

Supporting Information

Gold(I)-Catalyzed Cyclization–3-Aza-Cope–Mannich Cascade and its Application to the Synthesis of Cephalotaxine

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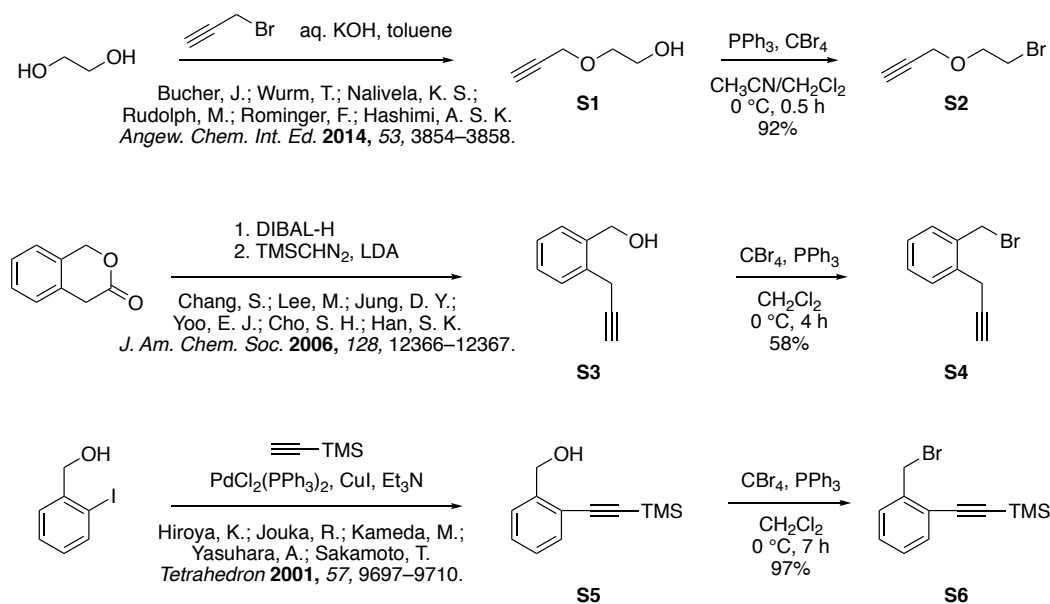
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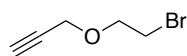
General Information

All air- and moisture-sensitive reactions were carried out under an argon atmosphere in commercially available dry solvents under anhydrous conditions. Heating reactions were performed using an oil bath except for microwave experiments, and the microwave experiments were carried out in a Discover SP microwave reactor (CEM corporation). Flash chromatography was carried out using silica gel (spherical, neutral, particle size 40-50 μm , Kanto Chemical), NH_2 silica gel (Silica gel 60 (spherical) NH_2 , particle size 40–50 μm , Kanto Chemical) or alumina (aluminum oxide 90, standardized, particle size 63–200 μm , Activity grade II-III, Merck). Preparative HPLC was performed using LaboACE LC-5060 and Analytical HPLC was performed Shimadzu LC-20 series. Specific optical rotations were recorded on a JASCO P-1020 polarimeter. IR spectra were recorded on a JASCO FT/IR-410 spectrophotometer. NMR spectra were recorded on AVANCE III HD (600 MHz), JNM-ECA (500 MHz), or JNM-ECZS (400 MHz) spectrometers. Chemical shifts are reported in ppm relative to the solvent signals (δ 1.94 ppm for CD_3CN) or internal TMS (δ 0.00 ppm for CDCl_3) for ^1H NMR spectra and to the solvent signals (δ 1.39 ppm for CD_3CN , δ 77.0 ppm for CDCl_3) for ^{13}C NMR spectra. Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad). High-resolution mass spectra were recorded on an Exactive Plus orbitrap mass spectrometer (DART, ESI) or a JMS-700 magnetic sector mass spectrometer (EI).

Synthesis of alkyl bromides **S2**, **S4**, and **S6**



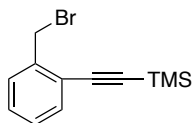
Scheme S1. Synthesis of alkyl halides **S2**, **S4**, and **S6**.



2-Bromoethyl propargyl ether (S2). To a solution of 2-(propargyloxy)ethanol¹ (**S1**) (2.28 g, 14.0 mmol) and CBr₄ (6.96 g, 21.0 mmol) in CH₃CN (28 mL) at 0 °C was slowly added a solution of PPh₃ (5.51 g, 21.0 mmol) in CH₂Cl₂ (14 mL) over 5-min period. The reaction mixture was stirred at 0 °C for 0.5 h and the reaction was quenched with saturated aqueous NaHCO₃ solution. The resulting mixture was extracted with Et₂O, washed with water and brine, dried over MgSO₄, filtered, and concentrated under reduced pressure. Flash chromatography on silica gel (8% Et₂O/*n*-hexane) afforded 2-bromoethyl propargyl ether (**S2**) (2.11 g, 92%) as a colorless oil. ¹H NMR (CDCl₃, 600 MHz) δ 4.23 (2H, d, *J* = 2.4 Hz), 3.87 (2H, t, *J* = 6.1 Hz), 3.50 (2H, t, *J* = 6.1 Hz), 2.47 (1H, t, *J* = 2.4 Hz). Spectroscopic data was consistent with that reported in Hu et al.²



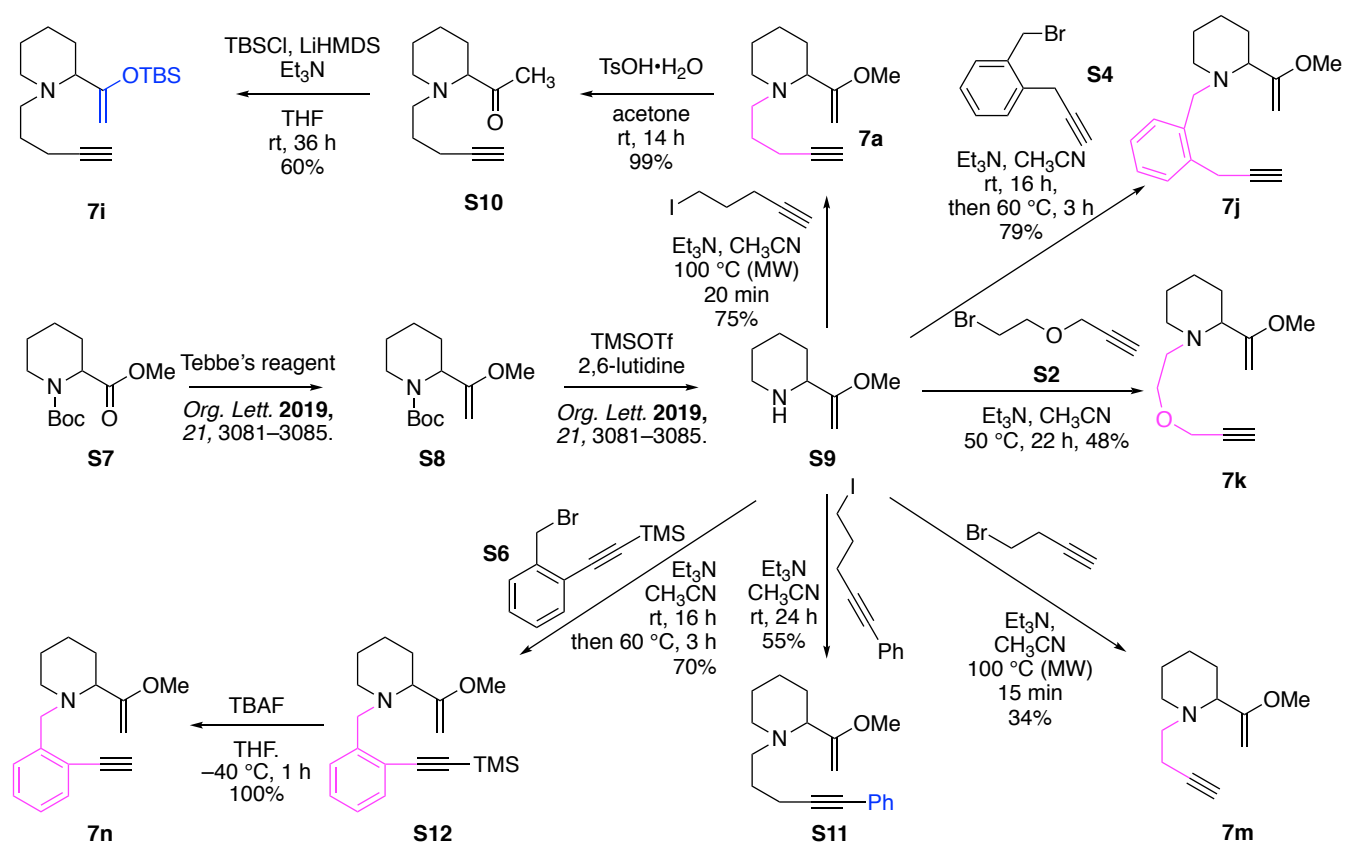
2-Propargylbenzyl bromide (S4). To a solution of 2-propargylbenzyl alcohol³ (**S3**) (983 mg, 6.72 mmol) and carbon tetrabromide (2.45 g, 7.40 mmol, 1.1 equiv) in CH₂Cl₂ (7 mL) at 0 °C was slowly added PPh₃ (1.94 g, 7.40 mmol) over 15-min period. The reaction mixture was stirred at 0 °C for 4 h and then concentrated under reduced pressure. The resulting mixture was diluted with *n*-hexane and resulting precipitate of triphenylphosphine oxide was removed by suction filtration. The residue was concentrated and purified by flash chromatography on silica gel (100% hexane) afforded 2-propargylbenzyl bromide (**S4**) (811 mg, 58%) as a colorless solid. Mp 40–41 °C; IR (film) 3284, 2123, 1456, 1225, 1210, 759, 715, 645, 602 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.51 (1H, d, *J* = 7.5 Hz), 7.34–7.31 (2H, m), 7.25 (1H, td, *J* = 7.5, 0.9 Hz), 4.58 (2H, s), 3.74 (2H, d, *J* = 2.8 Hz), 2.22 (1H, t, *J* = 2.8 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ 135.23, 135.21, 130.4, 129.39, 129.36, 127.6, 81.0, 71.2, 31.2, 22.0; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₀H₁₀Br⁺ 208.9960, found 208.9956.



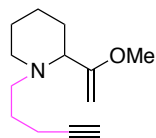
2-((Trimethylsilyl)ethynyl)benzyl bromide (S6). To a solution of 2-((trimethylsilyl)ethynyl)benzyl alcohol⁴ (**S5**) (2.04 g, 10.0 mmol) and carbon tetrabromide (3.98 g, 12.0 mmol, 1.2 equiv) in CH₂Cl₂ (10 mL) at 0 °C was slowly added PPh₃ (3.15 g, 12.0 mmol, 1.2 equiv) over 15-min period. The reaction mixture was stirred at 0 °C for 7 h and then concentrated under reduced pressure. The resulting mixture was diluted with *n*-hexane and resulting precipitate of triphenylphosphine oxide was removed by suction filtration. The residue was concentrated and purified by flash chromatography on silica gel (100% hexane) afforded 2-((trimethylsilyl)ethynyl)benzyl bromide (**S6**) (2.60 g, 97%) as a colorless oil. ¹H NMR (CDCl₃, 600 MHz) δ 7.46 (1H, dd, *J* = 7.5, 1.5 Hz), 7.41 (1H, dd, *J* = 7.5, 1.3 Hz), 7.30 (1H, td, *J* = 7.5, 1.5 Hz),

7.24 (1H, td, $J = 7.5, 1.3$ Hz), 4.67 (2H, s), 0.29 (9H, s). Spectroscopic data was consistent with that reported in Hu et al.⁵

Synthesis of piperidines **7a**, **7i–k**, **7m–n** and **S11**

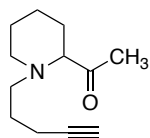


Scheme S2. Synthesis of piperidines **7a**, **7i–k**, **7m–n**, and **S11**.

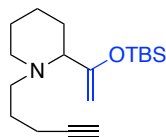


Tertiary amine 7a. A 10-mL, thick-walled reaction vessel was charged with a solution of piperidine **S9** (610 mg, 4.32 mmol), Et₃N (1.18 mL, 8.50 mmol, 2.0 equiv), and 5-iodo-1-pentyne (990 mg, 5.10 mmol, 1.2 equiv) in CH₃CN (4.3 mL). The vessel was sealed and the reaction mixture was heated with a microwave reactor at 100 °C for 20 min. After cooling to room temperature, saturated aqueous NaHCO₃ solution and saturated aqueous Na₂S₂O₃ solution were added. The resulting mixture was extracted with Et₂O, and the extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced

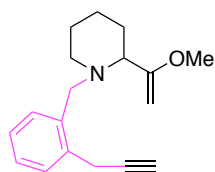
pressure. Flash chromatography on NH₂ silica gel (10% Et₂O/*n*-hexane) afforded tertiary amine **7a** (668 mg, 75%) as a colorless oil. IR (film) 3308, 2936, 1655, 1301 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 4.05 (1H, d, *J* = 1.8 Hz), 3.93 (1H, d, *J* = 1.7 Hz), 3.56 (3H, s), 3.03 (1H, dt, *J* = 11.2, 2.8 Hz), 2.75 (1H, ddd, *J* = 12.8, 10.0, 6.1 Hz), 2.60 (1H, dd, *J* = 10.8, 2.8 Hz), 2.18 (1H, ddd, *J* = 12.8, 9.5, 4.8 Hz), 2.16 (1H, dtd, *J* = 16.9, 7.2, 2.4 Hz), 2.13 (1H, dtd, *J* = 16.9, 7.3, 2.8 Hz), 1.99 (1H, td, *J* = 11.1, 4.1 Hz), 1.91 (1H, t, *J* = 2.6 Hz), 1.78–1.56 (7H, m), 1.25 (1H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 164.6, 84.5, 82.5, 68.1, 67.8, 54.9, 54.1, 52.9, 31.7, 25.7, 24.8, 24.4, 16.6; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₃H₂₂NO⁺ 208.1696, found 208.1696.



Ketone S10. To a solution of methyl vinyl ether **7a** (459 mg, 2.22 mmol) in acetone (10 mL) was added TsOH·H₂O (506 mg, 2.66 mmol, 1.2 equiv), and the mixture was stirred at room temperature for 14 h. The reaction was quenched with Et₃N (1.0 mL), and the resulting mixture was concentrated. Flash chromatography on alumina (0→50% Et₂O in *n*-hexane) afforded ketone **S10** (424 mg, 99%) as a colorless oil. IR (film) 3291, 2938, 2857, 2805, 1710 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.07 (1H, dt, *J* = 11.3, 3.6 Hz), 2.78 (1H, dd, *J* = 10.7, 3.2 Hz), 2.44 (1H, ddd, *J* = 12.6, 8.5, 7.5 Hz), 2.26–2.15 (3H, m), 2.19 (3H, s), 1.931 (1H, t, *J* = 2.7 Hz), 1.927 (1H, td, *J* = 11.4, 3.2 Hz), 1.78–1.62 (5H, m), 1.60–1.47 (2H, m), 1.28 (1H, qt, *J* = 12.1, 3.9 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ 212.2, 84.0, 74.4, 68.6, 55.6, 51.2, 28.7, 25.6, 25.3, 25.1, 23.4, 16.1; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₂H₂₀NO⁺ 194.1539, found 194.1537.

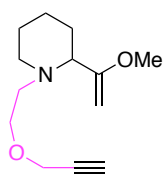


TBS ether 7i. To a solution of ketone **S10** (193 mg, 1.0 mmol) and Et₃N (0.42 mL, 3.0 mmol, 3.0 equiv) in THF (3.0 mL) at –80 °C was added a solution of TBSCl (181 mg, 1.2 mmol, 1.2 equiv) in THF (2 mL). LiHMDS (1.0 M solution in THF, 1.2 mL, 1.2 equiv) was added at –80 °C, and the reaction mixture was warmed to room temperature. After 36 h, the reaction was quenched with saturated NaHCO₃ solution and the resulting mixture was extracted with *n*-hexane. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (5→30% Et₂O/*n*-hexane) afforded TBS ether **7i** (185 mg, 60%) as a colorless oil. IR (film) 3313, 2933, 2857, 2118, 1626, 1300, 1255, 1027, 839 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.19 (1H, d, *J* = 0.9 Hz), 4.05 (1H, t, *J* = 0.9 Hz), 3.02 (1H, dtd, *J* = 11.2, 3.6, 1.0 Hz), 2.86 (1H, ddd, *J* = 12.6, 8.5, 7.3 Hz), 2.41 (1H, dd, *J* = 10.5, 2.7 Hz), 2.24 (1H, dddd, *J* = 16.9, 7.8, 7.0, 2.7 Hz), 2.11 (1H, dddd, *J* = 16.9, 8.0, 7.1, 2.7 Hz), 2.10 (1H, ddd, *J* = 12.6, 8.2, 5.0 Hz), 1.908 (1H, td, *J* = 11.4, 3.0 Hz), 1.906 (1H, t, *J* = 2.7 Hz), 1.73–1.64 (4H, m), 1.61–1.45 (3H, m), 1.23 (1H, qt, *J* = 12.6, 3.9 Hz), 0.94 (9H, s), 0.18 (3H, s), 0.17 (3H, s); ¹³C NMR (CDCl₃, 100 MHz) δ 160.6, 89.9, 84.9, 68.7, 67.9, 54.1, 52.8, 31.9, 25.9 (x2), 25.8, 24.4, 18.2, 16.5, –4.6, –4.7; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₈H₃₄NOSi⁺ 308.2404, found 308.2403.

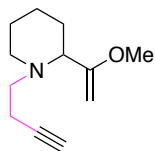


Tertiary amine 7j. A solution of piperidine **S9** (400 mg, 2.83 mmol, 1.0 equiv), Et₃N (2.0 mL, 14.2 mmol, 5.0 equiv), and 2-propargylbenzyl bromide (**S4**) (650 mg, 3.11 mmol, 1.1 equiv) in CH₃CN (2.0 mL) was stirred at room temperature. After 16 h, the mixture was warmed to 60 °C and then stirred for additional 3 h. After cooling to room temperature, saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with EtOAc. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (10% Et₂O/*n*-hexane) afforded tertiary amine **7j** (605 mg, 79%) as a pale yellow oil. IR (film) 3304, 2936, 2795, 1654, 1615,

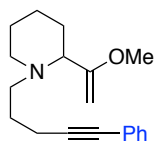
1299 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 7.49 (1H, d, $J = 7.7$ Hz), 7.24–7.22 (2H, m), 7.17 (1H, td, $J = 7.5, 1.3$ Hz), 4.15 (1H, d, $J = 2.0$ Hz), 4.09 (1H, d, $J = 13.0$ Hz), 3.99 (1H, d, $J = 2.0$ Hz), 3.84 (1H, dd, $J = 18.9, 2.8$ Hz), 3.80 (1H, dd, $J = 18.9, 2.8$ Hz), 3.59 (3H, s), 2.86 (1H, d, $J = 13.0$ Hz), 2.72 (1H, dtd, $J = 11.4, 3.5, 1.0$ Hz), 2.59 (1H, dd, $J = 10.8, 2.6$ Hz), 2.14 (1H, t, $J = 2.8$ Hz), 1.80–1.67 (4H, m), 1.47 (1H, m), 1.41 (1H, qt, $J = 12.2, 3.7$ Hz), 1.27 (1H, qt, $J = 12.5, 4.0$ Hz); ^{13}C NMR (CDCl_3 , 150 MHz) δ 165.0, 136.7, 135.8, 130.7, 128.5, 127.2, 126.3, 82.9, 82.5, 70.2, 69.0, 58.0, 54.9, 52.8, 31.8, 25.7, 24.5, 21.6; HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{24}\text{NO}^+$ 270.1852, found 270.1851.



Tertiary amine 7k. A solution of piperidine **S9** (385 mg, 2.73 mmol, 1.3 equiv), Et_3N (2.83 mL, 20.3 mmol, 7.4 equiv), and 2-bromoethyl propargyl ether (342 mg, 2.10 mmol, 1.0 equiv) in CH_3CN (6.0 mL) was stirred at 50 $^\circ\text{C}$ for 22 h. After cooling to room temperature, saturated aqueous NaHCO_3 solution was added, and the resulting mixture was extracted with Et_2O . The extract was washed with brine, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. Flash chromatography on alumina (50% $\text{Et}_2\text{O}/n$ -hexane \rightarrow Et_2O) afforded tertiary amine **7k** (226 mg, 48%) as a colorless oil. IR (film) 3295, 2937, 2855, 2789, 1654, 1621, 1101 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 4.138 (1H, dd, $J = 16.0, 2.4$ Hz), 4.136 (1H, dd, $J = 16.0, 2.4$ Hz), 4.06 (1H, d, $J = 2.0$ Hz), 3.95 (1H, d, $J = 2.0$ Hz), 3.66 (1H, dt, $J = 9.6, 6.4$ Hz), 3.62 (1H, ddd, $J = 9.6, 6.8, 5.5$ Hz), 3.57 (3H, s), 3.10 (1H, dtd, $J = 10.9, 3.5, 1.0$ Hz), 2.97 (1H, dt, $J = 13.4, 6.8$ Hz), 2.64 (1H, dd, $J = 10.9, 2.8$ Hz), 2.41 (1H, t, $J = 2.4$ Hz), 2.35 (1H, ddd, $J = 13.4, 6.4, 5.7$ Hz), 2.10 (1H, m), 1.80–1.72 (2H, m), 1.65–1.57 (3H, m), 1.26 (1H, m); ^{13}C NMR (CDCl_3 , 150 MHz) δ 164.4, 82.8, 79.9, 74.2, 67.9, 67.6, 58.1, 54.9, 53.92, 53.88, 31.5, 25.7, 24.3; HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{22}\text{NO}_2^+$ 224.1645, found 224.1646.

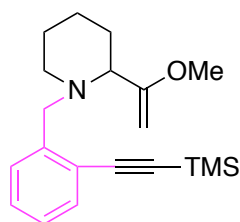


Tertiary amine 7m. A 10-mL, thick-walled reaction vessel was charged with a solution of piperidine **S9** (282 mg, 2.0 mmol, 1.0 equiv), Et₃N (1.39 mL, 10.0 mmol, 5.0 equiv), and 4-bromo-1-butyne (0.55 mL, 6.0 mmol, 3.0 equiv) in DMSO (0.5 mL) and CH₃CN (0.5 mL). The vessel was sealed and the reaction mixture was heated with a microwave reactor at 100 °C for 15 min. After cooling to room temperature, saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with Et₂O. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (10 → 80% Et₂O/*n*-hexane) afforded tertiary amine **7m** (132 mg, 34%) as colorless plates and recovered piperidine **S9** (90 mg, 32% recovery). Mp 91–92 °C; IR (KBr) 3193, 2951, 2940, 2926, 2107, 1626, 1302 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 4.08 (1H, d, *J* = 2.2 Hz), 3.97 (1H, d, *J* = 2.2, 0.9 Hz), 3.58 (3H, s), 3.01 (1H, dddd, *J* = 11.4, 3.9, 2.9, 1.5 Hz), 2.91 (1H, ddd, *J* = 13.2, 10.3, 5.9 Hz), 2.67 (1H, ddd, *J* = 10.8, 2.8, 0.9 Hz), 2.50 (1H, ddd, *J* = 13.2, 10.1, 5.3 Hz), 2.37 (1H, dddd, *J* = 16.3, 10.3, 5.3, 2.6 Hz), 2.33 (1H, dddd, *J* = 16.3, 10.1, 5.9, 2.6 Hz), 2.14 (1H, td, *J* = 11.2, 4.0 Hz), 1.94 (1H, t, *J* = 2.6 Hz), 1.77–1.70 (2H, m), 1.65–1.55 (3H, m), 1.25 (1H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 164.0, 83.3, 82.9, 68.7, 66.8, 55.0, 53.7, 52.7, 31.5, 25.6, 24.2, 15.1; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₂H₂₀NO⁺ 194.1539, found 194.1545.

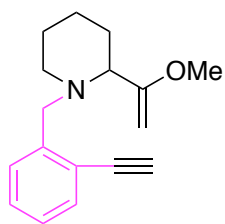


Tertiary amine S11. A solution of piperidine **S9** (220 mg, 1.56 mmol, 1.0 equiv), Et₃N (1.2 mL, 8.3 mmol, 5.0 equiv), and (5-iodo-1-pentynyl)benzene⁶ (635 mg, 2.35 mmol, 1.5 equiv) in CH₃CN (4.0 mL) was stirred at room temperature under dark condition. After 24 h, the reaction was quenched with saturated aqueous NaHCO₃ solution, and the resulting mixture was extracted with Et₂O. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography

on alumina (30% Et₂O/*n*-hexane) afforded tertiary amine **S11** (243 mg, 55%) as a pale yellow oil. IR (film) 2935, 2854, 2799, 2225, 1653 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.38–7.36 (2H, m), 7.28–7.23 (3H, m), 4.07 (1H, d, *J* = 2.0 Hz), 3.92 (1H, d, *J* = 2.0 Hz), 3.53 (3H, s), 3.06 (1H, dtd, *J* = 11.2, 3.4, 1.0 Hz), 2.84 (1H, ddd, *J* = 12.9, 10.2, 6.1 Hz), 2.62 (1H, dd, *J* = 10.8, 2.8 Hz), 2.37 (1H, dt, *J* = 16.9, 7.2 Hz), 2.36 (1H, dt, *J* = 16.9, 7.2 Hz), 2.24 (1H, ddd, *J* = 12.9, 9.8, 4.9 Hz), 2.01 (1H, m), 1.82–1.55 (7H, m), 1.26 (1H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 164.5, 131.4, 128.0, 127.3, 124.0, 90.0, 82.4, 80.5, 67.7, 54.8, 54.1, 52.8, 31.6, 25.7, 24.9, 24.3, 17.5; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₉H₂₆NO⁺ 284.2009, found 284.2008.

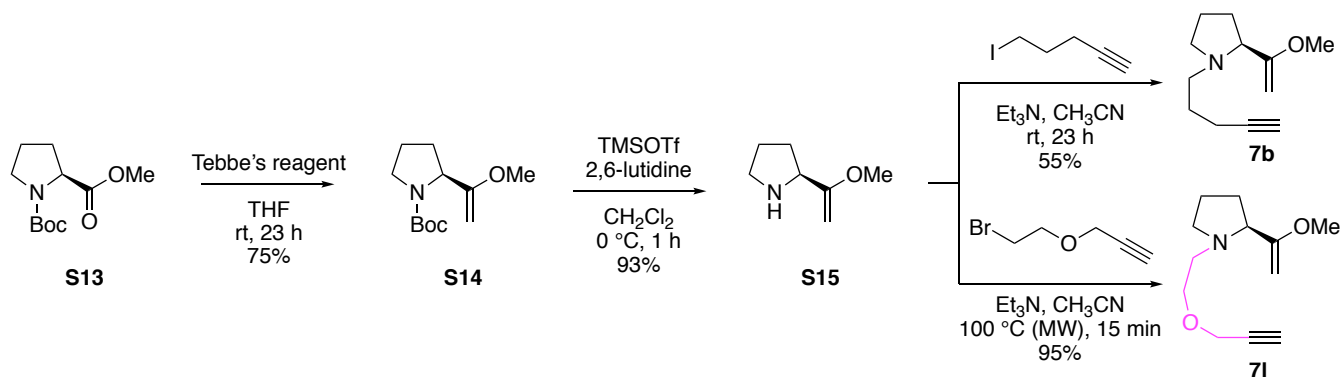


Tertiary amine S12. A solution of piperidine **S9** (413 mg, 2.91 mmol, 1.0 equiv), Et₃N (2 mL), and bromide **S6** (854 mg, 3.20 mmol, 1.1 equiv) in CH₃CN (2.0 mL) was stirred at room temperature. After 16 h, the mixture was warmed to 60 °C and then stirred for additional 3 h. After cooling to room temperature, saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with EtOAc. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (10% Et₂O/*n*-hexane) afforded tertiary amine **S12** (669 mg, 70%) as a pale yellow oil. IR (film) 2937, 2155, 1654, 1614, 1250, 869, 843 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.54 (1H, d, *J* = 7.7 Hz), 7.40 (1H, dd, *J* = 7.7, 1.3 Hz), 7.28 (1H, td, *J* = 7.6, 1.5 Hz), 7.14 (1H, td, *J* = 7.6, 1.0 Hz), 4.20 (1H, d, *J* = 2.0 Hz), 4.00 (1H, d, *J* = 2.0 Hz), 3.98 (1H, d, *J* = 13.9 Hz), 3.58 (3H, s), 3.46 (1H, d, *J* = 13.9 Hz), 2.92 (1H, dtd, *J* = 11.7, 3.4, 0.8 Hz), 2.74 (1H, dd, *J* = 10.2, 3.2 Hz), 1.96 (1H, m), 1.81–1.72 (3H, m), 1.53–1.48 (2H, m), 1.28 (1H, m), 0.26 (9H, s); ¹³C NMR (CDCl₃, 150 MHz) δ 165.3, 141.7, 132.0, 129.4, 128.6, 126.1, 122.9, 103.9, 98.3, 82.4, 68.0, 56.5, 54.9, 52.8, 32.1, 25.6, 24.5, 0.0; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₃₀NOSi⁺ 328.2091, found 328.2086.

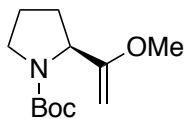


Terminal alkyne 7n. To a solution of TMS alkyne **S12** (308 mg, 0.940 mmol) in THF (9.4 mL) at $-40\text{ }^{\circ}\text{C}$ was added tetrabutylammonium fluoride (1.0 M solution in THF, 1.41 mL, 1.41 mmol, 1.5 equiv). The reaction mixture was stirred at $-40\text{ }^{\circ}\text{C}$ for 1 h, and the reaction was quenched with water. The resulting mixture was extracted with Et_2O , washed with brine, dried over Na_2SO_4 , filtered, and concentrated under reduced pressure. Flash chromatography on NH_2 silica gel (5% $\text{Et}_2\text{O}/n$ -hexane) afforded terminal alkyne **7n** (240 mg, 100%) as a colorless oil. IR (film) 3294, 2936, 1655, 1448, 1300, 1224 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 7.58 (1H, d, $J = 7.7$ Hz), 7.44 (1H, dd, $J = 7.7, 1.1$ Hz), 7.32 (1H, td, $J = 7.6, 1.4$ Hz), 7.16 (1H, td, $J = 7.6, 0.8$ Hz), 4.20 (1H, d, $J = 2.0$ Hz), 4.01 (1H, d, $J = 14.0$ Hz), 3.98 (1H, d, $J = 1.7$ Hz), 3.57 (3H, s), 3.44 (1H, d, $J = 14.0$ Hz), 3.24 (1H, s), 2.88 (1H, dt, $J = 11.5, 3.1$ Hz), 2.74 (1H, m), 1.96 (1H, m), 1.80–1.73 (3H, m), 1.53–1.46 (2H, m), 1.29 (1H, m); ^{13}C NMR (CDCl_3 , 150 MHz) δ 165.3, 142.1, 132.4, 129.3, 128.8, 126.2, 121.8, 82.28, 82.27, 81.2, 68.1, 56.6, 54.9, 52.9, 32.2, 25.6, 24.5; HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{22}\text{NO}^+$ 256.1696, found 256.1695.

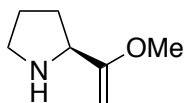
Synthesis of pyrrolidine **7b** and **7l**.



Scheme S3. Synthesis of pyrrolidine **7b** and **7l**.

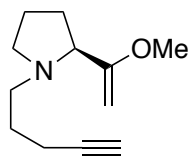


Vinyl ether S14. To a solution of *N*-Boc-L-Pro-OMe (**S13**) (2.60 mL, 2.78 g, 12.1 mmol, 1.0 equiv) in THF (38 mL) at 0 °C was added Tebbe reagent (0.5 M solution in toluene, 25 mL, 12.5 mmol, 1.03 equiv). The reaction mixture was stirred at room temperature for 23 h, and then the mixture was diluted with Et₂O (120 mL). 1 M NaOH (37.5 mL, 37.5 mmol, 3.0 equiv for Tebbe's reagent) was slowly added at room temperature to form orange precipitate. The resulting mixture was dried over Na₂SO₄, and the orange precipitate and Na₂SO₄ was removed by suction filtration. The filtrate was concentrated under reduced pressure and flash chromatography on alumina (30% Et₂O/*n*-hexane) afforded vinyl ether **S14** (2.07 g, 75%, 60:40 rotamers based on ¹H NMR analysis) as a pale yellow oil. [α]_D²⁶ −45.0 (*c* 0.97, CHCl₃); IR (film) 2975, 2878, 1696, 1390, 1163 cm^{−1}; ¹H NMR (CDCl₃, 600 MHz) δ 4.29 (0.4H, br s), 4.16 (0.6H, br s), 3.95 (0.8H, br s), 3.91 (1.2H, br s), 3.55 (3H, br s), 3.45–3.30 (2H, m), 2.02–1.85 (3H, m), 1.80–1.75 (1H, m), 1.46 (3.6H, br s), 1.42 (5.4H, br s); ¹³C NMR for the major rotamer (CDCl₃, 150 MHz) δ 164.1, 154.5, 112.0, 79.5, 59.6, 55.0, 46.3, 31.3, 28.4, 22.8; ¹³C NMR for the minor rotamer (CDCl₃, 150 MHz) δ 163.3, 154.4, 112.0, 79.2, 59.3, 55.0, 46.7, 30.3, 28.5, 23.5; HREIMS *m/z* M⁺ calcd for C₁₂H₂₁NO₃ 227.1521, found 227.1517.

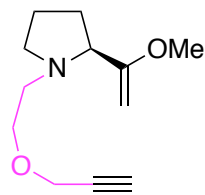


Pyrrolidine S15. To a solution of vinyl ether **S14** (1.012 g, 4.46 mmol) and 2,6-lutidine (1.02 mL, 8.92 mmol, 2.0 equiv) in CH₂Cl₂ (44 mL) at 0 °C was added TMSOTf (0.968 mL, 5.35 mmol, 1.2 equiv). The reaction mixture was stirred at 0 °C for 1 h, and the reaction was quenched with saturated aqueous NaHCO₃ solution. The resulting mixture was extracted with CH₂Cl₂, washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on alumina (0%→50%

MeOH/Et₂O) afforded pyrrolidine **S15** (32% solution in MeOH/Et₂O based on ¹H NMR analysis, 1.678 g, 93%). Because pyrrolidine **S15** was highly volatile and extremely stinky, we did not carried out further evaporation or distillation. **S15** was used as a MeOH/Et₂O solution in the next reaction. ¹H NMR (CDCl₃, 600 MHz) δ 4.09 (1H, d, *J* = 2.0 Hz), 3.92 (1H, d, *J* = 2.0 Hz), 3.58 (1H, t, *J* = 7.3 Hz), 3.56 (3H, s), 3.07 (1H, ddd, *J* = 10.2, 7.5, 5.2 Hz), 2.85 (1H, dt, *J* = 10.1, 7.1 Hz), 1.91 (1H, m), 1.80 (1H, m), 1.76–1.68 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 164.6, 80.4, 61.5, 54.9, 46.8, 30.3, 25.5; HRDARTMS *m/z* [M+H]⁺ calcd for C₇H₁₄NO⁺ 128.1070, found 128.1072. Specific optical rotation was not measured because **S15** was obtained as a MeOH/Et₂O solution as mention above.

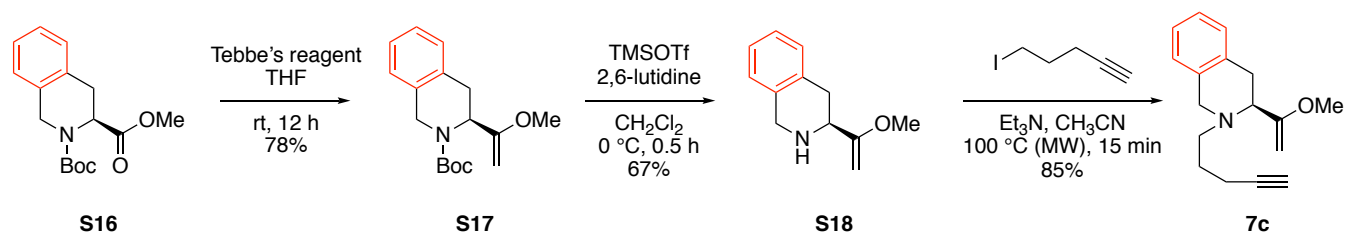


Tertiary amine 7b. A solution of pyrrolidine **S15** (32% solution in MeOH/Et₂O, 552.4 mg, 1.37 mmol), Et₃N (960 μL, 6.87 mmol, 5.0 equiv), and 5-iodo-1-pentyne (273 mg, 1.41 mmol, 1.02 equiv) in CH₃CN (1.3 mL) was stirred at room temperature for 23 h. The reaction was diluted with saturated aqueous NaHCO₃ solution, and the resulting mixture was extracted with Et₂O, washed with brine, dried, and concentrated under reduced pressure. Flash chromatography on alumina (20→50% Et₂O/*n*-hexane) afforded tertiary amine **7b** (145 mg, 55%) as a colorless oil; [α]²⁴_D -68.3 (*c* 0.89, CHCl₃); IR (film) 3303, 2948, 2799, 1657, 1620, 1236 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 4.11 (1H, d, *J* = 1.7 Hz), 3.96 (1H, d, *J* = 1.7 Hz), 3.57 (3H, s), 3.20 (1H, ddd, *J* = 8.8, 7.7, 2.4 Hz), 2.79 (1H, dt, *J* = 9.0, 8.1 Hz), 2.78 (1H, ddd, *J* = 12.1, 7.9, 4.2 Hz), 2.25 (1H, dtd, *J* = 16.9, 7.0, 2.6 Hz), 2.22 (1H, ddd, *J* = 12.1, 7.5, 6.6 Hz), 2.18 (1H, dtd, *J* = 16.9, 7.3, 2.6 Hz), 2.13 (1H, td, *J* = 9.0, 7.9 Hz), 1.93 (1H, t, *J* = 2.7 Hz), 1.93–1.79 (3H, m), 1.75–1.70 (3H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 163.9, 84.5, 82.1, 68.7, 68.1, 55.0, 53.8, 53.6, 29.7, 27.6, 22.5, 16.6; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₂H₂₀NO⁺ 194.1539, found 194.1540.

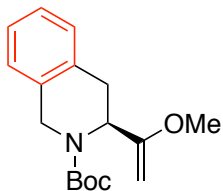


Tertiary amine 7l. A 10-mL reaction vessel was charged with a solution of pyrrolidine **S15** (32% solution in MeOH/Et₂O, 625 mg, 1.57 mmol), Et₃N (1.10 mL, 7.85 mmol, 5.0 equiv), and 2-bromoethyl propargyl ether (768 mg, 4.71 mmol, 3.0 equiv) in CH₃CN (3.0 mL). The vessel was sealed and the reaction mixture was heated with microwave reactor at 100 °C for 15 min. After cooling to room temperature, saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with EtOAc. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on alumina (15% Et₂O/n-hexane) afforded tertiary amine **7l** (313 mg, 95%) as a pale yellow oil. [α]_D²⁸ -59.4 (*c* 0.63, CHCl₃); IR (film) 3293, 2946, 2111, 1655, 1624, 1237 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 4.16 (2H, d, *J* = 2.4 Hz), 4.12 (1H, d, *J* = 2.0 Hz), 3.97 (1H, d, *J* = 1.8 Hz), 3.66 (1H, dt, *J* = 9.3, 6.2 Hz), 3.64 (1H, dt, *J* = 9.3, 6.2 Hz), 3.57 (3H, s), 3.25 (1H, ddd, *J* = 9.0, 7.7, 2.4 Hz), 2.99 (1H, dt, *J* = 12.7, 6.5 Hz), 2.83 (1H, t, *J* = 7.8 Hz), 2.41 (1H, t, *J* = 2.4 Hz), 2.40 (1H, dt, *J* = 12.7, 6.1 Hz), 2.23 (1H, td, *J* = 8.8, 8.1 Hz), 1.94–1.81 (3H, m), 1.74 (1H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 163.5, 82.5, 79.9, 74.2, 68.92, 68.91, 58.1, 55.0, 54.4, 53.4, 29.3, 22.6; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₂H₂₀NO₂⁺ 210.1489, found 210.1488.

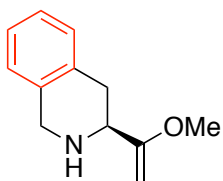
Synthesis of tetrahydroisoquinoline **7c**



Scheme S4. Synthesis of tetrahydroisoquinoline **7c**.

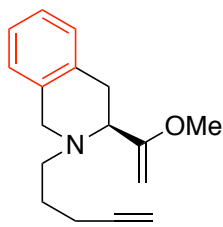


Vinyl ether S17. To a solution of *N*-Boc-L-Tic-OMe⁷ (**S16**) (901 mg, 3.09 mmol) in THF (13.5 mL) at 0 °C was added Tebbe reagent (0.5 M solution in toluene, 8.0 mL, 4.0 mmol, 1.3 equiv), and the mixture was stirred at room temperature for 12 h. Et₂O (10 mL) was added and the reaction was quenched with 10% aqueous NaOH (3 mL). The resulting orange precipitate was removed by suction filtration, and the residue was concentrated. Flash chromatography on alumina (20→30% Et₂O in *n*-hexane) afforded vinyl ether **S17** (697 mg, 78%) as a pale yellow oil; $[\alpha]_D^{23} +9.0$ (*c* 1.36, CHCl₃); IR (film) 2974, 1695, 1392, 1167 cm⁻¹; 56:44 rotamers (0 °C, CDCl₃); ¹H NMR for the major rotamer (CDCl₃, 600 MHz, 0 °C) δ 7.20–7.16 (2H, m), 7.14–7.09 (2H, m), 4.74 (1H, br s), 4.72 (1H, d, *J* = 16.1 Hz), 4.35 (1H, d, *J* = 16.1 Hz), 3.88 (1H, br s), 3.79 (1H, br s), 3.47 (3H, s), 3.12–3.02 (2H, m), 1.46 (9H, s); ¹H NMR for the minor rotamer (CDCl₃, 600 MHz, 0 °C) δ 7.20–7.16 (2H, m), 7.14–7.09 (2H, m), 5.04 (1H, br s), 4.69 (1H, d, *J* = 16.1 Hz), 4.35 (1H, d, *J* = 16.1 Hz), 3.91 (1H, br s), 3.79 (1H, br s), 3.47 (3H, s), 3.12–3.02 (2H, m), 1.52 (9H, s); ¹³C NMR (CDCl₃, 150 MHz, 0 °C) δ 162.1, 161.1, 155.3, 155.2, 133.9, 133.3, 133.12, 133.09, 128.5, 127.9, 126.52, 126.46, 126.2, 126.1, 126.0, 125.7, 81.2, 80.8, 80.1, 79.9, 55.0 (x2), 53.0, 51.2, 44.1, 43.6, 31.8, 31.0, 28.4, 28.3; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₇H₂₄NO₃⁺ 290.1751, found 290.1753.



Tetrahydroisoquinoline S18. To a solution of vinyl ether **S17** (690 mg, 2.38 mmol) and 2,6-lutidine (545 μL, 4.76 mmol, 2.0 equiv) in CH₂Cl₂ (24 mL) at 0 °C was added TMSOTf (517 μL, 2.86 mmol, 1.2 equiv), and the reaction mixture was stirred at 0 °C for 0.5 h. The reaction was quenched with saturated aqueous NaHCO₃ solution, and the resulting mixture was extracted with CH₂Cl₂. The extract was washed with brine, dried over K₂CO₃, filtered, and concentrated under reduced pressure. Flash chromatography on

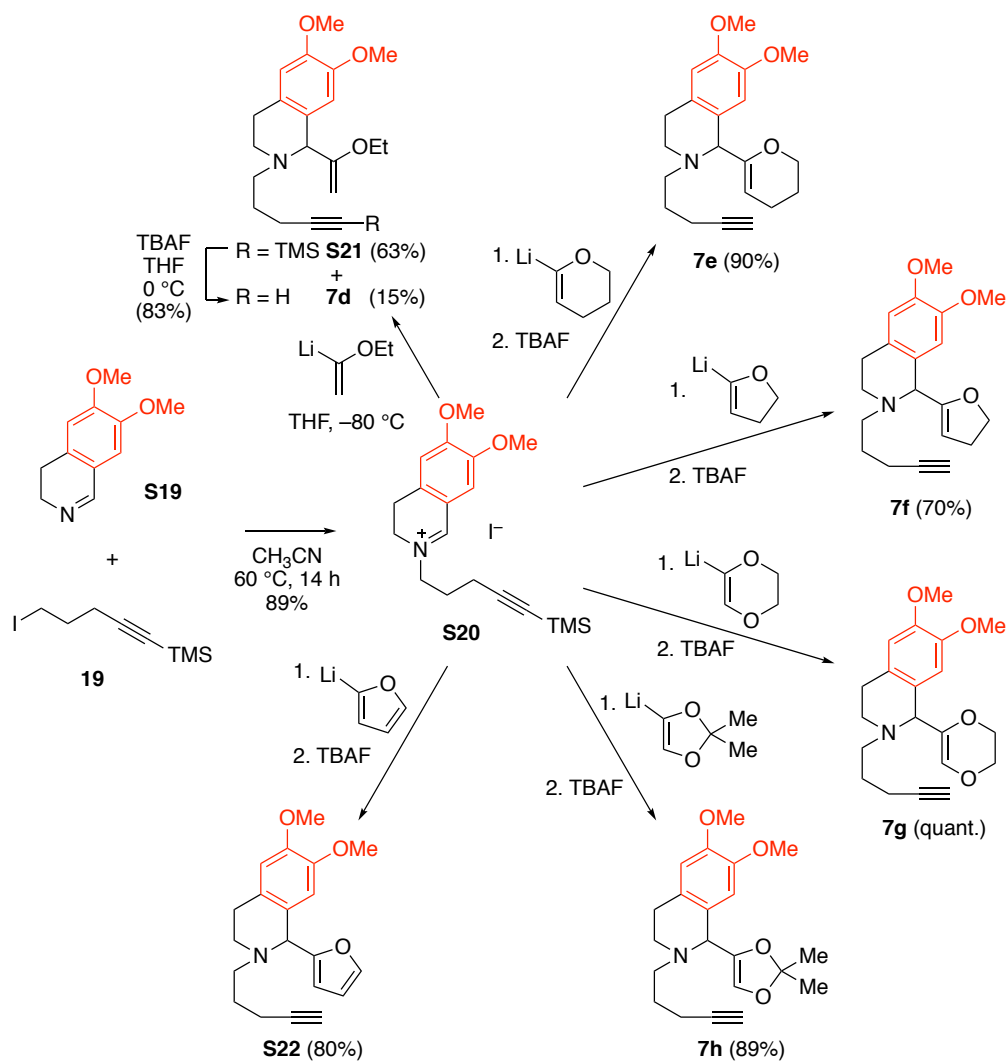
alumina (30% Et₂O/*n*-hexane→Et₂O→5% MeOH/Et₂O) afforded tetrahydroisoquinoline **S18** (300 mg, 67%) as a colorless oil. $[\alpha]_D^{24} -100.9$ (*c* 1.83, CHCl₃); IR (film) 3319, 2933, 2838, 1659, 1623, 1451, 1249, 1223 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.14–7.12 (2H, m), 7.09 (1H, m), 7.02 (1H, m), 4.16 (1H, d, *J* = 2.6 Hz), 4.12 (1H, d, *J* = 16.0 Hz), 4.09 (1H, d, *J* = 16.0 Hz), 4.02 (1H, d, *J* = 2.6 Hz), 3.61 (3H, s), 3.50 (1H, t, *J* = 7.3 Hz), 2.90 (1H, d, *J* = 7.3 Hz), 1.79 (1H, br s, NH); ¹³C NMR (CDCl₃, 150 MHz) δ 164.5, 135.2, 134.5, 129.2, 126.0, 125.9, 125.8, 80.5, 56.6, 54.9, 48.3, 33.2; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₂H₁₆NO⁺ 190.1226, found 190.1225.



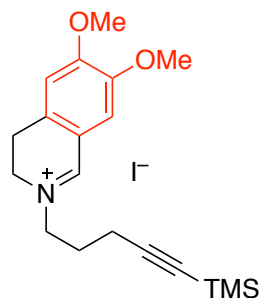
Tertiary amine 7c. A 10-mL, thick-walled reaction vessel was charged with a solution of 5-iodo-1-pentyne (886 mg, 4.57 mmol, 2.9 equiv), Et₃N (1.10 mL, 7.93 mmol, 5.0 equiv), and tetrahydroisoquinoline **S18** (300 mg, 1.59 mmol) in CH₃CN (1.6 mL). The vessel was sealed and the reaction mixture was heated at 100 °C for 15 min using a microwave reactor. After cooling to room temperature, saturated aqueous NaHCO₃ solution was added, and the resulting mixture was extracted with Et₂O. The extract was washed with brine, dried over K₂CO₃, filtered, and concentrated under reduced pressure. Flash chromatography on alumina gel (15%→20% Et₂O/*n*-hexane) afforded tertiary amine **7c** (345 mg, 85%) as a colorless oil. $[\alpha]_D^{24} -71.7$ (*c* 1.48, CHCl₃); IR (film) 3299, 2935, 2831, 2116, 1655, 1619, 1450, 1303, 1225, 1072 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 7.14–7.08 (2H, m), 7.07 (1H, m), 7.01 (1H, m), 4.09 (1H, d, *J* = 2.3 Hz), 4.05 (1H, d, *J* = 2.3 Hz), 3.96 (1H, d, *J* = 15.3 Hz), 3.60 (1H, d, *J* = 15.3 Hz), 3.58 (3H, s), 3.30 (1H, dd, *J* = 9.1, 4.8 Hz), 3.15 (1H, dd, *J* = 16.4, 8.8 Hz), 2.87 (1H, dd, *J* = 16.4, 4.5 Hz), 2.79 (1H, ddd, *J* = 12.5, 8.6, 6.4 Hz), 2.47 (1H, ddd, *J* = 12.6, 8.4, 6.0 Hz), 2.24 (1H, dtd, *J* = 16.7, 7.2, 2.7 Hz), 2.23 (1H, dtd, *J* = 16.7, 7.2, 2.7 Hz), 1.93 (1H, t, *J* = 2.7 Hz), 1.85–1.73 (2H, m);

^{13}C NMR (CDCl_3 , 125 MHz) δ 162.8, 134.3, 134.1, 128.2, 126.2, 126.1, 125.6, 84.5, 83.5, 68.2, 62.7, 54.8, 53.7, 52.6, 32.7, 26.1, 16.4; HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{22}\text{NO}^+$ 256.1696, found 256.1701.

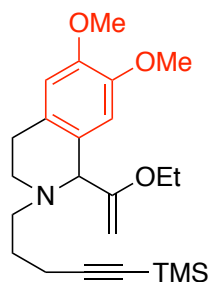
Synthesis of tetrahydroisoquinolines 7d–h, S21 and S22



Scheme S5. Synthesis of tetrahydroisoquinolines 7d–h, S21 and S22.



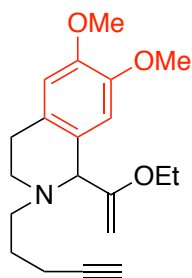
Iminium iodide S20. A mixture of 6,7-dimethoxy-3,4-dihydroisoquinoline hydrochloride (5.01 g, 22.0 mmol) and 10% aqueous K₂CO₃ solution (100 mL, 72.5 mmol, 3.3 equiv) was extracted with Et₂O. The extract was dried over K₂CO₃, filtered, and concentrated under reduced pressure to afford 6,7-dimethoxy-3,4-dihydroisoquinoline (**S19**) (4.12 g) as a yellow oil, which was used in the next alkylation without further purification. A solution of 6,7-dimethoxy-3,4-dihydroisoquinoline (**S19**) (4.12 g, 21.5 mmol) and (5-iodo-1-pentynyl)trimethylsilane⁸ (**19**) (7.29 g, 27.4 mmol, 1.27 equiv) in CH₃CN (50 mL) was stirred at 60 °C for 14 h. After cooling to room temperature, Et₂O (50 mL) was added. Resulting precipitation was collected with suction filtration to afford **S20** (9.00 g, 89%) as a pale yellow powder. Mp 166–167 °C; IR (KBr) 3011, 2958, 2173, 1659 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 9.88 (1H, s), 7.64 (1H, s), 6.90 (1H, s), 4.33 (2H, t, *J* = 6.9 Hz), 4.06 (2H, t, *J* = 8.3 Hz), 4.02 (3H, s), 3.94 (3H, s), 3.31 (2H, t, *J* = 8.3 Hz), 2.50 (2H, t, *J* = 6.6 Hz), 2.16 (2H, quintet, *J* = 6.7 Hz), 0.06 (9H, s); ¹³C NMR (CDCl₃, 150 MHz) δ 164.7, 157.6, 148.9, 132.2, 117.1, 115.7, 110.7, 104.3, 87.0, 59.5, 56.9, 56.7, 48.4, 25.8, 25.7, 17.1, -0.1; Anal Calcd for C₁₉H₂₈INO₂Si: C, 49.89; H, 6.17; N, 3.06. Found: C, 49.60; H, 5.97; N, 3.02.



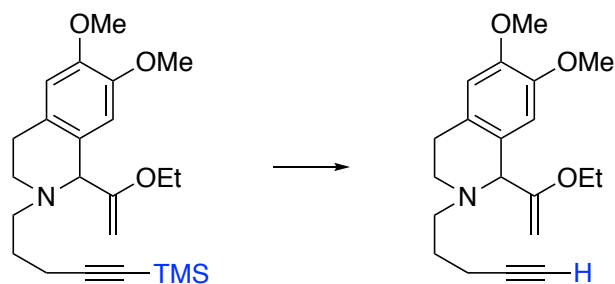
Addition of (1-ethoxyvinyl)lithium to iminium S20. To a solution of ethyl vinyl ether (0.48 mL, 5.0 mmol, 1.0 equiv) in THF (5 mL) at -80 °C was added *tert*-butyllithium (1.61 M solution in *n*-pentane, 3.11 mL, 5.0 mmol, 5.0 equiv). The reaction mixture was stirred at -80 °C for 2 h, and then warmed to roomed temperature. The resulting yellow solution of (1-ethoxyvinyl)lithium was added to a suspension of cyclic iminium **S20** (457 mg, 1.0 mmol, 1.0 equiv) in THF (5 mL) at -80 °C. The reaction mixture was warmed to 0 °C and stirred at 0 °C for 50 min. The reaction was quenched with brine, and the resulting mixture was extracted with Et₂O. The organic layer was dried over Na₂SO₄, filtered, and concentrated

under reduced pressure. Flash chromatography on NH₂ silica (5→20% Et₂O in *n*-hexane) afforded TMS alkyne **S21** (255 mg, 63%) and terminal alkyne **7d** (50 mg, 15%).

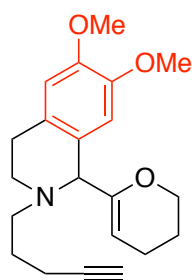
TMS alkyne S21. Colorless solid; Mp 50–52 °C; IR (film) 2954, 2924, 2853, 2172, 1516, 1250, 1225, 842 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.71 (1H, s), 6.56 (1H, s), 4.11 (1H, d, *J* = 1.7 Hz), 4.05 (1H, d, *J* = 1.7 Hz), 4.03 (1H, s), 3.84 (3H, s), 3.80 (3H, s), 3.80 (1H, dq, *J* = 9.9, 7.0 Hz), 3.73 (1H, dq, *J* = 9.9, 7.0 Hz), 3.22 (1H, ddd, *J* = 11.6, 6.5, 5.0 Hz), 2.84–2.71 (3H, m), 2.62–2.57 (2H, m), 2.30 (1H, dt, *J* = 17.1, 7.1 Hz), 2.27 (1H, dt, *J* = 17.1, 7.1 Hz), 1.81–1.71 (2H, m), 1.26 (3H, t, *J* = 7.1 Hz), 0.14 (9H, s); ¹³C NMR (CDCl₃, 150 MHz) δ 163.1, 147.5, 146.9, 127.7, 127.1, 110.9, 110.3, 107.7, 85.5, 84.3, 66.4, 62.9, 55.83, 55.77, 53.3, 46.6, 28.1, 26.2, 17.8, 14.3, 0.2; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₃H₃₆NO₃Si⁺ 402.2459, found 402.2459.



Terminal alkyne 7d. Colorless solid; Mp 56–57 °C; IR (film) 3287, 2933, 2832, 2116, 1612, 1516, 1256, 1224 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.70 (1H, s), 6.56 (1H, s), 4.12 (1H, d, *J* = 1.8 Hz), 4.03 (1H, s), 4.03 (1H, d, *J* = 1.8 Hz), 3.84 (3H, s), 3.80 (3H, s), 3.80 (1H, dq, *J* = 9.7, 7.0 Hz), 3.73 (1H, dq, *J* = 9.7, 7.0 Hz), 3.22 (1H, ddd, *J* = 11.7, 6.7, 5.0 Hz), 2.82–2.71 (3H, m), 2.62–2.57 (2H, m), 2.28 (1H, dtd, *J* = 16.9, 7.1, 2.6 Hz), 2.24 (1H, dtd, *J* = 16.9, 7.1, 2.6 Hz), 1.93 (1H, t, *J* = 2.6 Hz), 1.83–1.73 (2H, m), 1.26 (3H, t, *J* = 7.1 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ 163.1, 147.5, 146.9, 127.6, 127.1, 110.9, 110.3, 85.6, 84.7, 68.0, 66.3, 62.9, 55.82, 55.76, 53.2, 46.6, 27.9, 26.3, 16.3, 14.3; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₂₈NO₃⁺ 330.2064, found 330.2063.

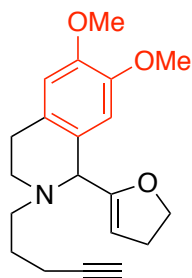


Deprotection of TMS group from S21 to 7d. To a solution of TMS alkyne **S21** (226 mg, 0.562 mmol, 1.0 equiv) in THF (5.6 mL) at 0 °C was added tetra-*n*-butylammonium fluoride (1 M solution in THF, 0.674 mL, 0.674 mmol, 1.2 equiv). The reaction mixture was stirred at 0 °C for 1 h, and the reaction was quenched with water. The resulting mixture was extracted with Et₂O, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (10% Et₂O in *n*-hexane) afforded **7d** (153 mg, 83%).



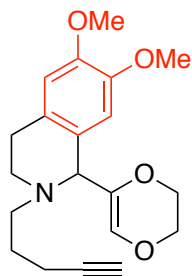
Dihydropyran 7e. To a solution of 3,4-dihydro-2*H*-pyran (0.42 mL, 5.0 mmol, 5.0 equiv) in THF (5 mL) at –80 °C was added *tert*-butyllithium (1.61 M solution in *n*-pentane, 3.11 mL, 5.0 mmol, 5.0 equiv). The reaction mixture was stirred at –80 °C for 2 h, and then warmed to room temperature. The resulting yellow solution of (3,4-dihydro-2*H*-pyran-6-yl)lithium was added to a suspension of cyclic iminium **S20** (457 mg, 1.0 mmol, 1.0 equiv) in THF (5 mL) at –80 °C. The reaction mixture was warmed to 0 °C and stirred at 0 °C for 50 min. The reaction was quenched with brine, and the resulting mixture was extracted with Et₂O. The organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford DHP adduct (471 mg), which was used in the desilylation step without further purification. To a solution of the DHP adduct in THF (11.2 mL) at 0 °C was added tetra-*n*-butylammonium fluoride (1 M solution in THF, 1.12 mL, 1.12 mmol, 1.1 equiv). The reaction mixture was stirred at 0 °C for 50 min,

and the reaction was quenched with water. The resulting mixture was extracted with Et₂O, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (5→20% Et₂O in *n*-hexane) afforded dihydropyran **7e** (308 mg, 90%) as a colorless solid. Mp 95–96 °C; IR (film) 3284, 2931, 2846, 1666, 1516, 1255, 1223 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.73 (1H, s), 6.56 (1H, s), 4.75 (1H, t, *J* = 3.8 Hz), 3.99 (1H, ddd, *J* = 10.5, 7.3, 3.3 Hz), 3.93 (1H, ddd, *J* = 10.6, 6.8, 3.1 Hz), 3.87 (1H, s), 3.84 (3H, s), 3.81 (3H, s), 3.20 (1H, dt, *J* = 11.7, 5.1 Hz), 2.90–2.85 (2H, m), 2.68 (1H, dt, *J* = 16.0, 4.8 Hz), 2.57–2.53 (2H, m), 2.28 (1H, dtd, *J* = 16.9, 7.0, 2.6 Hz), 2.23 (1H, dtd, *J* = 16.9, 7.2, 2.6 Hz), 2.11–2.08 (2H, m), 1.94 (1H, t, *J* = 2.6 Hz), 1.85–1.75 (4H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 154.0, 147.5, 147.0, 127.5, 127.2, 111.0, 110.2, 101.4, 84.7, 68.1, 66.8, 66.3, 55.9, 55.7, 53.2, 47.5, 28.2, 25.8, 22.3, 20.5, 16.4; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₁H₂₈NO₃⁺ 342.2064, found 342.2074.



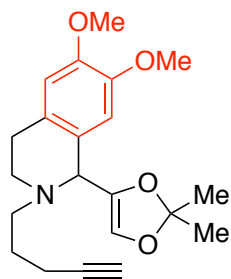
Dihydrofuran 7f. To a solution of 2,3-dihydrofuran (0.38 mL, 5.0 mmol, 5.0 equiv) in THF (5 mL) at –80 °C was added *tert*-butyllithium (1.61 M solution in *n*-pentane, 3.11 mL, 5.0 mmol, 5.0 equiv). The reaction mixture was stirred at –80 °C for 2 h, and then warmed to roomed temperature. The resulting yellow solution of (4,5-dihydrofuran-2-yl)lithium was added to a suspension of cyclic iminium **S20** (457 mg, 1.0 mmol, 1.0 equiv) in THF (5 mL) at –80 °C. The reaction mixture was warmed to 0 °C and stirred at 0 °C for 80 min. The reaction was quenched with brine, and the resulting mixture was extracted with Et₂O. The organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford DHF adduct (384 mg), which was used in the desilylation step without further purification. To a solution of the DHF adduct in THF (9.6 mL) at 0 °C was added tetra-*n*-butylammonium fluoride (1 M solution in

THF, 0.96 mL, 0.96 mmol, 1.1 equiv). The reaction mixture was stirred at 0 °C for 50 min, and the reaction was quenched with water. The resulting mixture was extracted with Et₂O, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (5→30% Et₂O in *n*-hexane) afforded dihydrofuran **7f** (228 mg, 70%) as a pale yellow solid. Mp 58–60 °C; IR (film) 3286, 2931, 1517, 1254, 1135 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.65 (1H, s), 6.57 (1H, s), 4.74 (1H, t, *J* = 2.4 Hz), 4.35 (1H, dt, *J* = 9.8, 9.0 Hz), 4.32 (1H, dt, *J* = 9.8, 8.8 Hz), 4.23 (1H, s), 3.84 (3H, s), 3.82 (3H, s), 3.18 (1H, ddd, *J* = 12.0, 7.2, 5.2 Hz), 2.81–2.71 (3H, m), 2.69–2.60 (4H, m), 2.27 (1H, dtd, *J* = 16.9, 7.1, 2.8 Hz), 2.26 (1H, dtd, *J* = 16.9, 7.1, 2.8 Hz), 1.93 (1H, t, *J* = 2.7 Hz), 1.82–1.75 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 158.7, 147.8, 147.0, 127.0, 126.6, 111.1, 110.5, 99.7, 84.6, 70.2, 68.1, 59.9, 55.9, 55.8, 53.0, 46.2, 29.7, 27.3, 26.3, 16.3; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₂₆NO₃⁺ 328.1907, found 328.1905.



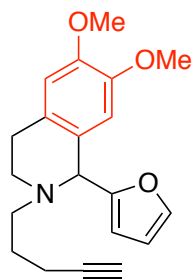
Dioxene 7g. To a solution of 1,4-dioxene (0.40 mL, 5.0 mmol, 1.0 equiv) in THF (5 mL) at –80 °C was added *tert*-butyllithium (1.61 M solution in *n*-pentane, 3.11 mL, 5.0 mmol, 1.0 equiv). The reaction mixture was stirred at –80 °C for 10 min, and then warmed to 0 °C. After stirring for 5 min at 0 °C, the resulting solution of (5,6-dihydro-1,4-dioxin-2-yl)lithium was added to a suspension of cyclic iminium **S20** (457 mg, 1.0 mmol, 1.0 equiv) in THF (5 mL) at –80 °C. The reaction mixture was warmed to 0 °C and stirred at 0 °C for 2 h. The reaction was quenched with brine, and the resulting mixture was extracted with Et₂O. The organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford 1,4-dioxene adduct (585 mg), which was used in the next desilylation step without further purification. To a solution of the 1,4-dioxene adduct in THF (14 mL) at 0 °C was added tetra-*n*-

butylammonium fluoride (1 M solution in THF, 1.41 mL, 1.41 mmol, 1.41 equiv). The reaction mixture was stirred at 0 °C for 50 min, and the reaction was quenched with water. The resulting mixture was extracted with Et₂O, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (5→30% Et₂O in *n*-hexane) afforded dioxane **7g** (343 mg, quantitative yield) as a pale yellow solid. Mp 110–113 °C; IR (film) 3284, 2933, 1676, 1517, 1255, 1137 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.74 (1H, s), 6.57 (1H, s), 6.02 (1H, s), 4.12–4.05 (2H, m), 4.02–3.99 (2H, m), 3.84 (3H, s), 3.83 (3H, s), 3.81 (1H, s), 3.21 (1H, t, *J* = 11.7, 5.3 Hz), 2.91 (1H, dt, *J* = 13.1, 7.5 Hz), 2.86 (1H, ddd, *J* = 15.9, 8.1, 5.2 Hz), 2.71 (1H, dt, *J* = 15.9, 5.2 Hz), 2.60–2.55 (2H, m), 2.27 (1H, dtd, *J* = 16.9, 7.0, 2.6 Hz), 2.26 (1H, dtd, *J* = 16.9, 7.0, 2.6 Hz), 1.95 (1H, t, *J* = 2.6 Hz), 1.80–1.75 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 147.7, 147.2, 136.9, 127.7, 126.65, 126.59, 111.1, 110.1, 84.6, 68.2, 64.5, 64.2, 63.3, 56.0, 55.8, 52.9, 47.2, 28.1, 25.9, 16.4; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₂₆NO₄⁺ 344.1856, found 344.1859.



Dimethyldioxolene 7h. To a solution of 2,2-dimethyl-1,3-dioxolene⁹ (994 mg, 9.93 mmol, 1.0 equiv) in THF (100 mL) at –80 °C was added *tert*-butyllithium (1.61 M solution in *n*-pentane, 6.78 mL, 10.9 mmol, 1.1 equiv). The reaction mixture was stirred at –80 °C for 1 h. A powder of cyclic iminium **S20** (4.77 mg, 10.4 mmol, 1.05 equiv) was added at –80 °C under argon stream, and the reaction mixture was stirred at –80 °C for 6.5 h. The reaction was quenched with brine, and THF was removed by rotary evaporator. The resulting mixture was extracted with Et₂O, and the organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford dimethyldioxolene adduct (4.77 g), which was used in the desilylation step without further purification. To a solution of the dimethyldioxolene adduct in THF (90

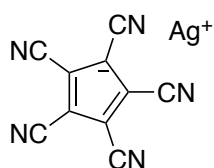
mL) at 0 °C was added tetra-*n*-butylammonium fluoride (1 M solution in THF, 12.0 mL, 12.0 mmol, 1.2 equiv). The reaction mixture was stirred at 0 °C for 30 min, and the reaction was quenched with water. THF was removed by rotary evaporator, and the resulting mixture was extracted with Et₂O. The organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on alumina (CH₂Cl₂) afforded dimethyldioxolene **7h** (3.16 g, 89%) as a pale yellow needles; Mp 94–96 °C; IR (film) 3283, 2934, 2832, 2114, 1516, 1254, 1224, 1139 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.70 (1H, s), 6.57 (1H, s), 5.97 (1H, s), 4.22 (1H, s), 3.85 (3H, s), 3.83 (3H, s), 3.14 (1H, ddd, *J* = 11.9, 7.0, 5.1 Hz), 2.84 (1H, ddd, *J* = 12.7, 8.1, 6.4 Hz), 2.82–2.74 (2H, m), 2.67–2.61 (2H, m), 2.28 (1H, dtd, *J* = 17.1, 7.3, 2.7 Hz), 2.25 (1H, dtd, *J* = 17.1, 7.0, 2.7 Hz), 1.94 (1H, t, *J* = 2.7 Hz), 1.83–1.75 (2H, m), 1.56 (3H, s), 1.45 (3H, s); ¹³C NMR (CDCl₃, 150 MHz) δ 147.9, 147.1, 138.7, 127.4, 126.0, 125.8, 114.7, 111.1, 110.3, 84.5, 68.3, 57.9, 55.9, 55.8, 53.2, 46.5, 27.9, 26.3, 25.6, 25.2, 16.4; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₁H₂₈NO₄⁺ 358.2013, found 358.2012.



Furan S22. To a solution of furan (0.150 mL, 140 mg, 2.06 mmol, 1.0 equiv) in THF (20 mL) at 0 °C was added *n*-butyllithium (1.59 M solution in *n*-hexane, 1.30 mL, 2.06 mmol, 1.0 equiv). The reaction mixture was stirred at 0 °C for 1 h and then cooled to –25 °C. A powder of cyclic iminium **S20** (943 mg, 2.06 mmol, 1.0 equiv) was added at –25 °C under argon stream, and the reaction mixture was stirred at –25 °C for 1.5 h. The reaction was quenched with brine, and THF was removed by rotary evaporator. The resulting mixture was extracted with Et₂O, and the organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford furan adduct (828 mg), which was used in the desilylation step without further purification. To a solution of the furan adduct in THF (10 mL) at 0 °C was added

tetra-*n*-butylammonium fluoride (1 M solution in THF, 2.06 mL, 2.06 mmol, 1.0 equiv). The reaction mixture was stirred at 0 °C for 10 min, and the reaction was quenched with water. THF was removed by rotary evaporator, and the resulting mixture was extracted with Et₂O. The organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (5→30% Et₂O in *n*-hexane) afforded furan **S22** (535 mg, 80%) as a yellow oil. IR (film) 3290, 2950, 2934, 2911, 2832, 1517 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.36 (1H, dd, *J* = 1.8, 0.7 Hz), 6.61 (1H, s), 6.40 (1H, s), 6.29 (1H, dd, *J* = 3.1, 1.8 Hz), 6.02 (1H, d, *J* = 3.1 Hz), 4.79 (1H, s), 3.85 (3H, s), 3.73 (3H, s), 3.04 (1H, ddd, *J* = 12.1, 8.5, 4.8 Hz), 2.88 (1H, ddd, *J* = 16.3, 8.5, 5.6 Hz), 2.78 (1H, dt, *J* = 16.3, 4.8 Hz), 2.71 (1H, dt, *J* = 11.8, 5.3 Hz), 2.61 (1H, ddd, *J* = 12.8, 8.1, 6.2 Hz), 2.56 (1H, ddd, *J* = 12.8, 8.1, 6.2 Hz), 2.26 (1H, dtd, *J* = 16.9, 7.2, 2.6 Hz), 2.21 (1H, dtd, *J* = 16.9, 7.2, 2.6 Hz), 1.92 (1H, t, *J* = 2.7 Hz), 1.79 (1H, d-quintet-d, *J* = 13.9, 7.2, 6.8 Hz), 1.77 (1H, d-quintet-d, *J* = 13.9, 7.2, 6.8 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ 155.6, 147.8, 147.1, 142.0, 127.0, 126.9, 111.1, 110.8, 109.8, 109.4, 84.4, 68.2, 59.7, 55.84, 55.78, 53.1, 45.7, 27.5, 26.4, 16.3; HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₂₄NO₃⁺ 326.1751, found 326.1751.

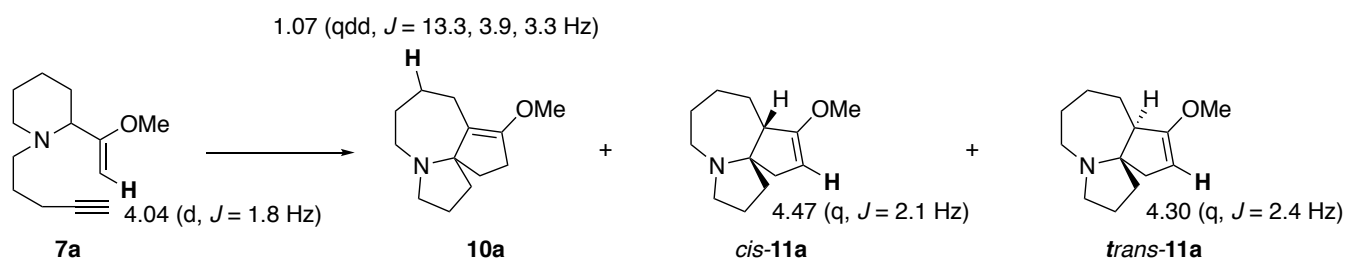
Preparation of Silver pentacyanocyclopentadienide



Silver pentacyanocyclopentadienide (Ag[C₅(CN)₅]). To a solution of Na[C₅(CN)₅]¹⁰ (5.68 g, 26.7 mmol) in H₂O (120 mL) was added Et₄NBr (7.0 g, 33 mmol, 1.2 equiv). Resulting precipitate was collected by filtration, washed with H₂O, and dried under reduced pressure to afford Et₄N[C₅(CN)₅] (8.22 g) as a beige powder, which was used in the next step without further purification. To a solution of Et₄N[C₅(CN)₅] (8.22 g) in acetone (250 mL) under dark condition was added a solution of AgNO₃ (13.1 g, 77.0 mmol, 3.0 equiv) in H₂O (50 mL). The solution was stirred at room temperature for 24 h under dark condition, and the resulting precipitate was collected by filtration, washed well with water, MeOH,

and Et₂O, and dried under reduced pressure to afford Ag[C₅(CN)₅] (6.37 g, 83%) as a beige powder. IR (KBr pellet) 2250, 2221, 1466 cm⁻¹; ¹³C NMR (CD₃CN, 600 MHz) δ 114.3, 103.3; Anal calcd for C₁₀N₅Ag: C, 40.30; H, 0.00; N, 23.50. Found: C, 39.97; H, 0.31; N, 23.20. Similar procedure was reported by Williams et al., in which elemental analysis showed Ag[C₅(CN)₅] with hydration waters.¹¹ Elemental analysis in our experiment supported the non-hydrated salt.

Reaction condition screening in Figure 1



To a solution of amine **7a** (10 mg, 0.048 mmol, 1.0 equiv) in CD₂Cl₂ (0.6 mL) in a NMR tube was added 1,2-dichloroethane (1 μL), and a ¹H NMR spectrum was collected for determination of signal ratio between amine **7a** (4.04 ppm) and 1,2-dichloroethane (3.76 ppm, internal standard). Au catalyst (0.008 mmol, 20 mol%) and Ag salt (0.010 mmol, 25 mol%) were added and the reaction mixture was heated under dark condition at 40 °C for the indicated time in Figure 1. After cooling to room temperature, a ¹H NMR spectrum was collected and product yields were determined based on the integration of 1.07 ppm (qdd, *J* = 13.3, 3.9, 3.3 Hz) for **10a**, 4.47 ppm (q, *J* = 2.1 Hz) for *cis*-**11a**, and 4.30 ppm (q, *J* = 2.4 Hz) for *trans*-**11a**.

Scope and Limitation of the cascade reaction

Table S1. Scope of the cascade reaction

entry	substrate	conditions	product(s)	total yield of products
1		Au 1 mol% Ag 1 mol% 80 °C 24 h		78% 13% 15%
2 ^a		Au 5 mol% Ag 5 mol% 80 °C 17 h		65% 44%
3 ^a		Au 1 mol% Ag 1 mol% 80 °C 24 h		73% 17% 22%
4		Au 1 mol% Ag 1 mol% 80 °C 24 h		60% 38%
5		Au 2 mol% Ag 2 mol% 80 °C 5 h		63% 63%
6		Au 2.5 mol% Ag 2.5 mol% 80 °C 5 h		58% 16%
7		Au 1.5 mol% Ag 1.5 mol% 80 °C 19 h		73% 38% 9% 26%

entry	substrate	conditions	product(s)	total yield of products
8		Au 2 mol% Ag 2 mol% 80 °C 27 h		7%
9		Au 1 mol% Ag 1 mol% Et ₃ N 80 °C 24 h		73% 73%
10		Au 4 mol% Ag 4 mol% 80 °C 48 h		34% 29% 5%
11		Au 2 mol% Ag 2 mol% 130 °C 0.5 h (Microwave)		16%, 46% brsm 9% (26% brsm) 7% (20% brsm)
12		Au 5 mol% Ag 5 mol% 120 °C 44 h (in C ₆ H ₅ Cl)		30% 30%
13 ^a		Au 5 mol% Ag 5 mol% 80 °C 22 h		21% 21%
14		Au 2 mol% Ag 2 mol% 80 °C 26 h		36%, 50% brsm 15% (21% brsm) 21% (29% brsm)
15		Au 1 mol% Ag 1 mol% 80 °C 2.5 h		56% 22% 34%
<p>For entries 14 and 15</p>				

^a S-Enantiomers were used in the reactions shown in entries 2, 3, and 13. The specific optical rotations of products **10b-c**, **11b**, *trans*- and *cis*-**11c**, and **11l** were very low.

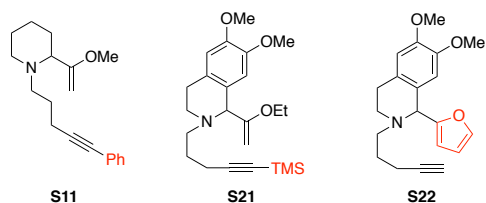
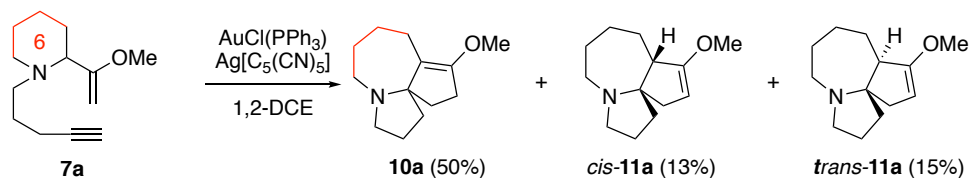


Figure S1. Substrates that did not give products.

As a limitation of this reaction, substrate **S11** or **S21** bearing a phenyl or trimethylsilyl group on the alkyne terminal did not afford any product derived from the cascade reaction. Furan **S22** did not give a product possibly because of its low reactivity during the 3-aza-Cope rearrangement stage.

Experimental procedure for the cascade reaction (Figure 2)



Cyclization reaction with 7a. To a solution of tertiary amine **7a** (98 mg, 0.47 mmol, 1.0 equiv) in 1,2-dichloroethane (5 mL) were added AuCl(PPh₃) (2.5 mg, 0.005 mmol, 1 mol%) and Ag[C₅(CN)₅] (1.5 mg, 0.005 mmol, 1 mol%). The reaction mixture was stirred at 80 °C under dark condition for 23 h. After cooling to room temperature, flash chromatography on NH₂ silica gel (20→30% Et₂O in *n*-hexane) gave a mixture of **10a** and *cis*-**11a** (67 mg) and a mixture including *trans*-**11a** (18 mg). The former mixture was further purified by flash chromatography on alumina (20% Et₂O in *n*-hexane → Et₂O) to afford **10a** (49 mg, 50%) and *cis*-**11a** (13 mg, 13%). The latter mixture was further purified by flash chromatography on alumina (20% Et₂O in *n*-hexane → Et₂O) to give *trans*-**11a** (15 mg, 15%).

Internal enol ether 10a. Pale yellow oil; IR (film) 2922, 2846, 1684 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 3.59 (3H, s), 3.09 (1H, ddd, *J* = 9.9, 8.1, 6.2 Hz), 3.06 (1H, ddt, *J* = 14.8, 4.8, 1.3 Hz), 2.89 (1H, ddd, *J* = 14.8, 11.9, 1.2 Hz), 2.74 (1H, m), 2.57 (1H, ddt, *J* = 13.3, 4.1, 1.9 Hz), 2.36 (1H, dd, *J* = 14.9, 9.2 Hz), 2.25 (1H, dddd, *J* = 14.9, 9.4, 7.4, 2.1 Hz), 1.86–1.79 (3H, m), 1.78–1.73 (3H, m), 1.67 (1H, dt, *J* = 12.1, 9.4 Hz), 1.58 (1H, m), 1.46 (1H, tt, *J* = 13.3, 2.3 Hz), 1.38 (1H, m), 1.11 (1H, qdd, *J* = 13.3, 3.9, 3.1 Hz);

^{13}C NMR (CDCl_3 , 600 MHz) δ 150.8 (C), 122.9 (C), 73.3 (C), 56.0 (CH_3), 50.0 (CH_2), 46.7 (CH_2), 37.6 (CH_2), 36.6 (CH_2), 29.8 (CH_2), 26.3 (CH_2), 24.7 (CH_2), 24.2 (CH_2), 23.1 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{22}\text{NO}^+$ 208.1696, found 208.1697.

Cis-fused compound *cis*-11a. Pale yellow oil; IR (film) 2923, 2849, 1653 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 4.46 (1H, q, $J = 2.1$ Hz), 3.60 (3H, s), 3.05 (1H, ddd, $J = 9.5, 8.6, 3.5$ Hz), 2.71 (1H, m), 2.68 (1H, dtd, $J = 13.5, 4.0, 1.5$ Hz), 2.65 (1H, td, $J = 9.7, 6.2$ Hz), 2.50 (1H, dt, $J = 15.4, 2.2$ Hz), 2.43 (1H, ddd, $J = 13.5, 9.0, 5.0$ Hz), 1.96 (1H, dd, $J = 15.2, 2.2$ Hz), 1.87–1.80 (4H, m), 1.70 (1H, m), 1.63 (1H, dddd, $J = 14.0, 11.6, 3.9, 1.3$ Hz), 1.57–1.50 (3H, m), 1.24 (1H, m); ^{13}C NMR (CDCl_3 , 600 MHz) δ 160.0 (C), 91.5 (CH), 70.7 (C), 56.0 (CH_3), 53.8 (CH_2), 52.4 (CH), 49.2 (CH_2), 42.3 (CH_2), 33.8 (CH_2), 30.8 (CH_2), 27.1 (CH_2), 24.4 (CH_2), 20.4 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{22}\text{NO}^+$ 208.1696, found 208.1696.

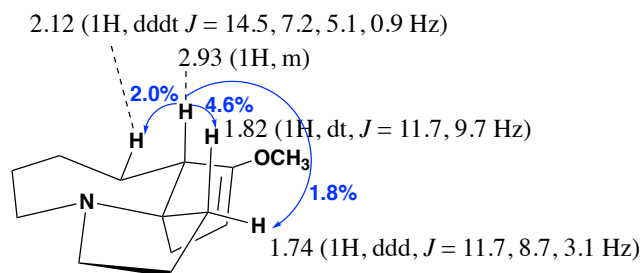


Figure S2. Difference NOE for *cis*-11a (C_6D_6 , 500 MHz).

Trans-fused compound *trans*-11a. Pale yellow oil; IR (film) 2931, 2843, 1639 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 4.31 (1H, q, $J = 2.4$ Hz), 3.56 (3H, s), 3.14–3.09 (2H, m), 3.04 (1H, dq, $J = 11.8, 2.0$ Hz), 2.86–2.80 (2H, m), 2.12 (1H, dd, $J = 13.4, 3.1$ Hz), 2.07 (1H, dq, $J = 13.4, 1.8$ Hz), 2.03 (1H, dqd, $J = 14.1, 3.3, 1.6$ Hz), 1.88–1.75 (3H, m), 1.69 (1H, tdd, $J = 11.0, 8.8, 1.1$ Hz), 1.63–1.57 (2H, m), 1.48 (1H, ddt, $J = 15.2, 7.2, 3.7$ Hz), 1.41 (1H, dtd, $J = 14.0, 11.9, 4.2$ Hz), 1.32 (1H, qdd, $J = 12.5, 5.1, 3.7$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 160.0 (C), 90.2 (CH), 76.0 (C), 55.8 (CH_3), 54.0 (CH_2), 53.5 (CH), 48.6 (CH_2), 43.5 (CH_2), 31.9 (CH_2), 28.7 (CH_2), 25.3 (CH_2), 22.9 (CH_2), 22.7 (CH_2). HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{22}\text{NO}^+$ 208.1696, found 208.1694.

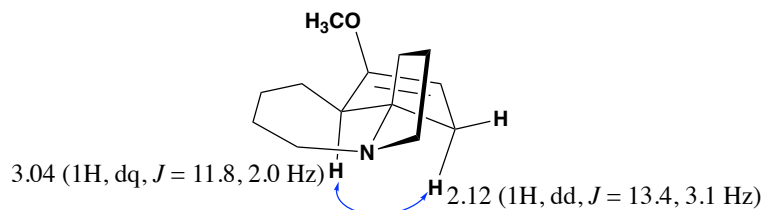
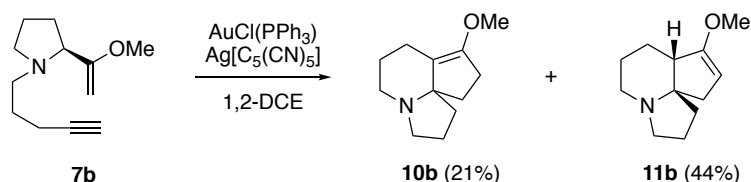


Figure S3. 2D NOESY for *trans*-**11a** (600 MHz, CDCl_3).



Cyclization reaction with 7b. To a solution of amine **7b** (173 mg, 1.29 mmol, 1.0 equiv) in 1,2-dichloroethane (13 mL) were added $\text{AuCl(PPh}_3\text{)}$ (31.6 mg, 0.065 mmol, 5 mol%) and $\text{Ag[C}_5\text{(CN)}_5\text{]}$ (20.0 mg, 0.065 mmol, 5 mol%). The reaction mixture was stirred at 80 °C under dark condition for 17 h. After cooling to room temperature, flash chromatography on alumina (50% Et_2O in *n*-hexane \rightarrow 5% MeOH in Et_2O) afforded **10b** (37 mg, 21%) and **11b** (76 mg, 44%).

Internal enol ether 10b. Pale yellow oil; IR (film) 2937, 2847, 1699 cm^{-1} ; $^1\text{H NMR}$ (CDCl_3 , 600 MHz) δ 3.63 (3H, s), 2.99 (1H, dt, $J = 12.3, 8.9$ Hz), 2.70 (1H, ddd, $J = 12.2, 8.8, 2.9$ Hz), 2.70 (1H, m), 2.52 (1H, dddd, $J = 14.2, 4.5, 2.4, 1.5$ Hz), 2.45 (1H, ddt, $J = 15.2, 9.7, 1.8$ Hz), 2.40 (1H, dddd, $J = 15.2, 9.2, 7.5, 4.2$ Hz), 2.37 (1H, td, $J = 11.9, 2.4$ Hz), 1.98 (1H, m), 1.85–1.72 (4H, m), 1.71–1.63 (2H, m), 1.54 (1H, qt, $J = 12.8, 4.1$ Hz), 1.47 (1H, ddd, $J = 12.2, 9.2, 2.9$ Hz); $^{13}\text{C NMR}$ (CDCl_3 , 600 MHz) δ 149.6 (C), 113.3 (C), 73.1 (C), 56.5 (CH_3), 52.6 (CH_2), 50.5 (CH_2), 36.8 (CH_2), 28.2 (CH_2), 26.7 (CH_2), 25.7 (CH_2), 21.3 (CH_2), 19.5 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{20}\text{NO}^+$ 194.1539, found 194.1541. Compound **10b** showed low specific optical rotation $[\alpha]_D^{24} = -0.9$ (c 1.90, CHCl_3) and enantiomeric excess of **10b** was not determined.

Cis-fused amine 11b. Pale yellow oil; IR (film) 2931, 2852, 2788, 1636 cm^{-1} ; $^1\text{H NMR}$ (CDCl_3 , 600 MHz) δ 4.38 (1H, t, $J = 2.0$ Hz), 3.59 (3H, s), 2.91 (1H, m), 2.85 (1H, m), 2.76–2.70 (2H, m), 2.49 (1H, d, $J = 14.1$ Hz), 2.18 (1H, dd, $J = 7.7, 6.1$ Hz), 1.91 (1H, dd, $J = 14.2, 2.7$ Hz), 1.87–1.80 (3H, m), 1.79–

1.71 (2H, m), 1.49–1.41 (2H, m), 1.34 (1H, m); ^{13}C NMR (CDCl_3 , 600 MHz) δ 163.2 (C), 90.6 (CH), 67.9 (C), 56.0 (CH_3), 51.7 (CH_2), 47.1 (CH_2), 45.1 (CH), 38.3 (CH_2), 36.4 (CH_2), 26.1 (CH_2), 21.4 (CH_2), 19.0 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{20}\text{NO}^+$ 194.1539, found 194.1541. Compound **11b** showed low specific optical rotation $[\alpha]_D^{24} = +3.0$ (c 1.10, CHCl_3) and enantiomeric excess of **11b** was not determined.

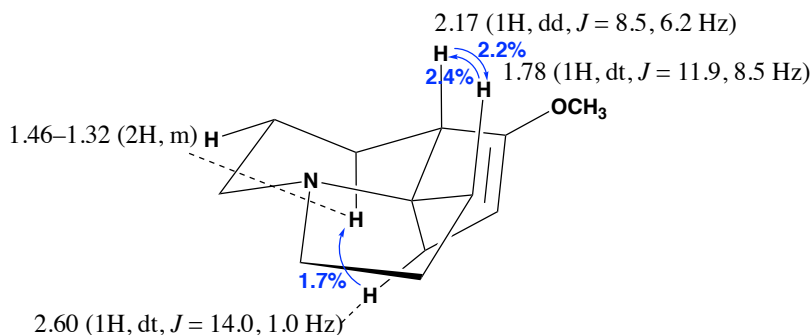
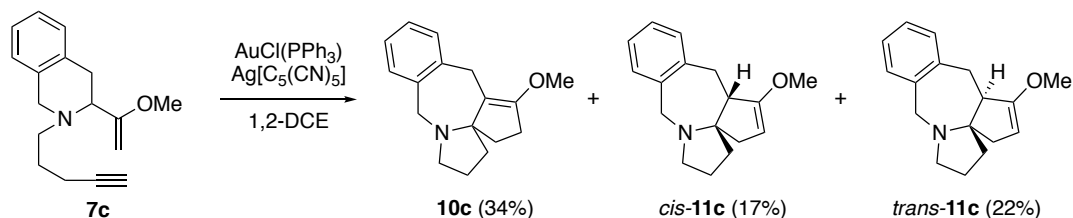


Figure S4. Difference NOE for **11b** (C_6D_6 , 500 MHz).



Cyclization reaction with 7c. To a solution of amine **7c** (325 mg, 1.27 mmol, 1.0 equiv) in 1,2-dichloroethane (13 mL) were added $\text{AuCl}(\text{PPh}_3)$ (6.3 mg, 0.013 mmol, 1 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (3.9 mg, 0.013 mmol, 1 mol%). The reaction mixture was stirred at 80 °C under dark condition for 24 h. After cooling to rt, flash chromatography on NH_2 silica gel (50% Et_2O in n -hexane) gave a mixture including *cis*-**11c** (101 mg) and a mixture including **10c** and *trans*-**11c** (212 mg). The former mixture was further purified by HPLC on NH_2 silica (Chomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 15% Et_2O in n -hexane, 19.0 mL/min, 11.9 min for *cis*-**11c**) to afford *cis*-**11c** (55 mg, 17%). The latter mixture was further purified by HPLC on NH_2 silica (Chomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 7% *i*-PrOH in n -hexane, collection between 8.9–11.2 min for **10c** and *trans*-**11c**) to afford a 61:39 mixture of **10c** and *trans*-**11c** (183 mg, 56%). Analytical samples of **10c** and *trans*-**11c** was obtained by separation of HPLC on NH_2 silica

(Chlomatorex NH₂, 5 μm, 2.0 cm x 25 cm, 7% *i*-PrOH in *n*-hexane, 19.0 mL/min, 6.6 min for **10c** and 6.2 min for *trans*-**11c**).

Internal enol ether 10c. Colorless solid; Mp 44–46 °C; IR (film) 2953, 2937, 2895, 1686, 1343, 1071 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.17–7.13 (2H, m), 7.08–7.03 (2H, m), 4.46 (1H, d, *J* = 15.8 Hz), 3.94 (1H, d, *J* = 17.6 Hz), 3.69 (1H, d, *J* = 15.8 Hz), 3.61 (3H, s), 3.31 (1H, dd, *J* = 17.6, 2.9 Hz), 2.83 (1H, m), 2.64 (1H, m), 2.51 (1H, dddd, *J* = 15.2, 8.9, 2.2, 1.5 Hz), 2.36 (1H, dtd, *J* = 15.2, 8.1, 3.6 Hz), 1.95 (1H, ddd, *J* = 12.8, 8.3, 2.4 Hz), 1.92 (1H, dt, *J* = 12.8, 8.6 Hz), 1.87–1.72 (4H, m); ¹³C NMR (CDCl₃, 600 MHz) δ 152.8 (C), 137.91 (C), 137.90 (C), 130.3 (CH), 129.3 (CH), 126.9 (CH), 125.4 (CH), 116.6 (C), 74.3 (C), 56.1 (CH₃), 51.9 (CH₂), 50.4 (CH₂), 37.3 (CH₂), 35.5 (CH₂), 29.4 (CH₂), 26.8 (CH₂), 23.8 (CH₂); HRDARTMS *m/z* [M+H]⁺ calcd for C₁₇H₂₂NO⁺ 256.1696, found 256.1692. Compound **10c** showed low specific optical rotation [α]²⁴_D = –3.8 (*c* 0.59, CHCl₃), and enantiomeric excess of **10c** was not determined.

Cis-fused compound cis-11c. Colorless oil; IR (film) 3065, 2931, 2900, 2845, 1641, 1456, 1341, 1245 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.15–7.08 (4H, m), 4.53 (1H, d, *J* = 15.2 Hz), 4.40 (1H, dd, *J* = 2.8, 1.7 Hz), 3.76 (1H, d, *J* = 15.2 Hz), 3.55 (3H, s), 3.01 (1H, dd, *J* = 14.2, 10.9 Hz), 2.90 (1H, dt, *J* = 14.3, 1.7 Hz), 2.89 (1H, dd, *J* = 14.2, 3.2 Hz), 2.80 (1H, td, *J* = 8.8, 4.6 Hz), 2.56 (1H, td, *J* = 8.8, 6.9 Hz), 2.25 (1H, ddd, *J* = 10.8, 3.2, 1.3 Hz), 1.99 (1H, dd, *J* = 14.3, 2.8 Hz), 1.96 (1H, ddd, *J* = 12.7, 9.0, 6.2 Hz), 1.86 (1H, ddd, *J* = 12.7, 9.5, 6.2 Hz), 1.72 (1H, dddt, *J* = 12.7, 9.2, 8.4, 6.4 Hz), 1.62 (1H, dtdd, *J* = 12.7, 8.8, 6.0, 4.6 Hz); ¹³C NMR (CDCl₃, 600 MHz) δ 162.2 (C), 139.2 (C x 2), 129.2 (CH), 128.8 (CH), 127.0 (CH), 125.9 (CH), 90.9 (CH), 70.5 (C), 55.9 (CH₃), 50.92 (CH₂), 50.88 (CH₂), 49.3 (CH), 43.0 (CH₂), 37.4 (CH₂), 35.0 (CH₂), 21.3 (CH₂); HRDARTMS *m/z* [M+H]⁺ calcd for C₁₇H₂₂NO⁺ 256.1696, found 256.1690. Compound *cis*-**11c** showed low specific optical rotation [α]²⁵_D = +0.7 (*c* 3.53, CHCl₃), and enantiomeric excess was not determined.

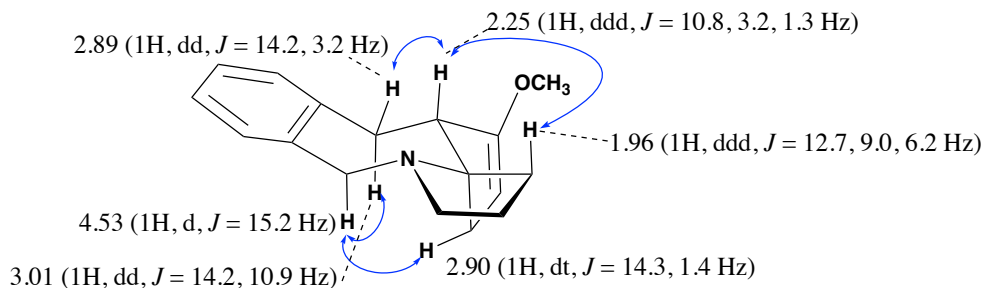


Figure S5. 2D NOESY for *cis*-**11c** (CDCl₃, 600 MHz).

Trans-fused compound *trans*-11c. Colorless solid; Mp 50–52 °C; IR (film) 2929, 2835, 1638, 1446, 1232 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.18–7.15 (2H, m), 7.05 (1H, ddd, 7.7, 6.4, 2.6 Hz), 6.99 (1H, dd, J = 7.3, 1.3 Hz), 4.79 (1H, d, J = 15.8 Hz), 4.36 (1H, ddd, J = 3.1, 2.4, 1.8 Hz), 3.85 (1H, ddt, J = 13.5, 4.5, 2.3 Hz), 3.60 (3H, s), 3.47 (1H, d, J = 15.8 Hz), 3.44 (1H, dd, J = 17.8, 4.6 Hz), 3.06 (1H, ddd, J = 9.4, 7.7, 1.3 Hz), 2.96 (1H, dd, J = 17.8, 13.6 Hz), 2.64 (1H, td, J = 10.1, 6.2 Hz), 2.22 (1H, dq, J = 13.4, 1.9 Hz), 2.11 (1H, dd, J = 13.5, 3.2 Hz), 1.71 (1H, m), 1.61–1.51 (3H, m); ¹³C NMR (CDCl₃, 600 MHz) δ 159.9 (C), 137.8 (C), 137.6 (C), 132.0 (CH), 130.6 (CH), 127.2 (CH), 125.0 (CH), 90.8 (CH), 74.3 (C), 56.2 (CH₂), 56.0 (CH₃), 52.3 (CH₂), 45.2 (CH), 43.8 (CH₂), 32.1 (CH₂), 32.0 (CH₂), 22.8 (CH₂); HRDARTMS *m/z* [M+H]⁺ calcd for C₁₇H₂₂NO⁺ 256.1696, found 256.1694. Compound *trans*-**11c** showed low specific optical rotation [α]²⁴_D = -7.0 (*c* 0.41, CHCl₃), and enantiomeric excess of *trans*-**11c** was not determined.

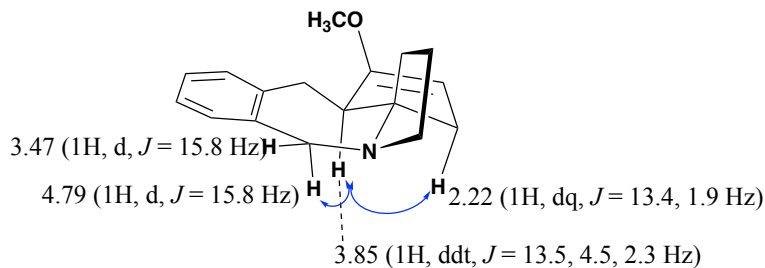
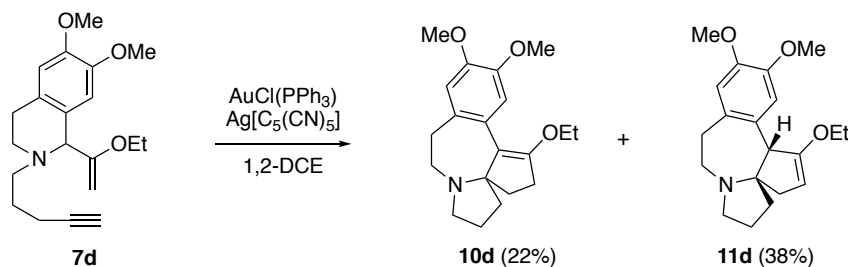


Figure S6. 2D NOESY for *trans*-**11c** (CDCl₃, 600 MHz).



Cyclization reaction with 7d. To a solution of amine **7d** (167 mg, 0.50 mmol, 1.0 equiv) in 1,2-dichloroethane (5 mL) were added AuCl(PPh₃) (2.8 mg, 0.005 mmol, 1 mol%) and Ag[C₅(CN)₅] (1.8 mg, 0.005 mmol, 1 mol%). The reaction mixture was stirred at 80 °C under dark condition for 25 h. After cooling to room temperature, the mixture was filtered through the alumina pad with aid of 5% MeOH in Et₂O, and the filtrate was concentrated. The residue was purified by flash chromatography on NH₂ silica (50% Et₂O in *n*-hexane → Et₂O → 5% MeOH in Et₂O) afforded **10d** (36 mg, 22%) and a mixture including **11d** (71 mg). The mixture was further purified by HPLC on NH₂ silica (Chomatorex NH₂, 5 μm, 2.0 cm x 25 cm, 5% *i*-PrOH in *n*-hexane, 7.9 min for **11d**) afforded **11d** (63 mg, 38%).

Internal enol ether 10d. Pale yellow amorphous solid; IR (film) 2925, 2851, 1512 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.87 (1H, s), 6.57 (1H, s), 3.90 (1H, dq, *J* = 9.7, 7.0 Hz), 3.89 (1H, dq, *J* = 9.7, 7.0 Hz), 3.844 (3H, s), 3.841 (3H, s), 3.52 (1H, ddd, *J* = 14.7, 11.5, 3.7 Hz), 3.25 (1H, ddd, *J* = 17.1, 11.9, 4.8 Hz), 2.96 (1H, ddd, *J* = 14.7, 4.5, 3.5 Hz), 2.90 (1H, dt, *J* = 17.1, 3.4 Hz), 2.86 (1H, m), 2.83 (1H, td, *J* = 8.8, 5.9 Hz), 2.56 (1H, ddd, *J* = 15.4, 7.7, 3.9 Hz), 2.44 (1H, dt, *J* = 15.4, 8.1 Hz), 1.89–1.84 (2H, m), 1.78–1.67 (3H, m), 1.56 (1H, m), 1.24 (3H, t, *J* = 7.0 Hz); ¹³C NMR (CDCl₃, 600 MHz) δ 152.2 (C), 147.2 (C), 146.1 (C), 130.0 (C), 125.4 (C), 119.0 (C), 114.4 (CH), 112.4 (CH), 76.9 (C), 64.3 (CH₂), 55.8 (CH₃), 55.7 (CH₃), 49.2 (CH₂), 44.0 (CH₂), 37.6 (CH₂), 35.9 (CH₂), 32.5 (CH₂), 27.9 (CH₂), 23.8 (CH₂), 15.5 (CH₃); HRDARTMS *m/z* [M+H]⁺ calcd for C₂₀H₂₈NO₃⁺ 330.2064, found 330.2061.

Cis-fused compound 11d. Pale yellow amorphous solid; IR (film) 2937, 2875, 2792, 1647, 1515 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.65 (1H, s), 6.61 (1H, s), 4.47 (1H, q, *J* = 2.4 Hz), 3.86 (3H, s), 3.84 (3H, s), 3.82 (1H, td, *J* = 2.3, 1.3 Hz), 3.79 (1H, dq, *J* = 9.7, 7.0 Hz), 3.74 (1H, dq, *J* = 9.7, 7.0 Hz), 3.22 (1H, ddd, *J* = 14.1, 12.2, 7.7 Hz), 3.03 (1H, ddd, *J* = 9.4, 8.3, 4.9 Hz), 2.93 (1H, td, *J* = 11.8, 7.2 Hz), 2.68 (1H,

dt, $J = 15.9, 2.4$ Hz), 2.60 (1H, dd, $J = 11.5, 7.4$ Hz), 2.44 (1H, td, $J = 9.6, 6.8$ Hz), 2.38 (1H, dd, $J = 14.1, 6.8$ Hz), 1.98 (1H, ddd, $J = 15.9, 2.2, 1.3$ Hz), 1.98–1.90 (2H, m), 1.75–1.69 (2H, m), 1.16 (3H, t, $J = 7.0$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 156.5 (C), 147.6 (C), 146.9 (C), 130.7 (C), 128.9 (C), 115.2 (CH), 112.5 (CH), 92.0 (CH), 66.7 (C), 64.4 (CH_2), 60.3 (CH), 56.0 (CH_3), 55.8 (CH_3), 53.0 (CH_2), 48.8 (CH_2), 42.8 (CH_2), 31.6 (CH_2), 30.3 (CH_2), 19.7 (CH_2), 14.3 (CH_3); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{28}\text{NO}_3^+$ 330.2064, found 330.2060.

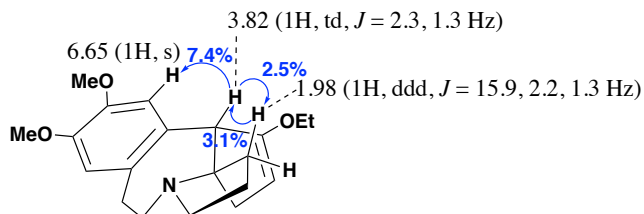
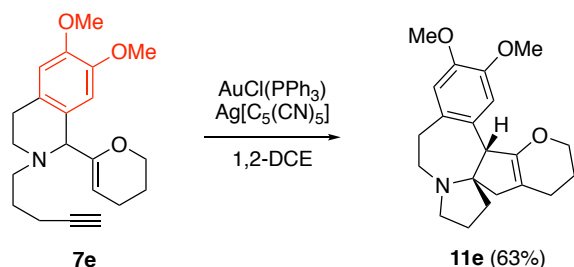


Figure S7. Difference NOE for compound **11d**.



Cyclization reaction with 7e. To a solution of cyclic amine **7e** (83.3 mg, 0.244 mmol, 1.0 equiv) in 1,2-dichloroethane (2.5 mL) were added $\text{AuCl}(\text{PPh}_3)$ (1.2 mg, 0.0025 mmol) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (0.7 mg, 0.0025 mmol). The reaction mixture was stirred under dark condition at 80 °C for 4 h. Additional $\text{AuCl}(\text{PPh}_3)$ (1.2 mg, 0.0025 mmol) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (0.7 mg, 0.0025 mmol) were added and the mixture was further stirred at 80 °C for 1 h. After cooling to room temperature, the mixture was concentrated. Flash chromatography on NH_2 silica gel (50% Et_2O in n -hexane \rightarrow Et_2O) afforded **11e** (52.1 mg, 63%) as a pale yellow amorphous solid. IR (film) 2935, 2837, 2832, 1695, 1510 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 6.65 (1H, s), 6.62 (1H, s), 3.97–3.92 (2H, m), 3.85 (3H, s), 3.84 (1H, s), 3.83 (3H, s), 3.25 (1H, ddd, $J = 13.8, 12.4, 7.6$ Hz), 3.03 (1H, td, $J = 8.9, 4.8$ Hz), 2.93 (1H, td, $J = 11.8, 7.2$ Hz), 2.61 (1H, d, $J = 14.5$ Hz), 2.59 (1H, dd, $J = 11.4, 7.7$ Hz), 2.44 (1H, td, $J = 9.6, 6.9$ Hz), 2.39 (1H, dd, $J = 13.9, 7.0$ Hz), 2.07–1.94 (4H, m), 1.92 (1H, d, $J = 14.5$ Hz), 1.86–1.82 (2H, m), 1.75–1.69 (2H, m); ^{13}C NMR (CDCl_3 , 600

MHz) δ 148.7 (C), 147.7 (C), 147.0 (C), 130.8 (C), 128.7 (C), 114.7 (CH), 112.7 (CH), 105.6 (C), 67.0 (CH₂), 65.4 (C), 60.0 (CH), 55.9 (CH₃), 55.7 (CH₃), 53.0 (CH₂), 48.7 (CH₂), 42.7 (CH₂), 34.7 (CH₂), 30.5 (CH₂), 22.7 (CH₂), 21.7 (CH₂), 19.7 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₂₁H₂₈NO₃⁺ 342.2064, found 342.2071.

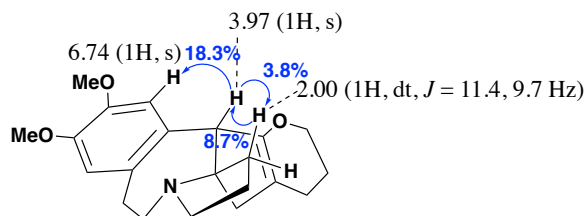
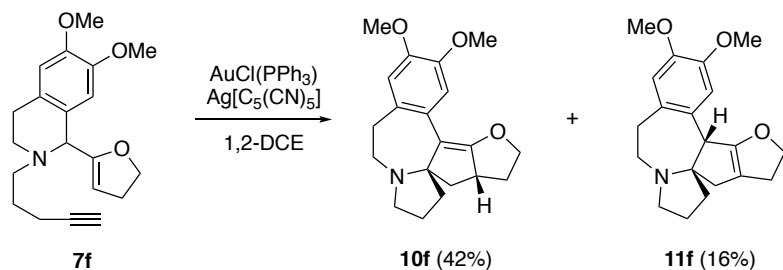


Figure S8. Difference NOE for compound **11e**.



Cyclization reaction with 7f. To a solution of cyclic amine **7f** (109 mg, 0.334 mmol, 1.0 equiv) in 1,2-dichloroethane (3.3 mL) were added AuCl(PPh₃) (1.7 mg, 0.0033 mmol, 1 mol%) and Ag[C₅(CN)₅] (1.0 mg, 0.0033 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 2 h. Additional AuCl(PPh₃) (2.5 mg, 0.0050 mmol, 1.5 mol%) and Ag[C₅(CN)₅] (1.5 mg, 0.0050 mmol, 1.5 mol%) were added and the mixture was stirred at 80 °C for 3 h. The reaction mixture was concentrated and flash chromatography on NH₂ silica gel (50% Et₂O in *n*-hexane → Et₂O) gave a mixture of **10f** and **11f**. The mixture was further purified by HPLC on NH₂ silica (Chlomatorex NH₂, 5 μ m, 2.0 cm x 25 cm, Et₂O, 16.0 mL/min, 24.0 min for **10f** and 19.0 min for **11f**) to give **10f** (46 mg, 42%) and **11f** (18 mg, 16%).

Conjugated enol ether 10f. Pale yellow amorphous solid; IR (film) 2930, 2895, 2852, 1683, 1514 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.81 (1H, s), 6.57 (1H, s), 4.49 (1H, t, J = 8.5 Hz), 4.38 (1H, ddd, J = 11.6, 8.7, 5.2 Hz), 3.86 (3H, s), 3.83 (3H, s), 3.57 (1H, ddd, J = 14.5, 12.3, 3.7 Hz), 3.40 (1H, ddd, J =

17.3, 12.3, 4.8 Hz), 3.05 (1H, ddd, $J = 14.5, 4.8, 3.3$ Hz), 2.96–2.91 (2H, m), 2.87 (1H, ddd, $J = 17.3, 3.7, 3.3$ Hz), 2.80 (1H, m), 2.13 (1H, ddd, $J = 11.8, 6.8, 5.2$ Hz), 2.07 (1H, dd, $J = 11.6, 6.4$ Hz), 2.01 (1H, m), 1.77–1.66 (3H, m), 1.63 (1H, qd, $J = 11.6, 8.4$ Hz), 1.53 (1H, dd, $J = 11.3, 9.4$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 158.8 (C), 147.1 (C), 146.4 (C), 129.1 (C), 124.3 (C), 114.2 (CH), 112.7 (CH), 111.0 (C), 82.6 (C), 76.2 (CH_2), 55.8 (CH_3), 55.7 (CH_3), 48.7 (CH_2), 44.7 (CH_2), 42.6 (CH_2), 42.3 (CH), 35.6 (CH_2), 32.2 (CH_2), 31.1 (CH_2), 24.1 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{26}\text{NO}_3^+$ 328.1907, found 328.1907.

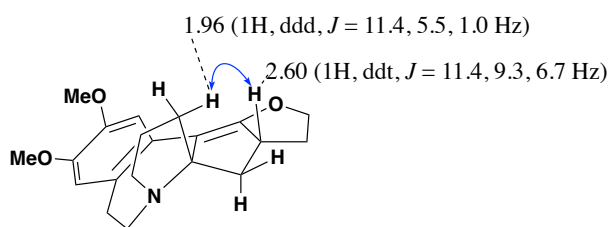


Figure S9. 1D Gradient NOESY for compound **10f** (C_6D_6 , 400 MHz).

Unconjugated enol ether 11f. Pale yellow amorphous solid; IR (film) 2929, 2853, 2797, 1706, 1514 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 6.65 (1H, s), 6.63 (1H, s), 4.65 (2H, t, $J = 8.8$ Hz), 3.84 (3H, s), 3.83 (3H, s), 3.77 (1H, s), 3.22 (1H, ddd, $J = 13.8, 12.8, 7.2$ Hz), 3.09 (1H, m), 2.98 (1H, ddd, $J = 12.8, 11.6, 6.6$ Hz), 2.66 (1H, dd, $J = 11.6, 7.2$ Hz), 2.64 (1H, d, $J = 15.6$ Hz), 2.62–2.53 (2H, m), 2.46 (1H, td, $J = 9.3, 7.7$ Hz), 2.41 (1H, dd, $J = 13.8, 6.6$ Hz), 2.10 (1H, ddd, $J = 11.7, 6.2, 5.0$ Hz), 2.01 (1H, dt, $J = 11.7, 10.1$ Hz), 1.89 (1H, dq, $J = 15.6, 1.1$ Hz), 1.79–1.74 (2H, m); ^{13}C NMR (CDCl_3 , 600 MHz) δ 159.0 (C), 147.8 (C), 147.2 (C), 130.6 (C), 128.0 (C), 114.3 (CH), 112.9 (CH), 110.8 (C), 76.2 (CH_2), 74.8 (C), 55.9 (CH_3), 55.8 (CH_3), 54.8 (CH), 53.4 (CH_2), 49.5 (CH_2), 43.6 (CH_2), 30.5 (CH_2), 29.0 (CH_2), 28.5 (CH_2), 19.8 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{26}\text{NO}_3^+$ 328.1907, found 328.1902.

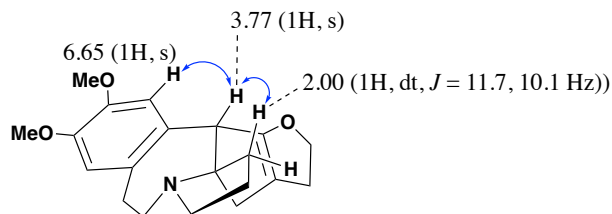
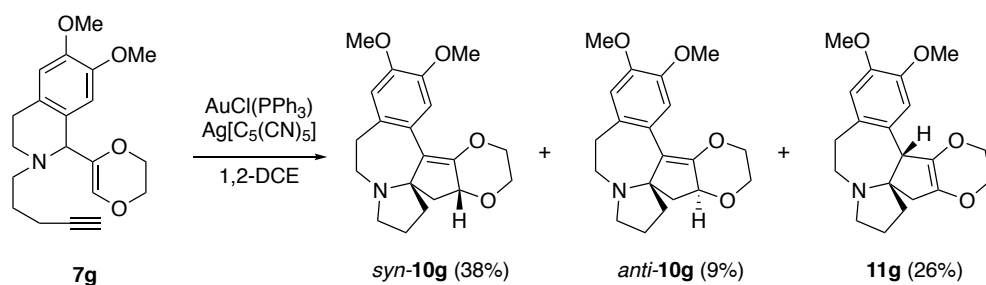


Figure S10. 2D NOESY for compound **11f** (CDCl_3 , 600 MHz).



Cyclization reaction with 7g. To a solution of cyclic amine **7g** (496 mg, 1.44 mmol, 1.0 equiv) in 1,2-dichloroethane (14 mL) were added $\text{AuCl}(\text{PPh}_3)$ (7.1 mg, 0.0144 mmol, 1 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (4.3 mg, 0.0144 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 17 h. Additional $\text{AuCl}(\text{PPh}_3)$ (3.5 mg, 0.0072 mmol, 0.5 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (2.1 mg, 0.0072 mmol, 0.5 mol%) were added and the mixture was stirred at 80 °C for 2 h. The reaction mixture was concentrated and flash chromatography on NH_2 silica gel (50% Et_2O in *n*-hexane \rightarrow Et_2O) afforded a mixture of **syn-10g** and **11g** (324 mg) and a mixture including **anti-10g** (66 mg). The former mixture was further purified by flash chromatography on alumina (Et_2O) to afford **syn-10g** (189 mg, 38%) and **11g** (130 mg, 26%). The latter mixture was further purified by HPLC on NH_2 silica (Chlomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 5% MeOH in Et_2O , 19.0 mL/min, 6.2 min for **anti-10g**) to give **anti-10g** (45 mg, 9%).

Conjugated *syn*-enol ether *syn-10g*. Colorless amorphous solid; Mp 168–170 °C; IR (film) 2956, 2929, 2853, 1514 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 6.71 (1H, s), 6.63 (1H, s), 4.35 (1H, dd, $J = 7.7, 7.0$ Hz), 3.97–3.86 (4H, m), 3.848 (3H, s), 3.847 (3H, s), 3.60 (1H, ddd, $J = 14.8, 12.3, 3.9$ Hz), 3.38 (1H, ddd, $J = 17.6, 12.3, 5.3$ Hz), 3.05 (1H, ddd, $J = 14.8, 5.3, 2.2$ Hz), 2.97 (1H, ddd, $J = 17.6, 3.9, 2.2$ Hz), 2.85 (1H, ddd, $J = 10.4, 8.4, 5.1$ Hz), 2.81 (1H, dd, $J = 8.4, 6.0$ Hz), 2.27 (1H, dd, $J = 11.8, 6.9$ Hz), 1.82–1.69 (4H, m), 1.56 (1H, td, $J = 12.1, 7.0$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 147.8 (C), 147.5 (C), 146.3 (C), 129.9 (C), 123.4 (C), 123.1 (C), 114.0 (CH), 112.7 (CH), 74.9 (CH), 72.0 (C), 67.7 (CH_2), 65.0 (CH_2), 55.9 (CH_3), 55.7 (CH_3), 48.3 (CH_2), 44.8 (CH_2), 41.9 (CH_2), 36.5 (CH_2), 32.3 (CH_2), 24.1 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{20}\text{H}_{26}\text{NO}_4^+$ 344.1856, found 344.1863.

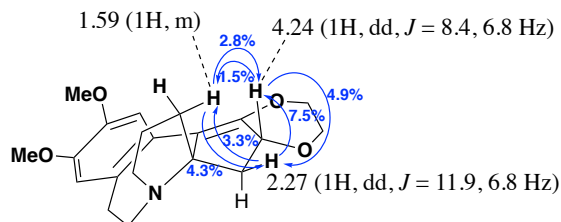


Figure S11. Difference NOE for compound *syn-10g* (C_6D_6 , 500 MHz).

Conjugated *anti*-enol ether *anti-10g*. Colorless solid; Mp 72–75 °C; IR (film) 2957, 2917, 2849, 1671, 1513 cm^{-1} ; 1H NMR ($CDCl_3$, 600 MHz) δ 6.88 (1H, s), 6.58 (1H, s), 4.67 (1H, t, $J = 7.1$ Hz), 4.09–4.07 (2H, m), 3.97–3.90 (2H, m), 3.88 (3H, s), 3.84 (3H, s), 3.32 (1H, dd, $J = 14.6, 11.5$ Hz), 3.18 (1H, ddd, $J = 11.1, 8.4, 3.7$ Hz), 3.10 (1H, ddd, $J = 17.4, 11.5, 1.8$ Hz), 2.88 (1H, dt, $J = 11.1, 8.1$ Hz), 2.79 (1H, ddd, $J = 14.6, 5.3, 1.8$ Hz), 2.62 (1H, dd, $J = 17.4, 4.9$ Hz), 2.43 (1H, dd, $J = 12.3, 7.3$ Hz), 1.87 (1H, dtdd, $J = 11.8, 8.5, 3.7, 1.6$ Hz), 1.79 (1H, tq, $J = 11.9, 8.1$ Hz), 1.71 (1H, td, $J = 11.7, 8.5$ Hz), 1.63 (1H, dd, $J = 12.3, 6.9$ Hz), 1.52 (1H, ddd, $J = 12.1, 7.0, 1.6$ Hz); ^{13}C NMR ($CDCl_3$, 600 MHz) δ 150.0 (C), 147.7 (C), 146.5 (C), 130.7 (C), 123.9 (C), 117.8 (C), 113.2 (CH), 112.4 (CH), 76.0 (CH), 74.8 (C), 67.6 (CH_2), 65.2 (CH_2), 55.8 (CH_3), 55.7 (CH_3), 50.9 (CH_2), 50.6 (CH_2), 45.8 (CH_2), 37.5 (CH_2), 31.9 (CH_2), 24.1 (CH_2); HRDARTMS m/z $[M+H]^+$ calcd for $C_{20}H_{26}NO_4^+$ 344.1856, found 344.1860.

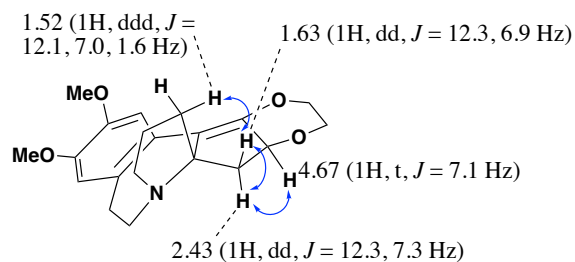


Figure S12. 1D Gradient NOESY for compound *anti-10g* ($CDCl_3$, 600 MHz).

Unconjugated enol ether 11g. Colorless amorphous solid. IR (film) 2932, 2867, 1719, 1515 cm^{-1} ; 1H NMR ($CDCl_3$, 600 MHz) δ 6.66 (1H, s), 6.65 (1H, s), 4.12–4.03 (4H, m), 3.93 (1H, dd, $J = 2.9, 0.9$ Hz), 3.85 (3H, s), 3.84 (3H, s), 3.37 (1H, ddd, $J = 13.7, 12.9, 7.5$ Hz), 3.07 (1H, td, $J = 9.5, 8.5, 4.9$ Hz), 2.95 (1H, ddd, $J = 12.9, 11.6, 6.9$ Hz), 2.77 (1H, dd, $J = 15.3, 3.0$ Hz), 2.63 (1H, dd, $J = 11.6, 7.5$ Hz), 2.44 (1H, dd, $J = 13.7, 6.9$ Hz), 2.43 (1H, td, $J = 9.5, 6.6$ Hz), 2.04 (1H, d, $J = 15.3$ Hz), 2.03 (1H, ddd, $J = 11.4, 8.3, 2.9$ Hz), 1.98 (1H, dt, $J = 11.4, 9.7$ Hz), 1.79–1.71 (2H, m); ^{13}C NMR ($CDCl_3$, 600 MHz) δ

147.9 (C), 147.1 (C), 130.7 (C), 129.3 (C), 128.9 (C), 128.3 (C), 114.7 (CH), 112.9 (CH), 65.4 (CH₂), 65.3 (CH₂), 63.3 (C), 57.8 (CH), 55.9 (CH₃), 55.8 (CH₃), 53.2 (CH₂), 49.0 (CH₂), 43.4 (CH₂), 30.4 (CH₂), 30.3 (CH₂), 19.6 (CH₂); HRDARTMS m/z $[M+H]^+$ calcd for C₂₀H₂₆NO₄⁺ 344.1856, found 344.1860.

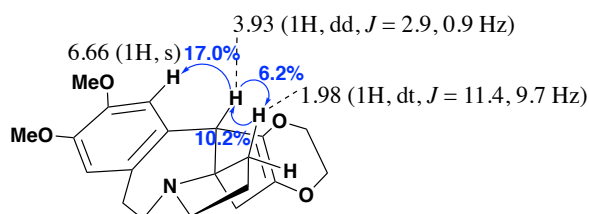
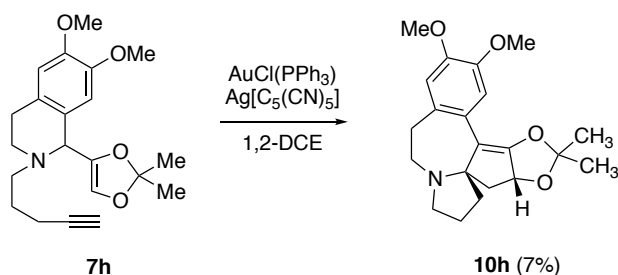


Figure S13. Difference NOE for compound **11g** (CDCl₃, 500 MHz).



Cyclization reaction with 7h. To a solution of cyclic amine **7h** (358 mg, 1.00 mmol, 1.0 equiv) in 1,2-dichloroethane (10 mL) were added AuCl(PPh₃) (4.9 mg, 0.010 mmol, 1 mol%) and Ag[C₅(CN)₅] (3.0 mg, 0.010 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 24 h. Additional AuCl(PPh₃) (4.9 mg, 0.010 mmol, 1 mol%) and Ag[C₅(CN)₅] (3.0 mg, 0.010 mmol, 1 mol%) were added and the mixture was stirred at 80 °C for 3 h. The reaction mixture was concentrated and flash chromatography on alumina (80% Et₂O in *n*-hexane → Et₂O) afforded **10h** (27 mg, 7%) as a pale yellow amorphous solid. IR (film) 2954, 2931, 2853, 1697, 1515 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.72 (1H, s), 6.62 (1H, s), 4.85 (1H, dd, $J = 7.7, 6.1$ Hz), 3.86 (3H, s), 3.84 (3H, s), 3.48 (1H, ddd, $J = 14.5, 10.9, 3.9$ Hz), 3.34 (1H, ddd, $J = 17.2, 10.9, 5.0$ Hz), 3.07 (1H, ddd, $J = 14.6, 5.0, 3.9$ Hz), 2.92 (1H, m), 2.91 (1H, dt, $J = 17.2, 3.9$ Hz), 2.76 (1H, ddd, $J = 7.6, 4.9, 3.3$ Hz), 2.22 (1H, dd, $J = 11.2, 6.1$ Hz), 1.90 (1H, m), 1.86 (1H, dd, $J = 11.2, 7.7$ Hz), 1.77–1.72 (3H, m), 1.56 (3H, s), 1.53 (3H, s); ¹³C NMR (CDCl₃, 600 MHz) δ 151.2 (C), 147.6 (C), 146.6 (C), 129.5 (C), 123.9 (C), 118.3 (C), 114.2 (CH), 113.4 (C), 112.7 (CH), 80.0 (C), 79.3 (CH), 55.9 (CH₃), 55.7 (CH₃), 48.6 (CH₂), 44.9 (CH₂), 42.4 (CH₂), 36.9 (CH₂), 32.1

(CH₂), 27.6 (CH₃), 25.4 (CH₃), 23.9 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₂₁H₂₈NO₄⁺ 358.2013, found 358.2017.

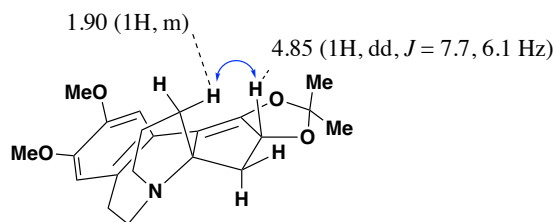
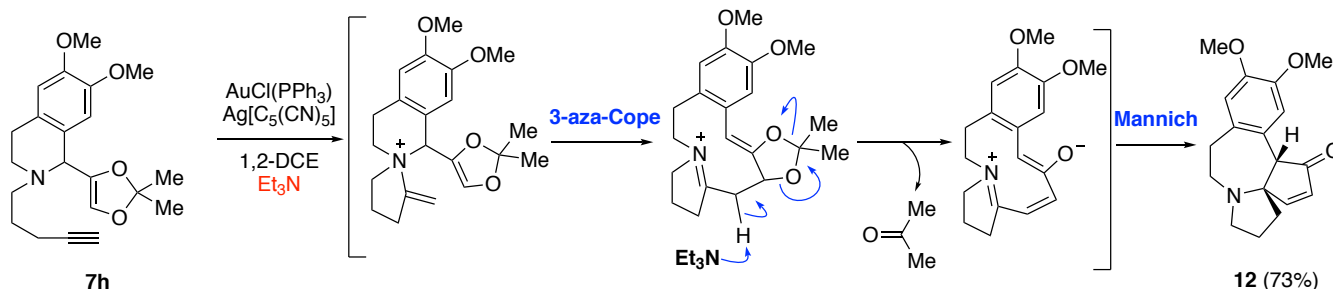


Figure S14. 1D gradient NOESY for compound **10h** (CDCl₃, 600 MHz).



Cyclization reaction with 7h in the presence of Et₃N. To a solution of cyclic amine **7h** (107 mg, 0.30 mmol, 1.0 equiv) in 1,2-dichloroethane (1 mL) and Et₃N (2 mL) were added Ph₃PAuCl (1.5 mg, 0.003 mmol, 1 mol%) and Ag[C₅(CN)₅] (0.9 mg, 0.003 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 24 h. The reaction mixture was concentrated and flash chromatography on NH₂ silica gel (Et₂O) afforded **12** (66 mg, 73%) as a pale yellow amorphous solid. IR (film) 2941, 2804, 1714, 1486 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.79 (1H, d, *J* = 5.9 Hz), 6.73 (1H, s), 6.68 (1H, s), 6.45 (1H, d, *J* = 6.1 Hz), 3.89 (3H, s), 3.85 (3H, s), 3.43 (1H, s), 3.13 (1H, td, *J* = 9.0, 4.2 Hz), 2.99 (1H, td, *J* = 11.4, 8.3 Hz), 2.79 (1H, td, *J* = 9.4, 7.7 Hz), 2.62 (1H, ddd, *J* = 11.4, 7.1, 1.2 Hz), 2.49 (1H, ddd, *J* = 14.9, 11.6, 7.3 Hz), 2.46 (1H, ddd, *J* = 14.9, 8.5, 1.2 Hz), 2.14 (1H, ddd, *J* = 12.7, 10.4, 8.6 Hz), 2.03 (1H, ddd, *J* = 12.6, 8.3, 4.2 Hz), 1.95–1.85 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 208.2 (C), 163.1 (CH), 148.2 (C), 147.3 (C), 135.1 (CH), 129.2 (C), 127.7 (C), 115.5 (CH), 113.2 (CH), 70.2 (C), 60.9 (CH), 55.9 (CH₃), 55.8 (CH₃), 53.1 (CH₂), 48.1 (CH₂), 37.9 (CH₂), 31.2 (CH₂), 20.2 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₁₈H₂₂NO₃⁺ 300.1594, found 300.1597.

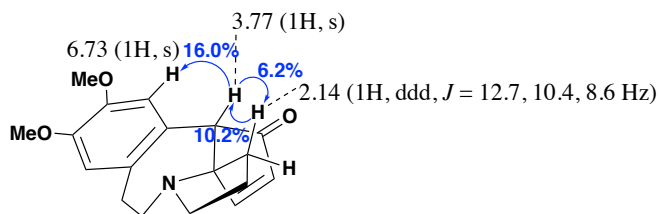
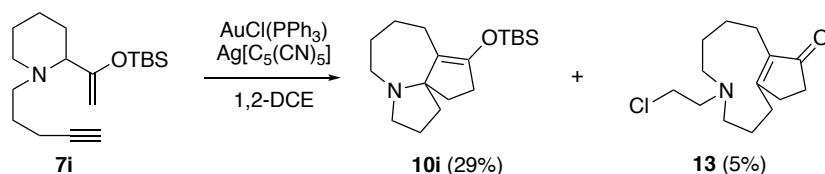


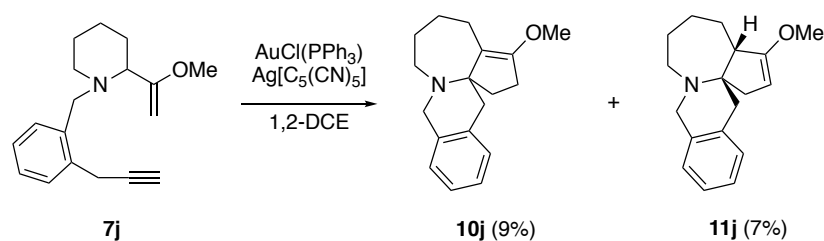
Figure S15. Difference NOE for compound **12** (CDCl_3 , 600 MHz).



Cyclization reaction with 7i. To a solution of cyclic amine **7i** (142 mg, 0.462 mmol, 1.0 equiv) in 1,2-dichloroethane (4.6 mL) were added $\text{AuCl}(\text{PPh}_3)$ (2.1 mg, 0.0046 mmol, 1 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (1.4 mg, 0.0046 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 1.5 h. Additional $\text{AuCl}(\text{PPh}_3)$ (6.3 mg, 0.014 mmol, 3 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (4.2 mg, 0.014 mmol, 3 mol%) were added and the mixture was stirred at 80 °C for 48 h. The reaction mixture was concentrated and flash chromatography on NH_2 silica gel (5→20% Et_2O in *n*-hexane) gave **10i** (41 mg, 29%) and a mixture including **13** (9 mg). Further purification for the mixture by flash chromatography on alumina (20→50% Et_2O in *n*-hexane) to give **13** (6 mg, 5%).

TBS enol ether 10i. Colorless oil; IR (film) 2952, 2928, 2857, 2792, 1681 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 3.09 (1H, td, $J = 8.4, 6.8$ Hz), 3.07 (1H, ddt, $J = 14.9, 4.8, 1.3$ Hz), 2.86 (1H, dd, $J = 14.9, 11.8$ Hz), 2.74 (1H, m), 2.20 (1H, dddd, $J = 14.9, 9.5, 7.3, 2.0$ Hz), 2.12 (1H, dd, $J = 14.9, 9.0$ Hz), 1.86–1.73 (6H, m), 1.70–1.65 (2H, m), 1.56 (1H, m), 1.46 (1H, tt, $J = 13.2, 2.4$ Hz), 1.37 (1H, m), 1.13 (1H, qdd, $J = 13.2, 4.0, 3.1$ Hz), 0.94 (9H, s), 0.133 (3H, s), 0.127 (3H, s); ^{13}C NMR (CDCl_3 , 150 MHz) δ 146.8 (C), 125.3 (C), 73.0 (C), 50.0 (CH_2), 46.8 (CH_2), 37.7 (CH_2), 36.5 (CH_2), 31.3 (CH_2), 29.5 (CH_2), 25.7 (CH_3), 24.7 (CH_2), 24.2 (CH_2), 23.3 (CH_2), 18.1 (C), –4.0 (CH_3), –4.1 (CH_3); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{34}\text{NOSi}^+$ 308.2404, found 308.2400.

2-Chloroethylated compound 13. Colorless oil; IR (film) 2957, 2926, 2854, 2796, 1691, 1636 cm^{-1} ; ^1H NMR (C_6D_6 , 600 MHz, 50 $^\circ\text{C}$) δ 3.19 (2H, m), 2.64 (2H, t, $J = 6.6$ Hz), 2.27 (2H, t, $J = 6.7$ Hz), 2.20 (2H, m), 2.05 (2H, AA'XX', $J_{\text{AA}'} = 12.5$ Hz, $J_{\text{XX}'} = 12.5$ Hz, $J_{\text{AX}} = 7.0$ Hz, $J_{\text{AX}'} = 3.0$ Hz), 1.99 (2H, m), 1.97 (2H, m), 1.86 (2H, m), 1.77 (2H, t, $J = 5.9$ Hz), 1.27 (2H, m), 1.09 (2H, dq, $J = 6.4, 5.3$ Hz); ^{13}C NMR (C_6D_6 , 150 MHz, 50 $^\circ\text{C}$) δ 208.3 (C), 171.0 (C), 142.0 (C), 56.9 (CH_2), 56.7 (CH_2), 48.3 (CH_2), 42.1 (CH_2), 34.6 (CH_2), 29.1 (CH_2), 27.3 (CH_2), 26.7 (CH_2), 23.9 (CH_2), 23.6 (CH_2), 23.4 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{23}\text{ONCl}^+$ 256.1463, found 256.1462.



Cyclization reaction with 7j. A 10-mL reaction vessel was charged with a solution of cyclic amine **7j** (69.4 mg, 0.258 mmol, 1.0 equiv) in 1,2-dichloroethane (2.6 mL). After addition of $\text{AuCl(PPh}_3\text{)}$ (2.6 mg, 0.0052 mmol, 2 mol%) and $\text{Ag[C}_5\text{(CN)}_5\text{]}$ (1.5 mg, 0.0052 mmol, 2 mol%), the vessel was sealed and the reaction mixture was heated at 130 $^\circ\text{C}$ for 0.5 h using a microwave reactor. After cooling to rt, the reaction mixture was concentrated and flash chromatography on NH_2 silica gel (5 \rightarrow 20% Et_2O in *n*-hexane) afforded recovered **7j** (45.3 mg, 65% recovery) and a mixture of **10j** and **11j**. The mixture was further purified by HPLC on NH_2 silica gel (Chlomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 20% Et_2O in *n*-hexane, 19.0 mL/min, 11.0 min for **10j** and 14.5 min for **11j**) to afford **10j** (6.4 mg, 9%) and **11j** (4.6 mg, 7%).

Internal enol ether 10j. Pale yellow oil; IR (film) 2926, 2846, 1686 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 7.12–7.06 (3H, m), 7.01 (1H, m), 3.89 (1H, d, $J = 15.4$ Hz), 3.84 (1H, d, $J = 15.4$ Hz), 3.64 (3H, s), 2.92 (1H, d, $J = 16.0$ Hz), 2.82 (1H, ddd, $J = 13.7, 11.6, 0.7$ Hz), 2.56 (1H, dt, $J = 12.7, 3.3$ Hz), 2.48 (1H, d, $J = 16.0$ Hz), 2.44–2.38 (2H, m), 2.35 (1H, ddd, $J = 13.7, 4.8, 2.6$ Hz), 1.83–1.75 (3H, m), 1.70 (1H, m), 1.63–1.56 (2H, m), 1.17 (1H, qdd, $J = 13.0, 4.8, 3.0$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 151.4 (C), 135.5

(C), 135.1 (C), 129.4 (CH), 126.0 (CH), 125.9 (CH), 125.2 (CH), 122.2 (C), 67.8 (C), 56.3 (CH₃), 54.1 (CH₂), 50.8 (CH₂), 38.4 (CH₂), 31.8 (CH₂), 29.6 (CH₂), 26.3 (CH₂), 24.3 (CH₂), 22.4 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₁₈H₂₄NO⁺ 270.1852, found 270.1847.

Cis-fused product 11j. Pale yellow oil; IR (film) 2922, 2846, 1653 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 7.14–7.09 (2H, m), 7.03 (1H, dd, $J = 7.1, 1.5$ Hz), 6.99 (1H, dd, $J = 6.9, 1.5$ Hz), 4.28 (1H, ddd, $J = 3.0, 1.7, 0.7$ Hz), 4.24 (1H, d, $J = 17.2$ Hz), 3.66 (1H, d, $J = 17.2$ Hz), 3.60 (3H, s), 2.97 (1H, d, $J = 17.0$ Hz), 2.80 (1H, d, $J = 17.0$ Hz), 2.75 (1H, m), 2.57 (1H, dt, $J = 14.3, 3.9$ Hz), 2.45 (1H, dq, $J = 14.1, 1.5$ Hz), 2.18 (1H, dt, $J = 10.6, 1.7$ Hz), 2.13 (1H, dd, $J = 14.1, 2.9$ Hz), 1.88 (1H, m), 1.86 (1H, dtd, $J = 14.0, 11.6, 2.0$ Hz), 1.71 (1H, ddt, $J = 14.0, 6.3, 2.1$ Hz), 1.68–1.63 (2H, m), 1.29 (1H, m); ¹³C NMR (CDCl₃, 600 MHz) δ 161.9 (C), 134.8 (C), 134.4 (C), 129.4 (CH), 126.1 (CH), 126.0 (CH), 125.6 (CH), 89.5 (CH), 64.4 (C), 58.9 (CH), 56.3 (CH₃), 55.6 (CH₂), 53.2 (CH₂), 38.8 (CH₂), 34.7 (CH₂), 31.5 (CH₂), 29.1 (CH₂), 27.0 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₁₈H₂₄NO⁺ 270.1852, found 270.1852.

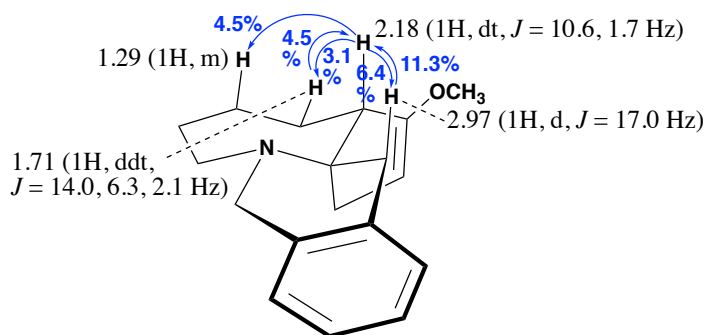
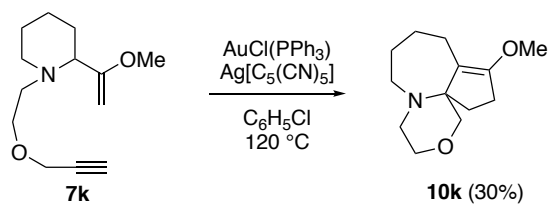
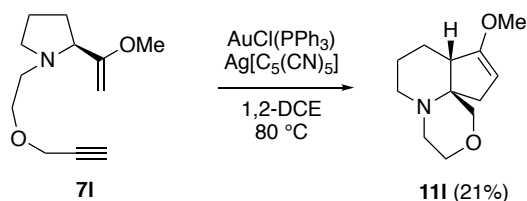


Figure S16. Difference NOE for compound **11j** (CDCl₃, 500 MHz).



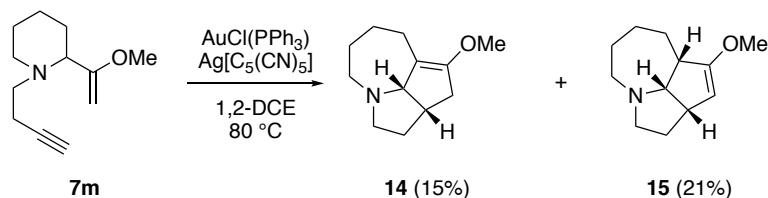
Cyclization reaction with 7k. To a solution of cyclic amine **7k** (50 mg, 0.22 mmol, 1.0 equiv) in chlorobenzene (2.2 mL) were added AuCl(PPh₃) (5.7 mg, 0.012 mmol, 5 mol%) and Ag[C₅(CN)₅] (3.4 mg, 0.012 mmol, 5 mol%), and the reaction mixture was heated at 120 °C for 24 h under dark condition.

After cooling to room temperature, the reaction mixture was concentrated and flash chromatography on alumina (30 → 40% Et₂O in *n*-hexane) afforded **10k** (15 mg, 30%) as a pale yellow oil. IR (film) 2926, 2846, 1684 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 3.74 (1H, ddt, *J* = 10.7, 3.2, 1.1 Hz), 3.61 (3H, s), 3.60 (1H, ddd, *J* = 11.6, 10.7, 3.0 Hz), 3.30 (1H, d, *J* = 10.3 Hz), 3.19 (1H, dd, *J* = 10.4, 2.5 Hz), 2.89 (1H, td, *J* = 11.6, 3.3 Hz), 2.77 (1H, ddd, *J* = 13.5, 11.7, 1.5 Hz), 2.44–2.38 (4H, m), 2.25 (1H, ddd, *J* = 12.4, 6.1, 2.6 Hz), 2.16 (1H, dddd, *J* = 13.8, 4.2, 2.9, 0.9 Hz), 1.77 (1H, m), 1.69 (1H, tdd, *J* = 12.7, 2.8, 1.5 Hz), 1.63 (1H, m), 1.59 (1H, m), 1.47 (1H, tddd, *J* = 13.2, 11.4, 4.6, 2.9 Hz), 1.07 (1H, qdd, *J* = 12.8, 4.8, 3.1 Hz); ¹³C NMR (CDCl₃, 600 MHz) δ 152.9 (C), 119.3 (C), 71.8 (CH₂), 68.5 (C), 67.9 (CH₂), 56.2 (CH₃), 51.5 (CH₂), 50.2 (CH₂), 32.0 (CH₂), 29.9 (CH₂), 26.4 (CH₂), 23.4 (CH₂), 22.6 (CH₂); HRDARTMS *m/z* [M+H]⁺ calcd for C₁₃H₂₂NO₂⁺ 224.1645, found 224.1646.



Cyclization reaction with 71. To a solution of cyclic amine **71** (133 mg, 0.64 mmol, 1.0 equiv) in chlorobenzene (6.4 mL) were added AuCl(PPh₃) (15.9 mg, 0.032 mmol, 5 mol%) and Ag[C₅(CN)₅] (9.7 mg, 0.032 mmol, 5 mol%), and the reaction mixture was heated at 80 °C for 22 h under dark condition. After cooling to room temperature, the reaction mixture was concentrated and the residue was purified by flash chromatography on alumina (30% Et₂O in *n*-hexane → Et₂O). Further purification was performed by HPLC on NH₂ silica gel (Chlomatorex NH₂, 10 μm, 2.0 cm x 25 cm, 20% Et₂O in *n*-hexane, 16.0 mL/min, 5.6 min for **11I**) to afford **11I** (28 mg, 21%) as a pale yellow oil. IR (film) 2936, 2848, 1643, 1447, 1106 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 4.35 (1H, t, *J* = 2.4 Hz), 3.77 (1H, ddt, *J* = 11.0, 3.7, 1.1 Hz), 3.72 (1H, td, *J* = 11.4, 2.8 Hz), 3.68 (1H, dd, *J* = 10.3, 0.9 Hz), 3.56 (3H, s), 3.19 (1H, d, *J* = 10.5 Hz), 2.70 (1H, td, *J* = 11.8, 3.9 Hz), 2.62 (1H, dt, *J* = 11.0, 5.2 Hz), 2.44 (1H, ddd, *J* = 11.3, 8.6, 4.9 Hz), 2.40 (2H, d, *J* = 2.4 Hz), 2.37 (1H, ddd, *J* = 11.8, 2.8, 1.1 Hz), 1.98 (1H, dtdd, *J* = 13.0, 6.3, 4.1, 1.1 Hz),

1.87 (1H, t, $J = 7.2$ Hz), 1.60–1.48 (2H, m), 1.30 (1H, dddd, $J = 13.2, 9.9, 8.3, 4.5$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 162.8 (C), 89.9 (CH), 75.4 (CH_2), 66.8 (CH_2), 62.1 (C), 56.1 (CH_3), 49.9 (CH_2), 49.2 (CH_2), 44.2 (CH), 25.9 (CH_2), 24.8 (CH_2), 21.2 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{20}\text{NO}_2^+$ 210.1489, found 210.1488. Compound **11l** showed low specific optical rotation $[\alpha]_D^{24} = +0.5$ (c 2.36, CHCl_3), and enantiomeric excess of **11l** was not determined.



Cyclization reaction with 7m. To a solution of cyclic amine **7m** (130 mg, 0.67 mmol, 1.0 equiv) in 1,2-dichloroethane (6.7 mL) were added $\text{AuCl}(\text{PPh}_3)$ (6.0 mg, 0.013 mmol, 2 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (4.0 mg, 0.013 mmol, 2 mol%). The reaction mixture was stirred under dark condition at 80 °C for 26 h. The reaction mixture was concentrated and flash chromatography on NH_2 silica gel (15→ 40% Et_2O in *n*-hexane) gave **14** (20 mg, 15%) and a mixture of **7m** and **15** (77 mg). The mixture was separated by HPLC on NH_2 silica gel (Chomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 10% Et_2O in *n*-hexane, 16.0 mL/min, 9.5 min for **7m** and 11.0 min for **15**) to afford **15** (28 mg, 21%) and recovered **7m** (36 mg, 28% recovery).

Internal enol ether 14. Colorless oil; IR (film) 2924, 2781, 1684 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 3.60 (3H, s), 3.27 (1H, d, $J = 6.8$ Hz), 3.06 (1H, ddd, $J = 11.9, 4.4, 3.6$ Hz), 2.96 (1H, dd, $J = 8.0, 7.4$ Hz), 2.71–2.67 (2H, m), 2.38–2.32 (3H, m), 2.26 (1H, ddd, $J = 11.0, 9.0, 5.6$ Hz), 2.17 (1H, td, $J = 11.0, 2.7$ Hz), 2.09 (1H, dddd, $J = 12.7, 7.8, 5.1, 1.3$ Hz), 1.85 (1H, m), 1.79 (1H, m), 1.66–1.55 (3H, m); ^{13}C NMR (CDCl_3 , 150 MHz) δ 149.8 (C), 115.0 (C), 74.2 (CH), 56.3 (CH_3), 56.0 (CH_2), 55.6 (CH_2), 36.8 (CH_2), 36.4 (CH), 33.3 (CH_2), 30.6 (CH_2), 25.7 (CH_2), 25.3 (CH_2). HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{20}\text{NO}^+$ 194.1539, found 194.1545.

Tricyclic compound 15. Colorless oil; IR (film) 2926, 2800, 1646, 1292, 1248, 1212 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 4.42 (1H, t, $J = 2.0$ Hz), 3.54 (3H, s), 3.17 (1H, qt, $J = 8.1, 1.5$ Hz), 3.11 (1H, dddd,

$J = 12.3, 4.4, 3.0, 1.1$ Hz), 2.96 (1H, dd, $J = 9.1, 7.6$ Hz), 2.89 (1H, t, $J = 7.7$ Hz), 2.72 (1H, dddt, $J = 12.1, 7.7, 6.2, 1.6$ Hz), 2.25 (1H, ddd, $J = 11.7, 9.2, 6.6$ Hz), 2.08 (1H, td, $J = 12.2, 3.6$ Hz), 2.00 (1H, ddd, $J = 14.0, 7.7, 6.6$ Hz), 1.96 (1H, ddd, $J = 12.2, 9.5, 6.6$ Hz), 1.87 (1H, m), 1.72–1.63 (2H, m), 1.54 (1H, tt, $J = 12.1, 7.0$ Hz), 1.49 (1H, dddd, $J = 14.0, 12.1, 11.0, 0.9$ Hz), 1.30 (1H, m); ^{13}C NMR (CDCl_3 , 150 MHz) δ 160.3 (C), 97.4 (CH), 72.0 (CH), 58.2 (CH_2), 57.7 (CH_2), 56.5 (CH_3), 47.9 (CH), 45.1 (CH), 31.6 (CH_2), 31.4 (CH_2), 28.7 (CH_2), 26.4 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{12}\text{H}_{20}\text{NO}^+$ 194.1539, found 194.1544.

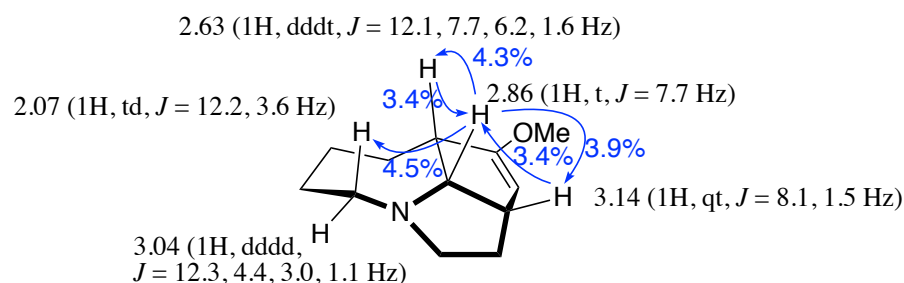
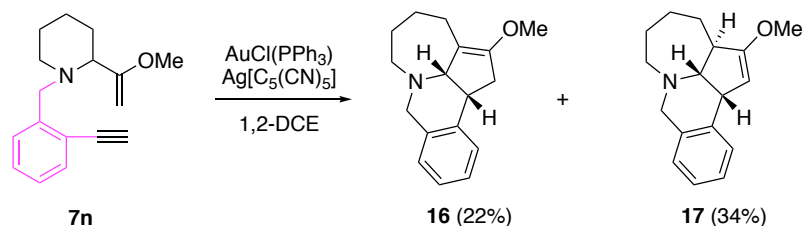


Figure S17. Difference NOE for compound **15** (CD_3CN , 500 MHz).



Cyclization reaction with 7n. To a solution of cyclic amine **7n** (233 mg, 0.912 mmol, 1.0 equiv) in 1,2-dichloroethane (9.1 mL) were added $\text{AuCl}(\text{PPh}_3)$ (4.5 mg, 0.0091 mmol, 1 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (2.7 mg, 0.0091 mmol, 1 mol%). The reaction mixture was stirred under dark condition at 80 °C for 2.5 h. The reaction mixture was concentrated and flash chromatography on NH_2 silica gel (5 \rightarrow 20% Et_2O in *n*-hexane) gave a mixture of **16** and **17**. The mixture was further purified by HPLC on NH_2 silica gel (Chomatorex NH_2 , 5 μm , 2.0 cm x 25 cm, 5% Et_2O in *n*-hexane, 19.0 mL/min, 13.0 min for **16** and 11.0 min for **17**) to afford **16** (52 mg, 22%) and **17** (79 mg, 34%).

Internal enol ether 16. Pale yellow oil; IR (film) 2920, 2845, 1680 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 7.17–7.14 (2H, m), 7.09 (1H, m), 7.01 (1H, d, $J = 7.5$ Hz), 3.84 (1H, d, $J = 14.6$ Hz), 3.70 (1H, d, $J = 6.4$ Hz), 3.58 (3H, s), 3.51 (1H, dt, $J = 8.2, 6.3$ Hz), 3.41 (1H, d, $J = 14.6$ Hz), 3.21 (1H, dddd, $J = 13.4, 4.6, 2.9, 1.0$ Hz), 2.97 (1H, dddd, $J = 15.0, 8.7, 2.2, 1.3$ Hz), 2.61–2.55 (2H, m), 2.41 (1H, m), 2.34 (1H, dddd, $J = 16.1, 10.8, 5.3, 2.0, 1.3$ Hz), 1.86 (1H, m), 1.79–1.70 (2H, m), 1.51 (1H, dddd, $J = 13.9, 10.8, 5.3, 3.3$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 152.6 (C), 138.2 (C), 135.5 (C), 128.2 (CH), 126.6 (CH), 125.8 (CH), 125.3 (CH), 115.7 (C), 68.5 (CH), 59.5 (CH_2), 56.2 (CH_3), 54.2 (CH_2), 38.7 (CH), 36.6 (CH_2), 28.6 (CH_2), 26.3 (CH_2), 25.0 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{22}\text{NO}^+$ 256.1696, found 256.1695.

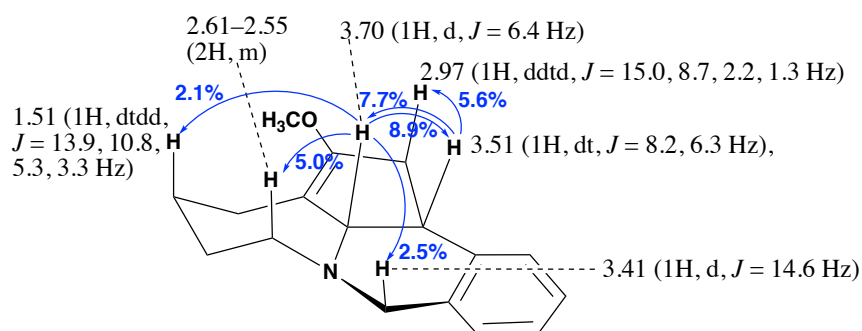


Figure S18. Difference NOE for compound **16** (CDCl_3 , 500 MHz).

Compound 17. Pale yellow oil; IR (film) 2920, 2845, 1680 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 7.19–7.15 (2H, m), 7.11–7.06 (2H, m), 4.78 (1H, dd, $J = 2.9, 2.0$ Hz), 3.97 (1H, dt, $J = 8.8, 2.2$ Hz), 3.76 (1H, d, $J = 14.3$ Hz), 3.62 (1H, t, $J = 8.4$ Hz), 3.58 (3H, s), 3.54 (1H, d, $J = 14.3$ Hz), 3.08 (1H, m), 2.91–2.86 (2H, m), 2.04 (1H, ddd, $J = 12.8, 11.4, 6.4$ Hz), 1.86–1.80 (2H, m), 1.74–1.70 (2H, m), 1.29 (1H, tt, $J = 12.6, 6.7$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 162.0 (C), 138.8 (C), 134.6 (C), 127.3 (CH), 126.8 (CH), 126.3 (CH), 125.2 (CH), 94.8 (CH), 64.4 (CH), 59.0 (CH_2), 56.3 (CH_3), 52.7 (CH_2), 42.1 (CH), 41.8 (CH), 29.2 (CH_2), 27.1 (CH_2), 24.9 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{22}\text{NO}^+$ 256.1696, found 256.1690.

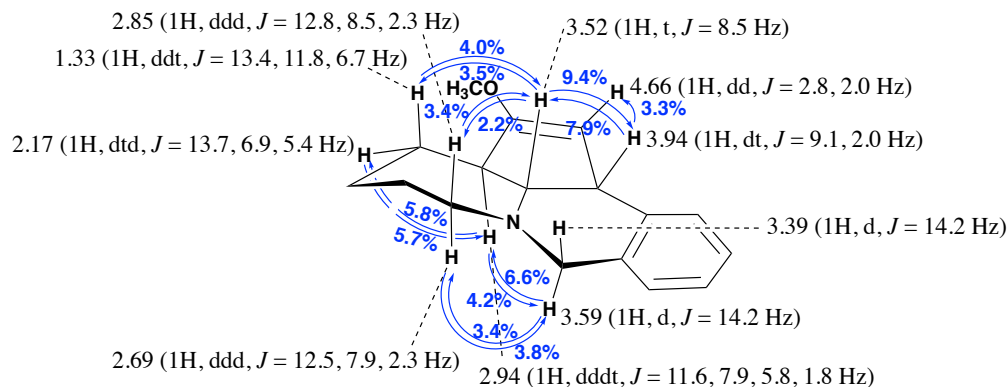
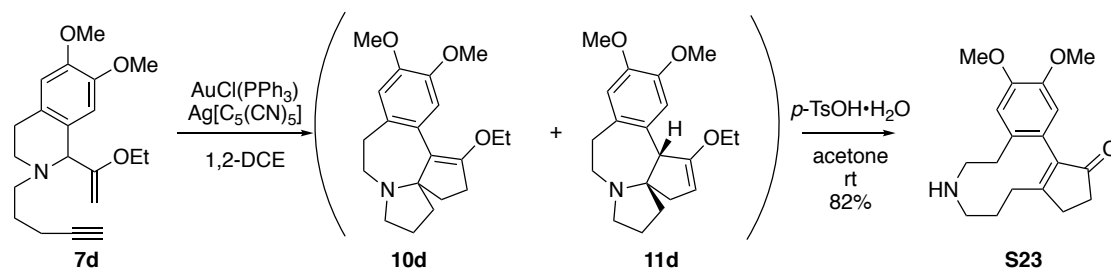


Figure S19. Difference NOE for compound **17** (C_6D_6 , 500 MHz).

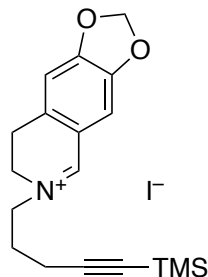
Attempted hydrolysis of vinyl ether **10d** and **11d** to ketone



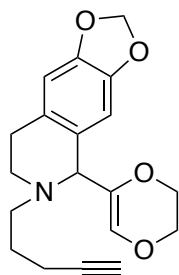
Macrocyclic ketone S23. To a solution of amine **7d** (146 mg, 0.44 mmol, 1.0 equiv) in 1,2-dichloroethane (5 mL) were added AuCl(PPh₃) (2.7 mg, 0.005 mmol, 1 mol%) and Ag[C₅(CN)₅] (1.9 mg, 0.006 mmol, 1 mol%). The reaction mixture was stirred at 80 °C under dark condition for 23 h. After cooling to room temperature, the mixture was concentrated to afford a mixture containing **10d** and **11d**, which was used in the next hydrolysis step without further purification. A solution of the mixture of **10d** and **11d** in acetone (8 mL) was added *p*-toluenesulfonic acid (449 mg, 2.61 mmol), and the reaction mixture was stirred at room temperature for 23 h. The reaction was quenched with Et₃N and the resulting mixture was concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (Et₂O) afforded conjugate ketone **S23** (109 mg, 82%) as a pale yellow oil. IR (film) 3352, 2923, 2852, 1695, 1513 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.75 (1H, s), 6.40 (1H, s), 3.87 (3H, s), 3.82 (3H, s), 2.87 (1H, td, *J* = 13.2, 3.5 Hz), 2.84–2.74 (3H, m), 2.72 (1H, dt, *J* = 11.5, 3.1 Hz), 2.59–2.52 (4H, m), 2.41 (1H, m), 2.25–2.20 (2H, m), 1.77 (1H, m), 1.69 (1H, m); ¹³C NMR (CDCl₃, 600 MHz) δ 208.3 (C), 175.9 (C), 149.0 (C), 147.3 (C), 144.2 (C), 131.7 (C), 125.5 (C), 111.8 (CH), 111.7 (CH), 55.7 (CH₃), 55.6 (CH₃), 49.0 (CH₂), 42.2 (CH₂),

34.6 (CH₂), 30.3 (CH₂), 28.4 (CH₂), 26.9 (CH₂), 24.8 (CH₂); HRDARTMS m/z [M+H]⁺ calcd for C₁₈H₂₄NO₃⁺ 302.1751, found 302.1748.

Synthesis of (–)-Cephalotaxine (Scheme 3)



Iminium 20. A mixture of 6,7-methylenedioxy-3,4-dihydroisoquinoline (Norhydrastinine; **18**)¹² (2.46 g, 14.0 mmol) and (5-iodo-1-pentynyl)trimethylsilane (**19**)⁸ (5.60 g, 21.1 mmol, 1.5 equiv) in CH₃CN (35 mL) was stirred at 65 °C for 21 h. After cooling to room temperature, Et₂O (50 mL) was added, and resulting precipitation was collected with suction filtration to afford iminium **20** (5.58 g, 97%) as a pale yellow powder. Mp 173–175 °C; IR (KBr) 2958, 2930, 2173, 1660 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 9.84 (1H, s), 7.49 (1H, s), 6.85 (1H, s), 6.15 (2H, s), 4.34 (2H, t, *J* = 6.9 Hz), 4.08 (2H, t, *J* = 8.3 Hz), 3.31 (2H, t, *J* = 8.3 Hz), 2.50 (2H, t, *J* = 6.6 Hz), 2.17 (2H, quintet, *J* = 6.8 Hz), 0.07 (9H, s); ¹³C NMR (CDCl₃, 150 MHz) δ 164.6, 156.2, 147.8, 134.8, 118.3, 112.7, 108.9, 104.4, 103.1, 87.0, 59.5, 48.3, 26.0, 25.8, 17.1, –0.1; HRESIMS m/z [M–I]⁺ calcd for C₁₈H₂₄NO₂Si⁺ 314.1571, found 314.1573.



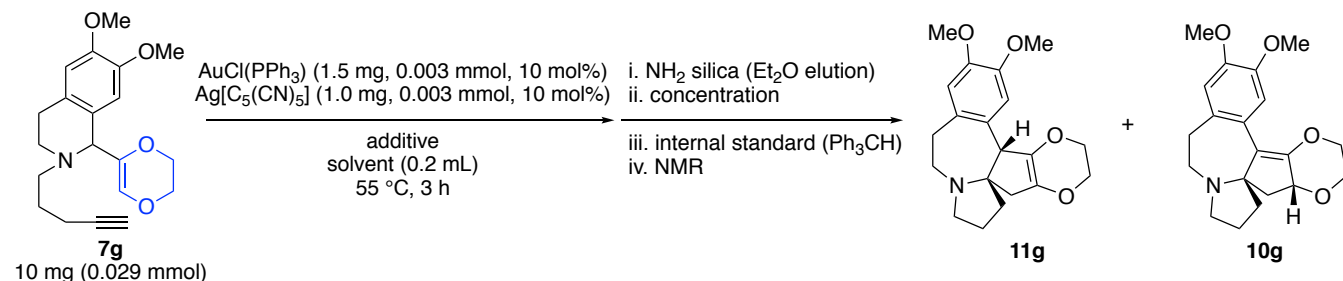
Dioxene adduct 22. To a solution of 1,4-dioxene (0.42 mL, 5.3 mmol, 1.5 equiv) in THF (30 mL) at –80 °C was added *tert*-butyllithium (1.60 M solution in *n*-pentane, 3.13 mL, 5.0 mmol, 1.4 equiv). The reaction mixture was stirred at –80 °C for 10 min, and then warmed to 0 °C. After stirring for 5 min at

0 °C, the resulting solution of (5,6-dihydro-1,4-dioxin-2-yl)lithium was added to a suspension of cyclic iminium **20** (1.545 g, 3.5 mmol, 1.0 equiv) in THF (15 mL) at –80 °C. The reaction mixture was stirred for –80 °C for 30 min. The reaction was quenched with brine, and the resulting mixture was extracted with Et₂O. The organic layer was dried over Na₂SO₄, filtered, and concentrated under reduced pressure to afford an addition product (2.03 g), which was used in the desilylation step without further purification. To a solution of the addition product (2.03 g) in THF (35 mL) at 0 °C was added tetra-*n*-butylammonium fluoride (1 M solution in THF, 3.6 mL, 3.6 mmol, 1.03 equiv). The reaction mixture was stirred at 0 °C for 30 min, and the reaction was quenched with water. The resulting mixture was extracted with Et₂O, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on NH₂ silica (50% Et₂O in *n*-hexane) afforded **22** (1.047 g, 91%) as a pale yellow solid. Mp 101–103 °C; IR (film) 3289, 2953, 2926, 1676, 1484 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.74 (1H, s), 6.54 (1H, s), 6.01 (1H, s), 5.89 (2H, s), 4.10–4.00 (4H, m), 3.76 (1H, s), 3.19 (1H, dt, *J* = 11.6, 5.3 Hz), 2.91 (1H, dt, *J* = 12.9, 8.0, 7.2 Hz), 2.85 (1H, m), 2.68 (1H, dt, *J* = 16.1, 5.0 Hz), 2.57–2.52 (2H, m), 2.27 (1H, dtd, *J* = 16.9, 7.0, 2.8 Hz), 2.24 (1H, dtd, *J* = 16.9, 7.0, 2.8 Hz), 1.95 (1H, t, *J* = 2.7 Hz), 1.79–1.74 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 146.2, 145.8, 136.7, 128.8, 127.9, 126.7, 108.2, 107.1, 100.6, 84.5, 68.3, 64.6, 64.2, 63.8, 52.9, 47.2, 28.6, 25.8, 16.4; HRDARTMS *m/z* [M+H]⁺ calcd for C₁₉H₂₂NO₄⁺ 328.1543, found 328.1546.

Condition screening for the cascade reaction of dioxane adduct 7g to 11g. To a solution of tertiary amine **7g** (10 mg, 0.029 mmol) in solvent (0.2 mL) were added AuCl(PPh₃) (1.5 mg, 0.003 mmol, 10 mol%), Ag[C₅(CN)₅] (1.0 mg, 0.003 mmol, 10 mol%), and additive in Table S1. The reaction mixture was stirred at 55 °C for 3 h, and then cooled to room temperature. The mixture was filtered through NH₂ silica with aid of Et₂O and the filtrate was concentrated under reduced pressure. Internal standard Ph₃CH was added and the mixture was subjected to ¹H NMR analysis. Product yield of **11g**, **10g**, and recovered

7g were calculated based on integration values of Ph₃CH (5.55 ppm), **11g** (6.66 ppm), **10g** (6.71 ppm), and **7g** (6.74 ppm).

Table S2. Condition Screening



entry	solvent	additive	11g (%)	10g (%)	Others (%)	recovered 7g (%)	11g /(11g + 10g)
1 ^a	1,2-DCE		26	38			0.41
2	EtOAc		16	24			0.40
3	THF		11	14			0.44
4	CH ₃ CN		31	30			0.51
5 ^b	Tol		10	23			0.30
6	<i>i</i> -PrOH		15	8	48 ^c		0.65
7	<i>t</i> -BuOH		33	25		9	0.57
8	1,2-DCE	<i>i</i> -Pr ₂ NEt (0.02 mL)	9	10			0.47
9	1,2-DCE	2,6-di- <i>tert</i> -BuPy (0.02 mL)	12	14			0.46
10	1,2-DCE	2,5-diClPy (20 mg)	15	22			0.41
11	1,2-DCE	CF ₃ CH ₂ OH (20 mg)	24	10	23 ^d	3	0.71
12	1,2-DCE	2,6-di- <i>tert</i> -BuC ₆ H ₃ OH (20 mg)	20	26		4	0.43
13	1,2-DCE	PhCONH ₂ (20 mg)	27	23		3	0.54
14	1,2-DCE	CH ₃ SO ₂ NH ₂ (20 mg) ^e	30	23		3	0.57
15	1,2-DCE	TolSO ₂ NH ₂ (20 mg, 4 equiv)	40	24		4	0.63
16	1,2-DCE	PhCOOH (20 mg)	16	33		4	0.33
17	<i>t</i> -BuOH	TolSO ₂ NH ₂ ⁻ (4 equiv)	44	24		3	0.65
18	<i>t</i> -BuOH	TolSO ₂ NH ₂ (10 equiv)	48	24		3	0.67
19	<i>t</i> -BuOH	TolSO ₂ NH ₂ (2 equiv)	38	25		3	0.60
20	<i>t</i> -BuOH	TolSO ₂ NH ₂ (1 equiv)	44	34		6	0.56
21	<i>t</i> -BuOH	TolSO ₂ NH ₂ (0.4 equiv)	39	29		8	0.57
22	<i>t</i> -BuOH	HFIP (0.02 mL)	38	21		4	0.64

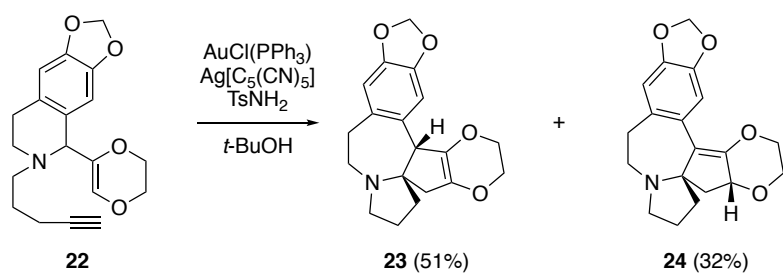
^a Isolation experiment in Table 2, entry 6. ^b Reaction time was 17 h.

^c Reaction with *i*-PrOH gave an unidentified byproduct in entry 6.

^d Reaction with TFE gave an unidentified byproduct in entry 11. ^e MsNH₂ did not dissolve in entry 14.

First, we examined the effect of solvents on the cascade reaction (entries 1–7). Aprotic solvents, including 1,2-DCE, EtOAc, THF, CH₃CN, and toluene, yielded conjugated product **10g** as a major isomer (entries 1–5). *i*-PrOH preferentially yielded the desired unconjugated isomer **11g**; however, ¹H NMR analysis revealed a high amount of an unidentified byproduct having an isopropyl group (entry 6). *tert*-BuOH slightly improved the selectivity for **11g** compared to the aprotic solvents (entry 7). Basic additives, such as *i*-Pr₂NEt, 2,6-di-*tert*-butylpyridine, and 2,5-dichloropyridine, did not improve the ratio of **11g** and decreased the product yield (entries 8–10). A weak-acid additive, CF₃CH₂OH (pK_a¹³ 12.5), increased the ratio of **11g**; however, an unidentified byproduct was observed (entry 11). 2,6-Di-*tert*-butylphenol (pK_a¹³

12.2), a bulky acid with an acidity similar to that of $\text{CF}_3\text{CH}_2\text{OH}$, did not improve the ratio of **11g** (entry 12). MsNH_2 (pK_a^{13} 10.9) was not completely dissolved in 1,2-DCE (entry 13); therefore, we investigated TsNH_2 (pK_a^{13} 10.2), and the best yield of **11g** was obtained without the formation of any byproduct, except for isomer **10g** (entry 15). Benzamide, which has a weaker acidity (pK_a^{13} 16.0), afforded **11g** and **10g** in almost 1:1 ratio (entry 13), whereas, benzoic acid, with a lower pK_a (pK_a^{13} 4.2), decreased both the product yield and ratio of **11g** (entry 16). Based on these results, *tert*-BuOH and TsNH_2 were the best solvent and additive, respectively. We further investigated the amount of TsNH_2 (entries 17–21). The runs with four or ten equivalents of TsNH_2 yielded **11g** and **10g** in almost 2:1 ratio (entries 17 and 18). With less TsNH_2 , the selectivity of **11g** decreased (entries 19–21). 1,1,1,3,3,3-Hexafluoro-2-propanol (HFIP) (pK_a^{13} 9.8), which has pK_a similar to that of TsNH_2 , gave a slightly lower yield of **11g** and **10g** (entry 22). In conclusion, using four equivalents of TsNH_2 as an additive in *tert*-BuOH (entry 17) is the best condition.



Cascade reaction with 22. To a solution of cyclic amine **22** (1.12 g, 3.42 mmol, 1.0 equiv) and *p*-toluenesulfonamide (2.34 g, 13.7 mmol, 4.0 equiv) in *tert*-butanol (17.1 mL) were added $\text{AuCl}(\text{PPh}_3)$ (33.9 mg, 0.069 mmol, 2 mol%) and $\text{Ag}[\text{C}_5(\text{CN})_5]$ (20.4 mg, 0.069 mmol, 2 mol%). The reaction mixture was stirred under dark condition at 50 °C for 5.5 h. After cooling to room temperature, the reaction mixture was filtered through NH_2 silica gel pad, and the filtrate was concentrated under reduced pressure. Flash chromatography on alumina (Et_2O) provided **23** (567 mg, 51%) and **24** (359 mg, 32%).

Compound 23. Pale yellow amorphous solid; IR (film) 2928, 2873, 2798, 1719, 1504, 1486 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 6.64 (1H, s), 6.61 (1H, s), 5.89 (1H, d, $J = 1.7$ Hz), 5.87 (1H, d, $J = 1.7$ Hz),

4.13–4.02 (4H, m), 3.91 (1H, dd, $J = 2.9, 1.2$ Hz), 3.35 (1H, ddd, $J = 13.8, 13.0, 7.3$ Hz), 3.07 (1H, td, $J = 9.0, 4.9$ Hz), 2.93 (1H, ddd, $J = 13.0, 11.5, 6.7$ Hz), 2.76 (1H, dd, $J = 15.4, 2.9$ Hz), 2.61 (1H, dd, $J = 11.5, 7.3$ Hz), 2.42 (1H, ddd, $J = 10.3, 9.0, 6.4$ Hz), 2.41 (1H, dd, $J = 13.8, 6.7$ Hz), 2.02 (1H, dd, $J = 15.4, 1.2$ Hz), 2.01 (1H, ddd, $J = 11.9, 8.1, 2.6$ Hz), 1.94 (1H, dt, $J = 11.6, 10.0$ Hz), 1.80–1.69 (2H, m); ^{13}C NMR (CDCl_3 , 150 MHz) δ 146.7 (C), 146.0 (C), 132.1 (C), 129.4 (C), 129.2 (C), 129.0 (C), 111.6 (CH), 109.9 (CH), 100.8 (CH_2), 65.4 (CH_2), 65.3 (CH_2), 63.2 (C), 58.0 (CH), 53.2 (CH_2), 49.1 (CH_2), 43.4 (CH_2), 30.6 (CH_2), 30.4 (CH_2), 19.6 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{22}\text{NO}_4^+$ 328.1543, found 328.1544.

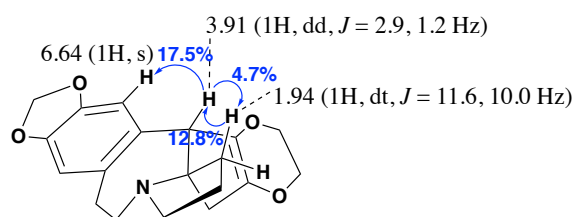


Figure S20. Difference NOE for **23** (CDCl_3 , 500 MHz).

Conjugated isomer 24. Pale yellow amorphous solid; IR (film) 2958, 2932, 2895, 2857, 1669, 1504, 1484 cm^{-1} ; ^1H NMR (CDCl_3 , 600 MHz) δ 6.67 (1H, s), 6.62 (1H, s), 5.92 (1H, d, $J = 1.5$ Hz), 5.89 (1H, d, $J = 1.5$ Hz), 4.34 (1H, dd, $J = 7.8, 6.8$ Hz), 3.98–3.85 (4H, m), 3.54 (1H, ddd, $J = 14.7, 11.5, 4.2$ Hz), 3.28 (1H, ddd, $J = 17.4, 11.5, 5.5$ Hz), 3.01 (1H, ddd, $J = 14.7, 5.5, 3.0$ Hz), 2.95 (1H, ddd, $J = 17.4, 4.2, 3.0$ Hz), 2.84 (1H, ddd, $J = 10.0, 8.4, 5.4$ Hz), 2.79 (1H, dd, $J = 8.4, 6.3$ Hz), 2.24 (1H, dd, $J = 11.9, 6.8$ Hz), 1.80–1.68 (4H, m), 1.59 (1H, tdd, $J = 12.3, 7.3, 0.7$ Hz); ^{13}C NMR (CDCl_3 , 600 MHz) δ 147.7 (C), 146.8 (C), 145.3 (C), 131.4 (C), 124.4 (C), 122.9 (C), 110.6 (CH), 109.8 (CH), 100.9 (CH_2), 74.9 (CH), 71.7 (C), 67.7 (CH_2), 65.0 (CH_2), 48.6 (CH_2), 44.0 (CH_2), 42.1 (CH_2), 36.3 (CH_2), 32.7 (CH_2), 23.9 (CH_2); HRDARTMS m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{22}\text{NO}_4^+$ 328.1543, found 328.1541.

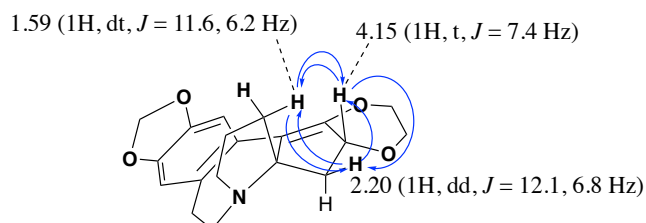
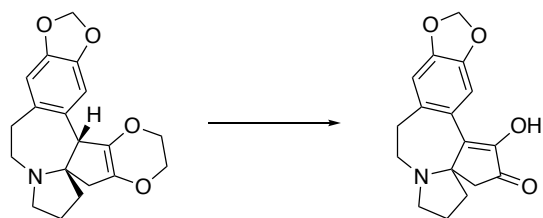
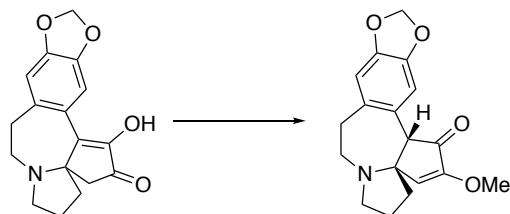


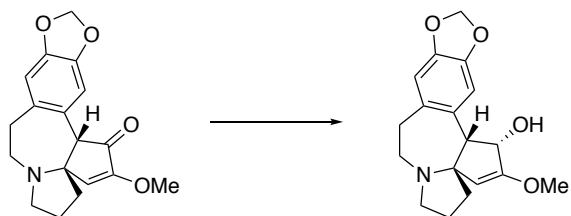
Figure S21. 1D Gradient NOESY for **24** (C₆D₆, 600 MHz).



(±)-Demethylcephalotaxinone (25). To a suspension of hexacyclic compound **23** (561 mg, 1.71 mmol) in THF/H₂O (1:1) (20 mL) at 0 °C was added TsOH·H₂O (400 mg, 2.10 mmol, 1.23 equiv). After 3 minutes, the mixture became a colorless solution and then *m*-CPBA (stabilized with 30% water, 444 mg, 1.80 mmol, 1.05 equiv) was added. The reaction mixture was stirred at 0 °C for 1 h and the reaction was quenched with saturated aqueous Na₂S₂O₃ solution and saturated aqueous NaHCO₃ solution. The resulting mixture was extracted with CH₂Cl₂, and the organic layer was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on silica gel (10% MeOH in CH₂Cl₂) afforded (±)-demethylcephalotaxinone (**25**) (447 mg, 87%) as a pale yellow amorphous solid. IR (film) 2894, 1698, 1487, 1226 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.92 (1H, s), 6.68 (1H, s), 5.97 (1H, d, *J* = 1.5 Hz), 5.96 (1H, d, *J* = 1.5 Hz), 4.70 (1H, br s), 3.39–3.31 (2H, m), 3.01 (1H, m), 2.96–2.86 (3H, m), 2.60 (1H, d, *J* = 18.5 Hz), 2.56 (1H, d, *J* = 18.5 Hz), 1.90–1.83 (3H, m), 1.70 (1H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 200.7 (C), 148.1 (C), 147.6 (C), 145.8 (C), 145.0 (C), 132.5 (C), 123.9 (C), 109.9 (CH), 109.7 (CH), 101.3 (CH₂), 70.6 (C), 51.0 (CH₂), 49.4 (CH₂), 44.7 (CH₂), 39.0 (CH₂), 32.7 (CH₂), 24.3 (CH₂); HRDARTMS *m/z* [M+H]⁺ calcd for C₁₇H₁₈NO₄⁺ 300.1230, found 300.1227.



(±)-Cephalotaxinone. To a solution of **24** (105 mg, 0.351 mmol, 1.0 equiv) in 1,2-dichloroethane (35.1 mL, 0.01 M) were added CH(OMe)₃ (230 μL, 2.10 mmol, 6.0 equiv) and *p*-TsOH·H₂O (134 mg, 0.702 mmol, 2.0 equiv), and the reaction mixture was stirred at 50 °C for 2.0 h. After cooling to room temperature, the reaction was quenched with Et₃N and the mixture was concentrated under reduced pressure. Flash chromatography on NH₂ silica gel (Et₂O) afforded (±)-cephalotaxinone (63 mg, 57%) as a colorless solid; mp 182–184 °C (lit¹⁴ 178–180 °C); IR (film) 2936, 2884, 2802, 1720, 1625, 1487, 1229, 1103 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.70 (1H, s), 6.64 (1H, s), 6.40 (1H, s), 5.92 (1H, d, *J* = 1.5 Hz), 5.90 (1H, d, *J* = 1.5 Hz), 3.80 (3H, s), 3.53 (1H, s), 3.09 (1H, td, *J* = 8.6, 5.1 Hz), 2.91 (1H, q, *J* = 10.2 Hz), 2.69 (1H, q, *J* = 8.6 Hz), 2.53 (1H, dt, *J* = 11.2, 4.2 Hz), 2.44 (2H, dd, *J* = 9.6, 4.1 Hz), 2.10 (1H, dt, *J* = 12.1, 10.1 Hz), 1.96 (1H, ddd, *J* = 12.2, 7.2, 4.8 Hz), 1.89–1.83 (2H, m); ¹³C NMR (CDCl₃, 150 MHz) δ 200.8, 158.2, 147.2, 146.2, 130.6, 128.4, 123.7, 112.4, 110.2, 101.0, 65.3, 60.7, 57.2, 52.9, 47.6, 38.9, 31.4, 20.0. HRDARTMS *m/z* [M+H]⁺ calcd for C₁₈H₂₀NO₄⁺ 314.1387, found 314.1387.



(–)-Cephalotaxine. The reaction was carried out according to the report by Ju and Beaudry in somewhat larger scale.¹⁵

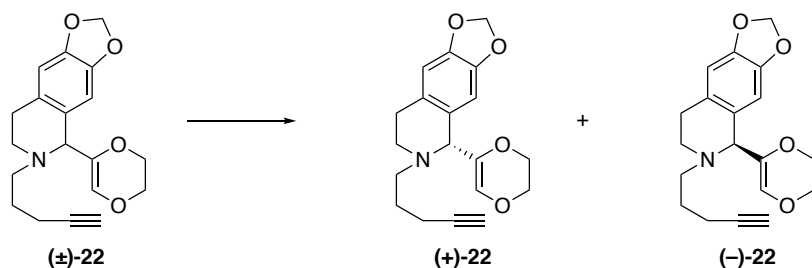
A 7-mL thick-walled reaction vessel was charged with a mixture of (±)-cephalotaxinone (60 mg, 0.19 mmol) and RuCl(*p*-cymene)-(S,S)-TsDPEN (5.0 mg, 0.0079 mmol, 4 mol%) in Et₃N (0.67 mL). Formic acid (0.13 mL) was added dropwise and the reaction tube was sealed with a Teflon cap. The resulting biphasic mixture was vigorously stirred at 40 °C for 45 h and then cooled to room temperature. The mixture was poured into saturated aqueous NaHCO₃ solution and the resulting mixture was extracted with EtOAc. The extract was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on silica gel (MeOH/CH₂Cl₂ (5:95) → Et₃N/MeOH/CH₂Cl₂ (1:10:90))

afforded (-)-cephalotaxine (25 mg, 41%, 99% ee) and recovered (+)-cephalotaxinone (31 mg, 52% recovered, 89% ee).

(-)-Cephalotaxine. Colorless solid; Mp 133–135 °C (lit¹⁶ 134–136 °C); 99% ee determined by HPLC (Daicel Chiralcel OD-3, 0.46 x 25 cm, 40% *i*-PrOH/*n*-Hexane, 1.0 mL/min, 254 nm. Major 5.70 min, Minor 6.51 min); $[\alpha]_D^{22}$ -182.1 (*c* 0.73, CHCl₃) (lit¹⁶ -189 (*c* 0.51, CHCl₃)); IR (film) 3541, 2933, 2880, 2804, 1651, 1486, 1222 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ 6.67 (1H, s), 6.64 (1H, s), 5.91 (1H, d, *J* = 1.5 Hz), 5.90 (1H, d, *J* = 1.5 Hz), 4.93 (1H, s), 4.76 (1H, dd, *J* = 9.4, 2.9 Hz), 3.73 (3H, s), 3.68 (1H, d, *J* = 9.5 Hz), 3.35 (1H, ddd, *J* = 14.4, 12.2, 7.9 Hz) 3.08 (1H, ddd, *J* = 9.5, 8.2, 4.5 Hz) 2.92 (1H, ddd, *J* = 12.2, 11.0, 7.2 Hz) 2.60 (1H, dd, *J* = 11.0, 7.9 Hz), 2.57 (1H, dd, *J* = 9.5, 7.3 Hz), 2.35 (1H, dd, *J* = 14.4, 7.2 Hz) 2.01 (1H, dt, *J* = 12.2, 9.6 Hz) 1.87 (1H, ddd, *J* = 12.2, 8.3, 4.0 Hz) 1.79–1.69 (2H, m) 1.62 (1H, d, *J* = 3.5 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ 160.4, 146.9, 146.1, 134.3, 128.0, 112.6, 110.3, 100.9, 97.6, 73.3, 70.5, 58.0, 57.2, 53.9, 48.6, 43.6, 31.7, 20.3. HRDARTMS *m/z* [M+H]⁺ calcd for C₁₈H₂₂NO₄⁺ 316.1543, found 316.1547.

(+)-Cephalotaxinone. Pale yellow amorphous solid; 89% ee determined by HPLC (Daicel Chiralcel OD-3, 0.46 x 25 cm, 40% *i*-PrOH/*n*-Hexane, 1.0 mL/min, 254 nm. Major 8.82 min, Minor 7.22 min); $[\alpha]_D^{21}$ +97.6 (*c* 1.03, CHCl₃) (lit¹⁷ -109.3 (CHCl₃)).

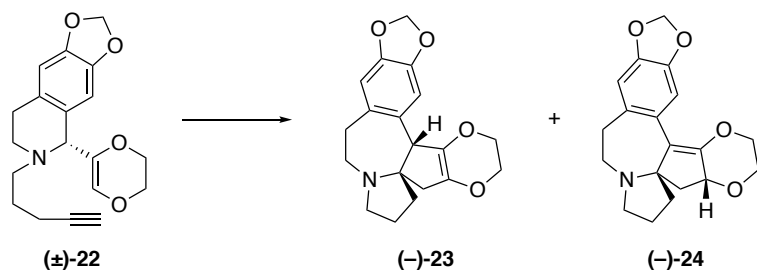
Chiral separation of racemic **22** and cascade reaction with optical enriched (+)-**22**



Separation of racemic **22 by chiral-phase HPLC.** Racemic **22** was separated by chiral-phase HPLC (20% *i*-PrOH/*n*-hexane, Chiralcel OD, 10 μm, 1.0 cm x 25 cm, 5 mL/min, 254 nm, 6.7 min for (+)-**22** and 14.4 min for (-)-**22**). From racemic **22** (151 mg), (+)-**22** (98% ee, 56 mg) and (-)-**22** (87% ee, 58 mg) were obtained.

(+)-**22**. Pale yellow oil; 98% ee determined by HPLC (20% *i*-PrOH/*n*-hexane, Chiralcel OD-3, 0.25 cm x 25 cm, 1 mL/min, 254 nm). $[\alpha]^{23}_D +38.0$ (*c* 0.64, CHCl₃).

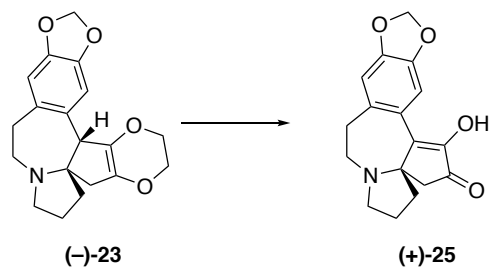
(-)-**22**. Pale yellow oil; 87% ee determined by HPLC (20% *i*-PrOH/*n*-hexane, Chiralcel OD-3, 0.25 cm x 25 cm, 1 mL/min, 254 nm). $[\alpha]^{23}_D -32.0$ (*c* 0.93, CHCl₃).



Cascade reaction with (+)-22. To a solution of cyclic amine (+)-**22** (56 mg, 0.17 mmol, 1.0 equiv) and *p*-toluenesulfonamide (117 mg, 0.681 mmol, 4.0 equiv) in *tert*-butanol (0.85 mL) were added AuCl(PPh₃) (1.7 mg, 0.0034 mmol, 2 mol%) and Ag[C₅(CN)₅] (1.0 mg, 0.0034 mmol, 2 mol%). The reaction mixture was stirred under dark condition at 50 °C for 2.5 h. After cooling to room temperature, the reaction mixture was filtered through NH₂ silica gel pad, and the filtrate was concentrated under reduced pressure. Flash chromatography on alumina (Et₂O) provided (-)-**23** (26.7 mg, 48%) and (-)-**24** (17.0 mg, 31%).

(-)-**23**. 98% ee determined by HPLC (30% *i*-PrOH/*n*-hexane, Chiralcel OD-3, 1.0 mL/min, 254 nm, major 9.4 min and minor 7.1 min); mp 142–147 °C; $[\alpha]^{24}_D -87.1$ (*c* 2.18, CHCl₃).

(-)-**24**. 96% ee determined by HPLC (50% *i*-PrOH/*n*-hexane, Chiralcel OD-3, 1.0 mL/min, 254 nm, major 12.7 min and minor 5.4 min); Pale yellow amorphous solid; $[\alpha]^{24}_D -6.9$ (*c* 1.45, CHCl₃).

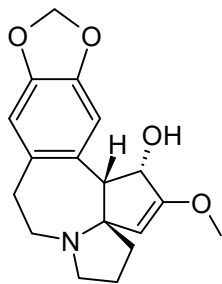


Oxidation with (–)-23. To a suspension of hexacyclic compound (–)-**23** (10.8 mg, 0.0330 mmol) in THF/H₂O (1:1) (0.5 mL) at 0 °C was added TsOH·H₂O (13 mg, 0.066 mmol, 2.0 equiv). After 3 minutes, the mixture became a colorless solution and then *m*-CPBA (stabilized with 30% water, 16 mg, 0.066 mmol, 2.0 equiv) was added. The reaction mixture was stirred at 0 °C for 1.0 h and the reaction was quenched with saturated aqueous Na₂S₂O₃ solution and saturated aqueous NaHCO₃ solution. The resulting mixture was extracted with CH₂Cl₂, was washed with brine, dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash chromatography on silica gel (10% MeOH in CH₂Cl₂) afforded enol (+)-**25** (8.8 mg, 89%) as a pale yellow amorphous solid. [α]_D²³ +382.5 (*c* 0.53, CHCl₃) (lit¹⁷ +371.9 (*c* 0.42, CHCl₃)).

References

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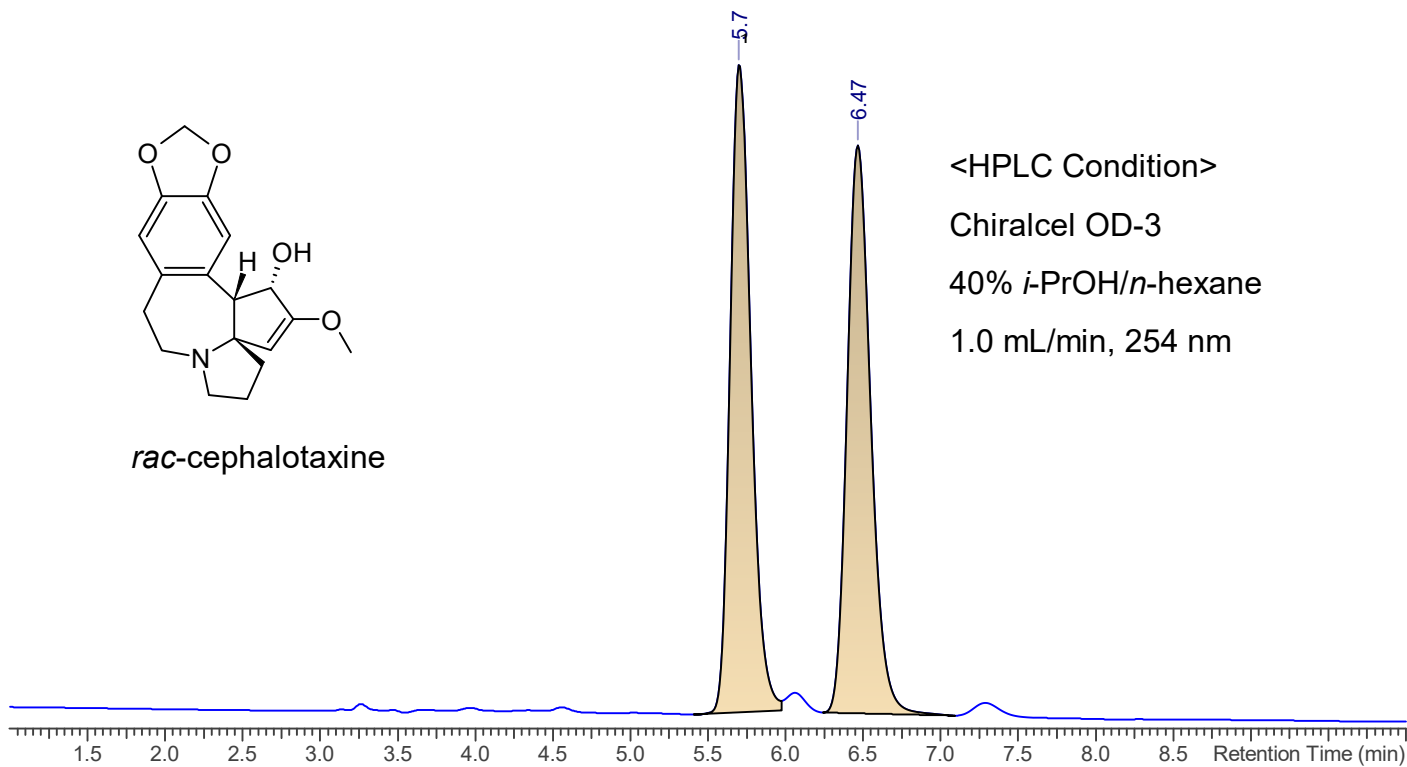
rac-cephalotaxine

<HPLC Condition>

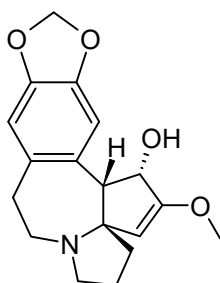
Chiralcel OD-3

40% *i*-PrOH/*n*-hexane

1.0 mL/min, 254 nm

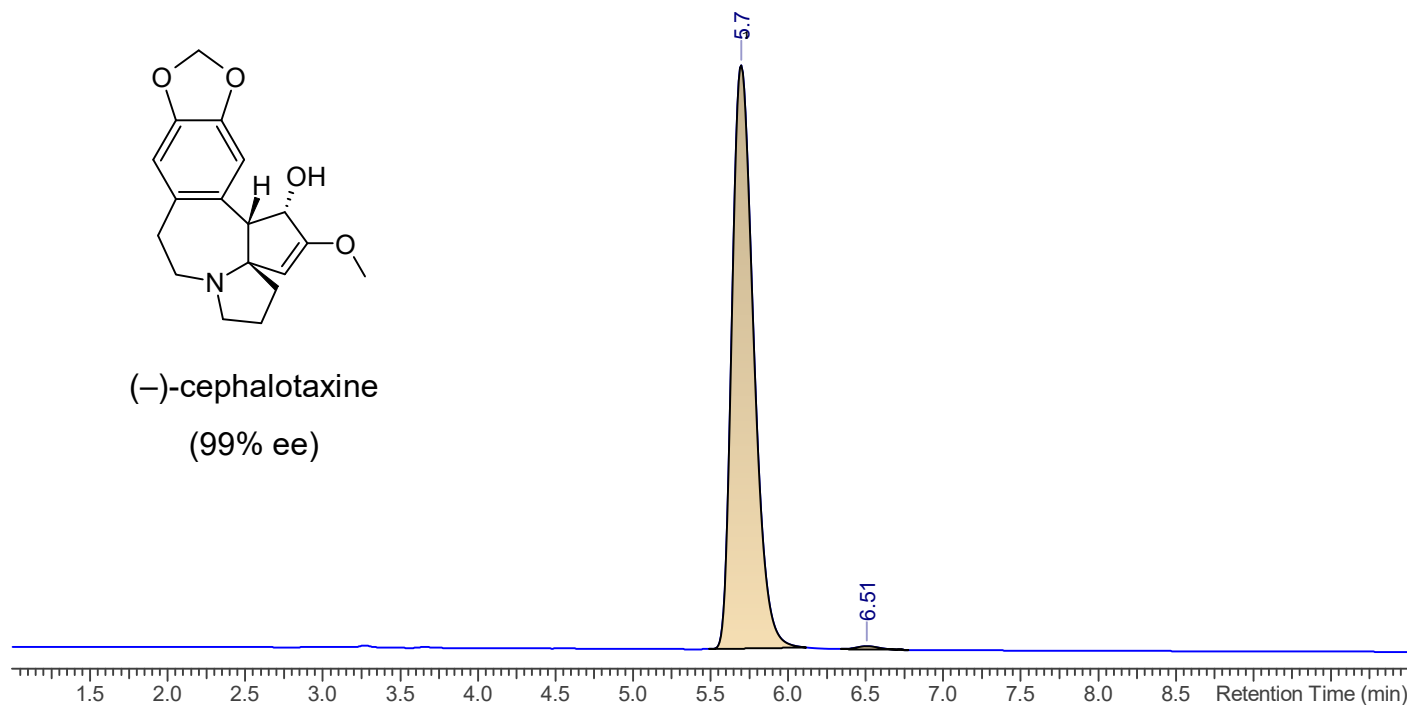


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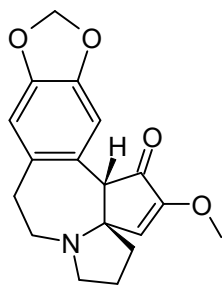


(-)-cephalotaxine

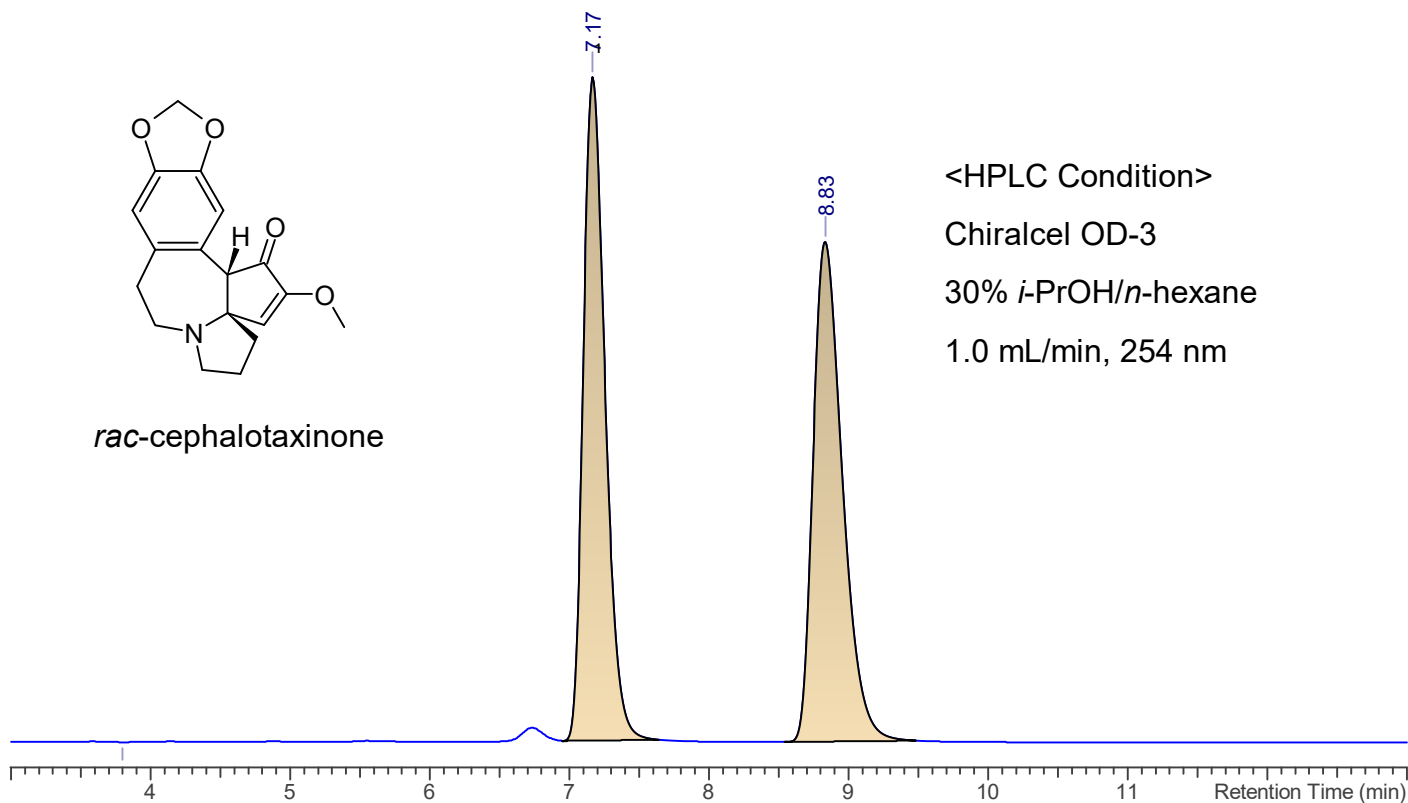
(99% ee)



No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	6.51	31071900.000	0.571	0.268
2	5.70	5410529792.000	99.429	0.253

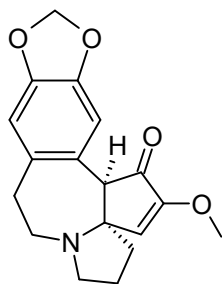


rac-cephalotaxinone

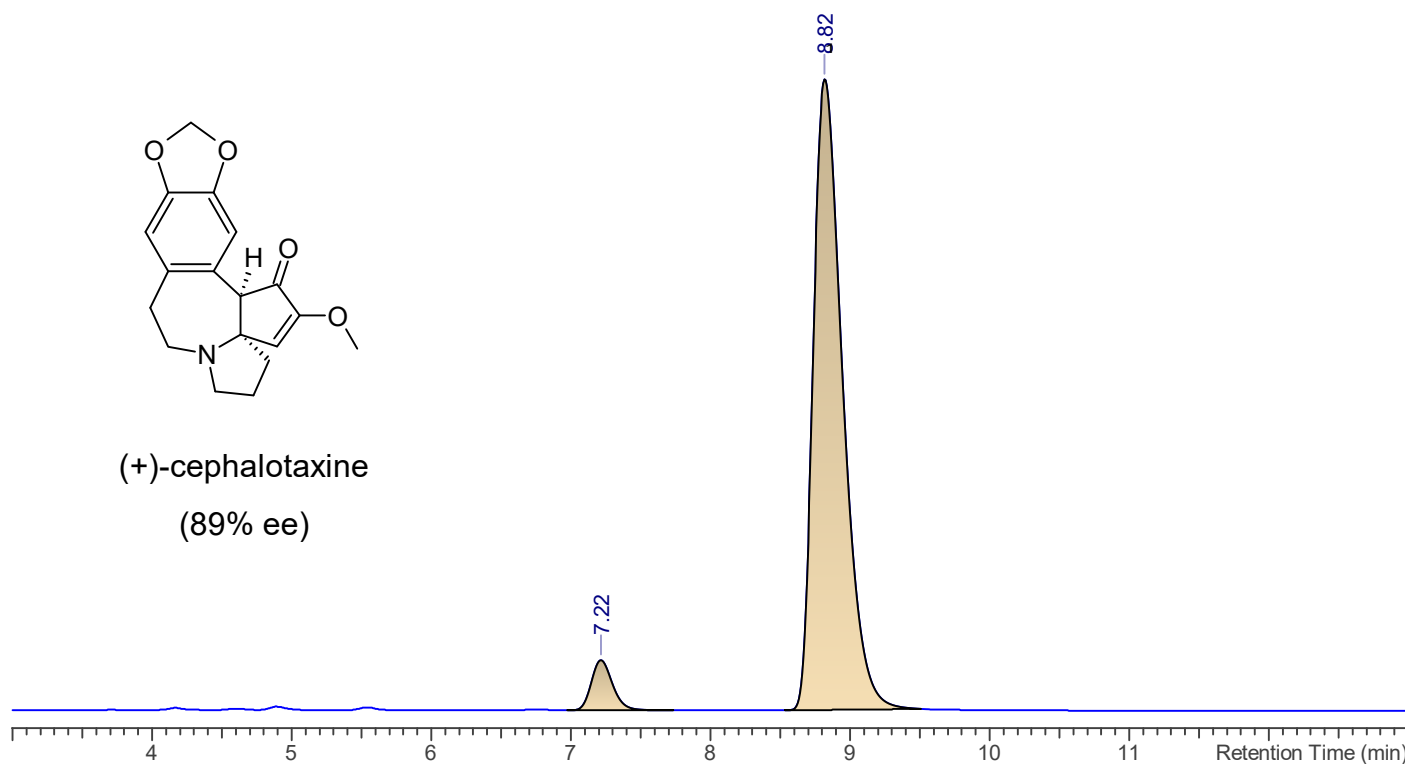


<HPLC Condition>
 Chiralcel OD-3
 30% *i*-PrOH/*n*-hexane
 1.0 mL/min, 254 nm

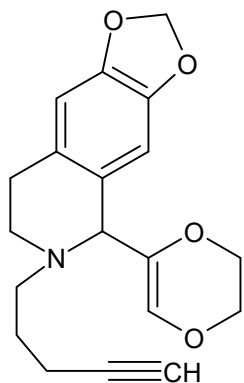
No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	7.17	8281185792.000	49.678	0.284
2	8.83	8388433920.000	50.322	0.380



(+)-cephalotaxine
 (89% ee)



No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	7.22	727704256.000	5.414	0.273
2	8.82	12713650176.000	94.586	0.381



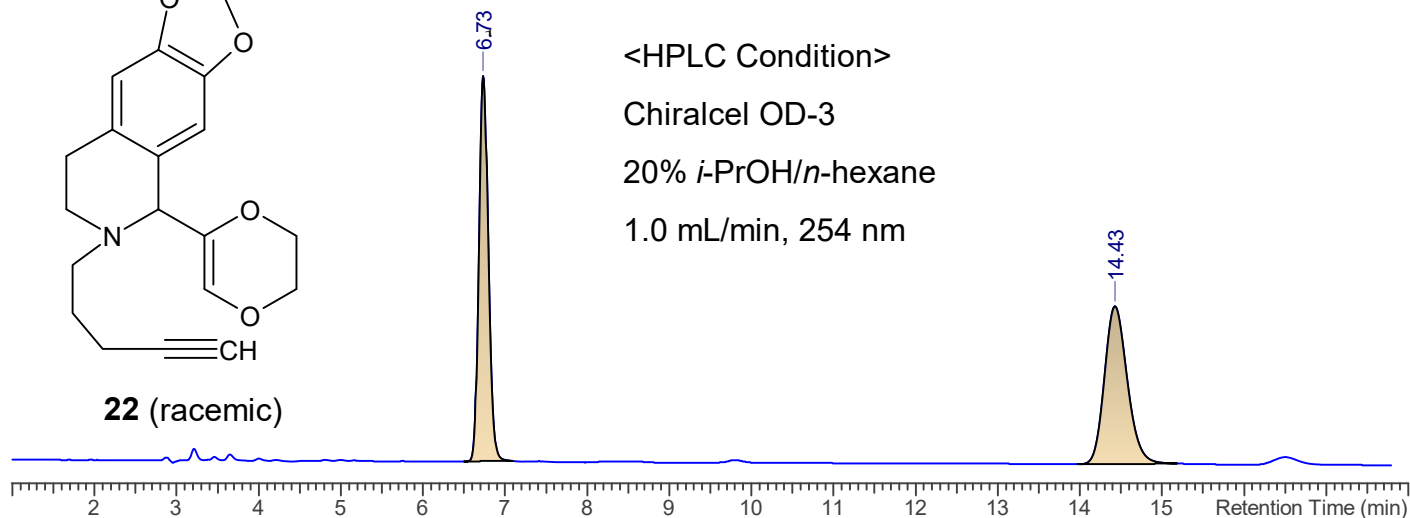
22 (racemic)

<HPLC Condition>

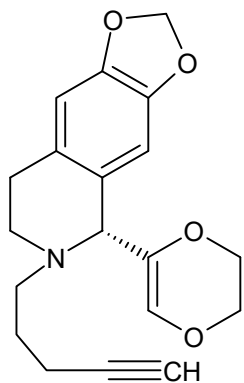
Chiralcel OD-3

20% *i*-PrOH/*n*-hexane

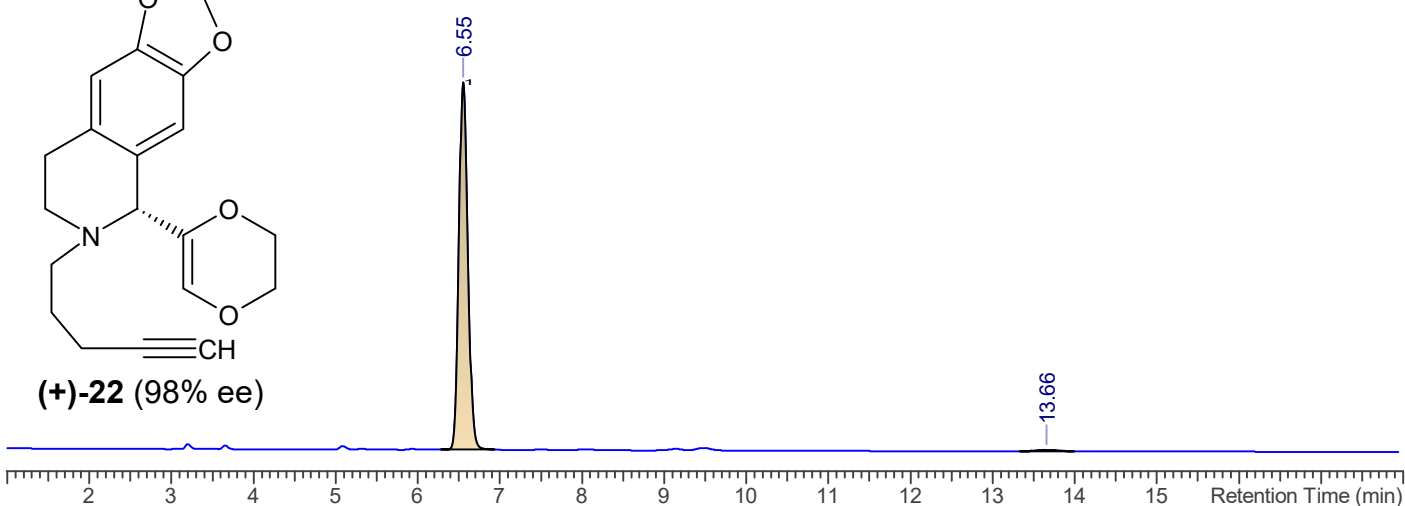
1.0 mL/min, 254 nm



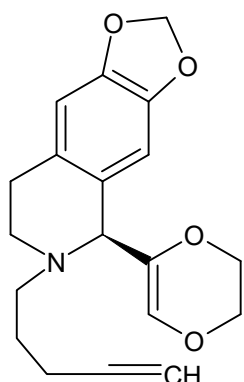
No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	14.43	251089248.047	49.555	0.502
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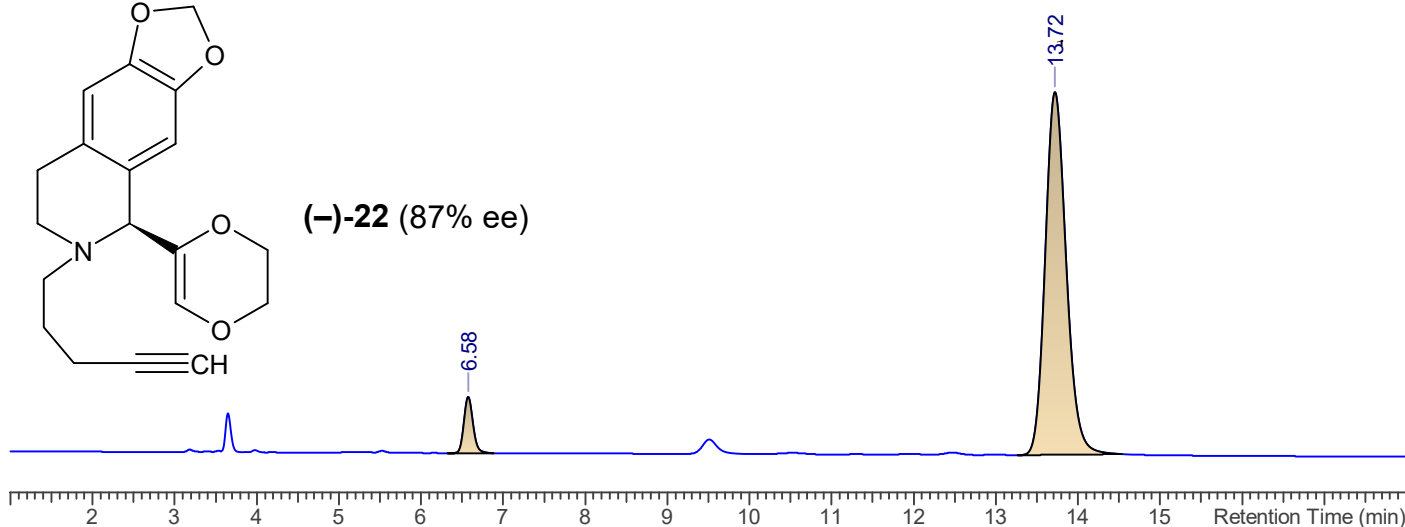
(+)-22 (98% ee)



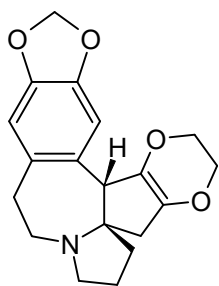
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1	13.66	88376982.422	0.920	0.456
2	6.55	9515654062.500	99.080	0.200



(-)-22 (87% ee)



No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	6.57	196093828.125	6.310	0.199
2	13.72	2911813593.750	93.690	0.460



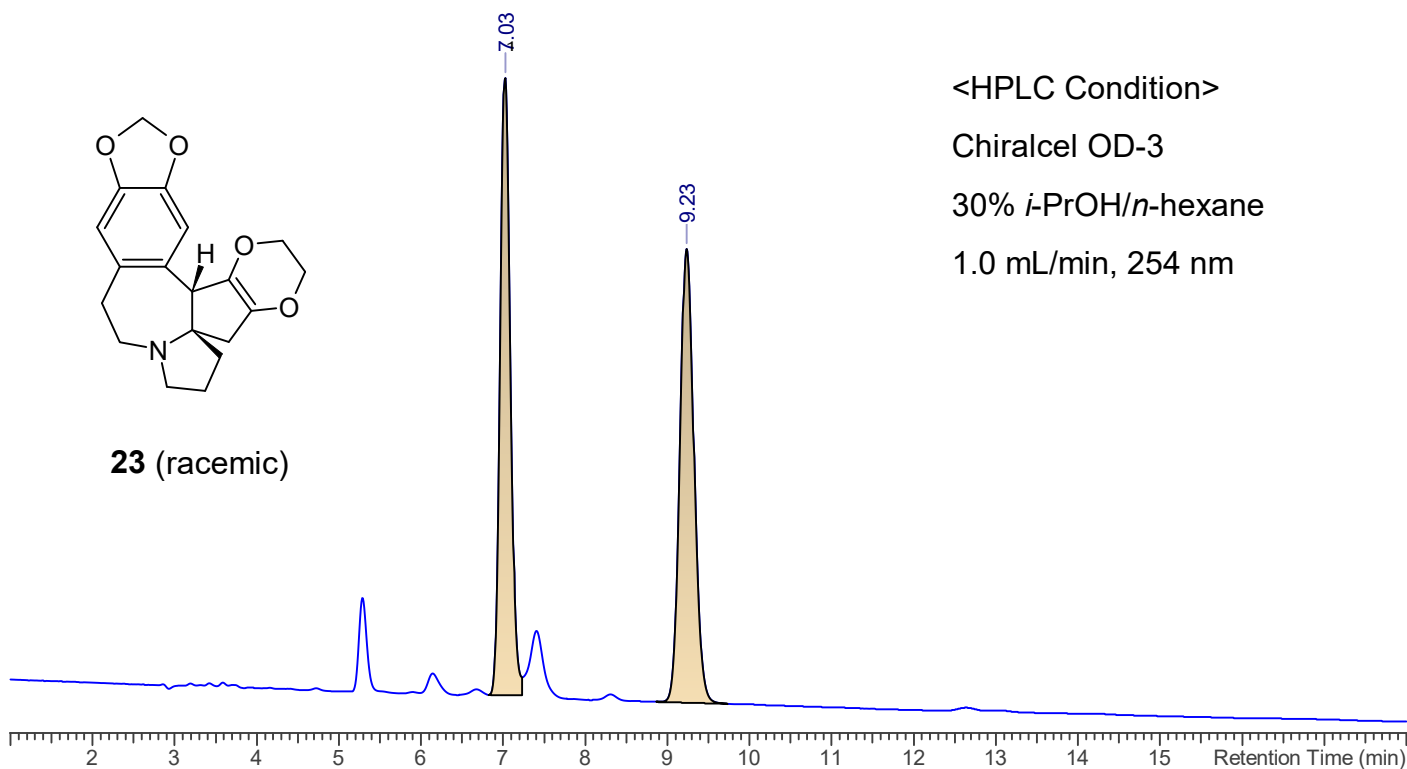
23 (racemic)

<HPLC Condition>

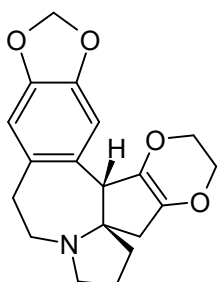
Chiralcel OD-3

30% *i*-PrOH/*n*-hexane

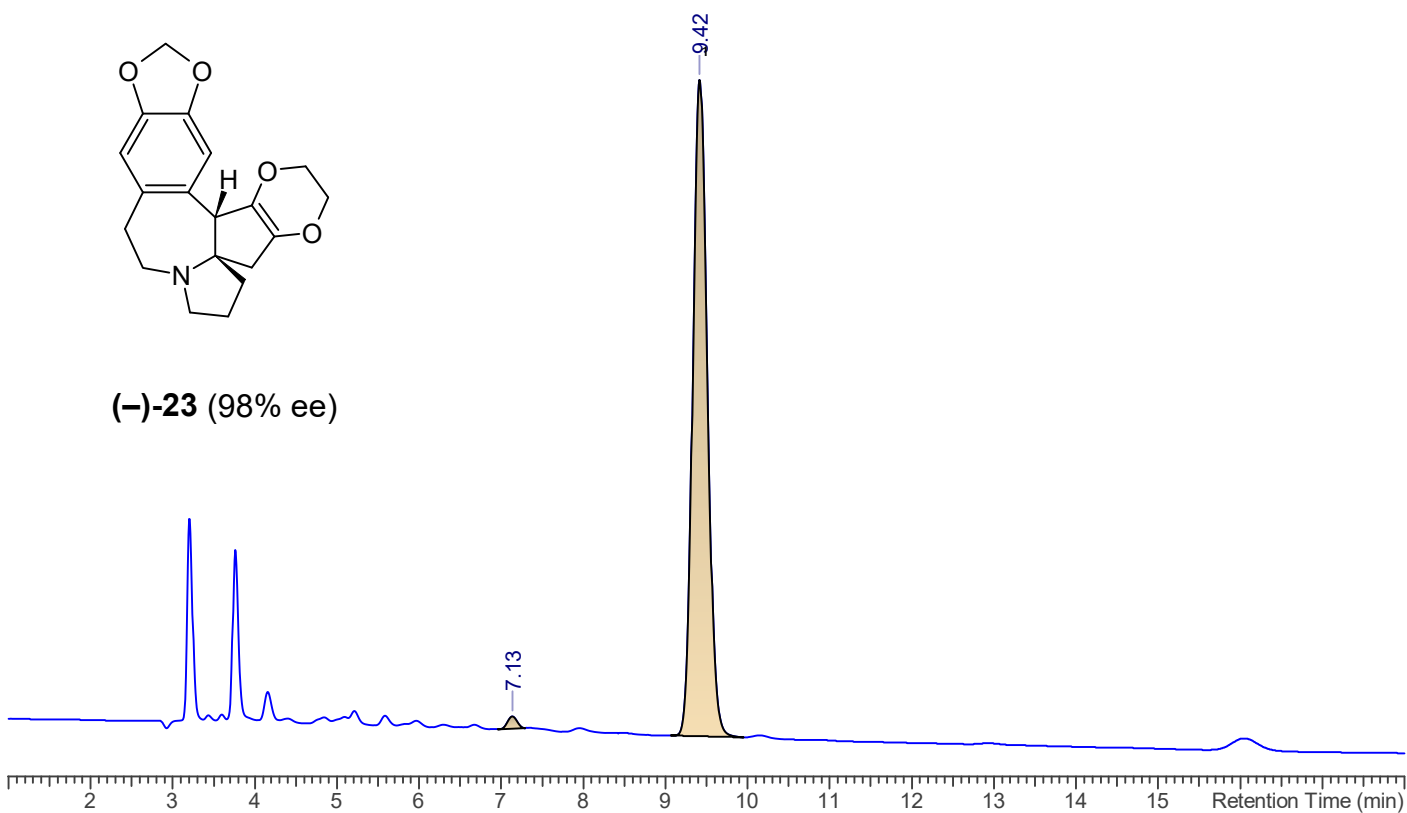
1.0 mL/min, 254 nm



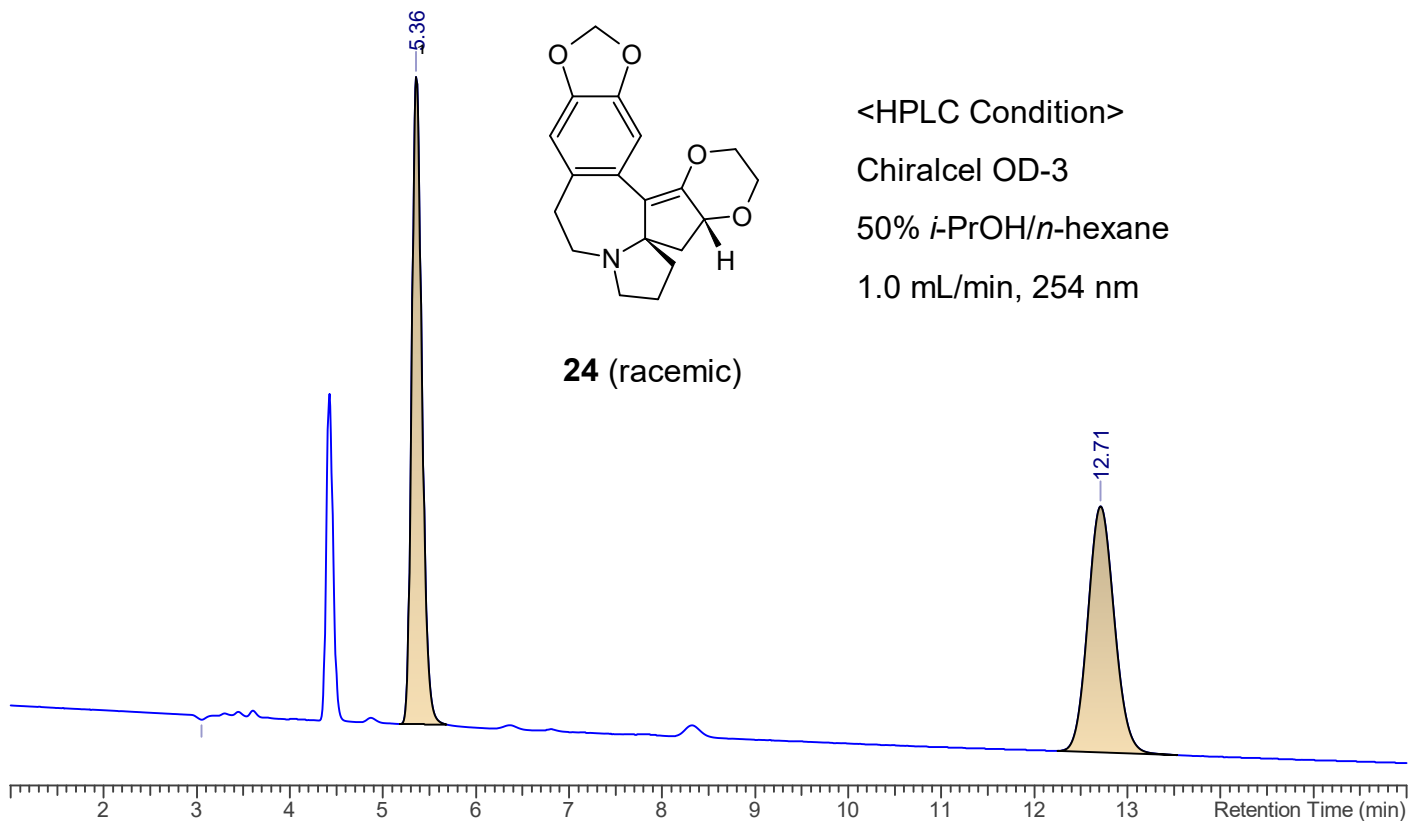
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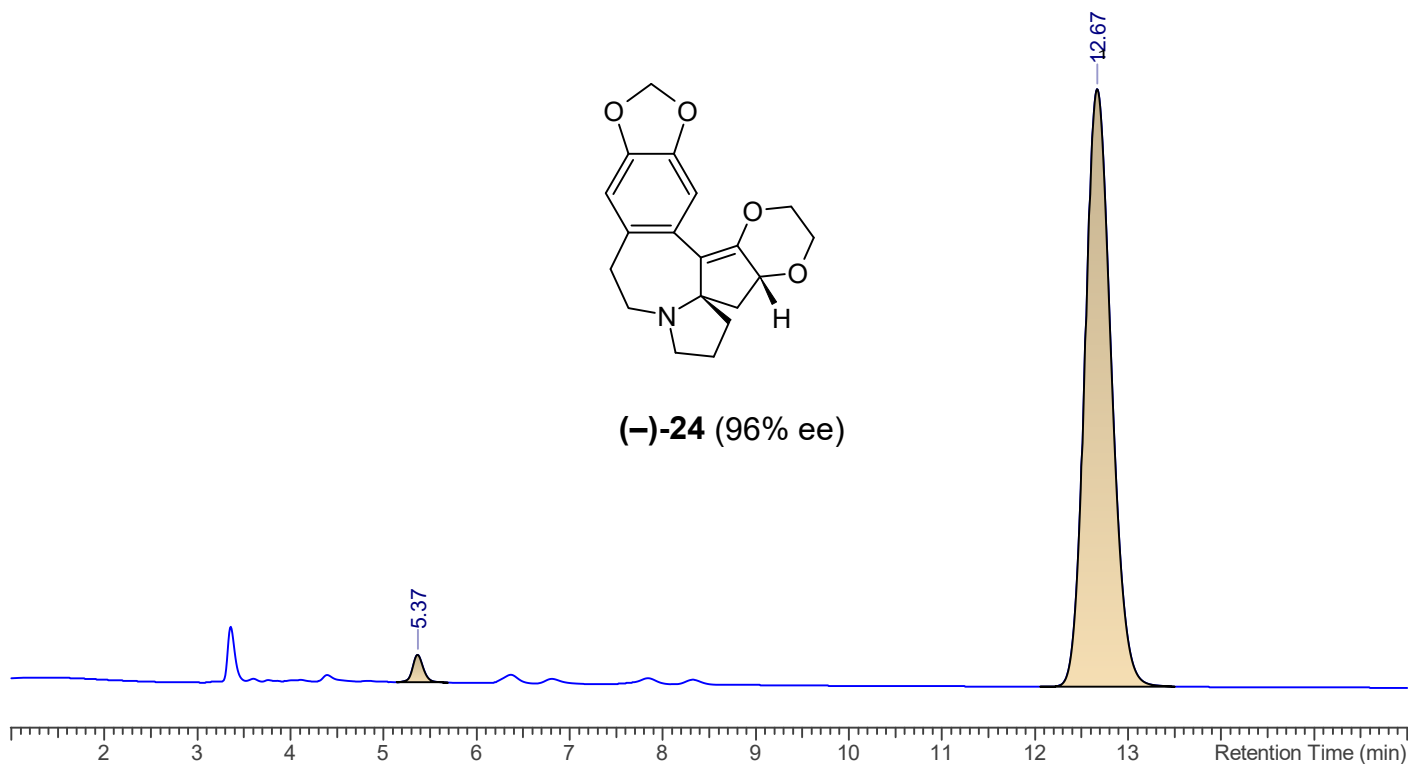
(-)-23 (98% ee)



No.	tR	Peak Area (Y units*ms)	Area (%)	Width
1	7.13	3347684.326	1.205	0.220
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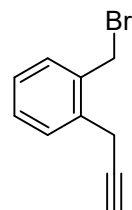


No.	tR	Peak Area (Y units*ms)	Area (%)	Width
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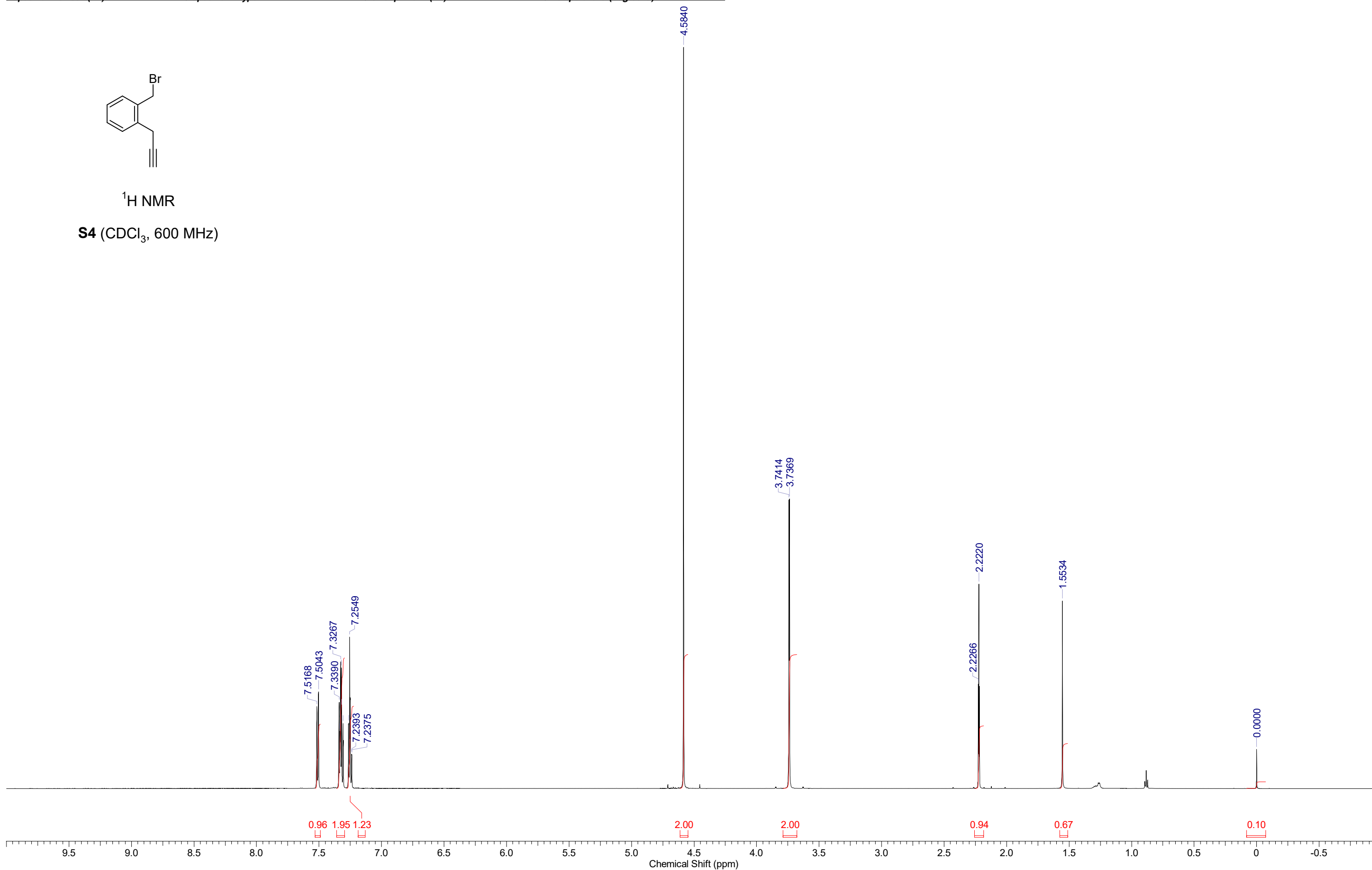
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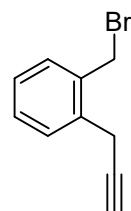


¹H NMR

S4 (CDCl₃, 600 MHz)

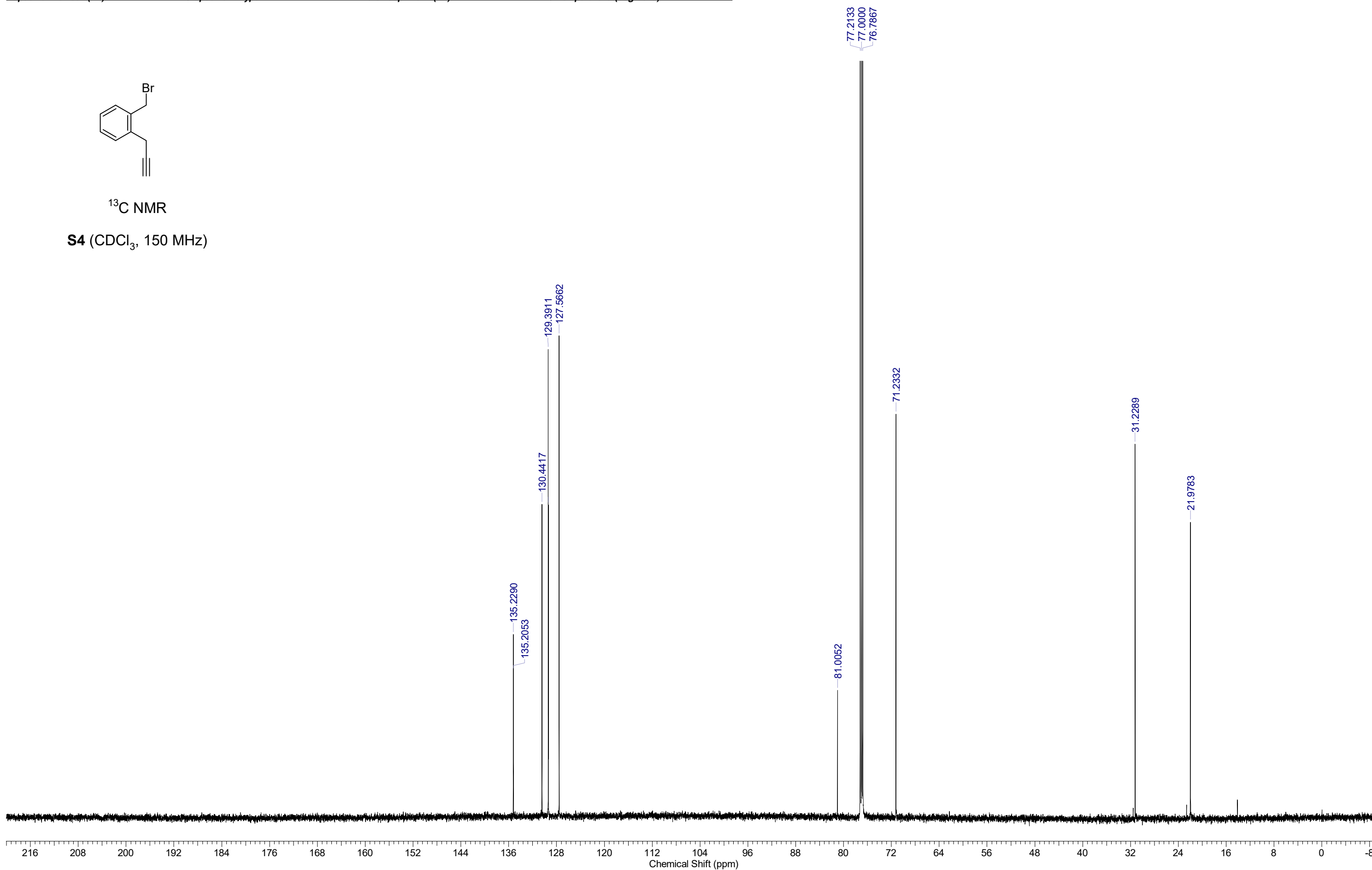


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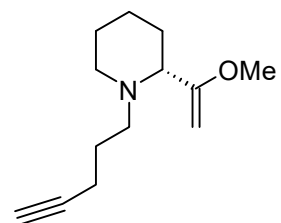


¹³C NMR

S4 (CDCl₃, 150 MHz)

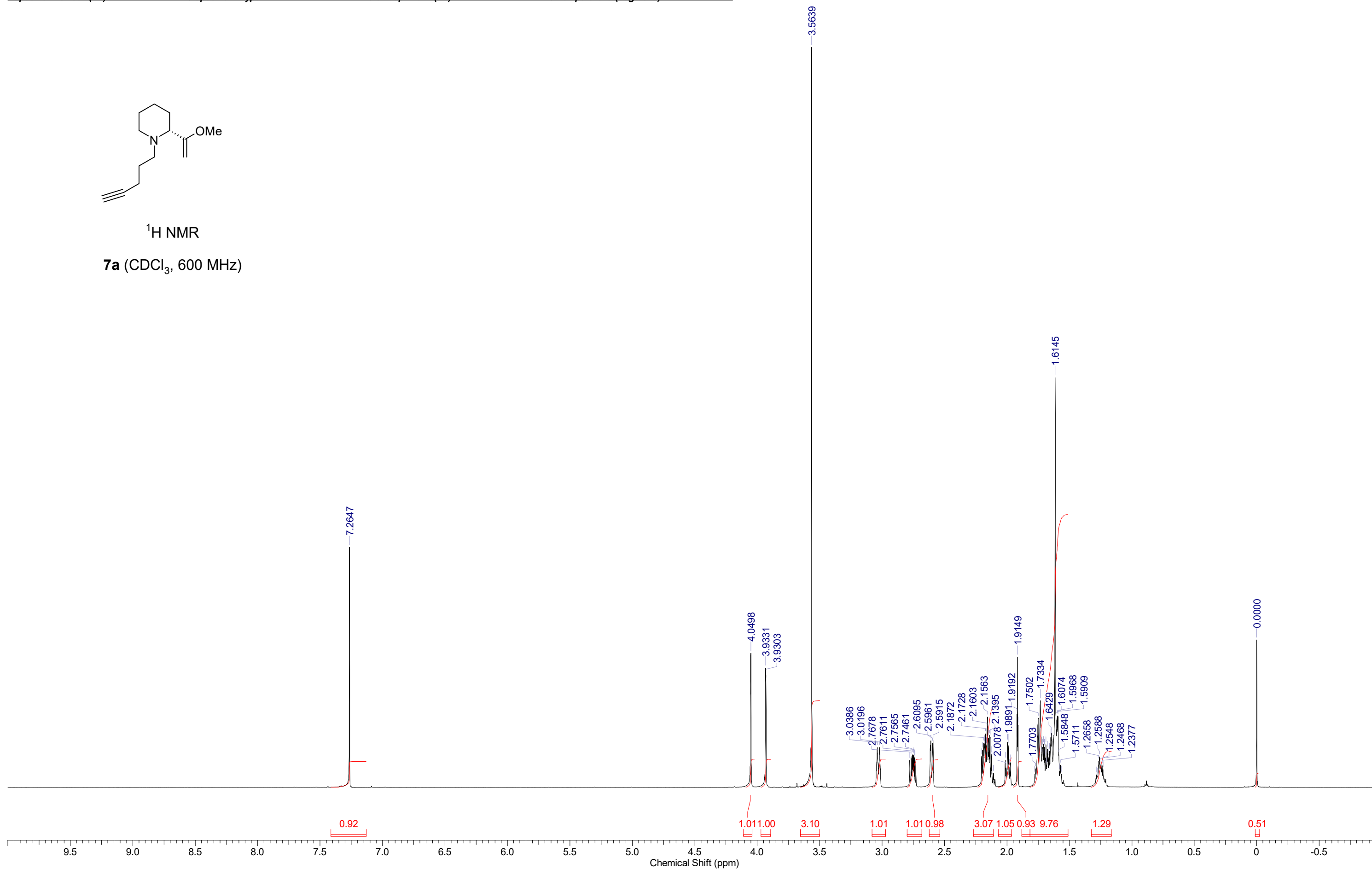


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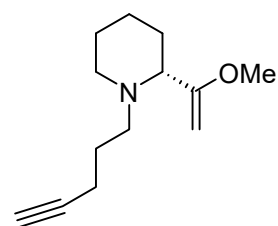


¹H NMR

7a (CDCl₃, 600 MHz)

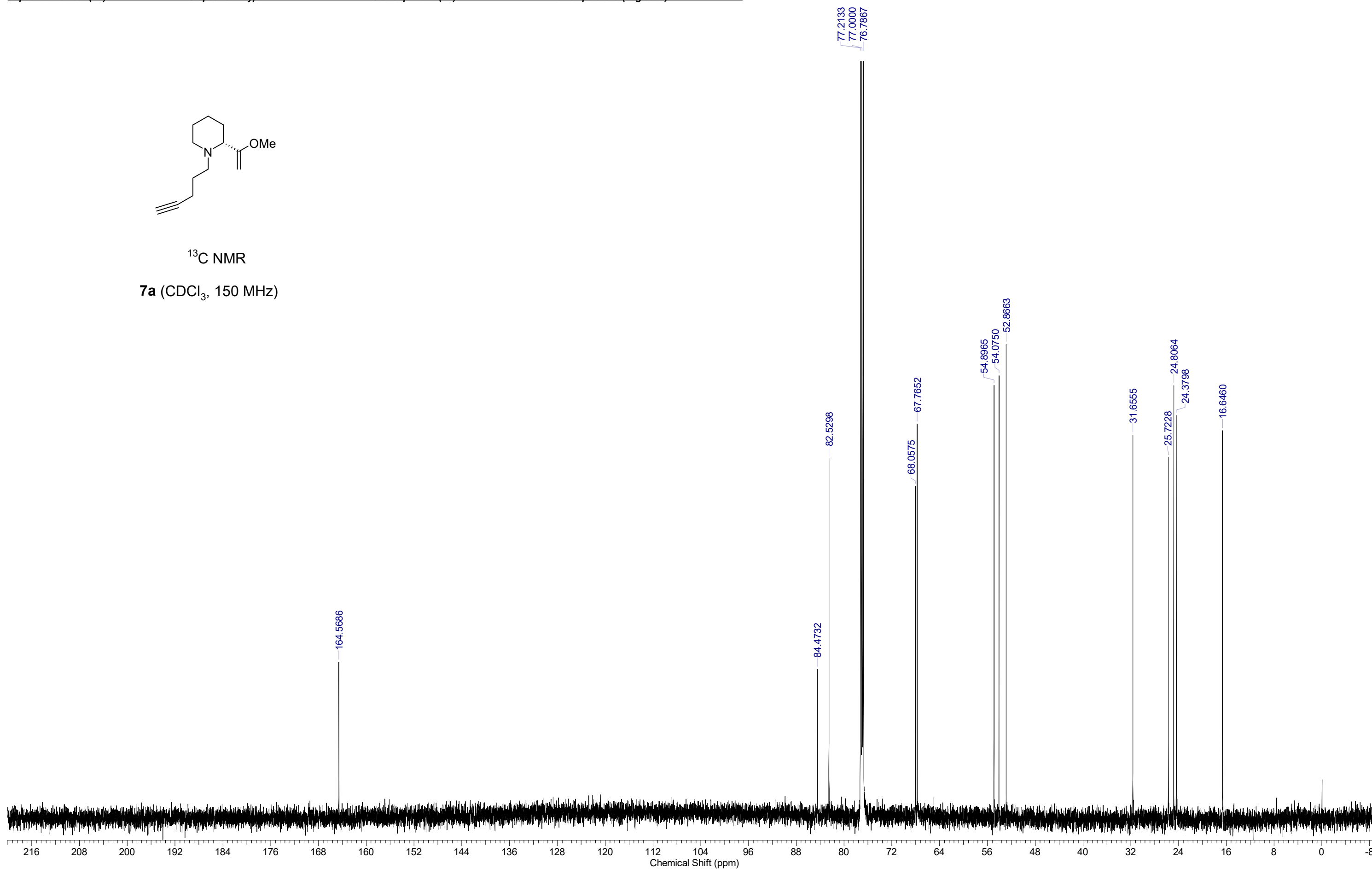


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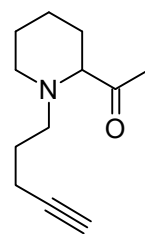


¹³C NMR

7a (CDCl₃, 150 MHz)

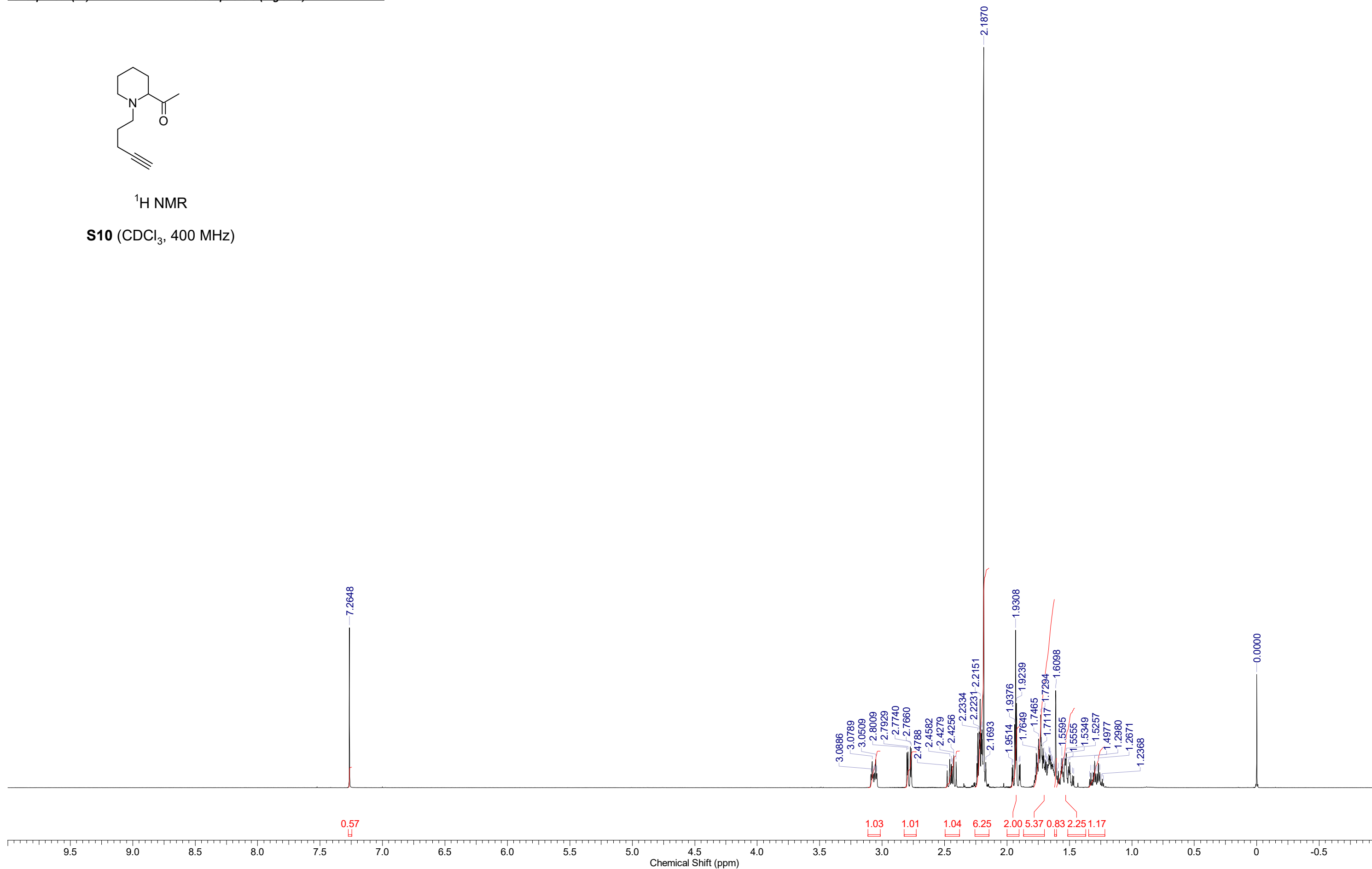


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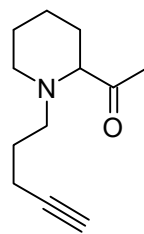


¹H NMR

S10 (CDCl₃, 400 MHz)

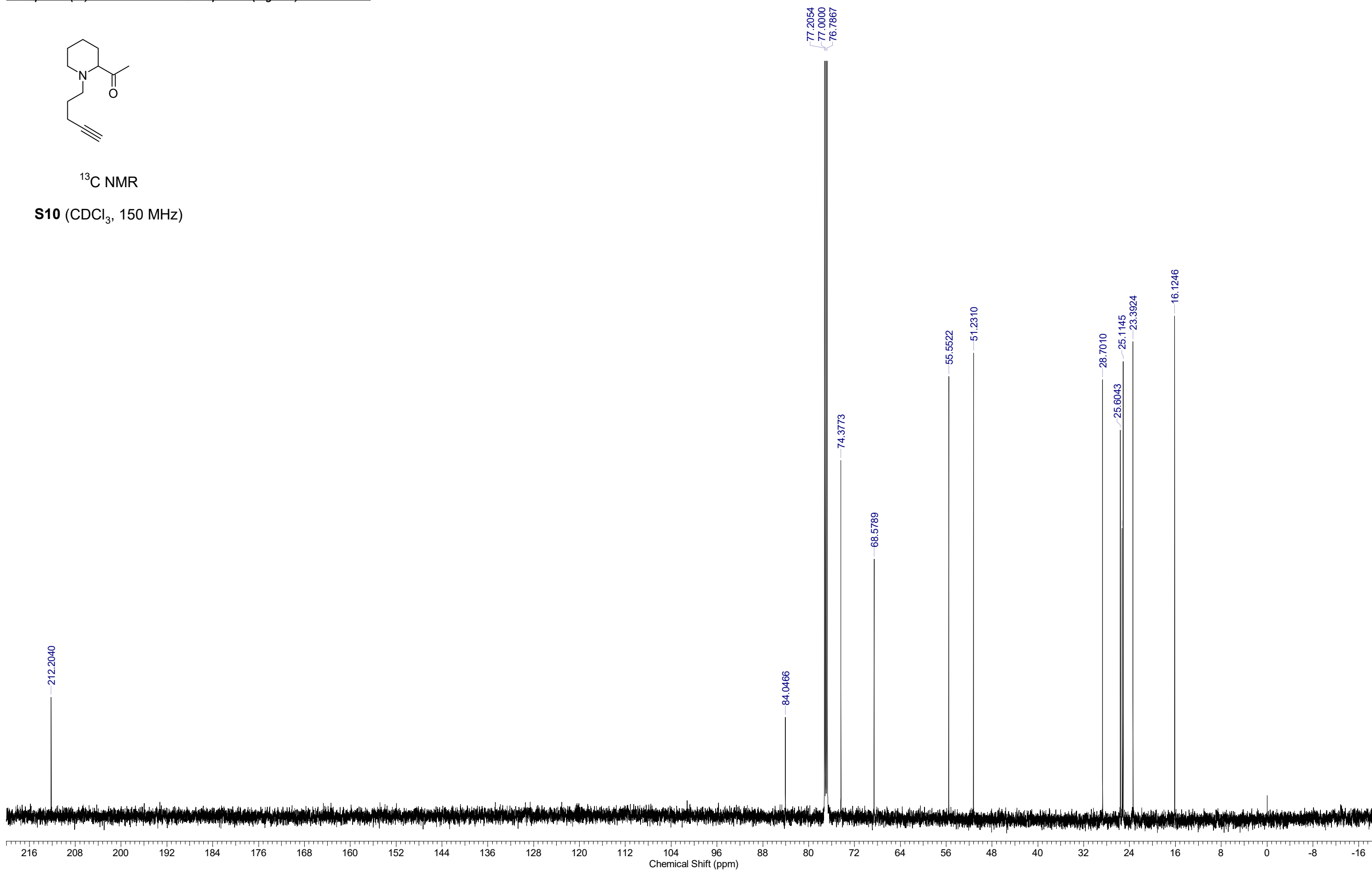


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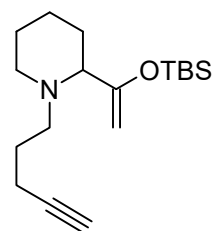


¹³C NMR

S10 (CDCl₃, 150 MHz)



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Sweep Width (Hz)	7494.00	Temperature (degree C)	25.000	Solvent	CHLOROFORM-d	Number of Transients	8
				Spectrum Offset (Hz)	2004.2638	Origin	ECA

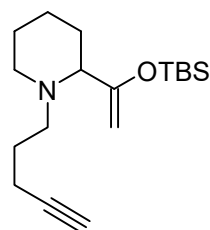


¹H NMR

7i (CDCl₃, 400 MHz)

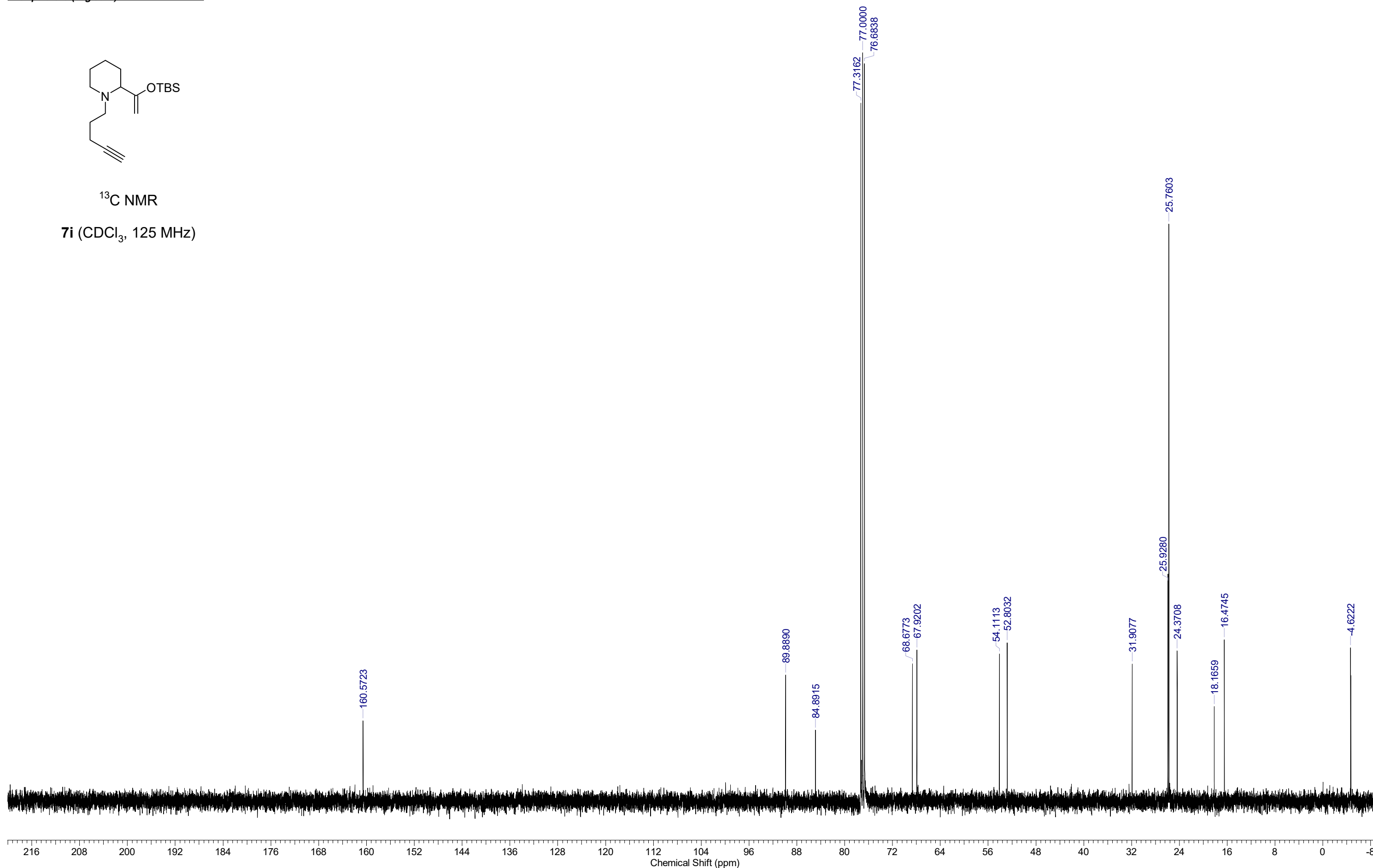


Acquisition Time (sec) 1.0381	Comment single_pulse_decoupled_gated_NOE	Date 17 Feb 2020 14:48:43	Date Stamp 17 Feb 2020 14:40:19
File Name ¥¥Mac¥Home¥Desktop¥mori_lab_NMR¥sakaiNMR¥sakai07032¥sakai07032ue_carbon-1-1.jdf		Frequency (MHz) 100.53	Nucleus 13C
Original Points Count 32768	Owner delta	Points Count 65536	Pulse Sequence carbon.jxp
Temperature (degree C) 24.700		Solvent CHLOROFORM-d	Number of Transients 256
			Spectrum Offset (Hz) 10043.9922
			Origin ECA
			Sweep Width (Hz) 31565.66

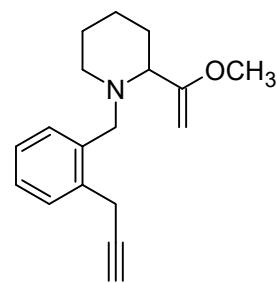


¹³C NMR

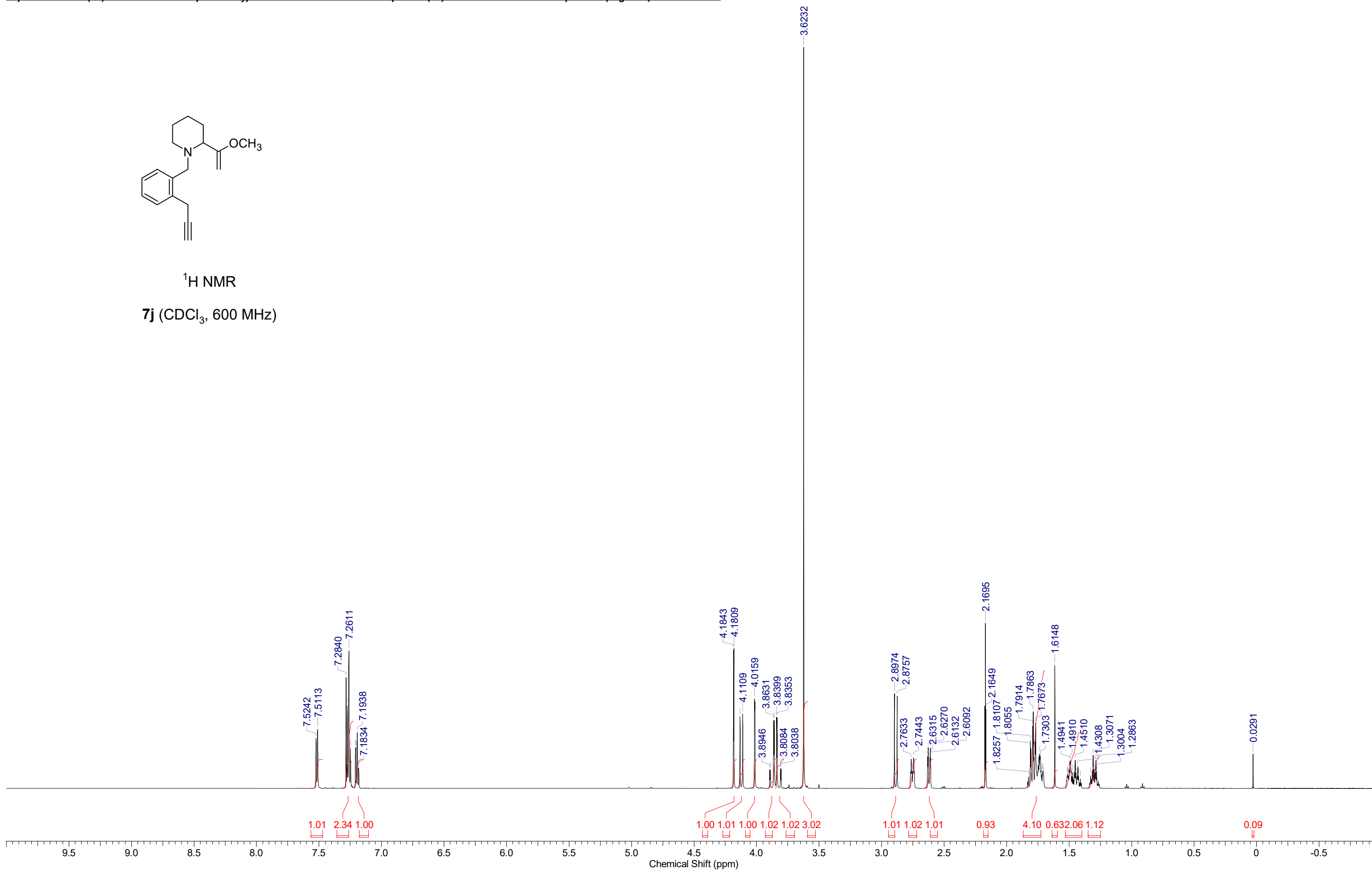
7i (CDCl₃, 125 MHz)



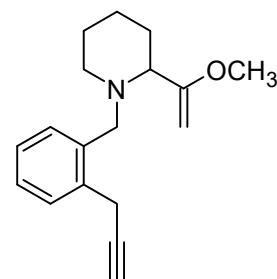
Acquisition Time (sec)	2.7263	Comment	proton	Date	05 Nov 2019 15:00:32	Date Stamp	05 Nov 2019 15:00:32		
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06104¥1¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8	
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	30.91
Spectrum Offset (Hz)	3706.0515	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.159	SW(cyclical) (Hz)	12019.23
								Origin	spect
								Solvent	CHLOROFORM-d



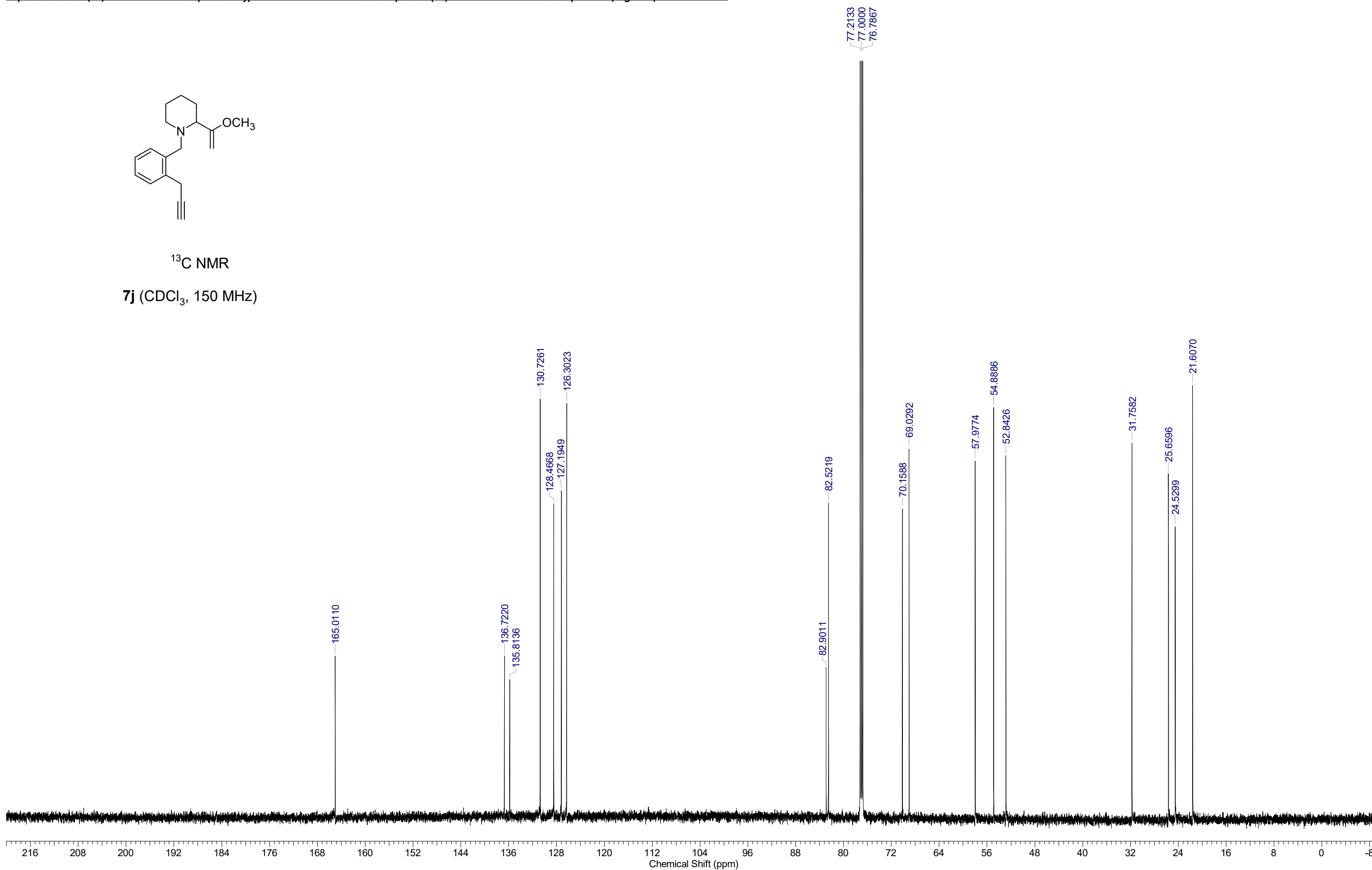
¹H NMR
7j (CDCl₃, 600 MHz)



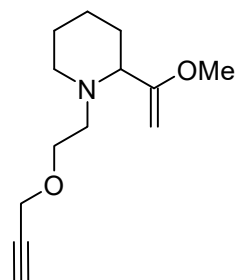
Acquisition Time (sec)	0.8389	Comment	carbon	Date	05 Nov 2019 15:06:56	Date Stamp	05 Nov 2019 15:06:56
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06104¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zpg30
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.152	Number of Transients	53
						SM(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d



¹³C NMR
7j (CDCl₃, 150 MHz)

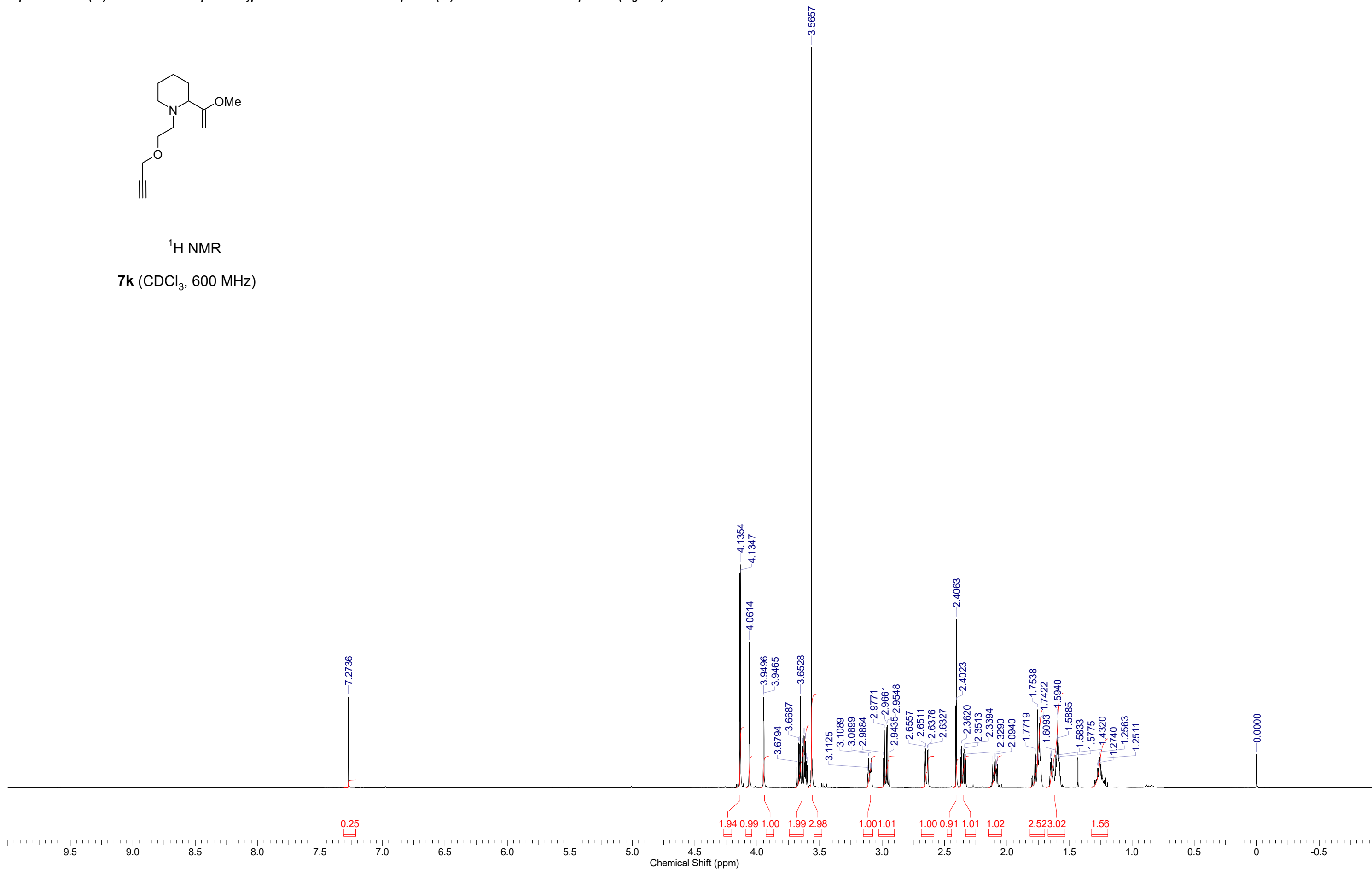


Acquisition Time (sec)	2.7263	Comment	proton	Date	04 Jun 2018 15:49:36	Date Stamp	04 Jun 2018 15:49:36
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamiva01102 fr21-¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3699.4309	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
				Temperature (degree C)	25.138	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

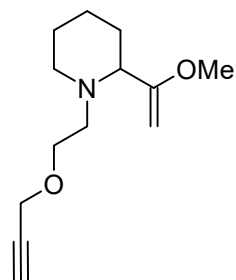


¹H NMR

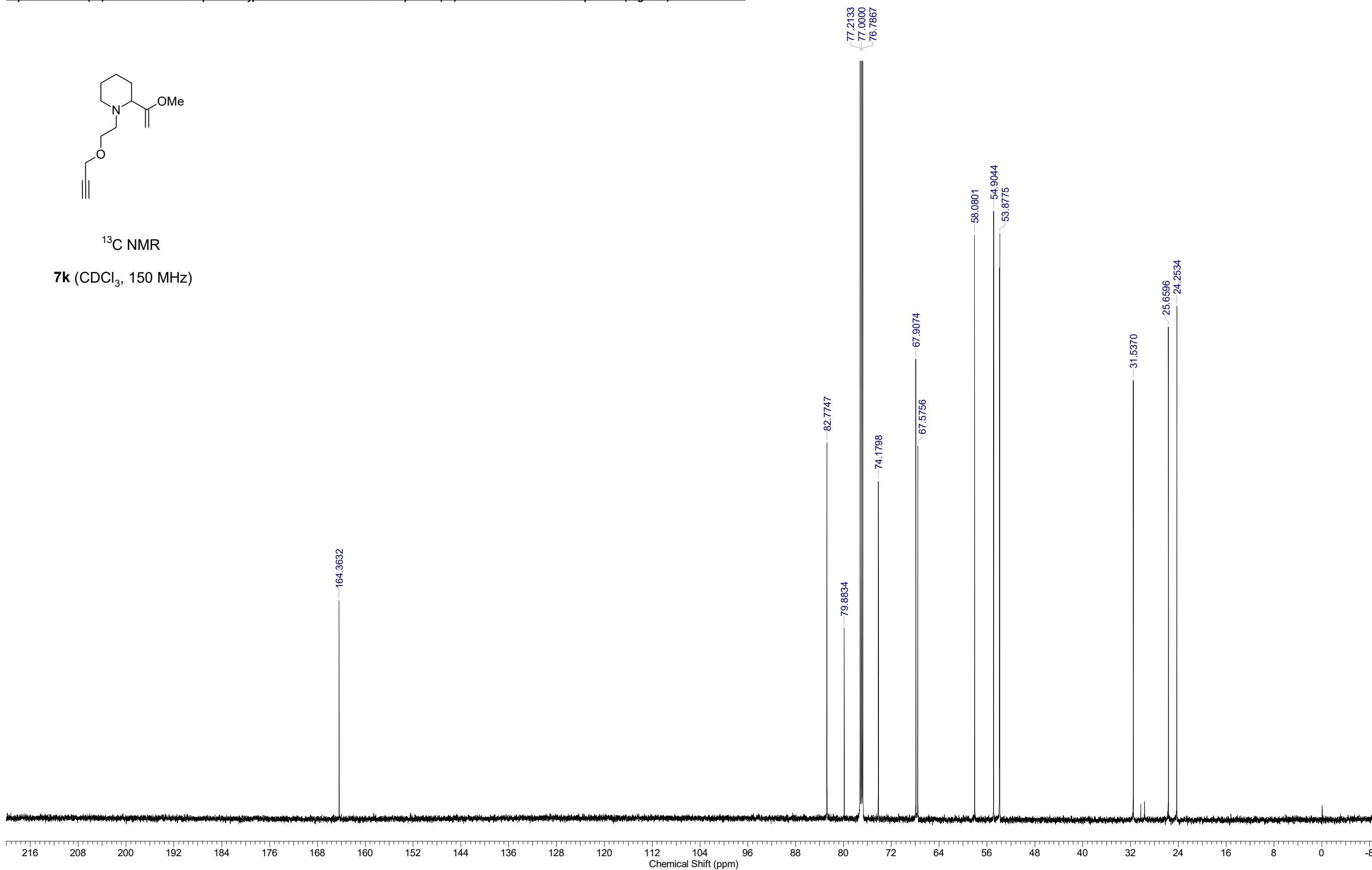
7k (CDCl₃, 600 MHz)



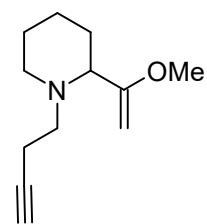
Acquisition Time (sec)	0.8389	Comment	carbon	Date	04 Jun 2018 16:34:24	Date Stamp	04 Jun 2018 16:34:24
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamiva01102 fr21-¥20¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	¹³ C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15083.1416	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.163	Number of Transients	128
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d



¹³C NMR
7k (CDCl₃, 150 MHz)

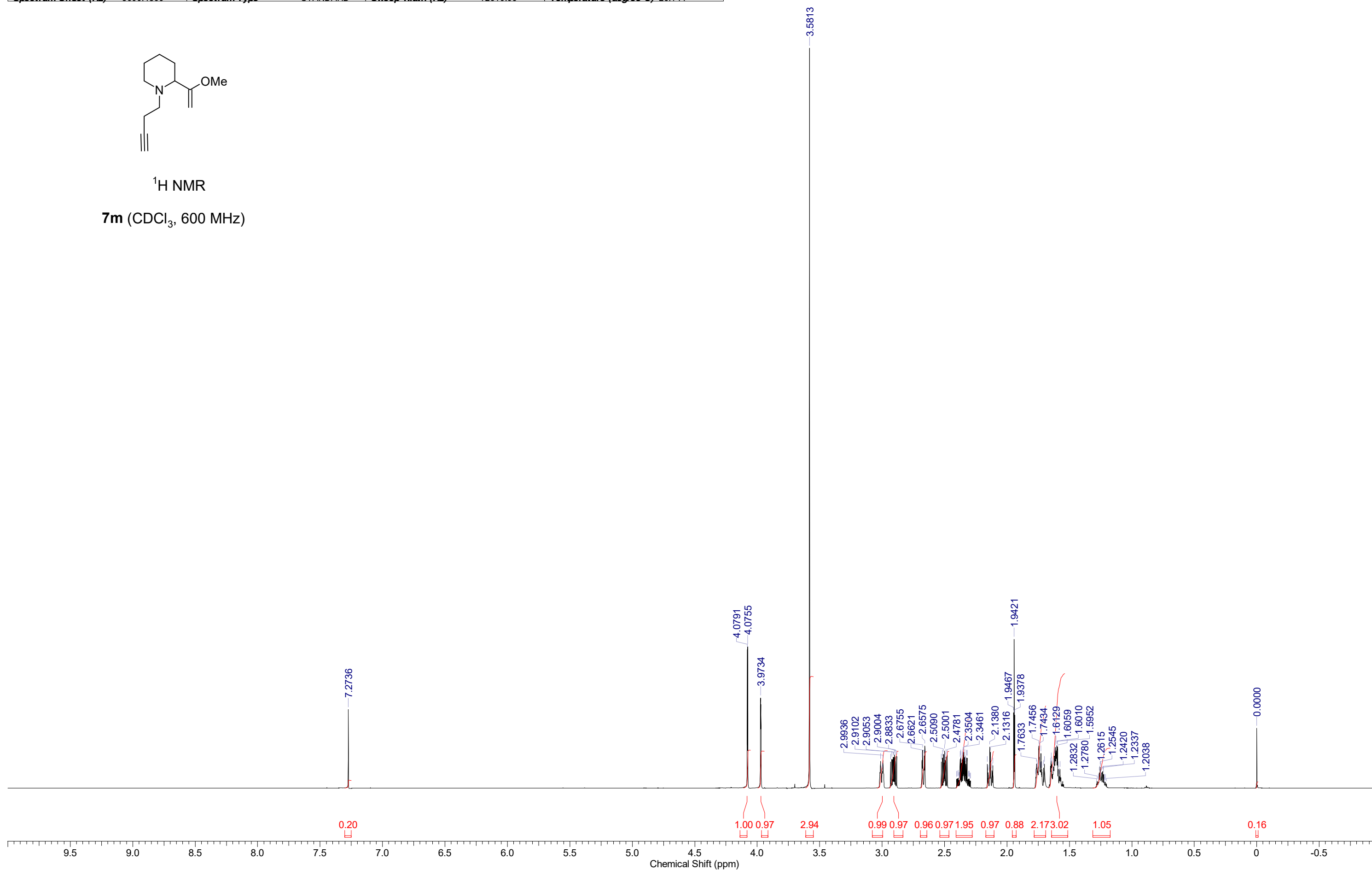


Acquisition Time (sec)	2.7263	Comment	proton	Date	24 Dec 2019 19:57:04	Date Stamp	24 Dec 2019 19:57:04
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07018ue¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3699.4309	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	22.72
				Temperature (degree C)	25.144	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

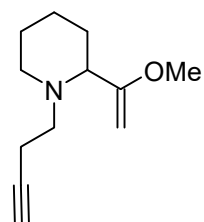


¹H NMR

7m (CDCl₃, 600 MHz)

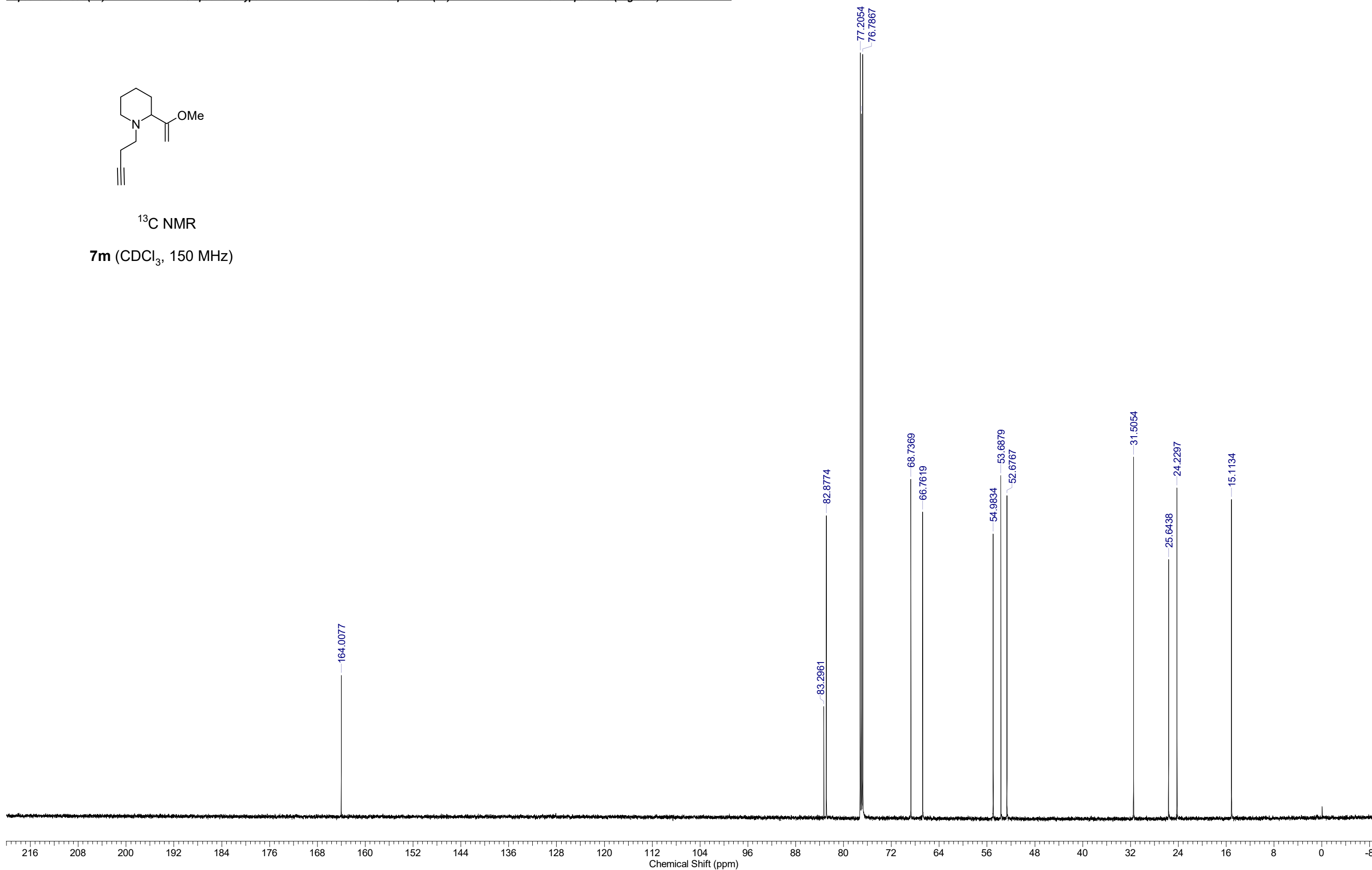


Acquisition Time (sec)	0.8389	Comment	carbon	Date	24 Dec 2019 20:05:36	Date Stamp	24 Dec 2019 20:05:36
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07018ue¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15083.1416	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.153	Number of Transients	128
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

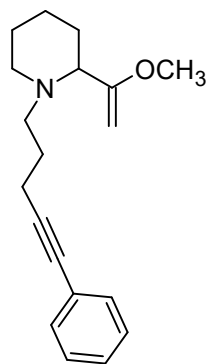


¹³C NMR

7m (CDCl₃, 150 MHz)

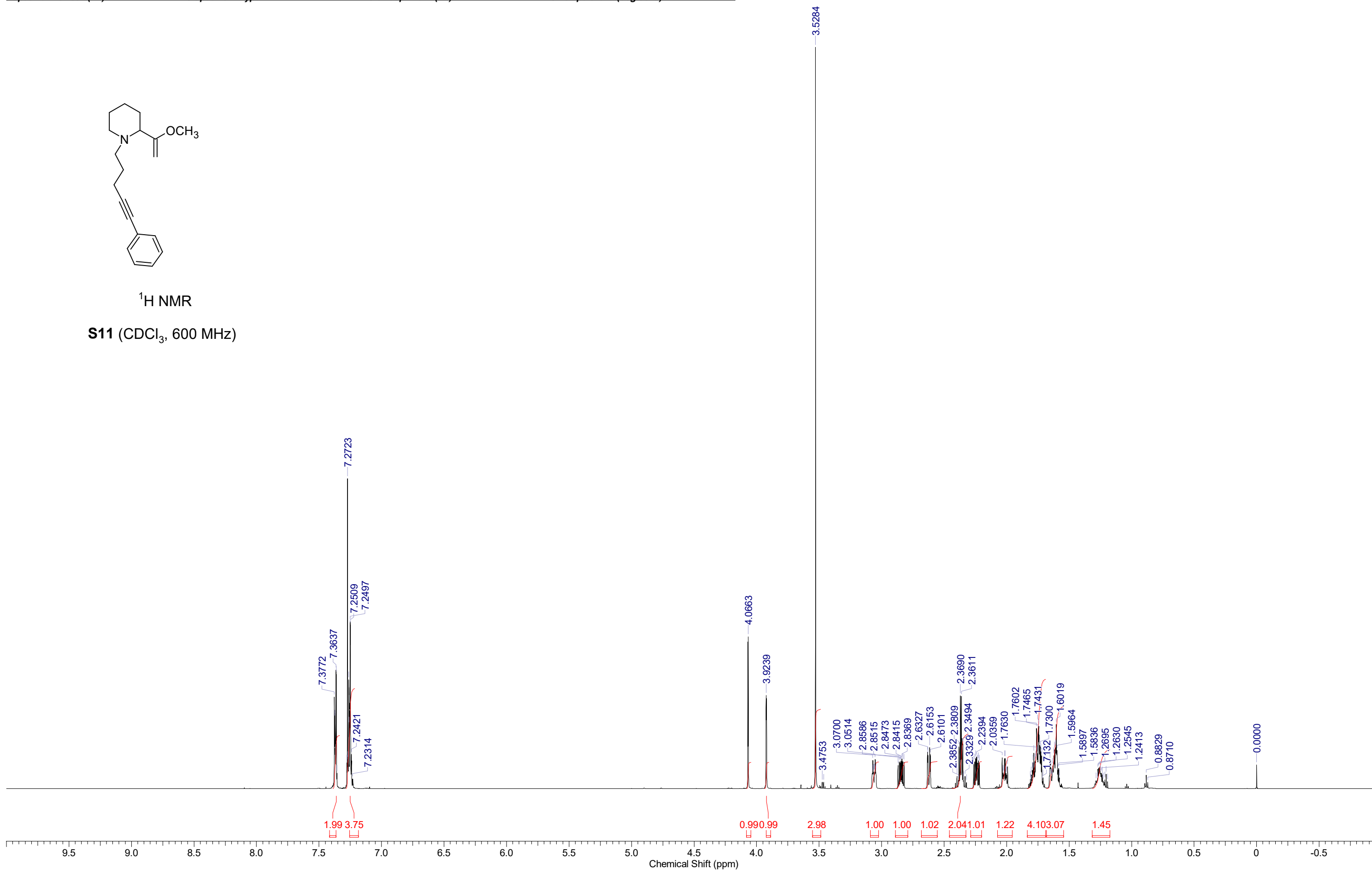


Acquisition Time (sec)	2.7263	Comment	proton	Date	06 Jul 2018 15:34:40	Date Stamp	06 Jul 2018 15:34:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamiva01106 fr19-¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3698.5139	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	14.11
				Temperature (degree C)	25.142	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Solvent	CHLOROFORM-d

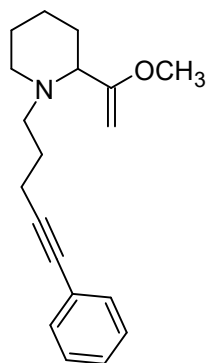


¹H NMR

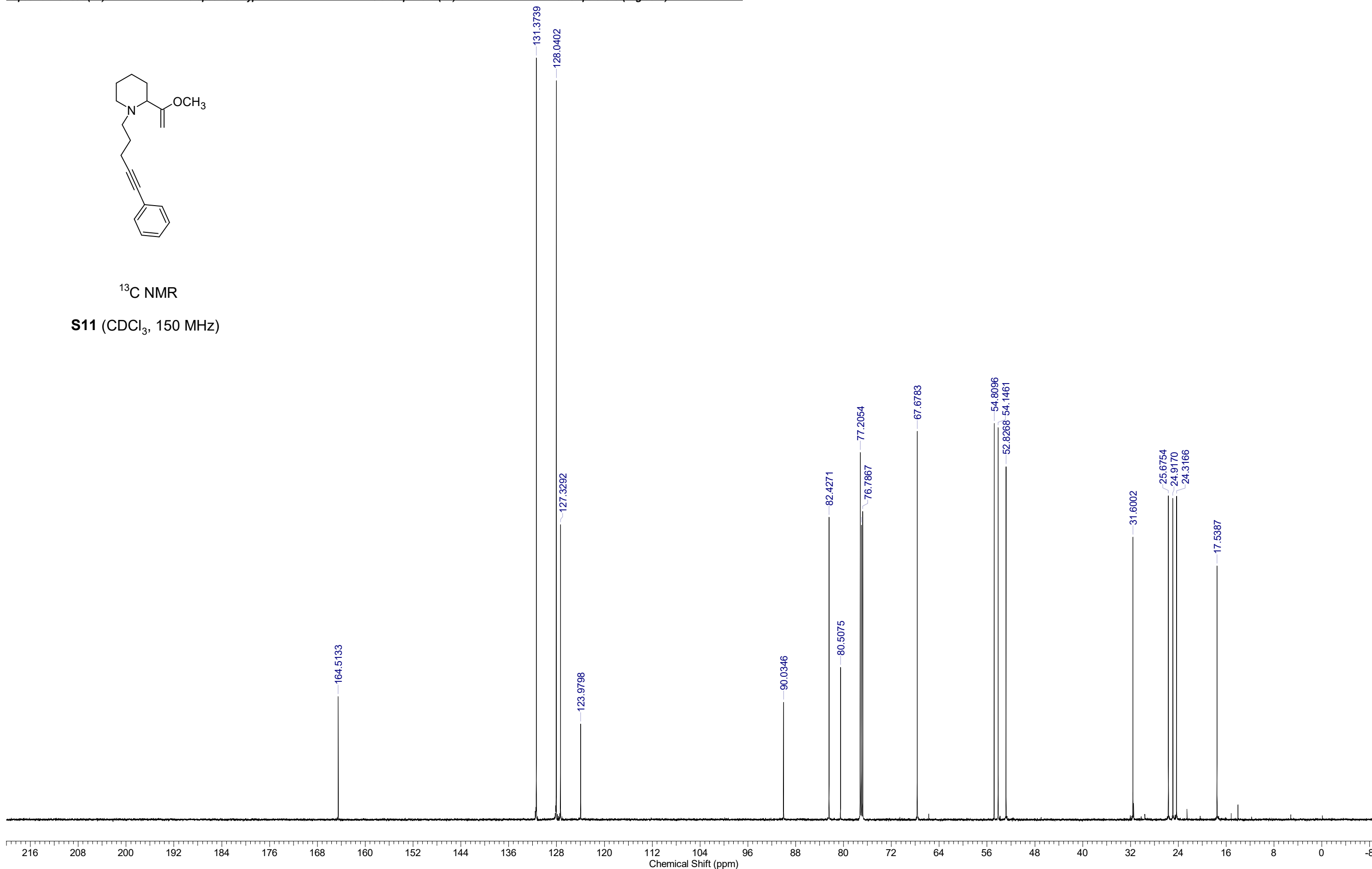
S11 (CDCl₃, 600 MHz)



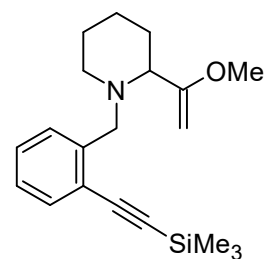
Acquisition Time (sec)	0.8389	Comment	carbon	Date	06 Jul 2018 15:38:56	Date Stamp	06 Jul 2018 15:38:56
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamija01106 fr19-¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15072.4131	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
						SW(cyclical) (Hz)	39062.50
						Temperature (degree C)	25.136
						Origin	spect
						Solvent	CHLOROFORM-d



¹³C NMR
S11 (CDCl₃, 150 MHz)

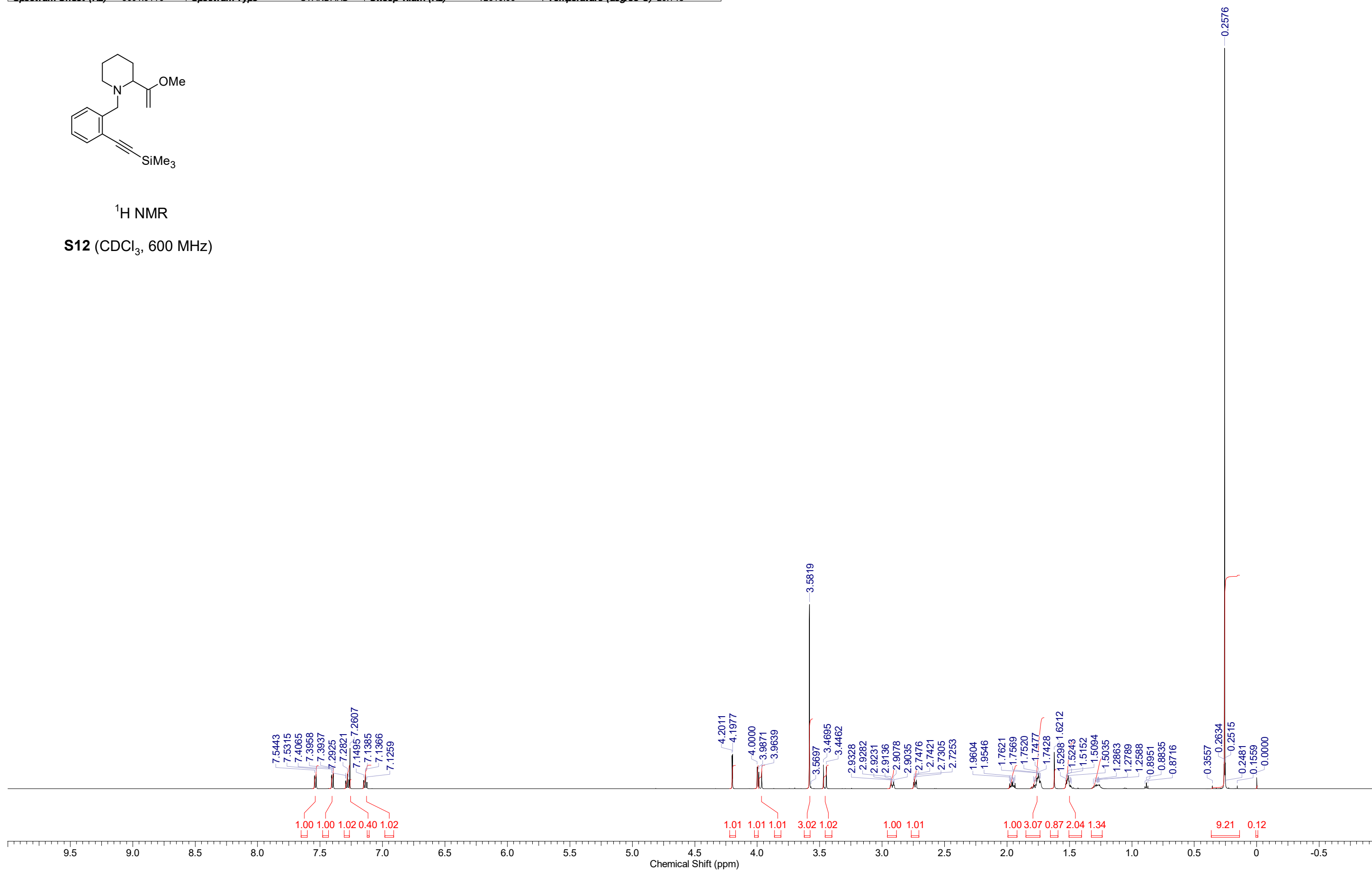


Acquisition Time (sec)	2.7263	Comment	proton	Date	05 Nov 2019 14:52:00	Date Stamp	05 Nov 2019 14:52:00
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06103¥1¥pdata¥1¥1r	Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3691.9116	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
				Temperature (degree C)	25.148	SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

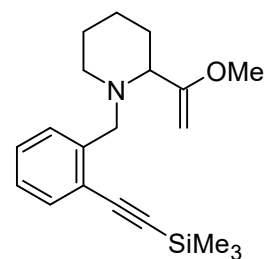


¹H NMR

S12 (CDCl₃, 600 MHz)

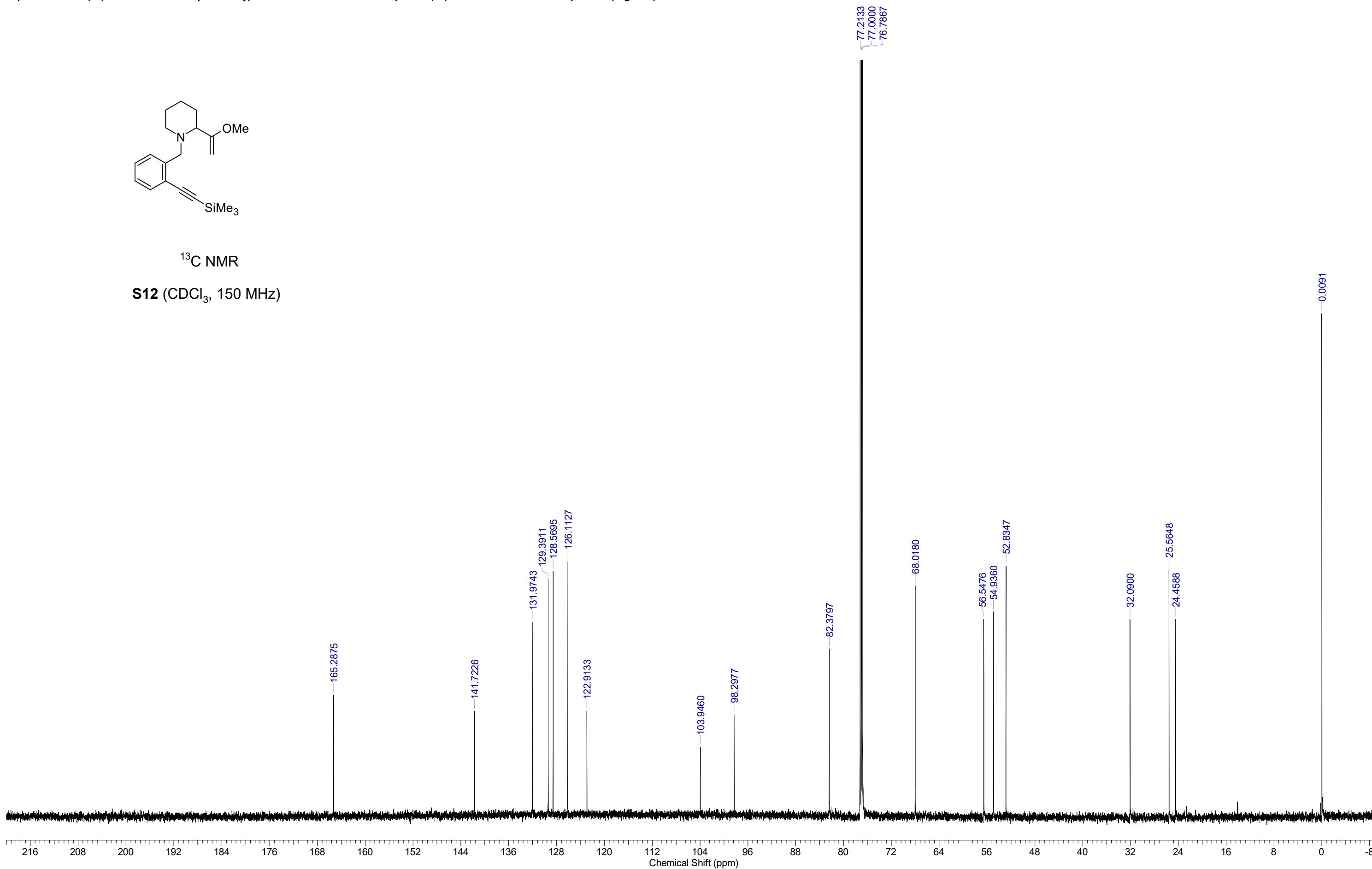


Acquisition Time (sec)	0.8389	Comment	carbon	Date	05 Nov 2019 14:56:16	Date Stamp	05 Nov 2019 14:56:16
File Name	¥¥Mac¥Home¥Desktop¥mori.lab.NMR¥sakaiNMR¥sakai06103¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.141	Number of Transients	48
						SM(cyclical) (Hz)	39062.50
						Solvent	CHLOROFORM-d

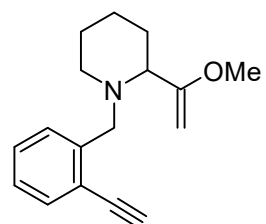


¹³C NMR

S12 (CDCl₃, 150 MHz)

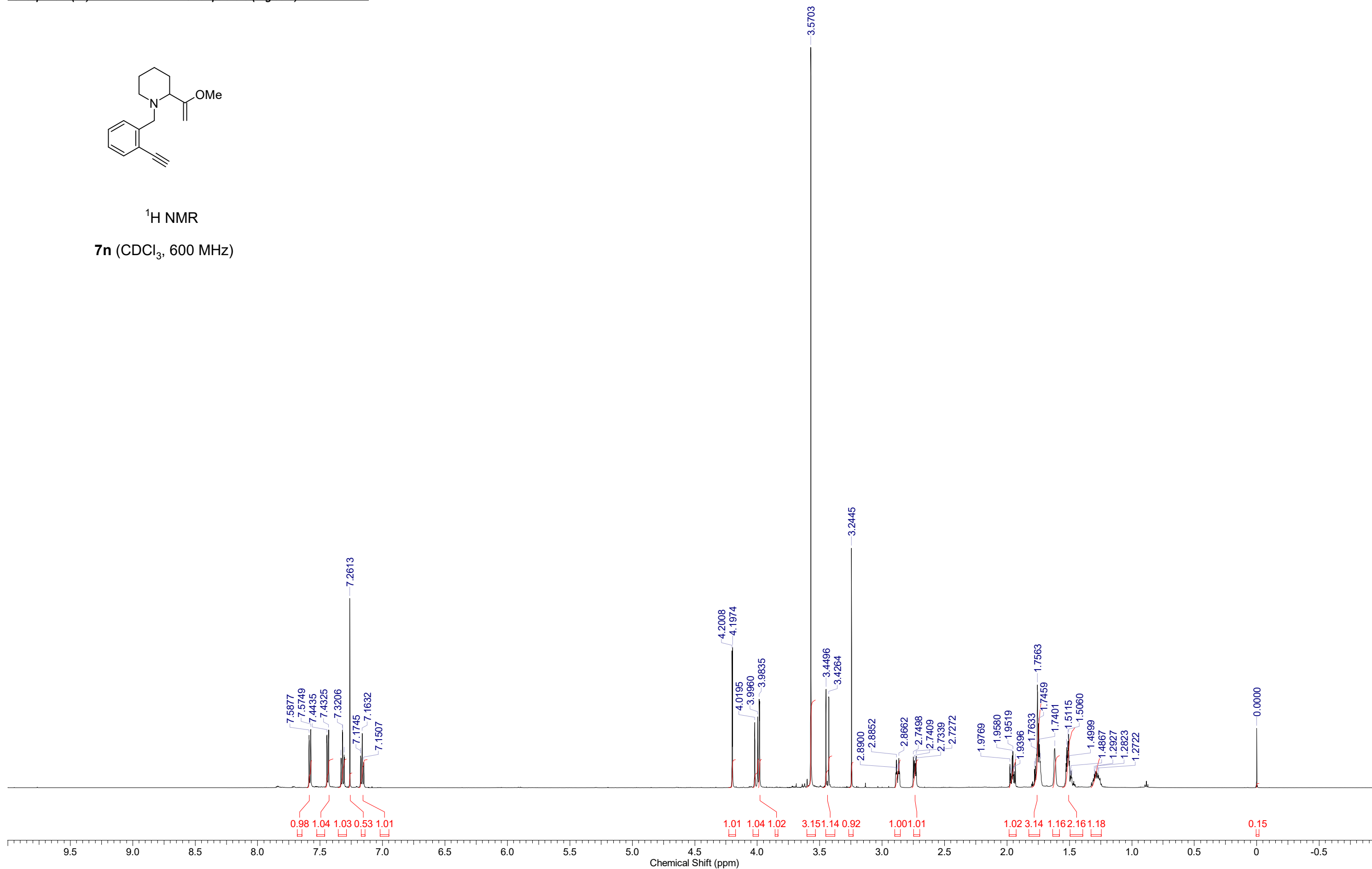


Acquisition Time (sec)	2.7263	Date	07 Nov 2019 15:49:36	Date Stamp	07 Nov 2019 15:49:36	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06105ue¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	30.91	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.153			Spectrum Offset (Hz)	3692.2783
						Points Count	65536
						Spectrum Type	STANDARD

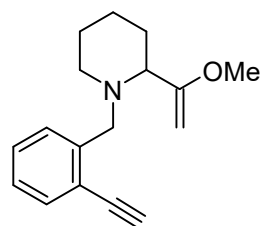


¹H NMR

7n (CDCl₃, 600 MHz)

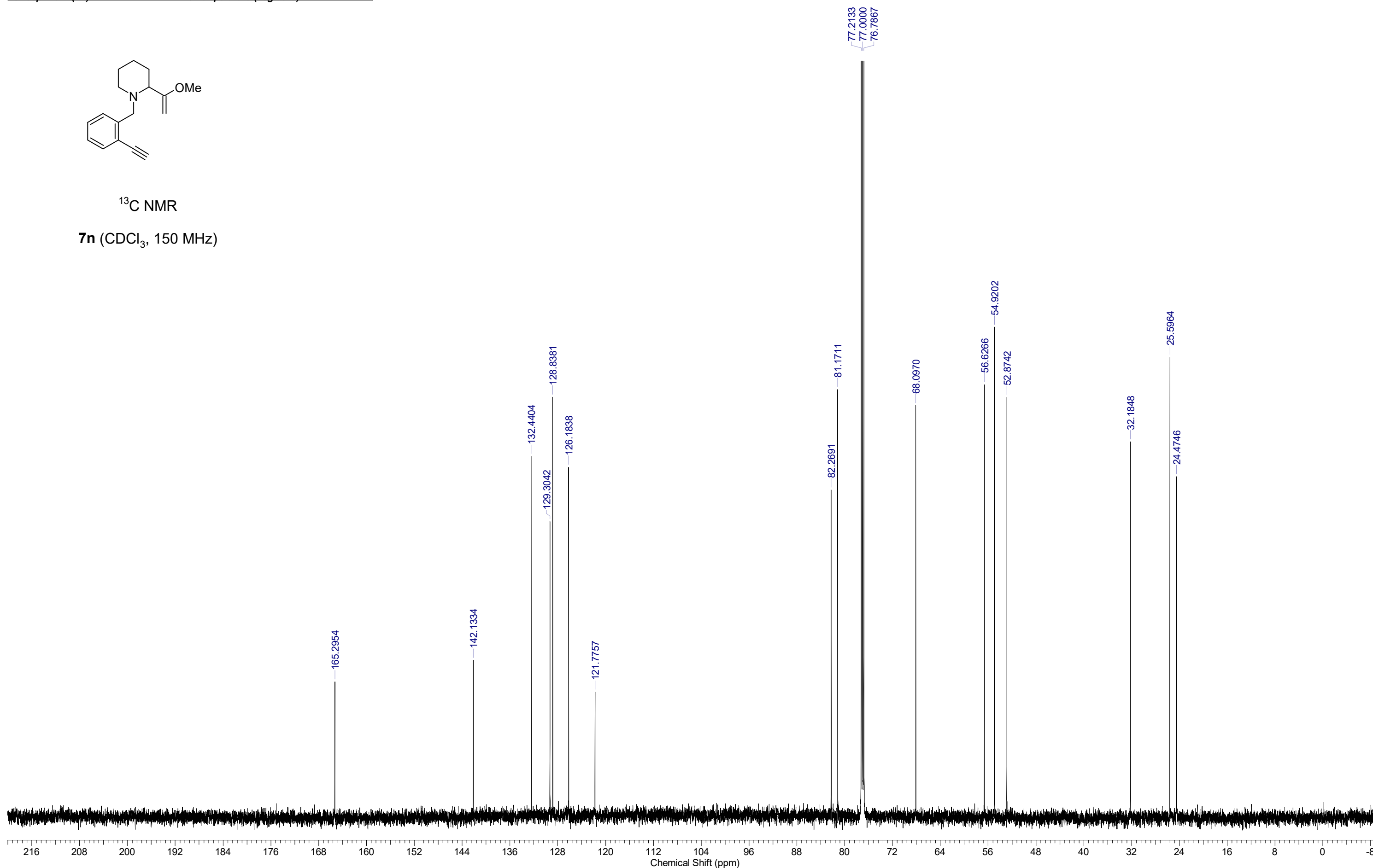


Acquisition Time (sec)	0.8389	Date	07 Nov 2019 15:53:52	Date Stamp	07 Nov 2019 15:53:52	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06105ue¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	13C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.148			Spectrum Offset (Hz)	15085.5254
						Points Count	32768
						Spectrum Type	STANDARD

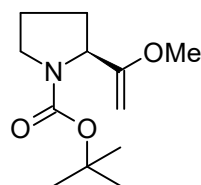


¹³C NMR

7n (CDCl₃, 150 MHz)

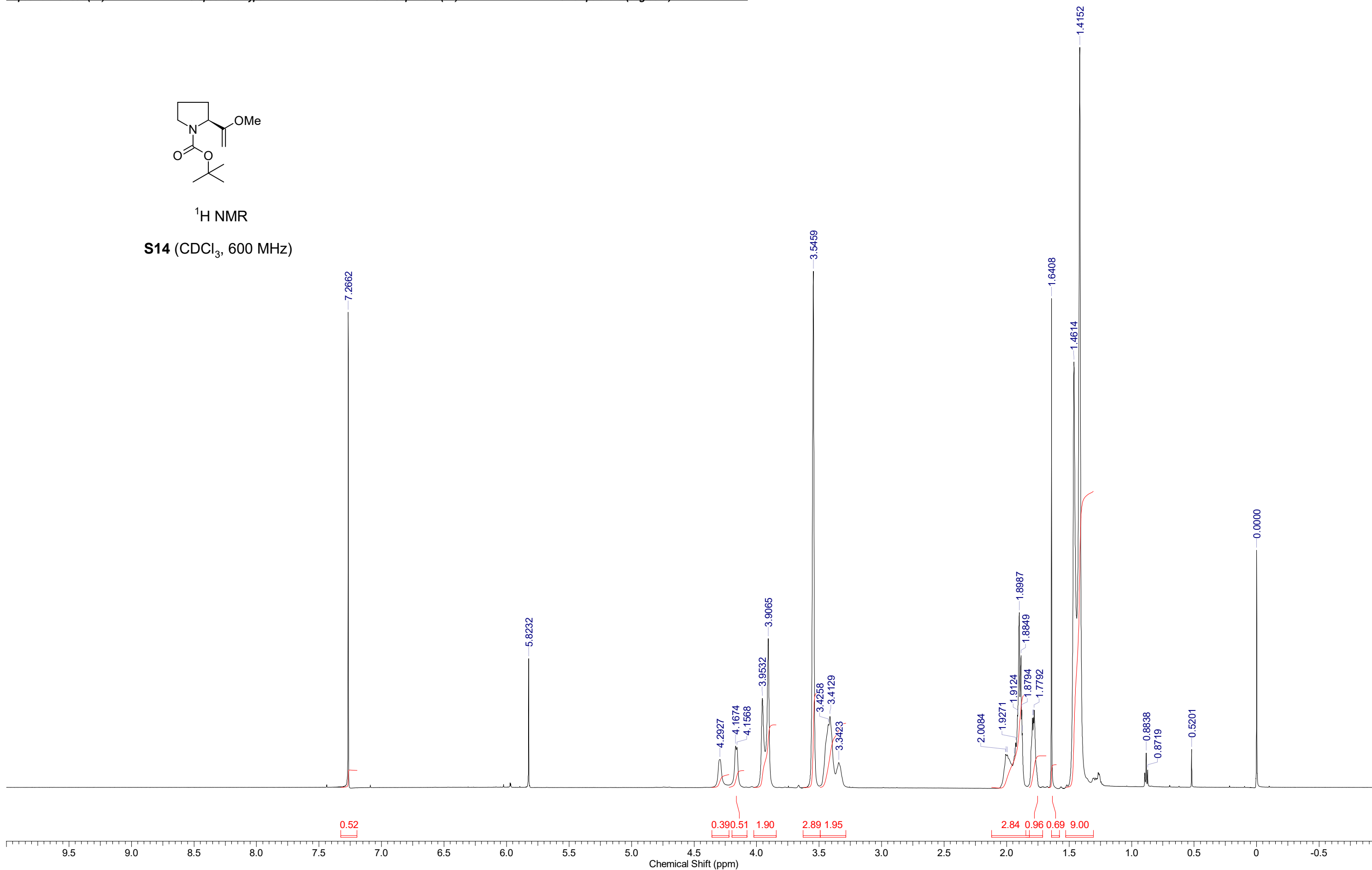


Acquisition Time (sec)	2.7263	Date	22 May 2017 12:24:48	Date Stamp	22 May 2017 12:24:48						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥竹内NMR¥takeuchi01087_1¥20¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H					
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30	Receiver Gain	56.41	Number of Transients	16	Origin	spect
Spectrum Offset (Hz)	3694.8459	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.141	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d

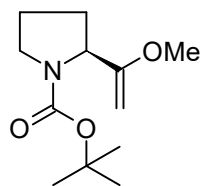


¹H NMR

S14 (CDCl₃, 600 MHz)

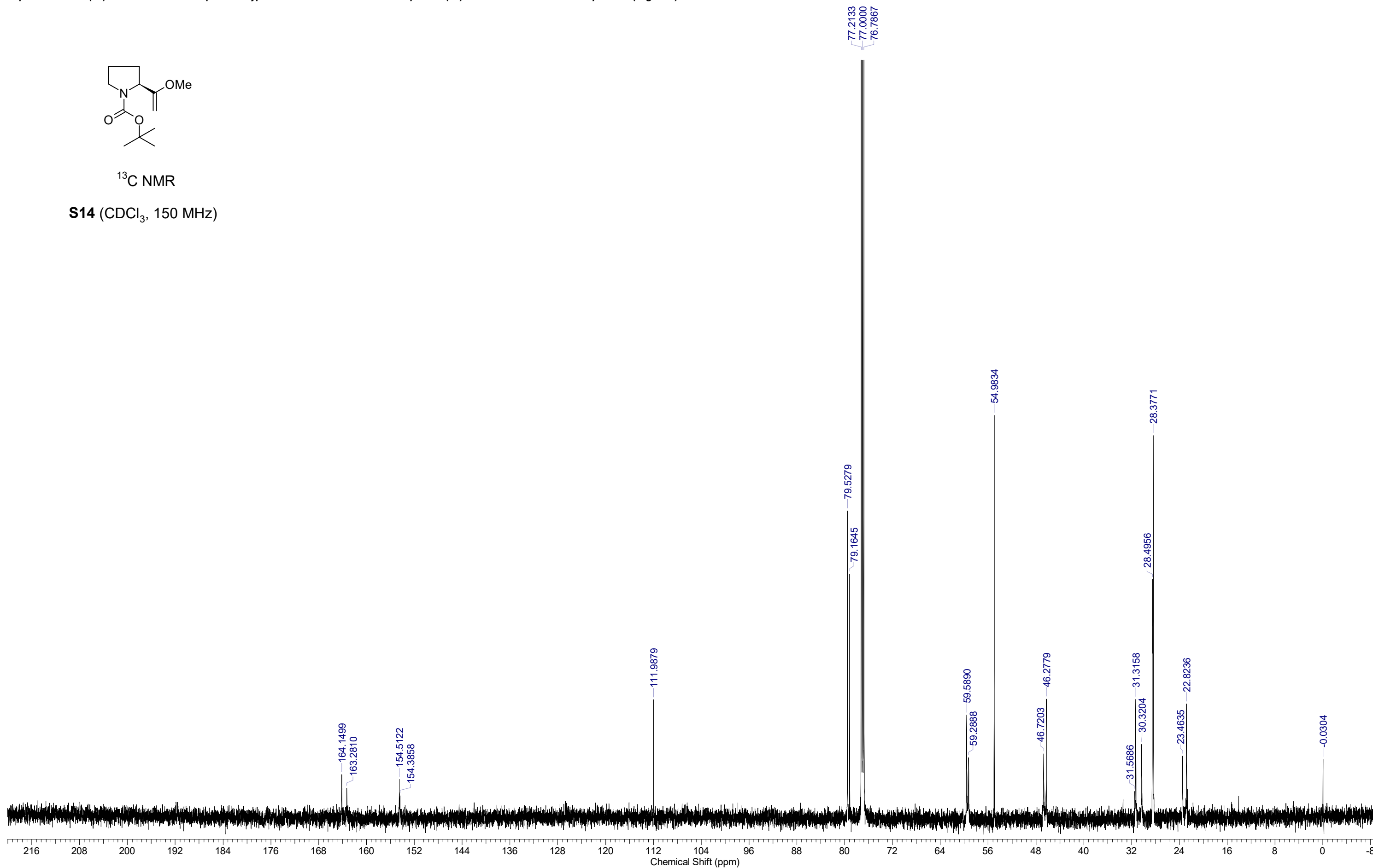


Acquisition Time (sec)	0.8389	Date	22 May 2017 12:39:44	Date Stamp	22 May 2017 12:39:44
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥竹内NMR¥takeuchi01087_1¥22¥pdata¥1¥1r			Frequency (MHz)	150.90
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Receiver Gain	193.26
		Points Count	32768	SW(cyclical) (Hz)	39062.50
		Sweep Width (Hz)	39061.31	Origin	spect
		Temperature (degree C)	25.132	Solvent	CHLOROFORM-d

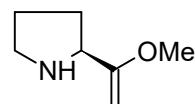


¹³C NMR

S14 (CDCl₃, 150 MHz)

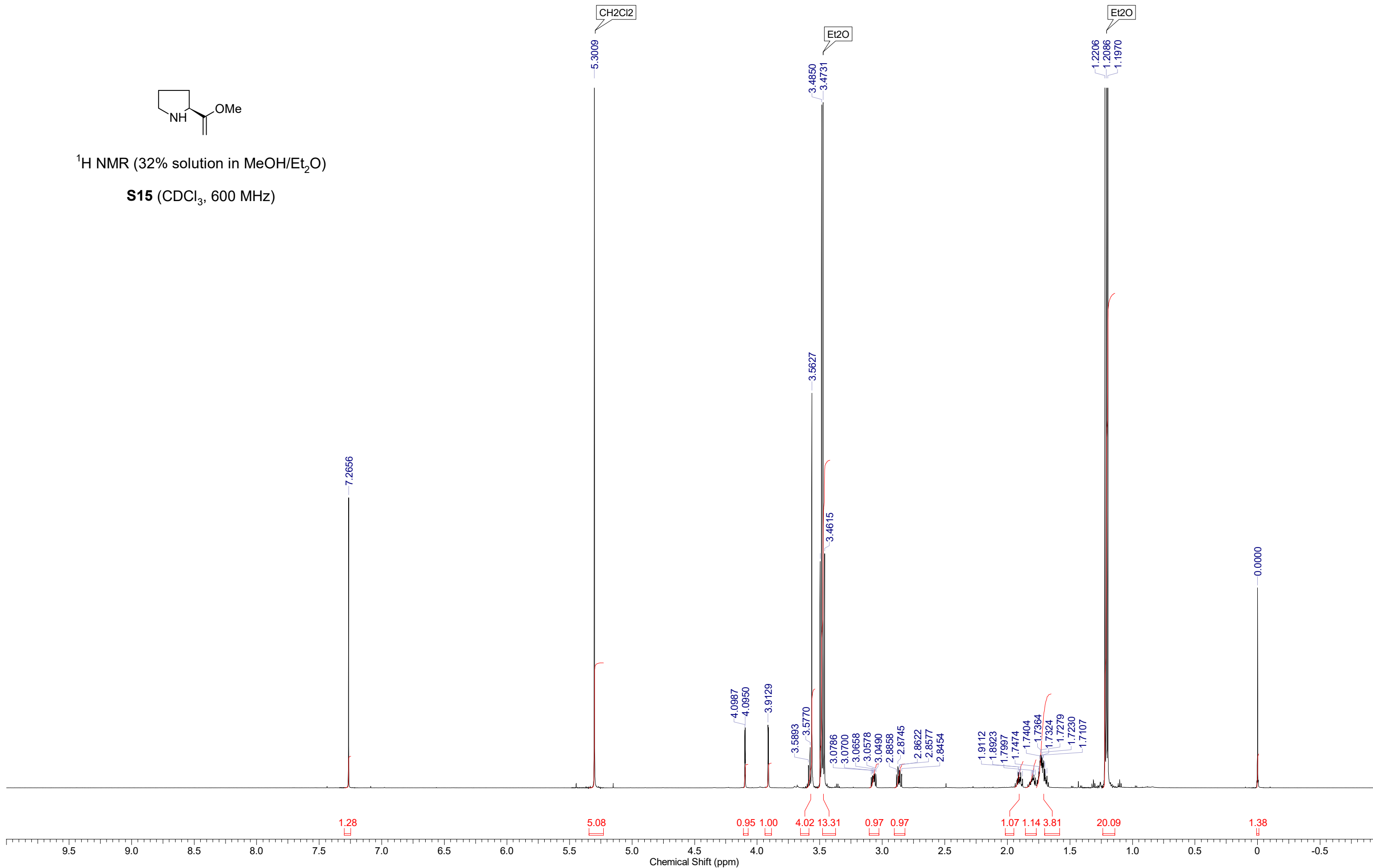


Acquisition Time (sec)	2.7263	Comment	proton	Date	06 Nov 2015 19:10:08	Date Stamp	06 Nov 2015 19:10:08
File Name	¥¥Mac¥Home¥Desktop¥mori.lab.NMR¥sakaiNMR¥sakai04s¥sakai04084crude¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3691.7280	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.150	Number of Transients	8
						SM(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

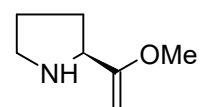


¹H NMR (32% solution in MeOH/Et₂O)

S15 (CDCl₃, 600 MHz)

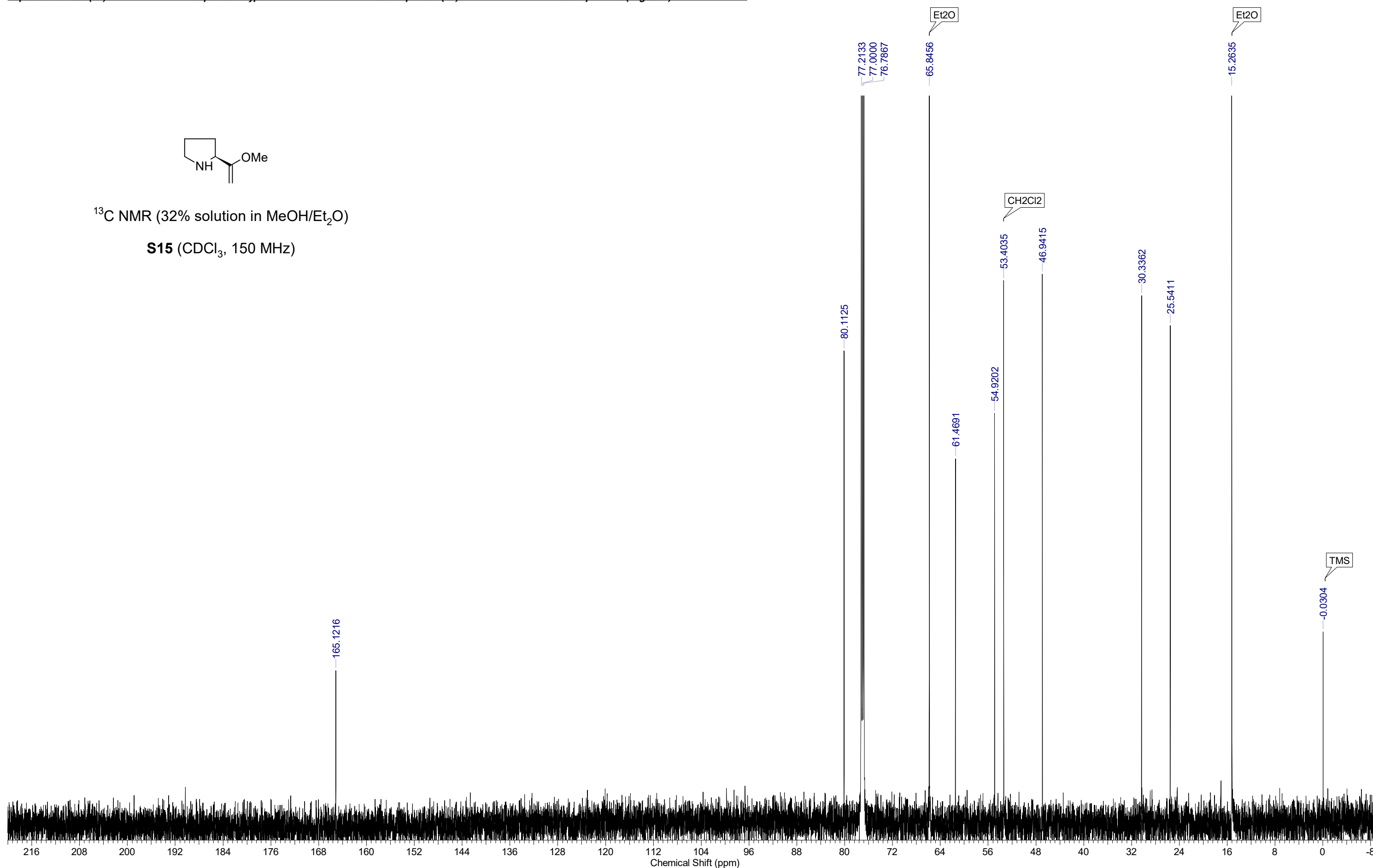


Acquisition Time (sec)	0.8389	Comment	carbon	Date	06 Nov 2015 19:20:48	Date Stamp	06 Nov 2015 19:20:48
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai04s¥sakai04084crude 1¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.152	Number of Transients	128
						SV(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

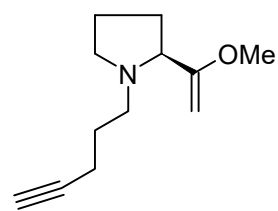


¹³C NMR (32% solution in MeOH/Et₂O)

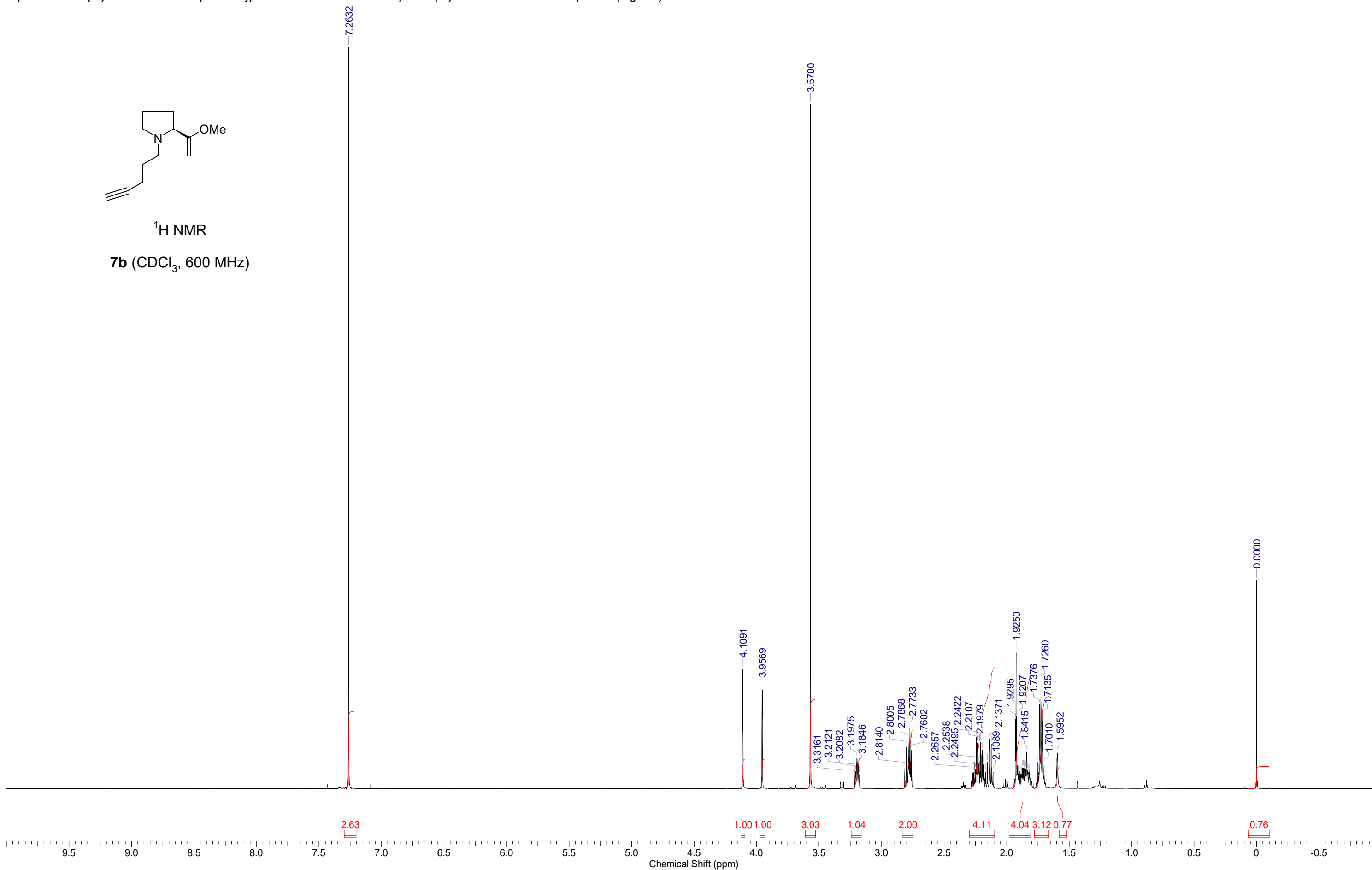
S15 (CDCl₃, 150 MHz)



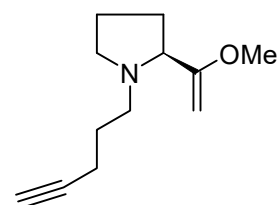
Acquisition Time (sec)	2.7263	Comment	1H	Date	01 Mar 2018 09:59:44	Date Stamp	01 Mar 2018 09:59:44
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02041fr1-5retry¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3693.3787	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.141	Number of Transients	8
						SM(cyclical) (Hz)	12019.23
						Solvent	CHLOROFORM-d



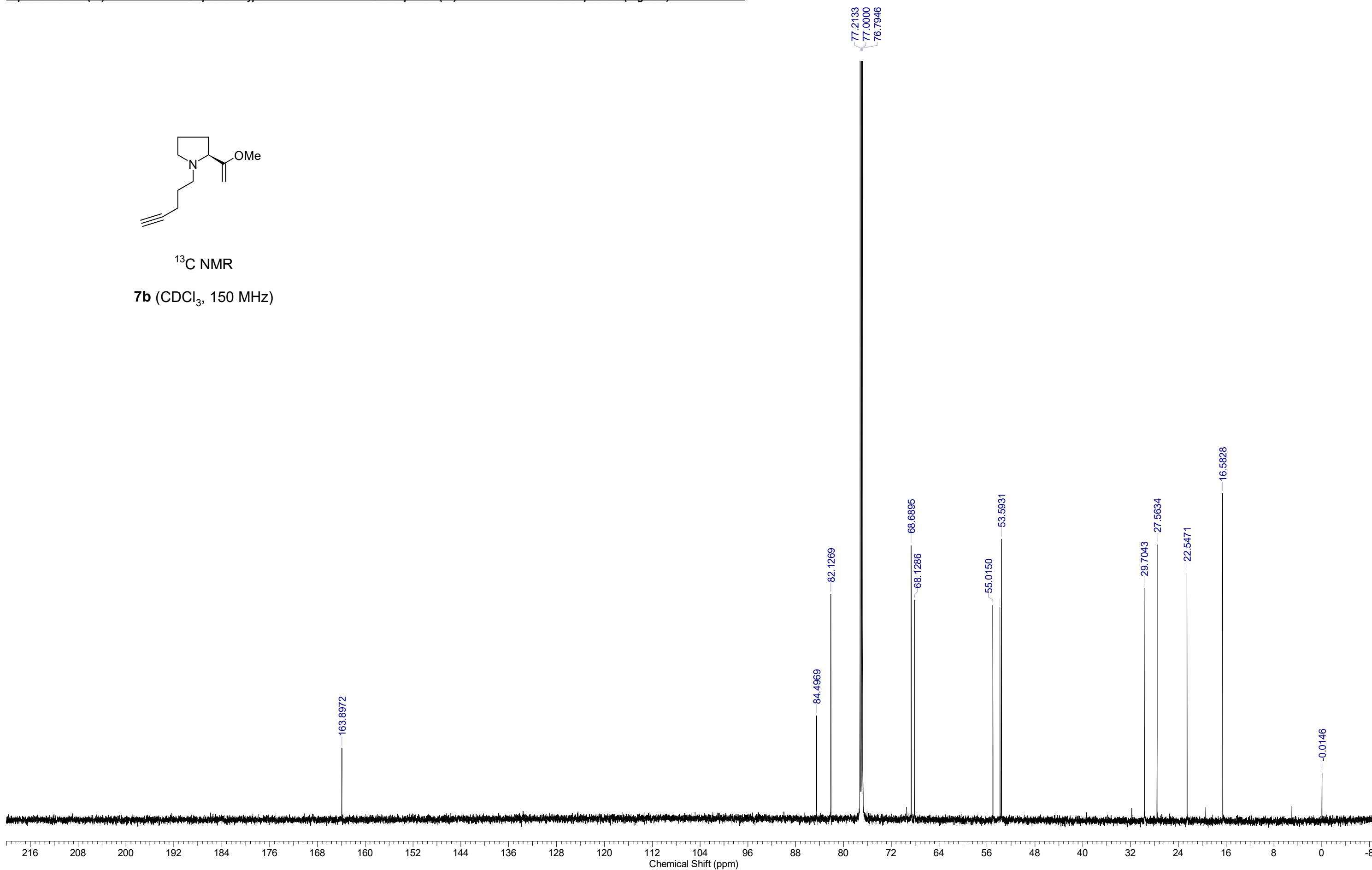
¹H NMR
7b (CDCl₃, 600 MHz)



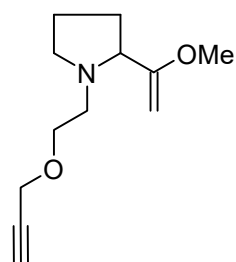
Acquisition Time (sec)	0.8389	Comment	13C	Date	01 Mar 2018 10:12:32	Date Stamp	01 Mar 2018 10:12:32
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02041fr1-5retry¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15086.7188	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.148	Number of Transients	256
						SV(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d



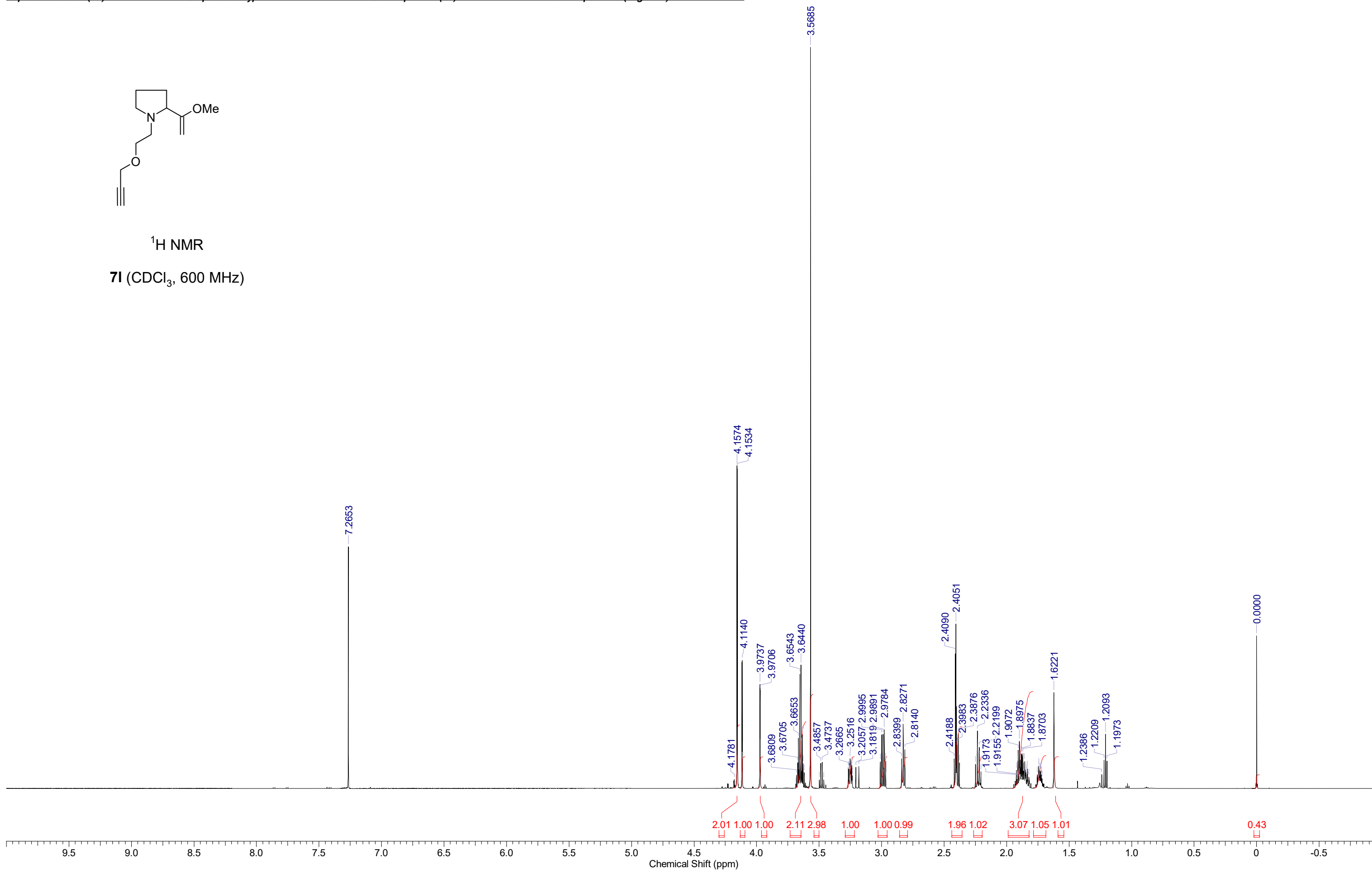
¹³C NMR
7b (CDCl₃, 150 MHz)



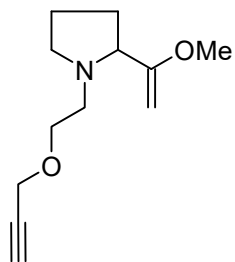
Acquisition Time (sec)	2.7263	Comment	1H	Date	22 May 2019 13:26:40	Date Stamp	22 May 2019 13:26:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab.NMR¥卒業予定2019¥二村将豪¥futamura01098fr27-45¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmrslu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3694.6626	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.147	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d



¹H NMR
7I (CDCl₃, 600 MHz)

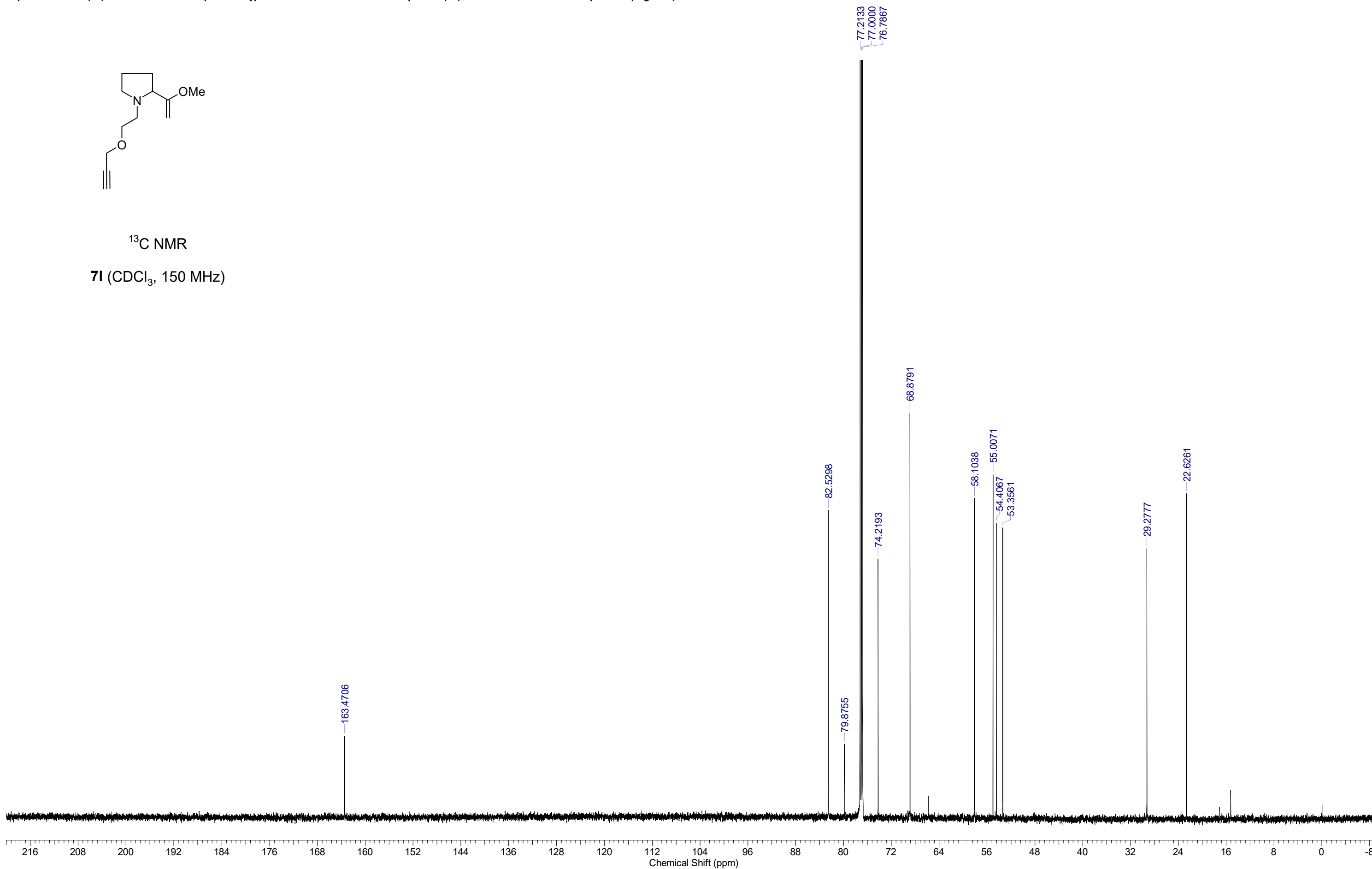


Acquisition Time (sec)	0.8389	Comment	13C	Date	27 May 2019 09:57:36	Date Stamp	27 May 2019 09:57:36
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01098fr27-45¥21¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.169	Number of Transients	128
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

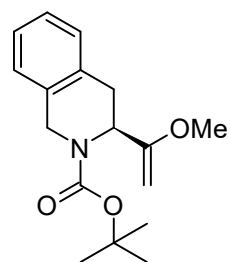


¹³C NMR

71 (CDCl₃, 150 MHz)

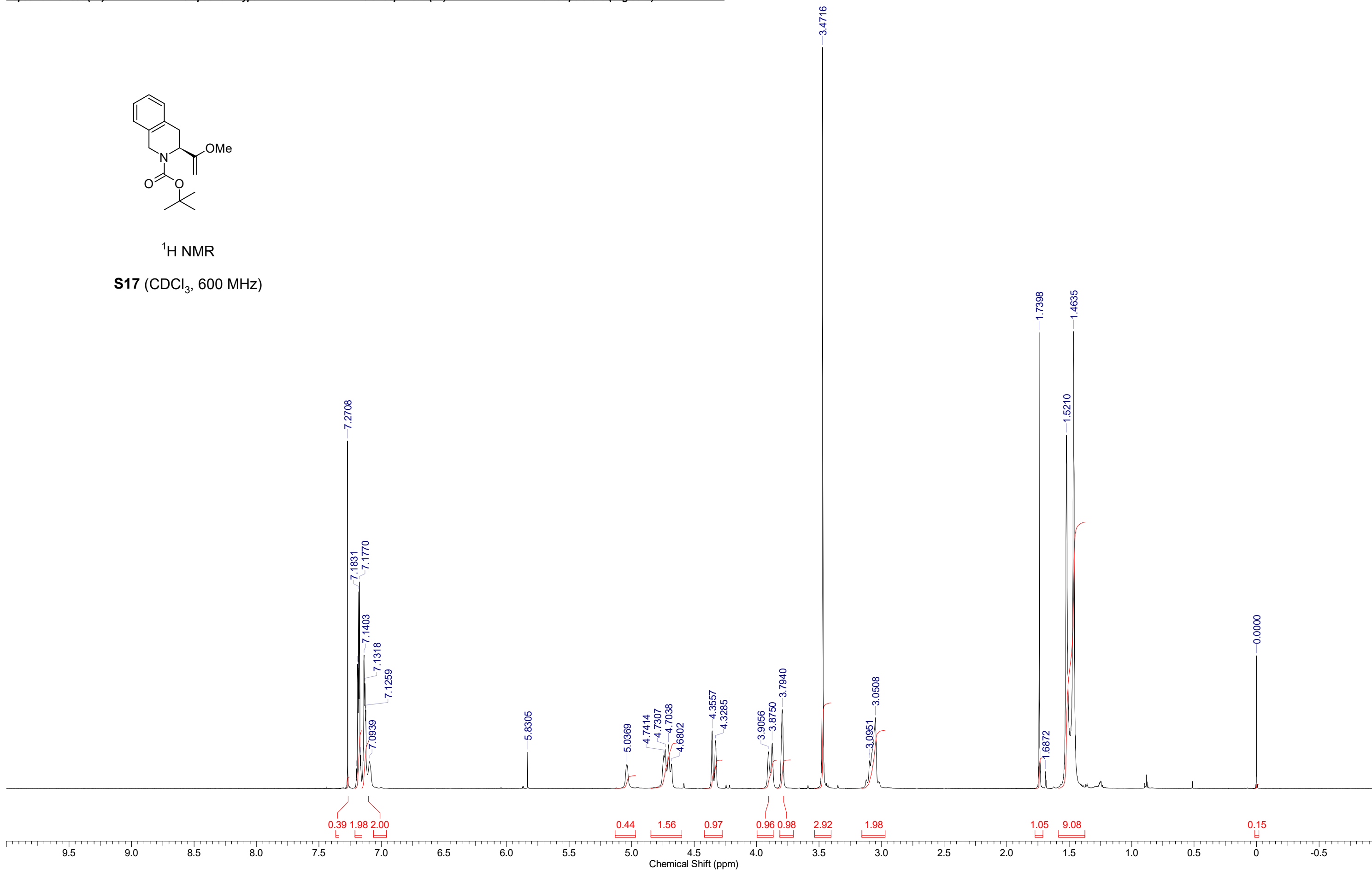


Acquisition Time (sec)	2.7263	Comment	proton	Date	25 Sep 2019 19:59:12	Date Stamp	25 Sep 2019 19:59:12
File Name	¥¥Mac¥Home¥Desktop¥mori_lab_NMR¥sakaiNMR¥sakai06093_0deg¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nrmrsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3697.2302	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
				Temperature (degree C)	0.158	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

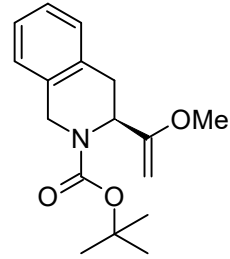


¹H NMR

S17 (CDCl₃, 600 MHz)

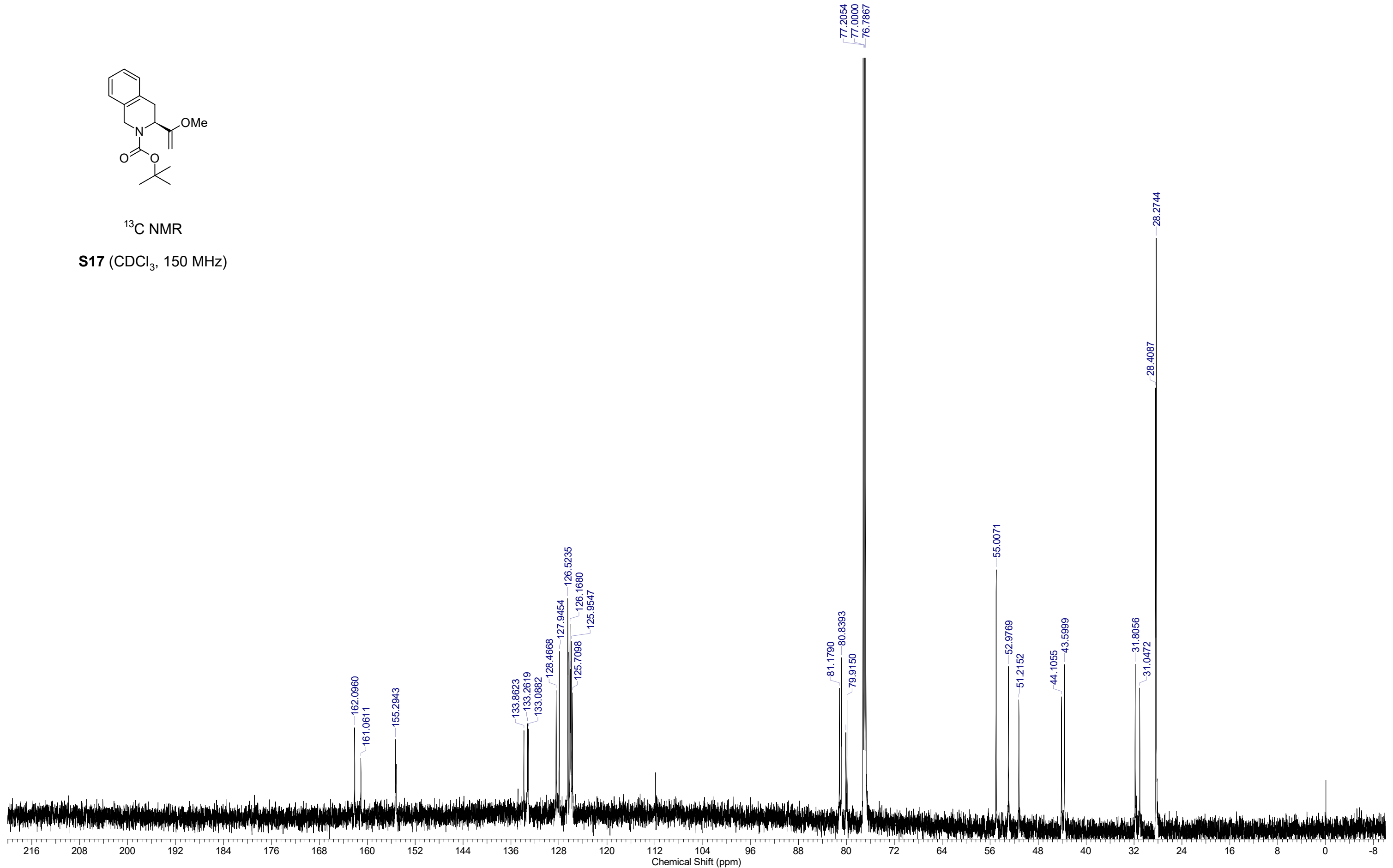


Acquisition Time (sec)	0.8389	Comment	carbon	Date	25 Sep 2019 20:05:36	Date Stamp	25 Sep 2019 20:05:36
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06093_0deg¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	¹³ C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15070.0283	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	0.129	Number of Transients	128
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

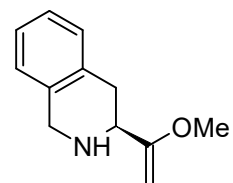


¹³C NMR

S17 (CDCl₃, 150 MHz)

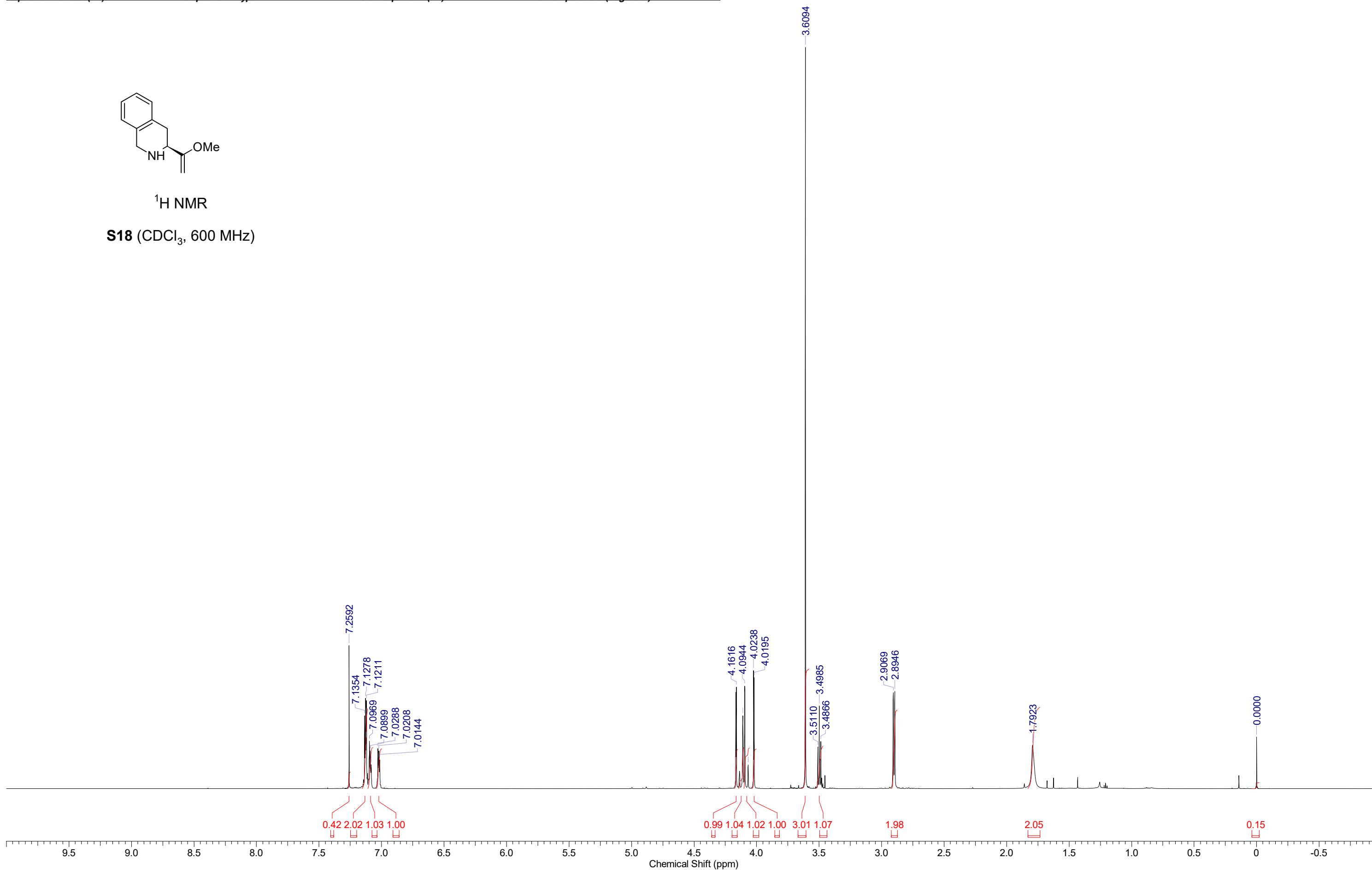


Acquisition Time (sec)	2.7263	Comment	proton	Date	22 Oct 2019 16:25:52	Date Stamp	22 Oct 2019 16:25:52
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06099¥1¥pdata¥1¥1r	Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3691.1780	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.143	SV(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

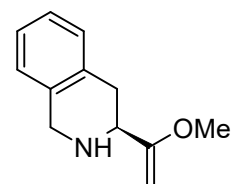


¹H NMR

S18 (CDCl₃, 600 MHz)

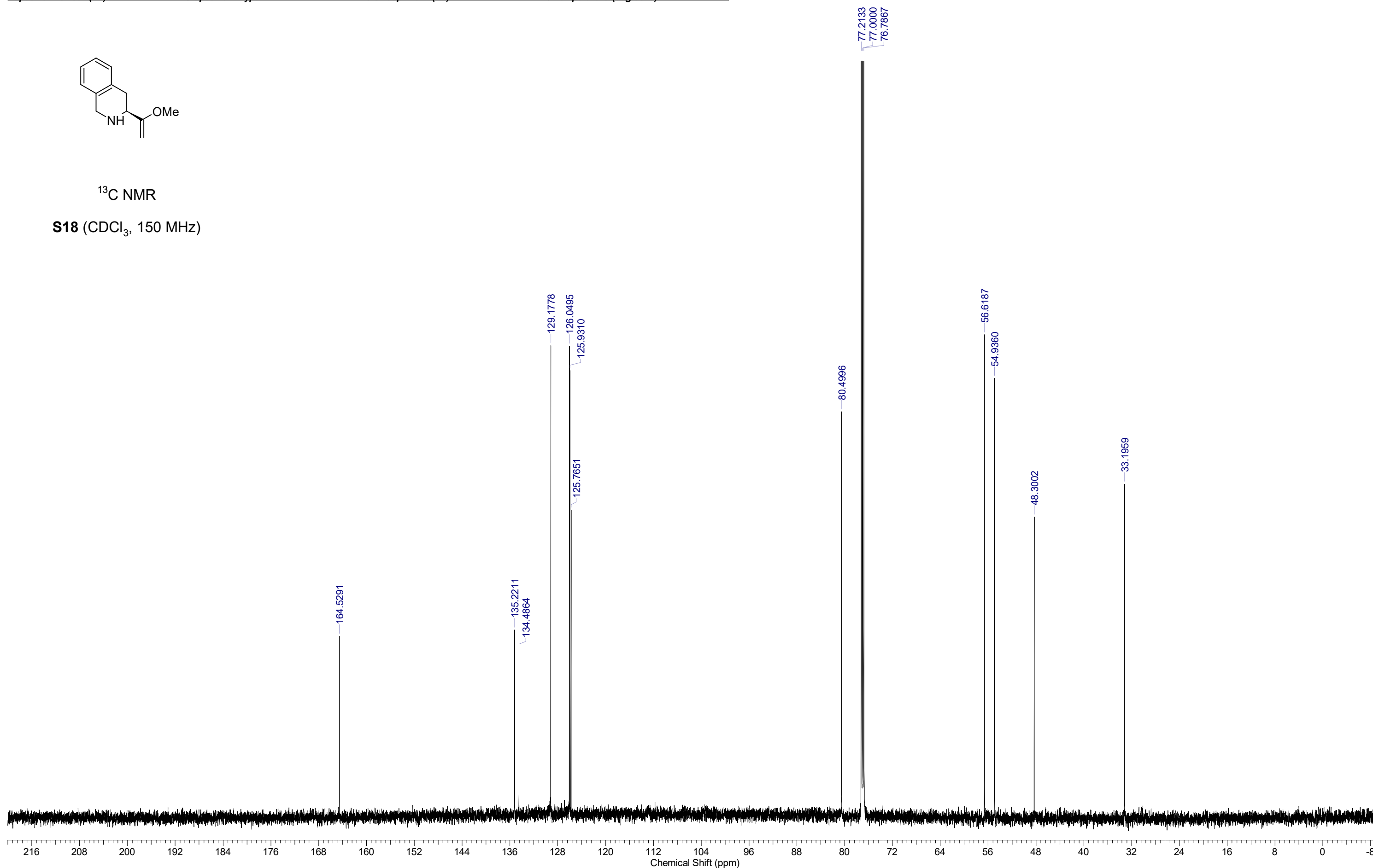


Acquisition Time (sec)	0.8389	Comment	carbon	Date	22 Oct 2019 16:32:16	Date Stamp	22 Oct 2019 16:32:16
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06099¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	n.mrsu	Points Count	32768	Pulse Sequence	zpg30
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.144	Number of Transients	64
						SV(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

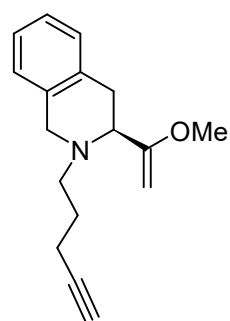


¹³C NMR

S18 (CDCl₃, 150 MHz)

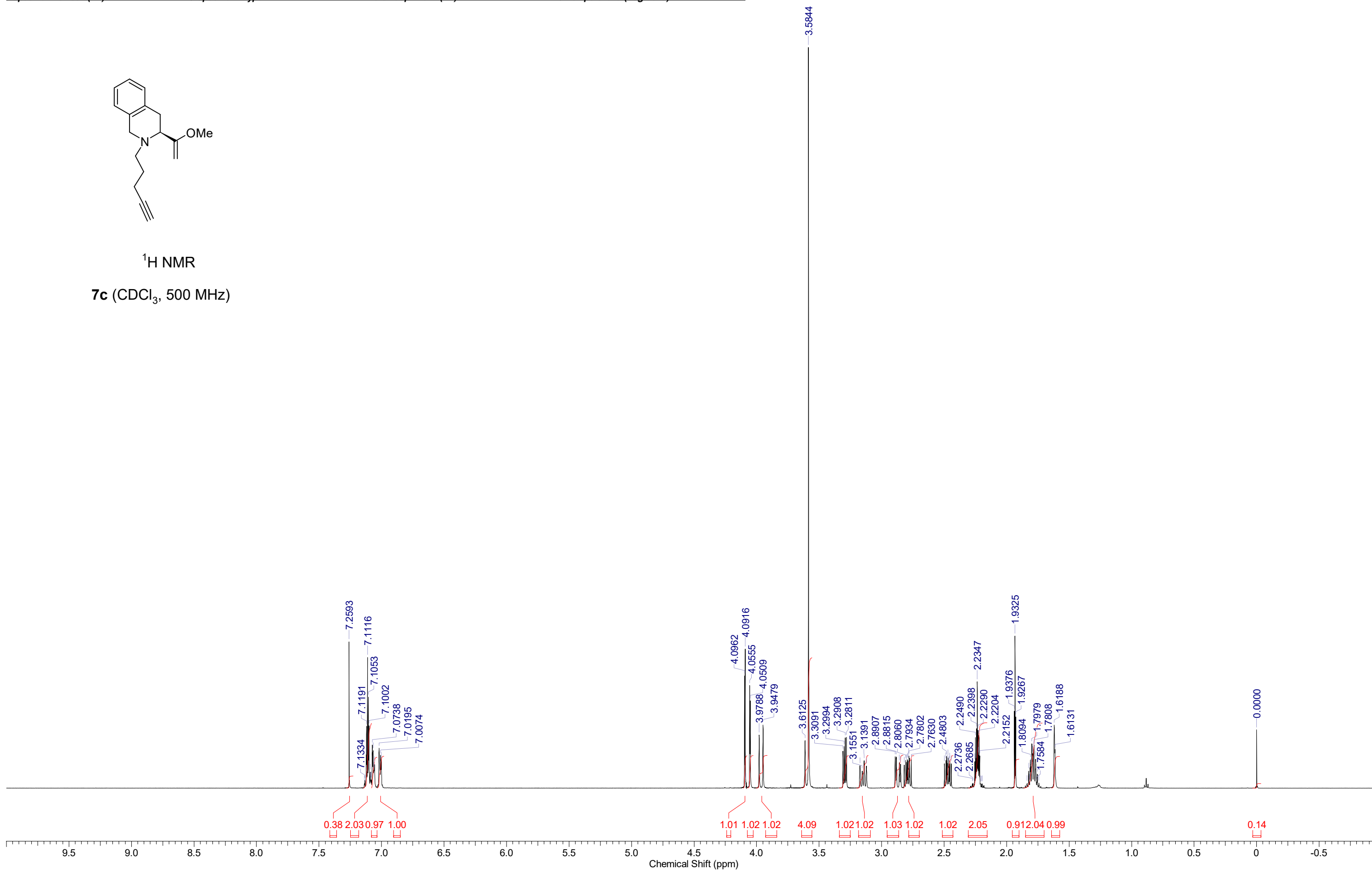


Acquisition Time (sec)	1.7642	Comment	single pulse	Date	23 Oct 2019 12:41:28	Date Stamp	23 Oct 2019 12:45:27
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06100¥sakai06100H-1.jdf			Frequency (MHz)	495.13	Nucleus	1H
Original Points Count	16384	Owner	delta	Points Count	32768	Pulse Sequence	single_pulse.ex2
Spectrum Offset (Hz)	2478.3535	Spectrum Type	STANDARD	Sweep Width (Hz)	9286.78	Receiver Gain	46.00
				Temperature (degree C)	23.500	Number of Transients	8
						Solvent	CHLOROFORM-d
						Origin	ECA 500

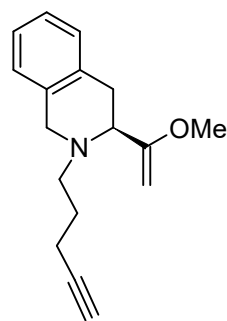


¹H NMR

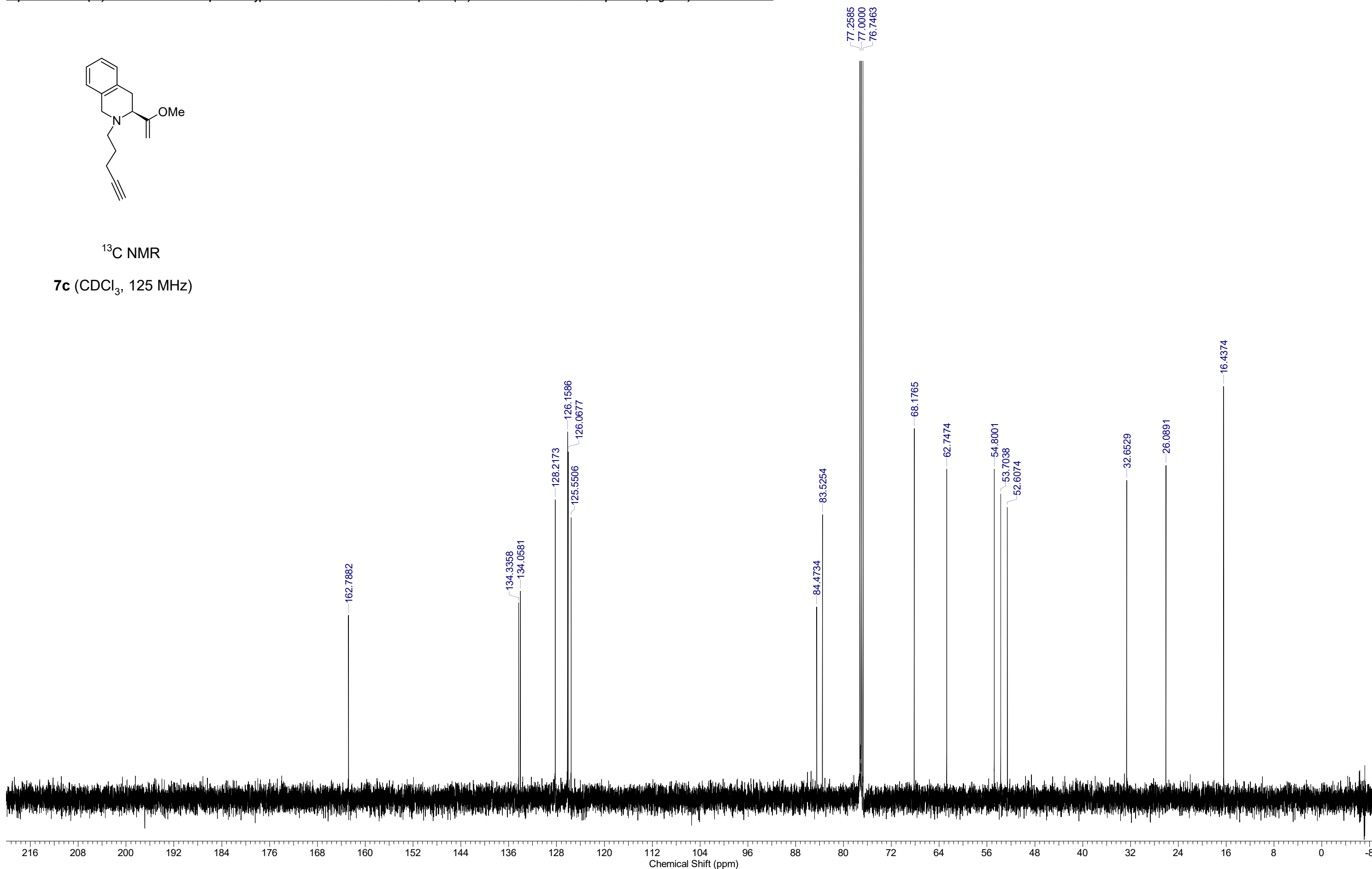
7c (CDCl₃, 500 MHz)



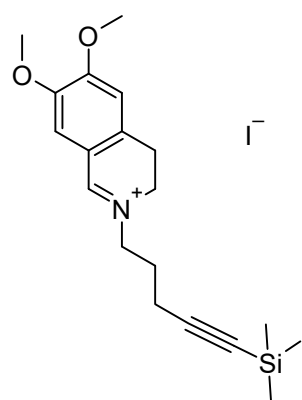
Acquisition Time (sec)	0.8389	Comment	single_pulse_decoupled_gated_NOE	Date	23 Oct 2019 12:52:27	Date Stamp	23 Oct 2019 12:56:26
File Name	¥¥Mac¥Home¥Desktop¥mori_lab_NMR¥sakaiNMR¥sakai06100¥sakai06100-1.jdf			Frequency (MHz)	124.50	Nucleus	13C
Original Points Count	32768	Owner	delta	Points Count	65536	Pulse Sequence	single_pulse_dec
Spectrum Offset (Hz)	12436.6172	Spectrum Type	STANDARD	Sweep Width (Hz)	39062.50	Receiver Gain	60.00
				Temperature (degree C)	23.800	Solvent	CHLOROFORM-d



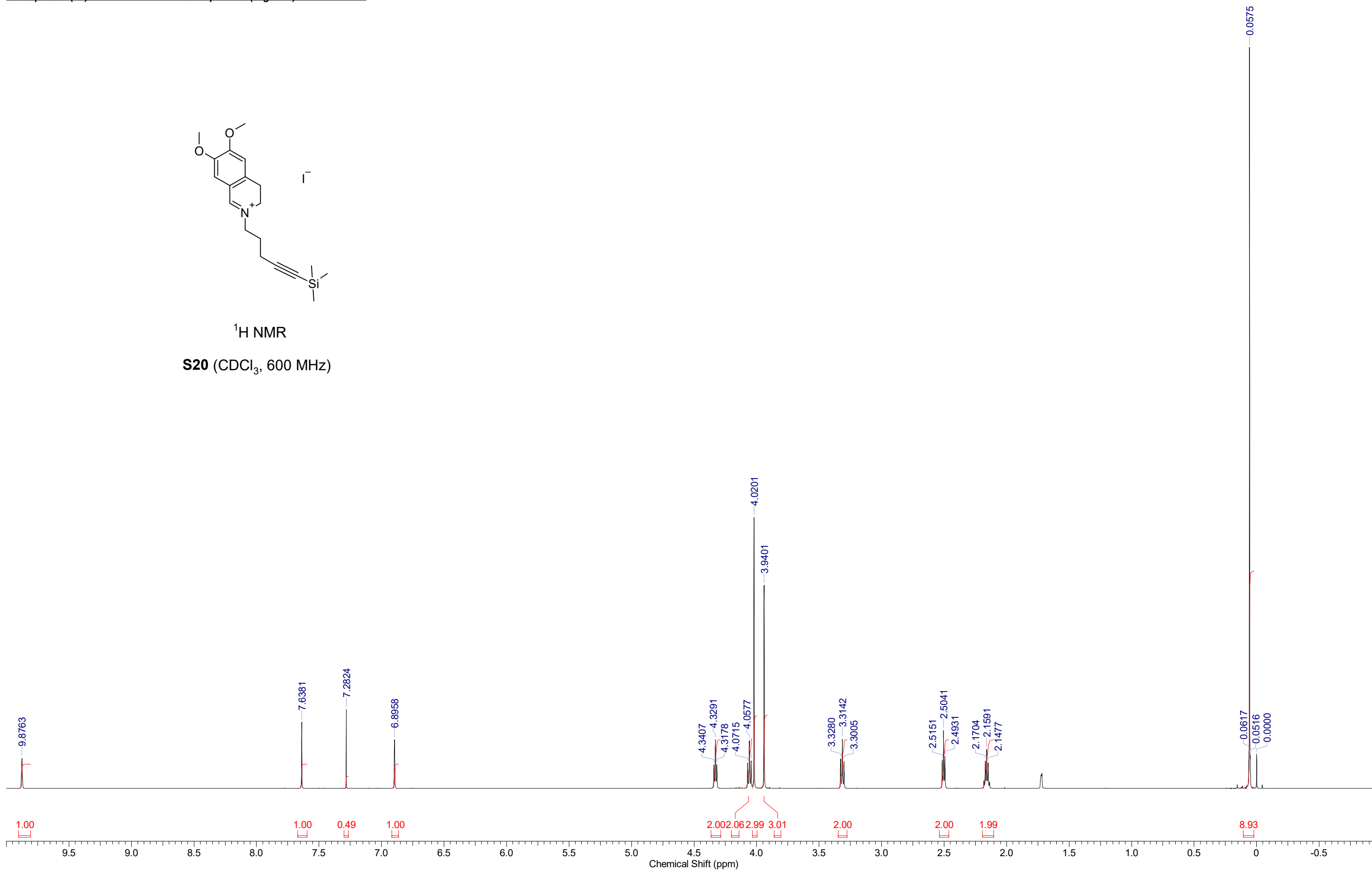
¹³C NMR
7c (CDCl₃, 125 MHz)



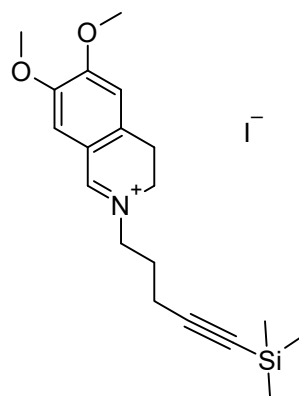
Acquisition Time (sec)	2.7263	Date	24 Mar 2020 14:37:04	Date Stamp	24 Mar 2020 14:37:04	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07045¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	30.91	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.154			Spectrum Offset (Hz)	3704.5659
						Points Count	65536
						Spectrum Type	STANDARD



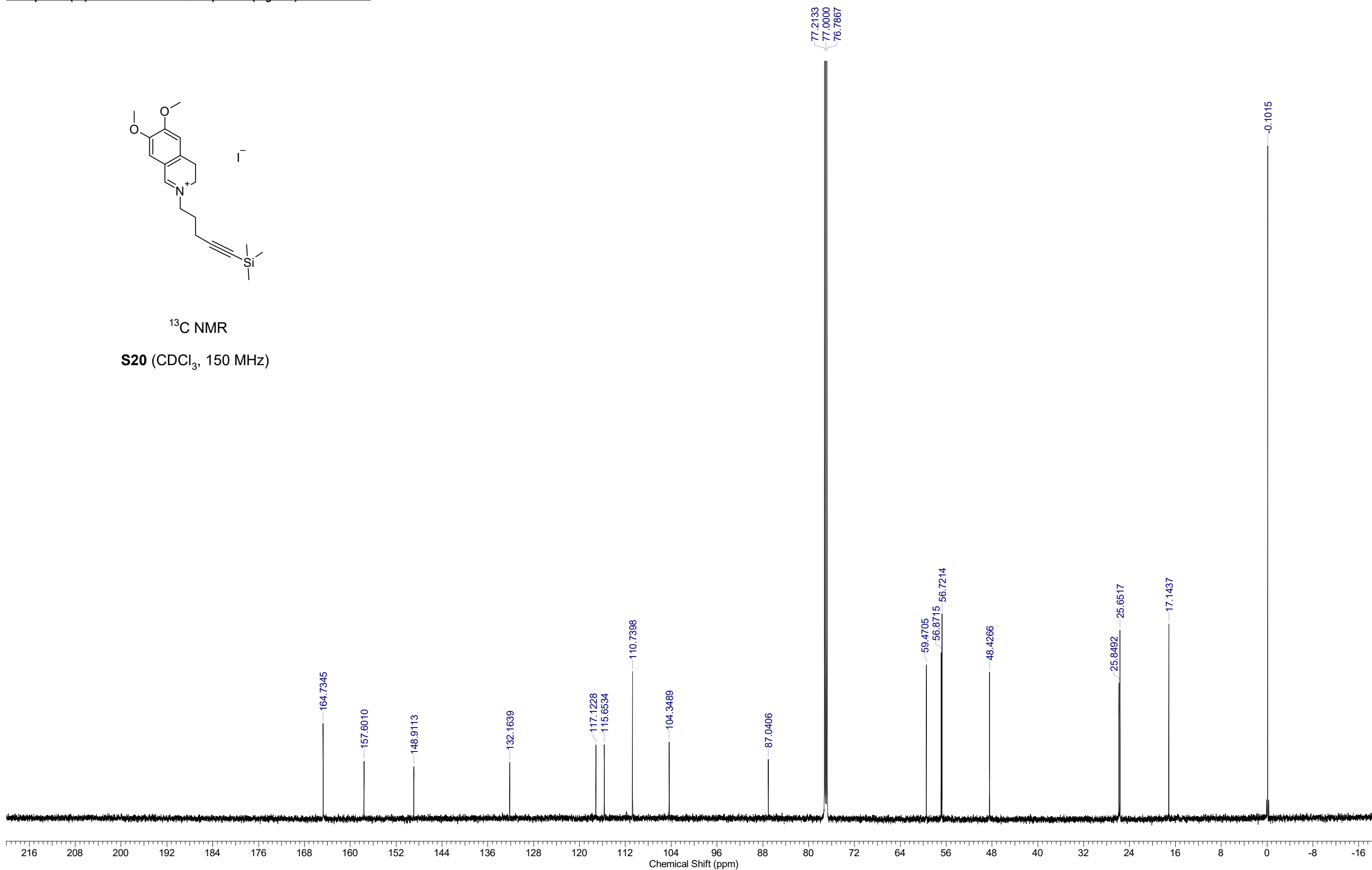
¹H NMR
S20 (CDCl₃, 600 MHz)



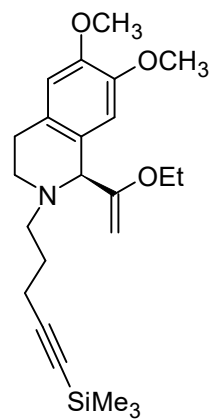
Acquisition Time (sec)	0.8389	Date	24 Mar 2020 14:43:28	Date Stamp	24 Mar 2020 14:43:28	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07045¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	13C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.126			Spectrum Offset (Hz)	15079.5664
						Points Count	32768
						Spectrum Type	STANDARD



¹³C NMR
S20 (CDCl₃, 150 MHz)

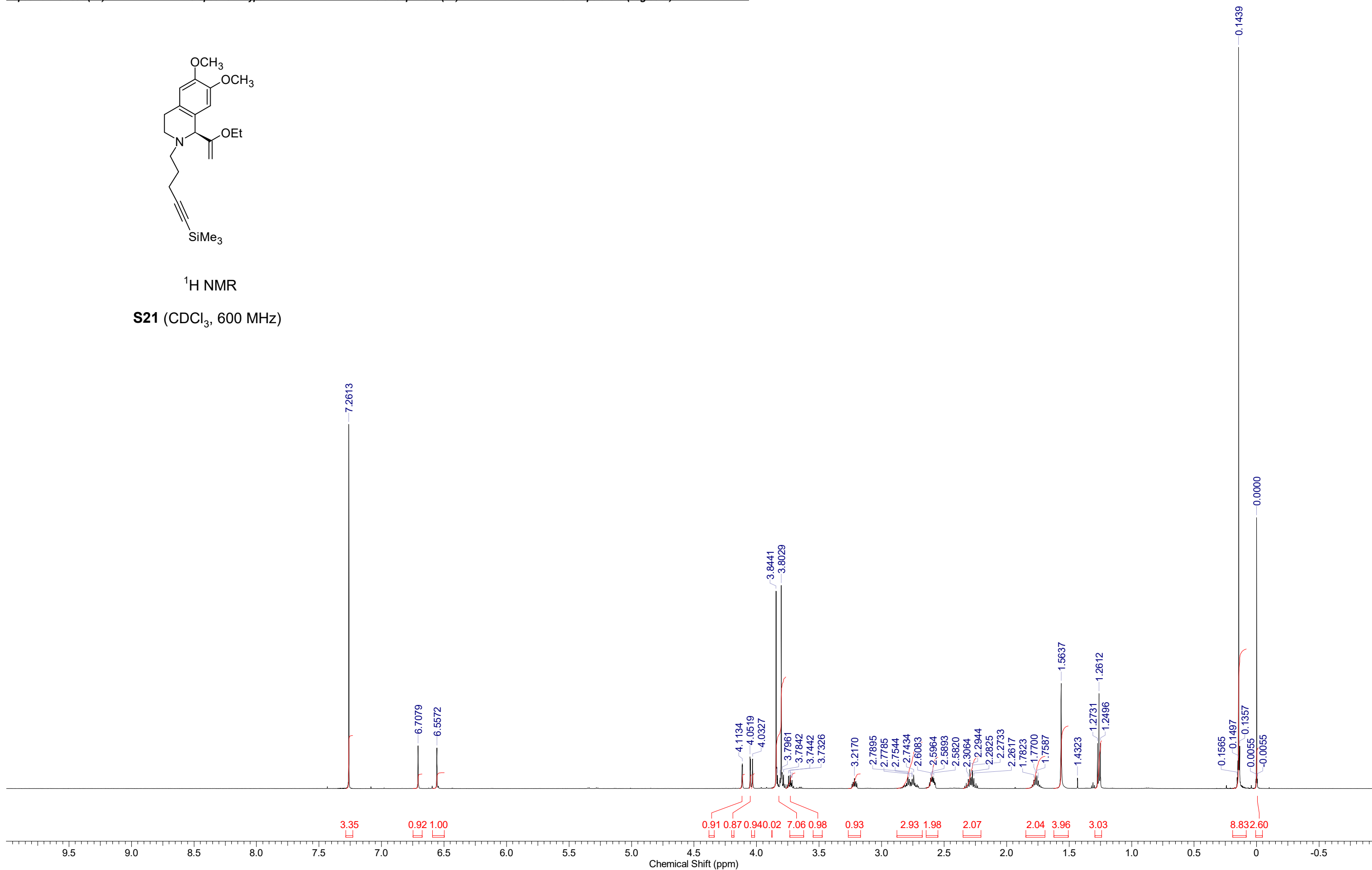


Acquisition Time (sec)	2.7263	Date	19 Nov 2018 12:14:08	Date Stamp	19 Nov 2018 12:14:08						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥長江紋加莉¥nagae01033_5¥10¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H					
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30	Receiver Gain	30.91	Number of Transients	8	Origin	spect
Spectrum Offset (Hz)	3692.2783	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.153	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d

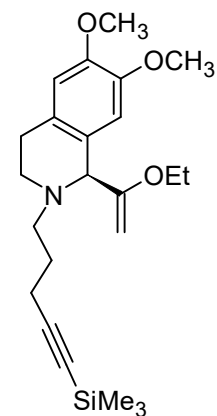


¹H NMR

S21 (CDCl₃, 600 MHz)

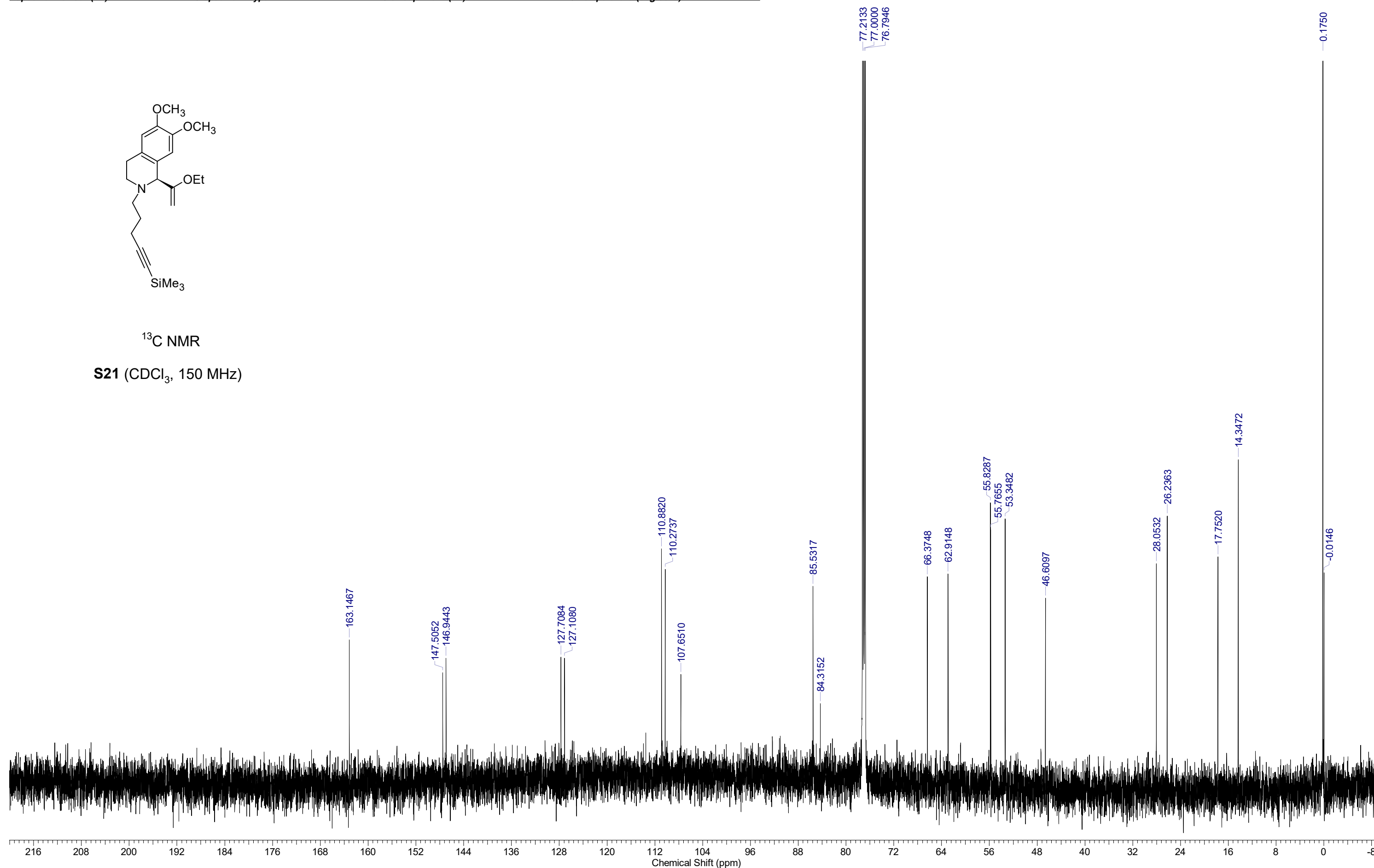


Acquisition Time (sec)	0.8389	Date	19 Nov 2018 12:22:40	Date Stamp	19 Nov 2018 12:22:40	
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥長江紋加莉¥nagae01033_6¥10¥pdata¥1¥1r			Frequency (MHz)	150.90	
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30	
Spectrum Offset (Hz)	15086.7197	Spectrum Type	STANDARD	Receiver Gain	193.26	
		Sweep Width (Hz)	39061.31	Number of Transients	128	
			Temperature (degree C)	25.135	SV(cyclical) (Hz)	39062.50
					Origin	spect
					Solvent	CHLOROFORM-d

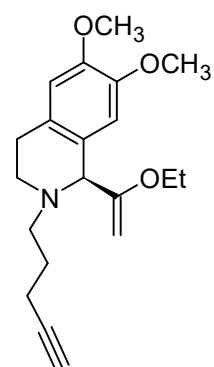


¹³C NMR

S21 (CDCl₃, 150 MHz)

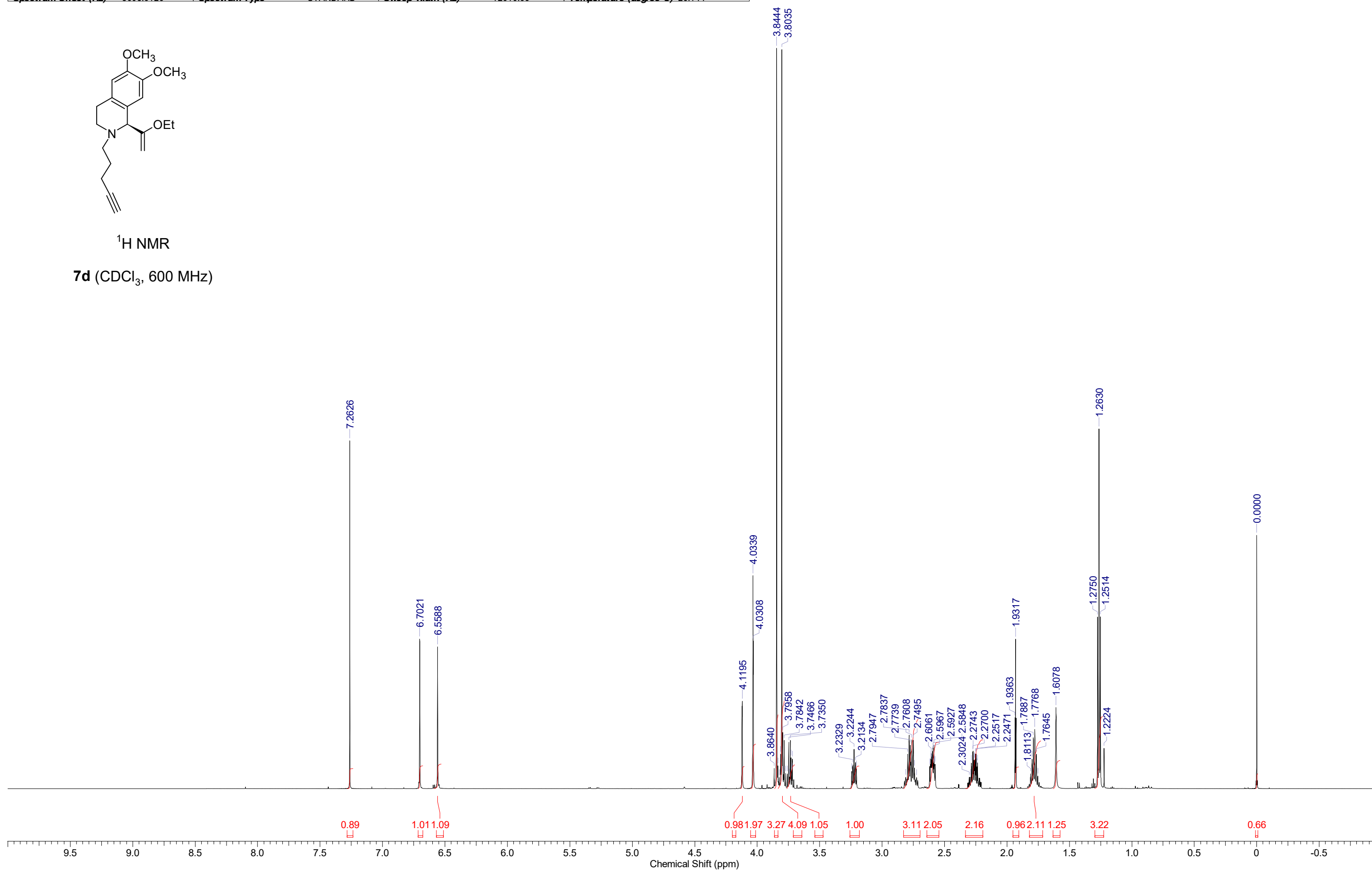


Acquisition Time (sec)	2.7263	Date	22 Nov 2018 14:09:20	Date Stamp	22 Nov 2018 14:09:20						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥長江紋加莉¥nagae01034.2¥10¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H					
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30	Receiver Gain	22.72	Number of Transients	8	Origin	spect
Spectrum Offset (Hz)	3693.0120	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.144	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d

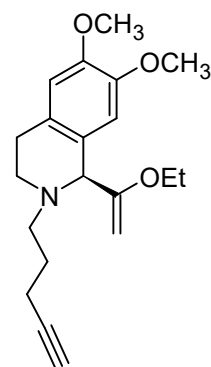


¹H NMR

7d (CDCl₃, 600 MHz)

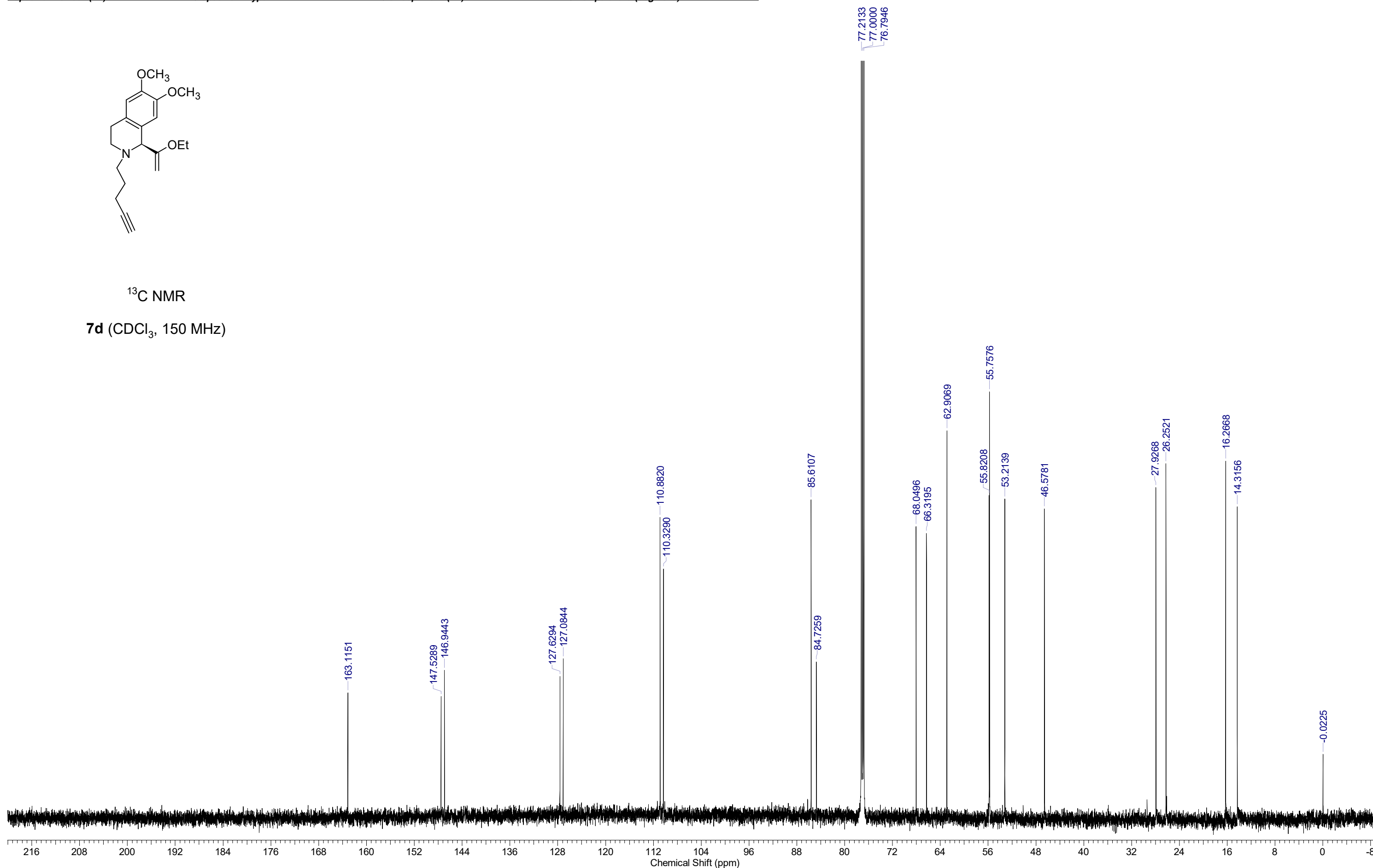


Acquisition Time (sec)	0.8389	Date	22 Nov 2018 14:17:52	Date Stamp	22 Nov 2018 14:17:52	
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥長江紋加莉¥nagae01034_3¥10¥pdata¥1¥1r			Frequency (MHz)	150.90	
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30	
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Receiver Gain	193.26	
		Sweep Width (Hz)	39061.31	Number of Transients	128	
			Temperature (degree C)	25.141	SV(cyclical) (Hz)	39062.50
				Nucleus	13C	
				Solvent	CHLOROFORM-d	

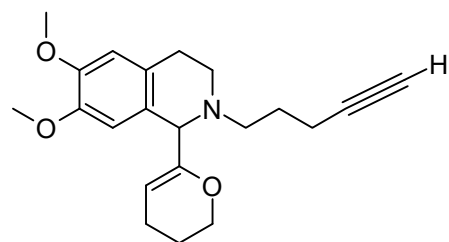


¹³C NMR

7d (CDCl₃, 150 MHz)

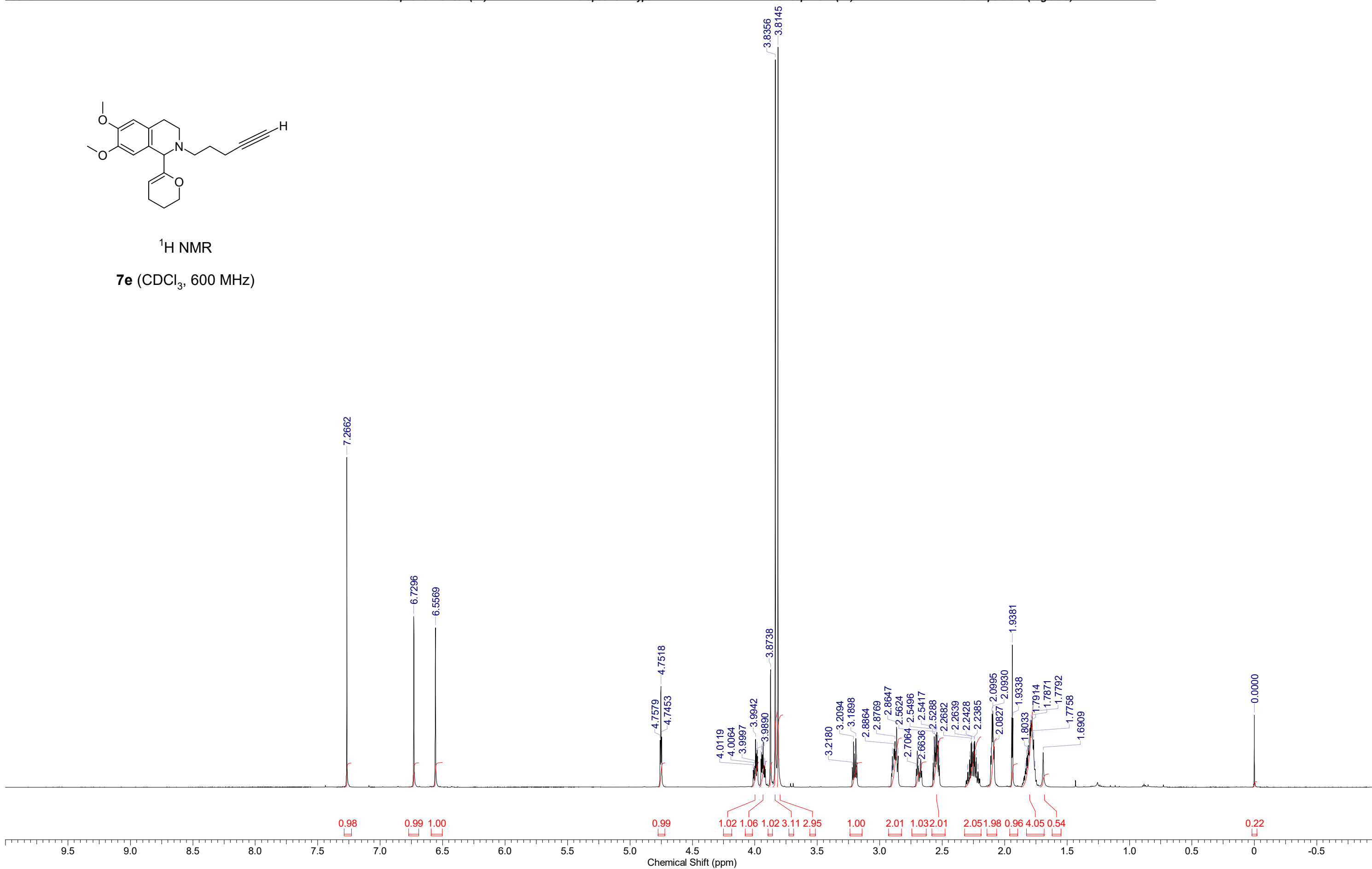


Acquisition Time (sec)	2.7263	Comment	1H	Date	15 Jan 2020 19:20:48	Date Stamp	15 Jan 2020 19:20:48
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01043¥も の ¥okumura01043_monofr13-30H¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3695.2126	Points Count	65536	Receiver Gain	25.50
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.151
						Number of Transients	8
						SW(cyclical) (Hz)	12019.23

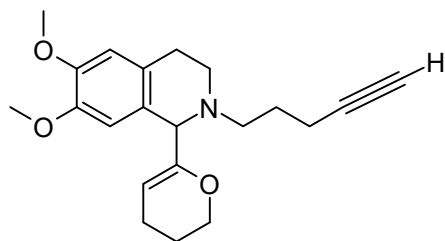


¹H NMR

7e (CDCl₃, 600 MHz)

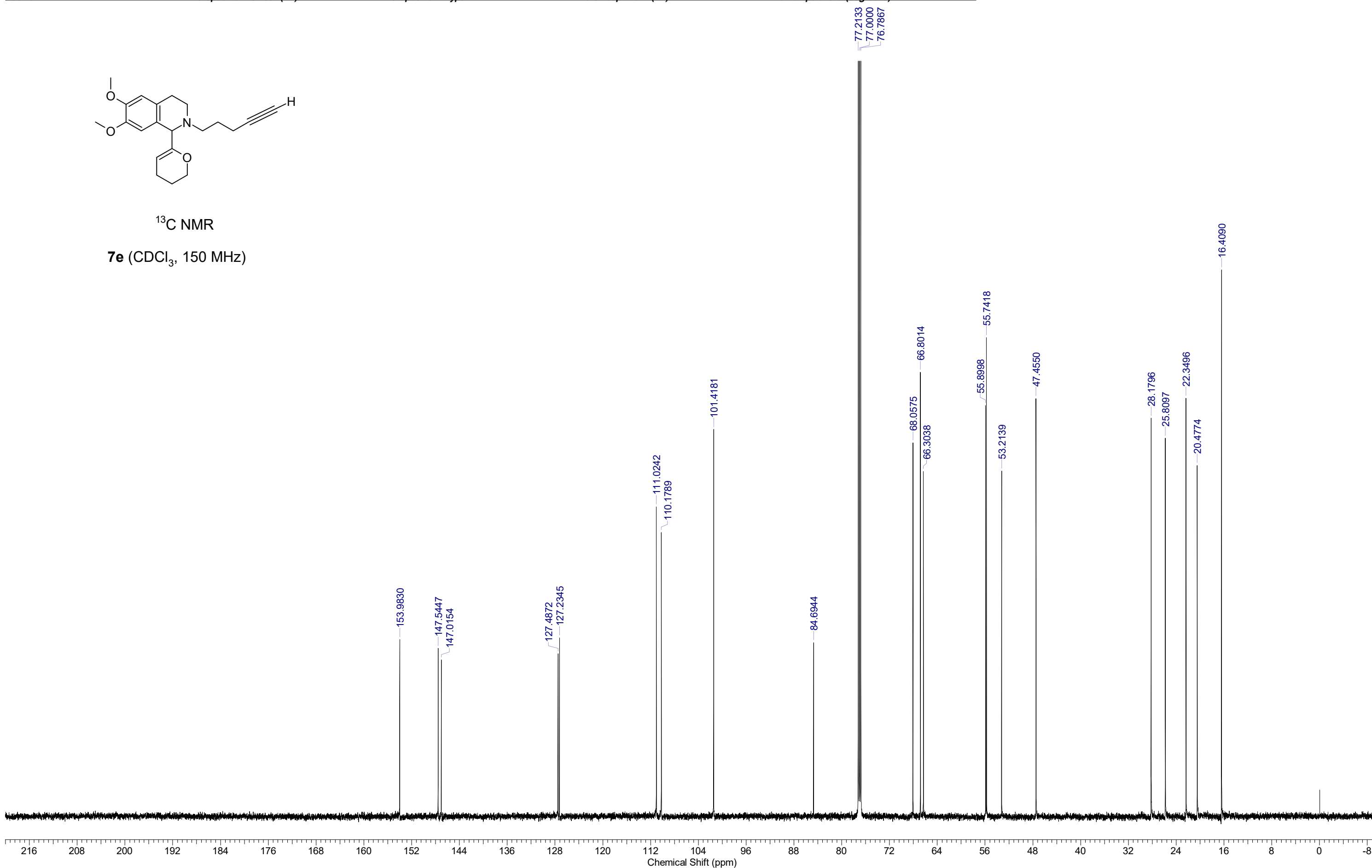


Acquisition Time (sec)	0.8389	Comment	13C	Date	15 Jan 2020 19:29:20	Date Stamp	15 Jan 2020 19:29:20
File Name	¥¥Mac¥Home¥Desktop¥mori_lab_NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01043¥もくの¥okumura01043_monofr13-30C¥10¥pdata¥1¥1r	Original Points Count	32768	Owner	nmsu	Frequency (MHz)	150.90
Origin	spect	Spectrum Offset (Hz)	15081.9492	Points Count	32768	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-d	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
						Temperature (degree C)	25.157
						Number of Transients	128
						SW(cyclical) (Hz)	39062.50

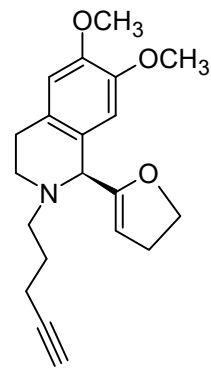


¹³C NMR

7e (CDCl₃, 150 MHz)

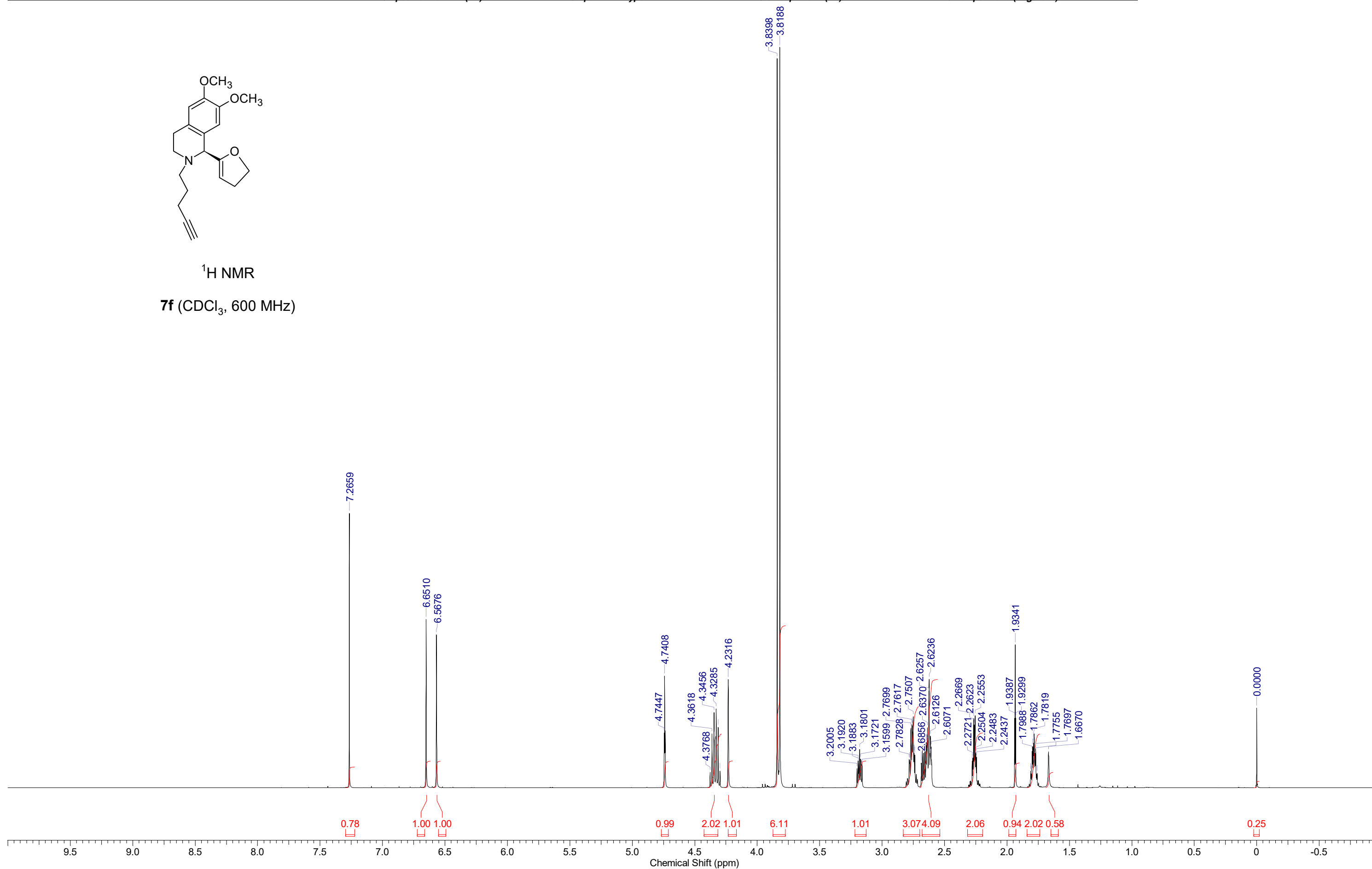


Acquisition Time (sec)	2.7263	Comment	1H	Date	23 Jan 2020 14:37:04	Date Stamp	23 Jan 2020 14:37:04
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01047¥okumura01047.fr8-12¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3695.0293	Points Count	65536	Receiver Gain	22.72
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.152
						Number of Transients	8
						SM(cyclical) (Hz)	12019.23

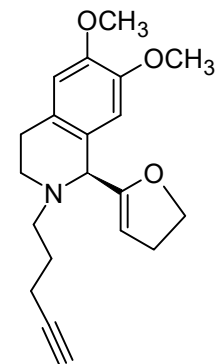


¹H NMR

7f (CDCl₃, 600 MHz)

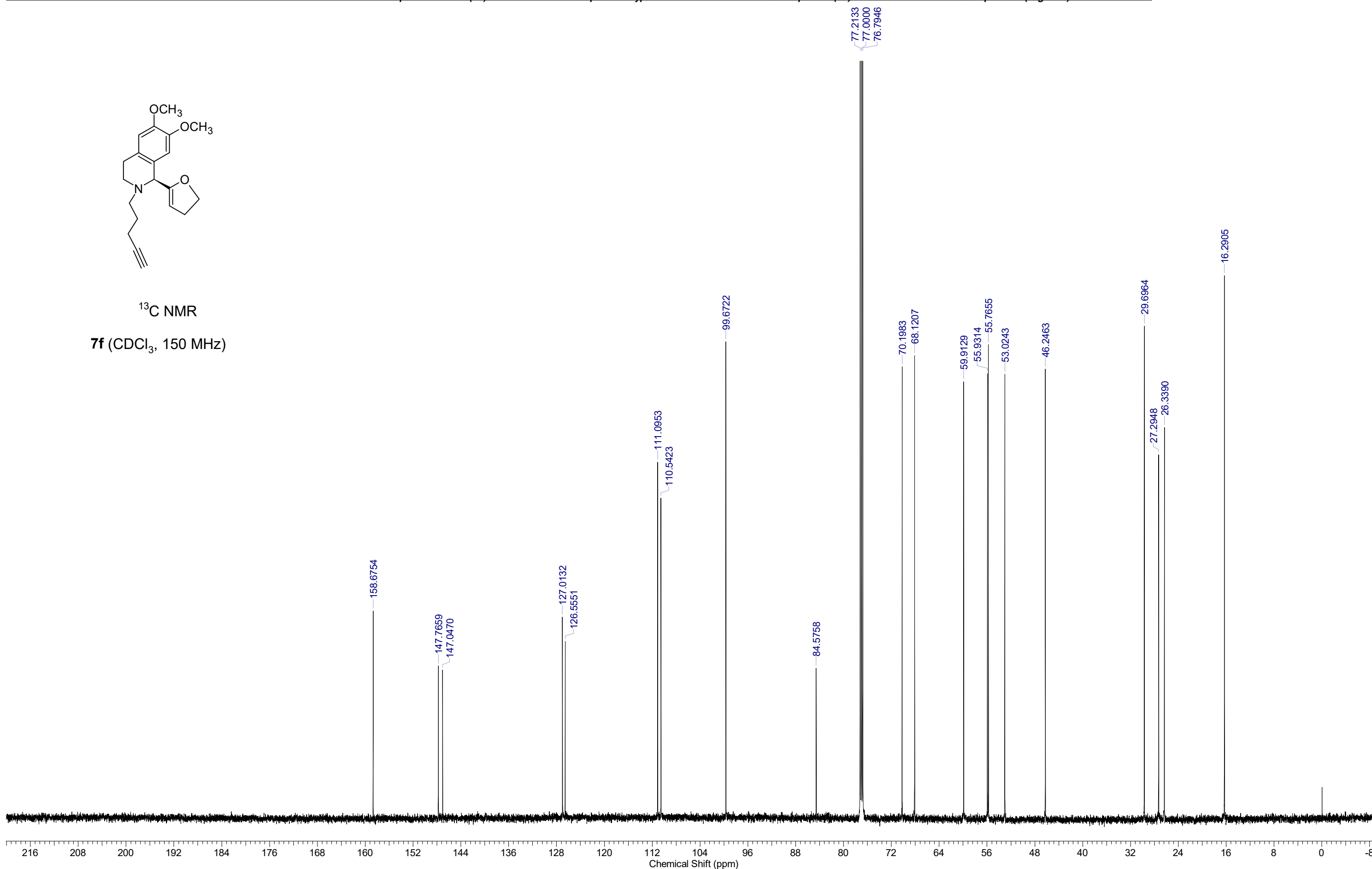


Acquisition Time (sec)	0.8389	Comment	13C	Date	23 Jan 2020 14:45:36	Date Stamp	23 Jan 2020 14:45:36
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01047¥okumura01047 fr8-12¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15083.1416	Points Count	32768	Receiver Gain	193.26
		Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.140
						Number of Transients	128
						SV(cyclical) (Hz)	39062.50

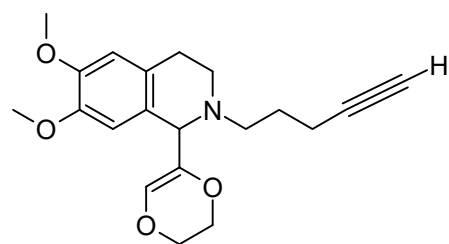


¹³C NMR

7f (CDCl₃, 150 MHz)

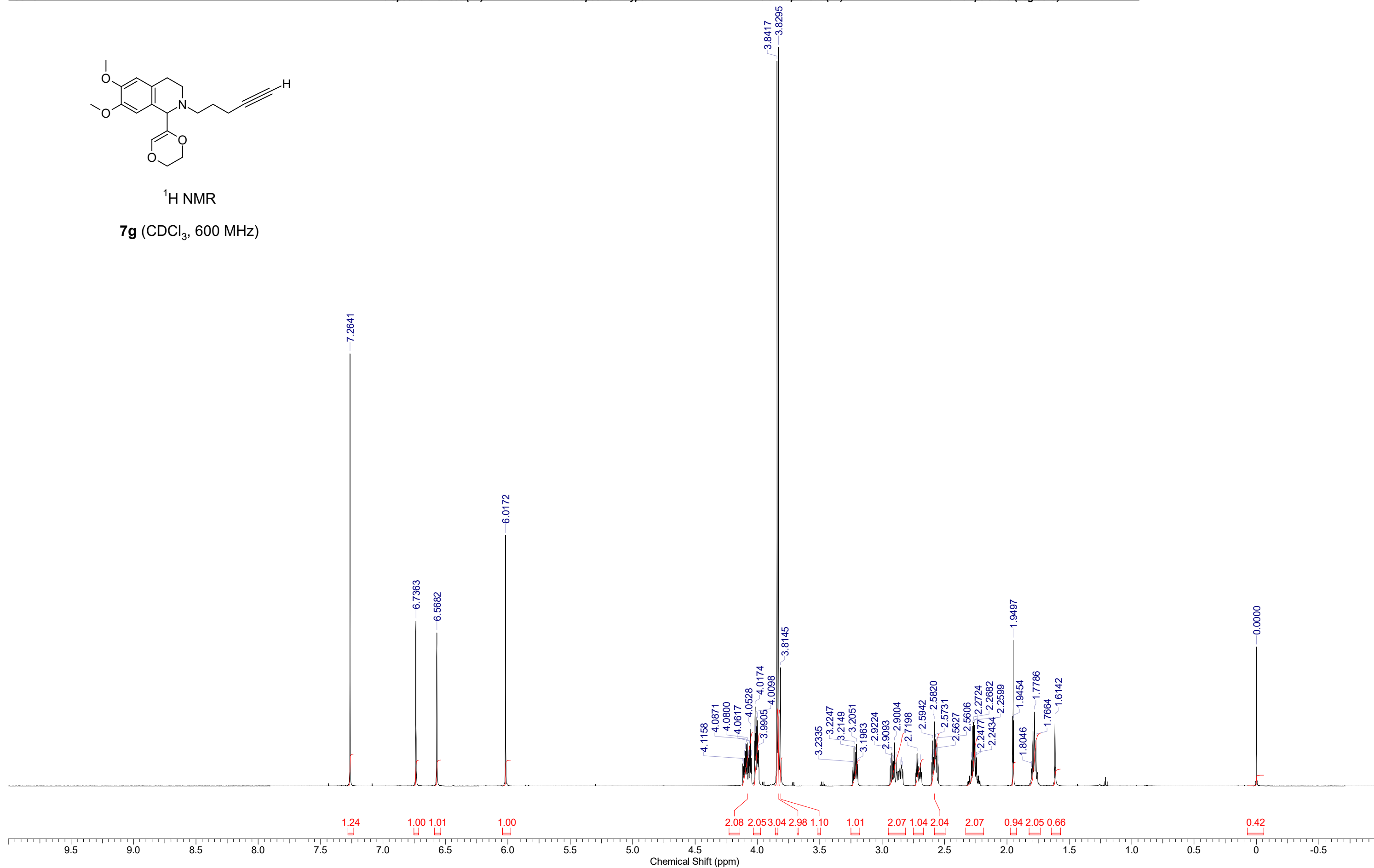


Acquisition Time (sec)	2.7263	Comment	1H	Date	20 Jan 2020 17:06:24	Date Stamp	20 Jan 2020 17:06:24
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01045¥okumura01045 fr18-20¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3693.9290	Points Count	65536	Receiver Gain	25.50
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.146
						Number of Transients	8
						SW(cyclical) (Hz)	12019.23

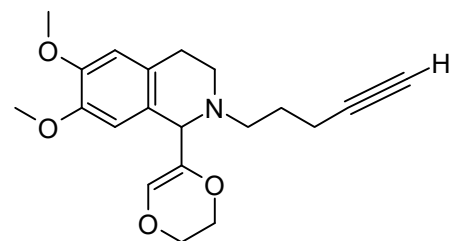


¹H NMR

7g (CDCl₃, 600 MHz)

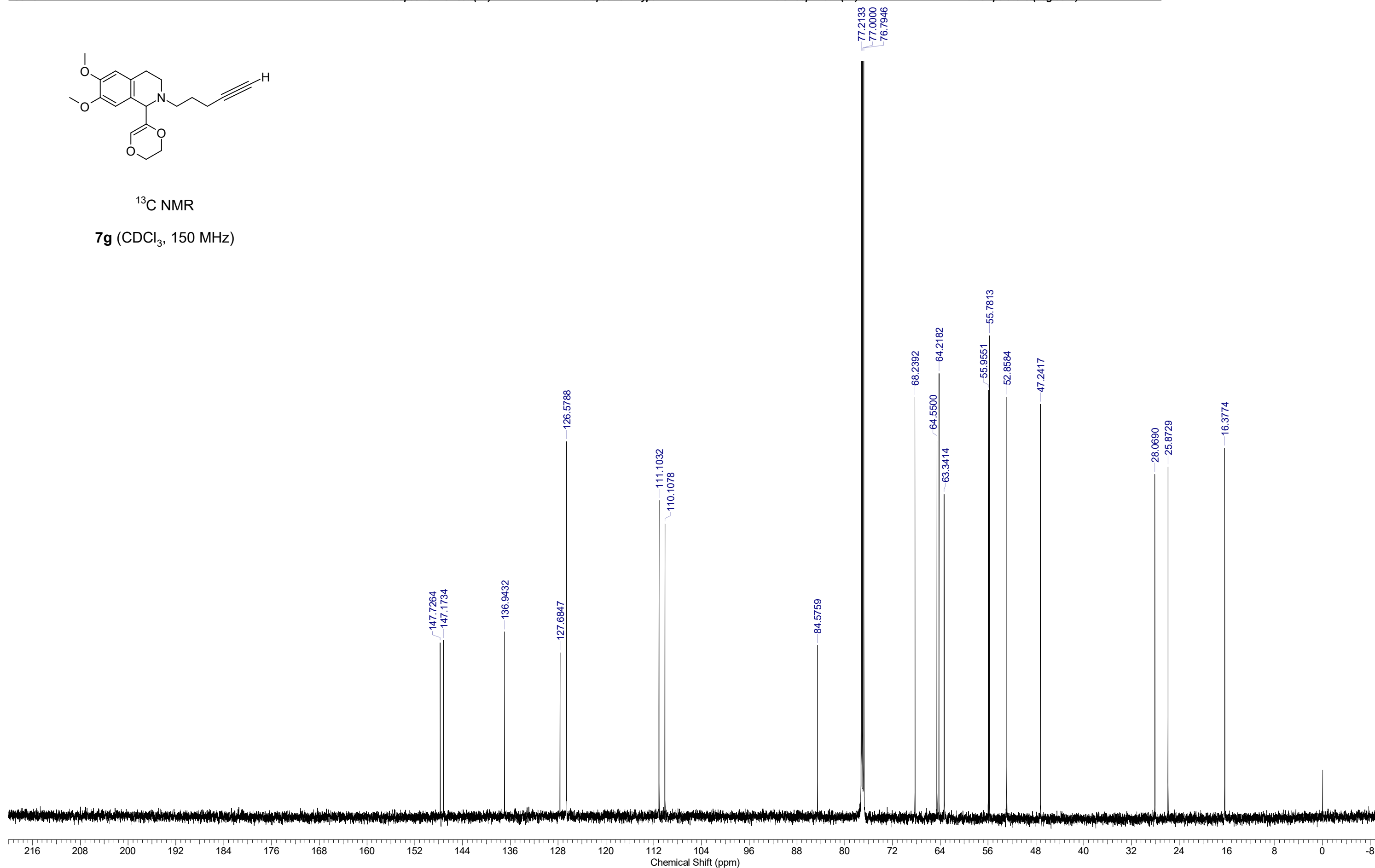


Acquisition Time (sec)	0.8389	Comment	13C	Date	20 Jan 2020 17:12:48	Date Stamp	20 Jan 2020 17:12:48
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01045¥okumura01045 fr18-20 13C¥10¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15084.3340	Points Count	32768	Receiver Gain	193.26
		Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.146
						Number of Transients	128
						SW(cyclical) (Hz)	39062.50

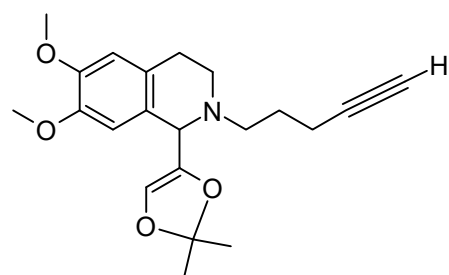


¹³C NMR

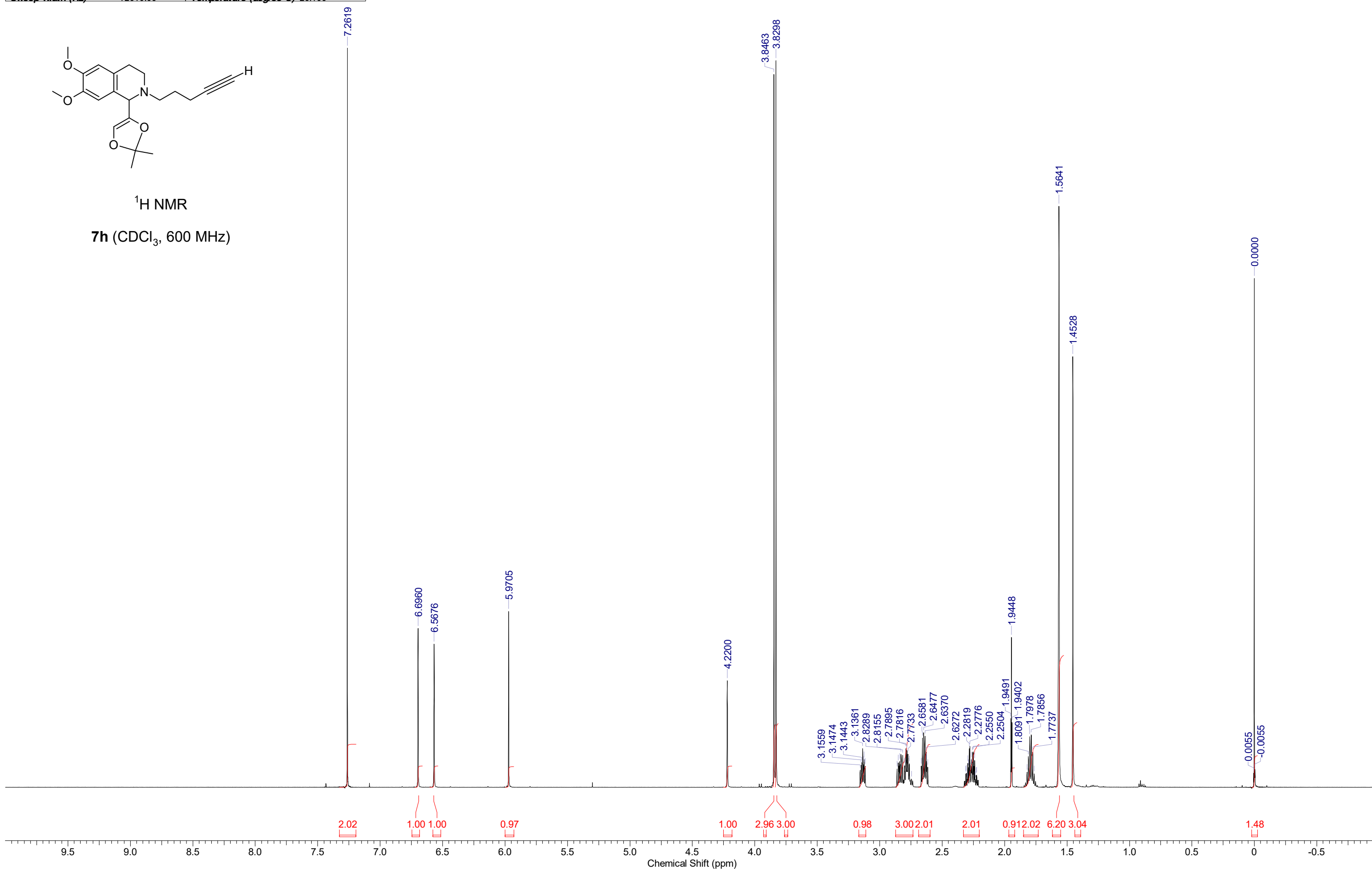
7g (CDCl₃, 150 MHz)



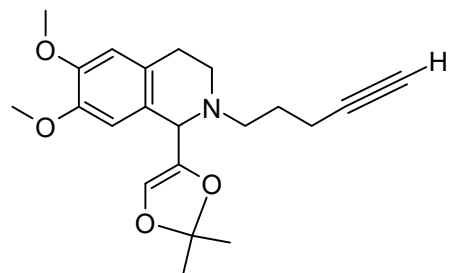
Acquisition Time (sec)	2.7263	Date	08 Apr 2020 09:32:00	Date Stamp	08 Apr 2020 09:32:00	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07053ue¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	¹ H	Number of Transients	8	Origin	spect
Pulse Sequence	zg30	Receiver Gain	30.91	SW(cyclical) (Hz)	12019.23	Original Points Count	32768
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.155	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3692.6450
						Points Count	65536
						Spectrum Type	STANDARD



¹H NMR
7h (CDCl₃, 600 MHz)

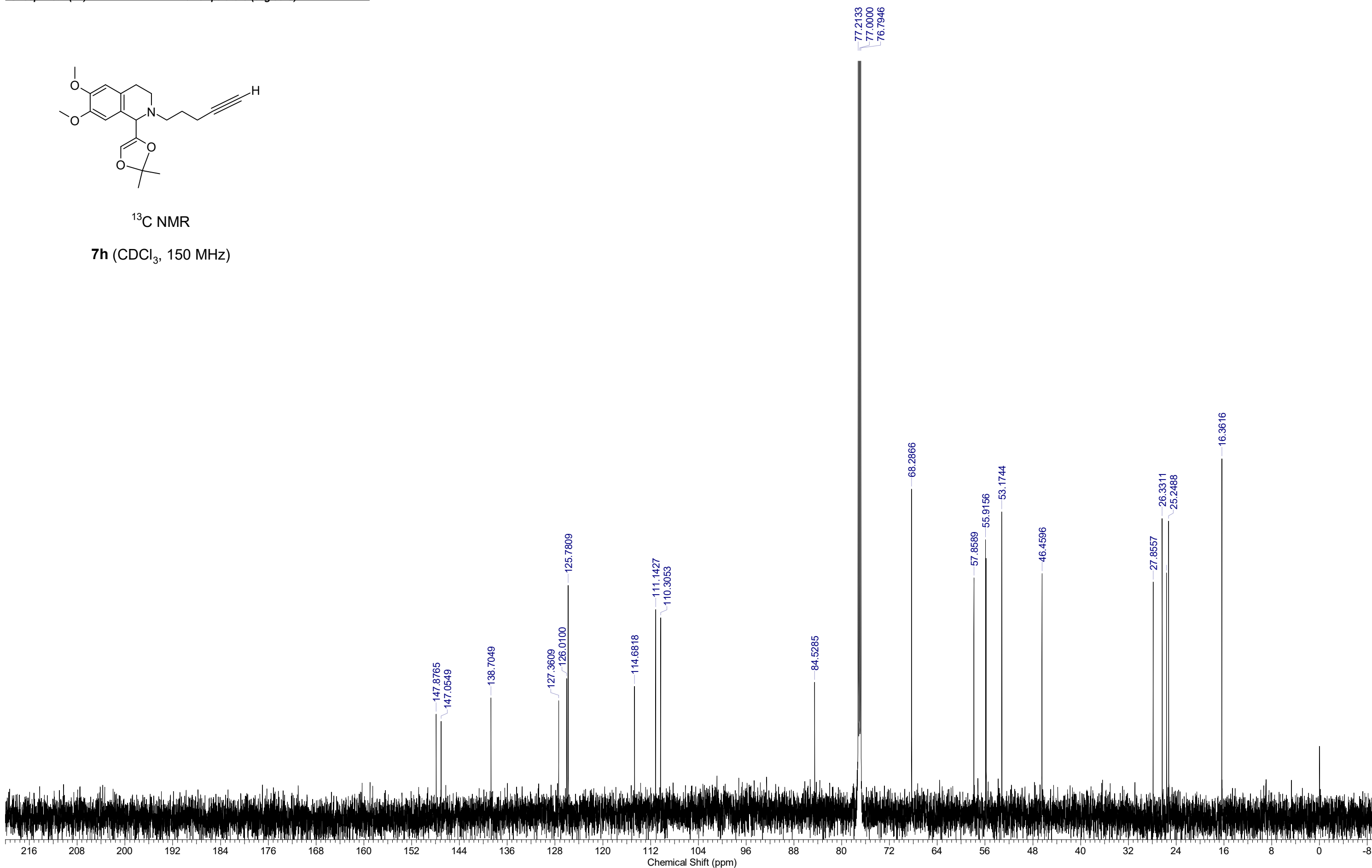


Acquisition Time (sec)	0.8389	Date	08 Apr 2020 09:36:16	Date Stamp	08 Apr 2020 09:36:16	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakai07053ue¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	64	Origin	spect
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Original Points Count	32768
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.155	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15086.7188
						Points Count	32768
						Spectrum Type	STANDARD

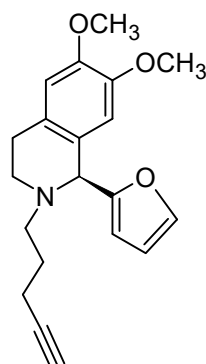


¹³C NMR

7h (CDCl₃, 150 MHz)

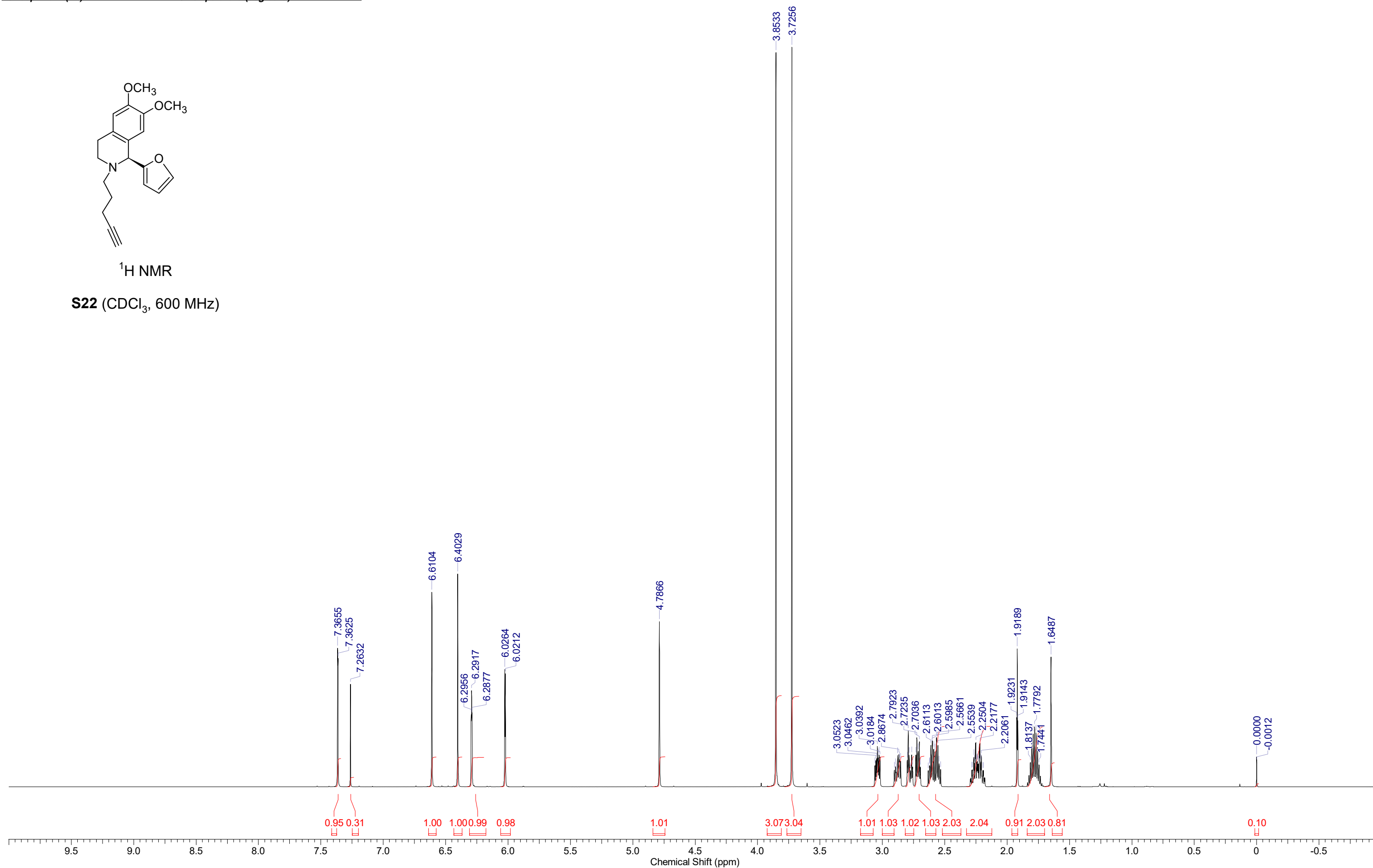


Acquisition Time (sec)	2.7263	Date	15 Jun 2020 19:48:32	Date Stamp	15 Jun 2020 19:48:32	File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07062¥10¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	30.91	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.156			Spectrum Offset (Hz)	3693.3787
						Points Count	65536
						Spectrum Type	STANDARD

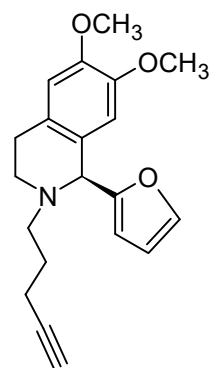


¹H NMR

S22 (CDCl₃, 600 MHz)

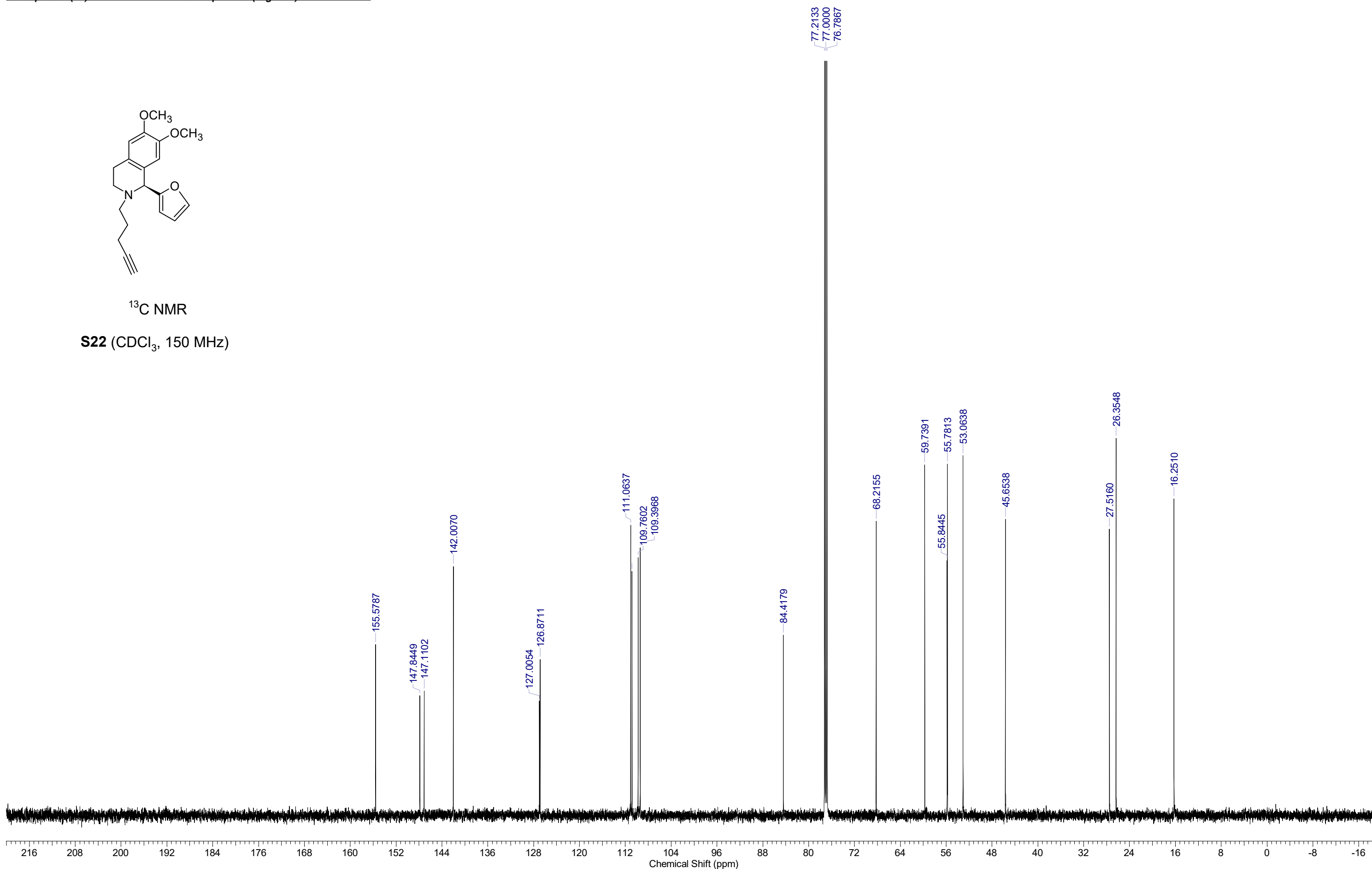


Acquisition Time (sec)	0.8389	Date	15 Jun 2020 19:46:24	Date Stamp	15 Jun 2020 19:46:24	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07062¥11¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	13C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.156			Spectrum Offset (Hz)	15083.1416
						Points Count	32768
						Spectrum Type	STANDARD

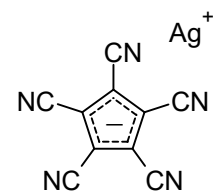


¹³C NMR

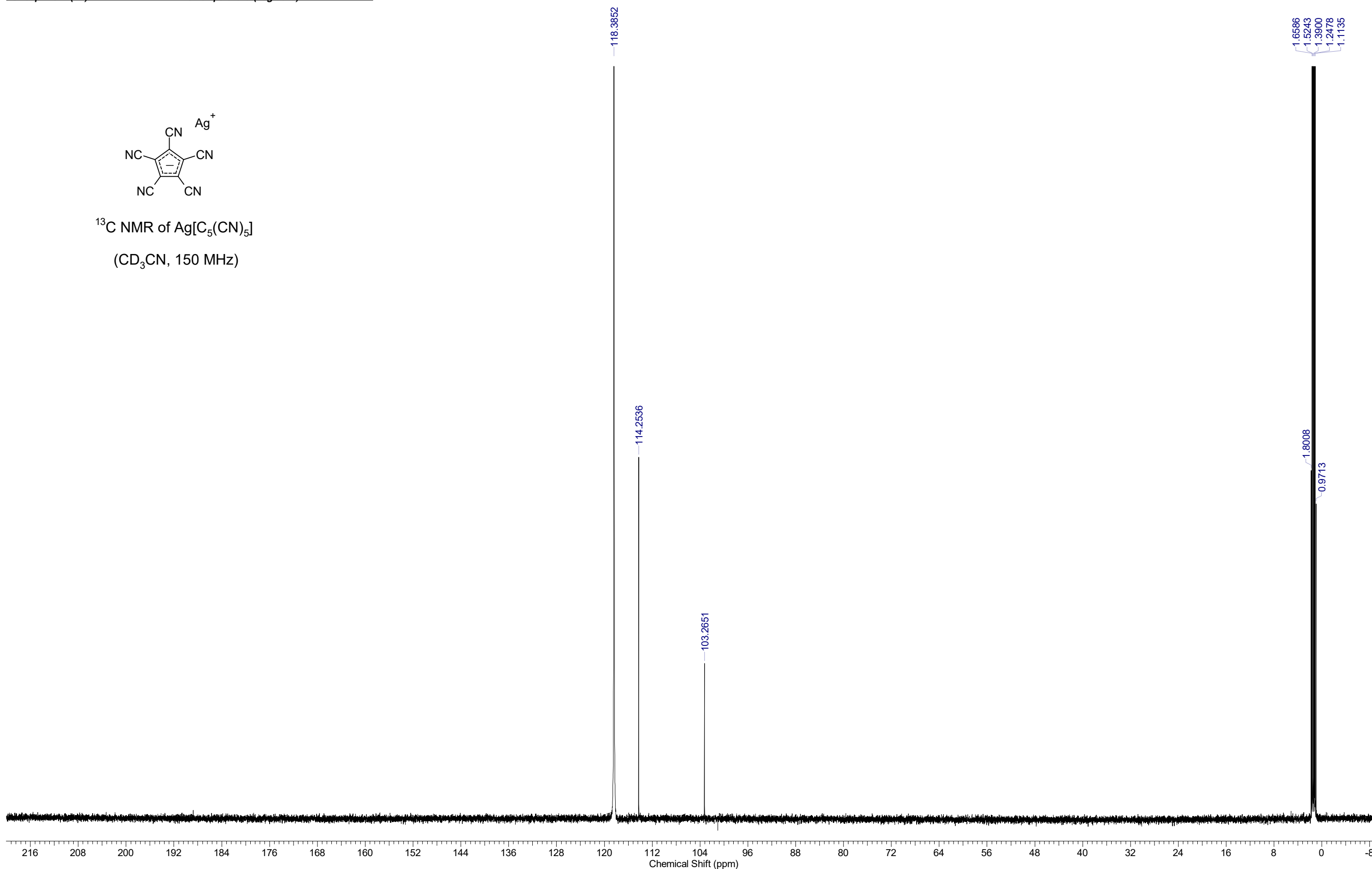
S22 (CDCl₃, 150 MHz)



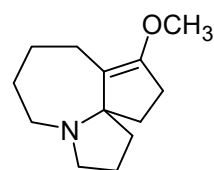
Acquisition Time (sec)	0.8389	Date	21 Nov 2020 13:28:48	Date Stamp	21 Nov 2020 13:28:48	File Name	¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai_AgCpCN¥10¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	ACETONITRILE-d3
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.157	Spectrum Offset (Hz)	15245.0264	Spectrum Type	STANDARD



¹³C NMR of $\text{Ag}[\text{C}_5(\text{CN})_5]$
(CD_3CN , 150 MHz)

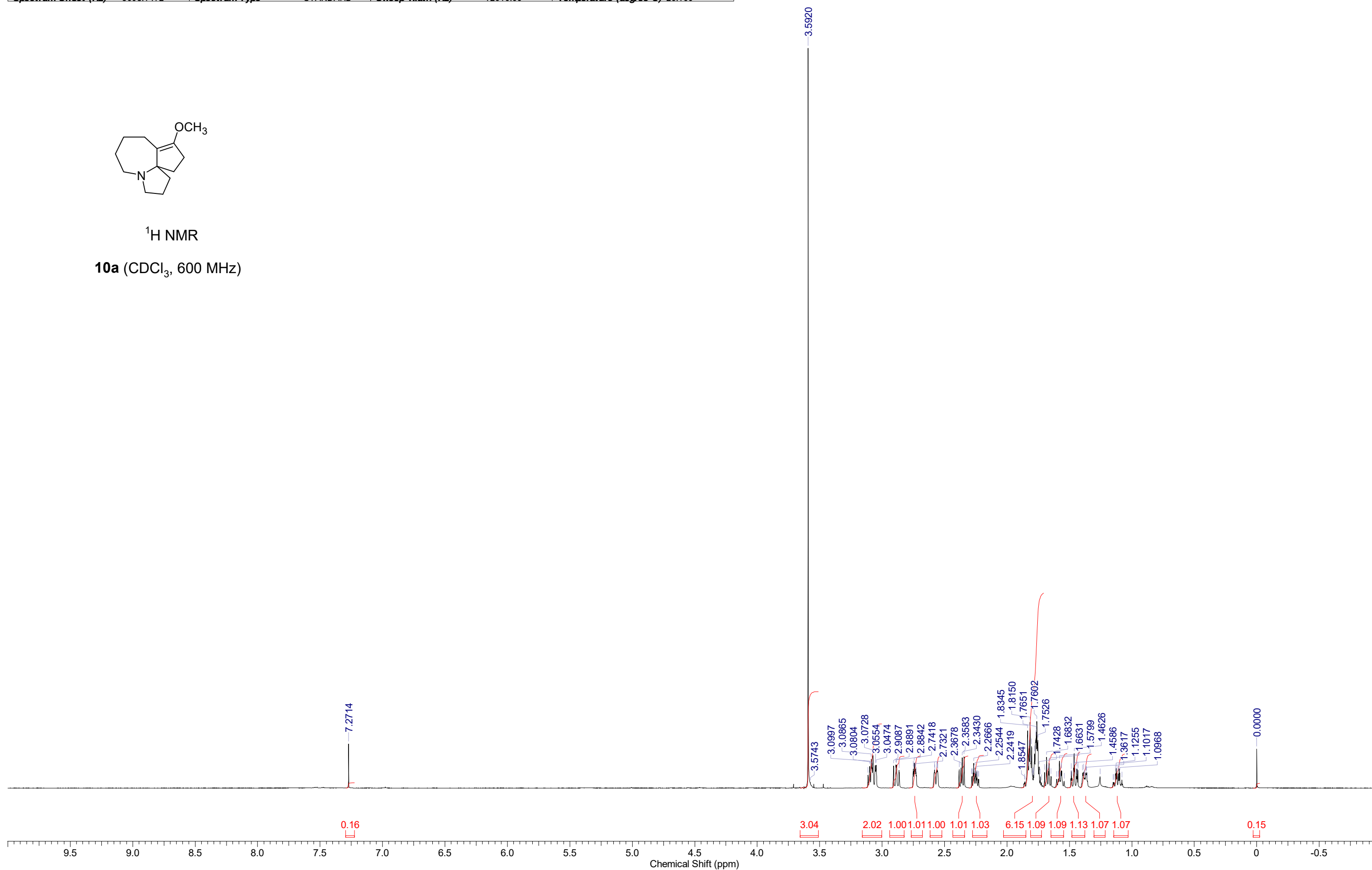


Acquisition Time (sec)	2.7263	Comment	1H	Date	03 Oct 2017 15:24:00	Date Stamp	03 Oct 2017 15:24:00
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02039fr11-24¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3698.1472	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
				Temperature (degree C)	25.159	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

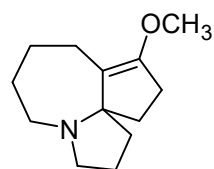


¹H NMR

10a (CDCl₃, 600 MHz)

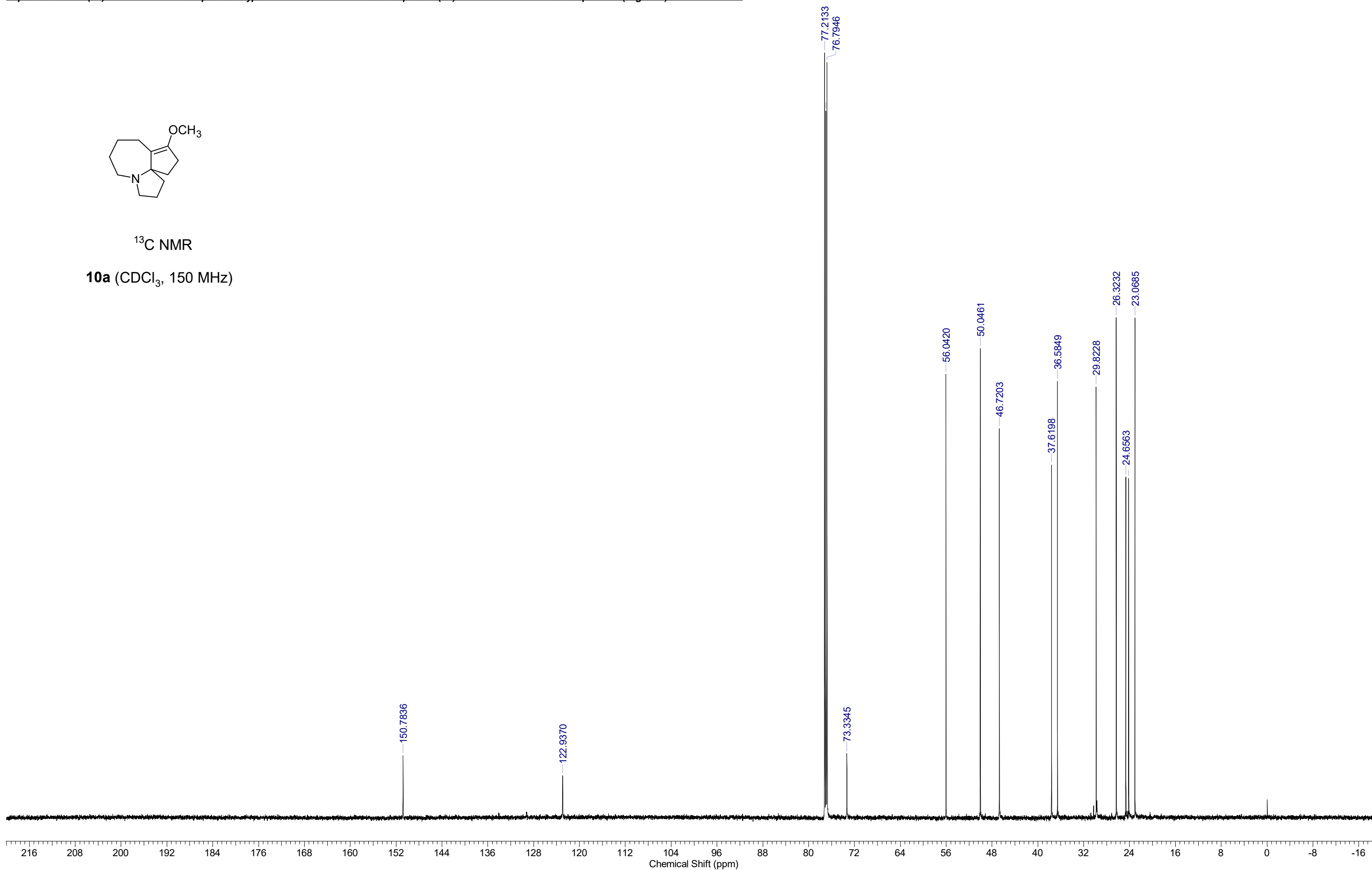


Acquisition Time (sec)	0.8389	Comment	13C	Date	05 Oct 2017 14:17:52	Date Stamp	05 Oct 2017 14:17:52
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02039fr11-24¥20¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmr-su	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.133	SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

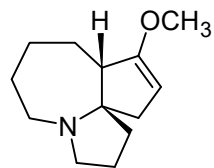


¹³C NMR

10a (CDCl₃, 150 MHz)

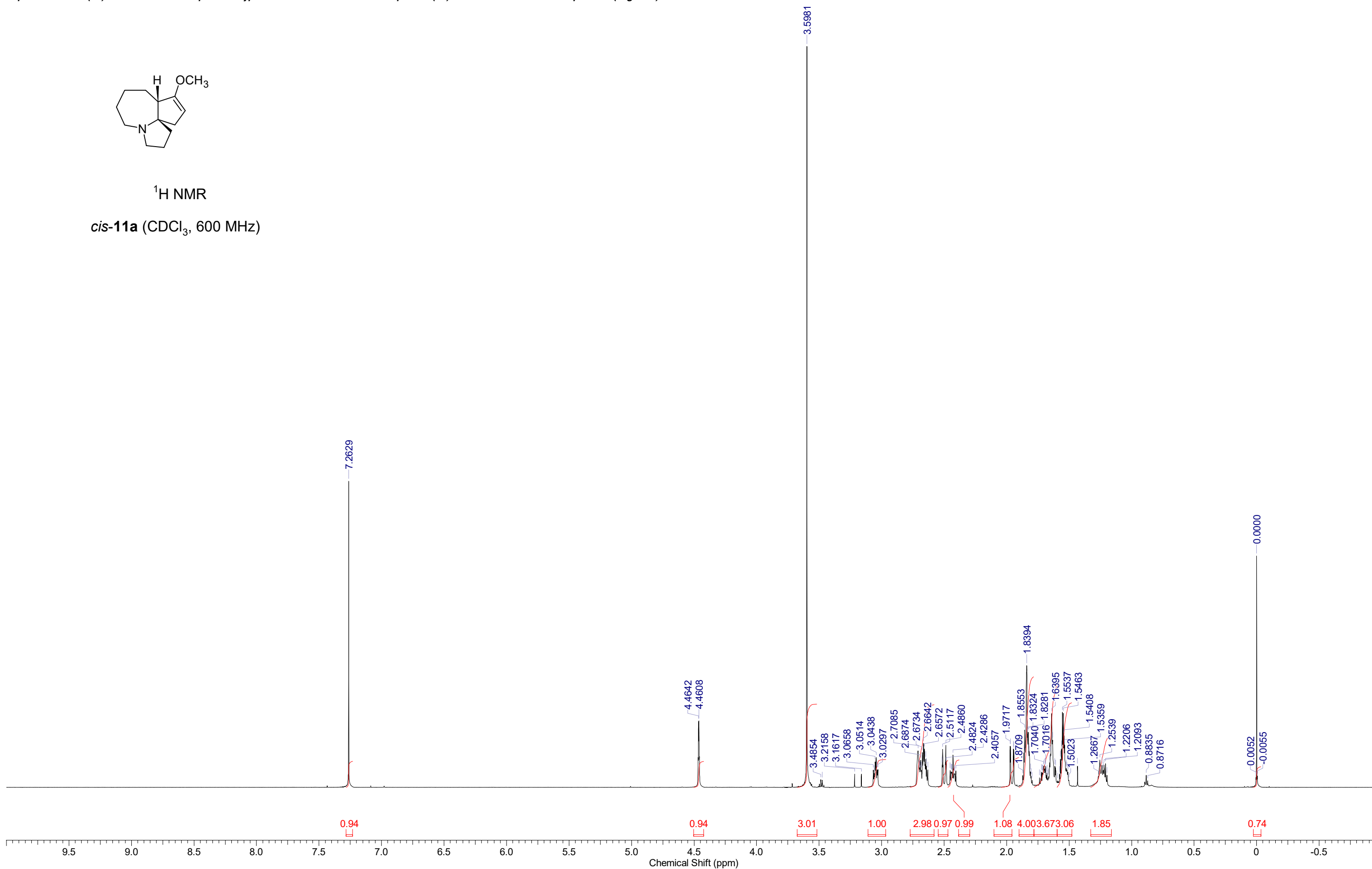


Acquisition Time (sec)	2.7263	Comment	1H	Date	03 Oct 2017 15:21:52	Date Stamp	03 Oct 2017 15:21:52
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02039fr3-8¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Receiver Gain	30.91
Spectrum Offset (Hz)	3692.8286	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	SW(cyclical) (Hz)	12019.23
				Temperature (degree C)	25.151	Origin	spect
						Solvent	CHLOROFORM-d

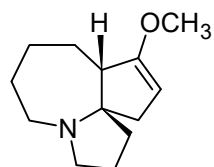


¹H NMR

cis-11a (CDCl₃, 600 MHz)

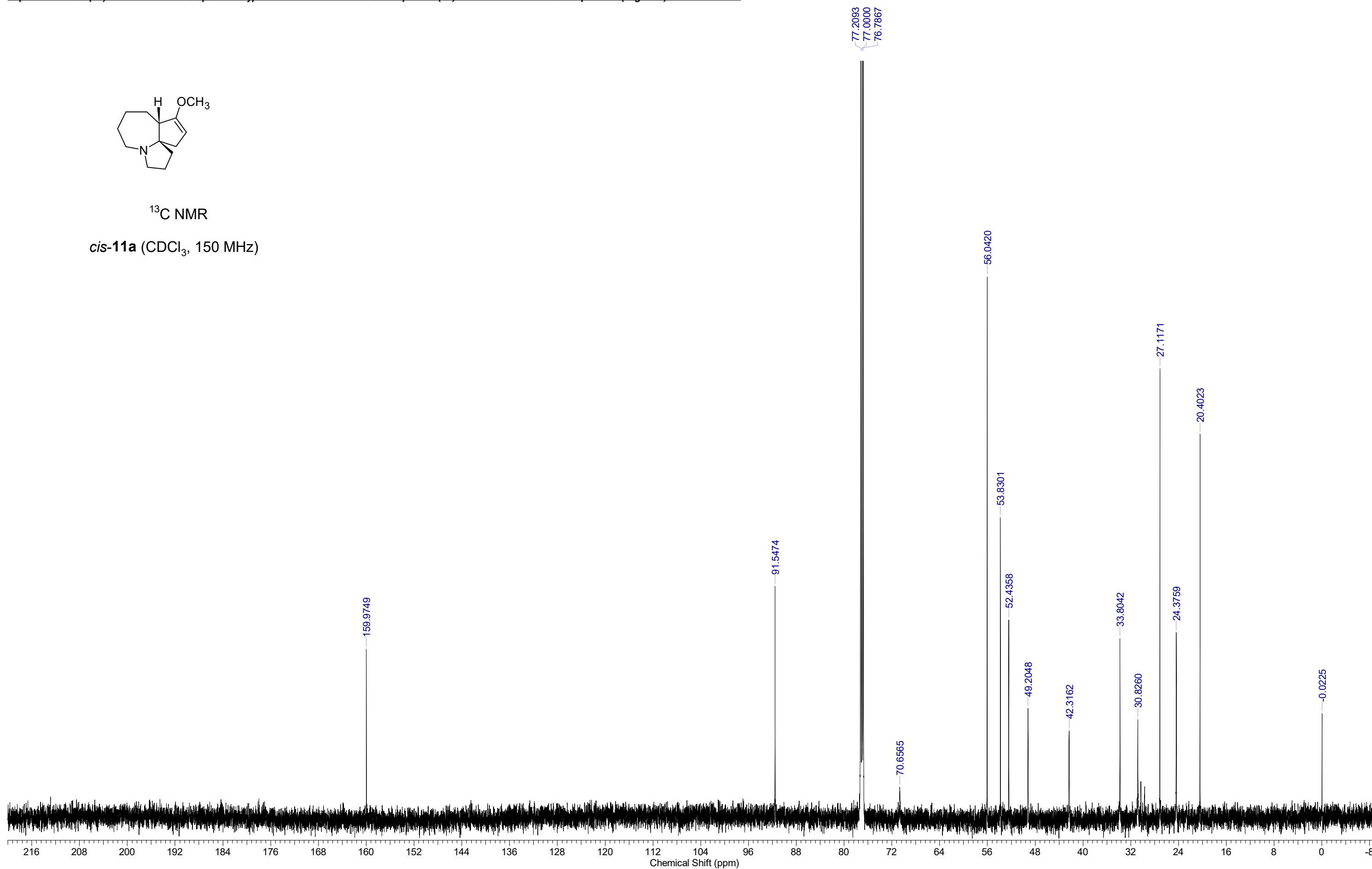


Acquisition Time (sec)	0.8389	Comment	13C	Date	05 Oct 2017 14:32:48	Date Stamp	05 Oct 2017 14:32:48
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2017¥野田¥noda02040fr1-6¥20¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	65536	Receiver Gain	193.26
Spectrum Offset (Hz)	15084.6318	Spectrum Type	STANDARD	Pulse Sequence	zgpg30	Number of Transients	128
				Sweep Width (Hz)	39061.90	SW(cyclical) (Hz)	39062.50
				Temperature (degree C)	25.156	Origin	spect
						Solvent	CHLOROFORM-d

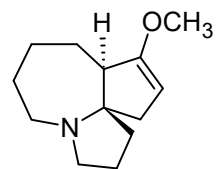


¹³C NMR

cis-11a (CDCl₃, 150 MHz)

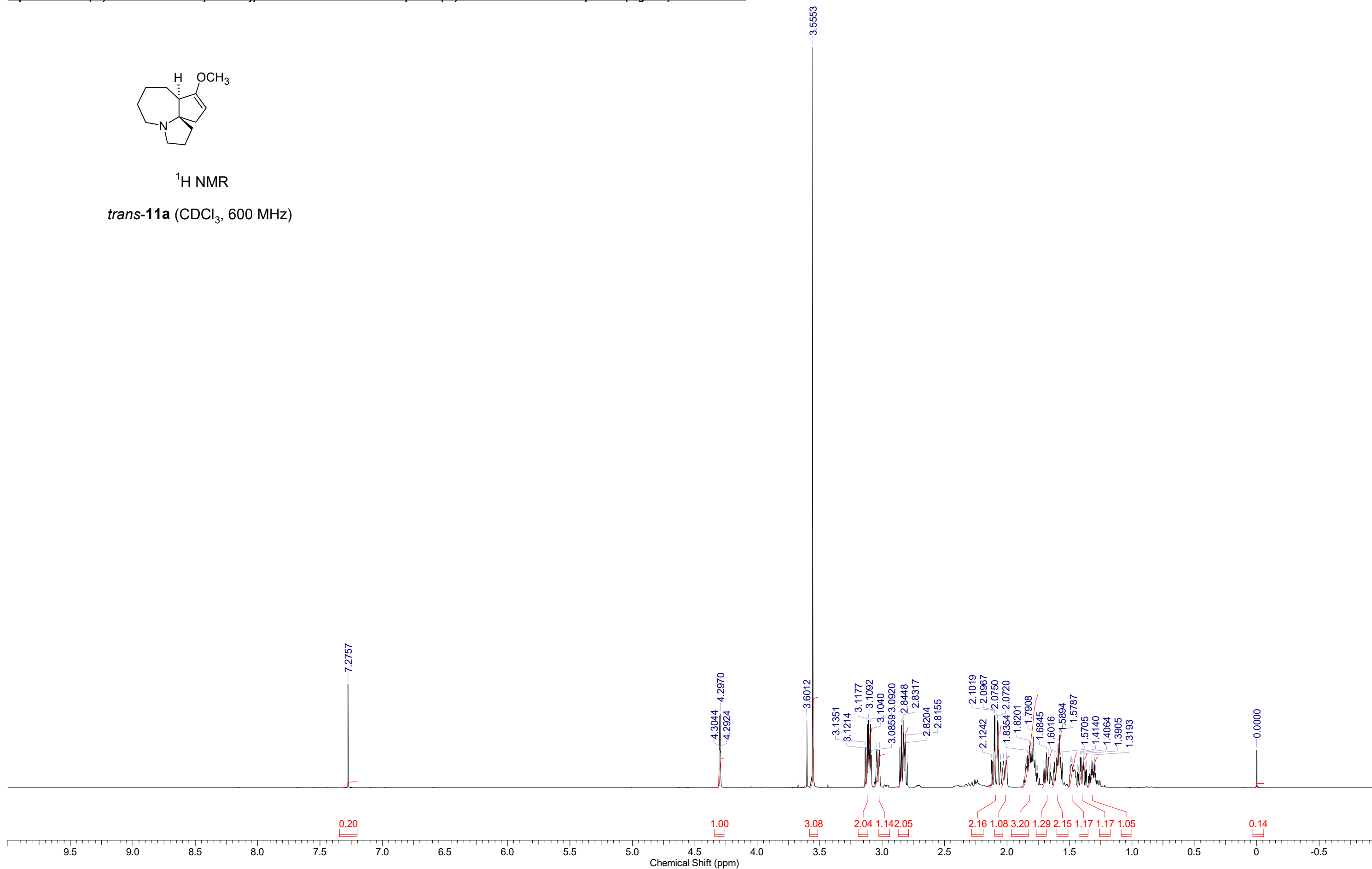


Acquisition Time (sec)	2.7263	Comment	proton	Date	09 Dec 2019 18:06:08	Date Stamp	09 Dec 2019 18:06:08
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamiya kankatai shita trans¥11¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3700.5313	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.144	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

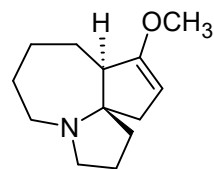
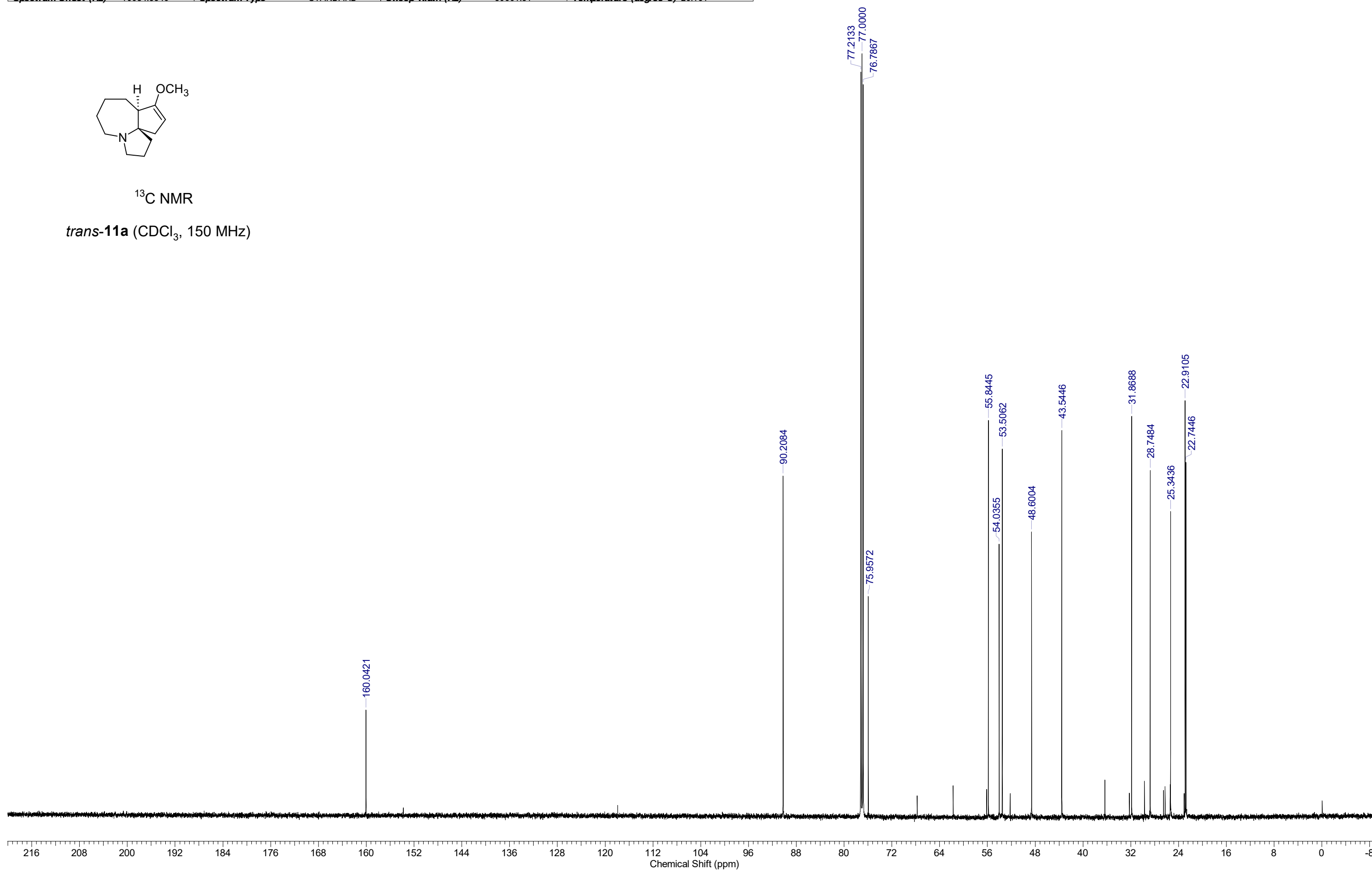


¹H NMR

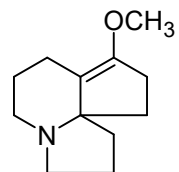
trans-11a (CDCl₃, 600 MHz)



Acquisition Time (sec)	0.8389	Comment	carbon	Date	09 Dec 2019 18:23:12	Date Stamp	09 Dec 2019 18:23:12						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2018¥神谷¥kamiya kankatai shita trans¥12¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	64	Origin	spect		
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.151						

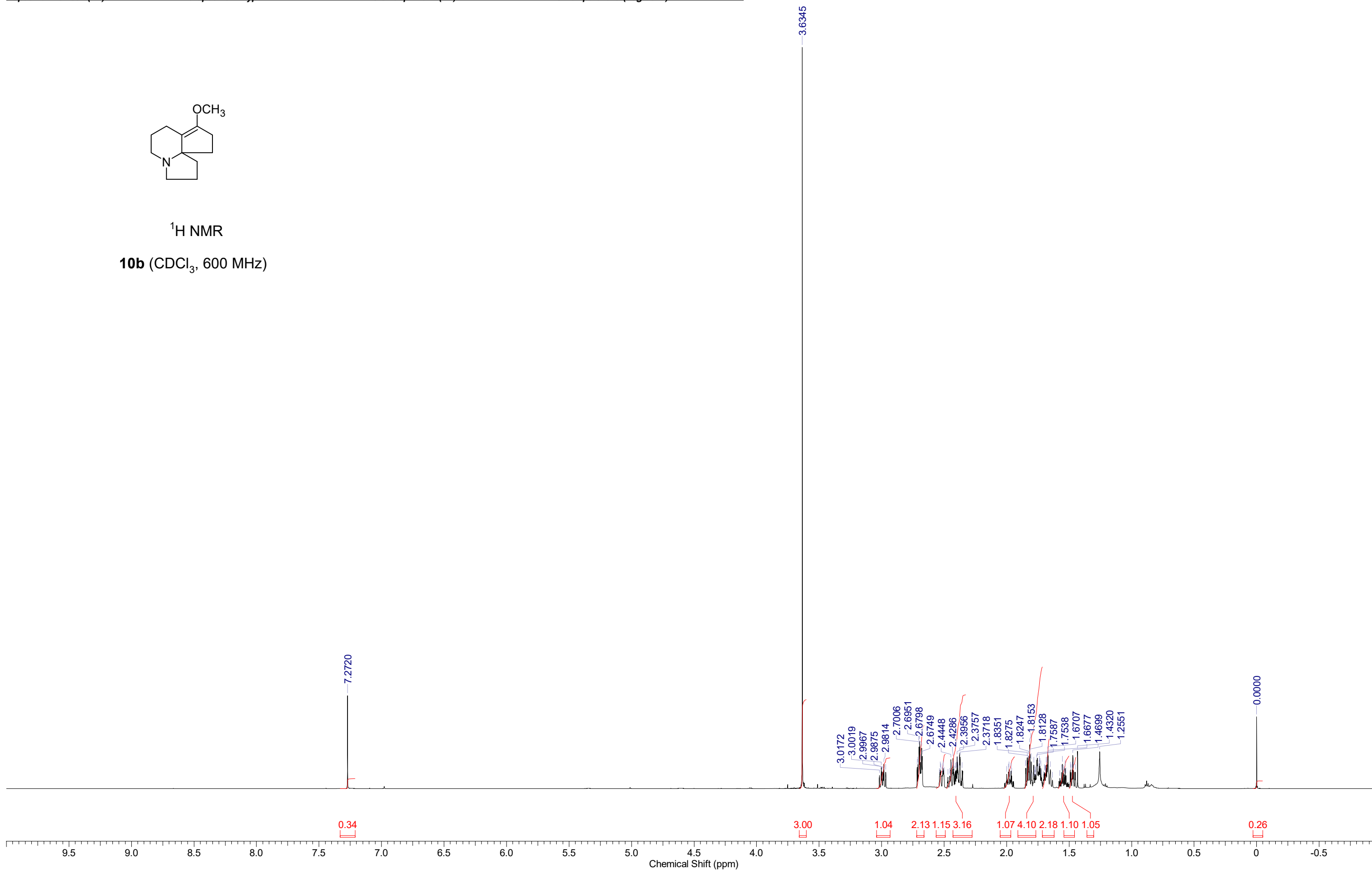
¹³C NMRtrans-11a (CDCl₃, 150 MHz)

Acquisition Time (sec)	2.7263	Comment	1H	Date	07 Jun 2019 14:49:52	Date Stamp	07 Jun 2019 14:49:52
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01106saishita¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nrmrsu	Points Count	65536	Receiver Gain	25.50
Spectrum Offset (Hz)	3698.5139	Spectrum Type	STANDARD	Pulse Sequence	zg30	SW(cyclical) (Hz)	12019.23
				Sweep Width (Hz)	12019.05	Temperature (degree C)	25.143
						Origin	spect
						Solvent	CHLOROFORM-d

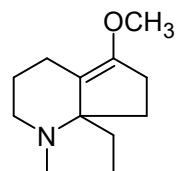


¹H NMR

10b (CDCl₃, 600 MHz)

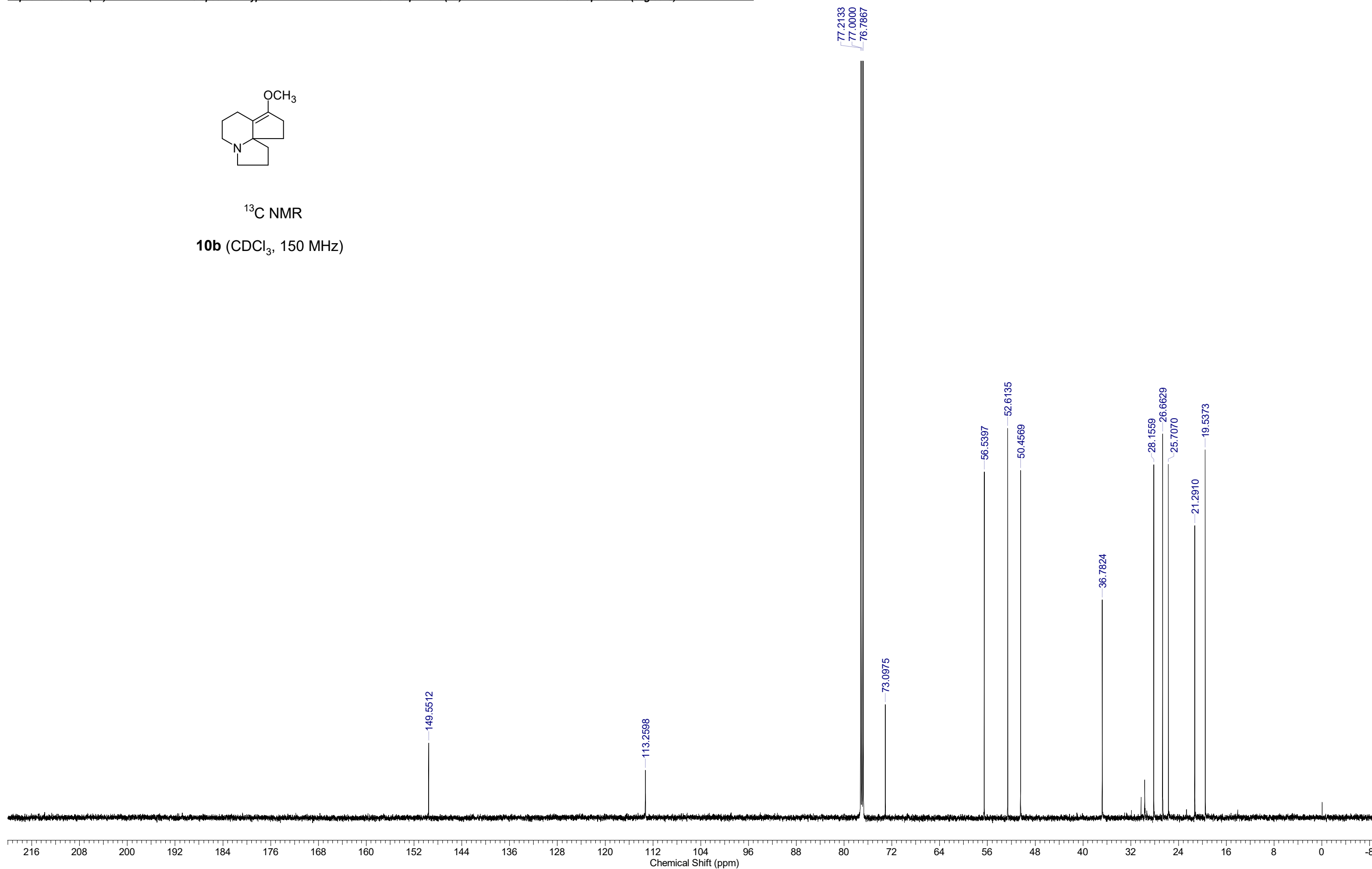


Acquisition Time (sec)	0.8389	Comment	13C	Date	07 Jun 2019 14:58:24	Date Stamp	07 Jun 2019 14:58:24						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01106saishita¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	128	Origin	spect		
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.165						

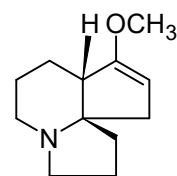


¹³C NMR

10b (CDCl₃, 150 MHz)

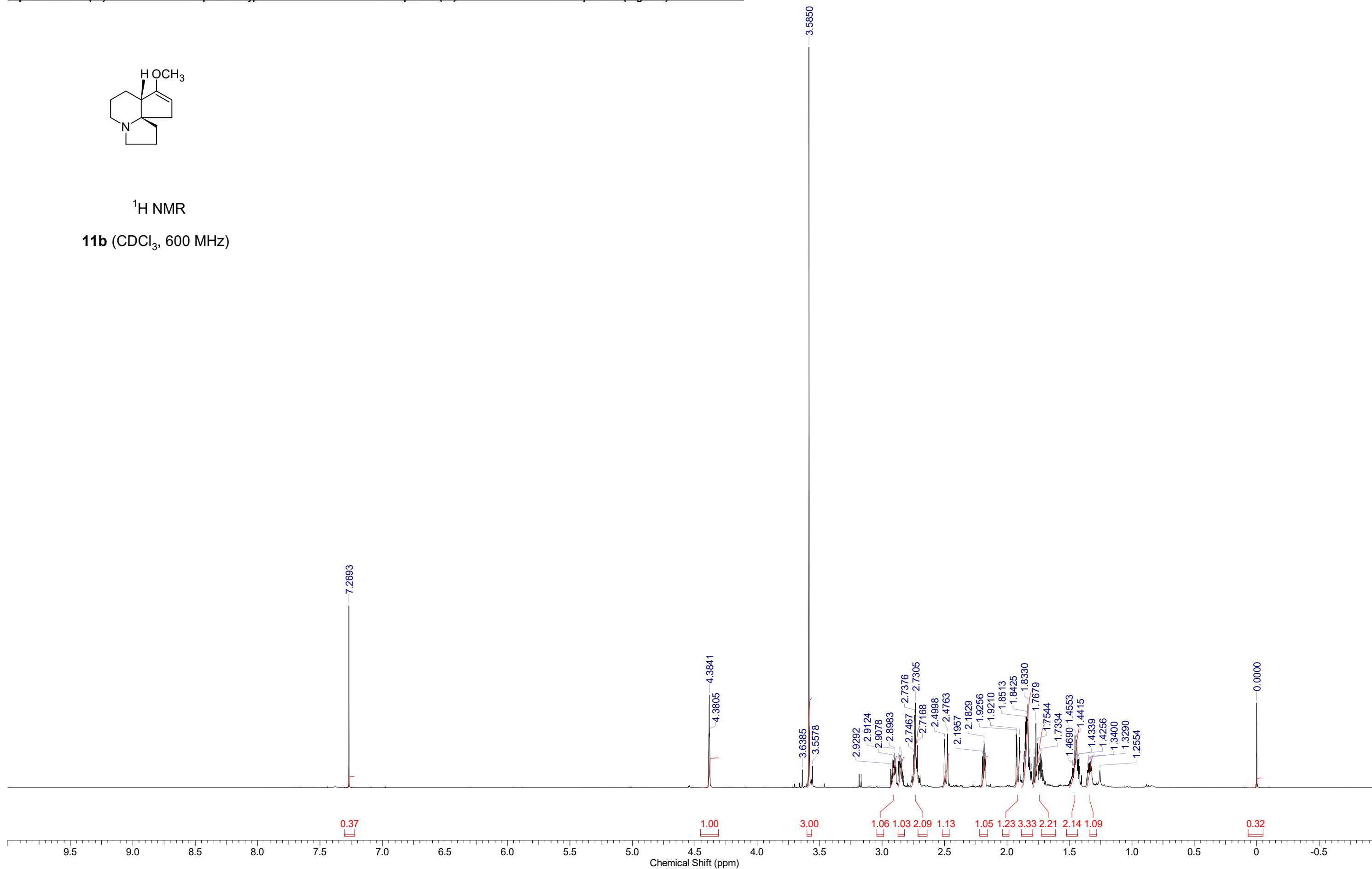


Acquisition Time (sec)	2.7263	Comment	proton	Date	07 Dec 2019 11:27:12	Date Stamp	07 Dec 2019 11:27:12
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01106.7ue¥3¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmrsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3696.8633	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.147	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

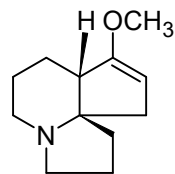


¹H NMR

11b (CDCl₃, 600 MHz)

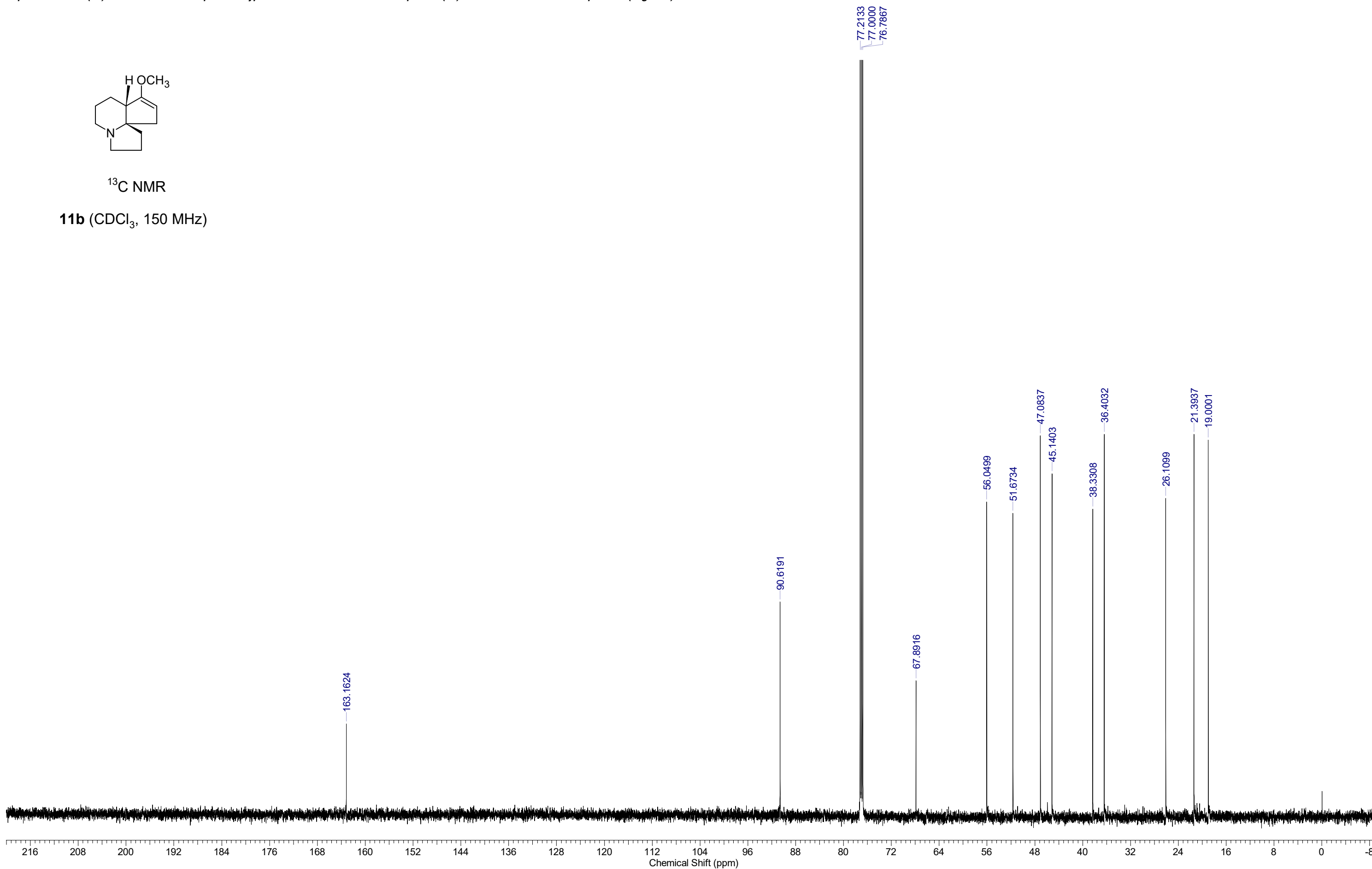


Acquisition Time (sec)	0.8389	Comment	carbon	Date	07 Dec 2019 11:31:28	Date Stamp	07 Dec 2019 11:31:28
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01106.7ue¥4¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.150	Number of Transients	32
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

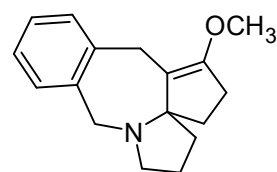


¹³C NMR

11b (CDCl₃, 150 MHz)

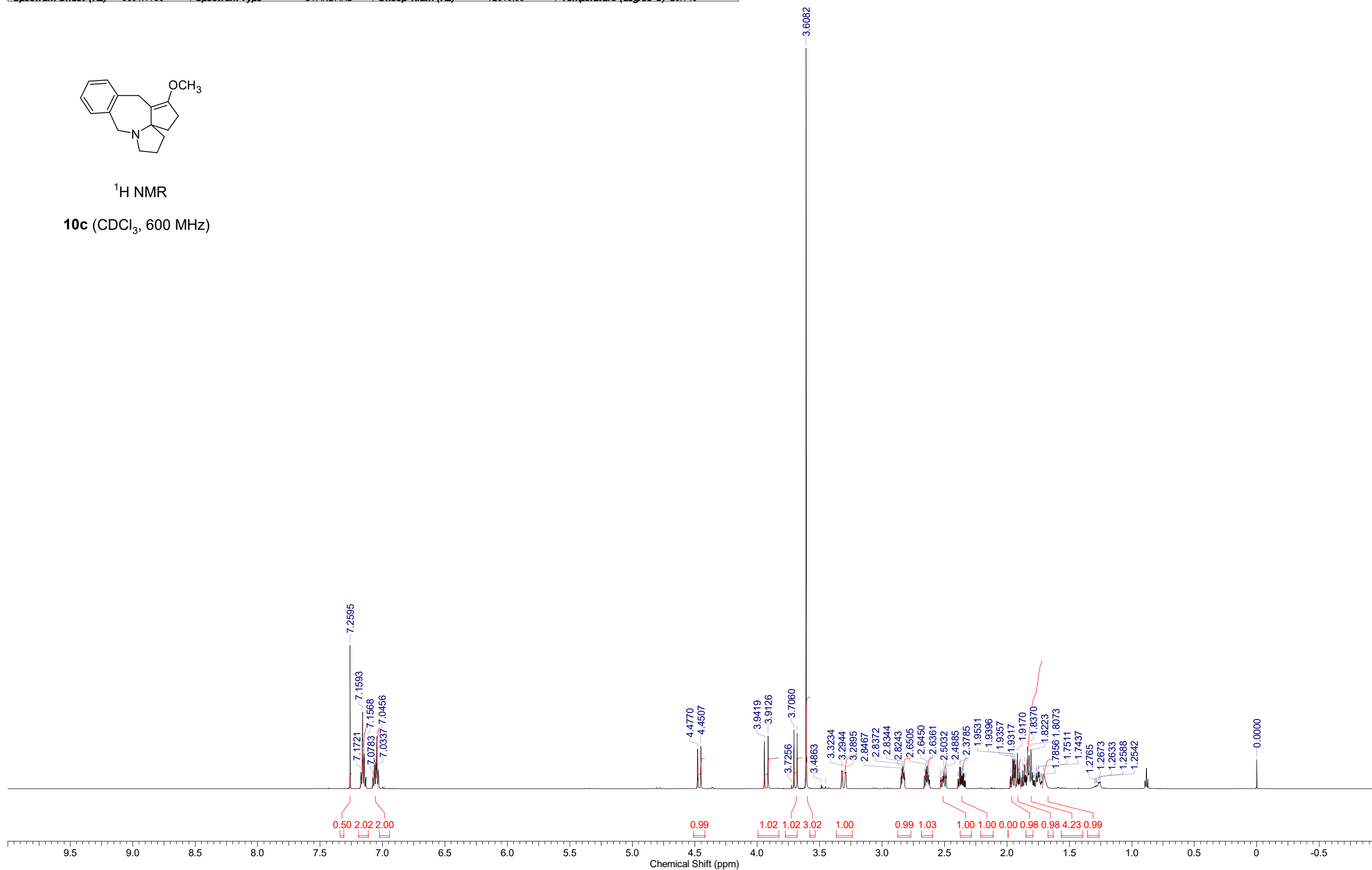


Acquisition Time (sec)	2.7263	Date	30 Oct 2019 17:36:16	Date Stamp	30 Oct 2019 17:36:16								
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06101shita_shita¥10¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H							
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	22.72	Number of Transients	8	Origin	spect
Spectrum Offset (Hz)	3691.1780	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.146	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d		

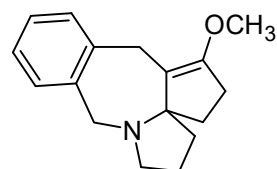


¹H NMR

10c (CDCl₃, 600 MHz)

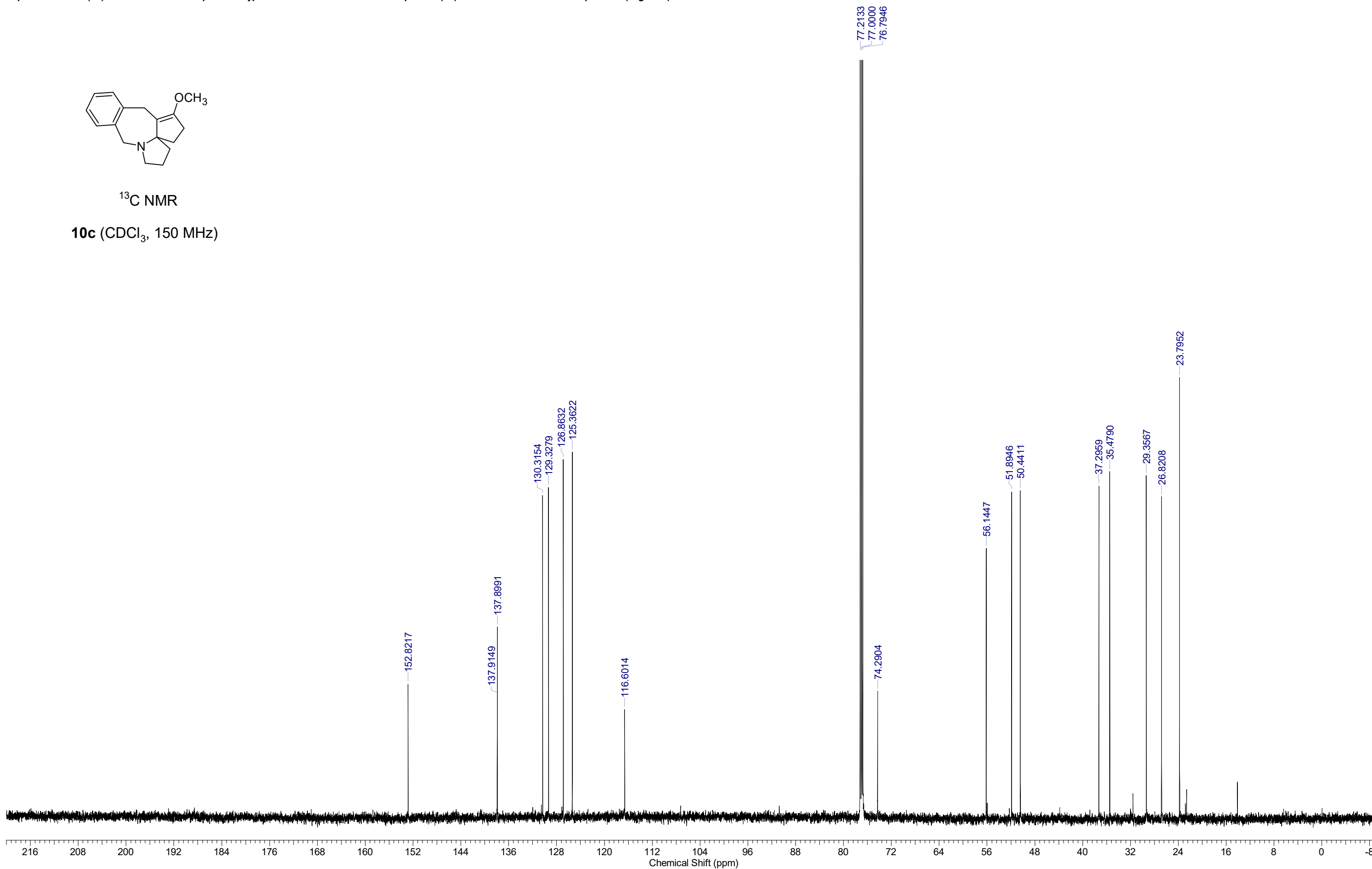


Acquisition Time (sec)	0.8389	Date	30 Oct 2019 17:44:48	Date Stamp	30 Oct 2019 17:44:48	
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06101shita_shita¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	
Original Points Count	32768	Owner	nmsu	Points Count	32768	
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Pulse Sequence	zgpg30	
			Sweep Width (Hz)	39061.31	Temperature (degree C)	25.147
				Receiver Gain	193.26	
				Number of Transients	128	
				SW(cyclical) (Hz)	39062.50	
				Origin	spect	
				Solvent	CHLOROFORM-d	

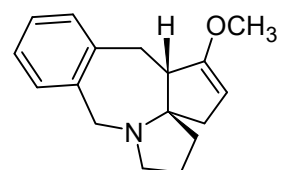


¹³C NMR

10c (CDCl₃, 150 MHz)

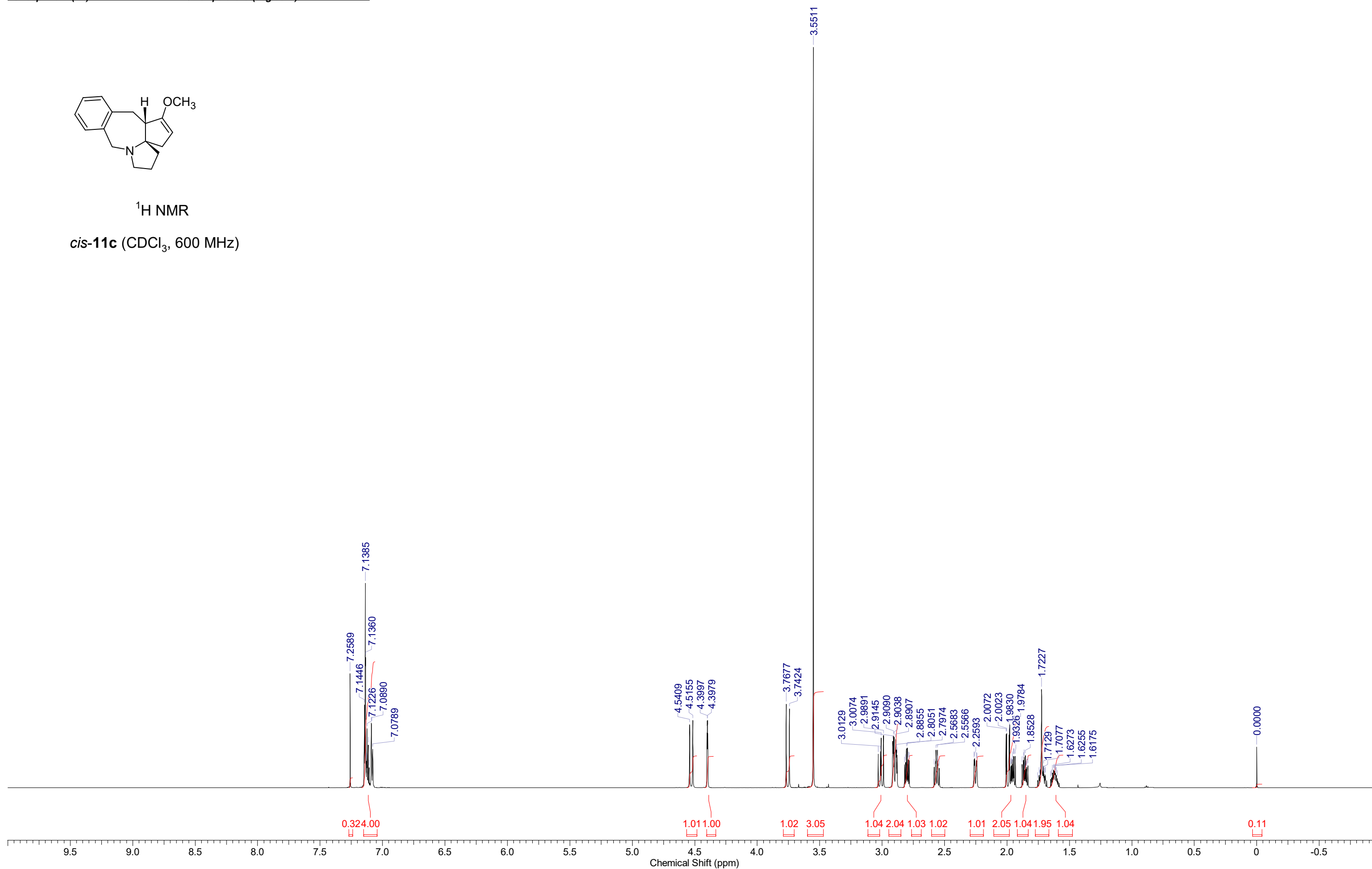


Acquisition Time (sec)	2.7263	Date	26 Oct 2019 21:37:20	Date Stamp	26 Oct 2019 21:37:20	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06101naka¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	25.50	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.147			Spectrum Offset (Hz)	3690.8110
						Points Count	65536
						Spectrum Type	STANDARD

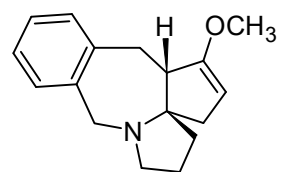


¹H NMR

cis-11c (CDCl₃, 600 MHz)

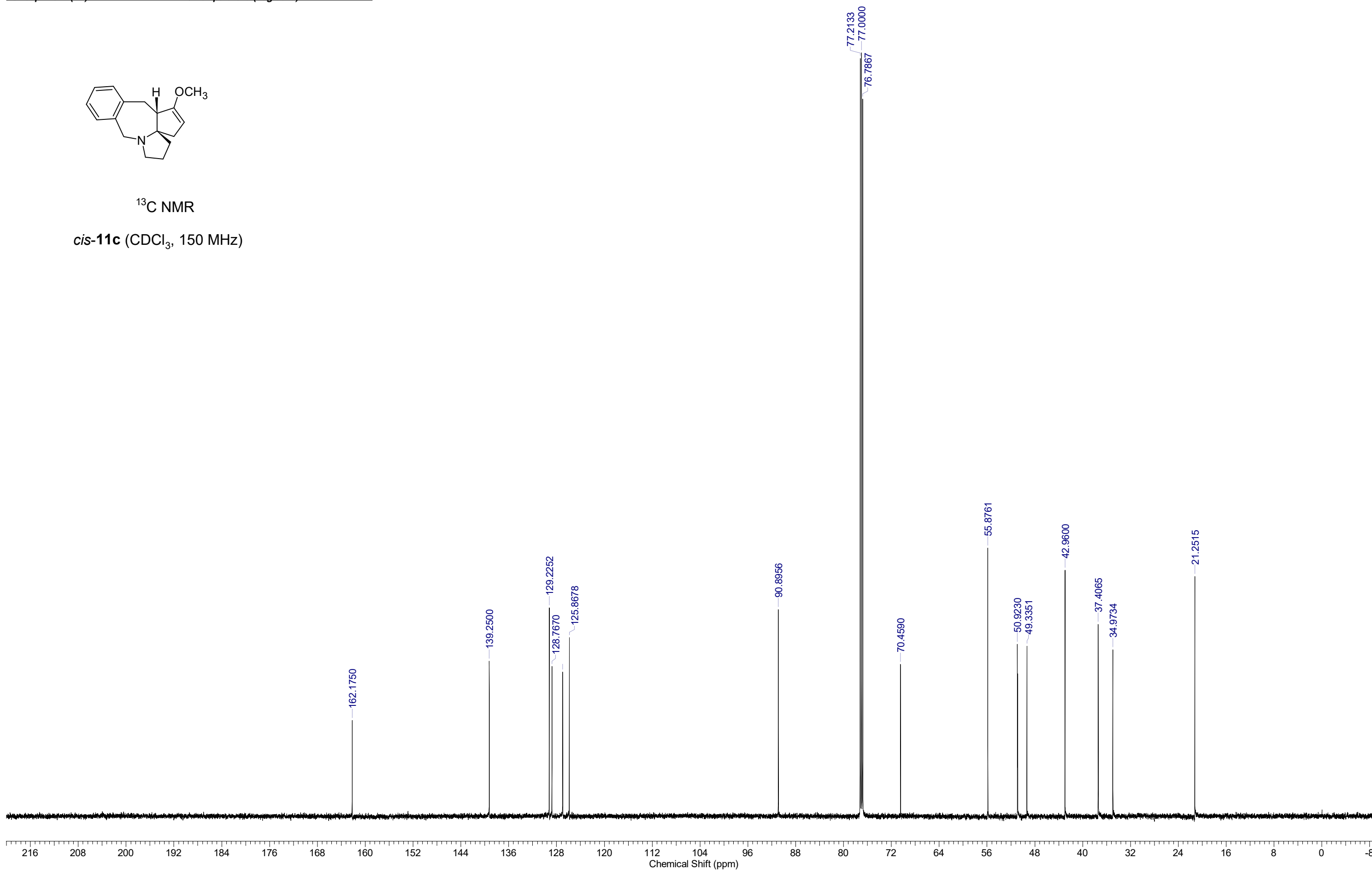


Acquisition Time (sec)	0.8389	Date	26 Oct 2019 21:41:36	Date Stamp	26 Oct 2019 21:41:36	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06101naka¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15084.3340
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.149	Number of Transients	64	Points Count	32768
				SV(cyclical) (Hz)	39062.50	Spectrum Type	STANDARD

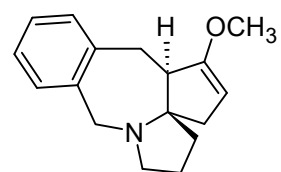


¹³C NMR

cis-11c (CDCl₃, 150 MHz)



Acquisition Time (sec)	2.7263	Date	30 Oct 2019 17:51:12	Date Stamp	30 Oct 2019 17:51:12
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06101shita_naka¥10¥pdata¥1¥1r			Frequency (MHz)	600.13
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Spectrum Offset (Hz)	3691.9116	Spectrum Type	STANDARD	Receiver Gain	30.91
		Points Count	65536	Number of Transients	8
		Sweep Width (Hz)	12019.05	SW(cyclical) (Hz)	12019.23
		Temperature (degree C)	25.147	Origin	spect
				Solvent	CHLOROFORM-d

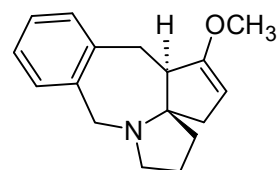


¹H NMR

trans-11c (CDCl₃, 600 MHz)

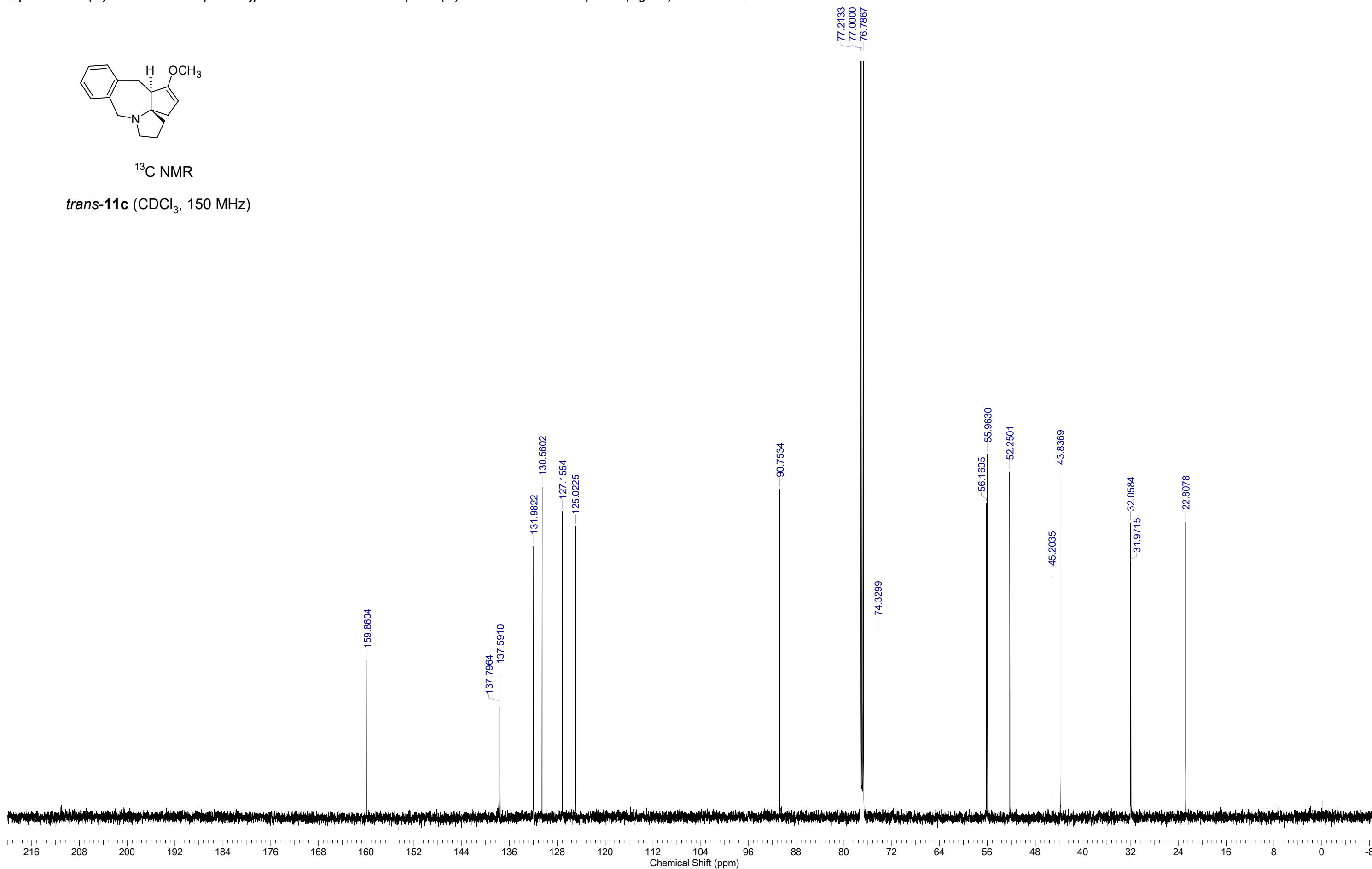


Acquisition Time (sec)	0.8389	Date	30 Oct 2019 17:59:44	Date Stamp	30 Oct 2019 17:59:44
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06101shita_naka¥11¥pdata¥1¥1r			Frequency (MHz)	150.90
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Receiver Gain	193.26
		Points Count	32768	Number of Transients	128
		Sweep Width (Hz)	39061.31	Temperature (degree C)	25.156
				Origin	spect
				Solvent	CHLOROFORM-d

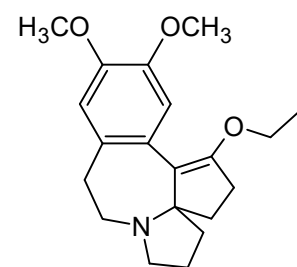


¹³C NMR

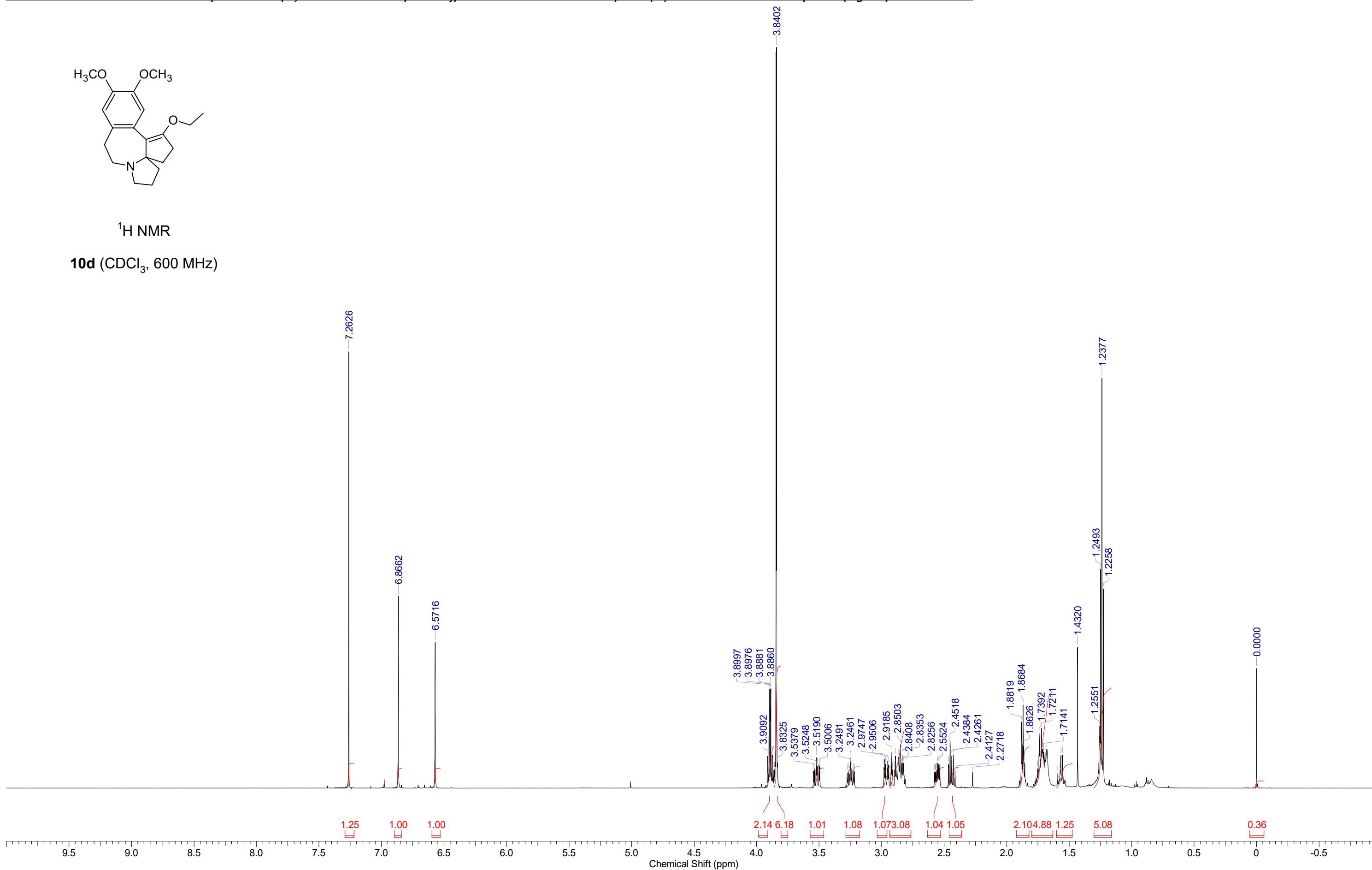
trans-11c (CDCl₃, 150 MHz)



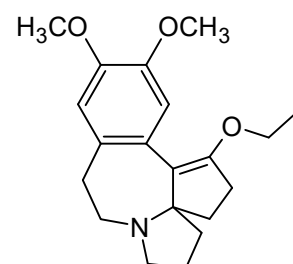
Acquisition Time (sec)	2.7263	Comment	1H	Date	06 Jan 2020 16:00:16	Date Stamp	06 Jan 2020 16:00:16
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01041¥4回目¥力ラ¥Yokumura01041.4.3fr23-30¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3693.0120	Spectrum Type	STANDARD	Receiver Gain	30.91
				Sweep Width (Hz)	12019.05	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Temperature (degree C)	25.147



¹H NMR
10d (CDCl₃, 600 MHz)

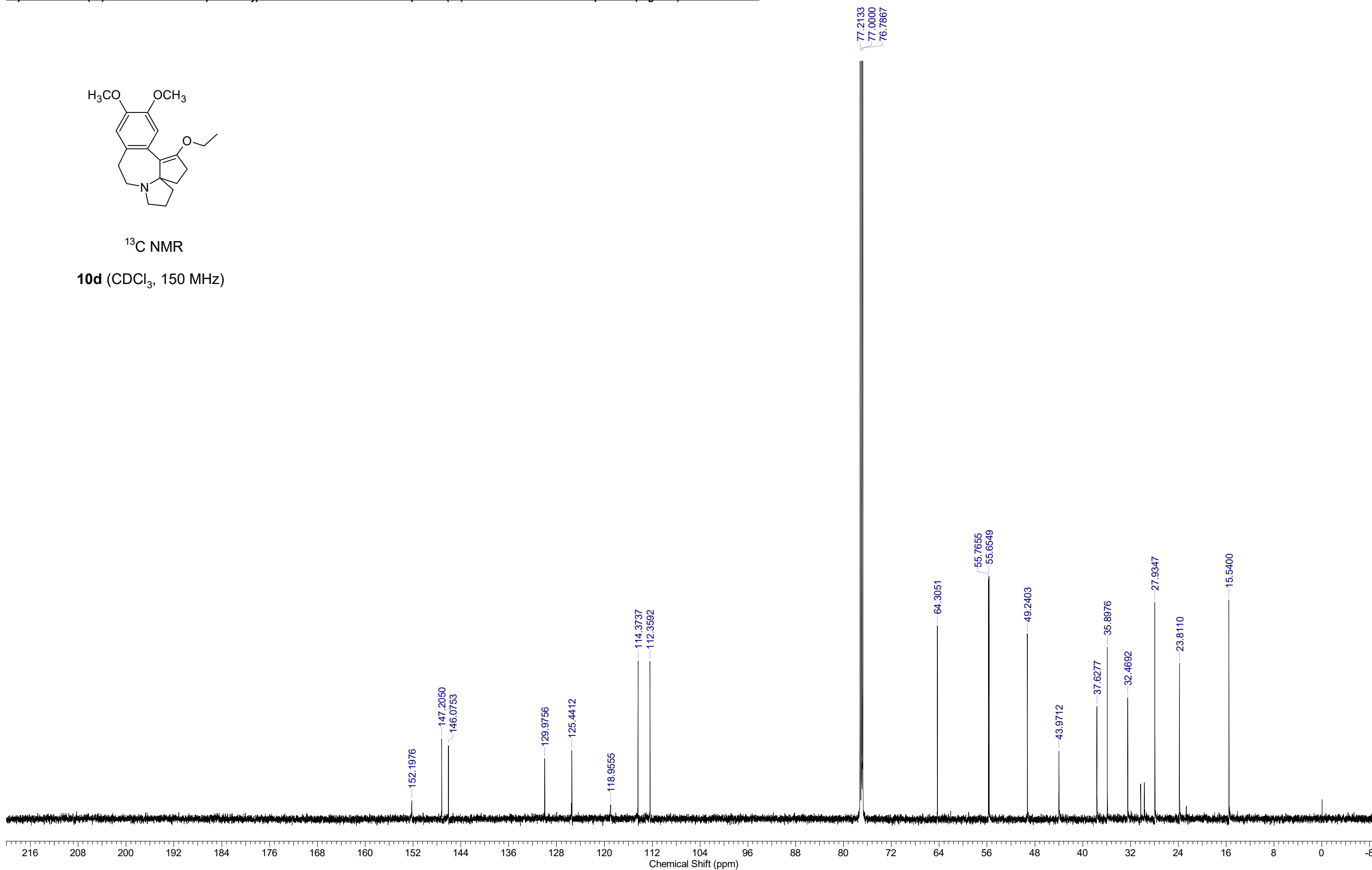


Acquisition Time (sec)	0.8389	Date	21 Nov 2020 15:47:28	Date Stamp	21 Nov 2020 15:47:28
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥okumura01041shita¥2¥pdata¥1¥1r			Frequency (MHz)	150.90
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31
				Temperature (degree C)	25.142
				Nucleus	13C
				Receiver Gain	193.26
				Number of Transients	128
				SM(cyclical) (Hz)	39062.50
				Origin	spect
				Solvent	CHLOROFORM-d

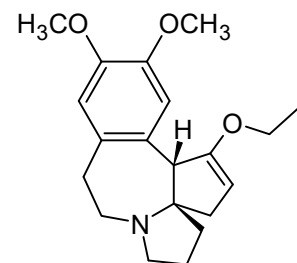


¹³C NMR

10d (CDCl₃, 150 MHz)

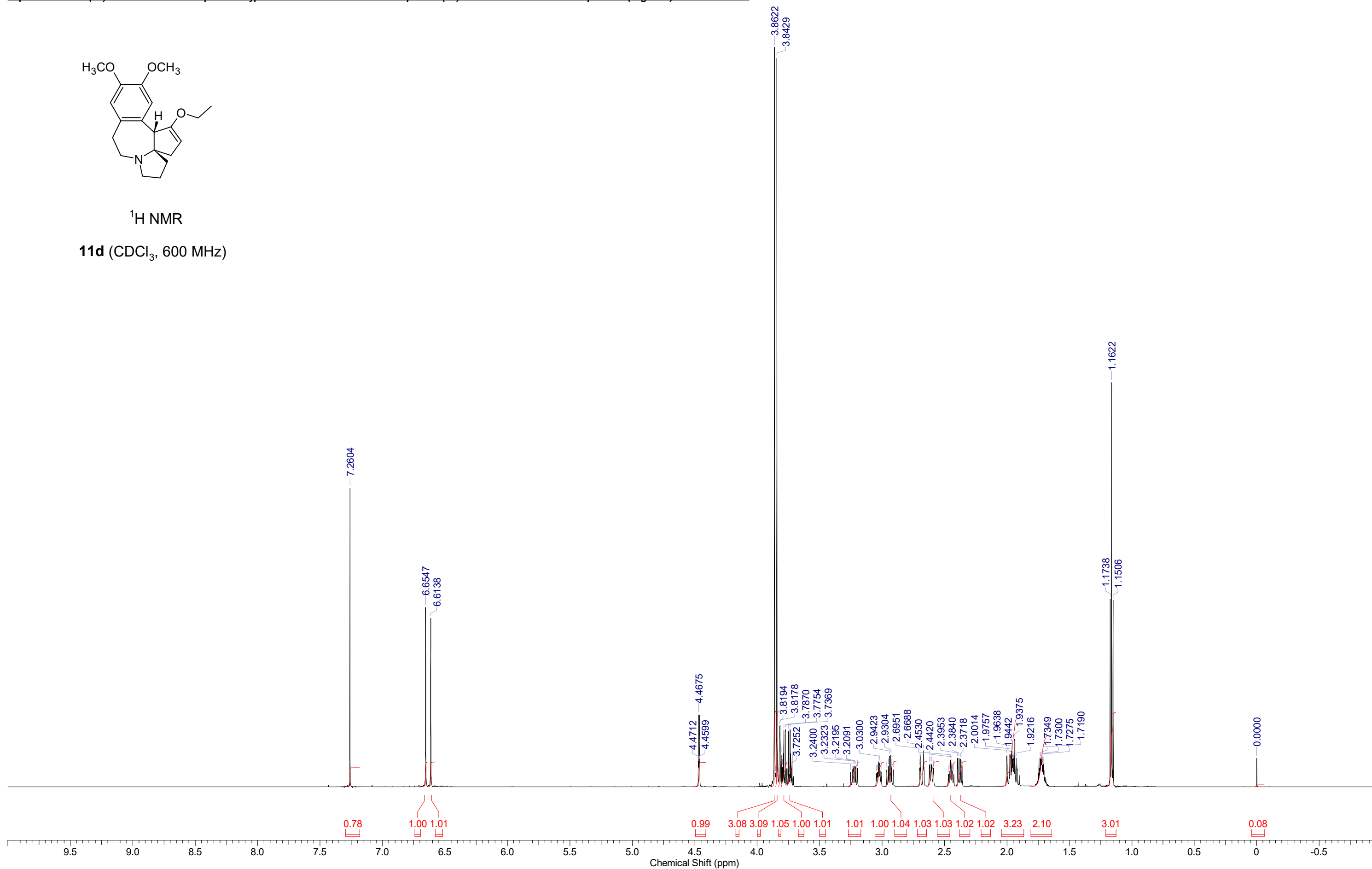


Acquisition Time (sec)	2.7263	Date	25 Jun 2019 14:11:28	Date Stamp	25 Jun 2019 14:11:28						
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2019¥長江紋加莉¥nagae01078_hr1¥10¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H					
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30	Receiver Gain	18.45	Number of Transients	8	Origin	spect
Spectrum Offset (Hz)	3692.0950	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.126	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d

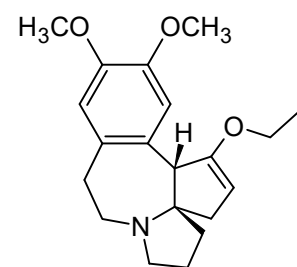


¹H NMR

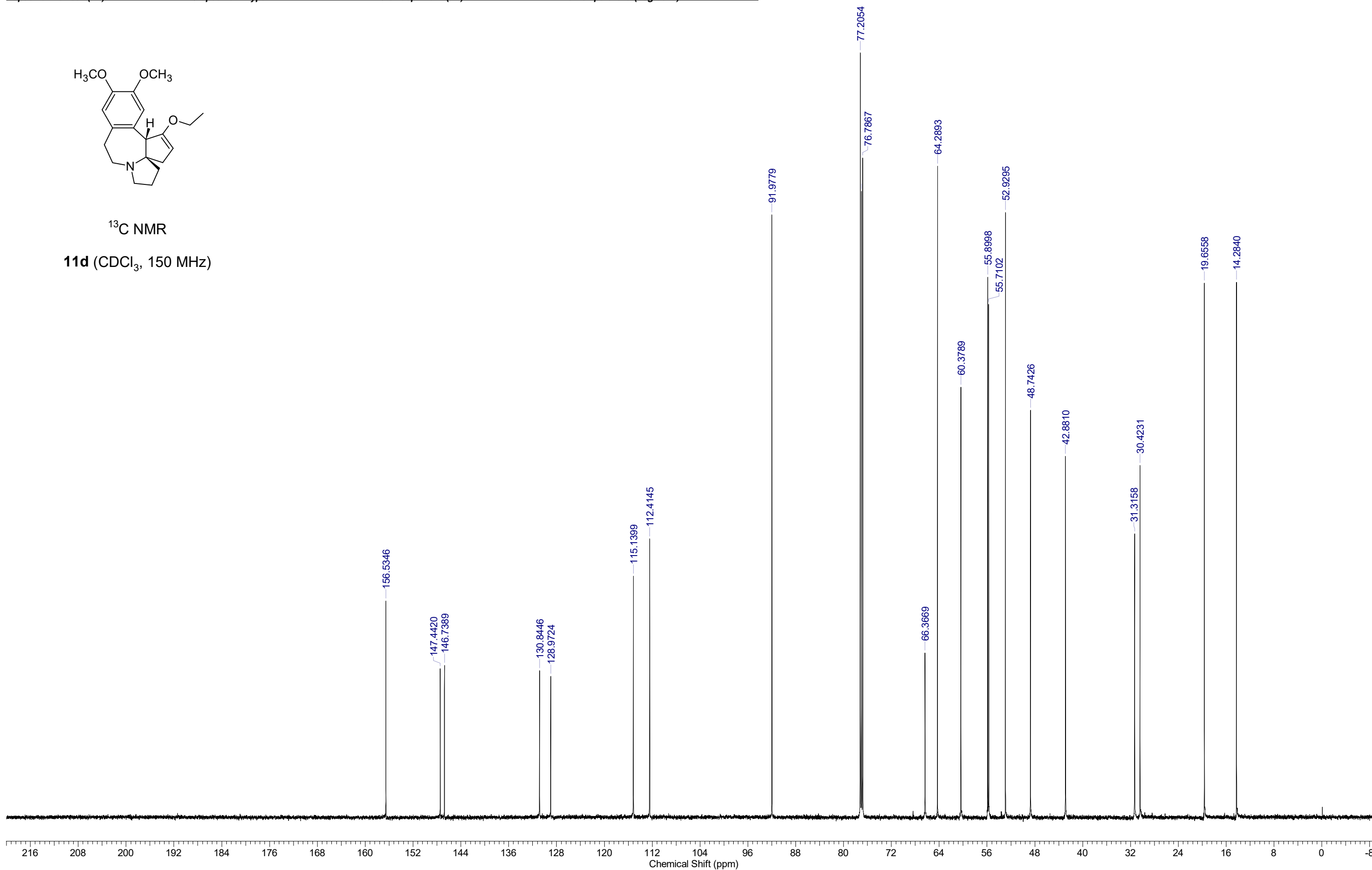
11d (CDCl₃, 600 MHz)



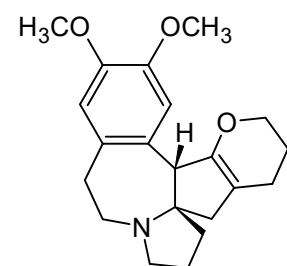
Acquisition Time (sec)	0.8389	Date	25 Jun 2019 14:20:00	Date Stamp	25 Jun 2019 14:20:00
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業生2019¥長江紋加莉¥nagae01078_hr1c¥10¥pdata¥1¥1r			Frequency (MHz)	150.90
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15074.7969	Spectrum Type	STANDARD	Receiver Gain	193.26
		Sweep Width (Hz)	39061.31	Number of Transients	128
				SW(cyclical) (Hz)	39062.50
				Temperature (degree C)	25.117
				Origin	spect
				Solvent	CHLOROFORM-d



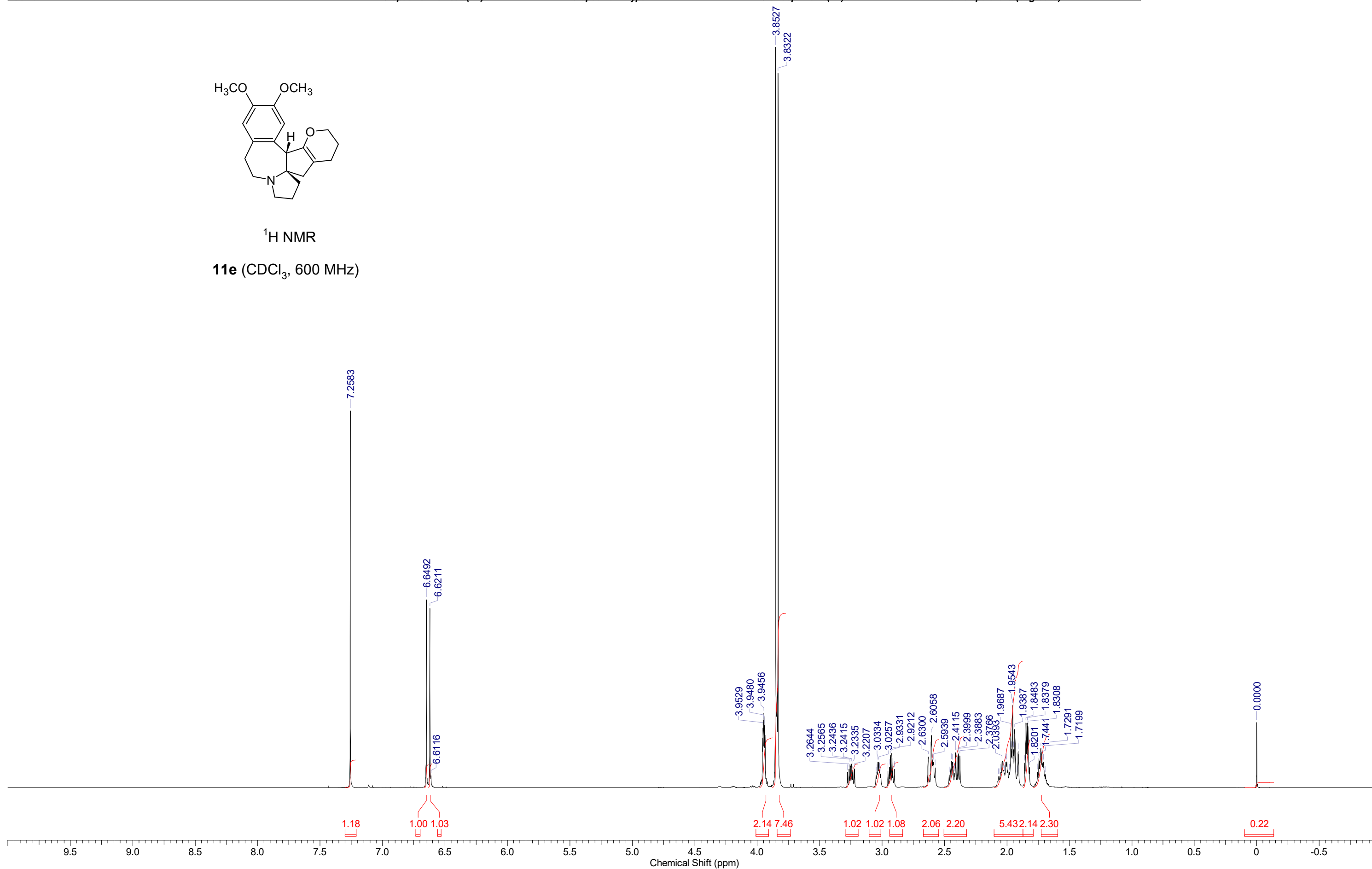
¹³C NMR
11d (CDCl₃, 150 MHz)



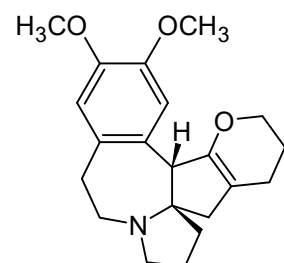
Acquisition Time (sec)	2.7263	Comment	1H	Date	26 Mar 2020 12:12:00	Date Stamp	26 Mar 2020 12:12:00
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01052¥①¥kokumura01052.1.1H¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3690.6277	Points Count	65536	Receiver Gain	22.72
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.146
						Number of Transients	8
						SW(cyclical) (Hz)	12019.23



¹H NMR
11e (CDCl₃, 600 MHz)

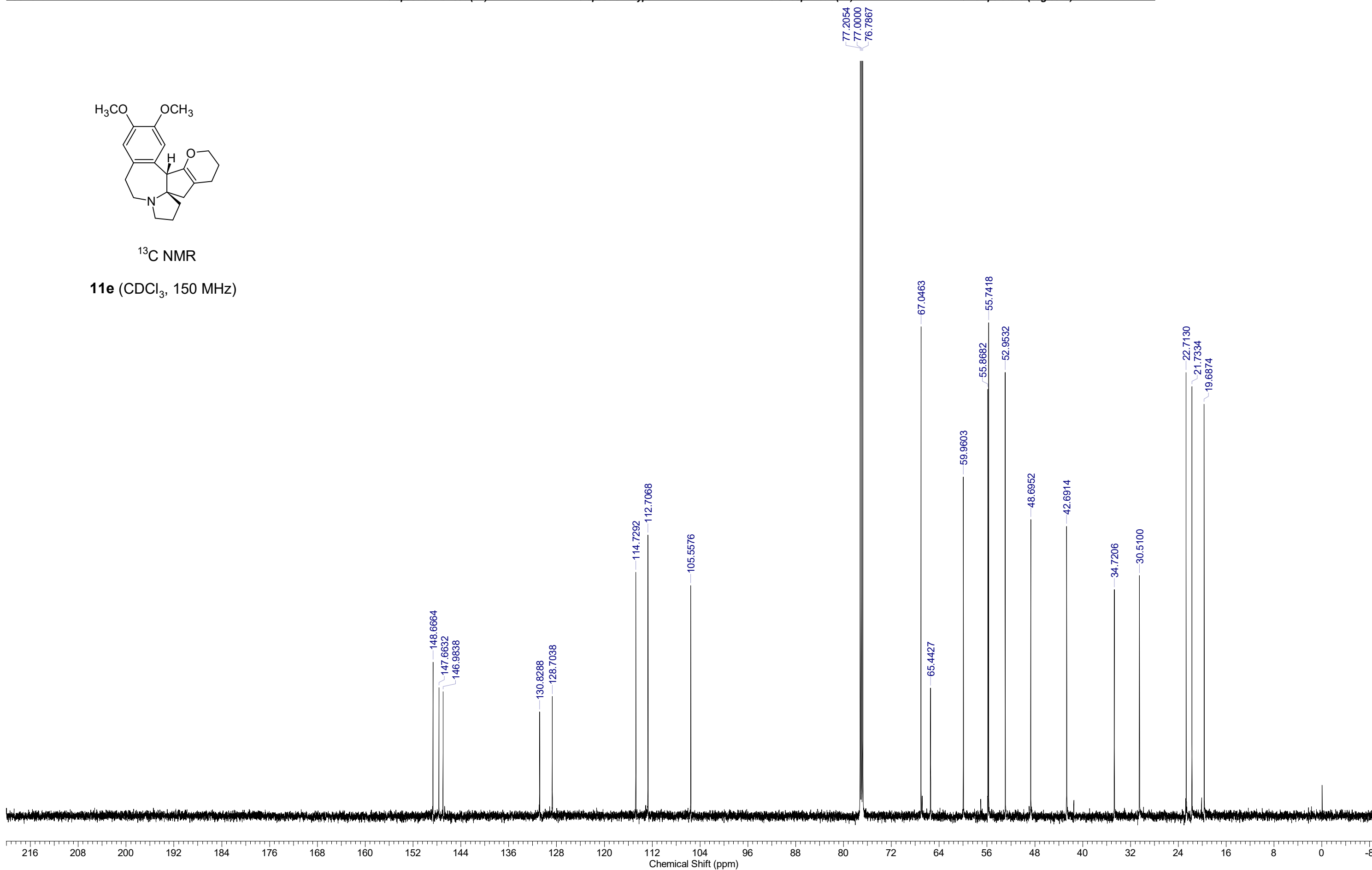


Acquisition Time (sec)	0.8389	Comment	13C	Date	26 Mar 2020 12:16:16	Date Stamp	26 Mar 2020 12:16:16
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01052¥(1)¥okumura01052_1_13C¥10¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15080.7578	Points Count	32768	Receiver Gain	193.26
		Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.148
						Number of Transients	64
						SW(cyclical) (Hz)	39062.50

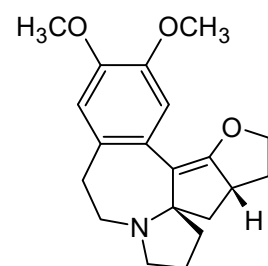


¹³C NMR

11e (CDCl₃, 150 MHz)

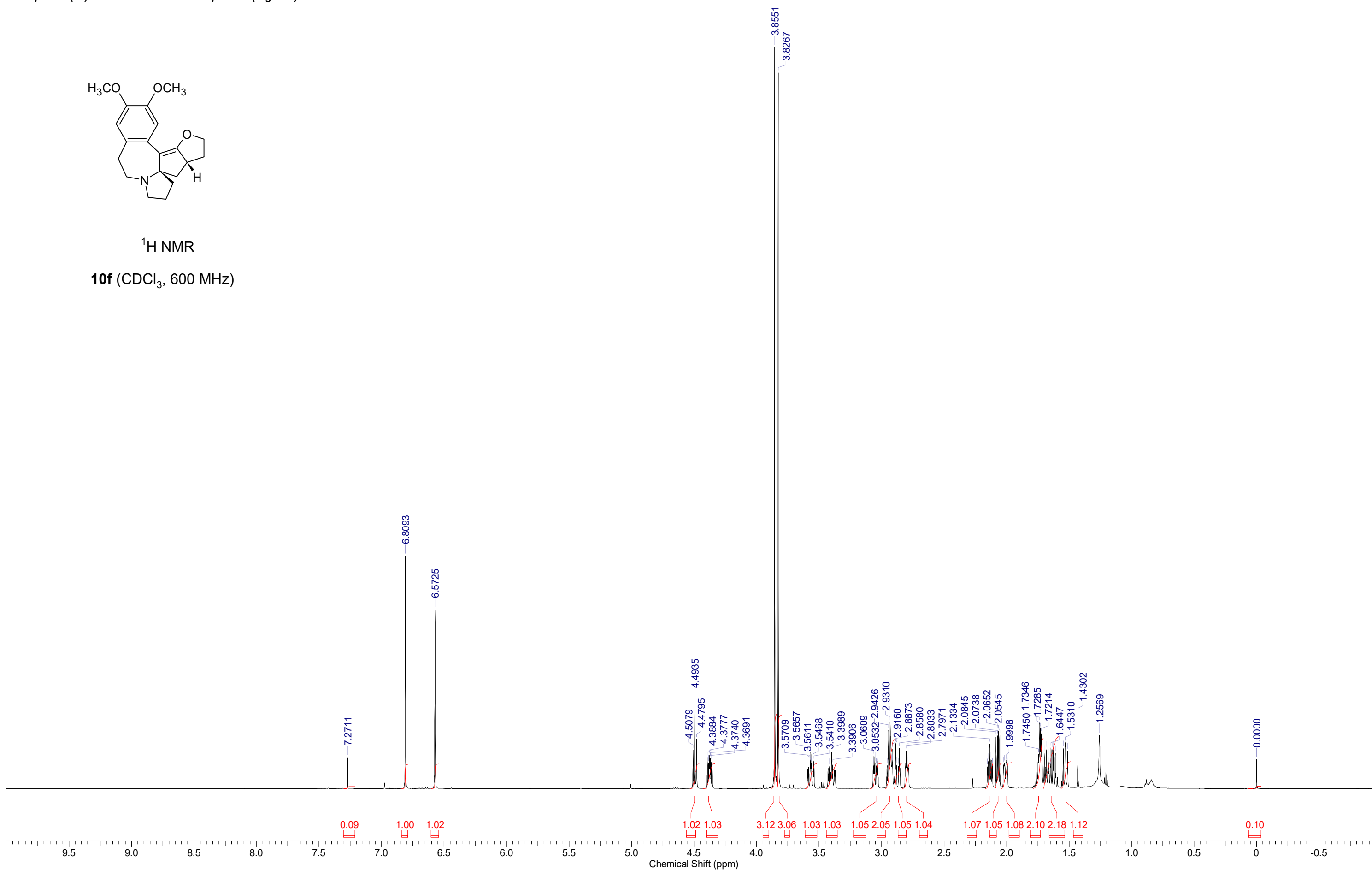


Acquisition Time (sec)	2.7263	Date	31 Oct 2020 20:35:28	Date Stamp	31 Oct 2020 20:35:28	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07110ueshita¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	16.31	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.157			Spectrum Offset (Hz)	3698.3306
						Points Count	65536
						Spectrum Type	STANDARD

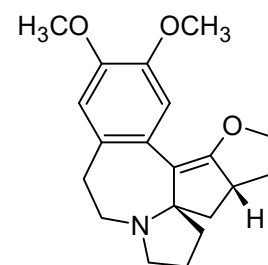


¹H NMR

10f (CDCl₃, 600 MHz)

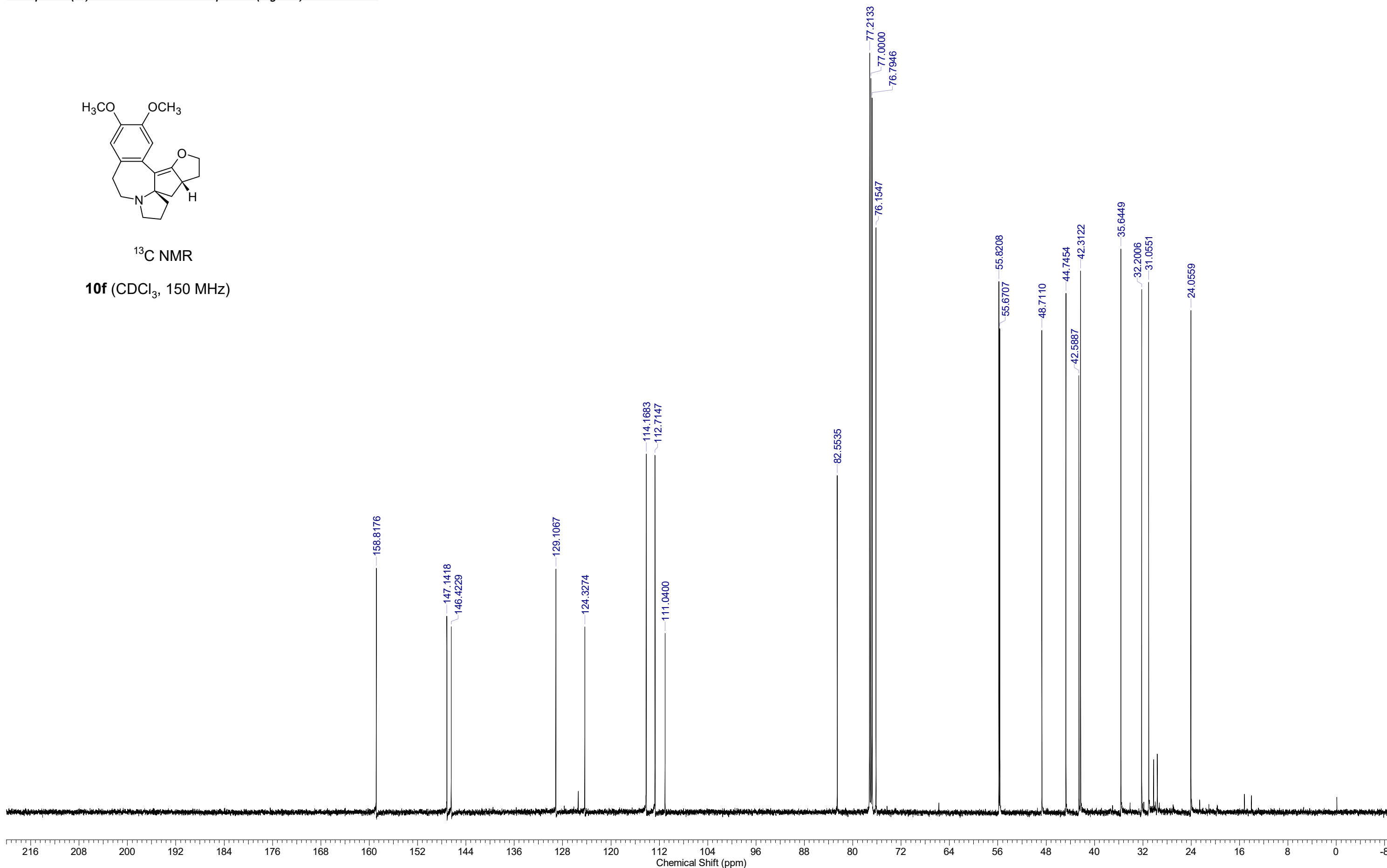


Acquisition Time (sec)	0.8389	Date	31 Oct 2020 20:39:44	Date Stamp	31 Oct 2020 20:39:44	File Name	¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai071110ueshita¥2¥pdata¥1¥1r						
Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	64	Origin	spect	Original Points Count	32768	Owner	nmsu	Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15075.9893	Spectrum Type	STANDARD		
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.156										

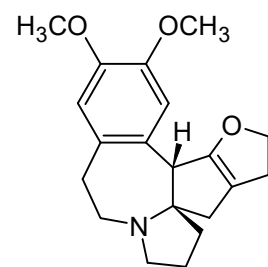


¹³C NMR

10f (CDCl₃, 150 MHz)

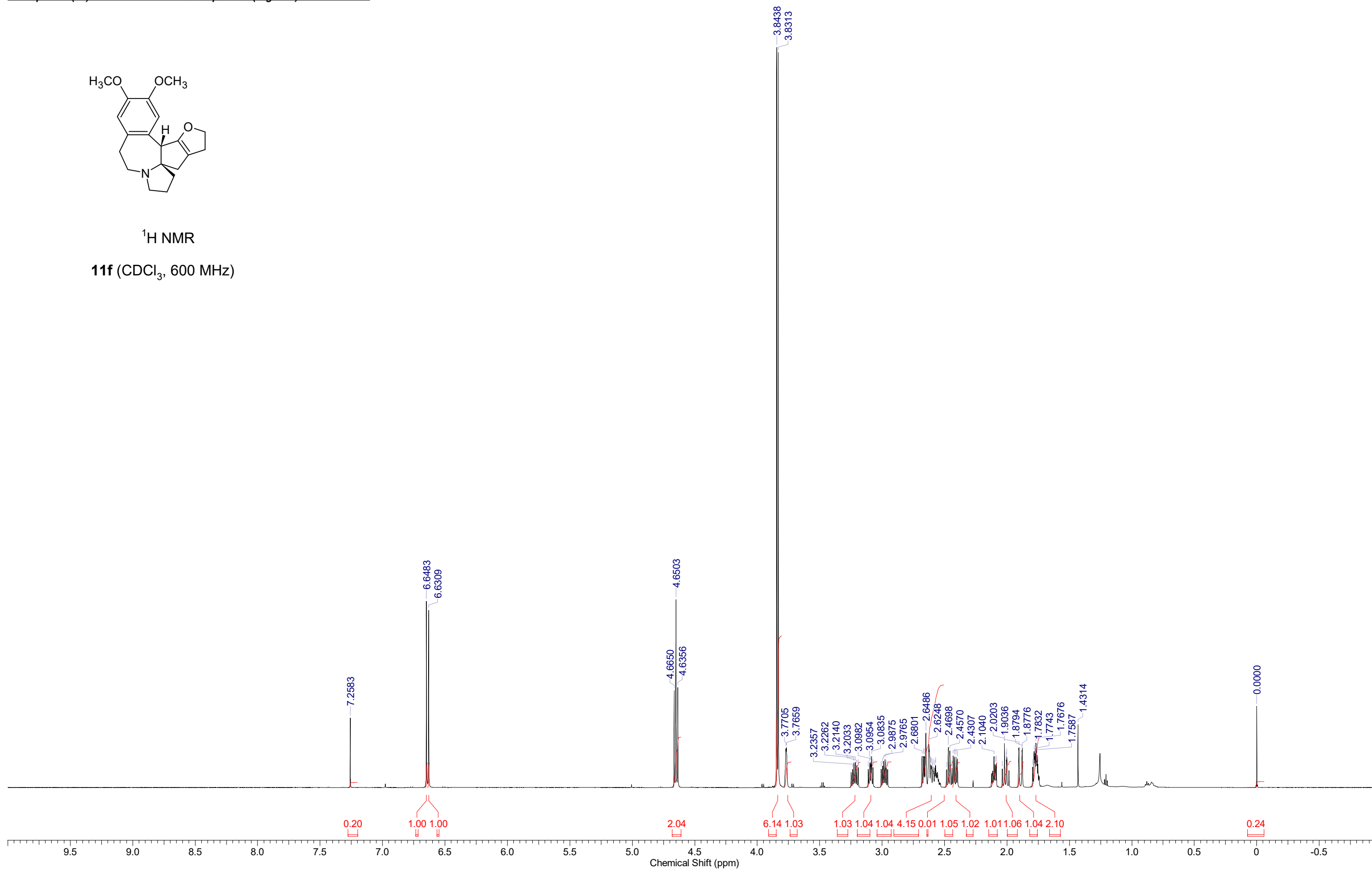


Acquisition Time (sec)	2.7263	Date	31 Oct 2020 20:16:16	Date Stamp	31 Oct 2020 20:16:16	File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07110ueue¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	19.87	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.130			Spectrum Offset (Hz)	3690.6277
						Points Count	65536
						Spectrum Type	STANDARD

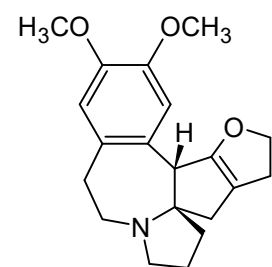


¹H NMR

11f (CDCl₃, 600 MHz)

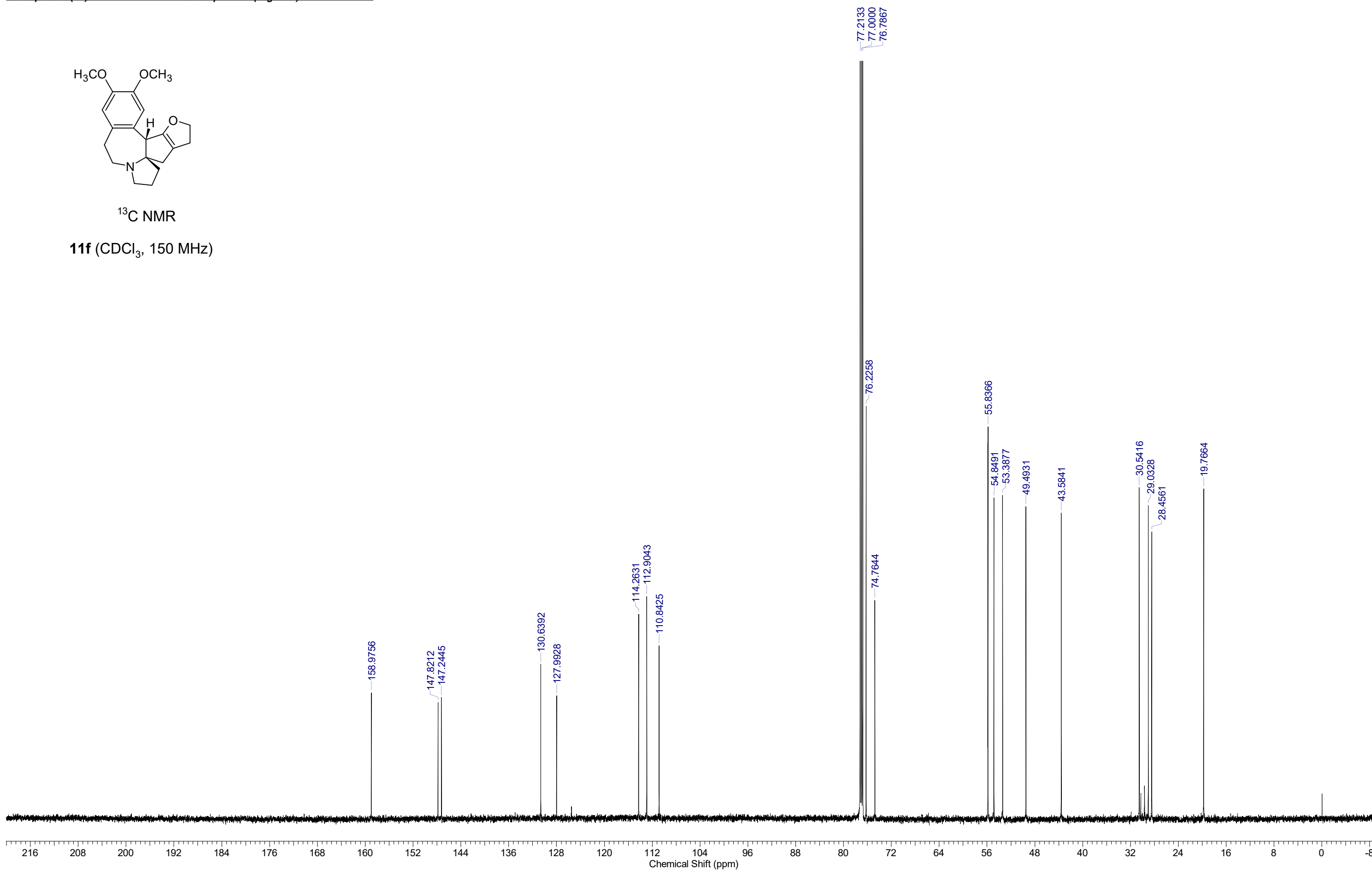


Acquisition Time (sec)	0.8389	Date	31 Oct 2020 20:22:40	Date Stamp	31 Oct 2020 20:22:40	File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07110ueue¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.151			Spectrum Offset (Hz)	15081.9492
						Points Count	32768
						Spectrum Type	STANDARD

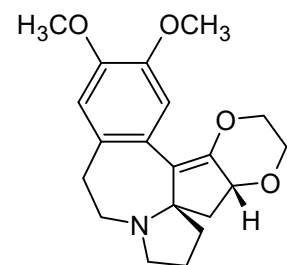


¹³C NMR

11f (CDCl₃, 150 MHz)

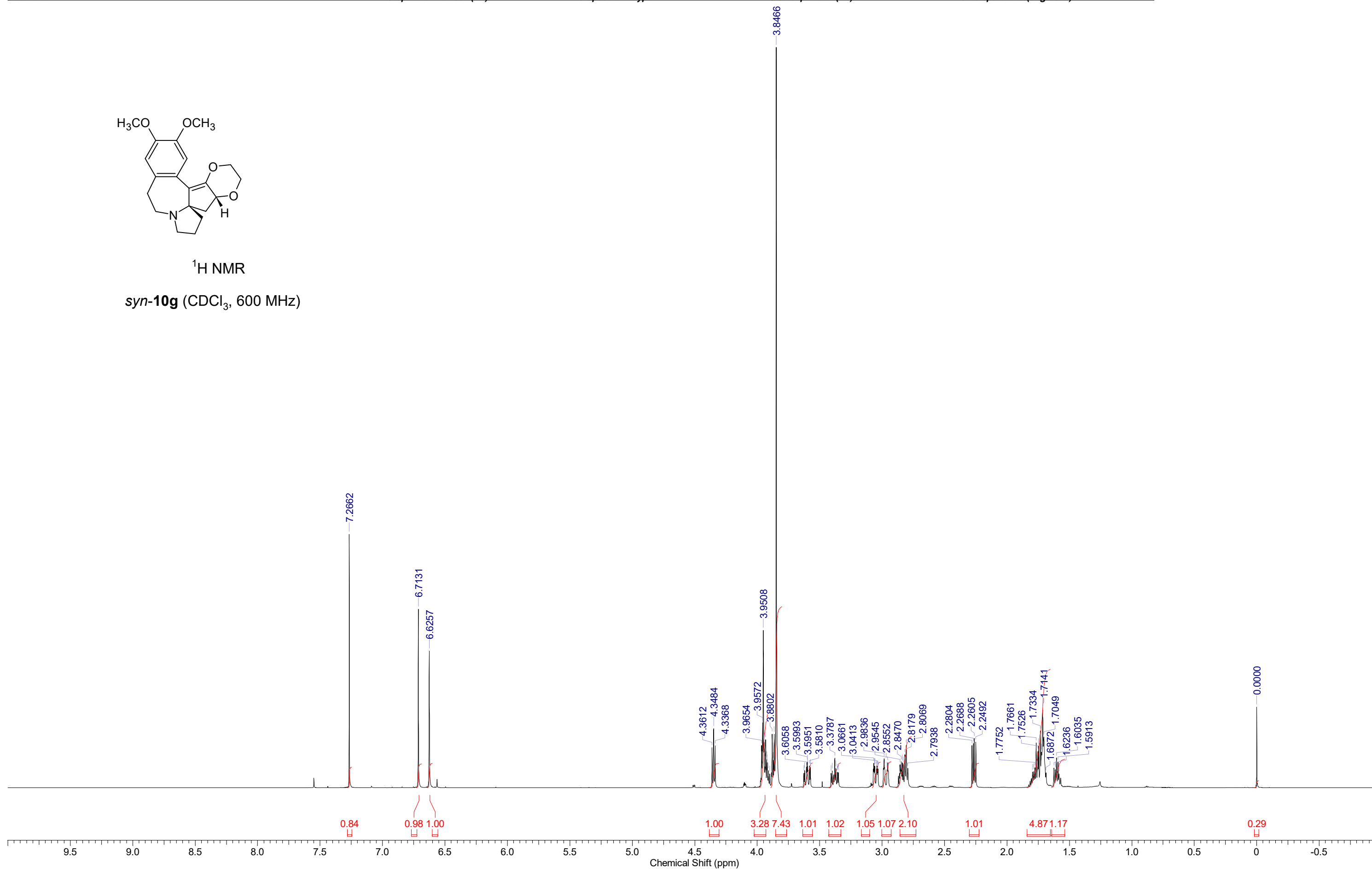


Acquisition Time (sec)	2.7263	Comment	1H	Date	31 Jan 2020 17:57:36	Date Stamp	31 Jan 2020 17:57:36
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01048¥③¥Yokumura01048.3.4column.1H¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3695.2126	Points Count	65536	Receiver Gain	25.50
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.147
						Number of Transients	8
						SW(cyclical) (Hz)	12019.23

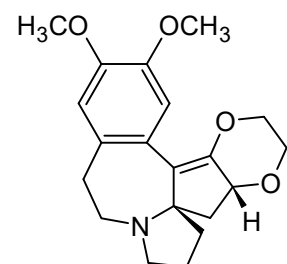


¹H NMR

syn-10g (CDCl₃, 600 MHz)

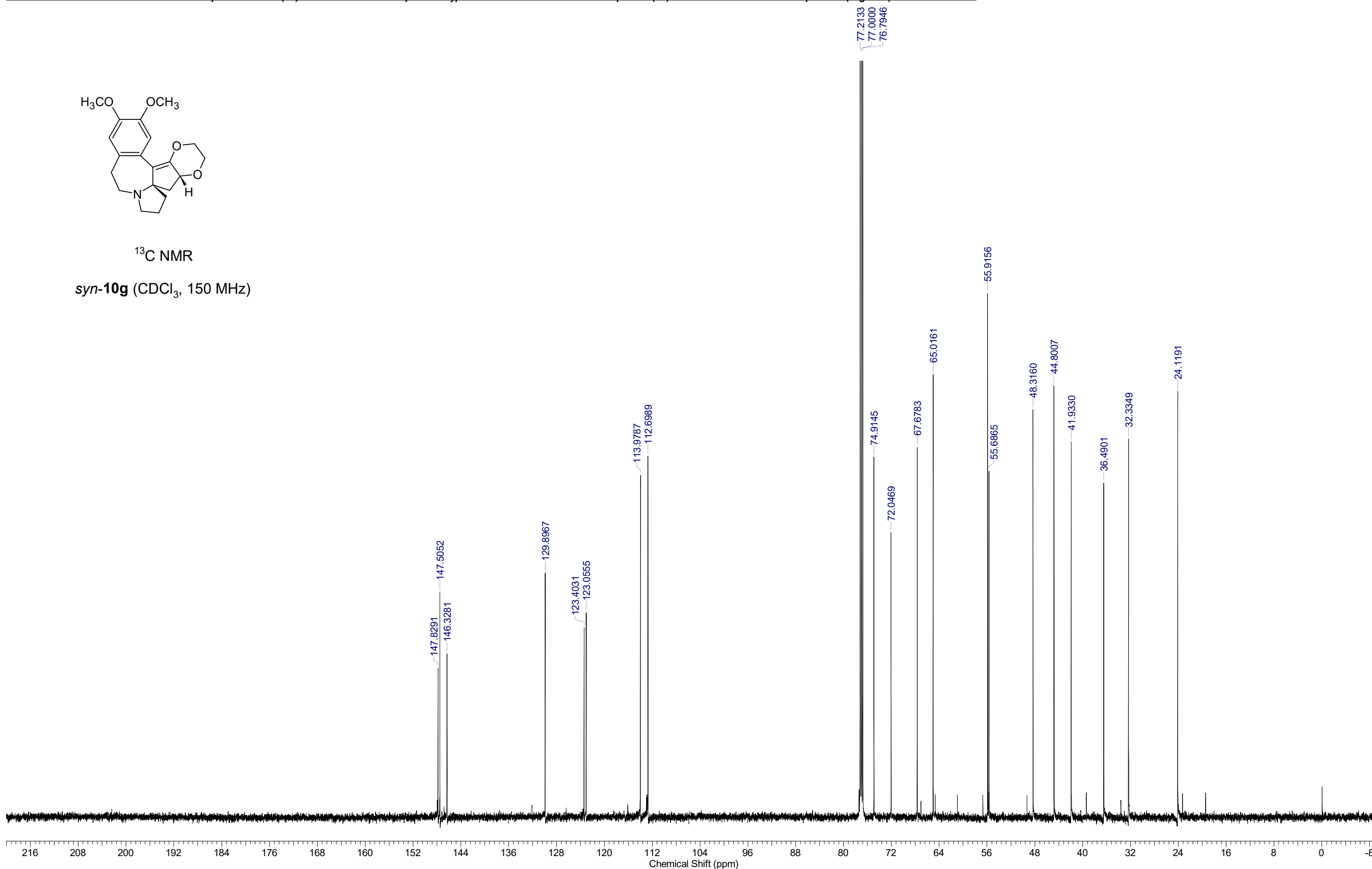


Acquisition Time (sec)	0.8389	Comment	13C	Date	31 Jan 2020 18:04:00	Date Stamp	31 Jan 2020 18:04:00
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01048¥③¥Yokumura01048.3.4column.13C¥10¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15083.1416	Points Count	32768	Receiver Gain	193.26
		Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	SW(cyclical) (Hz)	39062.50
				Temperature (degree C)	25.155		

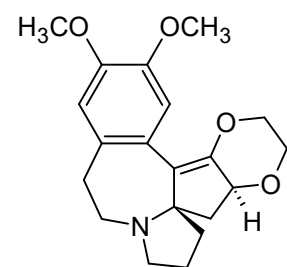


¹³C NMR

syn-10g (CDCl₃, 150 MHz)

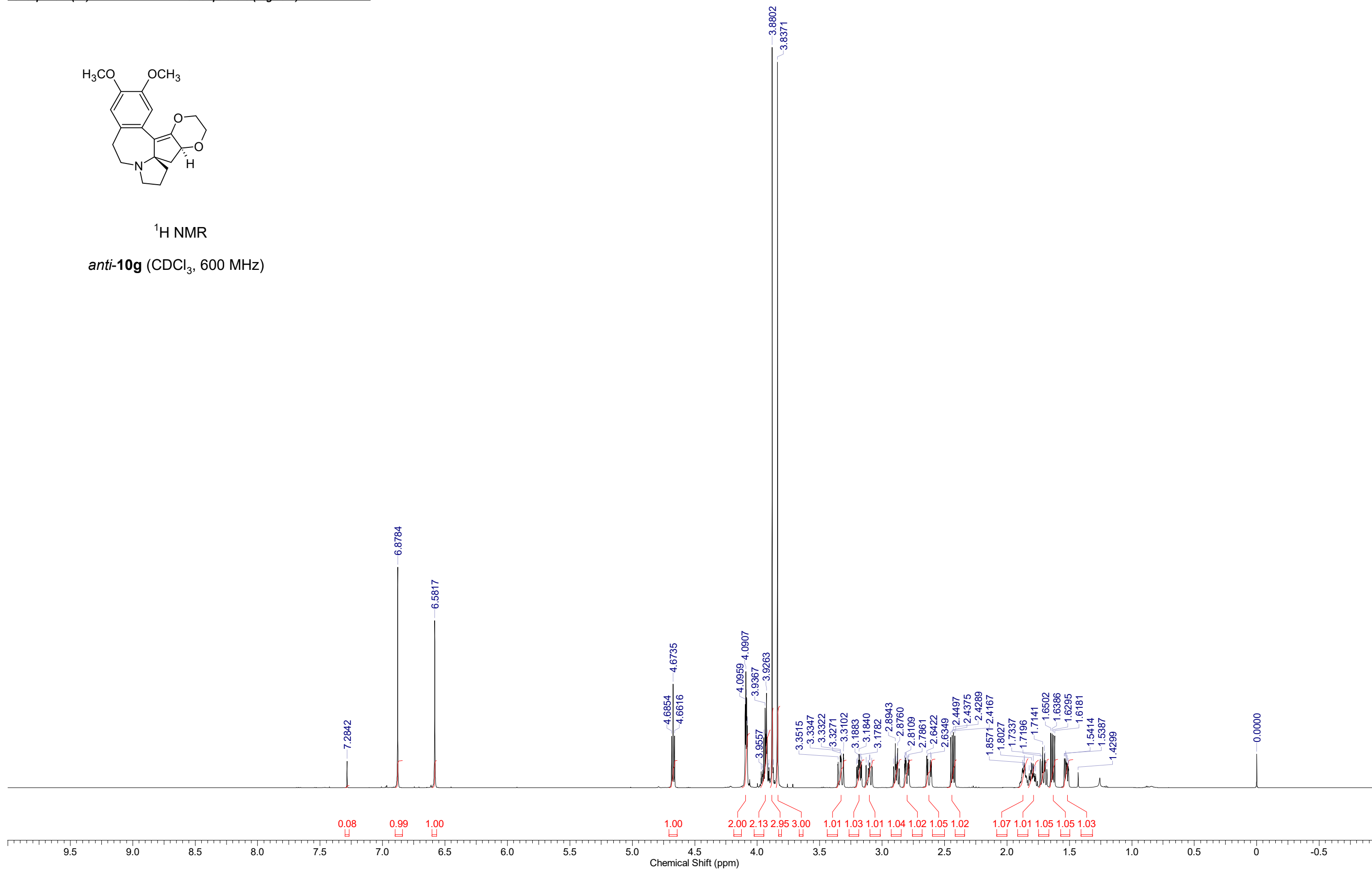


Acquisition Time (sec)	2.7263	Date	21 Nov 2020 13:18:08	Date Stamp	21 Nov 2020 13:18:08	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07070shita¥10¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	18.45	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.139			Spectrum Offset (Hz)	3706.0332
						Points Count	65536
						Spectrum Type	STANDARD

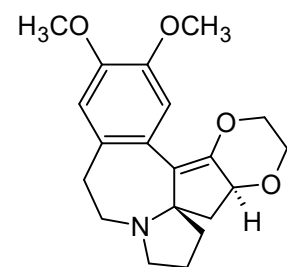


¹H NMR

anti-10g (CDCl₃, 600 MHz)

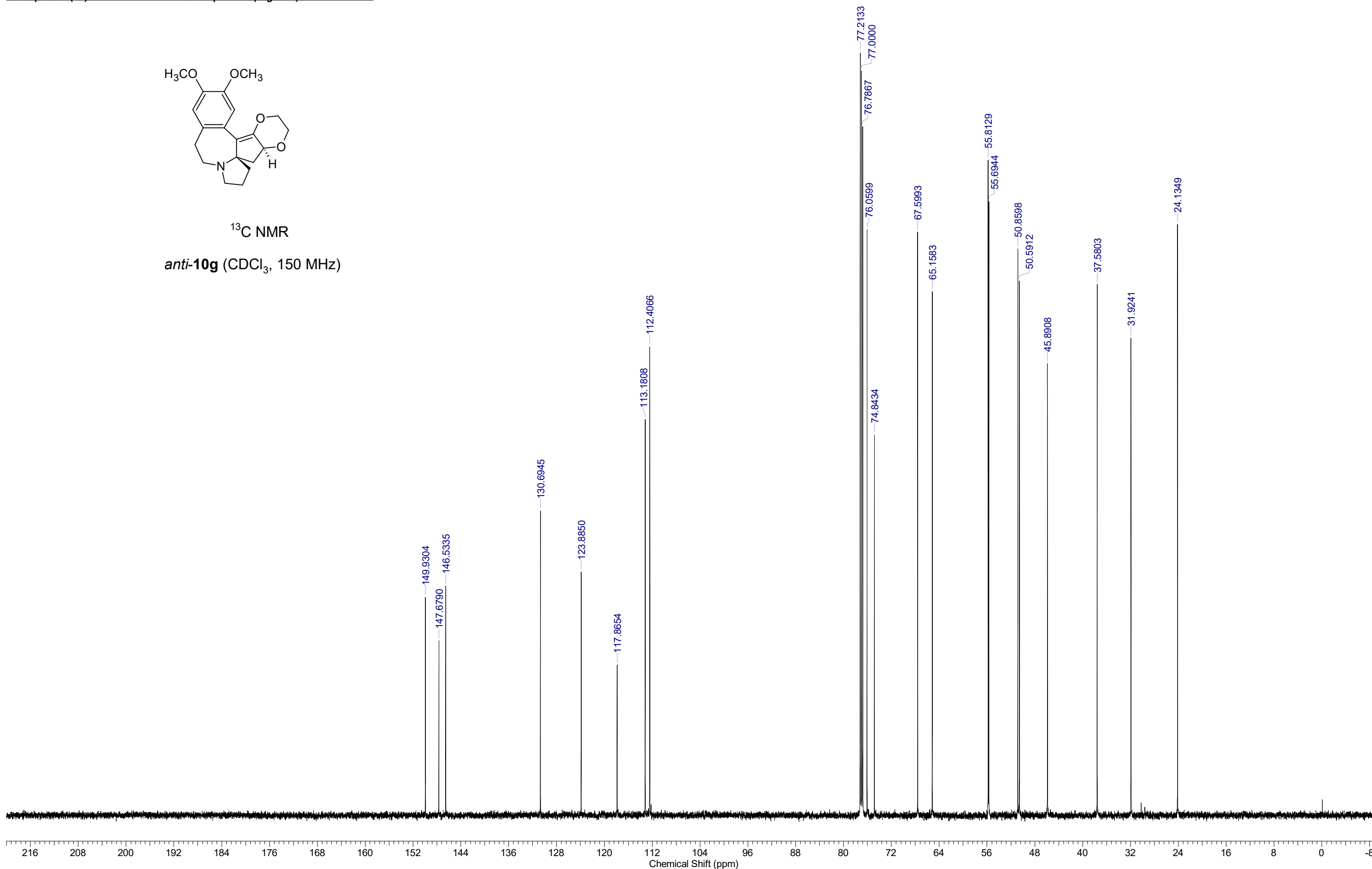


Acquisition Time (sec)	0.8389	Date	21 Nov 2020 13:20:16	Date Stamp	21 Nov 2020 13:20:16	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07070shita¥11¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.143			Spectrum Offset (Hz)	15075.9893
						Points Count	32768
						Spectrum Type	STANDARD

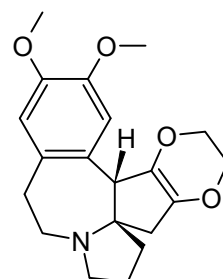


¹³C NMR

anti-10g (CDCl₃, 150 MHz)

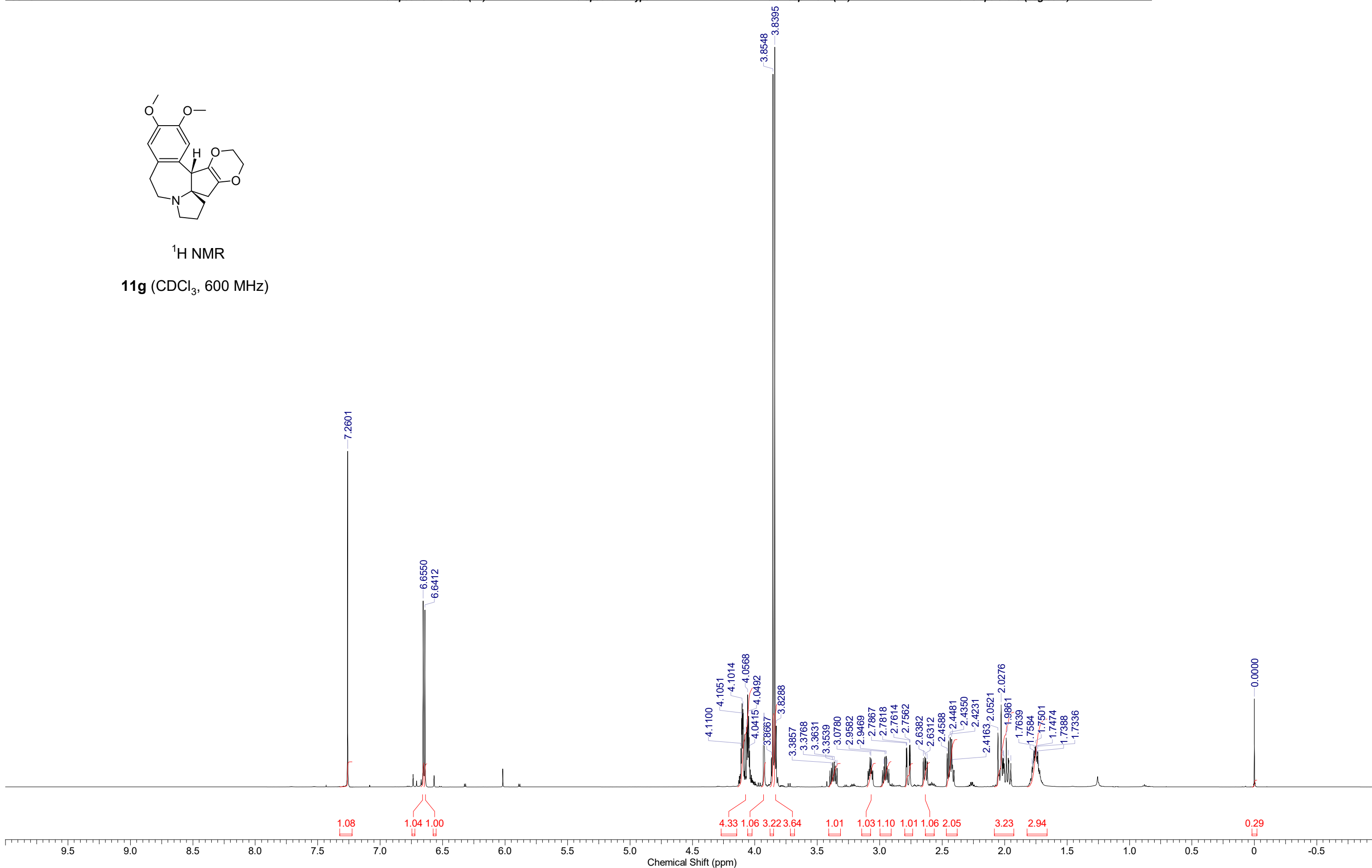


Acquisition Time (sec)	2.7263	Comment	1H	Date	31 Jan 2020 17:32:00	Date Stamp	31 Jan 2020 17:32:00
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01048¥2¥okumura01048_2_5column_1H¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Origin	spect	Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3691.5281	Points Count	65536	Receiver Gain	22.72
		Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.155
						Number of Transients	8
						SW(cyclical) (Hz)	12019.23

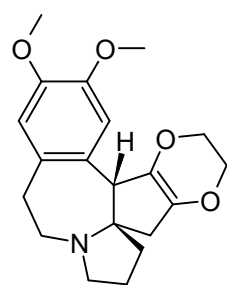


¹H NMR

11g (CDCl₃, 600 MHz)

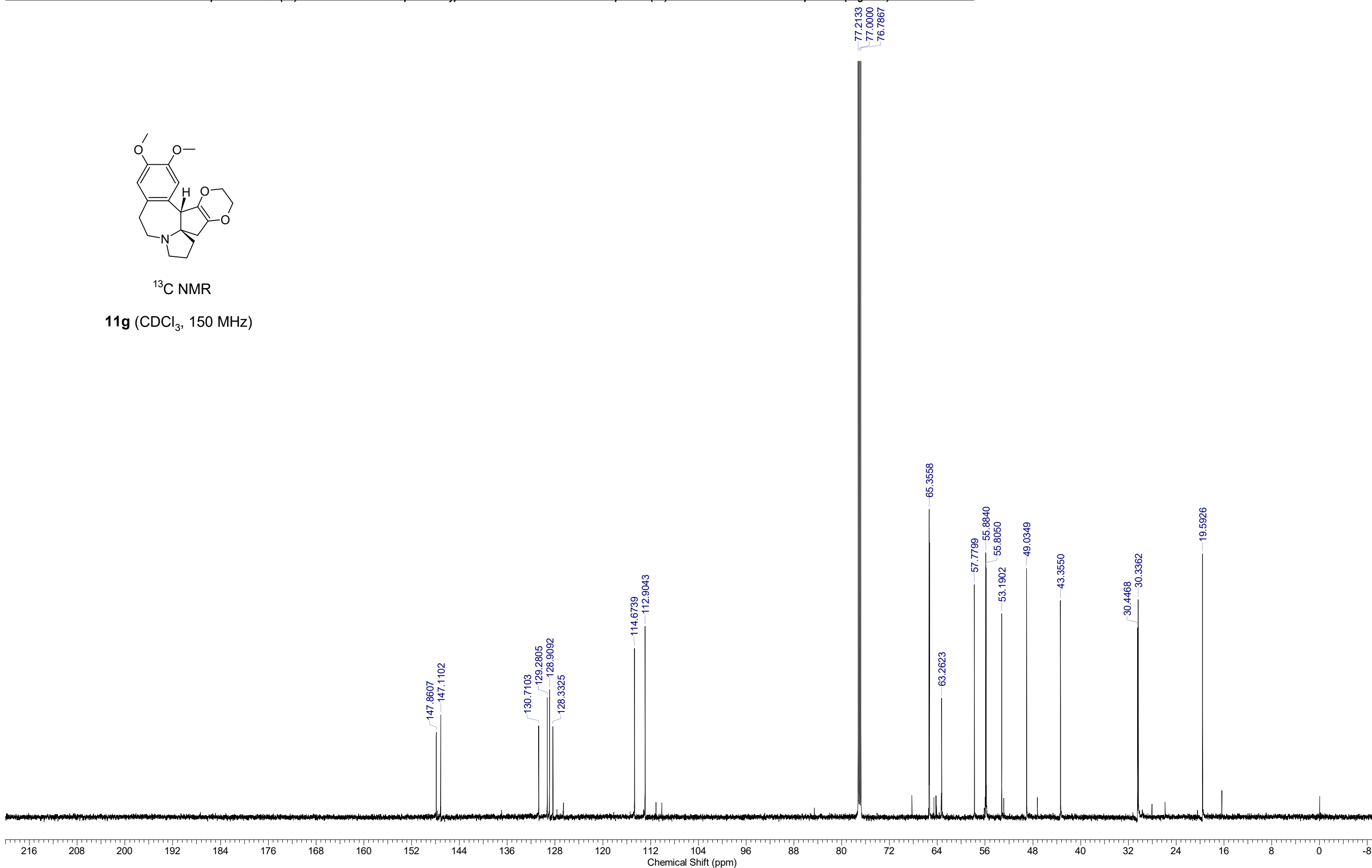


Acquisition Time (sec)	0.8389	Comment	13C	Date	31 Jan 2020 17:38:24	Date Stamp	31 Jan 2020 17:38:24
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01048¥2¥okumura01048 2 5column 13C¥10¥pdata¥1¥1r	Original Points Count	32768	Owner	nmsu	Frequency (MHz)	150.90
Origin	spect	Spectrum Offset (Hz)	15081.9492	Points Count	32768	Nucleus	13C
Solvent	CHLOROFORM-d	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
						SW(cyclical) (Hz)	39062.50
						Temperature (degree C)	25.148

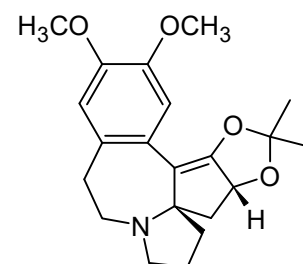


¹³C NMR

11g (CDCl₃, 150 MHz)

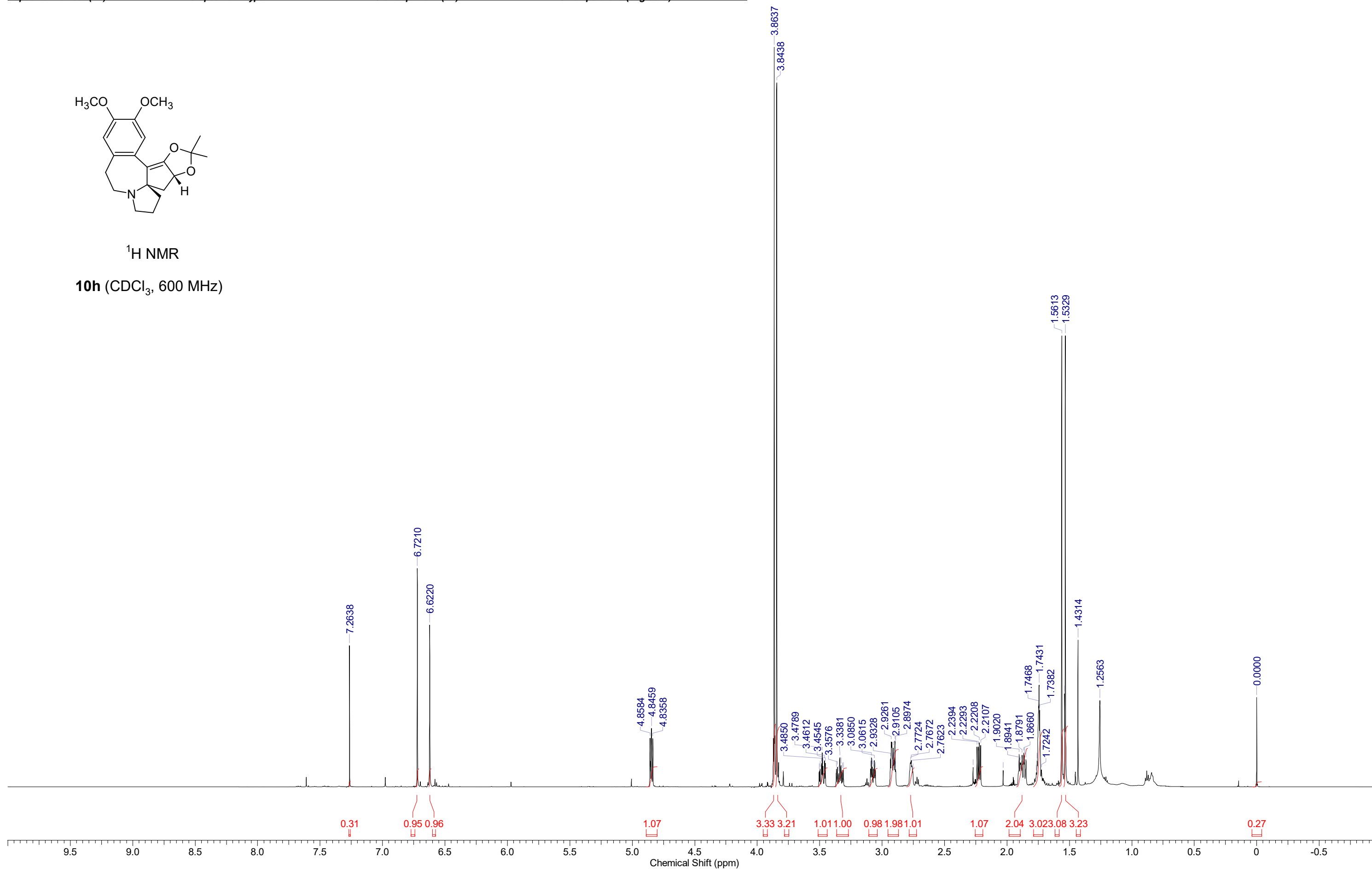


Acquisition Time (sec)	2.7263	Comment	proton	Date	21 Nov 2020 11:42:08	Date Stamp	21 Nov 2020 11:42:08
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥okumura01057_nakaue¥1¥pdata¥1¥1r	Owner	nmsu	Points Count	65536	Nucleus	1H
Original Points Count	32768	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
Spectrum Offset (Hz)	3693.9290	Temperature (degree C)			25.145	Number of Transients	8
						SM(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

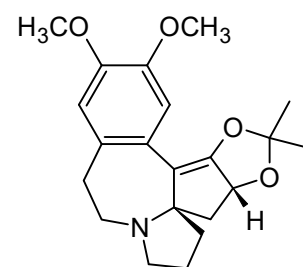


¹H NMR

10h (CDCl₃, 600 MHz)

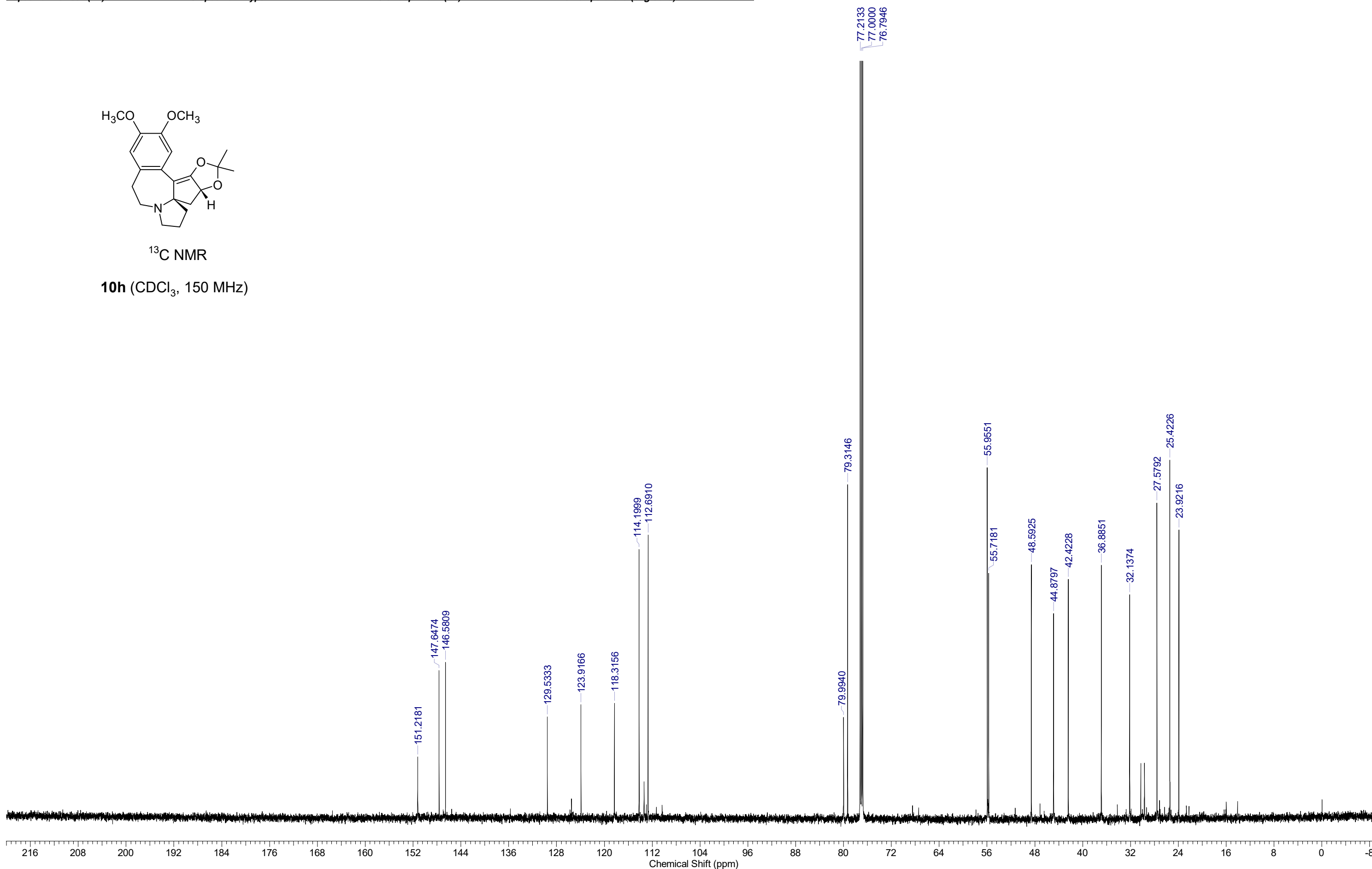


Acquisition Time (sec)	0.8389	Comment	carbon	Date	21 Nov 2020 11:46:24	Date Stamp	21 Nov 2020 11:46:24
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥okumura01057_nakaue¥2¥pdata¥1¥1r	Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	64
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15083.1416	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.150	SM(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

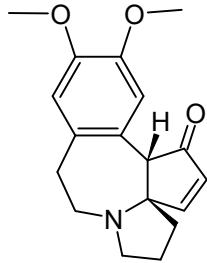
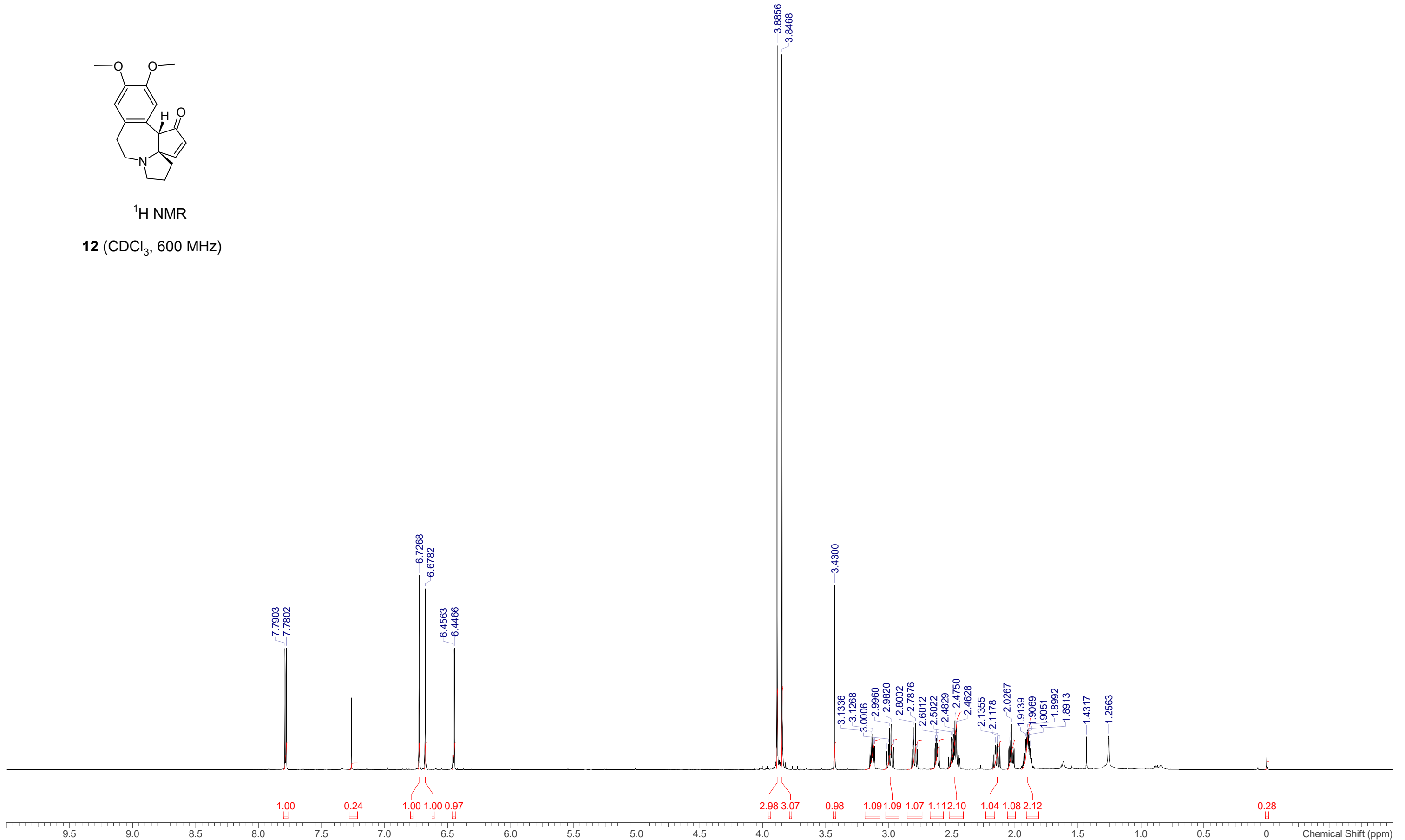


¹³C NMR

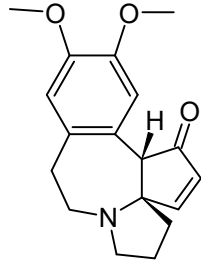
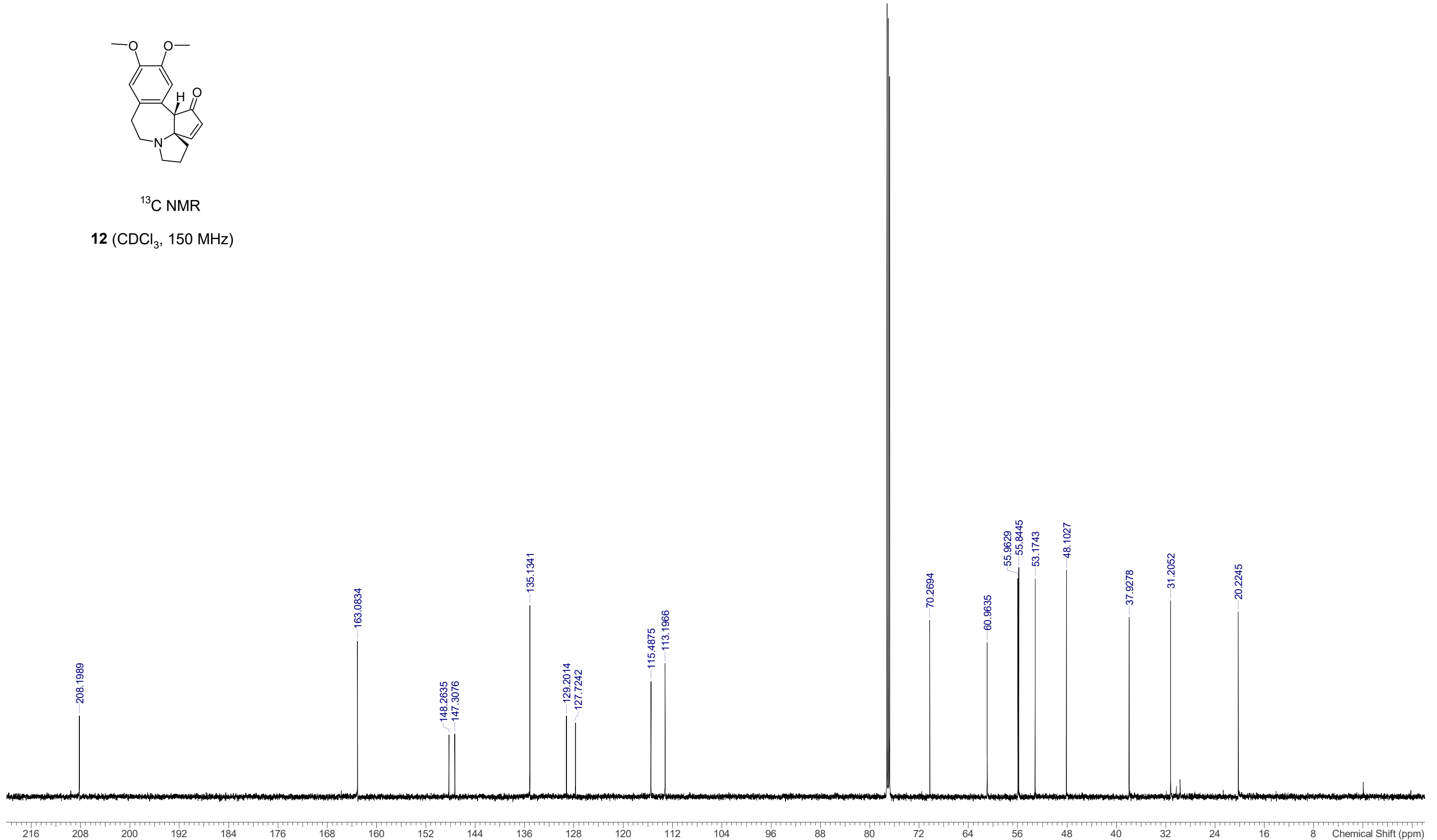
10h (CDCl₃, 150 MHz)



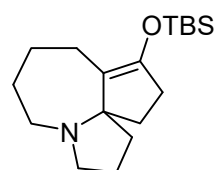
Acquisition Time (sec)	2.7263	Comment	Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	D	0.0002	D1	3	DE	10	DS	0
Date	21 Nov 2020 13:11:18 (GMT+09:00)	Date Stamp	21 Nov 2020 13:11:18 (GMT+09:00)	ExpNo	40	GB	0	INSTRUM	<spect>	LB	0.3
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥okumura01068¥40¥pdata¥1¥1r	Frequency (MHz)	600.1300	Original Points Count	32768	Owner	nmsu	PC	1	SF	600.130012942287
NS	8	Nucleus	1H	Number of Transients	8	Origin	spect	Pulse Sequence	zg30	Receiver Gain	50.88
PROBHD	<Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zg30>	Points Count	65536	SSB	0	SW(cyclical) (Hz)	12019.23	SWH	12019.2307692308
SFO1	600.133706048414	SI	65536	Spectrum Offset (Hz)	3693.1667	Spectrum Type	standard	Sweep Width (Hz)	12019.05	TD	65536
Solvent	CHLOROFORM-d	UNC1	<1H>	WDW	1						
TE	298.1535	Temperature (degree C)	25.153								

¹H NMR12 (CDCl₃, 600 MHz)

Acquisition Time (sec)	0.8389	Comment	Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	D	0.03	D1	2	DE	18	DS	4
Date	21 Nov 2020 13:14:52 (GMT+09:00)	Date Stamp	21 Nov 2020 13:14:52 (GMT+09:00)	ExpNo	41	ExpNo	41	INSTRUM	<spect>	LB	1
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業予定2020¥奥村知世¥okumura01068¥41¥pdata¥1¥1r	Frequency (MHz)	150.9028	GB	0	Original Points Count	32768	Owner	nmsu	PC	1.4
NS	64	Nucleus	13C	Number of Transients	64	Origin	spect	Pulse Sequence	zgpg30	Receiver Gain	193.26
PROBHD	<Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zgpg30>	Points Count	32768	SSB	0	SW(cyclical) (Hz)	39062.50	SWH	39062.5
SFO1	150.917898095859	SI	32768	TD	65536	TDO	1	TE	298.1489	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15081.9434	Spectrum Type	standard	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.149				
UNC1	<13C>	WDW	1								

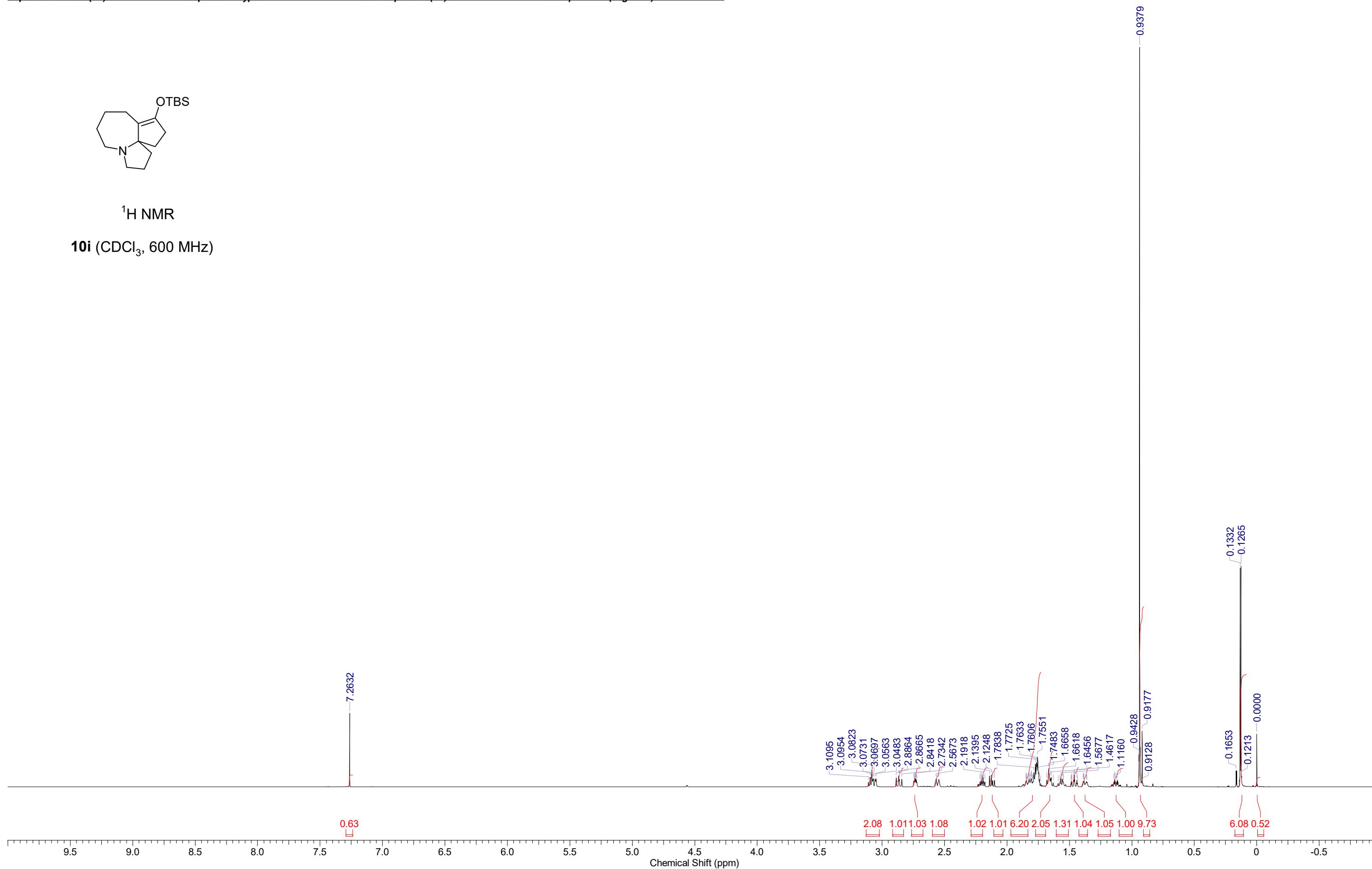
¹³C NMR12 (CDCl₃, 150 MHz)

Acquisition Time (sec)	2.7263	Comment	proton	Date	04 Mar 2020 18:14:40	Date Stamp	04 Mar 2020 18:14:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07036 fr2¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3693.3787	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.157	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

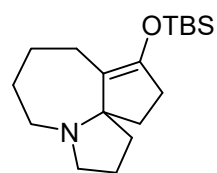


¹H NMR

10i (CDCl₃, 600 MHz)

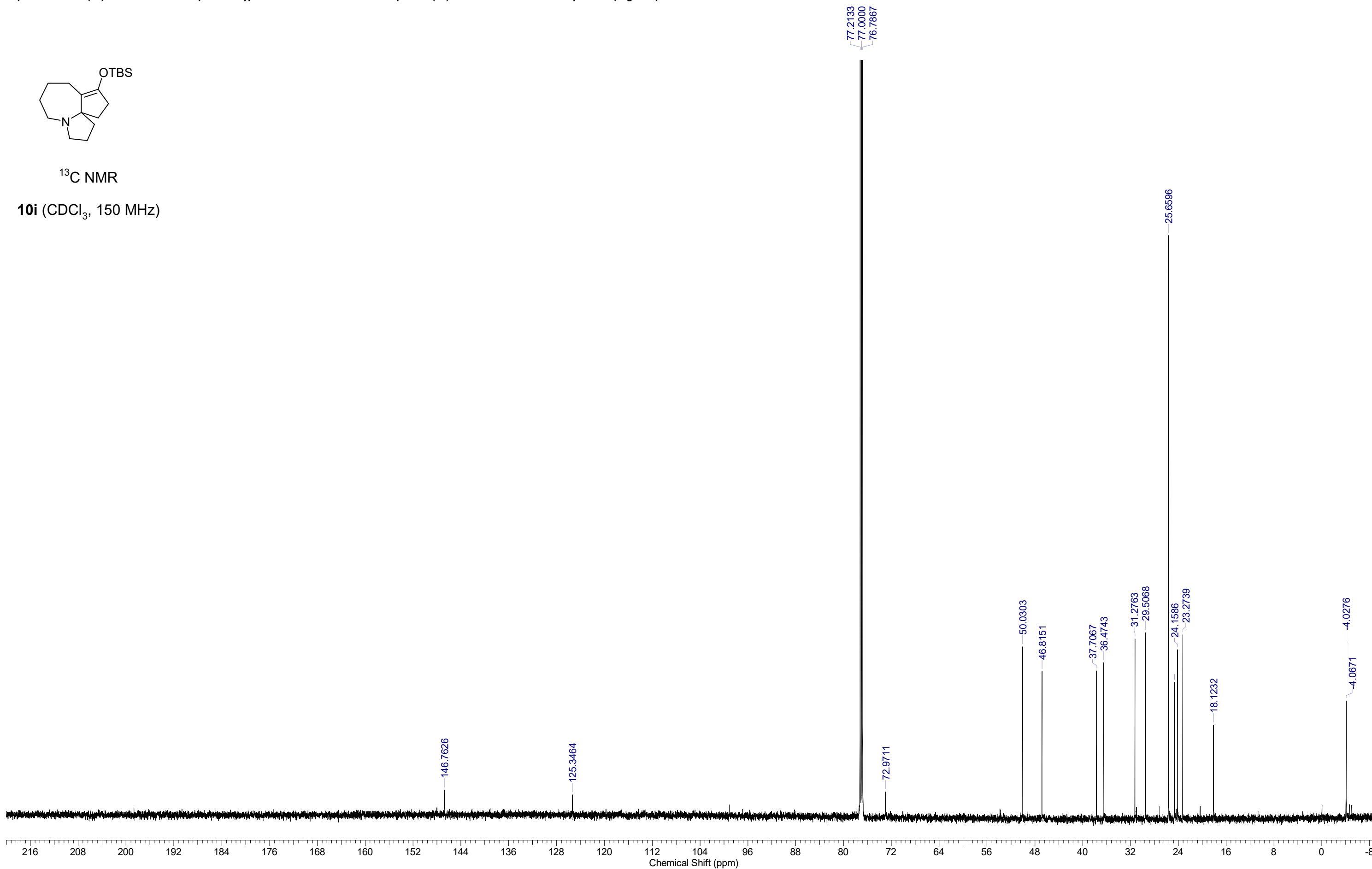


Acquisition Time (sec)	0.8389	Comment	carbon	Date	04 Mar 2020 18:48:48	Date Stamp	04 Mar 2020 18:48:48
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07036_fr2¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15086.7188	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.159	SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

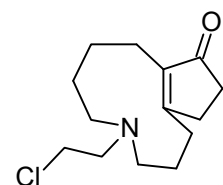


¹³C NMR

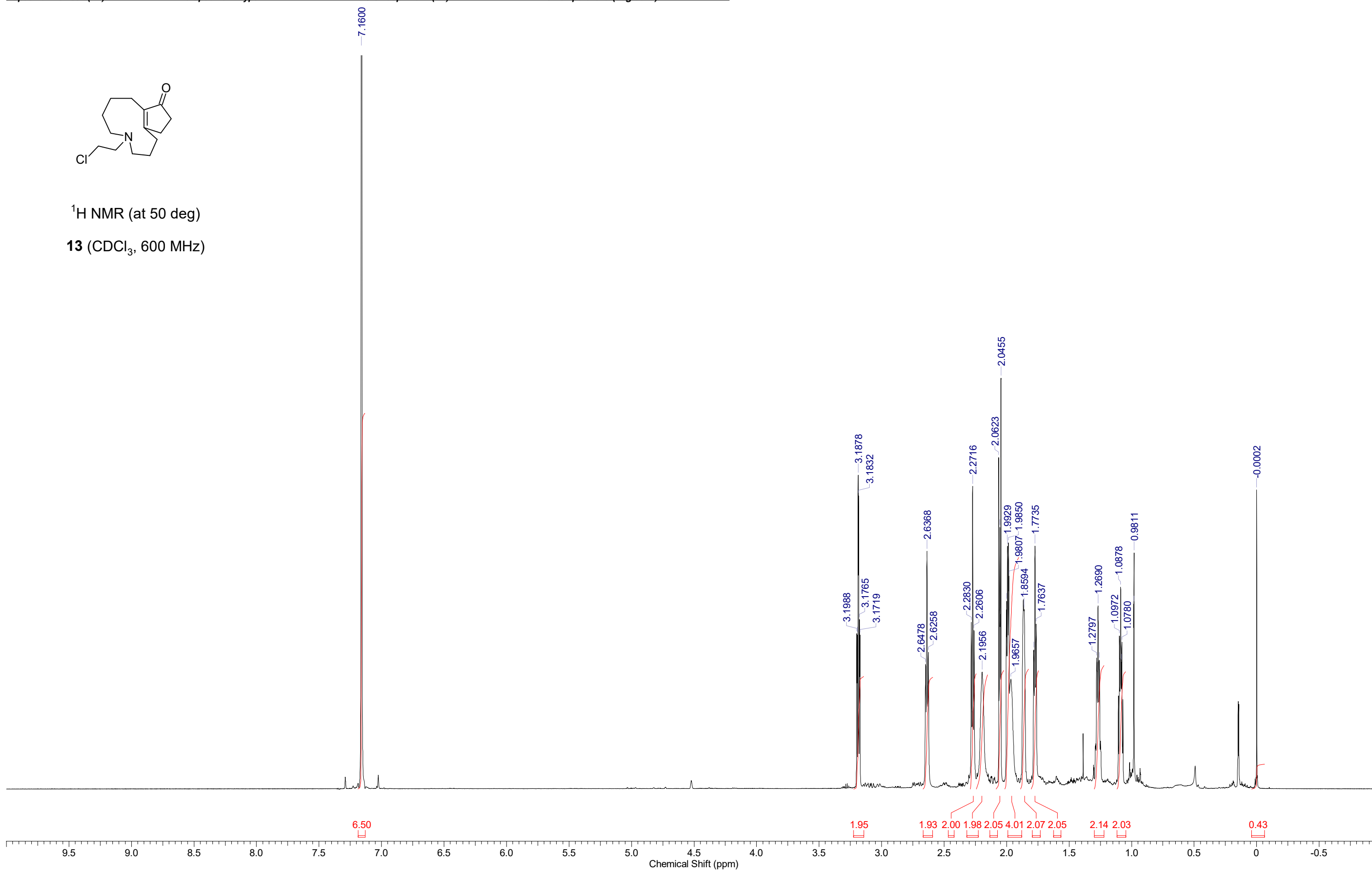
10i (CDCl₃, 150 MHz)



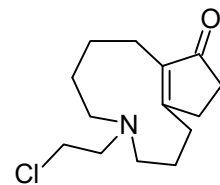
Acquisition Time (sec)	2.7263	Comment	proton50deg	Date	05 Mar 2020 12:29:04	Date Stamp	05 Mar 2020 12:29:04
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07036 fr4 ben¥3¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3711.7969	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	50.154	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	BENZENE-d6



¹H NMR (at 50 deg)
13 (CDCl₃, 600 MHz)

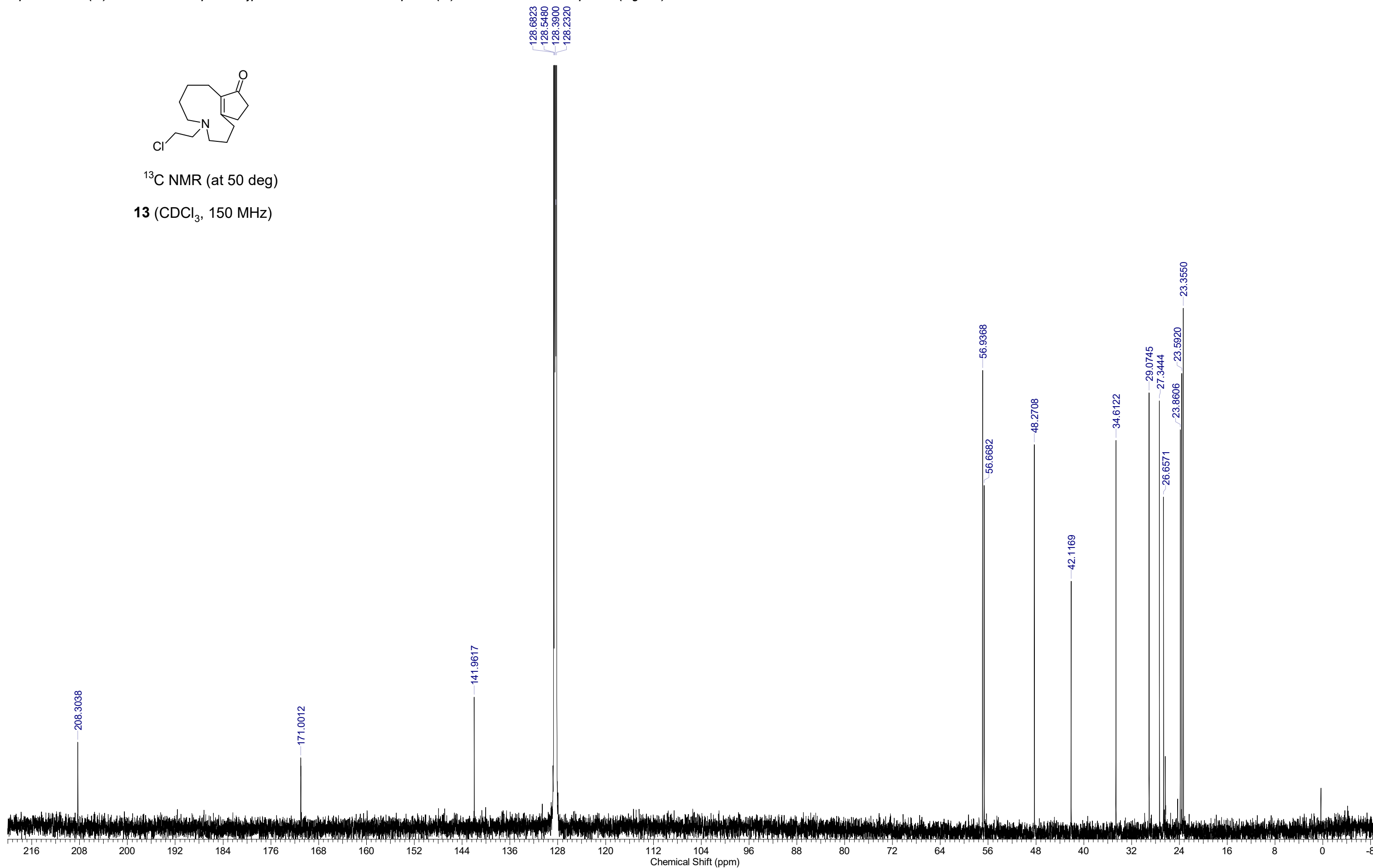


Acquisition Time (sec)	0.8389	Comment	carbon50deg	Date	05 Mar 2020 12:31:12	Date Stamp	05 Mar 2020 12:31:12
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07036 fr4 ben¥4¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15205.0645	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	50.159	Number of Transients	64
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	BENZENE-d6

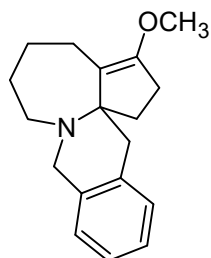


¹³C NMR (at 50 deg)

13 (CDCl₃, 150 MHz)

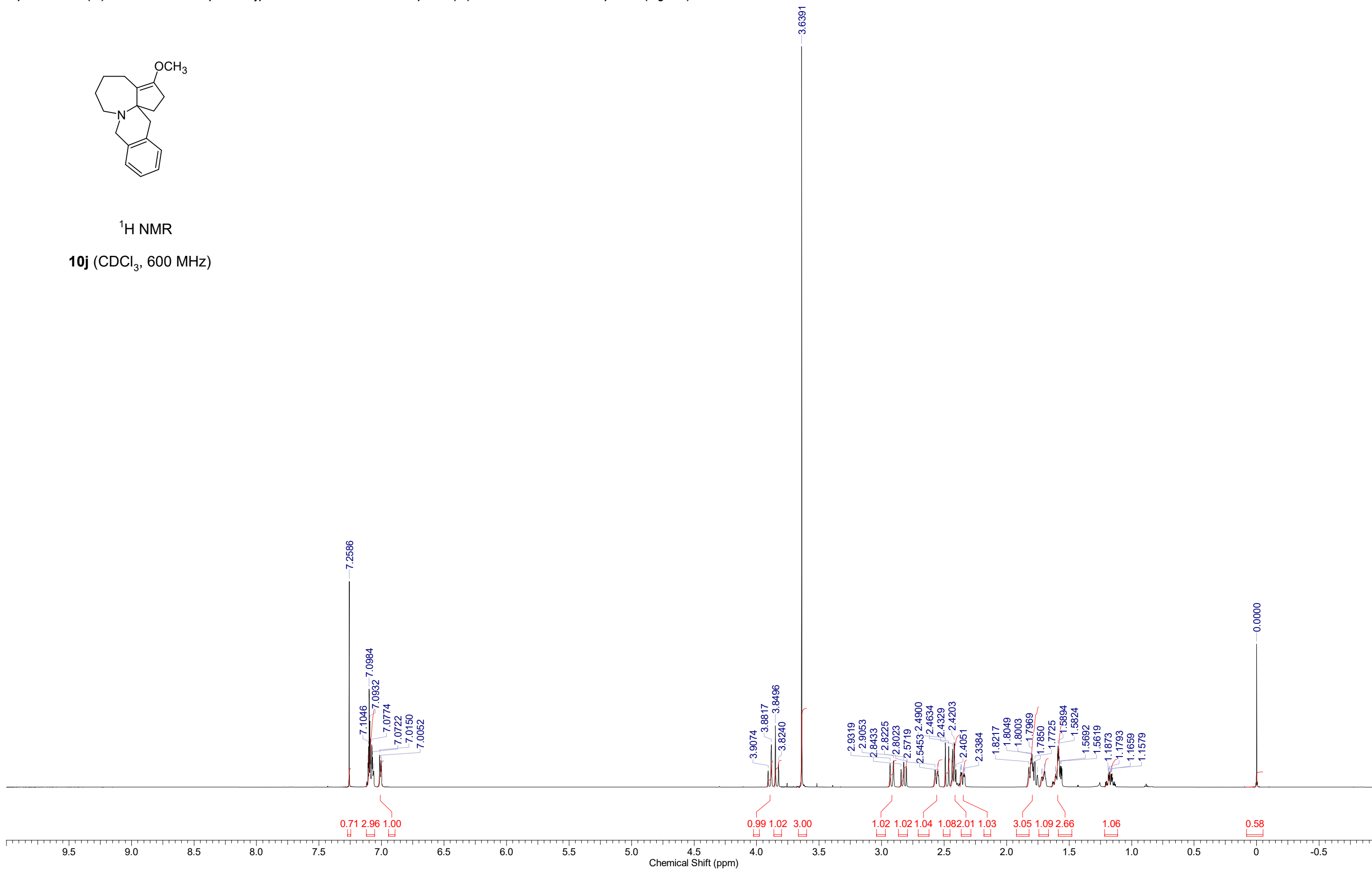


Acquisition Time (sec)	2.7263	Comment	Z126545.0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	Date	20 Nov 2019 18:18:56	Date Stamp	20 Nov 2019 18:18:56
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06115shita_fr3¥1¥pdata¥1¥1r	Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zg30	Receiver Gain	28.41
Spectrum Offset (Hz)	3690.6277	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.155
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

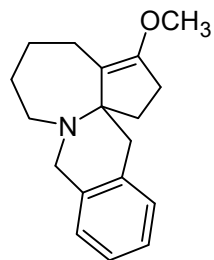


¹H NMR

10j (CDCl₃, 600 MHz)

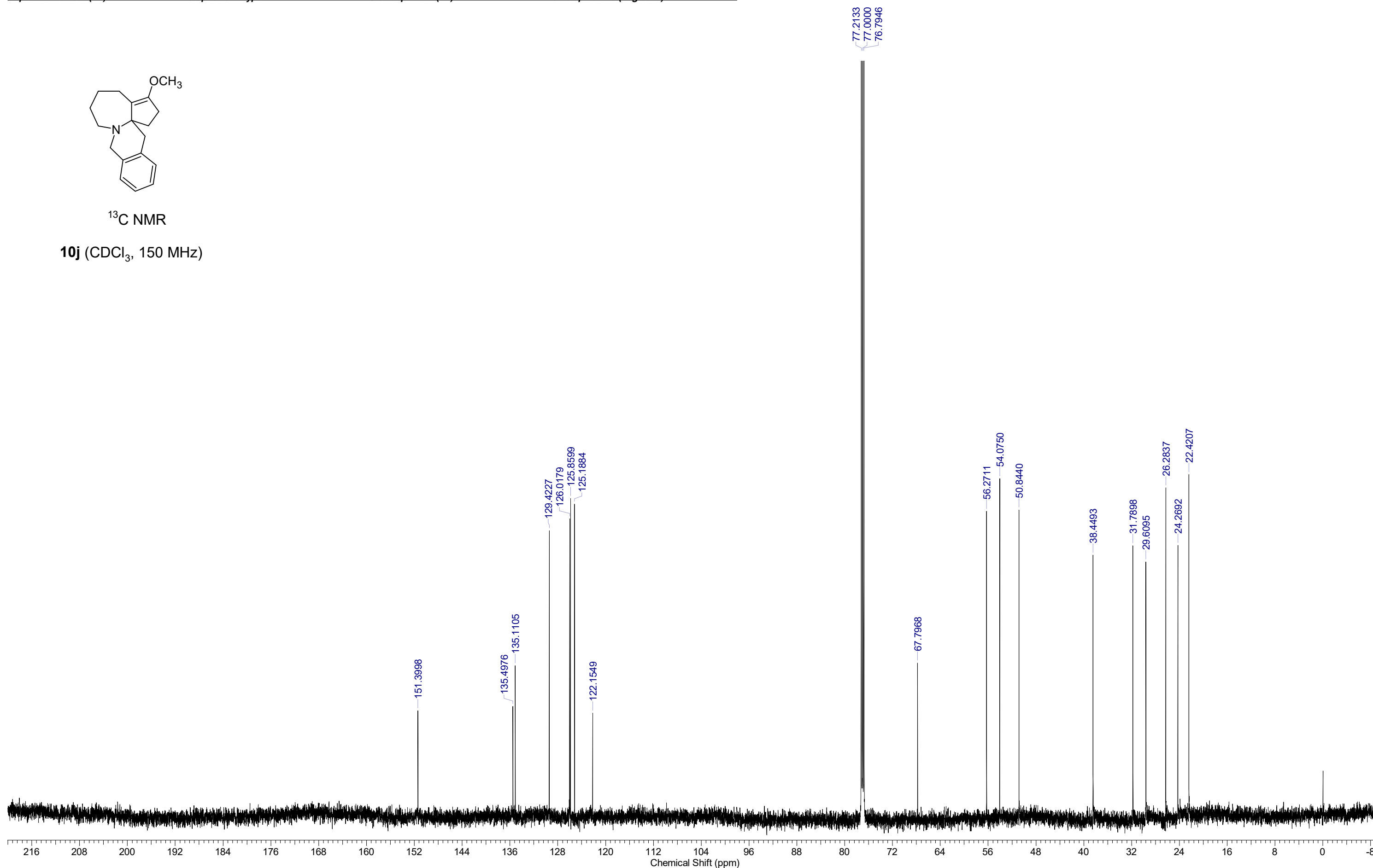


Acquisition Time (sec)	0.8389	Comment	carbon	Date	20 Nov 2019 18:23:12	Date Stamp	20 Nov 2019 18:23:12
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06115shita_fr3¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15086.7188	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.152	Number of Transients	64
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

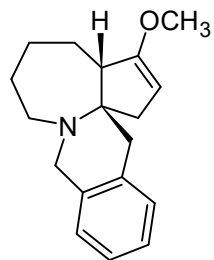


¹³C NMR

10j (CDCl₃, 150 MHz)

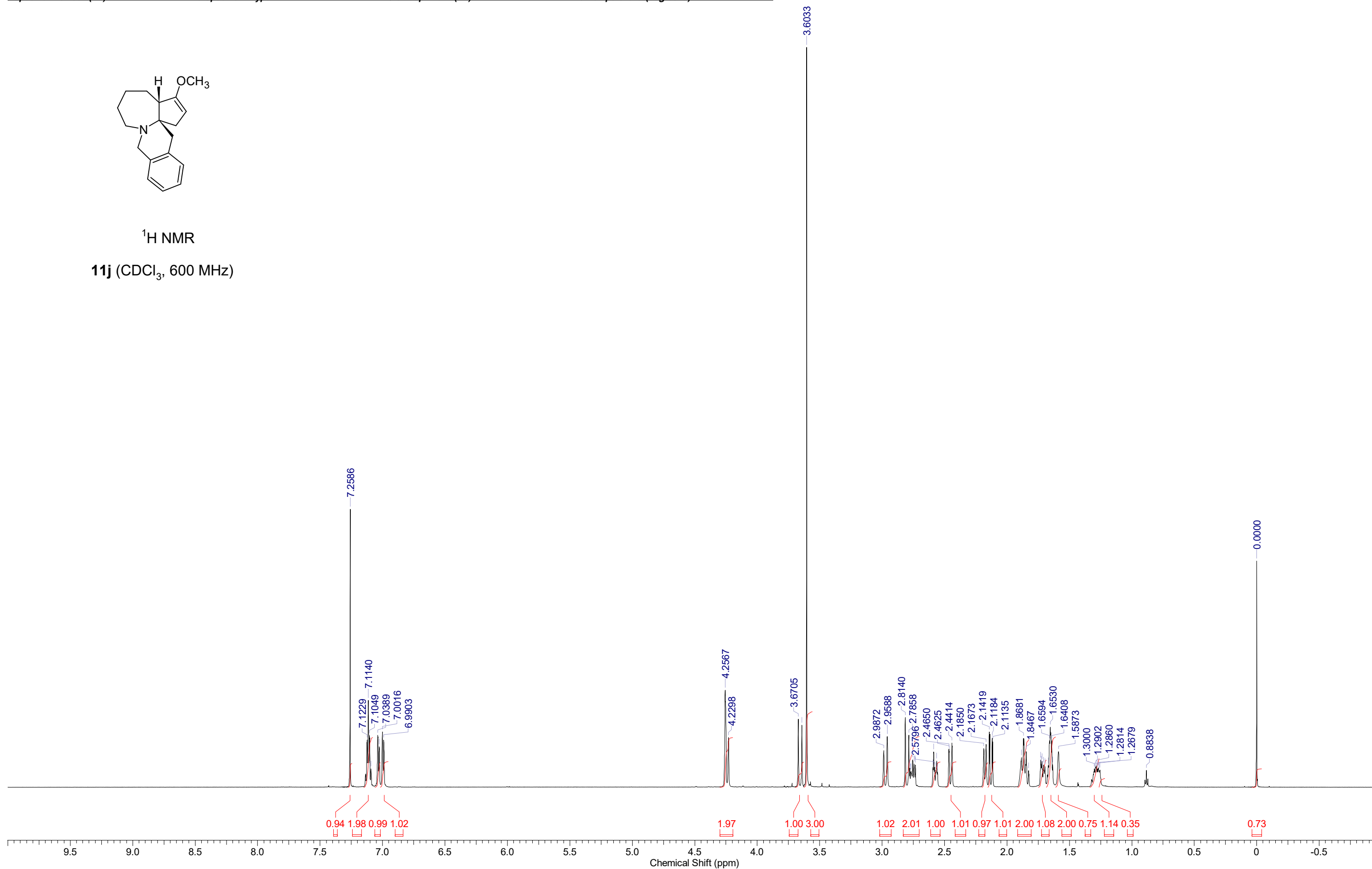


Acquisition Time (sec)	2.7263	Comment	Z126545.0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	Date	20 Nov 2019 18:40:16	Date Stamp	20 Nov 2019 18:40:16
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06115shita_fr4¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3691.1780	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.148	SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

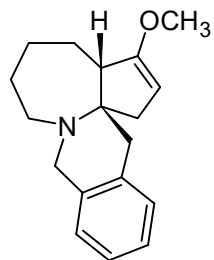


¹H NMR

11j (CDCl₃, 600 MHz)

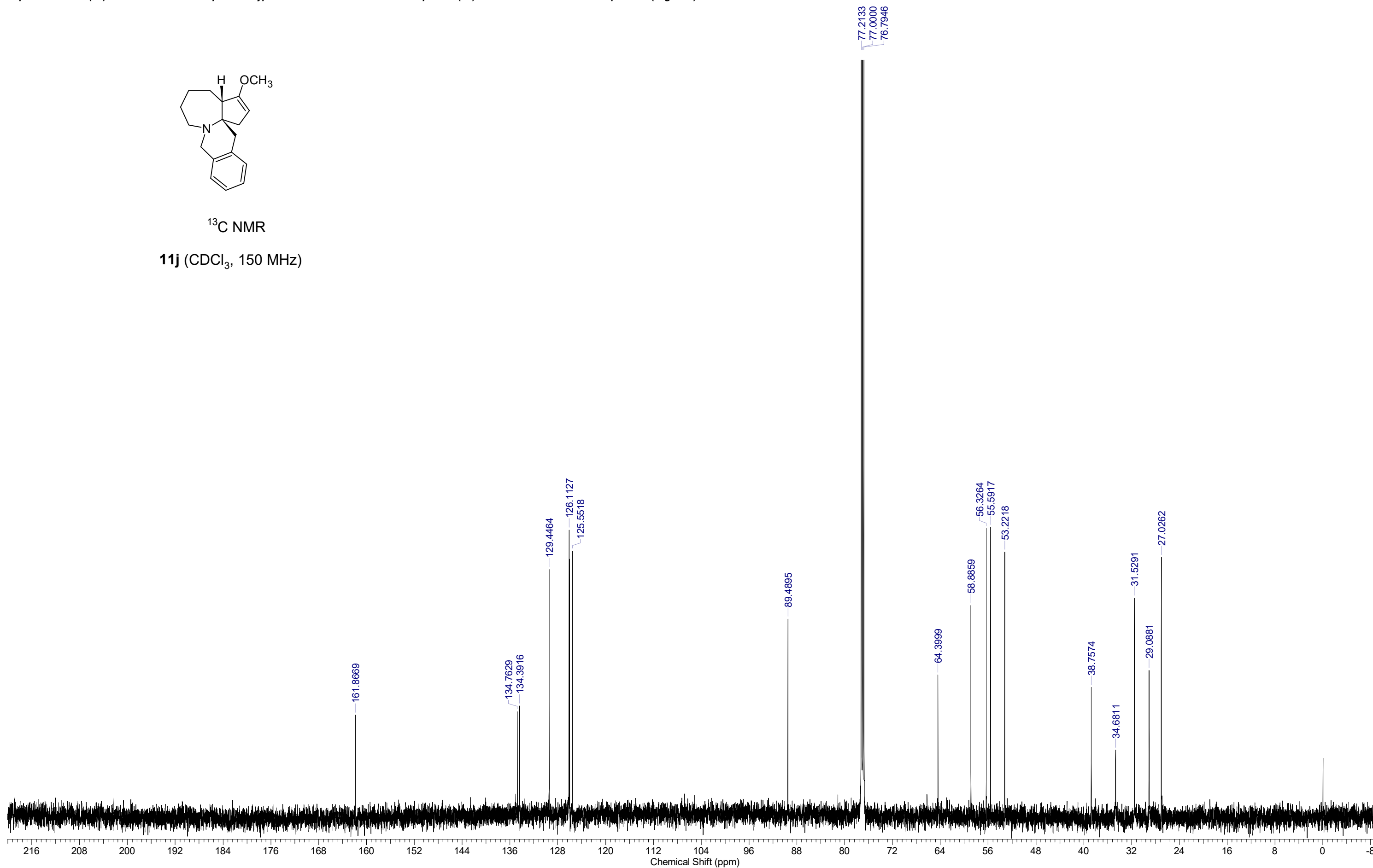


Acquisition Time (sec)	0.8389	Comment	Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	Date	20 Nov 2019 18:44:32	Date Stamp	20 Nov 2019 18:44:32
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai06115shita_fr4¥2¥pdata¥1¥1r	Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	64
Original Points Count	32768	Owner	nmsu	Pulse Sequence	zgpg30	Receiver Gain	193.26
Spectrum Offset (Hz)	15086.7188	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.153
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

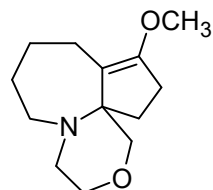


¹³C NMR

11j (CDCl₃, 150 MHz)

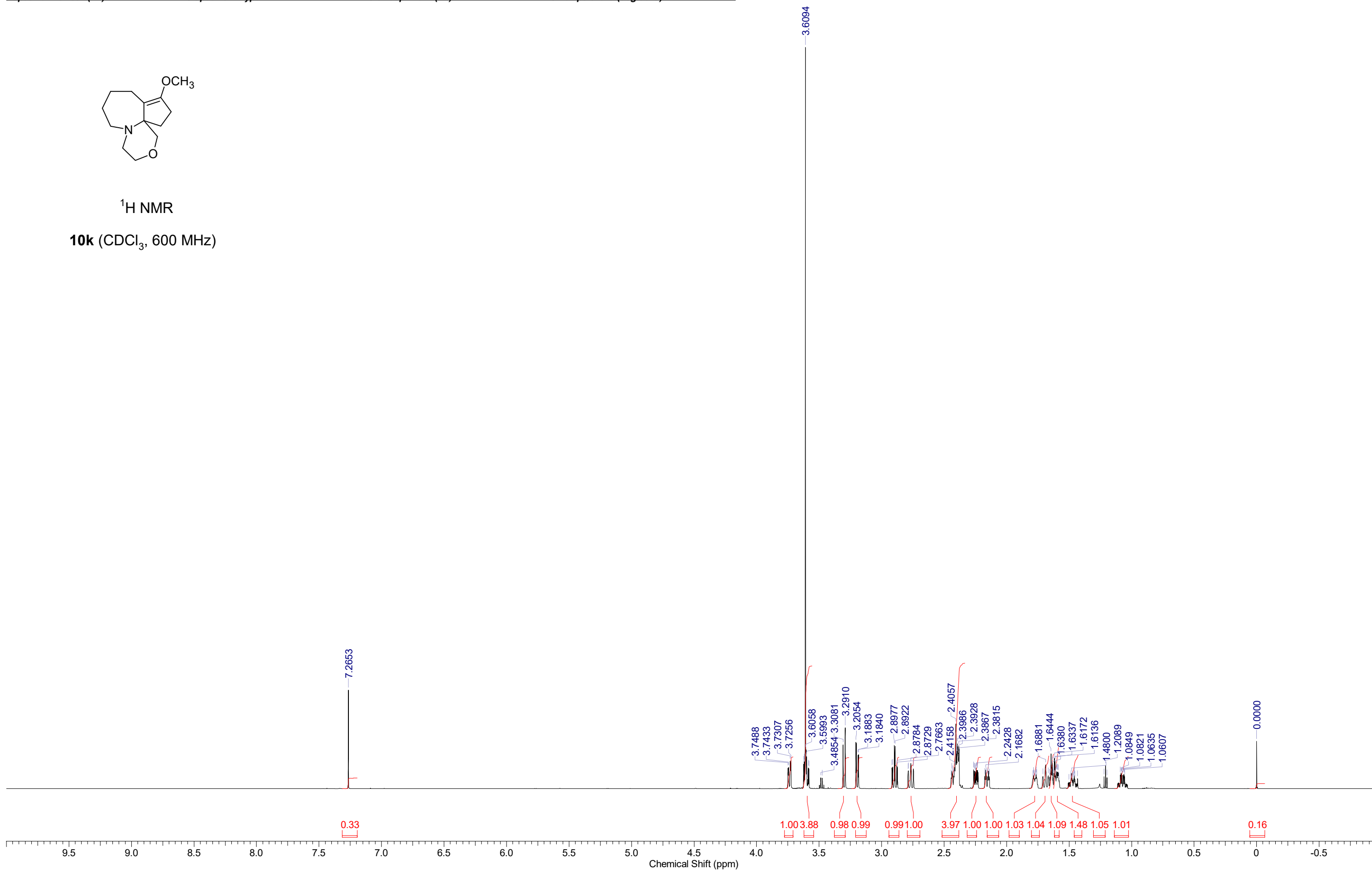


Acquisition Time (sec)	2.7263	Comment	1H	Date	23 Apr 2019 12:03:28	Date Stamp	23 Apr 2019 12:03:28
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01089¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3694.6626	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
				Temperature (degree C)	25.144	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Solvent	CHLOROFORM-d

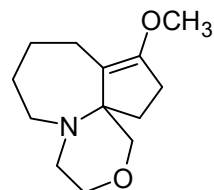


¹H NMR

10k (CDCl₃, 600 MHz)

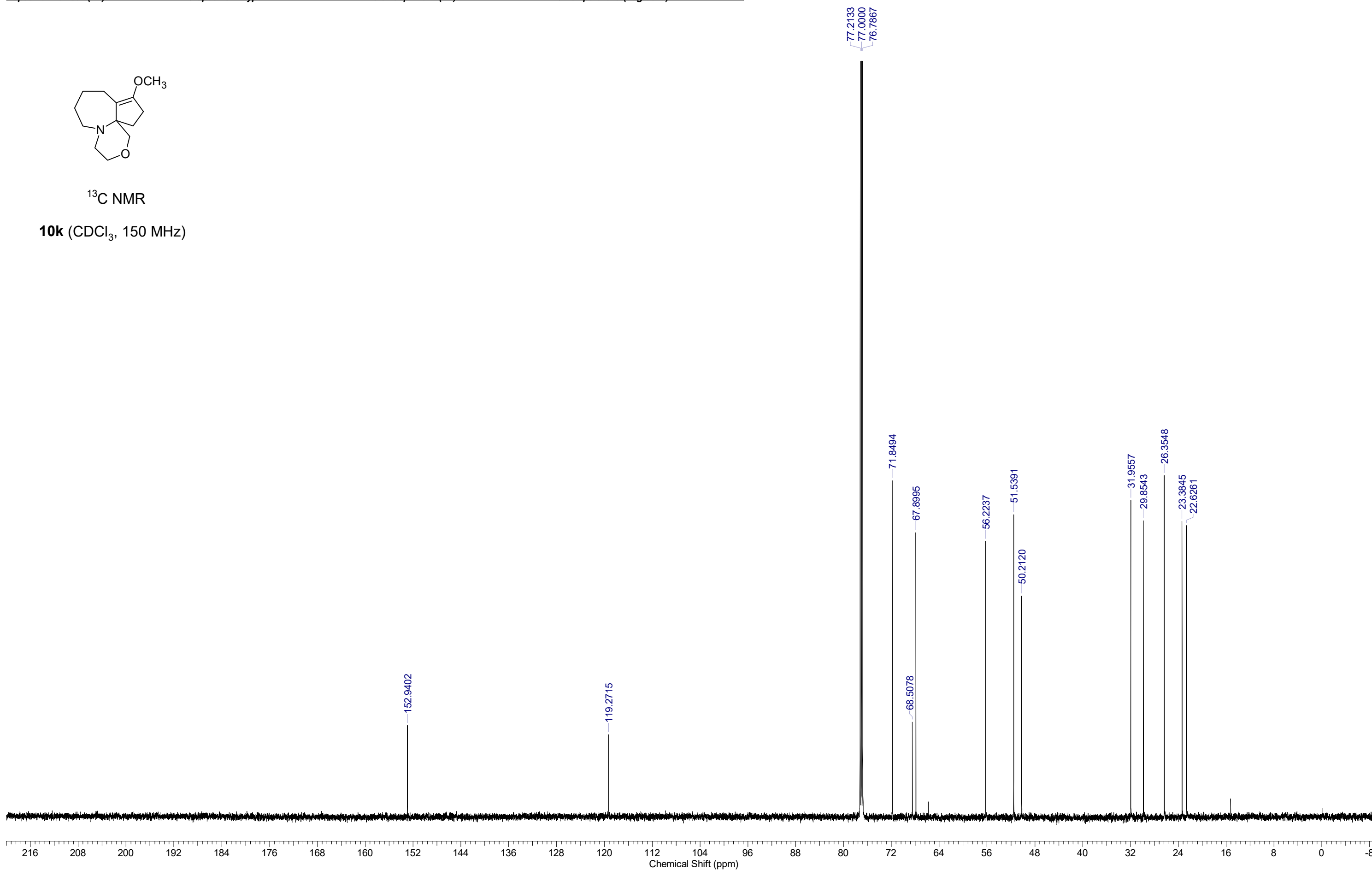


Acquisition Time (sec)	0.8389	Comment	13C	Date	23 Apr 2019 12:07:44	Date Stamp	23 Apr 2019 12:07:44
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01089¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Receiver Gain	193.26
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Pulse Sequence	zgpg30	Number of Transients	64
				Sweep Width (Hz)	39061.31	SW(cyclical) (Hz)	39062.50
				Temperature (degree C)	25.143	Origin	spect
						Solvent	CHLOROFORM-d

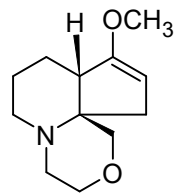


¹³C NMR

10k (CDCl₃, 150 MHz)



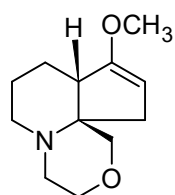
Acquisition Time (sec)	2.7263	Comment	1H	Date	19 Jun 2019 09:10:40	Date Stamp	19 Jun 2019 09:10:40		
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01102-mono¥10¥pdata¥1¥1r		Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8	
Original Points Count	32768	Owner	nrmrsu	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	19.87
Spectrum Offset (Hz)	3703.4656	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.129	SW(cyclical) (Hz)	12019.23
								Origin	spect
								Solvent	CHLOROFORM-d



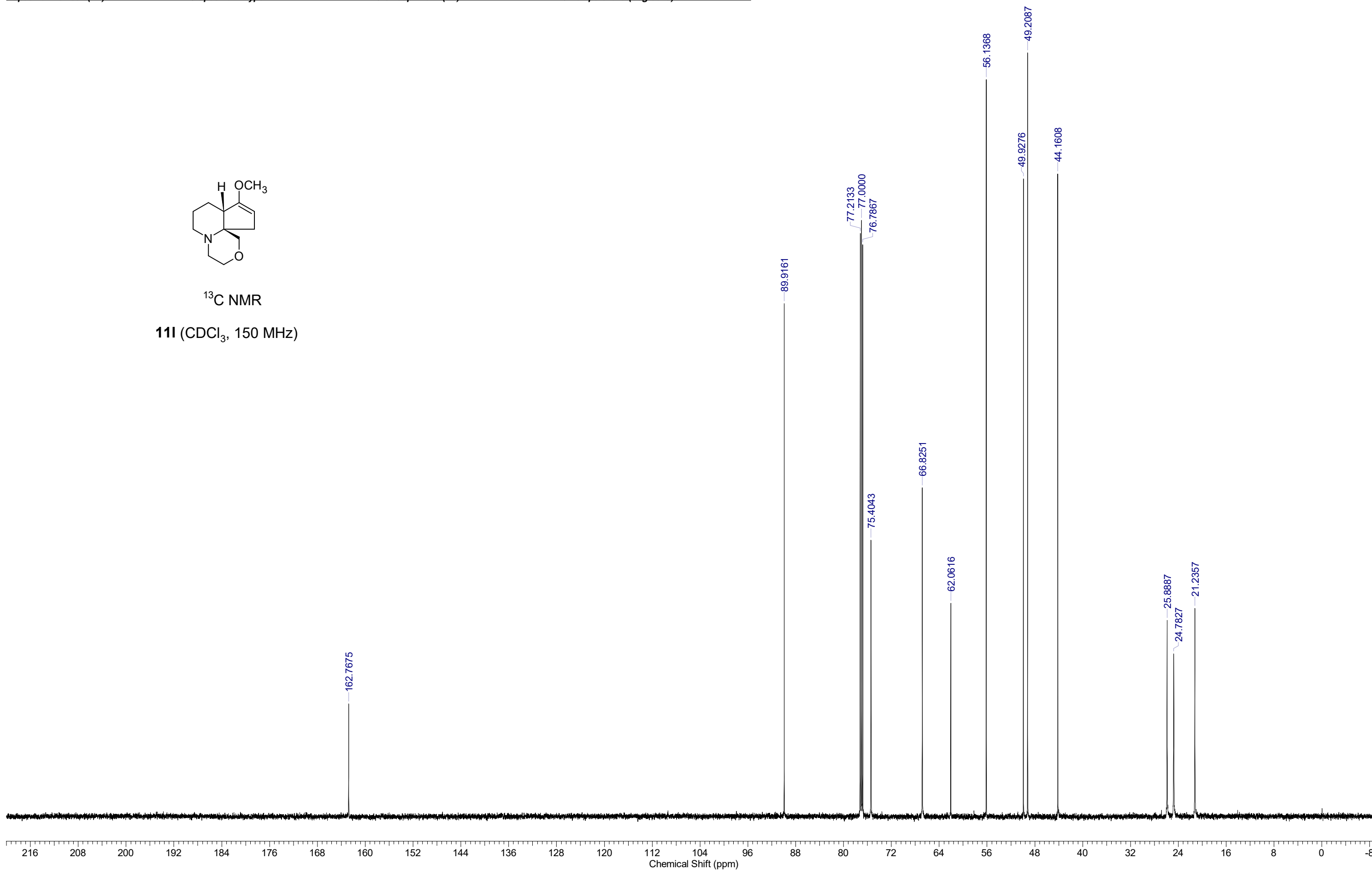
¹H NMR
11I (CDCl₃, 600 MHz)



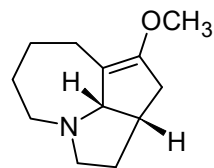
Acquisition Time (sec)	0.8389	Comment	13C	Date	19 Jun 2019 09:14:56	Date Stamp	19 Jun 2019 09:14:56
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2019¥二村将豪¥futamura01102-mono¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Receiver Gain	193.26
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Number of Transients	64
				Temperature (degree C)	25.158	SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d



¹³C NMR
11 (CDCl₃, 150 MHz)

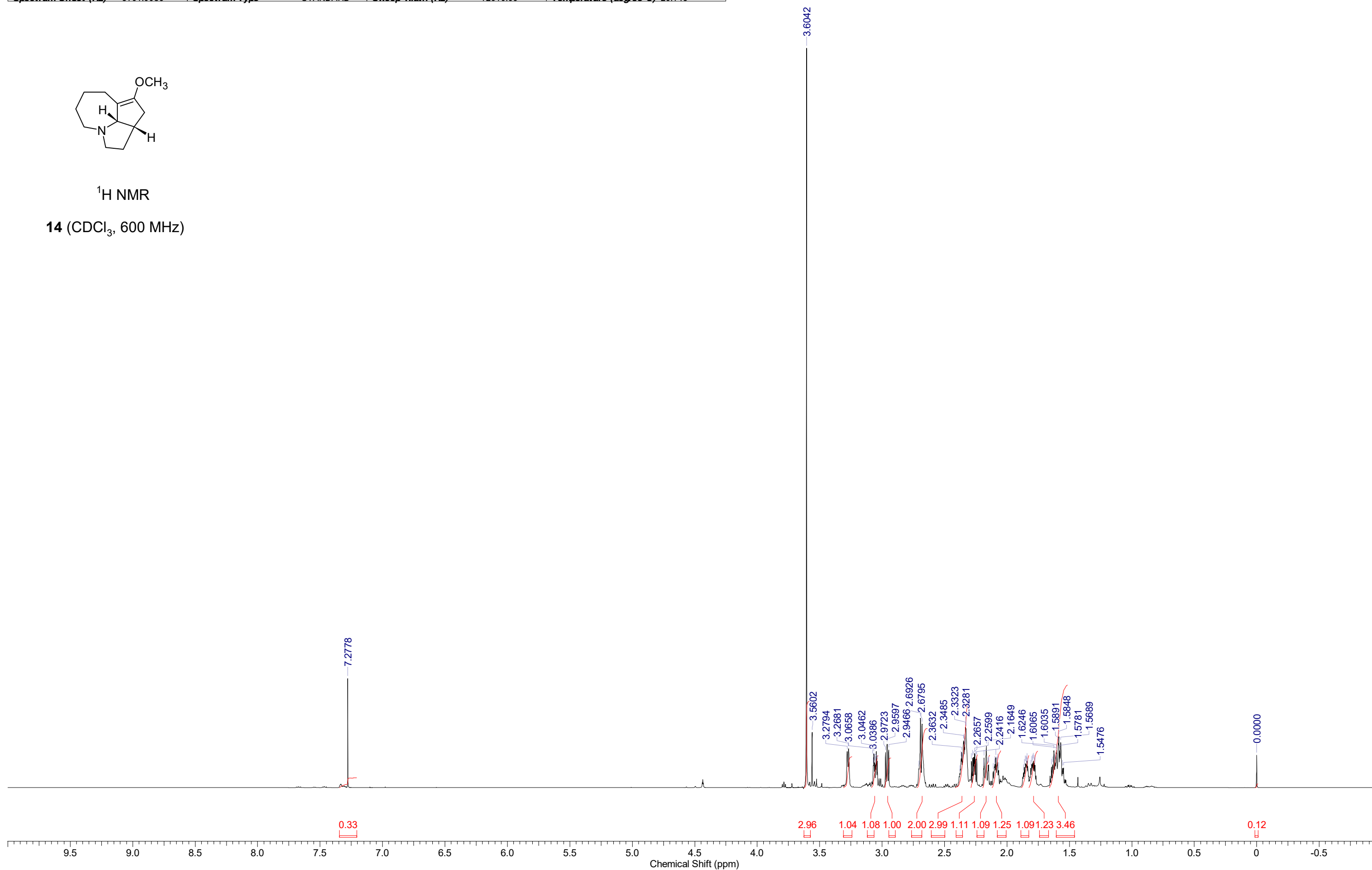


Acquisition Time (sec)	2.7263	Comment	proton	Date	26 Dec 2019 21:28:48	Date Stamp	26 Dec 2019 21:28:48
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07019shita¥1¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nrmrsu	Points Count	65536	Receiver Gain	19.87
Spectrum Offset (Hz)	3701.9985	Spectrum Type	STANDARD	Pulse Sequence	zg30	Number of Transients	8
				Sweep Width (Hz)	12019.05	SW(cyclical) (Hz)	12019.23
				Temperature (degree C)	25.143	Origin	spect
						Solvent	CHLOROFORM-d

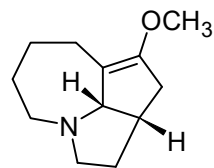


¹H NMR

14 (CDCl₃, 600 MHz)

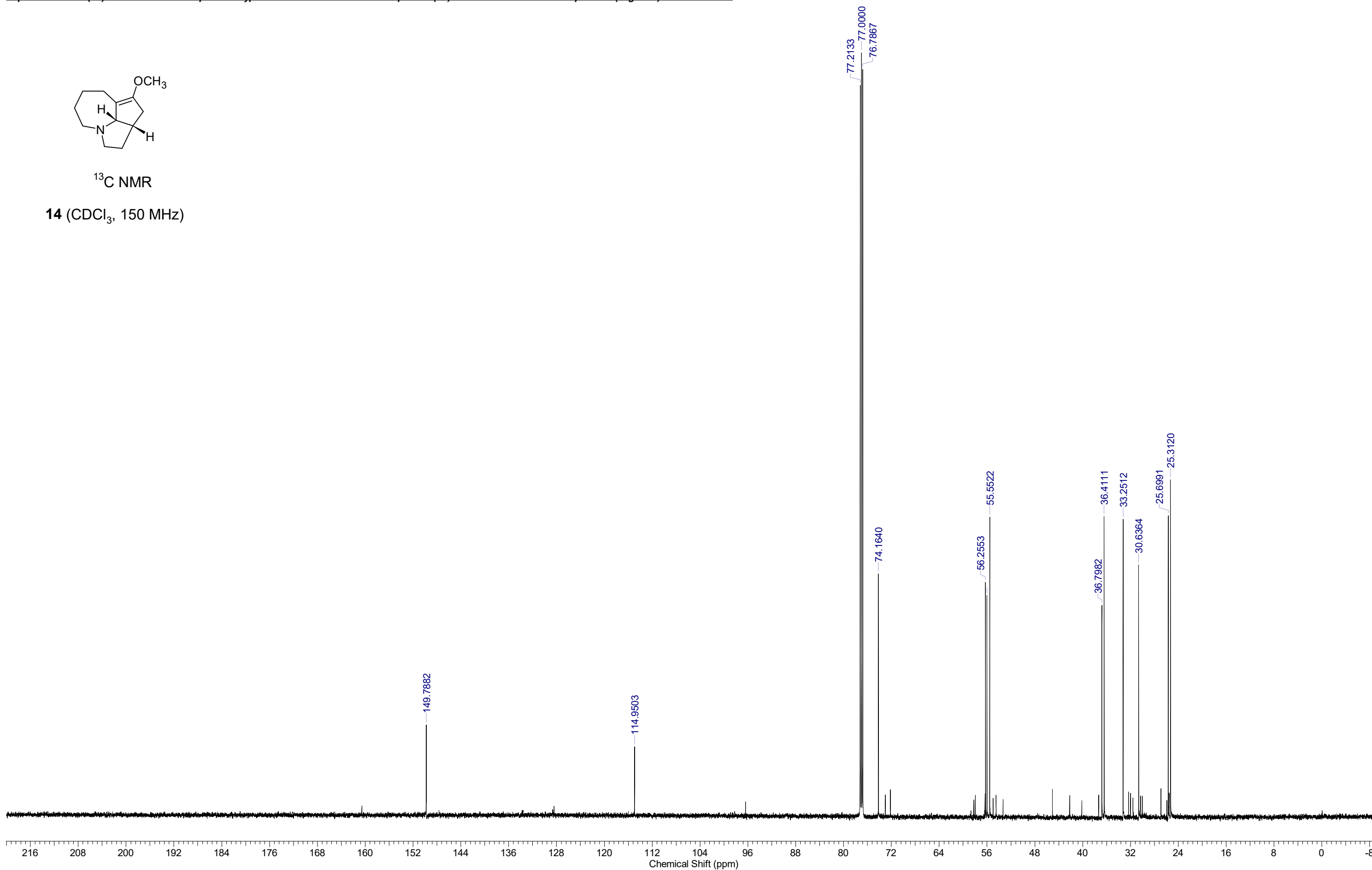


Acquisition Time (sec)	0.8389	Comment	carbon	Date	26 Dec 2019 21:33:04	Date Stamp	26 Dec 2019 21:33:04
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07019shita¥2¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15083.1416	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.147	Number of Transients	65
						SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

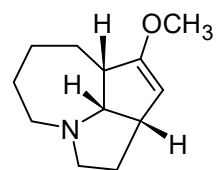


¹³C NMR

14 (CDCl₃, 150 MHz)

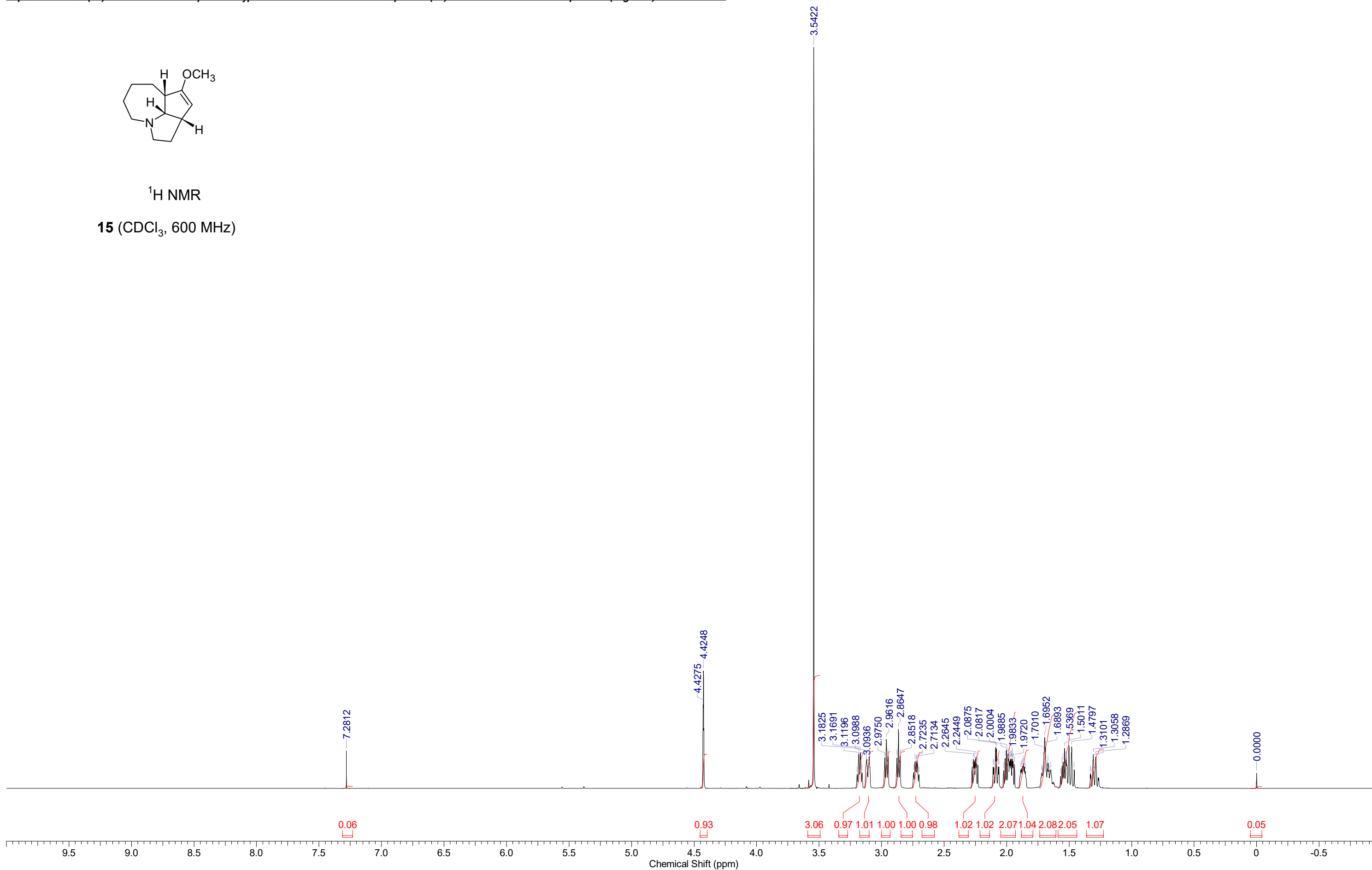


Acquisition Time (sec)	2.7263	Comment	proton	Date	26 Dec 2019 20:31:12	Date Stamp	26 Dec 2019 20:31:12
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07019uefr1¥1¥pdata¥1¥1r	Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	n.mrsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3703.8325	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	19.87
				Temperature (degree C)	25.148	SV(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

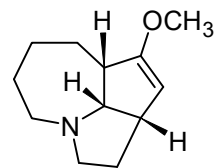


¹H NMR

15 (CDCl₃, 600 MHz)

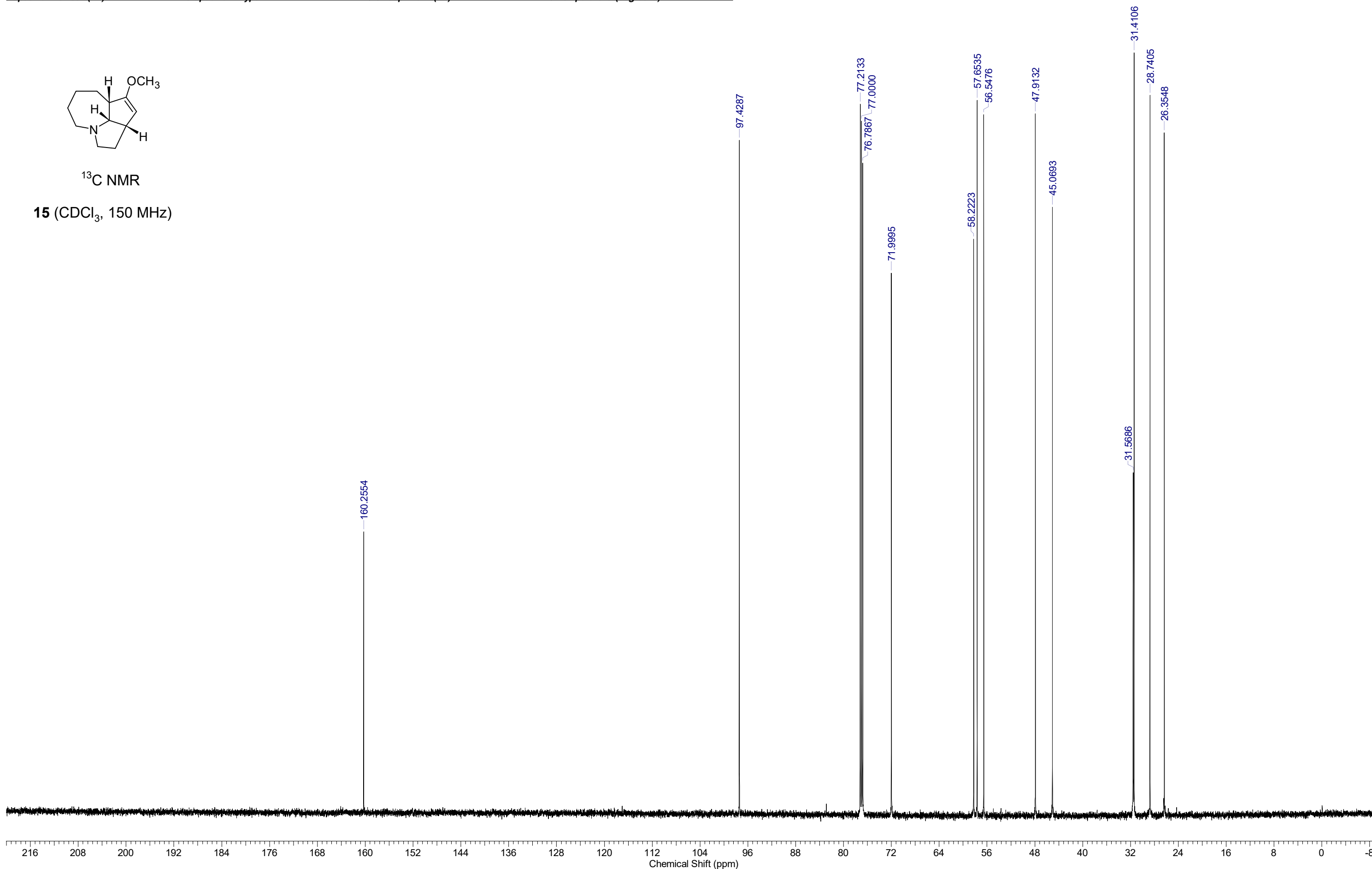


Acquisition Time (sec)	0.8389	Comment	carbon	Date	26 Dec 2019 20:35:28	Date Stamp	26 Dec 2019 20:35:28
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai07019uefr1¥2¥pdata¥1¥1r	Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	64
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15084.3340	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.149	SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

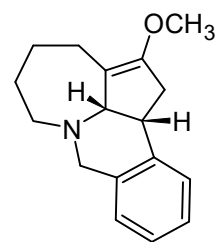


¹³C NMR

15 (CDCl₃, 150 MHz)

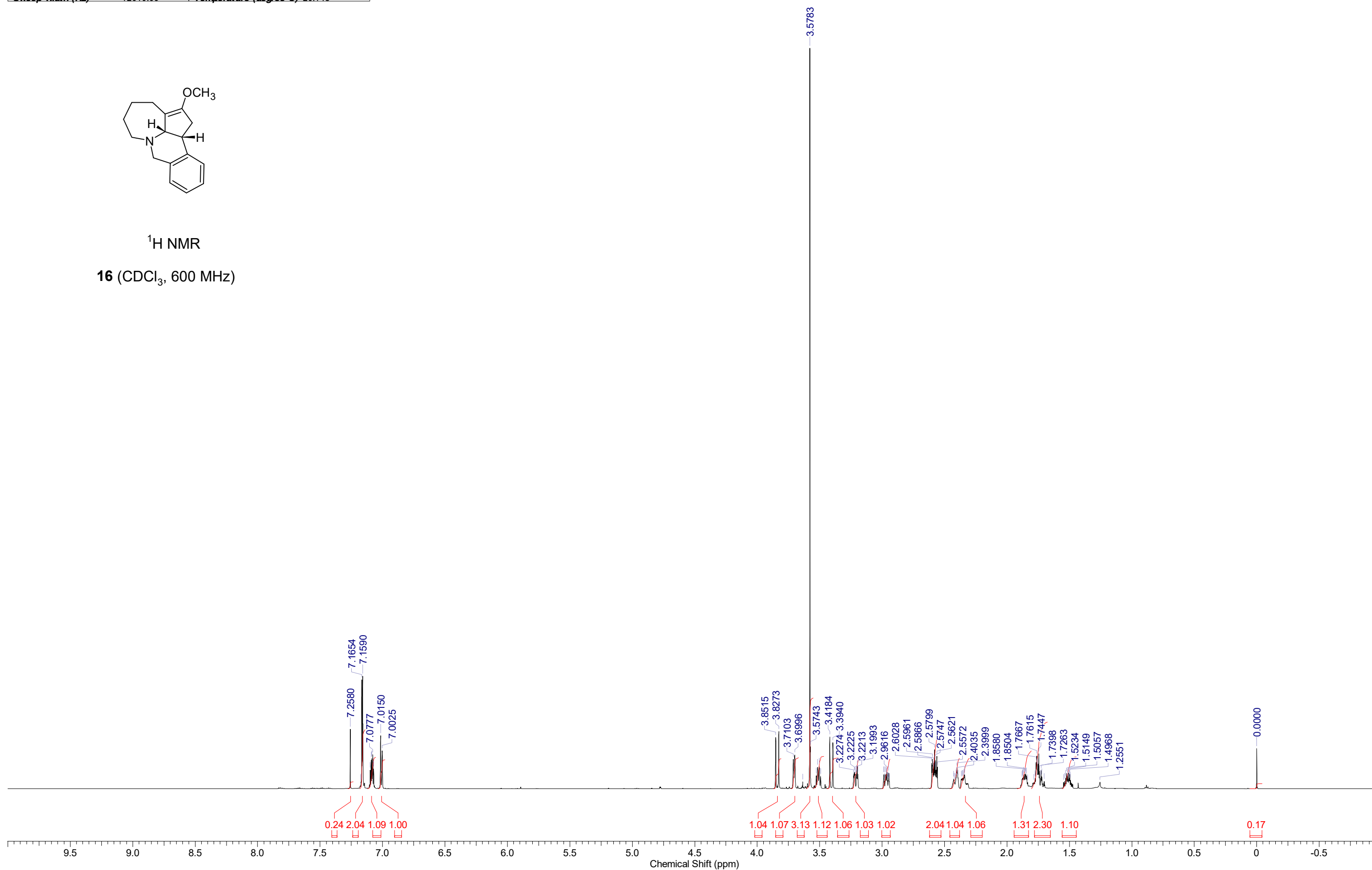


Acquisition Time (sec)	2.7263	Date	14 Nov 2019 20:50:24	Date Stamp	14 Nov 2019 20:50:24	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06113shita¥1¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	25.50	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.148			Spectrum Offset (Hz)	3690.2610
						Points Count	65536
						Spectrum Type	STANDARD

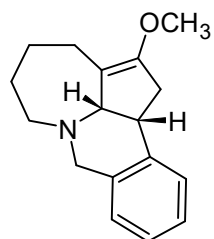


¹H NMR

16 (CDCl₃, 600 MHz)

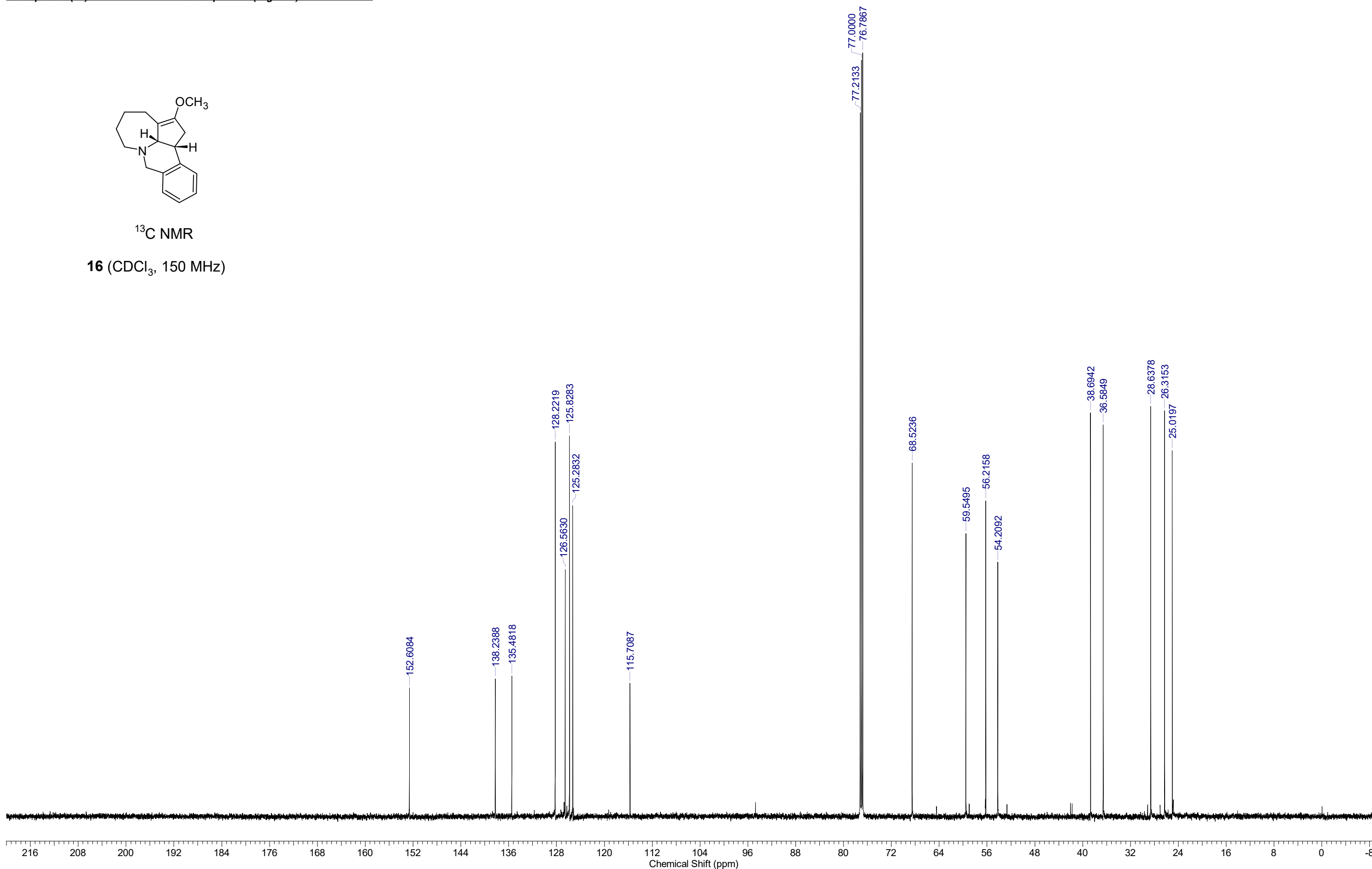


Acquisition Time (sec)	0.8389	Date	14 Nov 2019 20:54:40	Date Stamp	14 Nov 2019 20:54:40	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai06113shita¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Origin	spect	Original Points Count	32768
Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.145			Spectrum Offset (Hz)	15081.9492
						Points Count	32768
						Spectrum Type	STANDARD

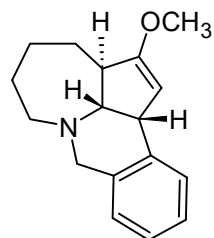


¹³C NMR

16 (CDCl₃, 150 MHz)

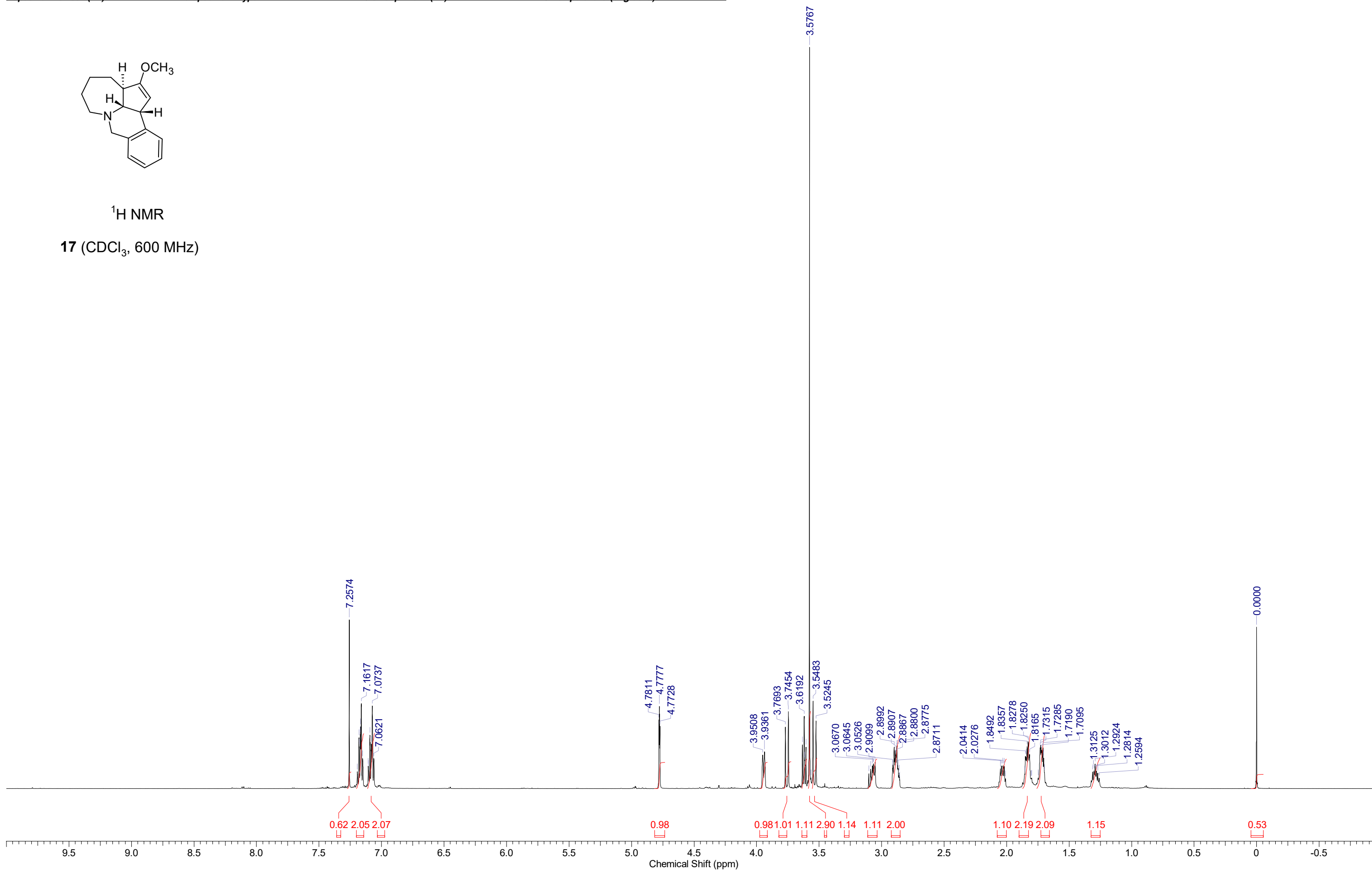


Acquisition Time (sec)	2.7263	Comment	proton	Date	16 Nov 2019 15:34:40	Date Stamp	16 Nov 2019 15:34:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai06114naka¥11¥pdata¥1¥1r	Frequency (MHz)	600.13	Nucleus	1H	Number of Transients	8
Original Points Count	32768	Owner	nrmrsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3690.4443	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.155	SV(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

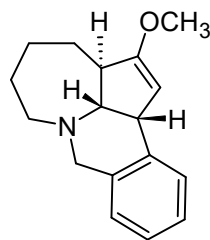


¹H NMR

17 (CDCl₃, 600 MHz)

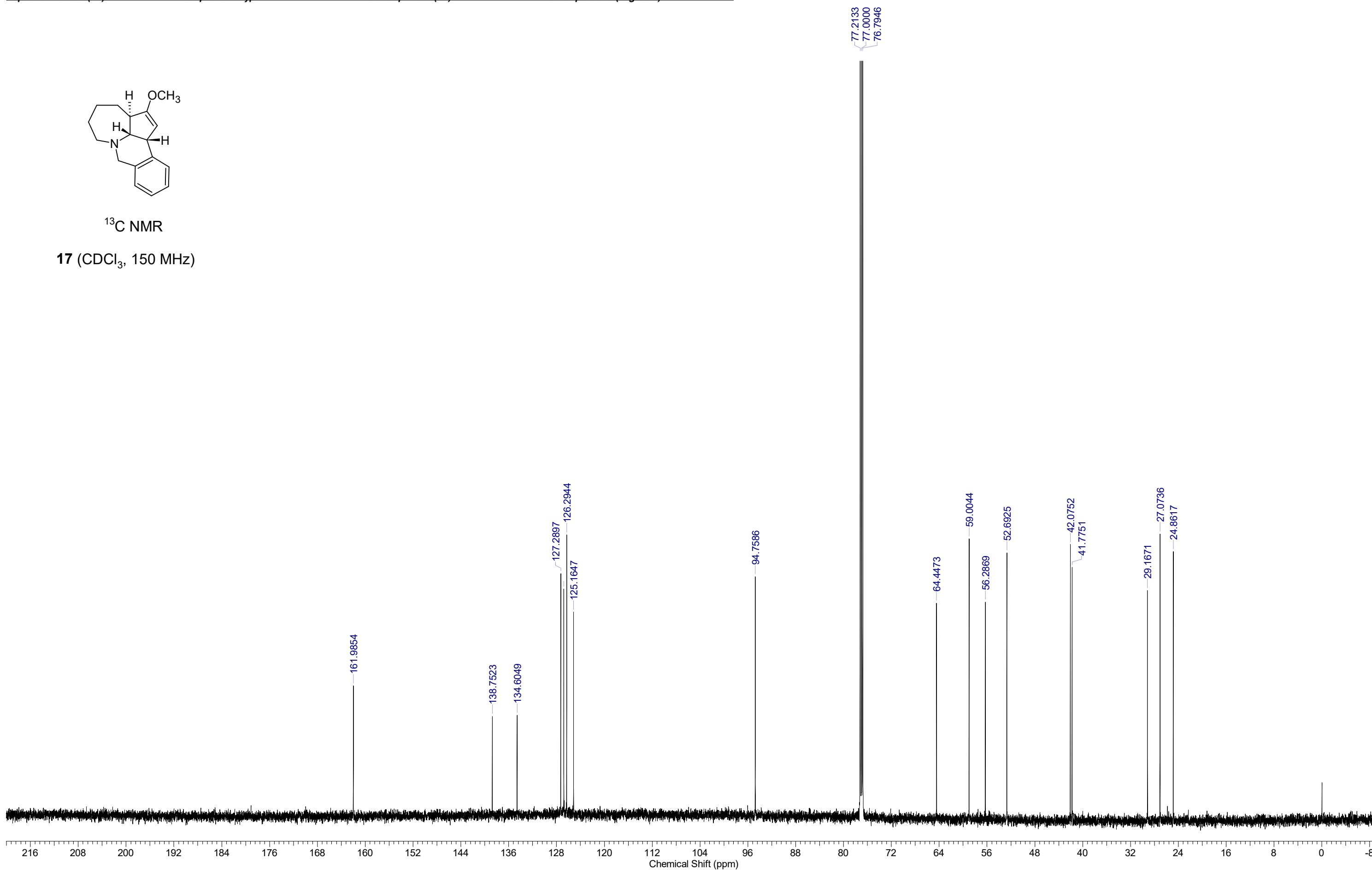


Acquisition Time (sec)	0.8389	Comment	carbon	Date	16 Nov 2019 15:38:56	Date Stamp	16 Nov 2019 15:38:56
File Name	¥¥Mac¥Home¥Desktop¥mori.lab.NMR¥sakaiNMR¥sakai06114naka¥12¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.150	Number of Transients	80
						SV(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

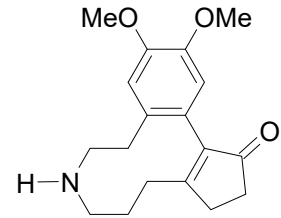
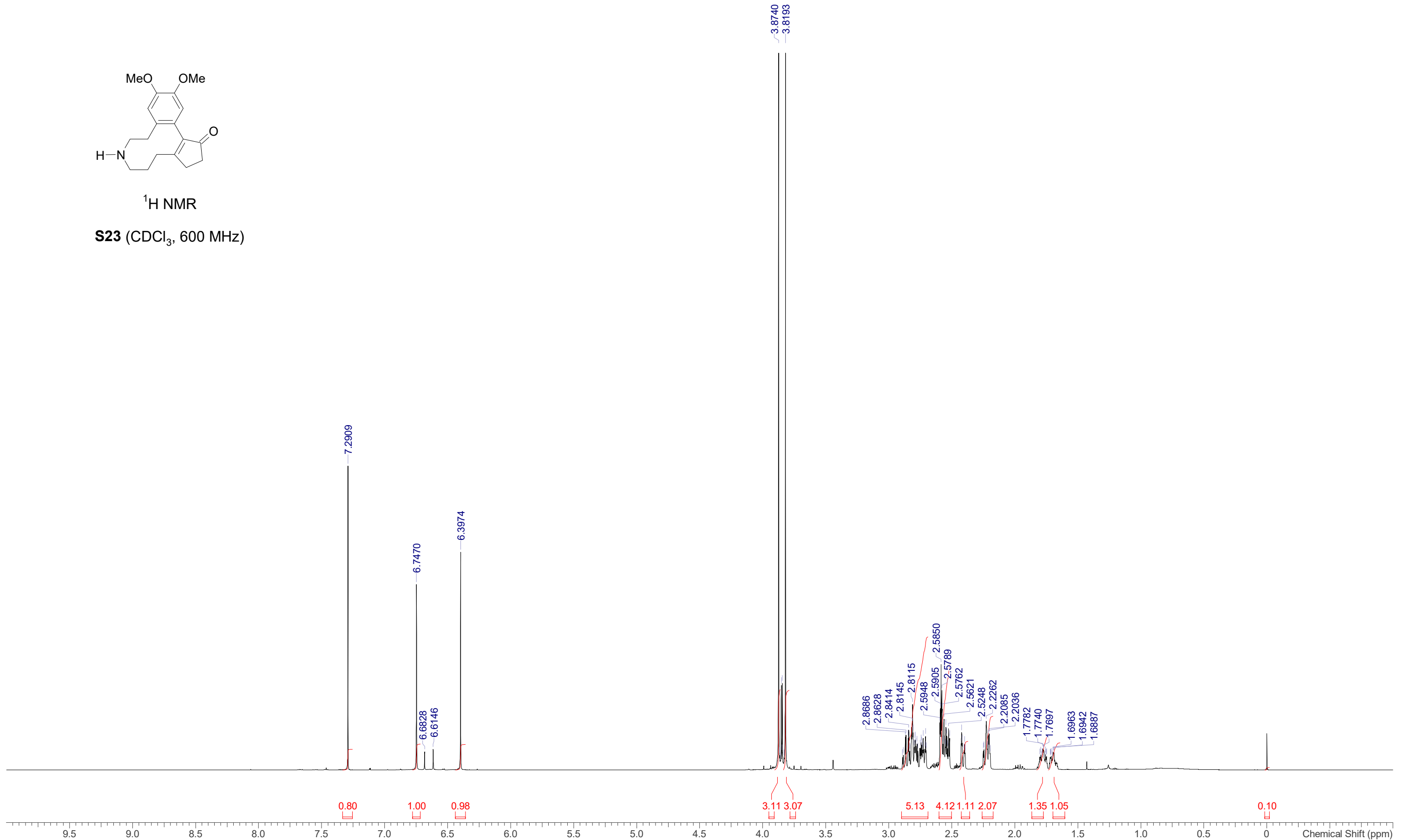


¹³C NMR

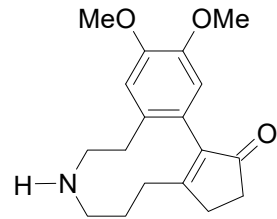
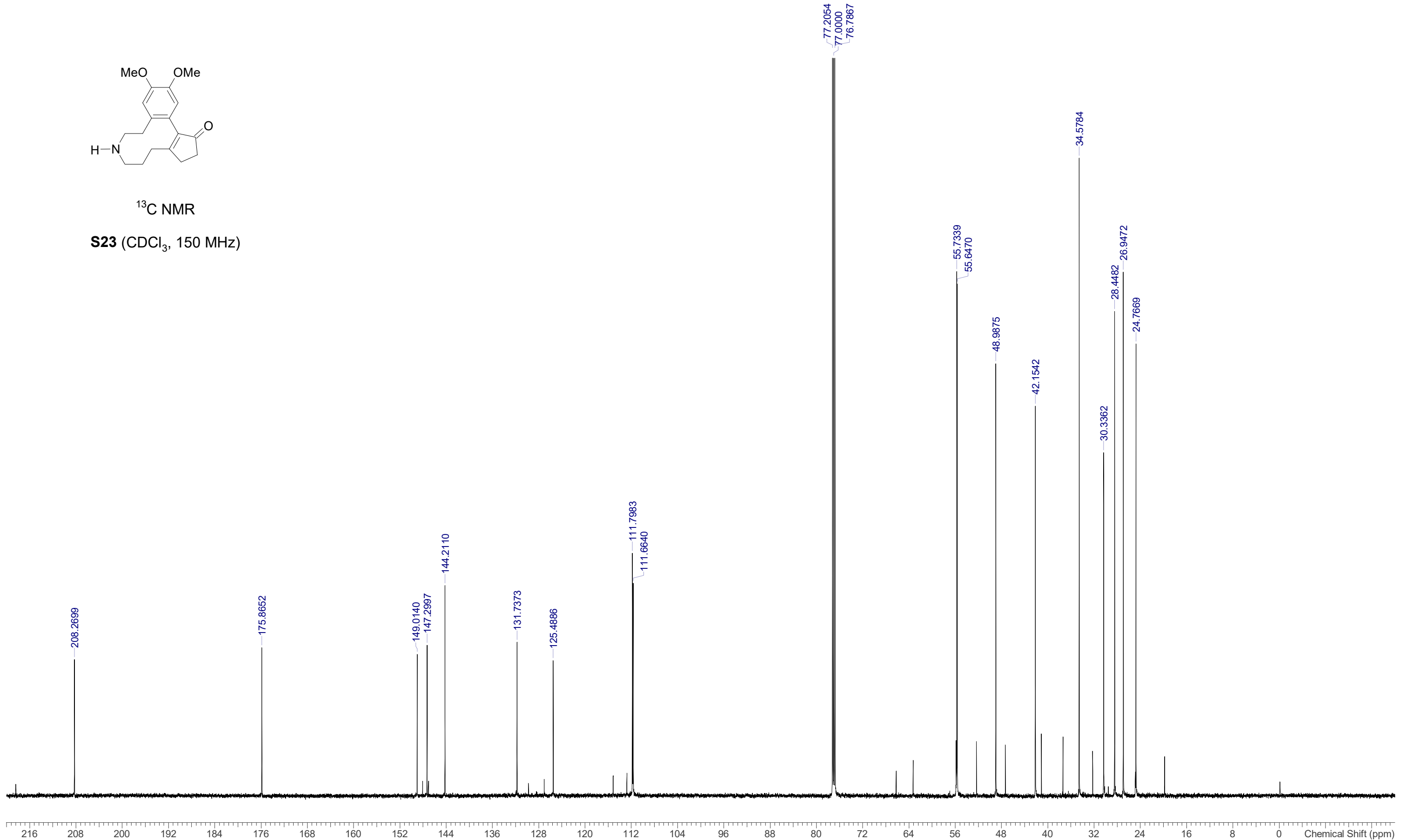
17 (CDCl₃, 150 MHz)



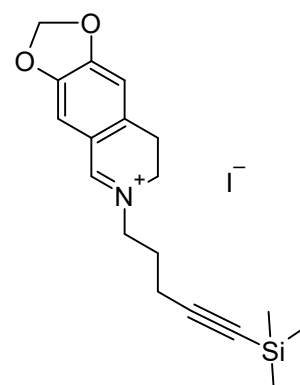
Acquisition Time (sec)	2.7263	Comment	Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	D	0.0002	D1	3	DE	10	DS	0
Date	07 Feb 2019 10:58:44 (GMT+09:00)	Date Stamp	07 Feb 2019 10:58:44 (GMT+09:00)	ExpNo	10	GB	0	INSTRUM	<spect>	LB	0.3
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業生2019¥長江紋加莉¥nagae01052_h¥10¥pdata¥1¥1r	Frequency (MHz)	600.1300	Original Points Count	32768	Owner	nmsu	PC	1	SF	600.129995452049
NS	8	Nucleus	1H	Number of Transients	8	Origin	spect	Pulse Sequence	zg30	Receiver Gain	16.31
PROBHD	<Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zg30>	Points Count	65536	SSB	0	SV(cyclical) (Hz)	12019.23	SWH	12019.2307692308
SFO1	600.133706048414	SI	65536	Spectrum Offset (Hz)	3710.5945	Spectrum Type	standard	Sweep Width (Hz)	12019.05	TD	65536
Solvent	CHLOROFORM-d	UNC1	<1H>	WDW	1						
TE	298.1459	Temperature (degree C)	25.146								

¹H NMRS23 (CDCl₃, 600 MHz)

Acquisition Time (sec)	0.8389	Comment	Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)	D	0.03	D1	2	DE	18	DS	4
Date	07 Feb 2019 11:06:17 (GMT+09:00)	Date Stamp	07 Feb 2019 11:06:17 (GMT+09:00)	ExpNo	10	GB	0	INSTRUM	<spect>	LB	1
File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥卒業生2019¥長江紋加莉¥nagae01052_13c¥10¥pdata¥1¥1r	Frequency (MHz)	150.9028	Original Points Count	32768	Owner	nmrsu	PC	1.4		
NS	128	Nucleus	13C	Number of Transients	128	Origin	spect	Pulse Sequence	zpgpg30	Receiver Gain	193.26
PROBHD	<Z126545_0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zpgpg30>	Points Count	32768	SSB	0	SW(cyclical) (Hz)	39062.50	SWH	39062.5
SFO1	150.917898095859	SI	32768	TD	65536	TDO	1	TE	298.1581	Solvent	CHLOROFORM-d
Spectrum Offset (Hz)	15067.6387	Spectrum Type	standard	Sweep Width (Hz)	39061.31			Temperature (degree C)	25.158		
UNC1	<13C>	WDW	1								

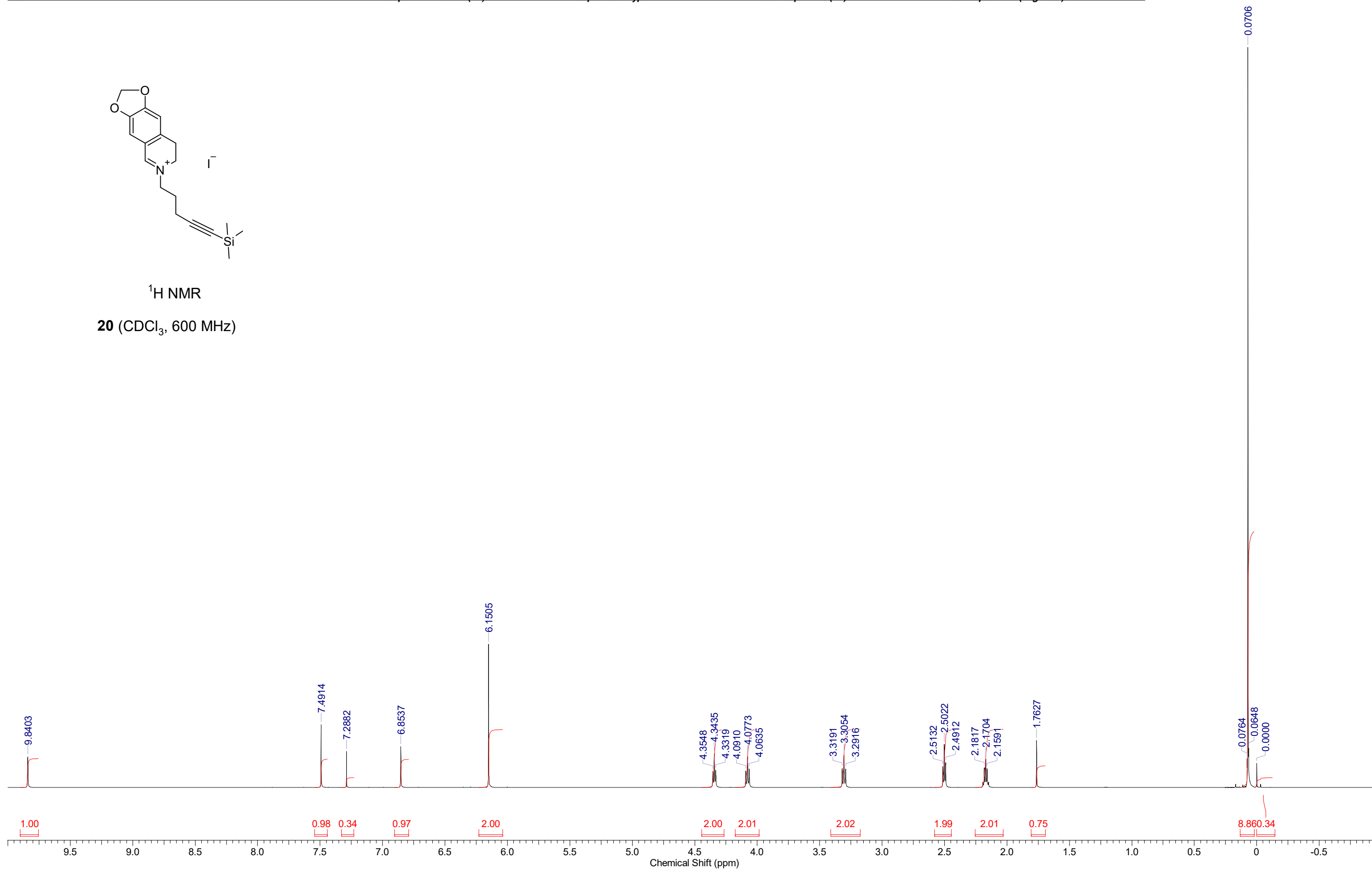
¹³C NMRS23 (CDCl₃, 150 MHz)

Acquisition Time (sec)	2.7263	Date	23 Jun 2020 13:54:24	Date Stamp	23 Jun 2020 13:54:24	Frequency (MHz)	600.13	Nucleus	¹ H	Number of Transients	8
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01069¥okumura01069¥1H¥pdata¥1¥1r			Points Count	65536	Pulse Sequence	zg30	Receiver Gain	25.50	SW(cyclical) (Hz)	12019.23
Origin	spect	Original Points Count	32768	Owner	nmsu	Spectrum Offset (Hz)	3707.8672	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3707.8672	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Temperature (degree C)	25.147		

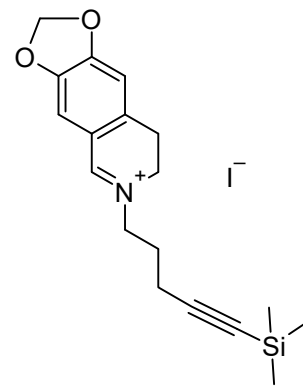


¹H NMR

20 (CDCl₃, 600 MHz)

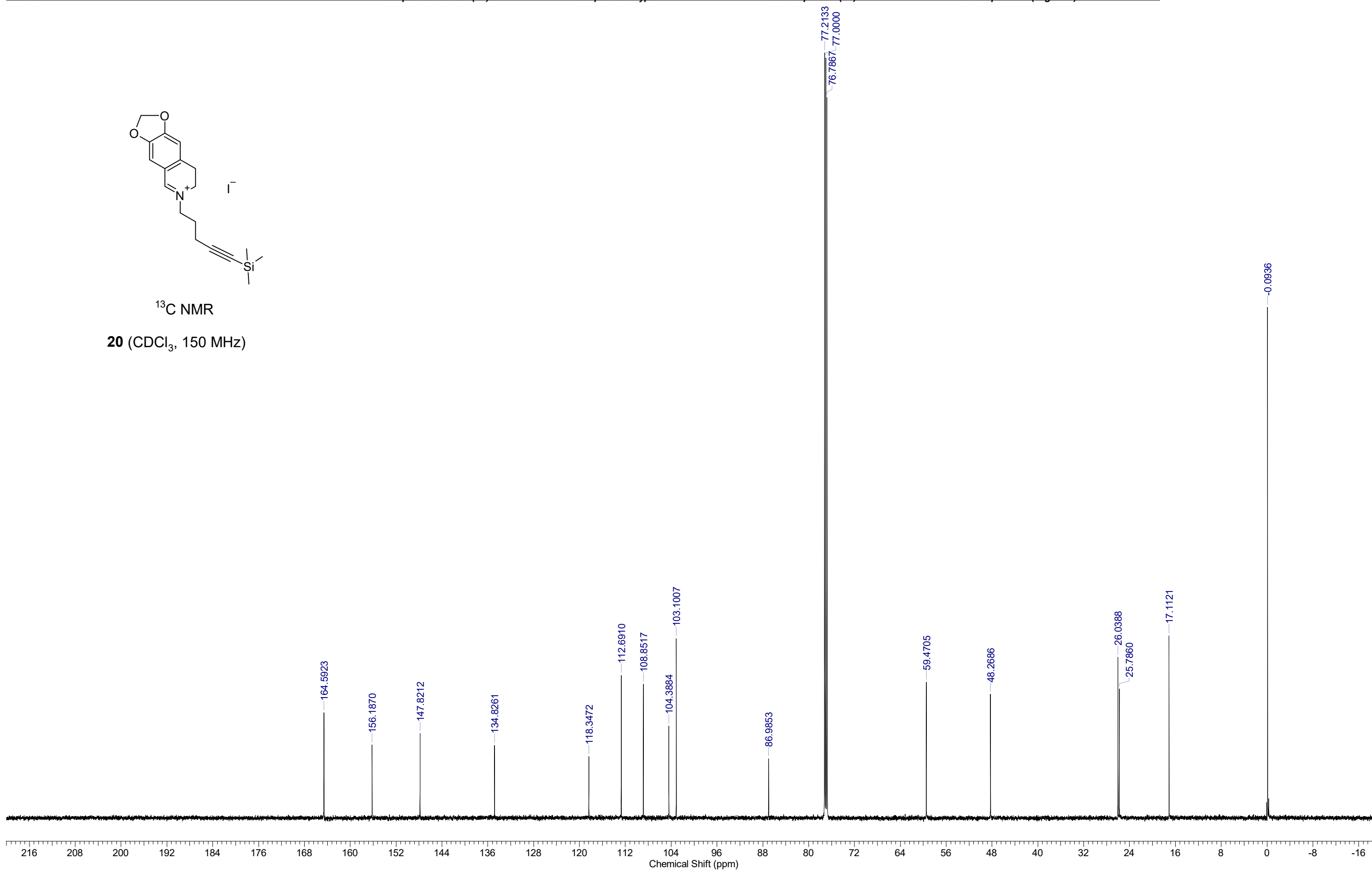


Acquisition Time (sec)	0.8389	Date	23 Jun 2020 14:00:48	Date Stamp	23 Jun 2020 14:00:48	Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	128		
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥卒業予定2020¥奥村知世¥奥村NMR¥01069¥okumura01069¥13C¥pdata¥1¥1r			Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50		
Origin	spect	Original Points Count	32768	Owner	nrmrsu	Spectrum Offset (Hz)	15077.1807	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Temperature (degree C)	25.143
Solvent	CHLOROFORM-d												

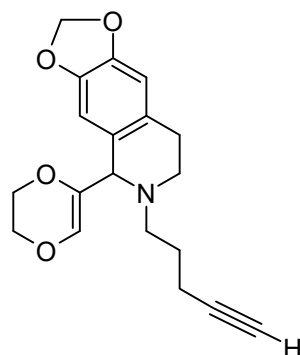


¹³C NMR

20 (CDCl₃, 150 MHz)

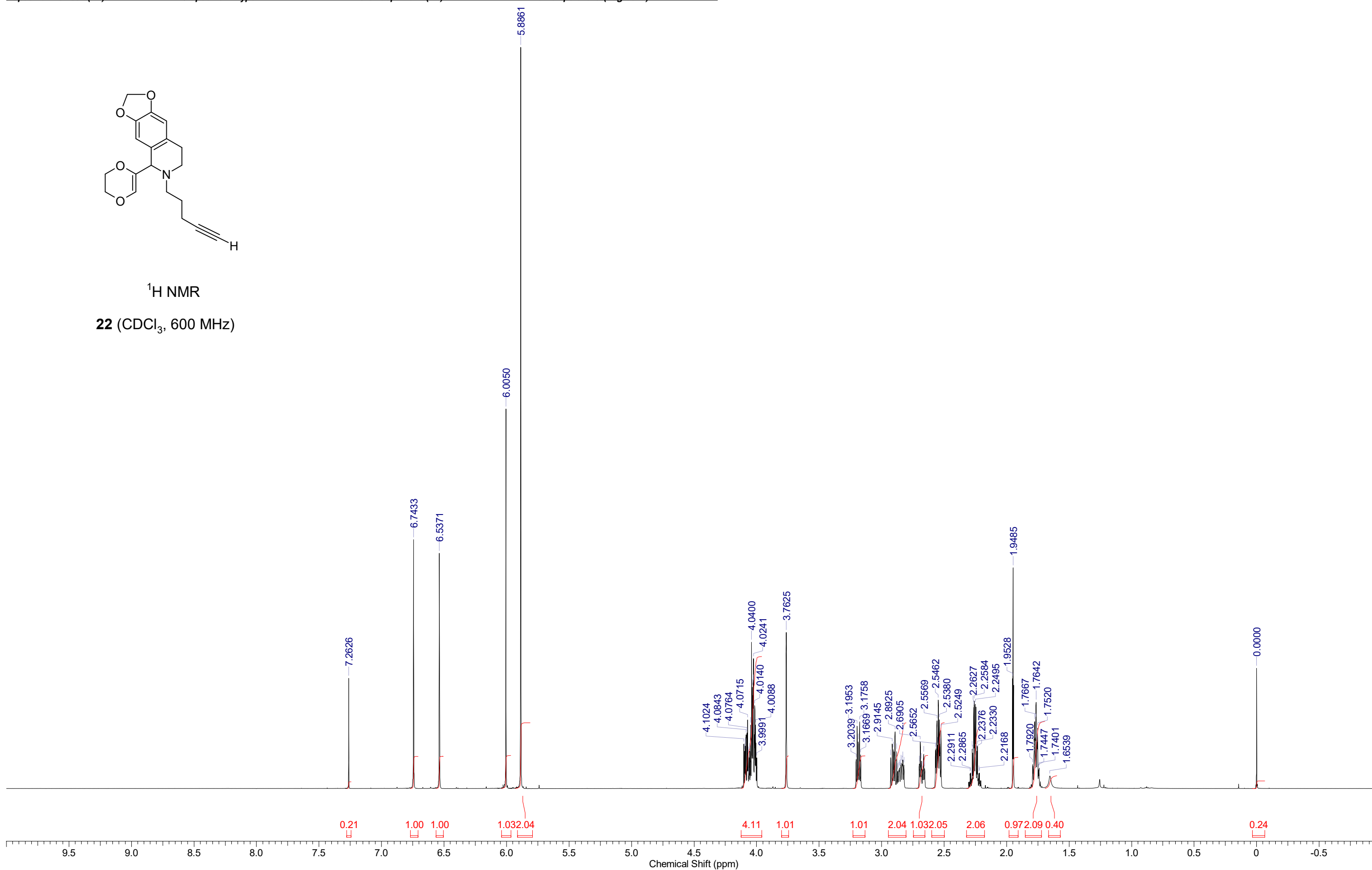


Acquisition Time (sec)	2.7263	Comment	1H	Date	07 Oct 2020 09:40:32	Date Stamp	07 Oct 2020 09:40:32
File Name	¥¥Mac¥Home¥Desktop¥mori.lab.NMR¥sakai.NMR¥sakai07091¥10¥pdata¥1¥1r			Frequency (MHz)	600.13	Nucleus	1H
Original Points Count	32768	Owner	nmsu	Points Count	65536	Pulse Sequence	zg30
Spectrum Offset (Hz)	3693.0120	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	30.91
				Temperature (degree C)	25.156	Number of Transients	8
						SW(cyclical) (Hz)	12019.23
						Origin	spect
						Solvent	CHLOROFORM-d

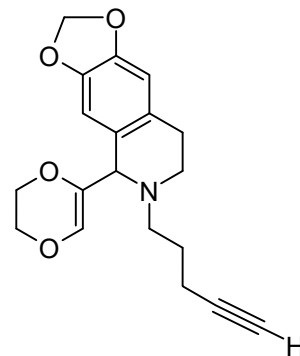


¹H NMR

22 (CDCl₃, 600 MHz)

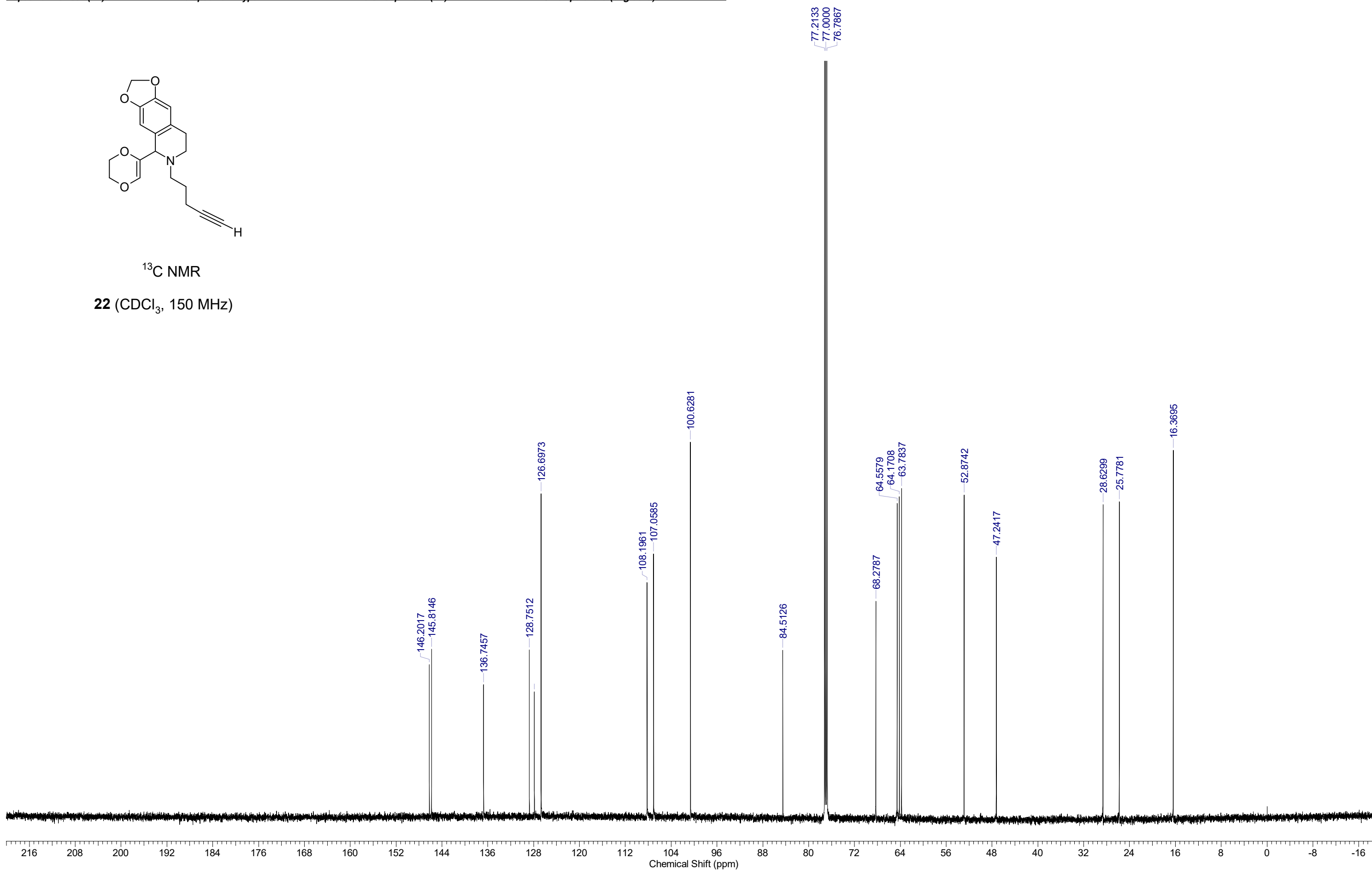


Acquisition Time (sec)	0.8389	Comment	13C	Date	07 Oct 2020 09:42:40	Date Stamp	07 Oct 2020 09:42:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai07091¥11¥pdata¥1¥1r			Frequency (MHz)	150.90	Nucleus	13C
Original Points Count	32768	Owner	nmsu	Points Count	32768	Pulse Sequence	zgpg30
Spectrum Offset (Hz)	15083.1416	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.152	Number of Transients	64
						SV(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

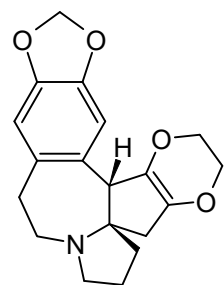


¹³C NMR

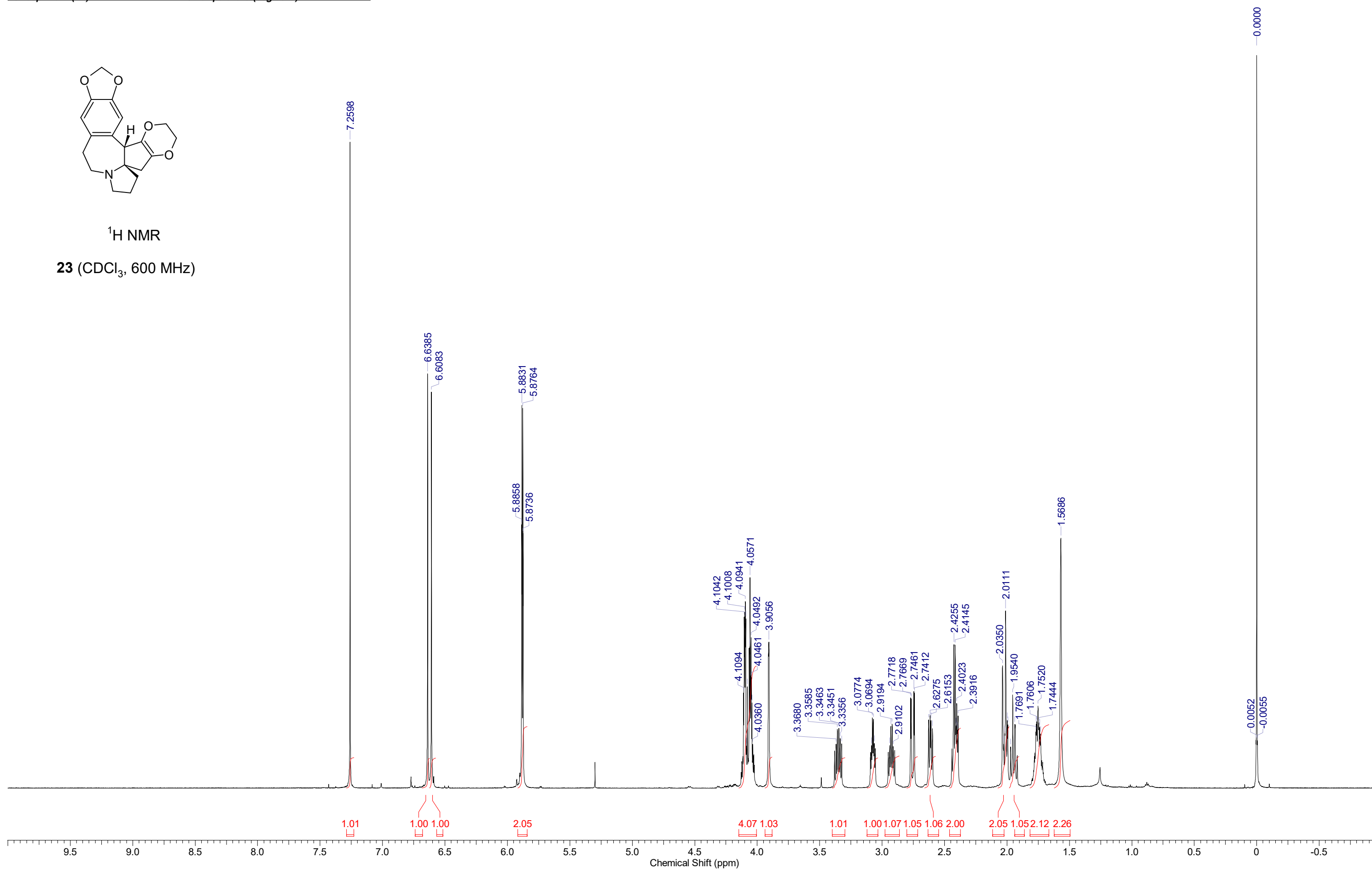
22 (CDCl₃, 150 MHz)



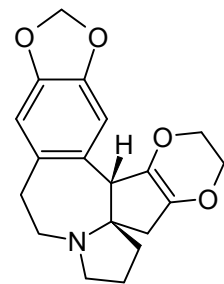
Acquisition Time (sec)	2.7263	Date	09 Oct 2020 17:21:20	Date Stamp	09 Oct 2020 17:21:20	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07093ueue¥10¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	30.91	SW(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.154			Spectrum Offset (Hz)	3691.3613
						Points Count	65536
						Spectrum Type	STANDARD



¹H NMR
23 (CDCl₃, 600 MHz)

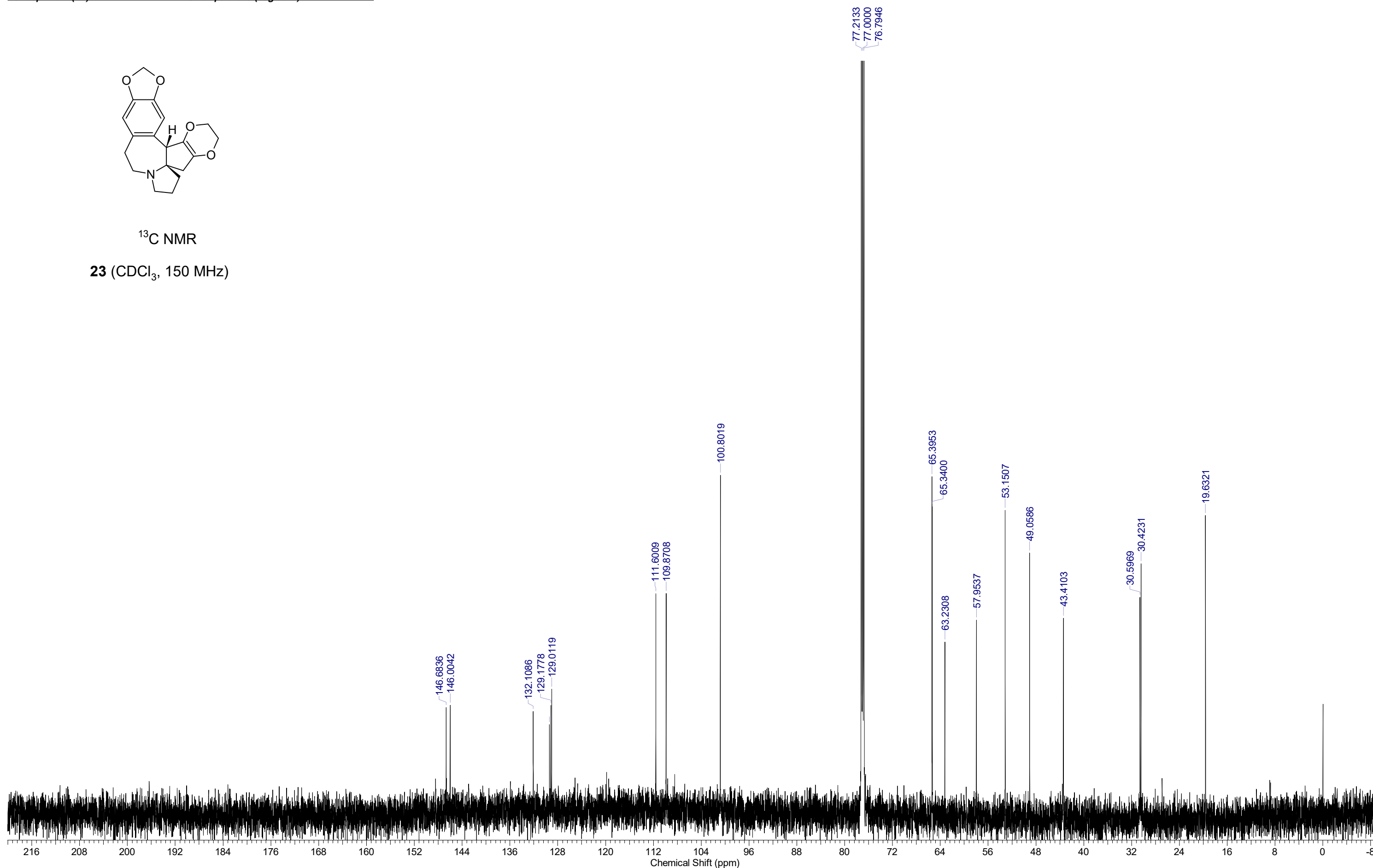


Acquisition Time (sec)	0.8389	Date	09 Oct 2020 15:02:40	Date Stamp	09 Oct 2020 15:02:40	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07093ueue¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	64	Origin	spect
Pulse Sequence	zgpg30	Receiver Gain	193.26	SV(cyclical) (Hz)	39062.50	Original Points Count	32768
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.145	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	15086.7188
						Points Count	32768
						Spectrum Type	STANDARD

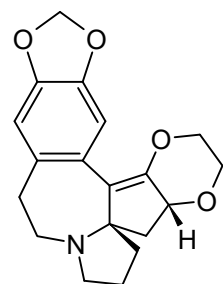


¹³C NMR

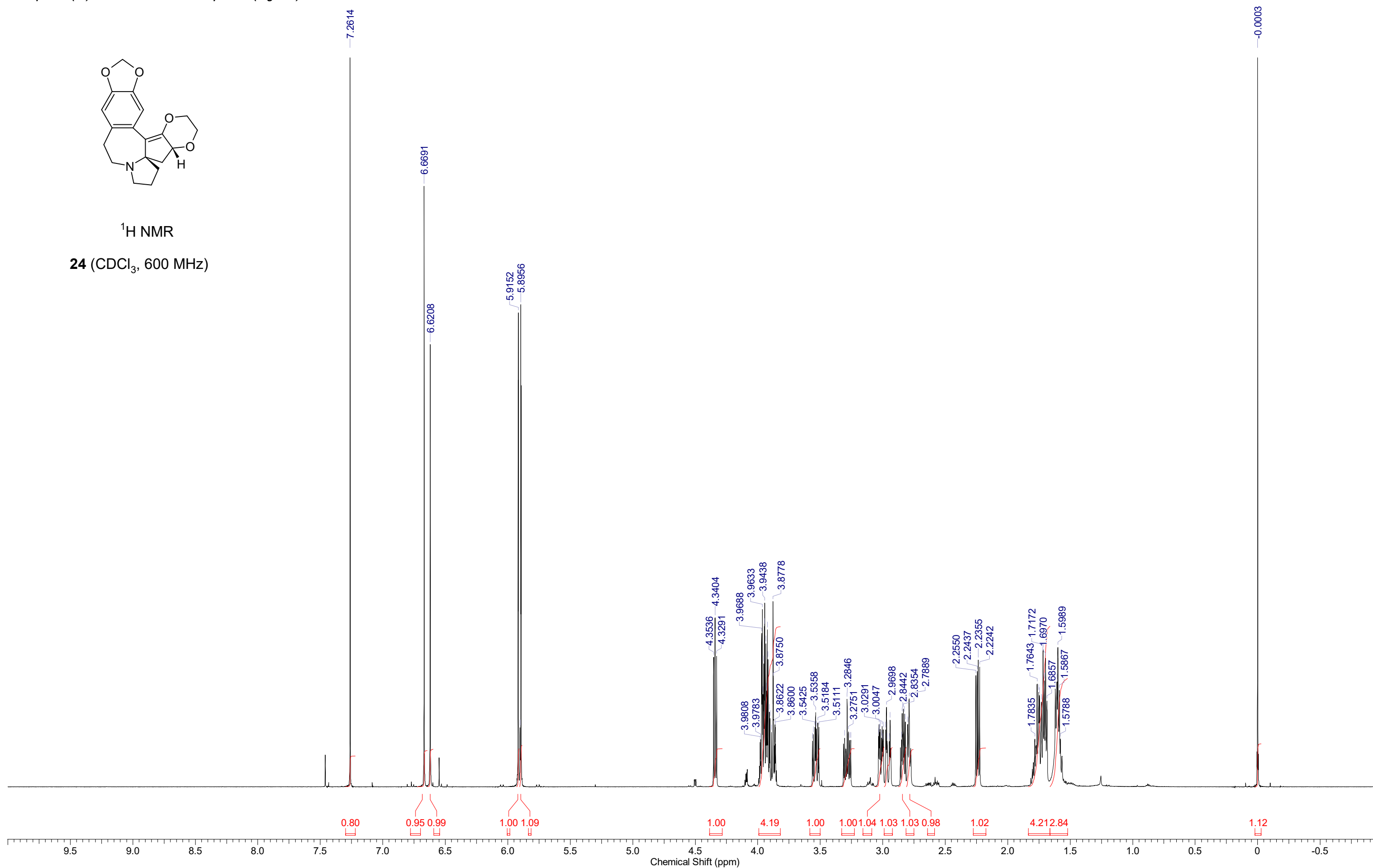
23 (CDCl₃, 150 MHz)



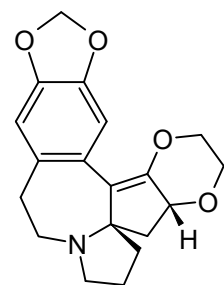
Acquisition Time (sec)	2.7263	Date	09 Oct 2020 17:25:36	Date Stamp	09 Oct 2020 17:25:36	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07093ueshita¥10¥pdata¥1¥1r
Frequency (MHz)	600.13	Nucleus	1H	Origin	spect	Original Points Count	32768
Pulse Sequence	zg30	Receiver Gain	30.91	SV(cyclical) (Hz)	12019.23	Solvent	CHLOROFORM-d
Sweep Width (Hz)	12019.05	Temperature (degree C)	25.152			Spectrum Offset (Hz)	3692.2961
						Points Count	65536
						Spectrum Type	STANDARD



¹H NMR
24 (CDCl₃, 600 MHz)

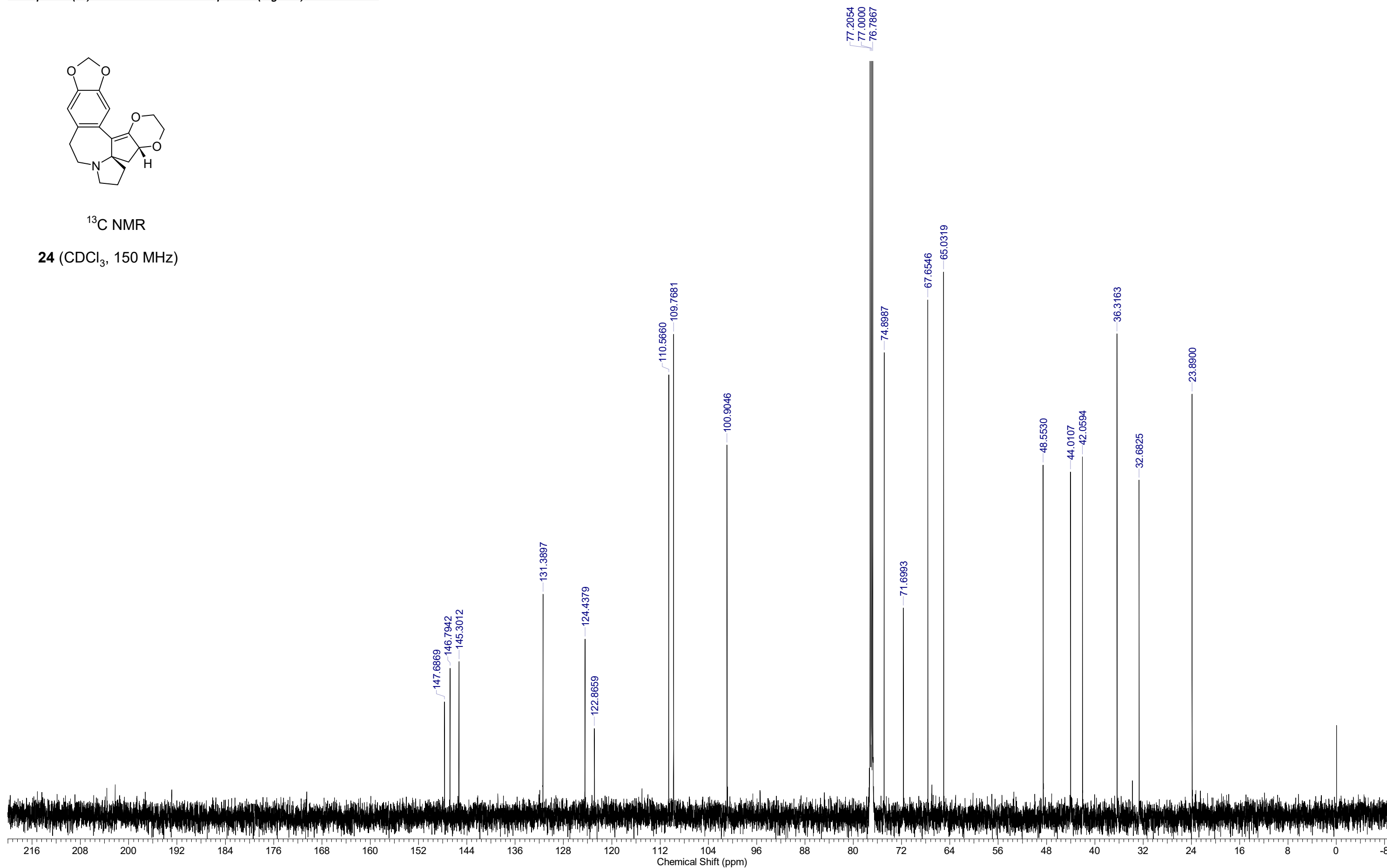


Acquisition Time (sec)	0.8389	Date	09 Oct 2020 15:19:44	Date Stamp	09 Oct 2020 15:19:44	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai07093ueshita¥2¥pdata¥1¥1r
Frequency (MHz)	150.90	Nucleus	¹³ C	Number of Transients	64	Origin	spect
Pulse Sequence	zgpg30	Receiver Gain	193.26	SW(cyclical) (Hz)	39062.50	Solvent	CHLOROFORM-d
Sweep Width (Hz)	39061.31	Temperature (degree C)	25.156	Original Points Count	32768	Owner	nmsu
				Spectrum Offset (Hz)	15085.5254	Points Count	32768
						Spectrum Type	STANDARD

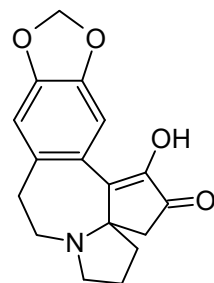


¹³C NMR

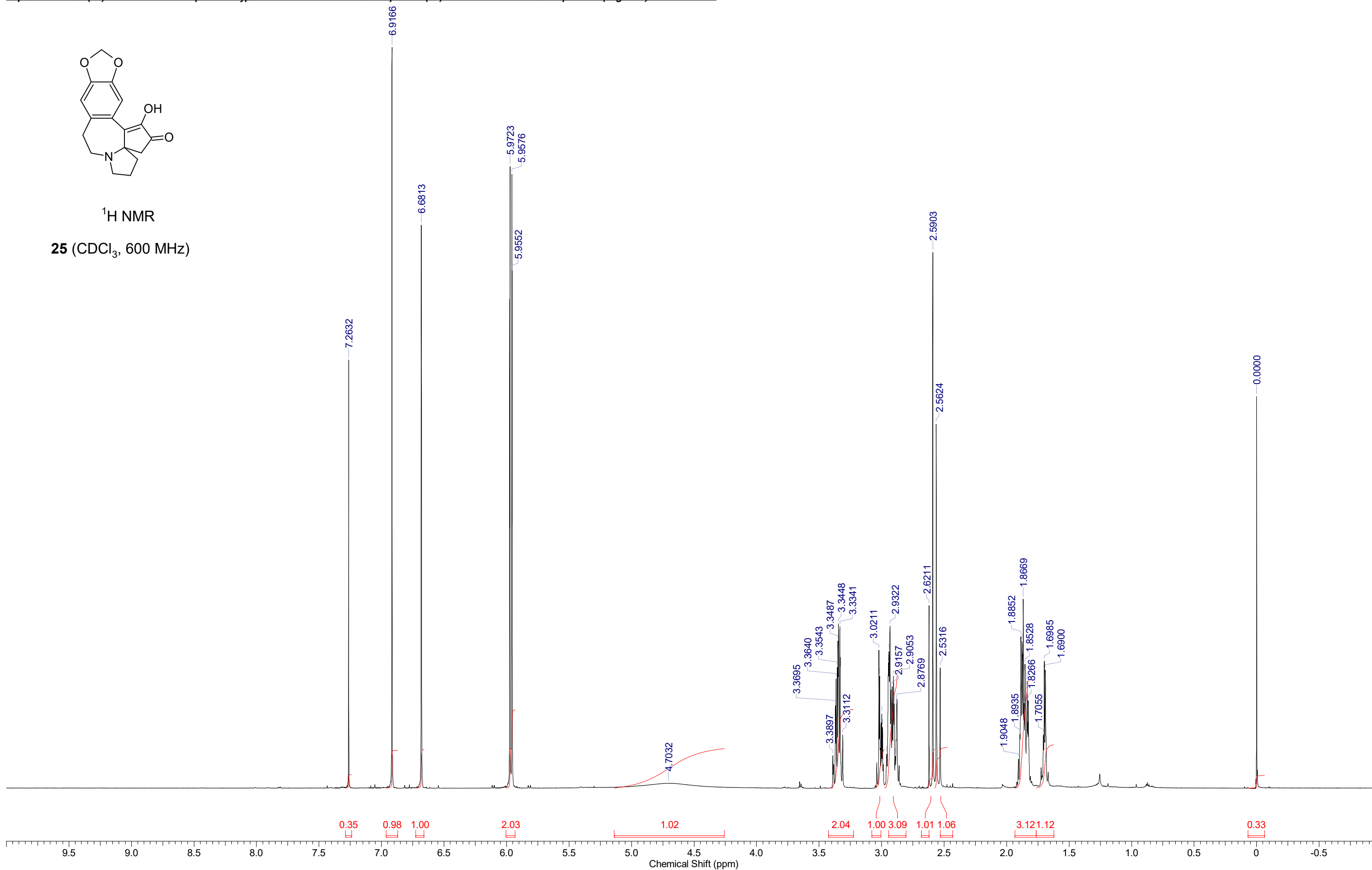
24 (CDCl₃, 150 MHz)



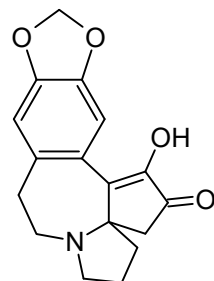
Acquisition Time (sec)	2.7263	Comment	1H	Date	05 Jan 2021 20:48:16	Date Stamp	05 Jan 2021 20:48:16
File Name	¥¥Mac¥Home¥Desktop¥mori_lab NMR¥sakaiNMR¥sakai08022¥1¥pdata¥1¥1r	Owner	nmsu	Points Count	65536	Nucleus	1H
Original Points Count	32768	Spectrum Type	STANDARD	Sweep Width (Hz)	12019.05	Receiver Gain	25.50
Spectrum Offset (Hz)	3693.3789			Temperature (degree C)	25.155	Number of Transients	8
						Origin	spect
						Solvent	CHLOROFORM-d



¹H NMR
25 (CDCl₃, 600 MHz)

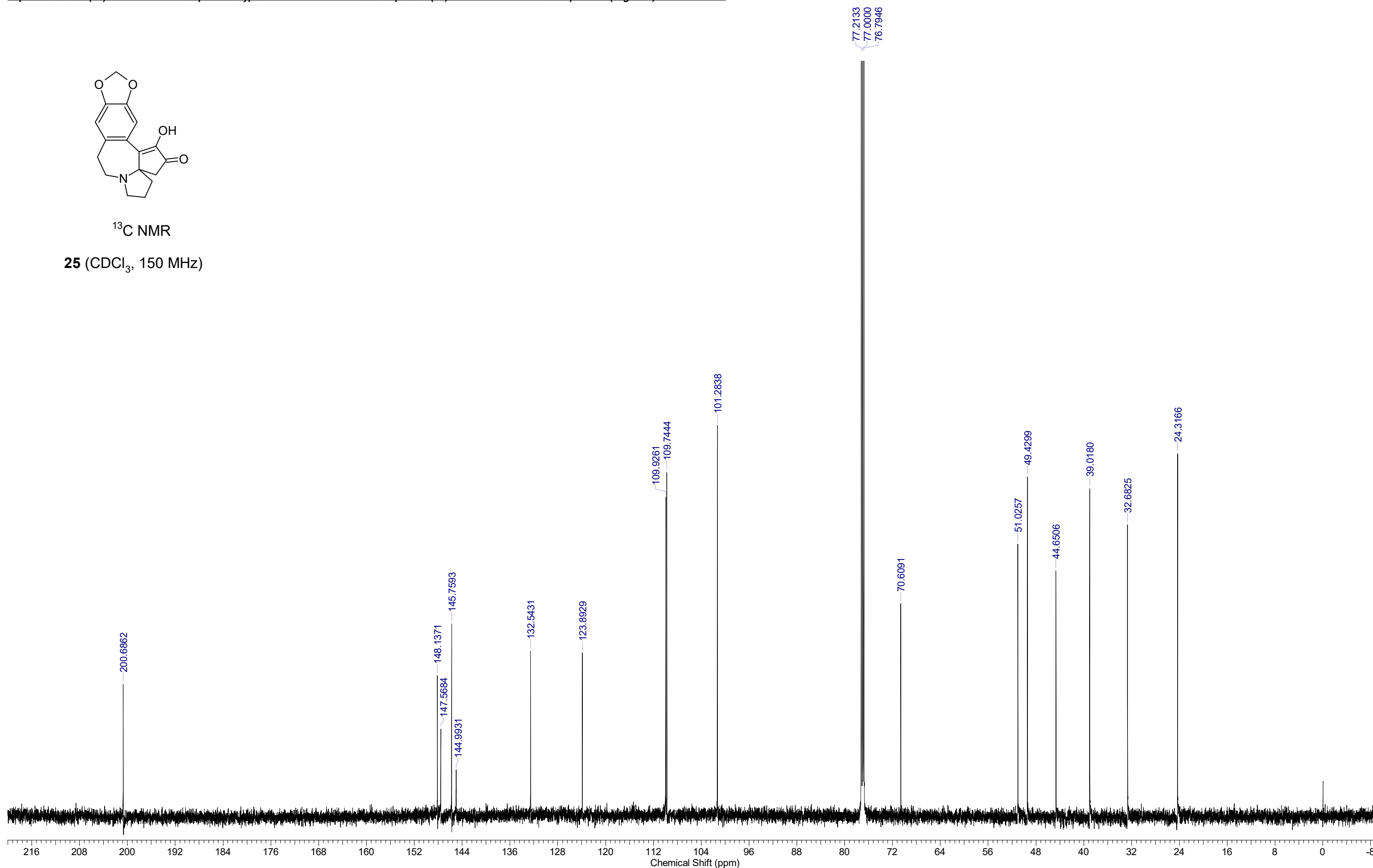


Acquisition Time (sec)	0.8389	Comment	13C	Date	05 Jan 2021 20:54:40	Date Stamp	05 Jan 2021 20:54:40
File Name	¥¥Mac¥Home¥Desktop¥mori.lab NMR¥sakaiNMR¥sakai08022¥2¥pdata¥1¥1r	Frequency (MHz)	150.90	Nucleus	13C	Number of Transients	80
Original Points Count	32768	Owner	nrmrsu	Points Count	32768	Pulse Sequence	zpgpg30
Spectrum Offset (Hz)	15085.5254	Spectrum Type	STANDARD	Sweep Width (Hz)	39061.31	Receiver Gain	193.26
				Temperature (degree C)	25.145	SW(cyclical) (Hz)	39062.50
						Origin	spect
						Solvent	CHLOROFORM-d

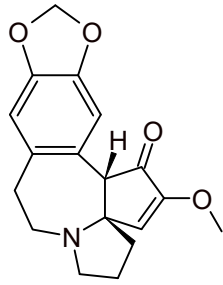


¹³C NMR

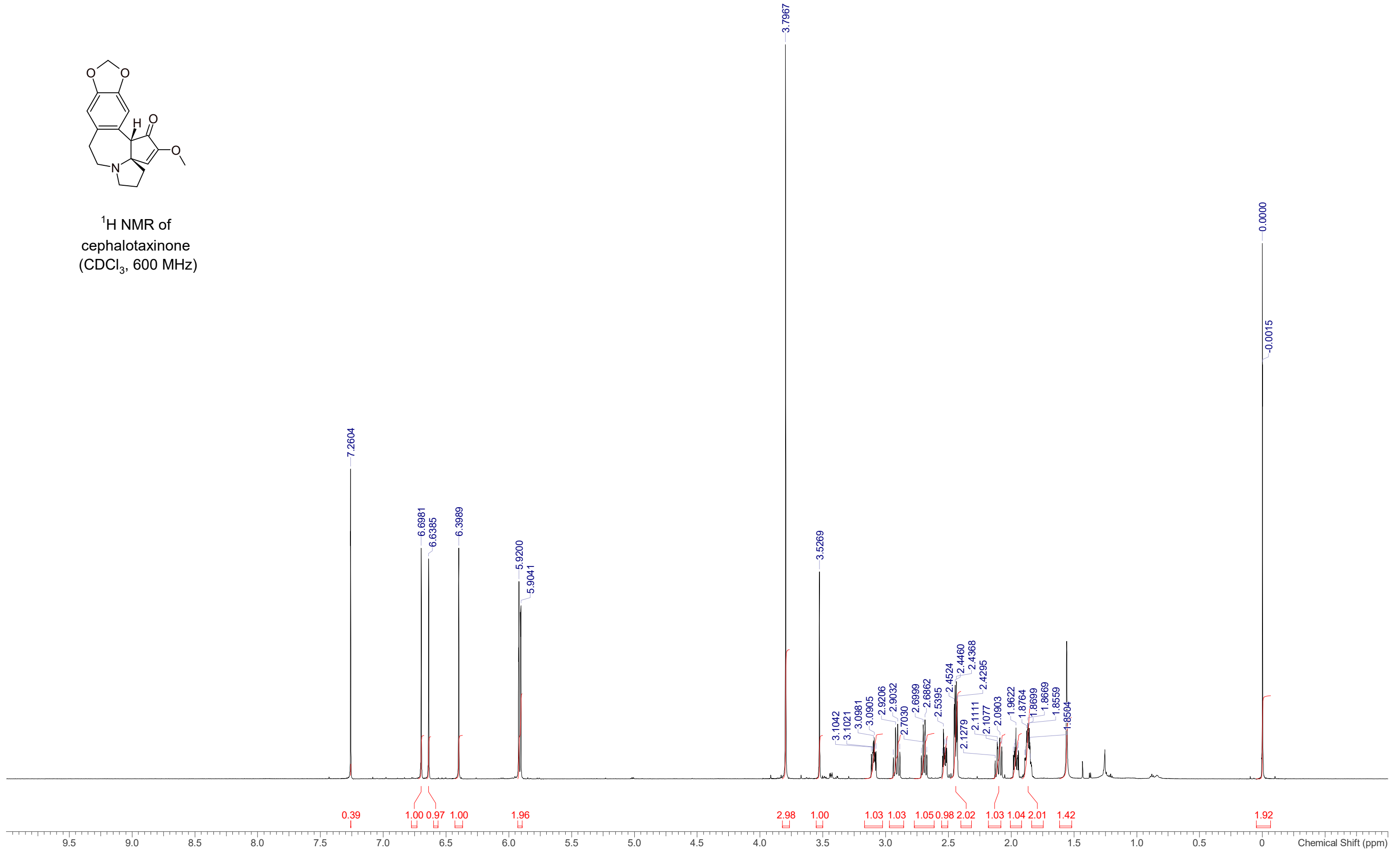
25 (CDCl₃, 150 MHz)



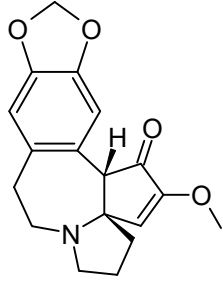
Acquisition Time (sec)	2.7263	Comment	proton	D	0.0001	D1	3	DE	10	DS	0	Date	26 Feb 2021 19:50:48 (GMT+09:00)		
Date Stamp	26 Feb 2021 19:50:48 (GMT+09:00)	ExpNo	1	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai08042shita¥1¥pdata¥1¥1r			Frequency (MHz)	600.1300	GB	0				
INSTRUM	<spect>	LB	0.3	NS	8	Nucleus	1H	Number of Transients	8	Origin	spect	Original Points Count	32768	Owner	nmsu
PC	1	PROBHD	<Z126545 0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zg30>	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	95.68	SF	600.13		
SFO1	600.133706048414	SI	65536	SSB	0	SW(cyclical) (Hz)	12019.23	SWH	12019.2307692308						
Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3691.3481	Spectrum Type	standard	Sweep Width (Hz)	12019.05	TD	65536	TDO	1	TE	298.153		
Temperature (degree C)	25.153	UNC1	<1H>	WDW	1										



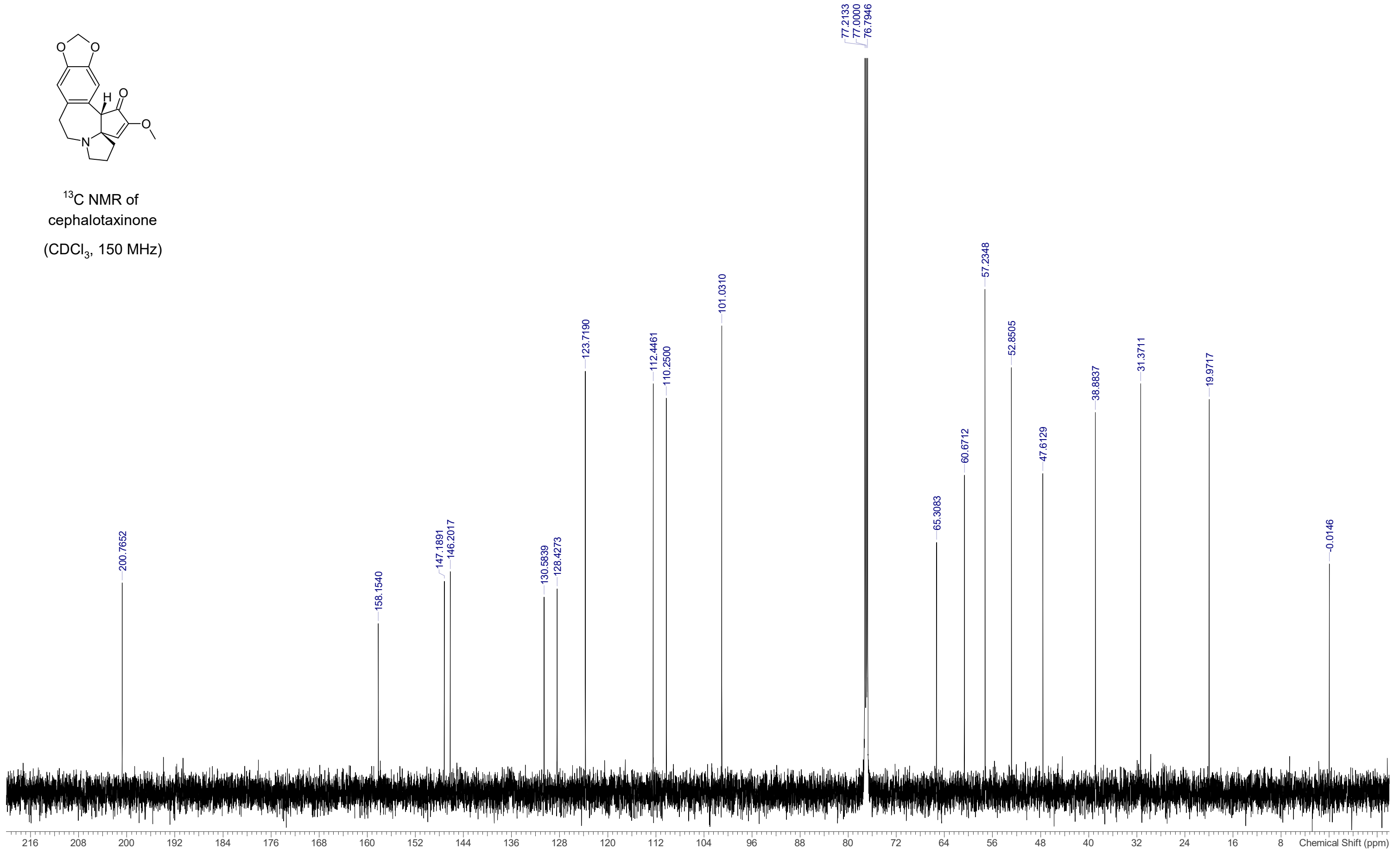
¹H NMR of cephalotaxinone (CDCl₃, 600 MHz)



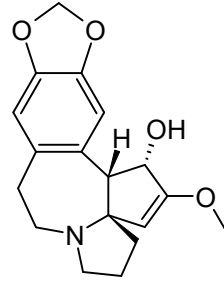
Acquisition Time (sec)	0.8389	Comment	carbon	D	0.03	D1	2	DE	18	DS	4	Date	26 Feb 2021 19:49:41 (GMT+09:00)		
Date Stamp	26 Feb 2021 19:49:41 (GMT+09:00)	ExpNo	2	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai08042shita¥2¥pdata¥1¥1r			Frequency (MHz)	150.9028	GB	0				
INSTRUM	<spect>	LB	1	NS	128	Nucleus	13C	Number of Transients	128	Origin	spect	Original Points Count	32768	Owner	nmsu
PC	1.4	PROBHD	<Z126545 0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zpgg30>	Points Count	32768	Pulse Sequence	zpgg30	Receiver Gain	193.26	SF	150.902809		
SFO1	150.917898095859	SI	32768	SSB	0	SM(cyclical) (Hz)	39062.50	SWH	39062.5	Solvent	CHLOROFORM-d				
Spectrum Offset (Hz)	15086.7119	Spectrum Type	standard	Sweep Width (Hz)	39061.31	TD	65536	TE	298.153	Temperature (degree C)	25.153	UNC1	<13C>		
WDW	1														



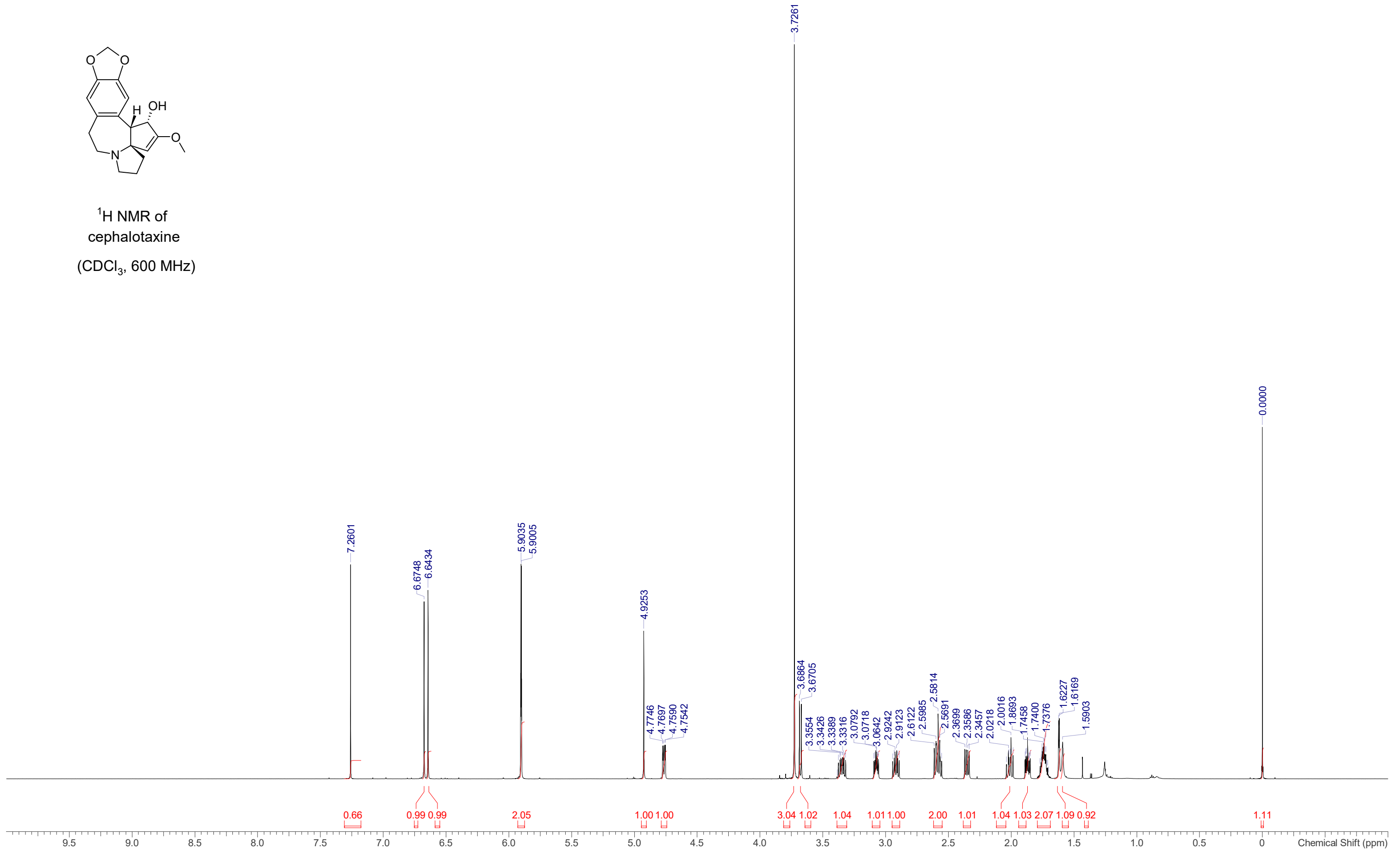
^{13}C NMR of
cephalotaxinone
(CDCl_3 , 150 MHz)



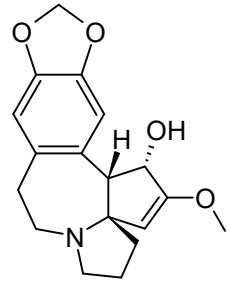
Acquisition Time (sec)	2.7263	Comment	proton	D	0.0002	D1	3	DE	10	DS	0	Date	01 Mar 2021 17:52:12 (GMT+09:00)		
Date Stamp	01 Mar 2021 17:52:12 (GMT+09:00)	ExpNo	1	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai08046¥1¥pdata¥1¥1r			Frequency (MHz)	600.1300	GB	0				
INSTRUM	<spect>	LB	0.3	NS	8	Nucleus	1H	Number of Transients	8	Origin	spect	Original Points Count	32768	Owner	nmsu
PC	1	PROBHD	<Z126545 0014 (CPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zg30>	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	25.50				
SF	600.130013805551	SFO1	600.133706048414	SI	65536	SSB	0	SW(cyclical) (Hz)	12019.23						
SWH	12019.2307692308	Solvent	CHLOROFORM-d	Spectrum Offset (Hz)	3691.5222	Spectrum Type	standard	Sweep Width (Hz)	12019.05	TD	65536				
TDO	1	TE	298.151	UNC1	<1H>	WDW	1								



¹H NMR of
cephalotaxine
(CDCl₃, 600 MHz)



Acquisition Time (sec)	0.8389	Comment	carbon	D	0.03	D1	2	DE	18	DS	4	Date	01 Mar 2021 17:55:56 (GMT+09:00)		
Date Stamp	01 Mar 2021 17:55:56 (GMT+09:00)	ExpNo	2	File Name	¥¥Mac¥Home¥Desktop¥mori lab NMR¥sakaiNMR¥sakai08046¥2¥pdata¥1¥1r			Frequency (MHz)	150.9028	GB	0				
INSTRUM	<spect>	LB	1	NS	64	Nucleus	13C	Number of Transients	64	Origin	spect	Original Points Count	32768	Owner	nmsu
PC	1.4	PROBHD	<Z126545 0014 (GPP BBO 600S3 BB-H&F-D-05 Z)>	PULPROG	<zpgpg30>	Points Count	32768	Pulse Sequence	zpgpg30	Receiver Gain	193.26	SF	150.902809		
SFO1	150.917898095859	SI	32768	SSB	0	SW(cyclical) (Hz)	39062.50	SWH	39062.5	Solvent	CHLOROFORM-d				
Spectrum Offset (Hz)	15085.5205	Spectrum Type	standard	Sweep Width (Hz)	39061.31	TD	65536	TDO	1	TE	298.1456	Temperature (degree C)	25.146	UNC1	<13C>
WDW	1														



^{13}C NMR of
cephalotaxine
(CDCl_3 , 150 MHz)

