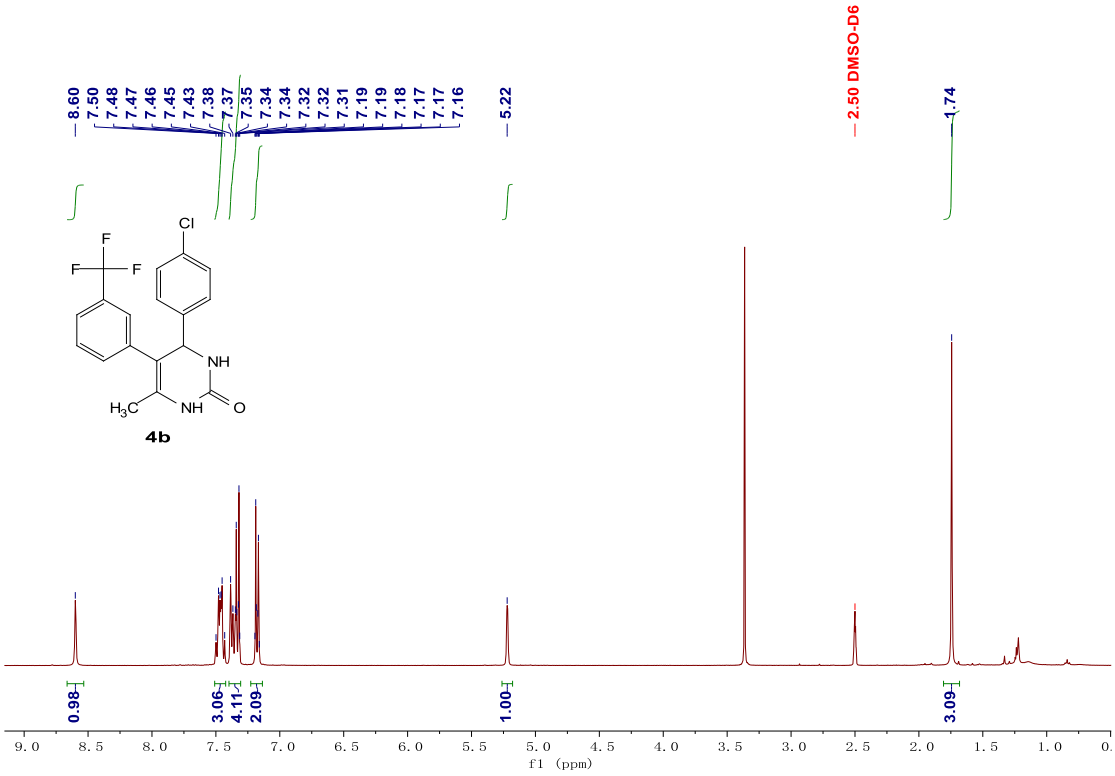
38.89 DMSO-D6
16.27
Cl
S
NH
H3C
NH
O
3j
f1 (ppm)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7.45
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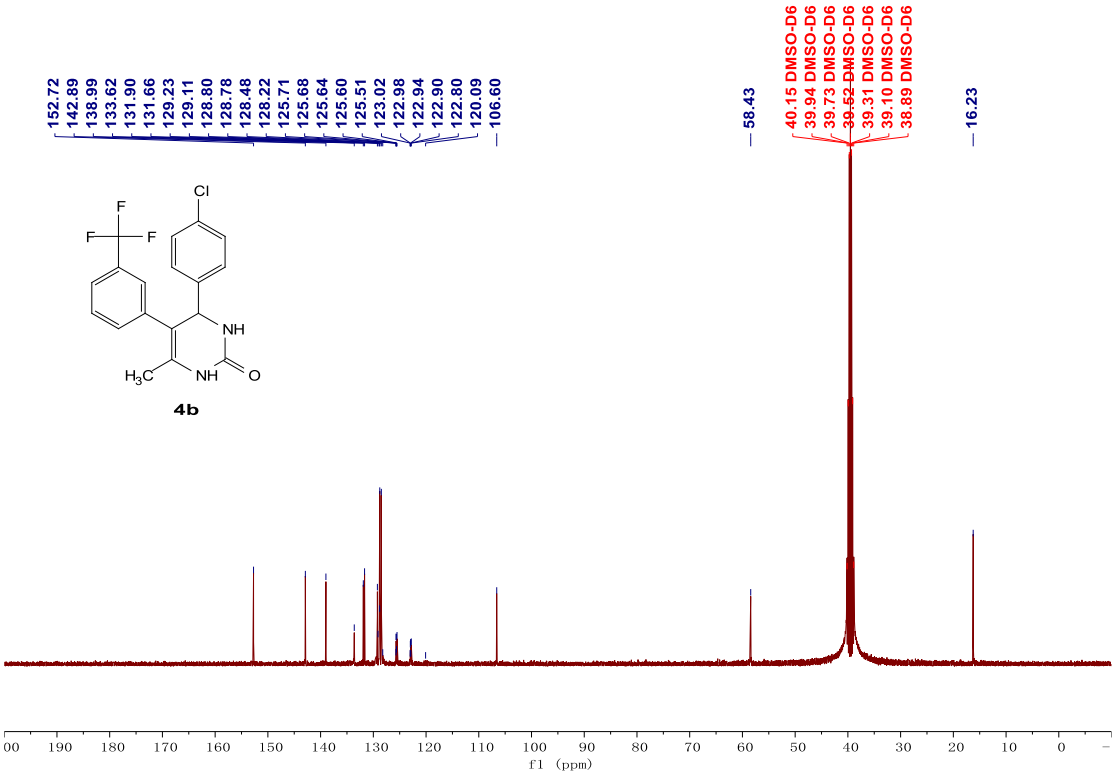
7.38
7.37
7.35
7.34
7.34
7.32
7.32
7.31
7.19
7.19
7.18
7.17
7.17
7.16
5.22
2.50 DMSO-D6
1.74
Cl
F
F
F
NH
H3C
NH
O
4b
0.98
3.06
4.11
2.09
1.00
3.09
f1 (ppm)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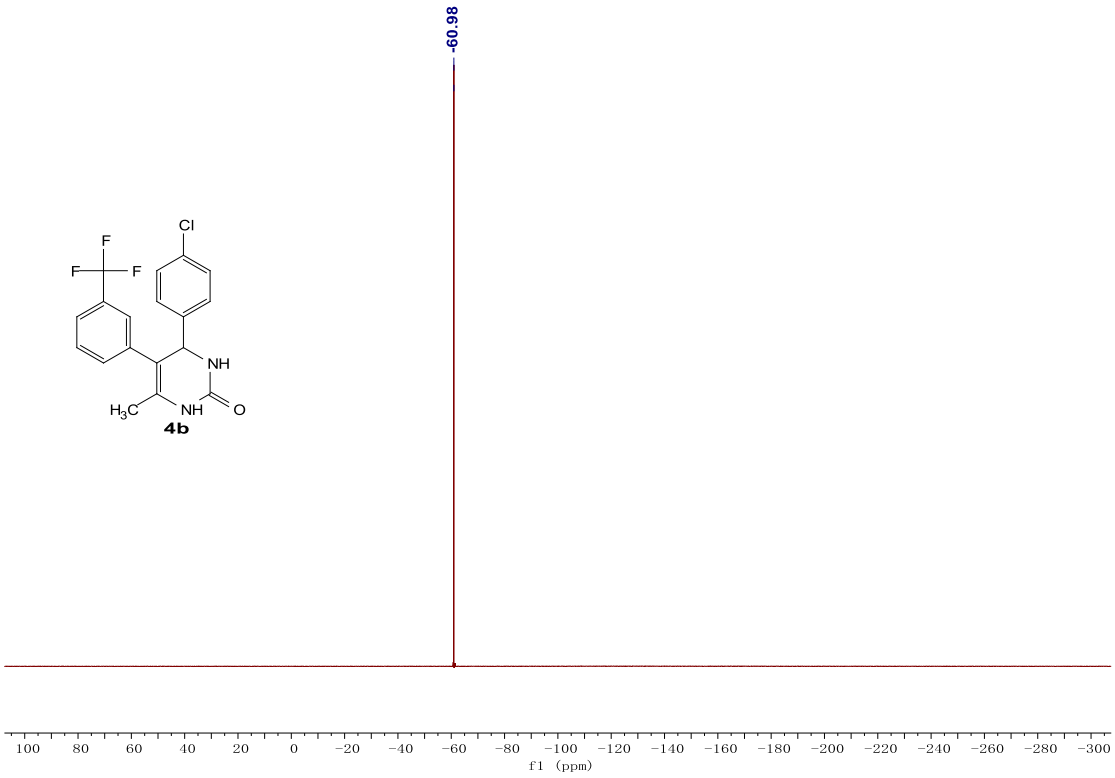
131.90
131.66
129.23
129.11
128.80
128.78
128.48
128.22
125.71
125.68
125.64
125.60
125.51
123.02
122.98
122.94
122.90
122.80
120.09
106.60
58.43
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.23
Cl
F
F
F
NH
H3C
NH
O
4b
f1 (ppm)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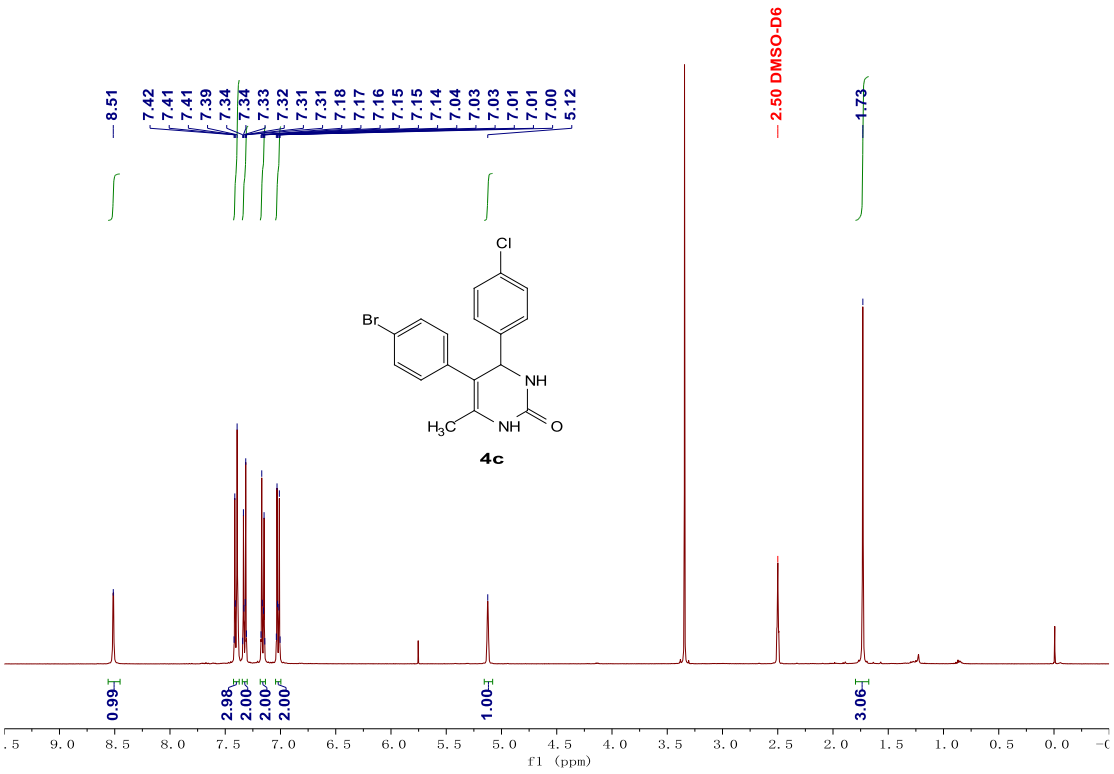
Cl
F
F
F
NH
H3C
NH
O
4b
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
f1 (ppm)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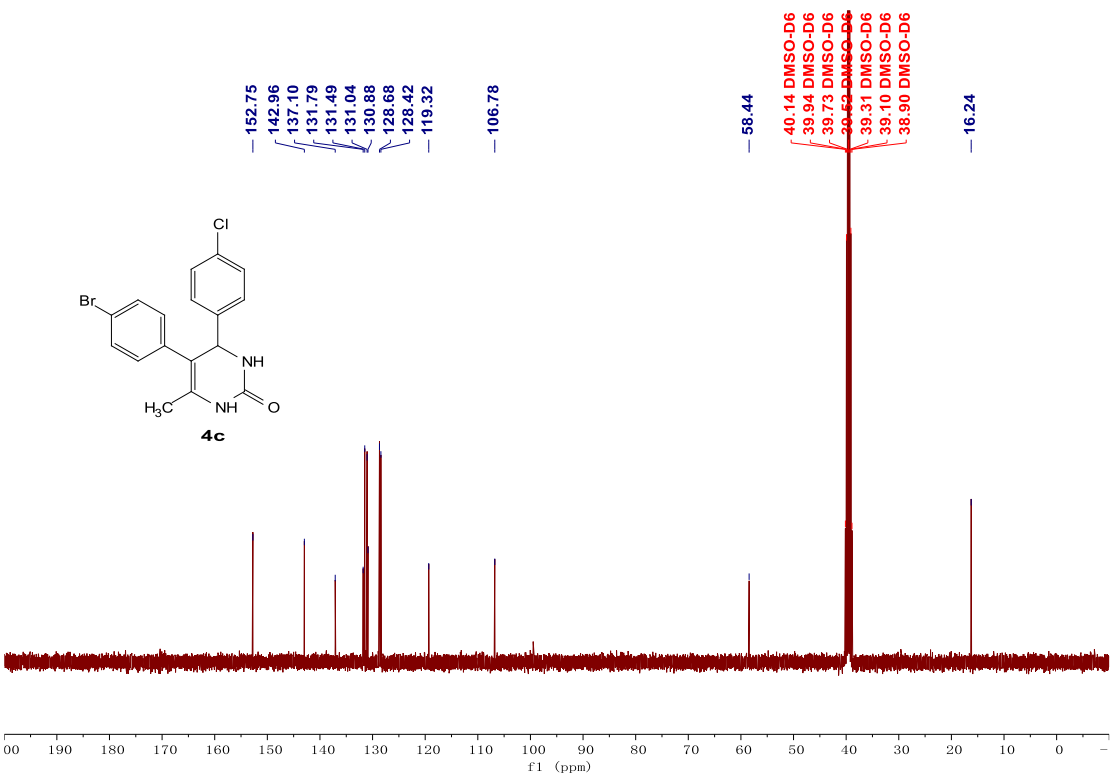
7.41
7.39
7.34
7.34
7.33
7.32
7.31
7.31
7.18
7.17
7.16
7.15
7.15
7.14
7.04
7.03
7.03
7.01
7.01
7.00
5.12
2.50 DMSO-D6
1.73
Cl
Br
NH
H3C
NH
O
4c
0.99
2.98
2.00
2.00
2.00
1.00
3.06
f1 (ppm)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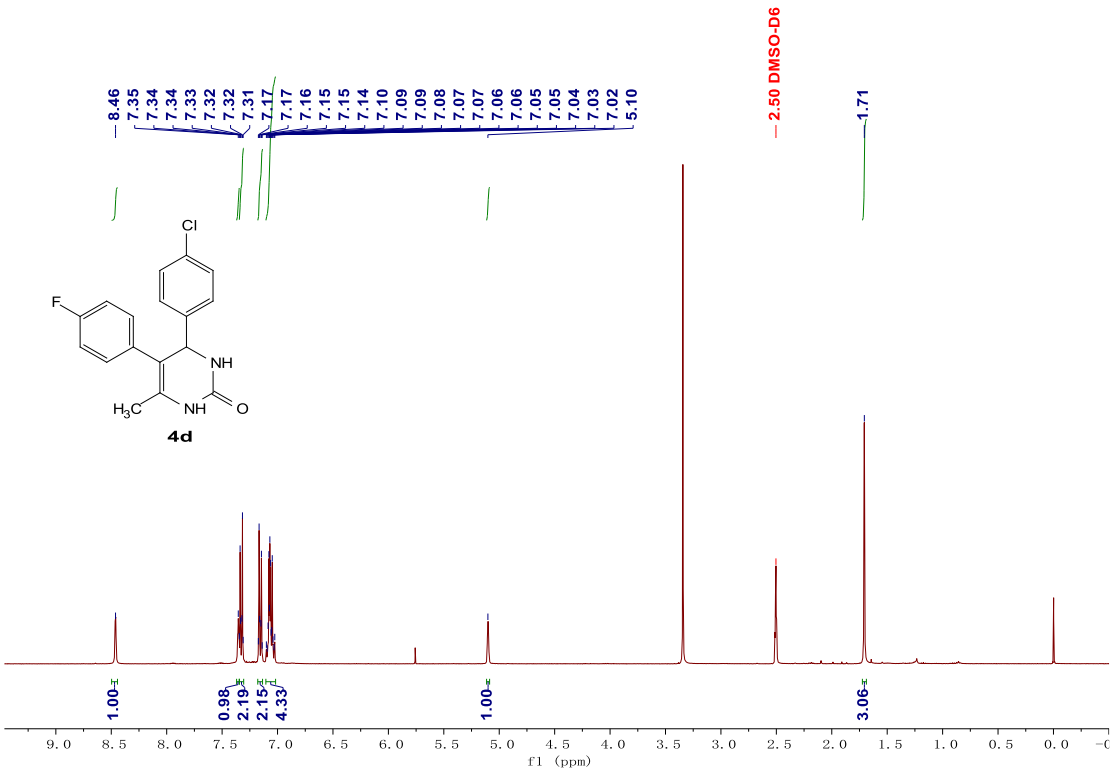
137.10
131.79
131.49
131.04
130.88
128.68
128.42
119.32
106.78
58.44
40.14 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.90 DMSO-D6
16.24
Cl
Br
NH
H3C
NH
O
4c
f1 (ppm)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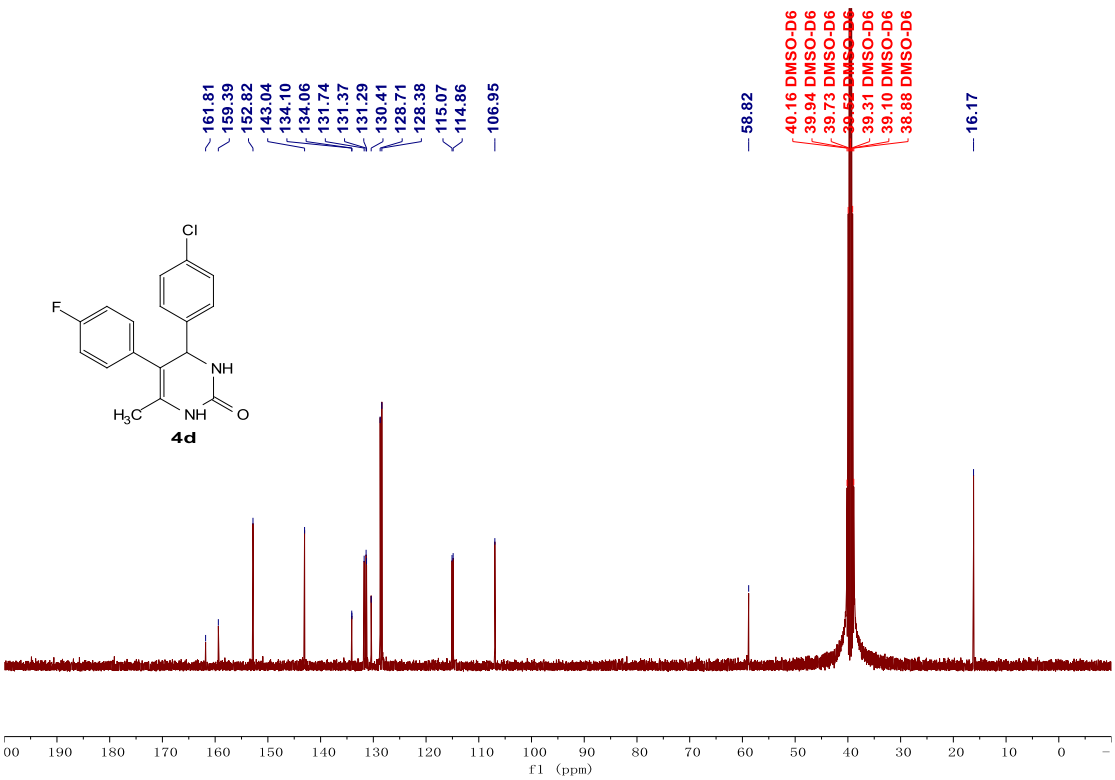
7.34
7.33
7.32
7.32
7.31
7.17
7.17
7.16
7.15
7.15
7.14
7.10
7.09
7.09
7.08
7.07
7.07
7.06
7.06
7.05
7.05
7.04
7.03
7.02
5.10
2.50 DMSO-D6
1.71
Cl
F
NH
H3C
NH
O
4d
1.00
0.98
2.19
2.15
4.33
1.00
3.06
f1 (ppm)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159.39
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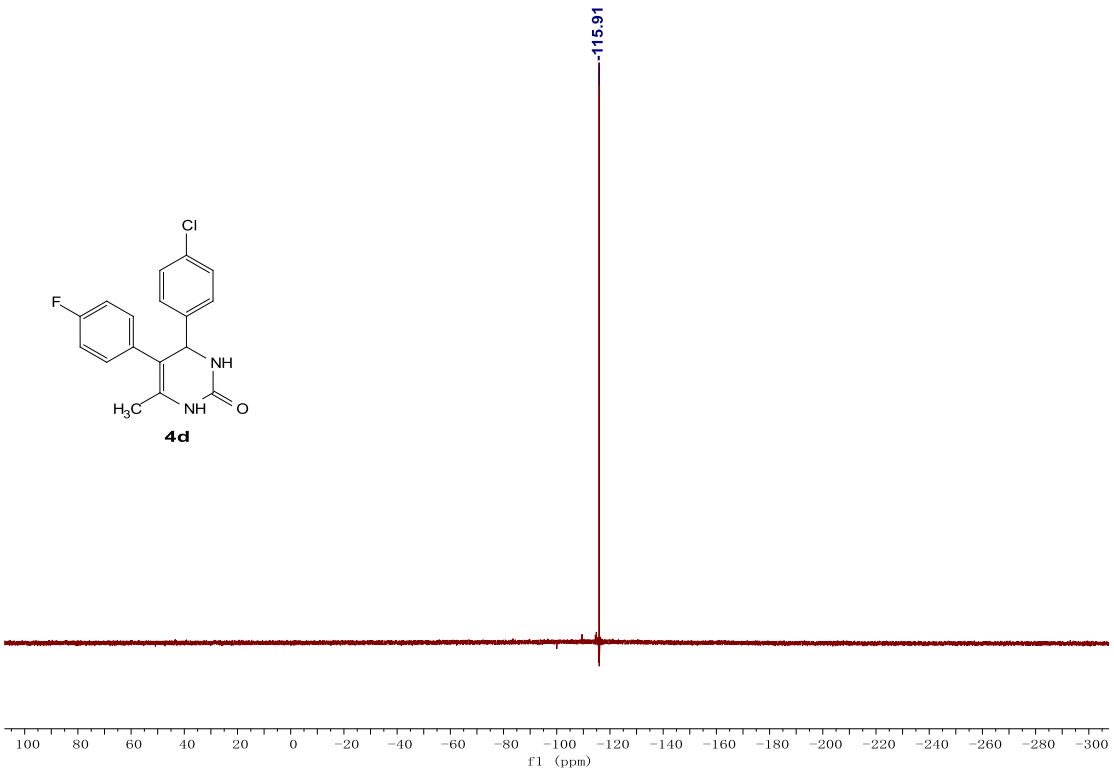
134.10
134.06
131.74
131.37
131.29
130.41
128.71
128.38
115.07
114.86
106.95
58.82
40.16 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.88 DMSO-D6
16.17
Cl
F
NH
H3C
NH
O
4d
f1 (ppm)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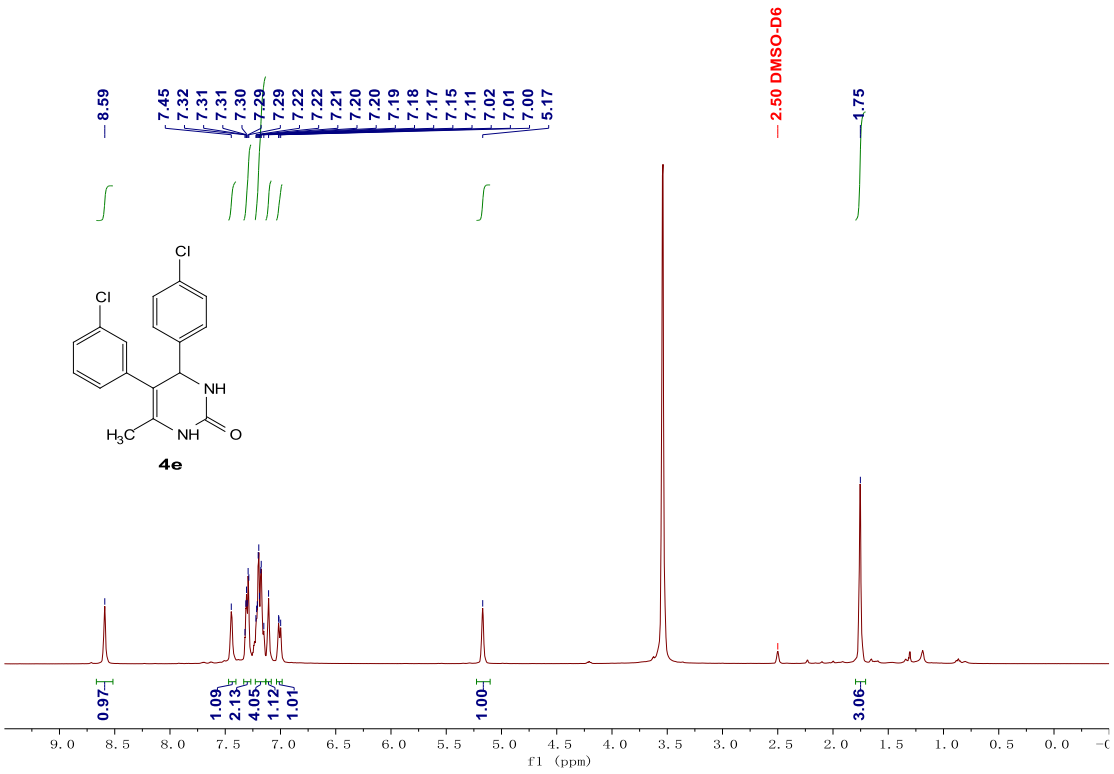
NH
H_3C
NH
O
4d
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
f1 (ppm)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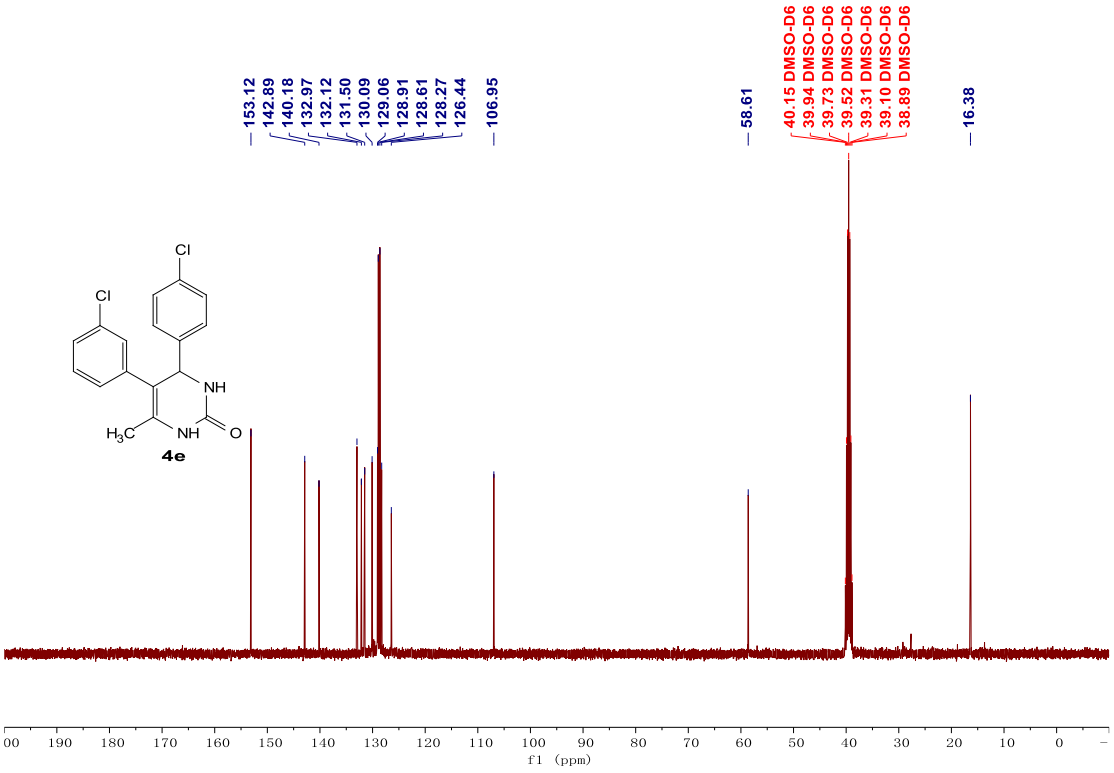
7.31
7.30
7.29
7.29
7.22
7.22
7.21
7.20
7.20
7.19
7.18
7.17
7.15
7.11
7.02
7.01
7.00
5.17
2.50 DMSO-D6
1.75
Cl
Cl
NH
H3C
NH
O
4e
0.97
1.09
2.13
4.05
1.12
1.01
1.00
3.06
f1 (ppm)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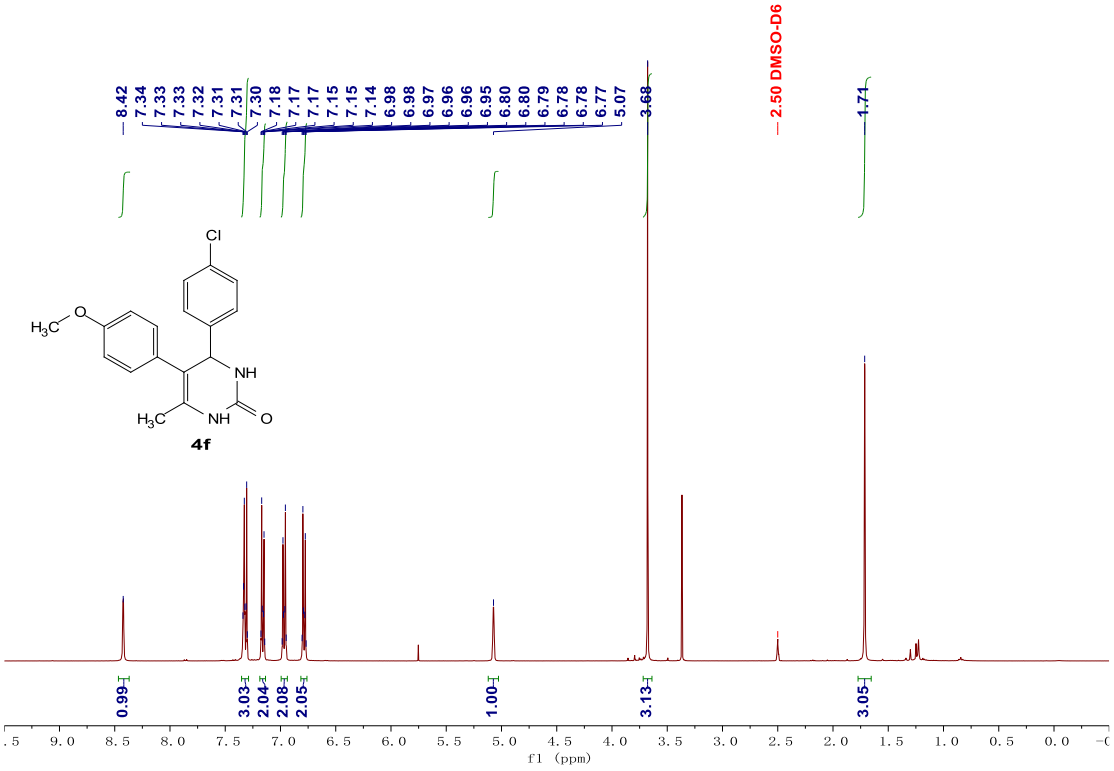
130.09
129.06
128.91
128.61
128.27
126.44
106.95
58.61
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.38
Cl
Cl
NH
H3C
NH
O
4e
f1 (ppm)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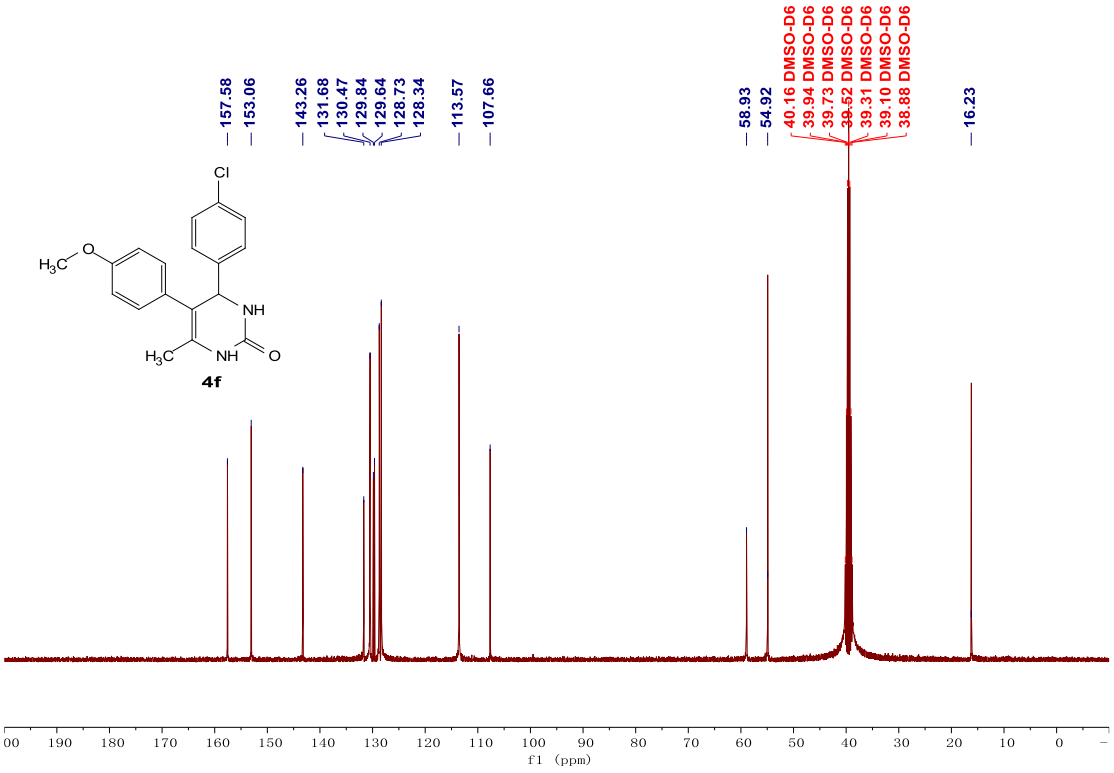
7.30
7.18
7.17
7.17
7.15
7.15
7.14
6.98
6.98
6.97
6.96
6.96
6.95
6.80
6.80
6.79
6.78
6.78
6.77
5.07
3.68
2.50 DMSO-D6
1.71
Cl
O
H3C
NH
H3C
NH
O
4f
0.99
3.03
2.04
2.08
2.05
1.00
3.13
3.05
f1 (ppm)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128.73
128.34
113.57
107.66
58.93
54.92
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39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
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38.88 DMSO-D6
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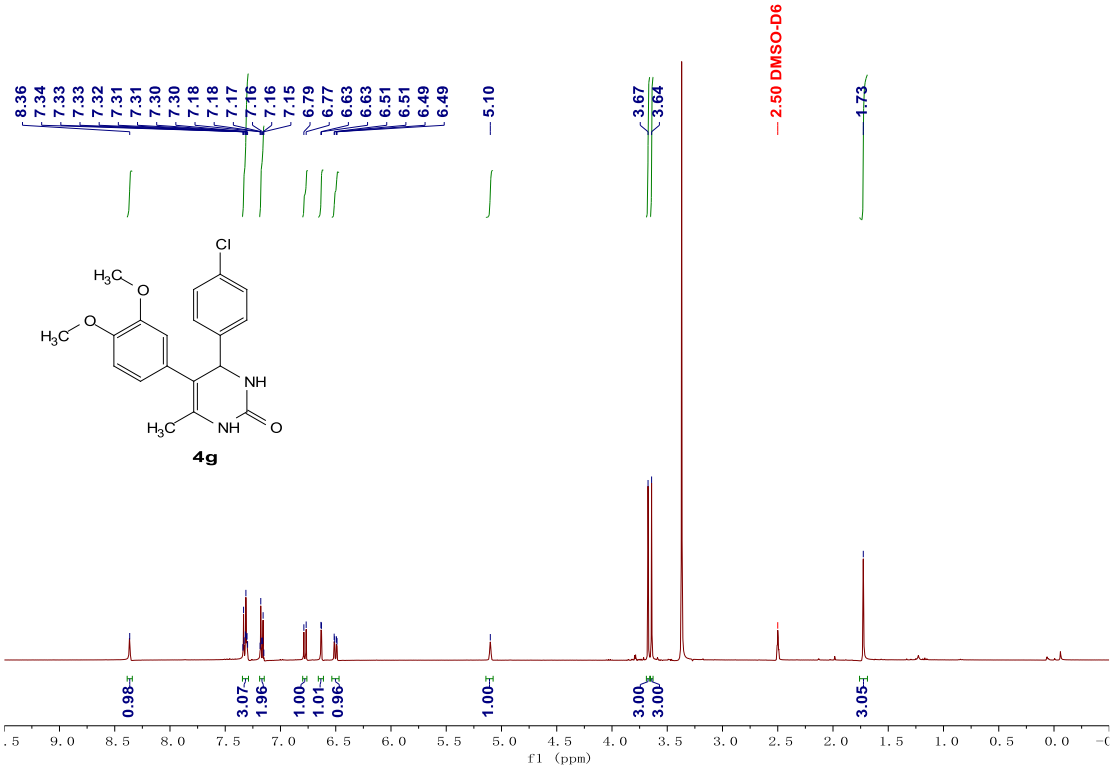
O
H3C
NH
H3C
NH
O
4f
f1 (ppm)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7.30
7.30
7.18
7.18
7.17
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7.16
7.15
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6.77
6.63
6.63
6.51
6.51
6.49
6.49
5.10
3.67
3.64
2.50 DMSO-D6
1.73
Cl
H3C
O
O
H3C
NH
H3C
NH
O
4g
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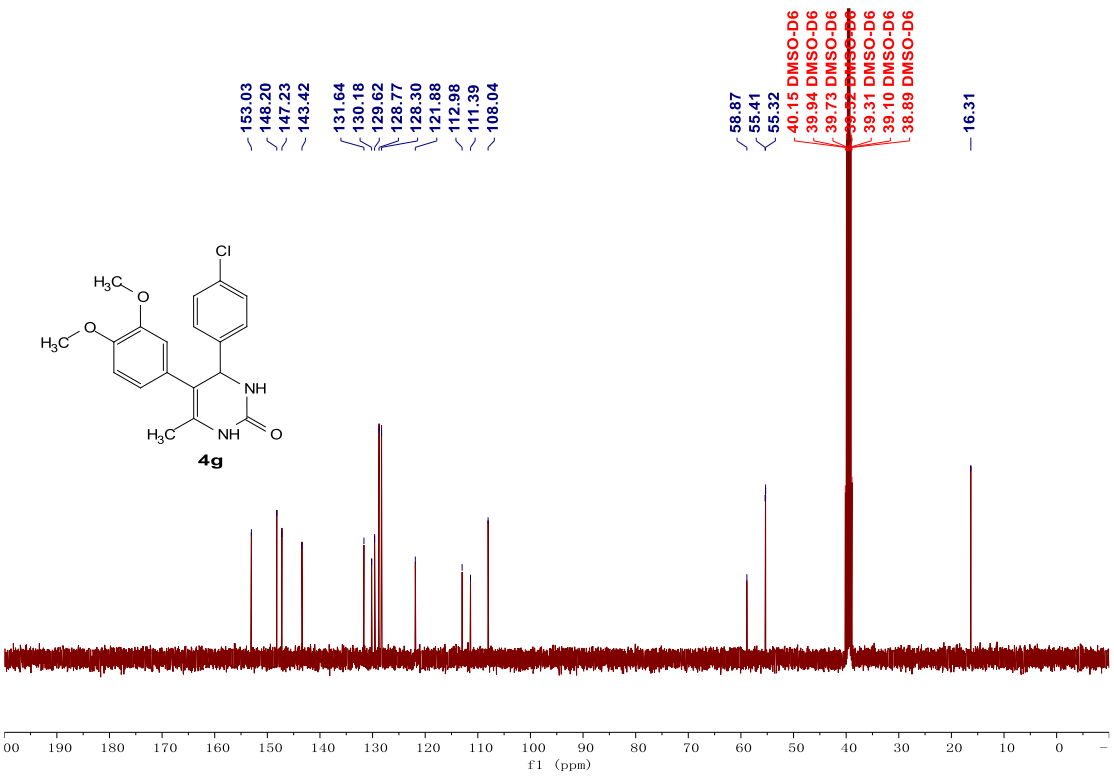
1.96
1.00
1.01
0.96
1.00
3.00
3.00
3.05
f1 (ppm)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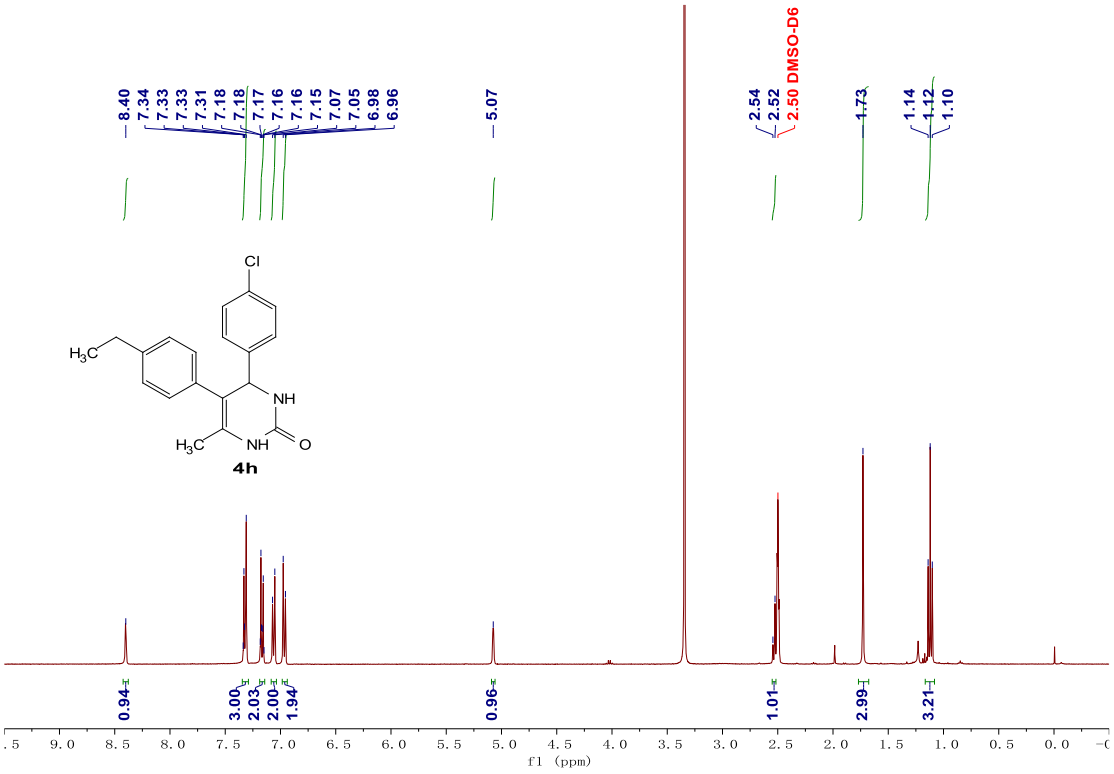
130.18
129.62
128.77
128.30
121.88
112.98
111.39
108.04
58.87
55.41
55.32
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
16.31
Cl
H3C
O
O
H3C
NH
H3C
NH
O
4g
f1 (ppm)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7.18
7.17
7.16
7.16
7.15
7.07
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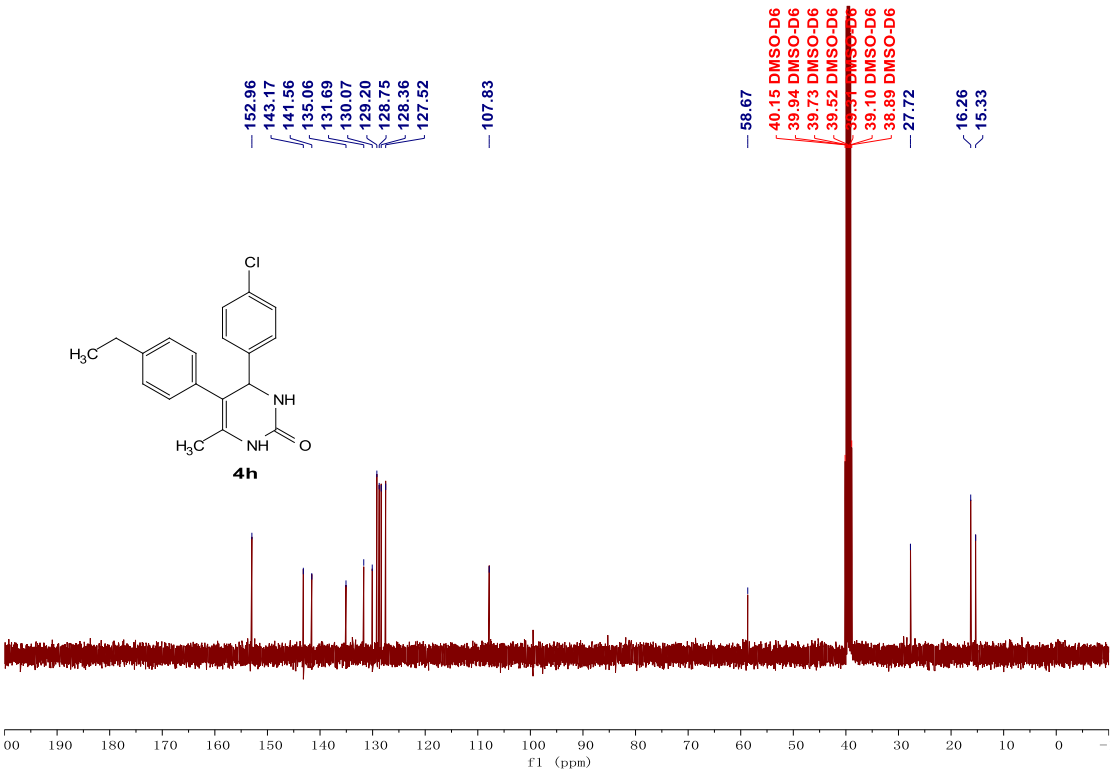
6.98
6.96
5.07
2.54
2.52
2.50 DMSO-D6
1.73
1.14
1.12
1.10
Cl
H3C
NH
H3C
NH
O
4h
0.94
3.00
2.03
2.00
1.94
0.96
1.01
2.99
3.21
f1 (ppm)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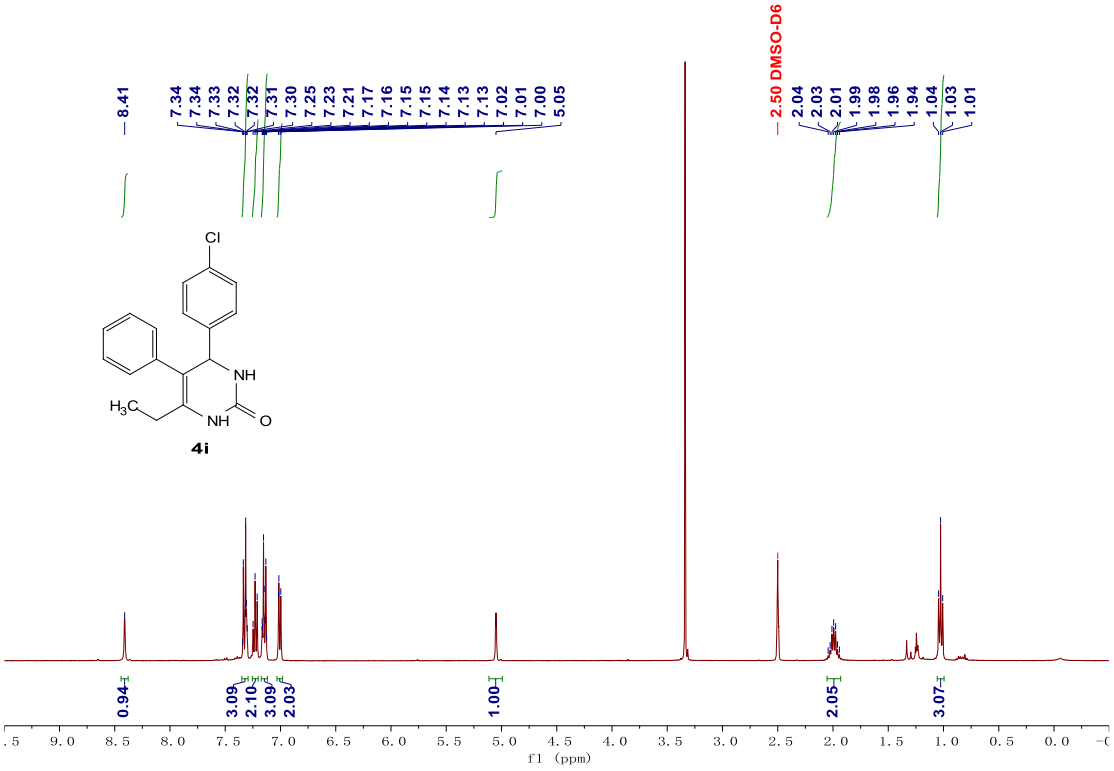
130.07
129.20
128.75
128.36
127.52
107.83
58.67
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
27.72
16.26
15.33
Cl
H3C
NH
H3C
NH
O
4h
f1 (ppm)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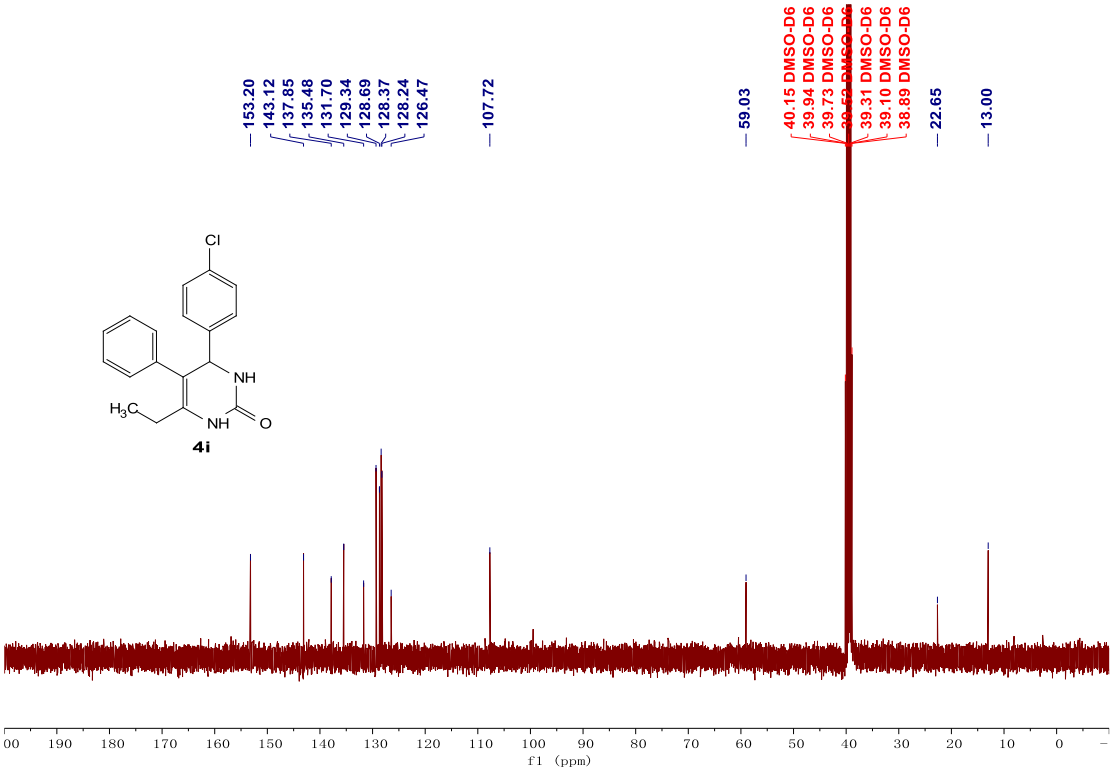
7.30
7.25
7.23
7.21
7.17
7.16
7.15
7.15
7.14
7.13
7.13
7.02
7.01
7.00
5.05
2.50 DMSO-D6
2.04
2.03
2.01
1.99
1.98
1.96
1.94
1.04
1.03
1.01
Cl
NH
H3C
NH
O
4i
0.94
3.09
2.10
3.09
2.03
1.00
2.05
3.07
f1 (ppm)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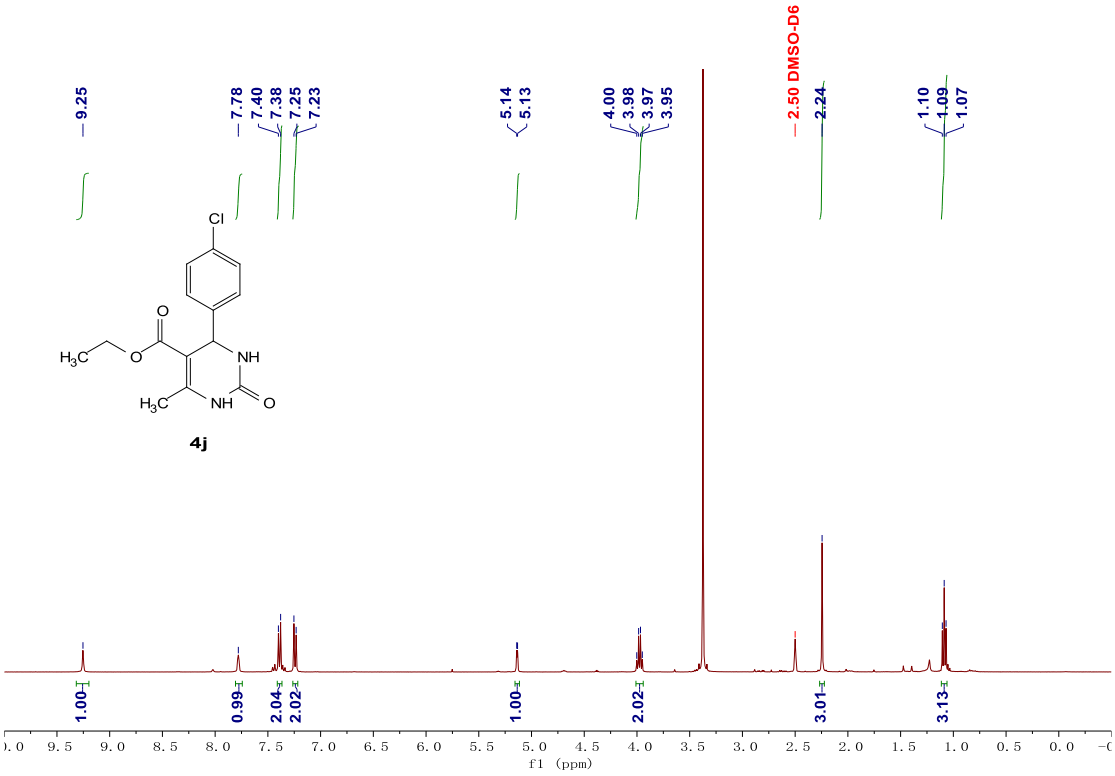
129.34
128.69
128.37
128.24
126.47
107.72
59.03
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
22.65
13.00
Cl
NH
H3C
NH
O
4i
f1 (ppm)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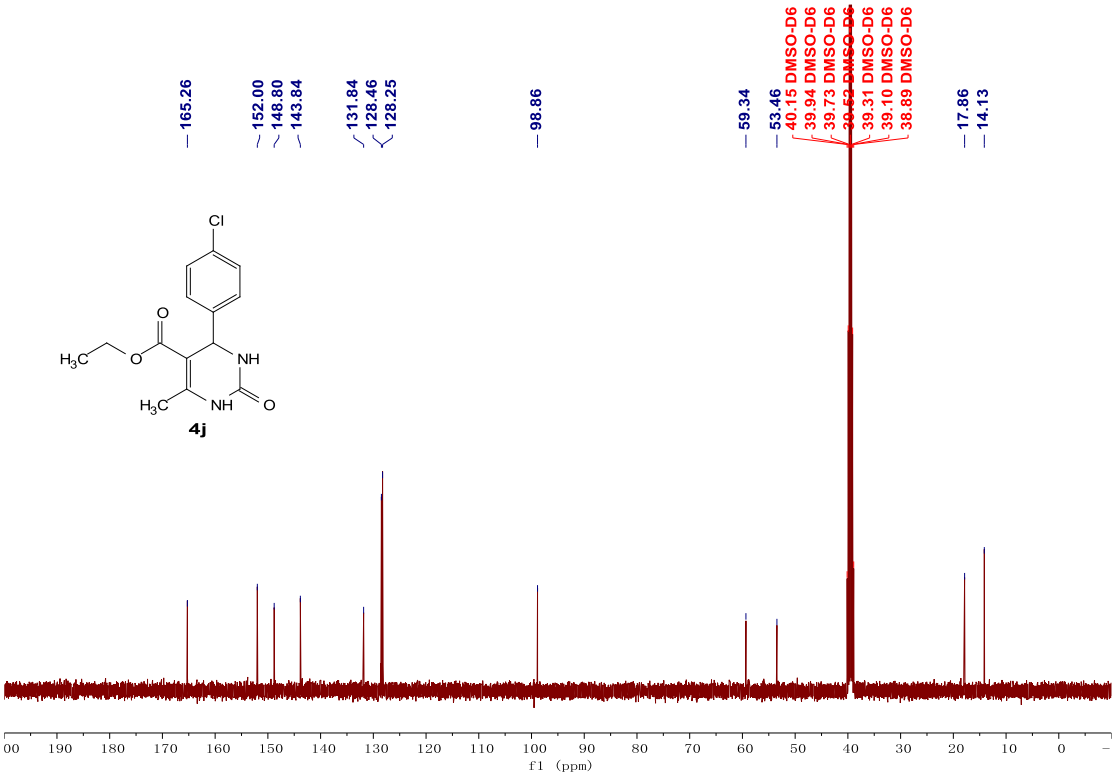
7.25
7.23
5.14
5.13
4.00
3.98
3.97
3.95
2.50 DMSO-D6
2.24
1.10
1.09
1.07
1.00
0.99
2.04
2.02
1.00
2.02
3.01
3.13
f1 (ppm)

Cl
O
O
NH
H3C
NH
H3C
O

4j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128.25
98.86
59.34
53.46
40.15 DMSO-D6
39.94 DMSO-D6
39.73 DMSO-D6
39.52 DMSO-D6
39.31 DMSO-D6
39.10 DMSO-D6
38.89 DMSO-D6
17.86
14.13
f1 (ppm)

Cl
O
O
NH
H3C
NH
H3C
O

4j