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SUPPORTING INFORMATION

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An intramolecular relay catalysis strategy for Knoevenagel condensation and 1,3-dipolar cycloaddition domino reactions

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1. Experimental Procedures

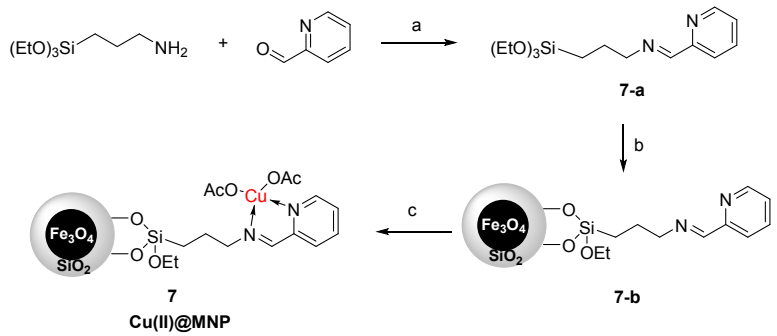
1.1 General information

Fourier transform infrared (FT-IR) spectra were recorded using a Nicolet spectrometer (KBr). X-ray diffraction (XRD) images were obtained using a Bruker D8 Advance instrument. Transmission electron microscopy (TEM) images were run on a Tecnai 12 instrument. Thermogravimetric analysis was performed under nitrogen atmosphere using a Shimadzu TGA-50 spectrometer. Magnetization curves were obtained using vibrating sample magnetometry (VSM; Lakeshore7400-s). ^{1}H NMR spectra were recorded on Bruker DRX500 (500 MHz) and ^{13}C NMR spectra on Bruker DRX500 (125 MHz) spectrometer. Elemental analysis was performed with an Elementar Vario EL β recorder. Copper content of the as-prepared catalysts was measured using inductively coupled plasma (ICP) with an L-PAD analyzer (Prodigy). X-ray photoelectron spectroscopy (XPS) spectra were obtained from an ESCALAB^{TM} 250Xi instrument. The surface mapping analysis was measured by SEM measurements done at FEI Nova NanoSEM 450, USA. Toluene was dehydrated according to standard operation and stored on 4 Å molecular sieves. Other solvents and all of the reagents were commercially available and were used without further purification.

1.2 Synthesis of silica-coated Fe_3O_4 nanoparticles (Fe_3O_4@SiO_2)

The core-shell Fe_3O_4@SiO_2 nanoparticles were prepared according to our previous work [1]. $FeCl_3$ (1.622 g, 10.0 mmol) and $FeCl_2{\cdot}4H_2O$ (0.994 g, 5.0 mmol) were dissolved in 250 mL deionized water under nitrogen with vigorous stirring at 85 ℃. The pH value of the solution was adjusted to 9 by concentrated ammonia. After continuously stirring for 4 h, the magnetite precipitates were collected by a permanent magnet and then washed by amounts of deionized water to reach pH = 7. The black precipitate (Fe_3O_4) was collected with a permanent magnet under the reaction flask. Coating of a layer of silica on the surface of the nano-Fe_3O_4 was achieved by pre-mixing (ultrasonic) the dispersion of the above black precipitate with ethanol (400 mL) for 30 min at room temperature. And $NH_3{\cdot}H_2O$ (7.0 mL) and tetraethyl orthosilicate (TEOS) (2.5 mL) were added successively. After stirring for 24 h, the black precipitate (Fe_3O_4@SiO_2) was collected using a permanent magnet, followed by washing with ethanol three times and drying in vacuum.
FT-IR (KBr, cm^{-1}): 3263, 1602, 1087, 586.

1.3 Synthesis of Cu(II)@MNP (7)



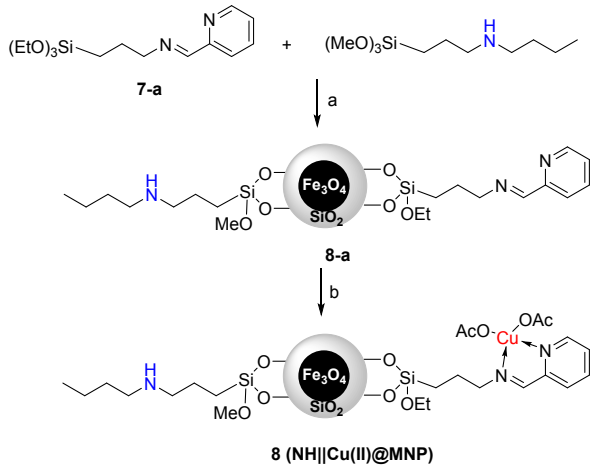
Scheme S1. Synthesis of catalyst **7**. Reaction conditions: (a) dry CH_2Cl_2, N_2, RT, 18 h; (b) Fe_3O_4@SiO_2, toluene, N_2, reflux, 24 h; (c) $Cu(OAc)_2{\cdot}H_2O$, acetone, N_2, RT, 24 h.

Synthesis of 7-a: The compounds **7-a** were synthesized according to the literature [2]. 3-Aminopropyltriethoxysilane (0.502 g, 2.27 mmol) and pyridine-2-carbaldehyde (0.243 g, 2.27 mmol), dried Na_2SO_4 (1.46 g, 10.27 mmol) and dry dichloromethane (10 mL) were added to a round-bottomed flask. The mixture was allowed to stir at room temperature under nitrogen atmosphere. After being stirred for 18 h, the resulting mixture was filtered for separation of Na_2SO_4, which was washed with dried dichloromethane. The filtrate was concentrated and dried under vacuum to afford brown viscous oil in 79.9% yield (0.5632 g, 1.81 mmol). The product was identified by comparison with the spectroscopic data in the literature [2, 3]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, J = 4.2 Hz, 1H), 8.38 (s, 1H), 7.99 (d, J = 8.4 Hz, 1H), 7.74 (td, J = 7.8, 1.8 Hz, 1H), 7.30 (ddd, J = 7.4, 4.9, 1.0 Hz, 1H), 3.83 (q, J = 7.0 Hz, 6H), 3.6 (m, 2H), 1.89-1.83 (m, 2H), 1.23 (t, J = 7.0 Hz, 9H), 0.71-0.68 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$): 161.89, 154.58, 149.32, 136.48, 124.57, 121.15, 64.03, 58.33, 24.06, 18.24, 8.03.

Synthesis of 7-b: 1.0 g of Fe_3O_4@SiO_2 NPs were dispersed in dry toluene (50 mL) by sonication for 1 h. ligand **7-a** (0.465 g, 1.5 mmol) and pyridine (0.589g, 7.45 mmol, 0.6 mL) were then added, and the reaction mixture was refluxed for 24 h under nitrogen. After being cooled to room temperature, the compound **7-b** was recycled by a permanent magnet. The resulting compound **7-b** was washed with dry toluene and acetone to remove no reacted species and then dried under vacuum overnight.

Synthesis of Cu(II)@MNP (7): To a round-bottomed flask, copper sulfate monohydrate (0.2 g, 1.0 mmol) and acetone (40 mL) were added. The solution was stirred at room temperature for 30 min, then 0.5 g of the above compound **7-b** was added. The mixture was stirred at room temperature for 24 h under nitrogen atmosphere, and the catalyst **7** was collected by a permanent magne, washed several times with acetone and dried under vacuum overnight.

1.4 Synthesis of NH||Cu(II)@MNP (8)



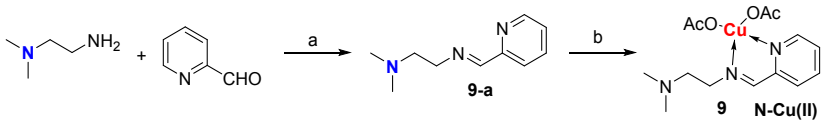
Scheme S2. Synthesis of catalyst **8**. Reaction conditions: (a) Fe_3O_4@SiO_2, toluene, N_2, reflux, 24 h; (b) $Cu(OAc)_2 \cdot H_2O$, acetone, N_2, RT, 24 h.

Synthesis of 8-a: 1.0 g of Fe_3O_4@SiO_2 NPs were dispersed in dry toluene (50 mL) by sonication for 1 h. ligand **7-a** (0.465 g, 1.5 mmol), N-(3-(trimethoxysilyl)propyl)butan-1-amine (0.353 g, 1.5 mmol) and pyridine (0.589g, 7.45 mmol, 0.6 mL) were then added, and the reaction mixture was refluxed for 24 h under nitrogen. After being cooled to room temperature, the compound **8-a** was recovered by a permanent magnet, washed with dry toluene and acetone to remove no reacted species and then dried under vacuum overnight.

Synthesis of NH||Cu(II)@MNP (8): To a round-bottomed flask, copper sulfate monohydrate (0.2 g, 1.0 mmol) and acetone (40 mL) were added. The solution was stirred at room temperature for 30 min,

then 0.5 g of the above compound **8-a** was added. The mixture was stirred at room temperature for 24 h under nitrogen atmosphere, and the catalyst **8** was collected by a permanent magnet, washed several times with acetone and dried under vacuum overnight.

1.5 Synthesis of N-Cu(II) (9)



Scheme S3. Synthesis of catalyst **9**. Reaction conditions: (a) anhydrous ethanol, N_2, reflux, 4h; (b) $Cu(OAc)_2 \cdot H_2O$, acetone, N_2, RT, 24h.

Synthesis of ligand 9-a: 2-Dimethylaminoethylamine (0.176 g, 2.0 mmol) and pyridine-2-carbaldehyde (0.214 g, 2.0 mmol) were dissolved in anhydrous ethanol (20 mL) and the mixture was refluxed under nitrogen atmosphere. After being stirred for 4 h, the resulting mixture was cooled to room temperature and evaporated under reduced pressure. The desired product was obtained as a brown oil in 95% yield. The product was identified by comparison with the spectroscopic data in the literature [4, 5]. ^{1}H NMR (500 MHz, CDCl3) δ 8.56 (d, J = 4.7 Hz, 1H), 8.34 (s, 1H), 7.91 (d, J = 7.9 Hz, 1H), 7.65 (td, J = 7.8 Hz, J = 1.5 Hz , 1H), 7.23 (t, J = 6.2 Hz, 1H), 3.73 (t, J = 6.9 Hz, 2H), 2.59 (t, J = 6.9 Hz, 2H), 2.23 (s, 6H). ^{13}C NMR (126 MHz, CDCl3): 162.74, 154.42, 149.39, 136.52, 124.71, 121.37, 77.40, 77.14, 76.89, 59.87, 59.48, 45.75.

Synthesis of 9: Copper sulfate monohydrate (0.200 g, 1.0 mmol) was dissolved in acetone (40 mL) and the solution was stirred at room temperature for 30 min, then ligand **9-a** (0.177 g, 1.0 mmol) was added. The mixture was stirred at room temperature for 24 h under nitrogen atmosphere. The mixture was evaporated under reduced pressure and used directly for the Knoevenagel condensation and 1,3-dipolar cycloaddition domino reactions without further purification.

2. Results and Discussion

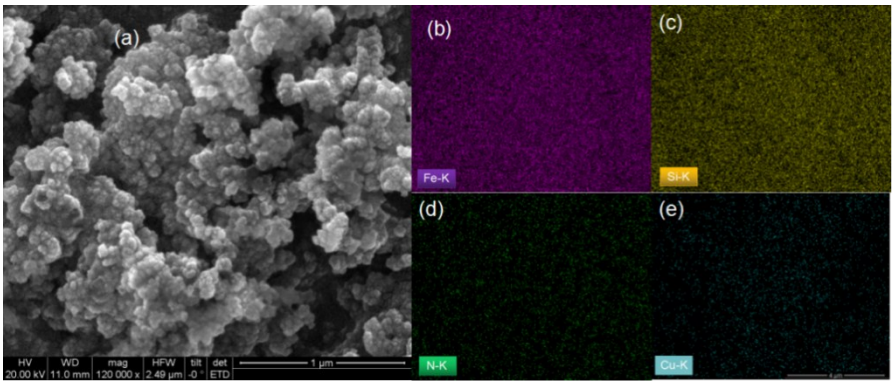
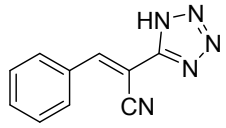
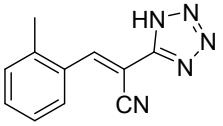


Figure S1. SEM images of NH-Cu(II)@MNP (a) and elemental mapping of Fe, Si, N and Cu (b-e).

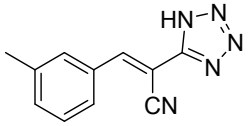
3. Spectroscopic Data of the Synthesized Compounds



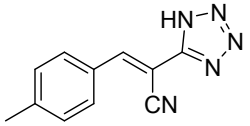
(*E*)-3-Phenyl-2-(1*H*-tetrazole-5-yl) acrylonitrile (6a). Powder. Yield : 92%. ^{1}H NMR (500 MHz, Acetone) δ 8.41 (s, 1H), 8.17 - 7.99 (m, 2H), 7.77 - 7.43 (m, 3H). ^{13}C NMR (126 MHz, DMSO): 156.21, 149.09, 133.01, 132.88, 130.55, 130.00, 116.21, 97.70. Elemental Analysis Calcd (%): C, 60.91; H, 3.58; N, 35.51; Found: C, 60.82; H, 3.95; N, 35.23. Spectroscopic data for the product were consistent with the literature[6].



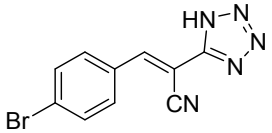
(*E*)-3-(2-Methylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6b). Powder. Yield : 82%. ^{1}H NMR (500 MHz, DMSO) δ 8.45 (s, 1H), 7.91 (d, J = 7.6 Hz, 1H), 7.39 (t, J = 7.3 Hz, 1H), 7.31 (t, J = 8.4 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (126 MHz, DMSO): 155.52, 148.02, 139.46, 132.38, 132.10, 131.48, 128.46, 127.02, 115.92, 99.62, 20.07. Elemental Analysis Calcd (%): C, 62.55; H, 4.29; N, 33.16; Found: C, 62.64; H, 4.25; N, 33.11. Spectroscopic data for the product were consistent with the literature[6].



(*E*)-3-(3-Methylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6c). Powder. Yield : 90%. ^{1}H NMR (500 MHz, DMSO) δ 8.35 (s, 1H), 7.86 (d, J = 7.7 Hz, 1H), 7.83 (s, 1H), 7.51 (t, J = 7.6 Hz, 1H), 7.45 (d, J = 7.5 Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (126 MHz, DMSO): 156.19, 149.10, 139.33, 133.73, 132.83, 131.07, 129.89, 127.63, 116.20, 97.38, 21.58. Elemental Analysis Calcd (%): C, 62.55; H, 4.29; N, 33.16; Found: C, 62.65; H, 4.22; N, 33.13. Spectroscopic data for the product were consistent with the literature[6].

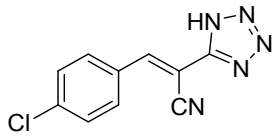


(*E*)-3-(4-Methylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6d). Powder. Yield : 87%. ^{1}H NMR (500 MHz, DMSO) δ 8.21 (s, 1H), 7.83 (d, J = 8.1 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 2.30 (s, 3H). ^{13}C NMR (126 MHz, DMSO): 156.10, 148.90, 143.67, 130.63, 130.55, 130.13, 116.32, 96.13, 21.90. Elemental Analysis Calcd (%): C, 62.55; H, 4.29; N, 33.16; Found: C, 62.65; H, 4.15; N, 33.20. Spectroscopic data for the product were consistent with the literature[6].

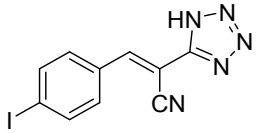


(*E*)-3-(4-Bromophenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6e). Powder. Yield : 81%. ^{1}H NMR (500 MHz, Acetone) δ 8.40 (s, 1H), 8.02 (d, J = 8.5 Hz, 2H), 7.79 (d, J = 8.5 Hz, 2H). ^{13}C NMR (126 MHz, DMSO): 156.40, 147.56, 133.00, 132.26, 131.98, 126.60, 115.97, 98.42. Elemental Analysis Calcd (%):

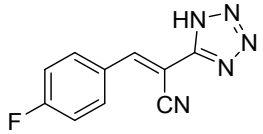
C, 43.50; H, 2.19; N, 25.37; Found: C, 43.58; H, 2.14; N, 25.31. Spectroscopic data for the product were consistent with the literature[6].



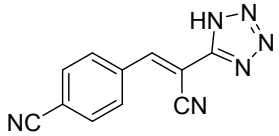
(*E*)-3-(4-Chlorophenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6f). Powder. Yield : 88%. ^{1}H NMR (500 MHz, DMSO) δ 8.40 (s, 1H), 8.04 (d, J = 8.5 Hz, 2H), 7.69 (d, J = 8.5 Hz, 2H). ^{13}C NMR (126 MHz, DMSO): 156.40, 147.55, 137.54, 132.23, 131.76, 130.12, 116.04, 98.45. Elemental Analysis Calcd (%): C, 51.85; H, 2.61; N, 30.23; Found: C, 51.79; H, 2.66; N, 30.27. Spectroscopic data for the product were consistent with the literature[7].



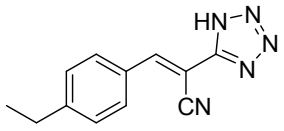
(*E*)-3-(4-Iodophenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6g). Powder. IR spectrum, v, cm^{-1}: 3050, 2220, 1610, 1580. Yield : 87%. ^{1}H NMR (500 MHz, Acetone) δ 8.37 (s, 1H), 8.00 (d, J = 8.4 Hz, 2H), 7.86 (d, J = 8.4 Hz, 2H). ^{13}C NMR (126 MHz, DMSO): 156.37, 147.98, 138.92, 132.28, 132.03, 116.05, 100.90, 98.32. MS (ESI) m/z for $C_{10}H_6IN_5$: 321.87 ([M-H]$^-$).



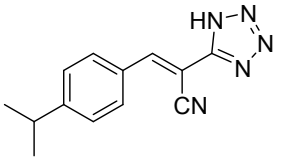
(*E*)-3-(4-Fluorophenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6h). Powder. IR spectrum, v, cm^{-1}: 3460, 3110, 2230, 1580, 1510. Yield : 84%. ^{1}H NMR (500 MHz, Acetone) δ 8.42 (s, 1H), 8.18 (dd, J = 8.6, 5.5 Hz, 2H), 7.40 (t, J = 8.7 Hz, 2H). ^{13}C NMR (126 MHz, DMSO): 164.69 (d, J = 252.5 Hz), 156.35, 147.72, 133.31, 133.24, 129.57, 117.32, 117.15, 116.19, 97.43. MS (ESI) m/z for $C_{10}H_6FN_5$: 214.00 ([M-H]$^-$).



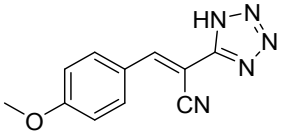
(*E*)-3-(4-Cyanophenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6i). Powder. Yield : 95%. IR spectrum, v, cm^{-1}: 3500, 3070, 2240, 2200, 1610, 1510, 1460. ^{1}H NMR (500 MHz, DMSO) δ 8.50 (s, 1H), 8.17 (d, J = 8.1 Hz, 2H), 8.08 (d, J = 6.7 Hz, 2H). ^{13}C NMR (126 MHz, DMSO): 156.68, 146.73, 137.15, 133.69, 130.95, 118.92, 115.72, 114.39, 101.31**.** MS (ESI) m/z for $C_{11}H_6N_6$: 220.98 ([M-H]$^-$).



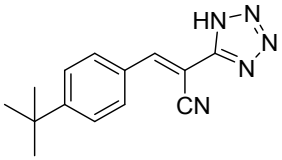
(*E*)-3-(4-Ethylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6j). Powder. Yield : 75%. IR spectrum, v, cm^{-1}: 3070, 2960, 2930, 2870, 2230, 1600, 1560. ^{1}H NMR (500 MHz, DMSO) δ 8.30 (s, 1H), 7.93 (d, J = 8.2 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 2.66 (q, J = 7.6 Hz, 2H), 1.19 (t, J = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, DMSO): 156.14, 149.74, 148.99, 130.77, 130.41, 129.42, 116.37, 96.24, 28.95, 15.77. MS (ESI) m/z for $C_{12}H_{11}N_5$: 224.08 ([M-H]$^-$).



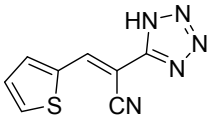
(*E*)-3-(4-Isopropylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6k). Powder. Yield : 77%. ^{1}H NMR (500 MHz, Acetone) δ 8.37 (s, 1H), 8.02 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.2 Hz, 2H), 3.00 (dt, J = 13.8, 6.9 Hz, 1H), 1.27 (d, J = 6.9 Hz, 6H). ^{13}C NMR (126 MHz, DMSO): 156.17, 154.20, 148.97, 130.82, 130.57, 128.00, 116.37, 96.34, 34.26, 24.09. Elemental Analysis Calcd (%): C, 65.25; H, 5.48; N, 29.27; Found: C, 65.50; H, 5.28; N, 29.22.



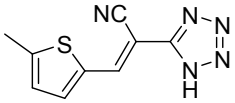
(*E*)-3-(4-Methoxyphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6l). Powder. Yield : 74%. ^{1}H NMR (500 MHz, DMSO) δ 8.30 (s, 1H), 8.04 (d, J = 8.7 Hz, 2H), 7.18 (d, J = 8.7 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (126 MHz, DMSO): 163.28, 156.04, 148.65, 132.94, 125.41, 116.70, 115.56, 93.68, 56.32. Elemental Analysis Calcd (%): C, 58.14; H, 3.99; N, 30.82; Found: C, 58.10; H, 4.15; N, 30.90.



(*E*)-3-(4-tert-Butylphenyl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6m). Powder. Yield : 70%. IR spectrum, ν, cm^{-1}: 3120, 2960, 2870, 2220, 1600, 1470. ^{1}H NMR (500 MHz, DMSO) δ 8.36 (s, 1H), 7.98 (d, J = 8.5 Hz, 2H), 7.63 (d, J = 8.5 Hz, 2H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, DMSO): 156.38, 156.17, 148.89, 130.57, 130.20, 126.88, 116.36, 96.45, 35.63, 31.42. MS (ESI) m/z for $C_{14}H_{15}N_5$: 252.05 ([M-H]$^-$).



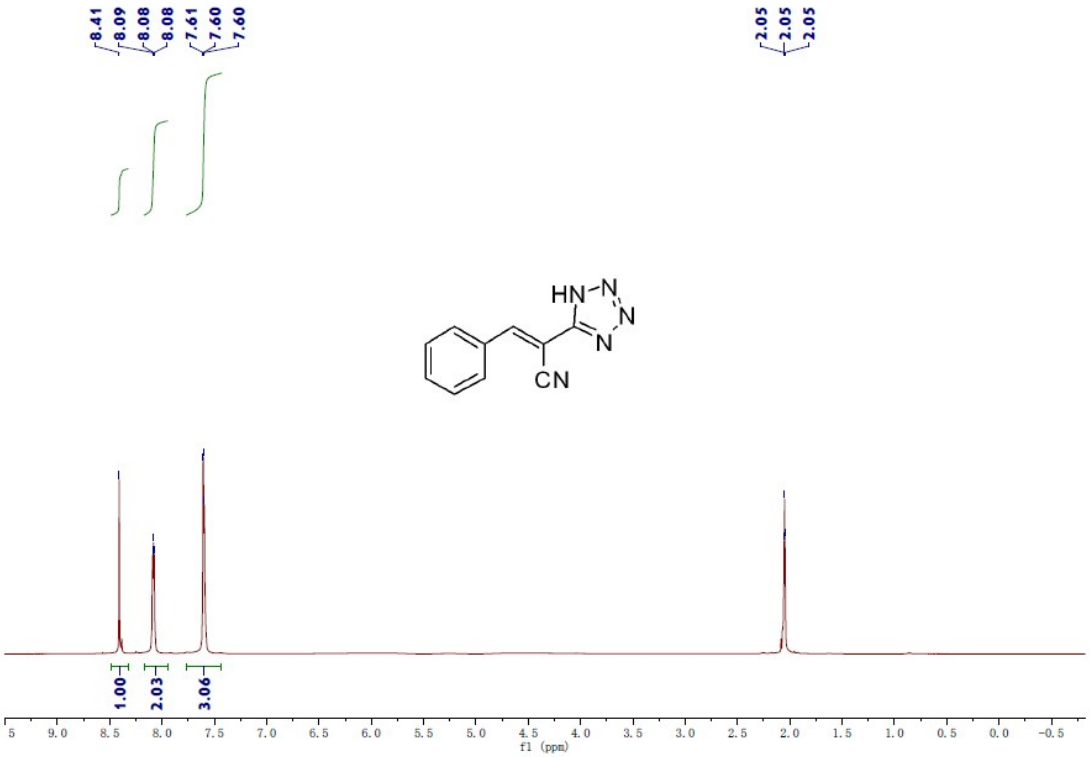
(*E*)-3-(Thiophen-2-yl)-2-(1*H*-tetrazol-5-yl) acrylonitrile (6n). Powder. Yield : 91%. ^{1}H NMR (500 MHz, Acetone) δ 8.58 (s, 1H), 8.02 (d, J = 5.0 Hz, 1H), 7.95 (d, J = 3.6 Hz, 1H), 7.41 - 7.26 (m, 1H). ^{13}C NMR (126 MHz, DMSO): 155.86, 141.82, 138.35, 136.77, 135.48, 129.27, 116.44, 93.06. Elemental Analysis Calcd (%): C, 47.28; H, 2.48; N, 34.46; Found: C, 47.33; H, 2.43; N, 34.50. Spectroscopic data for the product were consistent with the literature[6].



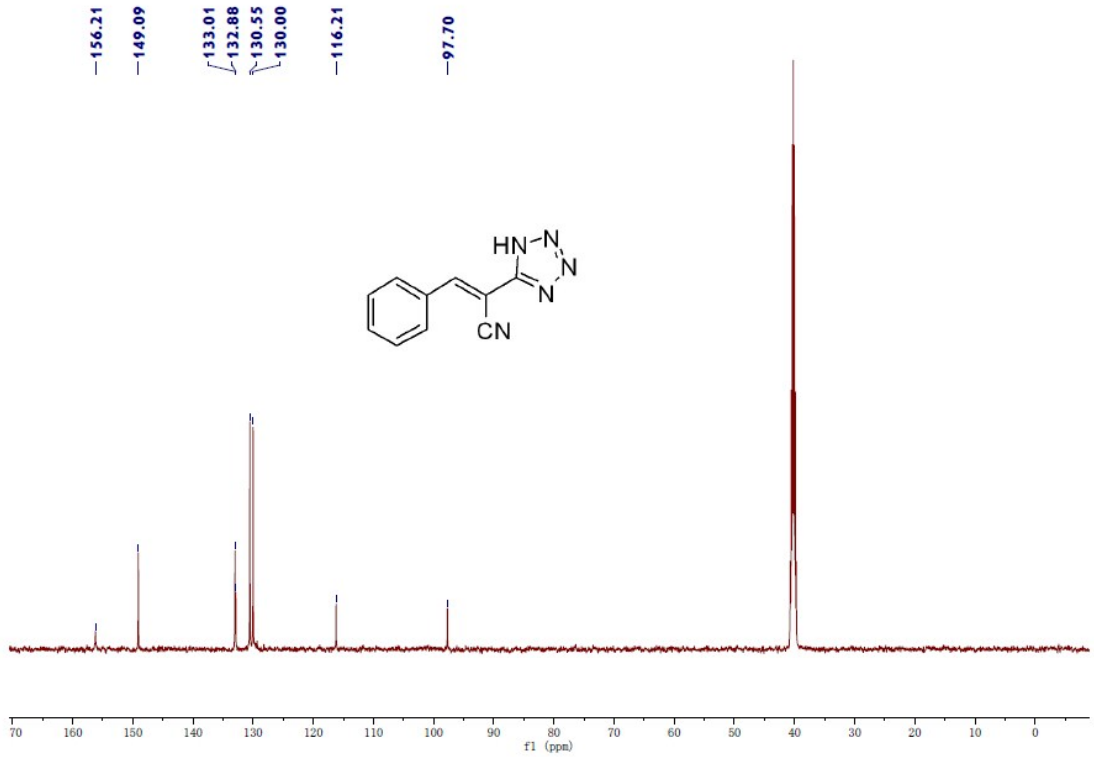
(*E*)-3-(5-Methyl-thiophen-2-yl)-2-(1*H*-tetrazol-5-yl)- acrylonitrile (6o). Powder. Yield : 95%. ^{1}H NMR (500 MHz, DMSO) δ 8.50 (s, 1H), 7.79 (d, J = 3.7 Hz, 1H), 7.11-7.08 (m, 1H), 2.63 (s, 3H). ^{13}C NMR (126 MHz, DMSO): 155.05, 149.90, 141.32, 138.79, 134.15, 127.45, 115.93, 90.47, 15.66. Elemental Analysis Calcd (%): C, 49.76; H, 3.25; N, 32.24; Found: C, 49.82; H, 3.21; N, 32.27. Spectroscopic data for the product were consistent with the literature[7].

4. ^{1}H-NMR and ^{13}C-NMR Spectra of Compounds

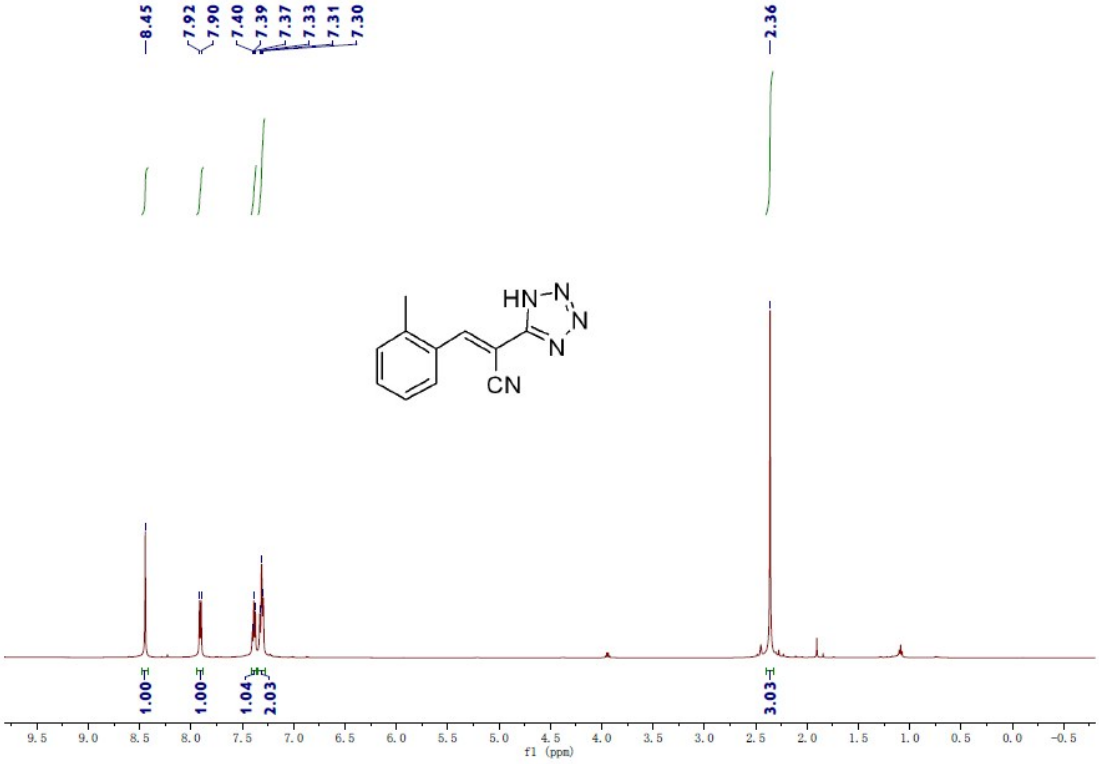
^{1}H NMR spectrum of the product of 6a (Acetone, 500 MHz)



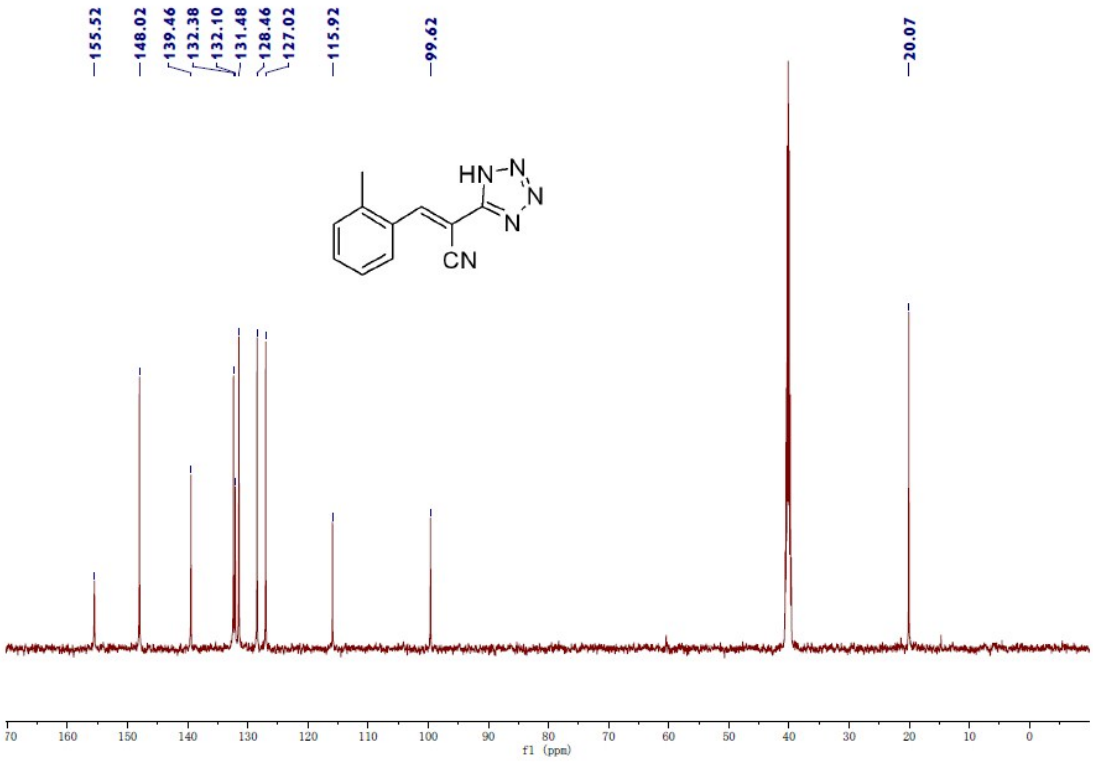
^{13}C NMR spectrum of the product of 6a (DMSO, 126 MHz)



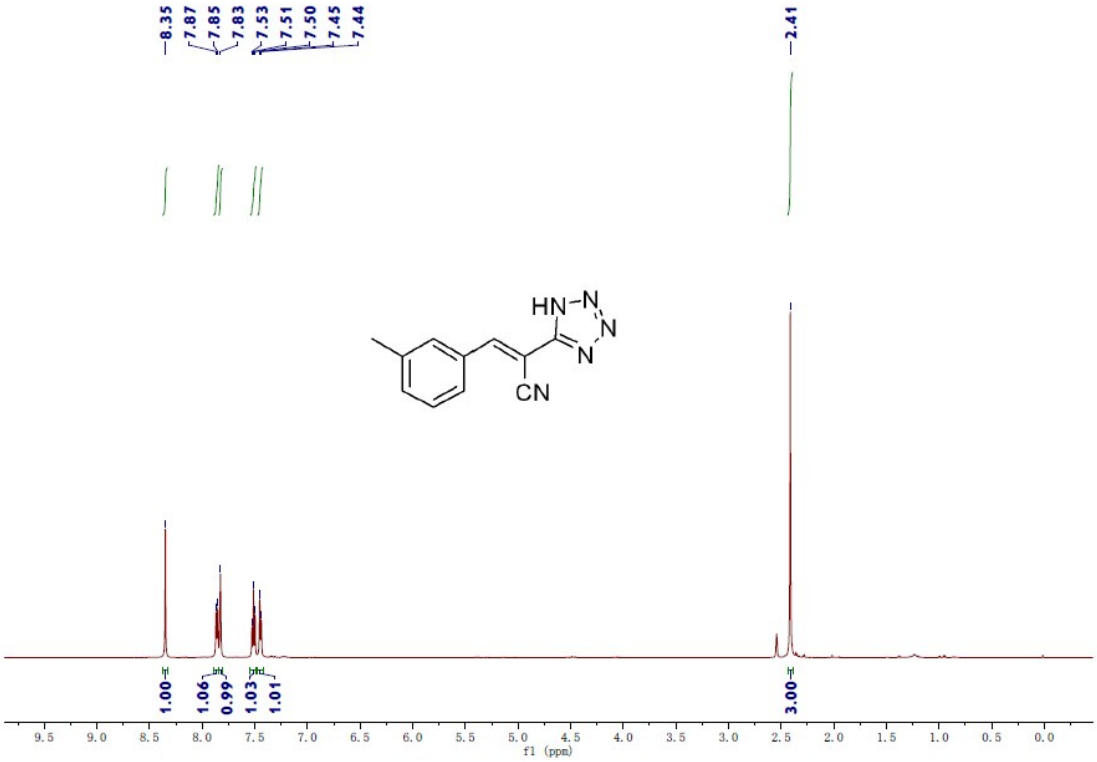
^{1}H NMR spectrum of the product of 6b (DMSO, 500 MHz)



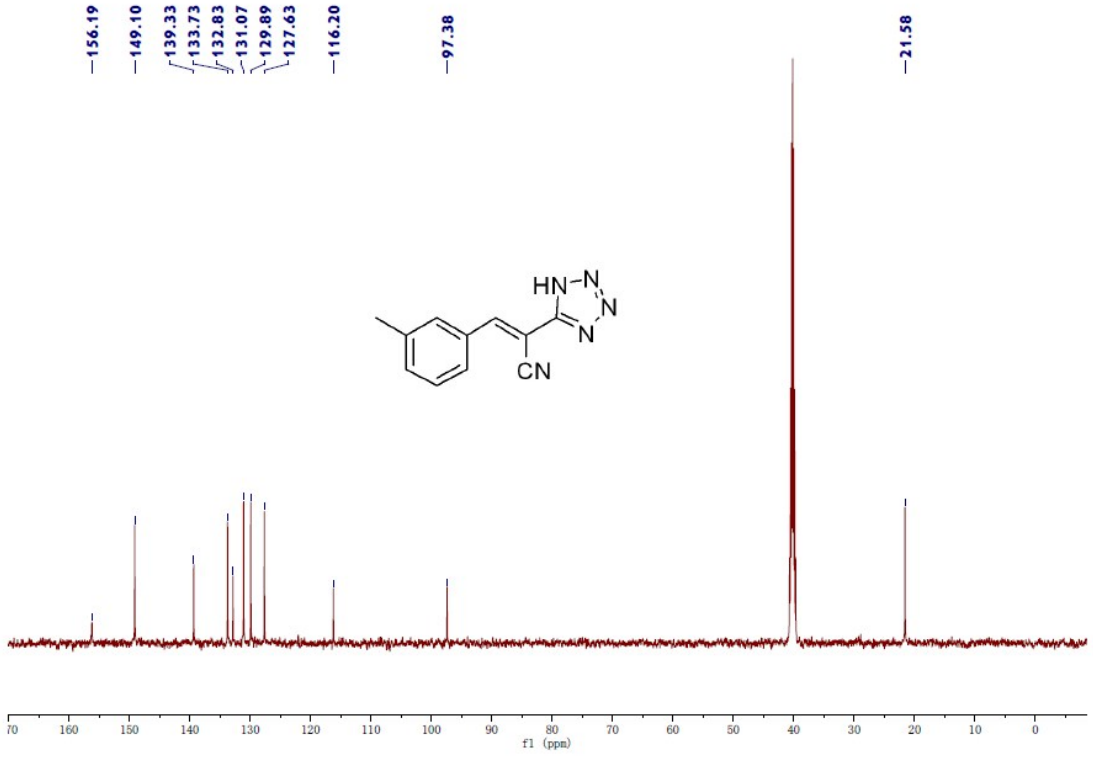
^{13}C NMR spectrum of the product of 6b (DMSO, 126 MHz)



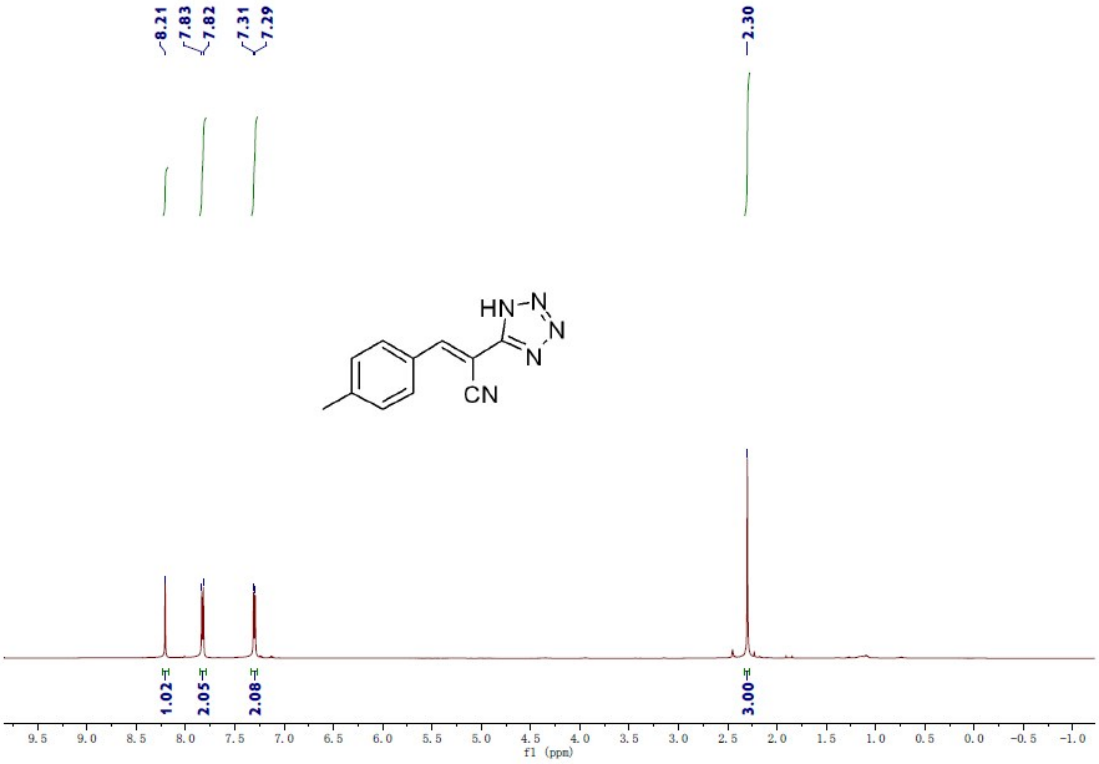
^{1}H NMR spectrum of the product of 6c (DMSO, 500 MHz)



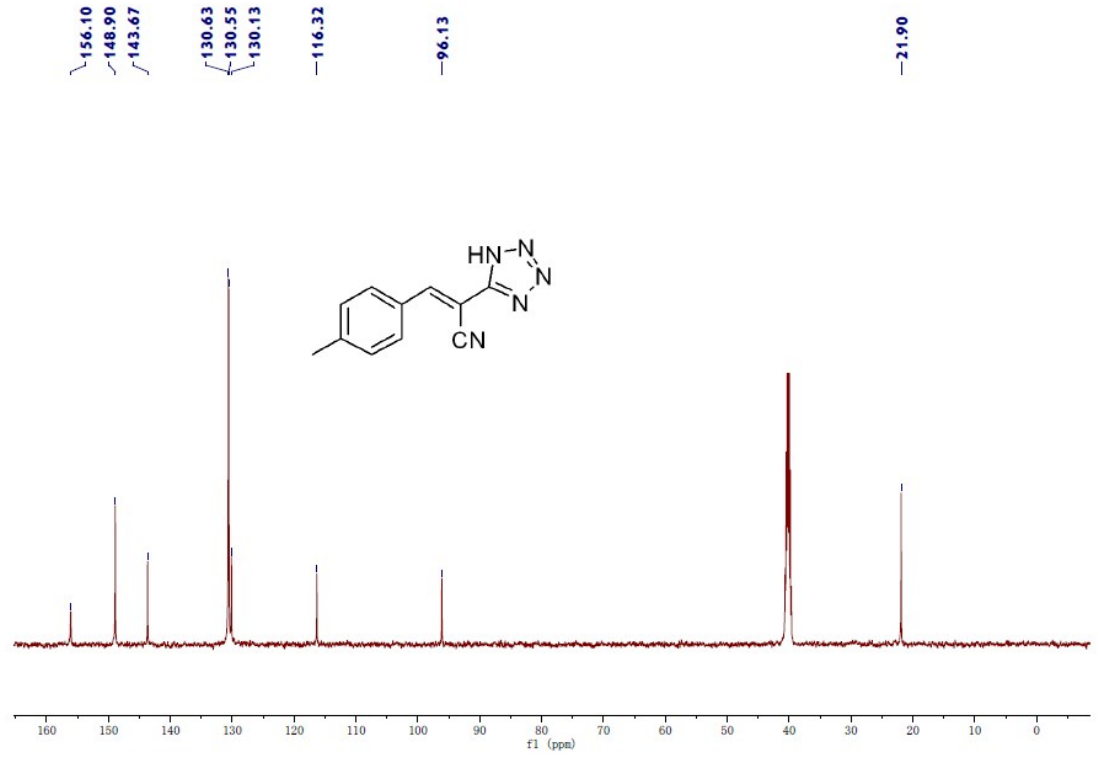
^{13}C NMR spectrum of the product of 6c (DMSO, 126 MHz)



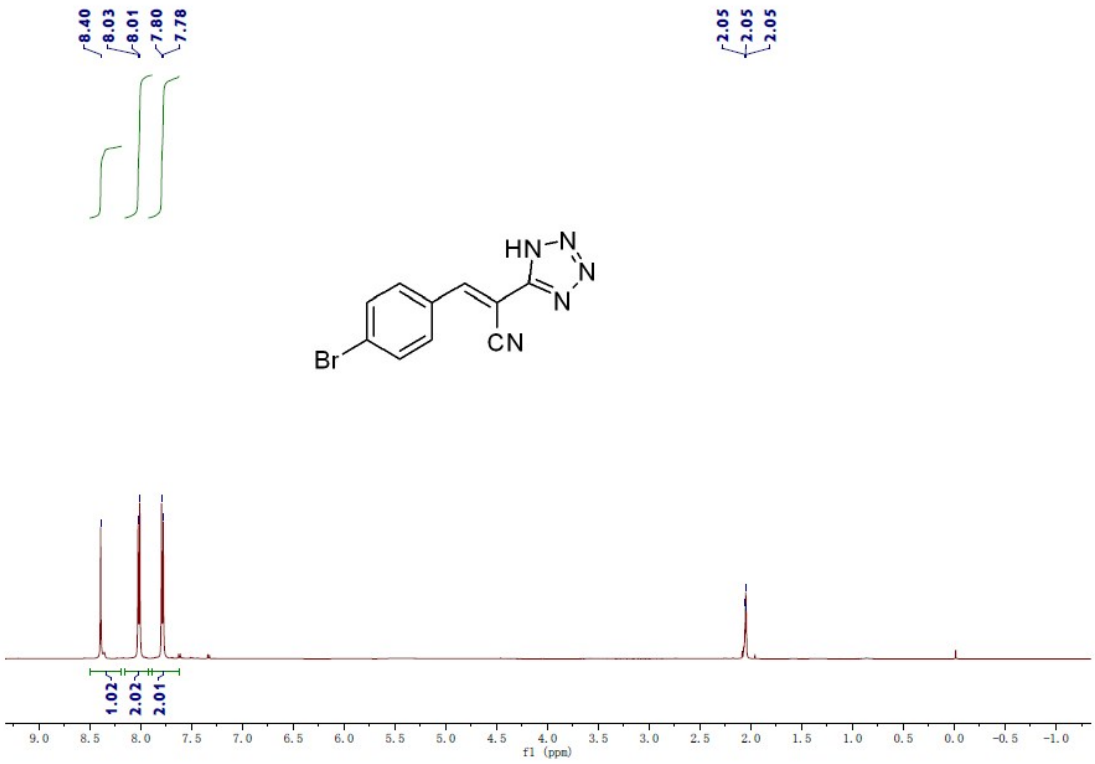
^{1}H NMR spectrum of the product of 6d (DMSO, 500 MHz)



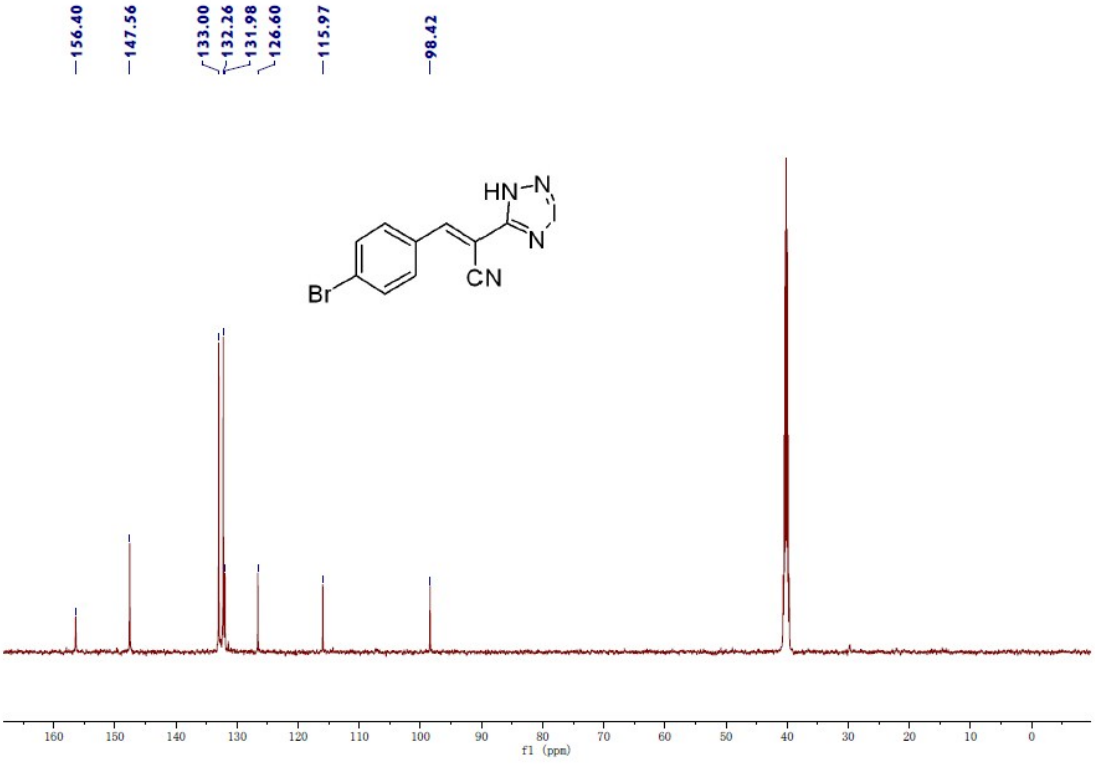
^{13}C NMR spectrum of the product of 6d (DMSO, 126 MHz)



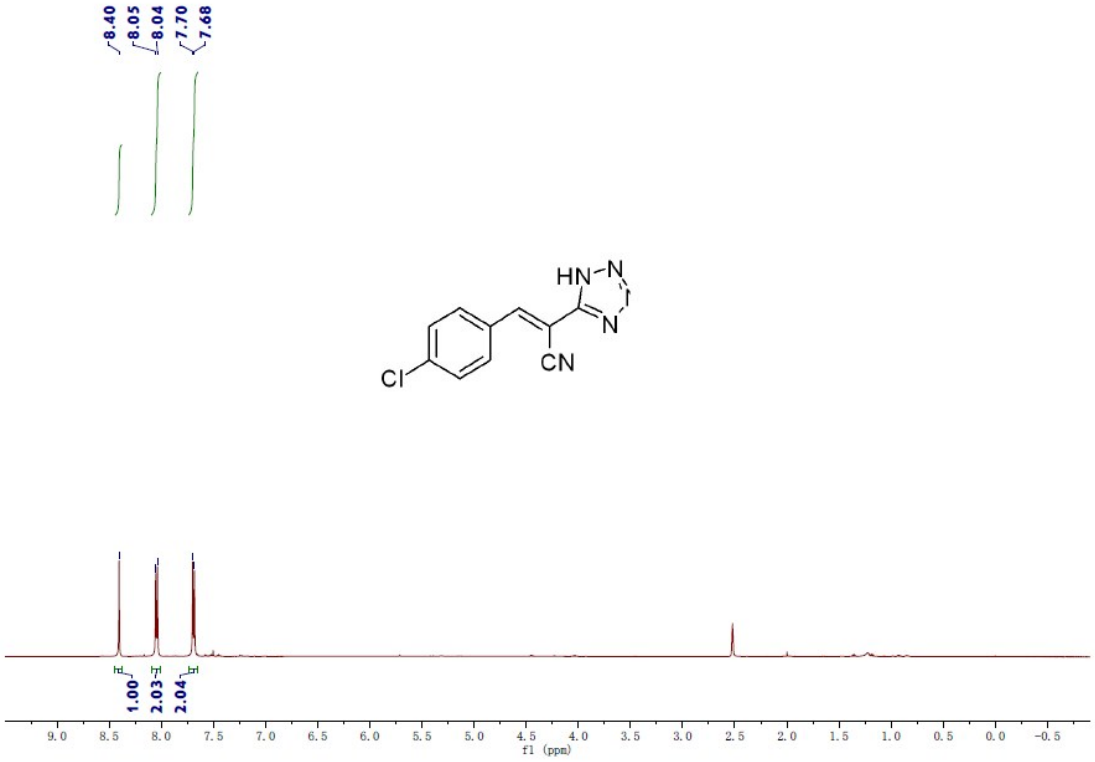
^{1}H NMR spectrum of the product of 6e (Acetone, 500 MHz)



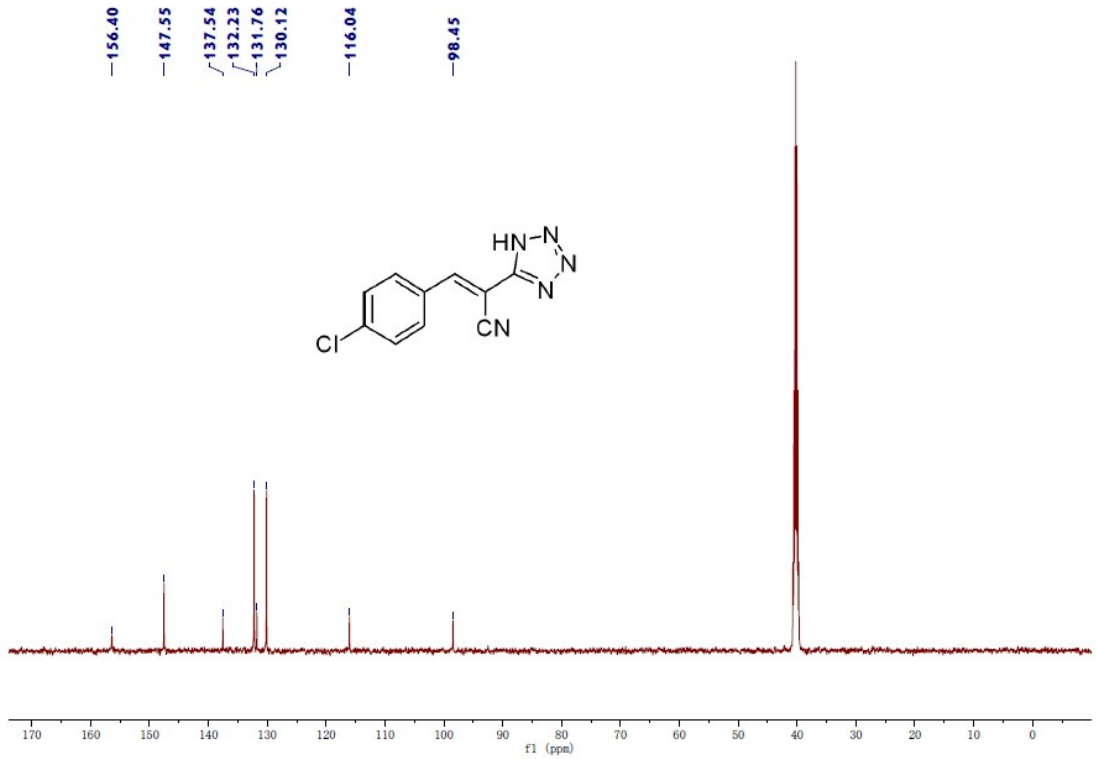
^{13}C NMR spectrum of the product of 6e (DMSO, 126 MHz)



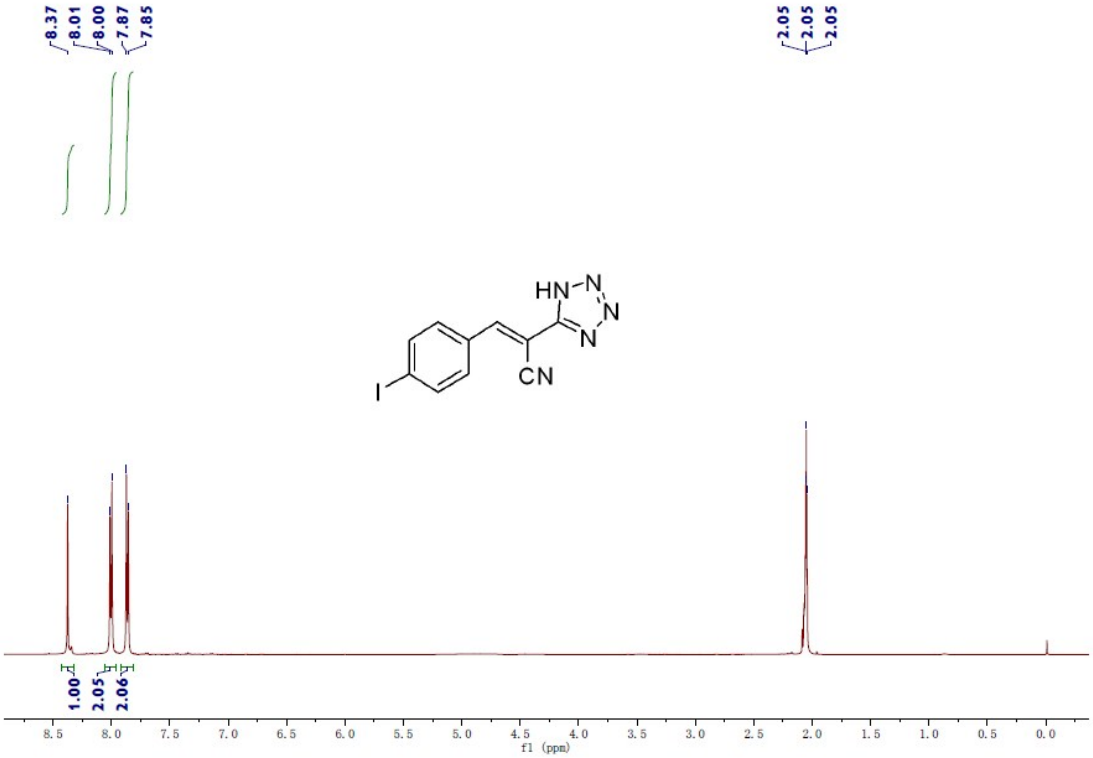
^{1}H NMR spectrum of the product of 6f (DMSO, 500 MHz)



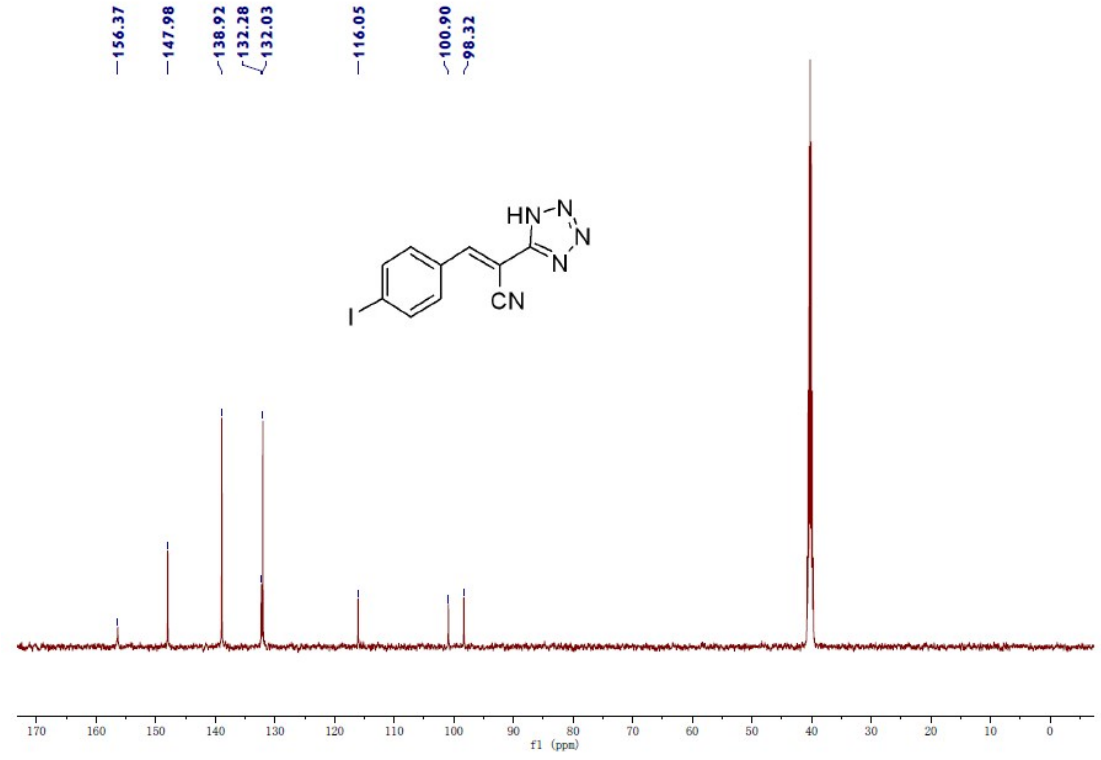
^{13}C NMR spectrum of the product of 6f (DMSO, 126 MHz)



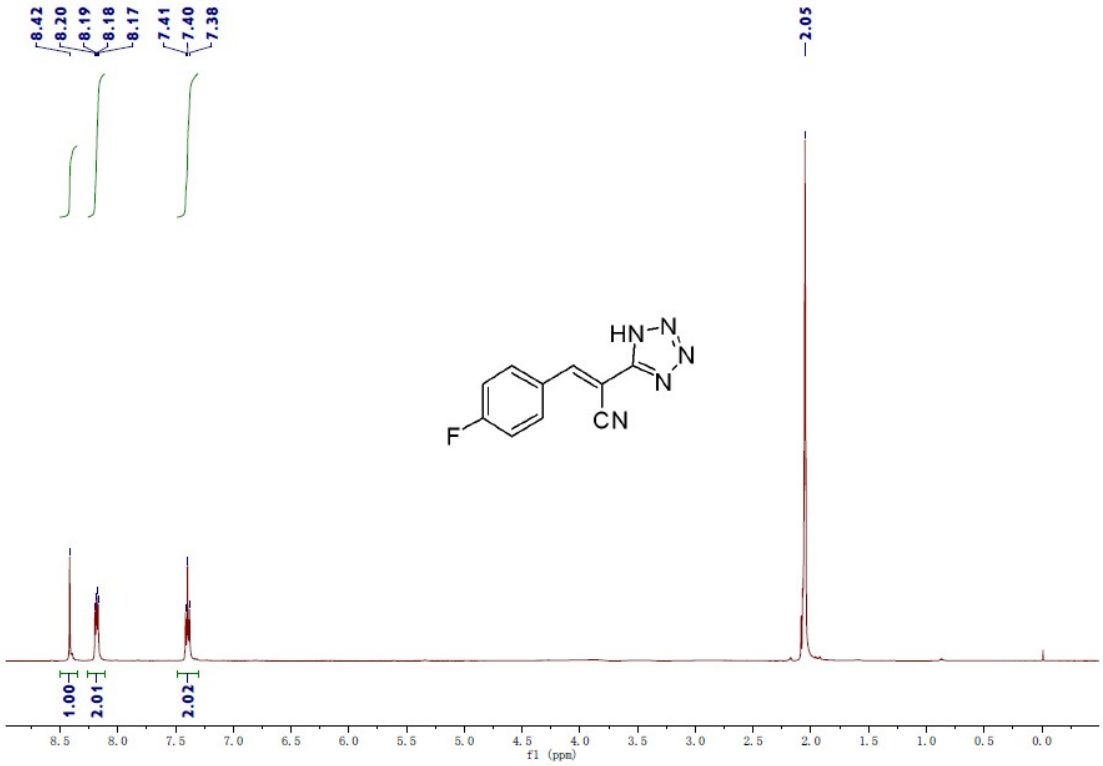
^{1}H NMR spectrum of the product of 6g (Acetone, 500 MHz)



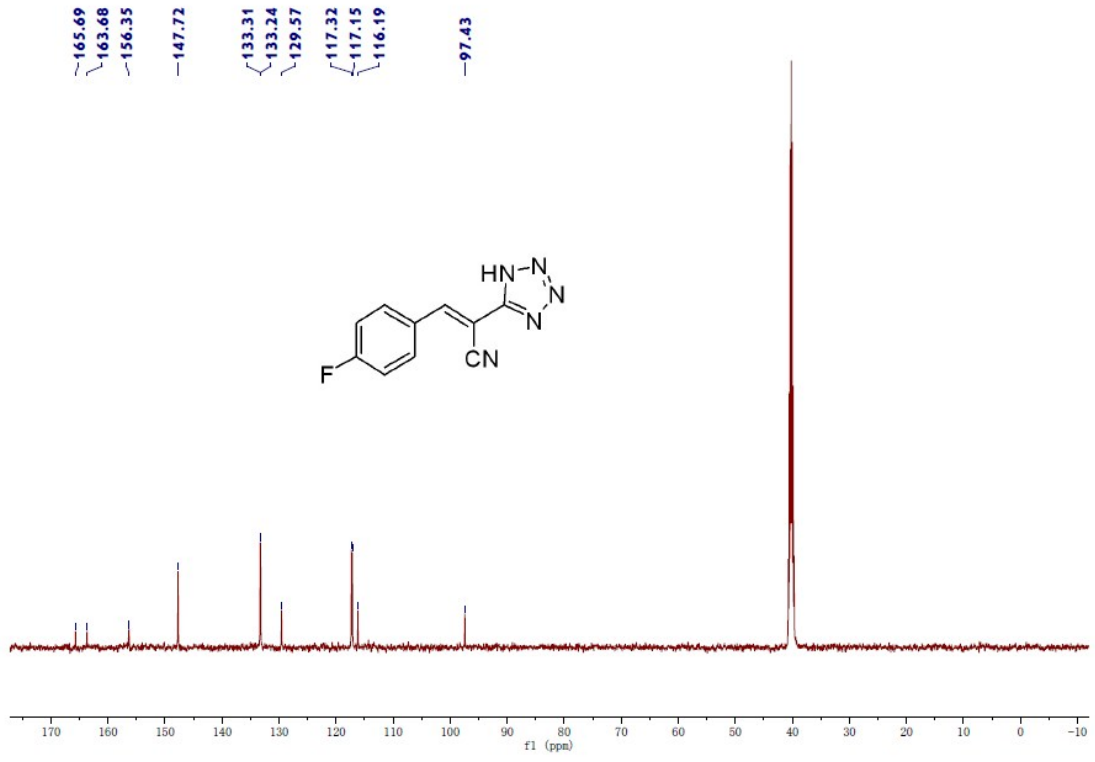
^{13}C NMR spectrum of the product of 6g (DMSO, 126 MHz)



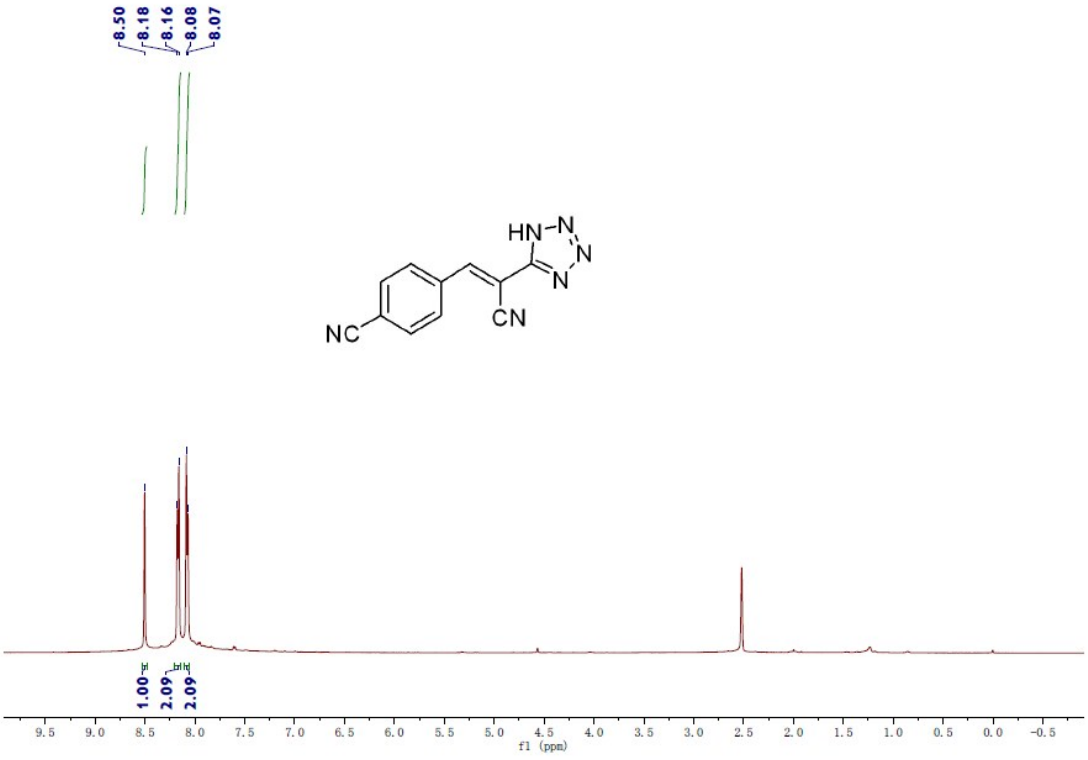
^{1}H NMR spectrum of the product of 6h (Acetone, 500 MHz)



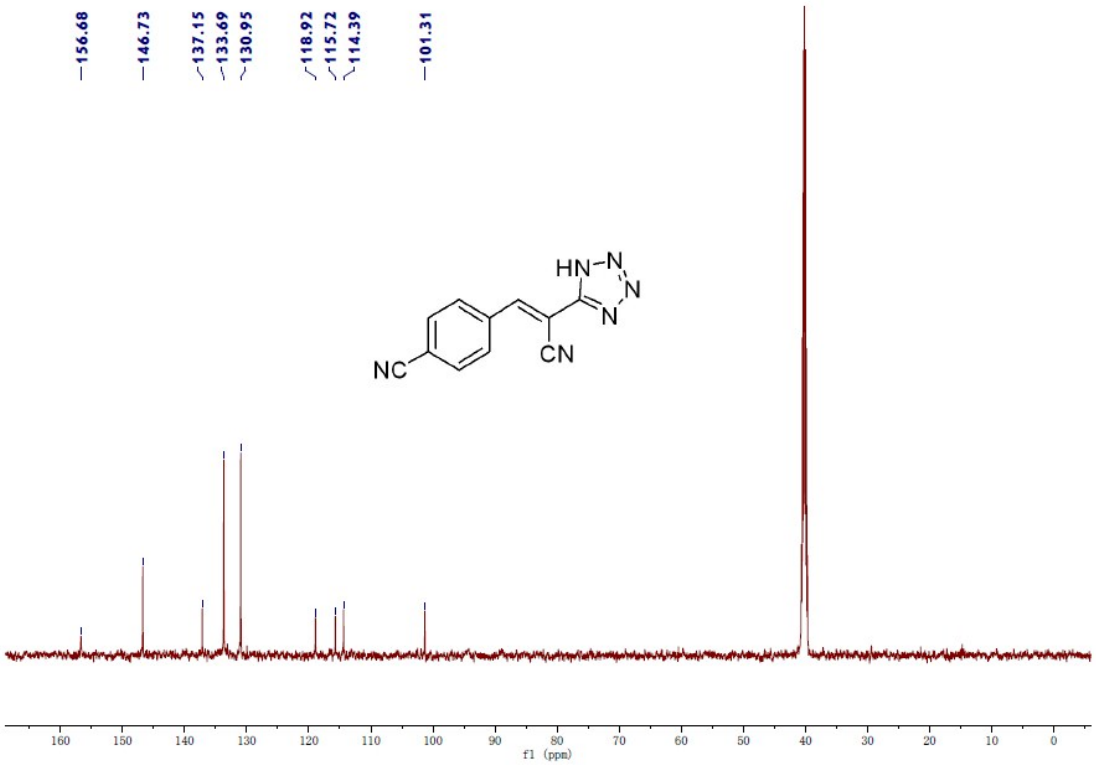
^{13}C NMR spectrum of the product of 6h (DMSO, 126 MHz)



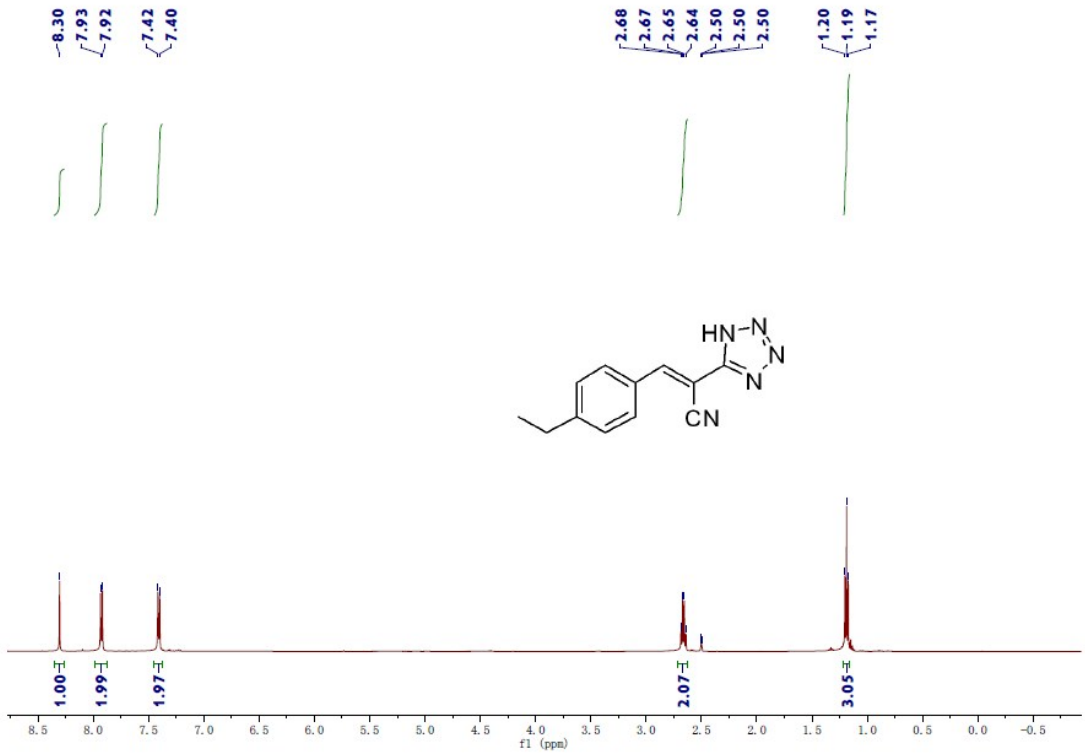
^{1}H NMR spectrum of the product of 6i (DMSO, 500 MHz)



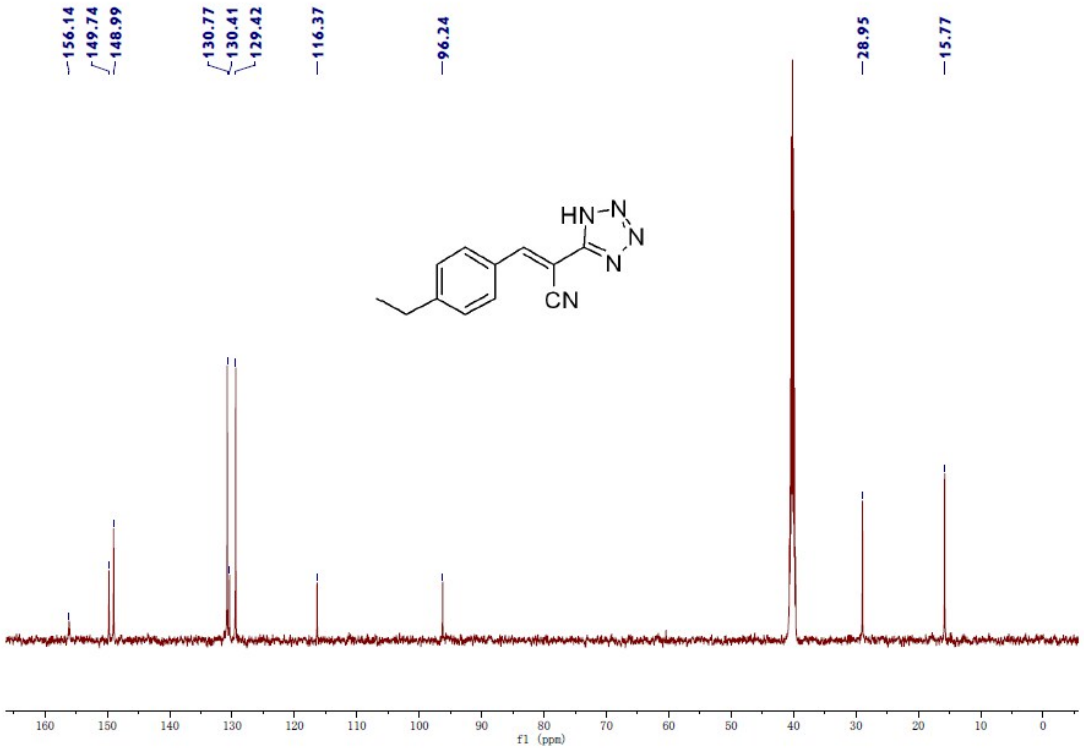
^{13}C NMR spectrum of the product of 6k (DMSO, 126 MHz)



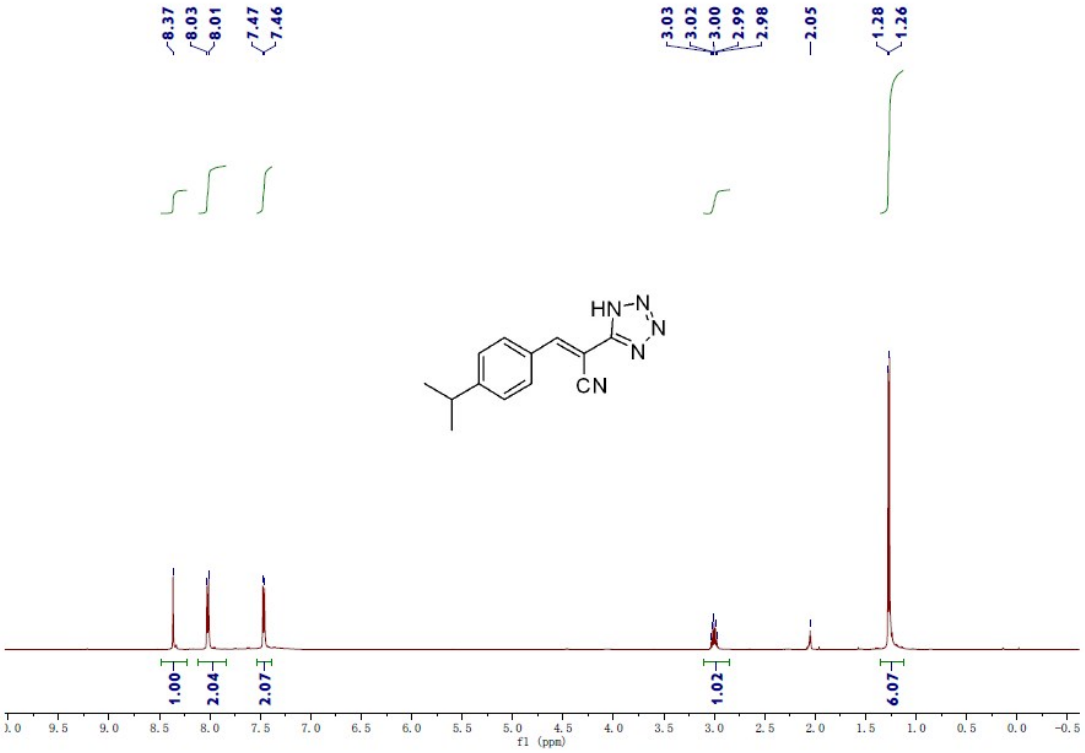
^{1}H NMR spectrum of the product of 6j (DMSO, 500 MHz)



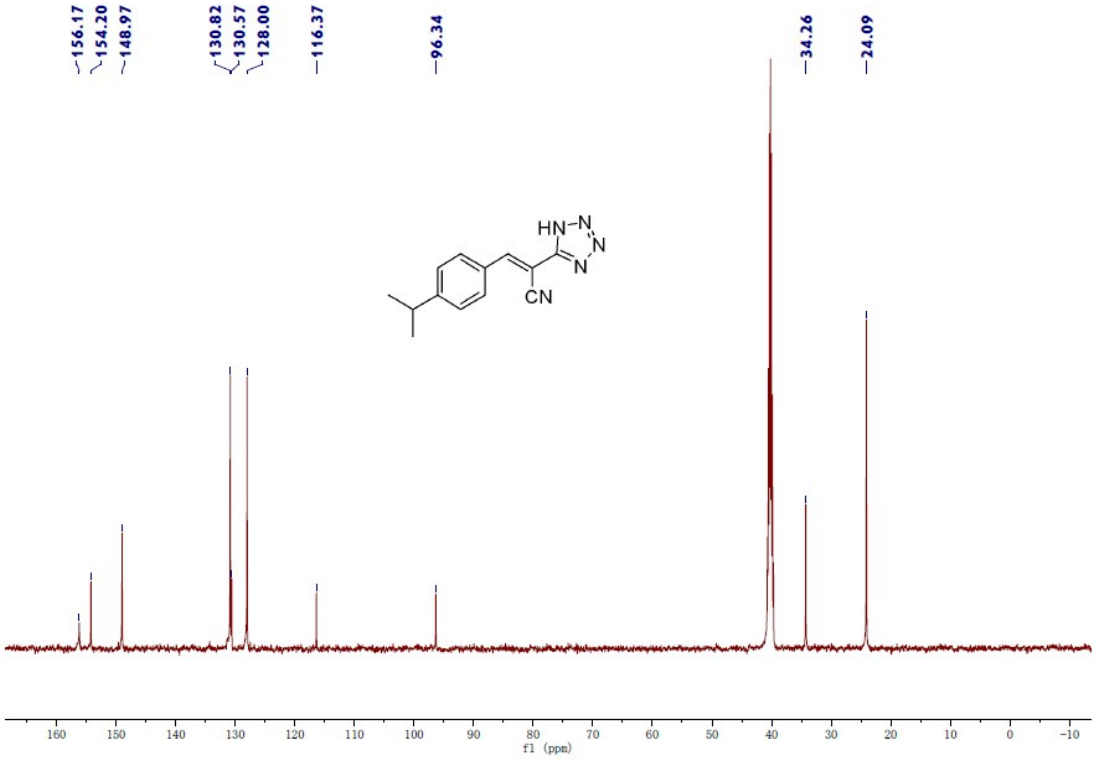
^{13}C NMR spectrum of the product of 6j (DMSO, 126 MHz)



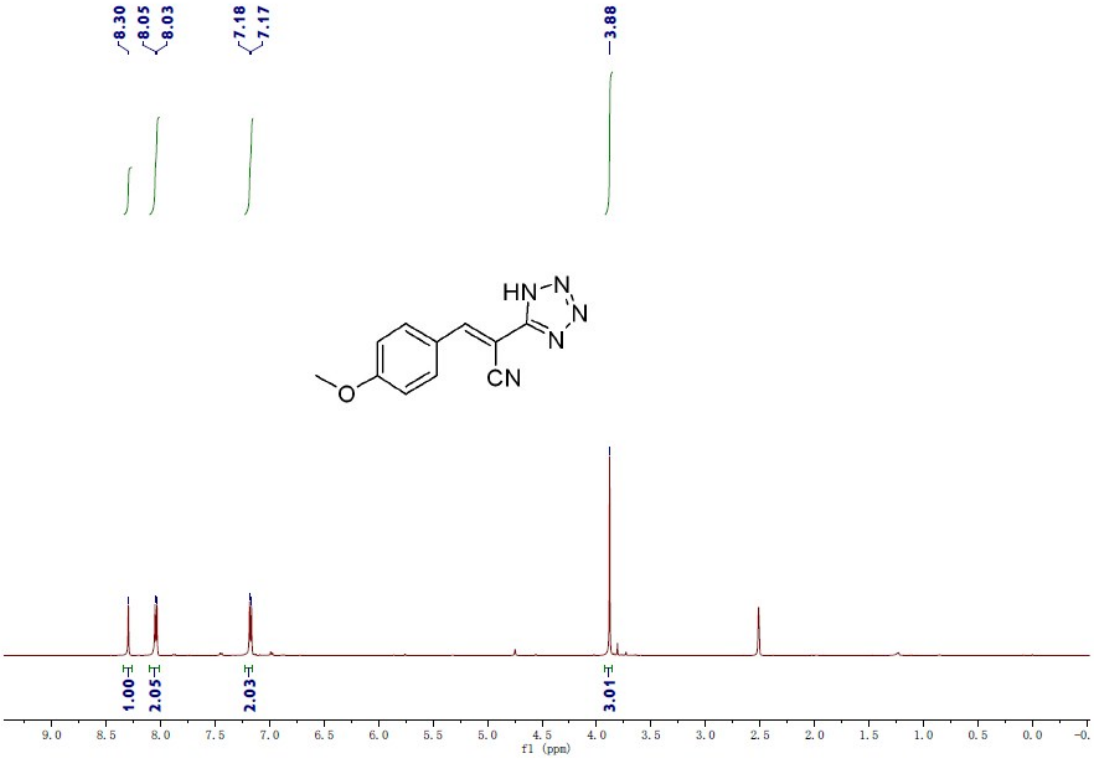
^{1}H NMR spectrum of the product of 6k (Acetone, 500 MHz)



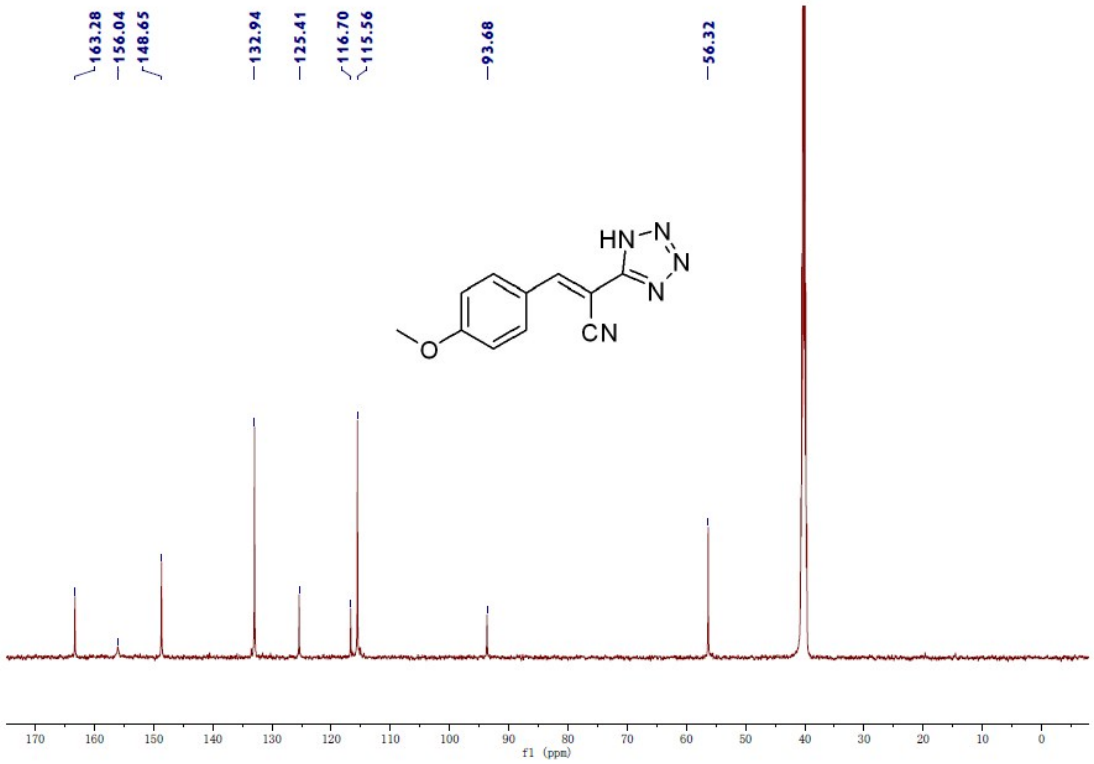
^{13}C NMR spectrum of the product of 6i (DMSO, 126 MHz)



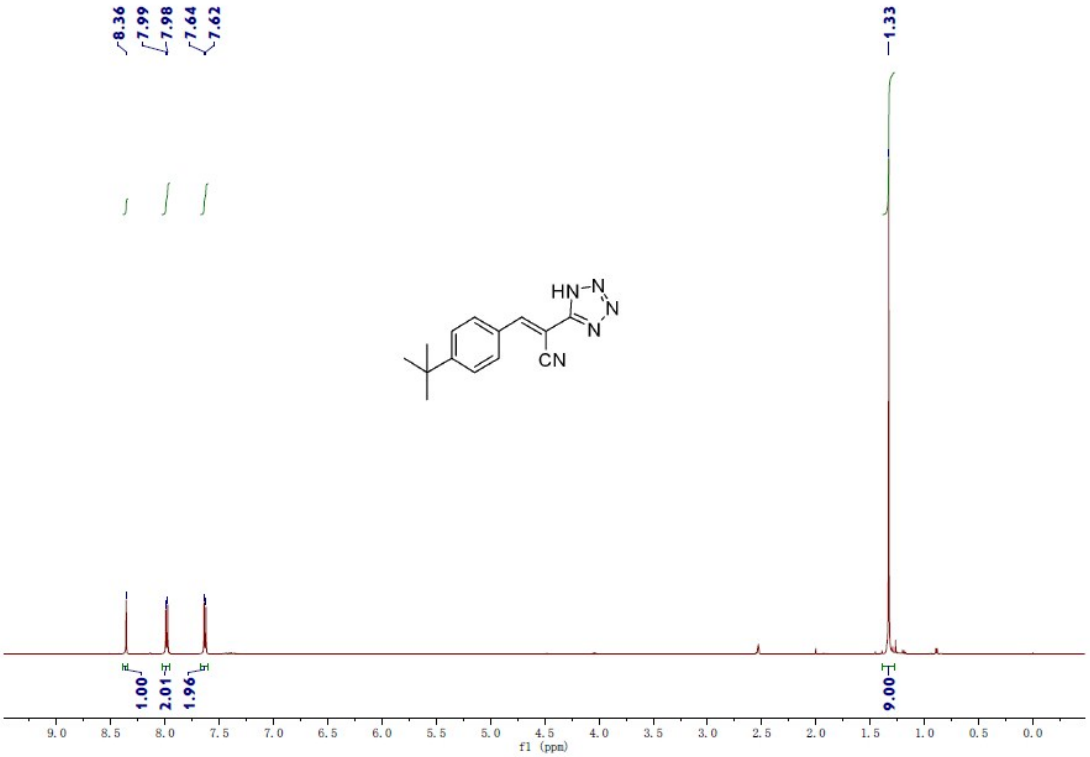
^{1}H NMR spectrum of the product of 6l (DMSO, 500 MHz)



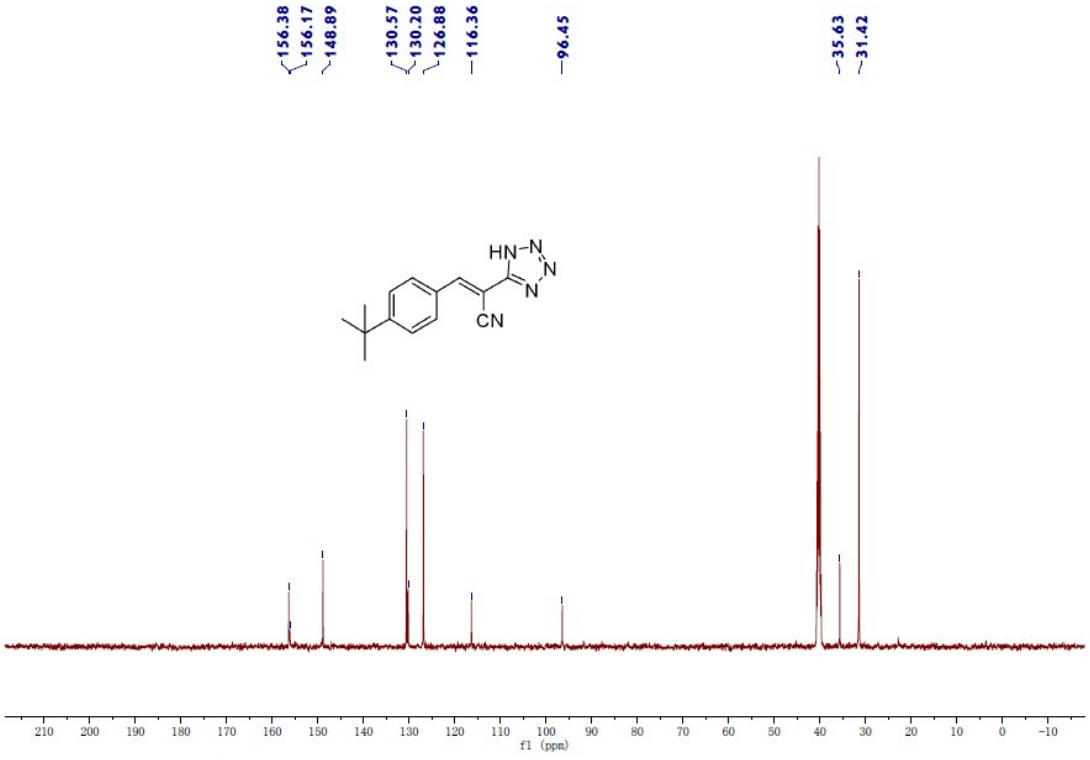
^{13}C NMR spectrum of the product of 6l (DMSO, 126 MHz)



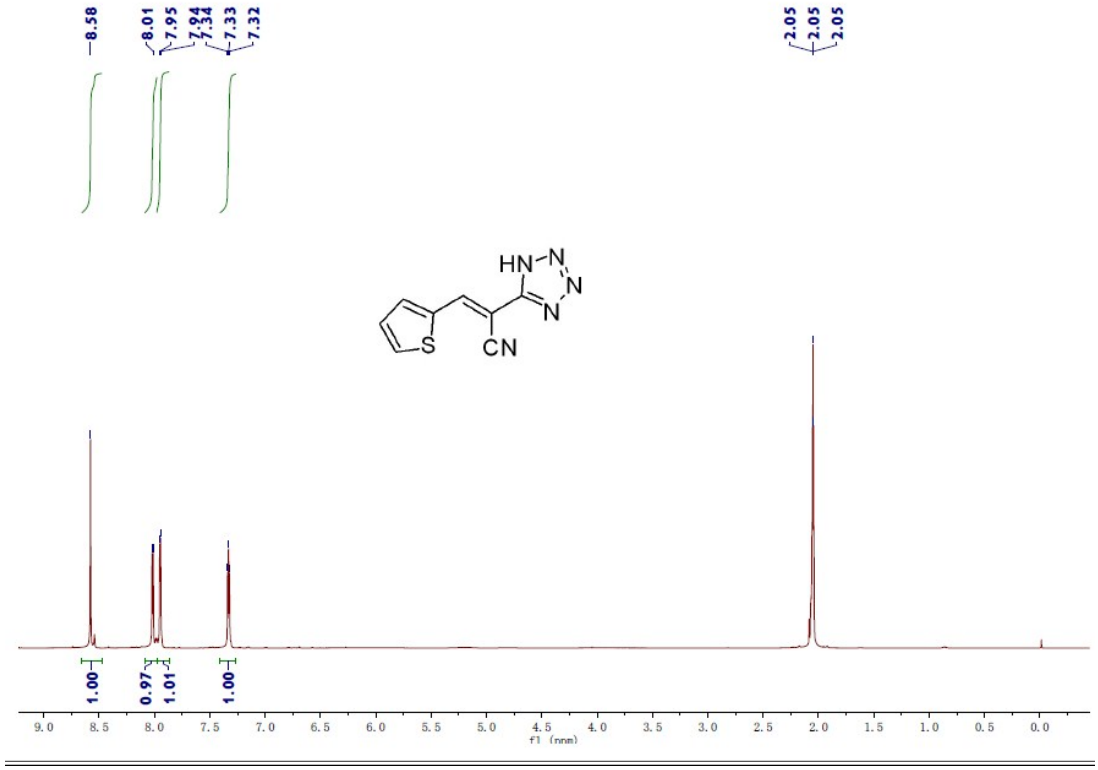
^{1}H NMR spectrum of the product of 6m (DMSO, 500 MHz)



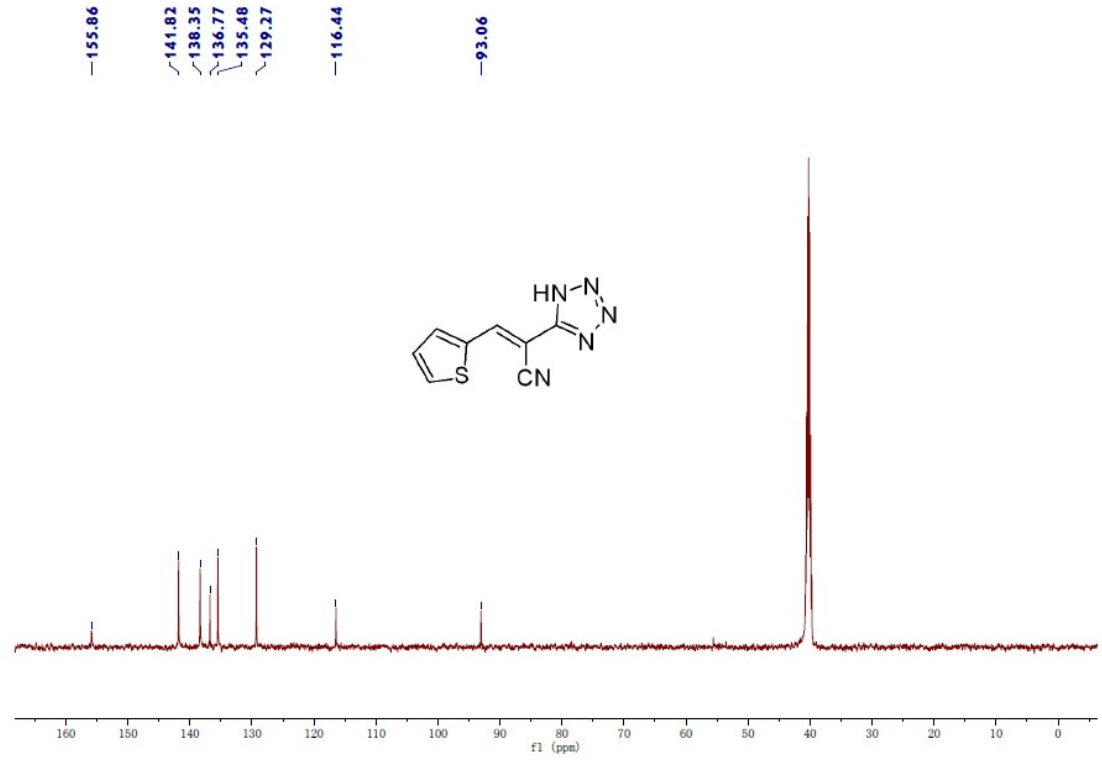
^{13}C NMR spectrum of the product of 6m (DMSO, 126 MHz)



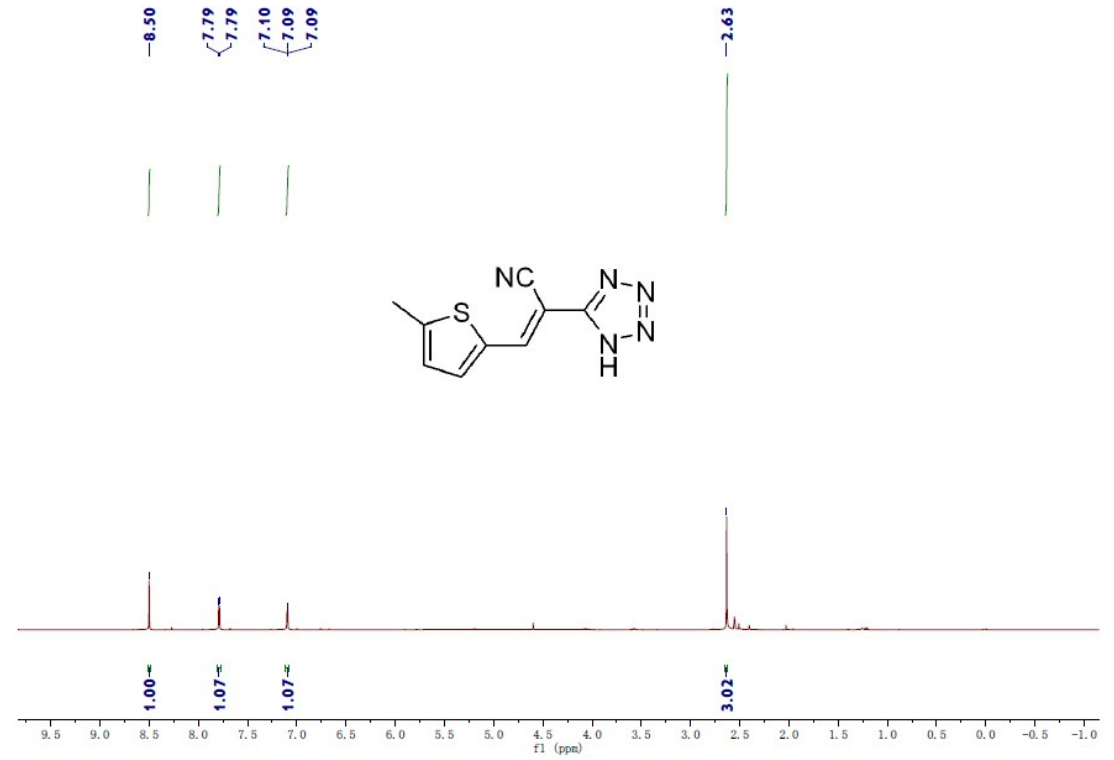
^{1}H NMR spectrum of the product of 6n (Acetone, 500 MHz)



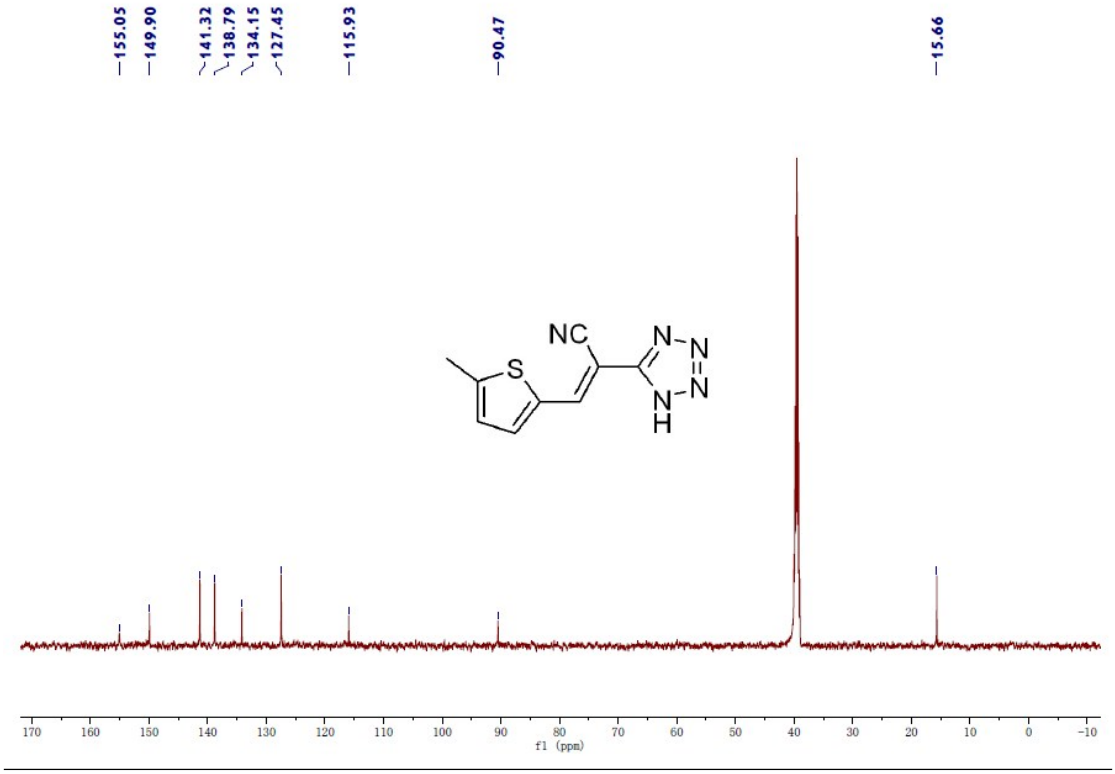
^{13}C NMR spectrum of the product of 6n (DMSO, 126 MHz)



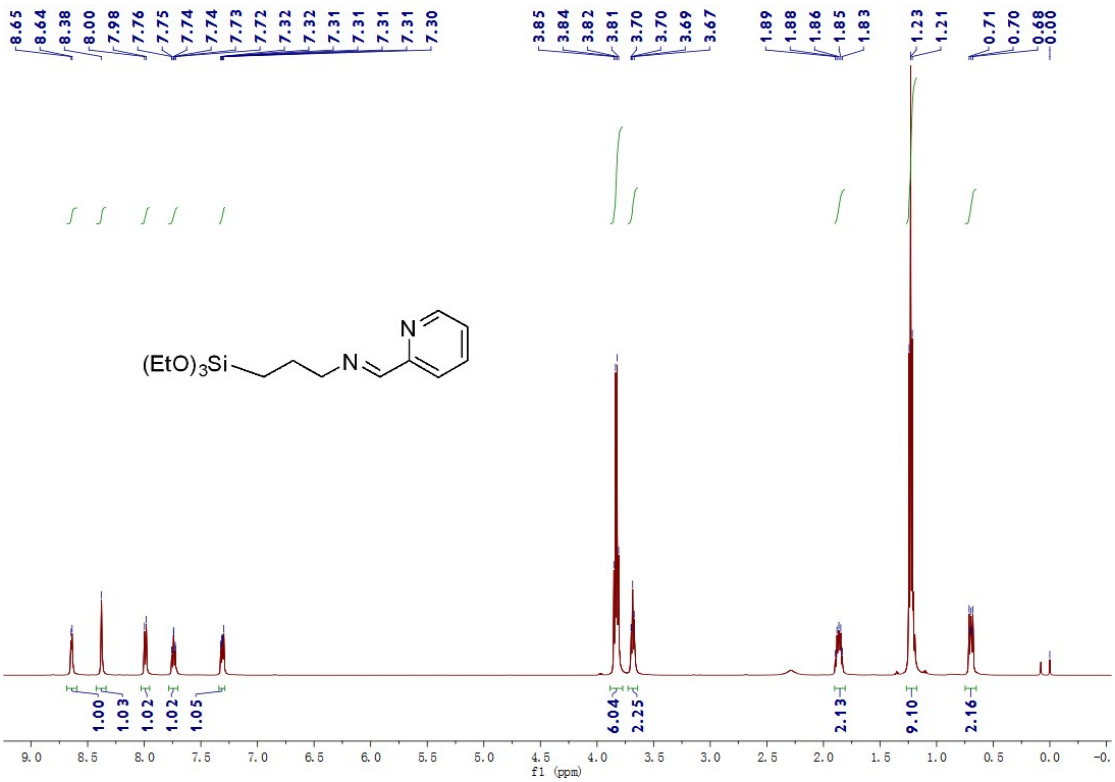
^{1}H NMR spectrum of the product of 6o (DMSO, 500 MHz)



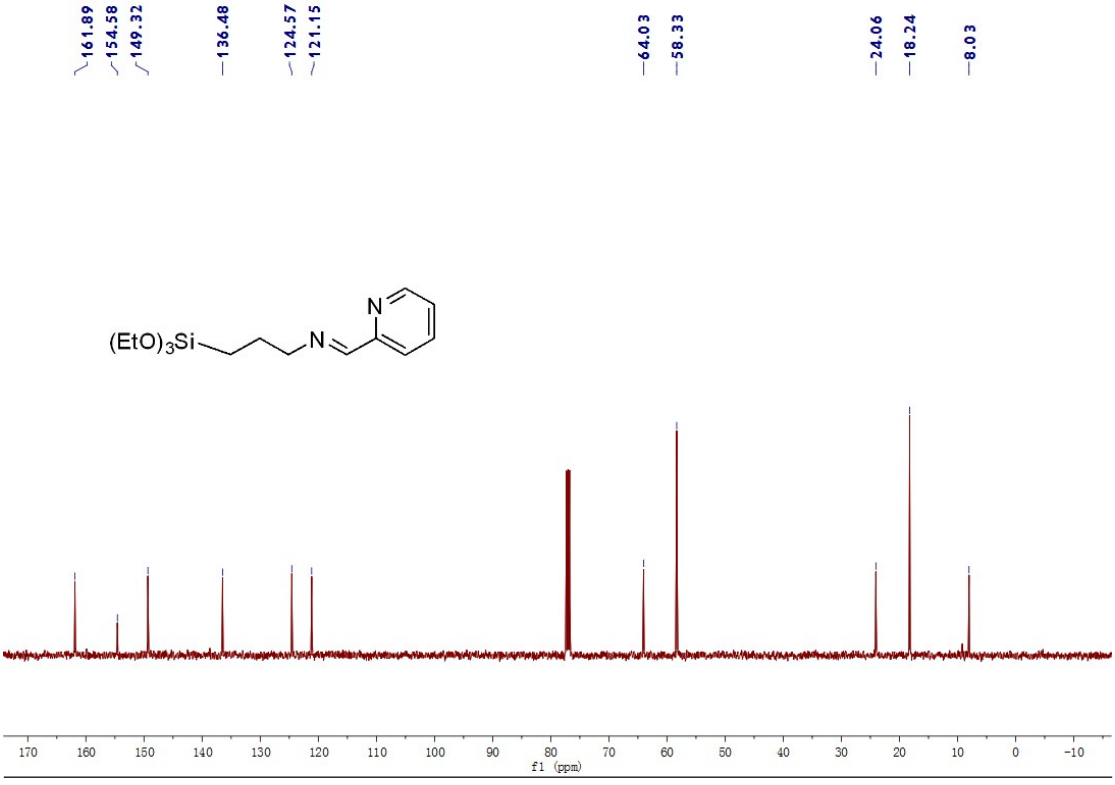
^{13}C NMR spectrum of the product of 6o (DMSO, 126 MHz)



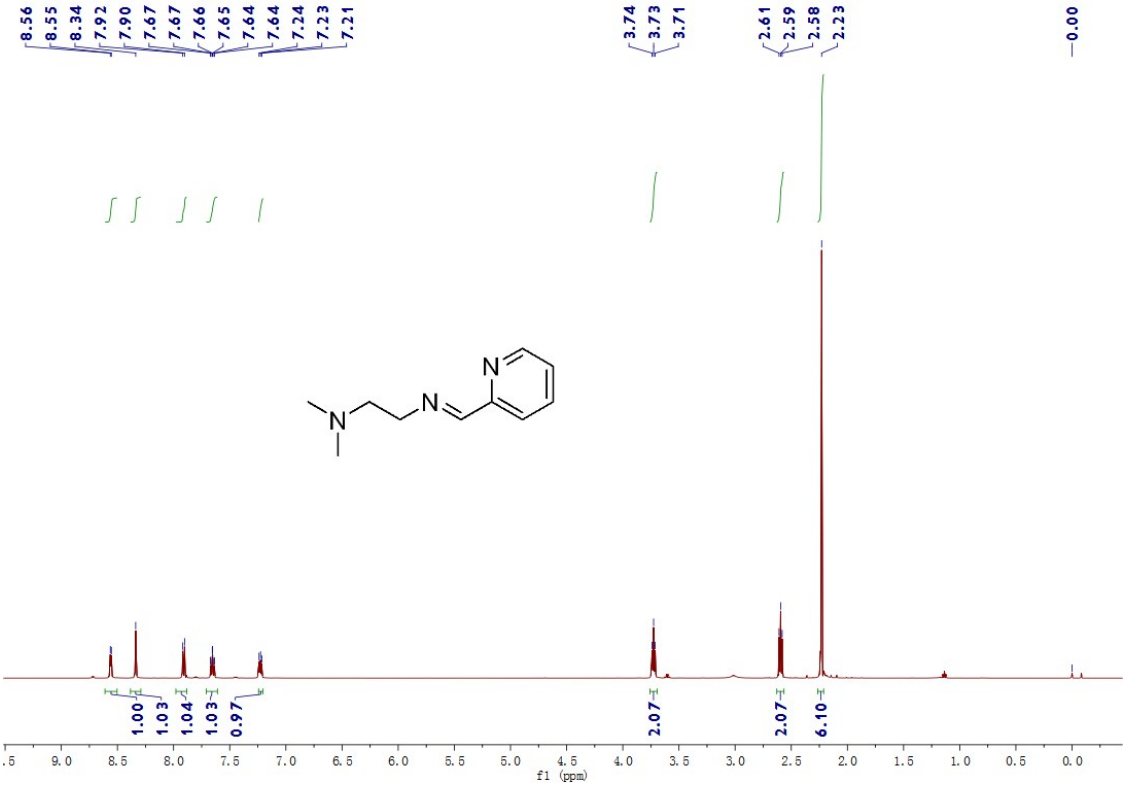
^{1}H NMR spectrum of the product of 7 ($CDCl_3$, 500 MHz)



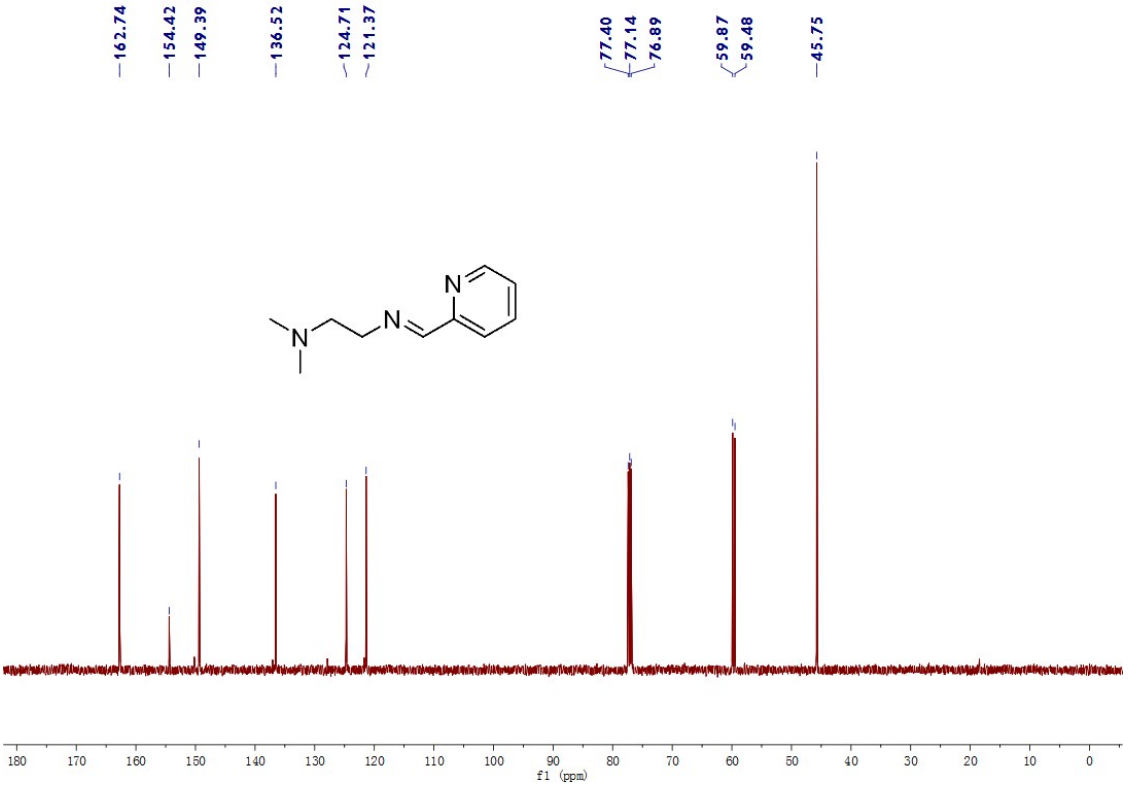
^{13}C NMR spectrum of the product of 7 ($CDCl_3$, 126 MHz)



^{1}H NMR spectrum of the product of 10 ($CDCl_3$, 500 MHz)



^{13}C NMR spectrum of the product of 10 ($CDCl_3$, 126 MHz)



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[2] K. Mandai, T. Korenaga, T. Ema, T. Sakai, M. Furutani, H. Hashimoto and J. Takada, *Tetrahedron Lett.*, **2012**, *53*, 329-332.
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