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Supplementary Information

Facile Access to Thioethers Using Sodium Thiosulfate as the Sulfur Surrogate under Base-Mediated Conditions

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General Information

All reactions were performed under nitrogen atmosphere. Solvents were purified following standard literature procedures. ^{1}H NMR and ^{13}C NMR spectra were reported on Varian 400 MHz NMR spectrometer and Jeol New 400 MHz NMR spectrometer with $CDCl_3$、*d*$_6$-DMSO as the solvent. Chemical shifts were reported in parts per million (ppm) relative to residual solvent peak ($CDCl_3$ δ H = 7.26 ppm, δ C = 77.16 ppm; $(CD_3)_2SO$ δ H = 2.50 ppm, δ C = 39.52 ppm). The IR spectra were measured on a Thermo Scientific Nicolet ATR-Diamond. Thin layer chromatography (TLC) was performed on pre-coated glass plates of Silica Gel 60 F254 (0.25 mm, E. Merck); TLC plates were visualized under UV light (254 or 365 nm) and by treatment with potassium permanganate ($KMnO_4$) and phosphomolybdic acid (PMA) as staining solution. Flash column chromatography was carried out by Silica Gel 60 (0.04-0.063 mm, Macherey-Nagel). High resolution mass spectrometry (HRMS) data were recorded on an EI and ESI spectrometer. Melting points were measured by Electrothermo (UK) "Mel- Temp " type melting point apparatus and are uncorrected. The following abbreviations were used to indicate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, quin. = quintet, sex = sextet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, ddd = doublet of doublet of doublets, br = broad, m = multiplet. Coupling constants (*J*) are reported in Hz. Yields of products refer to chromatographically purified products unless otherwise stated. All organic halides and sodium thiosulfate were purchase from Sigma Aldrich, Alfa Aesar, Merck, or TCI. All these compounds were used as received, unless otherwise stated.

Experimental Procedures

General Procedure I: For the synthesis of alkyl Bunte salt[1]

To a solution of the alkyl halide **1** (1.0 equiv) in MeOH/H_2O (3:1, 0.25 M) was added

$Na_2S_2O_3$ (1.2 equiv) and stirred at 65 °C for 8~10 hours, then was cooled to room temperature and concentrated on a rotary evaporator at a bath temperature of 40 °C to remove MeOH/H_2O. The processed solid was treated with heated MeOH (0.1 M) to let most of the solid dissolve. The mixture was passed through a frit funnel and the resulting liquid was concentrated on a rotary evaporator to yield a solid. The solid was then subjected to trituration with hexane, followed by filtration and vacuum drying to afford the desired product.

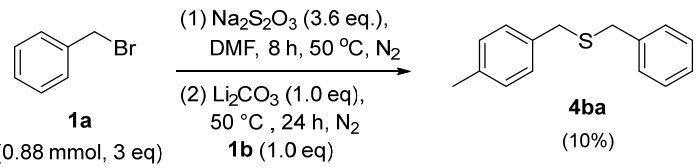
General Procedure II: For the synthesis of symmetric thioether

To a solution of the organic halide **1** (1.0 equiv) in DMF (1.5 M) was added anhydrous sodium thiosulfate (1.0 equiv), Li_2CO_3 (1.0 equiv) and stirred at 70 °C under nitrogen atmosphere for 24 hours. The resulting reaction mixture was completed as indicated by TLC. The mixture was mixed with silica gel first and purified by flash column chromatography to afford the desired product.

General procedure III: For the synthesis of unsymmetric thioether

To a solution of the alkyl Bunte salt **2** (3.0 equiv) in DMF (0.2 M) was added the organic halide **1** (1.0 equiv), Li_2CO_3 (1.0 equiv) and stirred at 50 °C under nitrogen atmosphere for 24 hours. The resulting reaction mixture was extracted with Et_2O and water for three times, and the combined organic phase was dried over $MgSO_4$, filtered and concentrated in rotary evaporators. Then the mixture was mixed with silica gel and purified by flash column chromatography to afford the desired product.

One-pot Synthesis of unsymmetric thioether

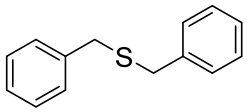


To a solution of the benzyl bromide **1a** (0.88 mmol, 3.0 equiv) in DMF (1.5 M) was

added anhydrous sodium thiosulfate (3.6 equiv) and stirred at 50 °C under a nitrogen atmosphere for 8 hours to generate the Bunte salt **2a**. Subsequently, organic bromide **1b** (1.0 equiv) and Li_2CO_3 (1.0 equiv) were added to the reaction mixture and stirred at 50 °C under nitrogen atmosphere for 24 hours. The resulting reaction mixture was extracted three times with Et_2O and water. The combined organic phase was dried over $MgSO_4$, filtered, and concentrated using a rotary evaporator. The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ba** (6.7 mg, 10 % yield) as a colorless oil.

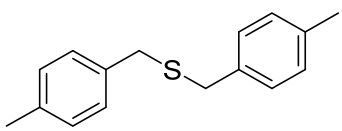
Characterization Data

Dibenzyl sulfide (3a)[2]



Following general procedure II, using benzyl bromide **1a** (100.0 mg, 0.585 mmol), anhydrous sodium thiosulfate (93.0 mg, 0.585 mmol), Li_2CO_3 (43.0 mg, 0.585 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3a** (52.5 mg, 83 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.34 – 7.25 (m, 10H), 3.61 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : 138.2, 129.1, 128.5, 127.0, 35.7; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{14}S$: 214.0816, found: 214.0812.
The spectral data of **3a** match those reported in the literature.[2]

Bis(4-methylbenzyl) sulfide (3b)[2]

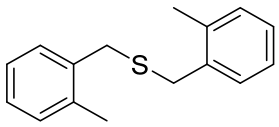


Following general procedure II, using 4-methylbenzyl bromide **1b** (146.0 mg, 0.789

mmol), anhydrous sodium thiosulfate (125.0 mg, 0.789 mmol), Li_2CO_3 (58.0 mg, 0.789 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3b** (79.9 mg, 84 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, *J* = 8.0 Hz, 4H), 7.12 (d, *J* = 8.0 Hz, 4H), 3.57 (s, 4H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.6, 135.2, 129.2, 129.0, 35.3, 21.2; HRMS (EI) m/z: $[M]^+$ calcd. for $C_{16}H_{18}S$: 242.1129, found: 242.1125.

The spectral data of **3b** match those reported in the literature.[2]

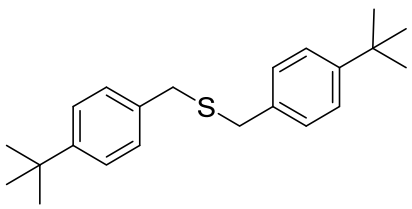
Bis(2-methylbenzyl) sulfide (3c)[2]



Following general procedure II, using 2-methylbenzyl bromide **1c** (146.0 mg, 0.789 mmol), anhydrous sodium thiosulfate (125.0 mg, 0.789 mmol), Li_2CO_3 (58.0 mg, 0.789 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3c** (66.0 mg, 69 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.11 (m, 8H), 3.67 (s, 4H), 2.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.0, 135.9, 130.8, 129.8, 127.4, 126.0, 34.4, 19.2; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{16}H_{19}S$: 243.1208, found: 243.1216.

The spectral data of **3c** match those reported in the literature.[2]

Bis(4-*tert*-butylbenzyl) sulfide (3d)[2]

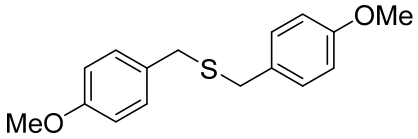


Following general procedure II, using 4-*tert*-butylbenzyl bromide **1d** (140.0 mg, 0.616

mmol), anhydrous sodium thiosulfate (97.4 mg, 0.616 mmol), Li_2CO_3 (45.5 mg, 0.616 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3d** (52.4 mg, 52 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.0 Hz, 4H), 7.23 (d, *J* = 8.0 Hz, 4H), 3.60 (s, 4H), 1.33 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 135.4, 128.8, 125.5, 35.4, 34.6, 31.5; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{22}H_{31}S$: 327.2147, found: 327.2148.

The spectral data of **3d** match those reported in the literature.[2]

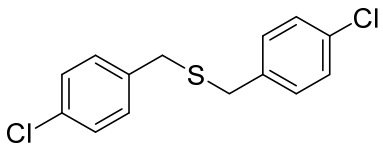
Bis(4-methoxybenzyl) sulfide (3e)[4]



Following general procedure II, using 4-methoxybenzyl bromide **1e** (385.0 mg, 1.915 mmol), anhydrous sodium thiosulfate (1.0 equiv. 302.0 mg, 1.915 mmol), Li_2CO_3 (1.0 equiv. 141.0 mg, 1.915 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 3:1, v/v) to afford the desired product **3e** (119.6 mg, 46 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 8.5 Hz, 4H), 6.86 (d, *J* = 8.5 Hz, 4H), 3.81 (s, 6H), 3.56 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.7, 130.3, 130.1, 114.0, 55.4, 35.0; HRMS (EI) m/z: $[M]^+$ calcd. for $C_{16}H_{18}SO_2$: 274.1028, found: 274.1023.

The spectral data of **3e** match those reported in the literature.[4]

Bis(4-chlorobenzyl) sulfide (3f)[2]

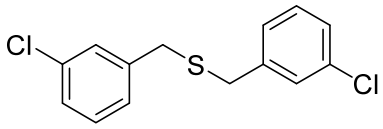


Following general procedure II, using 4-chlorobenzyl bromide **1f** (161.0 mg, 0.784

mmol), anhydrous sodium thiosulfate (124.0 mg, 0.784 mmol), Li_2CO_3 (58.0 mg, 0.784 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3f** (93.2 mg, 84 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 4H), 7.19 (d, *J* = 8.4 Hz, 4H), 3.54 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.5, 132.9, 130.4, 128.8, 34.9; HRMS (EI) m/z : $[M]^+$ calcd. for $^{12}C_{14}H_{12}Cl_2S$: 282.0037, found: 282.0033.

The spectral data of **3f** match those reported in the literature.[2]

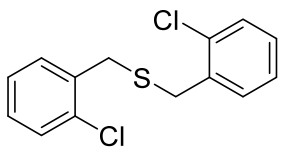
Bis(3-chlorobenzyl) sulfide (3g)[3]



Following general procedure II, using 3-chlorobenzyl bromide **1g** (161.0 mg, 0.784 mmol), anhydrous sodium thiosulfate (124.0 mg, 0.784 mmol), Li_2CO_3 (58.0 mg, 0.784 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3g** (90.0 mg, 81 % yield) as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.19 (m, 6H), 7.14 (d, *J* = 7.0 Hz, 2H), 3.56 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 134.5, 129.9, 129.2, 127.5, 127.3, 35.3; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{14}H_{13}Cl_2S$: 283.0115, found: 283.0108.

The spectral data of **3g** match those reported in the literature.[3]

Bis(2-chlorobenzyl) sulfide (3h)[2]

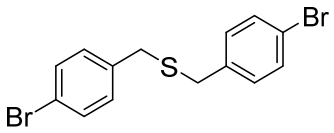


Following general procedure II, using 2-chlorobenzyl bromide **1h** (120.0 mg, 0.584 mmol), anhydrous sodium thiosulfate (92.3 mg, 0.584 mmol), Li_2CO_3 (43.2 mg, 0.584

mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3h** (54.6 mg, 66 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.35 (m, 4H), 7.22 – 7.18 (m, 4H), 3.82 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.8, 134.3, 130.8, 129.9, 128.6, 127.0, 33.9; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{14}H_{13}Cl_2S$: 283.0115, found: 283.0106.

The spectral data of **3h** match those reported in the literature.[2]

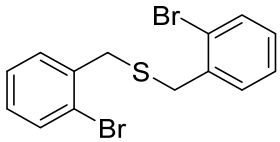
Bis(4-bromobenzyl) sulfide (3i)[2]



Following general procedure II, using 4-bromobenzyl bromide **1i** (200.0 mg, 0.800 mmol), anhydrous sodium thiosulfate (126.0 mg, 0.800 mmol), Li_2CO_3 (59.0 mg, 0.800 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3i** (128.1 mg, 86 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.43 (d, J = 8.2 Hz, 4H), 7.13 (d, J = 8.2 Hz, 4H), 3.52 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 137.0, 131.8, 130.8, 121.1, 35.1; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{12}Br_2S$: 369.9026, found: 369.9021.

The spectral data of **3i** match those reported in the literature.[2]

Bis(2-bromobenzyl) sulfide (3j)[3]

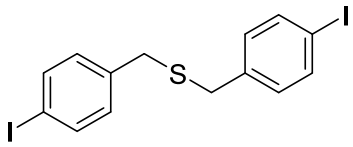


Following general procedure II, using 2-bromobenzyl bromide **1j** (195.0 mg, 0.780 mmol), anhydrous sodium thiosulfate (123.0 mg, 0.780 mmol), Li_2CO_3 (58.0 mg, 0.780 mmol). The crude product was then purified by column chromatography

(hexane/CH_2Cl_2, 6:1, v/v) to afford **3j** (118.1 mg, 81 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.54 (dd, *J* = 8.0, 1.0 Hz, 2H), 7.35 (dd, *J* = 7.6, 1.5 Hz, 2H), 7.24 (td, *J* = 7.5, 1.6 Hz, 2H), 7.09 (td, *J* = 7.8, 1.6 Hz, 2H), 3.83 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : 137.4, 133.2, 130.9, 128.8, 127.6, 124.8, 36.6; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{12}Br_2S$: 369.9026, found: 369.9033.

The spectral data of **3j** match those reported in the literature.[3]

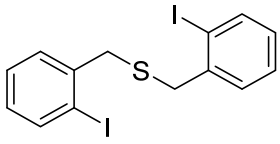
Bis(4-iodobenzyl) sulfide (3k)[4]



Following general procedure II, using 4-iodobenzyl bromide **1k** (125.9 mg, 0.424 mmol), anhydrous sodium thiosulfate (67.0 mg, 0.424 mmol), Li_2CO_3 (31.3 mg, 0.424 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3k** (70.2 mg, 71 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.2 Hz, 4H), 7.01 (d, *J* = 8.2 Hz, 4H), 3.51 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.7, 137.7, 131.1, 92.5, 35.1; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{13}I_2S$: 466.8827, found: 466.8843.

The spectral data of **3k** match those reported in the literature.[4]

Bis(2-iodobenzyl) sulfide (3l)[3]

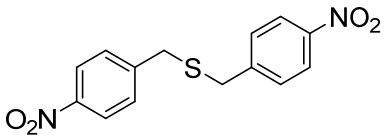


Following general procedure II, using 2-iodobenzyl bromide **1l** (126.0 mg, 0.424 mmol), anhydrous sodium thiosulfate (67.0 mg, 0.424 mmol), Li_2CO_3 (31.0 mg, 0.424

mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3l** (84.7 mg, 86 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.84 (d, *J* = 7.9 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 2H), 6.93 (t, *J* = 7.6 Hz, 2H), 3.80 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : 140.3, 139.9, 130.2, 128.9, 128.4, 101.0, 41.5; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{12}I_2S$: 465.8749, found : 465.8745.

The spectral data of **3l** match those reported in the literature.[3]

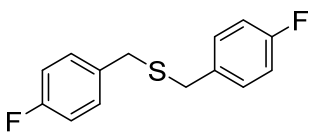
Bis(4-nitrobenzyl) sulfide (3m)[4]



Following general procedure II, using 4-nitrobenzyl bromide **1m** (162.0 mg, 0.750 mmol), anhydrous sodium thiosulfate (119.0 mg, 0.750 mmol), Li_2CO_3 (55.0 mg, 0.750 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 1:1, v/v) to afford **3m** (77.2 mg, 68 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 8.19 (d, *J* = 8.6 Hz, 4H), 7.44 (d, *J* = 8.6 Hz, 4H), 3.67 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 147.4, 145.3, 129.9, 124.1, 35.4; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{12}SN_2O_4$: 304.0518, found: 304.0521.

The spectral data of **3m** match those reported in the literature.[4]

Bis(4-Fluorobenzyl) sulfide (3n)[2]

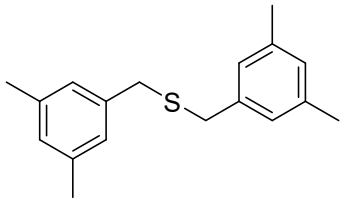


Following general procedure II, using 4-fluorobenzyl bromide **1n** (149.0 mg, 0.788

mmol), anhydrous sodium thiosulfate (124.0 mg, 0.788 mmol), Li_2CO_3 (58.0 mg, 0.788 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3n** (77.7 mg, 79 % yield) as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.23 (dd, J = 8.4, 5.4 Hz, 4H), 7.00 (t, J = 8.7 Hz, 4H), 3.57 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 162.0 (d, J_{C-F} = 245.4 Hz), 133.7 (d, J_{C-F} = 3.0 Hz), 130.6 (d, J_{C-F} = 8.0 Hz), 115.5 (d, J_{C-F} = 21.4 Hz), 34.9; ^{19}F NMR (376 MHz, $CDCl_3$) : δ -115.4 (s, C–F); HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{12}SF_2$: 250.0628, found: 250.0636.

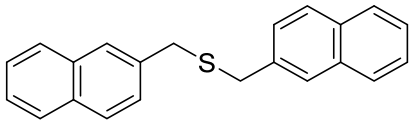
The spectral data of **3n** match those reported in the literature.[2]

Bis(3,5-dimethylbenzyl) sulfide (3o)



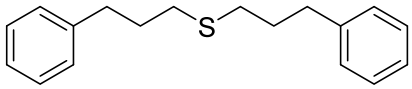
Following general procedure II, using 3,5-dimethylbenzyl bromide **1o** (182.0 mg, 0.914 mmol), anhydrous sodium thiosulfate (145.0 mg, 0.914 mmol), Li_2CO_3 (68.0 mg, 0.914 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3o** (94.2 mg, 76 % yield) as a white solid; m.p. 42-43 °C; IR (KBr) : ν = 2906, 2857, 1602, 1463, 1415 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) : δ 6.96 (s, 4H), 6.94 (s, 2H), 3.62 (s, 4H), 2.37 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 138.1, 138.0, 128.7, 126.9, 35.9, 21.3; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{18}H_{22}S$: 270.1442, found: 270.1436.

Bis(2-naphthylmethyl) sulfide (3p)



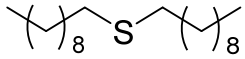
Following general procedure II, using 2-(bromomethyl)naphthalene **1p** (138.0 mg, 0.624 mmol), anhydrous sodium thiosulfate (99.0 mg, 0.624 mmol), Li_2CO_3 (46.0 mg, 0.624 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3p** (79.6 mg, 83 % yield) as a white solid; m.p. 121-122 °C; IR (KBr) : ν = 3050, 2915, 1598, 1505, 1357 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 6H), 7.65 (s, 2H), 7.51 – 7.45 (m, 6H), 3.76 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) 135.5, 133.4, 132.7, 128.5, 127.84, 127.76, 127.76, 127.4, 126.3, 125.9, 35.9; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{22}H_{18}S$: 314.1129, found: 314.1134.

Bis(3-phenylpropyl) sulfide (3q)[5]



Following general procedure III, using the alkyl Bunte salt (**2q**) synthesized from **1q** by procedure I (128.0 mg, 0.502 mmol), 1-bromo-3-phenylpropane **1q** (200.0 mg, 1.00 mmol), Li_2CO_3 (37.0 mg, 0.502 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford **3q** (45.4 mg, 34 % yield) as a white solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.32 (m, 4H), 7.28 – 7.19 (m, 6H), 2.75 (t, J = 7.6 Hz, 4H), 2.56 (t, J = 7.5 Hz, 4H), 1.93 (p, J = 7.5 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) 141.5, 128.4, 128.3, 125.9, 34.8, 31.3, 31.1. HRMS (MALDI-TOF; Matrix: Dihydroxybenzoic acid) m/z : $[M+H]^+$ calcd. for $C_{18}H_{23}S$: 271.1515, found: 271.1518. The spectral data of **3q** match those reported in the literature.[5]

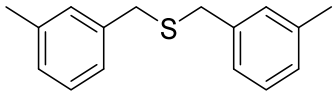
Bis(n-decyl) sulfide (3r)[6]



Following general procedure III, using the alkyl Bunte salt (**2r**) synthesized from **1r** by procedure I (62.5 mg, 0.226 mmol), 1-bromodecane **1r** (100.0 mg, 0.452 mmol), Li_2CO_3 (17.0 mg, 0.226 mmol). The crude product was then purified by column chromatography (hexane) to afford **3r** (25.6 mg, 35 % yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.49 (t, *J* = 7.4 Hz, 4H), 1.63 – 1.52 (m, 4H), 1.40 – 1.21 (m, 28H), 0.87 (t, *J* = 6.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) 32.2, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.0, 22.7, 14.1. HRMS (EI) m/z: $[M]^+$ calcd for $C_{20}H_{42}S$ 314.3002; found 314.2994.

The spectral data of **3r** match those reported in the literature.[6]

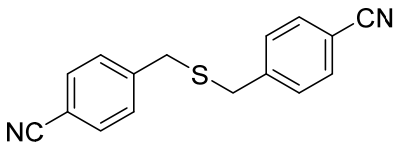
Bis(3-methylbenzyl) sulfide (3s)[7]



Following general procedure **E**, using 3-methylbenzyl bromide (100.0 mg, 0.54 mmol), anhydrous sodium thiosulfate (85.43 mg, 0.54 mmol), Li_2CO_3 (39.90 mg, 0.54 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford Bis(3-methylbenzyl) sulfide (54.0 mg, 83% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.5 Hz, 2H), 7.13 – 7.05 (m, 6H), 3.59 (s, 4H), 2.35 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.23, 138.21, 129.88, 128.45, 127.86, 126.18, 35.80, 21.51. HRMS (EI) m/z: $[M]^+$ calcd for $C_{16}H_{18}S$ 242.1124; found 242.1126.

The spectral data of **3r** match those reported in the literature.[7]

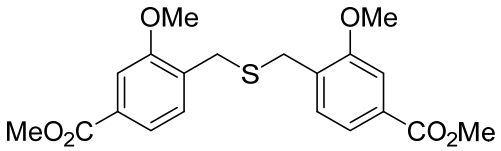
4,4'-(Thiobis(methylene))dibenzonitrile (3t)[5]



Following general procedure II, using 4-(bromomethyl)benzonitrile **1t** (165.0 mg, 0.839 mmol), anhydrous sodium thiosulfate (133.0 mg, 0.839 mmol), Li_2CO_3 (62.0 mg, 0.839 mmol). The crude product was then purified by column chromatography (hexane/EtOAc, 1:3, v/v) to afford **3t** (74.4 mg, 67% yield) as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ; δ 7.63 (d, *J* = 8.0 Hz, 4H), 7.38 (d, *J* = 8.0 Hz, 4H), 3.61 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.2, 132.5, 129.7, 118.7, 111.3, 35.5; HRMS (EI) m/z: [M^+] calcd for $C_{16}H_{12}N_2S$: 264.0716, found: 264.0721.

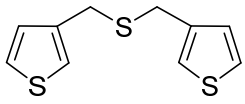
The spectral data of **3t** match those reported in the literature.[5]

Dimethyl 4,4'-(thiobis(methylene)) bis(3-methoxybenzoate) (3v)



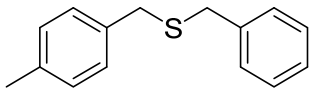
Following general procedure II, using methyl 4-(bromomethyl)-3-methoxybenzoate **1v** (194.3 mg, 0.750 mmol), anhydrous sodium thiosulfate (118.6 mg, 0.750 mmol), Li_2CO_3 (55.7 mg, 0.750 mmol). The crude product was then purified by column chromatography (hexane/EtOAc, 10:1, v/v) to afford **3v** (134.4 mg, 91% yield) as a white solid, m.p. 87-88 °C; 'H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 7.7 Hz, 2H), 7.50 (s, 2H), 7.32 (dd, *J* = 7.7 Hz, 2H), 3.90 (dd, *J* = 7.6 Hz, 12H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.1, 157.3, 132.4, 130.2, 130.1, 122.0, 111.4, 55.8, 52.3, 30.4; IR (ATR): v = 3070, 2949, 1725, 1714, 1606, 1580, 1504, 1288, 1273, 1228, 1216, 1192 cm^{-1}; HRMS (EI) m/z: [$M]^+$ calcd for $C_{20}H_{22}O_6S$: 390.1126, found:390.1132.

Bis(thiophen-3-ylmethyl) sulfane (3y)



Following general procedure II, using 3-(bromomethyl) thiophene **1y** (149.0 mg, 0.839 mmol), anhydrous sodium thiosulfate (133.0 mg, 0.839 mmol), Li_2CO_3 (62.0 mg, 0.839 mmol). The crude product was then purified by column chromatography (hexane, v/v) to afford **3y** (71.0 mg, 73% yield) as a yellow oil; 'H NMR (400 MHz, $CDCl_3$): δ 7.30-7.28 (m, 2H), 7.06-7.04 (m, 4H), 3.63 (s, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 128.3, 126.1, 122.6, 30.2; IR (ATR): v = 3100, 2915, 1464, 1412, 1405 cm^{-1}; HRMS (EI) m/z: $[M]^+$ calcd for $C_{10}H_{10}S_3$: 225.9935, found: 225.9939.

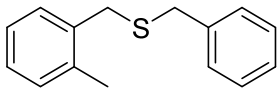
(4-Methylbenzyl)benzyl sulfide (4ba)[7]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-methylbenzyl bromide **1b** (40.9 mg, 0.221mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ba** (47.4 mg, 94 % yield) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.21 (m, 5H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 3.60 (s, 2H), 3.58 (s, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 136.7, 135.1, 129.3, 129.1, 129.0, 128.6, 127.0, 35.7, 35.4, 21.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{15}H_{17}S$: 229.1051, found: 229.1042.

The spectral data of **4ba** match those reported in the literature.[7]

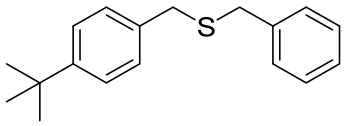
(2-Methylbenzyl)benzyl sulfide (4ca)[7]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-methylbenzyl bromide **1c** (40.9 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ca** (31.9 mg, 63 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 7.16 – 7.11 (m, 4H), 3.67 (s, 2H), 3.61 (s, 2H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 137.0, 135.9, 130.8, 129.8, 129.1, 128.6, 127.4, 127.1, 125.9, 36.3, 33.9, 19.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{15}H_{17}S$: 229.1051, found: 229.1042.

The spectral data of **4ca** match those reported in the literature.[7]

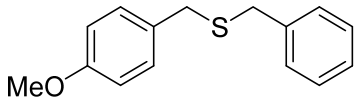
(4-*Tert*-butylbenzyl)benzyl sulfide (4da)[8]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-*tert*-butylbenzyl bromide **1d** (50.2 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4da** (35.9 mg, 60 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 6H), 7.28 – 7.19 (m, 3H), 3.62 (s, 2H), 3.59 (s, 2H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 138.5, 135.2, 129.1, 128.8, 128.6, 127.1, 125.5, 35.8, 35.3, 34.6, 31.5; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{18}H_{23}S$: 271.1521, found : 271.1512.

The spectral data of **4da** match those reported in the literature.[8]

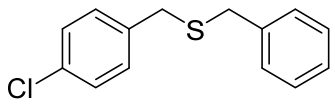
(4-Methoxybenzyl)benzyl sulfide (4ea)[9]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-methoxybenzyl bromide **1e** (44.4 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 3:1, v/v) to afford compound **4ea** (45.9 mg, 85 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.36 – 7.22 (m, 5H), 7.20 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 3.73 (s, 3H), 3.63 (s, 2H), 3.60 (s, 2H); ^{13}C NMR (100 MHz, $(CD_3)_2SO$) δ 158.2, 138.4, 130.0, 129.9, 128.8, 128.4, 126.8, 113.8, 55.0, 35.1, 34.6; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{15}H_{17}OS$: 245.1000, found: 245.0995.

The spectral data of **4ea** match those reported in the literature.[9]

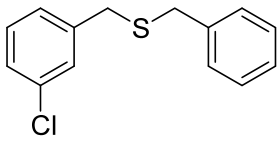
(4-Chlorobenzyl)benzyl sulfide (4fa)[3]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-chlorobenzyl bromide **1f** (45.4 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4fa** (49.5 mg, 90 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.16 (m, 9H), 3.58 (s, 2H), 3.55 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.0, 136.8, 132.9, 130.5, 129.1, 128.7, 128.6, 127.2, 35.7, 35.0; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{14}ClS$: 249.0505, found: 249.0503.

The spectral data of **4fa** match those reported in the literature.[3]

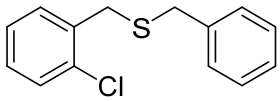
(3-Chlorobenzyl)benzyl sulfide (4ga)[7]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 3-chlorobenzyl bromide **1g** (45.4 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ga** (42.9 mg, 78 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.20 (m, 8H), 7.18 – 7.12 (m, 1H), 3.61 (s, 2H), 3.56 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 137.9, 134.4, 129.8, 129.2, 129.1, 128.7, 127.31, 127.28, 127.25, 35.8, 35.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{14}ClS$: 249.0505, found : 249.0505.

The spectral data of **4ga** match those reported in the literature.[7]

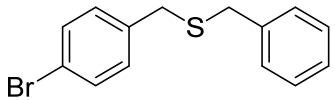
(2-Chlorobenzyl)benzyl sulfide (4ha)[10]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-chlorobenzyl bromide **1h** (45.4 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ha** (46.7 mg, 85 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.14 (m, 9H), 3.74 (s, 2H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.1, 136.1, 134.3, 130.9, 129.9, 129.1, 128.7, 128.5, 127.2, 126.9, 36.3, 33.5; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{14}ClS$: 249.0505, found: 249.0506.

The spectral data of **4ha** match those reported in the literature.[10]

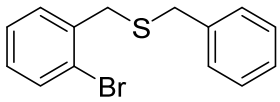
(4-Bromobenzyl)benzyl sulfide (4ia)[11]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-bromobenzyl bromide **1i** (55.2 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ia** (55.7 mg, 86 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.35 – 7.22 (m, 5H), 7.15 (d, *J* = 8.4 Hz, 2H), 3.59 (s, 2H), 3.54 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.9, 137.3, 131.7, 130.8, 129.1, 128.6, 127.2, 120.9, 35.7, 35.0; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{14}BrS$: 293.0000, found: 293.0005.

The spectral data of **4ia** match those reported in the literature.[11]

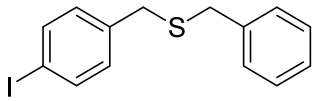
(2-Bromobenzyl)benzyl sulfide (4ja)[8]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-bromobenzyl bromide **1j** (55.2 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ja** (53.1 mg, 82 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.36 – 7.21 (m, 7H), 7.11 (td, *J* = 7.8, 1.8 Hz, 1H), 3.74 (s, 2H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.0, 137.7, 133.2, 130.8, 129.1, 128.7, 128.6, 127.5, 127.2, 124.8, 36.3, 36.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{14}BrS$: 293.0000, found: 292.9996.

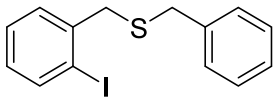
The spectral data of **4ja** match those reported in the literature.[8]

(4-Iodobenzyl)benzyl sulfide (4ka)



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-iodobenzyl bromide **1k** (65.6 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ka** (61.7 mg, 82 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 3027, 2918, 1739, 1482, 1230, 1007; ^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.35 – 7.20 (m, 5H), 7.10 (d, *J* = 8.2 Hz, 2H), 3.64 (s, 2H), 3.61 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.0, 137.9, 137.6, 131.1, 129.1, 128.6, 127.2, 92.4, 35.7, 35.1; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{13}IS$: 339.9783, found: 339.9786.

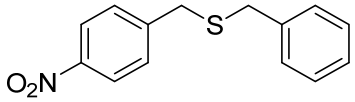
(2-Iodobenzyl)benzyl sulfide (4la)[12]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-iodobenzyl bromide **1l** (65.6 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4la** (57.1 mg, 76 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.0 Hz, 1H), 7.37 – 7.21 (m, 7H), 6.93 (dt, *J* = 8.0, 4.4 Hz, 1H), 3.72 (s, 2H), 3.70 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 140.0, 138.0, 130.1, 129.2, 128.8, 128.7, 128.3, 127.2, 101.0, 41.1, 36.3; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{14}H_{14}IS$: 340.9861, found: 340.9864.

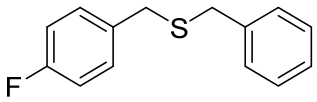
The spectral data of **4la** match those reported in the literature.[12]

(4-Nitrobenzyl)benzyl sulfide (4ma)



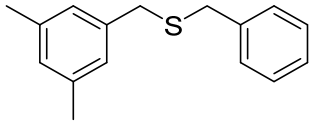
Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-nitrobenzyl bromide **1m** (47.7 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 3:1, v/v) to afford compound **4ma** (36.1 mg, 63 % yield) as a white solid; m.p. 54 - 55 °C; ATR-IR(cm^{-1}) : 2922, 2852, 1739, 1599, 1517, 1344; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.7 Hz, 2H), 7.42 (d, *J* = 8.7 Hz, 2H), 7.36 – 7.22 (m, 5H), 3.65 (s, 2H), 3.61 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 146.2, 137.5, 129.9, 129.1, 128.7, 127.4, 123.8, 35.9, 35.0; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{13}NO_2S$: 259.0667, found: 259.0669.

(4-Fluorobenzyl)benzyl sulfide (4na)



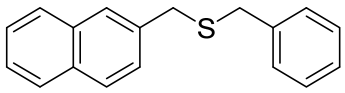
Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 4-fluorobenzyl bromide **1n** (41.8 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4na** (45.7 mg, 89 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 2920, 2852, 1740, 1601, 1507, 1220; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.21 (m, 7H), 7.00 (t, *J* = 8.7 Hz, 2H), 3.60 (s, 2H), 3.57 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0 (d, *J* = 244.1 Hz), 138.1, 133.9 (d, *J* = 2.9 Hz), 130.6 (d, *J* = 8.1 Hz), 129.1, 128.6, 127.2, 115.4 (d, *J* = 21.4 Hz), 35.7, 34.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -115.6; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{13}FS$: 232.0722, found: 232.0727.

(3,5-Dimethylbenzyl)benzyl sulfide (4oa)



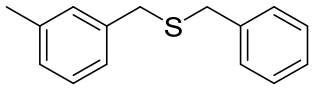
Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 3,5-dimethylbenzyl bromide **1o** (44.0 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4oa** (40.7 mg, 76 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 3027, 2918, 2852, 1739, 1604, 1453, 1229; ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 4H), 7.28 – 7.24 (m, 1H), 6.92 – 6.86 (m, 3H), 3.63 (s, 2H), 3.55 (s, 2H), 2.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 138.1, 138.0, 129.1, 128.8, 128.6, 127.1, 126.9, 35.9, 35.7, 21.4; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{16}H_{18}S$: 242.1129, found: 242.1124.

2-Naphthylmethyl benzyl sulfide (4pa)



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-(Bromomethyl)naphthalene **1p** (48.7 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4pa** (47.9 mg, 82 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 3028, 2970, 2922, 1739, 1366, 1217; ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.78 (m, 3H), 7.66 (s, 1H), 7.54 – 7.41 (m, 3H), 7.37 – 7.18 (m, 5H), 3.77 (s, 2H), 3.61 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.2, 135.6, 133.4, 132.7, 129.2, 128.6, 128.5, 127.8, 127.7, 127.6, 127.3, 127.1, 126.3, 125.9, 36.0, 35.6; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{18}H_{16}S$: 264.0973, found: 264.0970.

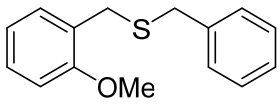
(3-Methylbenzyl)benzyl sulfide (4sa)[7]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 3-methylbenzyl bromide **1s** (40.9 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4sa** (38.7 mg, 77 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.16 (m, 6H), 7.13 – 7.03 (m, 3H), 3.62 (s, 2H), 3.58 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.3, 138.2, 138.1, 129.8, 129.1, 128.5, 128.4, 127.8, 127.0, 126.1, 35.8, 35.7, 21.5; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{15}H_{17}S$: 229.1051, found: 229.1045.

The spectral data of **4sa** match those reported in the literature.[7]

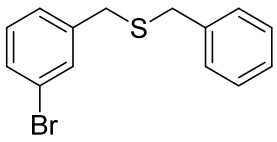
(2-Methoxybenzyl)benzyl sulfide (4za)[7]



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 2-methoxybenzyl bromide **1z** (44.4 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 3:1, v/v) to afford compound **4za** (45.4 mg, 84 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 7.39 – 7.14 (m, 7H), 6.98 (d, J = 8.1 Hz, 1H), 6.89 (t, J = 7.4 Hz, 1H), 3.77 (s, 3H), 3.68 (s, 2H), 3.62 (s, 2H); ^{13}C NMR (100 MHz, $(CD_3)_2SO$) δ 156.9, 138.5, 130.0, 128.8, 128.3, 128.3, 126.8, 126.3, 120.2, 111.1, 55.4, 35.5, 29.6; HRMS (ESI) m/z: $[M+H]^+$ calcd. for $C_{15}H_{17}OS$: 245.1000, found: 245.0996.

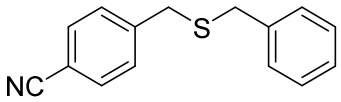
The spectral data of **4za** match those reported in the literature.[7]

(3-Bromobenzyl)benzyl sulfide (4xa)



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), 3-bromobenzyl bromide **1x** (55.2 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4xa** (48.6 mg, 75 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 3028, 2920, 1739, 1567, 1453, 1231, 1070; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.41 (m, 1H), 7.39 – 7.15 (m, 8H), 3.61 (s, 2H), 3.55 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 137.9, 132.1, 130.2, 130.1, 129.1, 128.7, 127.7, 127.3, 122.6, 35.8, 35.1; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{14}H_{13}BrS$: 291.9921, found: 291.9924.

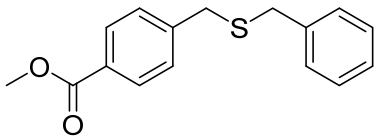
(4-Cyanobenzyl)benzyl sulfide (4ta)[13]



Following the general procedure III, using the alkyl Bunte salt **2a** (350.0 mg, 1.547 mmol), 4-cyanobenzyl bromide **1t** (101.2 mg, 0.516 mmol), Li_2CO_3 (38.1 mg, 0.516 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 2:1, v/v) to afford compound **4ta** (89.7 mg, 73 % yield) as a colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.2 Hz, 2H), 7.34 – 7.23 (m, 5H), 3.60 (s, 2H), 3.59 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 137.5, 132.2, 129.7, 129.0, 128.6, 127.3, 118.8, 110.8, 35.8, 35.3; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{15}H_{14}NS$: 240.0847, found: 240.0842.

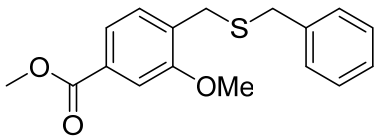
The spectral data of **4ta** match those reported in the literature.[13]

Methyl 4-((benzylthio)methyl)benzoate (4ua)



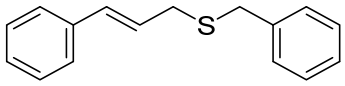
Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), Methyl 4-(bromomethyl)benzoate **1u** (43.6 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 1:1, v/v) to afford compound **4ua** (36.7 mg, 61 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 2920, 2851, 1721, 1610, 1278, 1107; ^1H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.32 – 7.21 (m, 5H), 3.92 (s, 3H), 3.62 (s, 2H), 3.59 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 143.7, 137.8, 129.9, 129.09, 129.07, 128.9, 128.6, 127.2, 52.2, 35.7, 35.3; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{16}H_{16}O_2S$: 272.0871, found: 272.0878.

Methyl 4-((benzylthio)methyl)-3-methoxybenzoate (4va)



Following the general procedure III, using the alkyl Bunte salt **2a** (150.0 mg, 0.663 mmol), methyl 4-(bromomethyl)-3-methoxybenzoate **1v** (50.2 mg, 0.221 mmol), Li_2CO_3 (16.3 mg, 0.221 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 1:1, v/v) to afford compound **4va** (55.5 mg, 83 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 2950, 2929, 1717, 1582, 1289, 1230, 1102; ^1H NMR (400 MHz, $(CD_3)_2SO$) δ 7.53 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.47 (d, *J* = 1.5 Hz, 1H), 7.36 (d, *J* = 7.7 Hz, 1H), 7.34 – 7.20 (m, 5H), 3.85 (s, 3H), 3.84 (s, 3H), 3.70 (s, 2H), 3.66 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 157.3, 138.2, 132.4, 130.2, 130.1, 129.1, 128.5, 127.1, 121.9, 111.4, 55.7, 52.3, 36.3, 29.9; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{17}H_{18}O_3S$: 302.0977, found: 302.0984.

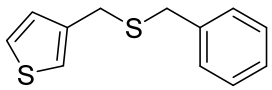
Cinnamyl benzyl sulfide (4wa)[14]



Following the general procedure III, using the alkyl Bunte salt **2a** (200.0 mg, 0.884 mmol), cinnamyl bromide **1w** (58.0 mg, 0.295 mmol), Li_2CO_3 (21.8 mg, 0.295 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 4:1, v/v) to afford compound **4wa** (60.5 mg, 85 % yield) as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.36 (m, 2H), 7.36 – 7.30 (m, 6H), 7.25 – 7.22 (m, 2H), 6.40 (d, *J* = 15.7 Hz, 1H), 6.18 (dt, *J* = 15.7, 7.3 Hz, 1H), 3.70 (s, 2H), 3.21 (dd, *J* = 7.3, 1.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 136.8, 132.6, 129.1, 128.7, 128.6, 127.7, 127.1, 126.4, 125.9, 35.1, 33.8; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{16}H_{17}S$: 241.1051, found: 241.1050.

The spectral data of **4wa** match those reported in the literature.[14]

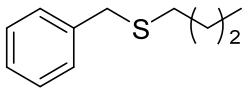
3-((Benzylthio)methyl)thiophene (4ya)



4ya, 89%

Following the general procedure III, using the alkyl Bunte salt **2a** (192.0 mg, 0.849 mmol), 3-(bromomethyl) thiophene **1y** (50.0 mg, 0.282 mmol), Li_2CO_3 (21.0 mg, 0.282 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 10:1, v/v) to afford compound **4ya** (56.0 mg, 89% yield) as a yellow oil; 'H NMR (400 MHz, $CDCl_3$): δ 7.34-7.23 (m, 6H), 7.07-7.05 (m, 2H), 3.62-3.60 (m, 4H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.7, 138.2, 129.1, 128.6, 128.4, 127.1, 126.1, 122.6, 35.7, 30.1; IR (ATR): v = 3027, 2917, 1494, 1451, 1421 cm^{-1}; HRMS (EI) m/z: $[M]^+$ calcd for $C_{12}H_{12}S_2$: 220.0378, found: 220.0375

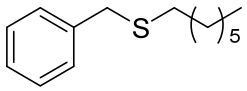
Benzyl butyl sulfide (4ab)[15]



Following the general procedure III, using the alkyl Bunte salt **2b** (500.0 mg, 2.600 mmol), benzyl bromide **1a** (148.3 mg, 0.867 mmol), Li_2CO_3 (64.3 mg, 0.867 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4ab** (82.9 mg, 53 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.28 (m, 4H), 7.24 – 7.21 (m, 1H), 3.70 (s, 2H), 2.41 (t, *J* = 7.4 Hz, 2H), 1.54 (quin, *J* = 7.4 Hz, 2H), 1.37 (sex, *J* = 7.4 Hz, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 128.9, 128.5, 126.9, 36.4, 31.4, 31.1, 22.1, 13.8; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{11}H_{17}S$: 181.1051, found: 181.1050.

The spectral data of **4ab** match those reported in the literature.[15]

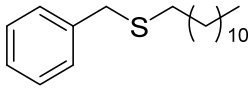
Benzyl heptyl sulfide (4ac)[16]



Following the general procedure III, using the alkyl Bunte salt **2c** (300.0 mg, 1.280 mmol), benzyl bromide **1a** (73.0 mg, 0.427 mmol), Li_2CO_3 (31.6 mg, 0.427 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4ac** (58.2 mg, 61 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.28 (m, 4H), 7.27 – 7.21 (m, 1H), 3.71 (s, 2H), 2.41 (t, *J* = 7.4 Hz, 2H), 1.55 (quin, *J* = 7.3 Hz, 2H), 1.37 – 1.21 (m, 8H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 128.9, 128.5, 126.9, 36.4, 31.8, 31.5, 29.3, 29.0, 28.9, 22.7, 14.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{23}S$: 223.1521, found: 223.1519.

The spectral data of **4ac** match those reported in the literature.[16]

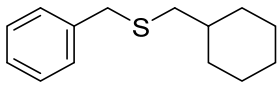
Benzyl dodecyl sulfide (4ad)[17]



Following the general procedure III, using the alkyl Bunte salt **2d** (450.0 mg, 1.479 mmol), benzyl bromide **1a** (84.3 mg, 0.493 mmol), Li_2CO_3 (36.4 mg, 0.493 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4ad** (98.2 mg, 68 % yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.29 (m, 4H), 7.26 – 7.21 (m, 1H), 3.70 (s, 2H), 2.40 (t, *J* = 7.4 Hz, 2H), 1.55 (quin, *J* = 7.4 Hz, 2H), 1.37 – 1.24 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 128.9, 128.5, 126.9, 36.3, 32.0, 31.4, 29.8, 29.8, 29.7, 29.6, 29.5, 29.3, 29.3, 29.0, 22.8, 14.3; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{19}H_{33}S$: 293.2303, found : 293.2308.

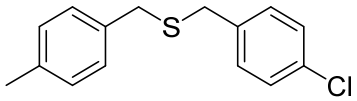
The spectral data of **4ad** match those reported in the literature.[17]

Benzyl(cyclohexylmethyl) sulfide (4ae)



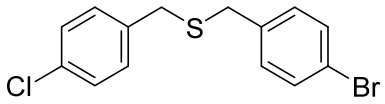
Following the general procedure III, using the alkyl Bunte salt **2e** (300.0 mg, 1.291 mmol), benzyl bromide **1a** (73.6 mg, 0.430 mmol), Li_2CO_3 (31.8 mg, 0.430 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4ae** (74.9 mg, 79 % yield) as a colorless oil. ATR-IR(cm^{-1}) : 2919, 2848, 1510, 1451, 1345, 1105; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.20 (m, 5H), 3.69 (s, 2H), 2.31 (d, *J* = 6.8 Hz, 2H), 1.84 – 1.76 (m, 2H), 1.72 – 1.60 (m, 3H), 1.48 – 1.37 (m, 1H), 1.28 – 1.05 (m, 3H), 0.96 – 0.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 128.9, 128.4, 126.8, 39.0, 37.6, 36.9, 32.9, 26.5, 26.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{21}S$: 221.1364, found : 221.1358.

(4-Methylbenzyl) (4-chlorobenzyl) sulfide (4bf)



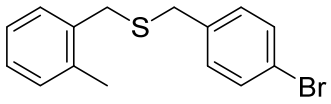
Following the general procedure III, using the alkyl Bunte salt **2f** (350.0 mg, 1.343 mmol), 4-methylbenzyl bromide **1b** (82.9 mg, 0.448 mmol), Li_2CO_3 (33.1 mg, 0.448 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4bf** (108.3 mg, 92 % yield) as a white solid; m.p. 56 - 57 °C; ATR-IR(cm^{-1}) : 2921, 2851, 1738, 1488, 1415, 1233, 1090; ^1H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.5 Hz, 2H), δ 7.20 (d, *J* = 8.5 Hz, 2H), δ 7.15 (d, *J* = 8.2 Hz, 2H), δ 7.11 (d, *J* = 8.2 Hz, 2H), 3.56 (s, 2H), 3.55 (s, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.9, 136.8, 134.8, 132.7, 130.4, 129.3, 129.0, 128.7, 35.4, 34.9, 21.2; HRMS (EI) m/z : $[M]^+$ calcd. for $C_{15}H_{15}ClS$: 262.0583, found: 262.0584.

(4-Chlorobenzyl) (4-bromobenzyl) sulfide (4fg)



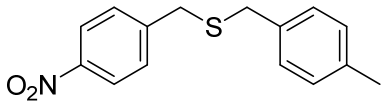
Following the general procedure III, using the alkyl Bunte salt **2g** (275.0 mg, 0.901 mmol), 4-chlorobenzyl bromide **1f** (61.6 mg, 0.300 mmol), Li_2CO_3 (22.2 mg, 0.300 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4fg** (71.5 mg, 73 % yield) as a white solid; m.p. 40 - 41 °C; ATR-IR(cm^{-1}) : 2917, 2848, 1486, 1399, 1086, 1011; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 3.54 (s, 2H), 3.53 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.0, 136.5, 132.9, 131.7, 130.8, 130.4, 128.8, 121.0, 35.0, 34.9; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{13}ClBrS$: 326.9610, found: 326.9622.

(2-Methylbenzyl) (4-bromobenzyl) sulfide (4cg)



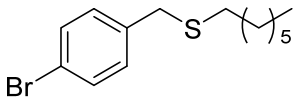
Following the general procedure III, using the alkyl Bunte salt **2g** (293.0 mg, 0.960 mmol), 2-methylbenzyl bromide **1c** (59.2 mg, 0.320 mmol), Li_2CO_3 (23.6 mg, 0.320 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4cg** (83.7 mg, 85 % yield) as a colorless oil; ATR-IR(cm^{-1}) : 3013, 2917, 2849, 1486, 1069, 1010; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 7.18 – 7.12 (m, 5H), 3.60 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 136.9, 135.5, 131.7, 130.8, 130.7, 129.8, 127.5, 125.9, 120.9, 35.6, 33.9, 19.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{15}H_{16}BrS$: 307.0156, found : 307.0143.

(4-Nitrolbenzyl) (4-methylbenzyl) sulfide (4mh)



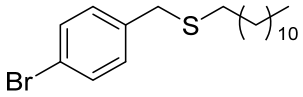
Following the general procedure III, using the alkyl Bunte salt **2h** (252.0 mg, 1.050 mmol), 4-nitrobenzyl bromide **1m** (75.6 mg, 0.350 mmol), Li_2CO_3 (25.9 mg, 0.350 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 3:1, v/v) to afford compound **4mh** (51.7 mg, 54 % yield) as a white solid; m.p. 79 - 80 °C; ATR-IR(cm^{-1}) : 2916, 2851, 1510, 1340, 1106; ^1H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.6 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.16 – 7.10 (m, 4H), 3.64 (s, 2H), 3.58 (s, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 146.3, 137.1, 134.3, 129.9, 129.4, 129.0, 123.8, 35.6, 35.0, 21.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{15}H_{16}NO_2S$: 274.0902, found : 274.0905.

(4-Bromobenzyl) heptyl sulfide (4ic)



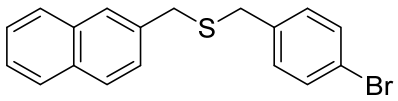
Following the general procedure III, using the alkyl Bunte salt **2c** (281.0 mg, 1.200 mmol), 4-bromobenzyl bromide **1i** (99.9 mg, 0.400 mmol), Li_2CO_3 (29.5 mg, 0.400 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4ic** (96.4 mg, 80 % yield) as a colorless oil; ATR-IR(cm^{-1}) : 2925, 2854, 1486, 1071, 1011; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 3.64 (s, 2H), 2.39 (t, *J* = 7.3 Hz, 2H), 1.53 (quin, *J* = 7.5 Hz, 2H), 1.36 – 1.22 (m, 8H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.9, 131.6, 130.6, 120.8, 35.8, 31.8, 31.5, 29.3, 29.0, 28.9, 22.7, 14.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{14}H_{22}BrS$: 301.0626, found : 301.0623.

(4-Bromobenzyl) dodecyl sulfide (4id)



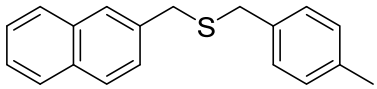
Following the general procedure III, using the alkyl Bunte salt **2d** (365.0 mg, 1.200 mmol), 4-bromobenzyl bromide **1i** (100.0 mg, 0.400 mmol), Li_2CO_3 (29.6 mg, 0.400 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 8:1, v/v) to afford compound **4id** (126.2 mg, 85 % yield) as a colorless oil; ATR-IR(cm^{-1}) : 2922, 2852, 1485, 1071, 1012; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 3.64 (s, 2H), 2.38 (t, *J* = 7.4 Hz, 2H), 1.53 (quin, *J* = 7.5 Hz, 2H), 1.36 – 1.22 (m, 18H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.9, 131.6, 130.6, 120.8, 35.8, 32.0, 31.5, 29.8, 29.8, 29.7, 29.6, 29.5, 29.3, 29.2, 29.0, 22.8, 14.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{19}H_{32}BrS$: 371.1408, found : 371.1416.

(2-Naphthylmethyl)(4-bromobenzyl) sulfide (4pg)



Following the general procedure III, using the alkyl Bunte salt **2g** (275.0 mg, 0.901 mmol), 2-(Bromomethyl)naphthalene **1p** (66.4 mg, 0.300 mmol), Li_2CO_3 (22.2 mg, 0.300 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4pg** (74.1 mg, 72 % yield) as a white solid; m.p. 76 - 77 °C; ATR-IR(cm^{-1}) : 2918, 2851, 1509, 1342, 1106; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 3H), 7.62 (s, 1H), 7.52 – 7.45 (m, 3H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 3.75 (s, 2H), 3.53 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.3, 135.2, 133.3, 132.7, 131.7, 130.8, 128.6, 127.8, 127.7, 127.6, 127.2, 126.4, 126.0, 120.9, 36.0, 34.9; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{18}H_{16}BrS$: 343.0156, found: 343.0169.

(2-Naphthylmethyl)(4-methylbenzyl) sulfide (4ph)



Following the general procedure III, using the alkyl Bunte salt **2h** (252.0 mg, 1.050 mmol), 2-(Bromomethyl)naphthalene **1p** (77.4 mg, 0.350 mmol), Li_2CO_3 (25.9 mg, 0.350 mmol). The crude product was then purified by column chromatography (hexane/CH_2Cl_2, 6:1, v/v) to afford compound **4ph** (51.7 mg, 53 % yield) as a white solid; m.p. 63 - 64 °C; ATR-IR(cm^{-1}) : 3051, 2914, 2850, 1509, 1208; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.77 (m, 3H), 7.66 (s, 1H), 7.51 – 7.44 (m, 3H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 3.76 (s, 2H), 3.58 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.8, 135.7, 135.1, 133.4, 132.7, 129.3, 129.1, 128.5, 127.8, 127.7, 127.6, 127.4, 126.3, 125.9, 35.9, 35.3, 21.2; HRMS (ESI) m/z : $[M+H]^+$ calcd. for $C_{19}H_{19}S$: 279.1208, found : 279.1200.

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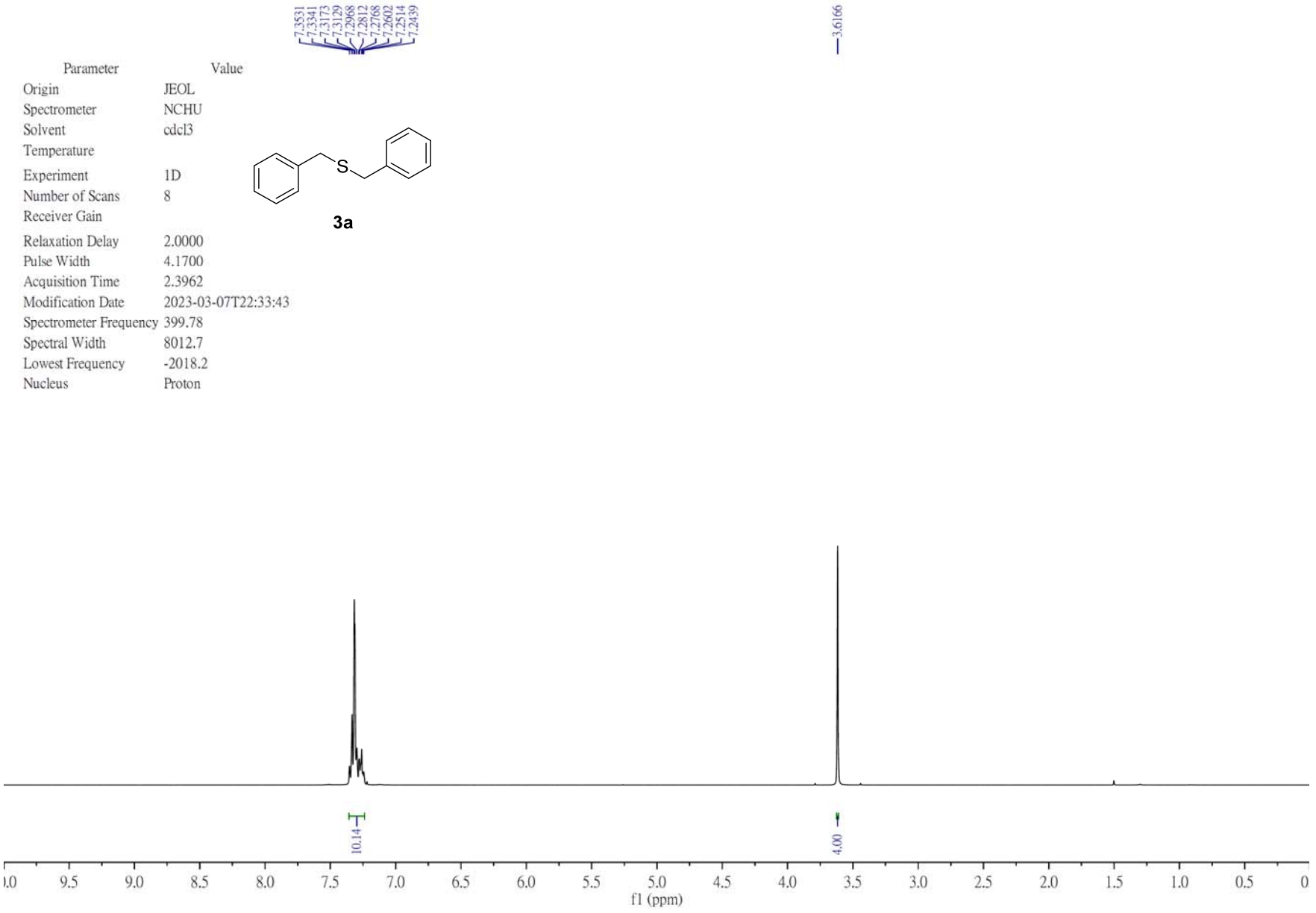
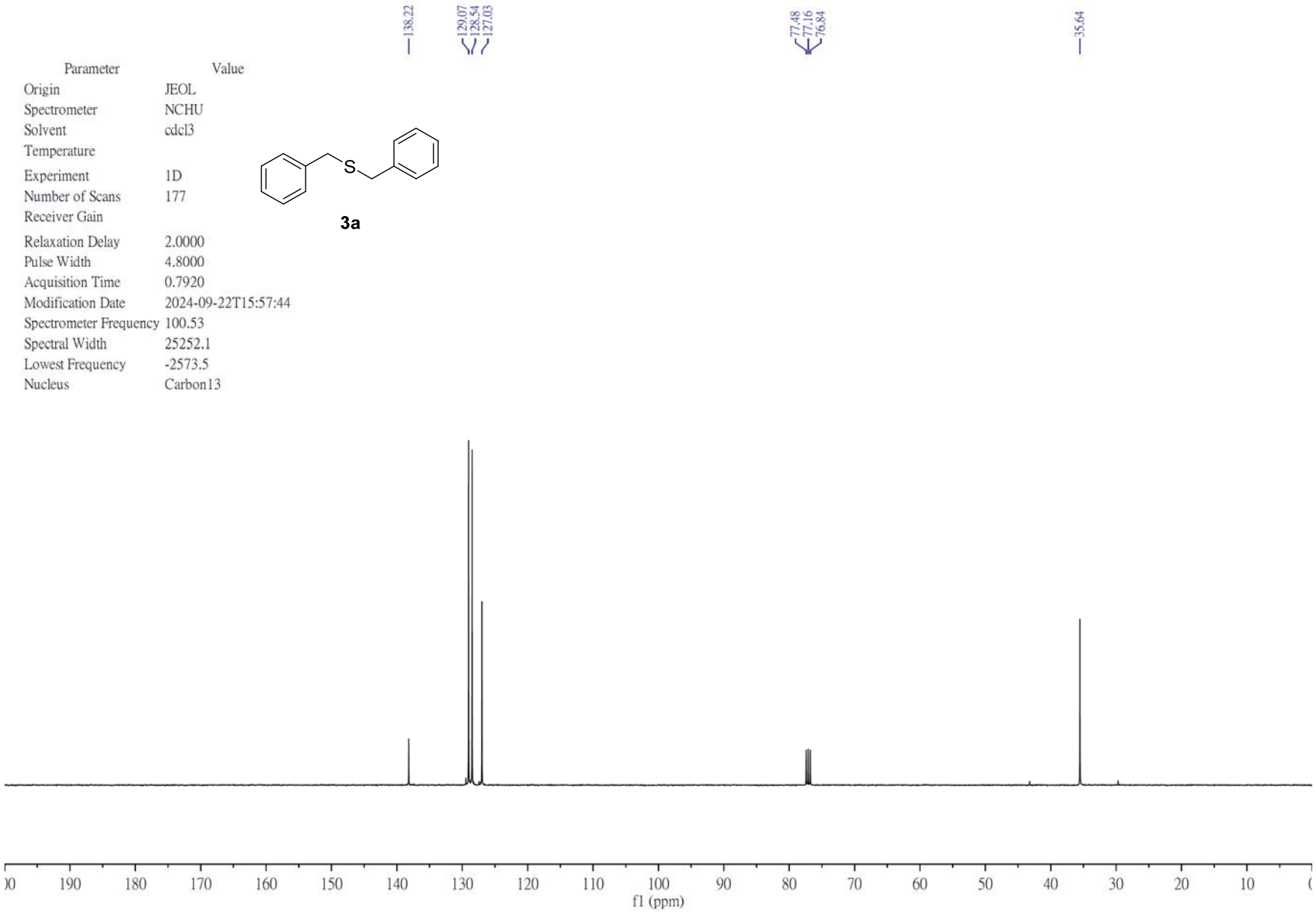
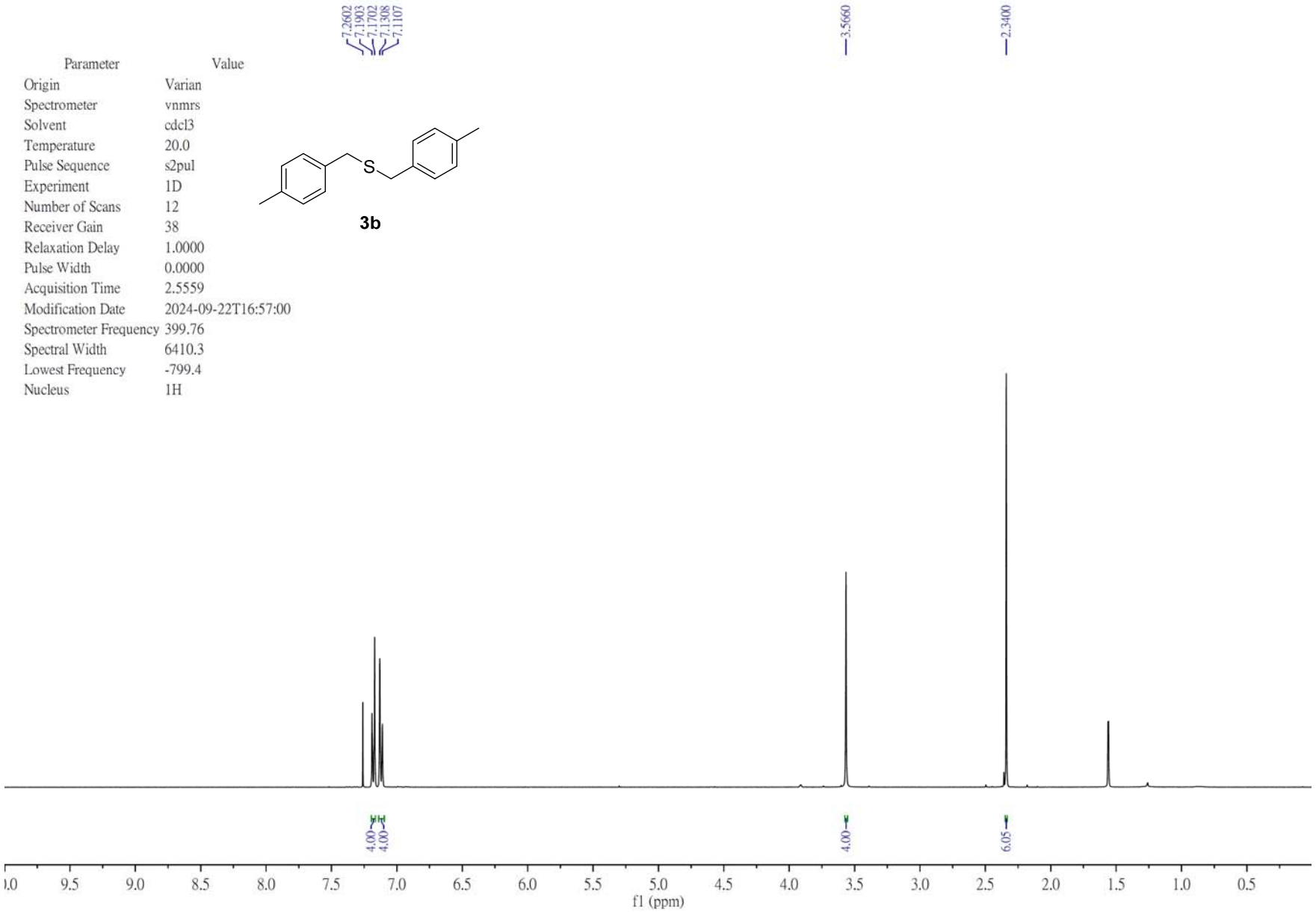


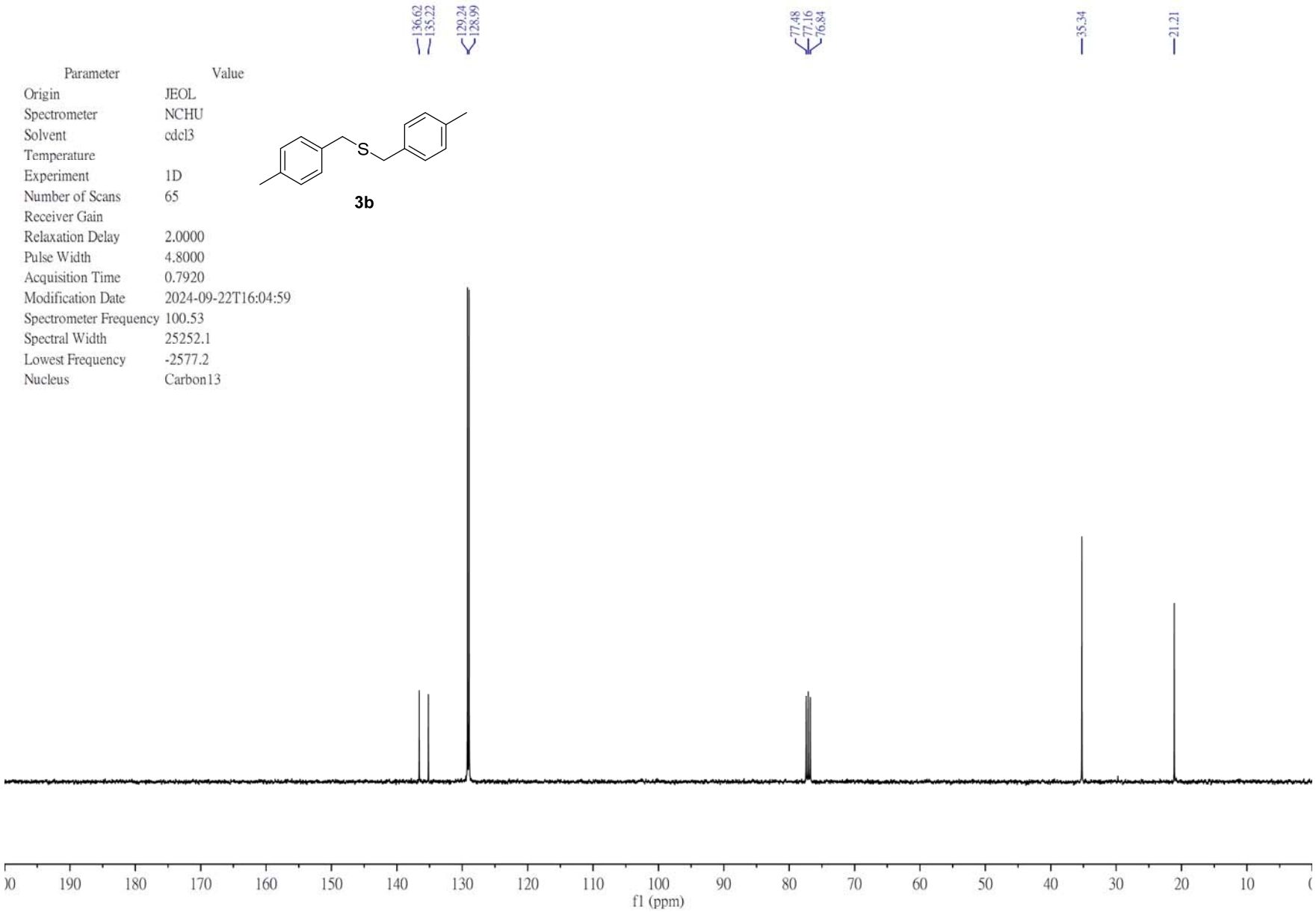
3a ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



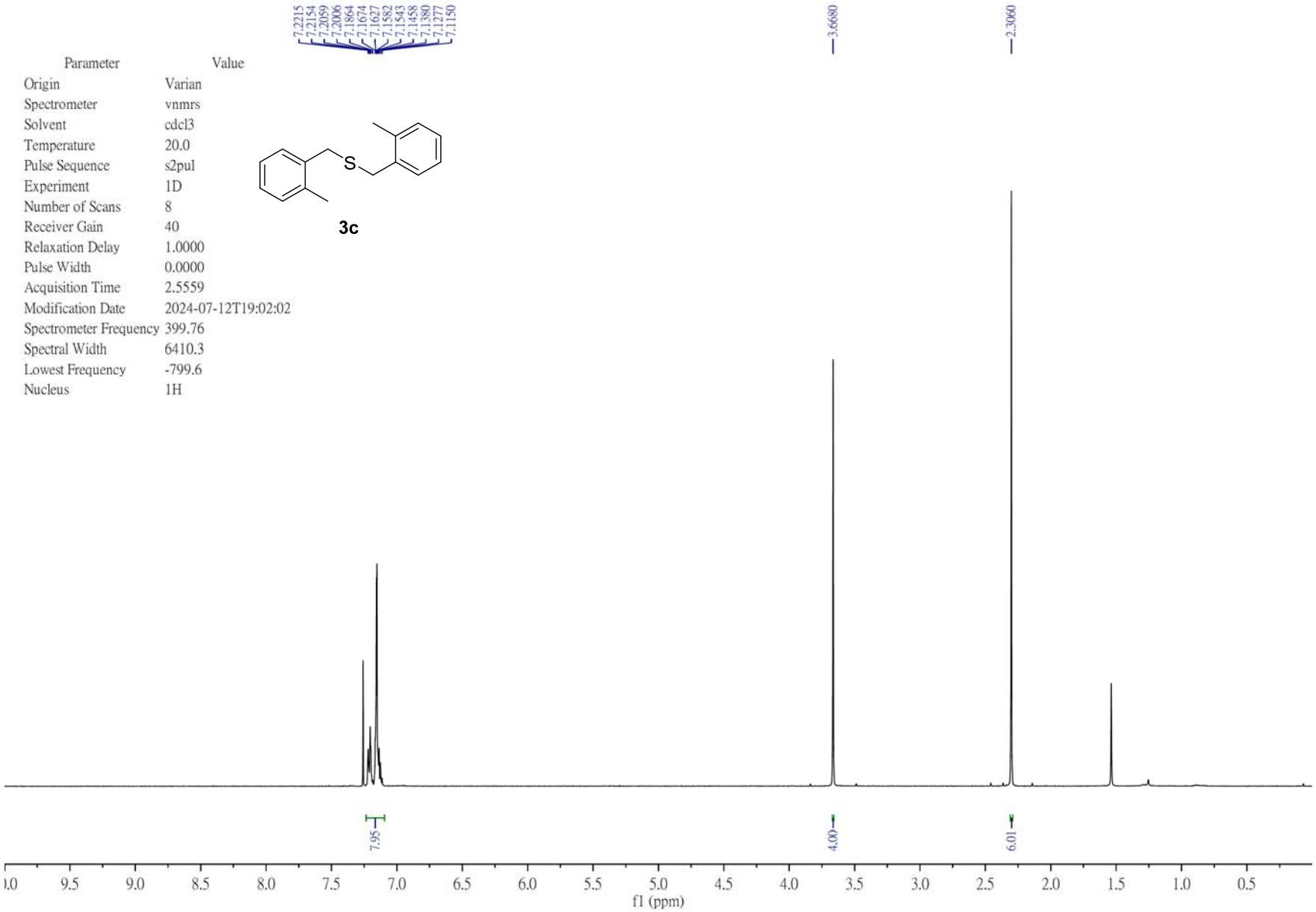
3a $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



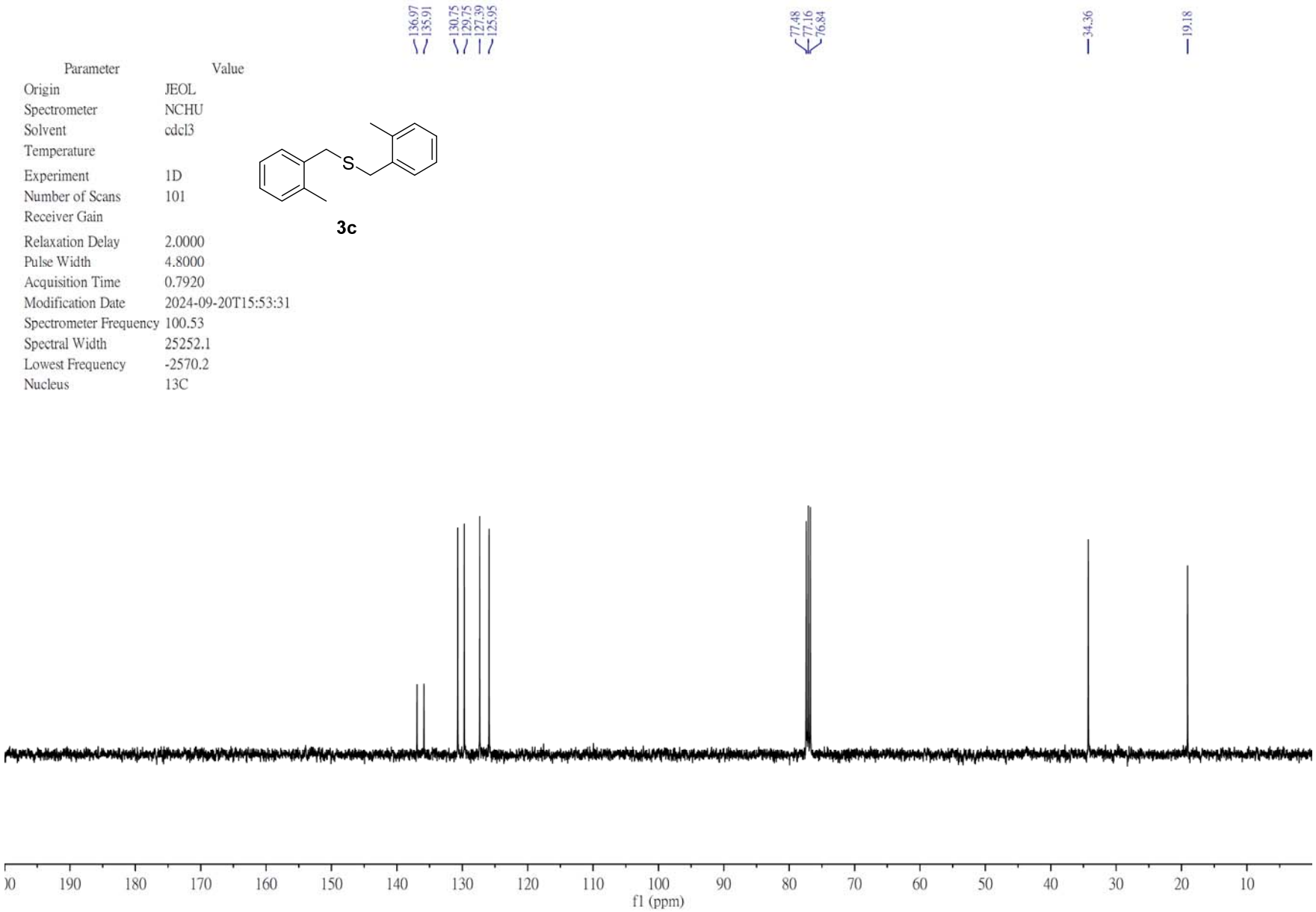
3b ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



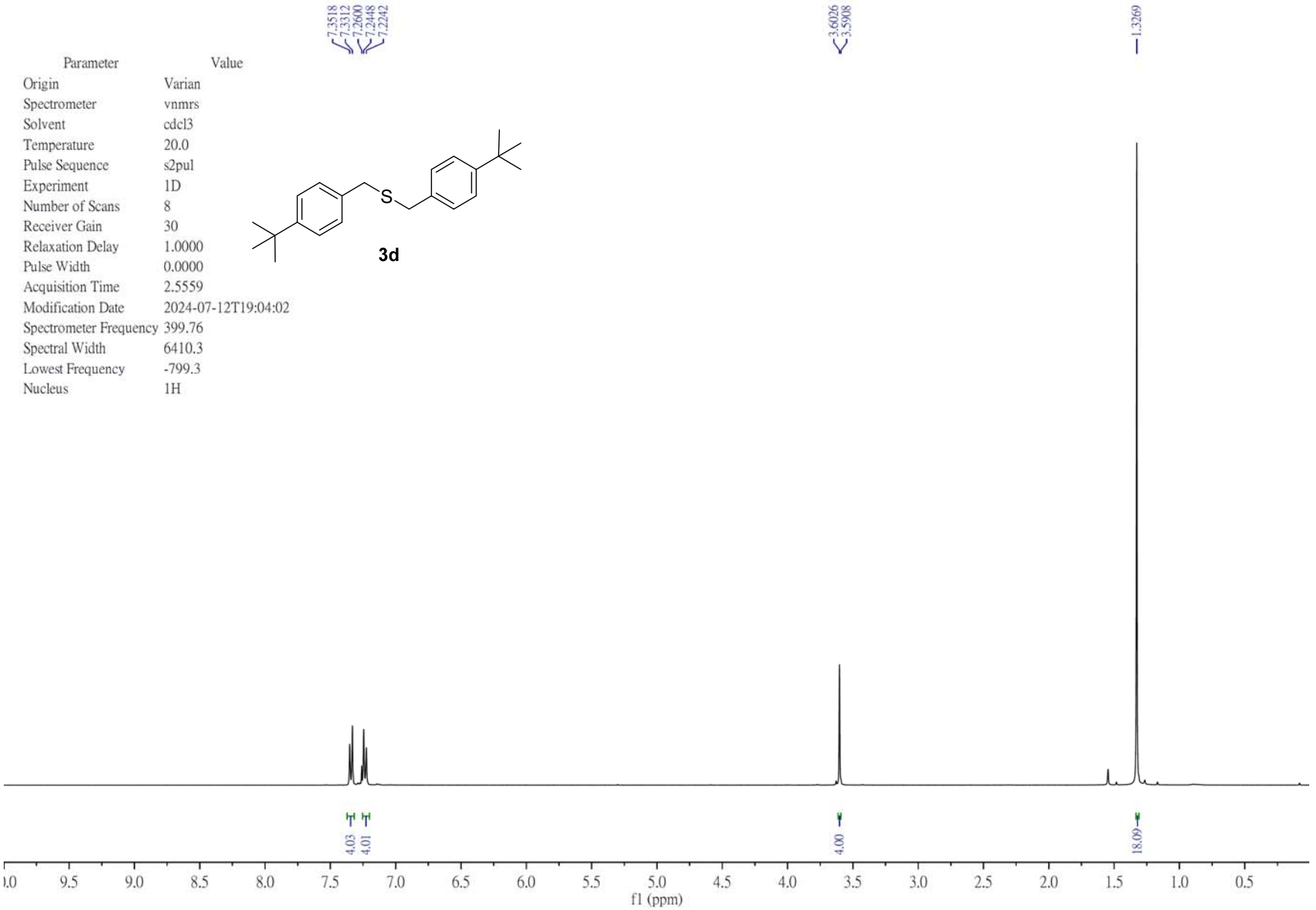
3b $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



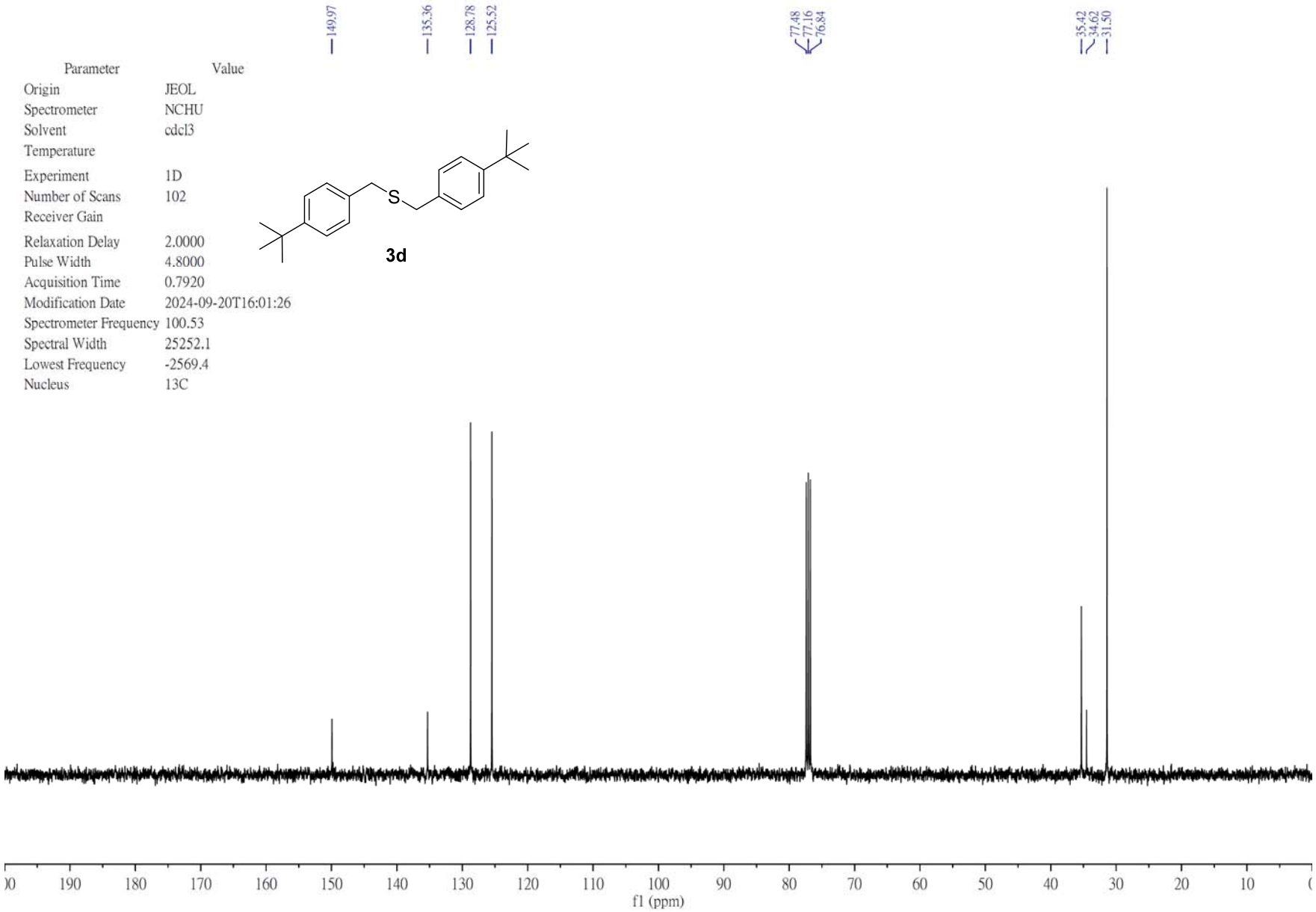
3c ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



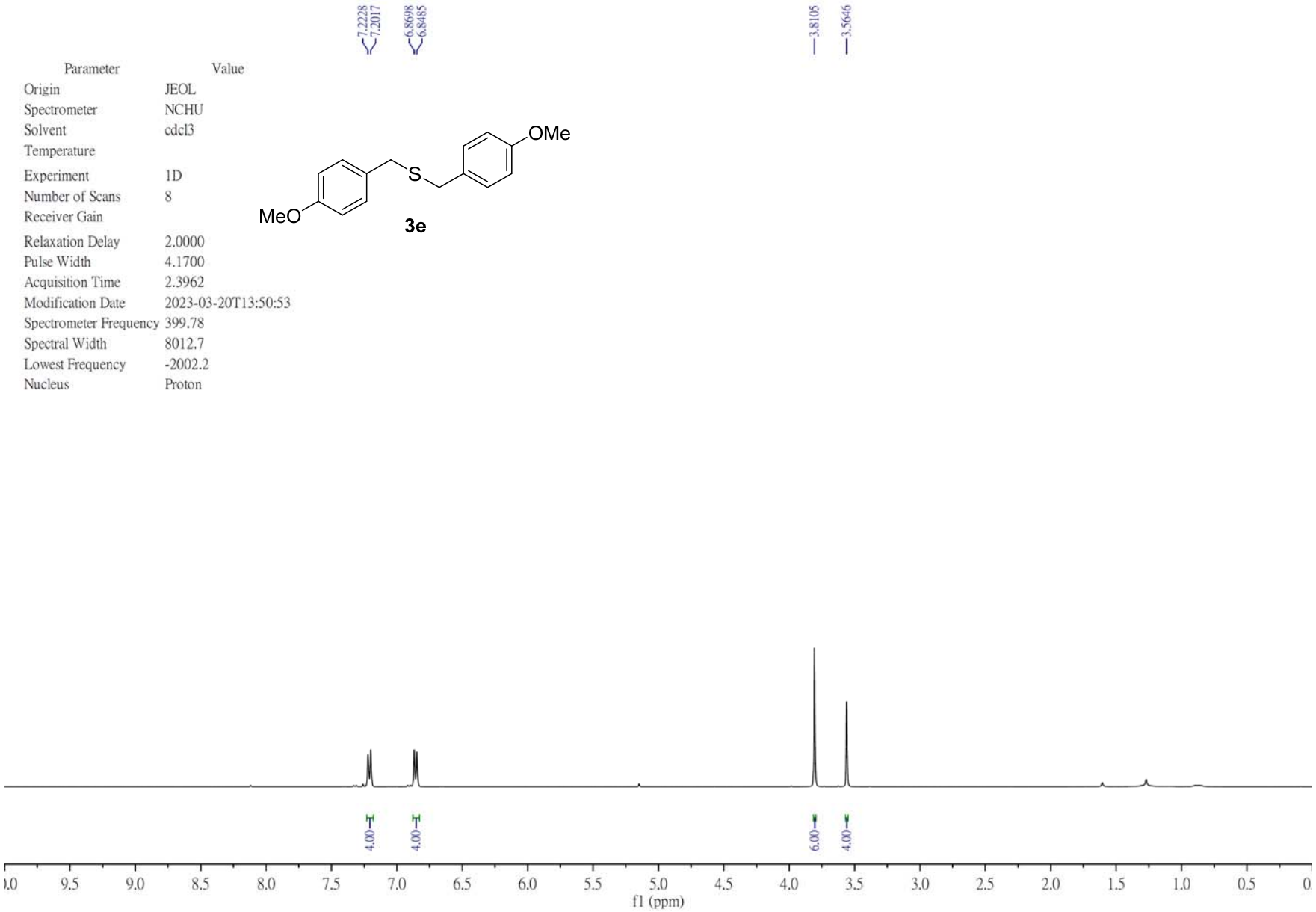
3c $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



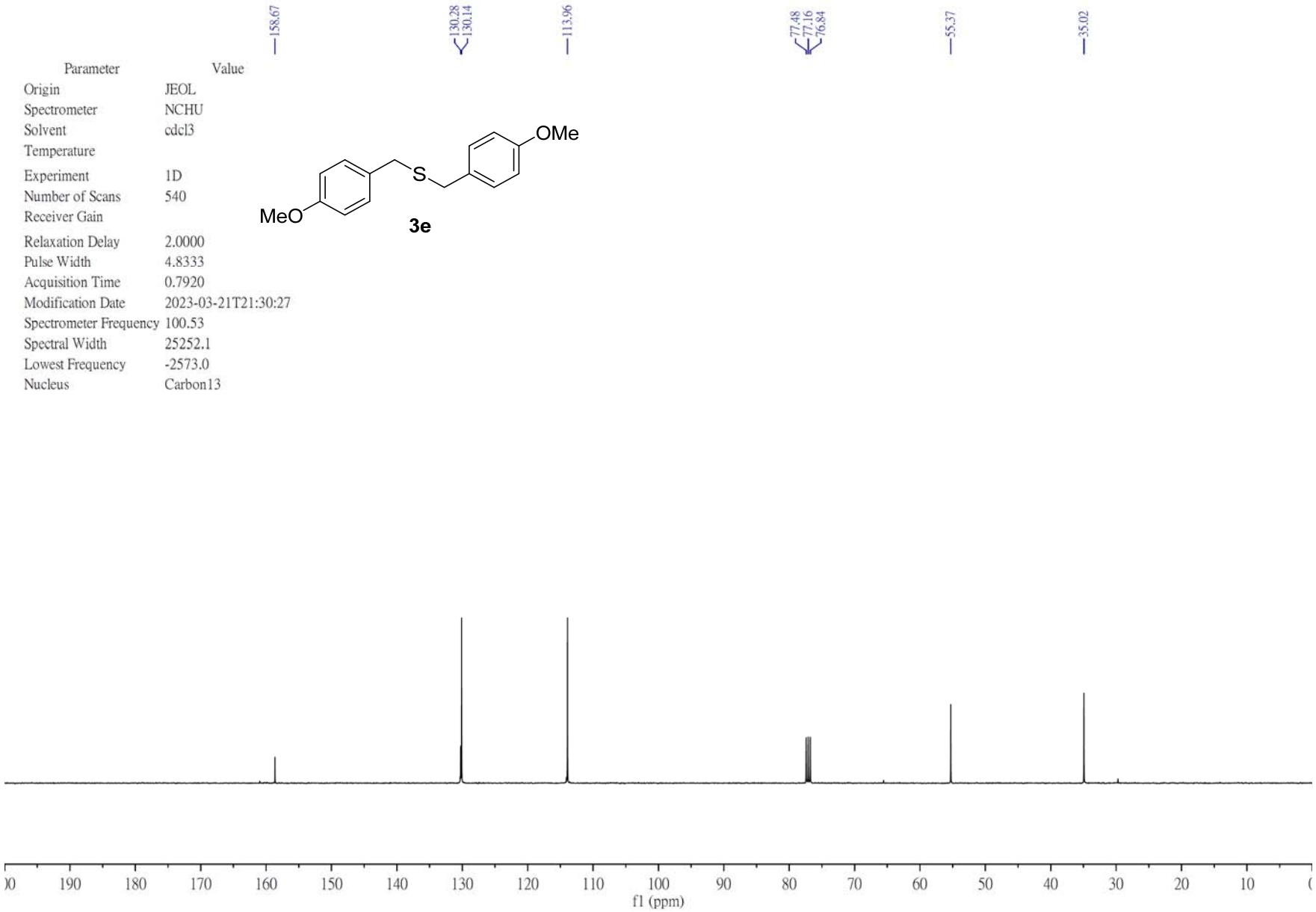
3d ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



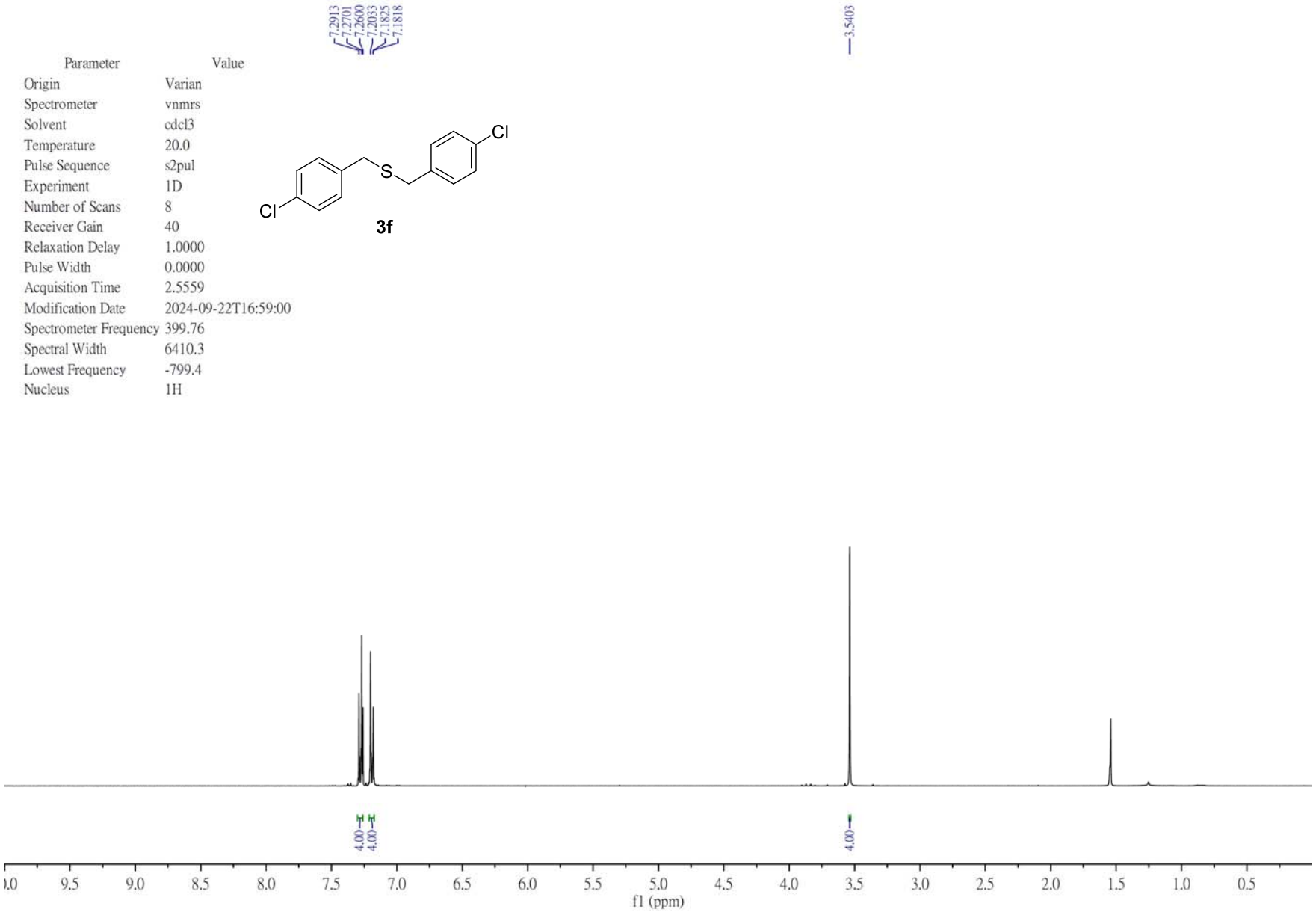
3d $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



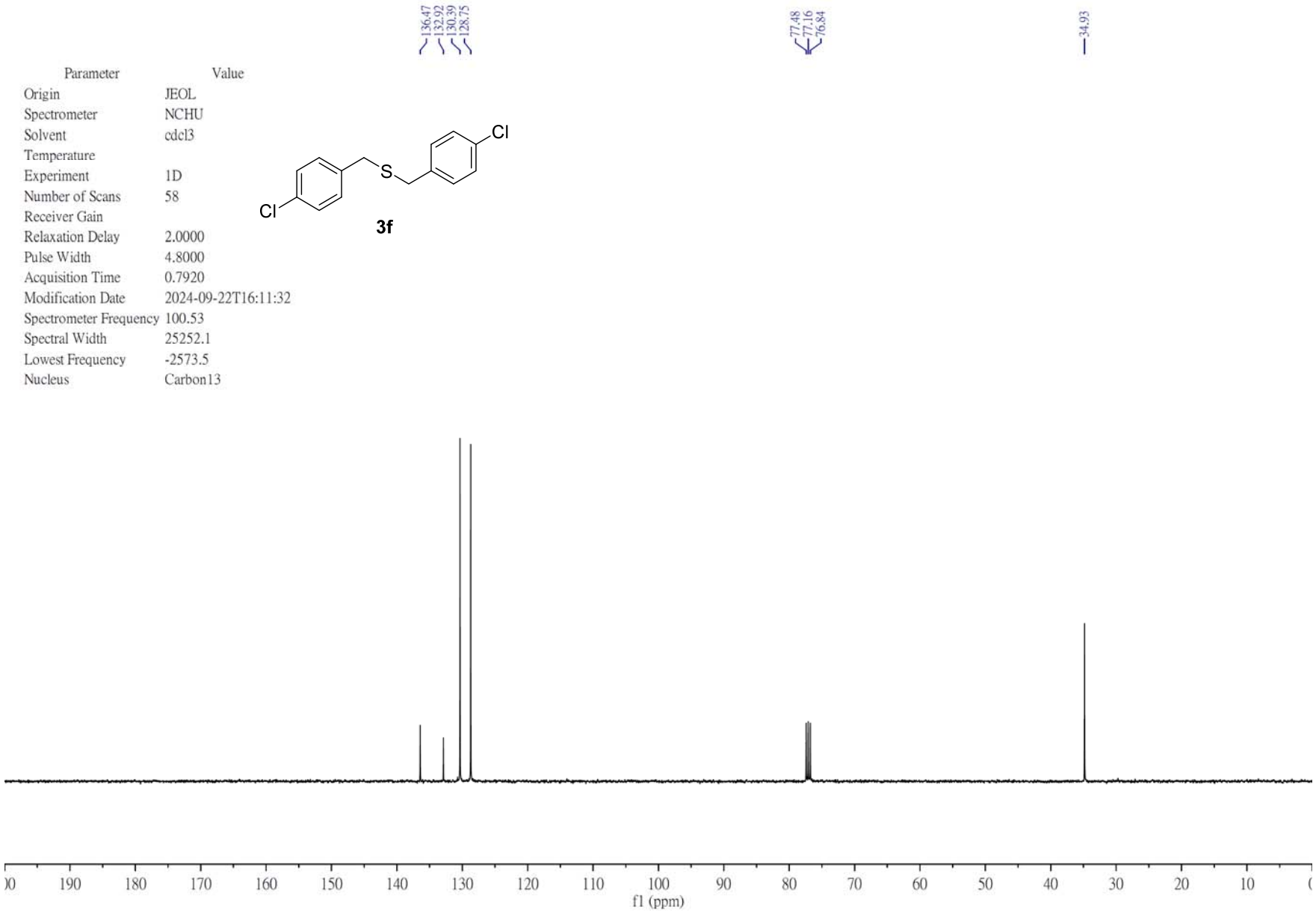
3e ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



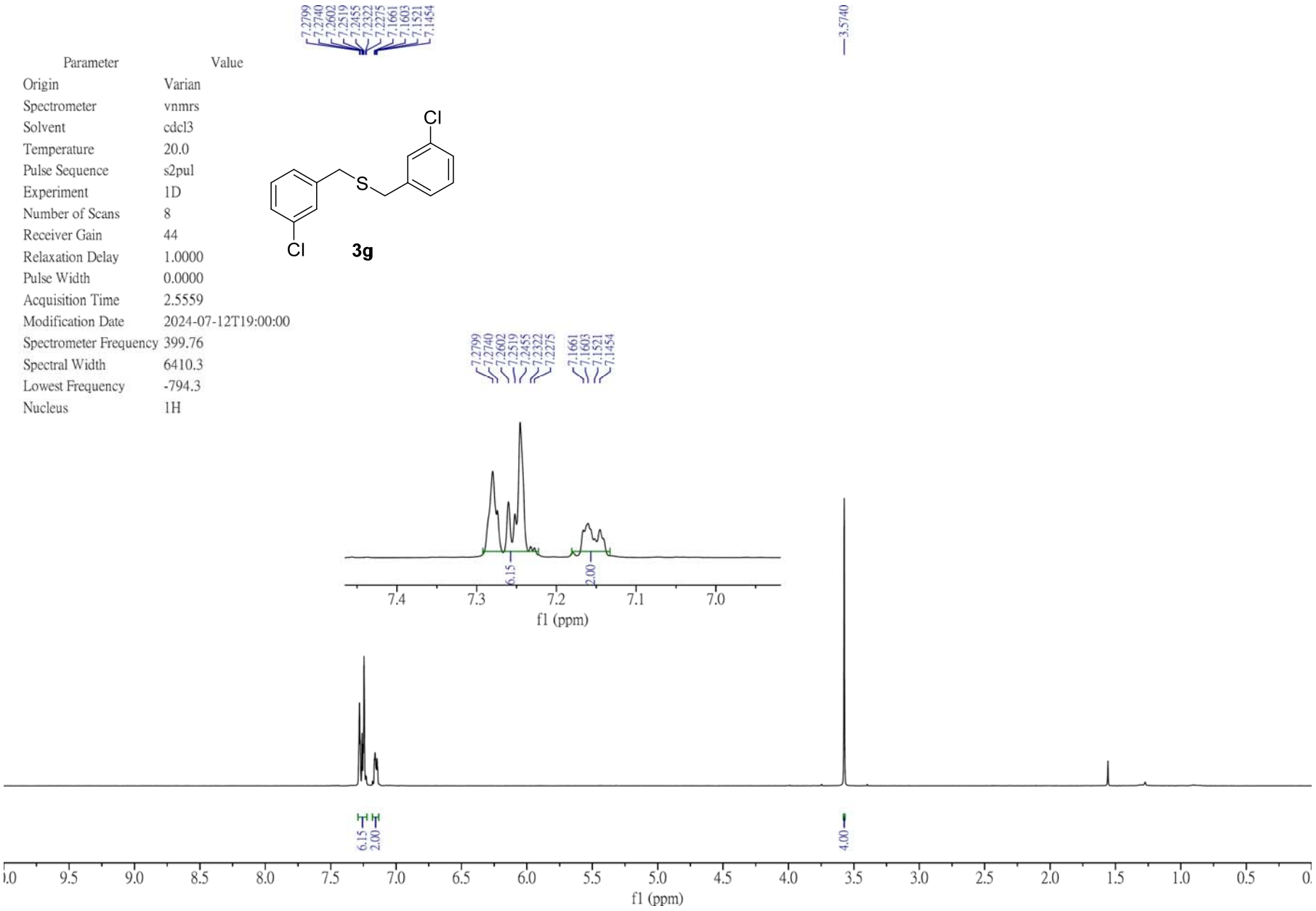
3e $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



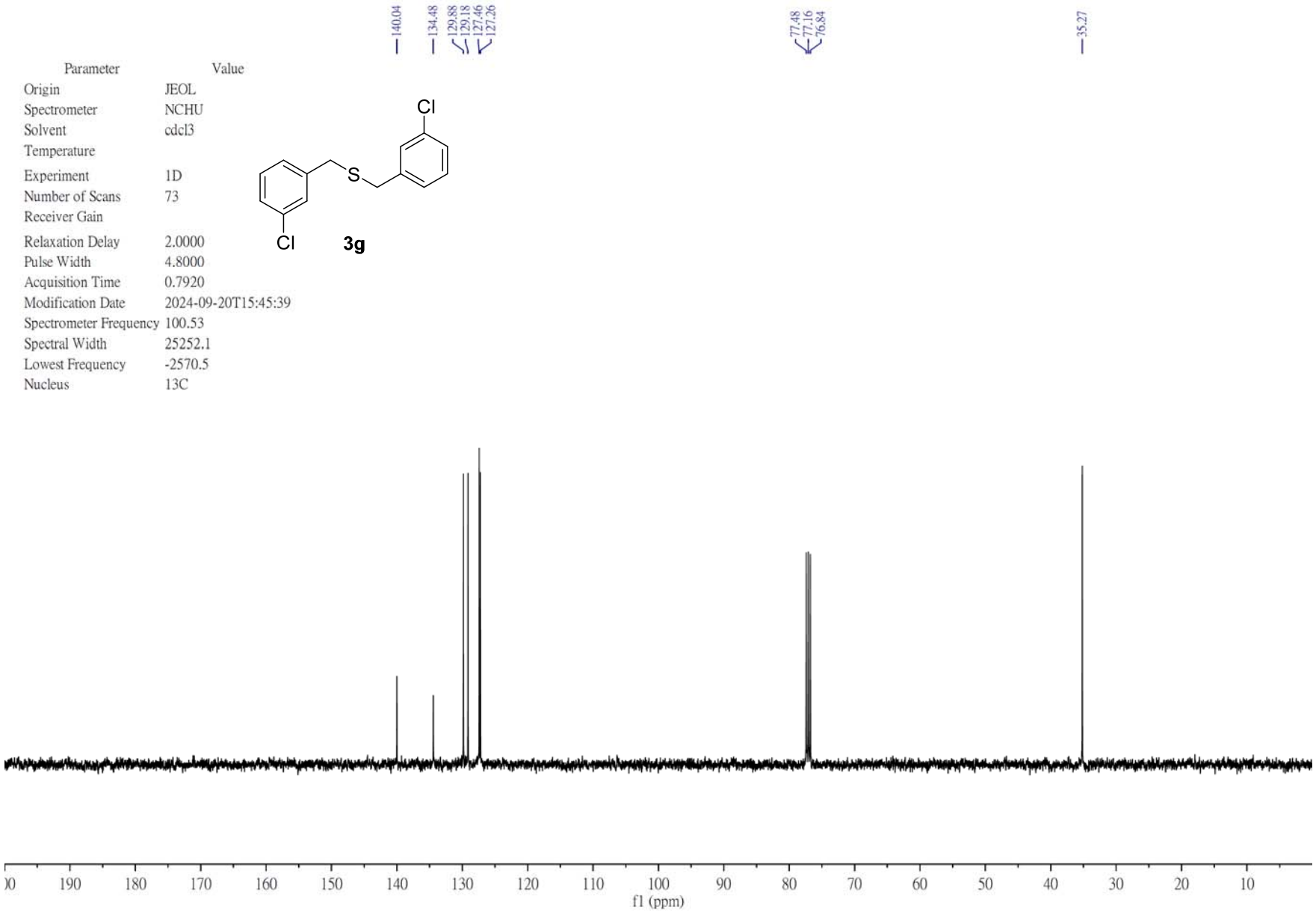
3f ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



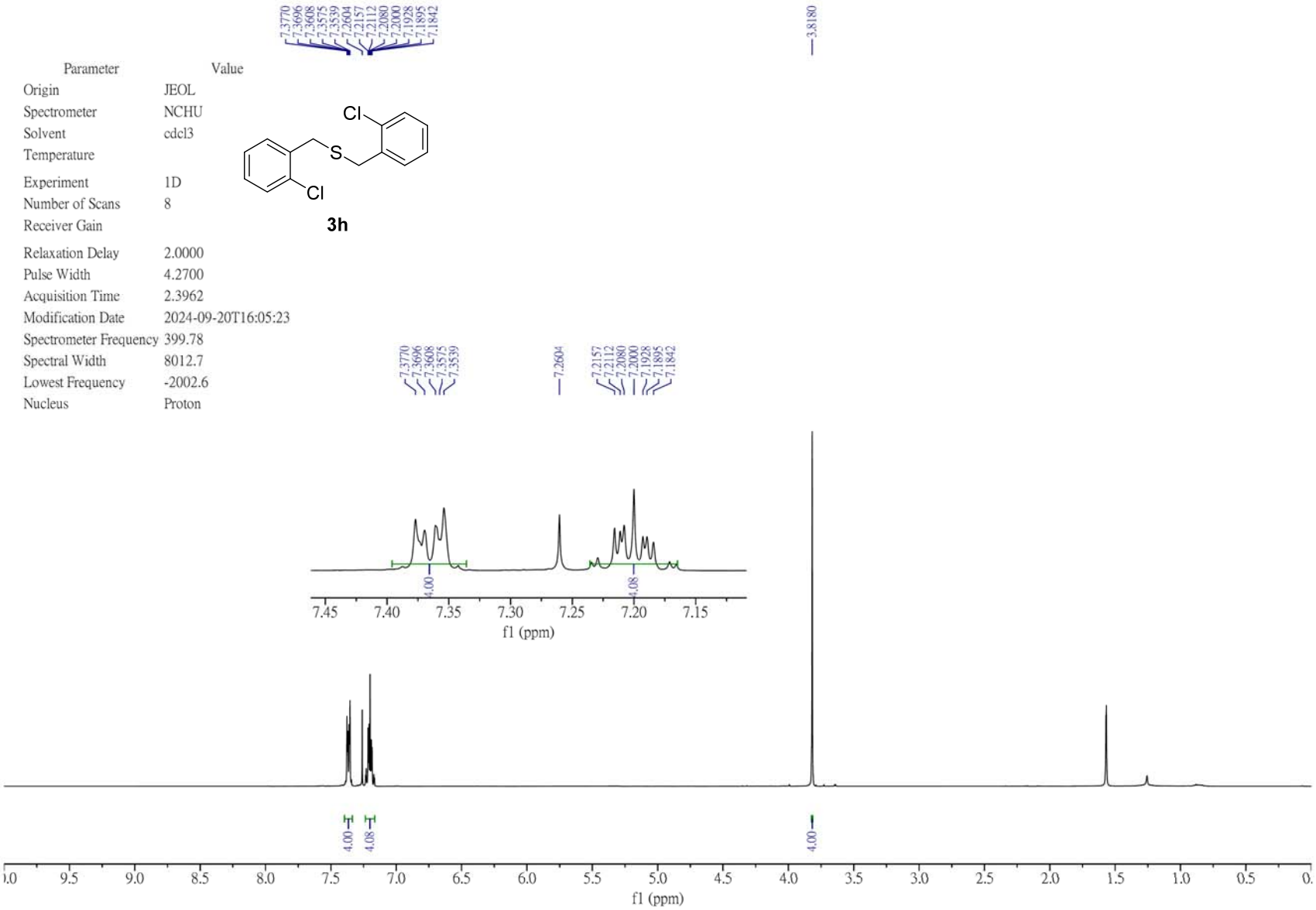
3f $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



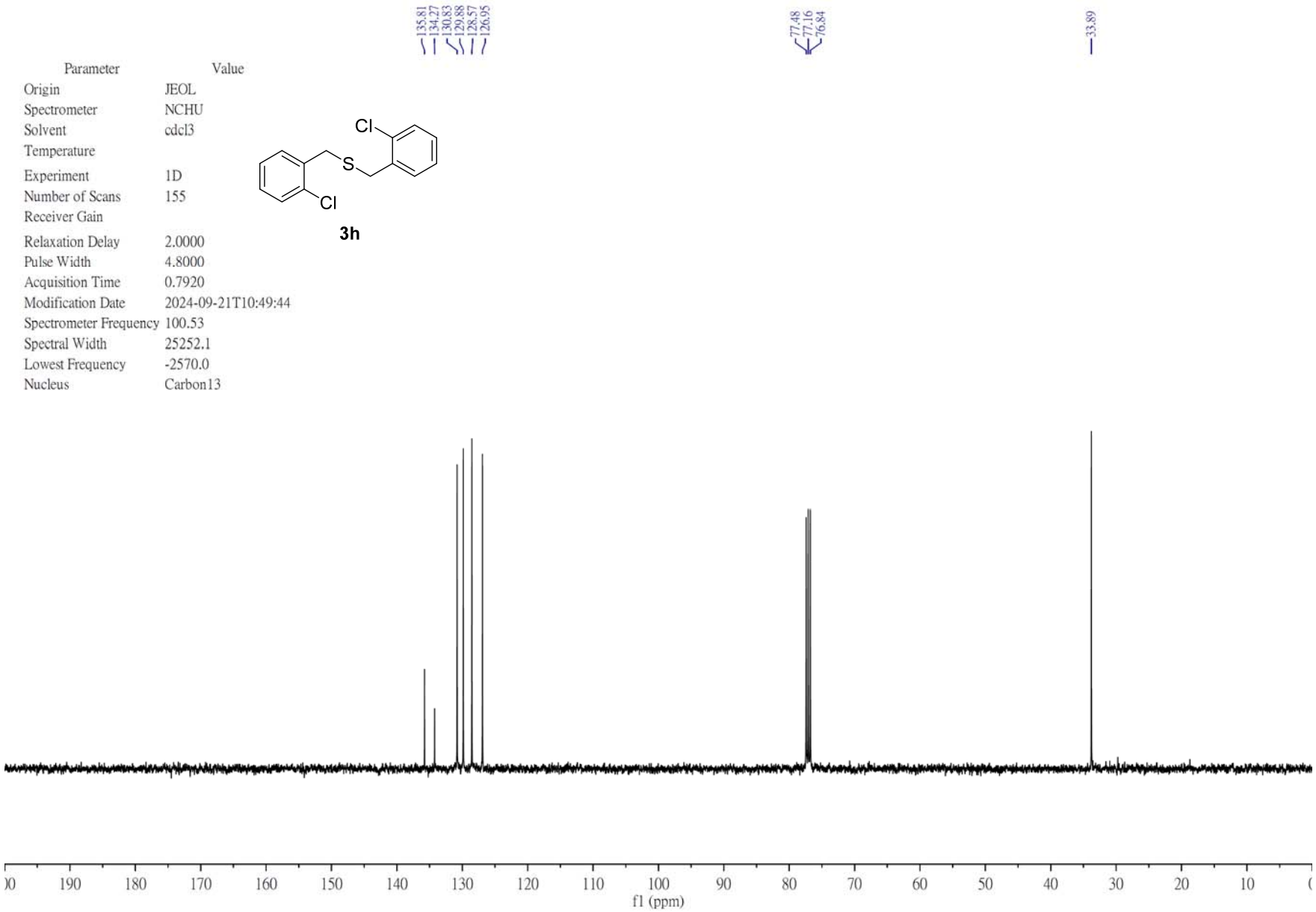
3g ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



3g $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

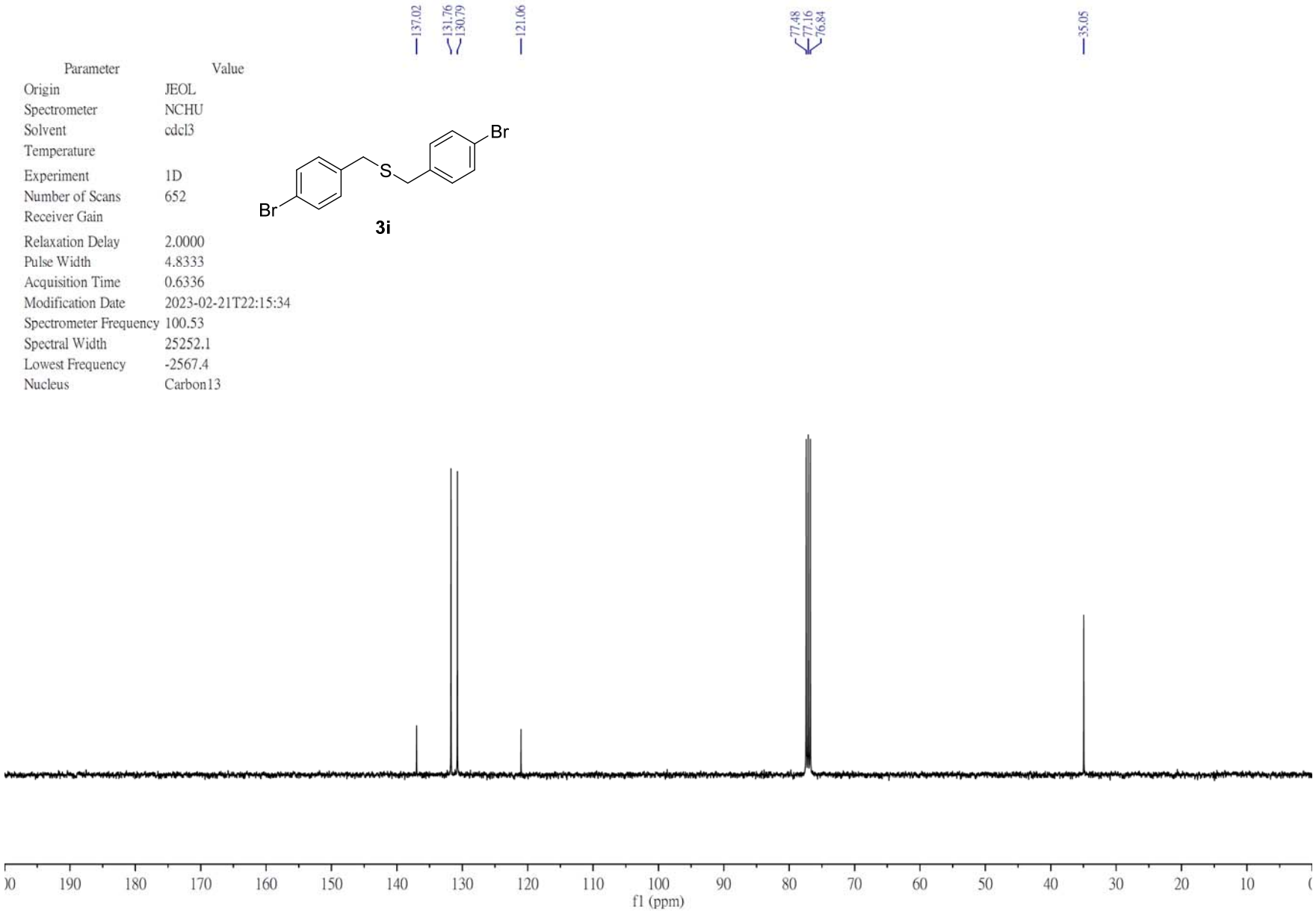


3h ^{1}H NMR spectrum（400 MHz in $CDCl_3$）

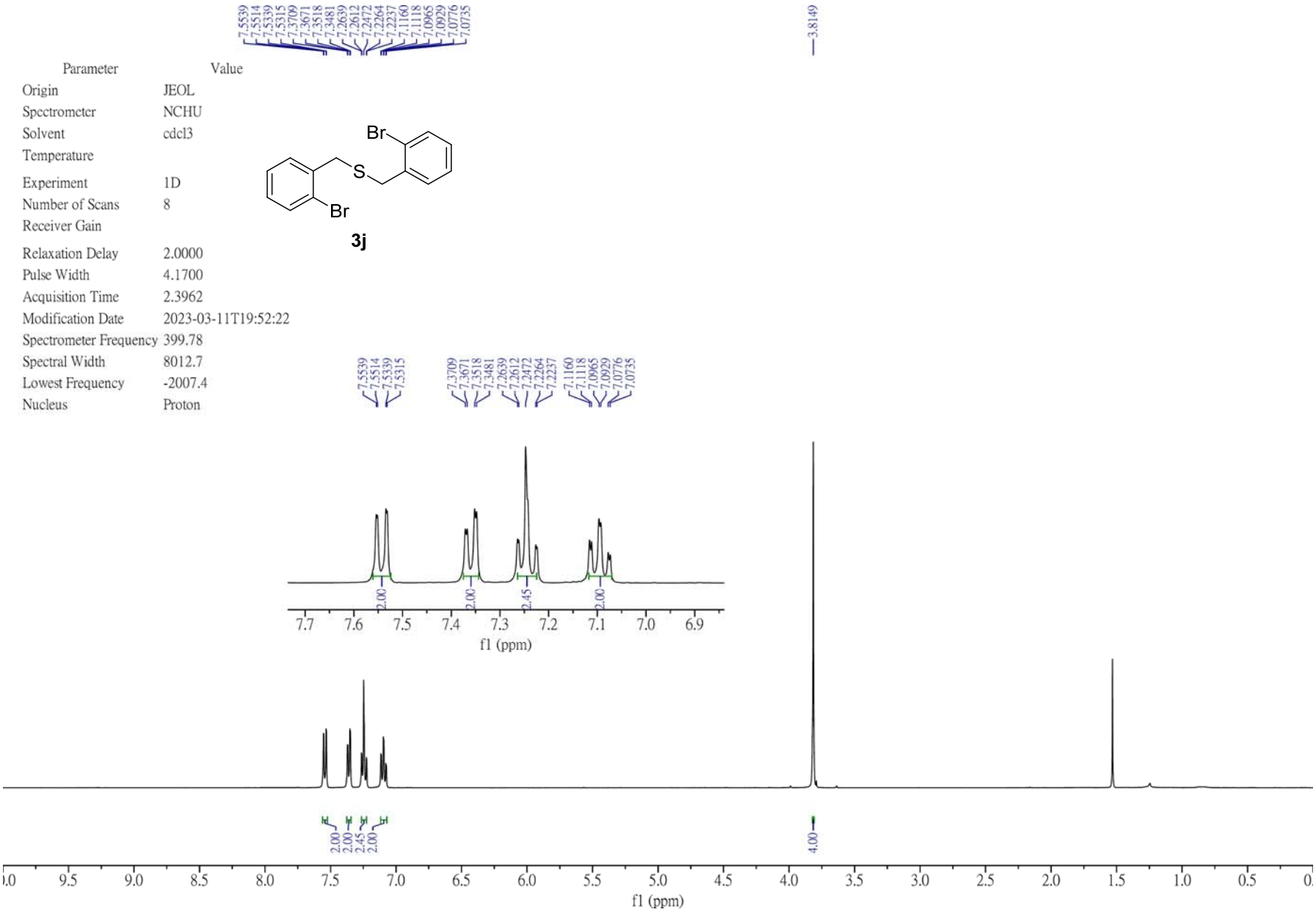


3h $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

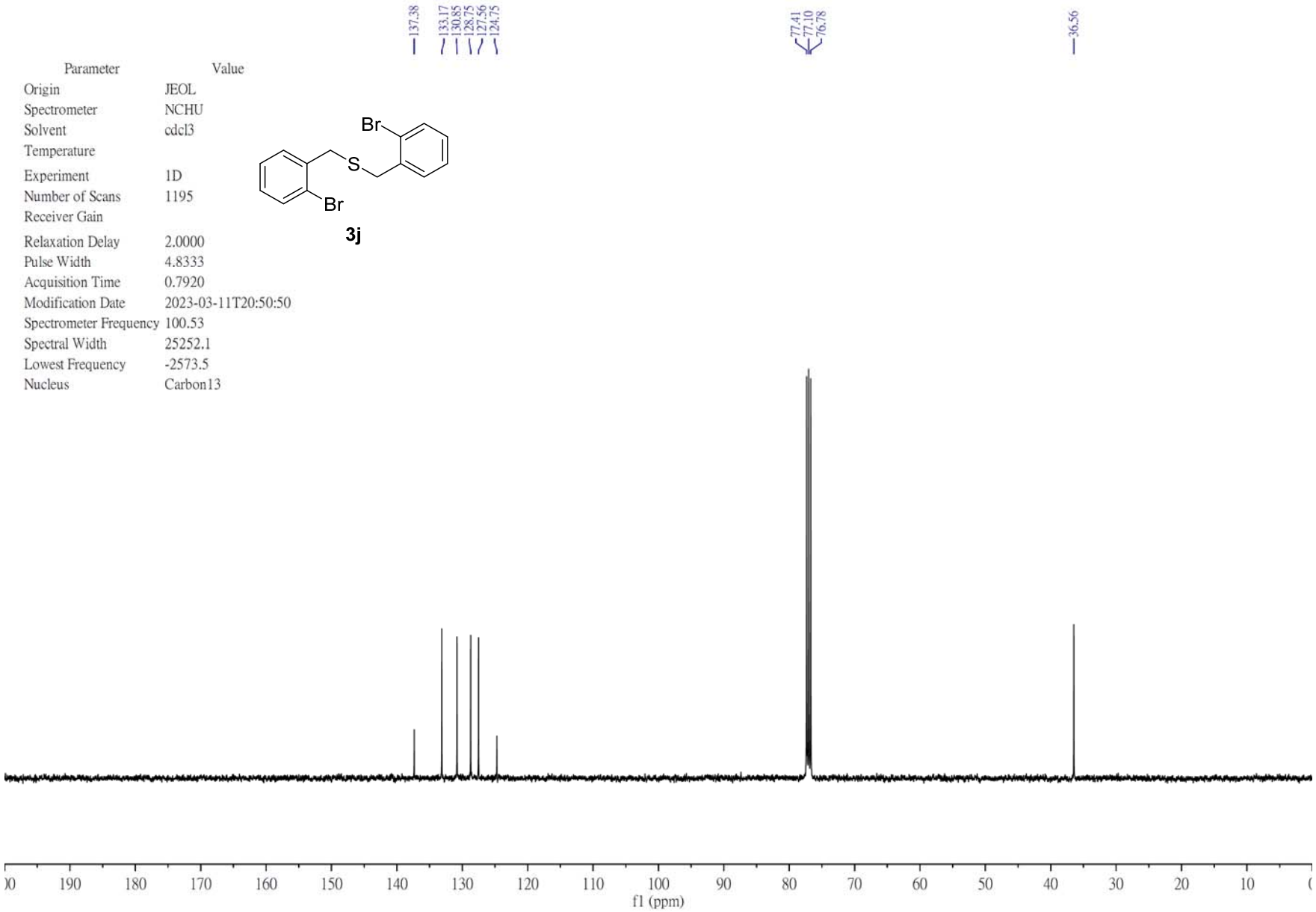
3i ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



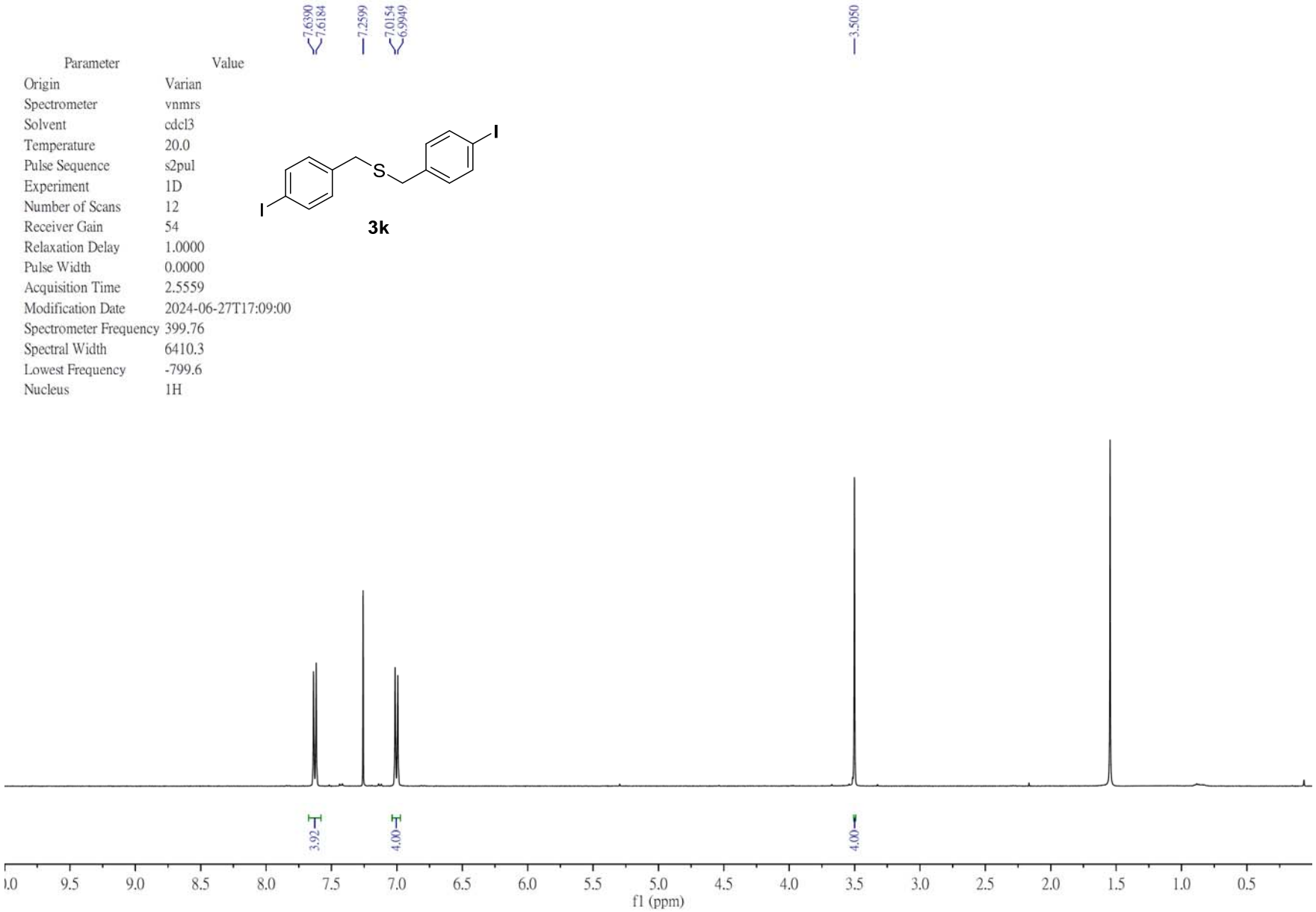
3i $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



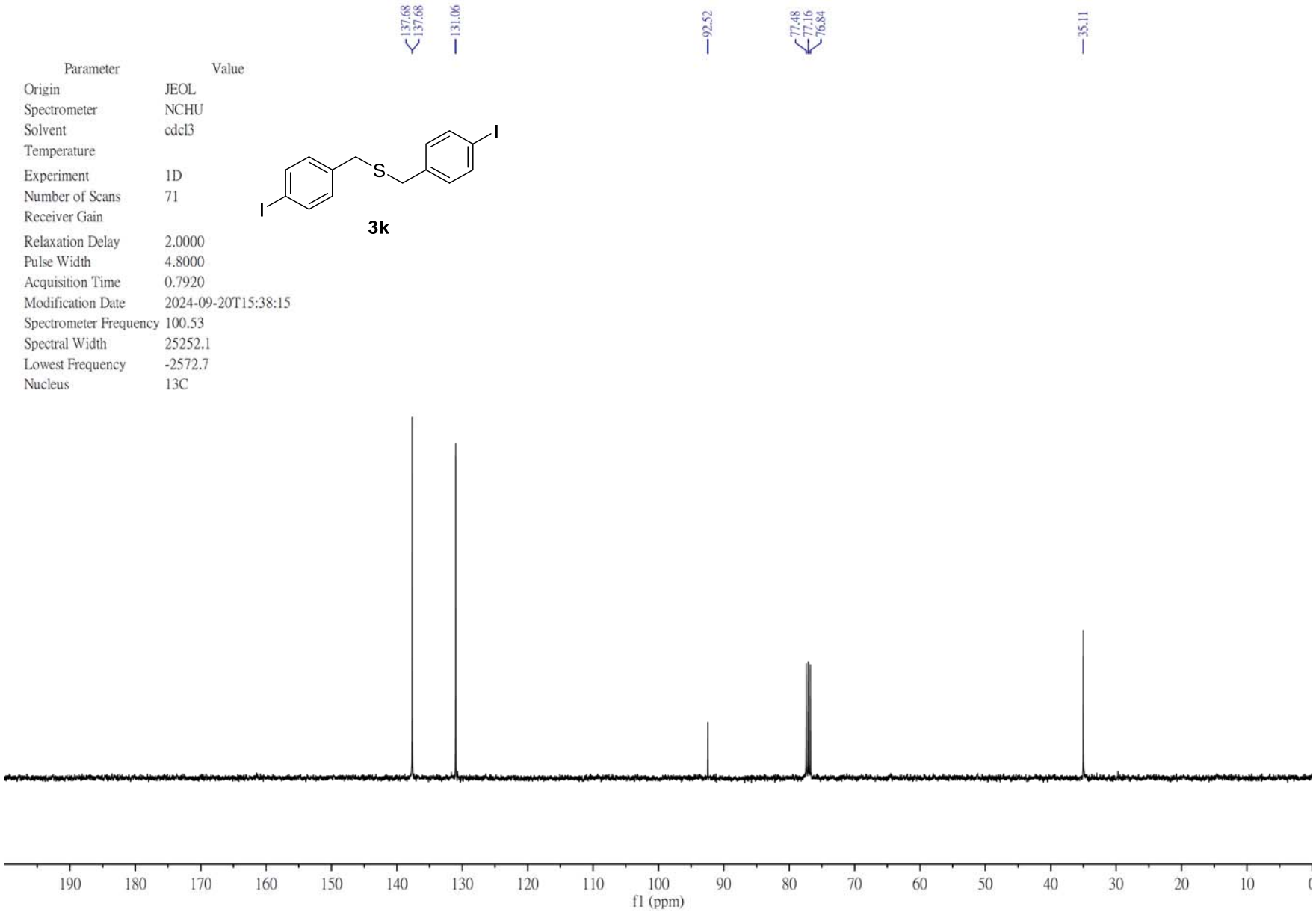
3j ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



3j $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

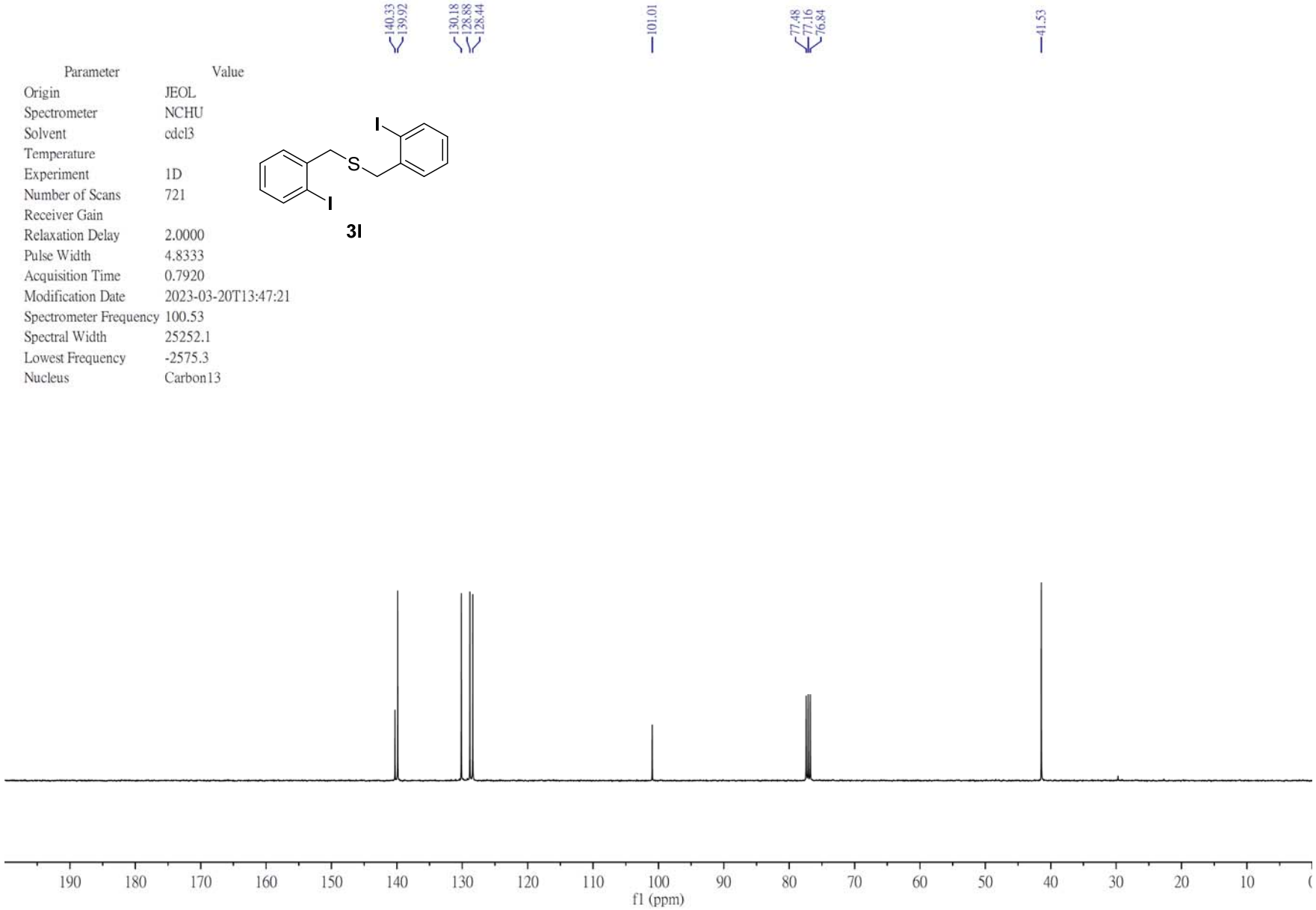


3k ^{1}H NMR spectrum（400 MHz in $CDCl_3$）

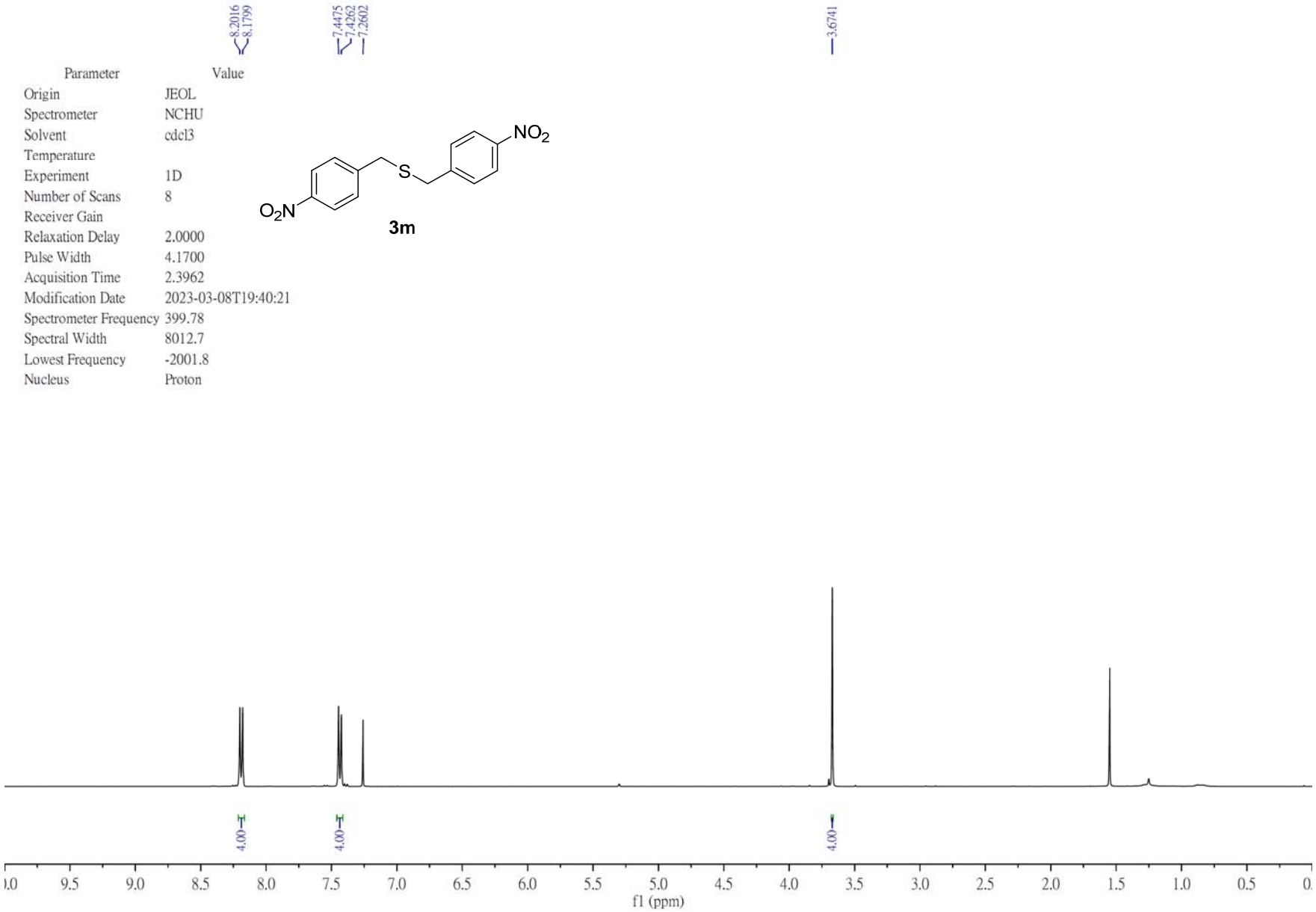


3k $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

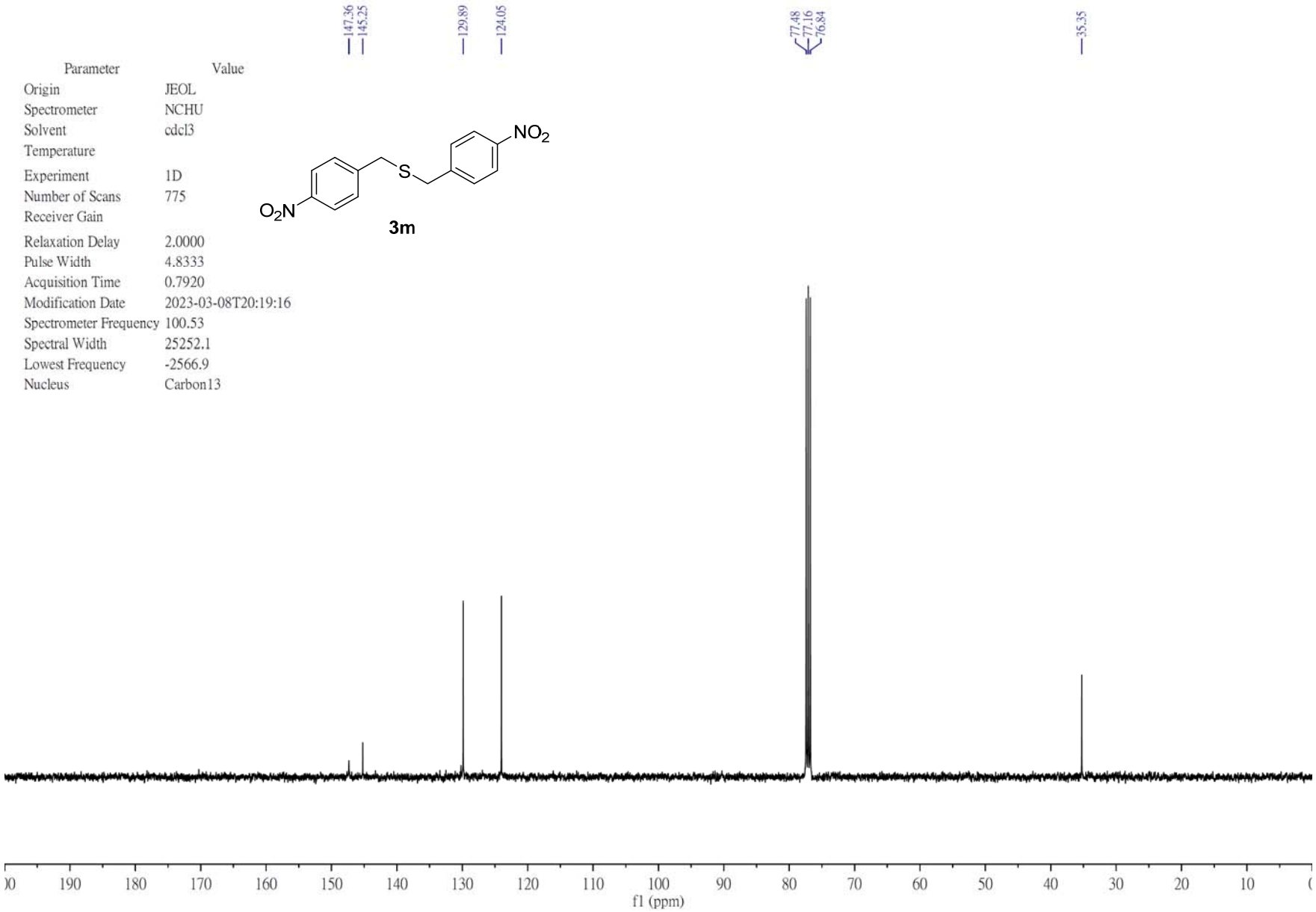
3l ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



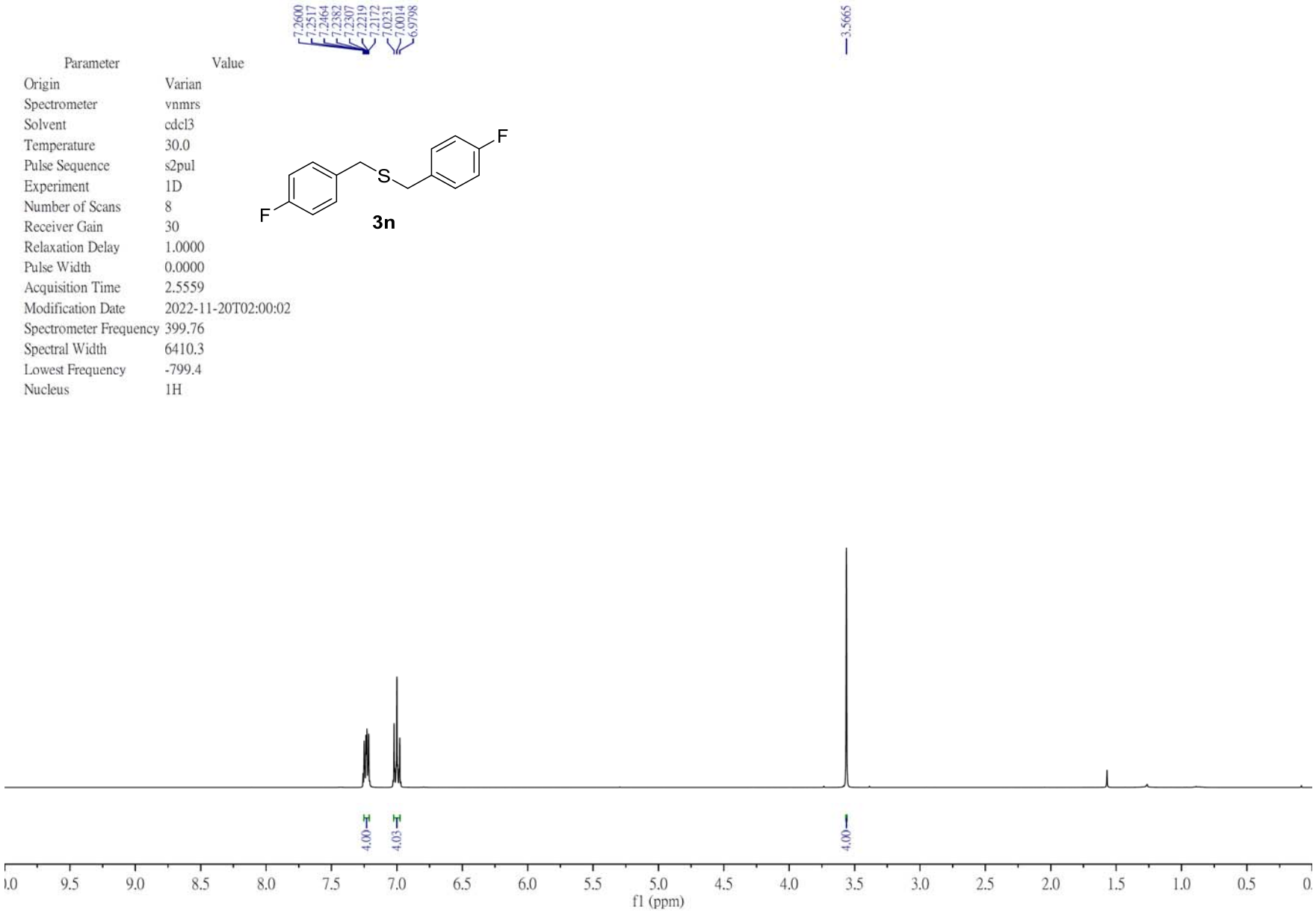
3l $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



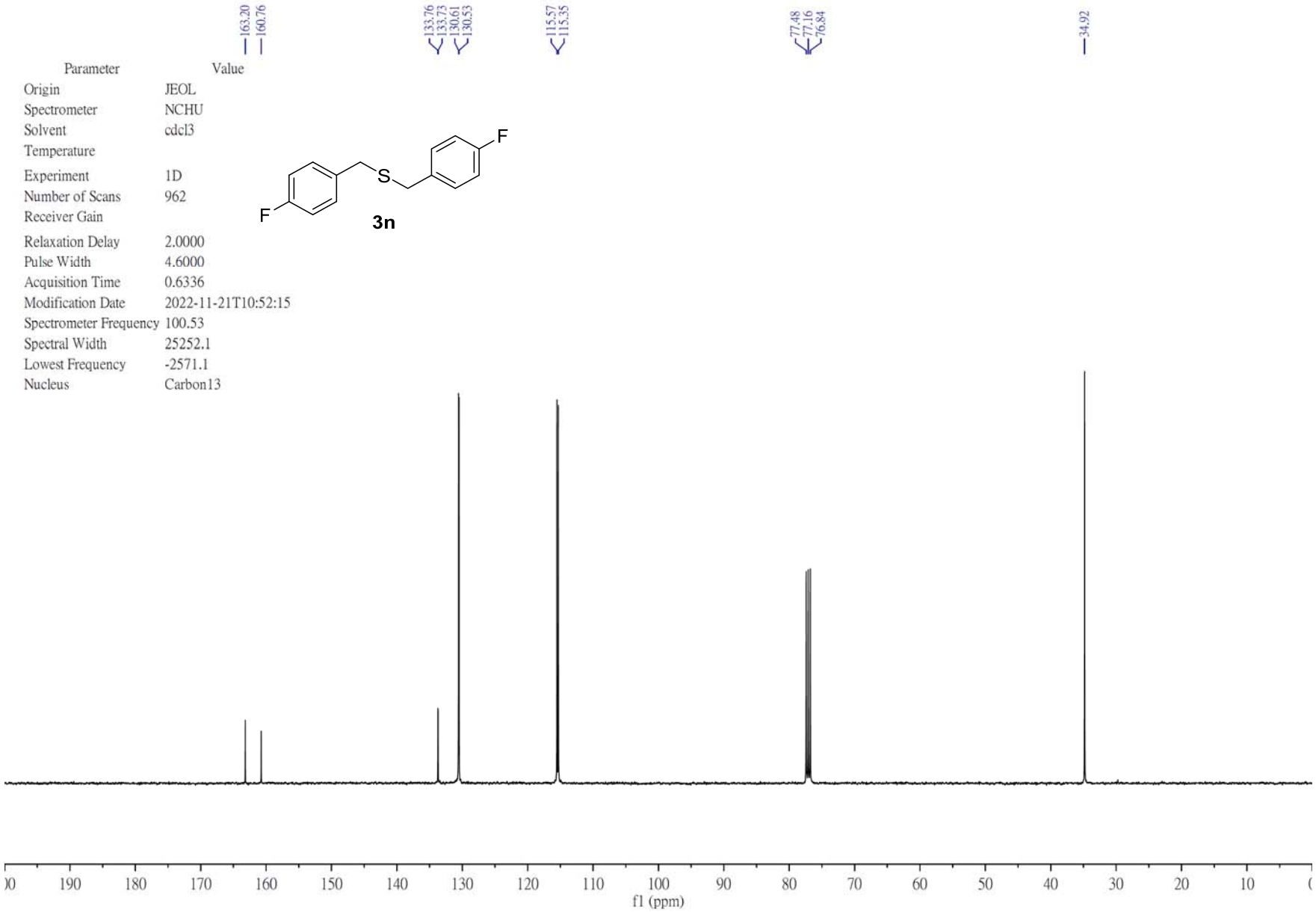
3m ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



3m $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



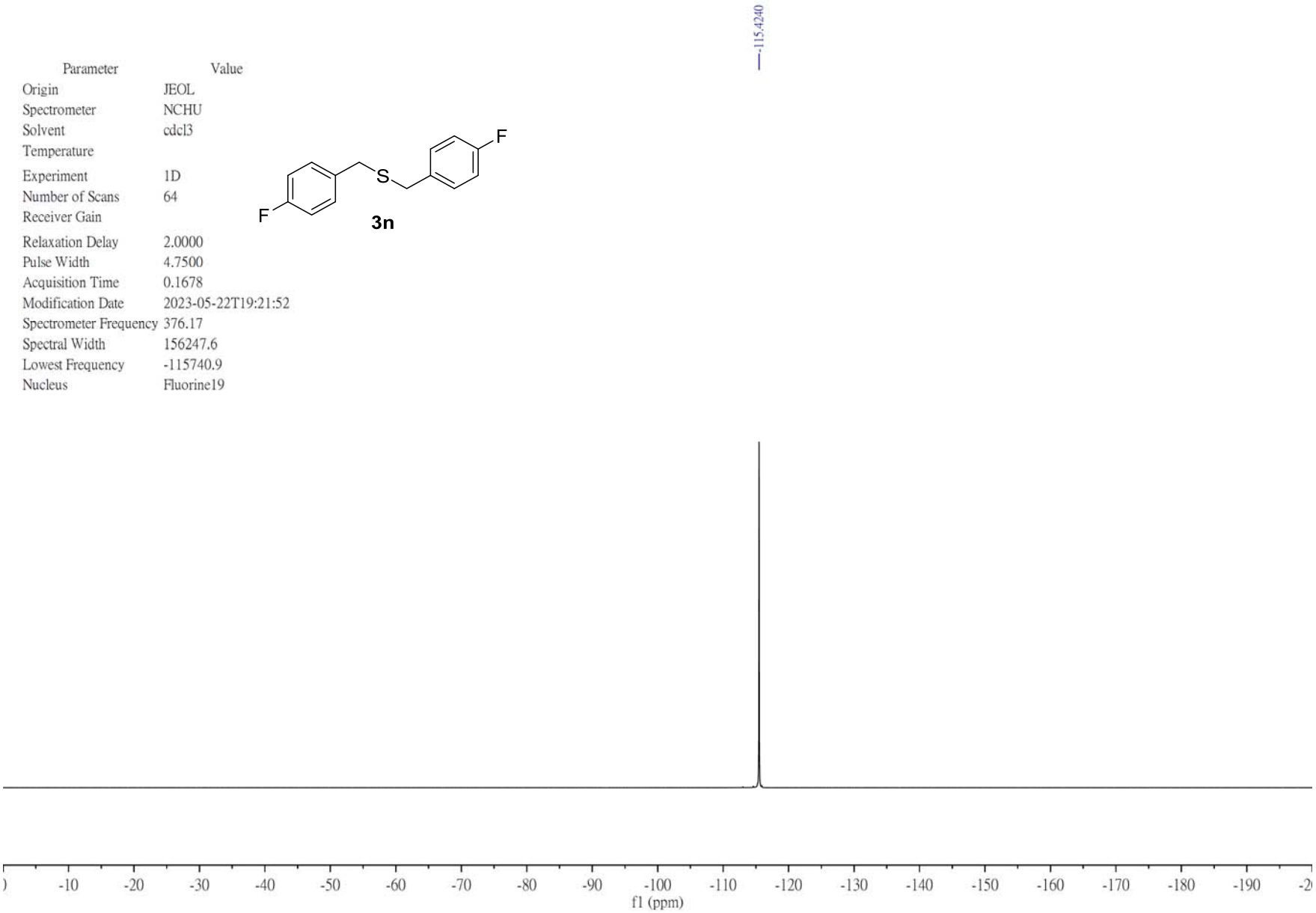
3n ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



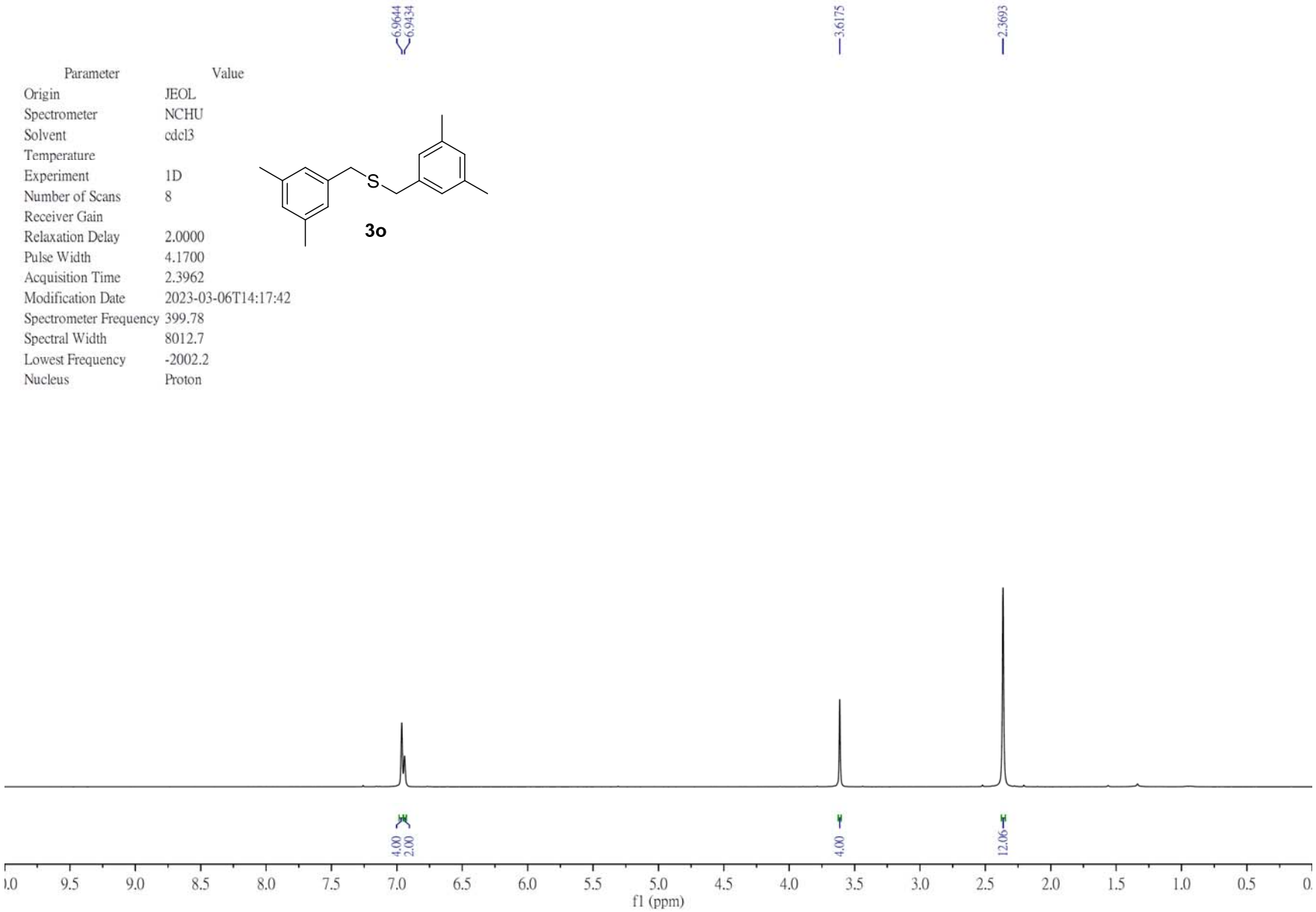
3n $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

Parameter	Value
Origin	JEOL
Spectrometer	NCHU
Solvent	cdcl3
Temperature	
Experiment	1D
Number of Scans	64
Receiver Gain	
Relaxation Delay	2.0000
Pulse Width	4.7500
Acquisition Time	0.1678
Modification Date	2023-05-22T19:21:52
Spectrometer Frequency	376.17
Spectral Width	156247.6
Lowest Frequency	-115740.9
Nucleus	Fluorine19

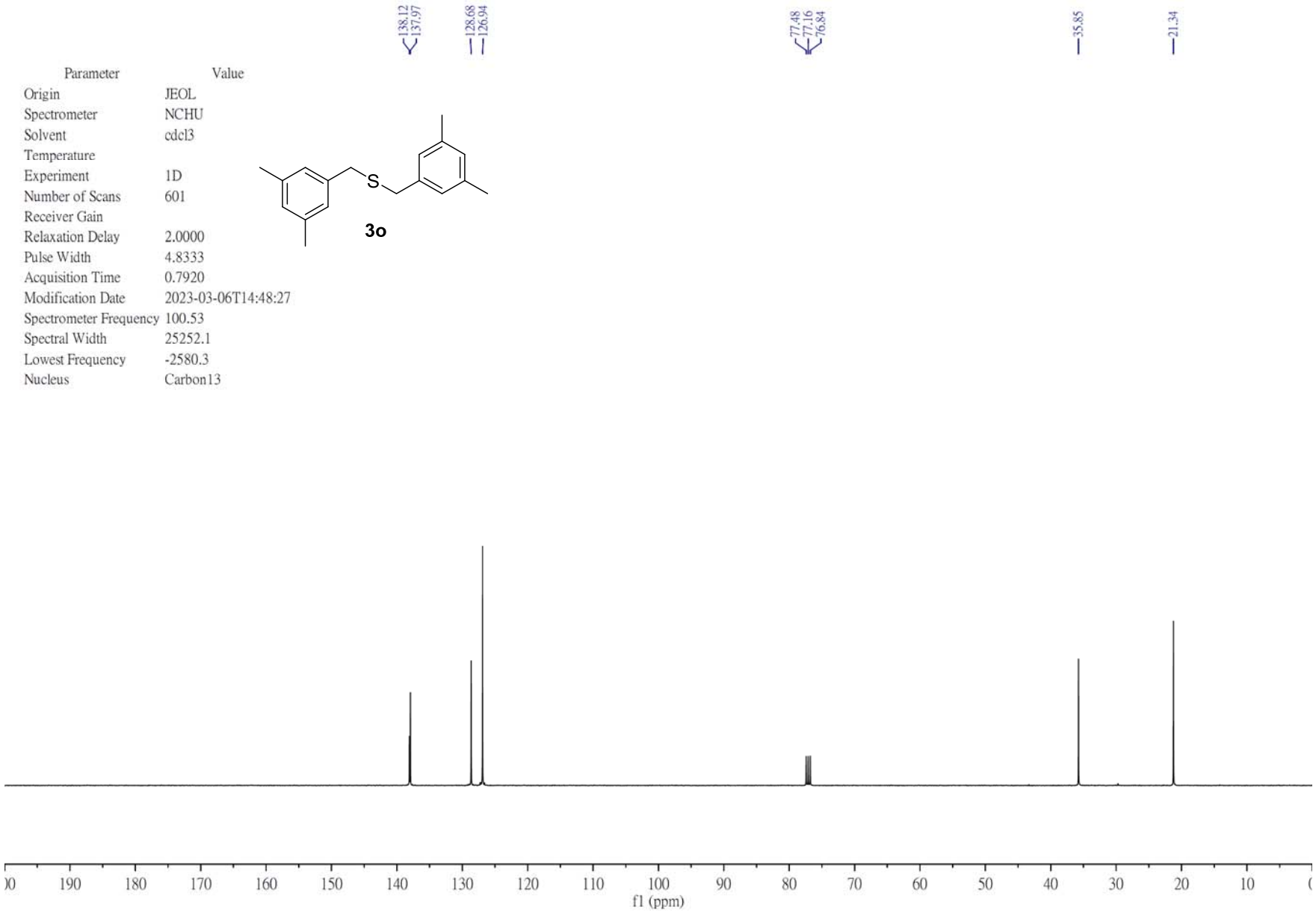
3n



3n ^{19}F NMR spectrum（376MHz in $CDCl_3$）

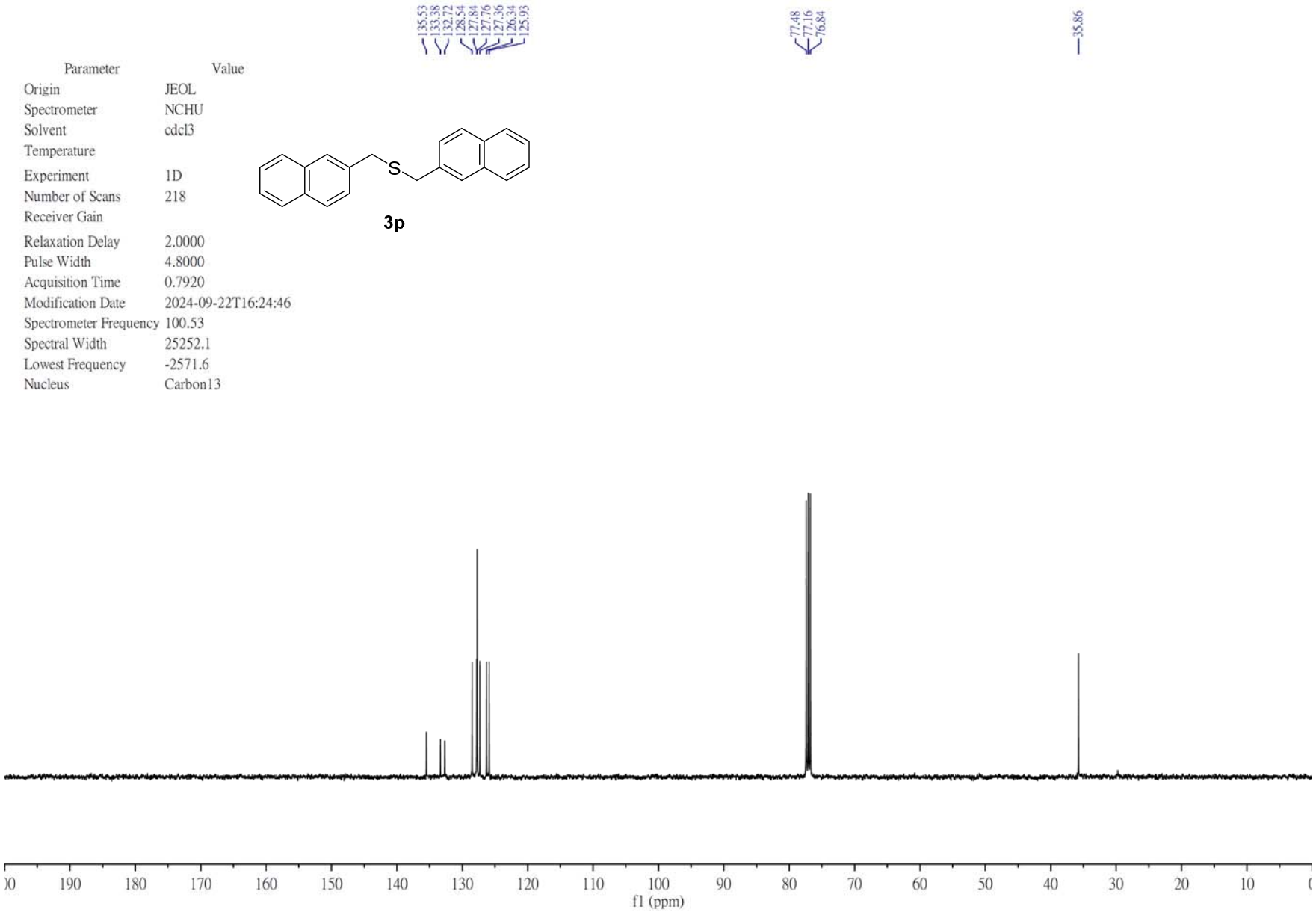


3o ^{1}H NMR spectrum（400 MHz in $CDCl_3$）

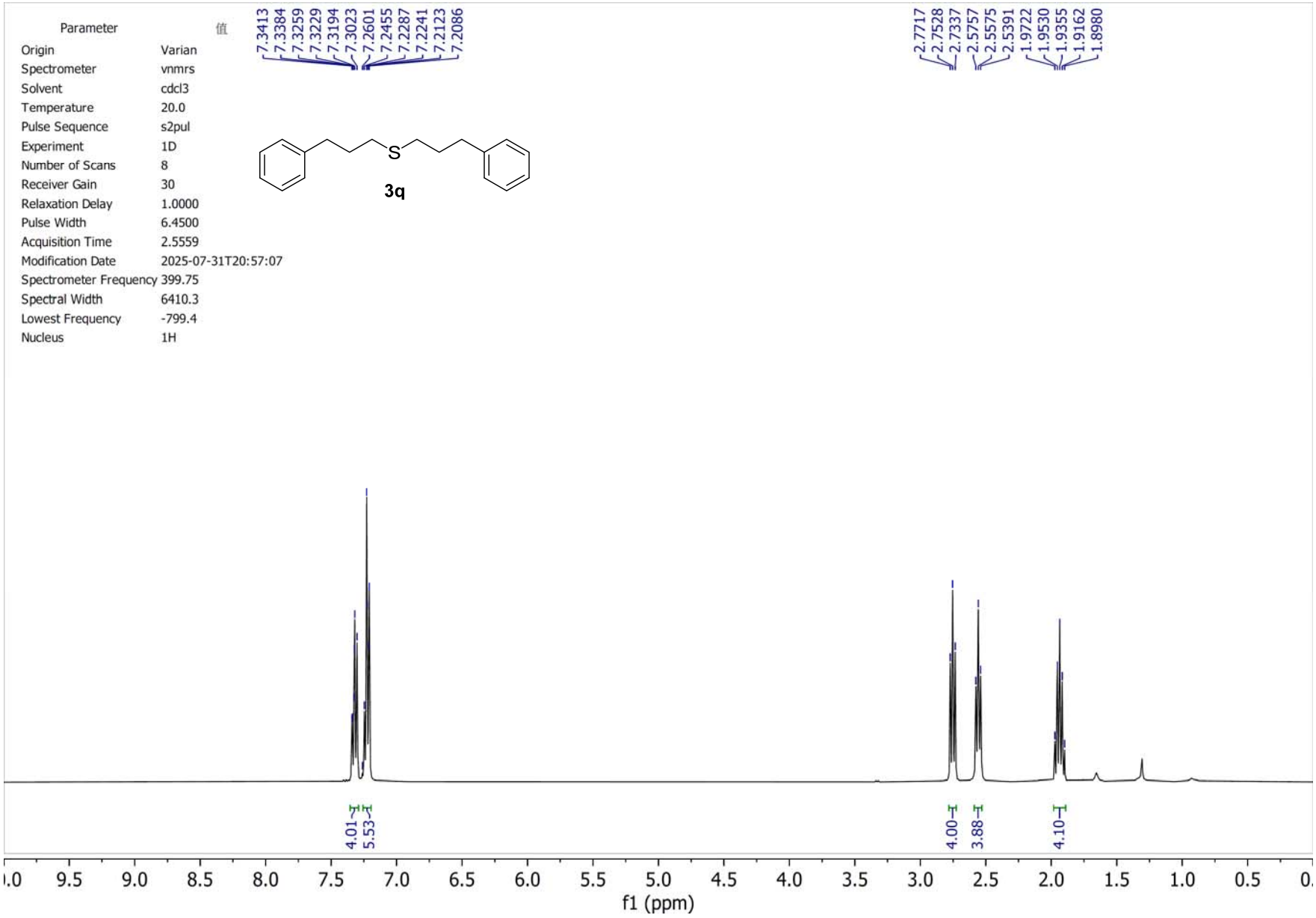


3o $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

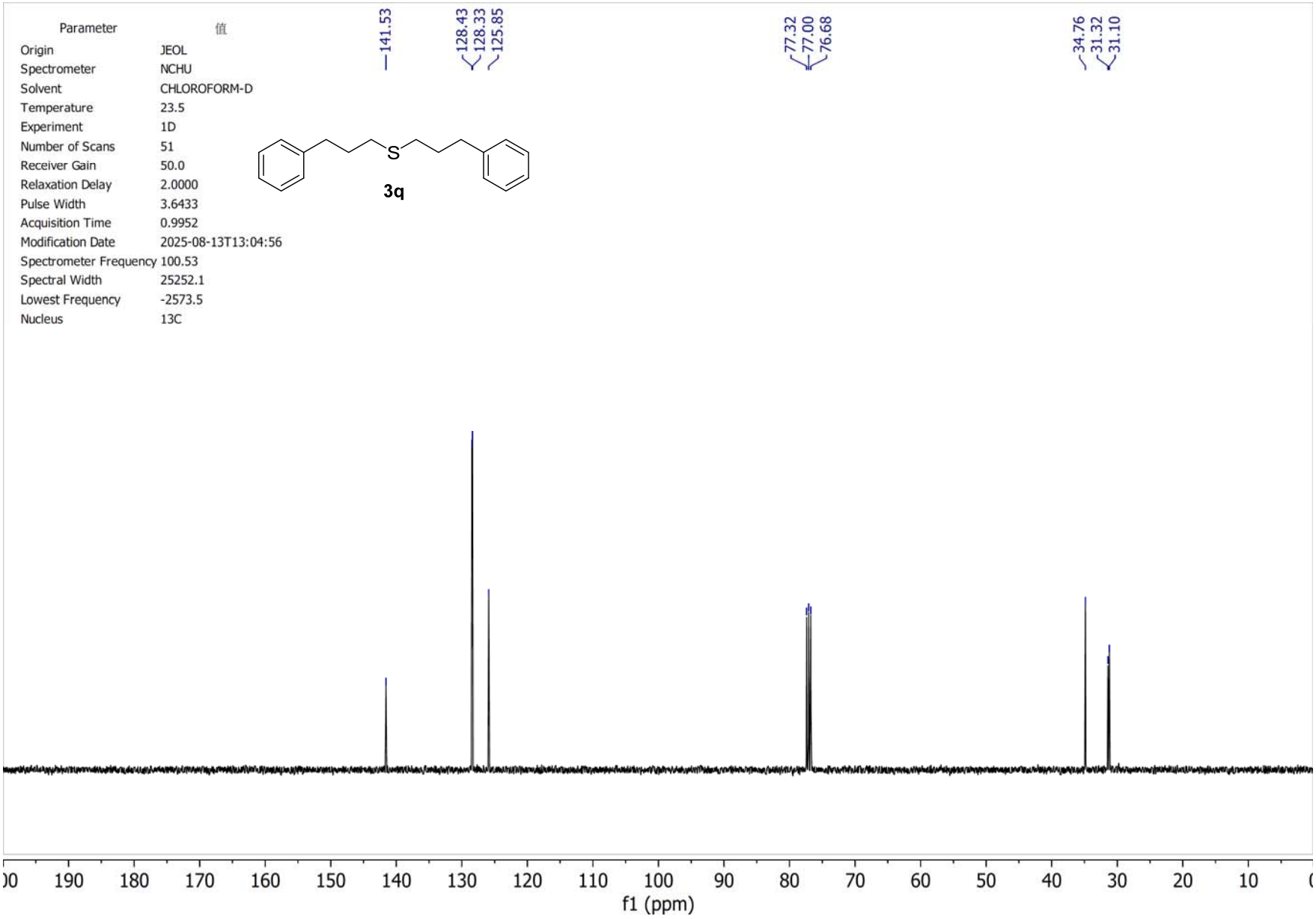
3p ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



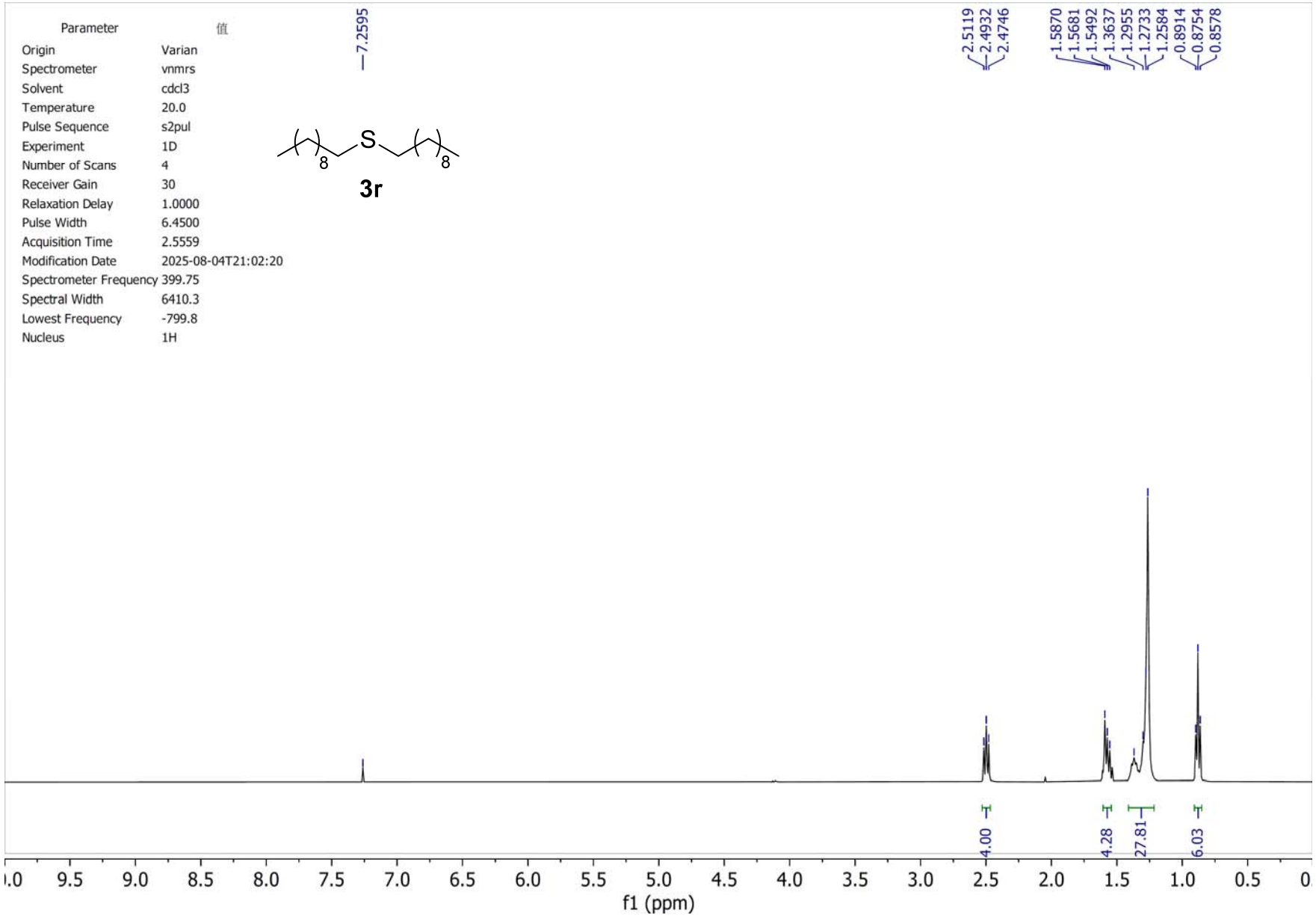
3p $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



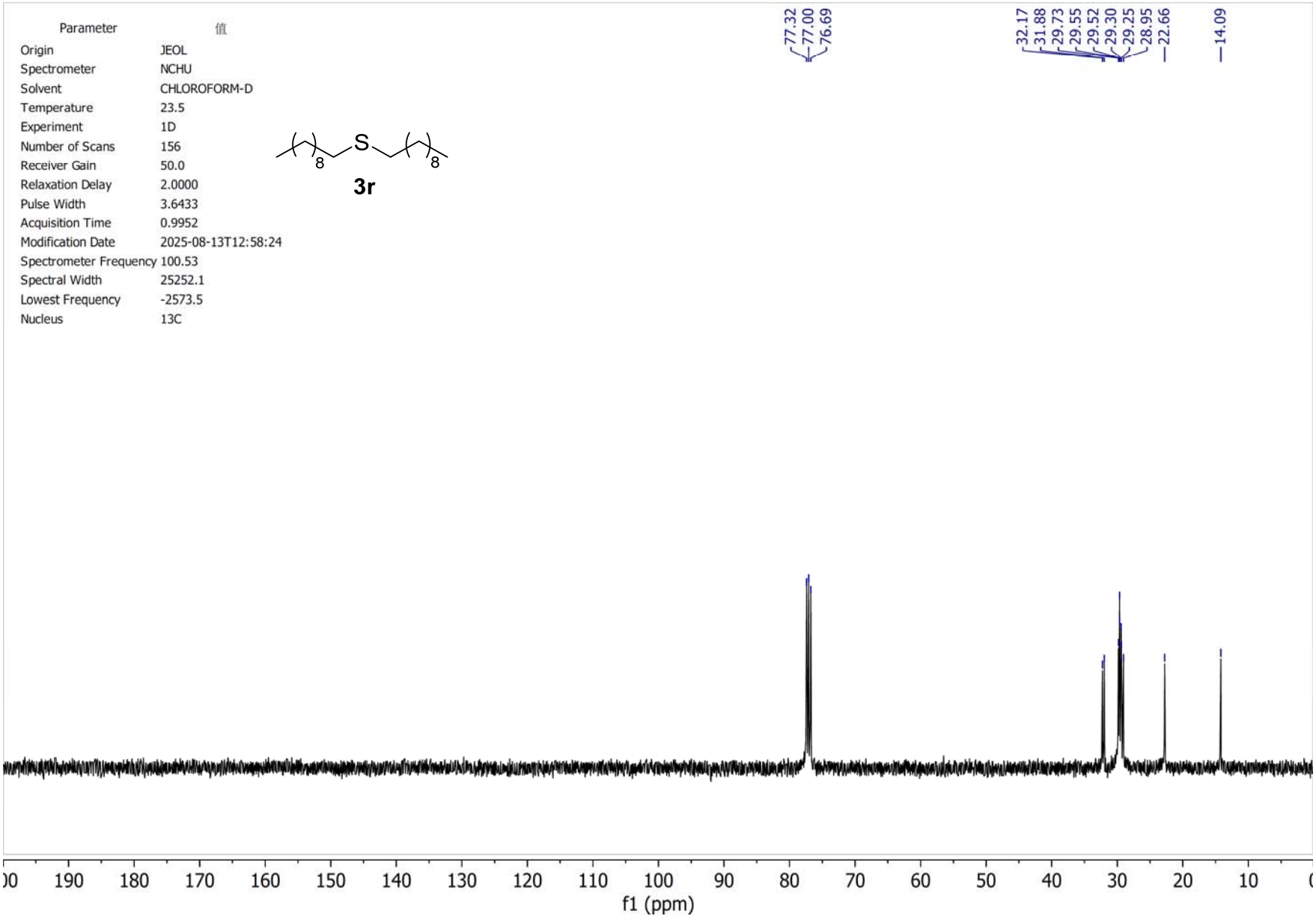
3q ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



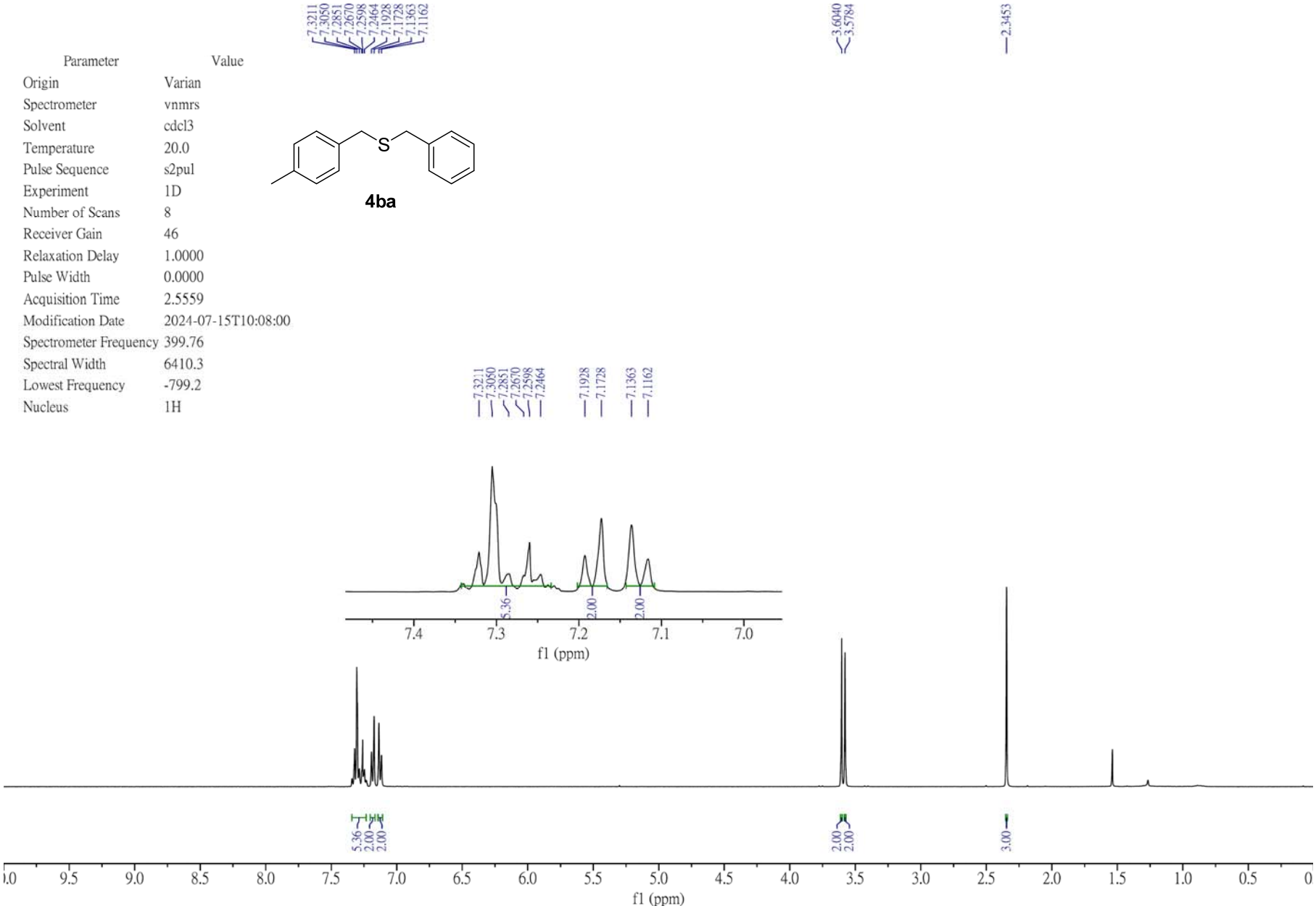
3q $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



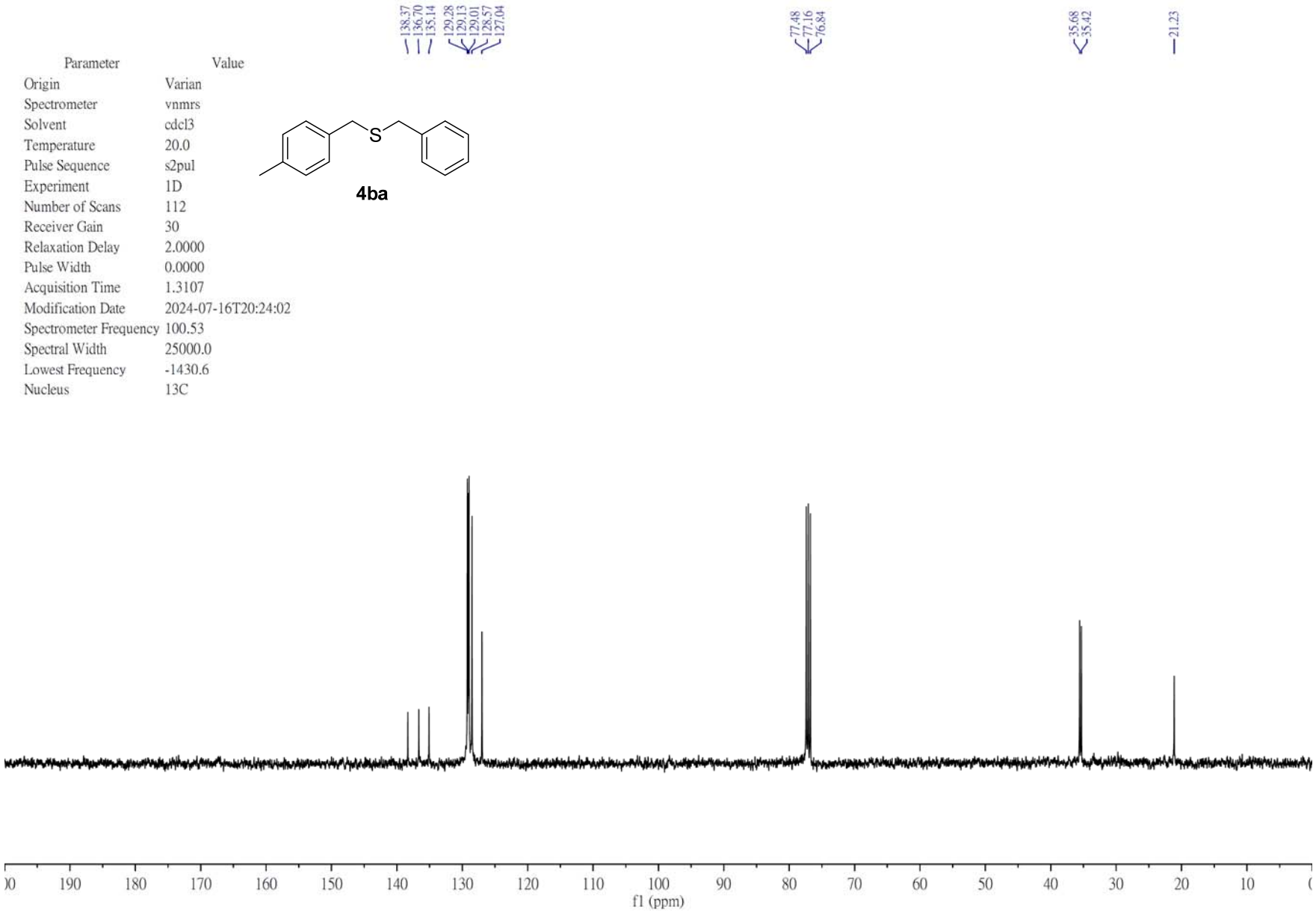
3r ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



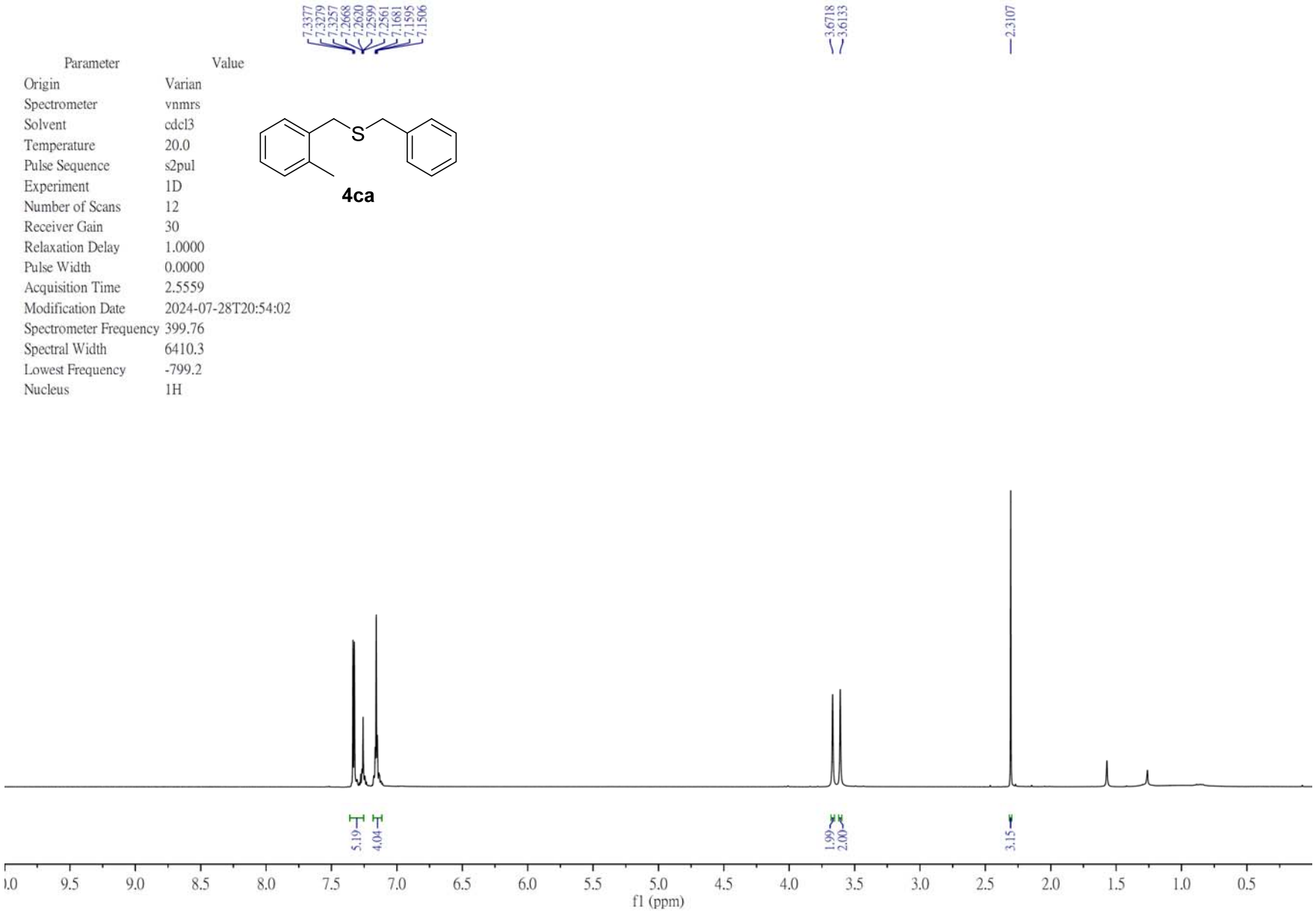
3r $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



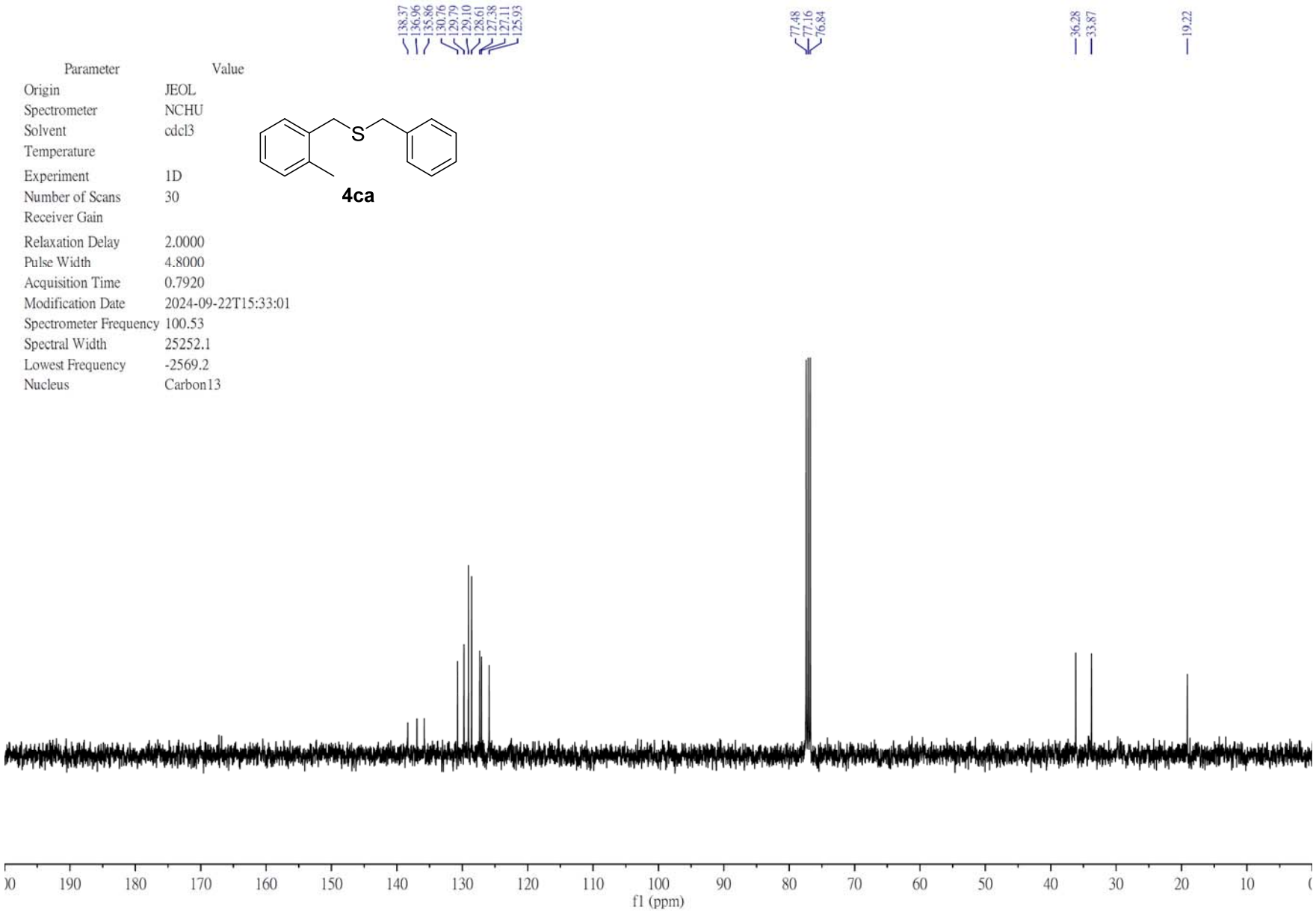
4ba ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



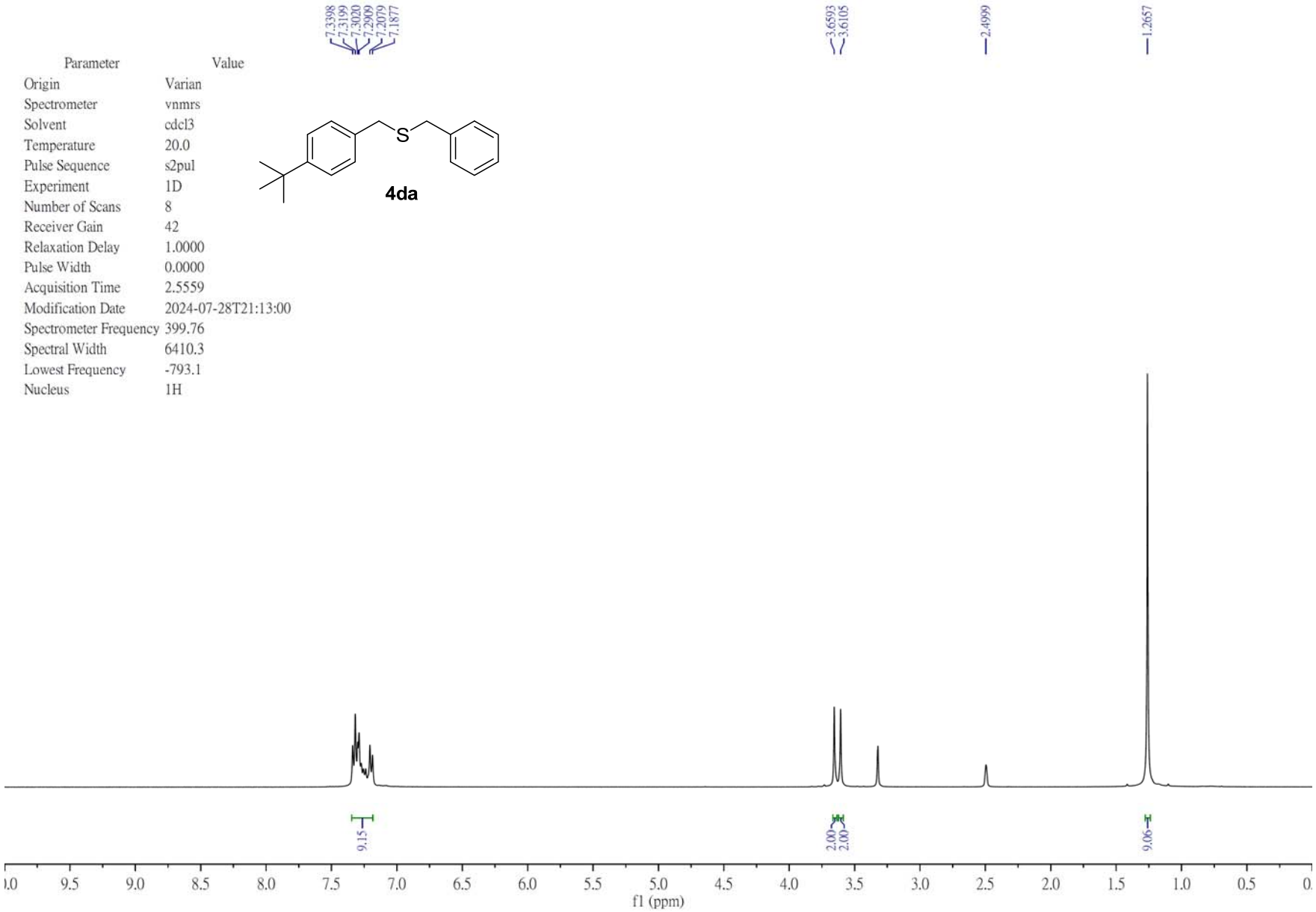
4ba $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



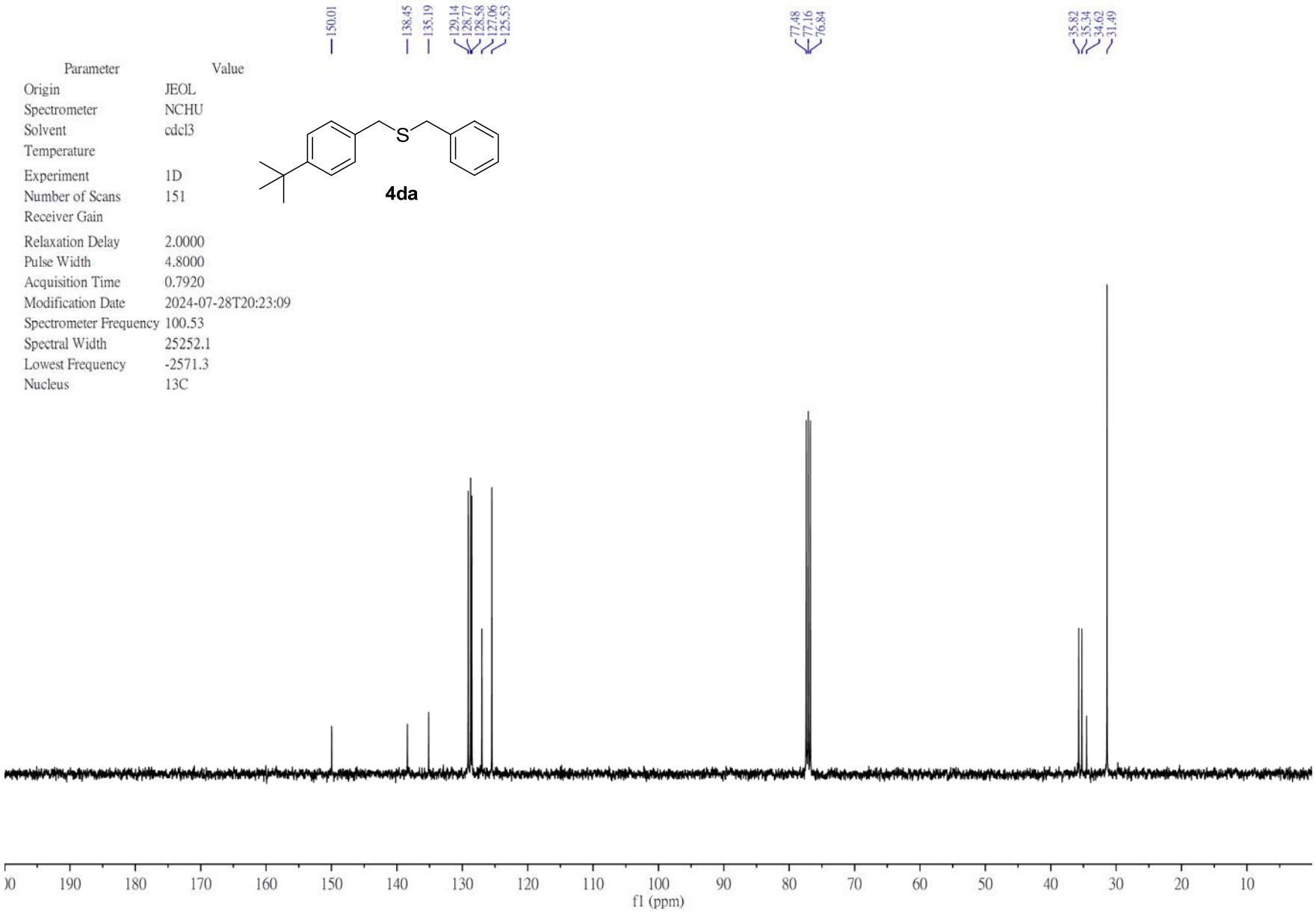
4ca ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



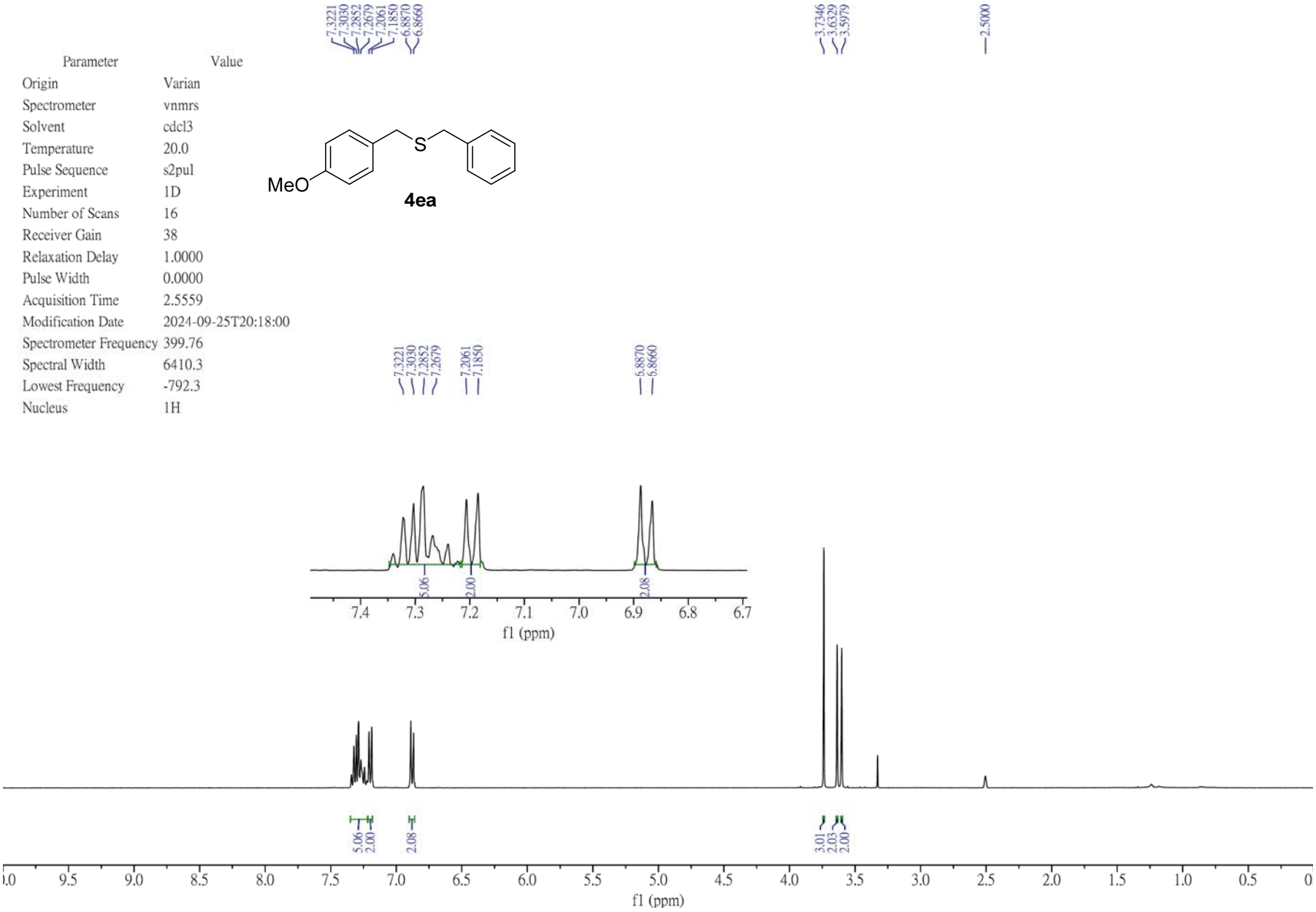
4ca $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



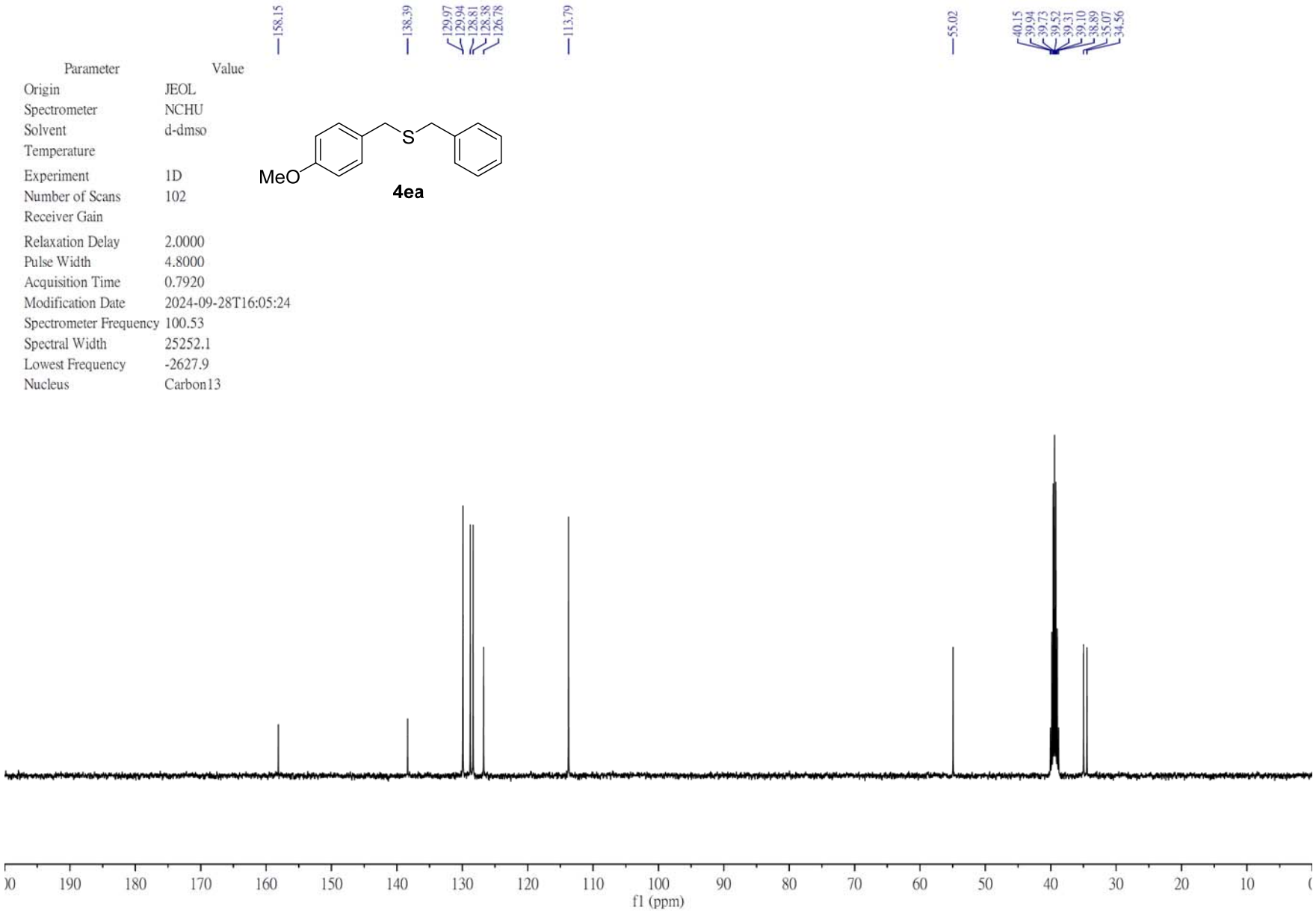
4da ^{1}H NMR spectrum（400 MHz in $(CD_3)_2SO$）



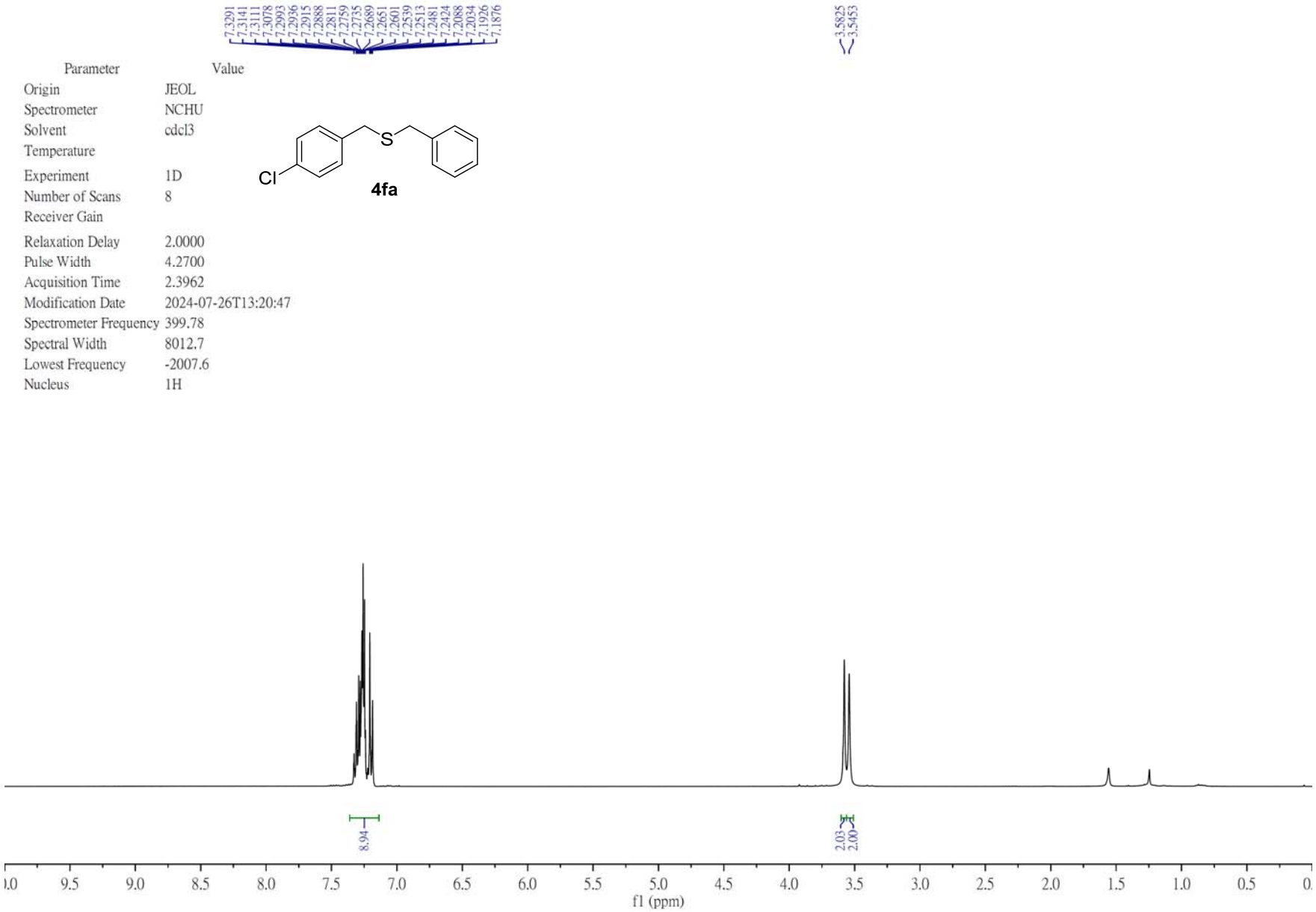
4da $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



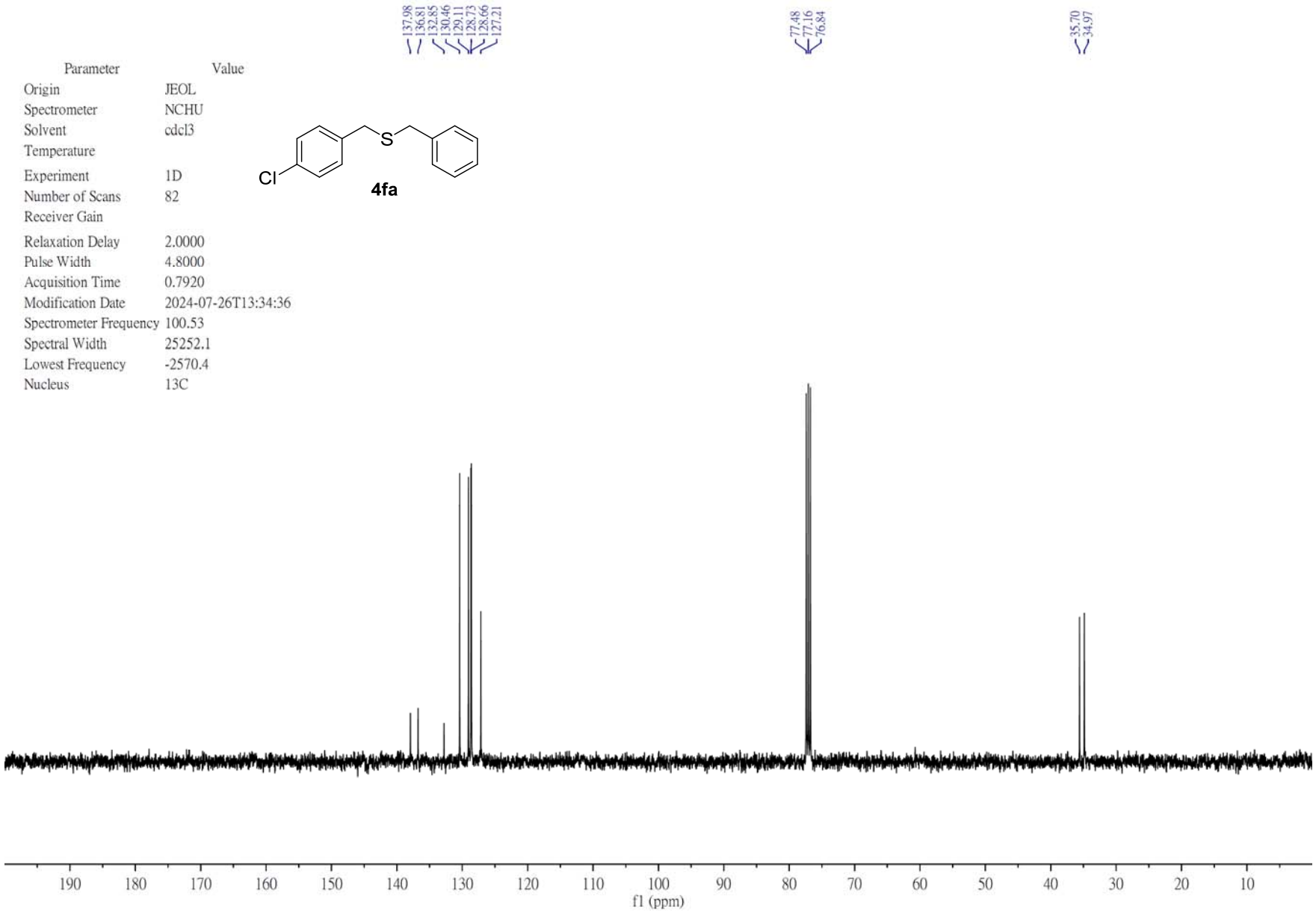
4ea ^{1}H NMR spectrum（400 MHz in $(CD_3)_2SO$）



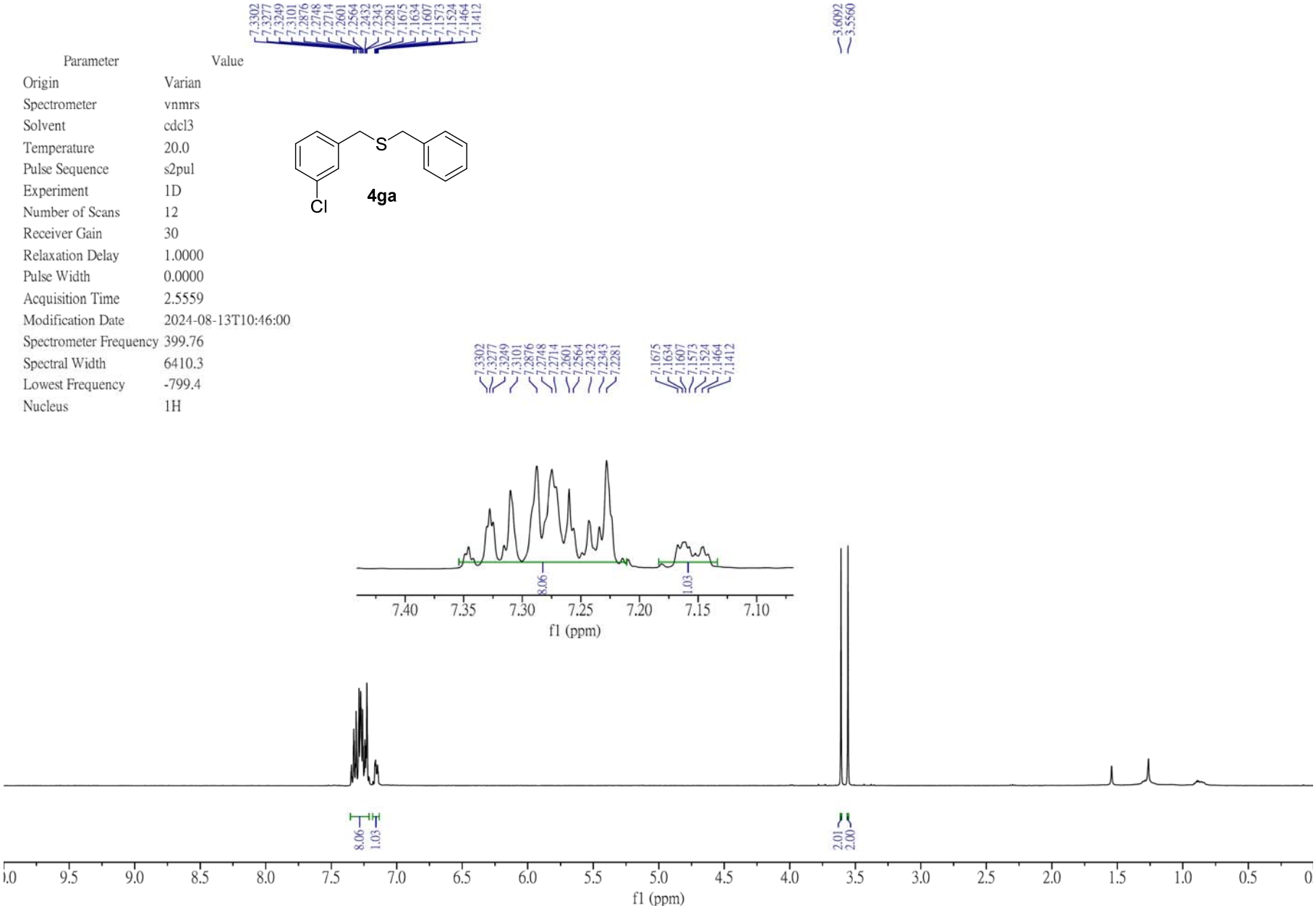
4ea $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $(CD_3)_2SO$）



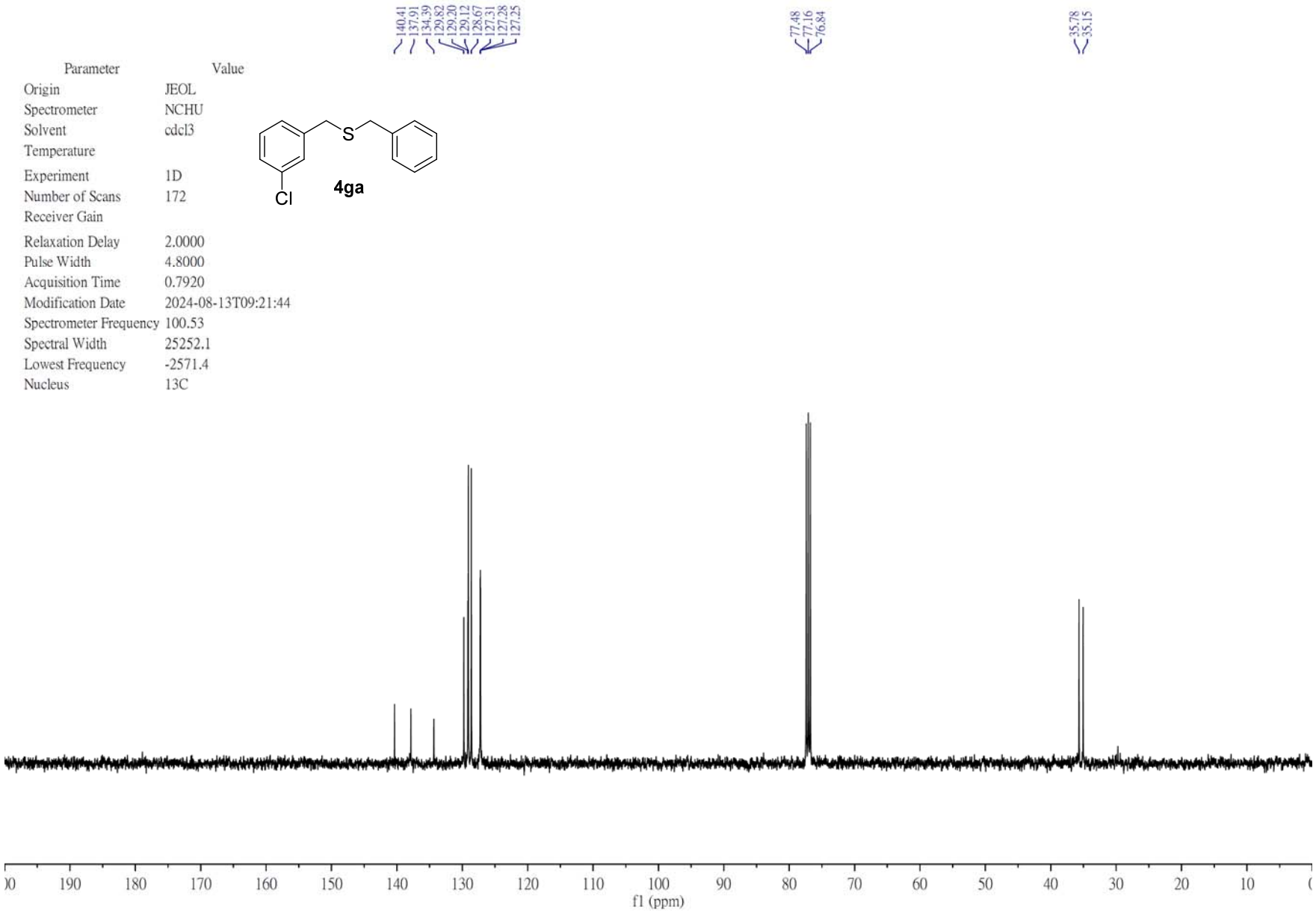
4fa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



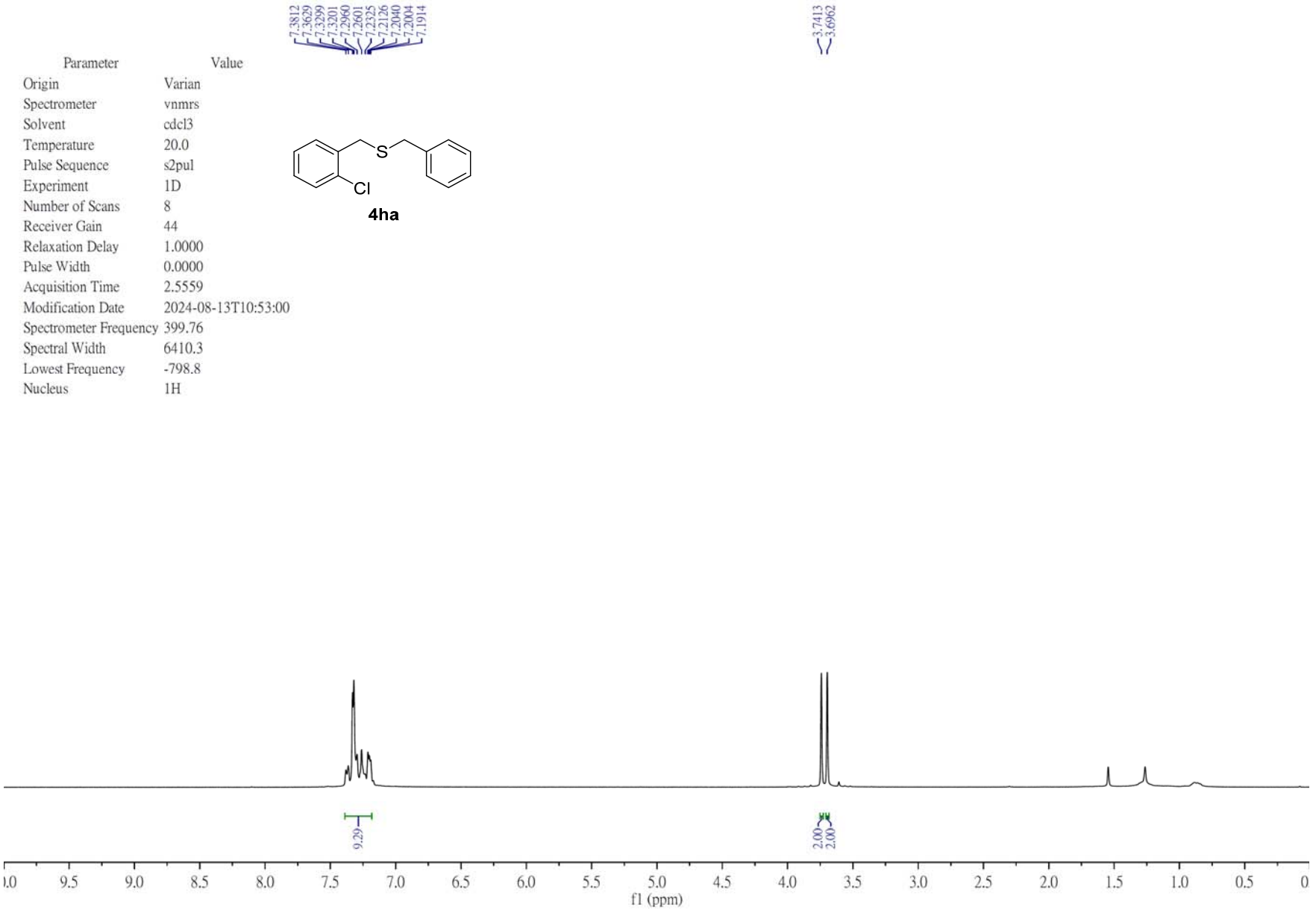
4fa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



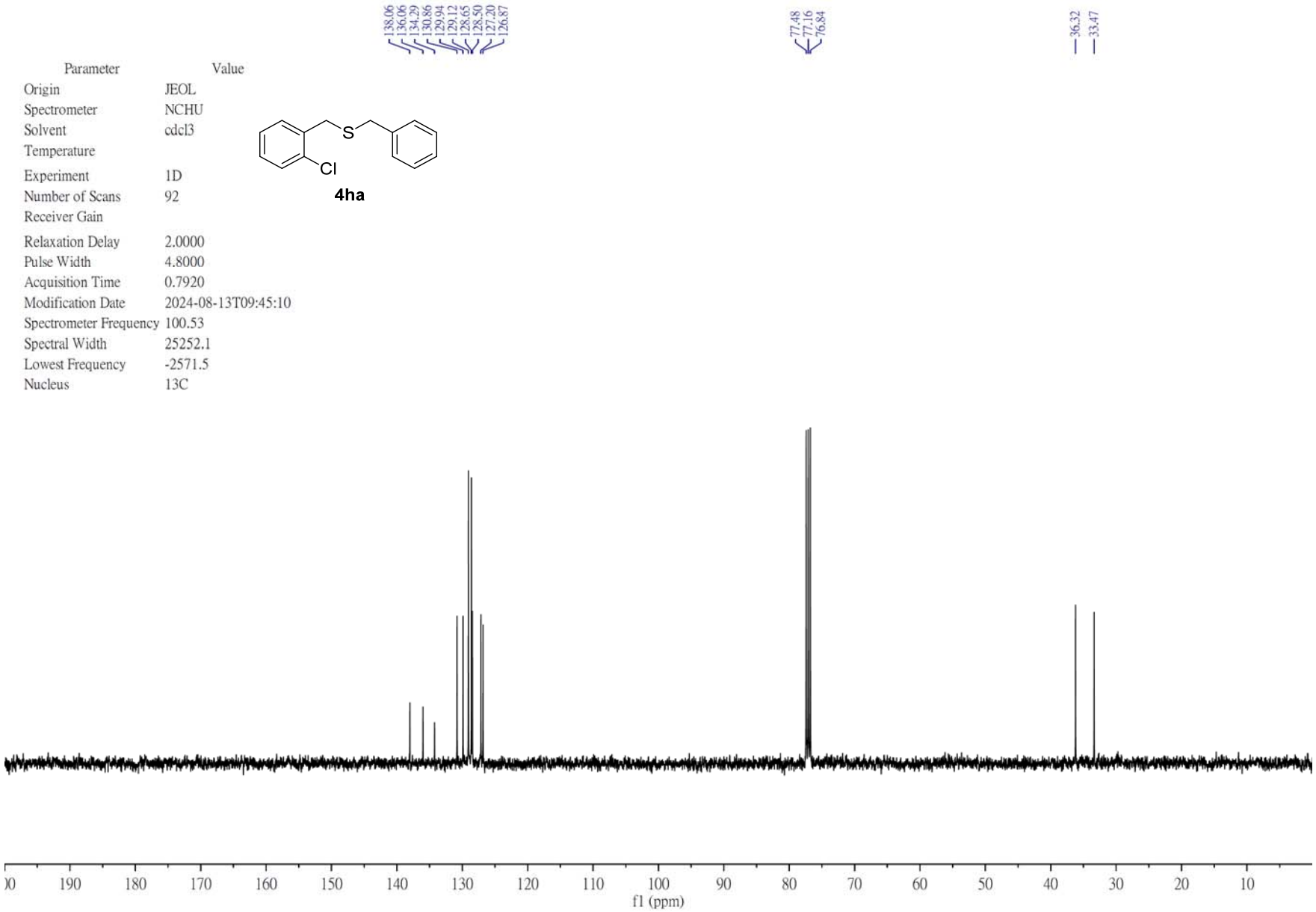
4ga ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4ga $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



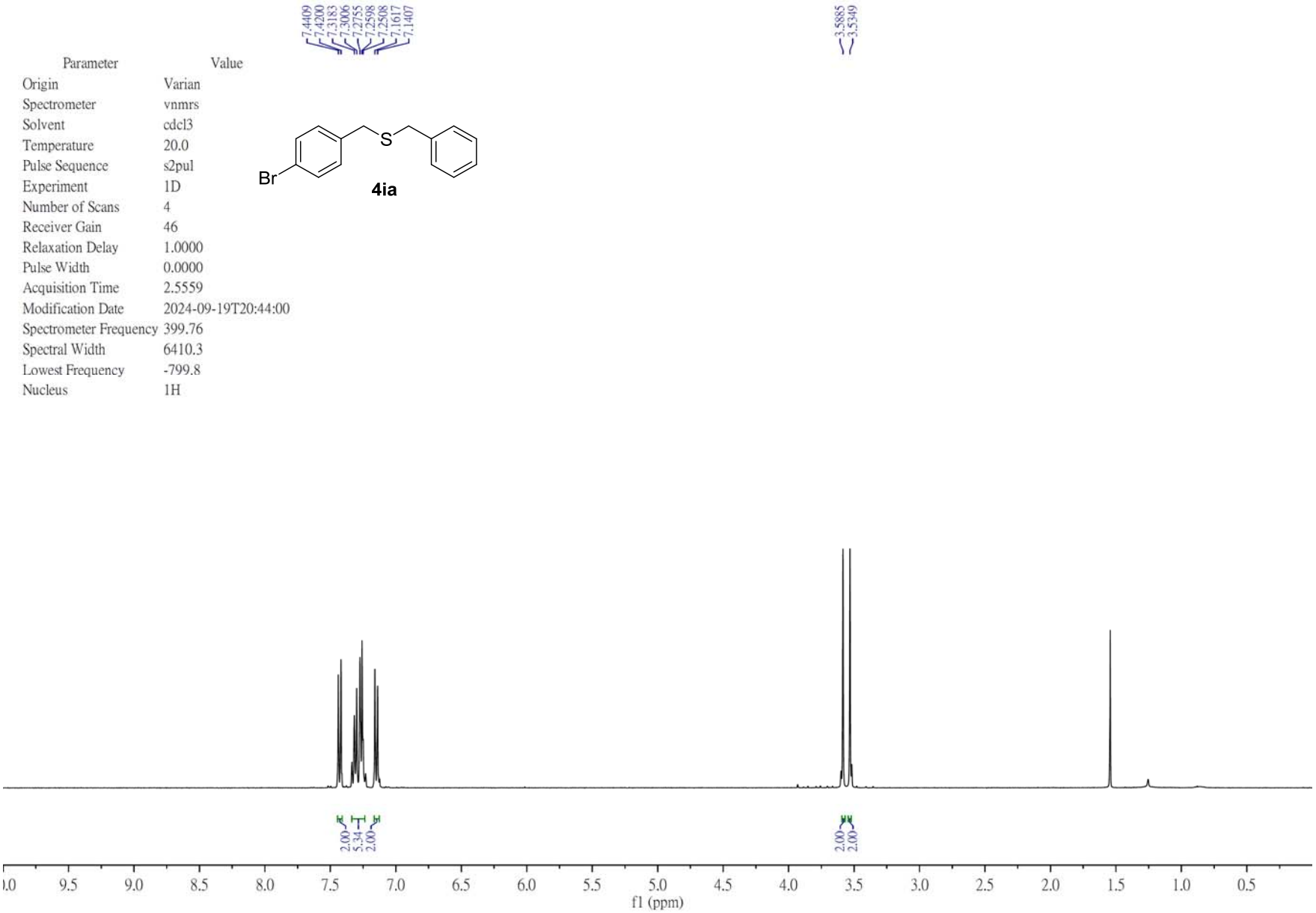
4ha ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



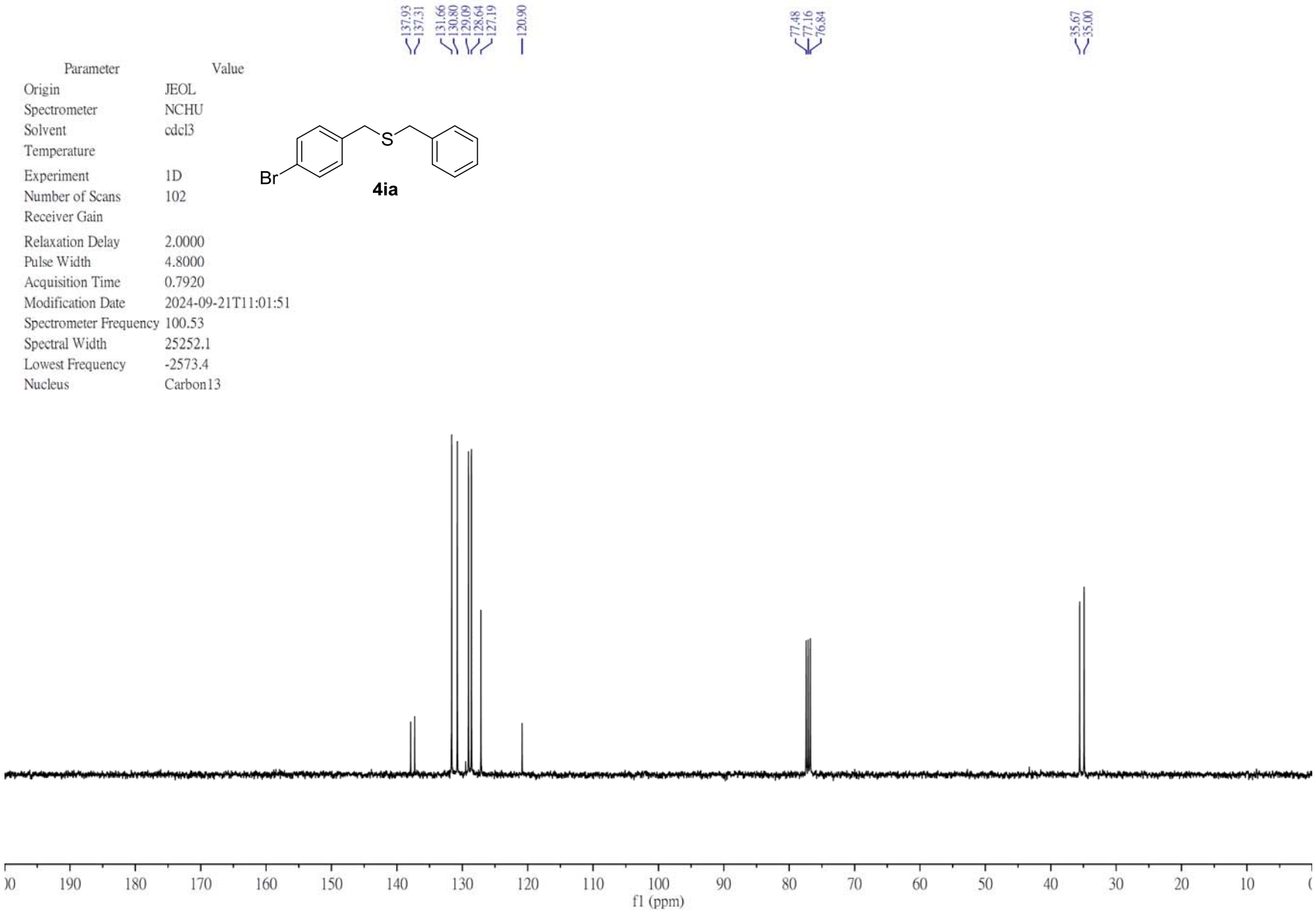
4ha $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	46
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Modification Date	2024-09-19T20:44:00
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.8
Nucleus	1H

4ia



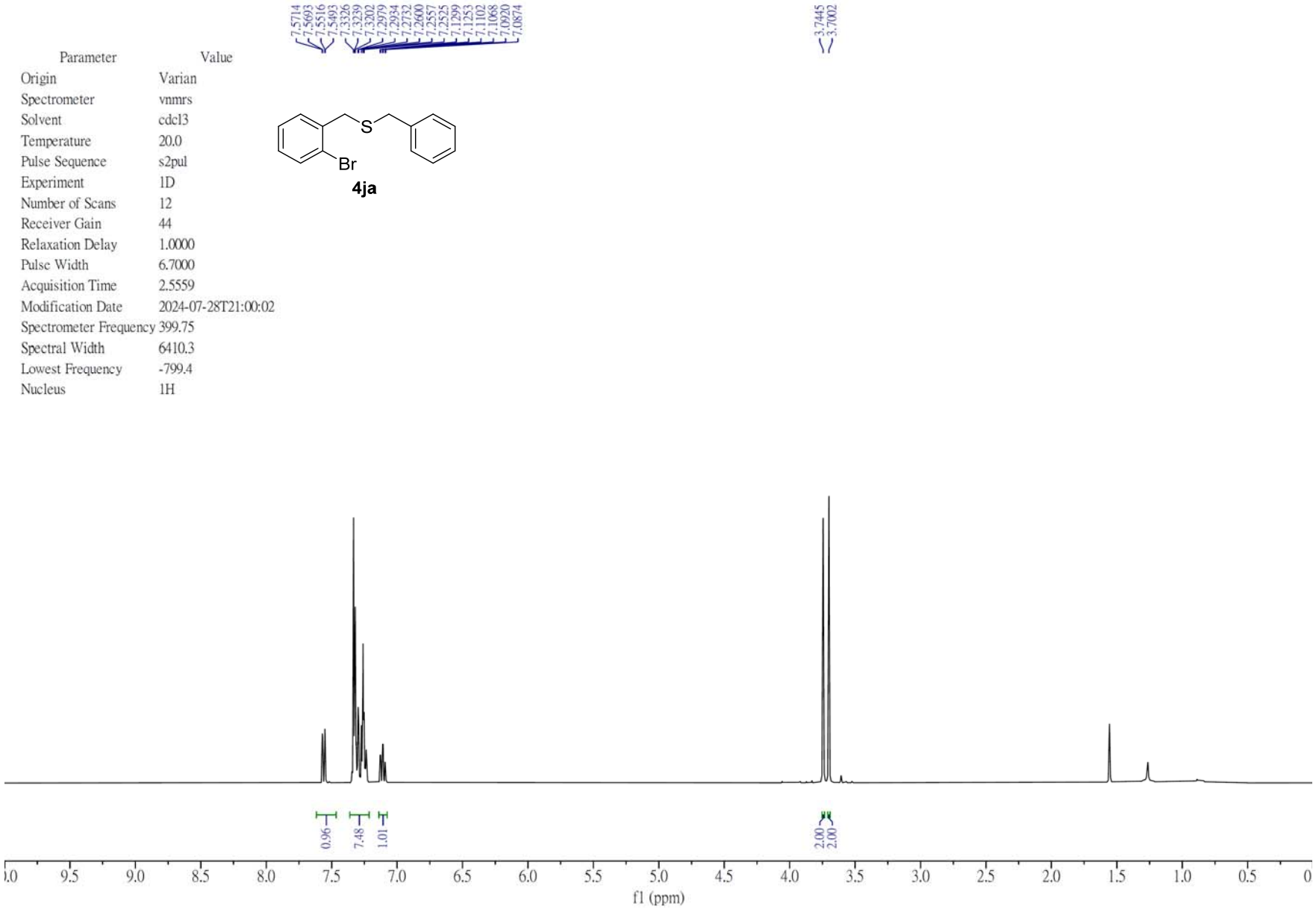
4ia ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



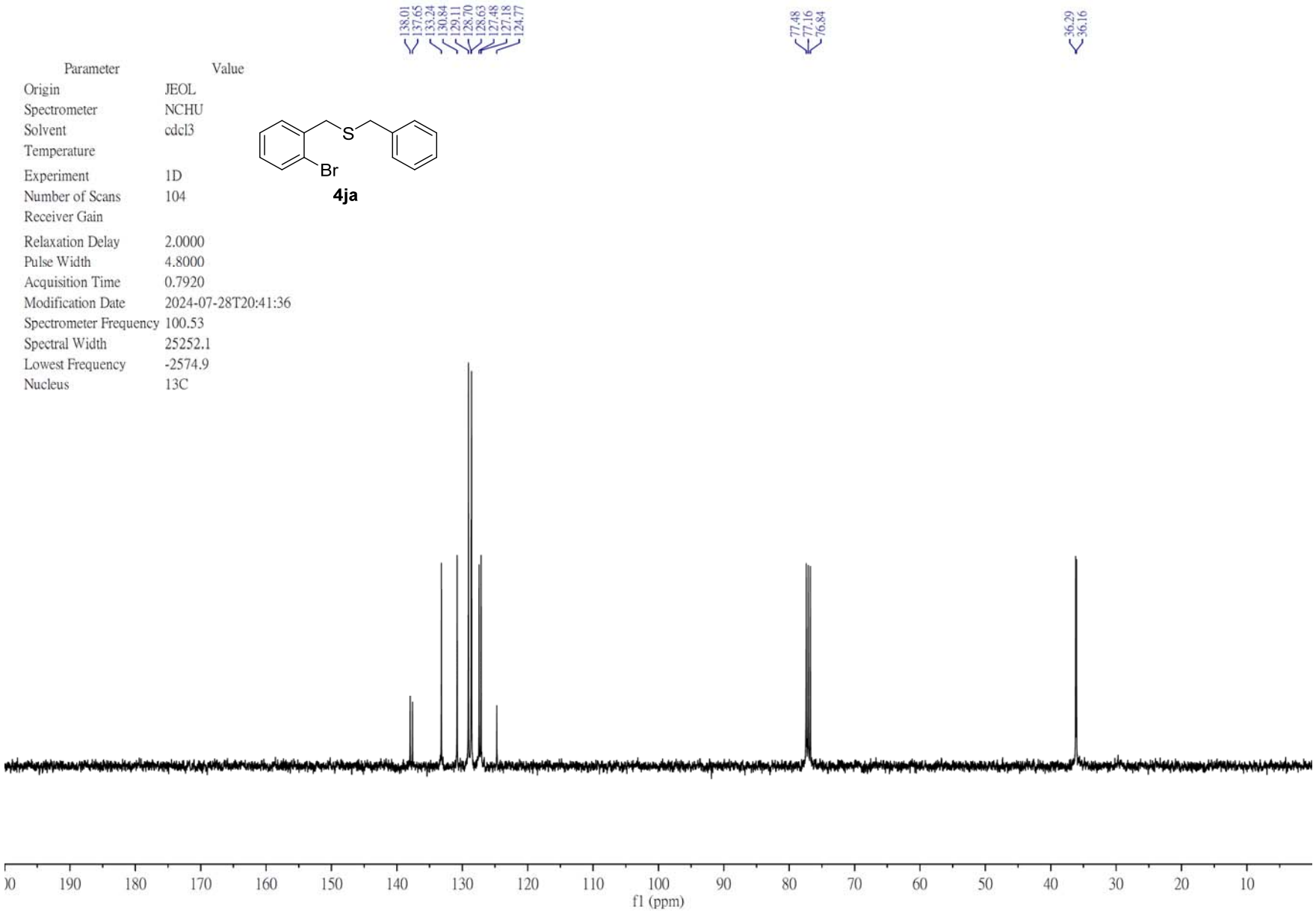
4ia $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

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	6.7000
Acquisition Time	2.5559
Modification Date	2024-07-28T21:00:02
Spectrometer Frequency	399.75
Spectral Width	6410.3
Lowest Frequency	-799.4
Nucleus	1H

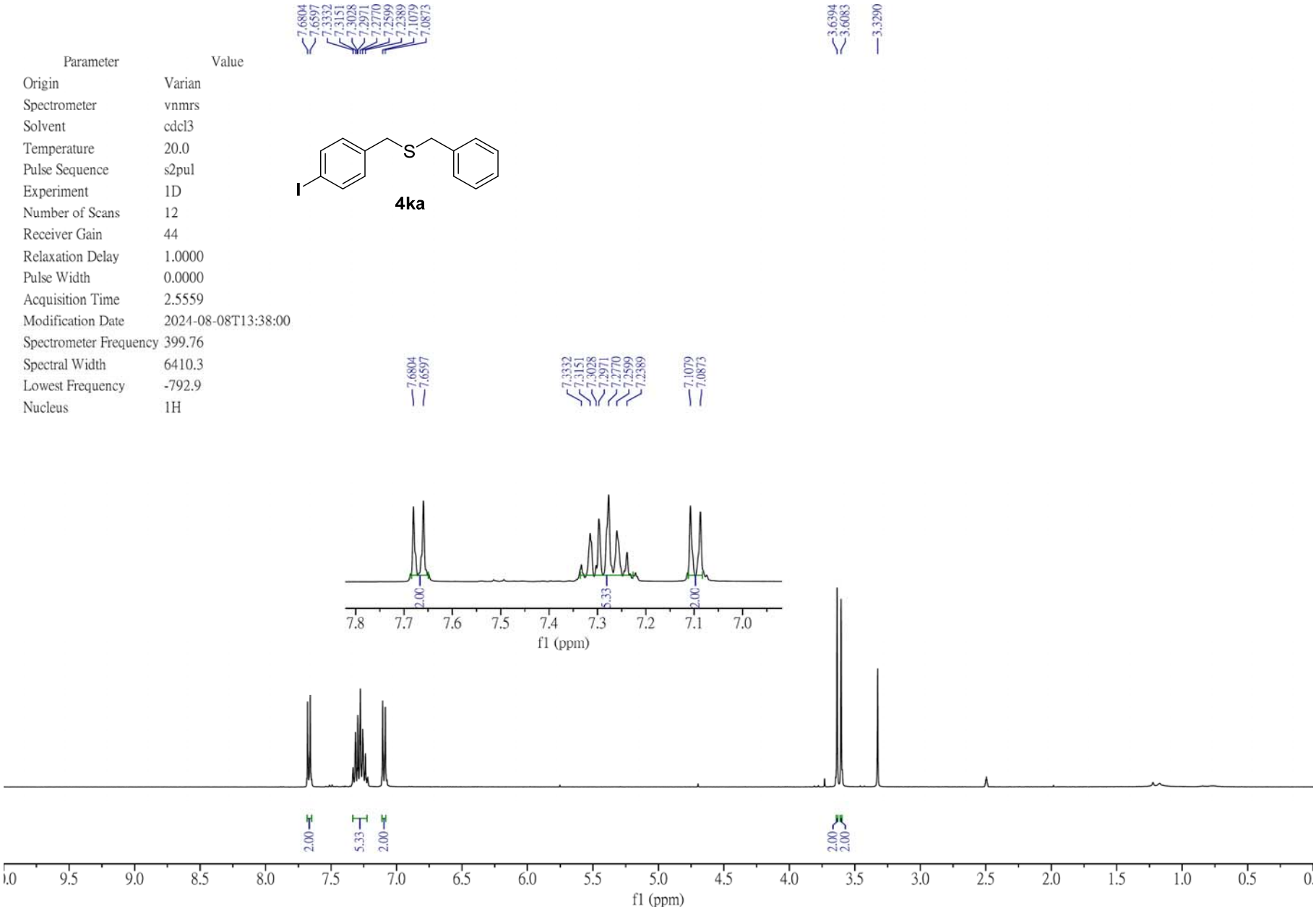
4ja



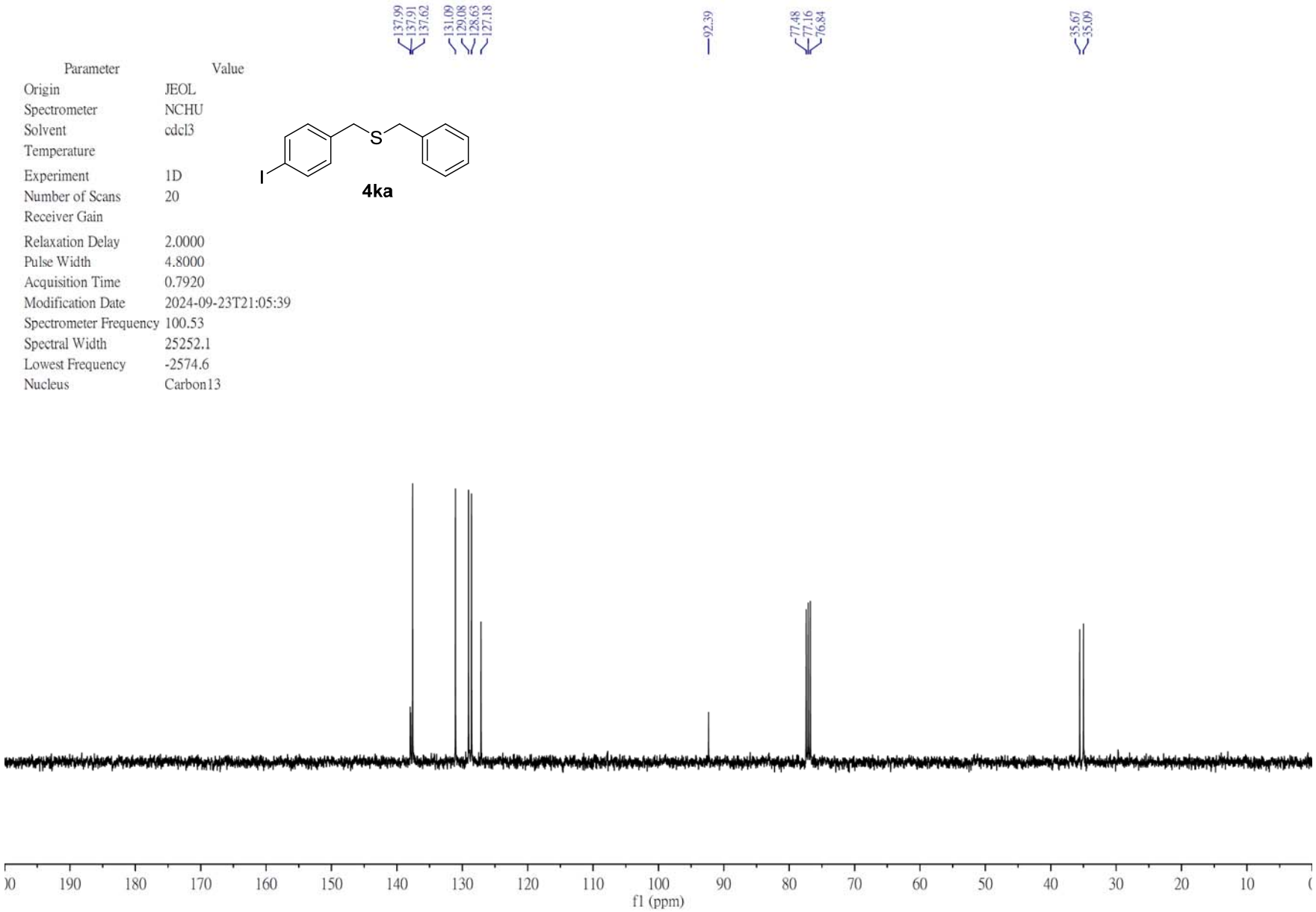
4ja ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



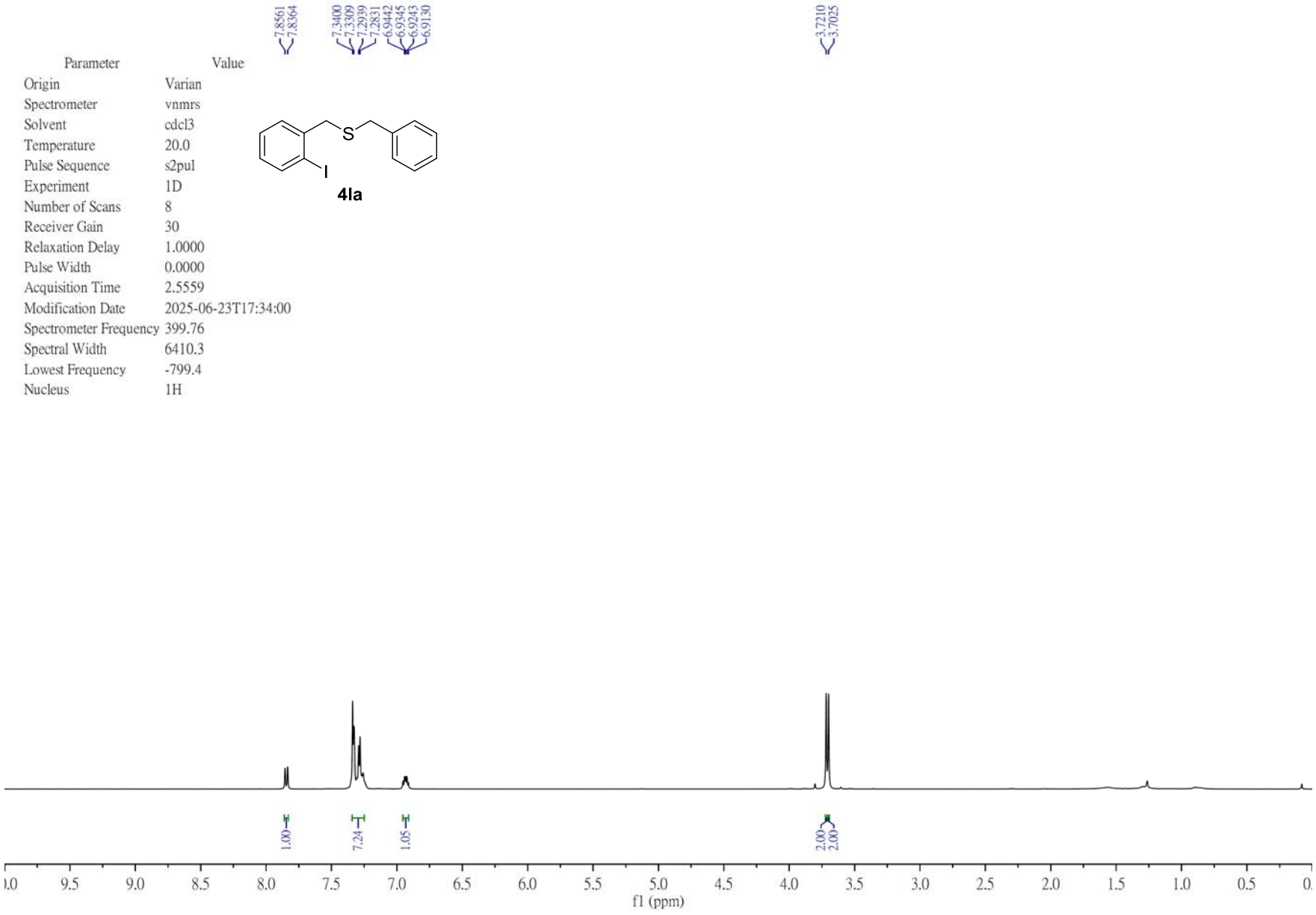
4ja $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



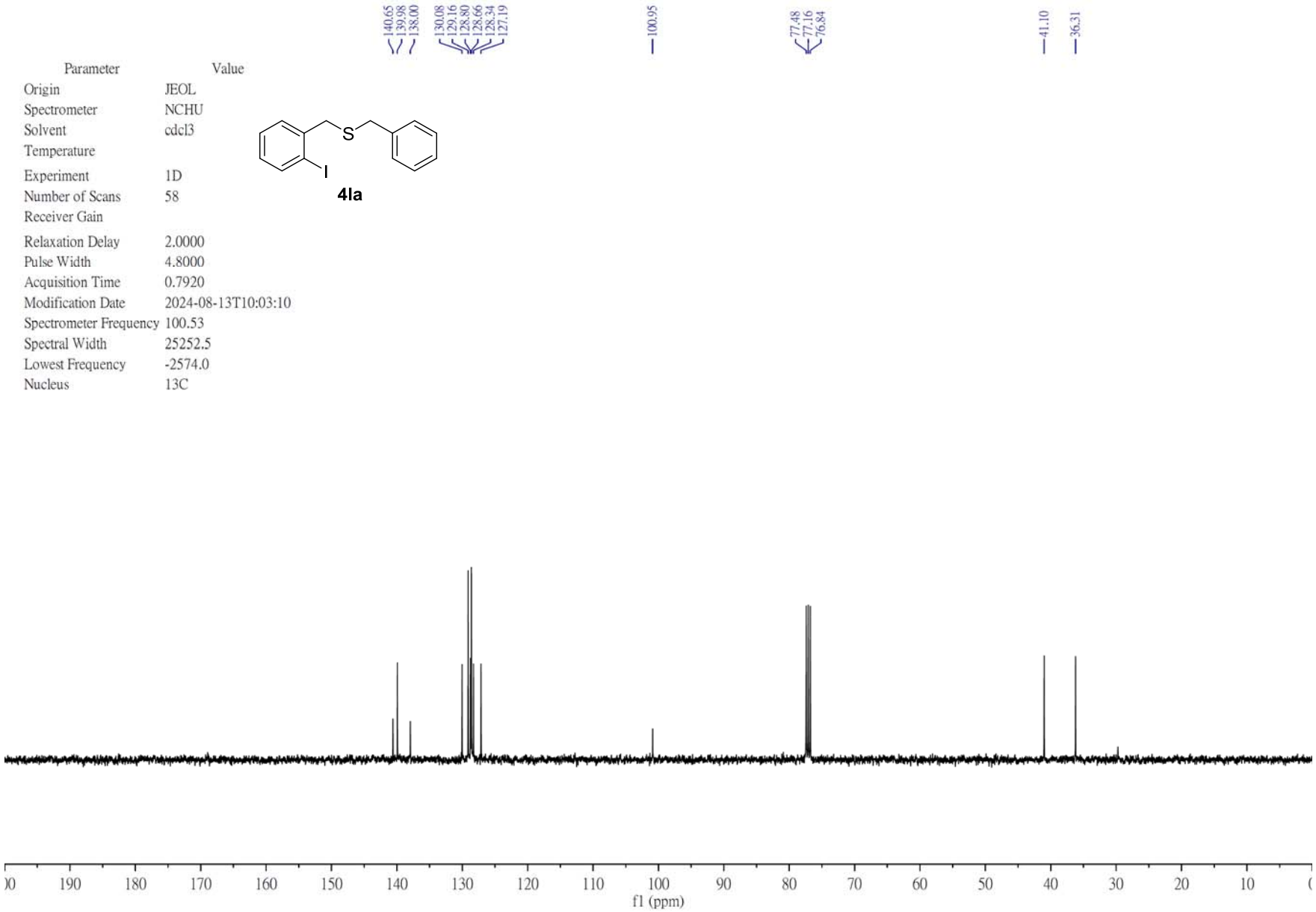
4ka ^{1}H NMR spectrum（400 MHz in $(CD_3)_2SO$）



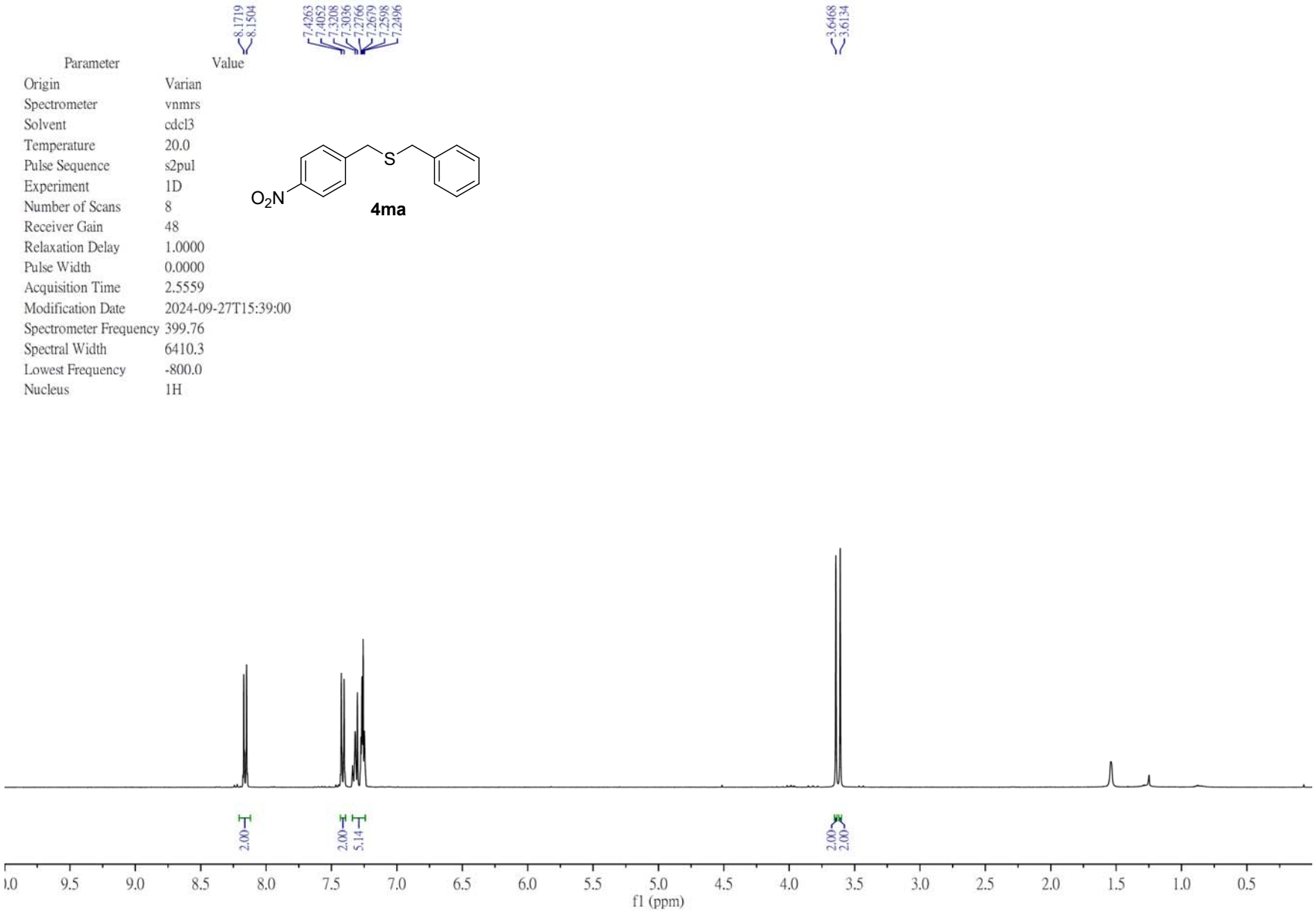
4ka $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



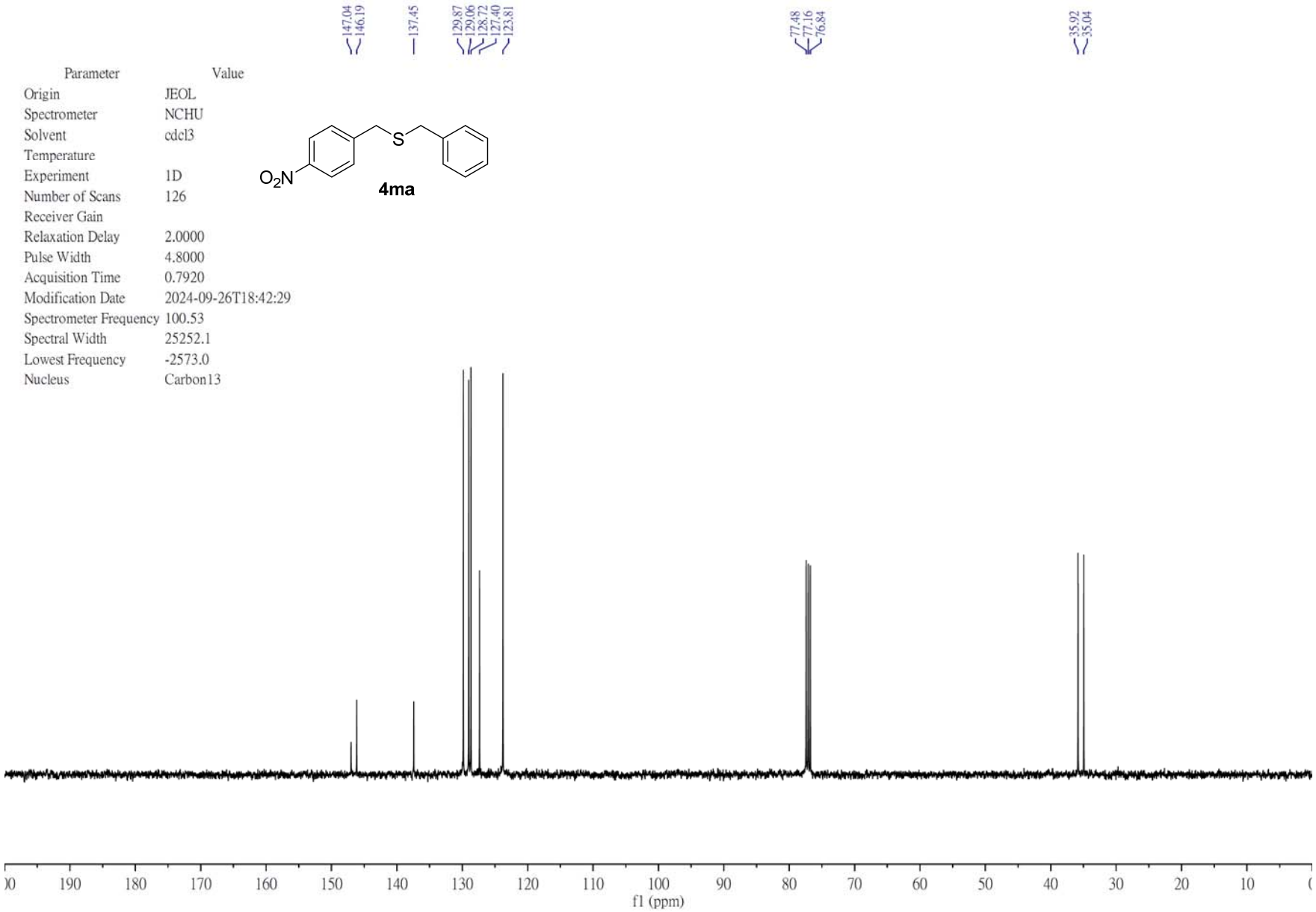
4la ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



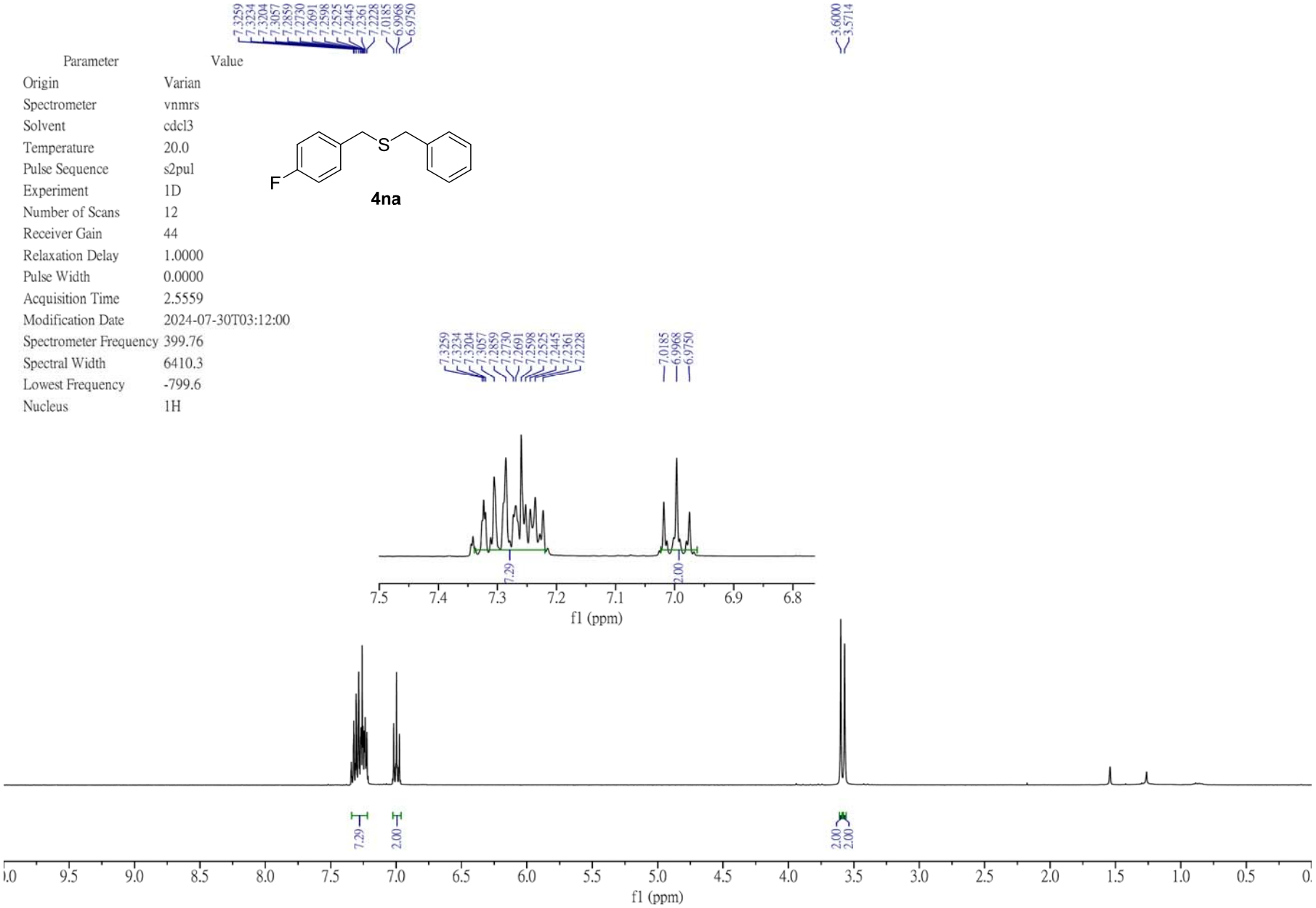
4la $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



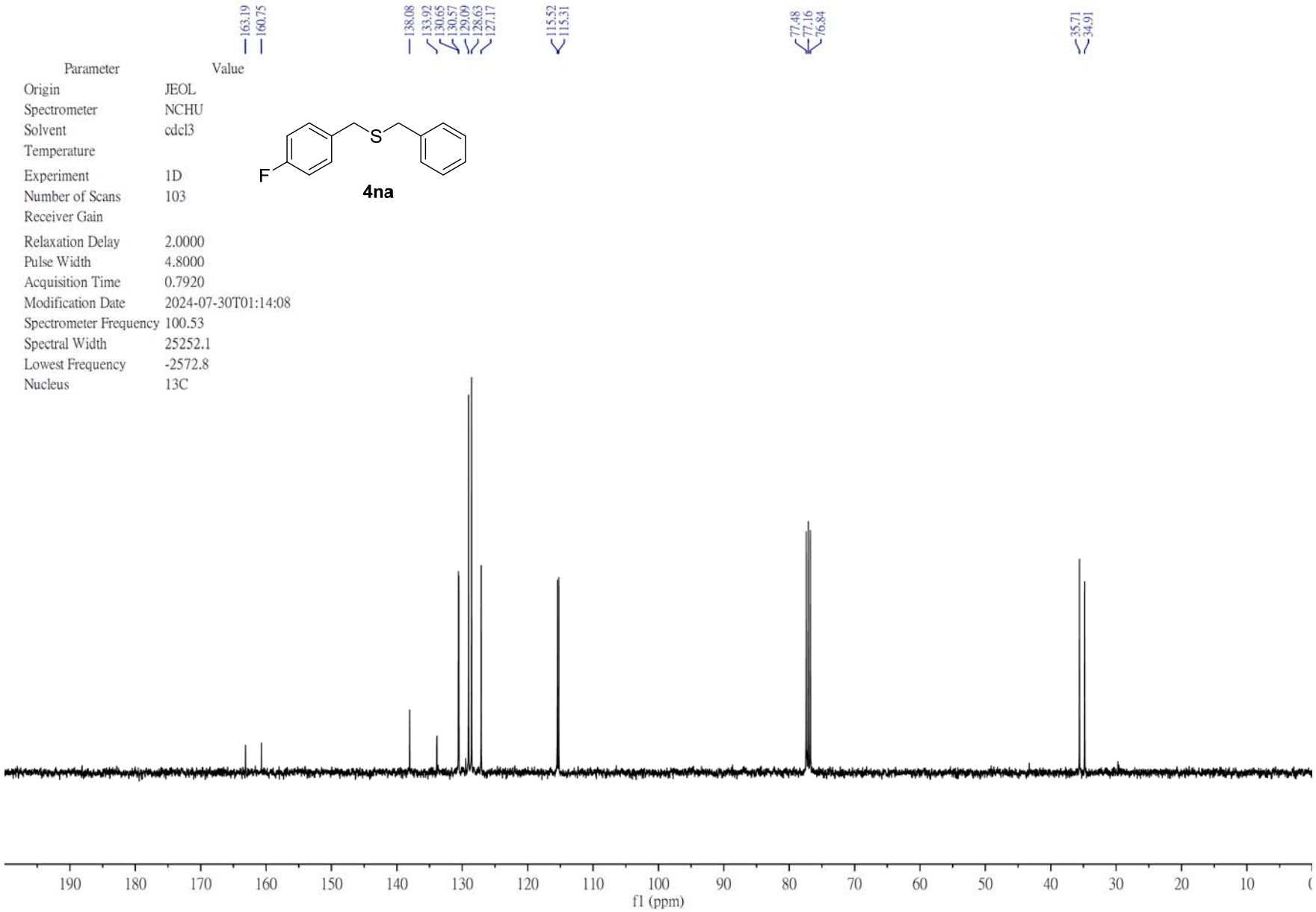
4ma ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4ma $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

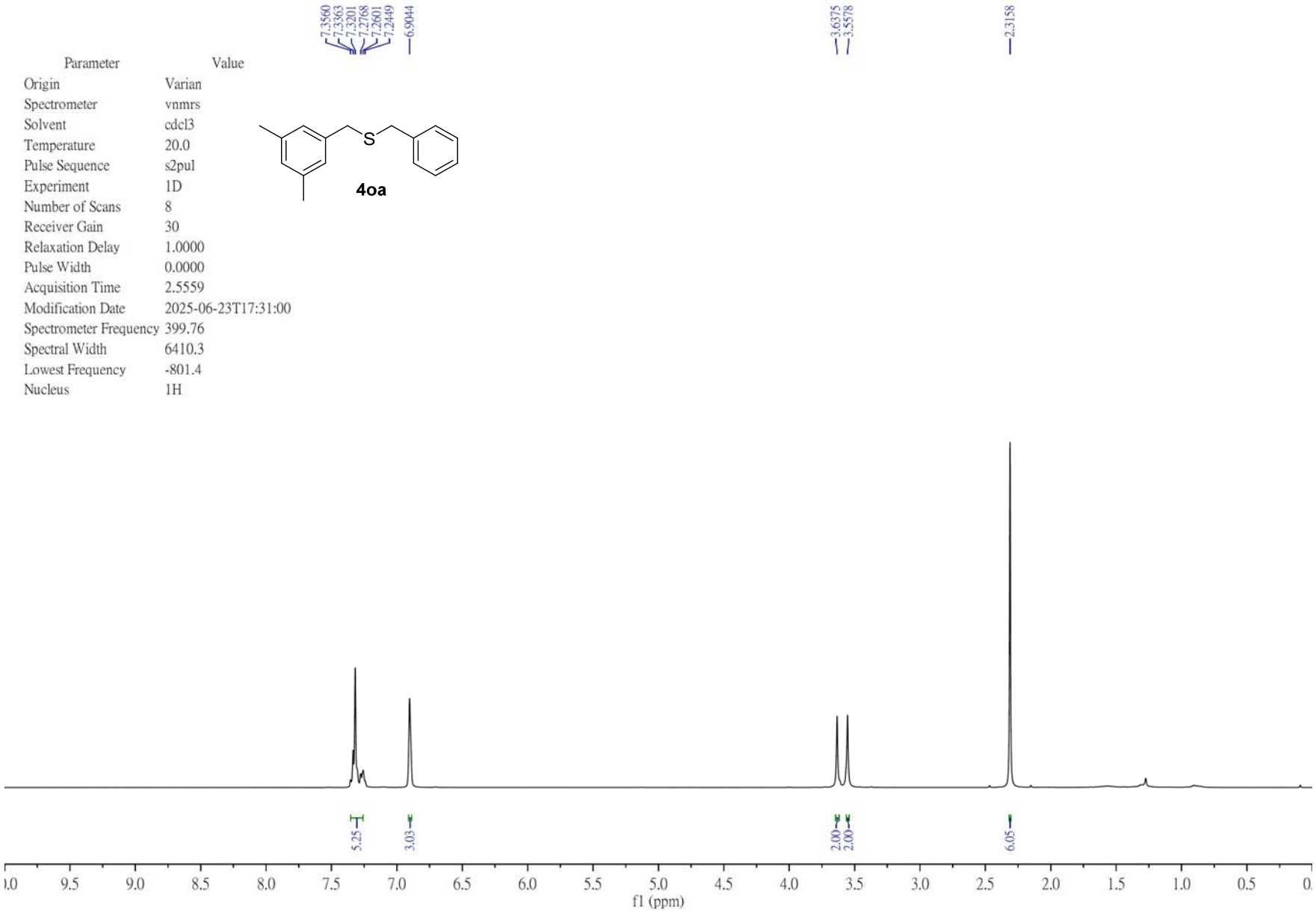


4na ^{1}H NMR spectrum（400 MHz in $CDCl_3$）

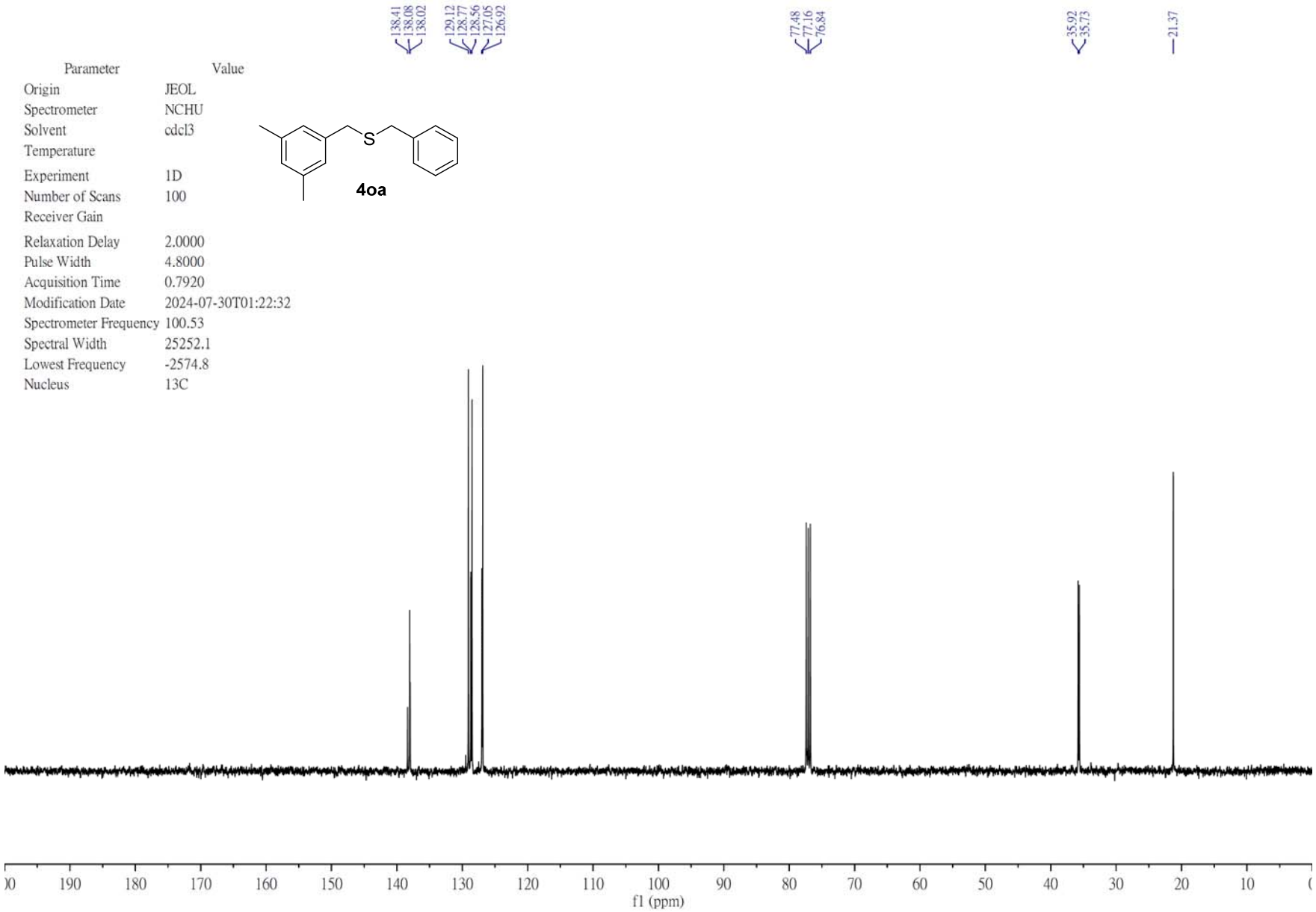


4na $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

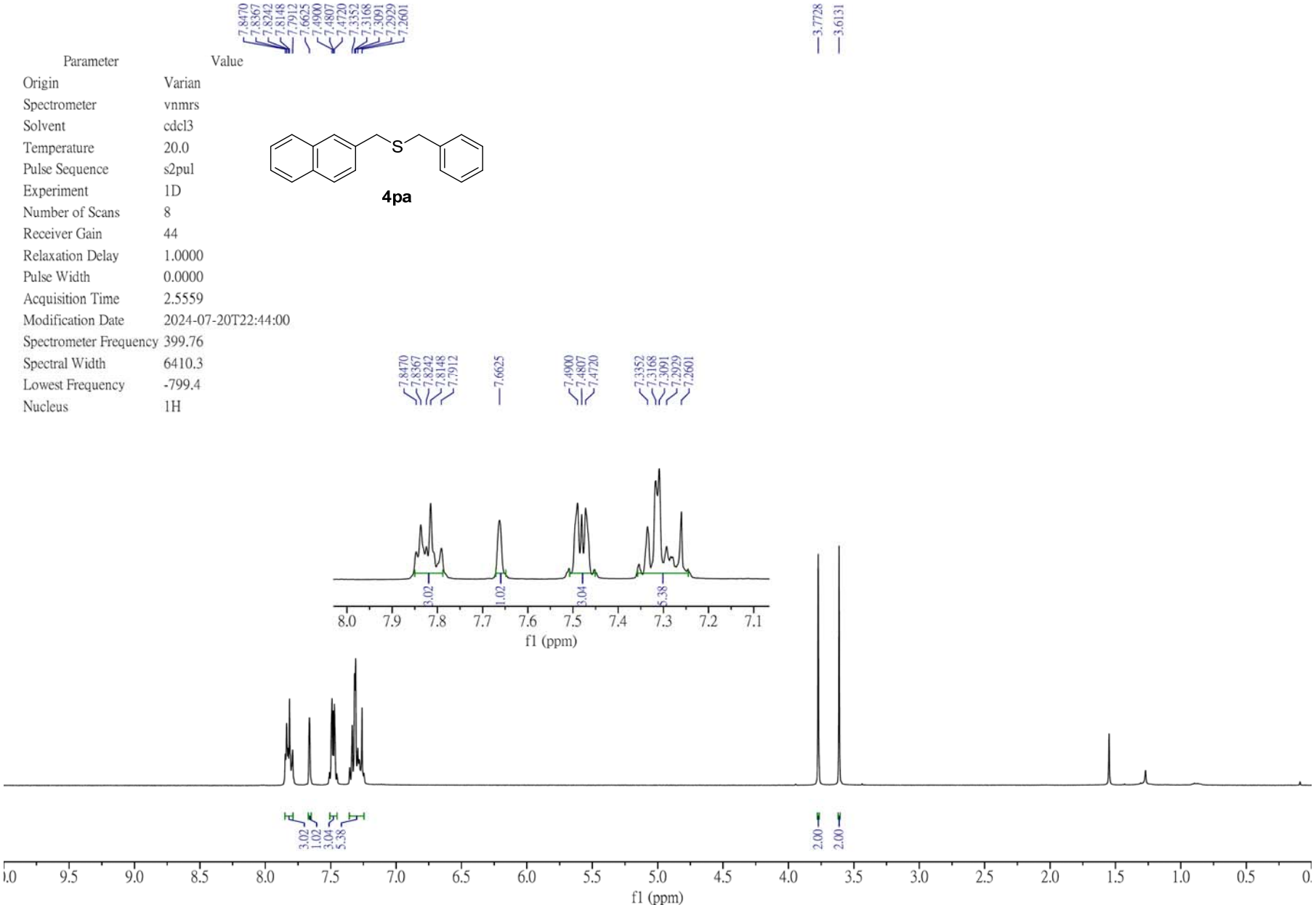
4na ^{19}F NMR spectrum（376MHz in $CDCl_3$）



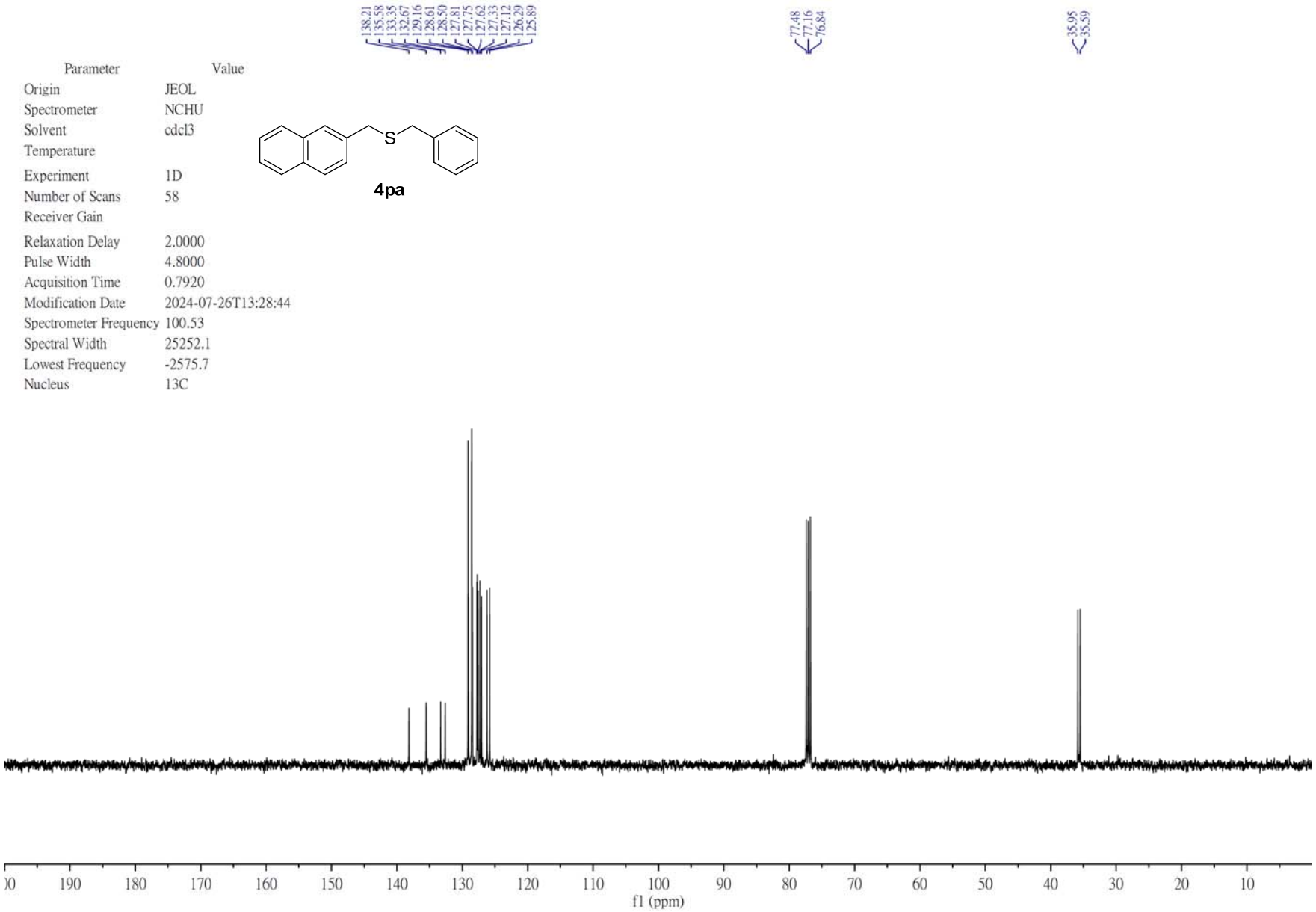
4oa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



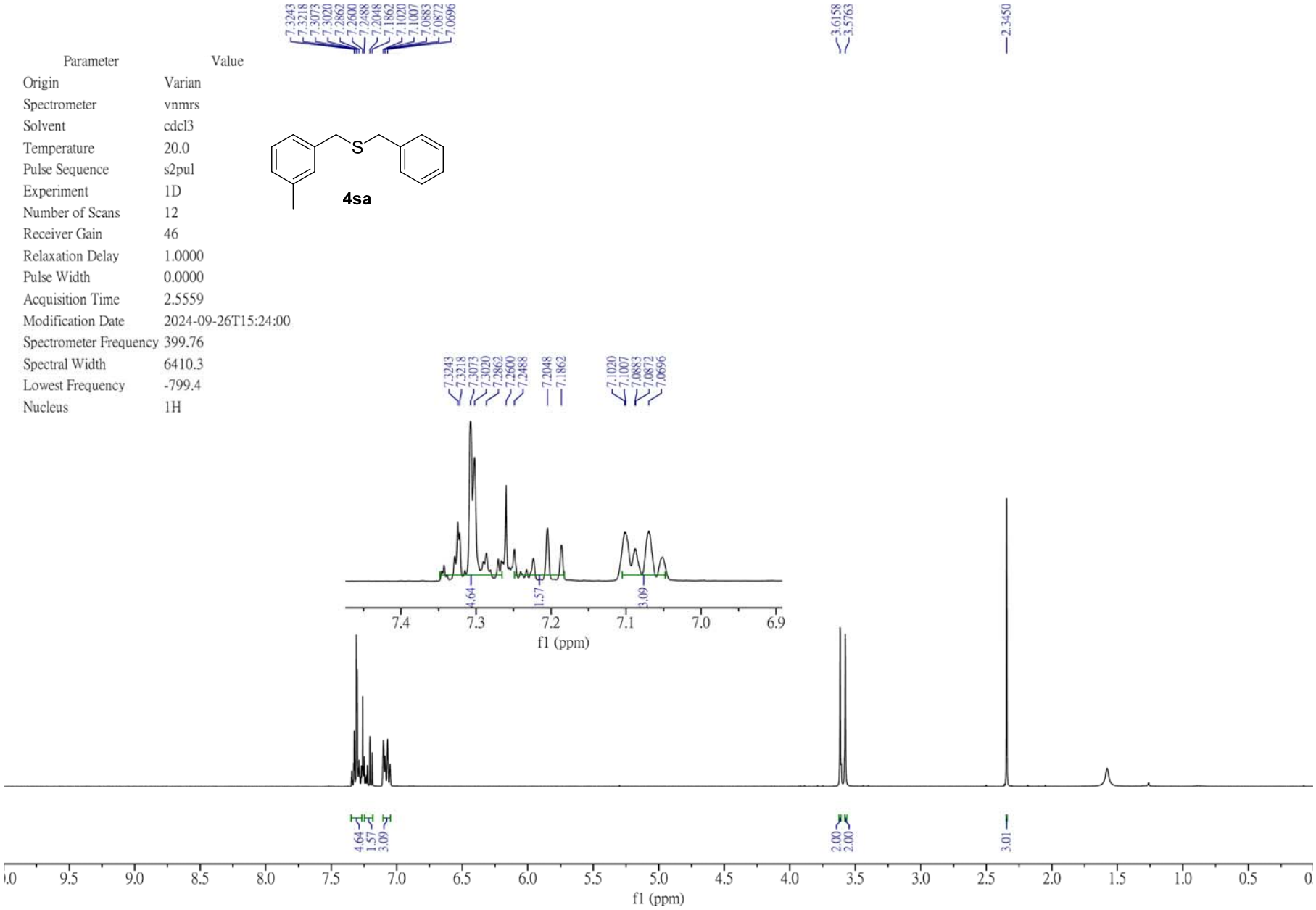
4oa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



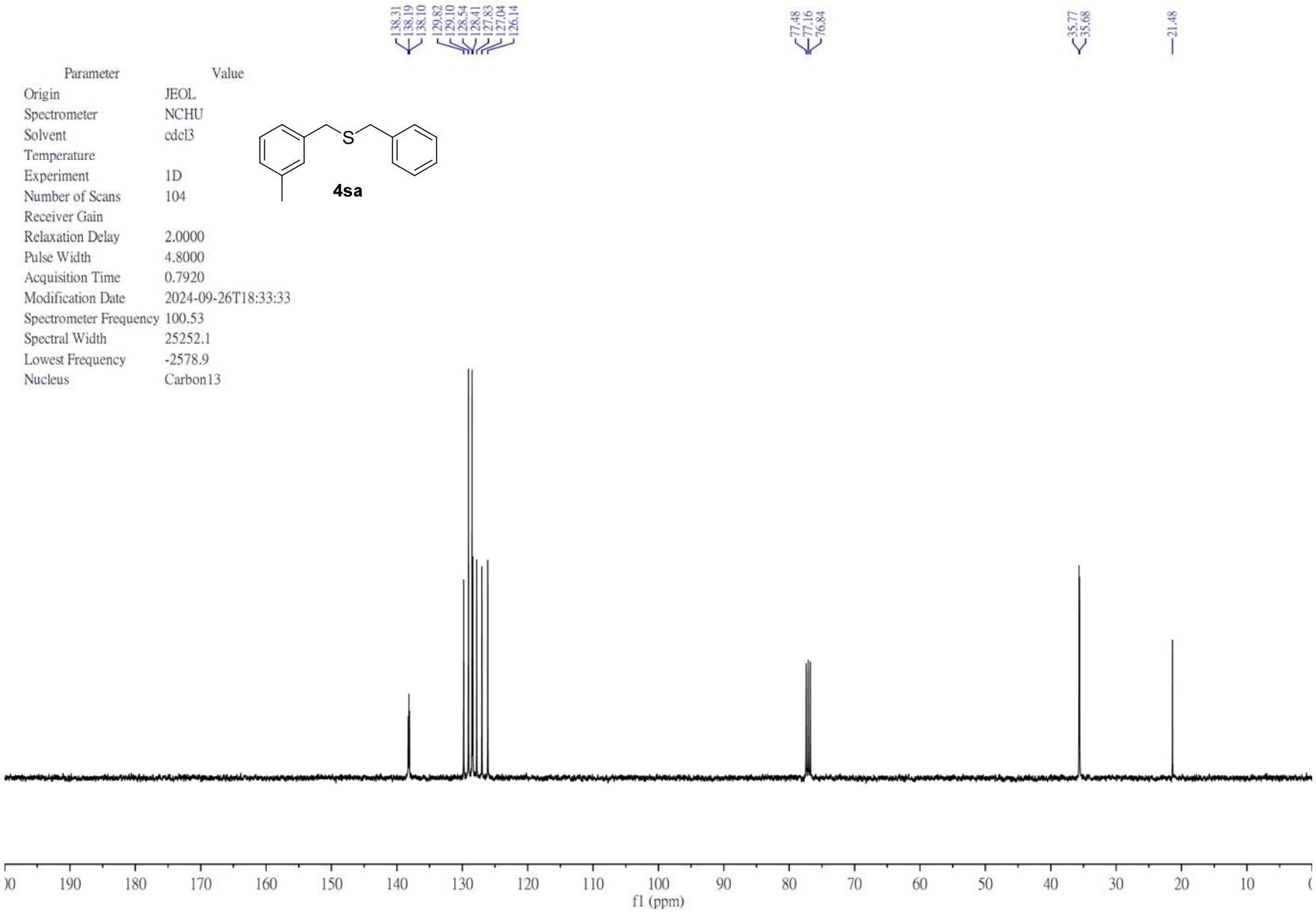
4pa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



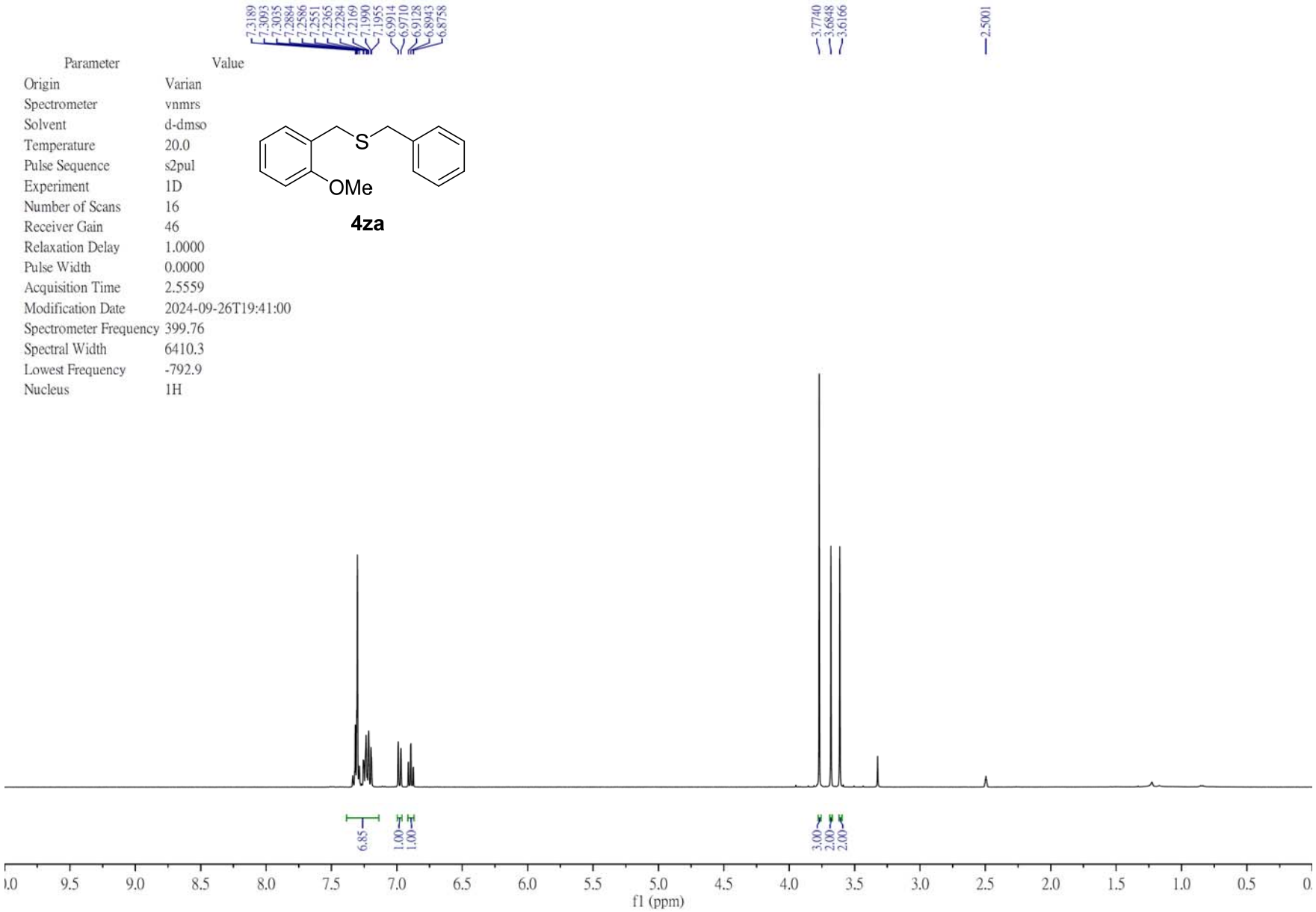
4pa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



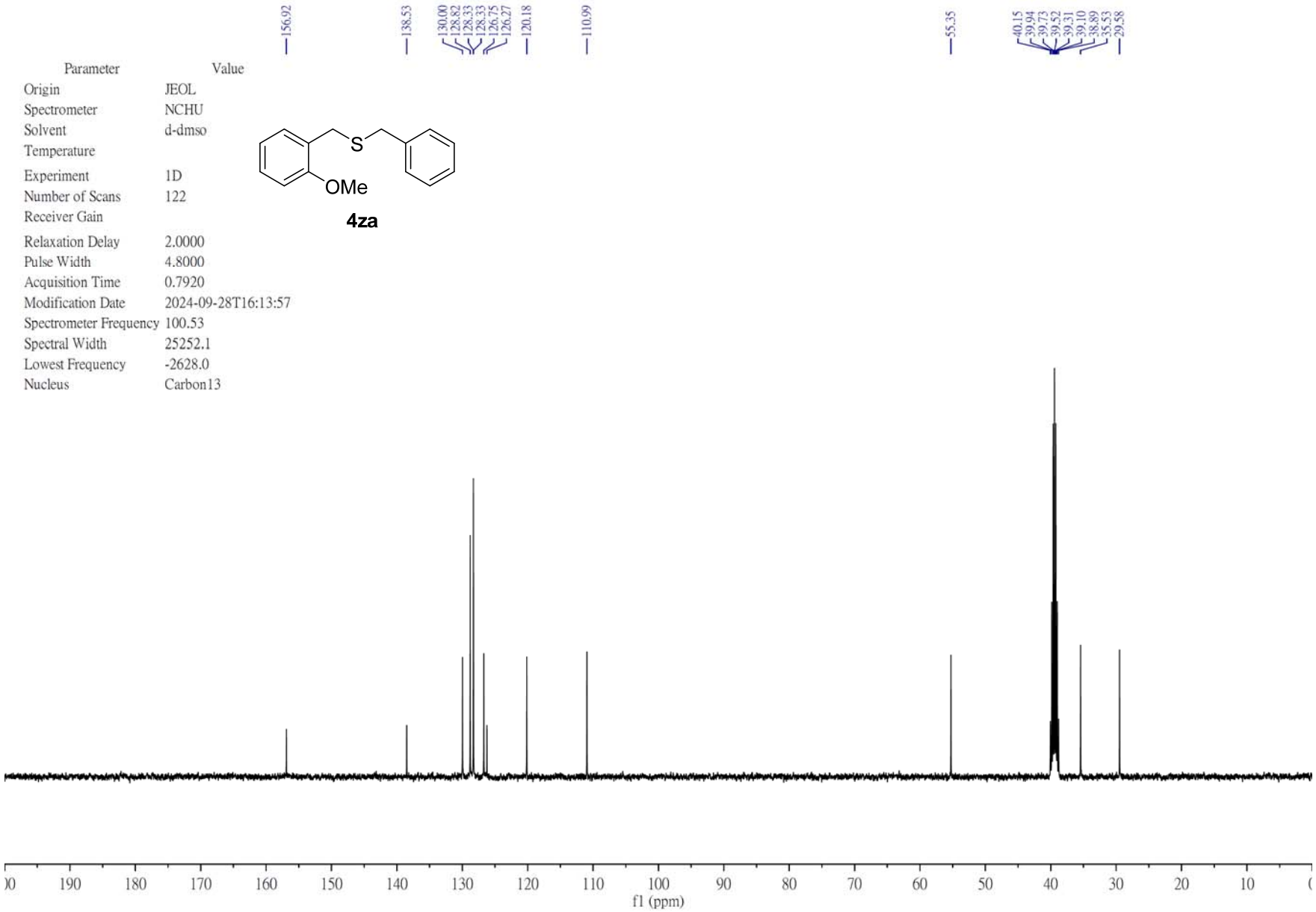
4sa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



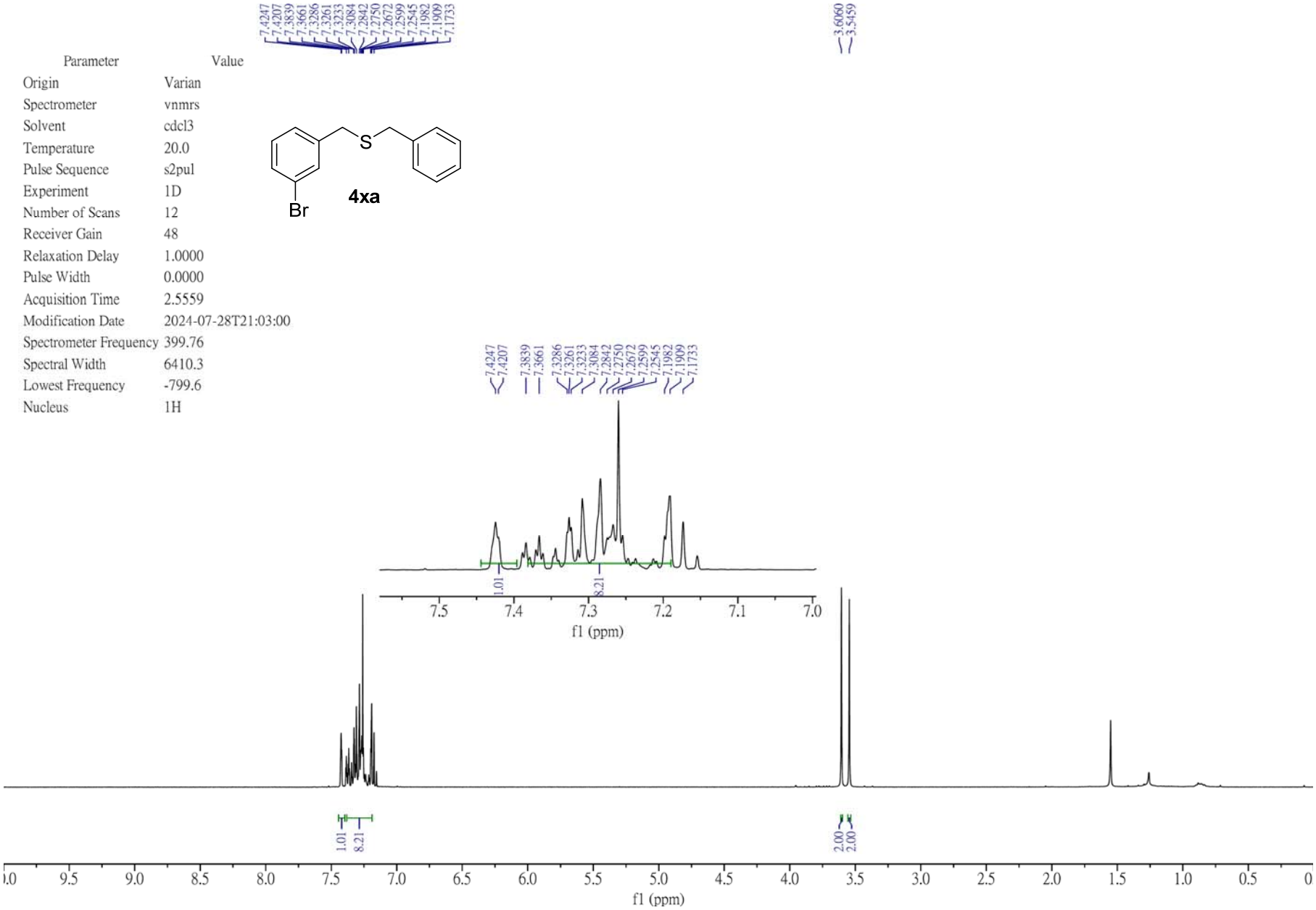
4sa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



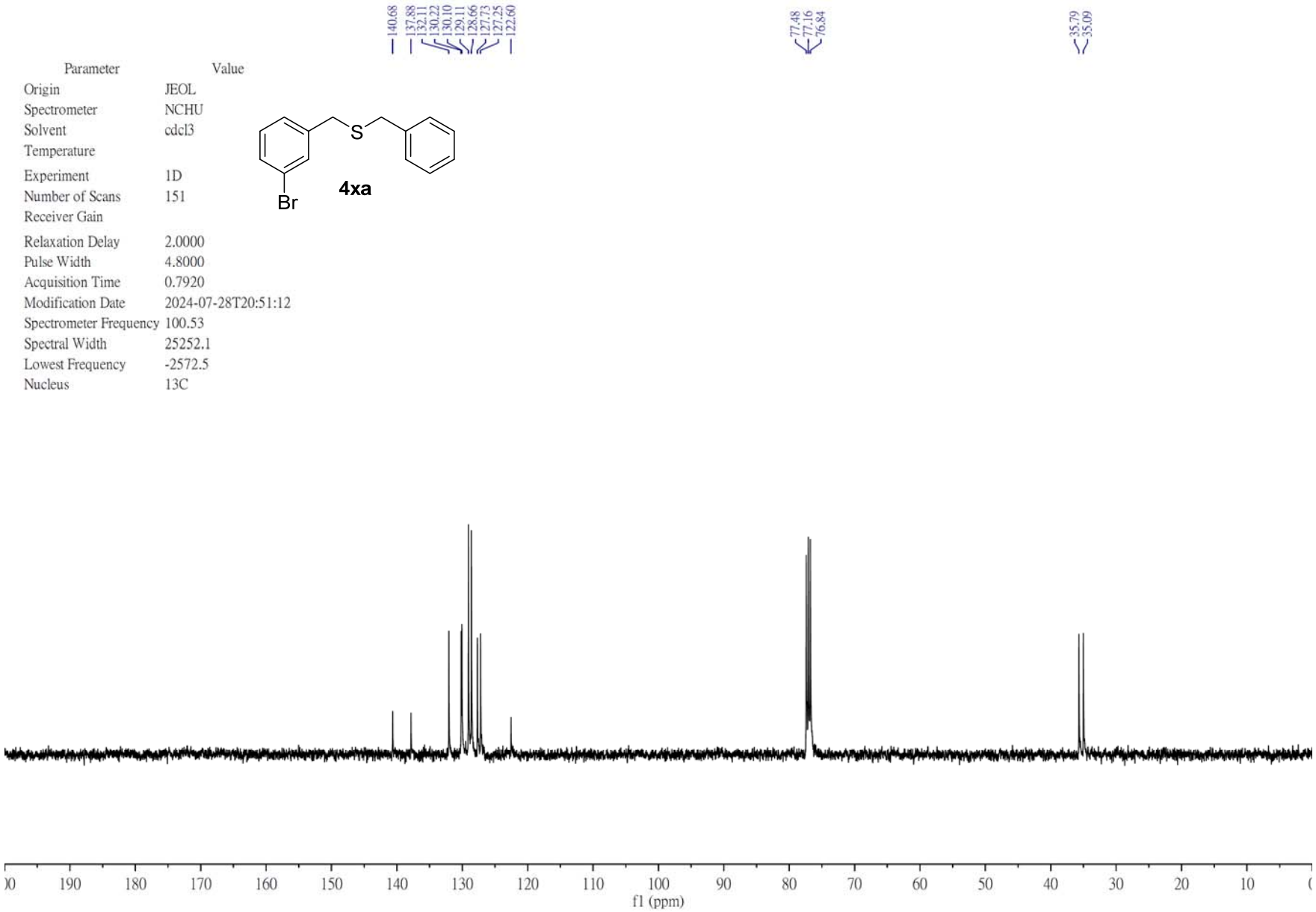
4za ^{1}H NMR spectrum（400 MHz in $(CD_3)_2SO$）



4za $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $(CD_3)_2SO$）



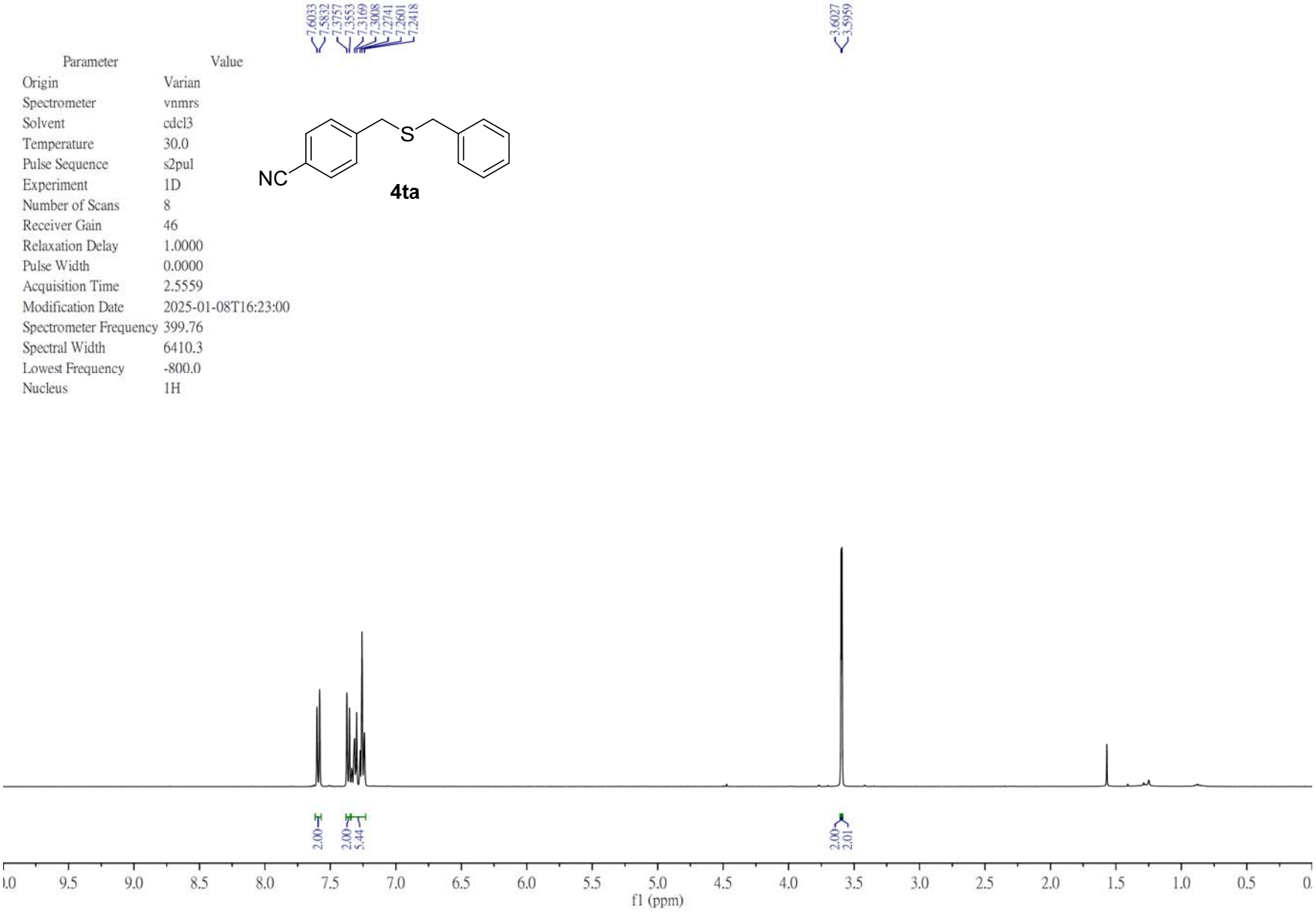
4xa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



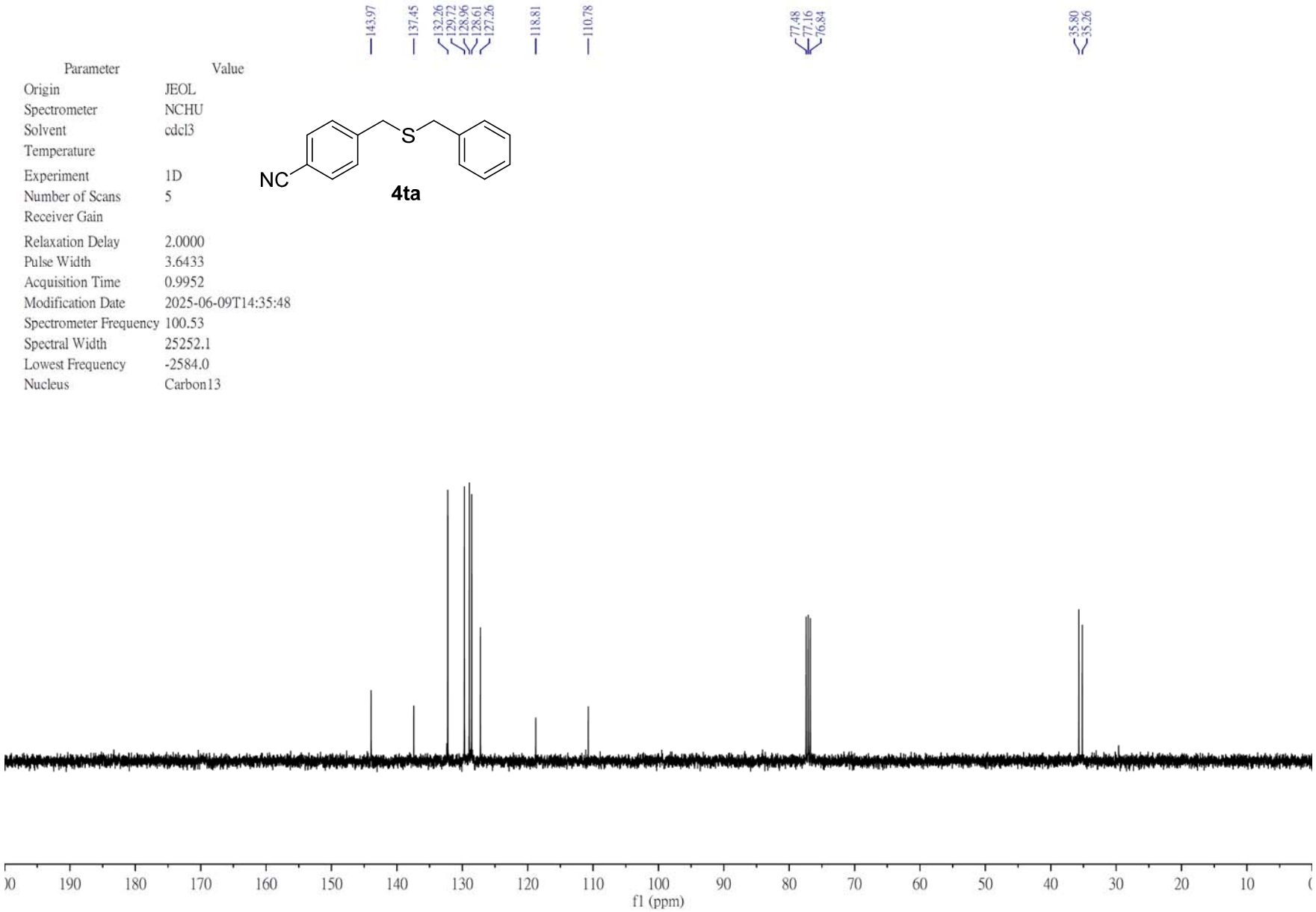
4xa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	30.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	8
Receiver Gain	46
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Modification Date	2025-01-08T16:23:00
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-800.0
Nucleus	1H

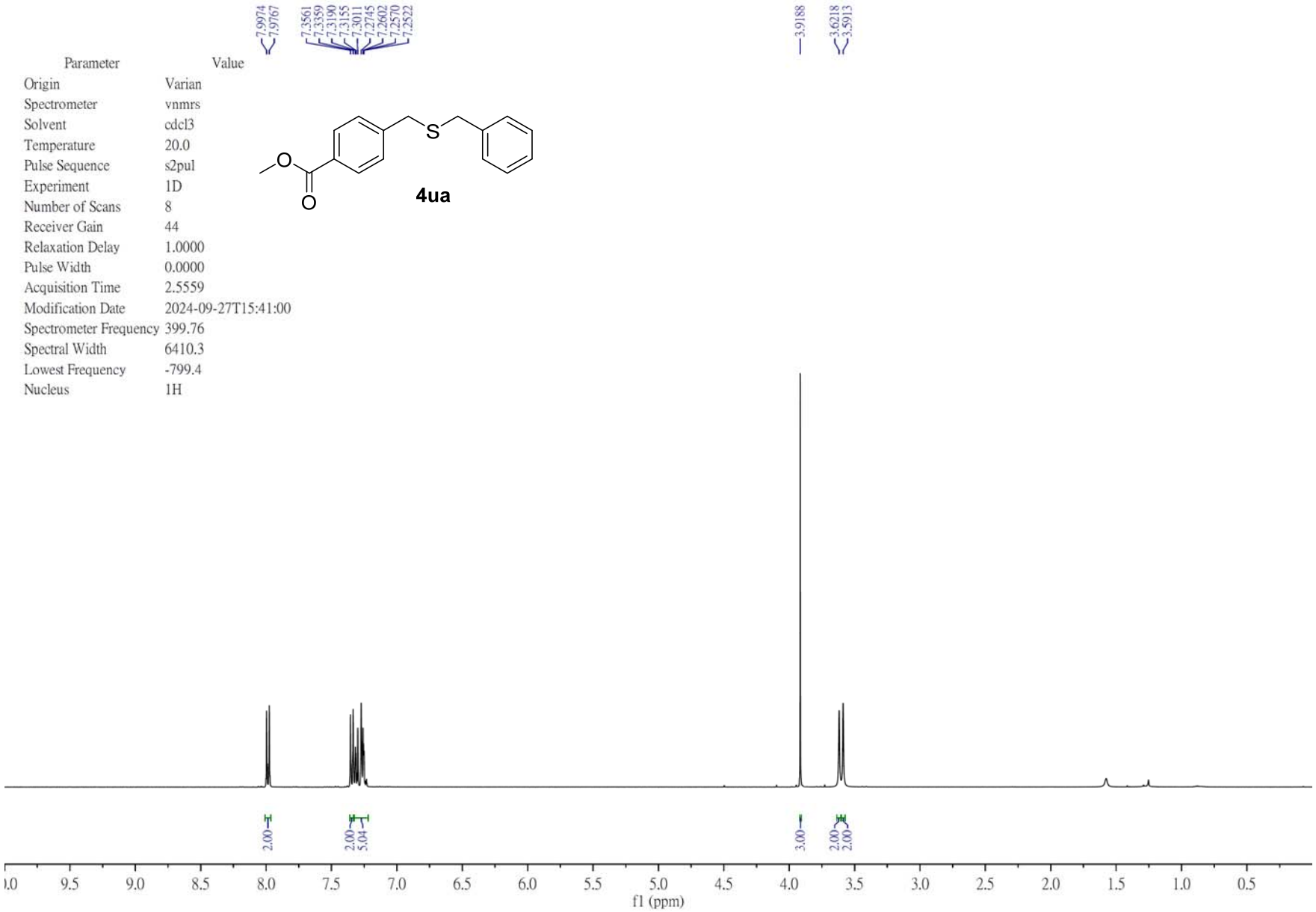
4ta



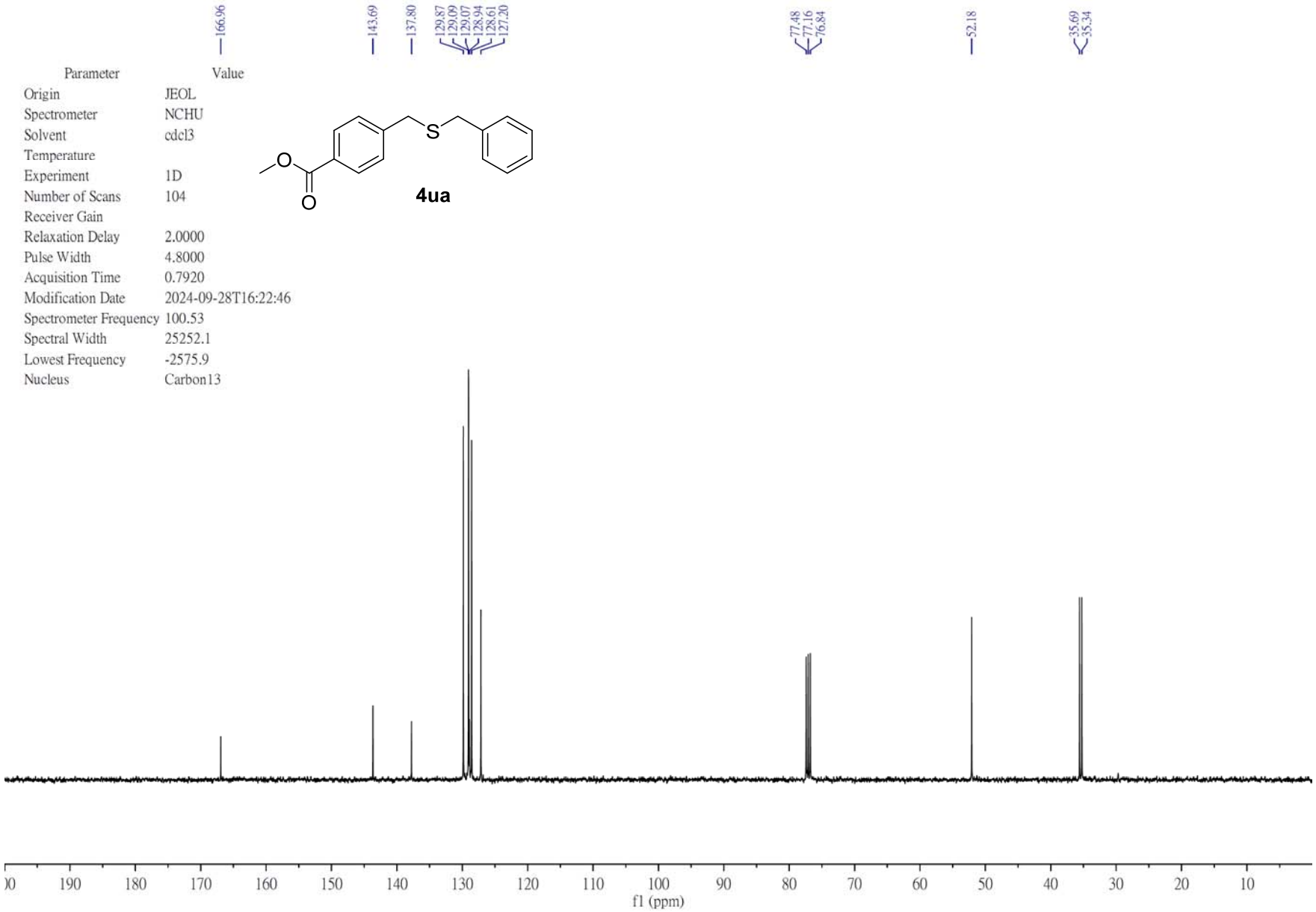
4ta ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



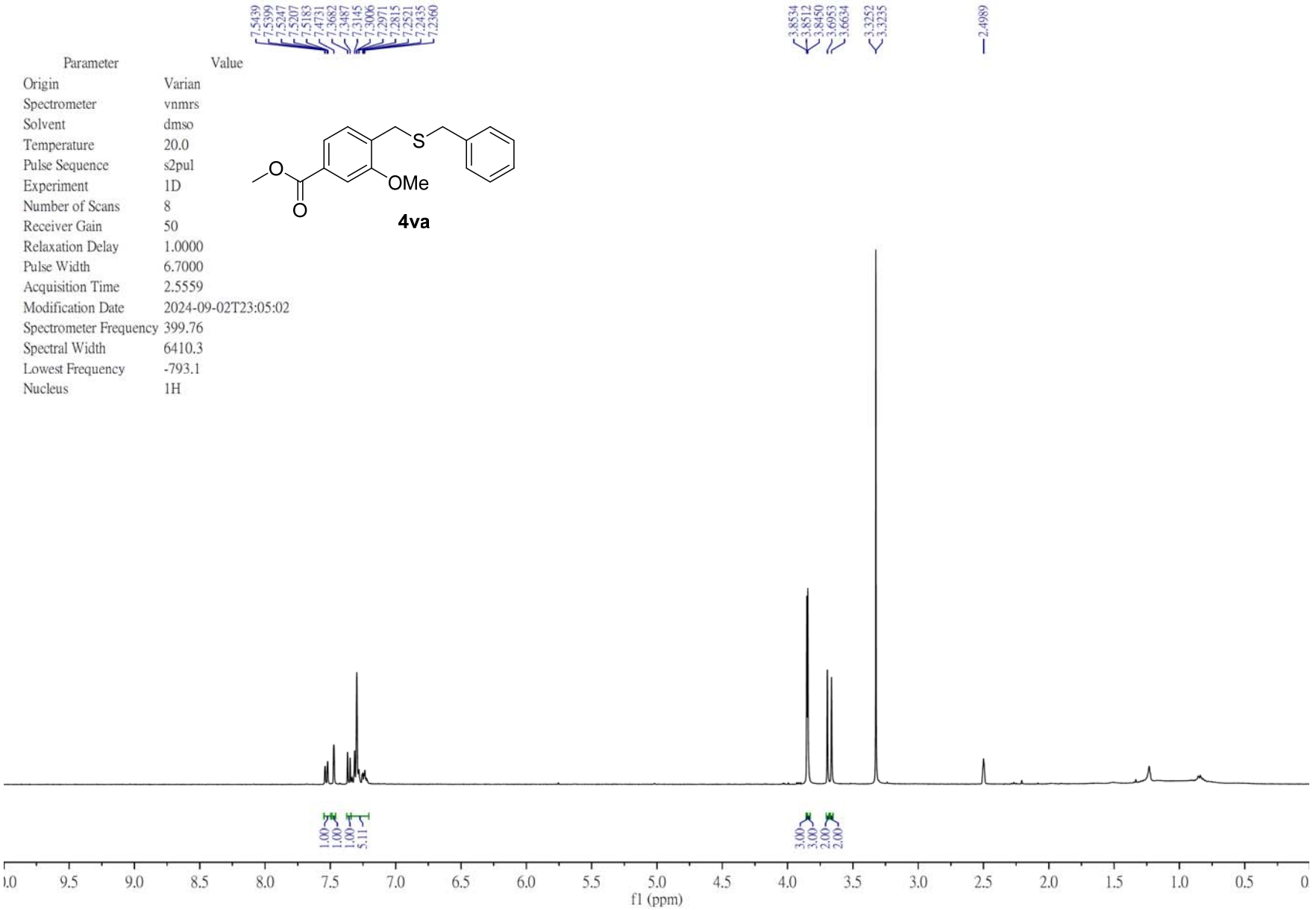
4ta $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



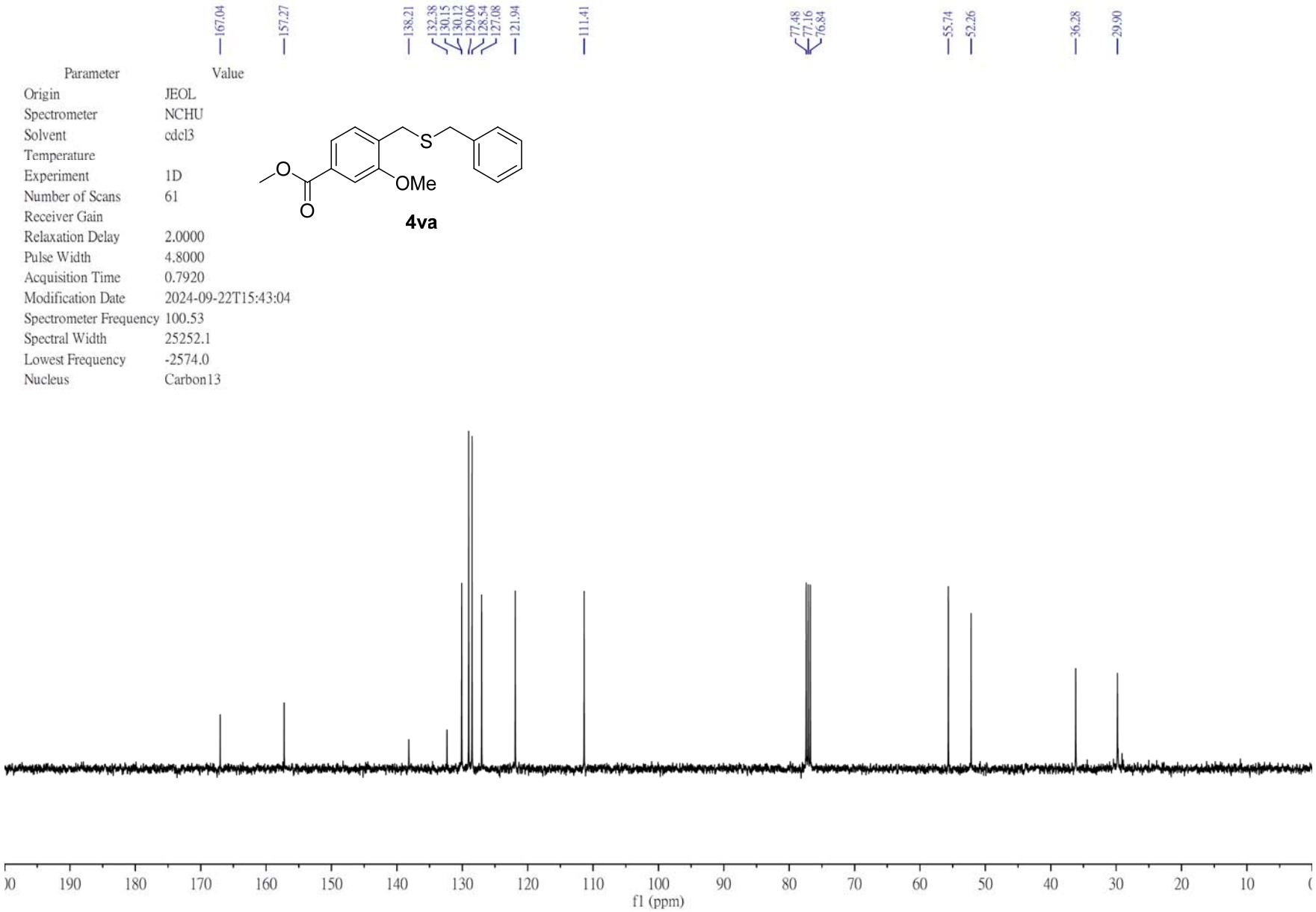
4ua ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



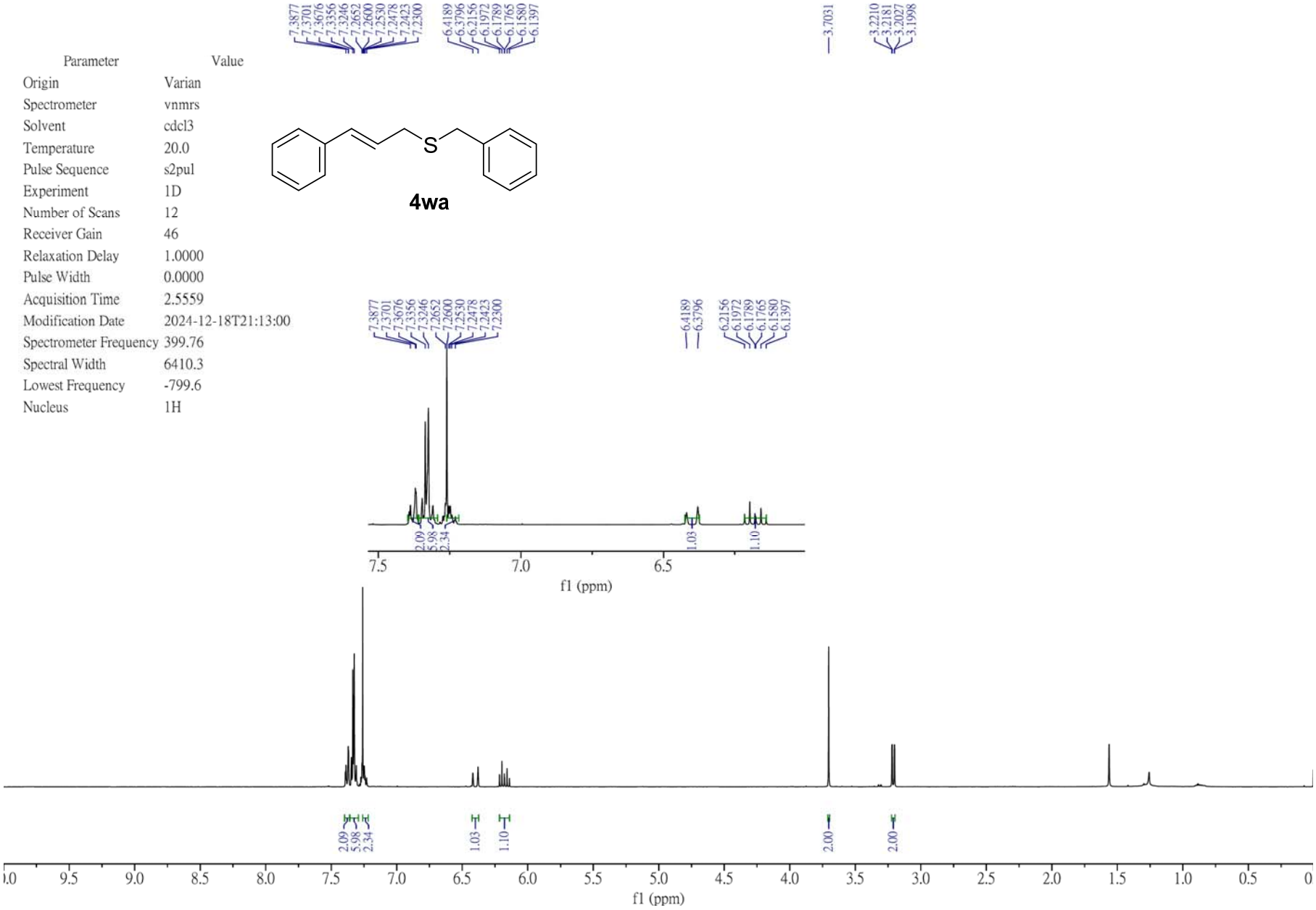
4ua $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



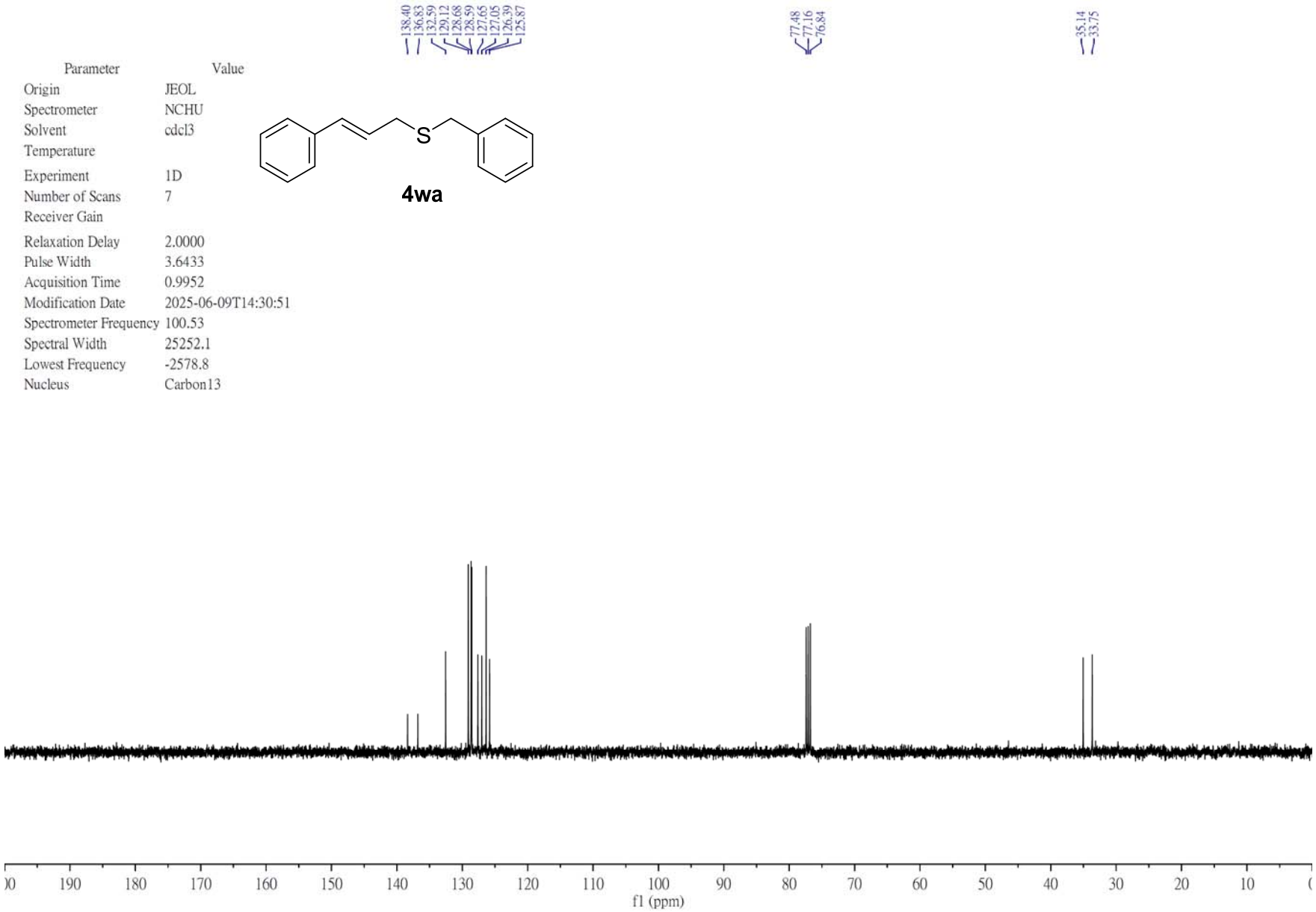
4va ^{1}H NMR spectrum（400 MHz in $(CD_3)_2SO$）



4va $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



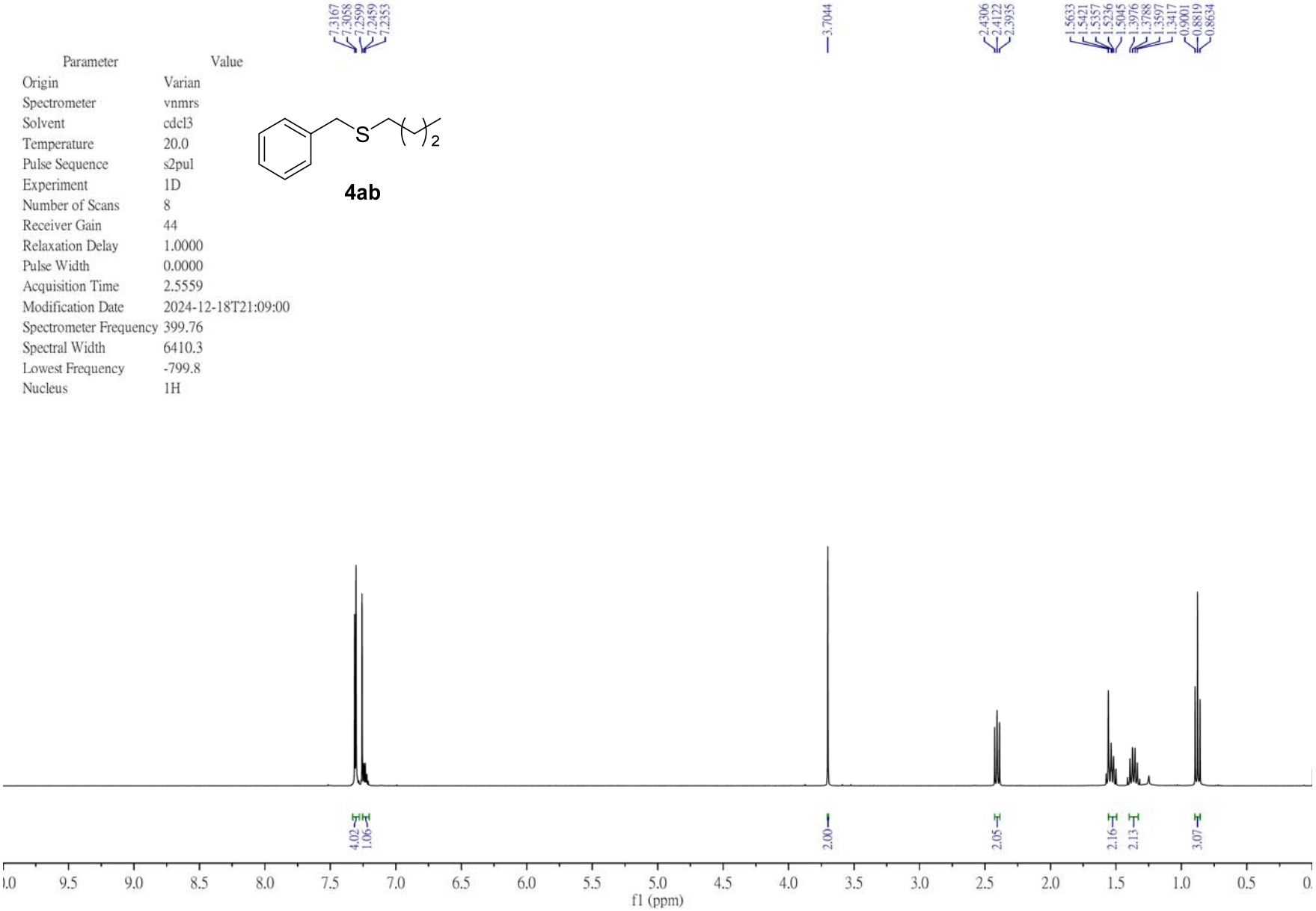
4wa ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



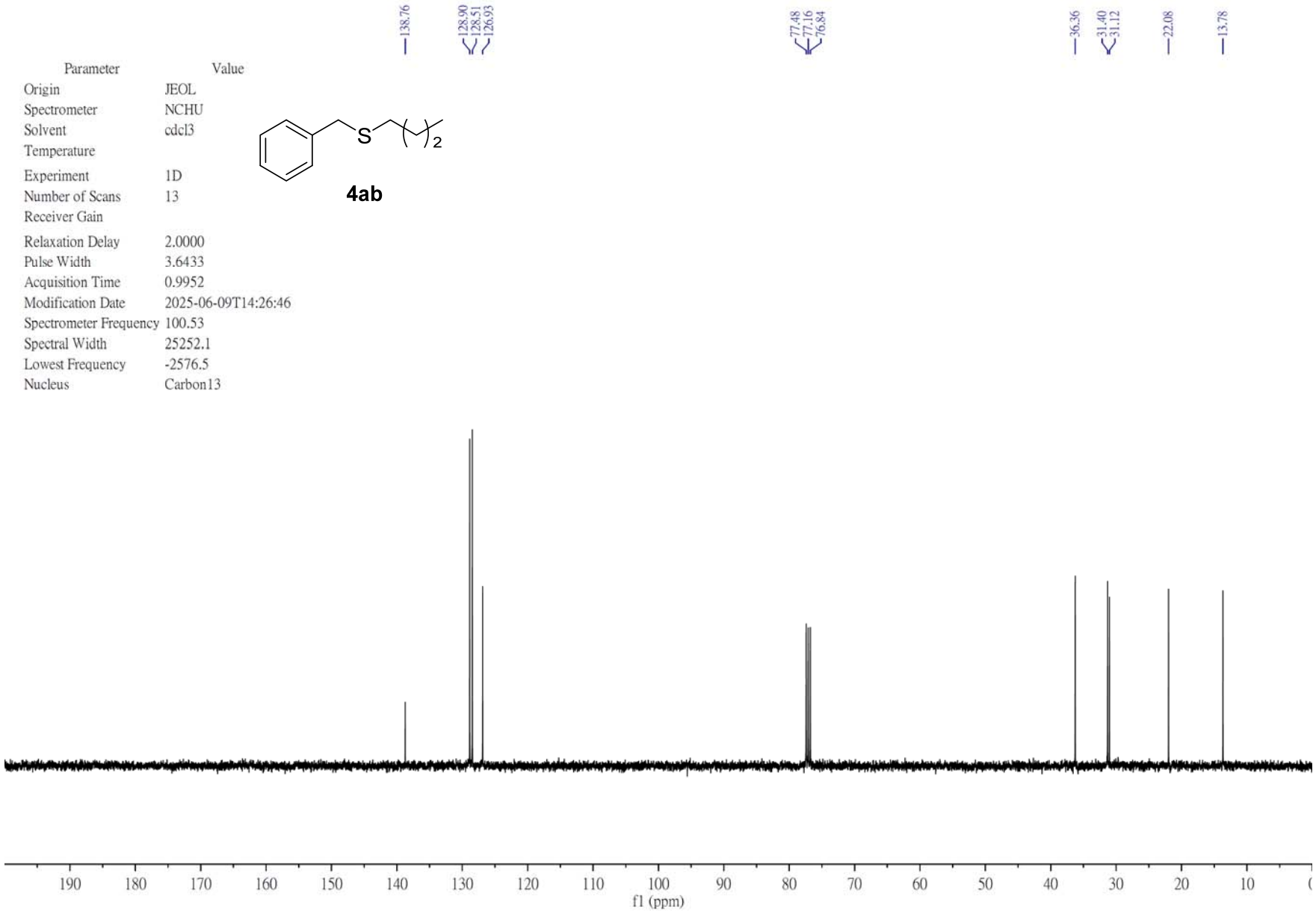
4wa $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

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
Modification Date	2024-12-18T21:09:00
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.8
Nucleus	1H

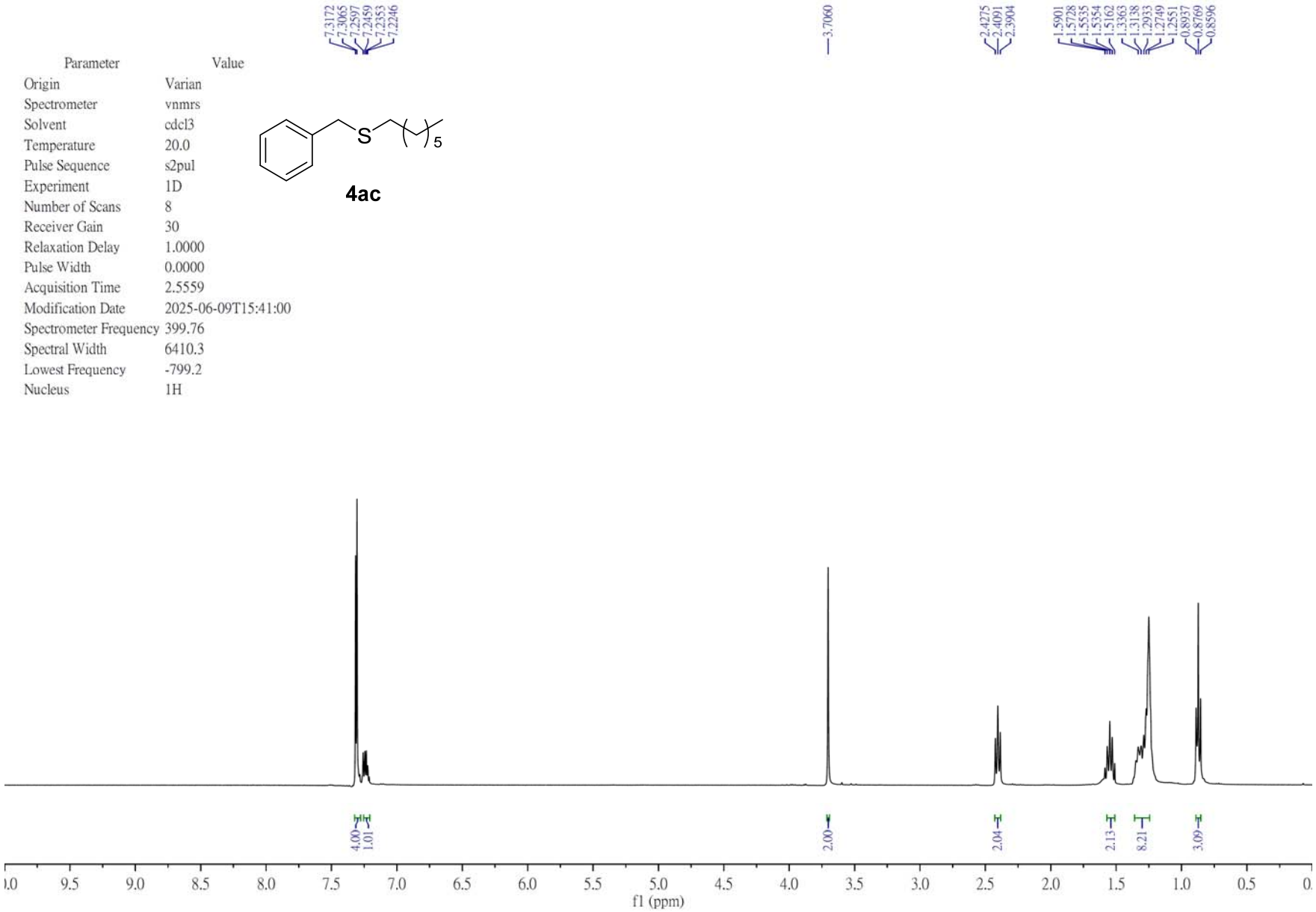
4ab



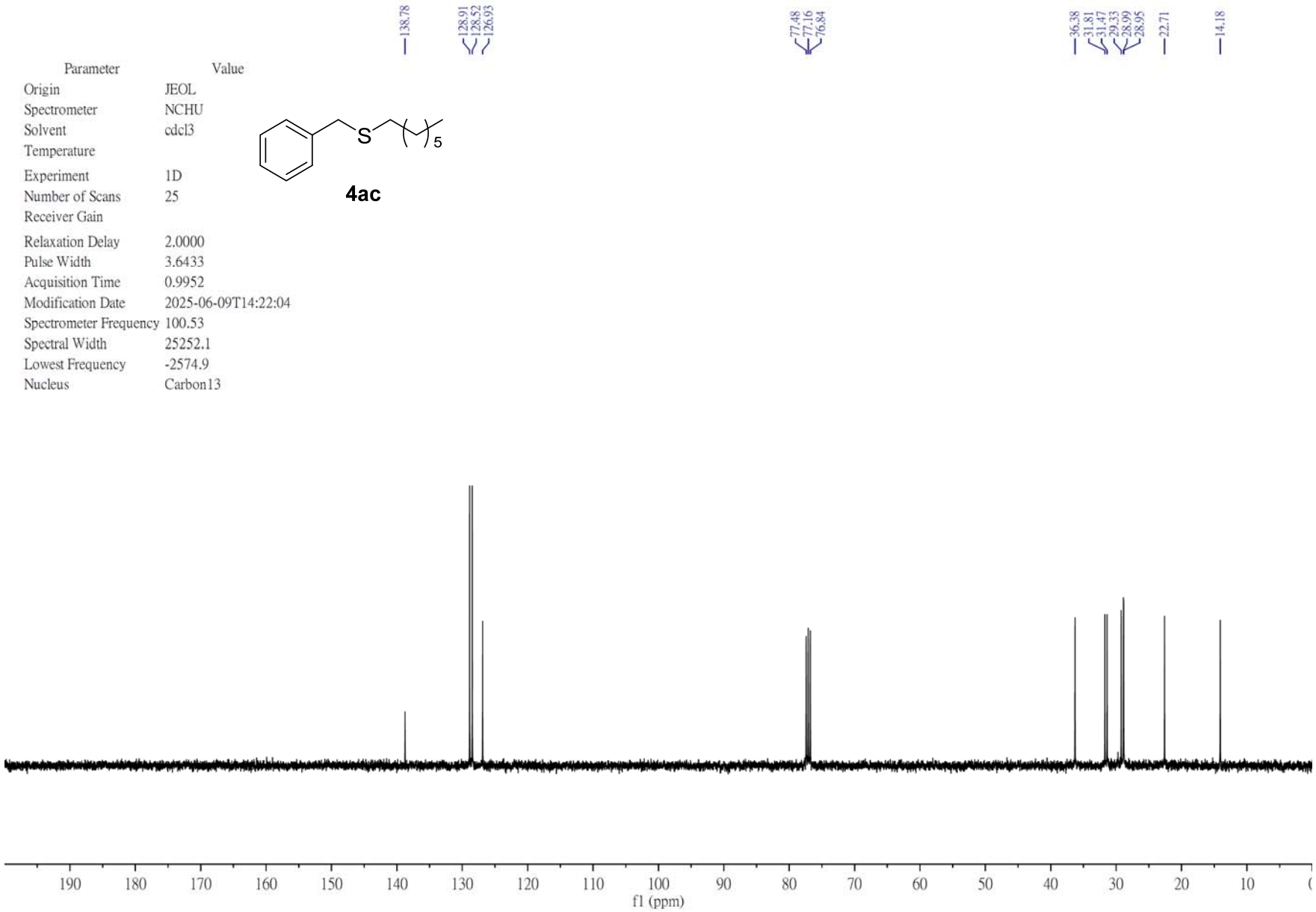
4ab ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



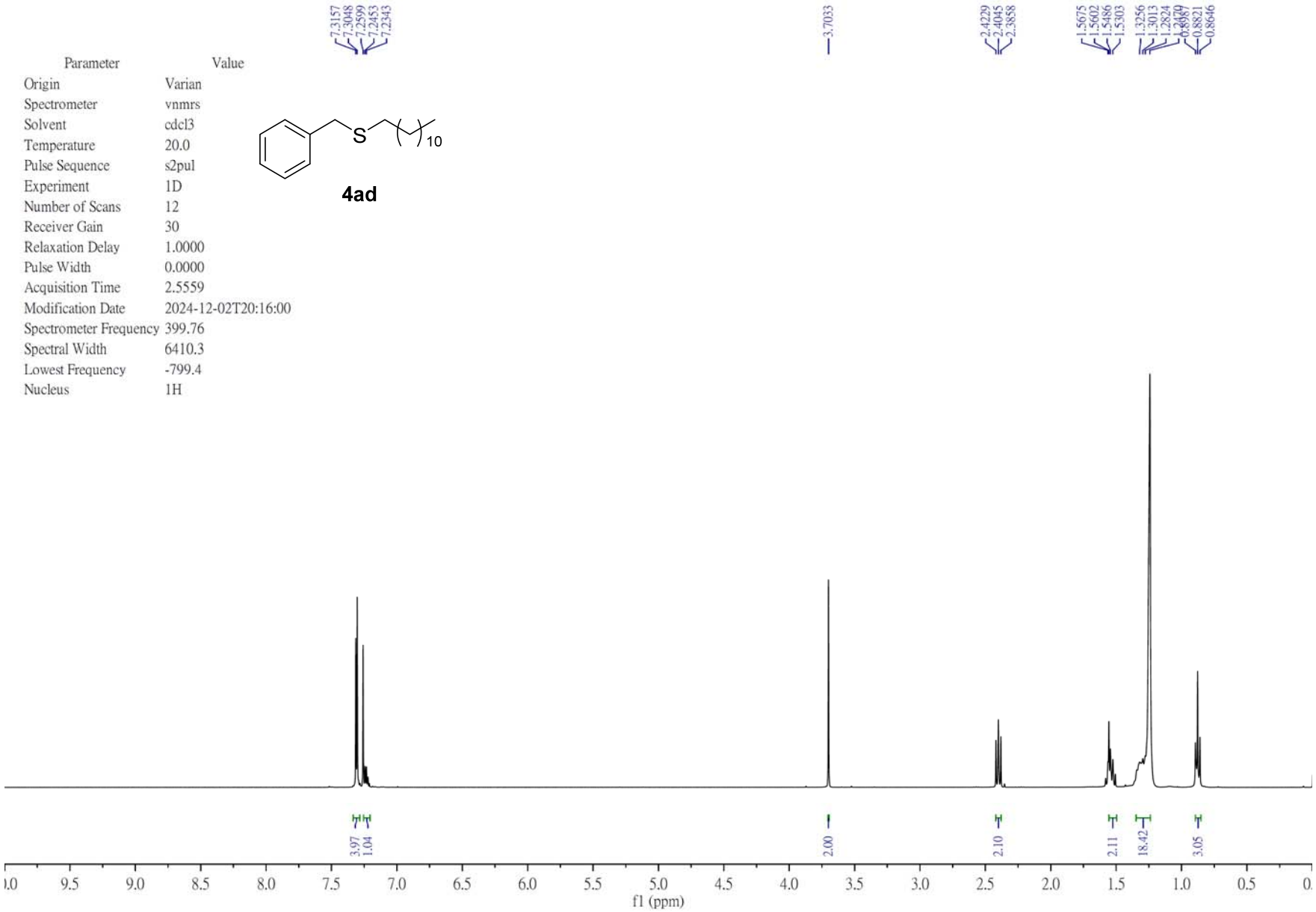
4ab $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



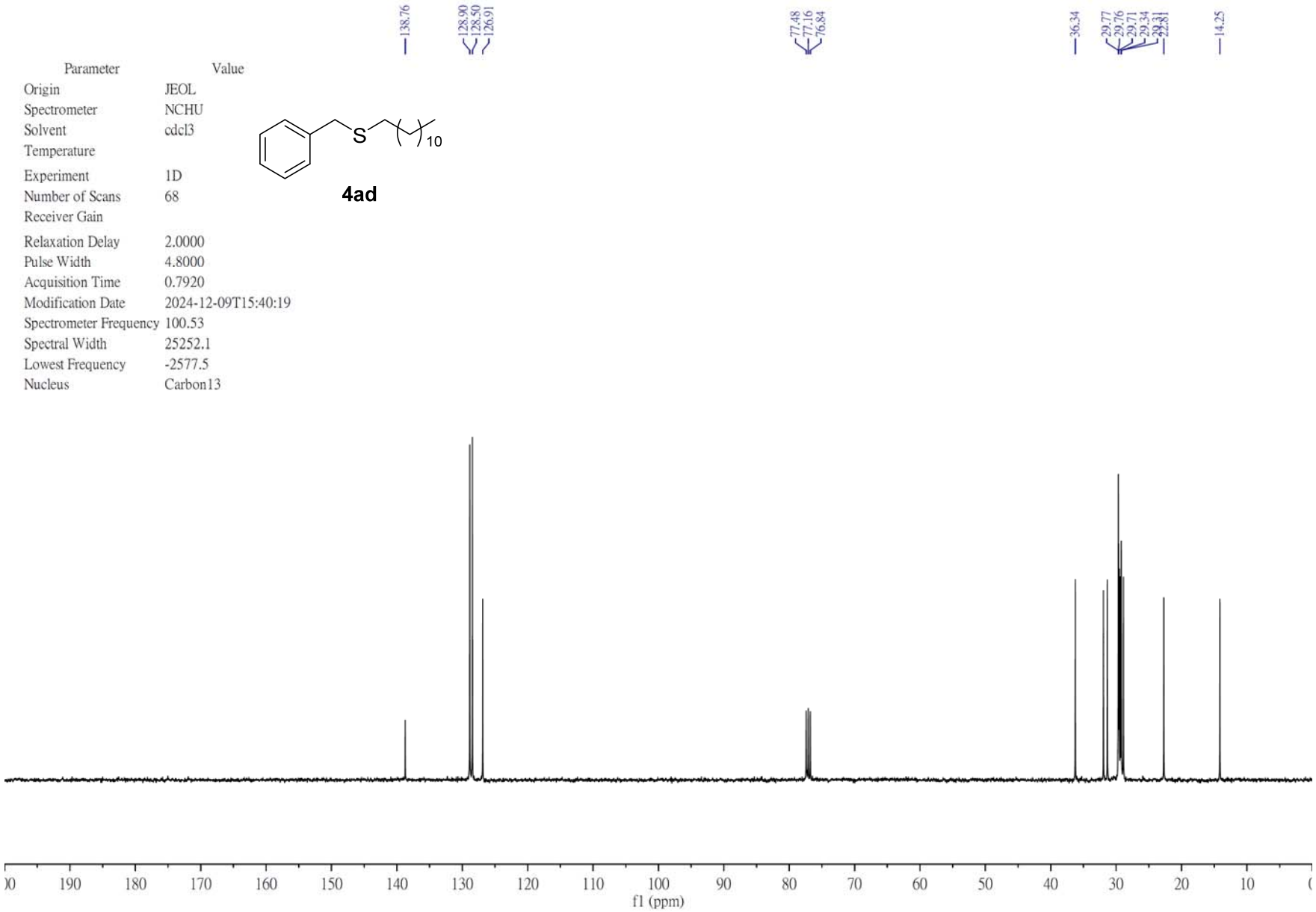
4ac ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



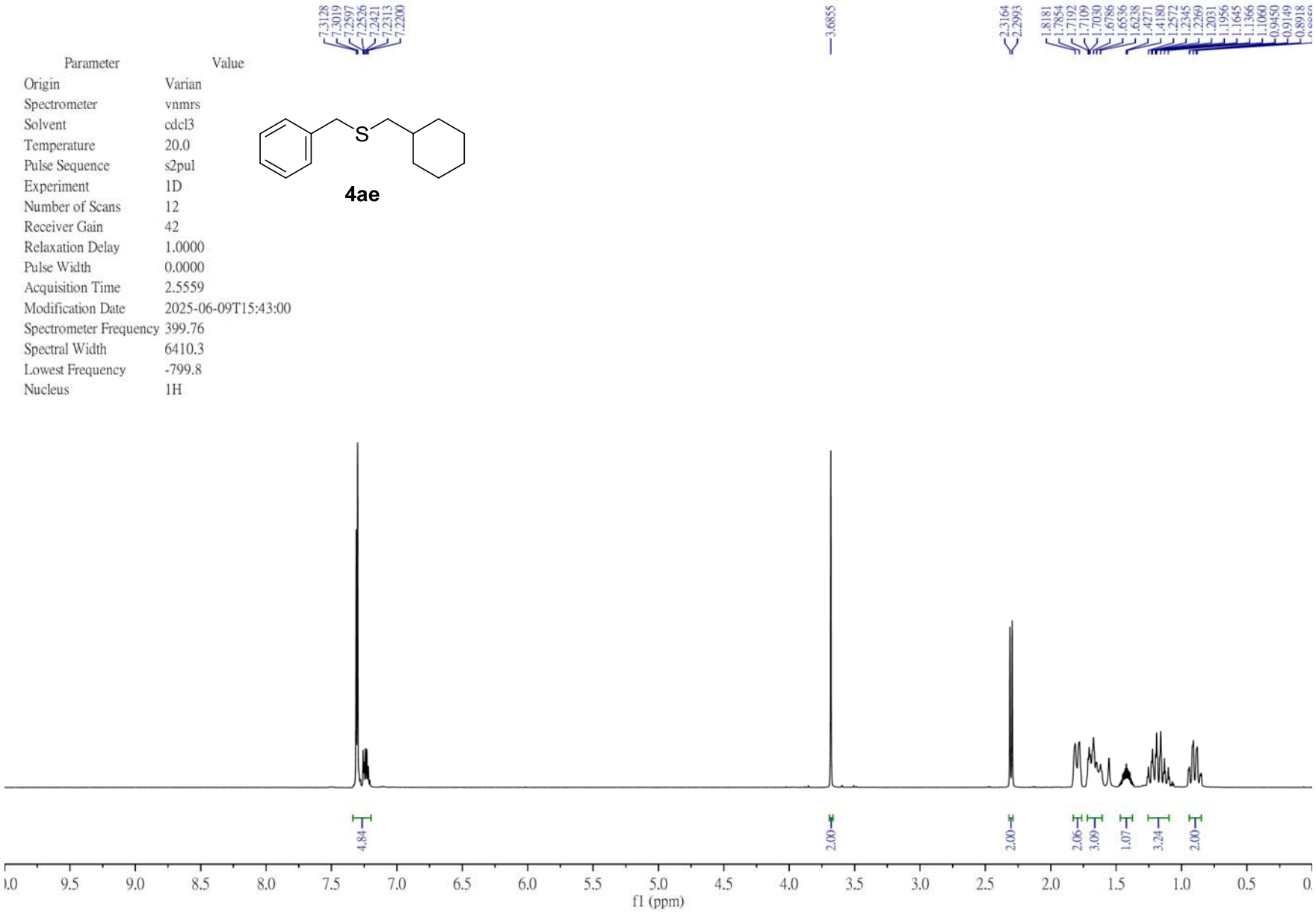
4ac $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



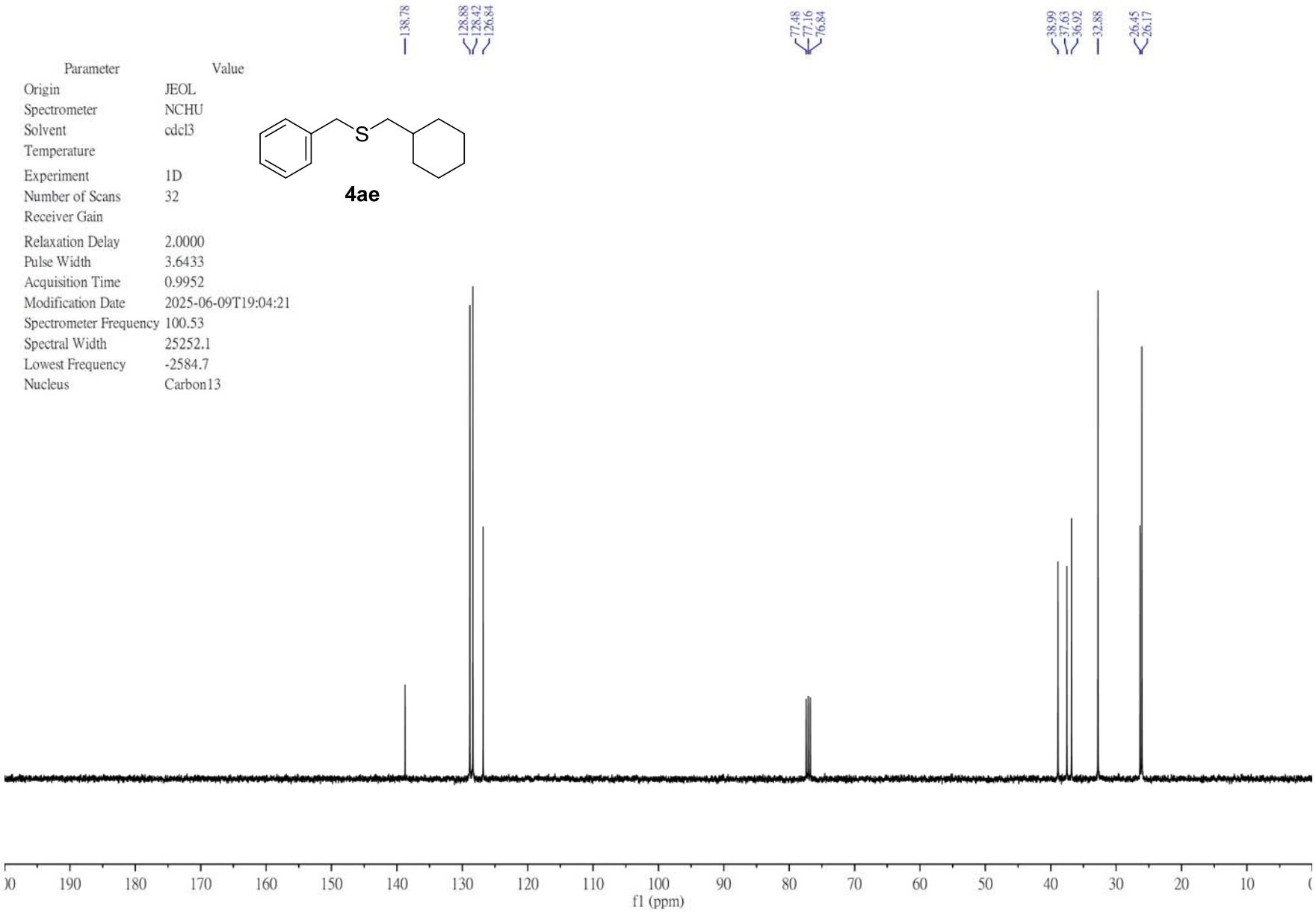
4ad ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



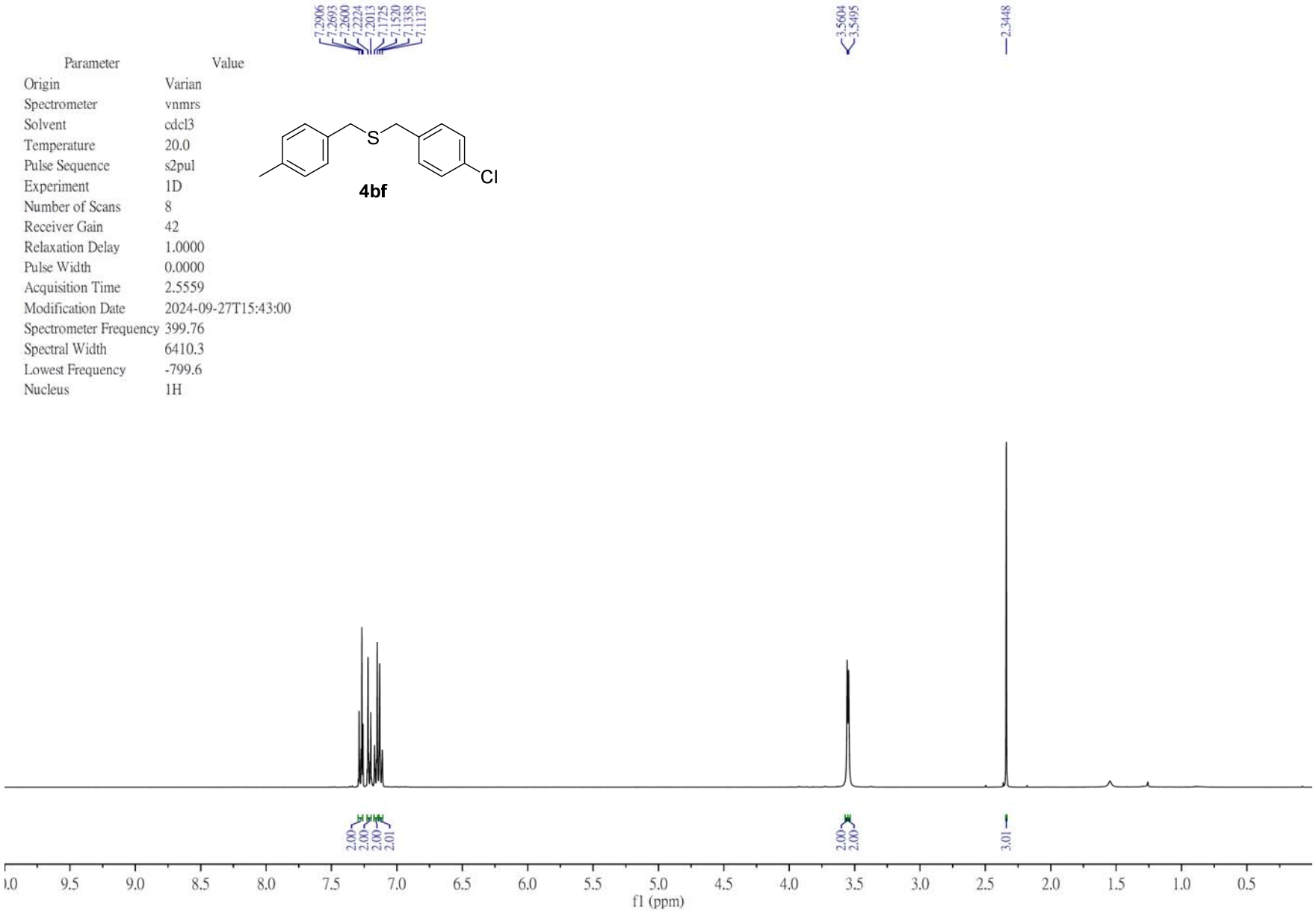
4ad $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



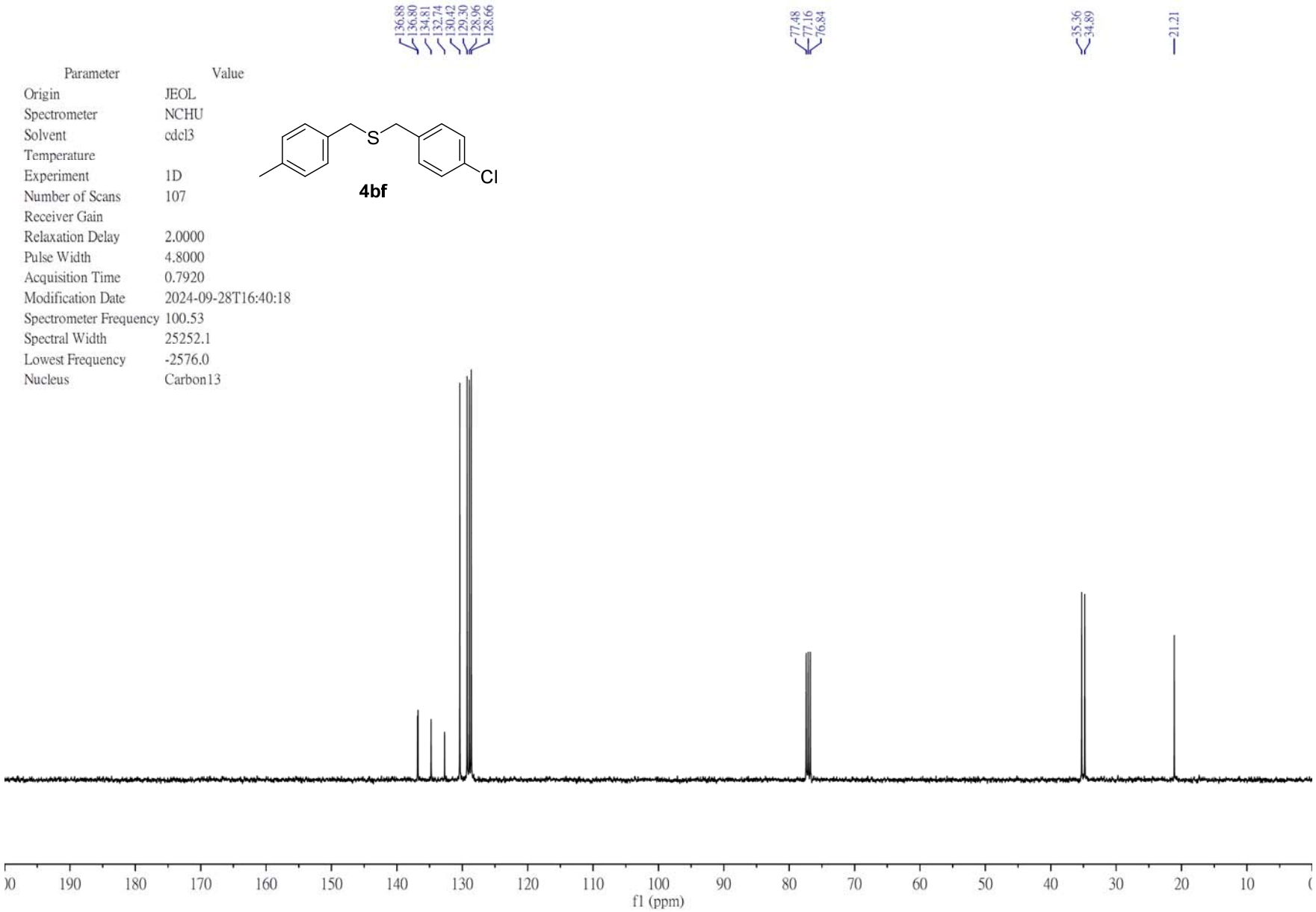
4ae ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4ae $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



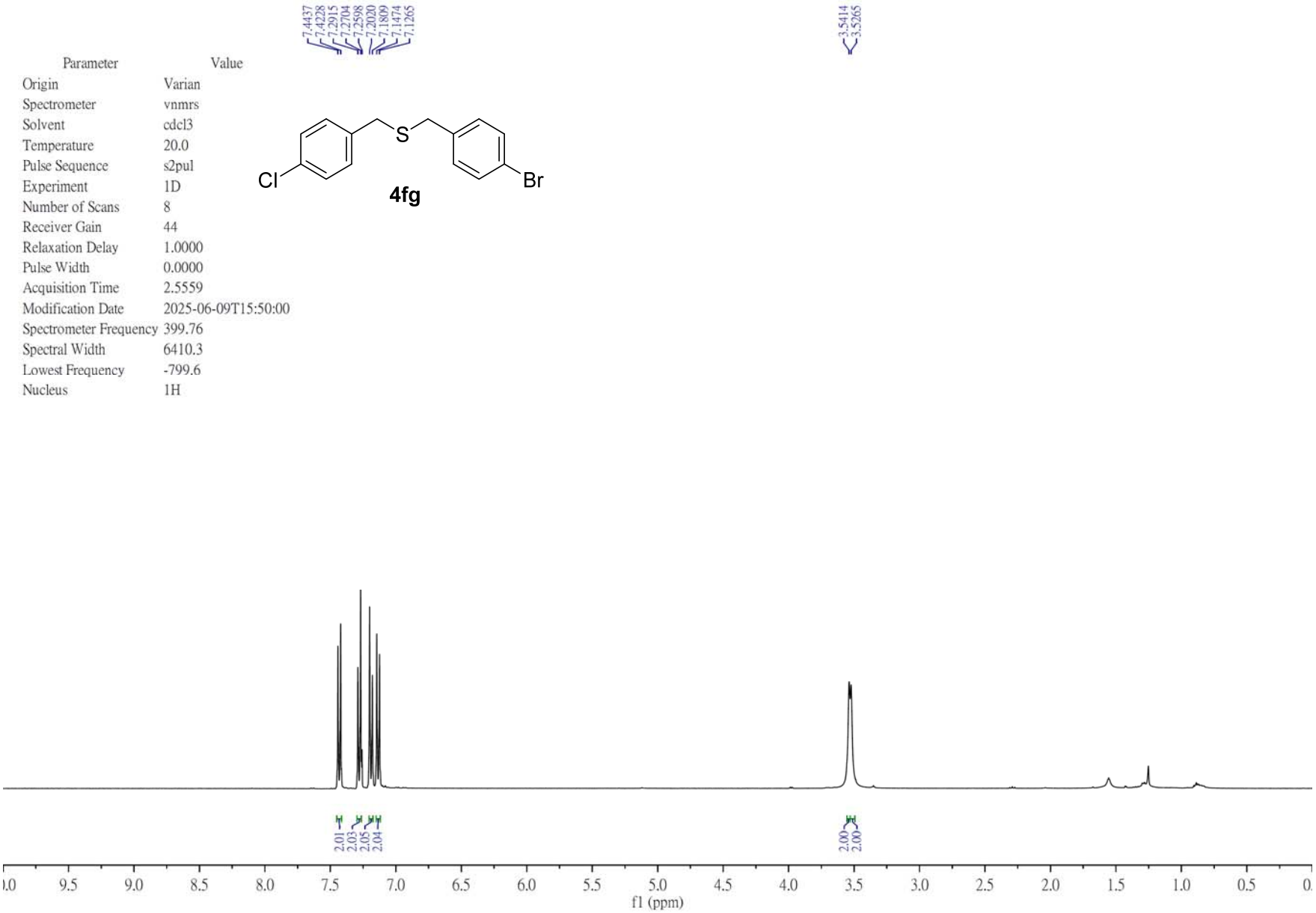
4bf ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



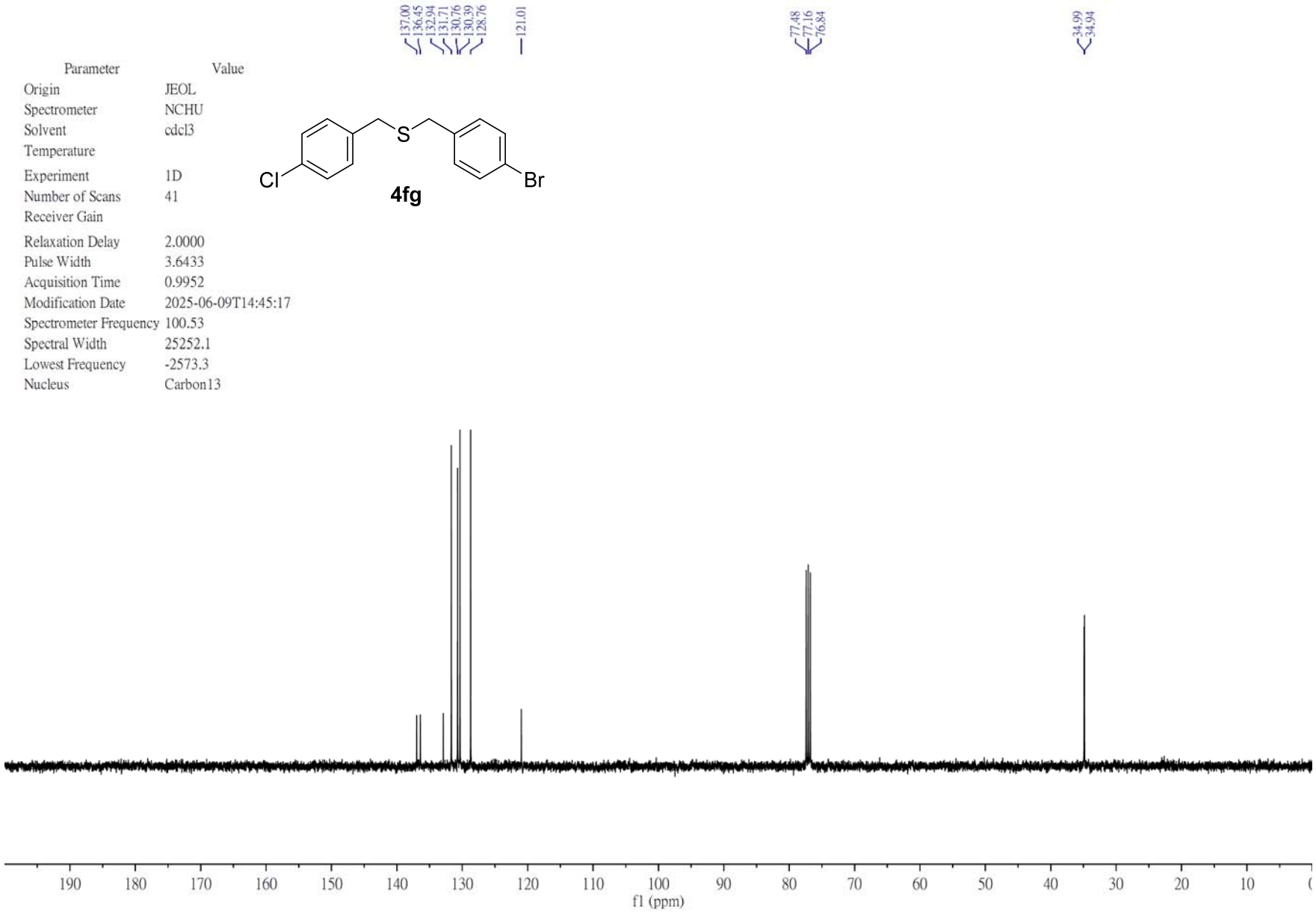
4bf $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

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
Modification Date	2025-06-09T15:50:00
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.6
Nucleus	1H

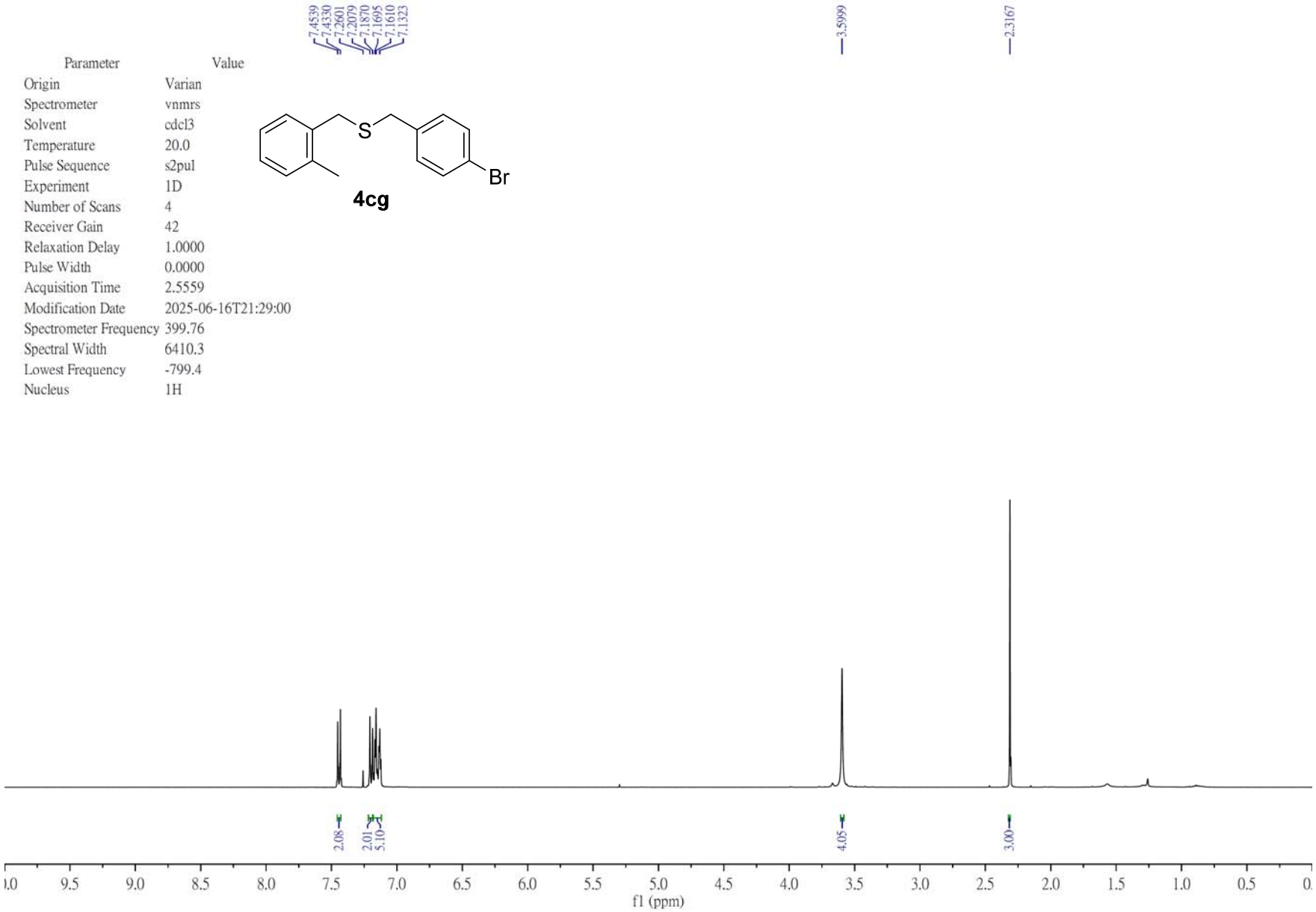
4fg



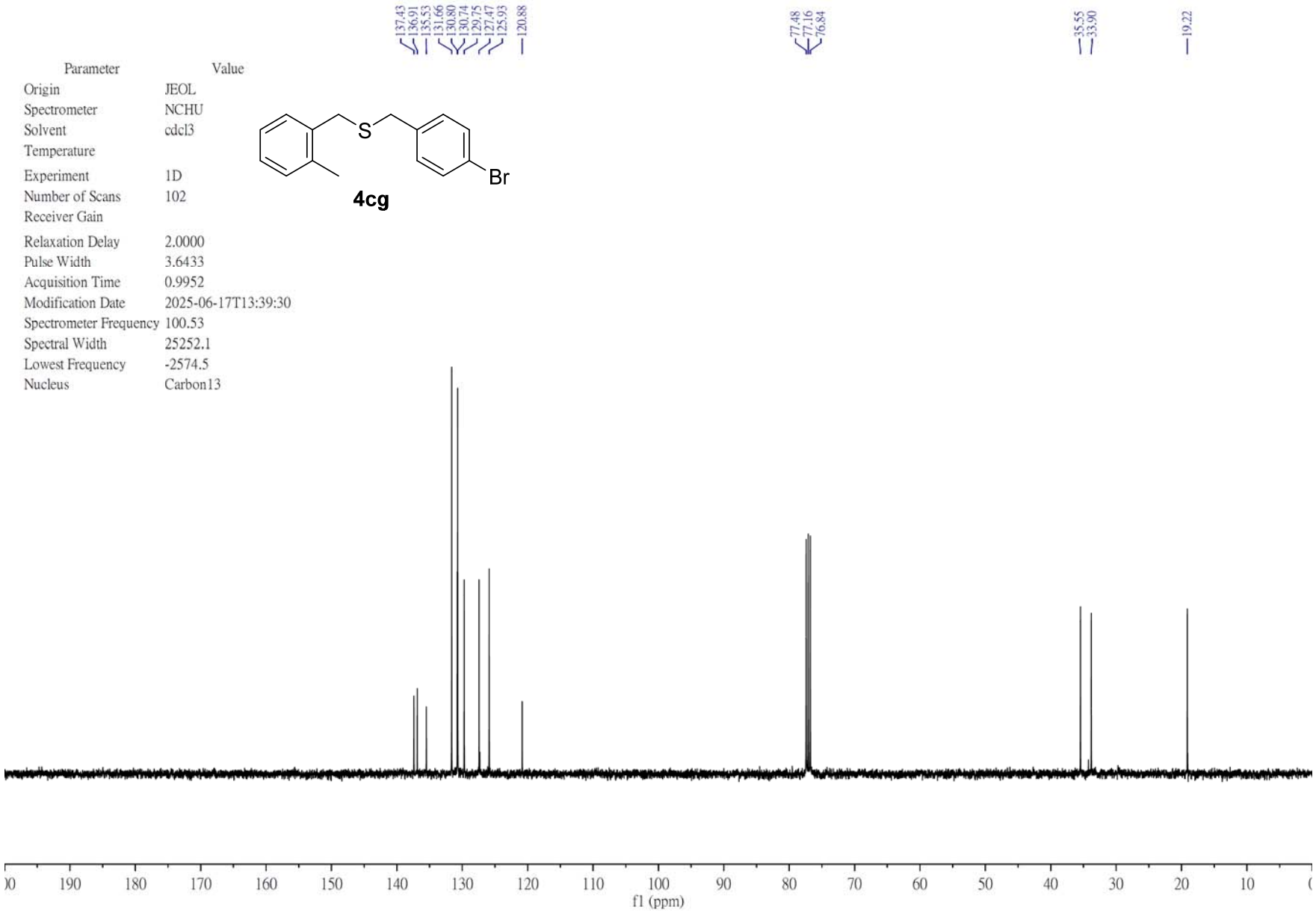
4fg ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



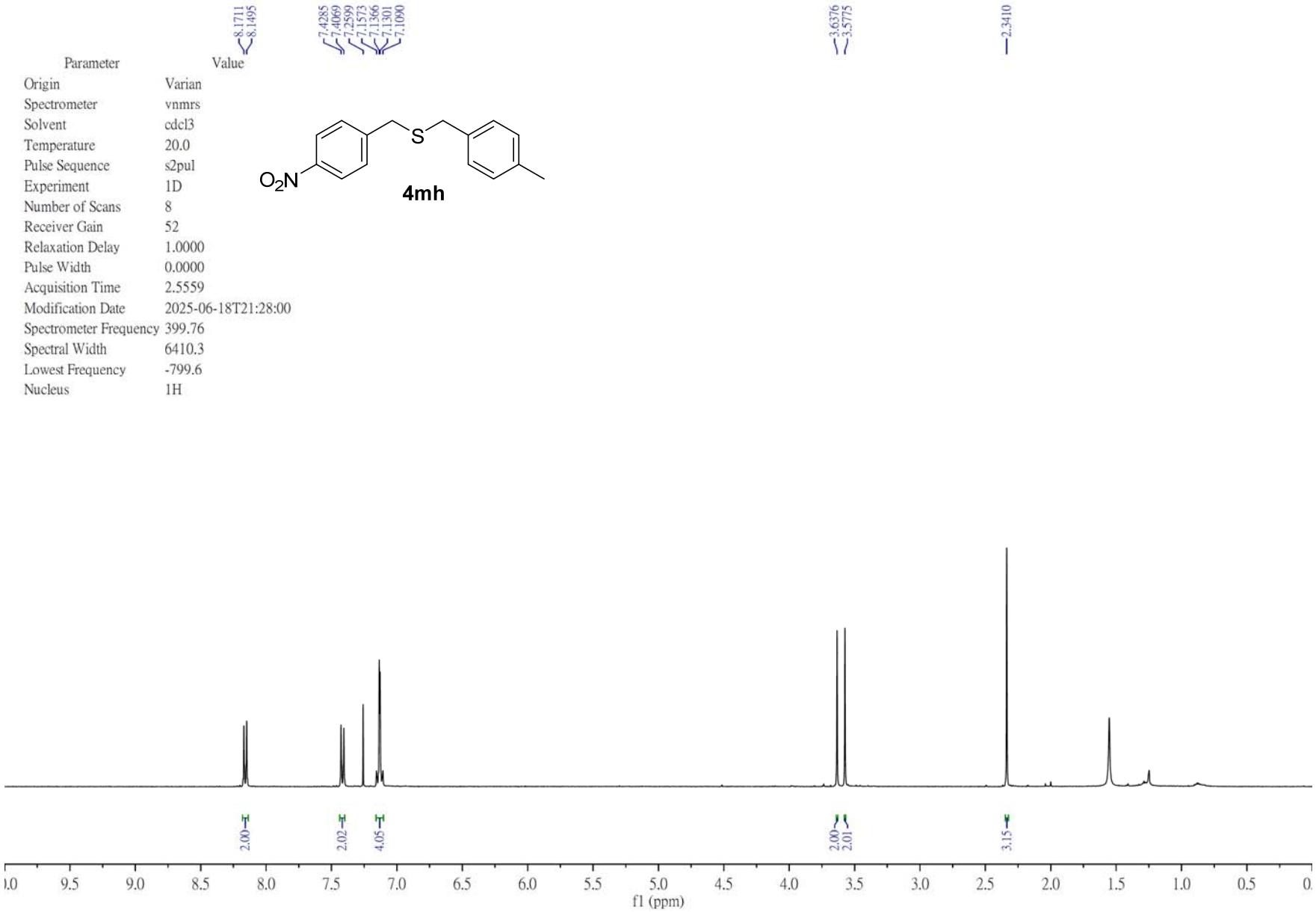
4fg $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



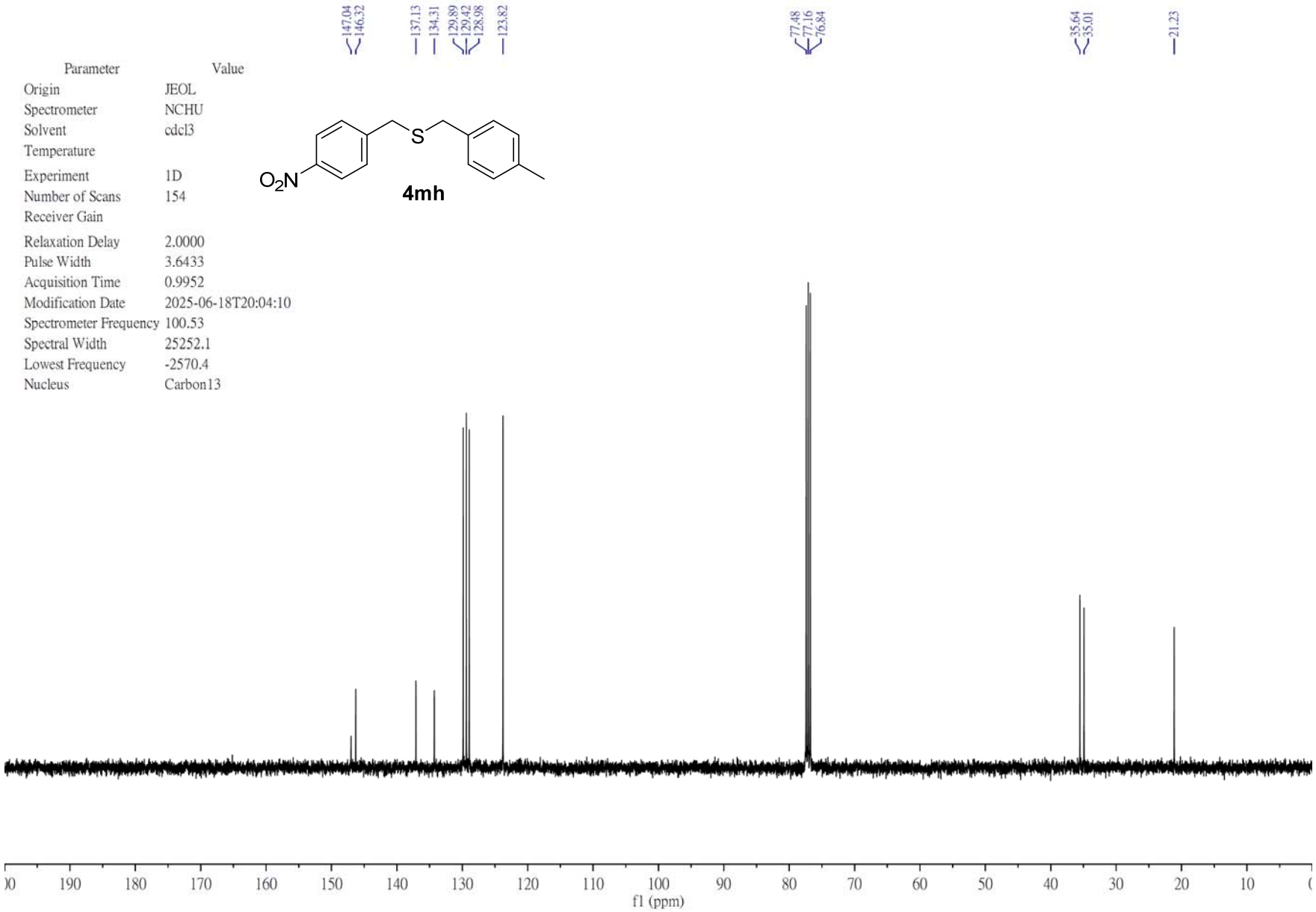
4cg ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4cg $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



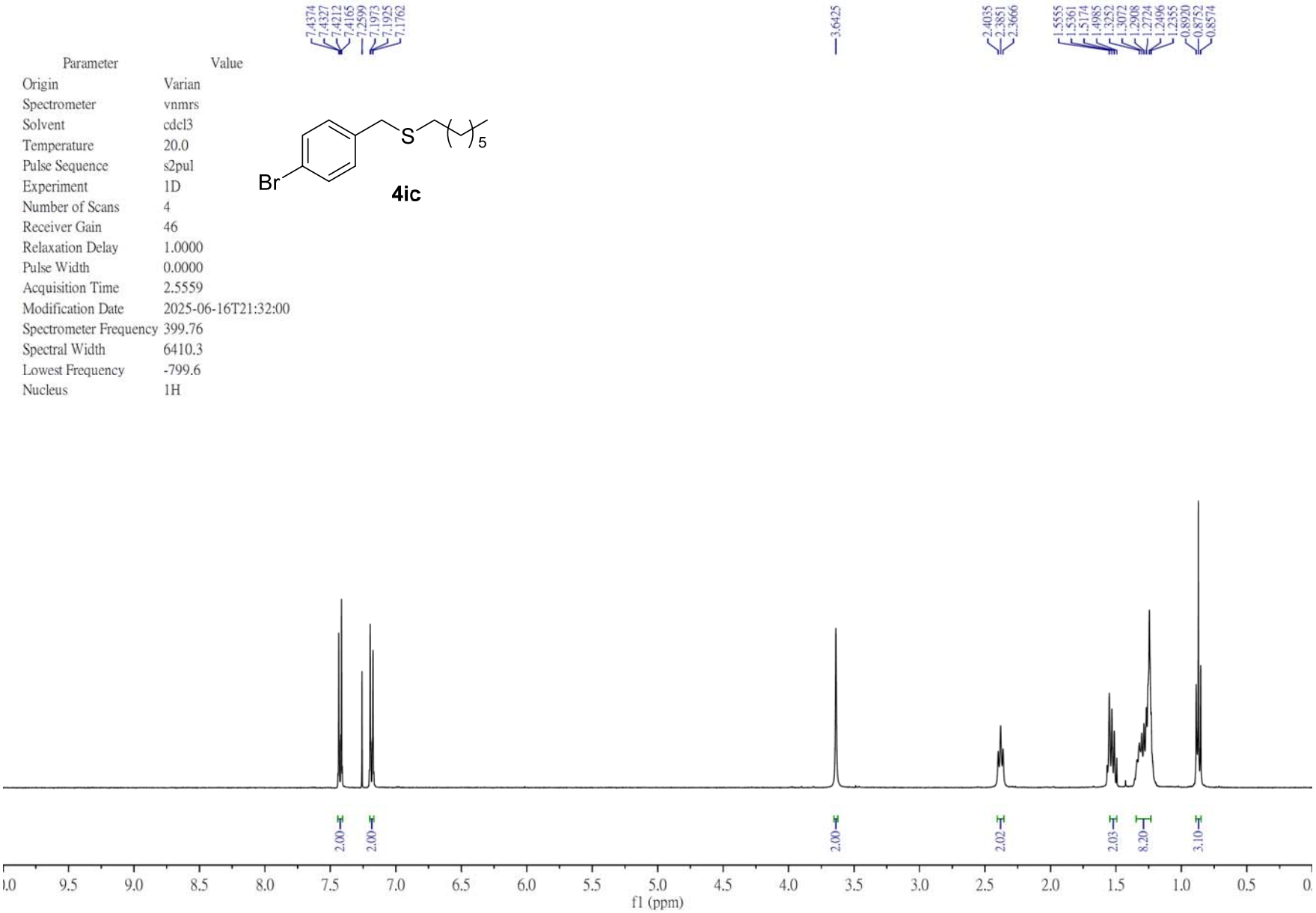
4mh ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



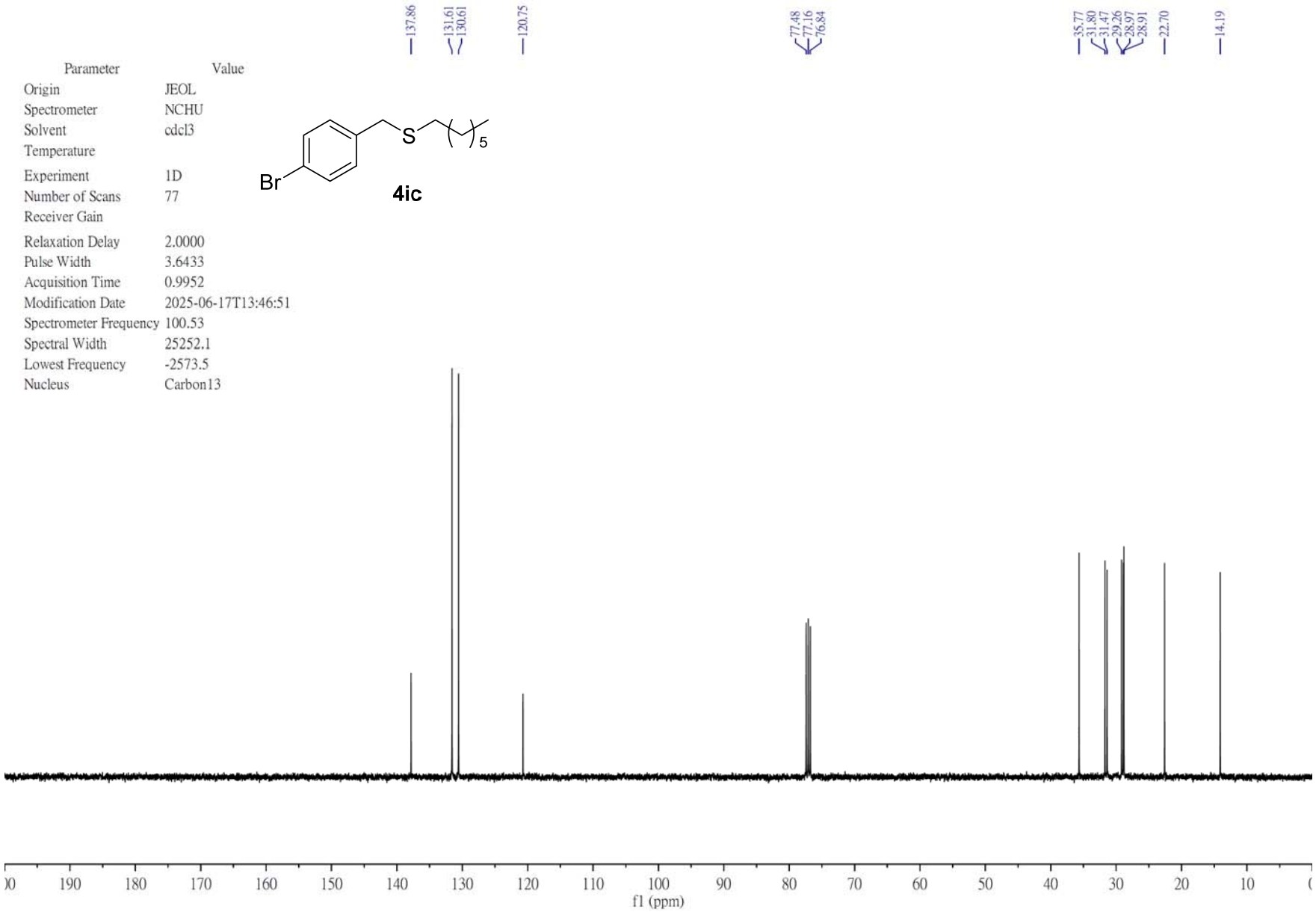
4mh $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

Parameter	Value
Origin	Varian
Spectrometer	vnmrs
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Experiment	1D
Number of Scans	4
Receiver Gain	46
Relaxation Delay	1.0000
Pulse Width	0.0000
Acquisition Time	2.5559
Modification Date	2025-06-16T21:32:00
Spectrometer Frequency	399.76
Spectral Width	6410.3
Lowest Frequency	-799.6
Nucleus	1H

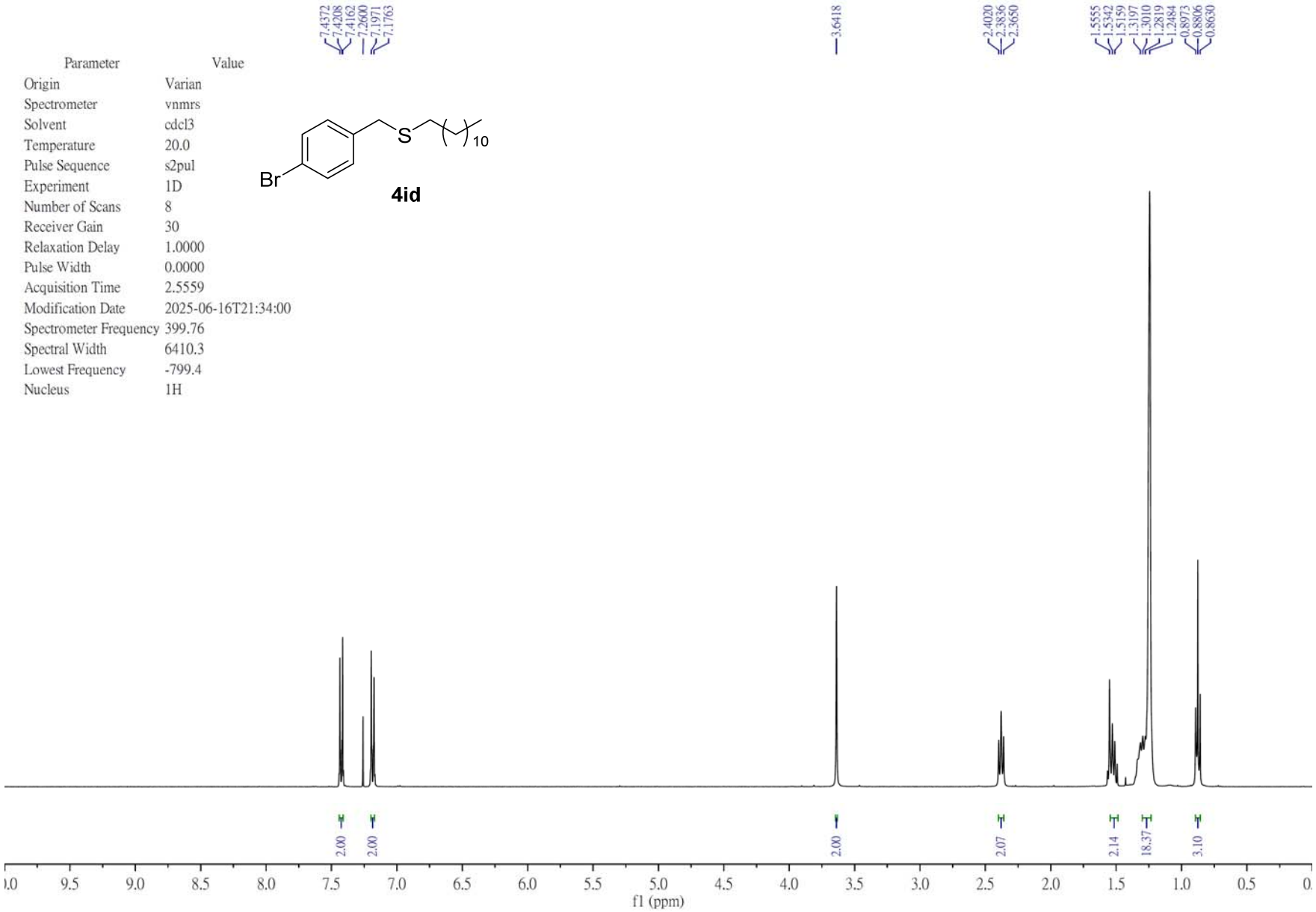
4ic



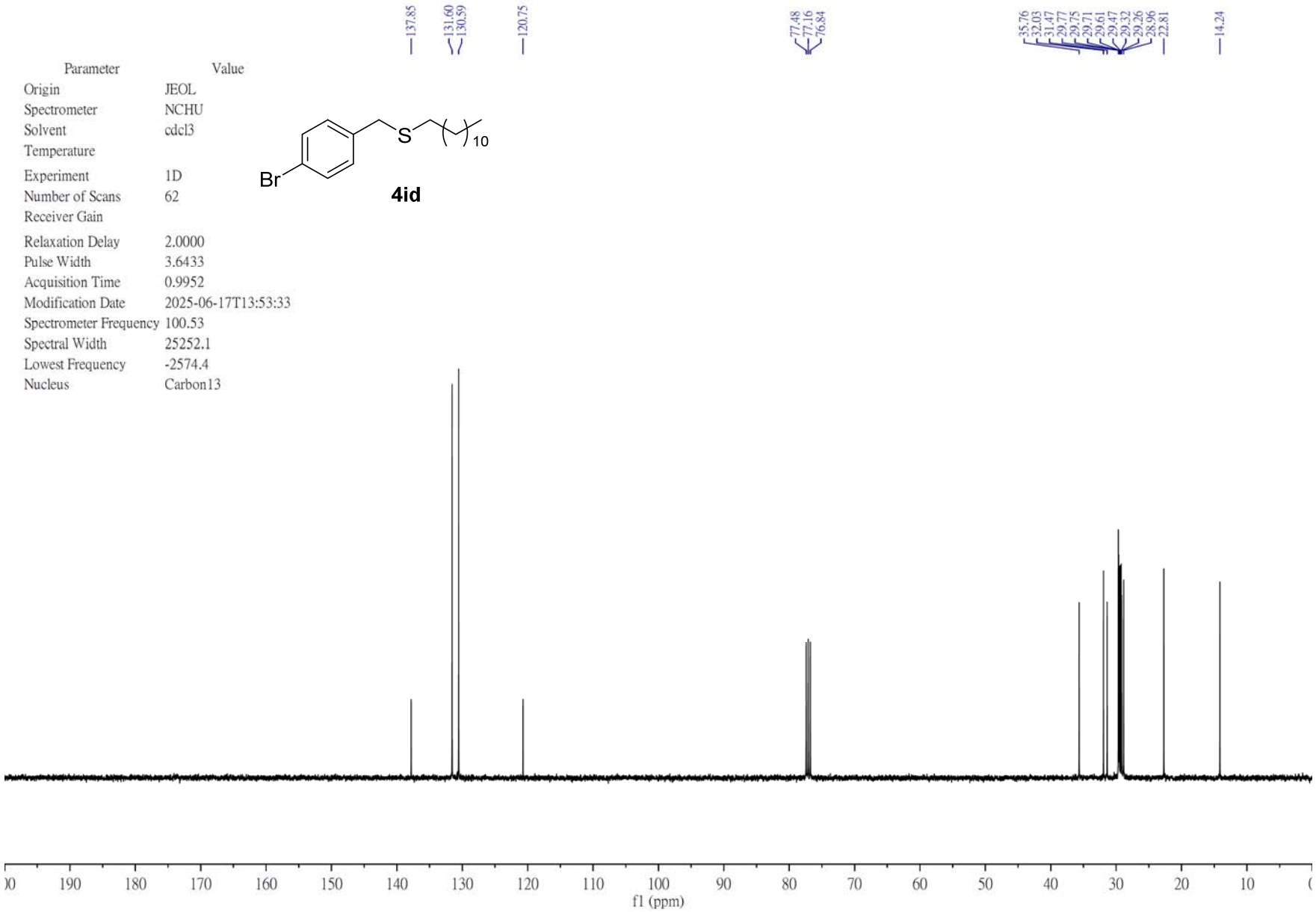
4ic ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



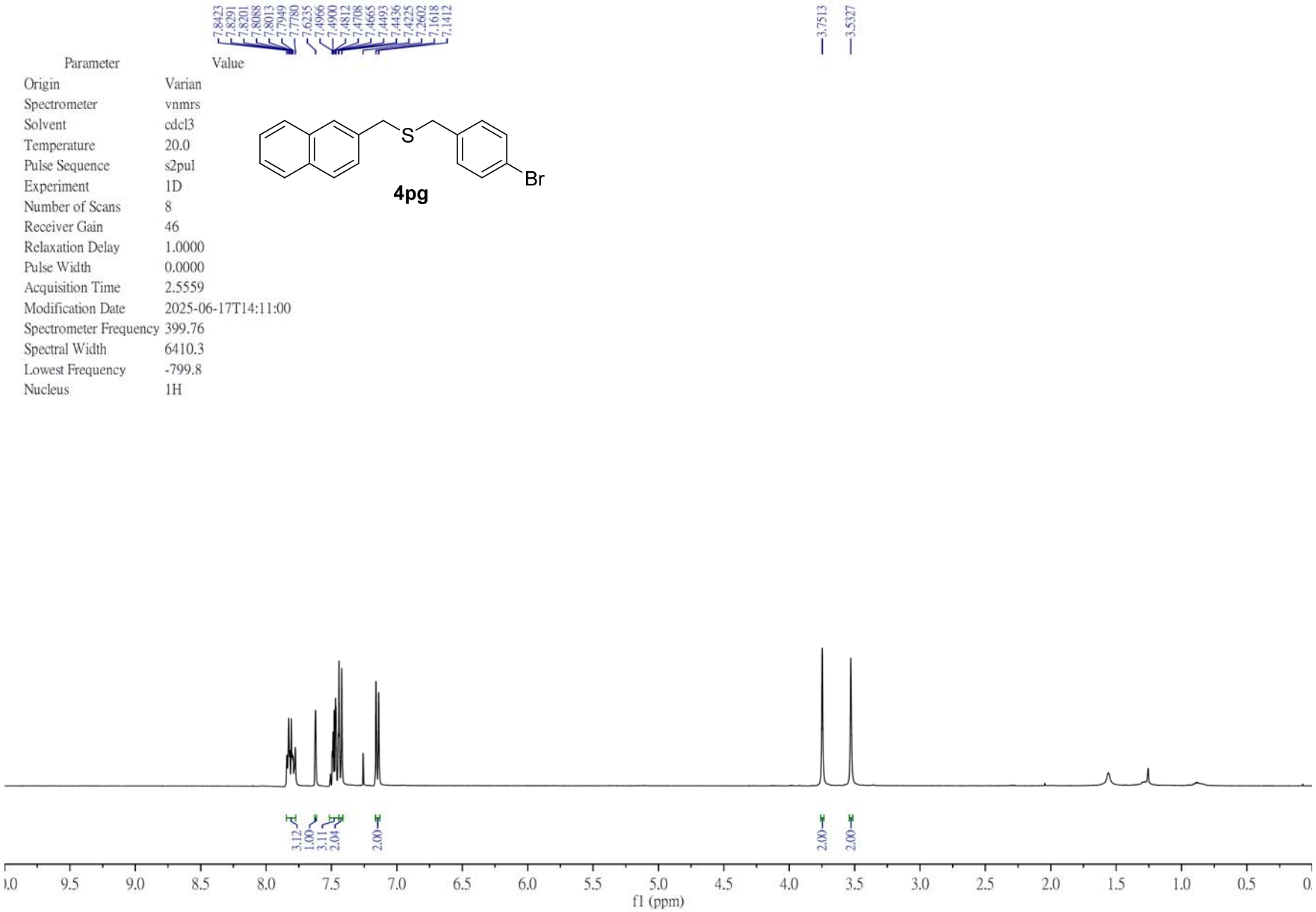
4ic $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



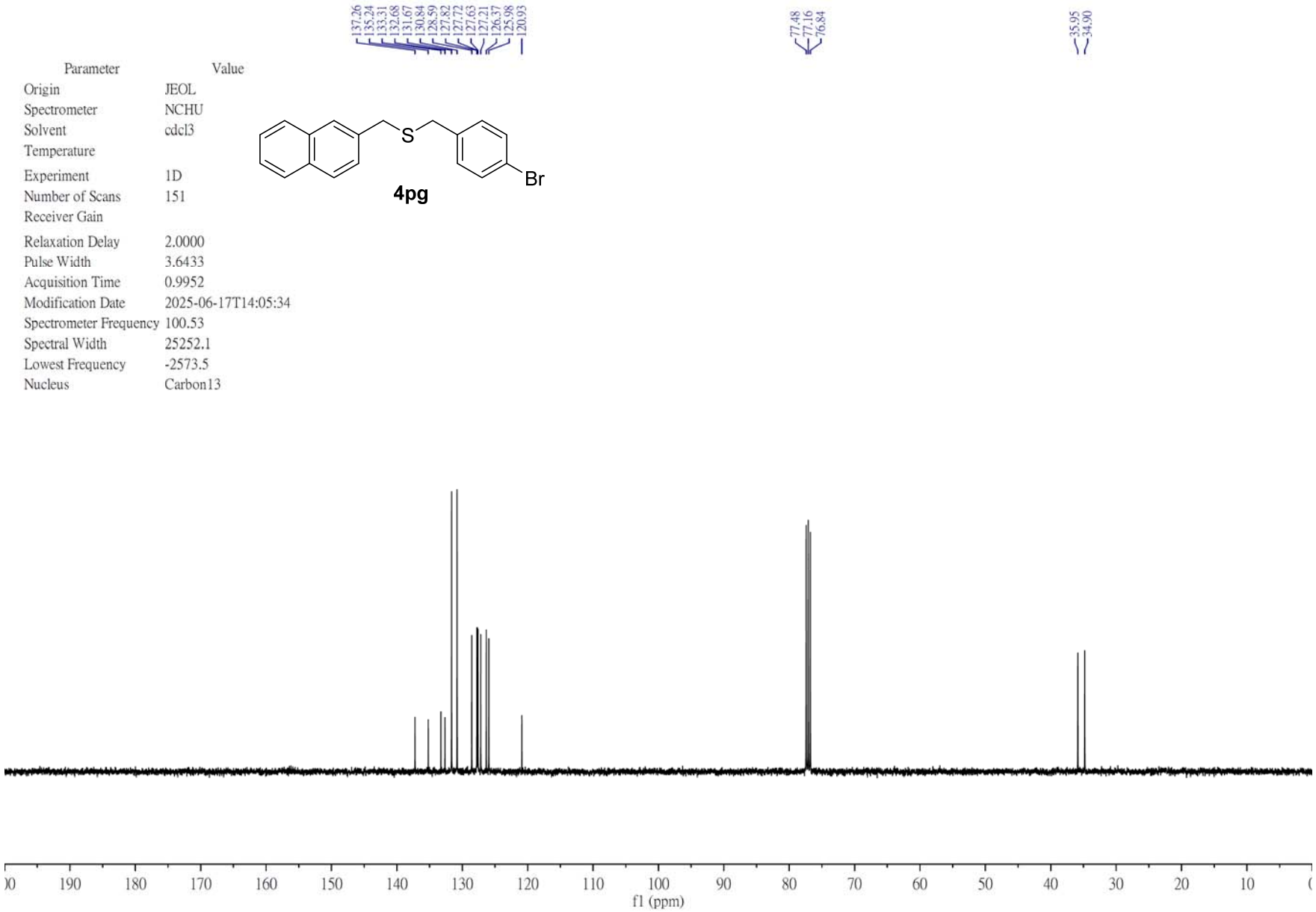
4id ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



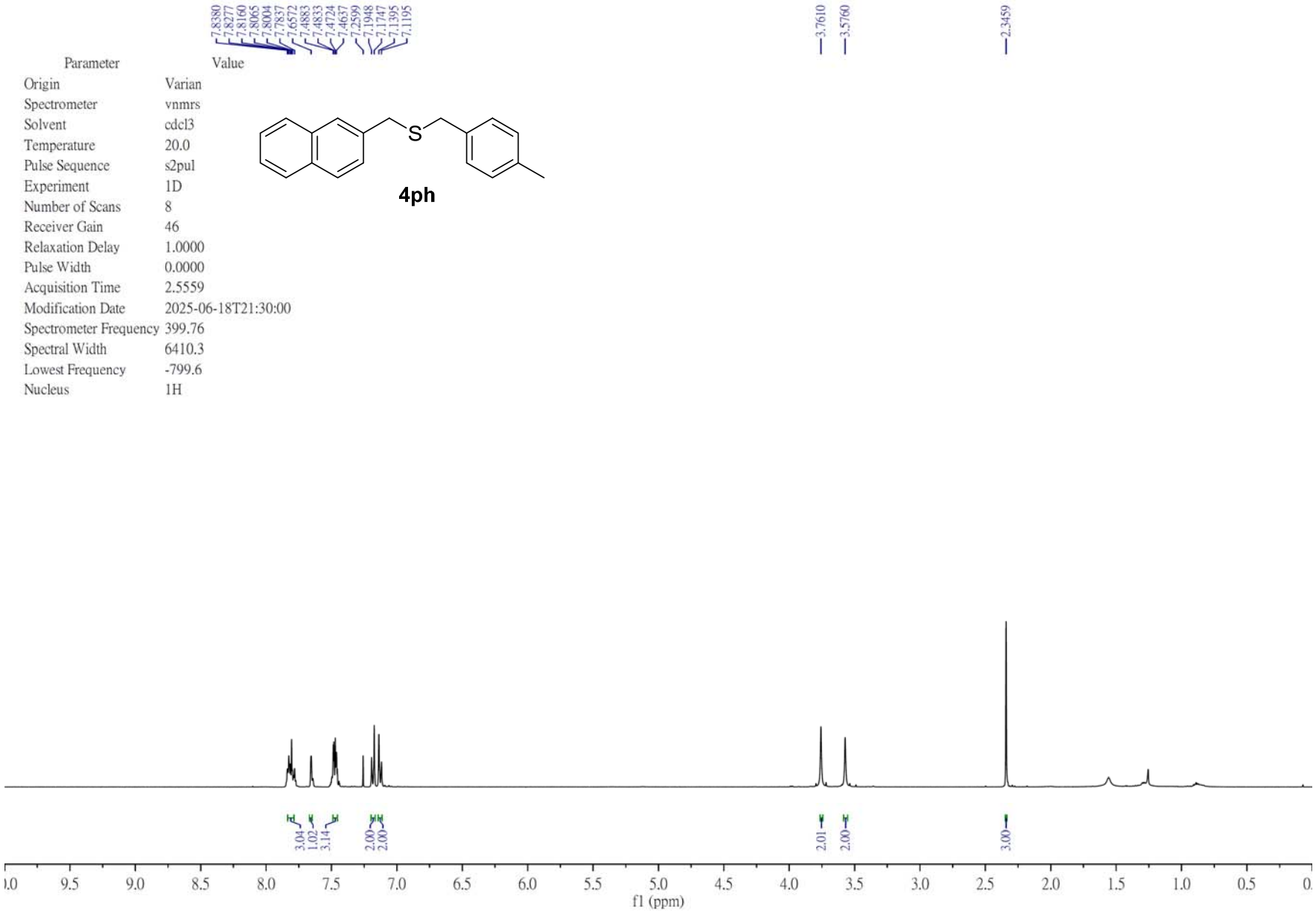
4id $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



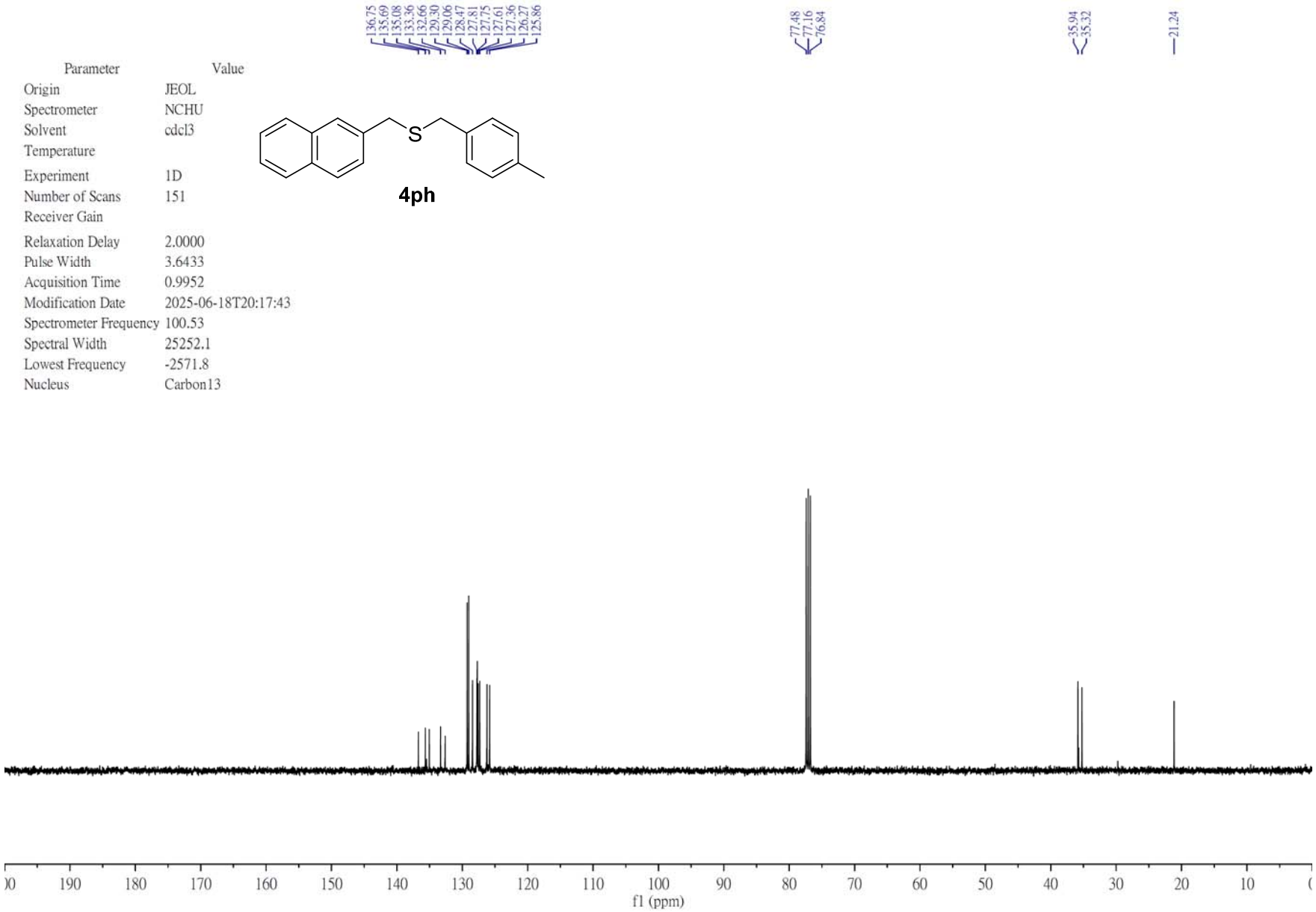
4pg ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4pg $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

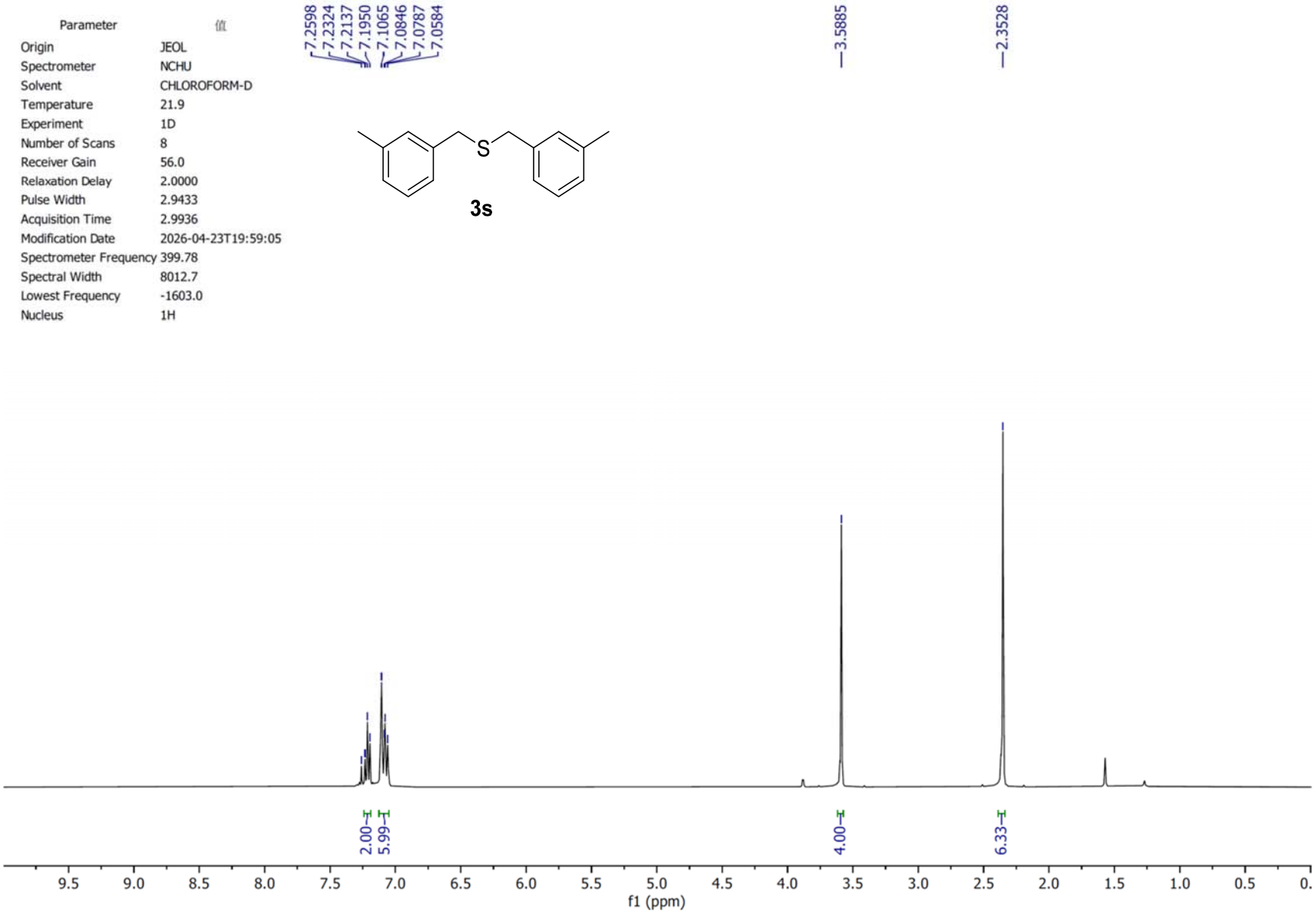


4ph ^{1}H NMR spectrum（400 MHz in $CDCl_3$）

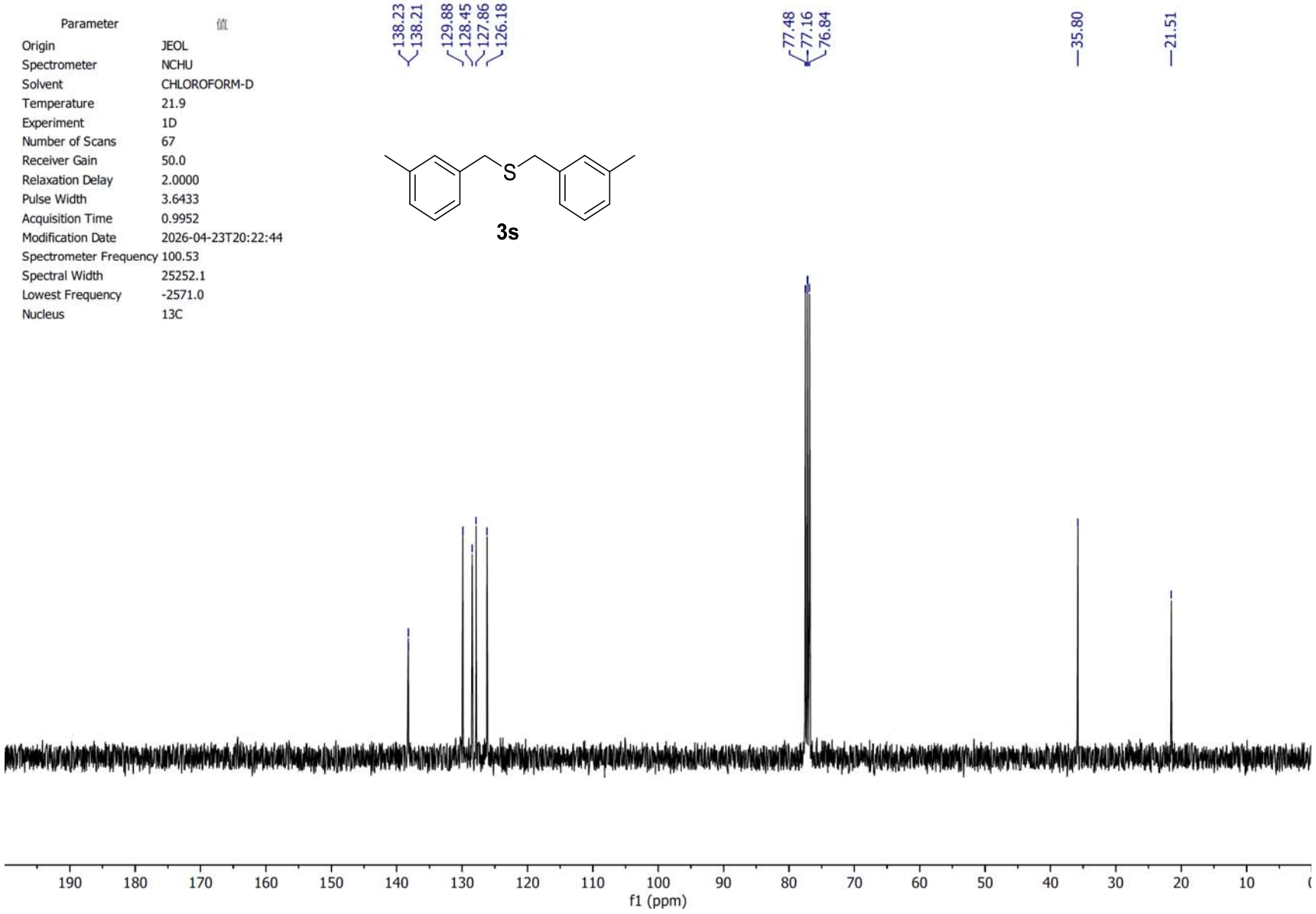


4ph $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）

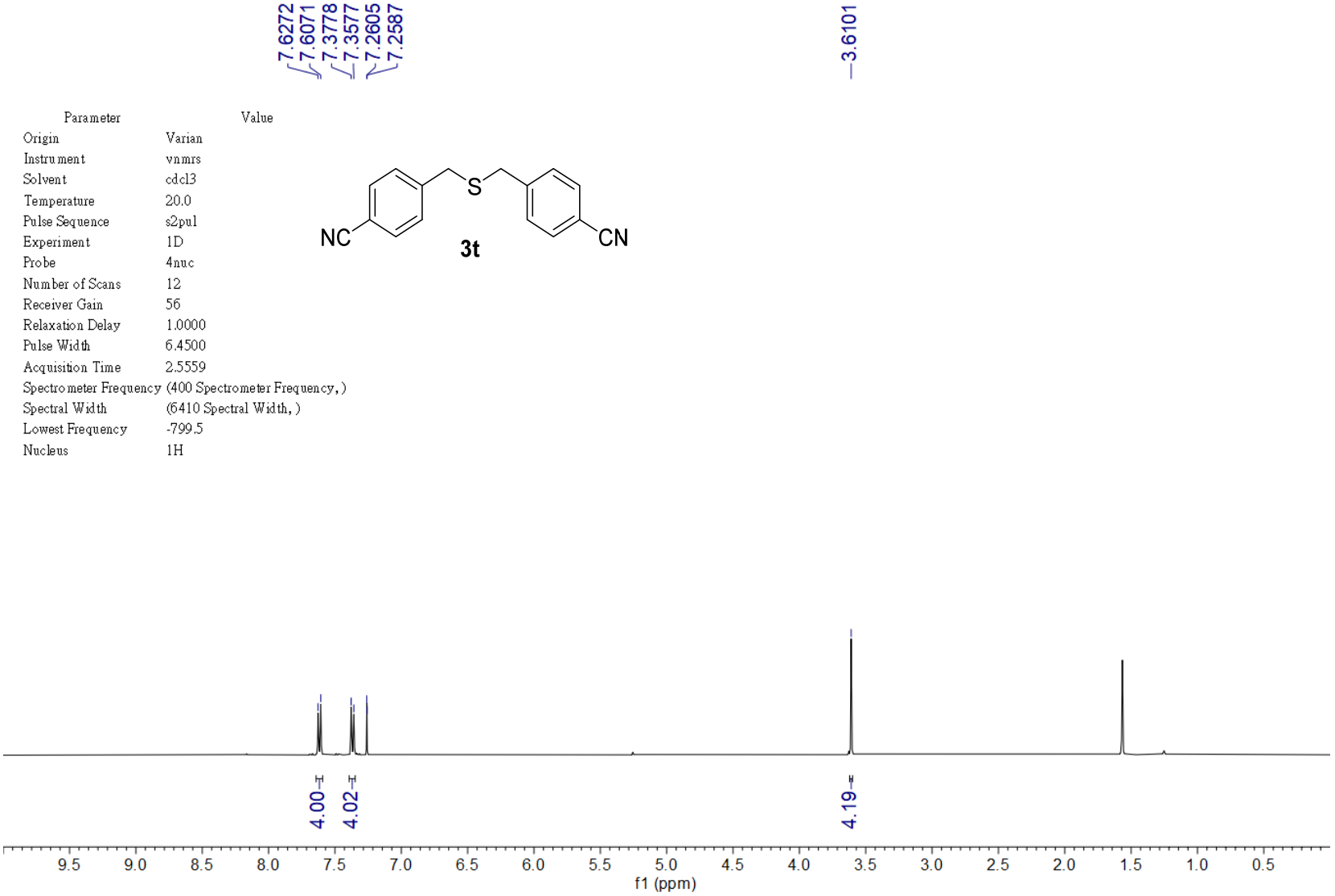
Parameter	值
Origin	JEOL
Spectrometer	NCHU
Solvent	CHLOROFORM-D
Temperature	21.9
Experiment	1D
Number of Scans	8
Receiver Gain	56.0
Relaxation Delay	2.0000
Pulse Width	2.9433
Acquisition Time	2.9936
Modification Date	2026-04-23T19:59:05
Spectrometer Frequency	399.78
Spectral Width	8012.7
Lowest Frequency	-1603.0
Nucleus	1H



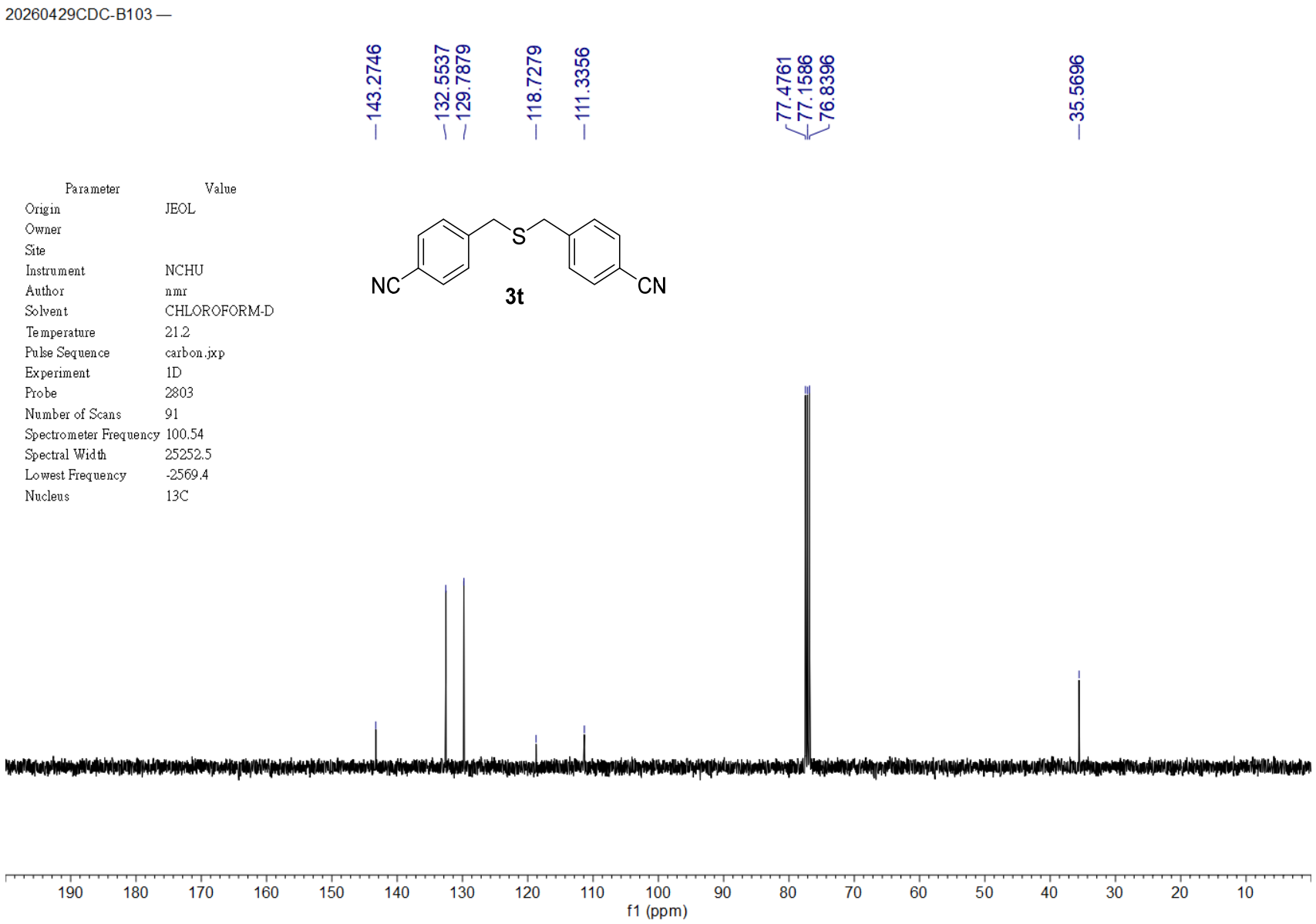
3s ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



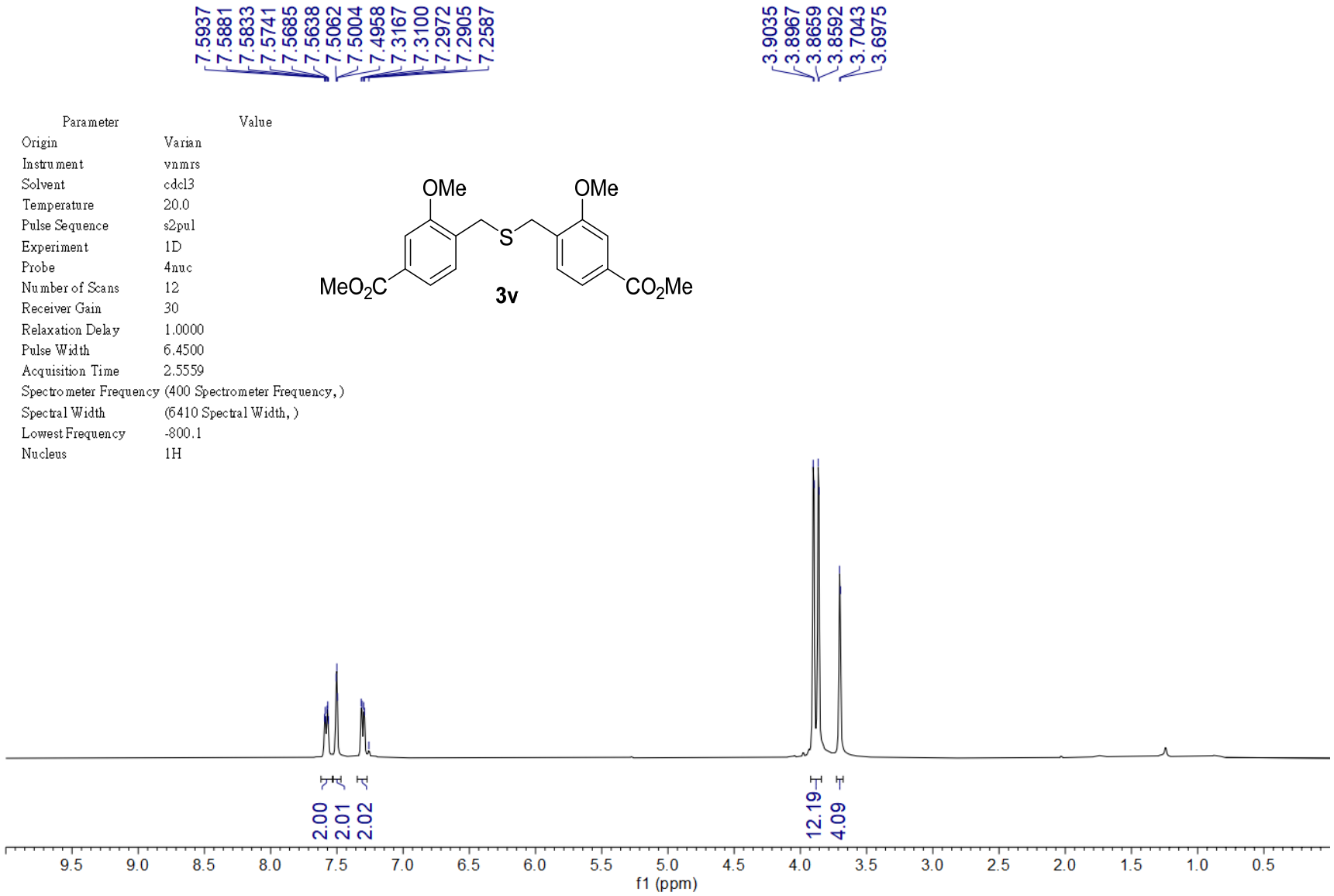
3s $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



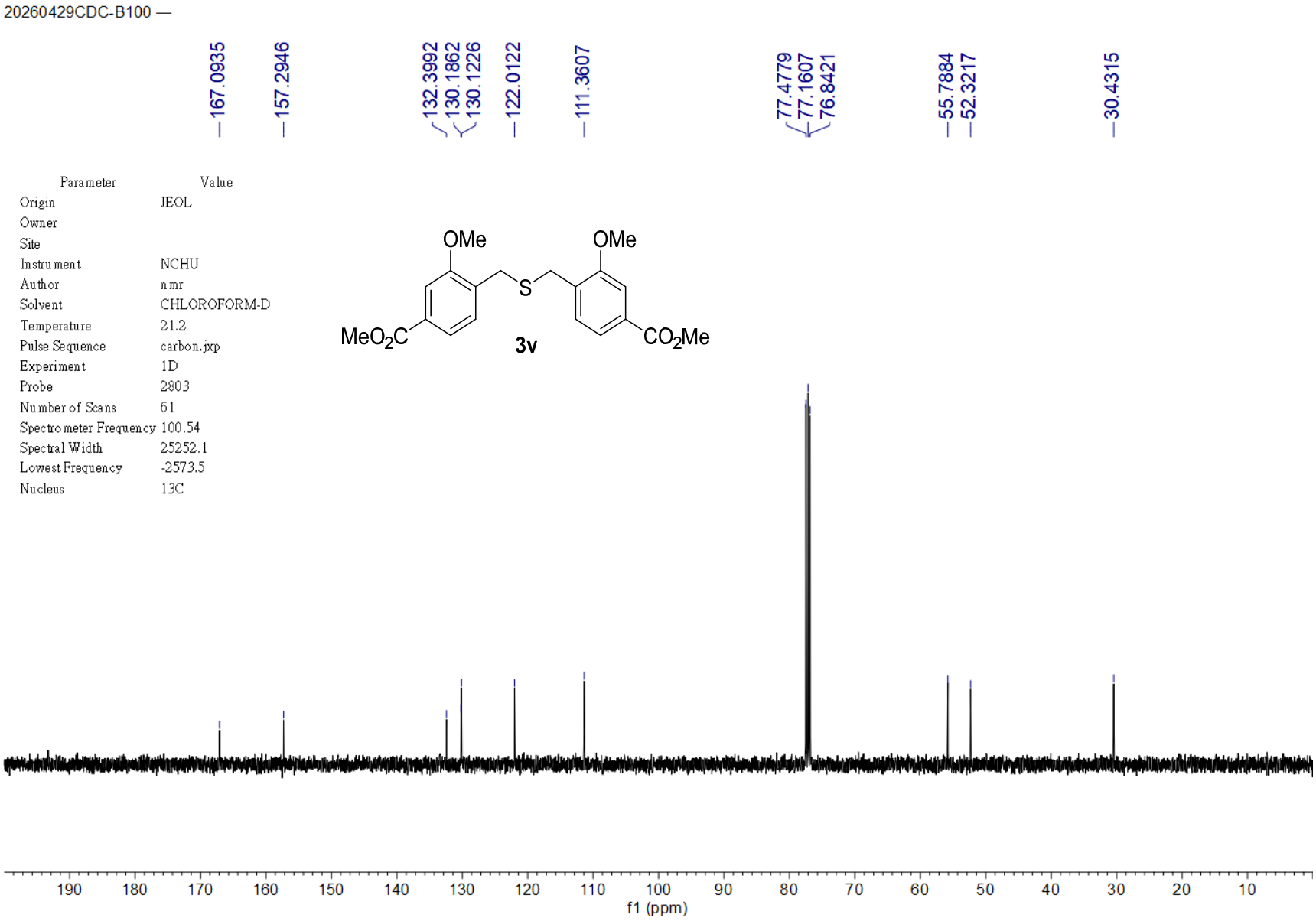
3t ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



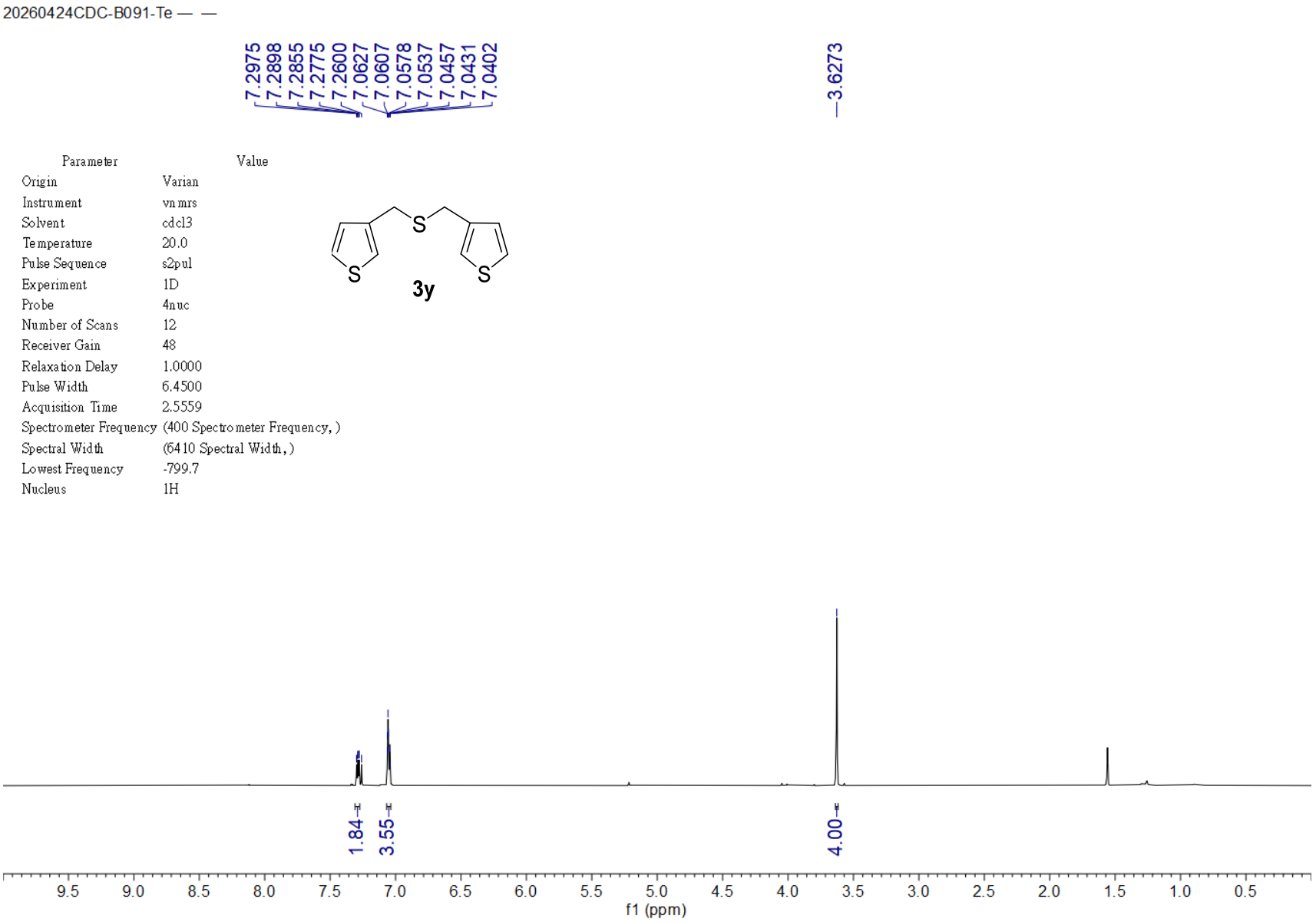
3t $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



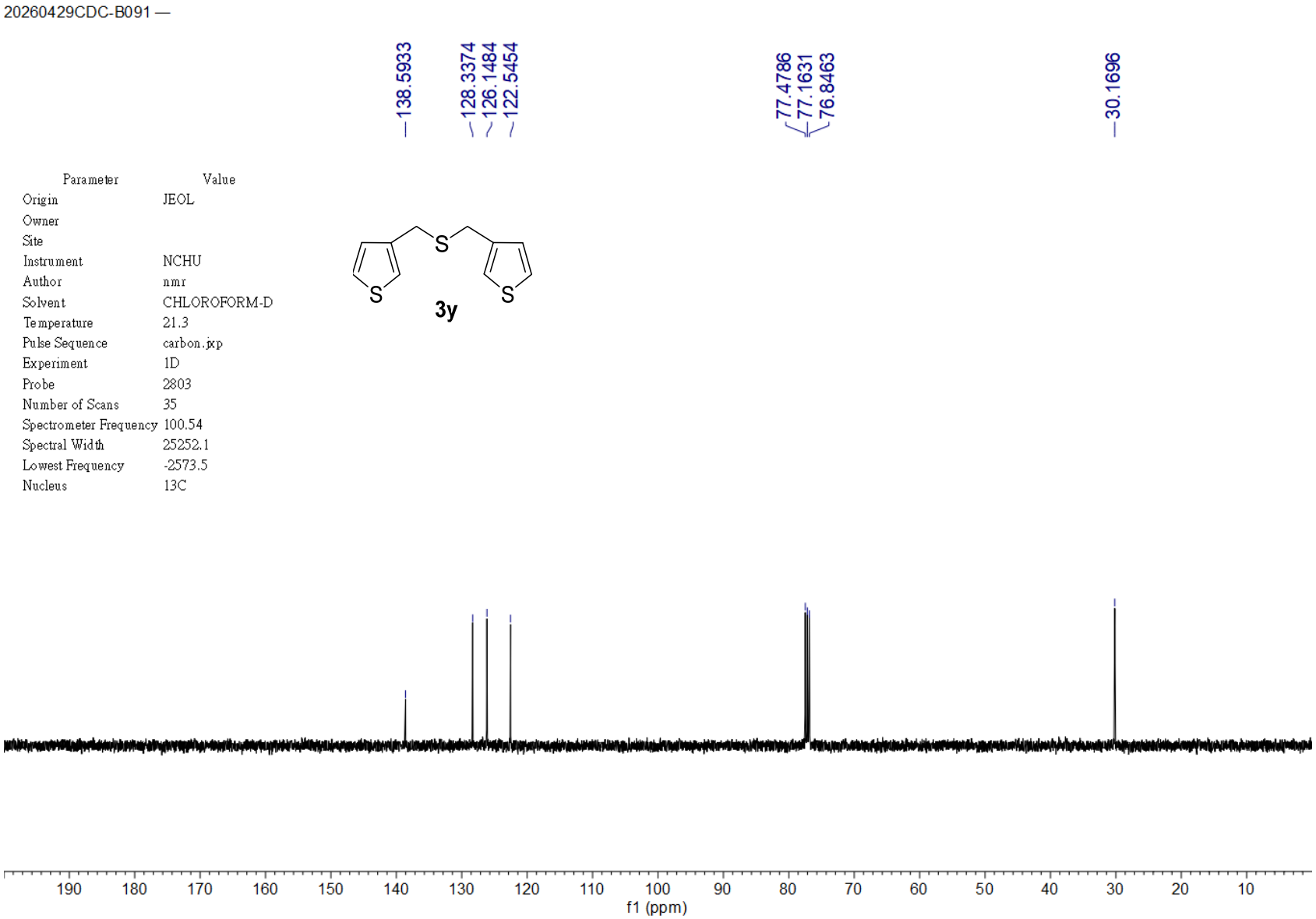
3v ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



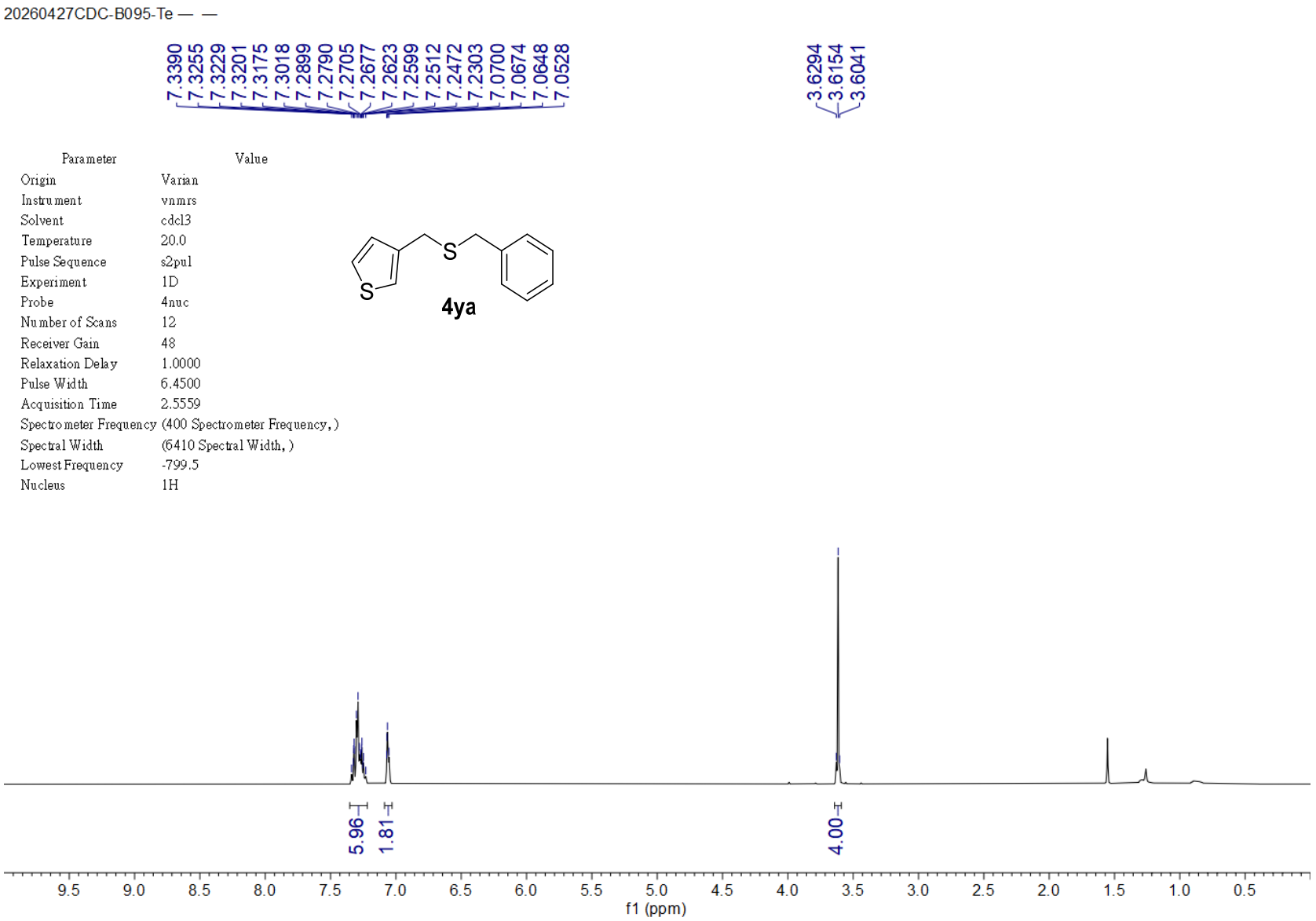
3v $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



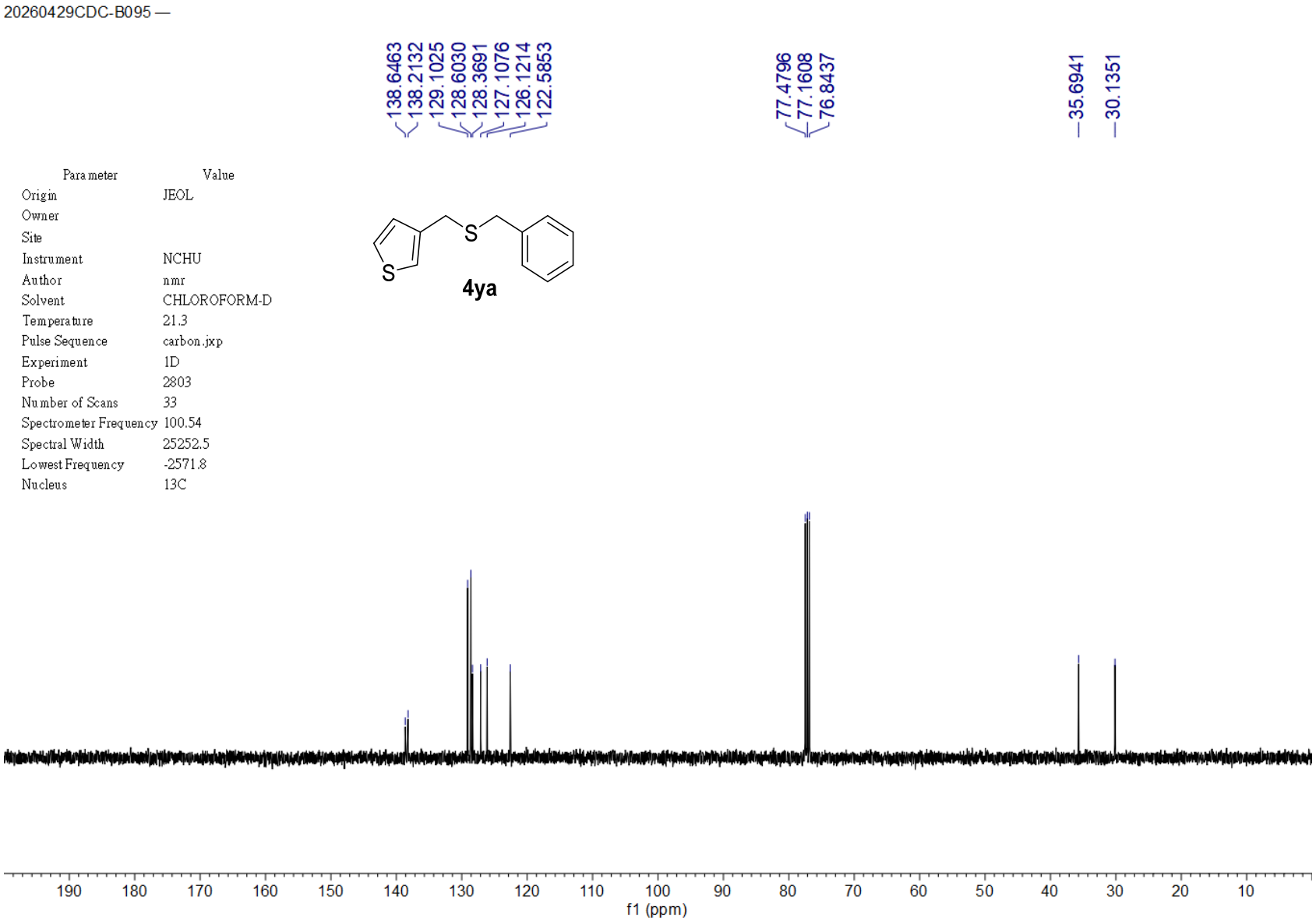
3y ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



3y $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）



4ya ^{1}H NMR spectrum（400 MHz in $CDCl_3$）



4ya $^{13}C\{^{1}H\}$ NMR spectrum（100 MHz in $CDCl_3$）