Supplementary Information (SI) for Organic Chemistry Frontiers.

SUPPORTING INFORMATION

Metal-Free Divergent Radical Reactions of *N*-Sulfonyl Ynamides: Substrate-Controlled Synthesis of *α*-Sulfonyl Amides and *α*-Sulfonyl Nitriles

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Part I Experimental Part

General Information

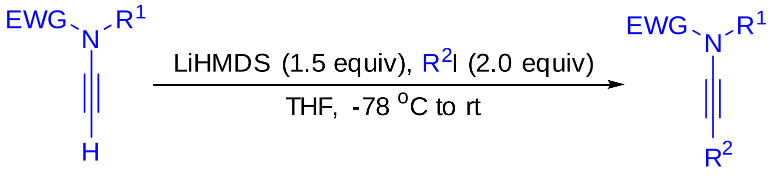
Unless otherwise indicated, all starting materials were obtained from commercial supplies and used as received. All reactions were performed in oven-dried glassware under a nitrogen atmosphere. Solvents were distilled before use. Chromatographic separations were performed using 200~300 mesh silica gel. ^{1}H NMR and ^{13}C NMR spectra were obtained on a Bruker's Ascend$^{\mathrm{TM}}$ 400 NMR spectrometer using $CDCl_3$ as solvent with TMS or residual solvent as standard unless otherwise noted. ^{13}C NMR (100 MHz) spectra were reported in ppm with the internal chloroform signal at 77.2 ppm as a standard. Infrared spectra were obtained on a PerkinElmer FT/IR spectrophotometer, and relative intensities are expressed qualitatively as s (strong), m (medium), and w (weak). TLC analysis was performed using 254 nm polyester-backed plates and visualized using UV and $KMnO_4$ stain. High-resolution mass spectra (HRMS) were performed on a Bruker MicrOTOF-Q II mass spectrometer.

1.1 Synthesis of ynamides 1 and 1′

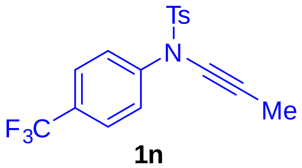
The known ynamides **1a**[1], **1b**[1], **1c**[1], **1d**[1], **1e**[11], **1f**[11], **1g**[1], **1h**[7], **1i**[7], **1j**[7], **1k**[7], **1l**[7], **1m**[7], **1o**[7], **1p**[7], **1u**[1], **1v**[5], **1w**[3], **1x**[10], **1z**[4], **1aa**[6], **1ab**[3], **1ac**[1], **1ad**[2], **1ae**[7], **1af**[7], **1ag**[7], **1ah**[7], **1ai**[7], **1aj**[8], **1ak**[7], **1al**[7], **1am**[7], **1an**[7], **1ap**[9], **1aq**[2], **1ar**[7], **1a′**[1], **1f′**[16], **1g′**[14], **1h′**[2], **1i′**[13], **1k′**[14] and **1l′**[15] were synthesized according to previously reported procedures, with analytical data consistent with literature values.

The novel ynamides **1n, 1q, 1r, 1s, 1t, 1y, 1ao, 1b′**, **1c′**, **1d′**, **1e′**, **1j′**, **1m′**, and **1n′** were prepared according to literatures.[7,10,17]

Synthesis of ynamides 1n, 1q-1t, 1b′-1e′, 1j′, 1m′, and 1n′[7]



S1 **1** or **1'**

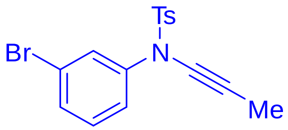


1n

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide

S1 (678.7 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 30:1~20:1 petroleum ether/EtOAc] to afford ynamide **1n** (473.5 mg, 1.34 mmol) in 67% yield.

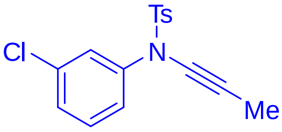
1n: R_f = 0.23 [20:1 petroleum ether/EtOAc]; white solid; mp = 104–105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, 4H, J = 8.3, 3.1 Hz), 7.43 (d, 2H, J = 8.0 Hz), 7.29 (d, 2H, J = 7.7 Hz), 2.44 (s, 3H), 1.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.3, 142.5, 132.9, 129.8, 129.7 (q, $J_{C\text{-}F}$ = 32.5 Hz), 128.2, 126.3 (q, $J_{C\text{-}F}$ = 3.8 Hz), 125.8, 123.9 (q, $J_{C\text{-}F}$ = 270.5 Hz), 72.0, 67.4, 21.9, 3.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.5; IR (neat) (cm^{-1}) 2048m, 1612w, 1372m, 1320s, 1162s, 1065s, 907m, 813s; HRMS (ESI): m/z calcd for $C_{17}H_{15}F_3NO_2S$ [M + H]$^+$ 354.0770, found 354.0773.



1q

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (700.5 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1q** (633.8 mg, 1.74 mmol) in 87% yield.

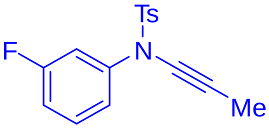
1q: R_f = 0.41 [10:1 petroleum ether/EtOAc]; white solid; mp = 124–125 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, 2H, J = 8.4 Hz), 7.47-7.37 (m, 2H), 7.31-7.23 (m, 3H), 7.18 (t, 1H, J = 8.3 Hz), 2.44 (s, 3H), 1.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 140.6, 132.8, 131.0, 130.3, 129.7, 128.9, 128.3, 124.8, 122.2, 72.2, 66.9, 21.9, 3.5; IR (neat) (cm^{-1}) 2356w, 1583m, 1369s, 1288m, 1165s, 924m, 817s, 665s; HRMS (ESI): m/z calcd for $C_{16}H_{15}BrNO_2S$ $[M + H]^+$ 364.0001, found 364.0007.



1r

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (611.6 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 µL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1r** (524.5 mg, 1.64 mmol) in 82% yield.

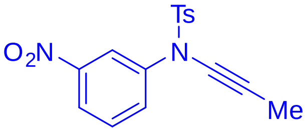
1r: R_f = 0.38 [10:1 petroleum ether/EtOAc]; white solid; mp = 120–121 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, 2H, J = 8.4 Hz), 7.32-7.19 (m, 6H), 2.44 (s, 3H), 1.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 140.5, 134.5, 132.9, 130.0, 129.7, 128.3, 128.1, 126.1, 124.3, 72.2, 66.9, 21.9, 3.5; IR (neat) (cm^{-1}) 2260w, 1588m, 1369s, 1265w, 1168s, 931s, 889m, 782m; HRMS (ESI): m/z calcd for $C_{16}H_{15}ClNO_2S$ $[M + H]^+$ 320.0507, found 320.0508.



1s

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (578.6 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 µL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1s** (473.2 mg, 1.56 mmol) in 78% yield.

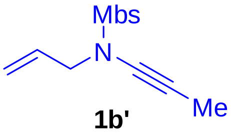
1s: R_f = 0.41 [10:1 petroleum ether/EtOAc]; white solid; mp = 85–86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, 2H, J = 8.2 Hz), 7.31-7.26 (m, 3H), 7.11 (d, 1H, J = 8.4 Hz), 7.07-6.89 (m, 2H), 2.44 (s, 3H), 1.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, $J_{C\text{-}F}$ = 245.8 Hz), 145.2, 140.8 (d, $J_{C\text{-}F}$ = 9.8 Hz), 132.9, 130.2 (d, $J_{C\text{-}F}$ = 8.9 Hz), 129.7, 128.3, 121.6 (d, $J_{C\text{-}F}$ = 3.3 Hz), 114.9 (d, $J_{C\text{-}F}$ = 21.0 Hz), 113.3 (d, $J_{C\text{-}F}$ = 24.2 Hz), 72.2, 66.9, 21.9, 3.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.2; IR (neat) (cm^{-1}) 2350w, 1592m, 1479m, 1361m, 1169s, 1226m, 890m, 874s; HRMS (ESI): m/z calcd for $C_{16}H_{15}FNO_2S$ $[M + H]^+$ 304.0802, found 304.0804.



1t

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (632.7 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1t** (409.6 mg, 1.24 mmol) in 62% yield.

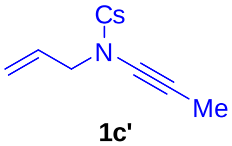
1t: R_f = 0.27 [10:1 petroleum ether/EtOAc]; white solid; mp = 131–132 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (ddd, 1H, J = 8.2, 2.2, 1.0 Hz), 8.09 (t, 1H, J = 2.1 Hz), 7.76 (ddd, 1H, J = 8.1, 2.2, 1.0 Hz), 7.58-7.51 (m, 3H), 7.30 (d, 2H, J = 8.7 Hz), 2.45 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 145.7, 140.7, 132.6, 132.0, 129.93, 129.91, 128.2, 122.5, 120.3, 71.6, 68.1, 21.9, 3.5; IR (neat) (cm^{-1}) 2930m, 2260w, 1530s, 1369m, 1171s, 1083m, 917m, 815m; HRMS (ESI): m/z calcd for $C_{16}H_{15}N_2O_4S$ $[M + H]^+$ 331.0747, found 331.0747.



1b'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (502.6 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1b′** (451.1 mg, 1.70 mmol) in 85% yield.

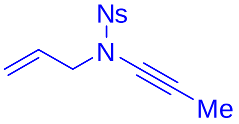
1b′: R_f = 0.26 [10:1 petroleum ether/EtOAc]; white solid; mp = 51–52 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, 2H, J = 8.9 Hz), 6.93 (d, 2H, J = 9.0 Hz), 5.70-5.58 (m, 1H), 5.18-5.07 (m, 2H), 3.82 (d, 2H, J = 6.2 Hz), 3.80 (s, 3H), 1.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6, 131.3, 129.9, 129.2, 119.6, 114.3, 72.1, 65.8, 55.7, 54.2, 3.3; IR (neat) (cm^{-1}) 2920w, 2255w, 1595s, 1496s, 1302m, 1161s, 936s, 829s; HRMS (ESI): m/z calcd for $C_{13}H_{16}NO_3S$ $[M + H]^+$ 266.0845, found 266.0849.



1c'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (511.4 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1c′** (442.4 mg, 1.64 mmol) in 82% yield.

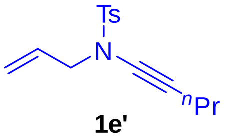
1c′: R_f = 0.52 [10:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, 2H, J = 8.7 Hz), 7.53 (d, 2H, J = 8.7 Hz), 5.77-5.66 (m, 1H), 5.27-5.18 (m, 2H), 3.94 (d, 2H, J = 6.3 Hz), 1.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.2, 136.4, 131.0, 129.6, 129.3, 120.1, 71.6, 66.4, 54.5, 3.4; IR (neat) (cm^{-1}) 3094w, 2261m, 1583m, 1477s, 1360s, 1235m, 988s, 838s; HRMS (ESI): m/z calcd for $C_{12}H_{13}ClNO_2S$ [M + H]$^+$ 270.0350, found 270.0352.



1d'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (532.5 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 µL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1d′** (353.2 mg, 1.26 mmol) in 63% yield.

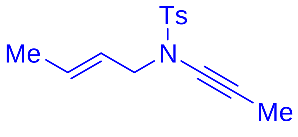
1d′: R_f = 0.35 [10:1 petroleum ether/EtOAc]; white solid; mp = 71–72 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, 2H, J = 8.9 Hz), 8.09 (d, 2H, J = 8.9 Hz), 5.77-5.65 (m, 1H), 5.29-5.19 (m, 2H), 4.00 (d, 2H, J = 6.3 Hz), 1.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.7, 143.4, 130.6, 129.1, 124.5, 120.5, 71.0, 67.0, 54.8, 3.4; IR (neat) (cm^{-1}) 2260w, 1527s, 1440m, 1345s, 1170s, 991m, 831s; HRMS (ESI): m/z calcd for $C_{12}H_{13}N_2O_4S$ [M + H]$^+$ 281.0591, found 281.0590.



1e'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (470.6 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodopropane (390.1 µL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1e′** (382.8 mg, 1.38 mmol) in 69% yield.

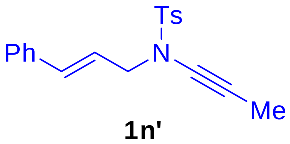
1e′: R_f = 0.50 [10:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, 2H, J = 8.3 Hz), 7.33 (d, 2H, J = 8.1 Hz), 5.78-5.65 (m, 1H), 5.25-5.15 (m, 2H), 3.91 (d, 2H, J = 6.2 Hz), 2.45 (s, 3H), 2.22 (t, 2H, J = 6.9 Hz), 1.54-1.44 (m, 2H), 0.93 (t, 3H, J = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 134.8, 131.3, 129.7, 127.8, 119.7, 73.2, 70.4, 54.4, 22.5, 21.8, 20.5, 13.5; IR (neat) (cm^{-1}) 2960m, 2252m, 1596m, 1493w, 1361s, 1166s, 987m, 813s; HRMS (ESI): m/z calcd for $C_{15}H_{20}NO_2S$ [M + H]$^+$ 278.1209, found 278.1211.



1m'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide **S1** (498.7 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 µL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1m′** (268.6 mg, 1.02 mmol) in 51% yield.

1m′: R_f = 0.33 [10:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, 2H, J = 8.4 Hz), 7.33 (d, 2H, J = 7.9 Hz), 5.69-5.60 (m, 1H), 5.40-5.32 (m, 1H), 3.84 (d, 2H, J = 6.7 Hz), 2.45 (s, 3H), 1.88 (s, 3H), 1.65 (d, 3H, J = 5.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 135.1, 131.7, 129.7, 127.8, 124.0, 72.2, 65.8, 53.9, 21.8, 17.9, 3.5; IR (neat) (cm^{-1}) 2922m, 2072w, 1363s, 1169s, 1091m, 966w, 814m; HRMS (ESI): m/z calcd for $C_{14}H_{18}NO_2S$ [M + H]$^+$ 264.1053, found 264.1053.



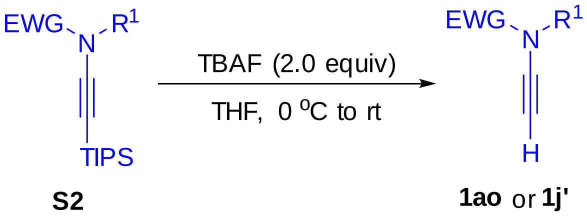
1n'

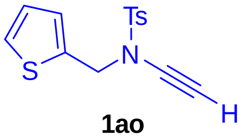
To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding ynamide

S1 (622.8 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to –78 ℃, then LiHMDS (3.0 mL, 1.0 M in THF) was added dropwise. The reaction mixture was gradually warmed to –60 °C and equilibrated for 1.0 h with vigorous stirring. At this temperature, iodomethane (249.0 μL, 4.00 mmol) was added. The resulting mixture was then allowed to warm gradually to room temperature and stirred for an additional 12.0 h under a nitrogen atmosphere. Upon confirmation of reaction completion by TLC, the mixture was quenched with distilled water (20 mL). The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1n′** (637.8 mg, 1.96 mmol) in 98% yield.

1n′: R_f = 0.25 [10:1 petroleum ether/EtOAc]; white solid; mp = 96–97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, 2H, J = 8.2 Hz), 7.31-7.24 (m, 7H), 6.49 (d, 1H, J = 15.6 Hz), 6.02 (dt, 1H, J = 15.8, 6.6 Hz), 4.08 (d, 2H, J = 6.7 Hz), 2.41 (s, 3H), 1.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 136.2, 135.0, 134.8, 129.8, 128.7, 128.1, 127.8, 126.7, 122.3, 72.2, 66.1, 54.2, 21.7, 3.5; IR (neat) (cm^{-1}) 2916w, 2261w, 1637m, 1368s, 1173m, 1407m, 991w; HRMS (ESI): m/z calcd for $C_{19}H_{20}NO_2S$ $[M + H]^+$ 326.1209, found 326.1205.

Synthesis of ynamides 1ao and 1j′[12,17]

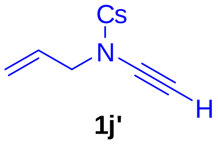




1ao

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding TIPS-protected ynamide **S2** (895.5 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to 0 ℃, then TBAF (4.0 mL of a 1.0 M solution in THF) was added dropwise. The reaction mixture was gradually warmed to room temperature and equilibrated for 0.5 h with vigorous stirring. Upon confirmation of reaction completion by TLC, the mixture was quenched with saturated ammonium chloride solution (4.0 mL), and distilled water (6.0 mL) was added. The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1ao** (495.4 mg, 1.70 mmol) in 85% yield.

1ao: R_f = 0.26 [10:1 petroleum ether/EtOAc]; white solid; mp = 68–69 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, 2H, J = 8.3 Hz), 7.30 (d, 2H, J = 7.8 Hz), 7.24 (dd, 1H, J = 5.1, 1.3 Hz), 7.00 (d, 1H, J = 3.5 Hz), 6.92 (dd, 1H, J = 5.1, 3.5 Hz), 4.73 (s, 2H), 2.76 (s, 1H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 136.3, 134.8, 129.9, 128.7, 127.9, 126.9, 126.8, 75.9, 60.6, 50.1, 21.8; IR (neat) (cm^{-1}) 2135m, 1595m, 1438w, 1356s, 1165s, 1135w, 914s; HRMS (ESI): m/z calcd for $C_{14}H_{14}NO_2S_2$ $[M + H]^+$ 292.0460, found 292.0464.

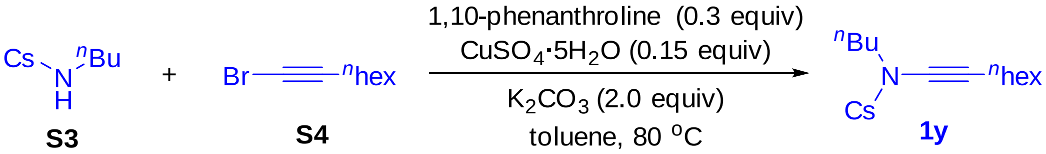


1j'

To an oven-dried round-bottom flask under N_2 atmosphere was added the corresponding TIPS-protected ynamide **S2** (824.1 mg, 2.00 mmol) and anhydrous THF (10.0 mL). The resulting solution was cooled to 0 ℃, then TBAF (4.0 mL of a 1.0 M solution in THF) was added dropwise. The reaction mixture was gradually warmed to room temperature and equilibrated for 0.5 h with vigorous stirring. Upon confirmation of reaction completion by TLC, the mixture was quenched with saturated ammonium chloride solution (4.0 mL), and distilled water (6.0 mL) was added. The aqueous phase was extracted with ethyl acetate (3 × 10.0 mL), and the combined organic extracts were washed with saturated brine, dried over anhydrous Na_2SO_4, filtered, concentrated in vacuo, and purified by flash silica gel column chromatography [gradient eluent: 15:1~10:1 petroleum ether/EtOAc] to afford ynamide **1j′** (439.8 mg, 1.72 mmol) in 86% yield.

1j′: R_f = 0.48 [10:1 petroleum ether/EtOAc]; white solid; mp = 64–65 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, 2H, J = 8.7 Hz), 7.54 (d, 2H, J = 8.6 Hz), 5.79-5.66 (m, 1H), 5.32-5.20 (m, 2H), 3.99 (d, 2H, J = 6.4 Hz), 2.76 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6, 136.1, 130.3, 129.6, 129.2, 120.6, 75.5, 59.7, 54.2; IR (neat) (cm^{-1}) 2922w, 2255w, 1594s, 1496s, 1355s, 1160s, 936m; HRMS (ESI): m/z calcd for $C_{11}H_{11}ClNO_2S$ $[M + H]^+$ 256.0194, found 256.0195.

Synthesis of ynamide 1y[10]

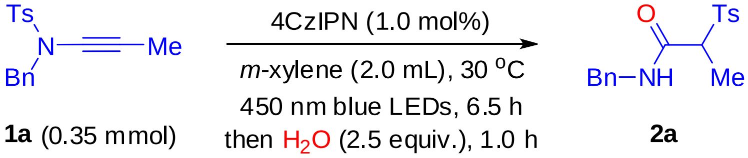


To an oven-dried round-bottom flask under N_2 atmosphere was added the sulfonamide **S3** (743.2 mg, 3.00 mmol), 1-bromooct-1-yne **S4** (680.7 mg, 3.60 mmol), 1,10-phenanthroline (162.2 mg, 0.90 mmol), $CuSO_4$•$5H_2O$ (112.4 mg, 0.45 mmol), K_2CO_3 (829.3 mg, 6.00 mmol) and toluene (15.0 mL) in sequence. The reaction was stirred at 80 ^{o}C in an oil bath for 4.0 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was cooled to room temperature, filtered through a short pad of silica gel, concentrated in vacuo, and purified by flash silica gel column chromatography [isocratic eluent: 30:1 petroleum ether/EtOAc] to afford ynamide **1y** (800.8 mg, 2.25 mmol) in 75% yield.

1y: R_f = 0.45 [30:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, 2H, J = 8.6 Hz), 7.52 (d, 2H, J = 8.7 Hz), 3.28 (t, 2H, J = 7.1 Hz), 2.26 (t, 2H, J = 6.9 Hz), 1.67-1.55 (m, 2H), 1.53-1.41 (m, 2H), 1.39-1.22 (m, 8H), 0.94-0.85 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 136.0, 129.3, 129.0, 72.6, 70.5, 51.3, 31.3, 29.8, 28.9, 28.5, 22.6, 19.4, 18.4, 14.1, 13.6; IR (neat) (cm^{-1}) 2956w, 2930m, 2251m, 1584m, 1366s, 1217w, 1171s, 825m; HRMS (ESI): m/z calcd for $C_{18}H_{27}ClNO_2S$ $[M + H]^+$ 356.1446, found 356.1451.

1.2 Reaction condition optimization for *α*-sulfonyl amide formation

Table 1. Reaction Condition Optimization for α-Sulfonyl Amide Formation



1a (0.35 mmol)

2a

Entry[a]	Variation from the standard reaction conditions	Yield (%)[b]
1	none	94
2	without 4CzIPN	NR
3	without light	NR
4	toluene instead of *m*-xylene	81
5	CH_2Cl_2 instead of *m*-xylene	78
6	DCE instead of *m*-xylene	84
7	THF instead of *m*-xylene	87
8	MeCN instead of *m*-xylene	80
9	1,4-dioxane instead of *m*-xylene	89
10	DMF instead of *m*-xylene	54
11	benzophenone instead of 4CzIPN	NR
12	405 nm instead of 450 nm LED	79
13	365 nm instead of 450 nm LED	NR
14[c]	none	90
15	thermal (130 °C), no photocatalyst/light	40
16	thermal (140 °C), no photocatalyst/light	75
17[c]	thermal (140 °C), no photocatalyst/light	73

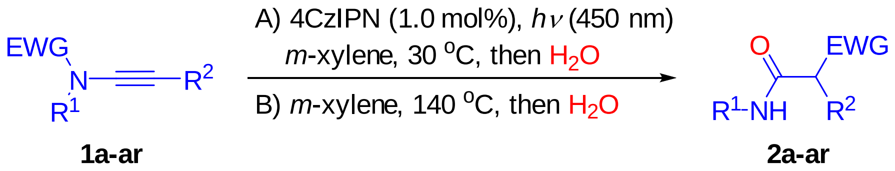
[a] Standard reaction conditions: **1a** (0.35 mmol), 4CzIPN (1.0 mol%), *m*-xylene (2.0 mL), 450 nm blue LED irradiation, 30 °C, 6.5 h, then H_2O (0.88 mmol, 2.5 equiv.), 1.0 h. [b] Isolated yields. [c] **1a** (1.00 mmol) was added. 4CzIPN = 2,4,5,6-tetra(9*H*-carbazol-9-yl)isophthalonitrile. NR = no reaction detected by TLC.

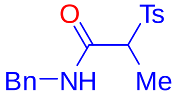
Entry 14 (1.0 mmol synthetic method): To a 10 mL Schlenk flask was added ynamide **1a** (299.4 mg, 1.0 mmol), 4CzIPN (7.9 mg, 0.01 mmol), and *m*-xylene (5.7 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. After 8 h of irradiation at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C, water (45.0 μL, 2.5 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (285.7 mg, 0.90 mmol) in 90% yield.

Entry 17 (1.0 mmol synthetic method): To an oven-dried tube was added ynamide **1a** (299.4 mg, 1.0 mmol), and *m*-xylene (5.7 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 8 h. Water (45.0 μL, 2.5 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (231.7 mg, 0.73 mmol) in 73% yield.

1.3 Substrate scope of *N*-sulfonyl ynamides in *α*-sulfonyl amides synthesis

All *α*-sulfonyl amides are novel except for **2v**, **2w**, and **2ap**, which are known compounds.



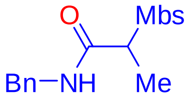


2a

Condition A: To a 10 mL Schlenk flask was added ynamide **1a** (104.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (104.5 mg, 0.33 mmol) in 94% yield.

Condition B: To an oven-dried tube was added ynamide **1a** (104.8 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 8.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (83.4 mg, 0.26 mmol) in 75% yield.

2a: R_f = 0.32 [2:1 petroleum ether/EtOAc]; white solid; mp = 141–142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.58 (d, 2H, *J* = 8.1 Hz), 7.37-7.24 (m, 7H), 7.09 (t, 1H, *J* = 5.7 Hz), 4.49-4.39 (m, 2H), 3.91 (q, 1H, *J* = 7.1 Hz), 2.42 (s, 3H), 1.50 (d, 3H, *J* = 5.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.6, 145.6, 137.6, 132.9, 130.0, 129.2, 128.8, 128.2, 127.8, 65.8, 44.2, 21.8, 12.2; IR (neat) (cm^{-1}) 3352m, 1665s, 1524m, 1288m, 1212w, 1139s, 813m, 700m; HRMS (ESI): m/z calcd for $C_{17}H_{20}NO_3S$ $[M + H]^+$ 318.1158, found 318.1163.



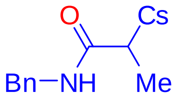
2b

Condition A: To a 10 mL Schlenk flask was added ynamide **1b** (110.4 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 7.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2b** (108.8 mg, 0.33 mmol) in 93% yield.

Condition B: To an oven-dried tube was added ynamide **1b** (110.4 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2b** (82.1 mg, 0.25 mmol) in 70% yield.

2b: R_f = 0.18 [2:1 petroleum ether/EtOAc]; white solid; mp = 139–140 °C; ^{1}H NMR (400 MHz,

$CDCl_3$) δ 7.61 (d, 2H, J = 9.0 Hz), 7.38-7.30 (m, 5H), 7.06 (t, 1H, J = 5.9 Hz), 6.93-6.89 (m, 2H), 4.48-4.41 (m, 2H), 3.92-3.86 (m, 4H), 1.51 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 164.4, 137.6, 131.4, 128.9, 128.3, 127.8, 127.3, 114.6, 66.0, 55.8, 44.3, 12.3; IR (neat) (cm^{-1}) 3353m, 1670s, 1594m, 1498m, 1318w, 1142s, 832s, 723s; HRMS (ESI): m/z calcd for $C_{17}H_{20}NO_4S$ $[M + H]^+$ 334.1108, found 334.1112.

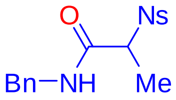


2c

Condition A: To a 10 mL Schlenk flask was added ynamide **1c** (111.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 7.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2c** (111.0 mg, 0.33 mmol) in 94% yield.

Condition B: To an oven-dried tube was added ynamide **1c** (111.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 5.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2c** (91.8 mg, 0.27 mmol) in 78% yield.

2c: R_f = 0.31 [2:1 petroleum ether/EtOAc]; white solid; mp = 149–150 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, 2H, J = 8.6 Hz), 7.43-7.29 (m, 7H), 6.98 (t, 1H, J = 5.7 Hz), 4.49-4.38 (m, 2H), 3.92 (q, 1H, J = 7.1 Hz), 1.51 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 141.4, 137.5, 134.2, 130.7, 129.7, 129.0, 128.3, 128.0, 66.0, 44.4, 12.2; IR (neat) (cm^{-1}) 3346m, 1684s, 1660s, 1581w, 1522s, 1312m, 1143s, 829m; HRMS (ESI): m/z calcd for $C_{16}H_{17}ClNO_3S$ $[M + H]^+$ 338.0612, found 338.0618.



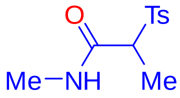
2d

Condition A: To a 10 mL Schlenk flask was added ynamide **1d** (115.6 mg, 0.35 mmol), 4CzIPN

(2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2d** (99.6 mg, 0.29 mmol) in 82% yield.

Condition B: To an oven-dried tube was added ynamide **1d** (115.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 12 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2d** (63.5 mg, 0.18 mmol) in 52% yield.

2d: R_f = 0.20 [2:1 petroleum ether/EtOAc]; white solid; mp = 160–161 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, 2H, J = 8.8 Hz), 7.82 (d, 2H, J = 8.8 Hz), 7.44-7.35 (m, 3H), 7.35-7.29 (m, 2H), 6.90 (t, 1H, J = 6.1 Hz), 4.50-4.37 (m, 2H), 3.97 (q, 1H, J = 7.1 Hz), 1.56 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 151.2, 141.3, 137.3, 130.9, 129.1, 128.5, 128.2, 124.4, 66.3, 44.5, 12.1; IR (neat) (cm^{-1}) 3340m, 1684s, 1532s, 1347m, 1297s, 1211w, 855m, 703s; HRMS (ESI): m/z calcd for $C_{16}H_{17}N_2O_5S$ [M + H]$^+$ 349.0853, found 349.0857.

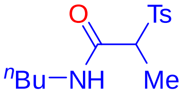


2e

Condition A: To a 10 mL Schlenk flask was added ynamide **1e** (78.2 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 5.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~1:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2e** (68.1 mg, 0.28 mmol) in 81% yield.

Condition B: To an oven-dried tube was added ynamide **1e** (78.2 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 9 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~1:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2e** (50.1 mg, 0.21 mmol) in 59% yield.

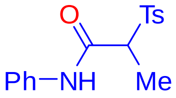
2e: R_f = 0.22 [1:1 petroleum ether/EtOAc]; white solid; mp = 140–141 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.72 (d, 2H, *J* = 8.3 Hz), 7.36 (d, 2H, *J* = 8.1 Hz), 6.79 (s, 1H), 3.92 (q, 1H , *J* = 7.2 Hz), 2.85 (d, 3H, *J* = 4.9 Hz), 2.45 (s, 3H), 1.49 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 165.3, 145.7, 133.1, 130.0, 129.2, 65.8, 27.0, 21.8, 12.3; IR (neat) (cm^{-1}) 1640s, 1559m, 1449m, 1315s, 1296w, 1146s, 815m, 727s; HRMS (ESI): m/z calcd for $C_{11}H_{16}NO_3S$ $[M + H]^+$ 242.0845, found 242.0849.


2f

Condition A: To a 10 mL Schlenk flask was added ynamide **1f** (92.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2f** (82.4 mg, 0.29 mmol) in 83% yield.

Condition B: To an oven-dried tube was added ynamide **1f** (92.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 °C with stirring for 5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2f** (70.6 mg, 0.25 mmol) in 71% yield.

2f: R_f = 0.23 [4:1 petroleum ether/EtOAc]; white solid; mp = 81–82 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.72 (d, 2H, *J* = 8.3 Hz), 7.36 (d, 2H, *J* = 8.0 Hz), 6.67 (t, 1H, *J* = 6.4 Hz), 3.86 (q, 1H, *J* = 7.2 Hz), 3.27 (q, 2H, *J* = 6.9 Hz), 2.45 (s, 3H), 1.56-1.48 (m, 5H), 1.43-1.32 (m, 2H), 0.94 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.6, 145.7, 133.3, 130.0, 129.2, 65.9, 40.1, 31.5, 21.9, 20.2, 13.9, 12.3; IR (neat) (cm^{-1}) 3363m, 2957w, 1662s, 1524m, 1302m, 1141s, 998w, 814m; HRMS (ESI): m/z calcd for $C_{14}H_{22}NO_3S$ $[M + H]^+$ 284.1315, found 284.1321.

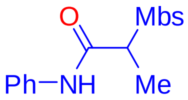


2g

Condition A: To a 10 mL Schlenk flask was added ynamide **1g** (99.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 12 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2g** (85.4 mg, 0.28 mmol) in 80% yield.

Condition B: To an oven-dried tube was added ynamide **1g** (99.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2g** (99.0 mg, 0.33 mmol) in 93% yield.

2g: R_f = 0.22 [4:1 petroleum ether/EtOAc]; white solid; mp = 160–161 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (s, 1H), 7.73 (d, 2H, J = 8.4 Hz), 7.51 (d, 2H, J = 7.4 Hz), 7.34 (t, 4H, J = 7.9 Hz), 7.15 (t, 1H, J = 7.4 Hz), 4.07 (q, 1H, J = 7.1 Hz), 2.43 (s, 3H), 1.58 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 146.0, 137.4, 132.7, 130.2, 129.28, 129.27, 125.2, 120.2, 66.8, 21.9, 12.1; IR (neat) (cm^{-1}) 3313m, 1659s, 1490m, 1342m, 1247w, 1133s, 813s, 766s; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_3S$ $[M + H]^+$ 304.1002, found 304.1006.

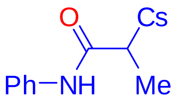


2h

Condition A: To a 10 mL Schlenk flask was added ynamide **1h** (105.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 9 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2h** (95.0 mg, 0.30 mmol) in 85% yield.

Condition B: To an oven-dried tube was added ynamide **1h** (105.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2h** (99.3 mg, 0.31 mmol) in 89% yield.

2h: R_f = 0.35 [2:1 petroleum ether/EtOAc]; white solid; mp = 154–155 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.53 (s, 1H), 7.77 (d, 2H, *J* = 8.9 Hz), 7.52 (d, 2H, *J* = 7.5 Hz), 7.35 (t, 2H, *J* = 7.8 Hz), 7.16 (t, 1H, *J* = 7.3 Hz), 6.99 (d, 2H, *J* = 8.9 Hz), 4.06 (q, 1H, *J* = 7.0 Hz), 3.86 (s, 3H), 1.59 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, CDCl$_3$) *δ* 164.6, 162.7, 137.4, 131.5, 129.3, 127.0, 125.2, 120.2, 114.8, 66.9, 55.9, 12.1; IR (neat) (cm^{-1}) 3321m, 1659m, 1572w, 1492s, 1264s, 1131s, 806m, 765m; HRMS (ESI): m/z calcd for C$_{16}$H$_{18}$NO$_4$S $[M + H]^+$ 320.0951, found 320.0955.

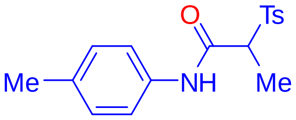


2i

Condition A: To a 10 mL Schlenk flask was added ynamide **1i** (107.0 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 7.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2i** (100.7 mg, 0.31 mmol) in 89% yield.

Condition B: To an oven-dried tube was added ynamide **1i** (107.0 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 3 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2i** (105.5 mg, 0.33 mmol) in 93% yield.

2i: R_f = 0.22 [4:1 petroleum ether/EtOAc]; white solid; mp = 128–129 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.41 (s, 1H), 7.78 (d, 2H, *J* = 8.6 Hz), 7.52 (dd, 4H, *J* = 8.0, 6.0 Hz), 7.36 (t, 2H, *J* = 8.0 Hz), 7.18 (t, 1H, *J* = 7.4 Hz), 4.08 (q, 1H, *J* = 7.1 Hz), 1.59 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 162.2, 141.8, 137.2, 134.1, 130.8, 129.9, 129.4, 125.4, 120.2, 66.9, 12.1; IR (neat) (cm^{-1}) 1653m, 1540m, 1444m, 1357w, 1322s, 1149s, 817m, 752s; HRMS (ESI): m/z calcd for $C_{15}H_{15}ClNO_3S$ $[M + H]^+$ 324.0456, found 324.0460.

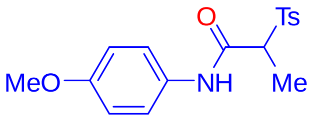


2j

Condition A: To a 10 mL Schlenk flask was added ynamide **1j** (104.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 5.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2j** (98.6 mg, 0.31 mmol) in 89% yield.

Condition B: To an oven-dried tube was added ynamide **1j** (104.8 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2j** (101.3 mg, 0.32 mmol) in 91% yield.

2j: R_f = 0.43 [2:1 petroleum ether/EtOAc]; white solid; mp = 163–164 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.49 (s, 1H), 7.72 (d, 2H, *J* = 8.0 Hz), 7.36 (dd, 4H, *J* = 26.9, 8.0 Hz), 7.13 (d, 2H, *J* = 8.0 Hz), 4.07 (q, 1H, *J* = 6.9 Hz), 2.42 (s, 3H), 2.32 (s, 3H), 1.57 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 162.4, 145.9, 134.83, 134.80, 132.7, 130.1, 129.7, 129.3, 120.2, 66.7, 21.9, 21.1, 12.1; IR (neat) (cm^{-1}) 3345s, 2948w, 1678s, 1512s, 1443w, 1290s, 1143s, 915m; HRMS (ESI): m/z calcd for $C_{17}H_{20}NO_3S$ $[M + H]^+$ 318.1158, found 318.1163.

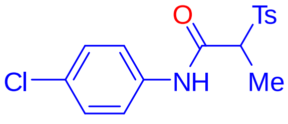


2k

Condition A: To a 10 mL Schlenk flask was added ynamide **1k** (110.4 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 7.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2k** (103.8 mg, 0.31 mmol) in 89% yield.

Condition B: To an oven-dried tube was added ynamide **1k** (110.4 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2k** (106.2 mg, 0.32 mmol) in 91% yield.

2k: R_f = 0.28 [2:1 petroleum ether/EtOAc]; white solid; mp = 140–141 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.73 (d, 2H, J = 8.4 Hz), 7.41 (d, 2H, J = 9.0 Hz), 7.33 (d, 2H, J = 8.1 Hz), 6.84 (d, 2H, J = 8.9 Hz), 4.10 (q, 1H, J = 7.0 Hz), 3.79 (s, 3H), 2.42 (s, 3H), 1.55 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 156.9, 145.9, 132.7, 130.5, 130.1, 129.3, 122.0, 114.3, 66.6, 55.6, 21.8, 12.2; IR (neat) (cm^{-1}) 3340m, 2946w, 1675s, 1544s, 1411m, 1292s, 918m, 725s; HRMS (ESI): m/z calcd for $C_{17}H_{20}NO_4S$ [M + H]$^+$ 334.1108, found 334.1113.

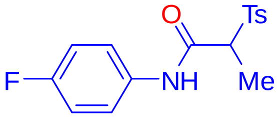


2l

Condition A: To a 10 mL Schlenk flask was added ynamide **1l** (111.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 10.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2l** (92.7 mg, 0.27 mmol) in 78% yield.

Condition B: To an oven-dried tube was added ynamide **1l** (111.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2l** (98.1 mg, 0.29 mmol) in 83% yield.

2l: R_f = 0.42 [2:1 petroleum ether/EtOAc]; white solid; mp = 179–180 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.59 (s, 1H), 7.71 (d, 2H, *J* = 8.3 Hz), 7.48 (d, 2H, *J* = 8.9 Hz), 7.32 (dd, 4H, *J* = 19.3, 8.4 Hz), 4.08 (q, 1H, *J* = 7.1 Hz), 2.44 (s, 3H), 1.56 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 162.6, 146.2, 136.0, 132.6, 130.3, 130.2, 129.3, 129.2, 121.4, 66.7, 21.9, 12.1; IR (neat) (cm^{-1}) 3340m, 1684s, 1535s, 1489m, 1290s, 1142s, 828m, 811m; HRMS (ESI): m/z calcd for $C_{16}H_{17}ClNO_3S$ $[M + H]^+$ 338.0612, found 338.0618.



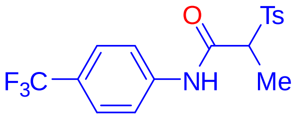
2m

Condition A: To a 10 mL Schlenk flask was added ynamide **1m** (106.2 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 39 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 5.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2m** (76.7 mg, 0.24 mmol) in 68% yield.

Condition B: To an oven-dried tube was added ynamide **1m** (106.2 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 4.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2m** (90.0 mg, 0.28

mmol) in 80% yield.

2m: R_f = 0.37 [2:1 petroleum ether/EtOAc]; white solid; mp = 165–166 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 7.73 (d, 2H, J = 8.4 Hz), 7.51-7.45 (m, 2H), 7.35 (d, 2H, J = 8.1 Hz), 7.02 (t, 2H, J = 8.7 Hz), 4.11 (q, 1H, J = 7.1 Hz), 2.44 (s, 3H), 1.56 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 159.8 (d, $J_{C\text{-}F}$ = 243.0 Hz), 146.1, 133.4 (d, $J_{C\text{-}F}$ = 2.9 Hz), 132.6, 130.2, 129.3, 122.0 (d, $J_{C\text{-}F}$ = 8.0 Hz), 115.9 (d, $J_{C\text{-}F}$ = 22.4 Hz), 66.6, 21.9, 12.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ -117.0; IR (neat) (cm^{-1}) 3354m, 2951w, 1678s, 1507s, 1344m, 1223m, 915m, 727s; HRMS (ESI): m/z calcd for $C_{16}H_{17}FNO_3S$ [M + H]$^+$ 322.0908, found 322.0913.

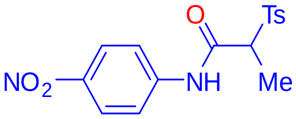


2n

Condition A: To a 10 mL Schlenk flask was added ynamide **1n** (123.7 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2n** (86.8 mg, 0.23 mmol) in 67% yield.

Condition B: To an oven-dried tube was added ynamide **1n** (123.7 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2n** (101.2 mg, 0.27 mmol) in 78% yield.

2n: R_f = 0.25 [4:1 petroleum ether/EtOAc]; white solid; mp = 164–165 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 7.72 (d, 2H, J = 8.3 Hz), 7.68-7.57 (m, 4H), 7.36 (d, 2H, J = 8.0 Hz), 4.14 (q, 1H, J = 7.1 Hz), 2.45 (s, 3H), 1.57 (d, 3H, J = 7.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 146.3, 140.4, 132.4, 130.3, 129.3, 126.9 (q, $J_{C\text{-}F}$ = 32.7 Hz), 126.5 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.1 (q, $J_{C\text{-}F}$ = 270.1 Hz), 119.8, 66.8, 21.9, 12.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.2; IR (neat) (cm^{-1}) 3322m, 2961w, 1671s, 1546s, 1368m, 1260s, 1149s, 813s; HRMS (ESI): m/z calcd for $C_{17}H_{17}F_3NO_3S$ $[M + H]^+$ 372.0876, found 372.0882.

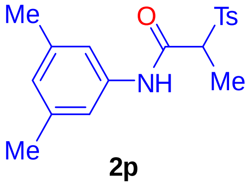


2o

Condition A: To a 10 mL Schlenk flask was added ynamide **1o** (115.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 16 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2o** (77.0 mg, 0.22 mmol) in 63% yield.

Condition B: To an oven-dried tube was added ynamide **1o** (115.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 17.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2o** (46.7 mg, 0.13 mmol) in 36% yield.

2o: R_f = 0.28 [2:1 petroleum ether/EtOAc]; white solid; mp = 199–200 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 8.24 (d, 2H, J = 9.2 Hz), 7.73 (dd, 4H, J = 10.8, 8.7 Hz), 7.37 (d, 2H, J = 7.8 Hz), 4.15 (q, 1H, J = 7.0 Hz), 2.46 (s, 3H), 1.57 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1, 146.5, 144.2, 143.1, 132.3, 130.4, 129.2, 125.3, 119.7, 66.8, 22.0, 12.1; IR (neat) (cm^{-1}) 3338m, 1699s, 1559m, 1408w, 1225m, 1138m, 857m, 736m; HRMS (ESI): m/z calcd for $C_{16}H_{17}N_2O_5S$ $[M + H]^+$ 349.0853, found 349.0858.

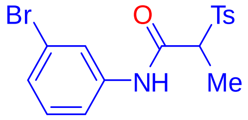


2p

Condition A: To a 10 mL Schlenk flask was added ynamide **1p** (109.7 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 8 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2p** (102.4 mg, 0.31 mmol) in 88% yield.

Condition B: To an oven-dried tube was added ynamide **1p** (109.7 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2p** (112.2 mg, 0.34 mmol) in 97% yield.

2p: R_f = 0.26 [4:1 petroleum ether/EtOAc]; white solid; mp = 172–173 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.42 (s, 1H), 7.72 (d, 2H, *J* = 8.3 Hz), 7.33 (d, 2H, *J* = 8.0 Hz), 7.15 (s, 2H), 6.80 (s, 1H), 4.02 (q, 1H, *J* = 7.1 Hz), 2.43 (s, 3H), 2.30 (s, 6H), 1.58 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 162.4, 146.0, 139.0, 137.2, 132.8, 130.2, 129.2, 126.9, 117.9, 66.8, 21.9, 21.5, 12.0; IR (neat) (cm^{-1}) 3345m, 2954w, 1686m, 1586s, 1310m, 1206w, 848s, 724w; HRMS (ESI): m/z calcd for $C_{18}H_{22}NO_3S$ [M + H]$^+$ 332.1315, found 332.1320.

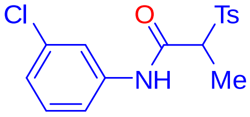


2q

Condition A: To a 10 mL Schlenk flask was added ynamide **1q** (127.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 21 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2q** (103.4 mg, 0.27 mmol) in 77% yield.

Condition B: To an oven-dried tube was added ynamide **1q** (127.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 °C with stirring for 4 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 °C for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2q** (117.9 mg, 0.31 mmol) in 88% yield.

2q: R_f = 0.20 [4:1 petroleum ether/EtOAc]; white solid; mp = 136–137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.71 (s, 1H), 7.80 (t, 1H, *J* = 2.0 Hz), 7.74 (d, 2H, *J* = 8.3 Hz), 7.41 (d, 1H, *J* = 8.1 Hz), 7.36 (d, 2H, *J* = 8.1 Hz), 7.25 (d, 1H, *J* = 8.4 Hz), 7.16 (t, 1H, *J* = 8.0 Hz), 4.16 (q, 1H, *J* = 7.1 Hz), 2.45 (s, 3H), 1.54 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 162.8, 146.2, 138.7, 132.4, 130.5, 130.2, 129.3, 128.0, 123.1, 122.8, 118.6, 66.7, 21.9, 12.2; IR (neat) (cm^{-1}) 3340m, 2953w, 1589s, 1445w, 1339m, 1142s, 864m, 776m; HRMS (ESI): m/z calcd for $C_{16}H_{17}BrNO_3S$ $[M + H]^+$ 382.0107, found 382.0114.

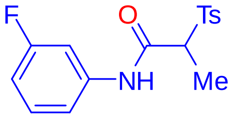


2r

Condition A: To a 10 mL Schlenk flask was added ynamide **1r** (111.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 13 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2r** (84.3 mg, 0.25 mmol) in 71% yield.

Condition B: To an oven-dried tube was added ynamide **1r** (111.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 °C with stirring for 3.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 °C for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2r** (85.9 mg, 0.25 mmol) in 73% yield.

2r: R_f = 0.25 [4:1 petroleum ether/EtOAc]; white solid; mp = 137–138 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.67 (s, 1H), 7.73 (d, 2H, *J* = 8.3 Hz), 7.66 (t, 1H, *J* = 2.1 Hz), 7.36 (d, 3H, *J* = 8.4 Hz), 7.23 (d, 1H, *J* = 8.1 Hz), 7.12 (d, 1H, *J* = 8.0 Hz), 4.13 (q, 1H, *J* = 7.0 Hz), 2.45 (s, 3H), 1.56 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, CDCl$_3$) δ 162.7, 146.2, 138.5, 134.9, 132.5, 130.3, 130.2, 129.3, 125.2, 120.3, 118.1, 66.7, 21.9, 12.1; IR (neat) (cm^{-1}) 3344m, 1686s, 1536m, 1478w, 1338w, 1144s, 809m, 738s; HRMS (ESI): m/z calcd for $C_{16}H_{17}ClNO_3S$ [M + H]$^+$ 338.0612, found 338.0618.

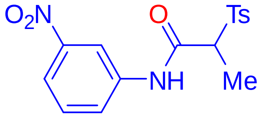


2s

Condition A: To a 10 mL Schlenk flask was added ynamide **1s** (106.2 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 13 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2s** (85.6 mg, 0.27 mmol) in 76% yield.

Condition B: To an oven-dried tube was added ynamide **1s** (106.2 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 3 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2s** (87.9 mg, 0.27 mmol) in 78% yield.

2s: R_f = 0.42 [2:1 petroleum ether/EtOAc]; white solid; mp = 123–124 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.73 (s, 1H), 7.73 (d, 2H, *J* = 8.3 Hz), 7.48 (dt, 1H, *J* = 10.8, 2.3 Hz), 7.35 (d, 2H, *J* = 8.1 Hz), 7.27 (d, 1H, *J* = 8.4 Hz), 7.18 (d, 1H, *J* = 8.1 Hz), 6.86-6.81 (m, 1H), 4.15 (dd, 1H, *J* = 7.0, 2.4 Hz), 2.44 (s, 3H), 1.55 (d, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, CDCl$_3$) δ 163.0 (d, $J_{C\text{-}F}$ = 243.6 Hz), 162.8, 146.2, 138.9 (d, $J_{C\text{-}F}$ = 10.7 Hz), 132.4, 130.3 (d, $J_{C\text{-}F}$ = 9.2 Hz), 130.2, 129.3, 115.4 (d, $J_{C\text{-}F}$ = 2.9 Hz), 111.8 (d, $J_{C\text{-}F}$ = 21.1 Hz), 107.7 (d, $J_{C\text{-}F}$ = 26.3 Hz), 66.7, 21.9, 12.2; ^{19}F NMR (376 MHz, CDCl$_3$) δ -111.2; IR (neat) (cm^{-1}) 1658s, 1543m, 1356w, 1230w, 1144s, 865w, 779m, 707m; HRMS (ESI): m/z calcd for $C_{16}H_{17}FNO_3S$ [M + H]$^+$ 322.0908, found 322.0913.

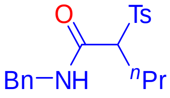


2t

Condition A: To a 10 mL Schlenk flask was added ynamide **1t** (115.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 14 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2t** (85.7 mg, 0.25 mmol) in 70% yield.

Condition B: To an oven-dried tube was added ynamide **1t** (115.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 22 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2t** (41.7 mg, 0.12 mmol) in 34% yield.

2t: R_f = 0.28 [2:1 petroleum ether/EtOAc]; white solid; mp = 194–195 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.95 (s, 1H), 8.46 (t, 1H, *J* = 2.2 Hz), 7.99 (dd, 1H, *J* = 8.3, 1.3 Hz), 7.89 (dd, 1H, *J* = 8.1, 1.2 Hz), 7.75 (d, 2H, *J* = 8.4 Hz), 7.51 (t, 1H, *J* = 8.2 Hz), 7.38 (d, 2H, *J* = 8.1 Hz), 4.16 (q, 1H, *J* = 7.1 Hz), 2.46 (s, 3H), 1.58 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 163.1, 148.7, 146.4, 138.5, 132.4, 130.4, 130.1, 129.3, 125.7, 119.7, 115.0, 66.7, 22.0, 12.2; IR (neat) (cm^{-1}) 3328m, 2927w, 1687m, 1529s, 1291m, 1144s, 737s, 675m; HRMS (ESI): m/z calcd for $C_{16}H_{17}N_2O_5S$ [M + H]$^+$ 349.0853, found 349.0859.

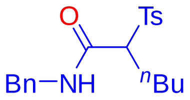


2u

Condition A: To a 10 mL Schlenk flask was added ynamide **1u** (114.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2u** (117.7 mg, 0.34 mmol) in 97% yield.

Condition B: To an oven-dried tube was added ynamide **1u** (114.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 4.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2u** (103.4 mg, 0.30 mmol) in 86% yield.

2u: R_f = 0.29 [4:1 petroleum ether/EtOAc]; white solid; mp = 155–156 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.59 (d, 2H, *J* = 8.4 Hz), 7.38-7.25 (m, 7H), 6.84 (t, 1H, *J* = 6.7 Hz), 4.43 (d, 2H, *J* = 5.9 Hz), 3.76 (dd, 1H, *J* = 11.1, 3.8 Hz), 2.43 (s, 3H), 2.03-1.85 (m, 2H), 1.46-1.28 (m, 2H), 0.92 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.3, 145.5, 137.6, 133.6, 129.9, 129.1, 128.9, 128.2, 127.8, 71.4, 44.2, 28.9, 21.9, 20.4, 13.8; IR (neat) (cm^{-1}) 2956m, 1638s, 1597w, 1437w, 1318s, 1147s, 811m, 746s; HRMS (ESI): m/z calcd for $C_{19}H_{24}NO_3S$ $[M + H]^+$ 346.1471, found 346.1475.



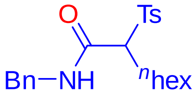
2v

Condition A: To a 10 mL Schlenk flask was added ynamide **1v** (119.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2v** (115.6 mg, 0.32 mmol) in 92% yield.

Condition B: To an oven-dried tube was added ynamide **1v** (119.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 4 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2v** (106.4 mg, 0.30 mmol) in 85% yield.

2v: R_f = 0.30 [4:1 petroleum ether/EtOAc]; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.59 (d, 2H, *J* = 8.3 Hz), 7.36-7.25 (m, 7H), 6.78 (t, 1H, *J* = 5.9 Hz), 4.43 (d, 2H, *J* = 5.9 Hz), 3.73 (dd, 1H, *J* =

11.2, 3.7 Hz), 2.43 (s, 3H), 2.08-2.00 (m, 1H), 1.95-1.85 (m, 1H), 1.39-1.28 (m, 4H), 0.87 (t, 3H, *J* = 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.4, 145.6, 137.6, 133.7, 130.0, 129.1, 128.9, 128.3, 127.9, 71.6, 44.2, 29.1, 26.6, 22.4, 21.9, 13.9. Spectral data are in agreement with literature values[18].

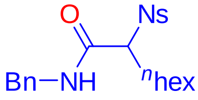


2w

Condition A: To a 10 mL Schlenk flask was added ynamide **1w** (129.3 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2w** (102.3 mg, 0.26 mmol) in 75% yield.

Condition B: To an oven-dried tube was added ynamide **1w** (129.3 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 4.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2w** (119.4 mg, 0.31 mmol) in 88% yield.

2w: R_f = 0.35 [4:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.59 (d, 2H, *J* = 8.3 Hz), 7.38-7.25 (m, 7H), 6.79 (t, 1H, *J* = 5.9 Hz), 4.43 (d, 2H, *J* = 5.9 Hz), 3.73 (dd, 1H, *J* = 11.1, 3.8 Hz), 2.43 (s, 3H), 2.06-1.98 (m, 1H), 1.94-1.84 (m, 1H), 1.37-1.20 (m, 8H), 0.85 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.3, 145.5, 137.6, 133.7, 130.0, 129.1, 128.9, 128.2, 127.9, 71.6, 44.2, 31.5, 28.9, 27.0, 26.9, 22.6, 21.9, 14.2. Spectral data are in agreement with literature values[18].

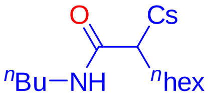


2x

Condition A: To a 10 mL Schlenk flask was added ynamide **1x** (140.2 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2x** (141.6 mg, 0.34 mmol) in 97% yield.

Condition B: To an oven-dried tube was added ynamide **1x** (140.2 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 12 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2x** (53.0 mg, 0.13 mmol) in 36% yield.

2x: R_f = 0.38 [4:1 petroleum ether/EtOAc]; white solid; mp = 157–158 ^{o}C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.25 (d, 2H, J = 8.8 Hz), 7.84 (d, 2H, J = 8.8 Hz), 7.40-7.28 (m, 5H), 6.76 (t, 1H, J = 6.0 Hz), 4.43 (d, 2H, J = 6.0 Hz), 3.81 (dd, 1H, J = 11.4, 3.6 Hz), 2.10-2.01 (m, 1H), 1.91-1.81 (m, 1H), 1.35-1.21 (m, 8H), 0.86 (t, 3H, J = 6.8 Hz); ^{13}C NMR (100 MHz, CDCl$_3$) δ 163.7, 151.1, 141.9, 137.4, 130.9, 129.0, 128.4, 128.1, 124.3, 71.9, 44.4, 31.5, 28.9, 26.95, 26.85, 22.6, 14.2; IR (neat) (cm^{-1}) 3353m, 2923w, 1669s, 1525s, 1348m, 1144s, 854m, 748m; HRMS (ESI): m/z calcd for $C_{21}H_{27}N_2O_5S$ [M + H]$^+$ 419.1635, found 419.1643.

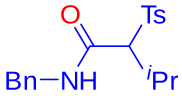


2y

Condition A: To a 10 mL Schlenk flask was added ynamide **1y** (124.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 17 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 5.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2y** (83.8 mg, 0.22 mmol) in 64% yield.

Condition B: To an oven-dried tube was added ynamide **1y** (124.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 °C with stirring for 2 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 4 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2y** (79.7 mg, 0.21 mmol) in 61% yield.

2y: R_f = 0.18 [10:1 petroleum ether/EtOAc]; white solid; mp = 125–126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.77 (d, 2H, *J* = 8.6 Hz), 7.53 (d, 2H, *J* = 8.6 Hz), 6.32 (t, 1H, *J* = 5.8 Hz), 3.70 (dd, 1H, *J* = 11.2, 3.7 Hz), 3.31-3.21 (m, 2H), 2.04-1.96 (m, 1H), 1.90-1.79 (m, 1H), 1.54-1.46 (m, 2H), 1.40-1.22 (m, 10H), 0.94 (t, 3H, *J* = 7.3 Hz), 0.86 (t, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.0, 141.4, 135.2, 130.7, 129.6, 71.9, 40.0, 31.53, 31.48, 28.9, 27.0, 26.9, 22.6, 20.2, 14.2, 13.9; IR (neat) (cm^{-1}) 3270m, 2925m, 1668s, 1638s, 1469m, 1361w, 830m, 754s; HRMS (ESI): m/z calcd for $C_{18}H_{29}ClNO_3S$ [M + H]$^+$ 374.1551, found 374.1556.

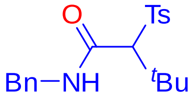


2z

Condition A: To a 10 mL Schlenk flask was added ynamide **1z** (114.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 8 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2z** (60.5 mg, 0.18 mmol) in 50% yield.

Condition B: To an oven-dried tube was added ynamide **1z** (114.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 6 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2z** (62.9 mg, 0.18 mmol) in 52% yield.

2z: R_f = 0.42 [2:1 petroleum ether/EtOAc]; white solid; mp = 152–153 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.64 (d, 2H, *J* = 8.4 Hz), 7.36-7.29 (m, 3H), 7.26-7.20 (m, 4H), 6.94 (t, 1H, *J* = 5.8 Hz), 4.42-4.29 (m, 2H), 3.66 (d, 1H, *J* = 7.2 Hz), 2.62-2.51 (m, 1H), 2.42 (s, 3H), 1.23 (d, 3H, *J* = 6.7 Hz), 1.10 (d, 3H, *J* = 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 164.1, 145.4, 137.5, 135.3, 130.0, 128.9, 128.7, 128.3, 127.9, 77.2, 44.1, 28.1, 22.1, 21.9, 20.3; IR (neat) (cm^{-1}) 3349m, 2954w, 1663s, 1521s, 1455m, 1286s, 1138s, 866m; HRMS (ESI): m/z calcd for $C_{19}H_{24}NO_3S$ [M + H]$^+$ 346.1471, found 346.1471.

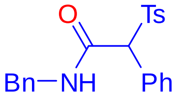


2aa

Condition A: To a 10 mL Schlenk flask was added ynamide **1aa** (119.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aa** (61.7 mg, 0.17 mmol) in 49% yield.

Condition B: To an oven-dried tube was added ynamide **1aa** (119.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 8.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aa** (56.6 mg, 0.16 mmol) in 45% yield.

2aa: R_f = 0.47 [2:1 petroleum ether/EtOAc]; white solid; mp = 150–151 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, 2H, *J* = 8.3 Hz), 7.35-7.28 (m, 3H), 7.19 (d, 4H, *J* = 9.1 Hz), 7.05 (t, 1H, *J* = 5.8 Hz), 4.30 (d, 2H, *J* = 4.5 Hz), 3.76 (s, 1H), 2.40 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 145.0, 137.4, 136.8, 129.9, 128.8, 128.39, 128.36, 127.8, 80.0, 44.1, 36.2, 29.6, 21.8; IR (neat) (cm^{-1}) 3375m, 2987w, 1667s, 1524s, 1456m, 1328s, 1139s, 827m; HRMS (ESI): m/z calcd for $C_{20}H_{26}NO_3S$ [M + H]$^+$ 360.1628, found 360.1631.

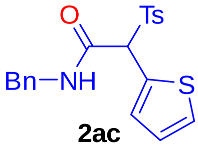


2ab

Condition A: To a 10 mL Schlenk flask was added ynamide **1ab** (126.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 20 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ab** (54.7 mg, 0.14 mmol) in 41% yield.

Condition B: To an oven-dried tube was added ynamide **1ab** (126.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 7 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ab** (48.7 mg, 0.13 mmol) in 37% yield.

2ab: R_f = 0.22 [4:1 petroleum ether/EtOAc]; white solid; mp = 218–219 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.43 (d, 2H, *J* = 8.1 Hz), 7.38-7.27 (m, 10H), 7.23 (t, 1H, *J* = 5.7 Hz), 7.17 (d, 2H, *J* = 8.0 Hz), 4.93 (s, 1H), 4.54 (d, 2H, *J* = 5.7 Hz), 2.40 (s, 3H); ^{13}C NMR (100 MHz, CDCl3) δ 163.2, 145.6, 137.5, 133.7, 130.5, 129.63, 129.61, 129.5, 128.97, 128.96, 128.8, 128.1, 127.9, 76.2, 44.4, 21.9; IR (neat) (cm^{-1}) 3356m, 2850w, 1669s, 1514m, 1285m, 1142s, 805s, 744s; HRMS (ESI): m/z calcd for $C_{22}H_{22}NO_3S$ $[M + H]^+$ 380.1315, found 380.1323.

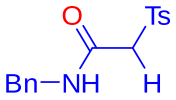


2ac

Condition A: To a 10 mL Schlenk flask was added ynamide **1ac** (128.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 9.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ac** (24.3 mg, 0.06 mmol) in 18% yield.

Condition B: To an oven-dried tube was added ynamide **1ac** (128.6 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 12 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ac** (11.0 mg, 0.03 mmol) in 8% yield.

2ac: R_f = 0.20 [4:1 petroleum ether/EtOAc]; white solid; mp = 214–215 °C; ^{1}H NMR (400 MHz, DMSO) δ 8.87 (t, 1H, *J* = 5.8 Hz), 7.56 (d, 1H, *J* = 6.4 Hz), 7.47 (d, 2H, *J* = 8.4 Hz), 7.42-7.19 (m, 6H), 7.15 (d, 2H, *J* = 6.5 Hz), 7.03- 6.96 (m, 2H), 4.36 (dd, 1H, *J* = 15.1, 3.9 Hz), 4.20 (dd, 1H, *J* = 15.2, 3.3 Hz), 2.40 (s, 3H); ^{13}C NMR (100 MHz, DMSO) *δ* 163.5, 145.4, 138.5, 134.0, 130.6, 129.8, 129.7, 129.5, 128.8, 127.79, 127.78, 127.5, 126.8, 70.6, 43.1, 21.6; IR (neat) (cm^{-1}) 3350m, 3180m, 2916s, 2848m, 1644m, 1139m, 813m, 696m; HRMS (ESI): m/z calcd for $C_{20}H_{20}NO_3S_2$ [M + H]$^+$ 386.0879, found 386.0883.

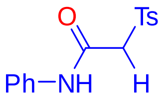


2ad

Condition A: To a 10 mL Schlenk flask was added ynamide **1ad** (99.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 4 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ad** (95.2 mg, 0.31 mmol) in 90% yield.

Condition B: To an oven-dried tube was added ynamide **1ad** (99.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ad** (56.0 mg, 0.18 mmol) in 53% yield.

2ad: R_f = 0.17 [2:1 petroleum ether/EtOAc]; white solid; mp = 143–144 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.67 (d, 2H, *J* = 8.3 Hz), 7.37-7.26 (m, 7H), 7.09 (t, 1H, *J* = 5.8 Hz), 4.43 (d, 2H, *J* = 5.9 Hz), 4.01 (s, 2H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.7, 145.8, 137.4, 135.1, 130.2, 128.9, 128.3, 128.2, 127.9, 62.1, 44.2, 21.9; IR (neat) (cm^{-1}) 3353s, 2923w, 1594m, 1492w, 1358w, 1287s, 900m, 809s; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_3S$ [M + H]$^+$ 304.1002, found 304.1006.

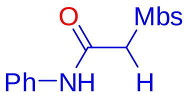


2ae

Condition A: To a 10 mL Schlenk flask was added ynamide **1ae** (95.0 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 2 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 4 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ae** (92.7 mg, 0.32 mmol) in 92% yield.

Condition B: To an oven-dried tube was added ynamide **1ae** (95.0 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 0.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 4.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ae** (12.3 mg, 0.04 mmol) in 12% yield.

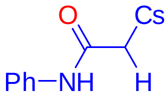
2ae: R_f = 0.29 [2:1 petroleum ether/EtOAc]; white solid; mp = 159–160 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 7.80 (d, 2H, J = 8.2 Hz), 7.50 (d, 2H, J = 7.4 Hz), 7.40-7.30 (m, 4H), 7.20-7.11 (m, 1H), 4.17 (s, 2H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 146.1, 137.1, 135.0, 130.4, 129.3, 128.3, 125.4, 120.3, 63.2, 21.9; IR (neat) (cm^{-1}) 3327s, 1661s, 1541s, 1404m, 1235w, 1075s, 907m, 766s; HRMS (ESI): m/z calcd for $C_{15}H_{16}NO_3S$ [M + H]$^+$ 290.0845, found 290.0849.



2af

Condition A: To a 10 mL Schlenk flask was added ynamide **1af** (100.6 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 2 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2af** (78.9 mg, 0.26 mmol) in 74% yield.

2af: R_f = 0.17 [2:1 petroleum ether/EtOAc]; white solid; mp = 138–139 $^{\circ}$C; ^{1}H NMR (400 MHz, CDCl$_3$) *δ* 8.56 (s, 1H), 7.84 (d, 2H, *J* = 8.9 Hz), 7.50 (d, 2H, *J* = 7.4 Hz), 7.34 (t, 2H, *J* = 7.9 Hz), 7.16 (t, 1H, *J* = 7.4 Hz), 7.01 (d, 2H, *J* = 8.9 Hz), 4.15 (s, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$) *δ* 164.6, 158.9, 137.1, 130.6, 129.31, 129.32, 125.4, 120.3, 115.0, 63.4, 55.9; IR (neat) (cm^{-1}) 3320m, 2344w, 1661s, 1441m, 1311m, 1260s, 1147s, 911m; HRMS (ESI): m/z calcd for $C_{15}H_{16}NO_4S$ $[M + H]^+$ 306.0795, found 306.0799.

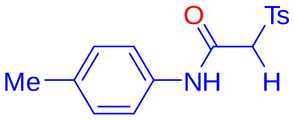


2ag

Condition A: To a 10 mL Schlenk flask was added ynamide **1ag** (102.1 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 0.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 $^{\circ}$C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ag** (101.3 mg, 0.33 mmol) in 93% yield.

Condition B: To an oven-dried tube was added ynamide **1ag** (102.1 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 0.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ag** (23.5 mg, 0.08 mmol) in 22% yield.

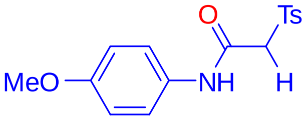
2ag: R_f = 0.37 [2:1 petroleum ether/EtOAc]; white solid; mp = 174–175 $^{\circ}$C; ^{1}H NMR (400 MHz, CDCl$_3$) *δ* 8.41 (s, 1H), 7.86 (d, 2H, *J* = 8.7 Hz), 7.58-7.45 (m, 4H), 7.36 (t, 2H, *J* = 7.9 Hz), 7.18 (t, 1H, *J* = 7.4 Hz), 4.17 (s, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) *δ* 158.3, 141.9, 136.9, 136.3, 130.2, 129.8, 129.4, 125.6, 120.3, 63.2; IR (neat) (cm^{-1}) 3372m, 2920w, 1663s, 1532s, 1393w, 1084s, 831m, 688s; HRMS (ESI): m/z calcd for $C_{14}H_{13}ClNO_3S$ $[M + H]^+$ 310.0299, found 310.0304.



2ah

Condition A: To a 10 mL Schlenk flask was added ynamide **1ah** (99.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 1.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 3 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ah** (81.3 mg, 0.27 mmol) in 77% yield.

2ah: R_f = 0.39 [2:1 petroleum ether/EtOAc]; white solid; mp = 145–146 ^{o}C; ^{1}H NMR (400 MHz, CDCl$_3$) *δ* 8.59 (s, 1H), 7.80 (d, 2H, *J* = 8.4 Hz), 7.35 (t, 4H, *J* = 8.9 Hz), 7.10 (d, 2H, *J* = 8.1 Hz), 4.18 (s, 2H), 2.42 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$) *δ* 158.8, 146.0, 135.1, 135.0, 134.6, 130.3, 129.7, 128.3, 120.4, 63.2, 21.9, 21.1; IR (neat) (cm^{-1}) 3338m, 2991m, 2922m, 1662s, 1508s, 1236w, 814s, 723s; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_3S$ [M + H]$^+$ 304.1002, found 304.1006.

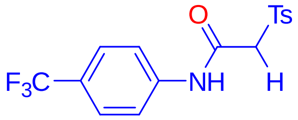


2ai

Condition A: To a 10 mL Schlenk flask was added ynamide **1ai** (105.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 3.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ai** (83.1 mg, 0.26 mmol) in 74% yield.

Condition B: To an oven-dried tube was added ynamide **1ai** (105.5 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 0.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ai** (13.6 mg, 0.04 mmol) in 12% yield.

2ai: R_f = 0.19 [2:1 petroleum ether/EtOAc]; white solid; mp = 144–145 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), 7.80 (d, 2H, J = 8.3 Hz), 7.37 (dd, 4H, J = 12.4, 8.5 Hz), 6.85 (d, 2H, J = 9.0 Hz), 4.15 (s, 2H), 3.79 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 157.1, 146.0, 135.1, 130.4, 130.2, 128.3, 122.1, 114.3, 63.0, 55.6, 21.9; IR (neat) (cm^{-1}) 3750m, 1658m, 1508s, 1412m, 1275w, 1144s, 823s, 721m; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_4S$ [M + H]$^+$ 320.0951, found 320.0954.



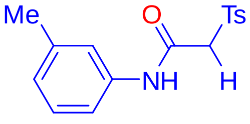
2aj

Condition A: To a 10 mL Schlenk flask was added ynamide **1aj** (118.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 12 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aj** (70.2 mg, 0.20 mmol) in 56% yield.

Condition B: To an oven-dried tube was added ynamide **1aj** (118.8 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 24 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aj** (10.2 mg, 0.03 mmol) in 8% yield.

2aj: R_f = 0.28 [2:1 petroleum ether/EtOAc]; white solid; mp = 182–183 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 7.79 (d, 2H, J = 8.4 Hz), 7.70-7.53 (m, 4H), 7.38 (d, 2H, J = 8.1 Hz), 4.18 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 146.4, 140.1, 134.8, 130.6, 128.2, 127.1 (q, $J_{C\text{-}F}$ = 33.1 Hz), 126.6 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.1 (q, $J_{C\text{-}F}$ = 270.3 Hz), 119.9, 63.1, 22.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.2; IR (neat) (cm^{-1}) 3375m, 2936w, 1683s, 1528s, 1182m, 1067s, 839m, 739m;

HRMS (ESI): m/z calcd for $C_{16}H_{15}F_3NO_3S$ $[M + H]^+$ 358.0719, found 358.0722.

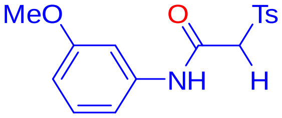


2ak

Condition A: To a 10 mL Schlenk flask was added ynamide **1ak** (99.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 3 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 2.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ak** (75.7 mg, 0.25 mmol) in 71% yield.

Condition B: To an oven-dried tube was added ynamide **1ak** (99.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 0.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ak** (9.8 mg, 0.03 mmol) in 9% yield.

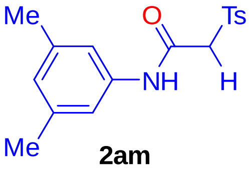
2ak: R_f = 0.30 [2:1 petroleum ether/EtOAc]; white solid; mp = 112–113 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (s, 1H), 7.80 (d, 2H, *J* = 8.3 Hz), 7.37-7.27 (m, 4H), 7.20 (t, 1H, *J* = 7.8 Hz), 6.97 (d, 1H, *J* = 7.5 Hz), 4.16 (s, 2H), 2.43 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.7, 146.1, 139.2, 137.1, 135.0, 130.4, 129.1, 128.3, 126.1, 120.9, 117.4, 63.2, 21.9, 21.6; IR (neat) (cm^{-1}) 3263m, 2962w, 1662s, 1528s, 1431w, 1288w, 1145s, 801m; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_3S$ $[M + H]^+$ 304.1002, found 304.1006.



2al

Condition A: To a 10 mL Schlenk flask was added ynamide **1al** (105.5 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 1 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2al** (94.6 mg, 0.30 mmol) in 85% yield.

2al: R_f = 0.22 [2:1 petroleum ether/EtOAc]; white solid; mp = 109–110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.66 (s, 1H), 7.80 (d, 2H, *J* = 8.3 Hz), 7.34 (d, 2H, *J* = 8.0 Hz), 7.23-7.16 (m, 2H), 7.01 (dd, 1H, *J* = 8.0, 2.0 Hz), 6.69 (d, 1H, *J* = 8.3 Hz), 4.19 (s, 2H), 3.75 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.2, 158.9, 146.0, 138.4, 135.0, 130.3, 129.9, 128.3, 112.4, 111.0, 105.9, 63.3, 55.4, 21.9; IR (neat) (cm^{-1}) 3282m, 2916w, 1666s, 1428m, 1217s, 1146s, 848s, 703m; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_4S$ $[M + H]^+$ 320.0951, found 320.0955.

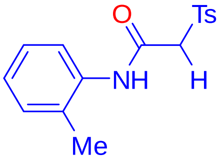


2am

Condition A: To a 10 mL Schlenk flask was added ynamide **1am** (104.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 2 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2am** (78.0 mg, 0.25 mmol) in 70% yield.

Condition B: To an oven-dried tube was added ynamide **1am** (104.8 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 °C with stirring for 1 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2am** (29.8 mg, 0.09 mmol) in 27% yield.

2am: R_f = 0.35 [2:1 petroleum ether/EtOAc]; white solid; mp = 139–140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.51 (s, 1H), 7.80 (d, 2H, *J* = 8.3 Hz), 7.35 (d, 2H, *J* = 8.0 Hz), 7.12 (s, 2H), 6.79 (s, 1H), 4.15 (s, 2H), 2.43 (s, 3H), 2.28 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 158.7, 146.0, 138.9, 137.0, 135.1, 130.3, 128.3, 127.0, 118.0, 63.2, 21.9, 21.5; IR (neat) (cm^{-1}) 3270m, 2919w, 1684s, 1534s, 1217m, 1147s, 804m, 705m; HRMS (ESI): m/z calcd for $C_{17}H_{20}NO_3S$ [M + H]$^+$ 318.1158 found 318.1164.

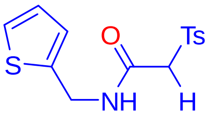


2an

Condition A: To a 10 mL Schlenk flask was added ynamide **1an** (99.9 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 4.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2an** (72.0 mg, 0.24 mmol) in 68% yield.

Condition B: To an oven-dried tube was added ynamide **1an** (99.9 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2an** (30.1 mg, 0.10 mmol) in 28% yield.

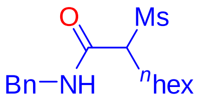
2an: R_f = 0.35 [2:1 petroleum ether/EtOAc]; white solid; mp = 117–118 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 7.81 (d, 2H, *J* = 8.3 Hz), 7.74 (d, 1H, *J* = 8.1 Hz), 7.36 (d, 2H, *J* = 8.1 Hz), 7.19 (dd, 2H, *J* = 11.6, 7.8 Hz), 7.10 (t, 1H, *J* = 7.5 Hz), 4.19 (s, 2H), 2.44 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 146.0, 135.2, 135.1, 130.8, 130.4, 129.5, 128.2, 126.9, 125.9, 122.8, 62.7, 21.9, 18.0; IR (neat) (cm^{-1}) 3334w, 2983w, 1654s, 1523s, 1412m, 1146m, 918m, 809m; HRMS (ESI): m/z calcd for $C_{16}H_{18}NO_3S$ [M + H]$^+$ 304.1002, found 304.1006.



2ao

Condition A: To a 10 mL Schlenk flask was added ynamide **1ao** (102.0 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ao** (74.8 mg, 0.24 mmol) in 69% yield.

2ao: R_f = 0.30 [2:1 petroleum ether/EtOAc]; yellow solid; mp = 129–130 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.68 (d, 2H, *J* = 8.4 Hz), 7.30 (d, 2H, *J* = 8.0 Hz), 7.26-7.23 (m, 1H), 7.20 (t, 1H, *J* = 5.8 Hz), 7.01-6.93 (m, 2H), 4.58 (d, 2H, *J* = 5.6 Hz), 4.00 (s, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.6, 145.7, 139.8, 135.1, 130.2, 128.3, 127.1, 126.8, 125.6, 62.0, 38.6, 21.9; IR (neat) (cm^{-1}) 3353m, 2919w, 2362m, 1664s, 1520s, 1316m, 1143s, 899m; HRMS (ESI): m/z calcd for $C_{14}H_{16}NO_3S_2$ $[M + H]^+$ 310.0566, found 310.0567.

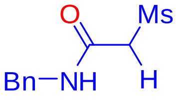


2ap

Condition A: To a 10 mL Schlenk flask was added ynamide **1ap** (102.7 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 7.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ap** (96.0 mg, 0.31 mmol) in 88% yield.

Condition B: To an oven-dried tube was added ynamide **1ap** (102.7 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 12 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2ap** (36.0 mg, 0.12 mmol) in 33% yield.

2ap: R_f = 0.27 [4:1 petroleum ether/EtOAc]; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.36-7.27 (m, 5H), 6.83 (t, 1H, *J* = 5.7 Hz), 4.48 (d, 2H, *J* = 5.8 Hz), 3.56 (dd, 1H, *J* = 9.7, 5.2 Hz), 2.90 (s, 3H), 2.08-1.99 (m, 2H), 1.39-1.24 (m, 8H), 0.88 (t, 3H, *J* = 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 165.2, 137.5, 128.9, 127.83, 127.80, 70.8, 44.2, 37.9, 31.5, 28.9, 27.13, 27.08, 22.6, 14.2. Spectral data are in agreement with literature values[18].

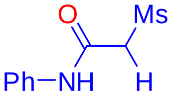


2aq

Condition A: To a 10 mL Schlenk flask was added ynamide **1aq** (73.2 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 2 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~1:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aq** (74.9 mg, 0.33 mmol) in 94% yield.

Condition B: To an oven-dried tube was added ynamide **1aq** (73.2 mg, 0.35 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.5 h. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 0.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~1:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2aq** (16.9 mg, 0.07 mmol) in 21% yield.

2aq: R_f = 0.25 [1:1 petroleum ether/EtOAc]; white solid; mp = 118–119 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.28 (m, 5H), 6.89 (t, 1H, J = 5.7 Hz), 4.47 (d, 2H, J = 5.8 Hz), 3.88 (s, 2H), 3.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.5, 137.2, 129.0, 128.0, 127.9, 60.9, 44.3, 41.4; IR (neat) (cm^{-1}) 3255m, 2953w, 1648s, 1550m, 1309s, 1128s, 900s, 759m; HRMS (ESI): m/z calcd for $C_{10}H_{14}NO_3S$ [M + H]$^+$ 228.0689, found 228.0693.



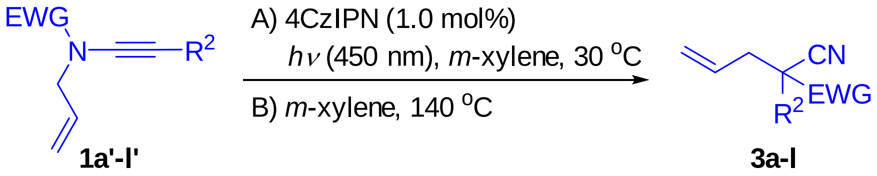
2ar

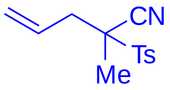
Condition A: To a 10 mL Schlenk flask was added ynamide **1ar** (68.3 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 4.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. Water (15.8 µL, 0.88 mmol) was then added, and the mixture was stirred for an additional 3 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford α-sulfonyl amide **2ar** (60.5 mg, 0.28 mmol) in 81% yield.

2ar: R_f = 0.17 [2:1 petroleum ether/EtOAc]; white solid; mp = 111–112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H), 7.52 (d, 2H, J = 7.5 Hz), 7.33 (t, 2H, J = 7.9 Hz), 7.16 (t, 1H, J = 7.5 Hz), 4.11 (s, 2H), 3.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 137.1, 129.3, 125.6, 120.4, 61.9, 41.5; IR (neat) (cm^{-1}) 3351m, 2964w, 1683s, 1538s, 1443m, 1294s, 1148m, 913m; HRMS (ESI): m/z calcd for $C_9H_{12}NO_3S$ $[M + H]^+$ 214.0532, found 214.0535.

1.4 Substrate scope of *N*-sulfonyl ynamides in *α*-sulfonyl nitriles synthesis

All α-sulfonyl nitriles are novel except for **3g** and **3h**, which are known compounds.





3a

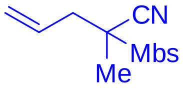
Condition A: To a 10 mL Schlenk flask was added ynamide **1a′** (99.7 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3a** (78.8 mg, 0.32 mmol) in 79% yield.

Condition B: To an oven-dried tube was added ynamide **1a′** (99.7 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3a** (77.8 mg, 0.31 mmol) in 78% yield.

Condition A (1.0 mmol synthetic method): To a 10 mL Schlenk flask was added ynamide **1a′** (249.3 mg, 1.0 mmol), 4CzIPN (7.9 mg, 0.01 mmol), and *m*-xylene (5.7 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3a** (187.0 mg, 0.75 mmol) in 75% yield.

Condition B (1.0 mmol synthetic method): To an oven-dried tube was added ynamide **1a′** (249.3 mg, 1.0 mmol), and *m*-xylene (5.7 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3a** (192.1mg, 0.77 mmol) in 77% yield.

3a: R_f = 0.38 [10:1 petroleum ether/EtOAc]; white solid; mp = 57–58 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.89 (d, 2H, *J* = 8.4 Hz), 7.44 (d, 2H, *J* = 8.1 Hz), 5.89-5.66 (m, 1H), 5.36-5.26 (m, 2H), 2.83 (dd, 1H, *J* = 13.7, 6.3 Hz), 2.58 (dd, 1H, *J* = 13.7, 8.4 Hz), 2.49 (s, 3H), 1.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 146.8, 130.9, 130.6, 130.2, 129.2, 122.6, 117.3, 61.6, 37.3, 21.9, 18.4; IR (neat) (cm^{-1}) 1639w, 1593m, 1405w, 1321s, 1177w, 1076s, 813s, 732w; HRMS (ESI): m/z calcd for $C_{13}H_{16}NO_2S$ [M + H]$^+$ 250.0896, found 250.0898.

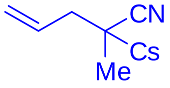


3b

Condition A: To a 10 mL Schlenk flask was added ynamide **1b′** (106.1 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3b** (96.6 mg, 0.36 mmol) in 91% yield.

Condition B: To an oven-dried tube was added ynamide **1b′** (106.1 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3b** (95.2 mg, 0.36 mmol) in 90% yield.

3b: R_f = 0.15 [10:1 petroleum ether/EtOAc]; white solid; mp = 88–89 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.93 (d, 2H, *J* = 9.0 Hz), 7.09 (d, 2H, *J* = 9.0 Hz), 5.87-5.72 (m, 1H), 5.39-5.25 (m, 2H), 3.91 (s, 3H), 2.83 (dd, 1H, *J* = 13.7, 6.2 Hz), 2.58 (dd, 1H, *J* = 13.6, 6.2 Hz), 1.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 165.1, 133.1, 129.3, 124.8, 122.4, 117.4, 114.7, 61.7, 55.9, 37.3, 18.4; IR (neat) (cm^{-1}) 2933w, 2242w, 1591s, 1493s, 1415m, 1305s, 1149s, 933s; HRMS (ESI): m/z calcd for $C_{13}H_{16}NO_3S$ [M + H]$^+$ 266.0845, found 266.0848.



3c

Condition A: To a 10 mL Schlenk flask was added ynamide **1c′** (107.9 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3c** (84.3 mg, 0.29 mmol) in 73% yield.

Condition B: To an oven-dried tube was added ynamide **1c′** (107.9 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3c** (97.2 mg, 0.36 mmol) in 90% yield.

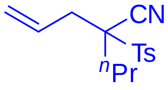
3c: R_f = 0.50 [10:1 petroleum ether/EtOAc]; white solid; mp = 77–78 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.97 (d, 2H, *J* = 8.6 Hz), 7.63 (d, 2H, *J* = 8.6 Hz), 5.85-5.72 (m, 1H), 5.38-5.29 (m, 2H), 2.84 (dd, 1H, *J* = 13.7, 6.3 Hz), 2.60 (dd, 1H, *J* = 13.6, 8.4 Hz), 1.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 142.6, 132.4, 132.2, 130.0, 128.9, 123.0, 117.1, 61.8, 37.3, 18.4; IR (neat) (cm^{-1}) 2919m, 2242w, 1639m, 1323s, 1280s, 1153s, 996m, 891w; HRMS (ESI): m/z calcd for $C_{12}H_{13}ClNO_2S$ [M + H]$^+$ 270.0350, found 270.0349.

3d

Condition A: To a 10 mL Schlenk flask was added ynamide **1d′** (112.1 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 18 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3d** (70.6 mg, 0.25 mmol) in 63% yield.

Condition B: To an oven-dried tube was added ynamide **1d′** (112.1 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3d** (104.9 mg, 0.37 mmol) in 94% yield.

3d: R_f = 0.23 [10:1 petroleum ether/EtOAc]; white solid; mp = 117–118 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.49 (d, 2H, *J* = 8.9 Hz), 8.25 (d, 2H, *J* = 8.9 Hz), 5.87-5.70 (m, 1H), 5.44-5.26 (m, 2H), 2.85 (dd, 1H, *J* = 13.6, 6.3 Hz), 2.65 (dd, 1H, *J* = 13.6, 8.3 Hz), 1.66 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 151.8, 139.4, 132.6, 128.4, 124.6, 123.4, 116.7, 62.1, 37.1, 18.3; IR (neat) (cm^{-1}) 2912w, 2242w, 1606m, 1531s, 1355s, 1153s, 970m, 855s; HRMS (ESI): m/z calcd for $C_{12}H_{12}N_2NaO_4S$ [M + Na]$^+$ 303.0410, found 303.0413.



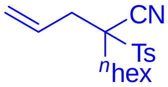
3e

Condition A: To a 10 mL Schlenk flask was added ynamide **1e′** (111.0 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3e** (80.3 mg, 0.29 mmol) in 72% yield.

Condition B: To an oven-dried tube was added ynamide **1e′** (111.0 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3e** (98.7 mg, 0.36 mmol) in 89% yield.

3e: R_f = 0.34 [10:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.89 (d, 2H, *J* = 8.4 Hz), 7.43 (d, 2H, *J* = 8.1 Hz), 5.90-5.75 (m, 1H), 5.31-5.20 (m, 2H), 2.71 (d, 2H, *J* = 6.8 Hz), 2.49 (s, 3H), 2.05-1.86 (m, 2H), 1.68-1.50 (m, 2H), 0.94 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 146.6, 131.5, 130.8, 130.1, 129.7, 121.6, 116.8, 65.8, 36.8, 33.4, 21.9, 18.5, 14.1; IR (neat) (cm^{-1}) 2967m, 2238w, 1595s, 1442m, 1326s, 1149s, 929s, 814s; HRMS (ESI): m/z calcd for

$C_{15}H_{20}NO_2S$ [M + H]$^+$ 278.1209, found 278.1212.

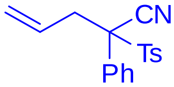


3f

Condition A: To a 10 mL Schlenk flask was added ynamide **1f′** (127.8 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3f** (103.4 mg, 0.32 mmol) in 81% yield.

Condition B: To an oven-dried tube was added ynamide **1f′** (127.8 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3f** (112.6 mg, 0.35 mmol) in 88% yield.

3f: R_f = 0.50 [10:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, 2H, *J* = 8.4 Hz), 7.34 (d, 2H, *J* = 8.1 Hz), 5.82-5.67 (m, 1H), 5.23-5.12 (m, 2H), 2.63 (d, 2H, *J* = 7.2 Hz), 2.41 (s, 3H), 1.98-1.80 (m, 2H), 1.54-1.37 (m, 2H), 1.26-1.14 (m, 6H), 0.79 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 131.5, 130.8, 130.1, 129.7, 121.6, 116.8, 65.8, 36.8, 31.41, 31.37, 29.2, 24.8, 22.5, 21.9, 14.1; IR (neat) (cm^{-1}) 2927m, 2237w, 1595m, 1328s, 1149s, 1084s, 928m, 814s; HRMS (ESI): m/z calcd for $C_{18}H_{26}NO_2S$ [M + H]$^+$ 320.1679, found 320.1682.



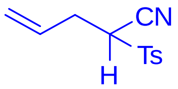
3g

Condition A: To a 10 mL Schlenk flask was added ynamide **1g′** (124.6 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 30 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3g** (63.0 mg, 0.20 mmol) in 51% yield.

Condition B: To an oven-dried tube was added ynamide **1g′** (124.6 mg, 0.4 mmol), and *m*-xylene

(2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 18 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3g** (76.8 mg, 0.25 mmol) in 62% yield.

3g: R_f = 0.29 [10:1 petroleum ether/EtOAc]; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.36 (m, 5H), 7.35-7.28 (m, 2H), 7.19 (d, 2H, J = 8.1 Hz), 5.61-5.48 (m, 1H), 5.30 (dd, 1H, J = 16.9, 1.4 Hz), 5.18 (d, 1H, J = 10.0 Hz), 3.46 (dd, 1H, J = 14.1, 6.7 Hz), 3.32 (dd, 1H, J = 14.1, 7.4 Hz), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 130.8, 130.7, 130.2, 129.6, 129.1, 128.88, 128.86, 128.4, 122.2, 116.3, 72.0, 35.9, 21.9. Spectral data are in agreement with literature values[19].

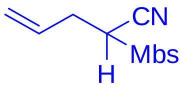


3h

Condition A: To a 10 mL Schlenk flask was added ynamide **1h′** (94.1 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 32 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3h** (63.4 mg, 0.27 mmol) in 67% yield.

Condition B: To an oven-dried tube was added ynamide **1h′** (94.1 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3h** (10.2 mg, 0.04 mmol) in 11% yield.

3h: R_f = 0.25 [10:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, 2H, J = 8.4 Hz), 7.45 (d, 2H, J = 8.0 Hz), 5.86-5.72 (m, 1H), 5.36-5.24 (m, 2H), 3.93 (dd, 1H, J = 10.9, 4.4 Hz), 3.00-2.91 (m, 1H), 2.67-2.56 (m, 1H), 2.50 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 132.6, 130.5, 130.0, 129.9, 121.3, 113.9, 57.4, 31.2, 22.0. Spectral data are in agreement with literature values[19].



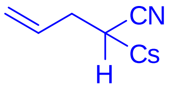
3i

Condition A: To a 10 mL Schlenk flask was added ynamide **1i′** (100.5 mg, 0.4 mmol), 4CzIPN

(3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 4 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~3:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3i** (78.1 mg, 0.31 mmol) in 78% yield.

Condition B: To an oven-dried tube was added ynamide **1i′** (100.5 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~3:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3i** (8.3 mg, 0.03 mmol) in 8% yield.

3i: R_f = 0.42 [3:1 petroleum ether/EtOAc]; white solid; mp = 62–63 ^{o}C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.93 (d, 2H, J = 9.0 Hz), 7.09 (d, 2H, J = 9.0 Hz), 5.86-5.71 (m, 1H), 5.35-5.23 (m, 2H), 3.97 (dd, 1H, J = 10.8, 4.4 Hz), 3.92 (s, 3H), 2.99-2.88 (m, 1H), 2.65-2.55 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 165.2, 132.1, 130.1, 126.7, 121.1, 115.0, 114.0, 57.5, 56.0, 31.3; IR (neat) (cm^{-1}) 2948m, 2241w, 1642w, 1496s, 1332s, 1142s, 941s, 814m; HRMS (ESI): m/z calcd for $C_{12}H_{14}NO_3S$ [M + H]$^+$ 252.0689, found 252.0691.



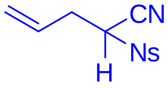
3j

Condition A: To a 10 mL Schlenk flask was added ynamide **1j′** (102.3 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 29 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3j** (59.3 mg, 0.23 mmol) in 58% yield.

Condition B: To an oven-dried tube was added ynamide **1j′** (102.3 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3j** (7.5 mg, 0.03 mmol) in 7% yield.

3j: R_f = 0.30 [10:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.96 (d, 2H, J

= 8.7 Hz), 7.64 (d, 2H, *J* = 8.6 Hz), 5.85-5.72 (m, 1H), 5.36-5.26 (m, 2H), 3.98 (dd, 1H, *J* = 10.8, 4.4 Hz), 3.00-2.91 (m, 1H), 2.70-2.56 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 142.7, 133.9, 131.3, 130.3, 129.7, 121.5, 113.6, 57.4, 31.1; IR (neat) (cm^{-1}) 2926w, 2246w, 1579s, 1475s, 1335s, 1218w, 991m, 830s; HRMS (ESI): m/z calcd for $C_{11}H_{10}ClNNaO_2S$ $[M + Na]^+$ 278.0013, found 278.0015.

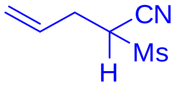


3k

Condition A: To a 10 mL Schlenk flask was added ynamide **1k′** (106.5 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 32 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3k** (43.7 mg, 0.16 mmol) in 41% yield.

Condition B: To an oven-dried tube was added ynamide **1k′** (106.5 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~4:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3k** (29.7 mg, 0.11 mmol) in 28% yield.

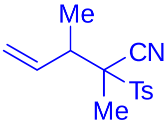
3k: R_f = 0.34 [4:1 petroleum ether/EtOAc]; colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.50 (d, 2H, *J* = 8.8 Hz), 8.25 (d, 2H, *J* = 8.8 Hz), 5.86-5.72 (m, 1H), 5.38-5.31 (m, 2H), 4.02 (dd, 1H, *J* = 10.6, 4.5 Hz), 3.06-2.96 (m, 1H), 2.74-2.63 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 152.0, 141.1, 131.5, 129.3, 125.0, 122.0, 113.2, 57.4, 30.9; IR (neat) (cm^{-1}) 2912w, 2246w, 1591s, 1531s, 1440m, 1147s, 939s, 855m; HRMS (ESI): m/z calcd for $C_{11}H_9N_2O_4S$ $[M - H]^-$ 265.0288, found 265.0290.



3l

Condition A: To a 10 mL Schlenk flask was added ynamide **1l′** (63.7 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 37 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3l** (36.7 mg, 0.23 mmol) in 58% yield.

3l: R_f = 0.50 [2:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 5.93-5.79 (m, 1H), 5.44-5.32 (m, 2H), 4.04-3.94 (m, 1H), 3.18 (s, 3H), 3.00-2.88 (m, 1H), 2.84-2.71 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 129.7, 121.7, 113.8, 55.4, 39.7, 30.2; IR (neat) (cm^{-1}) 2930m, 2362w, 1642w, 1412w, 1318s, 1146s, 985m, 836s; HRMS (ESI): m/z calcd for $C_6H_9NNaO_2S$ [M + Na]$^+$ 182.0246, found 182.0246.

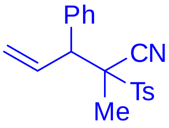


3m

Condition A: To a 10 mL Schlenk flask was added ynamide **1m′** (105.3 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 °C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 15:1~5:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3m** (64.7 mg, 0.25 mmol) in 61% yield.

Condition B: To an oven-dried tube was added ynamide **1m′** (105.3 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 3.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 15:1~5:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3m** (55.0 mg, 0.21 mmol) in 52% yield.

3m: R_f = 0.24 [10:1 petroleum ether/EtOAc]; yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.89 (d, 2H, *J* = 8.3 Hz), 7.42-7.40 (m, 2H), 5.92 (ddd, 1H, *J* = 17.6, 10.2, 7.9 Hz), 5.27-5.23 (m, 2H), 3.08 (p, 1H, *J* = 6.9 Hz), 2.49 (s, 3H), 1.58 (s, 3H), 1.34 (d, 3H, *J* = 6.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 146.6, 136.7, 132.0, 130.9, 130.0, 118.8, 117.8, 65.5, 41.0, 22.0, 16.5, 15.2; IR (neat) (cm^{-1}) 2918w, 1637m, 1329m, 1151s, 1075w, 816w, 785w; HRMS (ESI): m/z calcd for $C_{14}H_{18}NO_2S$ [M + H]$^+$ 264.1053, found 264.1051.



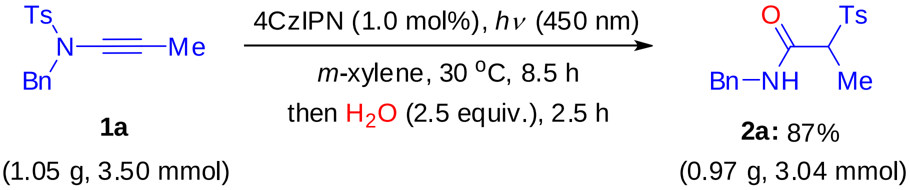
3n

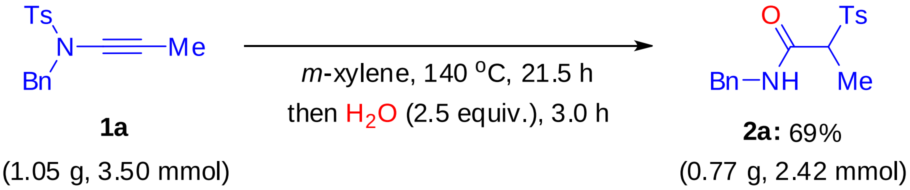
Condition A: To a 10 mL Schlenk flask was added ynamide **1n′** (130.2 mg, 0.4 mmol), 4CzIPN (3.2 mg, 0.004 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 15:1~5:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3n** (34.9 mg, 0.107 mmol) in 27% yield.

Condition B: To an oven-dried tube was added ynamide **1n′** (130.2 mg, 0.4 mmol), and *m*-xylene (2.3 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 1.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~5:1 petroleum ether /EtOAc] to afford α-sulfonyl nitrile **3n** (58.8 mg, 0.18 mmol) in 45% yield.

3n: R_f = 0.23 [10:1 petroleum ether/EtOAc]; white solid; mp = 86–87 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, 2H, J = 8.3 Hz), 7.40 (d, 2H, J = 8.1 Hz), 7.35-7.26 (m, 5H), 6.44 (ddd, 1H, J = 16.8, 10.3, 8.5 Hz), 5.24-5.18 (m, 2H), 4.11 (d, 1H, J = 8.6 Hz), 2.48 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 138.4, 135.5, 132.5, 131.4, 130.1, 129.3, 128.9, 128.3, 119.6, 117.4, 65.5, 52.2, 22.0, 21.1; IR (neat) (cm^{-1}) 2923m, 2852w, 2070w, 1452m, 1326m, 1148s, 926m; HRMS (ESI): m/z calcd for $C_{19}H_{20}NO_2S$ [M + H]$^+$ 326.1209, found 326.1207.

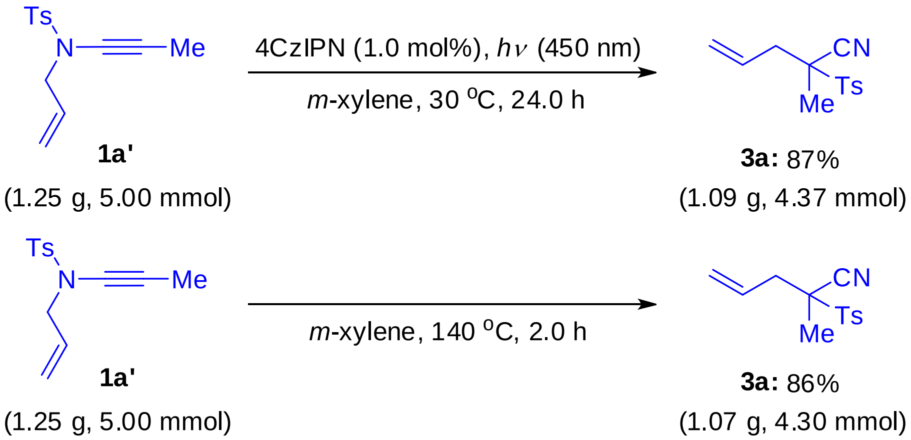
1.5 Gram-scale experiments and a synthetic application





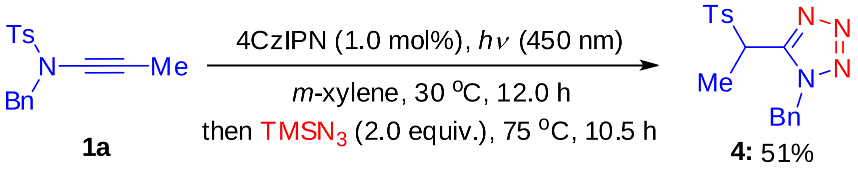
Condition A (gram-scale procedure): To a Schlenk flask was added ynamide **1a** (1.05 g, 3.50 mmol), 4CzIPN (27.6 mg, 0.035 mmol), and *m*-xylene (20.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. After 8.5 h of irradiation at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C, water (157.5 μL, 8.75 mmol) was then added, and the mixture was stirred for an additional 2.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (0.97 g, 3.04 mmol) in 87% yield.

Condition B (gram-scale procedure): To an oven-dried tube was added ynamide **1a** (1.05 g, 3.50 mmol), and *m*-xylene (20.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 21.5 h. Water (157.5 μL, 8.75 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 3.0 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **2a** (0.77 g, 2.42 mmol) in 69% yield.



Condition A (gram-scale procedure): To a Schlenk flask was added ynamide **1a′** (1.25 g, 5.00 mmol), 4CzIPN (39.4 mg, 0.05 mmol), and *m*-xylene (28.6 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 24.0 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3a** (1.09 g, 4.37 mmol) in 87% yield.

Condition B (gram-scale procedure): To an oven-dried tube was added ynamide **1a′** (1.25 g, 5.00 mmol), and *m*-xylene (28.6 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 2.0 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to afford *α*-sulfonyl nitrile **3a** (1.07 g, 4.30 mmol) in 86% yield.



1a

4: 51%

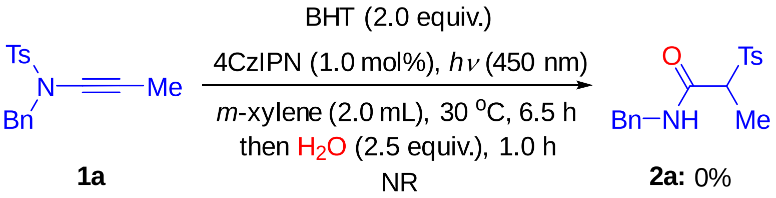
To a 10 mL Schlenk flask was added ynamide **1a** (104.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 12 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. $TMSN_3$ (80.6 mg, 0.7mmol) was then added, and the mixture was stirred in an oil bath and heated at 75 ^{o}C for 10.5 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [gradient eluent: 10:1~2:1 petroleum ether /EtOAc] to afford *α*-sulfonyl amide **4** (60.8 mg, 0.18 mmol) in 51% yield.

4: R_f = 0.35 [2:1 petroleum ether/EtOAc]; white solid; mp = 162–163 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.38 (m, 5H), 7.31 (d, 2H, *J* = 8.1 Hz), 7.24 (dd, 2H, *J* = 7.2, 2.3 Hz), 5.98 (d, 1H, *J* = 15.5 Hz), 5.80 (d, 1H, *J* = 15.6 Hz), 4.37 (q, 1H, *J* = 7.1 Hz), 2.44 (s, 3H), 1.62 (d, 3H, *J* = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.1, 146.5, 133.0, 130.7, 130.0, 129.8, 129.6, 129.3, 127.8, 56.3, 51.9, 21.9, 14.1; IR (neat) (cm^{-1}) 2922w, 1499m, 1312m, 1149s, 1085w, 820m, 751m; HRMS (ESI): m/z calcd for $C_{17}H_{19}N_4O_2S$ $[M + H]^+$ 343.1223, found 343.1222.

1.6 Control experiments

Condition A: To a 10 mL Schlenk flask was added ynamide **1a** (104.8 mg, 0.35 mmol), 4CzIPN (2.8 mg, 0.0035 mmol), BHT (154.2mg, 0.70 mmol) or TEMPO (109.4 mg, 0.70 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The reaction mixture was stirred and irradiated for 6.5 h at a distance of ~3.5 cm (light intensity: 20 mW/cm^2 at 3.5 cm distance) with 30 W blue LEDs (450 nm) at 30 ^{o}C. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to recover ynamide **1a** (100.2 mg, 0.33 mmol) with a recovery rate of 96% or recover ynamide **1a** (99.1 mg, 0.33 mmol) with a recovery rate of 95%.

Condition B: To an oven-dried tube was added ynamide **1a** (104.8 mg, 0.35 mmol), BHT (154.2mg, 0.70 mmol) or TEMPO (109.4 mg, 0.70 mmol), and *m*-xylene (2.0 mL, ynamide *concn* = 0.175 *M*) under a nitrogen atmosphere. The vessel was sealed and heated to 140 ℃ with stirring for 8.5 h. Water (15.8 μL, 0.88 mmol) was then added, and the mixture was stirred at 140 ℃ for an additional 1 h. After the reaction was determined to be complete by TLC analysis, the reaction mixture was purified by flash silica gel column chromatography [isocratic eluent: 10:1 petroleum ether /EtOAc] to recover ynamide **1a** (98.7 mg, 0.33 mmol) with a recovery rate of 94% or recover ynamide **1a** (98.2 mg, 0.33 mmol) with a recovery rate of 94%.



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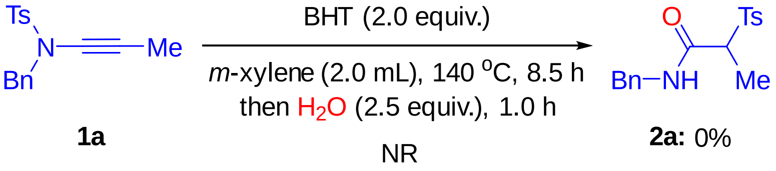

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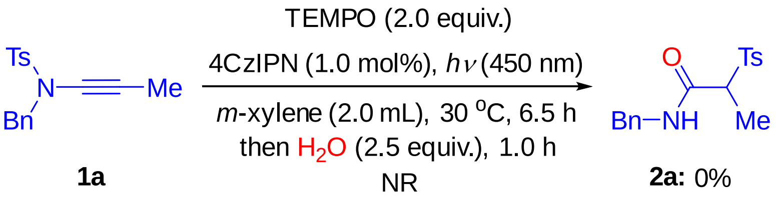

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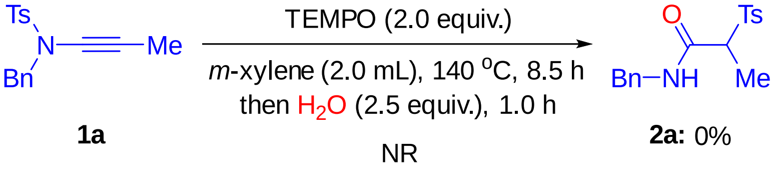

1a **2a:** 0%

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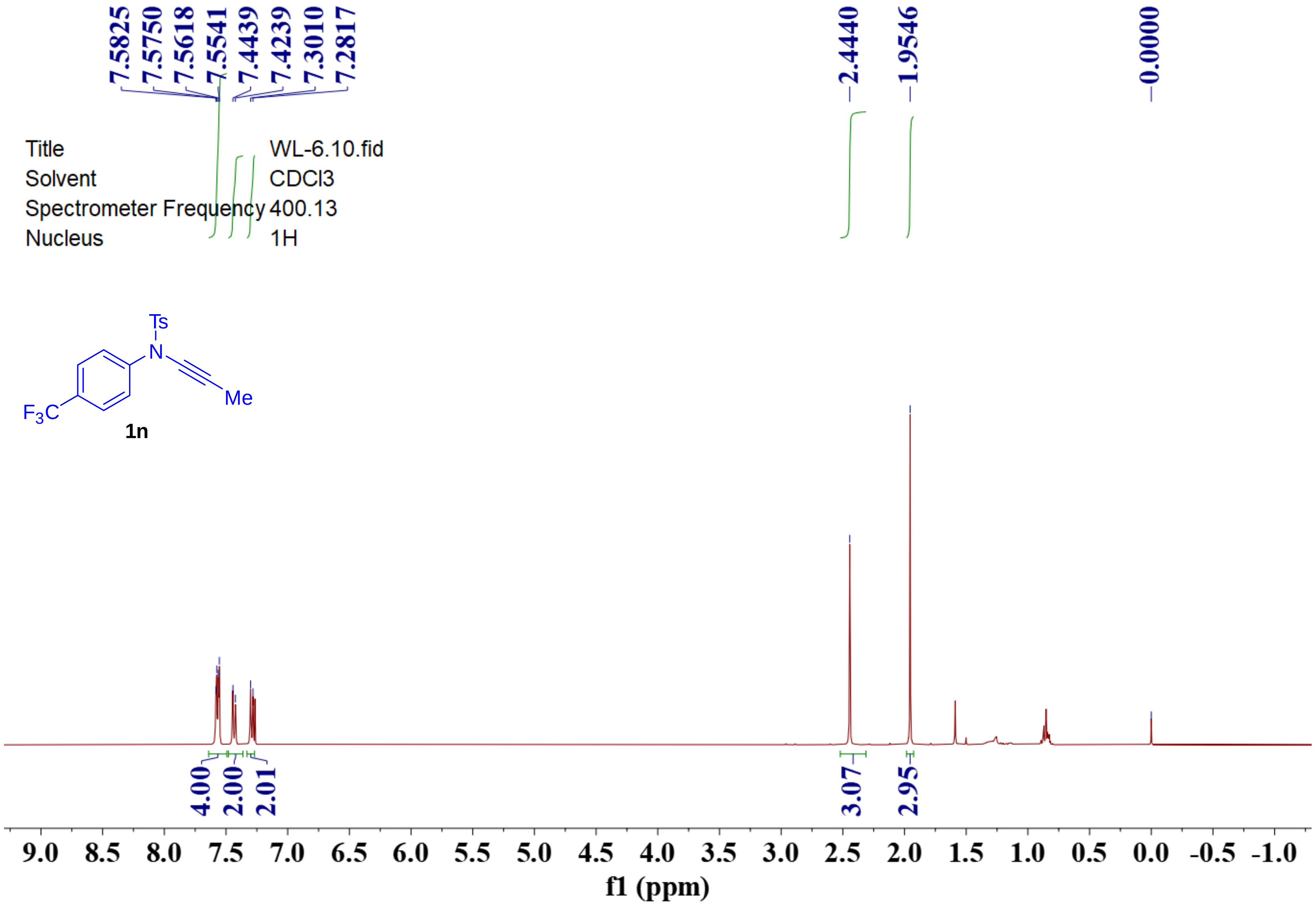
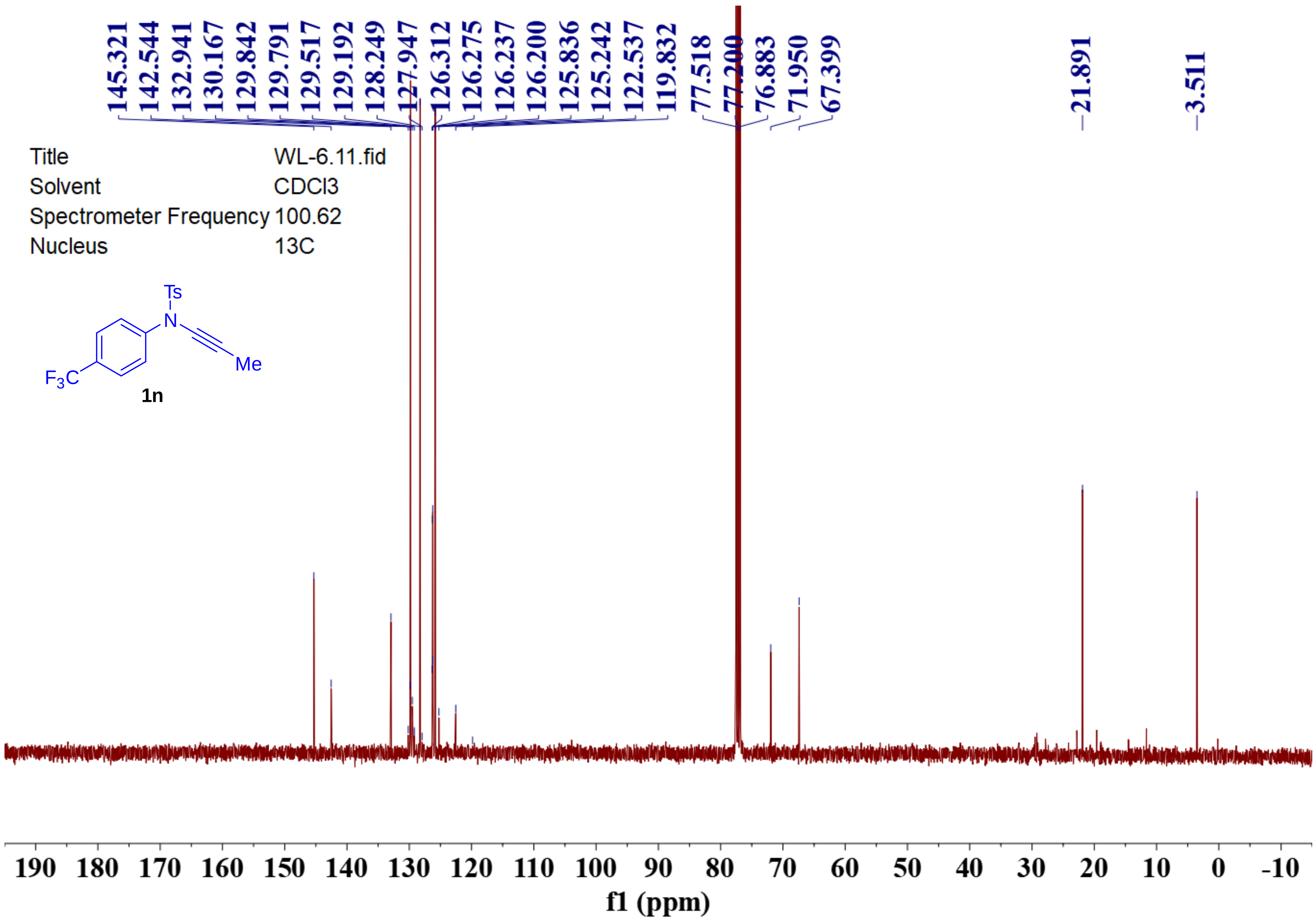
Part II Copies of ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR Spectra

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Title WL-6.11.fid
Solvent CDCl3
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Nucleus 13C
Ts
N
Me
F3C
1n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
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f1 (ppm)

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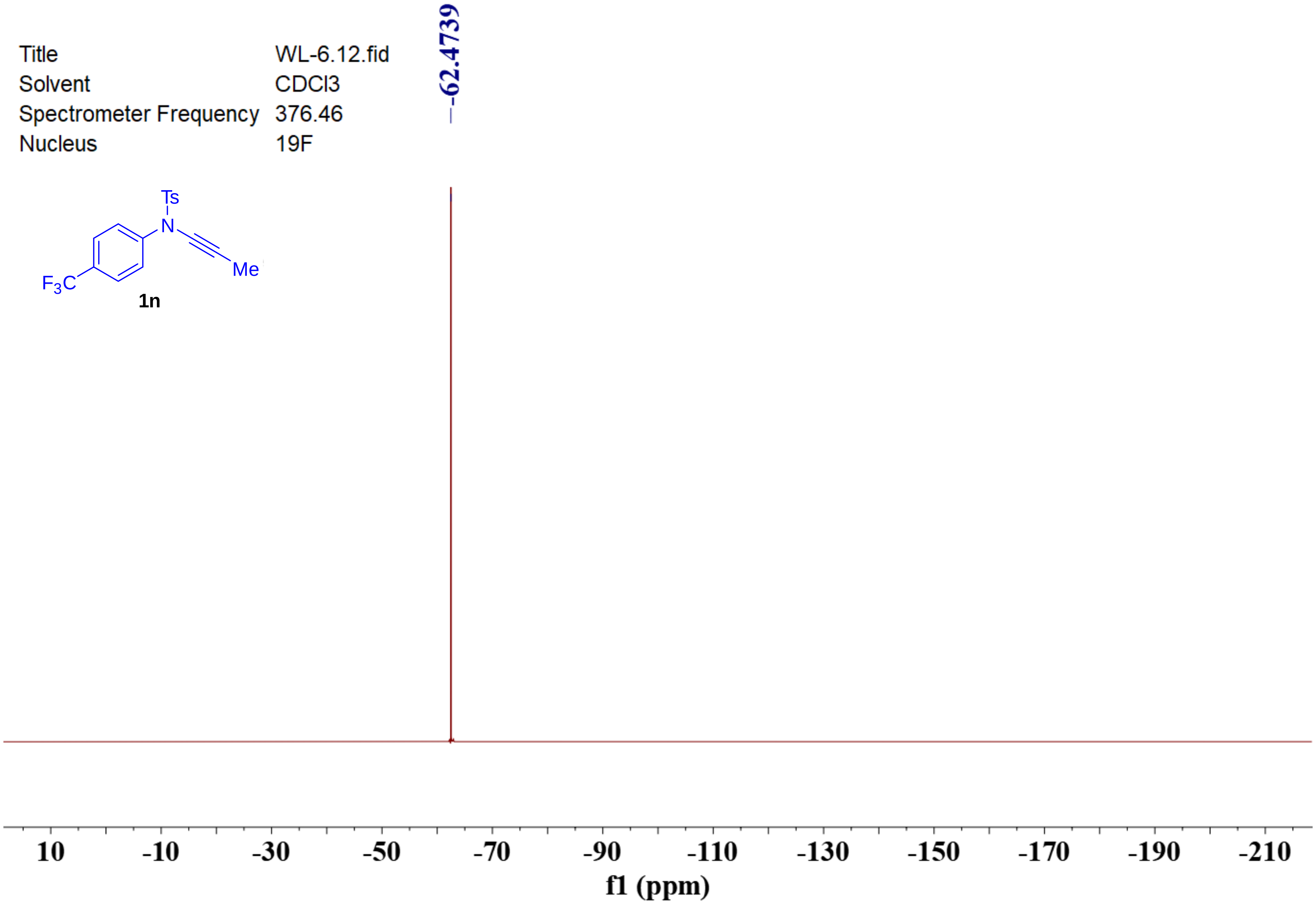
Solvent	CDCl3
Spectrometer Frequency	376.46
Nucleus	19F



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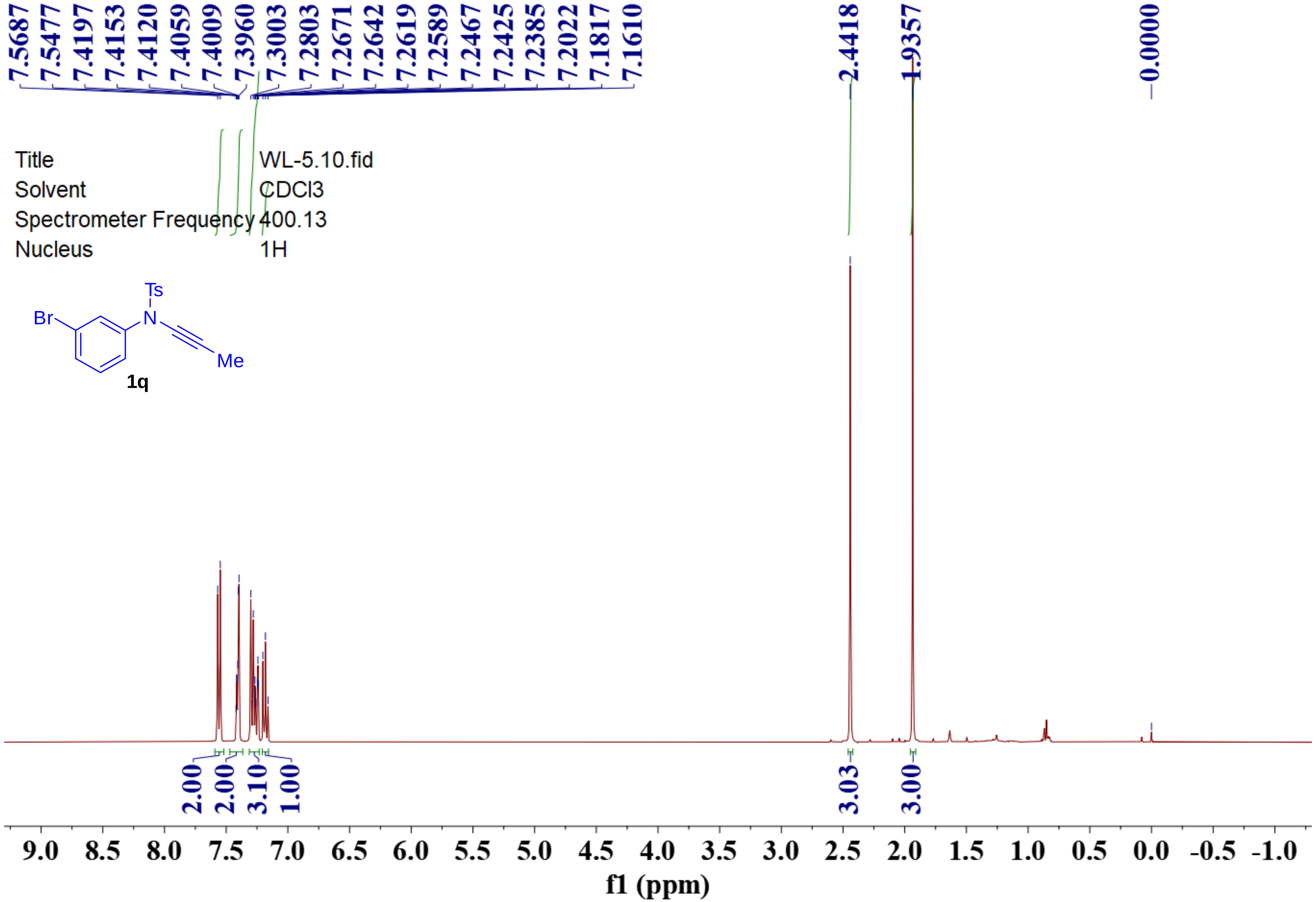
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Br
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Me
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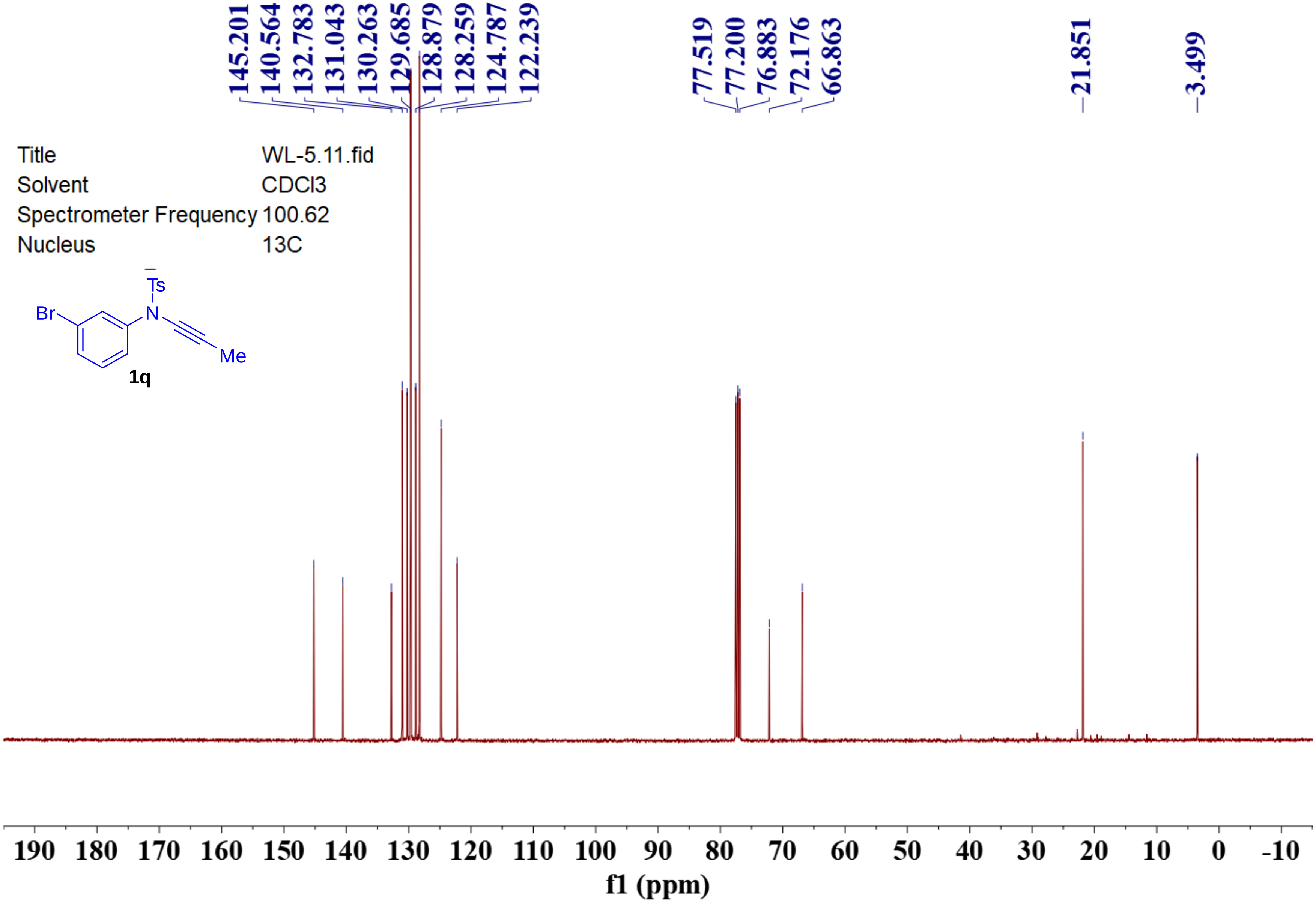
f1 (ppm)



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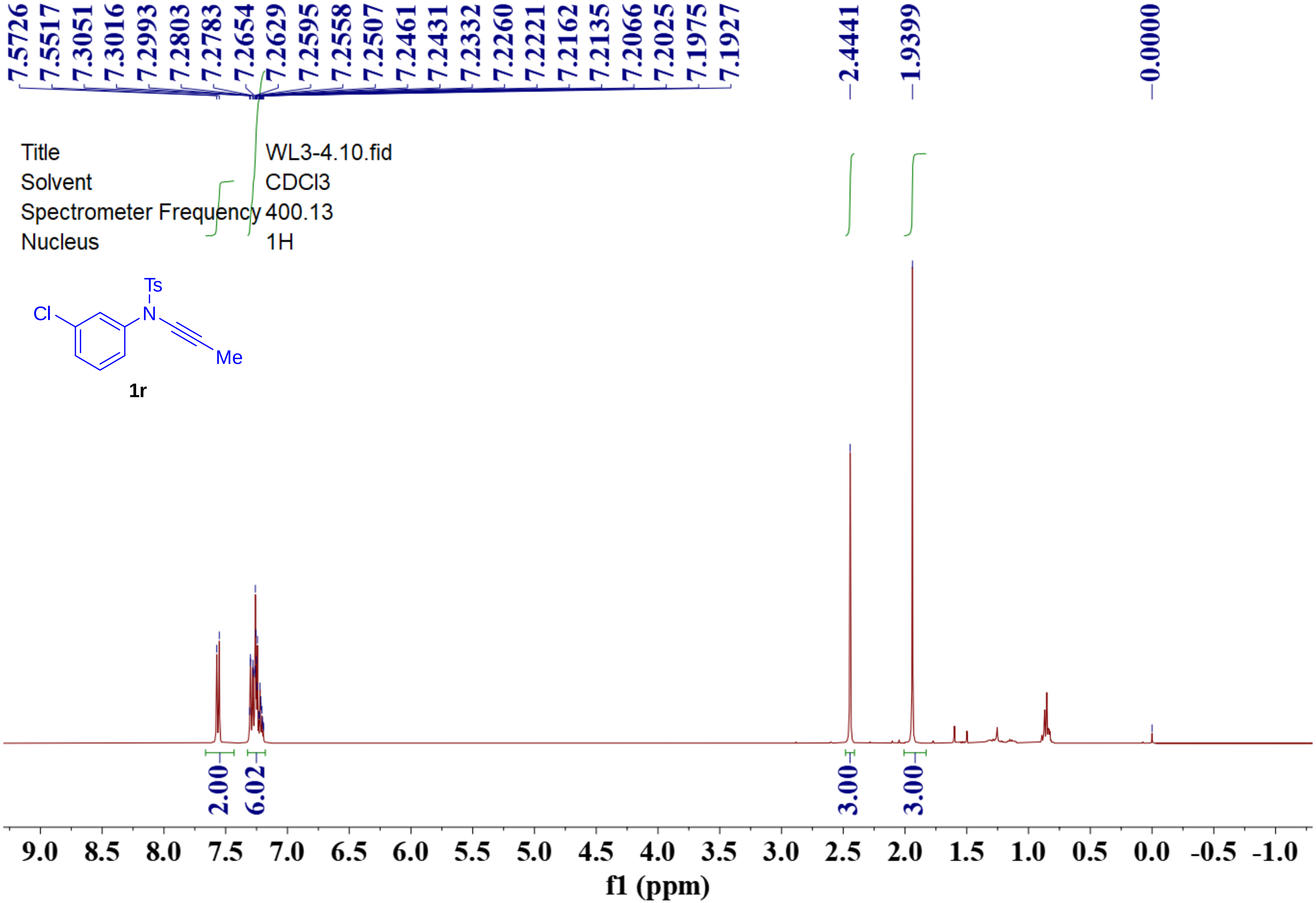
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C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Cl
Me
1r
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4.0
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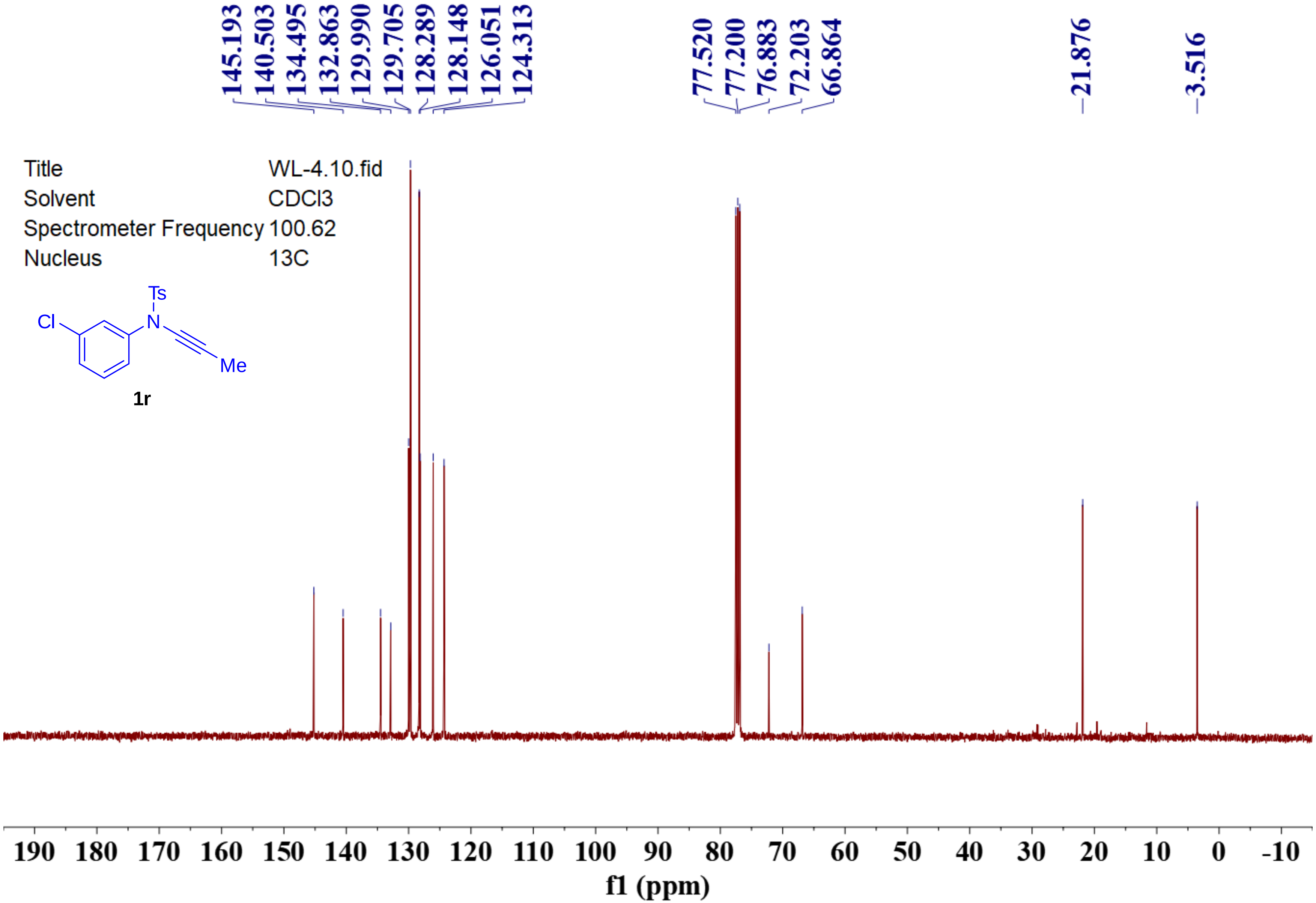
f1 (ppm)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Title WL-4.10.fid
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Ts
Cl
N
Me
1r
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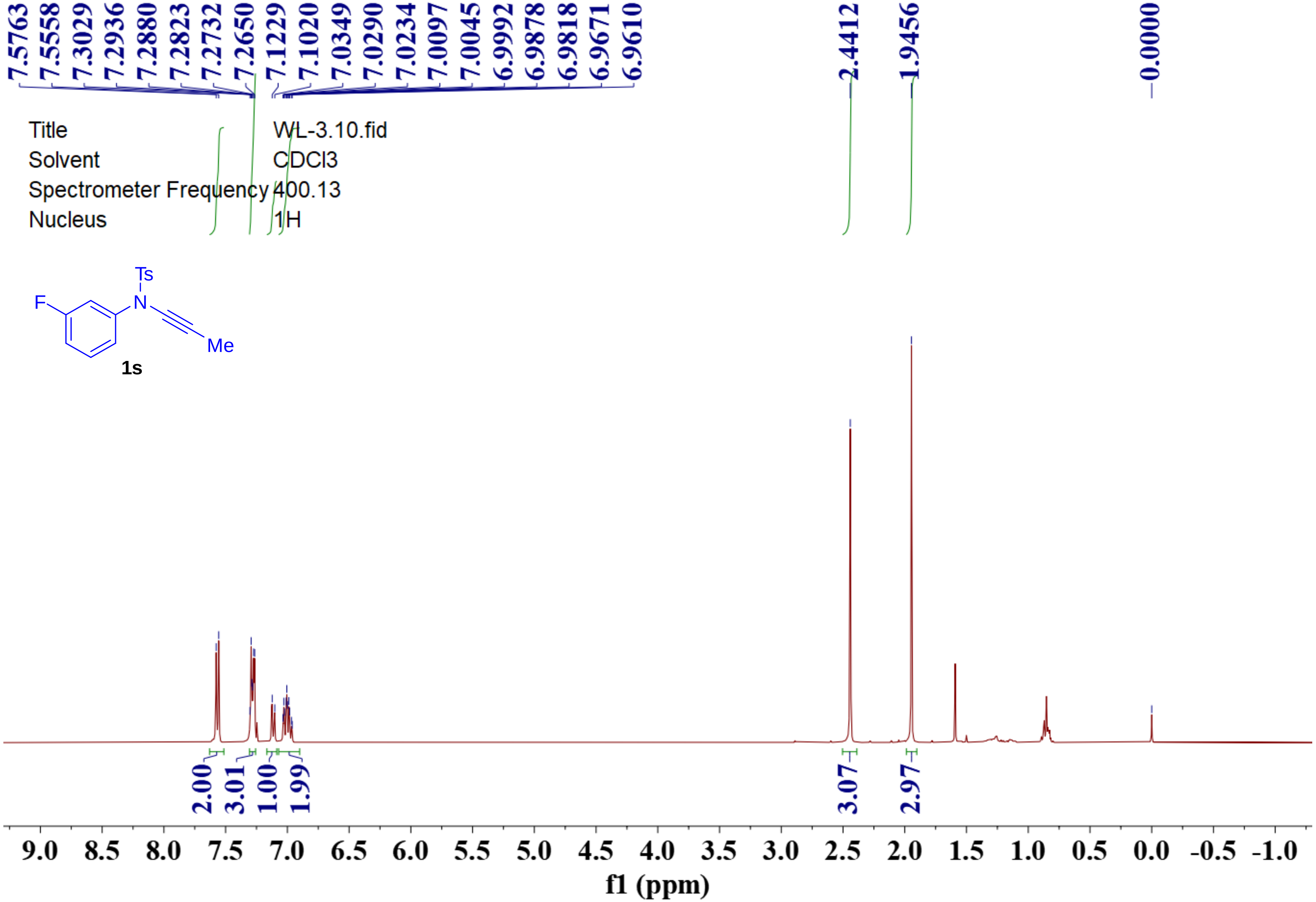
f1 (ppm)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Nucleus 1H
F
N
Ts
Me
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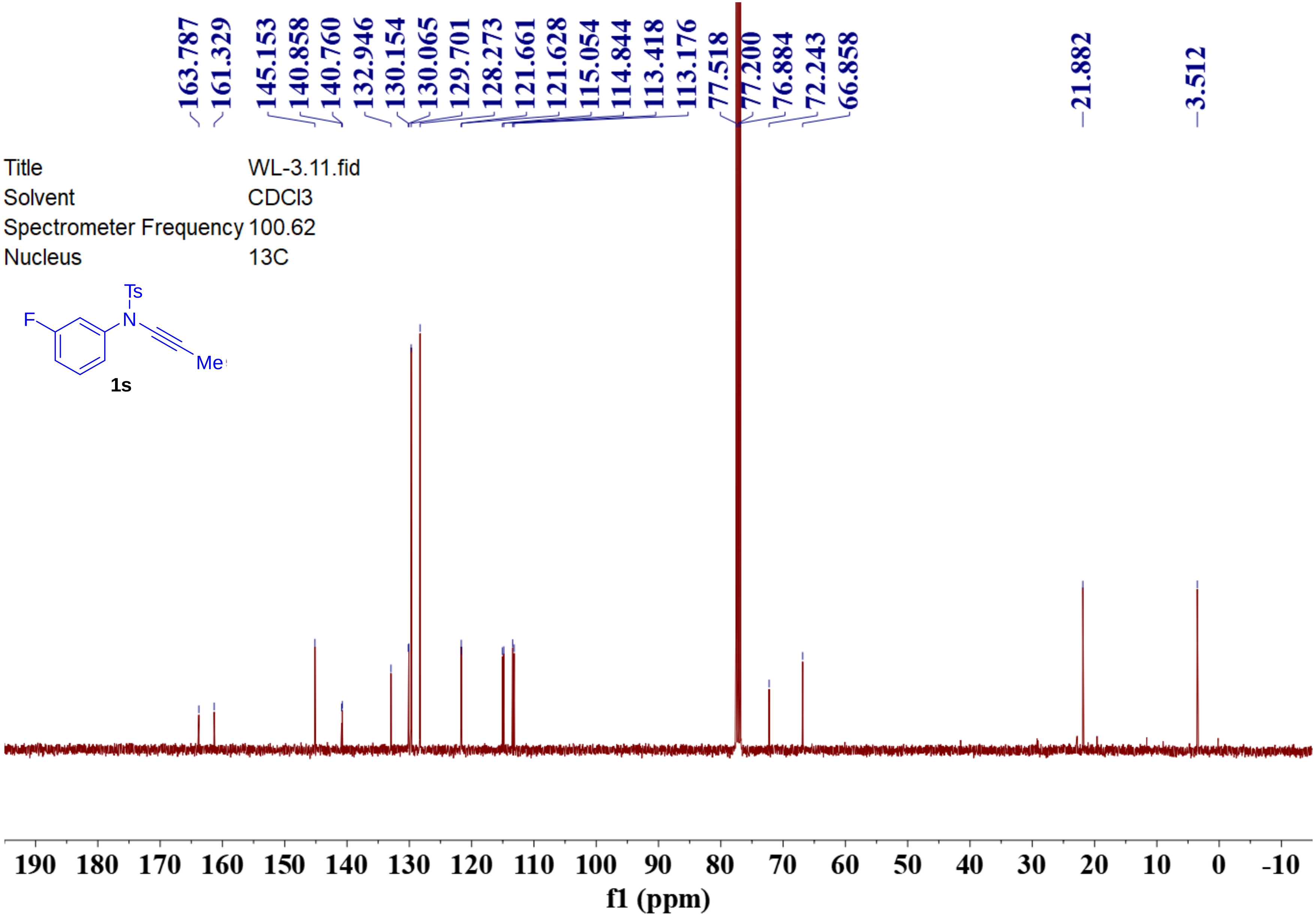
f1 (ppm)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Title WL-3.11.fid
Solvent CDCl3
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Nucleus 13C
Ts
F
N
Me
1s
190
180
170
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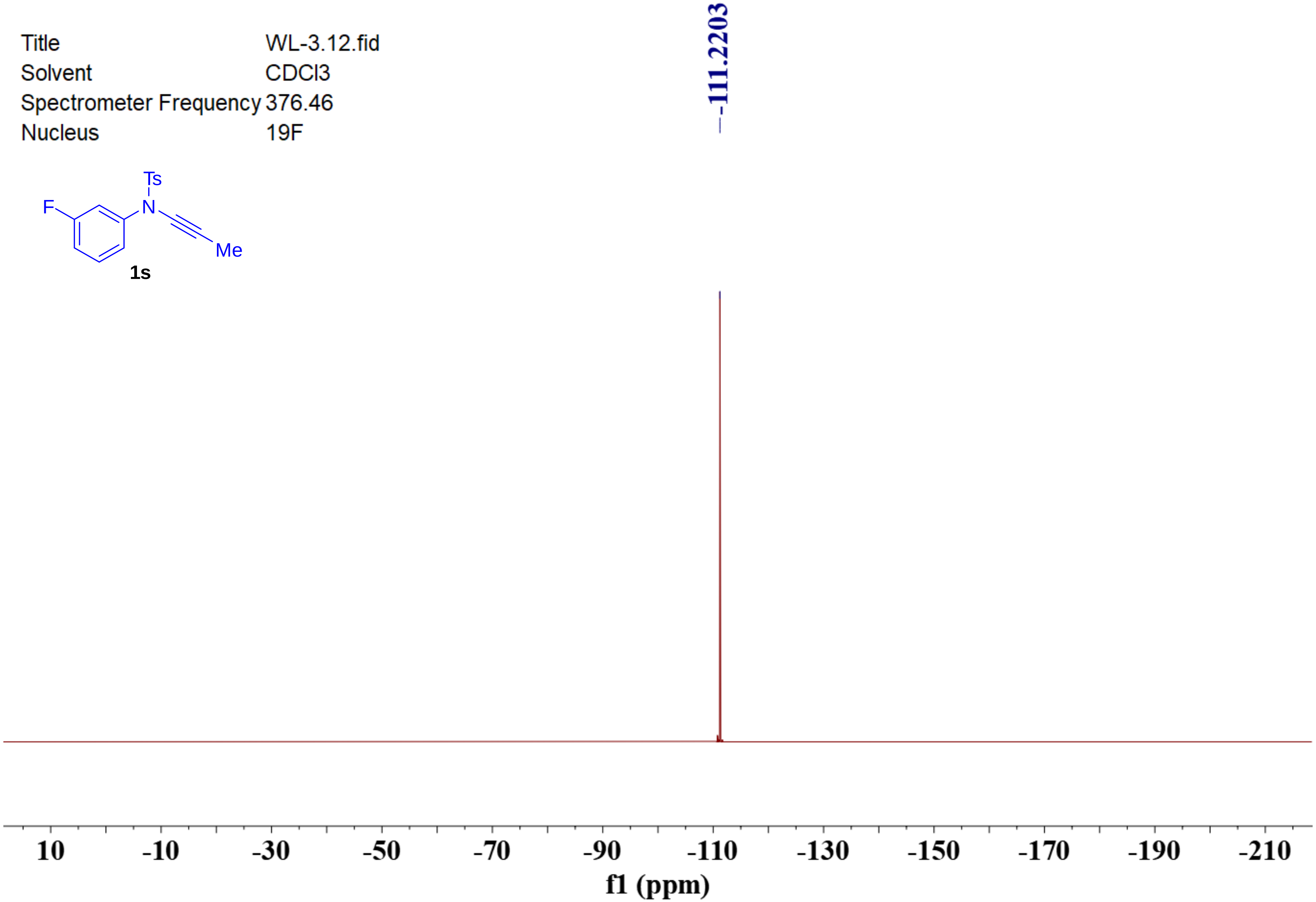
f1 (ppm)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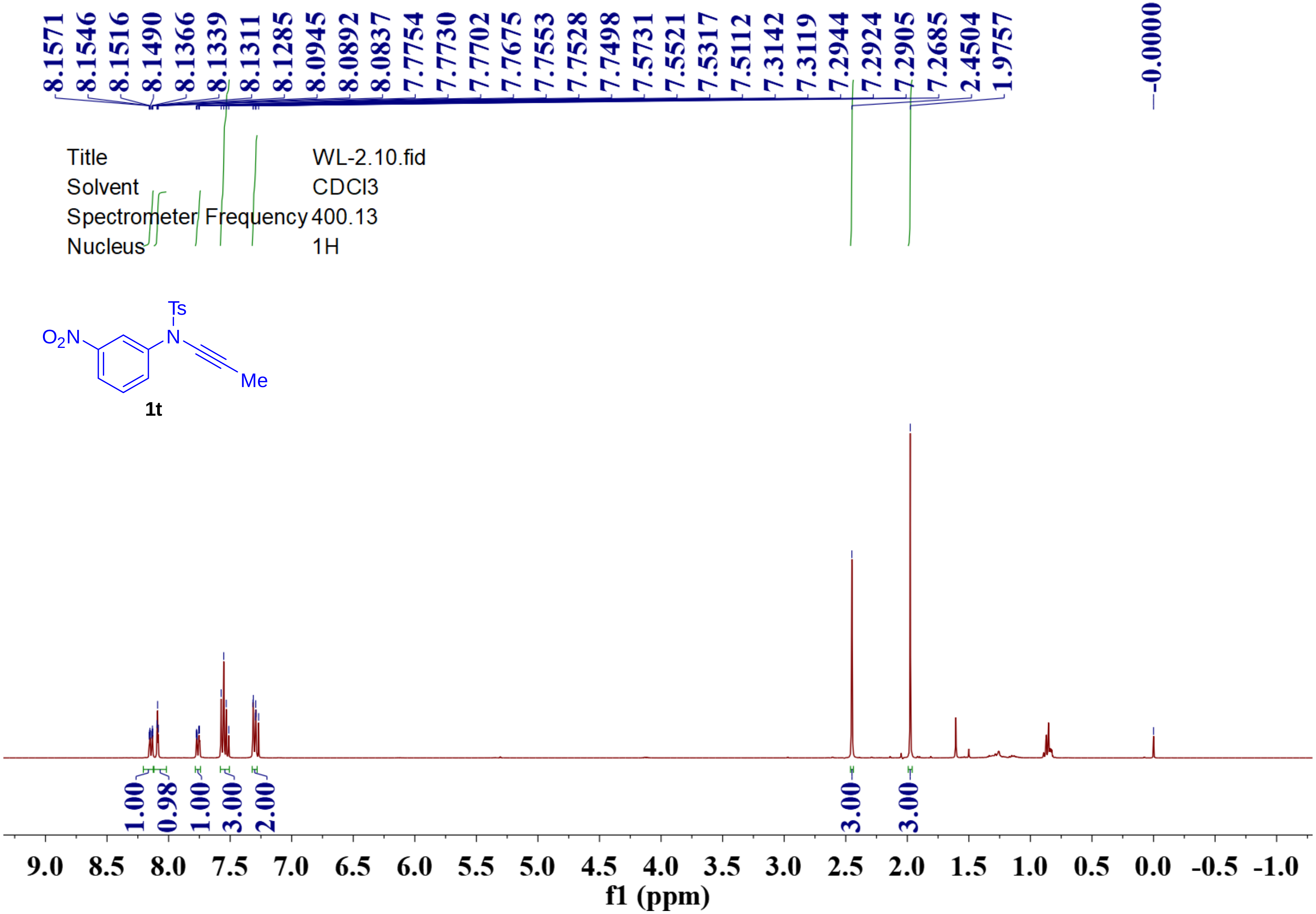
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Solvent CDCl3
Spectrometer Frequency 400.13
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N
Ts
Me
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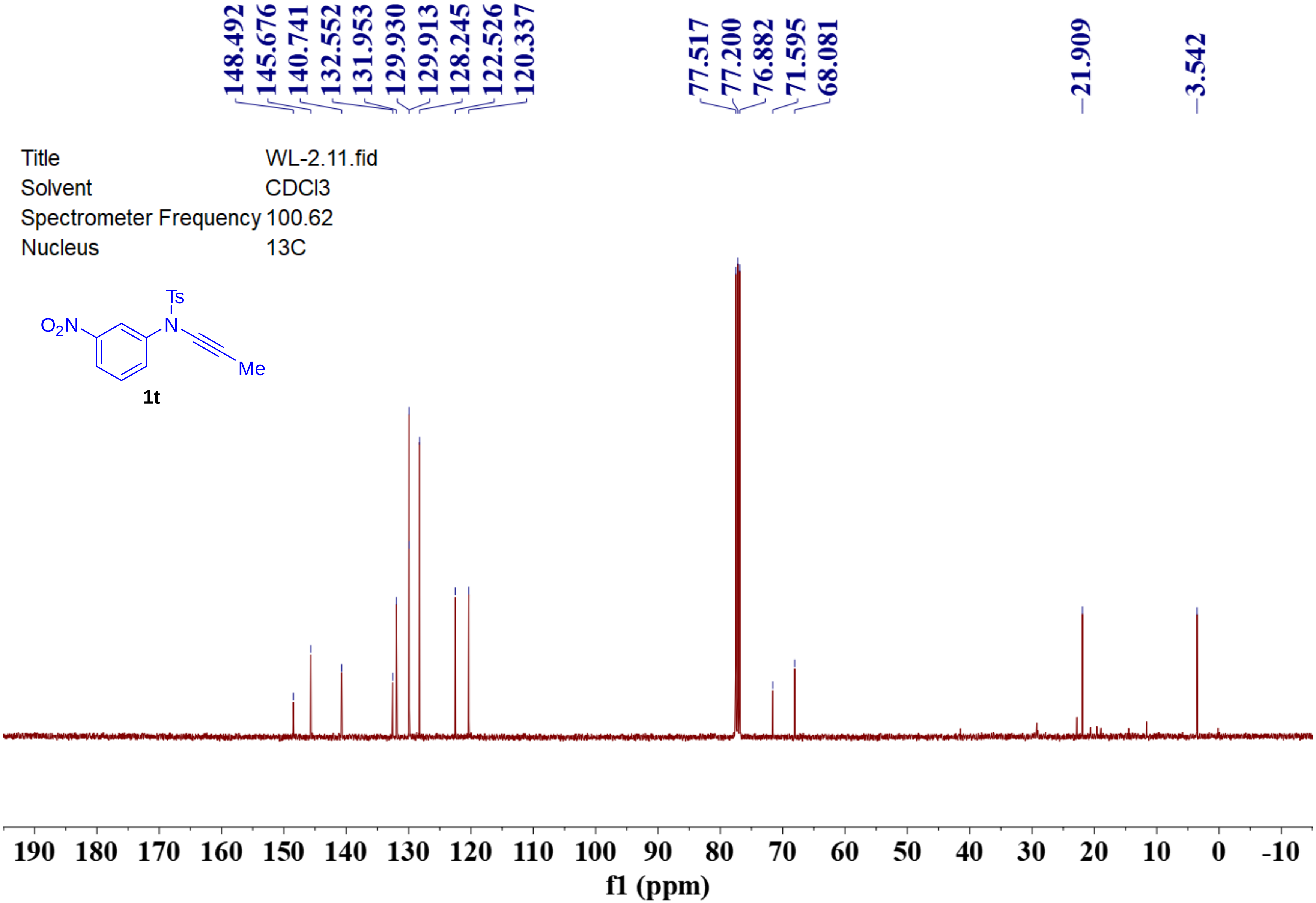
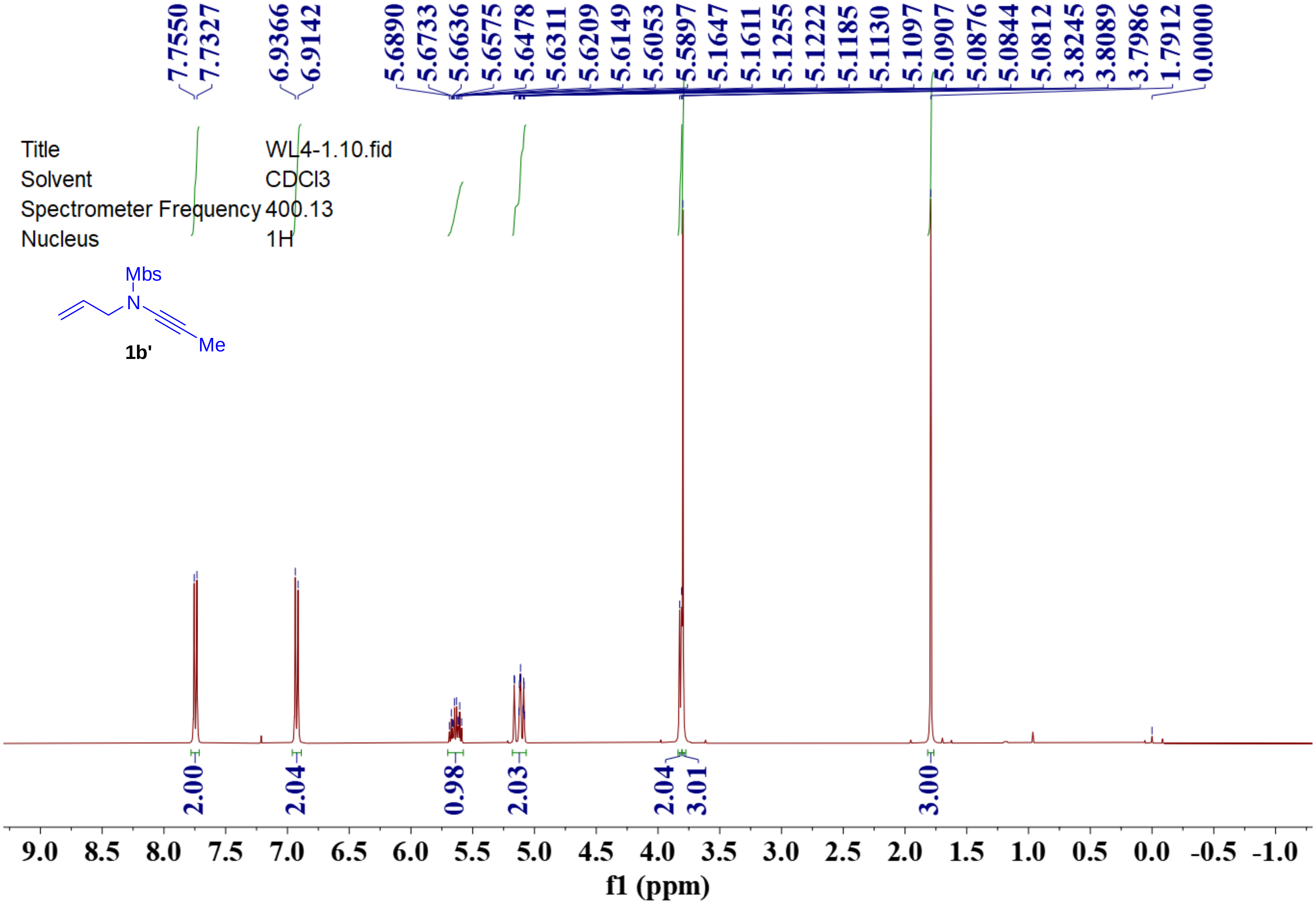
f1 (ppm)



Title WL-2.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

1t



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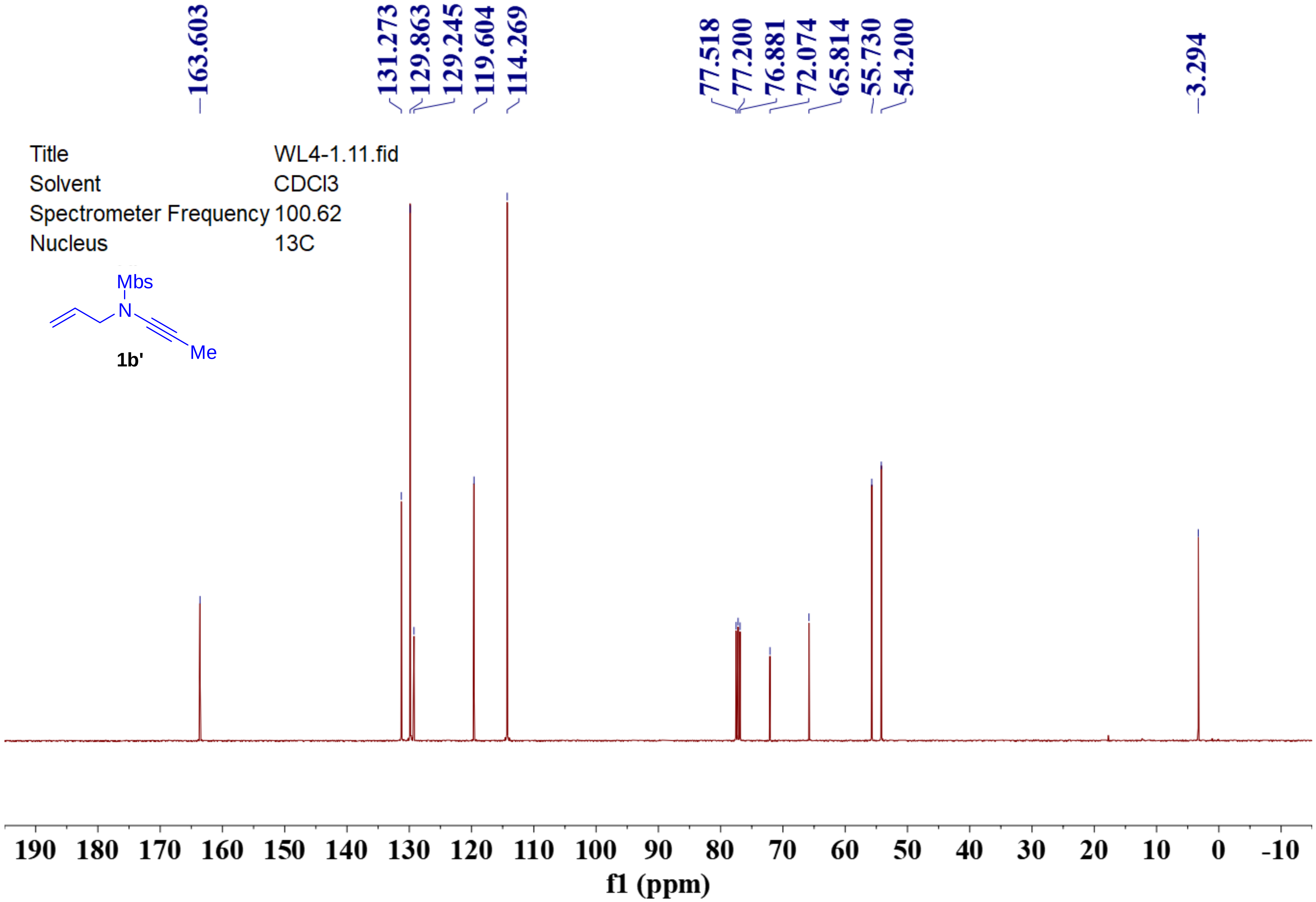
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

1b'



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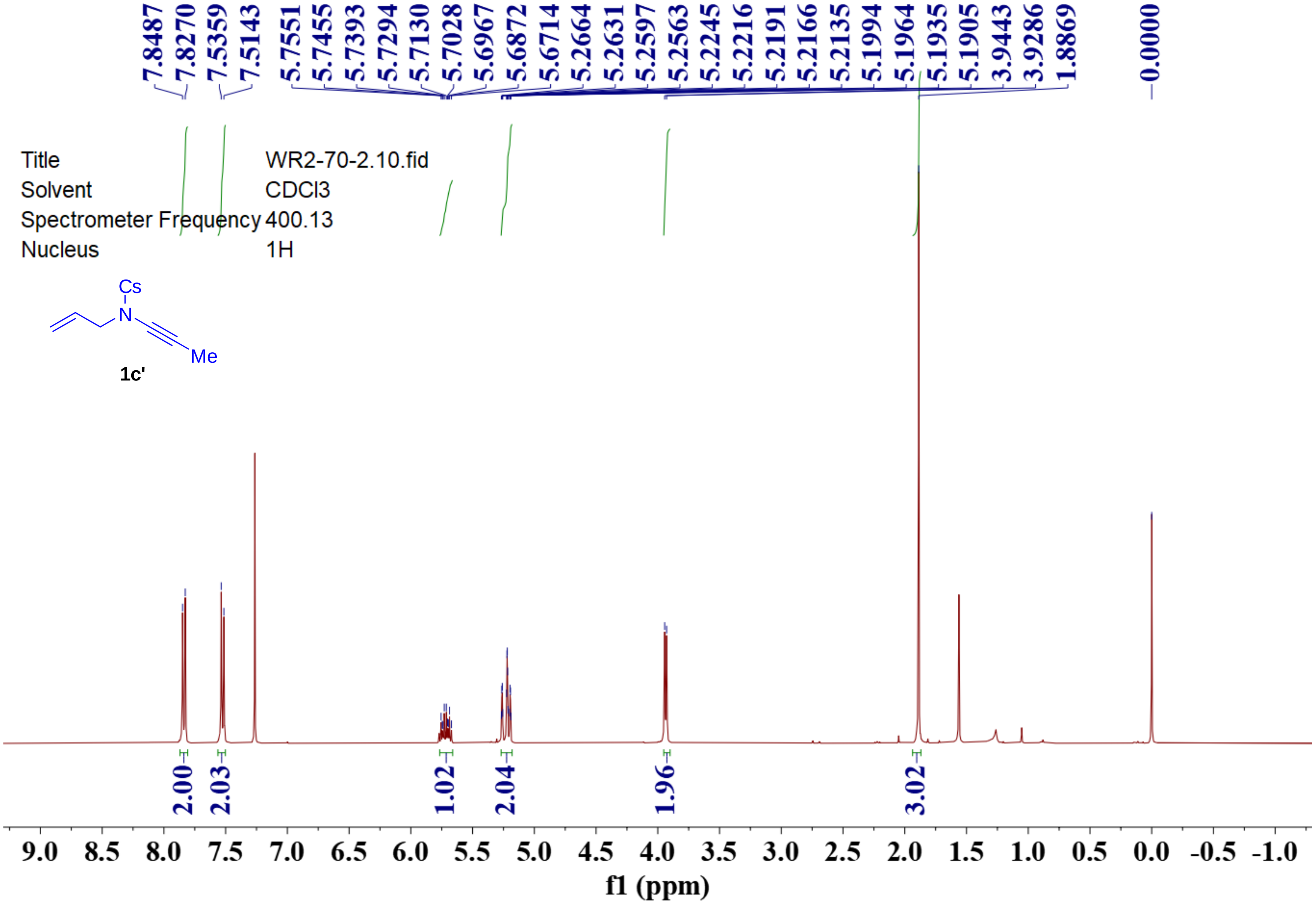
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C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5.2166
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Me
1c'
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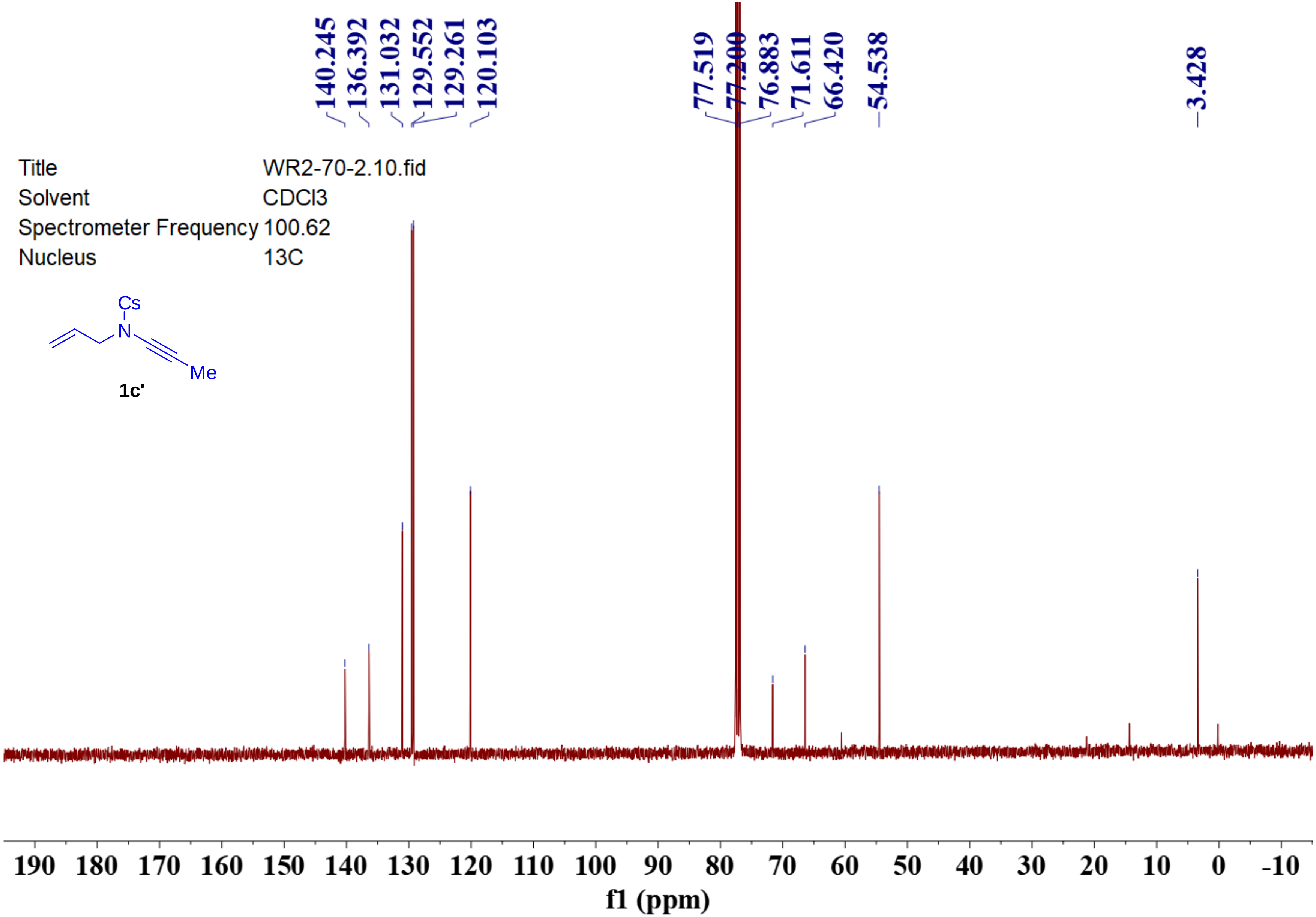
f1 (ppm)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Title WR2-70-2.10.fid
Solvent CDCl3
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Cs
N
Me
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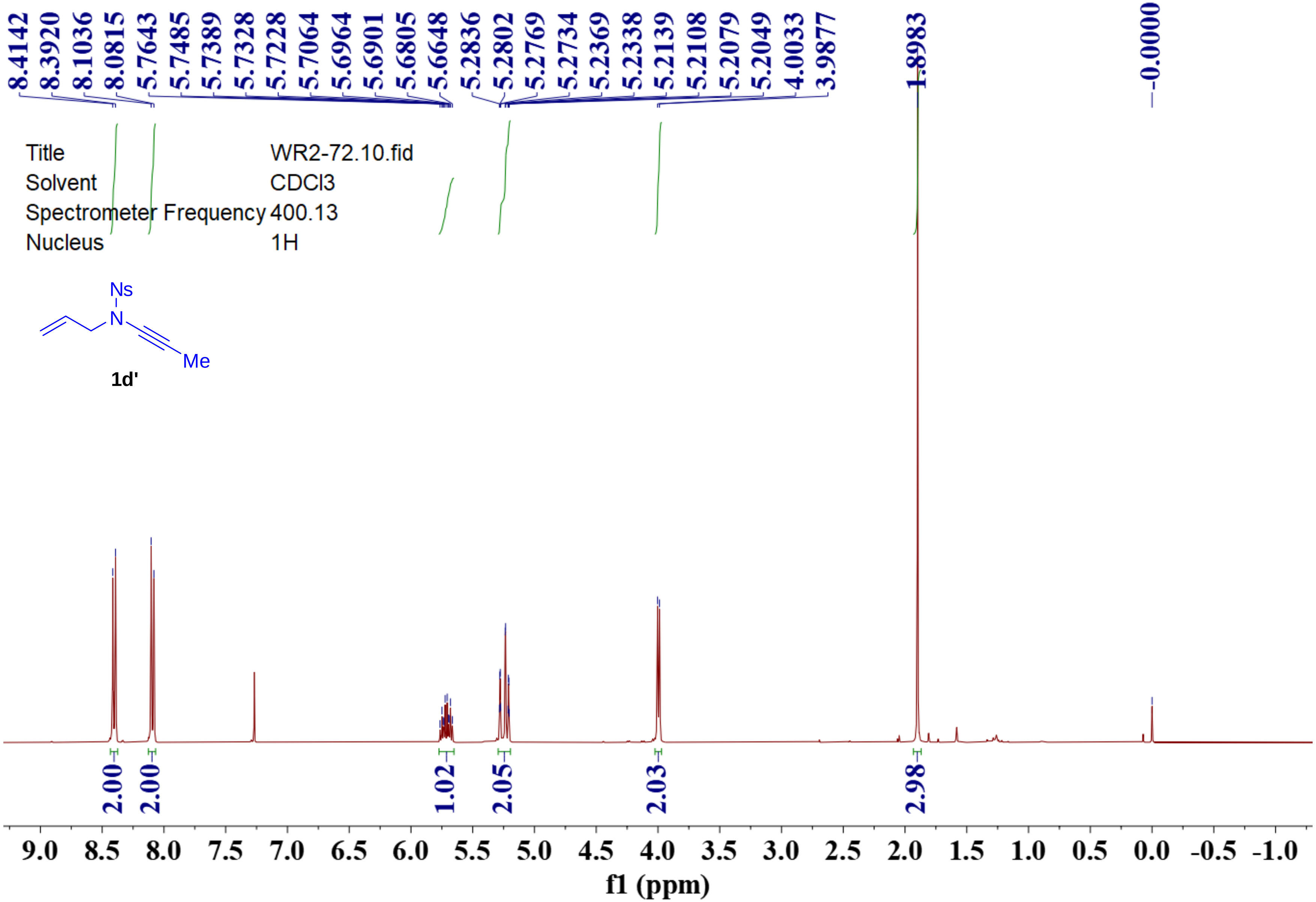
f1 (ppm)

1c'



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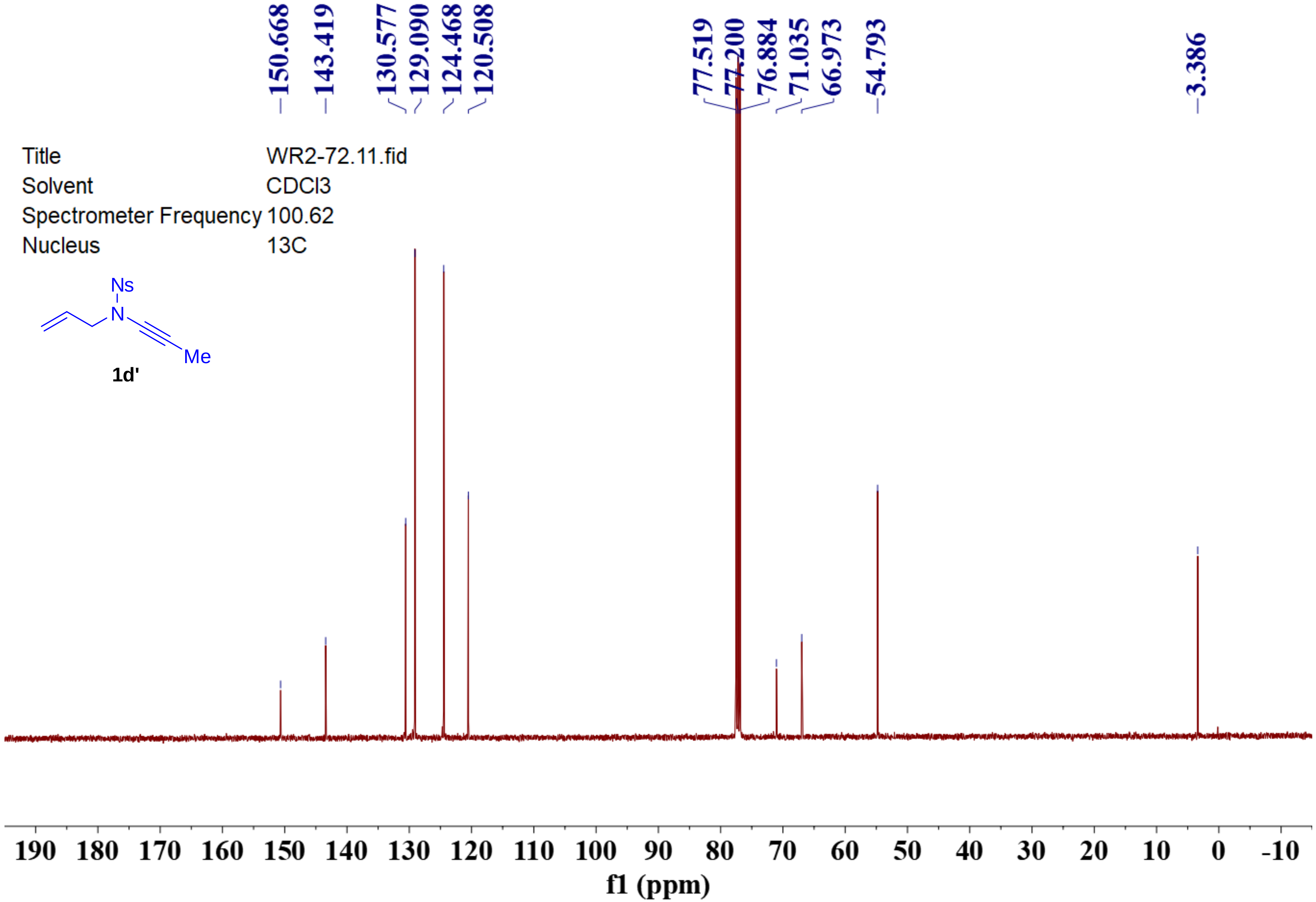
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

1d'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N
Me
190
180
170
160
150
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120
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100
90
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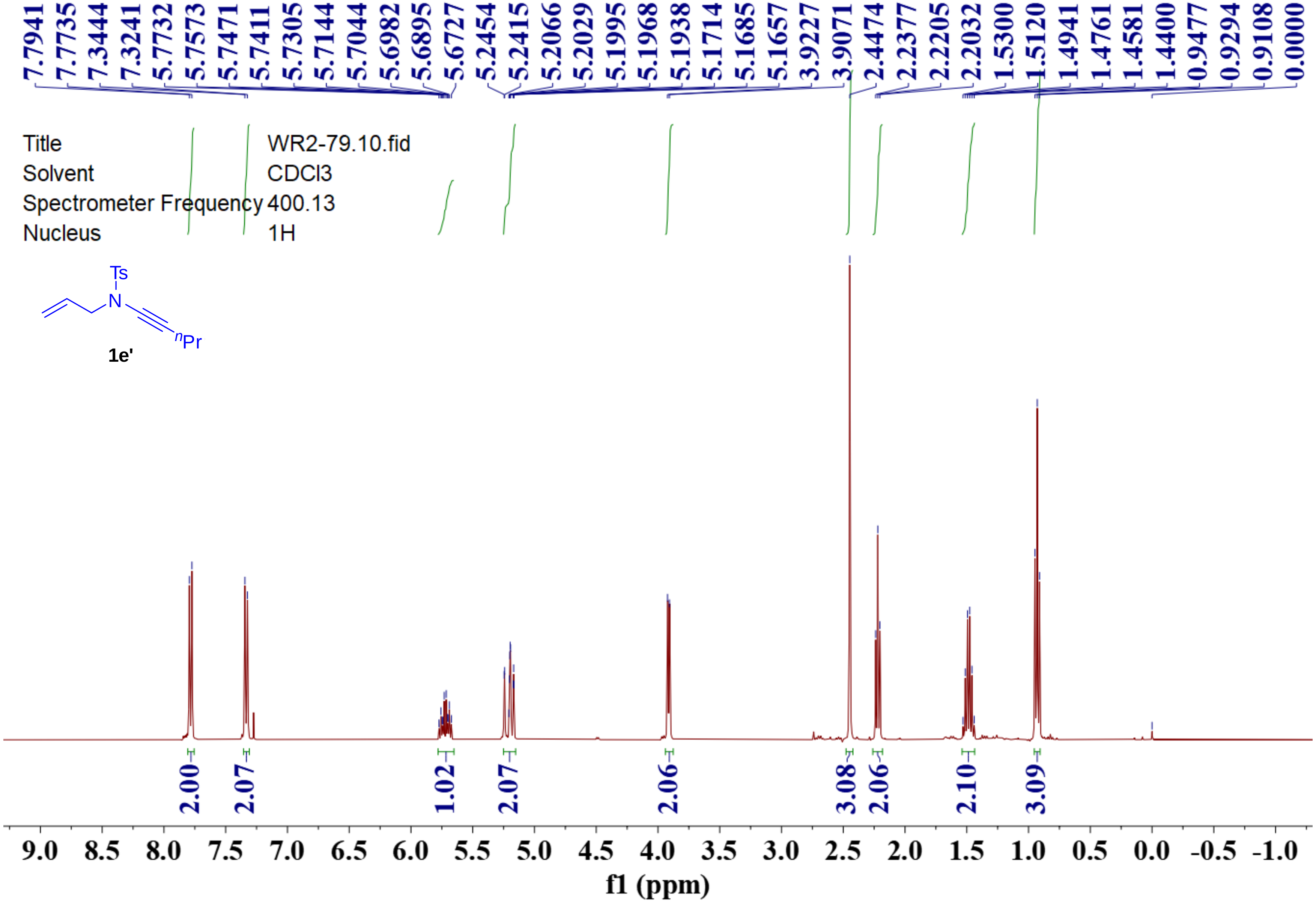
f1 (ppm)

1d'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f1 (ppm)

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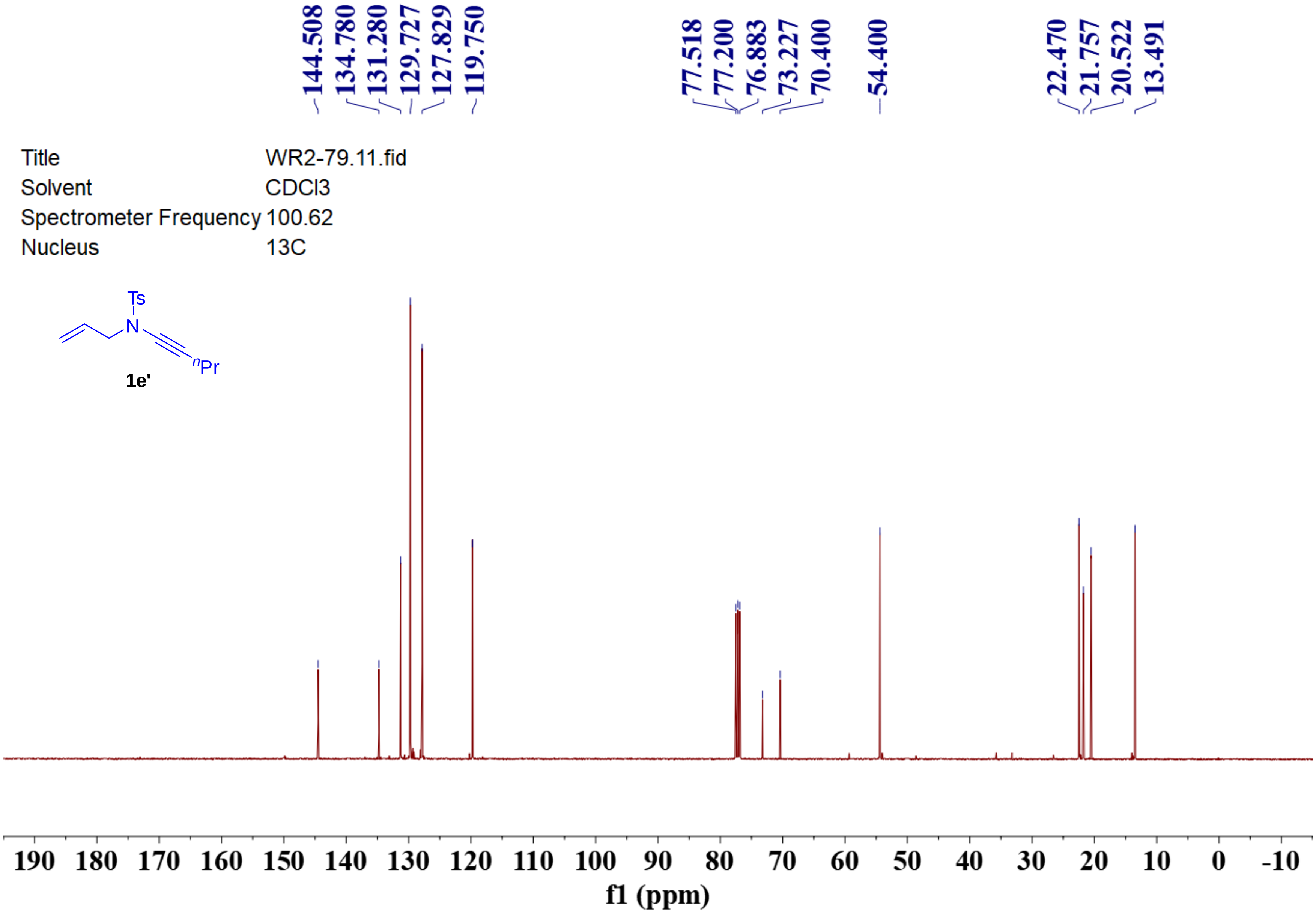
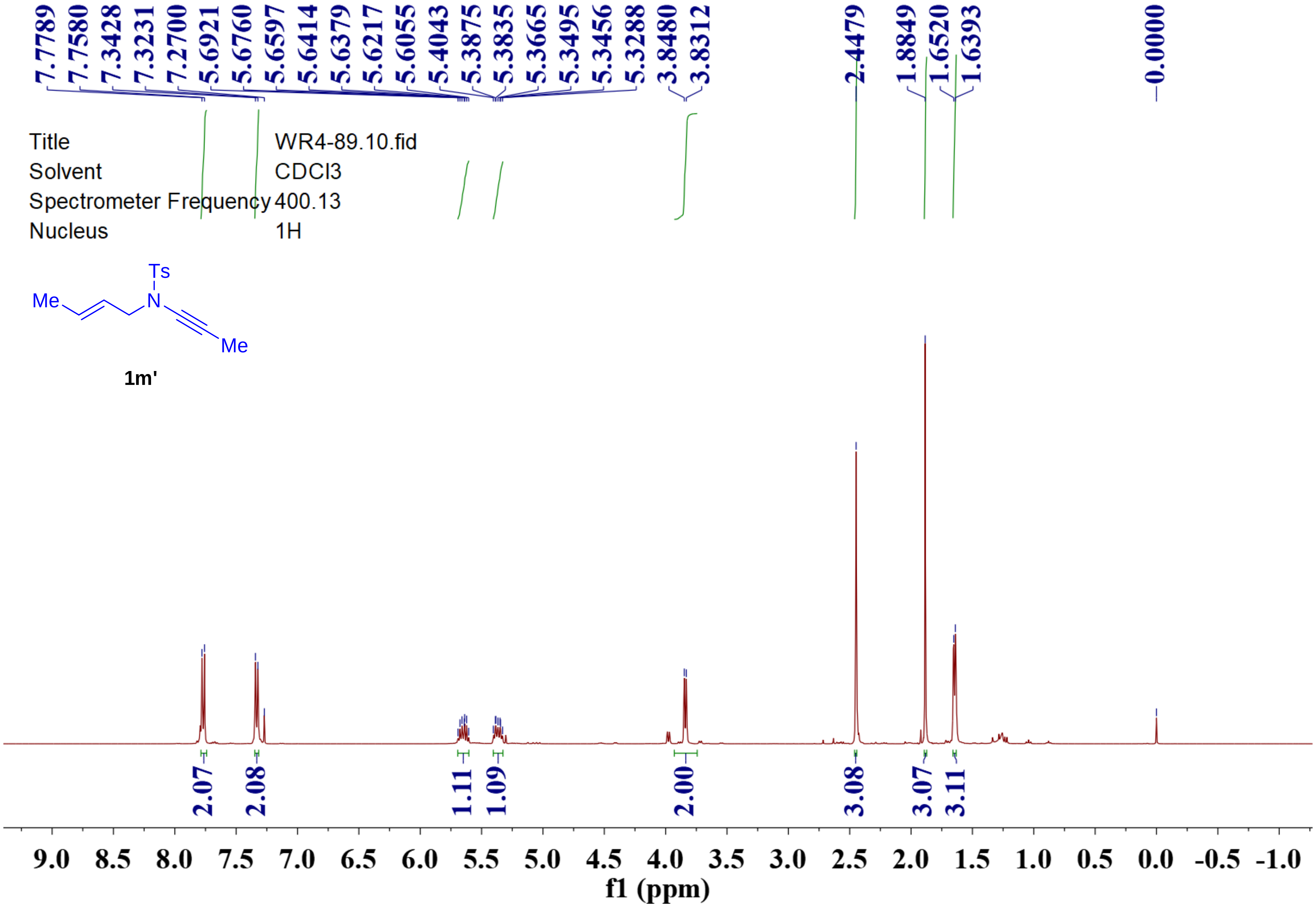
Spectrometer Frequency 100.62
Nucleus 13C

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N
Me
1m'
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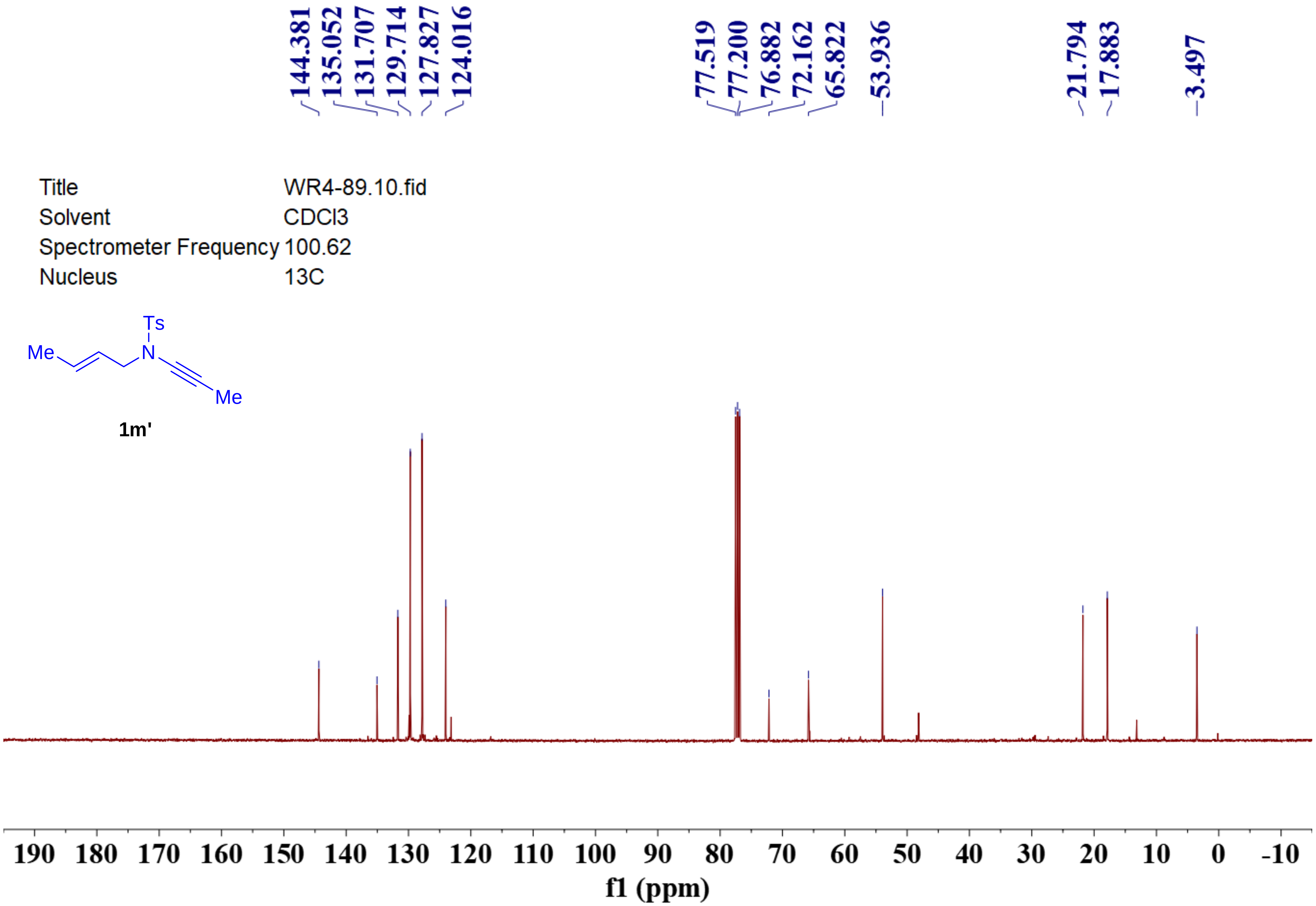

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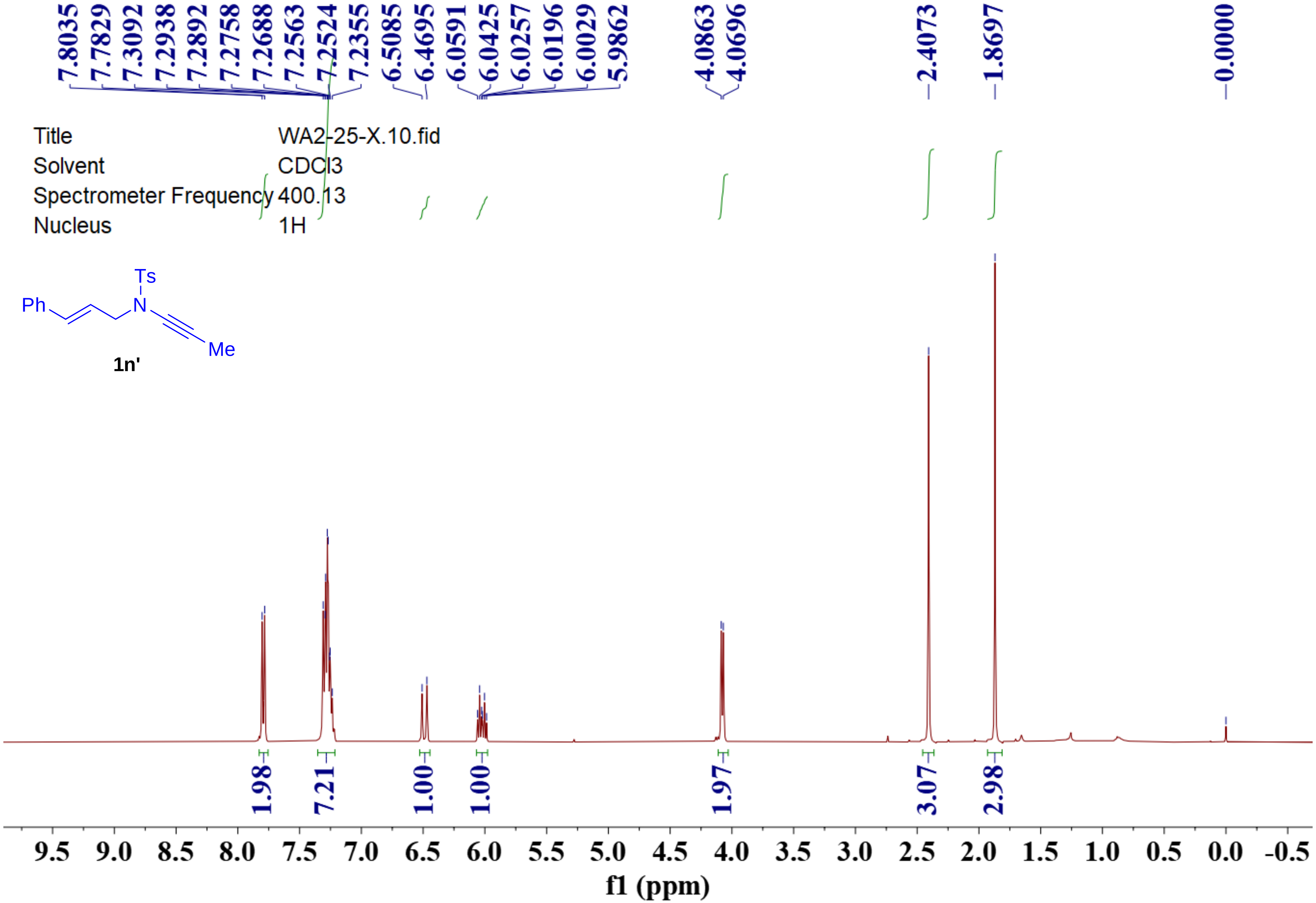
Spectrometer Frequency	100.62
Nucleus	13C

1m'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Spectrometer Frequency 400.13
Nucleus 1H
Ts
Ph
N
Me
1n'
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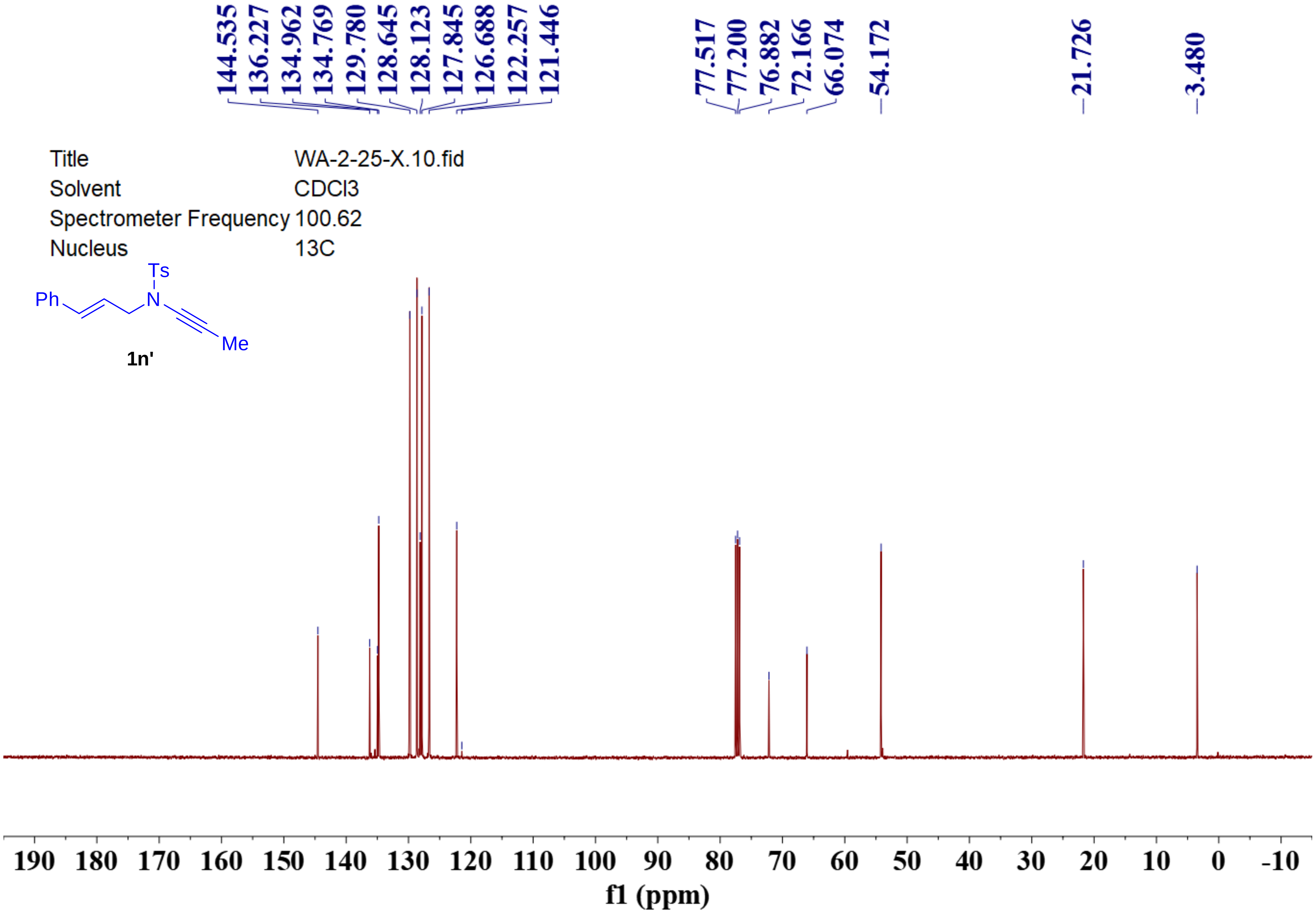
f1 (ppm)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77.517
77.200
76.882
72.166
66.074
54.172
21.726
3.480
Title WA-2-25-X.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
Ph
Ts
N
Me
1n'
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180
170
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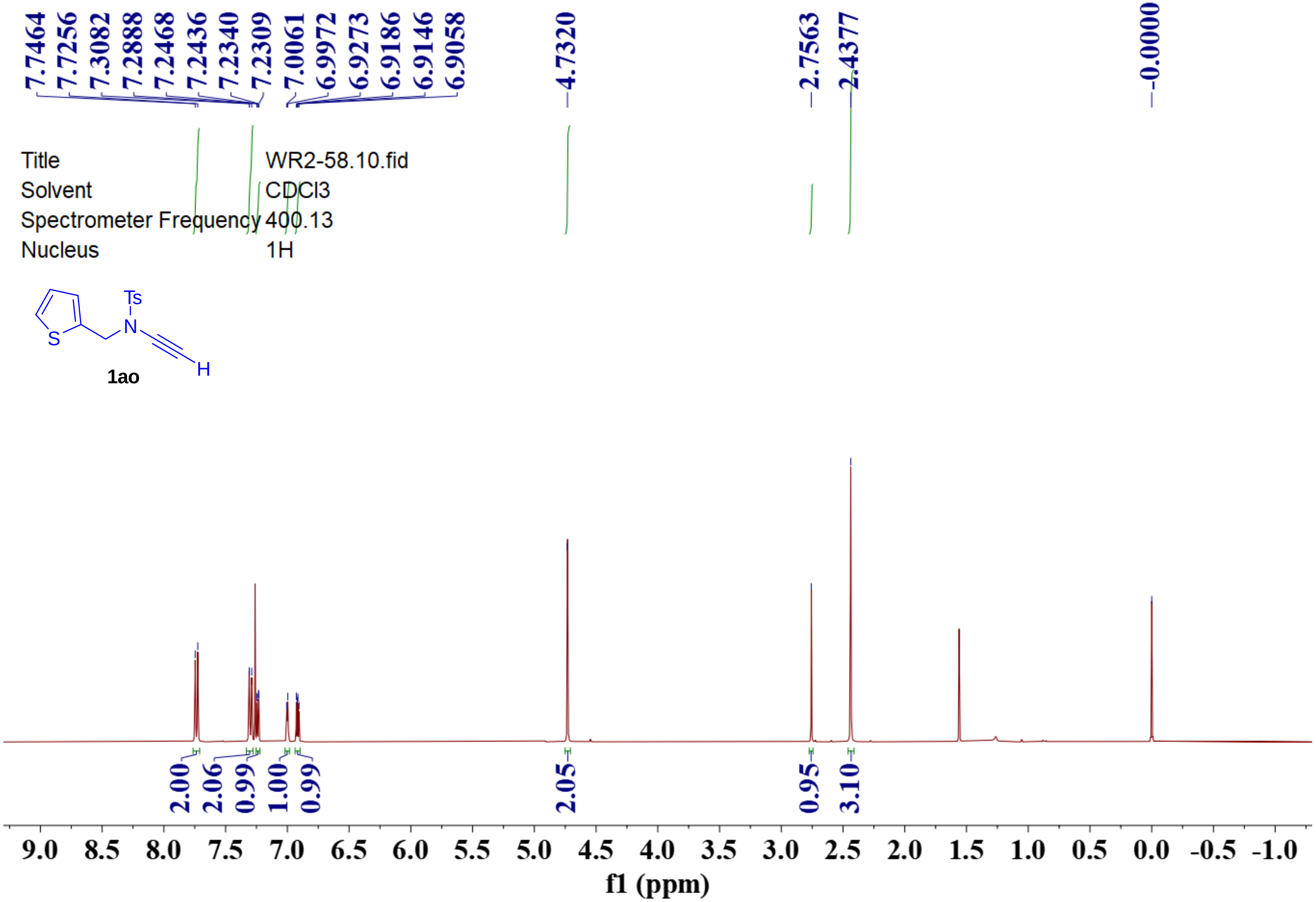
f1 (ppm)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Nucleus 1H
Ts
N
S
H
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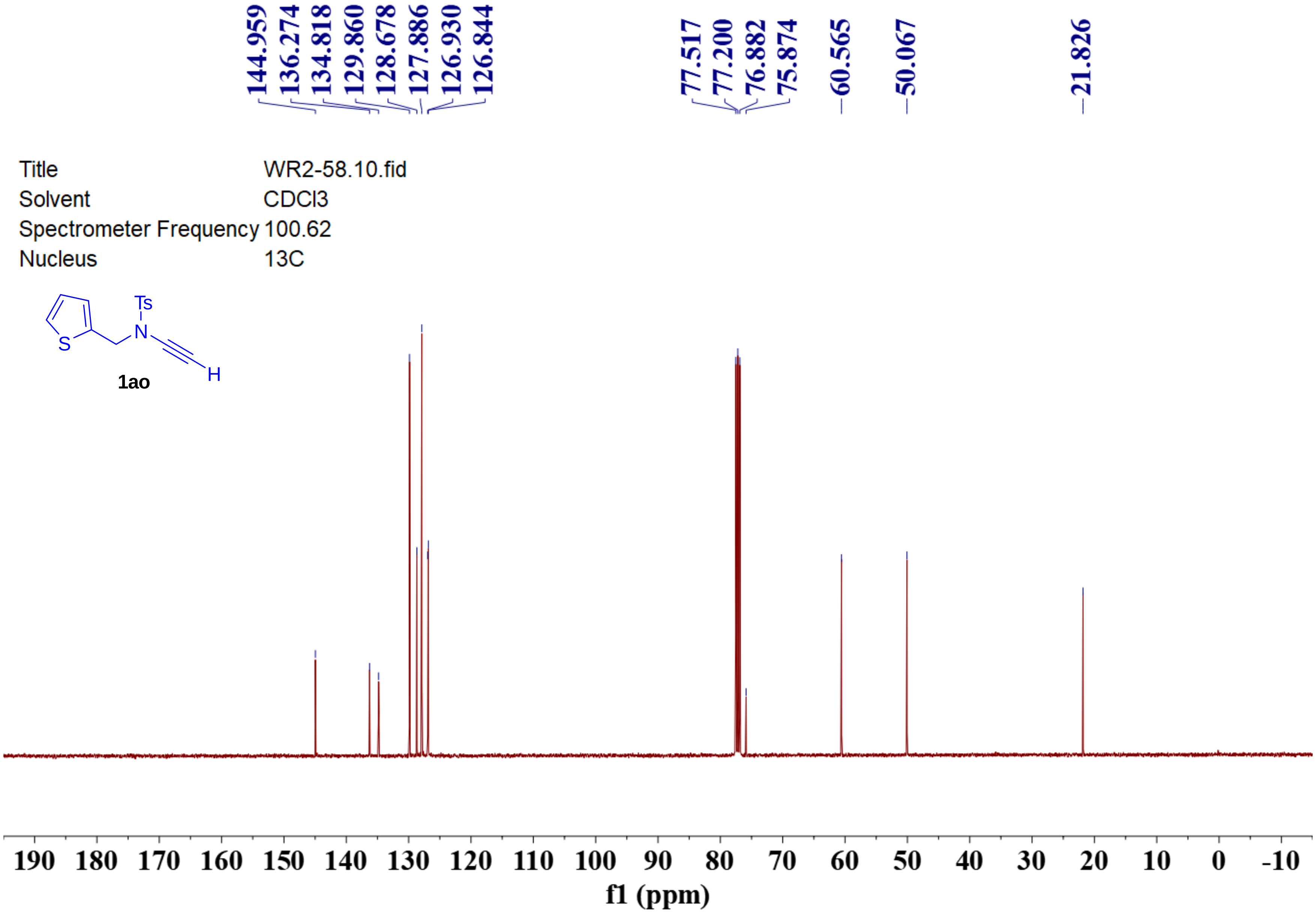
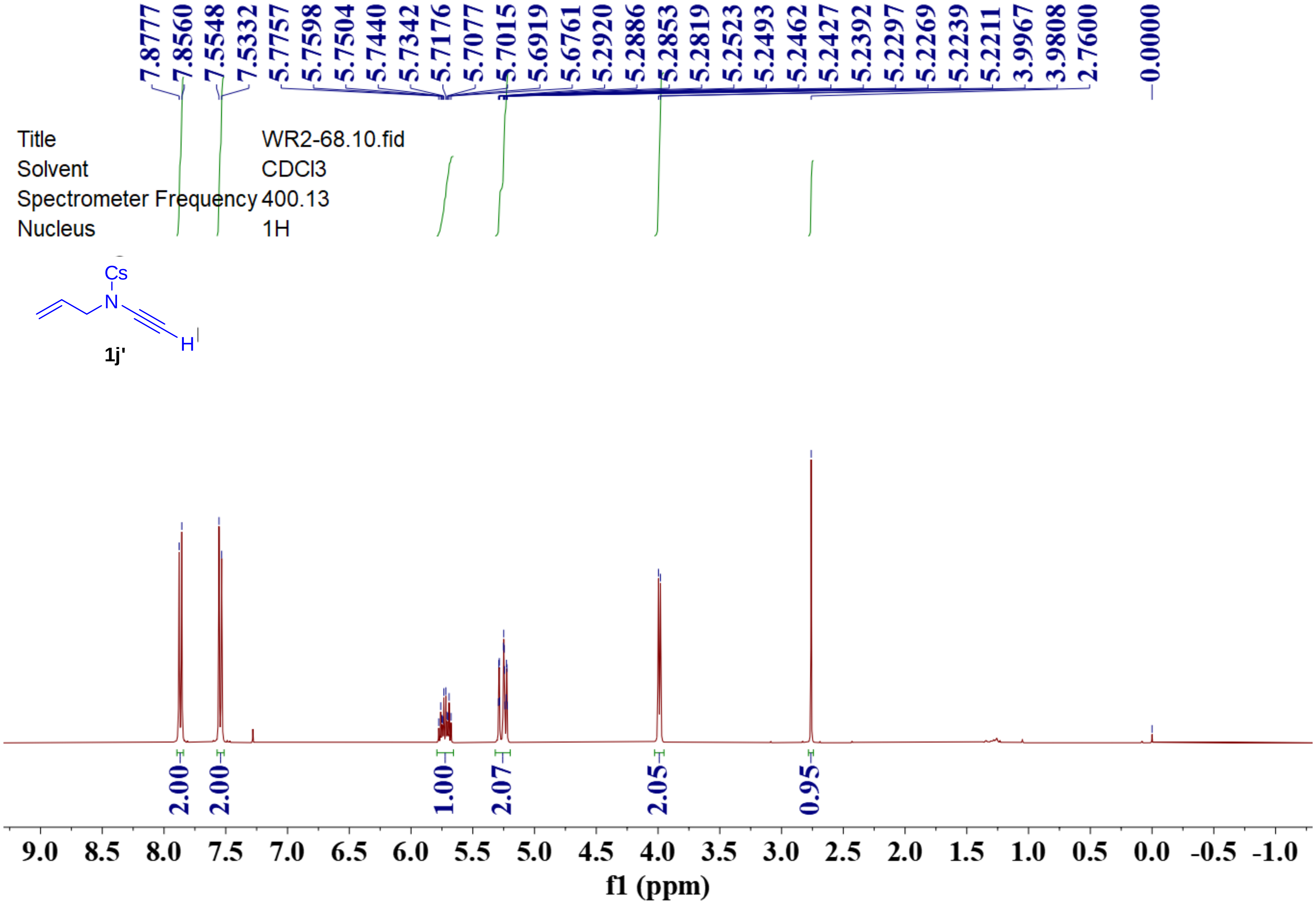
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

1ao

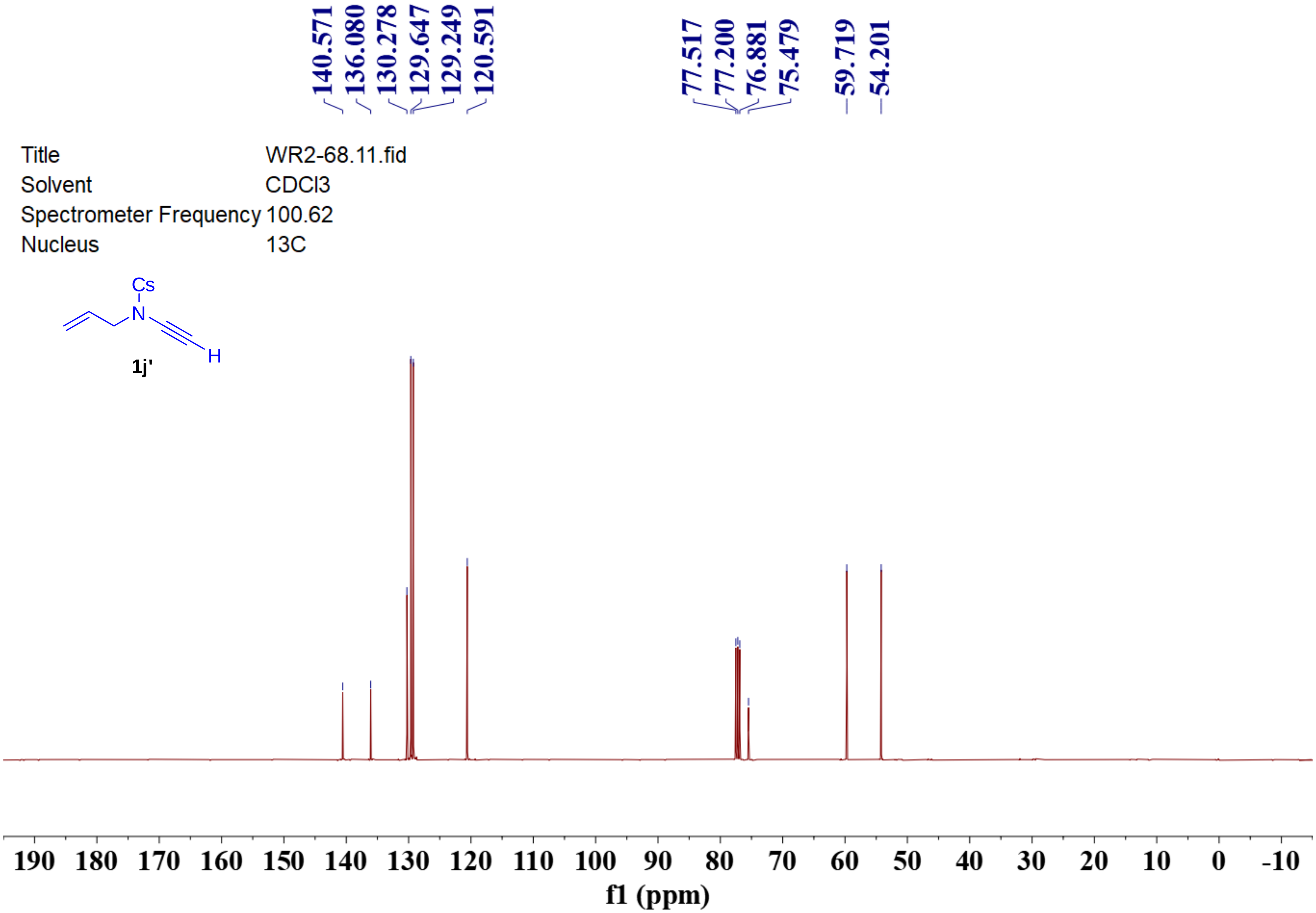


Title WR2-58.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

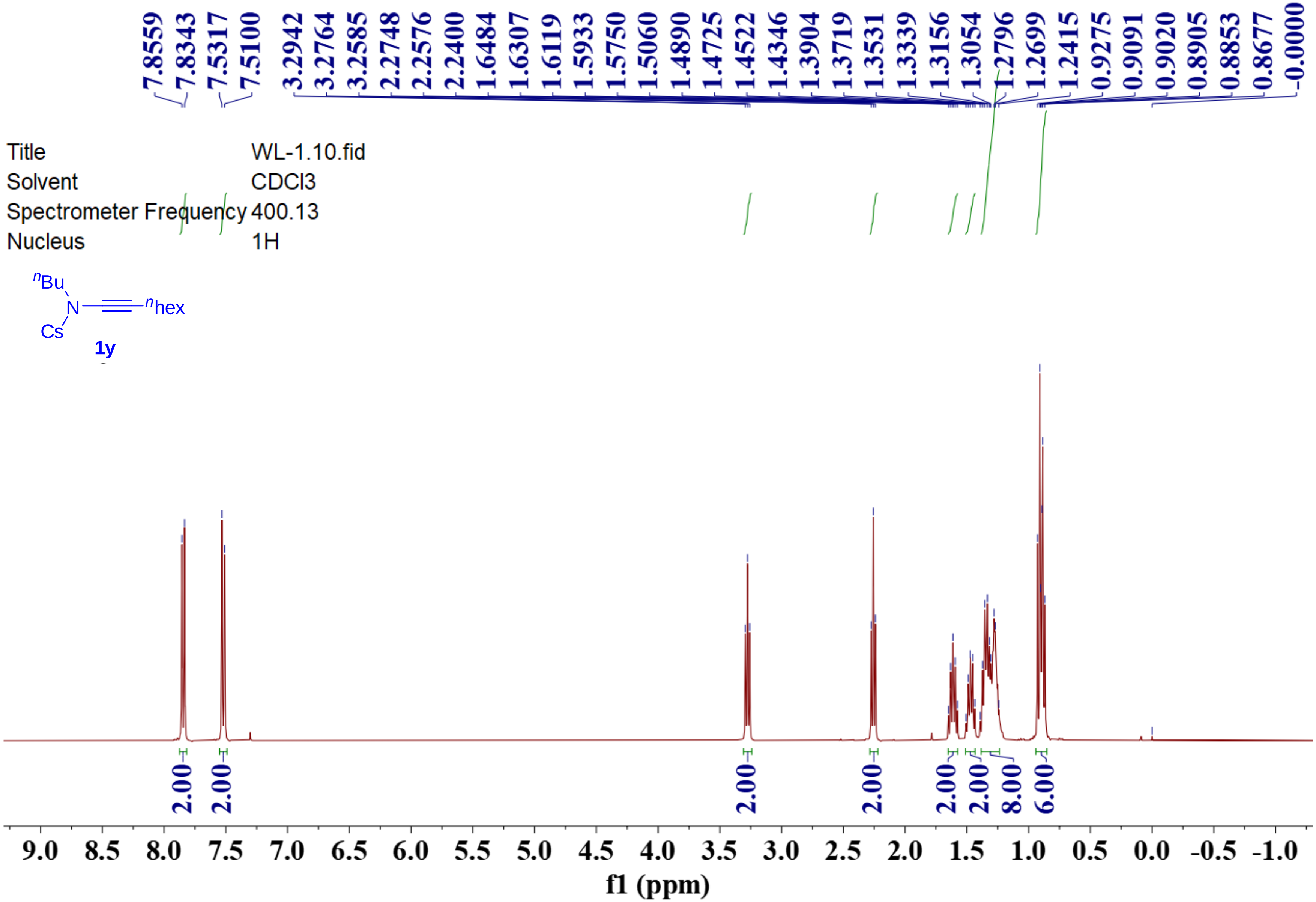
1ao

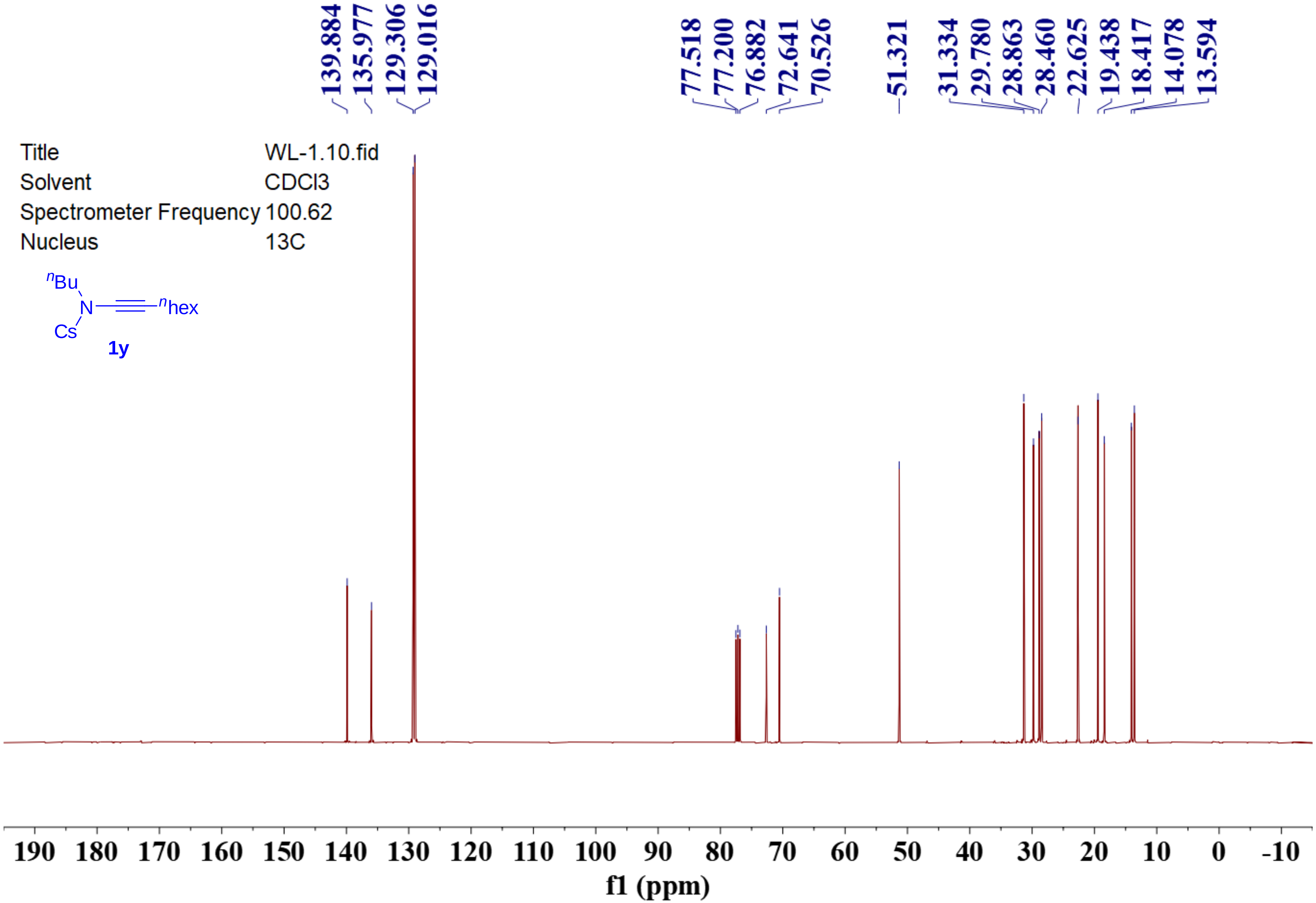

7.8777
7.8560
7.5548
7.5332
5.7757
5.7598
5.7504
5.7440
5.7342
5.7176
5.7077
5.7015
5.6919
5.6761
5.2920
5.2886
5.2853
5.2819
5.2523
5.2493
5.2462
5.2427
5.2392
5.2297
5.2269
5.2239
5.2211
3.9967
3.9808
2.7600
-0.0000
Title WR2-68.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Cs
N
H
1j'
2.00
2.00
1.00
2.07
2.05
0.95
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 -0.5 -1.0
f1 (ppm)

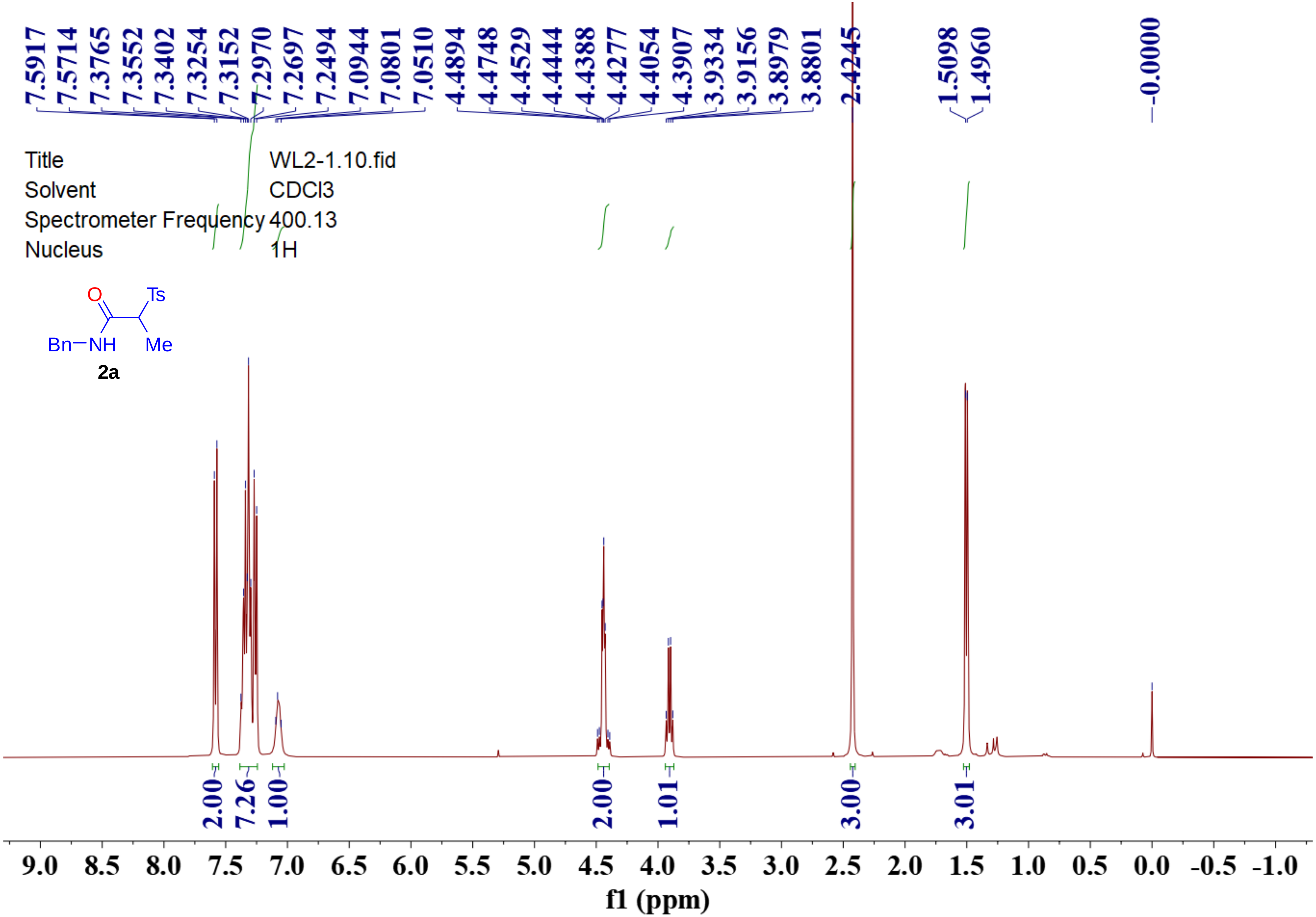
F

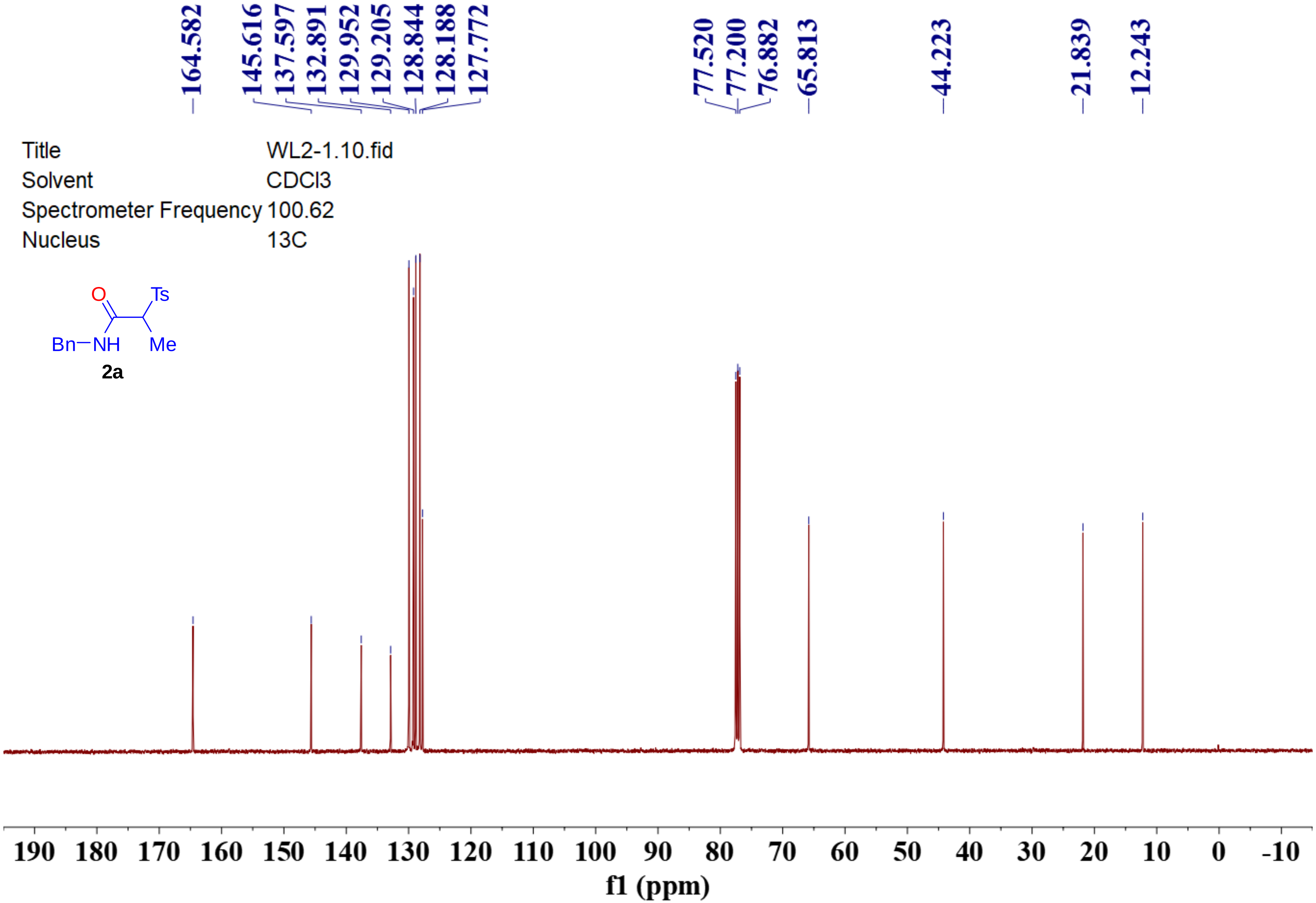


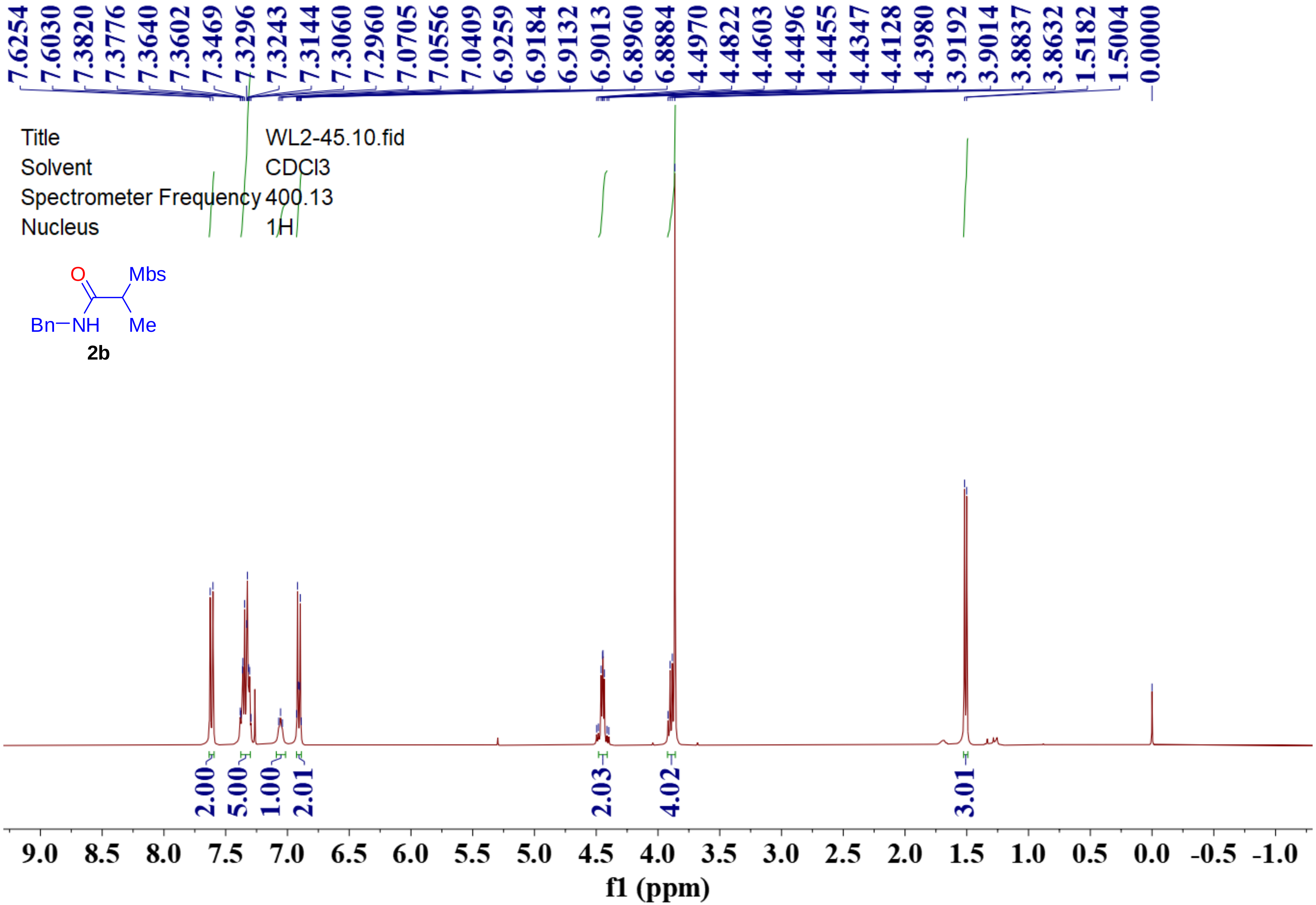
Title	WR2-68.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

7.8559
7.8343
7.5317
7.5100
3.2942
3.2764
3.2585
2.2748
2.2576
2.2400
1.6484
1.6307
1.6119
1.5933
1.5750
1.5060
1.4890
1.4725
1.4522
1.4346
1.3904
1.3719
1.3531
1.3339
1.3156
1.3054
1.2796
1.2699
1.2415
0.9275
0.9091
0.9020
0.8905
0.8853
0.8677
-0.0000
Title
WL-1.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
^{n}Bu
N
^{n}hex
Cs
1y
2.00
2.00
2.00
2.00
2.00
2.00
8.00
6.00
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
-0.5
-1.0
f1 (ppm)

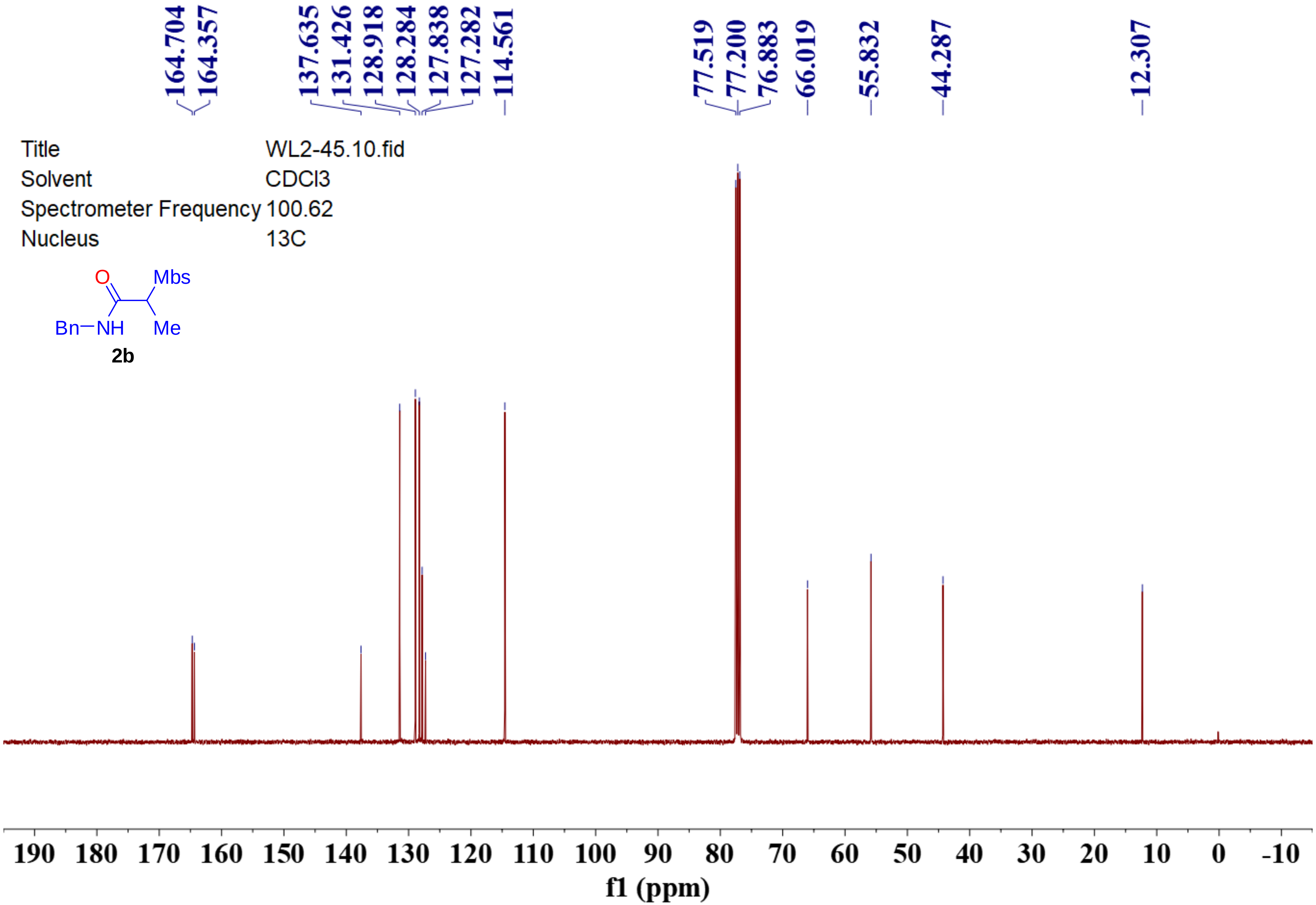

139.884
135.977
129.306
129.016
77.518
77.200
76.882
72.641
70.526
51.321
31.334
29.780
28.863
28.460
22.625
19.438
18.417
14.078
13.594
Title WL-1.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
ⁿBu
N
ⁿhex
Cs
1y
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.5917
7.5714
7.3765
7.3552
7.3402
7.3254
7.3152
7.2970
7.2697
7.2494
7.0944
7.0801
7.0510
4.4894
4.4748
4.4529
4.4444
4.4388
4.4277
4.4054
4.3907
3.9334
3.9156
3.8979
3.8801
2.4245
1.5098
1.4960
-0.0000
Title WL2-1.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn
NH
Me
2a
2.00
7.26
1.00
2.00
1.01
3.00
3.01
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
-0.5
-1.0
f1 (ppm)

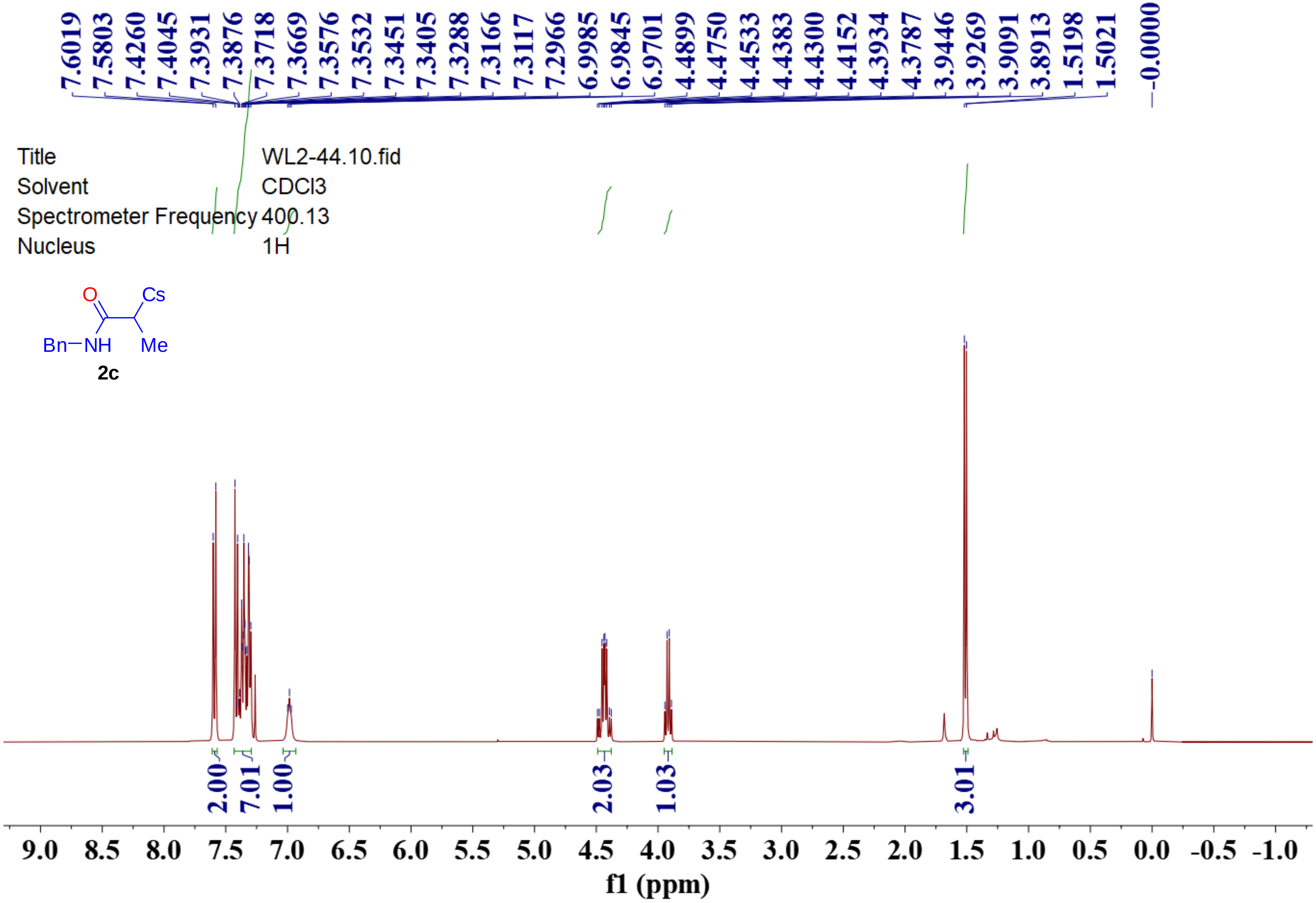

164.582
145.616
137.597
132.891
129.952
129.205
128.844
128.188
127.772
77.520
77.200
76.882
65.813
44.223
21.839
12.243
Title WL2-1.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Bn—NH
Me
2a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

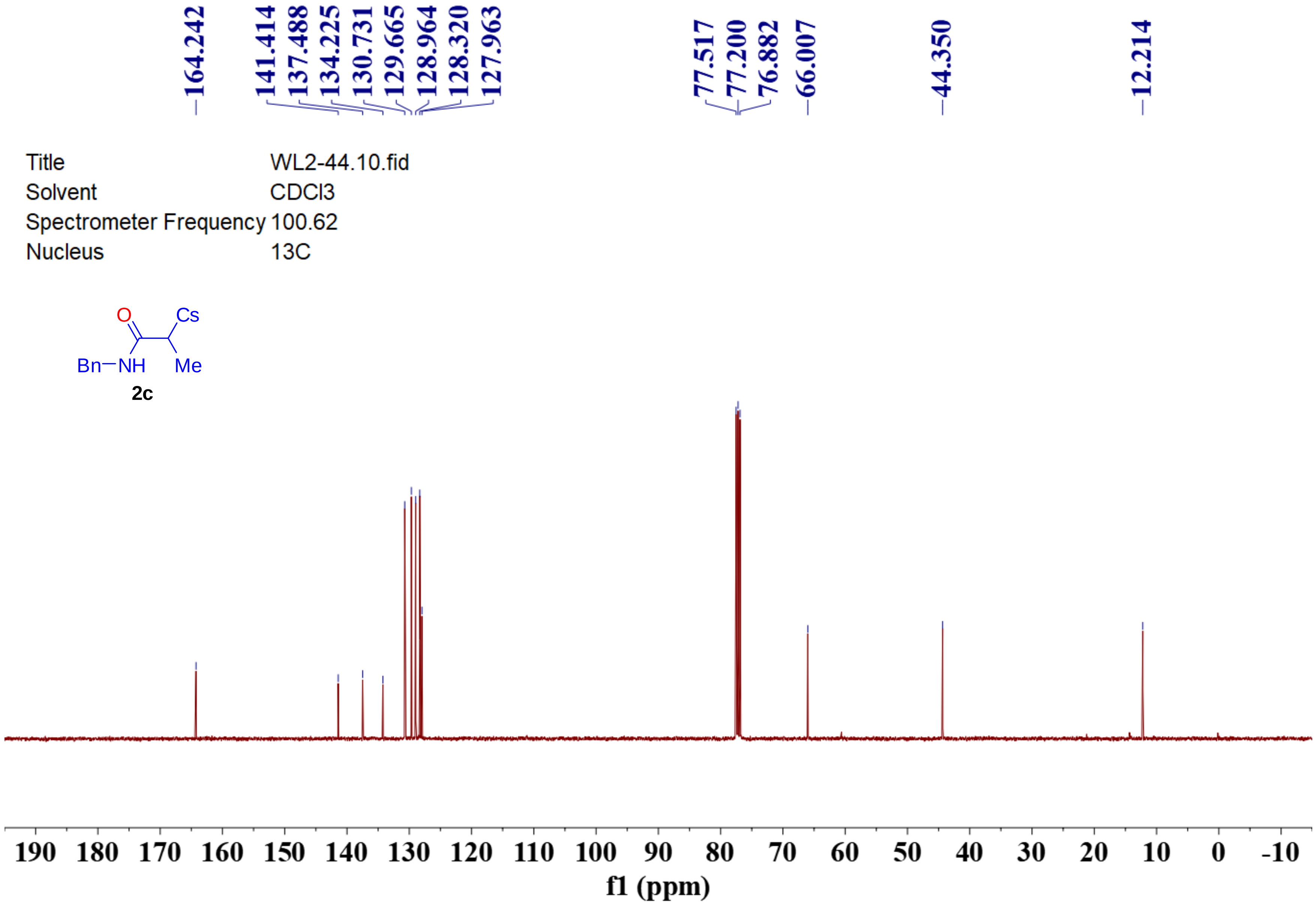

7.6254
7.6030
7.3820
7.3776
7.3640
7.3602
7.3469
7.3296
7.3243
7.3144
7.3060
7.2960
7.0705
7.0556
7.0409
6.9259
6.9184
6.9132
6.9013
6.8960
6.8884
4.4970
4.4822
4.4603
4.4496
4.4455
4.4347
4.4128
4.3980
3.9192
3.9014
3.8837
3.8632
1.5182
1.5004
0.0000
Title WL2-45.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Mbs
Bn
NH
Me
2.00
5.00
1.00
2.01
2.03
4.02
3.01
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
-0.5
-1.0
f1 (ppm)

2b



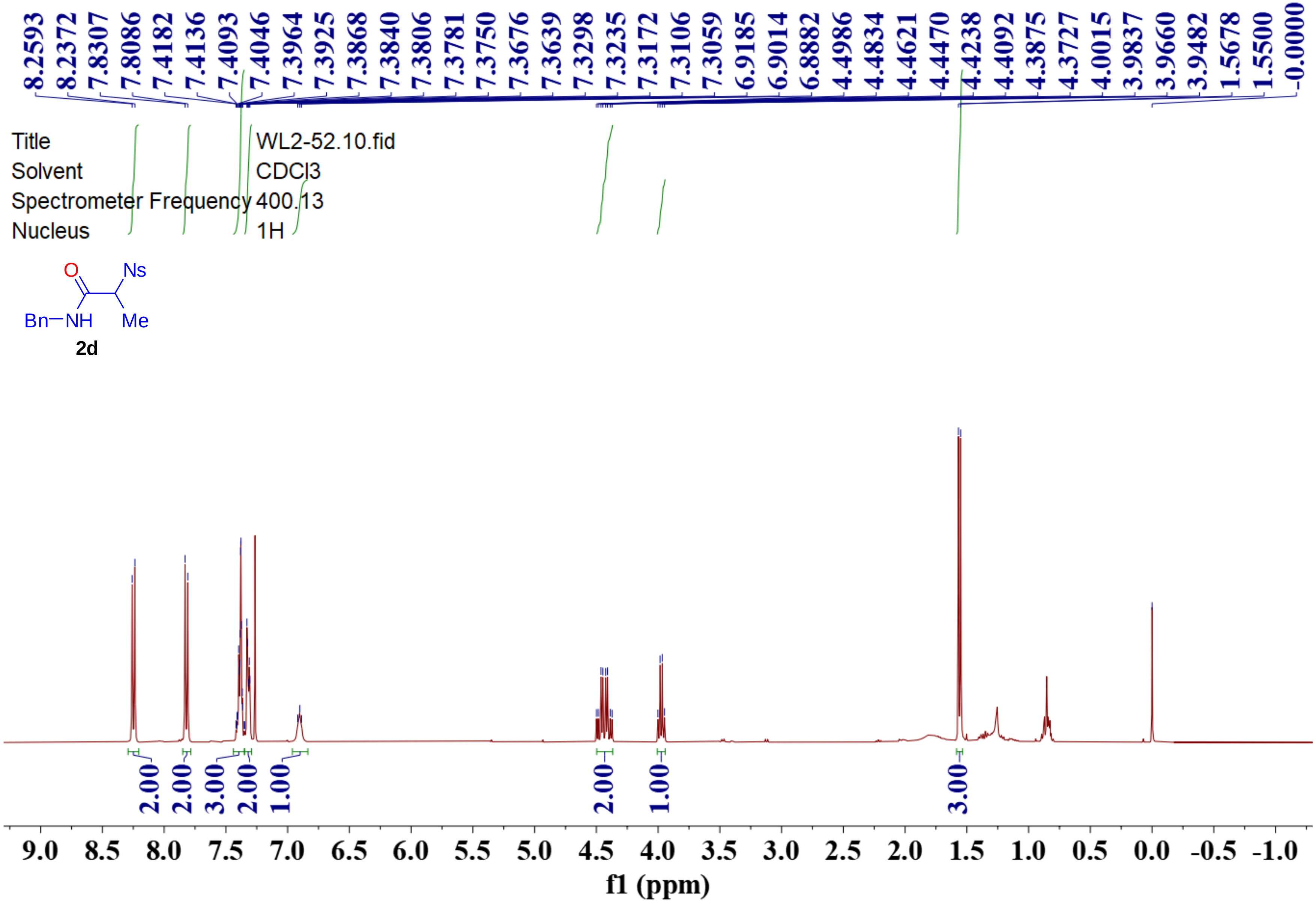
Title WL2-45.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

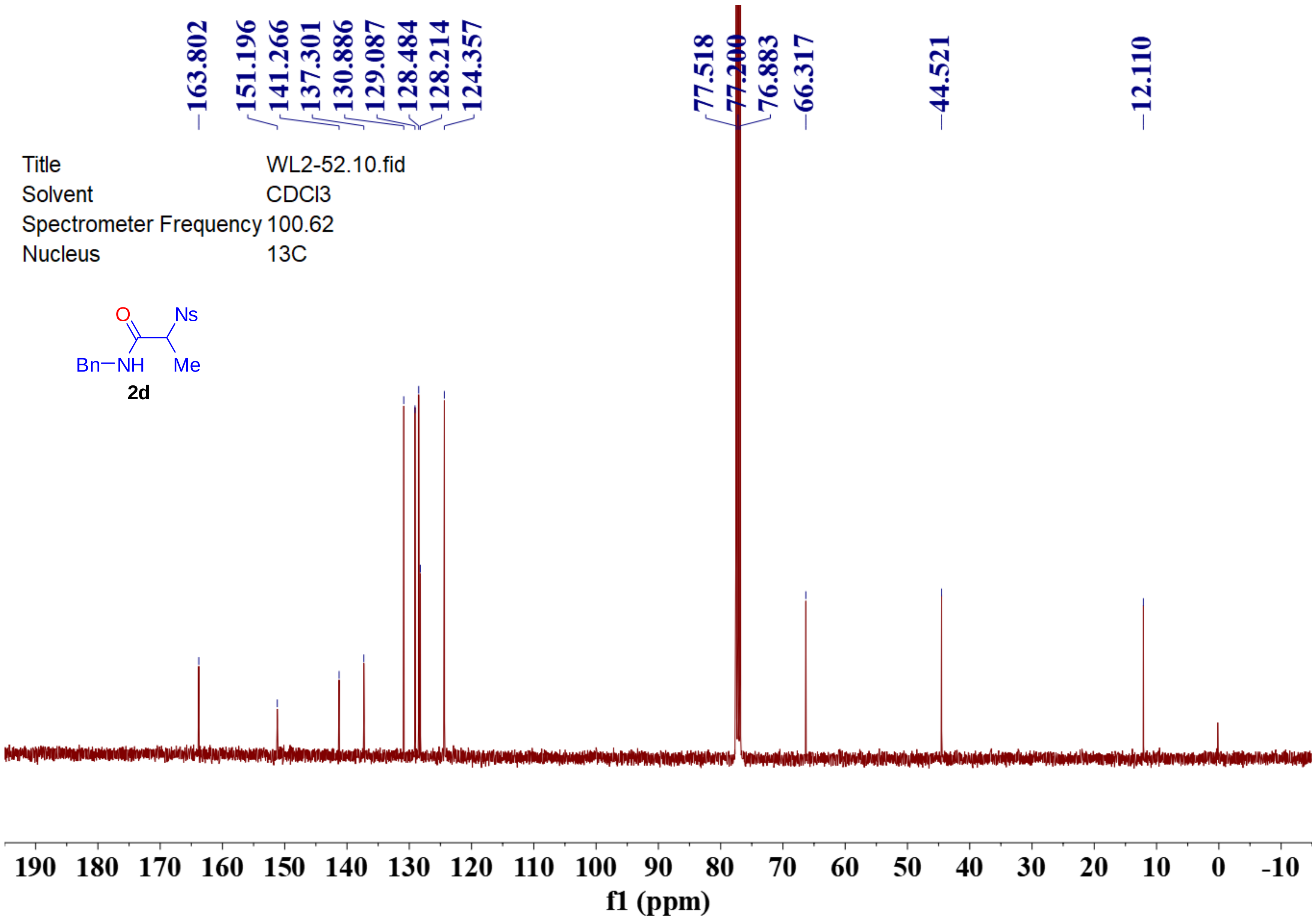

7.6019
7.5803
7.4260
7.4045
7.3931
7.3876
7.3718
7.3669
7.3576
7.3532
7.3451
7.3405
7.3288
7.3166
7.3117
7.2966
6.9985
6.9845
6.9701
4.4899
4.4750
4.4533
4.4383
4.4300
4.4152
4.3934
4.3787
3.9446
3.9269
3.9091
3.8913
1.5198
1.5021
-0.0000
Title WL2-44.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Cs
Bn−NH
Me
2c
2.00
7.01
1.00
2.03
1.03
3.01
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
-0.5
-1.0
f1 (ppm)



2c

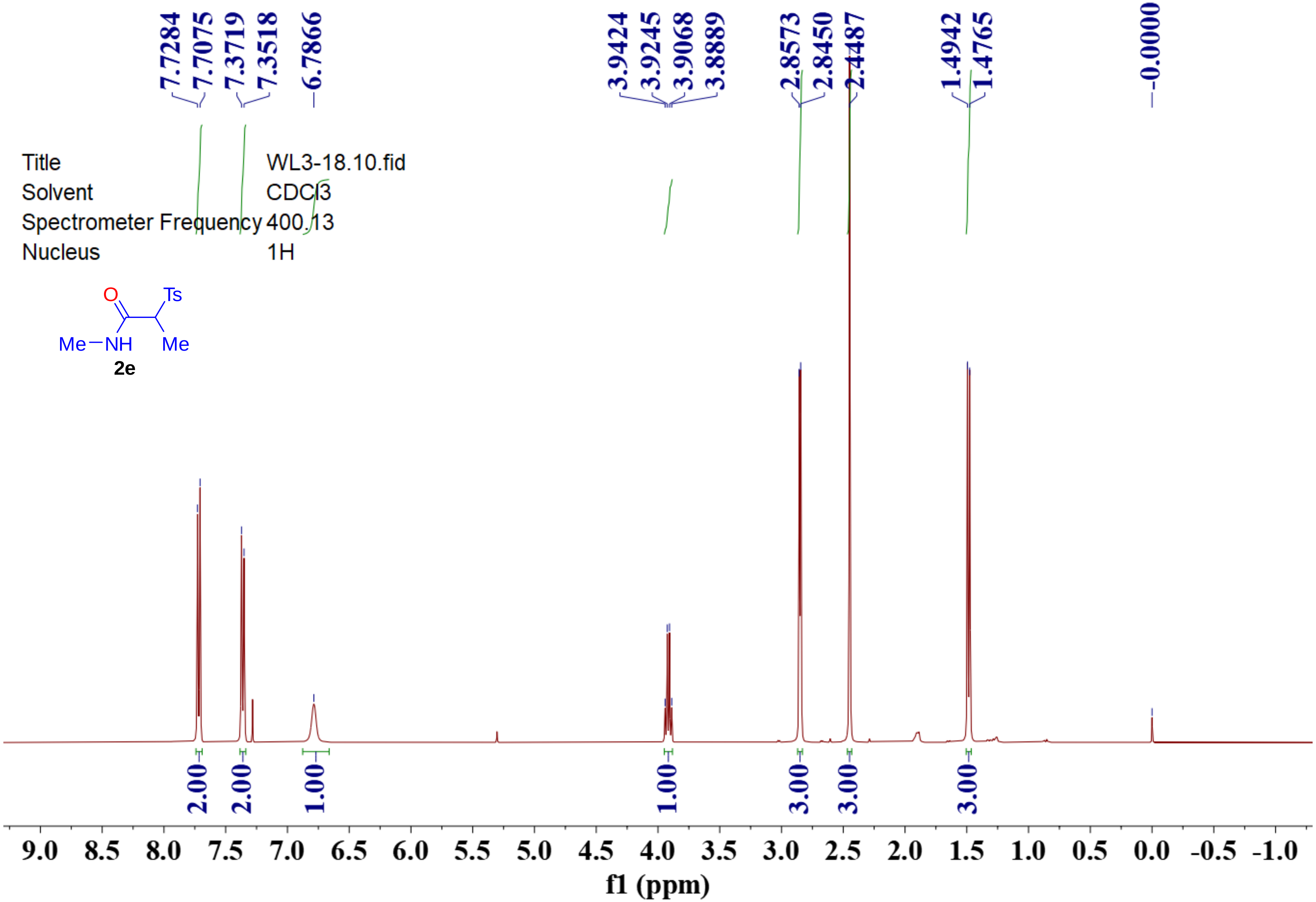
Title	WL2-44.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

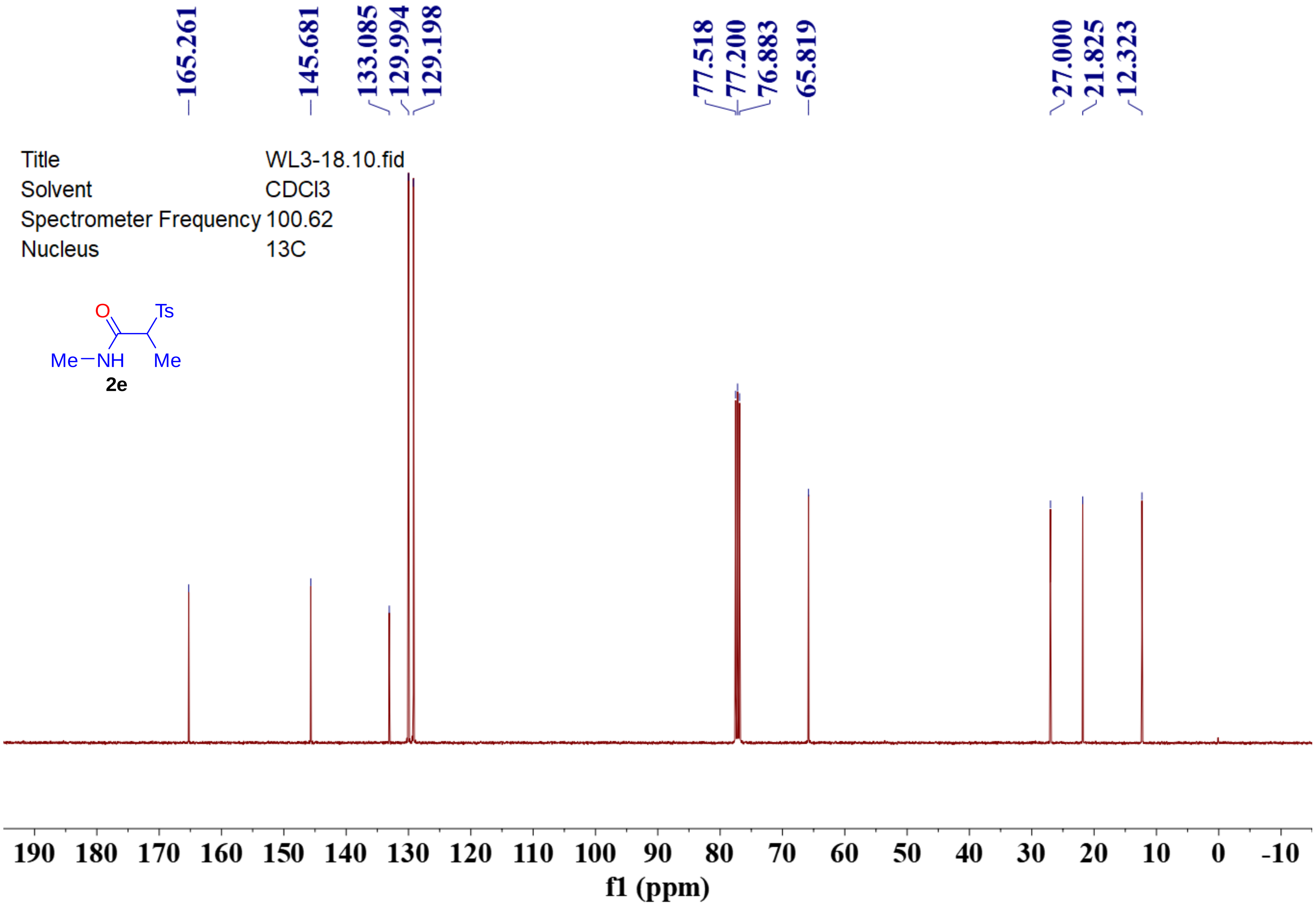

8.2593
8.2372
7.8307
7.8086
7.4182
7.4136
7.4093
7.4046
7.3964
7.3925
7.3868
7.3840
7.3806
7.3781
7.3750
7.3676
7.3639
7.3298
7.3235
7.3172
7.3106
7.3059
6.9185
6.9014
6.8882
4.4986
4.4834
4.4621
4.4470
4.4238
4.4092
4.3875
4.3727
4.0015
3.9837
3.9660
3.9482
1.5678
1.5500
-0.0000
Title WL2-52.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ns
Bn–NH
Me
2d
2.00
2.00
3.00
2.00
1.00
2.00
1.00
3.00
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
-0.5
-1.0
f1 (ppm)

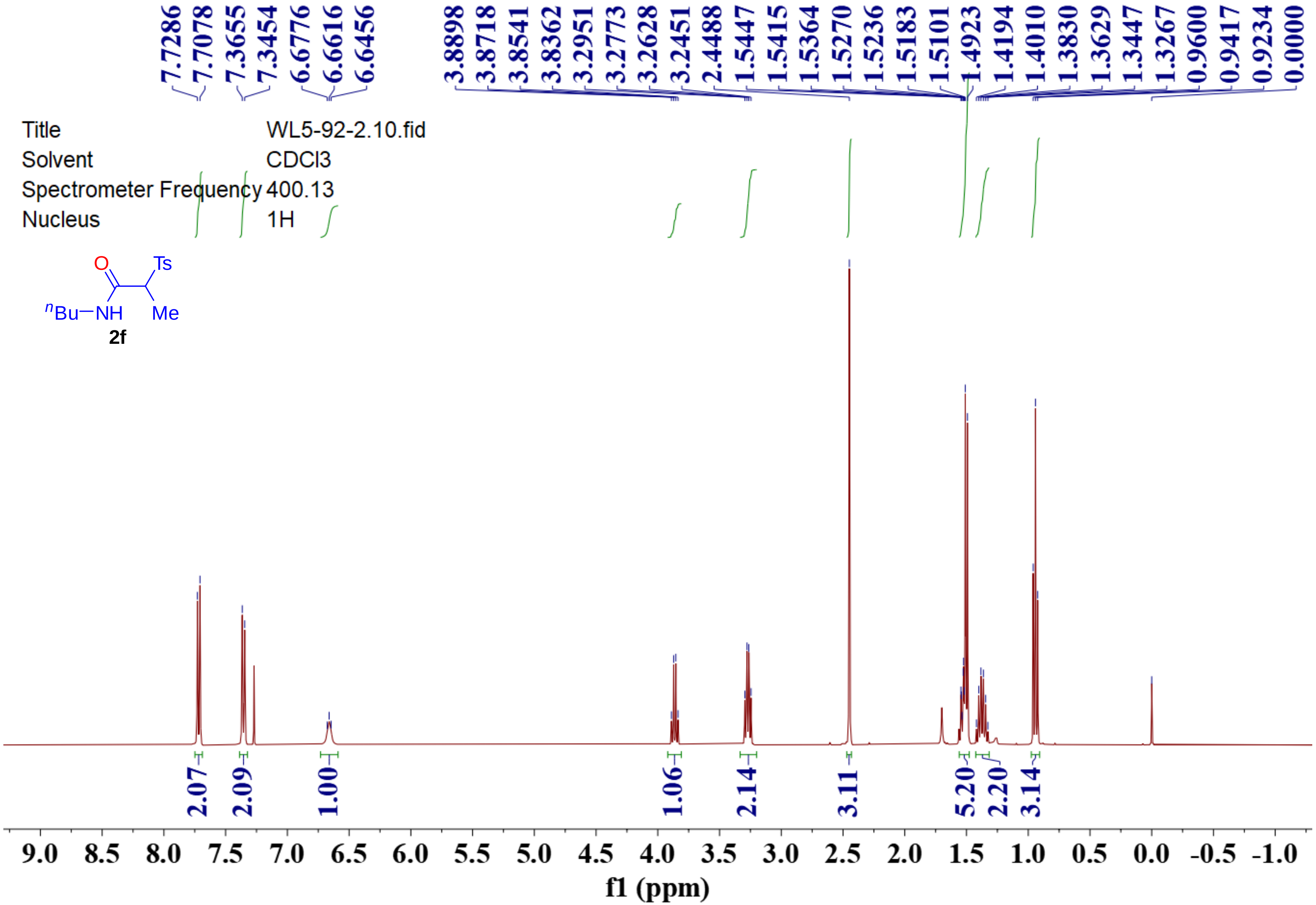


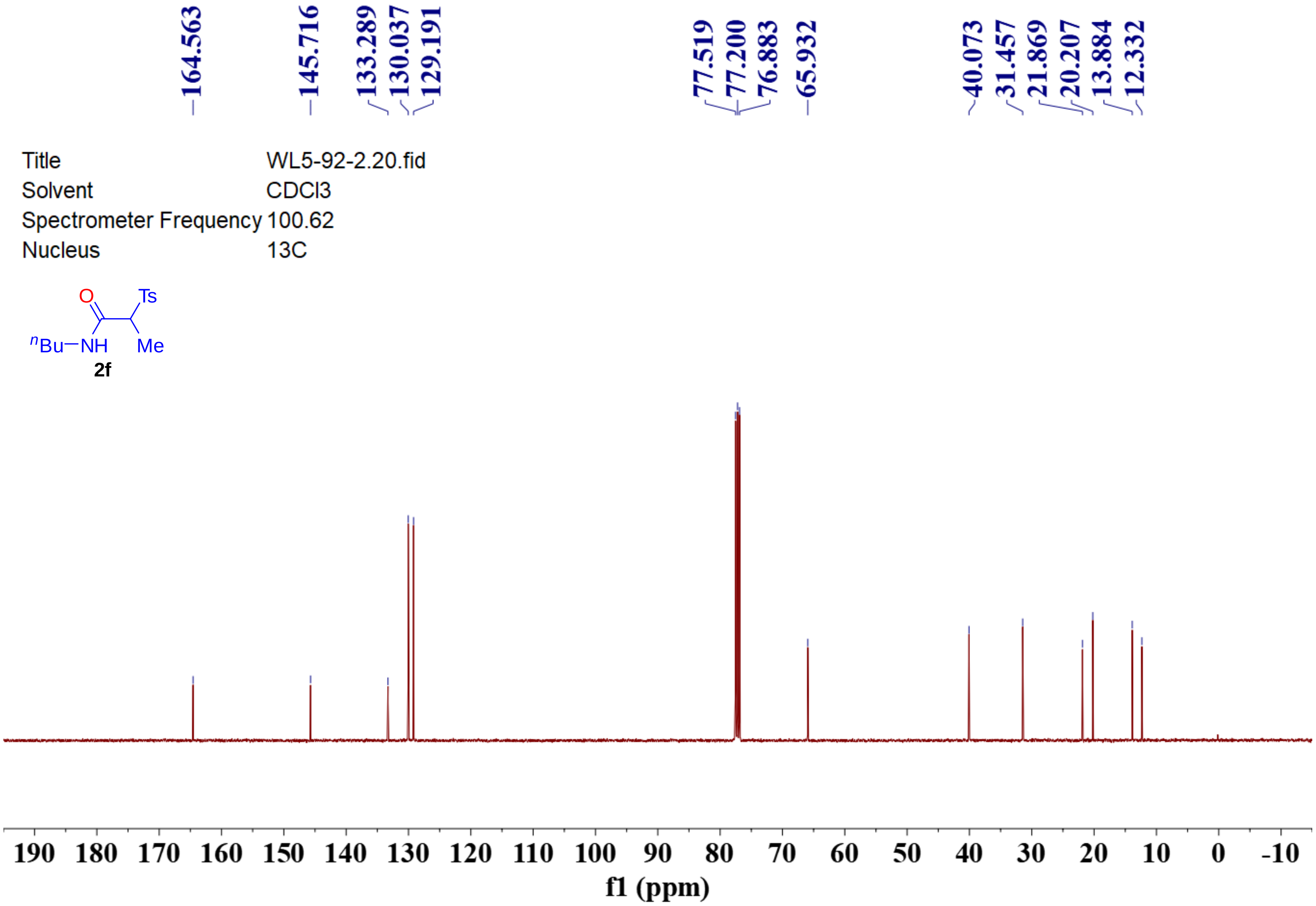
Title	WL2-52.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2d


7.7284
7.7075
7.3719
7.3518
6.7866
3.9424
3.9245
3.9068
3.8889
2.8573
2.8450
2.4487
1.4942
1.4765
-0.0000
Title
WL3-18.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
O
Ts
Me
NH
Me
2e
2.00
2.00
1.00
1.00
3.00
3.00
3.00
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
-0.5
-1.0
f1 (ppm)

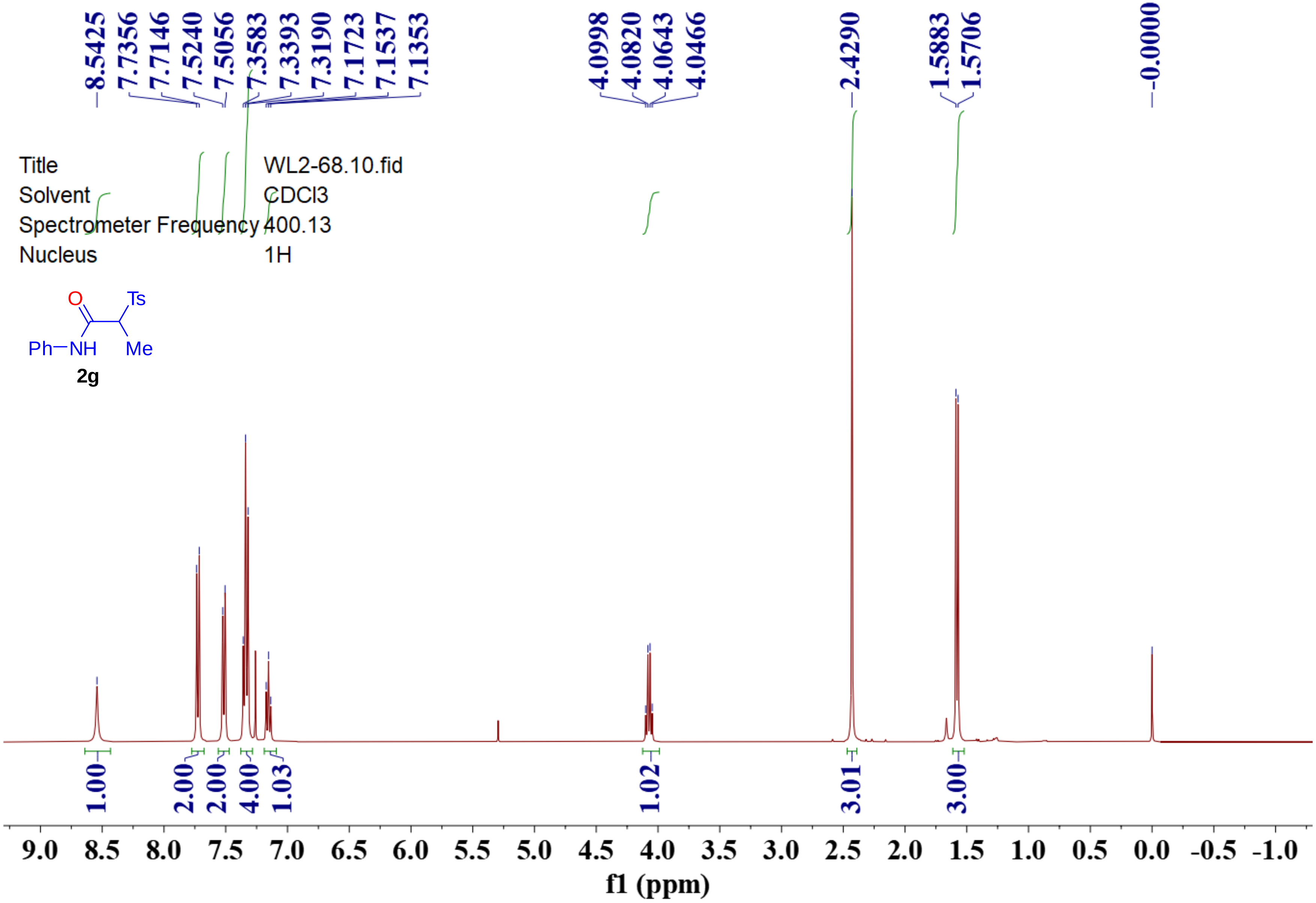

165.261
145.681
133.085
129.994
129.198
77.518
77.200
76.883
65.819
27.000
21.825
12.323
Title WL3-18.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Me
NH
Me
2e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

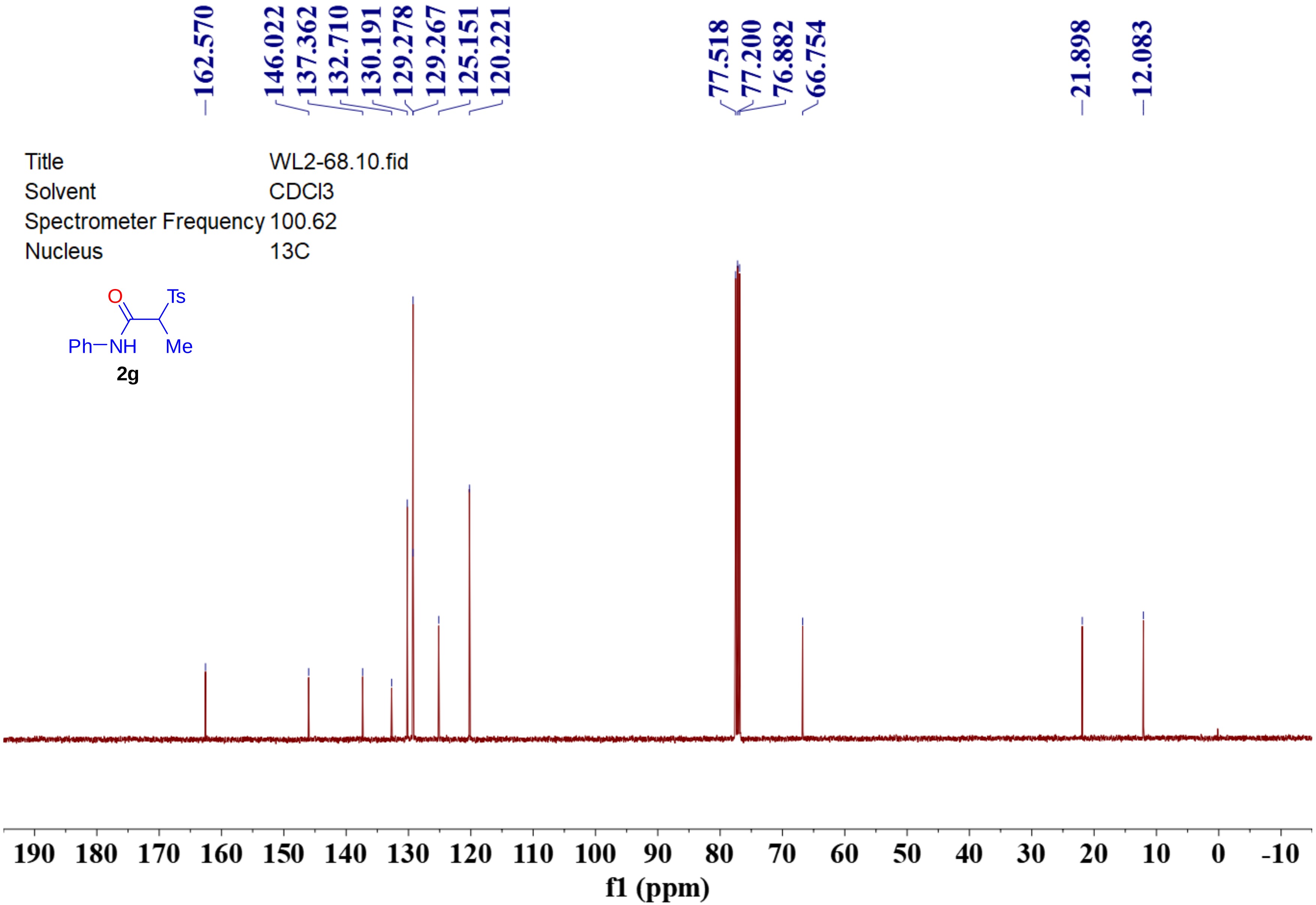

7.7286
7.7078
7.3655
7.3454
6.6776
6.6616
6.6456
3.8898
3.8718
3.8541
3.8362
3.2951
3.2773
3.2628
3.2451
2.4488
1.5447
1.5415
1.5364
1.5270
1.5236
1.5183
1.5101
1.4923
1.4194
1.4010
1.3830
1.3629
1.3447
1.3267
0.9600
0.9417
0.9234
0.0000
Title WL5-92-2.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
^{n}Bu–NH
Me
2f
2.07
2.09
1.00
1.06
2.14
3.11
5.20
2.20
3.14
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
-0.5
-1.0
f1 (ppm)



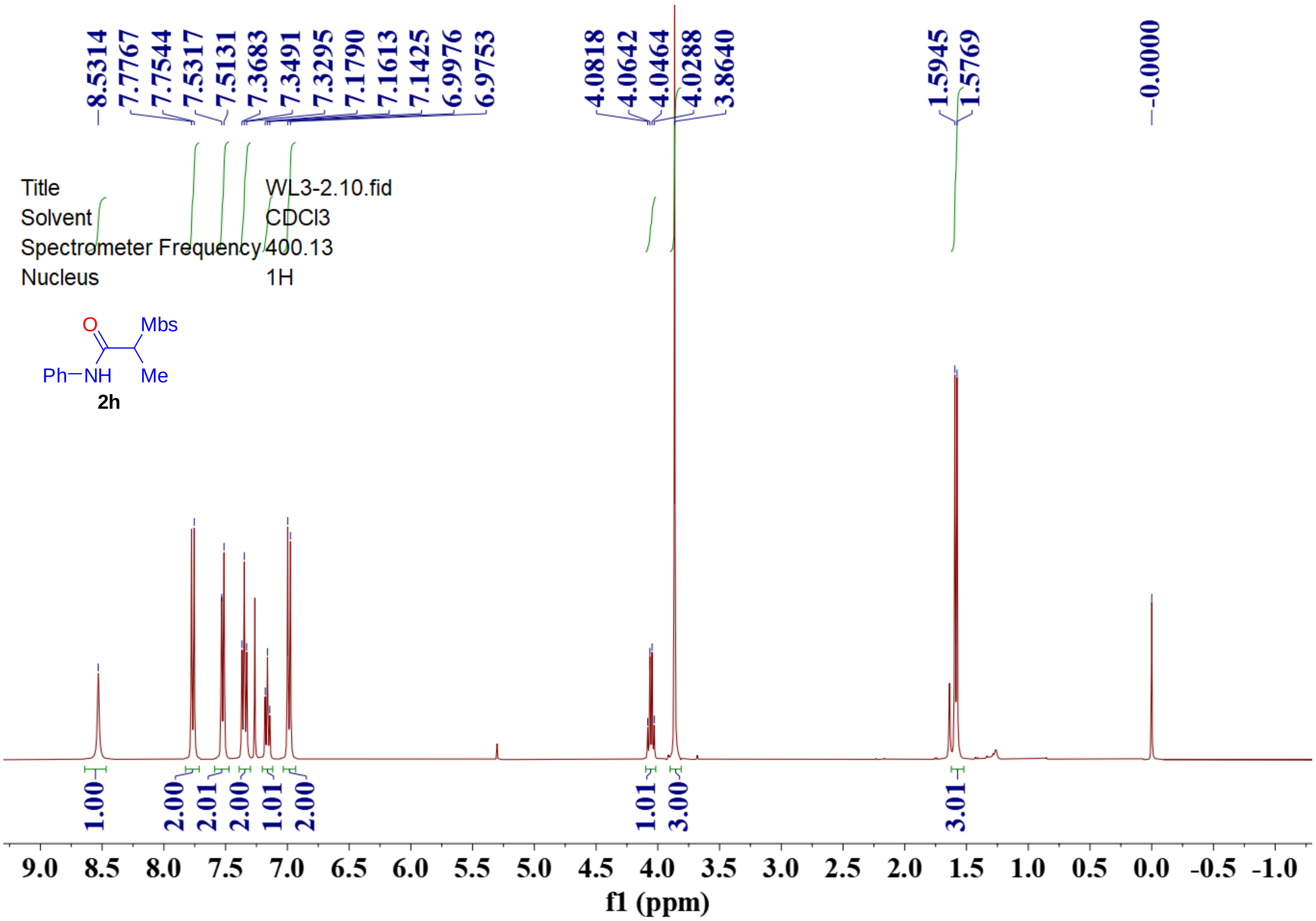
Title WL5-92-2.20.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

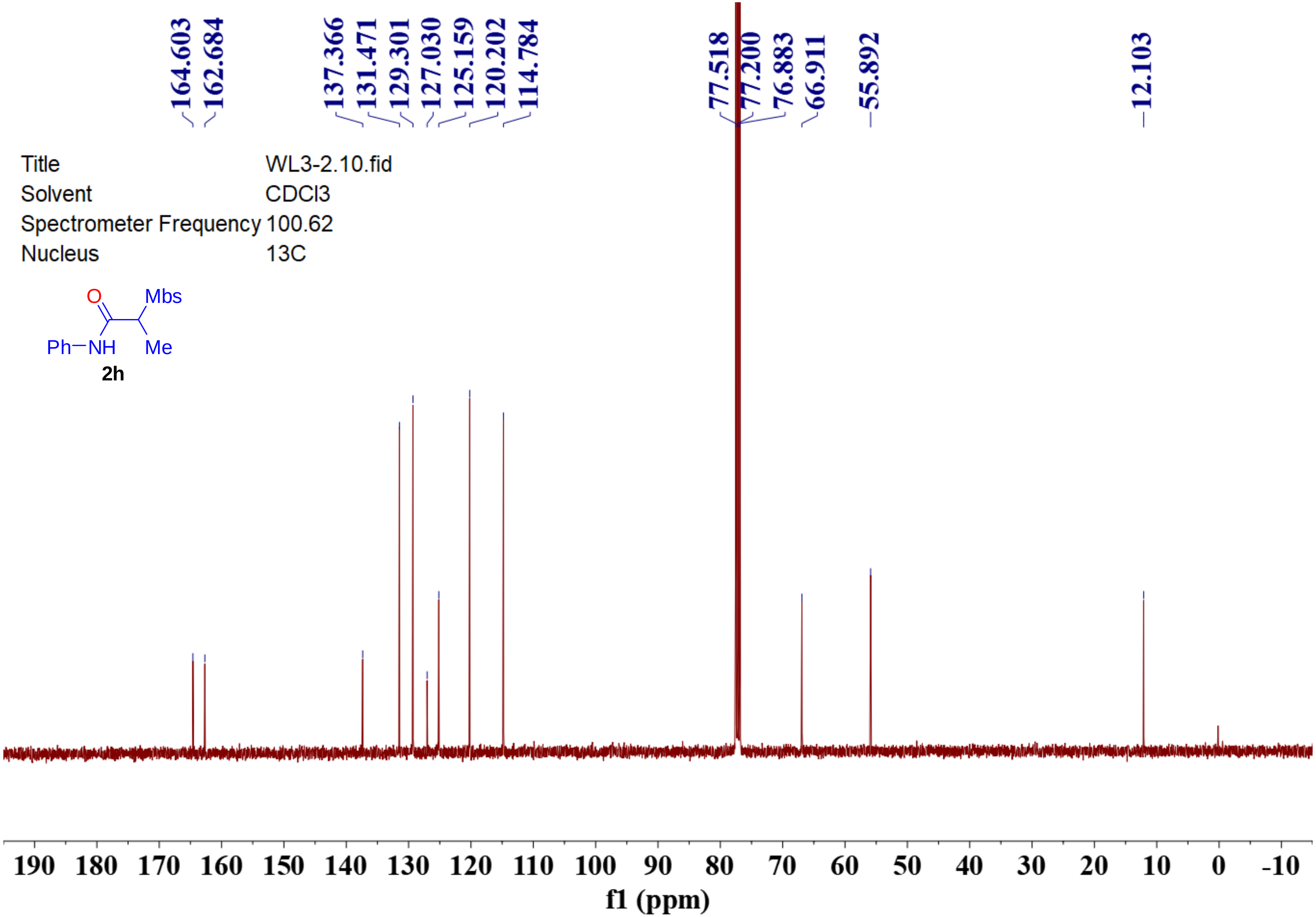
2f

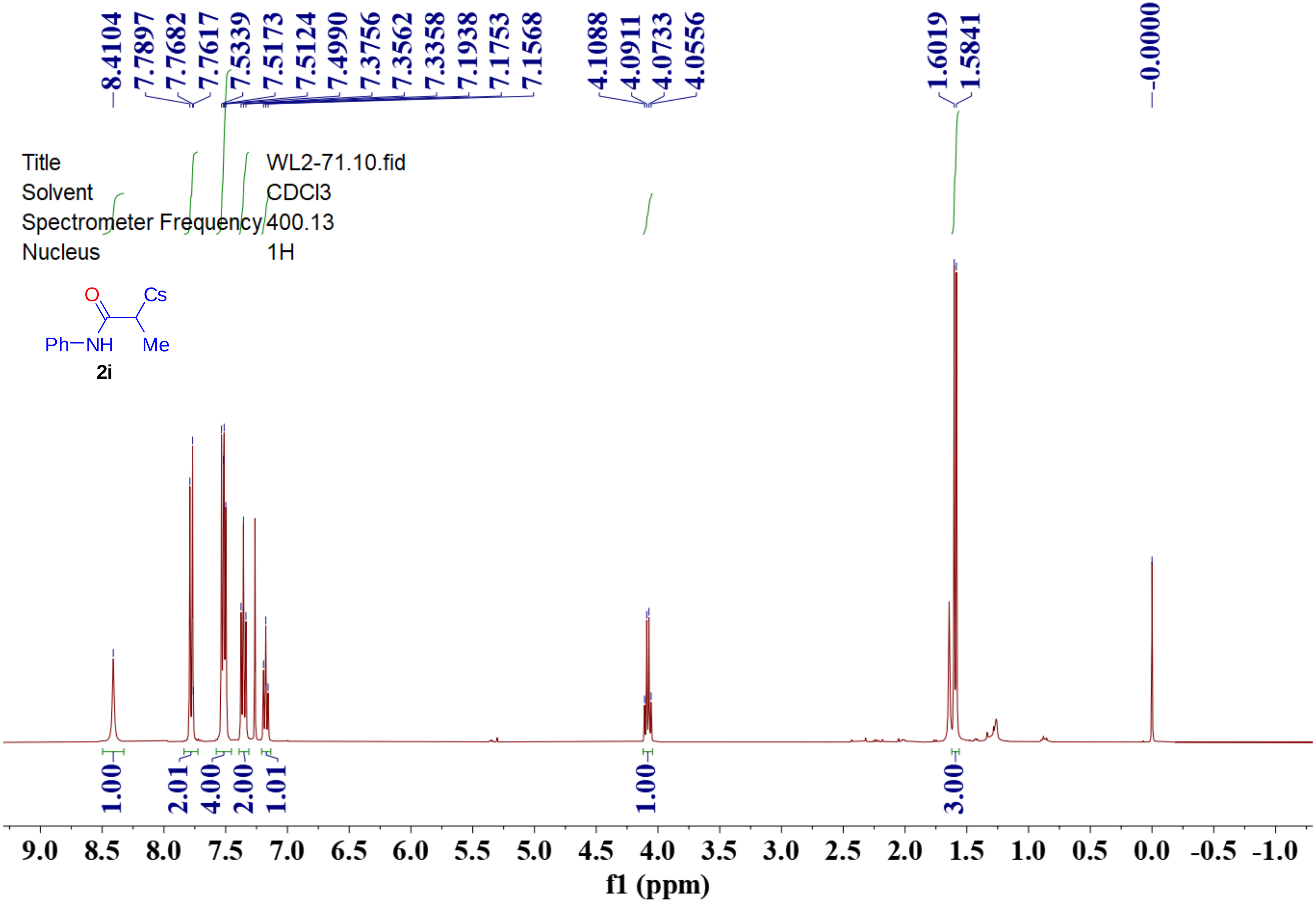

8.5425
7.7356
7.7146
7.5240
7.5056
7.3583
7.3393
7.3190
7.1723
7.1537
7.1353
4.0998
4.0820
4.0643
4.0466
2.4290
1.5883
1.5706
-0.0000
Title WL2-68.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Ph
NH
Me
2g
1.00
2.00
2.00
4.00
1.03
1.02
3.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



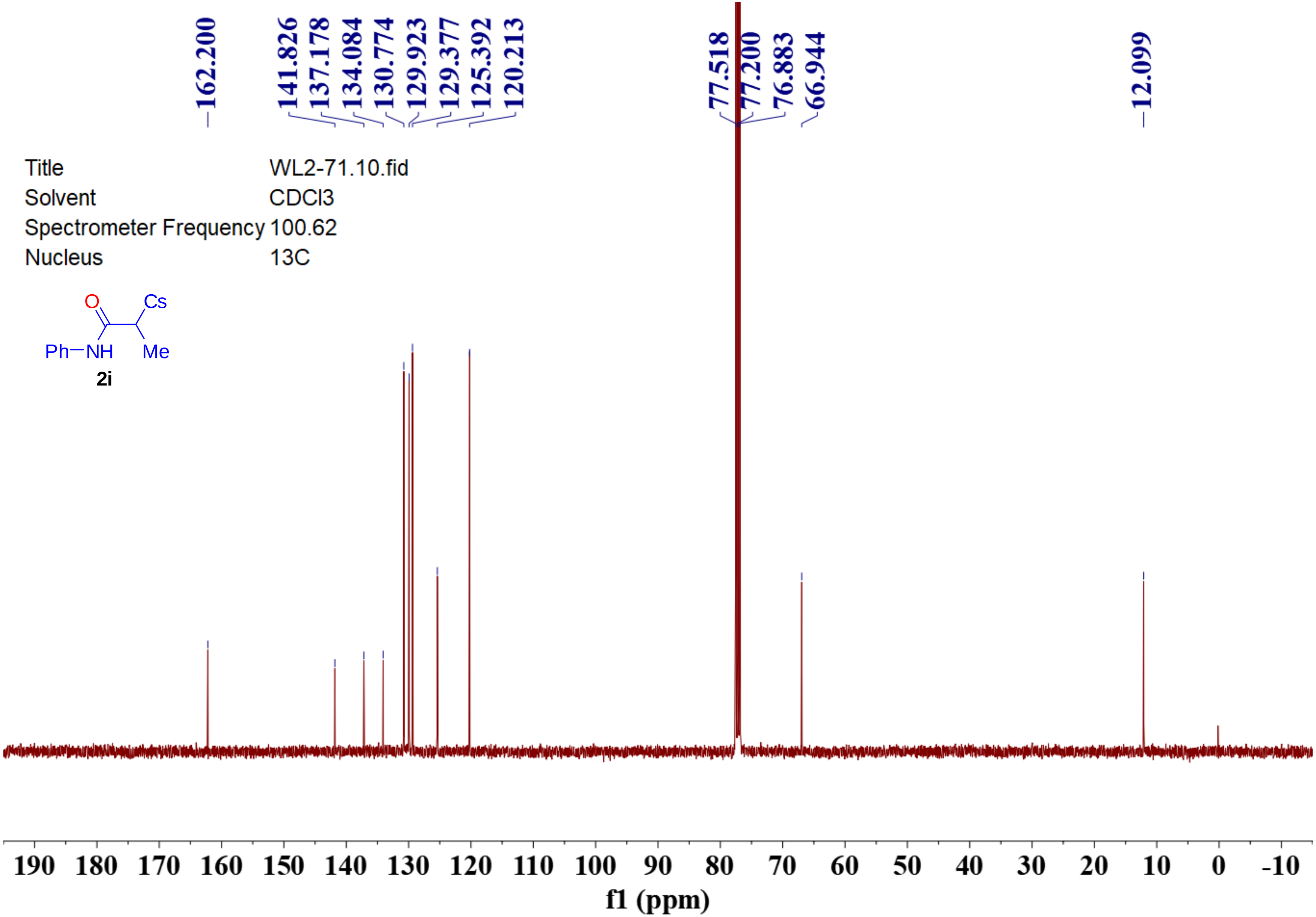
162.570
146.022
137.362
132.710
130.191
129.278
129.267
125.151
120.221
77.518
77.200
76.882
66.754
21.898
12.083
Title WL2-68.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Ph
NH
Me
2g
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.5314
7.7767
7.7544
7.5317
7.5131
7.3683
7.3491
7.3295
7.1790
7.1613
7.1425
6.9976
6.9753
4.0818
4.0642
4.0464
4.0288
3.8640
1.5945
1.5769
-0.0000
Title WL3-2.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Mbs
Ph
NH
Me
2h
1.00
2.00
2.01
2.00
1.01
2.00
1.01
3.00
3.01
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 -0.5 -1.0
f1 (ppm)


164.603
162.684
137.366
131.471
129.301
127.030
125.159
120.202
114.784
77.518
77.200
76.883
66.911
55.892
12.103
Title WL3-2.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Mbs
Ph−NH
Me
2h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



8.4104
7.7897
7.7682
7.7617
7.5339
7.5173
7.5124
7.4990
7.3756
7.3562
7.3358
7.1938
7.1753
7.1568
4.1088
4.0911
4.0733
4.0556
1.6019
1.5841
-0.0000
Title WL2-71.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Cs
Ph−NH
Me
2i
1.00
2.01
4.00
2.00
1.01
1.00
3.00
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 -0.5 -1.0
f1 (ppm)

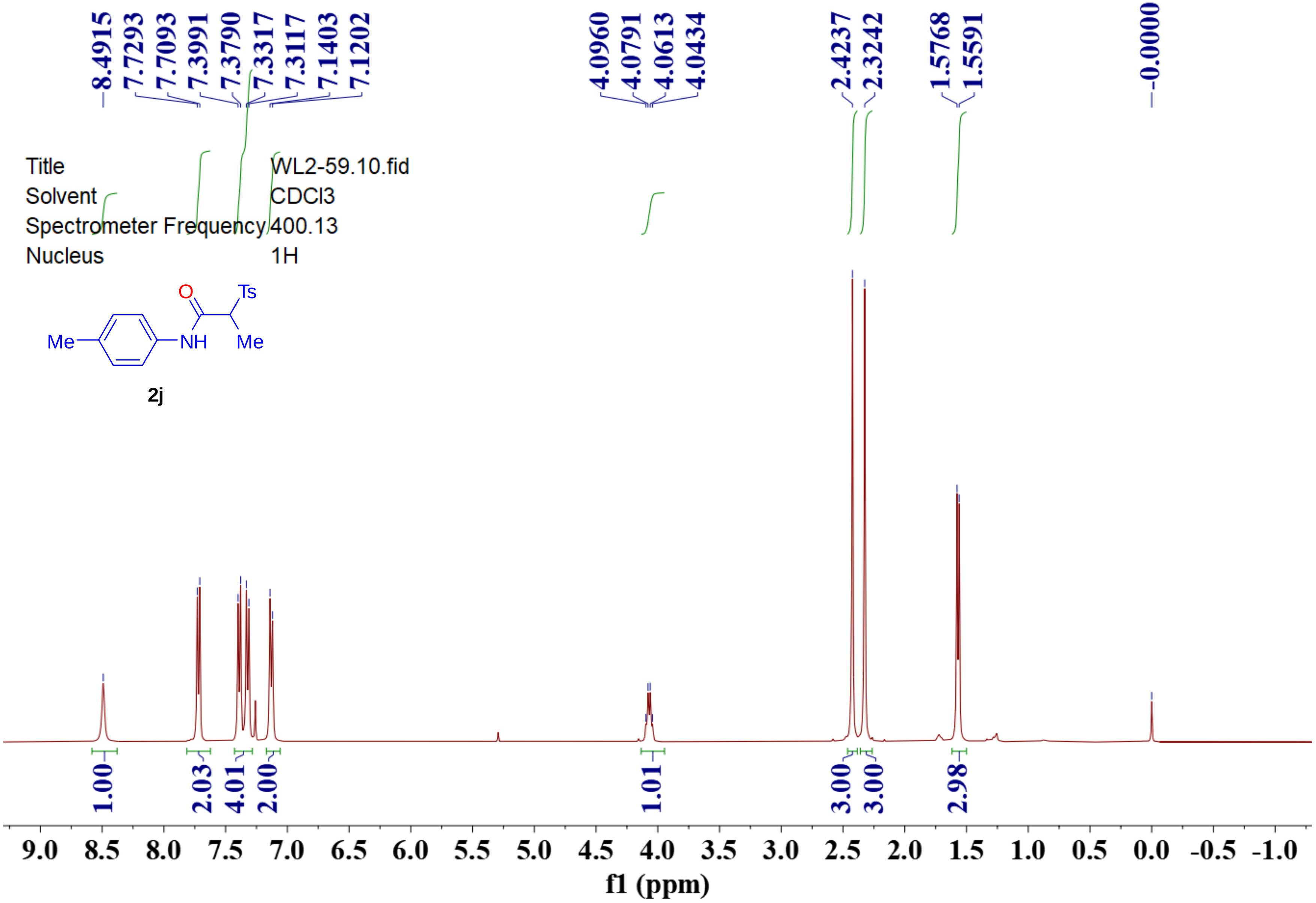


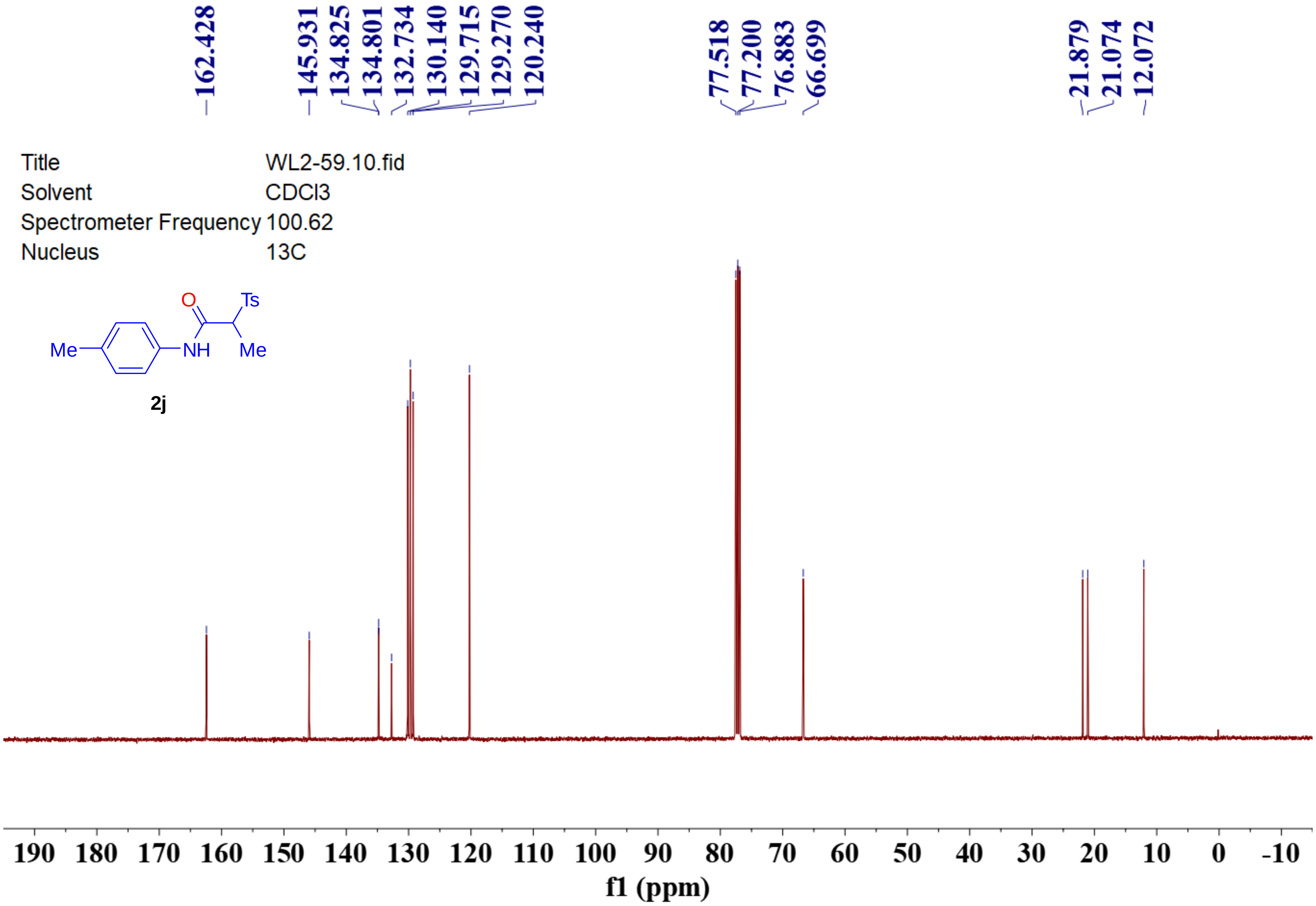
Title WL2-71.10.fid

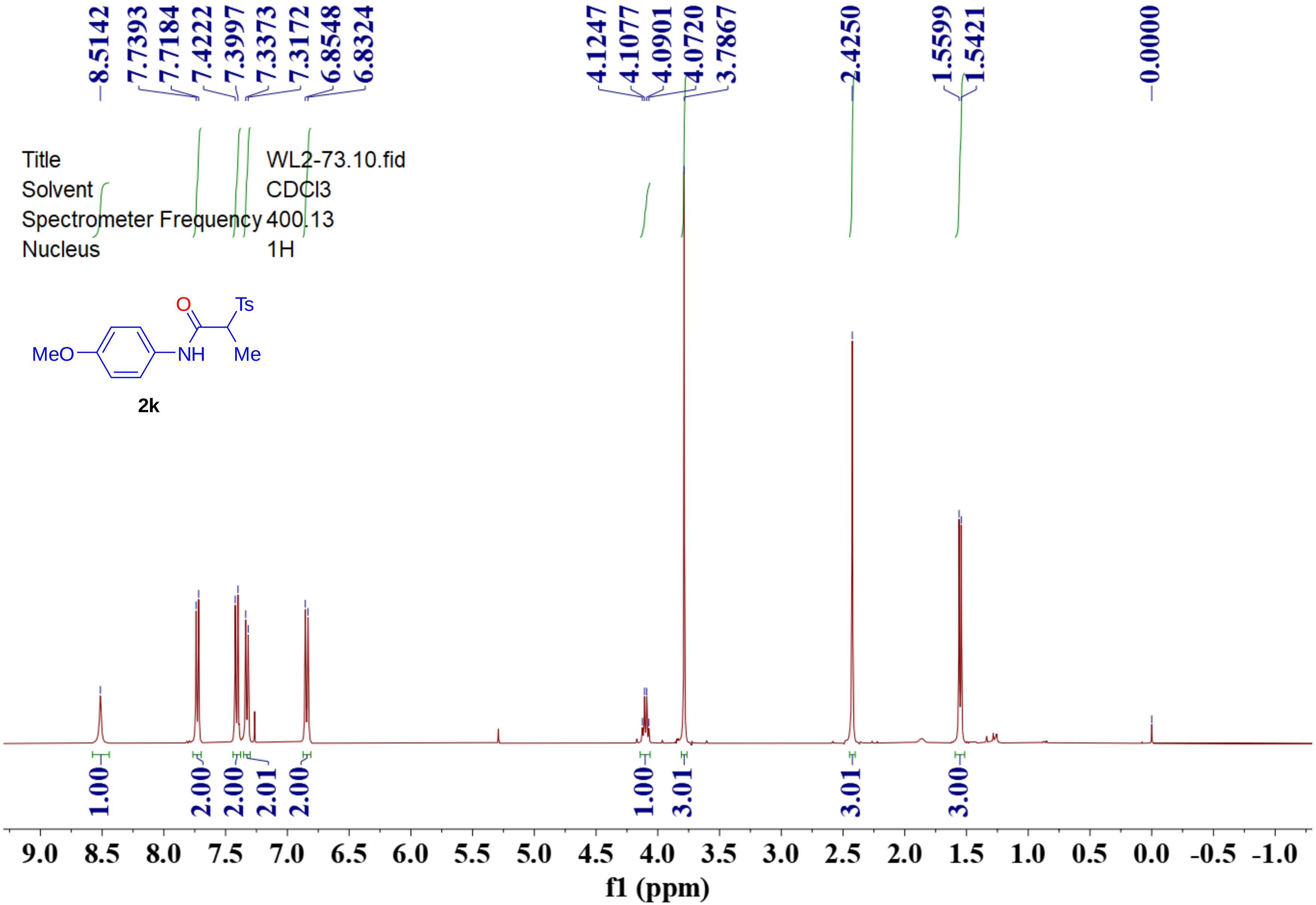
Solvent CDCl3

Spectrometer Frequency 100.62

Nucleus 13C

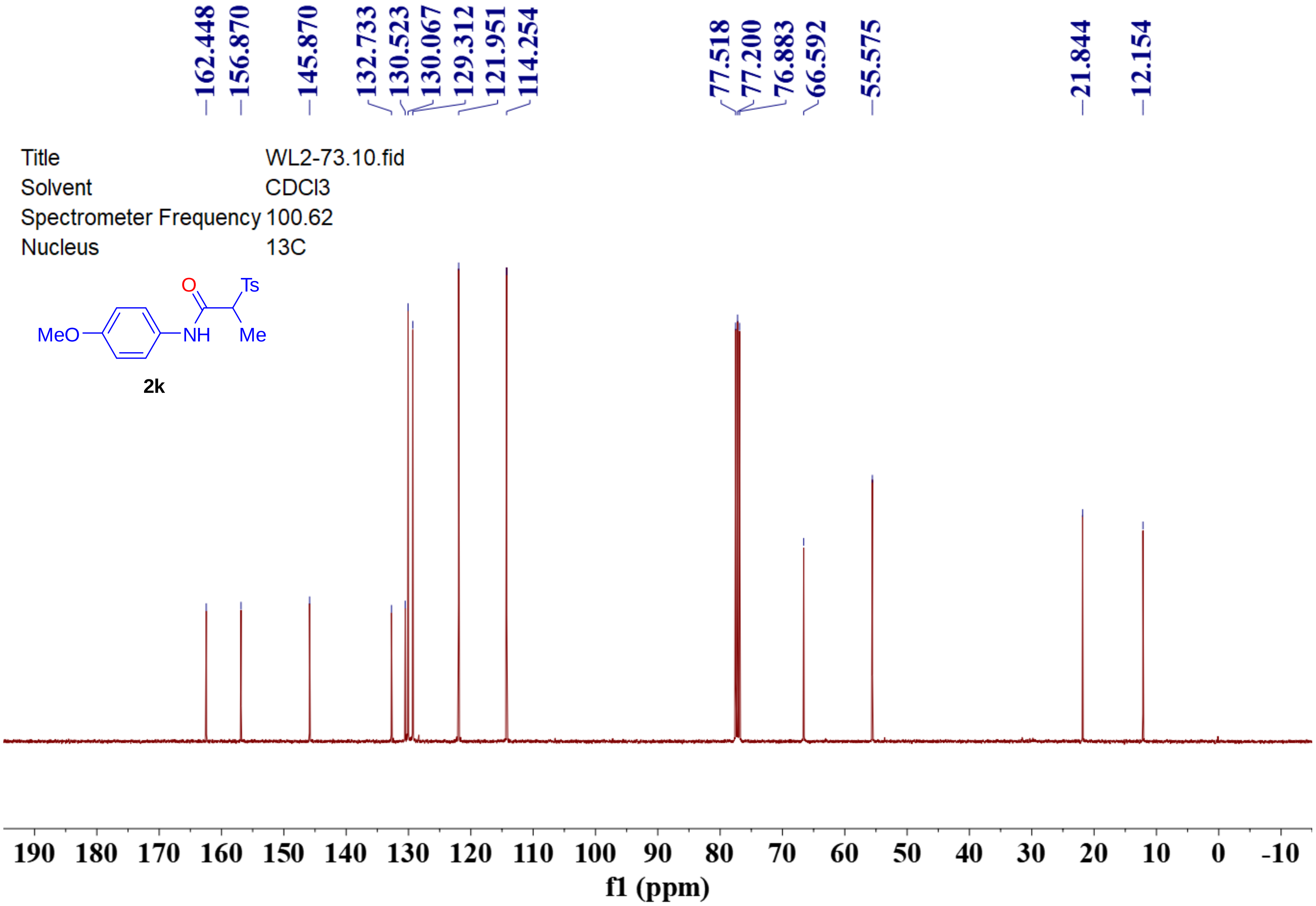

8.4915
7.7293
7.7093
7.3991
7.3790
7.3317
7.3117
7.1403
7.1202
4.0960
4.0791
4.0613
4.0434
2.4237
2.3242
1.5768
1.5591
-0.0000
Title WL2-59.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Me
NH
Me
2j
1.00
2.03
4.01
2.00
1.01
3.00
3.00
2.98
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


162.428
145.931
134.825
134.801
132.734
130.140
129.715
129.270
120.240
77.518
77.200
76.883
66.699
21.879
21.074
12.072
Title WL2-59.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Me
NH
Me
2j
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

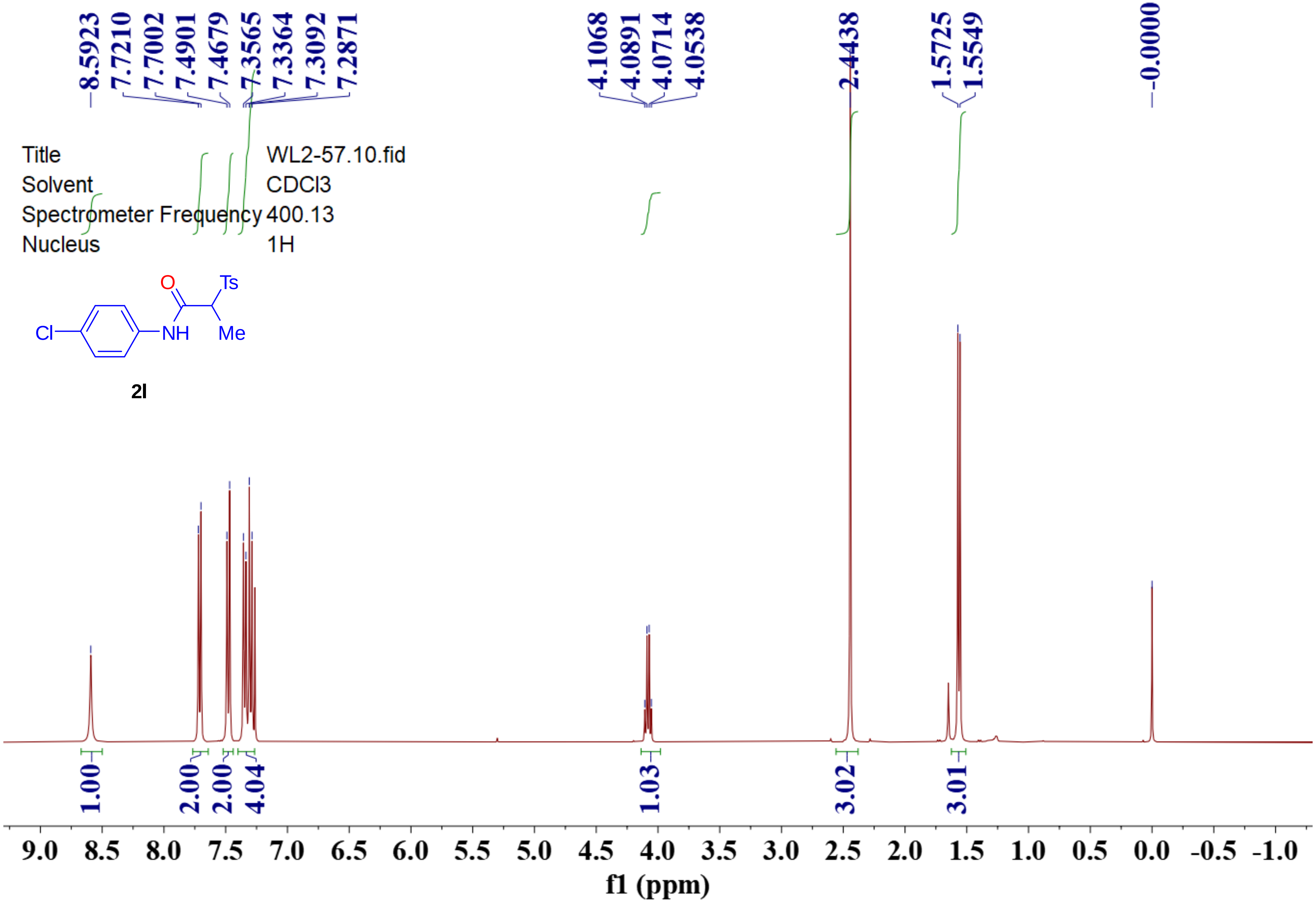


8.5142
7.7393
7.7184
7.4222
7.3997
7.3373
7.3172
6.8548
6.8324
4.1247
4.1077
4.0901
4.0720
3.7867
2.4250
1.5599
1.5421
0.0000
Title WL2-73.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
MeO
NH
O
Ts
Me
1.00
2.00
2.00
2.01
2.00
1.00
3.01
3.01
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

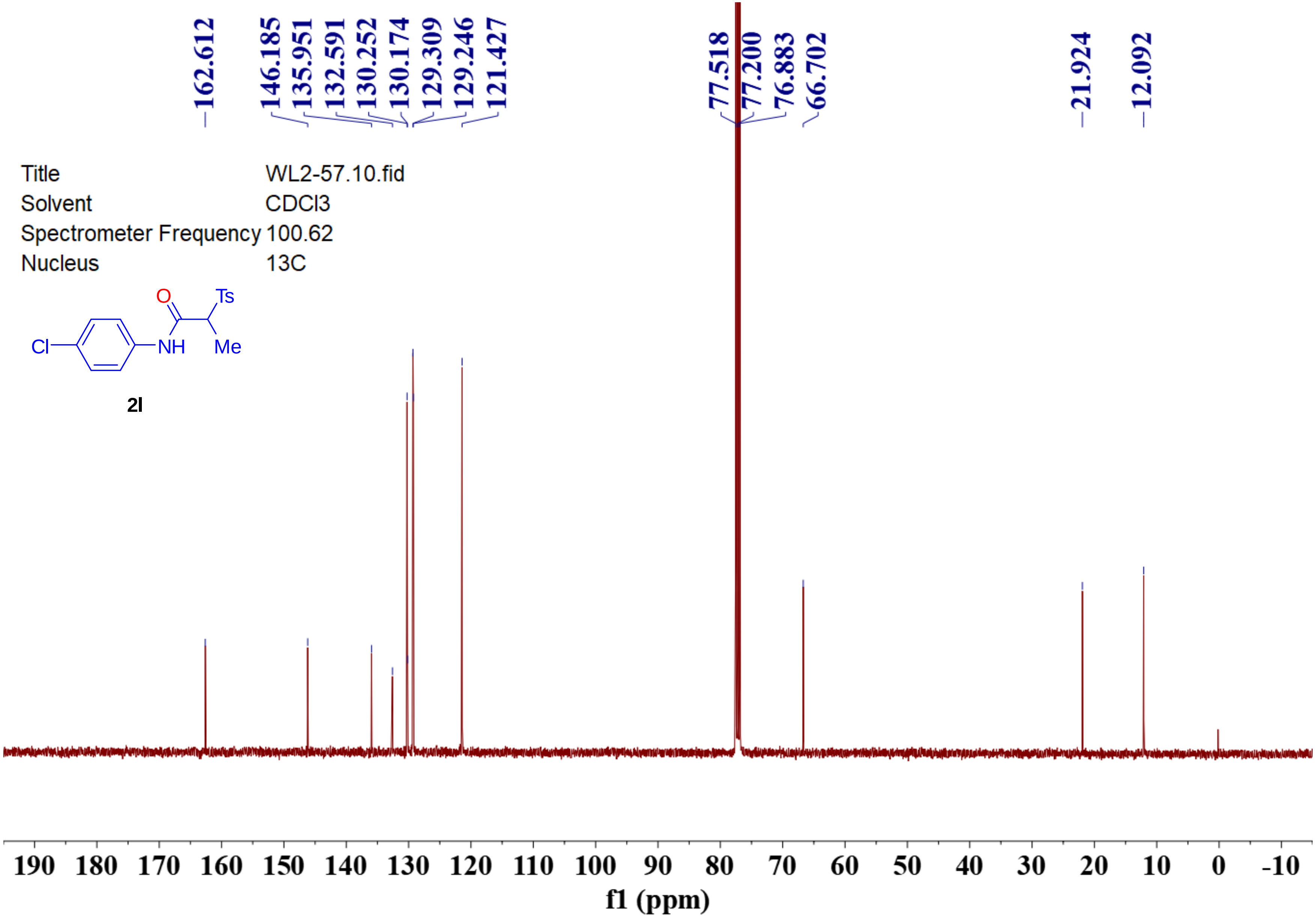
2k

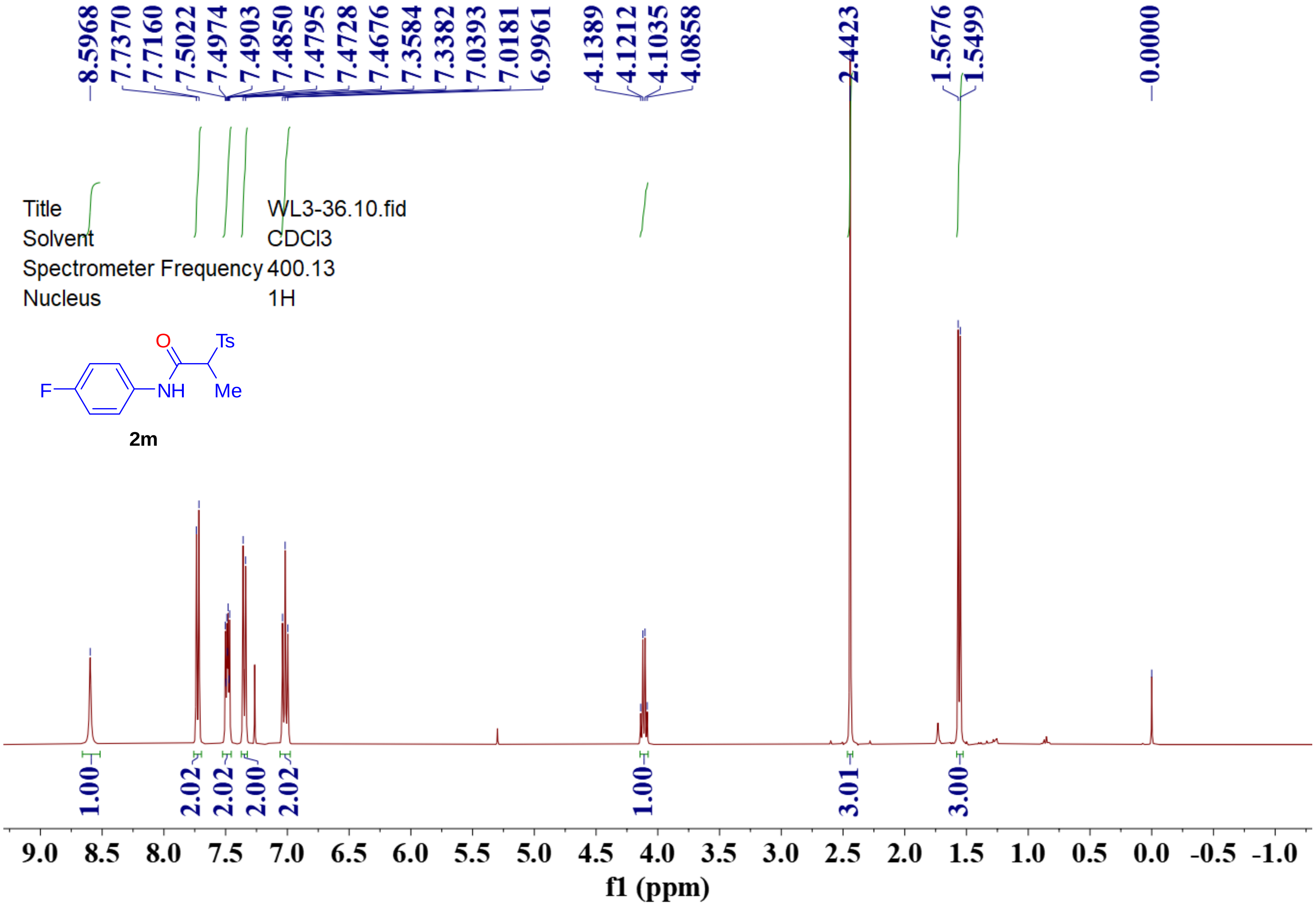


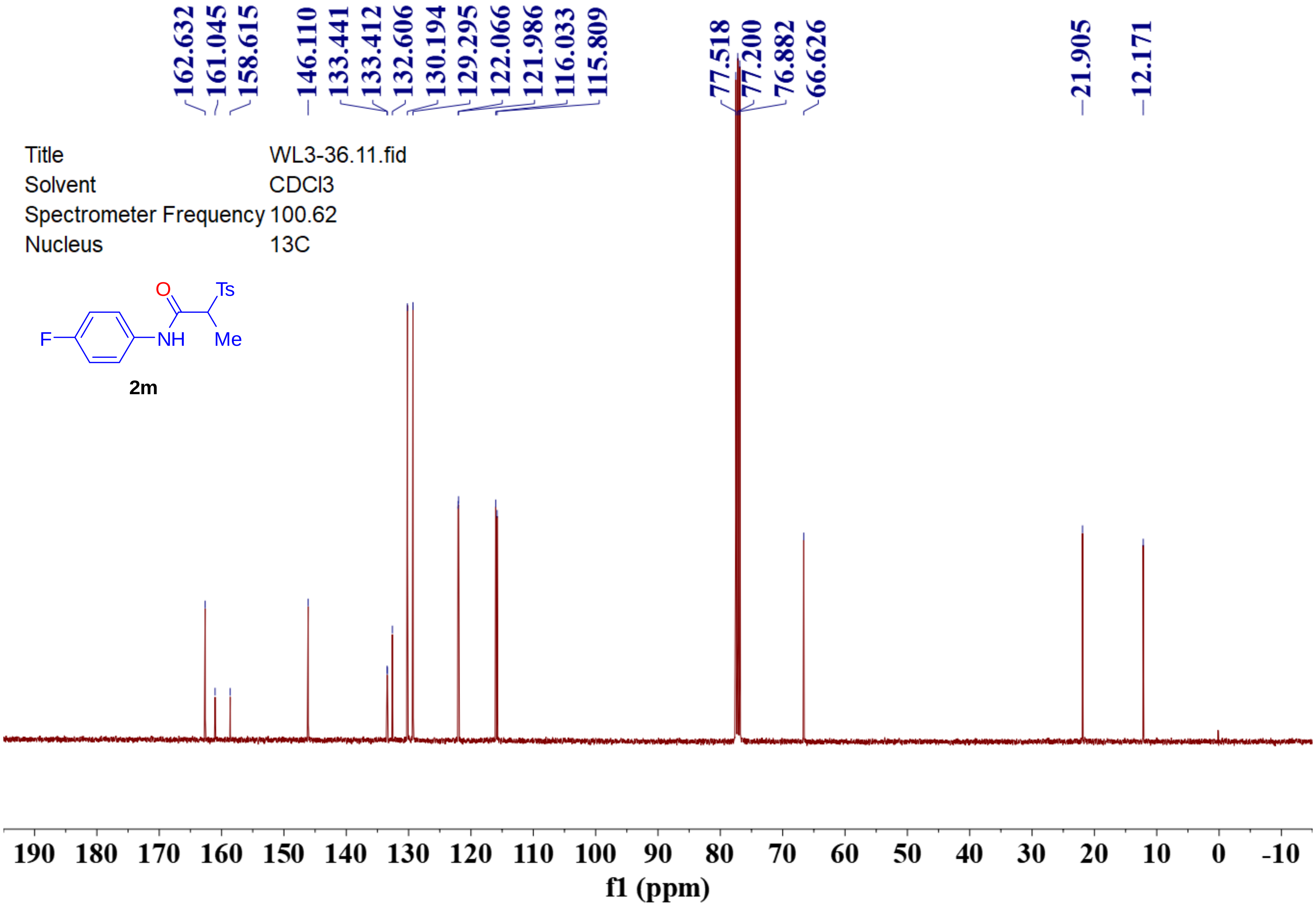
162.448
156.870
145.870
132.733
130.523
130.067
129.312
121.951
114.254
77.518
77.200
76.883
66.592
55.575
21.844
12.154
Title WL2-73.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
MeO
NH
Me
2k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.5923
7.7210
7.7002
7.4901
7.4679
7.3565
7.3364
7.3092
7.2871
4.1068
4.0891
4.0714
4.0538
2.4438
1.5725
1.5549
-0.0000
Title WL2-57.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Cl
NH
Me
1.00
2.00
2.00
4.04
1.03
3.02
3.01
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 -0.5 -1.0
f1 (ppm)

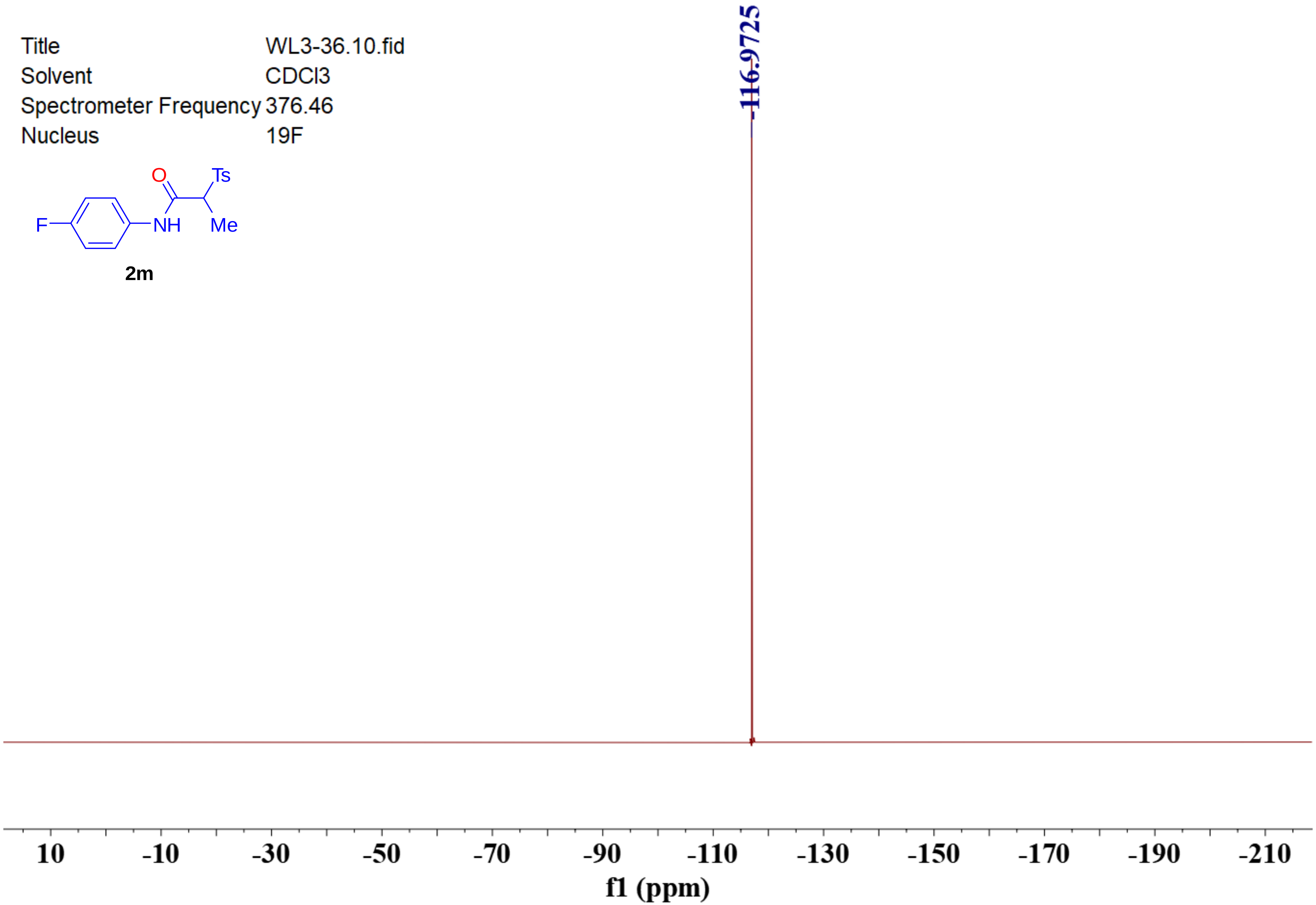
2l


162.612
146.185
135.951
132.591
130.252
130.174
129.309
129.246
121.427
77.518
77.200
76.883
66.702
21.924
12.092
Title WL2-57.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Cl
NH
Me
2l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

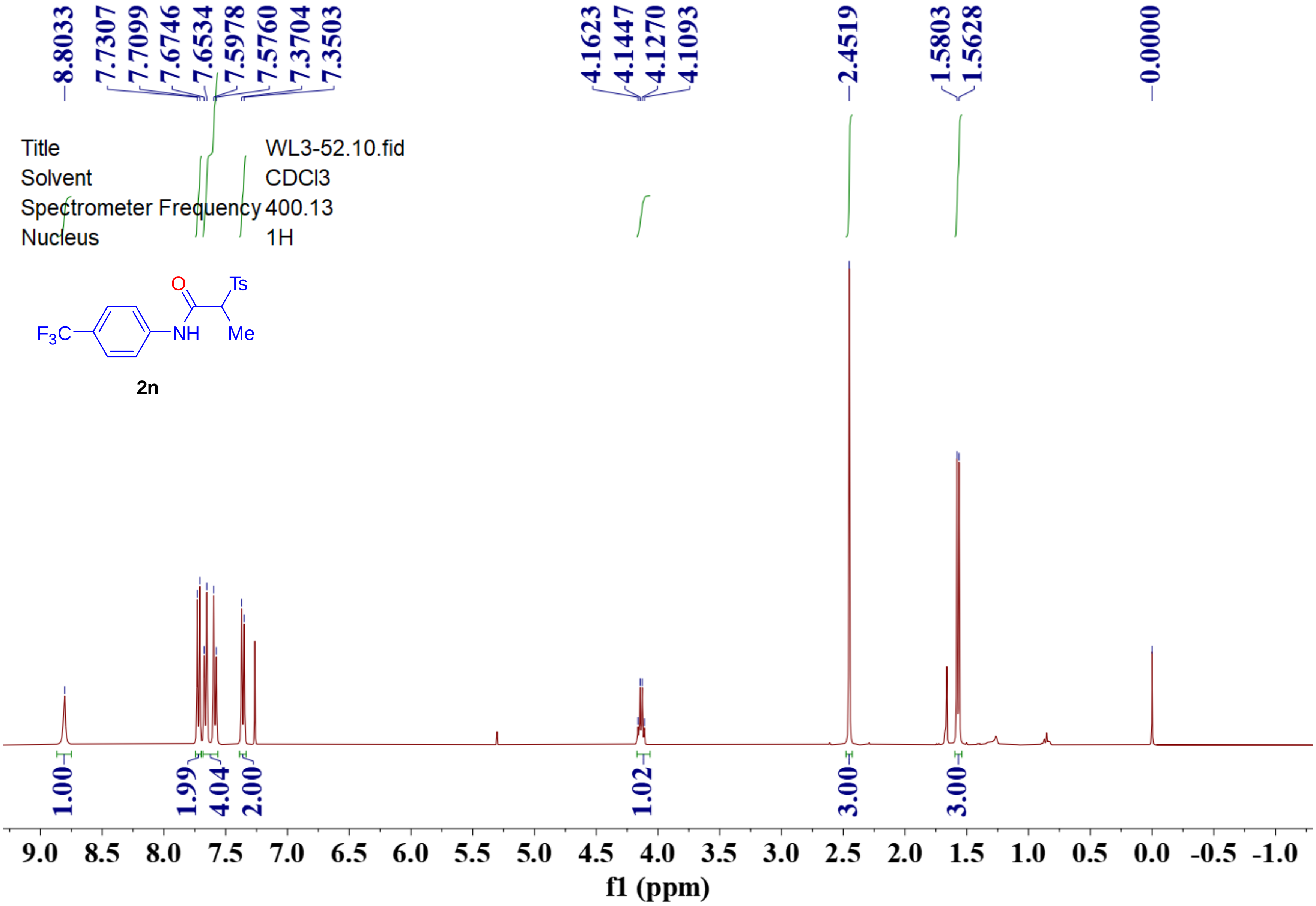

8.5968
7.7370
7.7160
7.5022
7.4974
7.4903
7.4850
7.4795
7.4728
7.4676
7.3584
7.3382
7.0393
7.0181
6.9961
4.1389
4.1212
4.1035
4.0858
2.4423
1.5676
1.5499
-0.0000
Title WL3-36.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
F
NH
Me
2m
1.00
2.02
2.02
2.00
2.02
1.00
3.01
3.00
9.0
8.5
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
-0.5
-1.0
f1 (ppm)


162.632
161.045
158.615
146.110
133.441
133.412
132.606
130.194
129.295
122.066
121.986
116.033
115.809
77.518
77.200
76.882
66.626
21.905
12.171
Title WL3-36.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
F
NH
Me
2m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

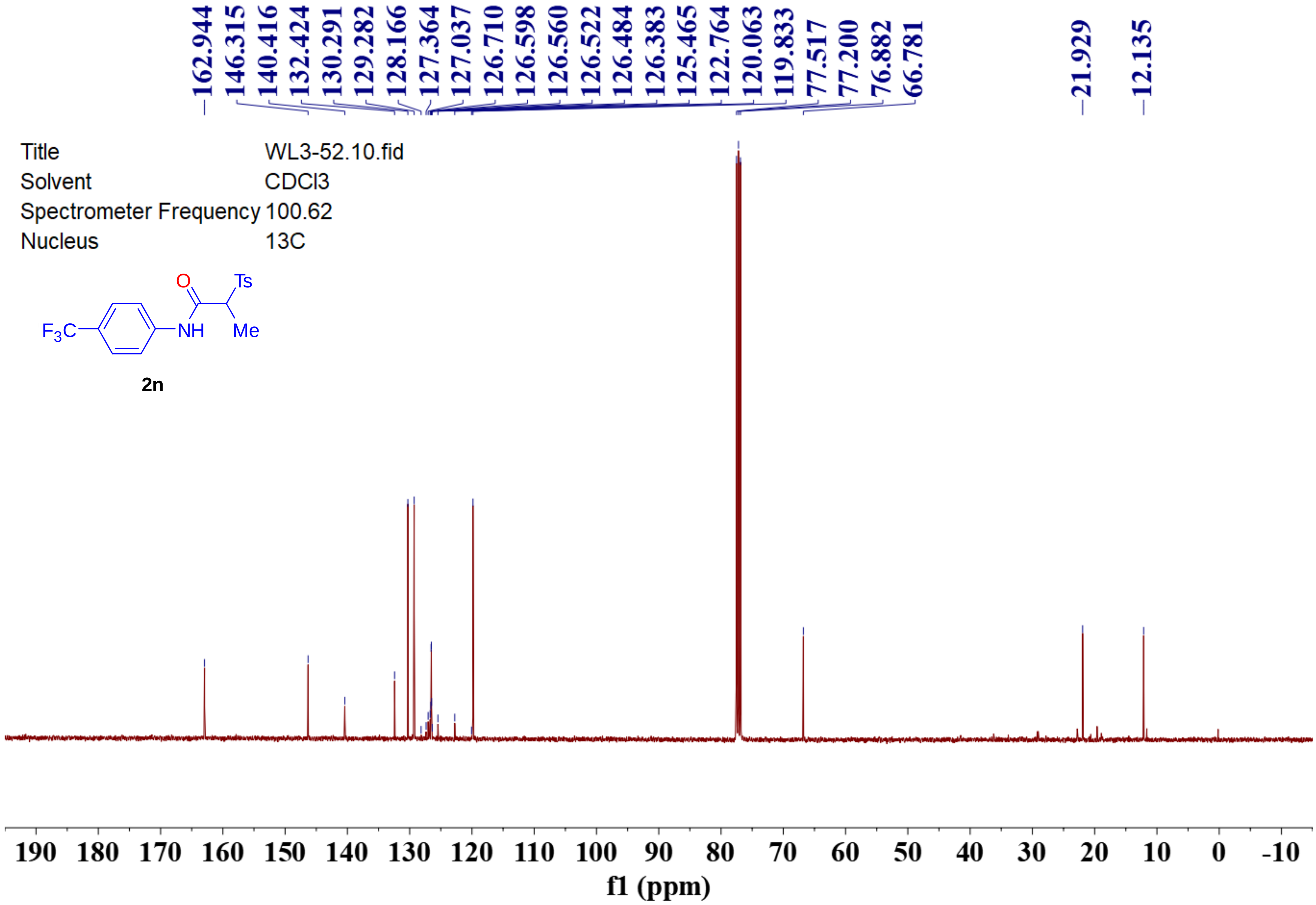
Title	WL3-36.10.fid
Solvent	CDCl3
Spectrometer Frequency	376.46
Nucleus	19F



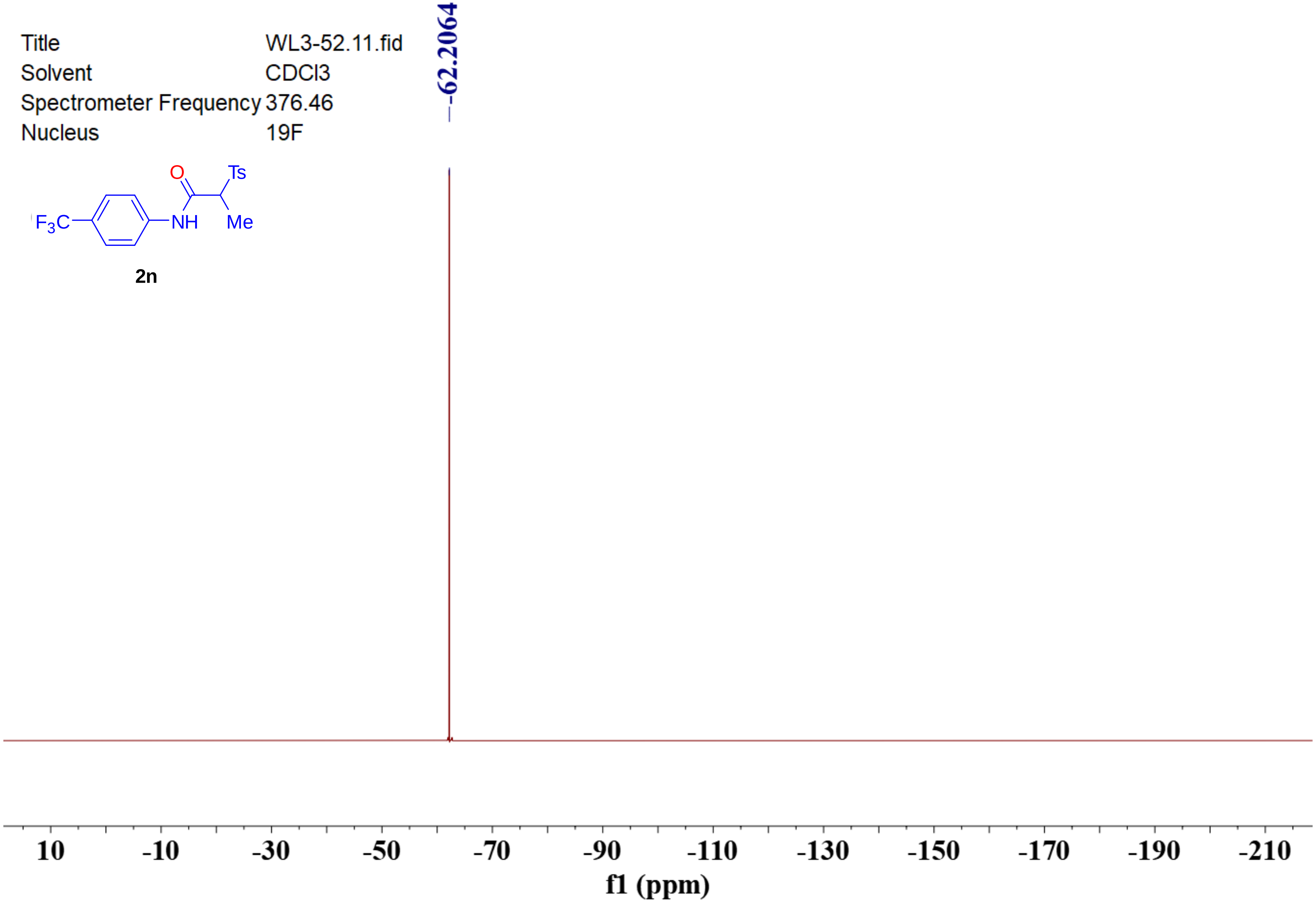
2m



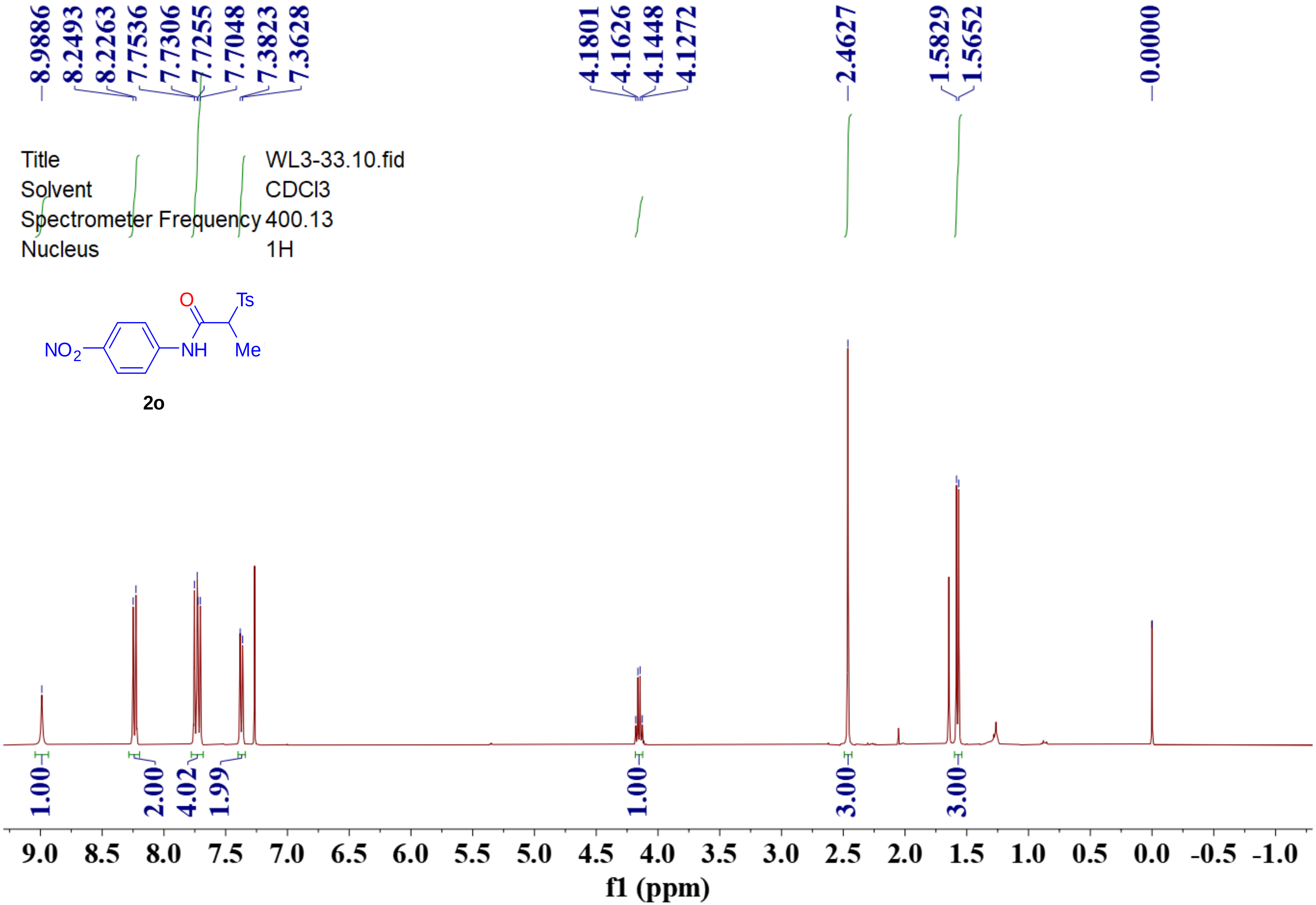
8.8033
7.7307
7.7099
7.6746
7.6534
7.5978
7.5760
7.3704
7.3503
4.1623
4.1447
4.1270
4.1093
2.4519
1.5803
1.5628
0.0000
Title WL3-52.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
F_3C
NH
Me
2n
1.00
1.99
4.04
2.00
1.02
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


162.944
146.315
140.416
132.424
130.291
129.282
128.166
127.364
127.037
126.710
126.598
126.560
126.522
126.484
126.383
125.465
122.764
120.063
119.833
77.517
77.200
76.882
66.781
21.929
12.135
Title WL3-52.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
F3C
NH
Me
2n
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

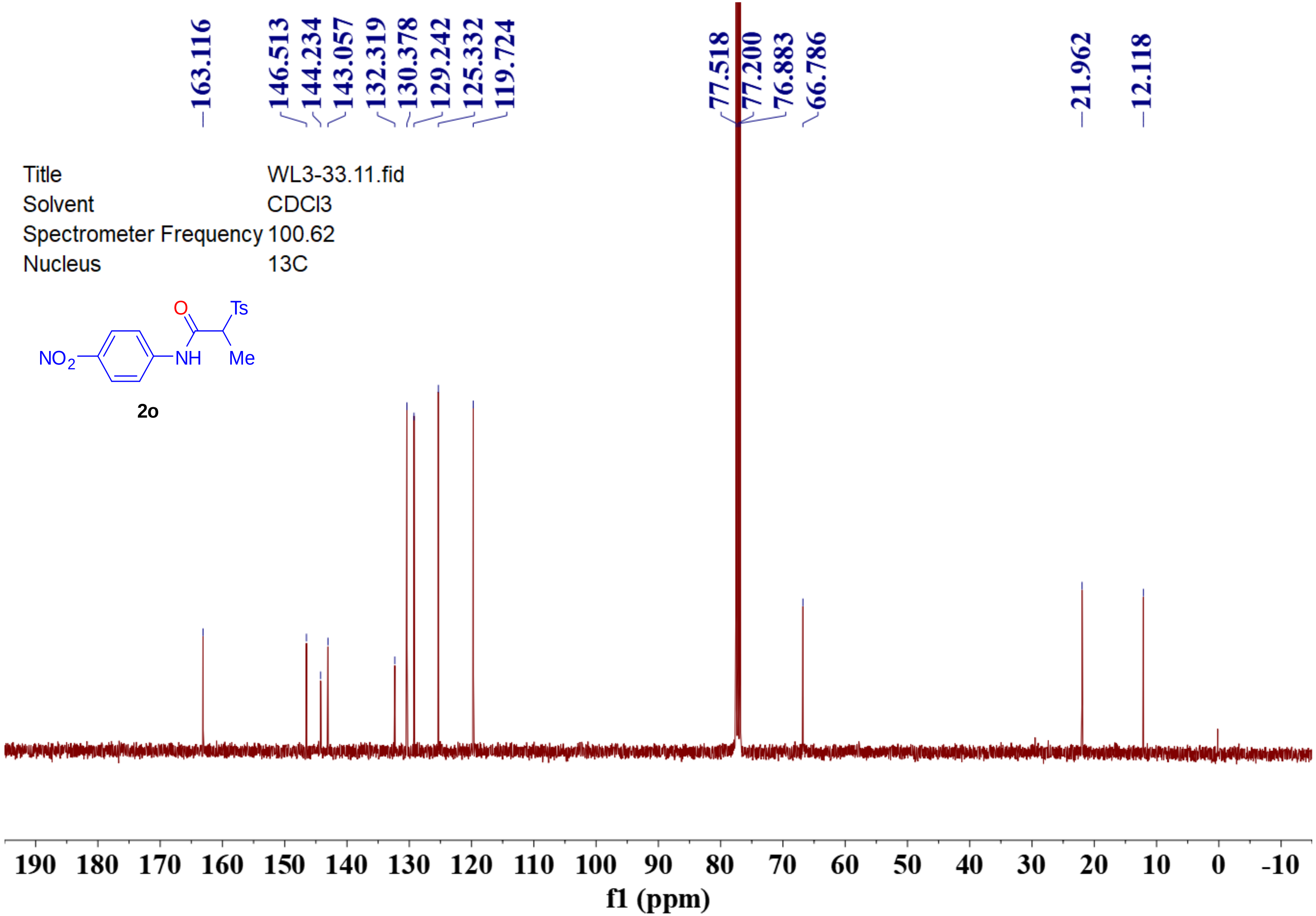
Title	WL3-52.11.fid
Solvent	CDCl3
Spectrometer Frequency	376.46
Nucleus	19F



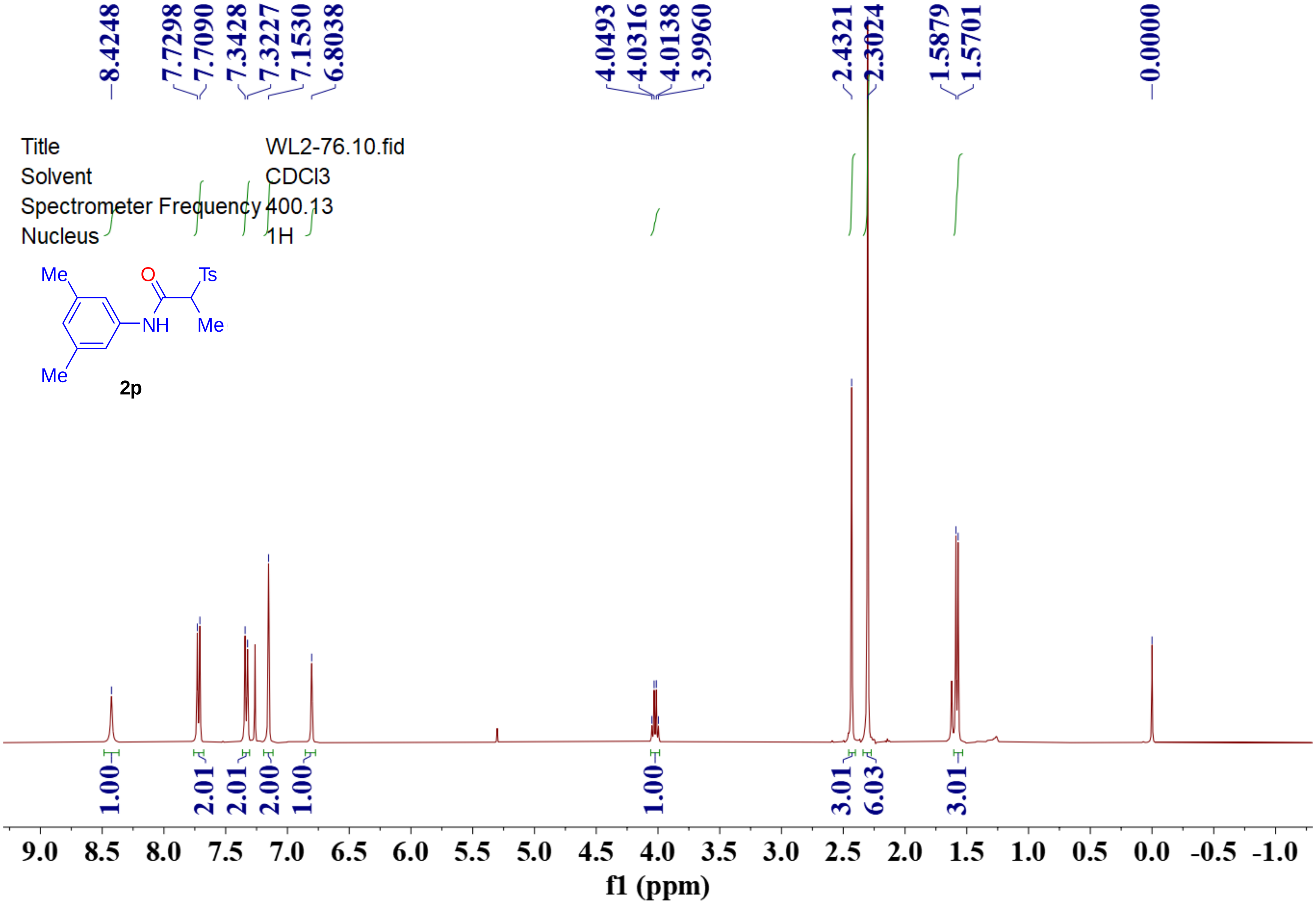
2n

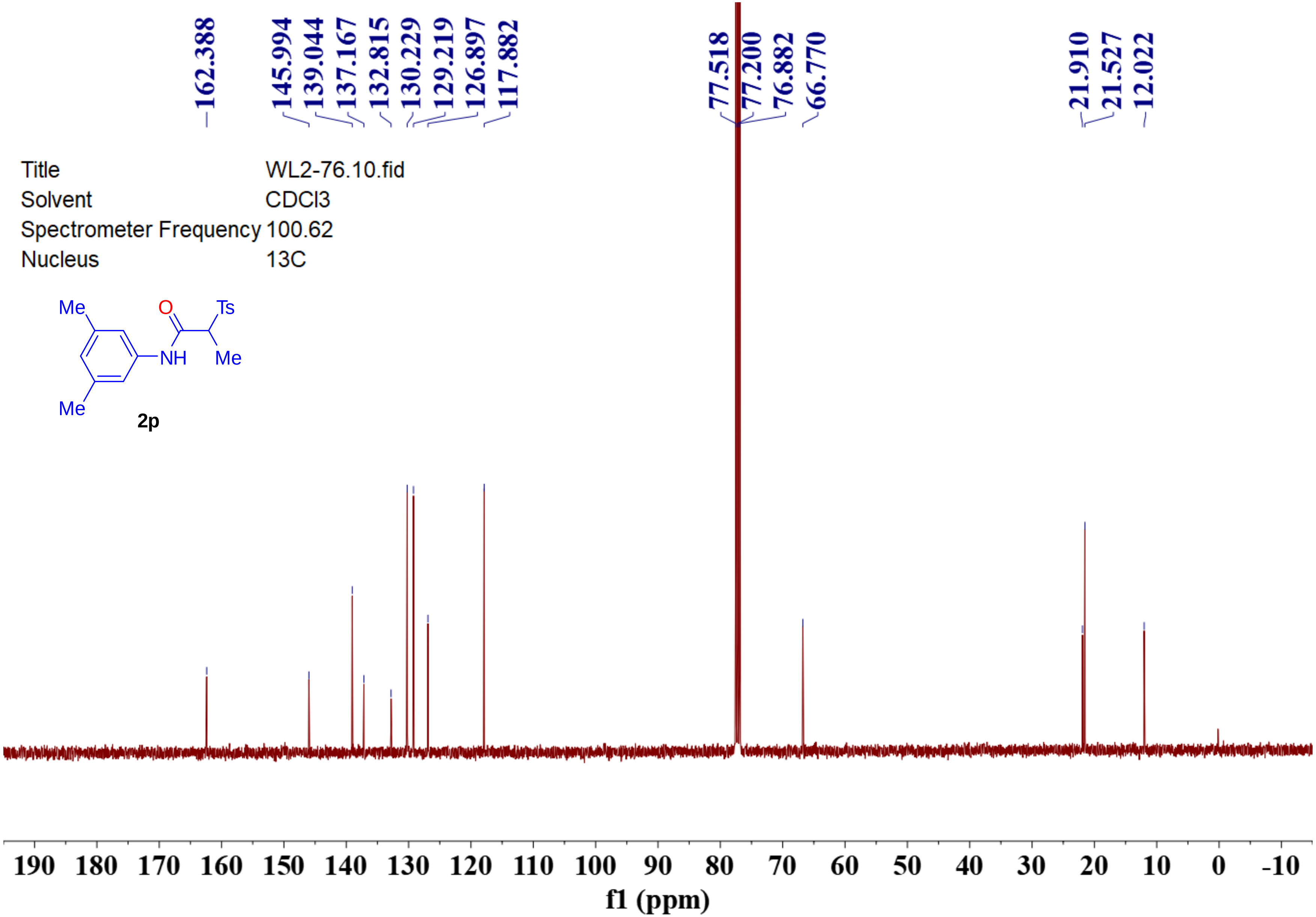

8.9886
8.2493
8.2263
7.7536
7.7306
7.7255
7.7048
7.3823
7.3628
4.1801
4.1626
4.1448
4.1272
2.4627
1.5829
1.5652
0.0000
Title WL3-33.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
NO2
NH
O
Ts
Me
1.00
2.00
4.02
1.99
1.00
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

2o

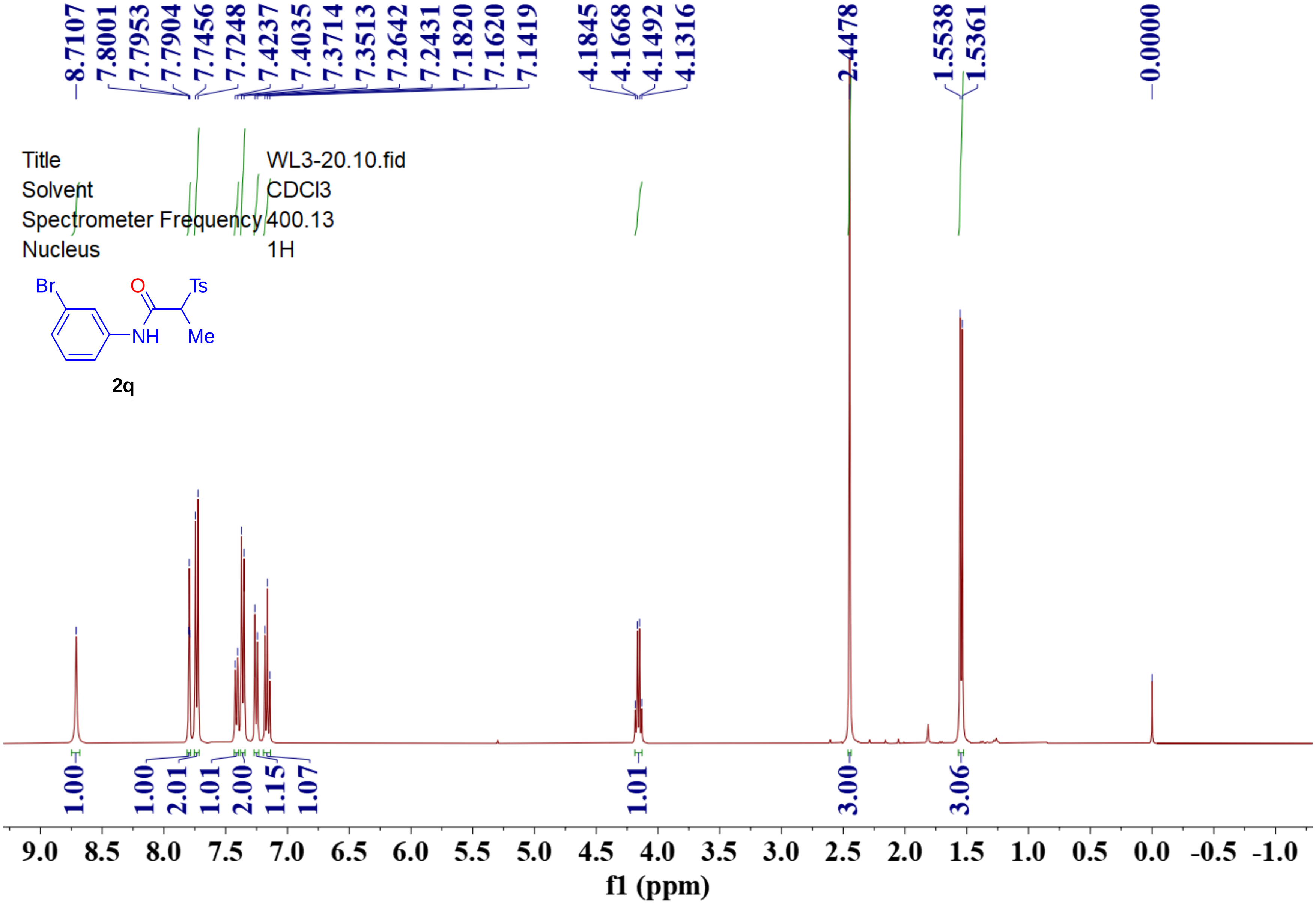

163.116
146.513
144.234
143.057
132.319
130.378
129.242
125.332
119.724
77.518
77.200
76.883
66.786
21.962
12.118
Title WL3-33.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
NO2
NH
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

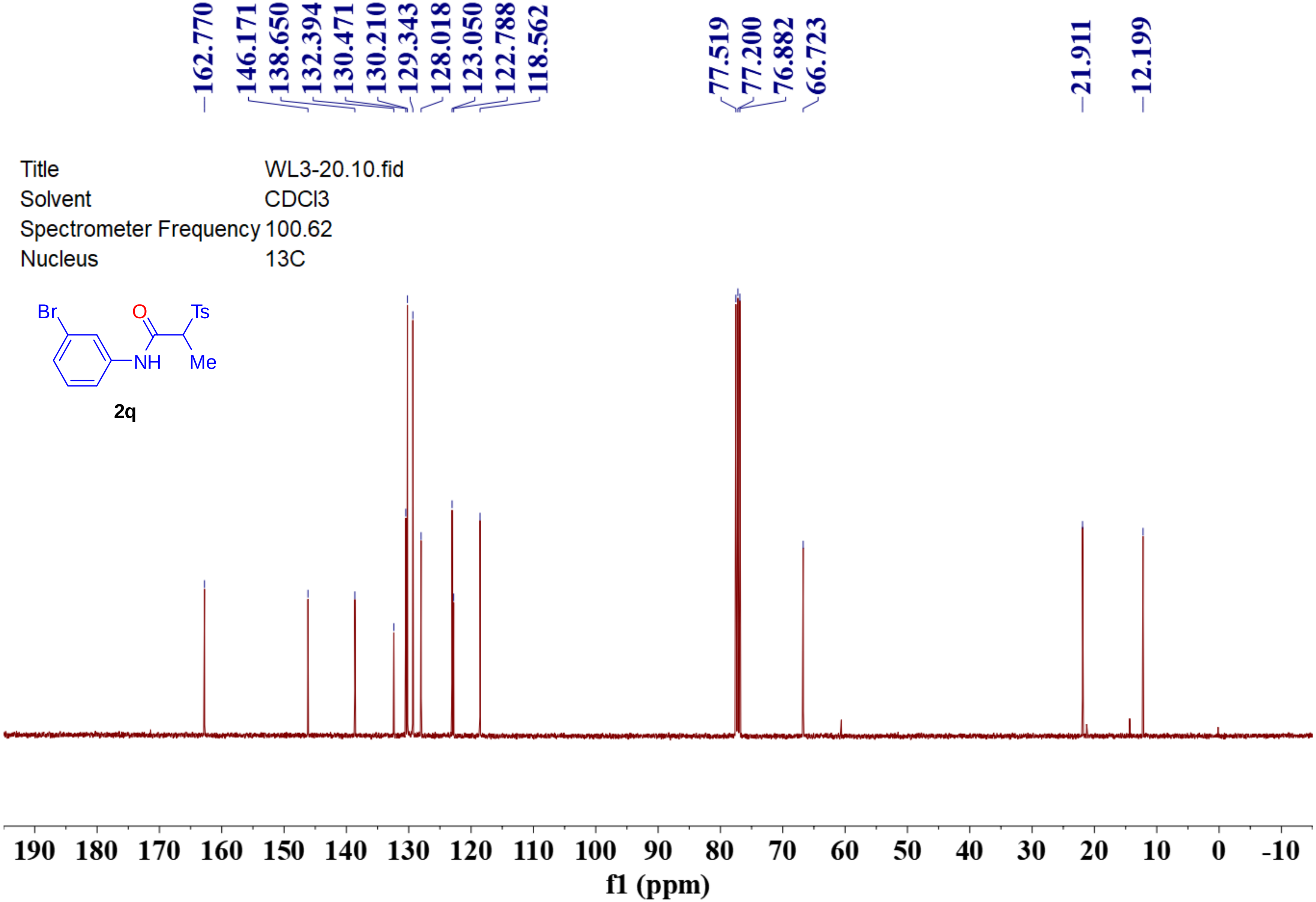
2o


8.4248
7.7298
7.7090
7.3428
7.3227
7.1530
6.8038
4.0493
4.0316
4.0138
3.9960
2.4321
2.3024
1.5879
1.5701
0.0000
Title WL2-76.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Me
O
Ts
NH
Me
Me
2p
1.00
2.01
2.01
2.00
1.00
1.00
3.01
6.03
3.01
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
-0.5
-1.0
f1 (ppm)


162.388
145.994
139.044
137.167
132.815
130.229
129.219
126.897
117.882
77.518
77.200
76.882
66.770
21.910
21.527
12.022
Title WL2-76.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
Me
O
Ts
NH
Me
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

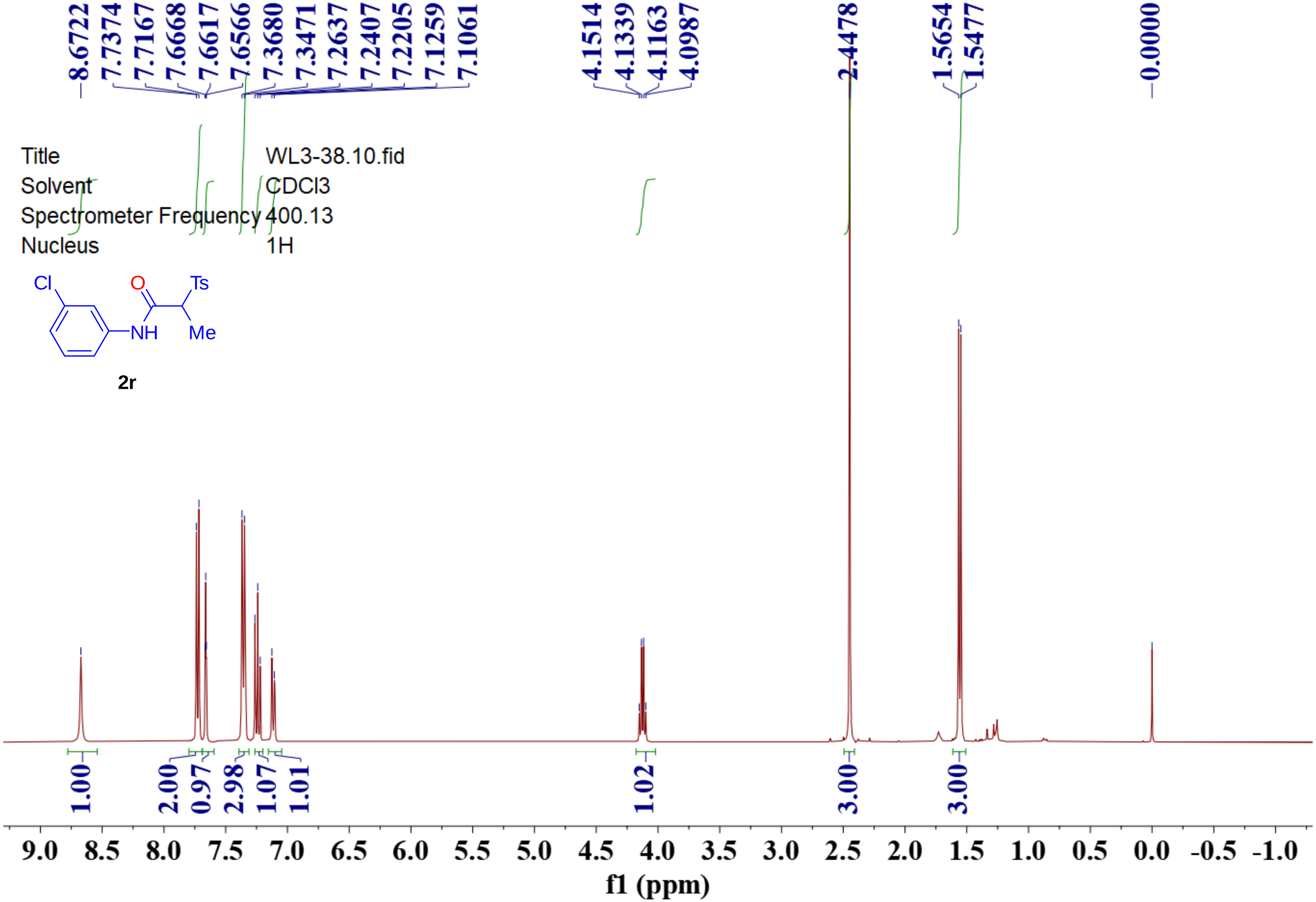
2p

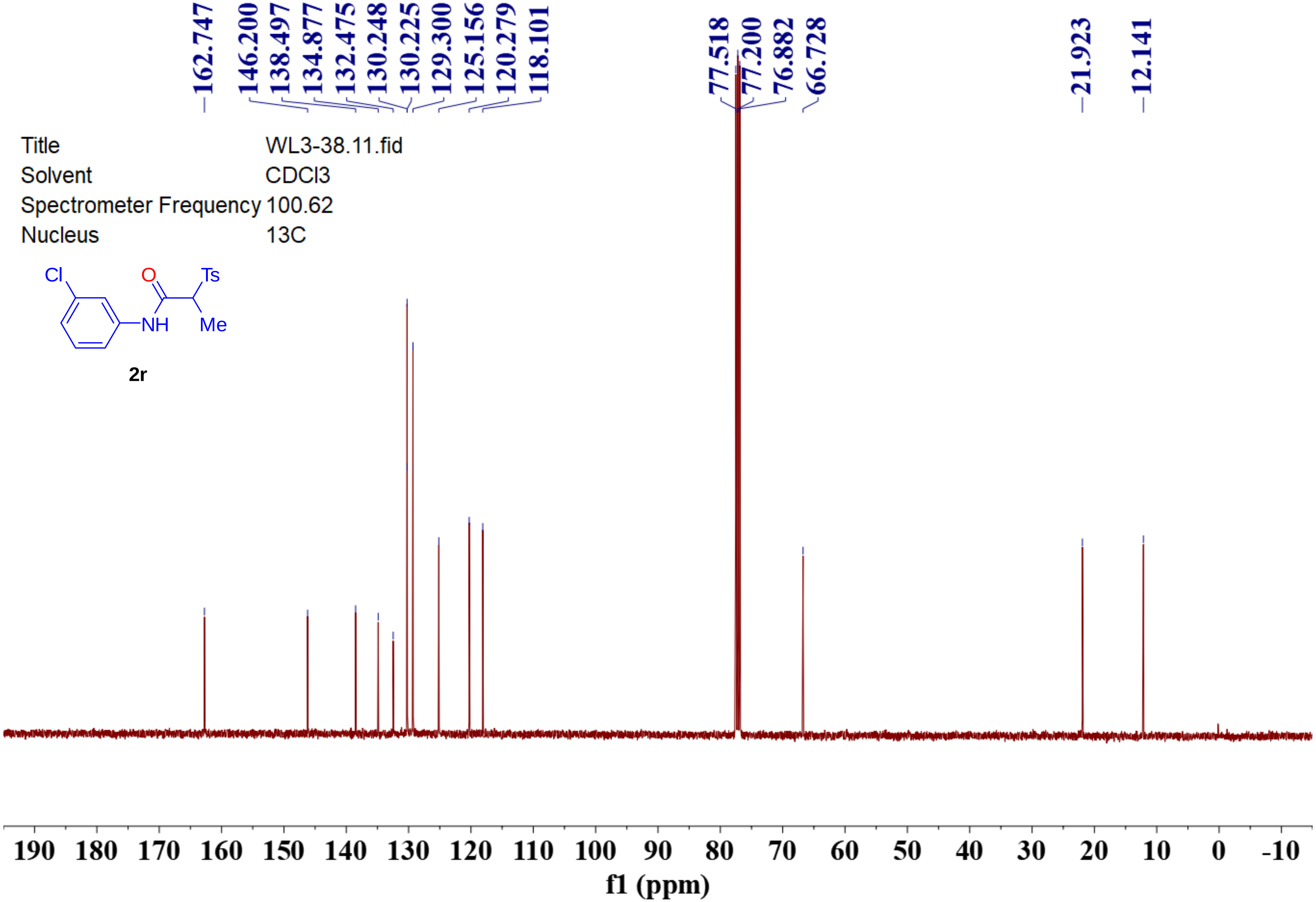

8.7107
7.8001
7.7953
7.7904
7.7456
7.7248
7.4237
7.4035
7.3714
7.3513
7.2642
7.2431
7.1820
7.1620
7.1419
4.1845
4.1668
4.1492
4.1316
2.4478
1.5538
1.5361
0.0000
Title WL3-20.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Br
O
Ts
NH
Me
2q
1.00
1.00
2.01
1.01
2.00
1.15
1.07
1.01
3.00
3.06
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
-0.5
-1.0
f1 (ppm)



Title WL3-20.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

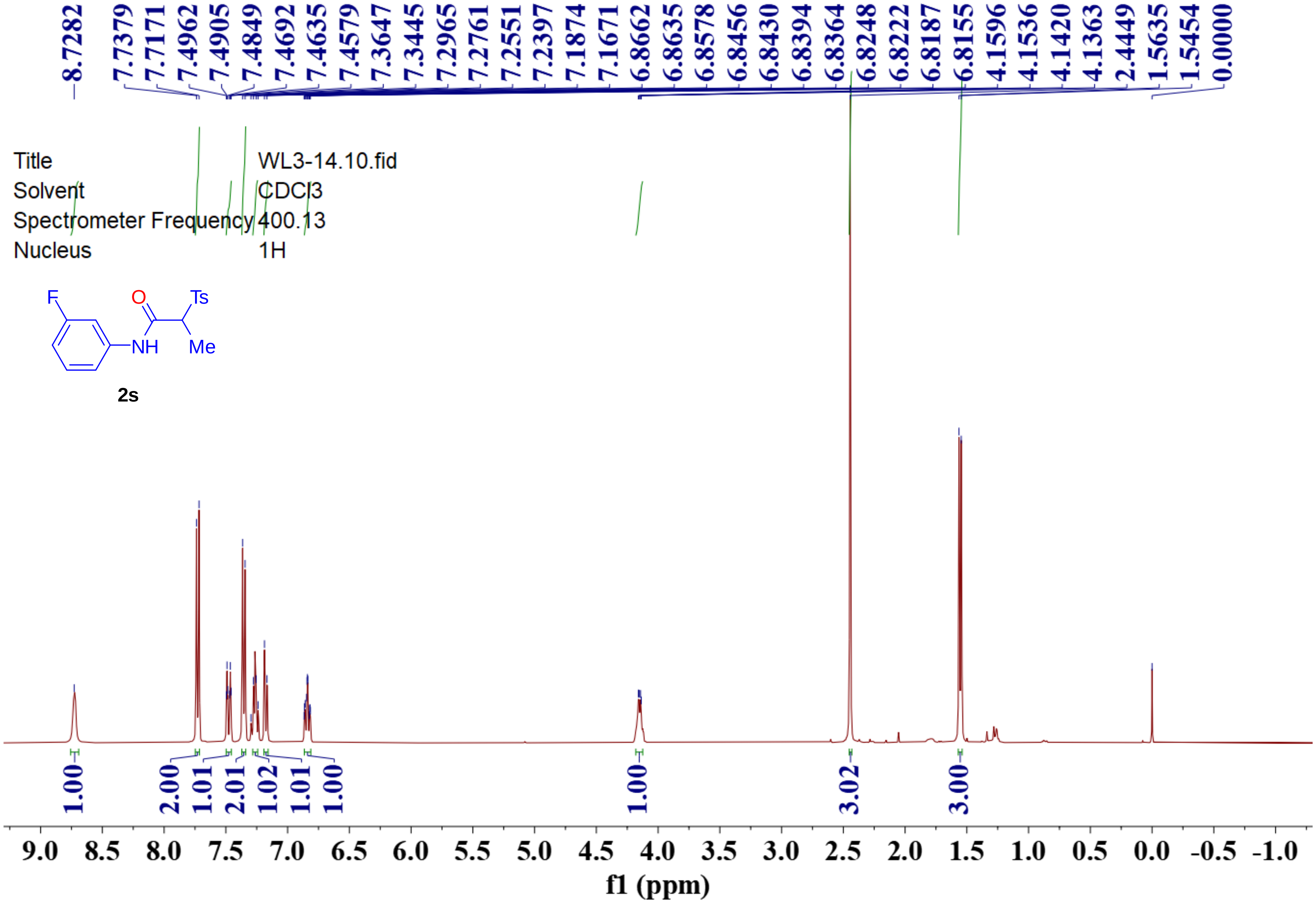
2q

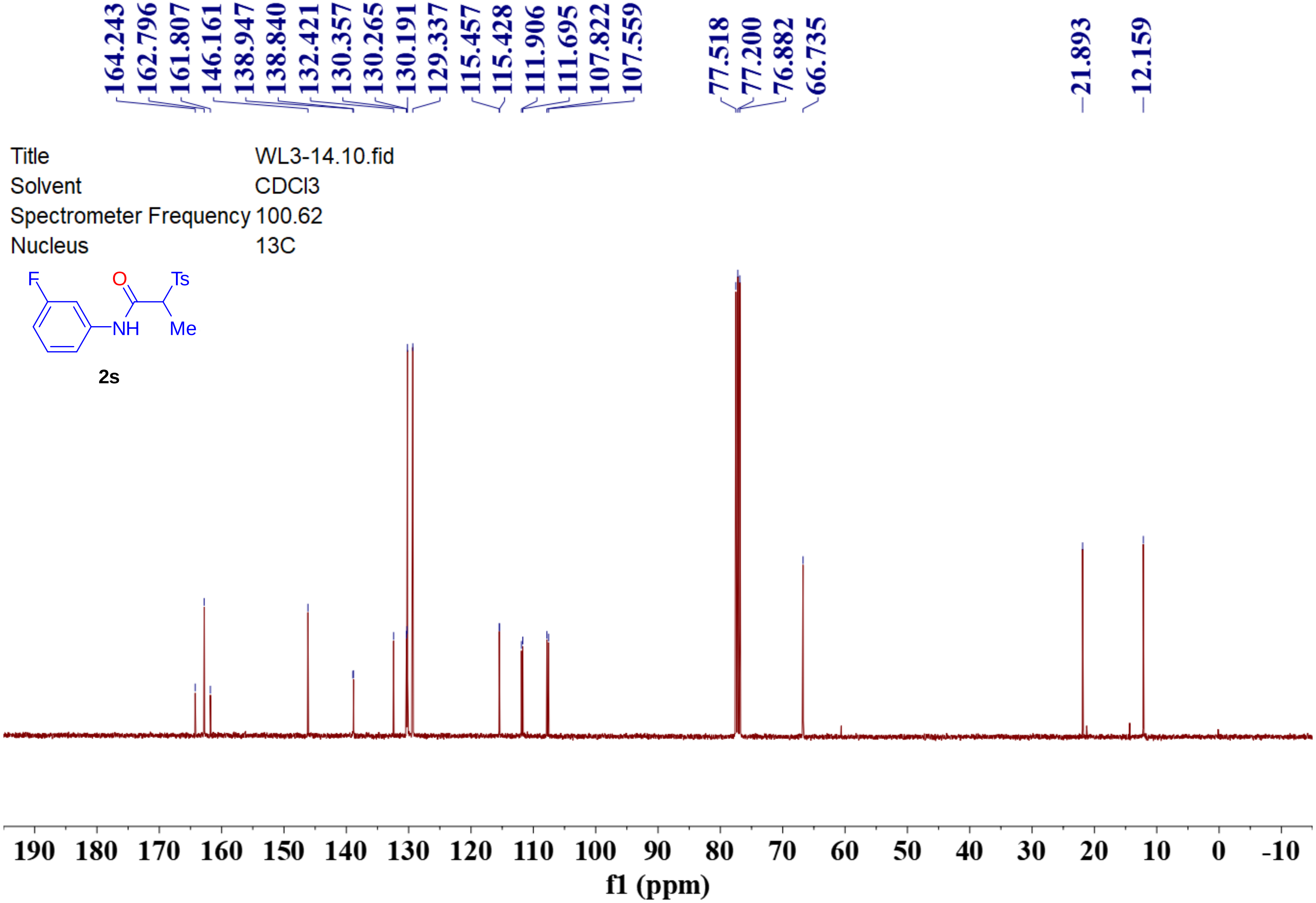

8.6722
7.7374
7.7167
7.6668
7.6617
7.6566
7.3680
7.3471
7.2637
7.2407
7.2205
7.1259
7.1061
4.1514
4.1339
4.1163
4.0987
2.4478
1.5654
1.5477
0.0000
Title
WL3-38.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
Cl
O
Ts
NH
Me
2r
1.00
2.00
0.97
2.98
1.07
1.01
1.02
3.00
3.00
9.0
8.5
8.0
7.5
7.0
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
-0.5
-1.0
f1 (ppm)



Title WL3-38.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

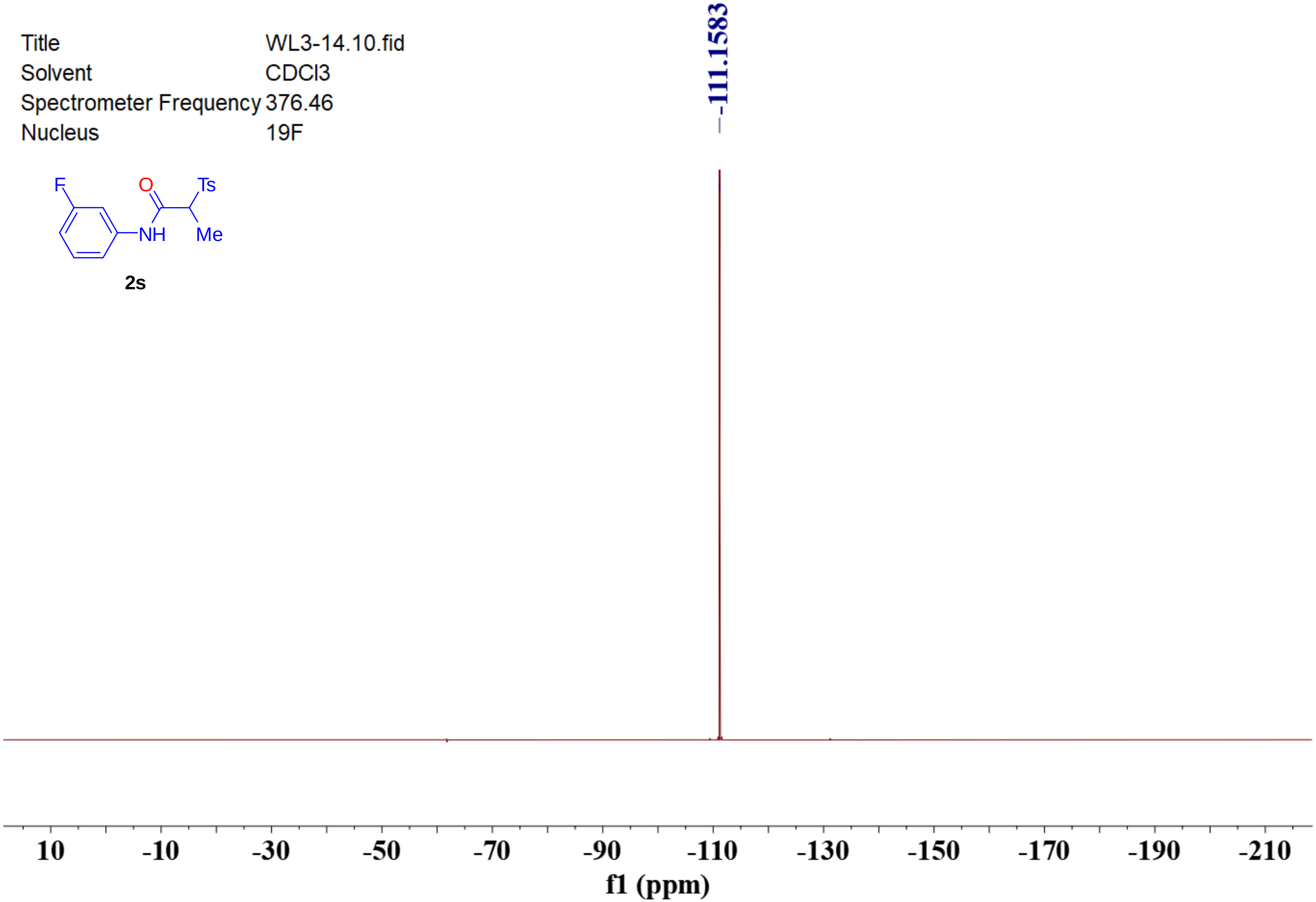
2r


8.7282
7.7379
7.7171
7.4962
7.4905
7.4849
7.4692
7.4635
7.4579
7.3647
7.3445
7.2965
7.2761
7.2551
7.2397
7.1874
7.1671
6.8662
6.8635
6.8578
6.8456
6.8430
6.8394
6.8364
6.8248
6.8222
6.8187
6.8155
4.1596
4.1536
4.1420
4.1363
2.4449
1.5635
1.5454
0.0000
Title WL3-14.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
F
O
Ts
NH
Me
2s
1.00
2.00
1.01
2.01
1.02
1.01
1.00
1.00
3.02
3.00
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
-0.5
-1.0
f1 (ppm)

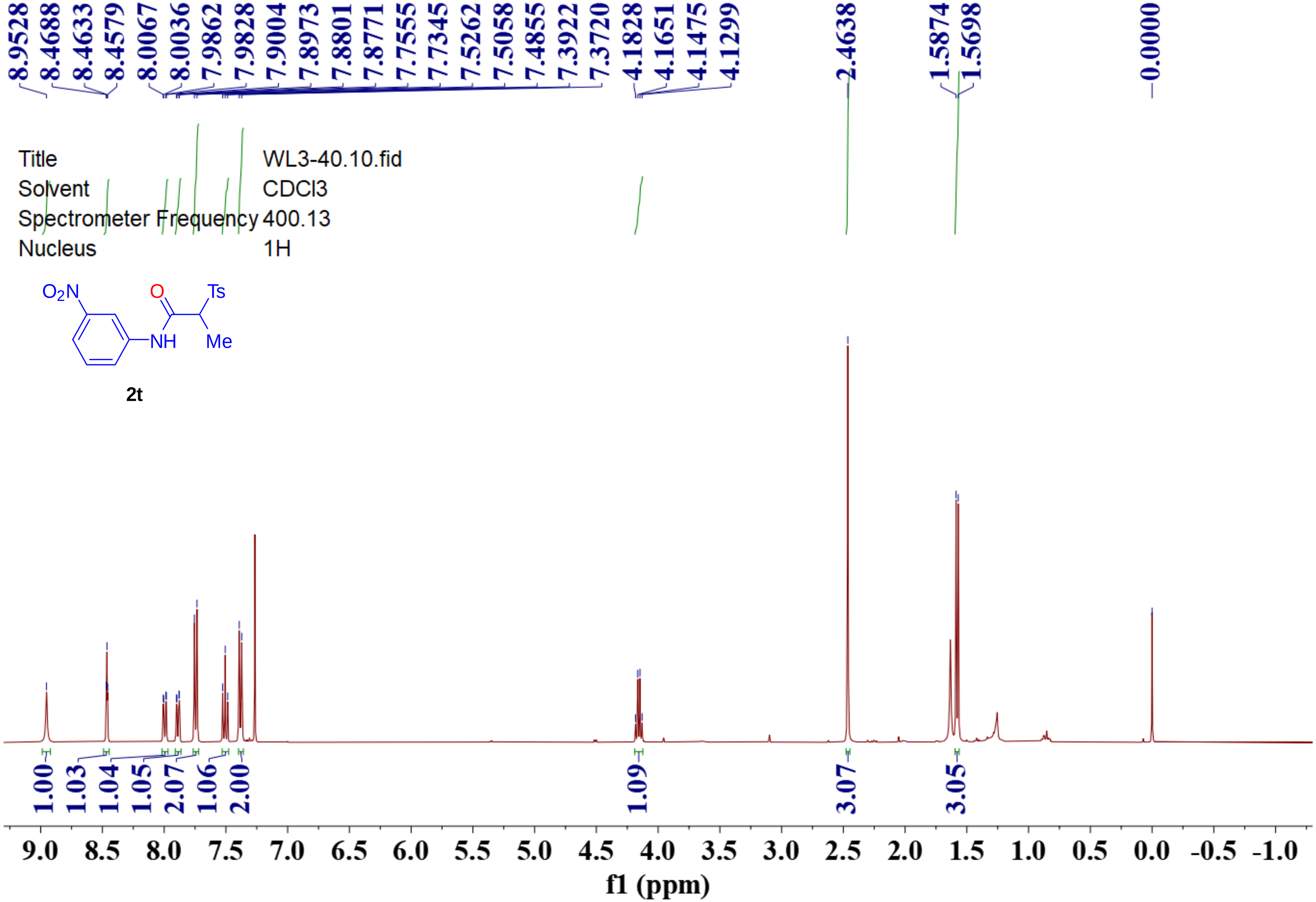


Title WL3-14.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

Title	WL3-14.10.fid
Solvent	CDCl3
Spectrometer Frequency	376.46
Nucleus	19F



2s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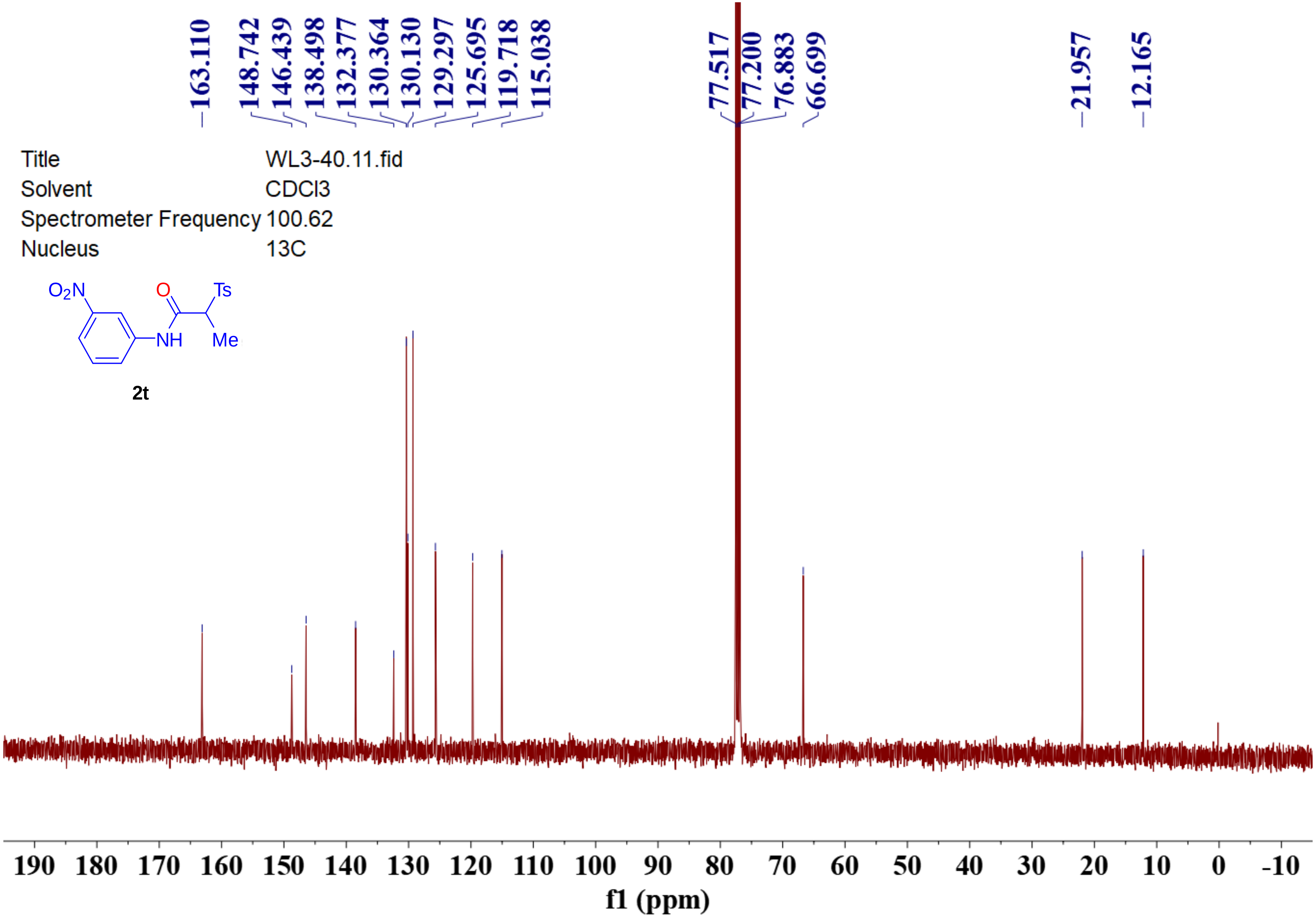
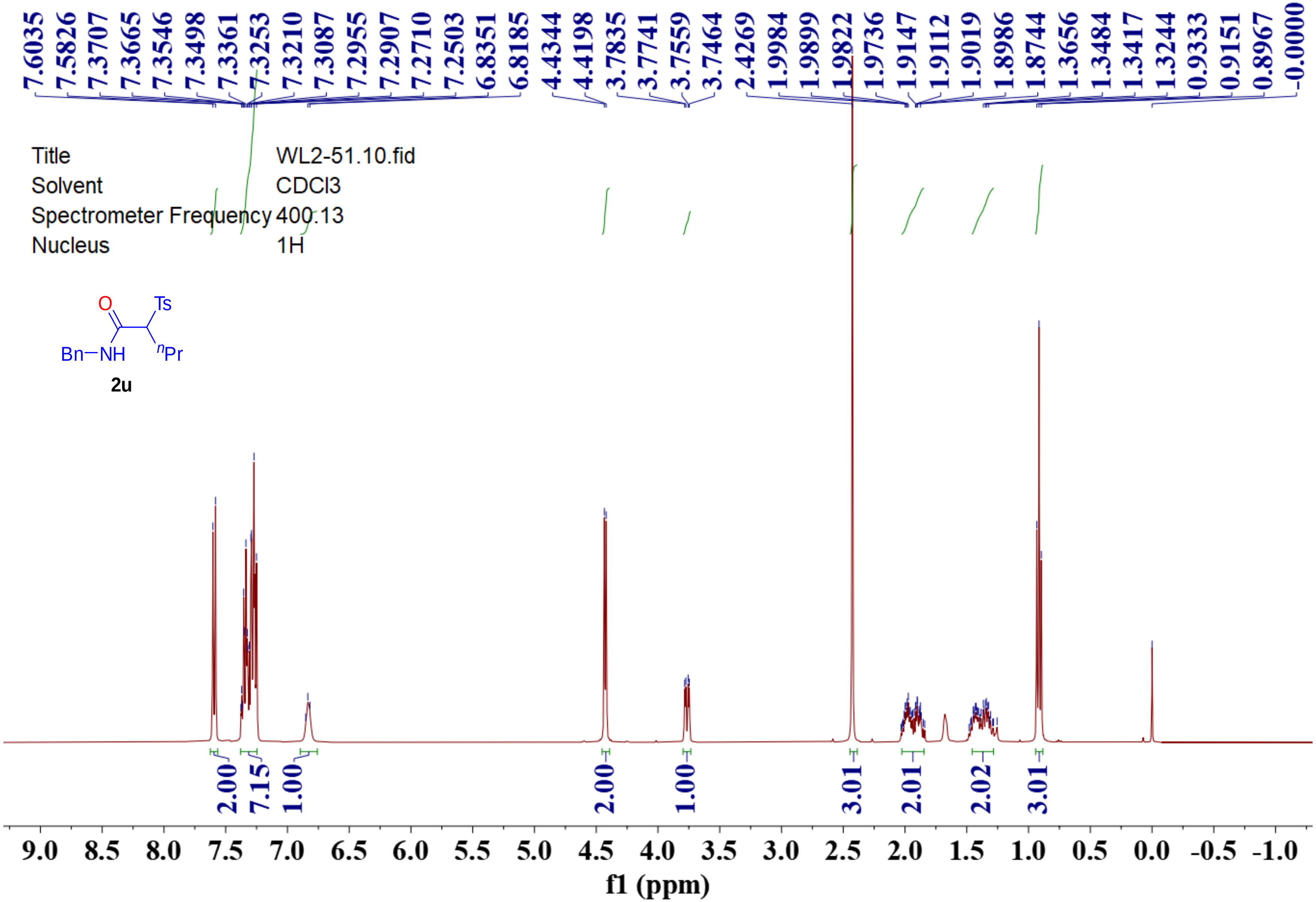
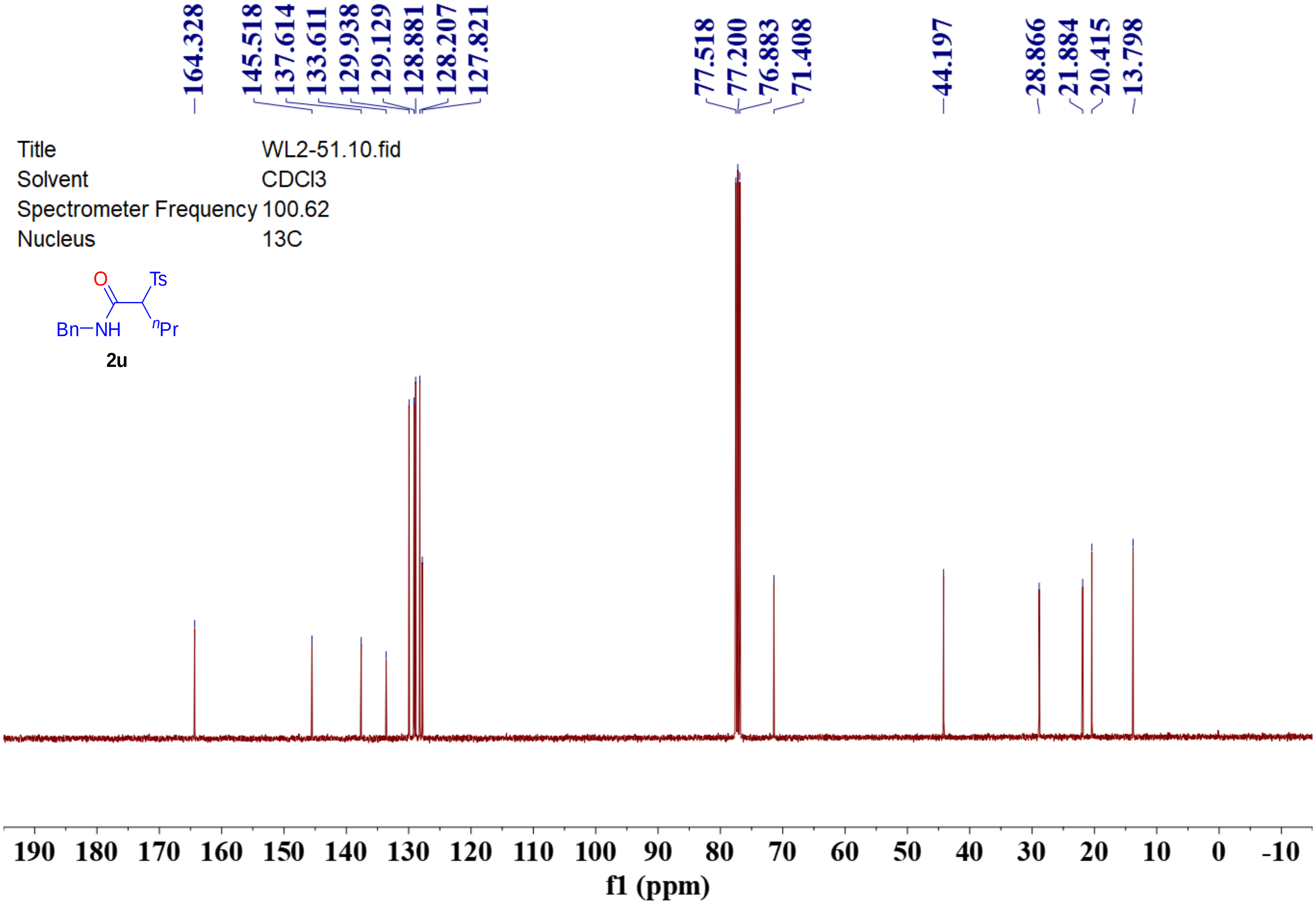
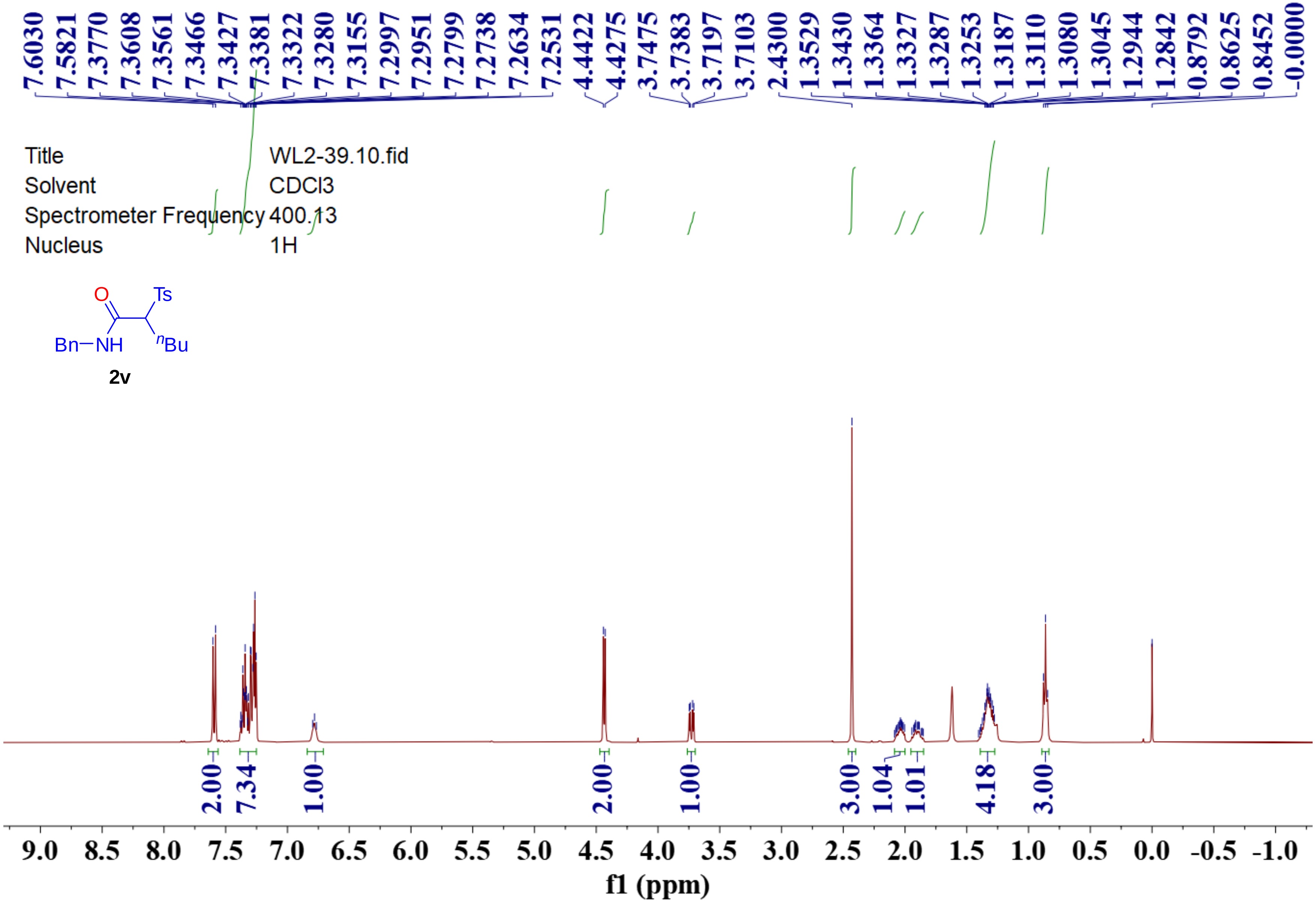
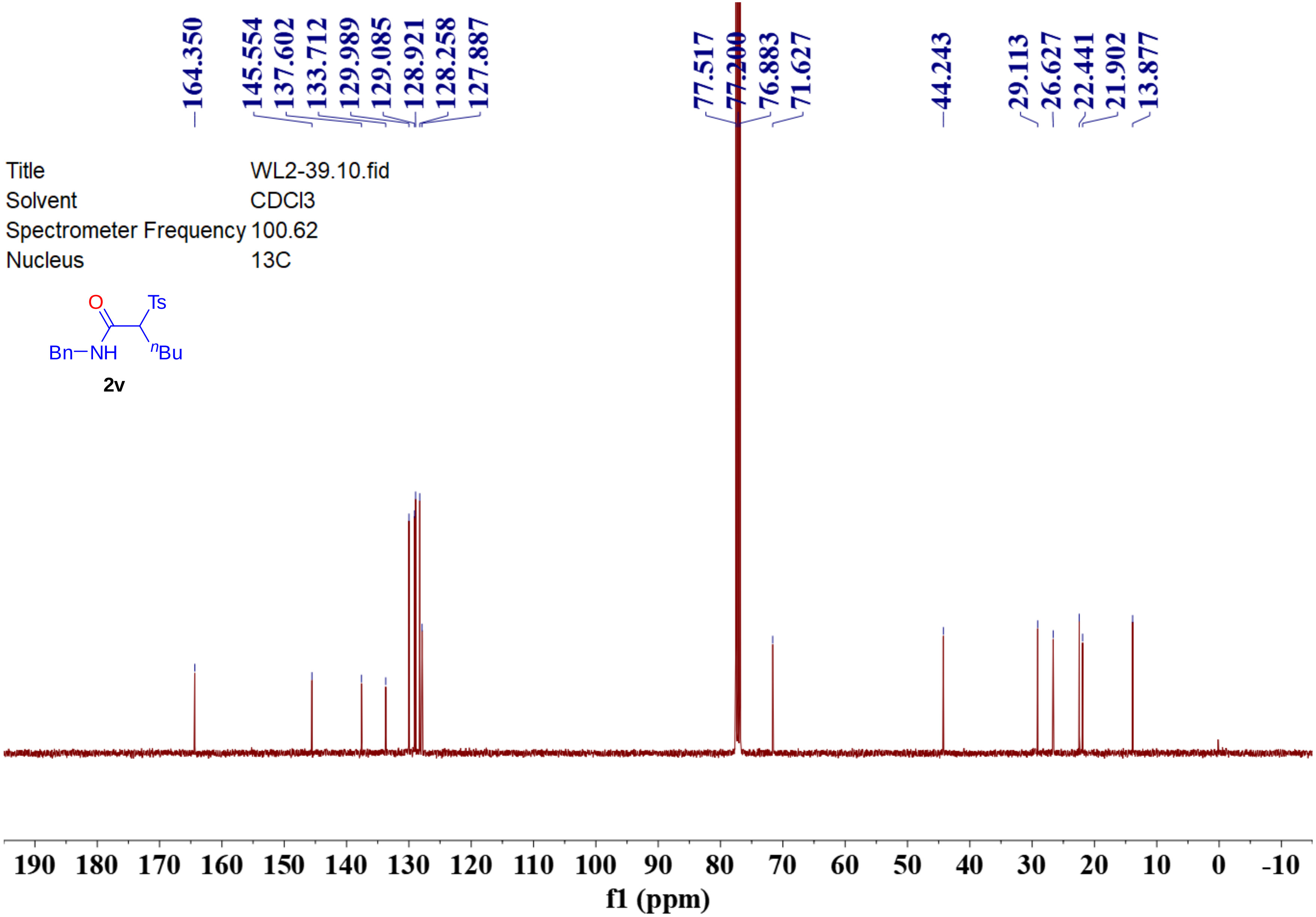
8.4688
8.4633
8.4579
8.0067
8.0036
7.9862
7.9828
7.9004
7.8973
7.8801
7.8771
7.7555
7.7345
7.5262
7.5058
7.4855
7.3922
7.3720
4.1828
4.1651
4.1475
4.1299
2.4638
1.5874
1.5698
-0.0000
Title WL3-40.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O2N
O
Ts
NH
Me
2t
1.00
1.03
1.04
1.05
2.07
1.06
2.00
1.09
3.07
3.05
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
-0.5
-1.0
f1 (ppm)


163.110
148.742
146.439
138.498
132.377
130.364
130.130
129.297
125.695
119.718
115.038
77.517
77.200
76.883
66.699
21.957
12.165
Title WL3-40.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O2N
O
Ts
NH
Me
2t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.6035
7.5826
7.3707
7.3665
7.3546
7.3498
7.3361
7.3253
7.3210
7.3087
7.2955
7.2907
7.2710
7.2503
6.8351
6.8185
4.4344
4.4198
3.7835
3.7741
3.7559
3.7464
2.4269
1.9984
1.9899
1.9822
1.9736
1.9147
1.9112
1.9019
1.8986
1.8744
1.3656
1.3484
1.3417
1.3244
0.9333
0.9151
0.8967
-0.0000
Title
WL2-51.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
O
Ts
Bn−NH
ⁿPr
2u
2.00
7.15
1.00
2.00
1.00
3.01
2.01
2.02
3.01
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
-0.5
-1.0
f1 (ppm)

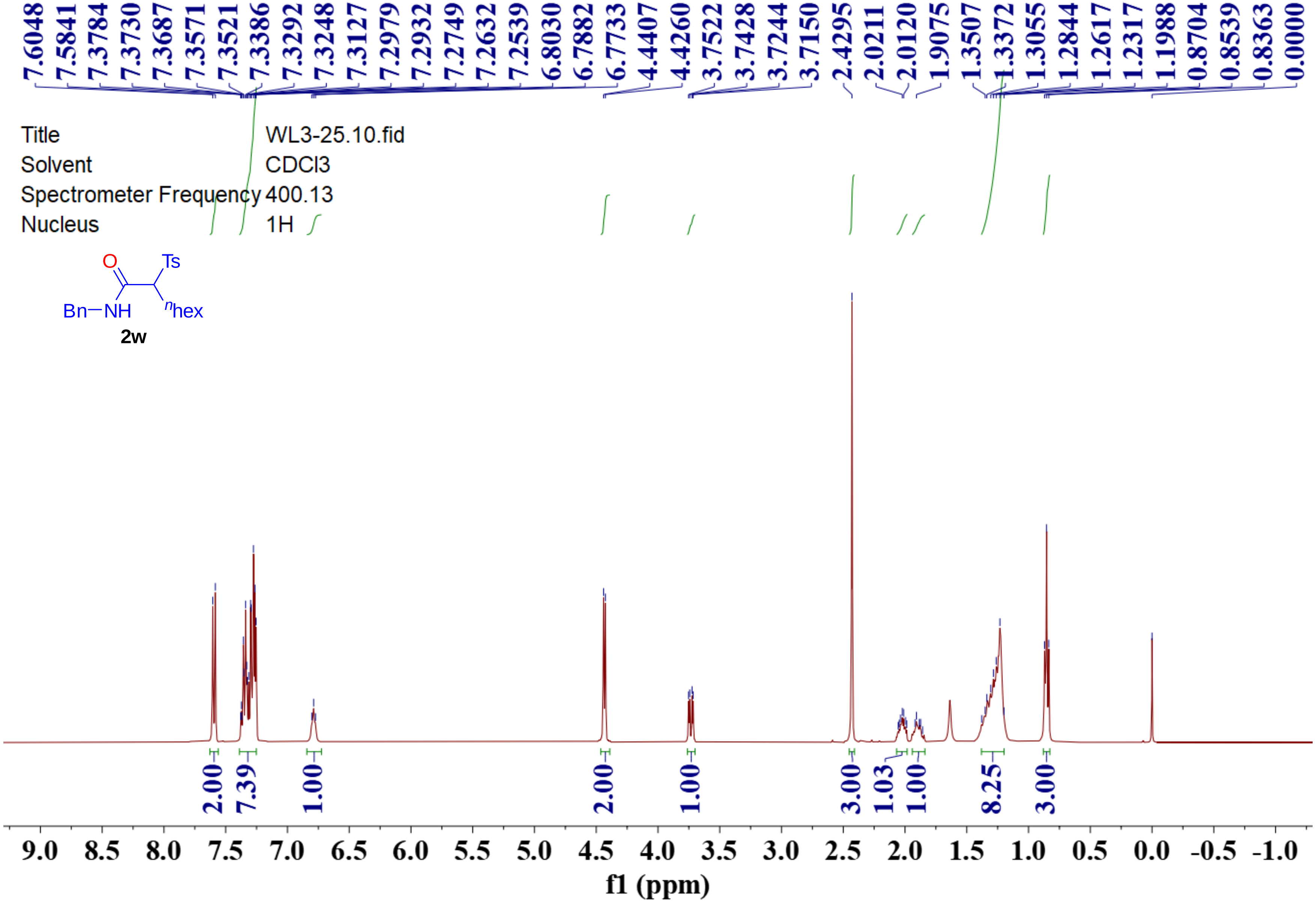

164.328
145.518
137.614
133.611
129.938
129.129
128.881
128.207
127.821
77.518
77.200
76.883
71.408
44.197
28.866
21.884
20.415
13.798
Title WL2-51.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Bn−NH
^{n}Pr
2u
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.6030
7.5821
7.3770
7.3608
7.3561
7.3466
7.3427
7.3381
7.3322
7.3280
7.3155
7.2997
7.2951
7.2799
7.2738
7.2634
7.2531
4.4422
4.4275
3.7475
3.7383
3.7197
3.7103
2.4300
1.3529
1.3430
1.3364
1.3327
1.3287
1.3253
1.3187
1.3110
1.3080
1.3045
1.2944
1.2842
0.8792
0.8625
0.8452
-0.0000
Title
WL2-39.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
O
Ts
Bn
NH
^{n}Bu
2v
2.00
7.34
1.00
2.00
1.00
3.00
1.04
1.01
4.18
3.00
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
-0.5
-1.0
f1 (ppm)



Title	WL2-39.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2v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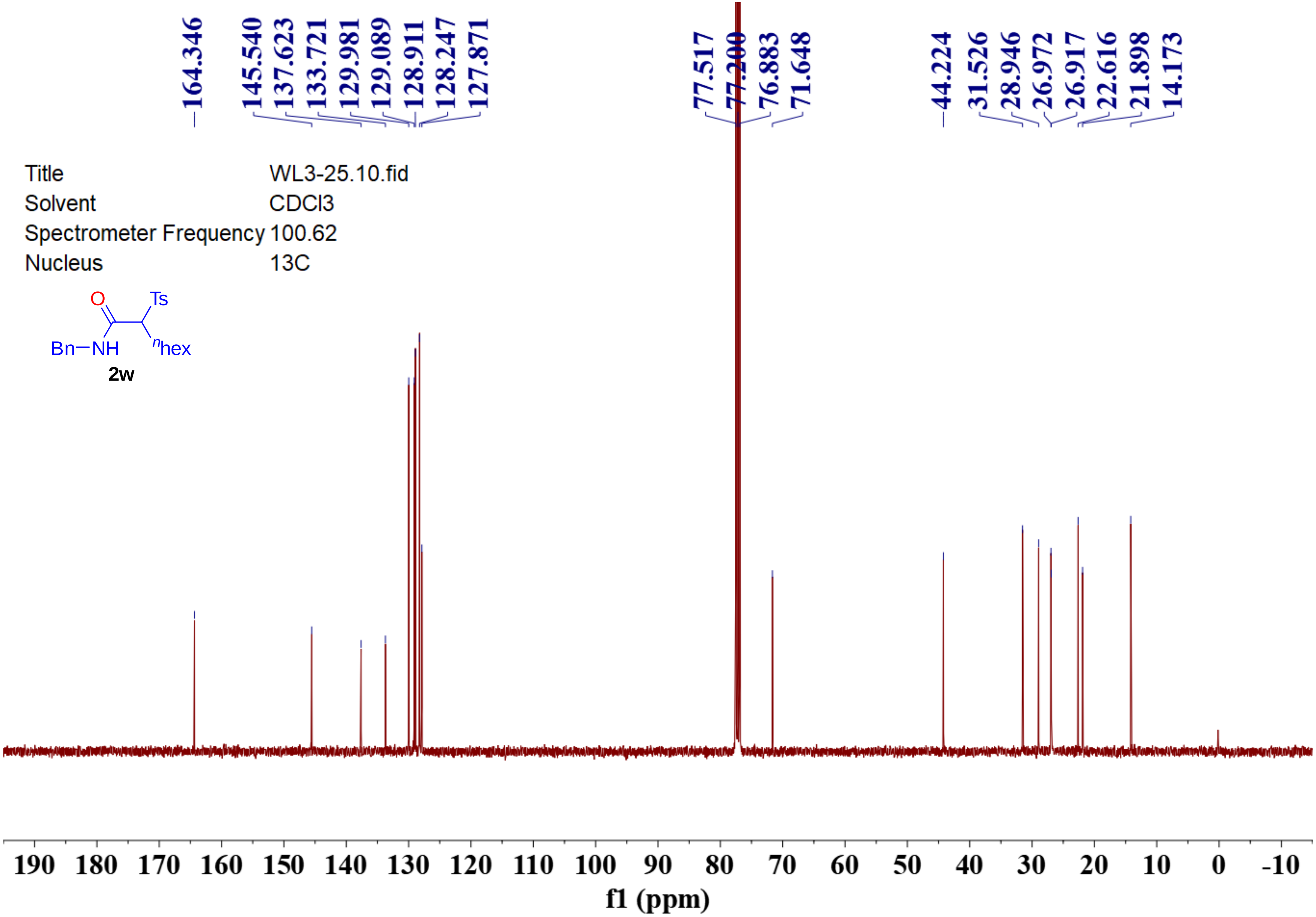
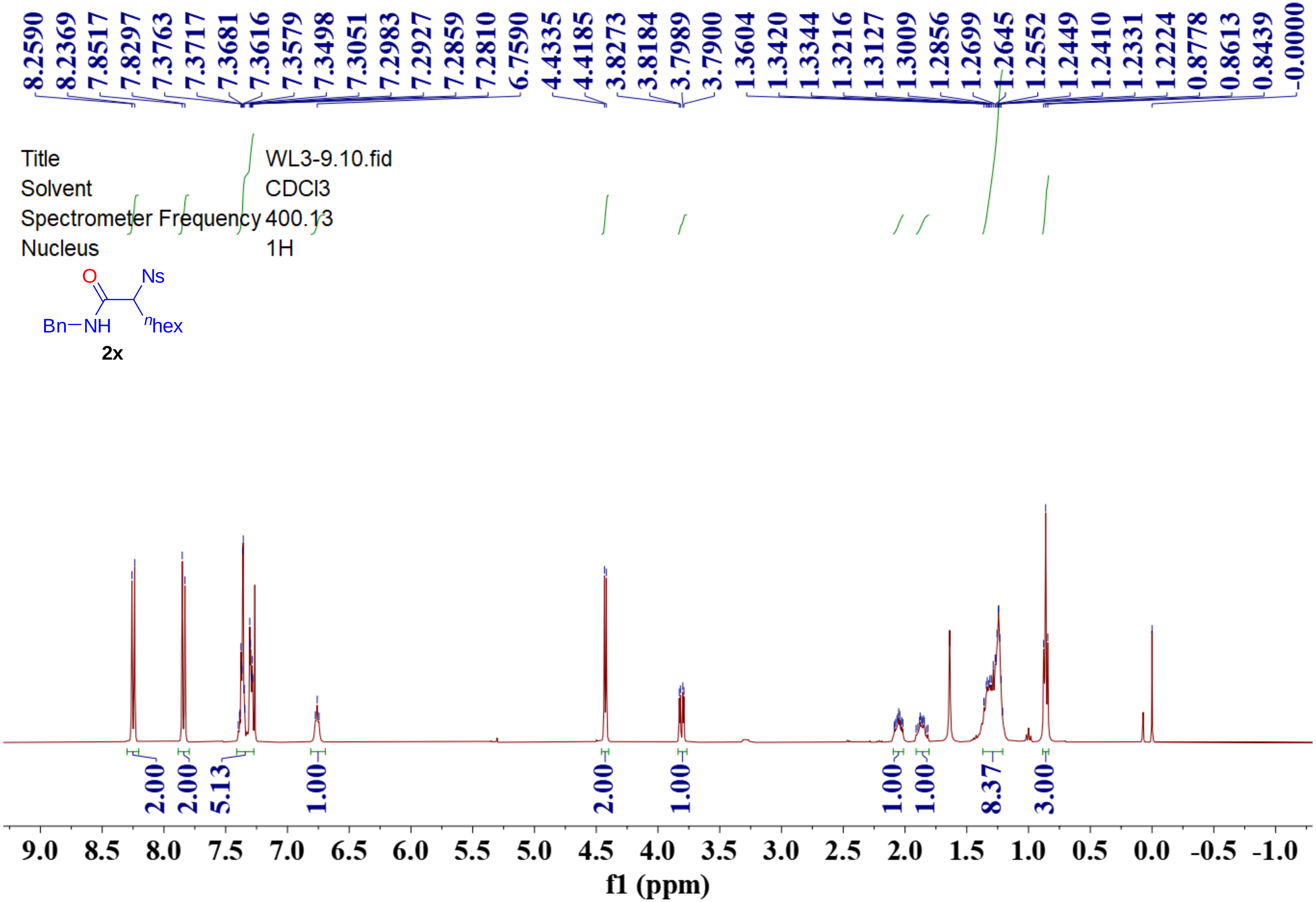
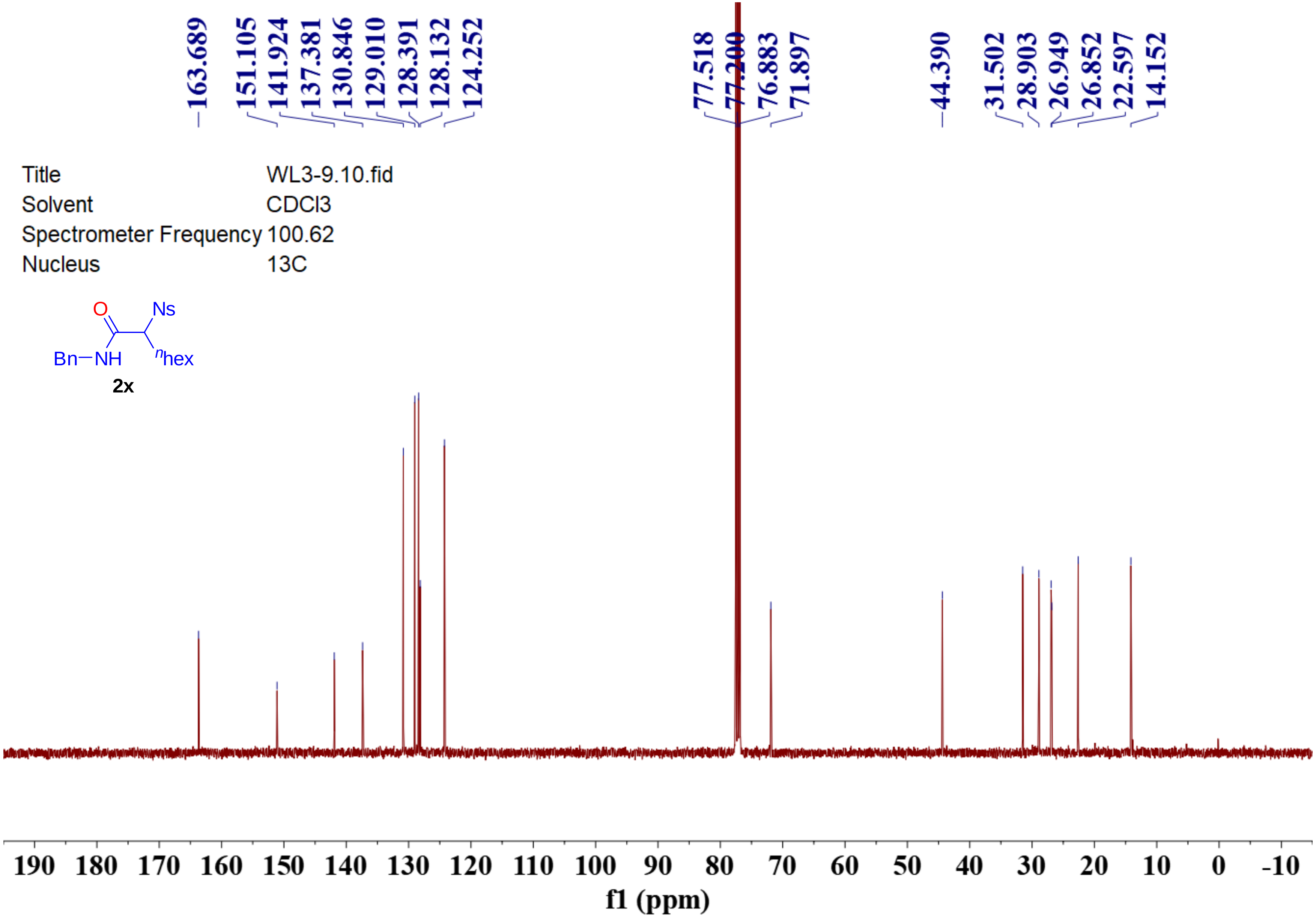
7.5841
7.3784
7.3730
7.3687
7.3571
7.3521
7.3386
7.3292
7.3248
7.3127
7.2979
7.2932
7.2749
7.2632
7.2539
6.8030
6.7882
6.7733
4.4407
4.4260
3.7522
3.7428
3.7244
3.7150
2.4295
2.0211
2.0120
1.9075
1.3507
1.3372
1.3055
1.2844
1.2617
1.2317
1.1988
0.8704
0.8539
0.8363
0.0000
Title WL3-25.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn—NH
nhex
2w
2.00
7.39
1.00
2.00
1.00
3.00
1.03
1.00
8.25
3.00
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
-0.5
-1.0
f1 (ppm)

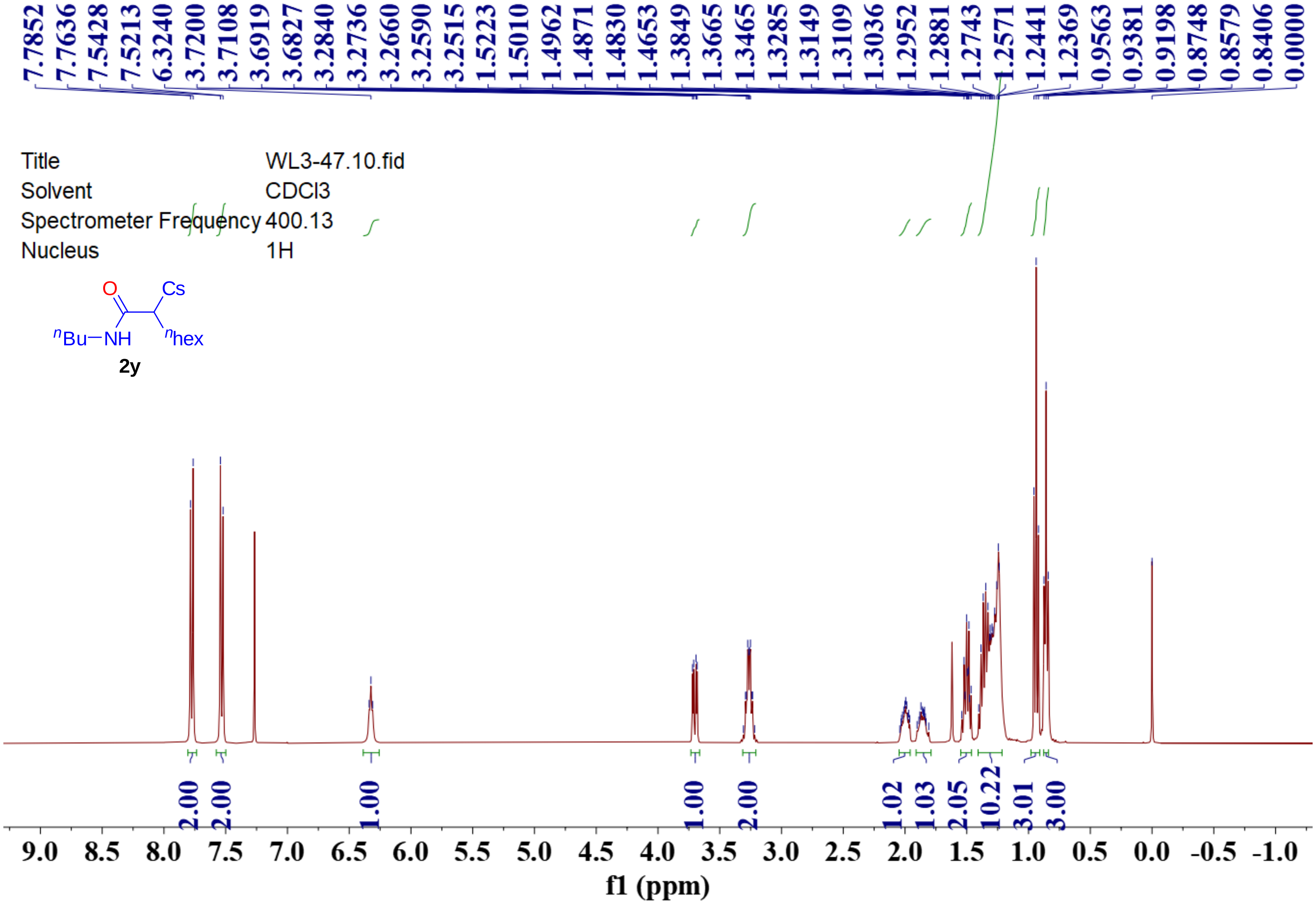


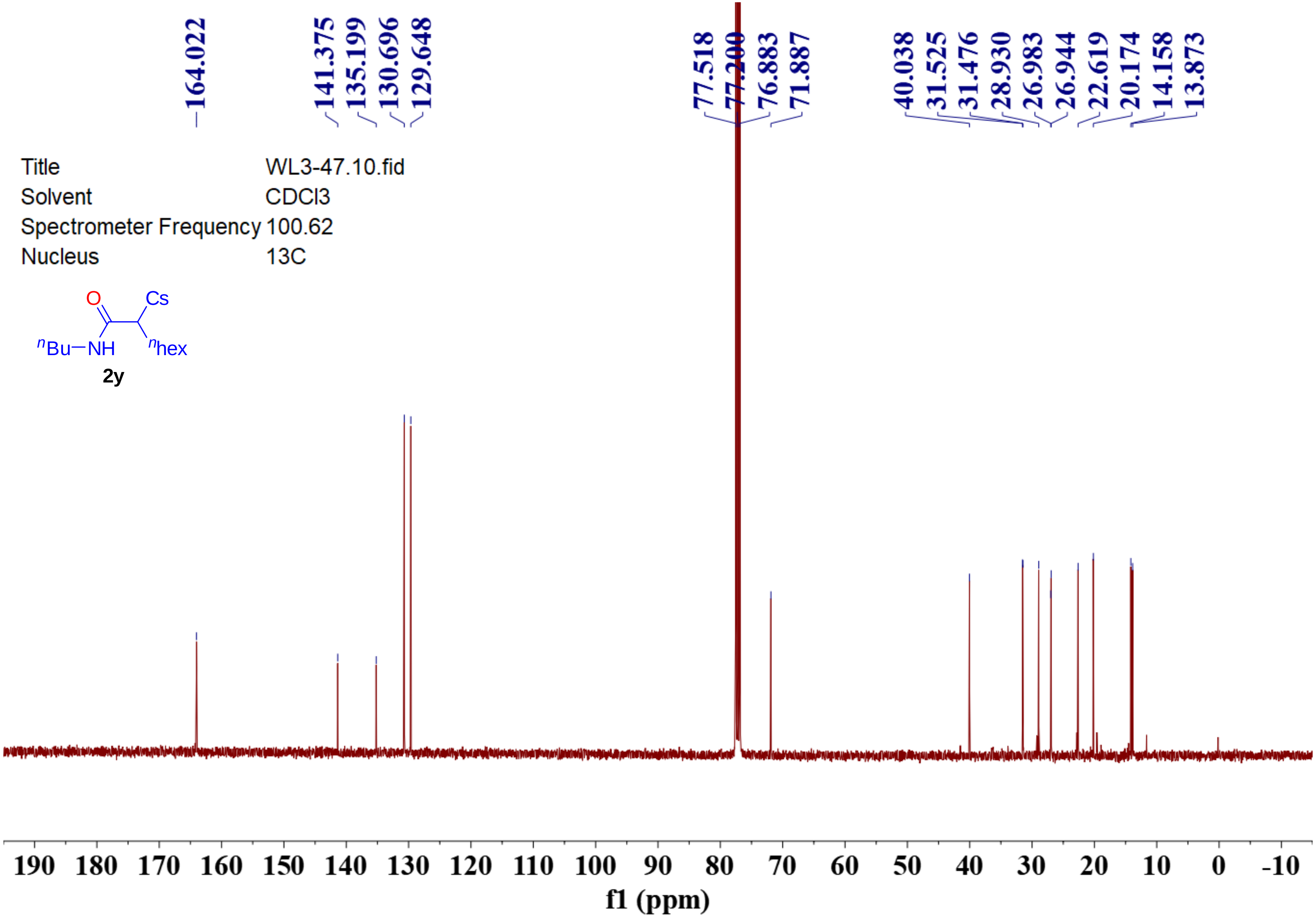
2w

Title	WL3-25.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C


8.2590
8.2369
7.8517
7.8297
7.3763
7.3717
7.3681
7.3616
7.3579
7.3498
7.3051
7.2983
7.2927
7.2859
7.2810
6.7590
4.4335
4.4185
3.8273
3.8184
3.7989
3.7900
1.3604
1.3420
1.3344
1.3216
1.3127
1.3009
1.2856
1.2699
1.2645
1.2552
1.2449
1.2410
1.2331
1.2224
0.8778
0.8613
0.8439
-0.0000
Title WL3-9.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ns
Bn—NH
^{n}hex
2x
2.00
2.00
5.13
1.00
2.00
1.00
1.00
1.00
8.37
3.00
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
-0.5
-1.0
f1 (ppm)


163.689
151.105
141.924
137.381
130.846
129.010
128.391
128.132
124.252
77.518
77.200
76.883
71.897
44.390
31.502
28.903
26.949
26.852
22.597
14.152
Title WL3-9.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ns
Bn−NH
^{n}hex
2x
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.7852
7.7636
7.5428
7.5213
6.3240
3.7200
3.7108
3.6919
3.6827
3.2840
3.2736
3.2660
3.2590
3.2515
1.5223
1.5010
1.4962
1.4871
1.4830
1.4653
1.3849
1.3665
1.3465
1.3285
1.3149
1.3109
1.3036
1.2952
1.2881
1.2743
1.2571
1.2441
1.2369
0.9563
0.9381
0.9198
0.8748
0.8579
0.8406
0.0000
Title WL3-47.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Cs
^{n}Bu—NH
^{n}hex
2y
2.00
2.00
1.00
1.00
2.00
1.02
1.03
2.05
10.22
3.01
3.00
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
-0.5
-1.0
f1 (ppm)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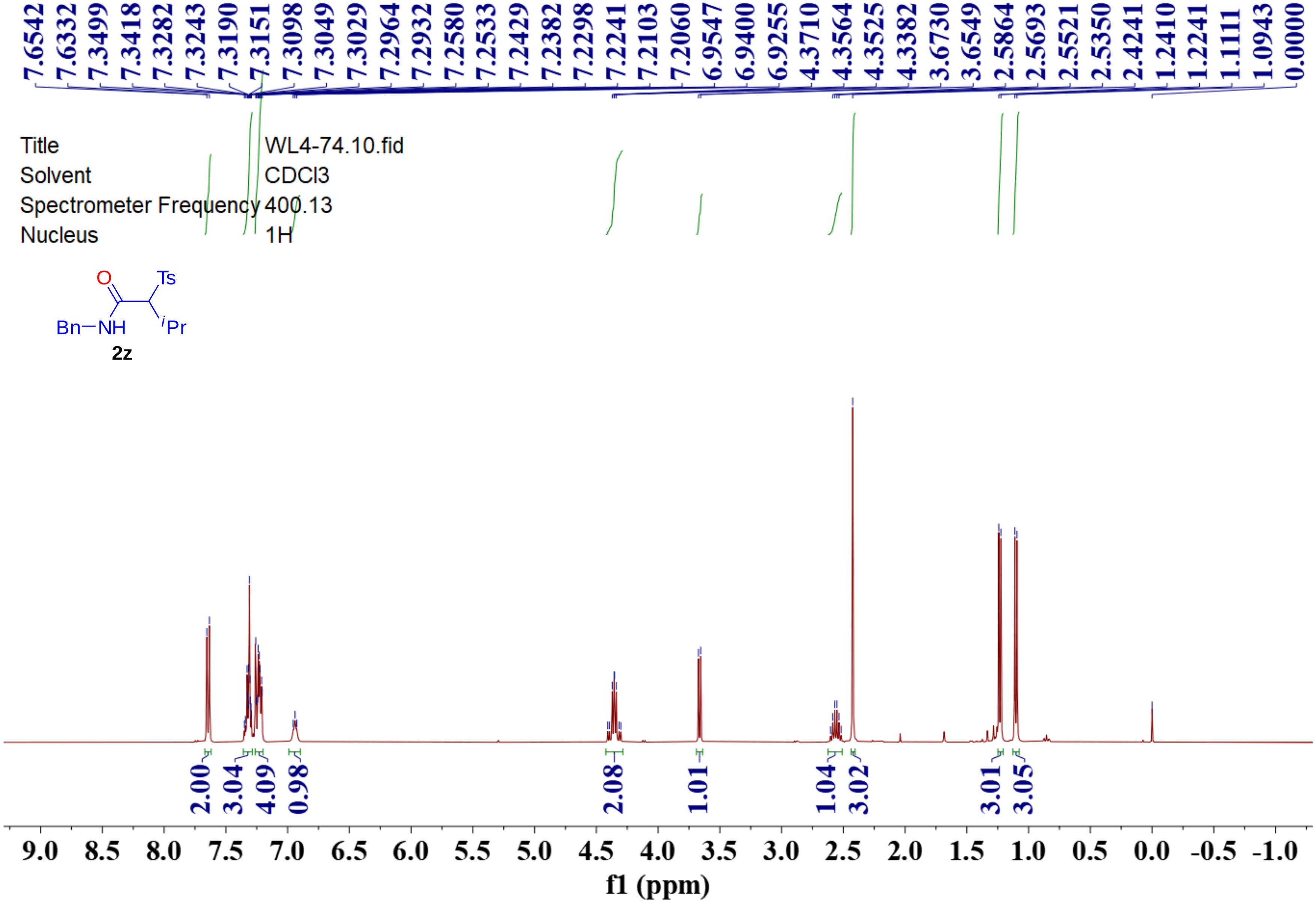
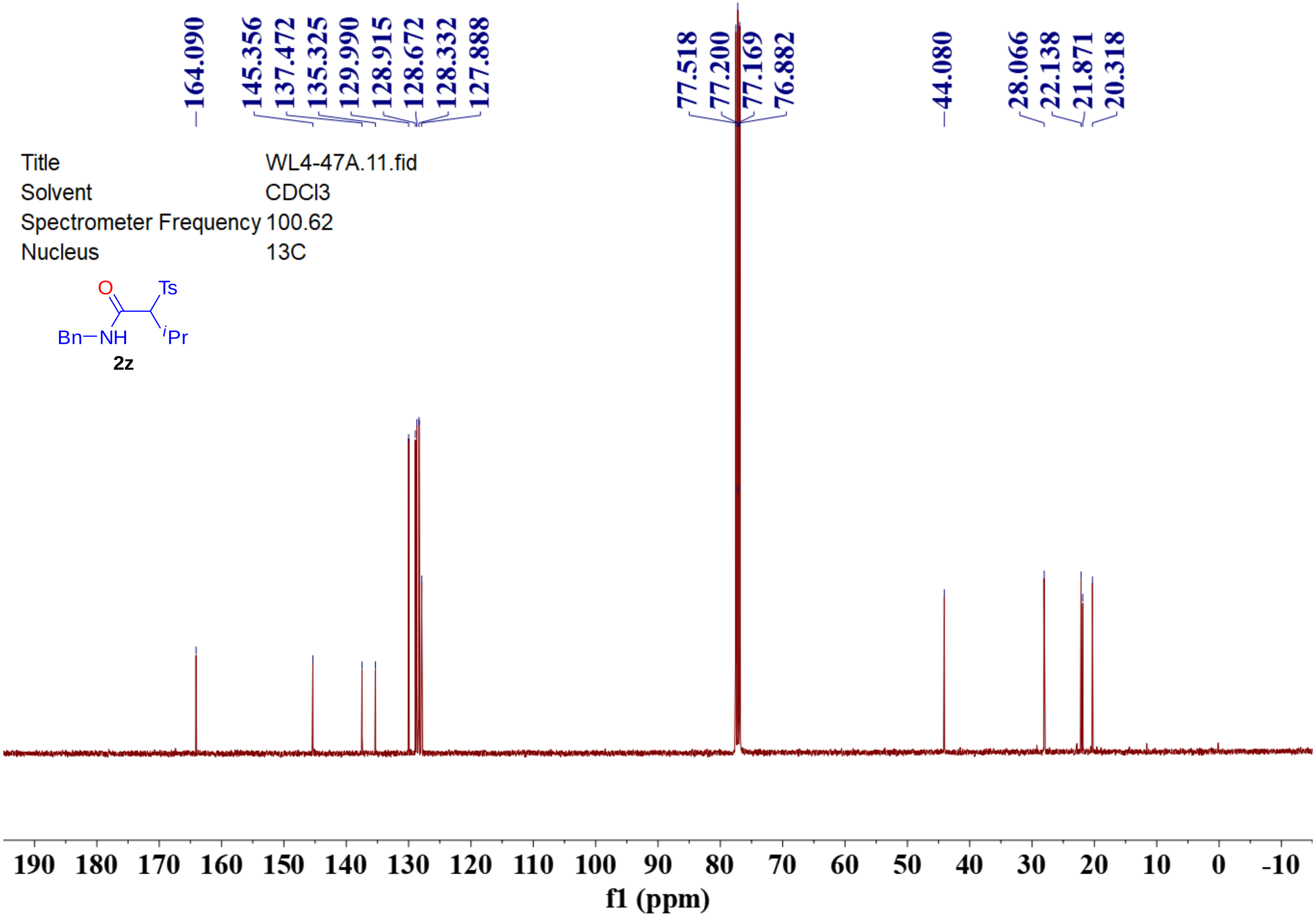
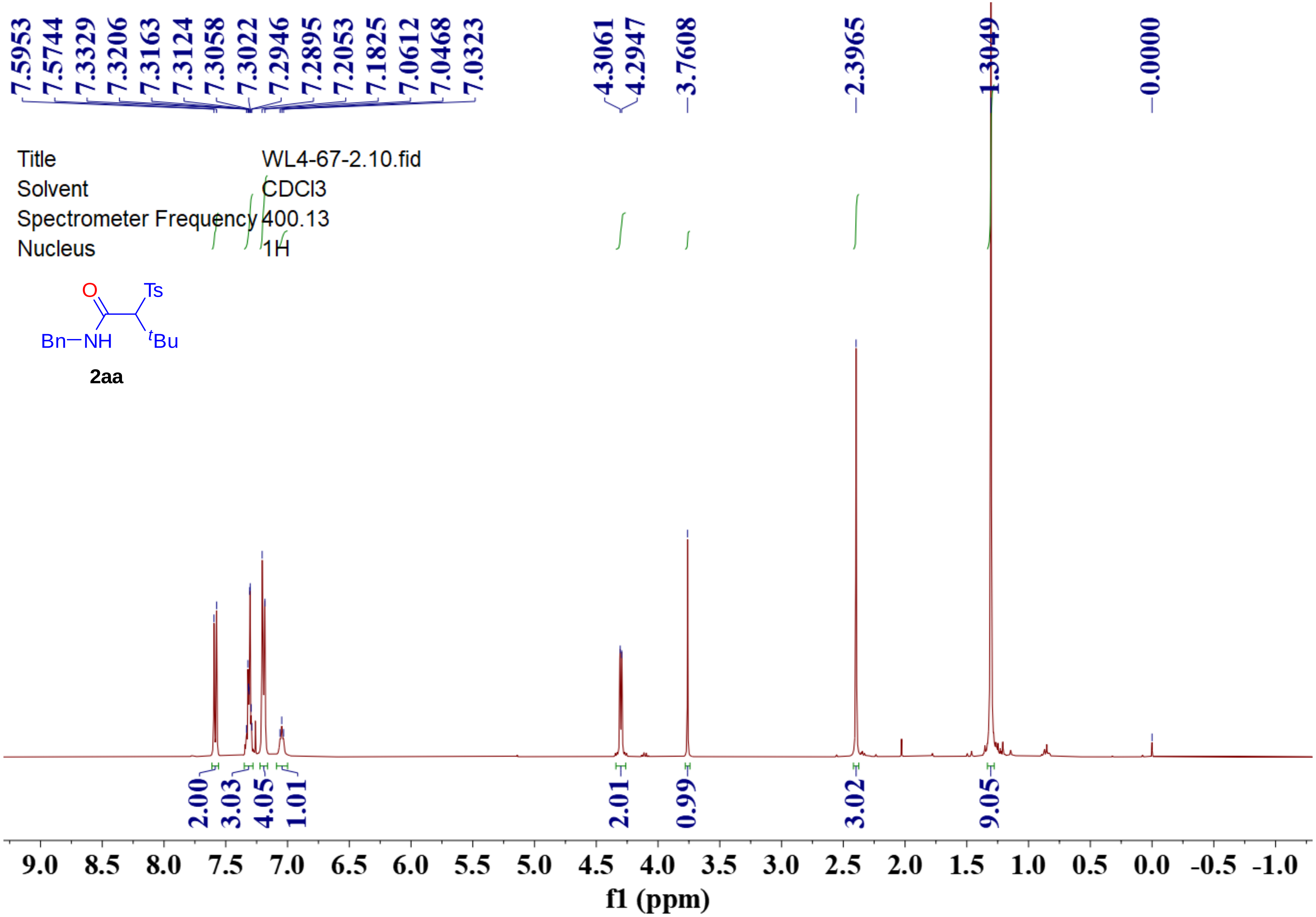
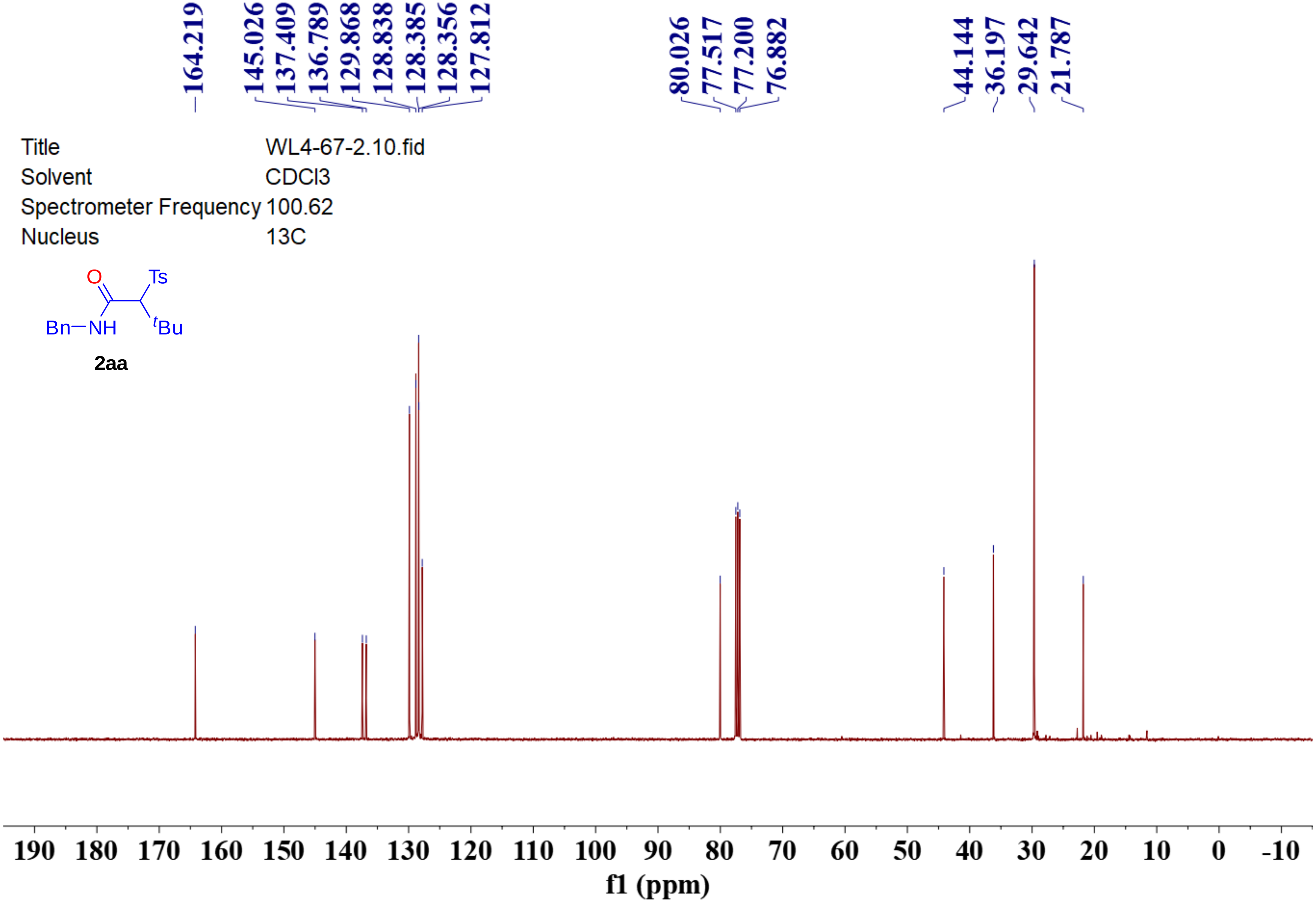
141.375
135.199
130.696
129.648
77.518
77.200
76.883
71.887
40.038
31.525
31.476
28.930
26.983
26.944
22.619
20.174
14.158
13.873
Title WL3-47.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Cs
nBu−NH
nhex
2y
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.6542
7.6332
7.3499
7.3418
7.3282
7.3243
7.3190
7.3151
7.3098
7.3049
7.3029
7.2964
7.2932
7.2580
7.2533
7.2429
7.2382
7.2298
7.2241
7.2103
7.2060
6.9547
6.9400
6.9255
4.3710
4.3564
4.3525
4.3382
3.6730
3.6549
2.5864
2.5693
2.5521
2.5350
2.4241
1.2410
1.2241
1.1111
1.0943
0.0000
Title WL4-74.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn
NH
^{i}Pr
2z
2.00
3.04
4.09
0.98
2.08
1.01
1.04
3.02
3.01
3.05
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
-0.5
-1.0
f1 (ppm)

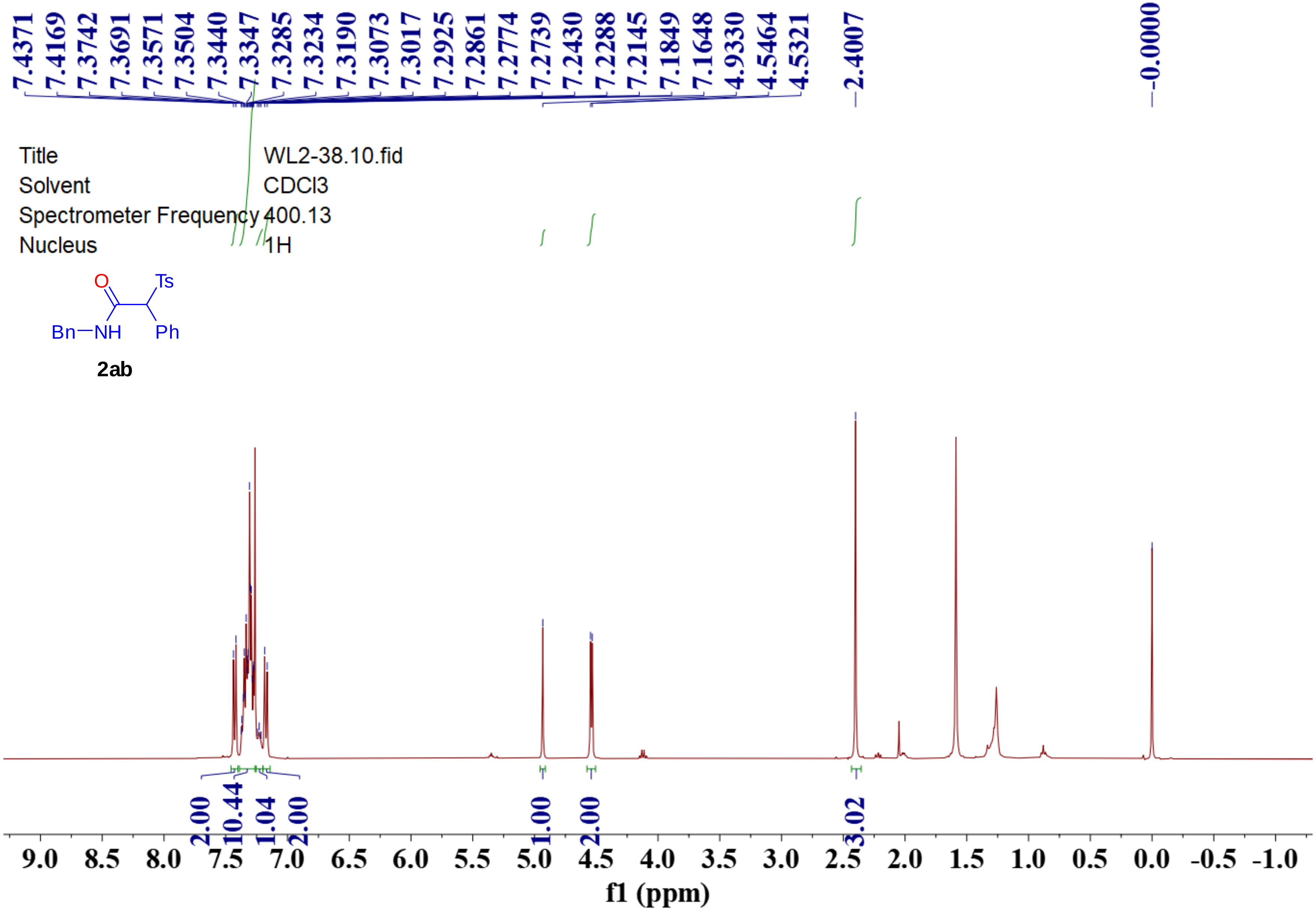

164.090
145.356
137.472
135.325
129.990
128.915
128.672
128.332
127.888
77.518
77.200
77.169
76.882
44.080
28.066
22.138
21.871
20.318
Title WL4-47A.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Bn−NH
^{i}Pr
2z
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.5953
7.5744
7.3329
7.3206
7.3163
7.3124
7.3058
7.3022
7.2946
7.2895
7.2053
7.1825
7.0612
7.0468
7.0323
4.3061
4.2947
3.7608
2.3965
1.3049
0.0000
Title WL4-67-2.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn—NH
tBu
2aa
2.00
3.03
4.05
1.01
2.01
0.99
3.02
9.05
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 -0.5 -1.0
f1 (ppm)



Title	WL4-67-2.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2aa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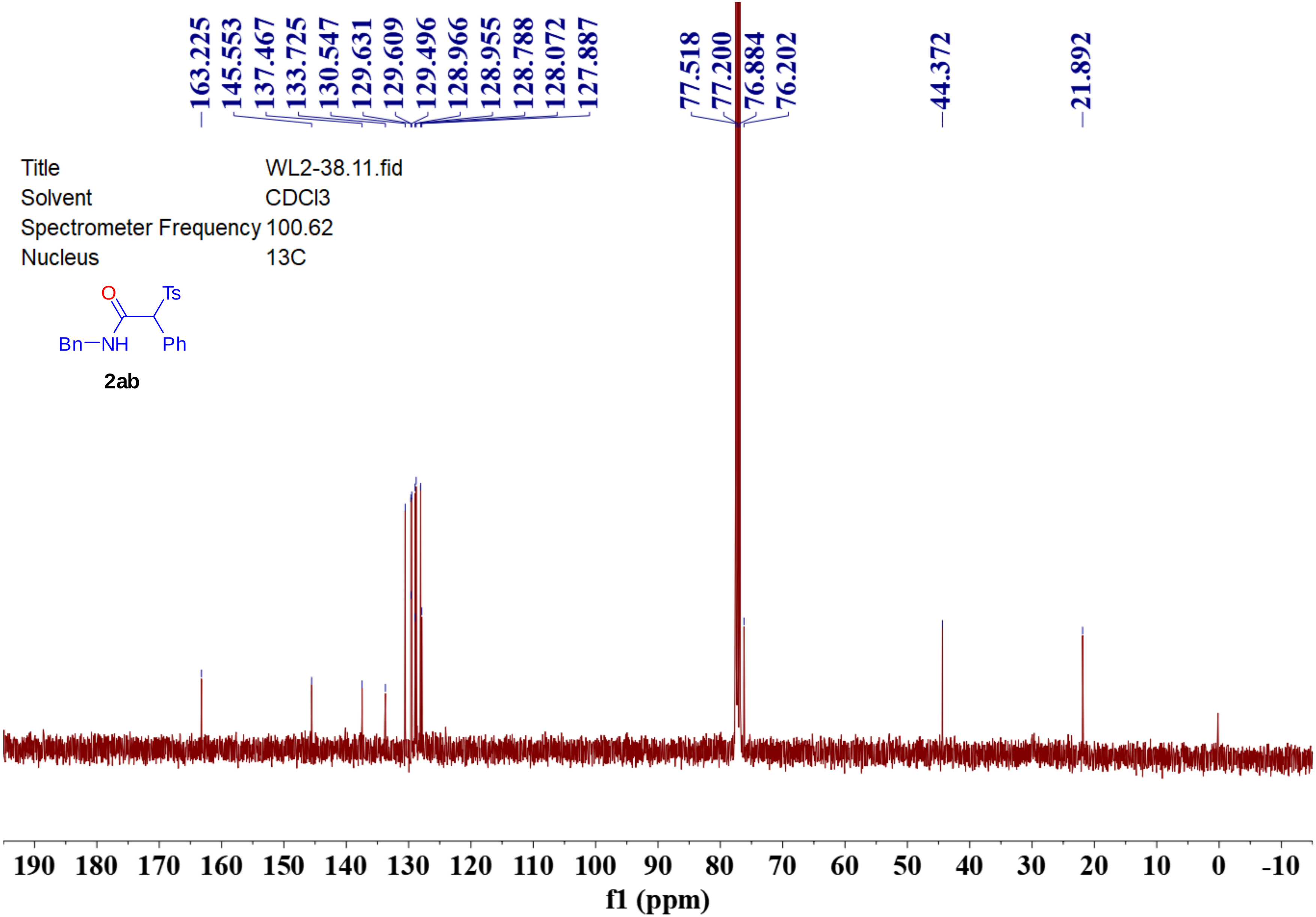
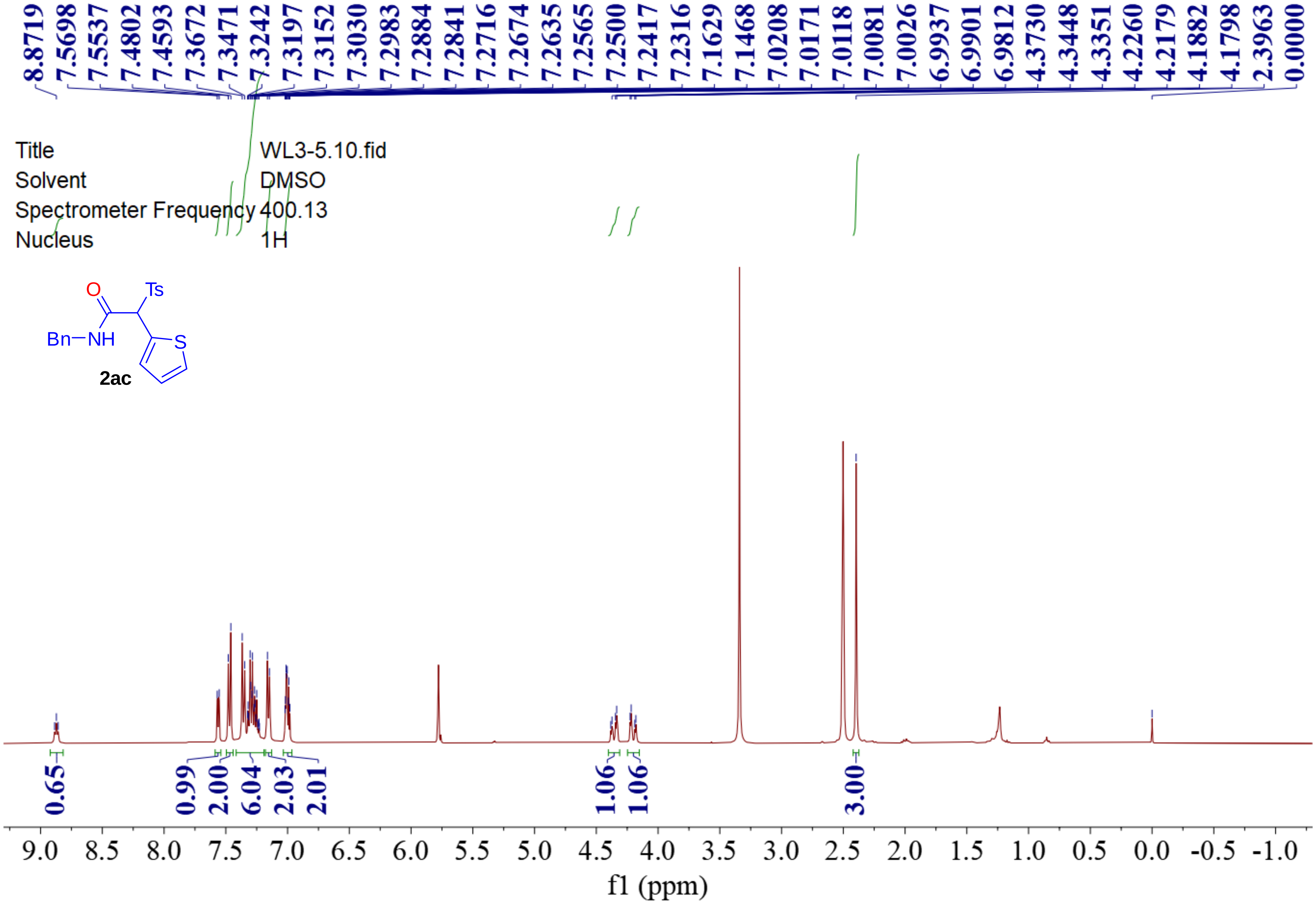
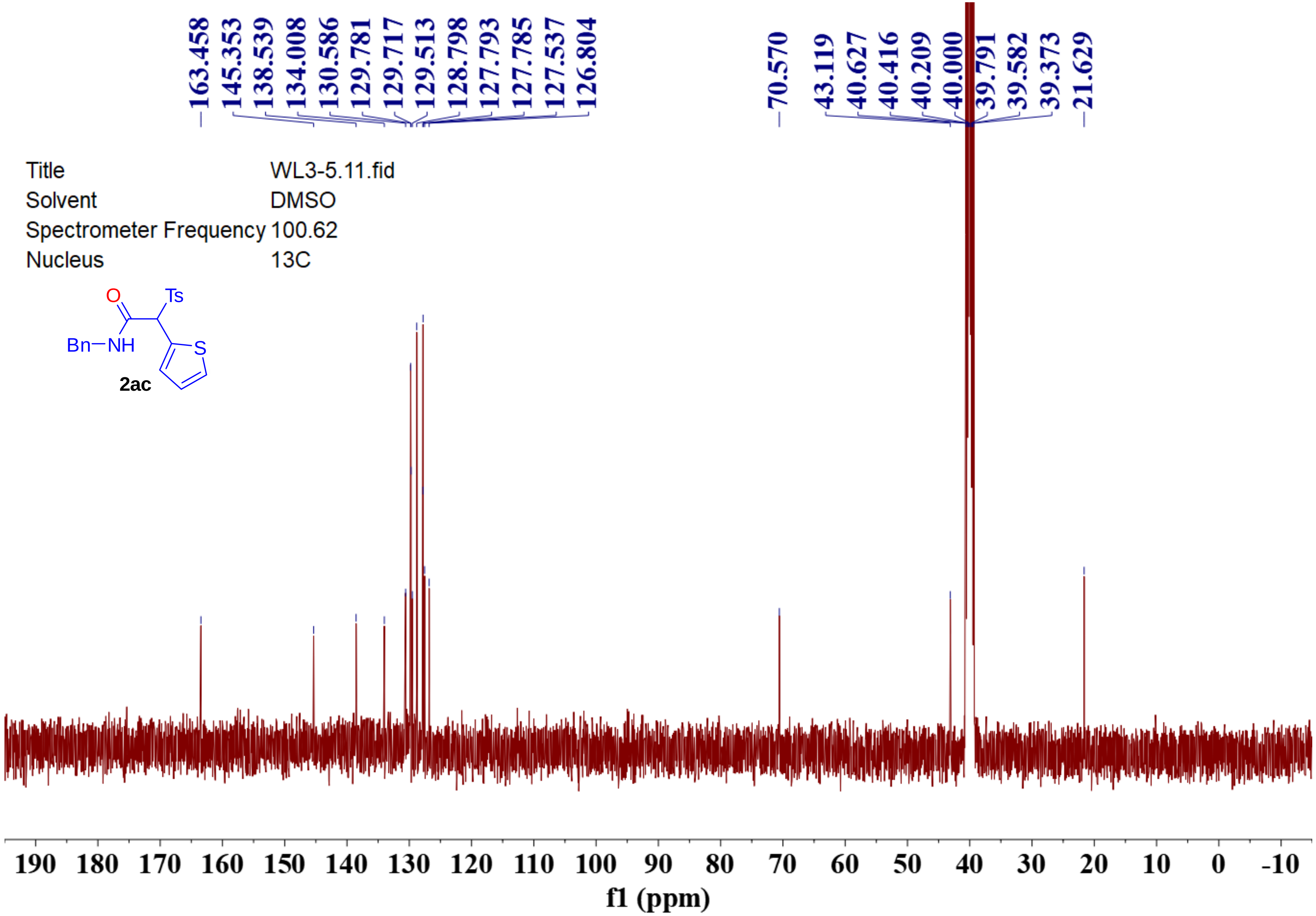
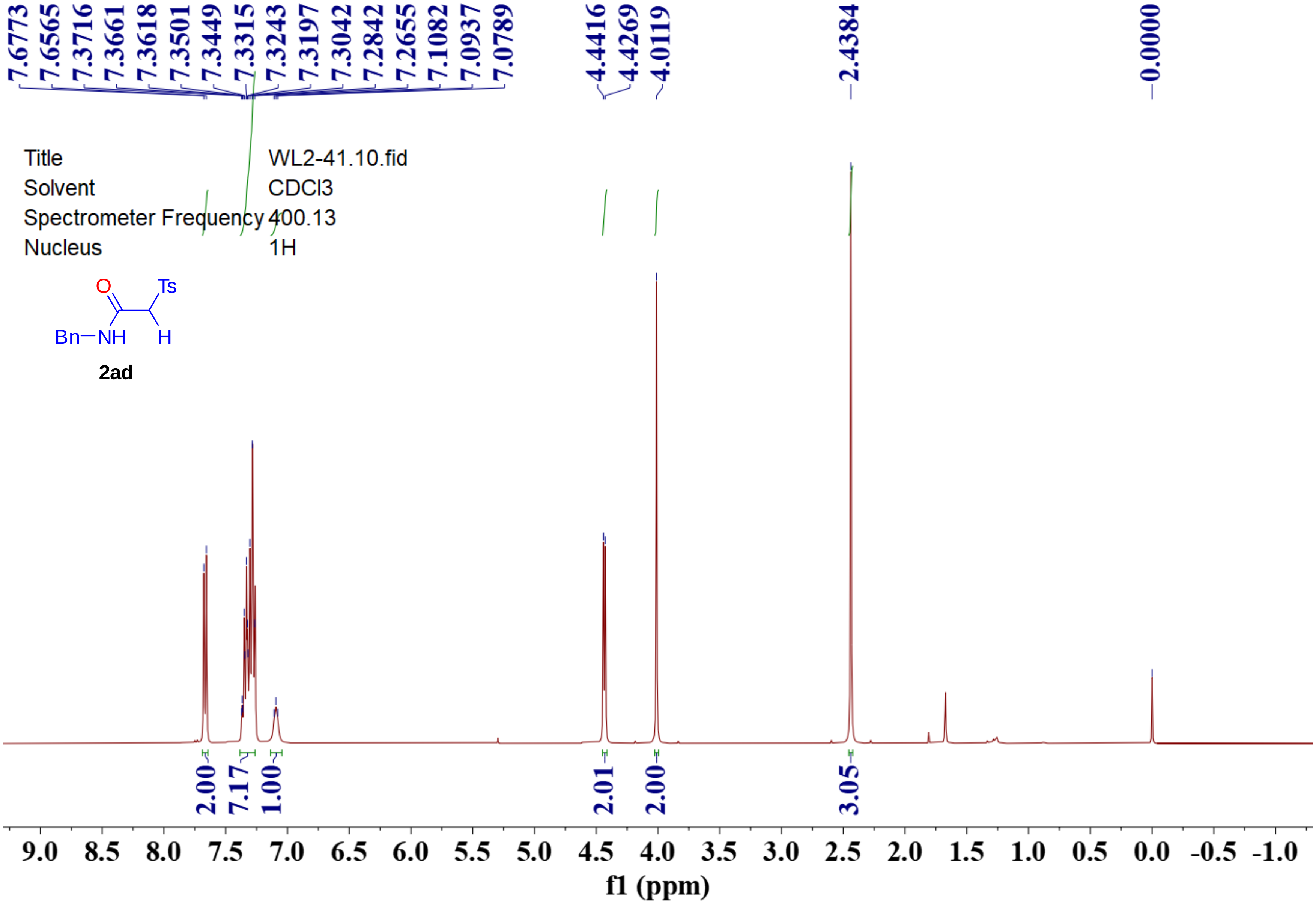
7.4169
7.3742
7.3691
7.3571
7.3504
7.3440
7.3347
7.3285
7.3234
7.3190
7.3073
7.3017
7.2925
7.2861
7.2774
7.2739
7.2430
7.2288
7.2145
7.1849
7.1648
4.9330
4.5464
4.5321
2.4007
-0.0000
Title WL2-38.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn—NH
Ph
2ab
2.00
10.44
1.04
2.00
1.00
2.00
3.02
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
-0.5
-1.0
f1 (ppm)

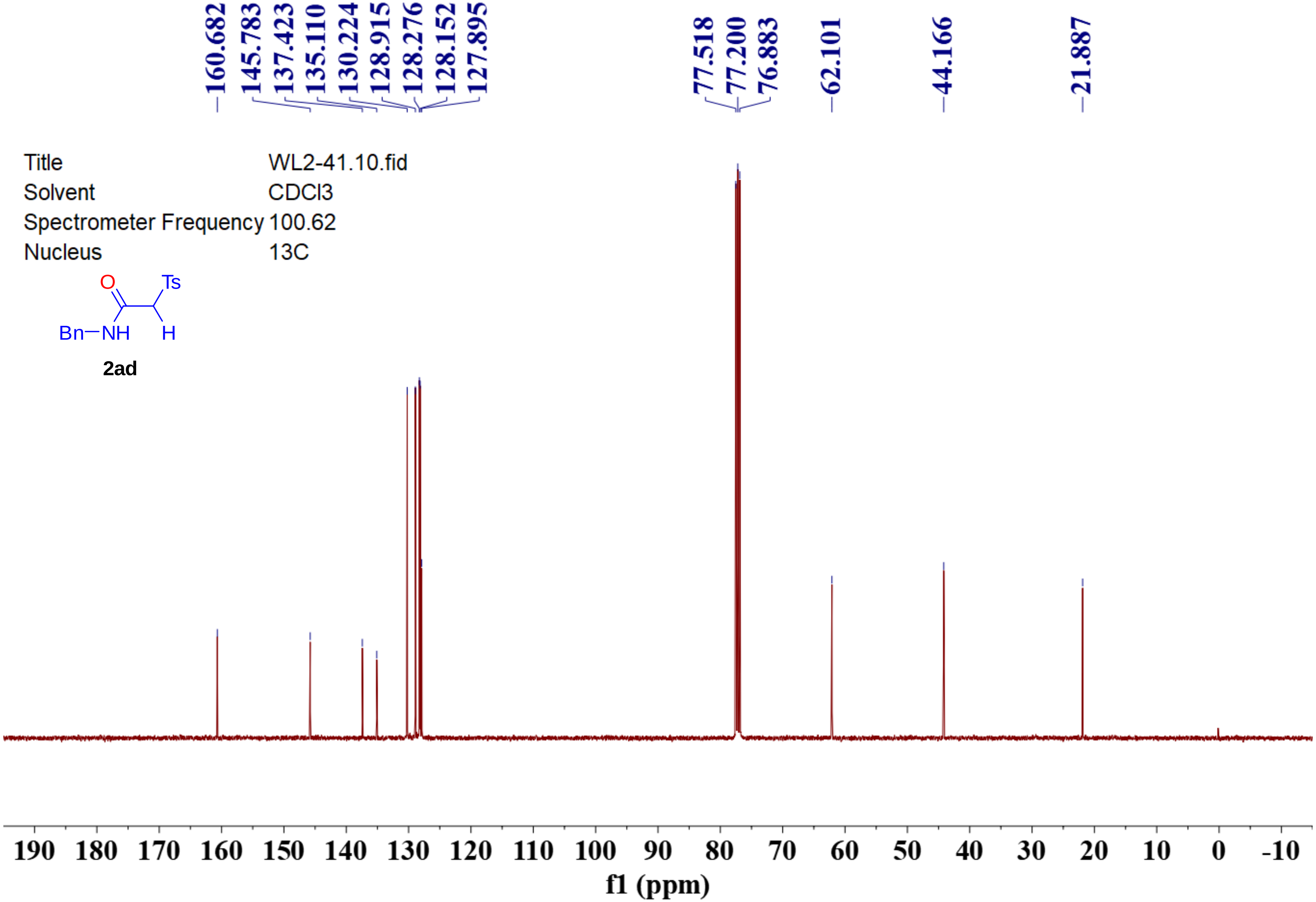

163.225
145.553
137.467
133.725
130.547
129.631
129.609
129.496
128.966
128.955
128.788
128.072
127.887
77.518
77.200
76.884
76.202
44.372
21.892
Title WL2-38.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Bn
NH
Ph
2ab
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.8719
7.5698
7.5537
7.4802
7.4593
7.3672
7.3471
7.3242
7.3197
7.3152
7.3030
7.2983
7.2884
7.2841
7.2716
7.2674
7.2635
7.2565
7.2500
7.2417
7.2316
7.1629
7.1468
7.0208
7.0171
7.0118
7.0081
7.0026
6.9937
6.9901
6.9812
4.3730
4.3448
4.3351
4.2260
4.2179
4.1882
4.1798
2.3963
0.0000
Title WL3-5.10.fid
Solvent DMSO
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn−NH
S
2ac
0.65
0.99
2.00
6.04
2.03
2.01
1.06
1.06
3.00
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
-0.5
-1.0
f1 (ppm)


Title WL3-5.11.fid
Solvent DMSO
Spectrometer Frequency 100.62
Nucleus 13C

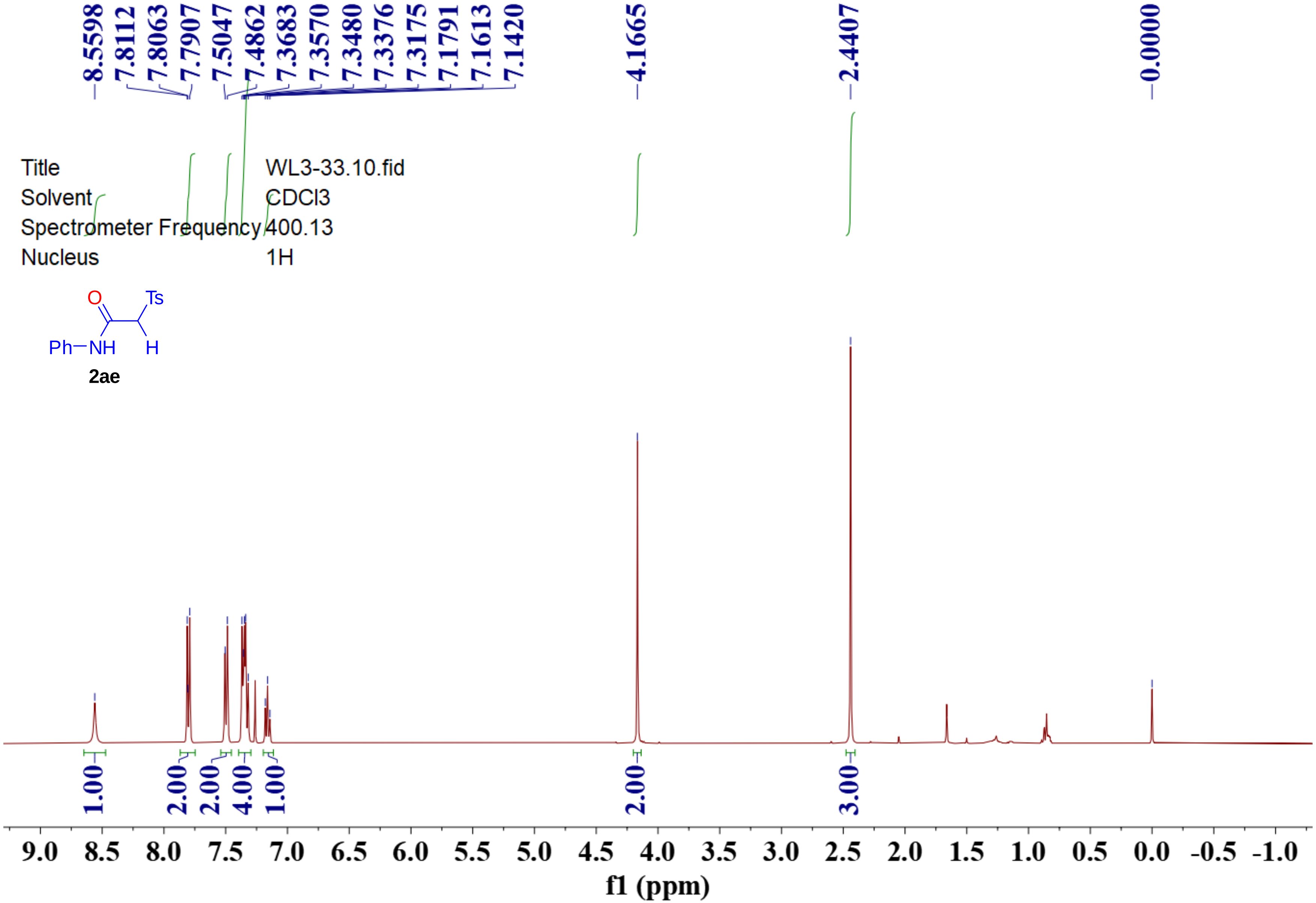
2ac

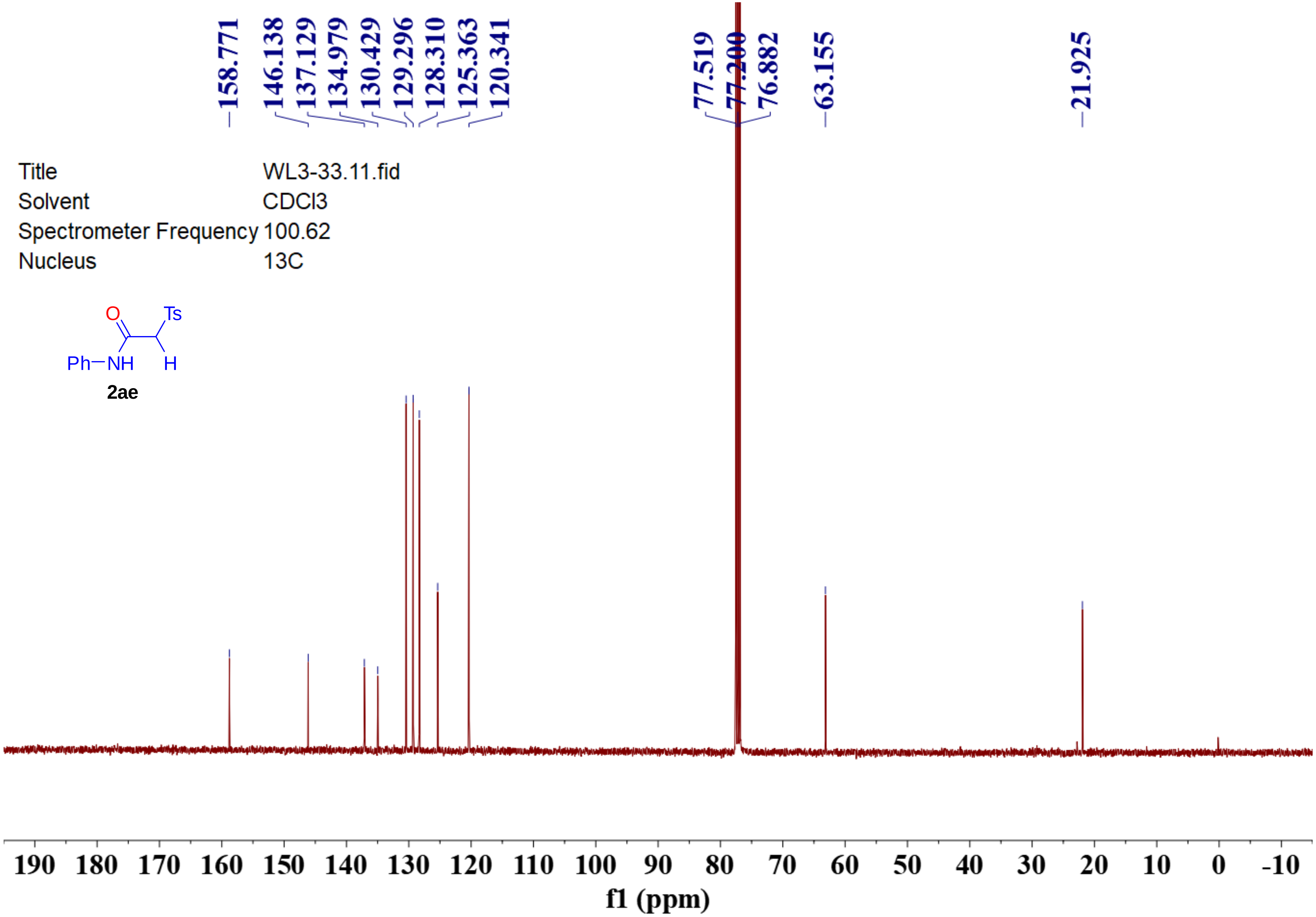

7.6773
7.6565
7.3716
7.3661
7.3618
7.3501
7.3449
7.3315
7.3243
7.3197
7.3042
7.2842
7.2655
7.1082
7.0937
7.0789
4.4416
4.4269
4.0119
2.4384
-0.0000
Title WL2-41.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Bn—NH
H
2ad
2.00
7.17
1.00
2.01
2.00
3.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)



Title	WL2-41.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

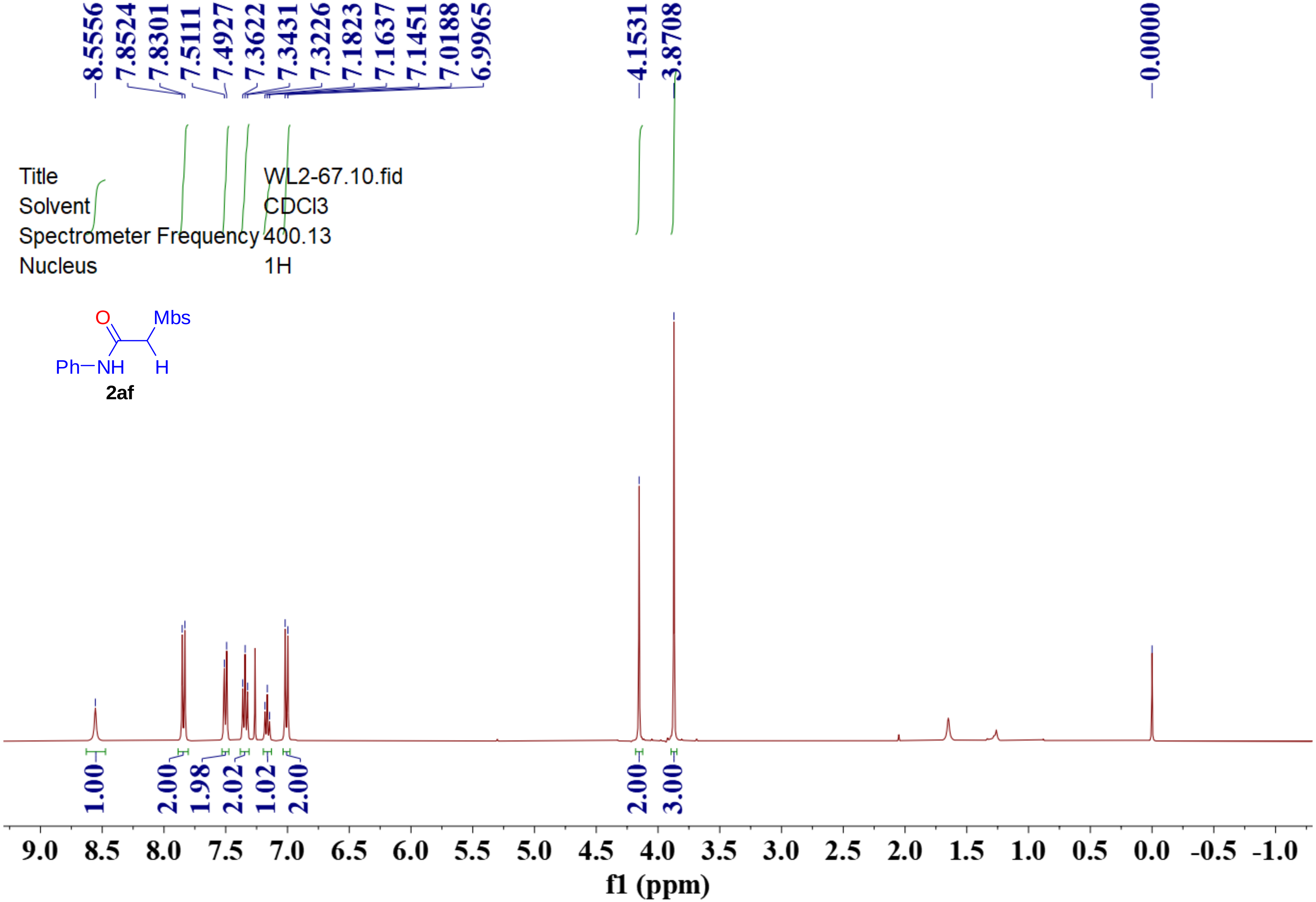
2ad

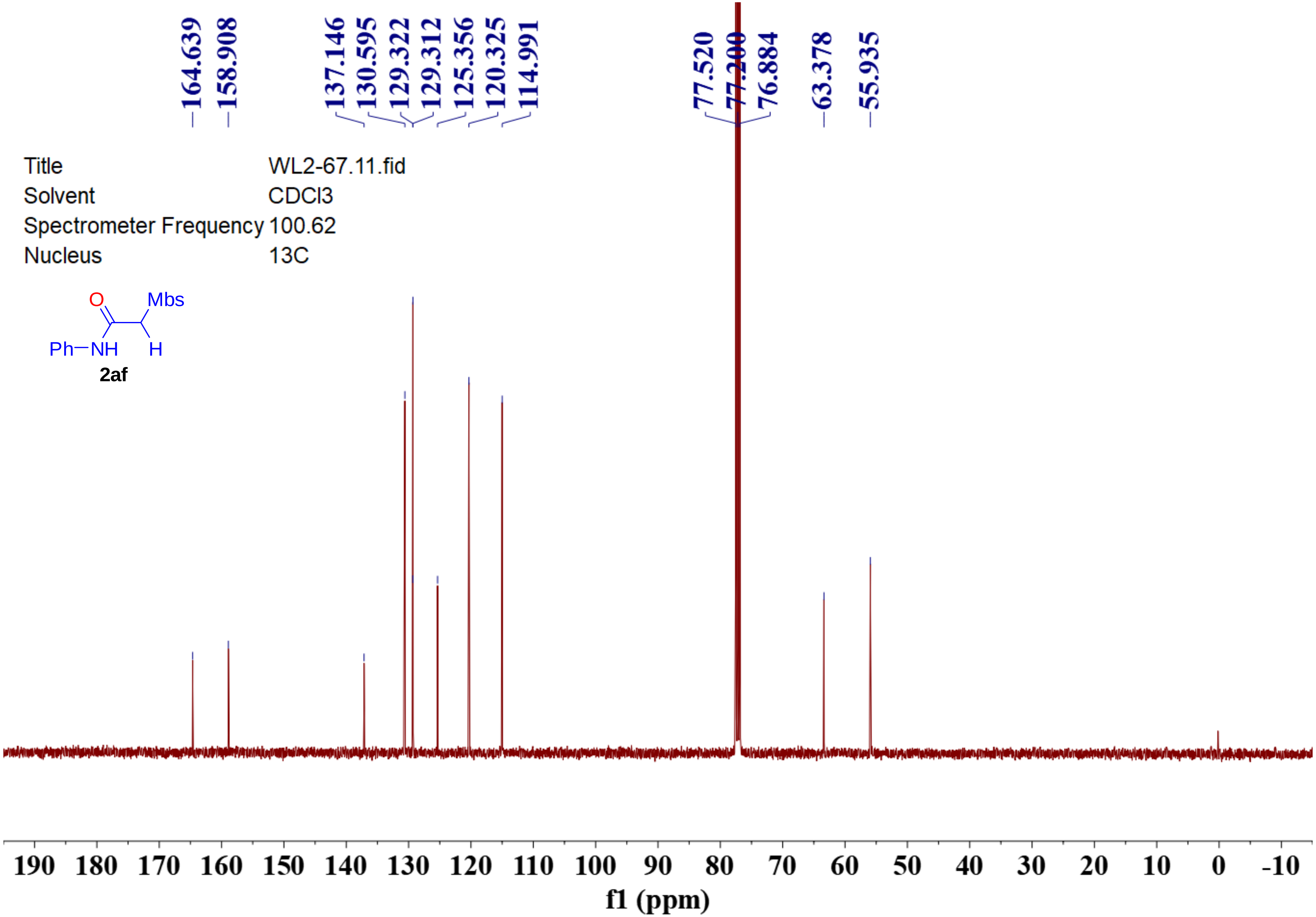

8.5598
7.8112
7.8063
7.7907
7.5047
7.4862
7.3683
7.3570
7.3480
7.3376
7.3175
7.1791
7.1613
7.1420
4.1665
2.4407
0.0000
Title WL3-33.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Ph
NH
H
2ae
1.00
2.00
2.00
4.00
1.00
2.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

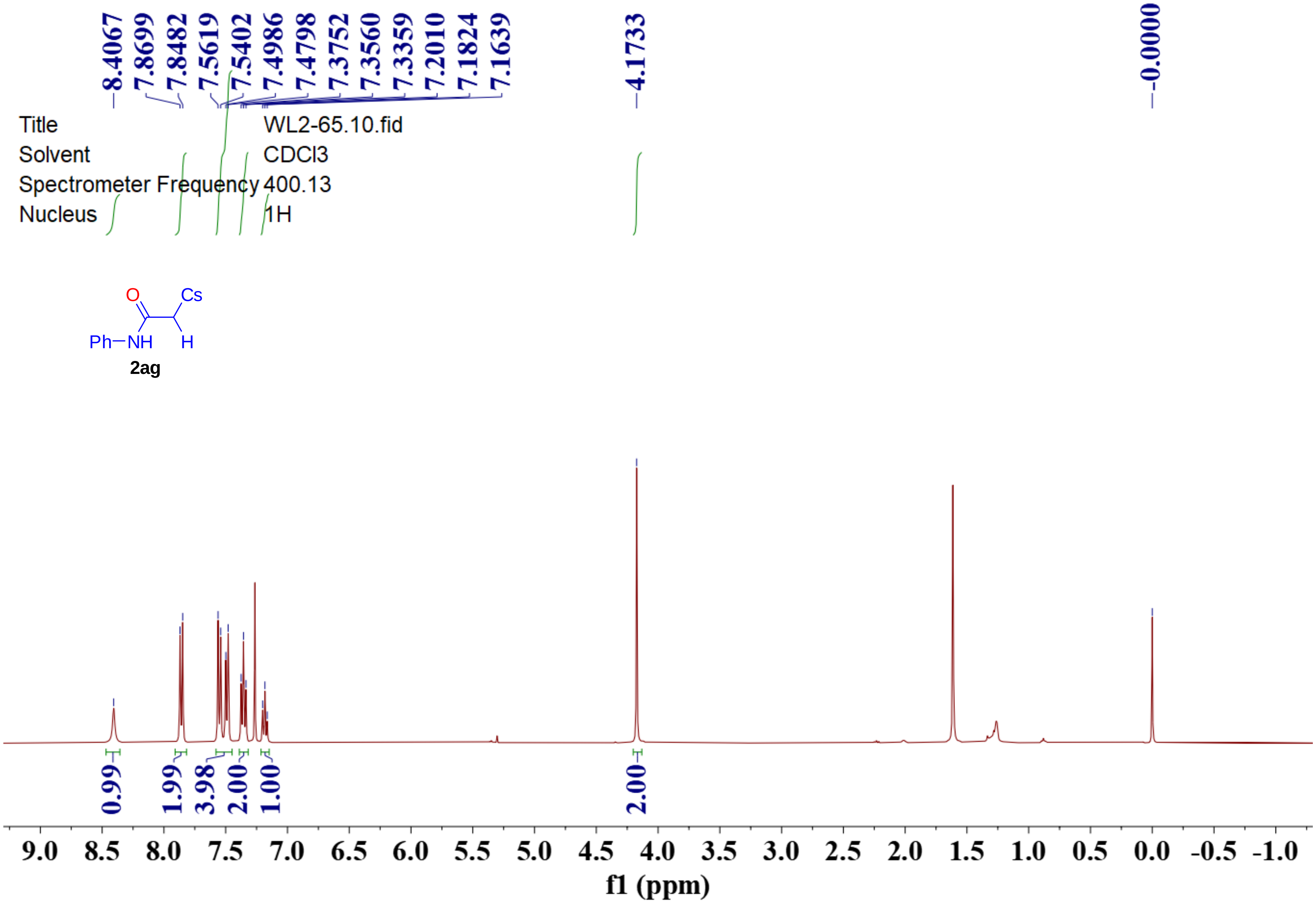


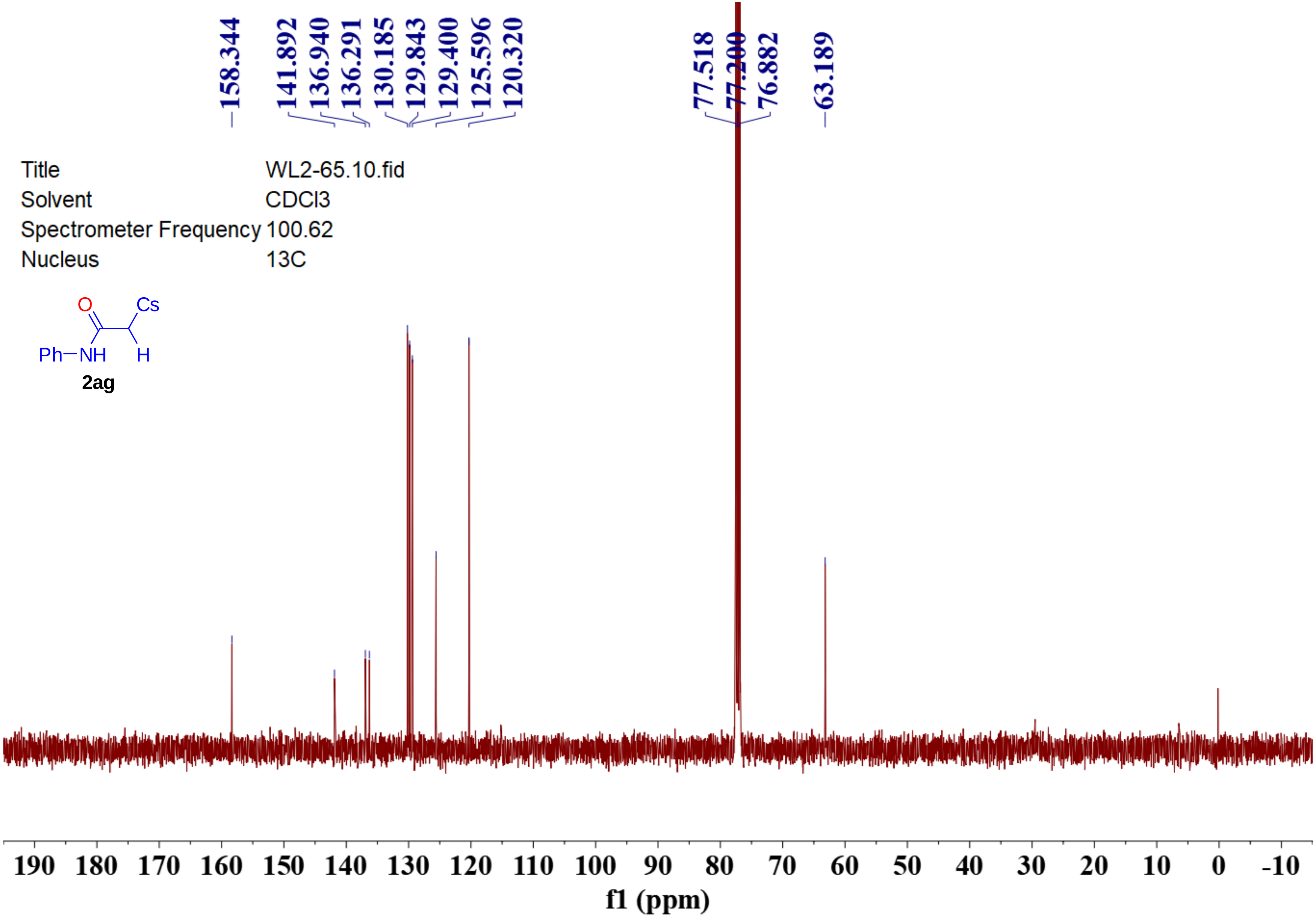
Title	WL3-33.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2ae


8.5556
7.8524
7.8301
7.5111
7.4927
7.3622
7.3431
7.3226
7.1823
7.1637
7.1451
7.0188
6.9965
4.1531
3.8708
0.0000
Title WL2-67.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Mbs
Ph
NH
H
2af
1.00
2.00
1.98
2.02
1.02
2.00
2.00
3.00
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
-0.5
-1.0
f1 (ppm)

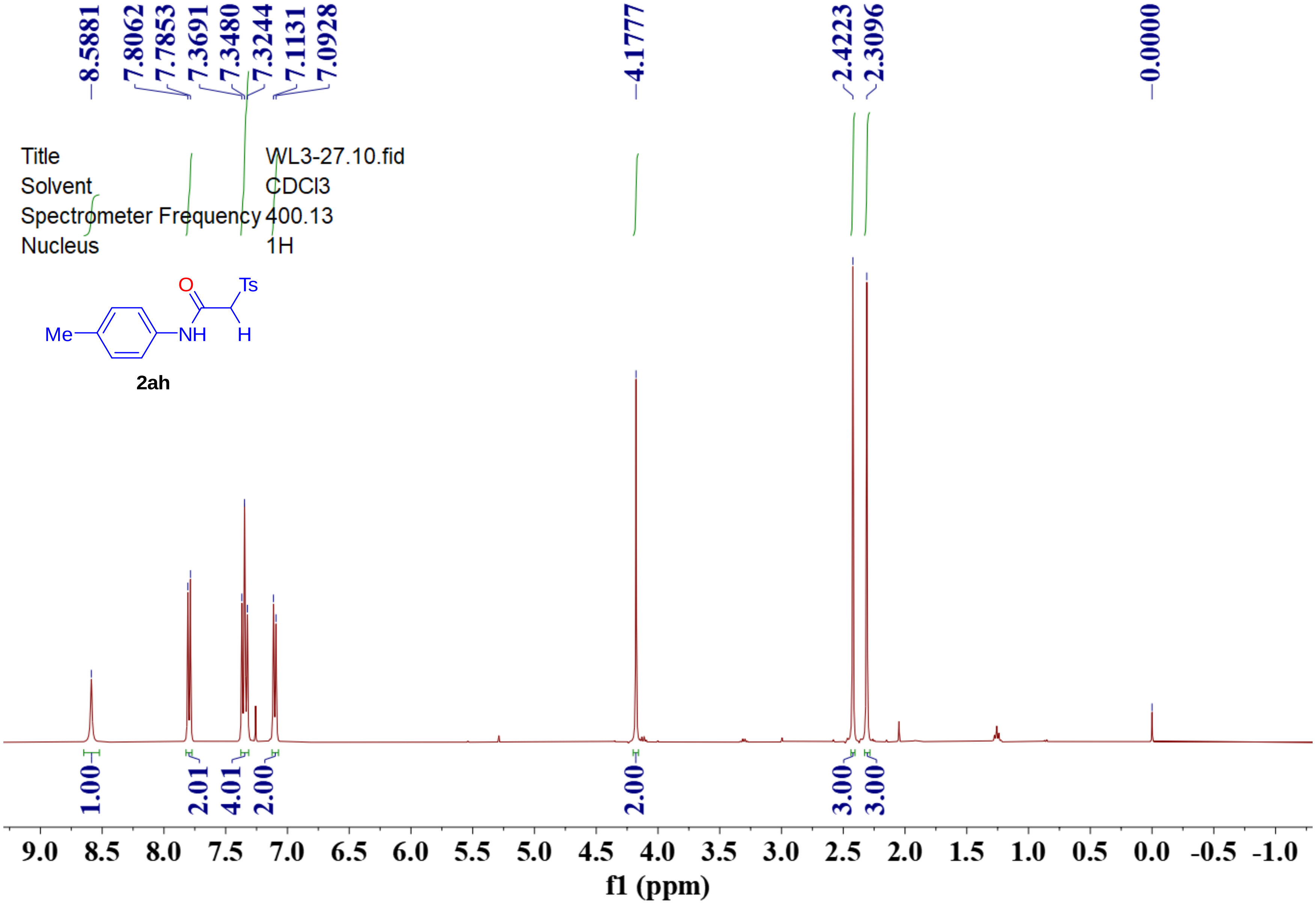

164.639
158.908
137.146
130.595
129.322
129.312
125.356
120.325
114.991
77.520
77.200
76.884
63.378
55.935
Title WL2-67.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Mbs
Ph—NH
H
2af
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

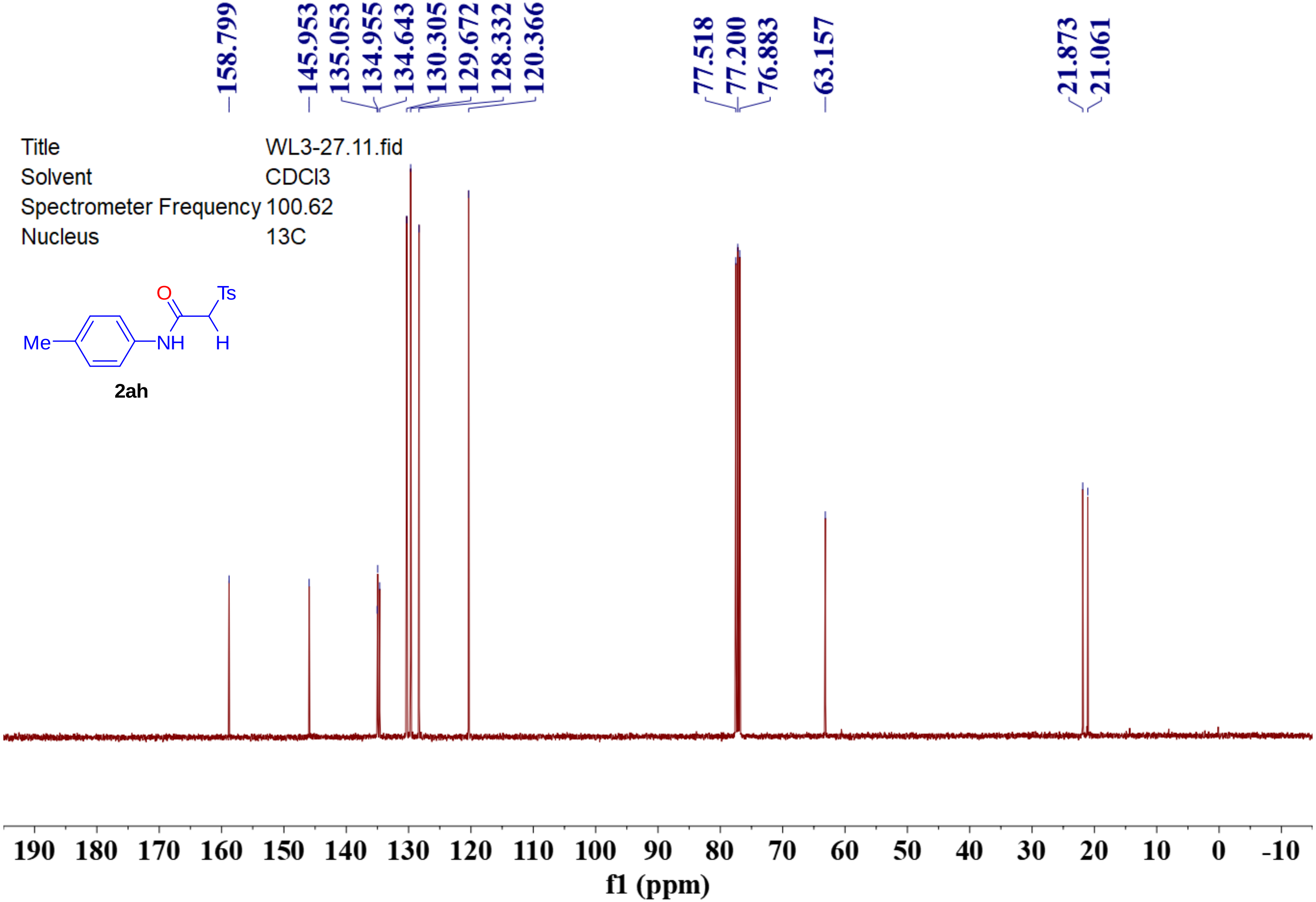

8.4067
7.8699
7.8482
7.5619
7.5402
7.4986
7.4798
7.3752
7.3560
7.3359
7.2010
7.1824
7.1639
4.1733
-0.0000
Title WL2-65.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Cs
Ph
NH
H
2ag
0.99
1.99
3.98
2.00
1.00
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

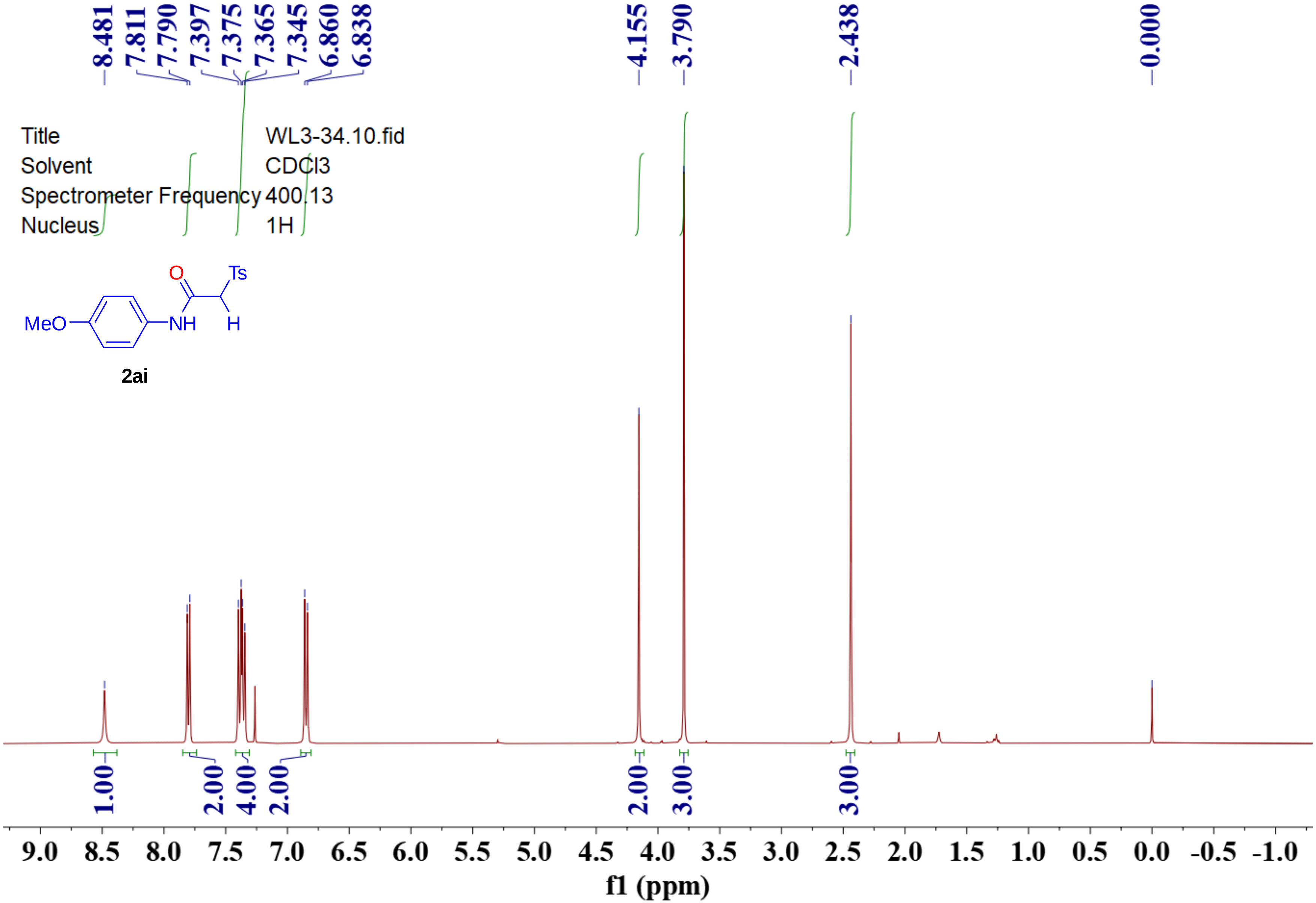


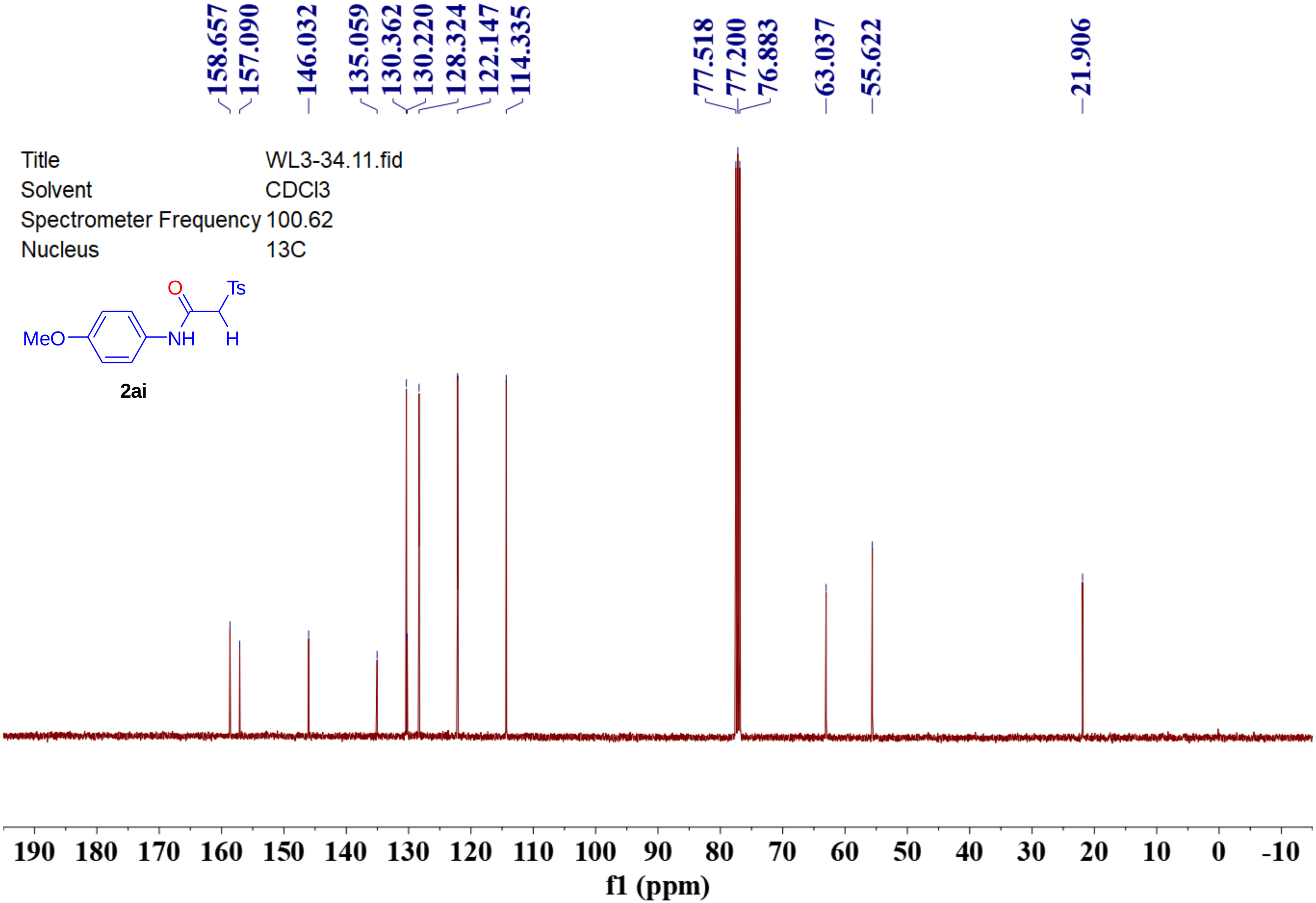
Title	WL2-65.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2ag

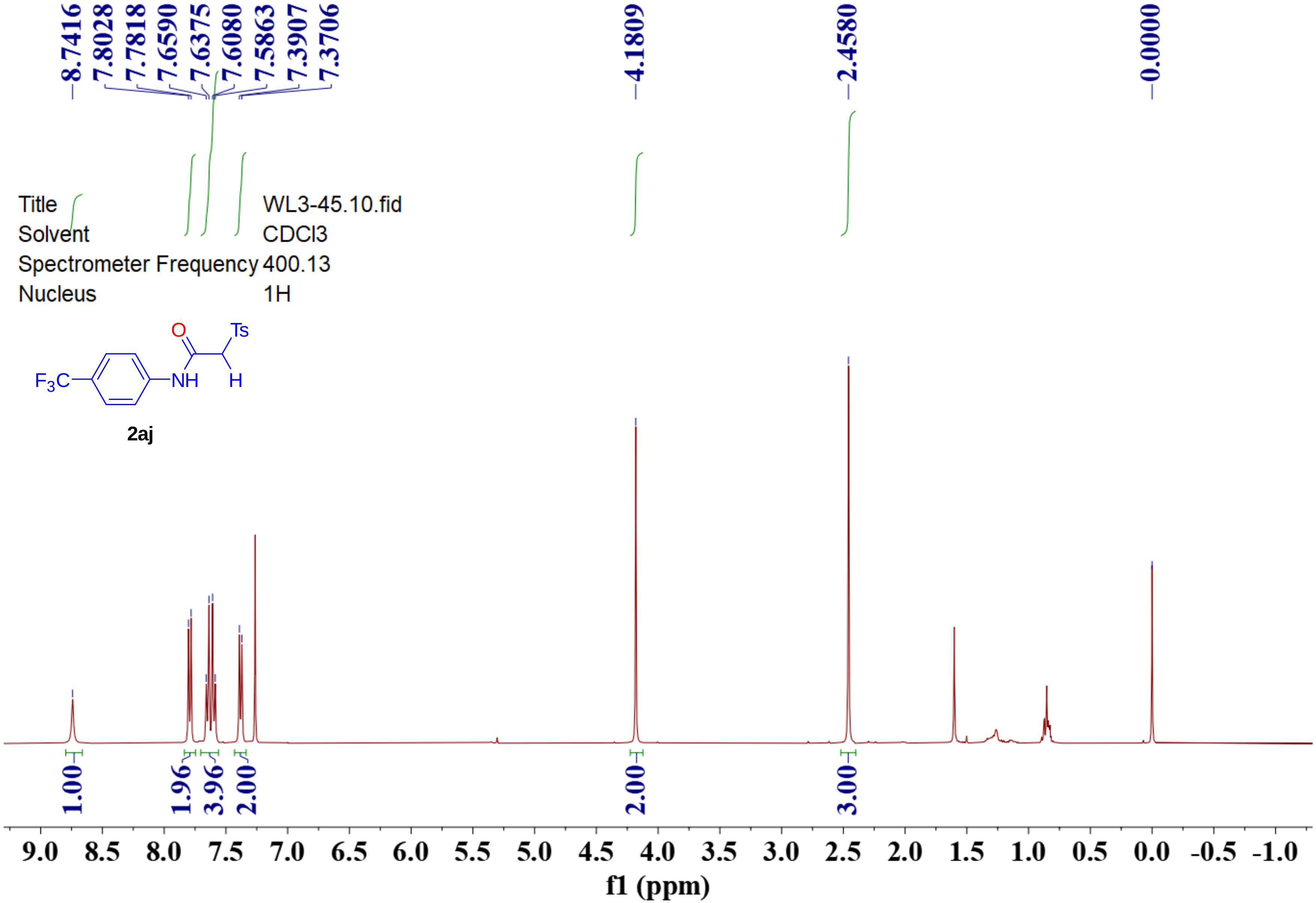

8.5881
7.8062
7.7853
7.3691
7.3480
7.3244
7.1131
7.0928
4.1777
2.4223
2.3096
0.0000
Title WL3-27.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
Me
NH
H
2ah
1.00
2.01
4.01
2.00
2.00
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

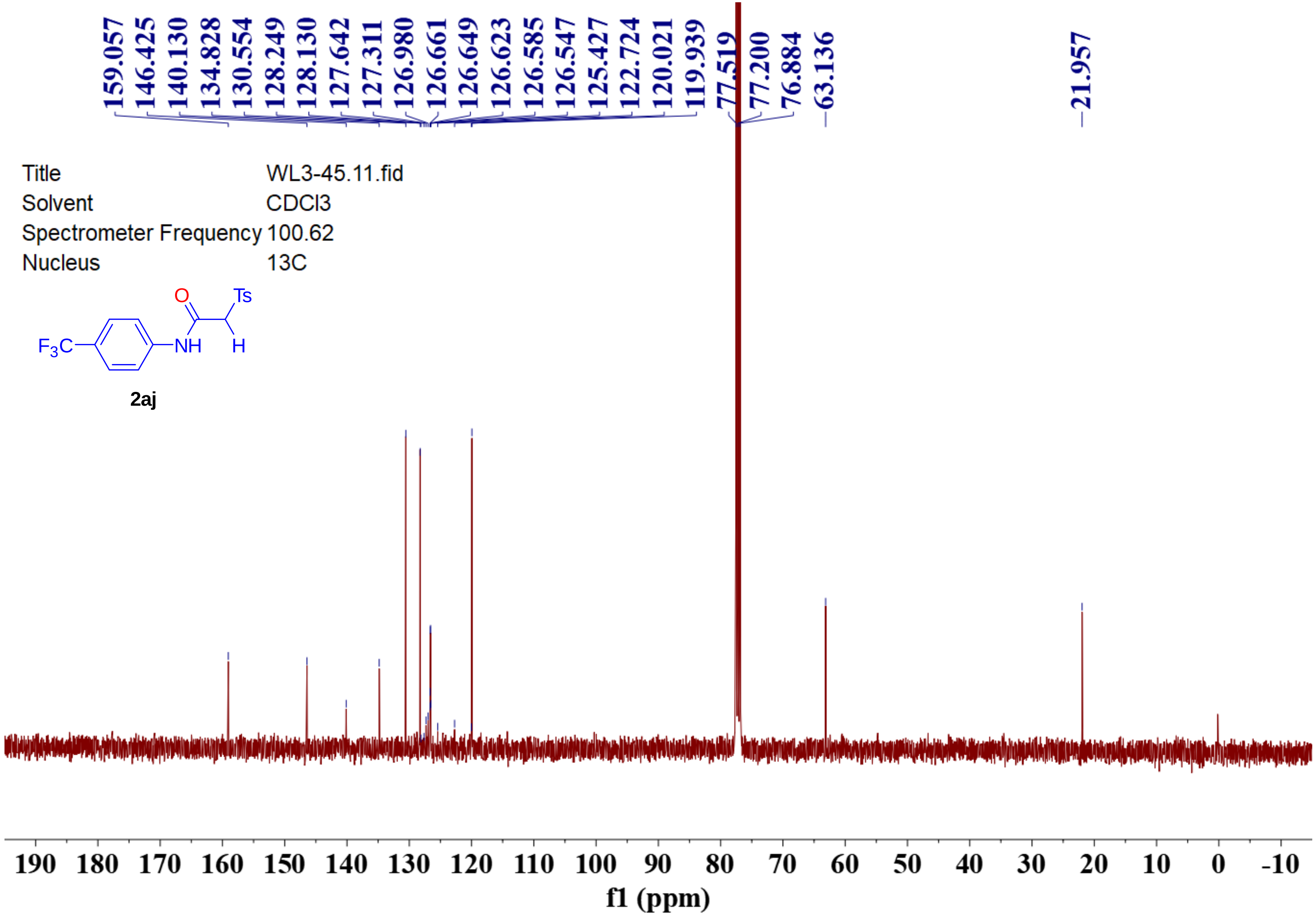

158.799
145.953
135.053
134.955
134.643
130.305
129.672
128.332
120.366
77.518
77.200
76.883
63.157
21.873
21.061
Title WL3-27.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
Me
NH
H
2ah
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

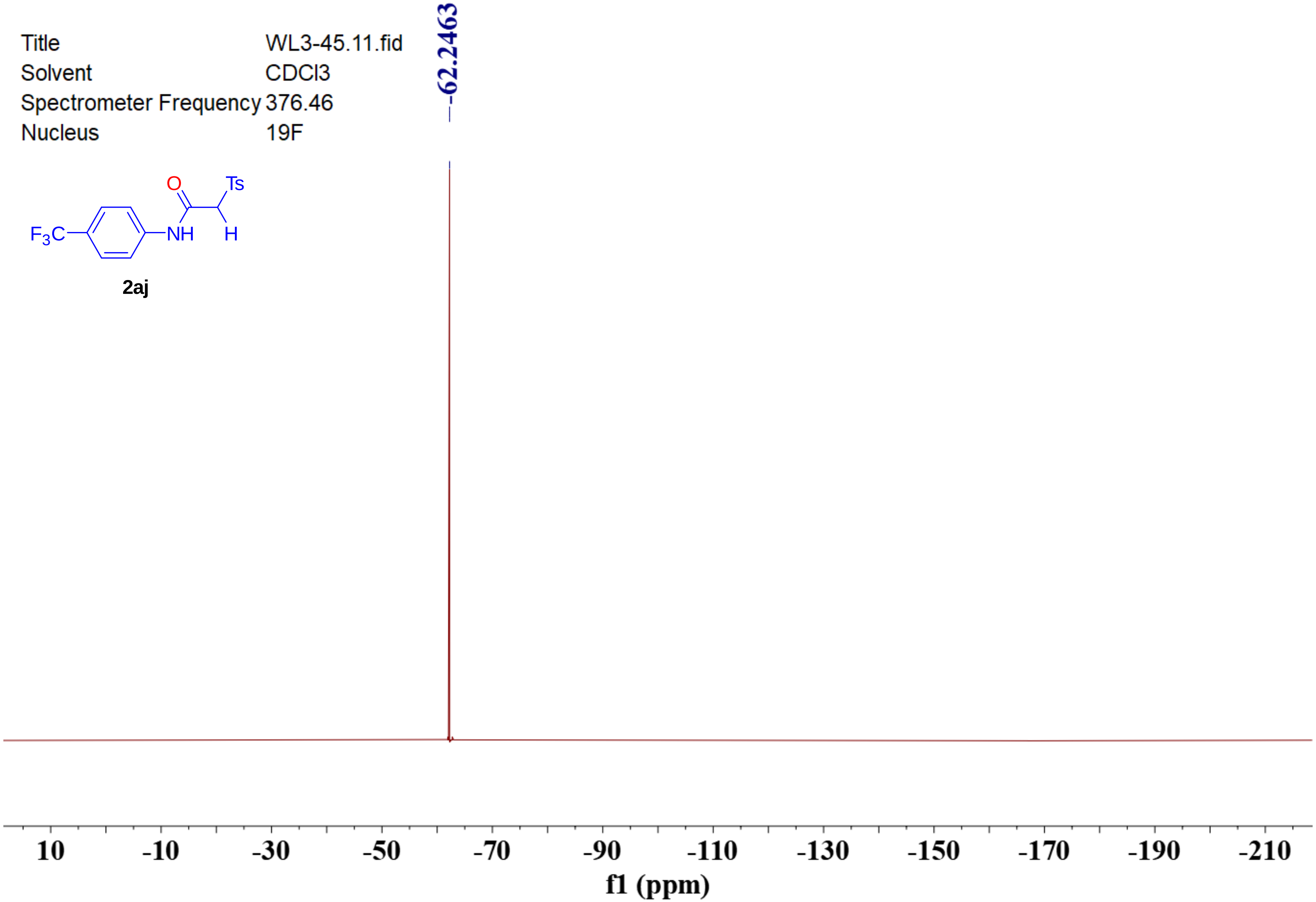

-8.481
7.811
7.790
7.397
7.375
7.365
7.345
6.860
6.838
-4.155
-3.790
-2.438
-0.000
Title WL3-34.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
MeO
NH
O
Ts
H
2ai
1.00
2.00
4.00
2.00
2.00
3.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


158.657
157.090
146.032
135.059
130.362
130.220
128.324
122.147
114.335
77.518
77.200
76.883
63.037
55.622
21.906
Title WL3-34.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
MeO
NH
O
Ts
H
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2ai

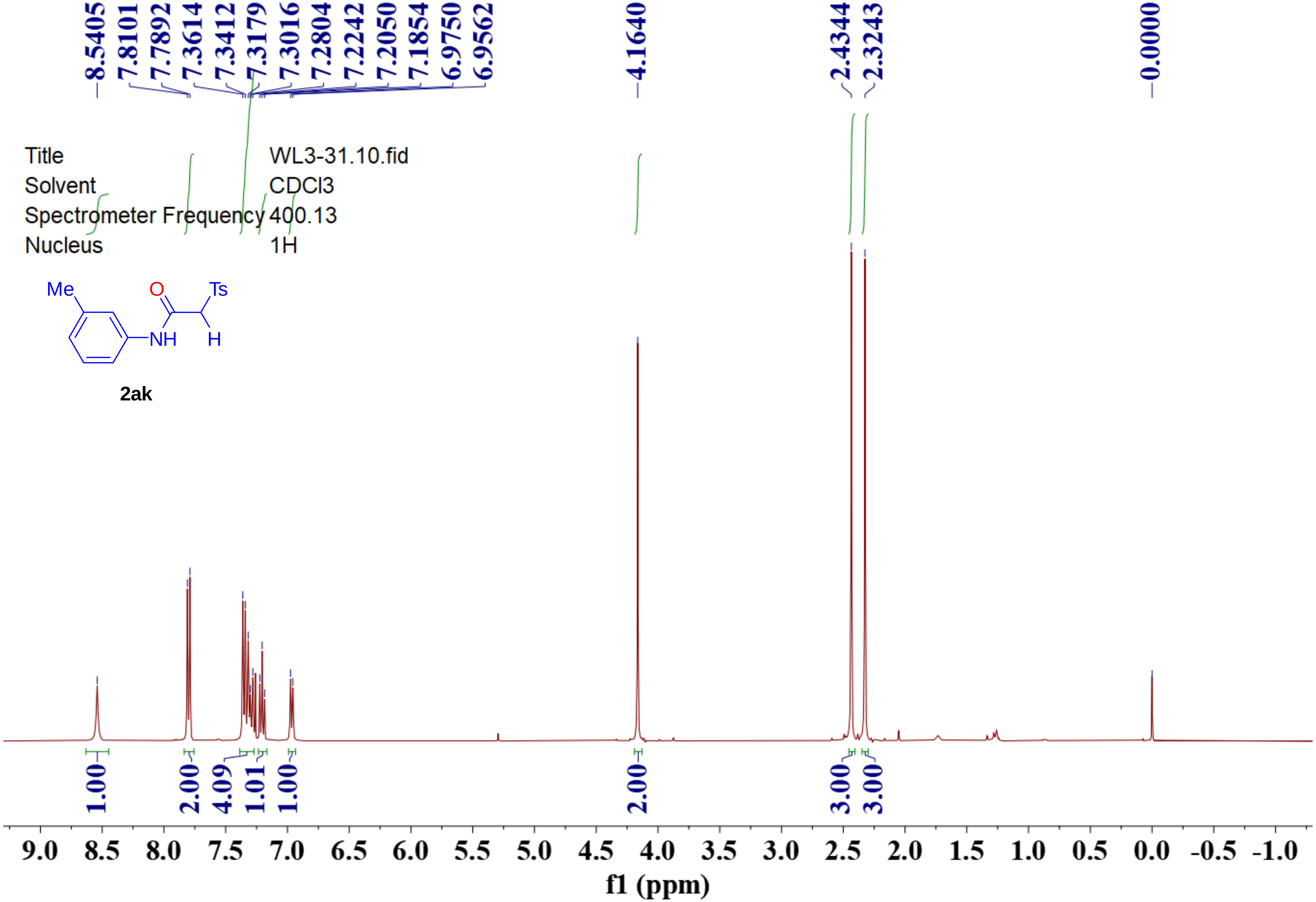

8.7416
7.8028
7.7818
7.6590
7.6375
7.6080
7.5863
7.3907
7.3706
4.1809
2.4580
0.0000
Title WL3-45.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
F3C
NH
H
2aj
1.00
1.96
3.96
2.00
2.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

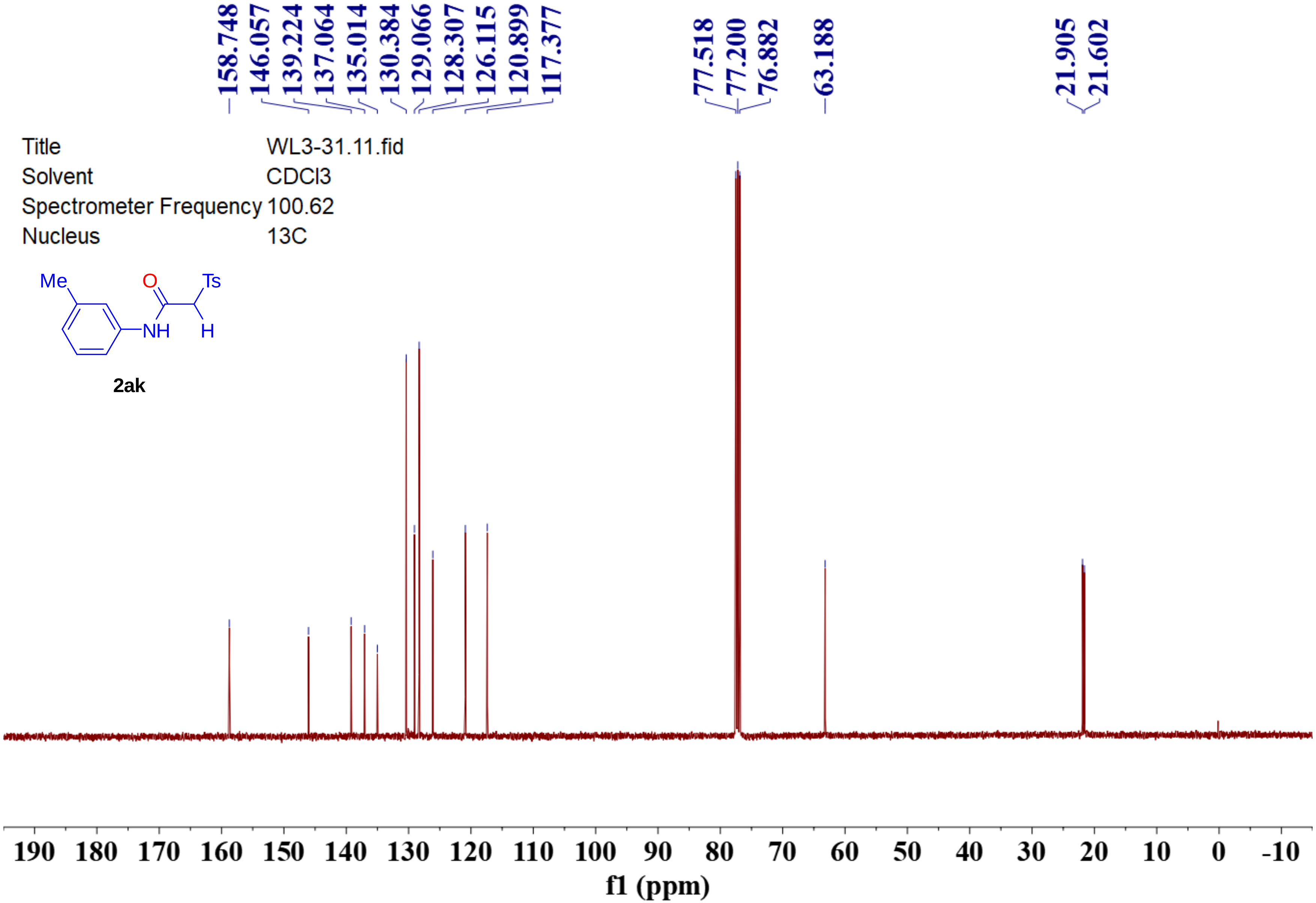

159.057
146.425
140.130
134.828
130.554
128.249
128.130
127.642
127.311
126.980
126.661
126.649
126.623
126.585
126.547
125.427
122.724
120.021
119.939
77.519
77.200
76.884
63.136
21.957
Title WL3-45.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
F3C
NH
H
2aj
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

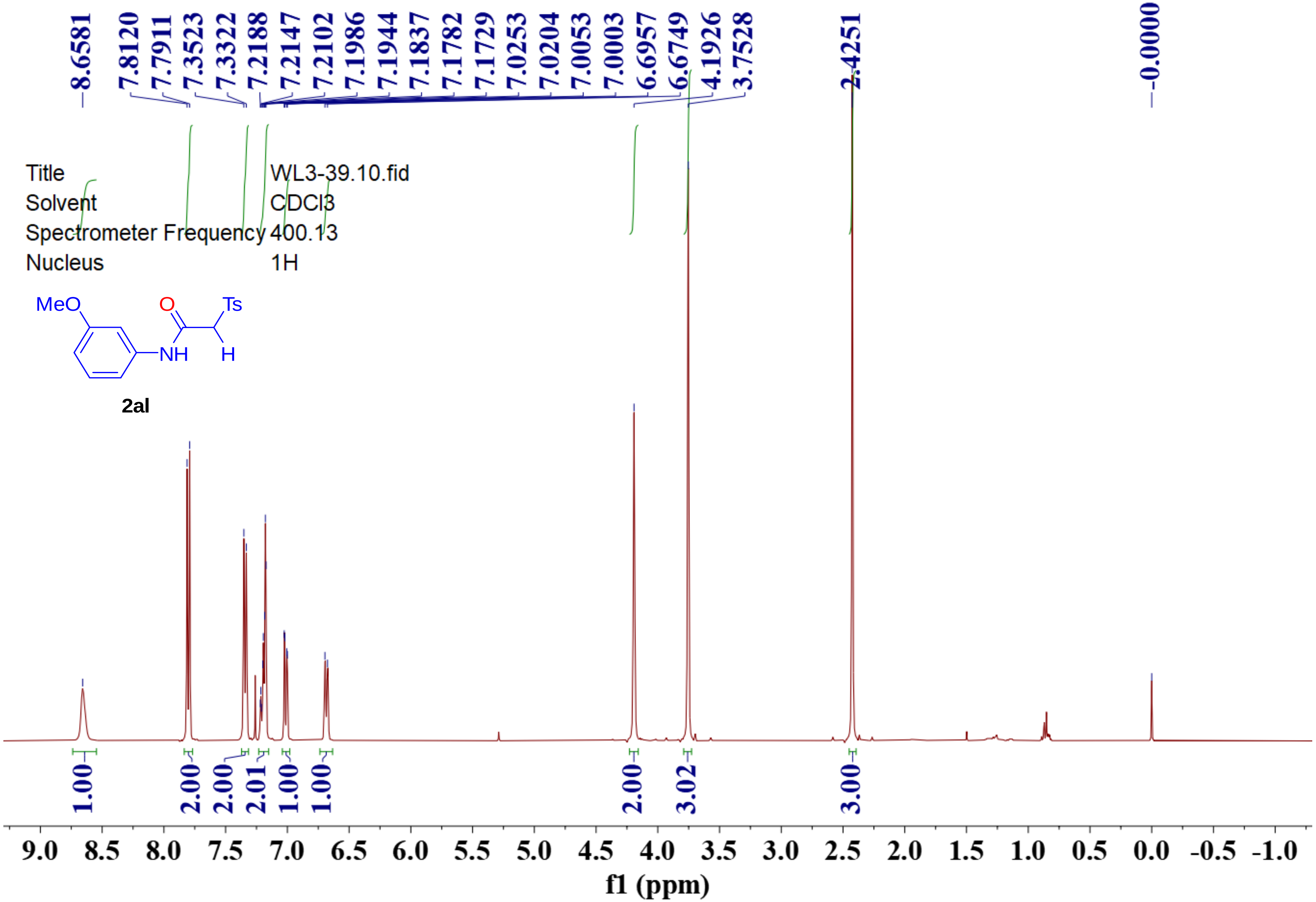


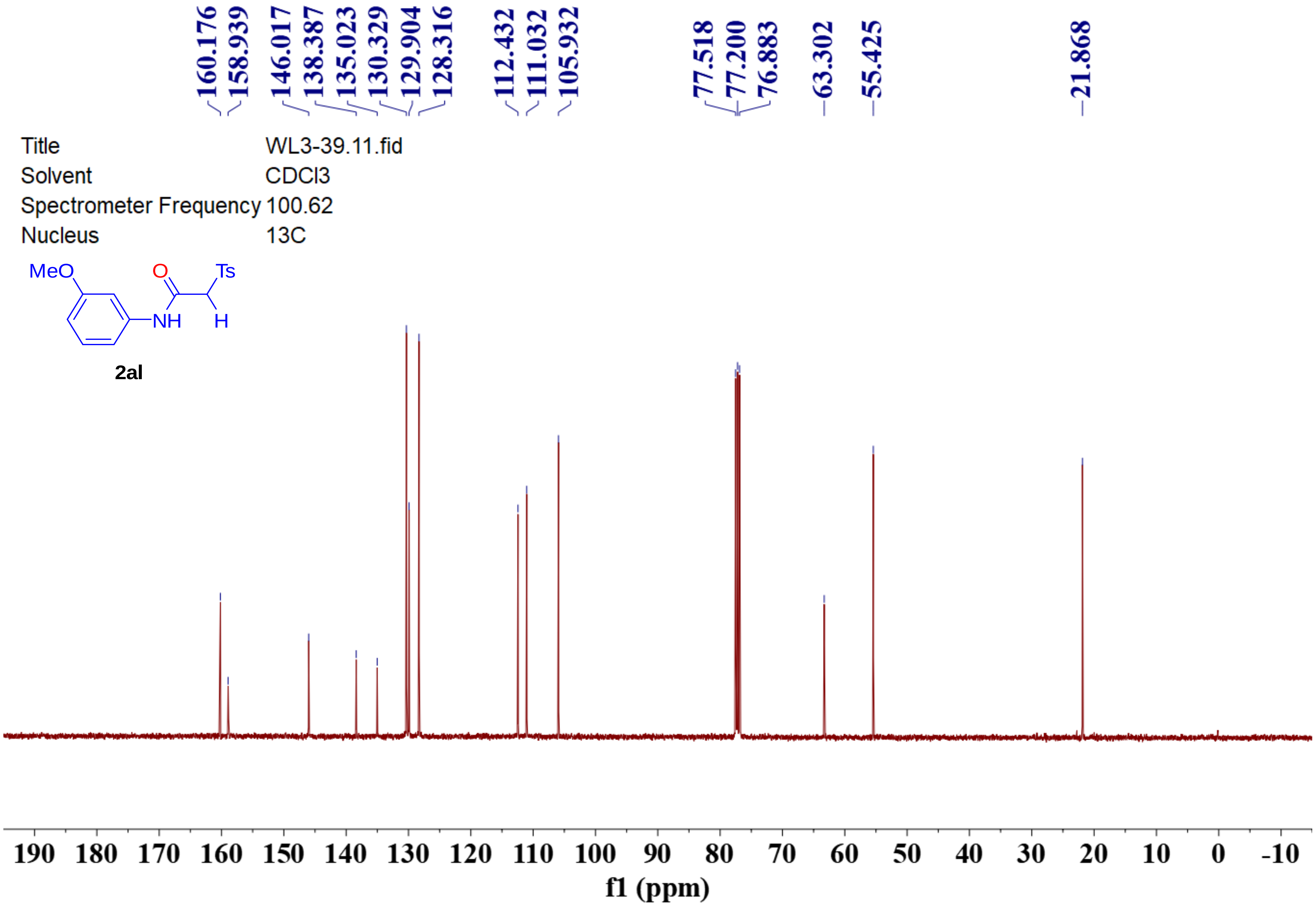
Title	WL3-45.11.fid
Solvent	CDCl3
Spectrometer Frequency	376.46
Nucleus	19F

2aj


8.5405
7.8101
7.7892
7.3614
7.3412
7.3179
7.3016
7.2804
7.2242
7.2050
7.1854
6.9750
6.9562
4.1640
2.4344
2.3243
0.0000
Title WL3-31.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Me
O
Ts
NH
H
2ak
1.00
2.00
4.09
1.01
1.00
2.00
3.00
3.00
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
-0.5
-1.0
f1 (ppm)

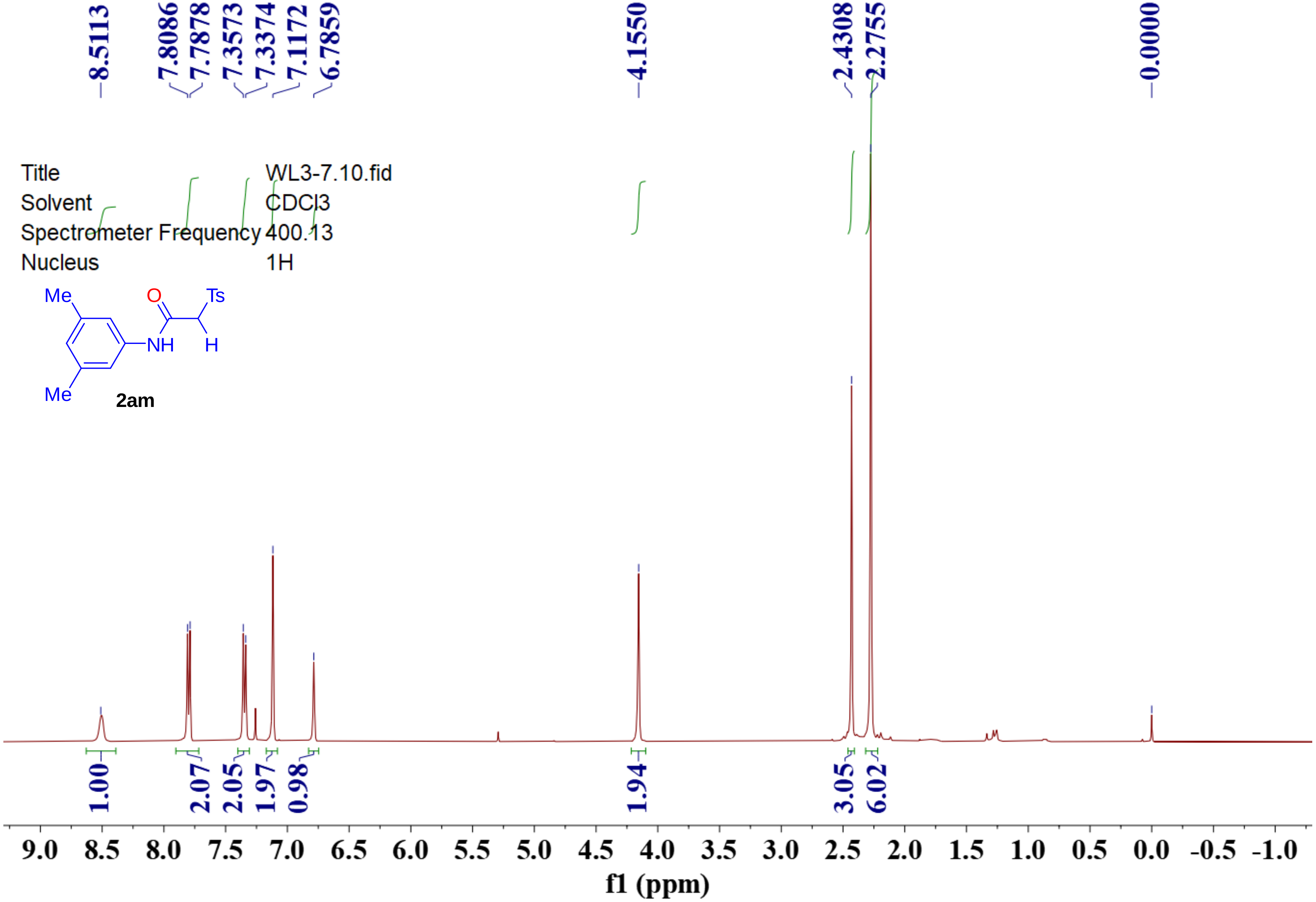

158.748
146.057
139.224
137.064
135.014
130.384
129.066
128.307
126.115
120.899
117.377
77.518
77.200
76.882
63.188
21.905
21.602
Title WL3-31.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
Me
O
Ts
NH
H
2ak
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

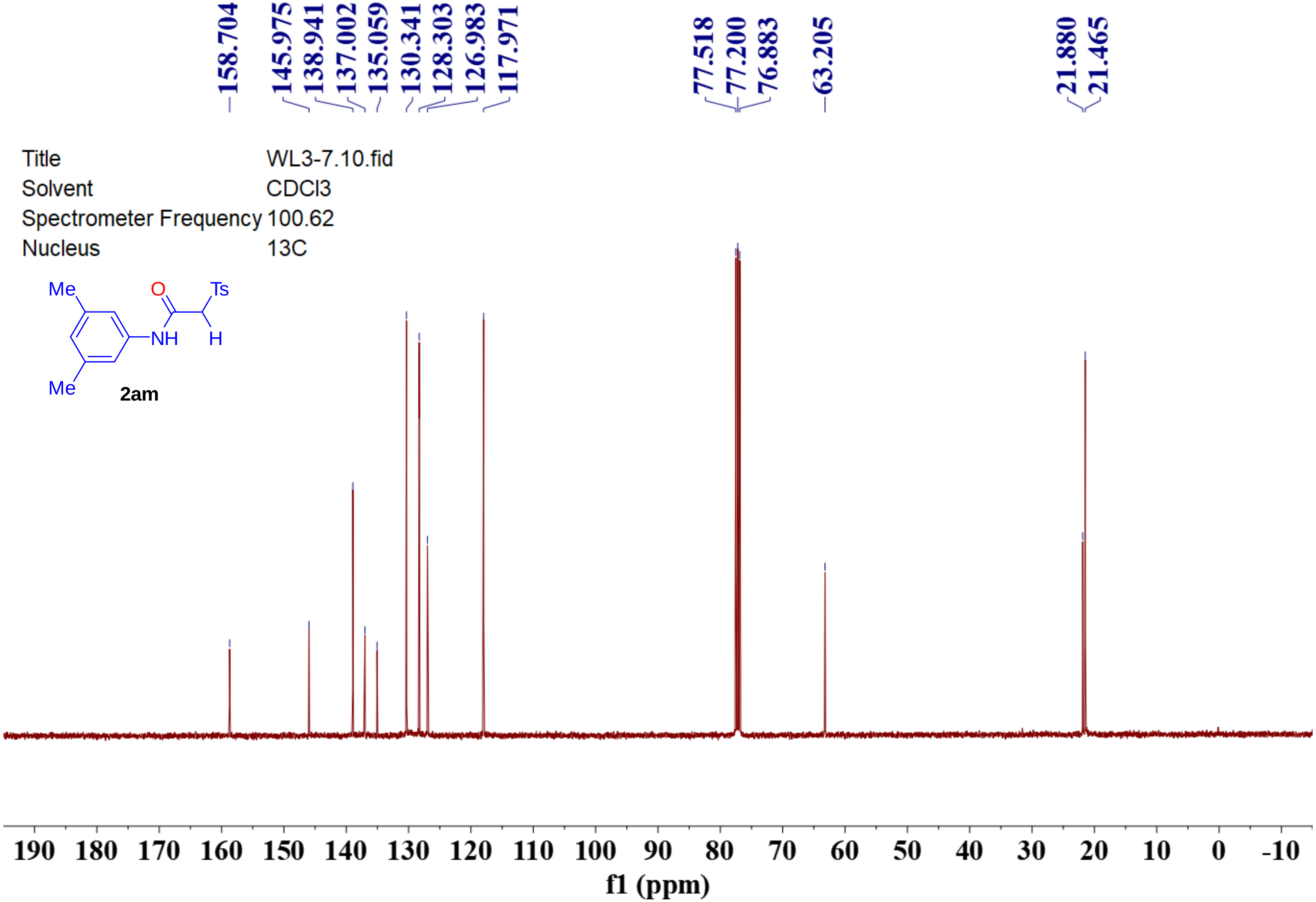

8.6581
7.8120
7.7911
7.3523
7.3322
7.2188
7.2147
7.2102
7.1986
7.1944
7.1837
7.1782
7.1729
7.0253
7.0204
7.0053
7.0003
6.6957
6.6749
4.1926
3.7528
2.4251
-0.0000
Title
WL3-39.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
MeO
O
Ts
NH
H
2al
1.00
2.00
2.00
2.01
1.00
1.00
2.00
3.02
3.00
9.0
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
-0.5
-1.0
f1 (ppm)



Title	WL3-39.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

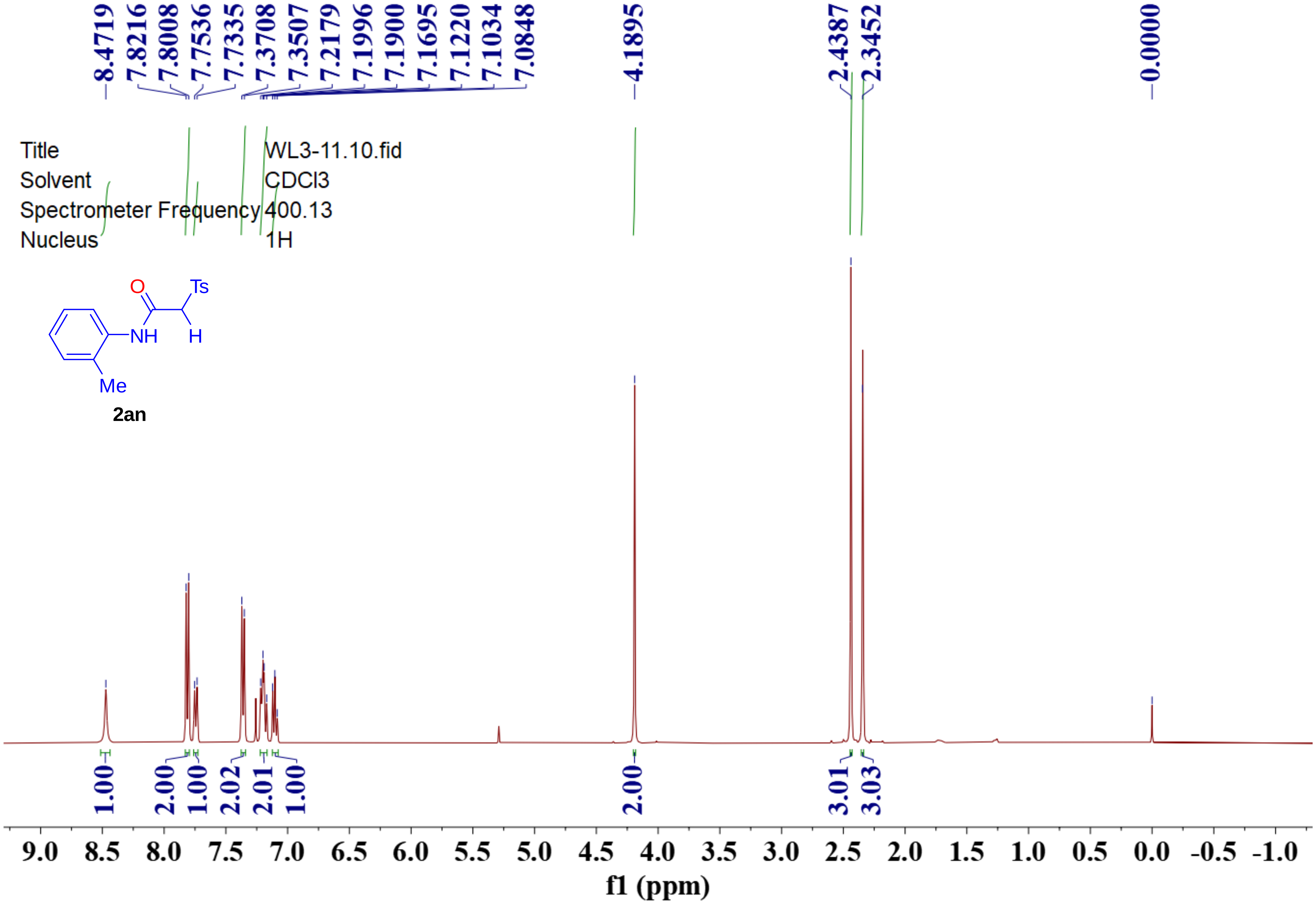
2al

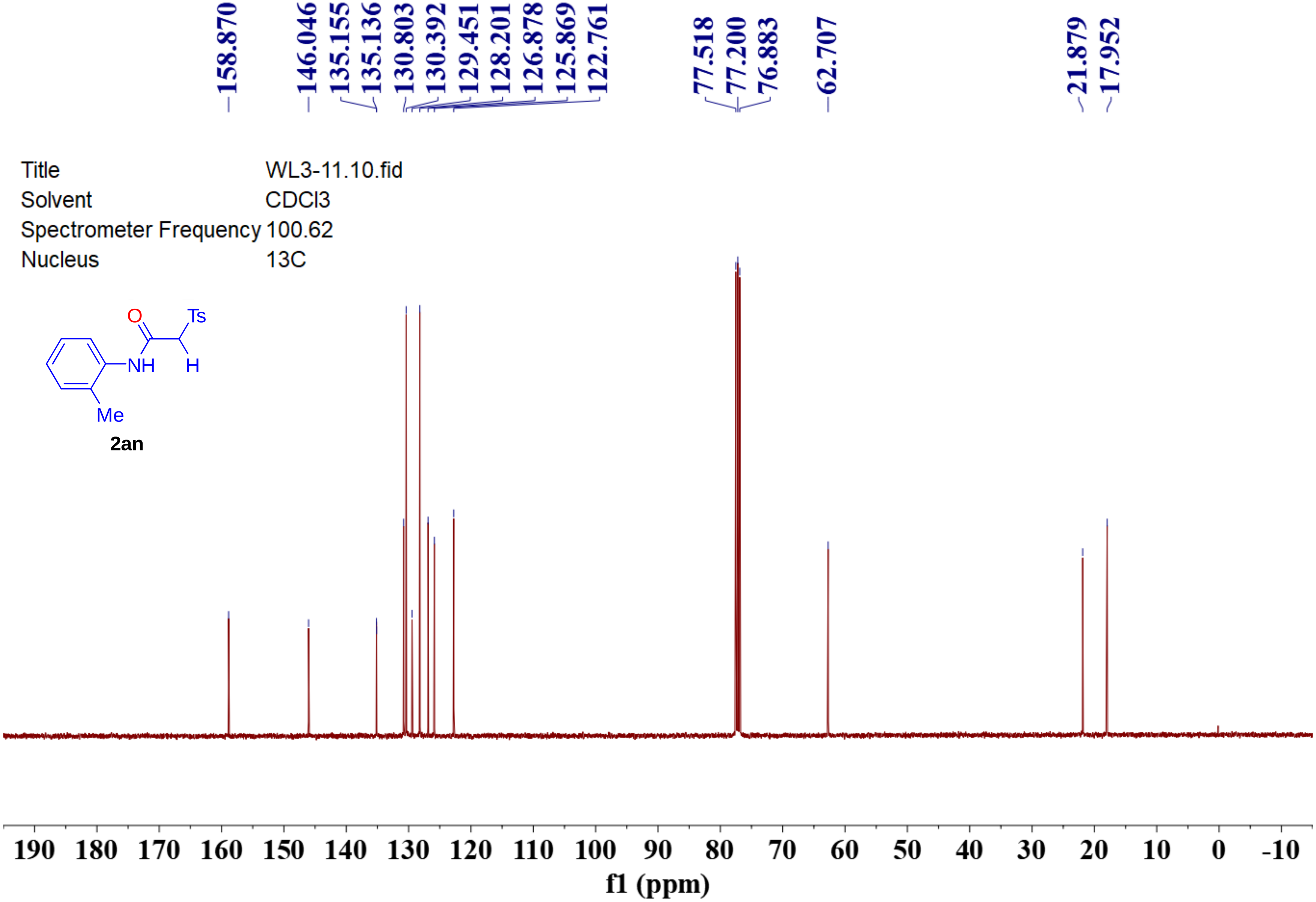

8.5113
7.8086
7.7878
7.3573
7.3374
7.1172
6.7859
4.1550
2.4308
2.2755
0.0000
Title WL3-7.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Me
O
Ts
NH
H
Me
2am
1.00
2.07
2.05
1.97
0.98
1.94
3.05
6.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

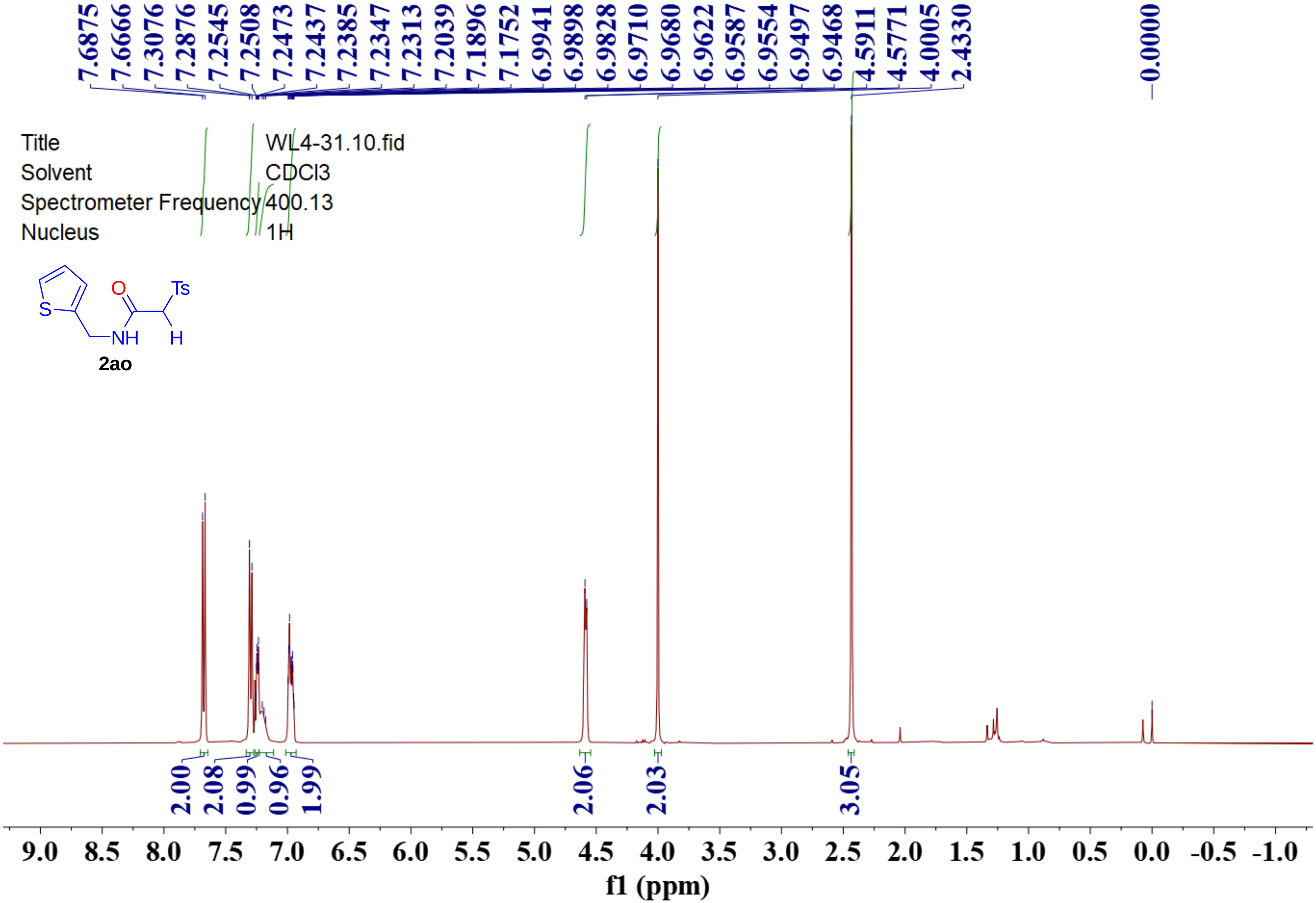


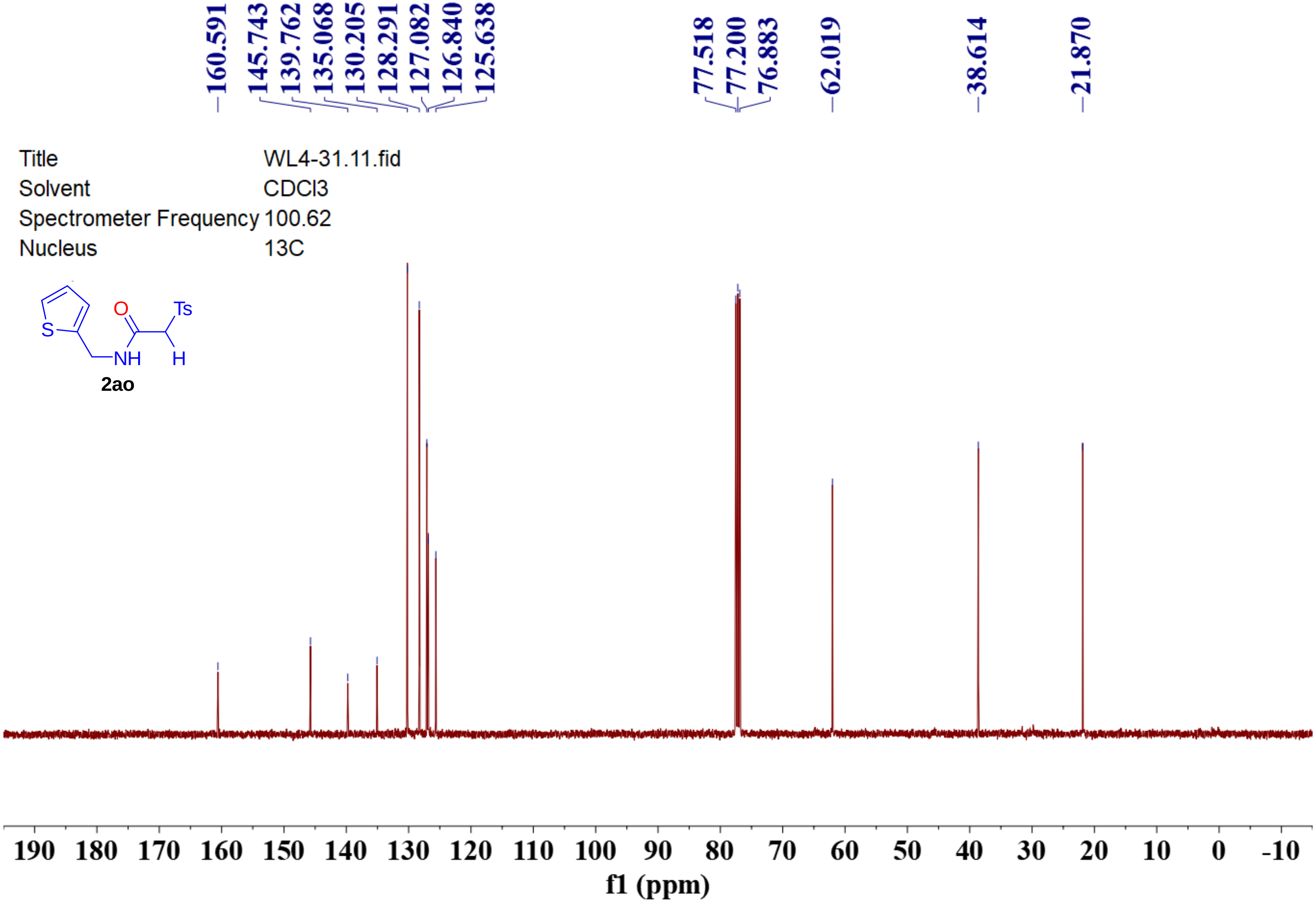
Title	WL3-7.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2am


8.4719
7.8216
7.8008
7.7536
7.7335
7.3708
7.3507
7.2179
7.1996
7.1900
7.1695
7.1220
7.1034
7.0848
4.1895
2.4387
2.3452
0.0000
Title WL3-11.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ts
NH
H
Me
2an
1.00
2.00
1.00
2.02
2.01
1.00
2.00
3.01
3.03
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
-0.5
-1.0
f1 (ppm)

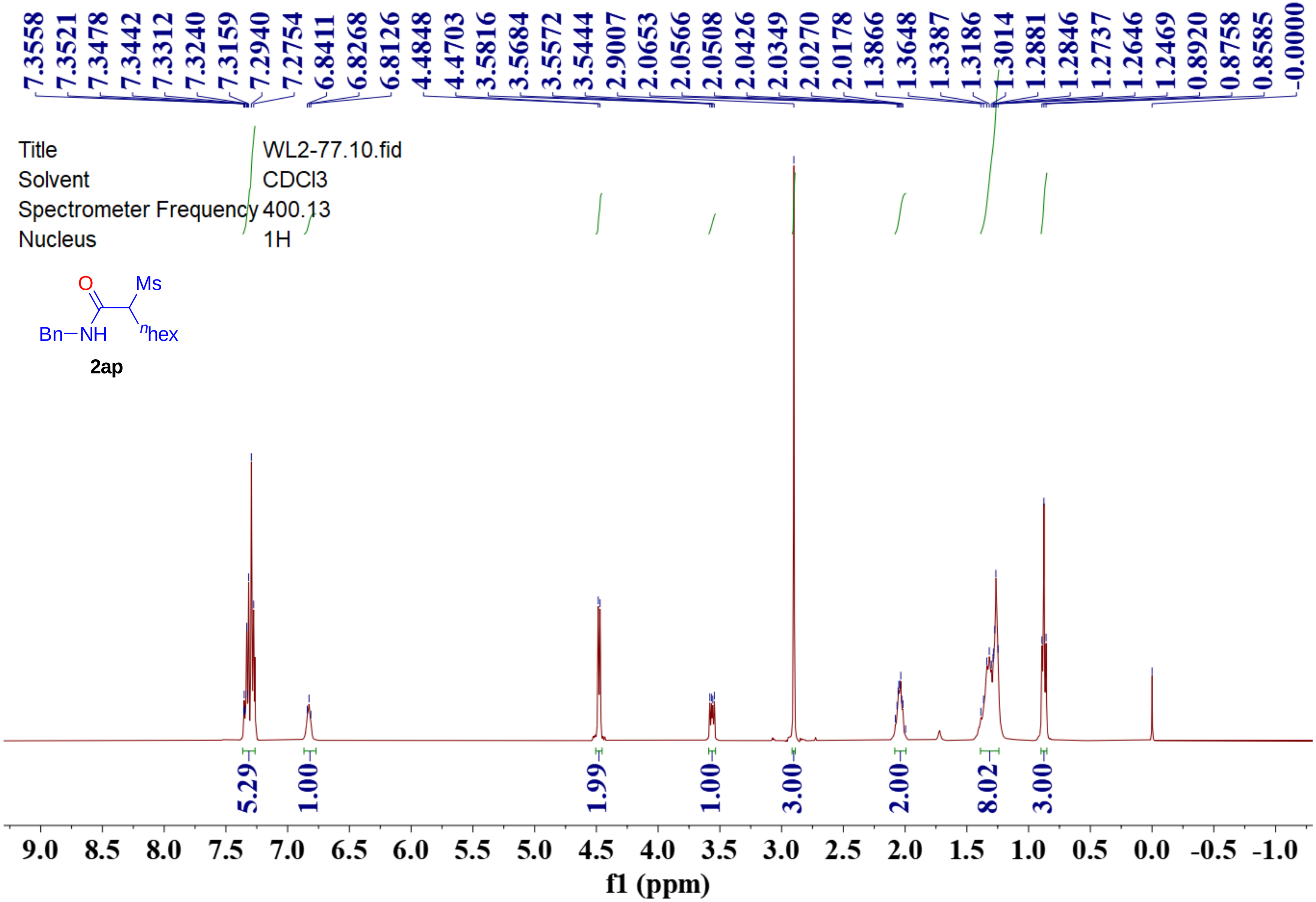

158.870
146.046
135.155
135.136
130.803
130.392
129.451
128.201
126.878
125.869
122.761
77.518
77.200
76.883
62.707
21.879
17.952
Title WL3-11.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
O
Ts
NH
H
Me
2an
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

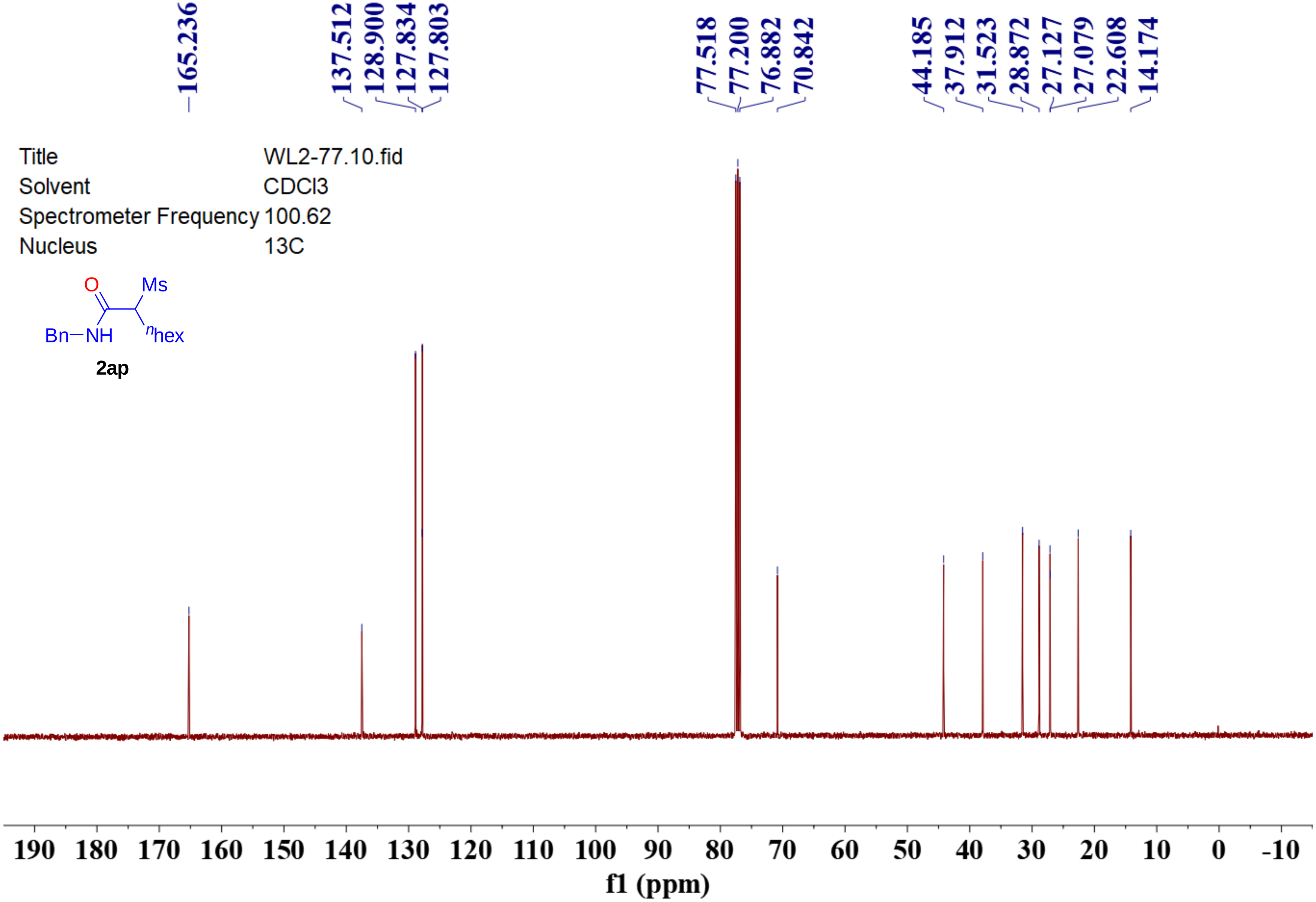

7.6875
7.6666
7.3076
7.2876
7.2545
7.2508
7.2473
7.2437
7.2385
7.2347
7.2313
7.2039
7.1896
7.1752
6.9941
6.9898
6.9828
6.9710
6.9680
6.9622
6.9587
6.9554
6.9497
6.9468
4.5911
4.5771
4.0005
2.4330
0.0000
Title
WL4-31.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
S
O
Ts
NH
H
2ao
2.00
2.08
0.99
0.96
1.99
2.06
2.03
3.05
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
-0.5
-1.0
f1 (ppm)



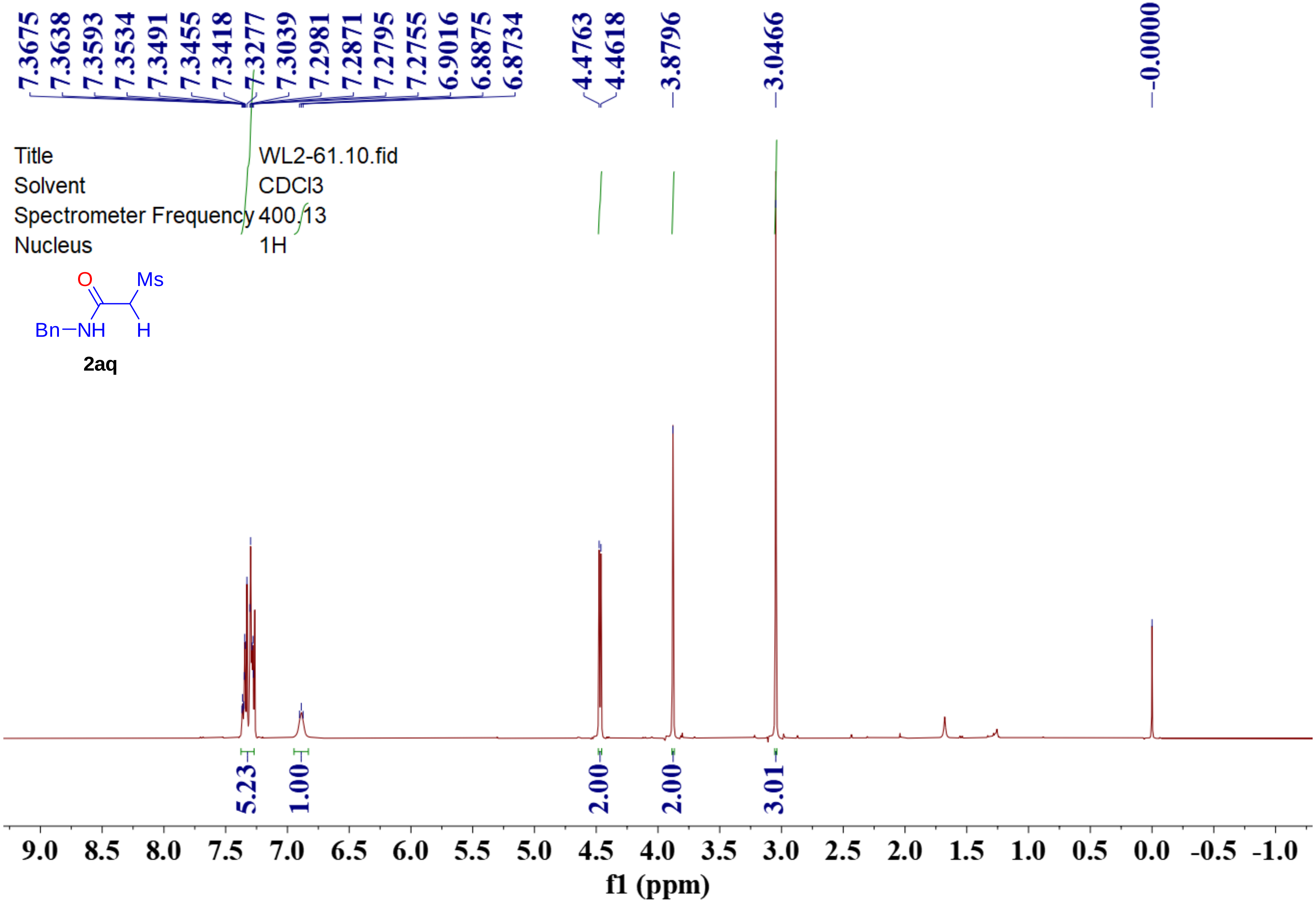
Title	WL4-31.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2ao

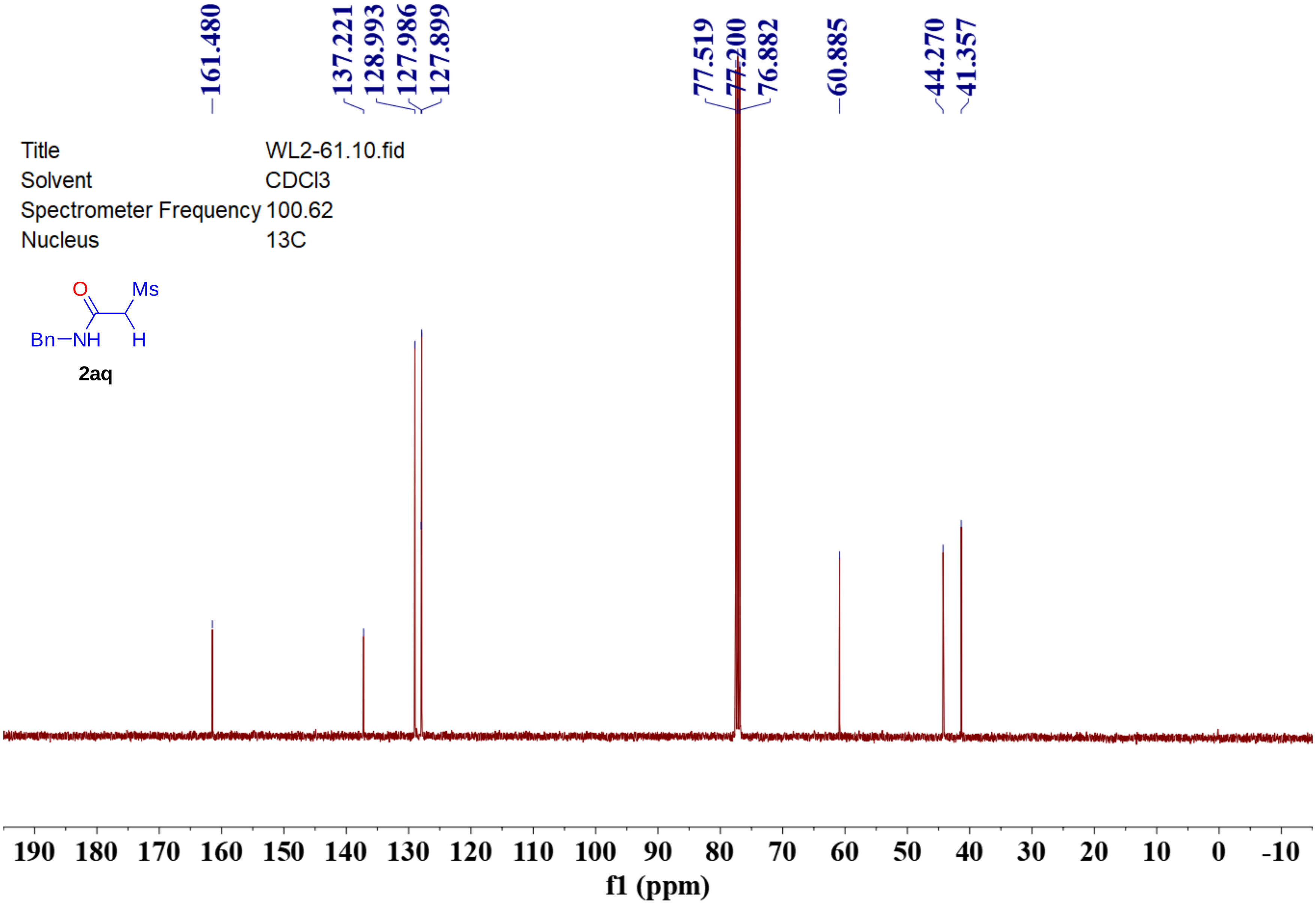

7.3558
7.3521
7.3478
7.3442
7.3312
7.3240
7.3159
7.2940
7.2754
6.8411
6.8268
6.8126
4.4848
4.4703
3.5816
3.5684
3.5572
3.5444
2.9007
2.0653
2.0566
2.0508
2.0426
2.0349
2.0270
2.0178
1.3866
1.3648
1.3387
1.3186
1.3014
1.2881
1.2846
1.2737
1.2646
1.2469
0.8920
0.8758
0.8585
-0.0000
Title
WL2-77.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
O
Ms
Bn—NH
ⁿhex
2ap
5.29
1.00
1.99
1.00
3.00
2.00
8.02
3.00
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
-0.5
-1.0
f1 (ppm)

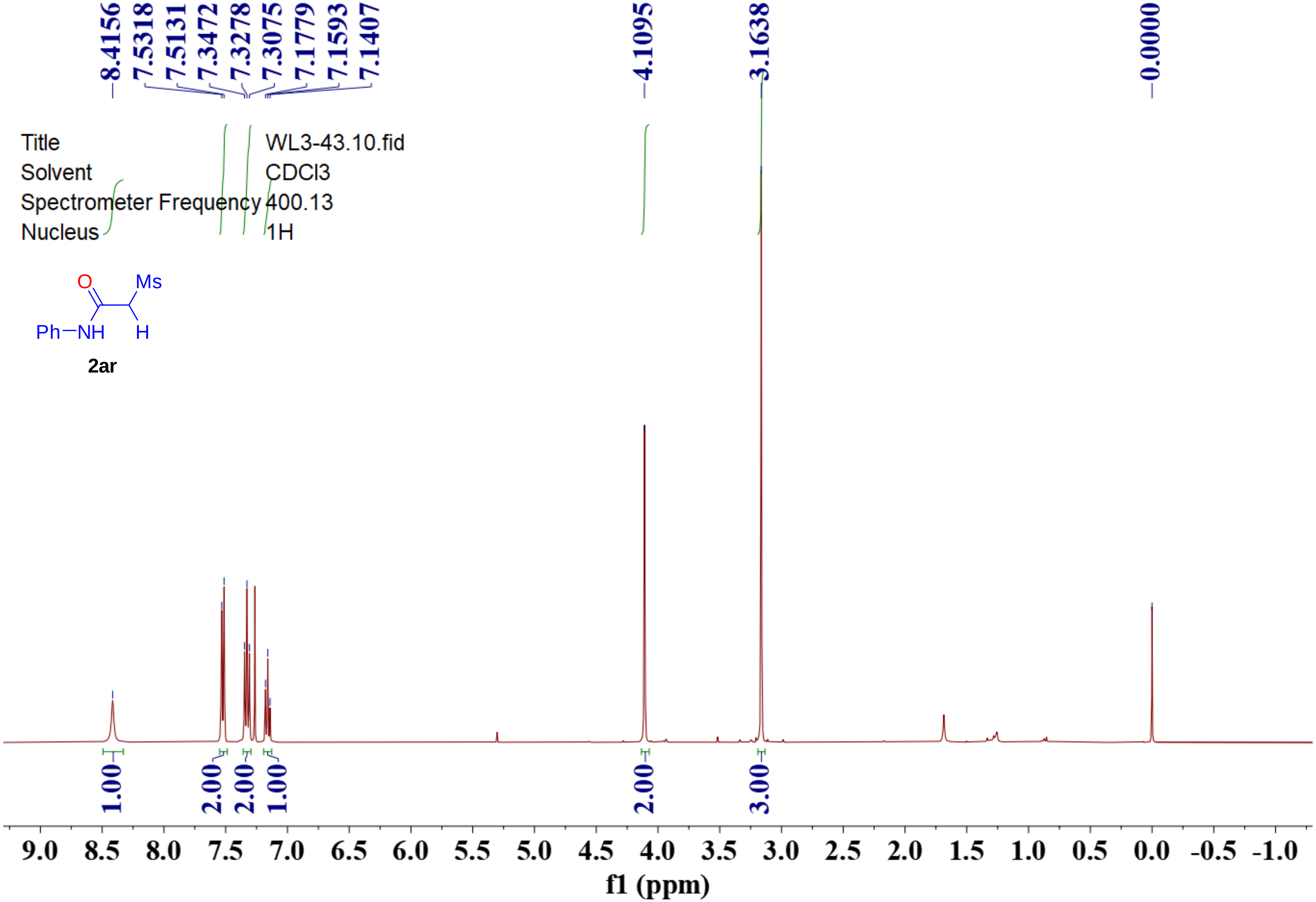


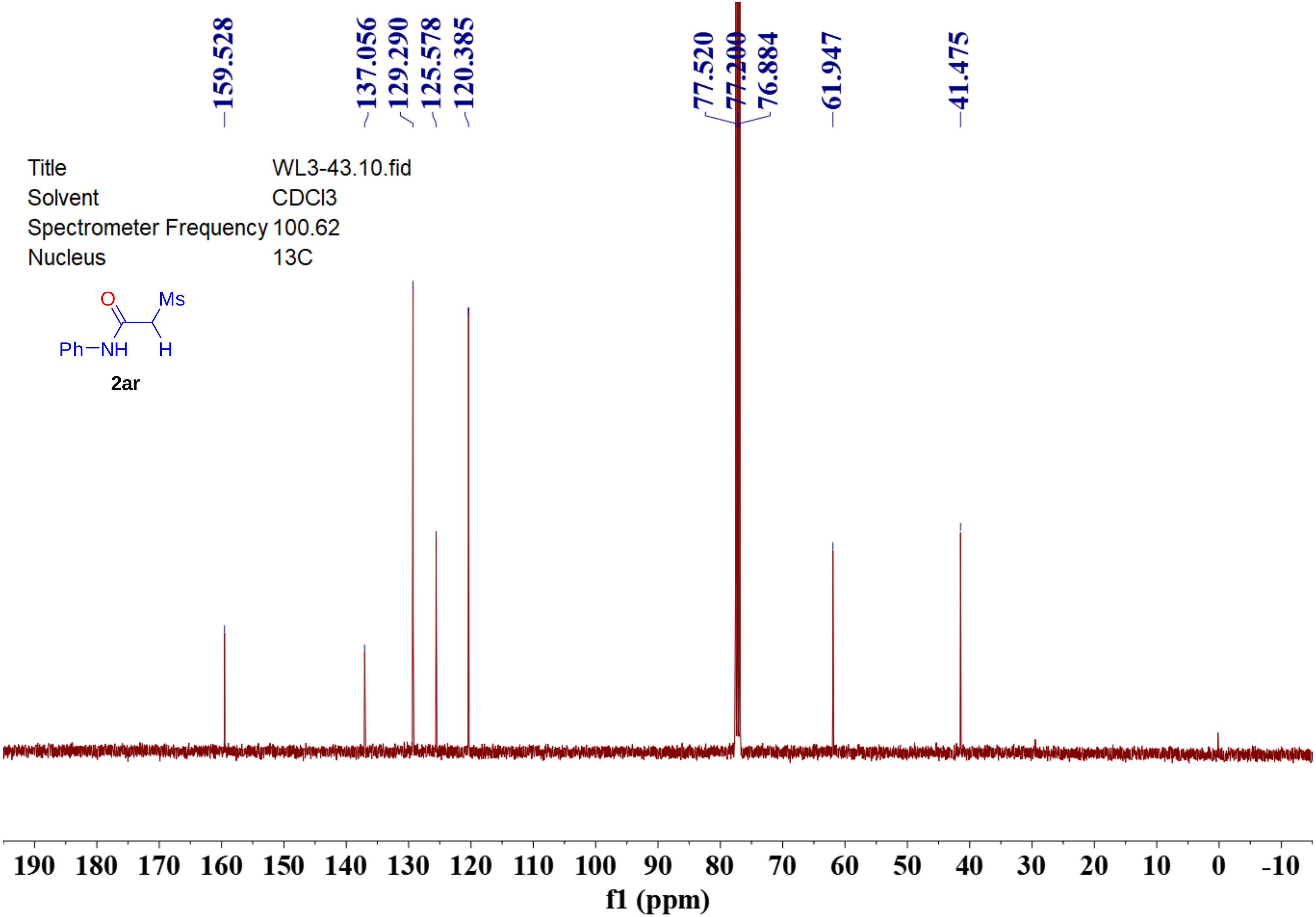
Title WL2-77.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C


7.3675
7.3638
7.3593
7.3534
7.3491
7.3455
7.3418
7.3277
7.3039
7.2981
7.2871
7.2795
7.2755
6.9016
6.8875
6.8734
4.4763
4.4618
3.8796
3.0466
-0.0000
Title WL2-61.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ms
Bn
NH
H
5.23
1.00
2.00
2.00
3.01
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 -0.5 -1.0
f1 (ppm)

2aq

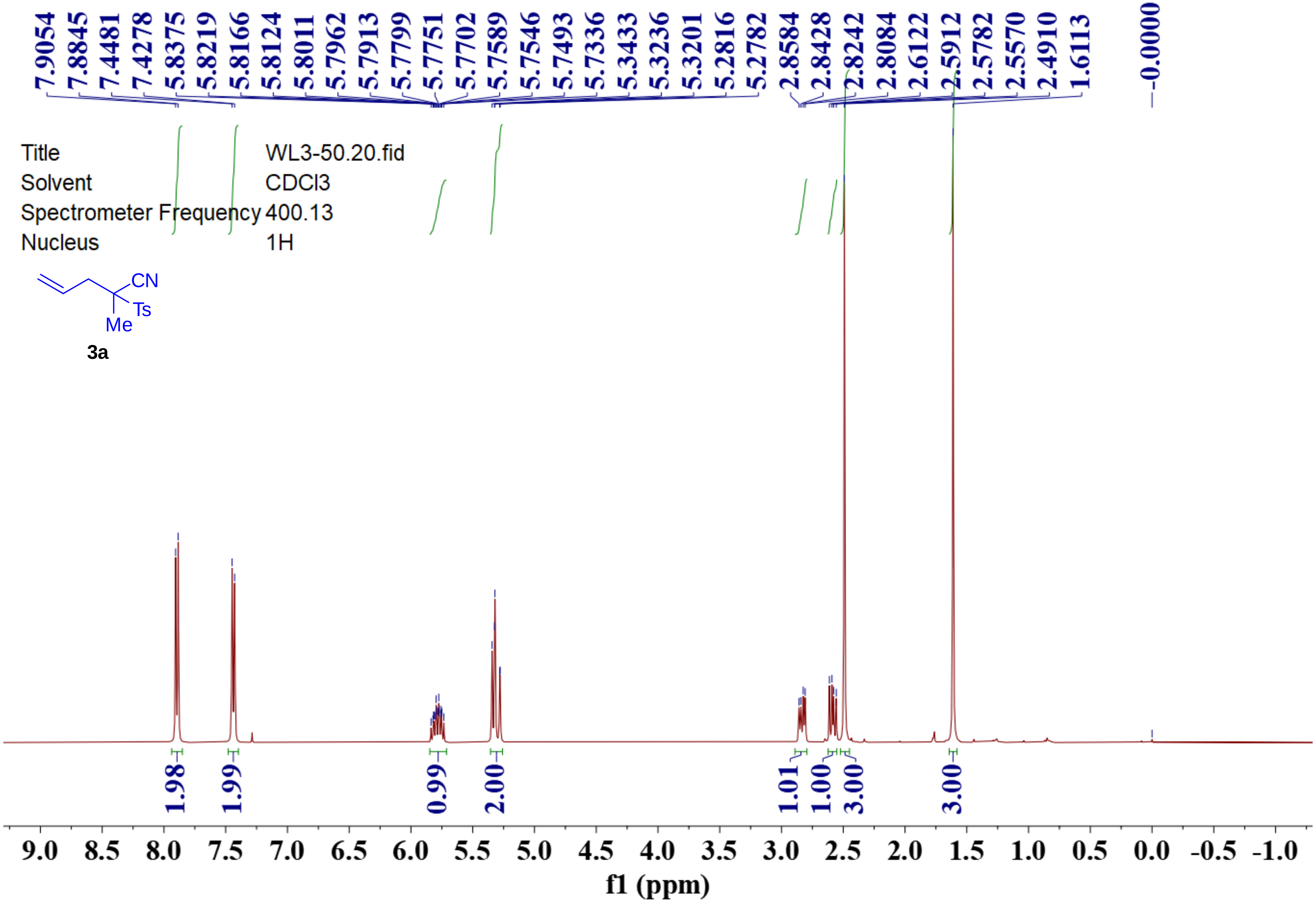

161.480
137.221
128.993
127.986
127.899
77.519
77.200
76.882
60.885
44.270
41.357
Title
WL2-61.10.fid
Solvent
CDCl3
Spectrometer Frequency
100.62
Nucleus
13C
O
Ms
Bn
NH
H
2aq
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

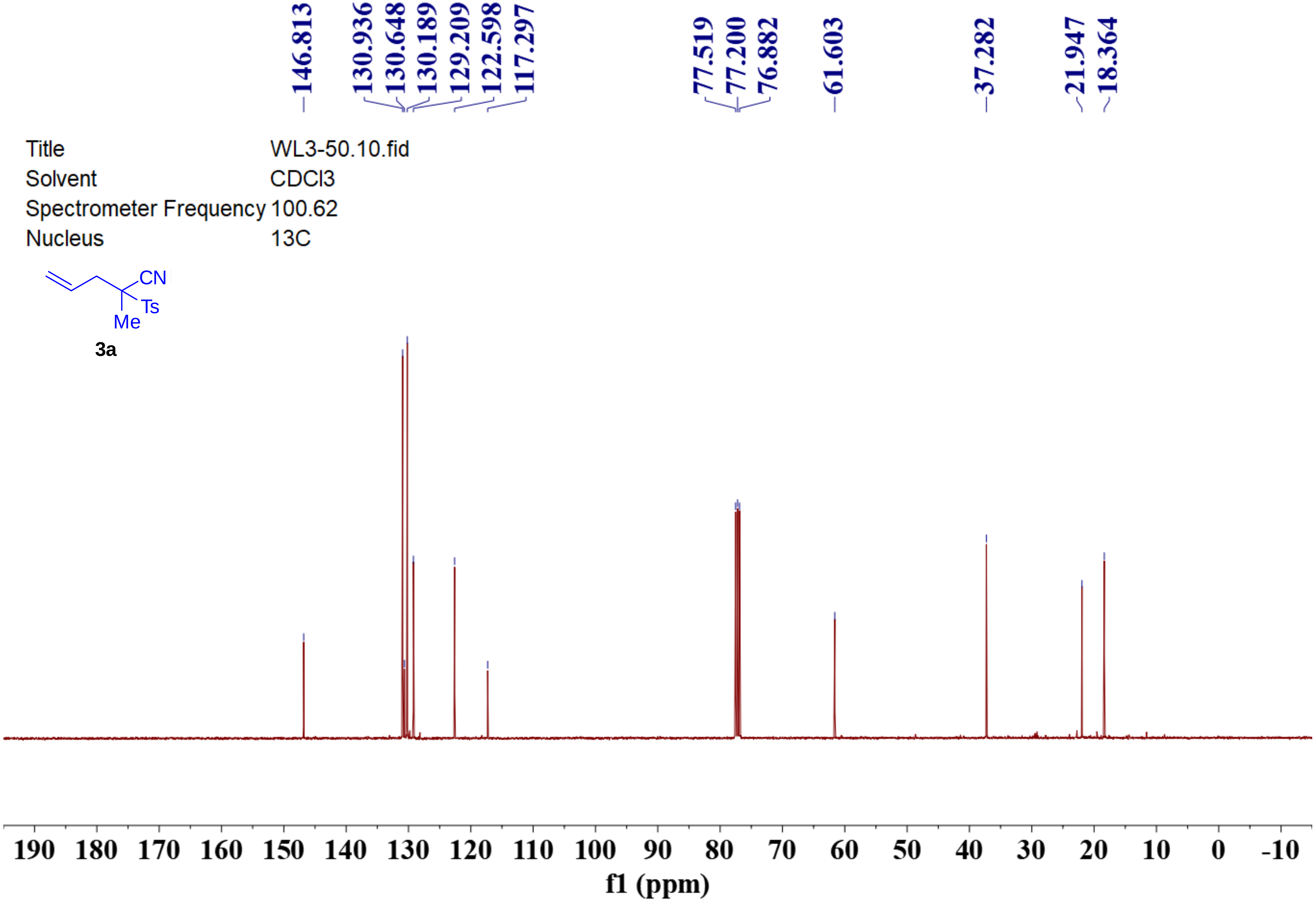

8.4156
7.5318
7.5131
7.3472
7.3278
7.3075
7.1779
7.1593
7.1407
4.1095
3.1638
0.0000
Title WL3-43.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
O
Ms
Ph—NH
H
2ar
1.00
2.00
2.00
1.00
2.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

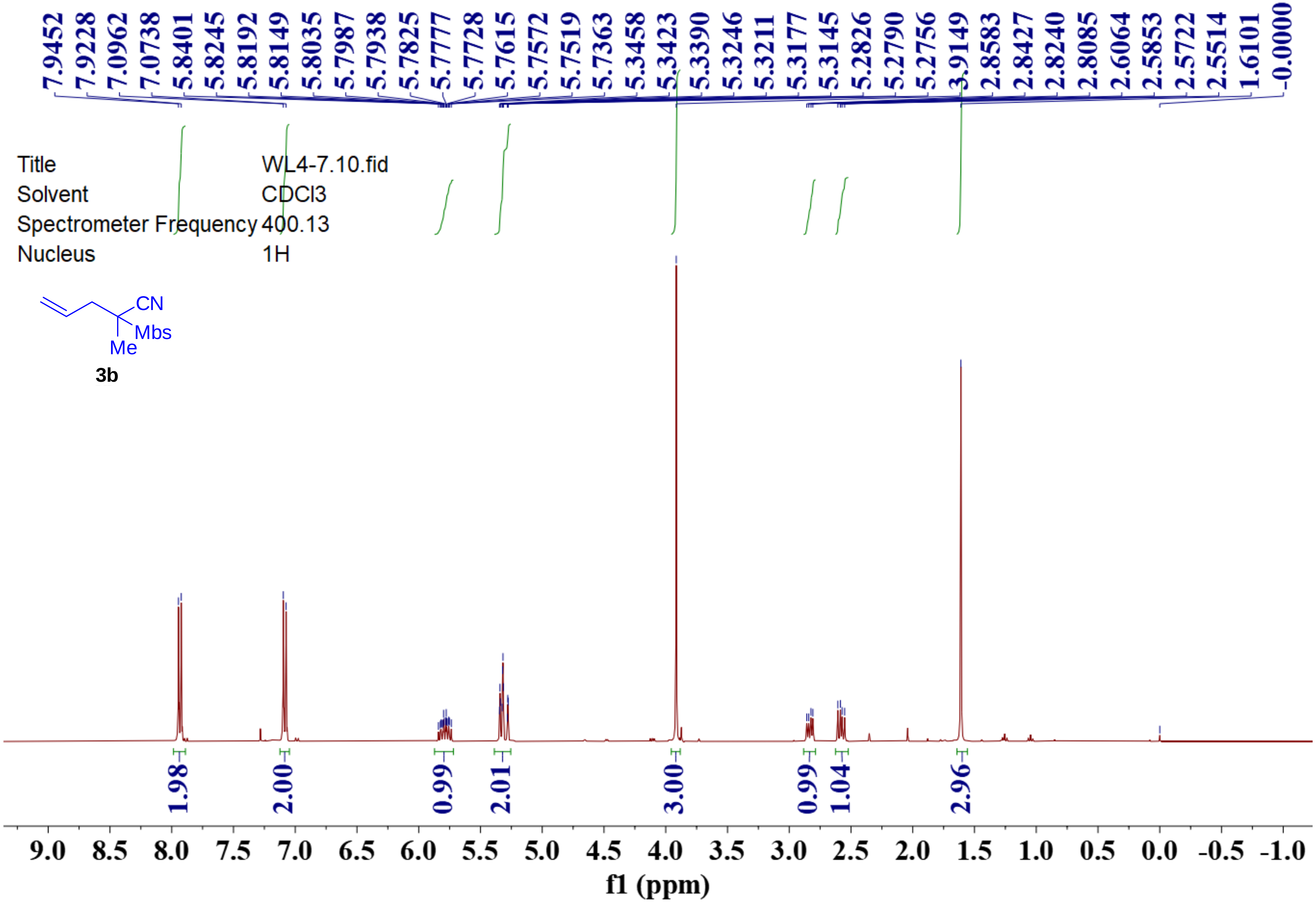


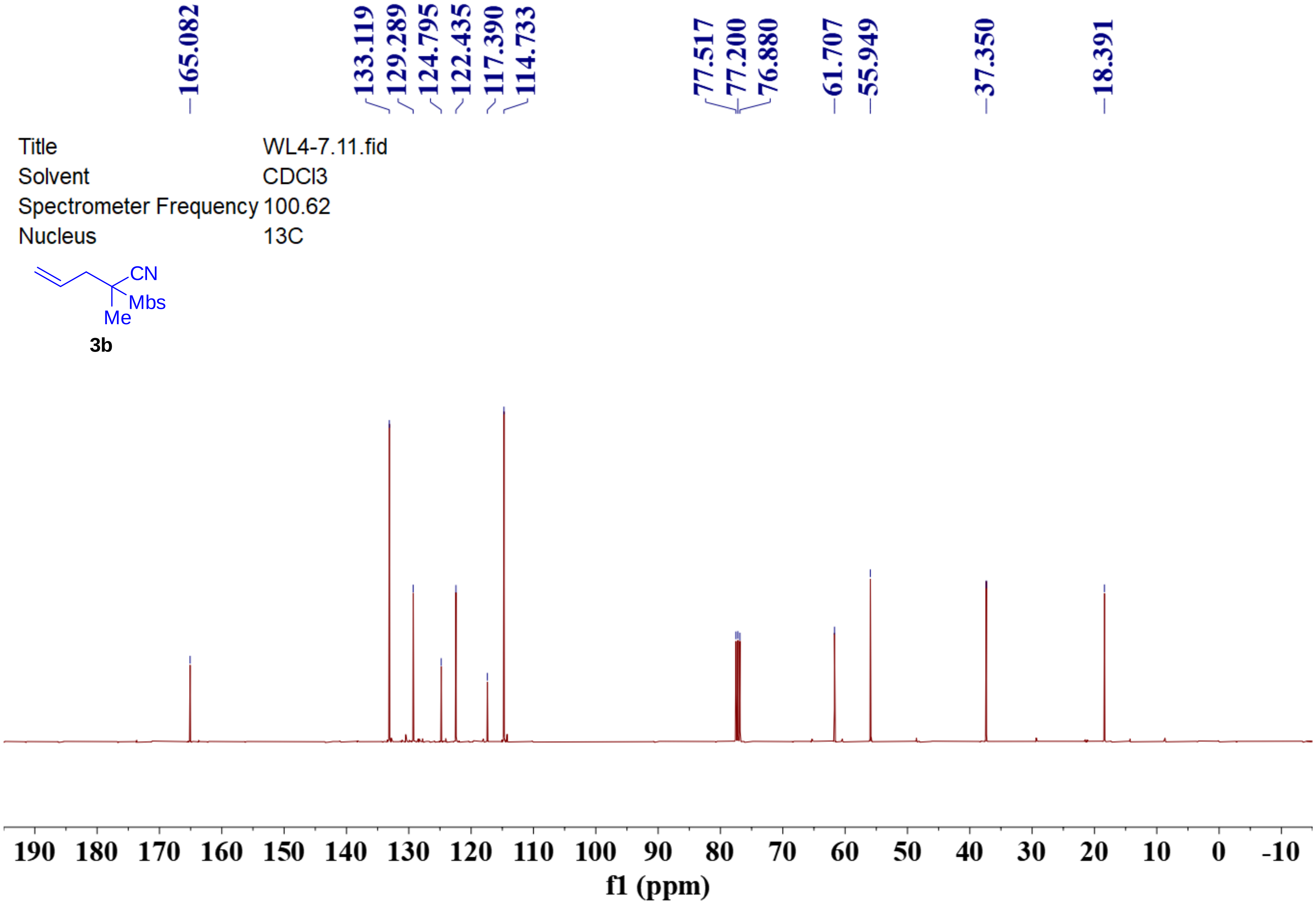
Title	WL3-43.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

2ar


7.9054
7.8845
7.4481
7.4278
5.8375
5.8219
5.8166
5.8124
5.8011
5.7962
5.7913
5.7799
5.7751
5.7702
5.7589
5.7546
5.7493
5.7336
5.3433
5.3236
5.3201
5.2816
5.2782
2.8584
2.8428
2.8242
2.8084
2.6122
2.5912
2.5782
2.5570
2.4910
1.6113
-0.0000
Title
WL3-50.20.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
CN
Ts
Me
3a
1.98
1.99
0.99
2.00
1.01
1.00
3.00
3.00
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
-0.5
-1.0
f1 (ppm)

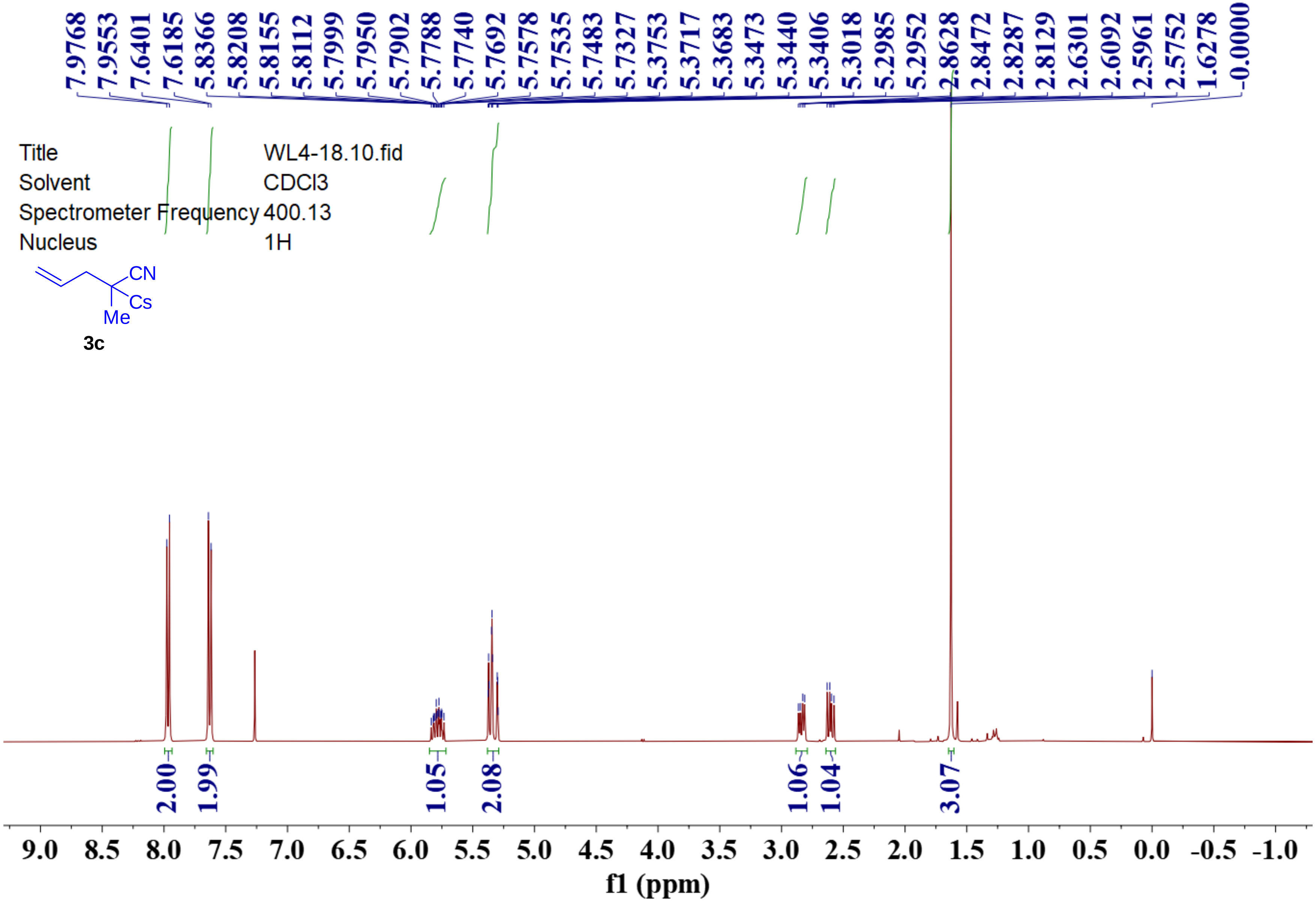

146.813
130.936
130.648
130.189
129.209
122.598
117.297
77.519
77.200
76.882
61.603
37.282
21.947
18.364
Title WL3-50.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ts
Me
3a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

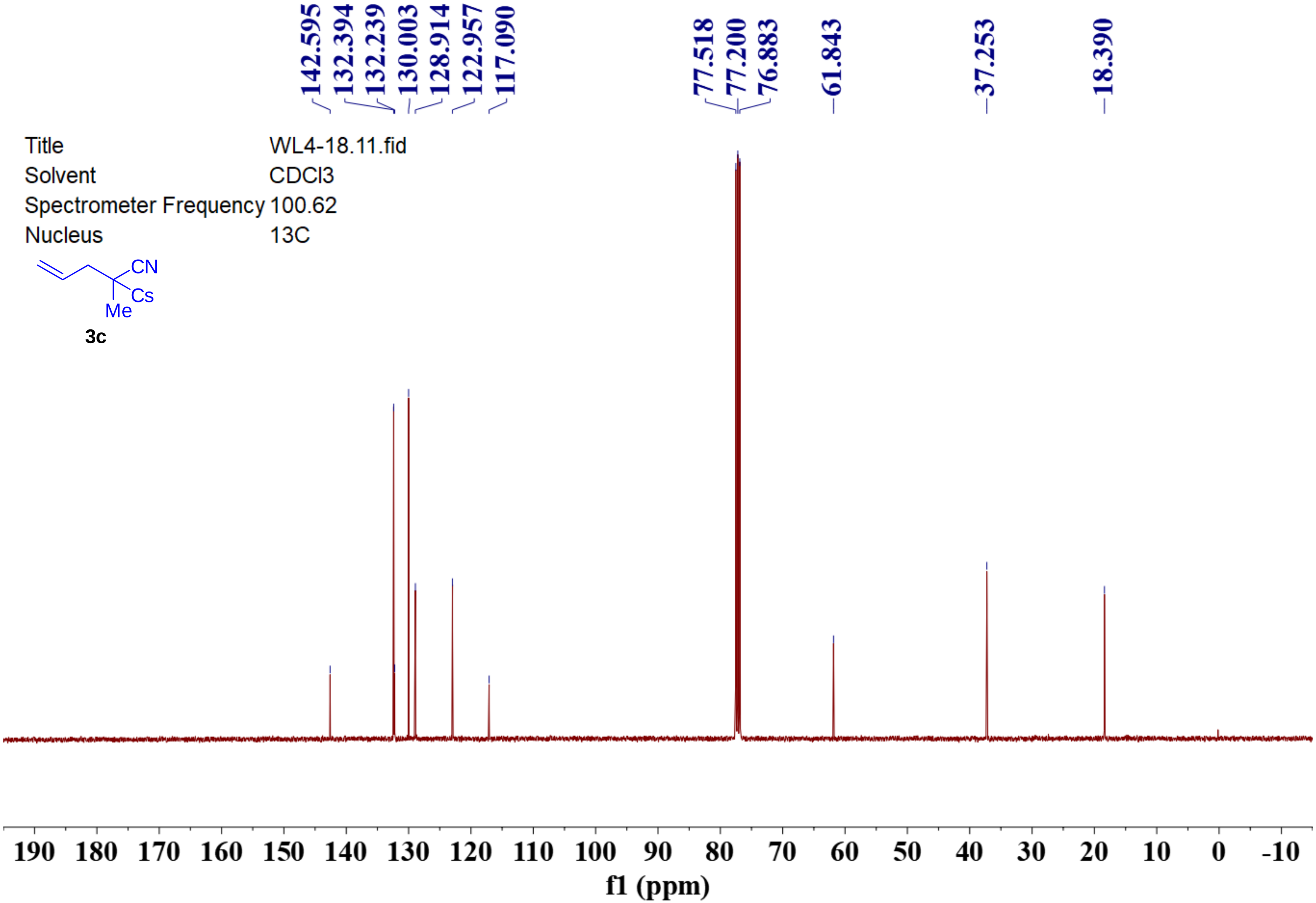

7.9452
7.9228
7.0962
7.0738
5.8401
5.8245
5.8192
5.8149
5.8035
5.7987
5.7938
5.7825
5.7777
5.7728
5.7615
5.7572
5.7519
5.7363
5.3458
5.3423
5.3390
5.3246
5.3211
5.3177
5.3145
5.2826
5.2790
5.2756
3.9149
2.8583
2.8427
2.8240
2.8085
2.6064
2.5853
2.5722
2.5514
1.6101
-0.0000
Title WL4-7.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Mbs
Me
3b
1.98
2.00
0.99
2.01
3.00
0.99
1.04
2.96
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
-0.5
-1.0
f1 (ppm)

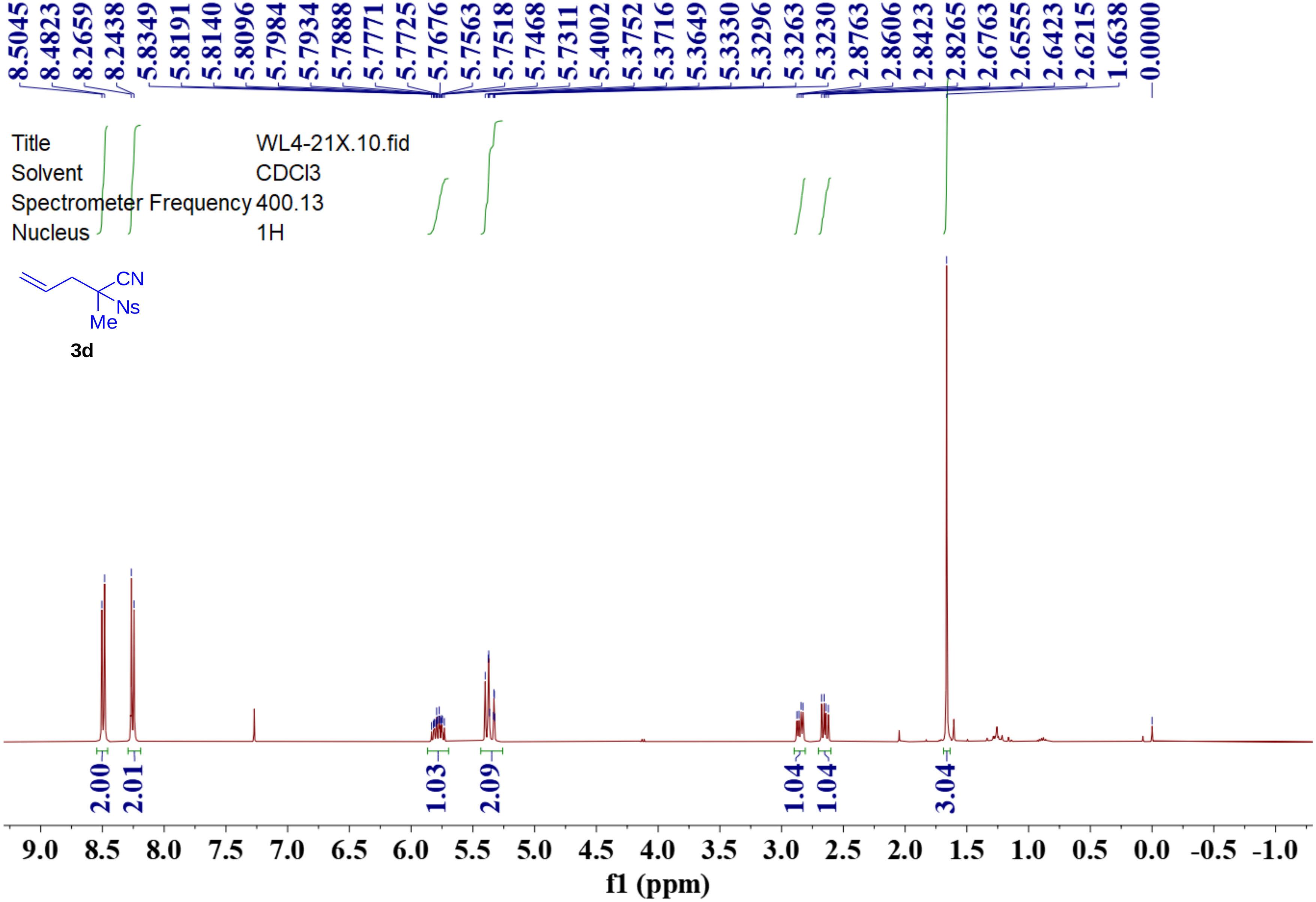


3b

Title	WL4-7.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

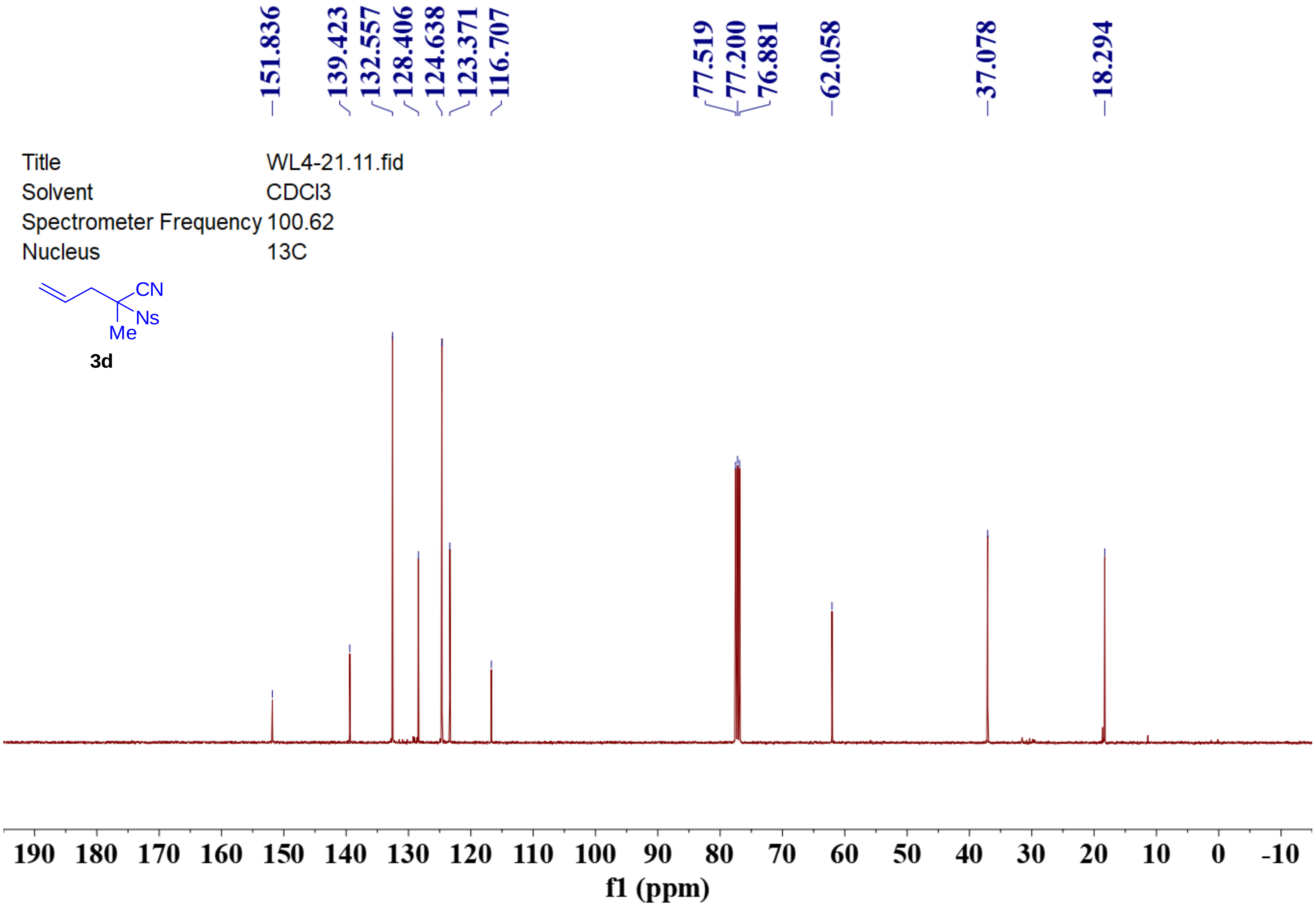

7.9768
7.9553
7.6401
7.6185
5.8366
5.8208
5.8155
5.8112
5.7999
5.7950
5.7902
5.7788
5.7740
5.7692
5.7578
5.7535
5.7483
5.7327
5.3753
5.3717
5.3683
5.3473
5.3440
5.3406
5.3018
5.2985
5.2952
2.8628
2.8472
2.8287
2.8129
2.6301
2.6092
2.5961
2.5752
1.6278
-0.0000
Title WL4-18.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Cs
Me
3c
2.00
1.99
1.05
2.08
1.06
1.04
3.07
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 -0.5 -1.0
f1 (ppm)

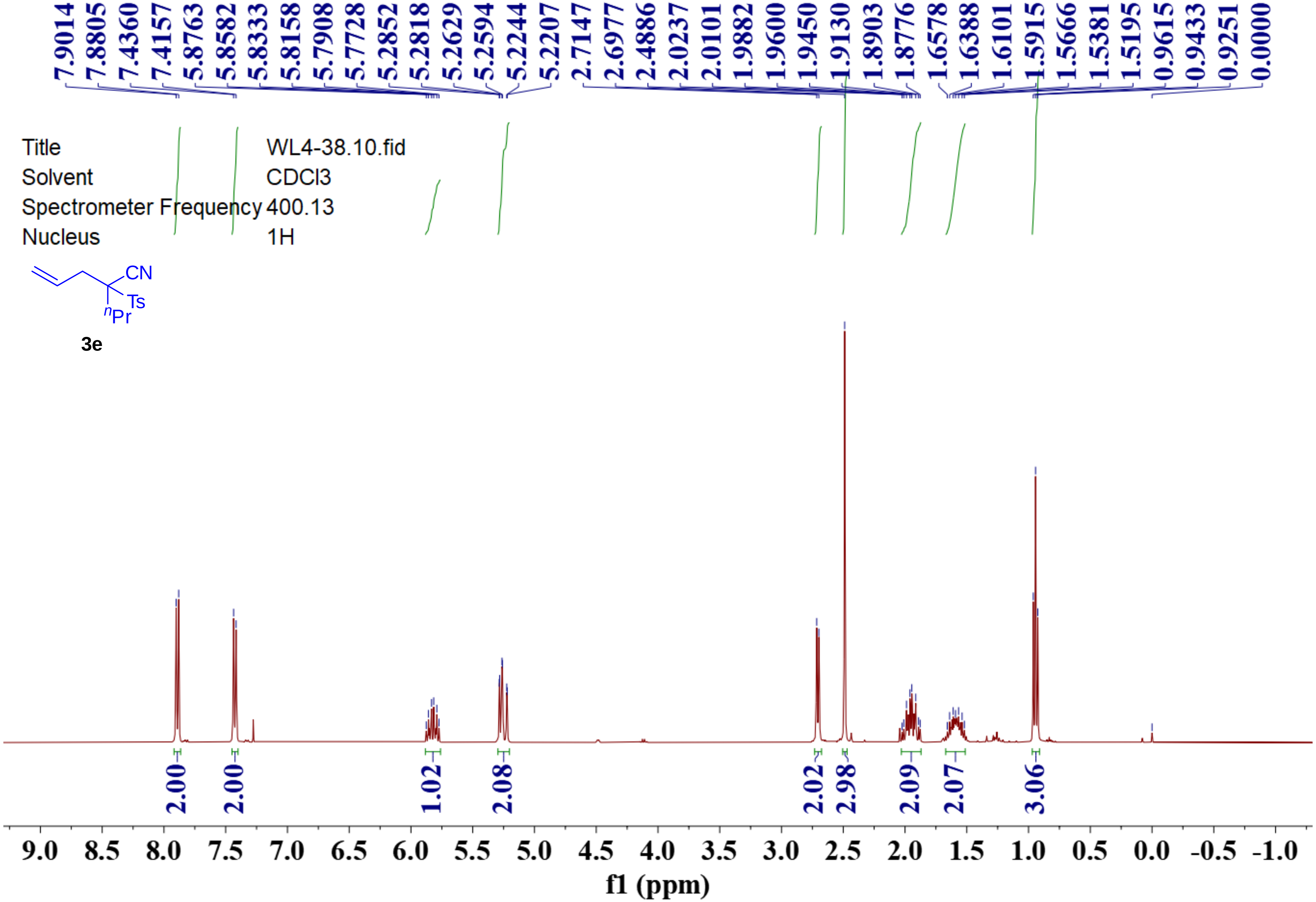

142.595
132.394
132.239
130.003
128.914
122.957
117.090
77.518
77.200
76.883
61.843
37.253
18.390
Title WL4-18.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Cs
Me
3c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

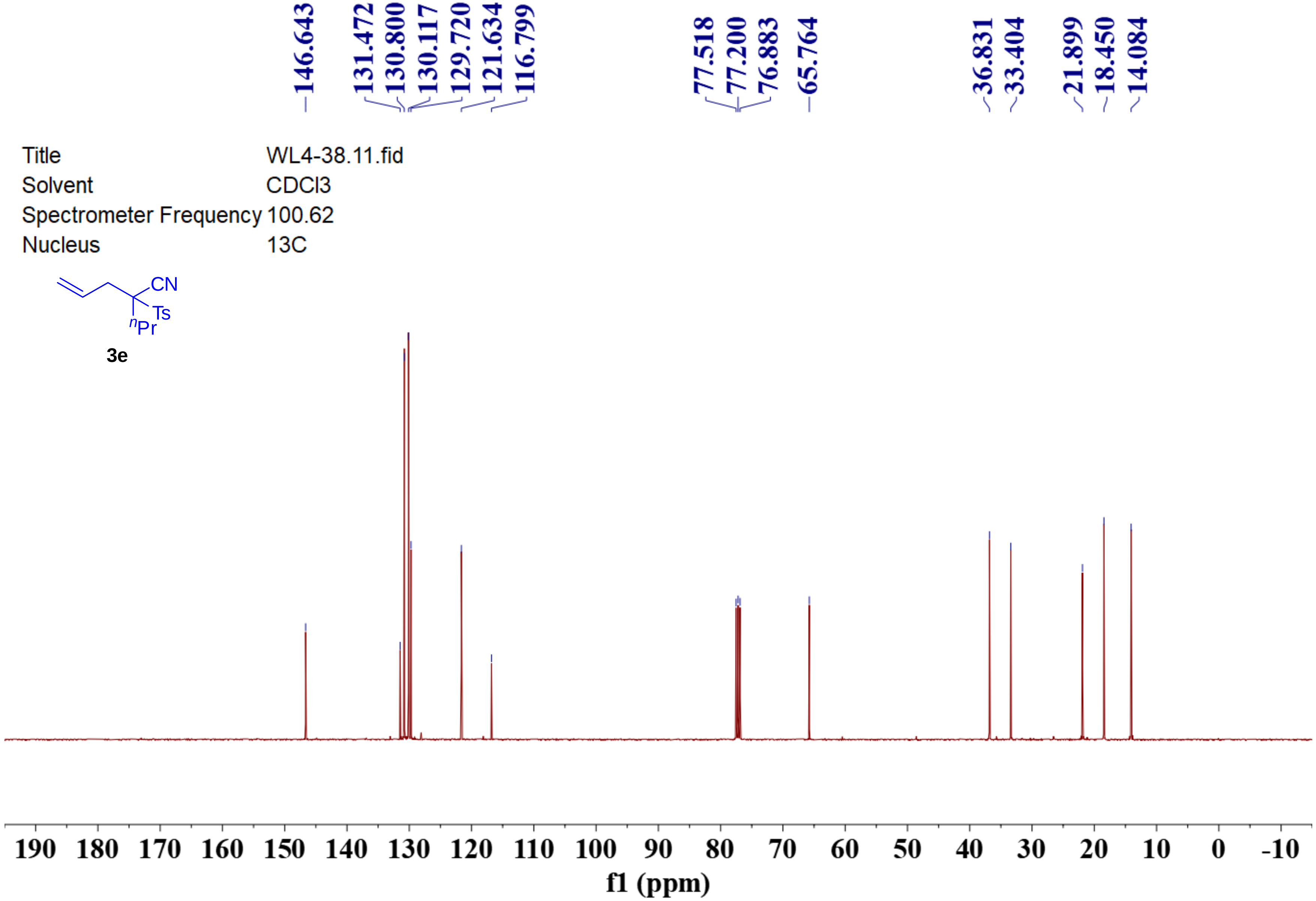


Title	WL4-21X.10.fid
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

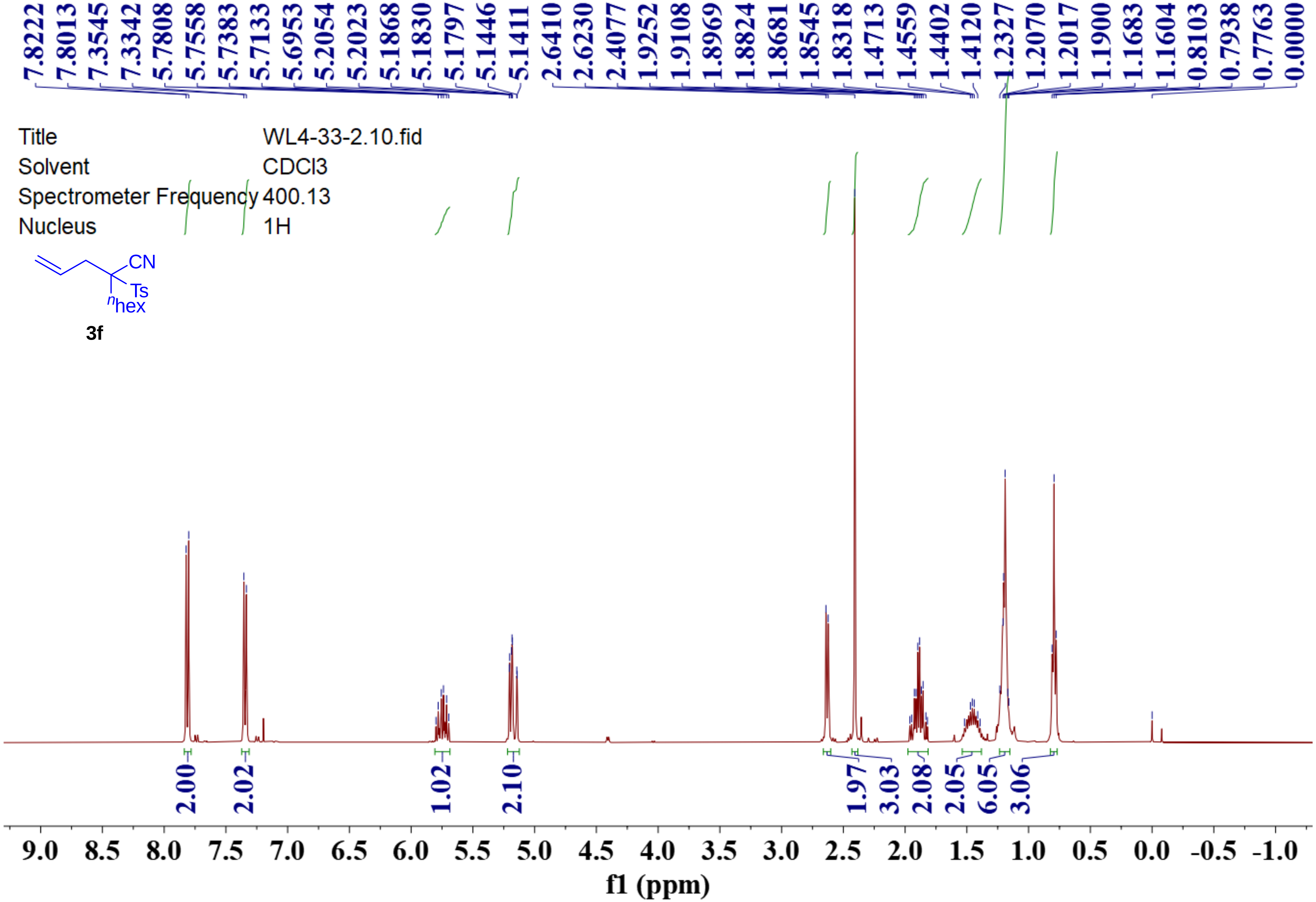
3d

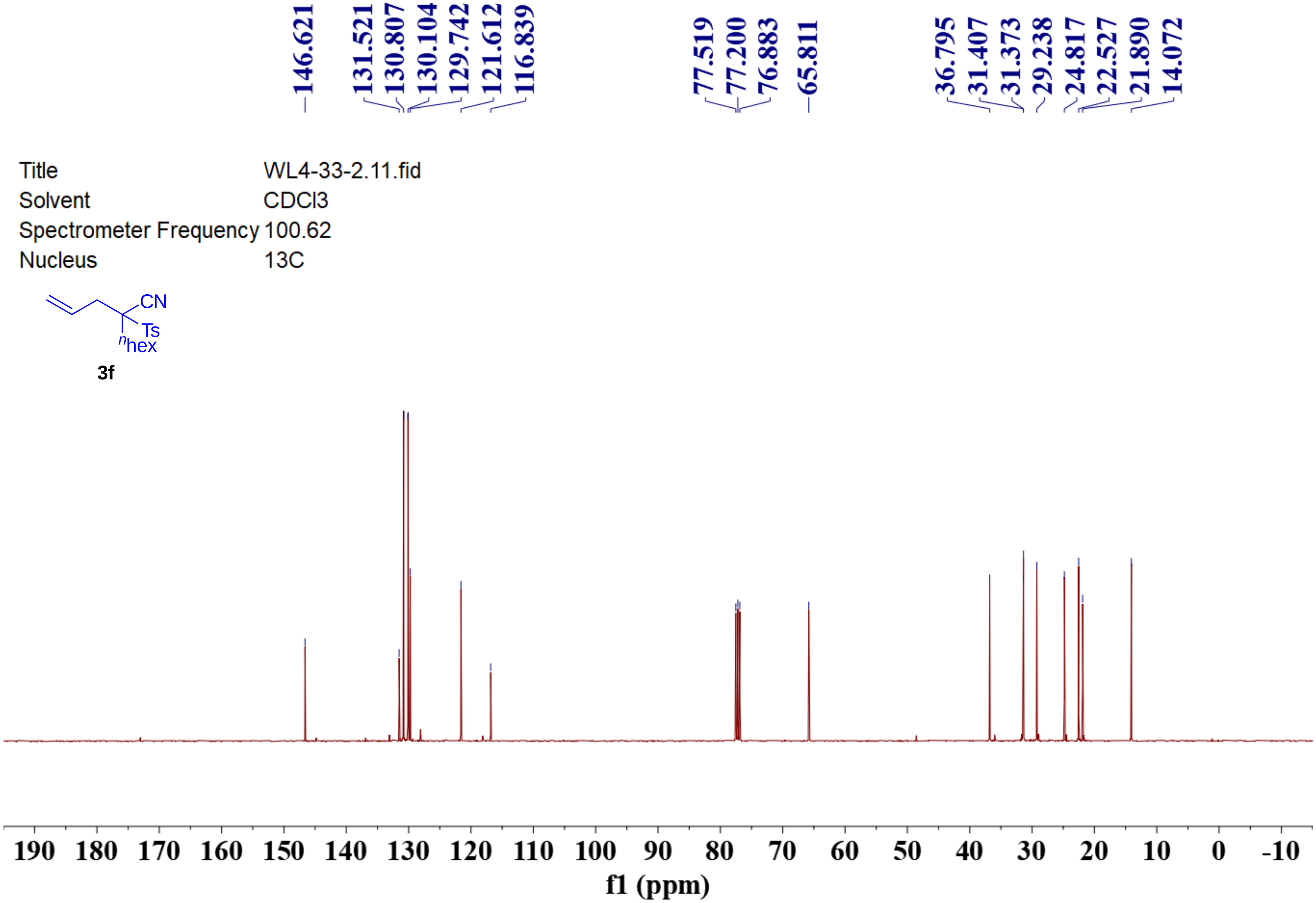

151.836
139.423
132.557
128.406
124.638
123.371
116.707
77.519
77.200
76.881
62.058
37.078
18.294
Title WL4-21.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ns
Me
3d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.9014
7.8805
7.4360
7.4157
5.8763
5.8582
5.8333
5.8158
5.7908
5.7728
5.2852
5.2818
5.2629
5.2594
5.2244
5.2207
2.7147
2.6977
2.4886
2.0237
2.0101
1.9882
1.9600
1.9450
1.9130
1.8903
1.8776
1.6578
1.6388
1.6101
1.5915
1.5666
1.5381
1.5195
0.9615
0.9433
0.9251
0.0000
Title WL4-38.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Ts
^{n}Pr
3e
2.00
2.00
1.02
2.08
2.02
2.98
2.09
2.07
3.06
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 -0.5 -1.0
f1 (ppm)


146.643
131.472
130.800
130.117
129.720
121.634
116.799
77.518
77.200
76.883
65.764
36.831
33.404
21.899
18.450
14.084
Title WL4-38.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ts
^{n}Pr
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3e


7.8222
7.8013
7.3545
7.3342
5.7808
5.7558
5.7383
5.7133
5.6953
5.2054
5.2023
5.1868
5.1830
5.1797
5.1446
5.1411
2.6410
2.6230
2.4077
1.9252
1.9108
1.8969
1.8824
1.8681
1.8545
1.8318
1.4713
1.4559
1.4402
1.4120
1.2327
1.2070
1.2017
1.1900
1.1683
1.1604
0.8103
0.7938
0.7763
0.0000
Title WL4-33-2.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Ts
^{n}hex
3f
2.00
2.02
1.02
2.10
1.97
3.03
2.08
2.05
6.05
3.06
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
-0.5
-1.0
f1 (ppm)



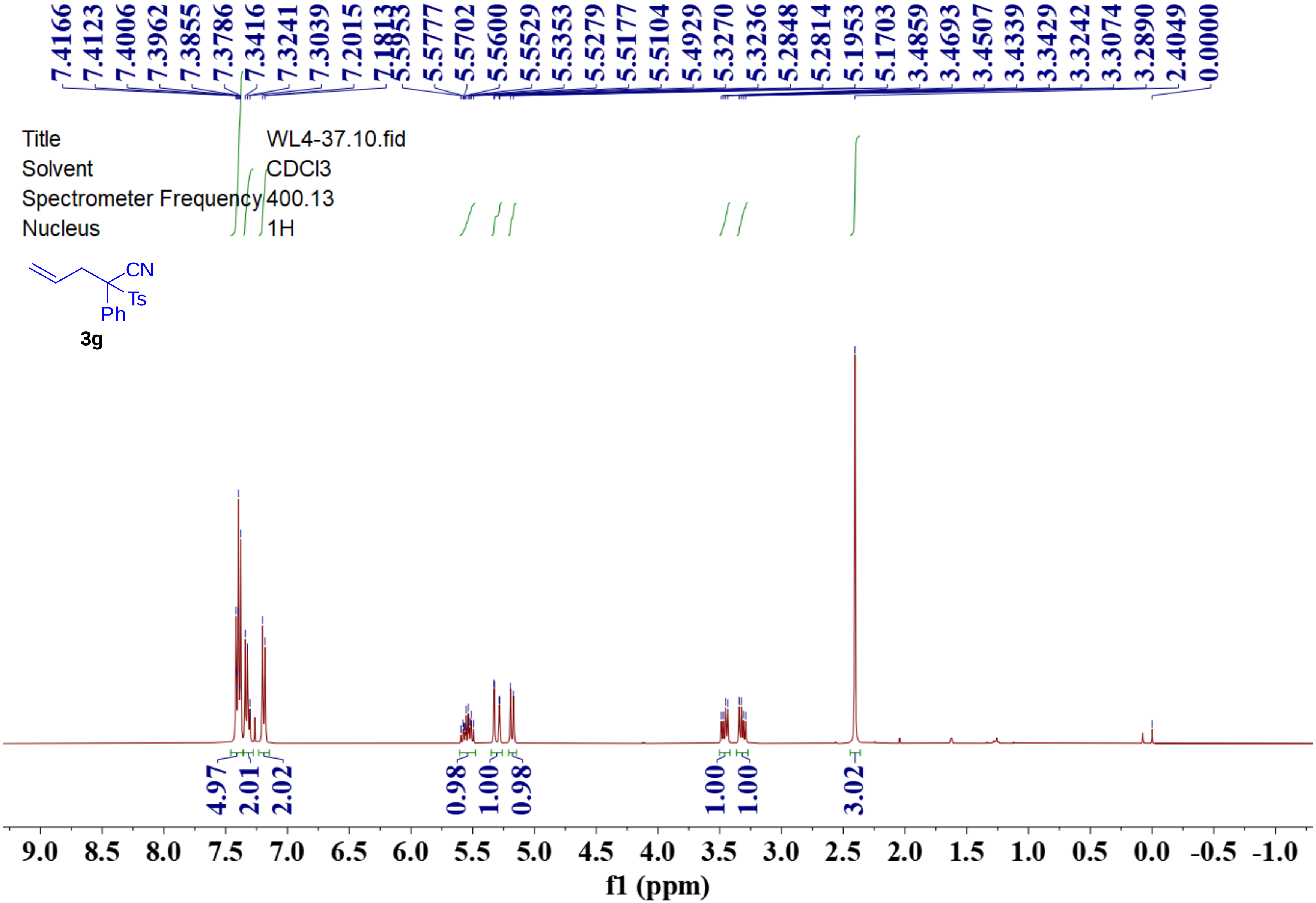
Title WL4-33-2.11.fid

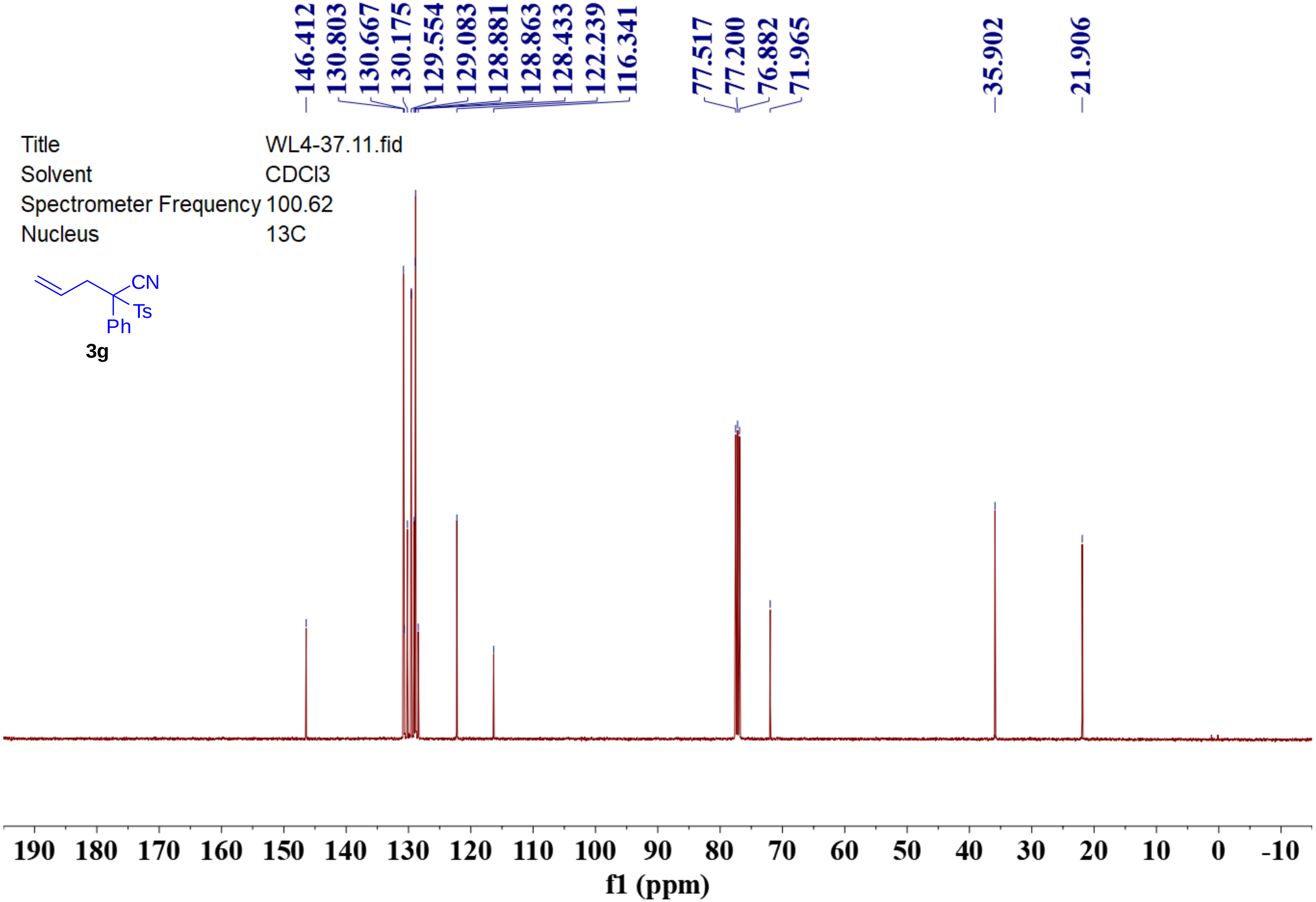
Solvent CDCl3

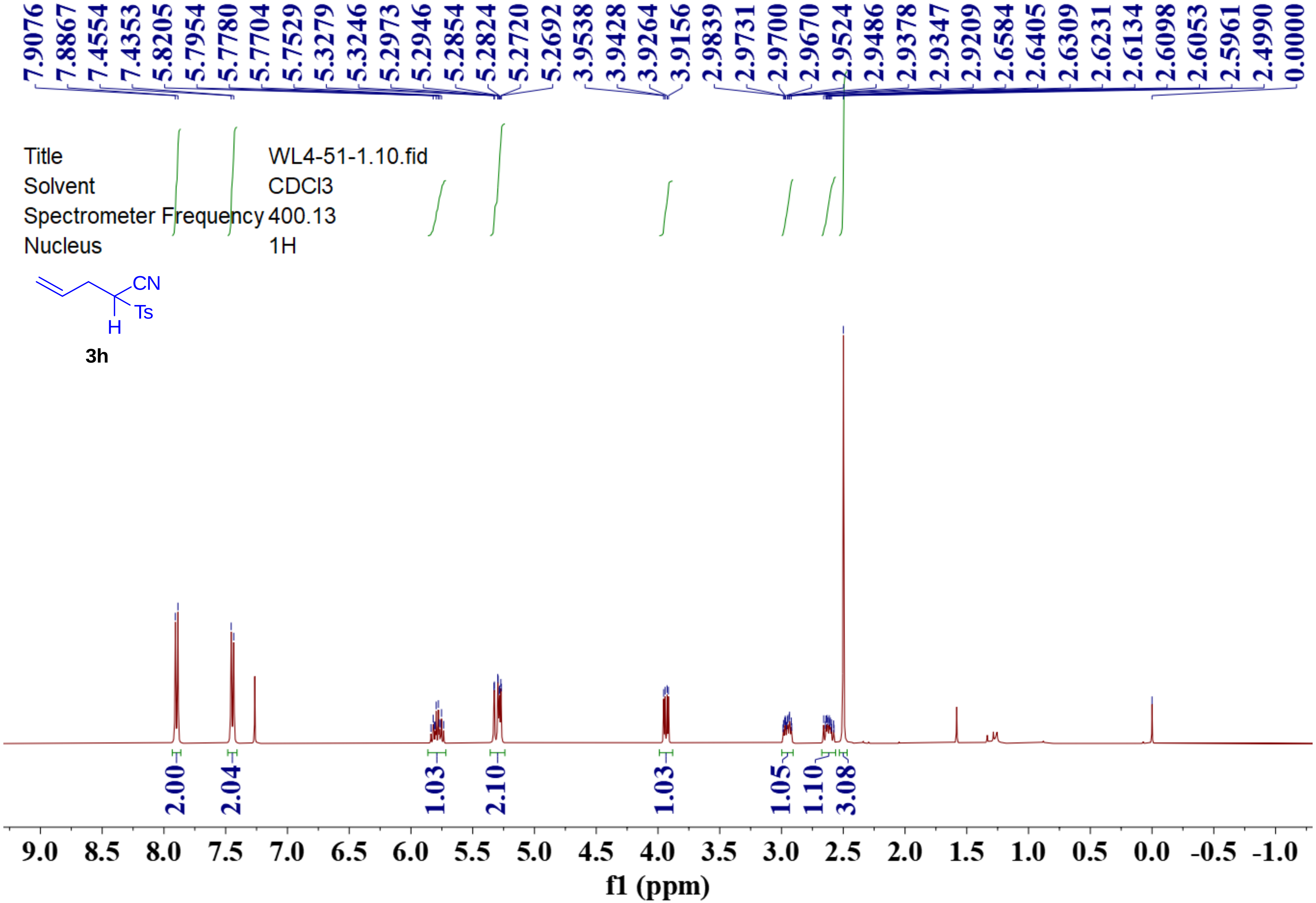
Spectrometer Frequency 100.62

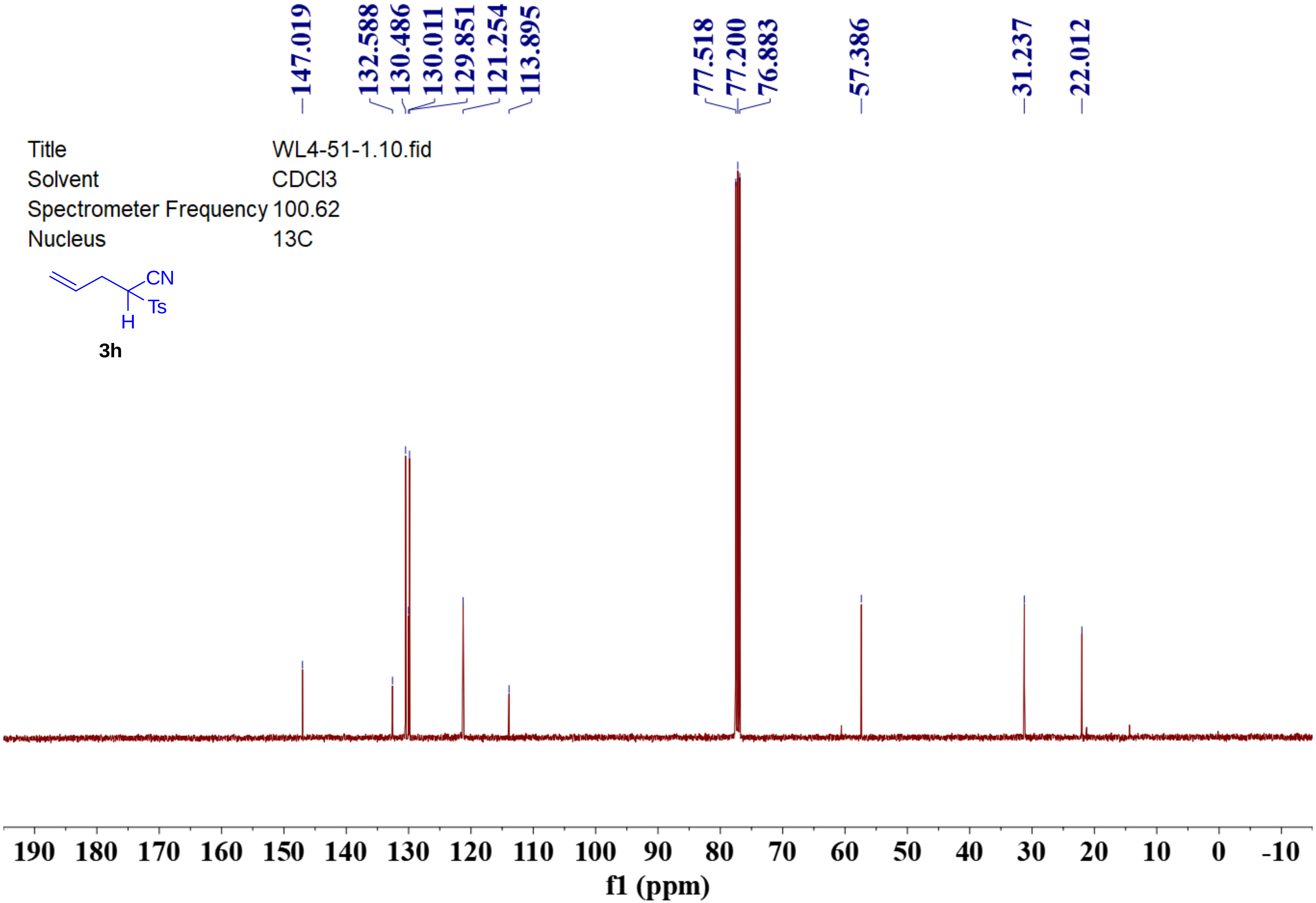
Nucleus 13C

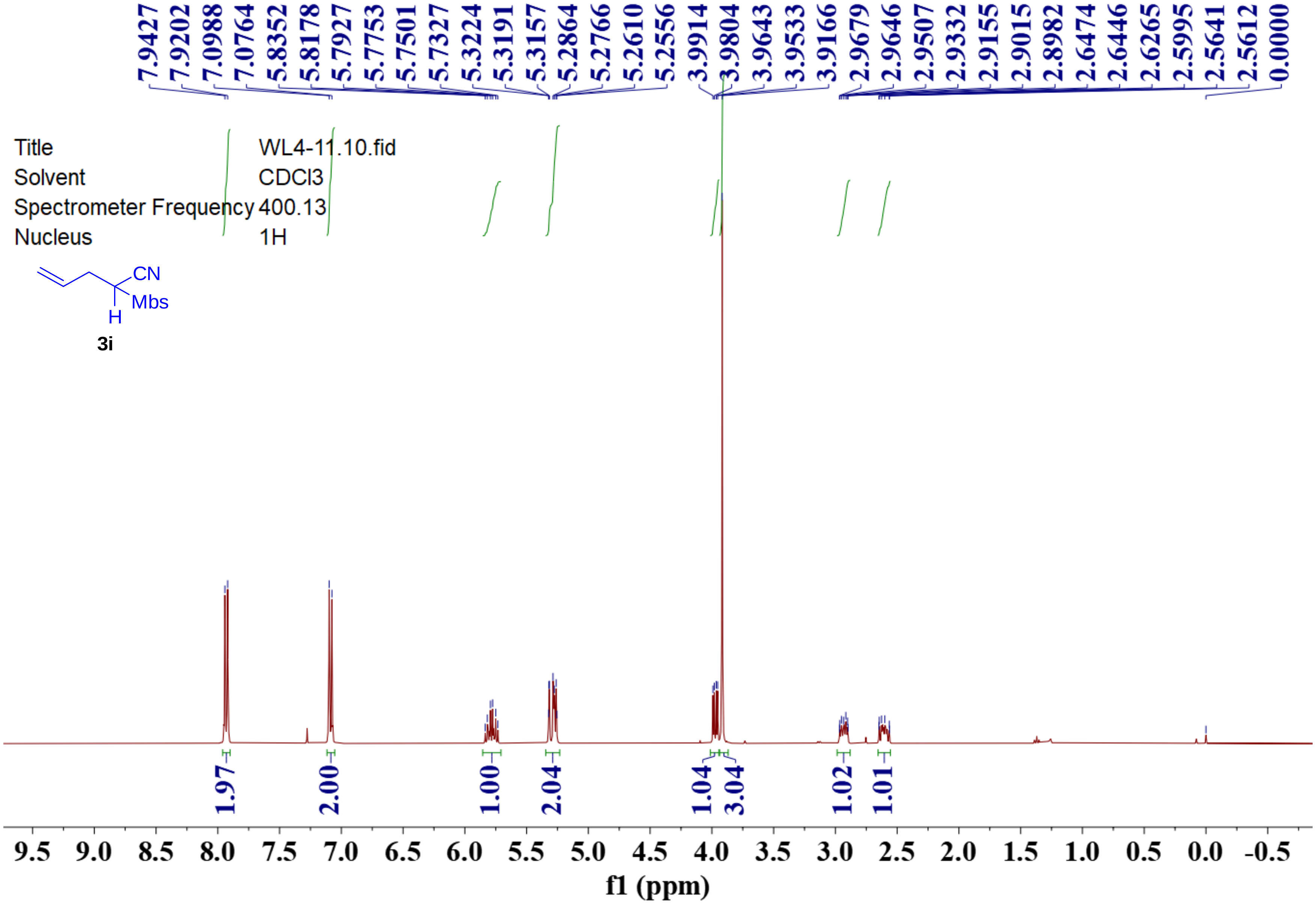
3f


7.4166
7.4123
7.4006
7.3962
7.3855
7.3786
7.3416
7.3241
7.3039
7.2015
7.1813
5.5953
5.5777
5.5702
5.5600
5.5529
5.5353
5.5279
5.5177
5.5104
5.4929
5.3270
5.3236
5.2848
5.2814
5.1953
5.1703
3.4859
3.4693
3.4507
3.4339
3.3429
3.3242
3.3074
3.2890
2.4049
0.0000
Title WL4-37.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Ts
Ph
3g
4.97
2.01
2.02
0.98
1.00
0.98
1.00
1.00
3.02
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
-0.5
-1.0
f1 (ppm)

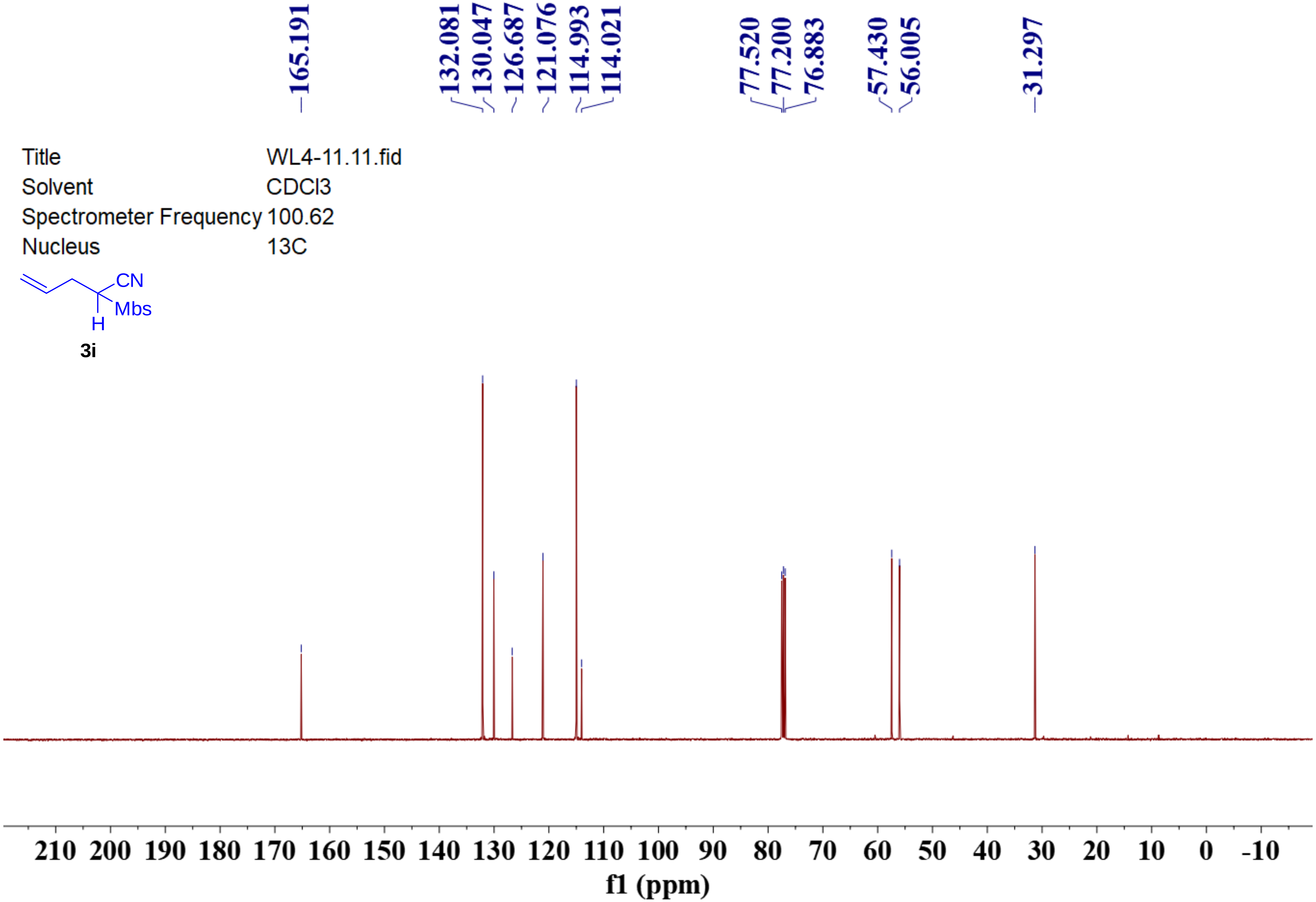

146.412
130.803
130.667
130.175
129.554
129.083
128.881
128.863
128.433
122.239
116.341
77.517
77.200
76.882
71.965
35.902
21.906
Title WL4-37.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ts
Ph
3g
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.9076
7.8867
7.4554
7.4353
5.8205
5.7954
5.7780
5.7704
5.7529
5.3279
5.3246
5.2973
5.2946
5.2854
5.2824
5.2720
5.2692
3.9538
3.9428
3.9264
3.9156
2.9839
2.9731
2.9700
2.9670
2.9524
2.9486
2.9378
2.9347
2.9209
2.6584
2.6405
2.6309
2.6231
2.6134
2.6098
2.6053
2.5961
2.4990
0.0000
Title WL4-51-1.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Ts
H
3h
2.00
2.04
1.03
2.10
1.03
1.05
1.10
3.08
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
-0.5
-1.0
f1 (ppm)


147.019
132.588
130.486
130.011
129.851
121.254
113.895
77.518
77.200
76.883
57.386
31.237
22.012
Title WL4-51-1.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ts
H
3h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

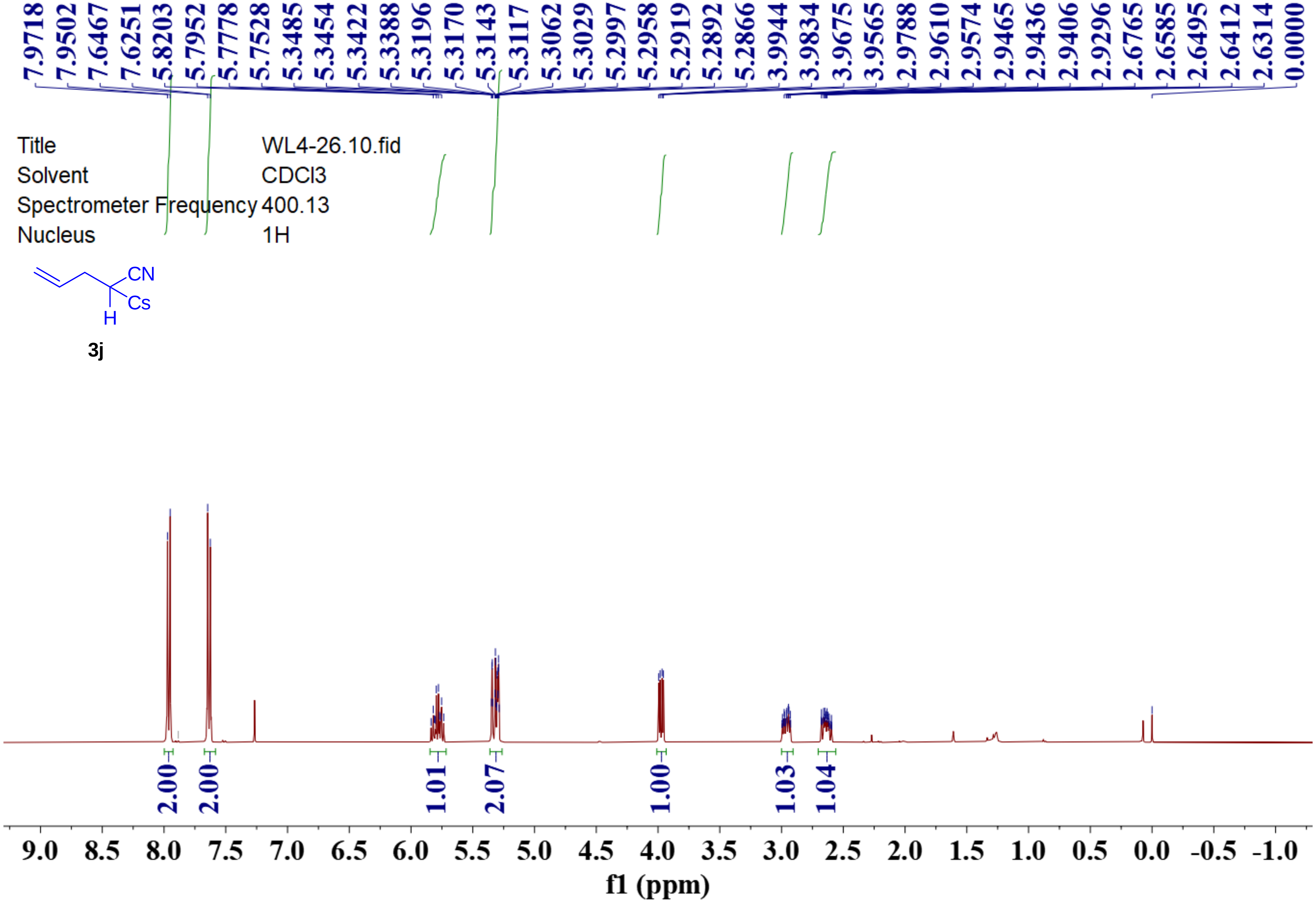


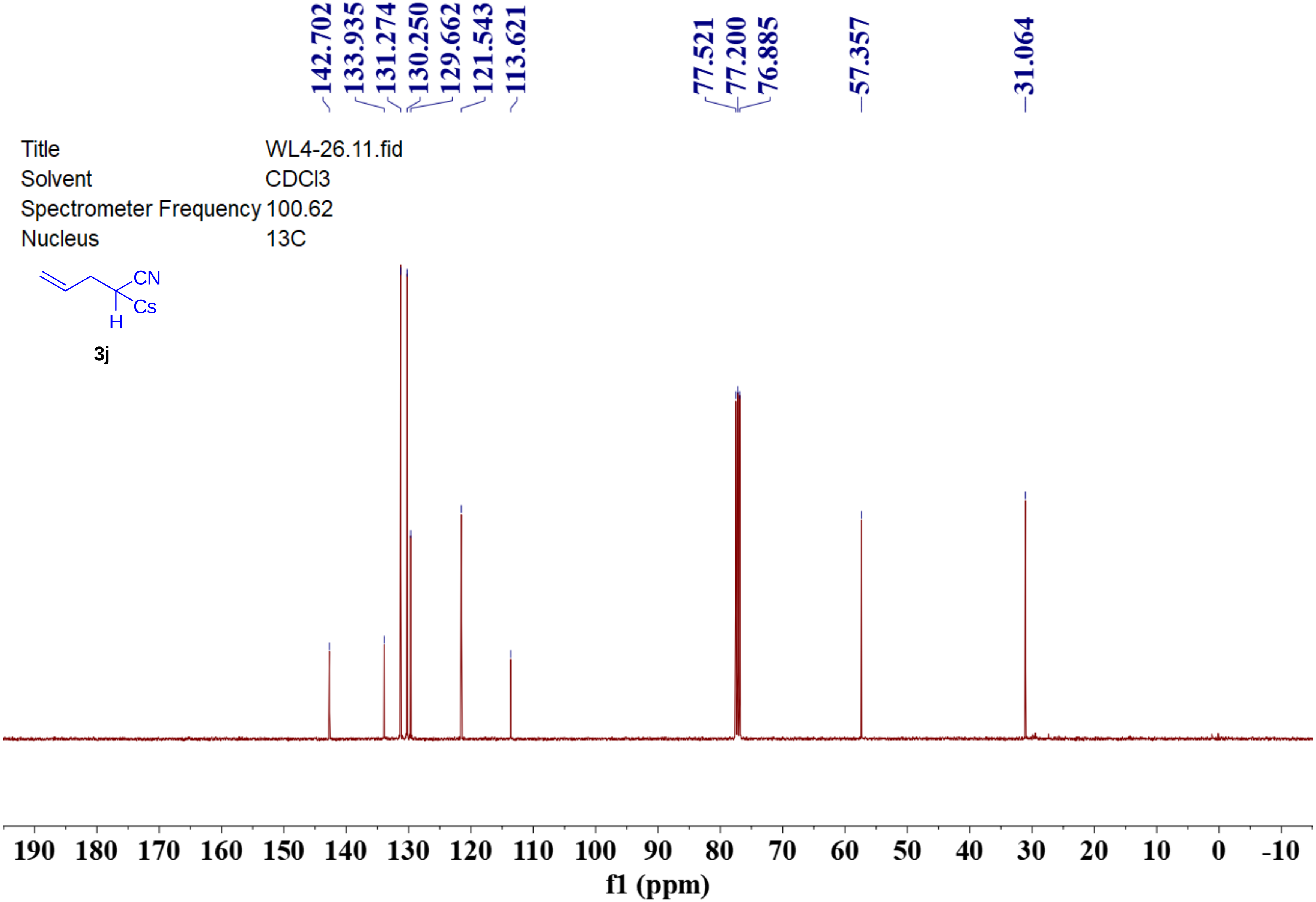
Title	WL4-11.10.fid
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

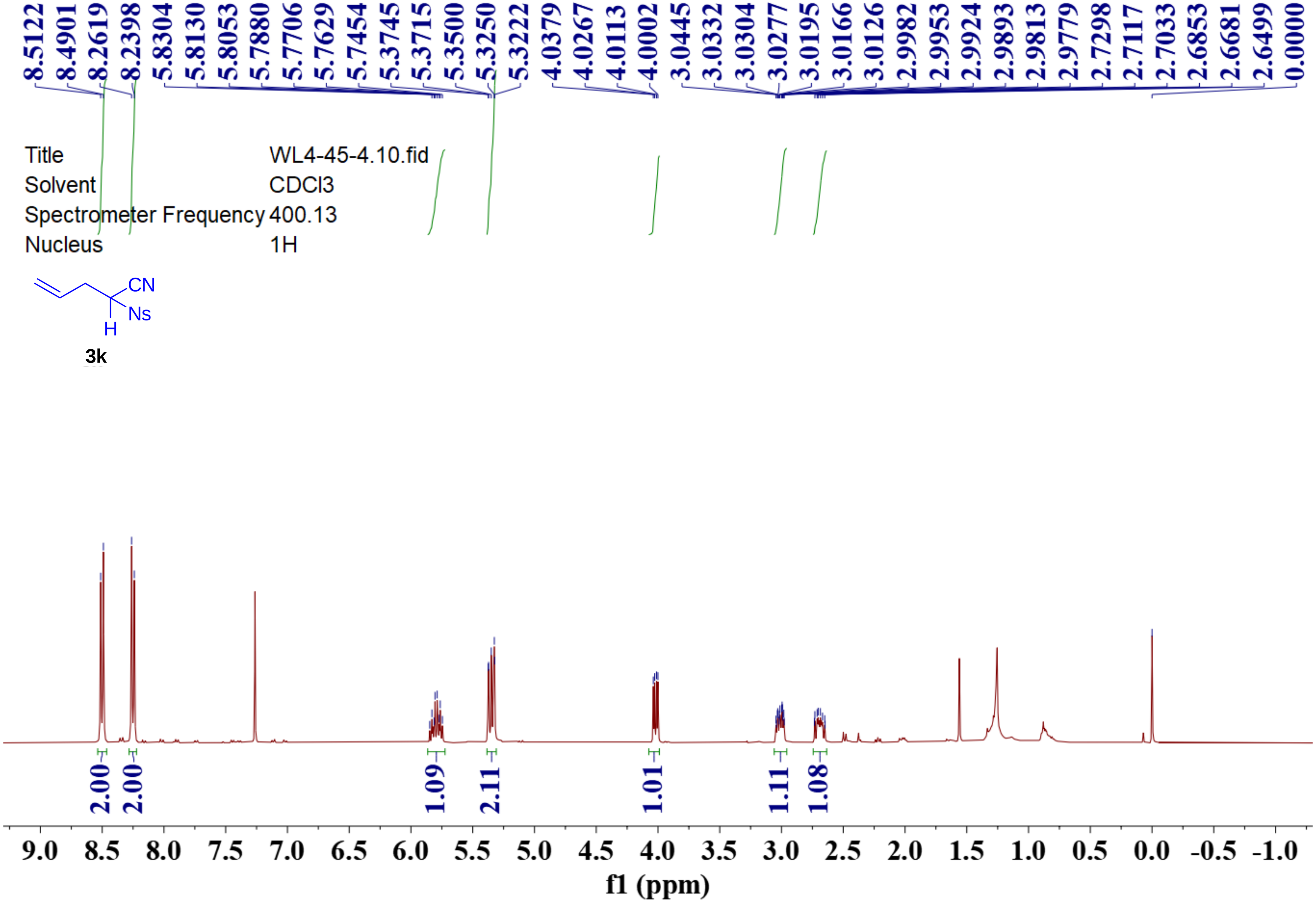


Title	WL4-11.11.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C

3i

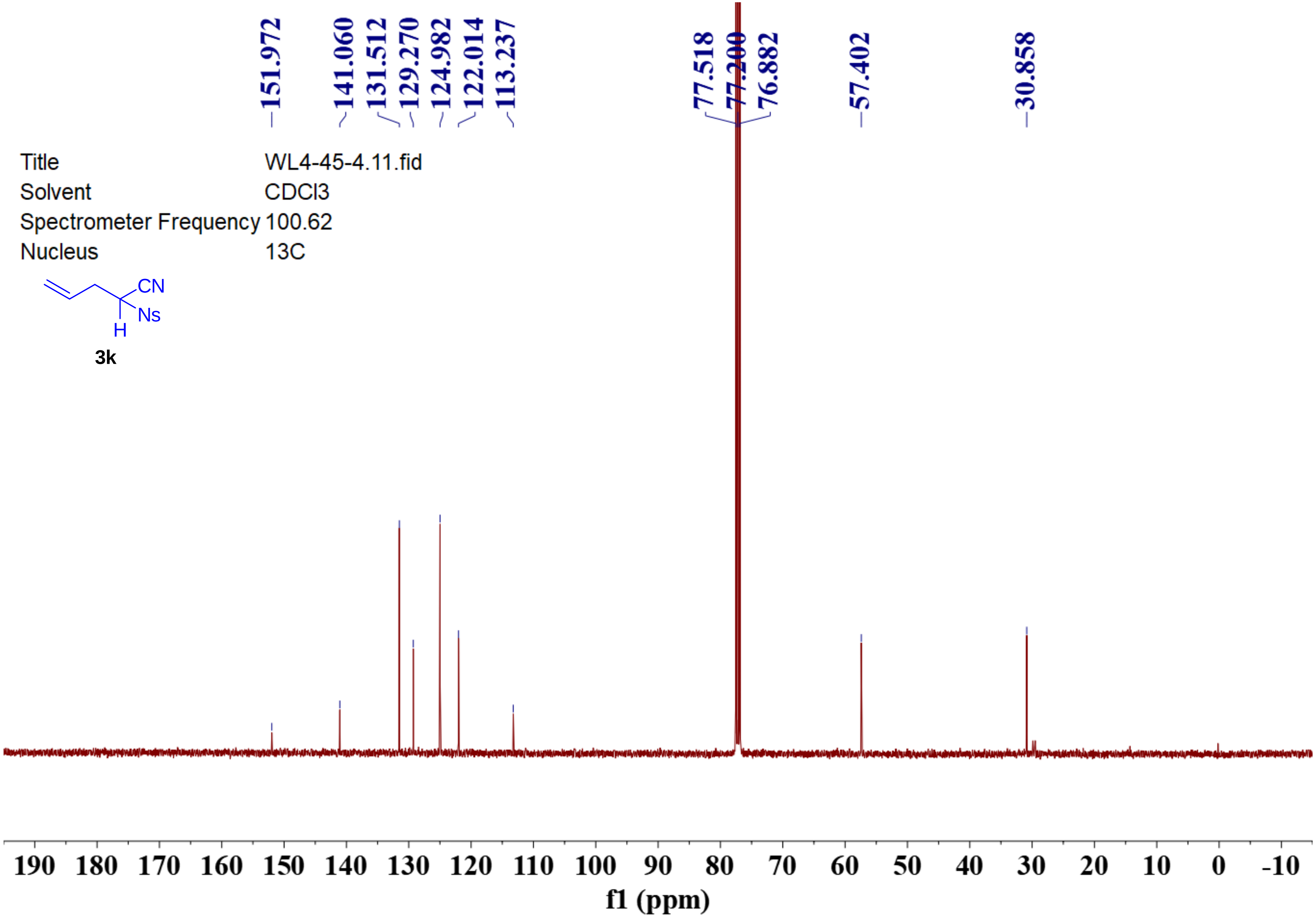

7.9718
7.9502
7.6467
7.6251
5.8203
5.7952
5.7778
5.7528
5.3485
5.3454
5.3422
5.3388
5.3196
5.3170
5.3143
5.3117
5.3062
5.3029
5.2997
5.2958
5.2919
5.2892
5.2866
3.9944
3.9834
3.9675
3.9565
2.9788
2.9610
2.9574
2.9465
2.9436
2.9406
2.9296
2.6765
2.6585
2.6495
2.6412
2.6314
0.0000
Title
WL4-26.10.fid
Solvent
CDCl3
Spectrometer Frequency
400.13
Nucleus
1H
CN
Cs
H
3j
2.00
2.00
1.01
2.07
1.00
1.03
1.04
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
-0.5
-1.0
f1 (ppm)

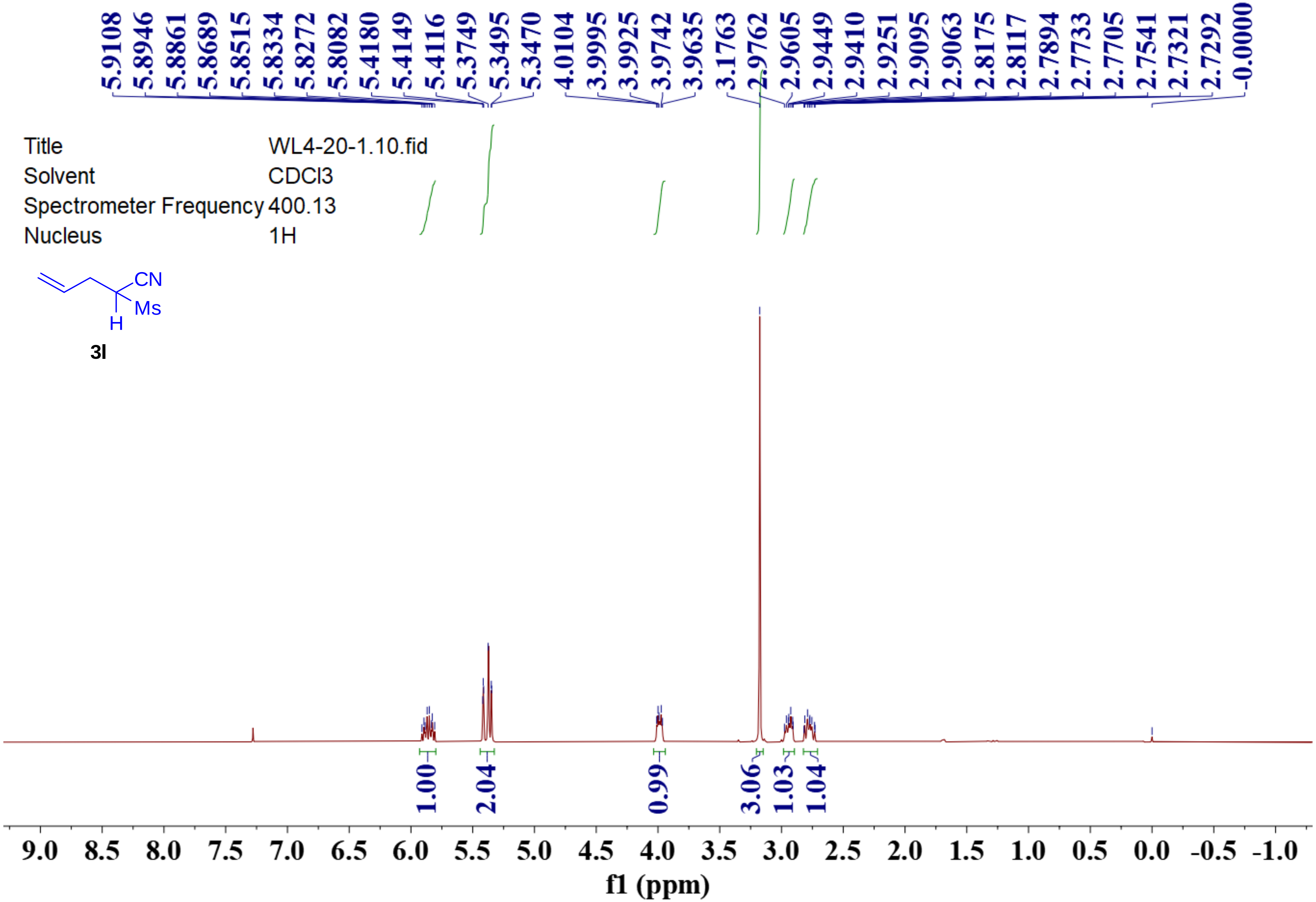

142.702
133.935
131.274
130.250
129.662
121.543
113.621
77.521
77.200
76.885
57.357
31.064
Title WL4-26.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Cs
H
3j
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

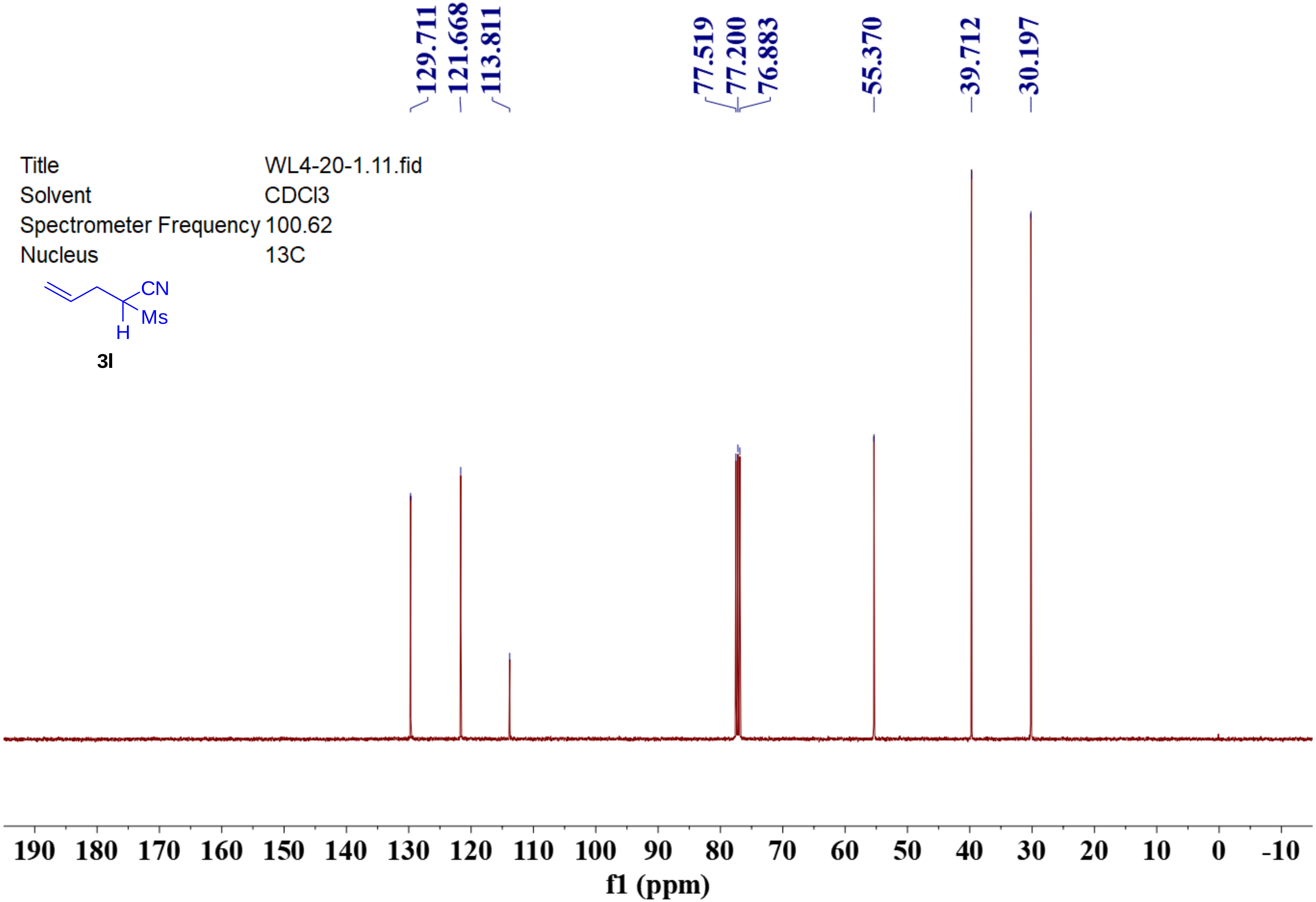


Title	WL4-45-4.10.fid
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

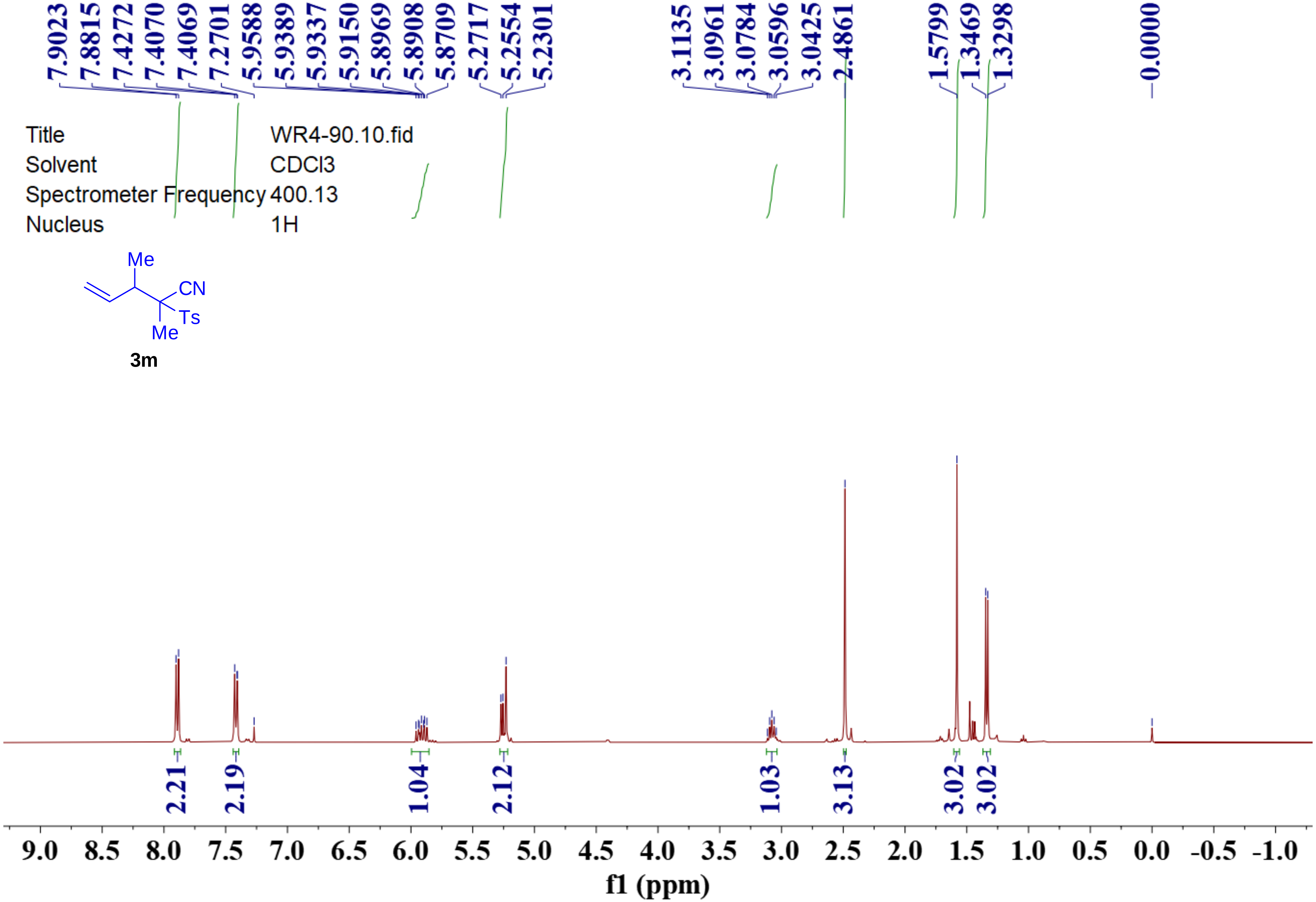
3k


151.972
141.060
131.512
129.270
124.982
122.014
113.237
77.518
77.200
76.882
57.402
30.858
Title WL4-45-4.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ns
H
3k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


5.9108
5.8946
5.8861
5.8689
5.8515
5.8334
5.8272
5.8082
5.4180
5.4149
5.4116
5.3749
5.3495
5.3470
4.0104
3.9995
3.9925
3.9742
3.9635
3.1763
2.9762
2.9605
2.9449
2.9410
2.9251
2.9095
2.9063
2.8175
2.8117
2.7894
2.7733
2.7705
2.7541
2.7321
2.7292
-0.0000
Title WL4-20-1.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
CN
Ms
H
3l
1.00
2.04
0.99
3.06
1.03
1.04
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
-0.5
-1.0
f1 (ppm)

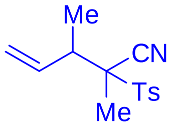

129.711
121.668
113.811
77.519
77.200
76.883
55.370
39.712
30.197
Title WL4-20-1.11.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C
CN
Ms
H
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3l

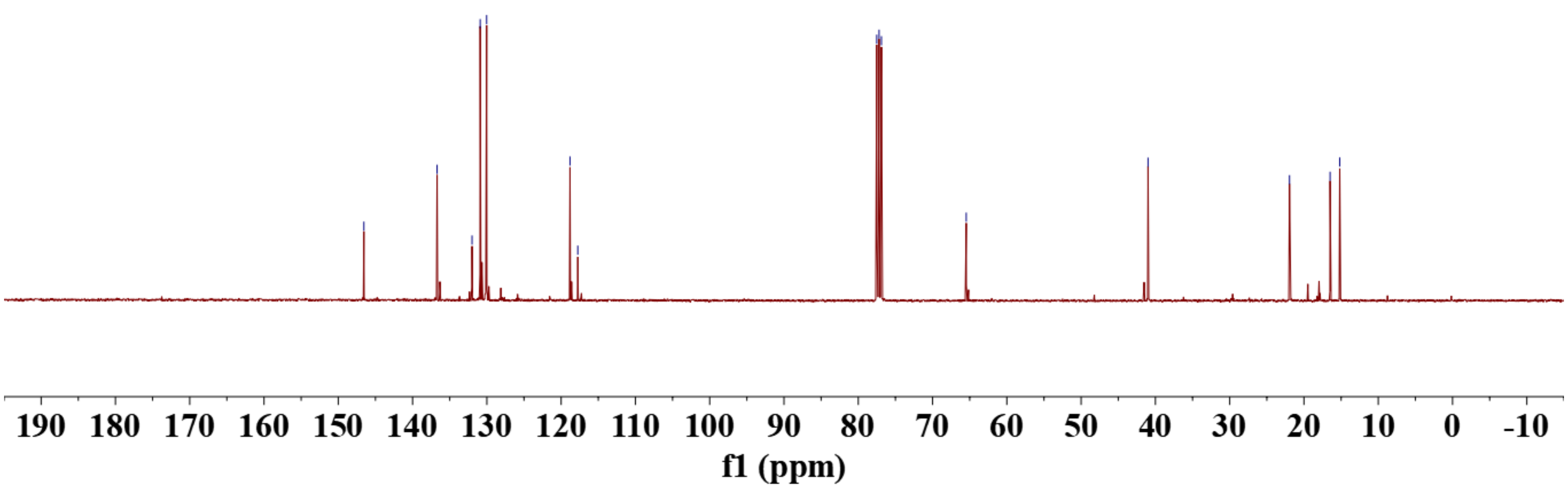

7.9023
7.8815
7.4272
7.4070
7.4069
7.2701
5.9588
5.9389
5.9337
5.9150
5.8969
5.8908
5.8709
5.2717
5.2554
5.2301
3.1135
3.0961
3.0784
3.0596
3.0425
2.4861
1.5799
1.3469
1.3298
-0.0000
Title WR4-90.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Me
CN
Ts
Me
2.21
2.19
1.04
2.12
1.03
3.13
3.02
3.02
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
-0.5
-1.0
f1 (ppm)

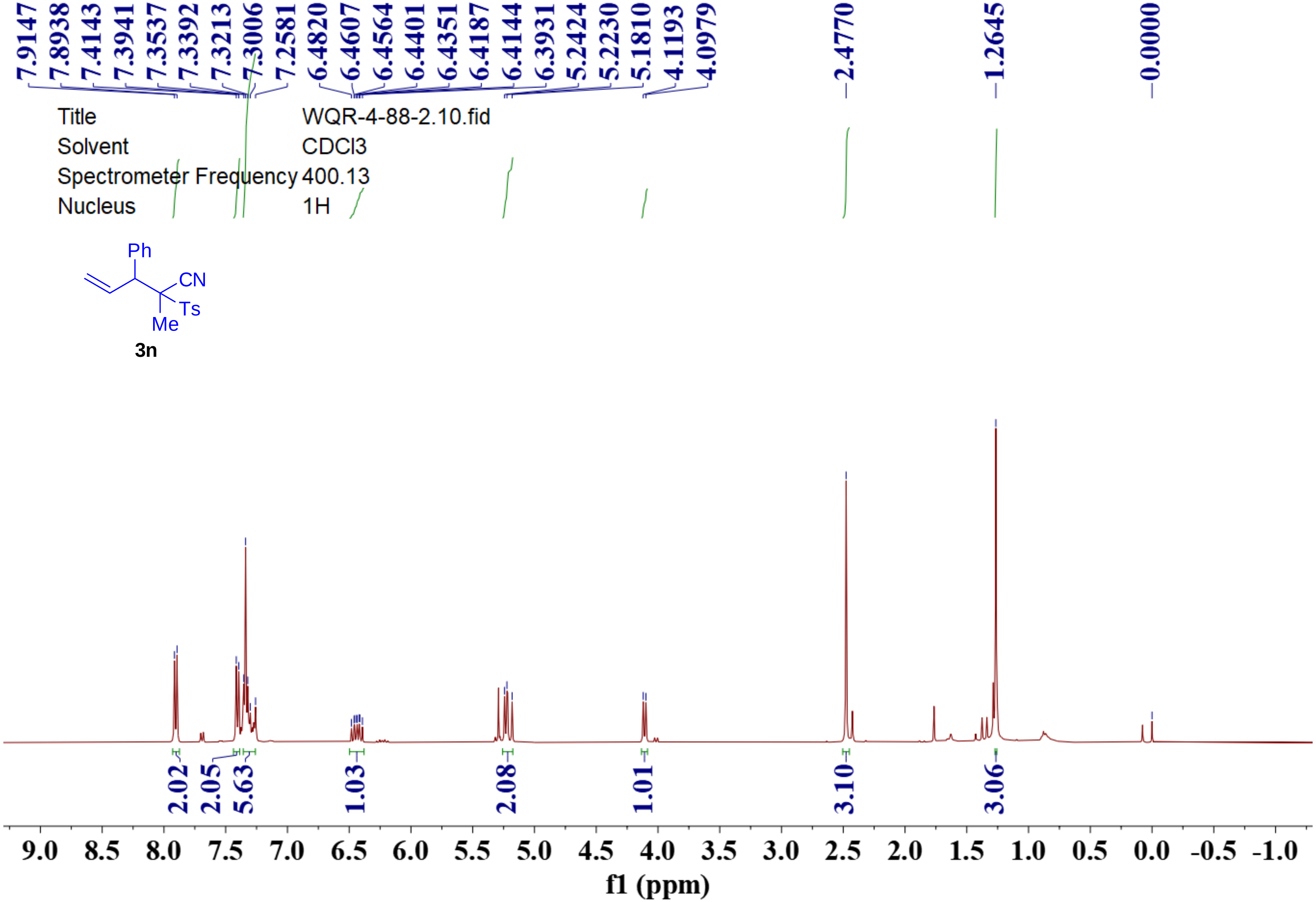
3m

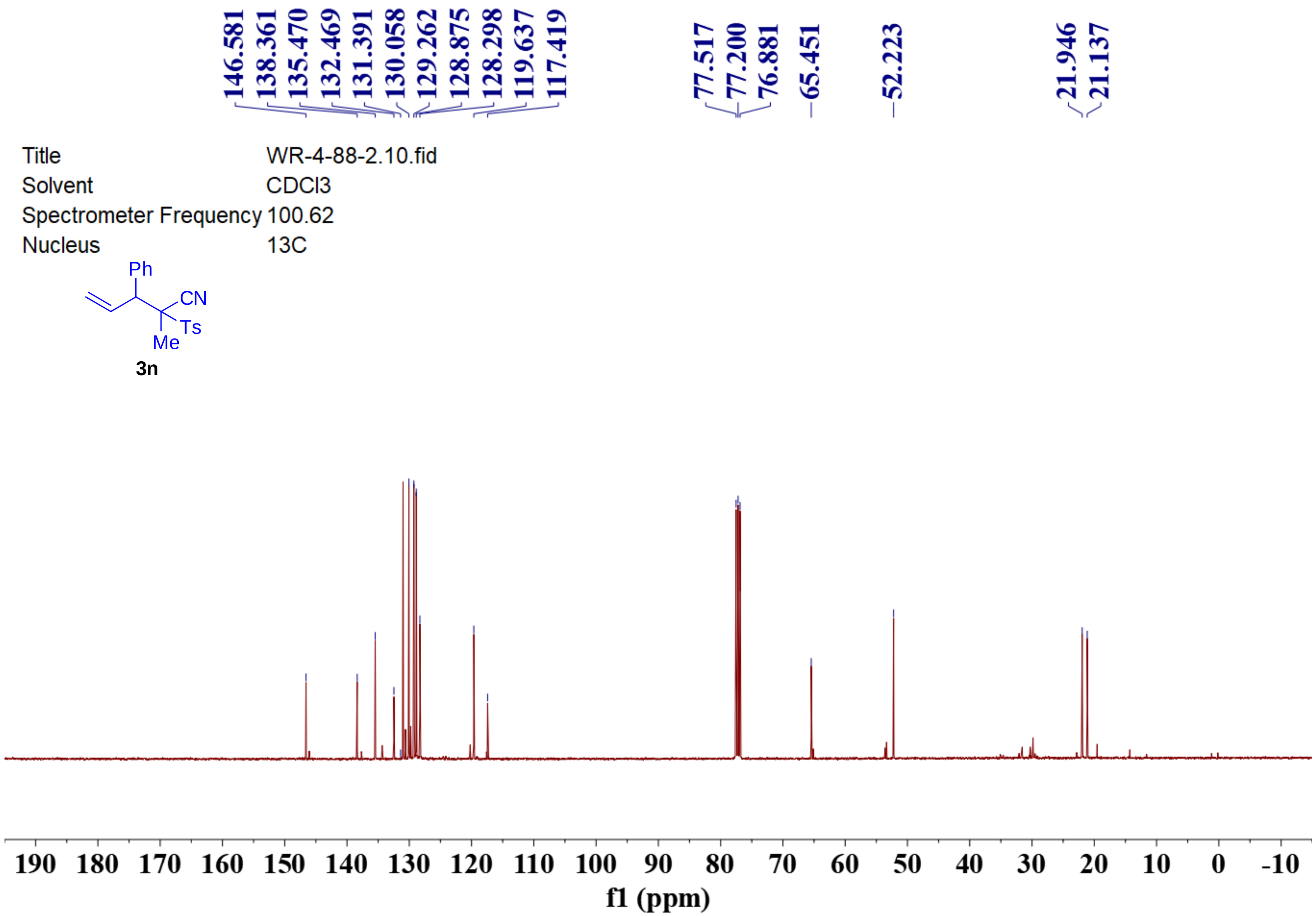
Title	WR4-90.10.fid
Solvent	CDCl3
Spectrometer Frequency	100.62
Nucleus	13C



3m

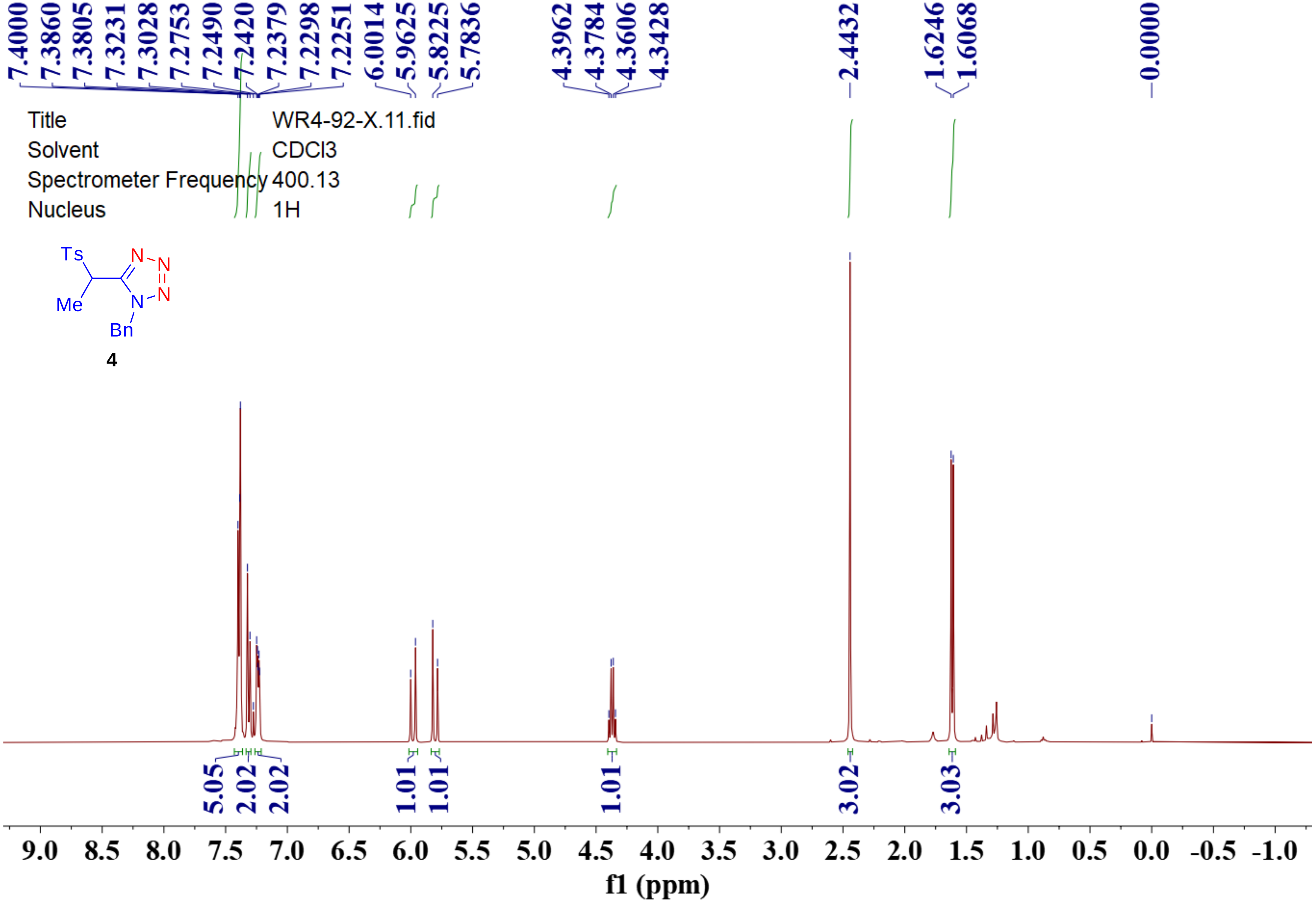



7.9147
7.8938
7.4143
7.3941
7.3537
7.3392
7.3213
7.3006
7.2581
6.4820
6.4607
6.4564
6.4401
6.4351
6.4187
6.4144
6.3931
5.2424
5.2230
5.1810
4.1193
4.0979
2.4770
1.2645
0.0000
Title WQR-4-88-2.10.fid
Solvent CDCl3
Spectrometer Frequency 400.13
Nucleus 1H
Ph
CN
Ts
Me
3n
2.02
2.05
5.63
1.03
2.08
1.01
3.10
3.06
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
-0.5
-1.0
f1 (ppm)



Title WR-4-88-2.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C

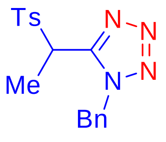
3n



Title	WR4-92-X.11.fid
Solvent	CDCl3
Spectrometer Frequency	400.13
Nucleus	1H

4

Title WR4-92-X.10.fid
Solvent CDCl3
Spectrometer Frequency 100.62
Nucleus 13C



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