Supporting Online Material for

Total Syntheses of (+)-Epilupinine *via* An Intramolecular Nitrile Oxide-Alkene Cycloaddition

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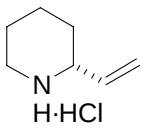
List of ^{1}H NMR and ^{13}C NMR spectra for compounds.

Materials and Methods

General Method: All melting points were determined on a Yanaco melting point apparatus and are uncorrected. IR spectra were recorded on a Nicolet FT-IR 5DX spectrometer. The ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a JEOL JNM-ECA 300 spectrometer in $CDCl_3$ with TMS as internal reference. The *J* values are given in Hz. MS were recorded on a VG-ZAB-MS spectrometer with 70 eV. Elementary analysis data were obtained on a Perkin-Elmer-241C apparatus.

The starting material **3**[18] and the intermediate **4**[10b] were prepared by the reference methods.

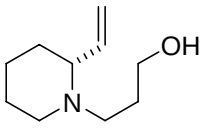


(*R*)-5·HCl

Preparation of (*R*)-2-vinylpiperidine Hydrochloride [(*R*)-5·HCl]: To a cold solution (ice-water bath) of compound **4** (1.02 g, 3 mmol) in dry THF (20 mL) was added $LiAlH_4$ (170 mg, 4.5 mmol) under N_2. After the reaction was stirred at 0 °C for 30 min (monitored by TLC), a saturated aqueous solution of NH_4Cl (30 mL) was added to quench the reaction. Then the resultant mixture was extracted with Et_2O (3 x 10 mL). The combined organic layers were washed with H_2O and brine and dried over Na_2SO_4.

After removal of the solvent, the residue was diluted by a solution of THF (4 mL), CH_3OH (4 mL) and aq. NaOH (6.0 M, 2 mL). The resultant mixture was stirred at 60

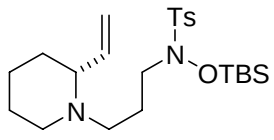
°C for 6 h and then was cooled to room temperature. It was extracted with CH_2Cl_2 (3 x 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. After the solid was filtered off, the filtrate was immediately treated with saturated solution of HCl in MeOH (6 mL) at 0 ^oC for 30 min. Then, the solvent was removed in vacuum and the residue was recrystallized from MeOH-Et_2O to give product **(*R*)-5·HCl** as a white crystal (410 mg, 93%). It had mp 199-200 ^oC (MeOH-Et_2O). $[\alpha]_D^{20}$ = +5.5 (c 0.2, $CHCl_3$); IR: *v* 3440, 2983, 1252, 1013 cm^{-1}. ^1H NMR: δ 9.53-9.40 (m, 2H), 6.13-6.02 (m, 1H), 5.56-5.37 (m, 2H), 3.60-3.43 (m, 2H), 2.95-2.88 (m, 1H), 2.04-1.82 (m, 5H), 1.55-1.52 (m, 1H); ^{13}C NMR: δ 133.3, 120.5, 58.1, 44.2, 28.1, 21.8, 21.5; MS *m/z* (%): 111 (M^+-HCl, 1.71), 84 (100). Anal. Calcd. For $C_7H_{14}ClN$: C, 56.94; H, 9.56; N, 9.49. Found: C, 56.67; H, 9.43; N, 9.66.



11a

Preparation of (*R*)-3-(2-vinylpiperidin-1-yl)propan-1-ol (11a): The mixture of compound **(*R*)-5·HCl** (2.00 g, 13.5 mmol), $ClCH_2CH_2CH_2OH$ (1.42 g, 15 mmol), dry K_2CO_3 (4.14 g, 30 mmol) and KI (2.49 g, 15 mmol) in acetone (50 mL) was refluxed for 2 h. Then the solid was filtrated off and the solvent was evaporated. The residue was purified by chromatography (silica gel, EtOAc) to give 2.18 g (95%) of product **11a** as a colorless oil, $[\alpha]_D^{20}$ = +76.4 (c 0.2, $CHCl_3$). IR: *v* 3387, 2933, 2857, 1053 cm^{-1}; ^1H NMR: δ 5.78-5.67 (m, 1H), 5.49 (s, 1H), 5.10-4.99 (m, 2H), 3.70-3.63 (m, 2H), 3.10-2.87 (m, 2H), 2.53-2.48 (m, 1H), 2.23-2.14 (m, 1H), 1.86-1.25 (m, 9H); ^{13}C NMR:

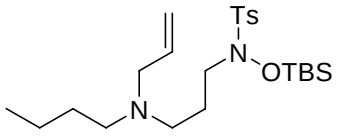
δ141.0, 116.0, 66.9, 64.2, 54.9, 51.7, 33.5, 27.1, 25.5, 23.3; MS *m/z* (%): 169 (M^+, 1.87), 124 (100). Anal. Calcd for $C_{10}H_{19}NO$: C, 70.96; H, 11.31; N, 8.28. Found: C, 71.21; H, 11.24; N, 8.32.



12a

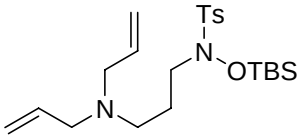
Preparation of *N*-[(2*R*)-2-vinyl-1-piperidine-propyl]-*N*-[(*tert*-butyldimethyl-silyl)oxy]-4-methyl-benzenesulfonamide (12a), **A typical procedure of Mutsunobu reaction:** To a stirred solution of **11a** (508 mg, 3 mmol), TsNHOTBS (915 mg, 3.15 mmol), PPh_3 (1.58 g, 6 mmol) and THF (3 mL) in toluene (9 mL) was added a solution of DEAD (784 mg, 4.5 mmol) in toluene (3 mL) at 0 ^oC under N_2. One hour later, the solvent was removed to give a residue, which was purified by chromatography (silica gel, 10% EtOAc in PE) to give 1.33 g (98%) of product **12a** as a colorless oil, $[\alpha]_D^{20}$ = +21.0 (c 0.2, $CHCl_3$). IR: *v* 2932, 2856, 1465 cm^{-1}; ^1H NMR: δ7.73 (d, *J* = 8.2, 2H), 7.33 (d, *J* = 7.9, 2H), 5.71-5.62 (m, 1H), 5.12-4.98 (m, 2H), 3.02-2.83 (m, 3H), 2.71-2.51 (m, 2H), 2.44 (s, 3H), 2.16-2.06 (m, 1H), 1.97-1.91 (m, 1H), 1.74-1.23 (m, 8H), 0.93 (s, 9H), 0.30 (s, 6H); ^{13}C NMR: δ144.3, 141.8, 129.9, 129.7 (2C), 129.1 (2C), 115.3, 66.4, 54.3, 52.4, 51.9, 33.5, 25.9 (3C), 25.8, 23.7, 23.5, 21.5, 18.0. -4,4 (2C); MS *m/z* (%): 452 (M^+, 1.08), 44 (100). Anal. Calcd for $C_{23}H_{40}N_2O_3SSi$: C, 61.02; H, 8.91; N, 6.19. Found: C, 61.25; H, 8.99; N, 6.06.

By similar procedure, 3-aminopropanols **11b-11k** were converted into the corresponding 4-methyl-benzenesulfonamides **12b-12k**.



12b

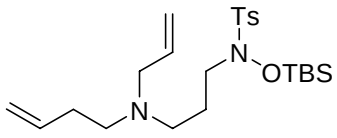
***N*-[3-[*N*-Allyl-*N*-butylamino]propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12b):** It is a yellowish oil (97%); IR: *v* 2956, 2931, 1359 cm^{-1}; ^{1}H NMR: δ 7.73 (d, *J* = 7.9, 2H), 7.33 (d, *J* = 7.9, 2H), 5.86-5.74 (m, 1H), 5.15-5.06 (m, 2H), 3.07-2.92 (m, 4H), 2.45 (s, 3H), 2.40-2.32 (m, 4H), 1.73-1.63 (m, 2H), 1.45-1.19 (m, 5H), 0.92 (s, 9H), 0.90-0.86 (m, 2H), 0.30 (s, 6H); ^{13}C NMR: δ 144.4, 136.0, 129.9, 129.8 (2C), 129.2 (2C), 116.9, 57.1, 54.3, 53.4, 50.9, 29.1, 26.0 (3C), 24.7, 21.6, 20.5, 18.2, 14.0, -4.3 (2C); MS *m/z* (%): 454 (M^{+}, 5.08), 84 (100). Anal. Calcd for $C_{23}H_{42}N_2O_3SSi$: C, 60.75; H, 9.31; N, 6.16. Found: C, 60.51; H, 9.18; N, 6.29.



12c

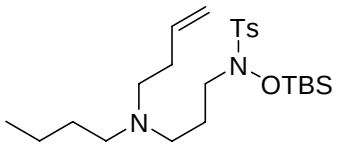
***N*-[3-(*N*,*N*-Diallylamino)propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12c):** It is a yellowish oil (96%); IR: *v* 3074, 2955, 2929, 1471, 1358 cm^{-1}; ^{1}H NMR: δ 7.73 (d, *J* = 7.9, 2H), 7.33 (d, *J* = 7.9, 2H), 5.86-5.74 (m, 2H), 5.16-5.08 (m, 4H), 3.03-3.01 (d, *J* = 6.2, 4H), 2.98-2.92 (t, *J* = 6.9, 2H), 2.44-2.38 (m, 5H), 1.74-1.67 (m, 2H), 0.93 (s, 9H), 0.30 (s, 6H); ^{13}C NMR: δ 144.3, 135.6 (2C),

130.0, 129.8 (2C), 129.1 (2C), 117.1 (2C), 56.7 (2C), 54.2, 50.4, 25.9 (3C), 24.7, 21.5, 18.1, -4.3 (2C). MS *m/z* (%): 438 (M^+, 0.28), 110 (100). Anal. Calcd for $C_{22}H_{38}N_2O_3SSi$: C, 60.23; H, 8.73; N, 6.39. Found: C, 60.46; H, 8.67; N, 6.46.



12d

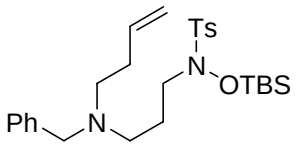
***N*-[3-(*N*-Allyl-*N*-but-3-enyl)aminopropyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12d):** It is a yellowish oil (96%); IR: *v* 3075, 2928, 1598, 1465, 1359 cm^{-1}; ^1H NMR: δ 7.73 (d, *J* = 8.3, 2H), 7.33 (d, *J* = 8.3, 2H), 5.83-5.70 (m, 2H), 5.16-4.94 (m, 4H), 3.04-3.02 (d, *J* = 6.2, 2H), 2.97-2.91 (m, 2H), 2.47-2.38 (m, 7H), 2.14-2.10 (m, 2H), 1.73-1.63 (m, 2H), 0.92 (s, 9H), 0.29 (s, 6H); ^{13}C NMR: δ 144.3, 136.6, 135.7, 129.9, 129.7 (2C), 129.1 (2C), 116.9, 115.3, 56.9, 54.2, 53.0, 50.7, 31.3, 25.9 (3C), 24.7, 21.5, 18.1, -4.3 (2C); MS *m/z* (%): 452 (M^+, 0.24), 84 (100). Anal. Calcd for $C_{23}H_{40}N_2O_3SSi$: C, 61.02; H, 8.91; N, 6.19. Found: C, 61.19; H, 9.10; N, 6.06.



12e

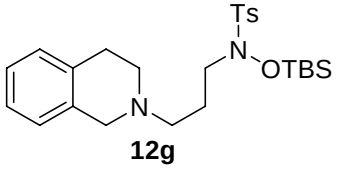
***N*-[3-(*N*-But-3-enyl-*N*-butyl)aminopropyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12e)**: It is a colorless oil (97%). IR: *v* 3073, 2955, 1598, 1462, 1357 cm^{-1}; ^1H NMR: δ 7.73 (d, *J* = 8.3, 2H), 7.33 (d, *J* = 7.9, 2H), 5.80-5.71 (m, 1H), 5.04-4.94 (m, 2H), 2.96-2.91 (m, 2H), 2.45-2.32 (m, 9H), 2.16-2.08 (m, 2H),

1.70-1.60 (m, 2H), 1.37-1.22 (m, 4H), 0.92 (s, 9H), 0.91-0.81 (m, 3H), 0.30 (s, 6H); ^{13}C NMR: δ 144.3, 136.9, 129.9, 129.8 (2C), 129.1 (2C), 115.2, 54.4, 53.5, 53.3, 51.2, 31.4, 29.2, 25.9 (3C), 24.8, 21.5, 20.5, 18.1, 14.0, -4.3 (2C); MS *m/z* (%): 468 (M^+, 1.42), 427 (100). Anal. Calcd for $C_{24}H_{44}N_2O_3SSi$: C, 61.49; H, 9.46; N, 5.98. Found: C, 61.57; H, 9.63; N, 5.88.



12f

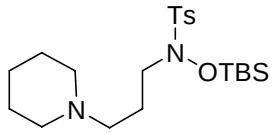
***N*-[3-(*N*-benzyl-*N*-but-3-enyl)aminopropyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12f):** It is a colorless oil (96%); IR: *v* 2959, 2931, 1457, 1358 cm^{-1}; ^{1}H NMR: δ 7.71 (d, *J* = 7.9, 2H), 7.32-7.20 (m, 7H), 5.80-5.65 (m, 1H), 5.02-4.92 (m, 2H), 3.50 (s, 2H), 2.95-2.90 (m, 2H), 2.48-2.37 (m, 7H), 2.23-2.14 (m, 2H), 1.74-1.65 (m, 2H), 0.91 (s, 9H), 0.27 (s, 6H); ^{13}C NMR: δ 144.3, 139.6, 136.8, 129.84, 129.79 (2C), 129.1 (2C), 128.6 (2C), 128.0 (2C), 126.7, 115.4, 58.3, 54.3, 53.0, 50.9, 31.4, 26.0 (3C), 24.8, 21.6, 18.1, -4.3 (2C); MS *m/z* (%): 502 (M^+, 1.10), 91 (100). Anal. Calcd for $C_{27}H_{42}N_2O_3SSi$: C, 64.50; H, 8.42; N, 5.57. Found: C, 64.55; H, 8.36; N, 5.61.



12g

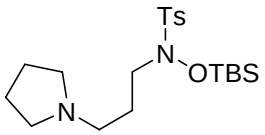
***N*-[3-[3,4-dihydroisoquinolin-2(1*H*)-yl]propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulfonamide (12g):** It is a colorless oil (99%); IR: *v* 3746, 2938,

1462, 1353 cm^{-1}; ^1H NMR: δ 7.73 (d, *J* = 7.9, 2H), 7.29 (d, *J* = 7.9, 2H), 7.12-7.05 (m, 3H), 6.98-6.95 (m, 1H), 3.55 (s, 2H), 3.05-3.00 (m, 2H), 2.86-2.82 (m, 2H), 2.68-2.63 (m, 2H), 2.53-2.47 (m, 2H), 2.40 (s, 3H), 1.88-1.83 (m, 2H), 0.93 (s, 9H), 0.31 (s, 6H); ^{13}C NMR: δ 144.3, 134.6, 134.1, 129.8, 129.7 (2C), 129.1 (2C), 128.4, 126.3, 125.9, 125.4, 55.9, 55.4, 54.2, 50.7, 28.9, 25.9 (3C), 24.7, 21.5, 18.0, -4.4 (2C); MS *m/z* (%): 474 (M^+, 0.69), 146 (100). Anal. Calcd for $C_{25}H_{38}N_2O_3SSi$: C, 63.25; H, 8.07; N, 5.90. Found: C, 63.37; H, 8.12; N, 5.83.



12h

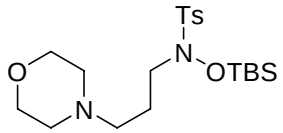
***N*-[3-(Piperidin-1-yl)propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzene sulfonamide (12h)**: It is a colorless oil (96%); IR: *v* 2932, 2857, 2804, 2768, 1467, 1358 cm^{-1}; ^1H NMR: δ 7.73 (d, *J* = 8.3, 2H), 7.34 (d, *J* = 8.3, 2H), 2.97-2.92 (m, 2H), 2.45 (s, 3H), 2.30-2.24 (m, 6H), 1.80-1.71 (m, 2H), 1.58-1.50 (m, 4H), 1.42-1.40 (m, 2H), 0.92 (s, 9H), 0.30 (s, 6H); ^{13}C NMR: δ 144.3, 129.8, 129.7 (2C), 129.1 (2C), 56.4, 54.4, 54.3 (2C), 25.9 (3C), 25.8 (2C), 24.34, 24.26, 21.5, 18.0, -4.4 (2C); MS *m/z* (%): 426 (M^+, 4.68), 271 (100). Anal. Calcd for $C_{21}H_{38}N_2O_3SSi$: C, 59.11; H, 8.98; N, 6.57. Found: C, 59.28; H, 8.79; N, 6.51.



12i

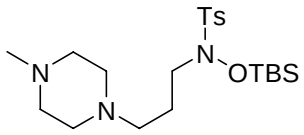
***N*-[3-(Pyrrolidin-1-yl)propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzen**

esulfonamide (12i): It is a colorless oil (93%); IR: *v* 2957, 2931, 2858, 2791, 1466, 1357 cm^{-1}; ^{1}H NMR: δ 7.73 (d, *J* = 7.9, 2H), 7.33 (d, *J* = 7.9, 2H), 3.00-2.96 (m, 2H), 2.44-2.39 (m, 9H), 1.83-1.74 (m, 6H), 0.92 (s, 9H), 0.30 (s, 6H); ^{13}C NMR: δ 144.4, 129.9, 129.8 (2C), 129.2 (2C), 54.4, 54.1 (2C), 53.8, 26.6, 25.9 (3C), 23.3 (2C), 21.6, 18.1, -4.3 (2C); MS *m/z* (%): 412 (M^{+}, 0.22), 84 (100). Anal. Calcd for $C_{20}H_{36}N_2O_3SSi$: C, 58.21; H, 8.79; N, 6.79. Found: C, 58.39; H, 8.67; N, 6.91.



12j

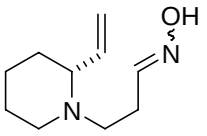
***N*-(3-Morpholinopropyl)-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl-benzenesulf onamide (12j)**: It is a colorless oil (98%); IR: *v* 2943, 1358, 1169 cm^{-1}; ^{1}H NMR: δ 7.73 (d, *J* = 7.9, 2H), 7.34 (d, *J* = 7.9, 2H), 3.69-3.66 (m, 4H), 3.00-2.95 (m, 2H), 2.45 (s, 3H), 2.40-2.31 (m, 6H), 1.78-1.73 (m, 2H), 0.92 (s, 9H), 0.30 (s, 6H); ^{13}C NMR: δ 144.4, 129.8, 129.7 (2C), 129.1 (2C), 66.8 (2C), 55.9, 54.1, 53.4 (2C), 25.9 (3C), 24.0, 21.5, 18.0, -4.4 (2C); MS *m/z* (%): 428 (M^{+}, 0.53), 100 (100); Anal. Calcd for $C_{20}H_{36}N_2O_4SSi$: C, 56.04; H, 8.46; N, 6.54. Found: C, 56.21; H, 8.30; N, 6.63.



12k

***N*-[3-(4-Methylpiperazin-1-yl)propyl]-*N*-(*tert*-butyldimethylsilyloxy)-4-methyl -benzenesulfonamide (12k)**: It is a colorless oil (93%), IR: *v* 2934, 1462, 1358 cm^{-1}; ^{1}H NMR: δ 7.73 (d, *J* = 8.3, 2H), 7.34 (d, *J* = 8.3, 2H), 2.99-2.93 (m, 2H), 2.45-2.28

(m, 16H), 1.76-1.71 (m, 2H), 0.92 (s, 9H), 0.29 (s, 6H); ^{13}C NMR: δ 144.3, 129.8, 129.7 (2C), 129.1 (2C), 55.5, 54.9 (2C), 54.1, 52.9 (2C), 45.9, 25.9 (3C), 24.2, 21.5, 18.0, -4.4 (2C); MS *m/z* (%): 442 (M^+ + H, 100). Anal. Calcd for $C_{21}H_{39}N_3O_3SSi$: C, 57.10; H, 8.90; N, 9.51. Found: C, 57.29; H, 8.73; N, 9.48.

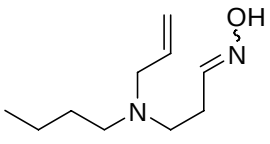


13a

Preparation of (*R*)-2-Vinyl-1-piperidinepropanal Oxime (13a), **A Typical Procedure of Oximation:** A stirred mixture of compound **12a** (453 mg, 1.0 mmol) and CsF (301 mg, 2 mmol) in MeCN (10 mL) was heated at 60 ^{o}C under N_2 for 2 h. After it was cooled to room temperature, saturated aqueous NH_4Cl was added to quench the reaction. The resultant mixture was extracted by EtOAc (3 x 30 mL) and combined organic layers were washed with brine (20 mL) and dried over Na_2SO_4. Removal of the solvent gave a residue, which was purified by chromatography (silica gel, 30% EtOAc in PE) to give 171 mg (94%, *E*:*Z* = 72:28) of product **13a** as a white crystal. It had mp 43-45 ^{o}C (PE/EtOAc), $[\alpha]_D^{20}$ = +11.2 (c 0.2, $CHCl_3$). IR: *v* 3305, 3179, 3073, 2931, 2854, 2789, 2726 cm^{-1}; ^{1}H NMR (as a mixture of *E*:*Z* isomers): δ 11.06 (s, br, 1H), 7.37-7.32 (m, 0.28H, minor), 6.70-6.67 (m, 0.72H, major), 5.88-5.74 (m, 1H), 5.18-5.04 (m, 2H), 3.02-2.87 (m, 2H), 2.75-2.63 (m, 1H), 2.62-2.33 (m, 3H), 2.17-2.08 (m, 1H), 1.74-1.45 (m, 5H), 1.37-1.25 (m, 1H); ^{13}C NMR: δ 149.8, 149.1, 140.7, 115.9, 66.2, 66.0, 51.9, 51.7, 51.5, 51.2, 32.8, 25.8, 25.2, 23.5, 21.4; MS *m/z* (%): 182 (M^+, 0.42), 124 (100). Anal. Calcd for $C_{10}H_{18}N_2O$: C, 65.90; H, 9.95; N, 15.37; Found: C, 65.95; H,

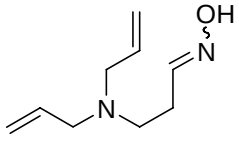
9.92; N,15.26.

By similar procedure, 4-methyl-benzenesulfonamides **12b-12k** were converted into **13b-13k**.



13b

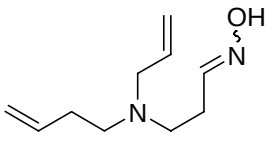
3-(*N*-Allyl-*N*-butylamino)propanal oxime (13b): It is a colorless oil (93%, dr = 72:28); IR: *v* 3207, 3077, 2957 cm^{-1}; ^{1}H NMR: δ10.40 (s, br, 1H), 7.42-7.38 (m, 0.28H, minor), 6.78-6.74 (m, 0.72H, major), 5.94-5.80 (m, 1H), 5.21-5.12 (m, 2H), 3.15-3.13 (m, 2H), 2.70-2.63 (m, 2H), 2.56-2.32 (m, 4H), 1.46-1.41 (m, 2H), 1.35-1.23 (m, 2H), 0.90 (t, *J* = 7.2, 3H), ^{13}C NMR: δ150.2, 149.7, 135.2, 117.7, 56.9, 56.8, 53.1, 53.0, 50.2, 49.4, 28.6, 28.5, 26.9, 22.5, 20.6, 14.0; MS *m/z* (%): 184 (M^{+}, 0.22), 84 (100). Anal. Calcd for $C_{10}H_{20}N_2O$: C, 65.18; H, 10.94; N, 15.20; Found: C, 65.35; H, 11.07; N, 15.04.



13c

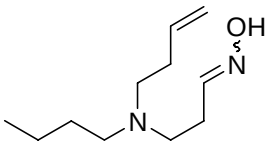
3-(*N*,*N*-Diallylamino)propanal oxime (13c): It is colorless oil (99%, dr = 66:34); IR: *v* 3200, 3076, 2977, 997 cm^{-1}; ^{1}H NMR: δ 9.00 (br, 0.66H), 8.25 (br, 0.34H), 7.44-7.40 (m, 0.34H, minor), 6.81-6.78 (m, 0.66H, major), 5.92-5.78 (m, 2H), 5.22-5.14 (m, 4H), 3.13 (d, *J* = 5.9, 4H); 2.68-2.62 (m, 2H), 2.56-2.50 (m, 1H), 2.40-2.33 (m, 1H); ^{13}C NMR: δ149.9; 149.4; 134.7, 134.6, 118.1, 118.0, 56.4, 56.2,

49.7, 48.9, 26.8, 22.3. MS *m/z* (%): 168 (M^+, 0.15), 110 (100). Anal. Calcd for $C_9H_{16}N_2O$: C, 64.25; H, 9.59; N, 16.65. Found: C, 64.07; H, 9.75; N, 16.46.



13d

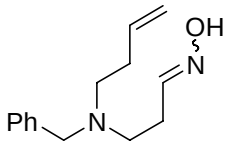
3-(*N*-Allyl-*N*-but-3-enyl)aminopropanal oxime (13d): It is a yellowish oil (97%, dr = 60:40); IR: *v* 3212, 3075, 2923, 2818 cm^{-1}; ^1H NMR: δ 10.18 (s, br, 0.6H), 9.74 (s, br, 0.4H), 7.43-7.39 (m, 0.4H, minor), 6.79-6.76 (m, 0.6H, major), 5.94-5.69 (m, 2H), 5.22-4.95 (m, 4H), 3.17-3.14 (m, 2H), 2.72-2.65 (m, 2H), 2.59-2.51 (m, 3H), 2.40-2.34 (m, 1H), 2.27-2.22 (m, 2H); ^{13}C NMR: δ 150.3, 149.9, 136.4, 135.0, 134.9, 117.8, 115.7, 56.8, 56.7, 52.7, 52.6, 50.2, 49.4, 31.1, 31.0, 27.0, 22.6; MS *m/z* (%): 182 (M^+, 0.05), 123 (100). Anal. Calcd for $C_{10}H_{18}N_2O$: C, 65.90; H, 9.95; N, 15.37. Found: C, 66.03; H, 9.91; N, 15.54.



13e

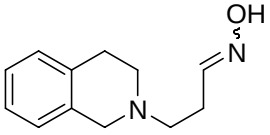
3-(but-3-enyl(butyl)amino)propanal oxime (13e): It is a yellowish oil (93%, dr = 67:33); IR: *v* 3216, 3076, 2956, 1080 cm^{-1}; ^1H NMR: δ10.06 (br, 1H), 7.44-7.40 (m, 0.33H, minor), 6.80-6.76 (m, 0.67H, major), 5.85-5.70 (m, 1H), 5.08-4.96 (m, 2H), 2.68-2.64 (m, 2H), 2.57-2.33 (m, 6H), 2.27-2.21 (m, 2H), 1.50-1.41 (m, 2H), 1.36-1.24 (m, 2H). 0.90 (t, *J* = 7.2, 3H); ^{13}C NMR: δ 150.4, 150.0, 136.5, 115.6, 55.33, 55.27, 53.1, 53.0, 50.6, 49.9, 31.1, 31.0, 28.7, 28.6, 27.1, 22.8, 20.6, 14.0; MS *m/z* (%): 198

(M^+, 0.28), 139 (100). Anal. Calcd for $C_{11}H_{22}N_2O$: C, 66.62; H, 11.18; N, 14.13. Found: C, 66.79; H, 11.03; N, 14.02.



13f

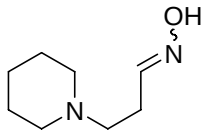
3-(*N*-Benzyl-*N*-but-3-enyl)aminopropanal oxime (13f): It is a yellowish oil (98%, dr = 70:30); IR: *v* 3221, 3068, 2940, 1453, 1368 cm^{-1}; ^1H NMR: δ10.31 (br, 1H), 7.40-7.18 (m, 5.30H), 6.75-6.72 (m, 0.70H, major), 5.80-5.67 (m, 1H), 5.04-4.94 (m, 2H), 3.58 (s, 2H), 2.62-2.20 (m, 8H); ^{13}C NMR: δ150.6, 150.2, 138.7, 138.6, 136.5, 128.8 (2C), 128.0 (2C), 126.8, 115.5, 58.0, 57.9, 52.6, 52.5, 50.2, 49.3, 31.1, 31.0, 27.0, 22.6; MS *m/z* (%): 232 (M^+, 0.72), 91 (100). Anal. Calcd for $C_{14}H_{20}N_2O$: C, 72.38; H, 8.68; N, 12.06. Found: C, 72.23; H, 8.77; N, 12.20.



13g

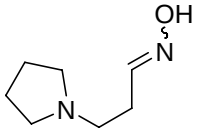
3-(3,4-Dihydroisoquinolin-2(1H)-yl)propanal oxime (13g): It is a white crystal (95%, dr = 64:36), mp 59-61 ^oC (PE/EtOAc); IR: *v* 3452, 3281, 2825, 935 cm^{-1}; ^1H NMR: δ9.91 (s, br, 1H), 7.45-7.40 (m, 0.36H, minor), 7.12-7.09 (m, 3H), 7.01-6.99 (m, 1H), 6.82-6.80 (m, 0.64H, major), 3.67 (s, 2H), 2.90-2.83 (m, 2H), 2.79-2.74 (m, 2H), 2.69-2.66 (m, 3H), 2.60-2.54 (m, 1H); ^{13}C NMR: δ150.1, 149.6, 134.1, 134.0, 128.5, 126.5, 126.1, 125.6, 55.4, 54.6, 54.0, 50.5, 50.4, 28.52, 28.47, 27.3, 23.0; MS *m/z* (%): 204 (M^+, 0.36), 146 (100). Anal. Calcd for $C_{12}H_{16}N_2O$: C, 70.56; H, 7.90; N, 13.71.

Found: C, 70.75; H, 7.94; N, 13.87.



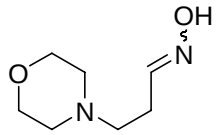
13h

3-(Piperidin-1-yl)propanal oxime (13h): It is a white crystal (94%, dr = 30:70), mp 85-87 ^{o}C (PE/EtOAc); IR: *v* 3411, 3248, 2945, 1123 cm^{-1}; ^{1}H NMR: δ 7.46-7.43 (m, 0.30H, minor), 6.86-6.85 (m, 0.70H, major), 2.58-2.39 (m, 8H), 1.65-1.57 (m, 4H), 1.49-1.34 (m, 2H); ^{13}C NMR: δ 149.7, 149.1, 55.6, 55.1, 54.1, 54.0, 26.8, 25.3, 25.2, 24.0, 22.7; MS *m/z* (%): 156 (M^{+}, 0.10), 98 (100). Anal. Calcd for $C_8H_{16}N_2O$: C, 61.50; H, 10.32; N, 17.93. Found: C, 61.33; H, 10.39; N, 17.80.



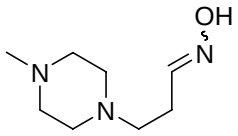
13i

3-(Pyrrolidin-1-yl)propanal oxime (13i): It is a yellow oil (70%, dr = 62:38); IR: *v* 3181, 2962, 2808, 1455, 910 cm^{-1}; ^{1}H NMR: δ 9.98 (s, br, 1H), 7.44-7.40 (m, 0.38H, minor), 6.80-6.76 (m, 0.62H, major), 2.69-2.40 (m, 8H), 1.85-1.76 (m, 4H); ^{13}C NMR: δ 149.4, 148.9, 53.8, 53.7, 53.0, 52.4, 29.0, 24.6, 23.2; MS *m/z* (%): 142 (M^{+}, 0.41), 84 (100). Anal. Calcd for $C_7H_{14}N_2O$: C, 59.12; H, 9.92; N, 19.70. Found: C, 59.33; H, 9.83; N, 19.89.



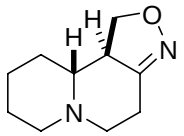
13j

3-Morpholinopropanal oxime (13j): It is a yellowish oil (89%, dr = 57:43); IR: *v* 3185, 3077, 2957, 2862, 1454, 1306 cm^{-1}; ^{1}H NMR: δ 9.64 (s, br, 0.43H), 9.23 (s, br, 0.57H), 7.46-7.42 (m, 0.43H, minor), 6.80-6.77 (m, 0.57H, major), 3.75-3.72 (m, 4H), 2.63-2.37 (m, 8H); ^{13}C NMR: δ 150.1, 149.7, 66.7, 66.6, 55.5, 54.9, 53.32, 53.25, 26.8, 22.4; MS *m/z* (%): 158 (M^+, 0.26), 100 (100). Anal. Calcd for $C_7H_{14}N_2O_2$: C, 53.15; H, 8.92; N, 17.71. Found: C, 53.02; H, 8.90; N, 17.53.



13k

3-(4-Methylpiperazin-1-yl)propanal oxime (13k): It is a colorless oil (65%, dr = 53:47); IR: *v* 3213, 2936, 1458, 1152 cm^{-1}; ^{1}H NMR: δ 8.55 (s, br, 1H), 7.42-7.39 (m, 0.47H, minor), 6.76-6.72 (m, 0.53H, major), 2.69-2.55 (m, 10H), 2.47-2.35 (m, 5H); ^{13}C NMR: δ 149.4, 148.8, 54.7, 54.2, 54.1, 51.52, 51.45, 45.0, 27.0, 22.6; MS *m/z* (%): 171 (M^+, 5.57), 113 (100). Anal. Calcd for $C_8H_{17}N_3O$: C, 56.11; H, 10.01; N, 24.54. Found: C, 56.32; H, 10.06; N, 24.63.

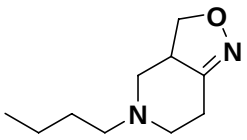


14a

Preaparation of (10a*R*,9b*R*)-octahydro-1H-isoxazolo[4,3-a]quinolizine (14a), A typical procedure of INOC: To a stirred solution of compound **13a** (91 mg, 0.5 mmol)

in CH_2Cl_2 (5 mL) was added an aqueous solution of NaOCl (10%, 0.9 mL) at 0 ^oC within 30 min. Then the reaction was stirred at room temperature for 2 h (monitored by TLC). After the reaction was quenched by H_2O (20 mL), the resultant mixture was extracted by CH_2Cl_2 (2 x 20 mL). The combined organic layers were washed with brine (20 mL) and dried over Na_2SO_4. Removal of the solvent gave a residue, which was purified by chromatography (silica gel, 50% EtOAc in PE) to give 79 mg (88%) of product **14a** as a yellowish oil, $[\alpha]_D^{20}$ = -30.4 (c 0.9, $CHCl_3$). IR: *v* 2929, 2856, 2800, 2763 cm^{-1}; ^1H NMR: δ 4.50-4.43 (m, 1H), 3.83-3.77 (m, 1H), 3.08-2.93 (m, 3H), 2.76-2.70 (m, 1H), 2.55-2.45 (m, 1H), 2.20-2.03 (m, 2H), 1.80-1.54 (m, 5H), 1.38-1.16 (m, 2H); ^{13}C NMR: δ 157.7, 70.5, 67.3, 55.2, 55.1, 54.0, 32.3, 25.2, 24.7, 23.4. MS *m/z* (%): 180 (M^+, 42.12), 179 (100). Anal. Calcd for $C_{10}H_{16}N_2O$: C, 66.63; H, 8.95; N, 15.54. Found: C, 66.81; H, 8.89; N, 15.42.

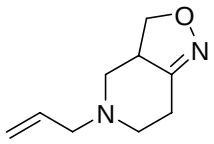
By similar procedure, 3-aminopropanal oximes **13b-13d** were converted into **14b-14d.**



14b

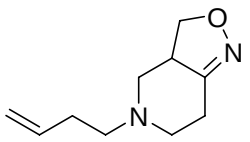
5-Butyl-3,3a,4,5,6,7-hexahydroisoxazolo[4,3-c]pyridine (14b): It is a yellow oil (90%); IR: *v* 2956, 2928, 1134 cm^{-1}; ^1H NMR: δ 4.49-4.43 (m, 1H), 3.80-3.74 (m, 1H), 3.46-3.40 (m, 1H), 3.30-3.23 (m, 1H), 3.17-3.12 (m, 1H), 2.73-2.68 (m, 1H), 2.50-2.38 (m, 3H), 2.13-1.95 (m, 2H), 1.53-1.43 (m, 2H), 1.40-1.25 (m, 2H), 0.93 (t, *J* = 7.2, 3H); ^{13}C NMR: δ 158.3, 70.7, 58.4, 57.0, 52.8, 47.7, 29.1, 24.9, 20.4, 13.8; MS *m/z* (%):

182 (M^+, 25), 139 (100). Anal. Calcd for $C_{10}H_{18}N_2O$: C, 65.90; H, 9.95; N, 15.37. Found: C, 66.04; H, 10.02; N, 15.43.



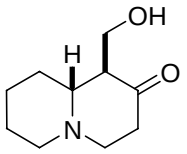
14c

5-Allyl-3,3a,4,5,6,7-hexahydroisoxazolo[4,3-c]pyridine (14c): It is a yellowish oil (86%); IR: *v* 3076, 2955, 2911, 1464, 1348 cm^{-1}; ^1H NMR: δ 5.92-5.78 (m, 1H), 5.24-5.18 (m, 2H), 4.51-4.44 (m, 1H), 3.82-3.75 (m, 1H), 3.50-3.03 (m, 5H), 2.74-2.71 (m, 1H), 2.51-2.39 (m, 1H), 2.13-1.98 (m, 2H); ^{13}C NMR: δ 157.9, 134.3, 118.1, 70.6, 60.2, 57.8, 52.4, 47.5, 24.7; MS *m/z* (%): 166 (M^+, 43), 41 (100). Anal. Calcd for $C_9H_{14}N_2O$: C, 65.03; H, 8.49; N, 16.85. Found: C, 65.20; H, 8.58; N, 16.81.



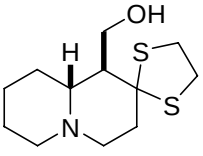
14d

5-(But-3-enyl)-3,3a,4,5,6,7-hexahydroisoxazolo[4,3-c]pyridine (14d): It is a yellow oil (85%). IR: *v* 2926, 2804, 1361, 913, 845 cm^{-1}; ^1H NMR: δ 5.88-5.73 (m, 1H), 5.13-5.00 (m, 2H), 4.52-4.44 (m, 1H), 3.82-3.75 (m, 1H), 3.46-3.37 (m, 1H), 3.31-3.25 (m, 1H), 3.19-3.13 (m, 1H), 2.75-2.69 (m, 1H), 2.58-2.39 (m, 3H), 2.31-2.24 (m, 2H), 2.18-2.03 (m, 2H); ^{13}C NMR: δ 158.2, 136.0, 115.8, 70.7, 58.2, 56.6, 52.6, 47.7, 31.4, 24.9; MS *m/z* (%): 180 (M^+, 0.93), 139 (100). Anal. Calcd for $C_{10}H_{16}N_2O$: C, 66.63; H, 8.95; N, 15.54. Found: C, 66.68; H, 8.81; N, 15.66.



15

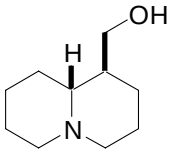
(1*S*,9a*R*)-Octahydro-1-hydroxymethyl-2*H*-quinolizin-2-one (15): At room temperature, a suspension of compound **14a** (400 mg, 2.22 mmol), Raney nickel (13 mg, 0.22 mmol) and HOAc (1.33 g, 22.2 mmol) in aqueous MeOH (75%, 20 mL) under H_2 (balloon) was vigorously stirred for 1.5 h. Then the reaction was quenched by saturated aqueous Na_2CO_3 (20 mL) and the catalyst was filtrated off. The filtration was extracted with EtOAc (3 x 20 mL) and combined organic layers were washed with brine (20 mL) and dried over Na_2SO_4. Removal of the solvent gave a residue, which was purified by chromatography (silica gel, EtOAc) to give 342 mg (84%) of product **15** as a white solid. It had mp 69-71 ^oC (EtOAc), $[\alpha]_D^{20} = +8.8$ (c 0.04, $CHCl_3$). IR: *v* 3440, 1714, 1092, 647 cm^{-1}; ^1H NMR: δ 3.97-3.93 (m, 1H), 3.74-3.68 (m, 1H), 3.14-3.05 (m, 1H), 3.00-2.96 (m, 1H), 2.83-2.70 (m, 2H), 2.46-2.33 (m, 3H), 2.17-2.07 (m, 2H), 1.98-1.95 (m, 1H), 1.83-1.56 (m, 3H), 1.40-1.20 (m, 2H); ^{13}C NMR: δ 211.9, 63.0, 58.3, 56.6, 55.69, 55.67, 41.7, 31.1, 25.3, 23.3; MS *m/z* (%): 183 (M^+, 28.76), 28 (100). Anal. Calcd for $C_{10}H_{17}NO_2$: C, 65.54; H, 9.35; N, 7.64. Found: C, 65.81; H, 9.42; N, 7.56.



16

(1*S*,9a*R*)-Octahydro-1-hydroxymethyl-spiro[1,3-dithiolane-2,2'-[2*H*]quinolizine] (16): To a solution of compound **15** (293 mg, 1.60 mmol) in dry CH_2Cl_2 was added a

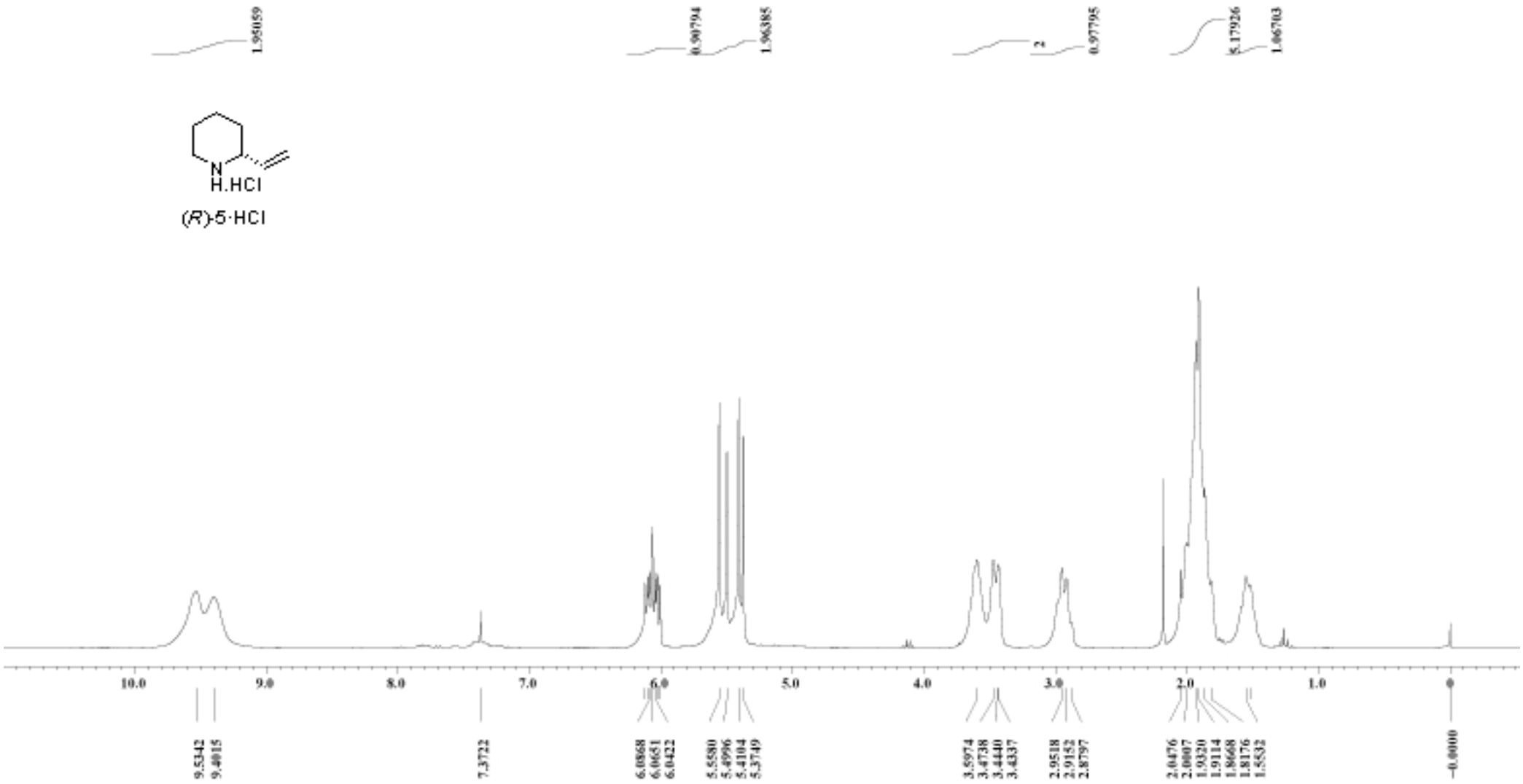
mixture of ethane-1,2-dithiol (1.23 mL, 14.7 mmol) in $BF_3 \cdot Et_2O$ (0.46 mL, 3.68 mmol). Two hours later, the reaction was quenched by aqueous NaOH (2.0 M, 3 mL). The resultant mixture was extracted by CH_2Cl_2 (3 x 20 mL) and combined organic layers were washed with H_2O (20 mL) and brine (20 mL) and dried over Na_2SO_4. Removal of the solvent gave a residue, which was purified by chromatography (silica gel, EtOAc) to give 344 mg (83%) of product **16** as a white solid. It had mp 128-130 ^oC (EtOAc), $[\alpha]_D^{20}$ = -13.0 (c 0.1, $CHCl_3$). IR: *v* 3452, 1641, 1048, 1025 cm^{-1}; ^1H NMR: δ 4.21-4.17 (m, 1H), 3.90-3.86 (m, 1H), 3.39-3.23 (m, 4H), 2.94-2.77 (m, 3H), 2.47-2.24 (m, 2H), 2.16-1.95 (m, 4H), 1.82-1.73 (m, 2H), 1.65-1.52 (m, 2H), 1.38-1.14 (m, 2H); ^{13}C NMR: δ 71.8, 63.7, 61.3, 56.3, 55.2, 52.7, 44.9, 39.3, 38.7, 29.9, 25.3, 24.2; MS *m/z* (%): 259 (M^+, 28.33), 97 (100). Anal. Calcd for $C_{12}H_{21}NOS_2$: C, 55.56; H, 8.16; N, 5.40. Found: C, 55.51; H, 8.17; N, 5.44.



2
(+)-Epilupinine

Preparation of (+)-Epilupinine (2): A suspension of compound **16** (260 mg, 1.00 mmol), Raney nickel (secondary grad, 900 mg, 15.3 mmol) in anhydrous EtOH (20 mL) was refluxed for 1.5 h. After the reaction was cooled to room temperature, the catalyst was filtrated off. Removal of the solvent gave a residue, which was purified by chromatography (silica gel, 25% MeOH in EtOAc) to give 161 mg (95%) of product **2** as a white solid. It had mp 77-79 ^oC (lit.[7b,7g] 78-79 ^oC), $[\alpha]_D^{20}$ = +31.8 (c 0.60, EtOH) [lit.[7b] $[\alpha]_D^{20}$= +32.6 (c 0.72, EtOH), lit.[7g] $[\alpha]_D^{22}$= +31.2 (c 0.86, EtOH)]. IR: *v* 3183,

2929, 2860, 1064 cm^{-1}; ^{1}H NMR: δ 3.62-3.56 (m, 1H), 3.50-3.45 (m, 1H), 2.77-2.69 (m, 2H), 2.33 (s, br, 1H), 2.00-1.50 (m, 9H), 1.38-1.28 (m, 1H), 1.26-1.11 (m, 4H); ^{13}C NMR: δ 64.4, 64.3, 56.8, 56.6, 43.9, 29.7, 28.2, 25.5, 24.9, 24.5; MS *m/z* (%): 169 (M^{+}, 42.96), 83 (100). Anal. Calcd for $C_{10}H_{19}NO$: C, 70.96; H, 11.31; N, 8.28. Found: C, 70.91; H, 11.34; N, 8.23.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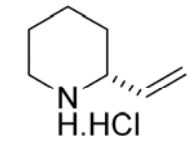
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H.HCl

(*R*)-**5**·HCl

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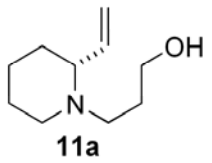
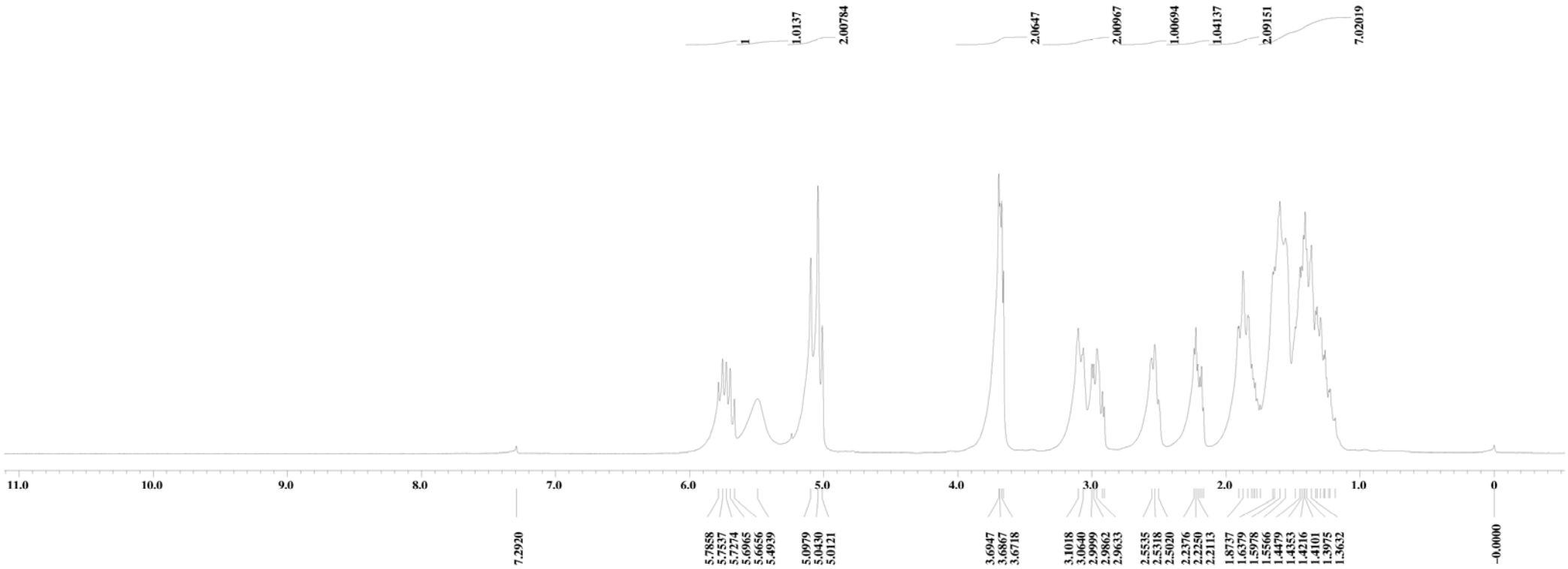
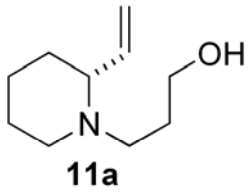


11a



X : parts per Million : 1H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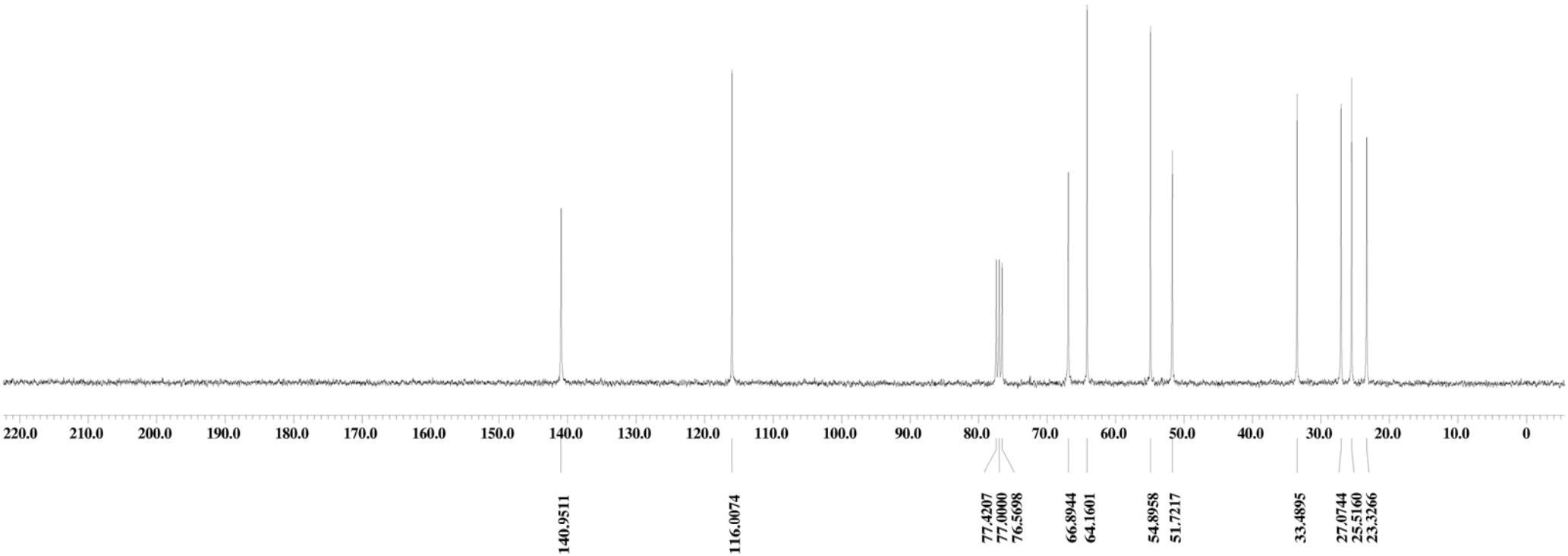
N

11a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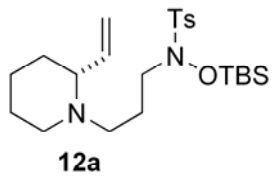
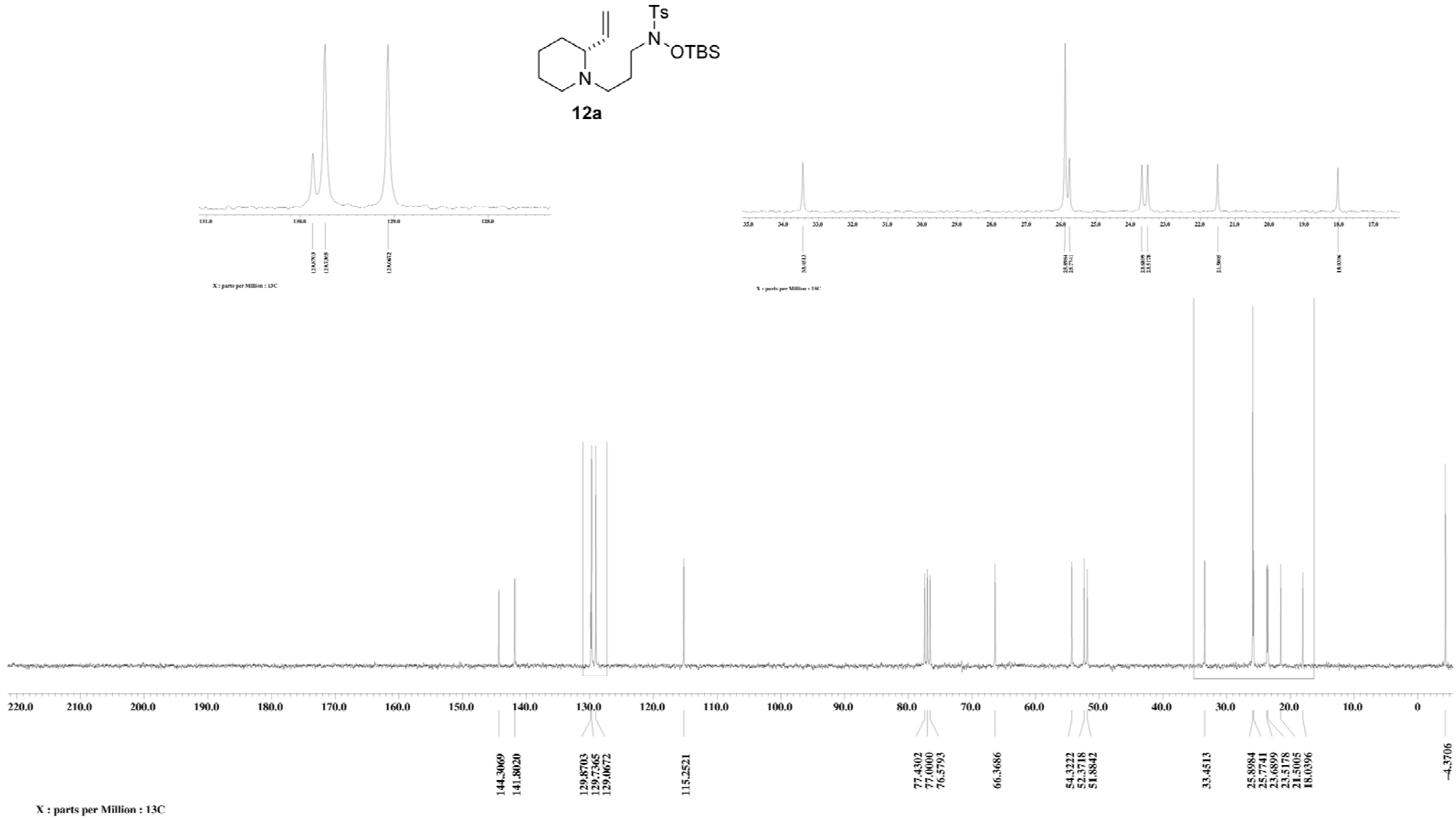


12a

X : parts per Million : 1H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X : parts per Million : 13C
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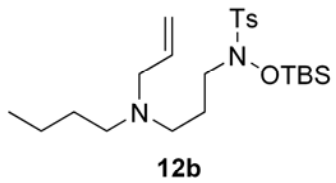

12b

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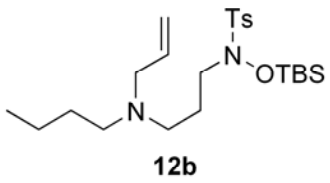
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OTBS
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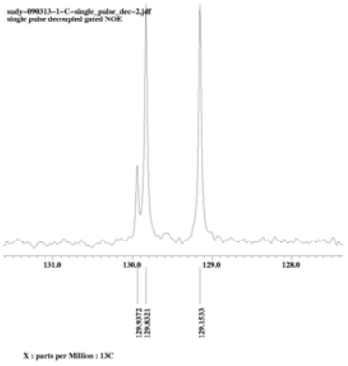
12b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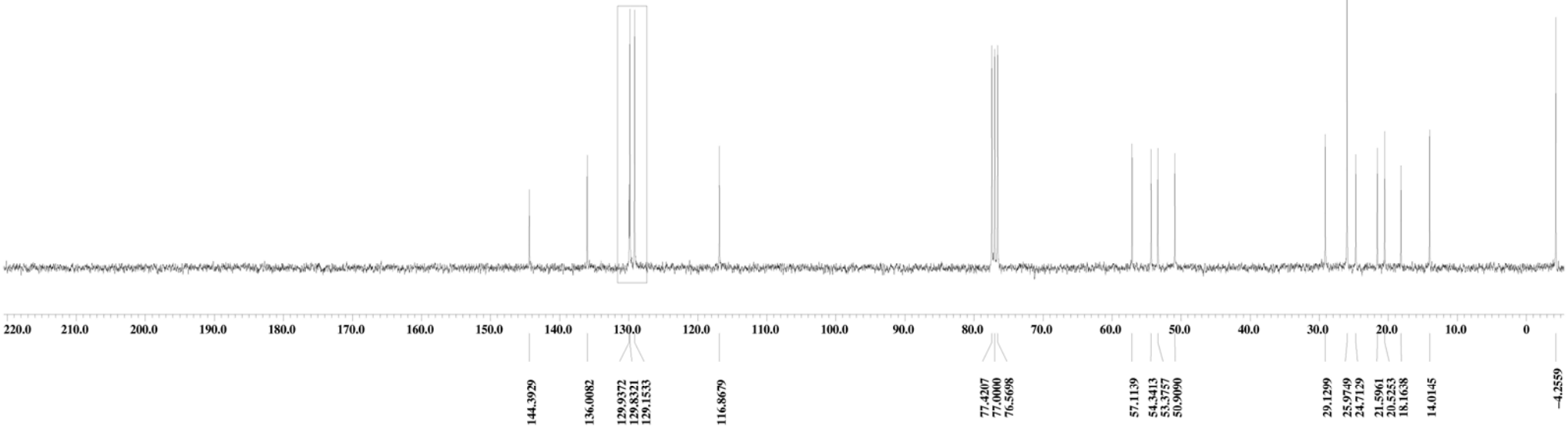
X : parts per Million : 13C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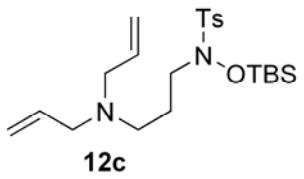

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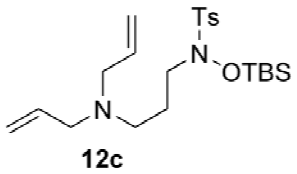
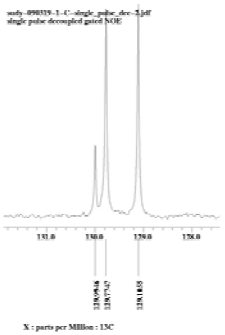

Ts
N
OTBS
N

12c


single pulse decoupled gated NOE
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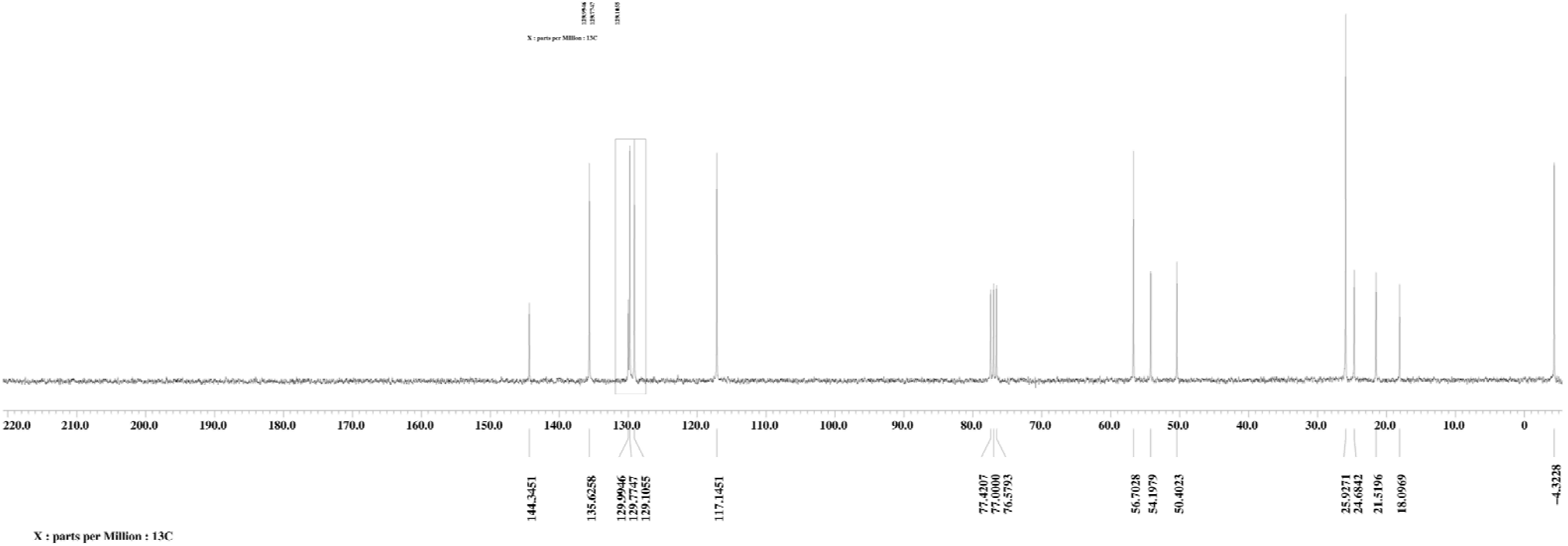
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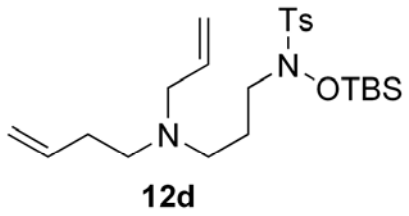
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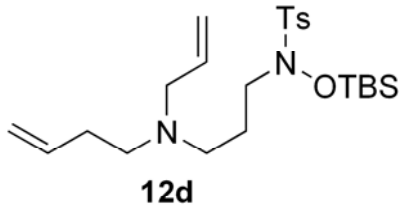
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OTBS
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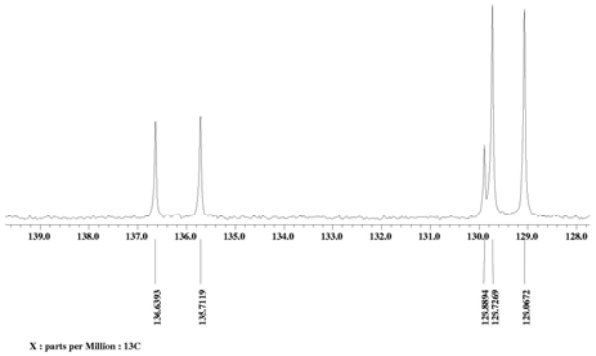
12d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X : parts per Million : 13C

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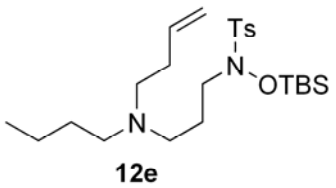

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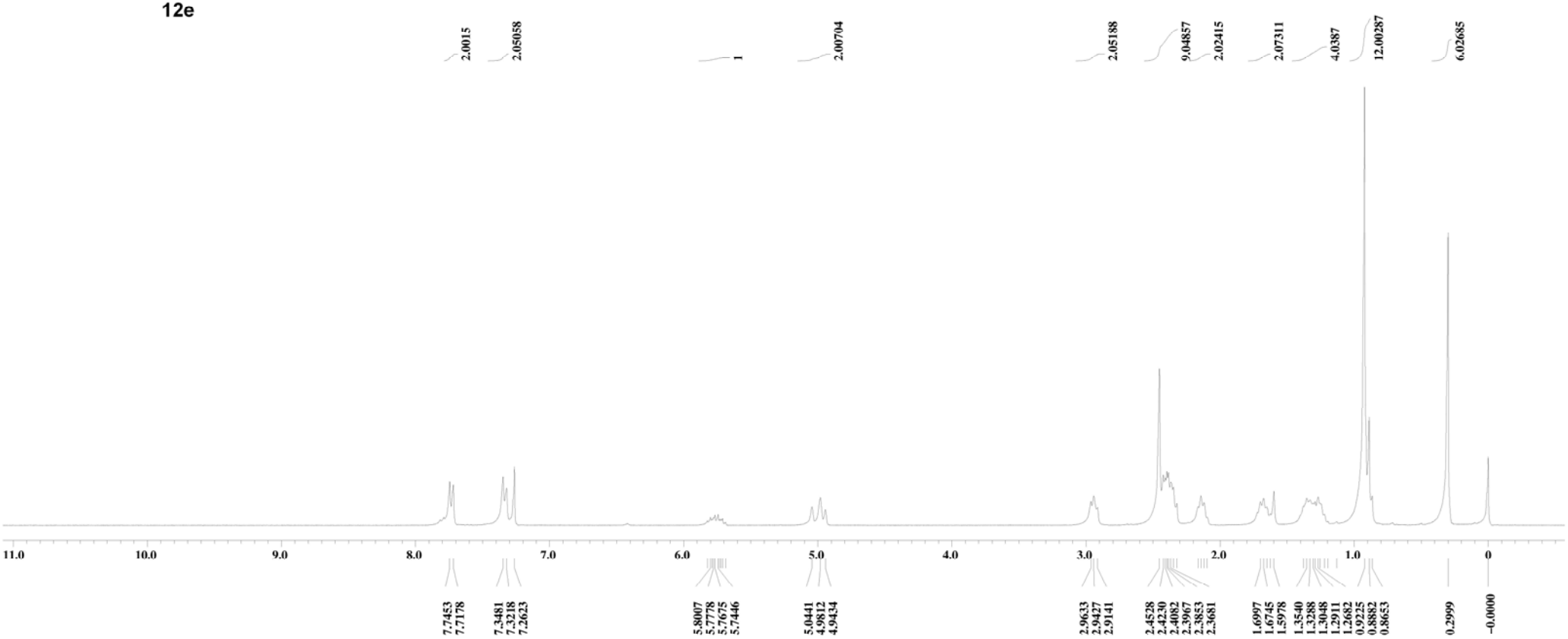

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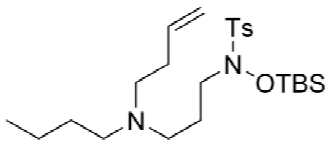
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OTBS
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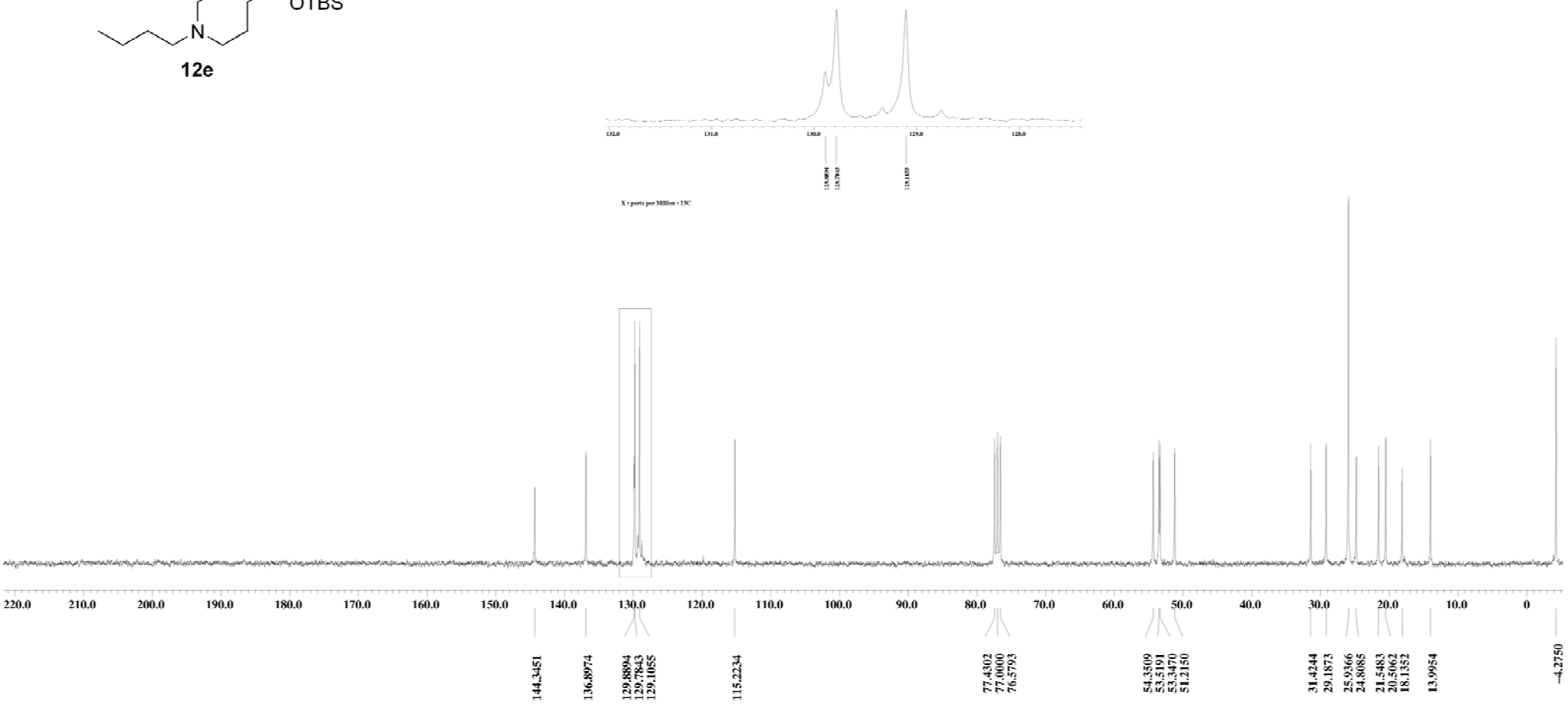
12e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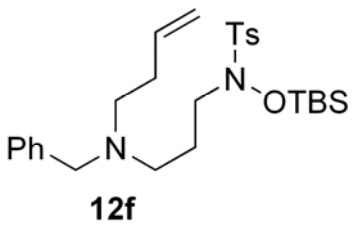
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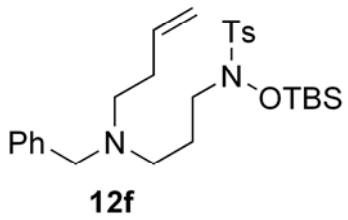
Ts
N
OTBS
Ph
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12f

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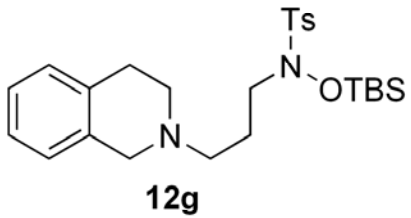
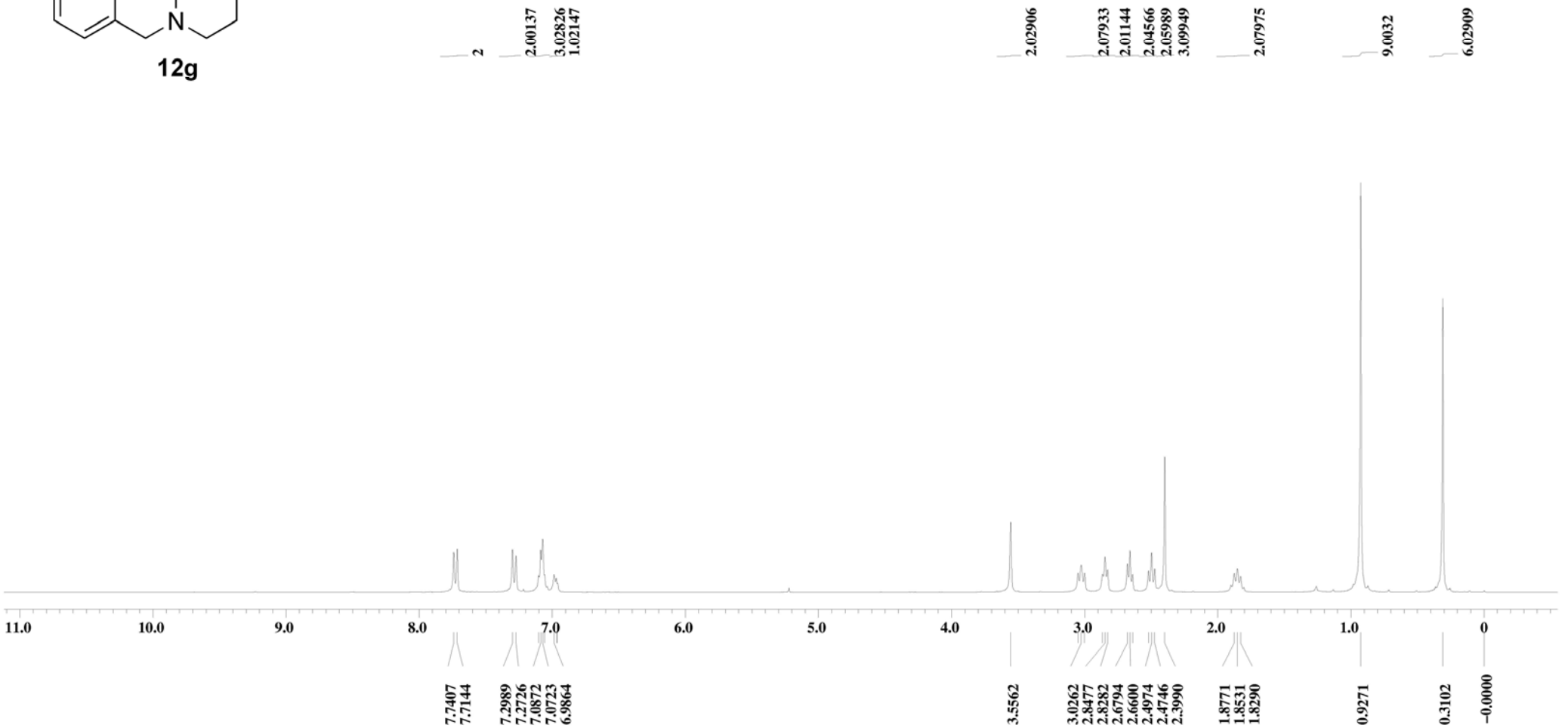
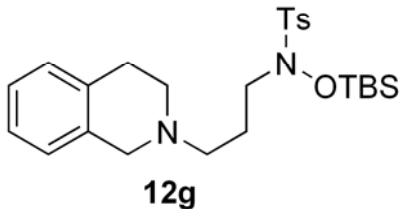
77.4302
77.0000
76.5793
58.3281
54.2552
52.9646
50.8899
31.3575
25.9558
24.7894
21.5674
18.1256
–4.2654

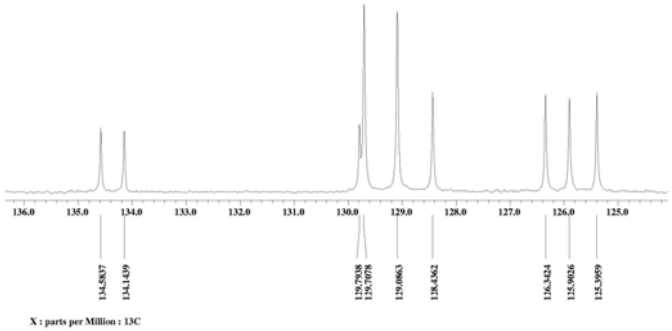

Ts
N
OTBS
N

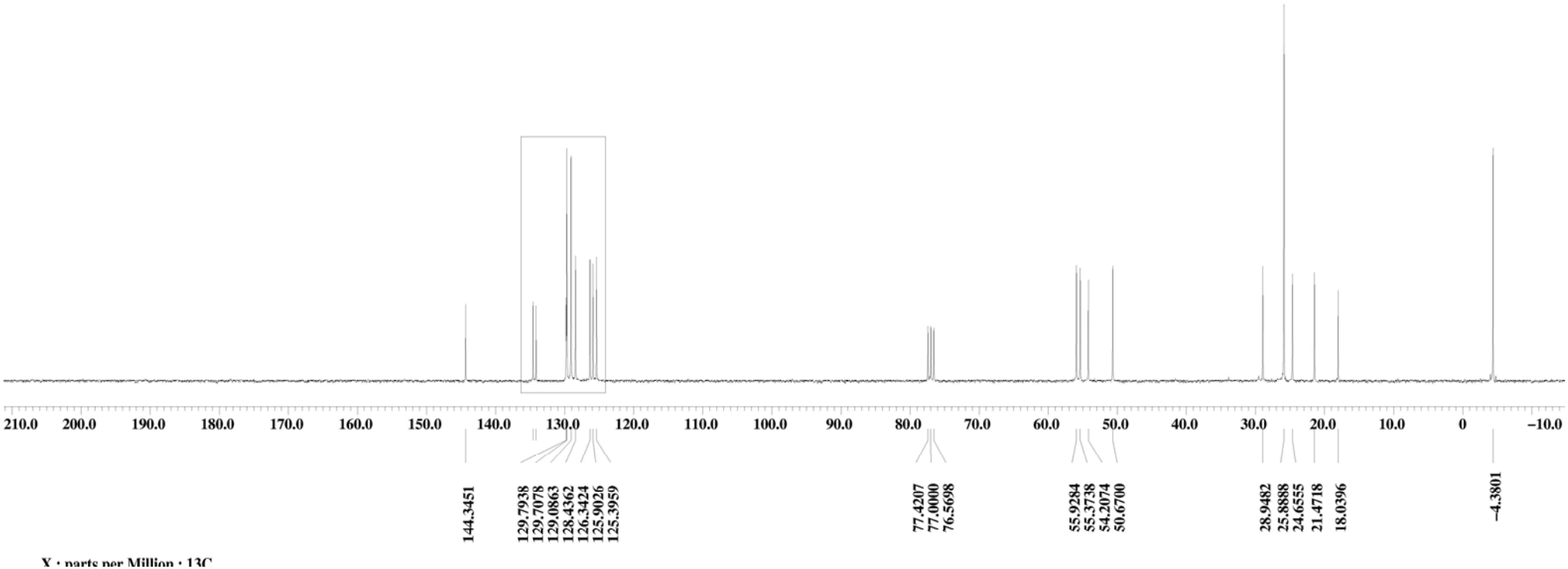
12g

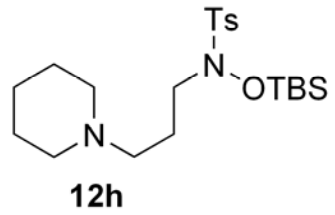

2
2.00137
3.02826
1.02147
2.02906
2.07933
2.01144
2.04566
2.05989
3.09949
2.07975
9.0032
6.02909
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.7407
7.7144
7.2989
7.2736
7.0872
7.0723
6.9864
3.5562
3.0262
2.8477
2.8282
2.6794
2.6600
2.4974
2.4746
2.3990
1.8771
1.8531
1.8290
0.9271
0.3102
−0.0000


Ts
N
OTBS
N

12g


136.0
135.0
134.0
133.0
132.0
131.0
130.0
129.0
128.0
127.0
126.0
125.0
134.5837
134.1439
129.7938
129.7078
129.0863
128.4362
126.3424
125.9026
125.3959
X : parts per Million : 13C


210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
144.3451
129.7938
129.7078
129.0863
128.4362
126.3424
125.9026
125.3959
77.4207
77.0000
76.5698
55.9284
55.3738
54.2074
50.6700
28.9482
25.8888
24.6555
21.4718
18.0396
−4.3801
X : parts per Million : 13C


Ts
N
OTBS
N

12h

2
2.00265
2.07734
3.00224
6.01628
2.08401
4.0938
2.04742
9.02094
6.03997
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.7499
7.7224
7.3493
7.3229
7.2680
2.9736
2.9484
2.9232
2.4505
2.3040
2.2926
2.2685
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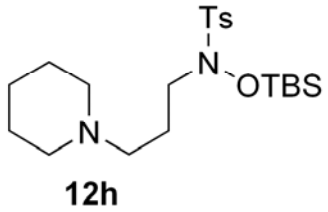
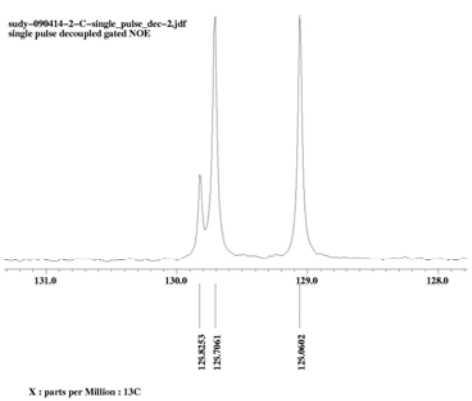
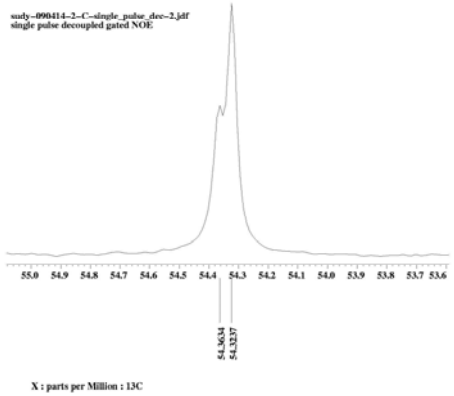
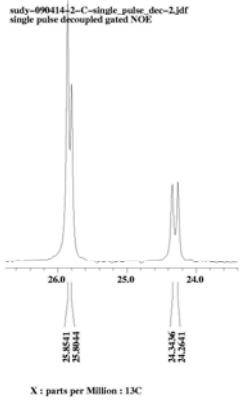
1.5600
1.5417
1.5246
1.4193
1.4032
0.9214
0.2953
−0.0000

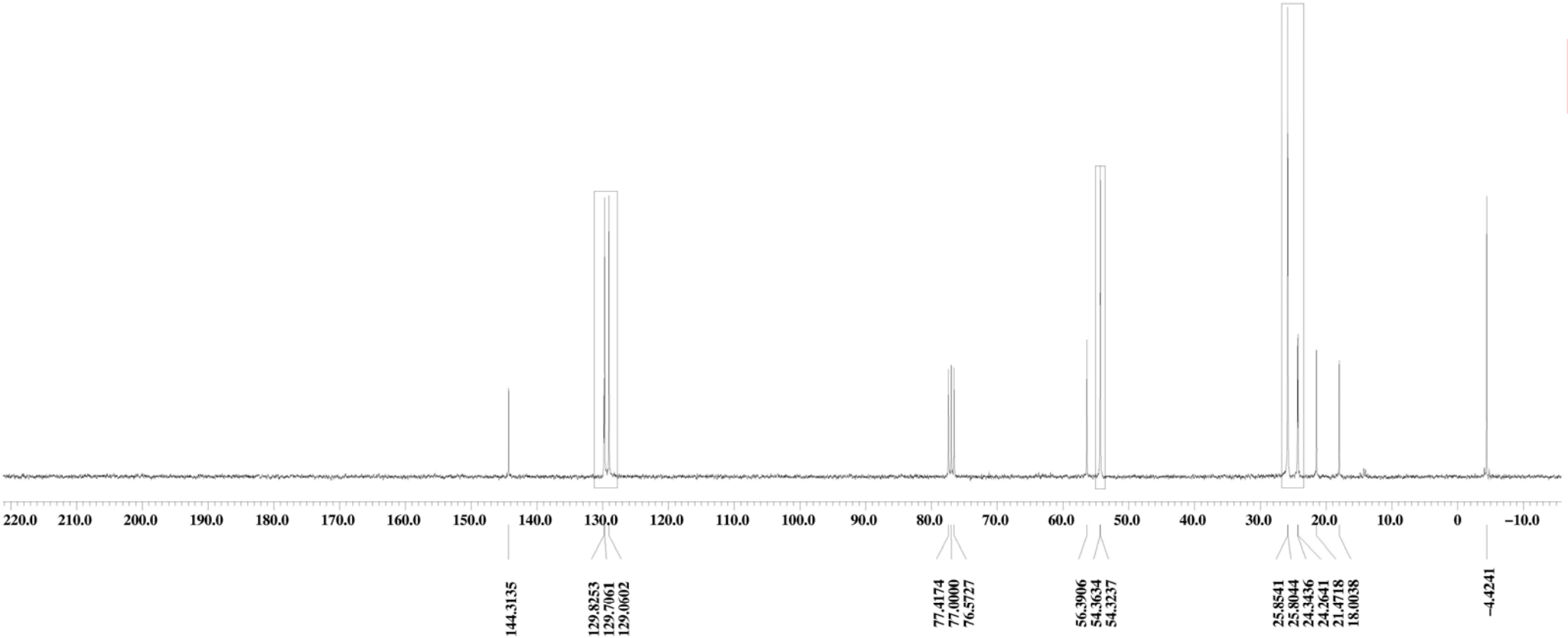

Ts
N
OTBS
N

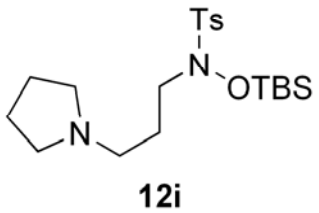
12h


sudy--090414--2--C--single_pulse_dec--2.jdf
single pulse decoupled gated NOE
131.0
130.0
129.0
128.0
125.8253
125.7061
125.0602
X : parts per Million : 13C

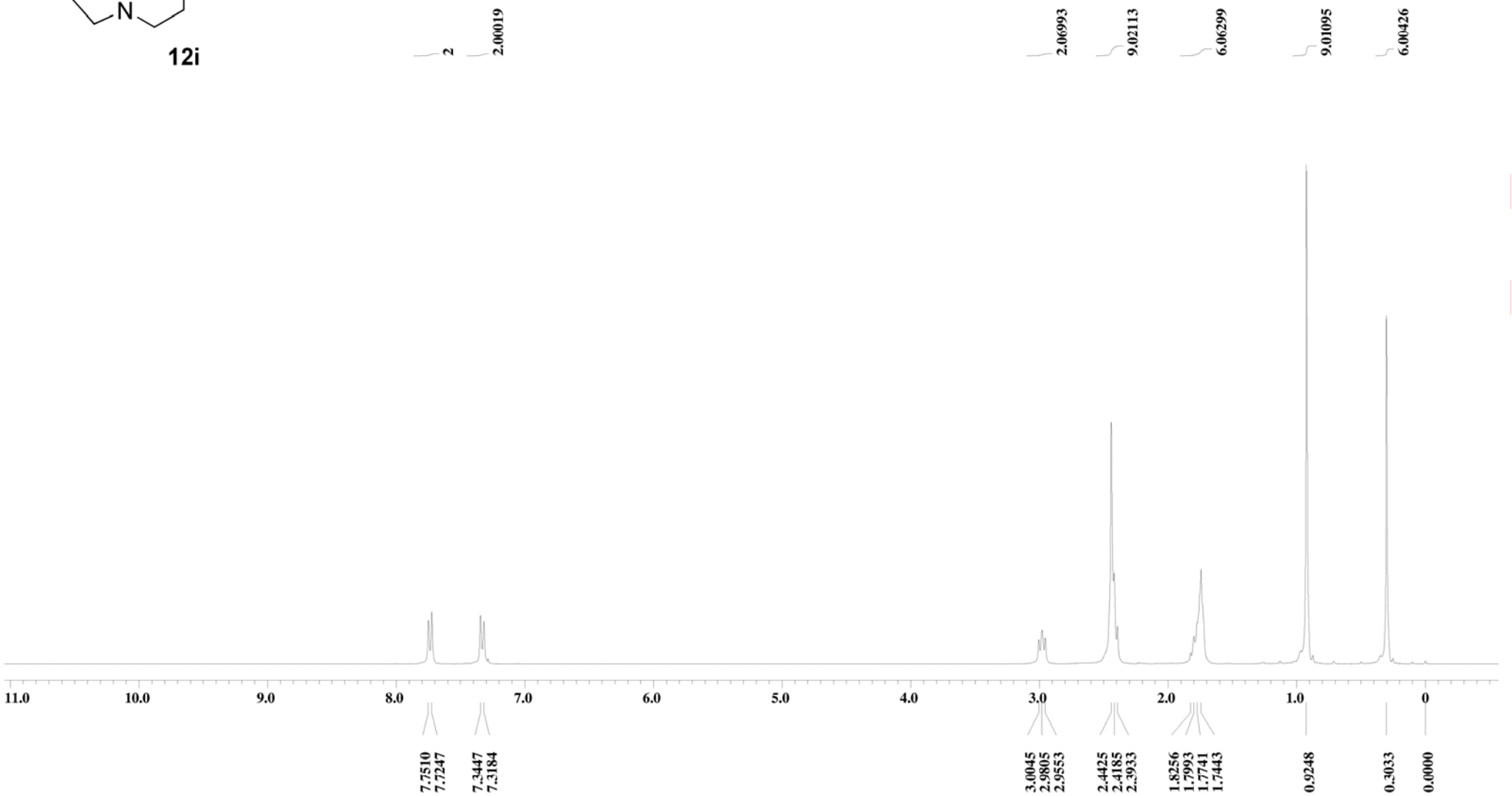

sudy--090414--2--C--single_pulse_dec--2.jdf
single pulse decoupled gated NOE
55.0 54.9 54.8 54.7 54.6 54.5 54.4 54.3 54.2 54.1 54.0 53.9 53.8 53.7 53.6
54.3634
54.3237
X : parts per Million : 13C

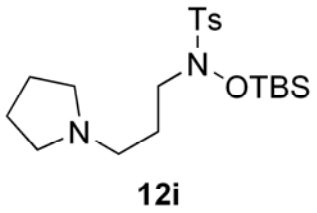

sudy--090414--2--C--single_pulse_dec--2.jdf
single pulse decoupled gated NOE
26.0
25.0
24.0
25.8541
25.8044
24.3436
24.2641
X : parts per Million : 13C


220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 −10.0
144.3135
129.8253
129.7061
129.0602
77.4174
77.0000
76.5727
56.3906
54.3634
54.3237
25.8541
25.8044
24.3436
24.2641
21.4718
18.0038
−4.4241

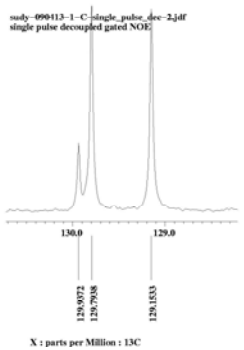

Ts
N
OTBS
N

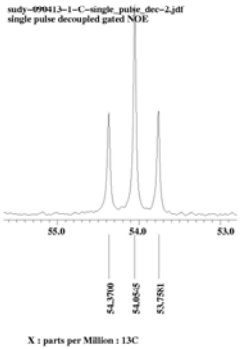
12i

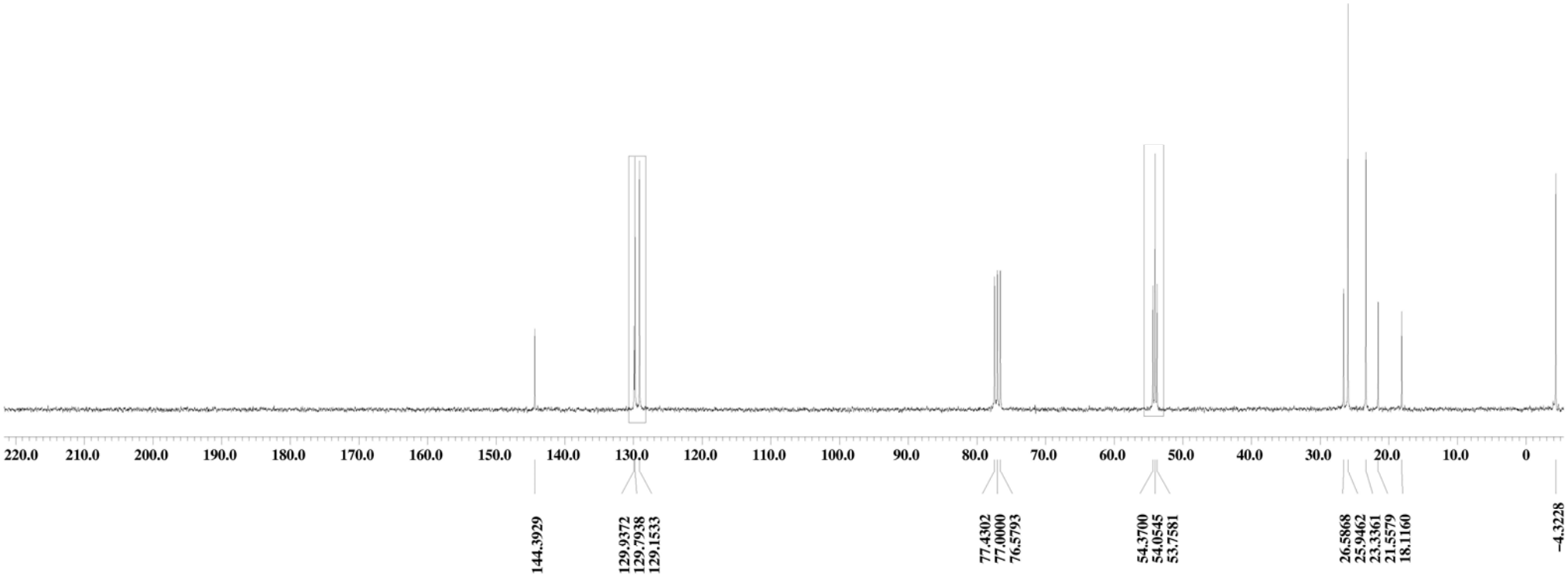

2
2.00019
2.06993
9.02113
6.06299
9.01095
6.00426
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.7510
7.7247
7.3447
7.3184
3.0045
2.9805
2.9553
2.4425
2.4185
2.3933
1.8256
1.7993
1.7741
1.7443
0.9248
0.3033
0.0000

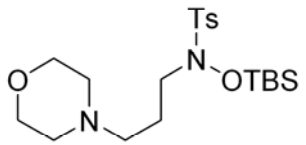

Ts
N
N
OTBS

12i


sudy-090413-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
130.0
129.0
129.9372
129.7938
129.1533
X : parts per Million : 13C

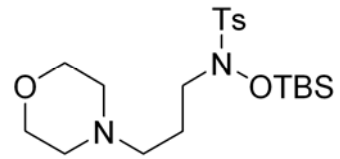

sudy-090413-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
55.0
54.0
53.0
54.3700
54.0545
53.7581
X : parts per Million : 13C


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
144.3929
129.9372
129.7938
129.1533
77.4302
77.0000
76.5793
54.3700
54.0545
53.7581
26.5868
25.9462
23.3361
21.5579
18.1160
-4.3228

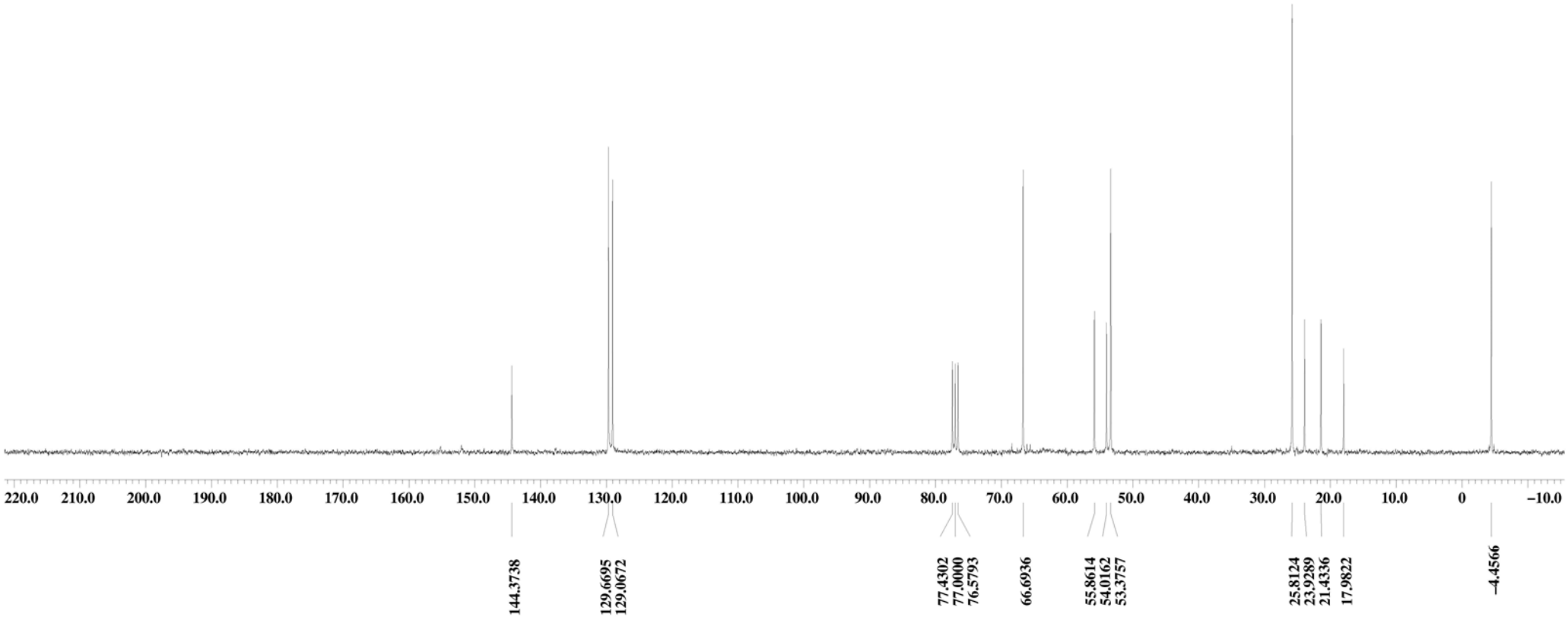


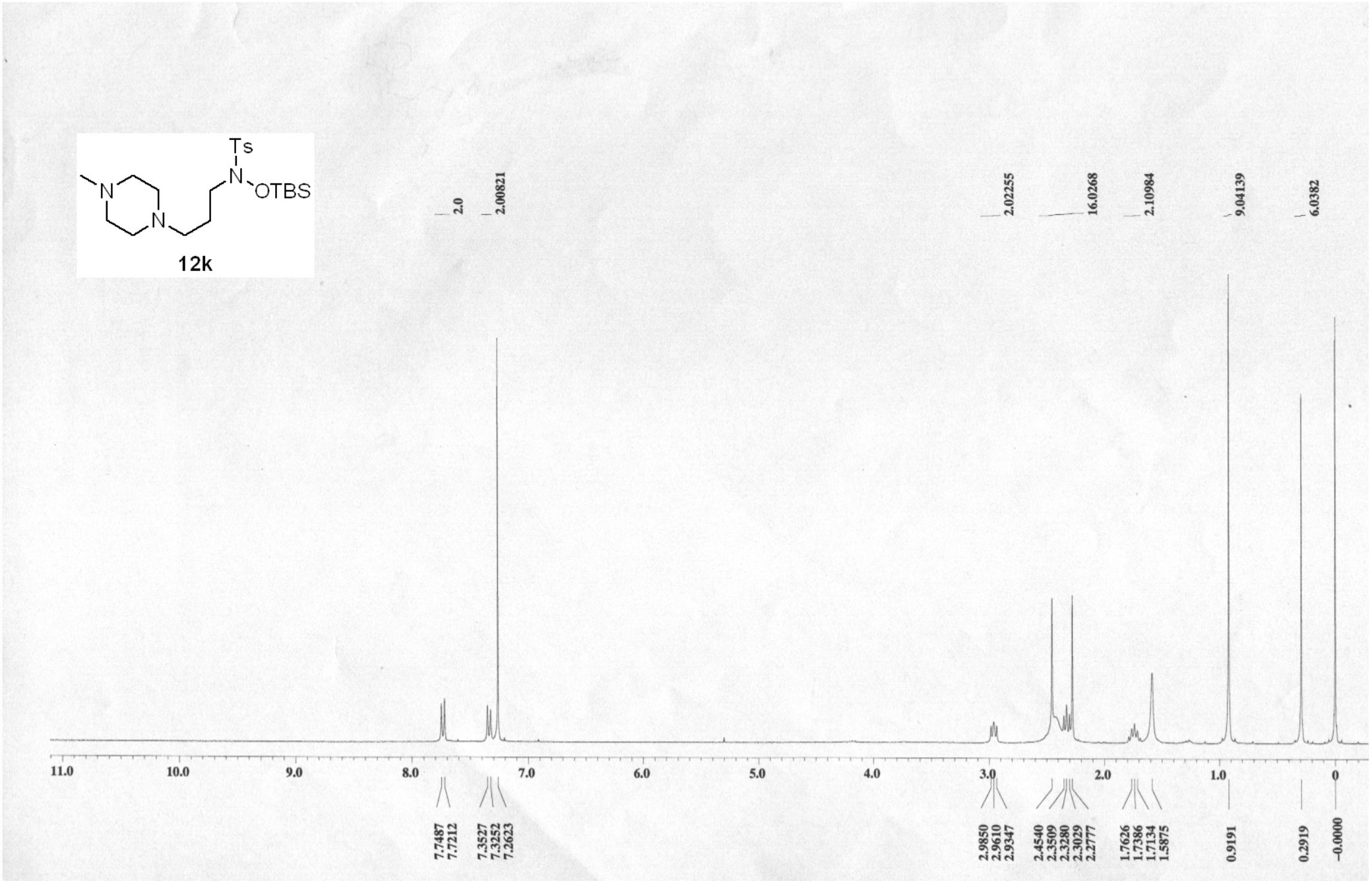
12j

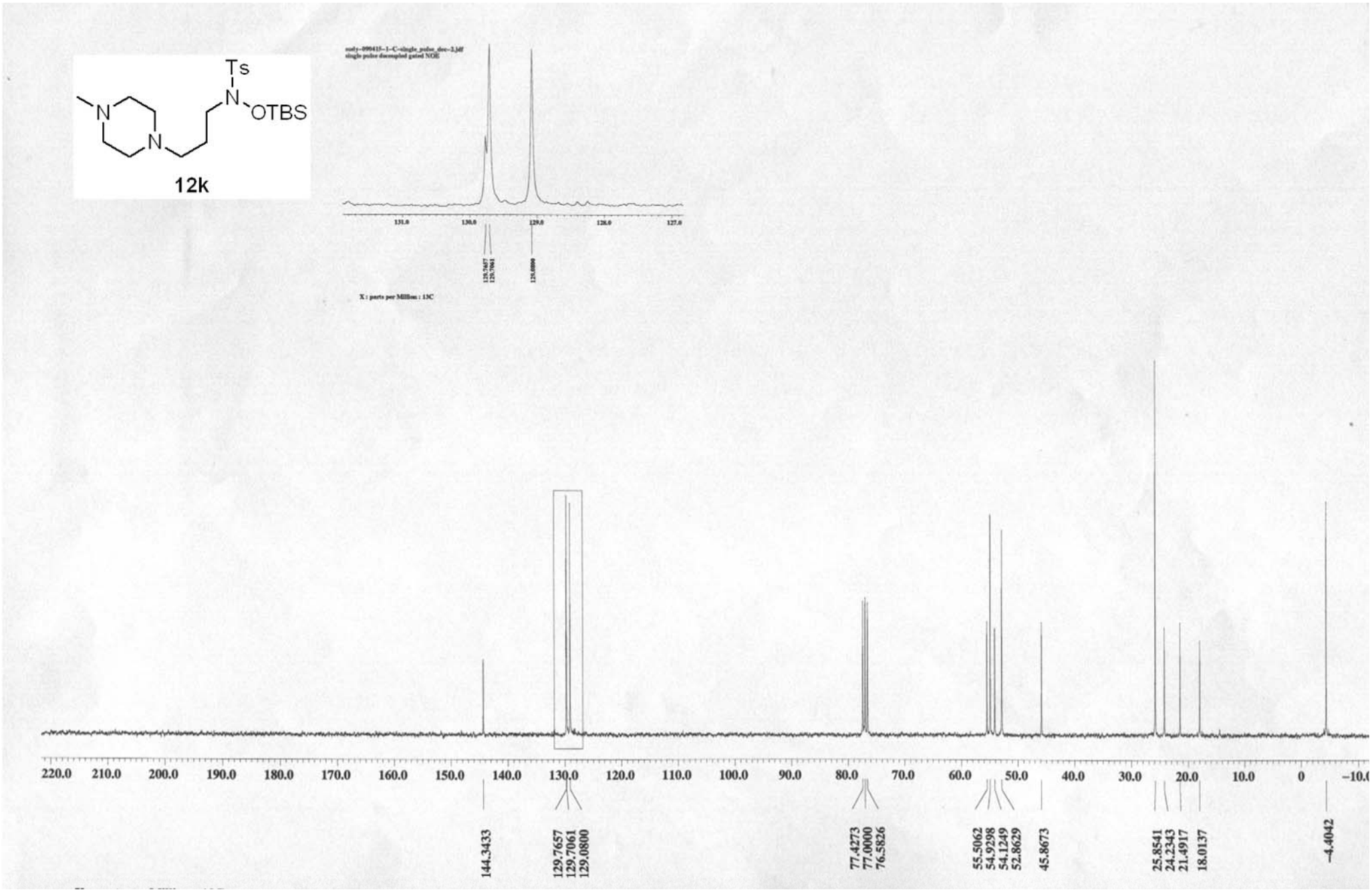
X : parts per Million : 1H


Ts
N
OTBS
O
N

12j


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
−10.0
144.3738
129.6695
129.0672
77.4302
77.0000
76.5793
66.6936
55.8614
54.0162
53.3757
25.8124
23.9289
21.4336
17.9822
−4.4566


Ts
N
N
OTBS
12k
2.0
2.00821
2.02255
16.0268
2.10984
9.04139
6.0382
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.7487
7.7212
7.3527
7.3252
7.2623
2.9850
2.9610
2.9347
2.4540
2.3509
2.3280
2.3029
2.2777
1.7626
1.7386
1.7134
1.5875
0.9191
0.2919
−0.0000


Ts
N
N
OTBS
N
12k
131.0
130.0
129.0
128.0
127.0
X : parts per Million : 13C
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
144.3433
129.7657
129.7061
129.0800
77.4273
77.0000
76.5826
55.5062
54.9298
54.1249
52.8629
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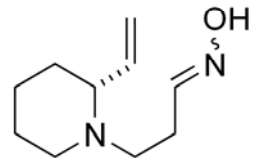
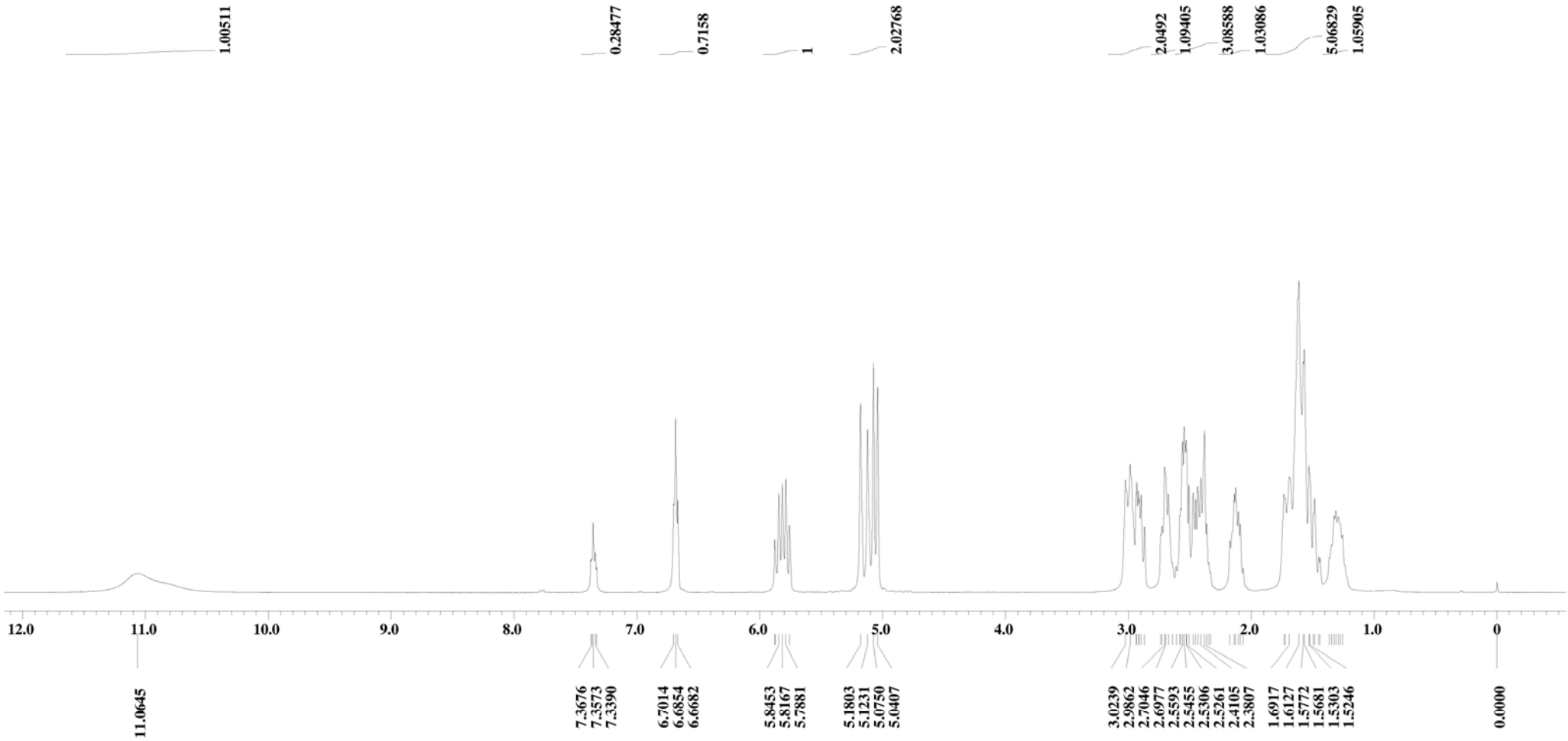
24.2343
21.4917
18.0137
-4.4042


OH
N
N

13a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0.28477
0.7158
1
2.02768
2.0492
1.09405
3.08588
1.03086
5.06829
1.05905
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.0645
7.3676
7.3573
7.3390
6.7014
6.6854
6.6682
5.8453
5.8167
5.7881
5.1803
5.1231
5.0750
5.0407
3.0239
2.9862
2.7046
2.6977
2.5593
2.5455
2.5306
2.5261
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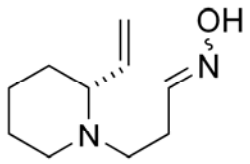
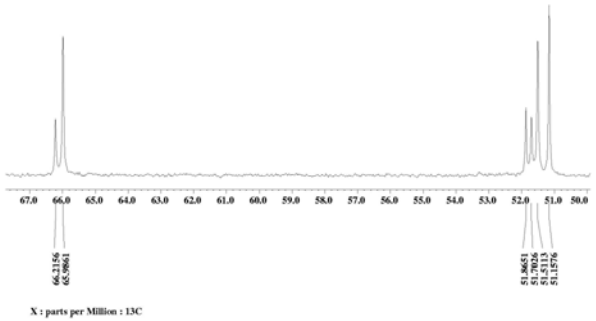
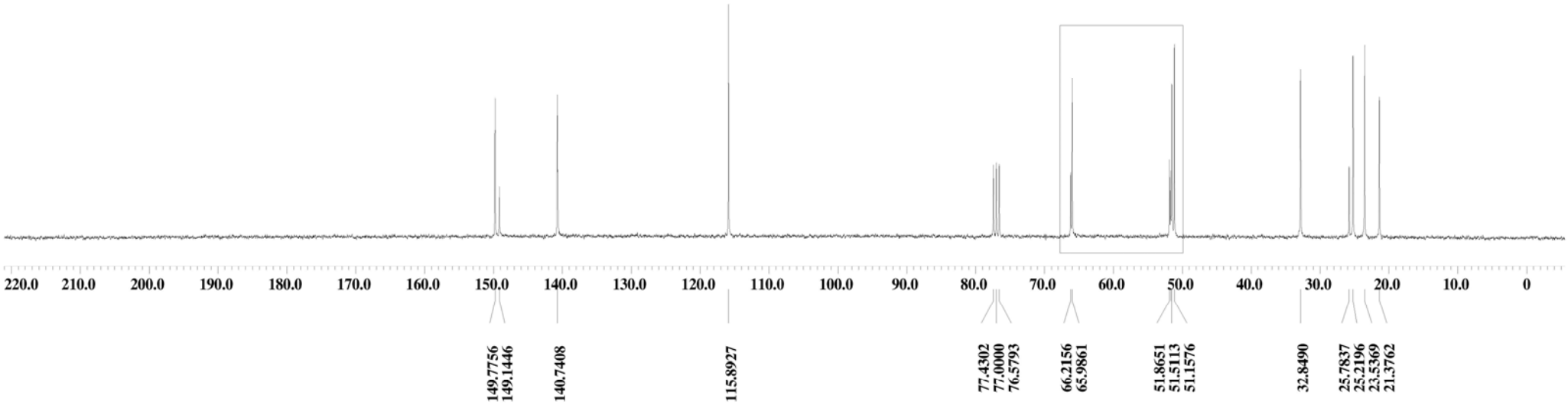
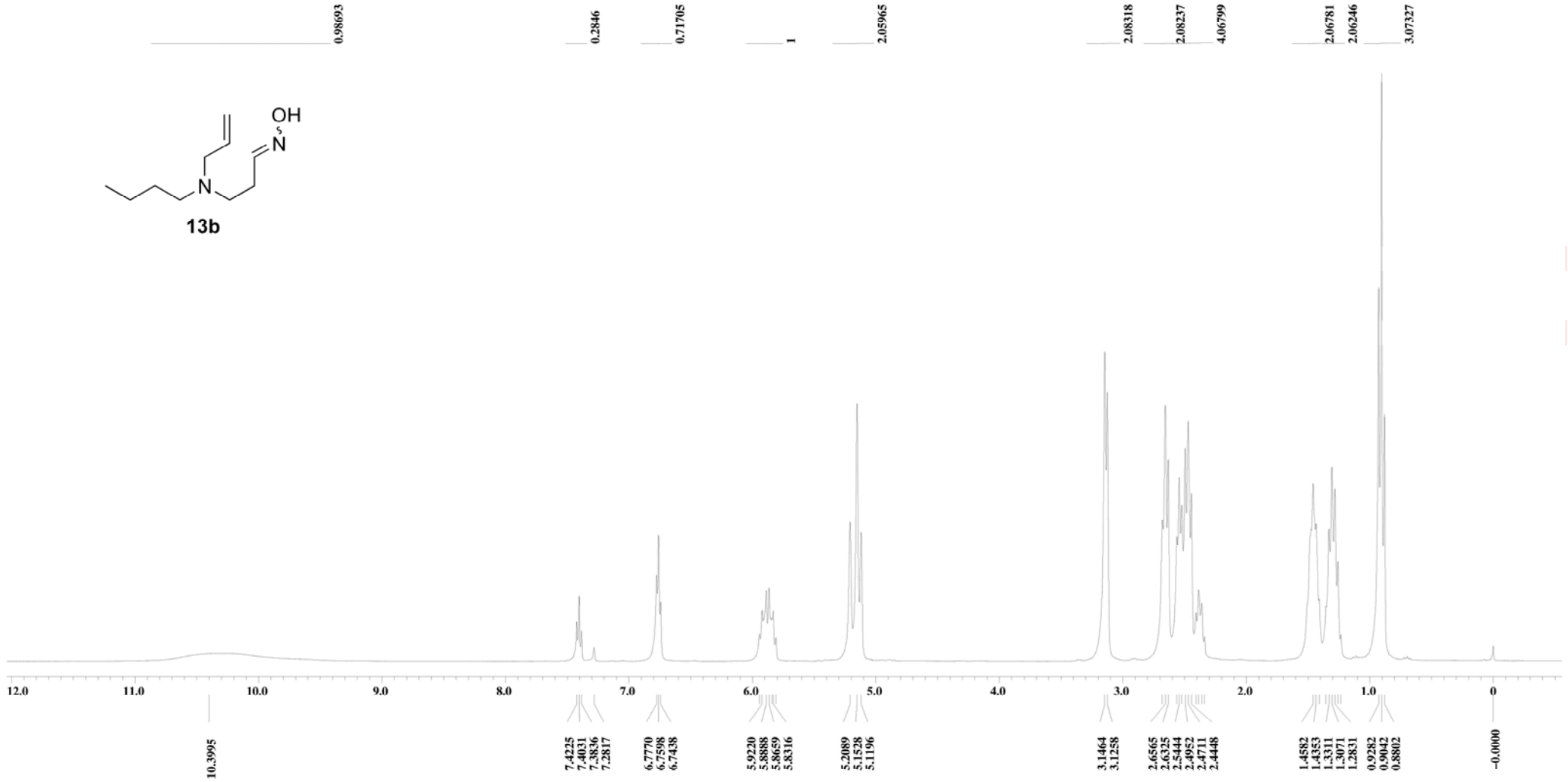
2.3807
1.6917
1.6127
1.5772
1.5681
1.5303
1.5246
0.0000

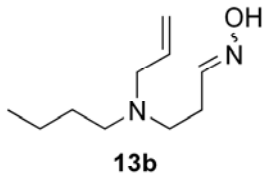

OH
N
N

13a

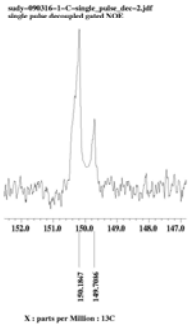

67.0 66.0 65.0 64.0 63.0 62.0 61.0 60.0 59.0 58.0 57.0 56.0 55.0 54.0 53.0 52.0 51.0 50.0
66.2156
65.9861
51.8651
51.7026
51.5113
51.1576
X : parts per Million : 13C

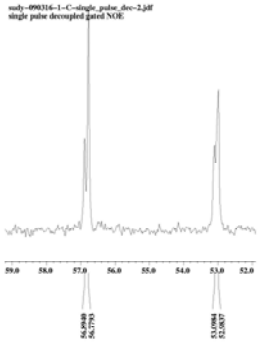

220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
149.7756
149.1446
140.7408
115.8927
77.4302
77.0000
76.5793
66.2156
65.9861
51.8651
51.5113
51.1576
32.8490
25.7837
25.2196
23.5369
21.3762

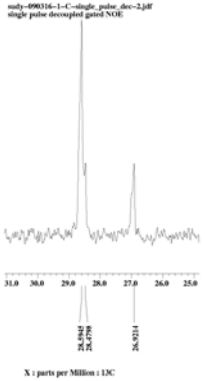

0.98693
0.2846
0.71705
1
2.05965
2.08318
2.08237
4.06799
2.06781
2.06246
3.07327
OH
N
N
13b
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.3995
7.4225
7.4031
7.3836
7.2817
6.7770
6.7598
6.7438
5.9220
5.8888
5.8659
5.8316
5.2089
5.1528
5.1196
3.1464
3.1258
2.6565
2.6325
2.5444
2.4952
2.4711
2.4448
1.4582
1.4353
1.3311
1.3071
1.2831
0.9282
0.9042
0.8802
-0.0000

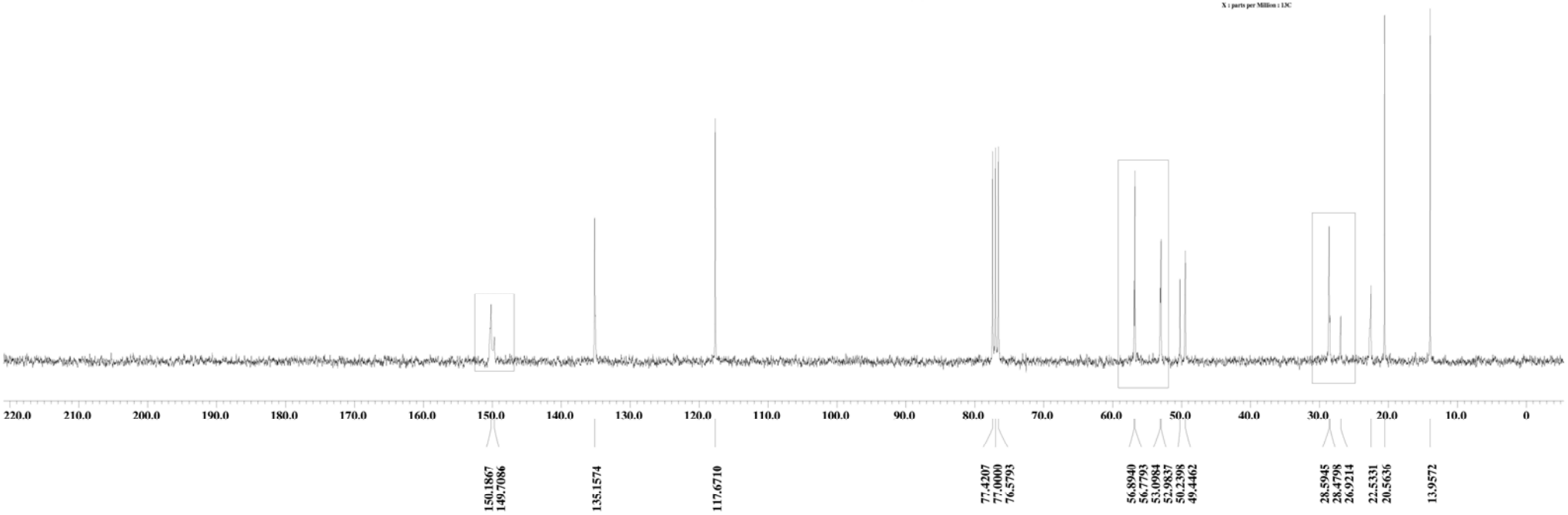

OH
N
N

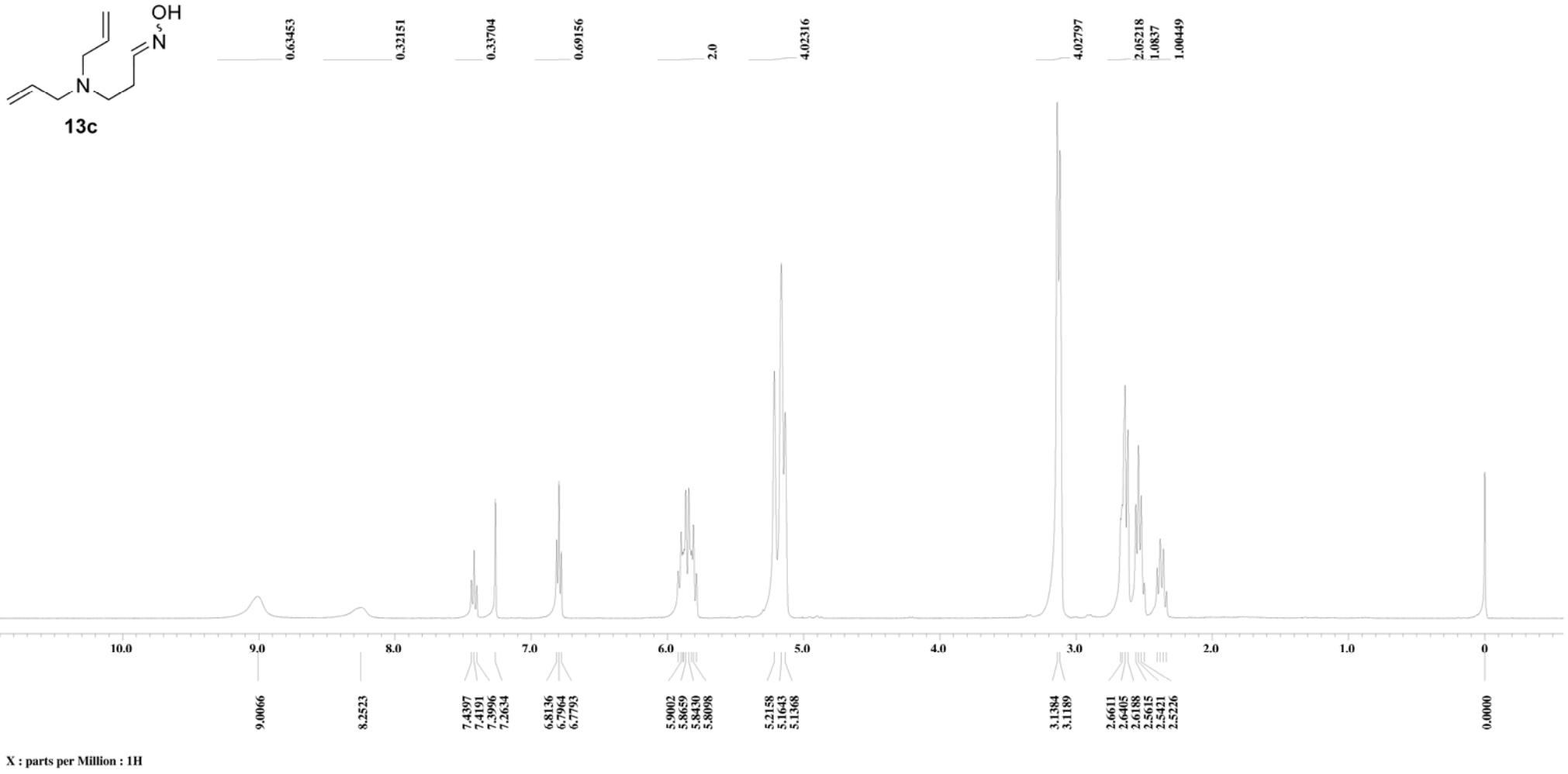
13b

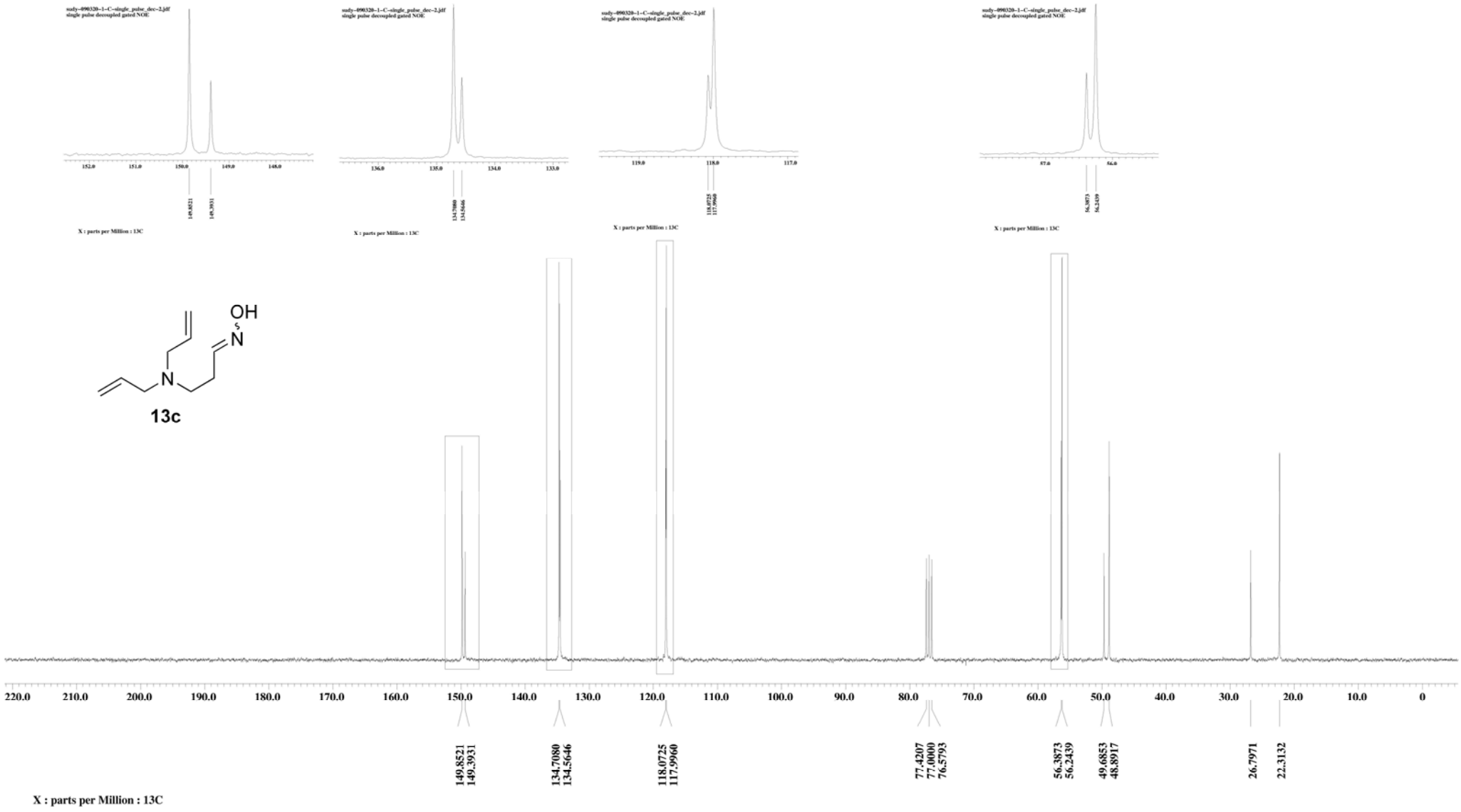

152.0 151.0 150.0 149.0 148.0 147.0
150.1867
149.7086
X : parts per Million : 13C

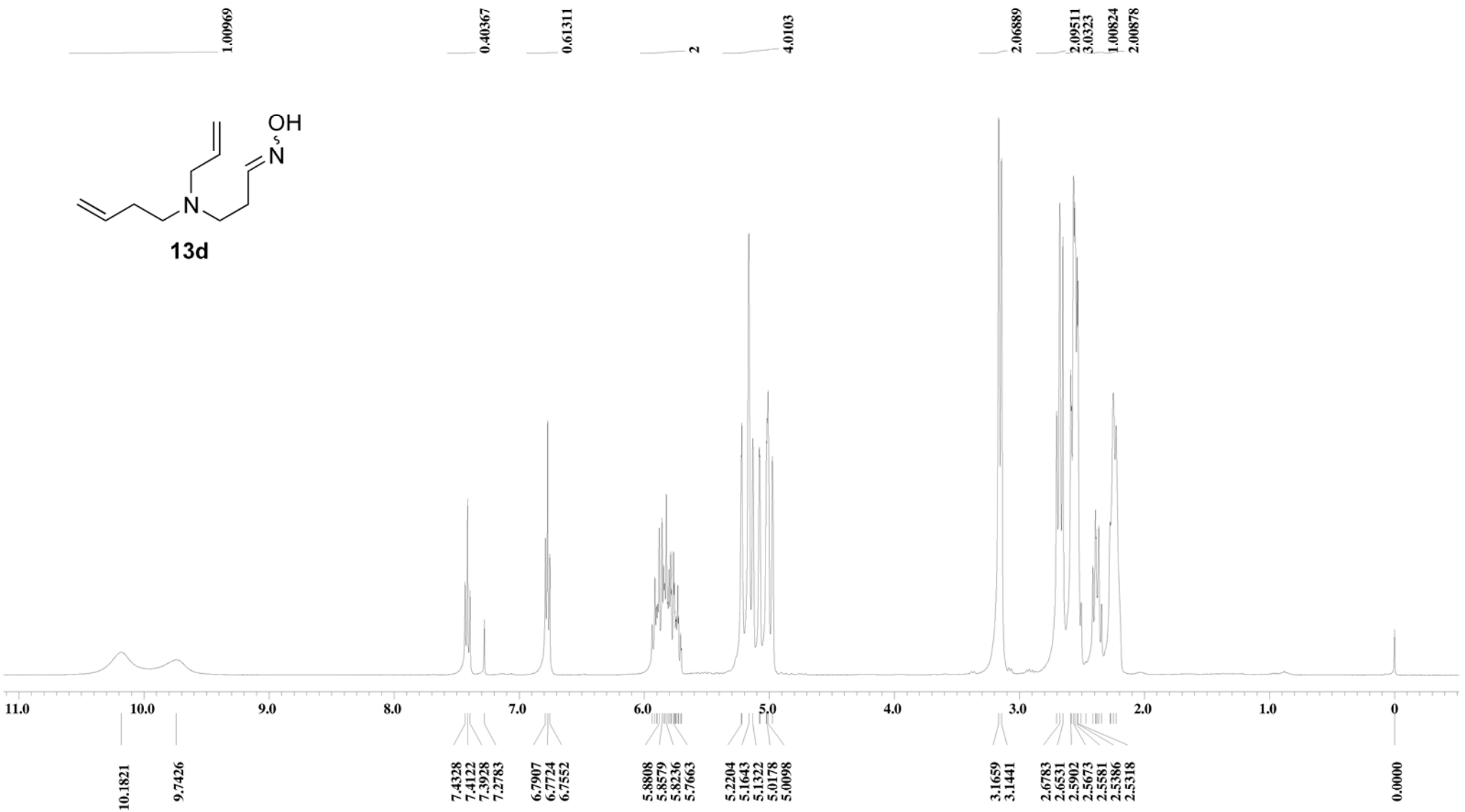

single pulse decoupled gated NOE
59.0 58.0 57.0 56.0 55.0 54.0 53.0 52.0
56.8940
56.7793
53.0984
53.0837
X : parts per Million : 13C

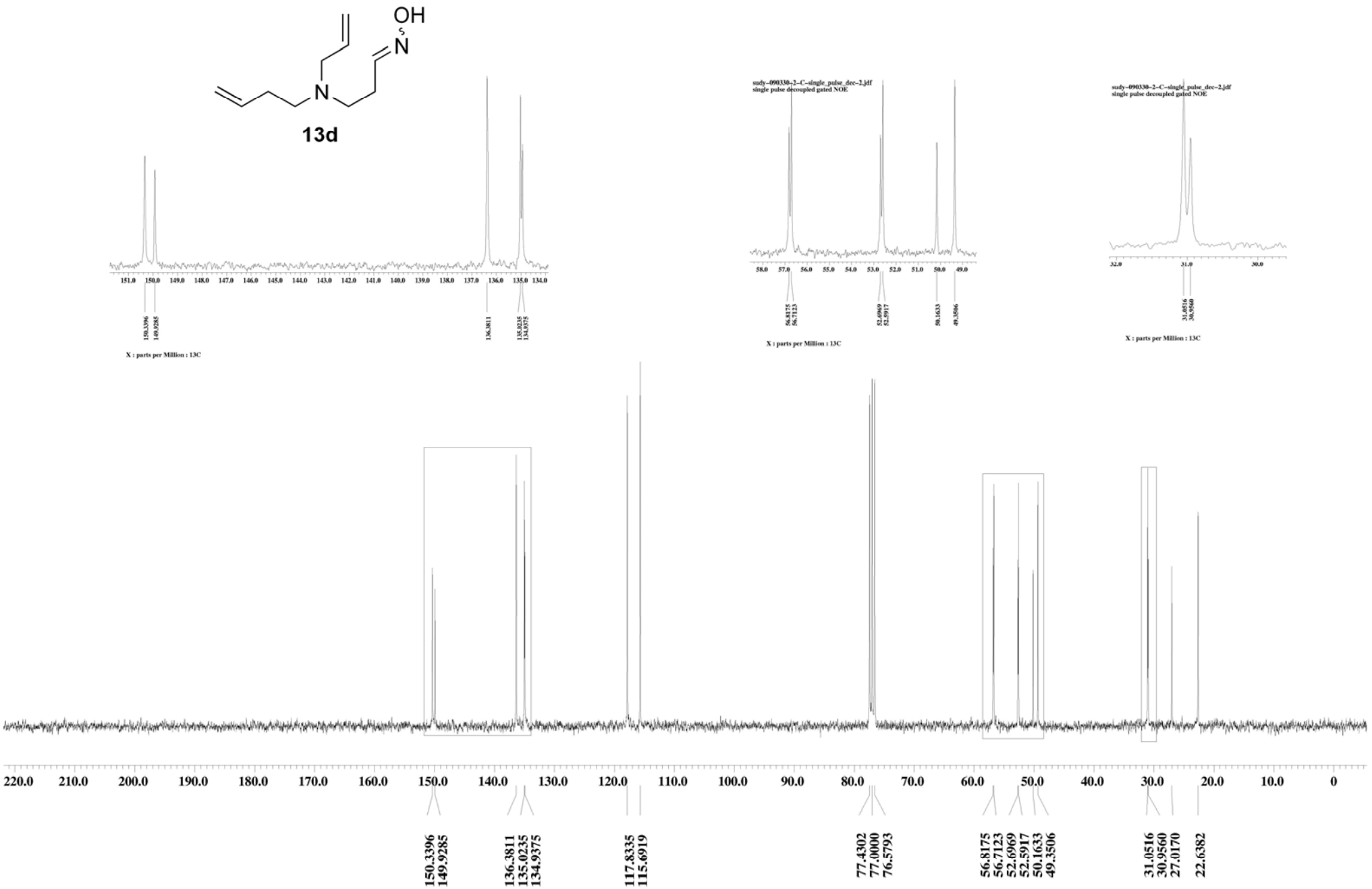

single pulse decoupled gated NOE
31.0 30.0 29.0 28.0 27.0 26.0 25.0
28.5945
28.4798
26.9214
X : parts per Million : 13C

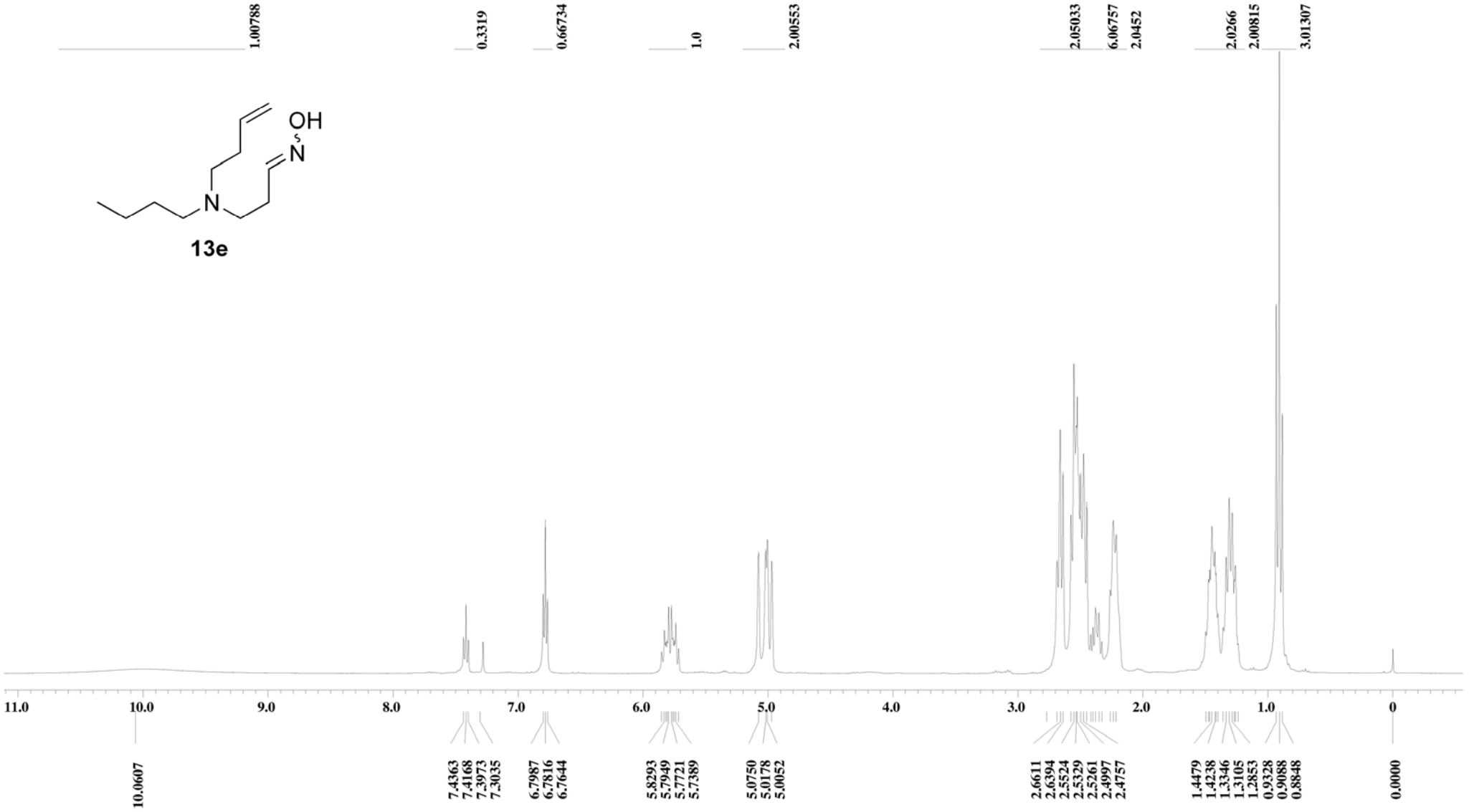

220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
150.1867
149.7086
135.1574
117.6710
77.4207
77.0000
76.5793
56.8940
56.7793
53.0984
52.9837
50.2398
49.4462
28.5945
28.4798
26.9214
22.5331
20.5636
13.9572
X : parts per Million : 13C

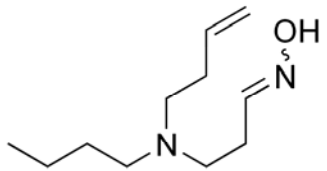

OH
N
N
13c
0.63453
0.32151
0.33704
0.69156
2.0
4.02316
4.02797
2.05218
1.0837
1.00449
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.0066
8.2523
7.4397
7.4191
7.3996
7.2634
6.8136
6.7964
6.7793
5.9002
5.8659
5.8430
5.8098
5.2158
5.1643
5.1368
3.1384
3.1189
2.6611
2.6405
2.6188
2.5615
2.5421
2.5226
0.0000
X : parts per Million : 1H


OH
N
N
13c
149.8521
149.3931
134.7080
134.5646
118.0725
117.9960
77.4207
77.0000
76.5793
56.3873
56.2439
49.6853
48.8917
26.7971
22.3132
X : parts per Million : 13C

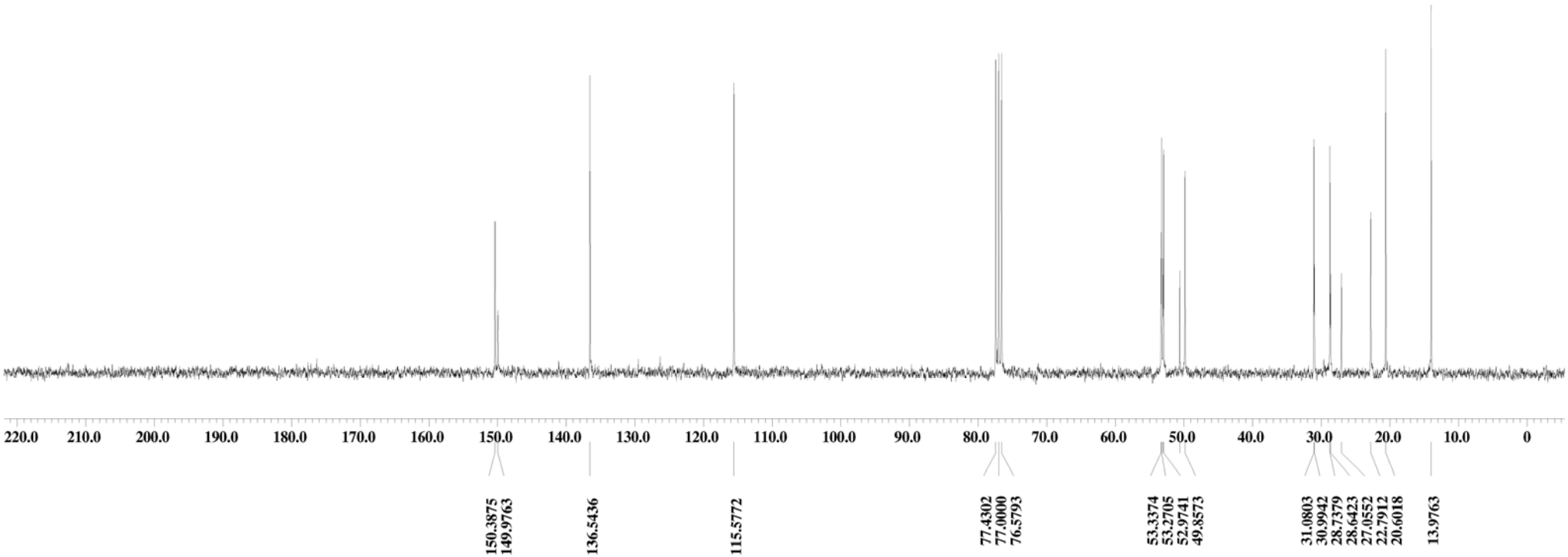

1.00969
0.40367
0.61311
2
4.0103
2.06889
2.09511
3.0323
1.00824
2.00878
OH
N
N
13d
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.1821
9.7426
7.4328
7.4122
7.3928
7.2783
6.7907
6.7724
6.7552
5.8808
5.8579
5.8236
5.7663
5.2204
5.1643
5.1322
5.0178
5.0098
3.1659
3.1441
2.6783
2.6531
2.5902
2.5673
2.5581
2.5386
2.5318
0.0000


OH
N
N
13d
X : parts per Million : 13C
X : parts per Million : 13C
X : parts per Million : 13C
150.3396
149.9285
136.3811
135.0235
134.9375
117.8335
115.6919
77.4302
77.0000
76.5793
56.8175
56.7123
52.6969
52.5917
50.1633
49.3506
31.0516
30.9560
27.0170
22.6382


1.00788
0.3319
0.66734
1.0
2.00553
2.05033
6.06757
2.0452
2.0266
2.00815
3.01307
OH
N
N
13e
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.0607
7.4363
7.4168
7.3973
7.3035
6.7987
6.7816
6.7644
5.8293
5.7949
5.7721
5.7389
5.0750
5.0178
5.0052
2.6611
2.6394
2.5524
2.5329
2.5261
2.4997
2.4757
1.4479
1.4238
1.3346
1.3105
1.2853
0.9328
0.9088
0.8848
0.0000


OH
N
N

13e


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
150.3875
149.9763
136.5436
115.5772
77.4302
77.0000
76.5793
53.3374
53.2705
52.9741
49.8573
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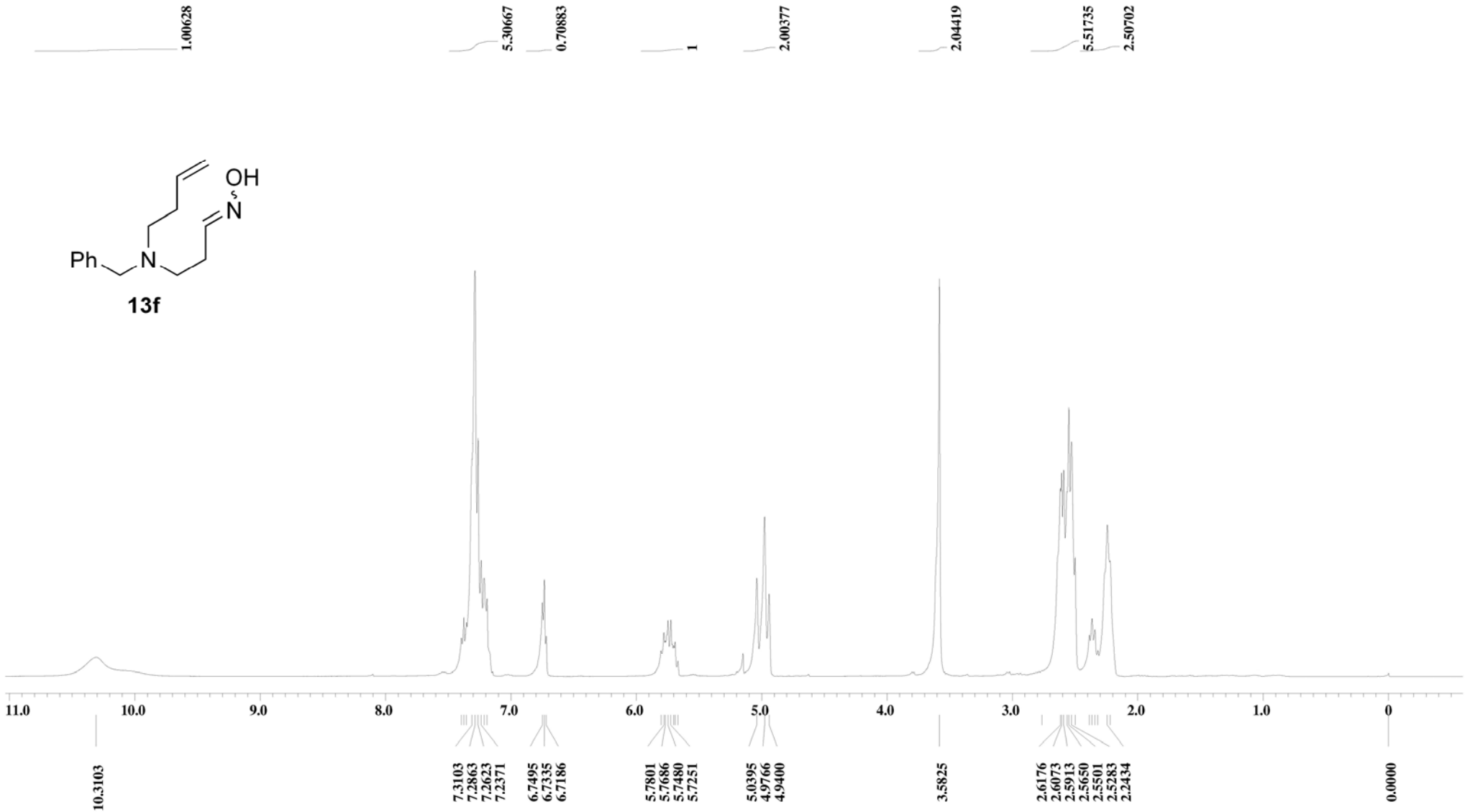
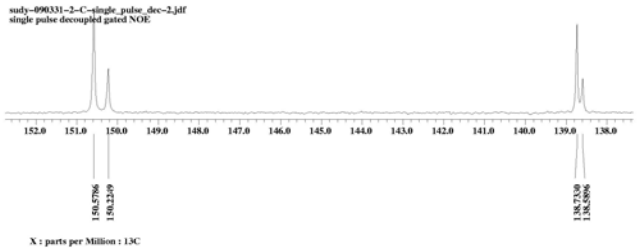
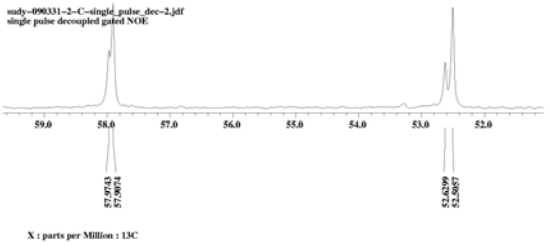
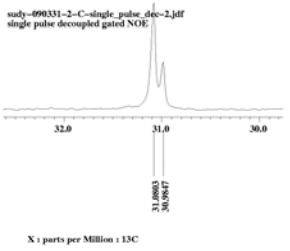
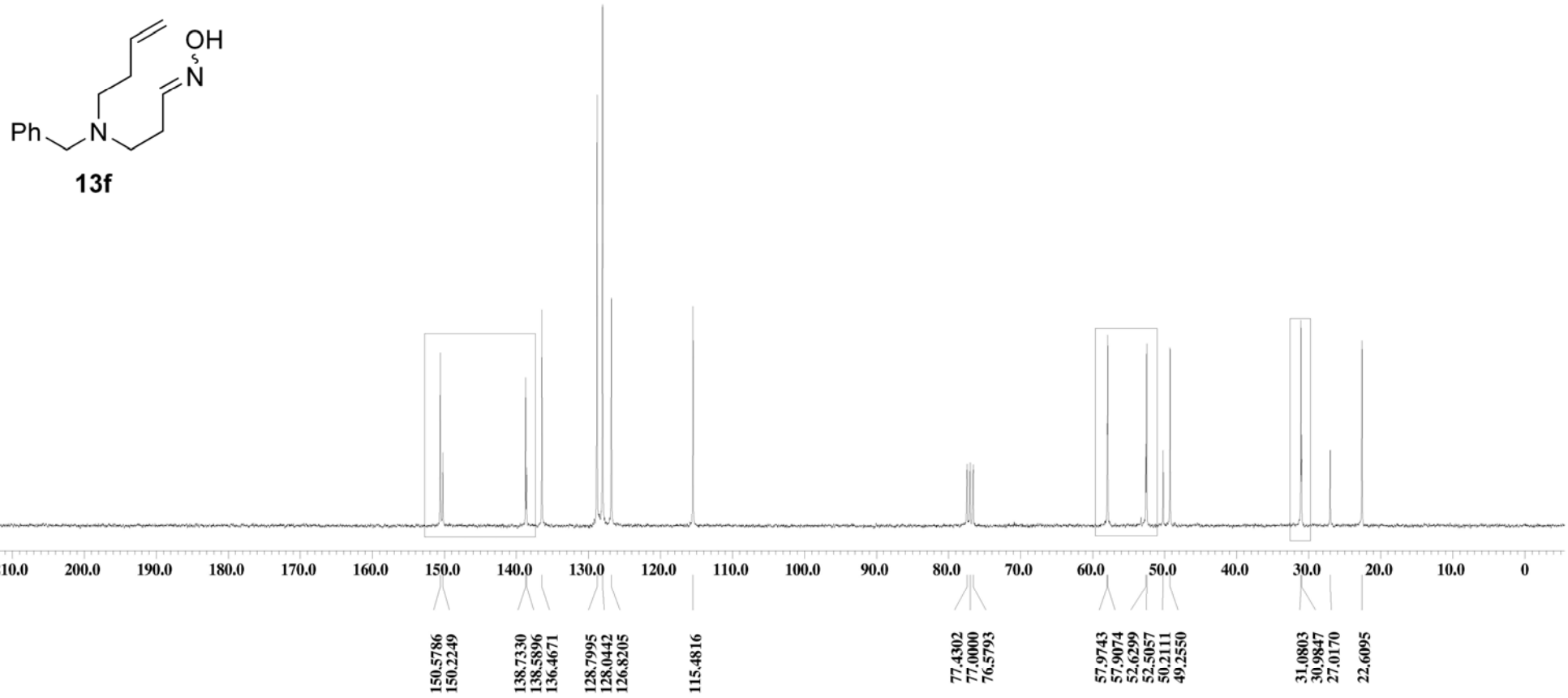
28.6423
27.0552
22.7912
20.6018
13.9763

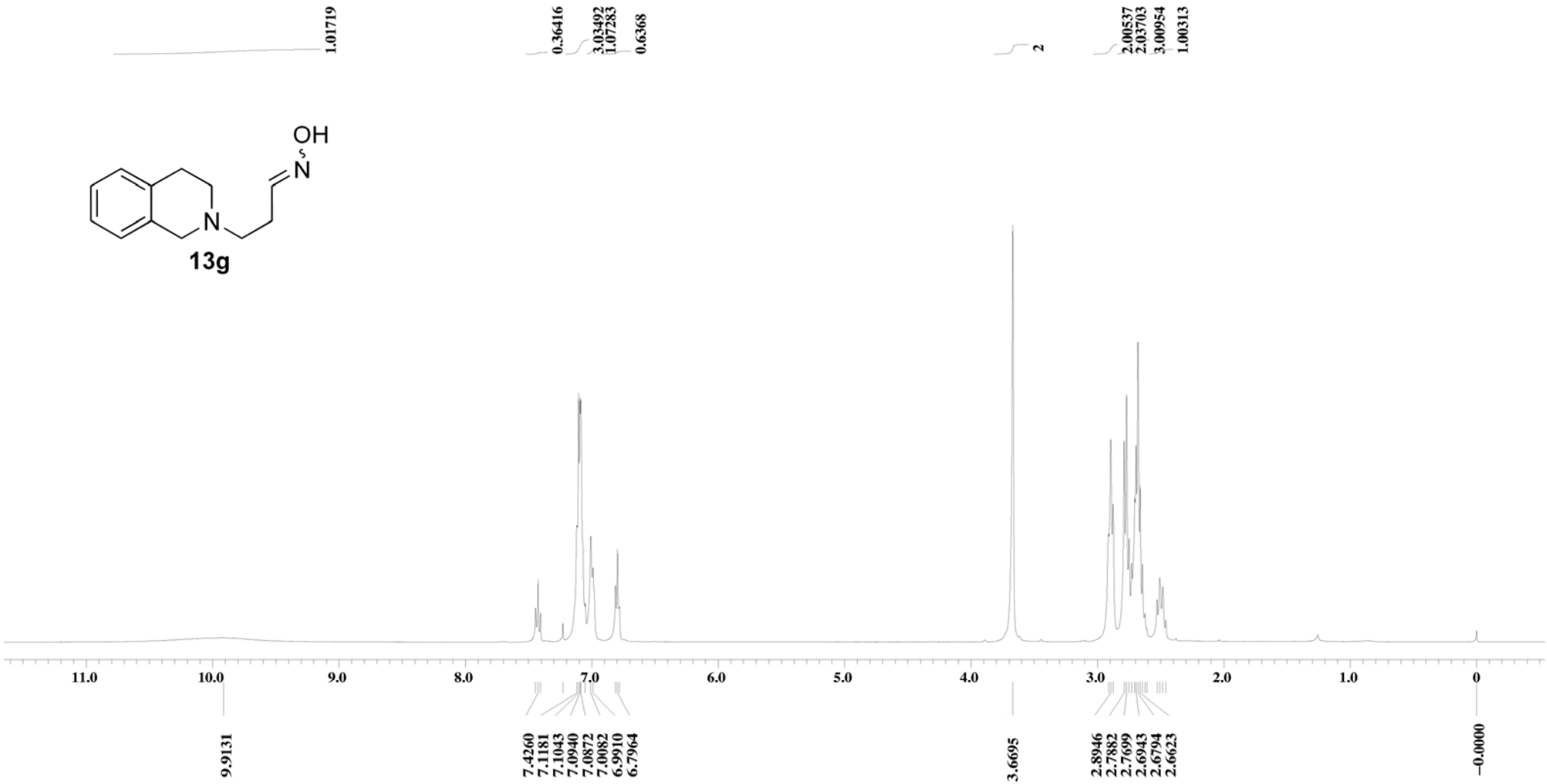

1.00628
5.30667
0.70883
1
2.00377
2.04419
5.51735
2.50702
OH
N
Ph
N
13f
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.3103
7.3103
7.2863
7.2623
7.2371
6.7495
6.7335
6.7186
5.7801
5.7686
5.7480
5.7251
5.0395
4.9766
4.9400
3.5825
2.6176
2.6073
2.5913
2.5650
2.5501
2.5283
2.2434
0.0000

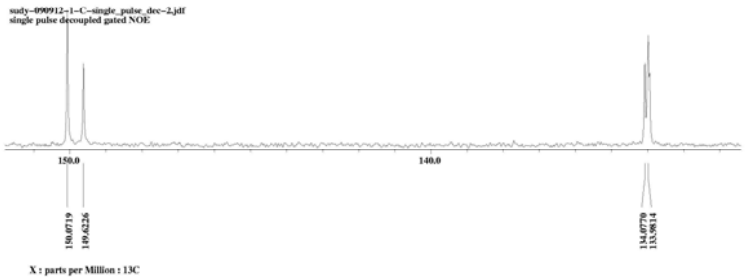

sudy-090331-2-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
152.0 151.0 150.0 149.0 148.0 147.0 146.0 145.0 144.0 143.0 142.0 141.0 140.0 139.0 138.0
150.5786
150.2249
138.7330
138.5896
X : parts per Million : 13C

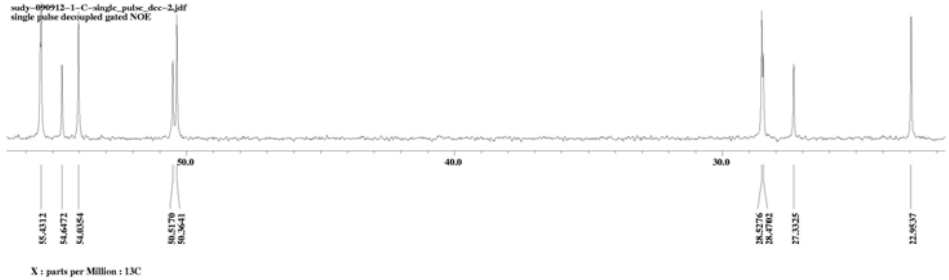

sudy-090331-2-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
59.0 58.0 57.0 56.0 55.0 54.0 53.0 52.0
57.9743
57.9074
52.6299
52.5057
X : parts per Million : 13C

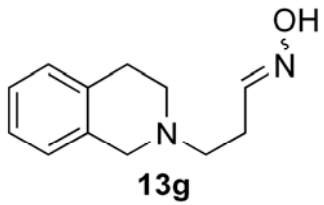

sudy-090331-2-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
32.0 31.0 30.0
31.0803
30.9847
X : parts per Million : 13C


OH
N
Ph
N
13f
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
150.5786
150.2249
138.7330
138.5896
136.4671
128.7995
128.0442
126.8205
115.4816
77.4302
77.0000
76.5793
57.9743
57.9074
52.6299
52.5057
50.2111
49.2550
31.0803
30.9847
27.0170
22.6095


1.01719
0.36416
3.03492
1.07283
0.6368
2
2.00537
2.03703
3.00954
1.00313
OH
N
N
13g
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.9131
7.4260
7.1181
7.1043
7.0940
7.0872
7.0082
6.9910
6.7964
3.6695
2.8946
2.7882
2.7699
2.6943
2.6794
2.6623
-0.0000

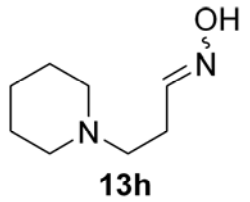

sudy-090912-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
150.0
140.0
150.0719
149.6226
134.0770
133.9814
X : parts per Million : 13C


sudy-090912-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
50.0
40.0
30.0
55.4312
54.6472
54.0354
50.5170
50.3641
28.5276
28.4702
27.3325
22.9537
X : parts per Million : 13C


OH
N
N

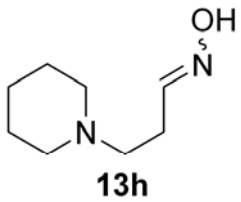
13g

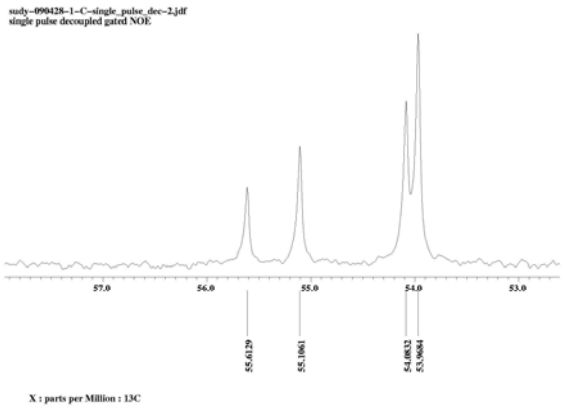
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
150.0719
149.6226
134.0770
133.9814
128.5414
126.5050
126.1130
125.5872
77.4207
77.0000
76.5793
55.4312
54.6472
54.0354
50.5170
50.3641
28.5276
28.4702
27.3325
22.9537

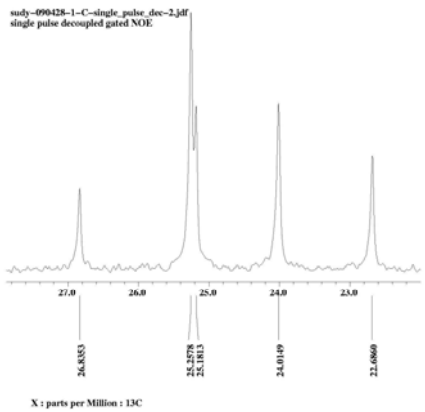

OH
N
N

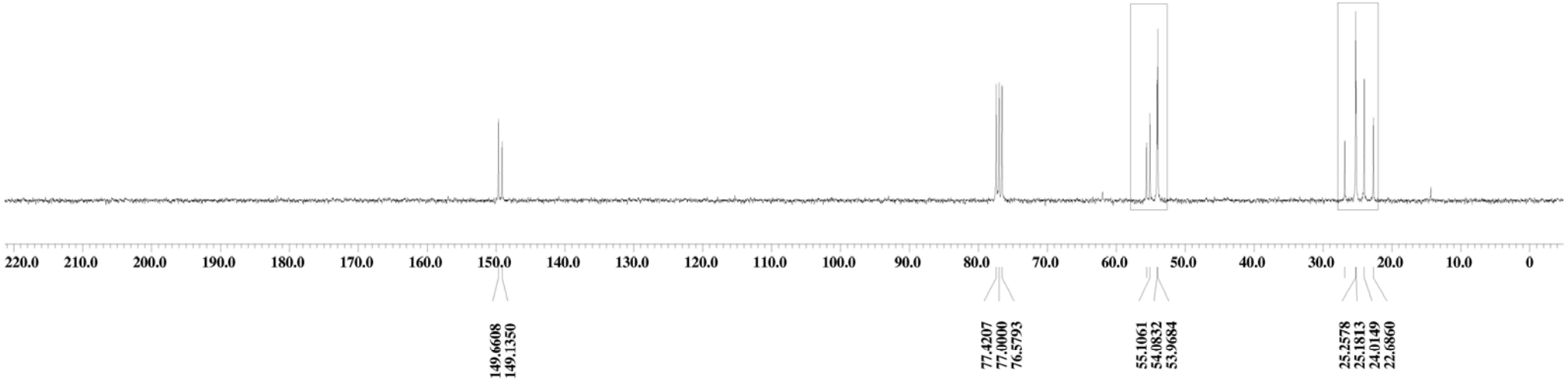
13h

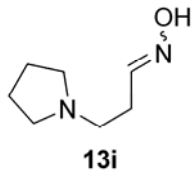
0.28471
0.70055
8.06691
4.06221
2.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4649
7.4454
7.4271
7.2623
6.8640
6.8479
2.5077
2.4952
2.4837
2.4631
1.6253
1.6070
1.5887
1.5715
0.0000


OH
N
N
13h

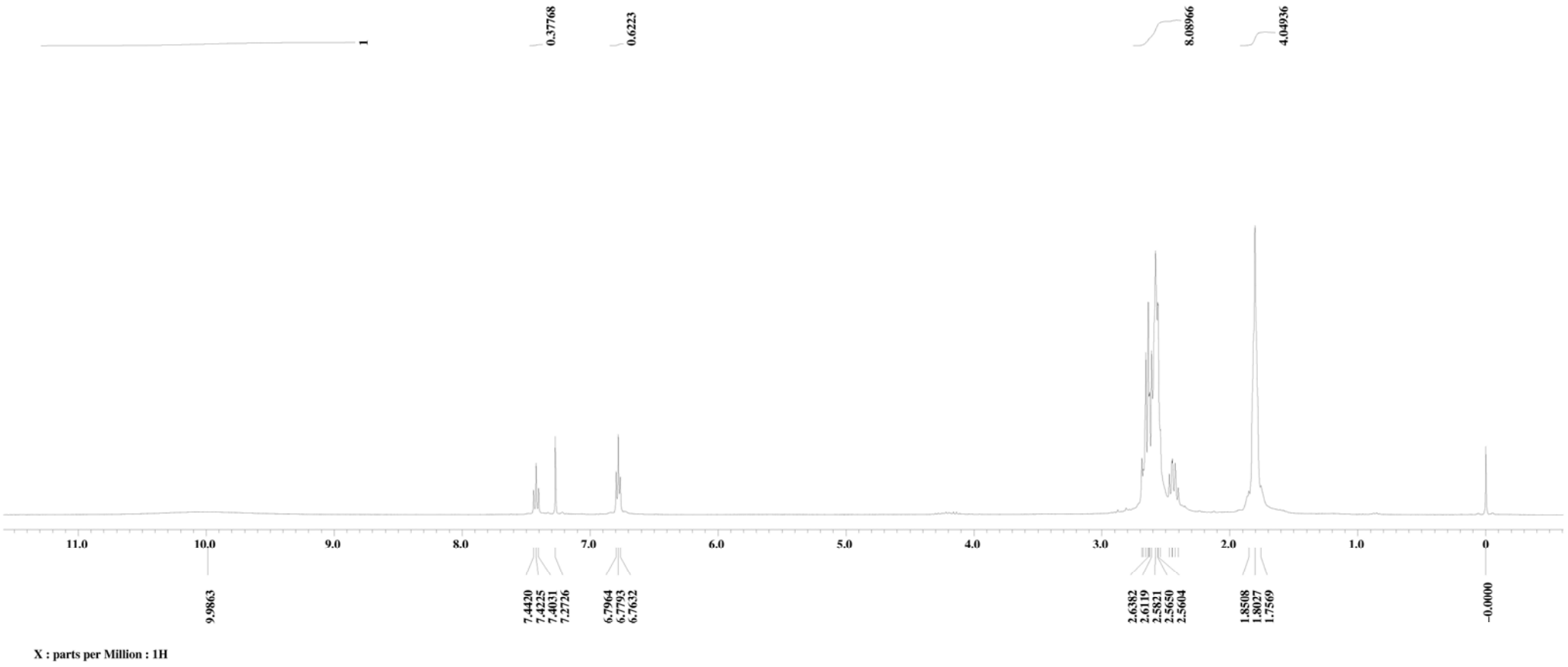

sudy-090428-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
57.0
56.0
55.0
54.0
53.0
55.6129
55.1061
54.0832
53.9684
X : parts per Million : 13C

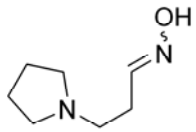

sudy-090428-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
27.0
26.0
25.0
24.0
23.0
26.8353
25.2578
25.1813
24.0149
22.6860
X : parts per Million : 13C


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
149.6608
149.1350
77.4207
77.0000
76.5793
55.1061
54.0832
53.9684
25.2578
25.1813
24.0149
22.6860

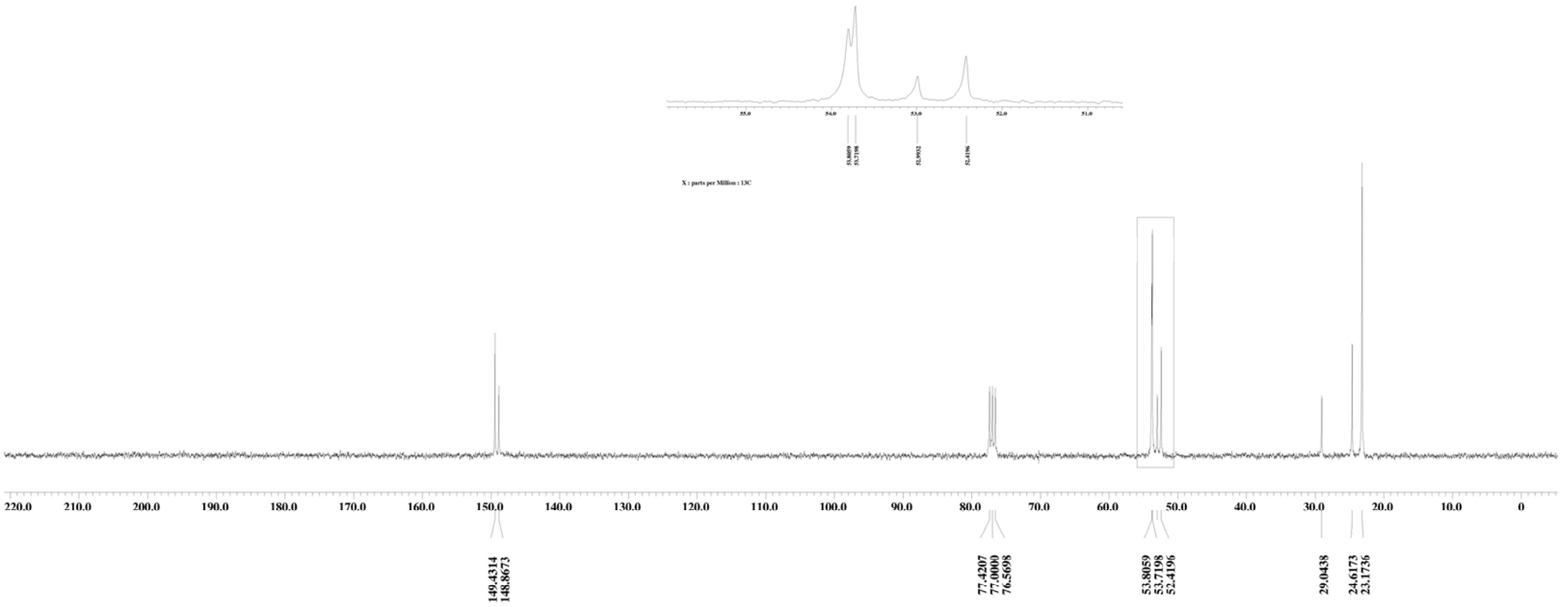

OH
N
N

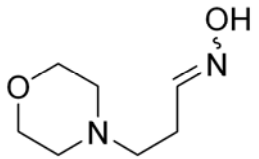
13i


1
0.37768
0.6223
8.08966
4.04936
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.9863
7.4420
7.4225
7.4031
7.2726
6.7964
6.7793
6.7632
2.6382
2.6119
2.5821
2.5650
2.5604
1.8508
1.8027
1.7569
-0.0000
X : parts per Million : 1H


OH
N
N

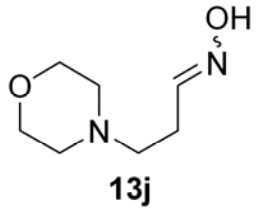
13i


55.0
54.0
53.0
52.0
51.0
53.8059
53.7198
52.9932
52.4196
X : parts per Million : 13C
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
149.4314
148.8673
77.4207
77.0000
76.5698
53.8059
53.7198
52.4196
29.0438
24.6173
23.1736
X : parts per Million : 13C

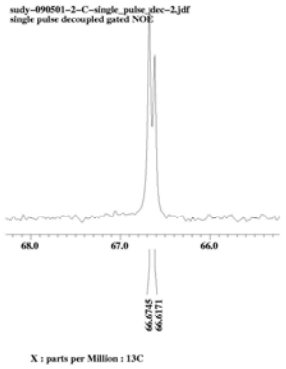

OH
N
O
N

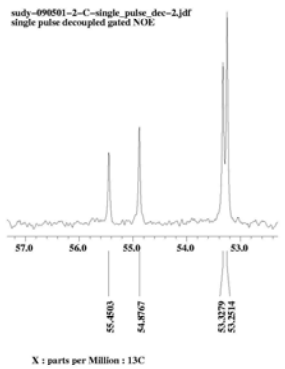
13j

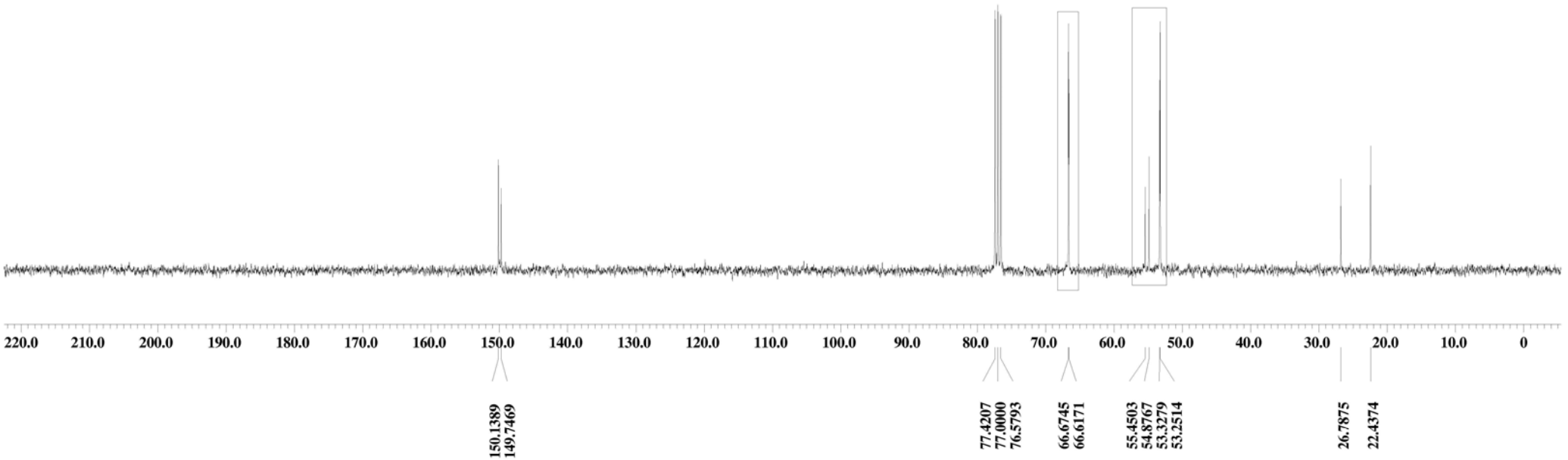
0.96481
0.42296
0.5733
4
8.03484
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.6453
9.2309
7.4580
7.4385
7.4191
7.2772
6.8033
6.7896
6.7724
3.7496
3.7336
3.7187
2.5799
2.5661
2.5558
2.5009
0.0000

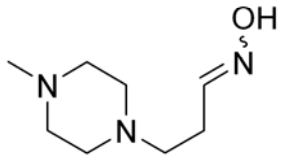

OH
N
O
N

13j

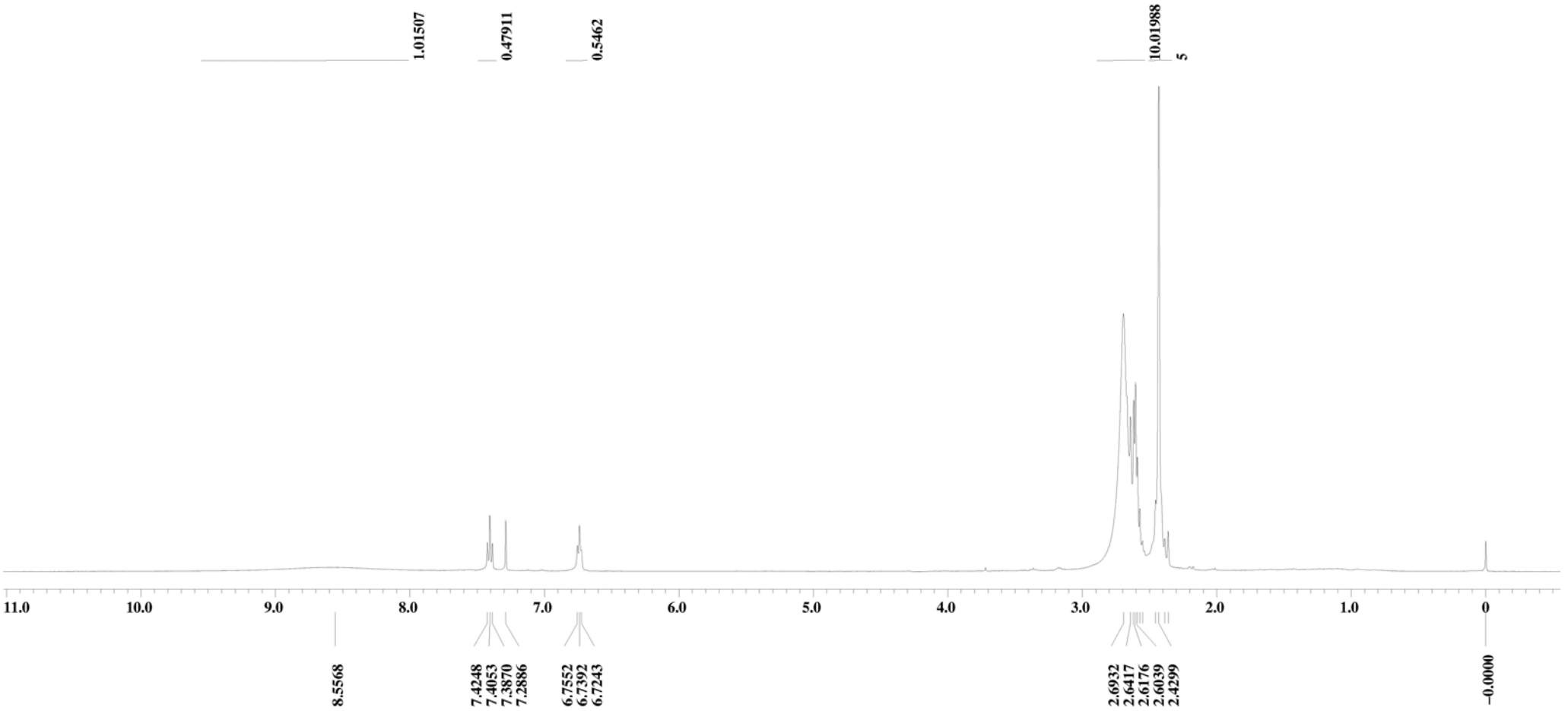

sudy-090501-2-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
68.0
67.0
66.0
66.6745
66.6171
X : parts per Million : 13C

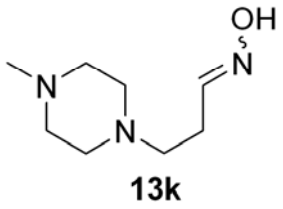

sudy-090501-2-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
57.0
56.0
55.0
54.0
53.0
55.4503
54.8767
53.3279
53.2514
X : parts per Million : 13C


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
150.1389
149.7469
77.4207
77.0000
76.5793
66.6745
66.6171
55.4503
54.8767
53.3279
53.2514
26.7875
22.4374

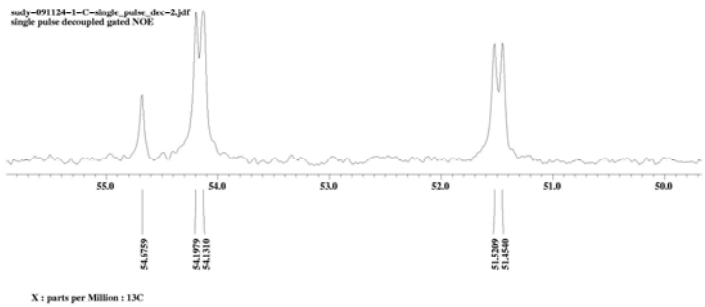

OH
N
N
N

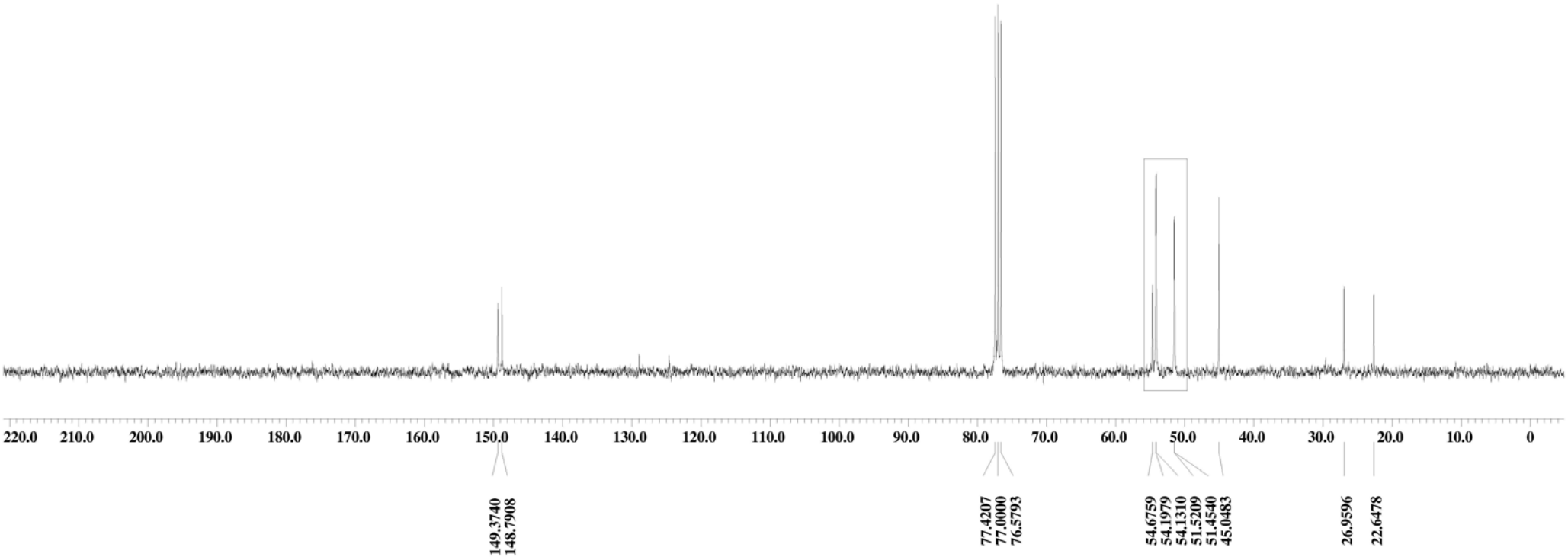
13k

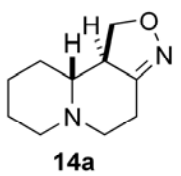

1.01507
0.47911
0.5462
10.01988
5
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.5568
7.4248
7.4053
7.3870
7.2886
6.7552
6.7392
6.7243
2.6932
2.6417
2.6176
2.6039
2.4299
-0.0000


OH
N
N
N

13k

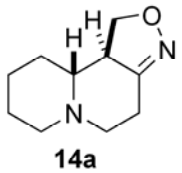

sudy-091124-1-C-single_pulse_dec-2.jdf
single pulse decoupled gated NOE
55.0
54.0
53.0
52.0
51.0
50.0
54.6759
54.1979
54.1310
51.5209
51.4540
X : parts per Million : 13C


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
149.3740
148.7908
77.4207
77.0000
76.5793
54.6759
54.1979
54.1310
51.5209
51.4540
45.0483
26.9596
22.6478

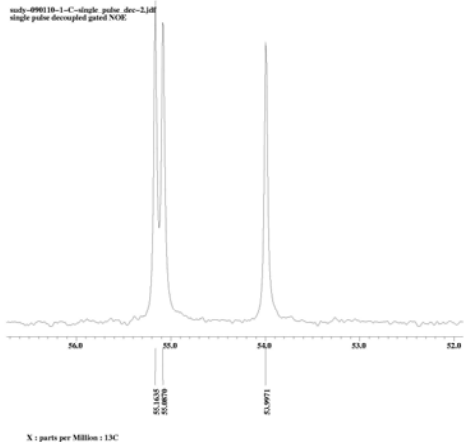

H
H
O
N
N

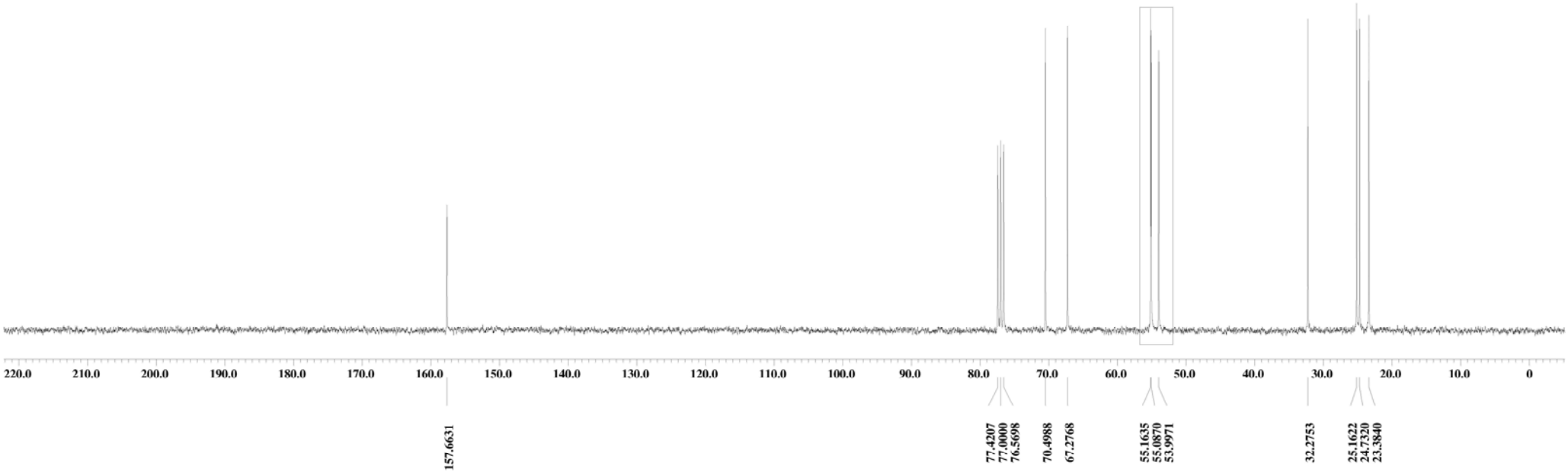
14a

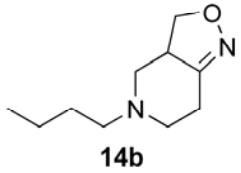
1
1.00683
3.01313
1.00245
1.00101
2.00313
5.08802
2.09216
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3367
4.4638
4.4558
4.4295
3.8343
3.8080
3.8000
3.0503
3.0159
3.0022
2.9759
2.7504
2.7092
2.7035
2.6989
2.5100
2.1644
2.1621
2.0797
2.0694
1.7764
1.7706
1.7432
1.6668
1.6276
1.6161
1.2956
1.2922
1.2636
0.0000
X : parts per Million : 1H



14a

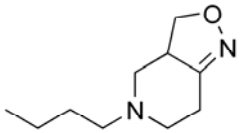

sudy-090110-1-C--single_pulse_dec--2.jdf
single pulse decoupled gated NOE
56.0
55.0
54.0
53.0
52.0
55.1635
55.0870
53.9971
X : parts per Million : 13C


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
157.6631
77.4207
77.0000
76.5698
70.4988
67.2768
55.1635
55.0870
53.9971
32.2753
25.1622
24.7320
23.3840
X : parts per Million : 13C

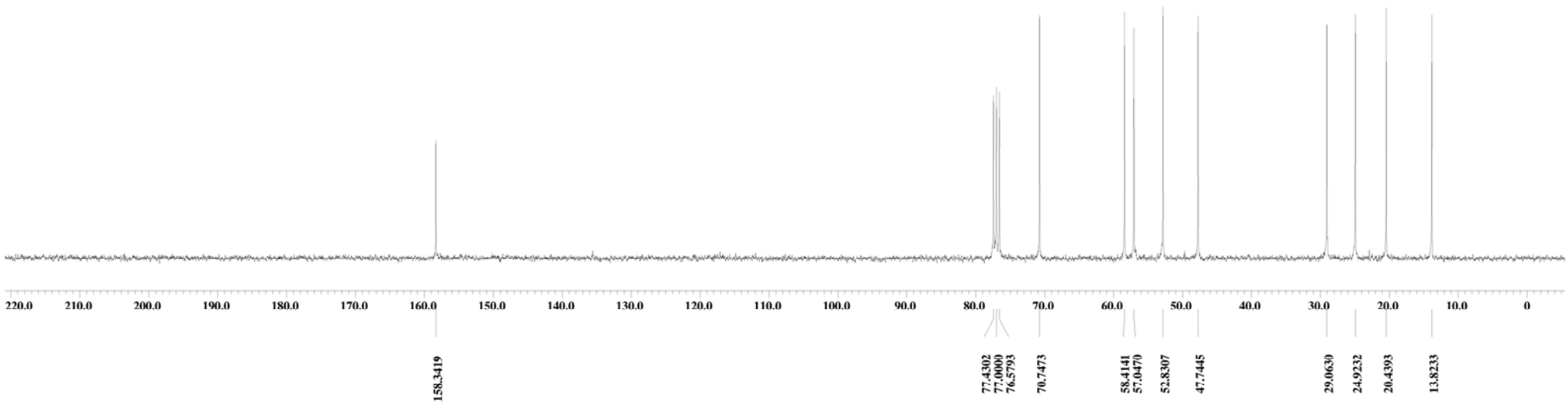


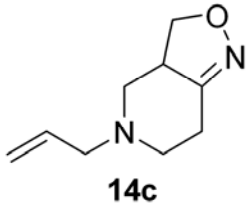
14b

X : parts per Million : 1H


O
N
N

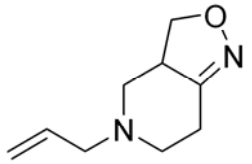
14b


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
158.3419
77.4302
77.0000
76.5793
70.7473
58.4141
57.0470
52.8307
47.7445
29.0630
24.9232
20.4393
13.8233
X : parts per Million : 13C

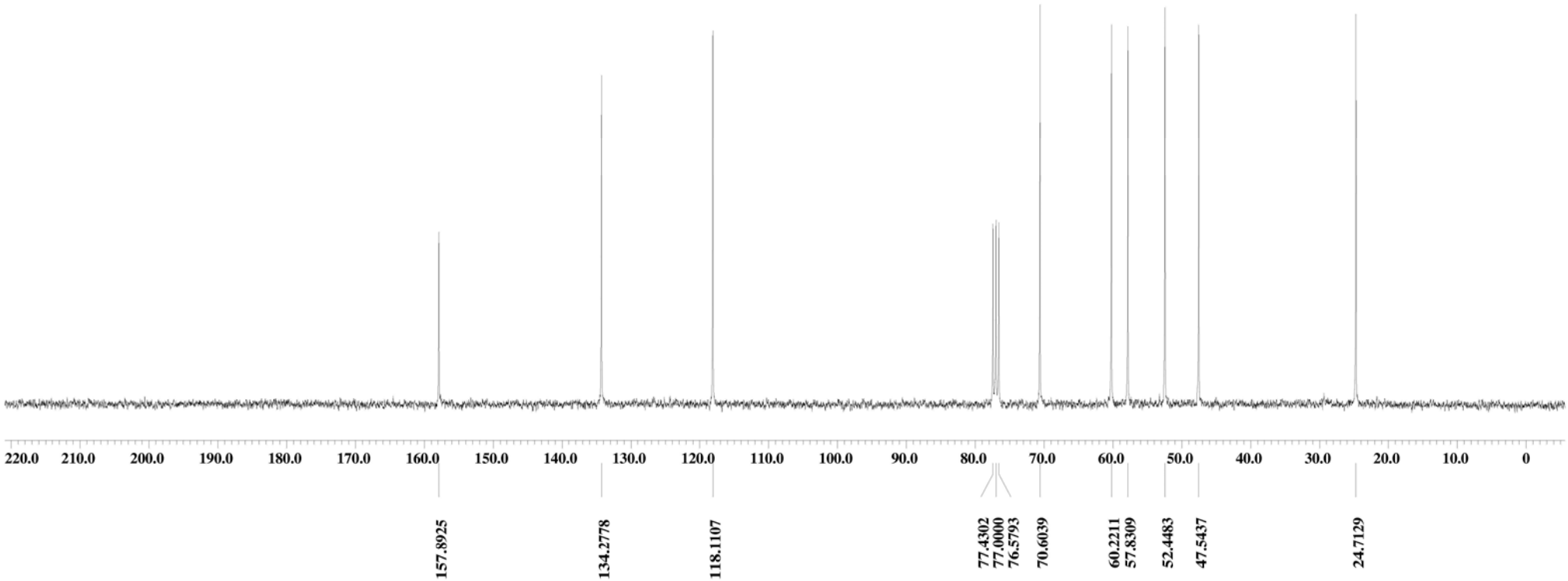


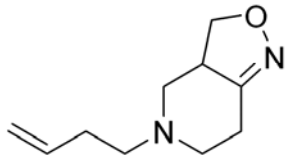
14c

1
2.04638
1.01817
1.00394
5.02516
1.07002
1.02433
2.0056
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3607
5.9014
5.8796
5.8682
5.8453
5.2421
5.2021
5.1849
5.1700
4.4890
4.4615
4.4272
3.7954
3.7679
3.7610
3.7336
3.3307
3.3044
3.2952
3.1945
3.1739
3.1556
3.1270
3.1075
3.0858
2.7309
2.6874
2.1037
2.0923
2.0236
1.9881
0.0000


O
N
N

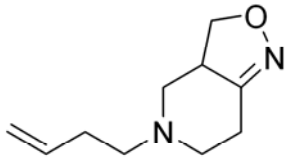
14c


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
157.8925
134.2778
118.1107
77.4302
77.0000
76.5793
70.6039
60.2211
57.8309
52.4483
47.5437
24.7129

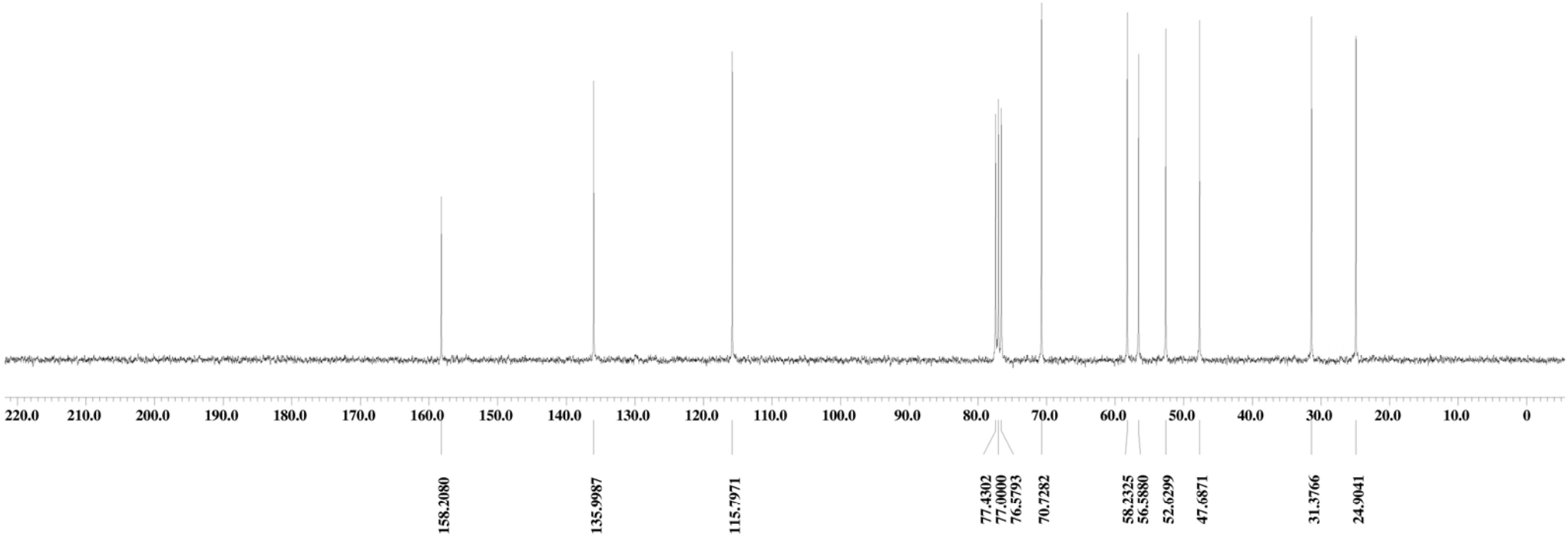


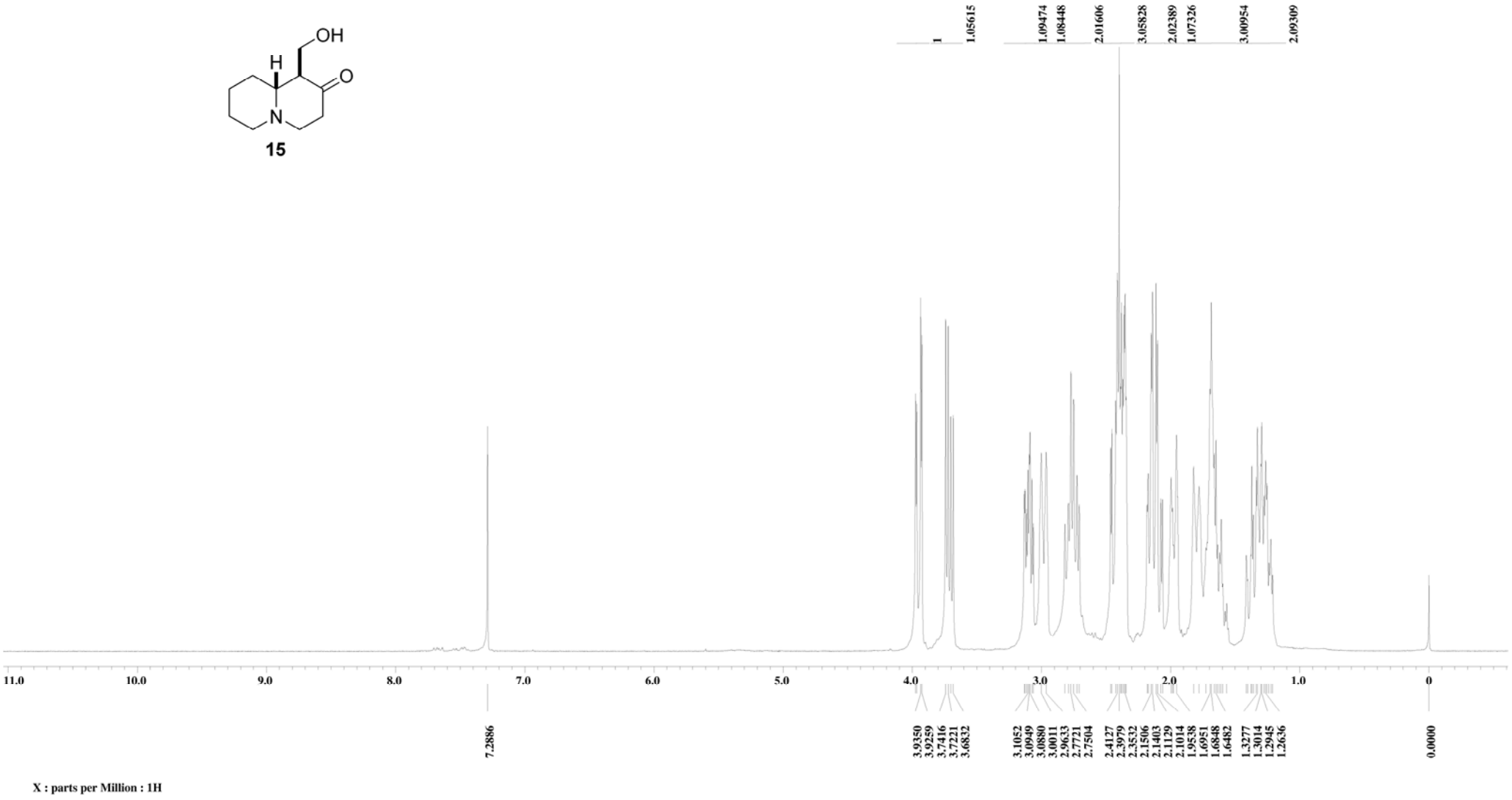
14d

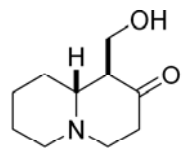
1
1.99552
1.01131
1.06516
1.07944
1.03246
1.07299
1.05048
3.07192
2.09461
2.02989
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2623
5.8167
5.7938
5.7824
5.7595
5.1036
5.0464
5.0418
4.4753
4.4684
4.4409
3.7851
3.7771
3.7496
3.3055
3.2700
3.1624
3.1453
3.1258
2.6897
2.5684
2.5524
2.5444
2.5249
2.5203
2.2800
2.2559
2.0419
0.0000


O
N
N

14d

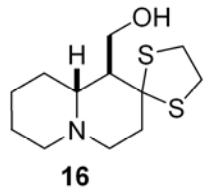

220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
158.2080
135.9987
115.7971
77.4302
77.0000
76.5793
70.7282
58.2325
56.5880
52.6299
47.6871
31.3766
24.9041


OH
H
O
N
15
1
1.05615
1.09474
1.08448
2.01606
3.05828
2.02389
1.07326
3.00954
2.09309
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2886
3.9350
3.9259
3.7416
3.7221
3.6832
3.1052
3.0949
3.0880
3.0011
2.9633
2.7721
2.7504
2.4127
2.3979
2.3532
2.1506
2.1403
2.1129
2.1014
1.9538
1.6951
1.6848
1.6482
1.3277
1.3014
1.2945
1.2636
0.0000
X : parts per Million : 1H


OH
H
O
N

15

220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
211.9484
77.4207
77.0000
76.5698
62.9937
58.3472
56.5594
55.6989
55.6702
41.6735
31.0707
25.2865
23.2501
X : parts per Million : 13C


OH
H
S
S
N

16

1
1.00076
4.01975
3.06041
2.00024
4.05448
2.04638
2.05072
2.05357
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3081
4.2143
4.1719
4.1662
3.9030
3.8904
3.8618
3.3353
3.3078
3.3055
3.2883
2.8568
2.8214
2.8122
2.7721
2.4253
2.1186
2.0797
2.0671
2.0614
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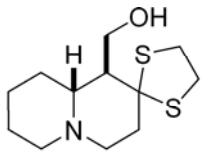
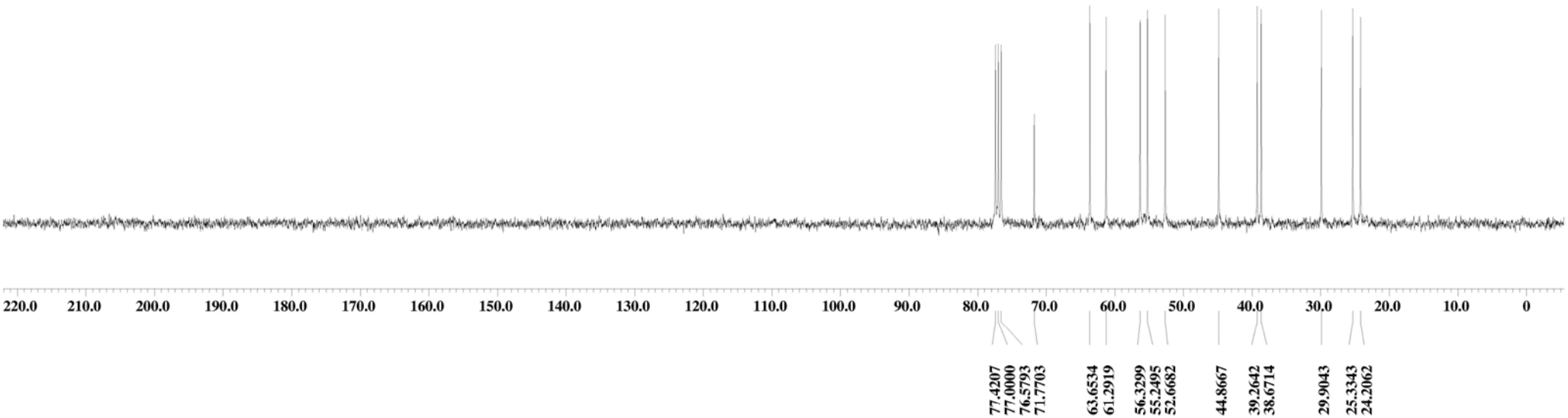
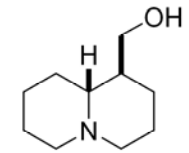
1.7649
1.7592
1.6047
1.2762
1.2625
1.2522
1.2224
-0.0000

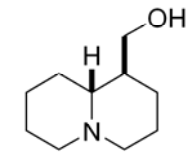

OH
H
S
S
N

16


220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
77.4207
77.0000
76.5793
71.7703
63.6534
61.2919
56.3299
55.2495
52.6682
44.8667
39.2642
38.6714
29.9043
25.3343
24.2062



(**2**)
(+)-Epilupinine



(**2**)
(+)-Epilupinine