Supporting Information

A facile access to *N*-sulfonylthioimidates and their use for the transformation to 3,4-dihydroquinazolines

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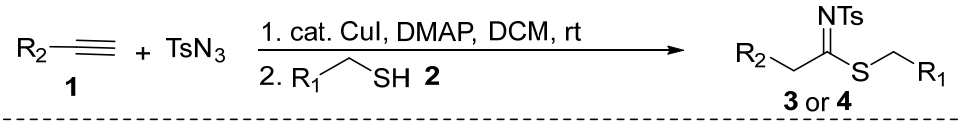
1. General Information:

Unless otherwise specified, all reactions were carried out under an atmosphere of nitrogen in flame-dried reaction vessels. The **1a** and **1g** were purchased from Acros and were used without further purification. The alkyne derivatives (**1b**-**1f**) were synthesized from the corresponding trimethylsilylacetylene following the literature procedure.[1] The **2o** was purchased from Aldrich and was used without further purification. The thiol molecules (**2a**-**2n, 2p**-**2r**) were synthesized by following the literature procedure.[2]

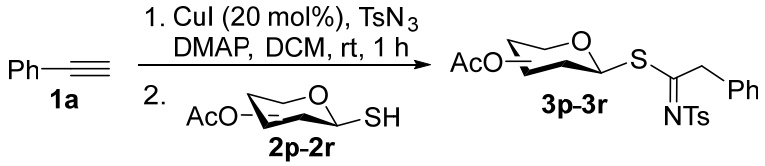
^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz), and ^{13}C DEPT (100 MHz) spectra were recorded on a Varian 400 MHz spectrometer. The NMR spectra were recorded in $CDCl_3$ or $(CD_3)_2CO$. *d*-Chloroform (δ 7.26 ppm in ^{1}H NMR; δ 77.0 ppm in ^{13}C NMR) and *d*-acetone (δ 2.05 ppm in ^{1}H NMR; δ 29.84 ppm in ^{13}C NMR) were used as internal standards. The IR spectra were measured on a Thermo Scientific Nicolet iS5 FT-IR spectrophotometer. Thin layer chromatography (TLC) was performed on Merck silica gel plates 60 F_{254} (0.25 mm). TLC plates were visualized under UV light (254 or 365 nm) and by treatment with *p*-anisaldehyde, $KMnO_4$ or cerium molybdate staining solution followed by heating. Flash column chromatography was carried out by using silica gel 60 (230–400 mesh, E. Merck). High-resolution mass spectrometry (HRMS) data were recorded on EI source. Melting points were measured by Thermo Scientific ‘‘Mel-Temp’’ type melting point apparatus and are uncorrected. Splitting patterns are reported as follows: s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; br, broad; dd, doublet of doublets; dt, doublet of triplets; td, triplet of doublets; tt, triplet of triplets. Coupling constants (J) are reported in Hz. Yields of products refer to chromatographically purified products unless otherwise stated.

2. General Procedure I:



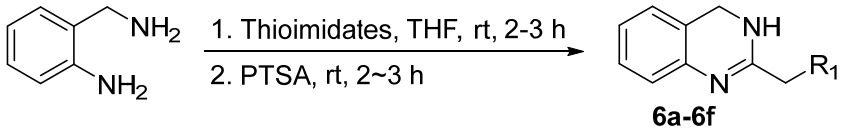
To a solution of terminal alkyne **1** (1.0 equiv) in DCM (0.3 M) was added *p*-toluenesulfonyl azide (1.2 equiv), DMAP (1.0 equiv) and CuI (0.2 equiv). The resulting solution was stirred at room temperature under nitrogen atmosphere for 1 hour. Then fresh prepared thiol **2** (3.0 equiv) was added to the reaction mixture and the reaction was stirred for several hours until the reaction was complete as indicated by TLC. The mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography to afford the desired products **3a**-**3o** and **4b**-**4n**.

3. General Procedure II:



To a solution of terminal alkyne **1a** (1.0 equiv) in DCM (0.3 M) was added *p*-toluenesulfonyl azide (1.2 equiv), DMAP (1.0 equiv) and CuI (0.2 equiv). The resulting solution was stirred at room temperature under nitrogen atmosphere for 1 hour. Then fresh prepared β-glycosyl 1-thiol **2p**-**2r** (1.5 equiv) were added to the reaction mixture and the reaction was stirred for 0.5 h-1.5 h. The mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography to afford the desired products **3p**-**3r**.

4. General Procedure III:

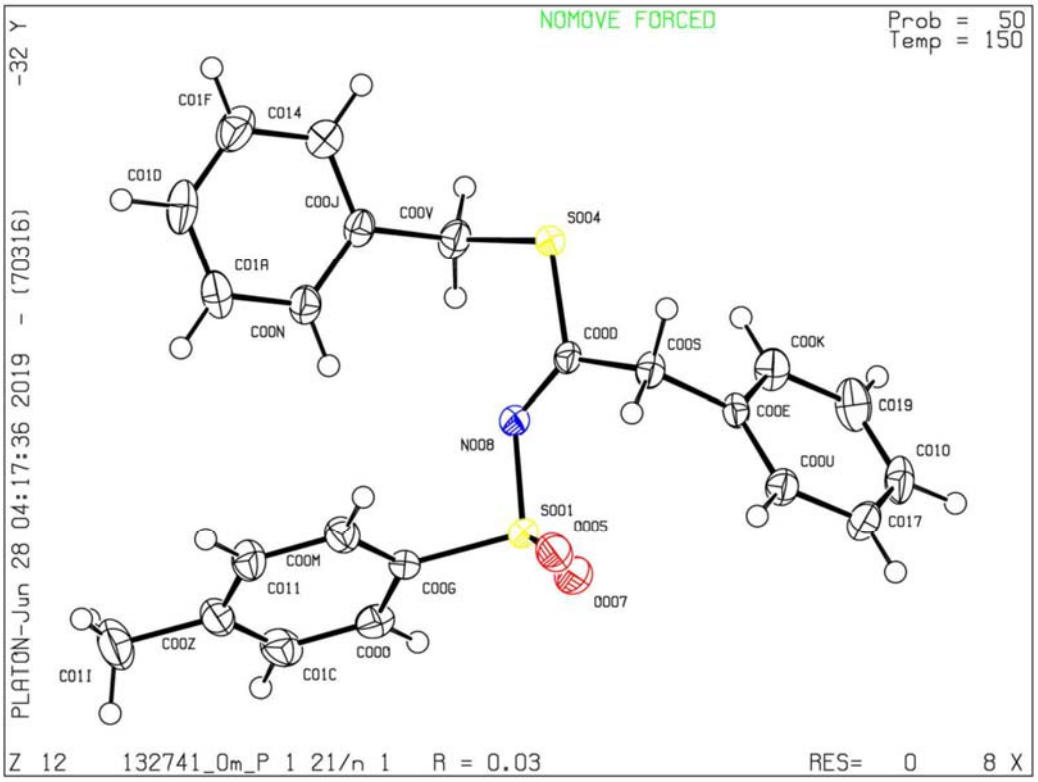


To a solution of thioimidates (1.0 equiv) in THF (0.3 M) was added 2-aminobenzylamine (1.2 equiv) stirred for 2-3 hours at room temperature under nitrogen atmosphere. Then, the *p*-toluenesulfonic acid monohydrate (2.0 equiv) was added to the reaction mixture and stirred for 2-3 hours until the reaction was complete as indicated by TLC. After the reaction was completed, the mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography to afford the desired products **6a-6f**.

X-ray Crystallographic Studies and the X-ray Data of 3b

Single crystals of compound **3b** suitable for X-ray diffraction measurements was mounted on the Bruker D8 VENTURE and the unit cell was determined using Bruker SMART APEX 3 software suite to employ graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å), and intensity data were collected with ω scans. The data collection and reduction were performed with the CrysAlisPro software, and the absorptions were corrected by the SCALE3 ABSPACK multiscan method. The space-group

determination was based on a check of the Laue symmetry and systematic absences, and it was confirmed using the structure solution. The structure was solved and refined with the Olex2 1.2-ac21 package. Anisotropic thermal parameters were used for all non-H atoms, and fixed isotropic parameters were used for H atoms. CCDC 2004076 (**3b**) contains the supplementary crystallographic data for this paper. The checkCIF report is given below:



Datablock: 132741_0m_pl

Bond precision: C-C = 0.0022 A Wavelength=0.71076

Cell: a=8.0525(5) b=15.8806(10) c=15.2504(10)
alpha=90 beta=94.189(2) gamma=90
Temperature: 150 K

	Calculated	Reported
Volume	1945.0(2)	1945.0(2)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C22 H21 N O2 S2	C22 H21 N O2 S2
Sum formula	C22 H21 N O2 S2	C22 H21 N O2 S2
Mr	395.52	395.52
Dx,g cm-3	1.351	1.351
Z	4	4
Mu (mm-1)	0.291	0.291
F000	832.0	832.0
F000'	833.29	
h,k,lmax	10,19,19	10,19,19
Nref	3974	3899
Tmin,Tmax		0.682,0.745
Tmin'		

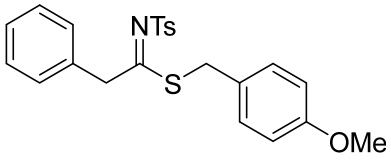
Correction method= # Reported T Limits: Tmin=0.682 Tmax=0.745
AbsCorr = MULTI-SCAN

Data completeness= 0.981 Theta(max)= 26.402

R(reflections)= 0.0333(3597) wR2(reflections)= 0.1018(3899)

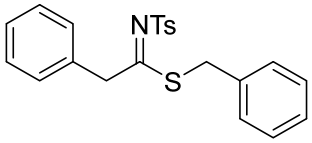
S = 1.126 Npar= 245

4-Methoxybenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3a)



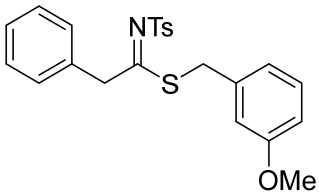
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (4-methoxyphenyl)methanethiol (226.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3a** (167.0 mg, 80% yield) as a yellow solid. m.p. 59-60 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.36-7.30 (m, 7H), 6.99 (d, *J* = 8.8, 2H), 6.70 (d, *J* = 8.8, 2H), 4.44 (s, 2H), 3.94 (s, 2H), 3.76 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.6, 158.9, 143.5, 138.5, 133.7, 130.2, 129.4, 129.0, 128.5, 127.6, 127.0, 113.8, 55.1, 44.1, 35.8, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{23}NO_3S_2$: 425.1119, found: 425.1116.

Benzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3b)



Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and phenylmethanethiol (182.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3b** (143.0 mg, 74% yield) as a yellow solid. m.p. 69-71 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.3, 2H), 7.35-7.30 (m, 7H), 7.21-7.15 (m, 3H), 7.07 (dd, *J* = 7.8, 1.7 Hz, 2H), 4.45 (s, 2H), 3.98 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.4, 143.5, 138.5, 135.2, 133.7, 130.2, 129.4, 129.0, 128.5, 128.4, 127.7, 127.4, 127.0, 44.2, 36.3, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{21}NO_2S_2$: 395.1014, found: 395.1010.
CCDC number: 2004076

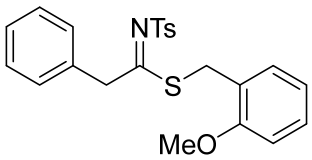
3-Methoxybenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3c)



Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (3-methoxyphenyl) methanethiol (226.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to

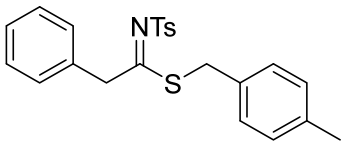
afford **3c** (137.0 mg, 66% yield) as a yellow solid. m.p. 71-72 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.1 Hz, 2H), 7.35-7.29 (m, 7H), 7.09 (t, *J* = 8.1 Hz, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 6.67 (d, *J* = 6.8 Hz, 1H), 6.65 (s, 1H), 4.45 (s, 2H), 3.97 (s, 2H), 3.71 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.4, 159.5, 143.4, 138.4, 136.6, 133.6, 130.2, 129.4, 129.3, 128.5, 127.7, 126.9, 121.2, 114.3, 113.1, 55.0, 44.1, 36.2, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{23}NO_3S_2$: 425.1119, found: 425.1114.

2-Methoxybenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3d)



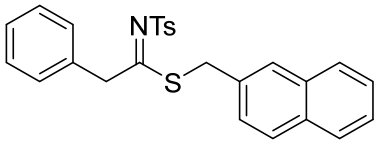
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (2-methoxyphenyl)methanethiol (226.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3d** (162.0 mg, 78% yield) as a yellow solid. m.p. 90-92 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.3 Hz, 2H), 7.35-7.27 (m, 7H), 7.19 (td, *J* = 8.2, 1.7 Hz, 1H), 6.93 (dd, *J* = 7.4, 1.6 Hz, 1H), 6.78 (d, *J* = 8.2 Hz, 1H), 6.69 (td, 7.4, 0.9 Hz, 1H), 4.43 (s, 2H), 4.02 (s, 2H), 3.74 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.0, 157.2, 143.3, 138.6, 133.8, 130.7, 130.0, 129.3, 128.9, 128.4, 127.5, 126.9, 123.3, 120.1, 110.2, 55.1, 44.1, 31.2, 21.4. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{23}NO_3S_2$: 425.1119, found: 425.1111.

4-Methylbenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3e)



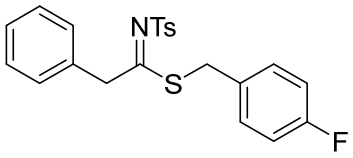
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and *p*-tolylmethanethiol (203.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3e** (163.0 mg, 81% yield) as a yellow solid. m.p. 72-73 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.35-7.28 (m, 7H), 6.98 (d, *J* = 8.2 Hz, 2H), 6.95 (d, *J* = 8.2 Hz, 2H), 4.44 (s, 2H), 3.95 (s, 2H), 2.46 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.5, 143.4, 138.5, 137.2, 133.7, 132.0, 130.2, 129.3, 129.1, 128.9, 128.5, 127.6, 126.9, 44.1, 36.0, 21.5, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{23}NO_2S_2$: 409.1170, found: 409.1180.

Naphthalen-2-ylmethyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3f)



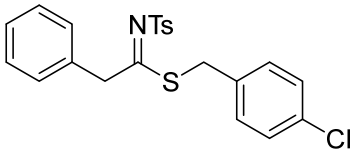
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and naphthalen-2-ylmethanethiol (256.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 4:1) to afford **3f** (168.0 mg, 77% yield) as a white solid. m.p. 104-105 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.77 (dd, *J* = 6.0, 3.3 Hz, 1H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.58 (dd, *J* = 6.1, 3.5 Hz, 1H), 7.49 (d, *J* = 1.0 Hz, 1H), 7.46 (t, *J* = 3.5 Hz, 1H), 7.43 (t, *J* = 3.5 Hz, 1H), 7.34-7.27 (m, 7H), 7.19 (dd, *J* = 8.4, 1.8 Hz, 1H), 4.46 (s, 2H), 4.15 (s, 2H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.3, 143.5, 138.5, 133.7, 133.0, 132.7, 132.5, 130.2, 129.4, 128.6, 128.3, 127.9, 127.7, 127.6, 127.5, 127.0, 126.7, 126.1, 126.0, 44.2, 36.5, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{26}H_{23}NO_2S_2$: 445.1170, found: 445.1165.

4-Fluorobenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3g)



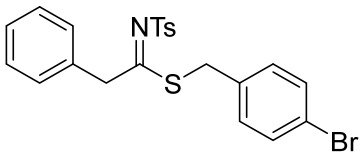
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (4-fluorophenyl)methanethiol (209.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 10:1) to afford **3g** (179.0 mg, 88% yield) as a white solid. m.p. 82-83 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.36-7.29 (m, 7H), 7.01 (d, *J* = 5.3 Hz, 1H), 6.99 (d, *J* = 5.3 Hz, 1H), 6.83 (t, *J* = 8.7 Hz, 2H), 4.44 (s, 2H), 3.93 (s, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.2, 161.9 (d, $J_{C\text{-}F}$ = 246.5 Hz), 143.6, 138.3, 133.6, 131.0 (d, $J_{C\text{-}F}$ = 2.9 Hz), 130.7 (d, $J_{C\text{-}F}$ = 8.1 Hz), 130.2, 129.4, 128.6, 127.8, 127.0, 115.4 (d, $J_{C\text{-}F}$ = 21.4 Hz), 44.1, 35.4, 21.5; ^{19}F NMR (376MHz, $CDCl_3$): δ -115.9 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{20}FNO_2S_2$: 413.0919, found: 413.0921.

4-Chlorobenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3h)



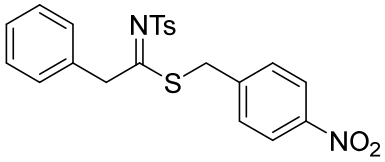
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (4-chlorophenyl)methanethiol (233.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 10:1) to afford **3h** (157.0 mg, 75% yield) as a white solid. m.p. 115-116 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, *J* = 8.3 Hz, 2H), 7.35-7.28 (m, 7H), 7.10 (d, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 4.43 (s, 2H), 3.92 (s, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.0, 143.6, 138.3, 134.0, 133.6, 133.2, 130.2, 130.1, 129.4, 128.6, 128.5, 127.8, 127.0, 44.1, 35.4, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{20}ClNO_2S_2$: 429.0624, found: 429.0626.

4-Bromobenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3i)



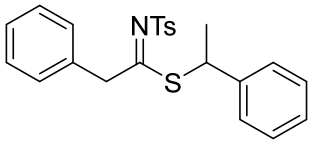
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (4-bromophenyl)methanethiol (298.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3i** (173.0 mg, 75% yield) as a yellow solid. m.p. 126-127 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.35-7.28 (m, 7H), 7.25 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.3 Hz, 2H), 4.43 (s, 2H), 3.89 (s, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.8, 143.6, 138.2, 134.5, 133.5, 131.3, 130.5, 130.1, 129.4, 128.5, 127.7, 126.9, 121.2, 44.0, 35.3, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{20}BrNO_2S_2$: 473.0119, found: 473.0114.

4-Nitrobenzyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3j)



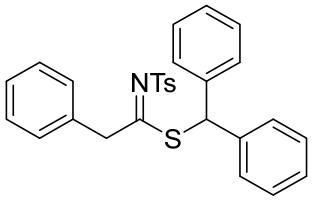
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and (4-nitrophenyl)methanethiolmethanethiol (249.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/DCM, 1:1) to afford **3j** (143.0 mg, 66% yield) as a white solid. m.p. 138-140 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.7 Hz, 2H), 7.75 (d, *J* = 8.3 Hz, 2H)), 7.36-7.28 (m, 7H), 7.14 (d, *J* = 8.7 Hz, 2H), 4.44 (s, 2H), 3.99 (s, 2H), 2.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.1, 146.8, 144.0, 143.4, 138.0, 133.3, 130.2, 129.6, 129.5, 128.6, 127.9, 127.0, 123.4, 44.0, 35.1, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{20}N_2O_4S_2$: 440.0864, found: 440.0869.

1-Phenylethyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3k)



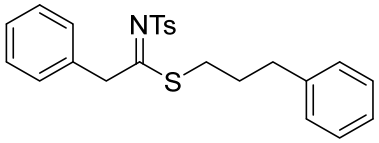
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and 1-phenylethane-1-thiol (203.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3k** (152.0 mg, 76% yield) as a white solid. m.p. 99-101 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.34-7.26 (m, 7H), 7.14-7.21 (m, 3H), 7.07 (dd, *J* = 7.5, 1.8 Hz, 2H), 4.56 (q, *J* = 7.2 Hz, 1H), 4.46 (d, *J* = 16.3 Hz, 1H), 4.32 (d, *J* = 16.3 Hz, 1H), 2.47 (s, 3H), 1.49 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.0, 143.4, 141.2, 138.4, 133.7, 130.1, 129.3, 128.5, 128.3, 127.5, 127.3, 126.9, 45.2, 44.1, 21.5, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{23}NO_2S_2$: 409.1170, found: 409.1176.

Benzhydryl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3l)



Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and diphenylmethanethiol (292.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **3l** (176.0 mg, 76% yield) as a white solid. m.p. 130-132 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.3 Hz, 2H), 7.36-7.30 (m, 5H), 7.24-7.18 (m, 8H), 7.16-7.12 (m, 4H), 5.75 (s, 1H), 4.45 (s, 2H), 2.45 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.1, 143.1, 139.0, 138.2, 133.6, 130.1, 129.1, 128.6, 128.4, 128.3, 127.7, 127.3, 126.8, 54.5, 44.1, 21.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{28}H_{25}NO_2S_2$: 471.1327, found: 471.1323.

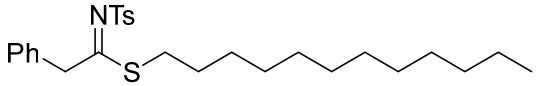
3-Phenylpropyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3m)



Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and 3-phenylpropane-1-thiol (224.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 10:1) to afford **3m** (157.0 mg, 76% yield) as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* =

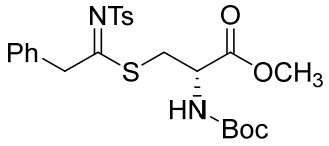
8.2 Hz, 2H), 7.36-7.28 (m, 7H), 7.25-7.14 (m, 3H), 7.0 (d, *J* = 7.2 Hz, 2H), 4.44 (s, 2H), 2.78 (t, *J* = 7.4 Hz, 2H), 2.51 (t, *J* = 7.6 Hz, 2H), 2.44 (s, 3H), 1.79 (p, *J* = 6.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.8, 143.3, 140.5, 138.5, 133.9, 130.0, 129.3, 128.4, 128.2, 128.1, 127.5, 126.7, 125.9, 44.2, 34.7, 31.0, 29.3, 21.4. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{24}H_{25}NO_2S_2$: 423.1327, found: 423.1337.

Dodecyl (*E*)-2-phenyl-*N*-tosylethanimidothioate (3n)



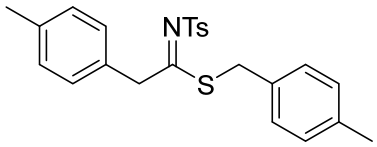
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and 12-phenyldodecane-1-thiol (297.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 15:1) to afford **3m** (103.0 mg, 51% yield) as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.36-7.28 (m, 7H), 4.43 (s, 2H), 2.75 (t, *J* = 7.4 Hz, 2H), 2.44 (s, 3H), 1.45 (p, *J* = 7.1 Hz, 2H), 1.30-1.10 (m, 18H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.1, 143.2, 138.8, 134.1, 130.1, 129.3, 128.5, 127.5, 126.8, 44.4, 31.8, 31.7, 29.5, 29.4, 29.3, 29.2, 29.0, 28.8, 27.8, 22.6, 21.5, 14.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{27}H_{39}NO_2S_2$: 473.2422, found: 473.2425.

Methyl (*E*)-*N*-(tert-butoxycarbonyl)-*S*-(2-phenyl-1-(tosylimino)ethyl)-*D*-cysteinate (3o)



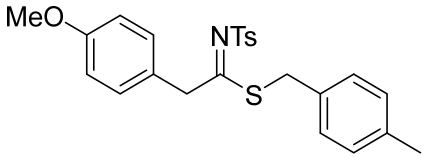
Following General Procedure I, using phenylacetylene (50.0 mg, 0.489 mmol), tosyl azide (116.0 mg, 0.587 mmol), and methyl (*tert*-butoxycarbonyl)-D-cysteinate (346.0 mg, 1.469 mmol). The product was purified by column chromatography (hexane/EtOAc, 2:1) to afford **3o** (186.0 mg, 75% yield) as a white solid. m.p. 81-83 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.36-7.30 (m, 7H), 5.08 (d, *J* = 7.5 Hz, 1H), 4.46-4.37 (m, 3H), 3.62 (s, 3H), 3.30 (dd, *J* = 13.5, 4.9 Hz, 1H), 3.16 (dd, *J* = 13.5, 7.5 Hz, 1H), 2.45 (s, 3H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.9, 170.5, 154.8, 143.5, 138.0, 133.4, 133.0, 129.3, 128.5, 127.6, 126.9, 80.1, 52.5, 52.0, 44.1, 33.4, 28.0, 21.4. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{24}H_{30}N_2O_6S_2$: 506.1545, found: 506.1548.

4-Methylbenzyl (*E*)-2-(p-tolyl)-*N*-tosylethanimidothioate (4b)



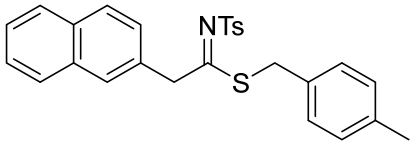
Following General Procedure I, using 1-ethynyl-4-methylbenzene (50.0 mg, 0.430 mmol), tosyl azide (103.0 mg, 0.516 mmol), and *p*-tolylmethanethiol (178.0 mg, 1.29 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4b** (115.0 mg, 63% yield) as a yellow solid. m.p. 75-76 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 6.95 (d, *J* = 8.2 Hz, 2H), 4.40 (s, 2H), 3.94 (s, 2H), 2.46 (s, 3H), 2.33 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.1, 143.3, 138.6, 137.4, 137.1, 132.0, 130.5, 130.1, 129.3, 129.2, 129.1, 128.9, 126.9, 43.8, 36.0, 21.5, 21.1, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{24}H_{25}NO_2S_2$: 423.1327, found: 423.1320.

4-Methylbenzyl (*E*)-2-(4-methoxyphenyl)-*N*-tosylethanimidothioate (4c)



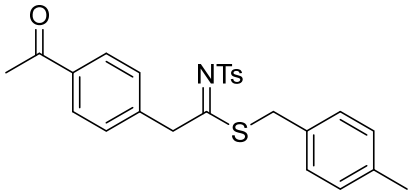
Following General Procedure I, using 1-ethynyl-4-methoxybenzene (50.0 mg, 0.378 mmol), tosyl azide (90.0 mg, 0.454 mmol), and *p*-tolylmethanethiol (157.0 mg, 1.136 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4c** (103.0 mg, 62% yield) as a yellow solid. m.p. 99-100 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 6.99 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 8.3 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.37 (s, 2H), 3.94 (s, 2H), 3.79 (s, 3H), 2.46 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.4, 159.1, 143.3, 138.5, 137.1, 132.0, 131.3, 129.3, 129.0, 128.8, 126.9, 125.5, 113.8, 55.0, 43.3, 36.0, 21.4, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{24}H_{25}NO_3S_2$: 439.1276, found: 439.1266.

4-Methylbenzyl (*E*)-2-(naphthalen-2-yl)-*N*-tosylethanimidothioate (4d)



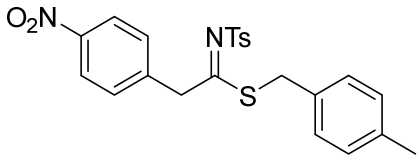
Following General Procedure I, using 2-ethynylnaphthalene (50.0 mg, 0.329 mmol), tosyl azide (79.0 mg, 0.394 mmol), and *p*-tolylmethanethiol (172.0 mg, 0.986 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4d** (100.0 mg, 66% yield) as a white solid. m.p. 143-144 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.83-7.75 (m, 3H), 7.73 (s, 1H), 7.50-7.44 (m, 2H), 7.41 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.01-6.92 (m, 4H), 4.60 (s, 2H), 3.97 (s, 2H), 2.45 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.5, 143.5, 138.5, 137.2, 133.2, 132.6, 132.0, 131.1, 129.4, 129.3, 129.1, 128.9, 128.2, 127.9, 127.8, 127.6, 127.0, 126.2, 126.1, 44.2, 36.1, 21.5, 21.1. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{27}H_{25}NO_2S_2$: 459.1327, found: 459.1331.

4-Methylbenzyl (*E*)-2-(4-acetylphenyl)-*N*-tosylethanimidothioate (4e)



Following General Procedure I, using 1-(4-ethynylphenyl)ethan-1-one (50.0 mg, 0.347 mmol), tosyl azide (82.0 mg, 0.416 mmol), and *p*-tolylmethanethiol (173.0 mg 1.040 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4e** (122.0 mg, 78% yield) as a yellow solid. m.p. 122-123 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.2Hz, 2H), 7.84 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H) , 7.33 (d, *J* = 8.2 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 8.2 Hz, 2H), 4.49 (s, 2H), 3.97 (s, 2H), 2.59 (s, 3H), 2.46 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 186.5, 143.6, 139.1, 138.2, 137.3, 136.3, 131.7, 130.2, 129.4, 129.1, 128.8, 128.5, 126.9, 43.8, 36.0, 26.5, 21.5, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{27}H_{25}NO_2S_2$: 451.1276, found: 451.1277.

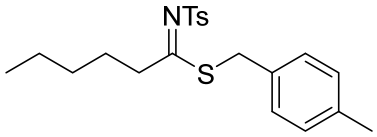
4-Methylbenzyl (*E*)-2-(4-nitrophenyl)-*N*-tosylethanimidothioate (4f)



Following General Procedure I, using 1-ethynyl-4-nitrobenzene (50.0 mg, 0.340 mmol), tosyl azide (80.0 mg, 0.408 mmol), and *p*-tolylmethanethiol (141.0 mg 1.040 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4f** (112.0 mg, 73% yield) as a yellow solid. m.p. 122-123 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 7.9 Hz, 2H), 7.83 (d, *J* = 7.8 Hz, 2H), 7.51 (d, *J* = 8.7 Hz, 2H) , 7.34 (d,

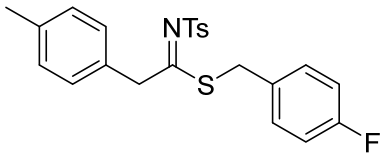
J = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 4.53 (s, 2H), 3.93 (s, 2H), 2.47 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 185.0, 147.3, 143.8, 141.3, 138.0, 137.4, 131.5, 130.8, 129.4, 129.2, 128.8, 127.0, 123.7, 43.5, 36.1, 21.5, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{22}N_2O_4S_2$: 454.1021, found: 454.1025.

4-Methylbenzyl (*E*)-*N*-tosylhexanimidothioate (4g)



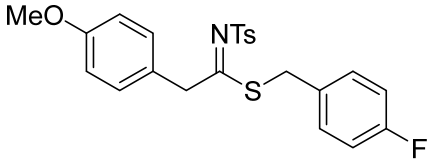
Following General Procedure I, using hex-1-yne (50.0 mg, 0.687 mmol), tosyl azide (146.0 mg, 0.730 mmol), and *p*-tolylmethanethiol (252.0 mg 1.826 mmol). The product was purified by column chromatography (hexane/EtOAc, 10:1) to afford **4g** (79.0 mg, 37% yield) as a light pink oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.01 (s, 4H), 4.00 (s, 2H), 3.05-2.97 (m, 2H), 2.45 (s, 3H), 2.30 (s, 3H), 1.78 (p, *J* = 7.7 Hz, 2H), 1.41-1.30 (m, 4H), 0.89 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.2, 143.2, 138.7, 137.2, 132.3, 129.3, 129.2, 128.8, 126.9, 38.8, 35.5, 31.4, 28.1, 22.1, 21.5, 21.0, 13.8. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{21}H_{27}NO_2S_2$: 389.1483, found: 389.1477.

4-Fluorobenzyl (*E*)-2-(*p*-tolyl)-*N*-tosylethanimidothioate (4h)



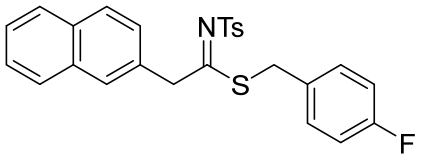
Following General Procedure I, using 1-ethynyl-4-methylbenzene (50.0 mg, 0.430 mmol), tosyl azide (102.0 mg, 0.517 mmol), and (4-fluorophenyl)methanethiol (184.0 mg, 1.291 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4h** (117.0 mg, 64% yield) as a yellow solid. m.p. 130-131 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 7.9 Hz, 2H), 7.02 (d, *J* = 5.6 Hz, 1H), 7.00 (d, *J* = 5.4 Hz, 1H), 6.84 (d, *J* = 8.6 Hz, 1H), 6.81 (d, *J* = 8.6 Hz, 1H), 4.40 (s, 2H), 3.93 (s, 2H), 2.46 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.6, 161.9 (d, J_{C-F} = 246.4 Hz), 143.5, 138.4, 137.5, 131.1 (d, J_{C-F} = 2.8 Hz), 130.6 (d, J_{C-F} = 8.1 Hz), 130.4, 130.0, 129.3, 129.2, 126.9, 115.2 (d, J_{C-F} = 21.4 Hz), 43.7, 35.3, 21.4, 21.0; ^{19}F NMR (376MHz, $CDCl_3$): δ -116.0 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{22}FNO_2S_2$: 427.1076, found: 427.1078.

4-Fluorobenzyl (*E*)-2-(4-methoxyphenyl)-*N*-tosylethanimidothioate (4i)



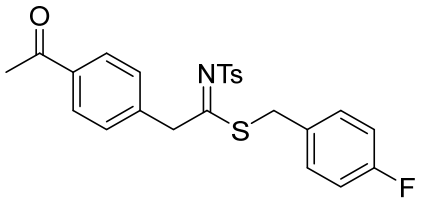
Following General Procedure I, using 1-ethynyl-4-methoxybenzene (50.0 mg, 0.378 mmol), tosyl azide (90.0 mg, 0.454 mmol), and (4-fluorophenyl)methanethiol (161.0 mg, 1.135 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4i** (109.0 mg, 65% yield) as a yellow solid. m.p. 102-103 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 6.6 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.7 Hz, 2H), 7.01 (d, *J* = 5.3 Hz, 1H), 6.99 (d, *J* = 5.3 Hz, 1H), 6.88-6.80 (m, 4H), 4.37 (s, 2H), 3.92 (s, 2H), 3.80 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.1, 161.9 (d, $J_{C\text{-}F}$ = 246.4 Hz), 159.2, 143.6, 138.4, 131.4, 131.1, 130.6 (d, $J_{C\text{-}F}$ = 8.0 Hz), 129.4, 127.0, 125.4, 115.2 (d, $J_{C\text{-}F}$ = 21.4 Hz), 113.9, 55.1, 43.3, 35.4, 21.5; ^{19}F NMR (376MHz, $CDCl_3$): δ -116.0 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{23}H_{22}FNO_3S_2$: 443.1025, found: 443.1020.

4-Fluorobenzyl (*E*)-2-(naphthalen-2-yl)-*N*-tosylethanimidothioate (4j)



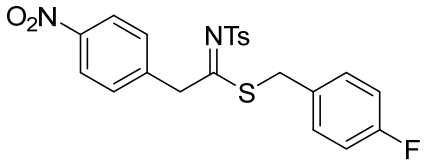
Following General Procedure I, using 2-ethynylnaphthalene (50.0 mg, 0.329 mmol), tosyl azide (78.0 mg, 0.394 mmol), and (4-fluorophenyl)methanethiol (140.0 mg, 0.986 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4j** (106.0 mg, 70% yield) as a white solid. m.p. 121-122 ℃; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86-7.75 (m, 5H), 7.73 (s, 1H), 7.51-7.45 (m, 2H), 7.40 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.32 (d, *J* = 8.3, 2H), 7.01 (d, *J* = 5.4 Hz, 1H), 6.99 (d, *J* = 5.4 Hz, 1H), 6.82 (t, *J* = 8.6, 2H), 4.60 (s, 2H), 3.94 (s, 2H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 188.1, 163.2 (d, $J_{C\text{-}F}$ = 245.0 Hz), 143.6, 138.2, 130.6 (d, $J_{C\text{-}F}$ = 8.1 Hz), 129.4, 128.2, 127.8, 127.7, 127.6, 127.0, 126.2, 126.1, 115.2 (d, $J_{C\text{-}F}$ = 21.4 Hz), 44.2, 35.4, 21.5; ^{19}F NMR (376MHz, $CDCl_3$): δ -115.9 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{26}H_{22}FNO_2S_2$: 463.1076, found: 463.1071.

4-Fluorobenzyl (*E*)-2-(4-acetylphenyl)-*N*-tosylethanimidothioate (4k)



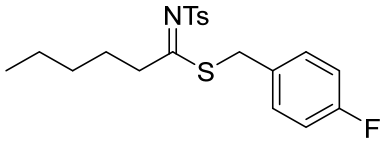
Following General Procedure I, using 1-(4-ethynylphenyl)ethan-1-one (50.0 mg, 0.347 mmol), tosyl azide (82.0 mg, 0.416 mmol), and (4-fluorophenyl)methanethiol (148.0 mg, 1.040 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4k** (120.0 mg, 76% yield) as a yellow solid. m.p. 118-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.82 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.01 (d, *J* = 5.3 Hz, 1H), 6.99 (d, *J* = 5.3 Hz, 1H), 6.83 (t, *J* = 8.6 Hz, 2H), 4.49 (s, 2H), 3.95 (s, 2H), 2.60 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.3, 186.0, 161.8 (d, $J_{C\text{-}F}$ = 245.3 Hz), 143.7, 138.9, 138.0, 136.2, 130.8 (d, $J_{C\text{-}F}$ = 2.8 Hz), 130.5 (d, $J_{C\text{-}F}$ = 8.1 Hz), 130.1, 129.3, 128.4, 126.8, 126.1, 115.2 (d, $J_{C\text{-}F}$ = 21.4 Hz), 43.7, 35.2, 26.4, 21.4; ^{19}F NMR (376MHz, $CDCl_3$): δ -115.7 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{24}H_{22}FNO_3S_2$: 455.1025, found: 455.1024.

4-Fluorobenzyl (*E*)-2-(4-nitrophenyl)-*N*-tosylethanimidothioate (4l)



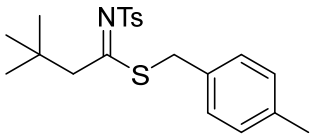
Following General Procedure I, using 1-ethynyl-4-nitrobenzene (50.0 mg, 0.340 mmol), tosyl azide (80.0 mg, 0.408 mmol), and (4-fluorophenyl)methanethiol (145.0 mg, 1.019 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4l** (113.7 mg, 73% yield) as a yellow solid. m.p. 134-135 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 8.7 Hz, 2H), 7.81 (d, *J* = 8.3 Hz, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.02 (d, *J* = 5.4 Hz, 1H), 7.00 (d, *J* = 5.3 Hz, 1H), 6.86 (d, *J* = 8.6 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 1H), 4.53 (s, 2H), 3.98 (s, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 184.6, 162.0 (d, $J_{C\text{-}F}$ = 246.9 Hz), 147.3, 144.0, 141.2, 137.8, 130.8, 130.6, 130.5, 129.5, 127.0, 123.7, 115.3 (d, $J_{C\text{-}F}$ = 21.5 Hz), 43.5, 35.4, 21.5; ^{19}F NMR (376MHz, $CDCl_3$): δ -114.5 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{19}FN_2O_4S_2$: 458.0770, found: 458.0778.

4-Fluorobenzyl (*E*)-*N*-tosylhexanimidothioate (4m)



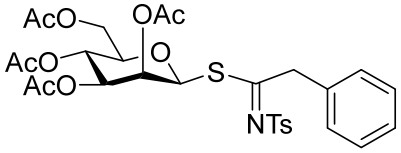
Following General Procedure I, using hex-1-yne (50.0 mg, 0.609 mmol), tosyl azide (144.0 mg, 0.731 mmol), and (4-fluorophenyl)methanethiol (260.0 mg, 1.826 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4m** (74.0 mg, 31% yield) as a pink solid. m.p. 44-45 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 5.3 Hz, 1H), 7.05 (d, *J* = 5.3 Hz, 1H), 6.87 (d, *J* = 8.5 Hz, 1H), 6.83 (d, *J* = 8.5 Hz, 1H), 3.99 (s, 2H), 3.11-2.96 (m, 2H), 2.45 (s, 3H), 1.78 (p, 7.28 Hz, 2H), 1.43-1.30 (m, 4H), 0.90 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.8, 162.0 (d, J_{C-F} = 246.3 Hz), 143.4, 138.6, 131.4 (d, J_{C-F} = 3.0 Hz), 130.5 (d, J_{C-F} = 8.1 Hz), 129.4, 126.9, 115.3 (d, J_{C-F} = 21.5 Hz), 38.8, 34.8, 31.5, 28.1, 22.1, 21.5, 13.8; ^{19}F NMR (376MHz, $CDCl_3$): δ -116.0 (s, C-F). HRMS (EI) m/z: $[M]^+$ calcd. for $C_{20}H_{24}FNO_2S_2$: 393.1232, found: 393.1228.

4-Methylbenzyl (*E*)-3,3-dimethyl-*N*-tosylbutanimidothioate (4n)



Following General Procedure I, using 3,3-dimethylbut-1-yne (50.0 mg, 0.6087 mmol), tosyl azide (144.0 mg, 0.730 mmol), and *p*-tolylmethanethiol (252.0 mg 1.826 mmol). The product was purified by column chromatography (hexane/EtOAc, 5:1) to afford **4n** (51.0 mg, 21% yield) as a white solid. m.p. 80-81 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 6.99 (s, 4H) , 4.04 (s, 2H), 3.09 (s, 2H), 2.44 (s, 3H), 2.30 (s, 3H), 1.13 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.1, 143.2, 138.9, 137.1, 132.4, 129.3, 129.2, 128.8, 127.0, 50.1, 36.0, 32.4, 30.3, 21.5, 21.1. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{21}H_{27}NO_2S_2$: 389.1483, found: 389.1490.

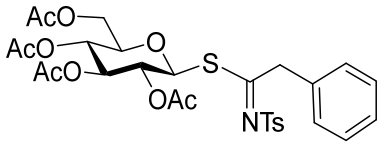
(2R,3R,4S,5S,6S)-2-(acetoxymethyl)-6-(((Z)-2-phenyl-1-(tosylimino)ethyl)thio)tetrahydro-2H-pyran-3,4,5-triyl triacetate (3p)



Following General Procedure II. To a solution of terminal alkyne **1a** (19.0 mg, 0.19 mmol, 1.0 equiv) in DCM (0.3 M) was added *p*-toluenesulfonyl azide (45.0 mg, 0.23 mmol, 1.2 equiv), DMAP (23.0 mg, 19 mmol, 1.0 equiv) and CuI (7.0 mg, 0.038 mmol,

0.2 equiv). The resulting solution was stirred at room temperature under nitrogen atmosphere for 1 hour. Then fresh prepared β-mannosyl 1-thiol **2p** (103.0 mg, 0.285 mmol, 1.5 equiv) were added to the reaction mixture and the reaction was stirred for 0.5 h. The mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography (hexane/EtOAc, 3:2) to afford the desired product **3p** (72 mg, 60% yield) as a colorless oil. $[\alpha]_D^{29}$ -18.3 (c = 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.2 Hz, 2H), 7.30 – 7.22 (m, TsNCH$_2$Ph, 5H), 5.33 (d, *J* = 2.8 Hz, H_2, 1H), 5.27 (s, H_1, 1H), 5.12 (t, *J* = 10.0 Hz, H_4, 1H), 4.92 (dd, *J* = 10.0, 3.4 Hz, H_3, 1H), 4.47 (d, *J* = 16.3 Hz,TsNCH$_2$Ph, 1H), 4.30 (d, *J* = 16.3 Hz, TsNCH$_2$Ph, 1H), 4.11 (dd, *J* = 12.5, 5.4 Hz, H_6, 1H), 3.82 (dd, *J* = 12.5, 1.9 Hz, H_6, 1H), 3.43 (ddd, *J* = 10.0, 5.4, 1.9 Hz, H_5, 1H), 2.43 (s, 3H), 2.04 (s, 3H), 2.03 (s, 3H), 1.97 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$):δ 184.95, 170.63, 169.74, 169.40, 144.11, 137.94, 132.80, 130.17, 129.64, 128.80, 128.04, 126.94, 80.06, 77.01, 71.33, 69.37, 65.09, 61.99, 44.14, 21.63, 20.72, 20.62, 20.52, 20.46; HRMS (ESI) *m/z* calc. for $C_{29}H_{33}NO_{11}NaS_2$ $[M+Na]^+$ 658.1387, found 658.1386.

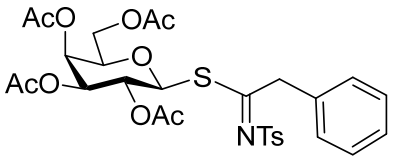
(2R,3R,4S,5R,6S)-2-(acetoxymethyl)-6-(((Z)-2-phenyl-1-(tosylimino)ethyl)thio)tetrahydro-2H-pyran-3,4,5-triyl triacetate (3q)



Following General Procedure II, To a solution of terminal alkyne **1a** (18.0 mg, 0.18 mmol, 1.0 equiv) in DCM (0.3 M) was added *p*-toluenesulfonyl azide (44.0 mg, 0.22 mmol, 1.2 equiv), DMAP (22.0 mg, 0.18 mmol, 1.0 equiv) and CuI (7.0 mg, 0.036 mmol, 0.2 equiv). The resulting solution was stirred at room temperature under nitrogen atmosphere for 1 hour. Then fresh prepared β-glucosyl 1-thiol **2q** (98.0 mg, 0.27 mmol, 1.5 equiv) were added to the reaction mixture and the reaction was stirred for 40 min. The mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography (hexane/EtOAc, 3:2)to afford the desired product **3q** (76.0 mg, 66% yield) as a white solid. $[\alpha]_D^{29}$ +10.5 (c = 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.32 (m, TsNCH$_2$Ph, 5H), 5.15 – 5.08 (m, H_1,H_3, 2H), 5.03 – 4.96 (m, H_2, H_4, 2H), 4.53 (d, *J* = 16.1 Hz, TsNCH$_2$Ph, 1H), 4.33 (d, *J* = 16.1 Hz, TsNCH$_2$Ph, 1H), 4.09 (dd, *J* = 12.5, 4.4 Hz, H_6, 1H), 3.70 (dd, *J* = 12.5, 2.1 Hz, H_6, 1H), 3.38 (ddd, *J* = 10.0,

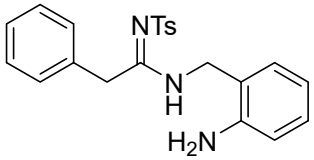
4.4, 2.1 Hz, H_5, 1H), 2.48 (s, 3H), 2.05 (s, 3H), 1.99 (s, 3H), 1.97 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.22, 170.60, 170.09, 169.17, 169.14, 144.18, 138.10, 132.88, 130.10, 129.60, 128.80, 128.09, 126.92, 80.79, 77.31, 73.67, 68.39, 67.54, 61.19, 44.36, 21.64, 20.68, 20.53, 20.38; HRMS (ESI) *m/z* calc. for $C_{29}H_{33}NO_{11}NaS_2$ $[M+Na]^+$ 658.1387, found 658.1387.

(2R,3S,4S,5R,6S)-2-(acetoxymethyl)-6-(((Z)-2-phenyl-1-(tosylimino)ethyl)thio)tetrahydro-2H-pyran-3,4,5-triyl triacetate (3r)



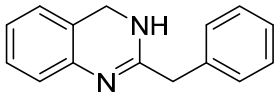
Following General Procedure II, To a solution of terminal alkyne **1a** (43.0 mg, 0.42 mmol, 1.0 equiv) in DCM (0.3 M) was added *p*-toluenesulfonyl azide (100.0 mg, 0.50 mmol, 1.2 equiv), DMAP (51.0 mg, 0.42 mmol, 1.0 equiv) and CuI (16.0 mg, 0.084 mmol, 0.2 equiv). The resulting solution was stirred at room temperature under nitrogen atmosphere for 1 hour. Then fresh prepared β-galactosyl 1-thiol **2r** (228.0 mg, 0.63 mmol, 1.5 equiv) were added to the reaction mixture and the reaction was stirred for 1.5 h. The mixture was extracted with DCM for three times and the combined organic phase was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude products were purified by flash column chromatography (hexane/EtOAc, 3:2)to afford the desired product **3r** (166.0 mg, 63% yield) as a colorless oil. $[\alpha]_D^{29}$ +23.6 (c = 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.26 (m, $TsNCH_2\underline{Ph}$, 5H), 5.28 (d, *J* = 3.2 Hz, H_4, 1H), 5.14 (t, *J* = 10.1 Hz, H_2, 1H), 5.05 (d, *J* = 10.5 Hz, H_1, 1H), 4.88 (dd, *J* = 9.7, 3.2 Hz, H_3, 1H), 4.46 (d, *J* = 16.1 Hz, $TsN\underline{CH_2}Ph$, 1H), 4.31 (d, *J* = 16.1 Hz, $TsN\underline{CH_2}Ph$, 1H), 3.90 (dd, *J* = 11.2, 6.6 Hz, H_6, 1H), 3.78 (dd, *J* = 11.2, 6.6 Hz, H_6, 1H), 3.60 (t, *J* = 6.6 Hz, H_5, 1H), 2.40 (s, 3H), 2.03 (s, 3H), 1.97 (s, 3H), 1.89 (s, 3H), 1.85 (s, 3H). ^{13}C NMR (101 MHz, cdcl$_3$) δ 185.26, 170.13, 170.03, 169.92, 169.31, 144.06, 138.16, 132.96, 130.08, 129.63, 128.78, 128.05, 126.84, 81.21, 74.85, 71.70, 66.86, 65.79, 60.88, 44.39, 21.61, 20.59, 20.55, 20.51, 20.46; HRMS (ESI) *m/z* calc. for $C_{29}H_{33}NO_{11}NaS_2$ $[M+Na]^+$ 658.1387, found: 658.1380.

(*E*)-*N*-(2-aminobenzyl)-2-phenyl-*N'*-tosylacetimidamide (5a)



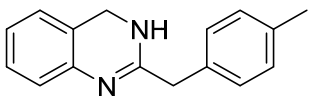
To a solution of **3b** (50.0 mg, 0.126 mmol, 1.0 equiv) in DCM (0.3M) was added 2-aminobenzylamine (0.151 mmol, 1.2 equiv) stirred at room temperature under nitrogen atmosphere. The resulting reaction mixture was stirred for 2 hours until the reaction was complete as indicated by TLC. After the reaction was completed, the mixture was extracted with DCM for three times and combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude product was purified by column chromatography (hexan/EtOAc, 2:1) to afford **5a** (47.0 mg, 95%) as a white solid, m.p. 148-149 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.37-7.27 (m, 5H), 7.17 (dd, *J* = 7.4, 1.7 Hz, 2H), 7.07 (td, *J* = 7.7, 1.5 Hz, 1H), 6.85 (dd, *J* = 7.5, 1.3 Hz, 1H), 6.64-6.58 (m, 2H), 5.53 (br, s, 1H), 4.34 (d, *J* = 6.0 Hz, 2H), 4.30 (s, 2H), 3.99 (br, s, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 145.3, 142.4, 140.2, 132.6, 130.4, 130.0, 129.3, 129.2, 128.0, 126.3, 120.2, 118.0, 116.0, 42.9, 39.4, 21.4. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{22}H_{23}SN_3O_2$: 393.1511, found: 393.1521.

2-Benzyl-3,4-dihydroquinazoline (6a)



Following General Procedure III, using **3b** (50.0 mg, 0.126 mmol, 1.0 equiv), 2-aminobenzylamine (19.0 mg, 0.152 mmol, 1.2 equiv) and PTSA (48.0 mg, 0.253 mmol, 2.0 equiv. The crude product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6a** (25.0 mg, 90%) as a yellow sticky oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 7.2 Hz, 2H), 7.31-7.20 (m, 3H), 7.11 (t, *J* = 7.6 HZ, 1H), 7.01 (t, *J* = 7.8 Hz, 2H), 6.86 (d, *J* = 7.4 Hz, 1H), 6.51 (br, s, 1H), 4.60 (s, 2H), 3.74 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.3, 137.6, 134.8, 129.2, 128.9, 128.3, 127.5, 125.8, 125.1, 119.5, 118.6, 43.8, 40.7. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{15}H_{14}N_2$: 222.1157, found: 222.1151.

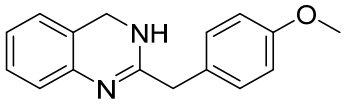
2-(4-Methylbenzyl)-3,4-dihydroquinazoline (6b)



Following General Procedure III, using **4h** (50.0 mg, 0.117 mmol), 2-aminobenzylamine (17.0 mg, 0.140 mmol), and PTSA (45.0 mg, 0.234 mmol). The product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6b**

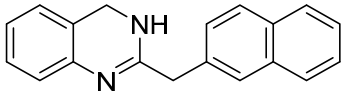
(25.0 mg, 88% yield) as a colorless sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 6.8 Hz, 2H), 7.15-7.07 (m, 3H), 7.04-6.98 (m, 2H), 6.87 (d, *J* = 7.4 Hz, 1H), 5.86 (br, s, 1H), 4.60 (s, 2H), 3.72 (s, 2H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.5, 139.8, 137.1, 132.2, 129.6, 129.1, 128.1, 125.6, 124.4, 120.3, 119.4, 44.5, 41.7, 21.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{16}H_{16}N_2$: 236.1313, found: 236.1314.

2-(4-Methoxybenzyl)-3,4-dihydroquinazoline (6c)



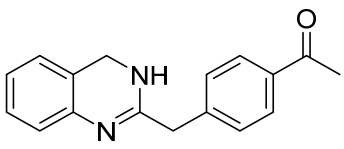
Following General Procedure III, using **4i** (50.0 mg, 0.113 mmol), 2-aminobenzylamine (17.0 mg, 0.135 mmol), and PTSA (43.0 mg, 0.225 mmol). The product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6c** (26.6 mg, 94% yield) as a colorless sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.4 Hz, 2H), 7.10 (t, *J* = 7.5 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 6.85 (d, *J* = 7.4 Hz, 1H), 6.79 (d, *J* = 8.5 Hz, 2H), 6.47 (s, br, 1H), 4.59 (s, 2H), 3.71 (s, 3H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 158.8, 137.4, 130.3, 128.3, 126.5, 125.8, 125.1, 119.3, 118.5, 114.3, 55.2, 43.7, 39.7. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{16}H_{16}ON_2$: 252.1263, found: 252.1257.

2-(Naphthalen-2-ylmethyl)-3,4-dihydroquinazoline (6d)



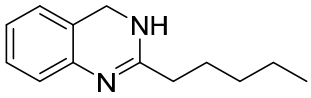
Following General Procedure III, using **4j** (50.0 mg, 0.108 mmol), 2-aminobenzylamine (16.0 mg, 0.129 mmol), and PTSA (41.0 mg, 0.216 mmol). The product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6d** (27.2 mg, 93% yield) as a sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80-7.69 (m, 4H), 7.47-7.39 (m, 3H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.00-6.93 (m, 2H), 6.79 (d, *J* = 7.5 Hz, 1H), 5.66 (br, s, 1H), 4.53 (s, 2H), 3.82 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 136.0, 133.3, 132.5, 131.7, 128.7, 128.4, 128.3, 127.6, 126.8, 126.3, 126.1, 125.8, 125.5, 119.0, 118.0, 43.3, 40.0. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{19}H_{16}N_2$: 272.1313, found: 272.1316.

1-(4-((3,4-Dihydroquinazolin-2-yl)methyl)phenyl)ethan-1-one (6e)



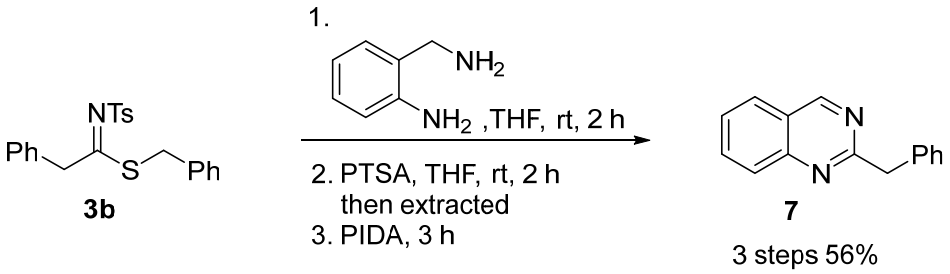
Following General Procedure IV, using **4k** (50.0 mg, 0.110 mmol), 2-aminobenzylamine (16.0 mg, 0.132 mmol), and PTSA (42.0 mg, 0.220 mmol). The product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6e** (27.2 mg, 94% yield) as a colorless sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.12-7.08 (m, 1H), 7.04-7.00 (m, 2H), 6.87 (d, *J* = 7.3 Hz, 1H), 6.18 (br, s, 1H), 4.63 (s, 2H), 3.85 (s, 2H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 157.9, 140.0, 136.5, 136.2, 129.4, 128.8, 128.5, 125.9, 125.6, 119.1, 118.2, 43.7, 40.1, 26.5. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{17}H_{16}ON_2$: 264.1263, found: 264.1270.

2-Pentyl-3,4-dihydroquinazoline (6f)



Following General Procedure III, using **4m** (110.0 mg, 0.280 mmol), 2-aminobenzylamine (41.0 mg, 0.335 mmol), and PTSA (106.0 mg, 0.559 mmol). The product was purified by column chromatography (DCM/MeOH, 10:1) to afford **6f** (29.0 mg, 52% yield) as a colorless sticky oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (d, *J* = 7.6 Hz, 1H), 7.18 (t, *J* = 7.1 Hz, 1H), 7.13 (t, *J* = 7.0 Hz, 1H), 6.96 (d, *J* = 7.2 Hz, 1H), 4.75 (s, 2H), 2.80 (t, *J* = 7.6 Hz, 2H), 1.87 (p, *J* = 7.6 Hz, 2H), 1.44-1.21 (m, 4H), 0.81 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 131.6, 128.9, 126.9, 126.1, 117.6, 116.5, 42.1, 31.8, 30.9, 27.1, 22.2, 13.8. HRMS (EI) m/z: $[M]^+$ calcd. for $C_{13}H_{18}N_2$: 202.1470, found: 202.1461.

One-pot synthesis of 2-benzylquinazoline (7)[3]



To a solution of **3b** (60.0 mg, 0.152 mmol, 1.0 equiv) in THF (0.3M) was added 2-aminobenzylamine (23.0 mg, 0.182 mmol, 1.2 equiv) stirred for 2 hours at room temperature under nitrogen atmosphere. Then the *p*-toluenesulfonic acid monohydrate (52.0 mg, 0.304 mmol, 2.0 equiv) was added to the reaction mixture and stirred for 2 hours. After the reaction was completed (as indicated by TLC), the mixture was extracted with DCM and $NaHCO_3$ for two times and the combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude was utilized directly in next step without purification. To a solution of crude in THF (0.3M) was

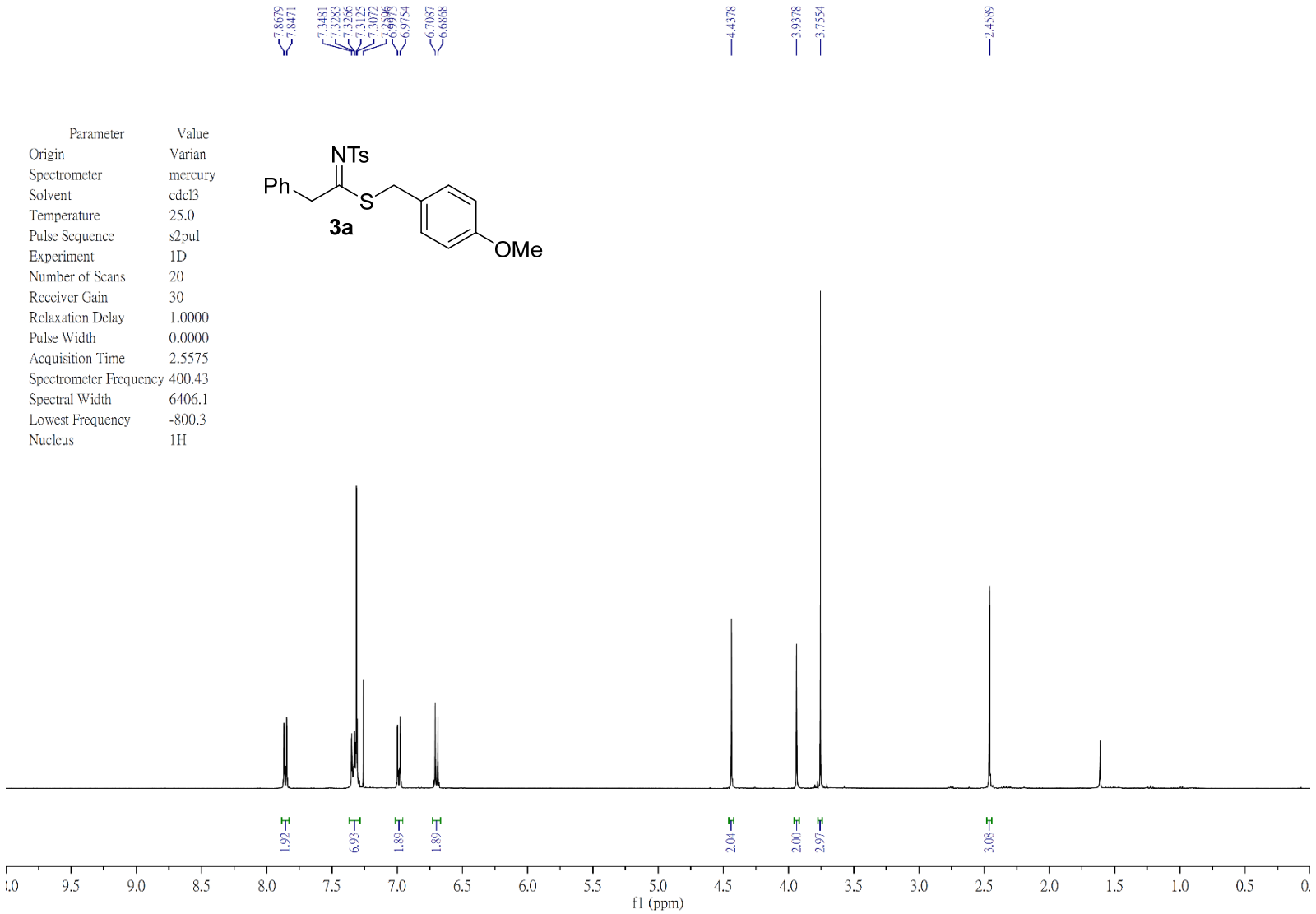
added PIDA (147.0 mg, 0.456 mmol, 3.0 equiv) at room temperature and stirred for 3 hours. After the reaction was completed, the mixture was extracted with ethyl acetate (EA) for three times and combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated. The crude product was purified by column chromatography (hexane/EtOAc, 3:1) to afford **9** (3 steps, 19.0 mg, 56% yield) as a yellow solid. m.p. 65-67 ℃ (reported: 68-69 ºC); ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.31 (s, 1H), 8.00 (d, *J* = 8.5 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.82 (d, *J* = 7.8 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.44 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.3 Hz, 1H), 4.47 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.9, 160.7, 150.3, 138.4, 133.9, 129.1, 128.4, 127.9, 127.1, 126.9, 126.4, 122.9, 46.2.

References:

[1] Elangovan, A.; Yang, S.-W.; Lin, J.-H.; Kao, K.-M.; Ho, T.-I. *Org. Biomol. Chem.,* 2004, **2**, 1597–1602.

[2] Guo, J.-R.; Huang, H.-Y.; Yan, Y.-L.; Liang, C.-F. *Asian J. Org. Chem*., 2018, **7**, 179–188.

[3] Fang, J.; Zhou, J.; Fang, Z. *RSC Adv*., 2013, **3**, 334−336.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6.7087
6.6868
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3.9378
3.7554
2.4589
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Origin Varian
Spectrometer mercury
Solvent cdcl3
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Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
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Spectral Width 6406.1
Lowest Frequency -800.3
Nucleus 1H
NTs
Ph
S
3a
OMe
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1.89
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2.00
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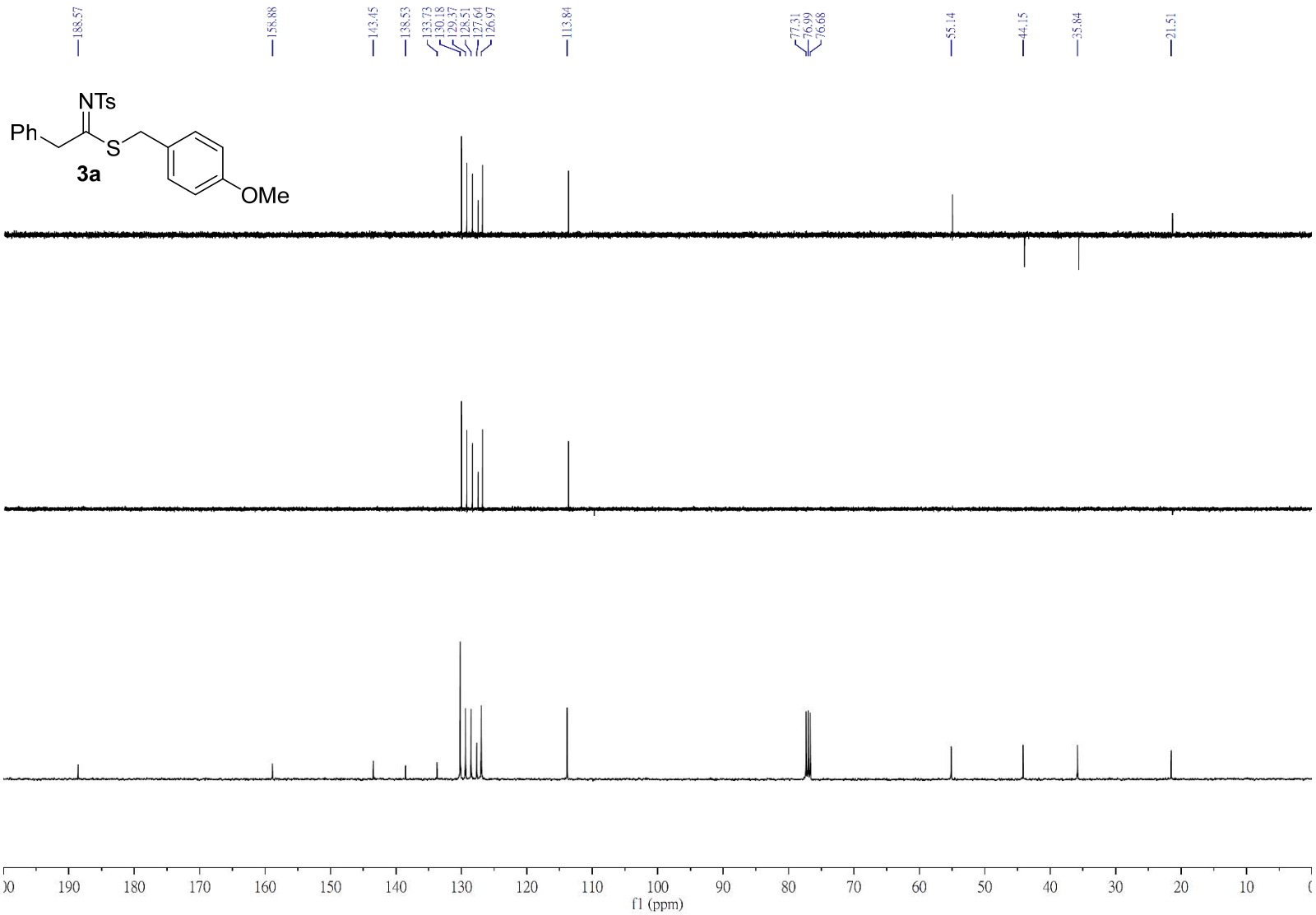
3.08
f1 (ppm)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129.37
128.51
127.64
126.97
113.84
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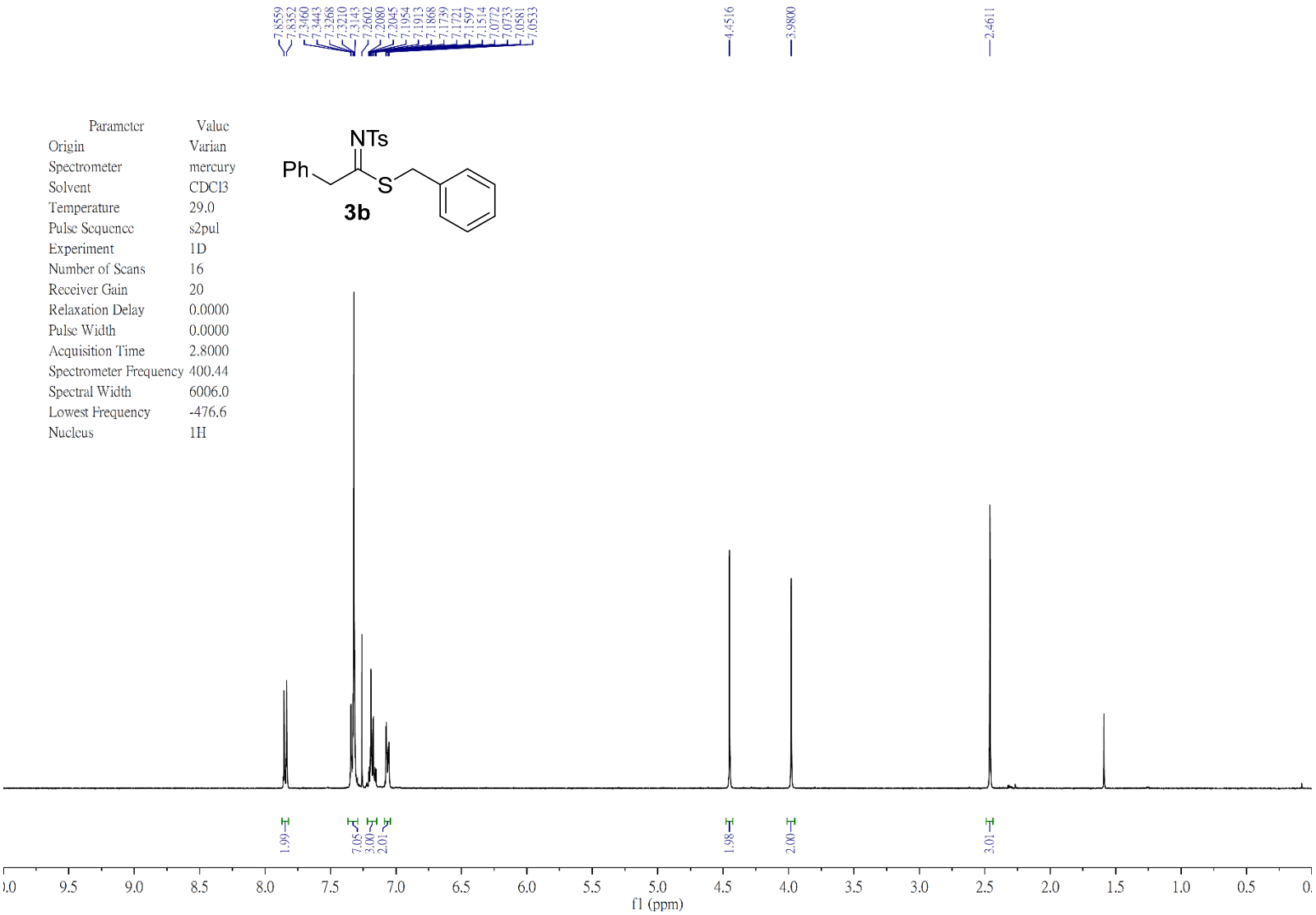
76.68
55.14
44.15
35.84
21.51
NTs
Ph
S
OMe
3a
f1 (ppm)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7.2080
7.2045
7.1954
7.1913
7.1868
7.1739
7.1721
7.1597
7.1514
7.0772
7.0733
7.0581
7.0533
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Spectrometer mercury
Solvent CDCl3
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Receiver Gain 20
Relaxation Delay 0.0000
Pulse Width 0.0000
Acquisition Time 2.8000
Spectrometer Frequency 400.44
Spectral Width 6006.0
Lowest Frequency -476.6
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Ph
S
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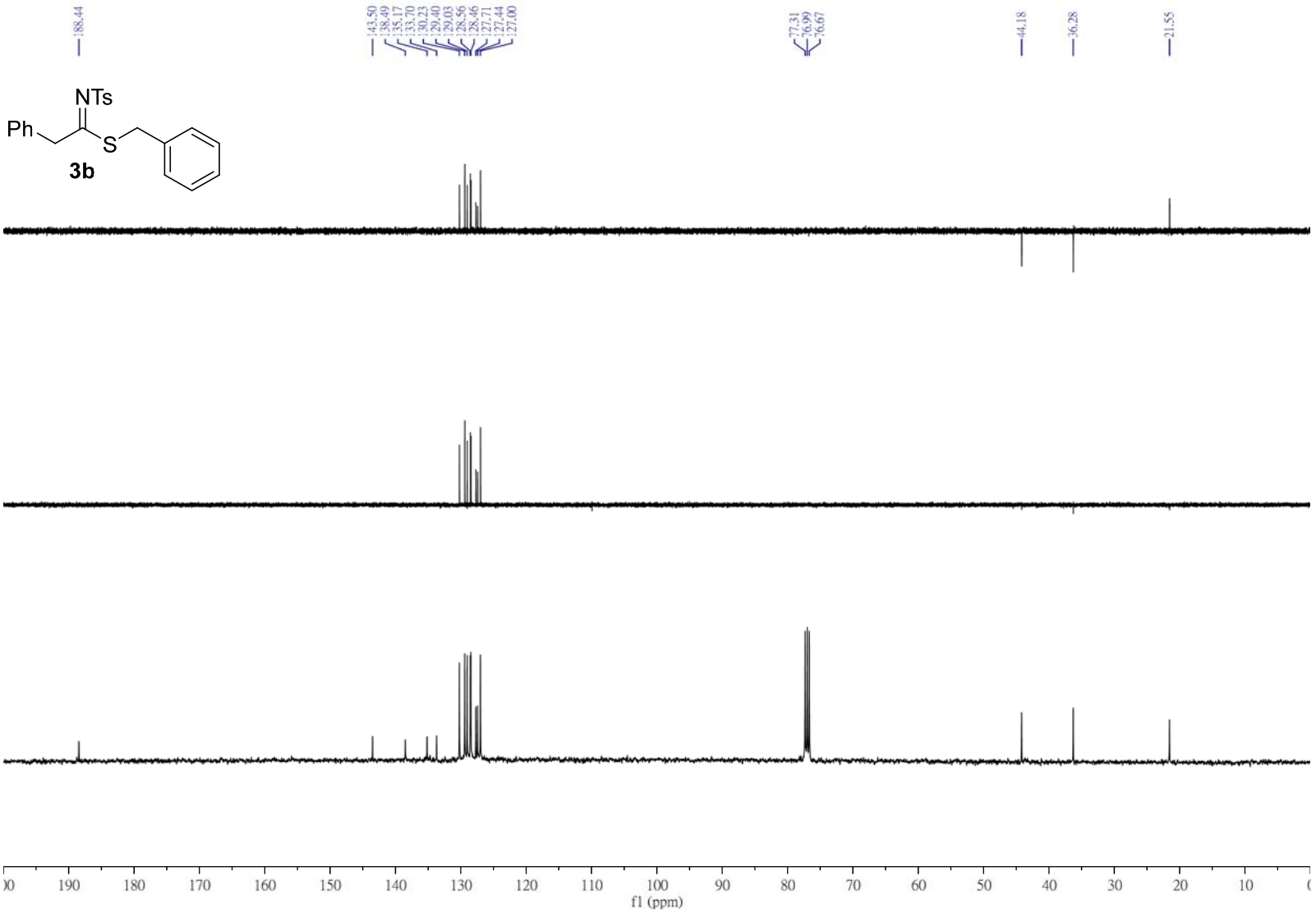
3.01
f1 (ppm)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129.03
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128.46
127.71
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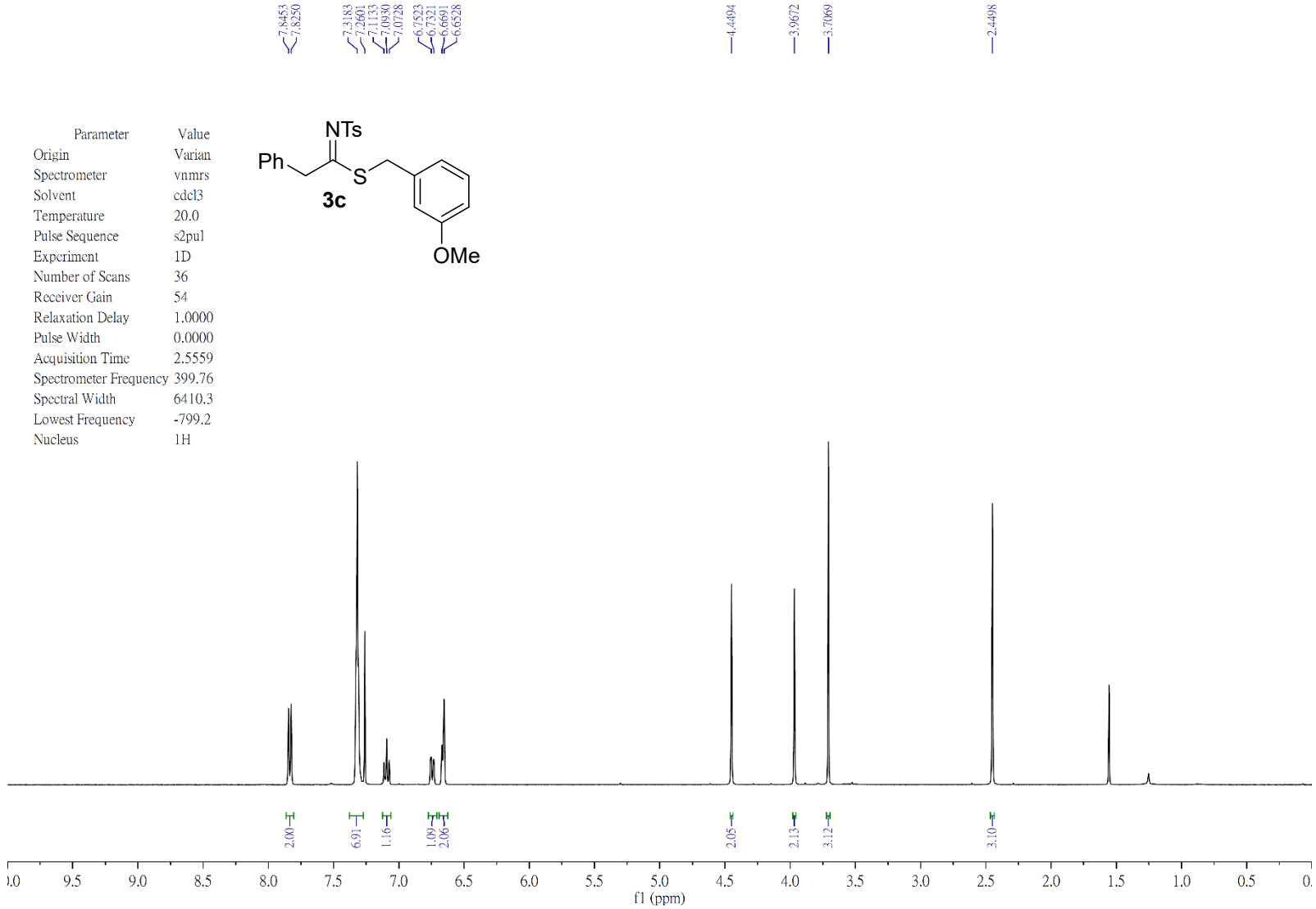
21.55
NTs
Ph
S
3b
f1 (ppm)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Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
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Pulse Width 0.0000
Acquisition Time 2.5559
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Spectral Width 6410.3
Lowest Frequency -799.2
Nucleus 1H
NTs
Ph
S
3c
OMe
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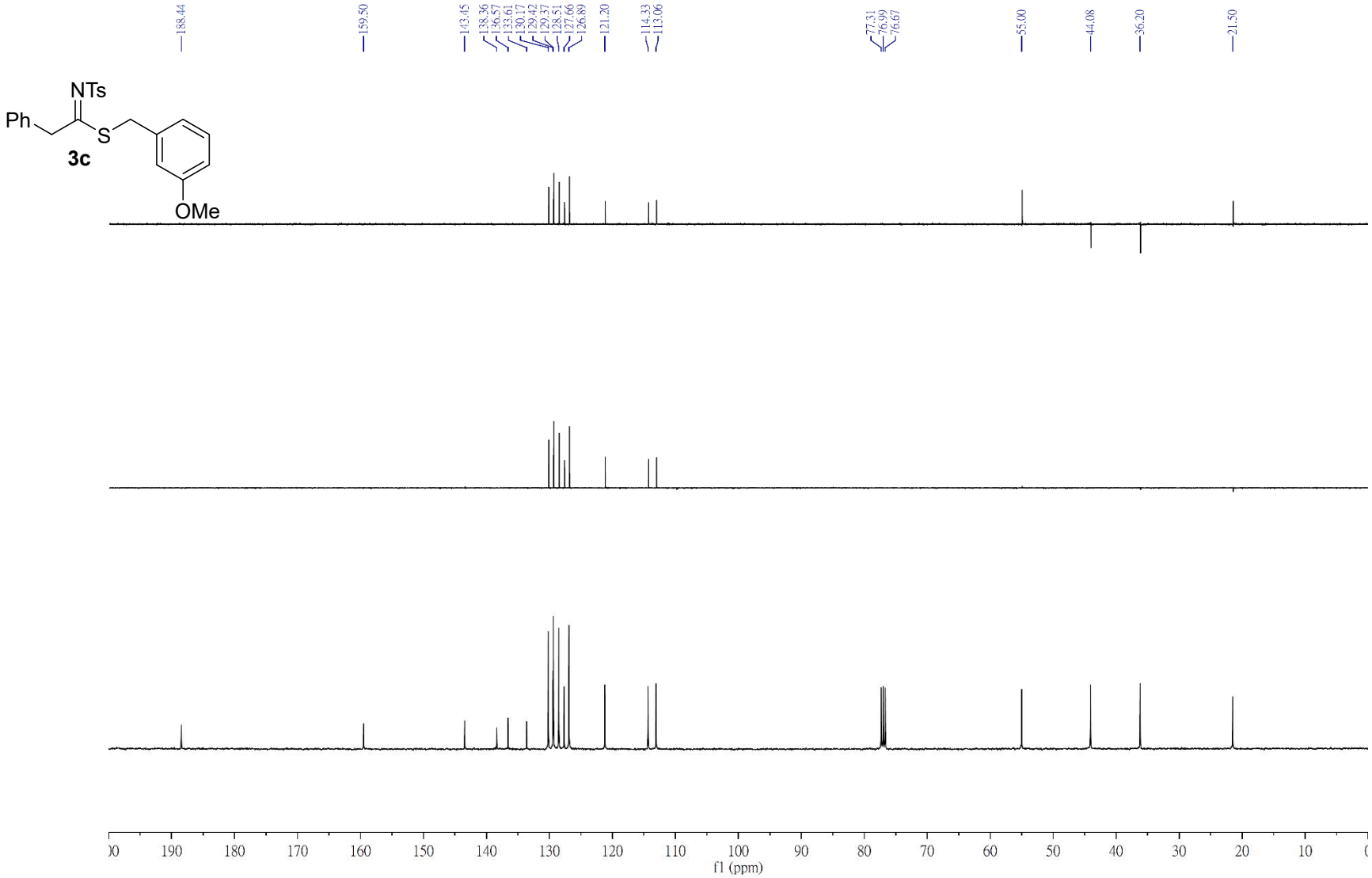
3.10
f1 (ppm)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129.42
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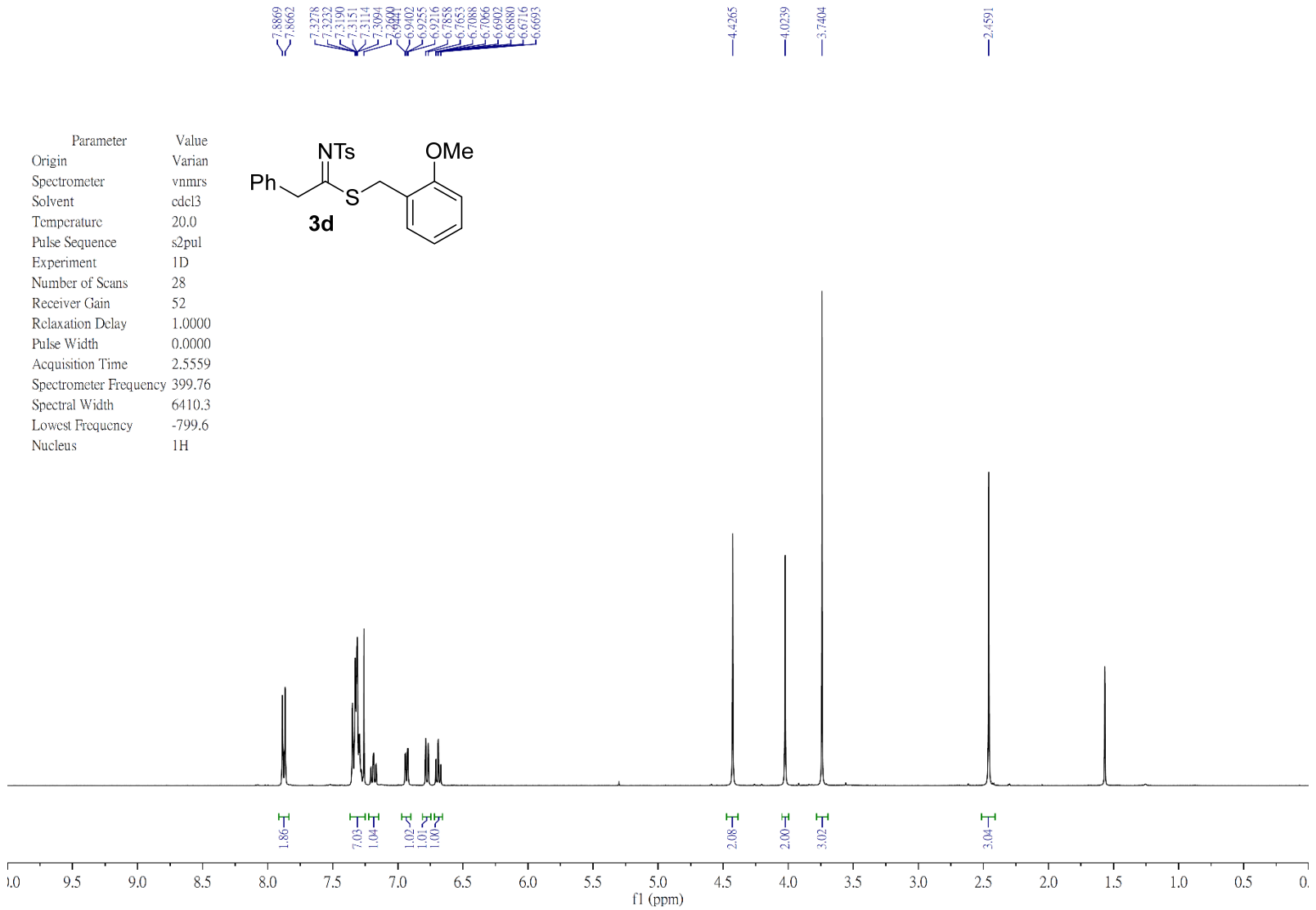
128.51
127.66
126.89
121.20
114.33
113.06
77.31
76.99
76.67
55.00
44.08
36.20
21.50
NTs
Ph
S
3c
OMe
f1 (ppm)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6.7088
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Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
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Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
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Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
NTs
OMe
Ph
S
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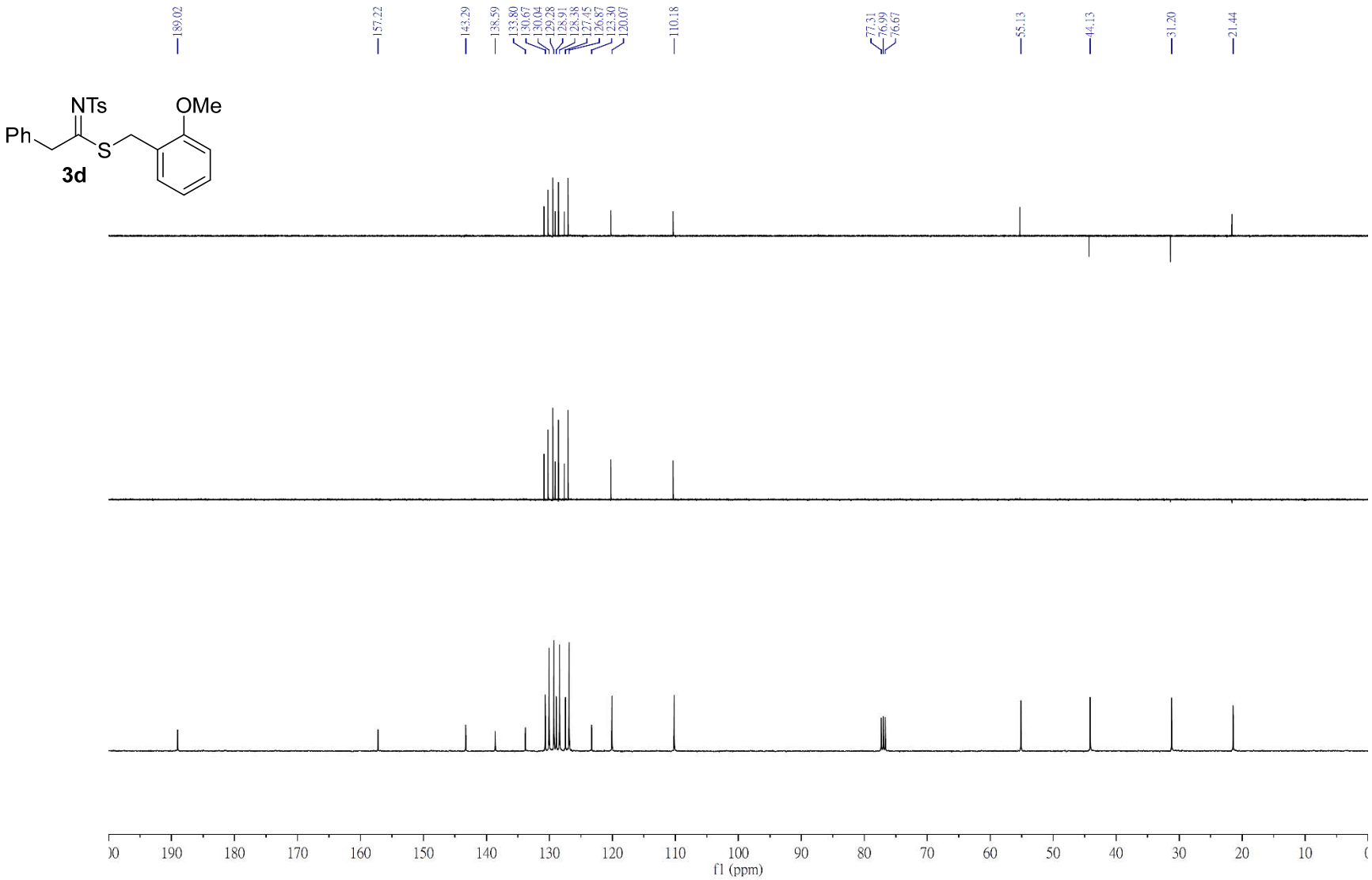
2.00
3.02
3.04
f1 (ppm)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55.13
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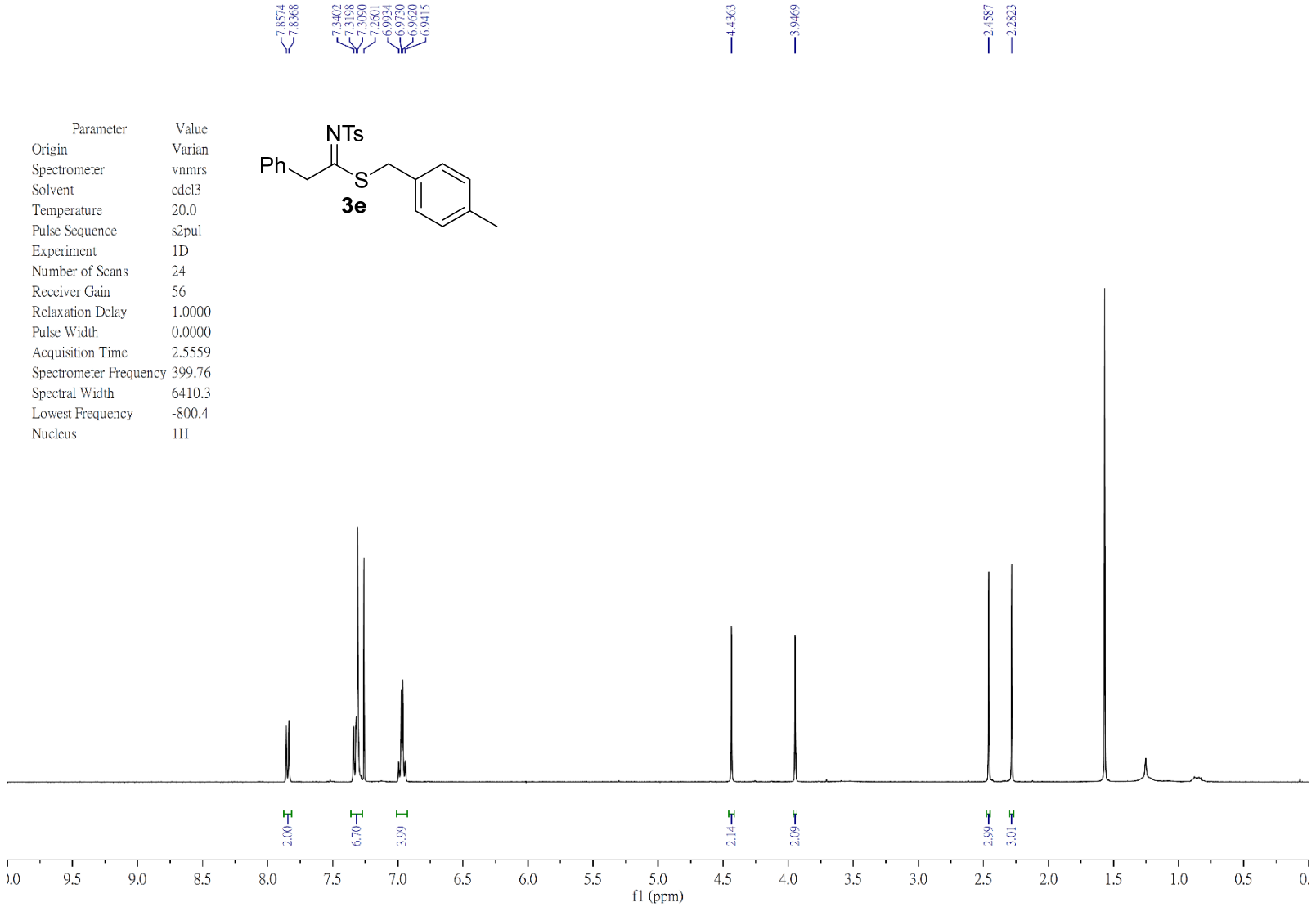
31.20
21.44
NTs
OMe
Ph
S
3d
f1 (ppm)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Parameter Value
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Spectrometer vnmrs
Solvent cdcl3
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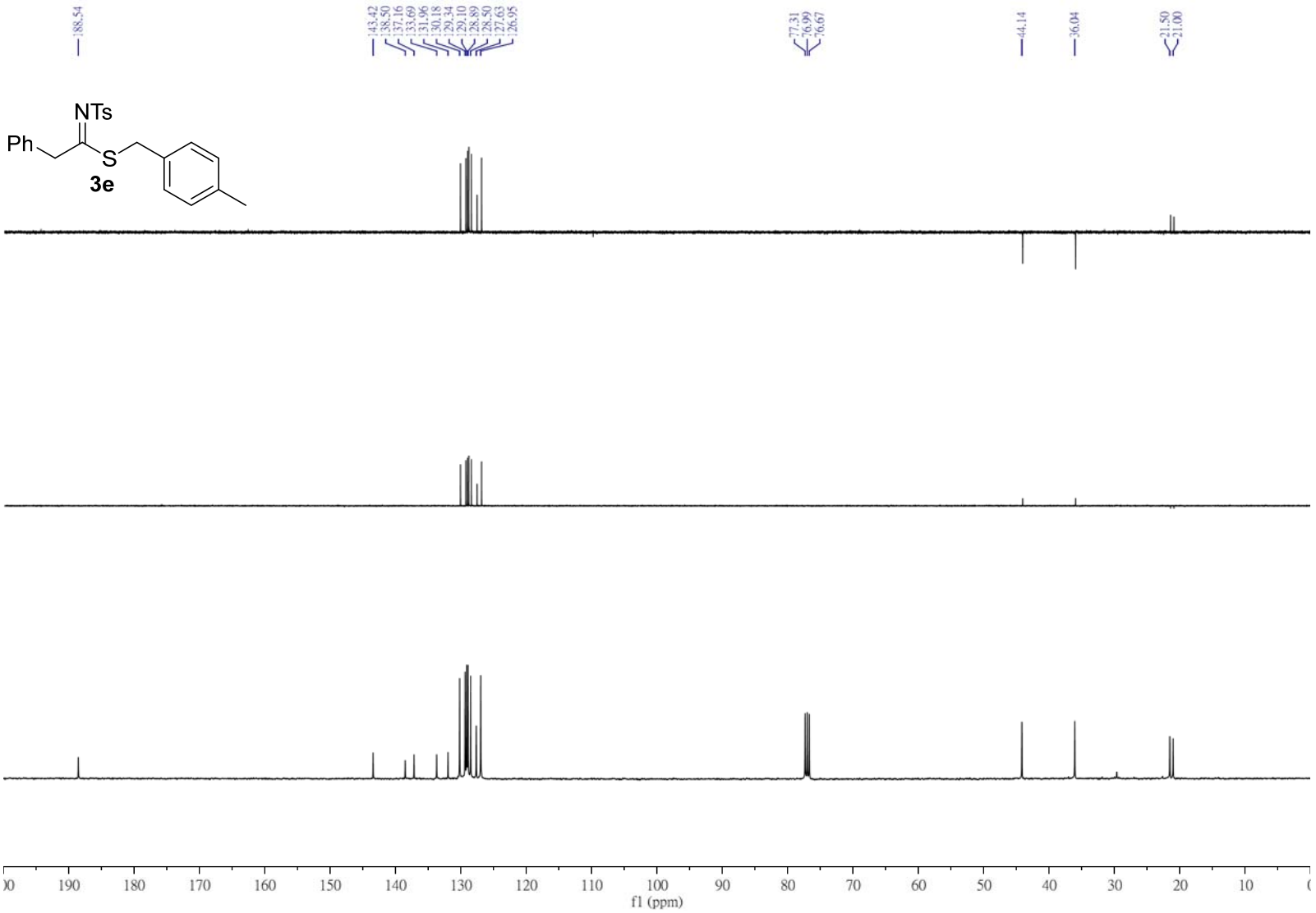
f1 (ppm)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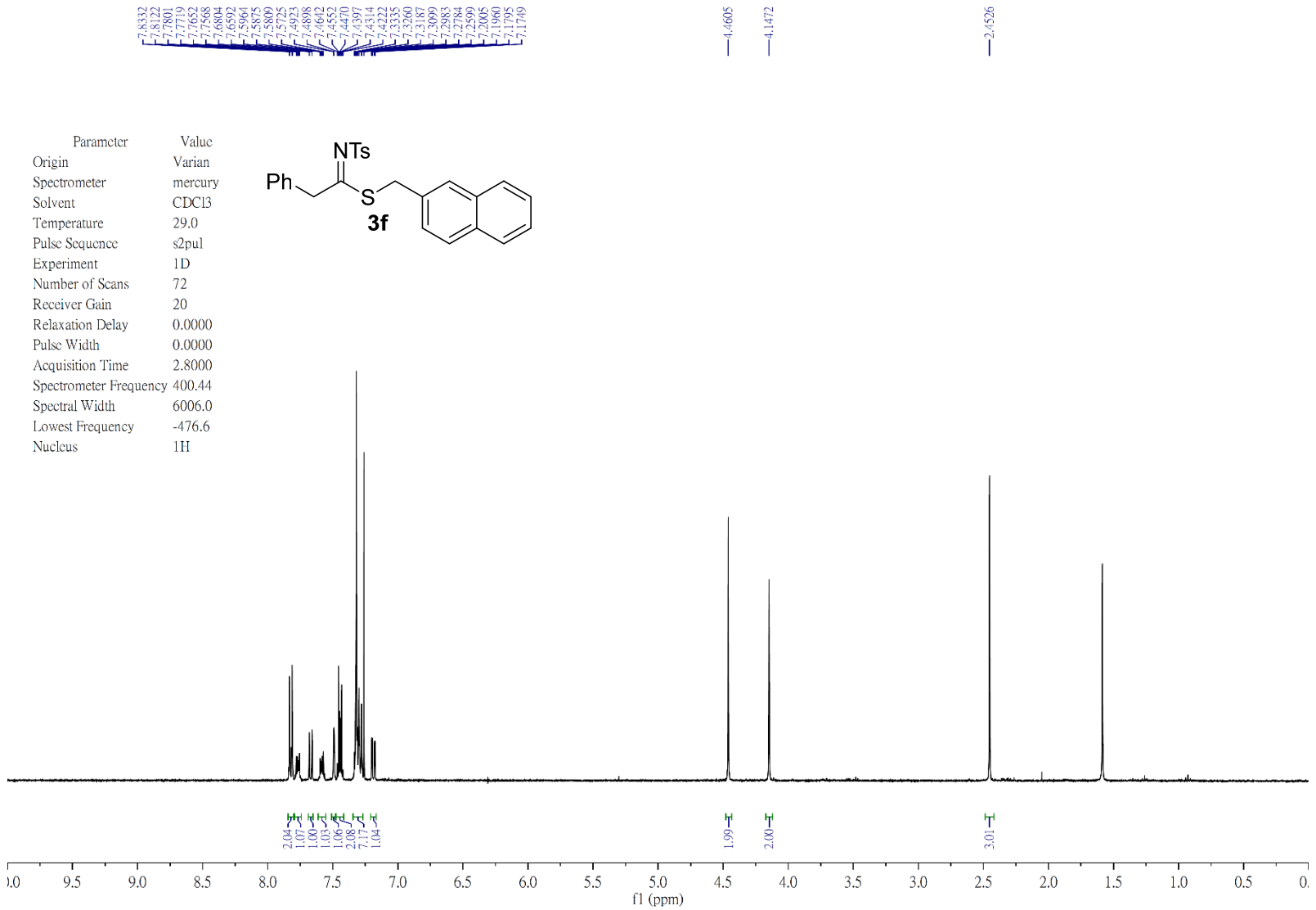
21.00
NTs
Ph
S
3e
f1 (ppm)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Spectrometer mercury
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Spectral Width 6006.0
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NTs
Ph
S
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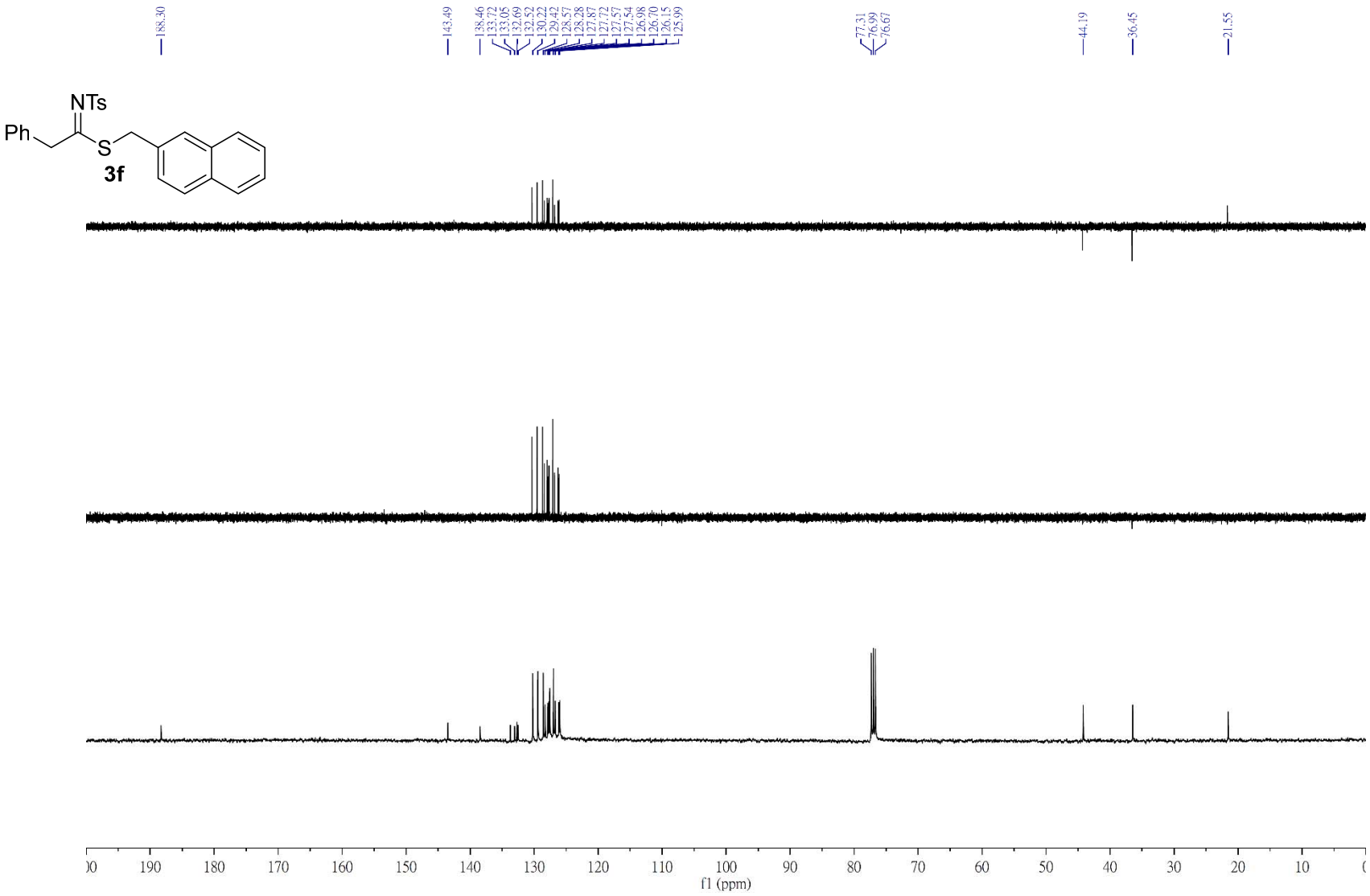
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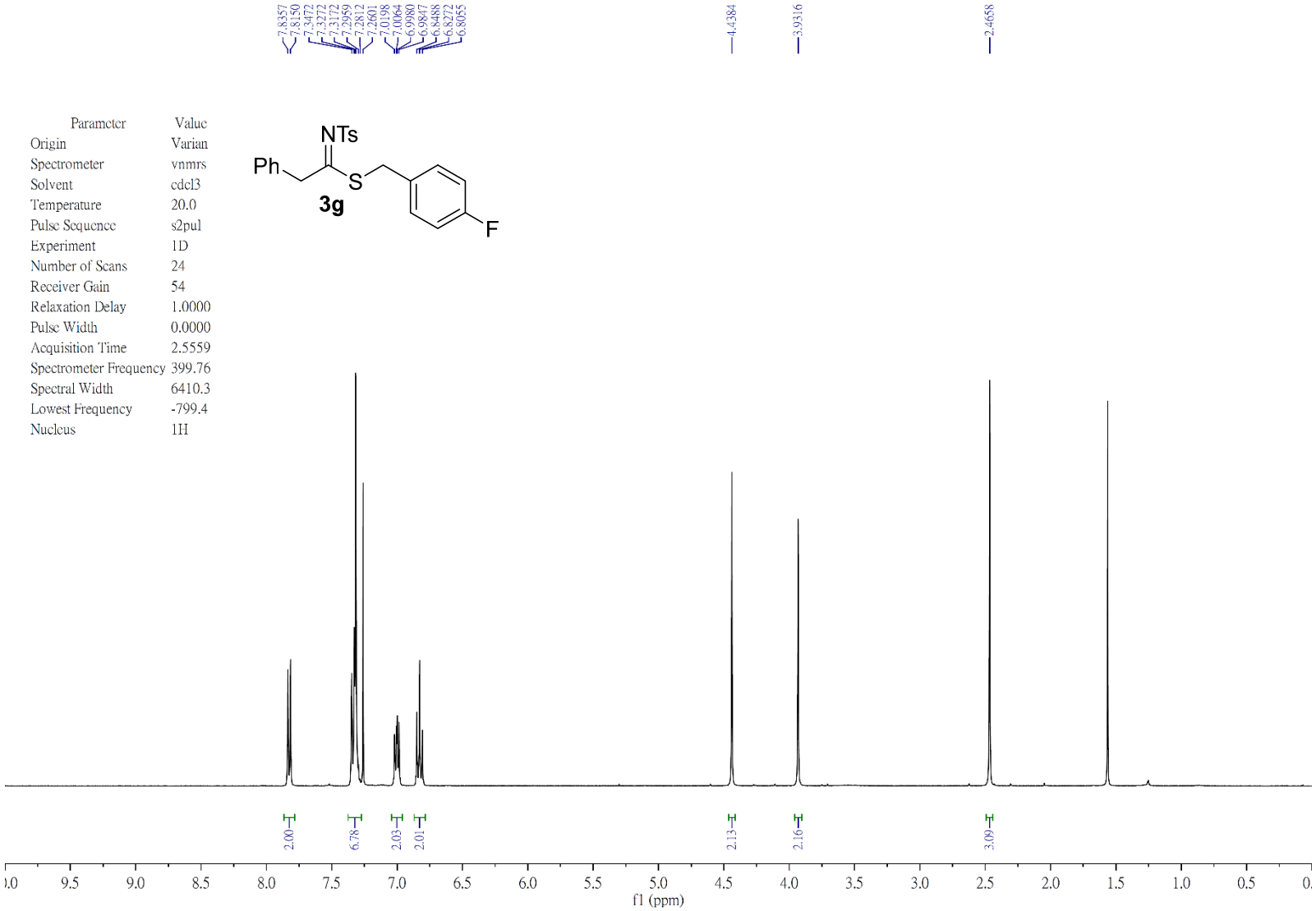
Ph
S
3f
f1 (ppm)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Parameter Value
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Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 24
Receiver Gain 54
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
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Spectral Width 6410.3
Lowest Frequency -799.4
Nucleus 1H
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F
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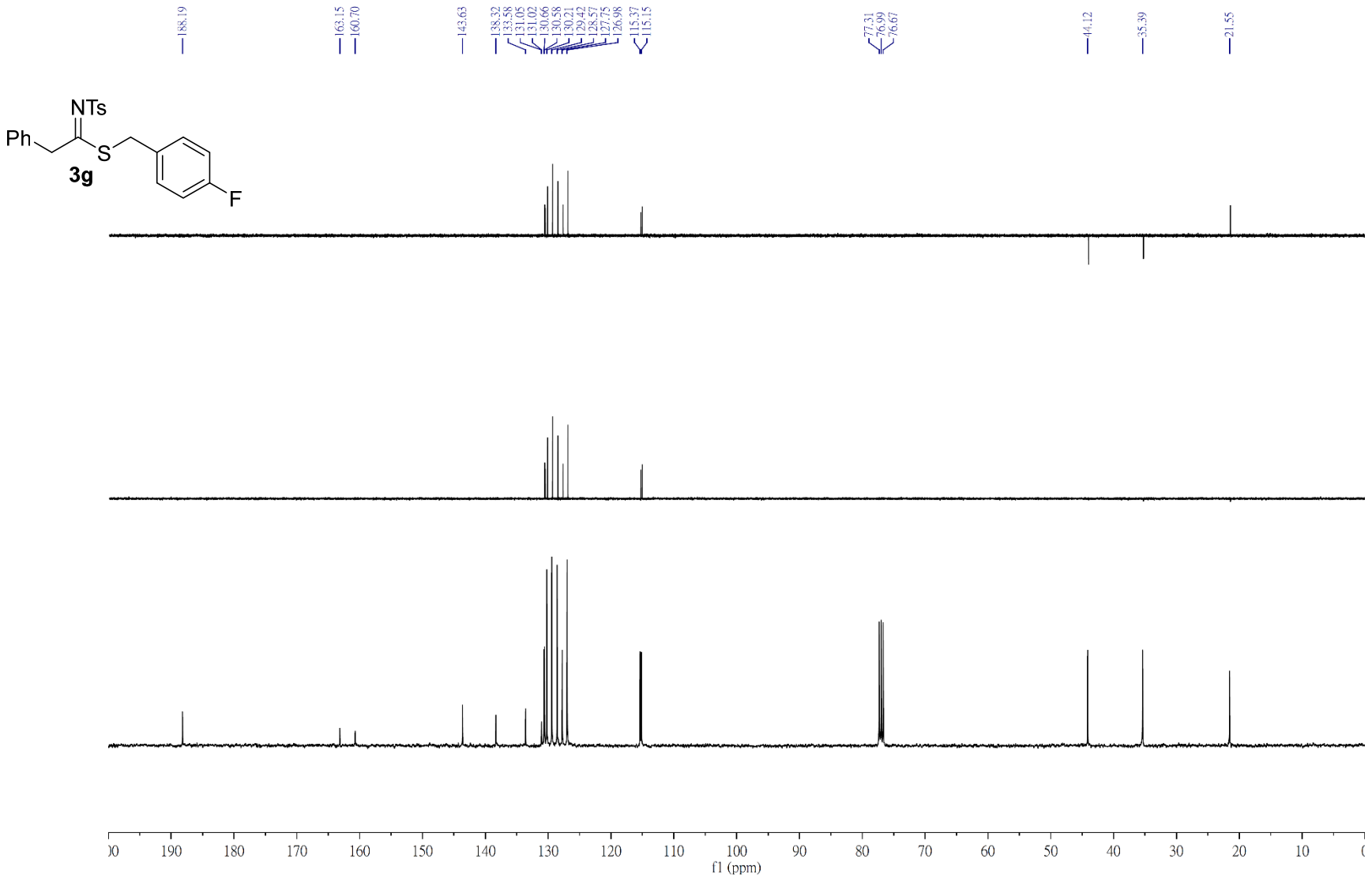
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115.15
77.31
76.99
76.67
44.12
35.39
21.55
NTs
Ph
S
F
3g
f1 (ppm)

Parameter	Value
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Spectrometer	vnmrs
Solvent	cdcl3
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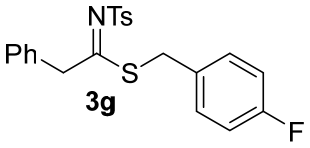
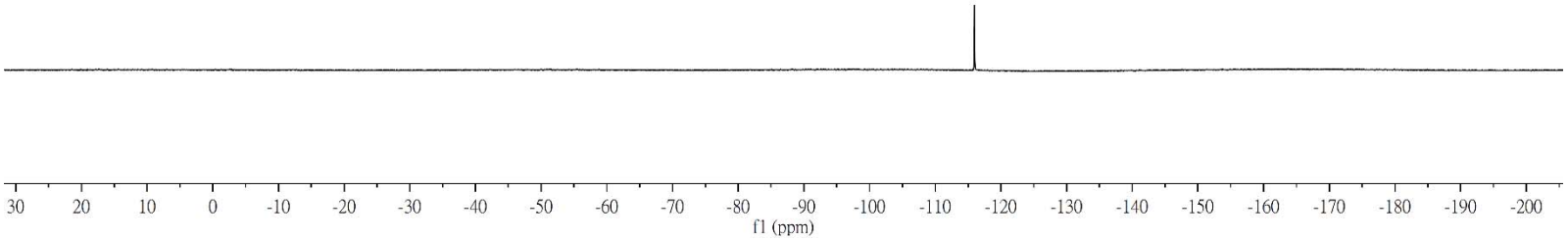
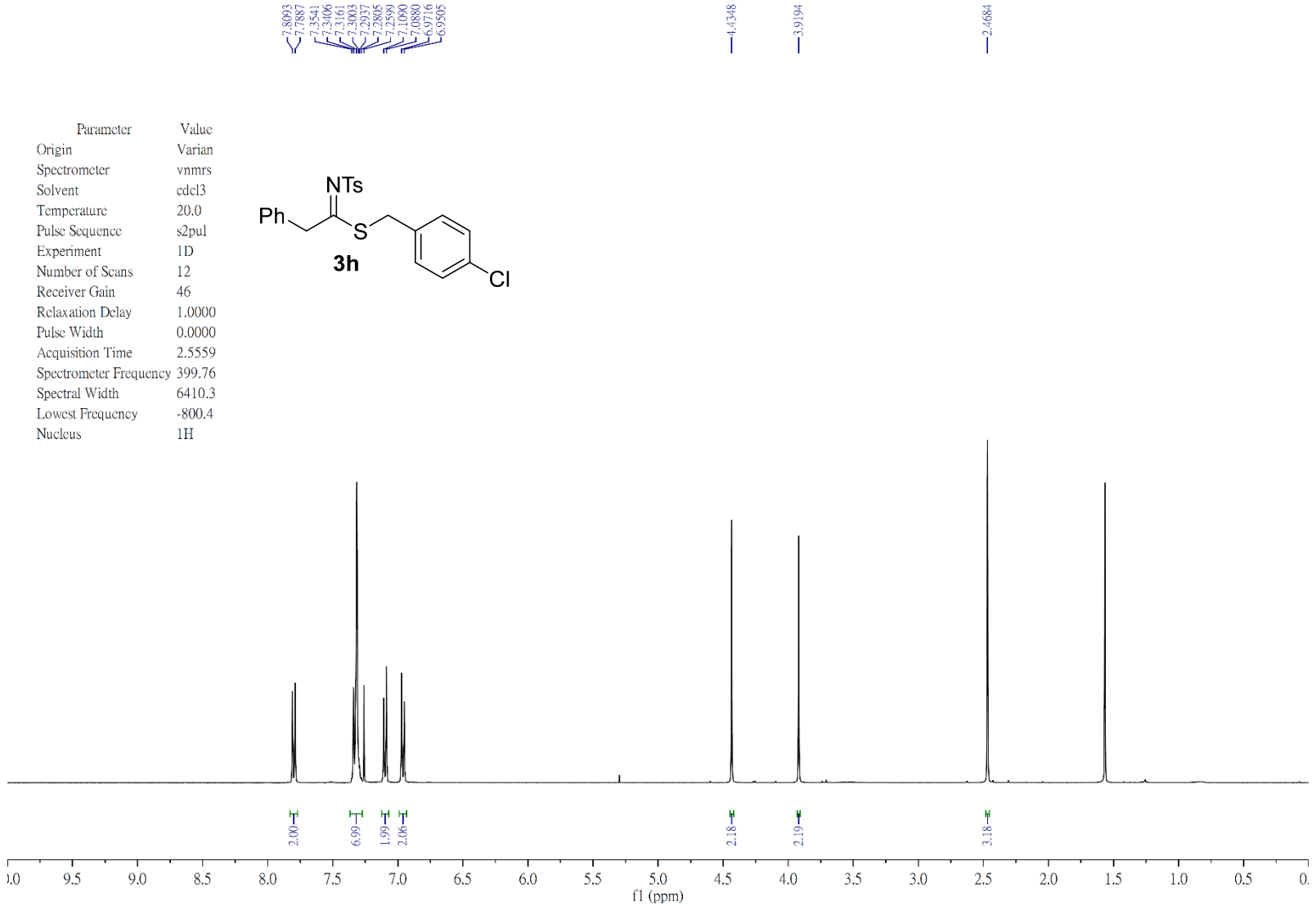
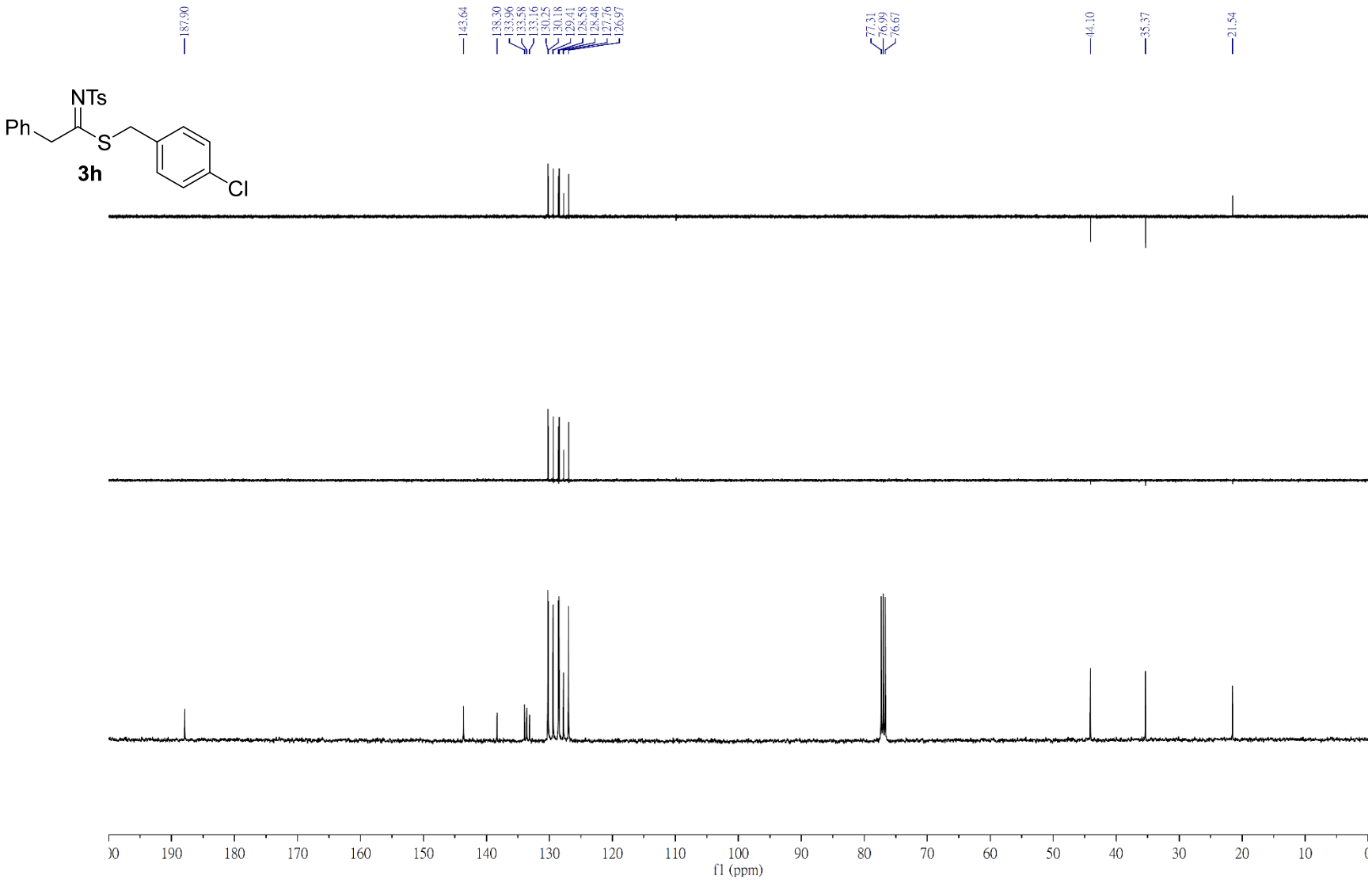


3g

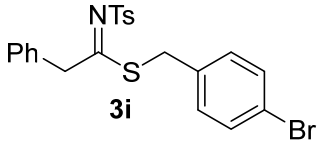
-115.978



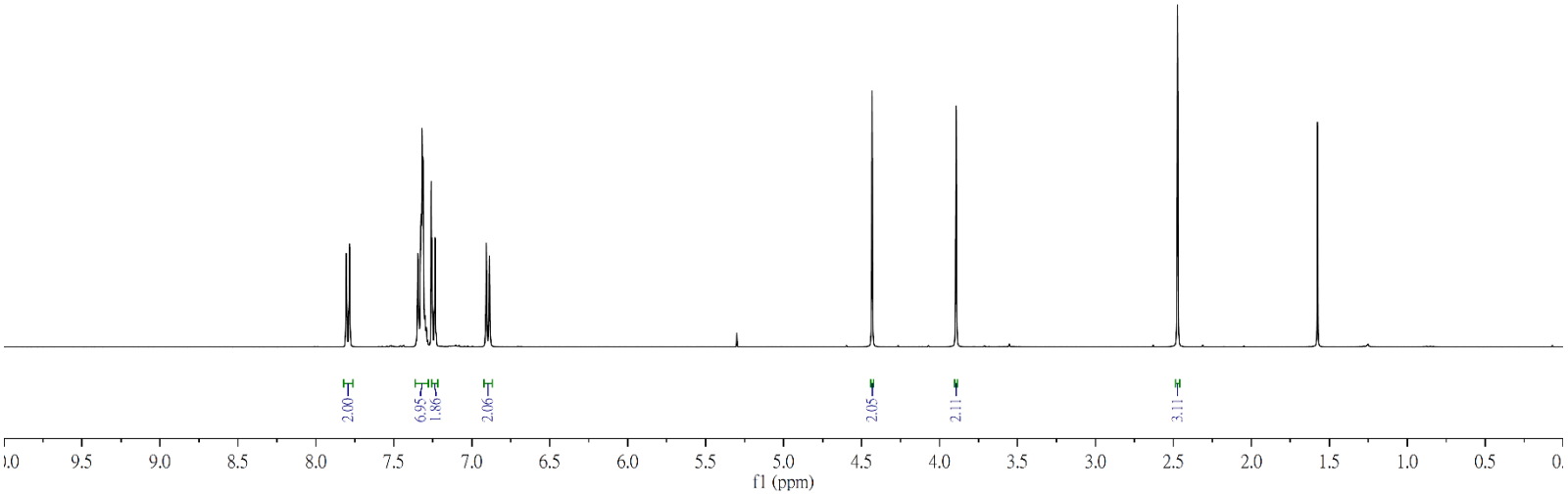

7.8093
7.7887
7.3541
7.3406
7.3161
7.3003
7.2937
7.2805
7.2599
7.1090
7.0880
6.9716
6.9505
4.4348
3.9194
2.4684
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 12
Receiver Gain 46
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.4
Nucleus 1H
NTs
Ph
S
Cl
3h
2.00
6.99
1.99
2.06
2.18
2.19
3.18
f1 (ppm)

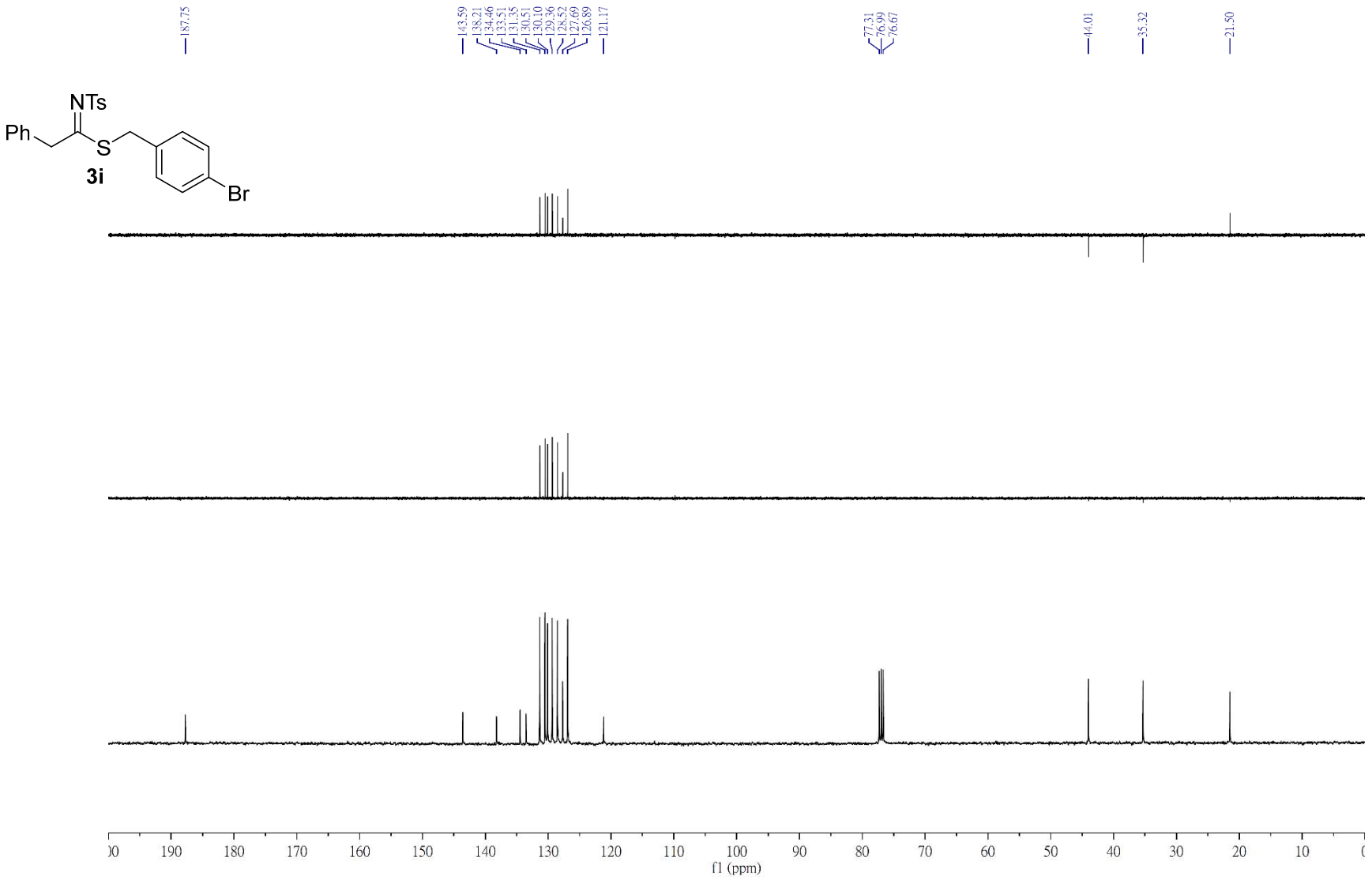

187.90
143.64
138.30
133.96
133.58
133.16
130.25
130.18
129.41
128.58
128.48
127.76
126.97
77.31
76.99
76.67
44.10
35.37
21.54
NTs
Ph
S
Cl
3h
f1 (ppm)

Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	36
Receiver Gain	50
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-800.4
Nucleus	1H



3i

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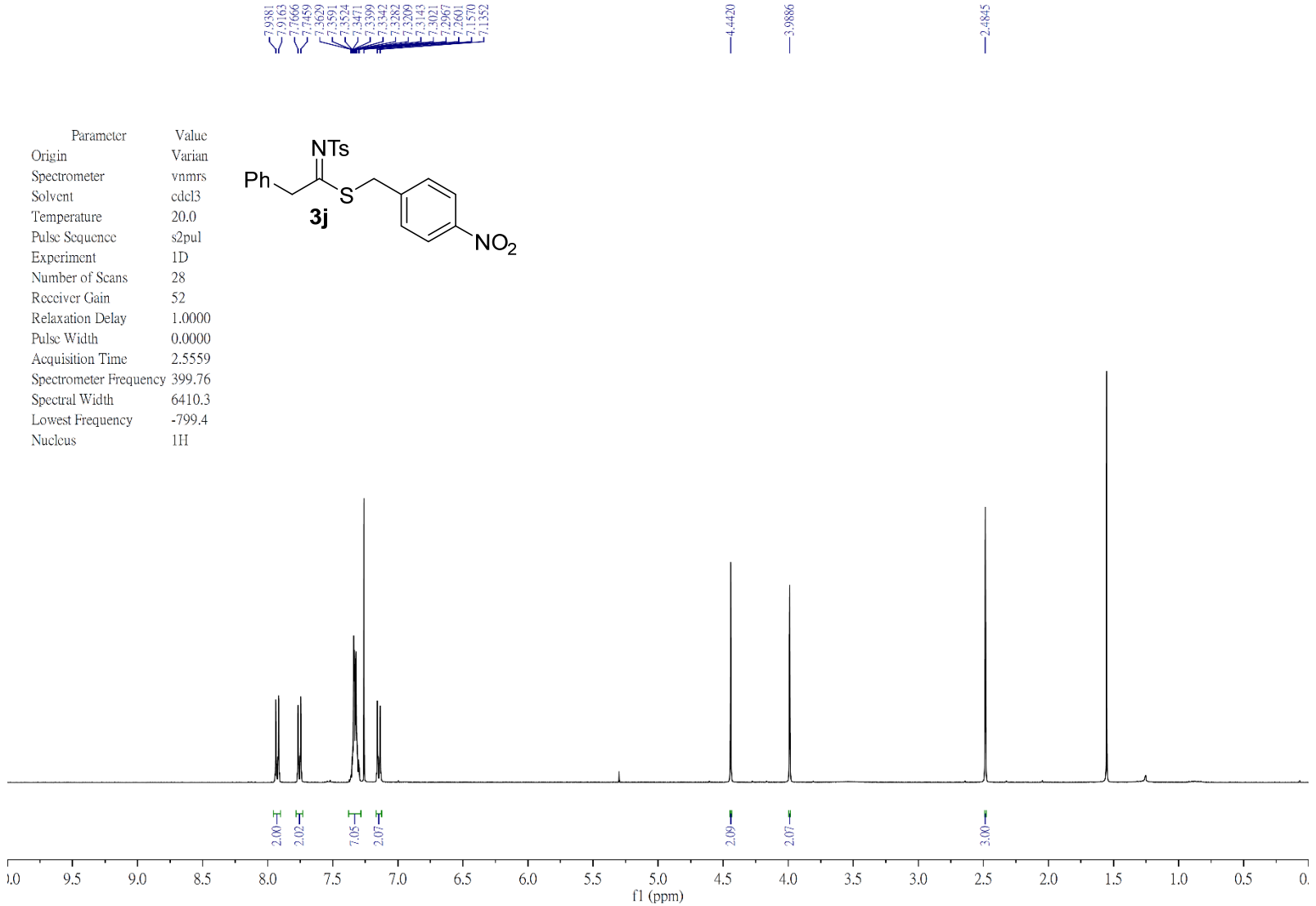
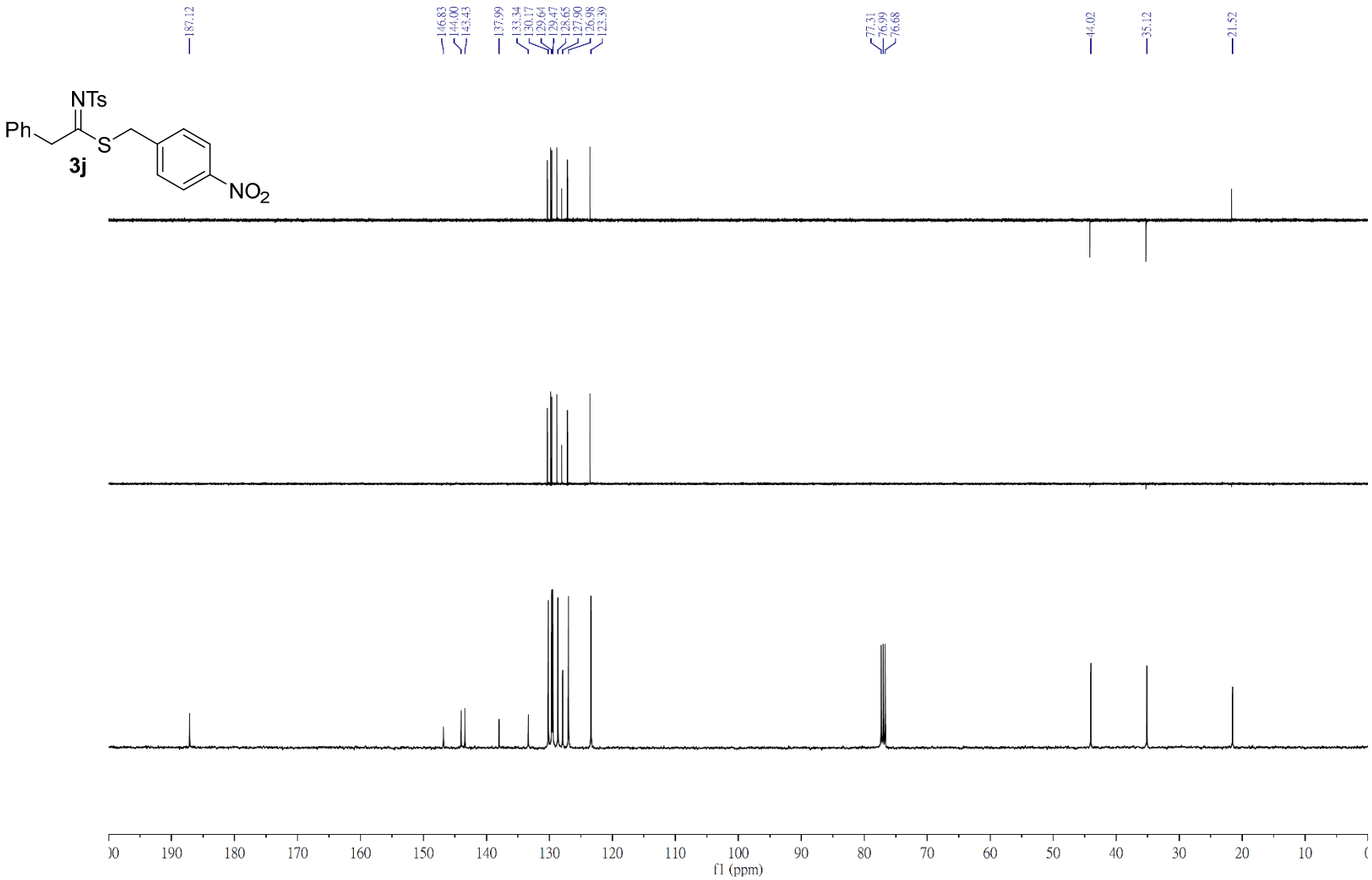
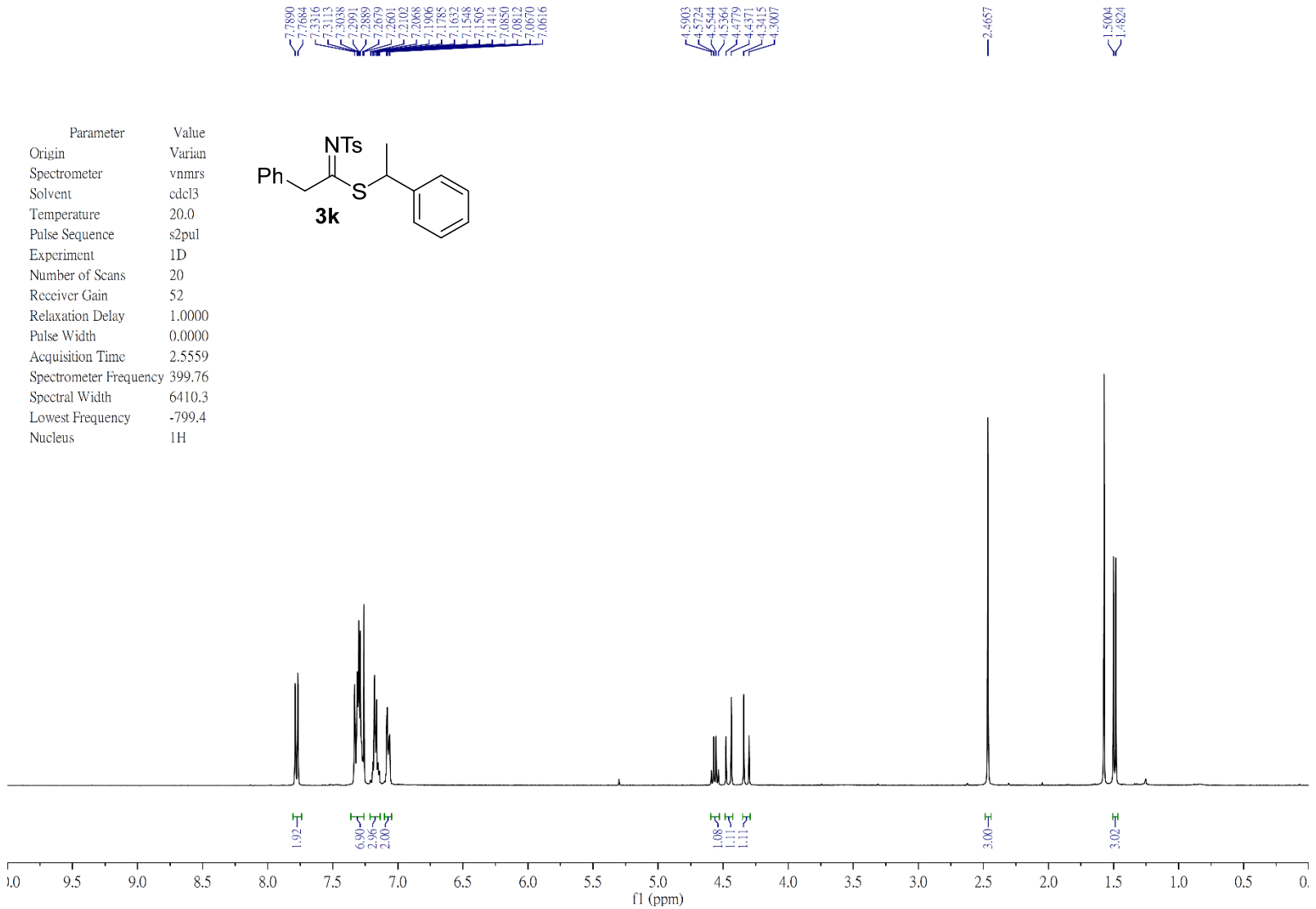
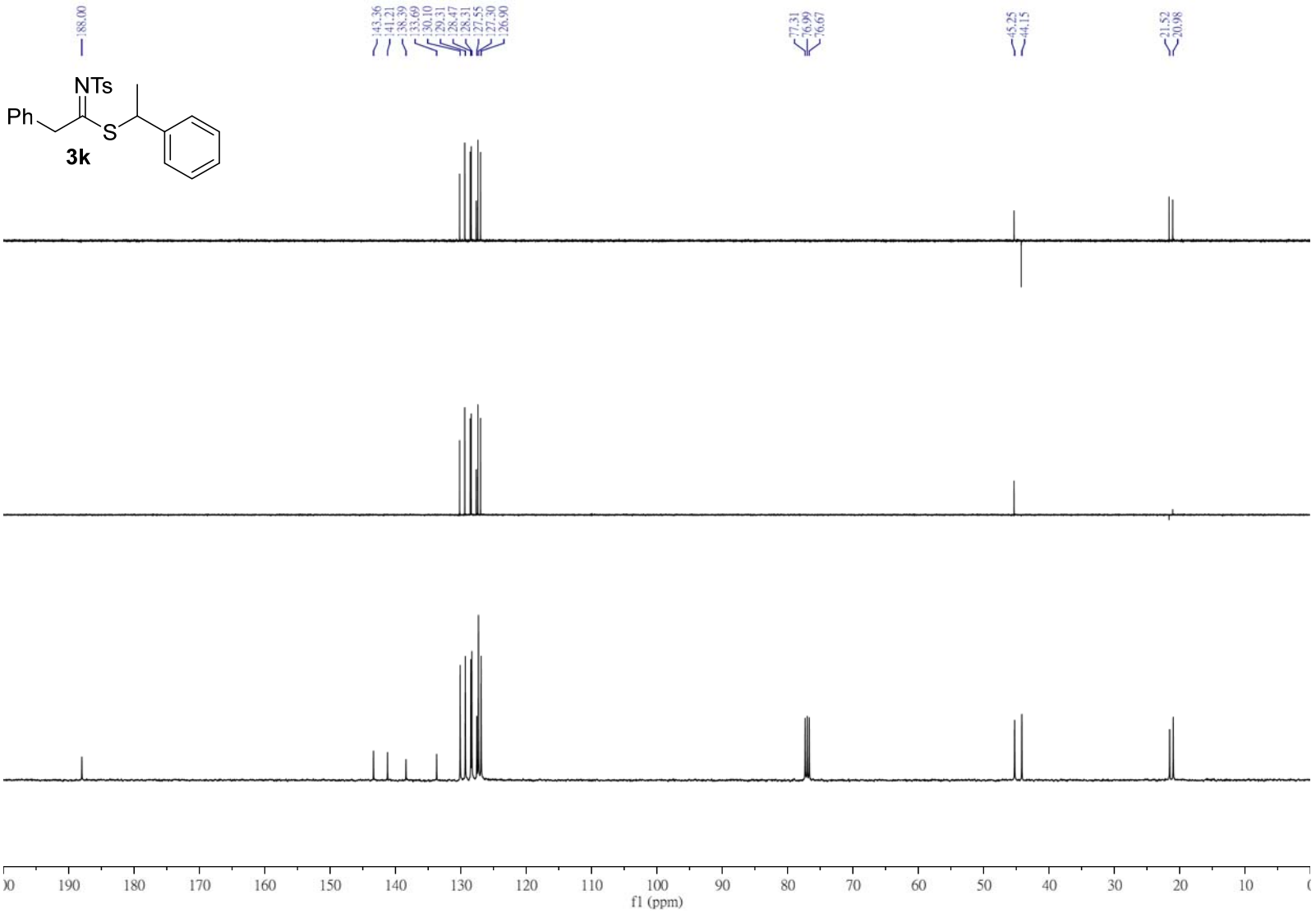
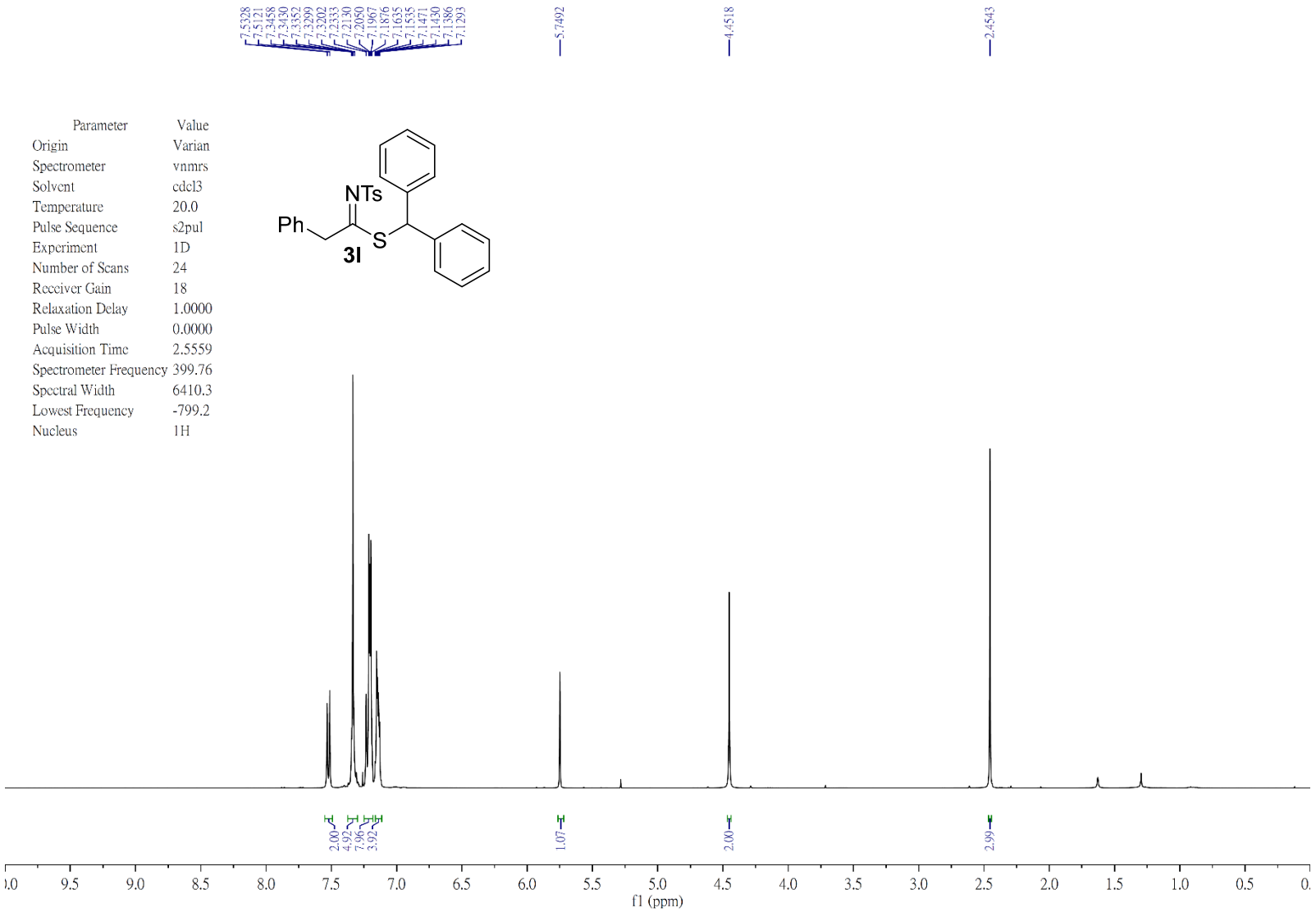
143.59
138.21
134.46
133.51
131.35
130.51
130.10
129.36
128.52
127.69
126.89
121.17
77.31
76.99
76.67
44.01
35.32
21.50
NTs
Ph
S
Br
3i
f1 (ppm)

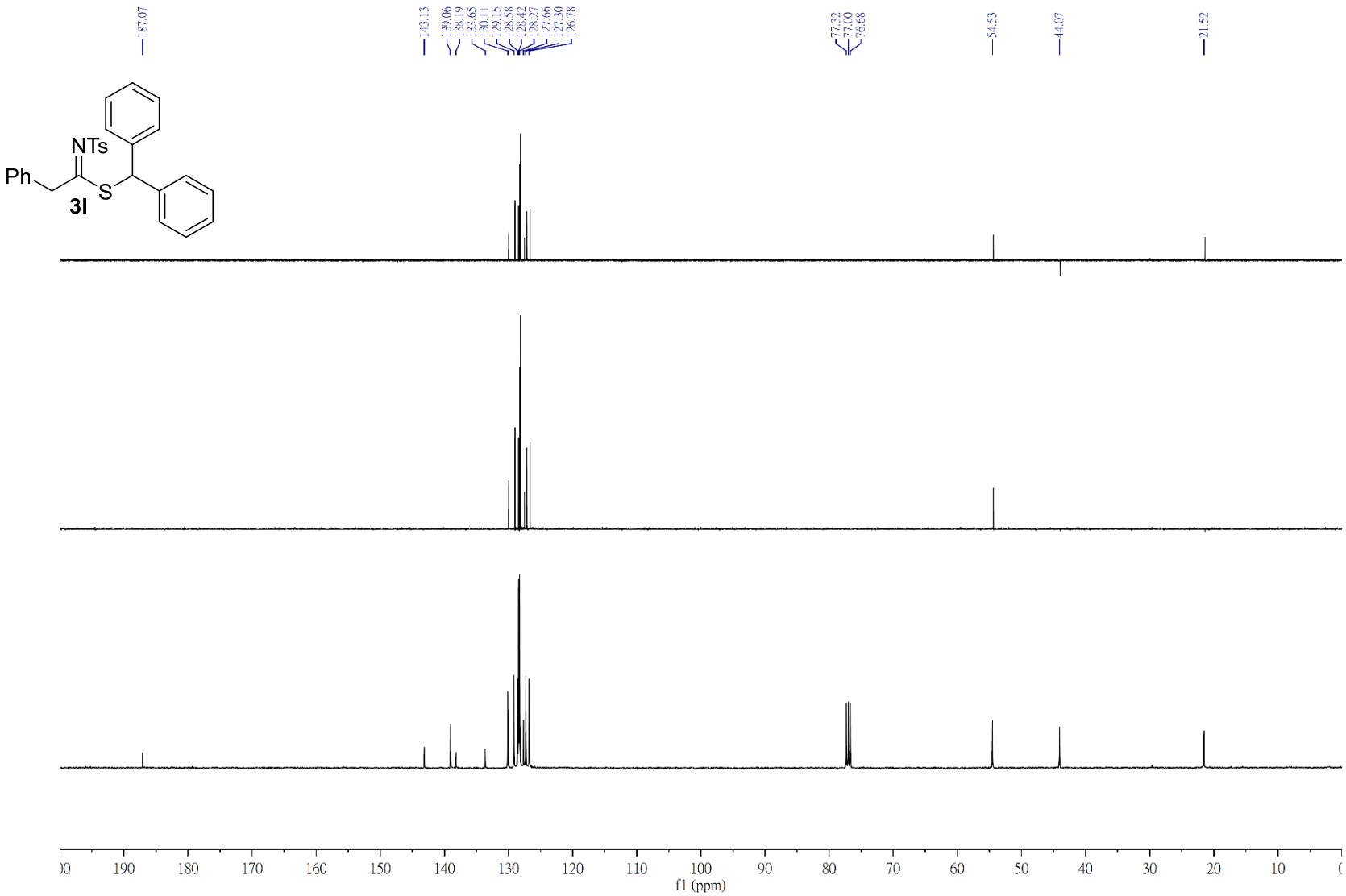

7.9381
7.9163
7.7666
7.7459
7.3629
7.3591
7.3524
7.3471
7.3399
7.3342
7.3282
7.3209
7.3143
7.3021
7.2967
7.2601
7.1570
7.1352
4.4420
3.9886
2.4845
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 28
Receiver Gain 52
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.4
Nucleus 1H
NTs
Ph
S
3j
NO2
2.00
2.02
7.05
2.07
2.09
2.07
3.00
f1 (ppm)

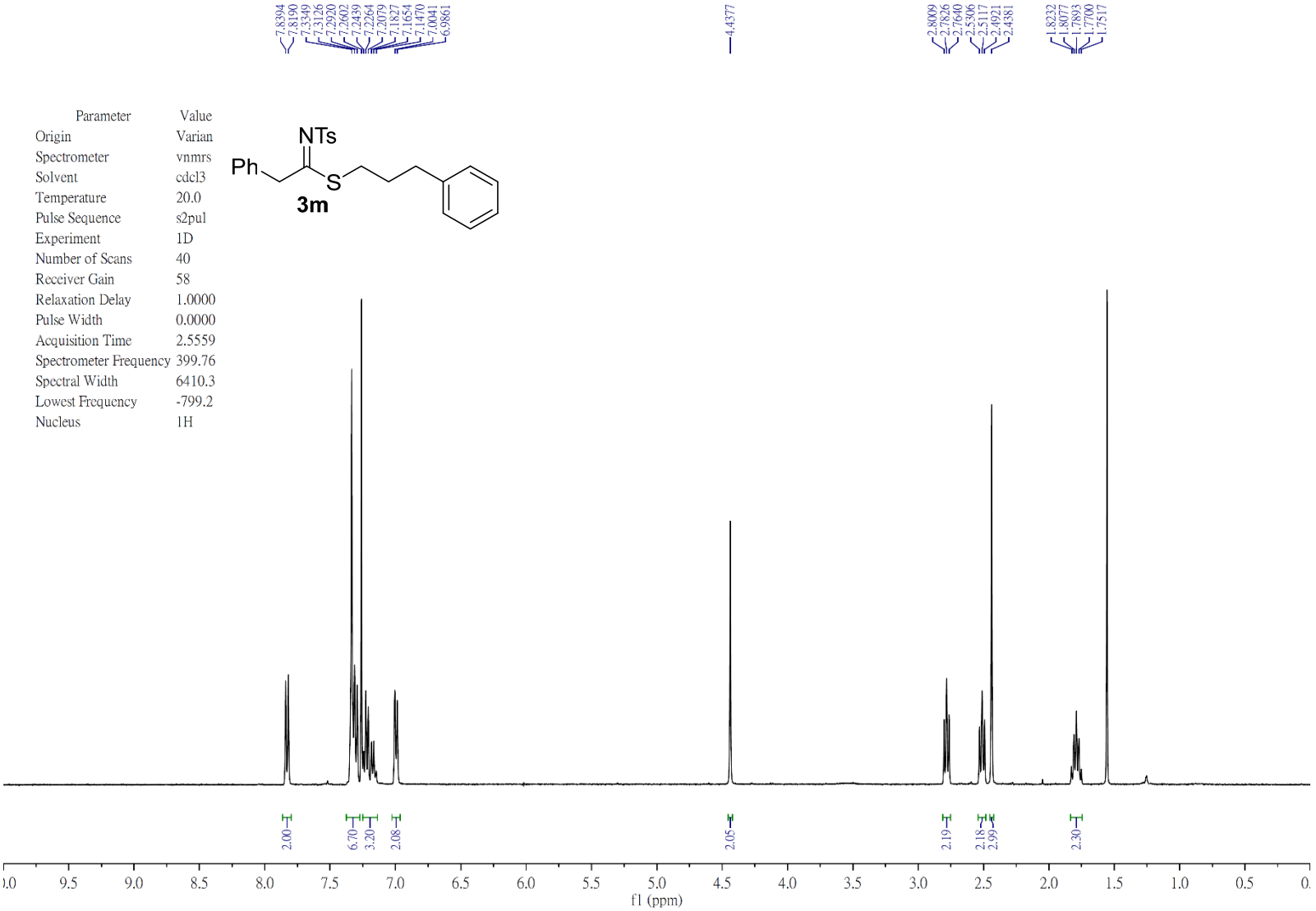

187.12
146.83
144.00
143.43
137.99
133.34
130.17
129.64
129.47
128.65
127.90
126.98
123.39
77.31
76.99
76.68
44.02
35.12
21.52
NTs
Ph
S
3j
NO2
f1 (ppm)

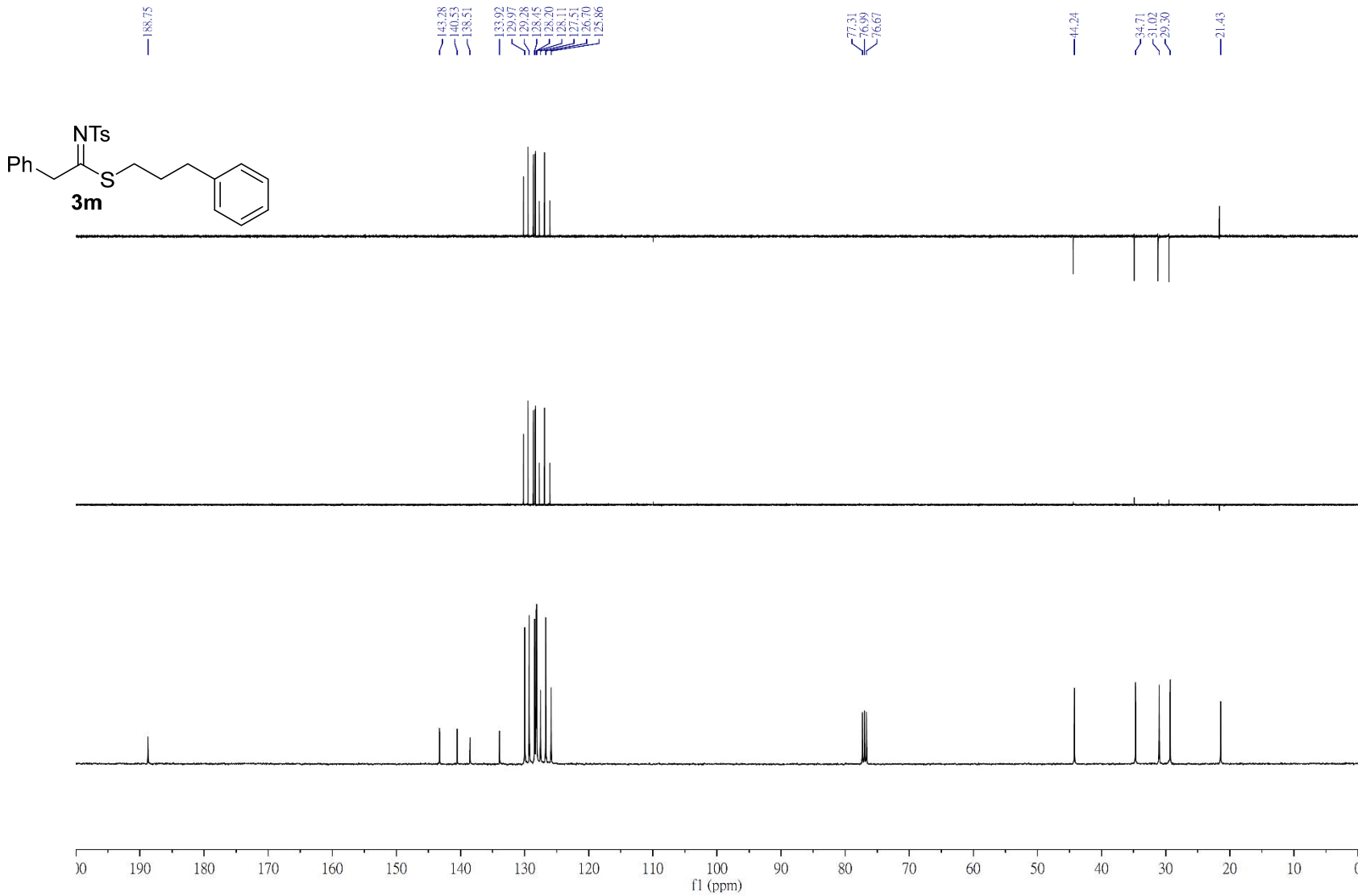

7.7890
7.7664
7.3316
7.3113
7.3038
7.2991
7.2889
7.2679
7.2601
7.2102
7.2068
7.1906
7.1785
7.1632
7.1548
7.1505
7.1414
7.0850
7.0812
7.0670
7.0616
4.5903
4.5724
4.5544
4.5364
4.4779
4.4371
4.3415
4.3007
2.4657
1.5004
1.4824
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 20
Receiver Gain 52
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.4
Nucleus 1H
NTs
Ph
S
3k
1.92
6.90
2.96
2.00
1.08
1.11
1.11
3.00
3.02
f1 (ppm)


188.00
143.36
141.21
138.39
133.69
130.10
129.31
128.47
128.31
127.55
127.30
126.90
77.31
76.99
76.67
45.25
44.15
21.52
20.98
NTs
Ph
S
3k
f1 (ppm)


7.5328
7.5121
7.3458
7.3430
7.3352
7.3299
7.3202
7.2333
7.2130
7.2050
7.1967
7.1876
7.1635
7.1535
7.1471
7.1430
7.1386
7.1293
5.7492
4.4518
2.4543
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 24
Receiver Gain 18
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.2
Nucleus 1H
NTs
Ph
S
3l
2.00
4.92
7.96
3.92
1.07
2.00
2.99
f1 (ppm)


187.07
143.13
139.06
138.19
133.65
130.11
129.15
128.58
128.42
128.27
127.66
127.30
126.78
77.32
77.00
76.68
54.53
44.07
21.52
NTs
Ph
S
3l
f1 (ppm)


7.8394
7.8190
7.3349
7.3126
7.2920
7.2602
7.2439
7.2264
7.2079
7.1827
7.1654
7.1470
7.0041
6.9861
4.4377
2.8009
2.7826
2.7640
2.5306
2.5117
2.4921
2.4381
1.8232
1.8077
1.7893
1.7700
1.7517
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 40
Receiver Gain 58
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.2
Nucleus 1H
NTs
Ph
S
3m
2.00
6.70
3.20
2.08
2.05
2.19
2.18
2.99
2.30
f1 (ppm)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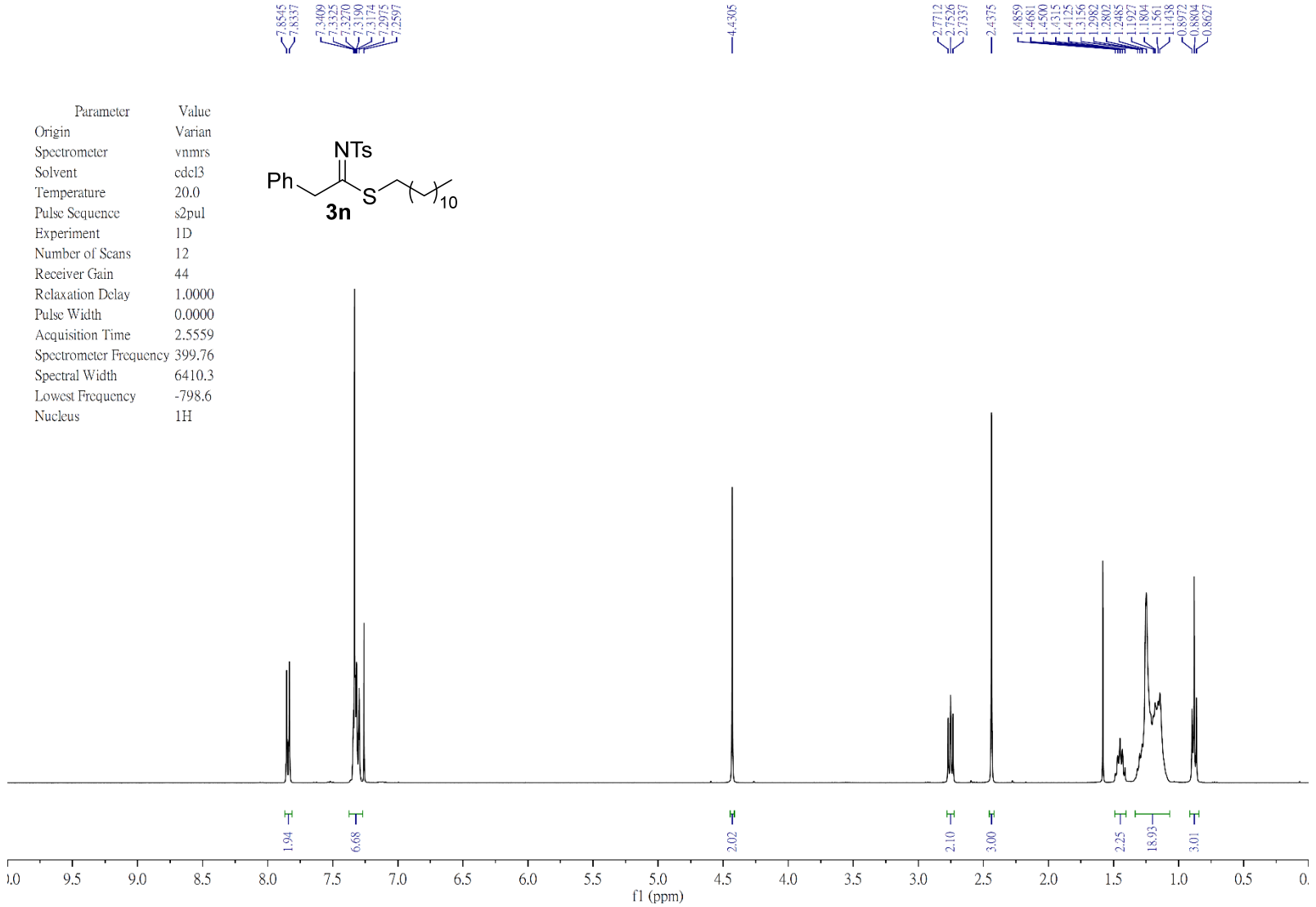
143.28
140.53
138.51
133.92
129.97
129.28
128.45
128.20
128.11
127.51
126.70
125.86
77.31
76.99
76.67
44.24
34.71
31.02
29.30
21.43
NTs
Ph
S
3m
f1 (ppm)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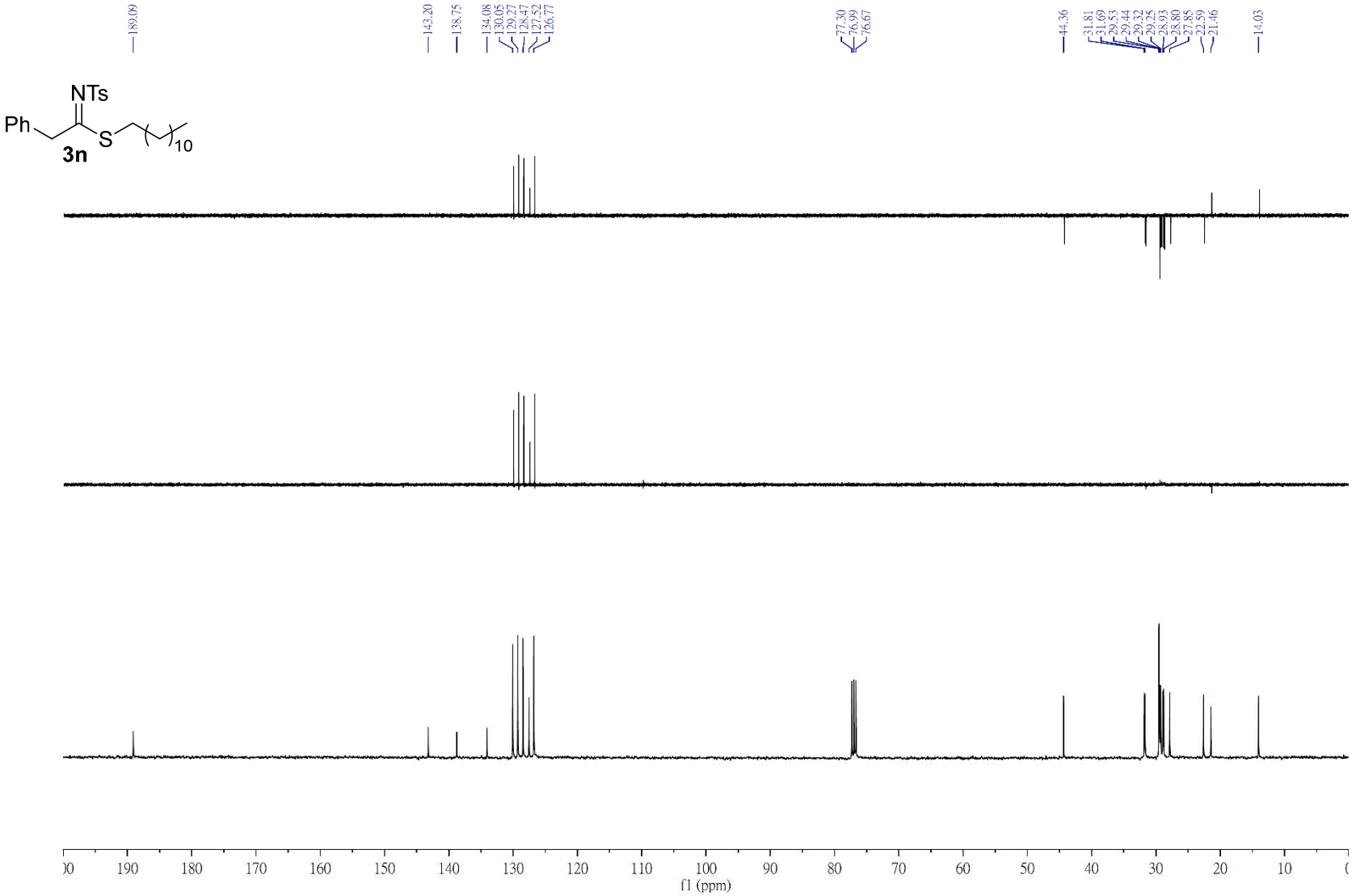
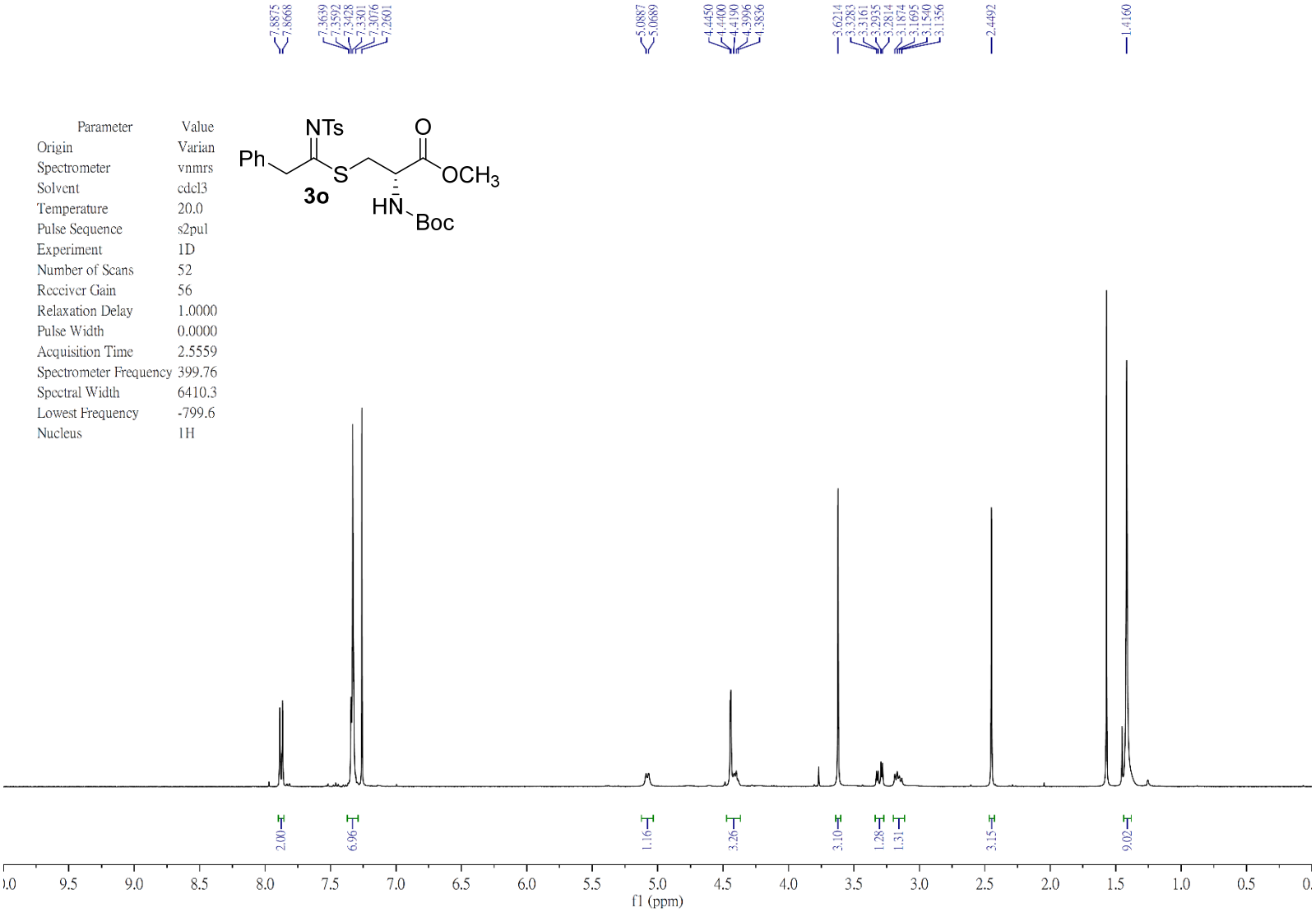
7.3409
7.3325
7.3270
7.3190
7.3174
7.2975
7.2597
4.4305
2.7712
2.7526
2.7337
2.4375
1.4859
1.4681
1.4500
1.4315
1.4125
1.3156
1.2982
1.2802
1.2485
1.1927
1.1804
1.1561
1.1438
0.8972
0.8804
0.8627
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 12
Receiver Gain 44
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -798.6
Nucleus 1H
NTs
Ph
S
10
3n
1.94
6.68
2.02
2.10
3.00
2.25
18.93
3.01
f1 (ppm)


189.09
143.20
138.75
134.08
130.05
129.27
128.47
127.52
126.77
77.30
76.99
76.67
44.36
31.81
31.69
29.53
29.44
29.32
29.25
28.93
28.80
27.85
22.59
21.46
14.03
NTs
Ph
S
10
3n
f1 (ppm)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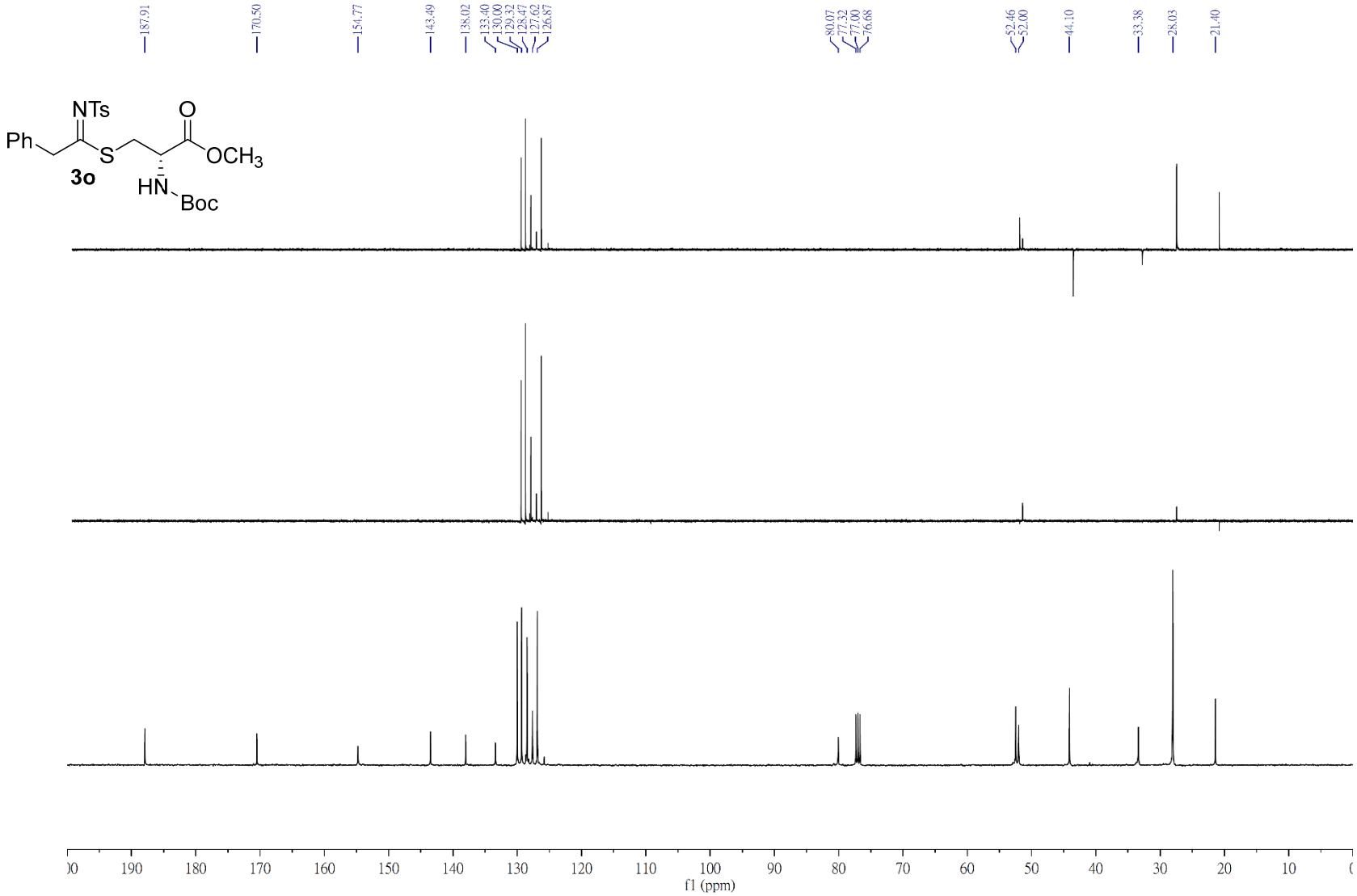
7.3639
7.3592
7.3428
7.3301
7.3076
7.2601
5.0887
5.0689
4.4450
4.4400
4.4190
4.3996
4.3836
3.6214
3.3283
3.3161
3.2935
3.2814
3.1874
3.1695
3.1540
3.1356
2.4492
1.4160
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 52
Receiver Gain 56
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
NTs
O
Ph
S
OCH3
3o
HN
Boc
2.00
6.96
1.16
3.26
3.10
1.28
1.31
3.15
9.02
f1 (ppm)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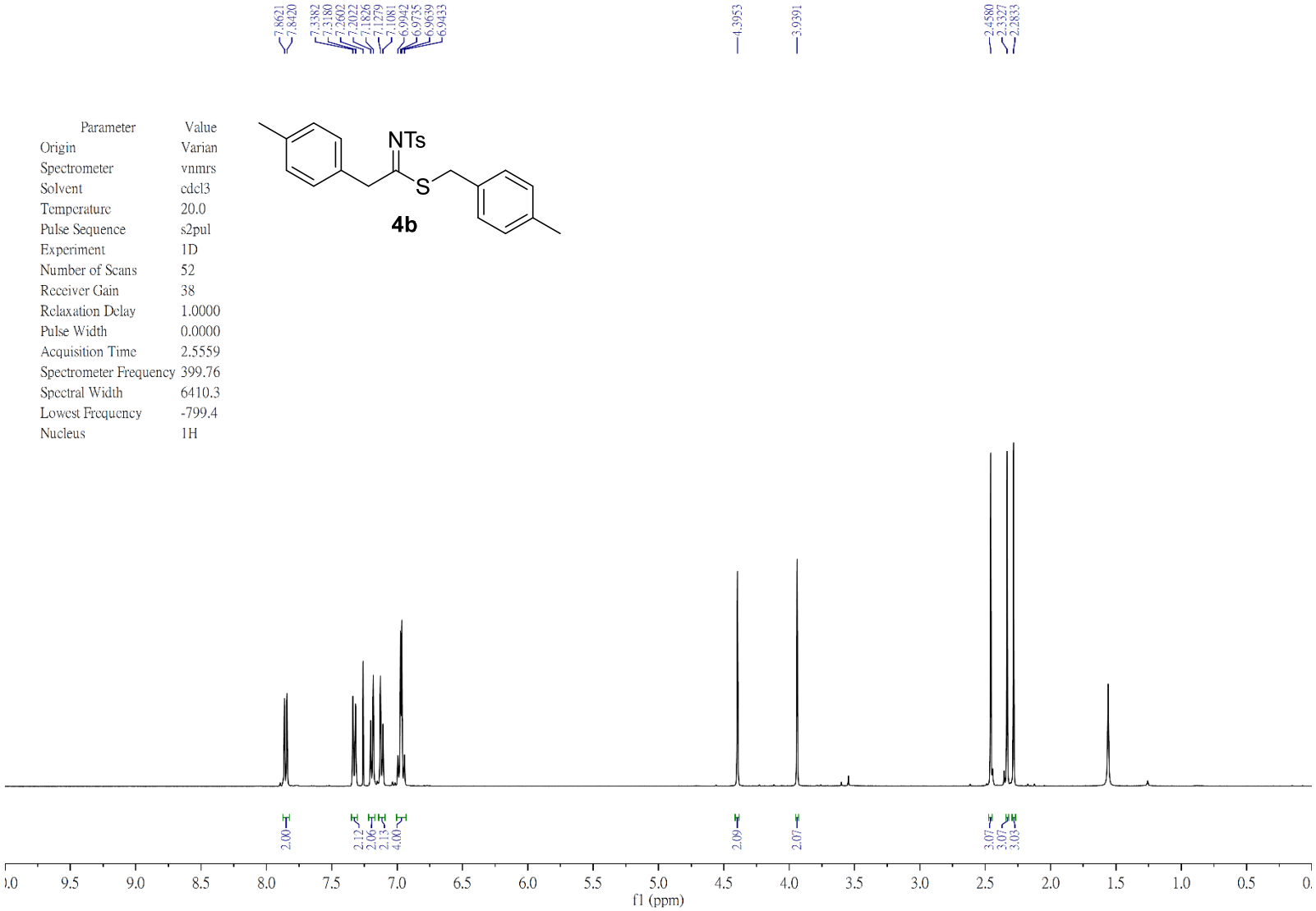
170.50
154.77
143.49
138.02
133.40
130.00
129.32
128.47
127.62
126.87
80.07
77.32
77.00
76.68
52.46
52.00
44.10
33.38
28.03
21.40
NTs
O
Ph
S
OCH3
3o
HN
Boc
f1 (ppm)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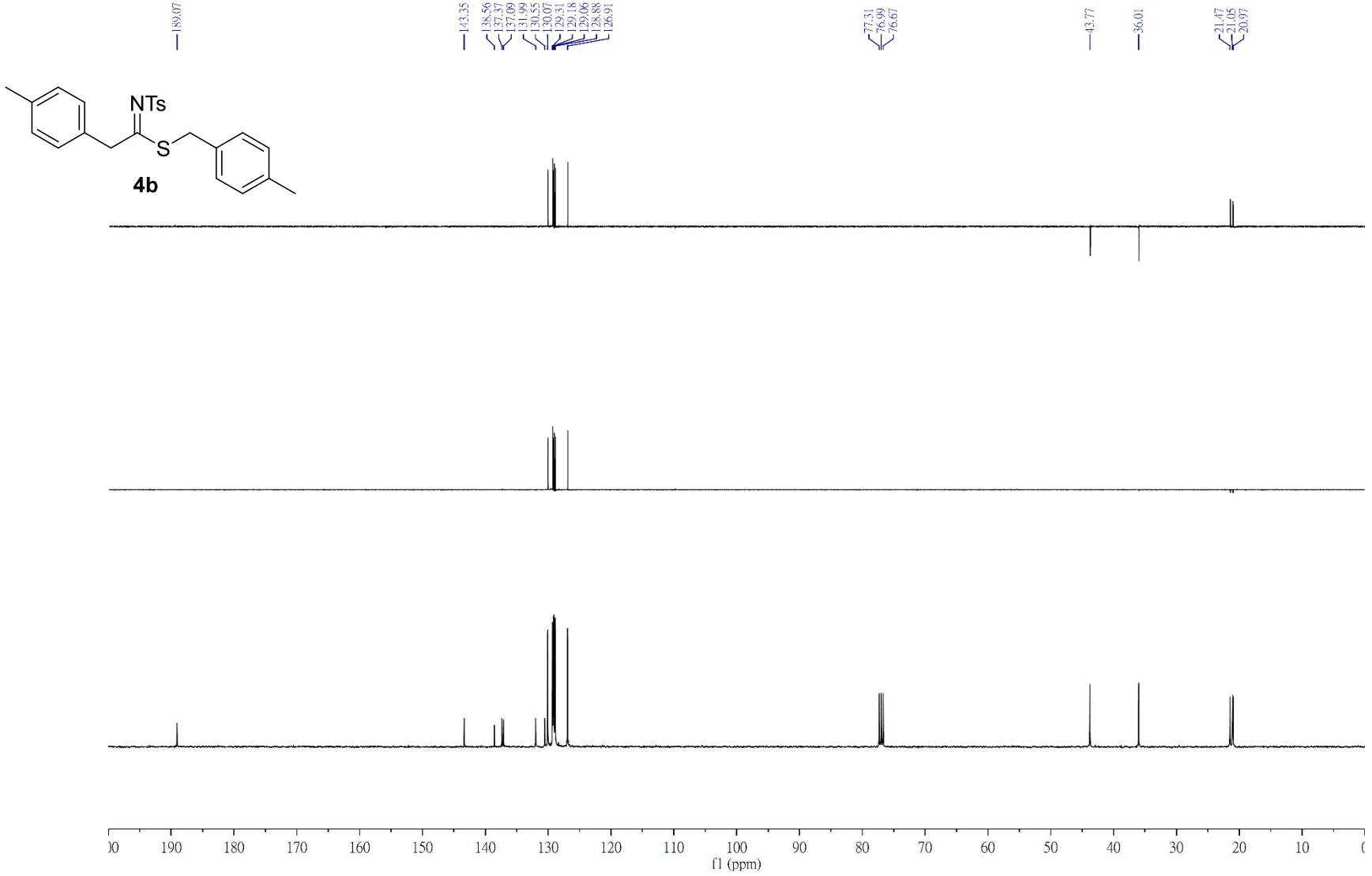
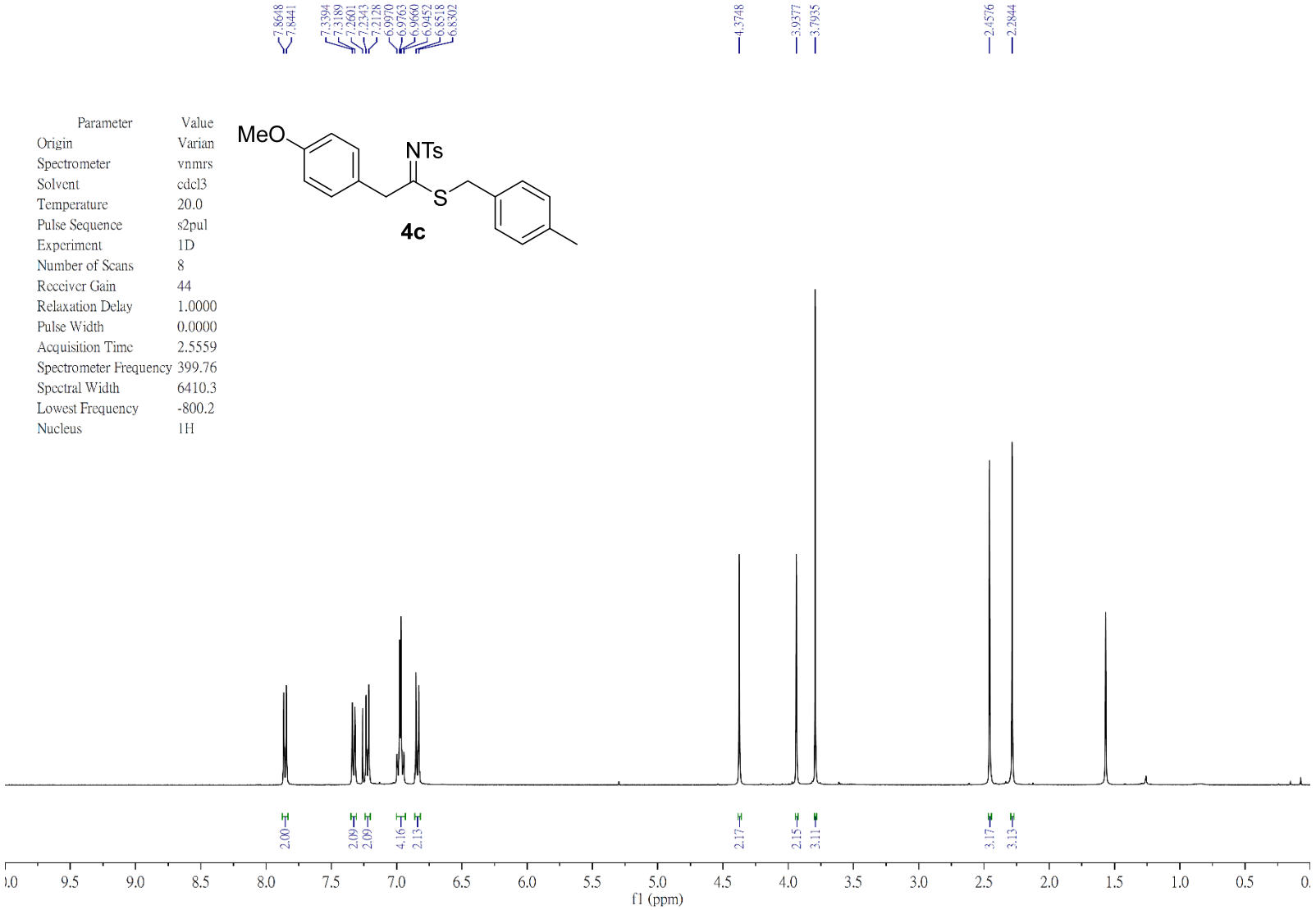
7.3382
7.3180
7.2602
7.2022
7.1826
7.1279
7.1081
6.9942
6.9735
6.9639
6.9433
4.3953
3.9391
2.4580
2.3327
2.2833
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 52
Receiver Gain 38
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.4
Nucleus 1H
NTs
S
4b
2.00
2.12
2.06
2.13
4.00
2.09
2.07
3.07
3.07
3.03
f1 (ppm)


189.07
143.35
138.56
137.37
137.09
131.99
130.55
130.07
129.31
129.18
129.06
128.88
126.91
77.31
76.99
76.67
43.77
36.01
21.47
21.05
20.97
NTs
S
4b
f1 (ppm)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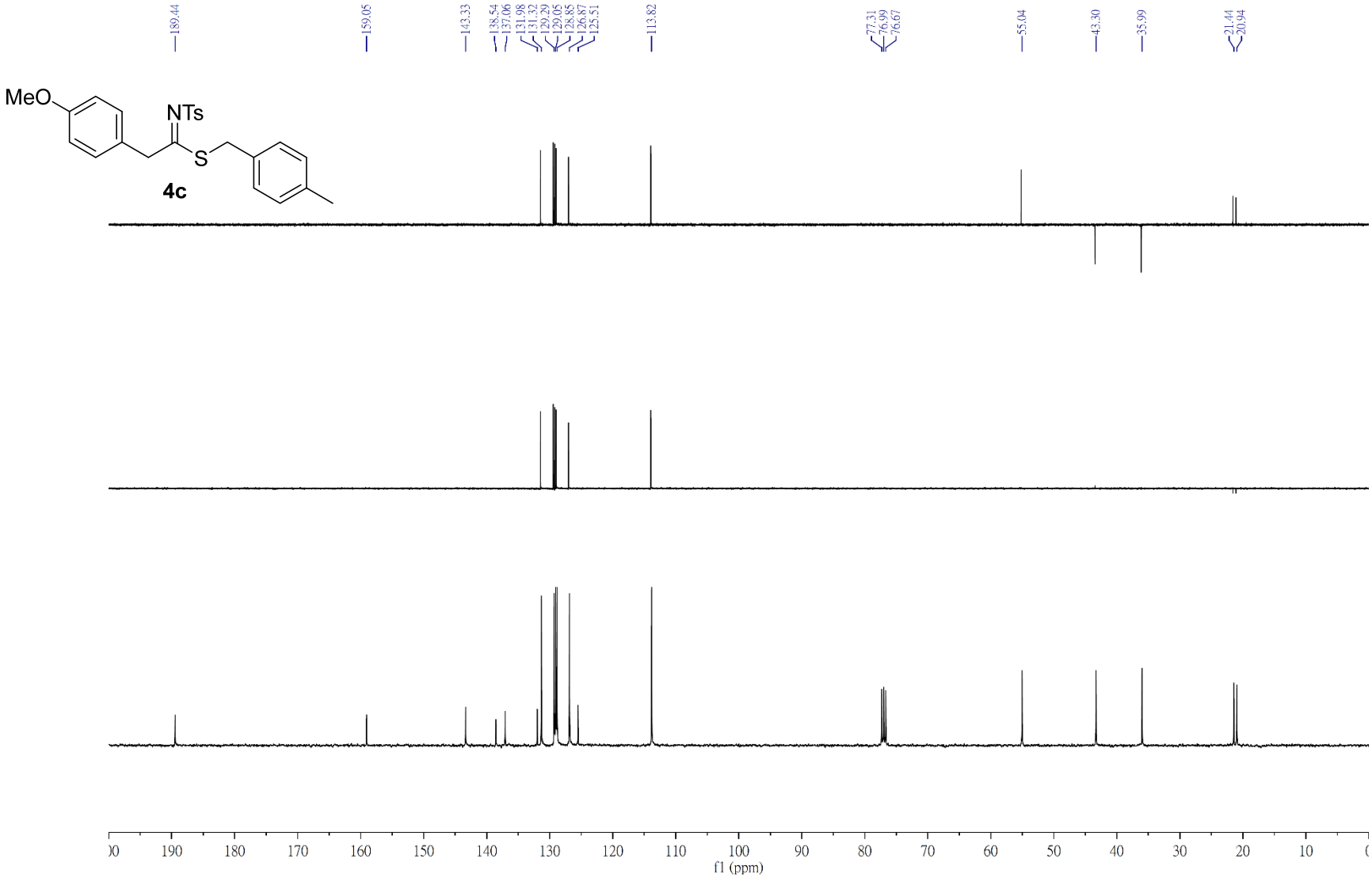
7.3189
7.2601
7.2343
7.2128
6.9970
6.9763
6.9660
6.9452
6.8518
6.8302
4.3748
3.9377
3.7935
2.4576
2.2844
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 8
Receiver Gain 44
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.2
Nucleus 1H
MeO
NTs
S
4c
2.00
2.09
2.09
4.16
2.13
2.17
2.15
3.11
3.17
3.13
f1 (ppm)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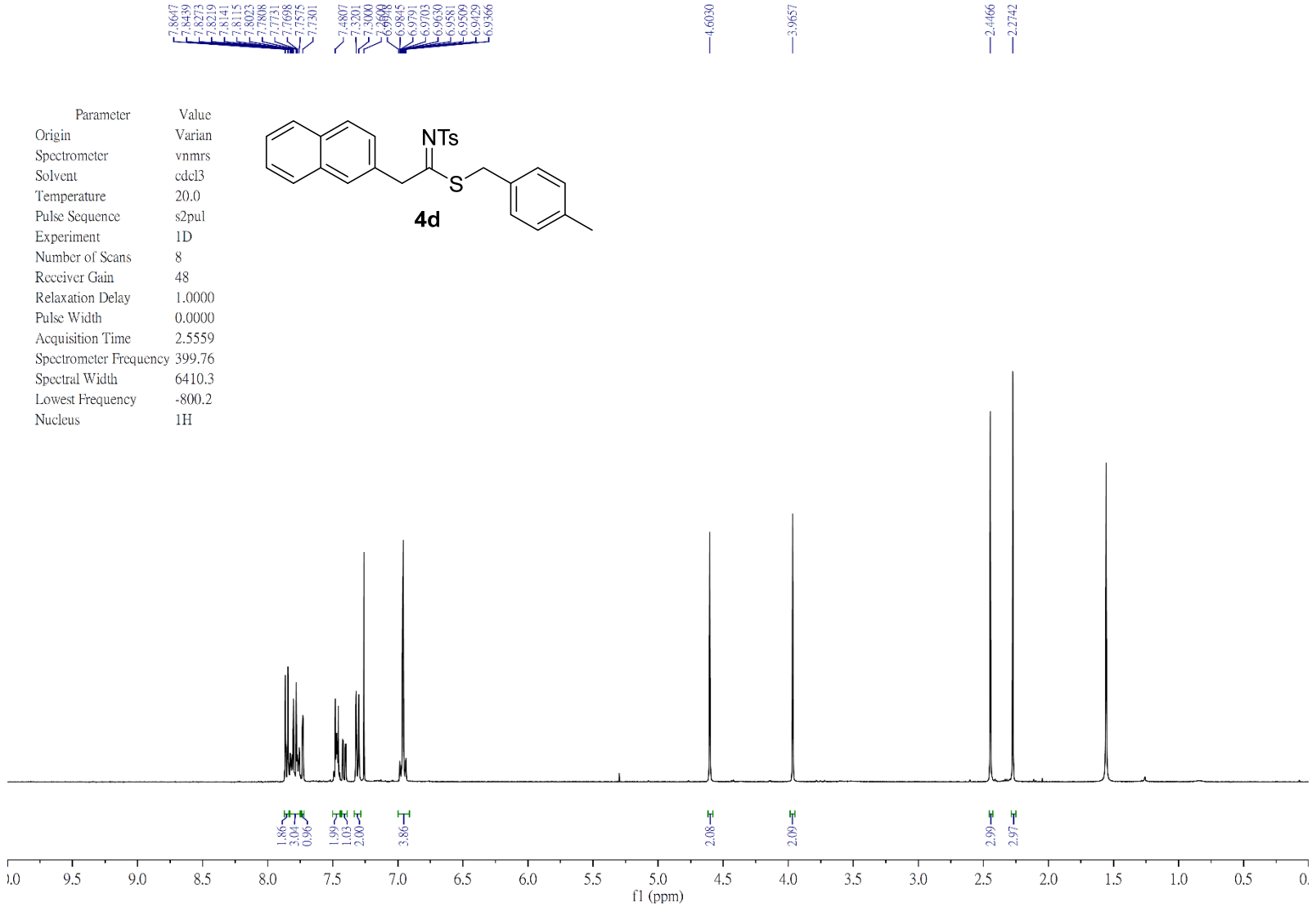
138.54
137.06
131.98
131.32
129.29
129.05
128.85
126.87
125.51
113.82
77.31
76.99
76.67
55.04
43.30
35.99
21.44
20.94
MeO
NTs
S
4c
f1 (ppm)


7.8647
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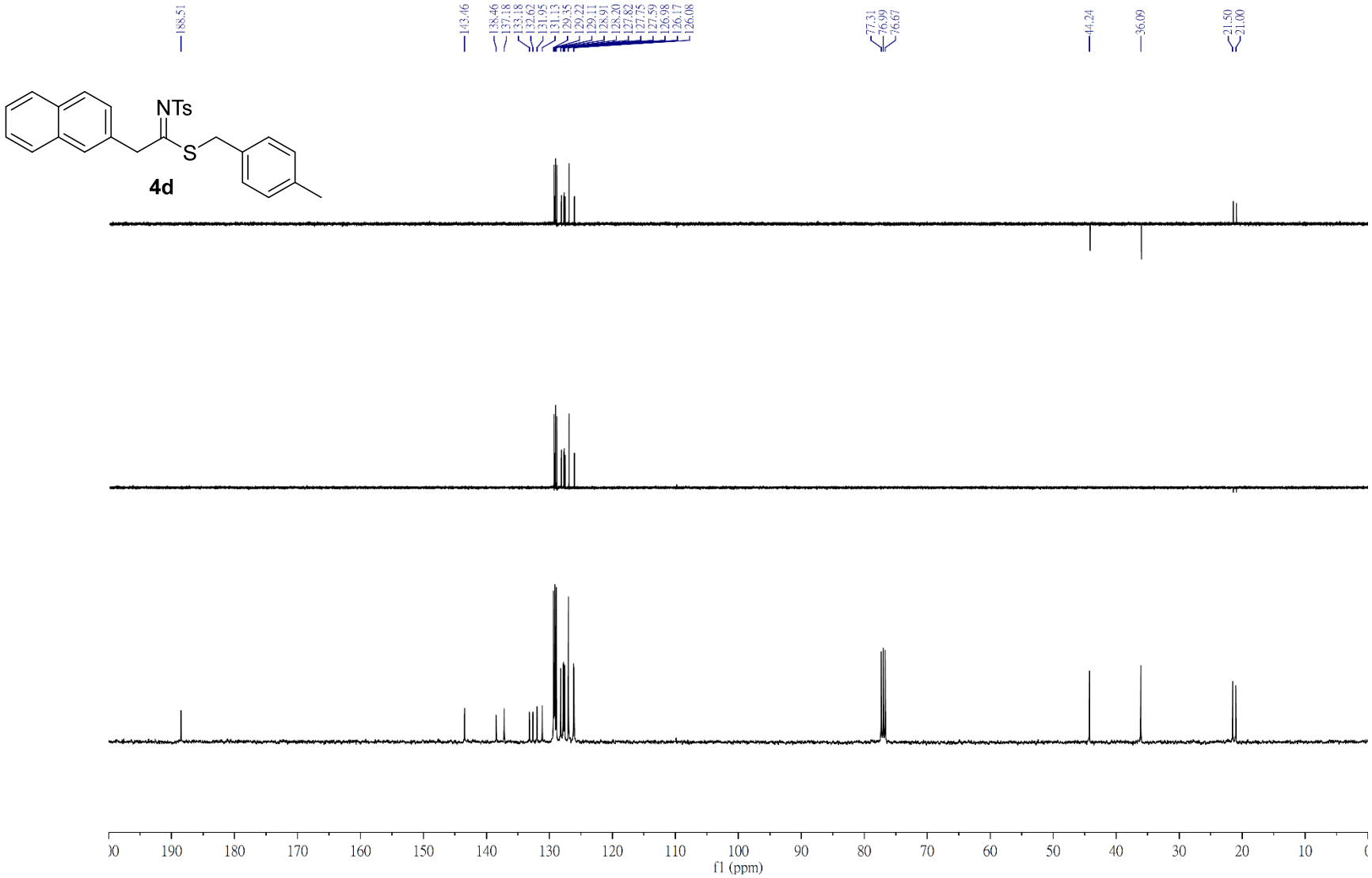
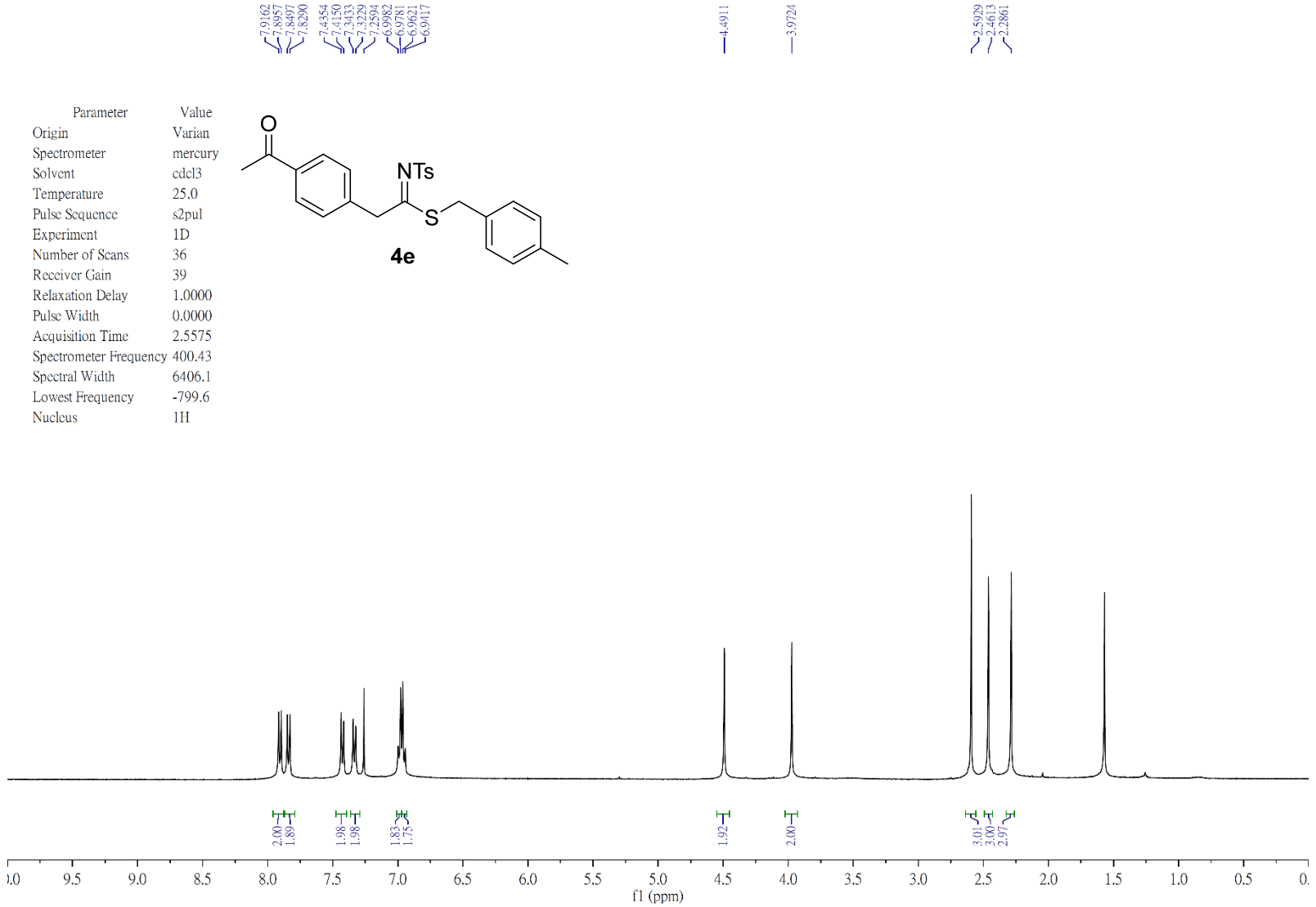
7.8273
7.8219
7.8141
7.8115
7.8023
7.7808
7.7731
7.7698
7.7575
7.7301
7.4807
7.3201
7.3000
7.2600
6.9845
6.9791
6.9703
6.9630
6.9581
6.9509
6.9429
6.9366
4.6030
3.9657
2.4466
2.2742
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 8
Receiver Gain 48
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.2
Nucleus 1H
NTs
S
4d
1.86
3.04
0.96
1.99
1.03
2.00
3.86
2.08
2.09
2.99
2.97
f1 (ppm)


188.51
143.46
138.46
137.18
133.18
132.62
131.95
131.13
129.35
129.22
129.11
128.91
128.20
127.82
127.75
127.59
126.98
126.17
126.08
77.31
76.99
76.67
44.24
36.09
21.50
21.00
NTs
S
4d
f1 (ppm)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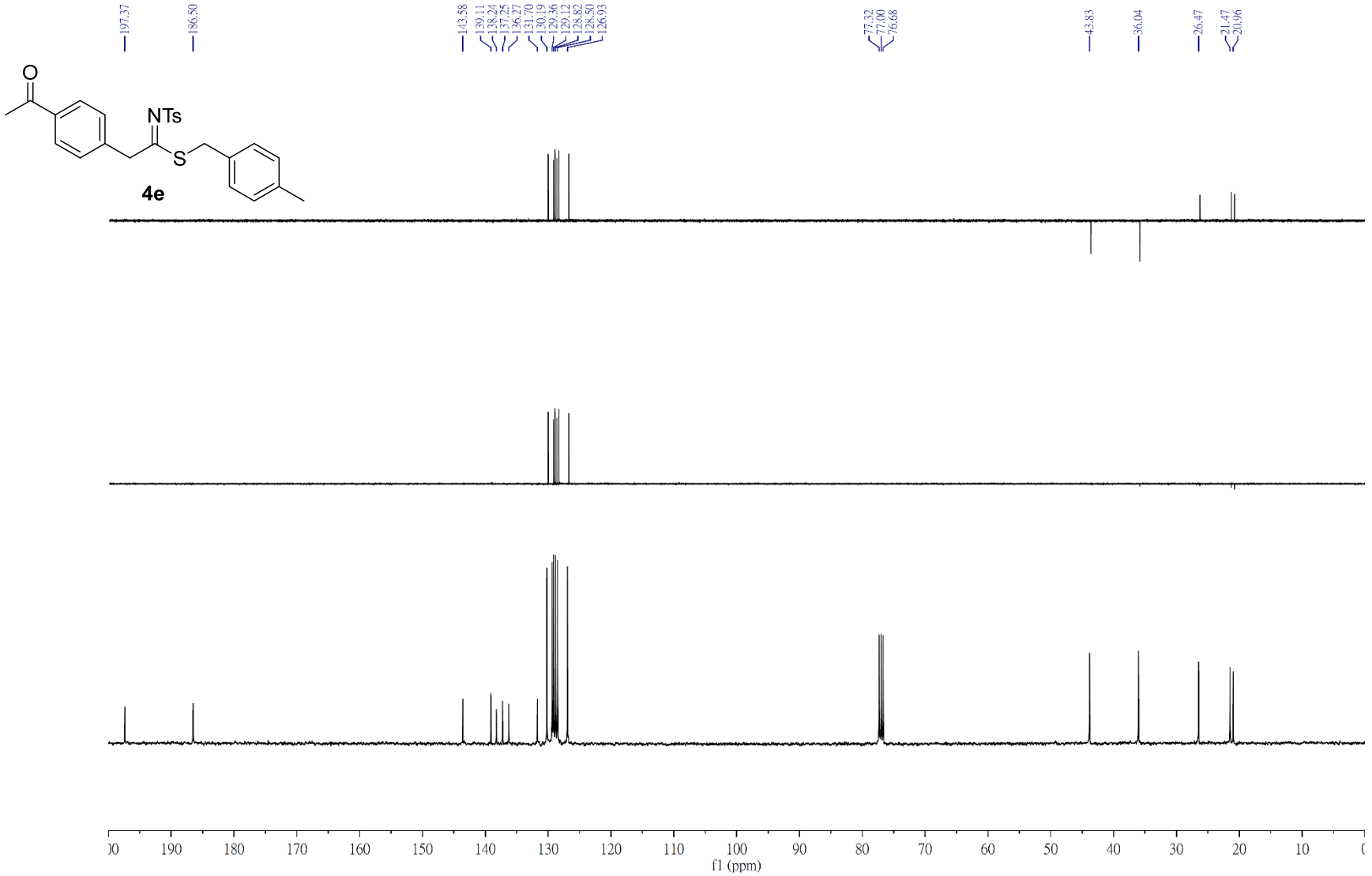
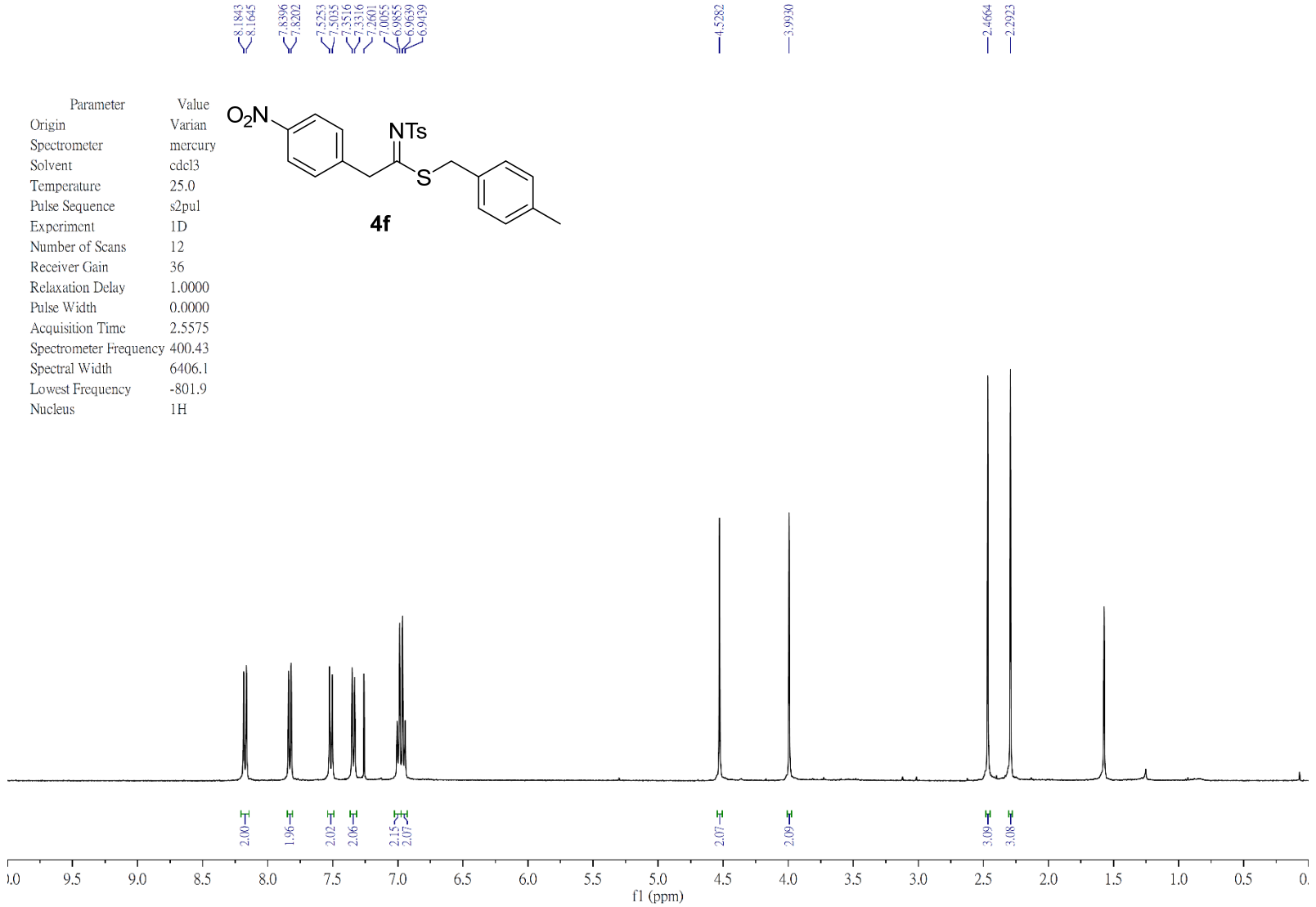
7.8957
7.8497
7.8290
7.4354
7.4150
7.3433
7.3229
7.2594
6.9982
6.9781
6.9621
6.9417
4.4911
3.9724
2.5929
2.4613
2.2861
Parameter Value
Origin Varian
Spectrometer mercury
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 36
Receiver Gain 39
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
Spectrometer Frequency 400.43
Spectral Width 6406.1
Lowest Frequency -799.6
Nucleus 1H
O
NTs
S
4e
2.00
1.89
1.98
1.98
1.83
1.75
1.92
2.00
3.01
3.00
2.97
f1 (ppm)


197.37
186.50
143.58
139.11
138.24
137.25
136.27
131.70
130.19
129.36
129.12
128.82
128.50
126.93
77.32
77.00
76.68
43.83
36.04
26.47
21.47
20.96
O
NTs
S
4e
f1 (ppm)


8.1843
8.1645
7.8396
7.8202
7.5253
7.5035
7.3516
7.3316
7.2601
7.0055
6.9855
6.9639
6.9439
4.5282
3.9930
2.4664
2.2923
Parameter Value
Origin Varian
Spectrometer mercury
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 12
Receiver Gain 36
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
Spectrometer Frequency 400.43
Spectral Width 6406.1
Lowest Frequency -801.9
Nucleus 1H
O2N
NTs
S
4f
2.00
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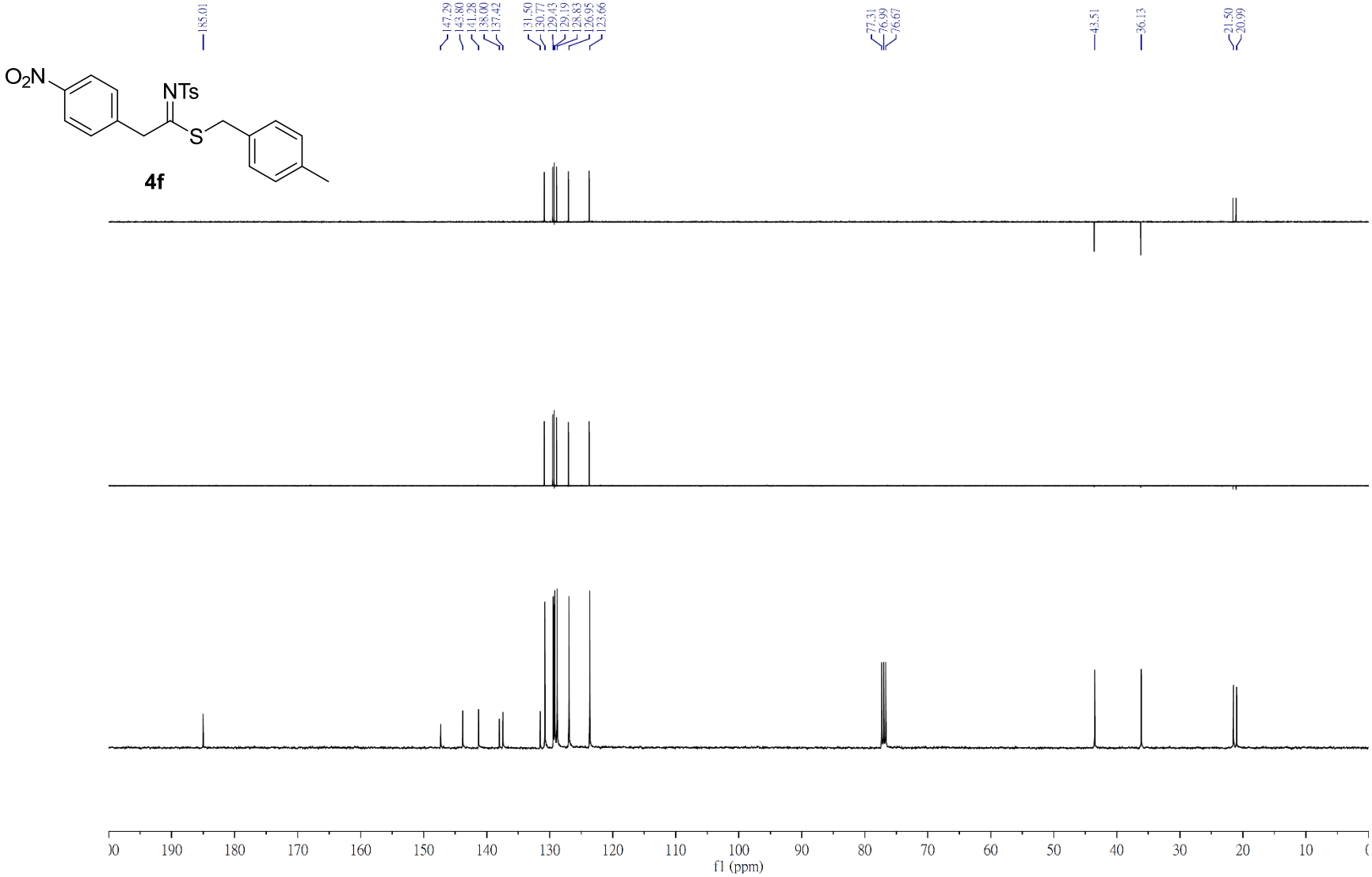
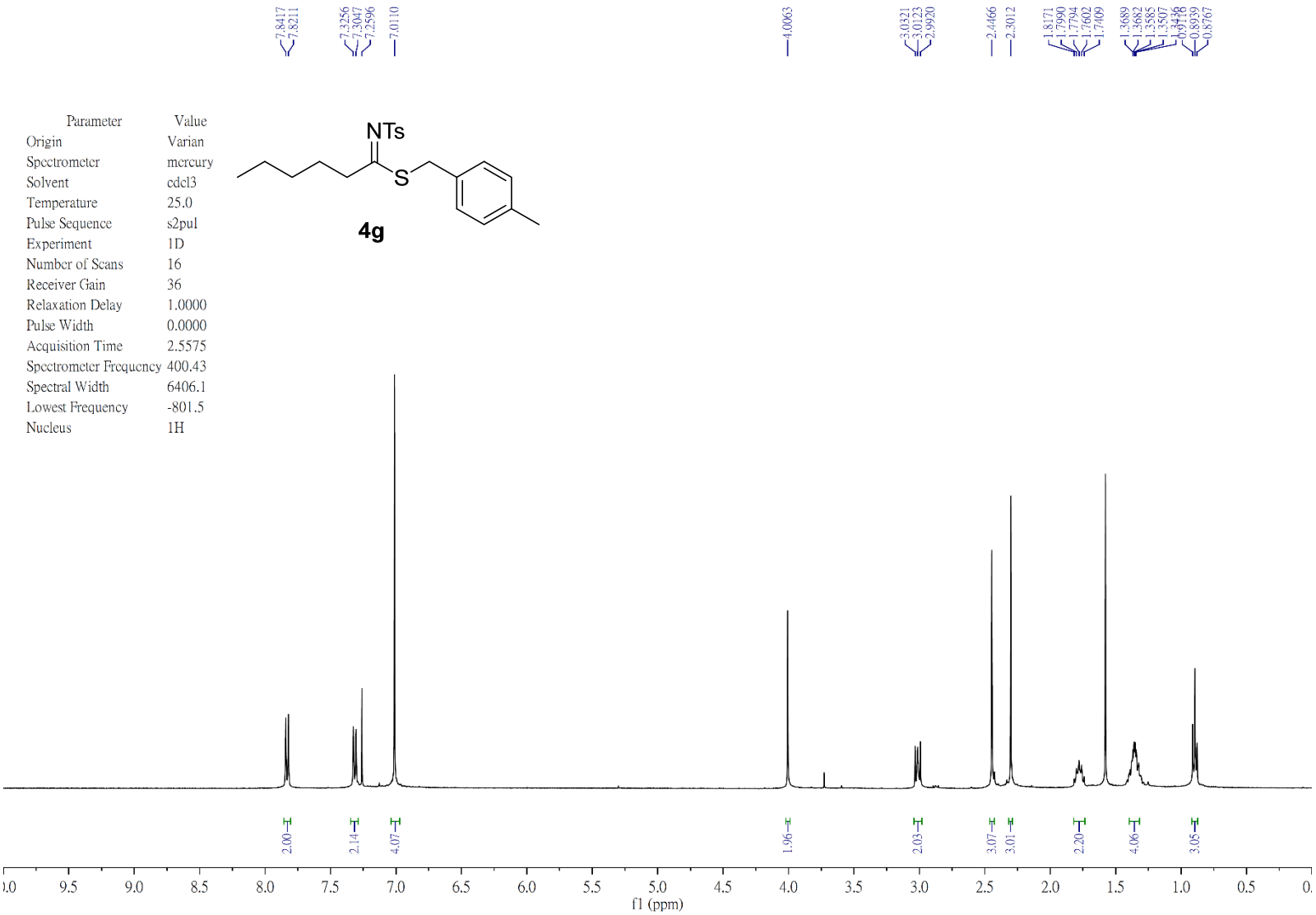
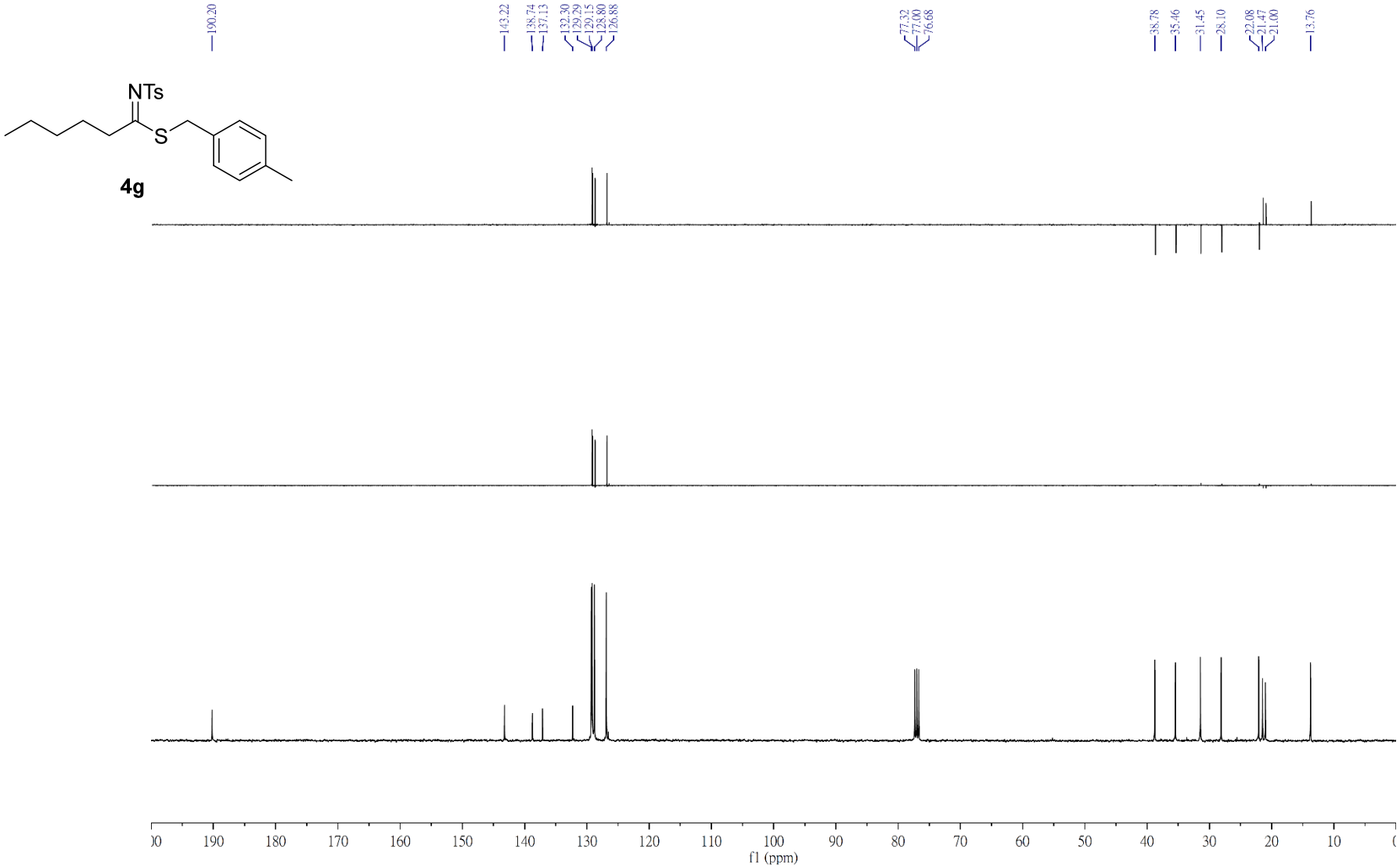
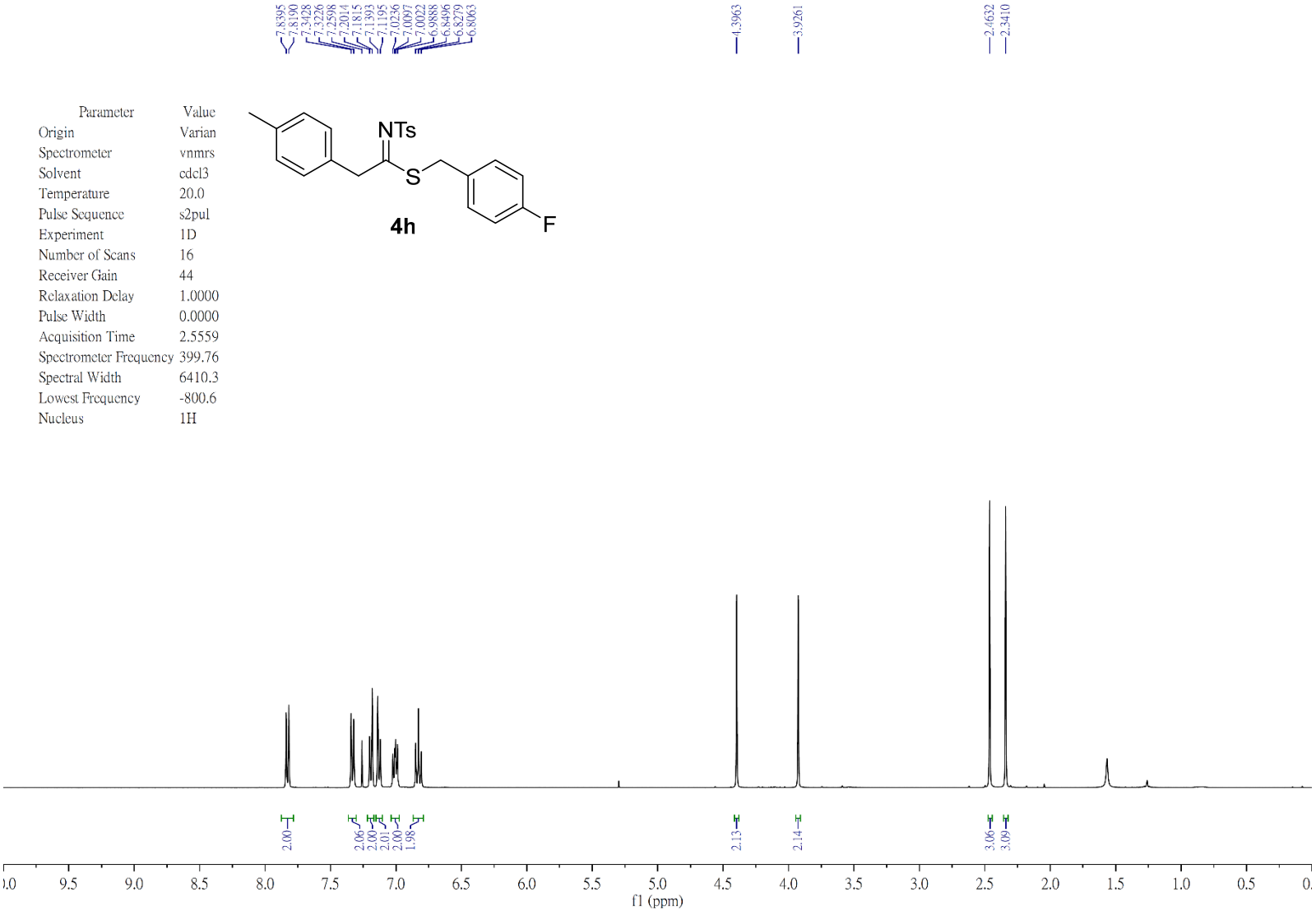
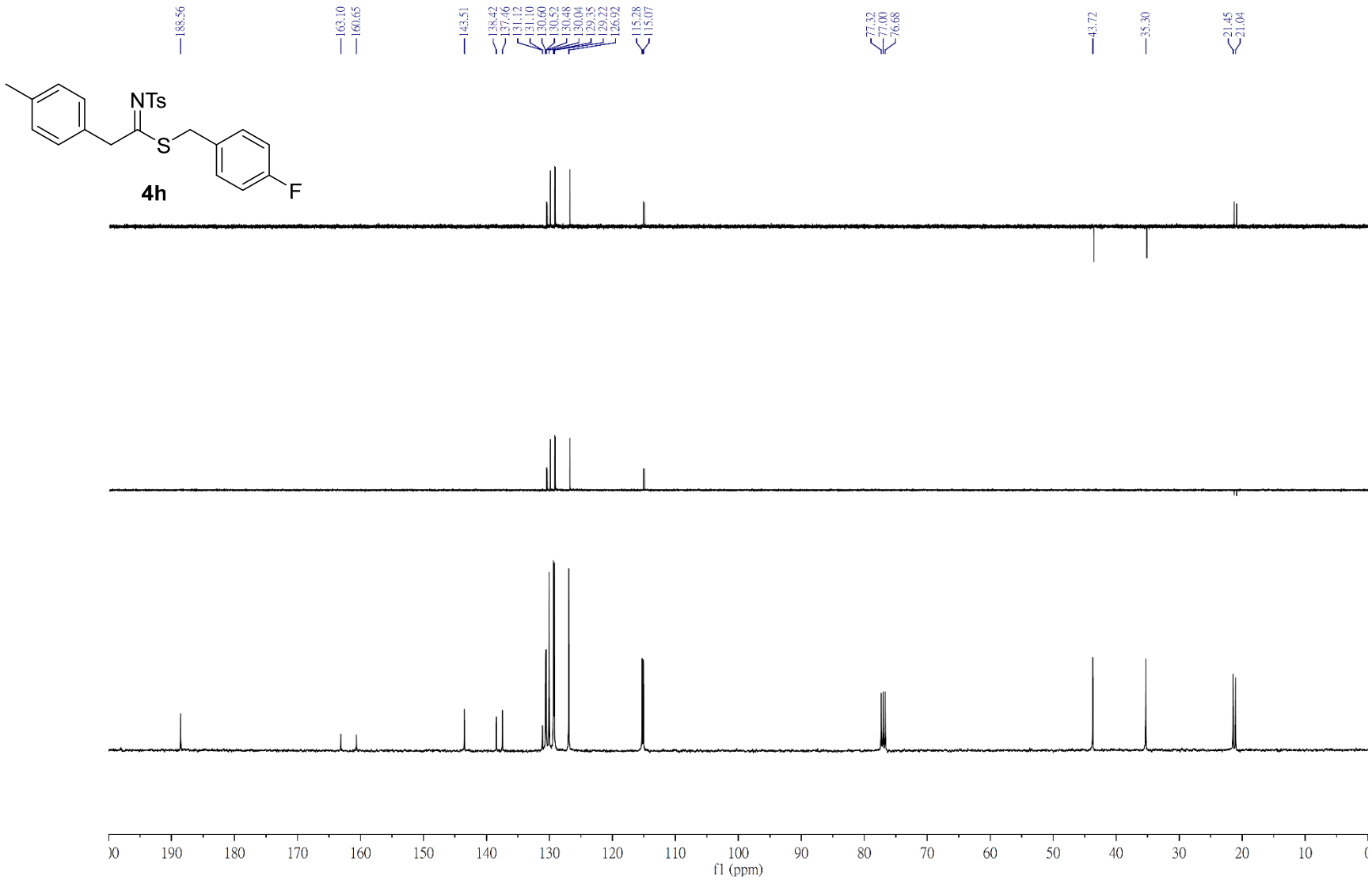
2.02
2.06
2.15
2.07
2.07
2.09
3.09
3.08
f1 (ppm)


185.01
147.29
143.80
141.28
138.00
137.42
131.50
130.77
129.43
129.19
128.83
126.95
123.66
77.31
76.99
76.67
43.51
36.13
21.50
20.99
O2N
NTs
S
4f
f1 (ppm)

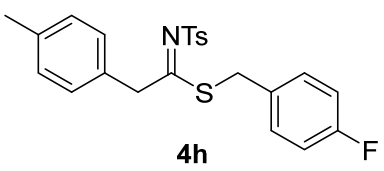

7.8417
7.8211
7.3256
7.3047
7.2596
7.0110
4.0063
3.0321
3.0123
2.9920
2.4466
2.3012
1.8171
1.7990
1.7794
1.7602
1.7409
1.3689
1.3682
1.3585
1.3507
0.9436
0.8939
0.8767
Parameter Value
Origin Varian
Spectrometer mercury
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 16
Receiver Gain 36
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
Spectrometer Frequency 400.43
Spectral Width 6406.1
Lowest Frequency -801.5
Nucleus 1H
NTs
S
4g
2.00
2.14
4.07
1.96
2.03
3.07
3.01
2.20
4.06
3.05
f1 (ppm)


190.20
143.22
138.74
137.13
132.30
129.29
129.15
128.80
126.88
77.32
77.00
76.68
38.78
35.46
31.45
28.10
22.08
21.47
21.00
13.76
NTs
S
4g
f1 (ppm)


7.8395
7.8190
7.3428
7.3226
7.2598
7.2014
7.1815
7.1393
7.1195
7.0236
7.0097
7.0022
6.9888
6.8496
6.8279
6.8063
4.3963
3.9261
2.4632
2.3410
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 16
Receiver Gain 44
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.6
Nucleus 1H
NTs
S
F
4h
2.00
2.06
2.00
2.01
2.00
1.98
2.13
2.14
3.06
3.09
f1 (ppm)

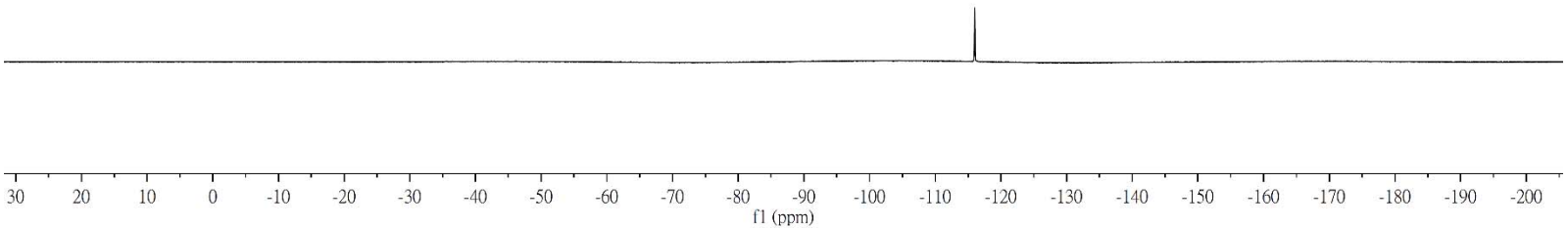

188.56
163.10
160.65
143.51
138.42
137.46
131.12
131.10
130.60
130.52
130.48
130.04
129.35
129.22
126.92
115.28
115.07
77.32
77.00
76.68
43.72
35.30
21.45
21.04
NTs
S
F
4h
f1 (ppm)

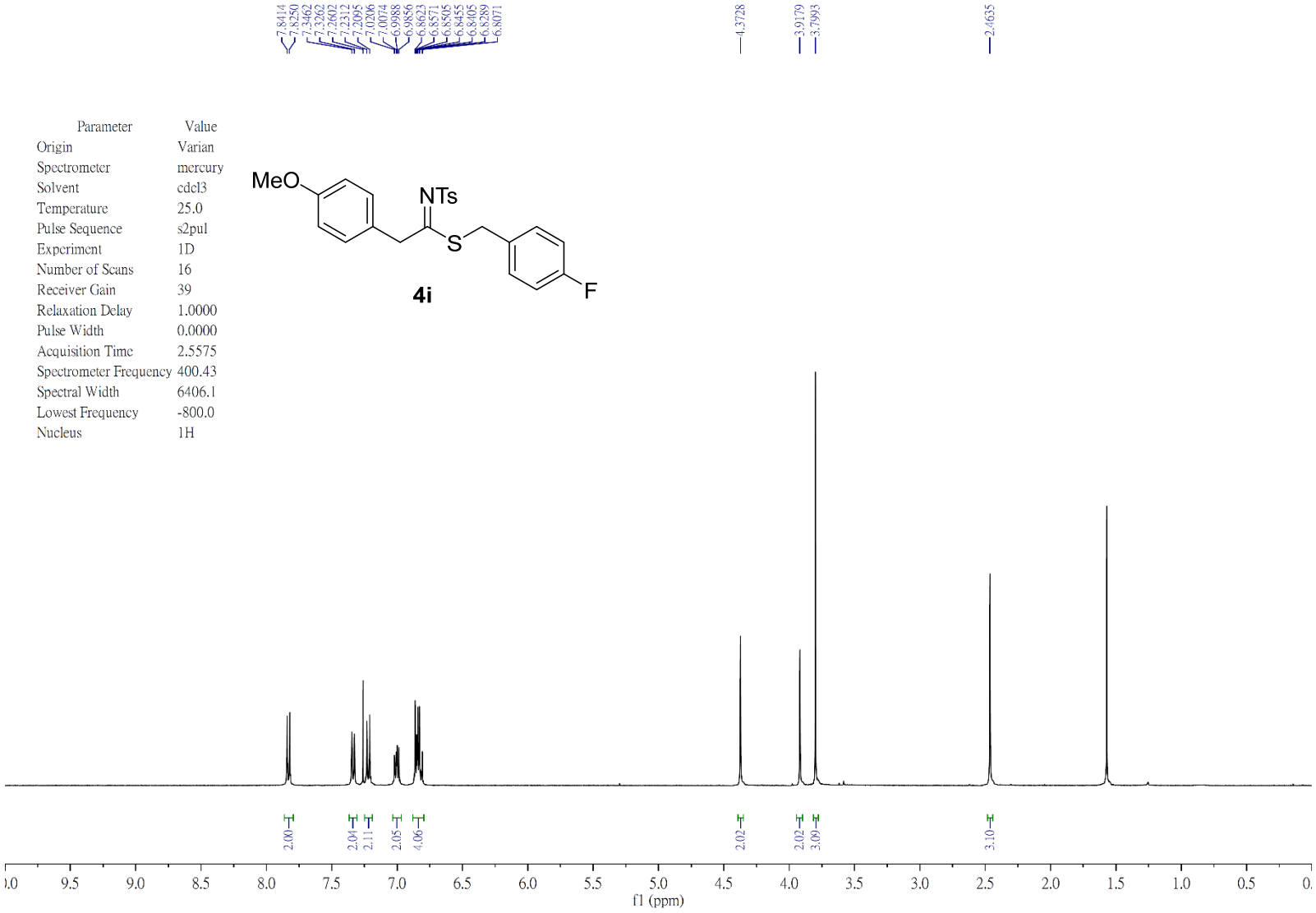
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77332.4
Nucleus	19F

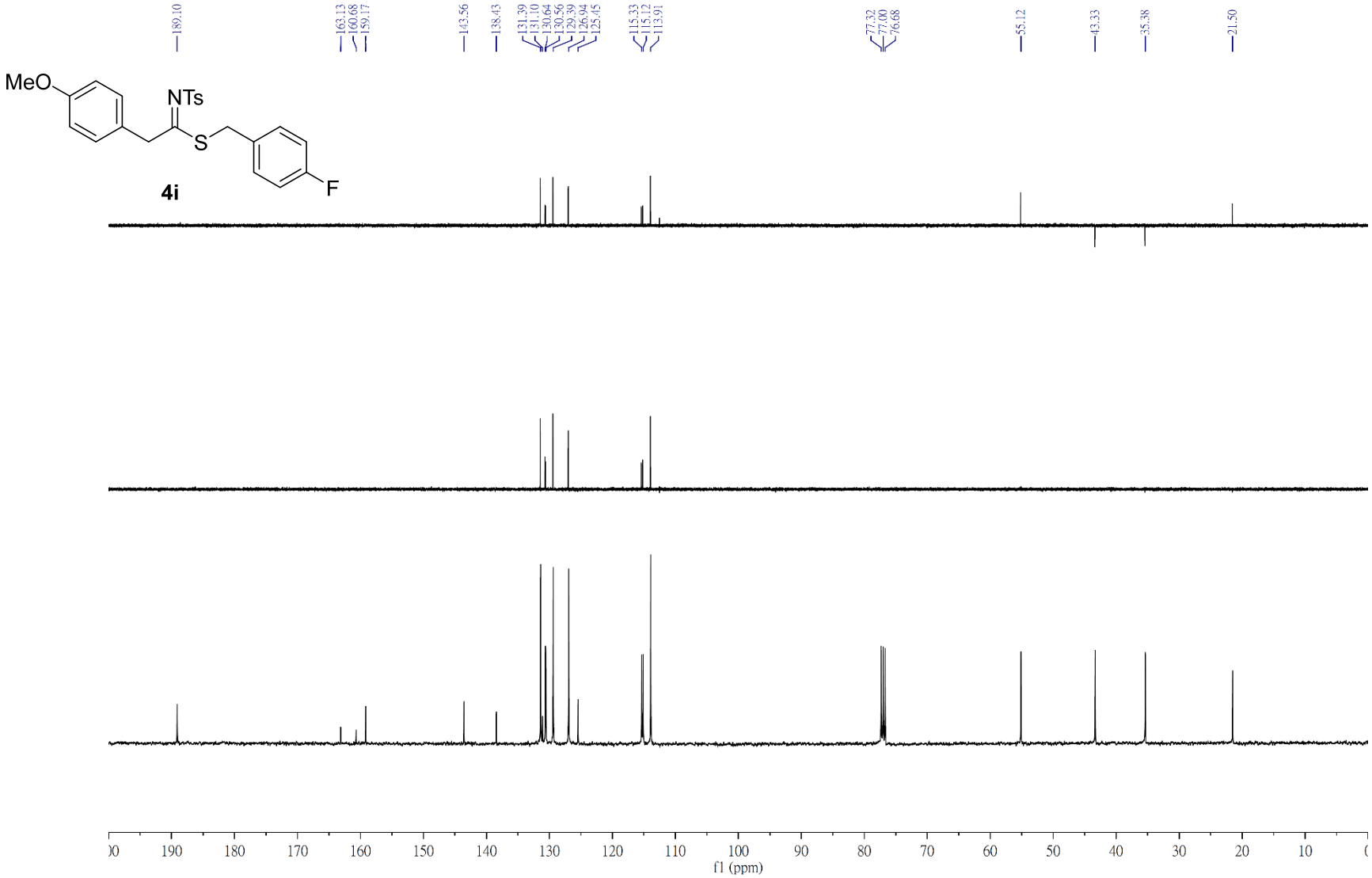


4h

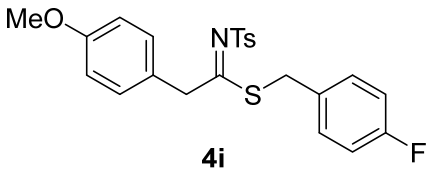
-116.027




7.8414
7.8250
7.3462
7.3262
7.2602
7.2312
7.2095
7.0206
7.0074
6.9988
6.9856
6.8623
6.8571
6.8505
6.8455
6.8405
6.8289
6.8071
4.3728
3.9179
3.7993
2.4635
Parameter Value
Origin Varian
Spectrometer mercury
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 16
Receiver Gain 39
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
Spectrometer Frequency 400.43
Spectral Width 6406.1
Lowest Frequency -800.0
Nucleus 1H
MeO
NTs
S
F
4i
2.00
2.04
2.11
2.05
4.06
2.02
2.02
3.09
3.10
f1 (ppm)

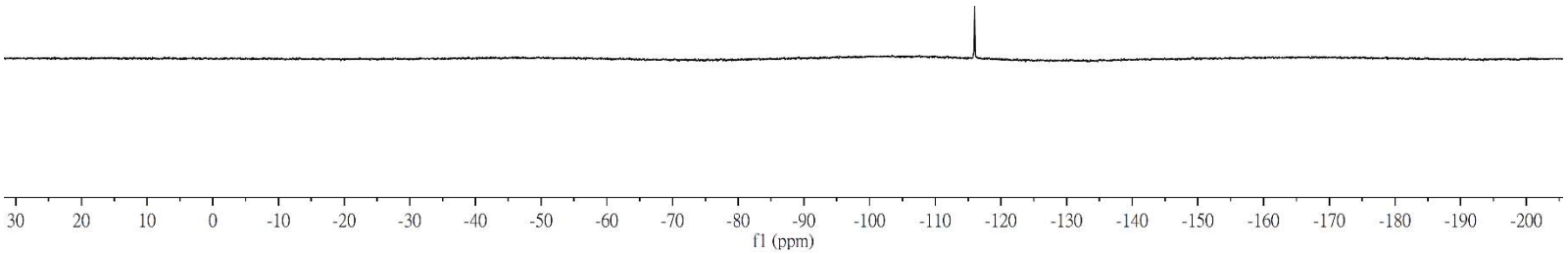

189.10
163.13
160.68
159.17
143.56
138.43
131.39
131.10
130.64
130.56
129.39
126.94
125.45
115.33
115.12
113.91
77.32
77.00
76.68
55.12
43.33
35.38
21.50
MeO
NTs
S
F
4i
f1 (ppm)

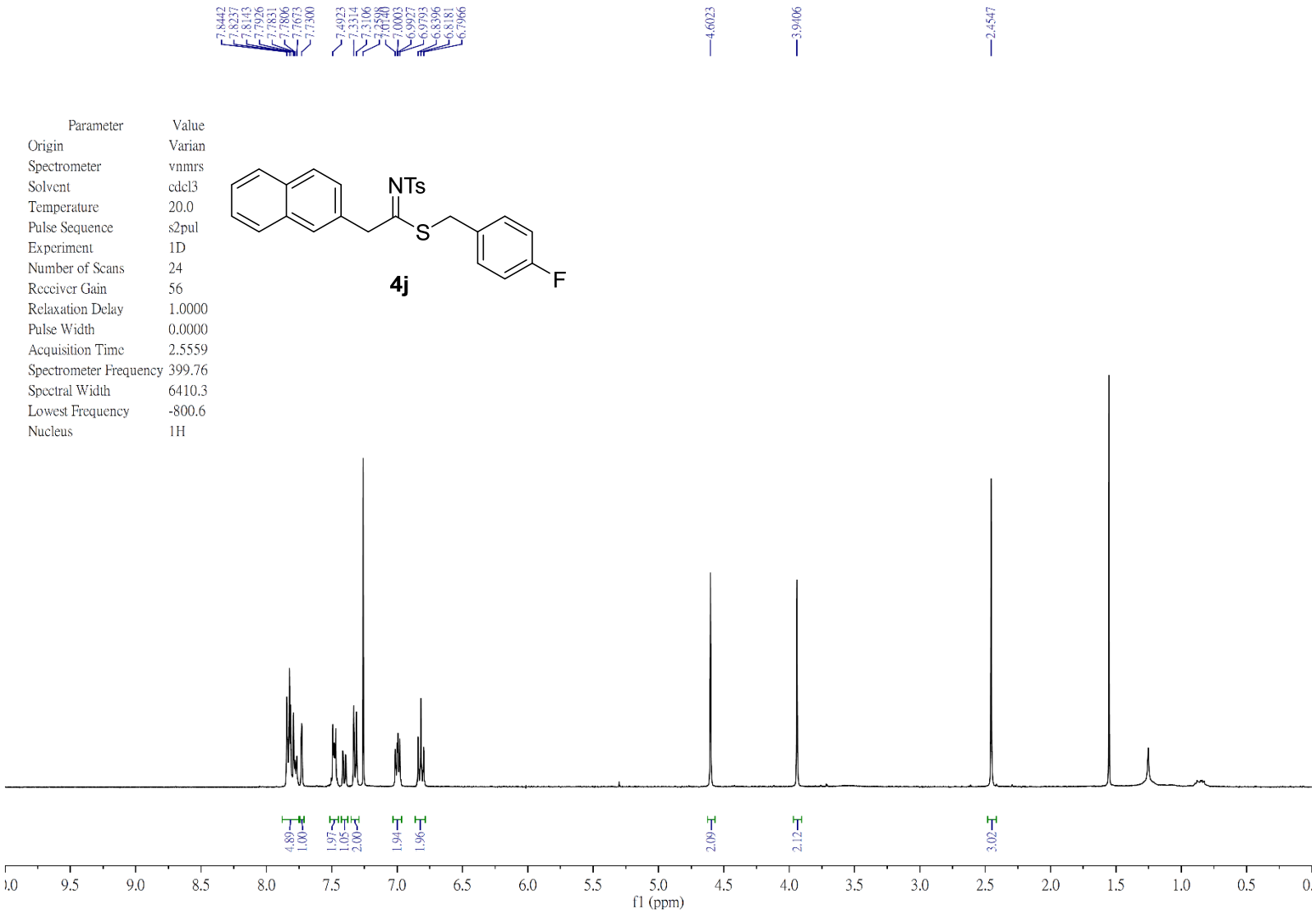
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77332.4
Nucleus	19F

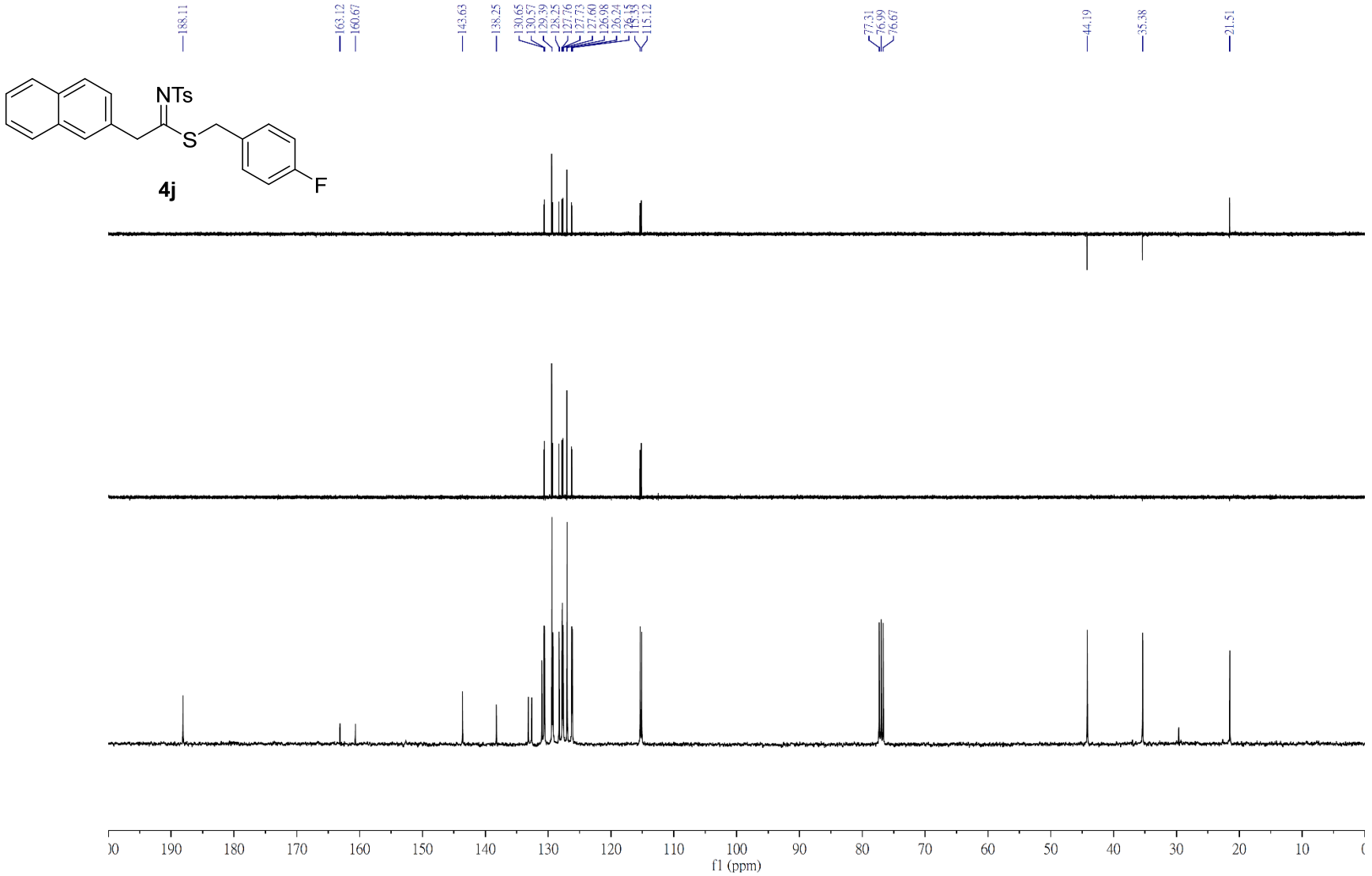


4i

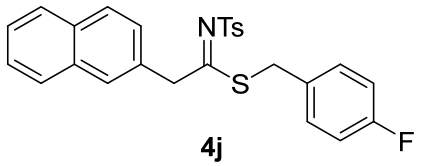
-116.015




7.8442
7.8237
7.8143
7.7926
7.7831
7.7806
7.7673
7.7300
7.4923
7.3314
7.3106
7.2598
7.0140
7.0003
6.9927
6.9793
6.8396
6.8181
6.7966
4.6023
3.9406
2.4547
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 24
Receiver Gain 56
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.6
Nucleus 1H
NTs
S
F
4j
4.89
1.00
1.97
1.05
2.00
1.94
1.96
2.09
2.12
3.02
f1 (ppm)

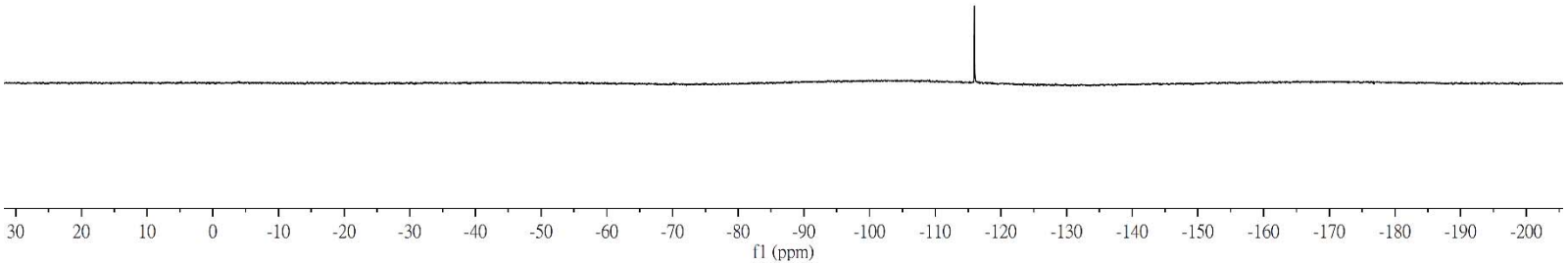

188.11
163.12
160.67
143.63
138.25
130.65
130.57
129.39
128.25
127.76
127.73
127.60
126.98
126.24
115.33
115.12
77.31
76.99
76.67
44.19
35.38
21.51
NTs
S
F
4j
f1 (ppm)

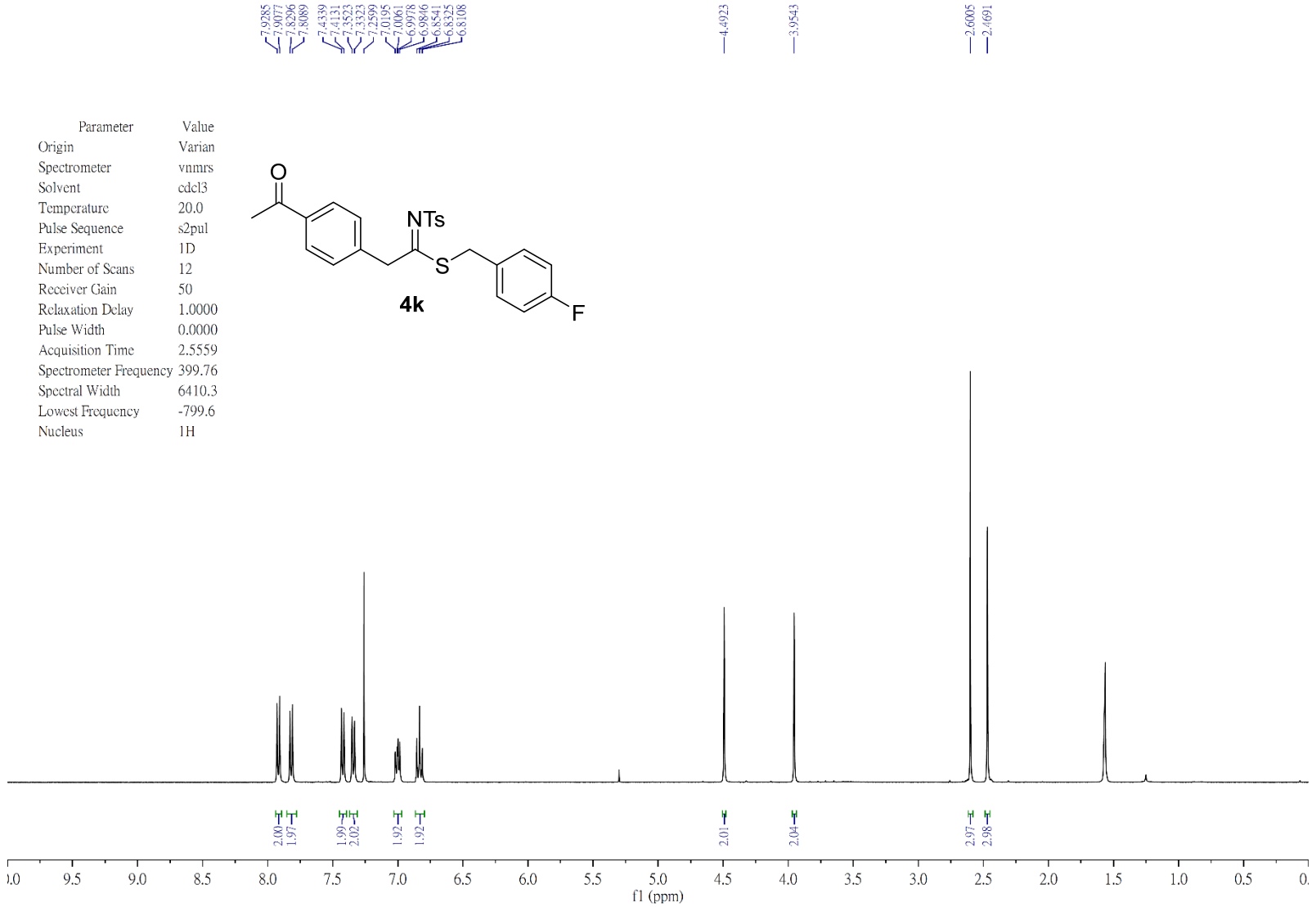
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77332.4
Nucleus	19F



4j

-115.976

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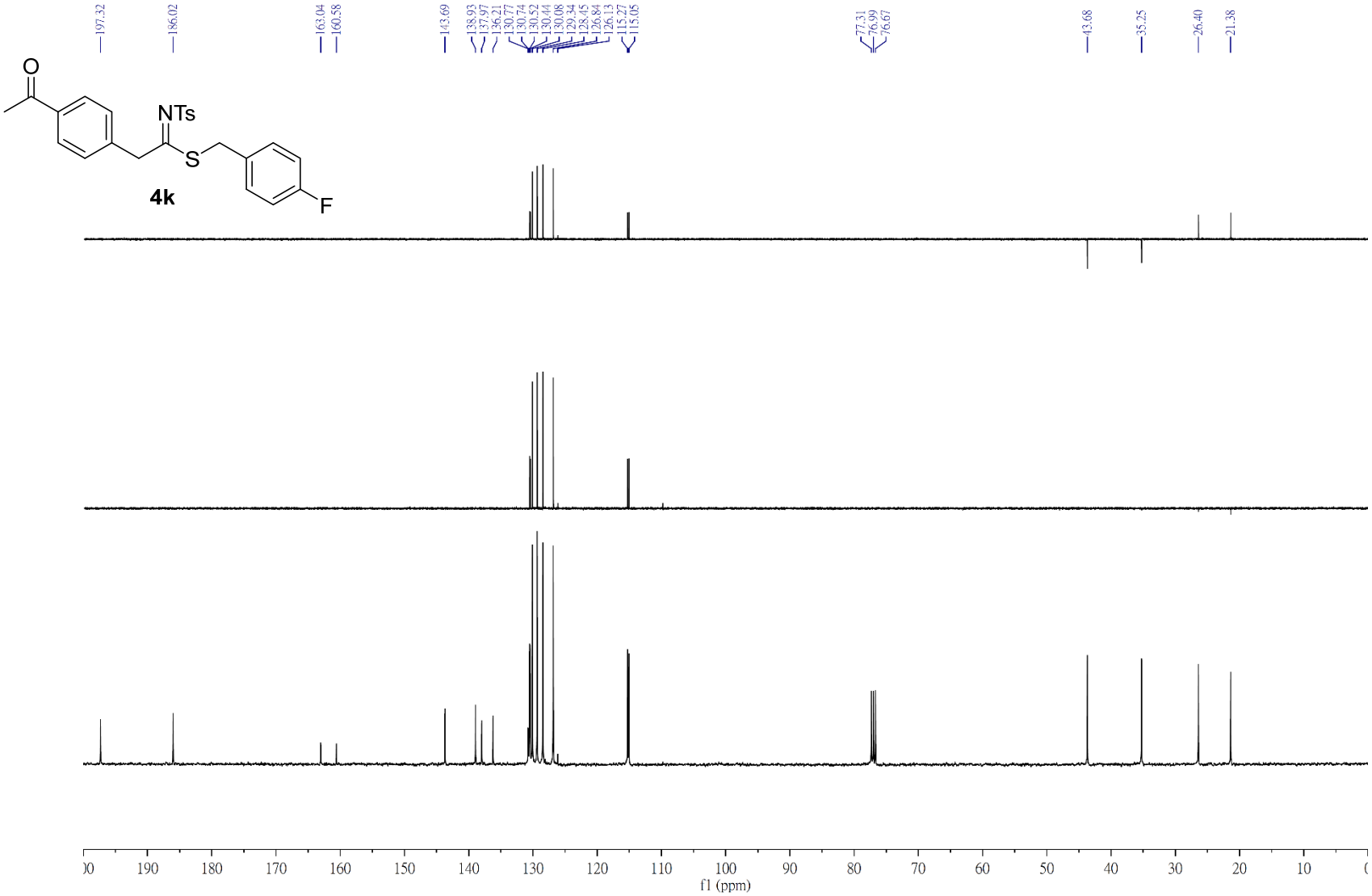
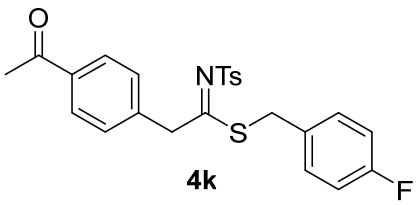
7.9077
7.8296
7.8089
7.4339
7.4131
7.3523
7.3323
7.2599
7.0195
7.0061
6.9978
6.9846
6.8541
6.8325
6.8108
4.4923
3.9543
2.6005
2.4691
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 12
Receiver Gain 50
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
O
NTs
S
F
4k
2.00
1.97
1.99
2.02
1.92
1.92
2.01
2.04
2.97
2.98
f1 (ppm)

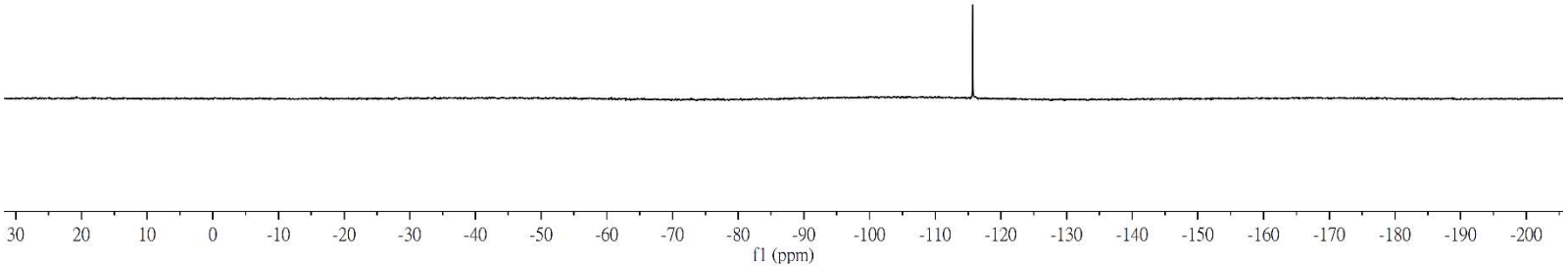

197.32
186.02
163.04
160.58
143.69
138.93
137.97
136.21
130.77
130.74
130.52
130.44
130.08
129.34
128.45
126.84
126.13
115.27
115.05
77.31
76.99
76.67
43.68
35.25
26.40
21.38
O
NTs
S
F
4k
f1 (ppm)

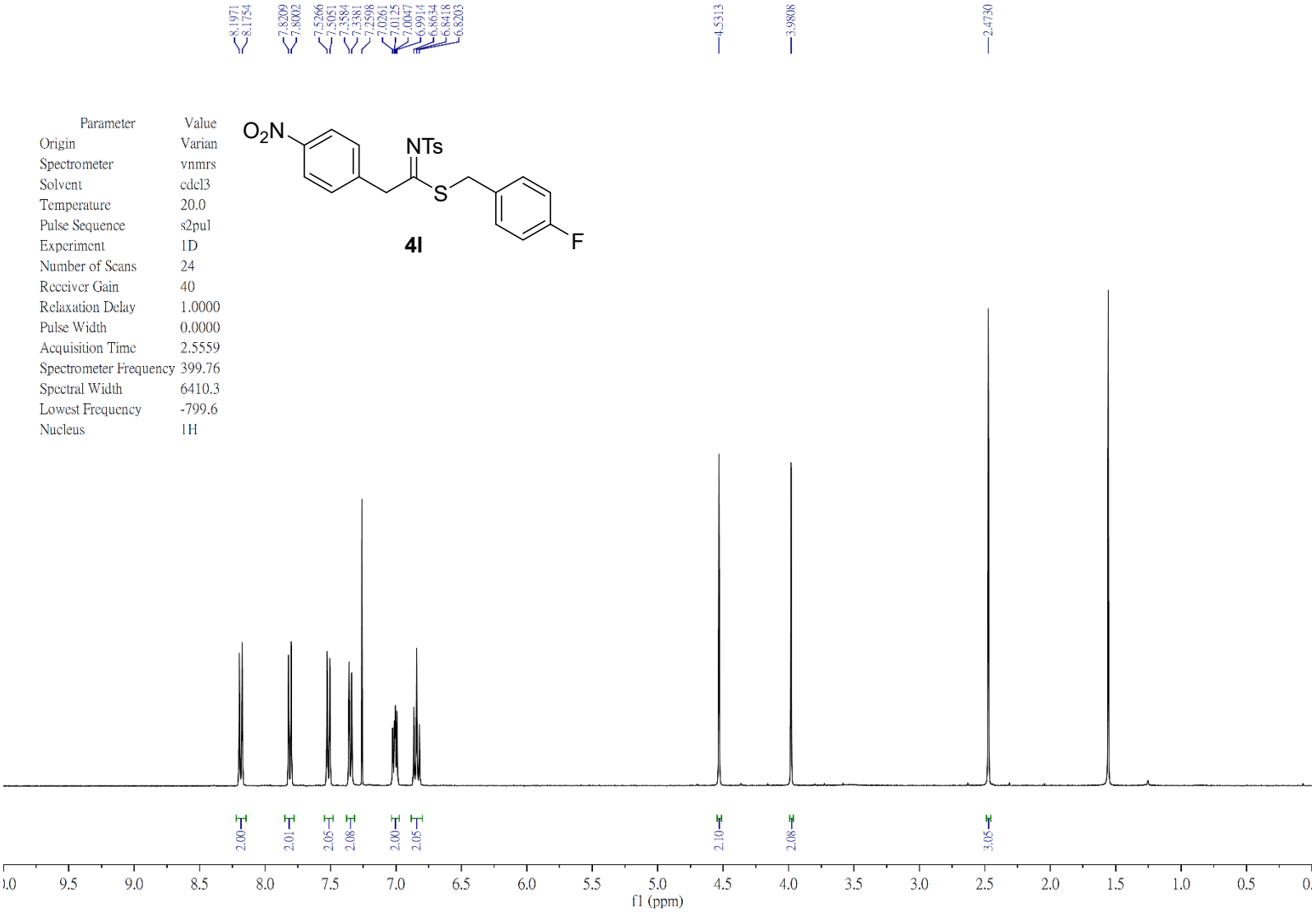
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77332.4
Nucleus	19F



4k

-115.705

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8.1754
7.8209
7.8002
7.5266
7.5051
7.3584
7.3381
7.2598
7.0261
7.0125
7.0047
6.9914
6.8634
6.8418
6.8203
4.5313
3.9808
2.4730
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 24
Receiver Gain 40
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
O2N
NTs
S
F
4l
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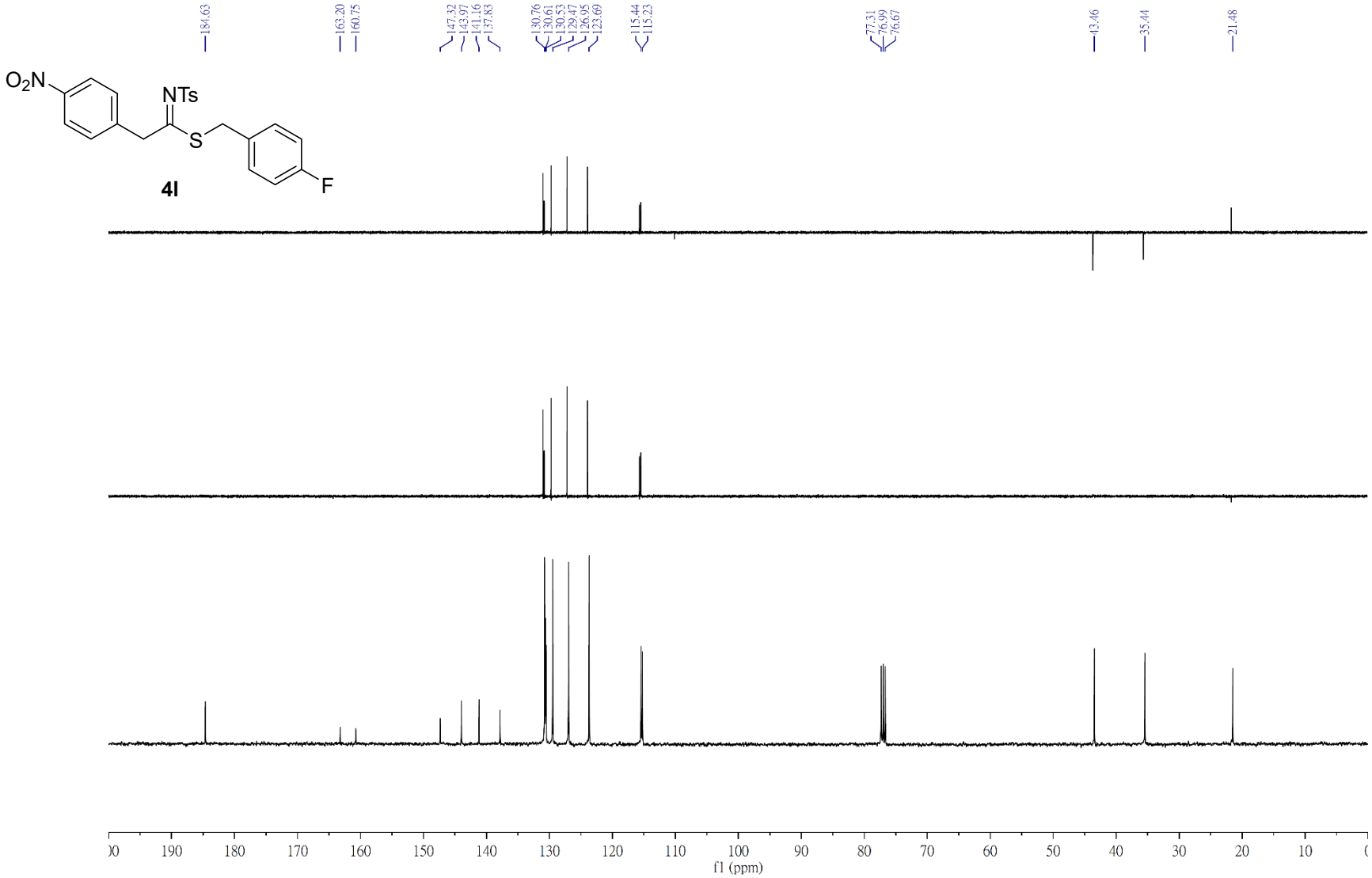
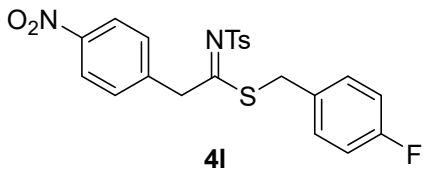
2.05
2.08
2.00
2.05
2.10
2.08
3.05
f1 (ppm)

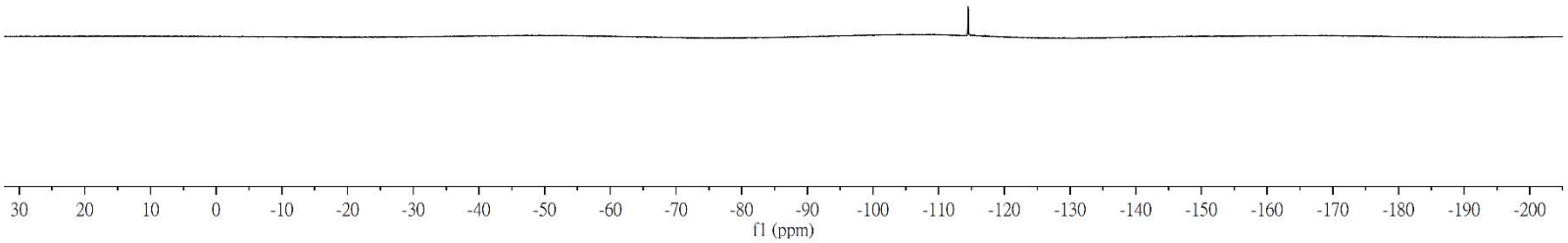

184.63
163.20
160.75
147.32
143.97
141.16
137.83
130.76
130.61
130.53
129.47
126.95
123.69
115.44
115.23
77.31
76.99
76.67
43.46
35.44
21.48
O2N
NTs
S
F
4l
f1 (ppm)

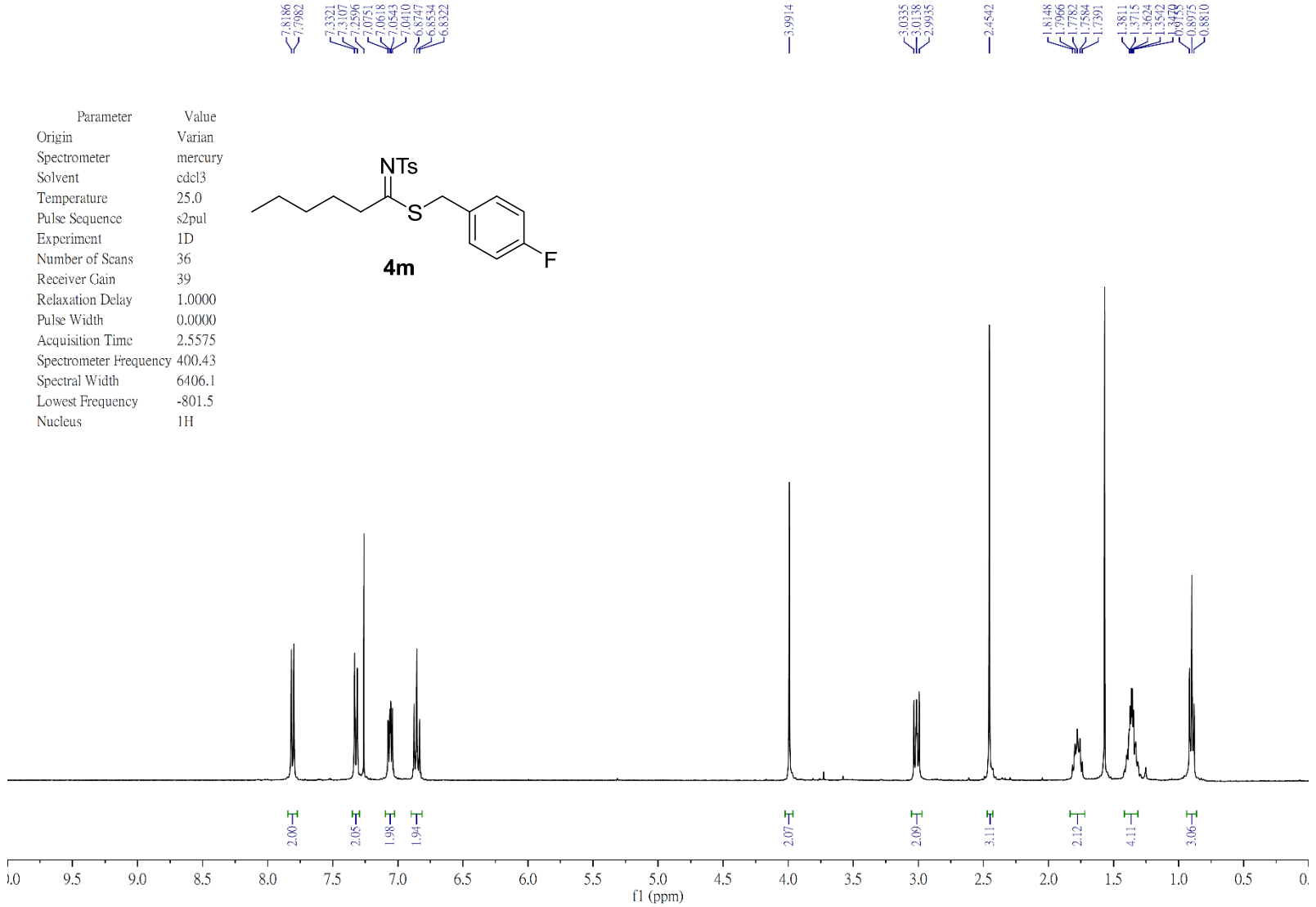
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	c6d6
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	196
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77133.5
Nucleus	19F



4l

-114.485

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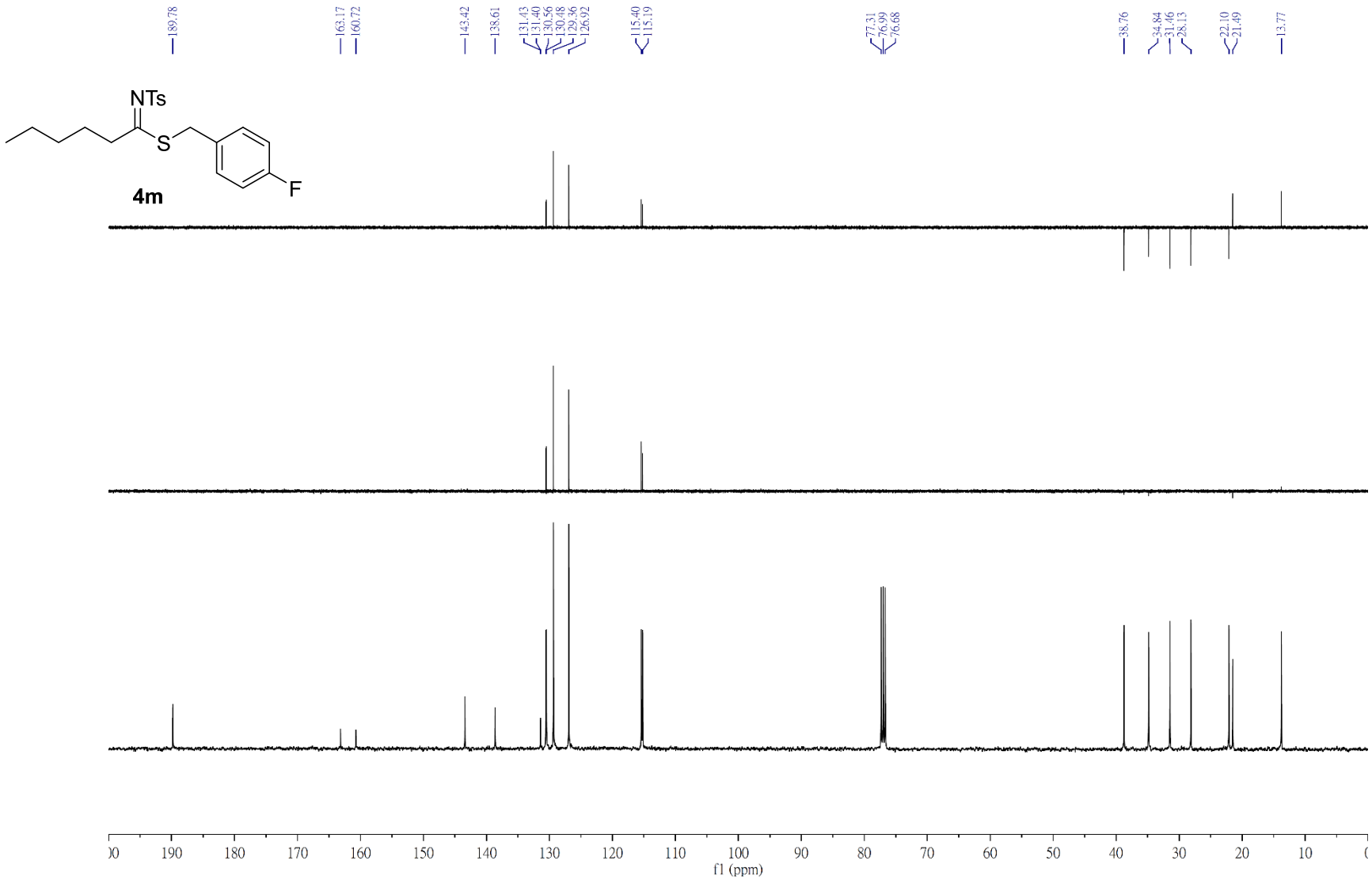
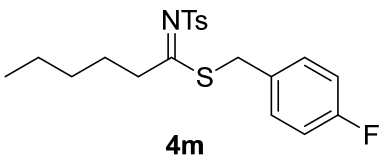
7.7982
7.3321
7.3107
7.2596
7.0751
7.0618
7.0543
7.0410
6.8747
6.8534
6.8322
3.9914
3.0335
3.0138
2.9935
2.4542
1.8148
1.7966
1.7782
1.7584
1.7391
1.3811
1.3715
1.3624
1.3542
0.9758
0.8975
0.8810
Parameter Value
Origin Varian
Spectrometer mercury
Solvent cdcl3
Temperature 25.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 36
Receiver Gain 39
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5575
Spectrometer Frequency 400.43
Spectral Width 6406.1
Lowest Frequency -801.5
Nucleus 1H
NTs
S
F
4m
2.00
2.05
1.98
1.94
2.07
2.09
3.11
2.12
4.11
3.06
f1 (ppm)

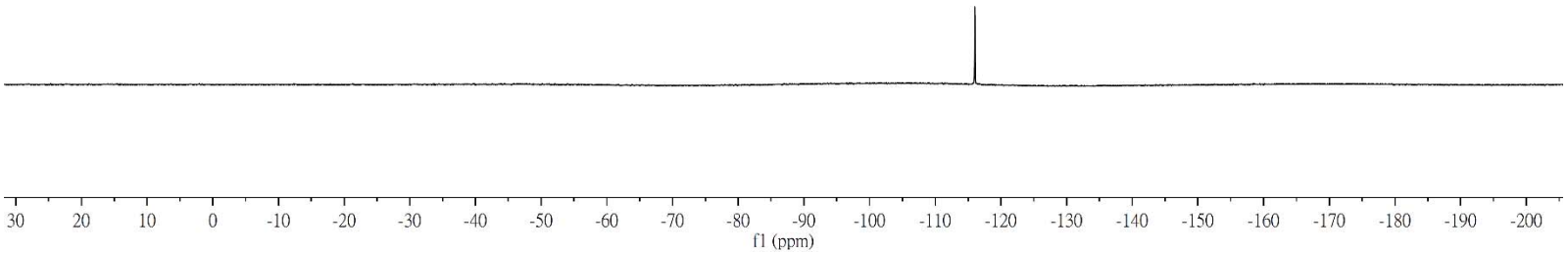

189.78
163.17
160.72
143.42
138.61
131.43
131.40
130.56
130.48
129.36
126.92
115.40
115.19
77.31
76.99
76.68
38.76
34.84
31.46
28.13
22.10
21.49
13.77
NTs
S
F
4m
f1 (ppm)

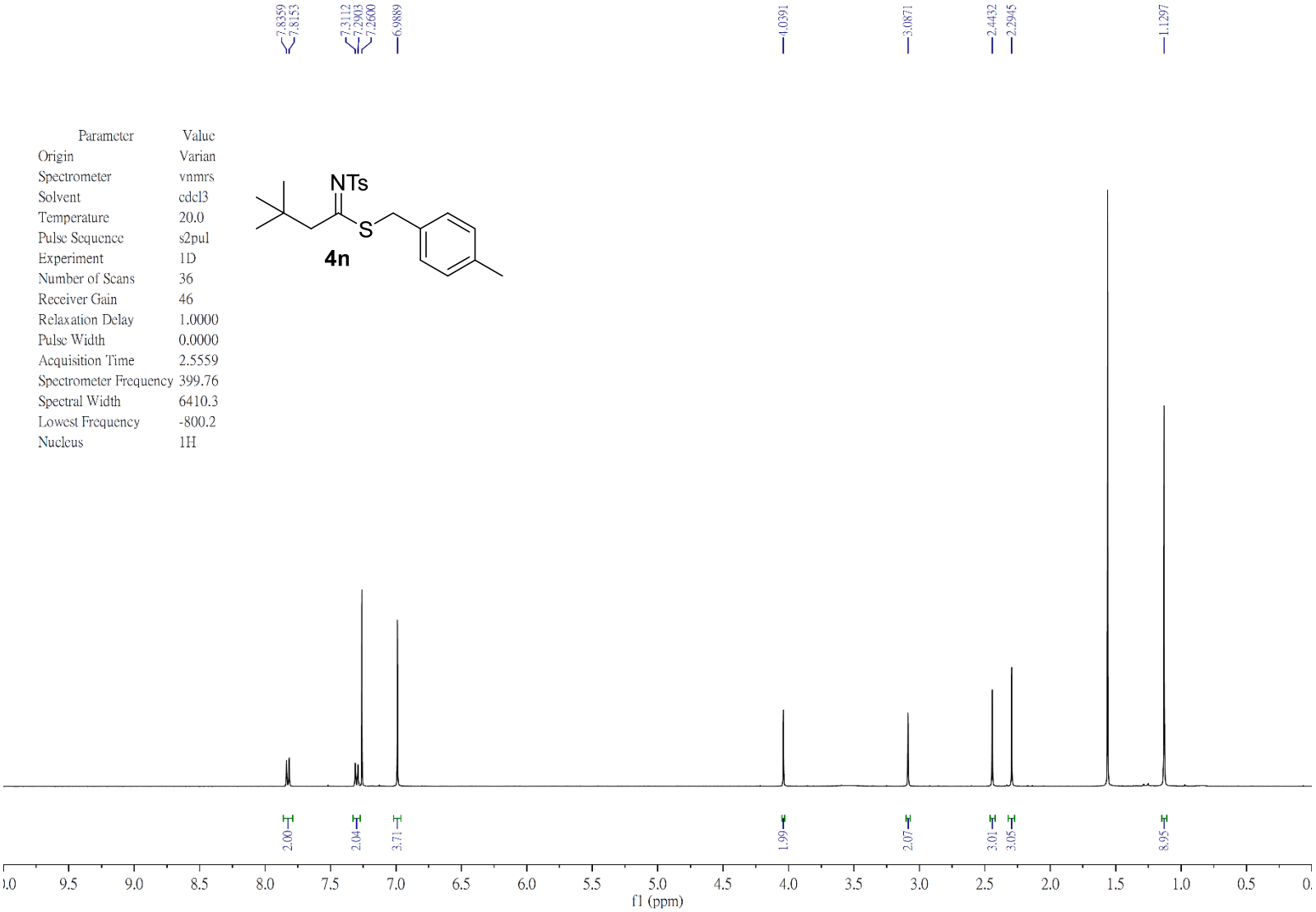
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	0.7340
Spectrometer Frequency	376.11
Spectral Width	89285.7
Lowest Frequency	-77332.4
Nucleus	19F

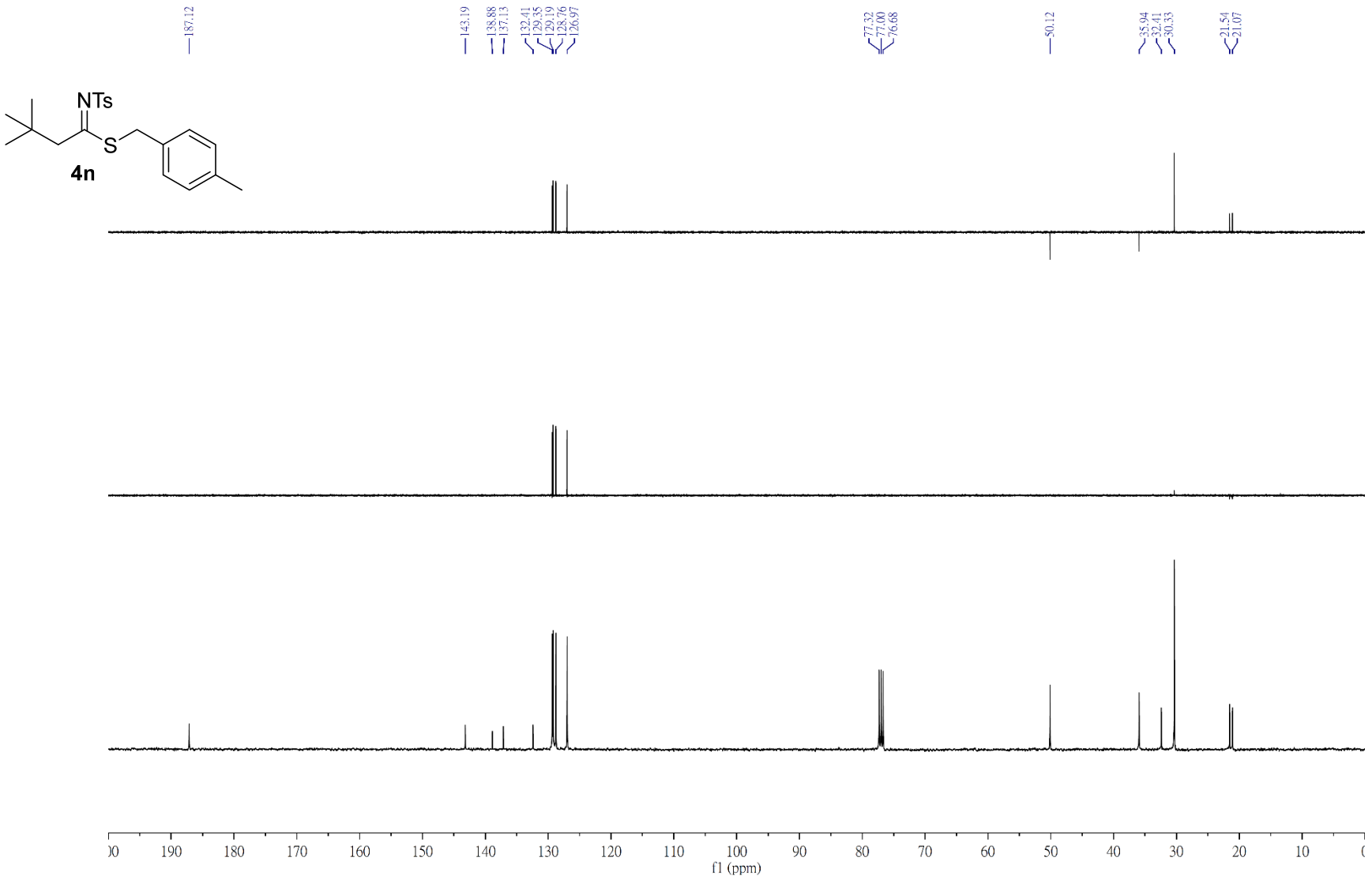


4m

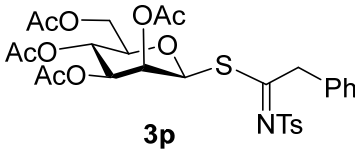
-116.046



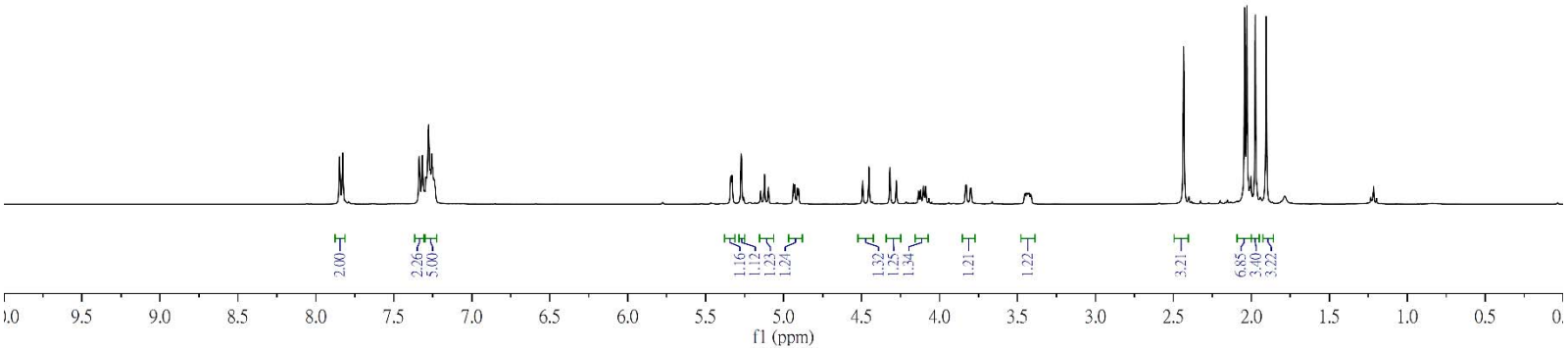

7.8359
7.8153
7.3112
7.2903
7.2600
6.9889
4.0391
3.0871
2.4432
2.2945
1.1297
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 36
Receiver Gain 46
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.2
Nucleus 1H
NTs
S
4n
2.00
2.04
3.71
1.99
2.07
3.01
3.05
8.95
f1 (ppm)

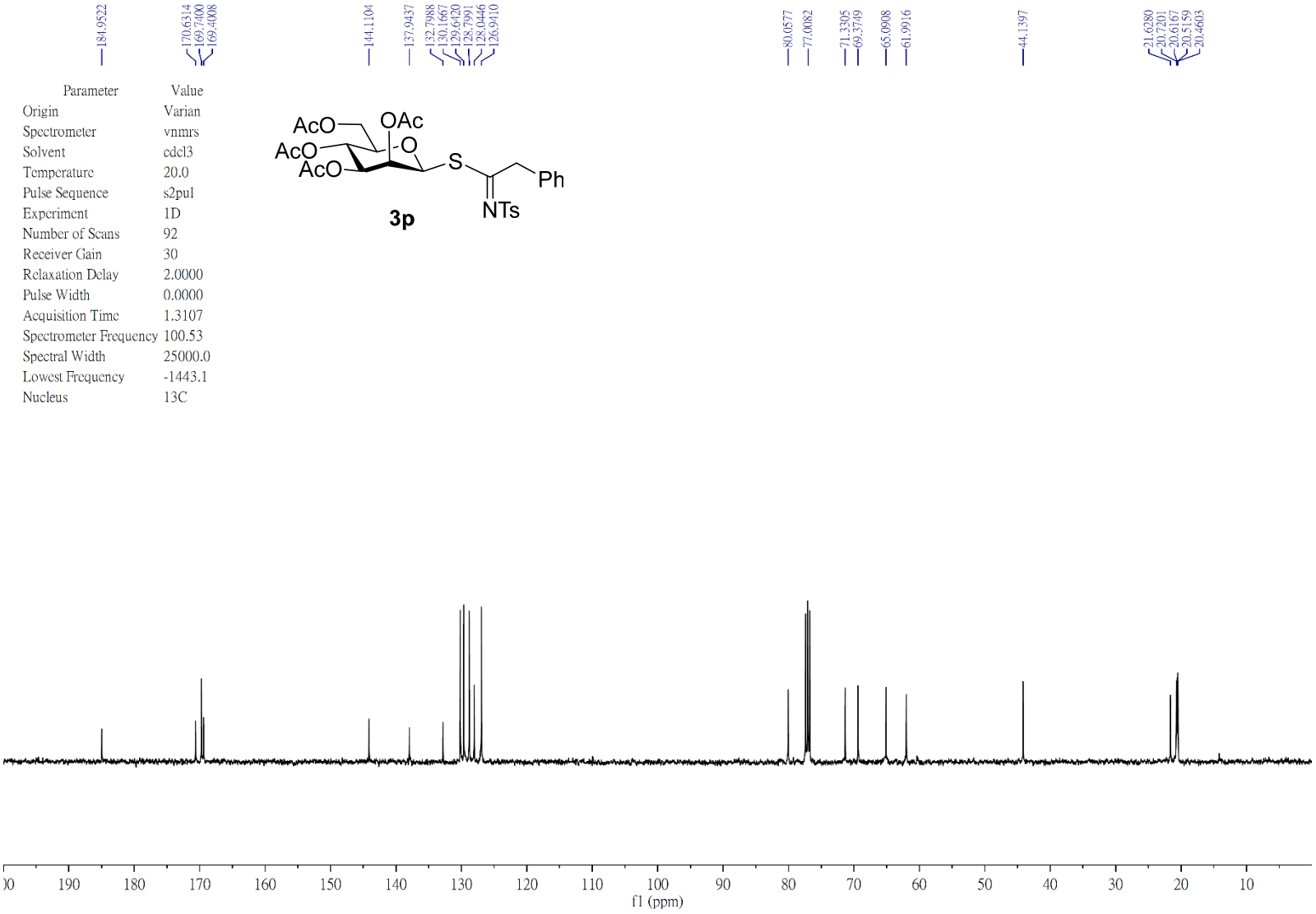

187.12
143.19
138.88
137.13
132.41
129.35
129.19
128.76
126.97
77.32
77.00
76.68
50.12
35.94
32.41
30.33
21.54
21.07
NTs
S
4n
f1 (ppm)

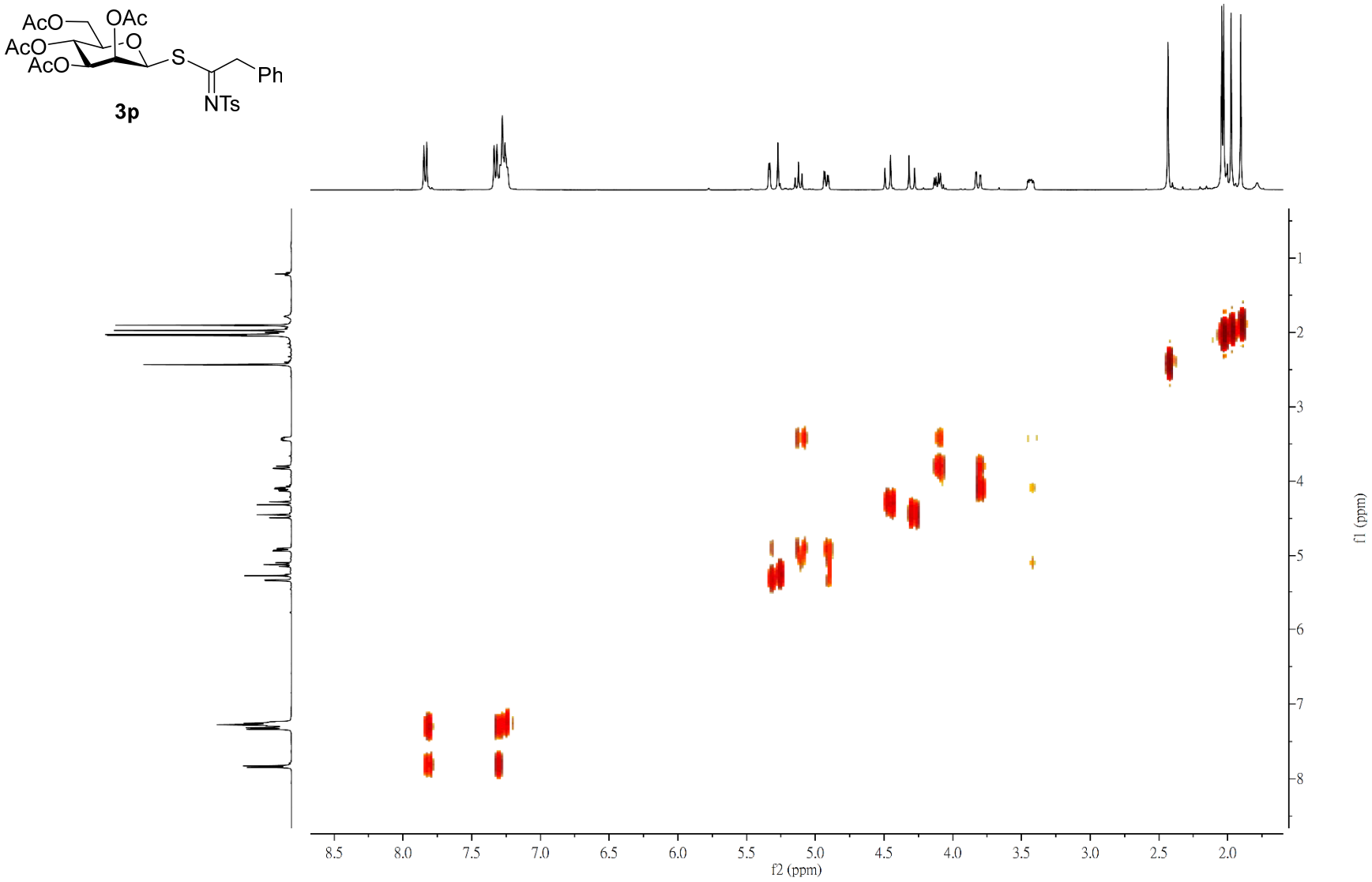
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	20
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-806.6
Nucleus	1H



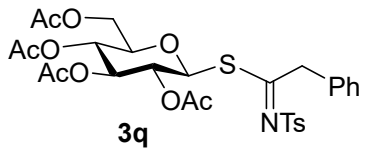
3p



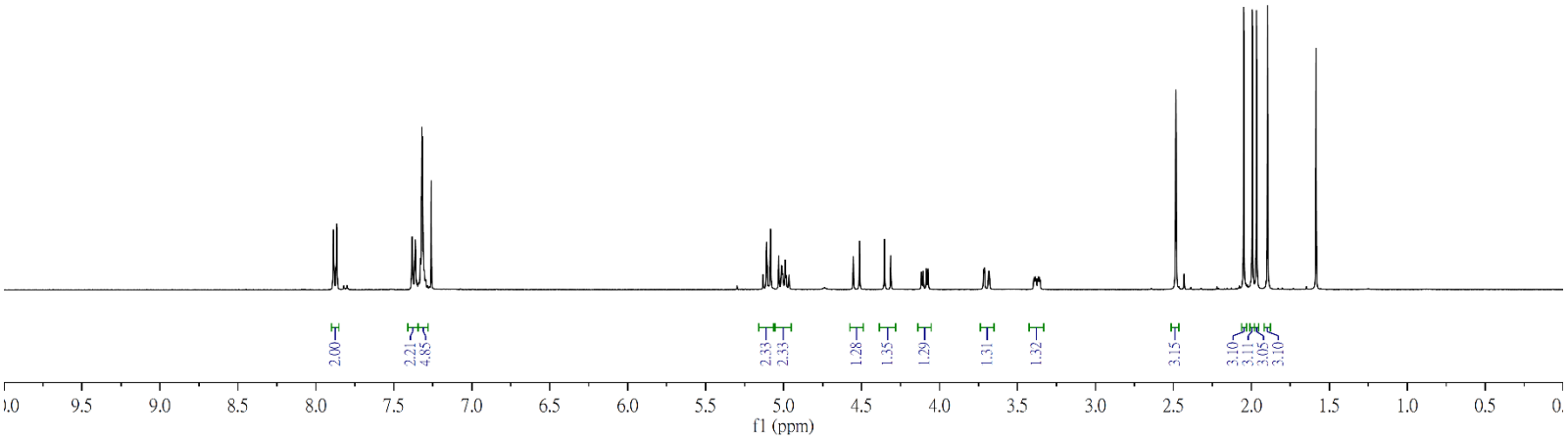

184.9522
170.6314
169.7400
169.4008
144.1104
137.9437
132.7988
130.1667
129.6420
128.7991
128.0446
126.9410
80.0577
77.0082
71.3305
69.3749
65.0908
61.9916
44.1397
21.6280
20.7201
20.6167
20.5159
20.4603
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 92
Receiver Gain 30
Relaxation Delay 2.0000
Pulse Width 0.0000
Acquisition Time 1.3107
Spectrometer Frequency 100.53
Spectral Width 25000.0
Lowest Frequency -1443.1
Nucleus 13C
AcO
OAc
AcO
O
AcO
S
Ph
NTs
3p
f1 (ppm)

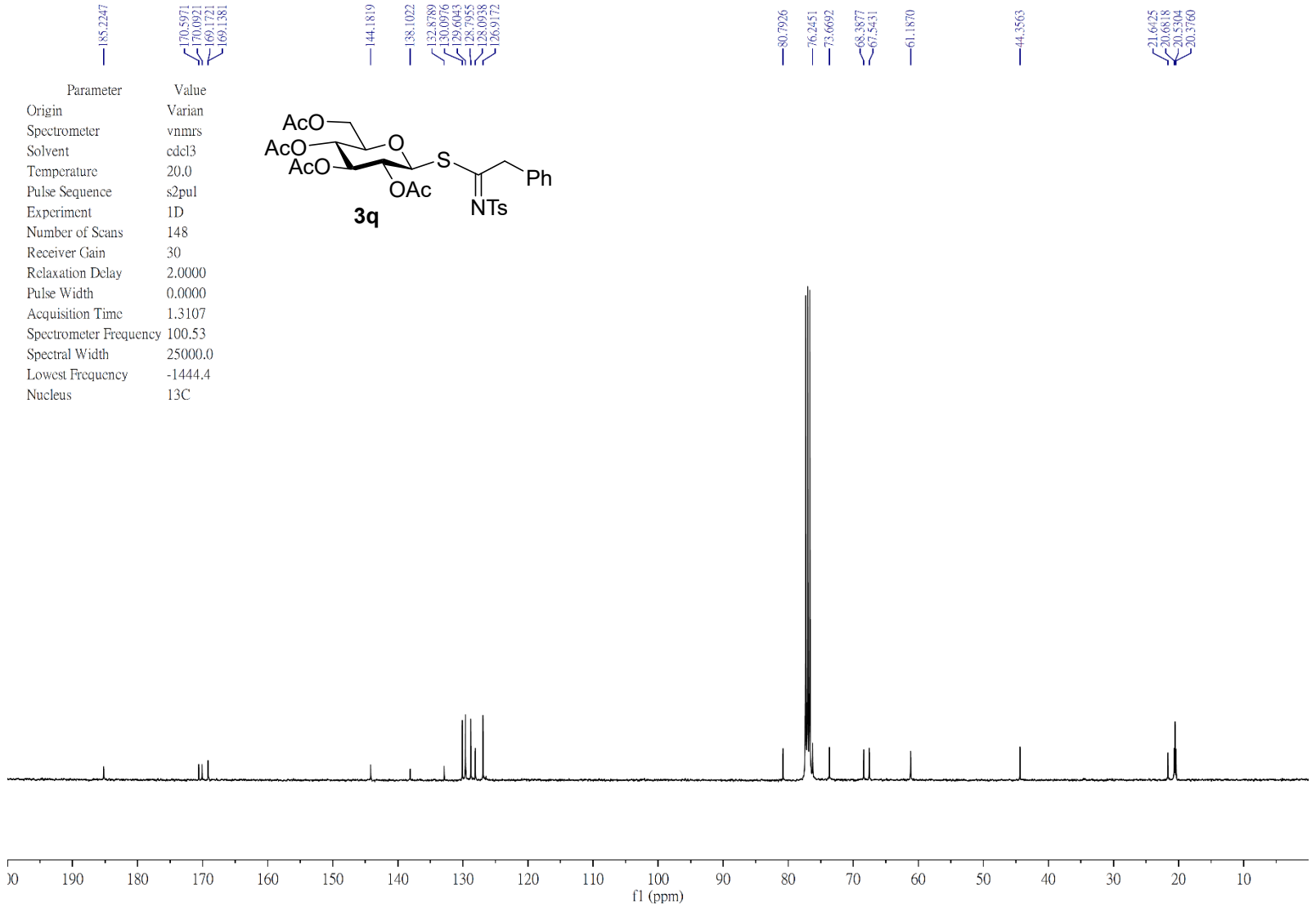

AcO
OAc
AcO
O
AcO
S
Ph
NTs
3p
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f2 (ppm)
1
2
3
4
5
6
7
8
f1 (ppm)

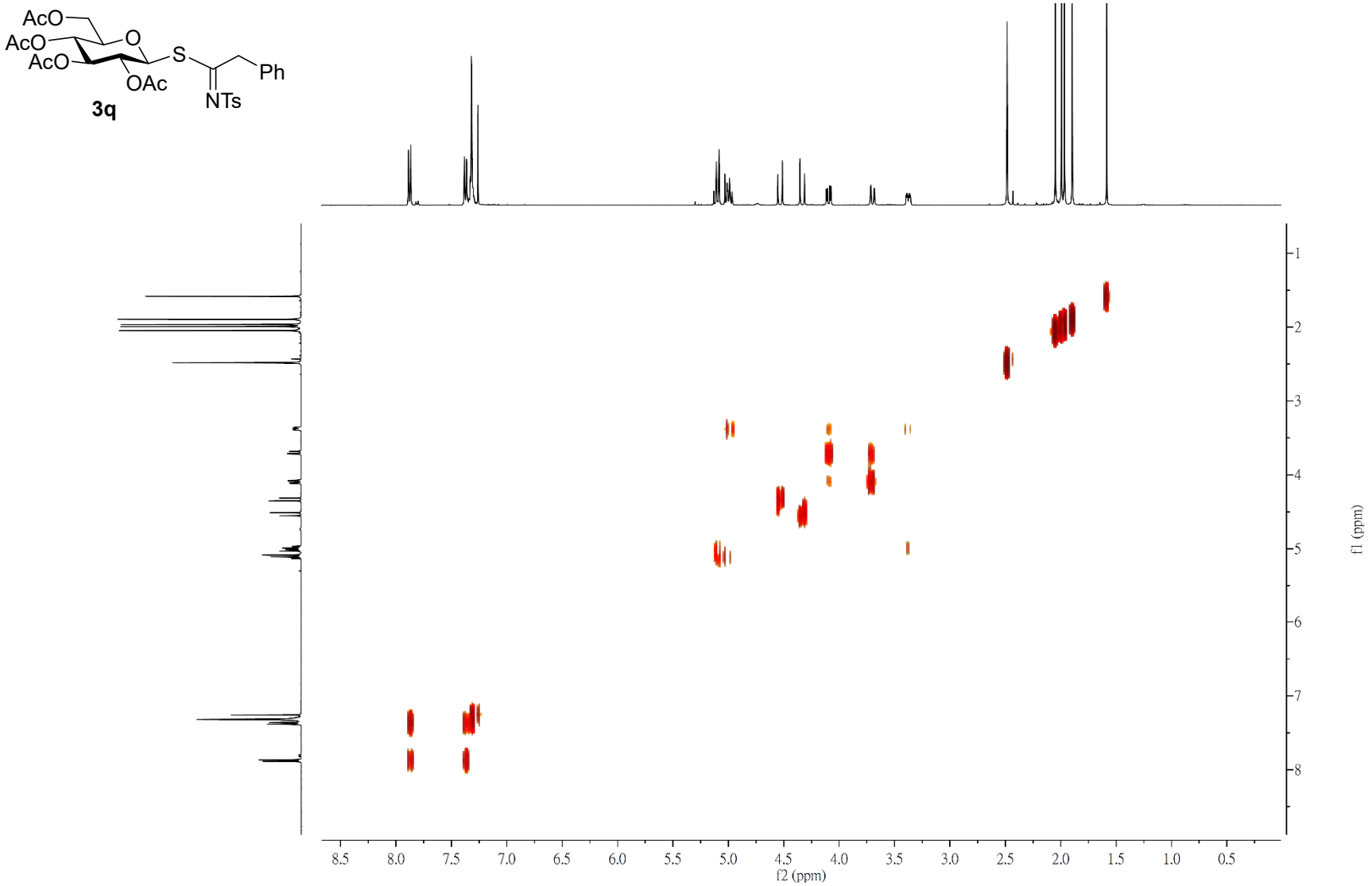
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	16
Receiver Gain	46
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.8
Nucleus	1H
Acquired Size	16384
Spectral Size	32768



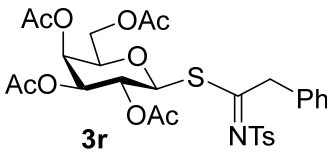
3q



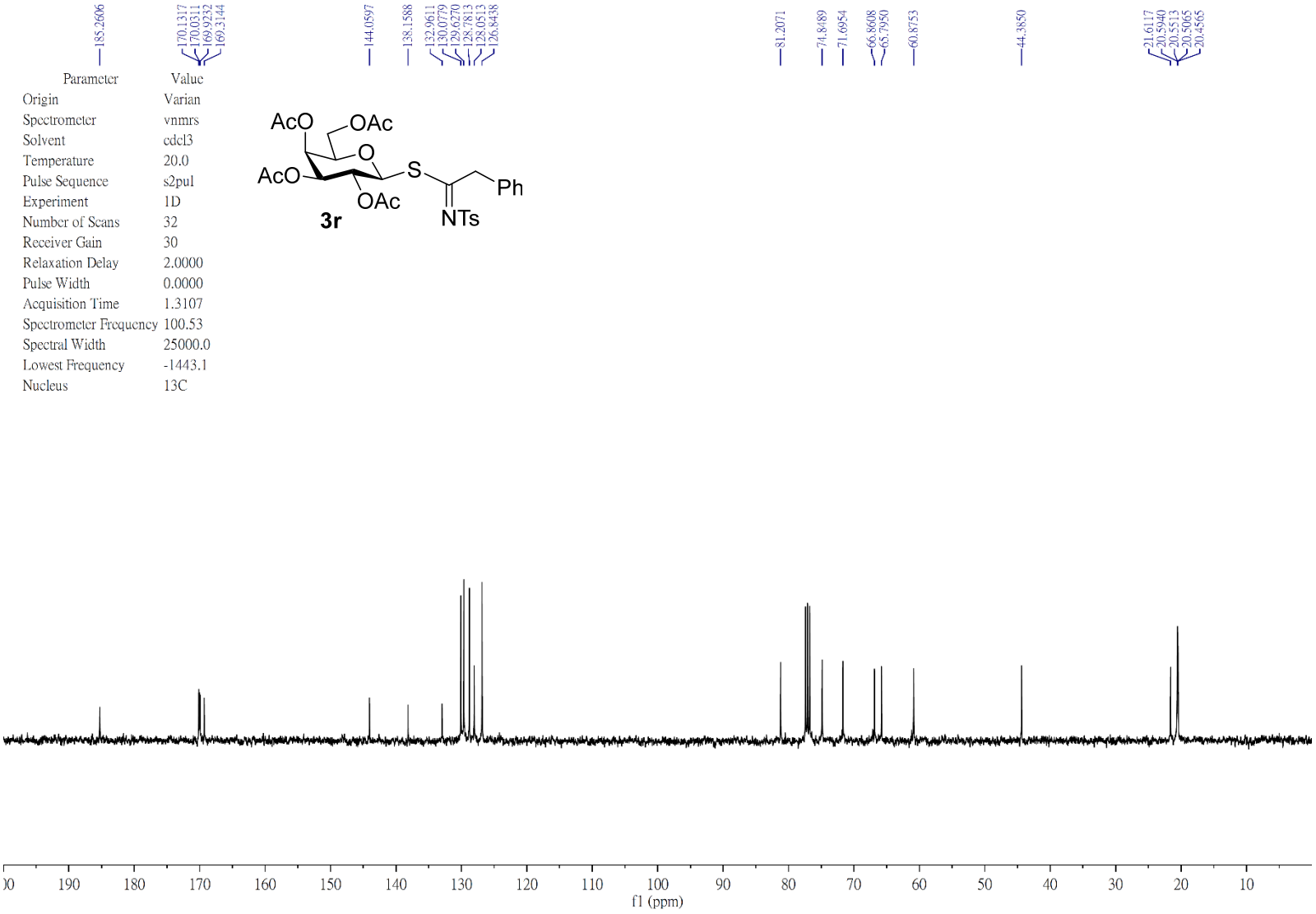

185.2247
170.5971
170.0921
169.1721
169.1381
144.1819
138.1022
132.8789
130.0976
129.6043
128.7955
128.0938
126.9172
80.7926
76.2451
73.6692
68.3877
67.5431
61.1870
44.3563
21.6425
20.6818
20.5304
20.3760
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 148
Receiver Gain 30
Relaxation Delay 2.0000
Pulse Width 0.0000
Acquisition Time 1.3107
Spectrometer Frequency 100.53
Spectral Width 25000.0
Lowest Frequency -1444.4
Nucleus 13C
AcO
AcO
AcO
O
S
Ph
OAc
NTs
3q
f1 (ppm)

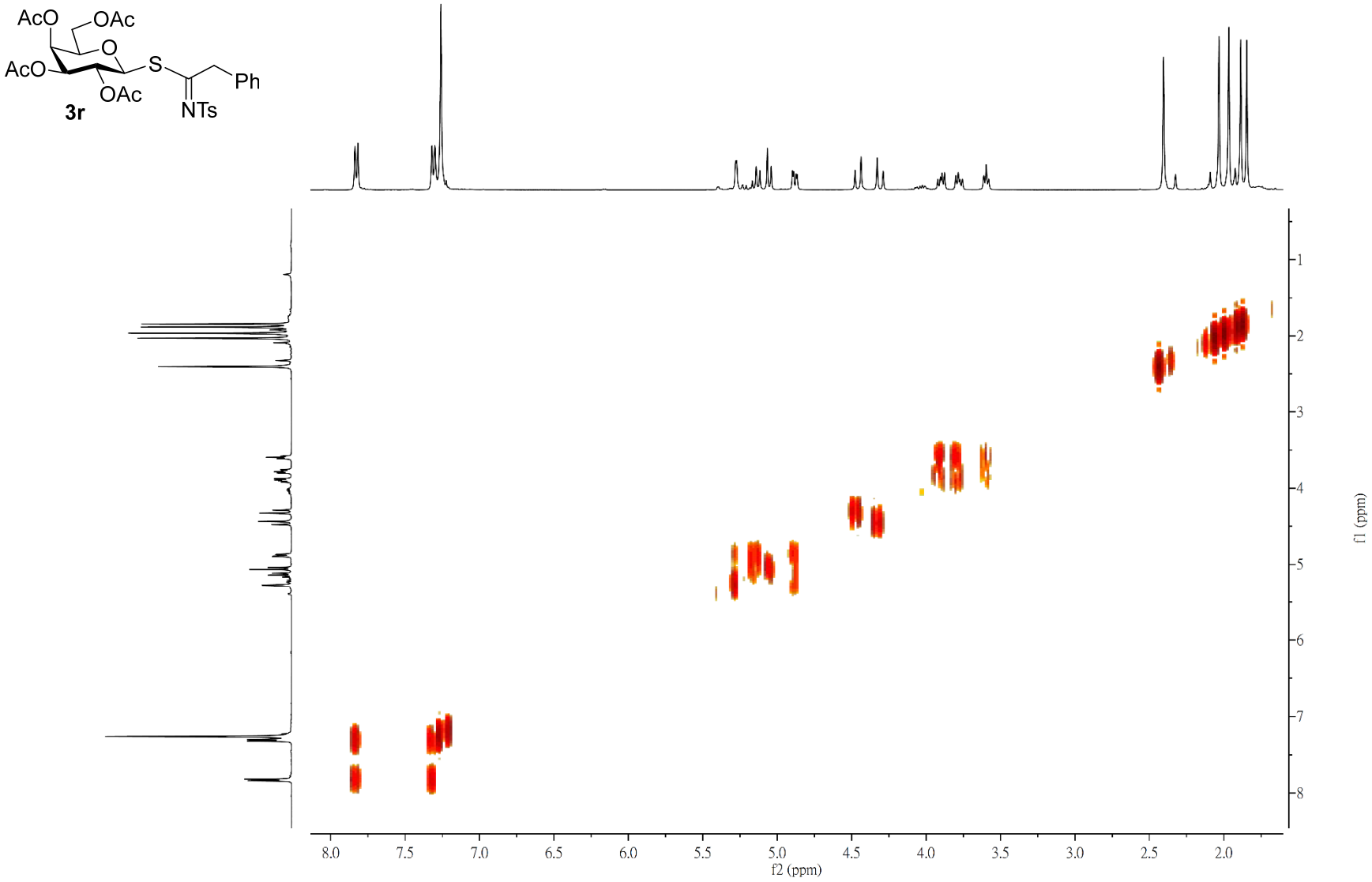

AcO
AcO
AcO
O
S
OAc
NTs
Ph
3q
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f2 (ppm)
1
2
3
4
5
6
7
8
f1 (ppm)

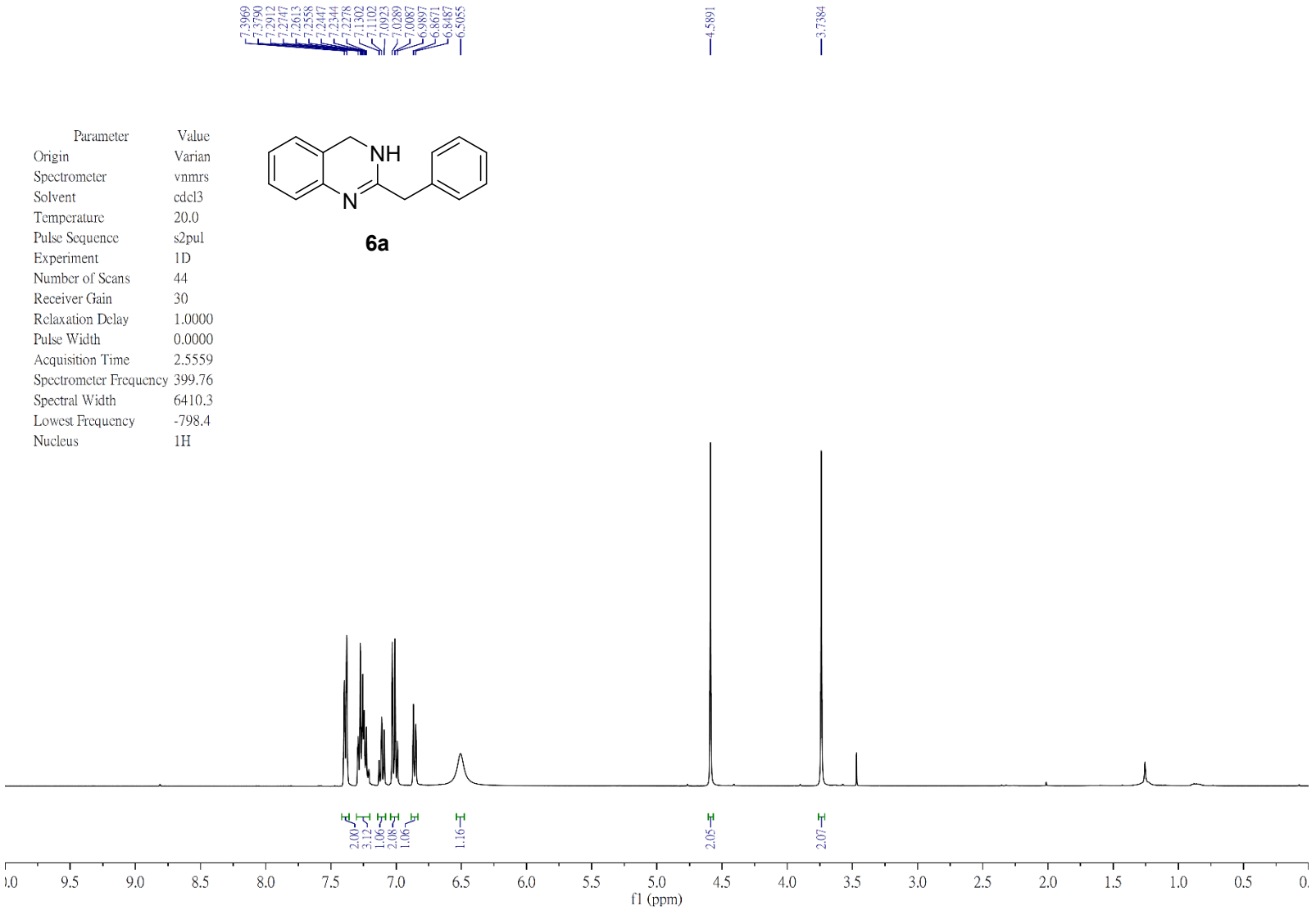
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	12
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-813.3
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

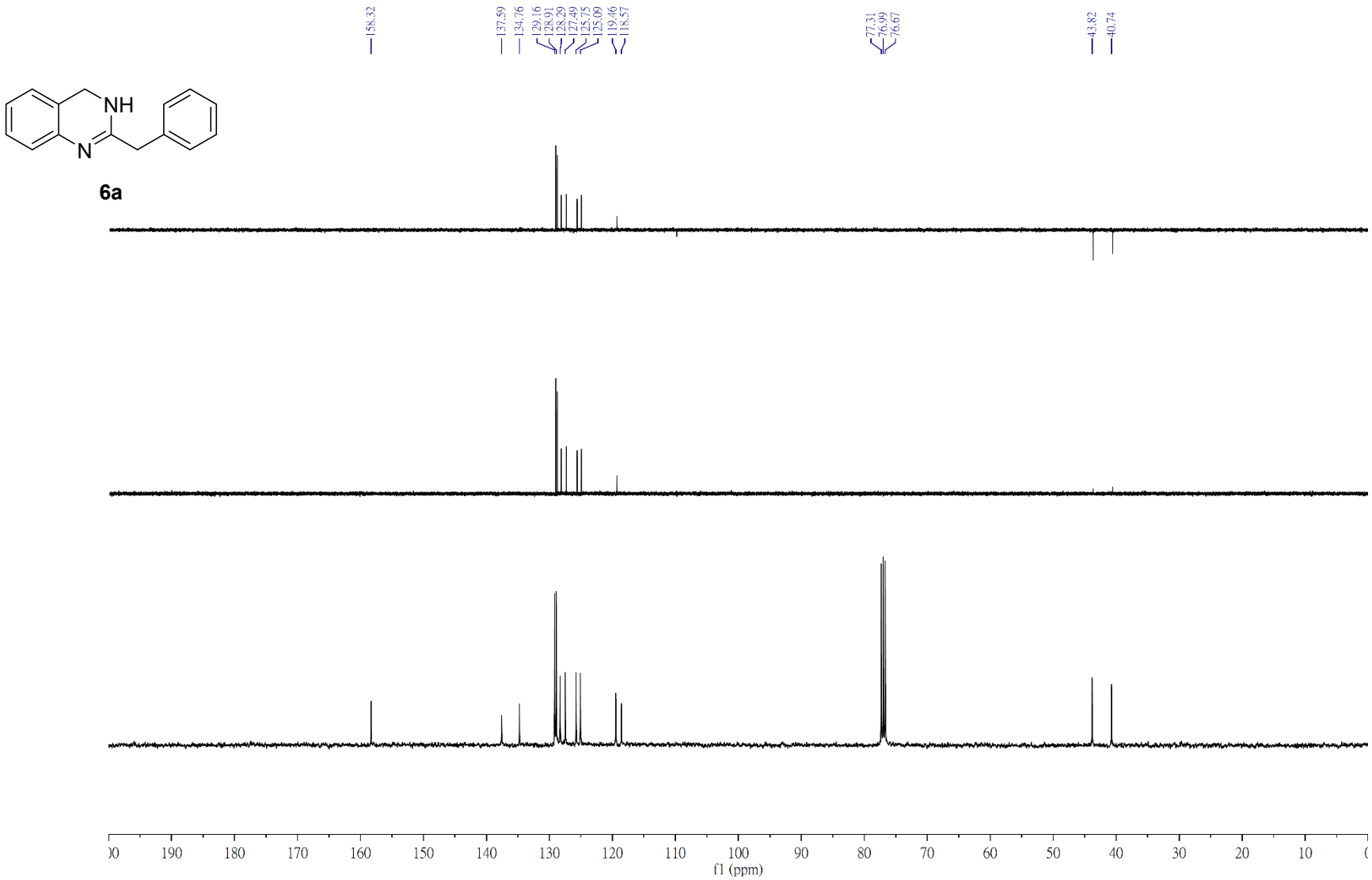


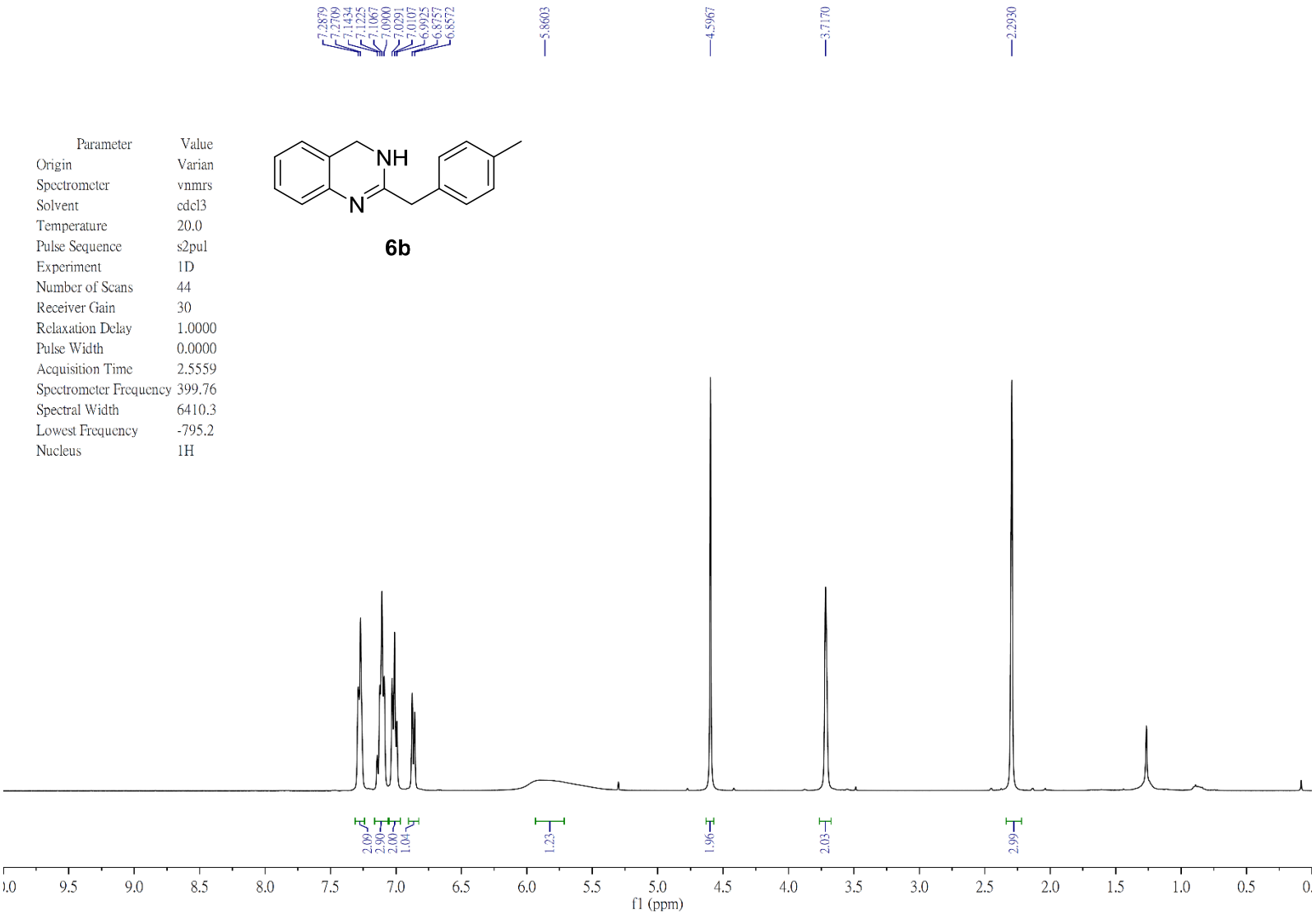
3r

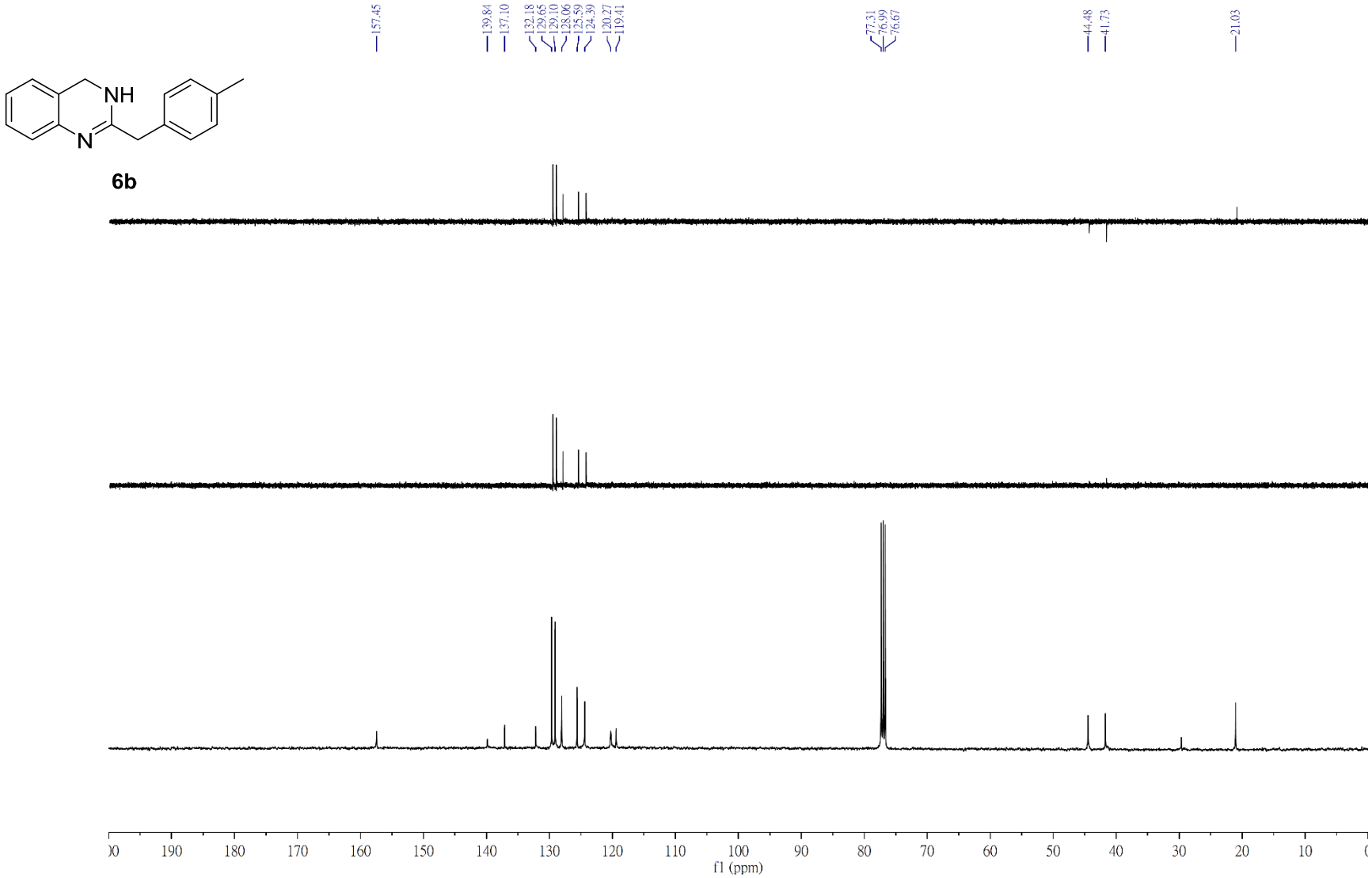

185.2606
170.1317
170.0311
169.9252
169.3144
144.0597
138.1588
132.9611
130.0779
129.6270
128.7813
128.0513
126.8438
81.2071
74.8489
71.6954
66.8608
65.7950
60.8753
44.3850
21.6117
20.5940
20.5513
20.5065
20.4565
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 32
Receiver Gain 30
Relaxation Delay 2.0000
Pulse Width 0.0000
Acquisition Time 1.3107
Spectrometer Frequency 100.53
Spectral Width 25000.0
Lowest Frequency -1443.1
Nucleus 13C
AcO
OAc
O
AcO
S
Ph
OAc
NTs
3r
f1 (ppm)

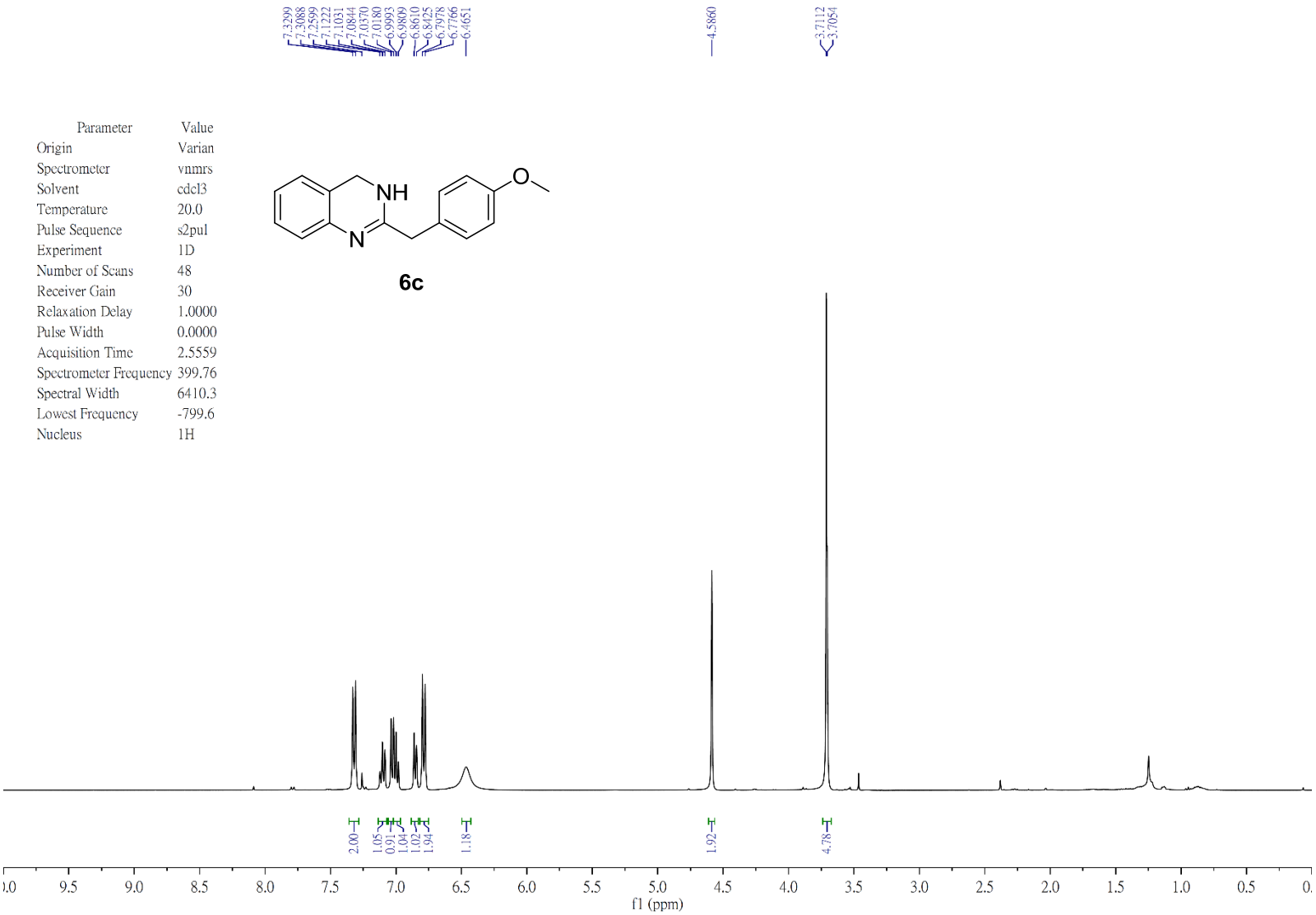

AcO
OAc
O
AcO
S
Ph
OAc
NTs
3r
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f2 (ppm)
1
2
3
4
5
6
7
8
f1 (ppm)

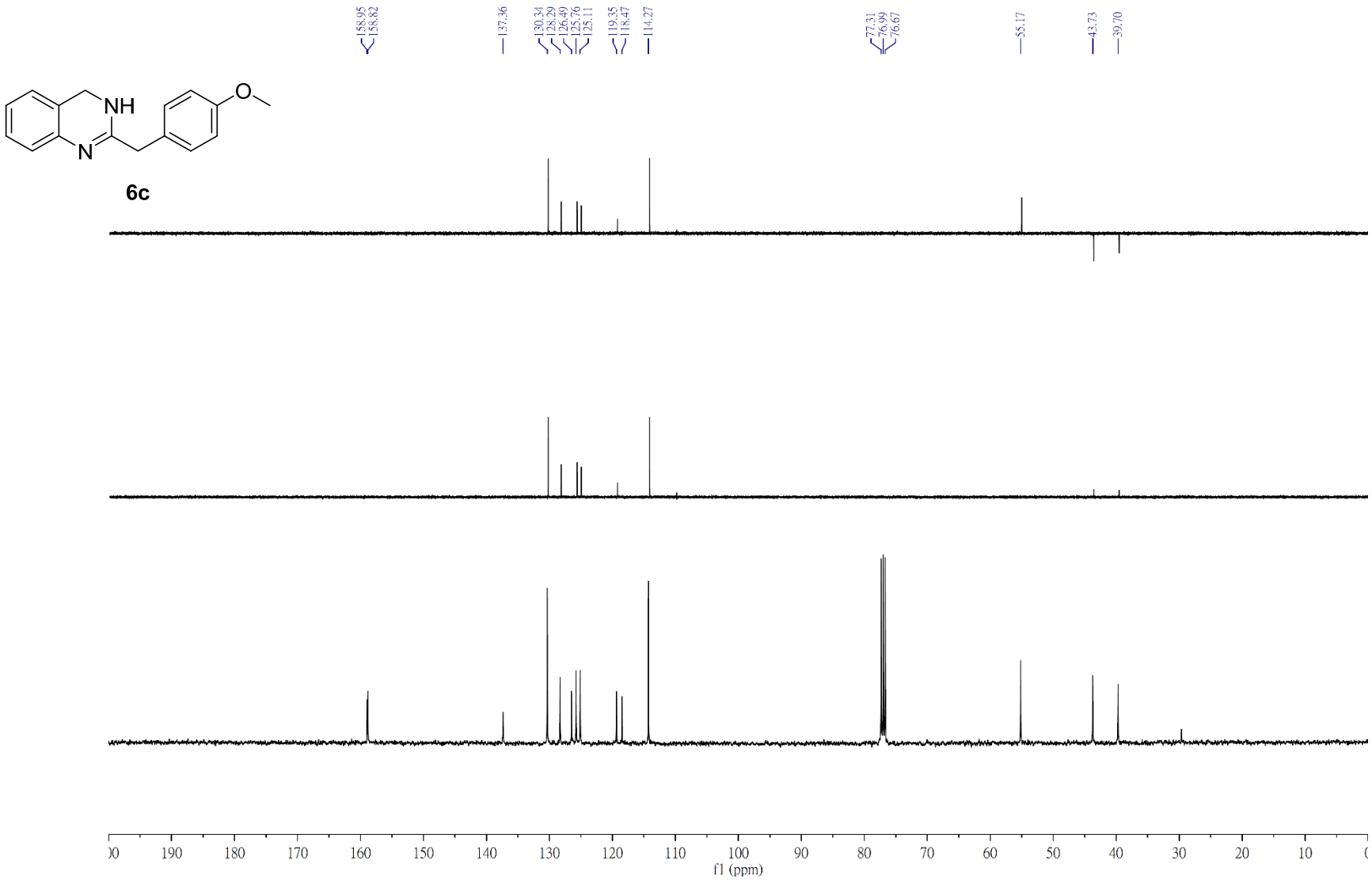

7.3969
7.3790
7.2912
7.2747
7.2613
7.2558
7.2447
7.2344
7.2278
7.1302
7.1102
7.0923
7.0289
7.0087
6.9897
6.8671
6.8487
6.5055
4.5891
3.7384
NH
N
6a
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 44
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -798.4
Nucleus 1H
2.00
3.12
1.06
2.08
1.06
1.16
2.05
2.07
f1 (ppm)

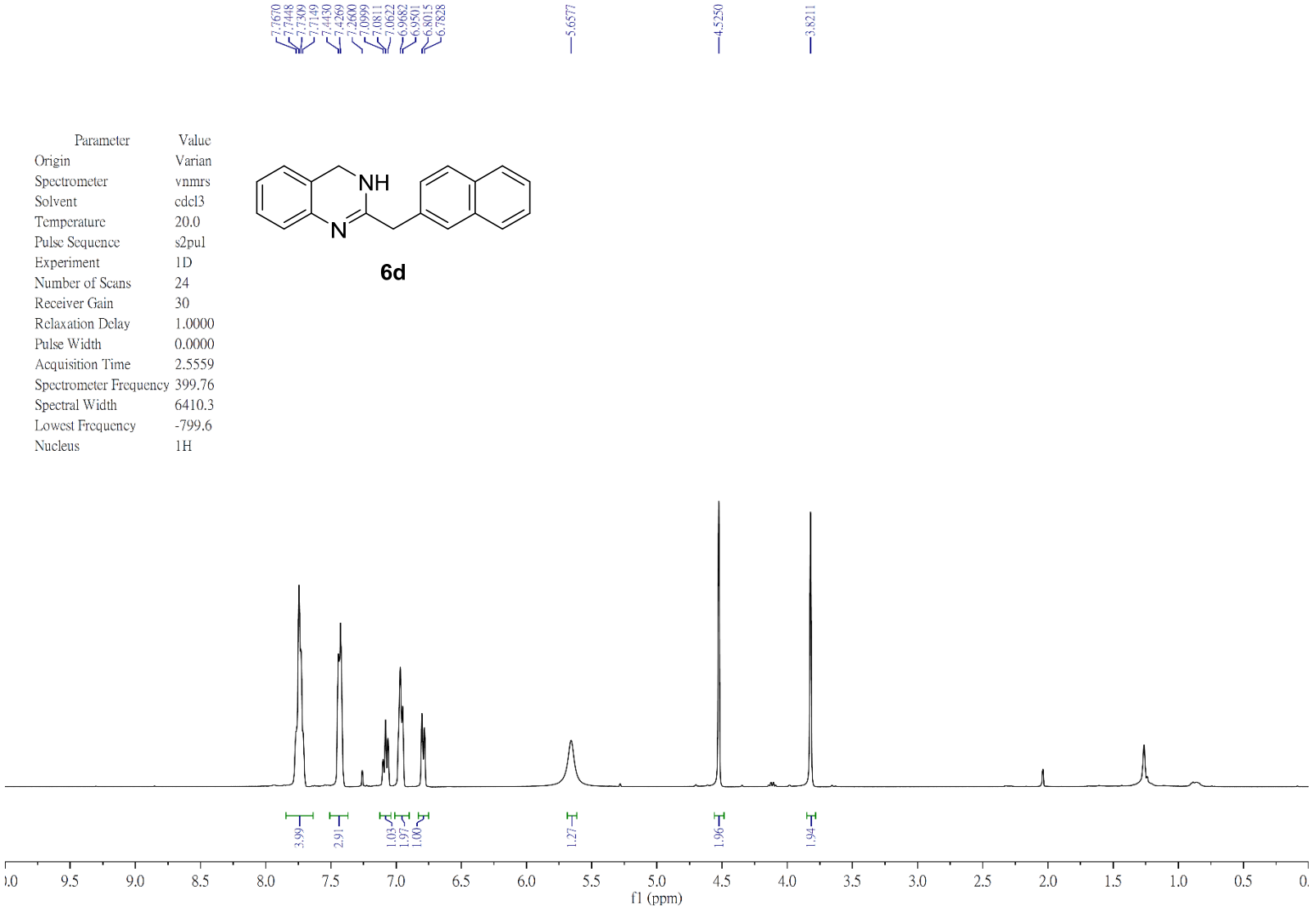

158.32
137.59
134.76
129.16
128.91
128.29
127.49
125.75
125.09
119.46
118.57
77.31
76.99
76.67
43.82
40.74
NH
N
6a
f1 (ppm)

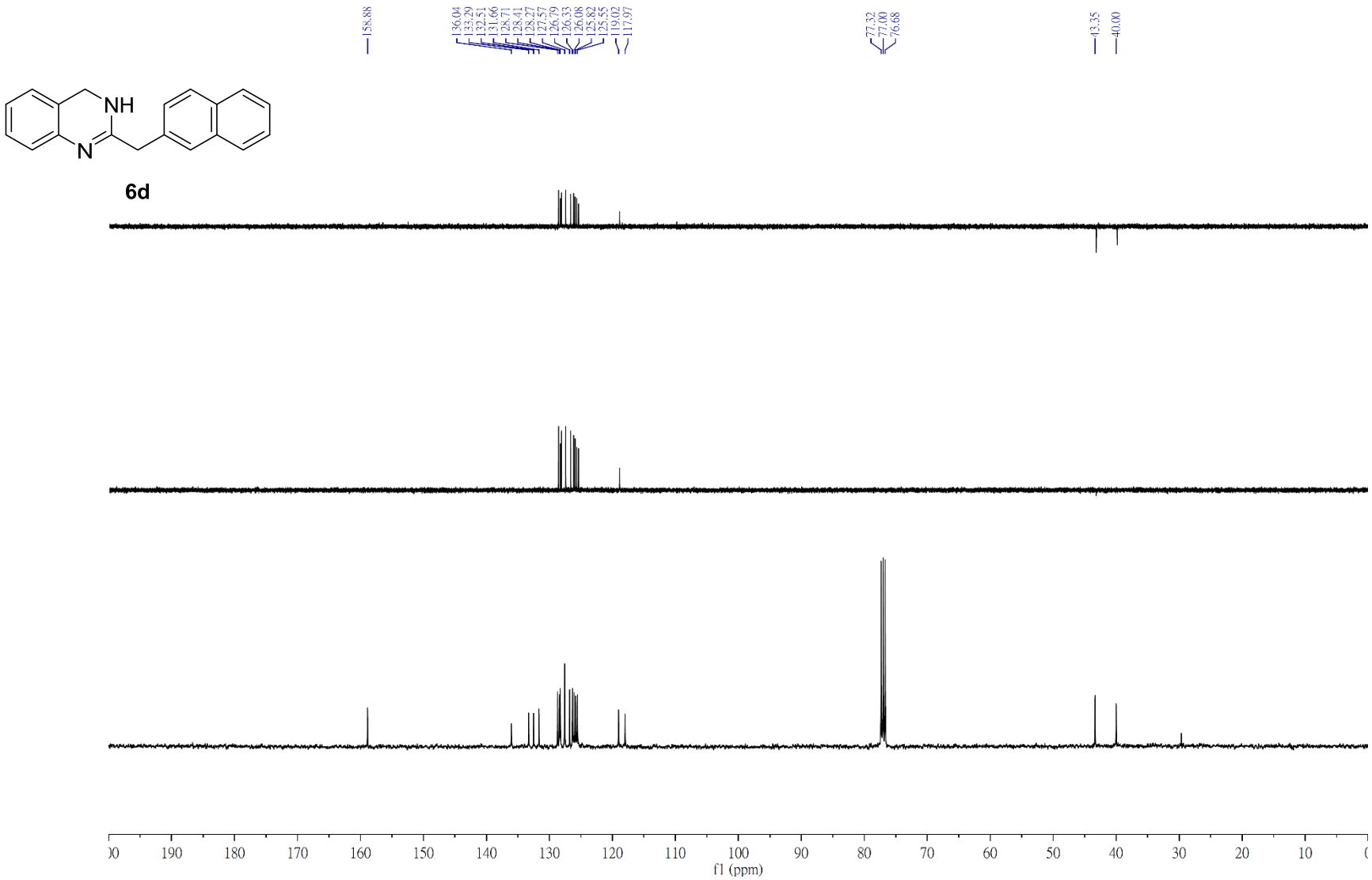

7.2879
7.2709
7.1434
7.1225
7.1067
7.0900
7.0291
7.0107
6.9925
6.8757
6.8572
5.8603
4.5967
3.7170
2.2930
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 44
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -795.2
Nucleus 1H
NH
N
6b
2.09
2.90
2.00
1.04
1.23
1.96
2.03
2.99
f1 (ppm)


157.45
139.84
137.10
132.18
129.65
129.10
128.06
125.59
124.39
120.27
119.41
77.31
76.99
76.67
44.48
41.73
21.03
NH
N
6b
f1 (ppm)

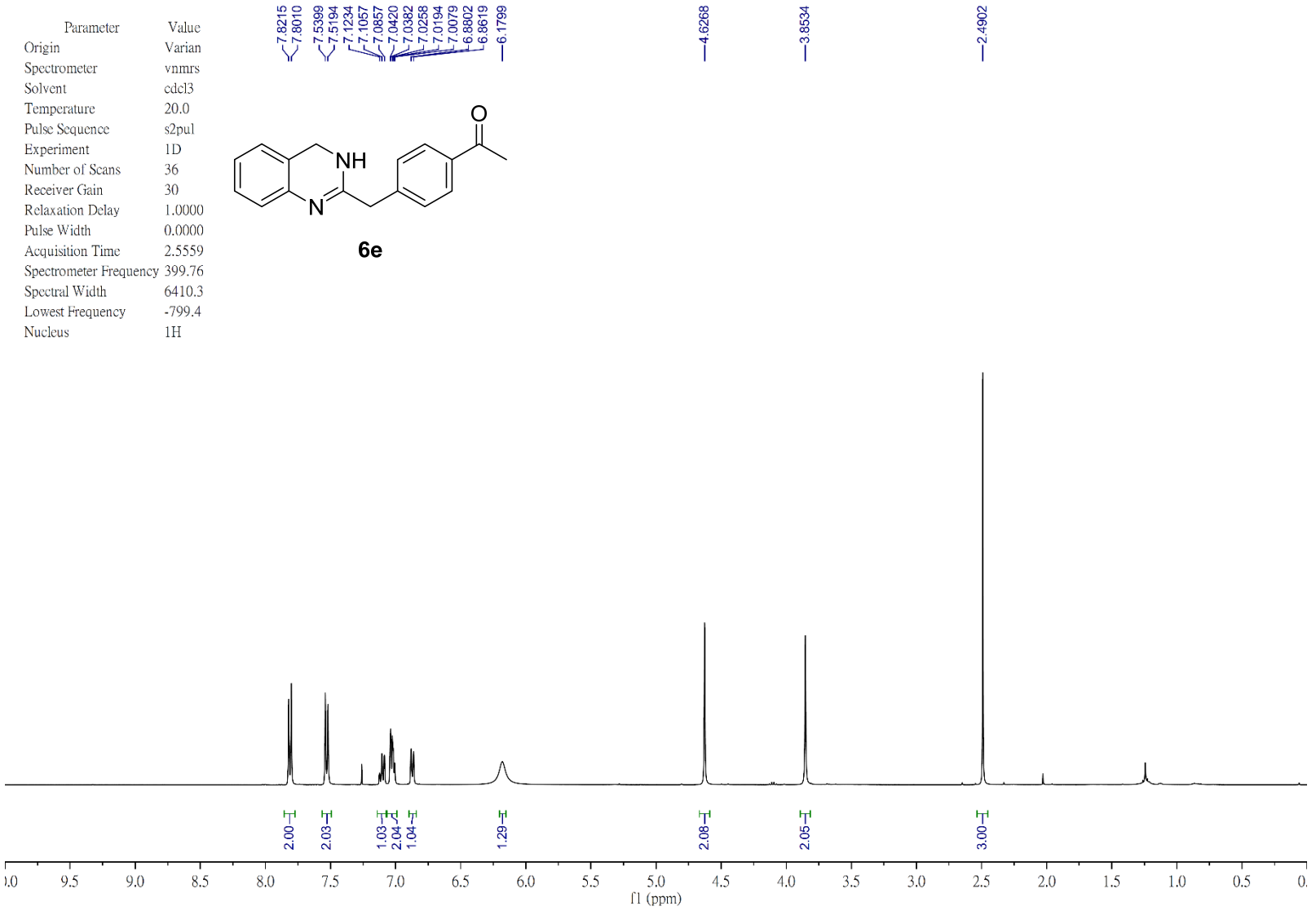

7.3209
7.3088
7.2599
7.1222
7.1031
7.0844
7.0370
7.0180
6.9993
6.9809
6.8610
6.8425
6.7978
6.7766
6.4651
4.5860
3.7112
3.7054
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 48
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
NH
N
O
6c
2.00
1.05
0.91
1.04
1.02
1.94
1.18
1.92
4.78
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


158.95
158.82
137.36
130.34
128.29
126.49
125.76
125.11
119.35
118.47
114.27
77.31
76.99
76.67
55.17
43.73
39.70
NH
N
O
6c
f1 (ppm)

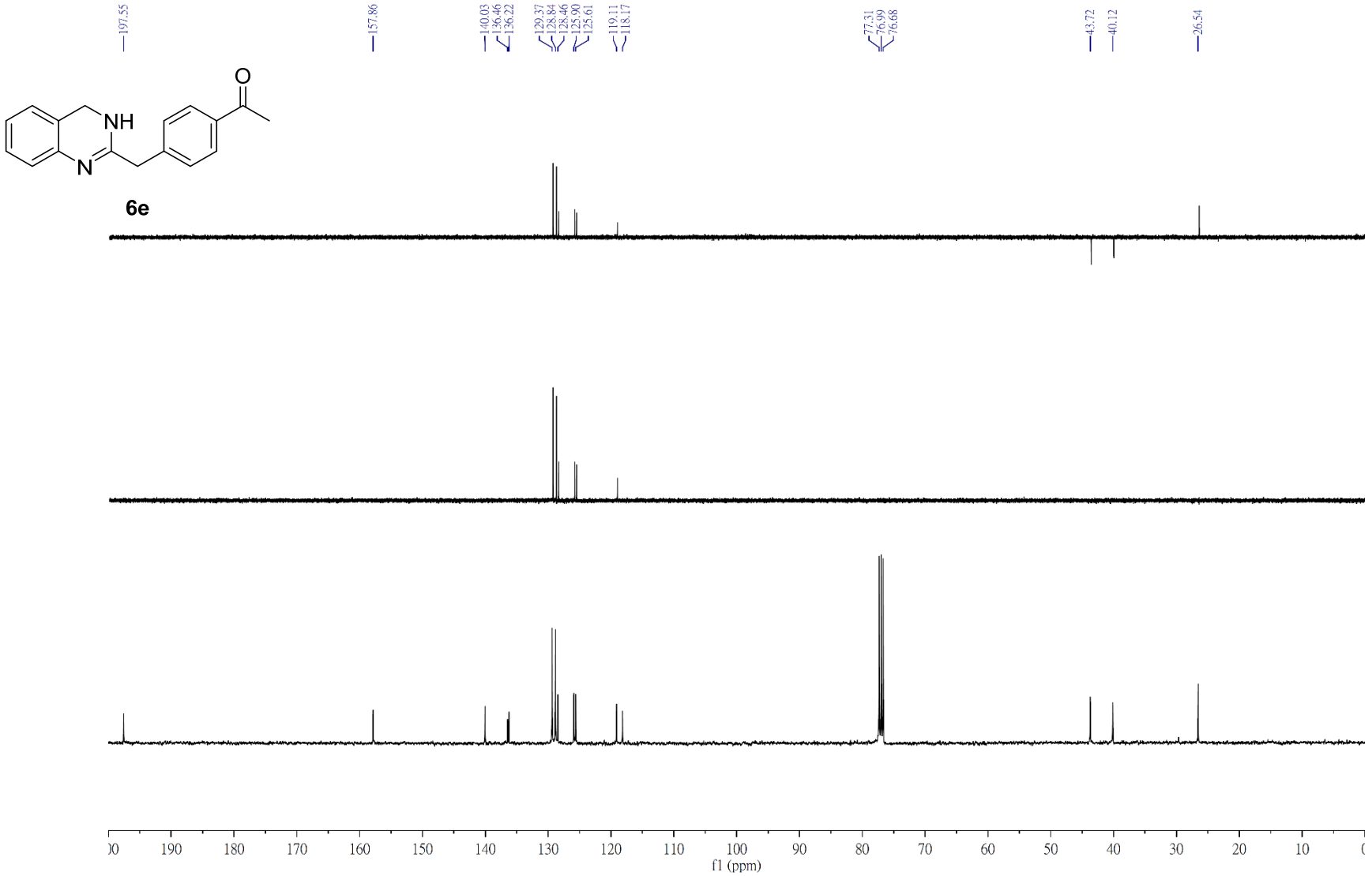

7.7670
7.7448
7.7309
7.7149
7.4430
7.4269
7.2600
7.0999
7.0811
7.0622
6.9682
6.9501
6.8015
6.7828
5.6577
4.5250
3.8211
NH
N
6d
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 24
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
3.99
2.91
1.03
1.97
1.00
1.27
1.96
1.94
f1 (ppm)

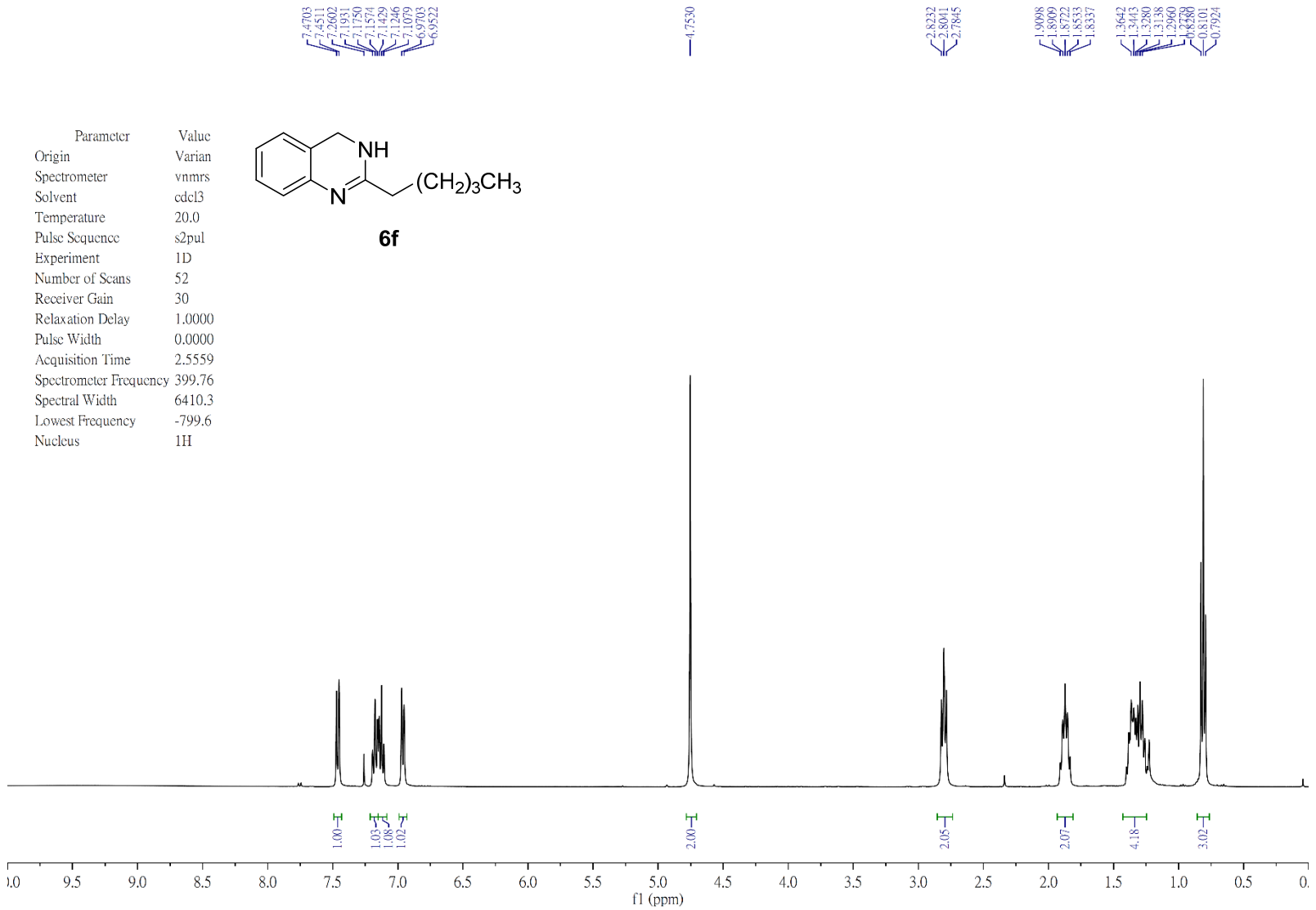

158.88
136.04
133.29
132.51
131.66
128.71
128.41
128.27
127.57
126.79
126.33
126.08
125.82
125.55
119.02
117.97
77.32
77.00
76.68
43.35
40.00
NH
N
6d
f1 (ppm)

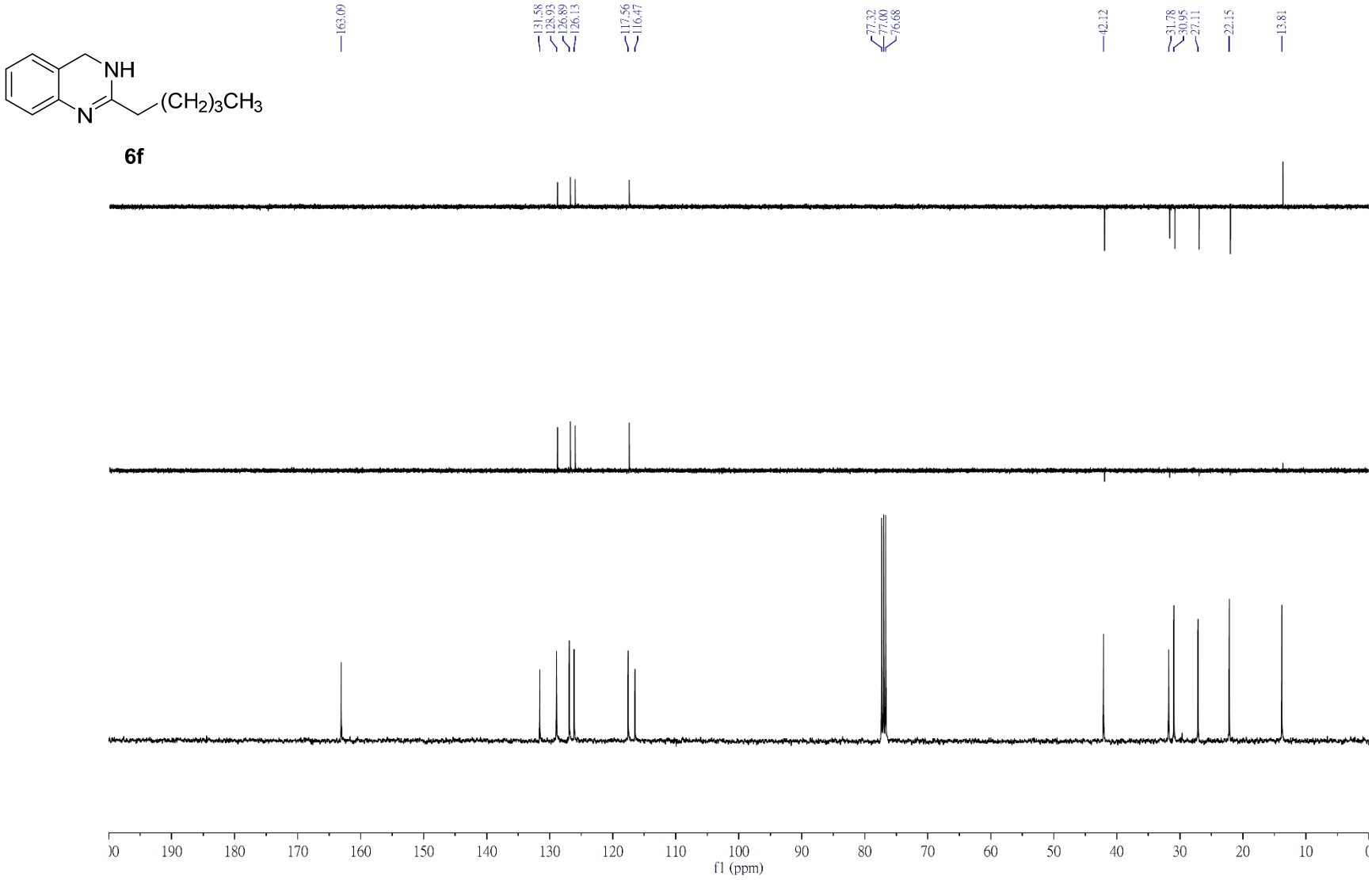
Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	36
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.4
Nucleus	1H

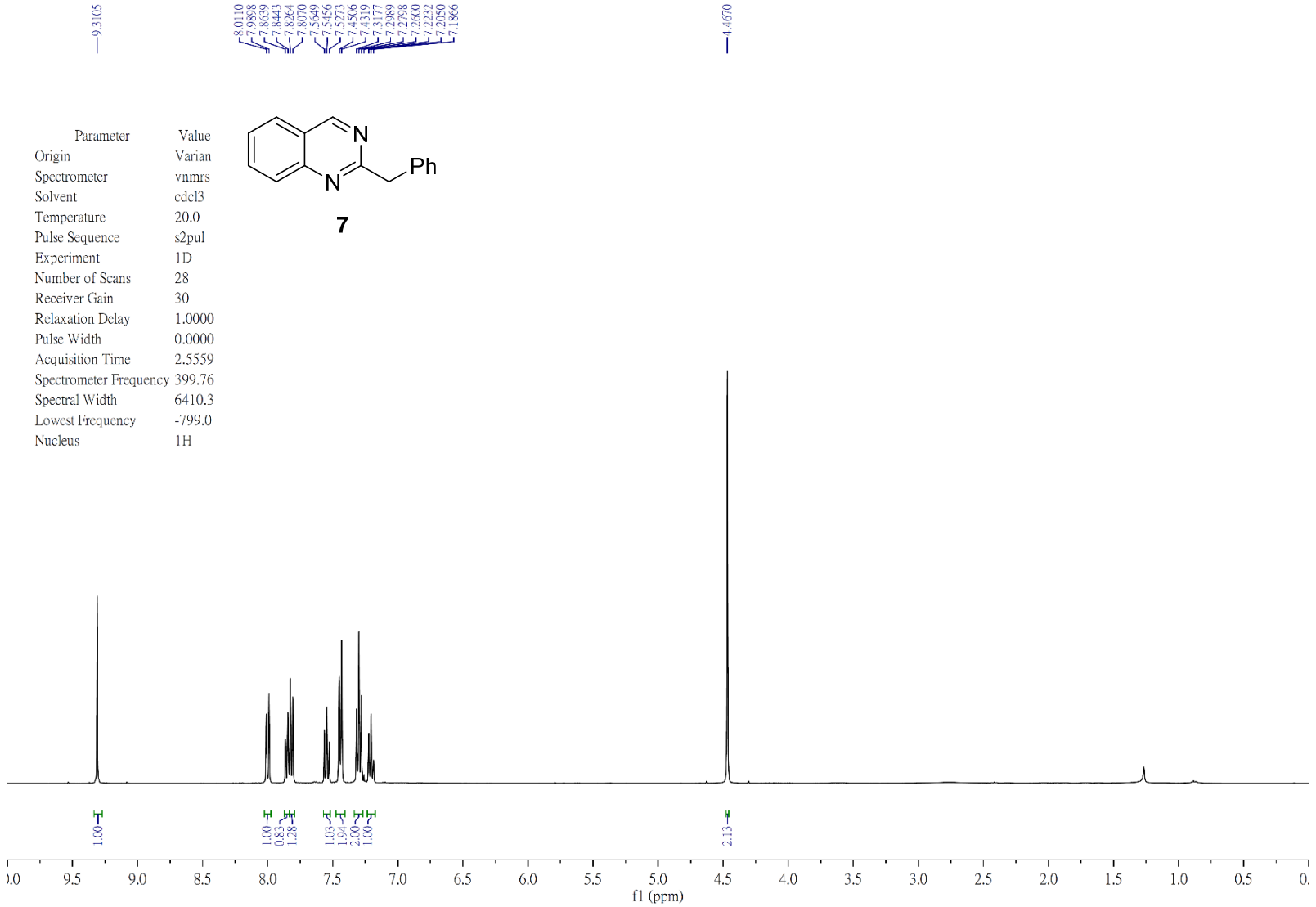


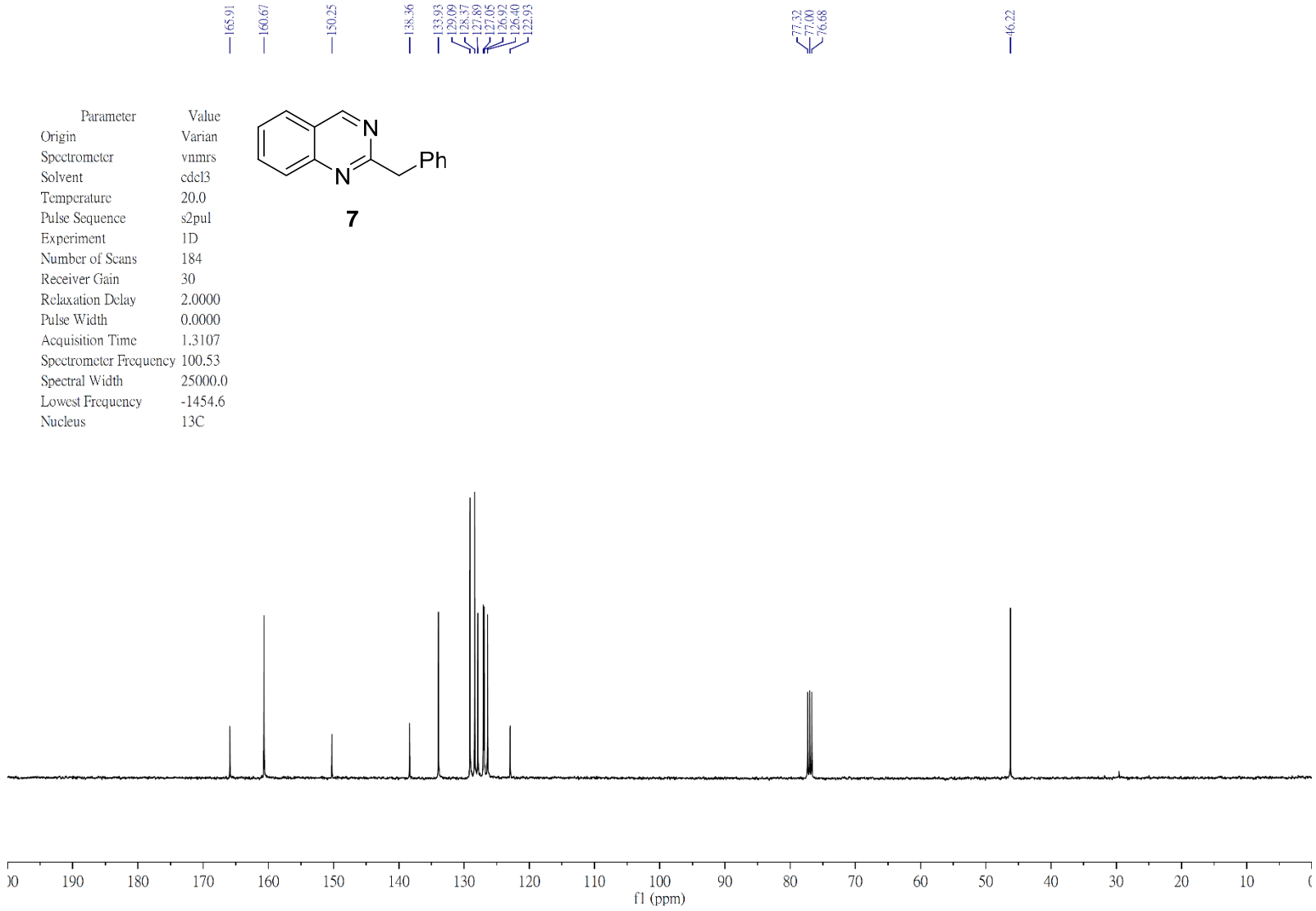
6e

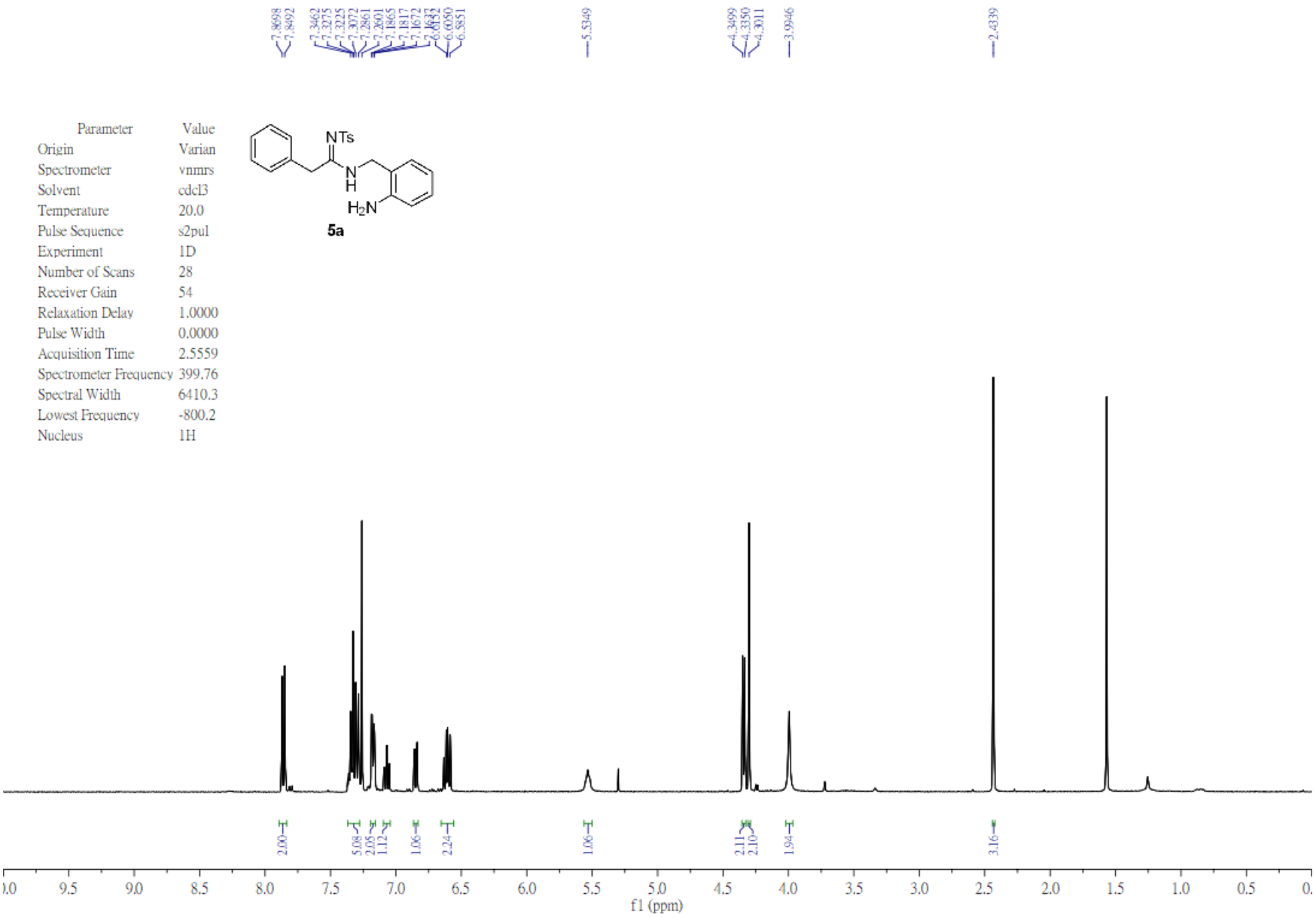

197.55
157.86
140.03
136.46
136.22
129.37
128.84
128.46
125.90
125.61
119.11
118.17
77.31
76.99
76.68
43.72
40.12
26.54
O
NH
N
6e
f1 (ppm)

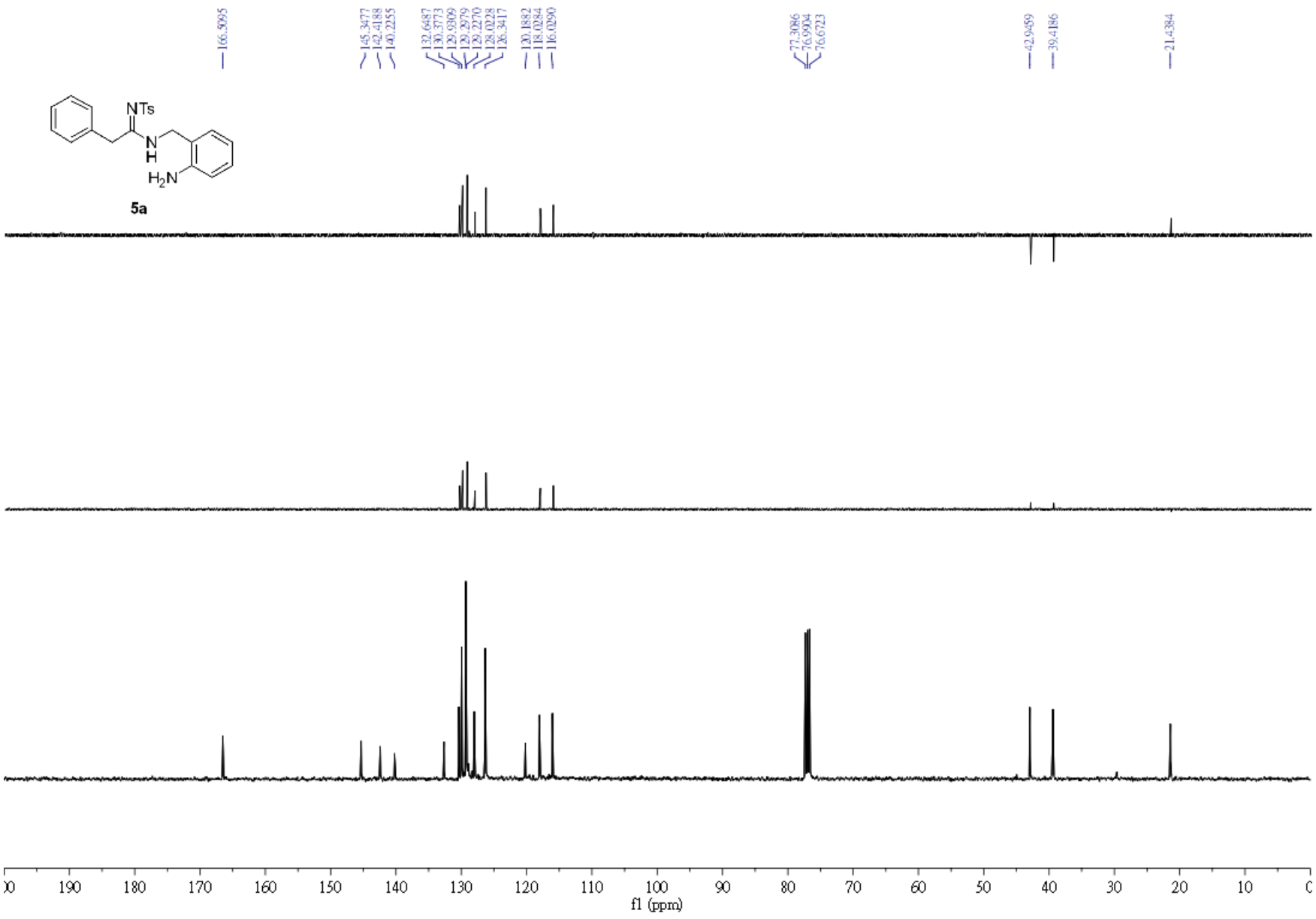

7.4703
7.4511
7.2602
7.1931
7.1750
7.1574
7.1429
7.1246
7.1079
6.9703
6.9522
1.7530
2.8232
2.8041
2.7845
1.9098
1.8909
1.8722
1.8533
1.8337
1.3642
1.3443
1.3280
1.3138
1.2960
1.2773
1.2580
0.8101
0.7924
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 52
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.6
Nucleus 1H
NH
N
(CH2)3CH3
6f
1.00
1.03
1.08
1.02
2.00
2.05
2.07
4.18
3.02
f1 (ppm)


NH
N
(CH2)3CH3
6f
163.09
131.58
128.93
126.89
126.13
117.56
116.47
77.32
77.00
76.68
42.12
31.78
30.95
27.11
22.15
13.81
f1 (ppm)


9.3105
8.0110
7.9808
7.8639
7.8443
7.8264
7.8070
7.5649
7.5456
7.5273
7.4506
7.4319
7.3177
7.2989
7.2798
7.2600
7.2232
7.2050
7.1866
4.4670
N
N
Ph
7
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 28
Receiver Gain 30
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -799.0
Nucleus 1H
1.00
1.00
0.83
1.28
1.03
1.94
2.00
1.00
2.13
f1 (ppm)


165.91
160.67
150.25
138.36
133.93
129.09
128.37
127.89
127.05
126.92
126.40
122.93
77.32
77.00
76.68
46.22
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 184
Receiver Gain 30
Relaxation Delay 2.0000
Pulse Width 0.0000
Acquisition Time 1.3107
Spectrometer Frequency 100.53
Spectral Width 25000.0
Lowest Frequency -1454.6
Nucleus 13C
N
N
Ph
7
f1 (ppm)


7.8698
7.8492
7.3462
7.3275
7.3225
7.3072
7.2861
7.2691
7.1865
7.1817
7.1672
6.6152
6.6050
6.5851
5.5349
4.3499
4.3350
4.3011
3.9946
2.4339
Parameter Value
Origin Varian
Spectrometer vnmrs
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Experiment 1D
Number of Scans 28
Receiver Gain 54
Relaxation Delay 1.0000
Pulse Width 0.0000
Acquisition Time 2.5559
Spectrometer Frequency 399.76
Spectral Width 6410.3
Lowest Frequency -800.2
Nucleus 1H
NTs
N
H
H2N
5a
2.00
5.08
2.05
1.12
1.06
2.24
1.06
2.11
2.10
1.94
3.16
f1 (ppm)


166.5095
145.3477
142.4188
140.2255
132.6487
130.3773
129.9309
129.2979
129.2270
128.0228
126.3417
120.1882
118.0284
116.0290
77.3086
76.9904
76.6723
42.9459
39.4186
21.4384
NTs
N
H
H2N
5a
f1 (ppm)