

Supporting Information

Visible-light-induced C(sp³)–H thiocyanation of pyrazolin-5-ones: A practical synthesis of 4-thiocyanated 5-hydroxy-1*H*-pyrazoles

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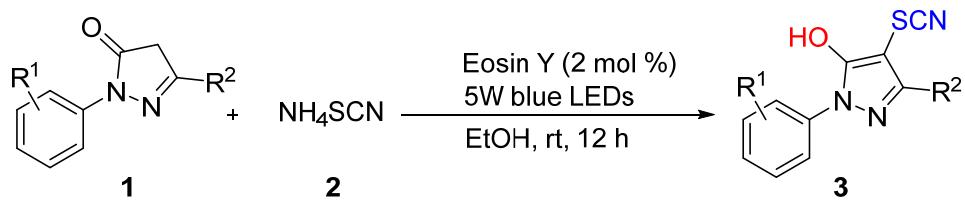
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1. General information

All reactions were carried out at room temperature under air unless otherwise stated. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on 400 MHz NMR spectrometer using DMSO or Methanol-D₄ as the solvent. Chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. Unless otherwise obtained, all reagents were obtained from commercial suppliers and used without further purification. Products were purified by flash chromatography on 200–300 mesh silica gels. Infrared spectra were recorded with a Nicolet 5700 spectrometer and are reported in reciprocal centimeter (cm^{-1}). HRMS data were measured on a Synapt G2-Si instrument.

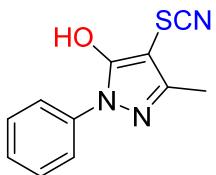
2. Experimental section

2.1 Representative procedure for the model reaction

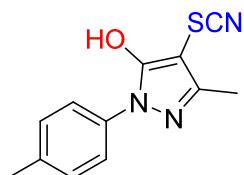


Pyrazolone (**1**, 0.5 mmol, 1.0 eq.) was added to a 10 mL Schlenk bottle with a high vacuum PTFE valve-glass sea. NH_4SCN (**2**, 1.5 mmol, 3.0 eq.), Eosin Y (2 mol%) and EtOH (2 mL) were then added. The reaction was stirred under blue LEDs irradiation for 12 h. At the end of the reaction, the solvent was removed in vacuum and the product was purified by silica gel chromatography using DCM/methanol (6:1 ~ 12:1, v/v) as eluent to give the pure product **3**.

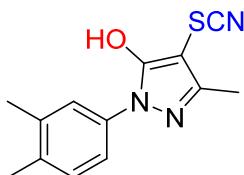
3. Characterization data of products



3-methyl-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3a). Yellow solid, Yield: 84%. ^1H NMR (400 MHz, DMSO- d_6) δ 7.77–7.62 (m, 2H), 7.55–7.45 (m, 2H), 7.38–7.26 (m, 1H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 157.03, 151.42, 137.87, 129.55, 126.87, 121.68, 112.64, 79.58, 12.69; IR (KBr) ν : 3433, 2123, 1655, 1496, 1361, 1015, 823, 761 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_8\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 230.0388; found: 230.0391.

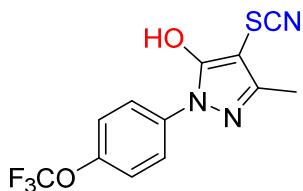


3-methyl-4-thiocyanato-1-(*p*-tolyl)-1*H*-pyrazol-5-ol (3b). Yellow solid, Yield: 77%; ^1H NMR (400 MHz, DMSO- d_6) δ 7.55 (d, $J = 8.2$ Hz, 2H), 7.29 (d, $J = 8.2$ Hz, 2H), 2.34 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 156.83, 151.04, 136.28, 135.53, 129.93, 121.73, 112.68, 79.19, 21.00, 12.68; IR (KBr) ν : 3441, 2121, 1653, 1516, 1363, 1022, 821, 762 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{10}\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 244.0545; found: 244.0547.



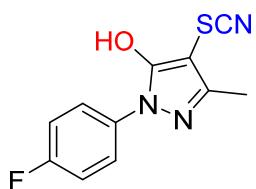
1-(3,4-dimethylphenyl)-3-methyl-4-thiocyanato-1*H*-pyrazol-5-ol (3c). Yellow solid, Yield: 85%; ^1H NMR (400 MHz, methanol- d_4) δ 7.35 (s, 1H), 7.28 (d, $J = 8.2$ Hz, 1H), 7.22 (d, $J = 8.2$ Hz, 1H), 2.37 (s, 3H), 2.29 (d, $J = 6.6$ Hz, 6H); ^{13}C NMR (101 MHz, methanol- d_4) δ 158.57, 150.94, 137.53, 136.05, 133.98, 129.82, 123.28,

119.72, 110.86, 80.68, 18.55, 18.07, 10.43; IR (KBr) ν : 3447, 2156, 1625, 1502, 1445, 1211, 974, 797 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{12}\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 258.0701; found: 258.0705.

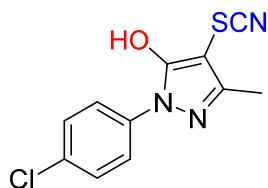


3-methyl-4-thiocyanato-1-(4-(trifluoromethoxy)phenyl)-1*H*-pyrazol-5-ol (3d)

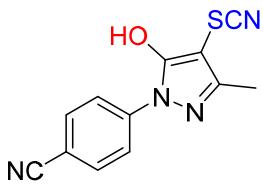
Yellow solid, Yield: 81%; ¹H NMR (400 MHz, methanol-*d*₄) δ 7.76 (d, *J* = 8.7 Hz, 2H), 7.41 (d, *J* = 8.7 Hz, 2H), 2.38 (s, 3H); ¹³C NMR (101 MHz, methanol-*d*₄) δ 158.03, 152.11, 147.36, 135.64, 123.30, 121.57, 120.49 (*q*, *J*_{CF} = 128.3 Hz), 120.49 (*q*, *J*_{CF} = 384.8 Hz), 110.80, 80.49, 10.74; ¹⁹F NMR (377 MHz, methanol-*d*₄) δ -59.63 (s, 3F); IR (KBr) ν : 3429, 2160, 1631, 1510, 1267, 1215, 1167, 841 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_7\text{F}_3\text{N}_3\text{O}_2\text{S}$ ($[\text{M}-\text{H}]^-$): 314.0211; found: 314.0208.



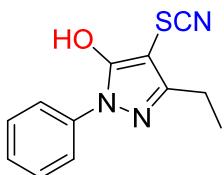
1-(4-fluorophenyl)-3-methyl-4-thiocyanato-1*H*-pyrazol-5-ol (3e). Yellow solid, Yield: 71%; ¹H NMR (400 MHz, methanol-*d*₄) δ 7.80–7.66 (m, 2H), 7.17–7.06 (m, 2H), 2.23 (s, 3H) ppm. ¹³C NMR (101 MHz, methanol-*d*₄) δ 163.83, 159.95 (*d*, *J*_{CF} = 121.2 Hz), 150.97, 136.16 (*d*, *J*_{CF} = 3.0 Hz), 122.89 (*d*, *J*_{CF} = 8.1 Hz), 114.67 (*d*, *J*_{CF} = 11.6 Hz), 113.30, 72.62, 11.71; ¹⁹F NMR (377 MHz, methanol-*d*₄) δ -120.29 (s, 1F); IR (KBr) ν : 3375, 2158, 1616, 1493, 1410, 1323, 833, 741 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_7\text{FN}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 248.0654; found: 248.0651.



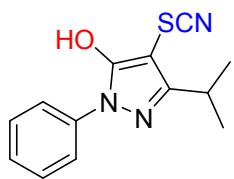
1-(4-chlorophenyl)-3-methyl-4-thiocyanato-1*H*-pyrazol-5-ol (3f). Yellow solid, Yield: 72%; ^1H NMR (400 MHz, DMSO-*d*6) δ 7.74 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 8.5 Hz, 2H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, DMSO-*d*6) δ 157.14, 151.84, 136.80, 130.83, 129.53, 122.94, 112.55, 79.78, 12.73; IR (KBr) ν : 3442, 2155, 1626, 1491, 1328, 834, 740 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_7\text{ClN}_3\text{OS}$ ([M-H] $^-$): 263.9999; found: 263.9995.



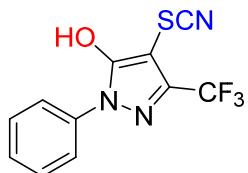
1-(4-cyanophenyl)-3-methyl-4-thiocyanato-1*H*-pyrazol-5-ol (3g). Yellow solid, Yield: 66%; ^1H NMR (400 MHz, methanol-*d*4) δ 8.14 (d, *J* = 8.7 Hz, 2H), 7.69 (d, *J* = 8.7 Hz, 2H), 2.25 (s, 3H); ^{13}C NMR (101 MHz, methanol-*d*4) δ 165.23, 152.96, 143.94, 132.49, 119.05, 118.75, 113.08, 105.72, 73.09, 11.92, IR (KBr) ν : 3445, 2233, 2142, 1606, 1513, 976, 829 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_7\text{N}_4\text{OS}$ ([M-H] $^-$): 255.0341; found: 255.0338.



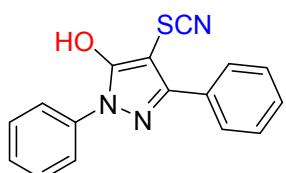
3-ethyl-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3h). Yellow solid, Yield: 61%; ^1H NMR (400 MHz, methanol-*d*4) δ 7.62–7.61 (m, 2H), 7.51–7.45 (m, 2H), 7.39–7.32 (m, 1H), 2.81–2.74 (m, 2H), 1.37–1.31 (m, 3H); ^{13}C NMR (101 MHz, methanol-*d*4) δ 158.52, 156.45, 136.63, 128.87, 127.07, 122.28, 111.21, 79.46, 19.73, 11.72; IR (KBr) ν : 2980, 2156, 1626, 1496, 1305, 824, 723 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{10}\text{N}_3\text{OS}$ ([M-H] $^-$): 244.0545; found: 244.0542.



3-(*iso*-propyl)-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3i**).** Yellow solid, Yield: 68%; ^1H NMR (400 MHz, DMSO- d_6) δ 8.08–8.06 (m, 2H), 7.29–7.23 (m, 2H), 6.96–6.90 (m, 1H), 2.96–2.87 (m, 1H), 1.27–1.23 (m, 6H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.93, 157.87, 142.26, 128.54, 121.88, 117.67, 115.32, 65.31, 28.08, 21.83; IR (KBr) ν : 3448, 2928, 2155, 1603, 1503, 1365, 1105, 756 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{12}\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 258.0701; found: 258.0698.

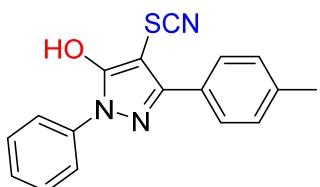


1-phenyl-4-thiocyanato-3-(trifluoromethyl)-1*H*-pyrazol-5-ol(3j**)** Yellow solid, Yield: 83%; ^1H NMR (400 MHz, DMSO- d_6) δ 8.04 (d, $J = 7.8$ Hz, 2H), 7.41–7.33 (m, 2H), 7.15–7.08 (m, 1H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 164.76, 141.00, 140.72, 128.93, 124.17, 121.96 (q, $J_{\text{CF}} = 270.7$ Hz), 121.96 (q, $J_{\text{CF}} = 406.0$ Hz), 118.97, 113.91, 66.33; ^{19}F NMR (377 MHz, DMSO- d_6) δ –62.23 (s, 3F); IR (KBr) ν : 3443, 2123, 1636, 1475, 1175, 1120, 1022, 762 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_5\text{F}_3\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 284.0106; found: 284.0102.

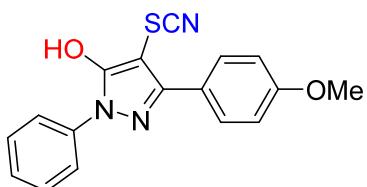


1,3-diphenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3k**).** Yellow solid, Yield: 73%; ^1H NMR (400 MHz, DMSO- d_6) δ 8.21–8.10 (m, 2H), 7.93–7.84 (m, 2H), 7.51–7.42 (m, 2H), 7.39–7.30 (m, 3H), 7.07–6.97 (m, 1H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 166.02, 150.28, 141.82, 134.88, 128.72, 128.65, 128.05, 127.47, 122.84, 118.30,

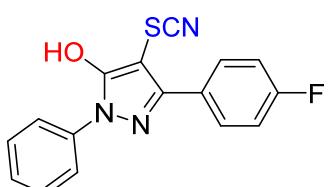
115.32, 66.66; IR (KBr) ν : 3454, 2153, 1638, 1495, 1251, 1121, 1020 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{10}\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 292.0545; found: 292.0540.



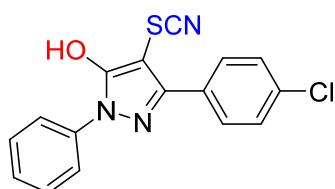
1-phenyl-4-thiocyanato-3-(*p*-tolyl)-1*H*-pyrazol-5-ol (3l). Yellow solid, Yield: 87%; ^1H NMR (400 MHz, methanol- d_4) δ 7.82–7.76 (m, 2H), 7.76–7.70 (m, 2H), 7.47–7.41 (m, 2H), 7.34–7.24 (m, 3H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, methanol- d_4) δ 159.26, 150.85, 137.27, 136.93, 127.39, 127.20, 126.94, 126.17, 124.42, 120.49, 111.12, 73.71, 18.43; IR (KBr) ν : 3381, 2930, 2152, 1611, 1497, 1310, 823, 762 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3\text{OS}$ ($[\text{M}-\text{H}]^-$): 306.0701; found: 306.0703.



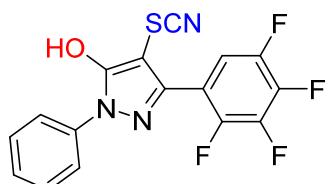
3-(4-methoxyphenyl)-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3m). Yellow solid, Yield: 77%; ^1H NMR (400 MHz, methanol- d_4) δ 7.80 (d, $J = 8.6$ Hz, 2H), 7.72 (d, $J = 8.6$ Hz, 2H), 7.55–7.44 (m, 2H), 7.41–7.31 (m, 1H), 7.08–7.06 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (101 MHz, methanol- d_4) δ 161.04, 158.63, 152.32, 137.20, 129.34, 128.81, 126.97, 122.30, 121.70, 113.82, 111.67, 78.25, 54.52; IR (KBr) ν : 3435, 2155, 1612, 1501, 1402, 1253, 1028, 831 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3\text{O}_2\text{S}$ ($[\text{M}-\text{H}]^-$): 322.0650; found: 322.0647.



3-(4-fluorophenyl)-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3n) Yellow solid, Yield: 61%; ^1H NMR (400 MHz, methanol- d_4) δ 7.90–7.82 (m, 4H), 7.42–7.36 (m, 2H), 7.23–7.14 (m, 3H); ^{13}C NMR (101 MHz, methanol- d_4) δ 164.21, 162.87 (d, $J_{\text{CF}} = 245.0$ Hz), 151.23, 139.85, 129.75 (d, $J_{\text{CF}} = 3.0$ Hz), 129.64 (d, $J_{\text{CF}} = 4.0$ Hz), 128.24, 124.85, 121.60, 114.71 (d, $J_{\text{CF}} = 10.0$ Hz), 113.89, 71.81; ^{19}F NMR (377 MHz, methanol- d_4) δ -115.76 (s, 1F); IR (KBr) ν : 3441, 2158, 1562, 1512, 1219, 1089, 839 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_9\text{FN}_3\text{OS}$ ([M-H] $^-$): 310.0451; found: 310.0455.

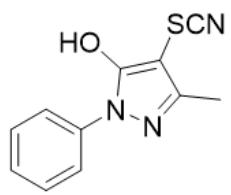


3-(4-chlorophenyl)-1-phenyl-4-thiocyanato-1*H*-pyrazol-5-ol (3o) Yellow solid, Yield: 69%; ^1H NMR (400 MHz, DMSO- d_6) δ 8.36–8.07 (m, 2H), 8.06–7.81 (m, 2H), 7.67–7.47 (m, 2H), 7.45–7.29 (m, 2H), 7.15–6.96 (m, 1H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.71, 148.84, 145.47, 141.67, 133.69, 132.64, 128.97, 128.76, 123.07, 118.47, 115.11, 67.02; IR (KBr) ν : 3439, 2124, 1654, 1020, 824, 762 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_9\text{ClN}_3\text{OS}$ ([M-H] $^-$): 326.0155; found: 326.0152.



1-phenyl-3-(2,3,4,5-tetrafluorophenyl)-4-thiocyanato-1*H*-pyrazol-5-ol (3p). Yellow solid, Yield: 65%; ^1H NMR (400 MHz, methanol- d_4) δ 7.85–7.83 (m, 2H), 7.44–7.33 (m, 3H), 7.24–7.18 (m, 1H); ^{13}C NMR (101 MHz, methanol- d_4) δ 164.22, 145.65, 141.61, 139.76, 139.08, 128.25, 125.01, 121.39, 118.48, 118.26, 115.28, 113.03, 112.06, 73.52; ^{19}F NMR (376 MHz, methanol- d_4) δ -140.33 ~ -140.43 (m, 1F), -142.03 ~ -142.14 (m, 1F), -158.61 ~ -158.81 (m, 2F); IR (KBr) ν : 3406, 2155, 1541, 1490, 1057, 982, 865, 762 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_6\text{F}_4\text{N}_3\text{OS}$ ([M-H] $^-$): 364.0168; found: 364.0165.

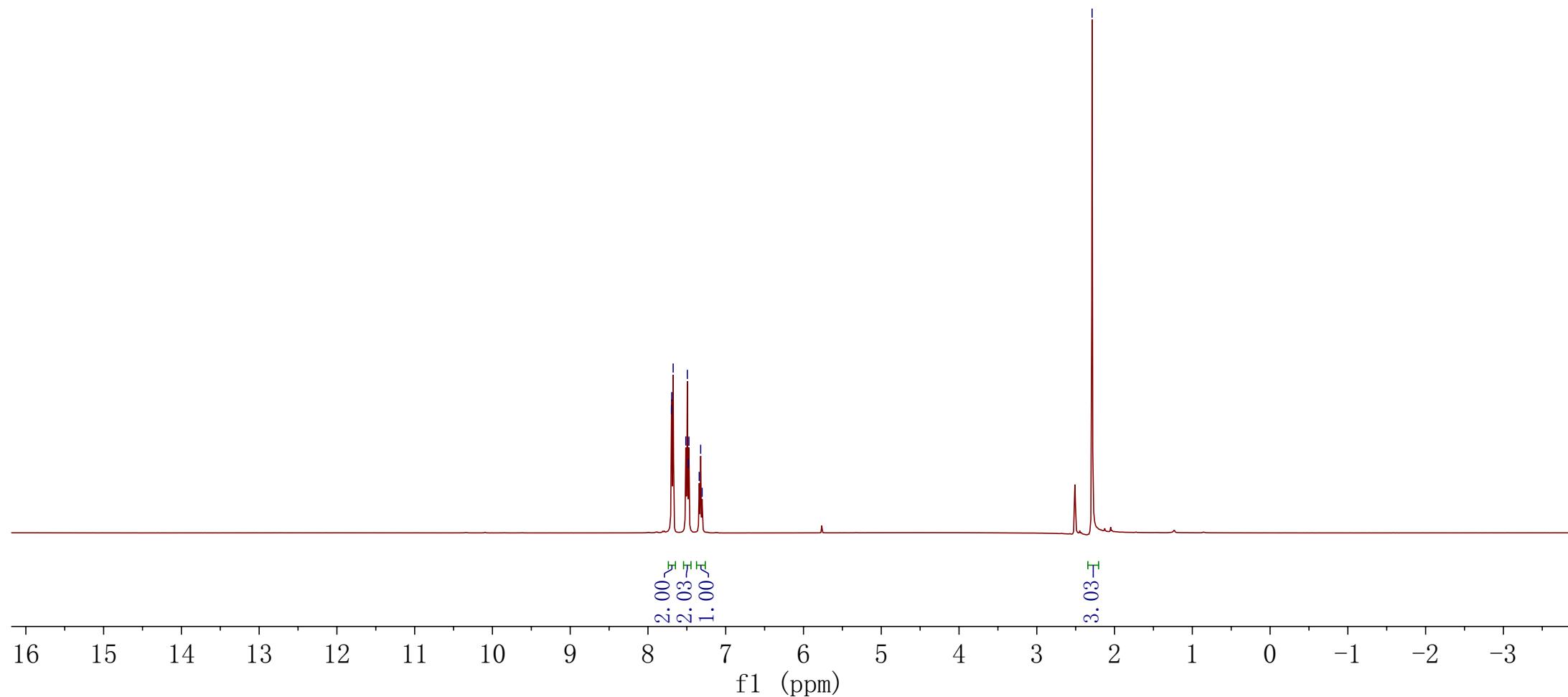
4. ^1H , ^{13}C and ^{19}F NMR spectra of the products

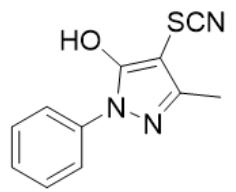


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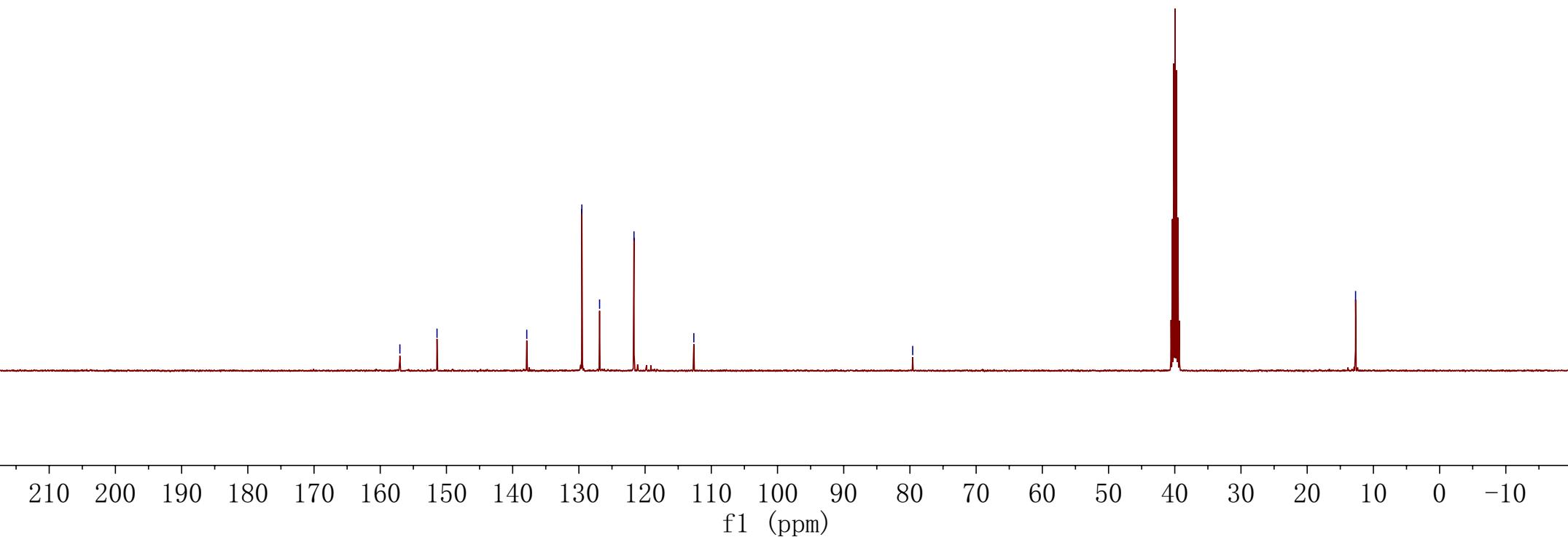
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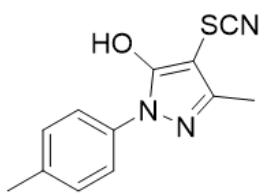
¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of **3a**



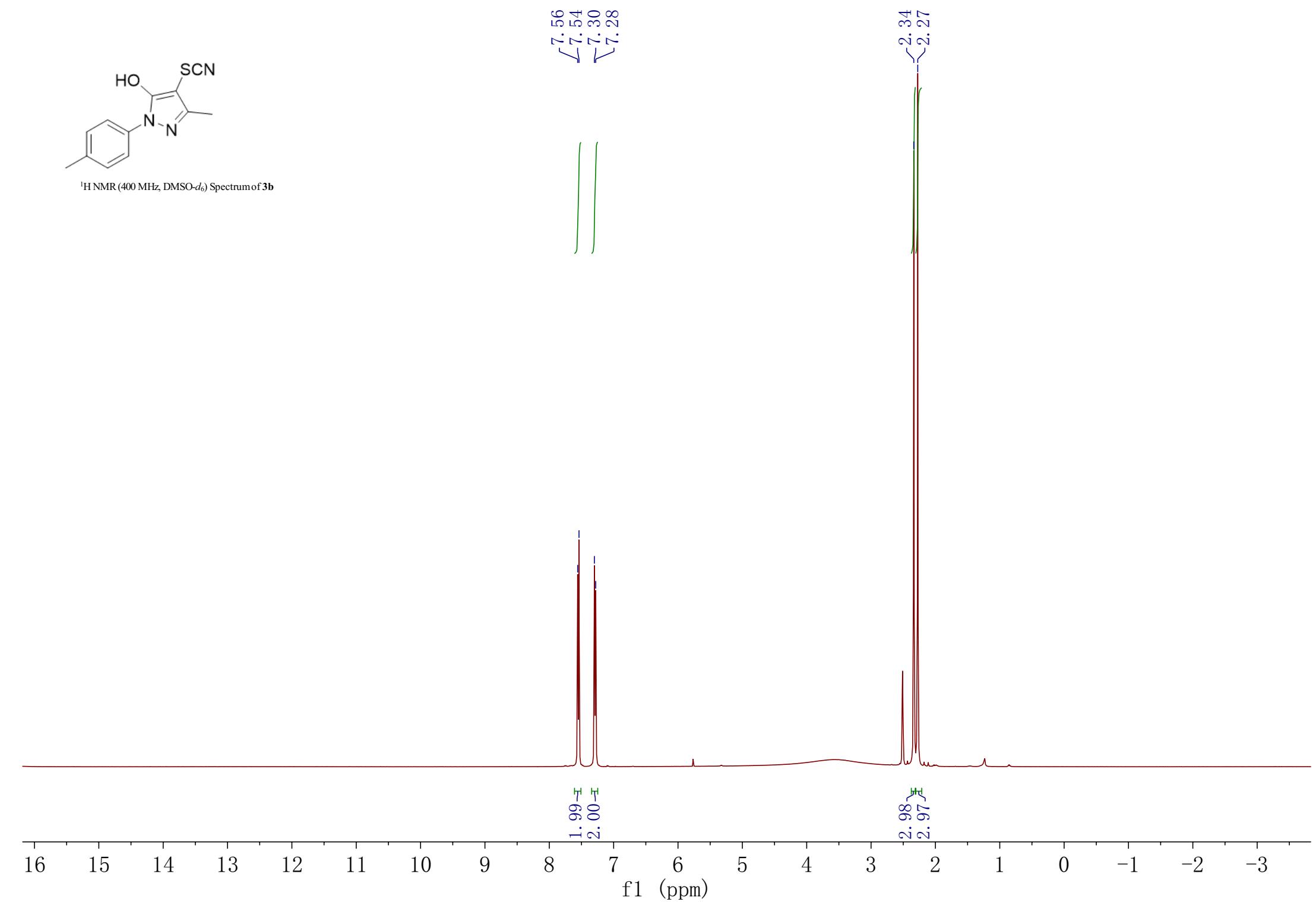


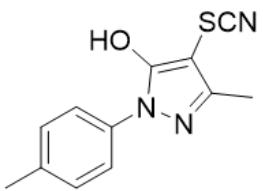
¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of **3a**



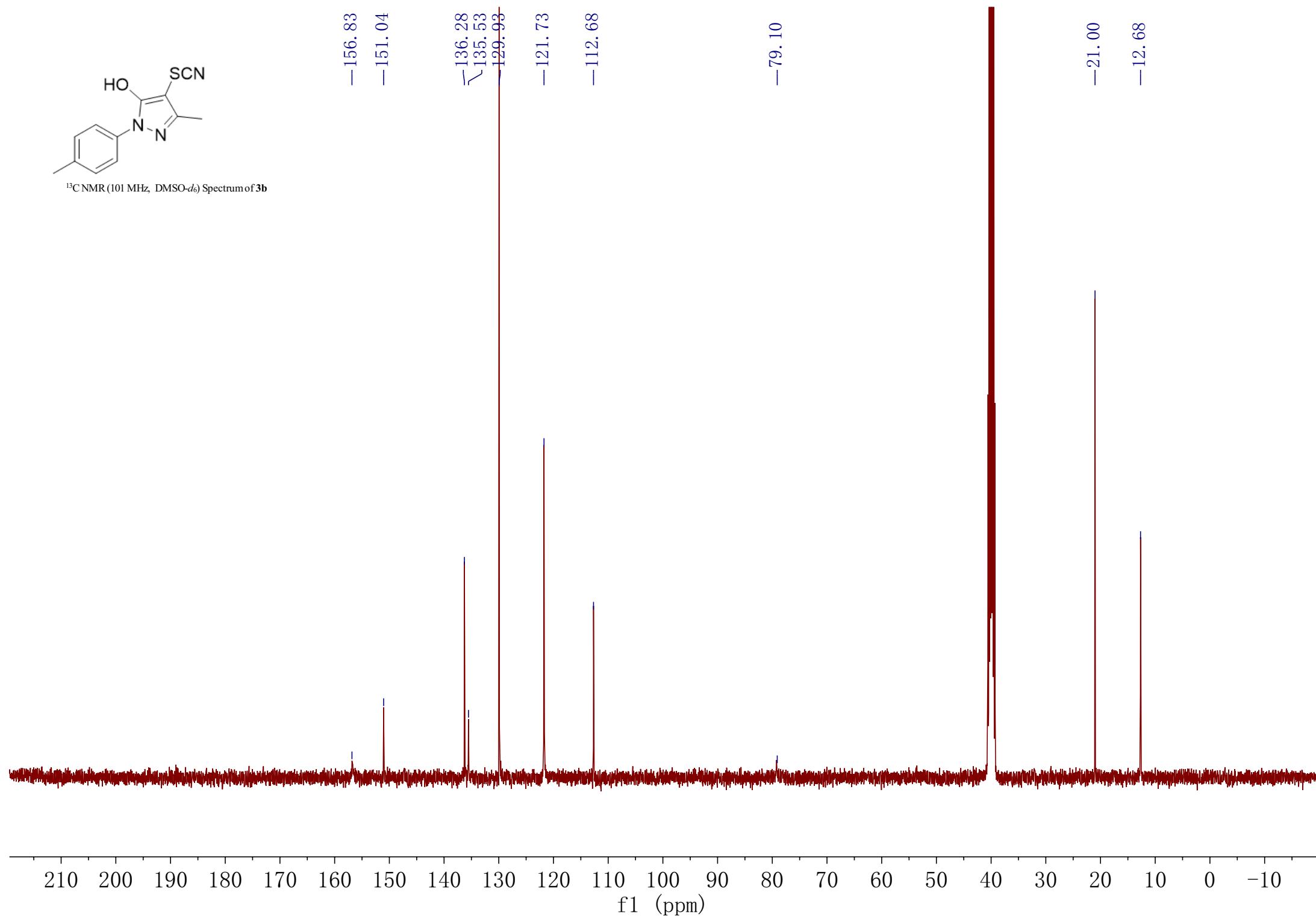


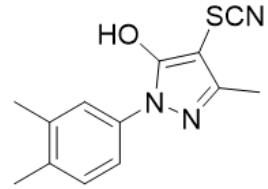
¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of 3b





¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of **3b**





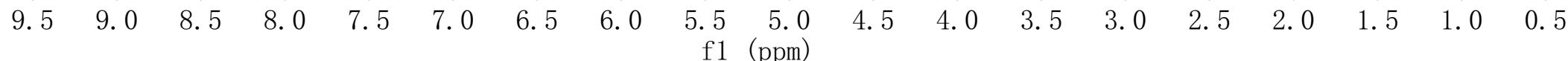
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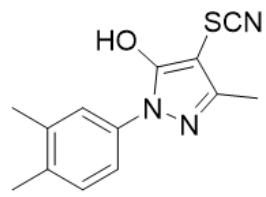
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of 3c

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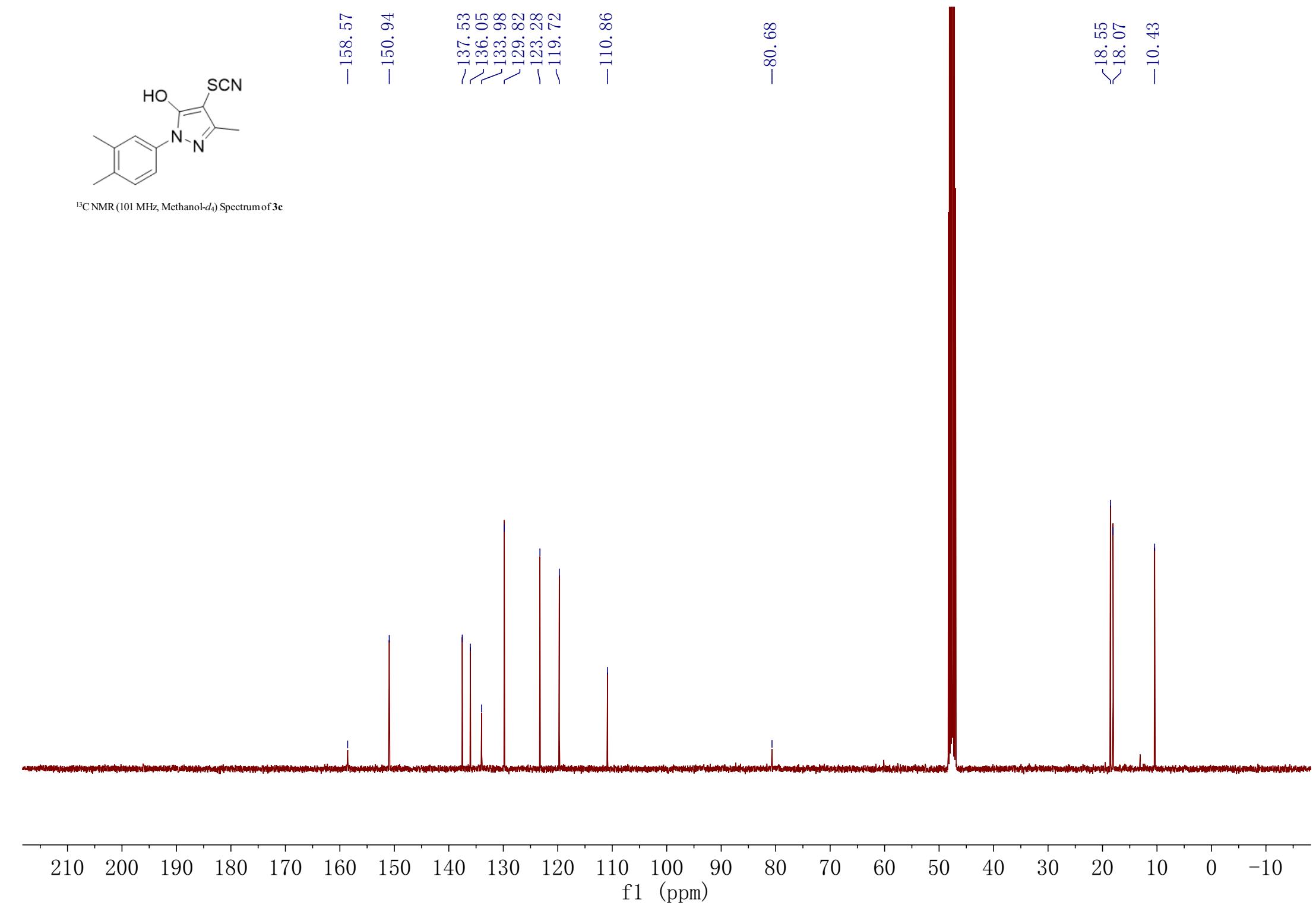
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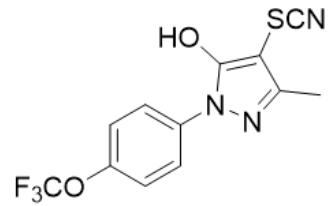
3.00
6.04



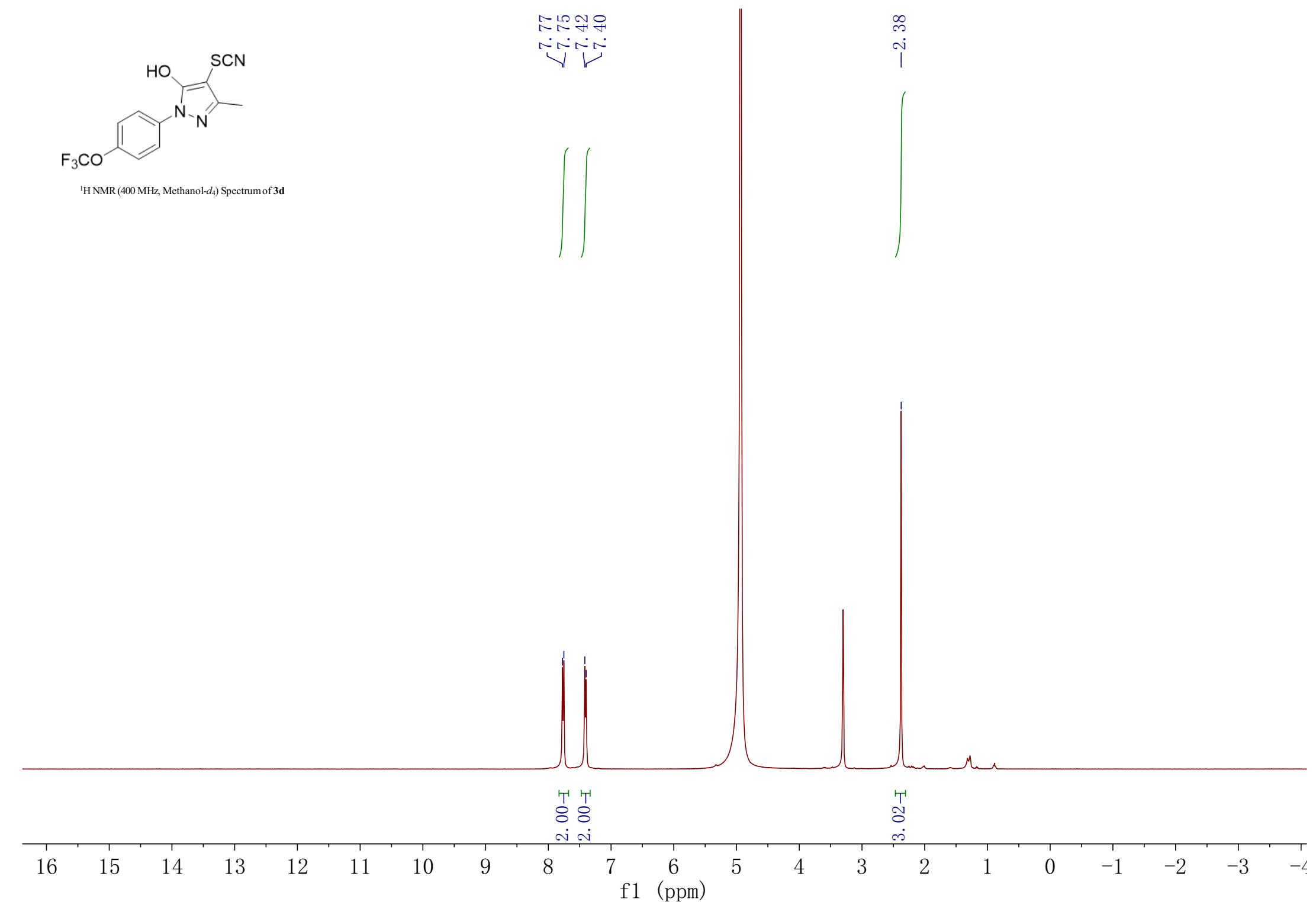


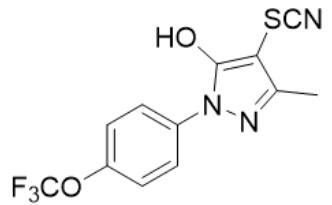
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of 3c



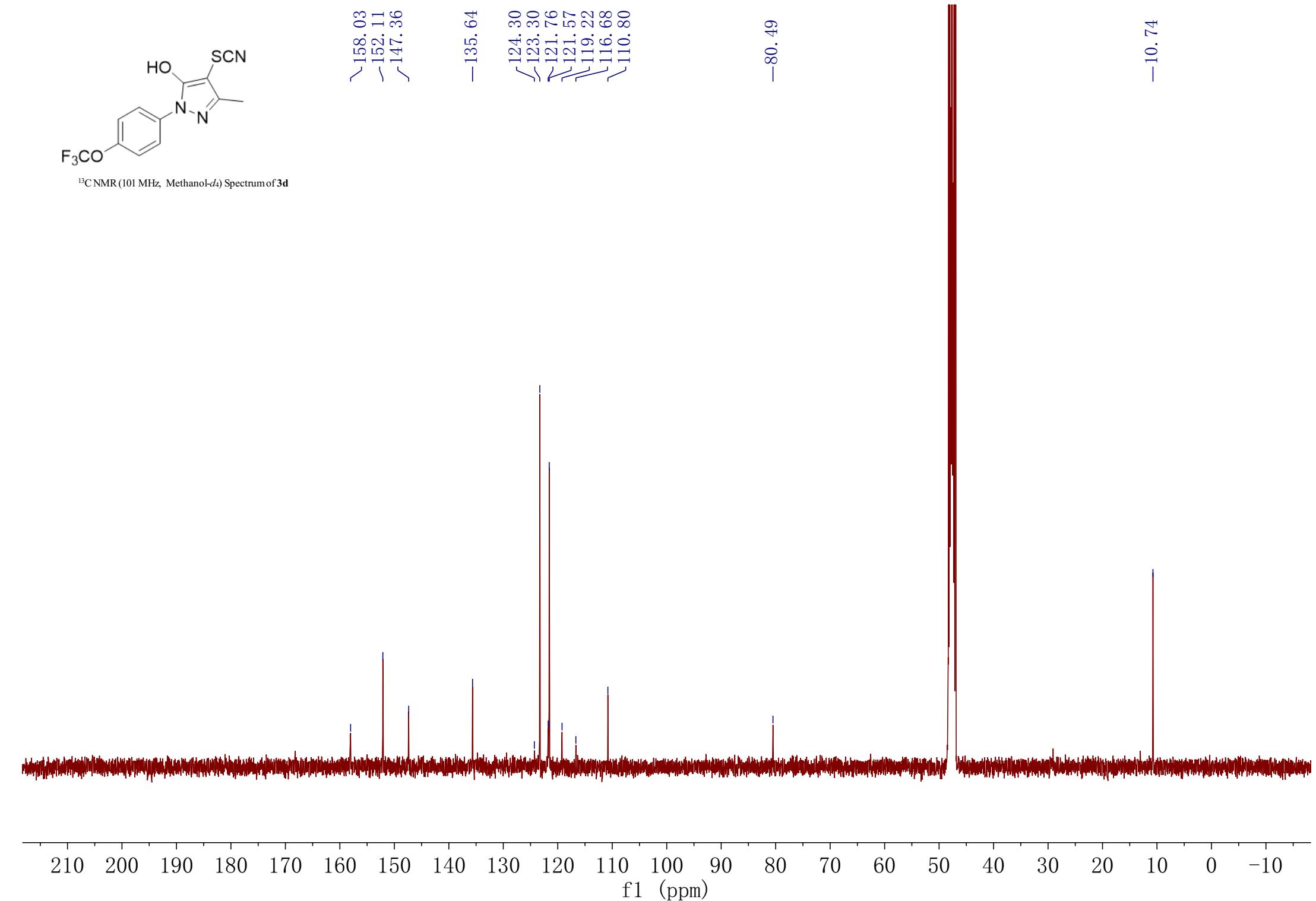


¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of **3d**





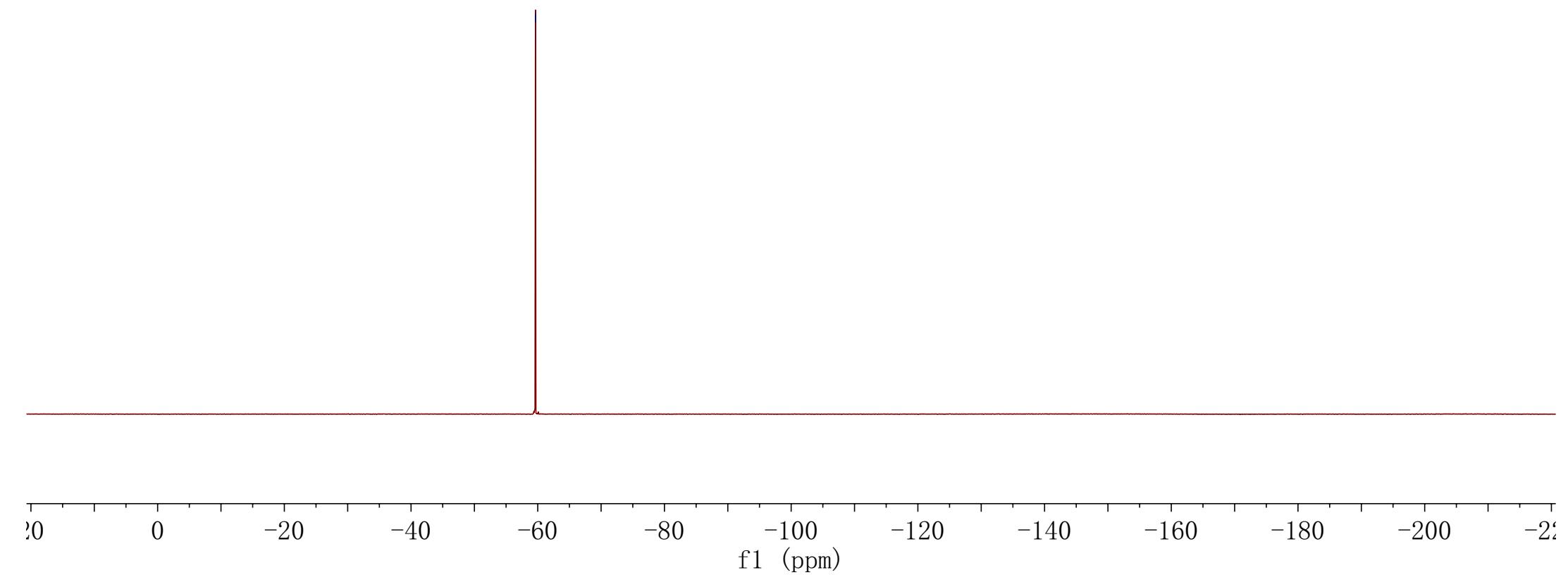
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3d**

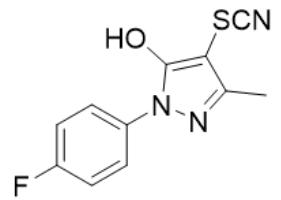




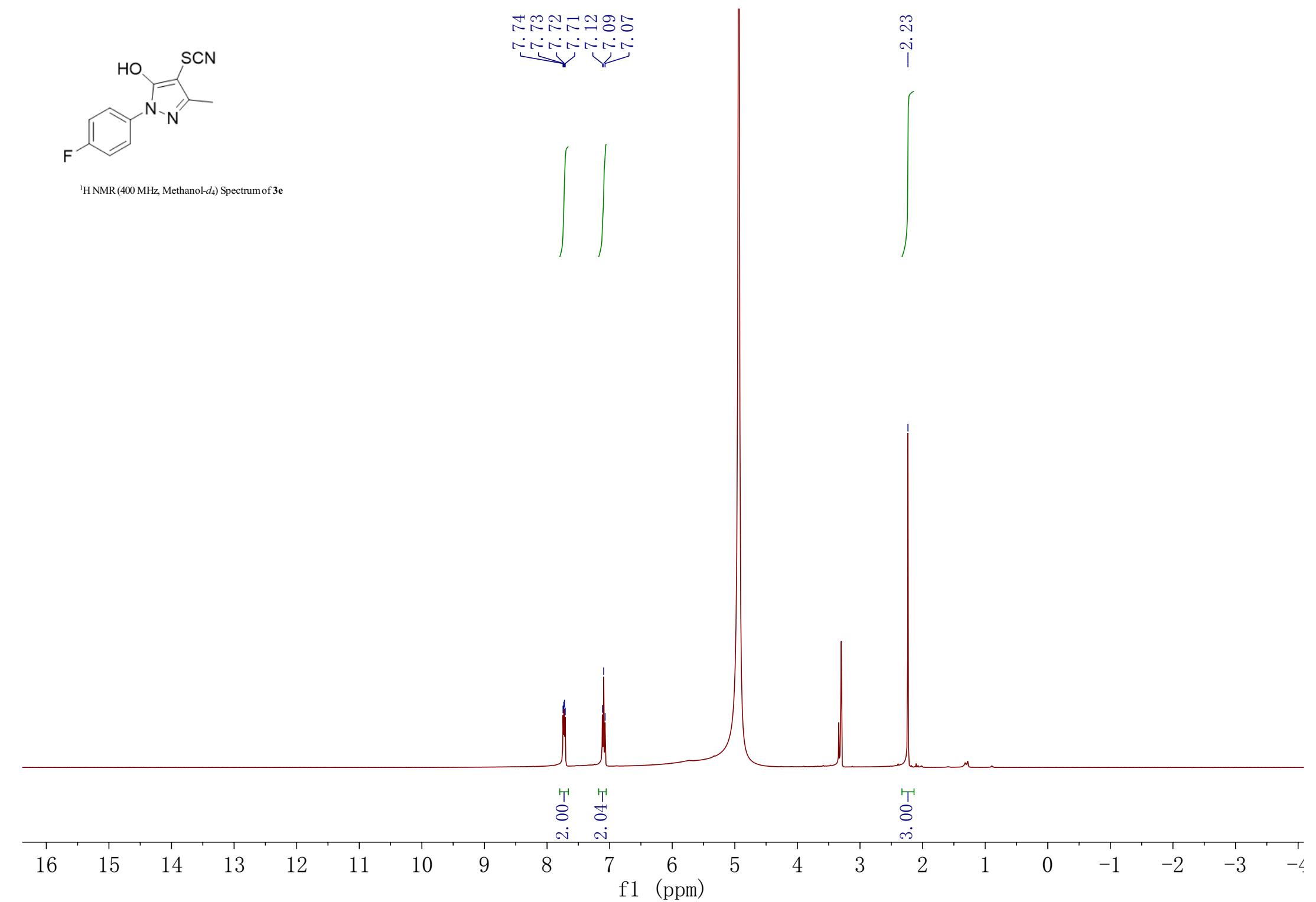
— -59.63

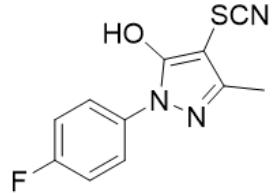
¹⁹F NMR (377 MHz, Methanol-*d*₄) Spectrum of 3d



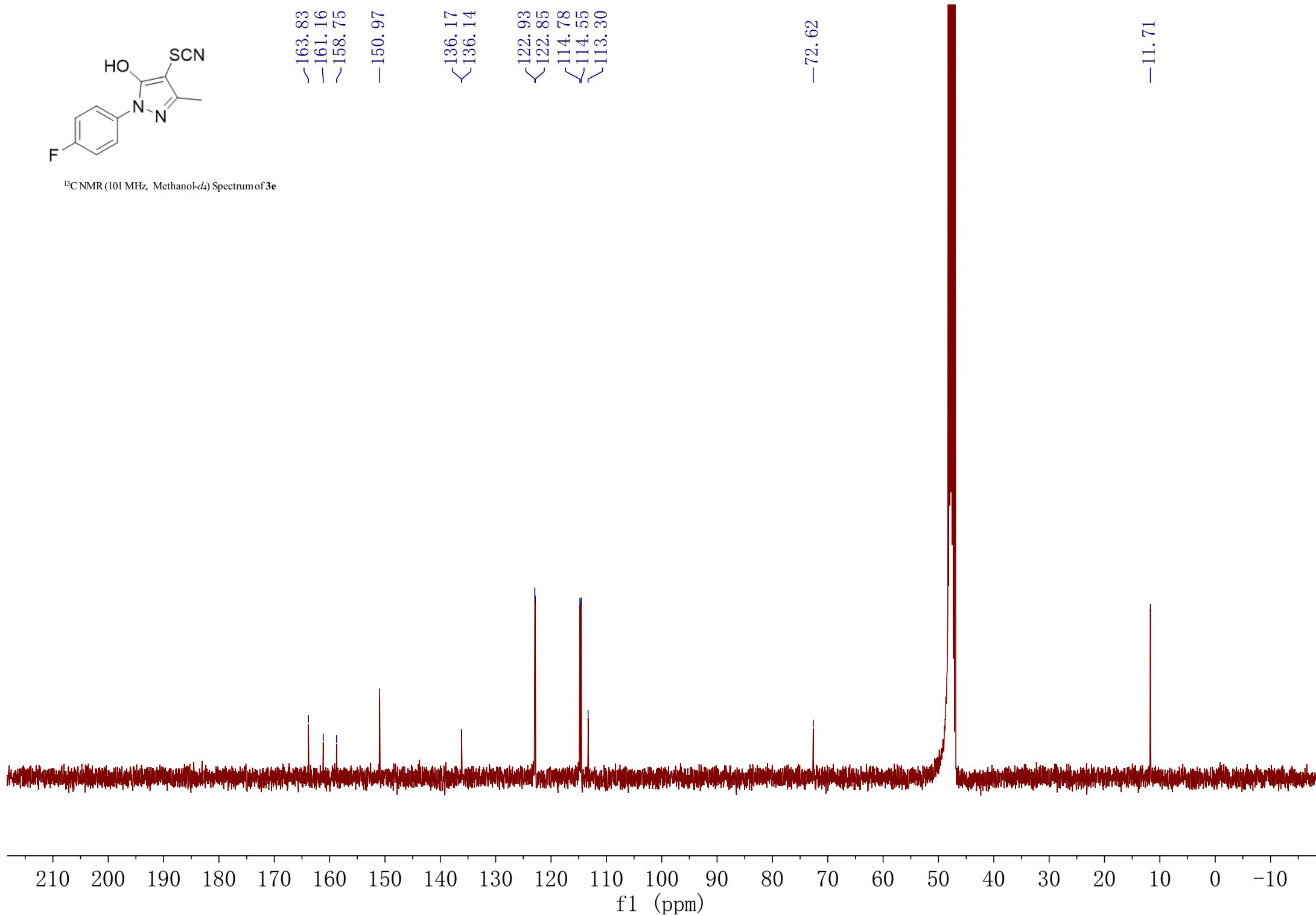


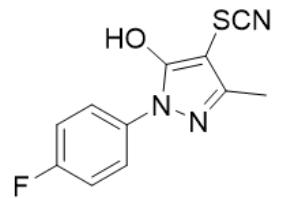
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of 3e



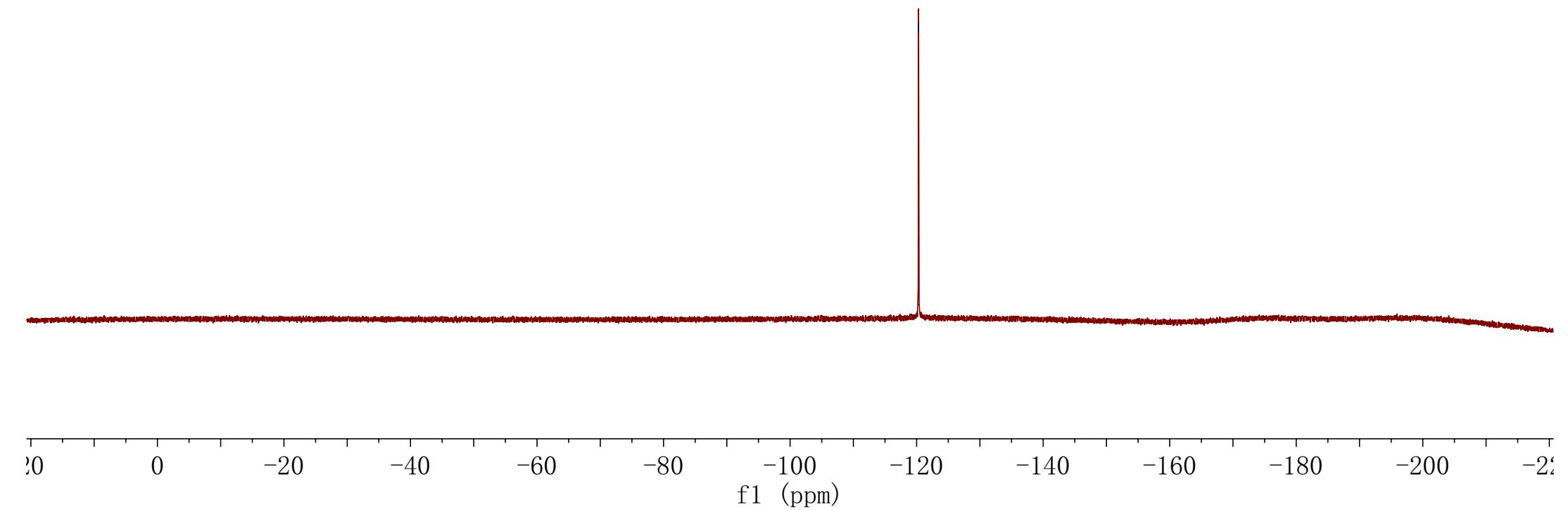


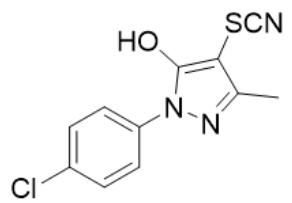
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3e**



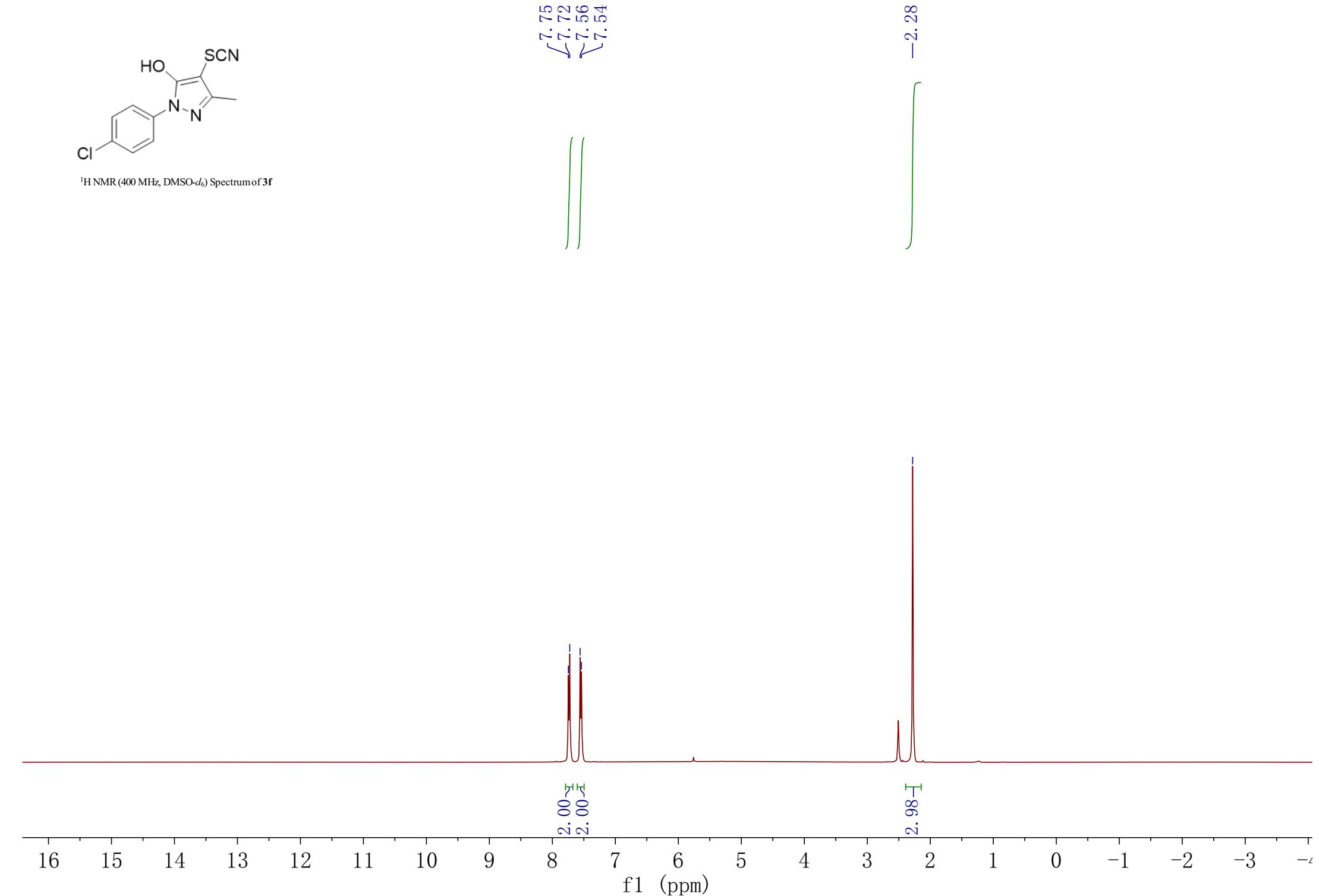


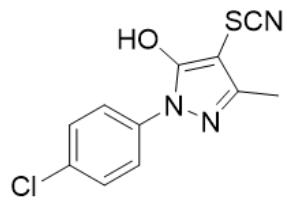
¹⁹F NMR (377 MHz, Methanol-*d*₄) Spectrum of **3e**



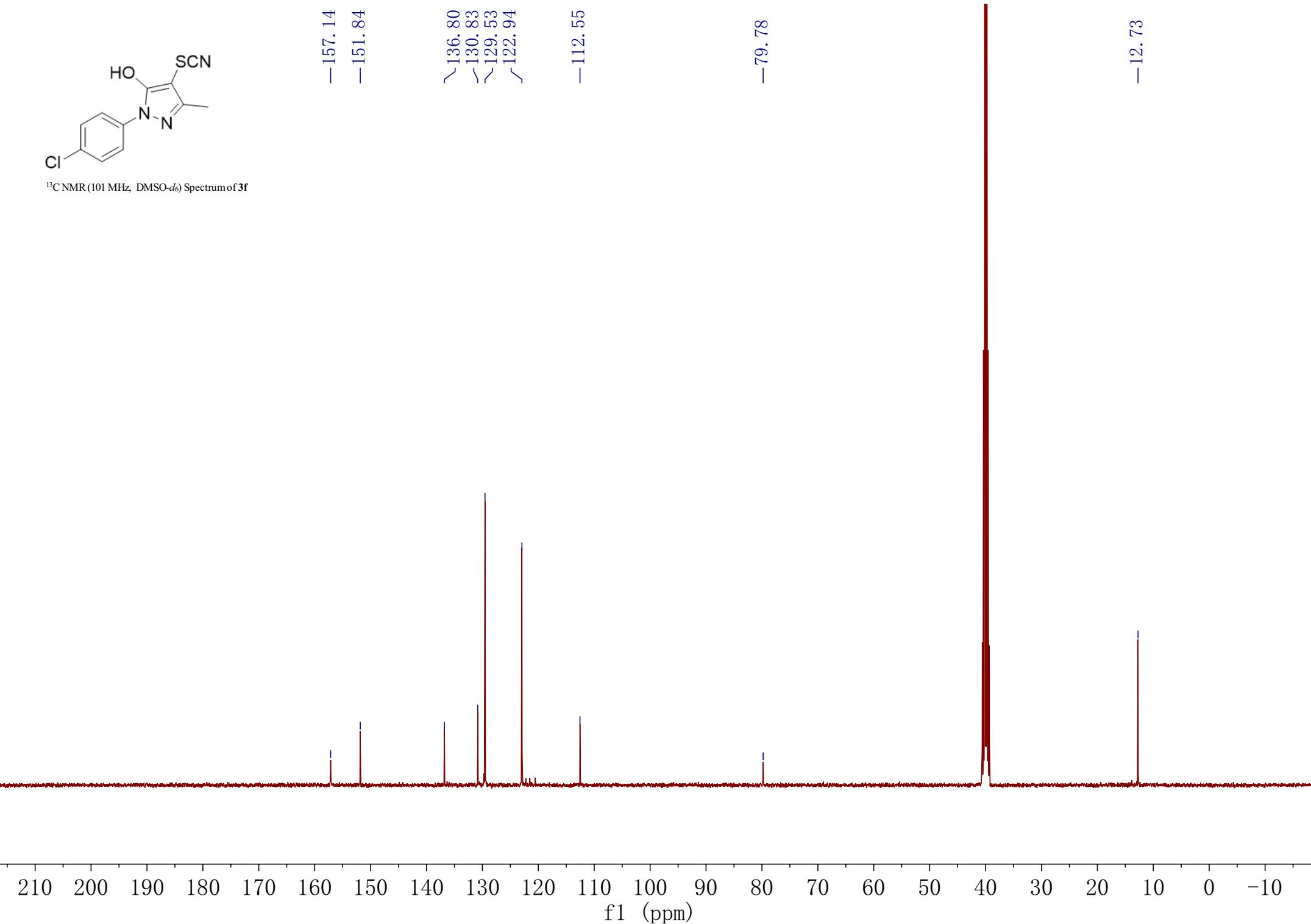


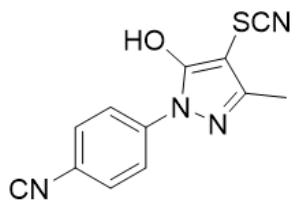
¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of 3f



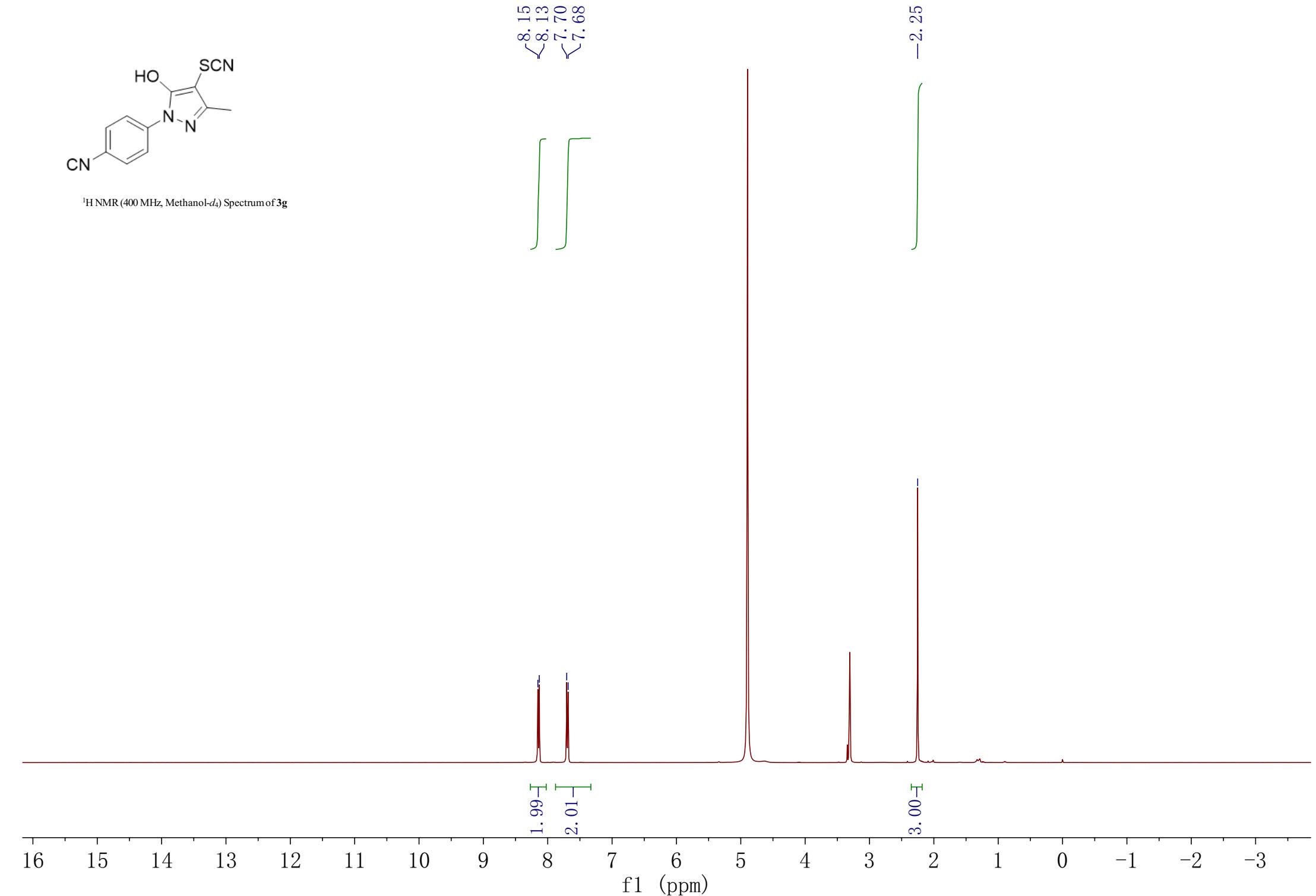


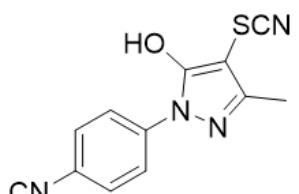
¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of **3f**



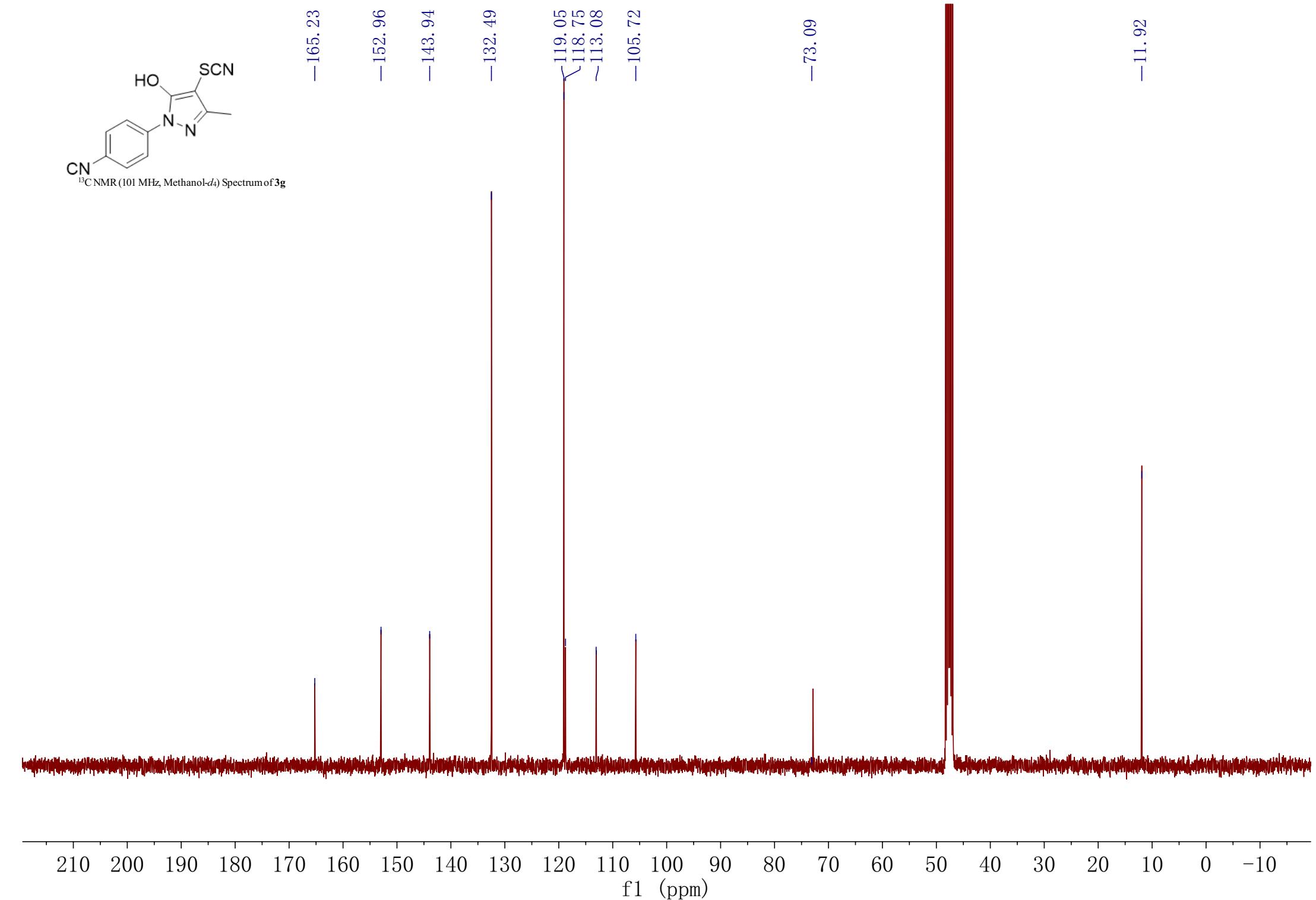


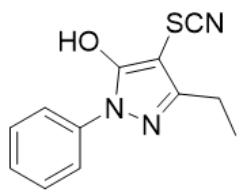
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of 3g



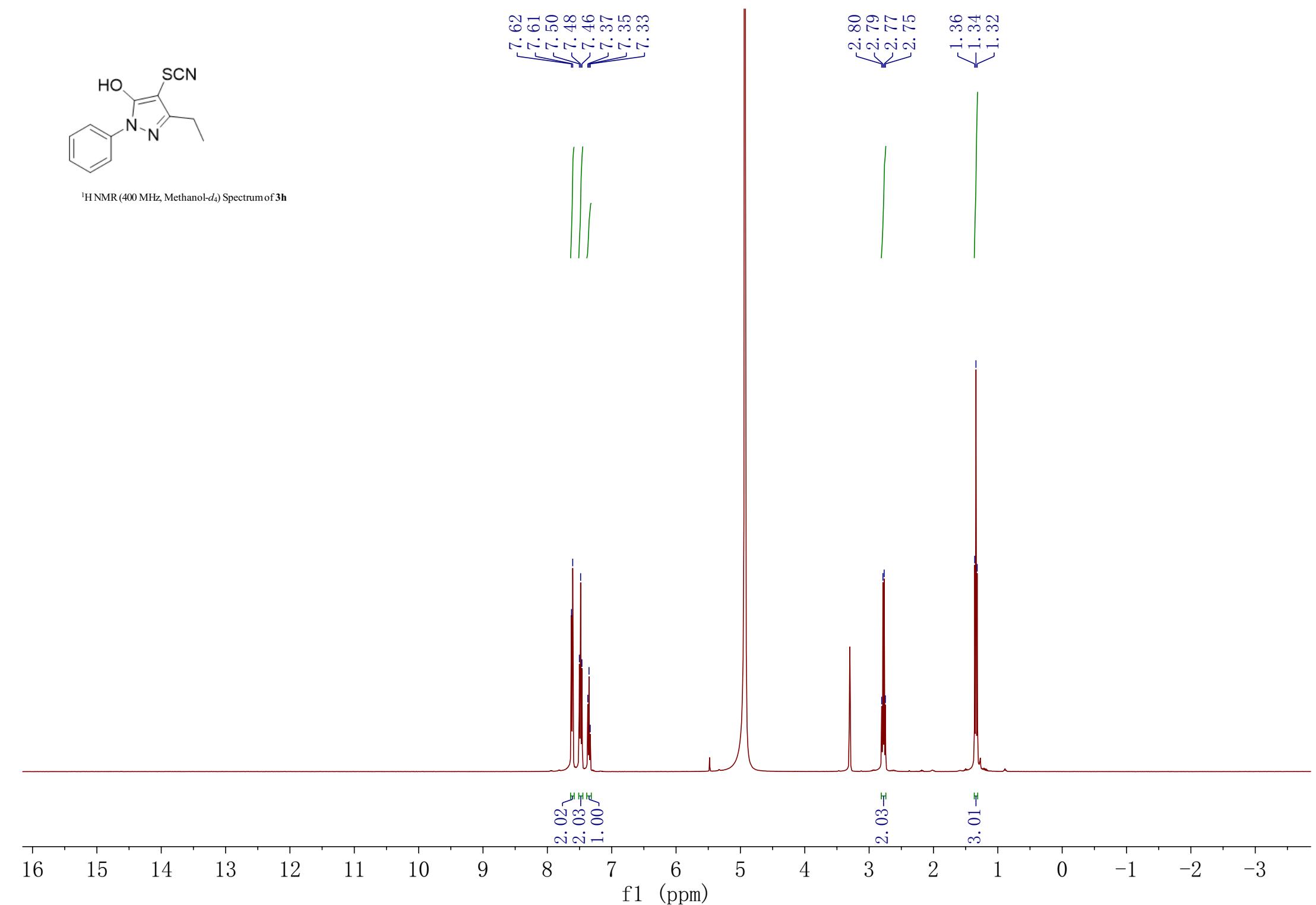


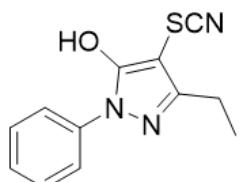
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3g**



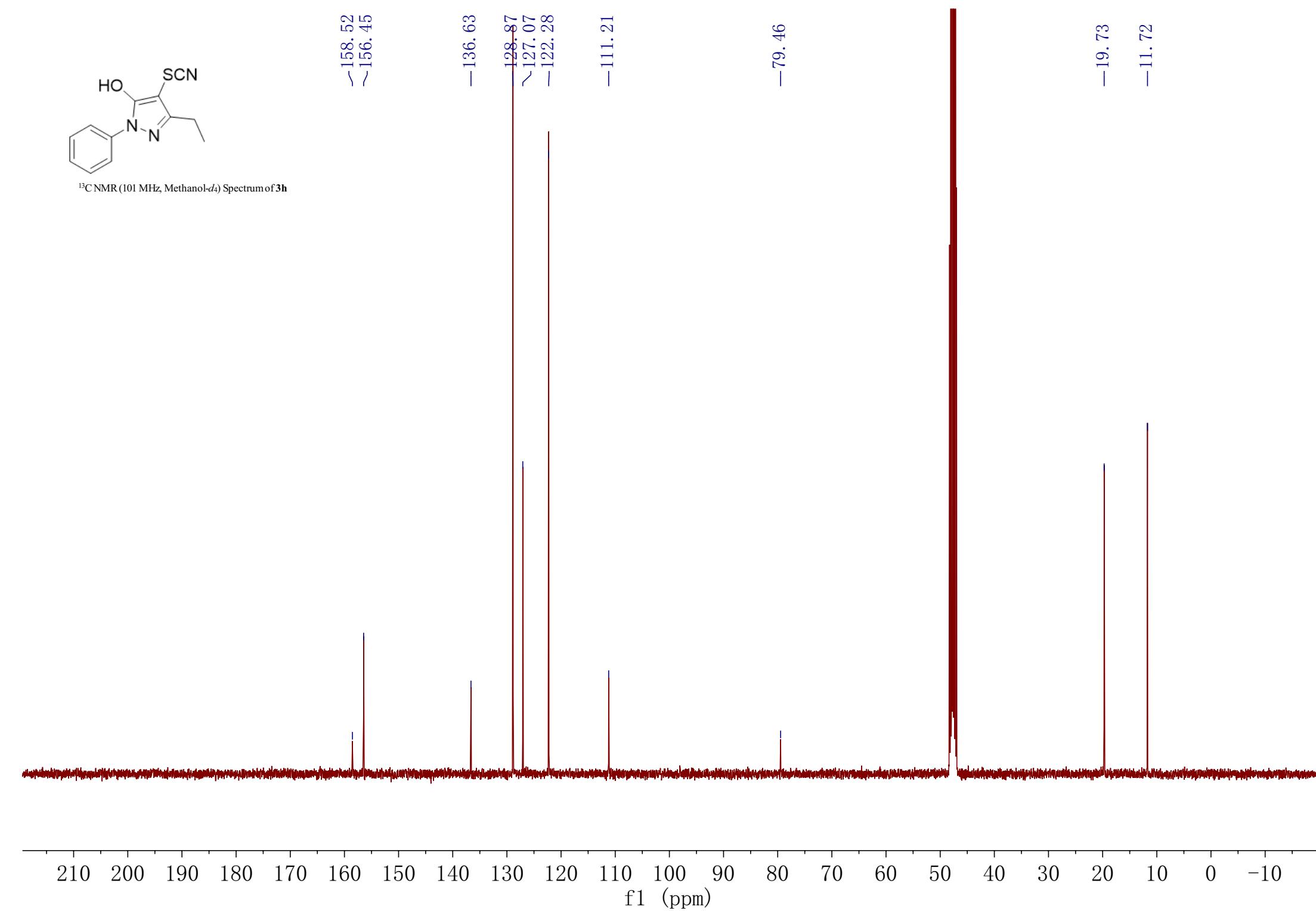


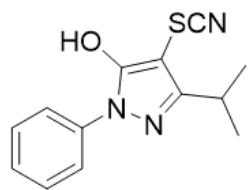
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of **3h**



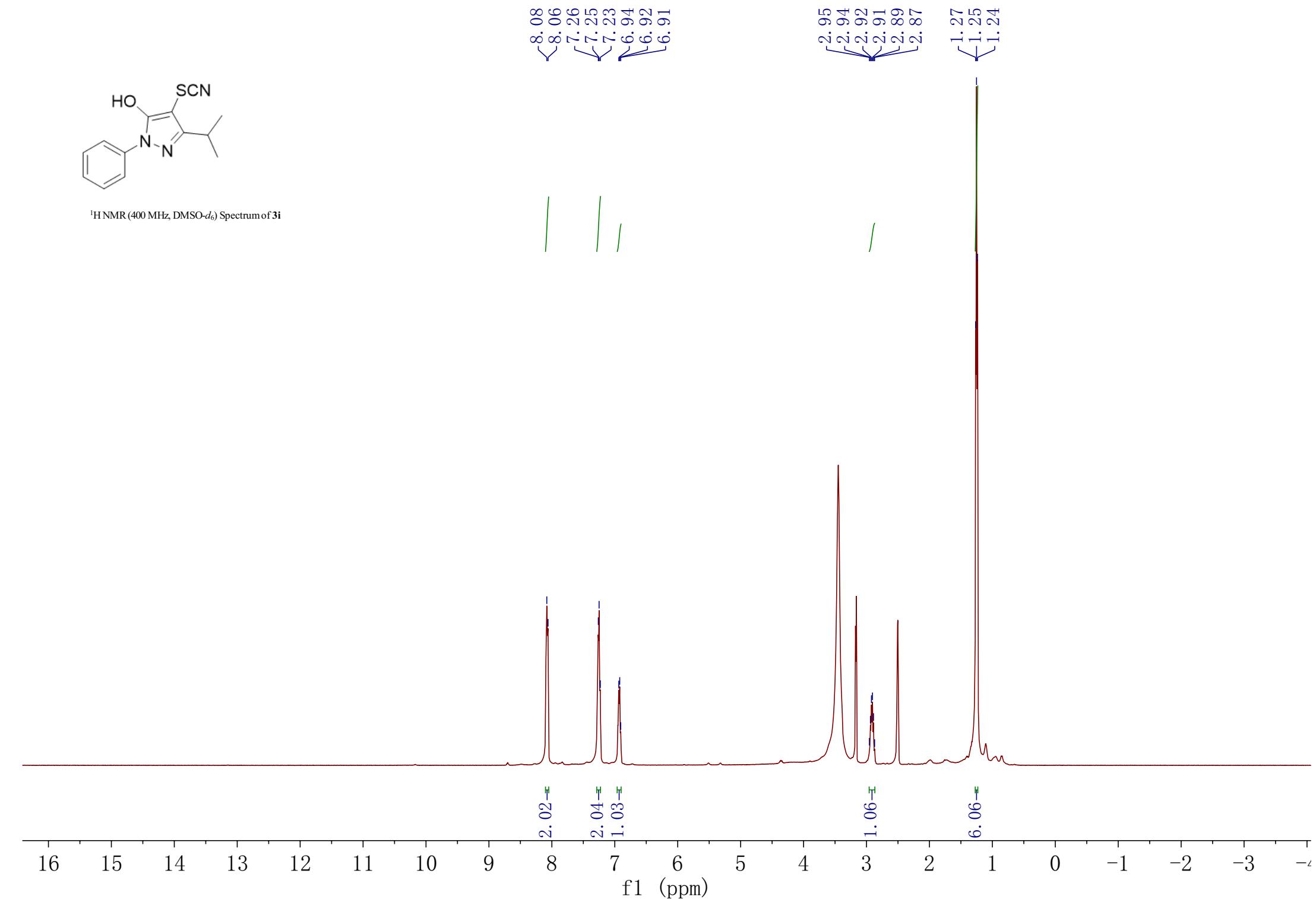


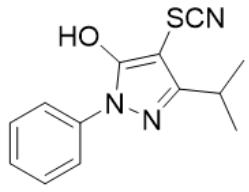
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3h**



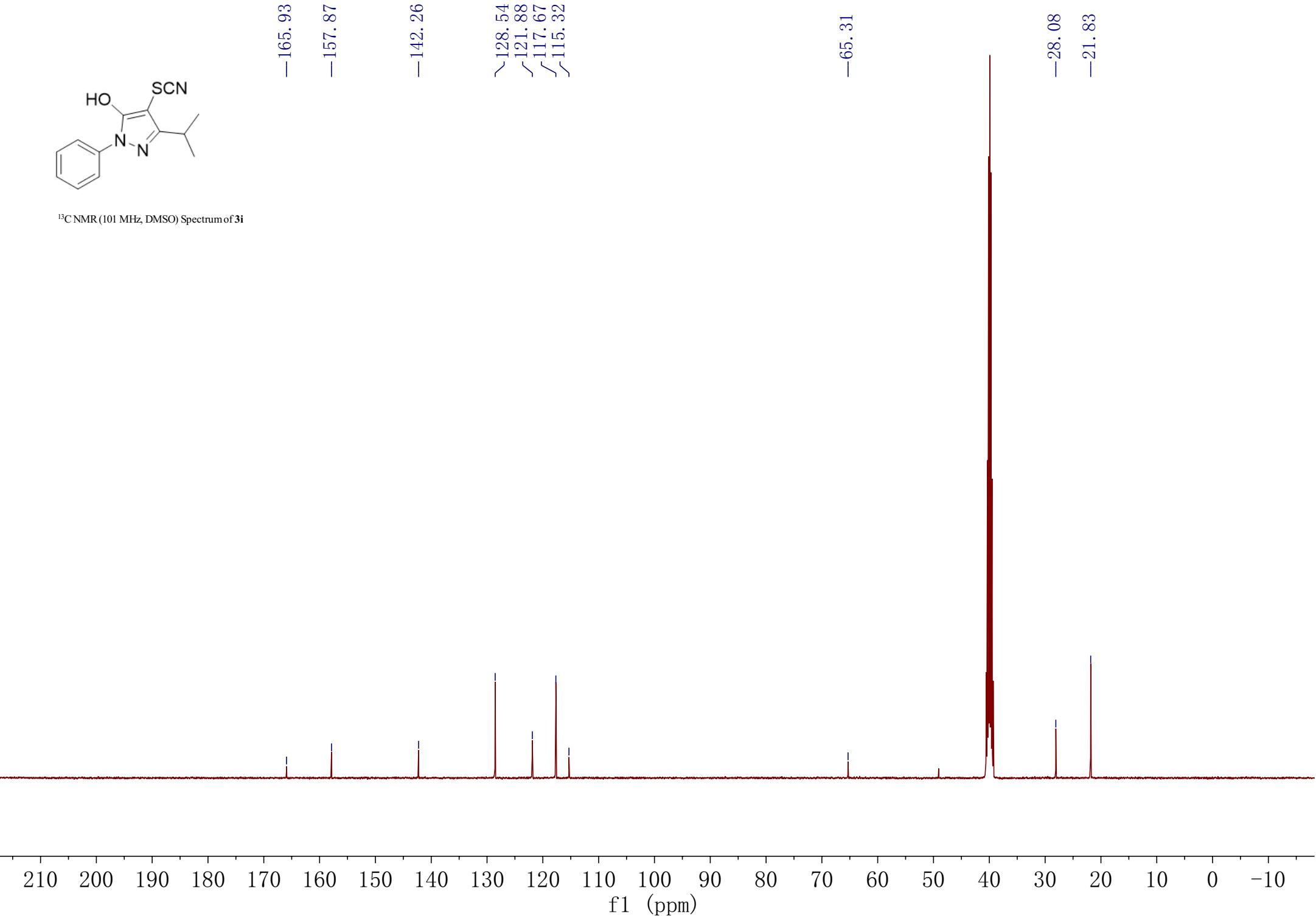


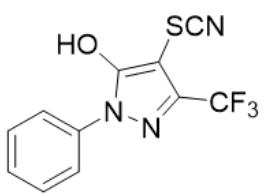
¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of **3i**





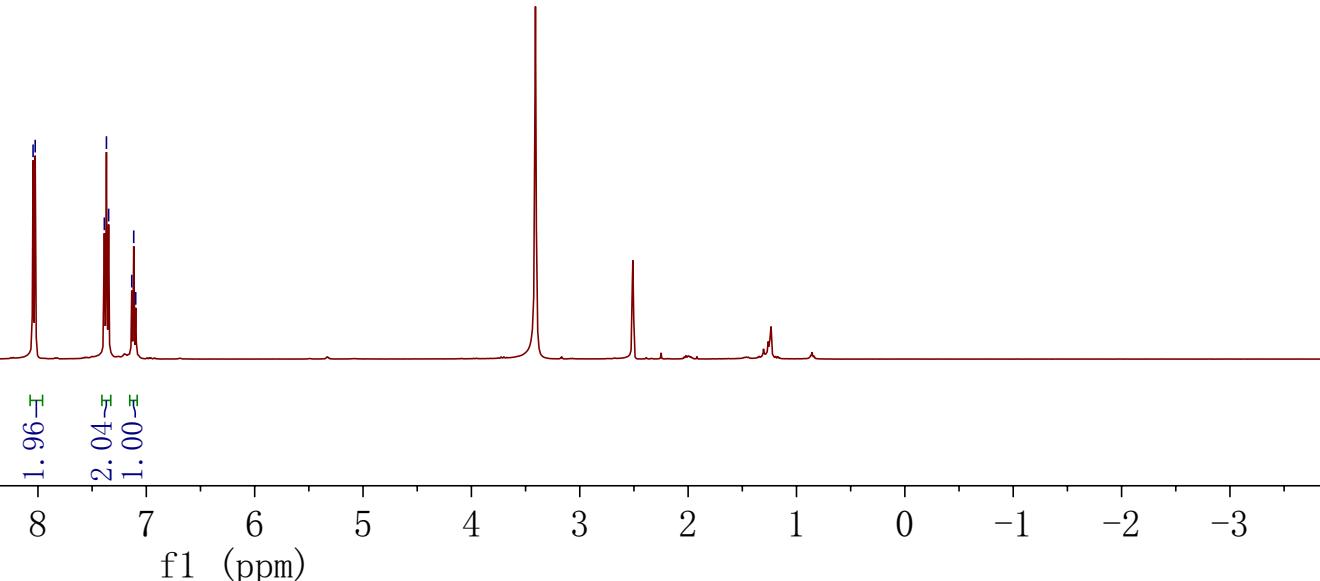
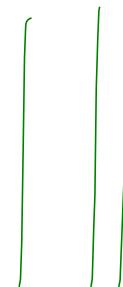
¹³C NMR (101 MHz, DMSO) Spectrum of **3i**

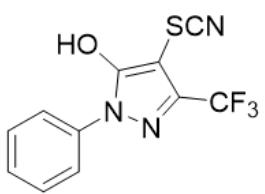




8.05
8.03
7.39
7.37
7.35
7.13
7.12
7.10

^1H NMR (400 MHz, $\text{DMSO}-d_6$) Spectrum of **3j**





-164.76

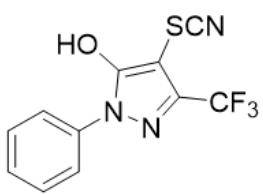
<141.00
<140.72
128.93
125.98
124.17
123.30
120.62
118.97
117.94
113.91

-66.33

^{13}C NMR (101 MHz, DMSO- d_6) Spectrum of **3j**

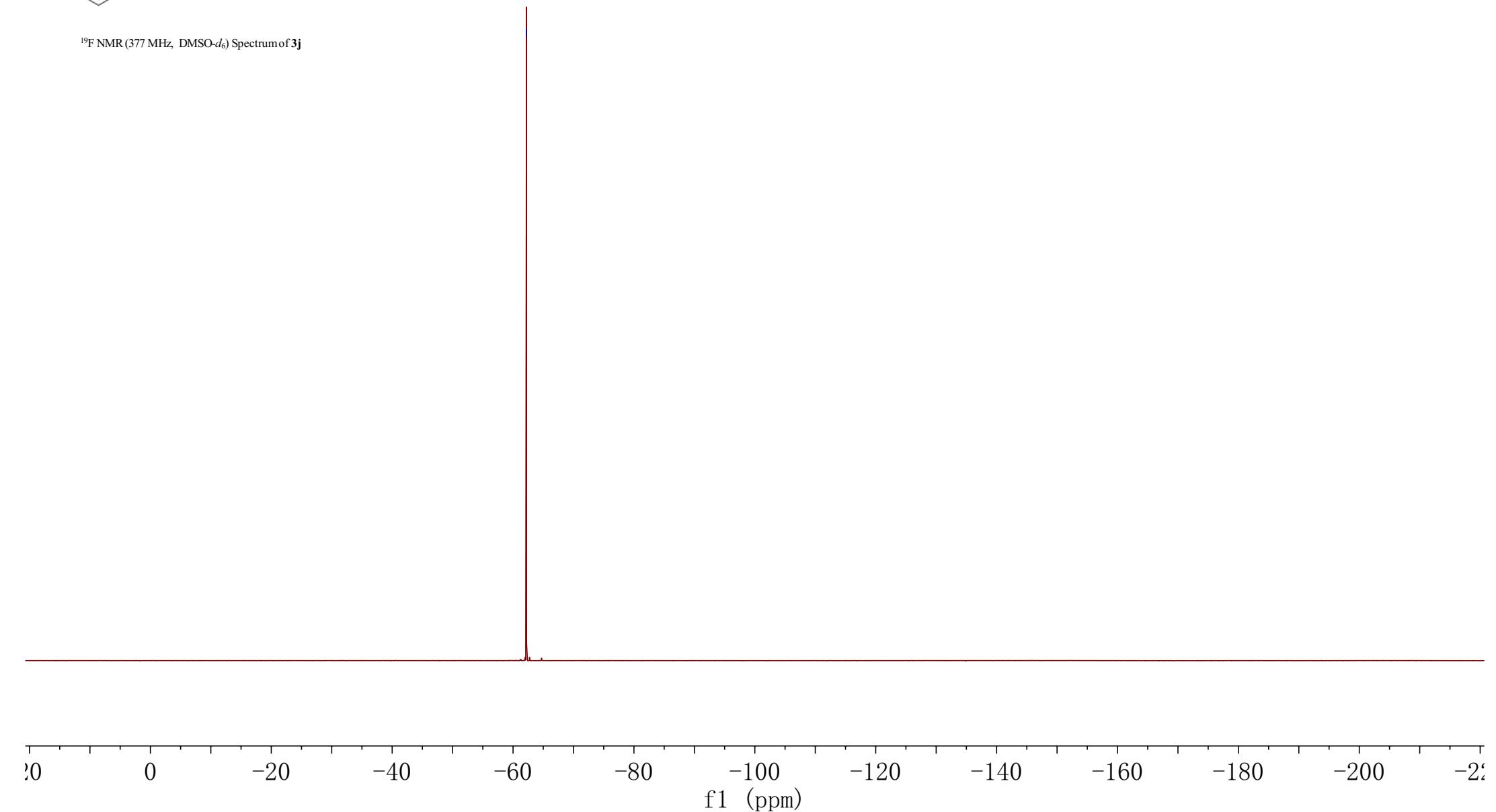
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

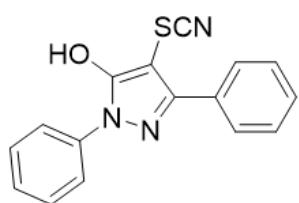
f1 (ppm)



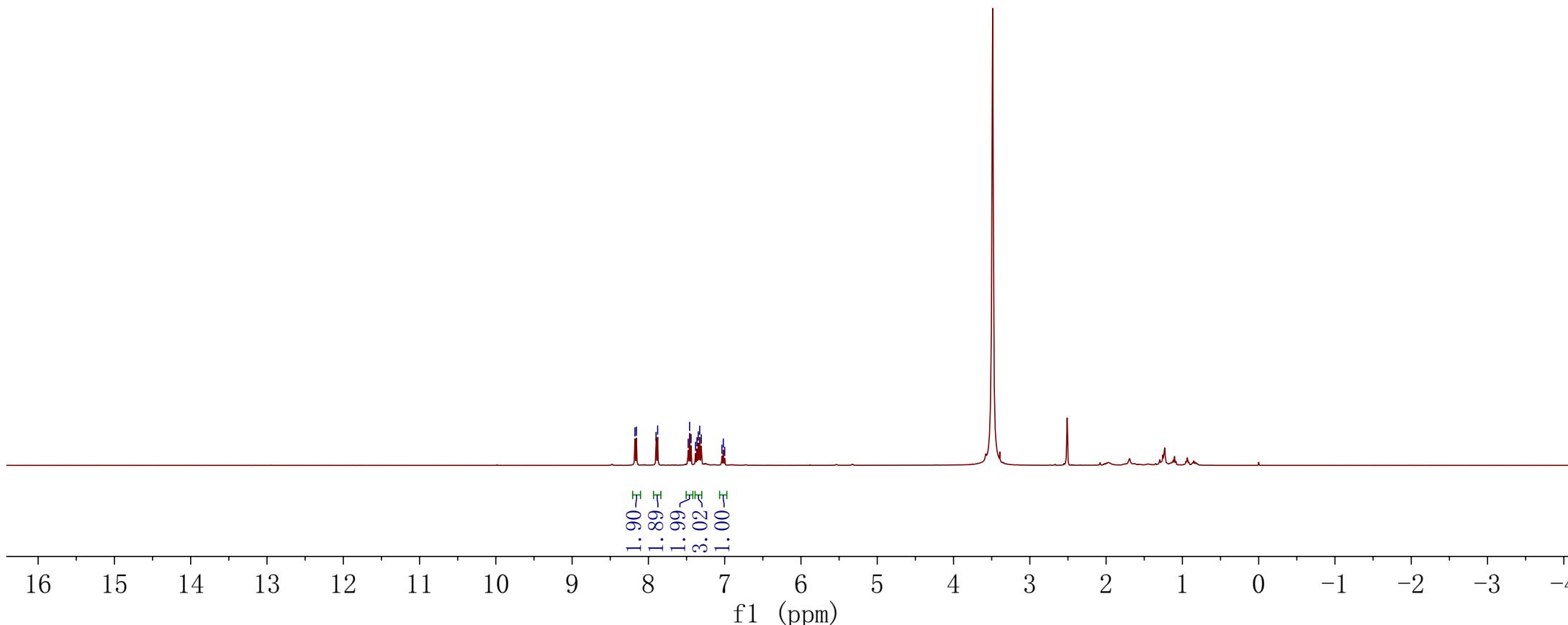
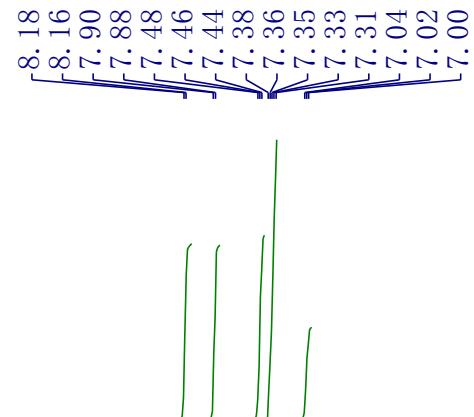
-62.23

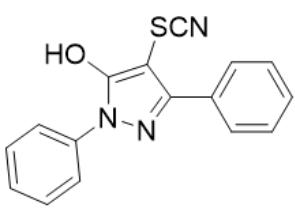
^{19}F NMR (377 MHz, DMSO- d_6) Spectrum of **3j**



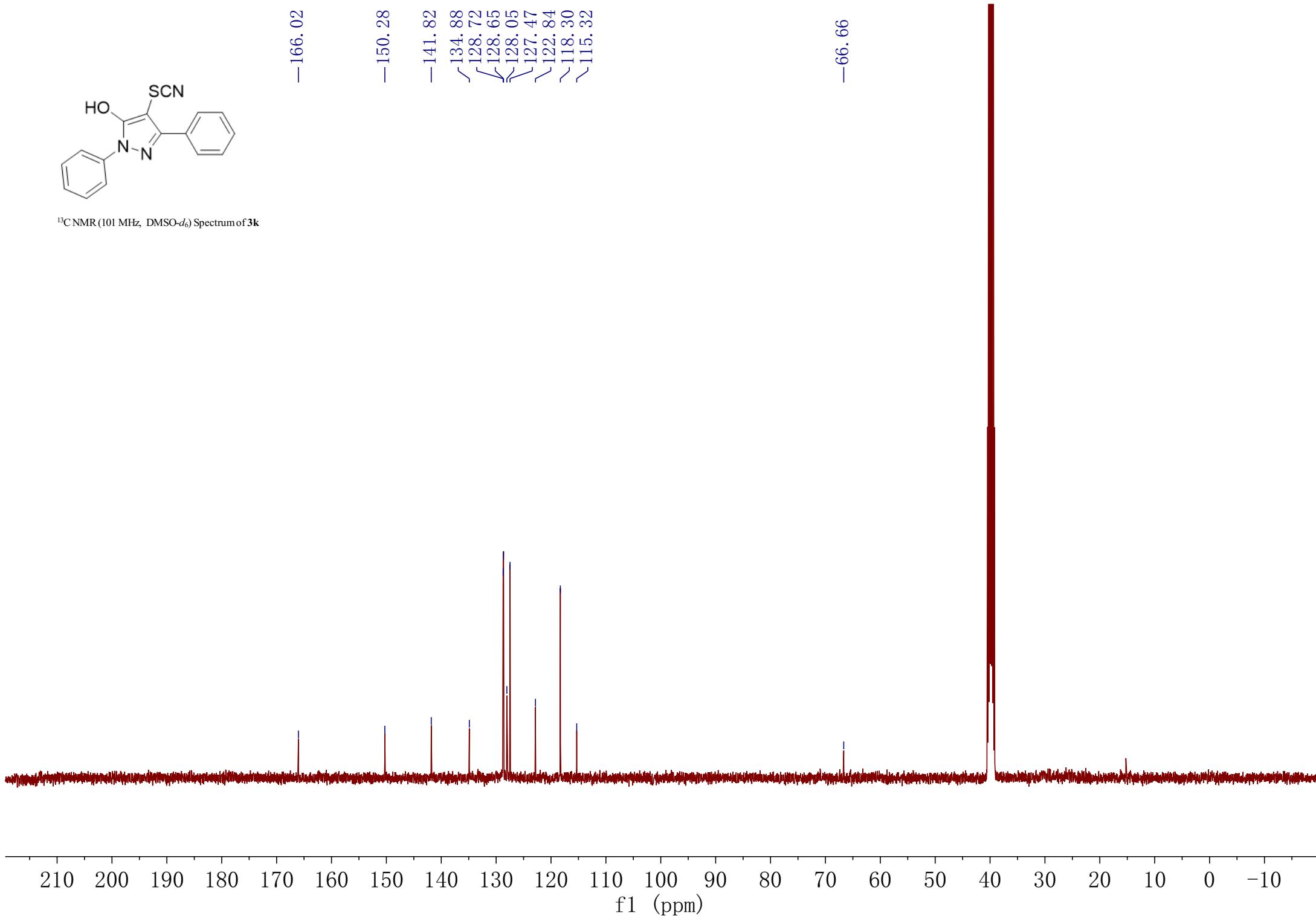


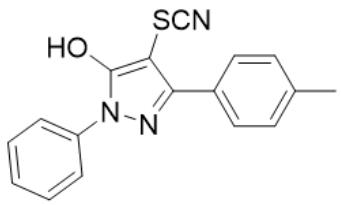
¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of **3k**



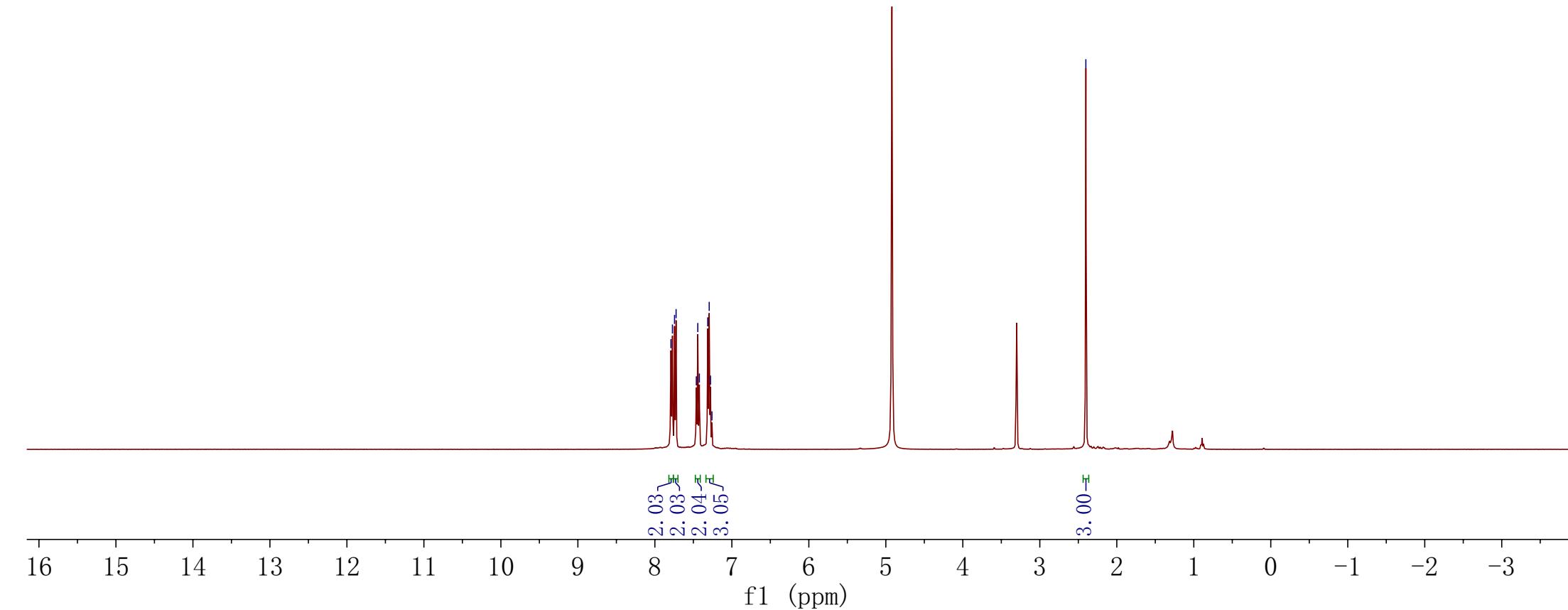
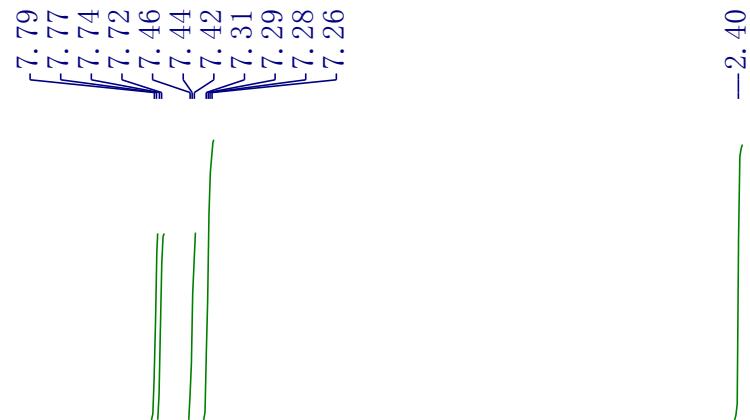


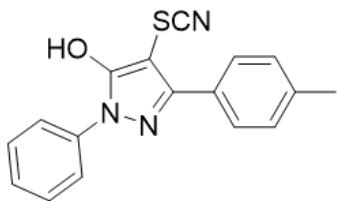
¹³C NMR (101 MHz, DMSO-*d*₆) Spectrum of **3k**



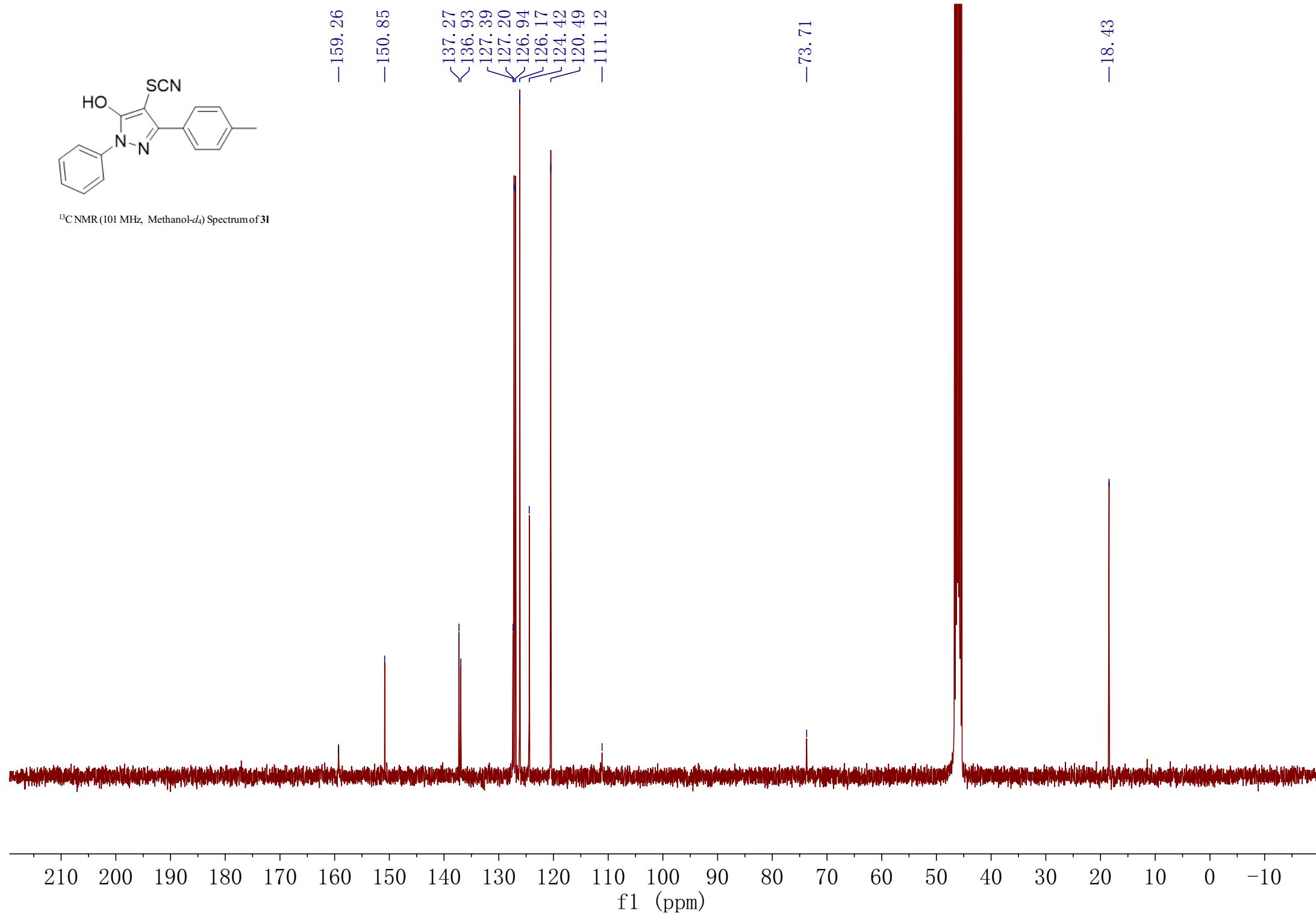


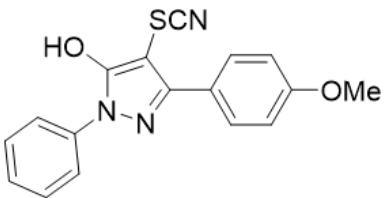
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of 3l



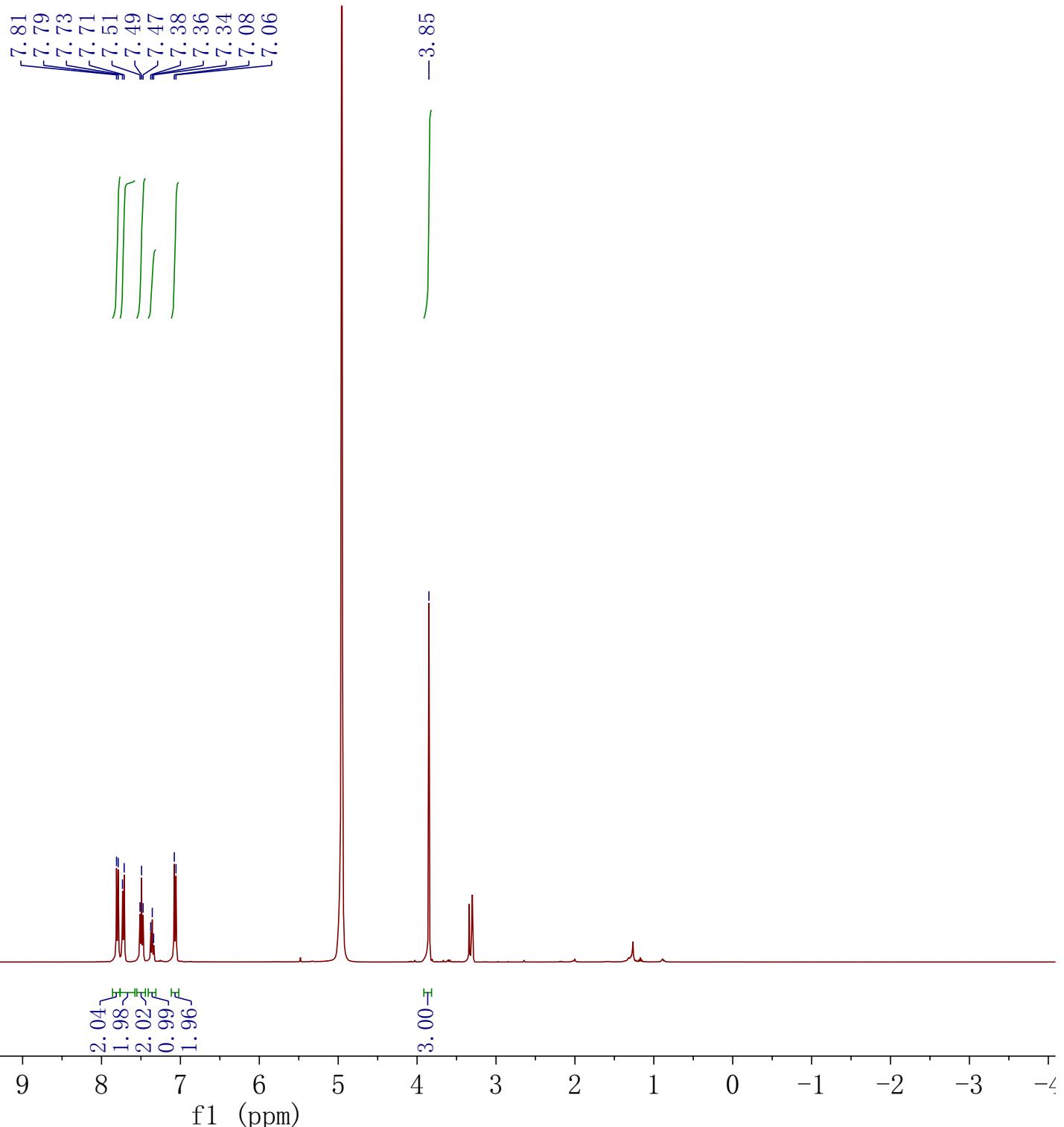


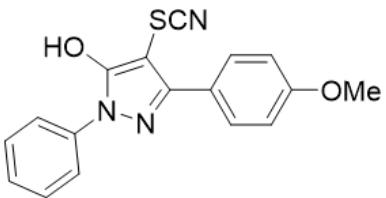
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3l**





¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of **3m**





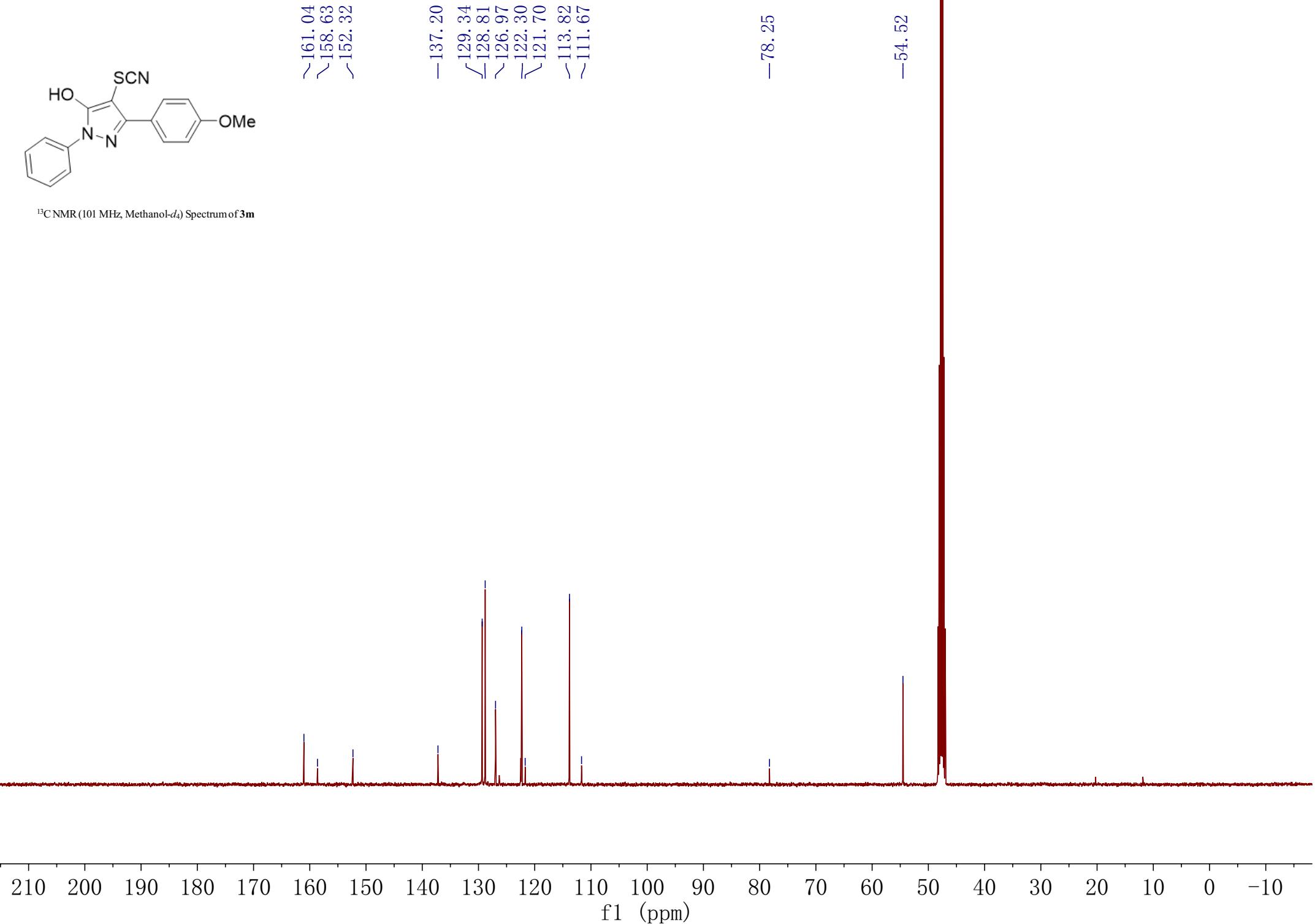
~161.04
~158.63
~152.32

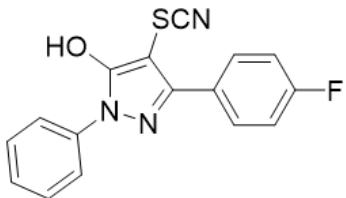
-137.20
129.34
128.81
126.97
122.30
121.70
-113.82
-111.67

-78.25

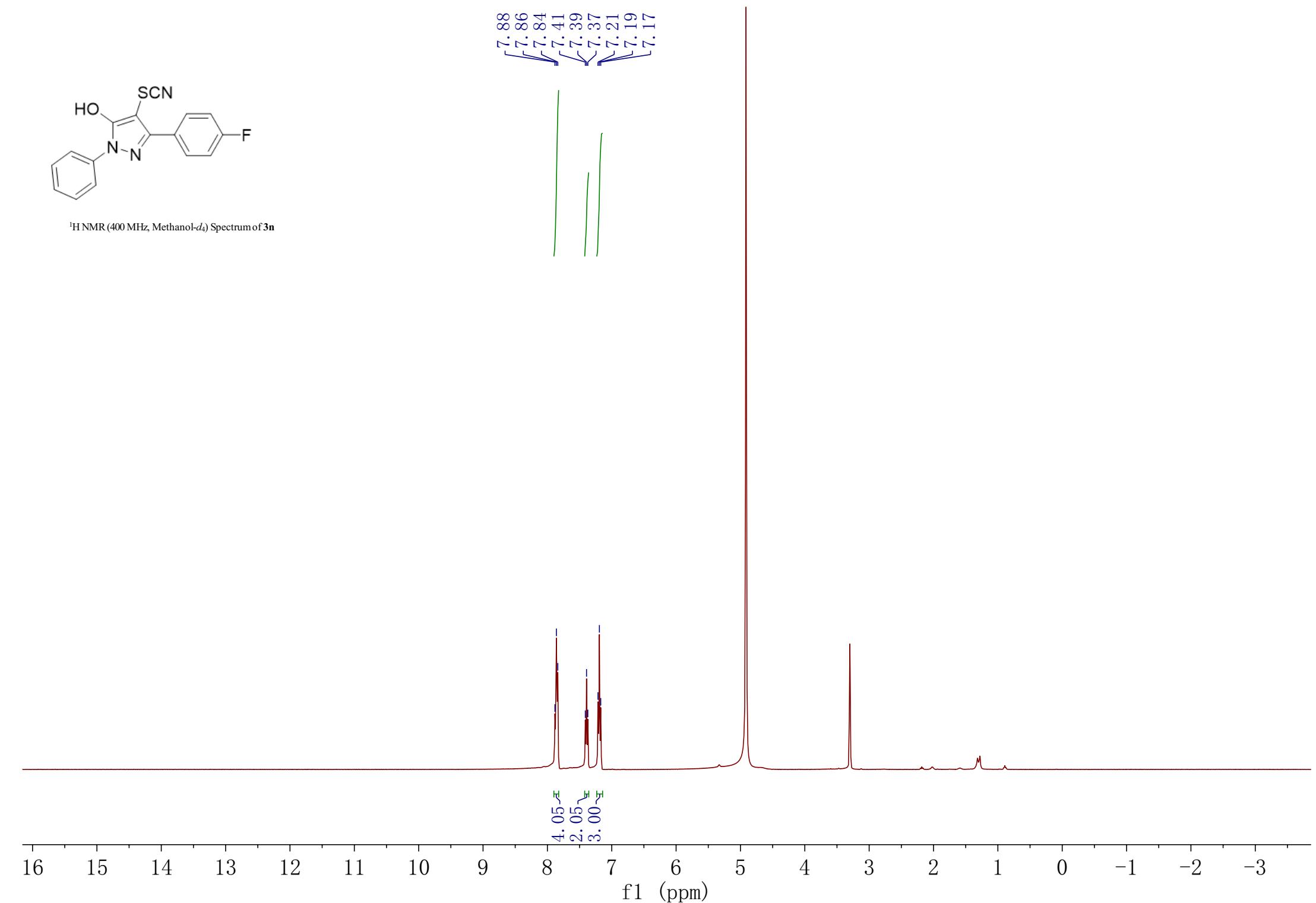
-54.52

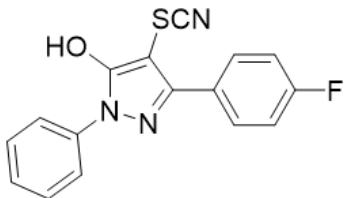
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of **3m**



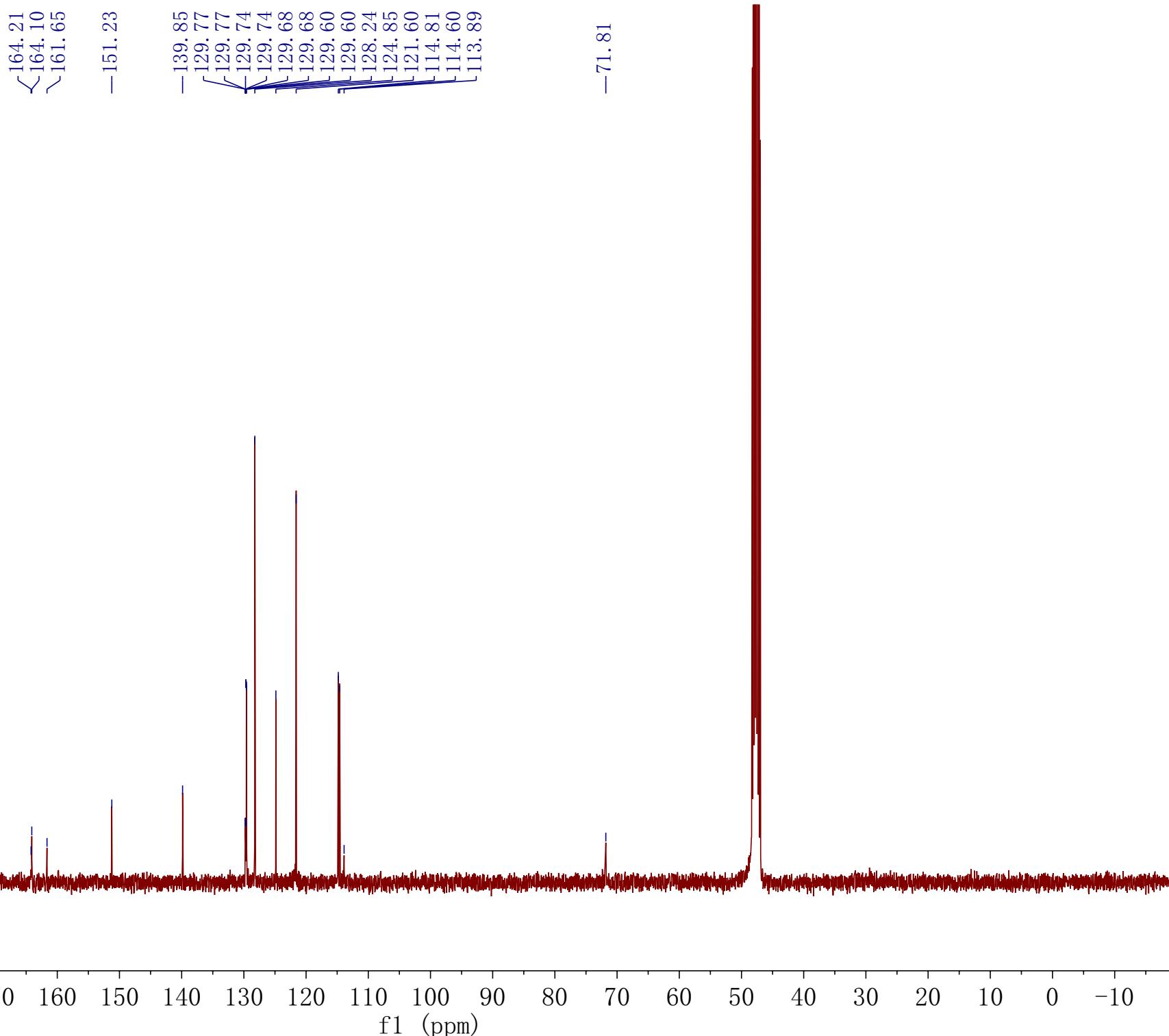


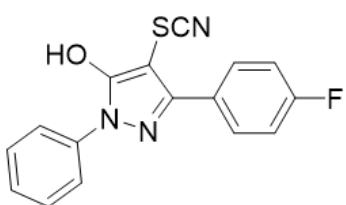
¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of **3n**



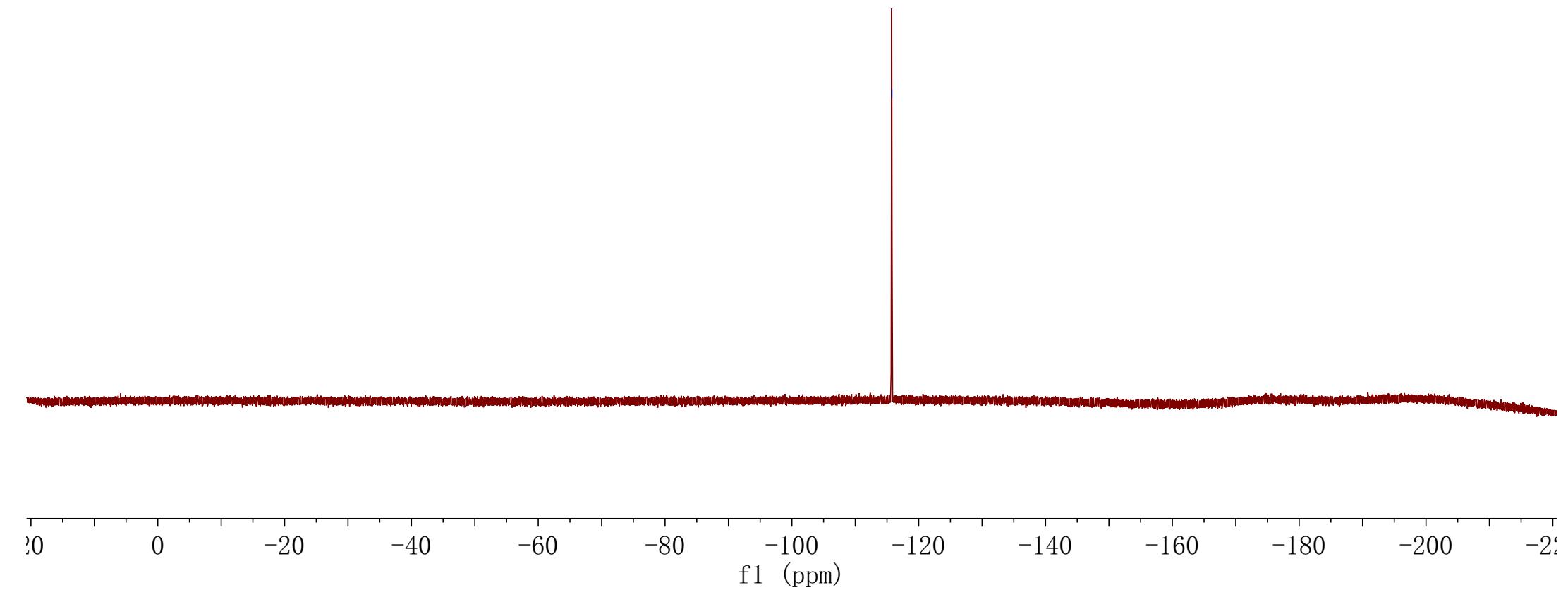


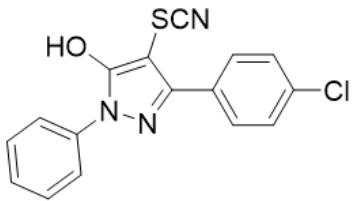
¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of 3n



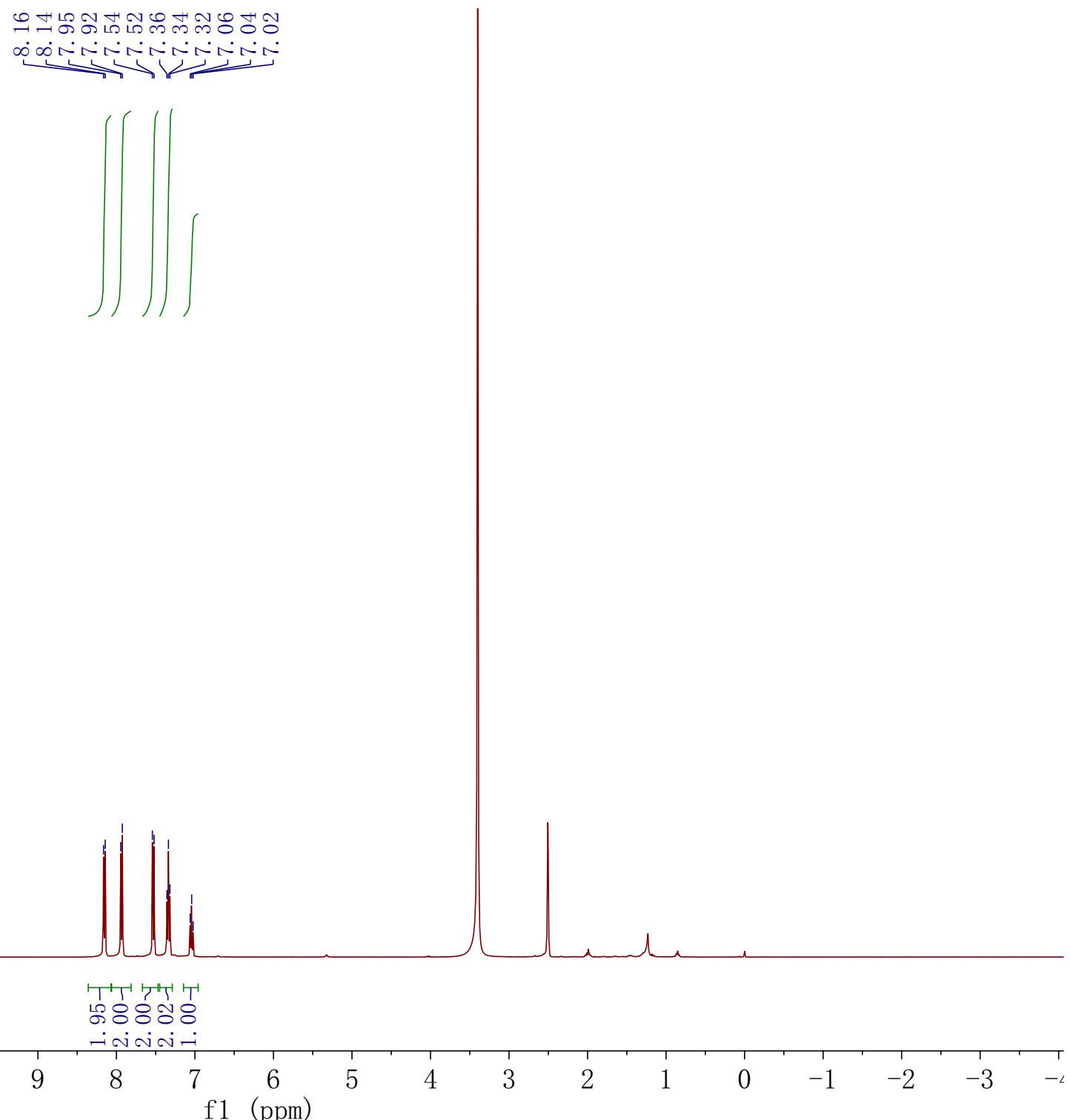


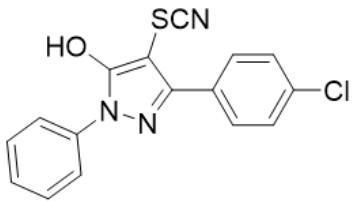
^{19}F NMR (377 MHz, Methanol- d_4) Spectrum of **3n**





¹H NMR (400 MHz, DMSO-*d*₆) Spectrum of 3o



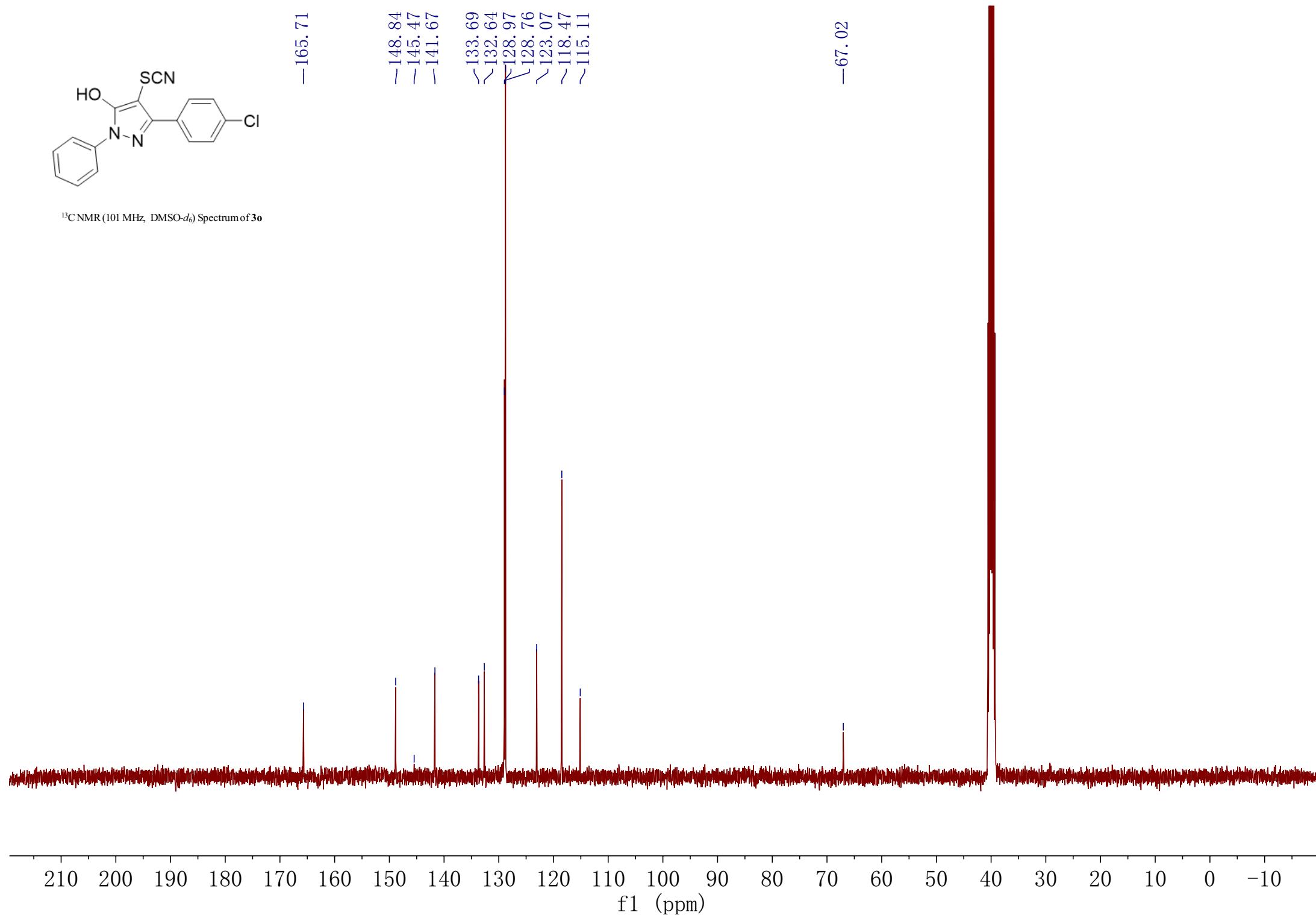


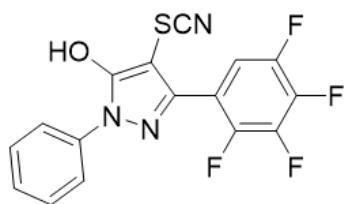
-165.71

-148.84
-145.47
-141.67
~133.69
~132.64
128.97
128.76
~123.07
~118.47
~115.11

-67.02

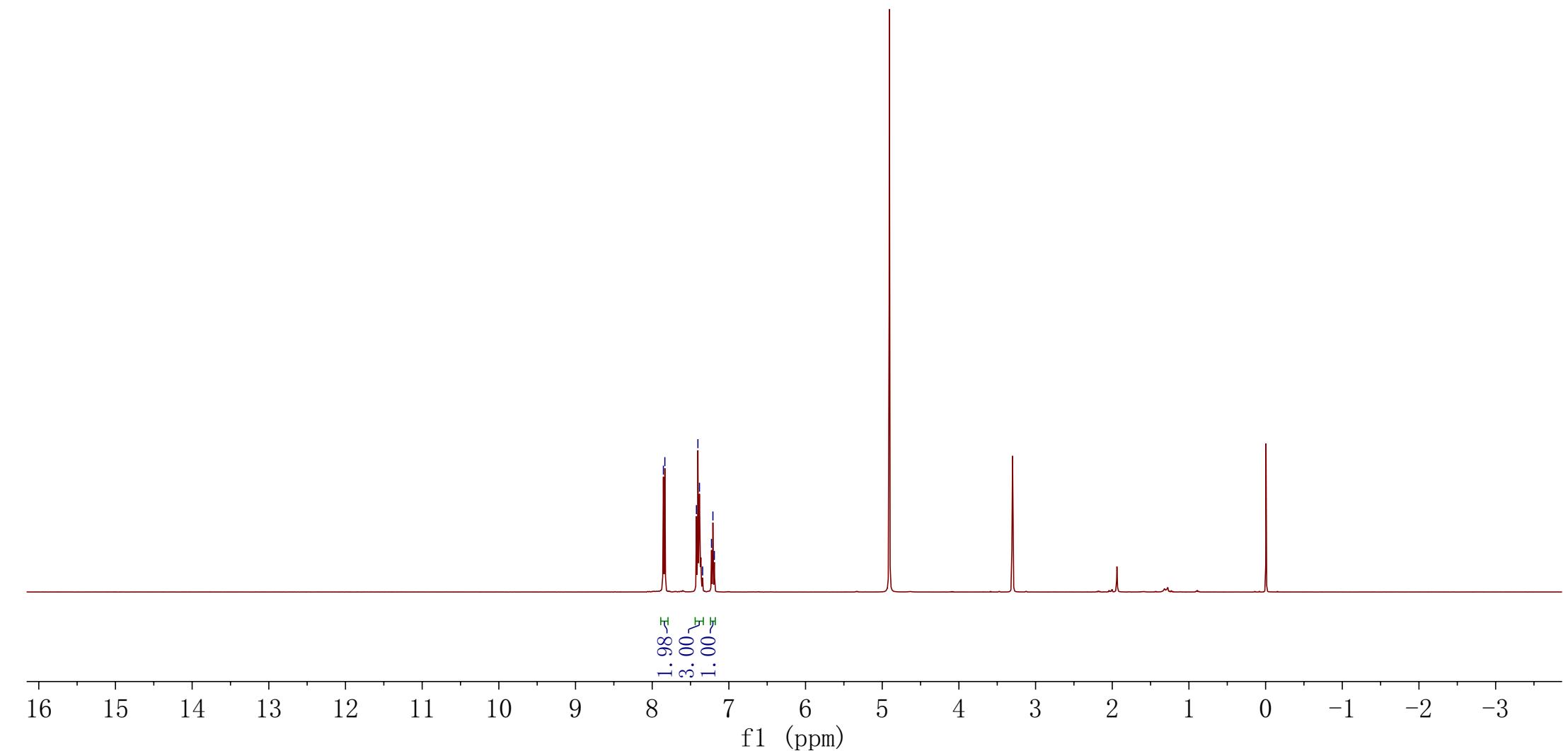
^{13}C NMR (101 MHz, DMSO- d_6) Spectrum of **3o**

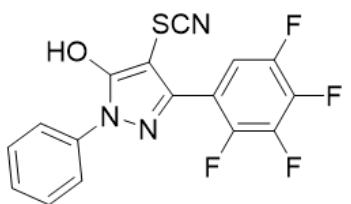




7.85
7.83
7.42
7.40
7.38
7.34
7.23
7.21
7.19

¹H NMR (400 MHz, Methanol-*d*₄) Spectrum of **3p**





-164.22

145.65
141.61
139.76
139.08
128.25
125.01
121.39
118.48
118.26
115.28
113.03
112.06

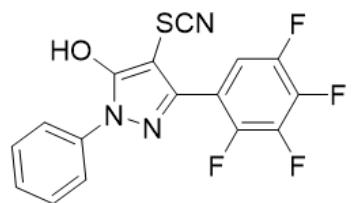
-73.52

-1.44

¹³C NMR (101 MHz, Methanol-*d*₄) Spectrum of 3p

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)



¹⁹F NMR (377 MHz, Methanol-*d*₄) Spectrum of **3p**

