Supporting Information

C–C bond cleavage arylation and alkenylation of cyclobutanone oxime ethers via photoredox/nickel catalysis

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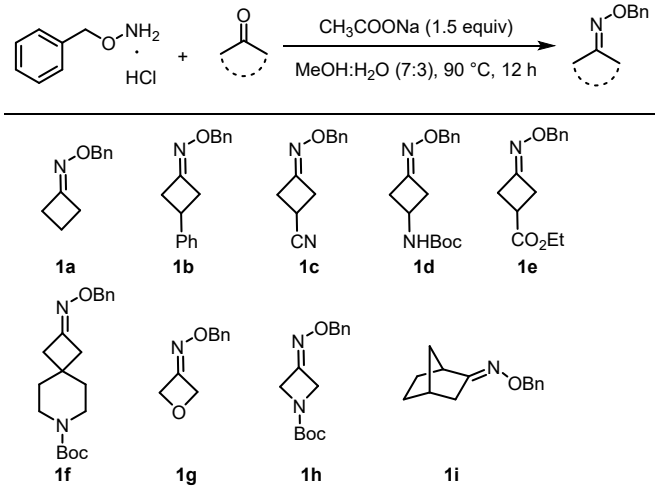
1. General Information

Unless otherwise noted, all reagents and solvents were obtained from commercial suppliers and were used without further purification. Analytical thin-layer chroma-tography (TLC) was performed on glass plates coated with 0.25 mm 230–400 mesh silica gel containing a fluores-cent indicator. Visualization was accomplished by exposure to a UV lamp. All the products in this article are compatible with standard silica gel chromatography. Column chromatography was performed on silica gel (200–300 mesh). Eluent generally contained ethyl acetate (EA), petroleum ether (PE). NMR spectra were measured on a Bruker Ascend 400 spectrometer and chemical shifts (δ) are reported in parts per million (ppm). ^{1}H NMR spectra were recorded at 400 MHz in NMR solvents and referenced internally to corresponding solvent resonance, and ^{13}C NMR spectra were recorded at 101 MHz and referenced to corre-sponding solvent resonance. Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Infrared spectra were collected on a Thermo Fisher Nicolet 6700 FT-IR spectrometer using ATR (Attenuated Total Reflectance) method. Absorption maxima (ν max) are reported in wavenumbers (cm^{-1}). High resolution mass spectra (HRMS) were acquired on Thermo Scientific LTQ Orbitrap XL with an ESI source. Melting points were measured with a micro-melting point apparatus.

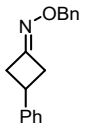
2. Starting Materials

2.1 General Procedure A for the Synthesis of Cyclobutanone *O*-benzyl Oxime Ether Derivatives[1]

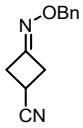


To a 250 mL dried tube was added the mixture of ketone (10 mmol), alkoxyamine hydrochloride (1.5 equiv), NaOAc (1.5 equiv) in MeOH (21 mL) and H_2O (9.0 mL) successively. The mixture was stirred at room temperature for 12 hours under 90 ℃. After the reaction was completed, the mixture diluted with H_2O (45 mL), and extracted with EtOAc (3 × 45 mL). The organic extract was washed with brine and dried over anhydrous $MgSO_4$. After removal of the EtOAc in vacuum, the crude product was purified by column chromatography on silica gel with hexanes or petroleum ether/ethyl acetate to give oximes.

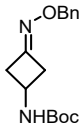
(1a) Cyclobutanone O-benzyl oxime Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.30 (m, 5H), 5.09 (s, 2H), 2.99 – 2.90 (m, 4H), 2.04 – 1.96 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.3, 138.3, 128.4, 128.1, 127.7, 75.6, 31.7, 31.3, 14.6. HRMS (APCI) m/z calcd for $C_{11}H_{14}ON$ $[M+H]^+$ 176.1075, found 176.1070. IR (cm^{-1}): 3718, 3149, 2933, 2860, 2667, 2522, 2407, 2291, 2246, 2166, 2033, 1809, 1497, 1364, 1165, 1022, 858, 733, 604, 430.



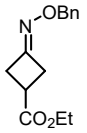
(1b) 3-Phenylcyclobutan-1-one O-benzyl oxime Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.34 (m, 7H), 7.30 – 7.27 (m, 3H), 5.15 (s, 2H), 3.67 – 3.59 (m, 1H), 3.52 – 3.36 (m, 2H), 3.12 – 3.04 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.0, 144.2, 138.2, 128.7, 128.5, 128.2, 127.9, 126.6, 126.5, 75.8, 39.7, 39.0, 33.0. HRMS (APCI) m/z calcd for $C_{17}H_{18}ON$ $[M+H]^+$ 252.1388, found 252.1383. IR (cm^{-1}): 3724, 3028, 2926, 2862, 2671, 2522, 2407, 2291, 2245, 2166, 2037, 1807, 1495, 1362, 1180, 1010, 851, 746, 696, 604, 428.



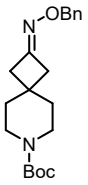
(1c) 3-((Benzyloxy)imino)cyclobutane-1-carbonitrile Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.30 (m, 5H), 5.06 (s, 2H), 3.42 – 3.25 (m, 4H), 3.18 – 3.10 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.8, 136.4, 127.4, 127.2, 127.0, 120.0, 75.2, 36.2, 35.9, 15.0. HRMS (APCI) m/z calcd for $C_{12}H_{13}ON_2$ $[M+H]^+$ 201.1028, found 201.1022. IR (cm^{-1}): 3719, 3163, 3062, 3036, 2939, 2864, 2716, 2540, 2413, 2243, 2164, 2041, 1813, 1699, 1499, 1454, 1400, 1364, 1180, 1026, 866, 743, 698, 604, 449.



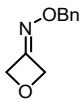
(1d) Tert-butyl (3-((benzyloxy)imino)cyclobutyl)carbamate Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 5H), 5.04 (s, 2H), 4.78 (s, 1H), 4.20 (s, 1H), 3.38 – 3.27 (m, 2H), 2.82 - 2.71 (m, 2H), 1.44 (m, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.1, 153.4, 138.0, 128.4, 128.2, 128.1, 127.8, 79.8, 75.8, 40.5, 39.8, 28.4. HRMS (APCI) m/z calcd for $C_{16}H_{23}O_3N_2$ $[M+H]^+$ 291.1709, found 297.1703. IR (cm^{-1}): 3730, 3350, 3146, 2937, 2677, 2521, 2413, 2291, 2247, 2166, 2041, 1933, 1809, 1682, 1526, 1371, 1275, 1165, 991, 862, 750, 652, 600, 426.



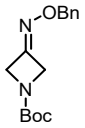
(1e) Ethyl 3-((benzyloxy)imino)cyclobutane-1-carboxylate Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 5H), 5.08 (s, 2H), 4.18 (q, J = 7.2 Hz, 2H), 3.21 – 3.09 (m, 5H), 1.27 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.0, 154.1, 138.0, 128.4, 128.1, 127.8, 75.8, 61.0, 35.5, 35.2, 31.4, 14.2. HRMS (APCI) m/z calcd for $C_{14}H_{18}O_3N$ $[M+H]^+$ 248.1287, found 248.1281. IR (cm^{-1}): 3707, 3148, 3036, 2984, 2939, 2866, 2725, 2523, 2407, 2291, 2243, 2164, 2029, 1728, 1495, 1369, 1346, 1184, 1018, 864, 733, 698, 604, 428.



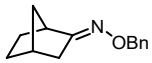
(1f) Tert-butyl 2-((benzyloxy)imino)-7-azaspiro[3.5]nonane-7-carboxylate Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 5H), 5.05 (s, 2H), 3.40 – 3.28 (m, 4H), 2.66 – 2.61 (m, 4H), 1.57 (t, J = 5.6 Hz, 4H), 1.45 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.8, 154.8, 138.0, 128.4, 128.1, 127.8, 79.5, 75.7, 41.8, 41.2, 36.4, 33.2, 28.5. HRMS (APCI) m/z calcd for $C_{20}H_{29}O_3N_2$ $[M+H]^+$ 345.2178, found 345.2173. IR (cm^{-1}): 3718, 3163, 2920, 2849, 2679, 2513, 2411, 2289, 2249, 2164, 2042, 1933, 1838, 1693, 1479, 1425, 1367, 1242, 1171, 1140, 1024, 970, 852, 746, 606, 426.



(1g) Oxetan-3-one O-benzyl oxime Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 5H), 5.31 – 5.29 (m, 2H), 5.27 – 5.25 (m, 2H), 5.07 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.0, 137.3, 128.5, 128.3, 128.1, 79.2, 79.0, 76.4. HRMS (APCI) m/z calcd for $C_{10}H_{12}O_2N$ $[M+H]^+$ 178.0868, found 178.0863. IR (cm^{-1}): 3724, 3146, 3065, 2935, 2862, 2704, 2521, 2410, 2297, 2249, 2162, 2031, 1807, 1497, 1366, 1205, 1015, 955, 864, 746, 700, 606, 465.

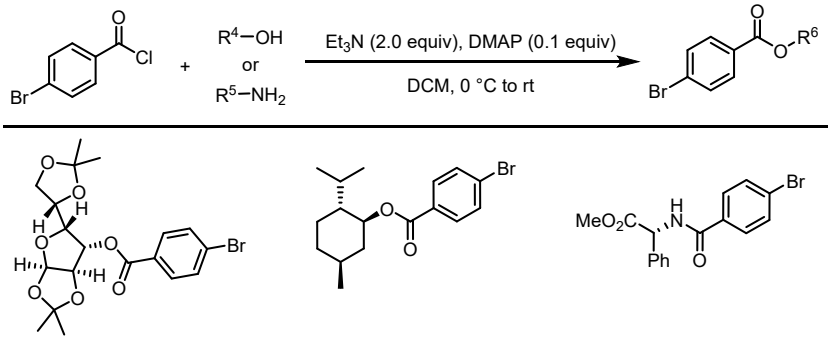


(1h) Tert-butyl 3-((benzyloxy)imino)azetidine-1-carboxylate Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 5H), 5.08 (s, 2H), 4.62 – 4.58 (m, 4H), 1.46 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.2, 148.2, 137.4, 128.5, 128.2, 128.1, 80.5, 76.4, 58.3, 28.3. HRMS (APCI) m/z calcd for $C_{15}H_{21}O_3N_2$ $[M+H]^+$ 277.1552, found 277.1547. IR (cm^{-1}): 3717, 3161, 3150, 2980, 2934, 2874, 2718, 2536, 2403, 2295, 2247, 2162, 2039, 1936, 1703, 1477, 1369, 1254, 1151, 1009, 918, 862, 741, 698, 613.

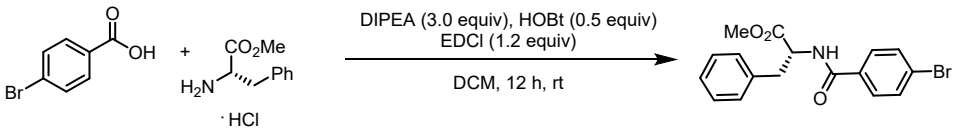


(1i) (1S,4R,E)-Bicyclo[2.2.1]heptan-2-one O-benzyl oxime Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 5H), 5.07 (s, 2H), 2.88 (d, J = 3.6 Hz, 1H), 2.50 – 2.48 (m, 1H), 2.33 – 2.23 (m, 1H), 2.14 – 1.98 (m, 1H), 1.78 – 1.61 (m, 2H), 1.51 – 1.27 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.7, 138.4, 128.3, 127.9, 127.6, 75.3, 42.4, 39.0, 35.6, 35.5, 27.8, 27.2. HRMS (APCI) m/z calcd for $C_{14}H_{18}ON$ $[M+H]^+$ 216.1388, found 216.1383. IR (cm^{-1}): 3716, 3431, 3165, 3087, 3066, 3030, 2964, 2874, 2715, 2536, 2409, 2291, 2245, 2168, 2039, 1942, 1809, 1497, 1452, 1364, 1209, 1159, 1161, 1092, 1026, 903, 864, 825, 737, 698, 613, 470.

2.2 General Procedure B for the Synthesis of Aryl Bromide Derivatives[2]

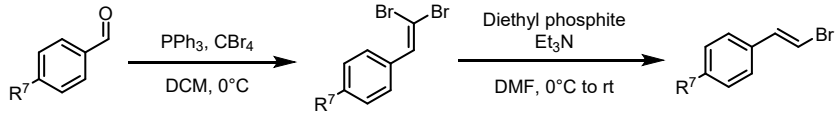


To a solution of alcohol or amine (5.0 mmol, 1.0 equiv), DMAP (0.5 mmol, 0.1 equiv) and Et_3N (10 mmol, 2.0 equiv) in DCM (10 mL) was added the solution of 4-bromobenzoylchloride (5.0 mmol, 1.0 equiv) in DCM (10 mL) dropwise using syringe at 0 °C. After stirring for 30 minutes, the mixture was allowed to stir at room temperature overnight. Then the mixture was diluted with saturated NH_4Cl solution (20 mL), and extracted with DCM (3 × 10 mL). The organic layer was washed with brine, dried over $MgSO_4$ and evaporated. The residue was purified with flash column chromatography to give the desired aryl bromide.



2,4-bromobenzoic acid (1.00 g, 5.0 mmol, 1.0 equiv), methyl *L*-phenylalaninate hydrochloride (0.99 g, 6.0 mmol, 1.2 equiv) and *N,N*-diisopropylethylamine (DIPEA) (1.94 g, 15 mmol, 3.0 equiv) were dissolved in DCM (15 mL). Then, 1-hydroxybenzotriazole (HOBt) (0.34 g, 2.5 mmol, 0.5 equiv) and *N*-(3-dimethylaminopropyl)-*N*'-ethylcarbodiimide hydrochloride (EDCI) (1.15 g, 6.0 mmol, 1.2 equiv) were added. The reaction was stirred overnight at room temperature. After the reaction was completed, the resulting mixture was diluted with DCM and washed with HCl (2.0 M, 10 mL), water (15 mL), brine (15 mL). The organic phase was dried over $MgSO_4$, filtered and the solvent was evaporated under reduced pressure. The resulting crude was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1 – 3:1) to give methyl (4-bromobenzoyl) phenylalaninate as a white solid in 89% yield.

2.3 General Procedure C for the Synthesis of Vinyl Bromides[3]



Step 1: A flame-dried round-bottom flask equipped with a stir bar under argon was charged with the corresponding aldehyde (10 mmol, 1.0 equiv), CBr_4 (15 mmol, 1.5 equiv) and DCM (80 mL). The reaction mixture was cooled at 0 °C, then a solution of PPh_3 (30 mmol, 3 equiv) in DCM (80 mL) was added dropwise over 20 minutes. After another 1 hour at 0 °C, the mixture was concentrated under reduce pressure to half of the volume. Next, pentane was added and triphenylphosphine oxide precipitated out. The mixture was filtered and concentrated under reduced pressure. Pentane was added again to further precipitate the triphenylphosphine oxide. After filtration and evaporation of the solvent, the crude dibromide was used directly in the next step without any further purification.

Step 2: To a mixture of the above dibromide and diethyl phosphite (30 mmol, 3.0 equiv) in DMF (10 mL) was added Et_3N (30 mmol, 3.0 equiv) at 0 °C. The reaction was then warmed to room temperature and stirred overnight. The mixture was quenched with water. The aqueous layer was extracted with DCM. The combined organics were washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The products were purified by chromatography.

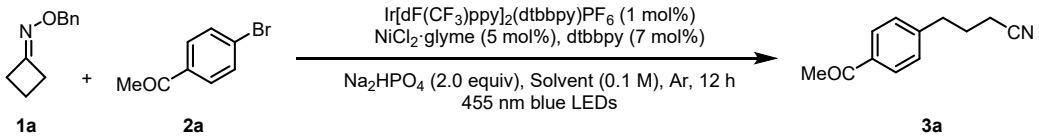
3. Optimization of Reaction Conditions

3.1 General Procedure for Radical Ring-Opening Arylation

To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added 1-(4-bromophenyl)ethan-1-one **2a** (0.2 mmol, 1.0 equiv), photocatalyst (0.002 mmol, 1 mol%), catalyst (0.01 mmol, 5 mol%), ligand (0.014 mmol, 7 mol%) and base (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in solvent (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **3a**.

3.2 Optimization of Reaction 1a with 2a[a]

Screening of Solvents

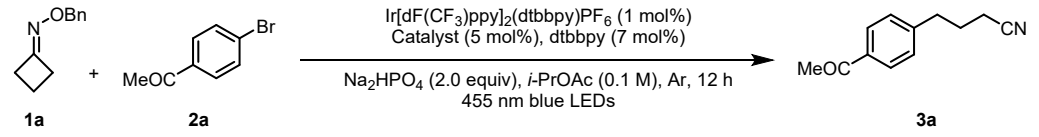


1a **2a** **3a**

Entry	Solvent	Yield (%)
1	***i*-PrOAc**	**49**
2	*n*-Butyl acetate	38
3	MTBE	<20
4	MeCN	<20
5	DCM	NR
6	DMSO	NR
7	EtOH	23
8	DMA	39
9	DMF	24
10[b]	***i*-PrOAc** +100 μL H_2O	22

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **2a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), $NiCl_2$·glyme (5 mol%), dtbbpy (7 mol%), Na_2HPO_4 (2.0 equiv) in Solvent (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. [b]Standard conditions. Isolated yields.

Screening of Catalysts

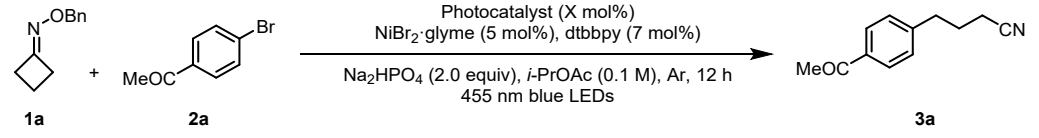


1a **2a** **3a**

Entry	Catalyst	Yield (%)
1	**$NiBr_2$·glyme**	**86**
2	$NiCl_2(PCy_3)_2$	NR
3	$NiCl_2$(dppp)	31
4	$NiCl_2$(dppe)	43
5	$NiCl_2$(dtppe)	67
6	$NiCl_2$(dppf)	<20

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **2a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), Catalyst (5 mol%), dtbbpy (7 mol%), Na_2HPO_4 (2.0 equiv) in *i*-PrOAc (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Photocatalysts



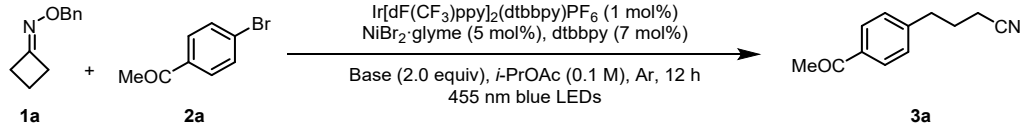
1a **2a** **3a**

Entry	Photocatalyst (X mol%)	Yield (%)
1	**$Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1)**	**86**
2	4CzIPN (2)	NR
3	$Ir(ppy)_3$ (1)	NR
4	TXO (10)	NR
5	Rhodamine B (5)	NR
6	Rose Bengal (5)	NR
7	EosinY (5)	NR

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **2a** (0.2 mmol, 1.0 equiv), Photocatalyst (X mol%), $NiBr_2$·glyme (5 mol%), dtbbpy (7

mol%), Na_2HPO_4 (2.0 equiv) in *i*-PrOAc (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Bases

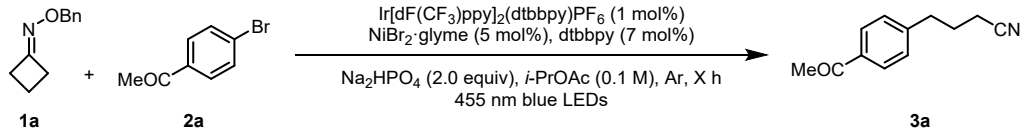


1a **2a** **3a**

Entry	Base	Yield (%)
1	**Na_2HPO_4**	**86**
2	Na_2CO_3	84
3	Na_3PO_4	50
4	LiOAc	45
5	^{t}BuOK	26
6	Cs_2CO_3	49

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **2a** (0.2 mmol, 1.0 equiv), Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ (1 mol%), NiBr$_2$·glyme (5 mol%), dtbbpy (7 mol%), base (2.0 equiv) in *i*-PrOAc (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Time



1a **2a** **3a**

Entry	Time (X h)	Yield (%)
1	**12**	**86**
2	3	20
3	6	40
4	9	65
5	15	89

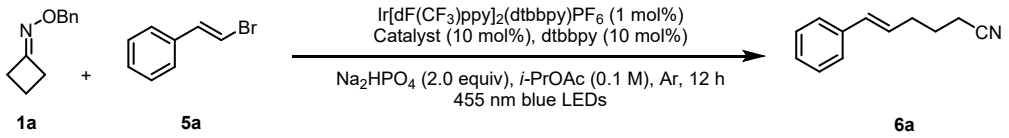
[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **2a** (0.2 mmol, 1.0 equiv), Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$ (1 mol%), NiBr$_2$·glyme (5 mol%), dtbbpy (7 mol%), Na_2HPO_4 (2.0 equiv) in *i*-PrOAc (2.0 mL) were irradiated by 10 W blue LEDs for X h under Ar at room temperature. Isolated yields.

3.3 General Procedure for Radical Ring-Opening Alkenylation

To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added (*E*)-(2-bromovinyl)benzene **5a** (0.2 mmol, 1.0 equiv), phtocatalyst (0.002 mmol, 1 mol%), catalyst (0.02 mmol, 10 mol%), ligand (0.02 mmol, 10 mol%) and base (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.5 mmol, 2.5 equiv) in sovent (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **6a**.

3.4 Optimization of Reaction 1a with 5a[a]

Screening of Catalysts

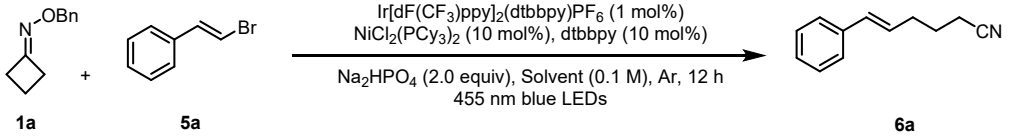


1a **5a** **6a**

Entry	Catalyst	Yield (%)
1	**$NiCl_2(PCy_3)_2$**	**28**
2	$NiBr_2$·glyme	17
3	$NiCl_2$(dppp)	12
4	$NiCl_2$(dppe)	17
5	$NiCl_2$(dtppe)	14
6	$NiCl_2$(dppf)	Trace

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (1 mol%), Catalyst (10 mol%), dtbbpy (10 mol%), Na_2HPO_4 (2.0 equiv) in *i*-PrOAc (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Solvents

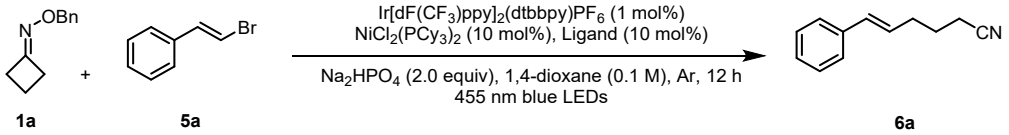


1a **5a** **6a**

Entry	Solvent	Yield (%)
1	**1,4-dioxane**	**55**
2	EA	17
3	DMC	25
4	MeCN	NR
5	*i*-PrOAc	28
6	MeOH	Trace
7	DCM	Trace
8	TFE	NR
9	DMSO	NR
10	NMP	NR
11	DMF	Trace
12	MTBE	36
13	DME	46
14	THF	Trace

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (1 mol%), $NiCl_2(PCy_3)_2$ (10 mol%), dtbbpy (10 mol%), Na_2HPO_4 (2.0 equiv) in Solvent (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Ligands

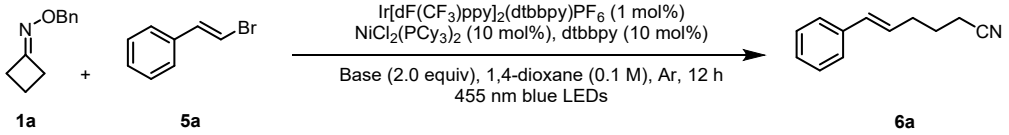


1a **5a** **6a**

Entry	Ligand	Yield (%)
1	**dtbbpy**	**55**
2	4,4'-dimethoxy-2,2'-bipyridyl	Trace
3	6,6'-dihydroxy-2,2'-bipyridyl	28
4	4,4'-dimethyl-2,2'-bipyridyl	Trace
5	5,5'-dimethyl-2,2'-bipyridyl	35
6	4-bromo-2-(4-bromopyridin-2-yl)pyridine	25
7	bpy	15
8	6,6'-dimethyl-2,2'-dipyridyl	17
9	4,7-diphenyl-1,10-phenanthroline	27
10	4,7-dimethoxy-1,10-phenanthroline	Trace
11	1,10-phenanthroline	Trace
12	2,2'-biquinoline	Trace
13	2,2,6,2-terpyridine	Trace
14	3,4,7,8-tetramethyl-1,10-phenanthroline	Trace

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), $NiCl_2(PCy_3)_2$ (10 mol%), Ligand (10 mol%), Na_2HPO_4 (2.0 equiv) in 1,4-dioxane (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Bases

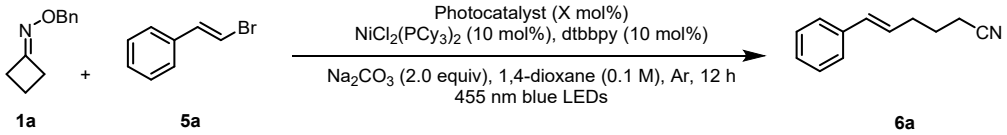


1a **5a** **6a**

Entry	Base	Yield (%)
1	**Na_2CO_3**	**75**
2	Cs_2CO_3	30
3	K_2HPO_4	55
4	$NaHCO_3$	42
5	K_2CO_3	40
6	$KHCO_3$	46
7	NH_4CO_3	NR
8	Li_2CO_3	41
9	Na_3PO_4	37
10	^{t}BuOK	NR
11	$NaPO_2$	Trace
12	K_3PO_4	30
13	DBU	Trace
14	NaOAc	43
15	Et_3N	Trace
16	KOAc	Trace
17	LiOAc	28
18	MeONa	NR

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), $NiCl_2(PCy_3)_2$ (10 mol%), dtbbpy (10 mol%), base (2.0 equiv) in 1,4-dioxane (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Photocatalysts

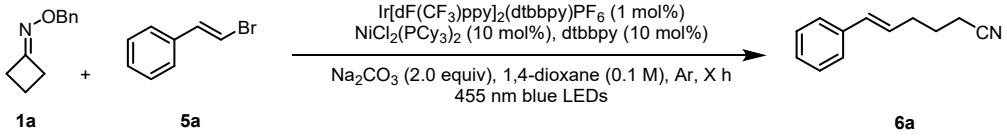


1a **5a** **6a**

Entry	Photocatalyst (X mol%)	Yield (%)
1	**$Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1)**	**75**
2	4CzIPN (2)	NR
3	$Ir(ppy)_3$ (1)	NR
4	TXO (10)	NR
5	Rhodamine B (5)	NR
6	Rose Bengal (5)	NR
7	EosinY (5)	NR

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), Phtotcatalyst (X mol%), $NiCl_2(PCy_3)_2$ (10 mol%), dtbbpy (10 mol%), Na_2CO_3 (2.0 equiv) in 1,4-dioxane (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

Screening of Time

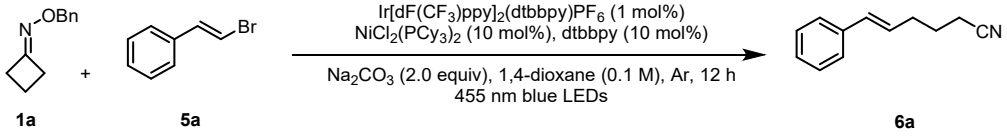


1a **5a** **6a**

Entry	Times (X h)	Yield (%)
1	**12**	**75**
2	6	42
3	18	71
4	24	62

[a] Reaction conditions: **1a** (0.4 mmol, 2.0 equiv), **5a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), $NiCl_2(PCy_3)_2$ (10 mol%), dtbbpy (10 mol%), Na_2CO_3 (2.0 equiv) in 1,4-dioxane (2.0 mL) were irradiated by 10 W blue LEDs for X h under Ar at room temperature. Isolated yields.

Screening of Substrate Ratio

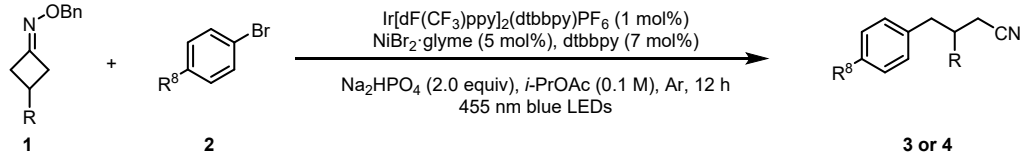


1a **5a** **6a**

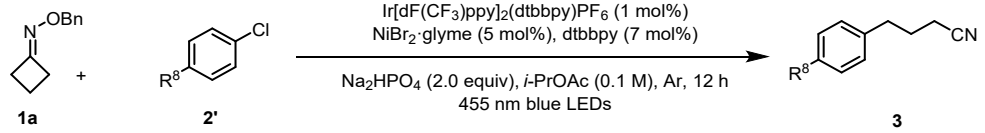
Entry	Ratio (**5a**:**1a**)	Yield (%)
1	**1:2.5**	**88**
2	1:2	75
3	1:3	90

[a] Reaction conditions: **1a** (X mmol), **5a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (1 mol%), $NiCl_2(PCy_3)_2$ (10 mol%), dtbbpy (10 mol%), Na_2CO_3 (2.0 equiv) in 1,4-dioxane (2.0 mL) were irradiated by 10 W blue LEDs for 12 h under Ar at room temperature. Isolated yields.

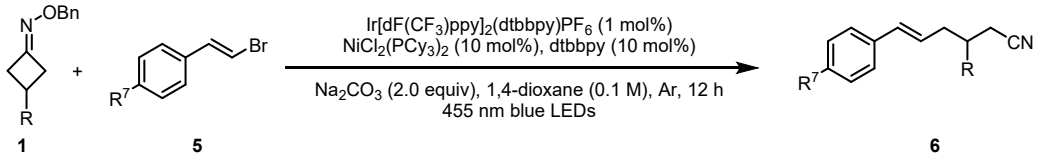
4. Representative Procedure for Schemes 2, 3 and 4



To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **2** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (0.002 mmol, 1 mol%), $NiBr_2$·glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%) and Na_2HPO_4 (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of **1** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **3** or **4**.

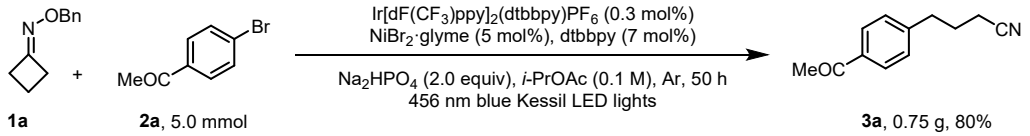


To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **2'** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (0.002 mmol, 1 mol%), $NiBr_2$·glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%) and Na_2HPO_4 (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **3**.



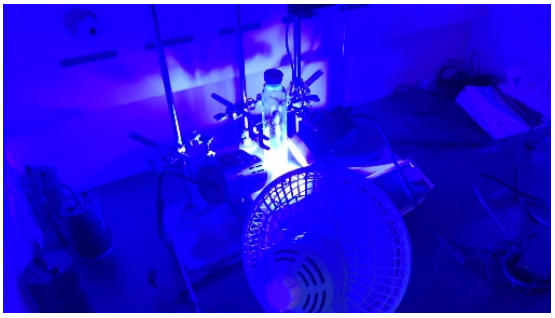
To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **5** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (0.002 mmol, 1 mol%), $NiCl_2(PCy_3)_2$ (0.02 mmol, 10 mol%), dtbbpy (0.02 mmol, 10 mol%) and Na_2CO_3 (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of **1** (0.5 mmol, 2.5 equiv) in 1,4-dioxane (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **6**.

5. Scale-up Synthesis

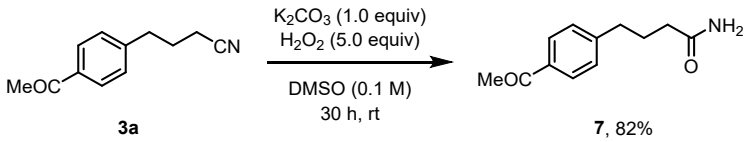


1a **2a**, 5.0 mmol **3a**, 0.75 g, 80%

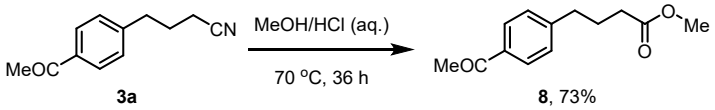
A flame-dried 100 mL quartz column reaction tube was placed with a magnetic stirrer was added **2a** (5.0 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (0.015 mmol, 0.3 mol%), $NiBr_2$·glyme (0.25 mmol, 5 mol%), dtbbpy (0.35 mmol, 7 mol%) and Na_2HPO_4 (10 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (10 mmol, 2.0 equiv) in *i*-PrOAc (50 mL) was added by syringe under nitrogen atmosphere. The reaction tube is placed in the centre of the stirrer and the two kessil lamps are placed perpendicular to the side wall of the reaction tube and then illuminated by a 40 W blue kessil lamp (approx 5 cm away from the light source). The fan is always on to evacuate the heat generated by the kessil lamps and to stabilise the reaction temperature (room temperature) to obtain reproducible results. After stirring for 50 hours at room temperature, concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target products **3a** (0.75 g, 80%).



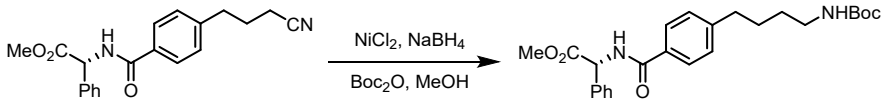
6. Procedures for Diverse Derivatizations of 3a and 3ag[4]



A 25 mL oven-dried reaction tube equipped with a magnetic stirrer was added a solution of **3a** (0.2 mmol, 1.0 equiv), K_2CO_3 (0.2 mmol, 1.0 equiv) in DMSO (2.0 mL). Then, H_2O_2 (1 mmol, 5.0 equiv) was added slowly at 25 ℃ for 12 hours. The reaction mixture was stirred until **3a** completely converted. The reaction mixture was diluted with EtOAc (5.0 mL) and H_2O (5.0 mL). The organic layer was separated and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was washed with saturated brine, dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the target product **7** in 82% yield.



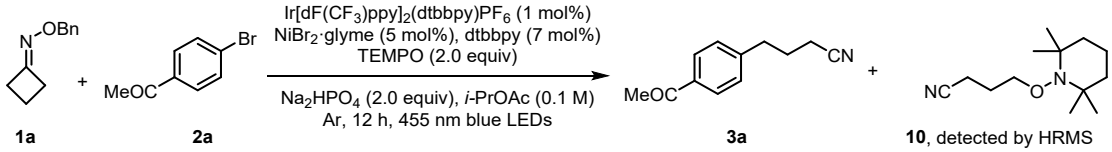
A 25 mL oven-dried reaction tube equipped with a magnetic stirrer was added a solution of **3a** (0.2 mmol, 1.0 equiv) in MeOH/HCl (aq.) (1:1, 2.0 mL). The reaction mixture was stirred at 70 ℃ for 36 hours until **3a** completely converted. After that, 30 mL saturated aqueous $NaHCO_3$ solution was added and the water layer was extracted with EtOAc (3 × 5 mL). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel to give the target product **8** in 73% yield.



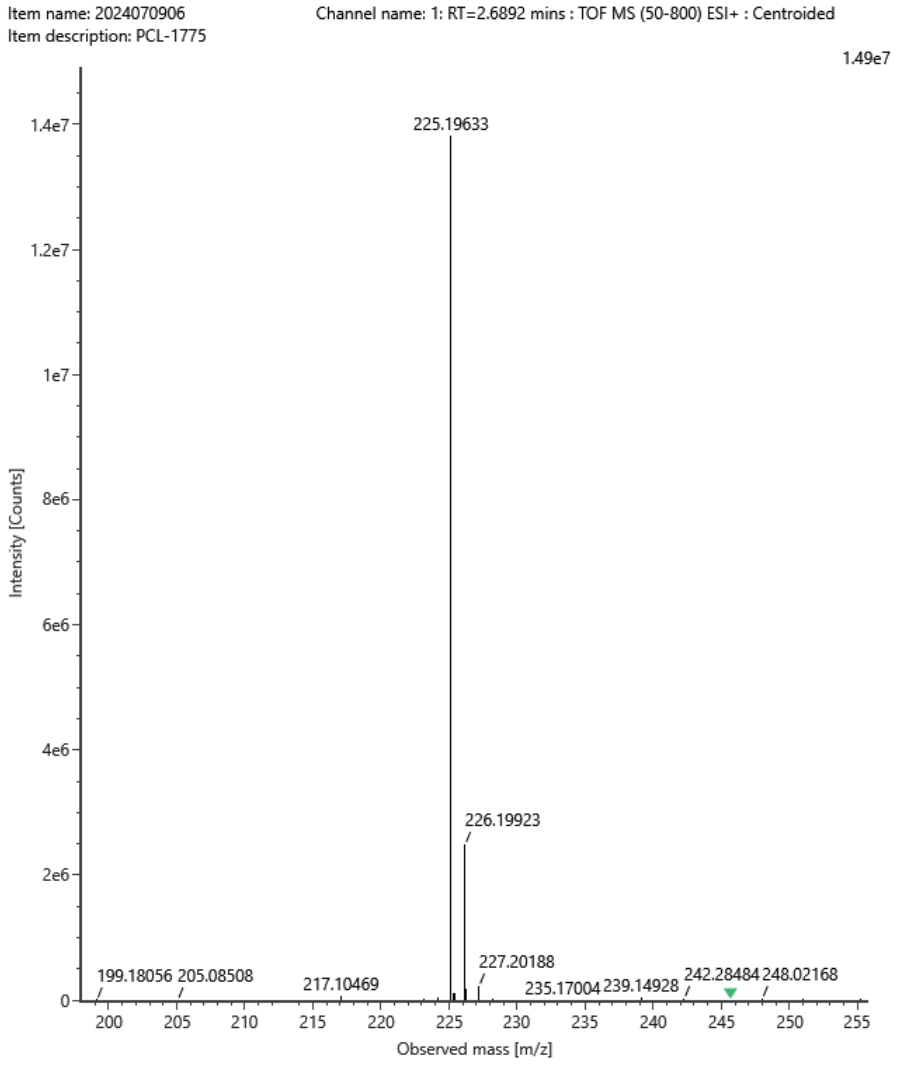
To a solution of compound **3ag** (50.4 mg, 0.15 mmol), $NiCl_2$ (29.2 mg, 1.5 equiv) and Boc_2O (98.2 mg, 3.0 equiv) in MeOH at 0 ℃ was added $NaBH_4$ (56.7 mg, 10 equiv). The mixture was allowed to stir for 4 hours at 50 ℃. The reaction mixture was quenched with a saturated aqueous solution of NH_4Cl and diluted with Et_2O. The layers were separated and the aqueous layer was washed with Et_2O. The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo, which was purified by column chromatography on silica gel to give the target product **9** in 63% yield.

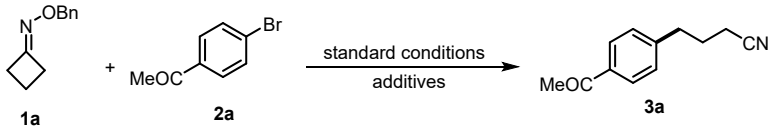
7. Investigation of the Reaction Mechanism

7.1 Radical Trapping and Inhibition Experiments



To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added 1-(4-bromophenyl)ethan-1-one **2a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (0.002 mmol, 1 mol%), $NiBr_2$·glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%), Na_2HPO_4 (0.4 mmol, 2.0 equiv) and TEMPO (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. At the end of the reaction, the reaction was found to be completely inhibited and the TEMPO adducts **10** were captured by UPLC-TOF-MS. These results indicate that a radical intermediate might be involved in this transformation.



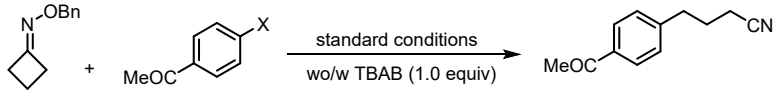


1a **2a** **3a**

Additives	Yield of **3a**
BHT (1.0 equiv)	55%
BHT (2.0 equiv)	35%

To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added 1-(4-bromophenyl)ethan-1-one **2a** (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (0.002 mmol, 1 mol%), $NiBr_2 \cdot$glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%), Na_2HPO_4 (0.4 mmol, 2.0 equiv) and BHT (X equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. The yield of **3a** was reduced to 55% yield when 1.0 equiv of BHT was added. When 2.0 equiv of BHT was added, the yield of **3a** was reduced to 35%. This result indicates that the reaction might proceed via a radical pathway.

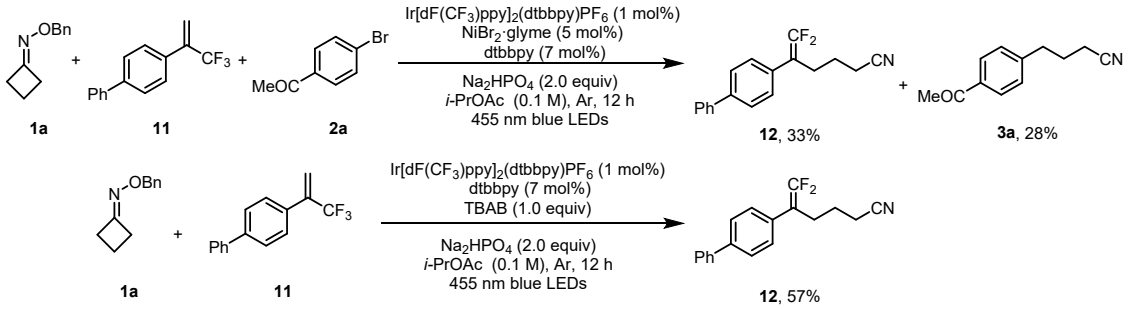
7.2 Reaction Mechanism Experiments



1a

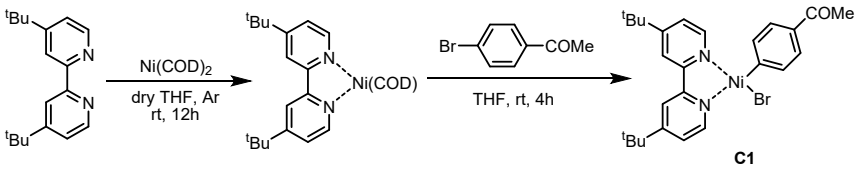
3a X = Cl, 57% / 55%
X = I, NR / 45%
X = OTf, N.R.
X = OTs, N.R.

To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added aryl halides (0.2 mmol, 1.0 equiv), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (0.002 mmol, 1 mol%), $NiBr_2 \cdot$glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%), Na_2HPO_4 (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **3a**. When 4-chloroacetophenone was used as an electrophilic reagent, the reaction yielded the target product **3a** in 57% yield. However, using 4-iodoacetophenone as an electrophilic reagent did not produce the target compound. Under the same conditions, when we added 1.0 equiv of TBAB, the yield of 4-chloroacetophenone as an electrophilic reagent did not change significantly. However, when 4-iodoacetophenone was used as an electrophilic reagent, the reaction was able to obtain the target compound **3a** in 45% yield. These experiments demonstrate that the iodine anion in the reaction did not promote the reaction process, while the bromine anion significantly enhanced the reaction. This result suggests that the reaction may proceed through the oxidation of bromine anions to bromine radical pathway. When using aryl-OTF or aryl-OT as electrophilic reagents, the reaction cannot occur.

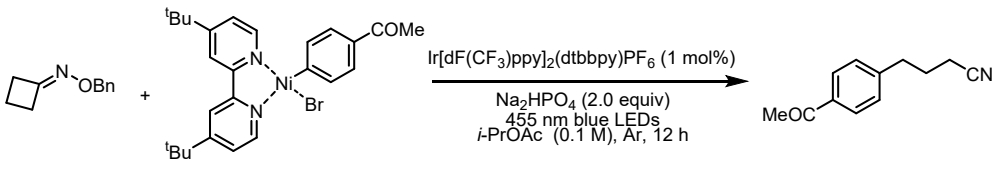


To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **11** (0.2 mmol, 1.0 equiv), **2a** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (0.002 mmol, 1 mol%), $NiBr_2$·glyme (0.01 mmol, 5 mol%), dtbbpy (0.014 mmol, 7 mol%), Na_2HPO_4 (0.4 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **12** (33%) and **3a** (28%). Furthermore, to a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added **11** (0.2 mmol, 1.0 equiv), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (0.002 mmol, 1 mol%), dtbbpy (0.014 mmol, 7 mol%), Na_2HPO_4 (0.4 mmol, 2.0 equiv) and TBAB (1.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.4 mmol, 2.0 equiv) in *i*-PrOAc (2.0 mL) was added by syringe under nitrogen atmosphere. Then the reaction mixture was irradiated with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. Then concentrated under vacuum to afford the crude product, which was purified by column chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the target product **12** (57%). This result suggests that the reaction may take place via the oxidation of bromine anions to bromine radical pathway.

7.3 Active Ni(II)-Complex Species Experiments

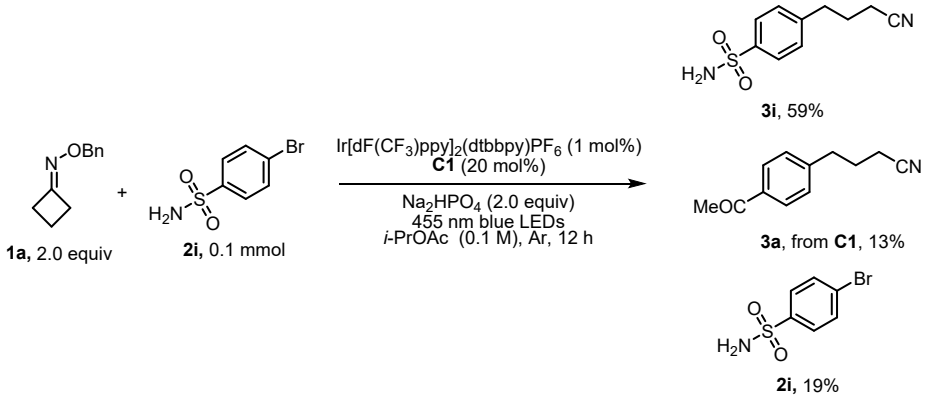


To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added Ni(COD)$_2$ (138 mg, 0.5 mmol, 1.0 equiv) and 4,4'-di-tert-butyl-2,2'-bipyridine (134 mg, 0.5 mmol, 1.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, the dry THF (5 mL) added by syringe under nitrogen atmosphere the mixture was stirred at room temperature for 12 hours. 1-(4-bromophenyl)ethan-1-one (10 mmol, 10.0 equiv) was added and stirred for additional 4 h. Dry pentane (30 mL) was added to the deep red colored mixture and filtered. The resulting precipitate was washed with pentane (3 x 10 mL) and dried under vacum to afford Ni(II) complex **C1** as a brown solid. The product was used without further purification.



1a, 2.0 equiv **C1**, 0.1 mmol **3a**, 49%

To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added Ni(II) complex **C1** (0.1 mmol), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (1 mol%) and Na_2HPO_4 (0.2 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.2 mmol, 2.0 equiv) in *i*-PrOAc (0.1 M) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. The crude product was purified by flash column chromatography on silica gel to afford compound **3a** as a colorless oil (9.2 mg, 49% yield).

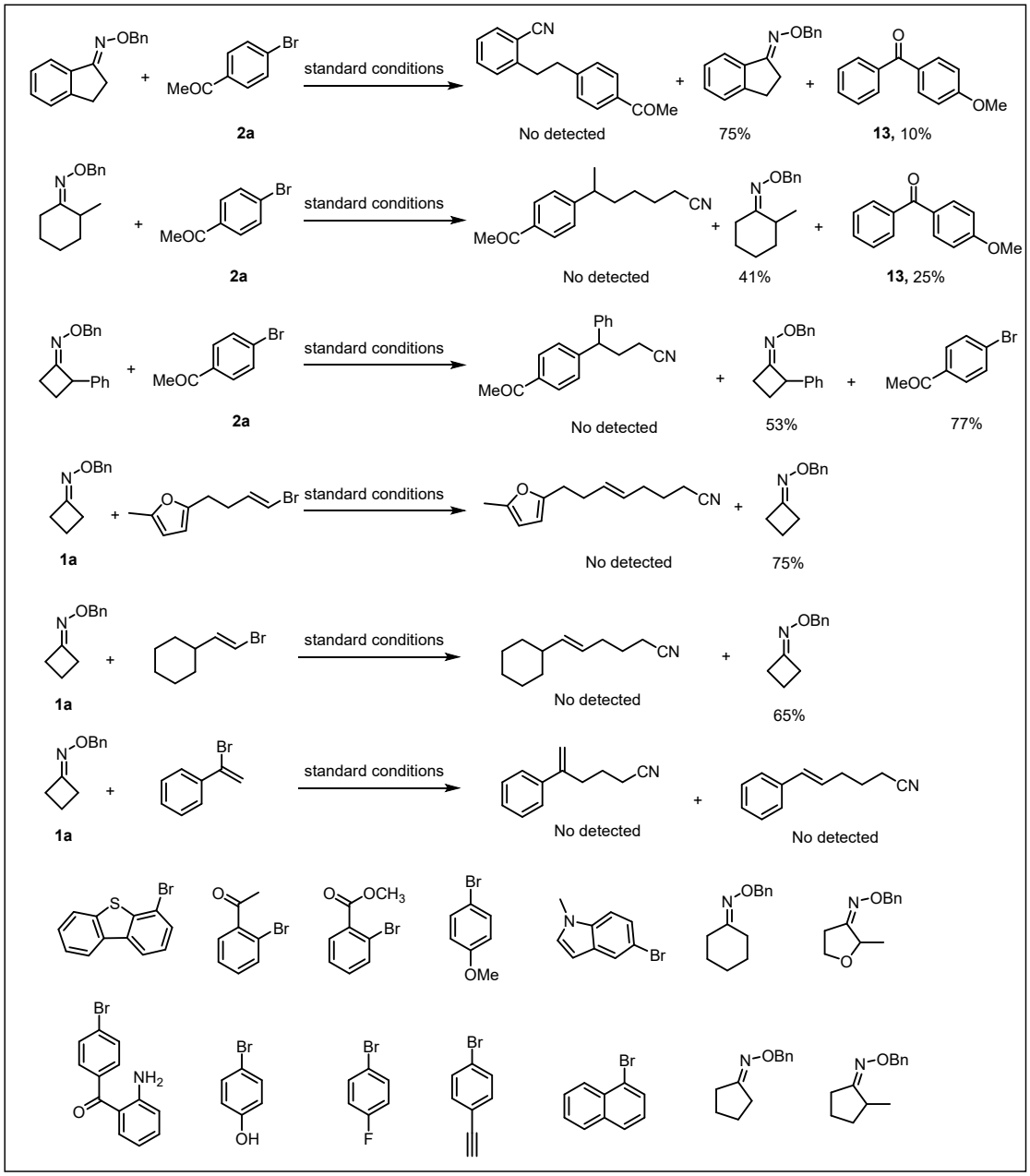


1a, 2.0 equiv **2i,** 0.1 mmol

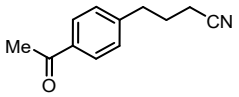
To a 25 mL oven-dried Schlenk-tube equipped with a magnetic stirrer was added Ni(II) complex **C1** (0.02 mmol, 20 mol%), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (1 mol%), **2i** (0.1 mmol, 1.0 equiv) and Na_2HPO_4 (0.2 mmol, 2.0 equiv). Then, the tube was evacuated and backfilled with nitrogen for three times. Subsequently, a solution of cyclobutanone *O*-benzyl oxime **1a** (0.2 mmol, 2.0 equiv) in *i*-PrOAc (0.1 M) was added by syringe under nitrogen atmosphere. The tube was sealed and was placed on a photocatalytic parallel reactor with the 10 W blue LEDs (at approximately 0.3 cm away from the light source) and the mixture was stirred at room temperature for 12 hours. The crude product was purified by flash column chromatography on silica gel to afford compound **3i** (13.2 mg, 59%) and compound **3a** (2.5 mg, 13%).

8. Failed Substrates

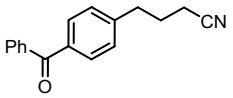
Failed substrates



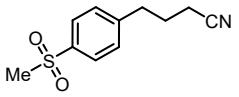
9. Characterizations of Products



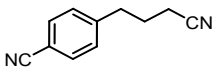
(3a) 4-(4-Acetylphenyl)butanenitrile[5] Colorless oil (32.3 mg, 86% or 21.3 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.4 Hz, 2H), 2.85 (t, J = 8.0 Hz, 2H), 2.59 (s, 3H), 2.34 (t, J = 6.8 Hz, 2H), 2.05 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 145.4, 135.7, 128.8, 128.7, 119.2, 34.3, 26.6, 26.5, 16.5.



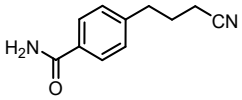
(3b) 4-(4-Benzoylphenyl)butanenitrile[5] Colorless oil (32.9 mg, 66% or 30 mg, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.75 (m, 4H), 7.61 – 7.57 (m, 1H), 7.51 – 7.46 (m, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.87 (t, J = 7.6 Hz, 2H), 2.37 (t, J = 6.8 Hz, 2H), 2.07 – 2.00 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.3, 144.7, 137.7, 136.0, 132.4, 130.6, 130.0, 128.4, 128.3, 119.2, 34.4, 26.6, 16.5.



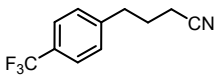
(3c) 4-(4-(Methylsulfonyl)phenyl)butanenitrile[5] Colorless oil (28.1 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.87 (m, 2H), 7.40 (d, J = 8.4 Hz, 2H), 3.04 (s, 3H), 2.88 (t, J = 7.6 Hz, 2H), 2.36 (t, J = 6.8 Hz, 2H), 2.05 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.3, 138.9, 129.4, 127.8, 119.0, 44.5, 34.2, 26.4, 16.5.



(3d) 4-(3-Cyanopropyl)benzonitrile[6] Colorless oil (20.1 mg, 59%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.59 (m, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.85 (t, J = 7.6 Hz, 2H), 2.36 (t, J = 6.8 Hz, 2H), 2.03 – 1.96 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.3, 132.5, 129.3, 119.0, 118.8, 110.7, 34.5, 26.4, 16.5.

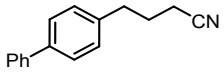


(3e) 4-(3-Cyanopropyl)benzamide Solid (21.8 mg, 58%). ^{1}H NMR (400 MHz, DMSO) δ 7.91 (br, 1H), 7.81 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 2.71 (t, J = 8.0 Hz, 2H), 2.51 – 2.47 (m, 2H), 1.91 – 1.84 (m, 2H); ^{13}C NMR (101 MHz, DMSO) δ 168.2, 144.3, 132.7, 128.6, 128.2, 120.9, 34.2, 26.6, 16.3. Melting point (℃): 132.9 – 134.4 ℃. HRMS (APCI) m/z calcd for $C_{11}H_{13}ON_2$ $[M+H]^+$ 189.1028, found 189.1019. IR (cm^{-1}): 3356, 3182, 2933, 2850, 2756, 2553, 2245, 2168, 2042, 1674, 1614, 1568, 1377, 1190, 1045, 864, 773, 628.

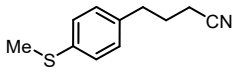


(3f) 4-(4-(Trifluoromethyl)phenyl)butanenitrile[6] Colorless oil (22.0 mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.85 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 7.2 Hz, 2H), 2.04 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.8, 129.0 (q, J = 32.5 Hz), 128.8, 125.6 (q,

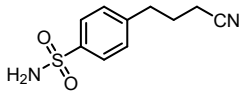
J = 3.7 Hz) 124.1 (q, J = 273.0 Hz), 119.1, 34.1, 26.6, 16.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.32.



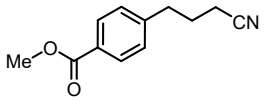
(3g) 4-([1,1'-Biphenyl]-4-yl)butanenitrile[7] Solid (20.3 mg, 46%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.53 (m, 4H), 7.44 (t, J = 7.6 Hz, 2H), 7.34 (t, J = 7.6 Hz, 1H), 7.26 (d, J = 7.6 Hz, 2H), 2.82 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 6.8 Hz, 2H), 2.06 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.8, 139.6, 138.8, 128.9, 128.8, 127.4, 127.3, 127.0, 119.5, 34.0, 26.9, 16.5.



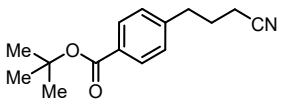
(3h) 4-(4-(Methylthio)phenyl)butanenitrile[8] Colorless oil (12.1 mg, 32%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.20 (m, 2H), 7.11 (d, J = 8.4 Hz, 2H), 2.74 (d, J = 7.2 Hz, 2H), 2.48 (s, 3H), 2.31 (t, J = 7.2 Hz, 2H), 2.00 – 1.92 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.6, 136.4, 129.0, 127.2, 119.4, 33.8, 26.9, 16.4, 16.1.



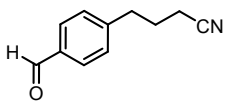
(3i) 4-(3-Cyanopropyl)benzenesulfonamide Colorless oil (35.9 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.86 (m, 2H), 7.34 (d, J = 8.4 Hz, 2H), 5.04 (s, 2H), 2.86 (t, J = 8.0 Hz, 2H), 2.36 (t, J = 7.2 Hz, 2H), 2.04 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.2, 140.3, 129.2, 126.9, 119.1, 34.2, 26.5, 16.5. HRMS (APCI) m/z calcd for $C_{10}H_{13}O_2N_2S$ $[M+H]^+$ 225.0698, found 225.0689. IR (cm^{-1}): 3182, 3047, 2935, 2868, 2690, 2245, 1599, 1495, 1435, 1408, 1335, 1161, 1028, 955, 837, 806, 679, 582, 553.



(3j) Methyl 4-(3-cyanopropyl)benzoate[9] Colorless oil (32.5 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.97 (m, 2H), 7.28 – 7.26 (m, 2H), 3.91 (s, 3H), 2.84 (t, J = 7.6 Hz, 2H), 2.34 (t, J = 7.2 Hz, 2H), 2.04 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.8, 145.0, 130.0, 128.5, 128.4, 119.2, 52.0, 34.3, 26.5, 16.4.

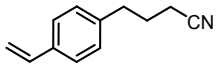


(3k) Tert-butyl 4-(3-cyanopropyl)benzoate Colorless oil (37.3 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 – 7.92 (m, 2H), 7.23 (d, J = 8.4 Hz, 2H), 2.83 (t, J = 7.6 Hz, 2H), 2.32 (t, J = 7.2 Hz, 2H), 2.03 – 1.96 (m, 2H), 1.59 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.5, 143.5, 129.5, 128.8, 127.3, 118.2, 80.0, 33.3, 27.2, 25.6, 15.4. HRMS (APCI) m/z calcd for $C_{15}H_{20}O_2N$ $[M+H]^+$ 246.1494, found 246.1485. IR (cm^{-1}): 2933, 2247, 1711, 1610, 1479, 1458, 1369, 1294, 1256, 1167, 1112, 1018, 982, 851, 768.

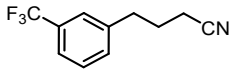


(3l) 4-(4-Formylphenyl)butanenitrile Colorless oil (24.2 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 7.85 – 7.82 (m, 2H), 7.37 (d, J = 8.0 Hz, 2H), 2.88 (t, J = 7.6 Hz, 2H), 2.36 (t, J = 6.8 Hz, 2H),

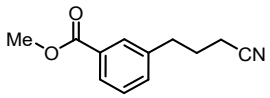
2.06 – 1.99 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.8, 147.0, 135.1, 130.2, 129.2, 119.1, 34.6, 26.5, 16.5. HRMS (APCI) m/z calcd for $C_{11}H_{12}ON$ $[M+H]^+$ 174.0919, found 174.0911. IR (cm^{-1}): 2935, 2854, 2739, 2245, 1699, 1607, 1576, 1306, 1213, 1171, 1057, 1016, 849, 825, 779, 486, 434.



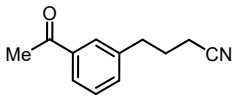
(3m) 4-(4-Vinylphenyl)butanenitrile Colorless oil (12.2 mg, 36%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 6.73 – 6.66 (m, 1H), 5.75 – 5.70 (m, 1H), 5.24 – 5.21 (m, 1H), 2.77 (t, J = 7.2 Hz, 2H), 2.32 (t, J = 6.8 Hz, 2H), 2.02 – 1.94 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 136.4, 136.0, 128.7, 126.5, 119.5, 113.6, 34.1, 26.9, 16.4. HRMS (APCI) m/z calcd for $C_{12}H_{14}N$ $[M+H]^+$ 172.1126, found 172.1118. IR (cm^{-1}): 3433, 2997, 2914, 2243, 1437, 1406, 1057, 955.



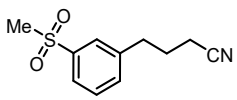
(3n) 4-(3-(Trifluoromethyl)phenyl)butanenitrile Colorless oil (23.8 mg, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.38 (m, 4H), 2.85 (t, J = 8.0 Hz, 2H), 2.36 (t, J = 7.2 Hz, 2H), 2.05 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.7, 131.9, 131.0 (q, J = 32.2 Hz), 129.2, 125.1 (q, J = 3.8 Hz), 124.1 (q, J = 273.4 Hz), 123.5 (q, J = 3.7 Hz), 119.1, 34.2, 26.7, 16.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.64. HRMS (APCI) m/z calcd for $C_{11}H_{11}NF_3$ $[M+H]^+$ 214.0844, found 214.0834. IR (cm^{-1}): 2937, 2862, 2768, 2247, 2154, 2041, 1595, 1493, 1454, 1333, 1201, 1163, 1124, 984, 800.



(3o) Methyl 3-(3-cyanopropyl)benzoate Colorless oil (20.5 mg, 50% or 19.5 mg, 48%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.89 (m, 1H), 7.87 (s, 1H), 7.39 – 7.35 (m, 2H), 3.91 (s, 3H), 2.83 (t, J = 7.6 Hz, 2H), 2.33 (t, J = 6.8 Hz, 2H), 2.04 – 1.97 (m, 2 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.0, 140.1, 133.1, 130.6, 129.4, 128.8, 127.9, 119.3, 52.2, 34.2, 26.8, 16.5. HRMS (ESI) m/z calcd for $C_{12}H_{14}NO_2$ $[M+H]^+$ 204.1025, found 204.1021. IR (cm^{-1}): 3431, 3034, 2949, 2866, 2245, 1720, 1589, 1435, 1286, 1203, 1109, 1059, 951, 752, 696.

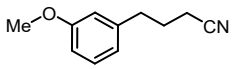


(3p) 4-(3-Acetylphenyl)butanenitrile Colorless oil (17.5 mg, 47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.79 (m, 2H), 7.44 – 7.39 (m, 2H), 2.85 (t, J = 8.0 Hz, 2H), 2.60 (s, 3H), 2.34 (t, J = 7.2 Hz, 2H), 2.05 – 1.98 (m, 2 H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 140.4, 137.6, 133.3, 129.0, 128.0, 126.8, 119.3, 34.3, 26.8, 26.7, 16.5. HRMS (APCI) m/z calcd for $C_{12}H_{14}ON$ $[M+H]^+$ 188.1075, found 188.1068. IR (cm^{-1}): 3348, 3049, 2937, 2868, 2243, 1684, 1603, 1585, 1485, 1435, 1360, 1271, 1190, 1059, 912, 802, 694, 590.

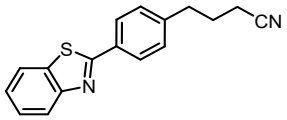


(3q) 4-(3-(Methylsulfonyl)phenyl)butanenitrile Colorless oil (20.8 mg, 47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.77 (m, 2H), 7.55 – 7.48 (m, 2H), 3.05 (s, 3H), 2.88 (t, J = 8.0 Hz, 2H), 2.37 (t, J = 6.8 Hz, 2H), 2.06 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.7, 141.1, 133.9, 129.8, 127.0, 125.6,

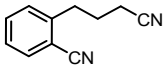
119.0, 44.5, 34.2, 26.6, 16.6. HRMS (APCI) m/z calcd for $C_{11}H_{14}O_2NS$ $[M+H]^+$ 224.0745, found 224.0736. IR (cm^{-1}): 3447, 3005, 2920, 2866, 2245, 1713, 1659, 1601, 1479, 1433, 1298, 1215, 1146, 1057, 955, 930, 802, 760, 698, 536, 484.



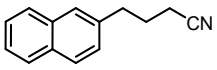
(3r) 4-(3-Methoxyphenyl)butanenitrile[10] Colorless oil (10.2 mg, 30%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.23 (t, J = 8.0 Hz, 1H), 6.79 – 6.76 (m, 2H), 6.73 – 6.72 (m, 1H), 3.81 (s, 3H), 2.76 (t, J = 7.6 Hz, 2H), 2.32 (t, J = 7.2 Hz, 2H), 2.02 – 1.95 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.9, 141.3, 129.7, 120.8, 119.5, 114.3, 111.7, 55.2, 34.4, 26.8, 16.4.



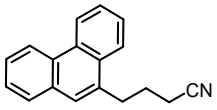
(3s) 4-(4-(Benzo[d]thiazol-2-yl)phenyl)butanenitrile Solid (38.8 mg, 70%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, J = 8.0 Hz, 1H), 8.03 (d, J = 8.4 Hz, 2H), 7.90 (d, J = 7.6 Hz, 1H), 7.51 – 7.47 (m, 1H), 7.40 – 7.36 (m, 1H), 7.31 (d, J = 8.4 Hz, 2H), 2.84 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 6.8 Hz, 2H), 2.05 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.7, 154.2, 143.0, 135.0, 132.1, 129.2, 127.9, 126.4, 125.2, 123.2, 121.7, 119.3, 34.3, 26.7, 16.5. Melting point (°C): 107.5 – 112.0 °C. HRMS (APCI) m/z calcd for $C_{17}H_{15}N_2S$ $[M+H]^+$ 279.0956, found 279.0947. IR (cm^{-1}): 3446, 2999, 2914, 2243, 1680, 1485, 1437, 1414, 1312, 1252, 1045, 953, 818, 760.



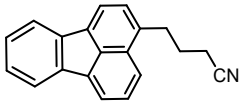
(3t) 2-(3-Cyanopropyl)benzonitrile Colorless oil (21.5 mg, 63%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.63 (m, 1H), 7.58 – 7.54 (m, 1H), 7.37 – 7.33 (m, 2H), 3.00 (t, J = 7.6 Hz, 2H), 2.40 (t, J = 7.2 Hz, 2H), 2.10 – 2.03 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.7, 133.2, 133.2, 129.8, 127.4, 119.0, 117.7, 112.4, 33.3, 26.3, 16.7. HRMS (APCI) m/z calcd for $C_{11}H_{11}N_2$ $[M+H]^+$ 171.0922, found 171.0914. IR (cm^{-1}): 3429, 3062, 2933, 2866, 2761, 2229, 1668, 1597, 1487, 1454, 1365, 1049, 767.



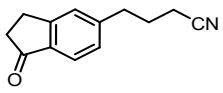
(3u) 4-(Naphthalen-2-yl)butanenitrile[6] Colorless oil (19.6 mg, 50%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.79 (m, 3H), 7.64 (s, 1H), 7.51 – 7.43 (m, 2H), 7.33 – 7.31 (m, 1H), 2.96 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 7.2 Hz, 2H), 2.12 – 2.05 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.1, 133.6, 132.3, 128.4, 127.7, 127.5, 126.9, 126.8, 126.2, 125.6, 119.5, 34.5, 26.8, 16.4.



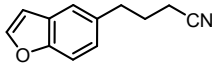
(3v) 4-(Phenanthren-9-yl)butanenitrile Solid (19.8 mg, 40%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.77 – 8.75 (m, 1H), 8.67 (d, J = 8.0 Hz, 1H), 8.07 – 8.04 (m, 1H), 7.86 – 7.84 (m, 1H), 7.71 – 7.59 (m, 5H), 3.29 (t, J = 7.6 Hz, 2H), 2.41 (t, J = 7.2 Hz, 2H), 2.22 – 2.14 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.8, 131.6, 130.9, 130.7, 129.9, 128.2, 127.0, 126.9, 126.9, 126.5, 126.5, 124.0, 123.5, 122.5, 119.6, 32.0, 25.6, 16.8. Melting point (°C): 85.1 – 88.1 °C. HRMS (APCI) m/z calcd for $C_{18}H_{16}N$ $[M+H]^+$ 246.1283, found 246.1274. IR (cm^{-1}): 3074, 2937, 2868, 2750, 2245, 2164, 2039, 1682, 1607, 1497, 1427, 1362, 1250, 1211, 1057, 980, 750, 617.



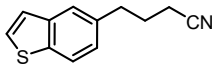
(3w) 4-(Fluoranthen-3-yl)butanenitrile Colorless oil (22.8 mg, 42%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.94 (m, 2H), 7.91 – 7.84 (m, 3H), 7.68 – 7.65 (m, 1H), 7.44 (d, J = 7.2 Hz, 1H), 7.40 – 7.35 (m, 2H), 3.32 (t, J = 7.6 Hz, 2H), 2.39 (t, J = 7.2 Hz, 2H), 2.20 – 2.13 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.4, 139.2, 137.6, 136.9, 136.1, 132.9, 129.0, 128.1, 128.1, 127.7, 127.5, 123.1, 121.5, 121.4, 120.1, 120.1, 119.5, 30.8, 27.3, 16.7. HRMS (APCI) m/z calcd for $C_{20}H_{16}N$ $[M+H]^+$ 270.1283, found 270.1273. IR (cm^{-1}): 3651, 3142, 3059, 2932, 2864, 2770, 2241, 2168, 2035, 1736, 1495, 1452, 1371, 1236, 1153, 1057, 991, 831, 781, 758, 685, 584, 521, 457, 428.



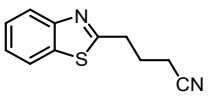
(3x) 4-(1-Oxo-2,3-dihydro-1*H*-inden-5-yl)butanenitrile[11] Colorless oil (21.8 mg, 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.0 Hz, 1H), 7.30 (s, 1H), 7.19 (d, J = 8.0 Hz, 1H), 3.11 (t, J = 6.0 Hz, 2H), 2.86 (t, J = 7.6 Hz, 2H), 2.70 – 2.67 (m, 2H), 2.37 – 2.33 (m, 2H), 2.05 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.5, 156.0, 147.3, 135.8, 127.9, 126.6, 124.1, 119.2, 36.4, 34.7, 26.7, 25.7, 16.5.



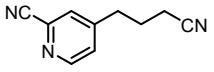
(3y) 4-(Benzofuran-5-yl)butanenitrile Colorless oil (12.2 mg, 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 2.0 Hz, 1H), 7.45 – 7.41 (m, 2H), 7.12 – 7.10 (m, 1H), 6.73 – 6.72 (m, 1H), 2.88 (t, J = 7.6 Hz, 2H), 2.32 (t, J = 6.8 Hz, 2H), 2.06 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.9, 145.4, 134.1, 127.8, 124.8, 120.8, 119.6, 111.5, 106.4, 34.3, 27.4, 16.3. HRMS (ESI) m/z calcd for $C_{12}H_{11}NONa$ $[M+Na]^+$ 208.0738, found 208.0734. IR (cm^{-1}): 3113, 2932, 2864, 2245, 1537, 1468, 1445, 1329, 1263, 1200, 1126, 1030, 947, 881, 769, 737, 424.



(3z) 4-(Benzo[b]thiophen-5-yl)butanenitrile[12] Colorless oil (17.2 mg, 43%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.4 Hz, 1H), 7.56 (d, J = 0.8 Hz, 1H), 7.45 (d, J = 5.6 Hz, 1H), 7.30 (d, J = 5.6 Hz, 1H), 7.19 – 7.17 (m, 1H), 2.91 (t, J = 7.6 Hz, 2H), 2.33 (t, J = 6.8 Hz, 2H), 2.08 – 2.01 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.1, 138.0, 135.8, 127.0, 125.0, 123.6, 123.3, 122.7, 119.5, 34.3, 27.2, 16.4.

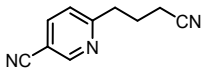


(3aa) 4-(Benzo[d]thiazol-2-yl)butanenitrile[13] Colorless oil (30.3 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 8.0 Hz, 1H), 7.87 – 7.85 (m, 1H), 7.50 – 7.45 (m, 1H), 7.40 – 7.36 (m, 1H), 3.28 (t, J = 7.2 Hz, 2H), 2.56 (t, J = 7.2 Hz, 2H), 2.33 – 2.26 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 153.2, 135.1, 126.2, 125.1, 122.7, 121.6, 119.0, 32.5, 24.8, 16.6.

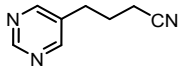


(3ab) 4-(3-Cyanopropyl)picolinonitrile Colorless oil (27.5 mg, 80% or 12 mg, 35%). ^{1}H NMR (400

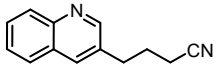
MHz, $CDCl_3$) δ 8.62 (d, J = 5.2 Hz, 1H), 7.55 (s, 1H), 7.38 – 7.37 (m, 1H), 2.86 (t, J = 8.0 Hz, 2H), 2.41 (t, J = 6.8 Hz, 2H), 2.06 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.3, 150.6, 134.3, 128.5, 127.1, 118.6, 117.2, 33.4, 25.5, 16.7. HRMS (APCI) m/z calcd for $C_{10}H_{10}N_3$ $[M+H]^+$ 172.0875, found 172.0866. IR (cm^{-1}): 3433, 3051, 2925, 2872, 2239, 1659, 1600, 1554, 1461, 1434, 1558, 1462, 1406, 1053, 951, 870, 835, 739, 480.



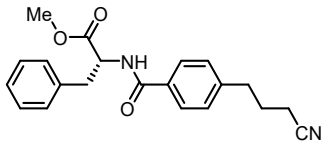
(3ac) 6-(3-Cyanopropyl)nicotinonitrile Colorless oil (23.9 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.31 (d, J = 8.0 Hz, 1H), 3.02 (t, J = 7.2 Hz, 2H), 2.43 (t, J = 6.8 Hz, 2H), 2.18 – 2.11 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.1, 152.2, 139.7, 123.3, 119.1, 116.7, 107.9, 36.5, 24.2, 16.7. HRMS (APCI) m/z calcd for $C_{10}H_{10}N_3$ $[M+H]^+$ 172.0875, found 172.0866. IR (cm^{-1}): 3425, 3140, 2933, 2847, 2766, 2542, 2233, 2154, 2040, 1595, 1485, 1371, 1203, 1028, 982, 916, 756, 646.



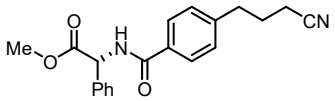
(3ad) 4-(Pyrimidin-5-yl)butanenitrile Colorless oil (13.9 mg, 47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (s, 1H), 8.62 (s, 2H), 2.81 (t, J = 8.0 Hz, 2H), 2.42 (t, J = 6.8 Hz, 2H), 2.05 – 1.98 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.4, 156.7, 133.0, 118.6, 29.1, 26.3, 16.7. HRMS (ESI) m/z calcd for $C_8H_{10}N_3$ $[M+H]^+$ 148.0875, found 148.0870. IR (cm^{-1}): 3045, 2935, 2858, 2771, 2245, 2156, 2035, 1682, 1562, 1462, 1412, 1358, 1171, 980, 729, 633.



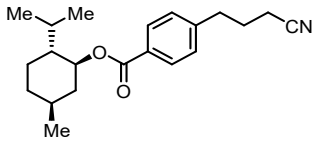
(3ae) 4-(Quinolin-3-yl)butanenitrile Solid (16.5 mg, 42% or 10.6 mg, 27%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.79 (d, J = 2.0 Hz, 1H), 8.10 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 1.2 Hz, 1H), 7.79 (d, J = 8.0 Hz, 1H), 7.72 – 7.68 (m, 1H), 7.57 – 7.54 (m, 1H), 2.99 (t, J = 7.6 Hz, 2H), 2.40 (t, J = 7.2 Hz, 2H), 2.13 – 2.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.3, 147.1, 134.8, 132.4, 129.2, 129.2, 128.0, 127.4, 127.0, 119.1, 31.8, 26.6, 16.6. Melting point (°C): 46.6 – 52.0 °C. HRMS (APCI) m/z calcd for $C_{13}H_{13}N_2$ $[M+H]^+$ 197.1079, found 197.1071. IR (cm^{-1}): 3437, 3267, 3063, 2935, 2864, 2766, 2243, 1607, 1570, 1495, 1126, 1057, 789, 756, 685, 611, 480.



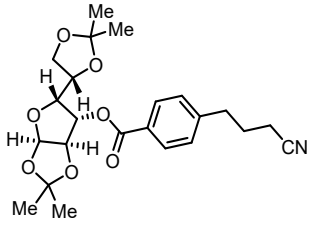
(3af) Methyl (4-(3-cyanopropyl)benzoyl)phenylalaninate Colorless oil (47.6 mg, 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 8.0 Hz, 2H), 7.31 – 7.23 (m, 5H), 7.13 (d, J = 7.2 Hz, 2H), 6.59 (d, J = 7.2 Hz, 1H), 5.10 – 5.06 (m, 1H), 3.76 (s, 3H), 3.31 – 3.19 (m, 2H), 2.82 (t, J = 7.6 Hz, 2H), 2.32 (t, J = 6.8 Hz, 2H), 2.02 – 1.95 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 166.5, 143.9, 135.9, 132.3, 129.3, 128.7, 128.7, 127.5, 127.2, 119.3, 53.5, 52.5, 37.9, 34.2, 26.6, 16.4. HRMS (ESI) m/z calcd for $C_{21}H_{22}N_2O_3Na$ $[M+Na]^+$ 373.1528, found 373.1523. IR (cm^{-1}): 3254, 3061, 3030, 2947, 2862, 2766, 2249, 1743, 1647, 1537, 1500, 1454, 1273, 1219, 1099, 1030, 982, 862, 760, 702.



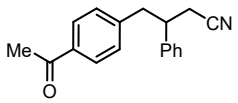
(3ag) Methyl 2-(4-(3-cyanopropyl)benzamido)-2-phenylacetate Colorless oil (48.3 mg, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 8.4 Hz, 2H), 7.45 – 7.43 (m, 2H), 7.40 – 7.32 (m, 3H), 7.27 – 7.25 (m, 2H), 7.14 (d, J = 6.4 Hz, 1H), 5.77 (d, J = 6.8 Hz, 1H), 3.77 (s, 3H), 2.83 (t, J = 7.2 Hz, 2H), 2.32 (t, J = 6.8 Hz, 2H), 2.04 – 1.95 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.5, 166.2, 144.0, 136.6, 132.0, 129.1, 128.7, 128.6, 127.6, 127.4, 119.2, 56.8, 53.0, 34.2, 26.6, 16.4. HRMS (ESI) m/z calcd for $C_{20}H_{20}N_2O_3Na$ $[M+Na]^+$ 359.1372, found 359.1365. IR (cm^{-1}): 3254, 3063, 3034, 2953, 2858, 2245, 1745, 1647, 1610, 1535, 1500, 1456, 1335, 1267, 1215, 1175, 1057, 982, 951, 860, 760, 698, 600, 544.



(3ah) 2-Isopropyl-5-methylcyclohexyl 4-(3-cyanopropyl)benzoate Colorless oil (49.1 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 4.96 – 4.89 (m, 1H), 2.84 (t, J = 7.6 Hz, 2H), 2.33 (t, J = 6.8 Hz, 2H), 2.12 (d, J = 11.6 Hz, 1H), 2.04 – 1.92 (m, 3H), 1.75 – 1.71 (m, 2H), 1.59 – 1.52 (m, 2H), 1.19 – 1.05 (m, 2H), 0.97 – 0.89 (m, 7H), 0.79 (d, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.9, 144.9, 130.0, 129.3, 128.5, 119.2, 74.8, 47.3, 41.0, 34.3, 34.3, 31.5, 26.6, 26.5, 23.7, 22.1, 20.8, 16.6, 16.4. HRMS (APCI) m/z calcd for $C_{21}H_{30}O_2N$ $[M+H]^+$ 328.2277, found 328.2262. IR (cm^{-1}): 2954, 2870, 2247, 1707, 1612, 1462, 1367, 1277, 1178, 1113, 982, 862, 764.

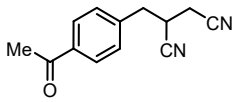


(3ai) 5-(2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-yl 4-(3-cyanopropyl)benzoate Colorless oil (60.5 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.90 (d, J = 4.0 Hz, 1H), 5.09 – 5.06 (m, 1H), 4.97 (t, J = 4.8 Hz, 1H), 4.39 – 4.32 (m, 2H), 4.14 – 4.10 (m, 1H), 4.00 – 3.96 (m, 1H), 2.86 (t, J = 7.6 Hz, 2H), 2.35 (t, J = 6.8 Hz, 2H), 2.05 – 1.97 (m, 2H), 1.55 (s, 3H), 1.41 (s, 3H), 1.33 (d, J = 2.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5, 143.8, 128.4, 126.7, 126.0, 117.3, 111.3, 108.1, 102.4, 76.0, 76.0, 73.3, 71.3, 63.9, 32.5, 24.9, 24.8, 24.7, 24.5, 23.1, 14.6. HRMS (ESI) m/z calcd for $C_{23}H_{29}NO_7Na$ $[M+Na]^+$ 454.1842, found 454.1837. IR (cm^{-1}): 2989, 2937, 2872, 2245, 1726, 1610, 1458, 1375, 1275, 1215, 1105, 1059, 1020, 868, 766, 609, 513.

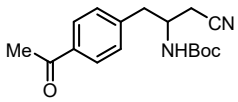


(4b) 4-(4-Acetylphenyl)-3-phenylbutanenitrile Colorless oil (46.4 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.0 Hz, 2H), 7.34 – 7.28 (m, 3H), 7.17 (t, J = 8.0 Hz, 4H), 3.29 – 3.07 (m, 3H), 2.62 – 2.60 (m, 2H), 2.56 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.7, 144.0, 140.6, 135.7, 129.3, 129.0, 128.6, 127.7, 127.2, 118.2, 43.6, 41.1, 26.6, 23.9. HRMS (APCI) m/z calcd for $C_{18}H_{18}ON$ $[M+H]^+$

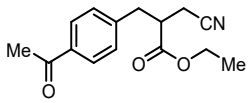
264.1388, found 264.1378. IR (cm^{-1}): 3346, 3031, 2930, 2860, 2245, 1678, 1605, 1572, 1497, 1454, 1416, 1362, 1267, 1182, 1059, 953, 820, 764, 704, 609.



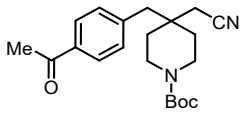
(4c) 2-(4-Acetylbenzyl)succinonitrile Colorless oil (28.0 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 8.0 Hz, 2H), 3.25 – 3.18 (m, 1H), 3.15 – 3.14 (m, 2H), 2.70 (d, J = 6.0 Hz, 2H), 2.60 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 139.7, 136.9, 129.5, 129.2, 118.2, 115.4, 36.9, 29.9, 26.7, 20.3. HRMS (APCI) m/z calcd for $C_{13}H_{13}ON_2$ $[M+H]^+$ 213.1028, found 213.1018. IR (cm^{-1}): 3346, 2932, 2862, 2245, 1682, 1610, 1572, 1416, 1360, 1269, 1186, 1115, 1055, 955, 849, 820, 694, 598, 496.



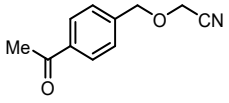
(4d) Tert-butyl (1-(4-acetylphenyl)-3-cyanopropan-2-yl)carbamate Solid (38.5 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 4.84 (d, J = 7.6 Hz, 1H), 4.11 – 4.10 (m, 1H), 3.08 – 2.91 (m, 2H), 2.71 (dd, J = 16.4 Hz, 4.4 Hz, 1H), 2.58 (s, 3 H), 2.43 (dd, J = 16.8 Hz, 4.4 Hz, 1H), 1.40 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 154.8, 141.8, 136.2, 129.4, 129.0, 117.1, 80.5, 48.3, 39.4, 28.3, 26.6, 22.8. Melting point (℃): 123.6 – 126.8 ℃. HRMS (APCI) m/z calcd for $C_{17}H_{23}O_3N_2$ $[M+H]^+$ 303.1709, found 303.1698. IR (cm^{-1}): 3641, 3219, 2932, 2854, 2766, 2247, 2164, 2040, 1684, 1608, 1526, 1363, 1267, 1169, 1045, 980, 818, 679, 600.



(4e) Ethyl 2-(4-acetylbenzyl)-3-cyanopropanoate Colorless oil (30.9 mg, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 4.21 – 4.14 (m, 2H), 3.23 – 3.17 (m, 1H), 3.05 – 2.98 (m, 2H), 2.58 (s, 3H), 2.53 (d, J = 6.0 Hz, 2H), 1.23 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 171.5, 142.4, 136.2, 129.3, 128.9, 117.4, 61.7, 42.9, 36.7, 26.6, 18.7, 14.1. HRMS (APCI) m/z calcd for $C_{15}H_{18}O_3N$ $[M+H]^+$ 260.1287, found 260.1276. IR (cm^{-1}): 3346, 2937, 2870, 2249, 1734, 1682, 1607, 1570, 1448, 1416, 1362, 1269, 1186, 1117, 1039, 957, 854, 600, 580.

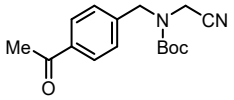


(4f) Tert-butyl 4-(4-acetylbenzyl)-4-(cyanomethyl)piperidine-1-carboxylate[5] Colorless oil (64.8 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.4 Hz, 2H), 3.67 – 3.61 (m, 2H), 3.32 – 3.26 (m, 2H), 2.87 (s, 2H), 2.59 (s, 3H), 2.23 (s, 2H), 1.64 – 1.57 (m, 2H), 1.54 – 1.49 (m, 2H), 1.45 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 154.7, 141.5, 136.0, 130.6, 128.5, 117.6, 80.0, 43.2, 39.3, 35.7, 34.1, 28.4, 26.6, 24.9.

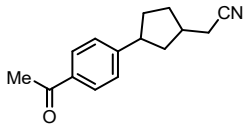


(4g) 2-((4-Acetylbenzyl)oxy)acetonitrile Colorless oil (16.2 mg, 43%). ^{1}H NMR (400 MHz, DMSO) δ

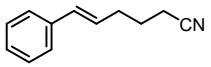
7.97 (d, J = 8.0 Hz, 2H), 7.50 (d, J = 8.0 Hz, 2H), 4.69 (s, 2H), 4.57 (s, 2H), 2.59 (s, 3H); ^{13}C NMR (101 MHz, DMSO) δ 198.1, 142.3, 136.9, 128.8, 128.2, 117.7, 72.3, 56.3, 27.2. HRMS (ESI) m/z calcd for $C_{11}H_{12}NO_2$ $[M+H]^+$ 190.0868, found 190.0864. IR (cm^{-1}): 3344, 2930, 2854, 2752, 2546, 2249, 2125, 2042, 1684, 1609, 1356, 1269, 1097, 1057, 822, 758, 594.



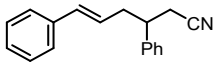
(4h) Tert-butyl (4-acetylbenzyl)(cyanomethyl)carbamate Colorless oil (25.2 mg, 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 7.6 Hz, 2H), 4.60 (s, 2H), 4.15 – 4.01 (m, 2H), 2.60 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.5, 141.4, 136.9, 129.0, 128.0, 115.5, 82.5, 50.3, 34.8, 29.7, 28.2, 26.7. HRMS (APCI) m/z calcd for $C_{16}H_{21}O_3N_2$ $[M+H]^+$ 289.1552, found 289.1542. IR (cm^{-1}): 2978, 2932, 2872, 1686, 1610, 1454, 1402, 1367, 1267, 1254, 1163, 1059, 943, 876, 814, 771, 683, 607.



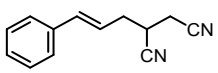
(4i) 3-(4-Acetylbenzyl)cyclopentane-1-carbonitrile Colorless oil (13.8 mg, 30%, dr = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.2 Hz, 2H), 7.31 (t, J = 7.8 Hz, 2H), 3.33 – 3.24 (m, 0.5H), 3.23 – 3.14 (m, 0.5H), 2.59 (s, 3H), 2.49 – 2.43 (m, 2H), 2.39 – 2.33(m, 1H), 2.25– 2.06 (m, 2H), 2.02 – 1.93 (m, 1H), 1.87 – 1.60 (m, 2H), 1.54 – 1.43 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 197.8, 151.0, 150.5, 135.4, 128.6, 127.2, 127.2, 118.9, 45.7, 44.4, 40.9, 39.3, 36.2, 35.5, 34.6, 33.0, 32.6, 31.1, 26.6, 23.4, 23.0. HRMS (ESI) m/z calcd for $C_{15}H_{17}NONa$ $[M+Na]^+$ 250.1208, found 250.1202. IR (cm^{-1}): 3466, 2999, 2955, 2916, 2864, 2243, 1680, 1607, 1419, 1358, 1308, 1271, 1184, 1055, 951, 831, 698, 600.



(6a) (*E*)-6-Phenylhex-5-enenitrile[14] Colorless oil (30.1 mg, 88%, *E/Z* = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 4H), 7.27 – 7.22 (m, 1H), 6.53 (d, J = 11.6 Hz, 0.2H), 6.47 (d, J = 15.6 Hz, 0.8H), 6.17 – 6.10 (m, 0.8H), 5.63 – 5.56 (m, 0.2H), 2.51 – 2.32 (m, 4H), 1.89 – 1.79 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.1, 132.1, 128.6, 127.6, 127.3, 126.1, 119.5, 31.6, 25.0, 16.4.

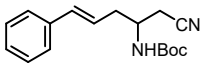


(6b) (*E*)-3,6-Diphenylhex-5-enenitrile[14] Colorless oil (23.7 mg, 48%, *E/Z* = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.35 (m, 2H), 7.31 – 7.23 (m, 7H), 7.22 – 7.18 (m, 1H), 6.68 (d, J = 15.6 Hz, 0.2H), 6.48 (d, J = 15.6 Hz, 0.8H), 6.11 – 6.00 (m, 0.94H), 5.57 – 5.47 (m, 0.15H), 4.27 – 4.23 (m, 0.2H), 3.90 – 3.63 (m, 1.2H), 3.45 – 3. 39 (m, 0.2H), 3.16 – 3.06 (m, 0.87H), 2.72 – 2.58 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.2, 137.0, 133.2, 128.9, 128.5, 127.5, 127.4, 127.1, 126.1, 126.1, 118.4, 42.1, 38.4, 23.9.

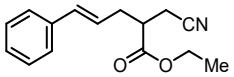


(6c) (*E*)-2-Cinnamylsuccinonitrile[17] Colorless oil (16.5 mg, 42%, *E/Z* = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.22 (m, 5H), 6.78 (d, J = 12.0 Hz, 0.06H), 6.65 (d, J = 16.0 Hz, 0.93H), 6.18 – 6.11

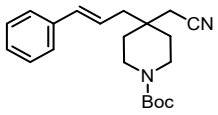
(m, 0.92H), 5.70 – 5.64 (m, 0.06H), 3.10 – 3.03 (m, 1H), 2.82 – 2.66 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.2, 135.9, 128.7, 128.3, 126.5, 121.4, 118.4, 115.4, 34.5, 28.4, 20.2.



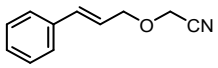
(6d) Tert-butyl (*E*)-(1-cyano-5-phenylpent-4-en-2-yl)carbamate[15] Colorless oil (52.5 mg, 92%, *E/Z* = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 4H), 7.26 – 7.23 (m, 1H), 6.64 (d, *J* = 11.6 Hz, 0.2H), 6.54 (d *J* = 16.0 Hz, 0.5H), 6.14 – 6.06 (m, 0.5H), 5.63 – 5.57 (m, 0.2H), 4.86 – 4.69 (m, 1H), 3.96 (s, 1H), 3.37 – 3.24 (m, 0.5H), 2.82 – 2.71 (m, 1H), 2.67 – 2.62 (m, 0.7H), 2.60 – 2.49 (m, 1.5H), 1.44 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.0, 136.6, 134.4, 128.7, 127.8, 126.3, 123.7, 117.3, 80.3, 47.1, 37.0, 28.3, 23.0.



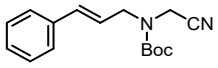
(6e) Ethyl (*E*)-2-(cyanomethyl)-5-phenylpent-4-enoate[14] Colorless oil (34.0 mg, 70%, *E/Z* = 15:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 4H), 7.25 – 7.22 (m, 1H), 6.61 (d, *J* = 11.6 Hz, 0.06H), 6.52 (d, *J* = 16.0 Hz, 0.90H), 6.10 – 6.02 (m, 0.91H), 5.59 – 5.55 (m, 0.07H), 4.26 – 4.16 (m, 2H), 2.92 – 2.85 (m, 1H), 2.75 – 2.58 (m, 4H), 1.29 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 136.6, 134.2, 128.6, 127.7, 126.3, 124.1 117.8, 61.6, 41.4, 34.5, 18.6, 14.2.



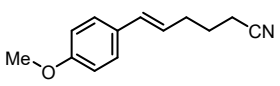
(6f) (*E*)-Tert-butyl 4-cinnamyl-4-(cyanomethyl)piperidine-1-carboxylate[14] Colorless oil (64.6 mg, 95%, *E/Z* = 4:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 5H), 6.68 (d, *J* = 11.6 Hz, 0.2H), 6.54 (d, *J* = 15.6 Hz, 0.8H), 6.17 – 6.09 (m, 0.8H), 5.67 – 5.60 (m, 0.2H), 3.48 – 3.39 (m, 3H), 3.26 – 3.18 (m, 1H), 2.81 (s, 0.4H), 2.43 – 2.29 (m, 3.9H), 1.64 – 1.56 (m, 4H), 1.46 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.7, 136.7, 134.9, 128.6, 127.6, 126.2, 123.1, 117.5, 79.8, 40.0, 35.9, 35.2, 34.1, 28.4, 26.0.



(6g) (*E*)-2-(Cinnamyloxy)acetonitrile[14] Colorless oil (15.9 mg, 46%, *E/Z* = 2:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.20 (m, 5H), 6.75 – 6.68 (m, 0.94H), 6.27 – 6.20 (m, 0.67H), 5.84 – 5.78 (m, 0.30H), 5.30 – 5.26 (m, 0.15H), 4.40 – 4.26 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.3, 128.7, 128.4, 128.3, 126.7, 123.0, 115.9, 71.7, 54.6.

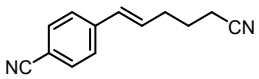


(6h) (*E*)-Tert-butyl cinnamyl(cyanomethyl)carbamate[14] Colorless oil (27.2 mg, 50%, *E/Z* = 9:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.35 (m, 2H), 7.32 – 7.29 (m, 2H), 7.25 – 7.22 (m, 1H), 6.71 – 6.68 (m, 0.1H), 6.57 – 6.53 (m, 0.9H), 6.13 – 6.05 (m, 0.9H), 5.65 – 5.59 (m, 0.1H), 4.12 – 4.09 (m, 4H), 1.49 (s, 8H), 1.46 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.0, 128.7, 128.1, 126.5, 123.2, 116.0, 82.0, 49.2, 34.3, 28.2.

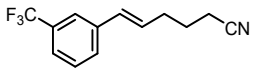


(6i) (*E*)-6-(4-Methoxyphenyl)hex-5-enenitrile[15] Colorless oil (19.3 mg, 55%, *E/Z* = 5:1). ^{1}H NMR (400

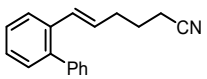
MHz, $CDCl_3$) δ 7.30 – 7.18 (m, 2H), 6.89 – 6.84 (m, 2H), 6.47 – 6.38 (m, 1H), 6.02 – 5.94 (m, 0.91H), 5.52 – 5.46 (m, 0.2H), 3.82 (s, 0.5H), 3.81 (s, 2.5H), 2.51 – 2.33 (m, 4H), 1.87 – 1.79 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.0, 131.4, 129.9, 129.9, 127.2, 125.3, 114.0, 55.3, 31.6, 25.1, 16.4.



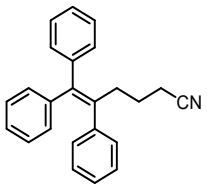
(6j) (*E*)-4-(5-Cyanopent-1-en-1-yl)benzonitrile[16] Colorless oil (16.3 mg, 48%, *E/Z* = 1.5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.58 (m, 2H), 7.43 – 7.41 (m, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 6.72 – 6.66 (m, 0.08H), 6.54 – 6.66 (m, 0.98H), 6.32 – 6.18 (m, 0.67H), 5.78 – 5.68 (m, 0.44H), 2.50 – 2.34 (m, 4H), 1.91- 1.79 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.5, 132.5, 131.9, 130.6, 126.6, 119.3, 119.0, 110.6, 31.8, 24.7, 16.6.



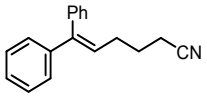
(6k) (E)-6-(3-(Trifluoromethyl)phenyl)hex-5-enenitrile[22] Colorless oil (27.7 mg, 58%, *E/Z* = 3:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (s, 1H), 7.52 – 7.40 (m, 3H), 6.56 – 6.48 (m, 0.98H), 6.26 – 6.14 (m, 0.79H), 5.73 – 5.66 (m, 0.26H), 2.49 – 2.33 (m, 4H), 1.90 – 1.79 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.0, 131.1 (q, *J* = 32.3 Hz), 130.9, 129.9, 129.5, 129.2, 127.0 (q, *J* = 273.6 Hz), 124.0 (q, *J* = 3.8 Hz), 122.8 (q, *J* = 3.7 Hz), 119.6, 31.8, 25.0, 16.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.78.



(6l) (E)-6-([1,1'-Biphenyl]-2-yl)hex-5-enenitrile Colorless oil (21.3 mg, 43%, *E/Z* = 5:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.56 (m, 1H), 7.45 – 7.27 (m, 8H), 6.46 – 6.40 (m, 0.98H), 6.09 – 6.01 (m, 0.84H), 5.54 – 5.47 (m, 0.18H), 2.35 – 2.26 (m, 4H), 1.83 – 1.65 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0, 140.5, 135.3, 131.0, 130.2, 129.7, 128.6, 128.1, 127.5, 127.3, 127.1, 126.0, 119.5, 31.8, 25.0, 16.4. HRMS (APCI) m/z calcd for $C_{18}H_{18}N$ $[M+H]^+$ 248.1439, found 248.1434. IR (cm^{-1}): 3718, 3142, 2935, 2845, 2677, 2540, 2411, 2289, 2245, 2166, 2030, 1807, 1691, 1560, 1475, 1367, 1169, 968, 746, 615, 432.

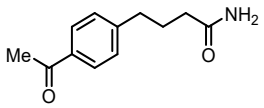


(6m) 5,6,6-Triphenylhex-5-enenitrile Colorless oil (22.6 mg, 35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (t, *J* = 7.6 Hz, 2H), 7.30 (d, *J* = 7.2 Hz, 1H), 7.24 – 7.10 (m, 7H), 7.05 – 7.01 (m, 3H), 6.90 – 6.88 (m, 2H), 2.60 – 2.56 (m, 2H), 2.19 (t, *J* = 7.2 Hz, 2H), 1.73 – 1.65 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.9, 142.3, 141.3, 141.0, 138.4, 130.5, 129.5, 129.1, 128.5, 128.2, 127.5, 127.0, 126.7, 126.1, 34.9, 24.8, 17.1. HRMS (ESI) m/z calcd for $C_{24}H_{21}NNa$ $[M+Na]^+$ 346.1572, found 346.1566. IR (cm^{-1}): 3466, 2993, 2908, 2851, 2586, 2334, 2243, 2083, 1990, 1491, 1435, 1406, 1315, 1057, 947, 762, 698.

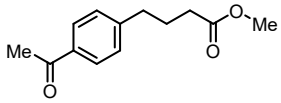


(6n) 6,6-Diphenylhex-5-enenitrile[21] Colorless oil (11.5 mg, 23%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.37 (m, 2H), 7.35 – 7.31 (m, 1H), 7.30 – 7.22 (m, 5H), 7.17 – 7.15 (m, 2H), 6.02 (t, *J* = 7.6 Hz, 1H),

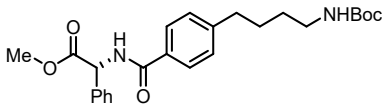
2.33 – 2.24 (m, 4H), 1.84 – 1.77 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.8, 142.1, 139.6, 129.6, 128.3, 128.1, 127.2, 127.1, 126.6, 119.5, 28.7, 25.7, 16.6.



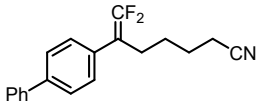
(7) 4-(4-Acetylphenyl)butanamide Colorless oil (33.6 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.4 Hz, 2H), 5.50 (d, J = 40.0 Hz, 2H), 2.74 (t, J = 7.6 Hz, 2H), 2.59 (s, 3H), 2.24 (t, J = 7.6 Hz, 2H), 2.04 – 1.97 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.9, 174.7, 147.3, 135.3, 128.7, 128.6, 35.1, 34.8, 26.6, 26.4. HRMS (ESI) m/z calcd for $C_{12}H_{15}NO_2Na$ $[M+Na]^+$ 228.1000, found 228.0995. IR (cm^{-1}): 3391, 3200, 2955, 2918, 2872, 1670, 1605, 1425, 1360, 1269, 1184, 960, 835, 661, 598, 461.



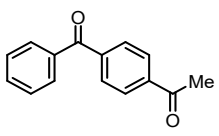
(8) Methyl 4-(4-acetylphenyl)butanoate[18] Colorless oil (32.1 mg, 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 3.67 (s, 3H), 2.72 (t, J = 7.6 Hz, 2H), 2.59 (s, 3H), 2.34 (t, J = 7.6 Hz, 2H), 2.02 – 1.94 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.8, 173.7, 147.2, 135.3, 128.7, 128.6, 51.6, 35.1, 33.3, 26.6, 26.1.



(9) Methyl (*R*)-2-(4-(4-((tert-butoxycarbonyl)amino)butyl)benzamido)-2-phenylacetate Colorless oil (55.5 mg, 63%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.0 Hz, 2H), 7.46 – 7.43 (m, 2H), 7.40 – 7.31 (m, 3H), 7.26 – 7.22 (m, 2H), 7.11 (d, J = 6.8 Hz, 1H), 5.78 (d, J = 6.8 Hz, 1H), 4.50 (s, 1H), 3.77 (s, 3H), 3.13 (d, J = 6.0 Hz, 2H), 2.67 (t, J = 7.6 Hz, 2H), 1.66 – 1.61 (m, 3H), 1.51 – 1.44 (m, 10H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.6, 166.5, 156.0, 146.6, 136.7, 131.2, 129.0, 129.0, 128.6, 128.6, 127.3, 127.3, 56.8, 52.9, 40.3, 35.4, 29.7, 28.4, 28.3. HRMS (ESI) m/z calcd for $C_{25}H_{32}N_2O_5Na$ $[M+Na]^+$ 463.2209, found 463.2204. IR (cm^{-1}): 3294, 2978, 2926, 2858, 1745, 1703, 1649, 1537, 1497, 1435, 1364, 1248, 1169, 1047, 947, 864, 766, 702.



(12) 6-([1,1'-Biphenyl]-4-yl)-7,7-difluorohept-6-enenitrile[19] Colorless oil (33.9 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, J = 8.0 Hz, 4H), 7.45 (t, J = 8.0 Hz, 2H), 7.39 – 7.34 (m, 3H), 2.52 – 2.48 (m, 2H), 2.33 (t, J = 7.2 Hz, 2H), 1.73 – 1.66 (m, 2H), 1.61 – 1.54 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.8 (t, J = 289.5 Hz), 140.4, 140.3, 132.0, 128.8, 128.5 (t, J = 3,2 Hz), 127.5, 127.3, 127.0, 119.4, 91.3 (dd, J = 19.3 Hz, 15.9 Hz), 26.8, 26.7, 24.7, 16.9.



(13) 1-(4-Benzoylphenyl)ethan-1-one[20] Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.0

Hz, 2H), 7.87 (d, J = 8.4 Hz, 2H), 7.81 (d, J = 6.8 Hz, 2H), 7.63 (t, J = 7.2 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 2.67 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 196.0, 141.4, 139.6, 137.0, 133.0, 130.1, 130.1, 128.5, 128.2, 26.9.

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Yang, Visible-Light-Promoted Cross-Coupling of *O*-Aryl Oximes and Nitrostyrenes to Access Cyanoalkylated Alkenes, *Org. Lett.*, 2022, **24**, 4640–4644.
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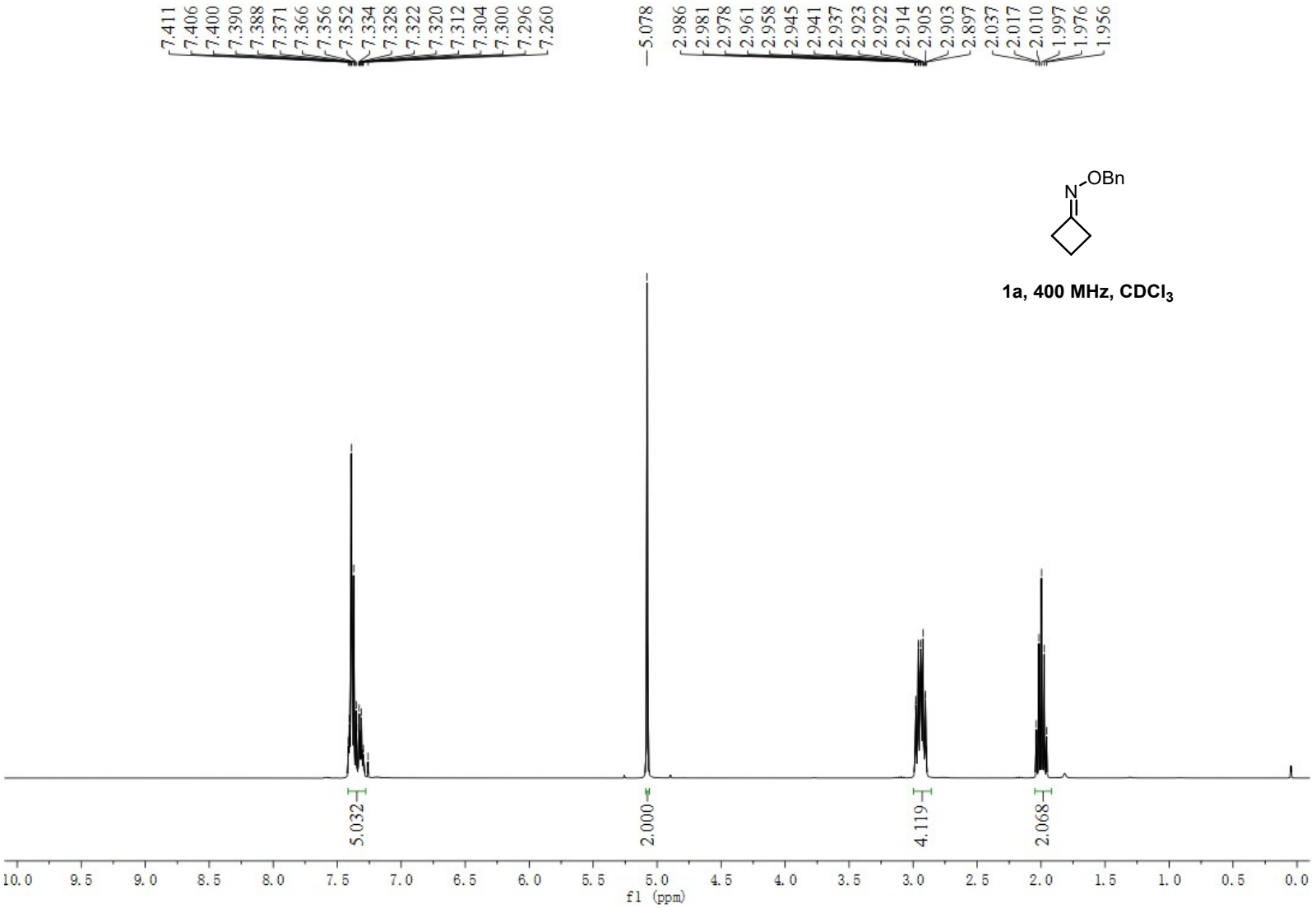
11. Copies of NMR spectra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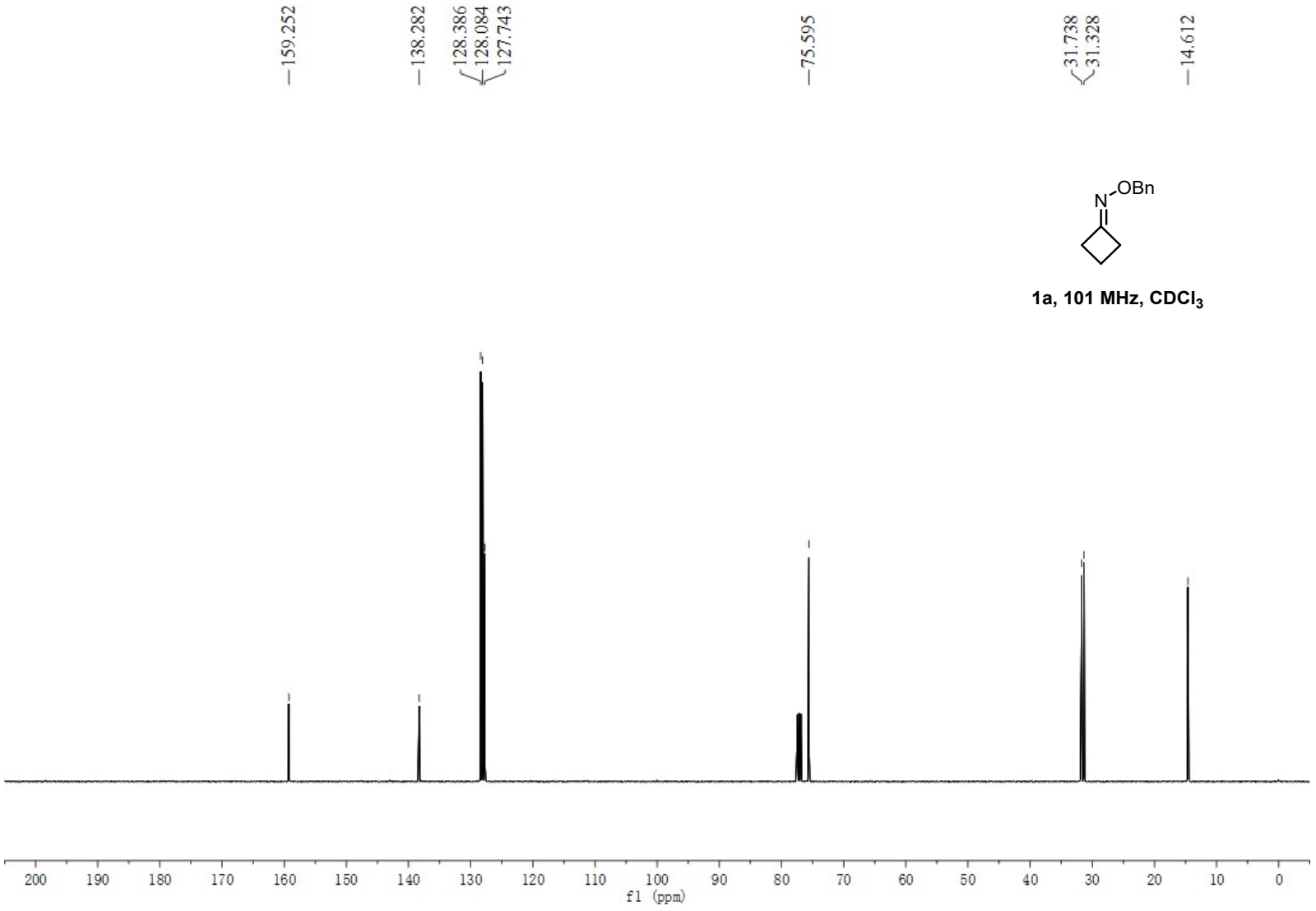

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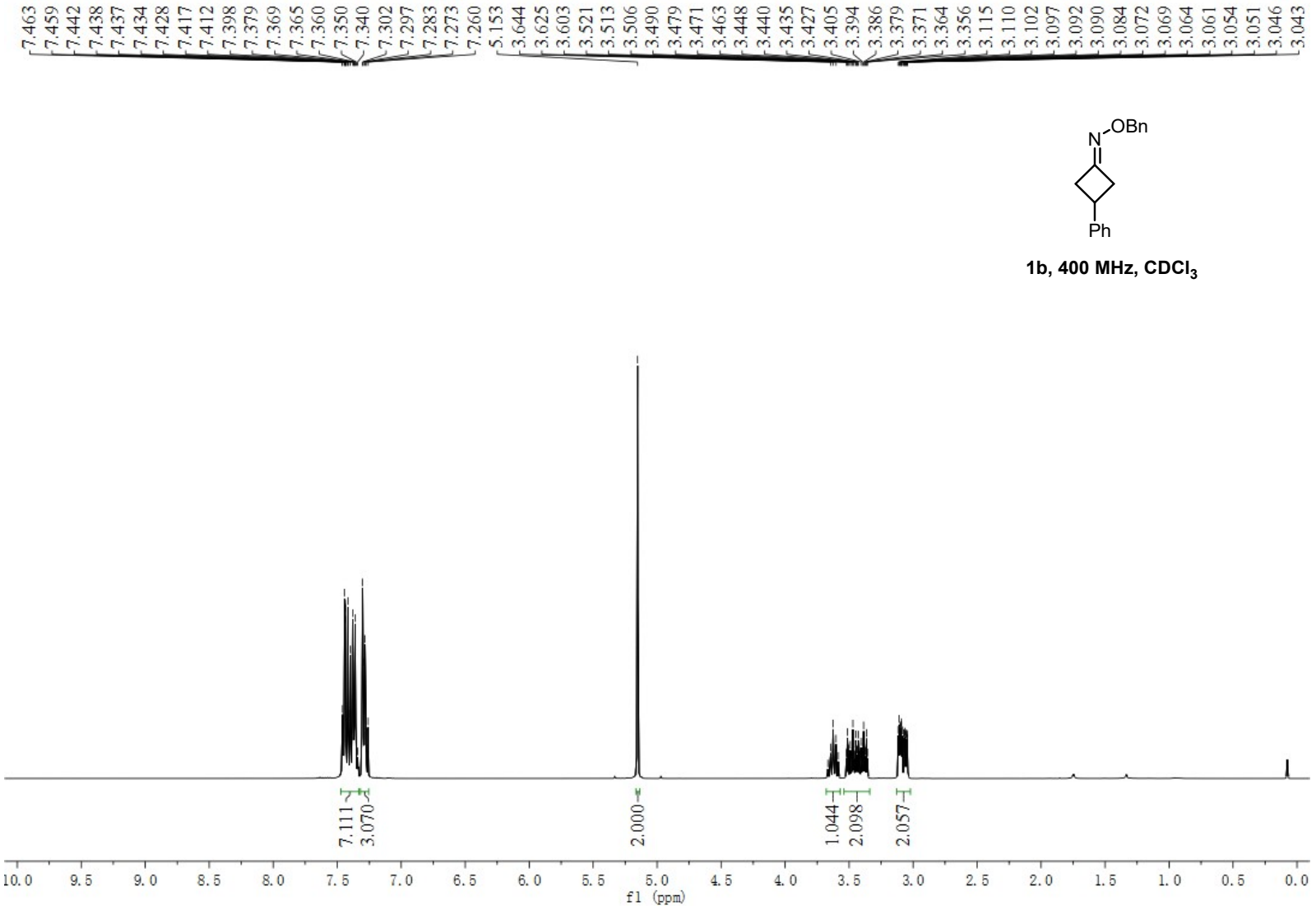

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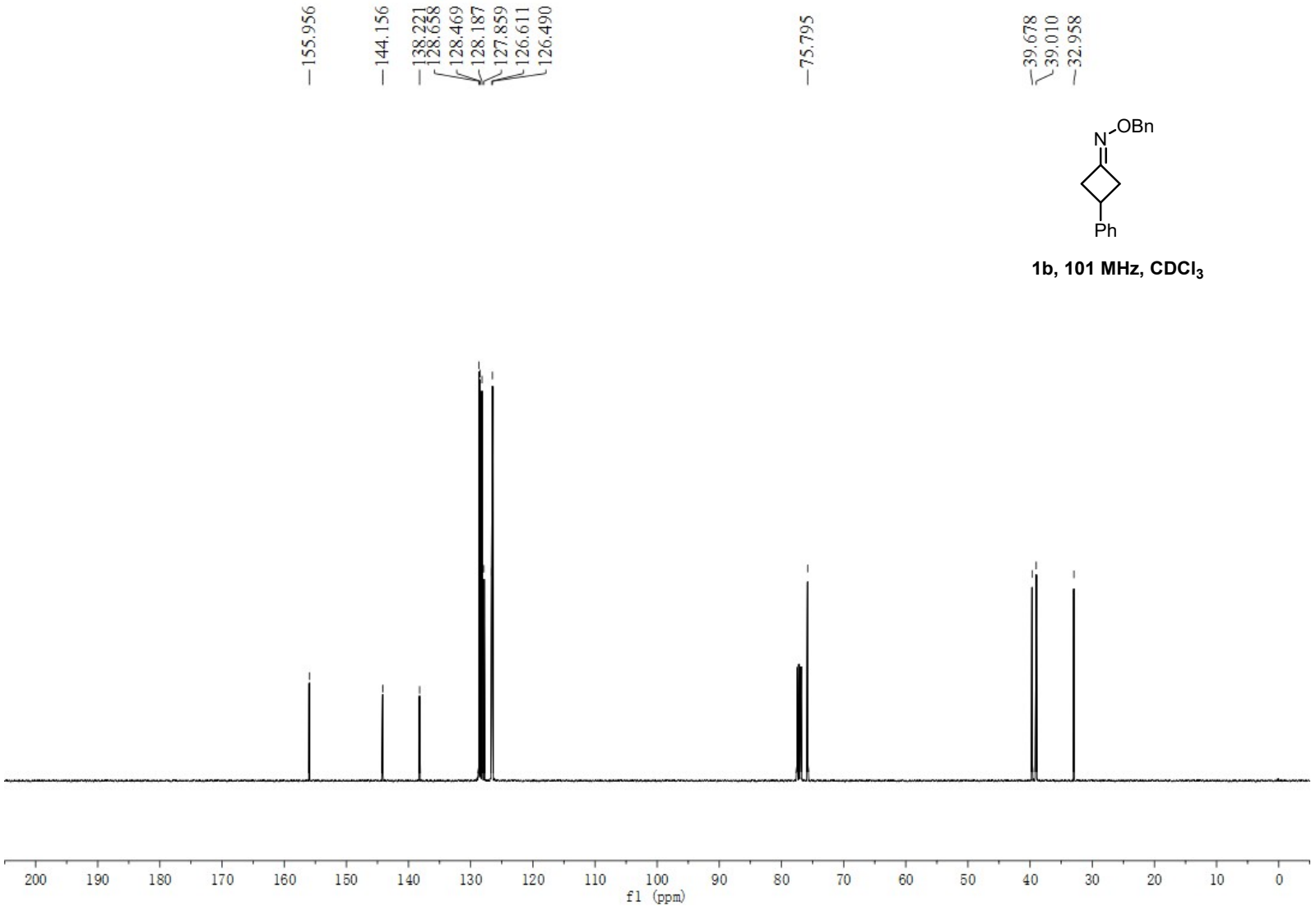

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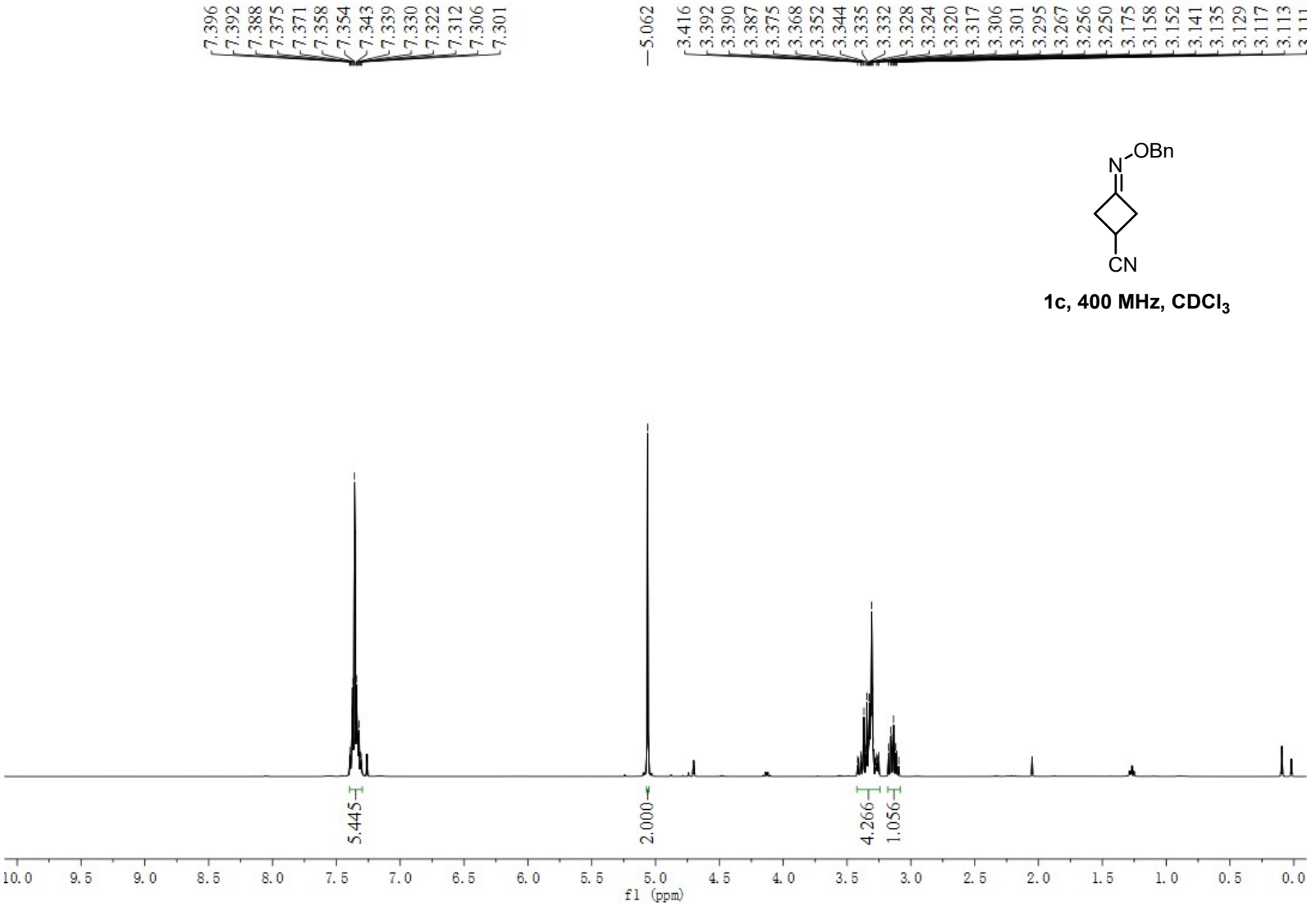

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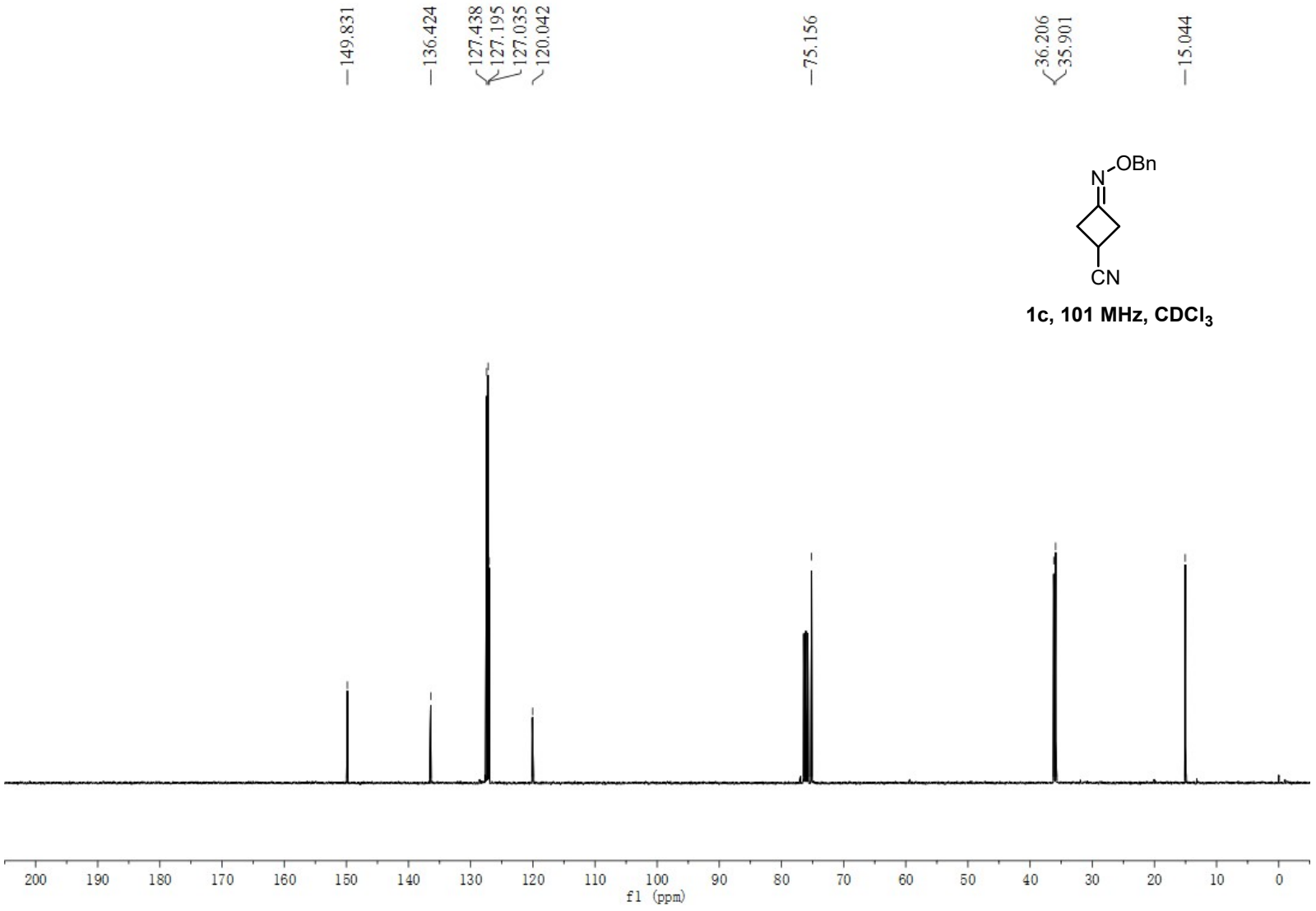

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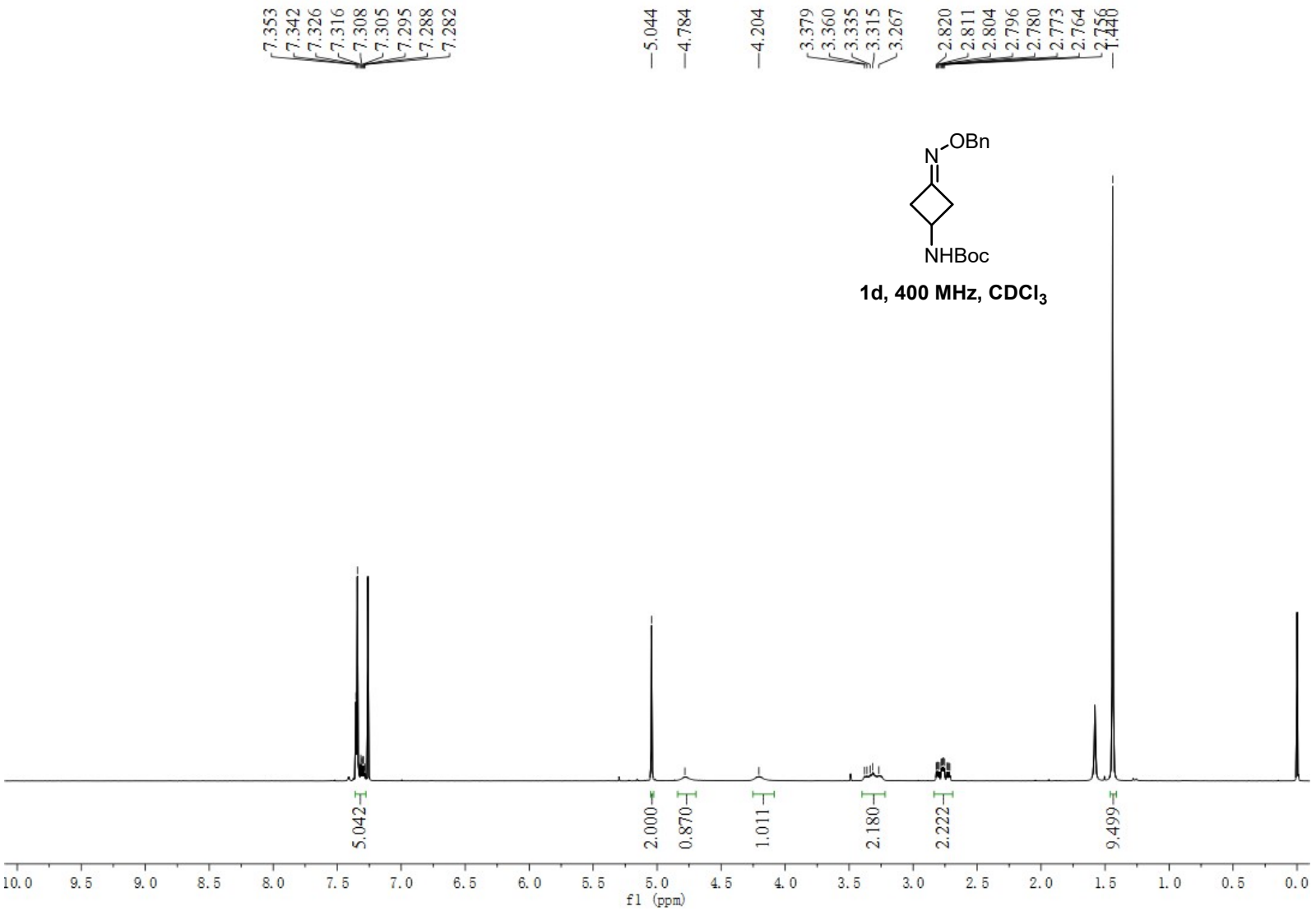
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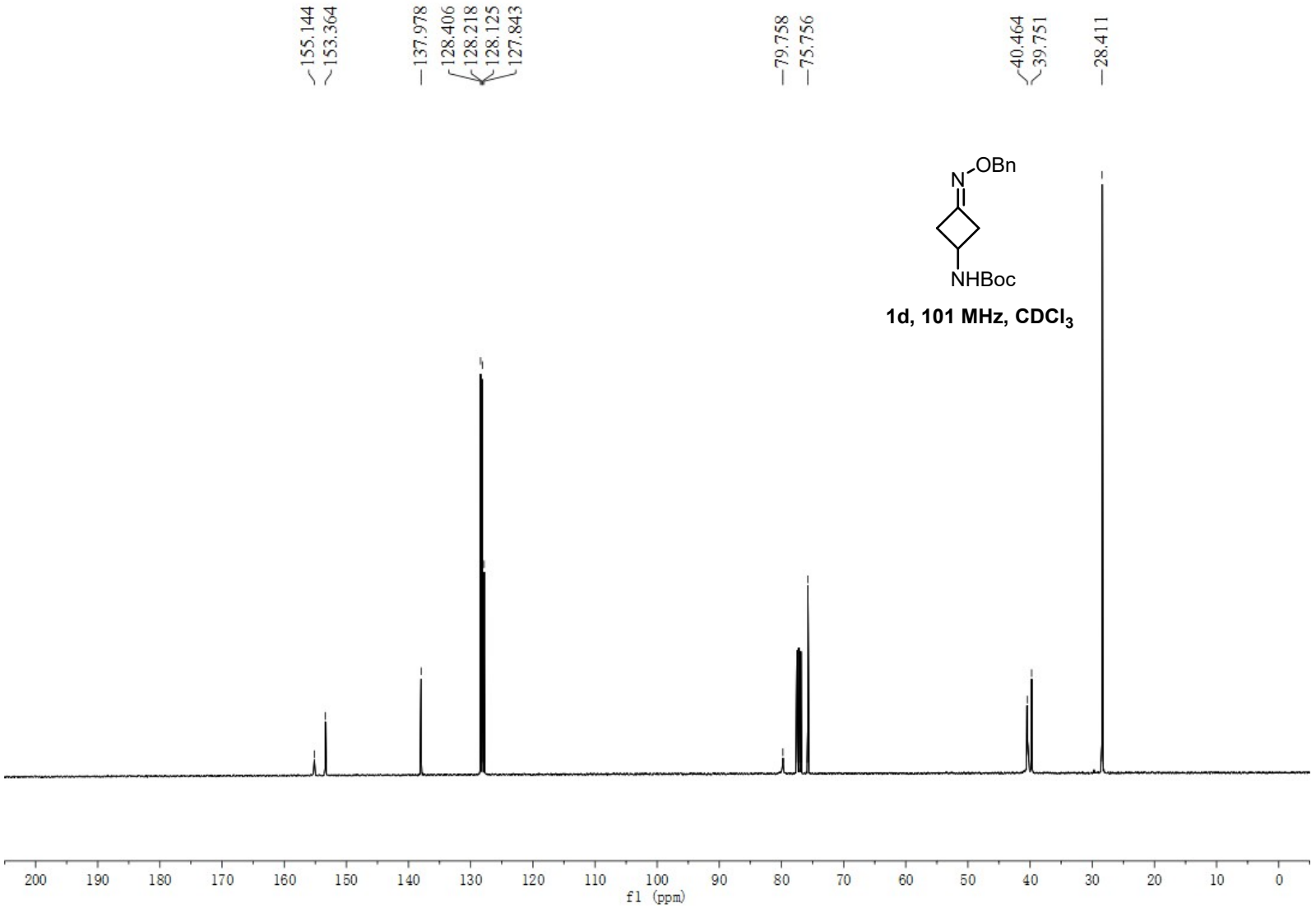
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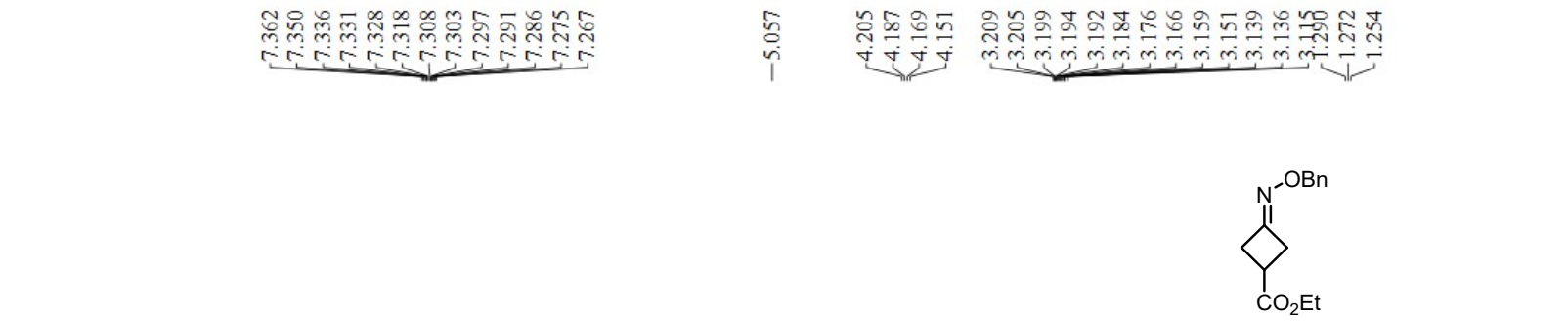
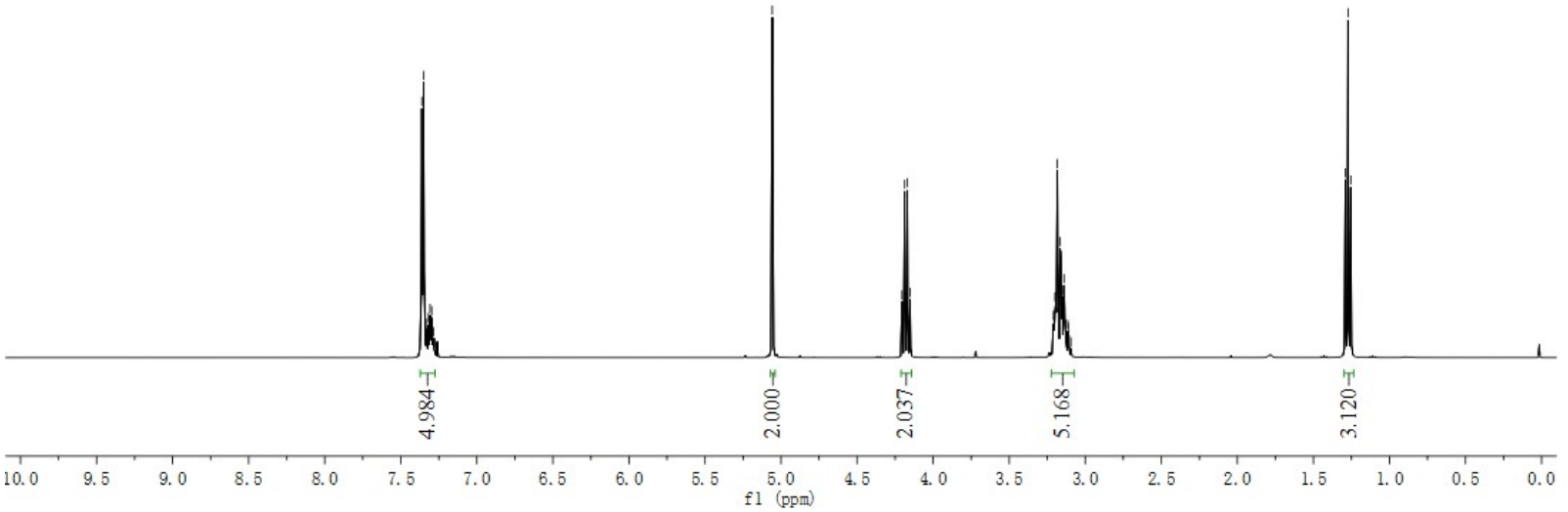
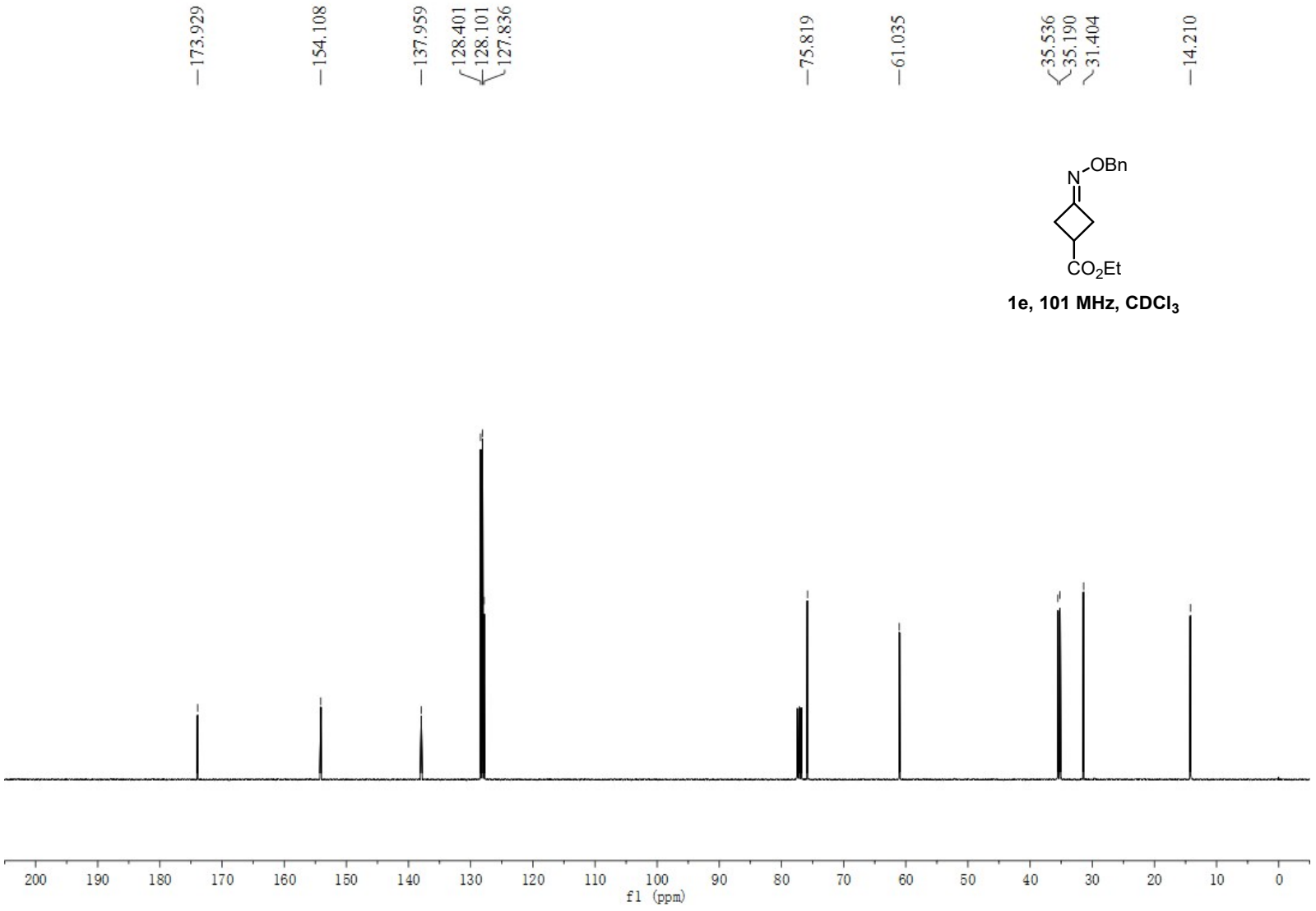
f1 (ppm)

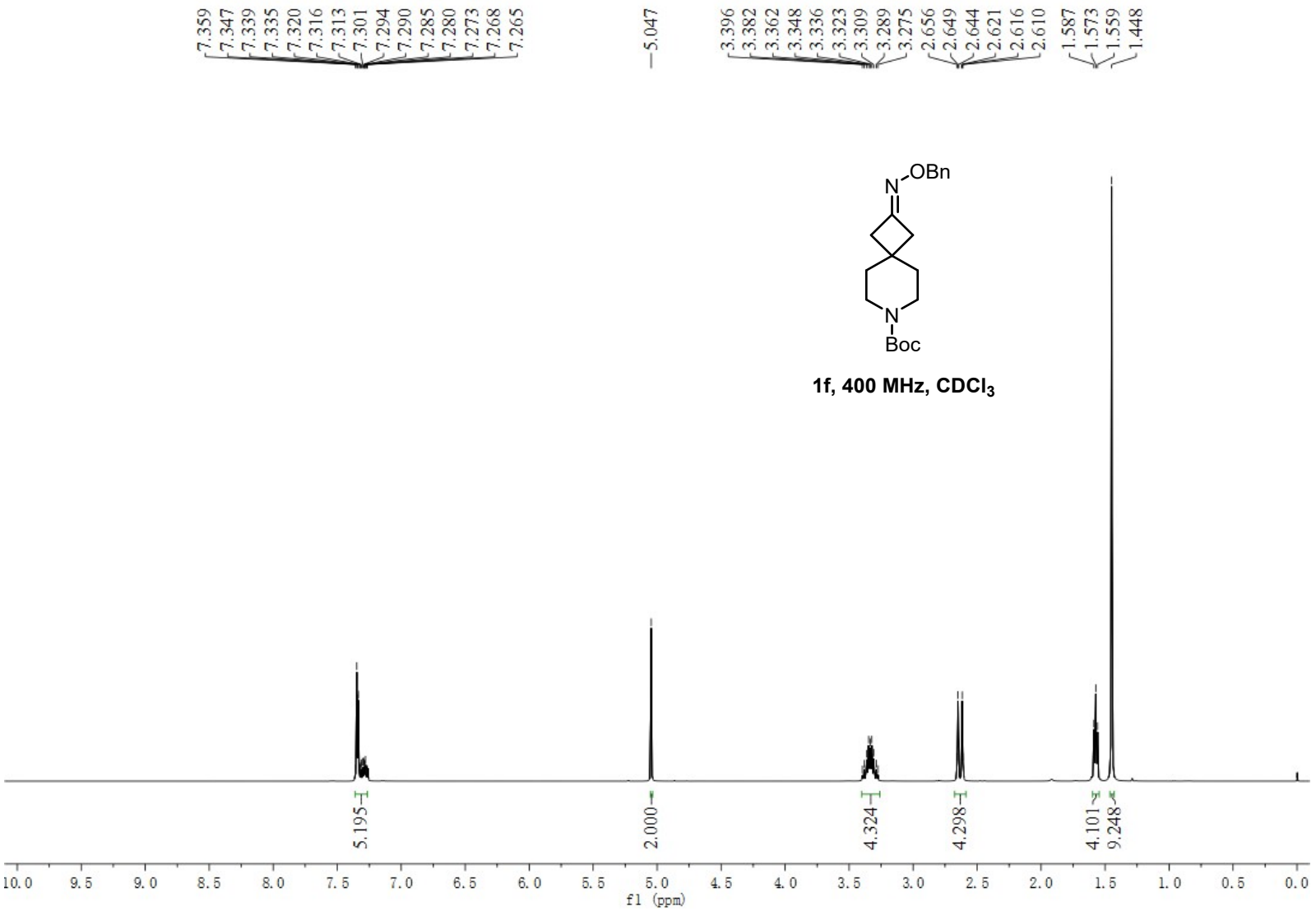


1e, 400 MHz, $CDCl_3$

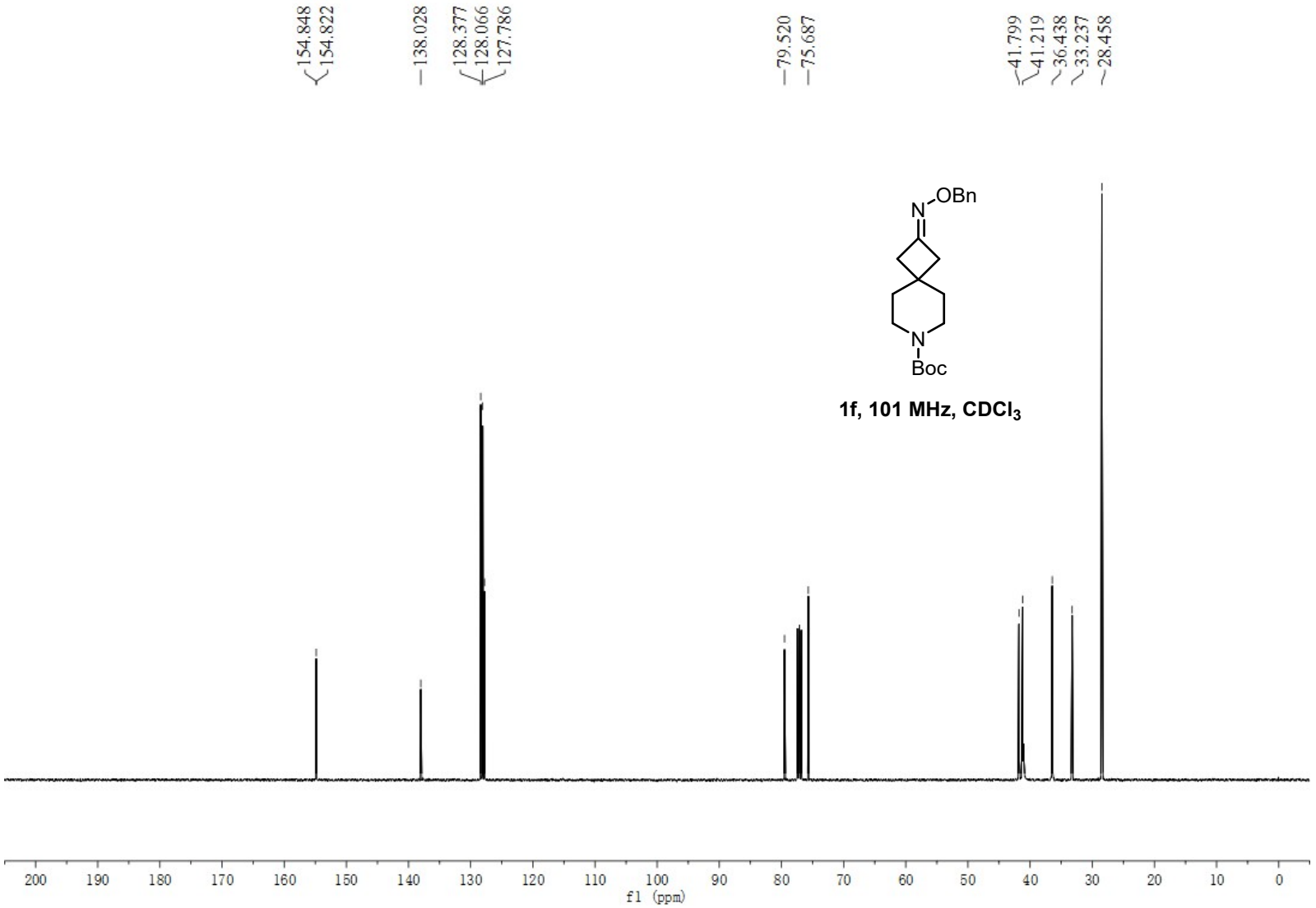




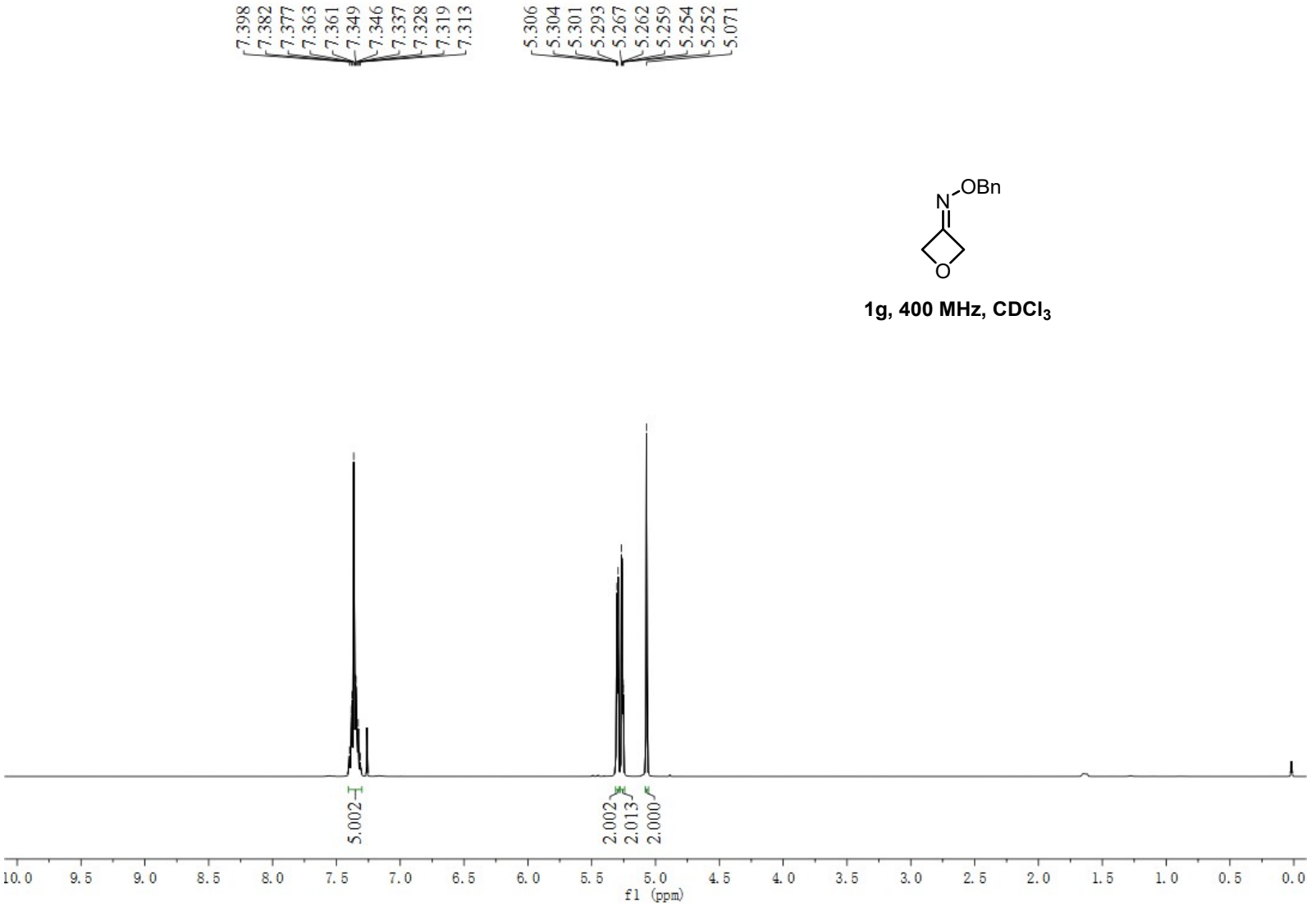
1e, 101 MHz, $CDCl_3$



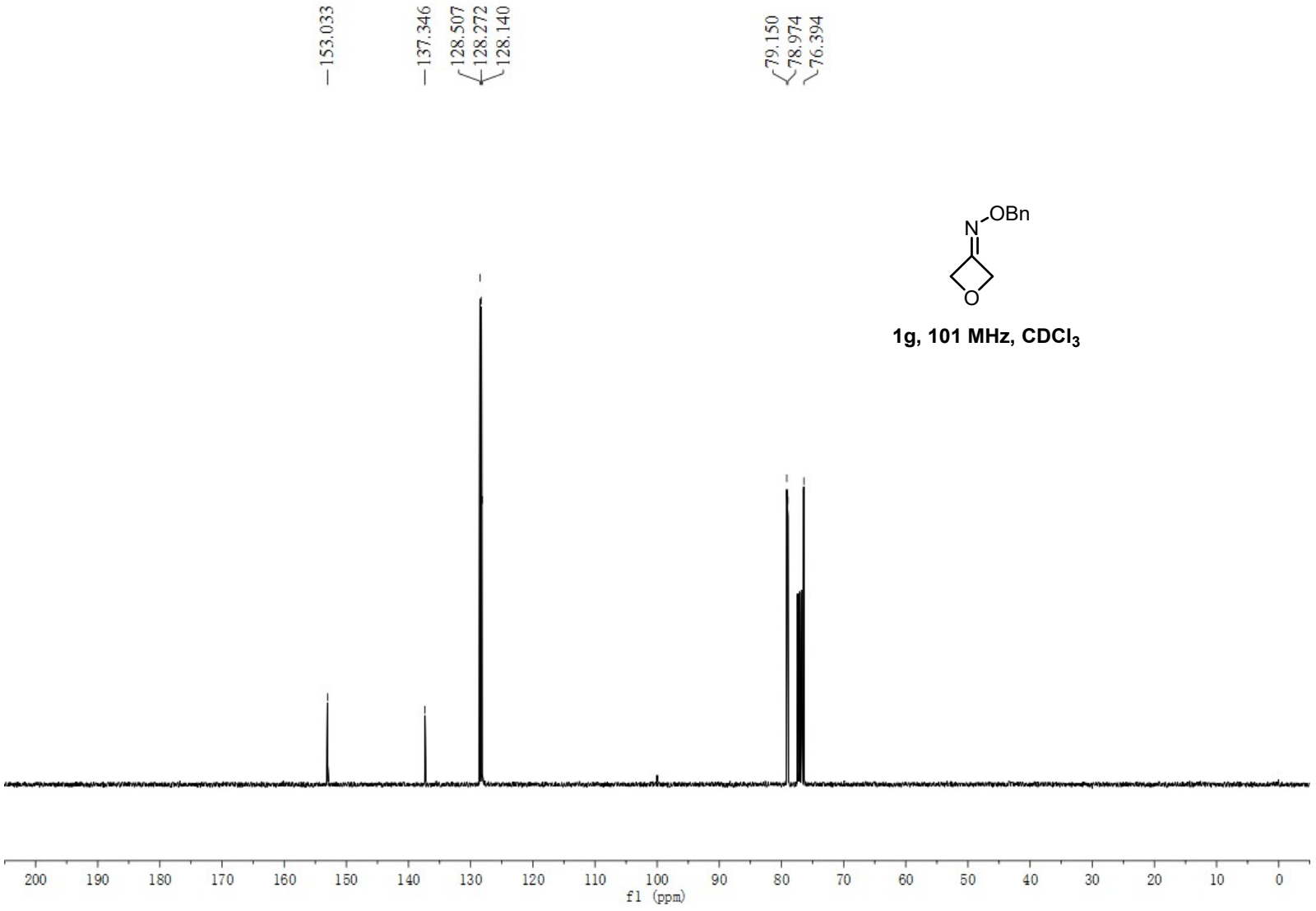
1f, 400 MHz, $CDCl_3$



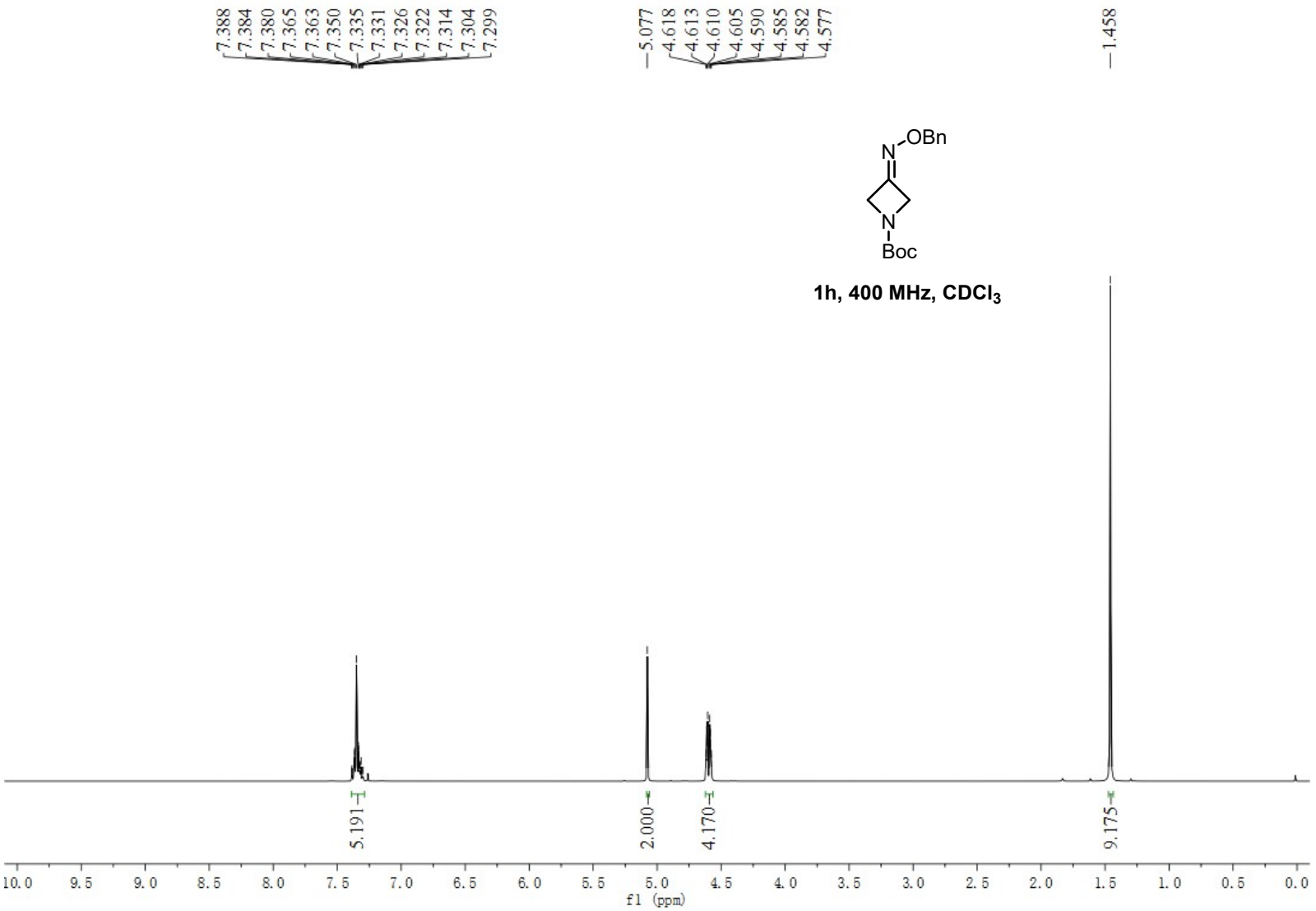
1f, 101 MHz, $CDCl_3$

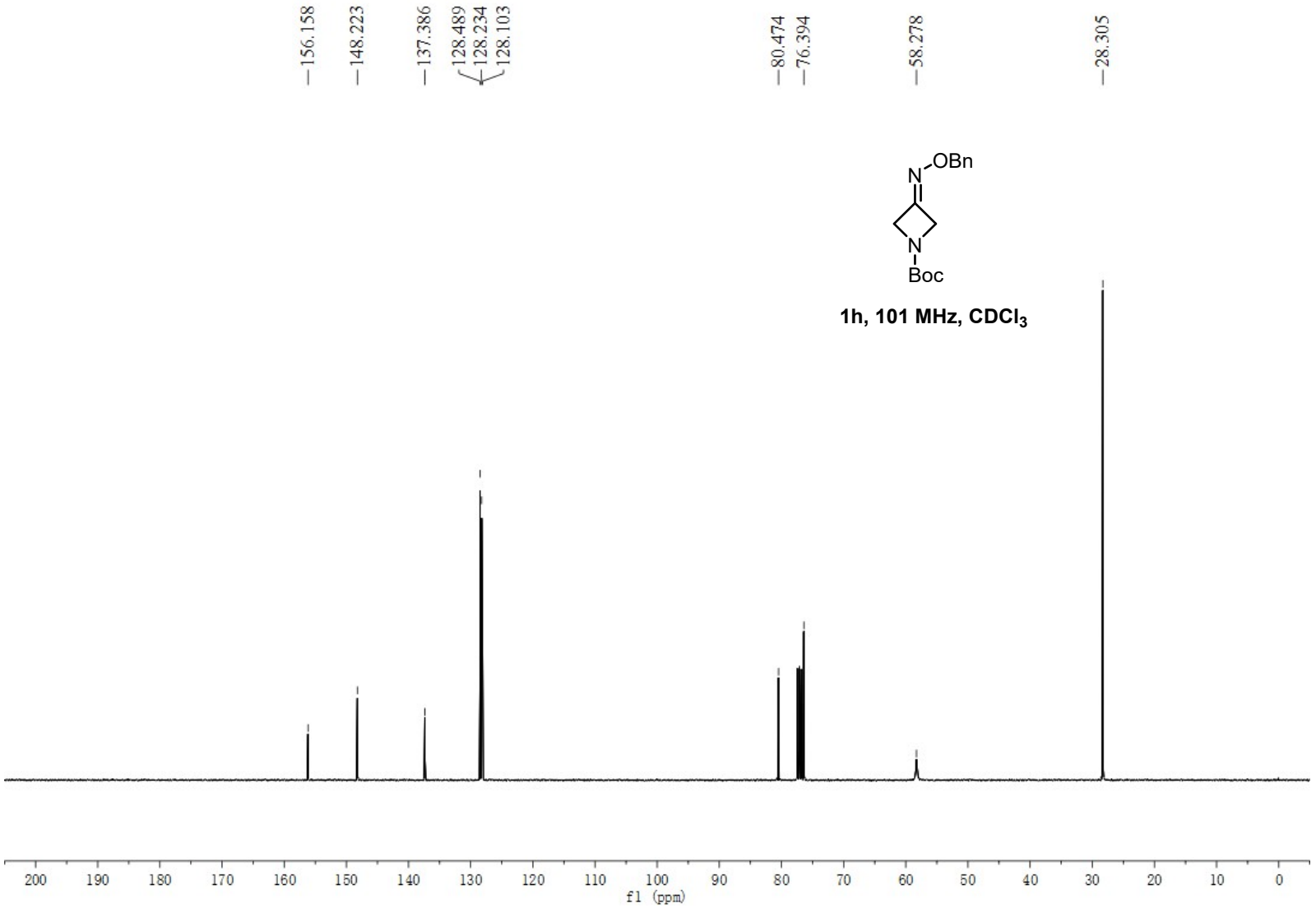


1g, 400 MHz, $CDCl_3$

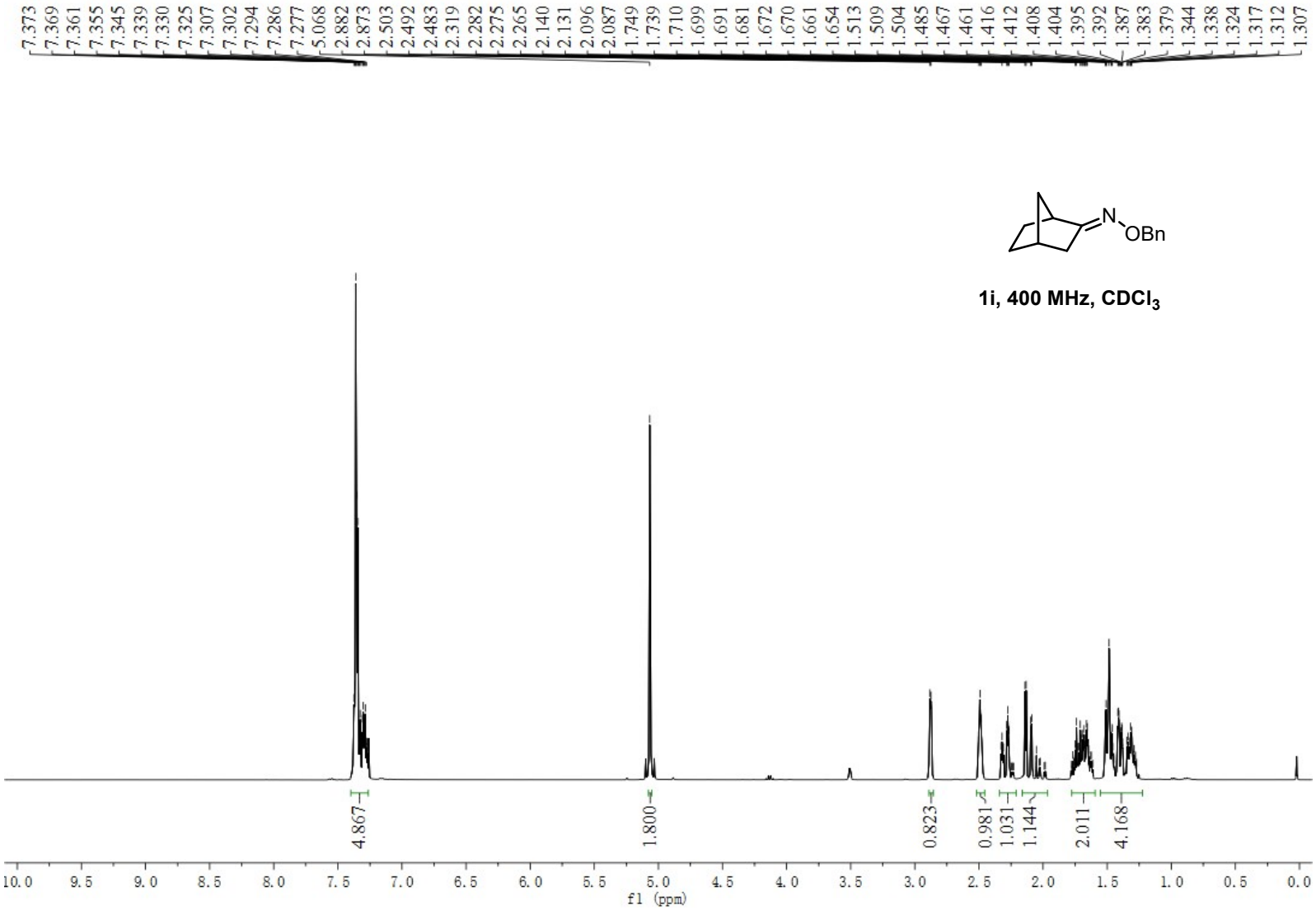


1g, 101 MHz, $CDCl_3$

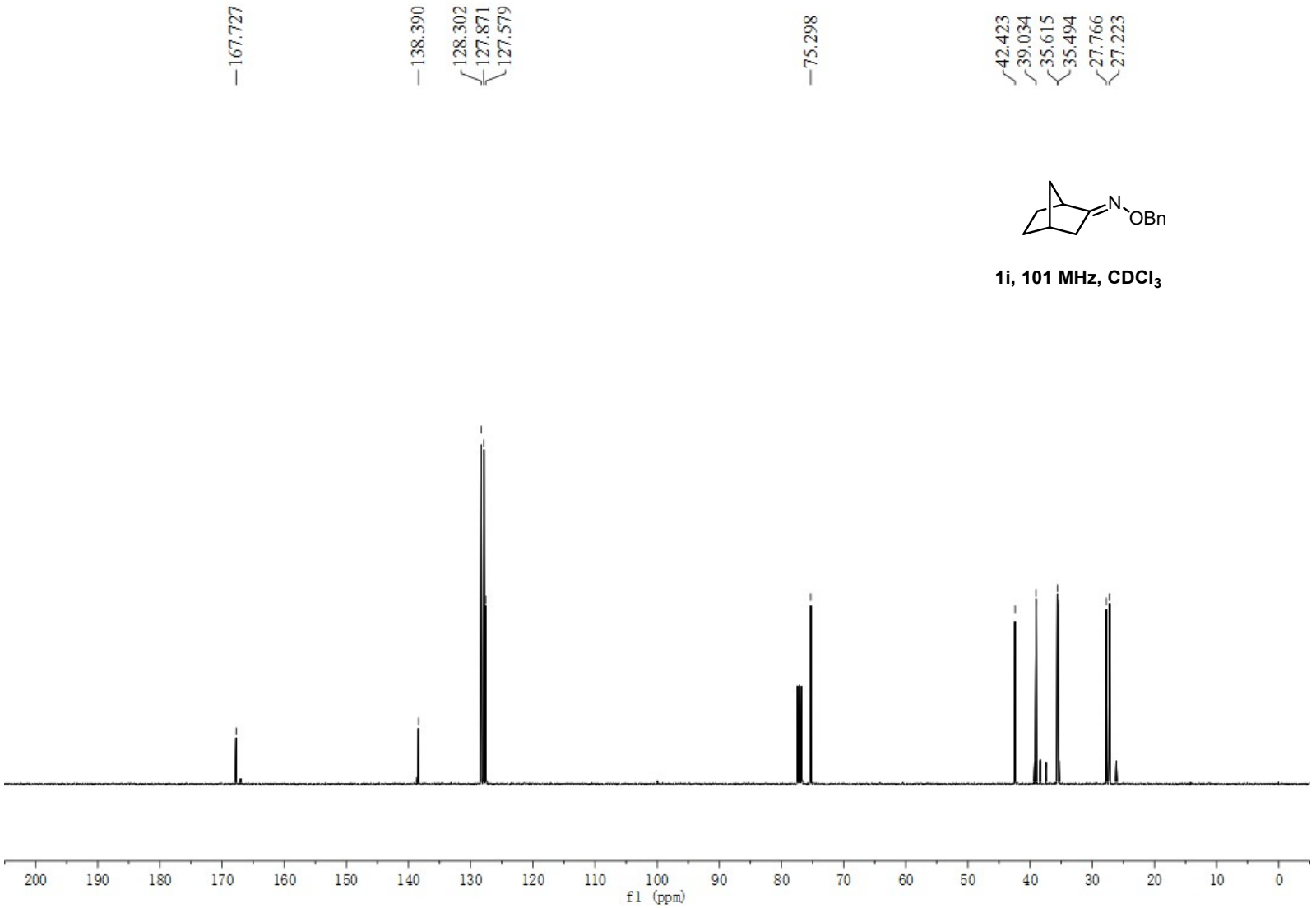

7.388
7.384
7.380
7.365
7.363
7.350
7.335
7.331
7.326
7.322
7.314
7.304
7.299
5.077
4.618
4.613
4.610
4.605
4.590
4.585
4.582
4.577
1.458
OBn
N
N
Boc
1h, 400 MHz, CDCl3
5.191
2.000
4.170
9.175
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



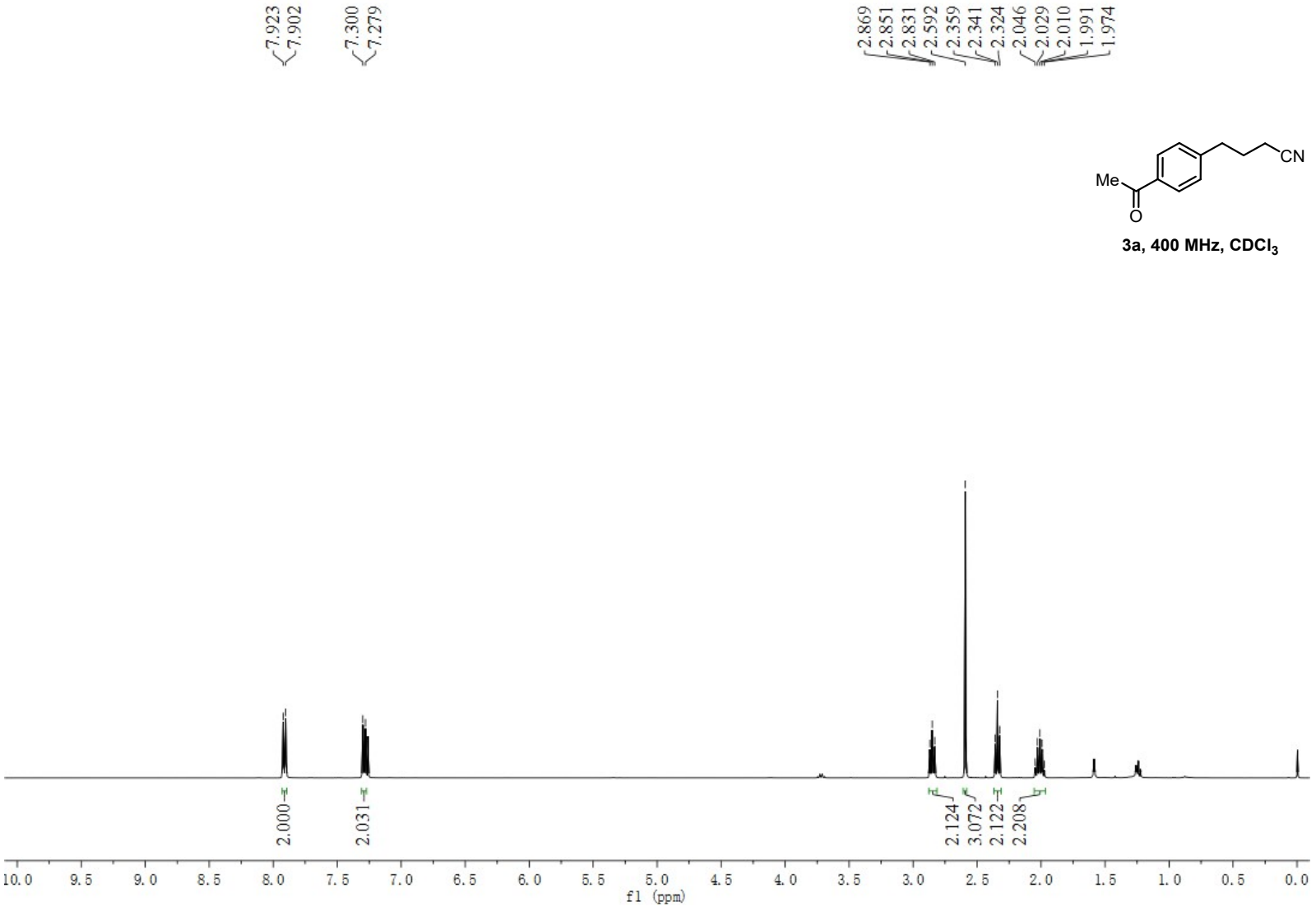
1h, 101 MHz, $CDCl_3$



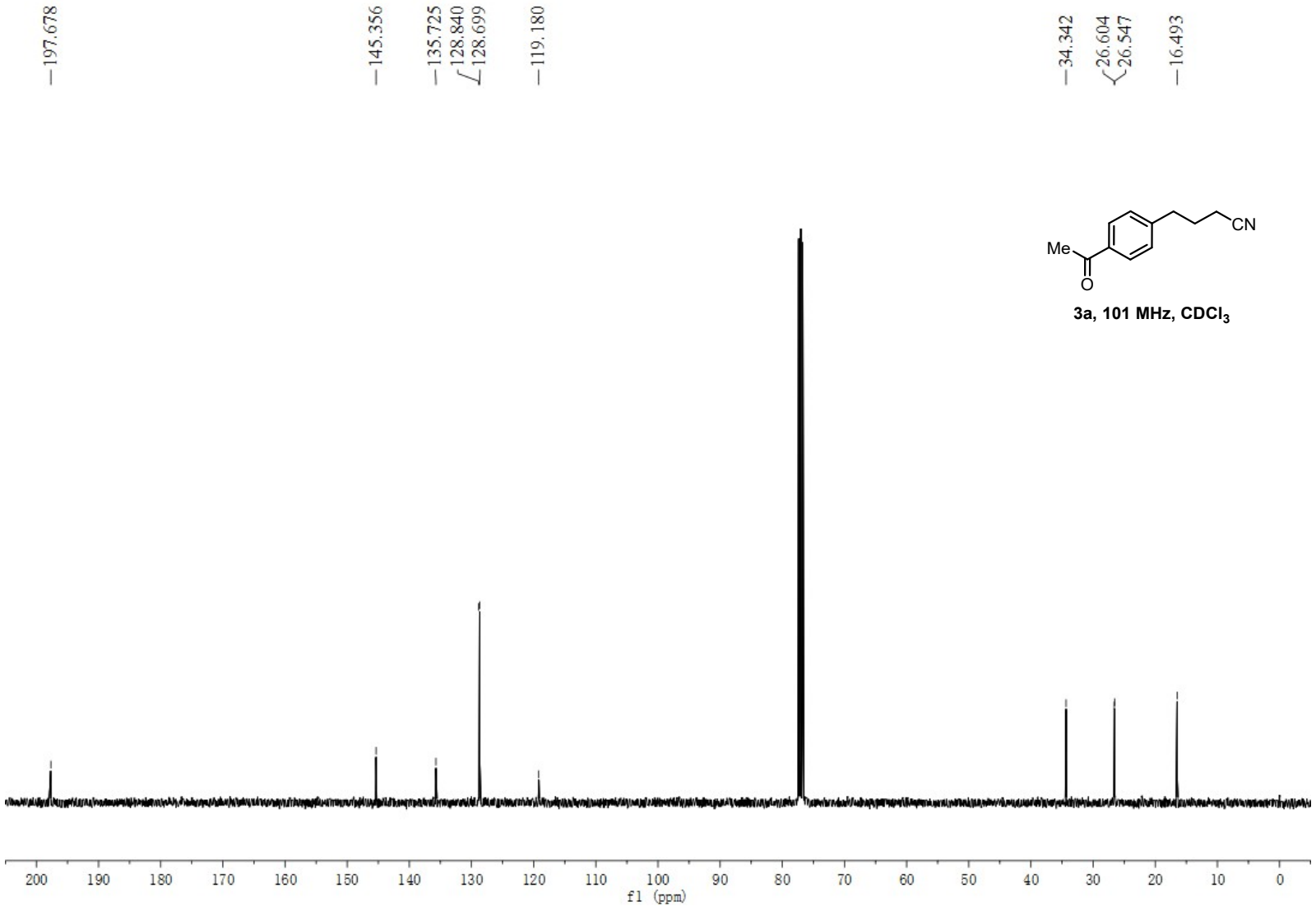
1i, 400 MHz, $CDCl_3$



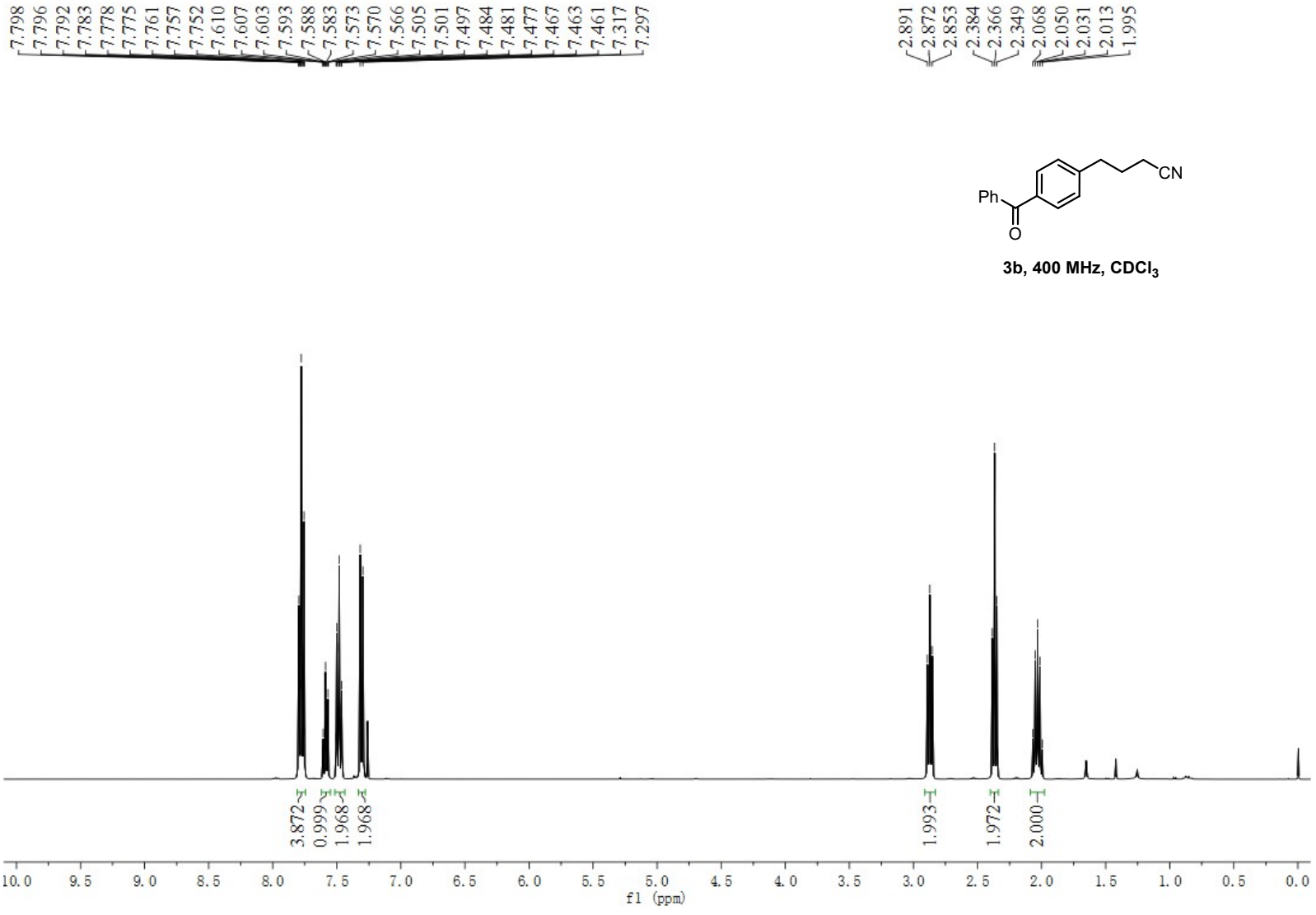
1i, 101 MHz, $CDCl_3$



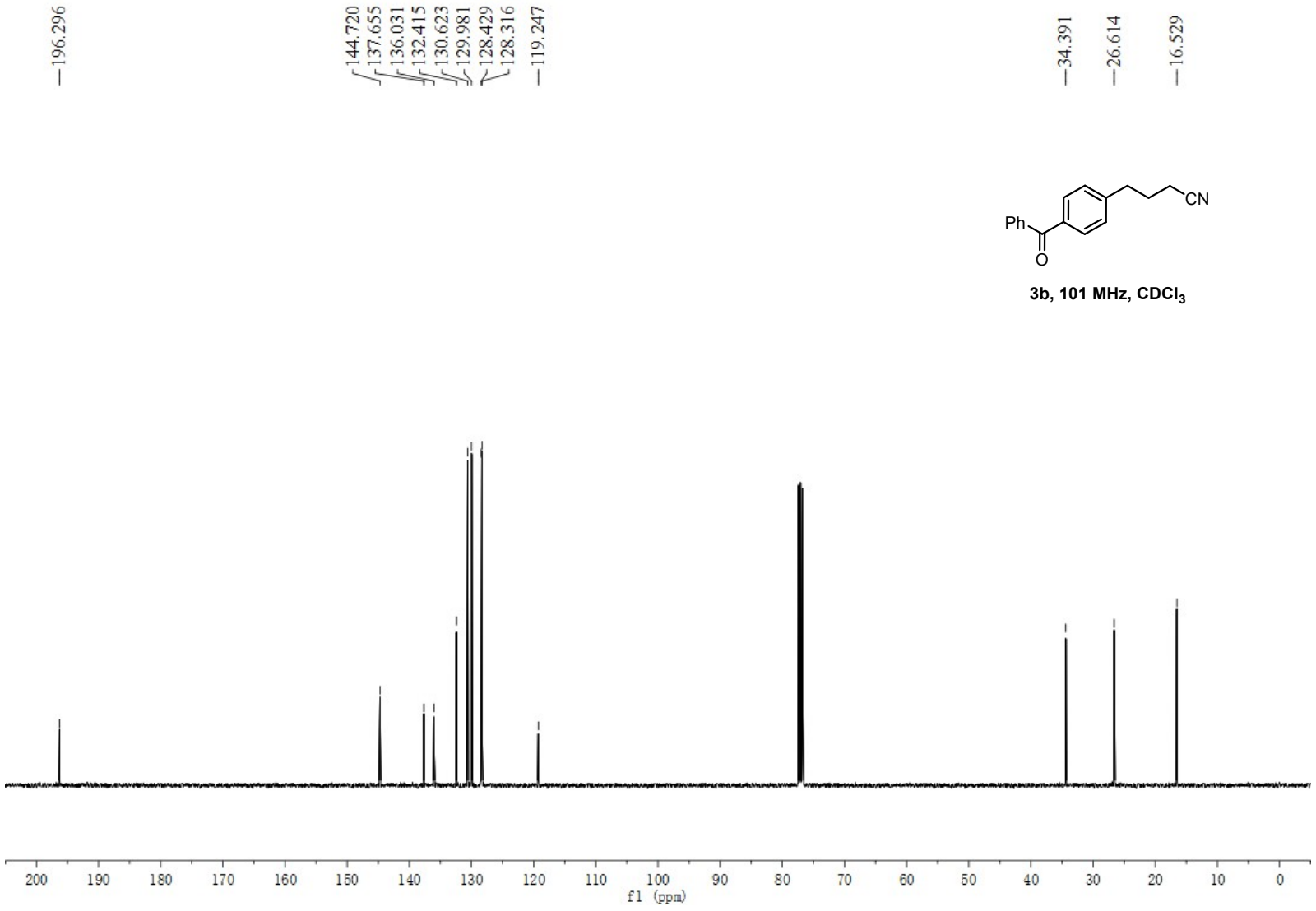
3a, 400 MHz, $CDCl_3$



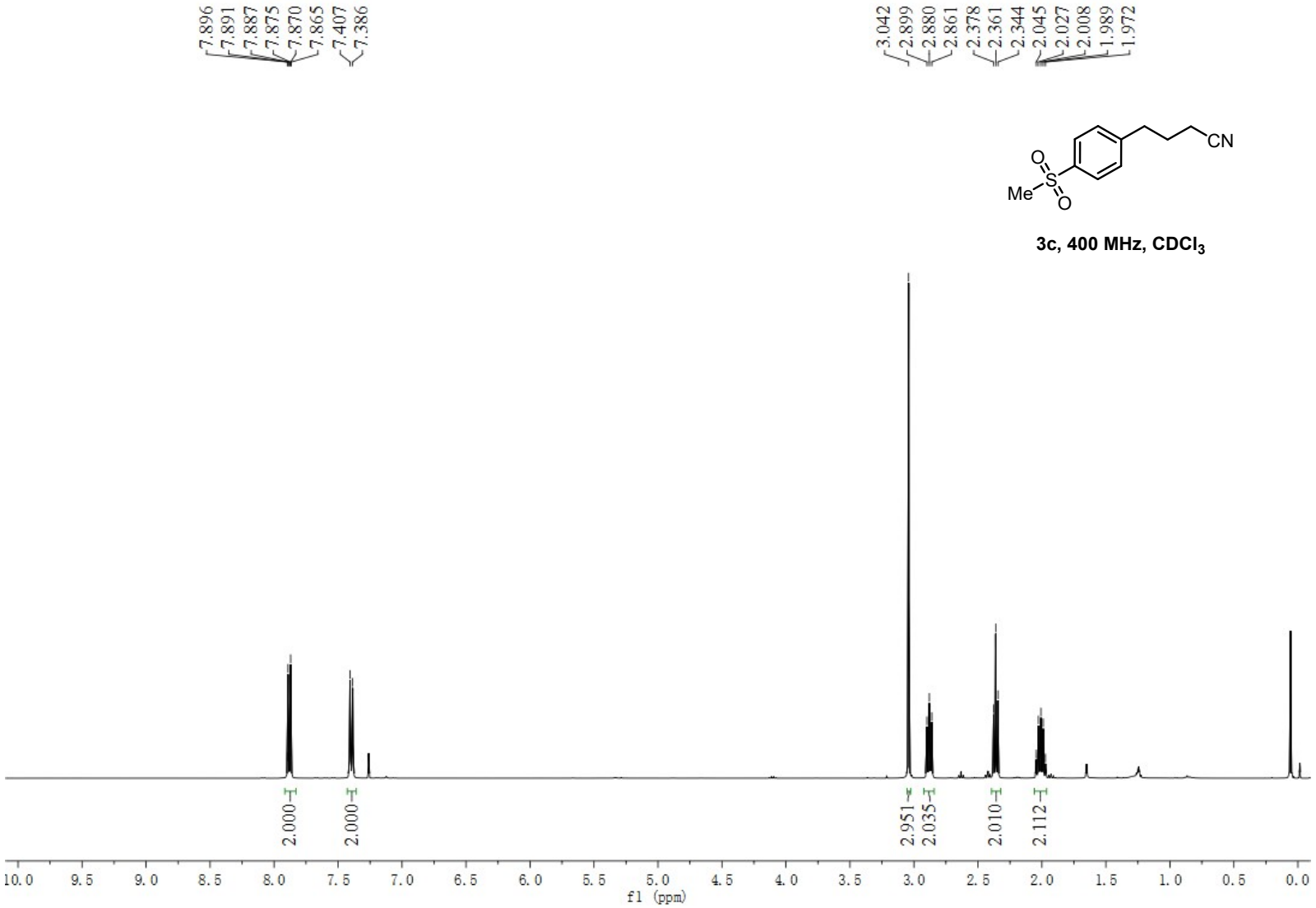
3a, 101 MHz, $CDCl_3$



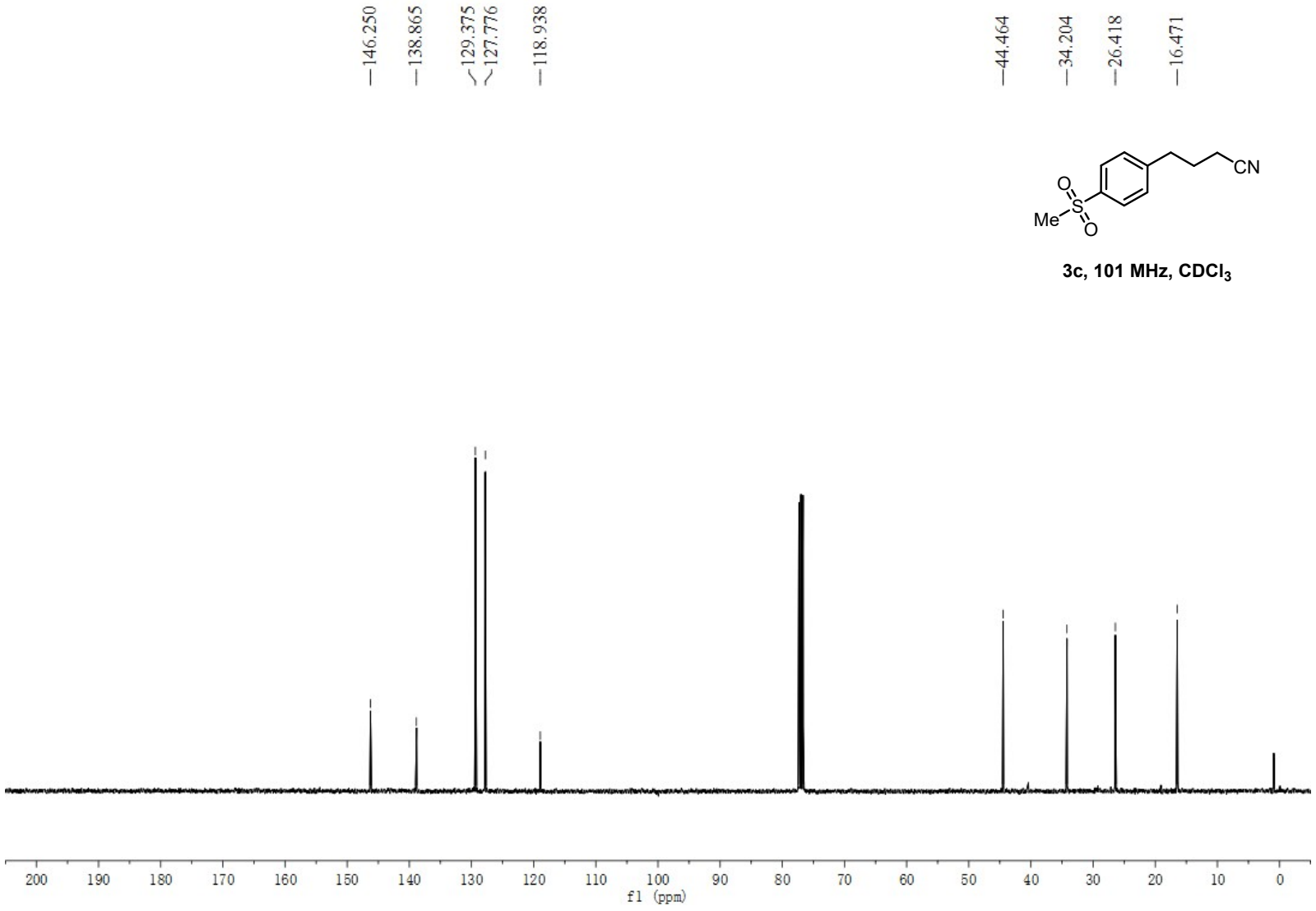
3b, 400 MHz, $CDCl_3$



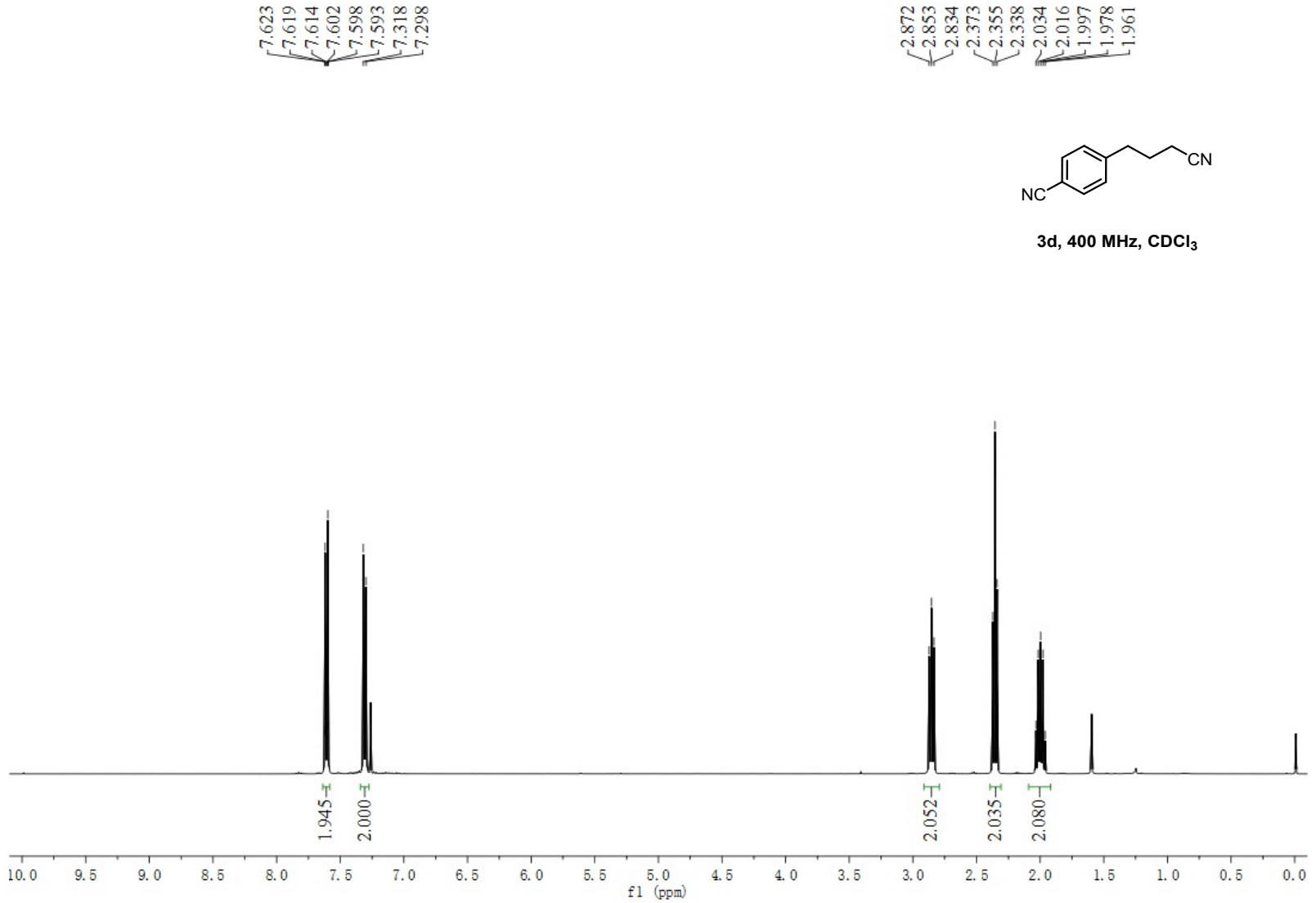
3b, 101 MHz, $CDCl_3$



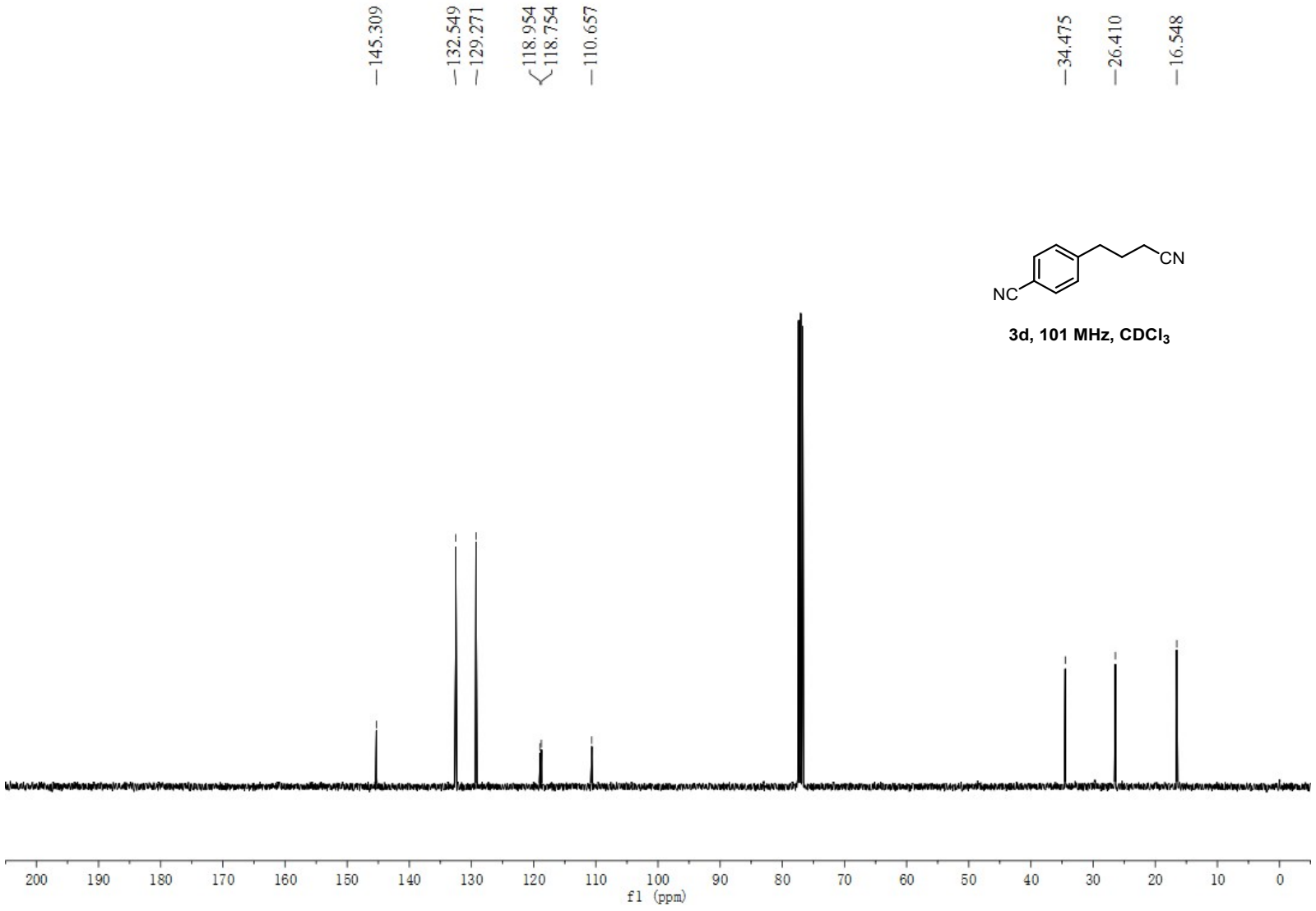
3c, 400 MHz, $CDCl_3$

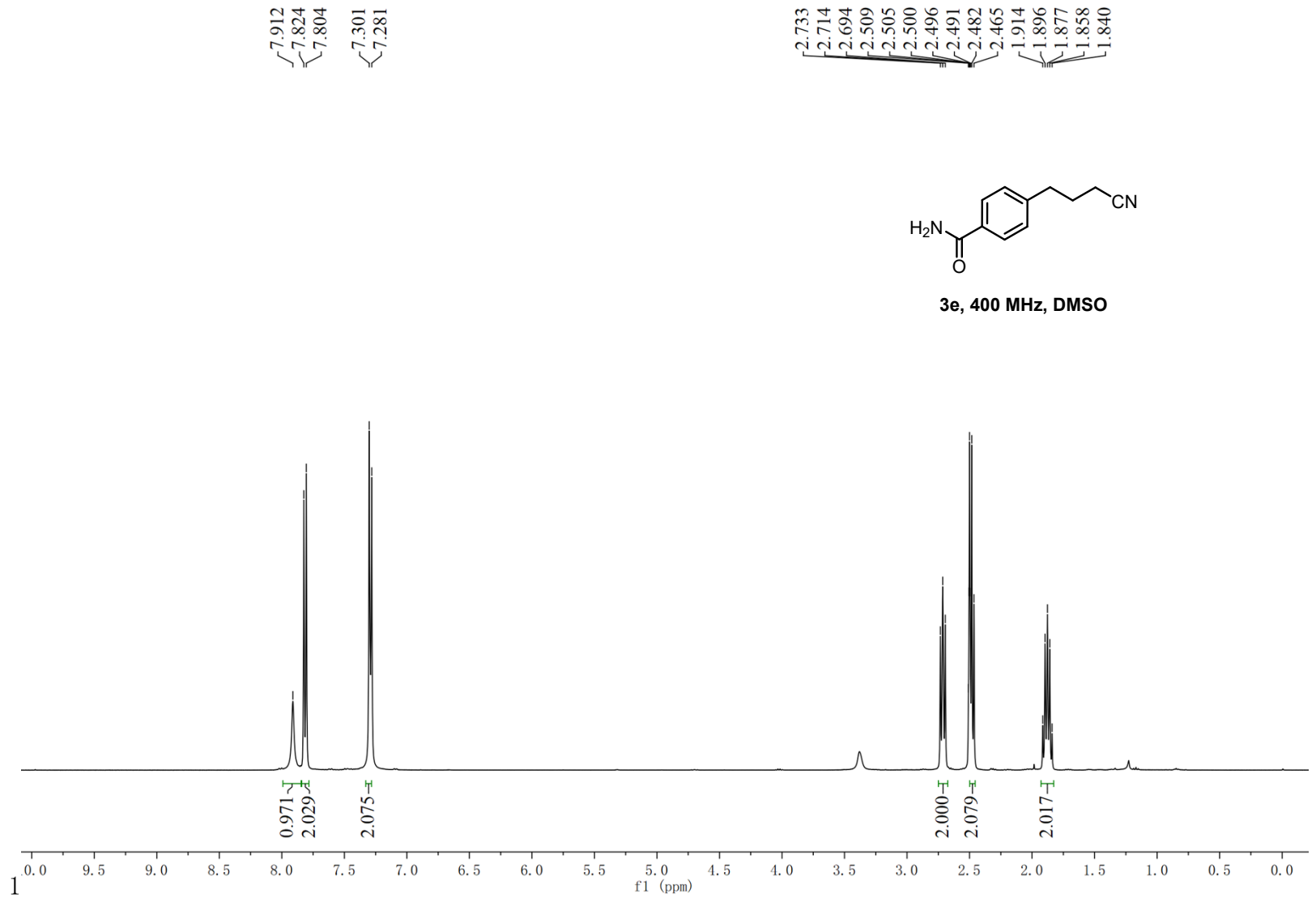


3c, 101 MHz, $CDCl_3$

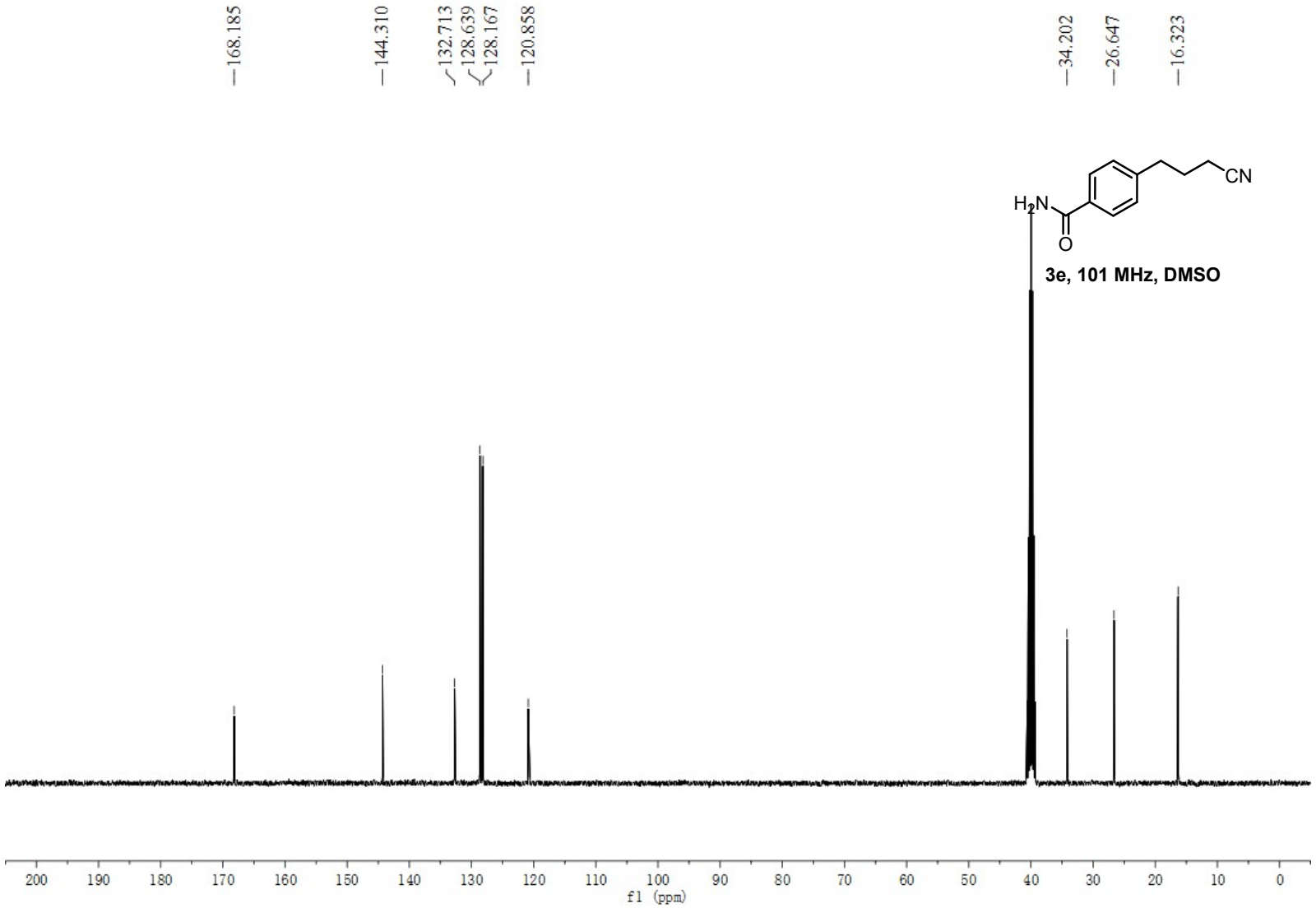


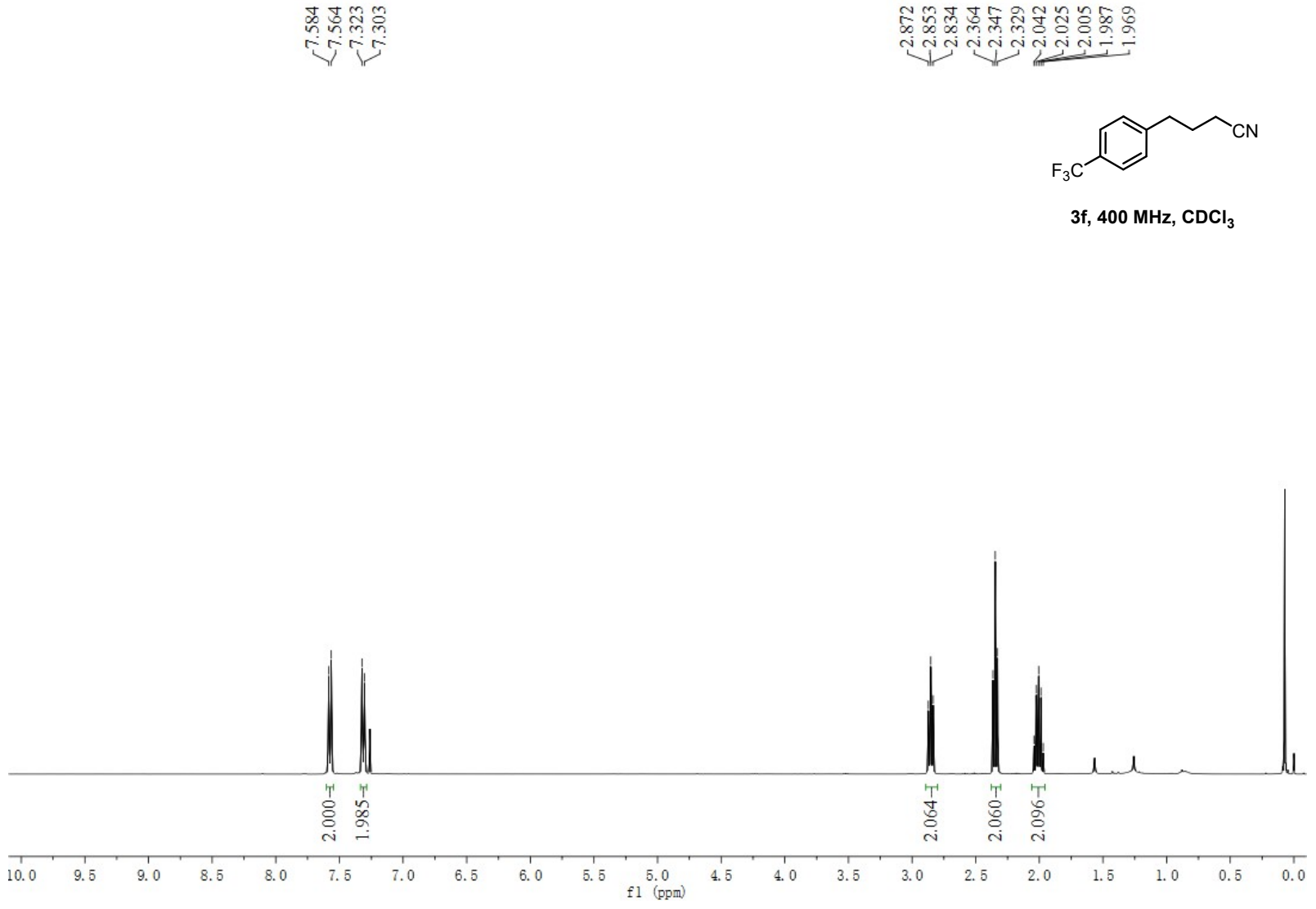
3d, 400 MHz, $CDCl_3$


145.309
132.549
129.271
118.954
118.754
110.657
34.475
26.410
16.548
NC
CN
3d, 101 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

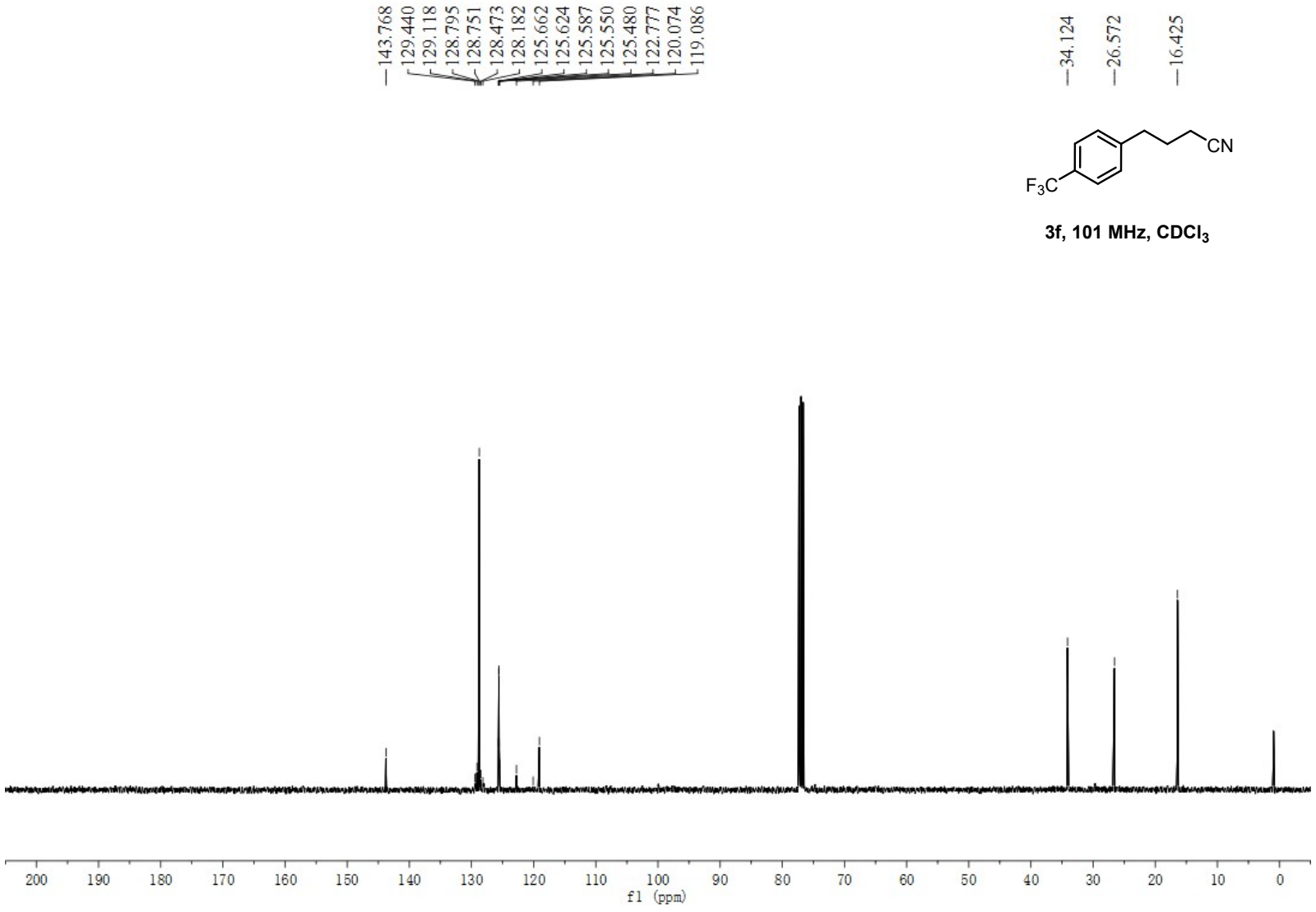


3e, 400 MHz, DMSO

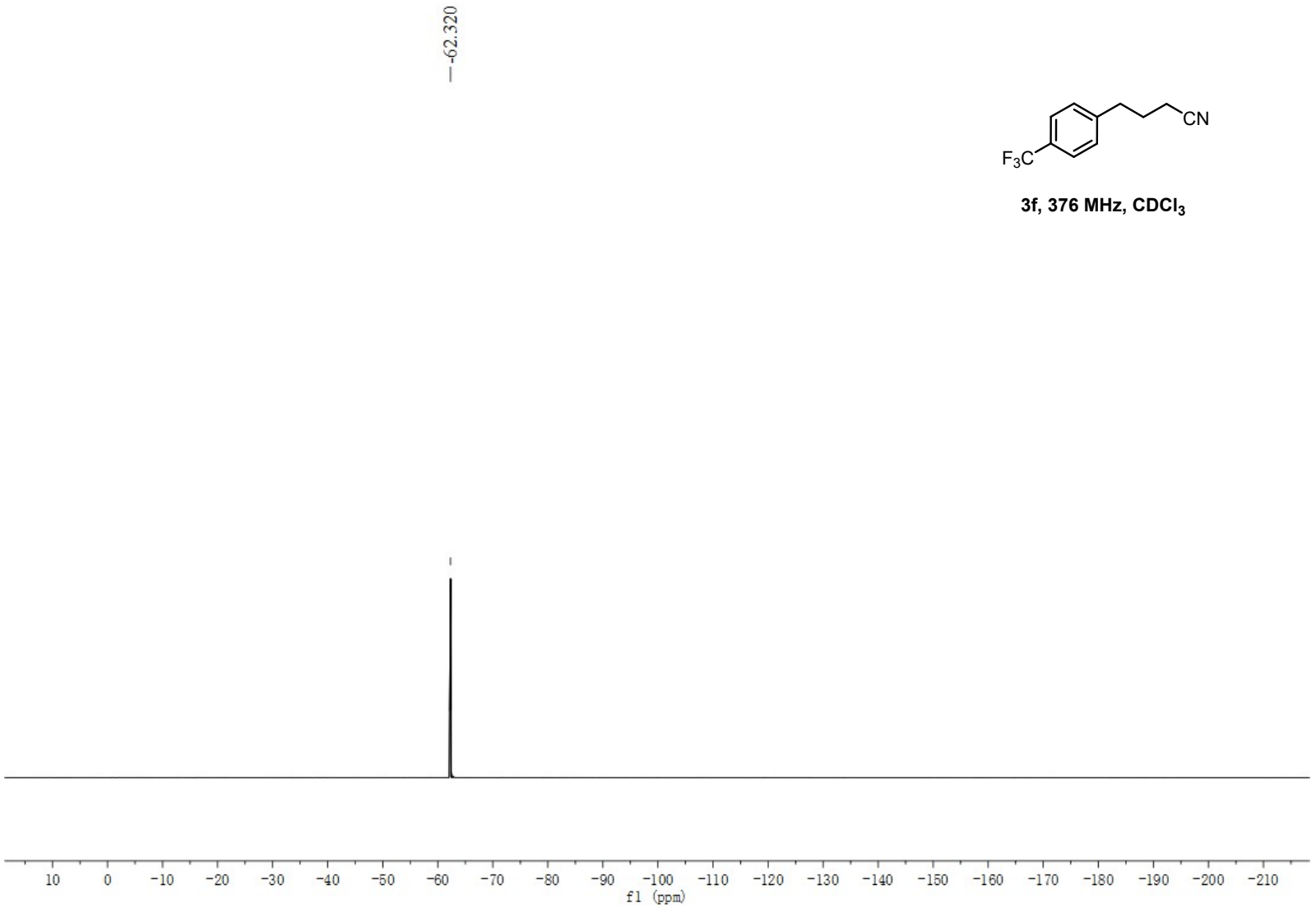

168.185
144.310
132.713
128.639
128.167
120.858
34.202
26.647
16.323
CN
H2N
O
3e, 101 MHz, DMSO
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



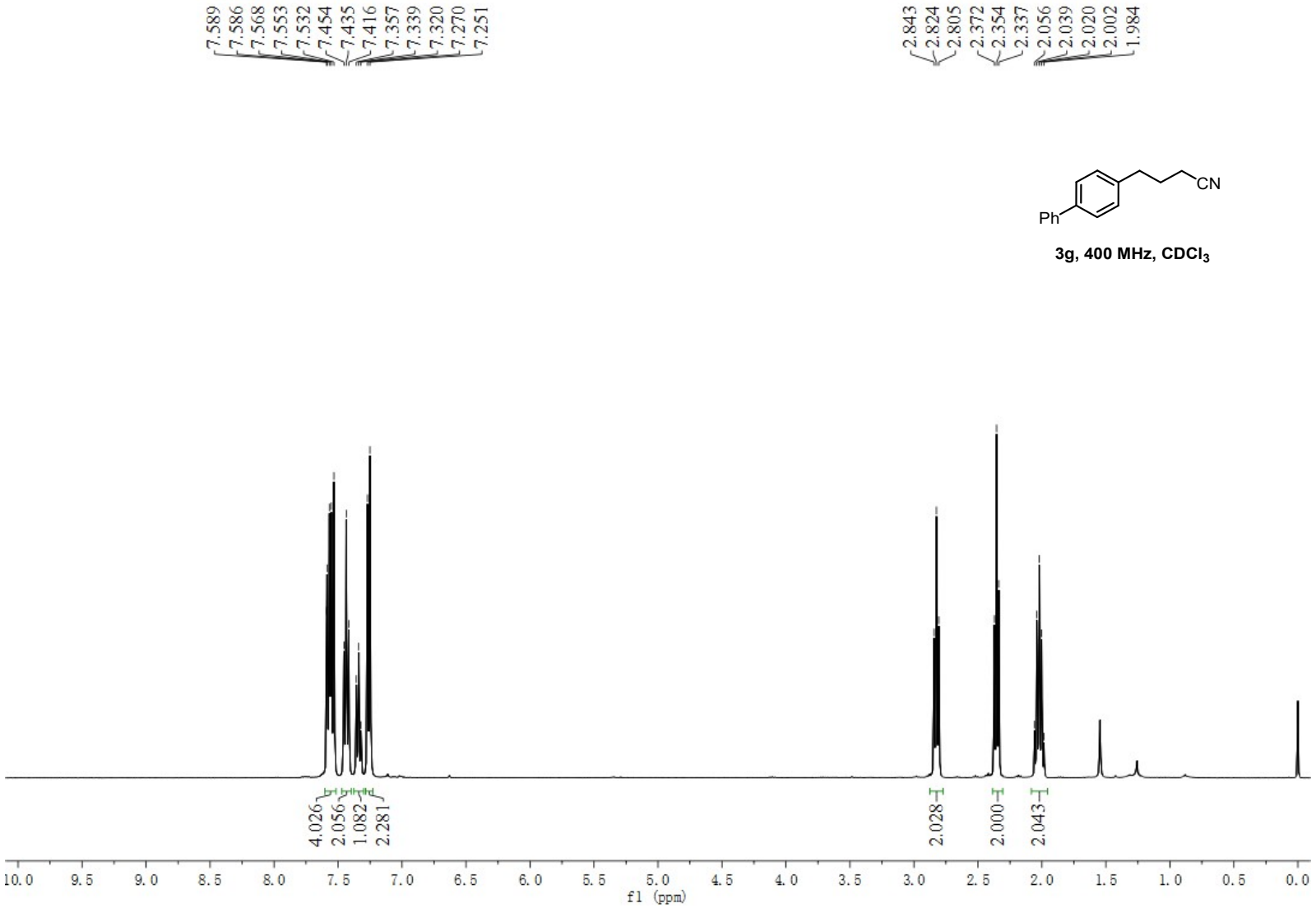
3f, 400 MHz, $CDCl_3$



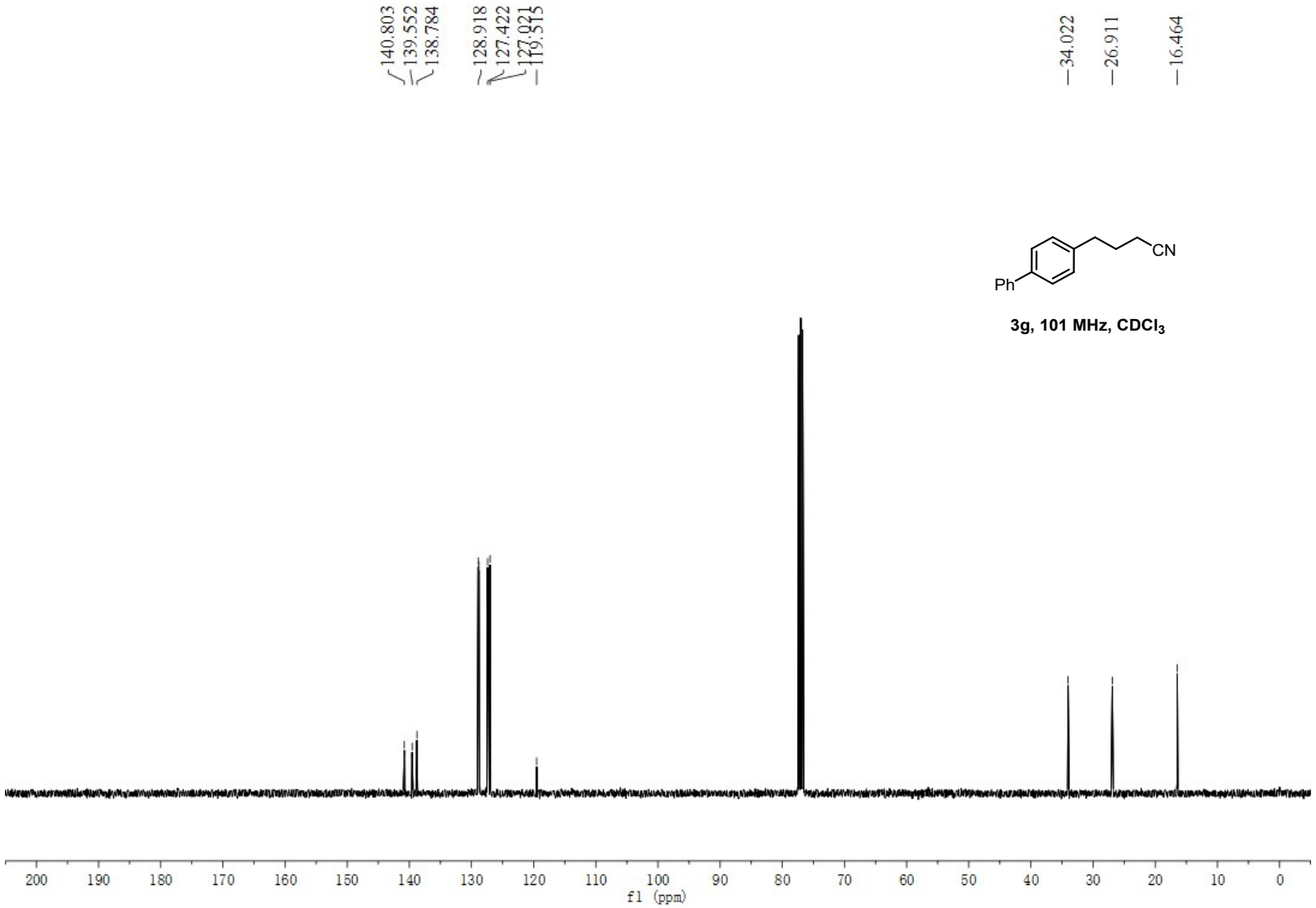
3f, 101 MHz, $CDCl_3$

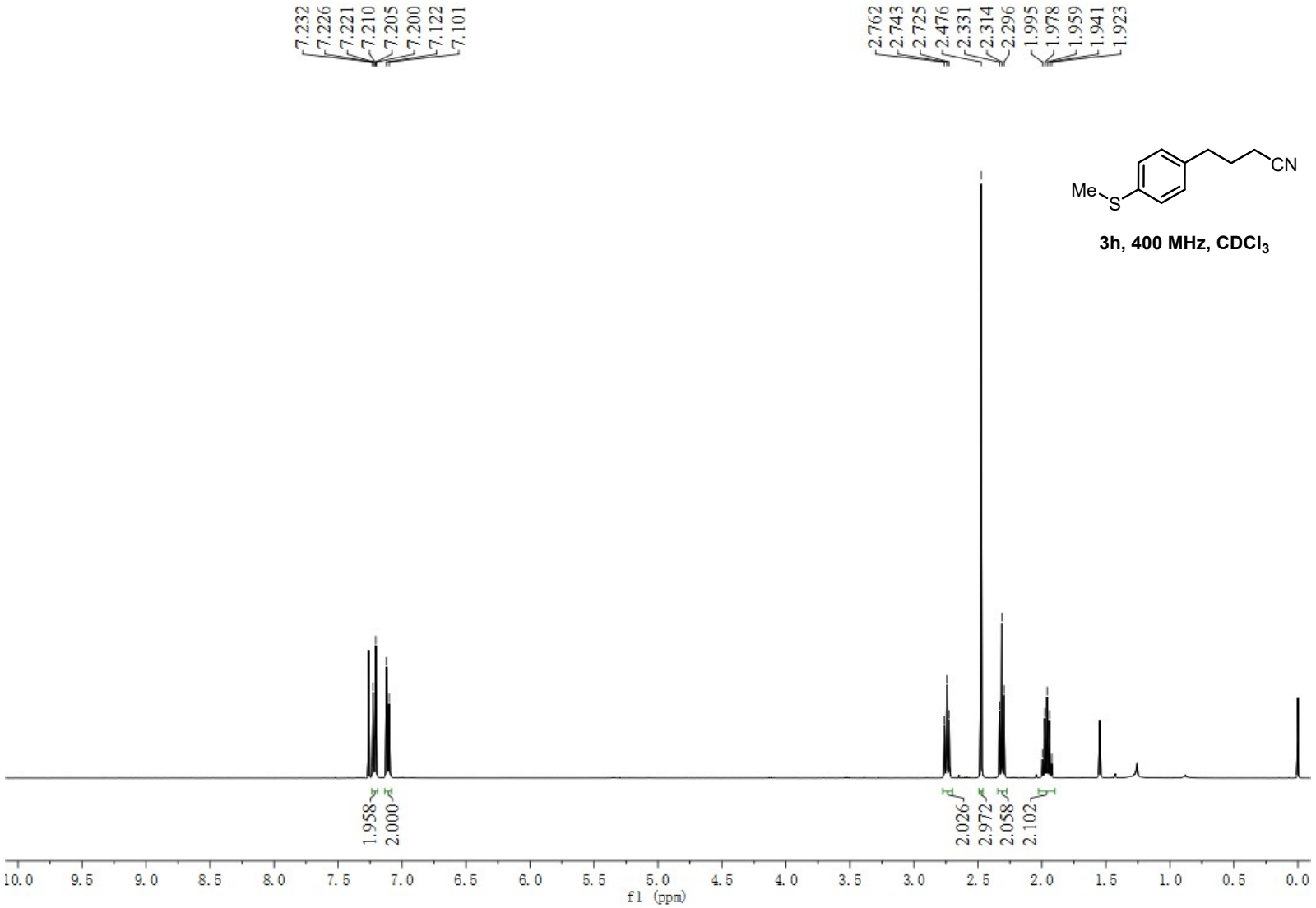


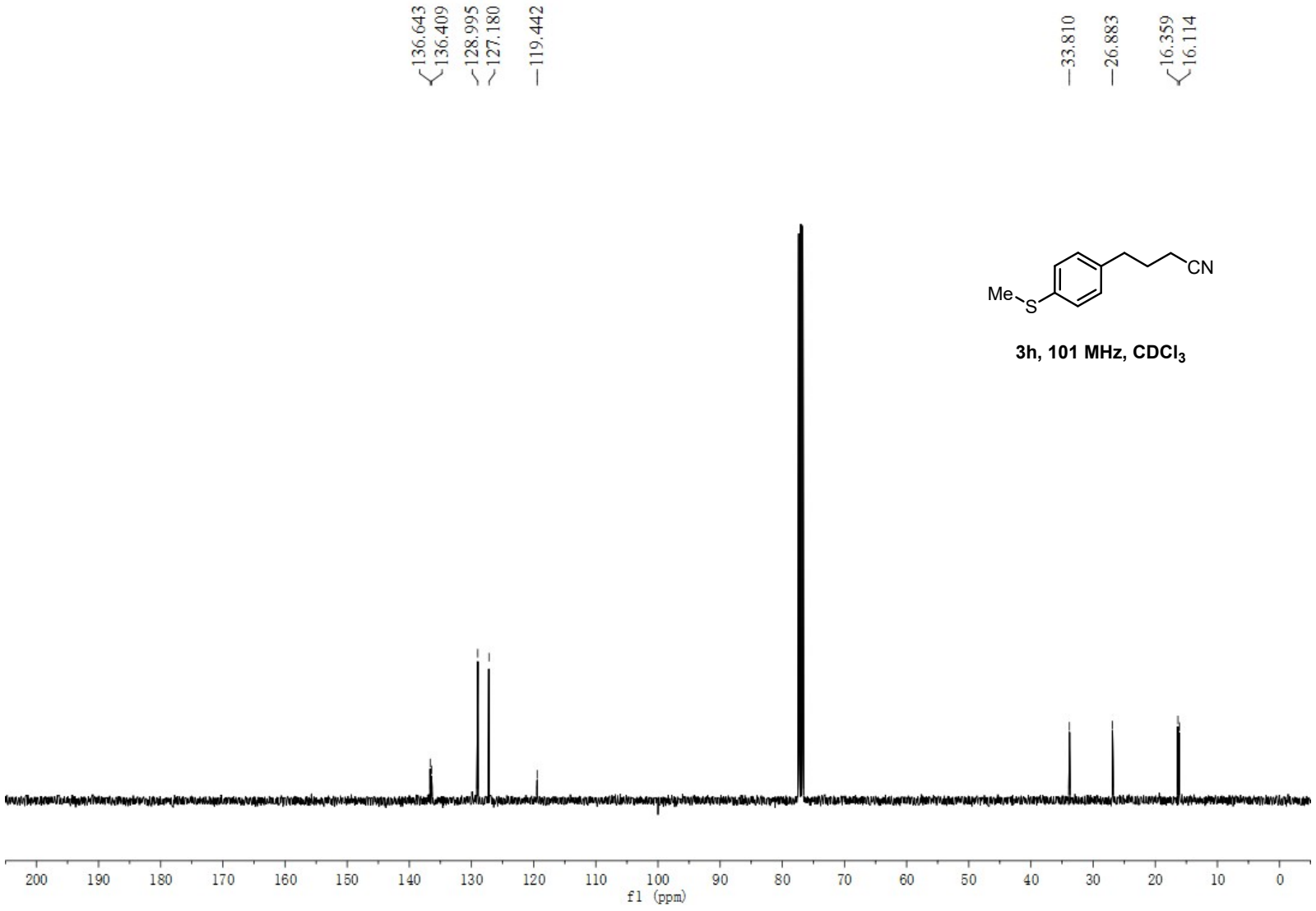
3f, 376 MHz, $CDCl_3$



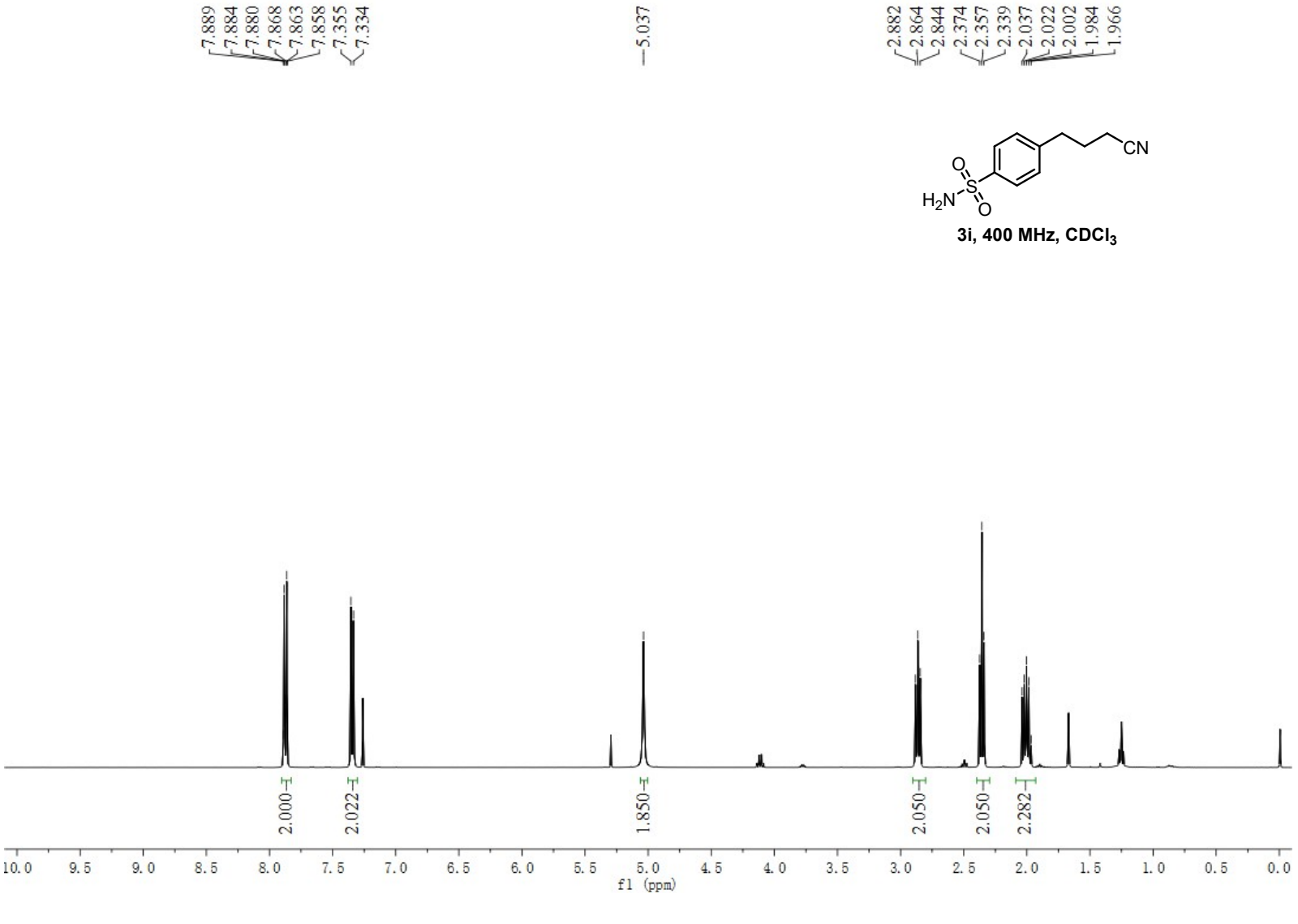
3g, 400 MHz, $CDCl_3$


140.803
139.552
138.784
128.918
127.422
127.021
119.515
34.022
26.911
16.464
CN
Ph
3g, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

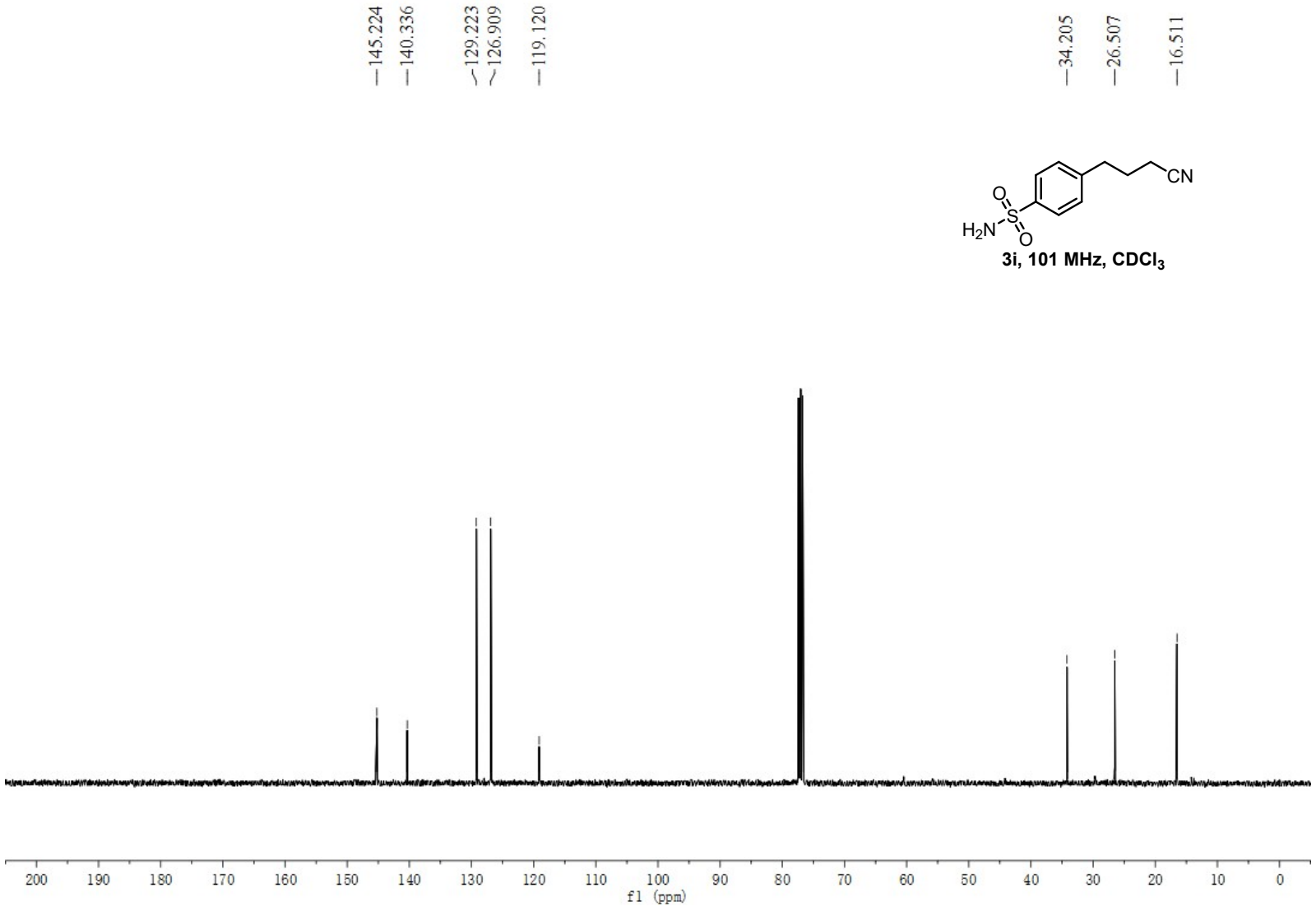

7.232
7.226
7.221
7.210
7.205
7.200
7.122
7.101
2.762
2.743
2.725
2.476
2.331
2.314
2.296
1.995
1.978
1.959
1.941
1.923
Me
S
CN
3h, 400 MHz, CDCl3
1.958
2.000
2.026
2.972
2.058
2.102
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



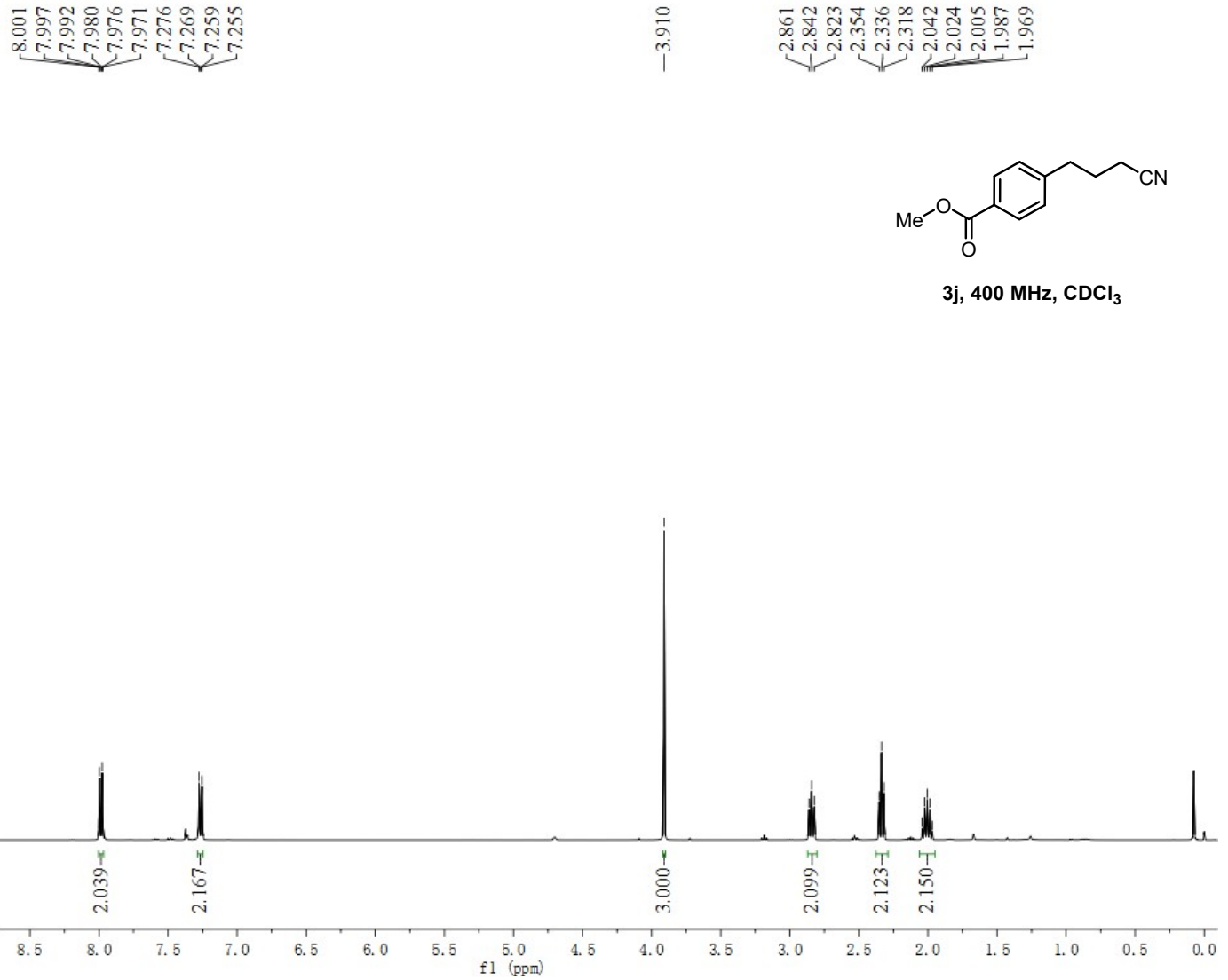
3h, 101 MHz, $CDCl_3$



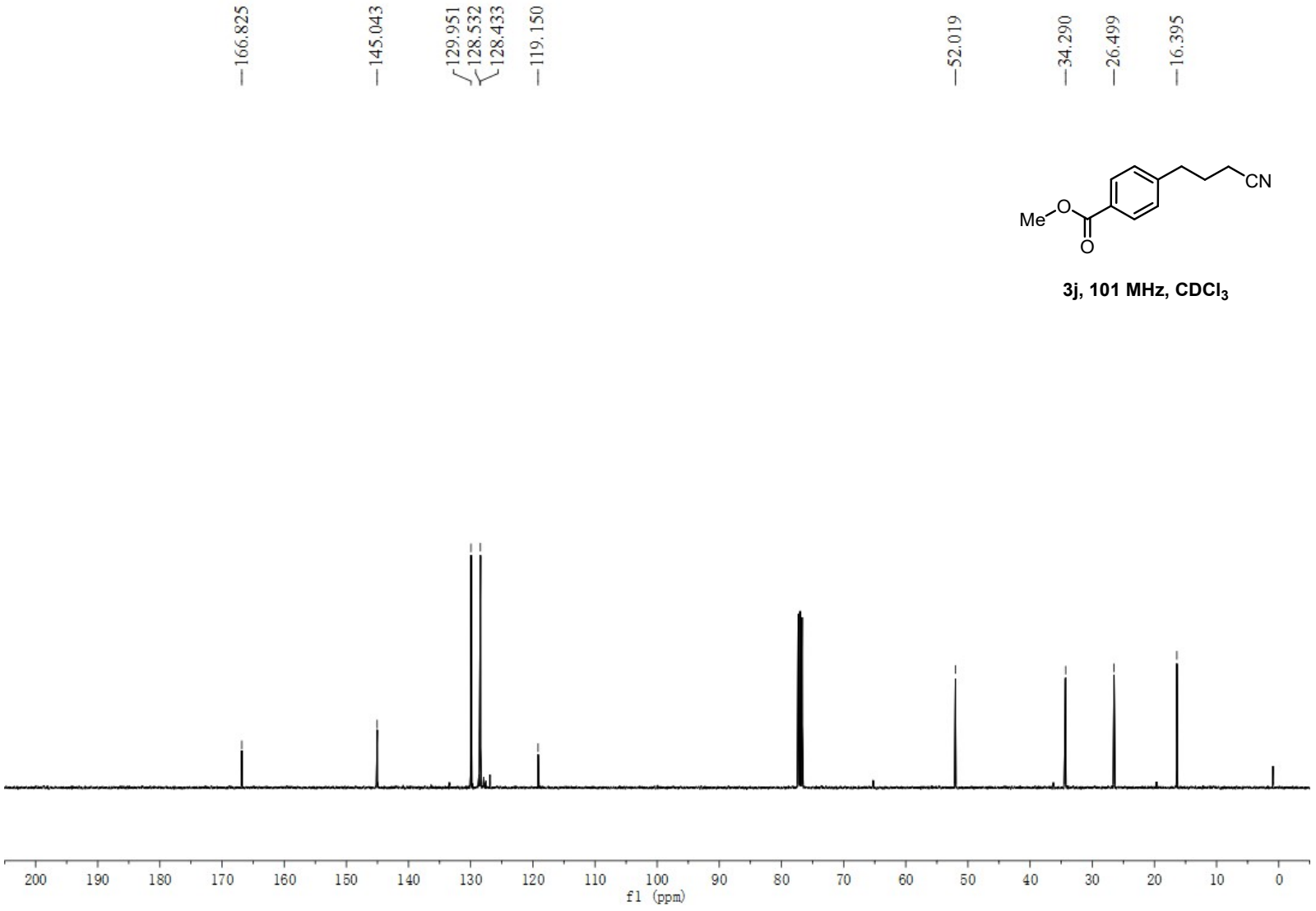
3i, 400 MHz, $CDCl_3$



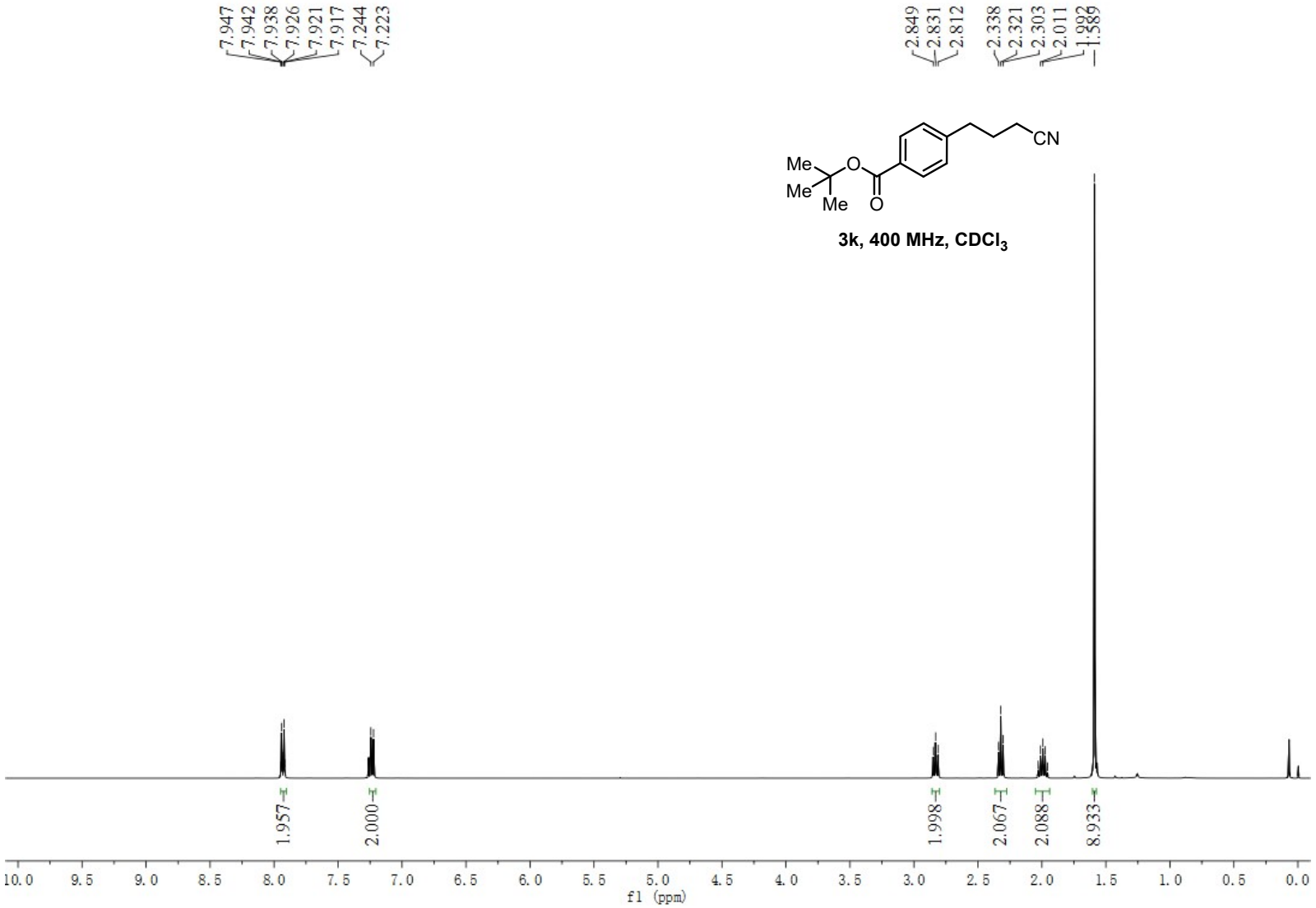
3i, 101 MHz, $CDCl_3$



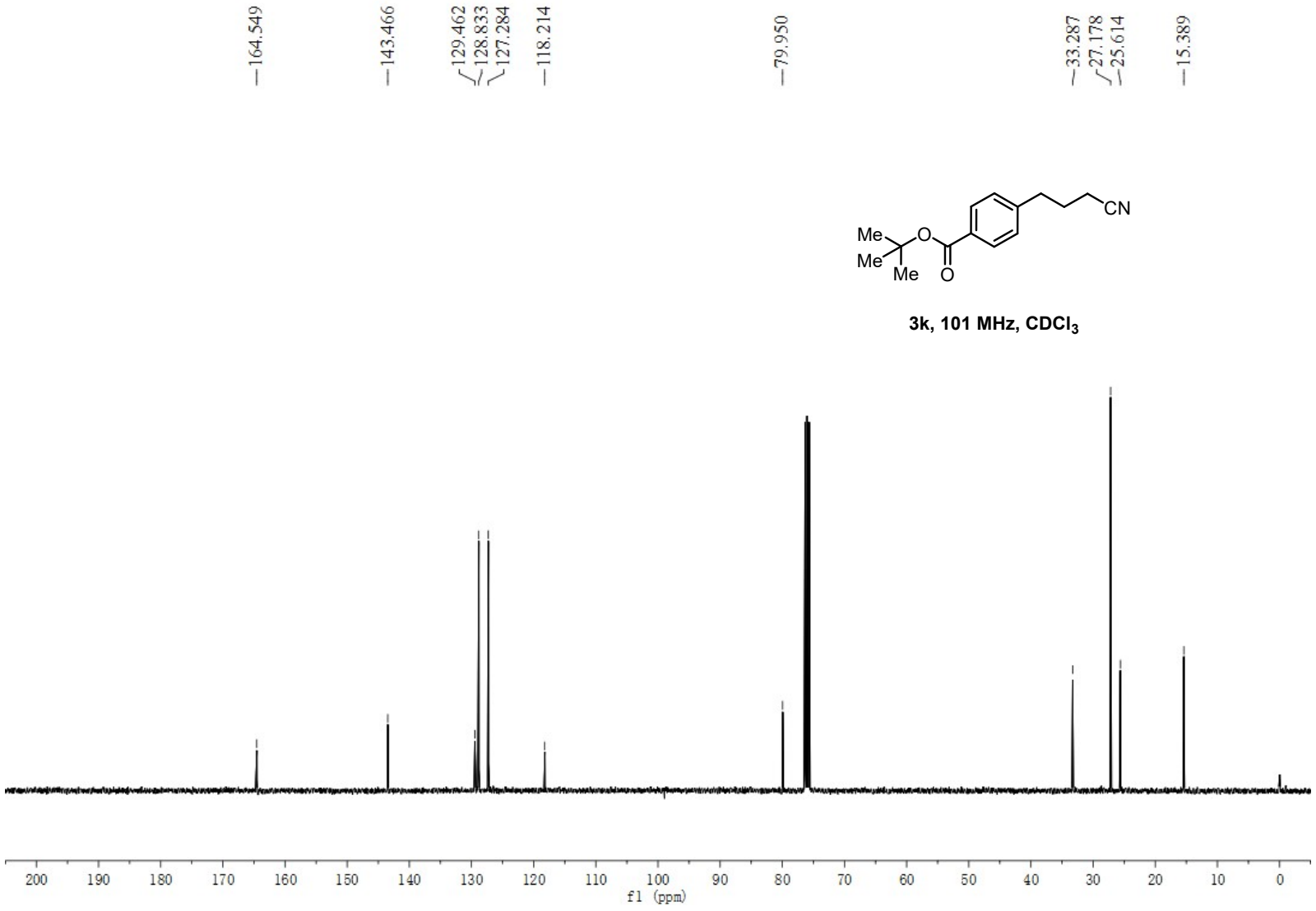
3j, 400 MHz, $CDCl_3$



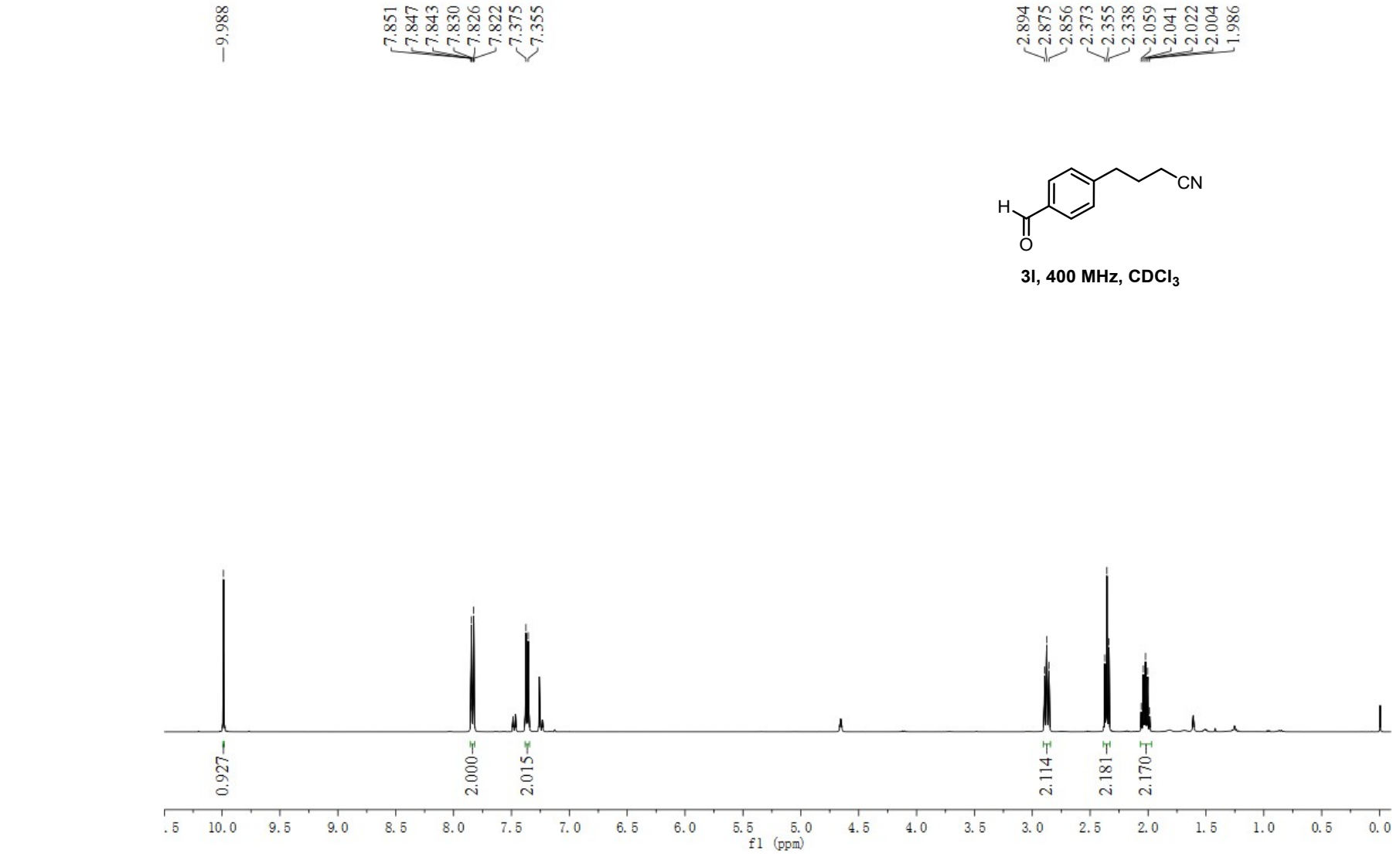
3j, 101 MHz, $CDCl_3$



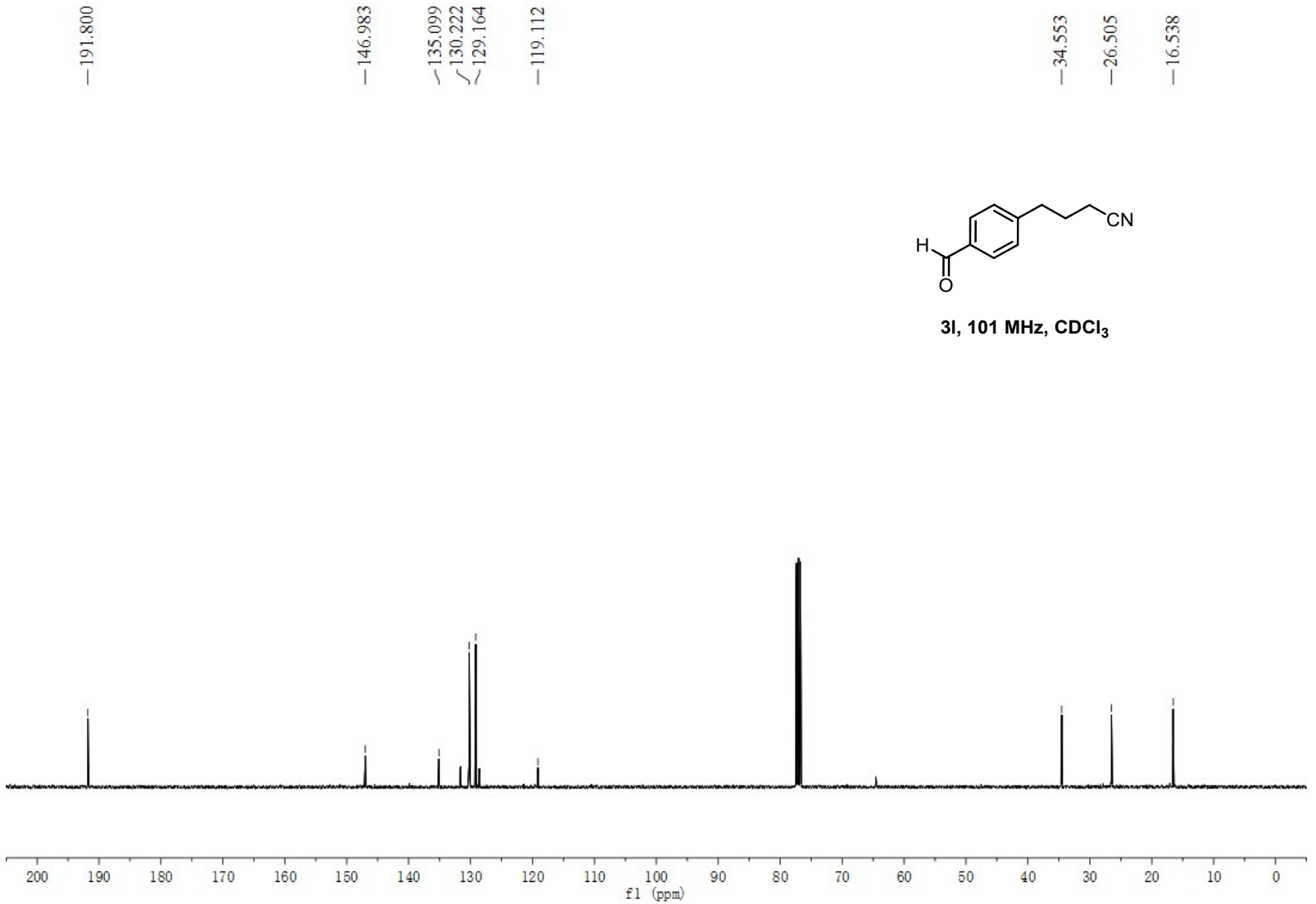
3k, 400 MHz, $CDCl_3$



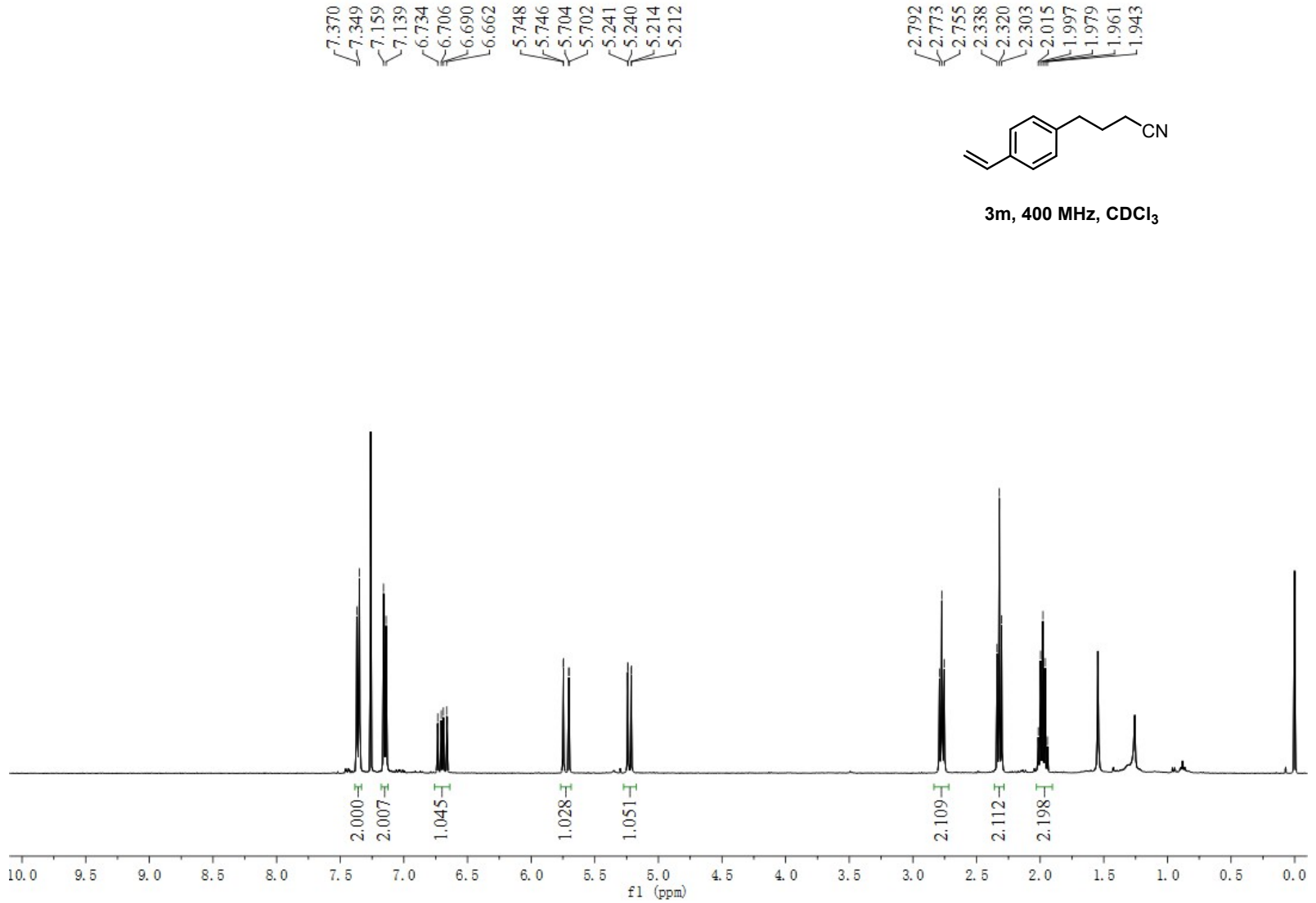
3k, 101 MHz, $CDCl_3$



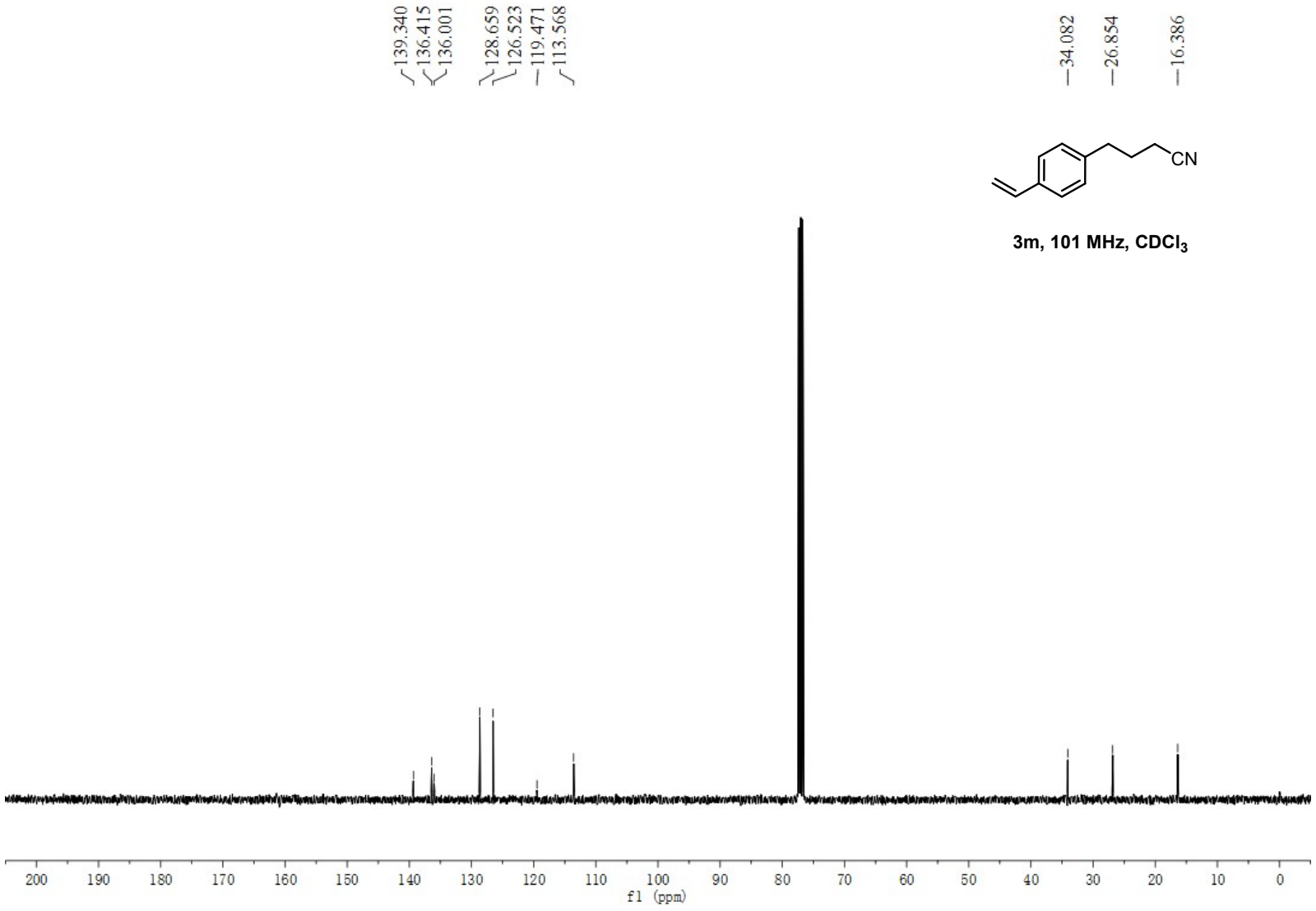
3l, 400 MHz, $CDCl_3$



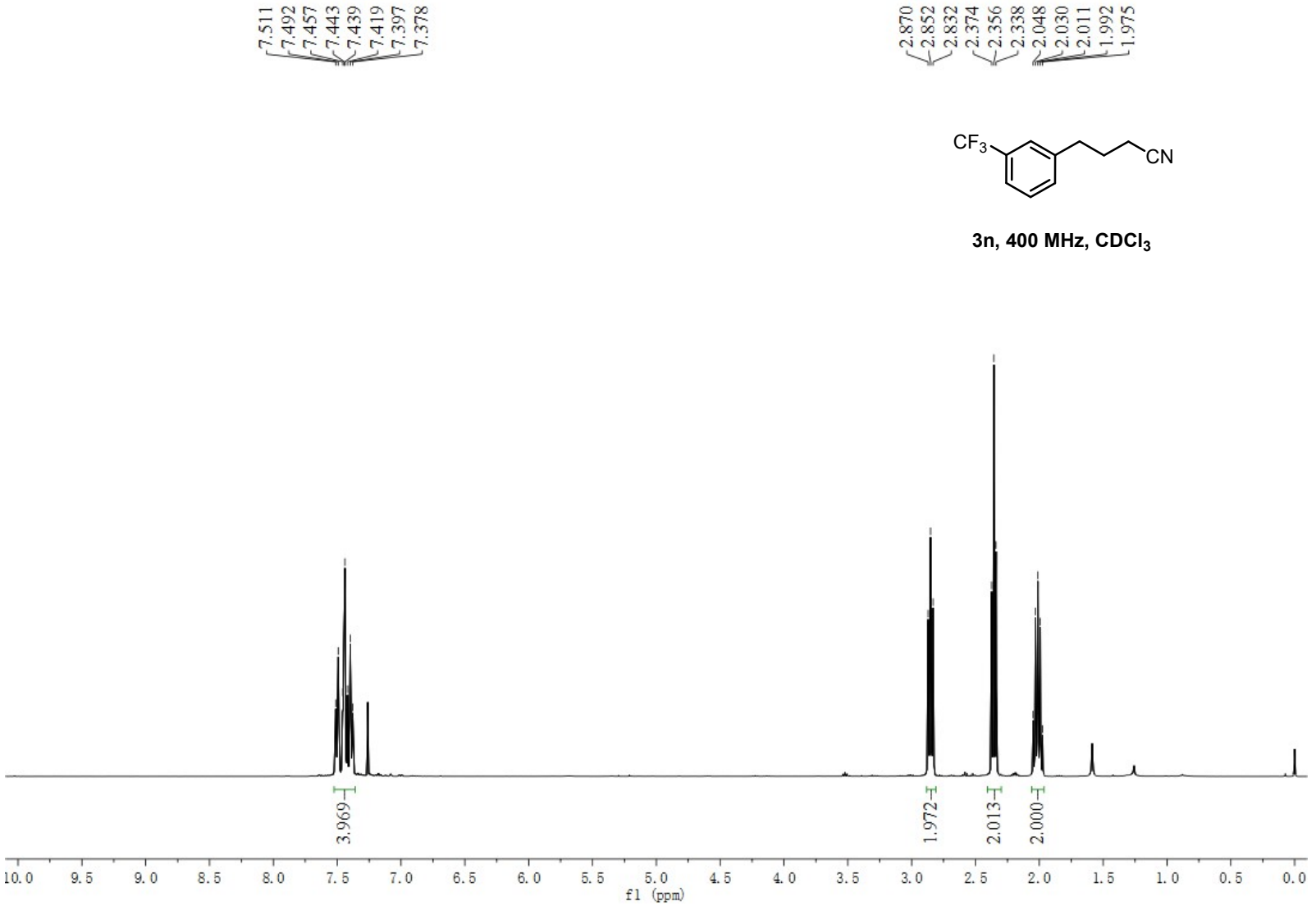
3l, 101 MHz, $CDCl_3$



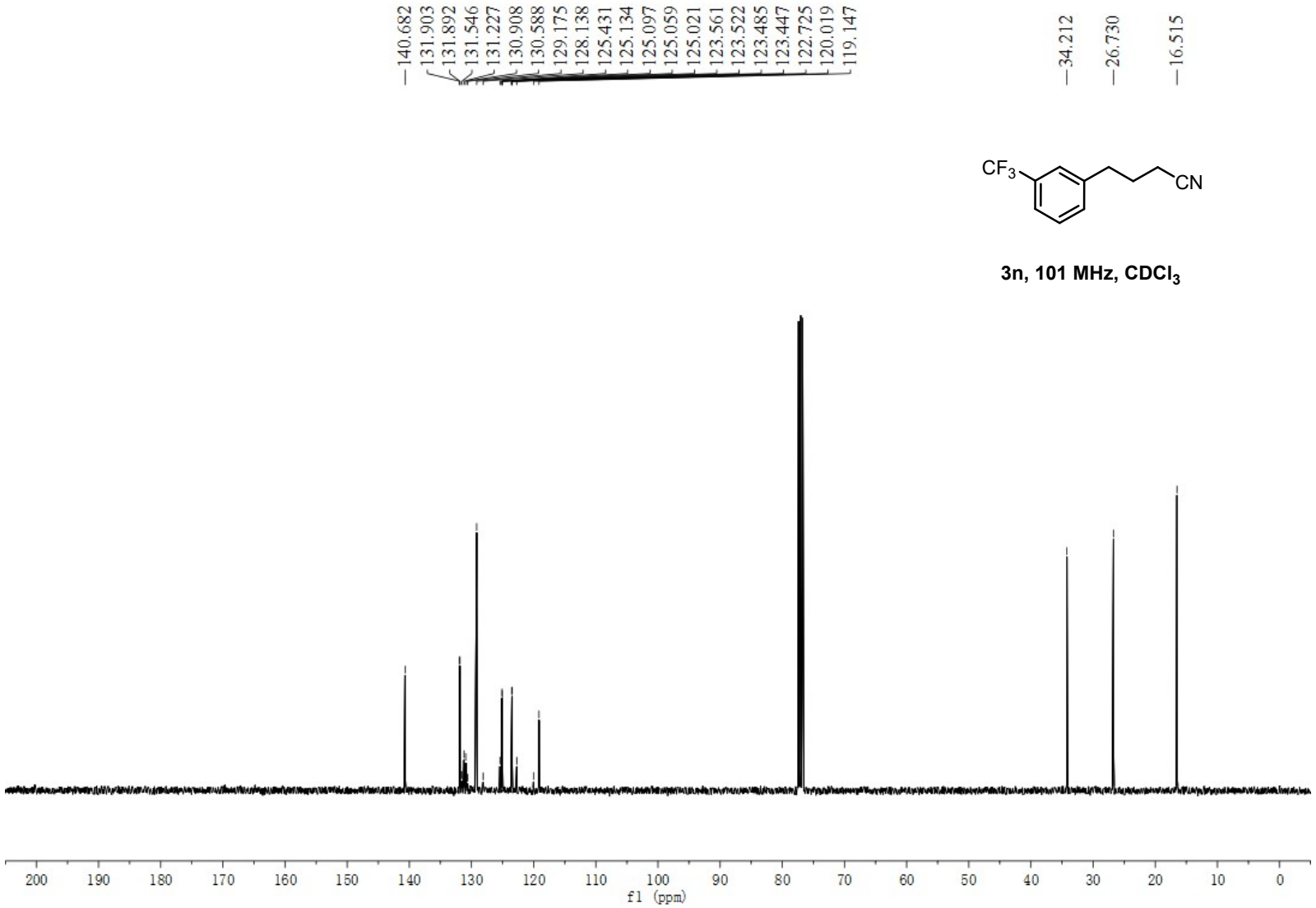
3m, 400 MHz, $CDCl_3$



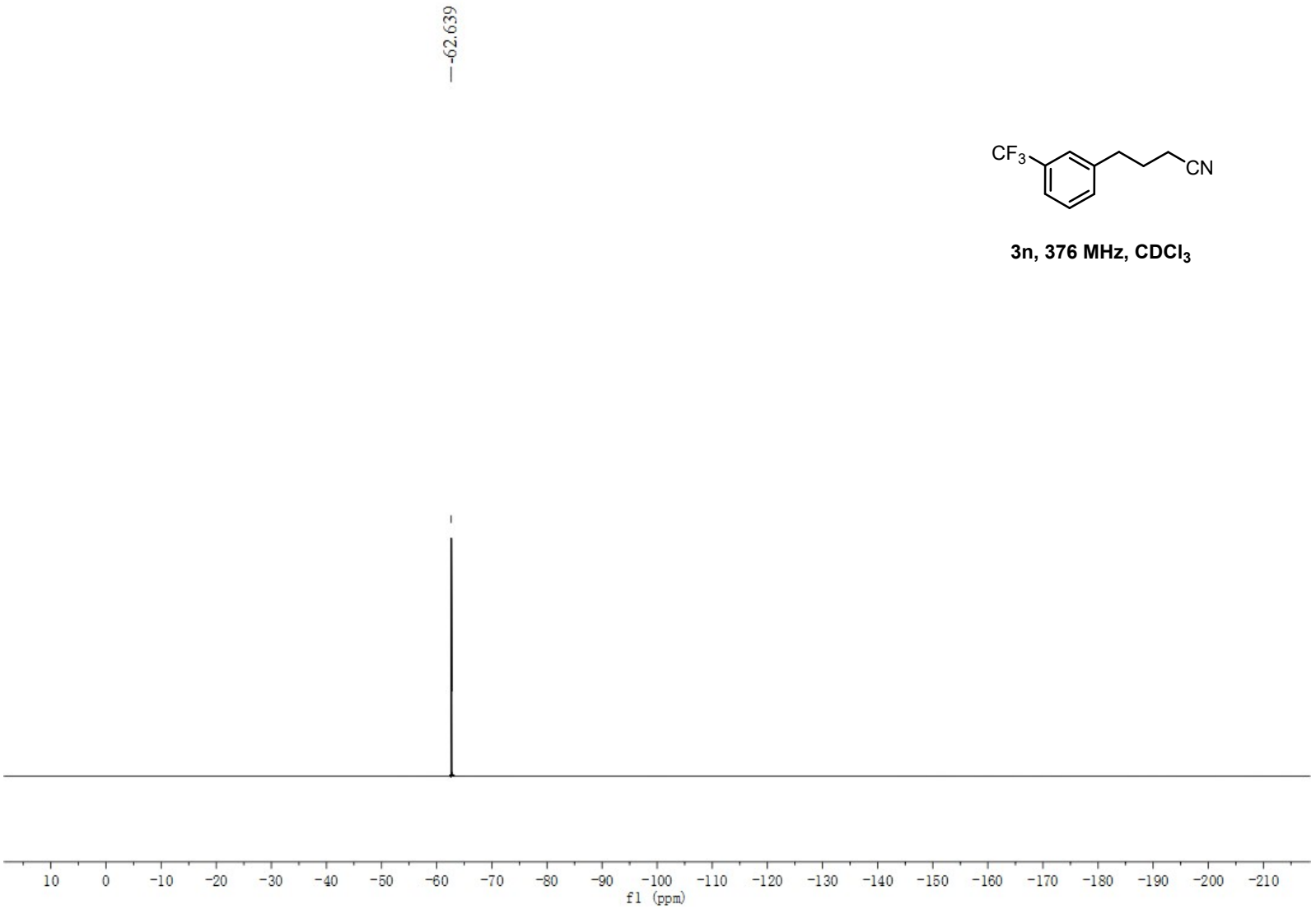
3m, 101 MHz, $CDCl_3$



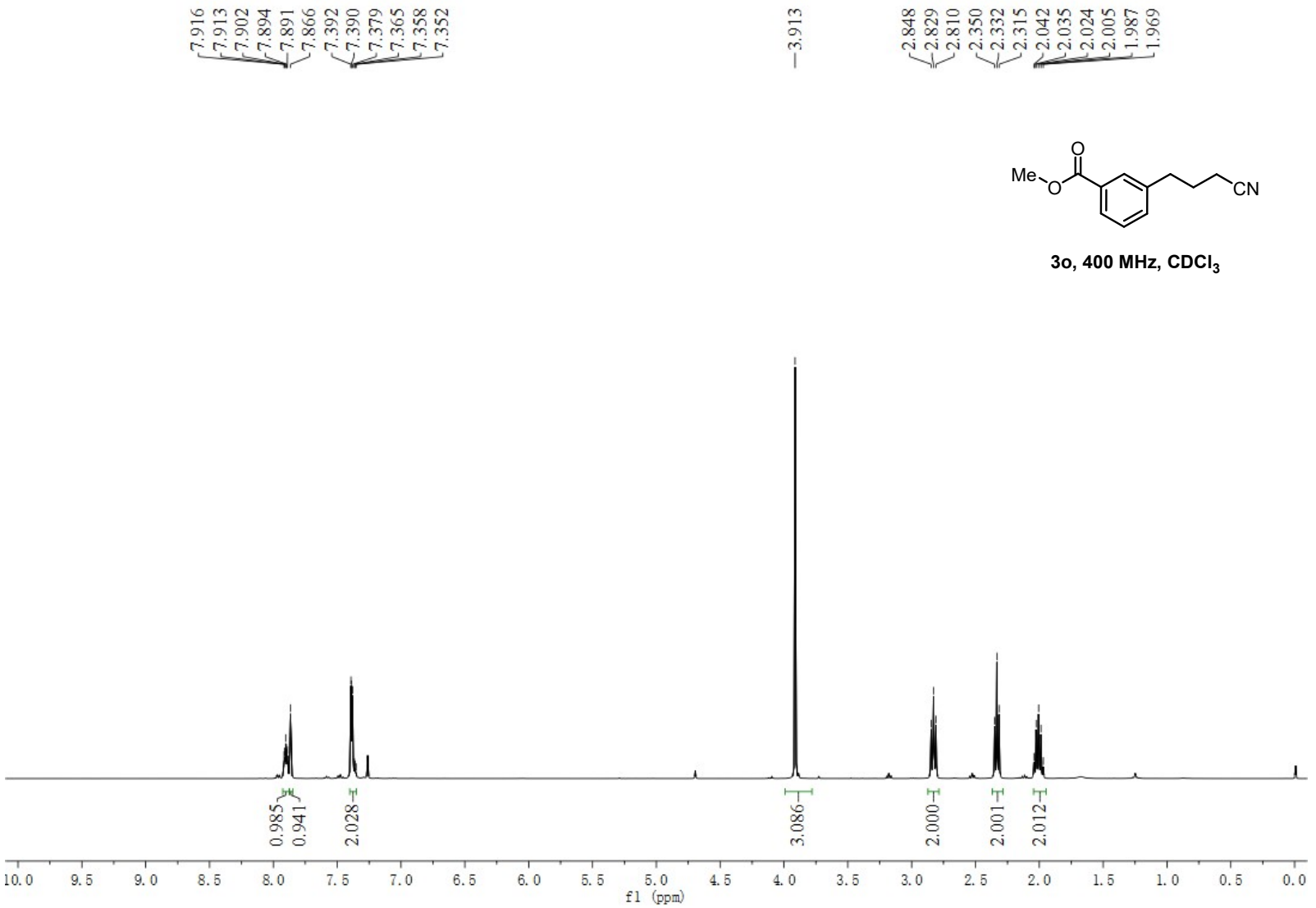
3n, 400 MHz, $CDCl_3$



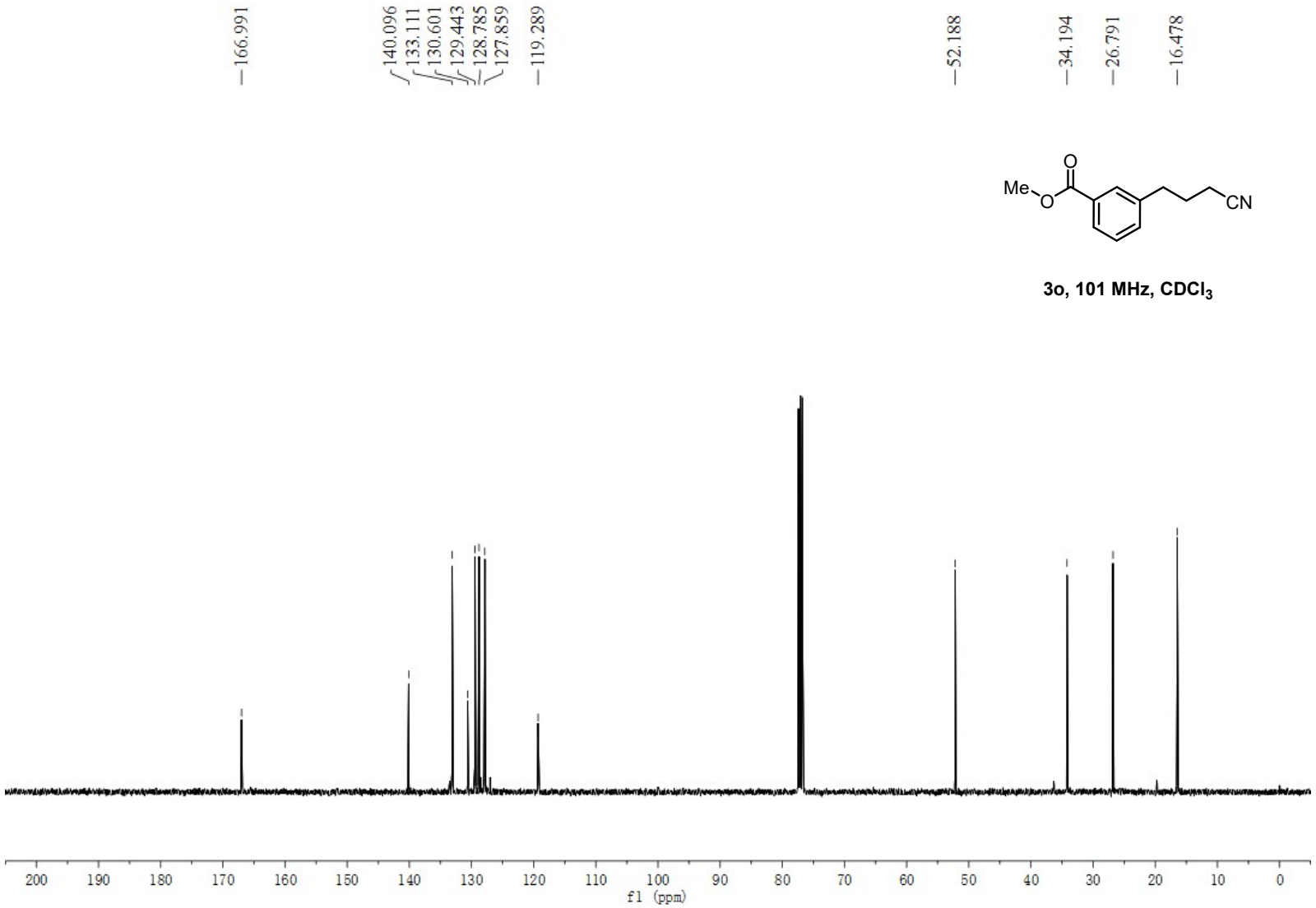
3n, 101 MHz, $CDCl_3$



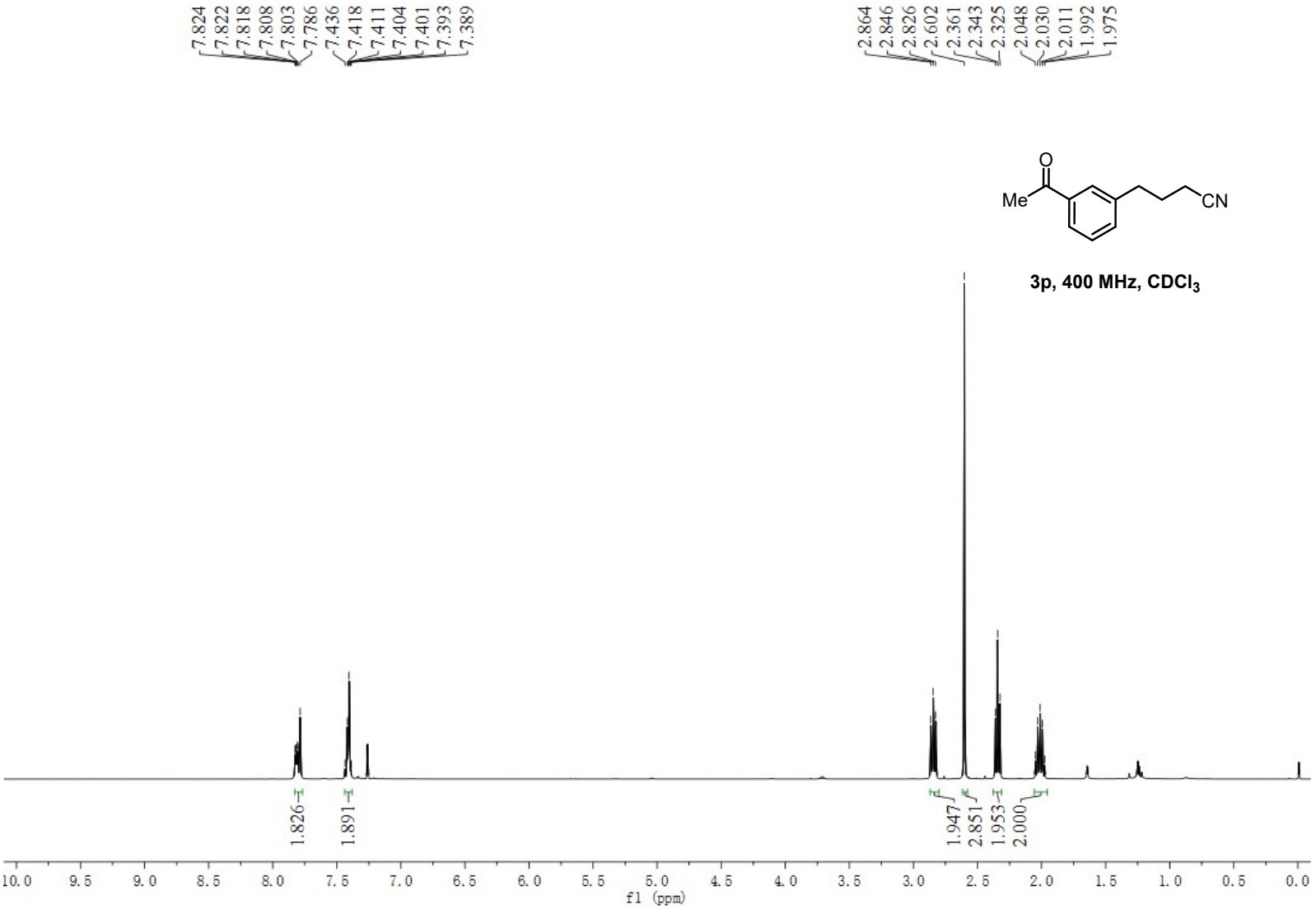
3n, 376 MHz, $CDCl_3$

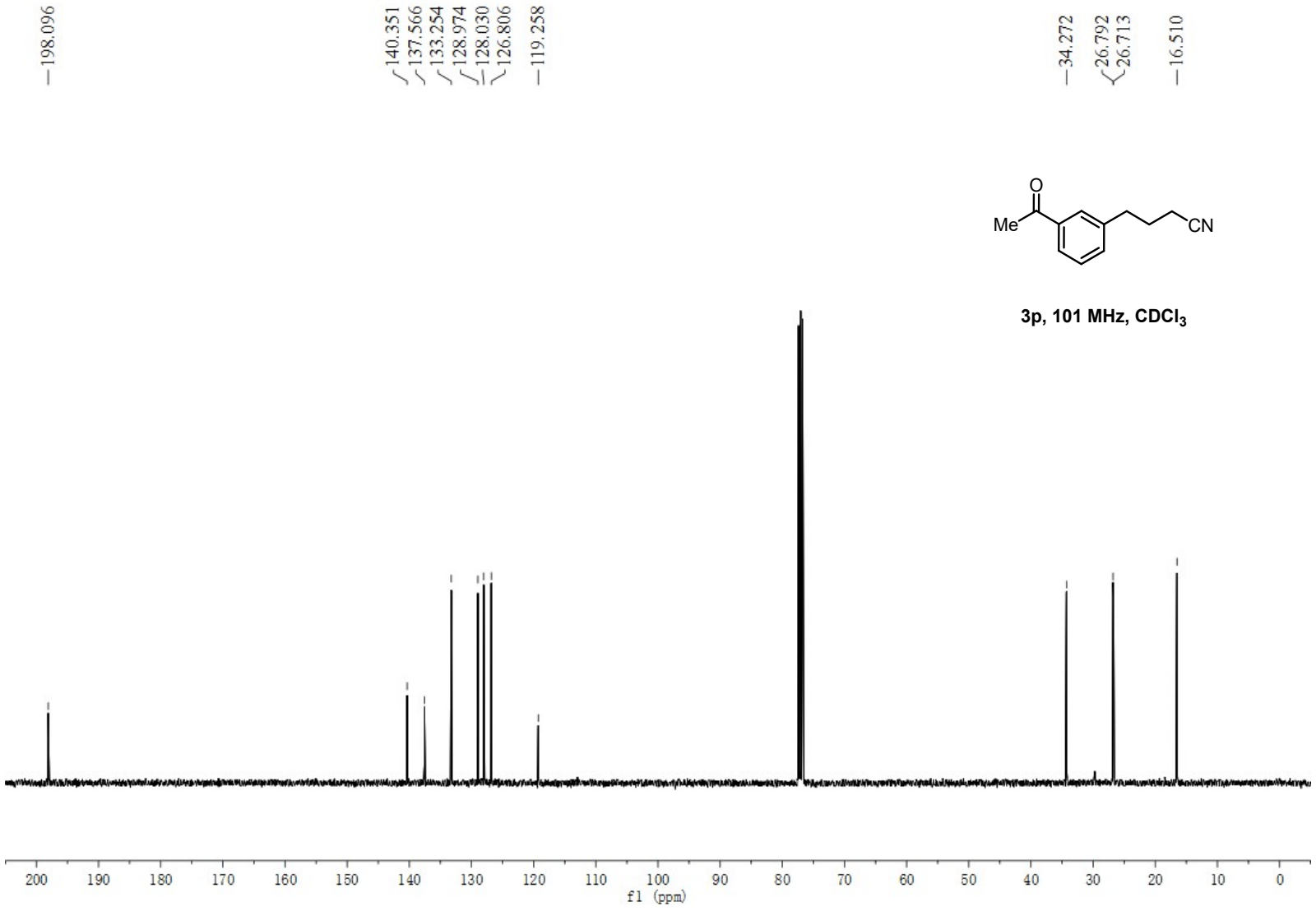


3o, 400 MHz, $CDCl_3$



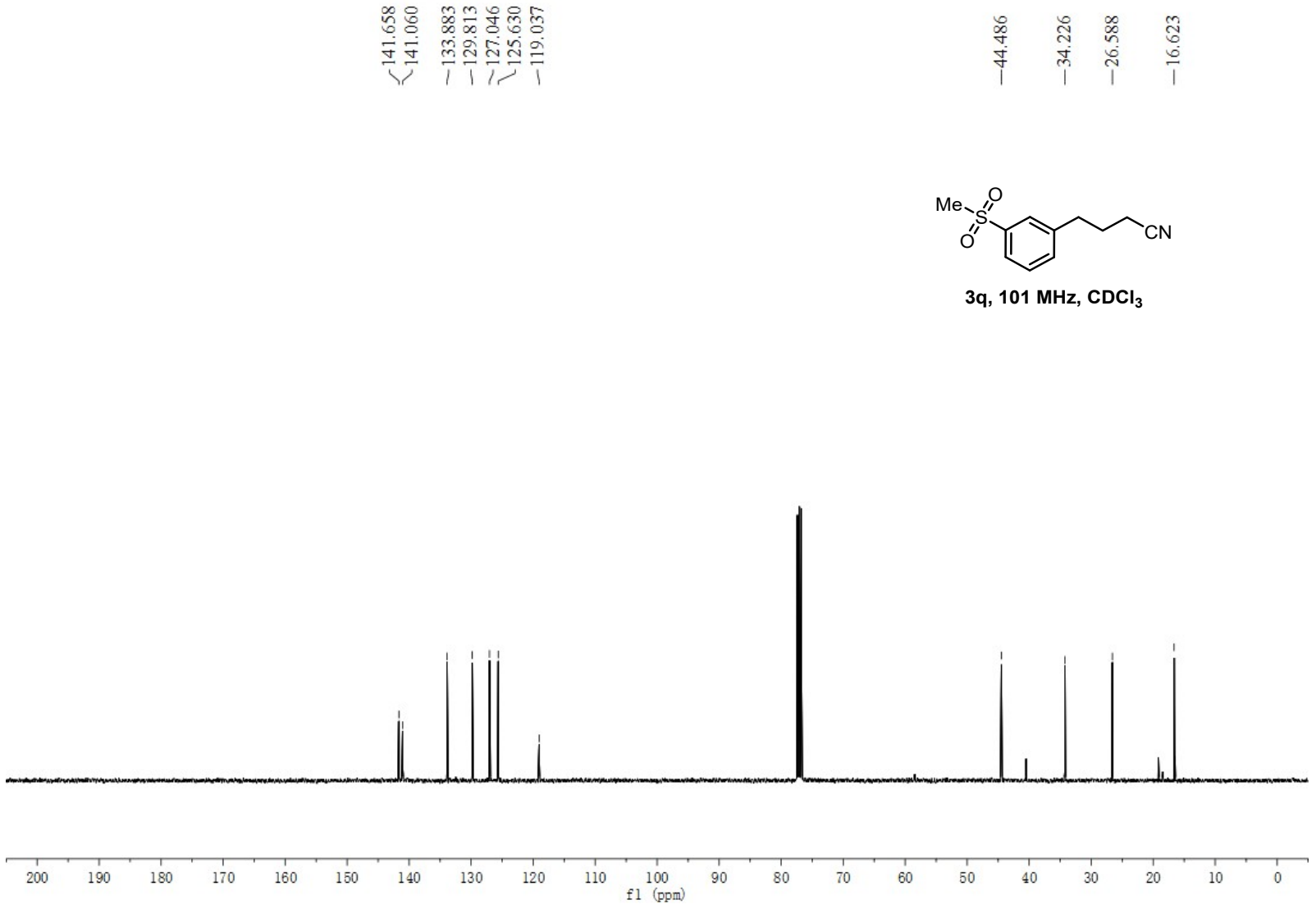
3o, 101 MHz, $CDCl_3$


7.824
7.822
7.818
7.808
7.803
7.786
7.436
7.418
7.411
7.404
7.401
7.393
7.389
2.864
2.846
2.826
2.602
2.361
2.343
2.325
2.048
2.030
2.011
1.992
1.975
O
Me
CN
3p, 400 MHz, CDCl3
1.826
1.891
1.947
2.851
1.953
2.000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

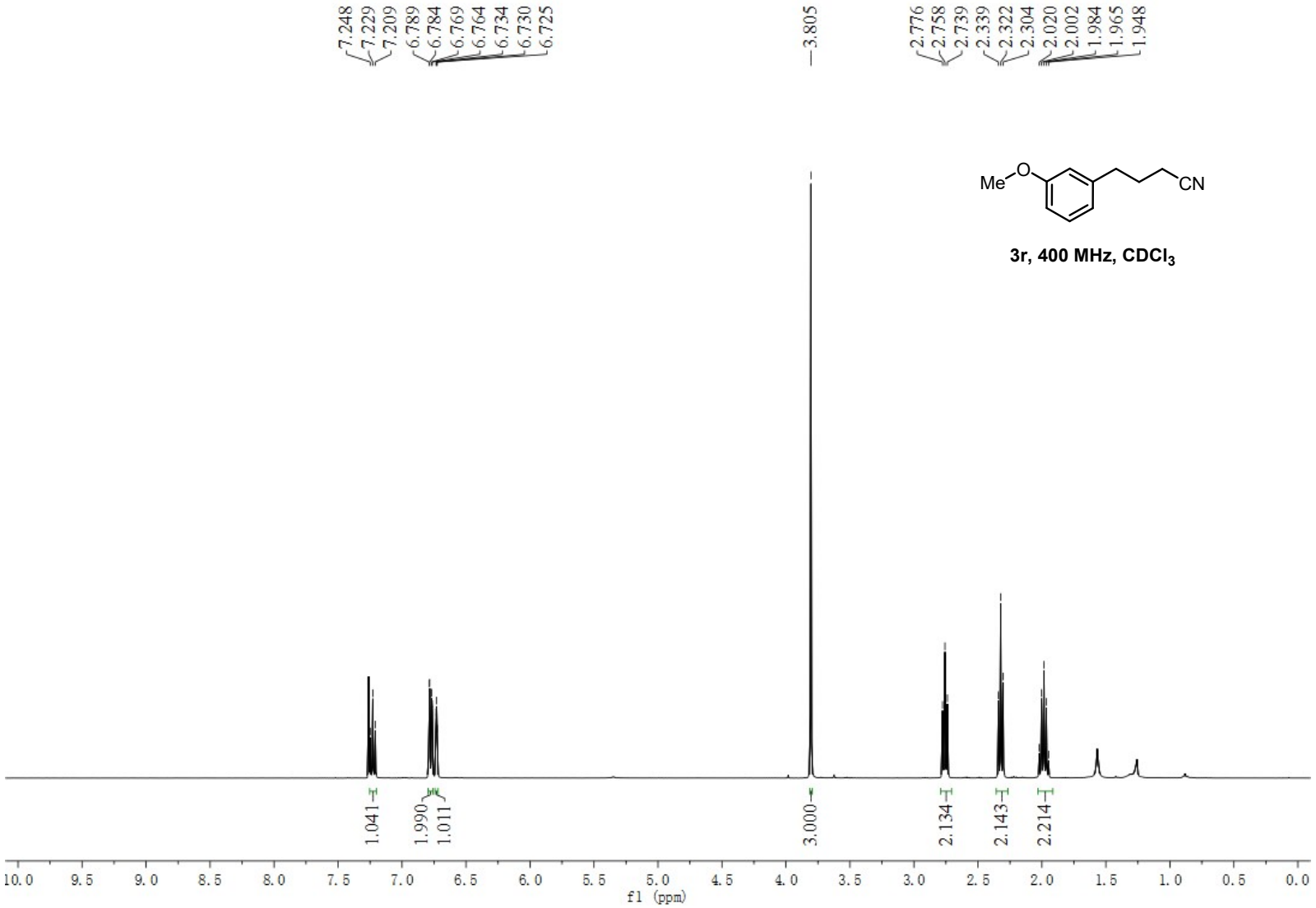


3p, 101 MHz, $CDCl_3$

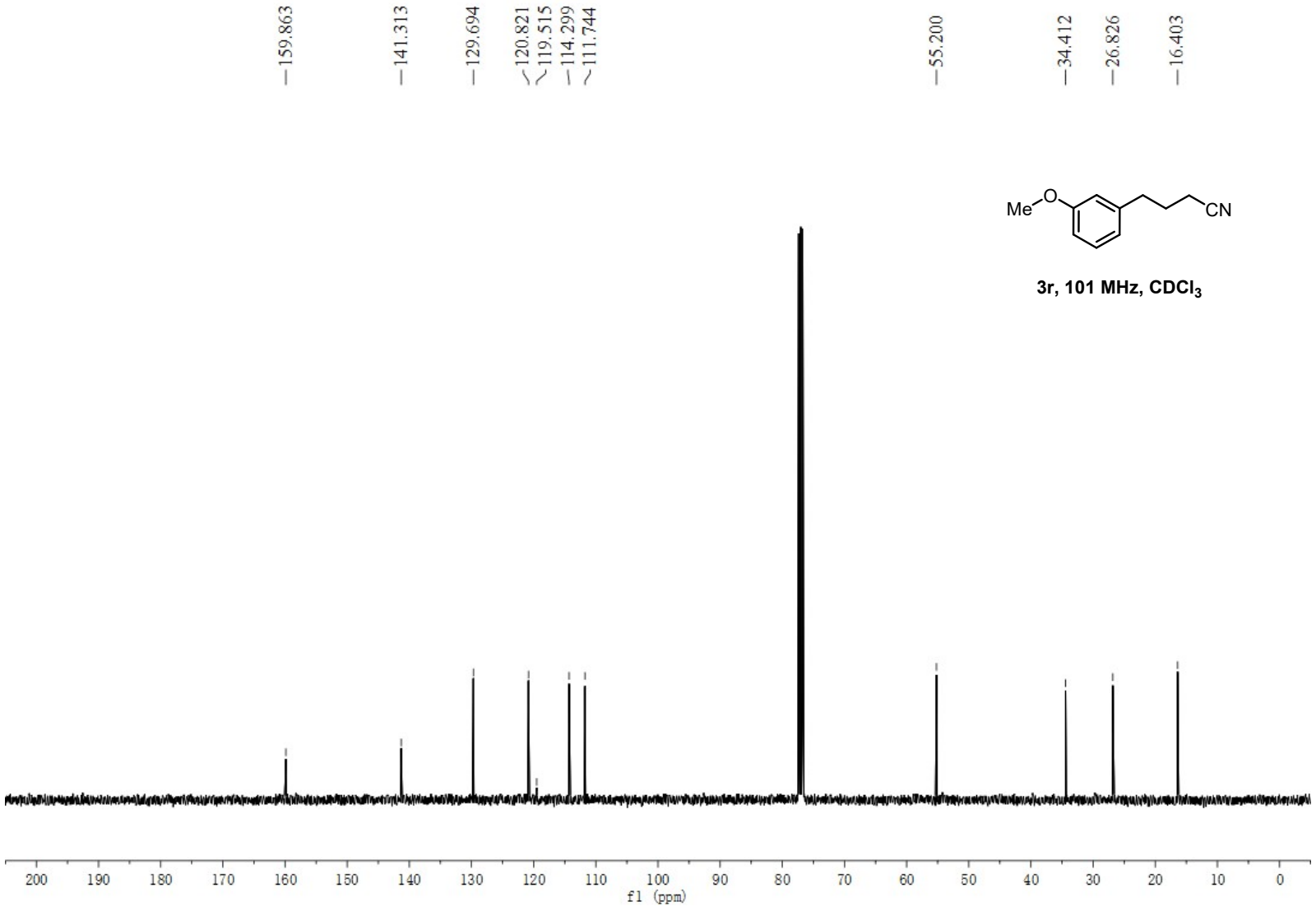
3q, 400 MHz, $CDCl_3$



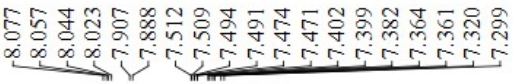
3q, 101 MHz, $CDCl_3$

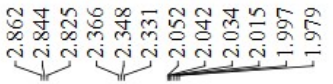


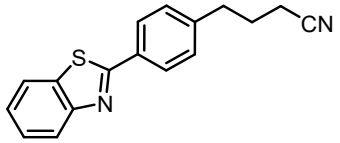
3r, 400 MHz, $CDCl_3$



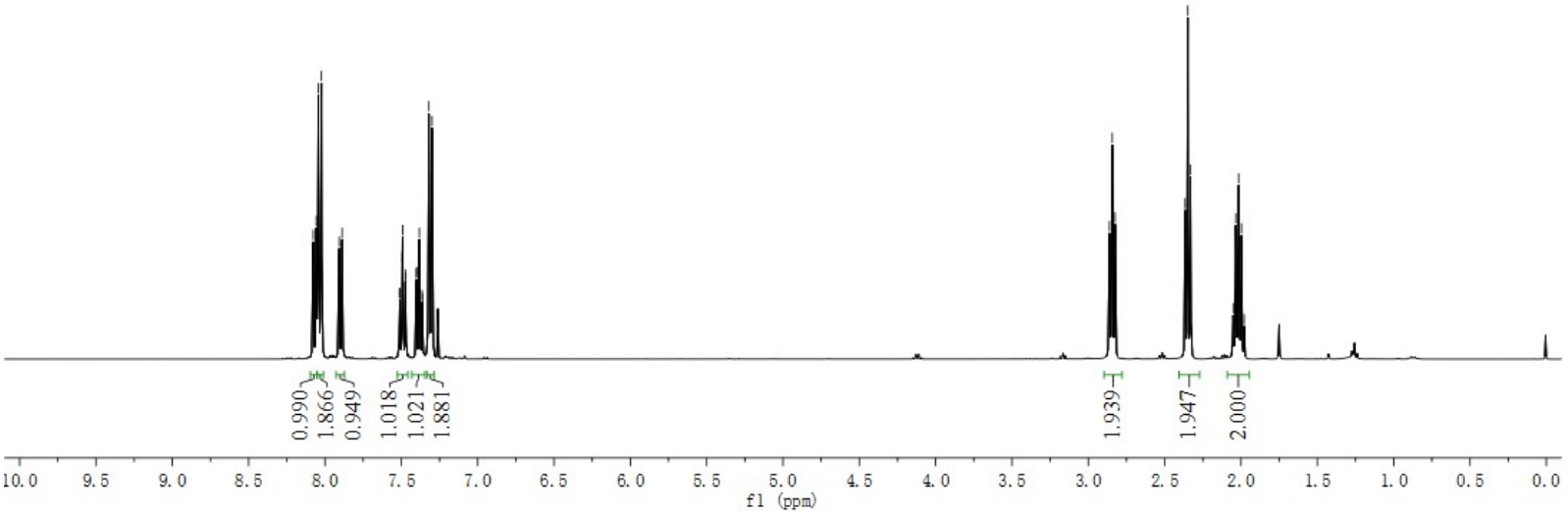
3r, 101 MHz, $CDCl_3$

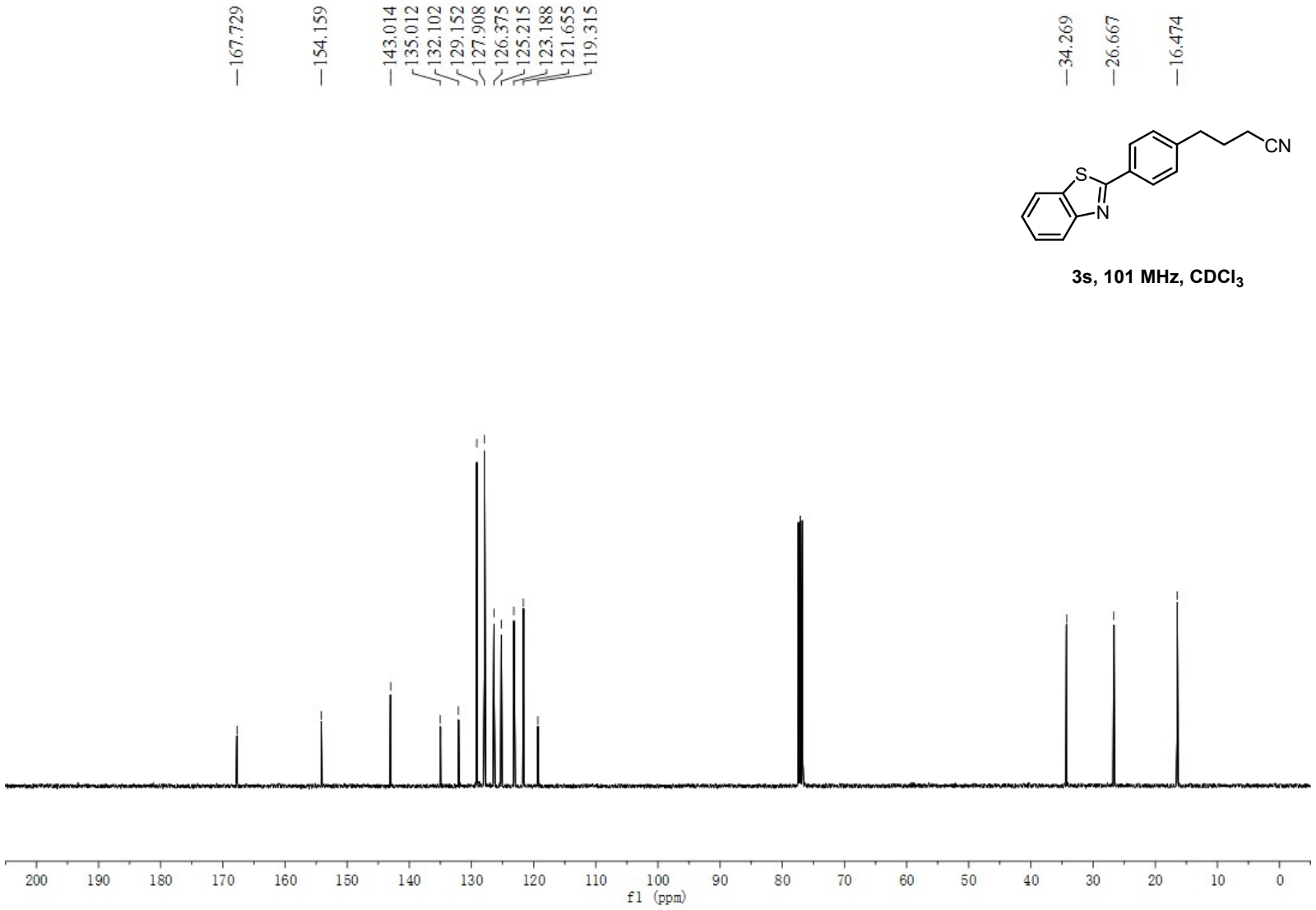




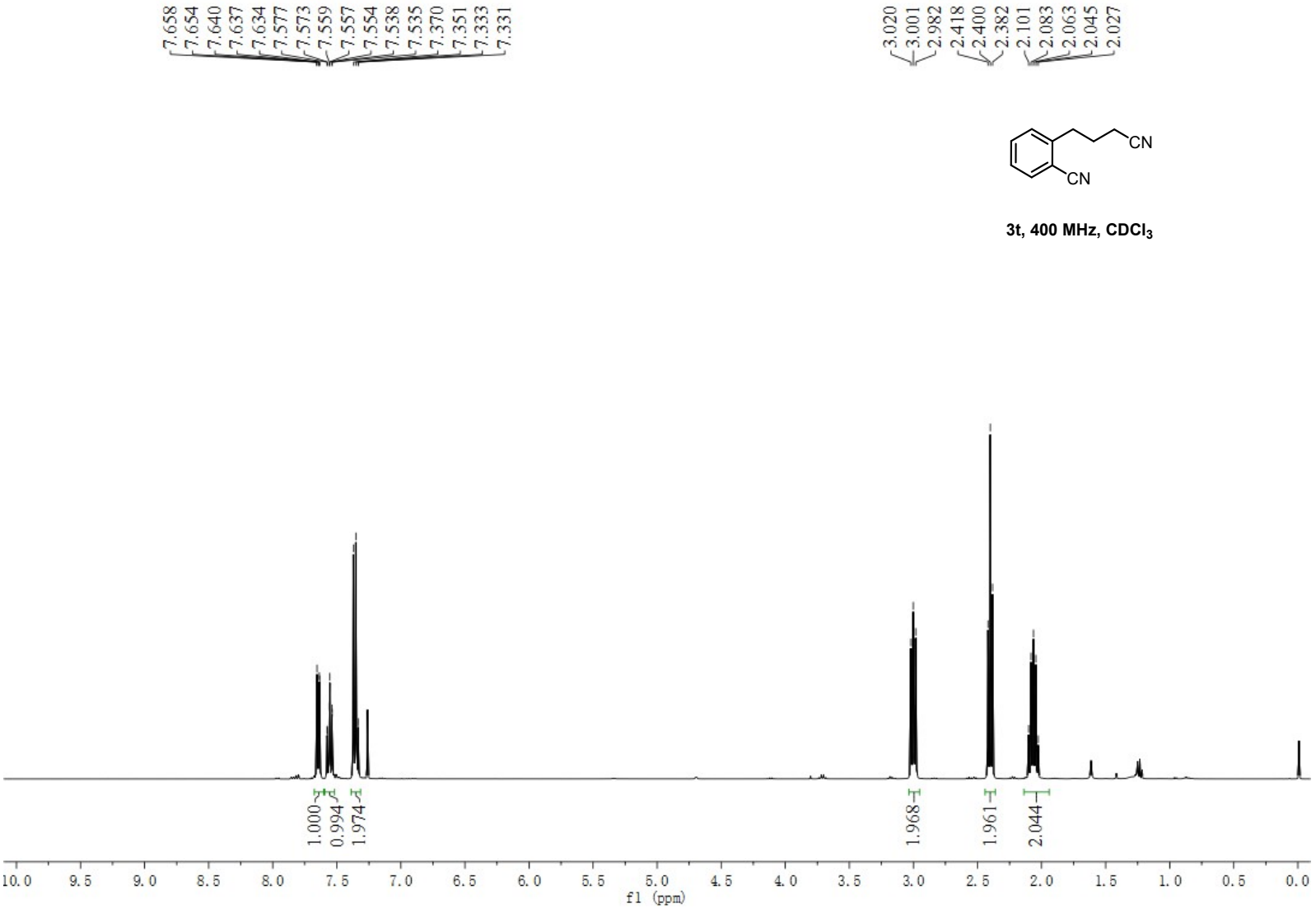


3s, 400 MHz, $CDCl_3$

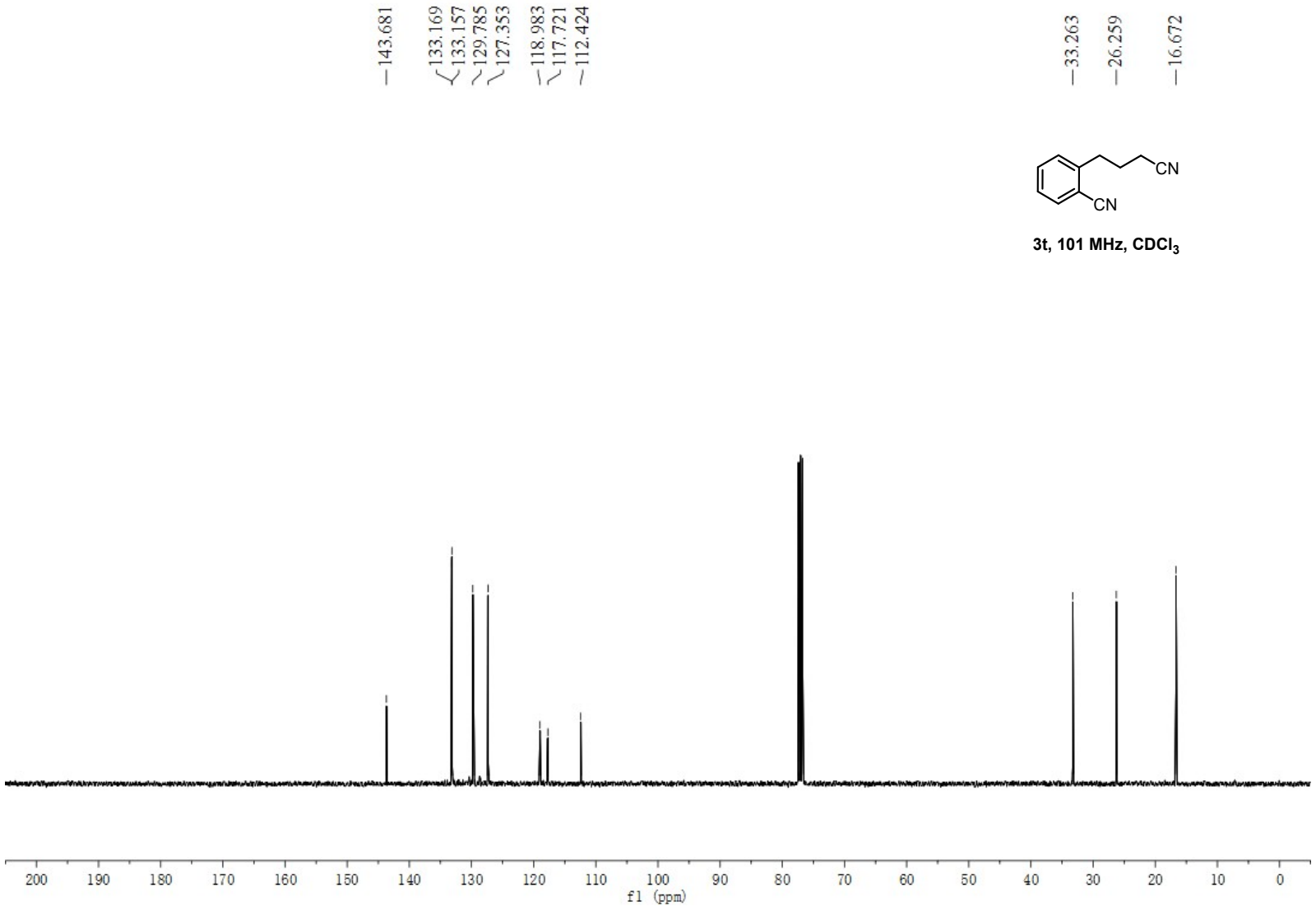




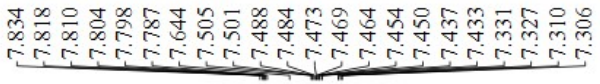
3s, 101 MHz, $CDCl_3$

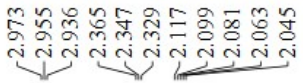


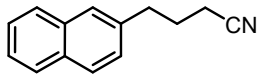
3t, 400 MHz, $CDCl_3$



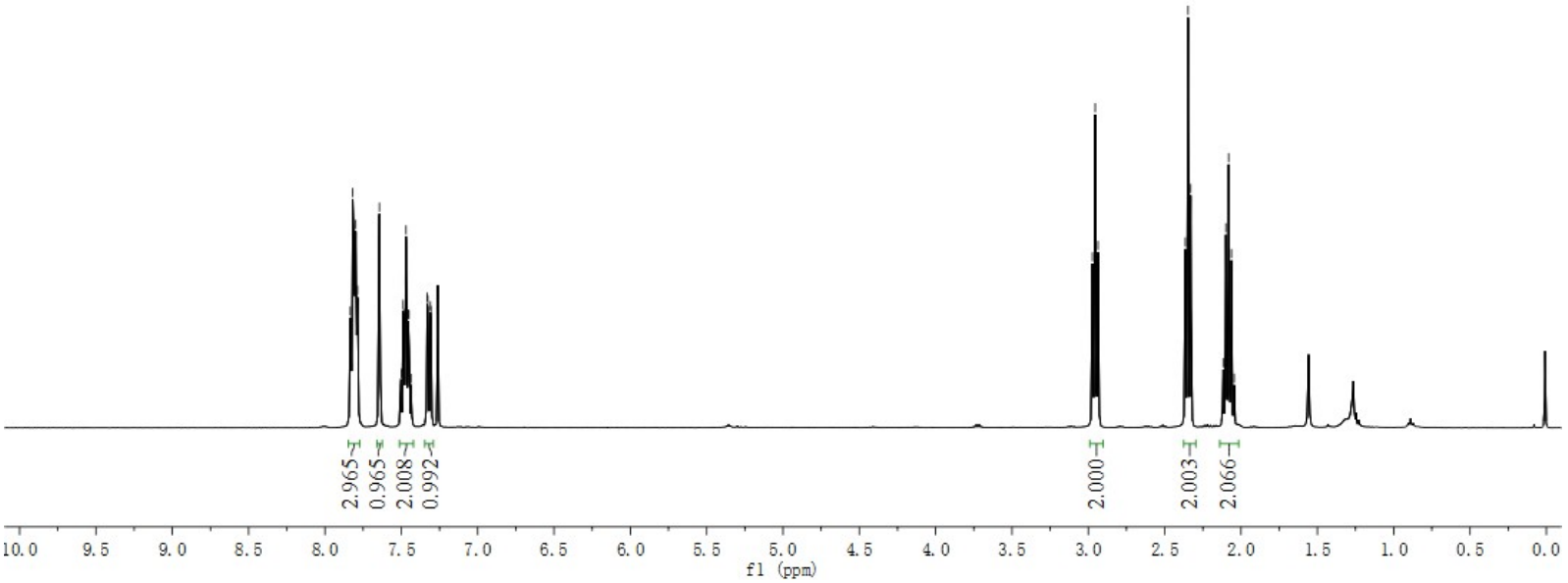
3t, 101 MHz, $CDCl_3$

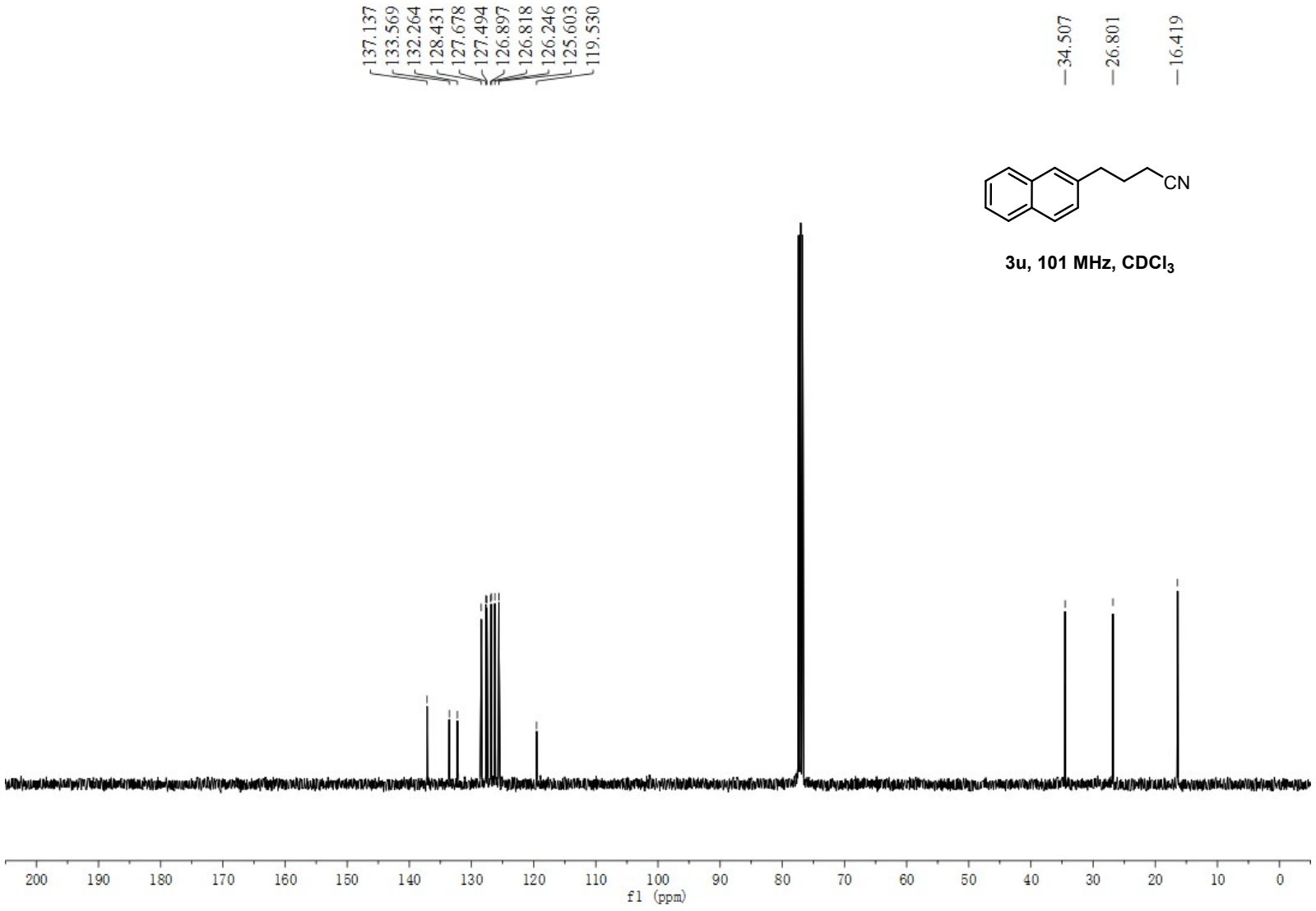




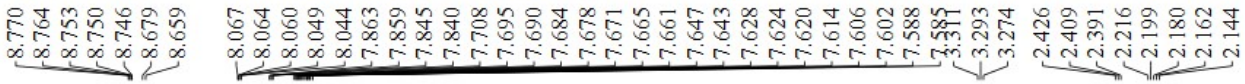


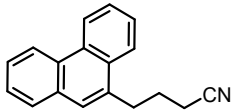
3u, 400 MHz, $CDCl_3$



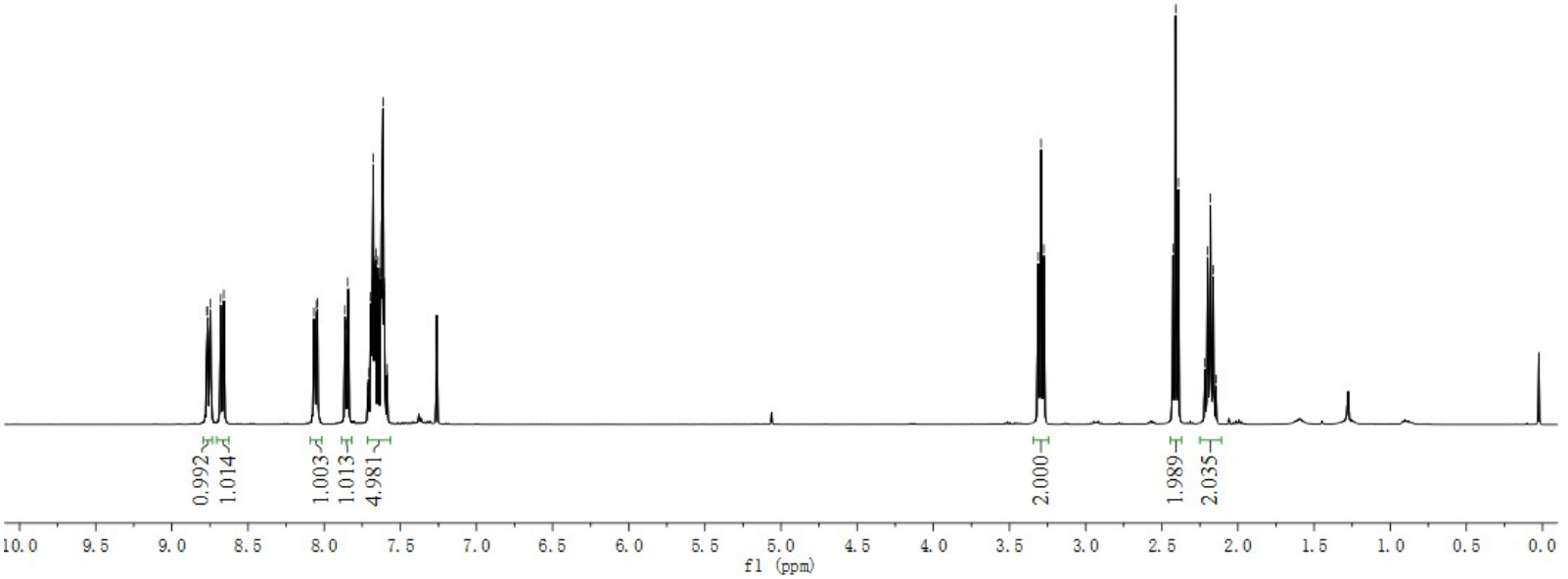


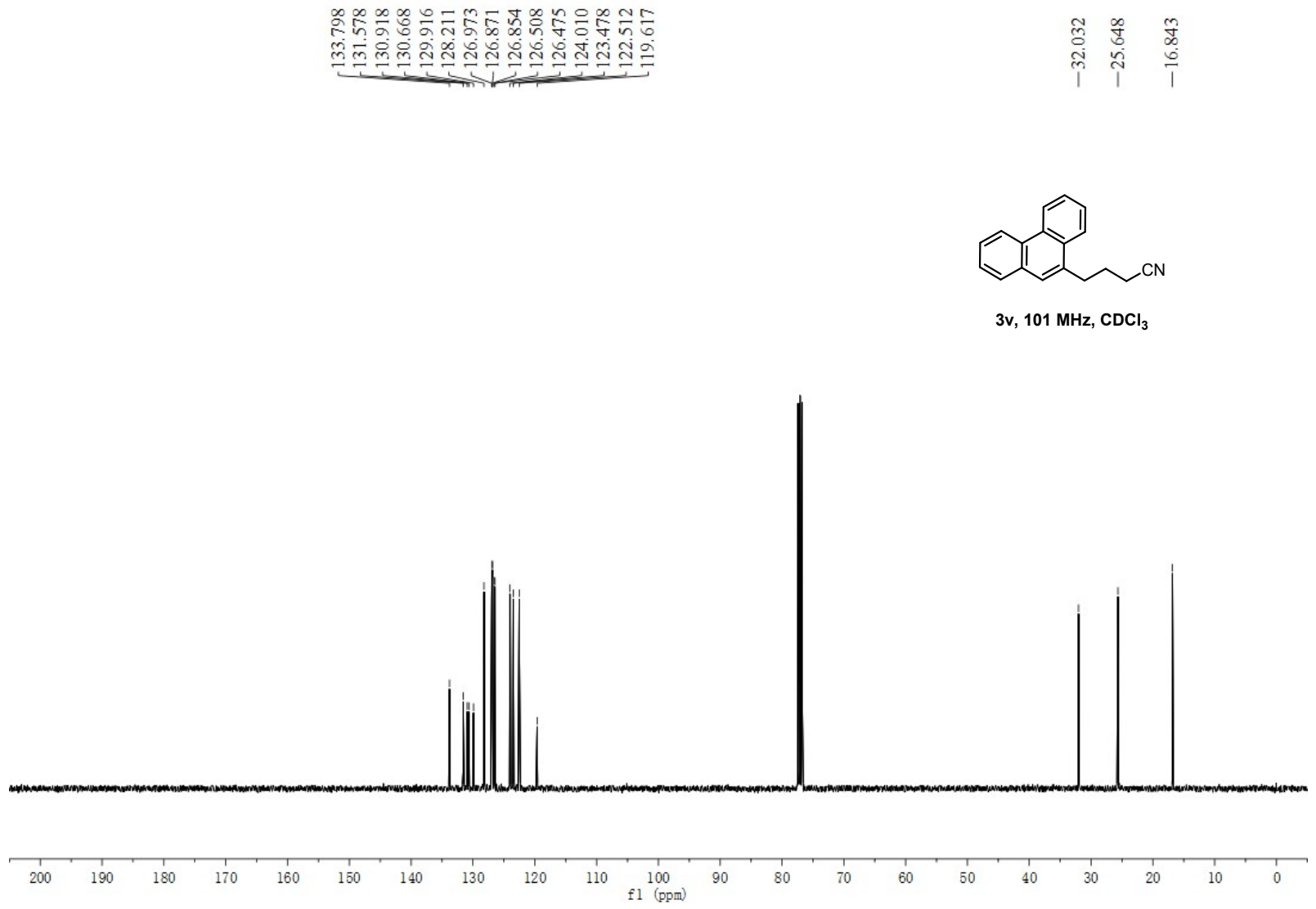
3u, 101 MHz, $CDCl_3$



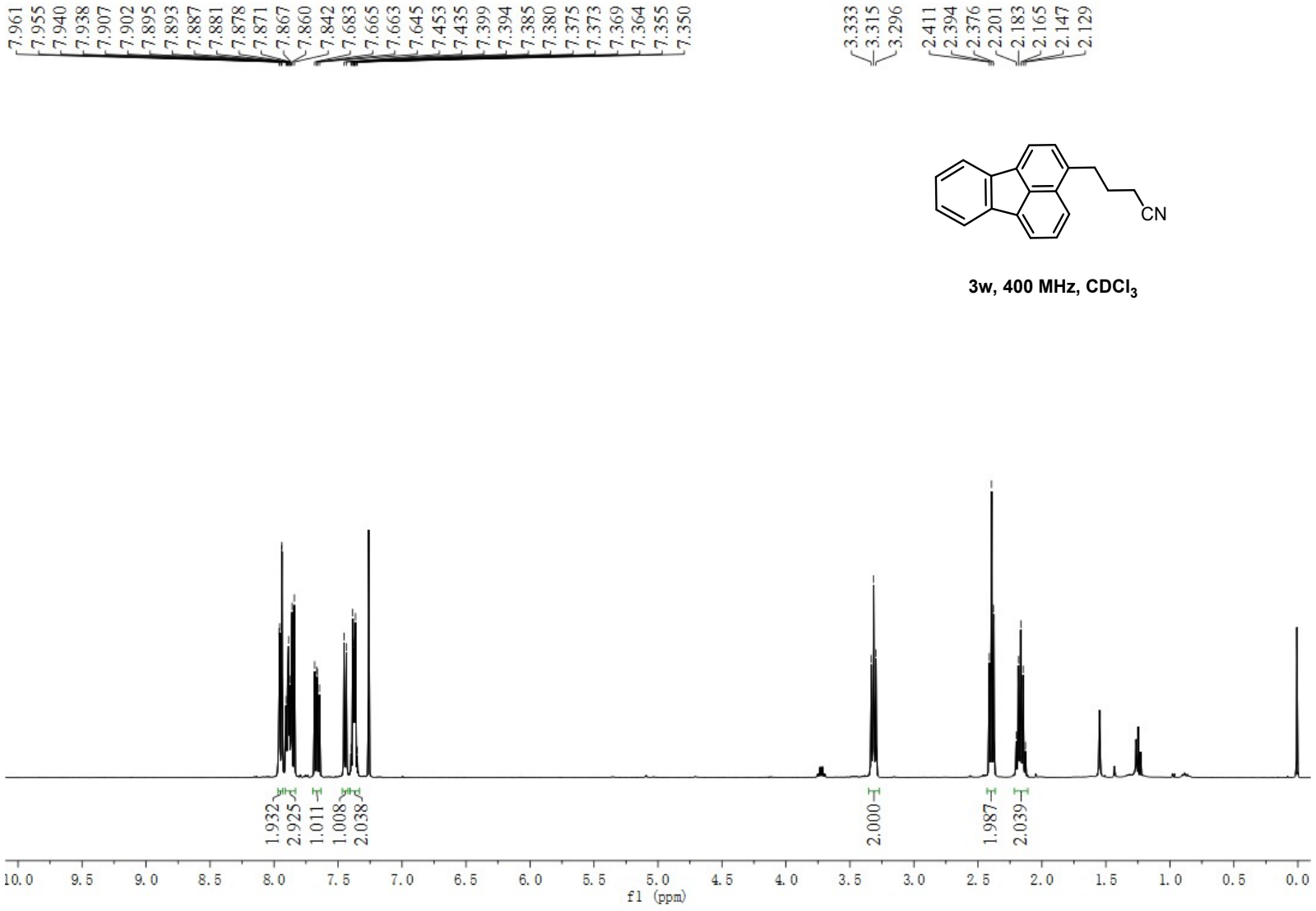


3v, 400 MHz, $CDCl_3$

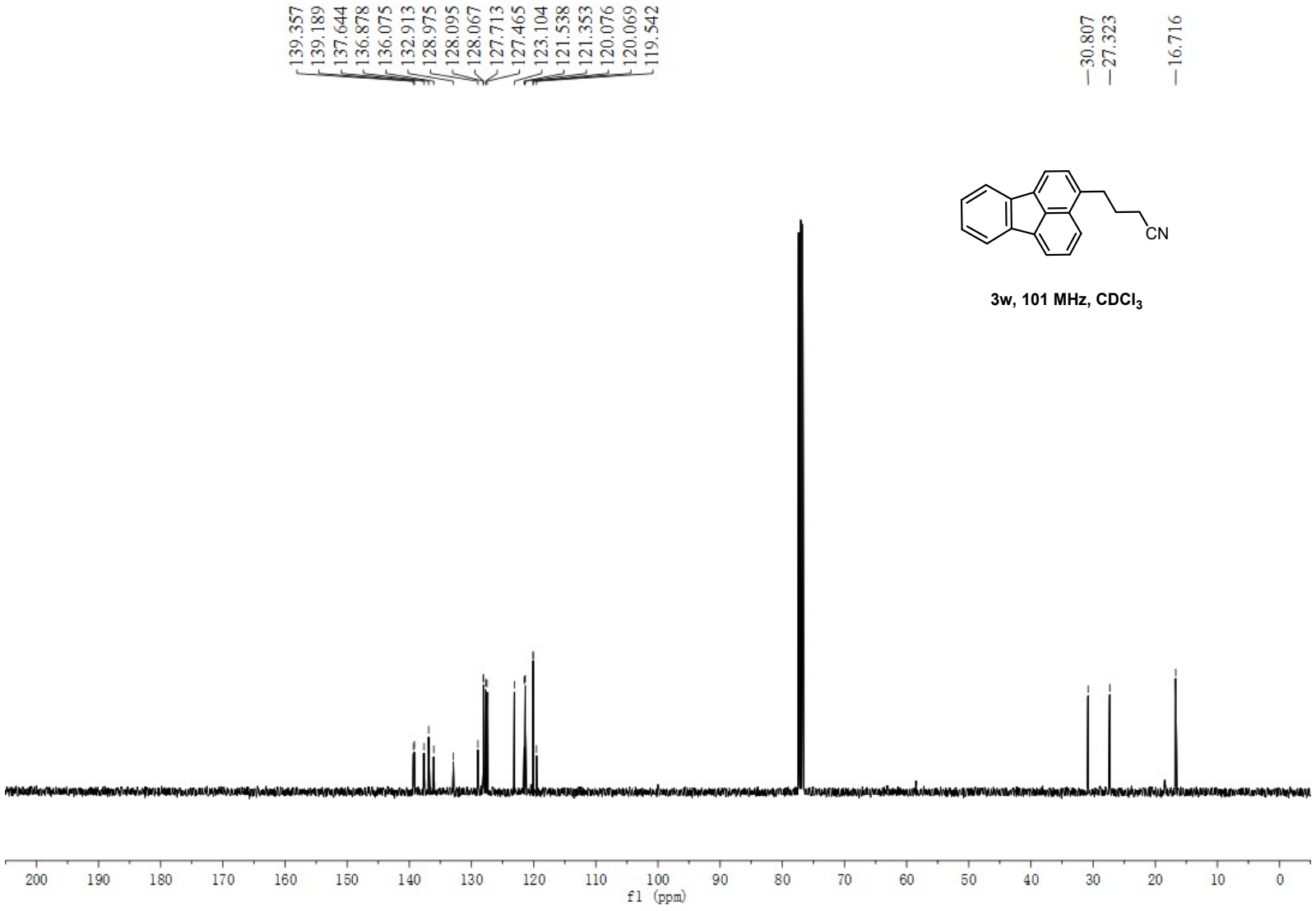


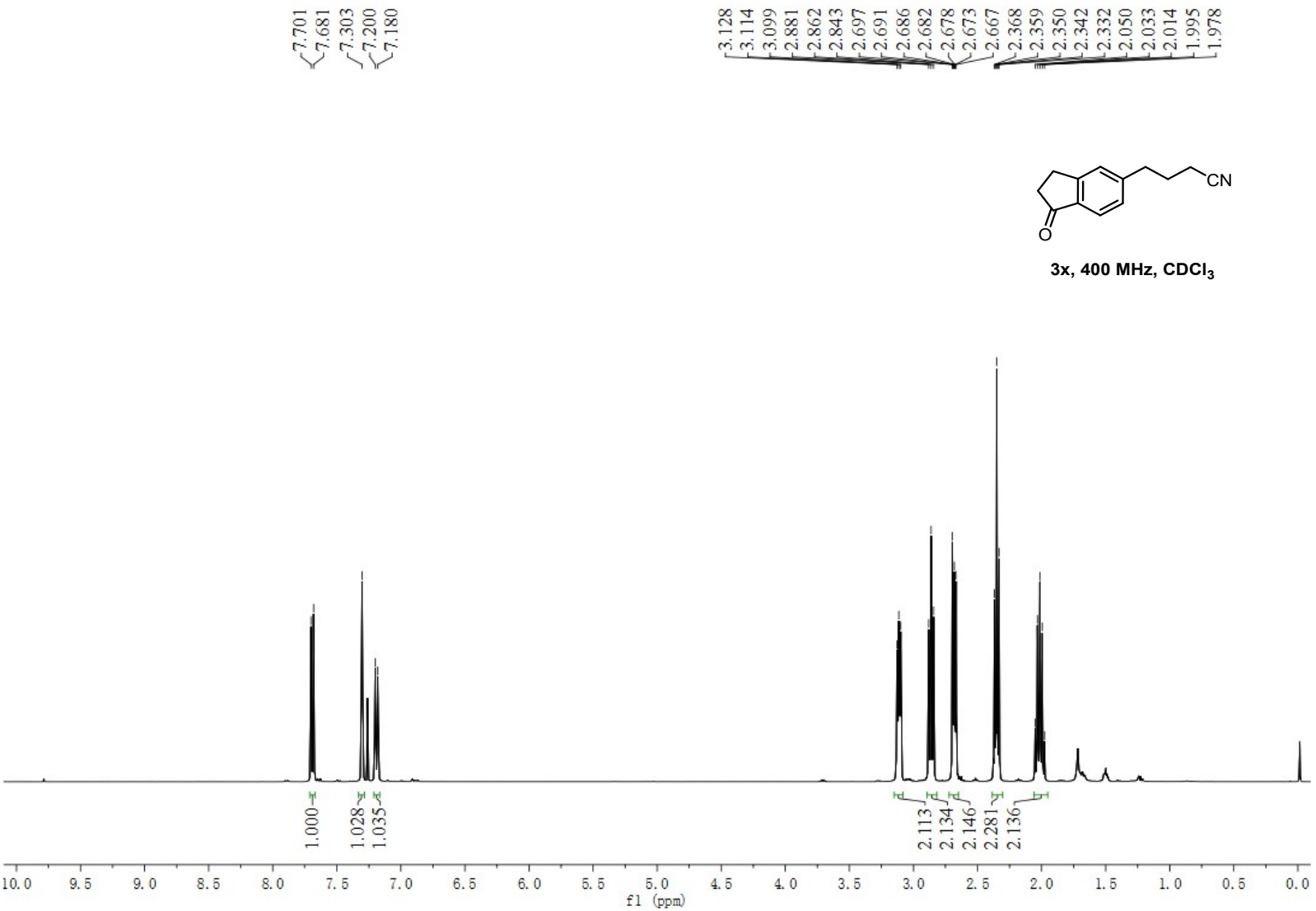


3v, 101 MHz, $CDCl_3$

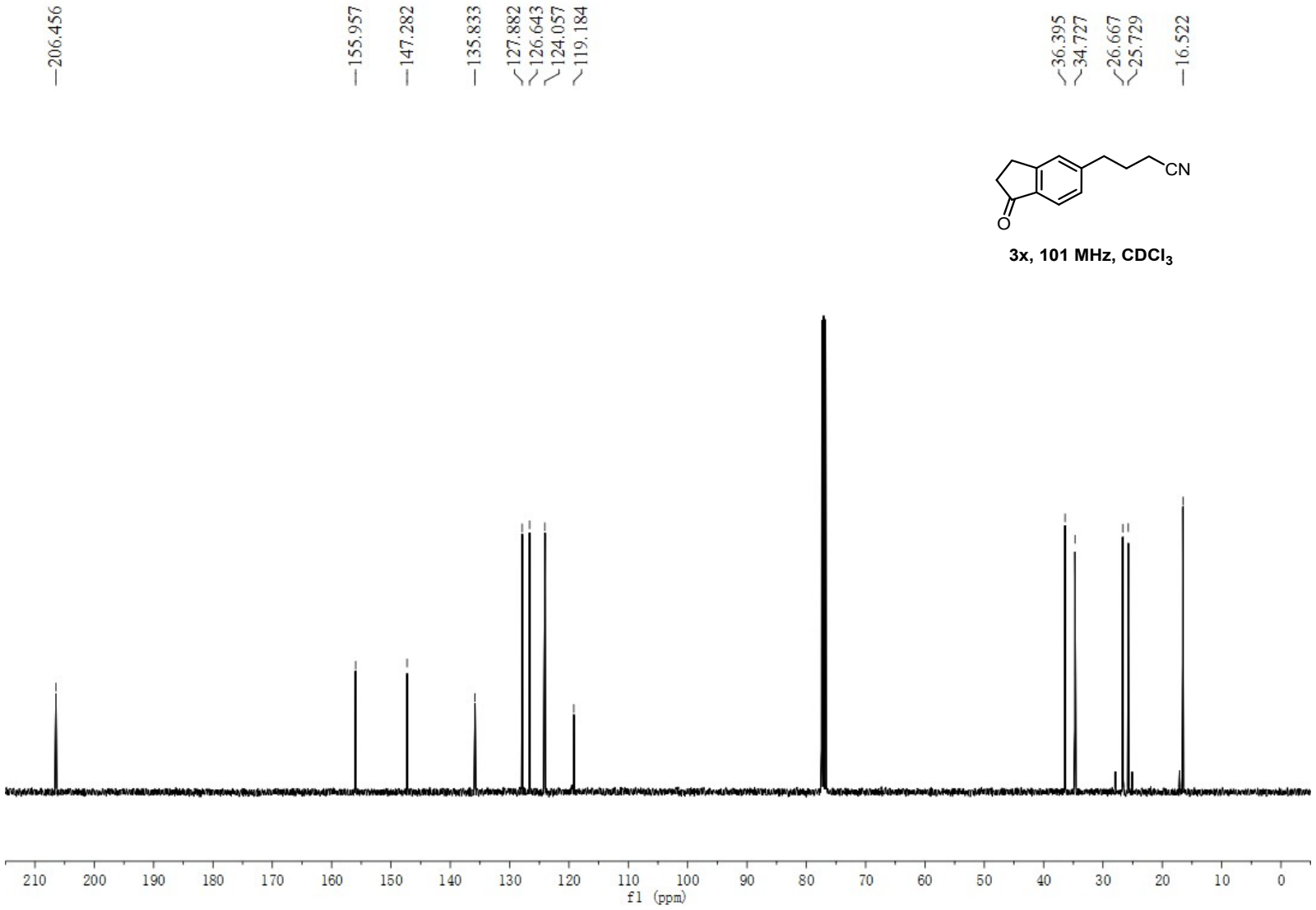


3w, 400 MHz, $CDCl_3$

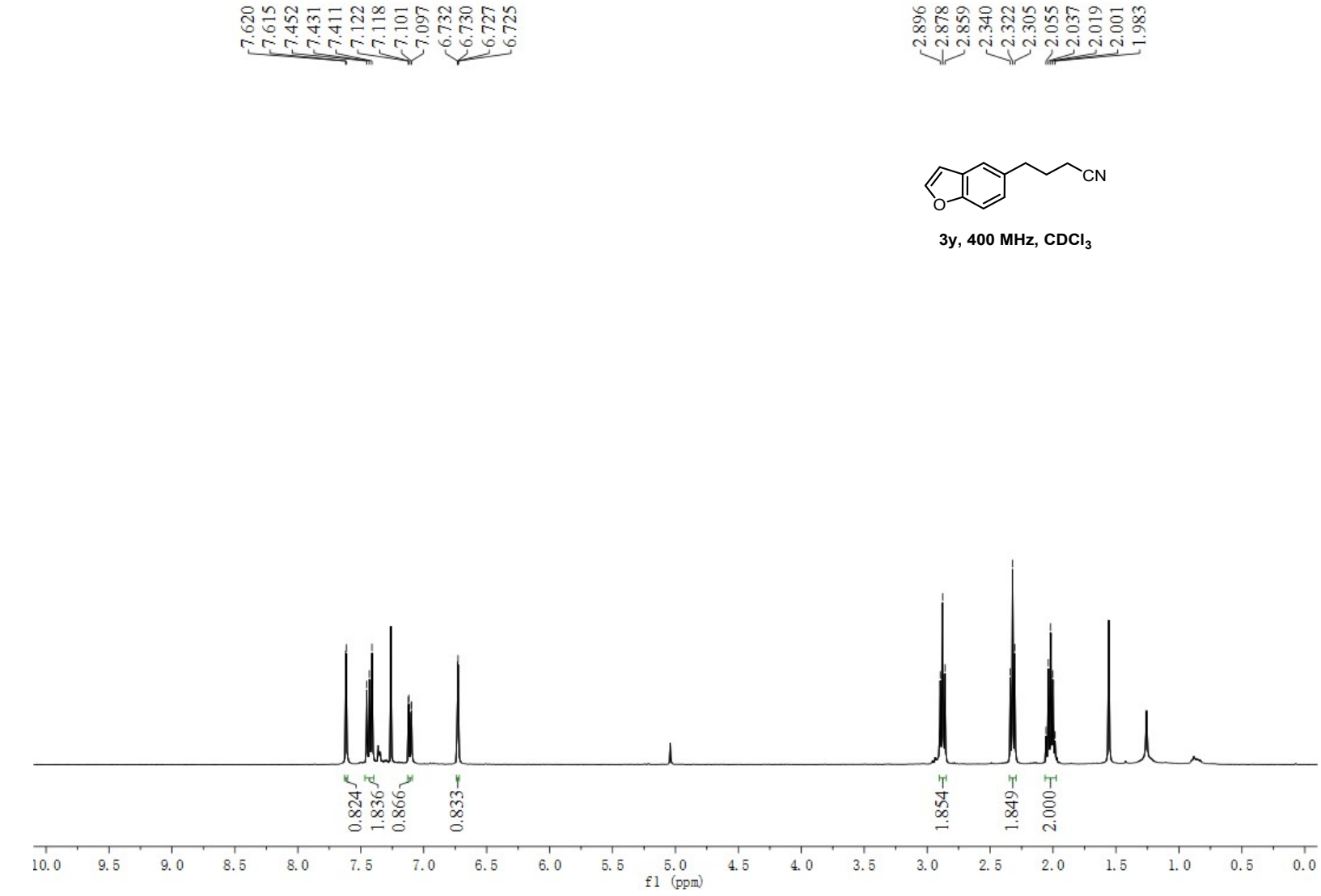

139.357
139.189
137.644
136.878
136.075
132.913
128.975
128.095
128.067
127.713
127.465
123.104
121.538
121.353
120.076
120.069
119.542
30.807
27.323
16.716
CN
3w, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



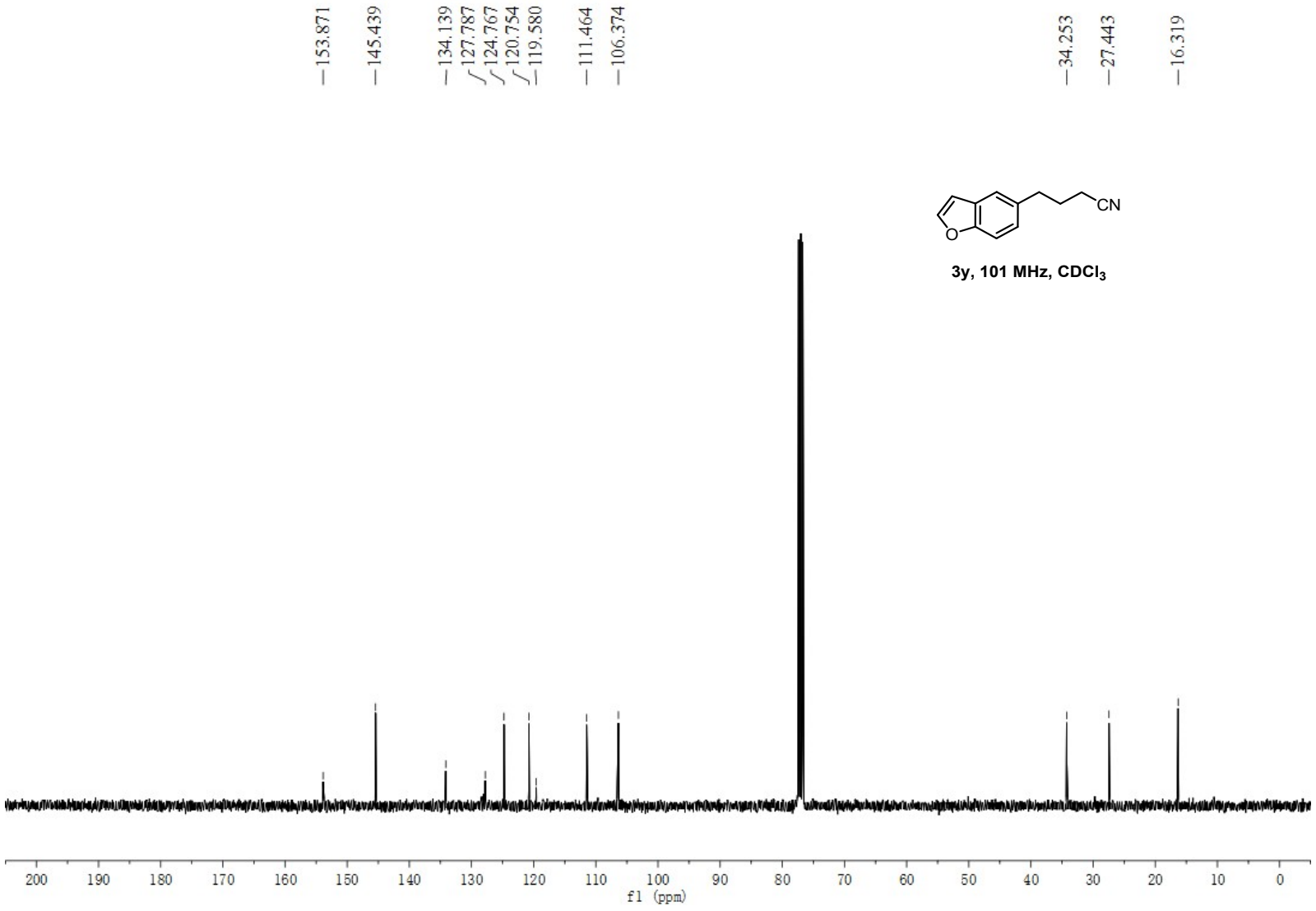
3x, 400 MHz, $CDCl_3$

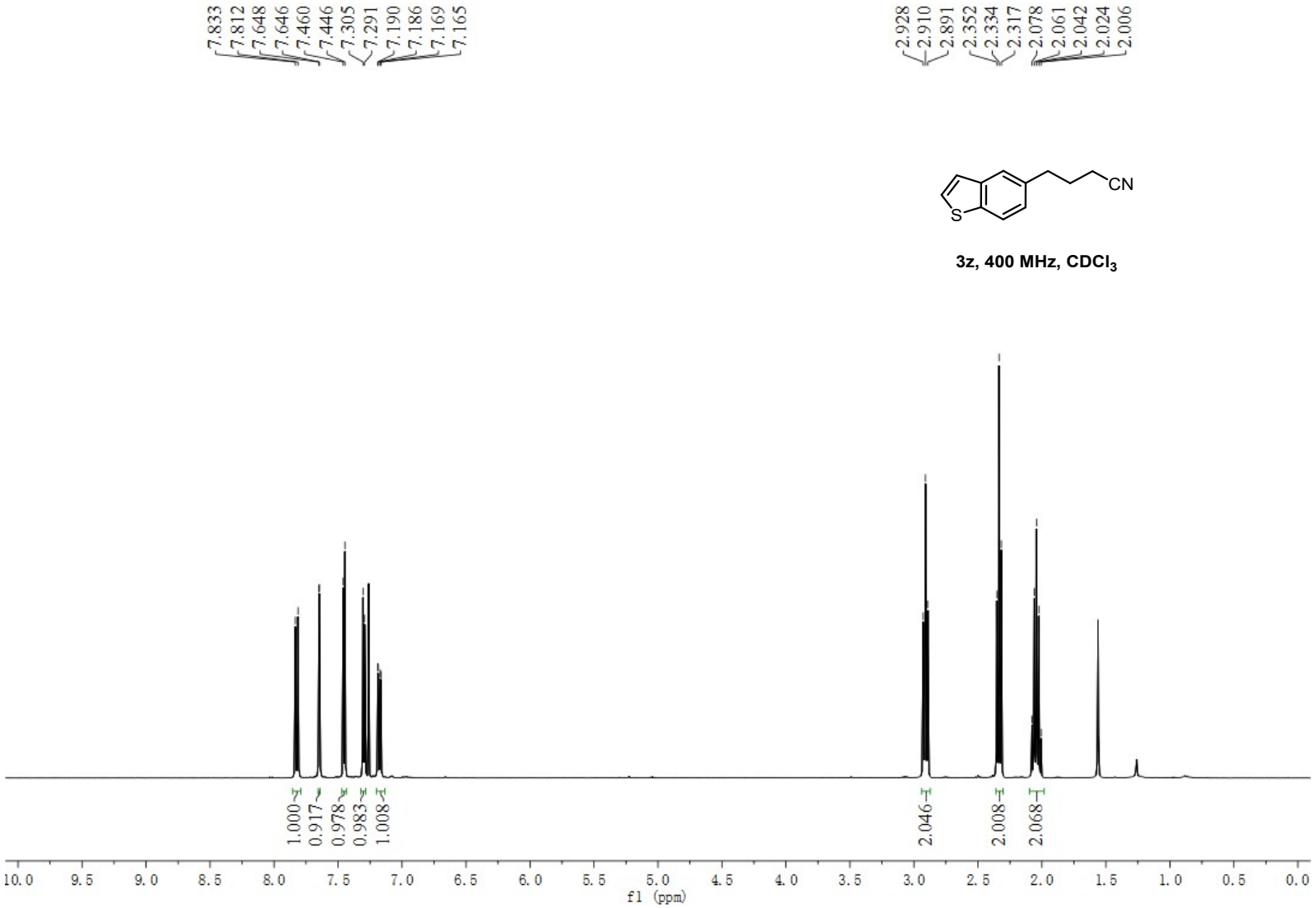


3x, 101 MHz, $CDCl_3$

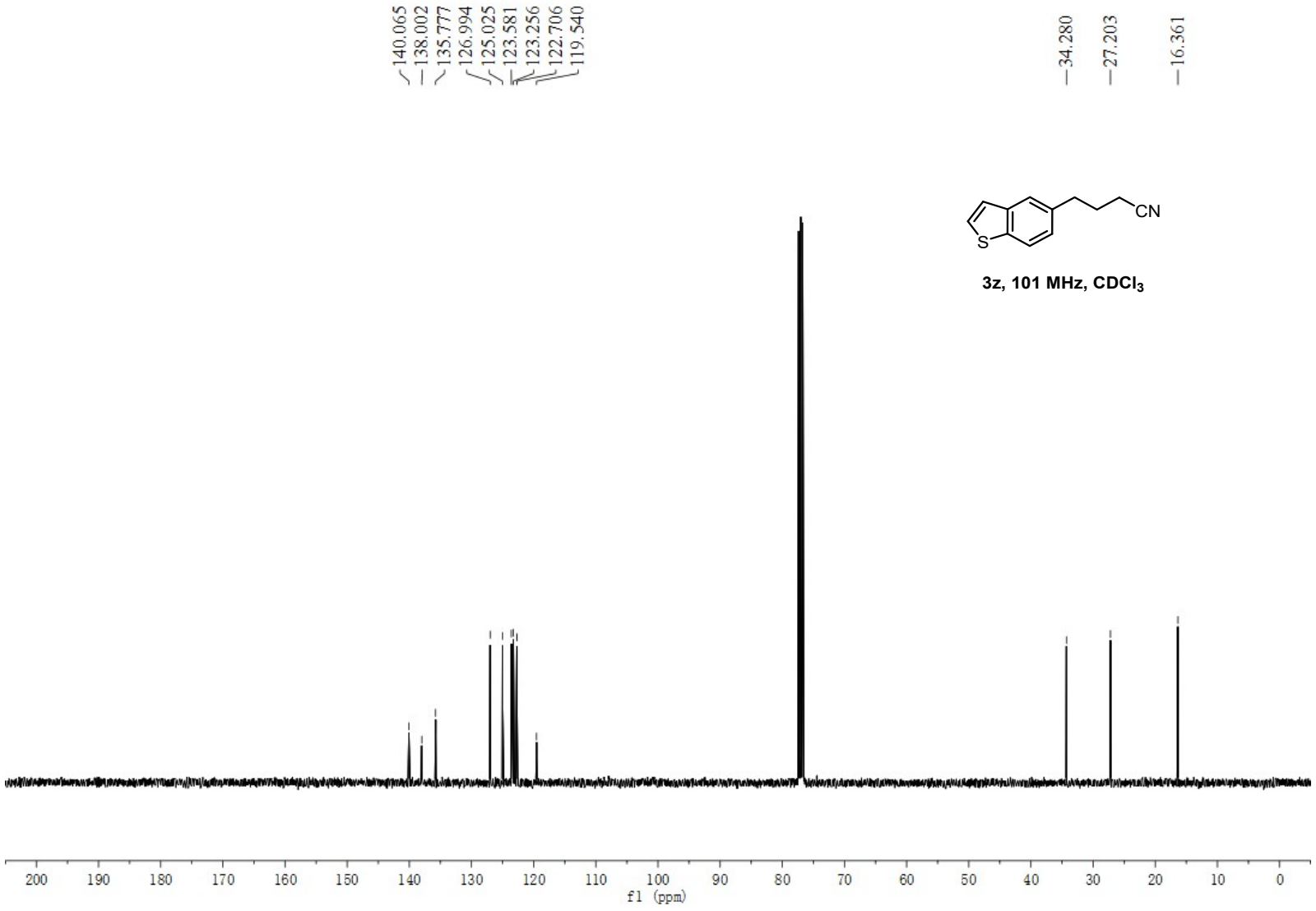


3y, 400 MHz, $CDCl_3$

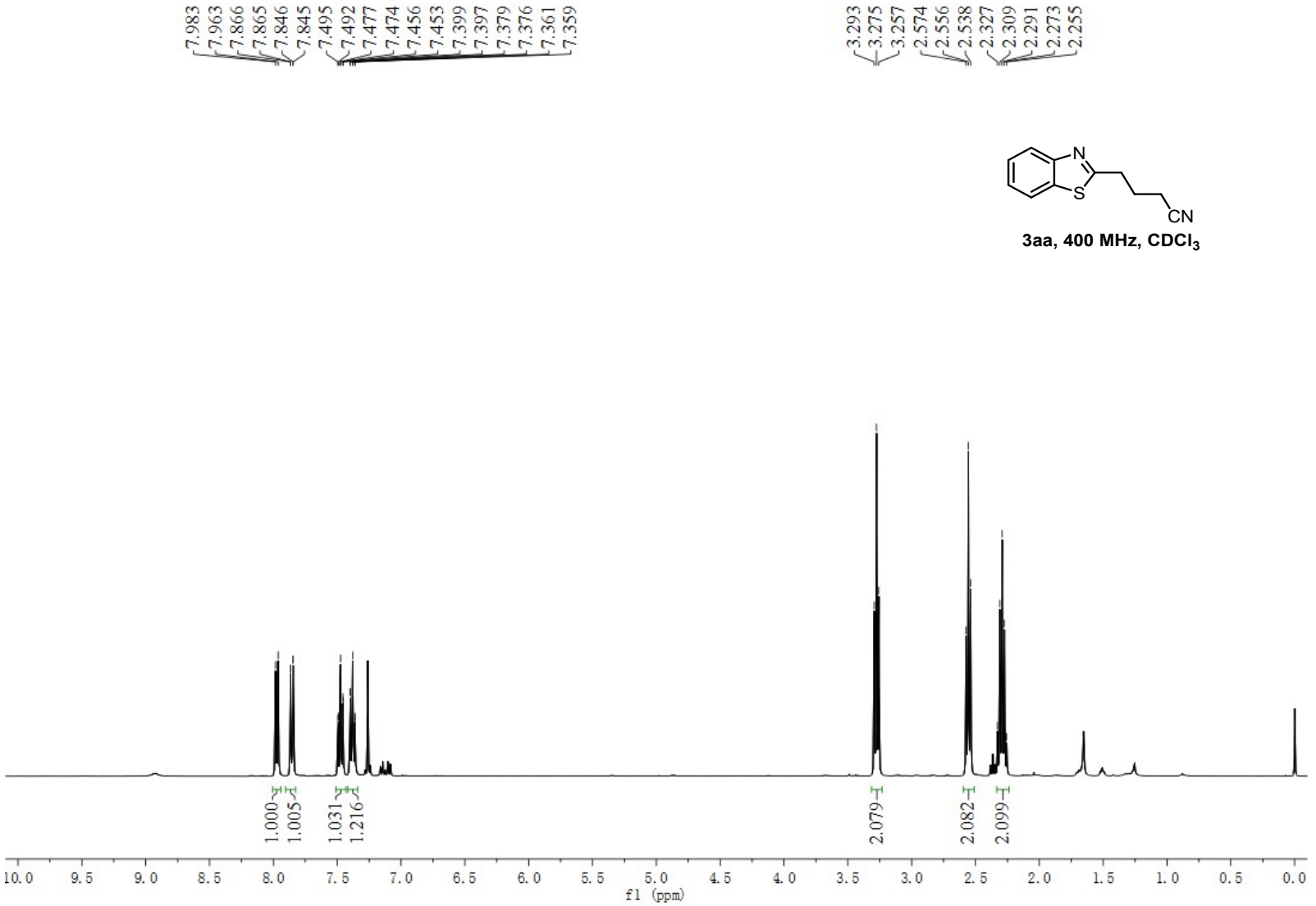

153.871
145.439
134.139
127.787
124.767
120.754
119.580
111.464
106.374
34.253
27.443
16.319
CN
O
3y, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



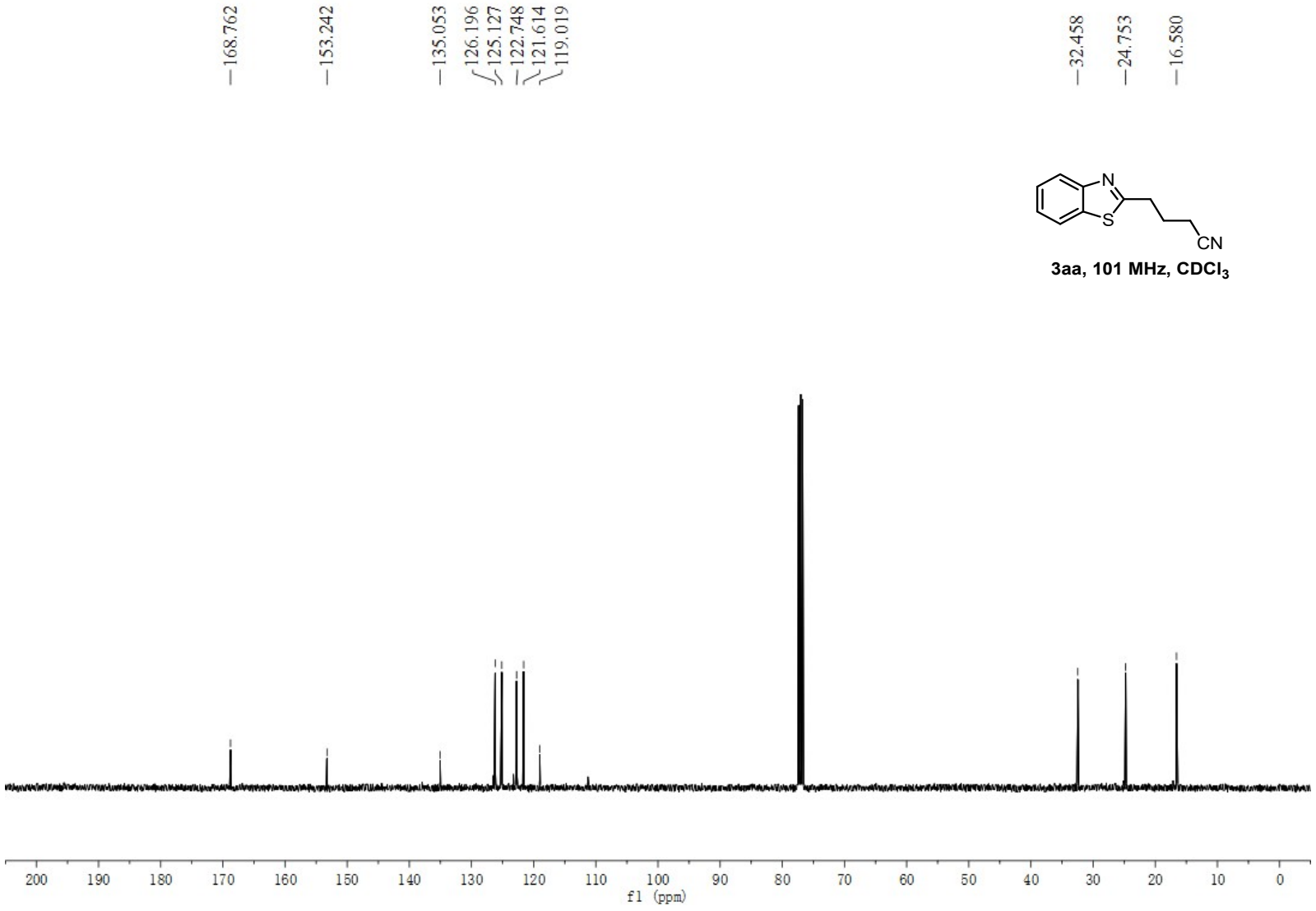
3z, 400 MHz, $CDCl_3$



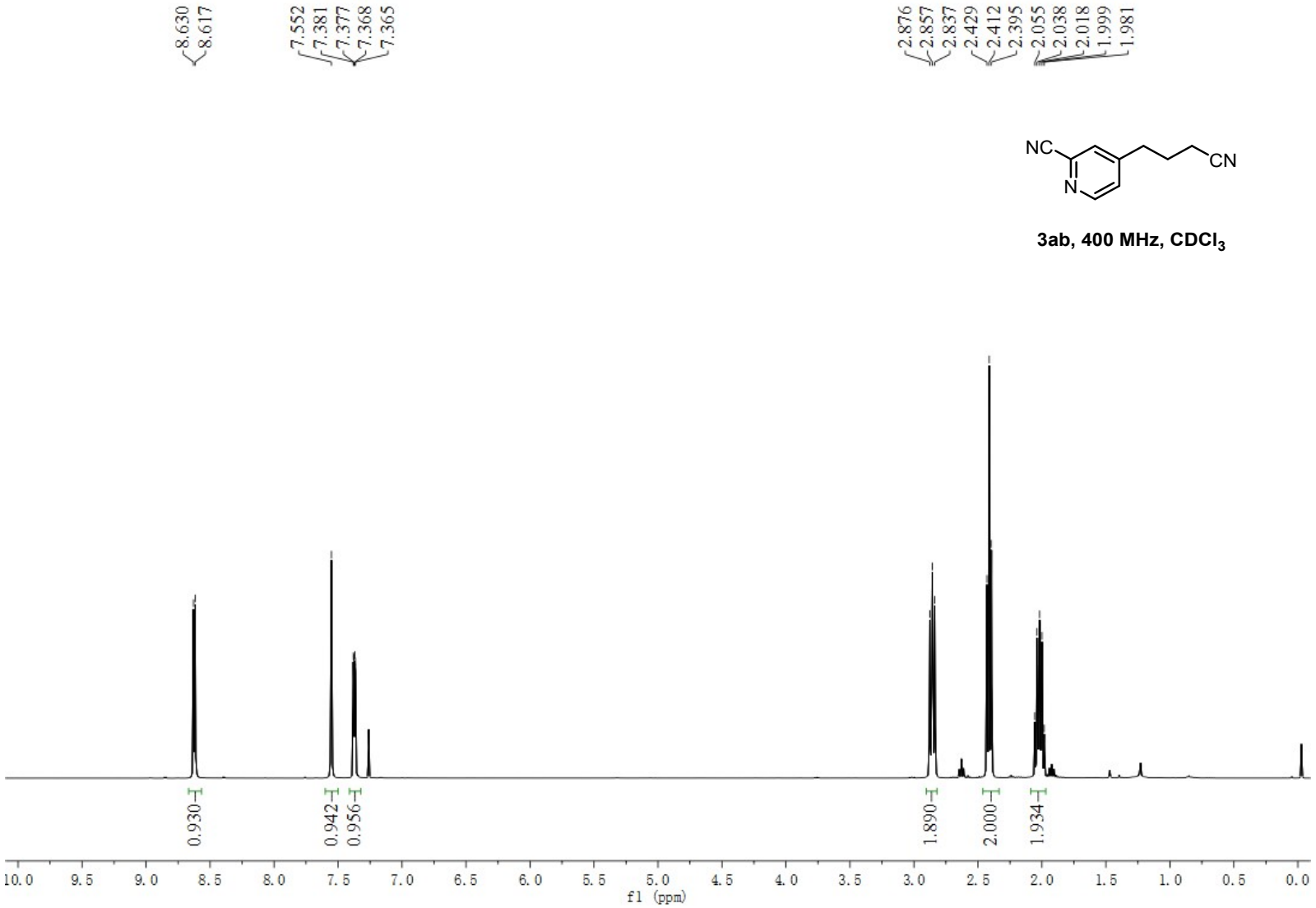
3z, 101 MHz, $CDCl_3$



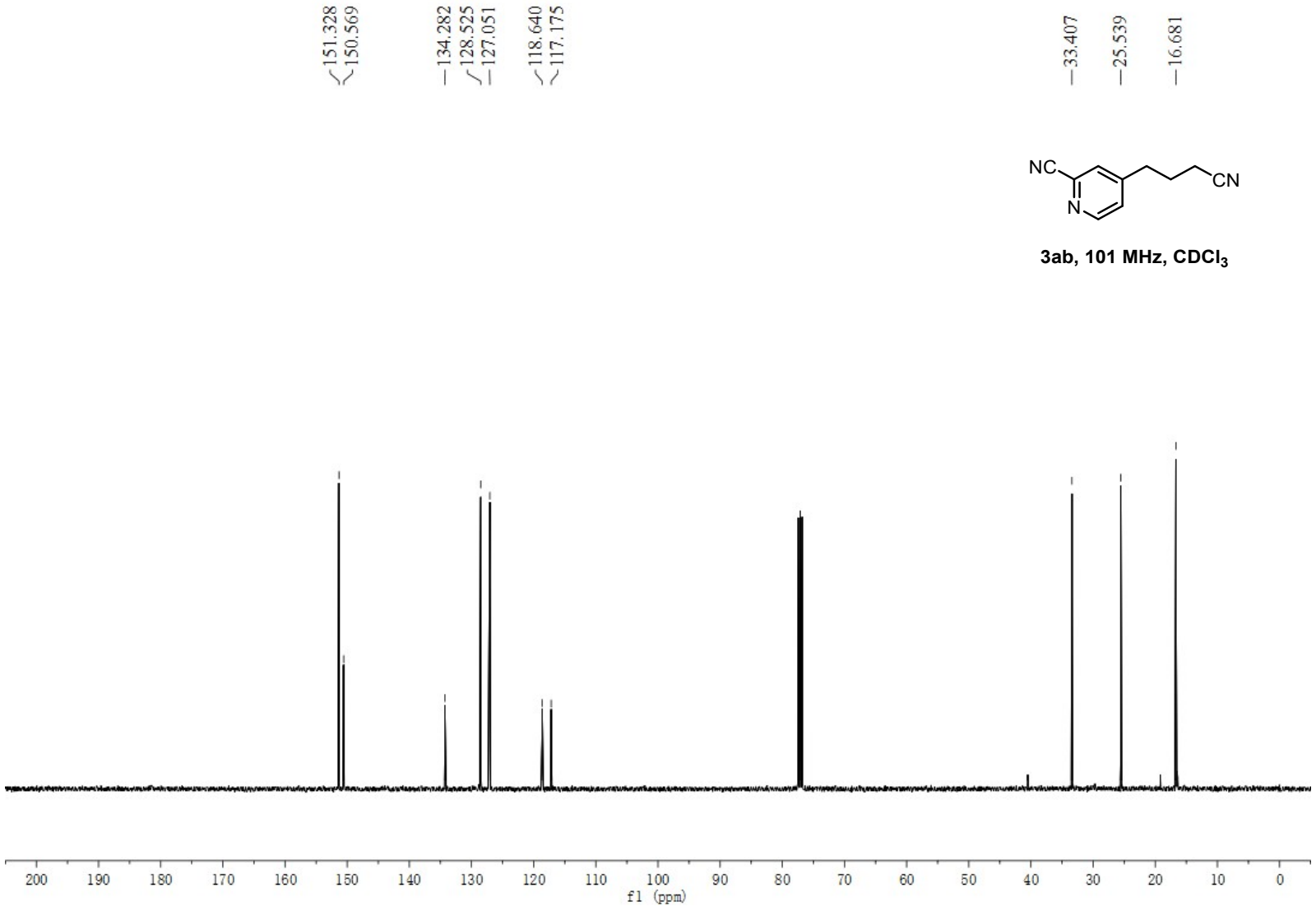
3aa, 400 MHz, $CDCl_3$



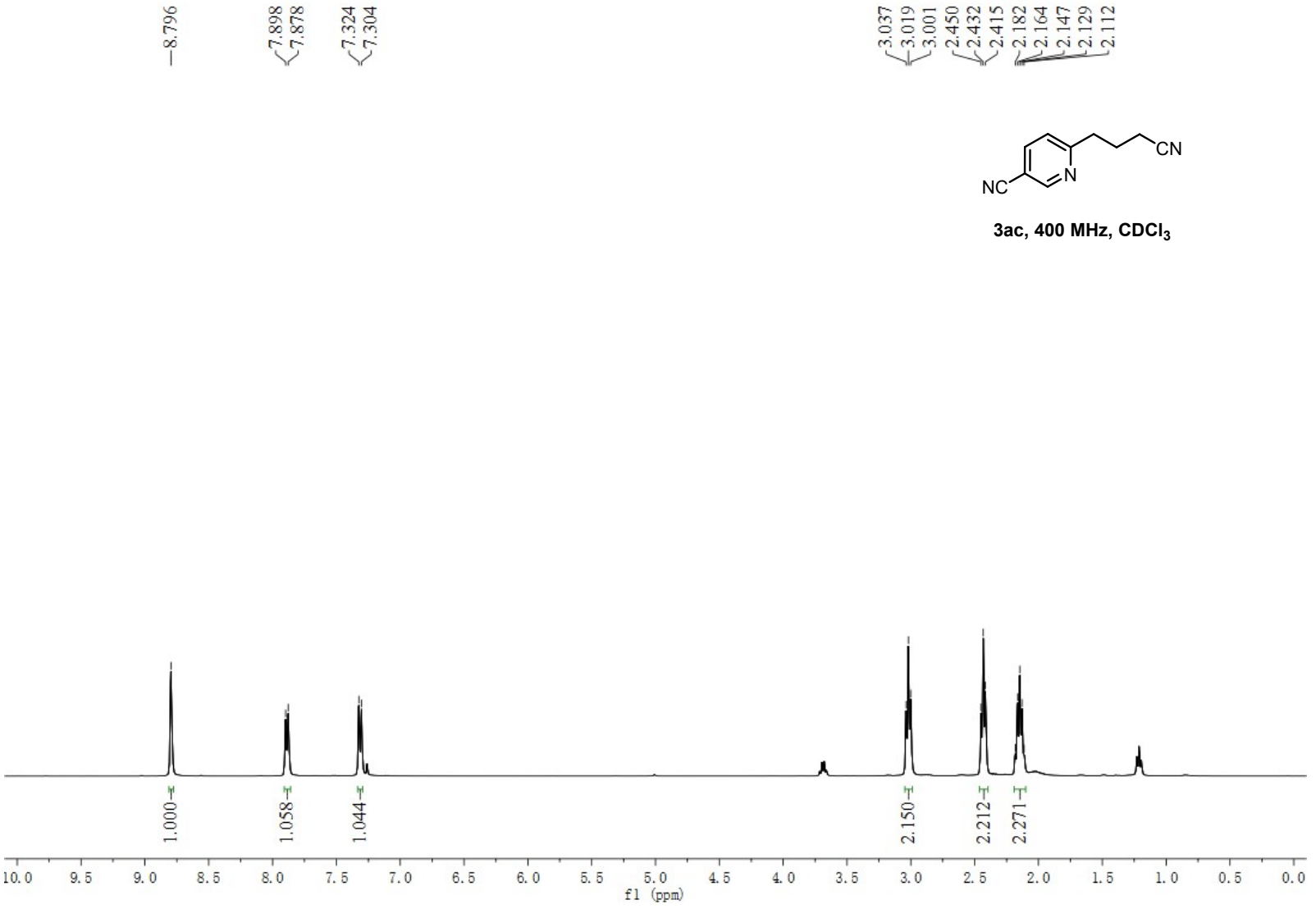
3aa, 101 MHz, $CDCl_3$



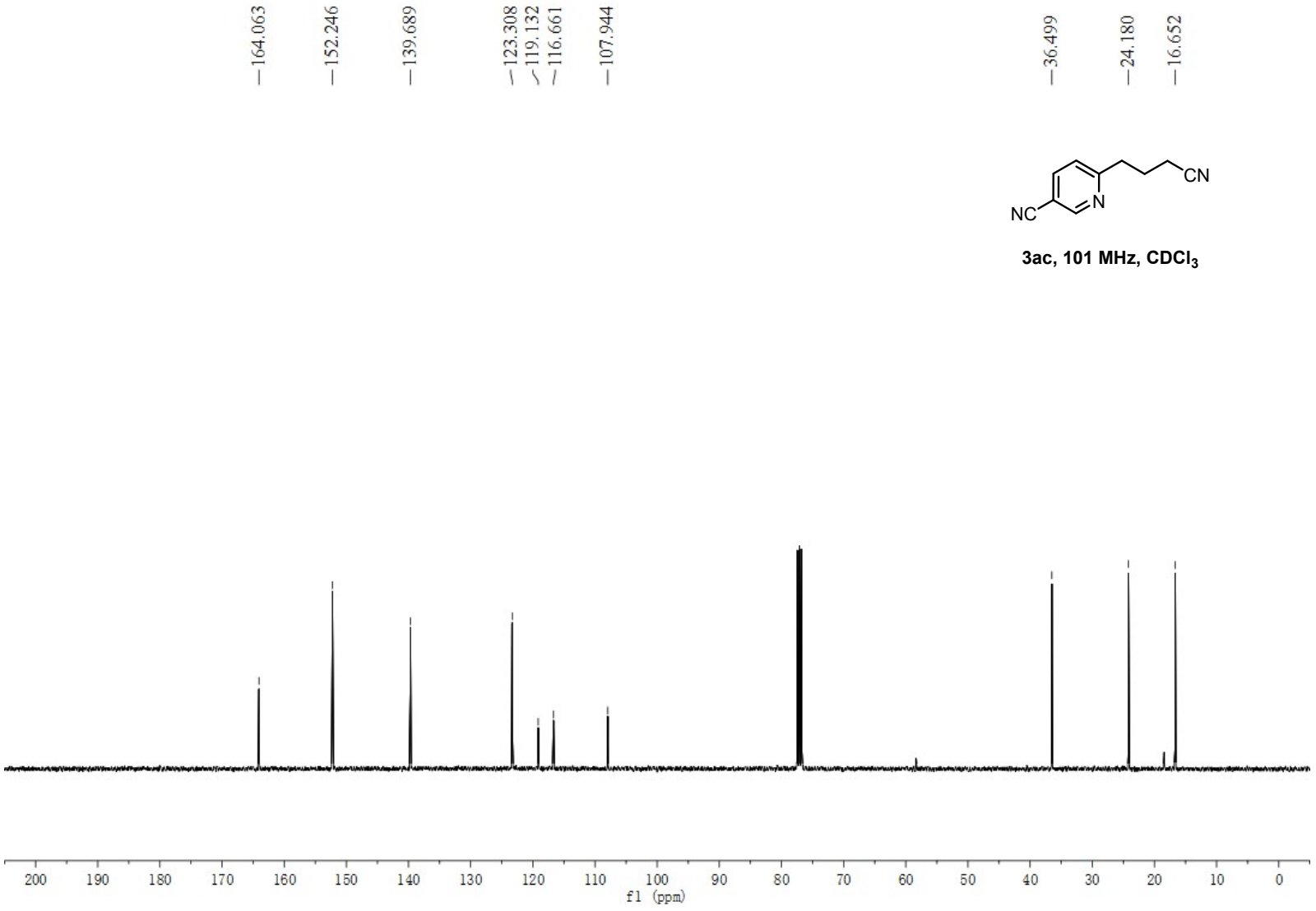
3ab, 400 MHz, $CDCl_3$



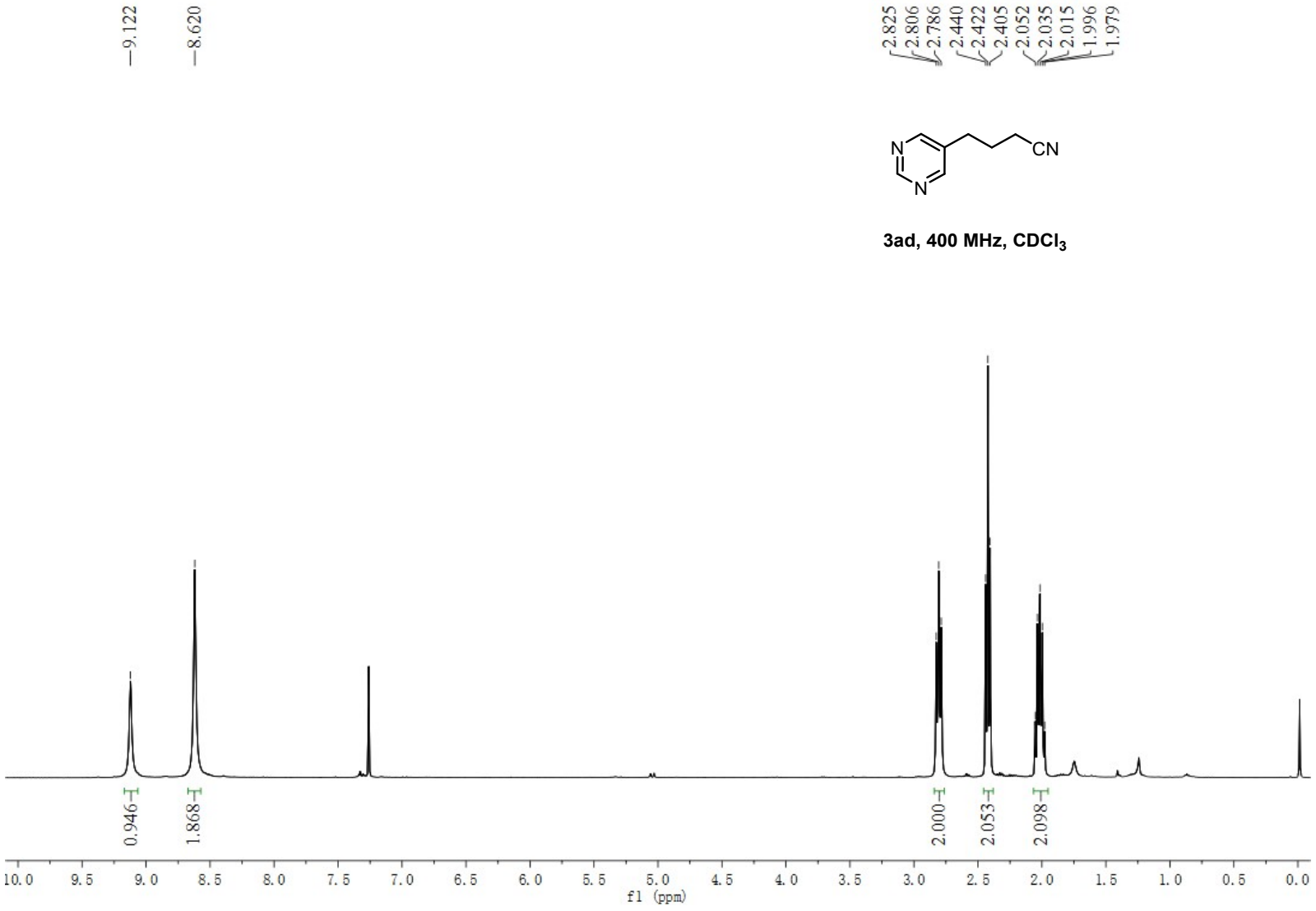
3ab, 101 MHz, $CDCl_3$



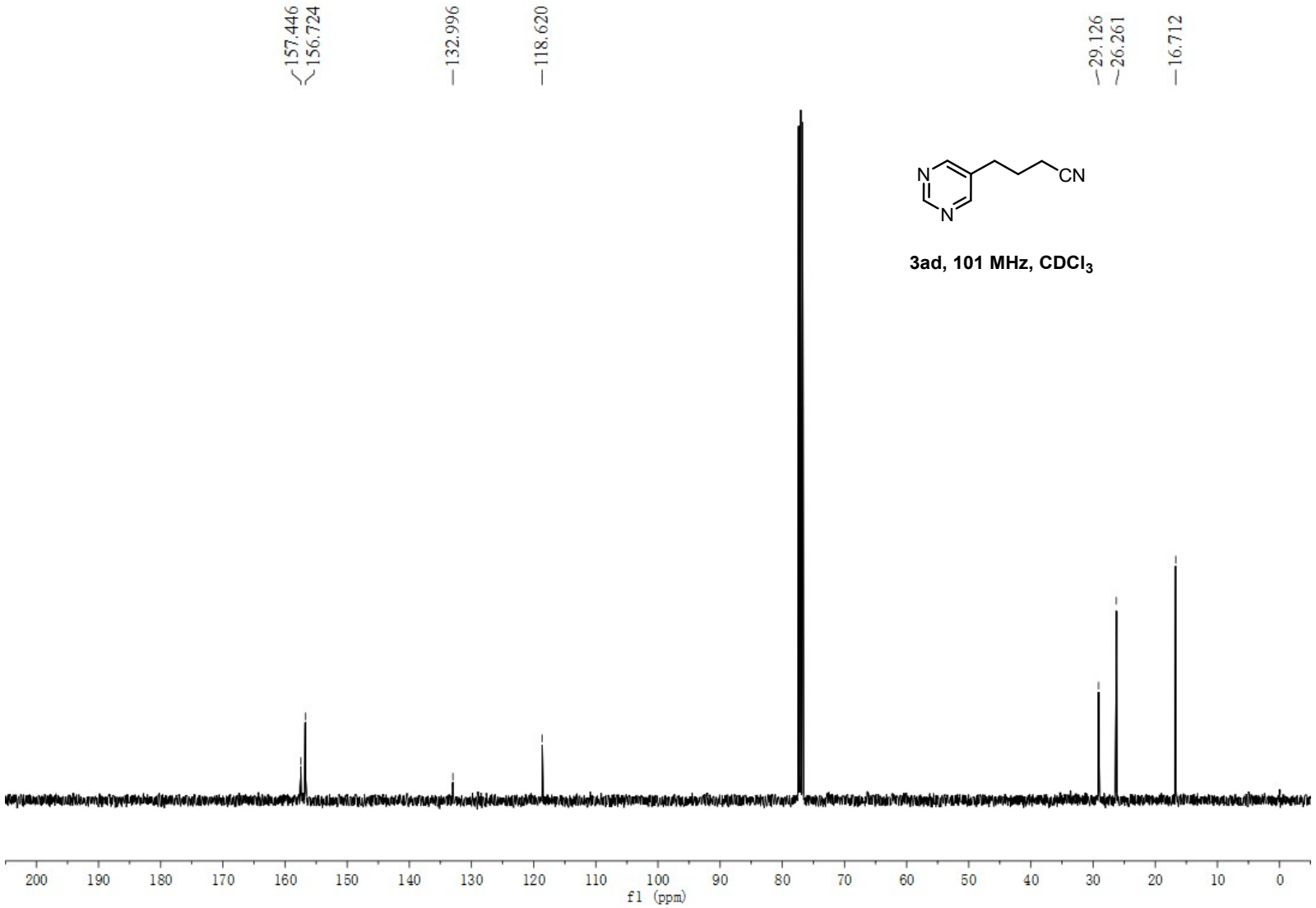
3ac, 400 MHz, $CDCl_3$

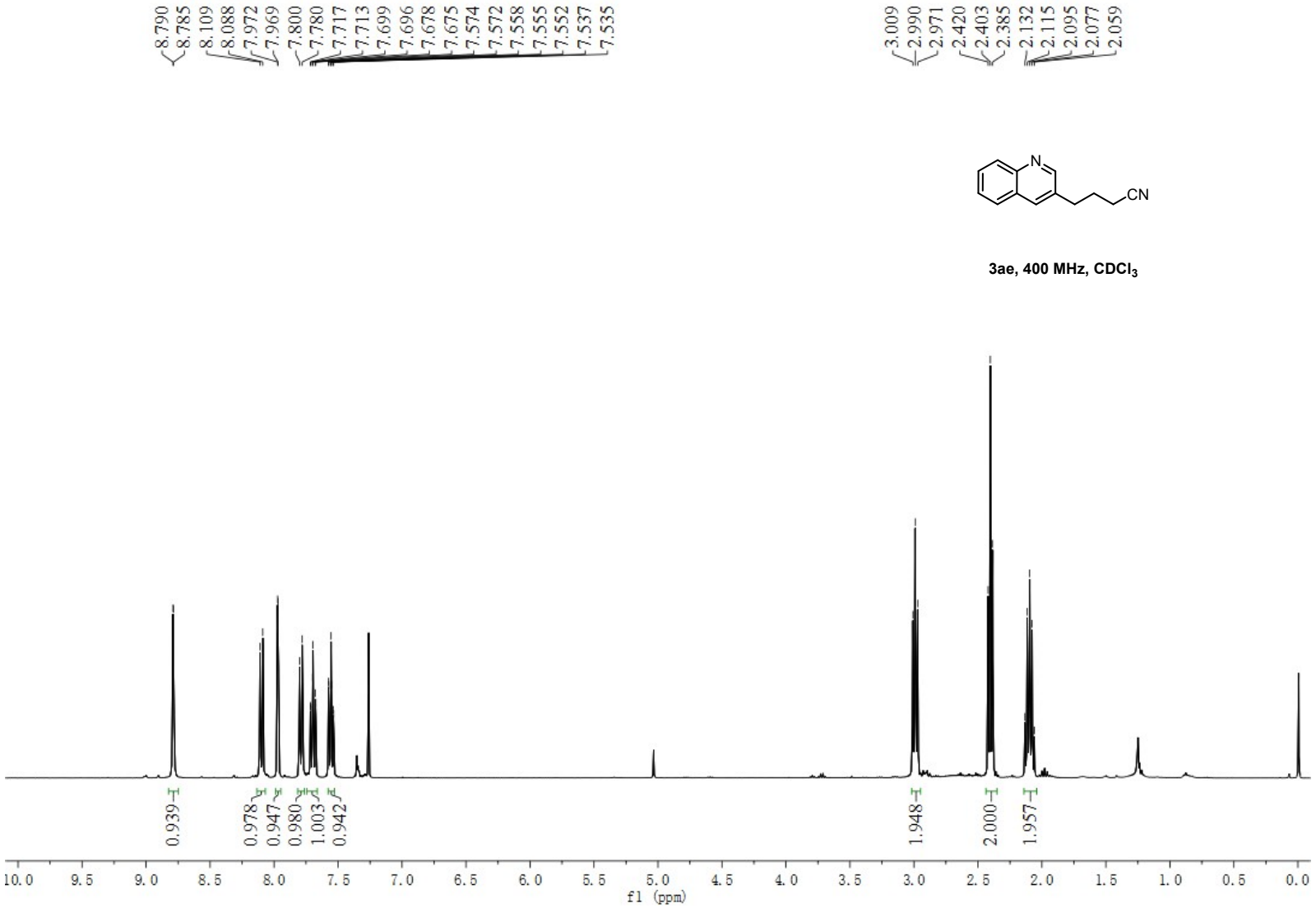


3ac, 101 MHz, $CDCl_3$

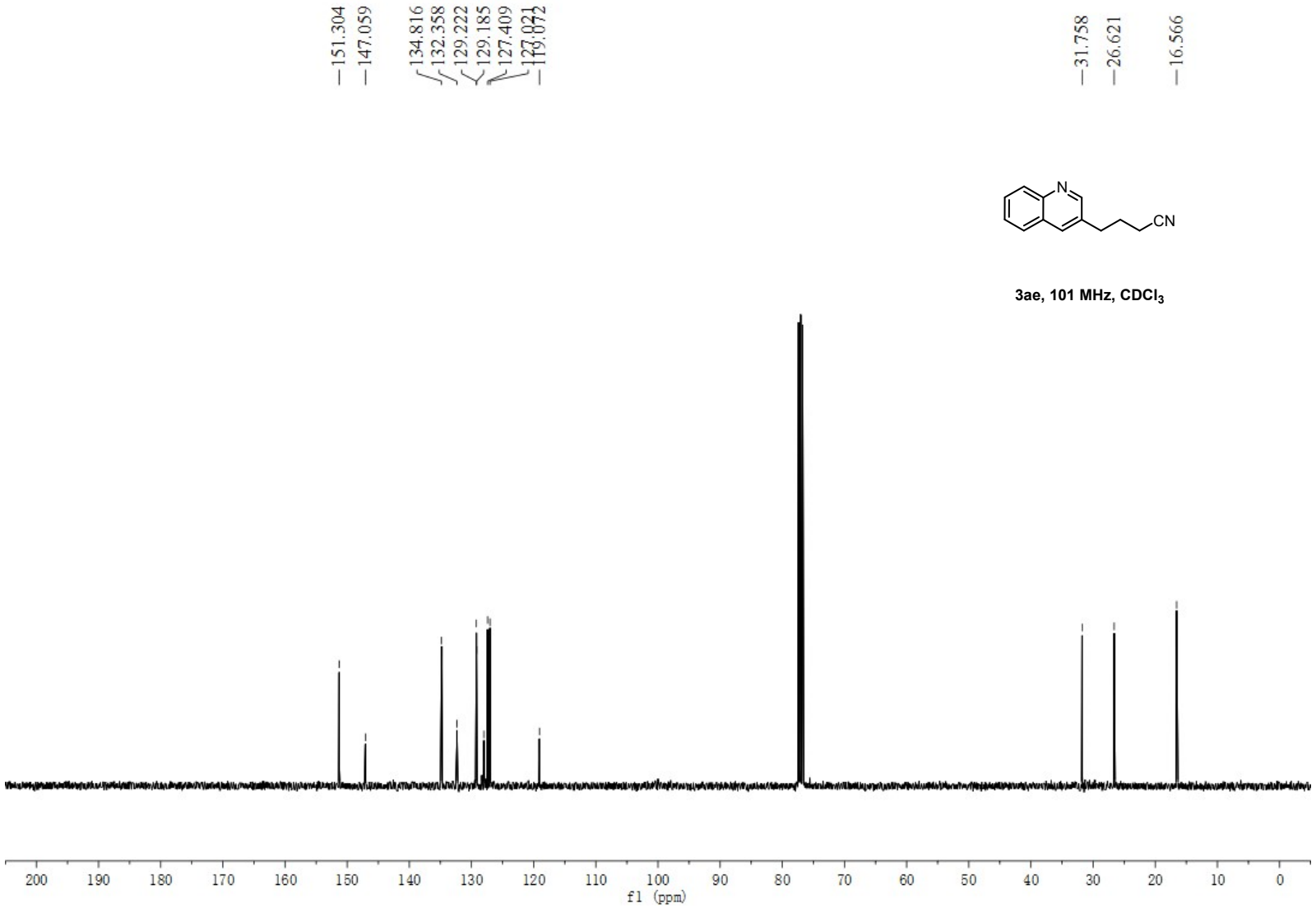


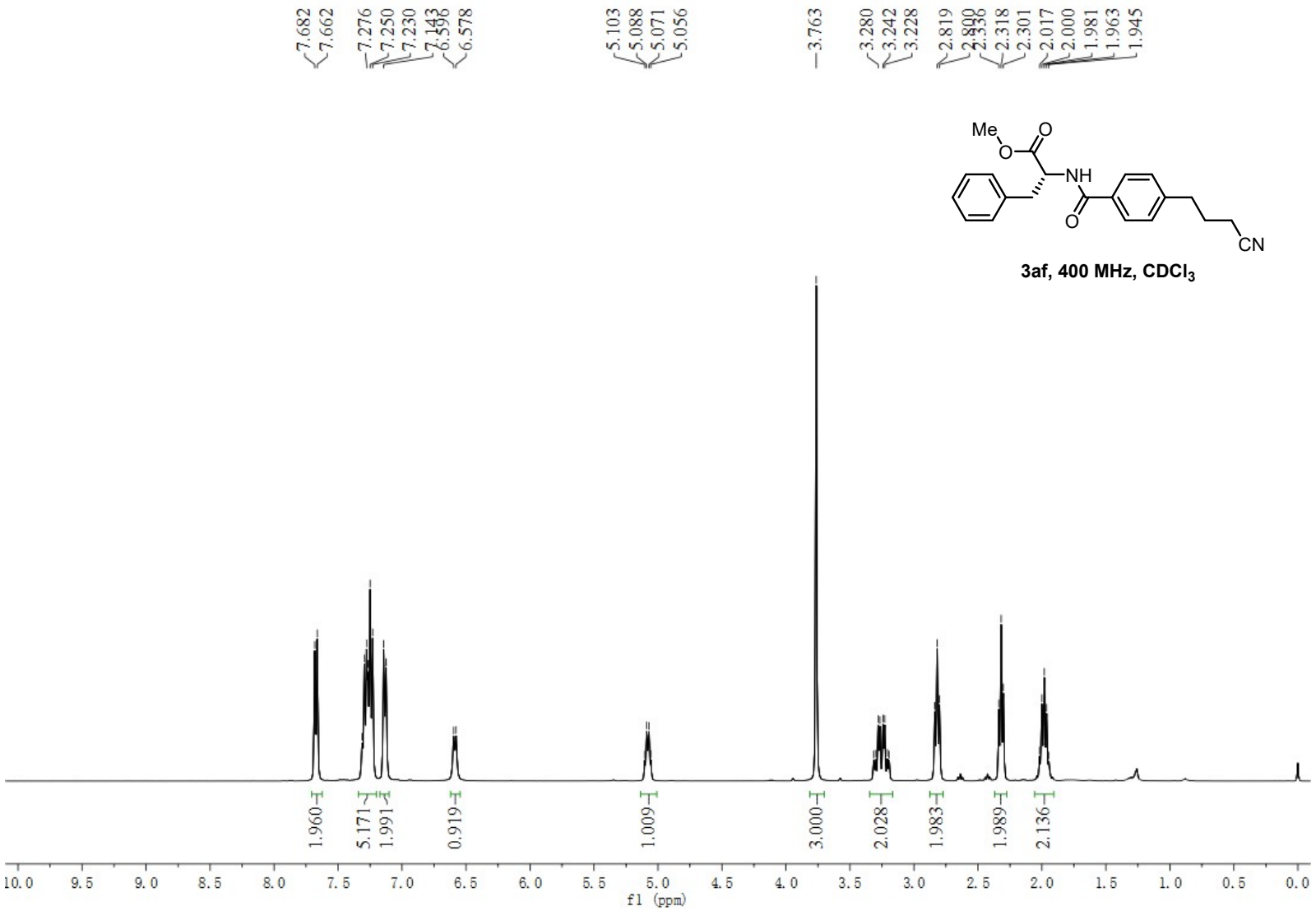
3ad, 400 MHz, $CDCl_3$


157.446
156.724
132.996
118.620
29.126
26.261
16.712
N
N
CN
3ad, 101 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

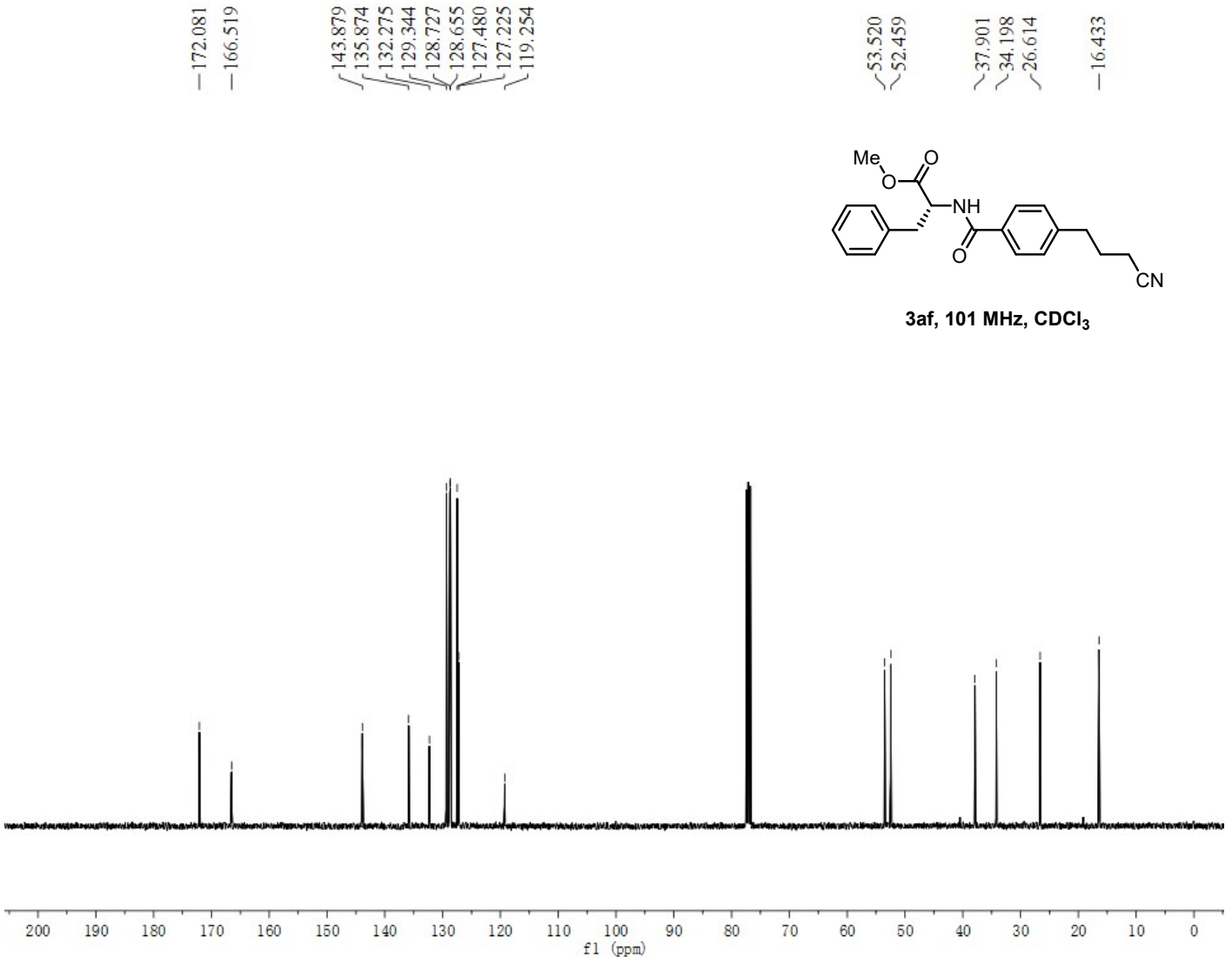


3ae, 400 MHz, $CDCl_3$

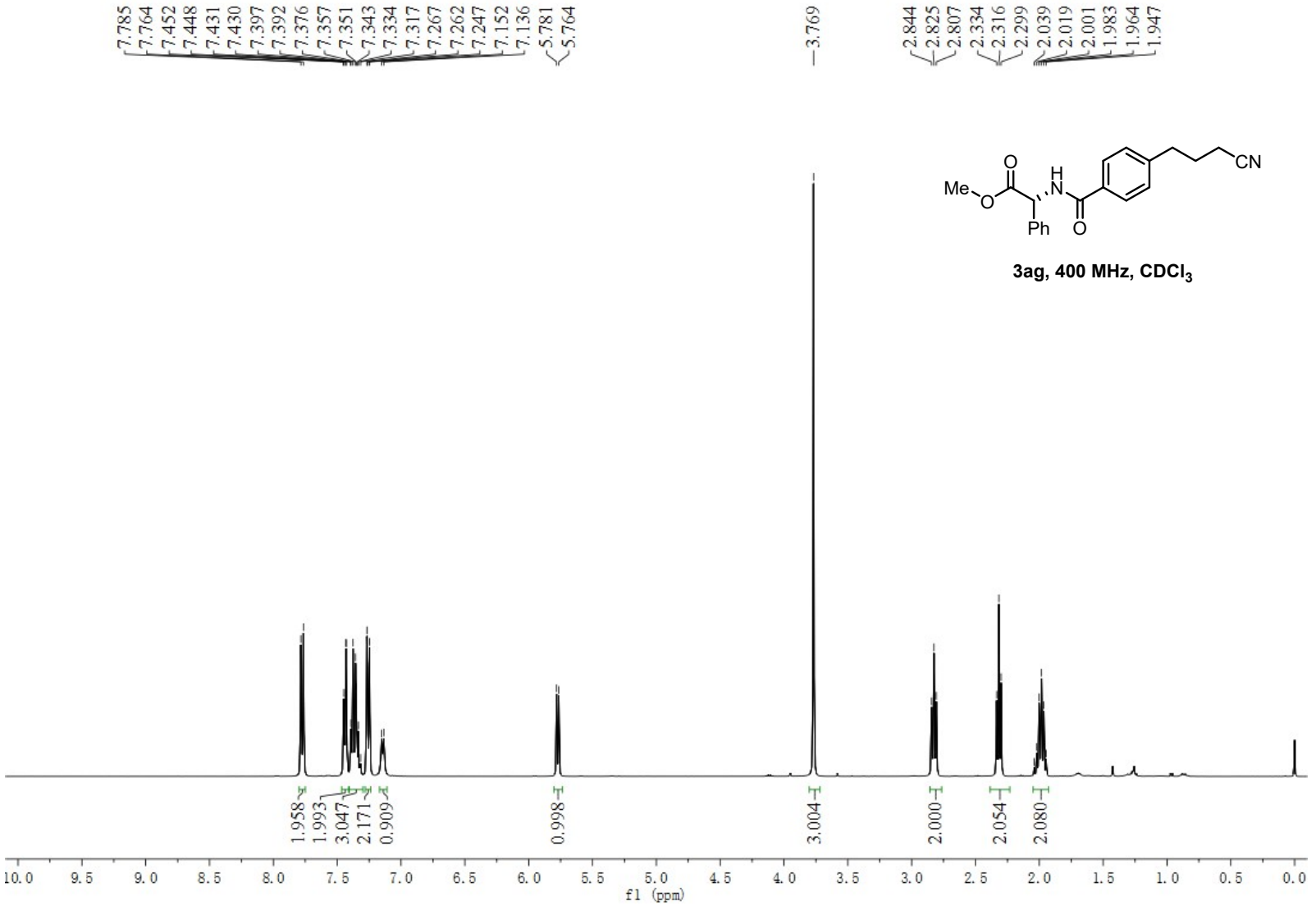

151.304
147.059
134.816
132.358
129.222
129.185
127.409
127.021
119.872
31.758
26.621
16.566
N
CN
3ae, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



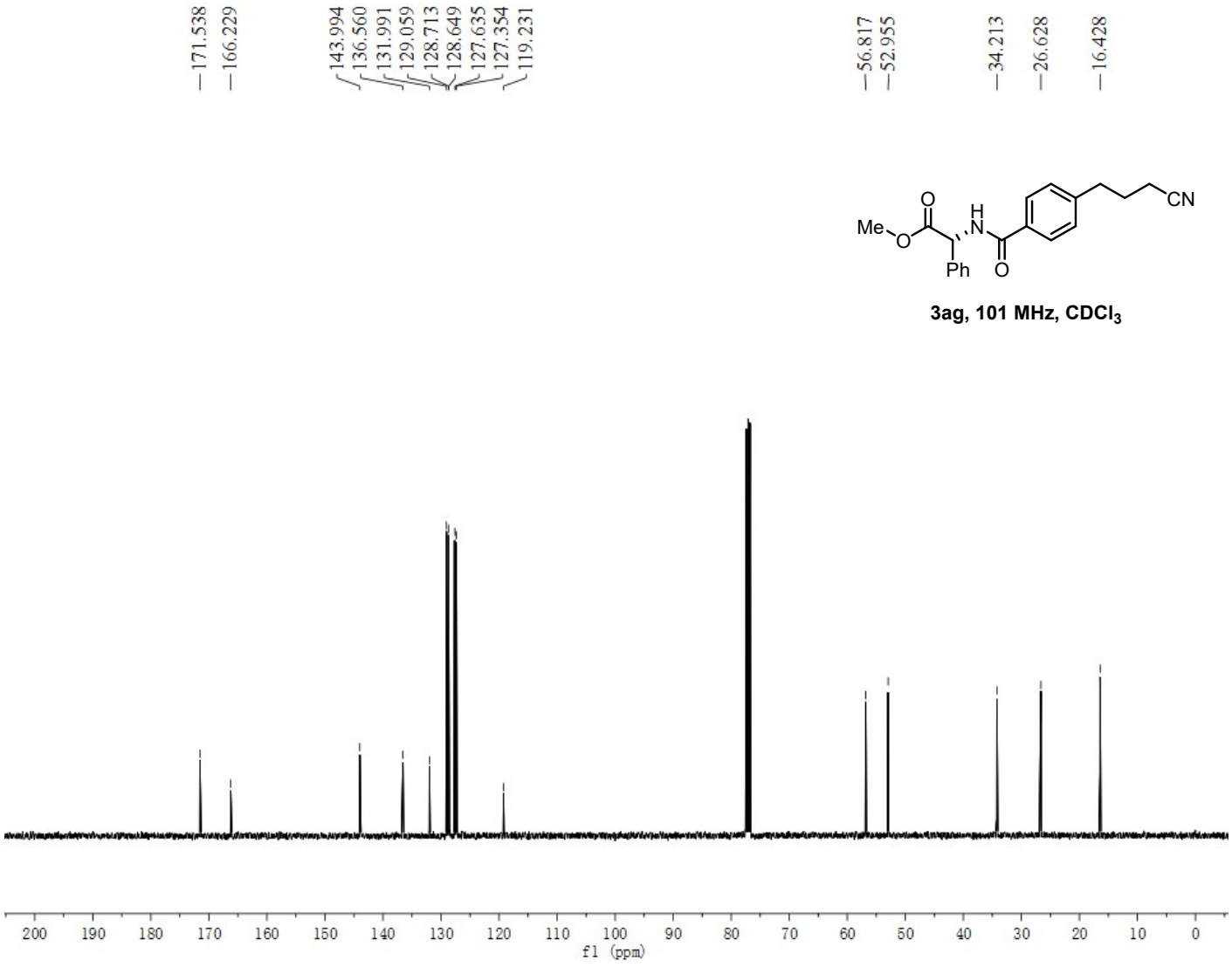
3af, 400 MHz, $CDCl_3$



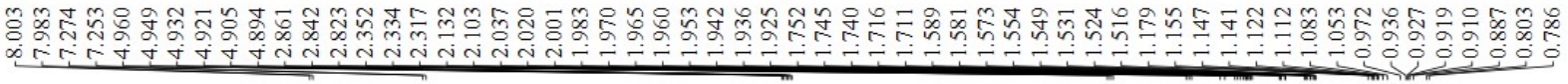
3af, 101 MHz, $CDCl_3$

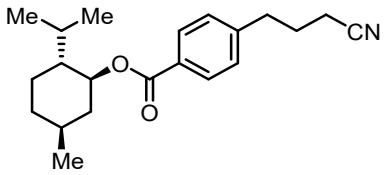


3ag, 400 MHz, $CDCl_3$

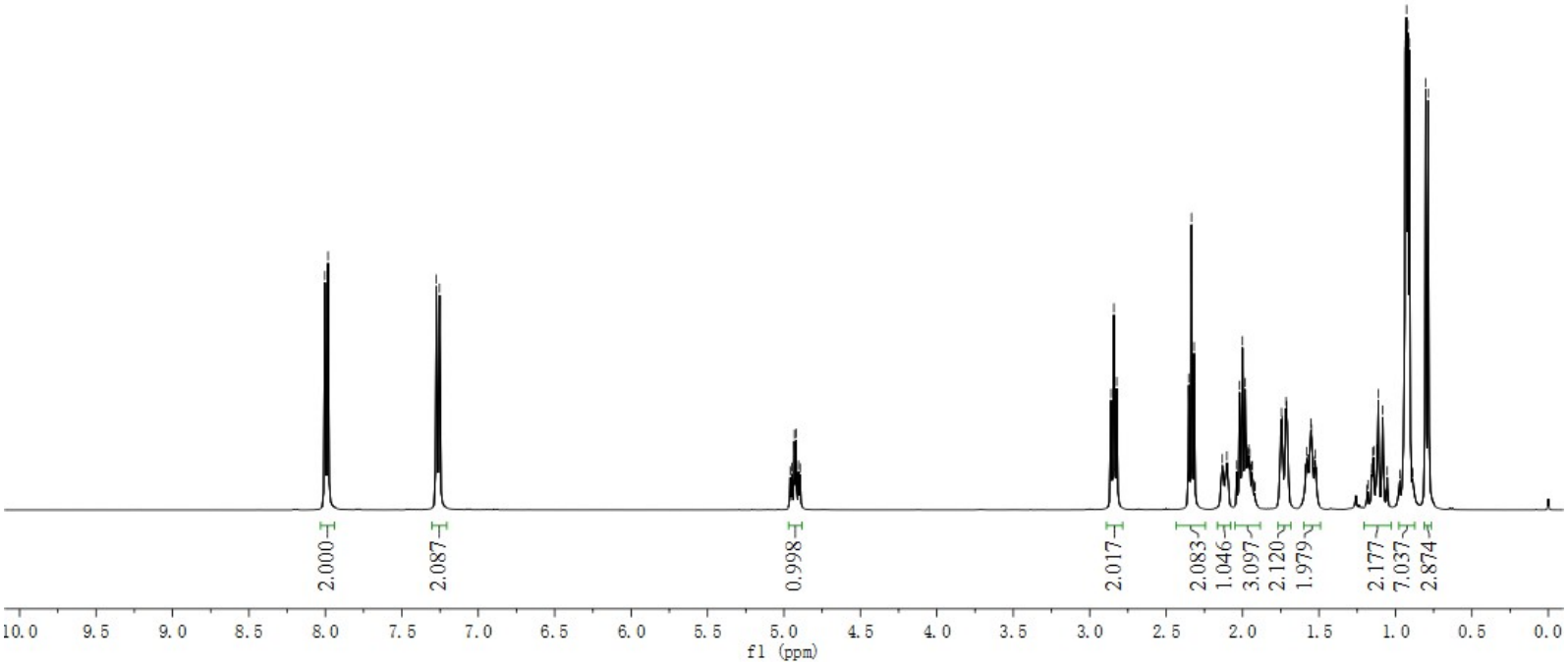


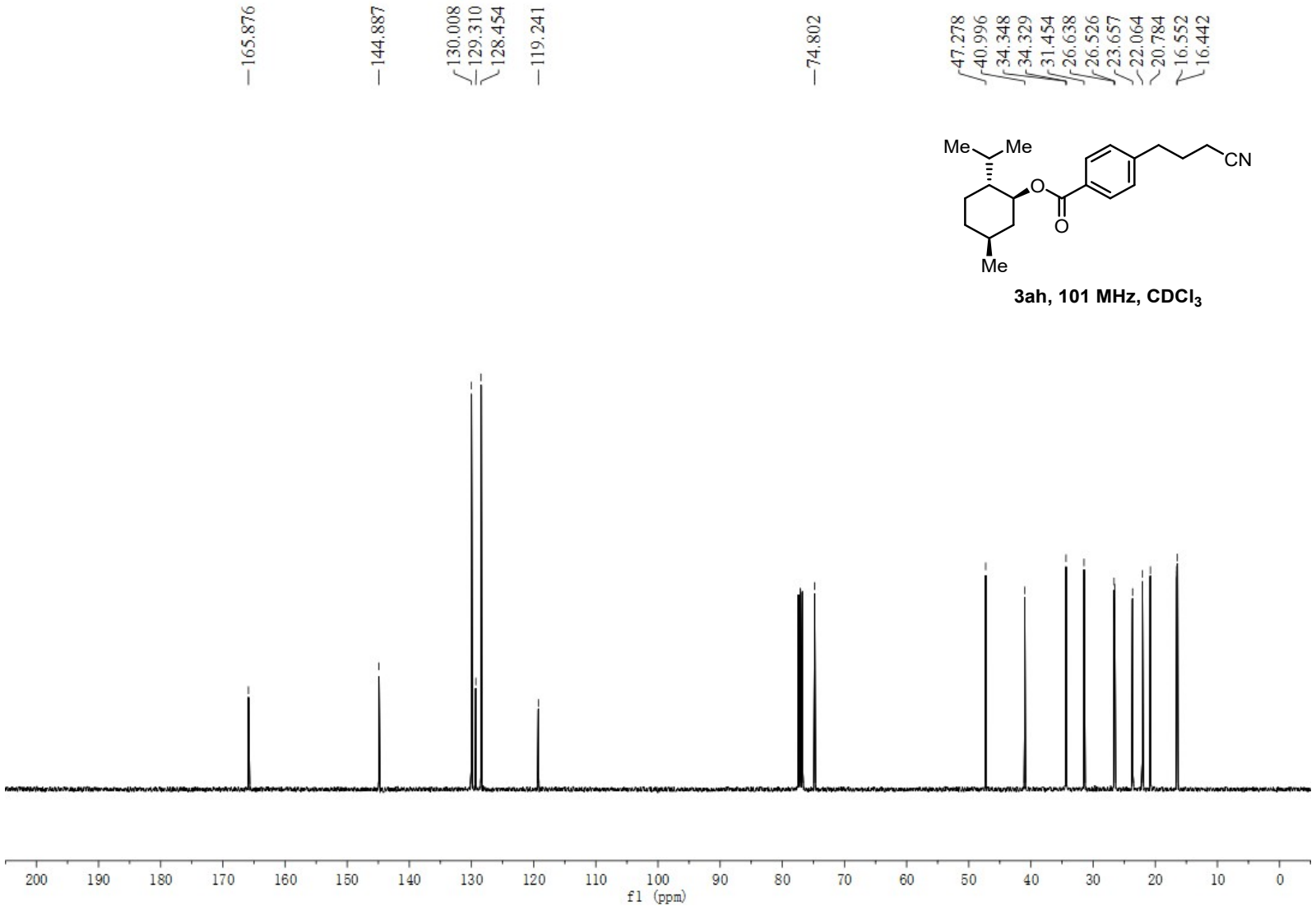
3ag, 101 MHz, $CDCl_3$



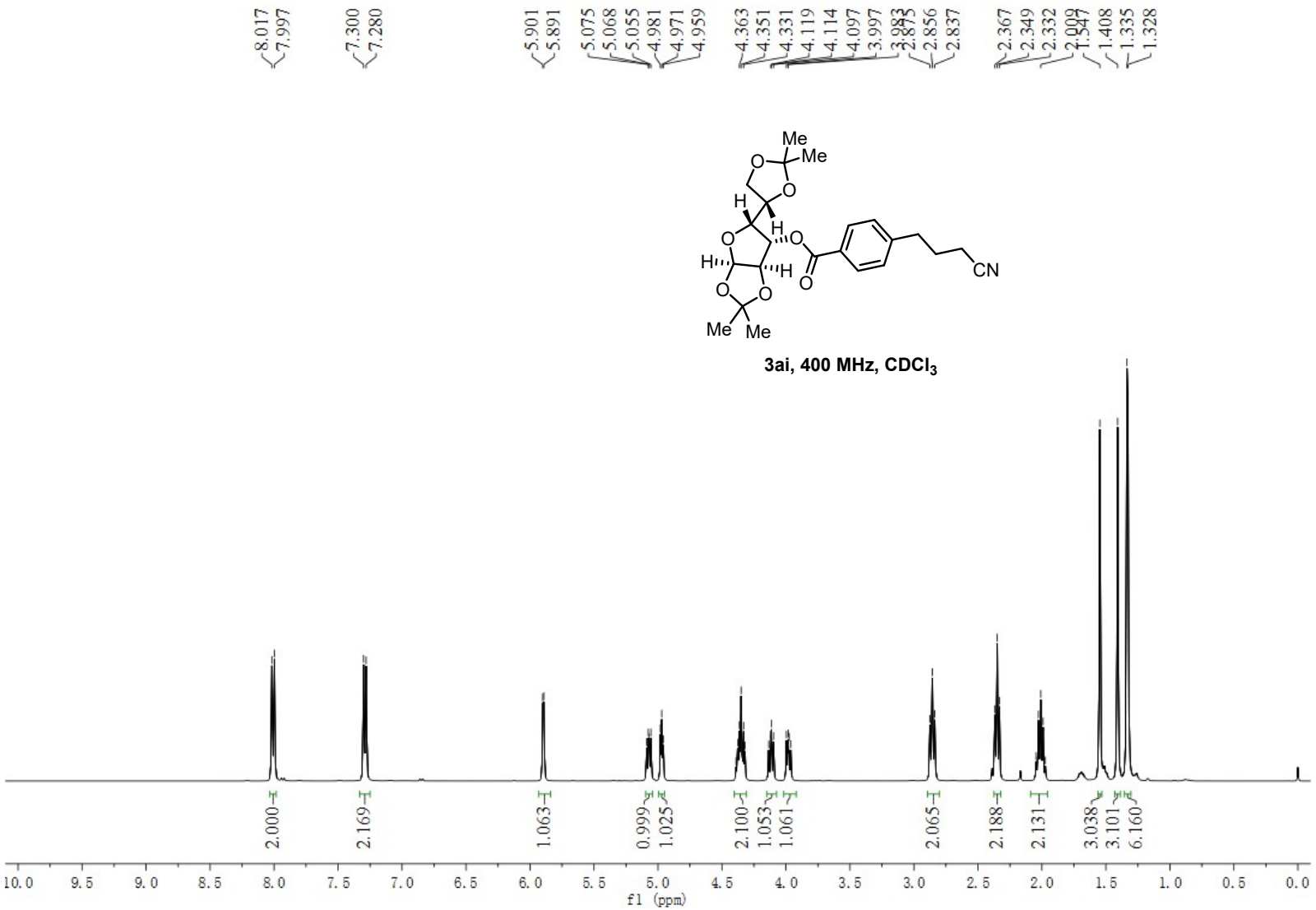


3ah, 400 MHz, $CDCl_3$

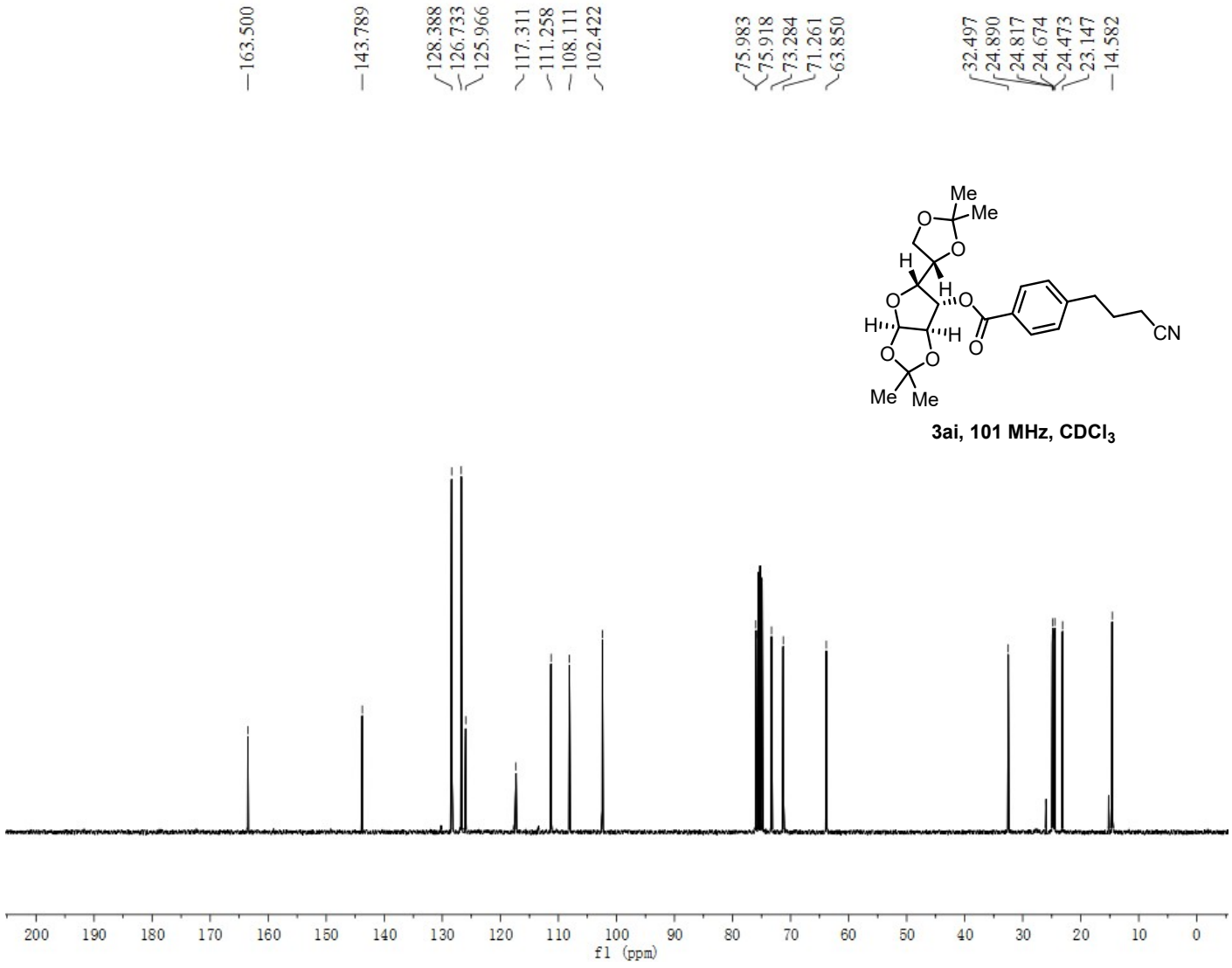




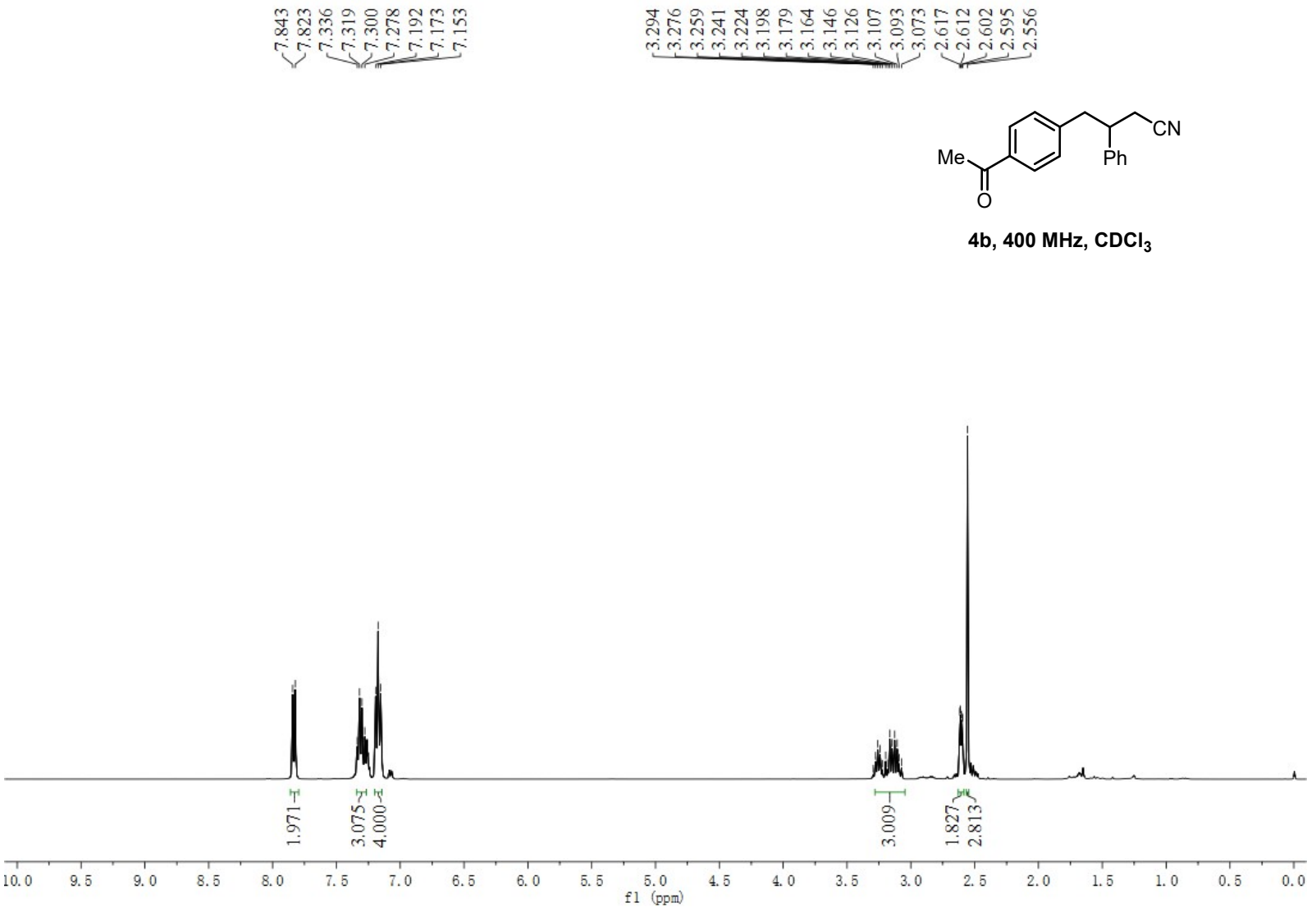
3ah, 101 MHz, $CDCl_3$



3ai, 400 MHz, $CDCl_3$

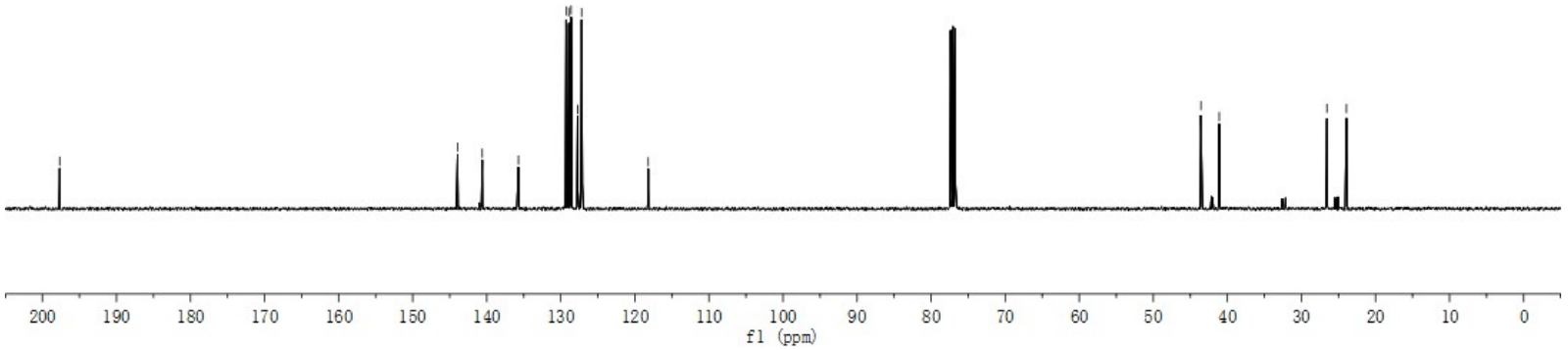


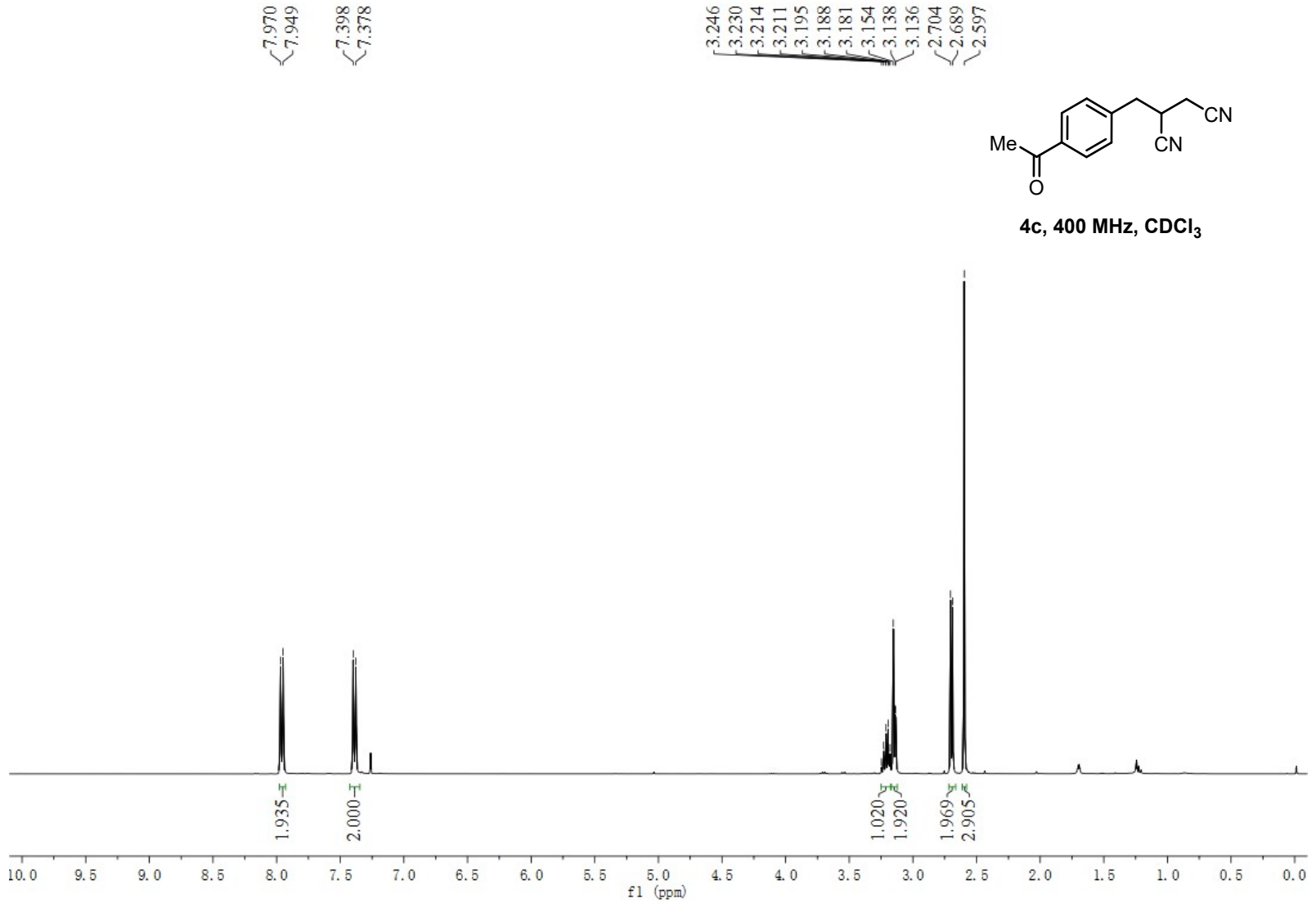
3ai, 101 MHz, $CDCl_3$



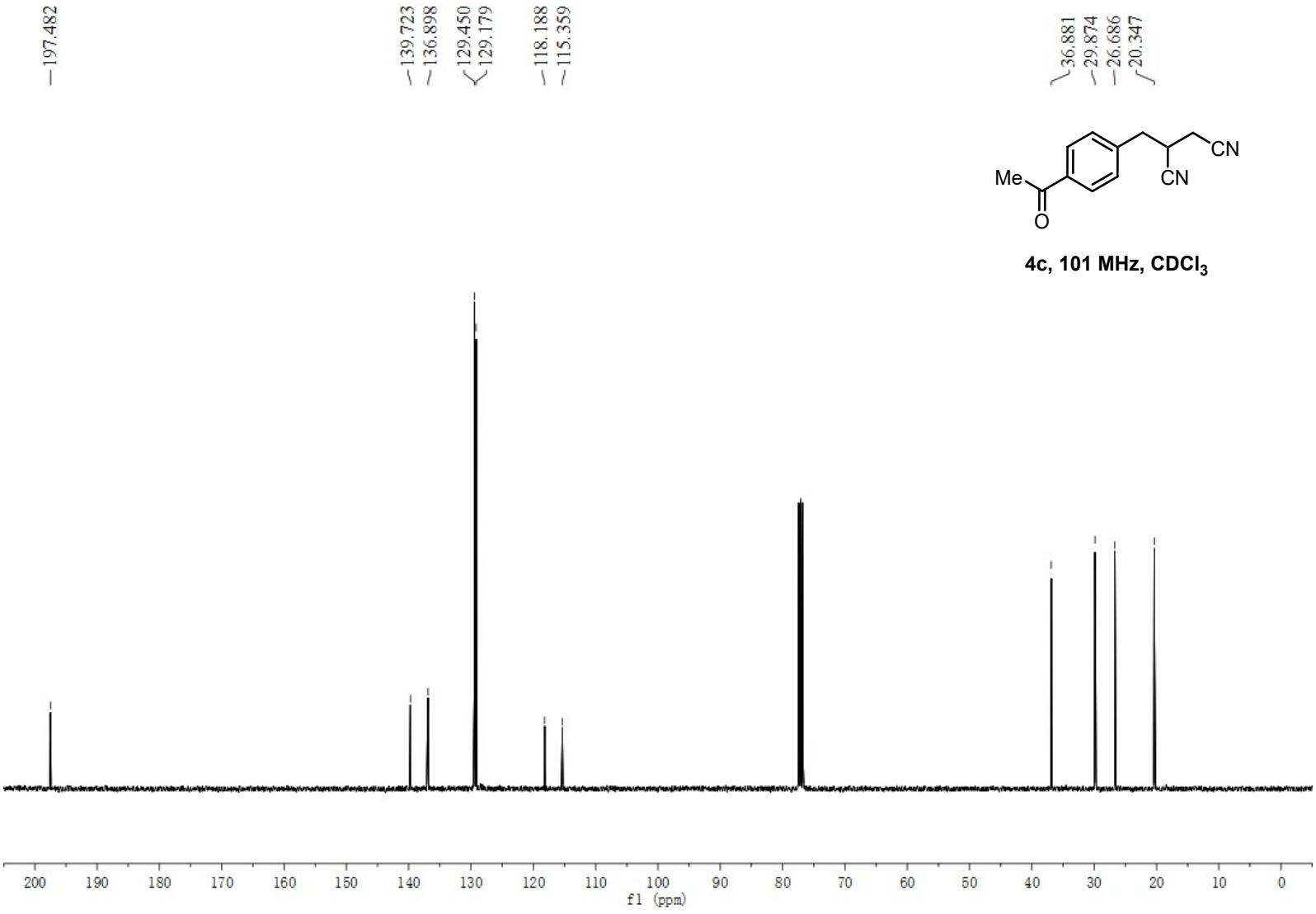
4b, 400 MHz, $CDCl_3$

4b, 101 MHz, $CDCl_3$

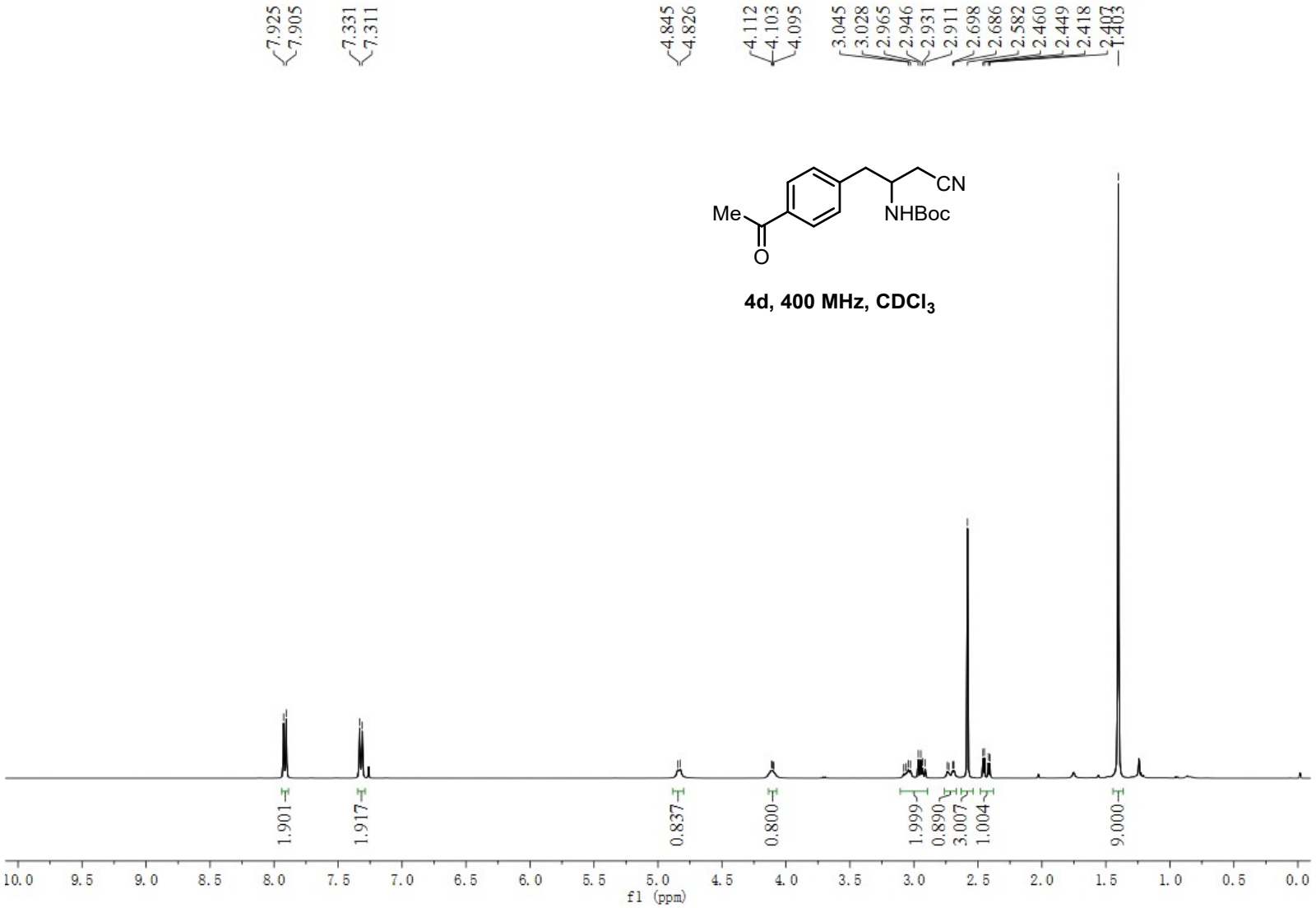




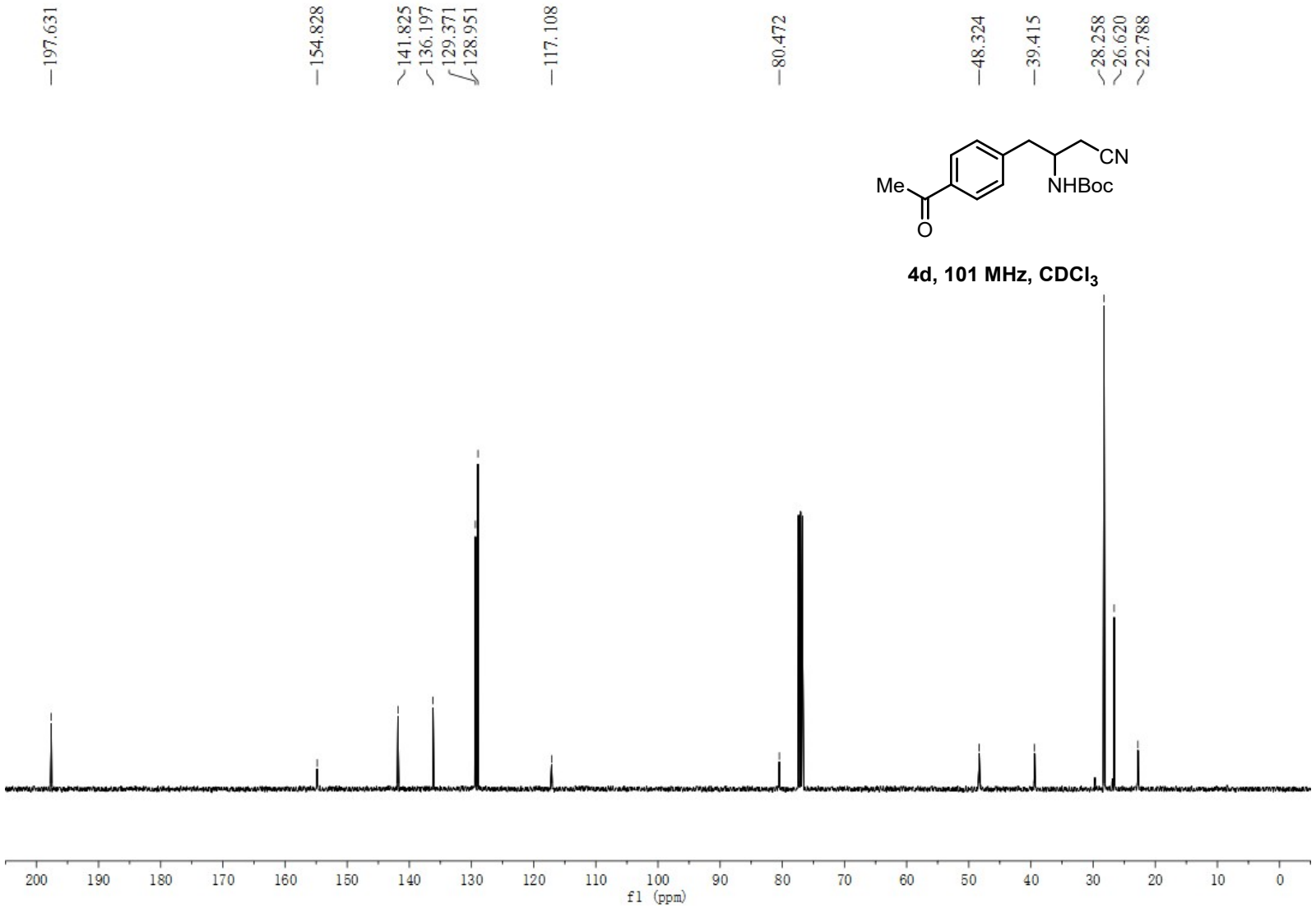
4c, 400 MHz, $CDCl_3$

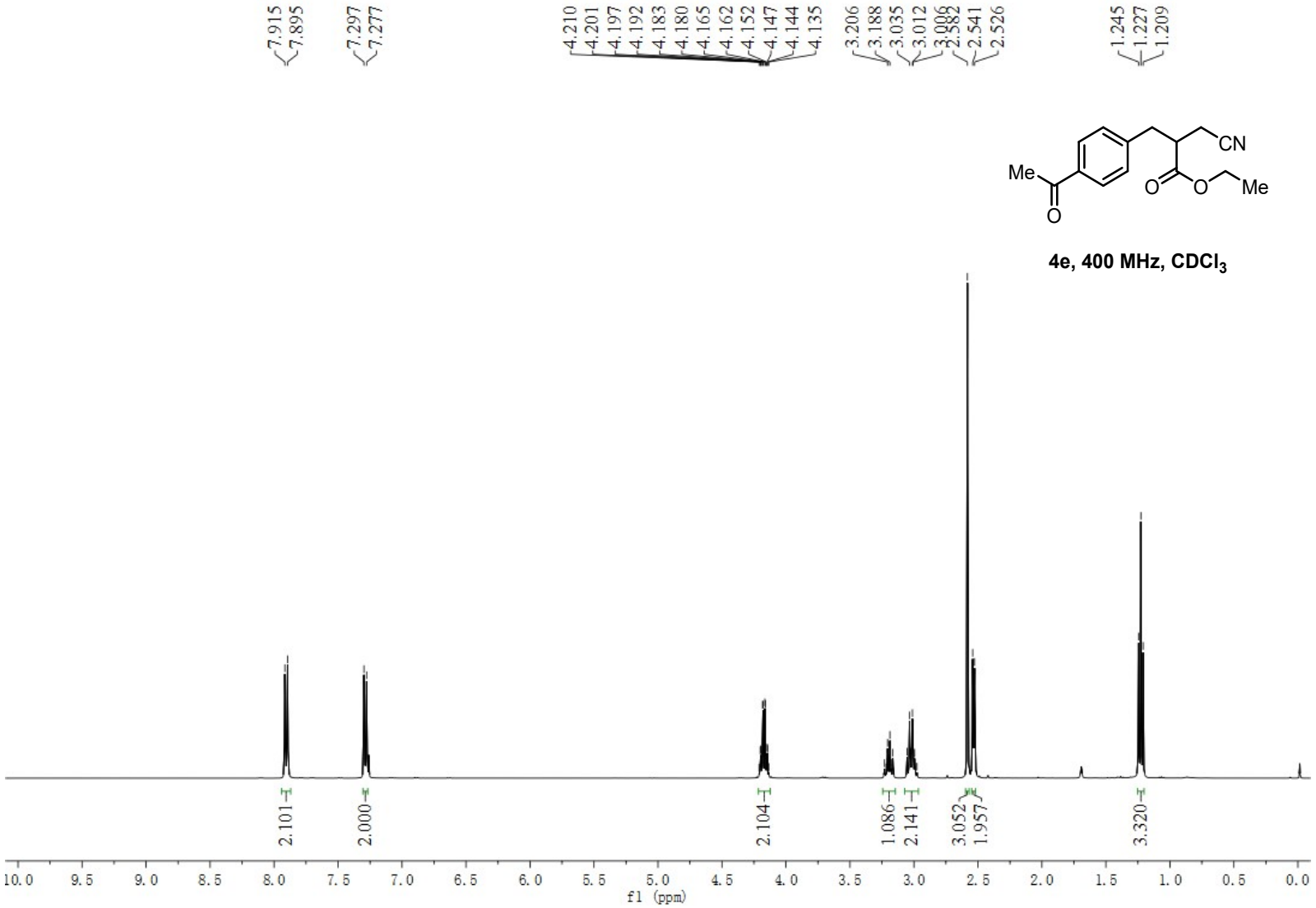


4c, 101 MHz, $CDCl_3$

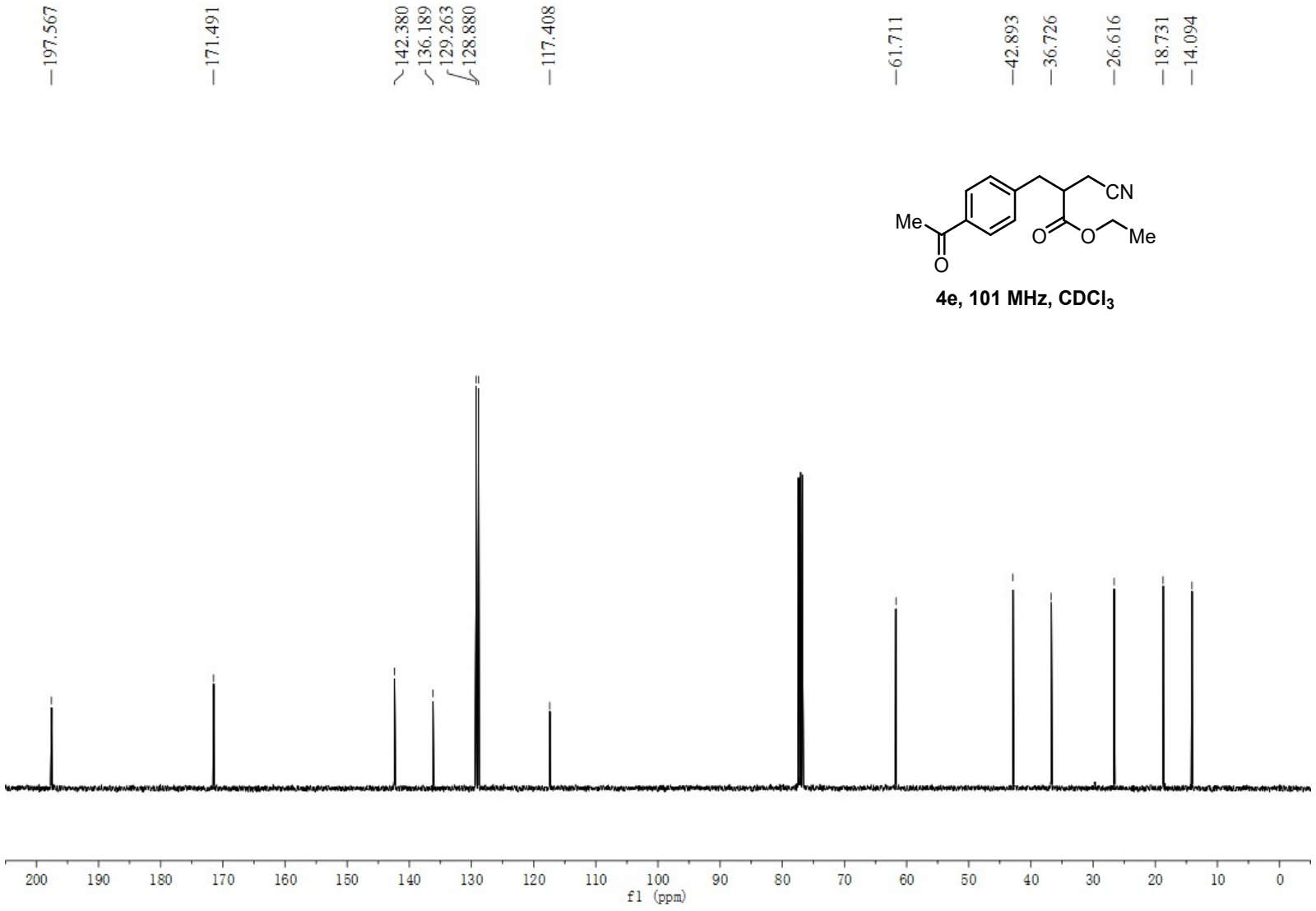


4d, 400 MHz, $CDCl_3$

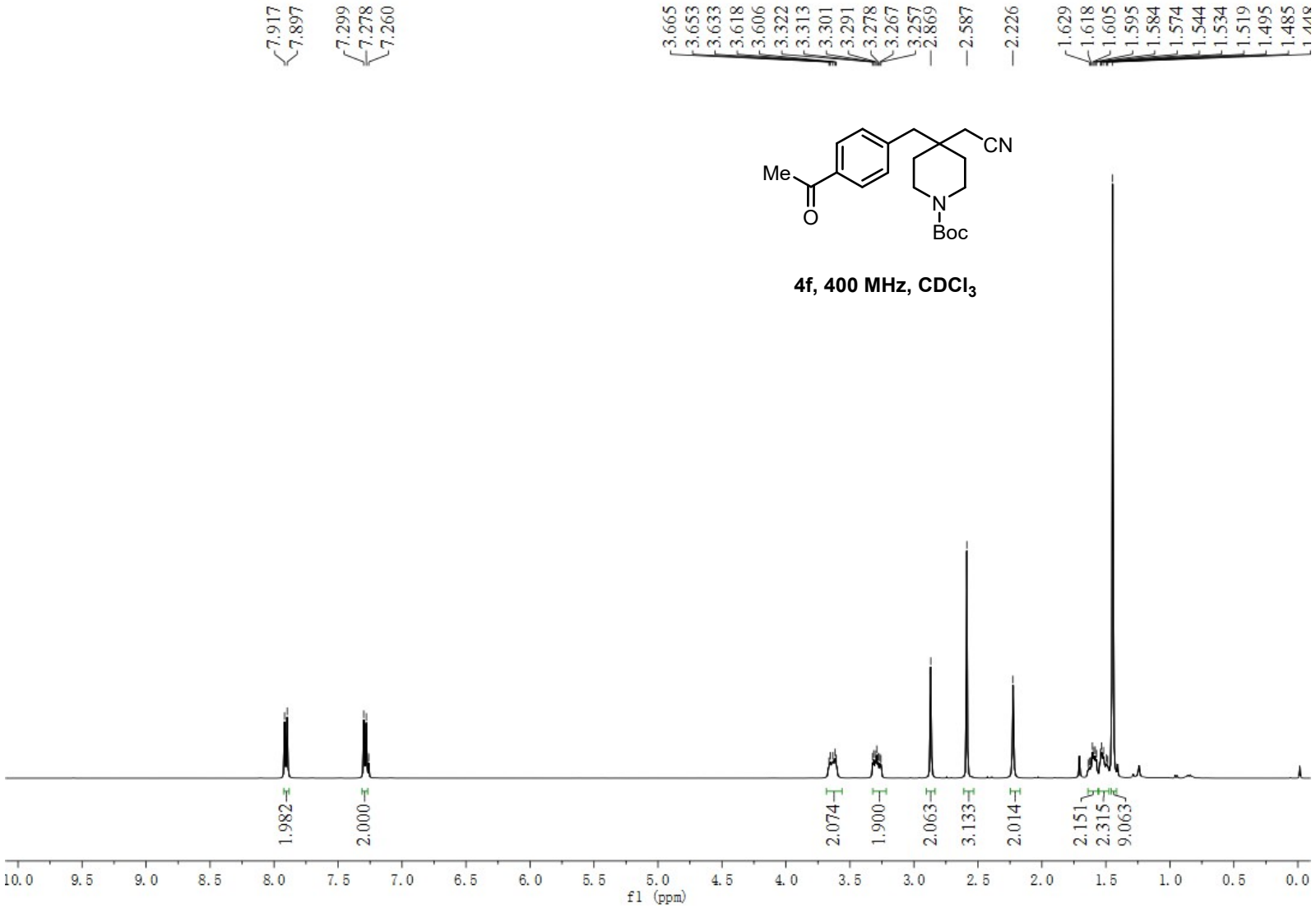

197.631
154.828
141.825
136.197
129.371
128.951
117.108
80.472
48.324
39.415
28.258
26.620
22.788
Me
O
NHBoc
CN
4d, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



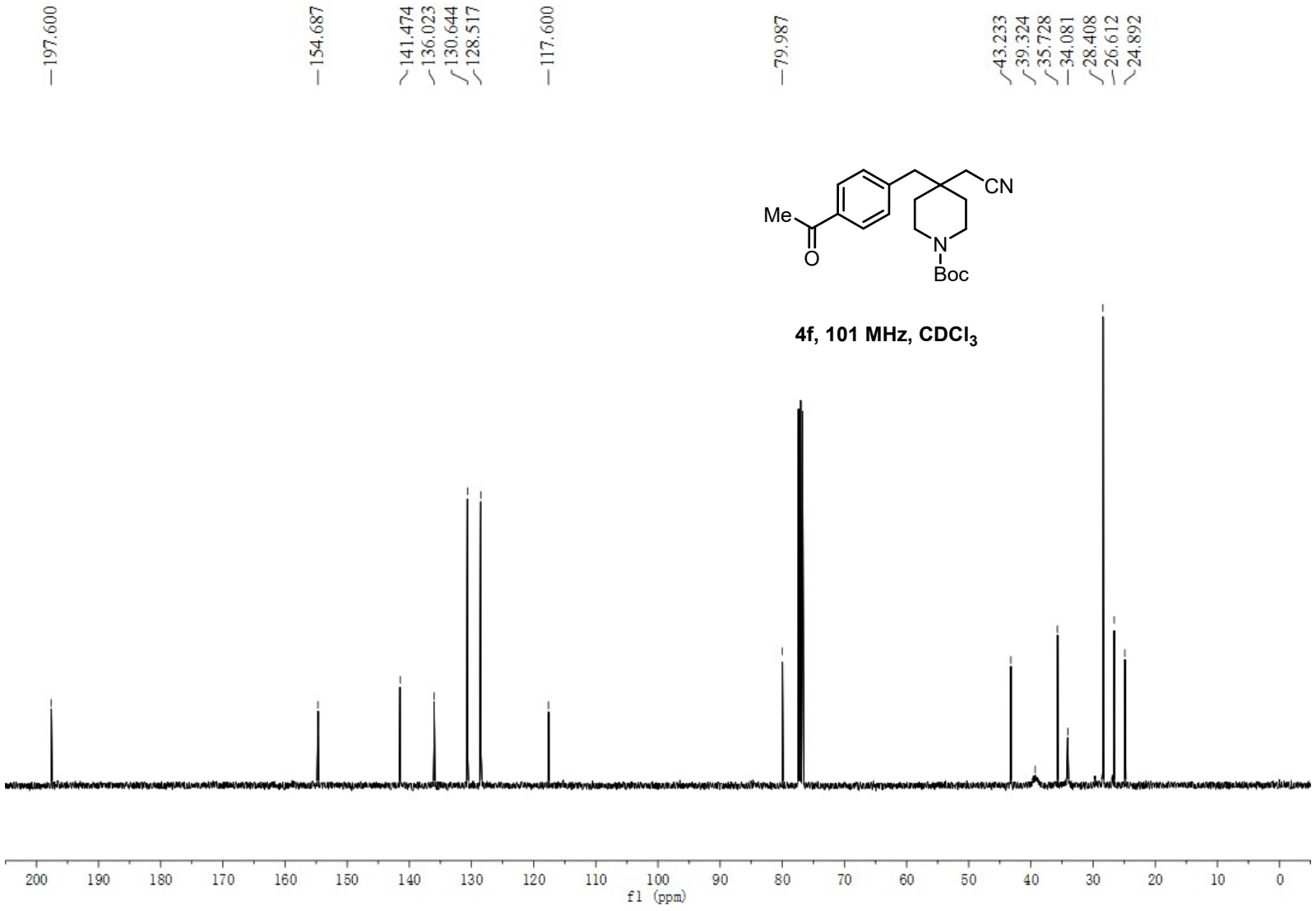
4e, 400 MHz, $CDCl_3$

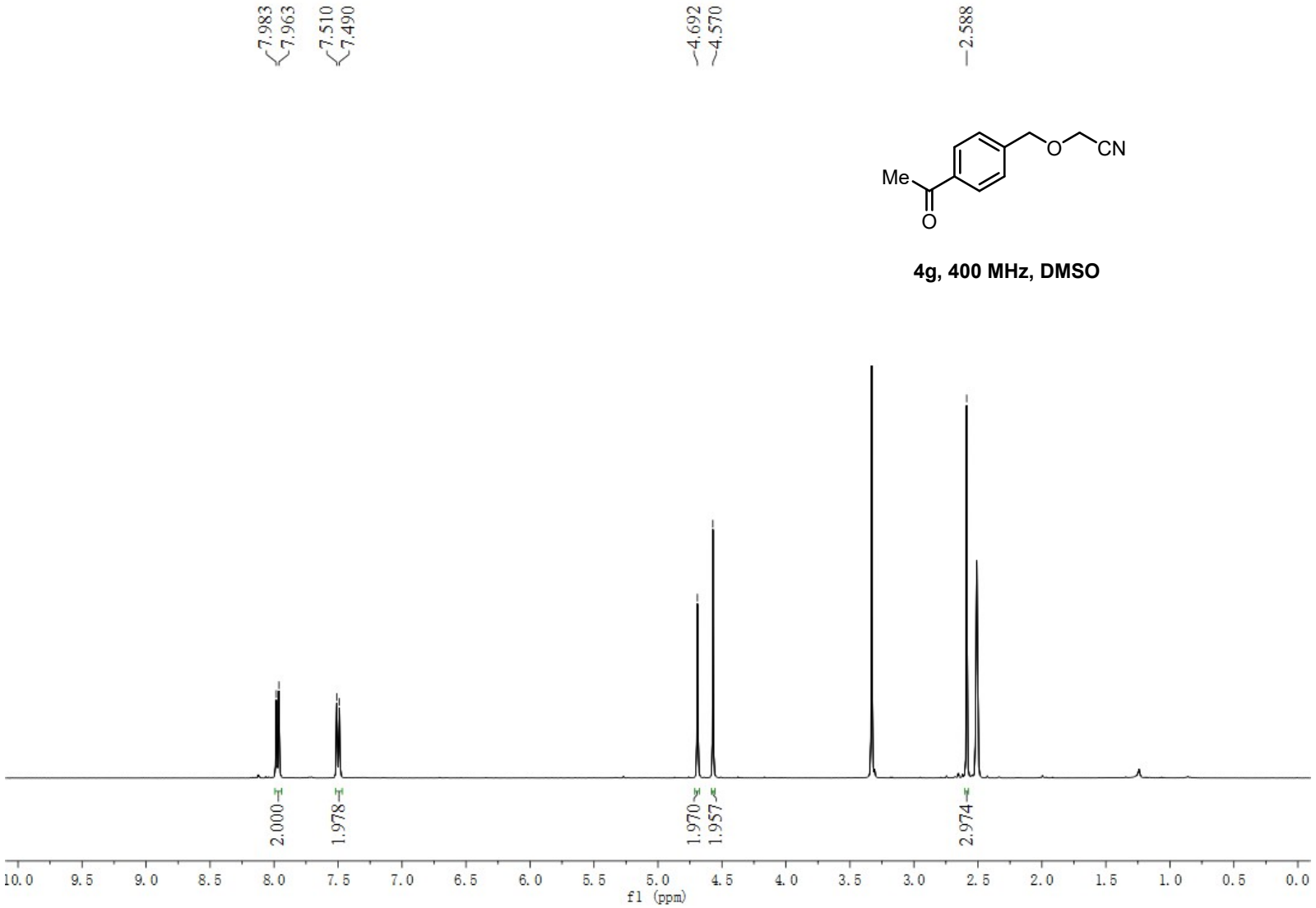


4e, 101 MHz, $CDCl_3$

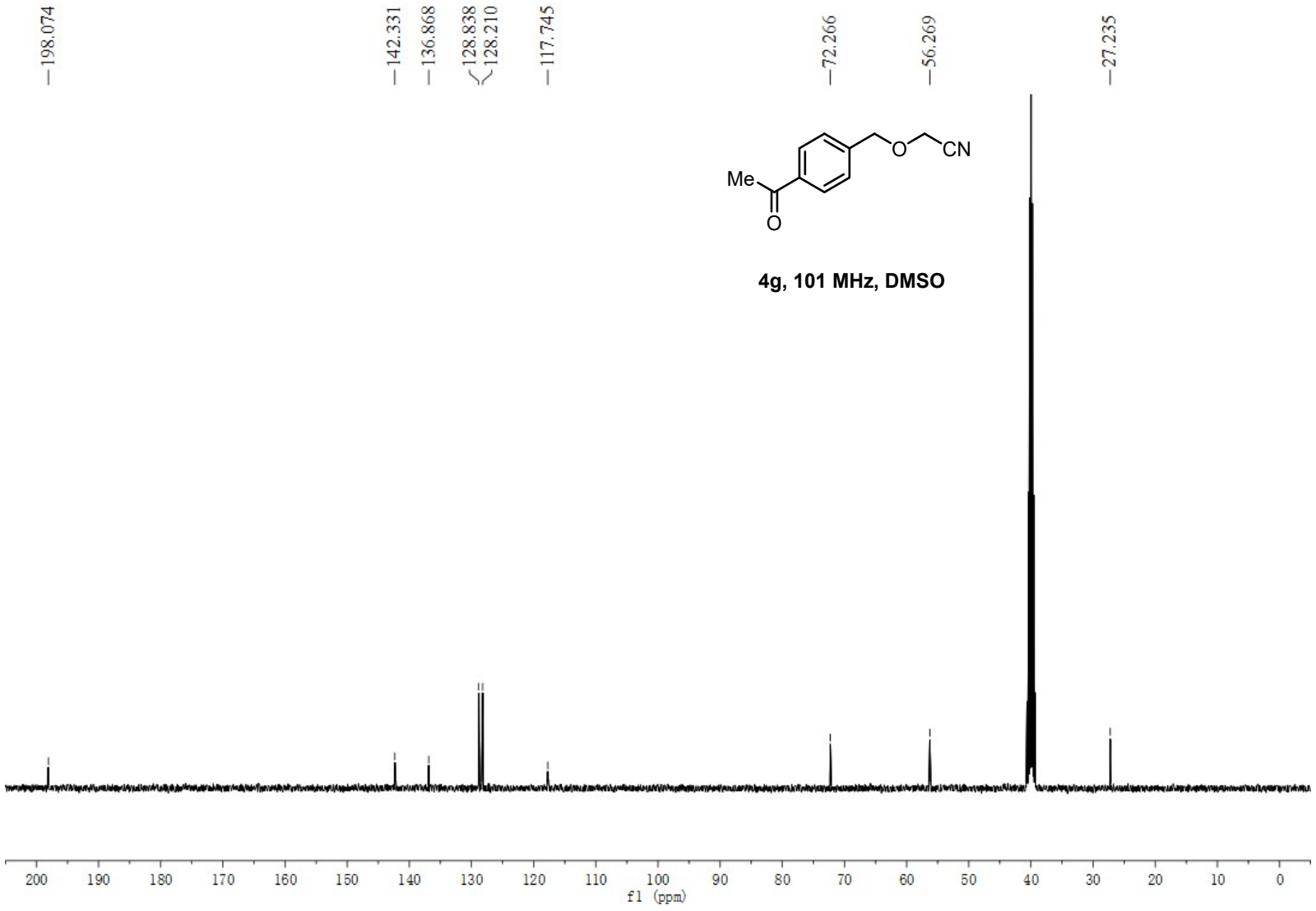


4f, 400 MHz, $CDCl_3$

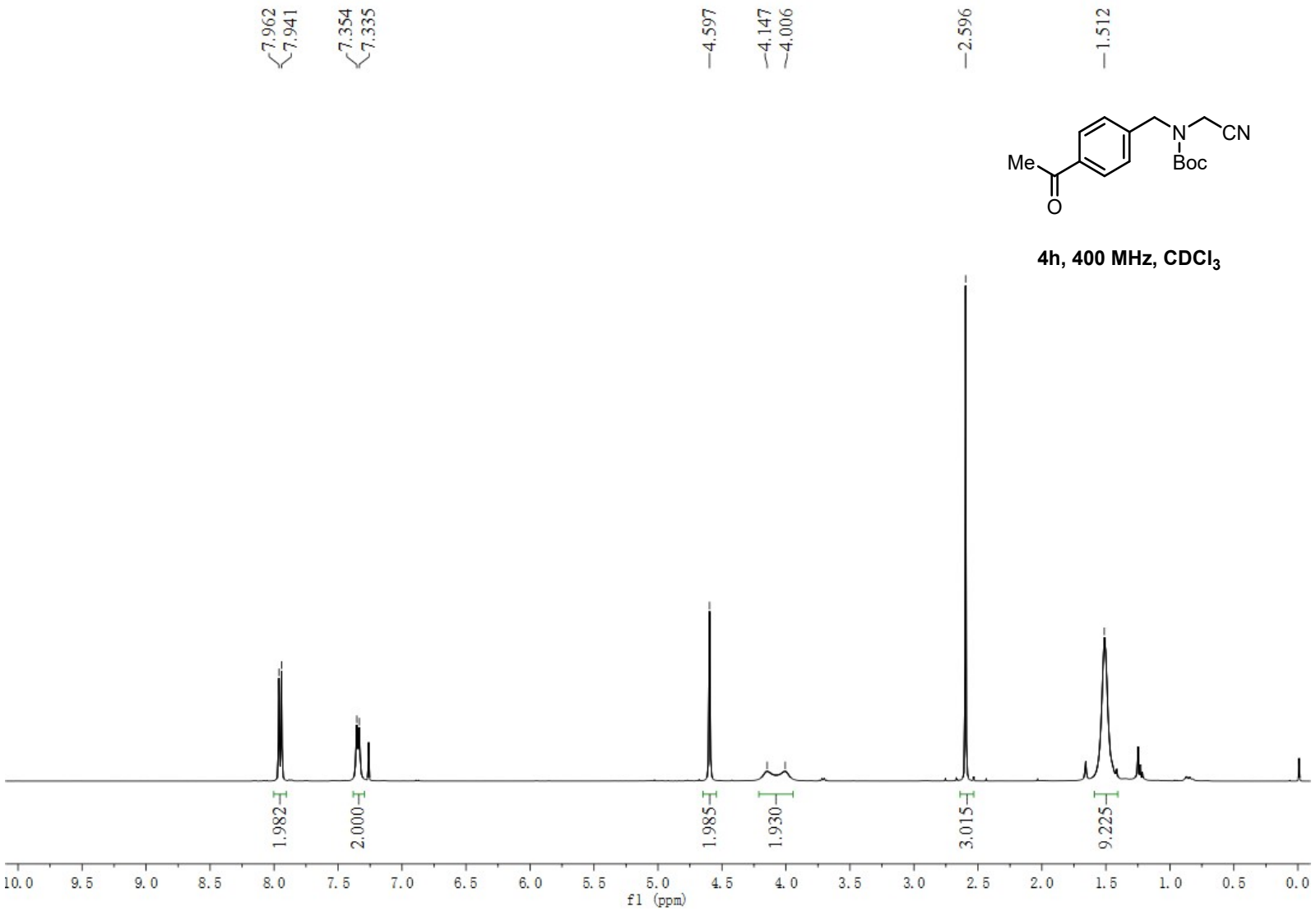

197.600
154.687
141.474
136.023
130.644
128.517
117.600
79.987
43.233
39.324
35.728
34.081
28.408
26.612
24.892
Me
O
N
Boc
CN
4f, 101 MHz, CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

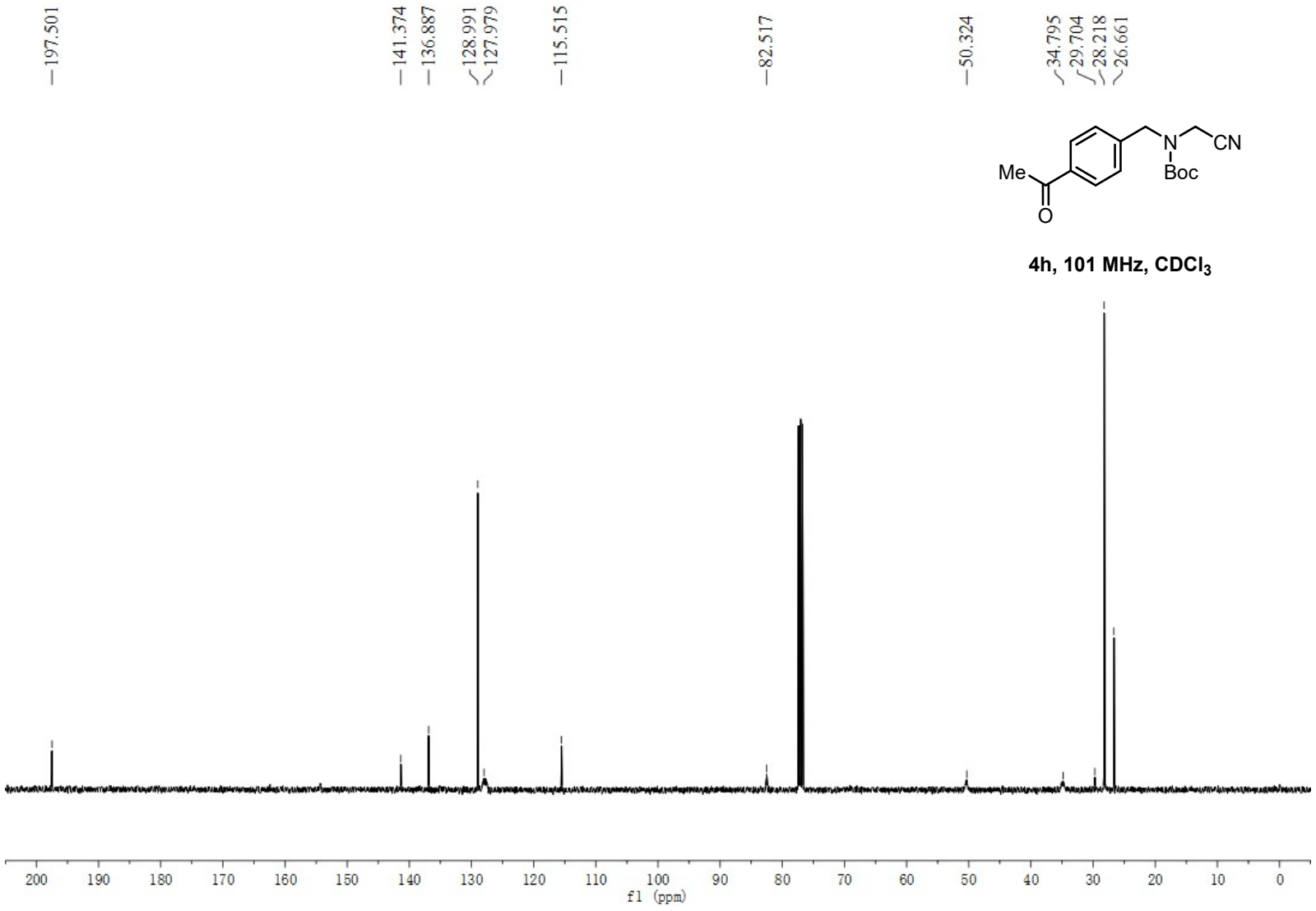


4g, 400 MHz, DMSO

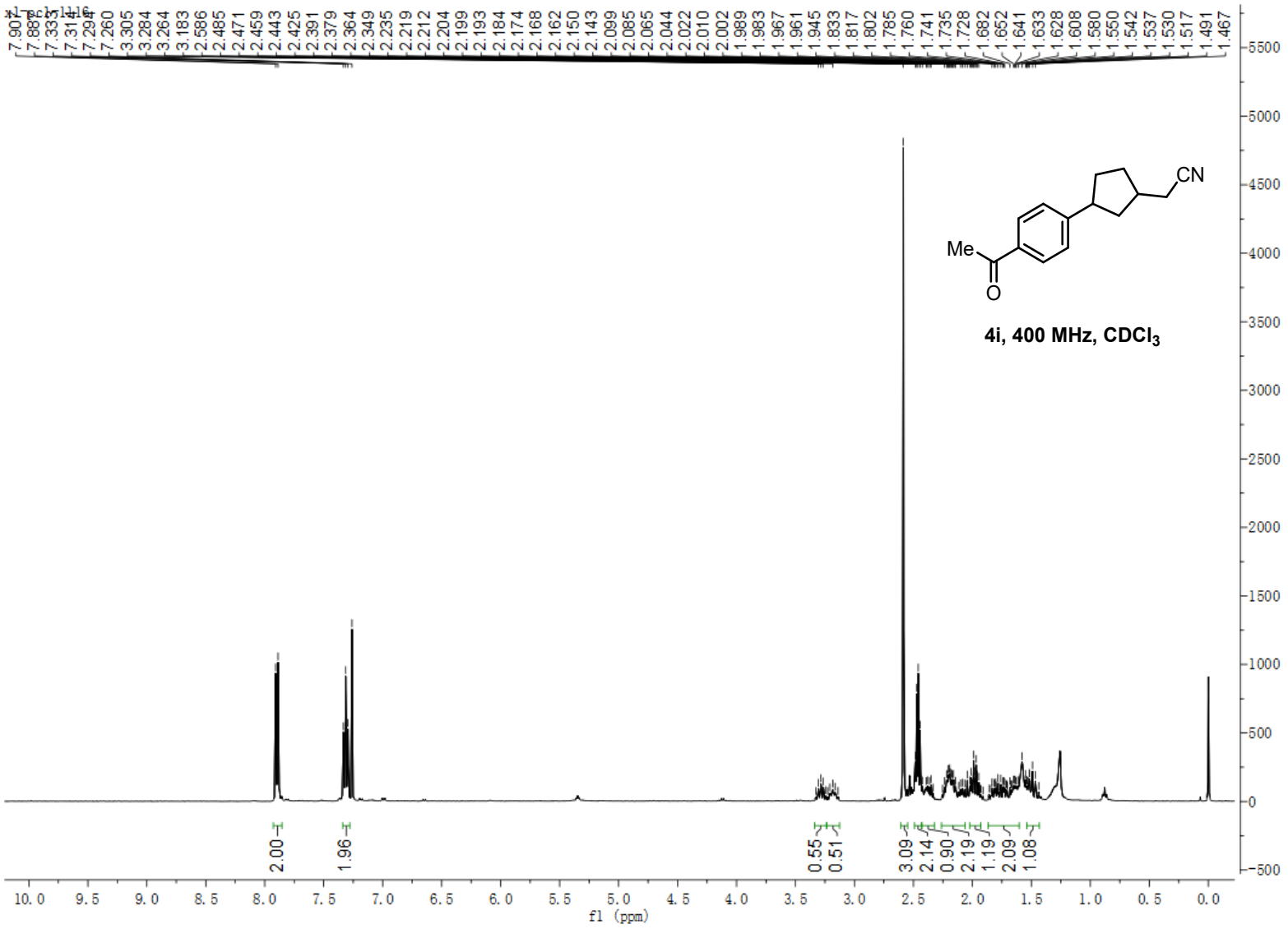


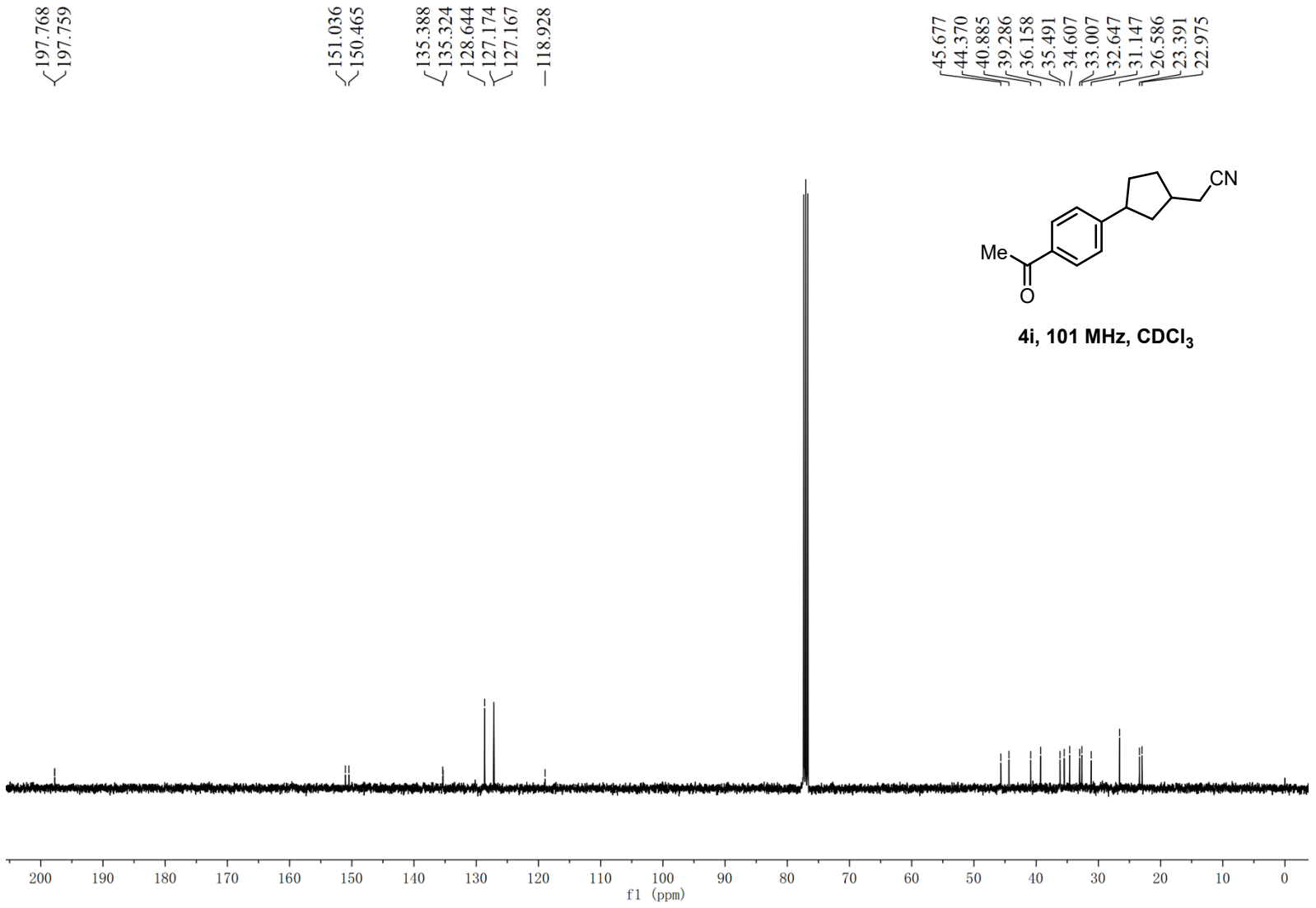
4g, 101 MHz, DMSO


7.962
7.941
7.354
7.335
4.597
4.147
4.006
2.596
1.512
Me
O
N
Boc
CN
4h, 400 MHz, CDCl3
1.982
2.000
1.985
1.930
3.015
9.225
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

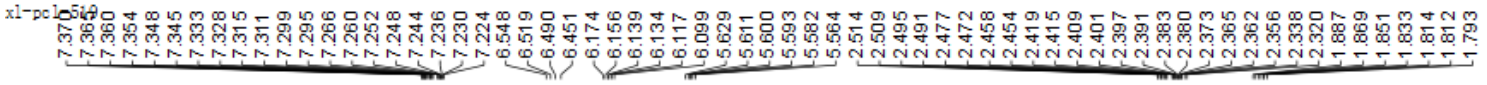


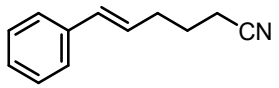
4h, 101 MHz, $CDCl_3$


CN
Me
O
4i, 400 MHz, CDCl3
f1 (ppm)

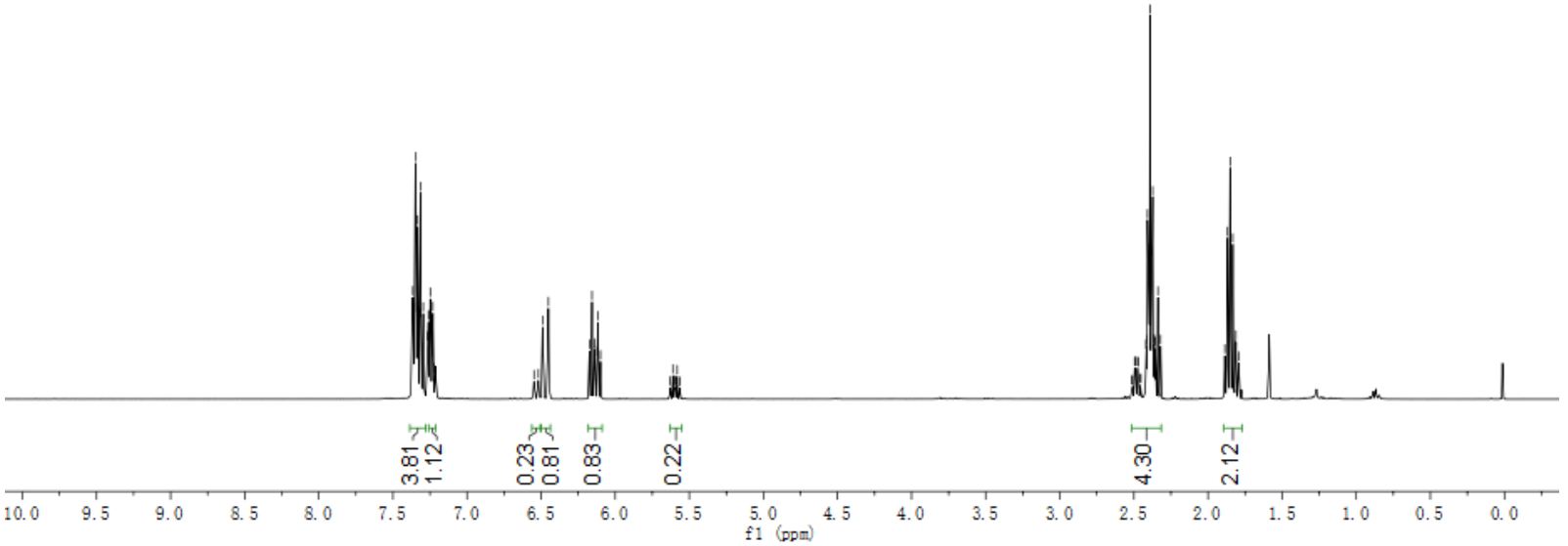


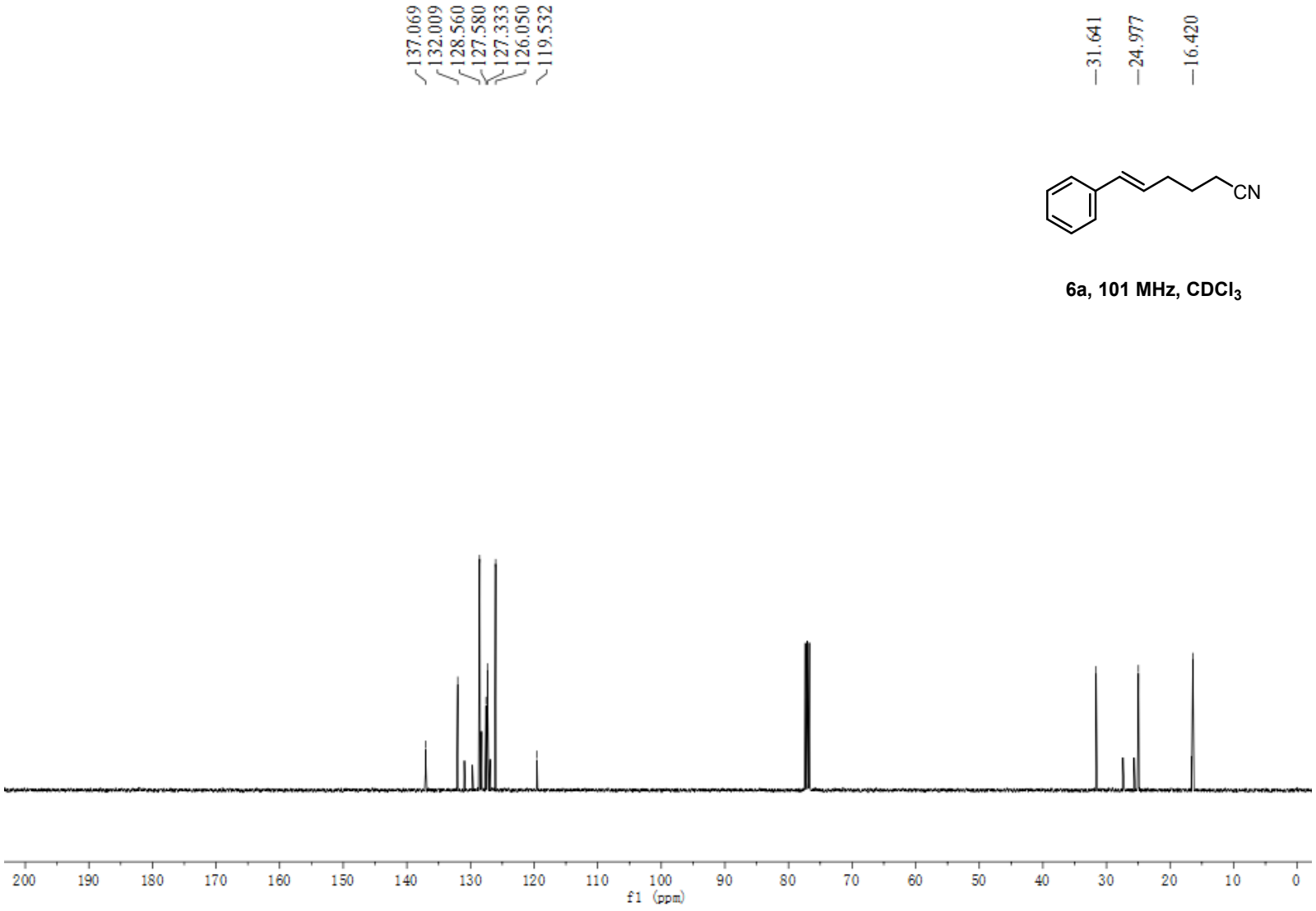
4i, 101 MHz, $CDCl_3$



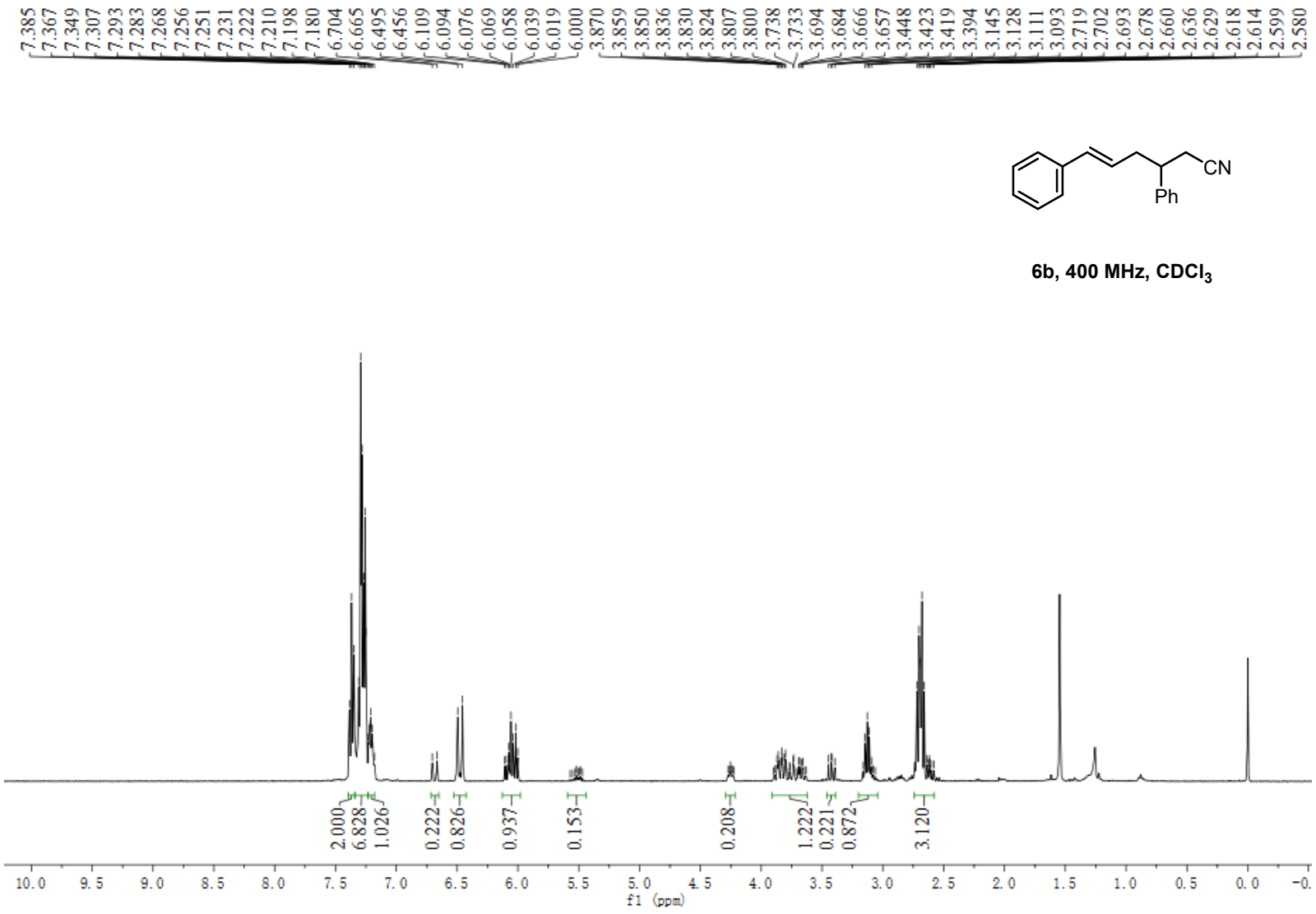


6a, 400 MHz, $CDCl_3$

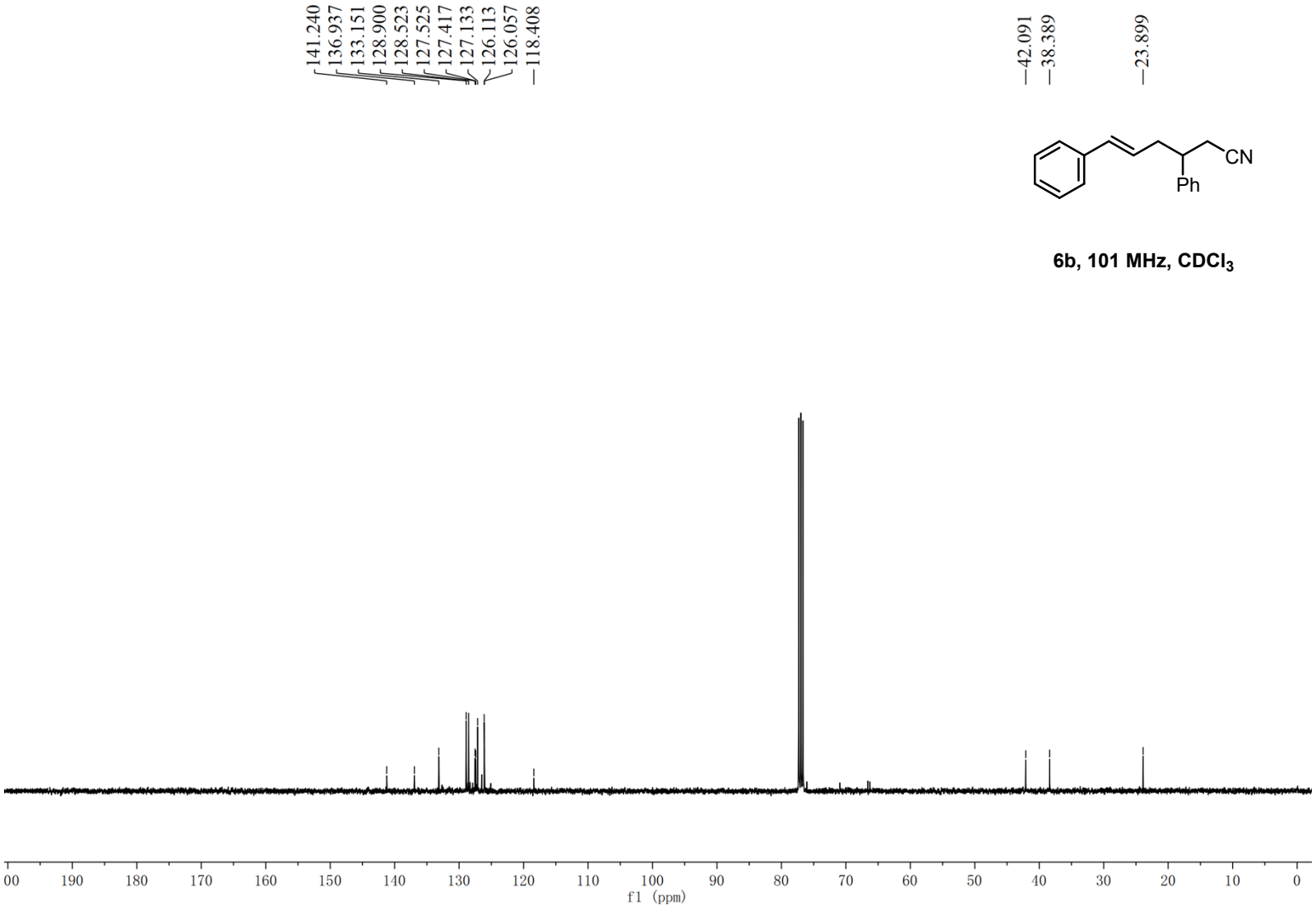




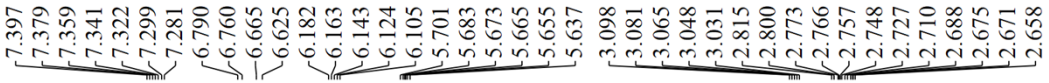
6a, 101 MHz, $CDCl_3$

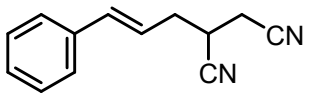


6b, 400 MHz, $CDCl_3$

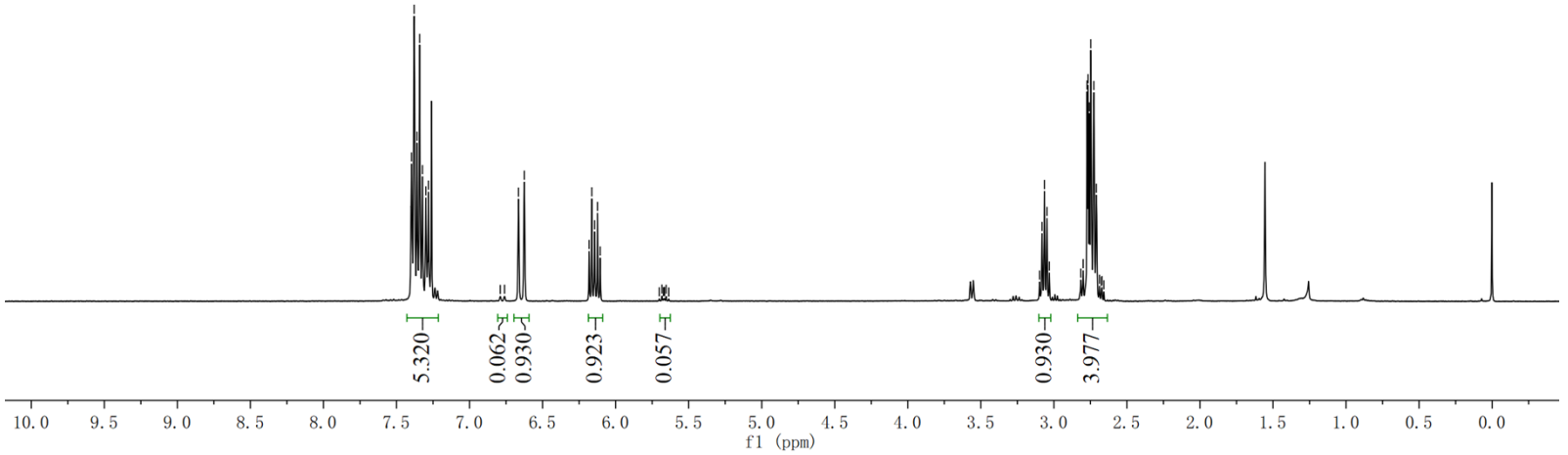


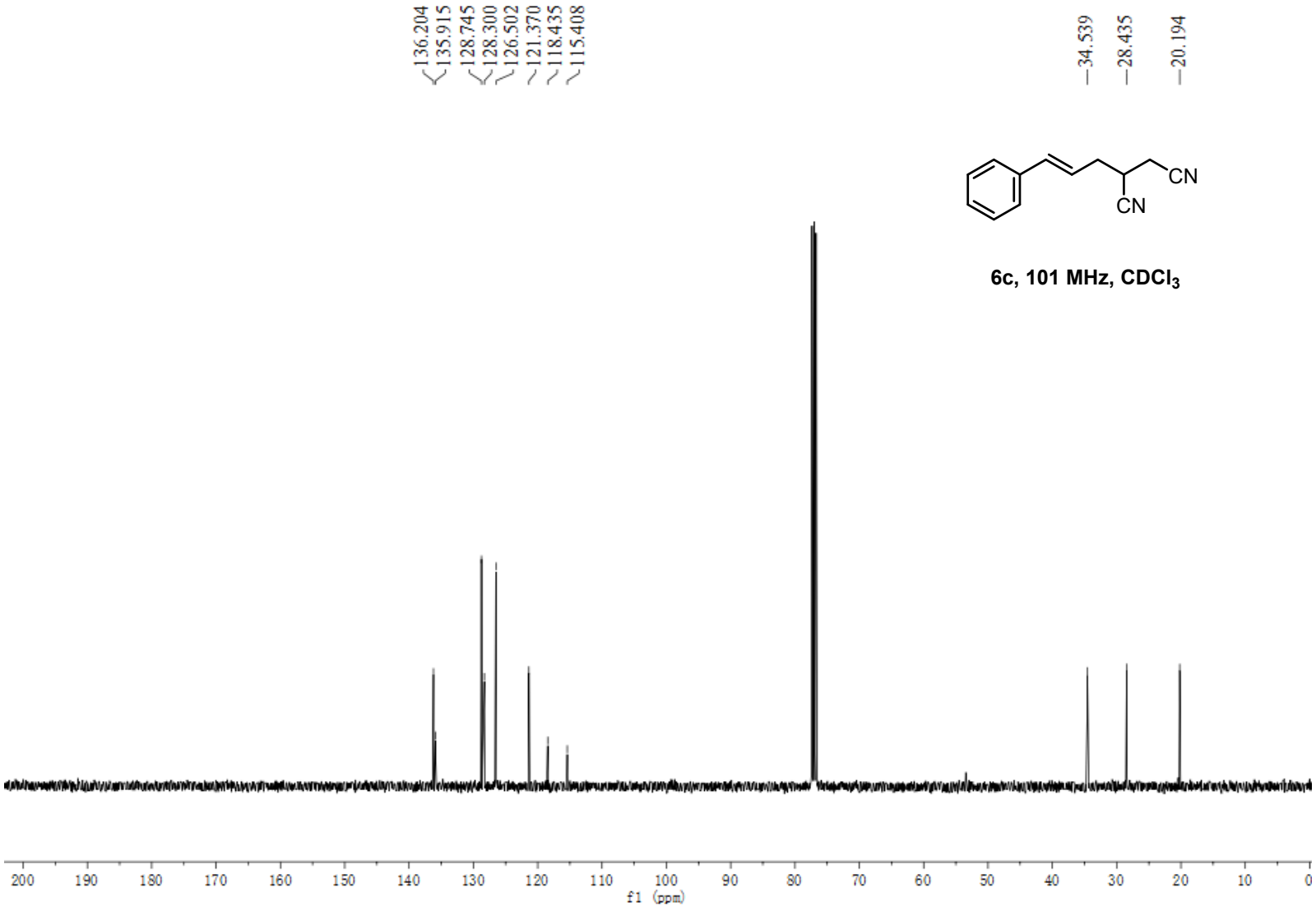
6b, 101 MHz, $CDCl_3$

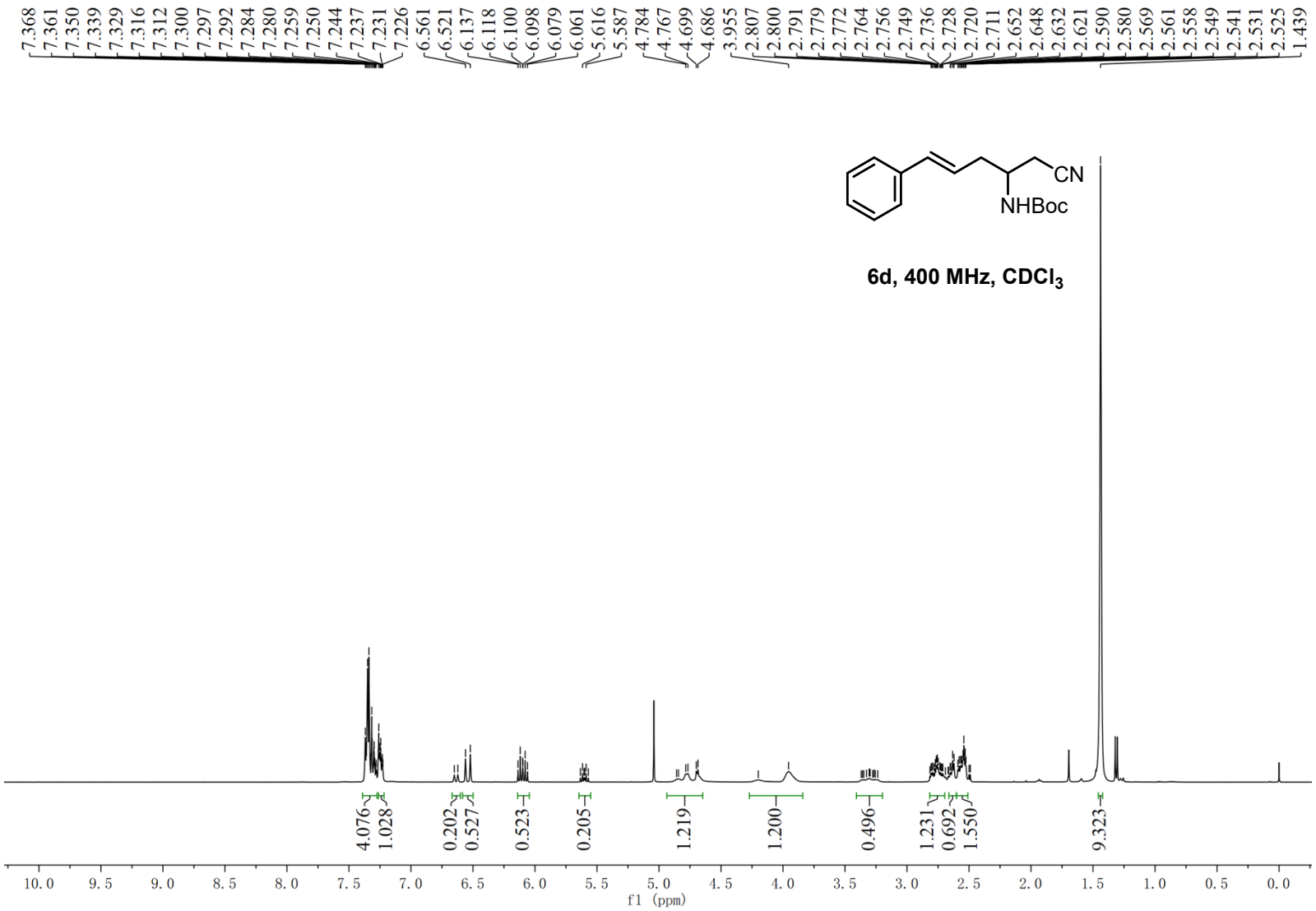


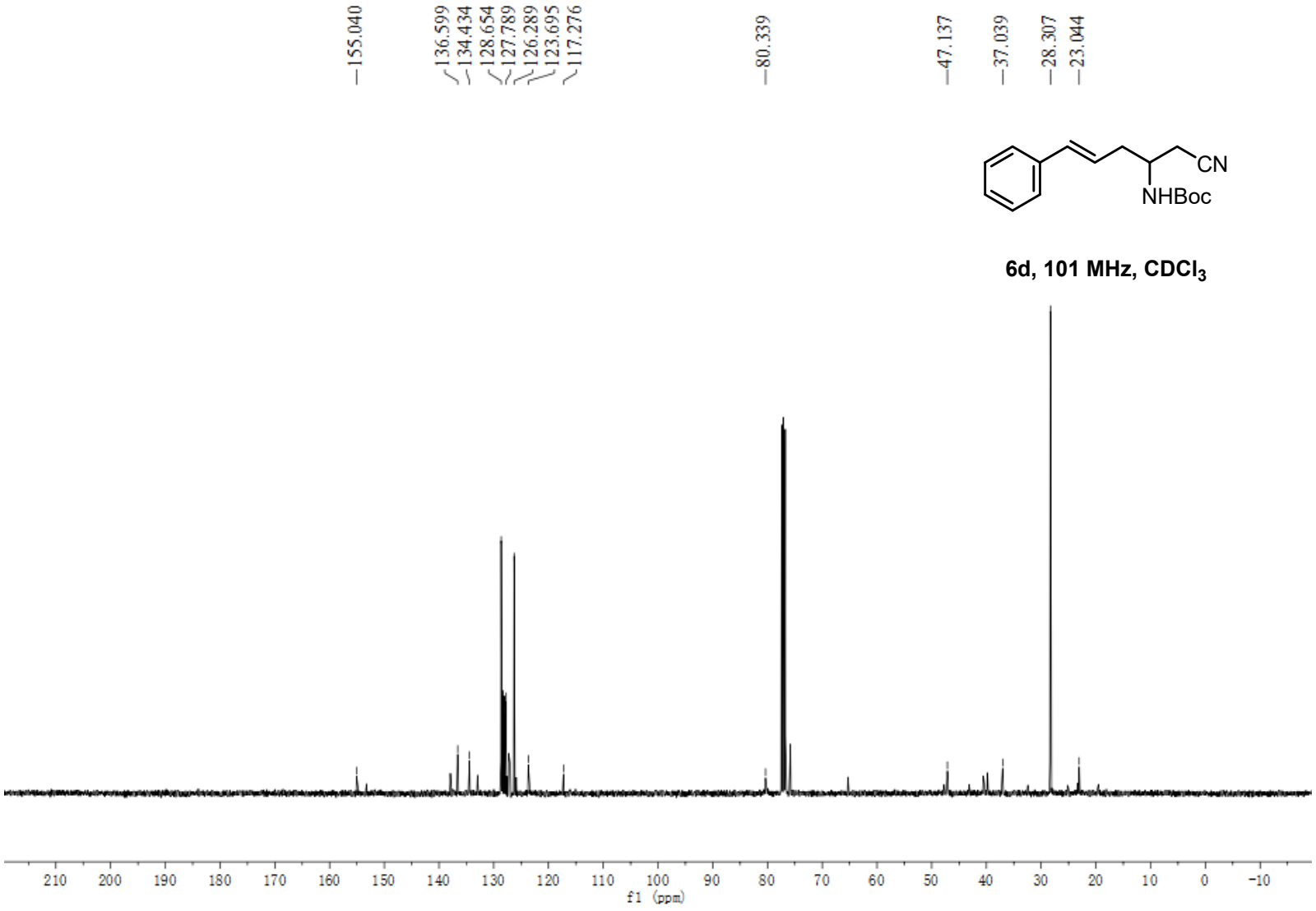


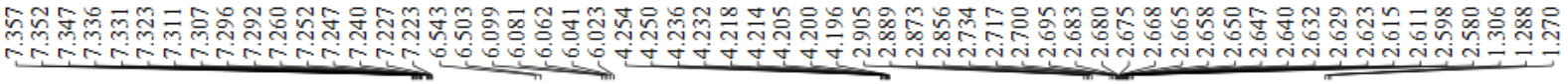
6c, 400 MHz, $CDCl_3$

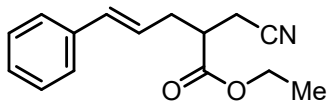



136.204
135.915
128.745
128.300
126.502
121.370
118.435
115.408
34.539
28.435
20.194
CN
CN
6c, 101 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

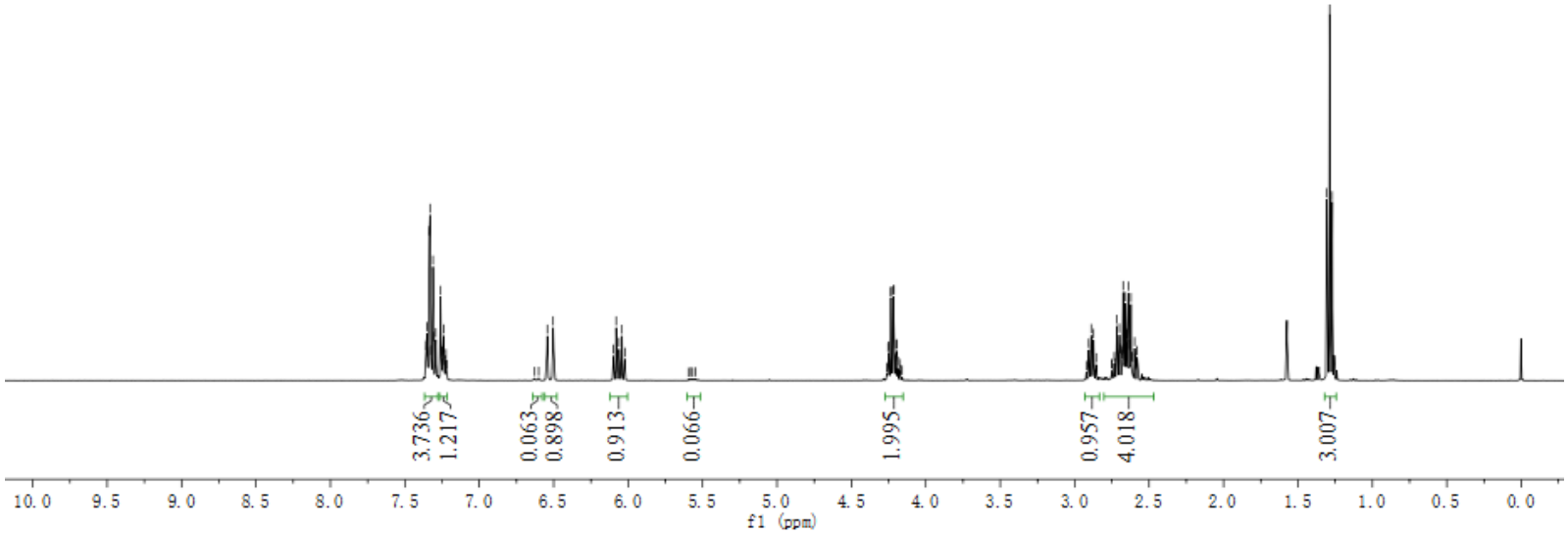

7.368
7.361
7.350
7.339
7.329
7.316
7.312
7.300
7.297
7.292
7.284
7.280
7.259
7.250
7.244
7.237
7.231
7.226
6.561
6.521
6.137
6.118
6.100
6.098
6.079
6.061
5.616
5.587
4.784
4.767
4.699
4.686
3.955
2.807
2.800
2.791
2.779
2.772
2.764
2.756
2.749
2.736
2.728
2.720
2.711
2.652
2.648
2.632
2.621
2.590
2.580
2.569
2.561
2.558
2.549
2.541
2.531
2.525
1.439
CN
NHBoc
6d, 400 MHz, CDCl3
4.076
1.028
0.202
0.527
0.523
0.205
1.219
1.200
0.496
1.231
0.692
1.550
9.323
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

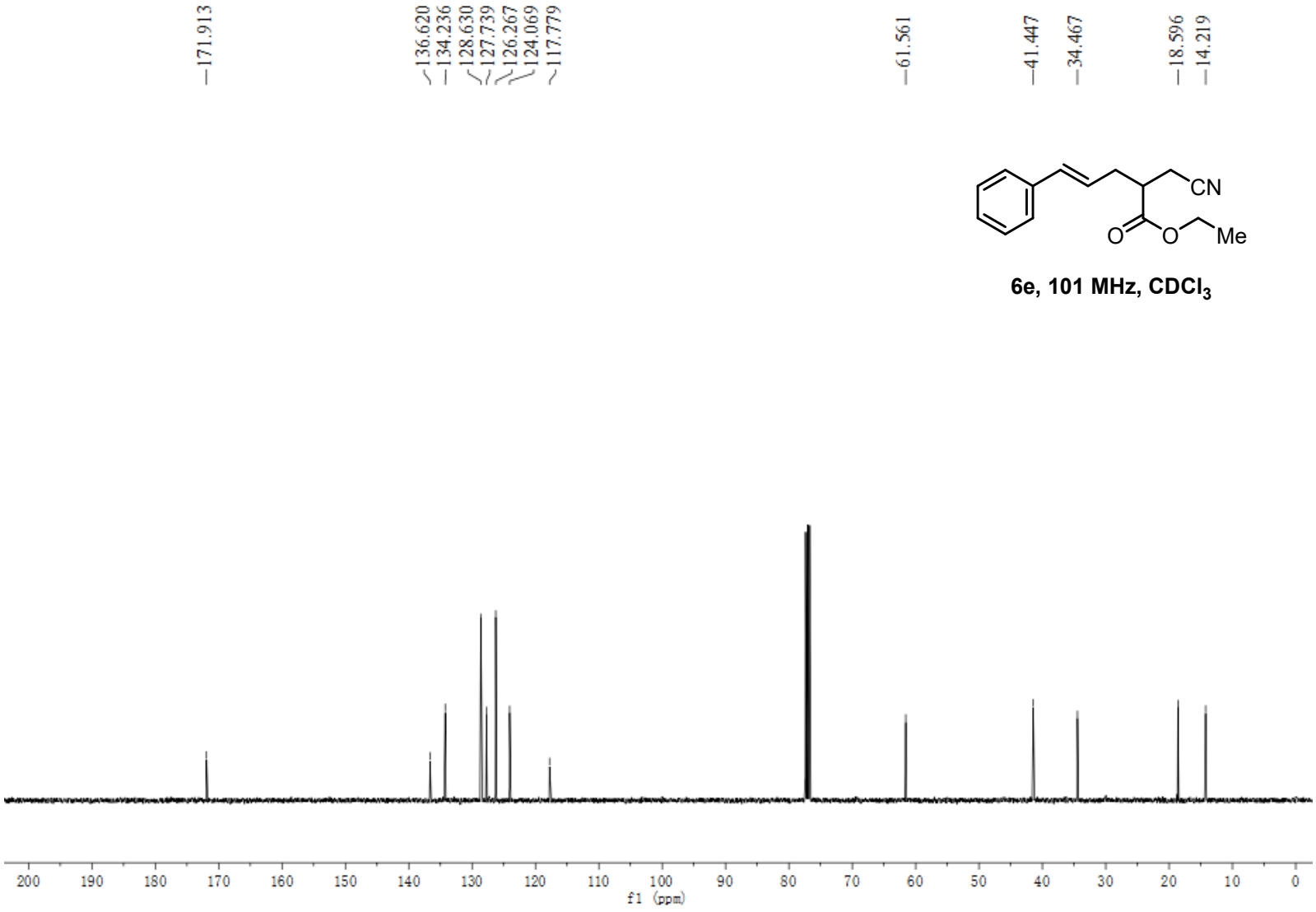

155.040
136.599
134.434
128.654
127.789
126.289
123.695
117.276
80.339
47.137
37.039
28.307
23.044
CN
NHBoc
6d, 101 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



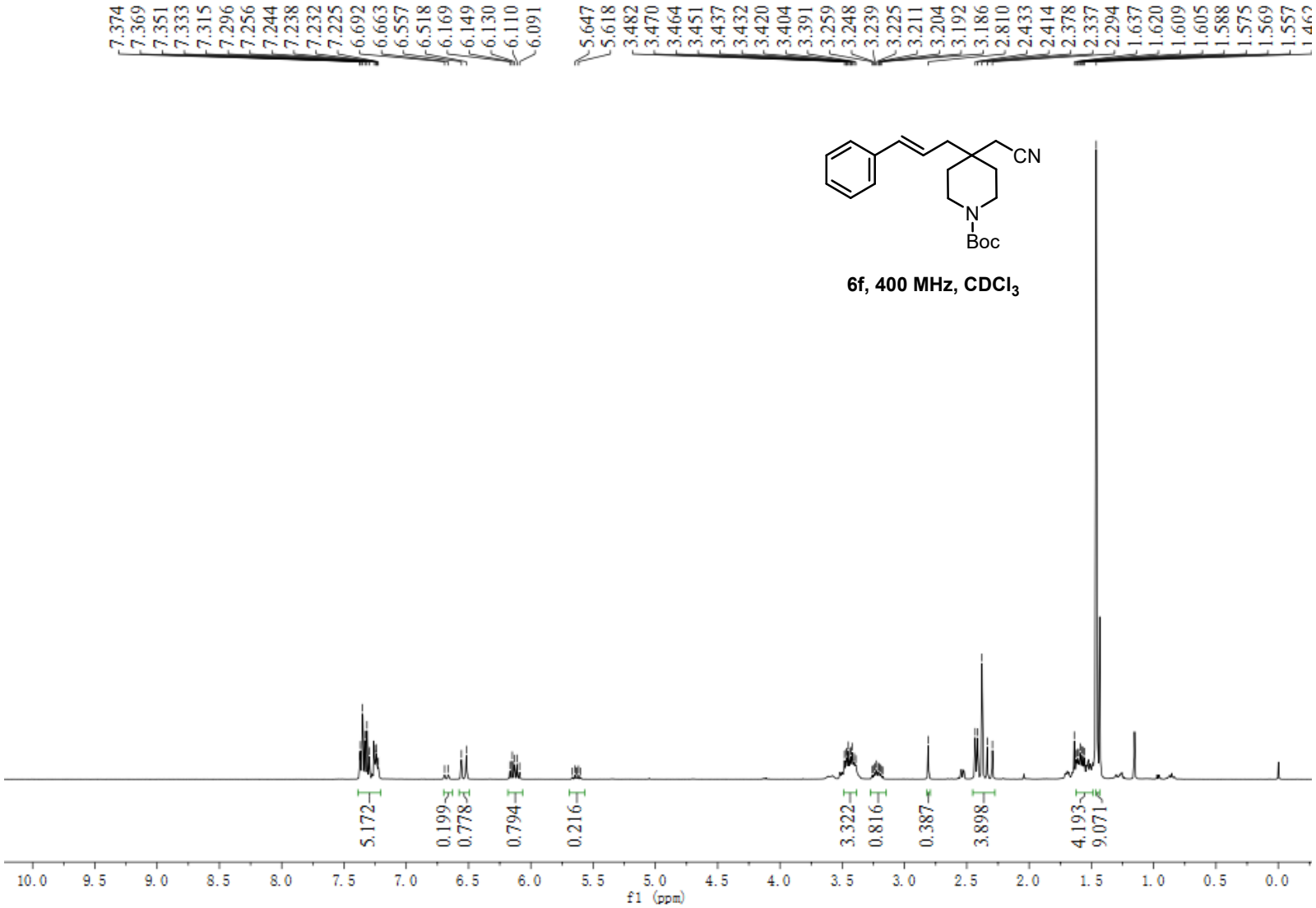


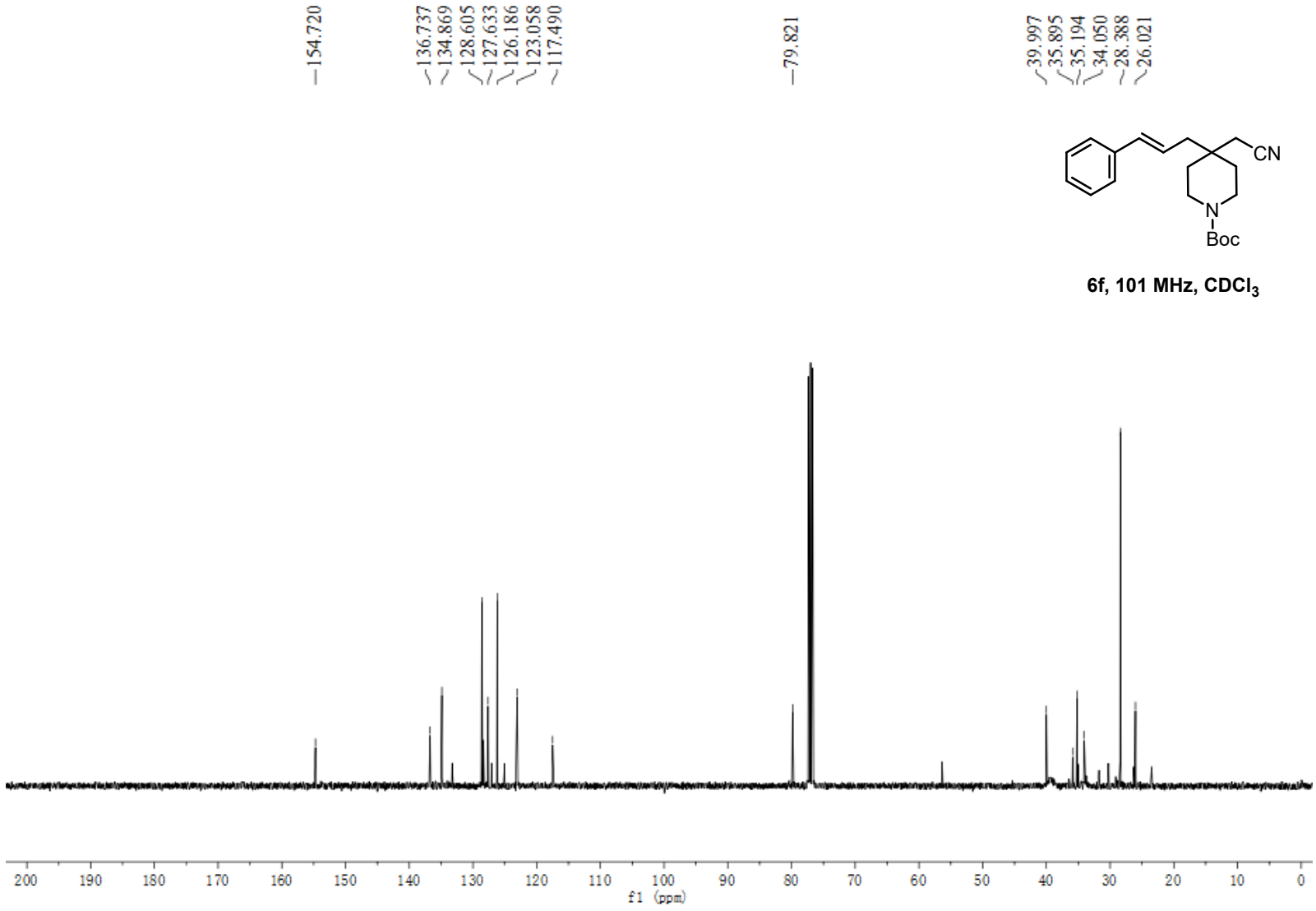
6e, 400 MHz, $CDCl_3$



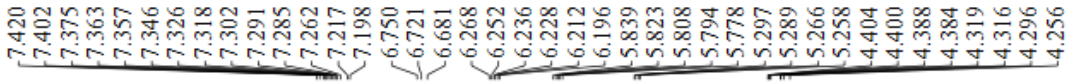


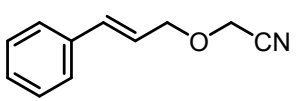
6e, 101 MHz, $CDCl_3$


7.374
7.369
7.351
7.333
7.315
7.296
7.256
7.244
7.238
7.232
7.225
6.692
6.663
6.557
6.518
6.169
6.149
6.130
6.110
6.091
5.647
5.618
3.482
3.470
3.464
3.451
3.437
3.432
3.420
3.404
3.391
3.259
3.248
3.239
3.225
3.211
3.204
3.192
3.186
2.810
2.433
2.414
2.378
2.337
2.294
1.637
1.620
1.609
1.605
1.588
1.575
1.569
1.557
CN
N
Boc
6f, 400 MHz, CDCl3
5.172
0.199
0.778
0.794
0.216
3.322
0.816
0.387
3.898
4.193
9.071
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

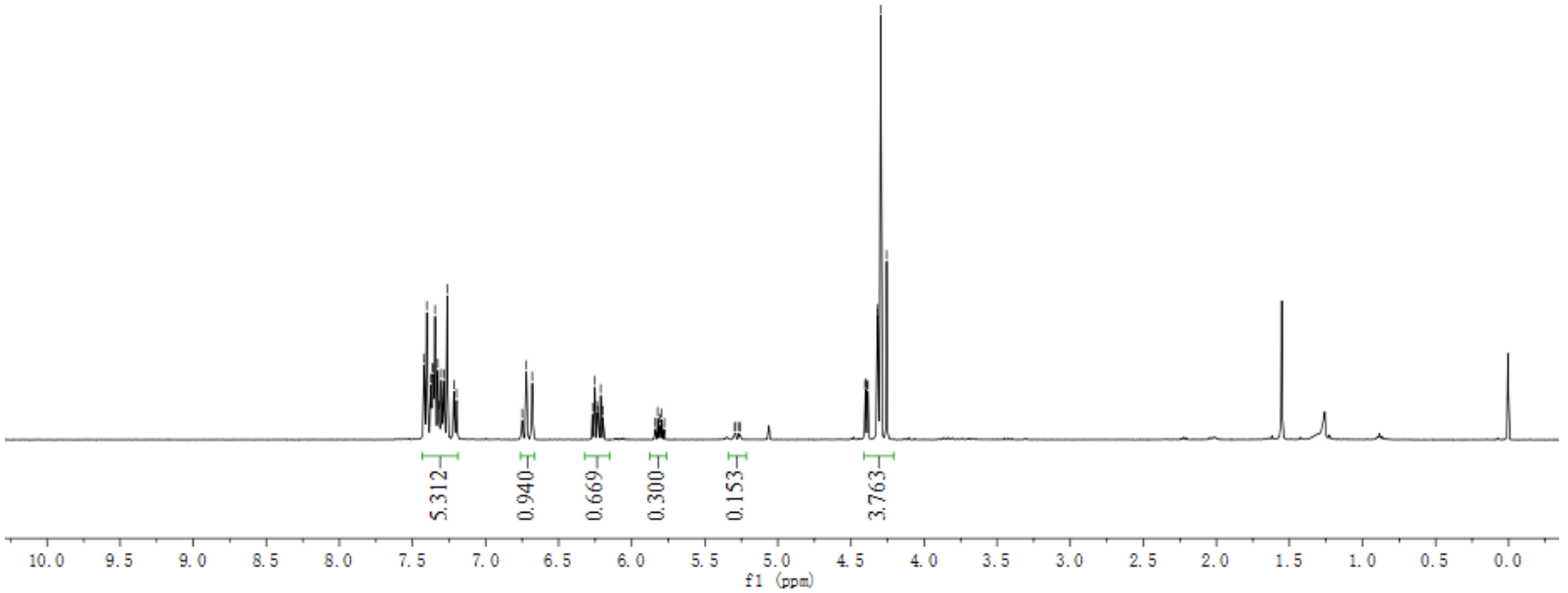


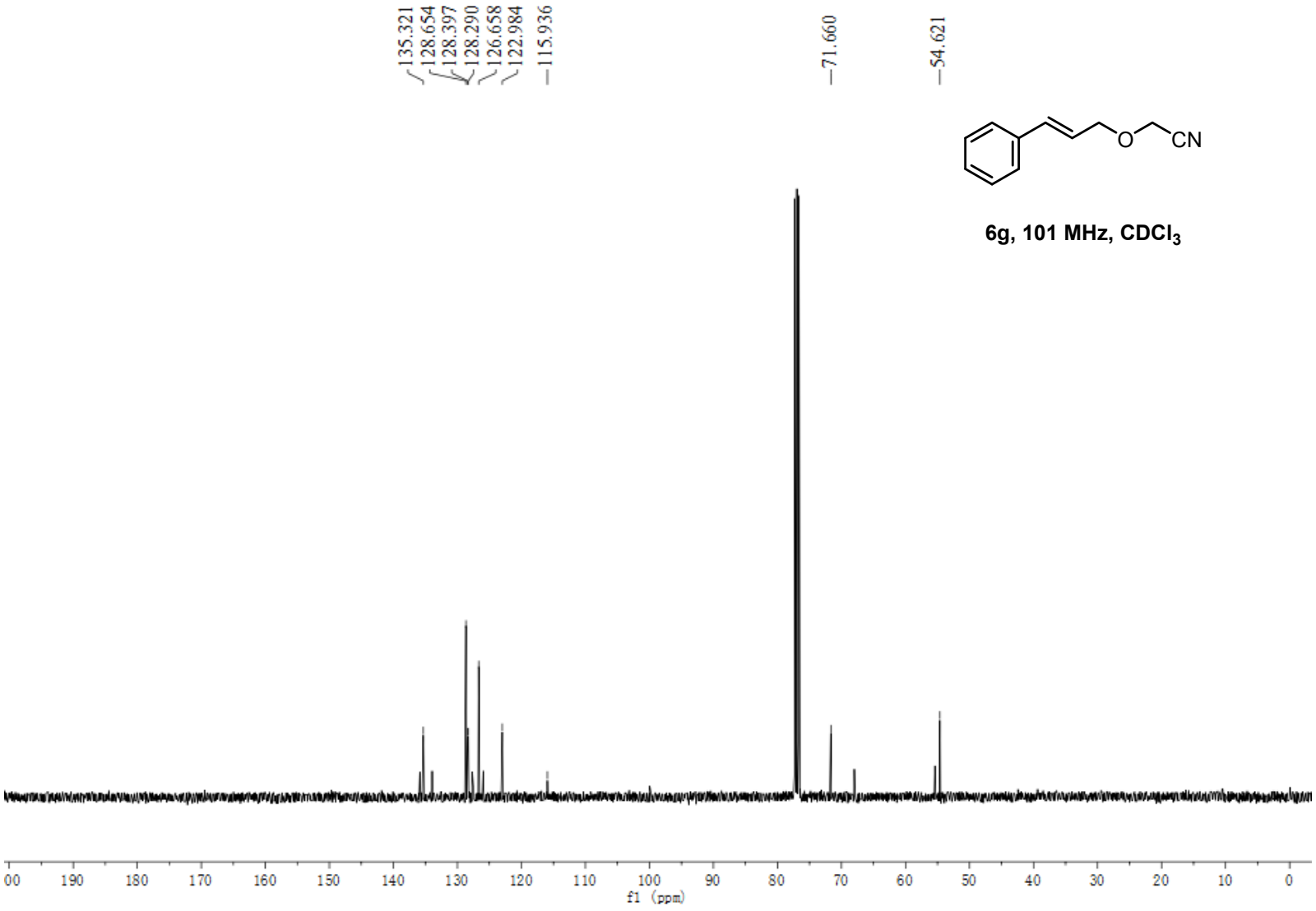
6f, 101 MHz, $CDCl_3$



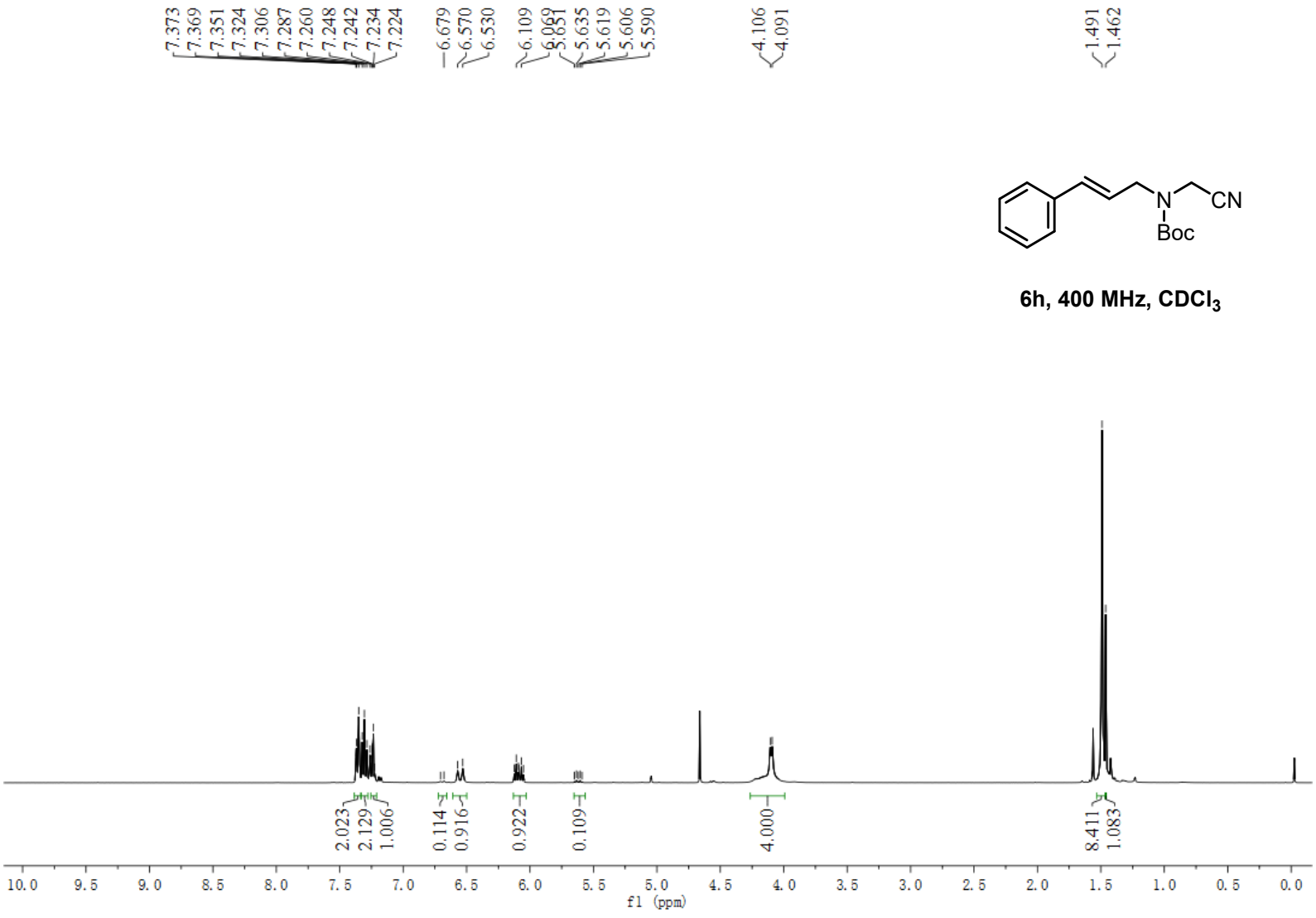


6g, 400 MHz, $CDCl_3$

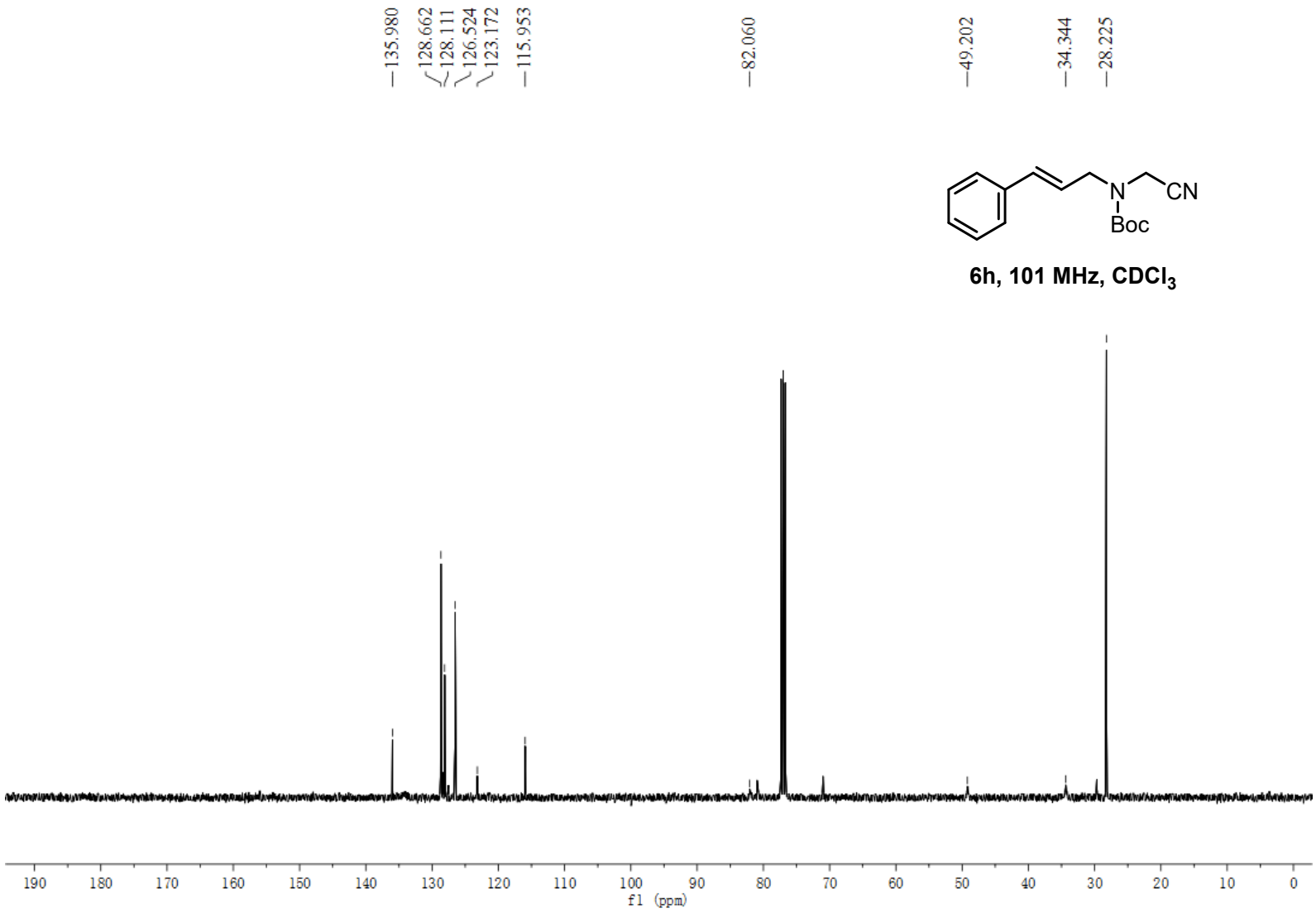




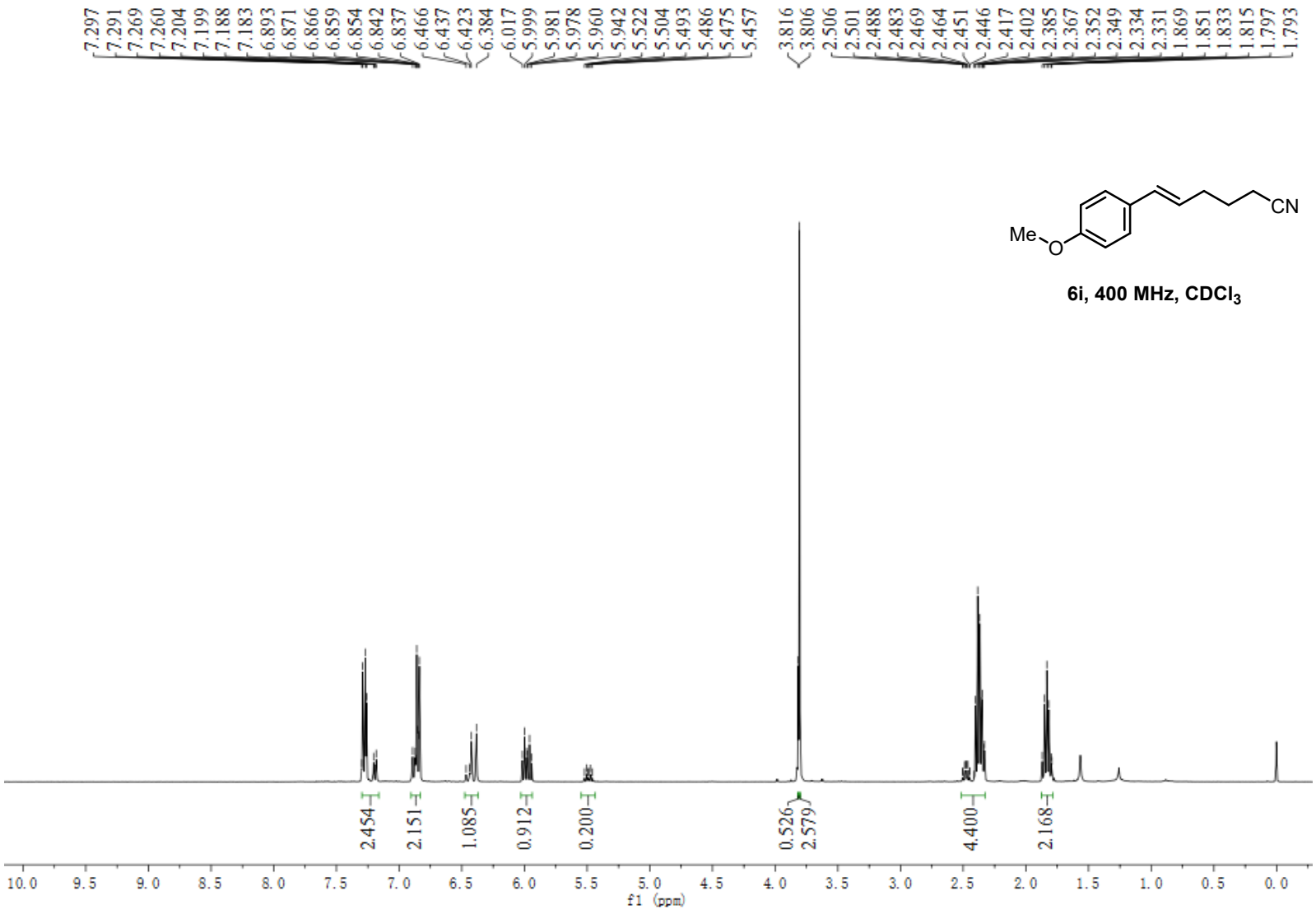
6g, 101 MHz, $CDCl_3$

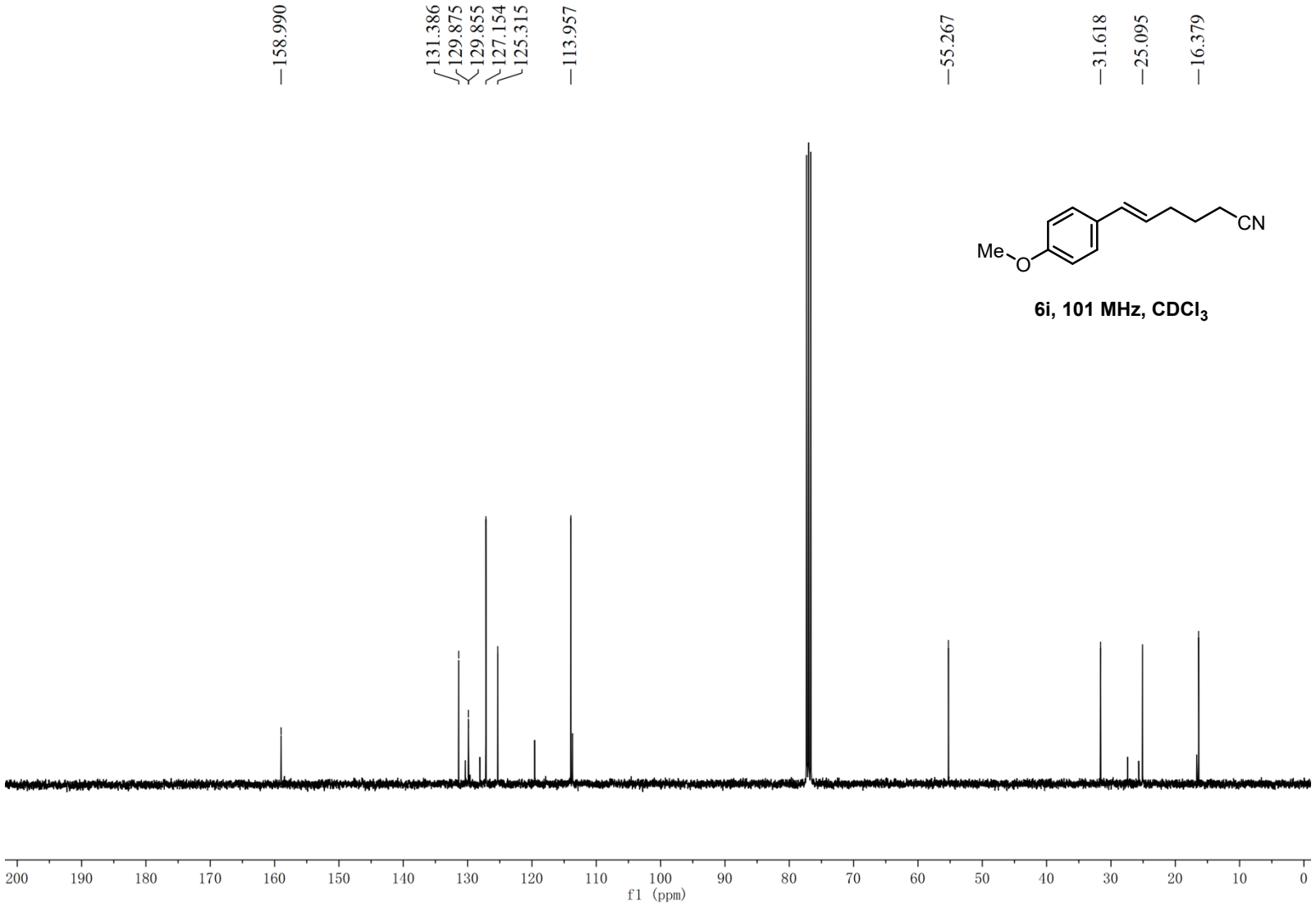


6h, 400 MHz, $CDCl_3$

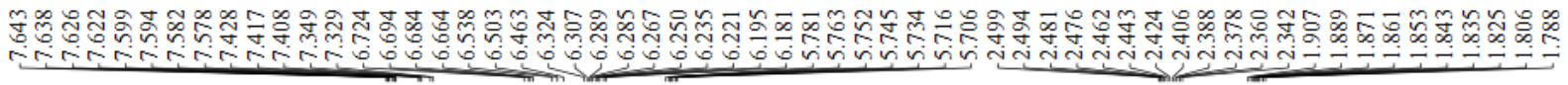


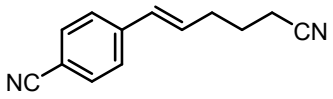
6h, 101 MHz, $CDCl_3$


7.297
7.291
7.269
7.260
7.204
7.199
7.188
7.183
6.893
6.871
6.866
6.859
6.854
6.842
6.837
6.466
6.437
6.423
6.384
6.017
5.999
5.981
5.978
5.960
5.942
5.522
5.504
5.493
5.486
5.475
5.457
3.816
3.806
2.506
2.501
2.488
2.483
2.469
2.464
2.451
2.446
2.417
2.402
2.385
2.367
2.352
2.349
2.334
2.331
1.869
1.851
1.833
1.815
1.797
1.793
Me
O
CN
6i, 400 MHz, CDCl3
2.454
2.151
1.085
0.912
0.200
0.526
2.579
4.400
2.168
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

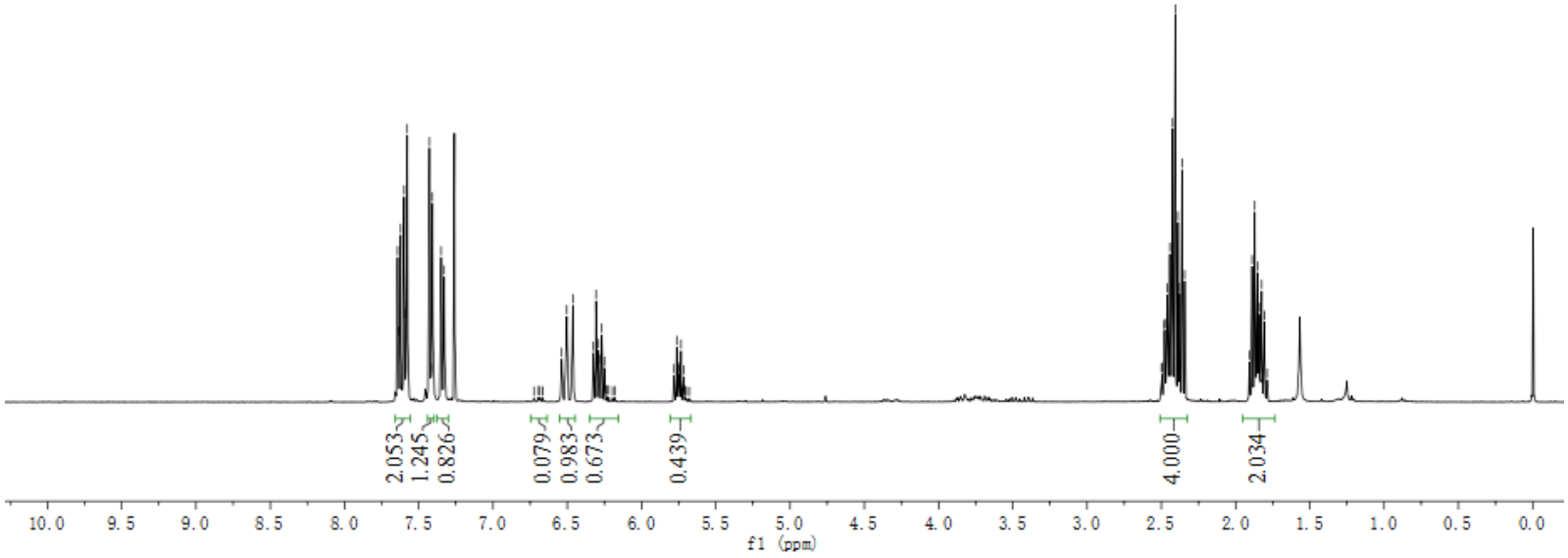


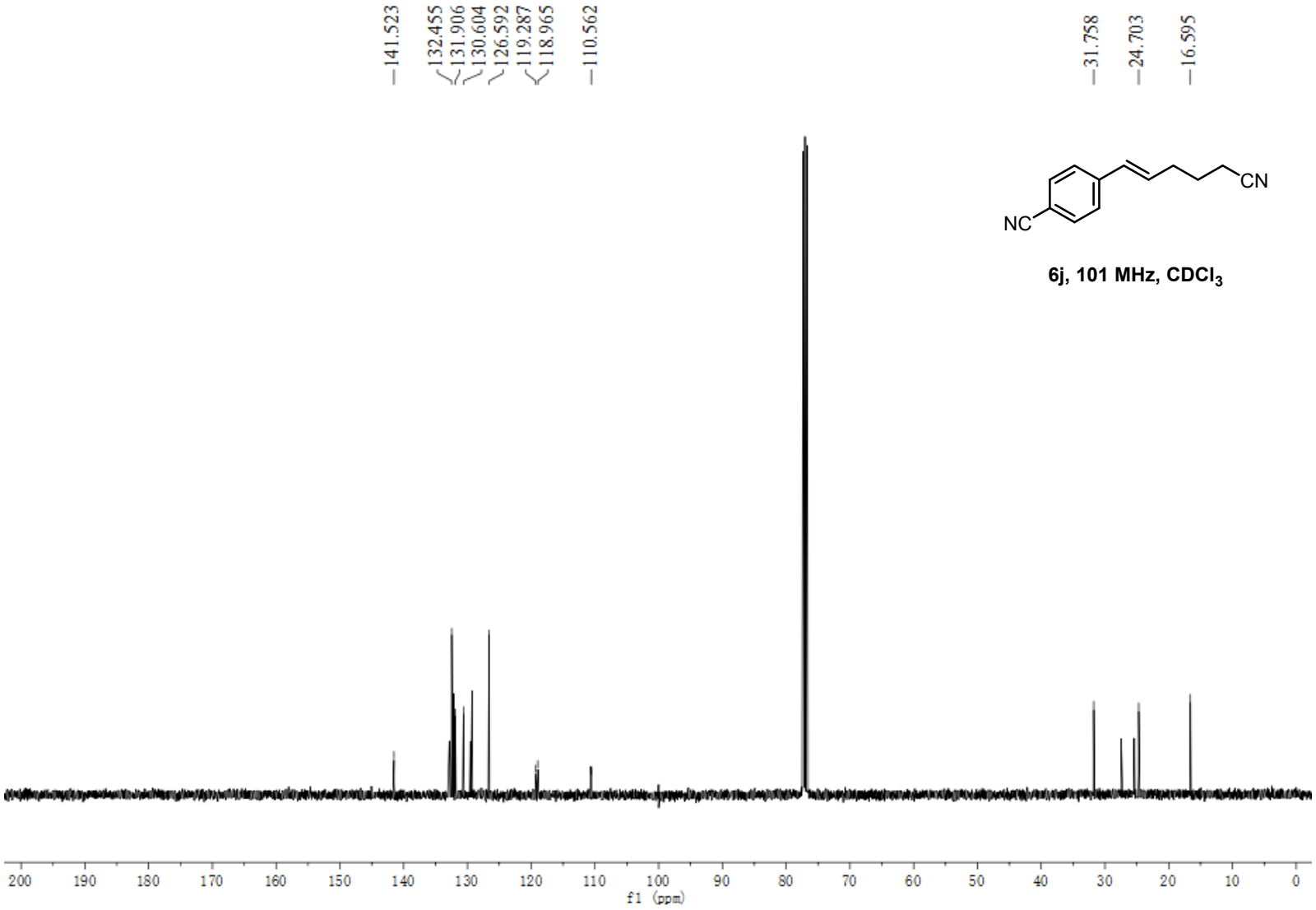
6i, 101 MHz, $CDCl_3$



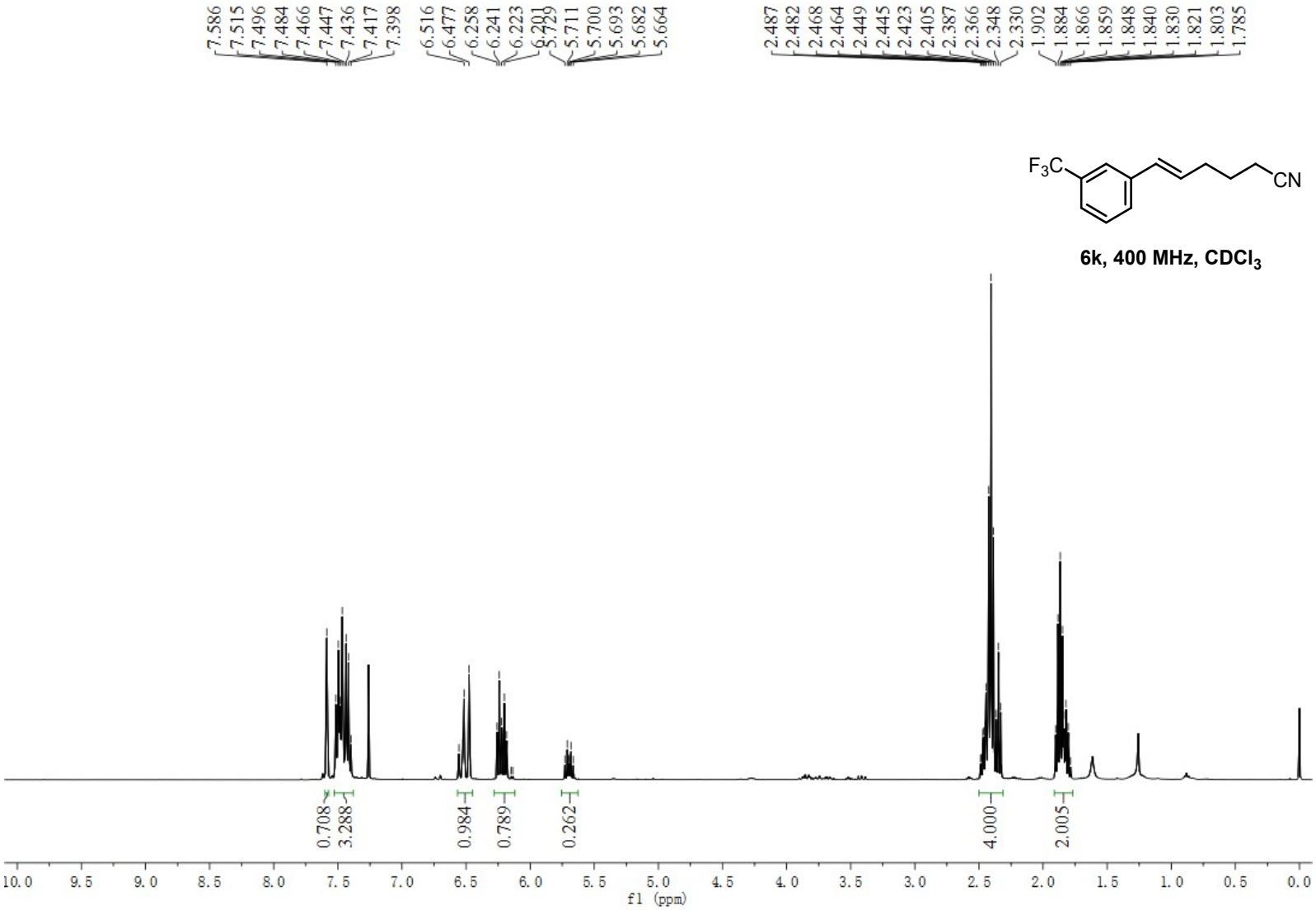


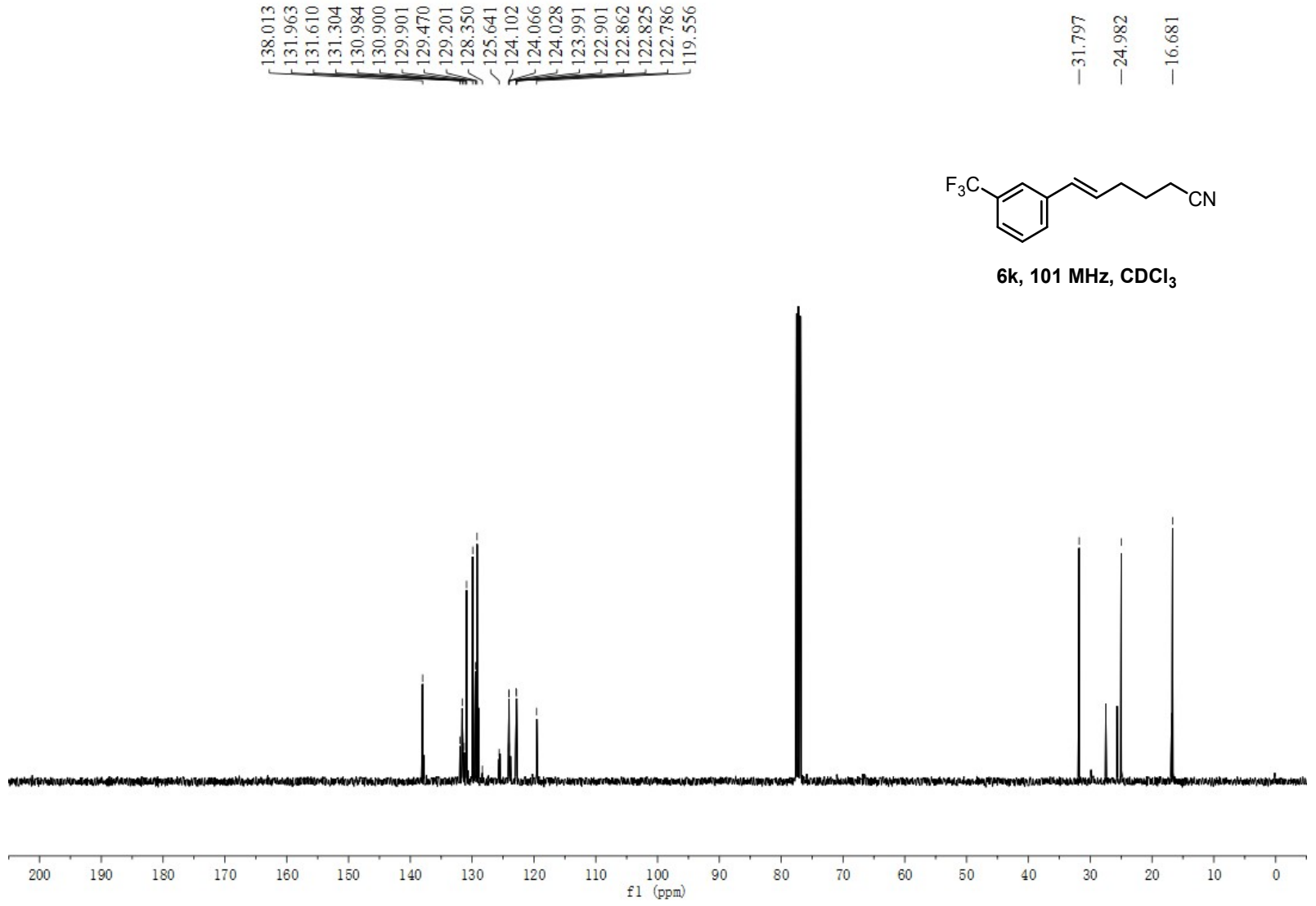
6j, 400 MHz, $CDCl_3$



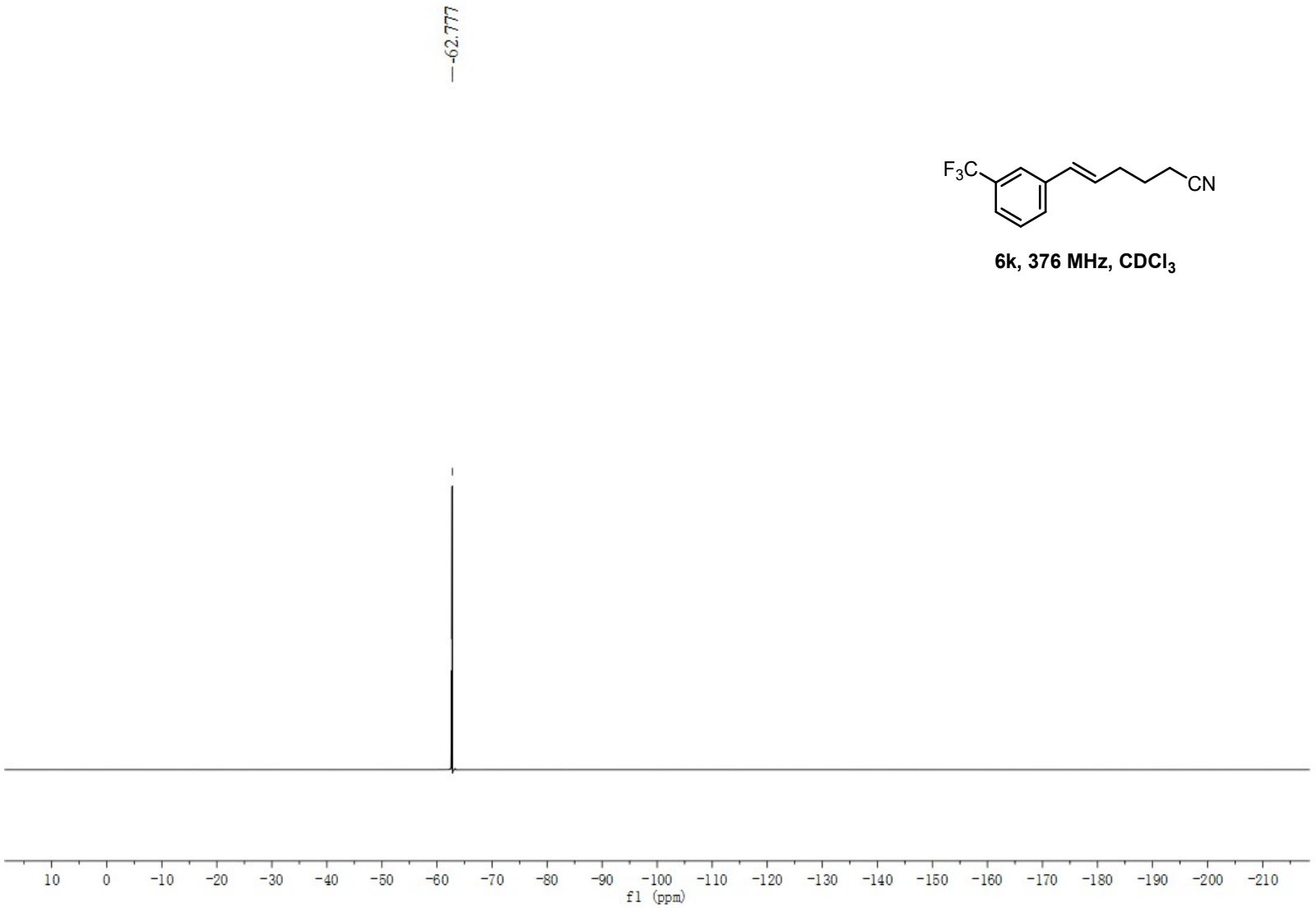


6j, 101 MHz, $CDCl_3$

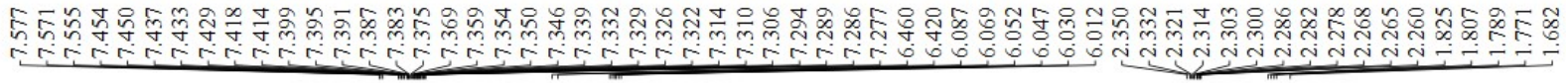

7.586
7.515
7.496
7.484
7.466
7.447
7.436
7.417
7.398
6.516
6.477
6.258
6.241
6.223
6.201
5.729
5.711
5.700
5.693
5.682
5.664
2.487
2.482
2.468
2.464
2.449
2.445
2.423
2.405
2.387
2.366
2.348
2.330
1.902
1.884
1.866
1.859
1.848
1.840
1.830
1.821
1.803
1.785
F3C
CN
6k, 400 MHz, CDCl3
0.708
3.288
0.984
0.789
0.262
4.000
2.005
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

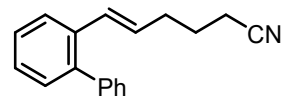


6k, 101 MHz, $CDCl_3$

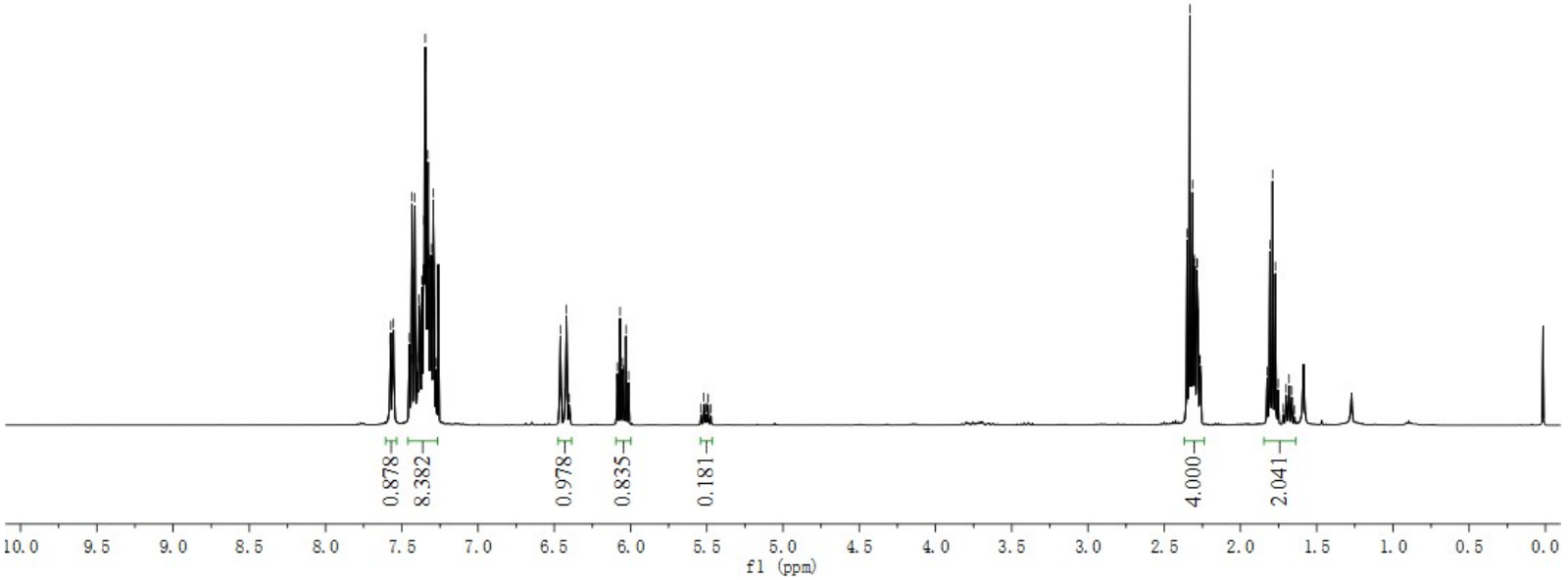


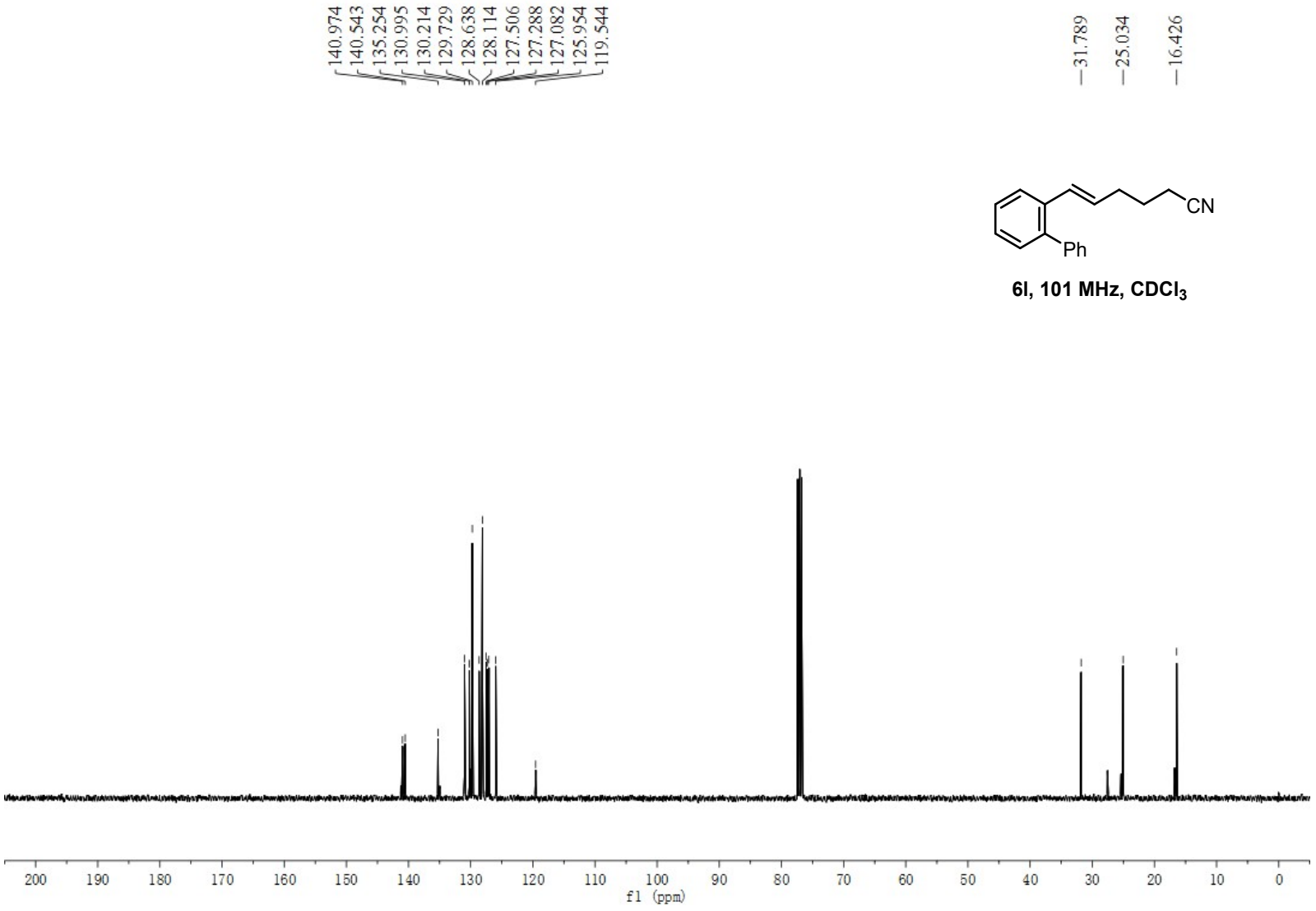
6k, 376 MHz, $CDCl_3$



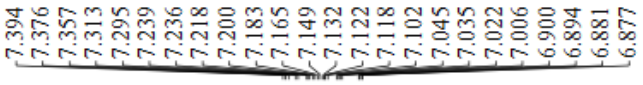


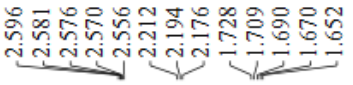
6l, 400 MHz, $CDCl_3$

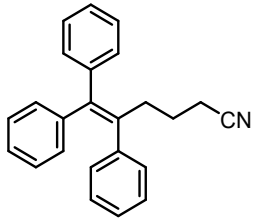




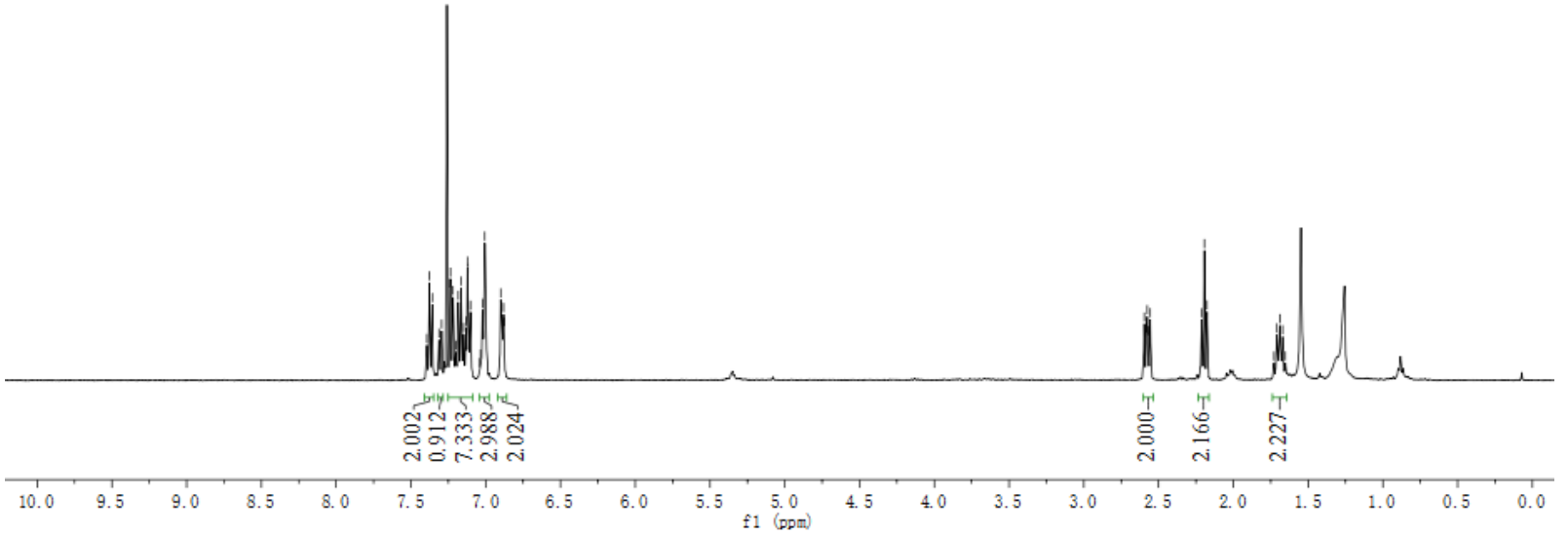
6l, 101 MHz, $CDCl_3$

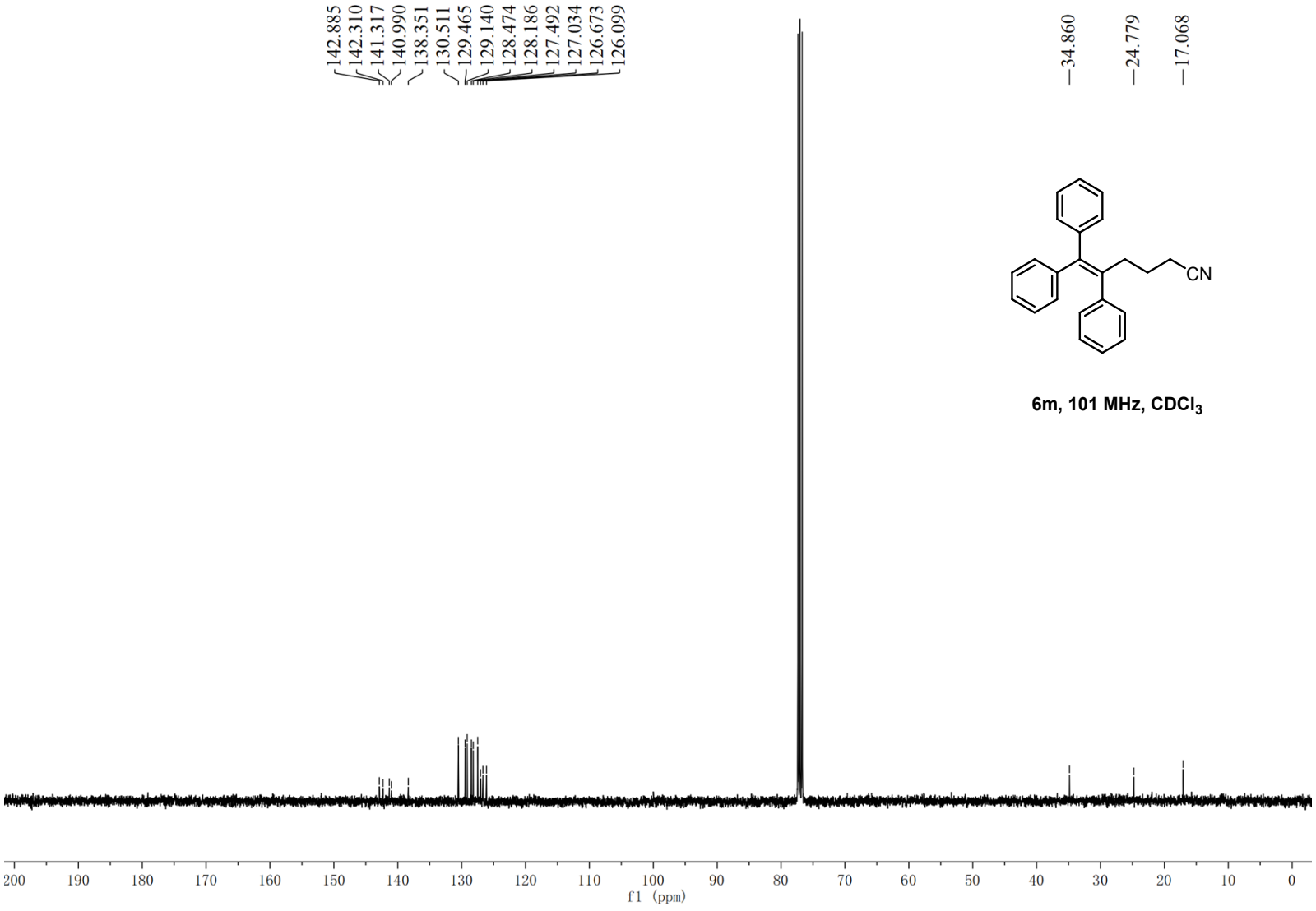




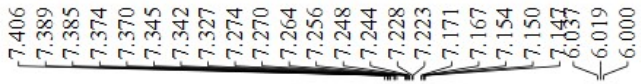


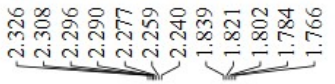
6m, 400 MHz, $CDCl_3$

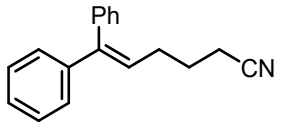




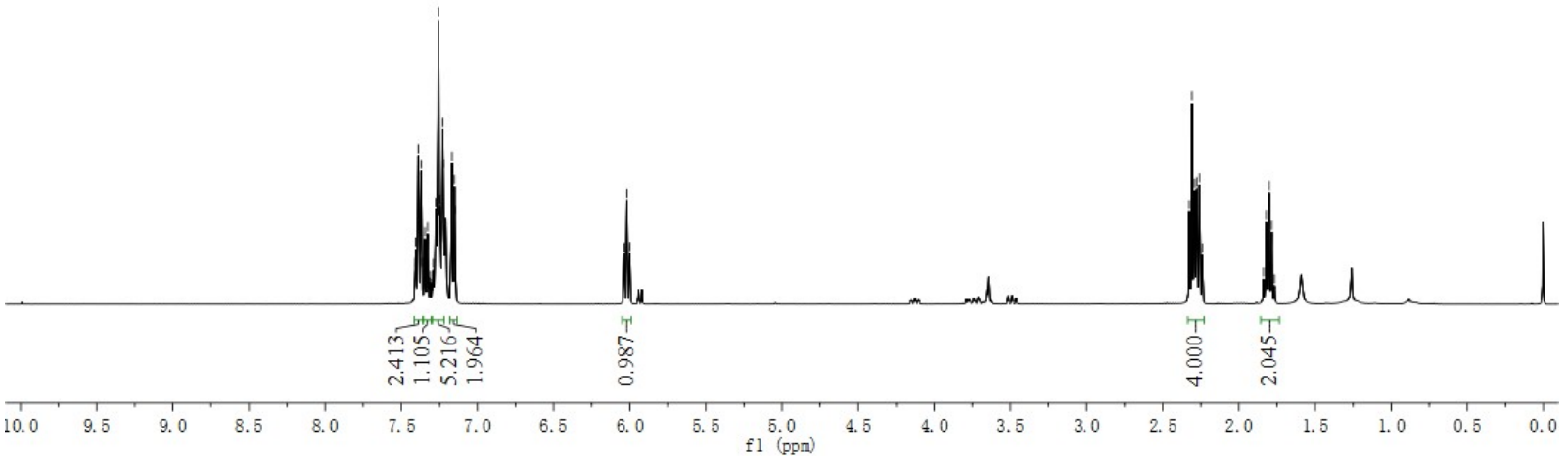
6m, 101 MHz, $CDCl_3$

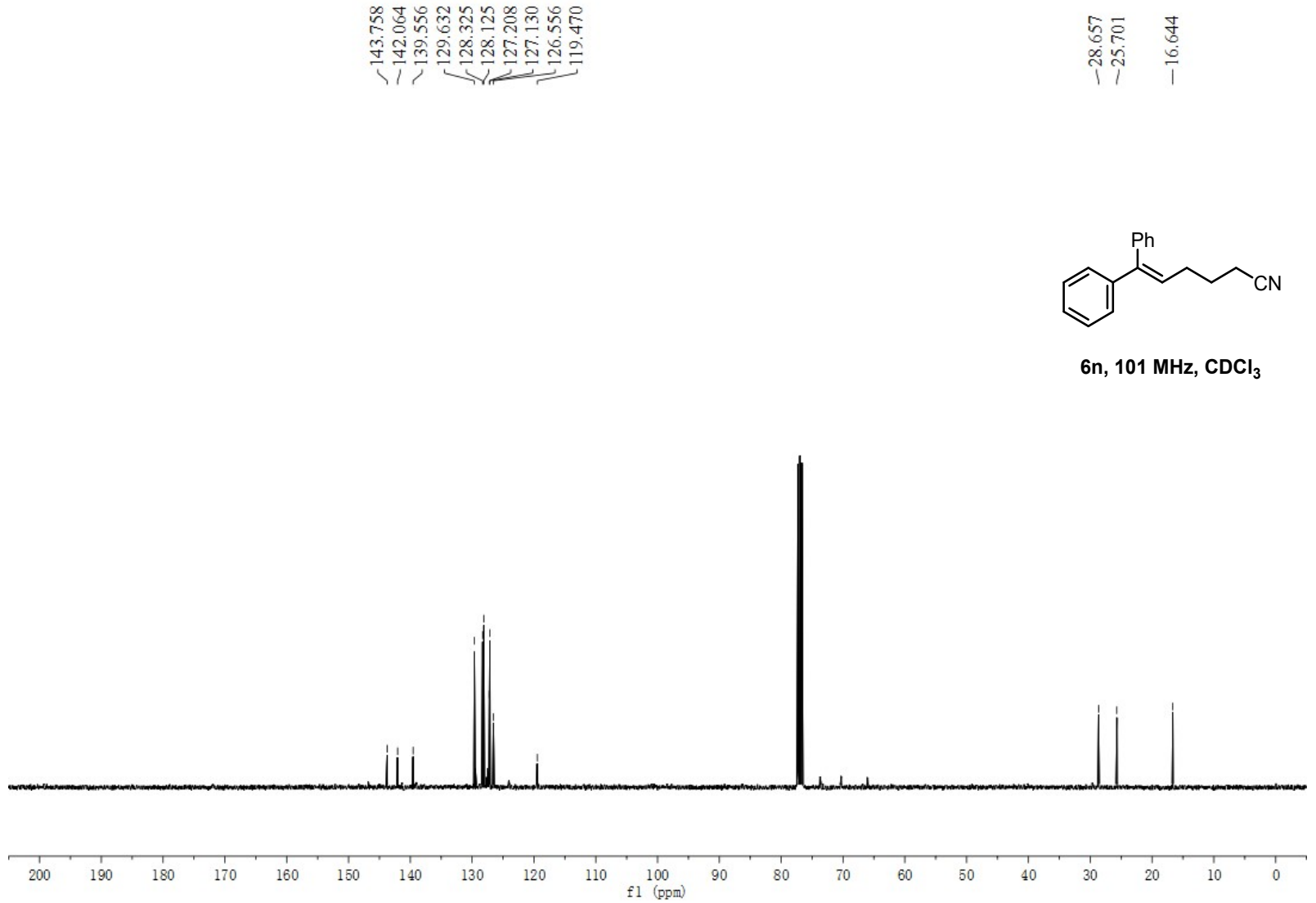




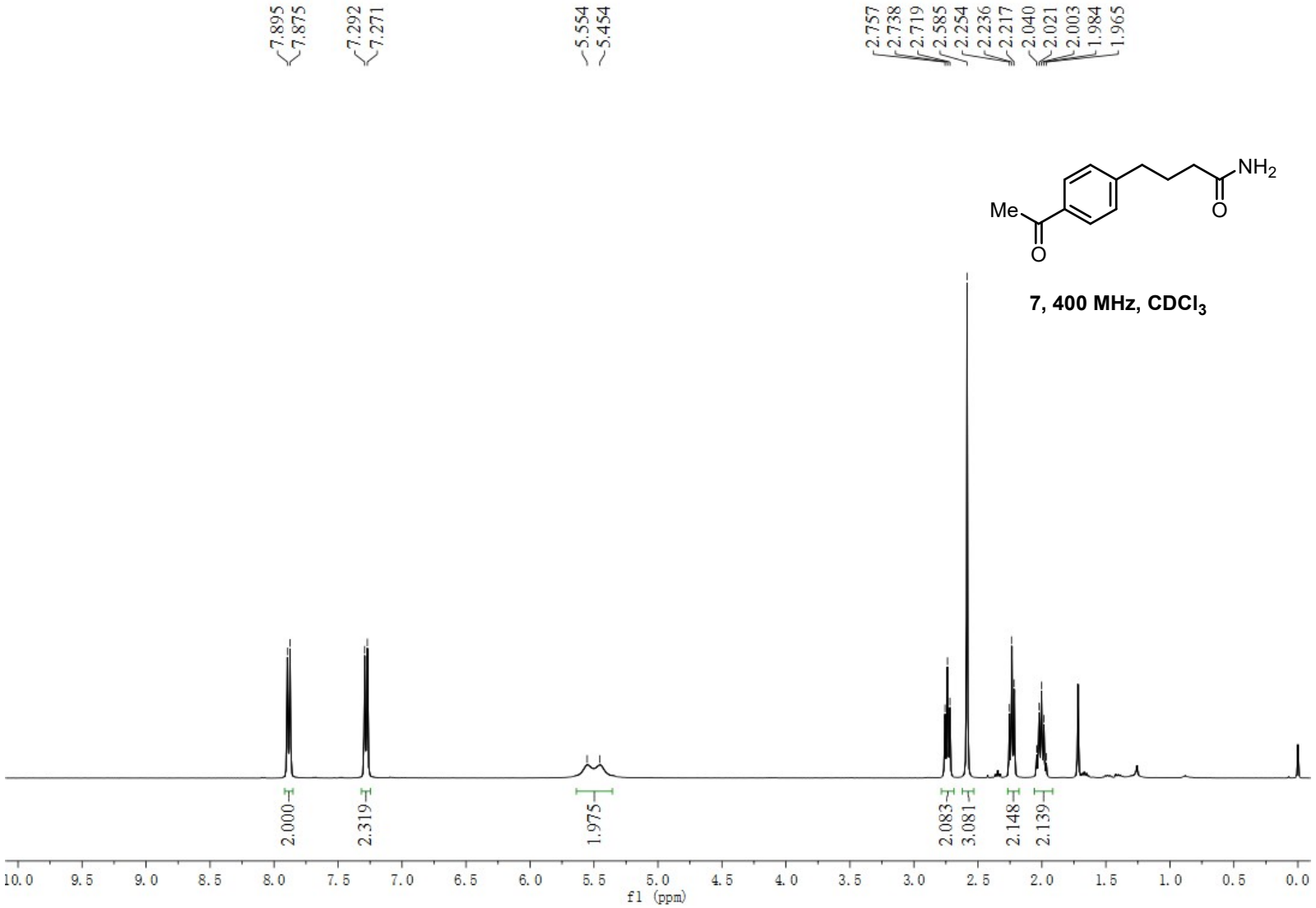


6n, 400 MHz, $CDCl_3$

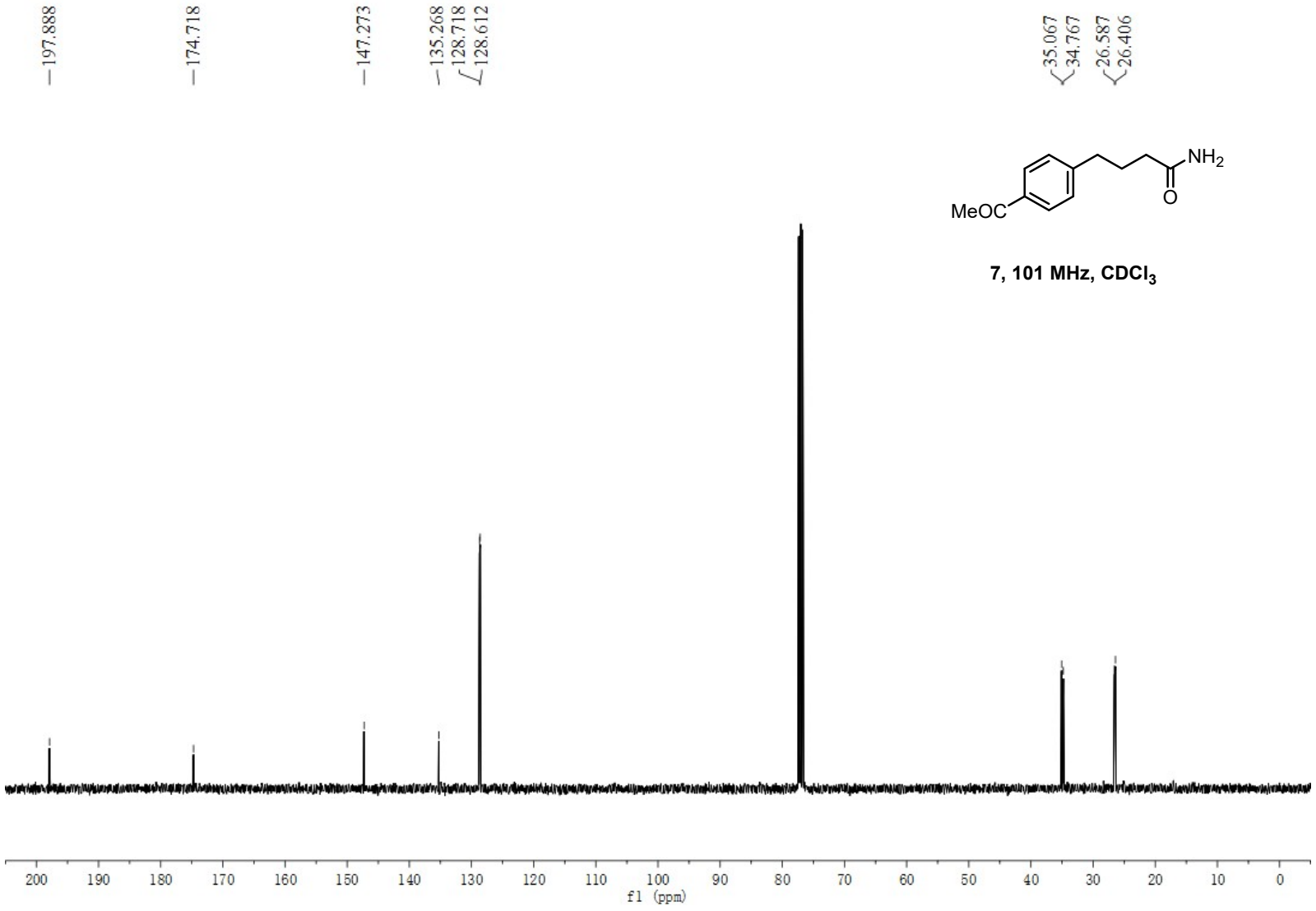




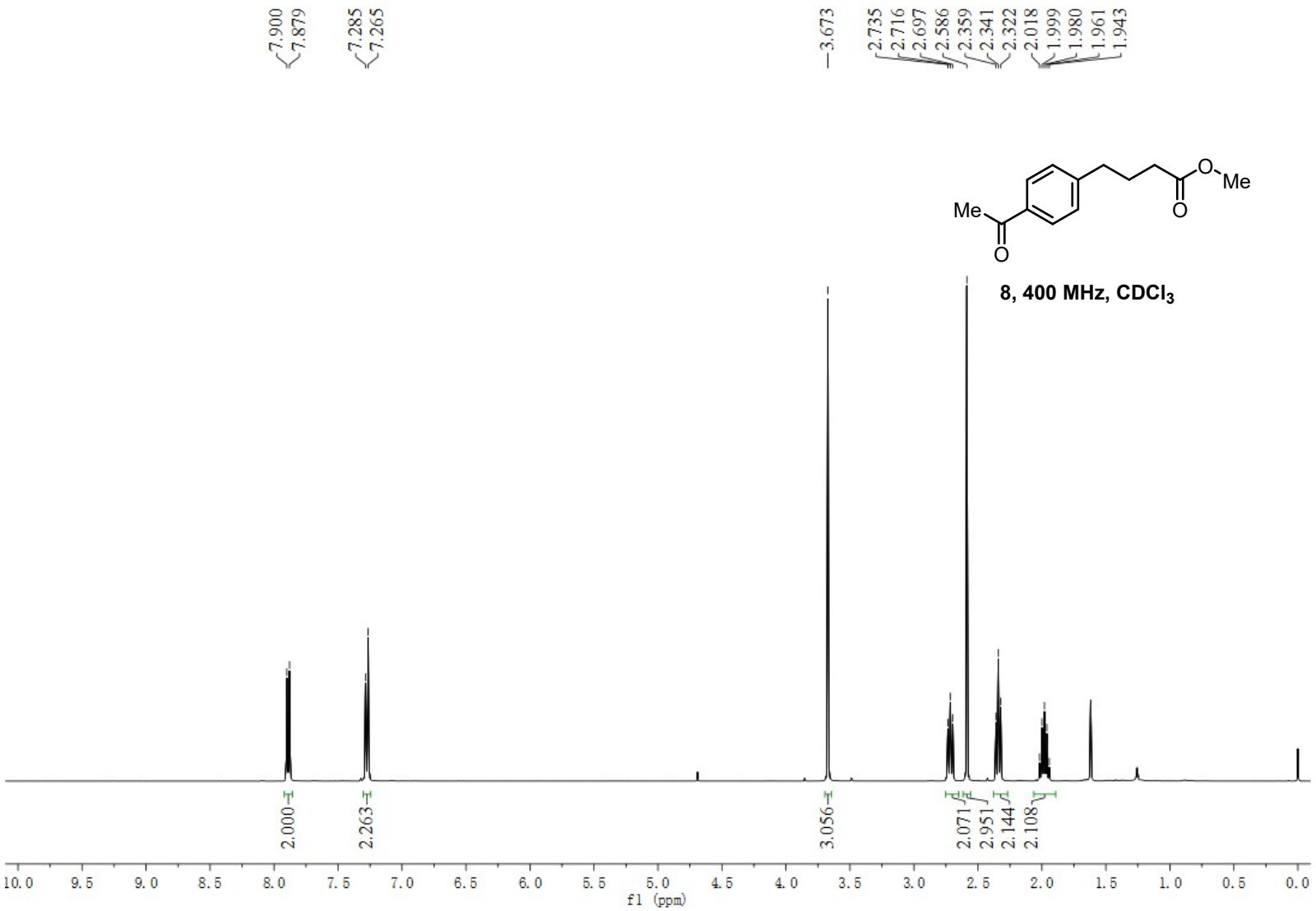
6n, 101 MHz, $CDCl_3$

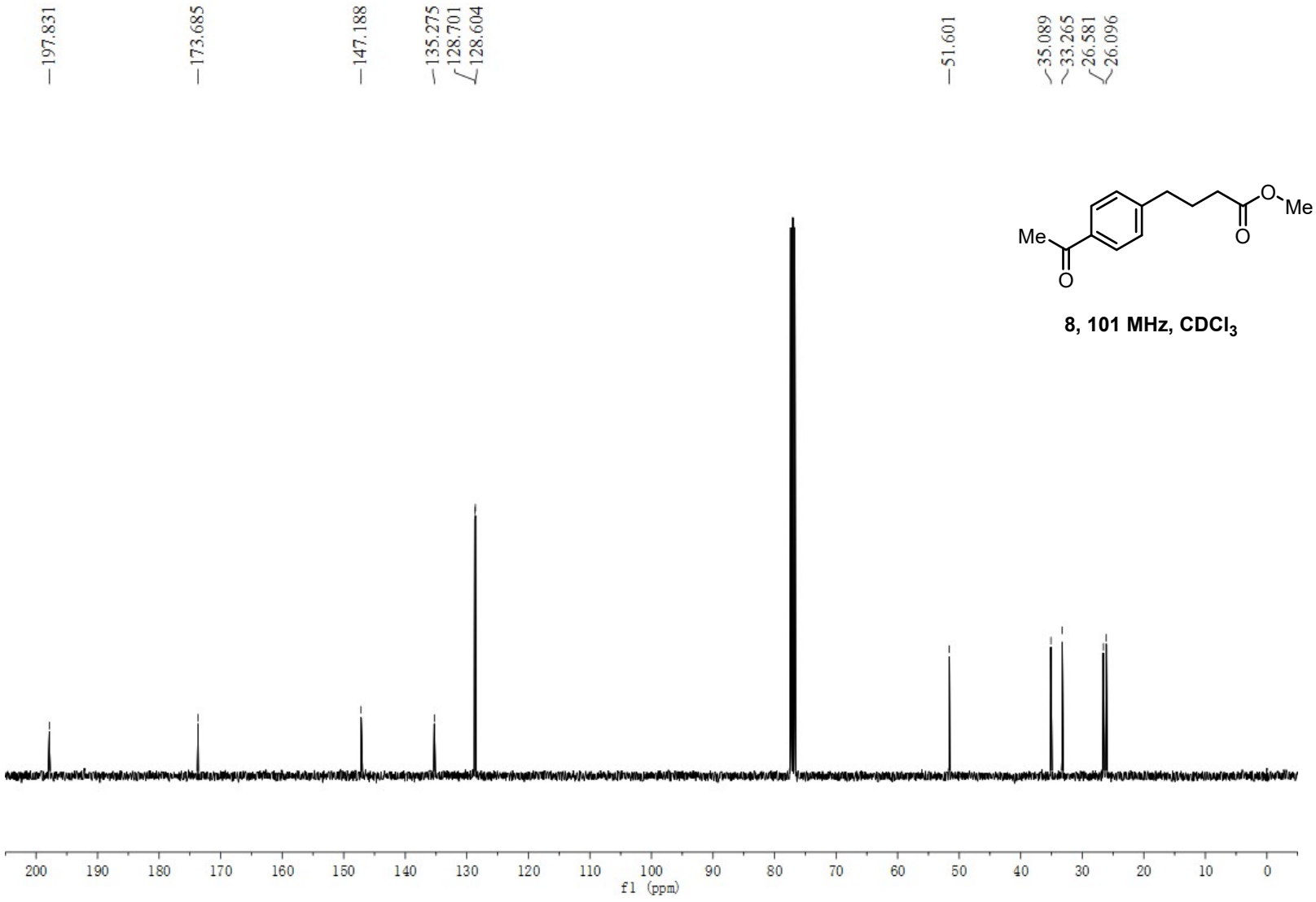


7, 400 MHz, $CDCl_3$

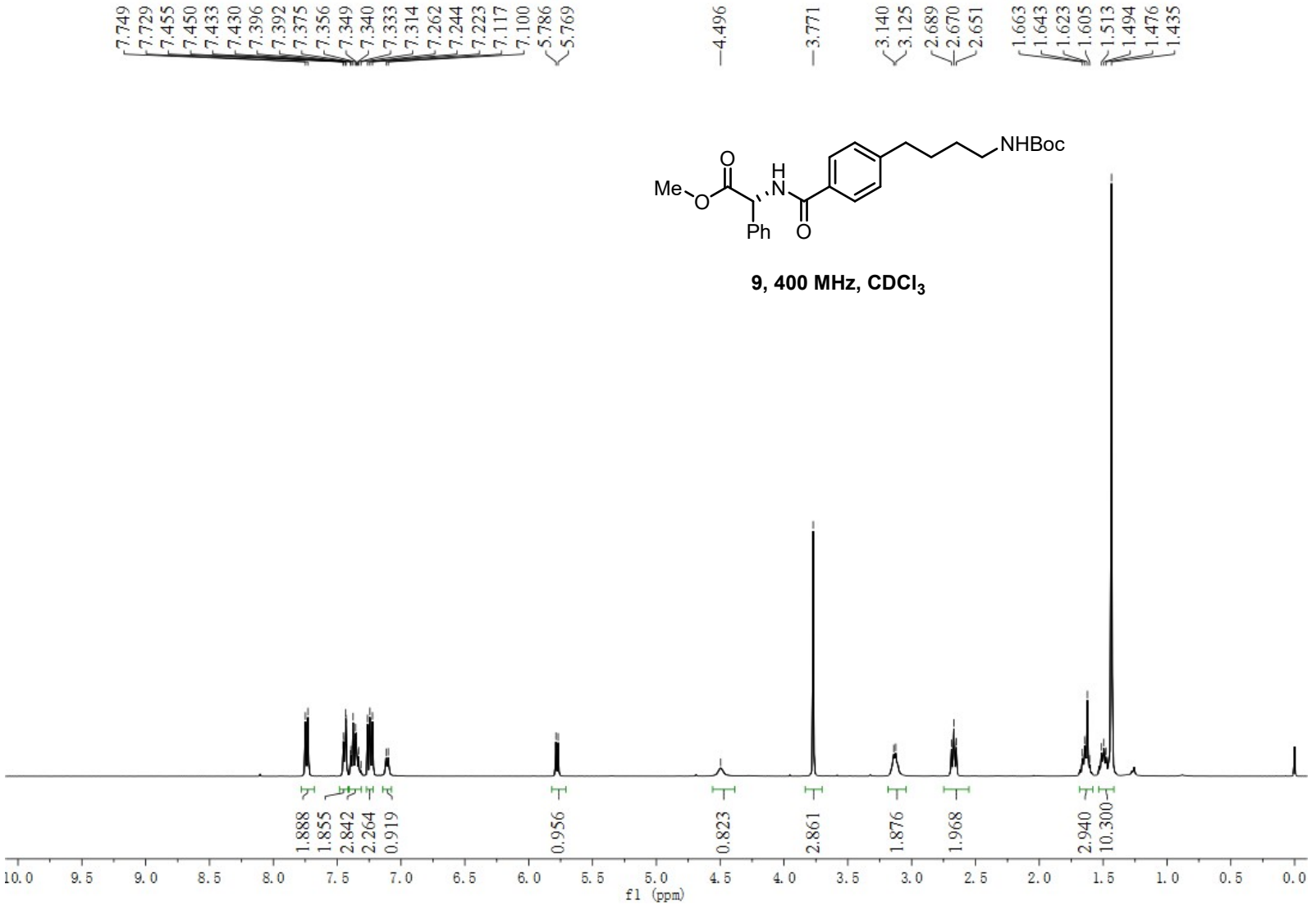


7, 101 MHz, $CDCl_3$

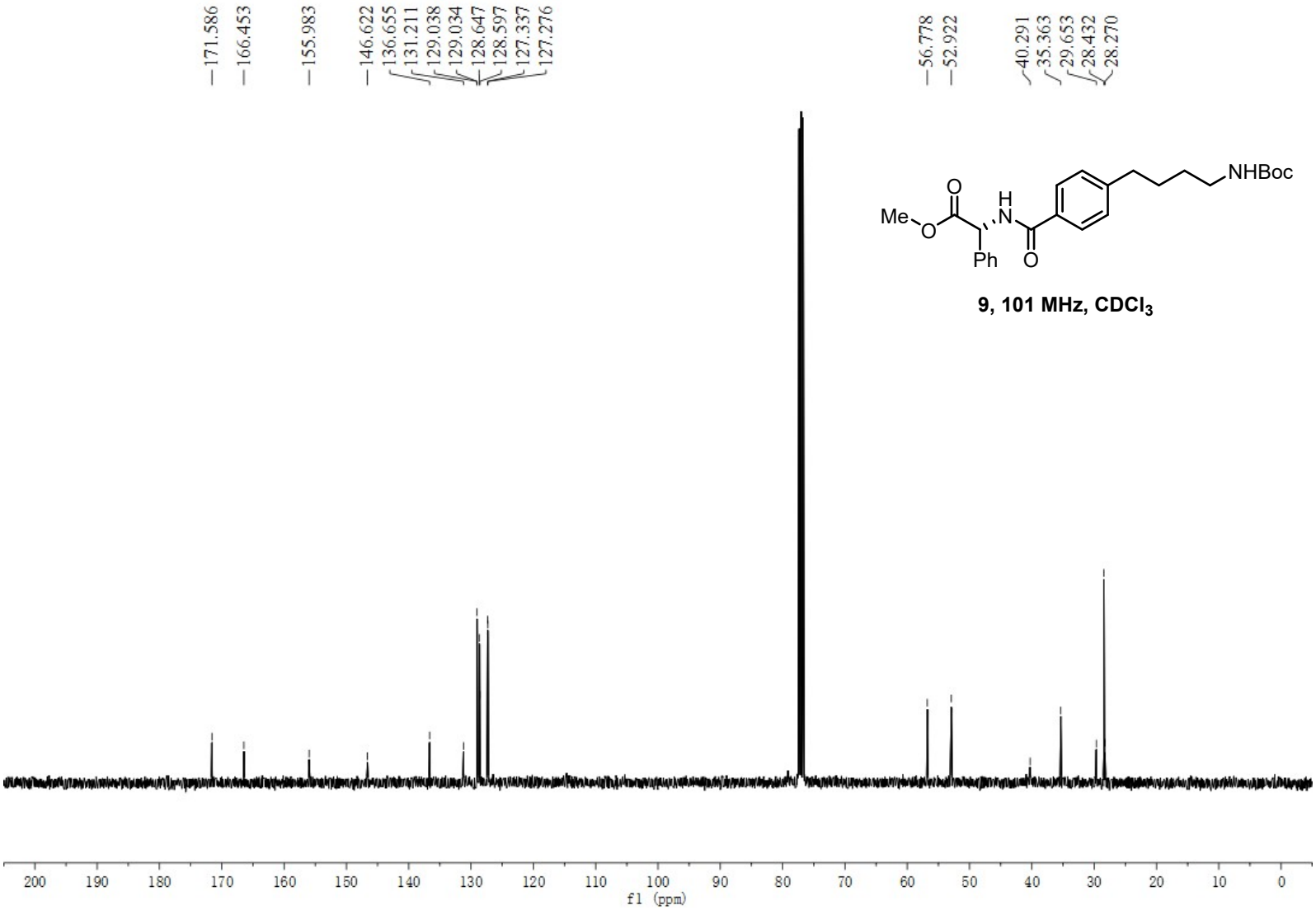

7.900
7.879
7.285
7.265
3.673
2.735
2.716
2.697
2.586
2.359
2.341
2.322
2.018
1.999
1.980
1.961
1.943
Me
O
O
Me
O
8, 400 MHz, CDCl3
2.000
2.263
3.056
2.071
2.951
2.144
2.108
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



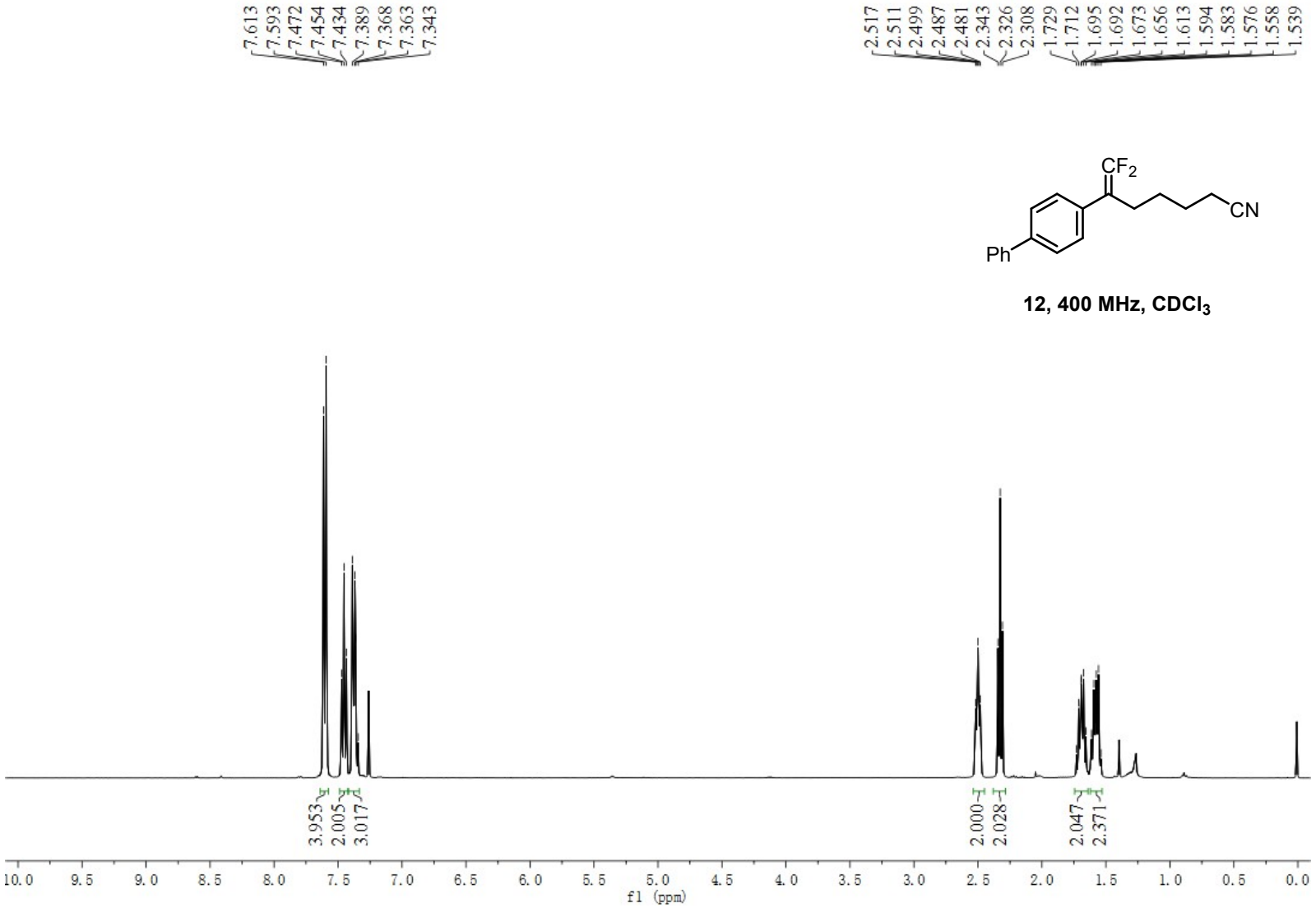
8, 101 MHz, $CDCl_3$



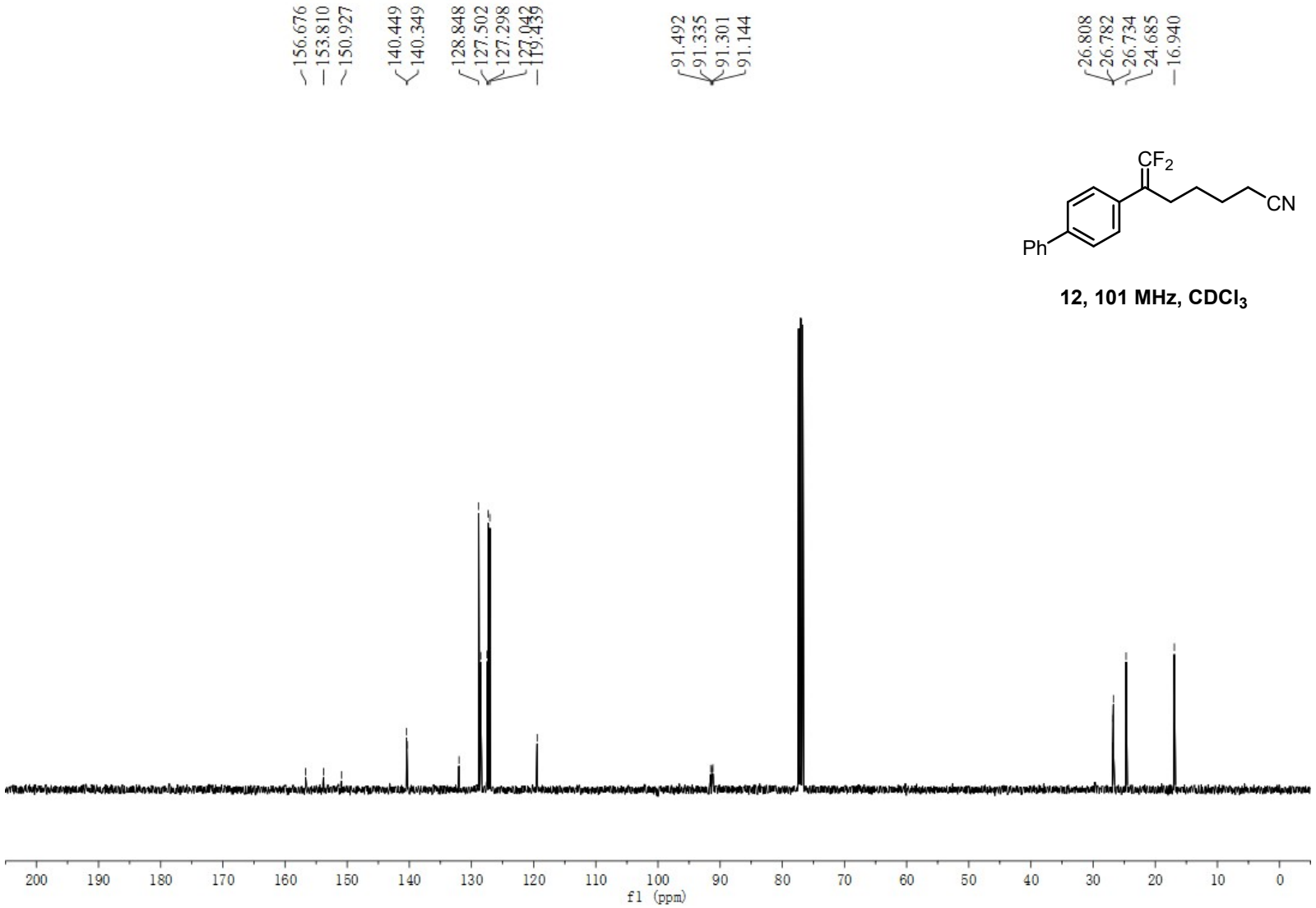
9, 400 MHz, $CDCl_3$

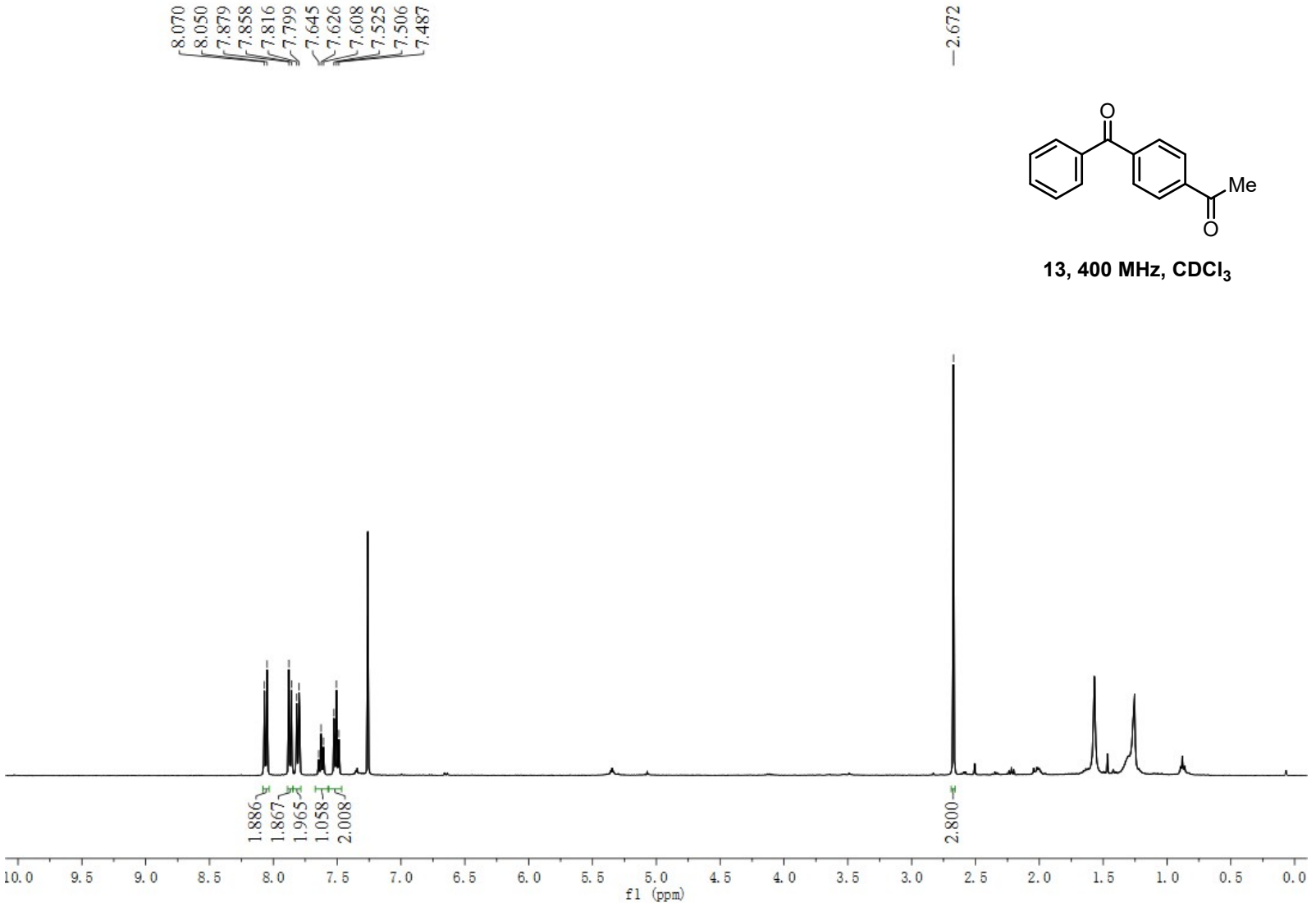


9, 101 MHz, $CDCl_3$

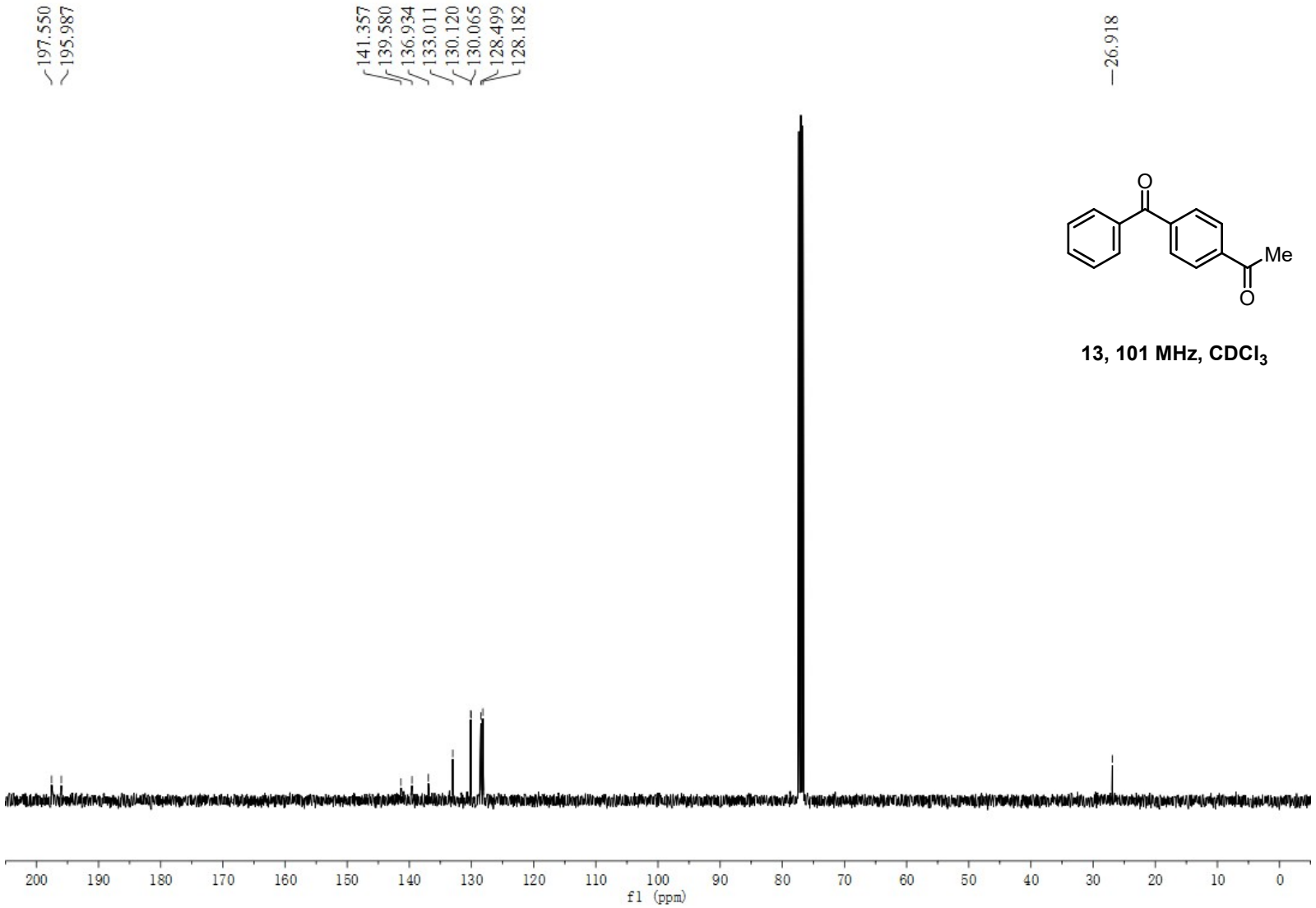


12, 400 MHz, $CDCl_3$


156.676
153.810
150.927
140.449
140.349
128.848
127.502
127.298
127.042
119.435
91.492
91.335
91.301
91.144
26.808
26.782
26.734
24.685
16.940
CF2
CN
Ph
12, 101 MHz, CDCl3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



13, 400 MHz, $CDCl_3$



13, 101 MHz, $CDCl_3$