Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

N-Hydroxyphthalimide-catalyzed chemoselective intermolecular benzylic C–H amination of unprotected arylalkanols

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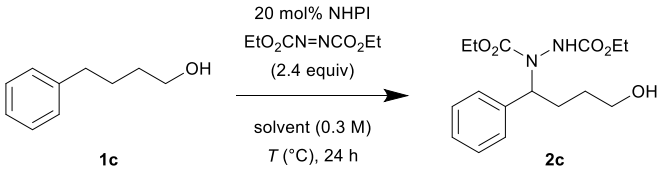
1. Experimental data and possible reaction mechanism

Table S1 Solvent screening using DEAD

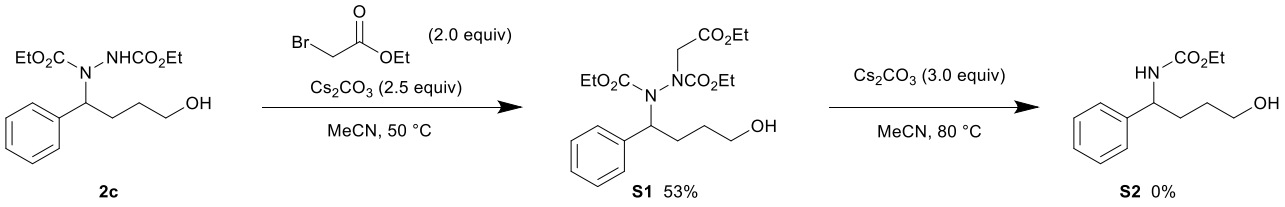


1c **2c**

solvent	*T* (°C)	yield (%) **2c**	**1c** (RSM)
DCE	80	40	17
AcOEt	60	17	31
MeCN	80	25	33
^{t}BuOH	60	no reaction	
AcOH	60	6	10
TFE	60	28	54
HFIP	60	85	5
HFIP[a]	60	87	trace

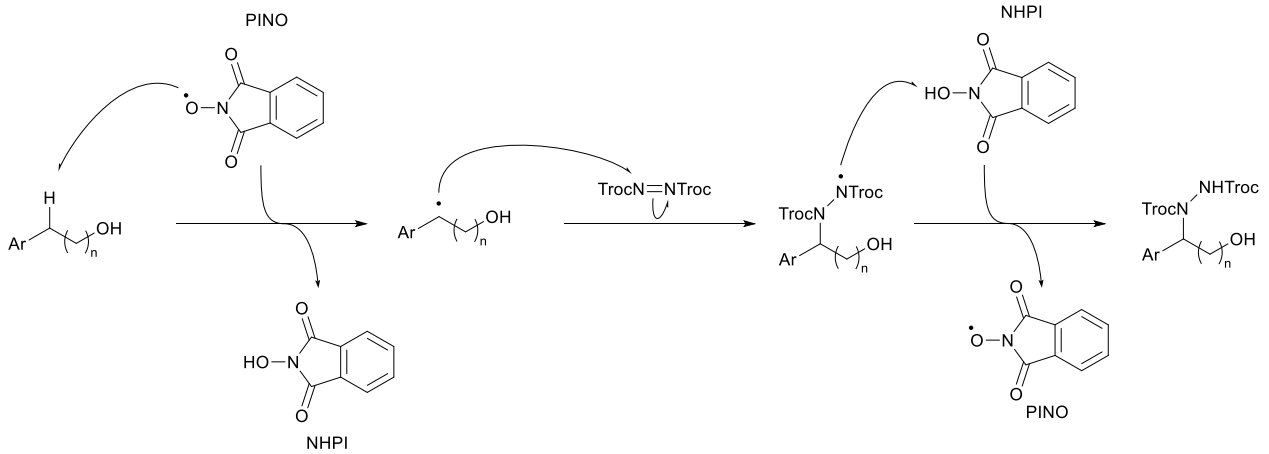
a) 0.5 M

Scheme S1 Examination for the cleavage of the N–N bond of **2c**

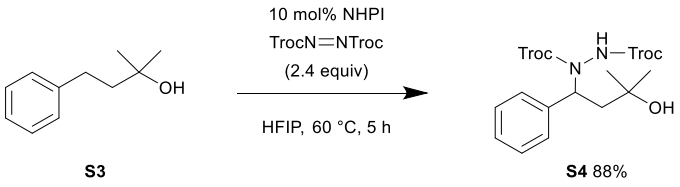


2c **S1** 53% **S2** 0%

Figure S1 Possible reaction mechanism[1]

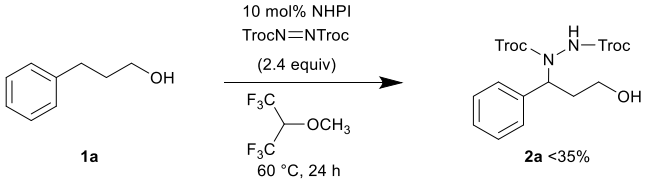


Scheme S2　C–H amination of tertiary alkanol **S3**



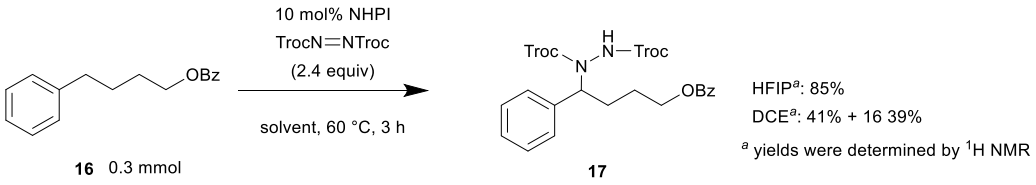
S3　　**S4** 88%

Scheme S3　C–H amination of **1a** in hexafluoroisopropyl methyl ether



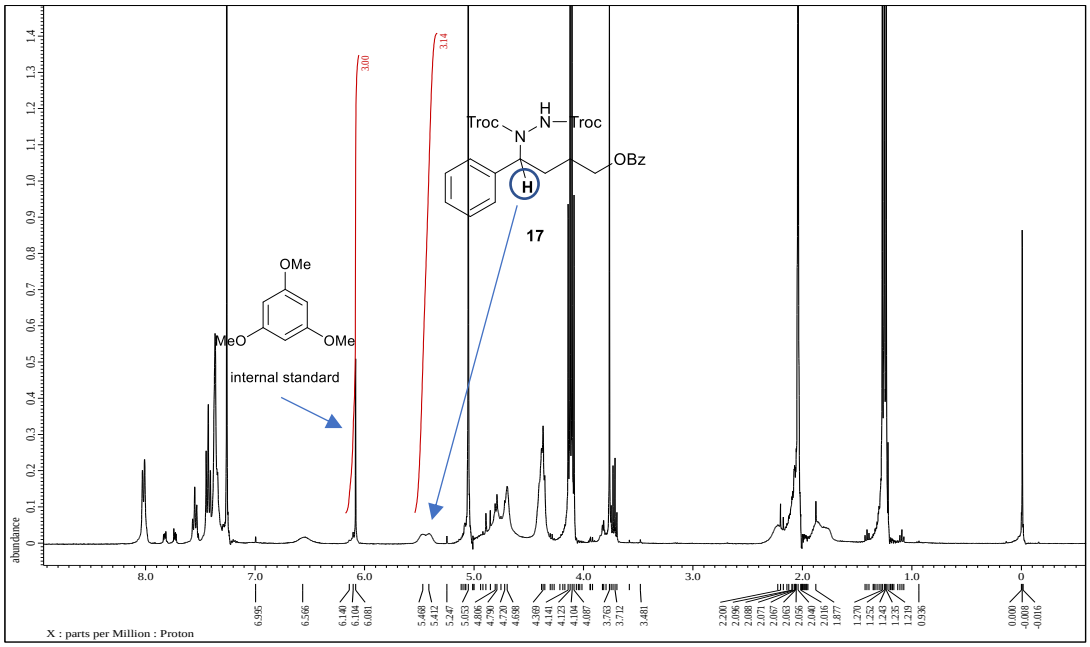
1a　　**2a** <35%

Figure S2　Experimental data for C–H amination of benzoate **16**



NMR charts of the crude materials

solvent: HFIP, internal standard: trimethoxybenzene 13.4 mg (79.7 μmol)



solvent: DCE, internal standard: trimethoxybenzene 5.84 mg (34.7 μmol)

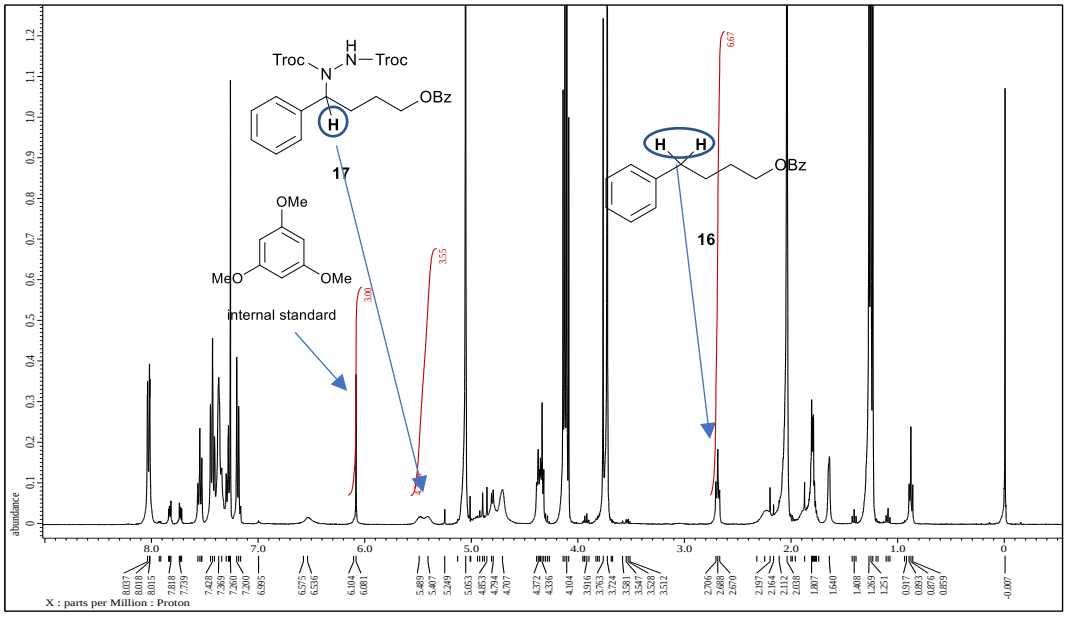
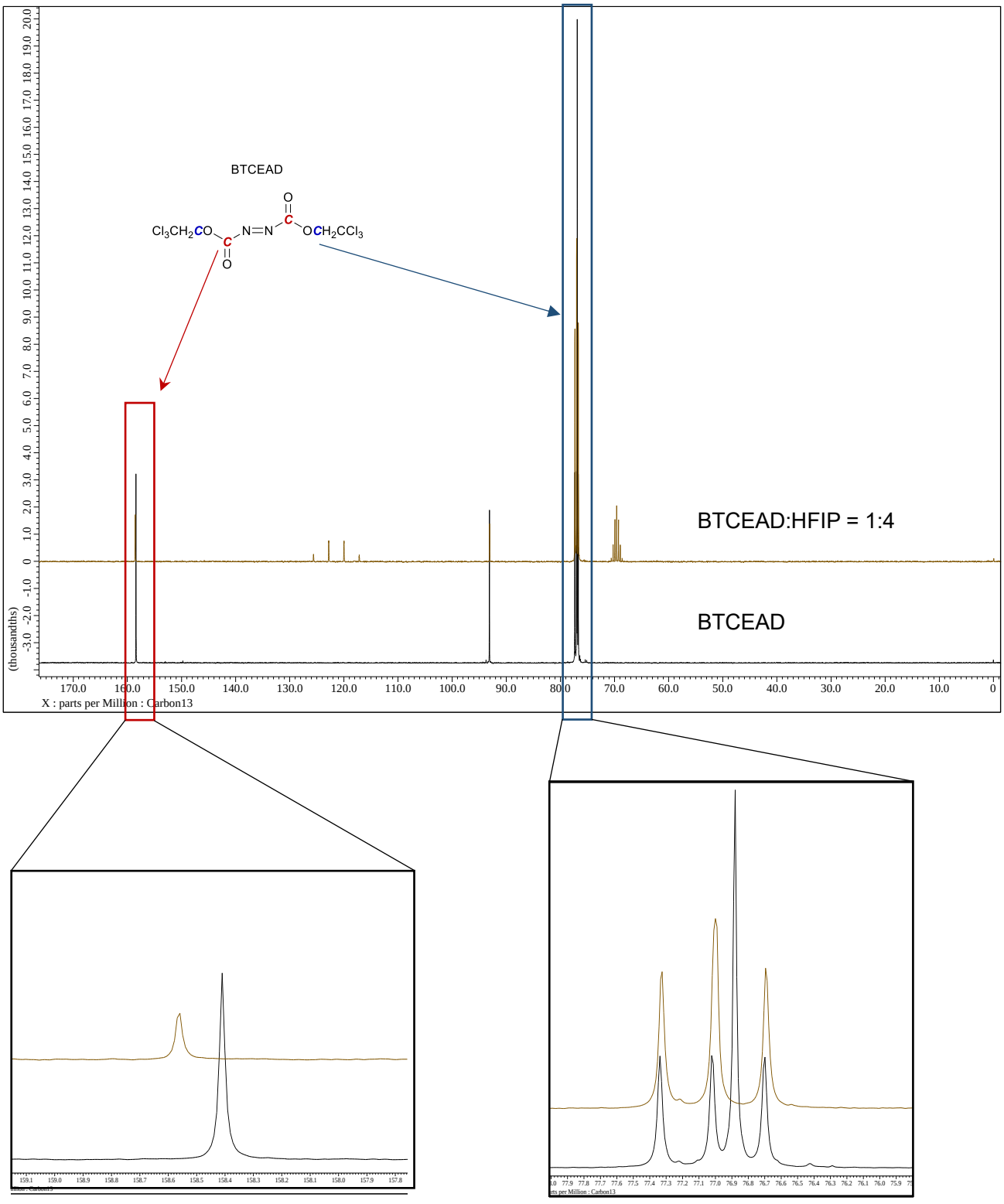


Figure S3　NMR charts of BTCEAD and BTCEAD+HFIP

The signals assigned to the carbonyl carbon and -CH_2- of BTCEAD were shifted downfield by the addition of HFIP.



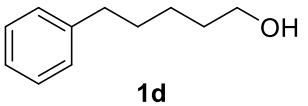
2. Experimental procedures

2-1. General considerations

All reactions were carried out under an argon atmosphere. Solvents and other reagents were purchased from chemical suppliers and used as receive. Reactions were monitored by thin-layer chromatography (TLC: Merck Silica Gel 60 F_{254}). Column chromatography was carried out using neutral silica gel (Cica silica gel 60N, particle size 0.040-0.050 mm, neutral, KANTO CHEMICAL CO., INC.). NMR spectra were measured by JEOL ECS-400 (400 MHz). ^{1}H and ^{13}C NMR chemical shifts are reported in parts per million (ppm, δ scale) relative to residual solvents or internal/external references (^{1}H NMR: $CHCl_3$ at 7.26 ppm or tetramethylsilane at 0.00 ppm as an internal reference in $CDCl_3$; ^{13}C NMR: $CDCl_3$ at 77.00 ppm as an internal reference in $CDCl_3$). Coupling constants (*J*) are reported in Hz. Multiplicities are reported using the following abbreviations; s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; m, multiplet; br, broad. Because the rotamers were observed in the NMR charts at 25 °C, ^{1}H and ^{13}C NMR spectra of the C-H amination products were corrected at 50 °C in $CDCl_3$ or 100 °C in toluene-d_8.[1] Infrared (IR) spectra were recorded on a JASCO FT/IR-4200 at 4.0 cm^{-1} resolution and reported in wavenumbers. Mass spectra were measured by JEOL JMS-T100LP using Electronspray Ionization (ESI) or Direct Analysis in Real Time (DART).

Preparations and Analytical data of arylalkanols

1a–**1c, 1j** and **9** were purchased from chemical suppliers. **1f**, **1g**, **1l**–**1n**, **1p**–**1r**, **11,** were prepared from the corresponding aromatic aldehyde or ketone (for **11**) by the three-step method consisting of HWE reaction using triethyl phosphonoacetate, hydrogenation (H_2, Pd/C), and $LiAlH_4$ of $LiBH_4$ (for **1l**) reduction. **1d**, **1h**, **1i**, **1k**, anc **1o** was prepared from the corresponding carboxylic acid by $LiAlH_4$ or BH_3•THF reduction. **7** was prepared from the corresponding methyl ester by $LiAlH_4$ reduction.[2] **13** was prepared by methylation of 3-phenylpropanal using MeMgBr.



1d

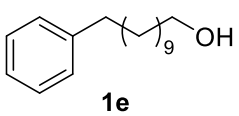
colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.32－7.24 (m, 2H), 7.20－7.14 (m, 3H), 3.64 (t, *J* = 6.4 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 1.70－1.56 (m, 4H), 1.45－1.37 (m, 2H), 1.23 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 142.5, 128.4, 128.2, 125.6, 62.8, 35.9, 32.6, 31.2, 25.4; IR (neat, cm^{-1}) 3334; HRMS (ESI, *m/z*) Calcd. for $C_{11}H_{16}NaO$ ($[M+Na]^+$): 187.1099, found 187.1101.

Preparation of 1e

To a degassed solution methyl 10-undecenoate (980 μL, 4.4 mmol,) in THF (8.8 mL) was added 9-BBN (0.5 M solution in THF, 10 mL, 5.0 mmol) at 0 °C. The solution was stirred for 6 h at room temperature. Then, K_2CO_3 (1.12 g, 8.0 mmol) was added to the solution. After the resultant solution was degassed, a solution of $Pd(dppf)Cl_2$ (87.2 mg, 0.12 mmol) and PhBr (420 μL, 4.0 mmol) in degassed DMF (20 mL) was added. After the reaction mixture was stirred at 50 °C for 12 h, H_2O was added. The aqueous layer was extracted with E_2O and the combined organic layers were washed with brine, dried over $MgSO_4$, and

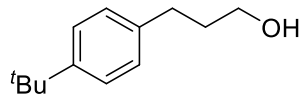
concentrated in vacuo. The crude product was purified by silica gel column chromatography (hexane/AcOEt 50:1) to afford methyl 11-phenyludecenoate (859 mg, 78%).

To a solution of $LiAlH_4$ (281 mg, 7.4 mmol) in THF (6 mL) was added a solution of methyl 11-phenyldecenoate (858 mg, 3.1 mmol) in THF (6 mL) at 0 °C. The reaction mixture was stirred for 25 min at room temperature. The reaction mixture was carefully quenched with H_2O (1.8 mL), 15% NaOH (1.8 mL), and H_2O (5.4 mL) at 0 °C, and filtered through a pad of Celite. The filtrate was extracted with AcOEt, washed with brine, dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AtOEt 6:1) to afford phenylalkanol **1e** (704 mg 91%) as a colorless oil.



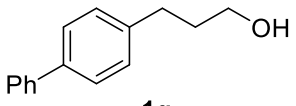
1e

^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.30－7.25 (m, 2H), 7.20－7.14 (m, 3H), 3.64 (td, *J* = 6.8, 5.6 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.65－1.52 (m, 4H), 1.38－1.20 (m, 14H), 1.20 (t, *J* = 5.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 142.9, 128.3, 128.1, 125.5, 62.9, 35.9, 32.7, 31.5, 29.5, 29.4, 29.4, 29.3, 25.7; IR (neat, cm^{-1}) 3332; HRMS (DART, *m/z*) Calcd. for $C_{17}H_{32}NO$ ([M+ NH_4]$^+$): 266.2484, found 266.2494.



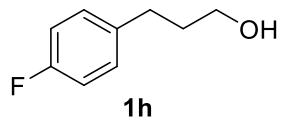
1f

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.32 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 3.69 (t, *J* = 6.4 Hz, 2H), 2.68 (t, *J* = 8.0 Hz, 2H), 1.94－1.85 (m, 2H), 1.31 (s, 9H), 1.26 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 148.6, 138.7 , 128.0, 125.2, 62.4, 34.3, 34.2, 31.5, 31.4; IR (neat, cm^{-1}) 3325; HRMS (ESI, *m/z*) Calcd. for $C_{13}H_{20}NaO$ ([M+Na]$^+$): 215.1407, found 215.1412.



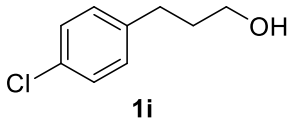
1g

white solid; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.58 (d, *J* = 7.2 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.72 (q, *J* = 5.6 Hz, 2H), 2.76 (t, *J* = 7.6 Hz, 2H), 1.98－1.90 (m, 2H), 1.27 (t, *J* = 5.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 140.96, 140.89, 138.8, 128.8, 128.7, 127.1, 127.0, 126.9, 62.2, 34.1, 31.6; IR (neat, cm^{-1}) 3276; HRMS (ESI, *m/z*) Calcd. for $C_{15}H_{16}NaO$ ([M+Na]$^+$): 235.1099, found 235.1082.



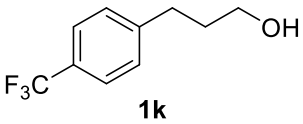
1h

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.17－7.12 (m, 2H), 7.00－6.92 (m, 2H), 3.67 (td, *J* = 6.4, 5.2 Hz, 2H), 2.69 (t, *J* = 8.0 Hz, 2H), 1.91－1.83 (m, 2H), 1.24 (t, *J* = 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 161.2 (d, *J* = 242.1 Hz), 137.4, 129.7 (d, *J* = 7.6 Hz), 115.1 (d, *J* = 20.0 Hz), 62.0, 34.3, 31.2; IR (neat, cm^{-1}) 3335; HRMS (DART, *m/z*) Calcd. for $C_9H_{15}FNO$ ([M+ NH_4]$^+$): 172.1138, found 172.1109.



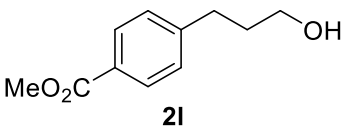
1i

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.25 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 3.67 (q, *J* = 5.6 Hz, 2H), 2.69 (t, *J* = 7.6 Hz, 2H), 1.91－1.83 (m, 2H), 1.26 (t, *J* = 5.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 140.2, 131.5, 129.7, 128.4, 62.0, 34.0, 31.3; IR (neat, cm^{-1}) 3333; HRMS (DART, *m/z*) Calcd. for $C_9H_{15}ClNO$ ($[M+NH_4]^+$): 188.0842, found 188.0828.



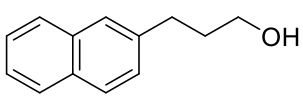
1k

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.69 (td, *J* = 6.4, 5.2 Hz, 2H), 2.78 (t, *J* = 7.6 Hz, 2H), 1.94－1.87 (m, 2H), 1.29 (t, *J* = 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 146.0, 128.7, 128.2 (q, *J* = 32.4 Hz), 125.3 (q, *J* = 2.9 Hz), 124.3 (q, *J* = 271 Hz), 61.9, 33.8, 31.9; IR (neat, cm^{-1}) 3334; HRMS (DART, *m/z*) Calcd. for $C_{10}H_{15}F_3NO$ ($[M+ NH_4]^+$): 222.1106, found 222.1130.



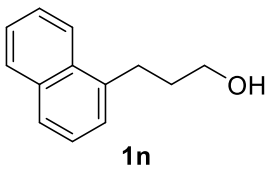
2l

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 3.90 (s, 3H), 3.68 (td, *J* = 6.4, 5.2 Hz, 2H), 2.77 (t, *J* = 8.0 Hz, 2H), 1.95－1.86 (m, 2H), 1.28 (t, *J* = 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 167.1, 147.4, 129.7, 128.4, 127.9, 62.0, 52.0, 33.8, 32.1; IR (neat, cm^{-1}) 3420; HRMS (ESI, *m/z*) Calcd. for $C_{11}H_{14}NaO_3$ ($[M+Na]^+$): 217.0841, found 217.0822.



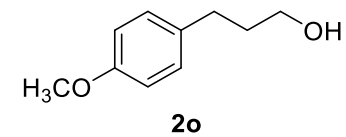
1m

white solid; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.82－7.76 (m, 3H), 7.64 (s, 1H), 7.48－7.39 (m, 2H), 7.35 (dd, *J* = 8.0, 2.0 Hz, 1H), 3.72 (dt, *J* = 6.4, 4.8 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.04－1.95 (m, 2H), 1.28 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 139.3, 133.6, 132.0, 127.9, 127.6, 127.4, 127.2, 126.4, 125.9, 125.2, 62.2, 34.0, 32.2; IR (neat, cm^{-1}) 3269; HRMS (ESI, *m/z*) Calcd. for $C_{13}H_{14}NaO$ ($[M+Na]^+$): 209.0942, found 209.0922.



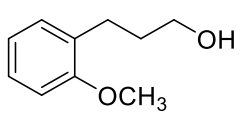
1n

colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 8.06 (d, *J* = 8.0 Hz, 1H), 7.88－7.84 (m, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.54－7.45 (m, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.35 (d, *J* = 6.4 Hz, 2H), 3.79－3.72 (m, 2H), 3.19 (t, *J* = 8.0 Hz, 2H), 2.08－2.00 (m, 2H), 1.33 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 137.8, 133.8, 131.8, 128.7, 126.5, 125.9, 125.7, 125.4, 125.3, 123.7, 62.3, 33.3, 29.0; IR (neat, cm^{-1}) 3335; HRMS (ESI, *m/z*) Calcd. for $C_{13}H_{14}NaO$ ($[M+Na]^+$): 209.0942, found 209.0930.



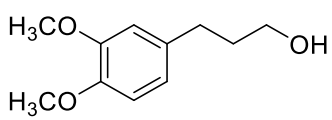
2o

A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.12 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 3.79 (s, 3H), 3.67 (q, *J* = 6.0 Hz, 2H), 2.66 (t, *J* = 8.0 Hz, 2H), 1.91－1.83 (m, 2H), 1.25 (t, *J* = 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 157.8, 133.8, 129.3, 113.8, 62.3, 55.2, 34.4, 31.1; IR (neat, cm^{-1}) 3348; HRMS (ESI, *m/z*) Calcd. for $C_{10}H_{14}NaO_2$ ($[M+Na]^+$): 189.0892, found 189.0886.



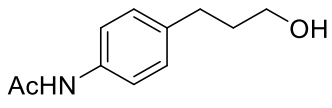
2p

A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.19 (t, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 1H), 6.90 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 3.84 (s, 3H), 3.60 (q, *J* = 6.0 Hz, 2H), 2.73 (t, *J* = 7.2 Hz, 2H), 1.90－1.81 (m, 2H), 1.75 (t, *J* = 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 157.3, 130.1, 129.9, 127.1, 120.6, 110.2, 61.7, 55.3, 32.9, 25.8; IR (neat, cm^{-1}) 3348; HRMS (ESI, *m/z*) Calcd. for $C_{10}H_{14}NaO_2$ ($[M+Na]^+$): 189.0892, found 189.0862.



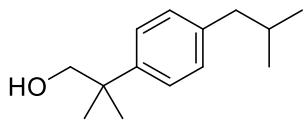
2q

A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 6.80 (d, *J* = 8.4 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 6.73 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.69 (td, *J* = 6.4, 5.2 Hz, 2H), 2.67 (t, *J* = 8.0 Hz, 2H), 1.93－1.84 (m, 2H), 1.25 (t, *J* = 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 148.8, 147.2, 134.4, 120.1, 111.7, 111.2, 62.3, 55.9, 55.8, 34.4, 31.7; IR (neat, cm^{-1}) 3365; HRMS (ESI, *m/z*) Calcd. for $C_{11}H_{20}NO_3$ ($[M+NH_4]^+$): 214.1443, found 214.1456.



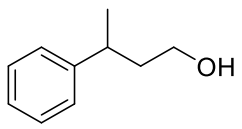
2r

A white solid; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 7.10 (br s, 1H), 3.67 (td, *J* = 6.0, 4.4 Hz, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 2.17 (s, 3 H), 1.91－1.83 (m, 2H), 1.27 (br d, *J* = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 157.9; IR (neat, cm^{-1}) 3277; HRMS (ESI, *m/z*) Calcd. for $C_{11}H_{15}NaO_2$ ($[M+Na]^+$): 216.1001, found 216.0995.



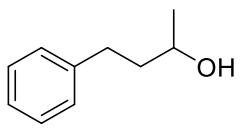
7

The corresponding ester was prepared from ibuprofen according to the reported protocol.[2] A white solid; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 3.60 (d, *J* = 6.8 Hz, 2H), 2.45 (d, *J* = 7.2 Hz, 2H), 1.91－1.79 (m, 1H), 1.33 (s, 6H), 1.19 (t, *J* = 6.8 Hz, 1H), 0.90 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 143.4, 139.4, 129.1, 125.9, 73.1, 44.9, 39.7, 30.1, 25.3, 22.4; IR (neat, cm^{-1}) 3230; HRMS (ESI, *m/z*) Calcd. for $C_{14}H_{26}NO$ ($[M+NH_4]^+$): 224.2014, found 224.2038.



11

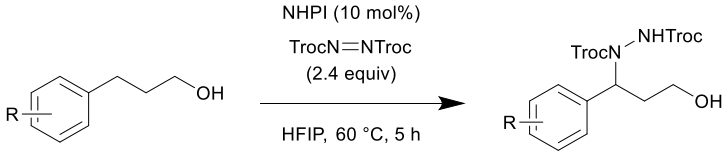
A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.40－7.20 (m, 5H), 3.69－3.54 (m, 2H), 2.99－2.88 (m, 1H), 1.91 (q, *J* = 6.8 Hz, 2H), 1.32 (d, *J* = 6.8 Hz, 3H), 1.20－1.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 146.8, 128.4, 126.9, 126.0, 61.0, 40.9, 36.4, 22.3; IR (neat, cm^{-1}) 3335; HRMS (ESI, *m/z*) Calcd. for $C_{10}H_{18}NO$ ($[M+NH_4]^+$): 168.1388, found 168.1359.



13

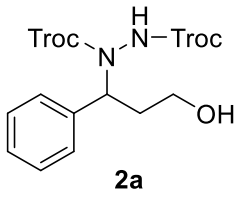
A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.32－7.26 (m, 2H), 7.23－7.16 (m, 3H), 3.90－3.78 (m, 1H), 2.81－2.63 (m, 2H), 1.85－1.70 (m, 2H), 1.34 (br s, 1H), 1.23 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 142.0, 128.3, 128.3, 125.7, 67.4, 40.8, 32.1, 23.5; IR (neat, cm^{-1}) 3357; HRMS (ESI, *m/z*) Calcd. for $C_{10}H_{18}NO$ ($[M+NH_4]^+$): 168.1388, found 168.1361.

2-2. Representative procedures for chemoselective benzylic C–H amination of arylalkanols and analytical data of the products



1 **2**

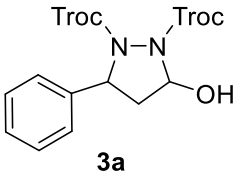
Representative procedure A: To a solution of **1a** (40.9 mg, 0.3 mmol) and NHPI (4.9 mg, 0.03 mmol) in HFIP (0.6 mL) was added TrocN=NTroc (BTCEAD, 274 mg, 0.72 mmol) at ambient temperature. The solution was degassed. The reaction mixture was stirred at 60 °C for 5 h. The reaction progress was monitored by TLC analysis. The solvent was removed in vacuo, and the crude product was purified by silica gel column chromatography (hexane/AcOEt 5:2) to afford **2a** (138 mg, 89%) as an amorphous colorless solid.



2a

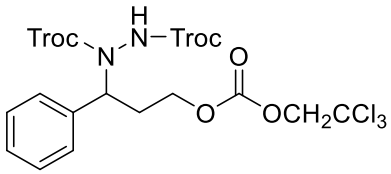
^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.45－7.30 (m, 5H), 6.68 (br s, 1H), 5.59 (br s, 1H), 5.00－4.60 (m, 4H), 4.04－3.86 (m, 1H), 3.85－3.70 (m, 1H), 2.50－2.30 (m, 1H), 2.25－2.03 (m, 1H); ^{13}C NMR (100 MH, $CDCl_3$, 50 °C) δ 154.5, 154.5, 137.8, 128.8, 128.5, 128.1, 94.9, 94.8, 75.9, 75.2, 59.7, 59.7, 33.2; IR (neat, cm^{-1}) 3291, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{15}H_{16}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ($[M+Na]^+$): 538.9059, found 538.9037.

Analytical data of byproducts obtained from the reaction in DCE



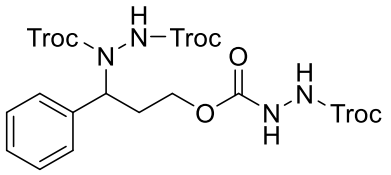
3a

An amorphous blue green solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.41－7.24 (m, 5H), 6.07 (br d, *J* = 4.4 Hz, 1H), 5.58 (t, *J* = 7.6 Hz, 1H), 4.91 (d, *J* = 12.0 Hz, 1H), 4.81 (s, 2H), , 4.80 (d, *J* = 12.0 Hz, 1H), 2.91 (br s, 1H), 2.74 (ddd, *J* = 14.0, 7.6, 1.2 Hz, 1H), 2.41－2.32 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 155.9, 152.9, 139.8, 128.8, 127.8, 125.9, 94.9, 94.8, 83.3, 75.7, 75.6, 63.1, 43.0; IR (neat, cm^{-1}) 3437, 1727; HRMS (DART, *m/z*) Calcd. for $C_{15}H_{18}{}^{35}Cl_5{}^{37}ClN_3O_5$ ($[M+NH_4]^+$): 531.9348, found 531.9334



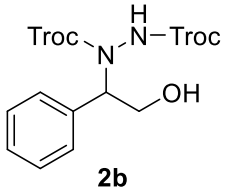
4a

An amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.44-7.30 (m, 5H), 6.45 (br s, 1H), 5.66－5.52 (m, 1H), 5.00－4.60 (m, 6H), 4.60－4.45 (m, 1H), 4.44－4.26 (m, 1H), 2.65-2.45 (m, 1H), 2.44－2.26 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.0, 153.8, 153.8, 136.9, 129.0, 128.8, 128.0, 94.7, 94.7, 94.3, 75.9, 75.7, 75.1, 66.0, 58.8, 29.6; IR (neat, cm^{-1}) 3304, 1759; HRMS (ESI, *m/z*) Calcd. for $C_{18}H_{17}{}^{35}Cl_7{}^{37}Cl_2N_2NaO_7$ ([M+Na]$^+$): 714.8071, found 714.8063.



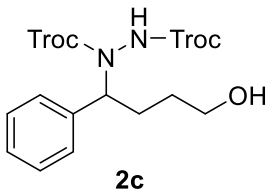
5

An amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.42－7.30 (m, 5H), 6.72 (br s, 1H), 6.50 (br s, 1H), 5.64－5.50 (m, 1H), 4.90－4.60 (m, 7H), 4.54－4.40 (m, 1H), 4.37－4.26 (m, 1H), 2.60－2.40 (m, 1H), 2.37－2.20 (m, 1H); IR (neat, cm^{-1}) 3300, 1732; HRMS (ESI, *m/z*) Calcd. for $C_{19}H_{19}{}^{35}Cl_7{}^{37}Cl_2N_4NaO_8$ ([M+Na]$^+$): 772.8238, found 772.8242.



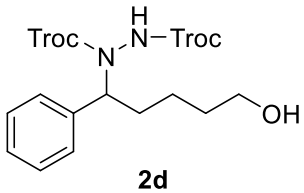
2b

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 9:2) afford **2b** (123.6 mg, 83%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 100 °C) δ 7.42－7.28 (m, 5H), 6.67 (br s, 1H), 5.70－5.50 (m, 1H), 5.10－4.70 (m, 4H), 4.18－4.06 1H), 3.94－3. 84 (m, 1H), 3.72 (br s, 1H); ^{13}C NMR (100 MHz, toluene-d_8, 100 °C) δ 156.6, 154.7, 136.9, 129.2, 128.8, 128.5, 95.8, 95.5, 76.4, 76.0, 65.0, 61.6; IR (neat, cm^{-1}) 3460, 3196, 1732; HRMS (ESI, *m/z*) Calcd. for $C_{14}H_{14}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 524.8902, found 524.8895.



2c

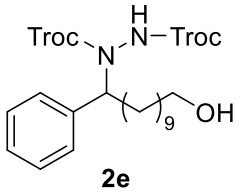
The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) afford **2c** (140.5 mg, 89%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.41－7.30 (m, 5H), 6.55 (br s, 1H), 5.48－5.34 (m, 1H), 5.00－4.60 (m, 4H), 3.78－3.64 (m, 2H), 2.30－2.27 (m, 1H), 2.10－1.95 (m, 1H), 1.95－1.77 (m, 1H), 1.77－1.60 (m, 1H), 1.56－1.36 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.6, 154.2, 138.0, 128.8, 128.5, 128.2, 95.0, 94.9, 75.9, 75.2, 62.4, 62.4, 29.6, 26.9; IR (neat, cm^{-1}) 3290, 1724; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{18}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 552.9215, found 552.9211.



2d

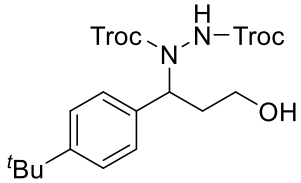
The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) afford **2d** (149.2 mg, 91%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.42－7.28 (m, 5H), 6.52 (br s, 1H), 5.44－5.30 (m, 1H), 5.0－4.66 (m, 4H), 3.68－3.57 (m, 2H), 2.24－2.08 (m, 1H), 2.01－1.86 (m, 1H), 1.78－1.38 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.6, 154.2, 138.0, 128.8, 128.4, 128.2, 95.0, 94.9, 75.9, 75.2, 62.3, 62.3, 32.2, 30.1, 22.6; IR (neat, cm^{-1}) 3288, 1725; HRMS (ESI, *m/z*) Calcd. for $C_{17}H_{20}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 566.9372, found

566.9356.



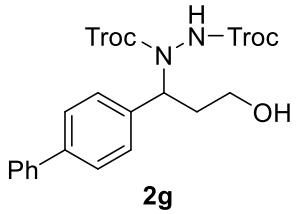
2e

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 4:1) afford **2e** (166.3 mg, 88%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.40－7.30 (m, 5H), 6.45 (br s, 1H), 5.40－5.30 (m, 1H), 4.94－4.66 (m, 4H), 3.63 (t, *J* = 6.8 Hz, 2H), 2.18－2.01 (m, 1H), 2.00－1.88 (m, 1H), 1.60－1.50 (m, 2H), 1.40－1.20 (m, 14H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.4, 154.1, 138.2, 128.6, 128.2, 128.2, 95.0, 95.0, 75.8, 75.0, 63.0, 62.2, 32.7, 30.5, 29.43, 29.38, 29.34, 29.31, 29.31, 26.3, 25.7; IR (neat, cm^{-1}) 3289, 1728; HRMS (ESI, *m/z*) Calcd. for $C_{23}H_{32}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ($[M+Na]^+$): 651.0311, found 651.0317.



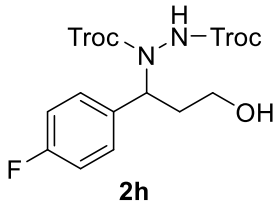
2f

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2f** (163.1 mg, 95%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.39 (br d, *J* = 8.0 Hz, 2H), 7.31 (br d, *J* = 8.0 Hz, 2H), 6.57 (br s, 1H), 5.70－5.50 (s, 1H), 5.00－4.60 (m, 4H), 4.01－3.74 (m, 2H), 2.50－2.00 (m, 2H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.5, 154.5, 151.6, 134.7, 127.8, 125.8, 95.0, 94.8, 75.9, 75.2, 59.8, 59.8, 34.6, 33.4, 31.3; IR (neat, cm^{-1}) 3289, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{19}H_{24}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ($[M+Na]^+$): 594.9685, found 594.9658.



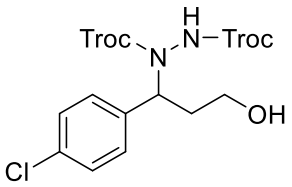
2g

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2g** (162.9 mg, 92%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.62－7.54 (m, 4H), 7.48－7.42 (m, 4H), 7.36 (t, *J* = 7.8 Hz, 1H), 6.67 (br s, 1H), 5.70－5.58 (m, 1H), 5.00－4.65 (m, 4H), 4.05－3.93 (m, 1H), 3.89－3.79 (m, 1H), 2.50－2.36 (m, 1H), 2.23－2.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.6, 154.4, 141.3, 140.3, 136.7, 128.8, 128.7, 127.4, 127.1, 126.9, 94.9, 94.9, 75.8, 75.1, 59.6, 59.6, 33.3; IR (neat, cm^{-1}) 3291, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{21}H_{20}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ($[M+Na]^+$): 614.9372, found 614.9351.



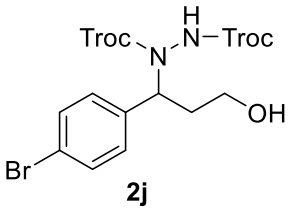
2h

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2h** (131.0 mg, 80%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.42－7.34 (m, 2H), 7.08－6.98 (m, 2H), 6.68 (br s, 1H), 5.62－5.52 (m, 1H), 4.94－4.60 (m, 4H), 4.00－3.86 (m, 1H), 3.85－3.74 (m, 1H), 2.46－2.30 (m, 1H), 2.26－2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 162.7 (d, *J* = 247 Hz), 154.5, 154.4, 133.8, 129.8 (d, *J* = 6.7 Hz), 115.7 (d, *J* = 20.0 Hz), 94.9, 94.8, 75.9, 75.2, 59.7, 59.7, 33.4; IR (neat, cm^{-1}) 3291, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{15}H_{15}{}^{35}Cl_5{}^{37}ClFN_2NaO_5$ ($[M+Na]^+$): 556.8964, found 556.8966.



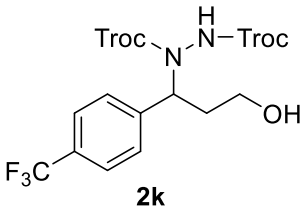
2i

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2i** (141.9 mg, 85%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.38－7.30 (m, 4H), 6.70 (br s, 1H), 5.62－5.50 (m, 1H), 4.90－4.60 (m, 4H), 4.00－3.90 (m, 1H), 3.85－3.75 (m, 1H), 2.37－1.70 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.5, 154.5, 136.4, 134.5, 129.5, 129.0, 94.9, 94.8, 75.9, 75.3, 59.7, 59.7, 33.2; IR (neat, cm^{-1}) 3290, 1727; HRMS (ESI, *m/z*) Calcd. for $C_{15}H_{15}{}^{35}Cl_6{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 572.8668, found 572.8678.



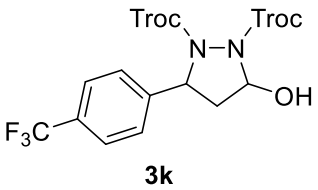
2j

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2j** (152.6 mg, 85%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.50 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 8.8 Hz, 2H), 6.71 (br s, 1H), 5.60－5.50 (m, 1H), 4.96－4.64 (m, 4H), 4.00－3.90 (m, 1H), 3.85－3.74 (m, 1H), 2.45－2.31 (m, 1H), 2.14－2.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.6, 154.3, 136.9, 131.9, 129.9, 122.5, 94.8, 94.8, 75.8, 75.1, 59.5, 59.5, 33.1; IR (neat, cm^{-1}) 3391, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{15}H_{15}Br^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 616.8164, found 616.8183.



2k

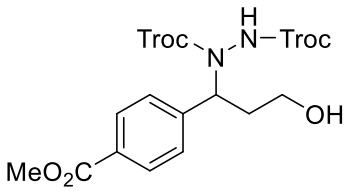
The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2k** (115.3 mg, 66%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.63 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 6.83 (br s, 1H), 5.67－5.58 (m, 1H) , 4.98－4.60 (m, 4H), 4.01－3.91 (m, 1H), 3.84－3.75 (m, 1H), 2.52－2.35 (m, 1H), 2.20－2.04 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.5, 154.5, 141.8 (q, *J* = 20.0 Hz), 130.6 (q, *J* = 32.4 Hz), 128.5, 125.7, 123.8 (q, *J* = 271 Hz), 94.7, 94.7, 75.7, 75.0, 59.7, 59.7, 32.7; IR (neat, cm^{-1}) 3290, 1731; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{15}{}^{37}Cl_5{}^{37}ClF_3N_2NaO_5$ ([M+Na]$^+$): 606.8932, found 606.8921.



3k

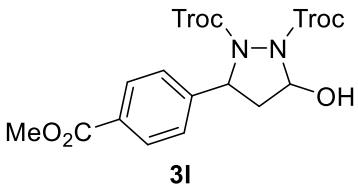
An amorphous colorless solid, 11.2 mg (6%); ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 6.09 (d, *J* = 5.2 Hz, 1H), 5.61 (d, *J* = 7.6 Hz, 1H), 4.94 (d, *J* = 12.0 Hz, 1H), 4.86－4.70 (m, 3H), 3.03 (br s, 1H), 2.80 (ddd, *J* = 13.2, 7.6, 1.6 Hz, 1H), 2.27 (ddd, *J* = 13.2, 7.6, 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 155.9, 152.9, 144.0, 130.2, 126.3, 125.9, 124.0 (q, *J* = 271 Hz), 94.8, 83.3, 75.8, 75.7, 62.7, 43.1;IR (neat, cm^{-1}) 3437, 1731; HRMS (DART, *m/z*) Calcd. for $C_{16}H_{17}{}^{35}Cl_5{}^{37}ClF_3N_3O_5$ ([M+NH_4]$^+$): 599.9222, found 599.9246.

The amount of this compound is too small to obtain ^{13}C NMR spectra.



2l

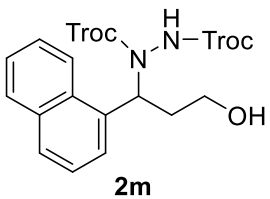
The crude was first purified by flash column chromatography on silica gel (AcOEt), and then by PTLC separation to afford **2l** (112.0 mg, 65%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 8.03 (d, *J* = 8.0 Hz, 2H), 7.48 (d, *J* = 8.0 Hz, 2H), 6.73 (br s, 1H), 5.67－5.57 (m, 1H), 4.90－4.64 (m, 4H), 4.01－3.90 (m, 1H), 3.92 (s, 3H), 3.85－3.75 (m, 1H) , 2.50－2.32 (m, 1H) , 2.18－2.05 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 166.6, 154.6, 154.4, 142.9, 130.2, 130.0, 128.1, 94.8, 94.8, 75.9, 75.2, 59.6, 59.6, 52.1, 33.0; IR (neat, cm^{-1}) 3280, 1719; HRMS (ESI, *m/z*) Calcd. for $C_{17}H_{18}{}^{35}Cl_5{}^{37}ClN_2NaO_7$ ([M+Na]$^+$): 596.9113, found 596.9123.



3l

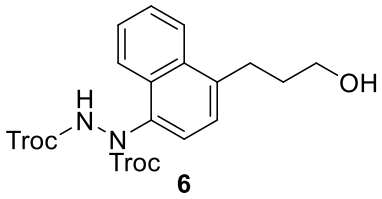
An amorphous light brown solid, 17.2 mg (10%); ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 6.09－6.06 (m, 1H), 5.61 (t, *J* = 7.6 Hz, 1H), 4.93 (d, *J* = 11.2 Hz, 1H), 4.85－4.77 (m, 2H), 4.79 (d, *J* = 11.2 Hz, 1H), 3.91 (s, 3H), 2.88－2.83 (m, 1H), 2.79 (ddd, *J* = 13.2, 8.0, 1.6 Hz, 1H), 2.33－2.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 166.6, 155.9, 152.9, 145.0, 130.2, 129.9, 125.9, 94.84, 94.80, 83.2, 75.72, 75.66, 62.9, 52.1, 43.1; IR (neat, cm^{-1}) 3430, 3305, 1722; HRMS (DART, *m/z*) Calcd. for $C_{17}H_{20}Cl_6N_3O_7$ ([M+NH_4]$^+$): 589.9403, found 589.9387.

Representative procedure B: To a solution of **1m** (55.4 mg, 0.3 mmol) and NHPI (5.0 mg, 0.03 mmol) in HFIP (0.3 mL) and DCE (0.3 mL) was added TrocN=NTroc (BTCEAD, 275 mg, 0.72 mmol) at ambient temperature. The solution was degassed. The reaction mixture was stirred at 60 °C for 6 h. The reaction progress was monitored by TLC analysis. The solvent was removed in vacuo, and the crude product was purified by silica gel column chromatography (hexane/AcOEt 5:2) to afford **2m** (117 mg, 70%) as an amorphous colorless solid and the purification by silica gel column chromatography (hexane/AcOEt 2:1) afforded **6** (4.7 mg, 3%) as an amorphous colorless solid.



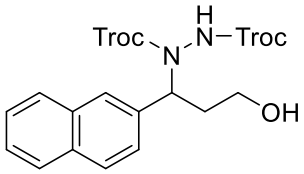
2m

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 8.30－7.80 (m, 3H), 7.60－7.40 (m, 4H), 6.48－6.40 (m, 1H), 6.15 (br s, 1H), 5.10－4.60 (m, 4H), 4.16－3.80 (m, 2H), 2.76－2.44 (m, 1H), 2.30－2.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.9, 154.6, 133.9, 132.5, 131.4, 131.6, 129.4, 129.0, 127.1, 126.2, 125.1, 122.8, 94.8, 94.7, 75.6, 75.0, 59.0, 53.8, 33.4; IR (neat, cm^{-1}) 3294, 1720; HRMS (ESI, *m/z*) Calcd. for $C_{19}H_{18}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 588.9215, found 588.9226.



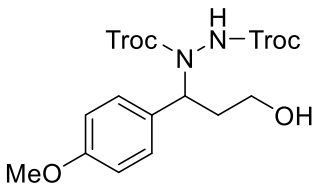
6

An amorphous colorless solid;^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 8.17－8.09 (m, 1H), 8.08－7.95 (m, 1H), 7.78－7.68 (m, 1H), 7.62－7.50 (m, 2H), 7.42－7.33 (m, 1H), 5.00－4.60 (m, 4H), 3.80－3.70 (m, 2H), 3.26－3.14 (m, 2H), 2.08－1.97 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.3, 154.3, 140.2, 135.3, 132.8, 130.2, 126.8, 126.4, 125.8, 125.8, 124.5, 122.9, 94.7, 94.7, 75.9, 75.4, 62.3, 33.4, 29.2; IR (neat, cm^{-1}) 3292, 2954, 1734; HRMS (DART, *m/z*) Calcd. for $C_{19}H_{22}{}^{35}Cl_5{}^{37}ClN_3O_5$ ($[M+NH_4]^+$): 583.9661, found 583.9681.



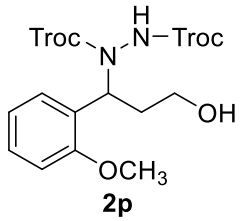
2n

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2n** (147.4 mg, 87%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.88－7.80 (m, 4H), 7.54－7.48 (m, 3H), 6.64 (br s, 1H), 5.82－5.71 (m, 1H), 5.00－4.64 (m, 4H), 4.07－3.94 (m, 1H), 3.92－3.82 (m, 1H), 2.58－2.40 (m, 1H), 2.30－2.20 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.5, 154.5, 135.1, 133.3, 133.2, 128.7, 128.1, 127.7, 127.1, 126.5, 126.4, 126.0, 94.9, 94.8, 75.9, 75.1, 59.8, 59.8, 33.2; IR (neat, cm^{-1}) 3291, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{19}H_{18}{}^{35}Cl_6{}^{37}ClN_2NaO_5$ ($[M+Na]^+$): 588.9215, found 588.9191.



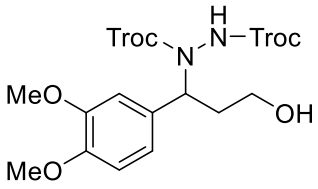
2o

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 2:1) to afford **2o** (145.9 mg, 89%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.30 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 6.55 (br s, 1H), 5.61-5.49 (m, 1H), 5.00－4.60 (m, 4H), 4.00－3.88 (m, 1H), 3.85－3.74 (m, 1H), 3.81 (s, 3H), 2.42－2.26 (m, 1H), 2.20－2.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 159.8, 154.4, 154.4, 129.9, 129.4, 114.2, 95.0, 94.8, 75.8, 75.2, 59.8, 59.8, 55.3, 33.5; IR (neat, cm^{-1}) 3287, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{18}{}^{35}Cl_5{}^{37}ClN_2NaO_6$ ($[M+Na]^+$): 568.9164, found 568.9143.



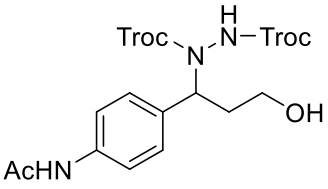
2p

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 5:2) to afford **2p** (153.7 mg, 93%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.37－7.27 (m, 2H), 7.00－6.93 (m, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 6.52 (br s, 1H), 5.96－5.80 (m, 1H), 4.90－4.60 (m, 4H), 4.05－3.92 (m, 1H), 3.90－3.76 (m, 1H), 3.84 (s, 3H), 2.43－2.25 (m, 1H), 2.14－2.00 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 157.5, 154.6, 154.6, 129.7, 128.9, 125.8, 120.6, 110.9, 95.0, 94.9, 75.9, 75.1, 59.6, 55.6, 54.2, 33.3; IR (neat, cm^{-1}) 3286, 1725; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{22}{}^{35}Cl_5{}^{37}ClN_3O_6$ ($[M+Na]^+$): 563.9610, found 563.9639.



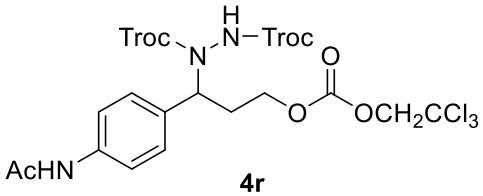
2q

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 2:1) to afford **2q** (110.3 mg, 64%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 6.92 (d, *J* = 8.0 Hz, 1H), 6.91 (s, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.58 (br s, 1H), 5.57－5.48 (br s, 1H), 4.90－4.60 (m, 4H), 4.00－3.75 (m, 2H), 3.86 (s, 3H), 3.85 (s, 3H), 2.40－2.25 (m, 1H), 2.15－2.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.4, 154.4, 149.4, 149.4, 130.4, 120.4, 112.0, 111.5, 95.0, 94.9, 75.8, 75.1, 59.7, 59.7, 56.1, 56.0, 33.5; IR (neat, cm^{-1}) 3289, 1730; HRMS (ESI, *m/z*) Calcd. for $C_{17}H_{20}{}^{35}Cl_5{}^{37}ClN_2NaO_7$ ([M+Na]$^+$): 598.9270, found 598.9269.



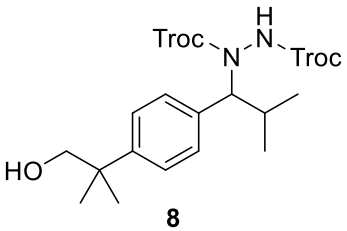
2r

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 3:5) to afford **2r** (108.8 mg, 63%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.47 (br d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.19 (br s, 1H), 6.77 (br s, 1H), 5.62－5.48 (m, 1H), 4.96－4.54 (m, 4H), 4.00－3.86 (m, 1H), 3.84－3.74 (m, 1H), 2.44－2.28 (m, 1H), 2.16 (s, 3H), 2.16－2.12 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 168.7, 154.8, 154.4, 138.0, 133.8, 128.8, 120.3, 94.9, 94.9, 75.8, 75.1, 59.7, 59.7, 33.3, 24.4; IR (neat, cm^{-1}) 3305, 1719; HRMS (ESI, *m/z*) Calcd. for $C_{17}H_{19}{}^{35}Cl_5{}^{37}ClN_3NaO_6$ ([M+Na]$^+$): 595.9273, found 595.9280.



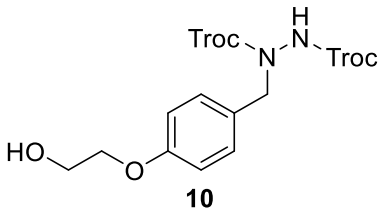
4r

An amorphous light-brown solid, 1.72 mg (5%, as small amounts of inseparable impurities were contained, the yield was determined by ^{1}H NMR spectroscopy with mesitylene as internal standard.): ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.60－7.42 (m, 2H), 7.36－7.28 (m, 2H), 7.20 (br s, 1H), 6.61 (br s, 1H), 5.60－5.55 (m, 1H), 5.00－4.60 (m, 6H), 4.57－4.44 (m, 1H), 4.40－4.30 (m, 1H), 2.58－2.44 (m, 1H), 2.38－2.25 (m, 1H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 168.5, 153.9, 153.9, 153.8, 138.2, 132.0, 128.7, 120.1, 94.7, 94.7, 94.3, 75.7, 75.3, 75.1, 66.0, 57.4, 24.6; IR (neat, cm^{-1}) 3306, 2959, 1760; HRMS (ESI, *m/z*) Calcd. for $C_{20}H_{20}{}^{35}Cl_7{}^{37}Cl_2N_3NaO_8$ ([M+Na]$^+$): 771.8286, found 771.8279.



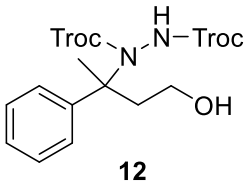
8

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 4:1) to afford **8** (156.8 mg, 89%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.38－7.27 (m, 4H), 6.46 (br s, 1H), 4.98－4.60 (m, 5H), 3.61 (s, 2H), 2.48－2.34 (m, 1H), 1.33 (s, 6H), 1.25 (br s, 3H), 0.80 (br d, *J* = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 154.3, 154.3, 146.7, 134.7, 129.0, 126.5, 95.0, 95.0, 75.8, 75.1, 72.9, 69.2, 40.0, 28.3, 25.3, 20.4, 20.2; IR (neat, cm^{-1}) 3279, 1725; HRMS (ESI, *m/z*) Calcd. for $C_{20}H_{26}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 608.9841, found 608.9847.



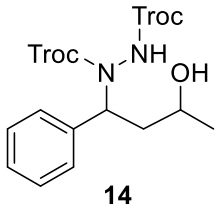
10

The crude was first purified by flash column chromatography on silica gel (hexane/AcOEt = 2:1), and then purified by PTLC to afford **10** (123.8 mg, 77%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.26 (d, *J* = 8.4 Hz, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.81 (br s, 1H), 4.95－4.65 (m, 6H), 4.09 (t, *J* = 4.4 Hz, 2H), 3.96 (br t, *J* = 4.4 Hz, 2H), 1.94 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 158.8, 154.2, 153.6, 130.3, 127.8, 115.0, 94.9, 94.8, 75.9, 75.1, 69.4, 61.4, 53.4; IR (neat, cm^{-1}) 3295, 1731; HRMS (DART, *m/z*) Calcd. for $C_{15}H_{20}{}^{35}Cl_5{}^{37}ClN_3O_6$ ([M+NH_4]$^+$): 549.9454, found 549.9458.



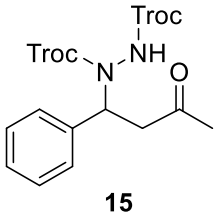
12

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 3:1) afford **12** (66.2 mg, <42%) with unidentified byproducts; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.67－7.19 (m, 6H), 4.95－4.56 (m, 4H), 4.05－3.96 (m, 2H), 2.90－2.82 (m, 1H), 2.20－2.10 (m, 1H), 1.61 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 155.2, 147.2, 128.6, 128.4, 126.7, 124.8, 95.1, 94.9, 75.5, 75.1, 66.0, 59.0, 37.3, 27.2; IR (neat, cm^{-1}) 3309, 2956, 1733; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{18}Cl_6N_2NaO_5$ ([M+Na]$^+$): 552.9210, found 552.9215.



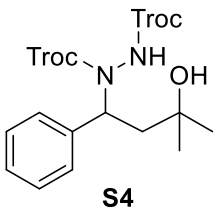
14

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 3:1) afford **14** (117.6 mg, 74%, an 5:4 inseparable mixture of diastereomers) as an amorphous colorless solid: major diastereomer; ^{1}H NMR (400 MHz, toluene-d_8, 100 °C) δ 7.35－7.27 (m, 2H), 7.17－6.94 (m, 3H), 6.14 (s, 1H), 5.56－5.46 (m, 1H), 4.70－4.30 (m, 4H), 3.70－3.58 (m, 1H), 2.30－1.60 (m, 2H), 1.00－0.91 (m, 3H); minor diastereomer; ^{1}H NMR (400 MHz, toluene-d_8, 100 °C) δ 7.28－7.20 (m, 2H), 7.17－6.94 (m, 3H), 6.47 (s, 1H), 5.67－5.57 (m, 1H), 4.70－4.30 (m, 4H), 4.15－4.00 (m, 1H), 2.30－1.60 (m, 2H), 1.20－1.10 (m, 3H); major and minor diastereomers ^{13}C NMR (100 MHz, toluene-d_8, 100 °C) δ 154.9, 154.9, 154.8, 154.5, 139.4, 139.4, 129.1, 129.0, 128.8, 128.7, 128.53, 128.47, 96.0, 95.9, 95.9, 95.8, 76.3, 76.3, 75.7, 75.7, 66.4, 64.8, 61.4, 60.4, 40.9, 40.5, 24.7, 23.6; IR (neat, cm^{-1}) 3290, 1726; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{18}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 552.9215, found 552.9215 .



15

An amorphous colorless solid (8.0 mg, 5%). The following data were collected using the material obtained from the reaction in DCE; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.41－7.30 (m, 5H), 6.66 (br, 1H), 5.94－5.82 (m, 1H), 4.90－4.68 (m, 4H), 3.40－3.28 (m, 1H), 3.12－2.98 (m, 1H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 204.9, 154.7, 153.7, 137.5, 128.8, 128.5, 127.7, 94.8, 94.8, 75.9, 75.2, 58.0, 44.7, 30.1; IR (neat, cm^{-1}) 3285, 1728; HRMS (ESI, *m/z*) Calcd. for $C_{16}H_{16}{}^{35}Cl_5{}^{37}ClN_2NaO_6$ ([M+Na]$^+$): 550.9059, found 550.9059.

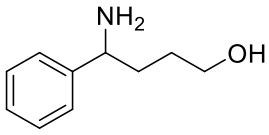


S4

The crude was purified by flash column chromatography on silica gel (hexane/AcOEt = 8:1) afford **S4** (143.5 mg, 88%) as an amorphous colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$, 50 °C) δ 7.46－7.24 (m, 5H), 6.94 (br s, 1H), 5.68 (s, 1H), 5.10－4.50 (m, 4H), 3.98－3.85 (m, 0.25H), 2.70－2.54 (m, 0.25H), 2.54－2.48 (m, 0.75H), 1.94－1.82 (m, 0.75H), 1.80－1.68 (m, 0.25H), 1.70－1.52 (m, 0.75H), 1.33 (s, 6H); ^{13}C NMR (100 MHz, toluene-d_8, 100 °C) δ 155.1, 153.9, 139.3, 128.7, 128.2, 127.8, 94.5, 94.9, 75.8, 75.2, 70.1, 57.9, 42.2, 32.4, 28.0; IR (neat, cm^{-1}) 3292, 1727; HRMS (ESI, *m/z*) Calcd. for $C_{17}H_{20}{}^{35}Cl_5{}^{37}ClN_2NaO_5$ ([M+Na]$^+$): 566.9372, found 566.9354.

Preparation of free amino alcohol 18 from 2c

To a solution of **2c** (48.9 mg, 0.092 mmol) in glacial acetic acid (1.3 mL) was added zinc dust (477 mg, 7.3 mmol) at room temperature. After the solution was stirred for 30 min, acetone (66.9 μL, 0.9 mmol) was added. The reaction mixture was stirred at room temperature for 30 min. Then, CH_2Cl_2 (6 ml) was added and the resultant mixture was sonicated for 1 min. The mixture was filtered through a pad of Celite. Sat. aqueous Na_2CO_3 was added to the filtrate, and the mixture was extracted with CH_2Cl_2. The combined organic layer was washed with brine and dried over $MgSO_4$. and concentrated in vacuo. The crude product was purified by silica gel column chromatography ($CHCl_3$/MeOH 5:1) to afford (11.7 mg, 78%) as a white solid. Analytical data were identical with those of an authentic sample which was prepared from benzoate **17**.



18

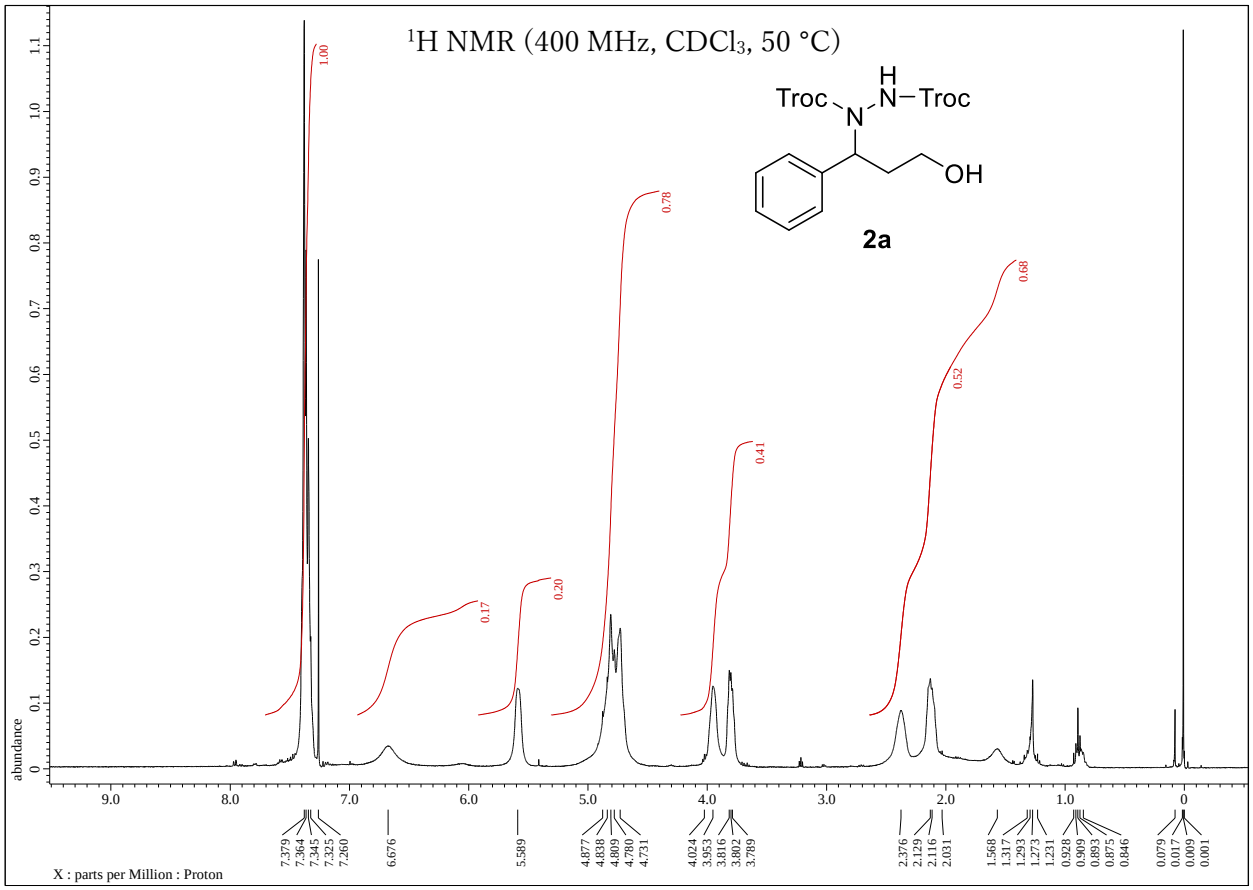
Authentic sample: A white solid; Mp 72.2－73.7 ºC; ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ 7.27－7.22 (m, 5H), 3.95－3.88 (m, 1H), 3.72－3.57 (m, 2H), 3.40－3.15 (m, 3H), 1.79－1.64 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ 146.5, 128.6, 127.1, 125.8, 62.8, 56.2, 37.4, 30.7; IR (neat, cm^{-1}) 3344, 3277, 2935; HRMS (DART, *m/z*) Calcd. for $C_{10}H_{16}NO$ ([M+H]$^+$): 166.1205, found 166.1232.

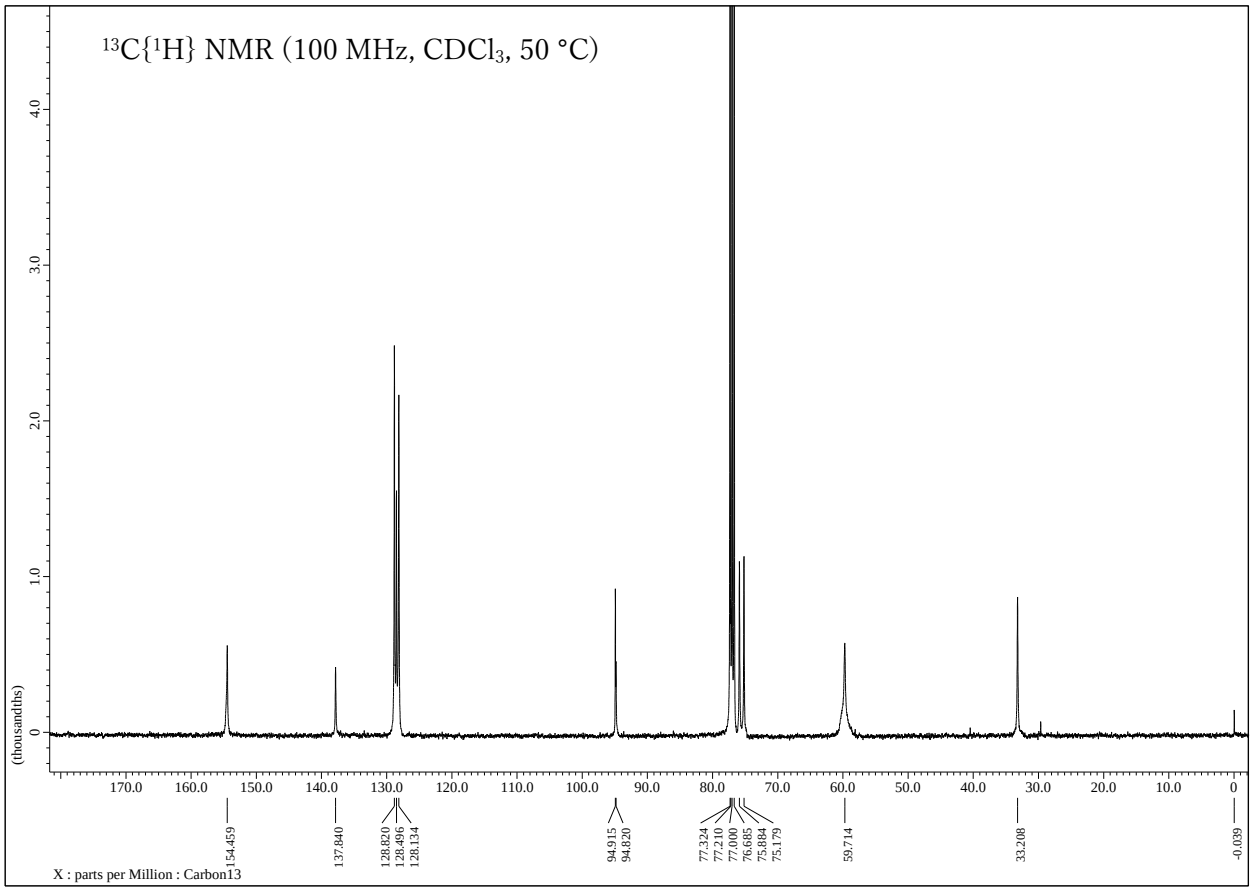
3. References

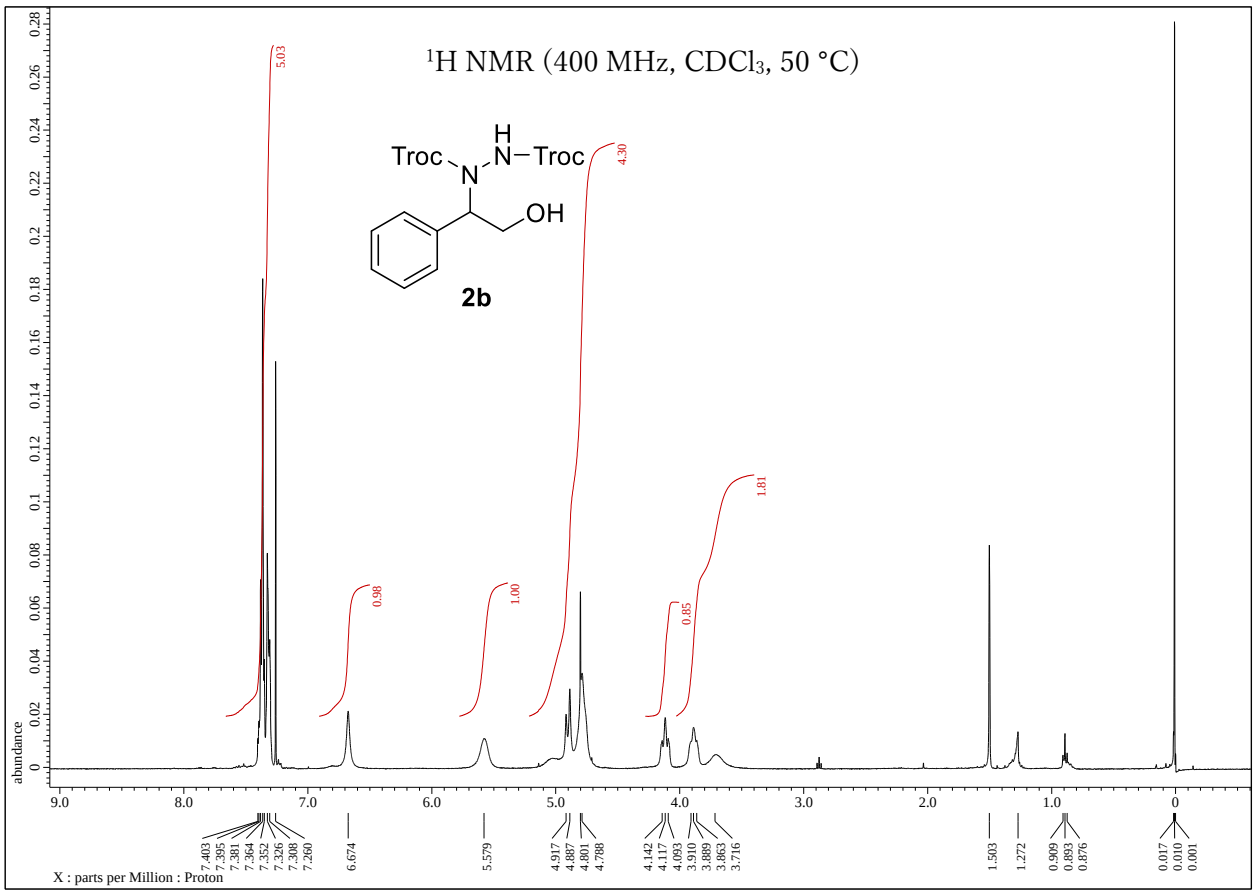
1) Y. Amaoka, S. Kamijo, T. Hoshikawa and M. Inoue, *J. Org. Chem.*, 2012, **77**, 9959-9969.

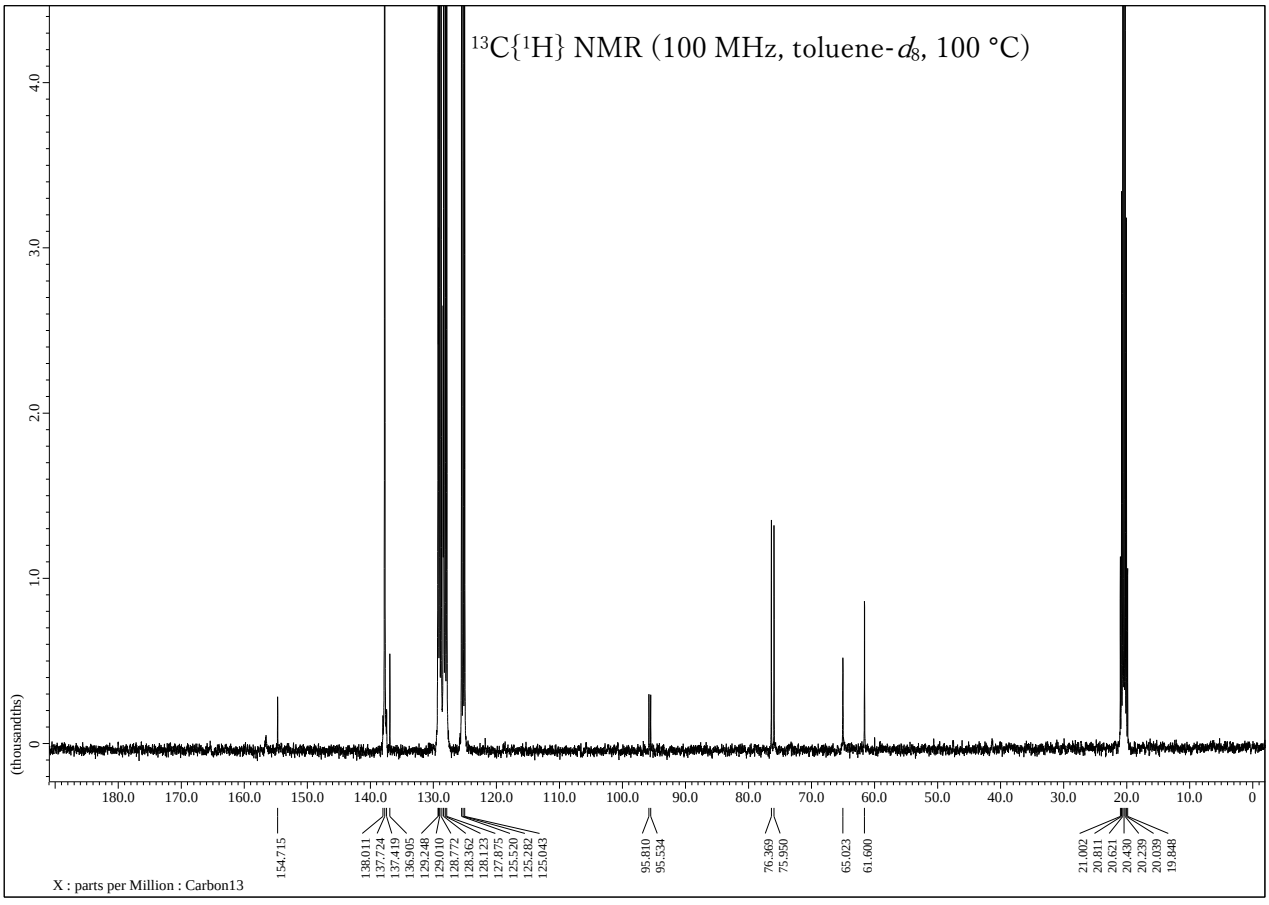
2) E. Banoglu, B. Caliskan, S. Luderer, G. Eren, Y. Ozkan, W. Altenhofen, C. Weinigel, D. Barz, J. Gerstmeier, C. Pergola and O. Werz, *Bioorg. Med. Chem.*, 2012, **20**, 3728-3741.

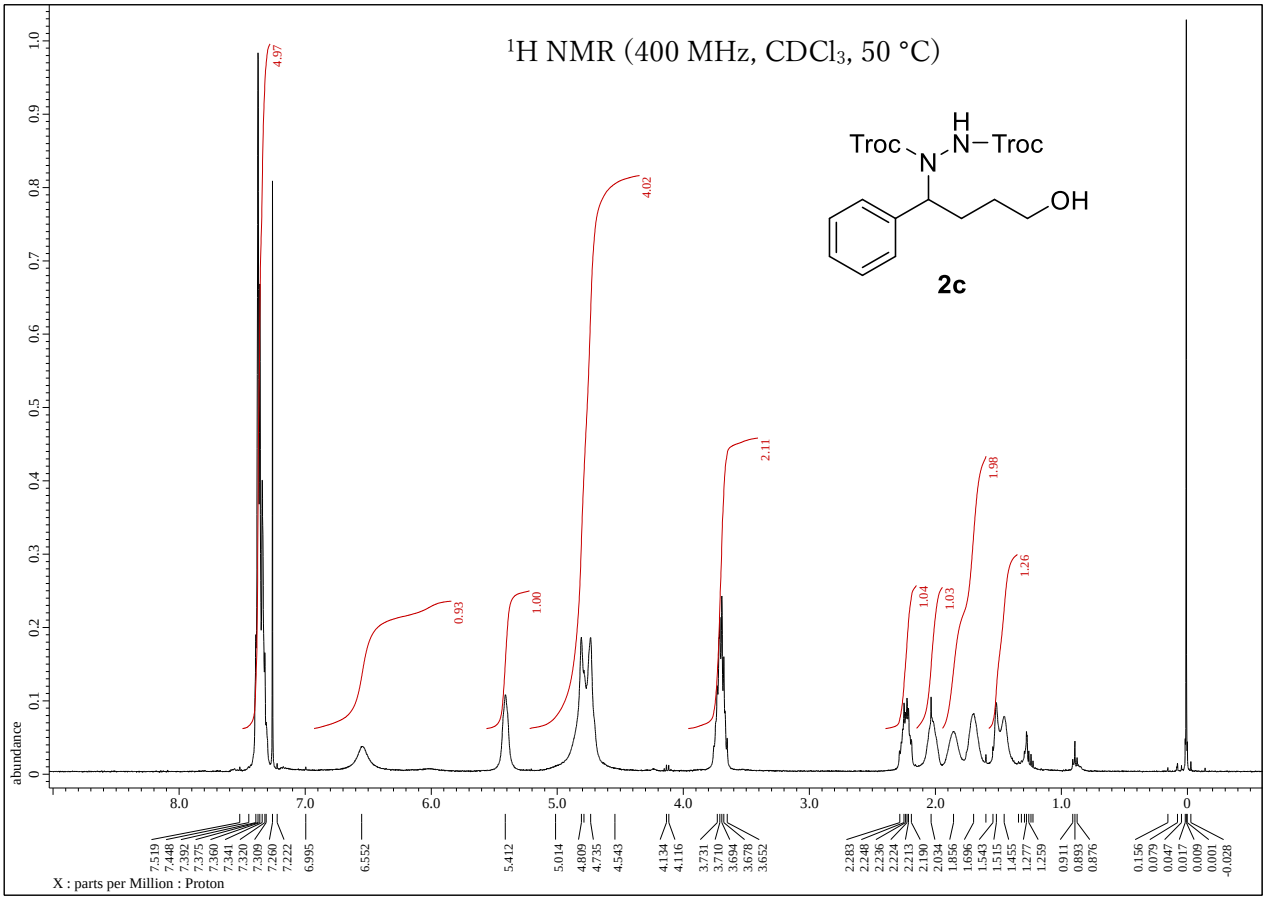
4. NMR Charts

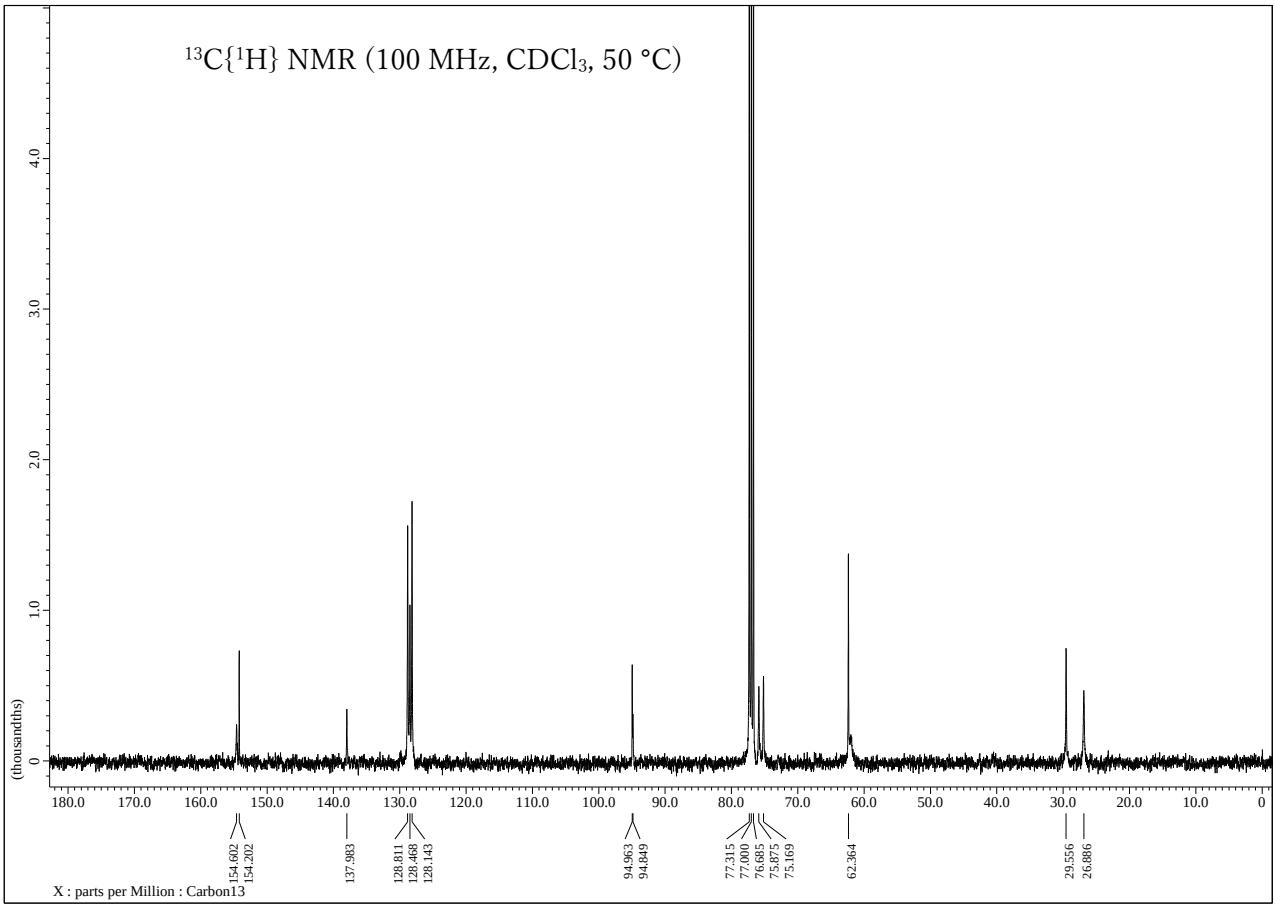

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N–Troc
OH
2a
abundance
X : parts per Million : Proton

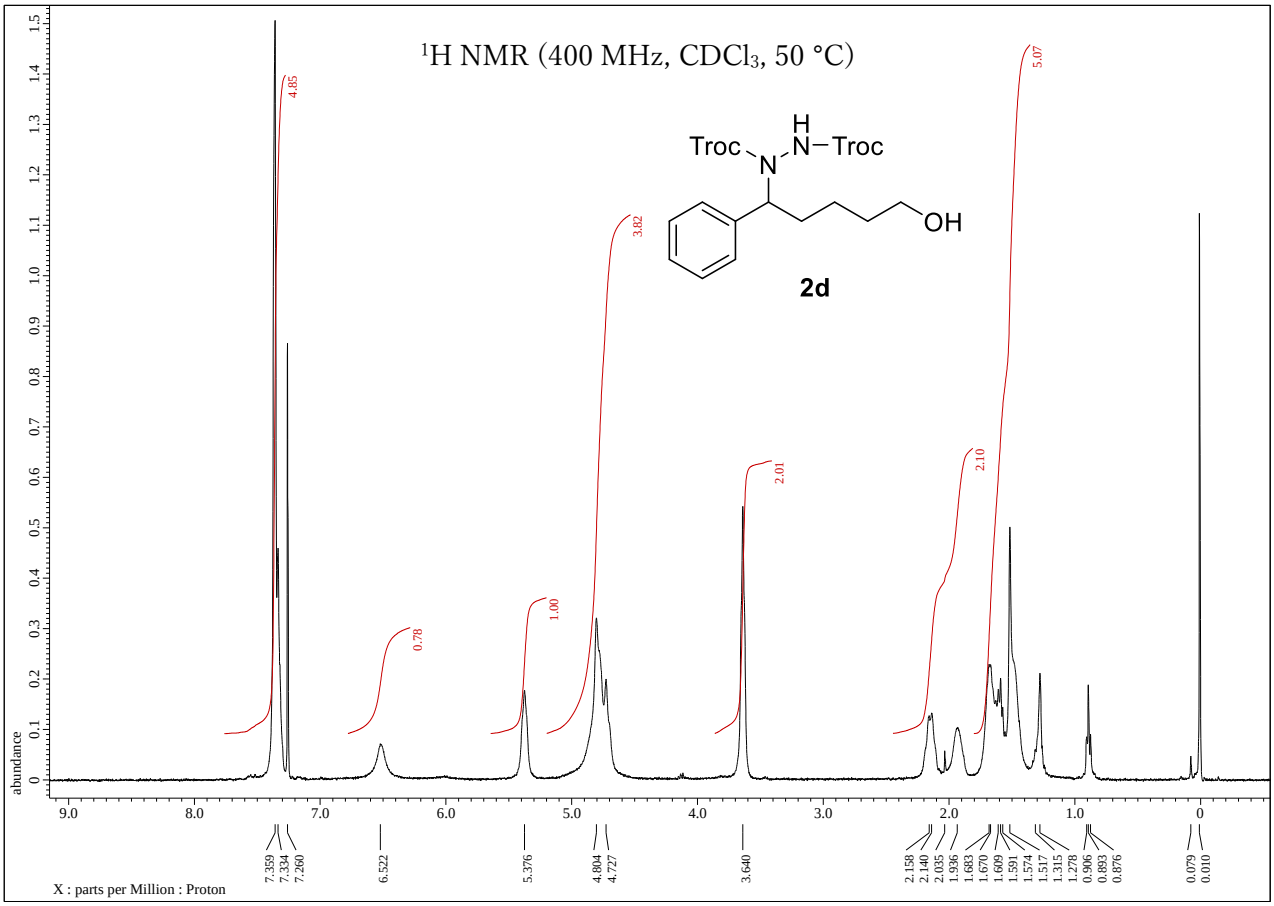

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

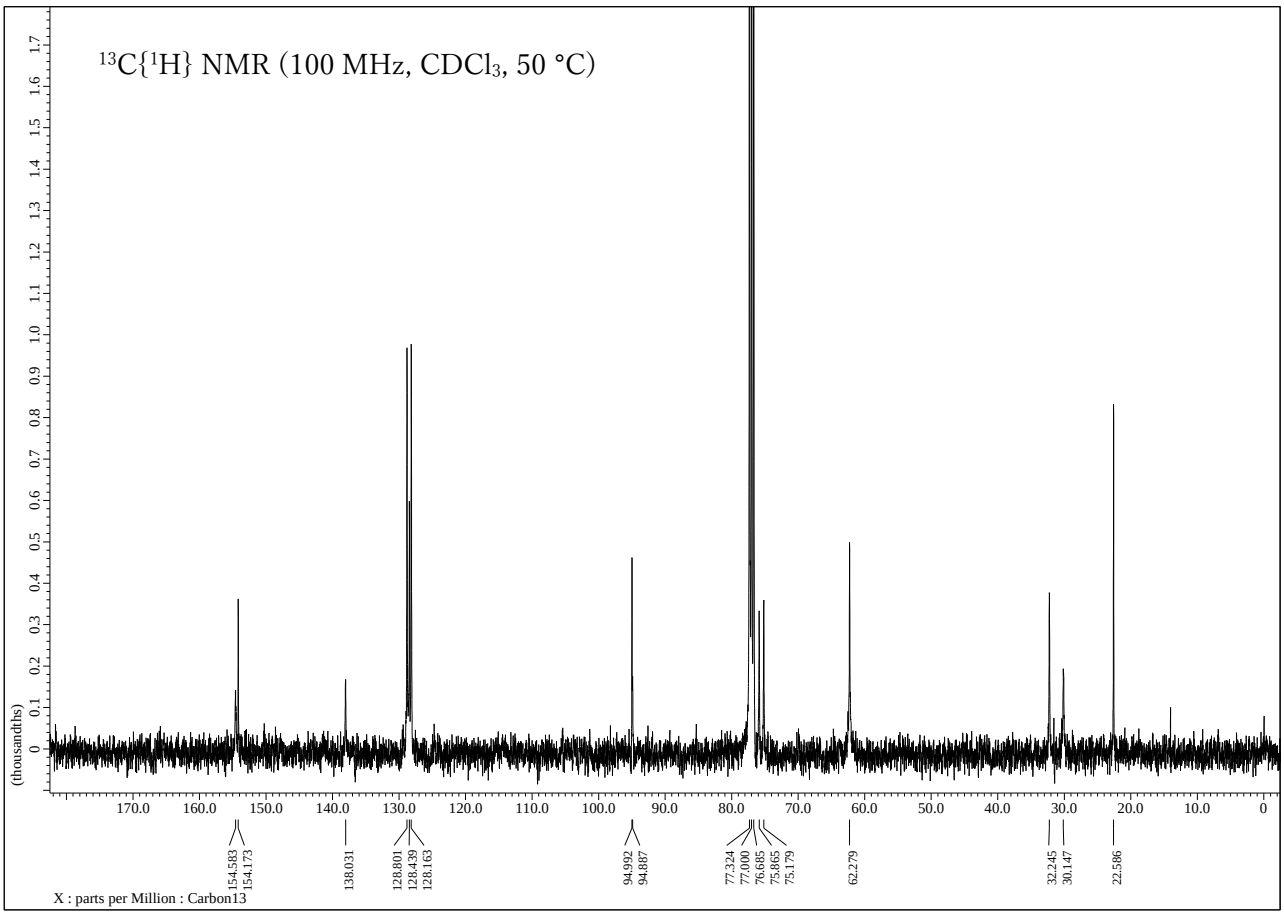

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N–Troc
OH
2b
5.03
4.30
1.81
0.98
1.00
0.85
abundance
X : parts per Million : Proton

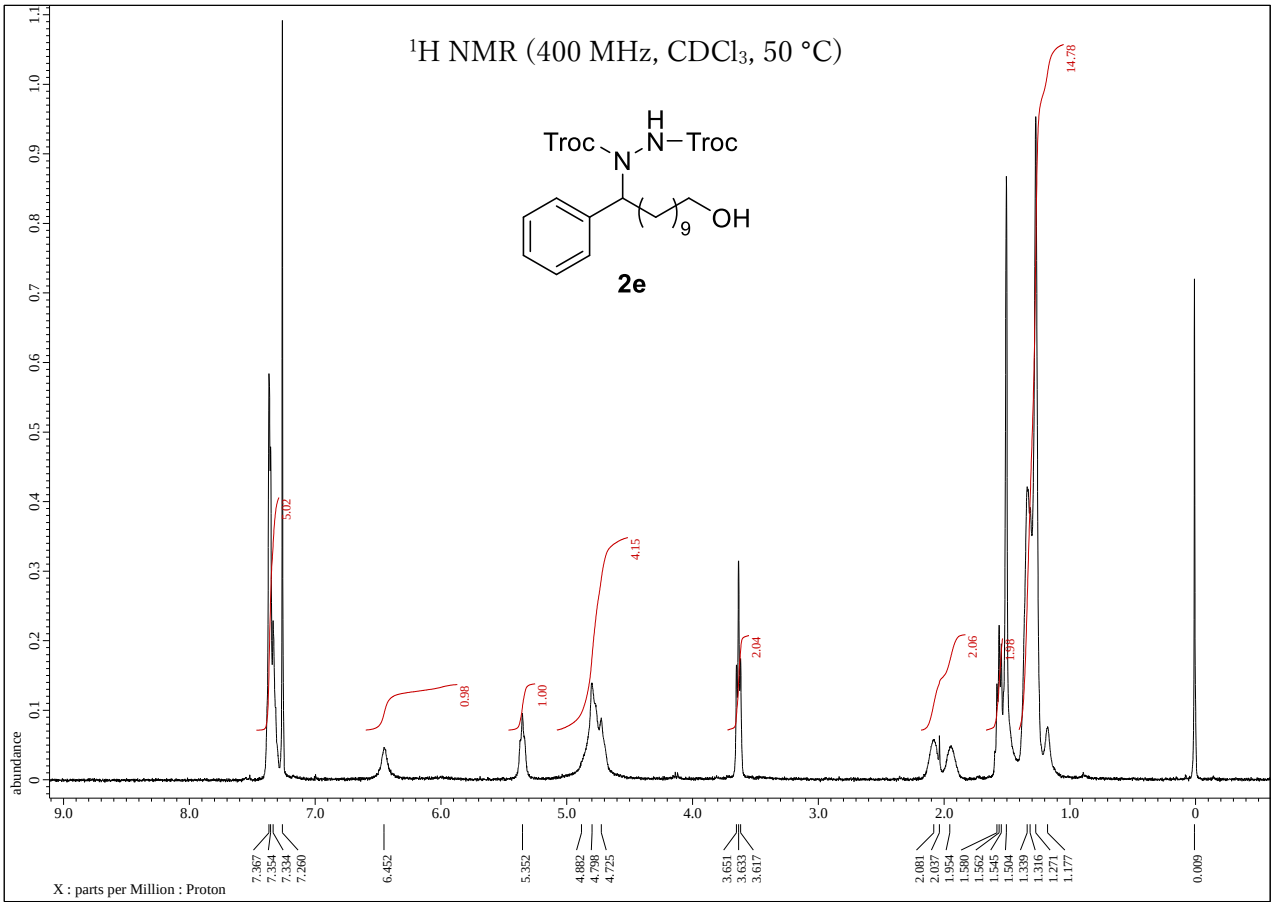

^{13}C{^{1}H} NMR (100 MHz, toluene-d_8, 100 °C)
(thousandths)
X : parts per Million : Carbon13

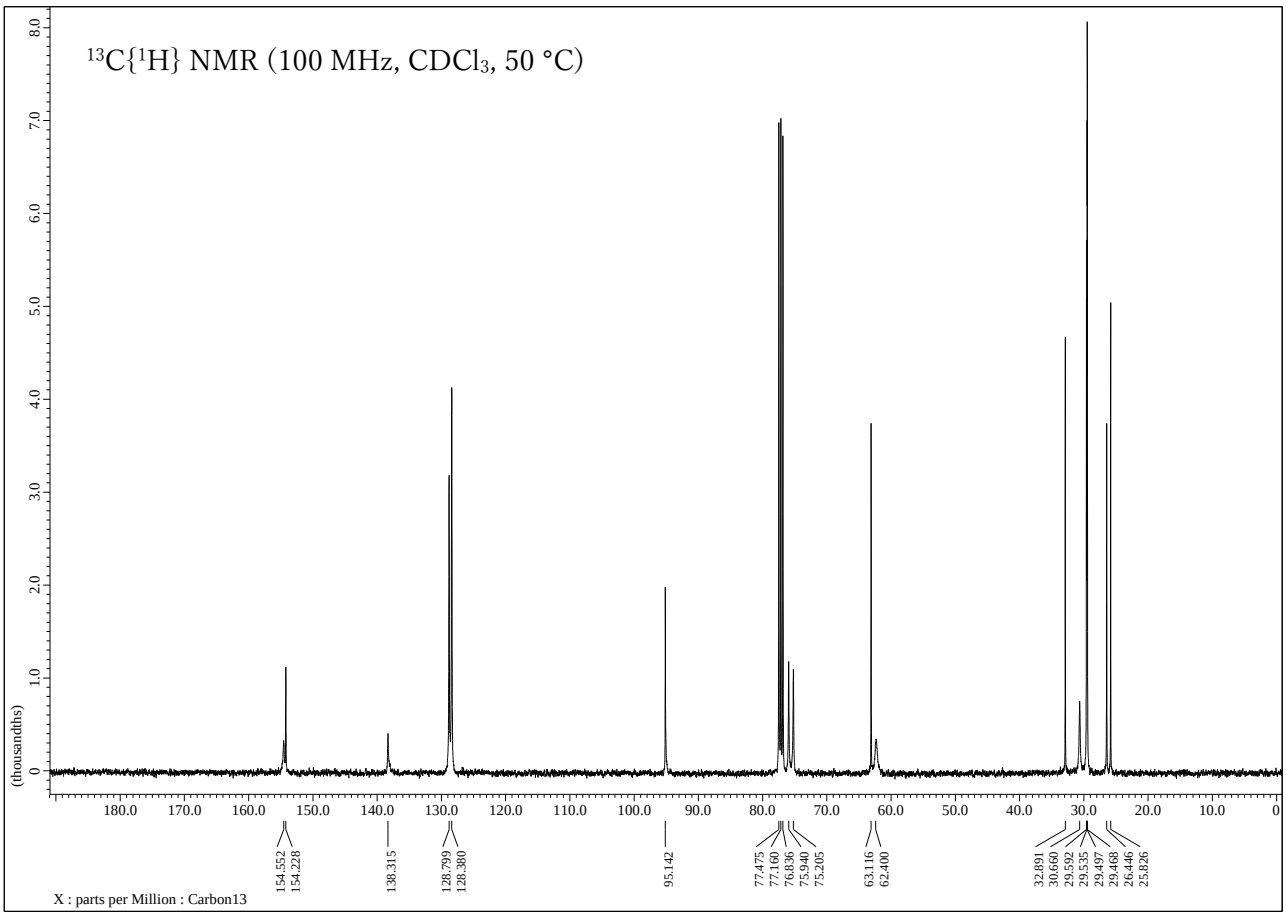

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
2c
4.97
0.93
1.00
4.02
2.11
1.04
1.03
1.98
1.26
abundance
X : parts per Million : Proton

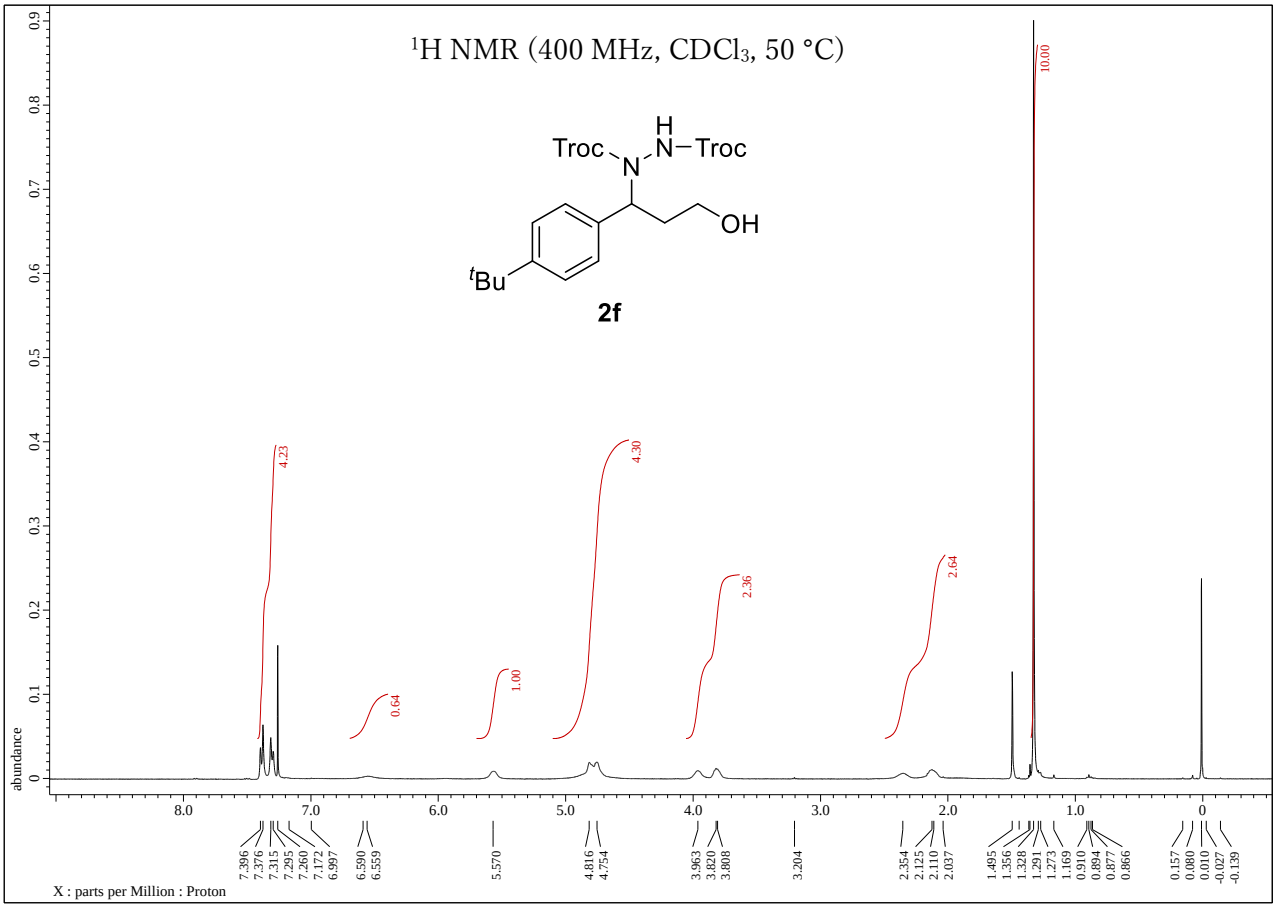

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

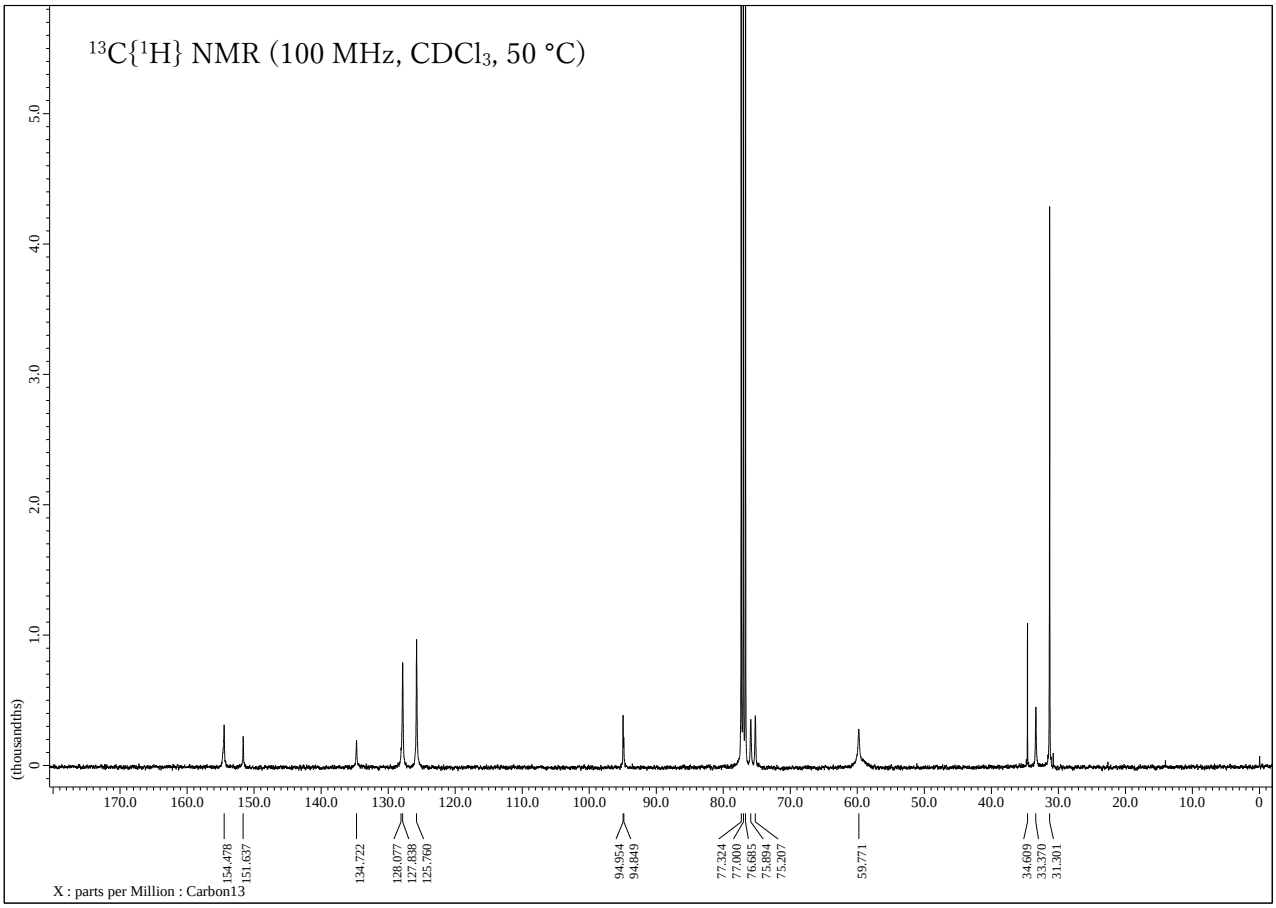

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N–Troc
OH
2d
4.85
0.78
1.00
3.82
2.01
2.10
5.07
abundance
X : parts per Million : Proton
7.359
7.334
7.260
6.522
5.376
4.804
4.727
3.640
2.158
2.140
2.035
1.936
1.683
1.670
1.609
1.591
1.574
1.517
1.315
1.278
0.906
0.893
0.876
0.079
0.010

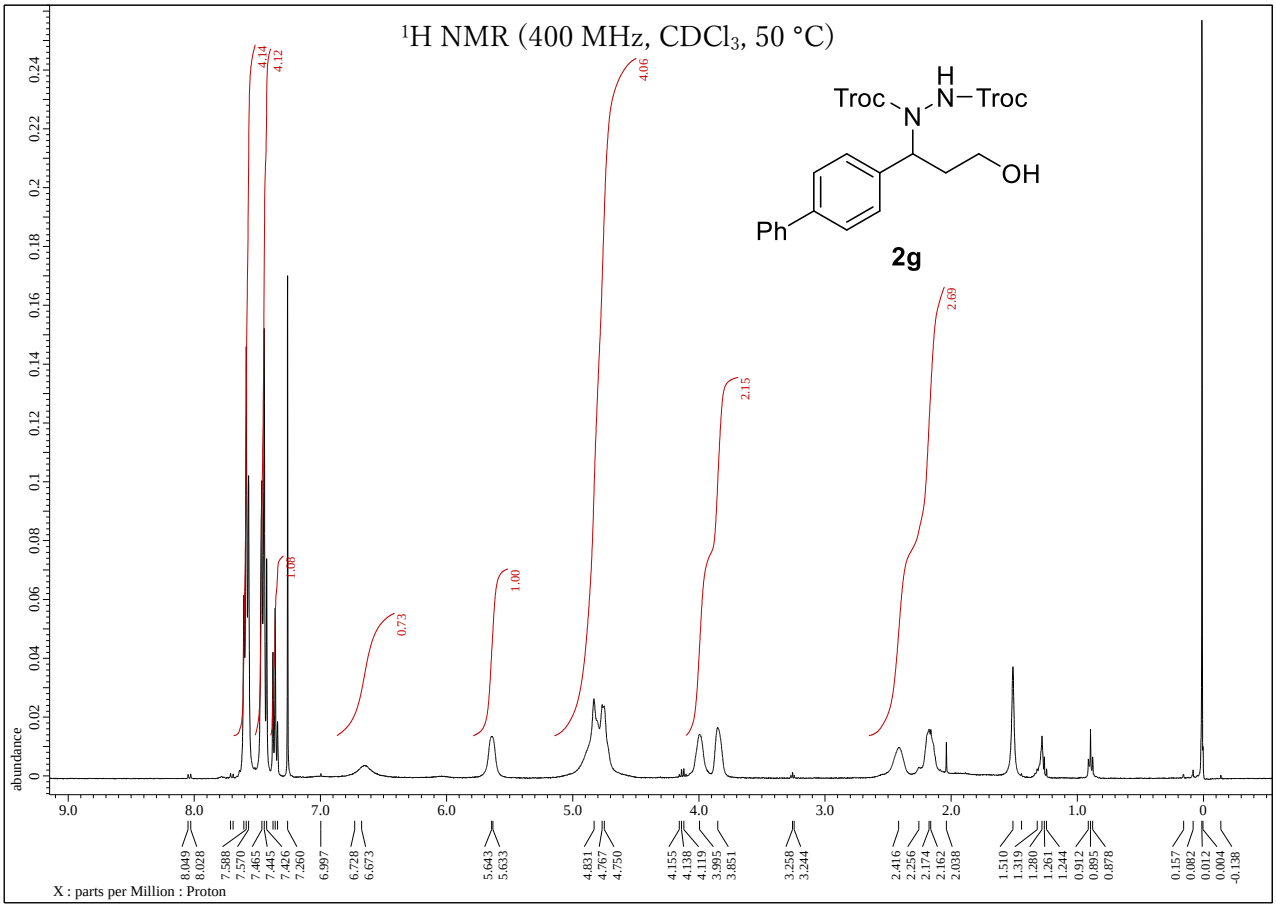

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13
154.583
154.173
136.031
128.801
128.439
128.163
94.992
94.887
77.324
77.000
76.685
75.865
75.179
62.279
32.245
30.147
22.586

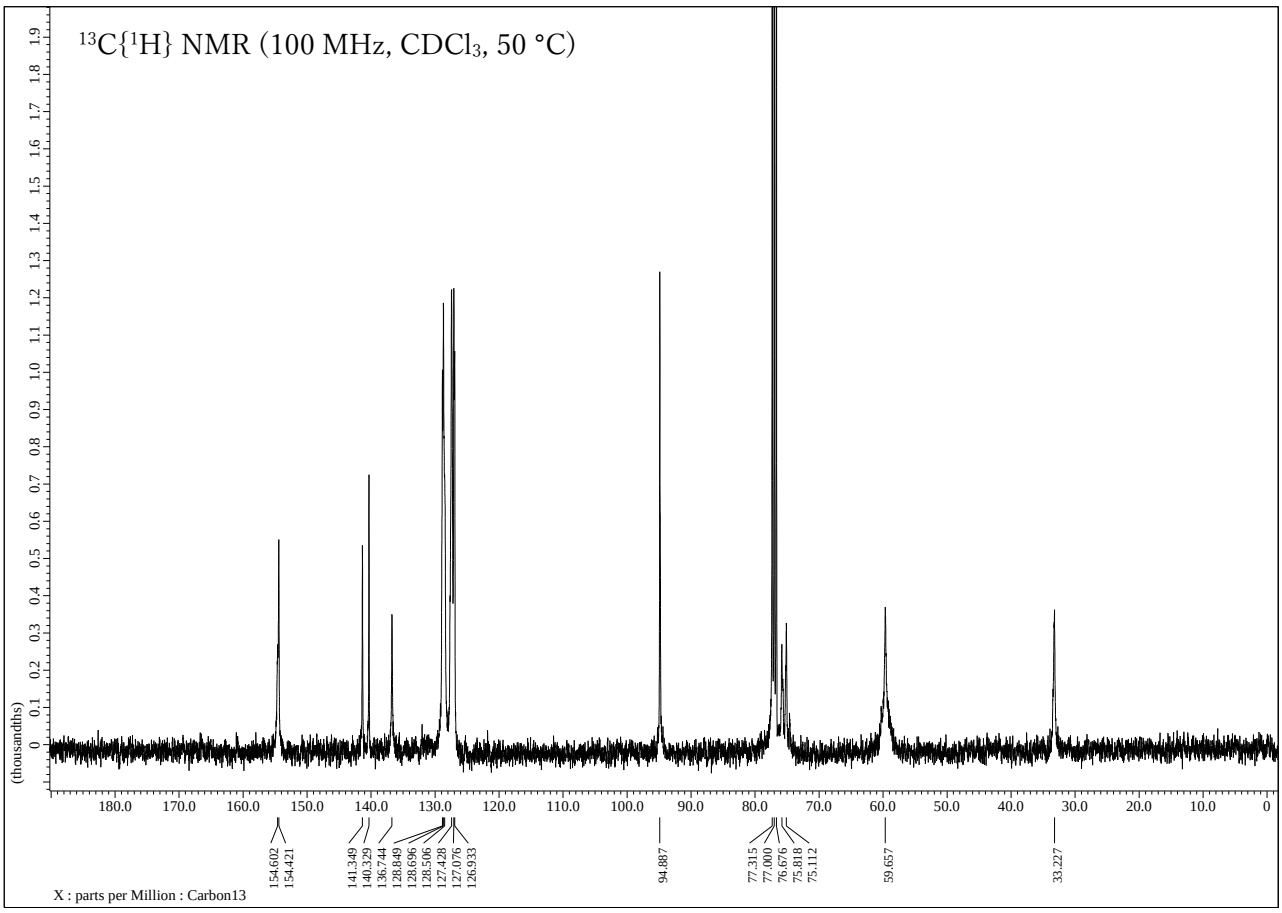

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N
Troc
9
OH
2e
abundance
X : parts per Million : Proton

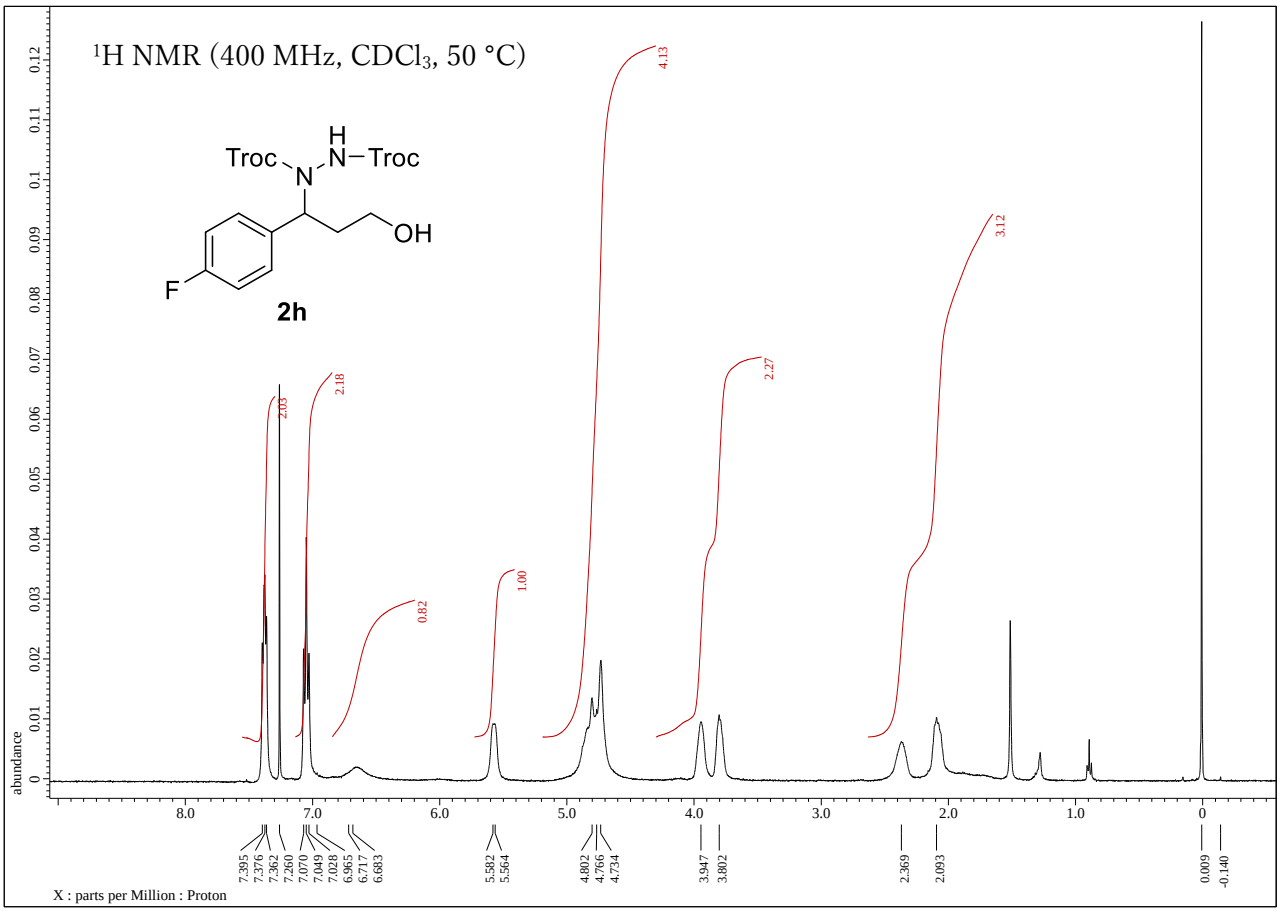

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

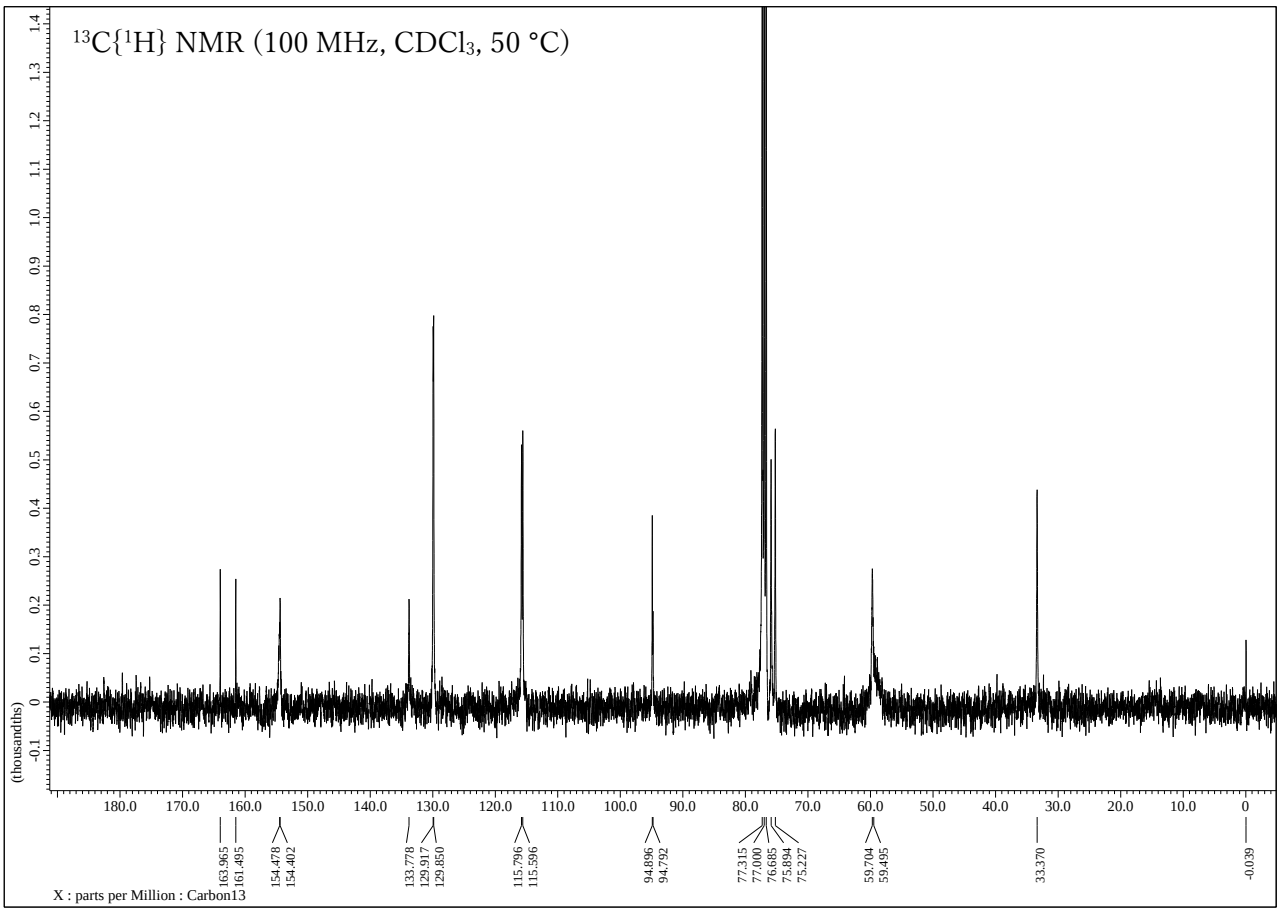

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N–Troc
OH
^{t}Bu
2f
abundance
X : parts per Million : Proton

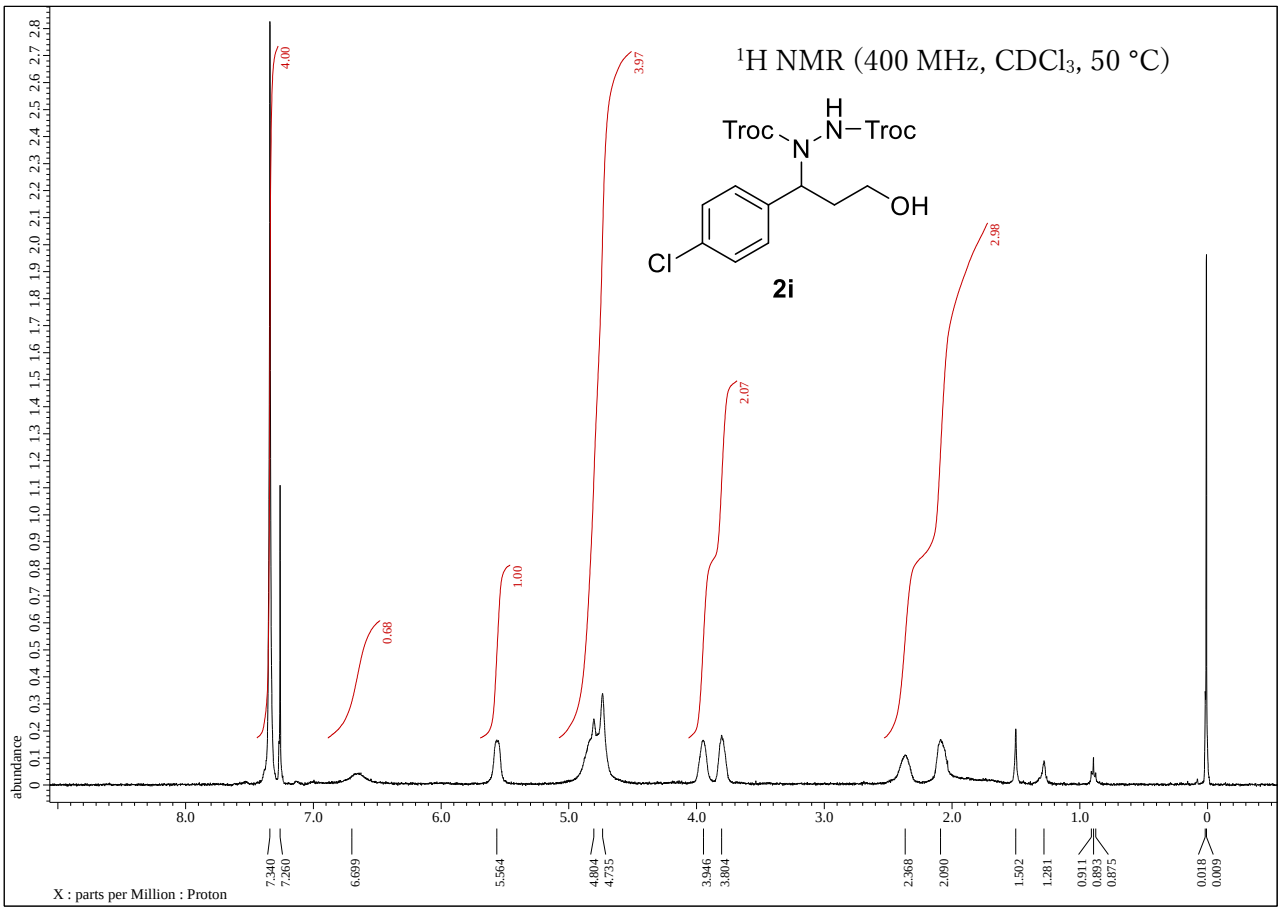

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

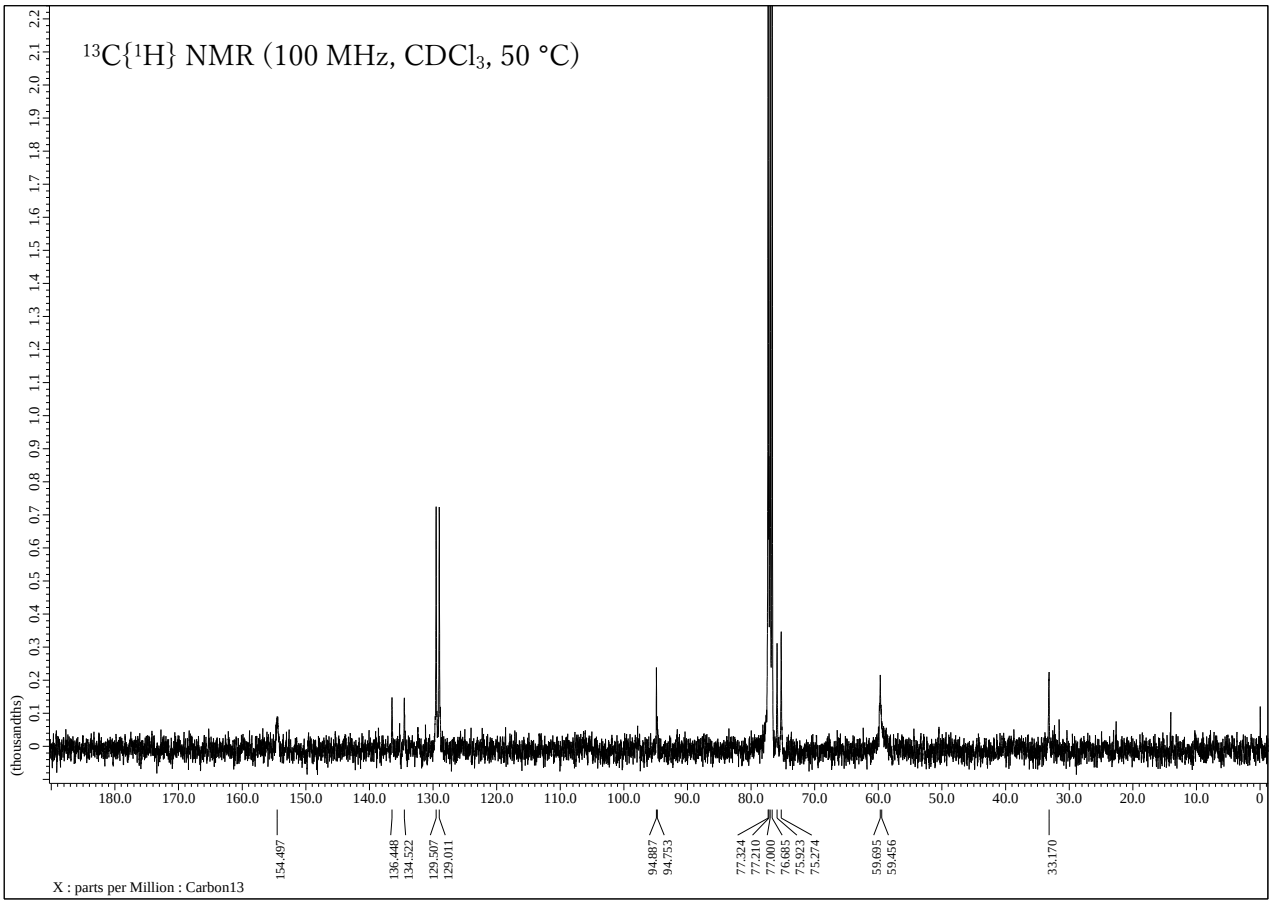

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N—Troc
OH
Ph
2g
4.14
4.12
4.06
2.69
2.15
1.08
1.00
0.73
abundance
X : parts per Million : Proton

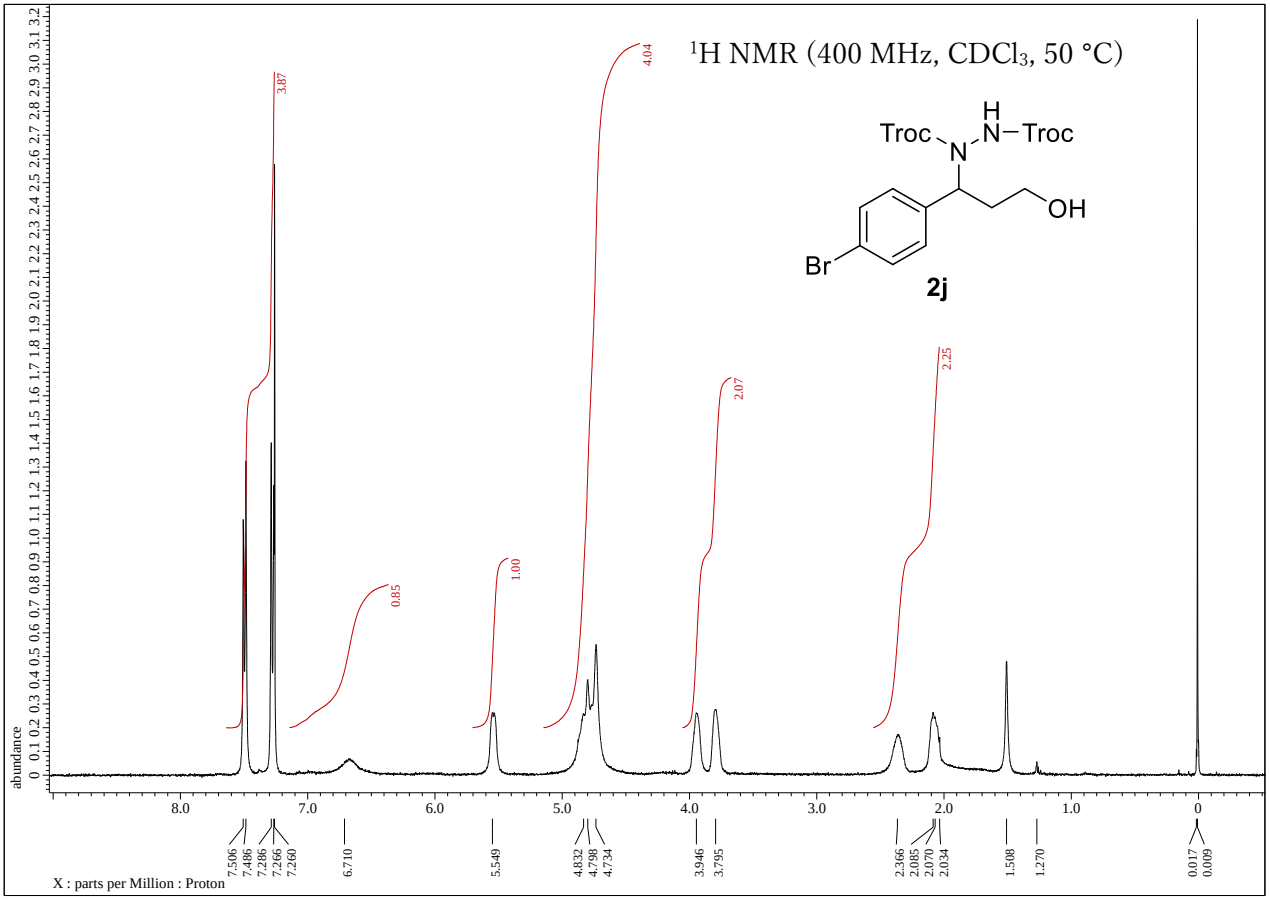

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

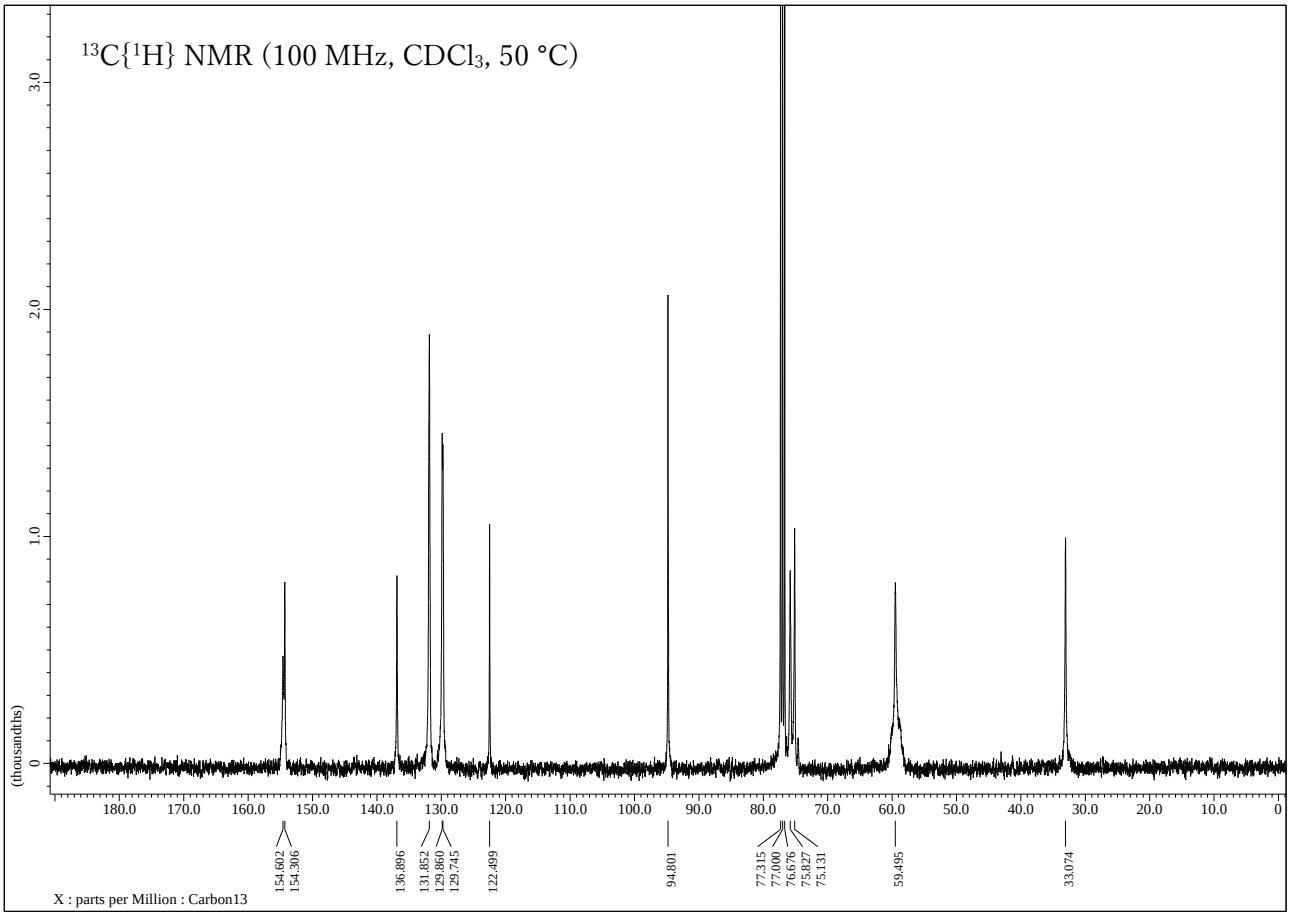

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N–Troc
OH
F
2h
4.13
3.12
2.27
2.18
2.03
1.00
0.82
abundance
X : parts per Million : Proton

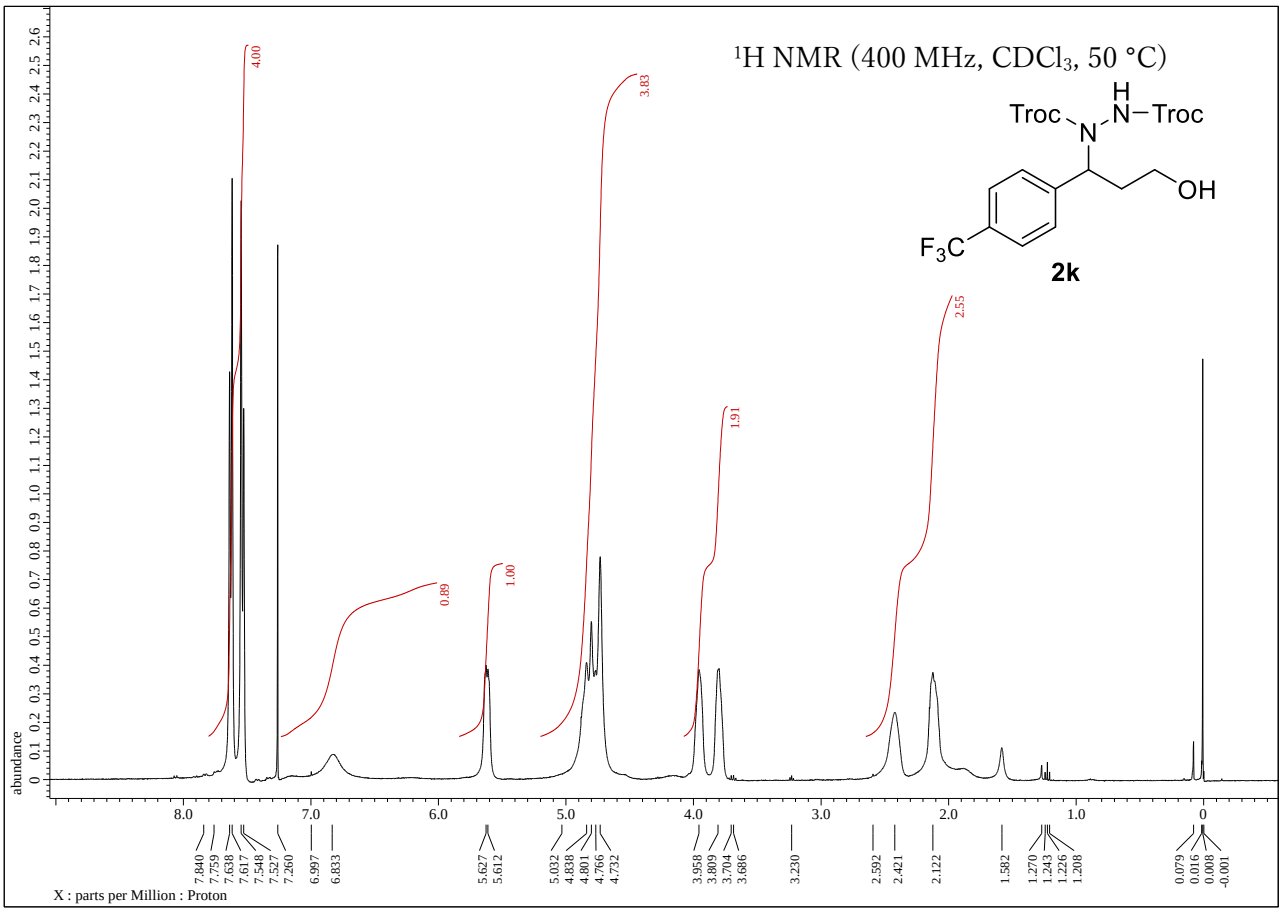

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

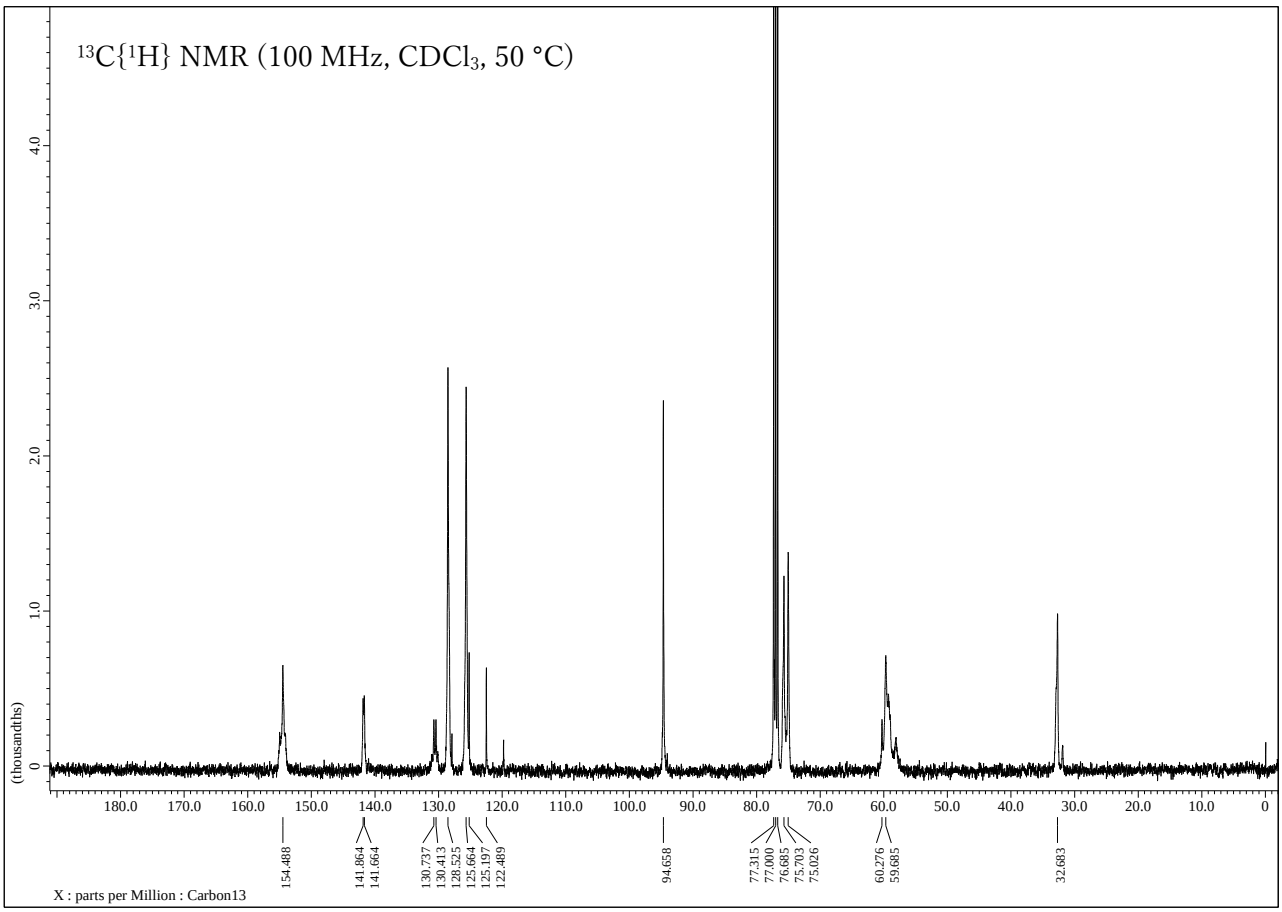

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
Cl
2i
4.00
3.97
2.98
2.07
1.00
0.68
abundance
X : parts per Million : Proton
7.340
7.260
6.699
5.564
4.804
4.735
3.946
3.804
2.368
2.090
1.502
1.281
0.911
0.893
0.875
0.018
0.009

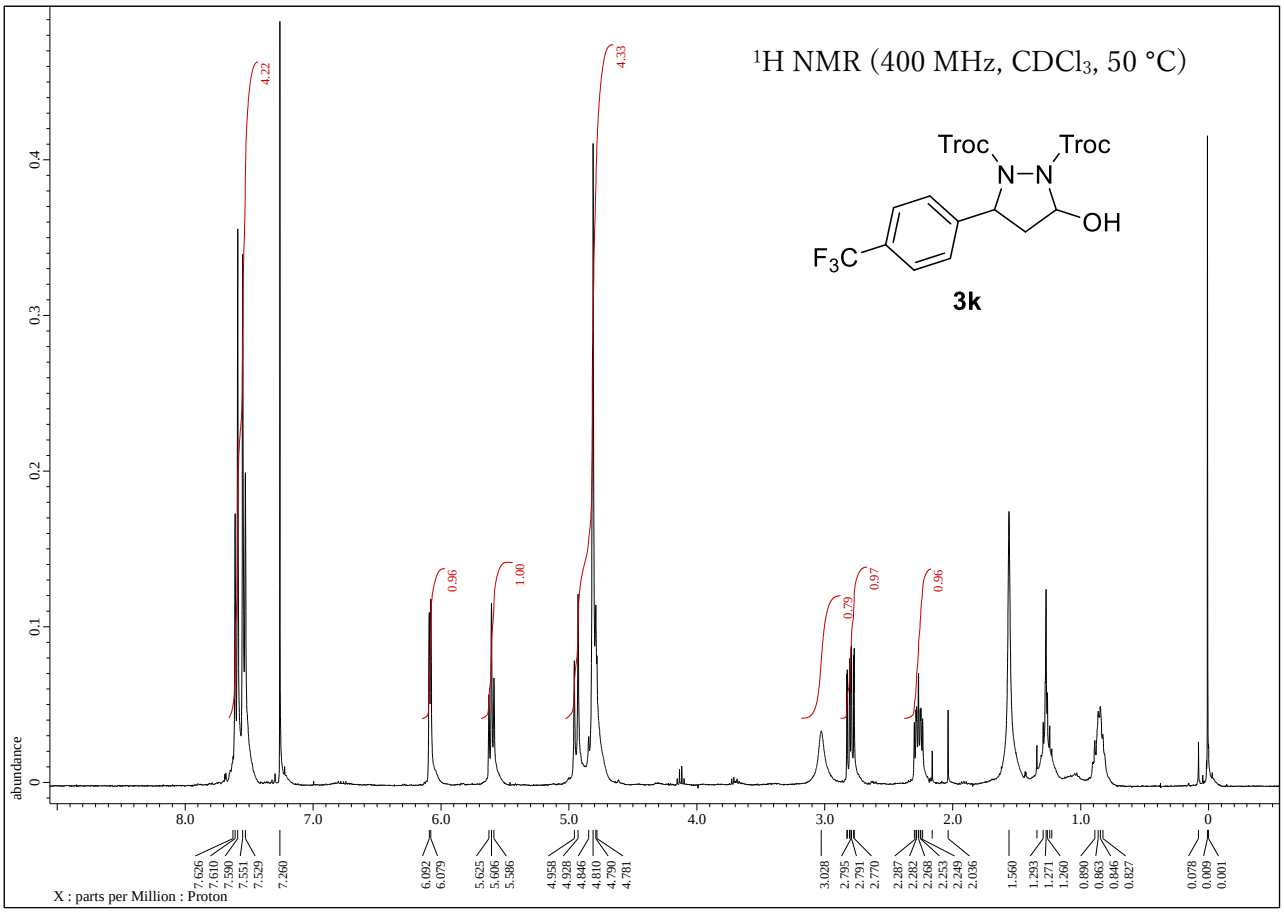

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13
154.497
136.448
134.522
129.507
129.011
94.887
94.753
77.324
77.210
77.000
76.685
75.923
75.274
59.695
59.456
33.170

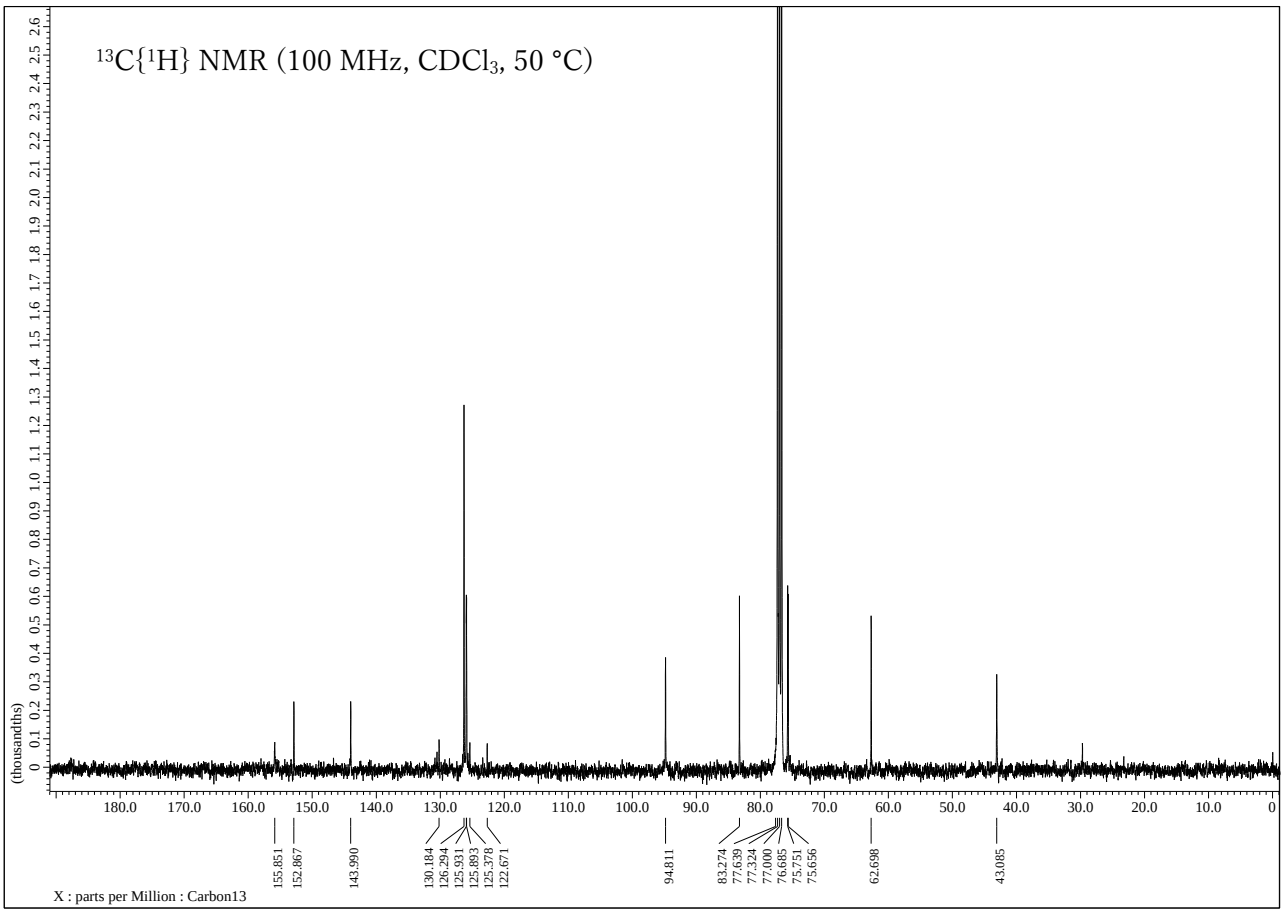

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
Br
2j
abundance
X : parts per Million : Proton

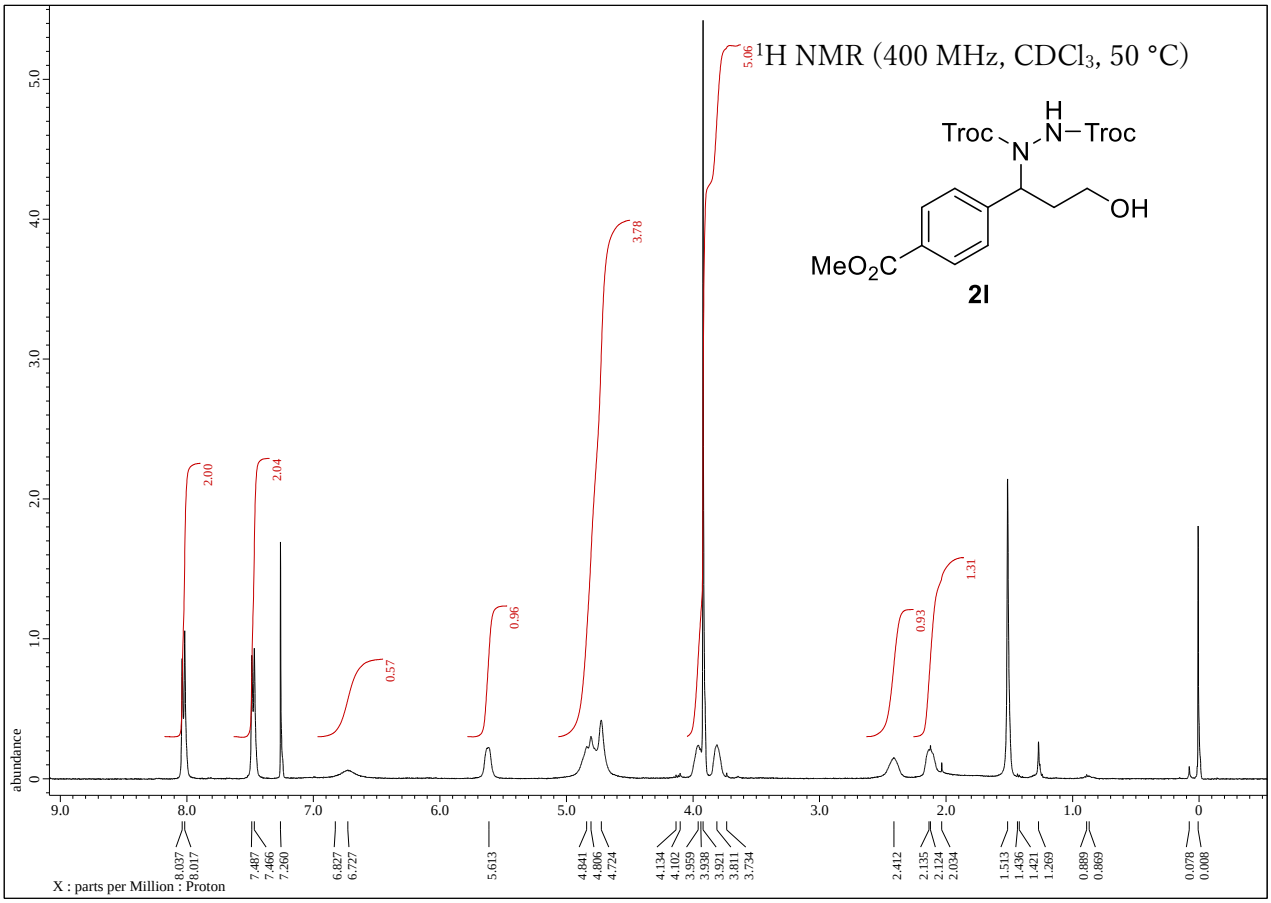

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

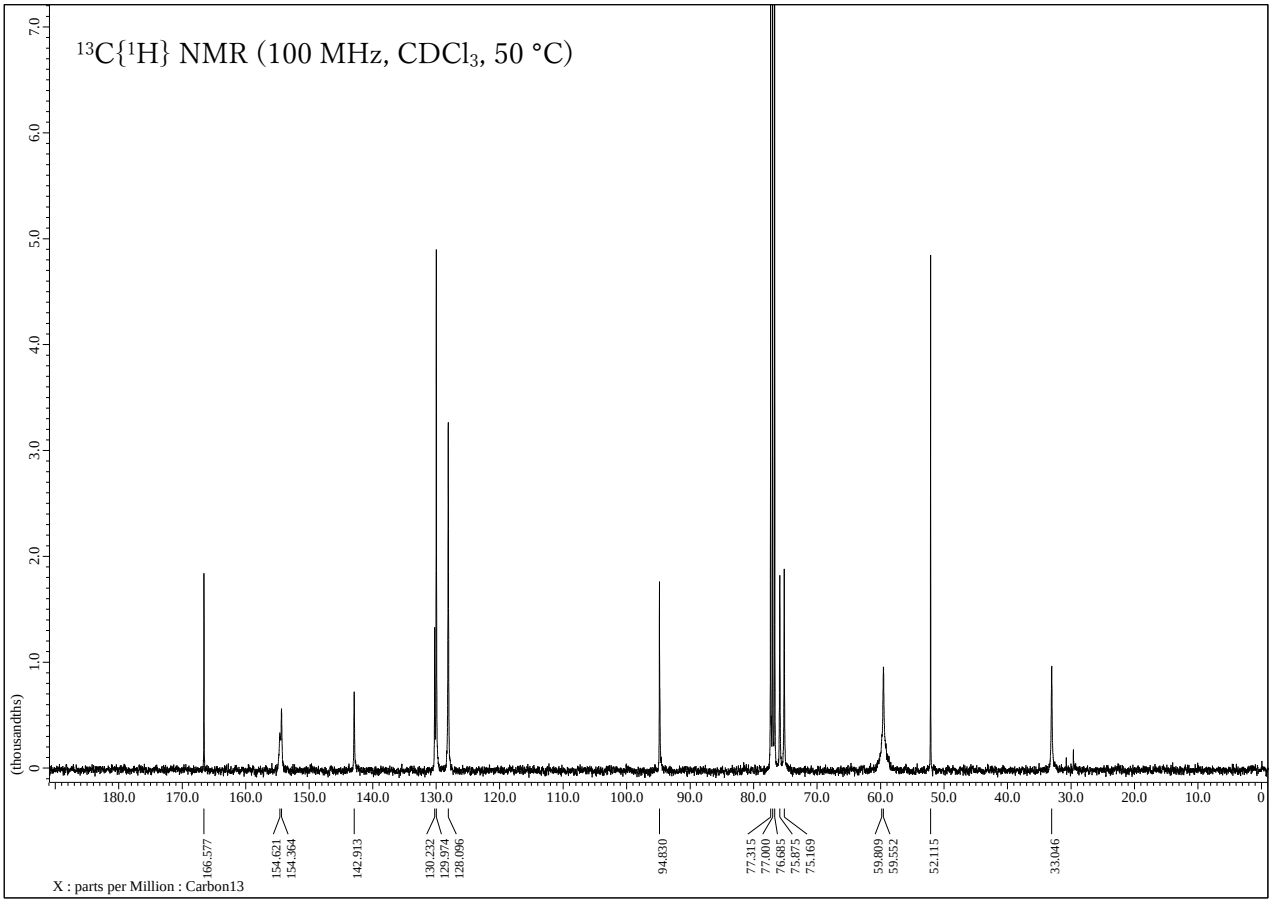

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
F_3C
2k
abundance
4.00
3.83
2.55
1.91
1.00
0.89
X : parts per Million : Proton

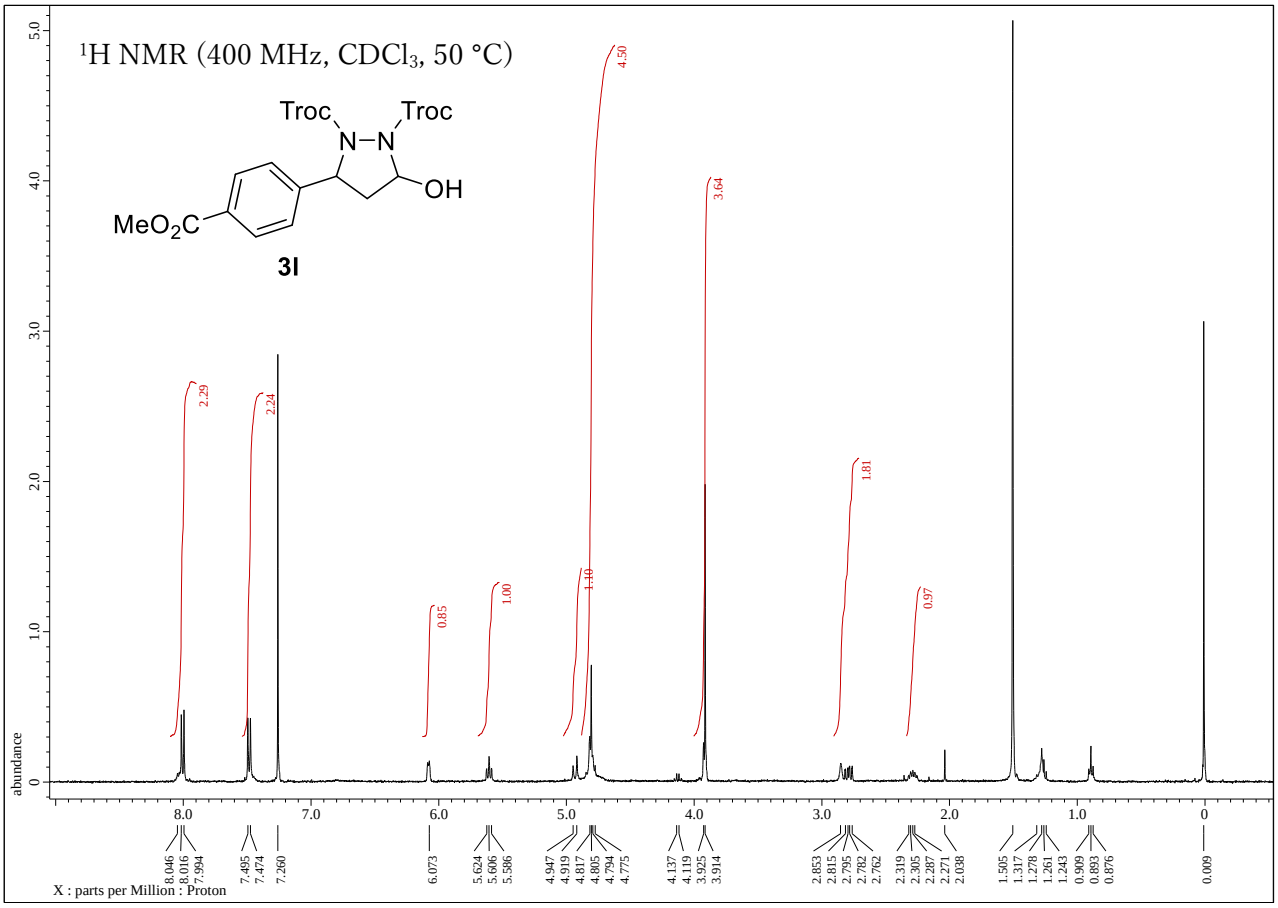

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

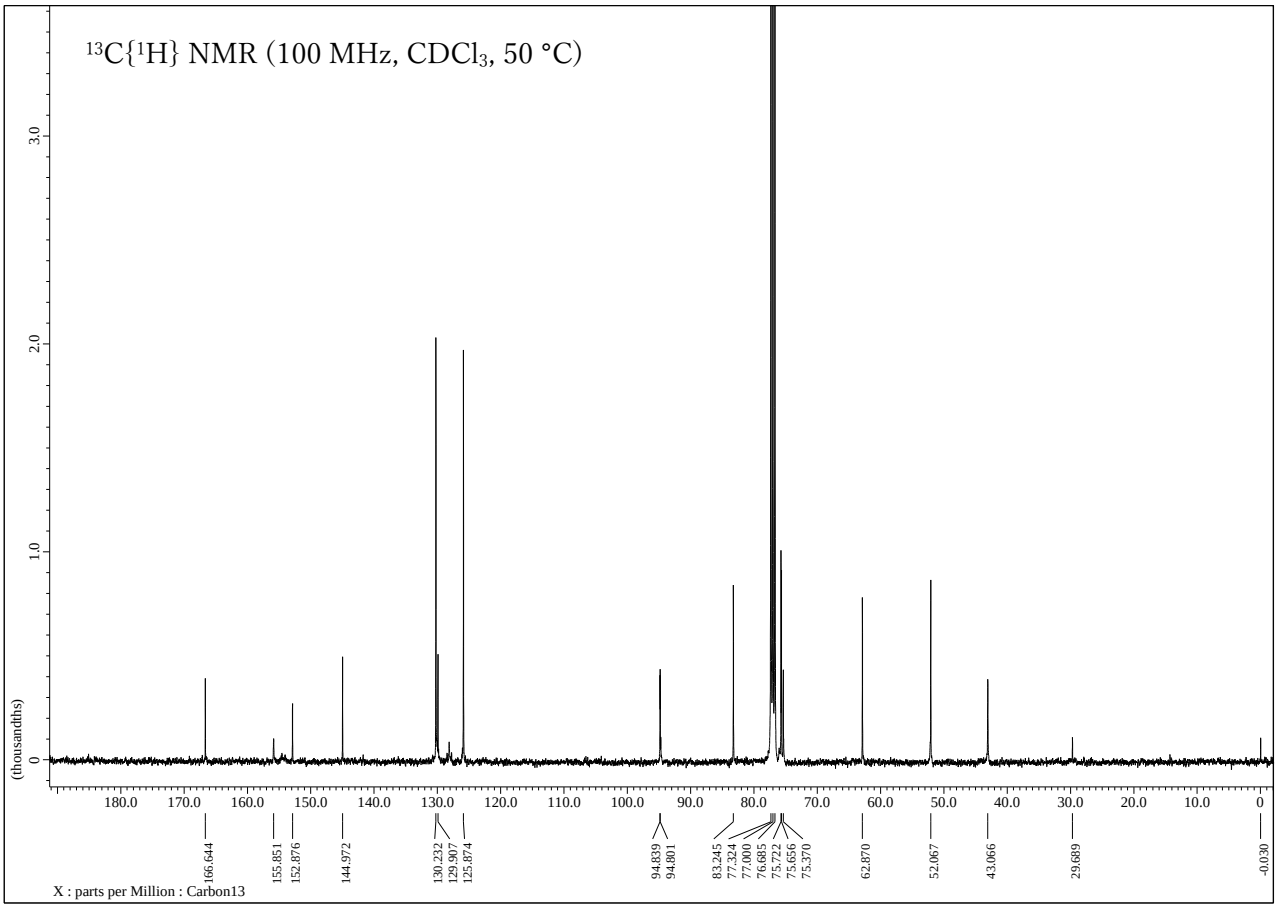

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
Troc
N–N
OH
F_3C
3k
abundance
X : parts per Million : Proton

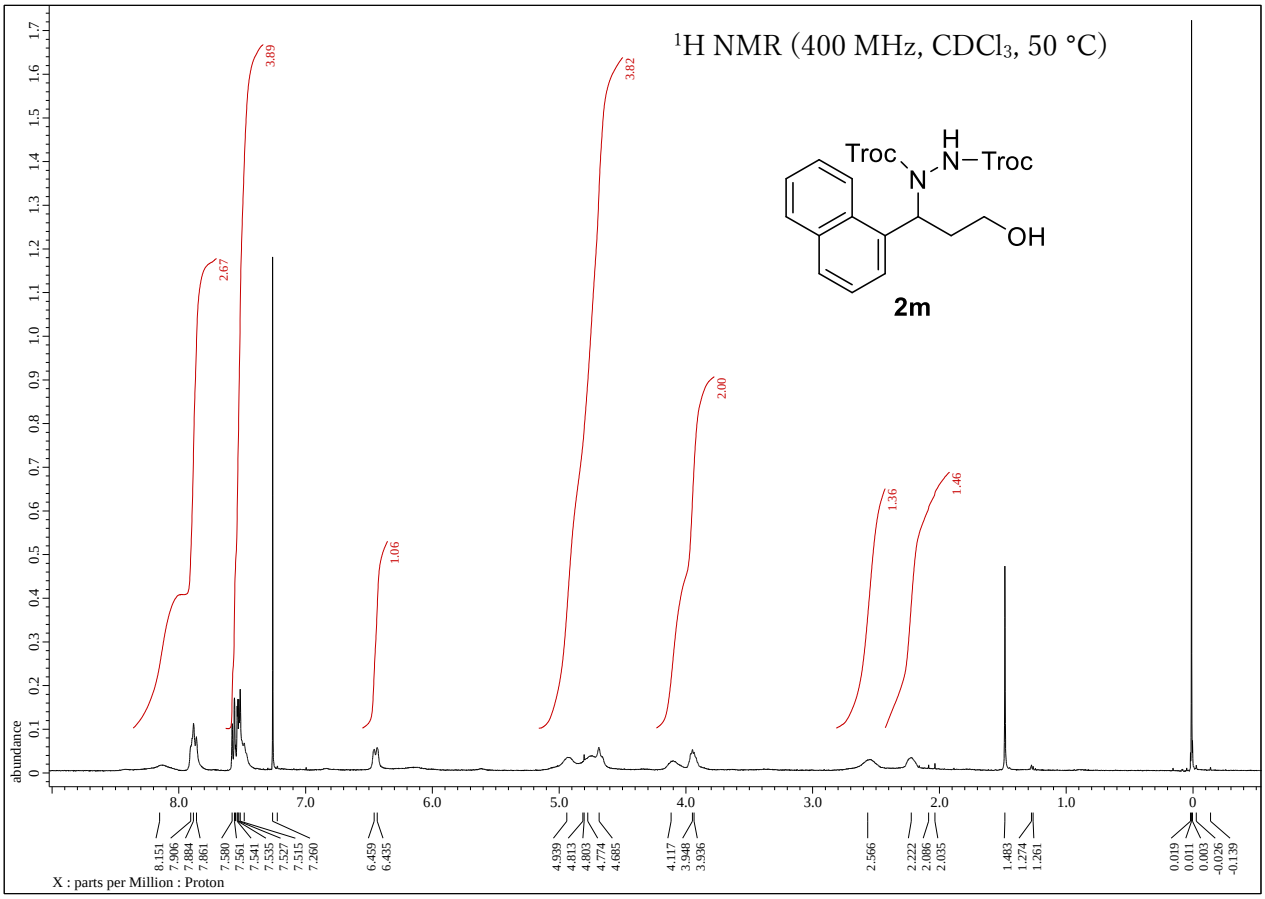

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

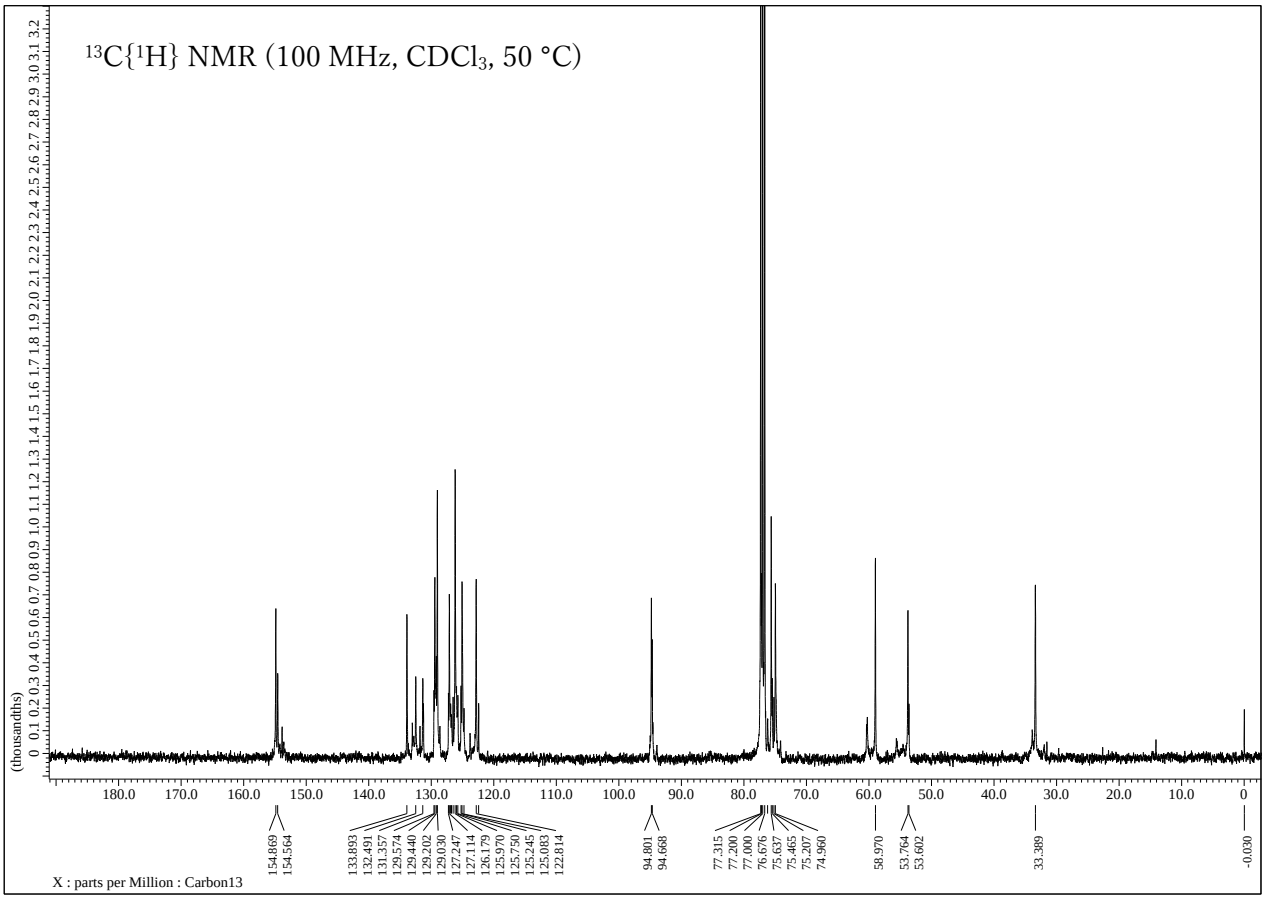

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N–Troc
OH
MeO$_2$C
2l
abundance
X : parts per Million : Proton

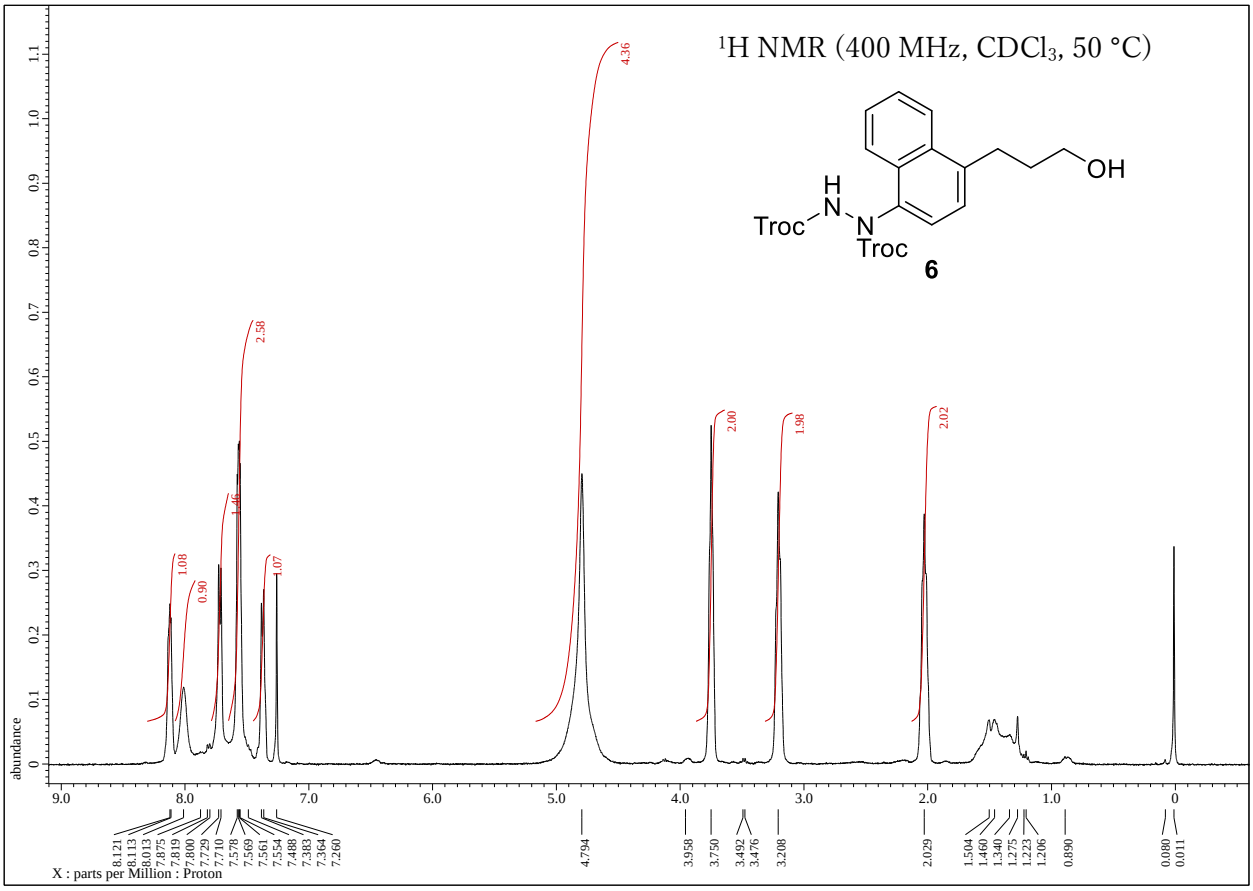

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

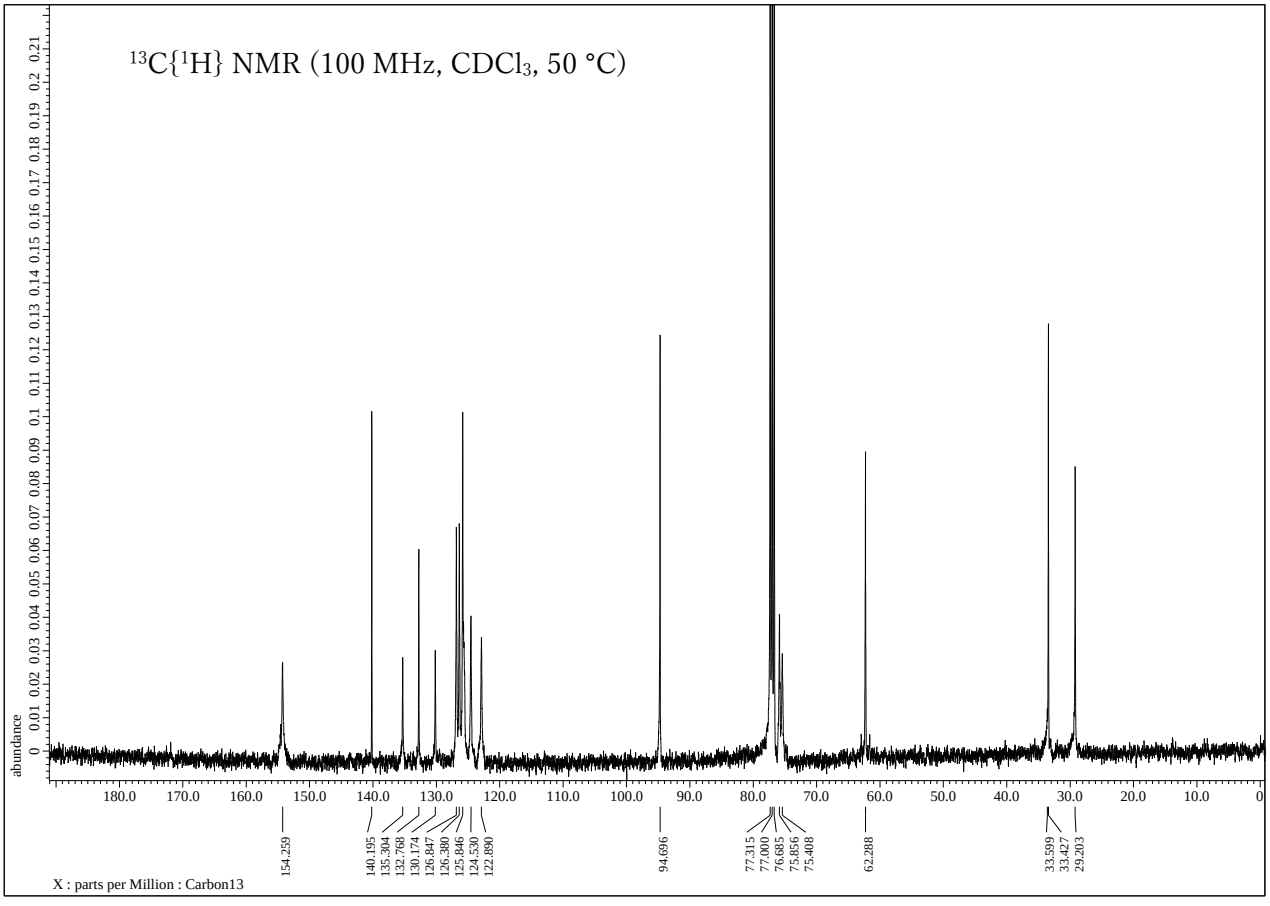

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N–N
Troc
OH
MeO_2C
3l
abundance
X : parts per Million : Proton

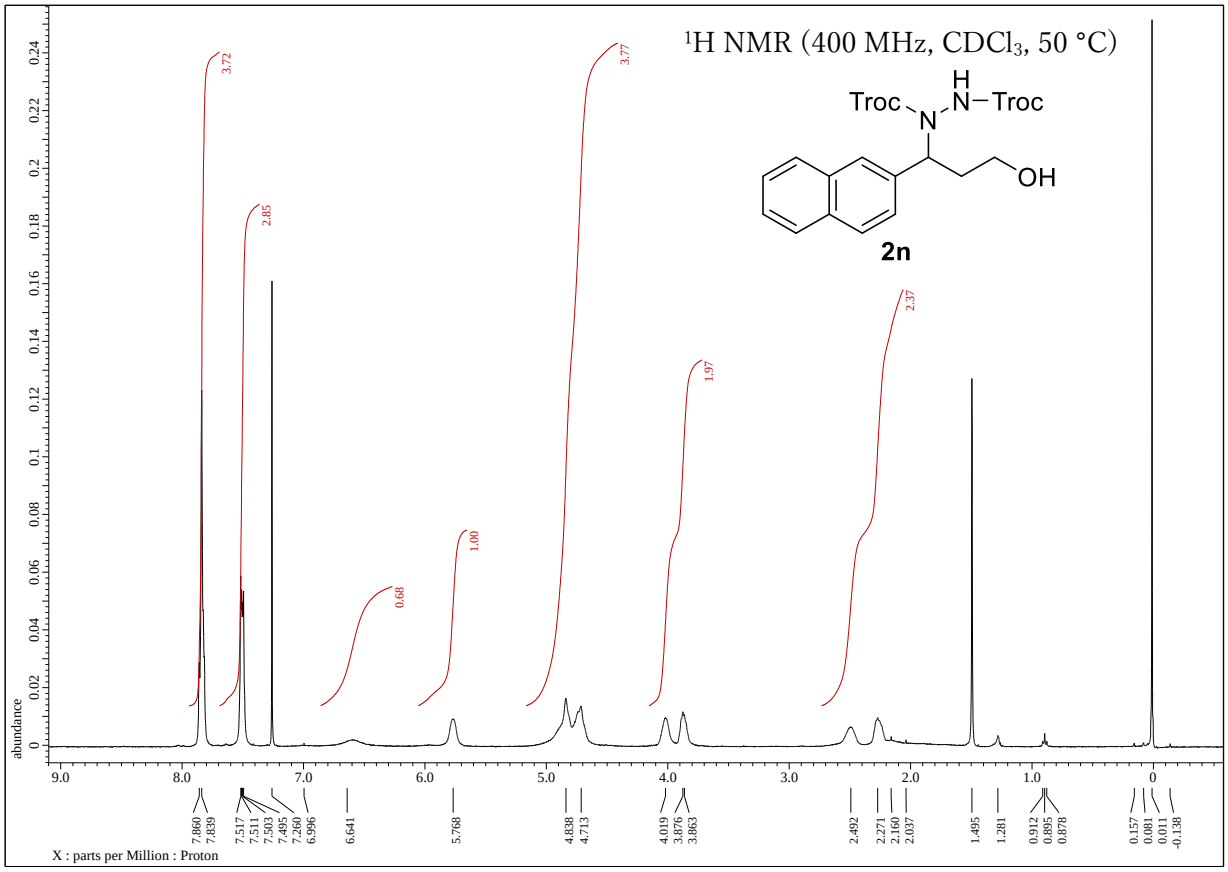

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

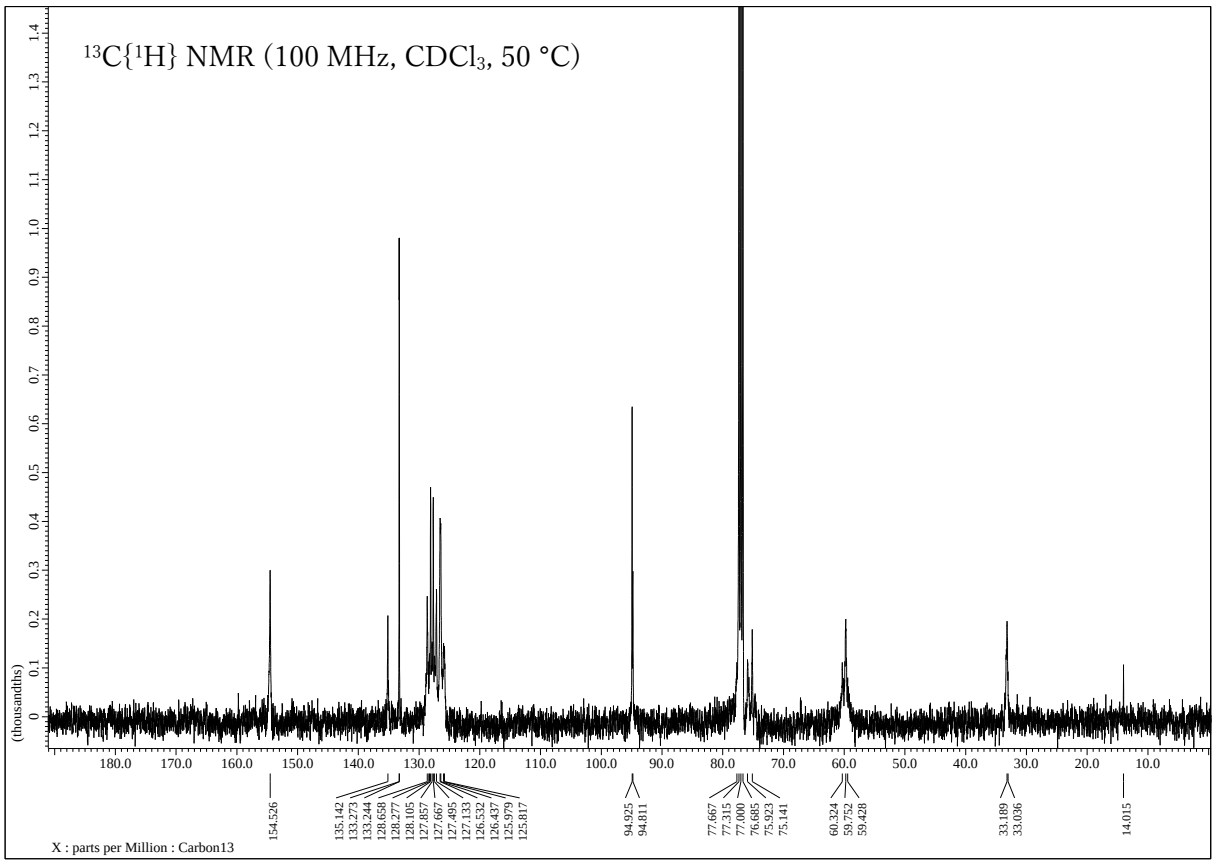

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N–Troc
OH
2m
3.89
3.82
2.67
2.00
1.36
1.46
1.06
abundance
X : parts per Million : Proton

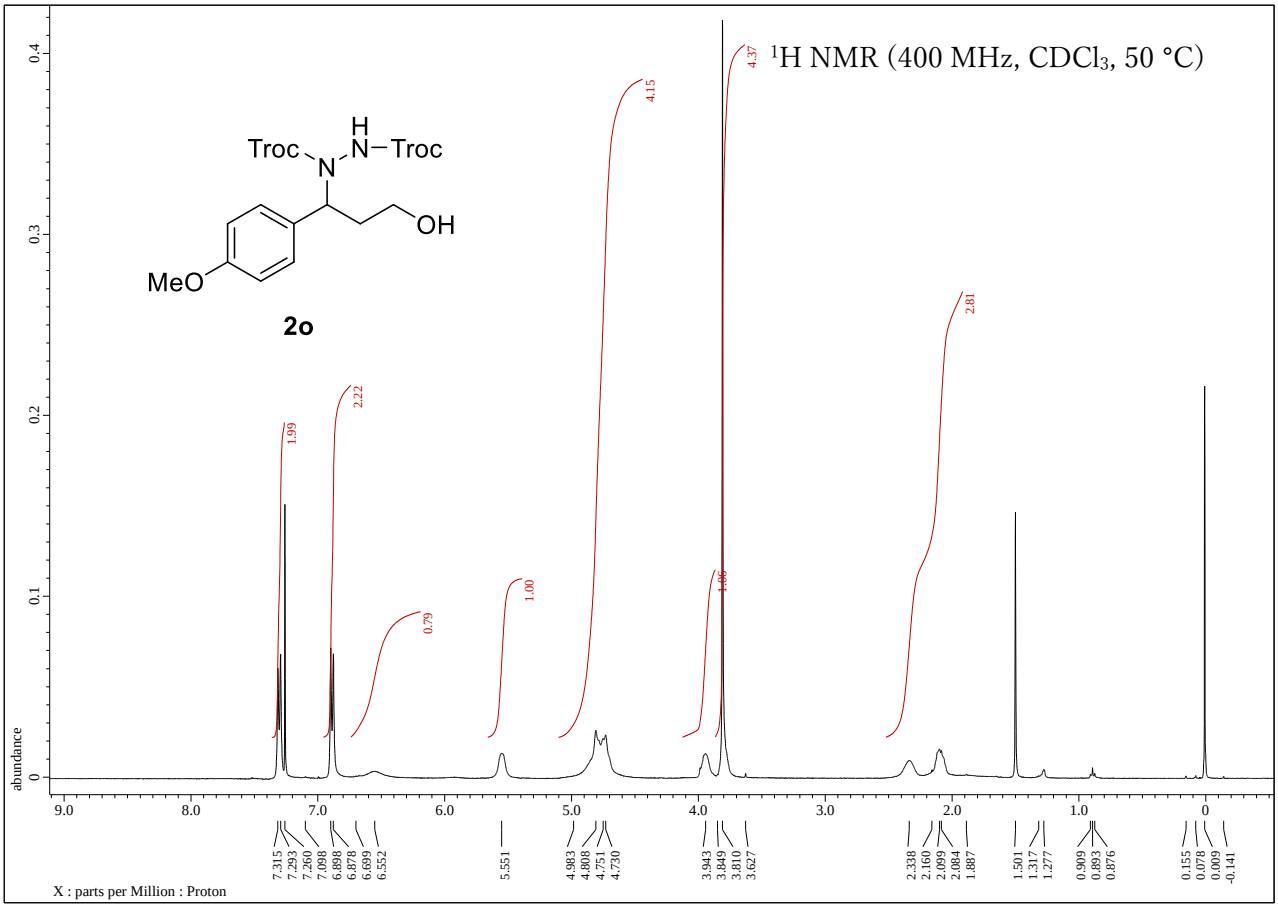

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

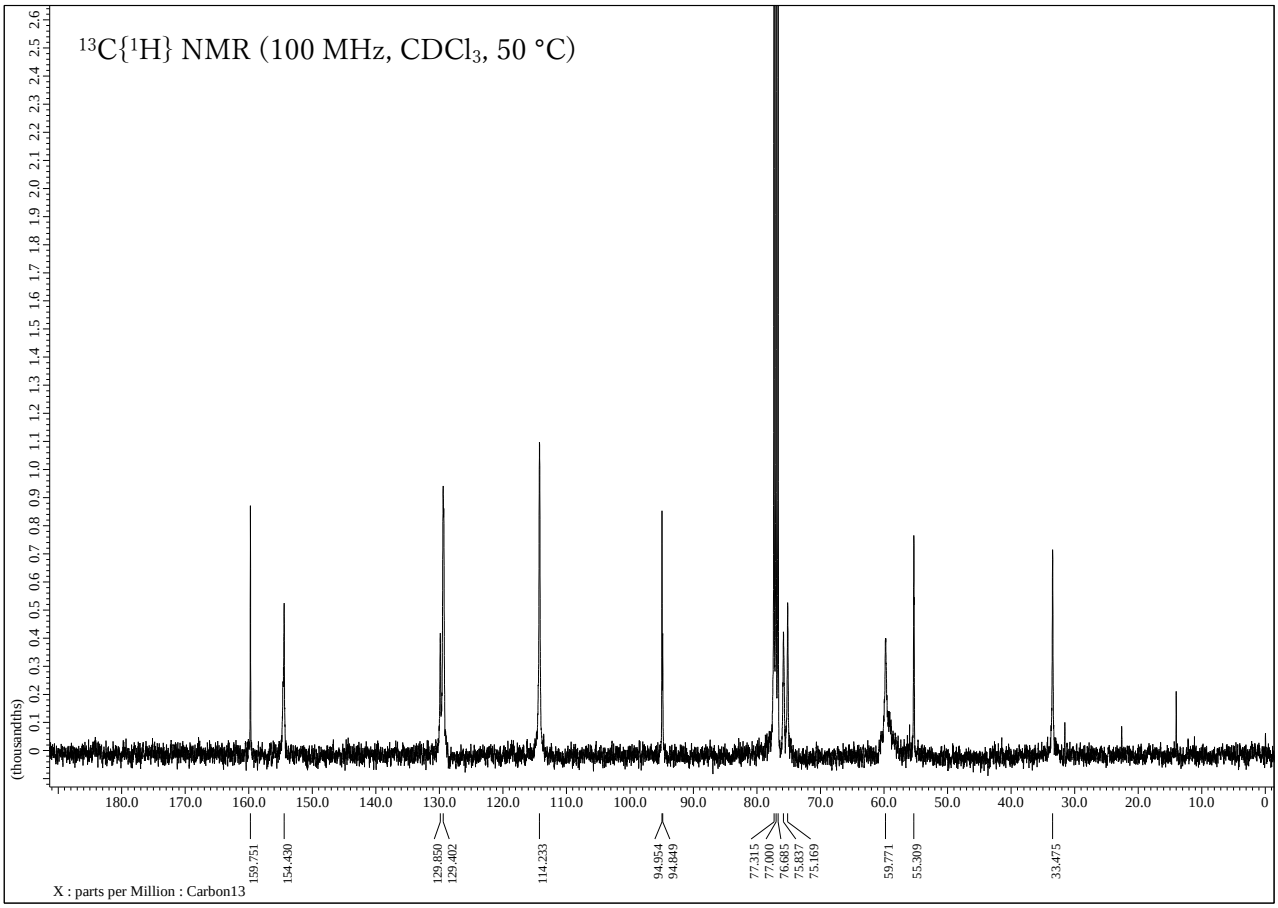

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
OH
H
N
N
Troc
Troc
6
4.36
2.58
2.00
1.98
2.02
1.08
0.90
1.46
1.07
abundance
X : parts per Million : Proton

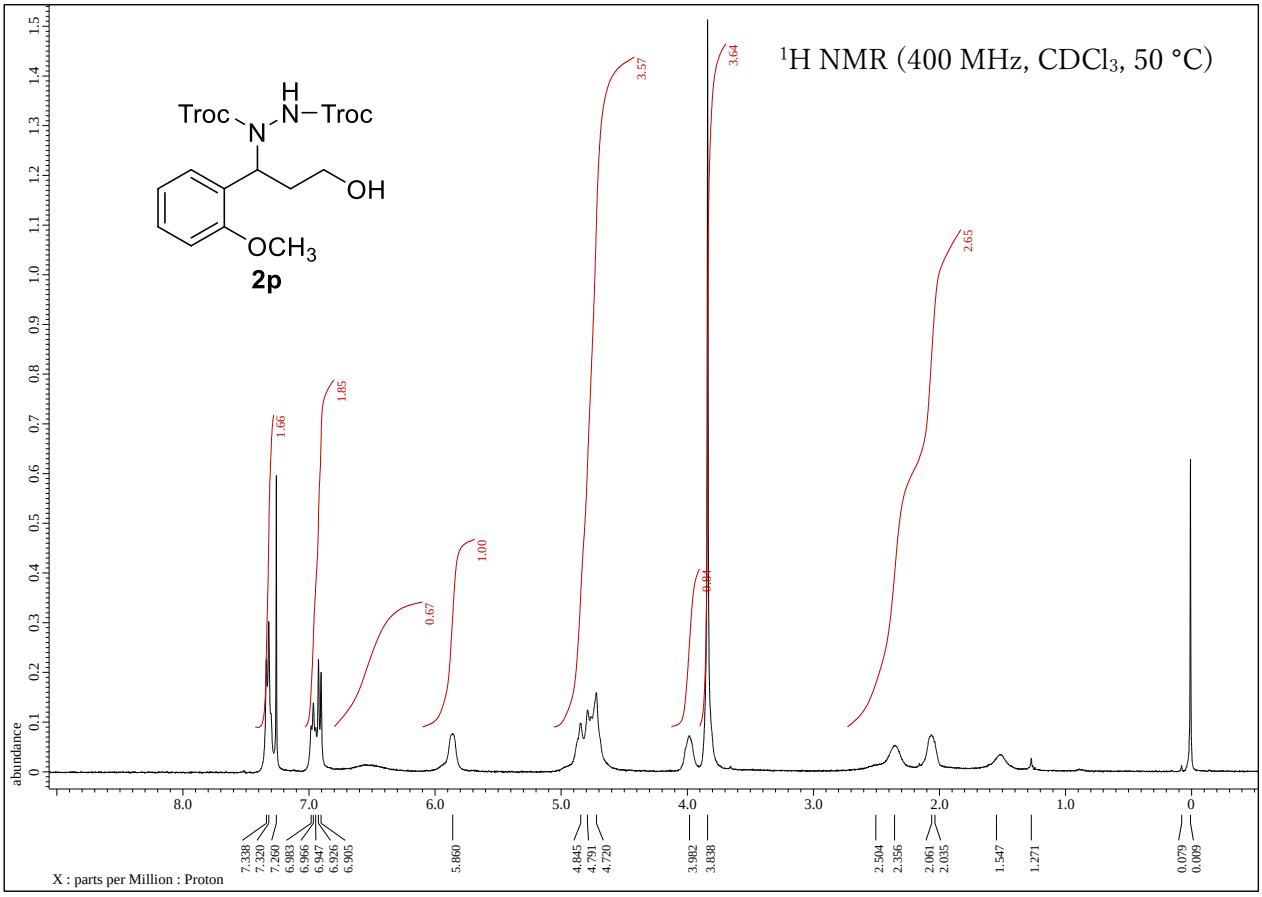

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
abundance
X : parts per Million : Carbon13

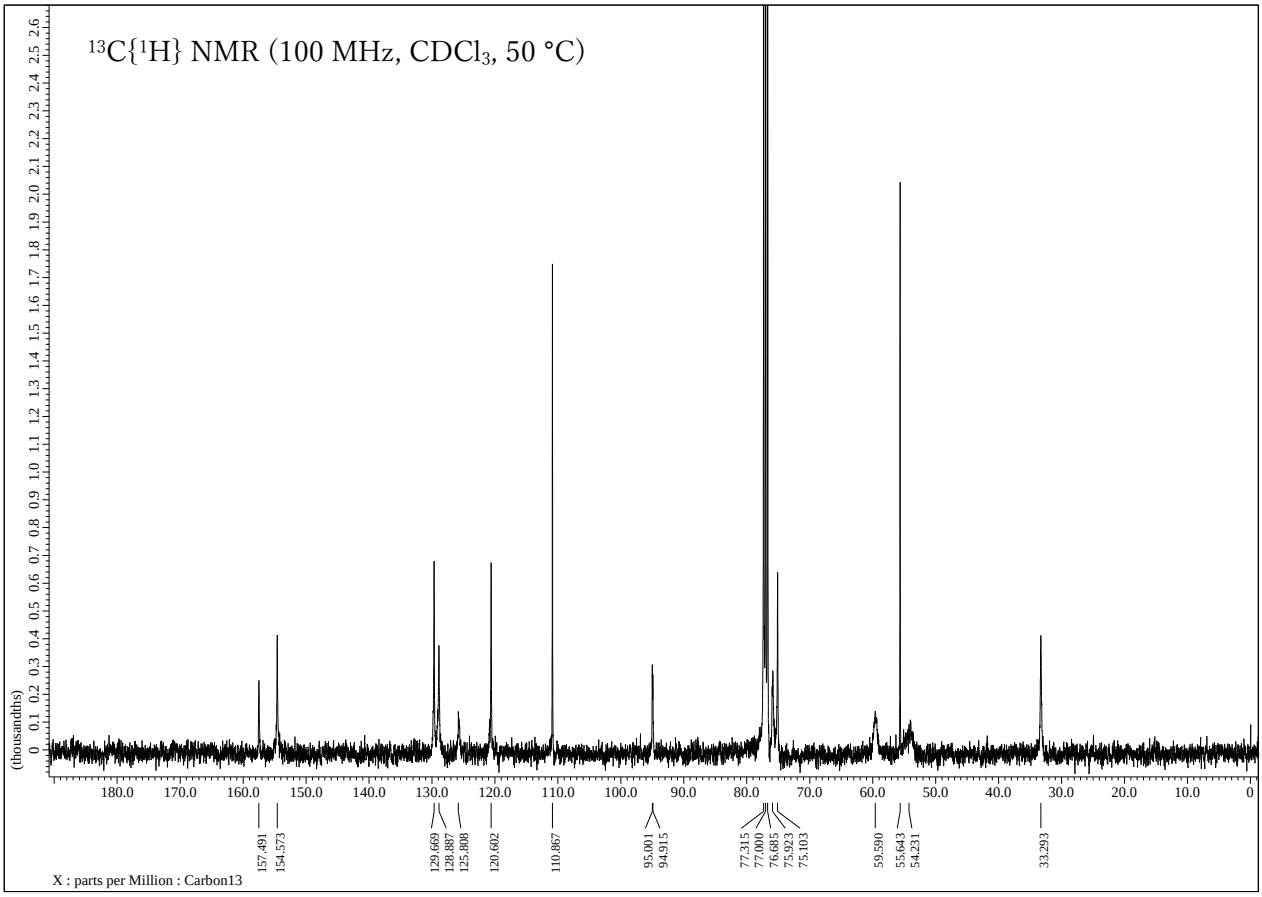

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
2n
3.72
2.85
0.68
1.00
3.77
1.97
2.37
abundance
X : parts per Million : Proton

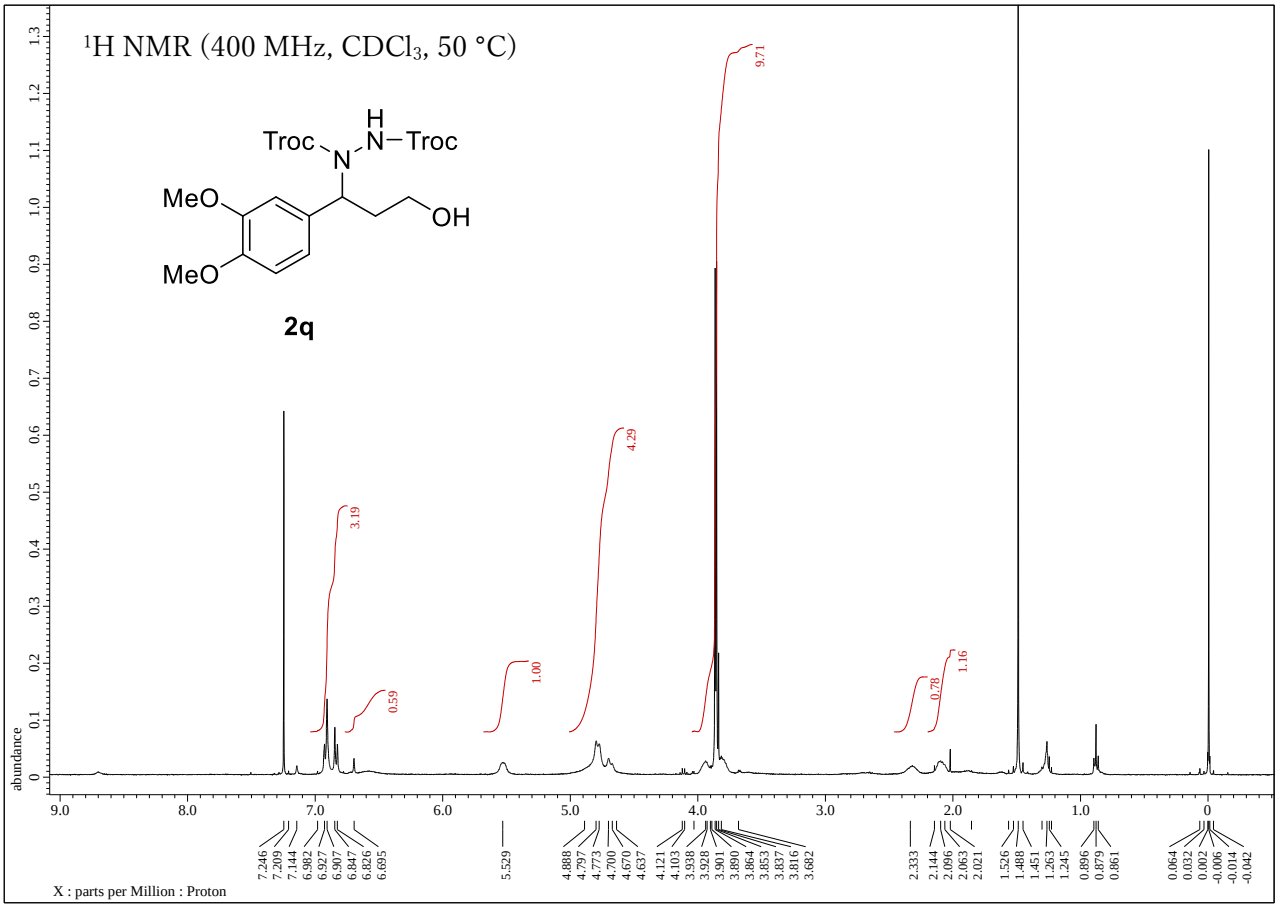

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

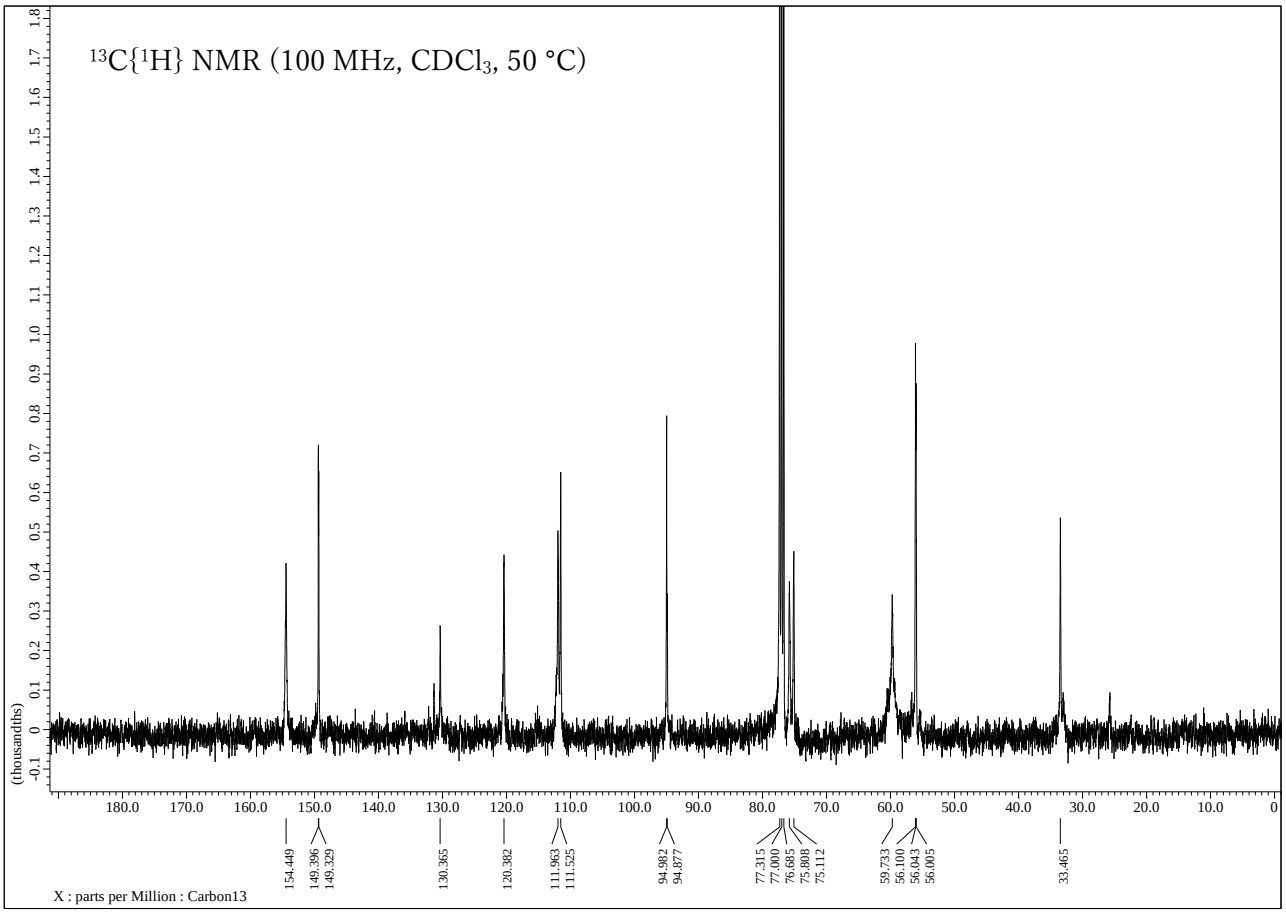

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N
Troc
OH
MeO
2o
abundance
X : parts per Million : Proton

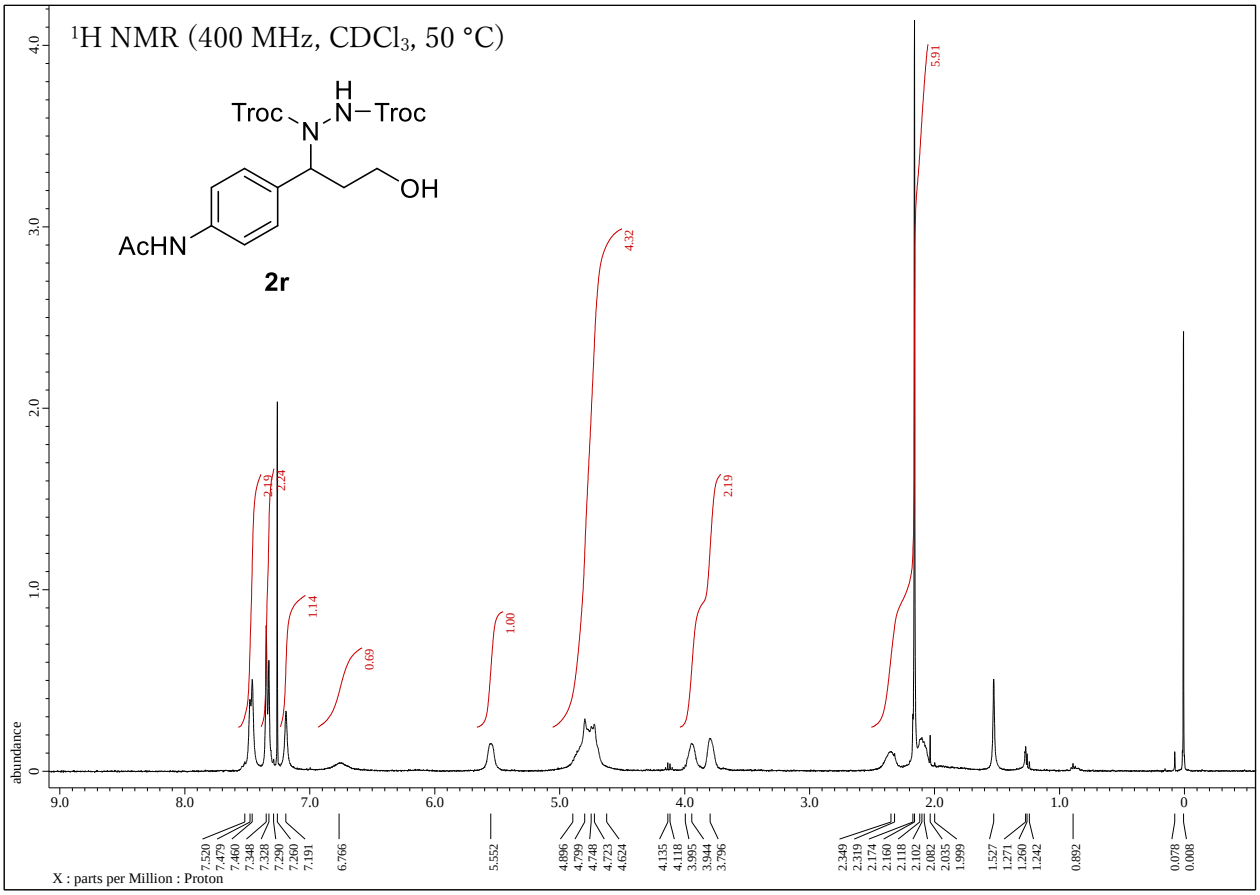

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

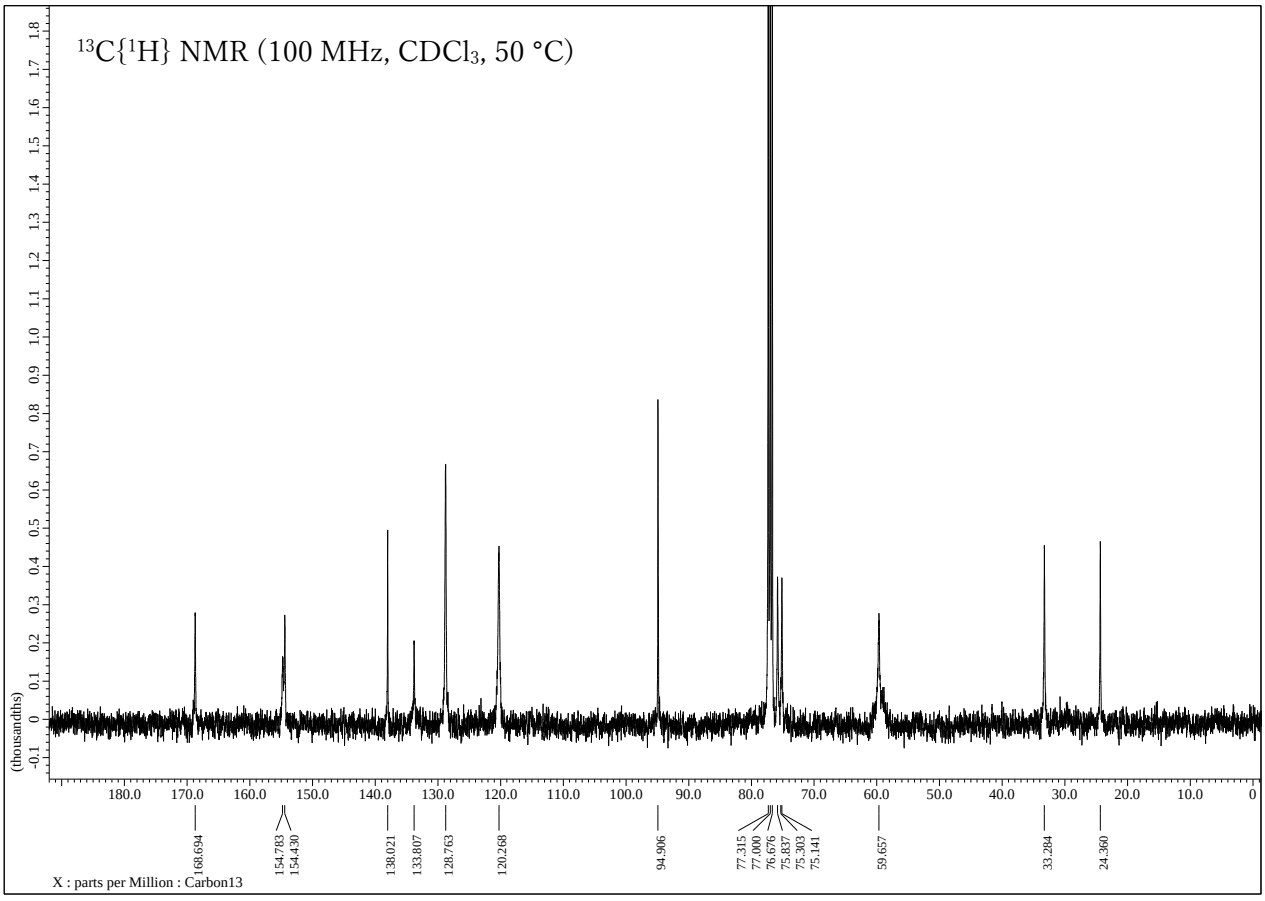

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N
Troc
OH
OCH$_3$
2p
abundance
X : parts per Million : Proton

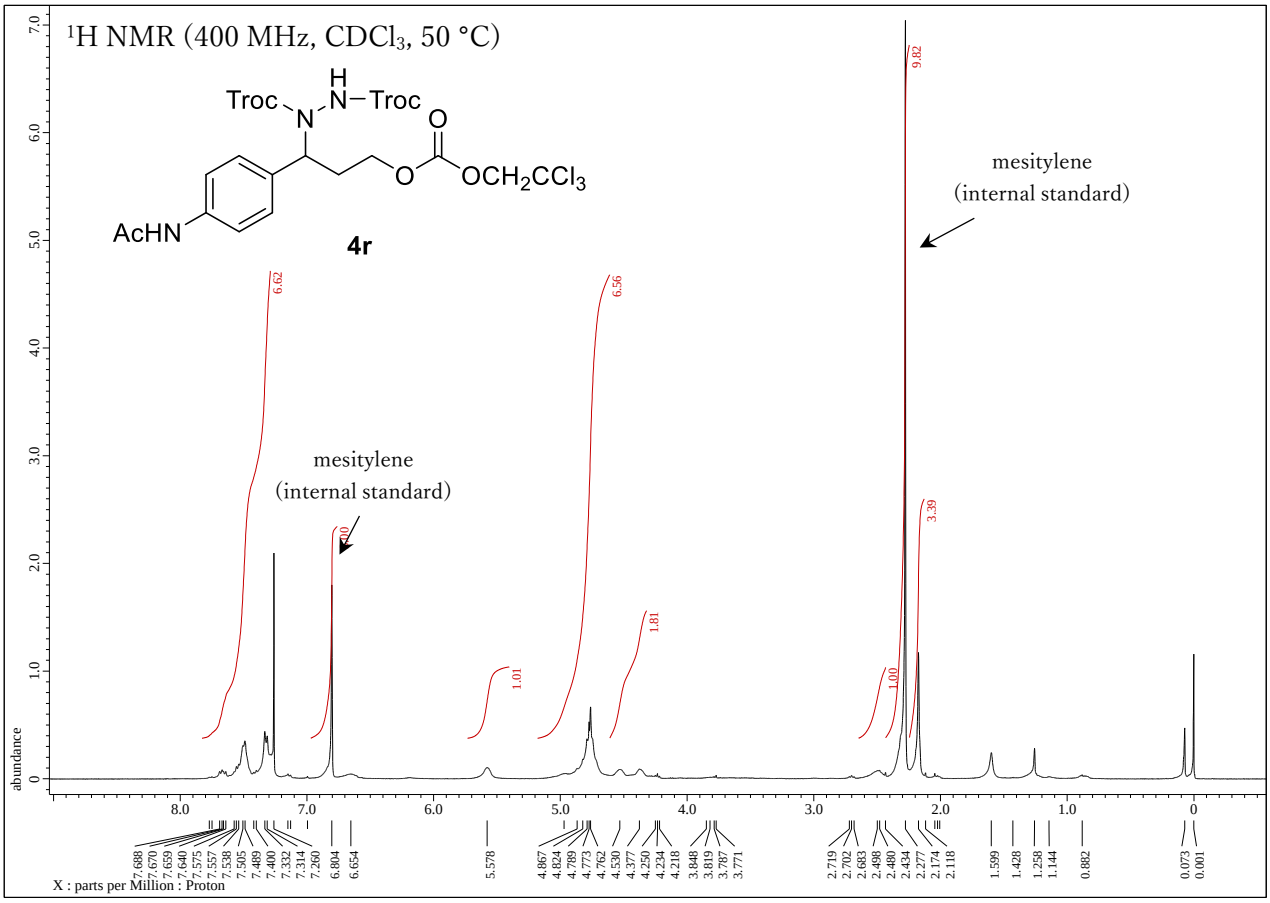

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

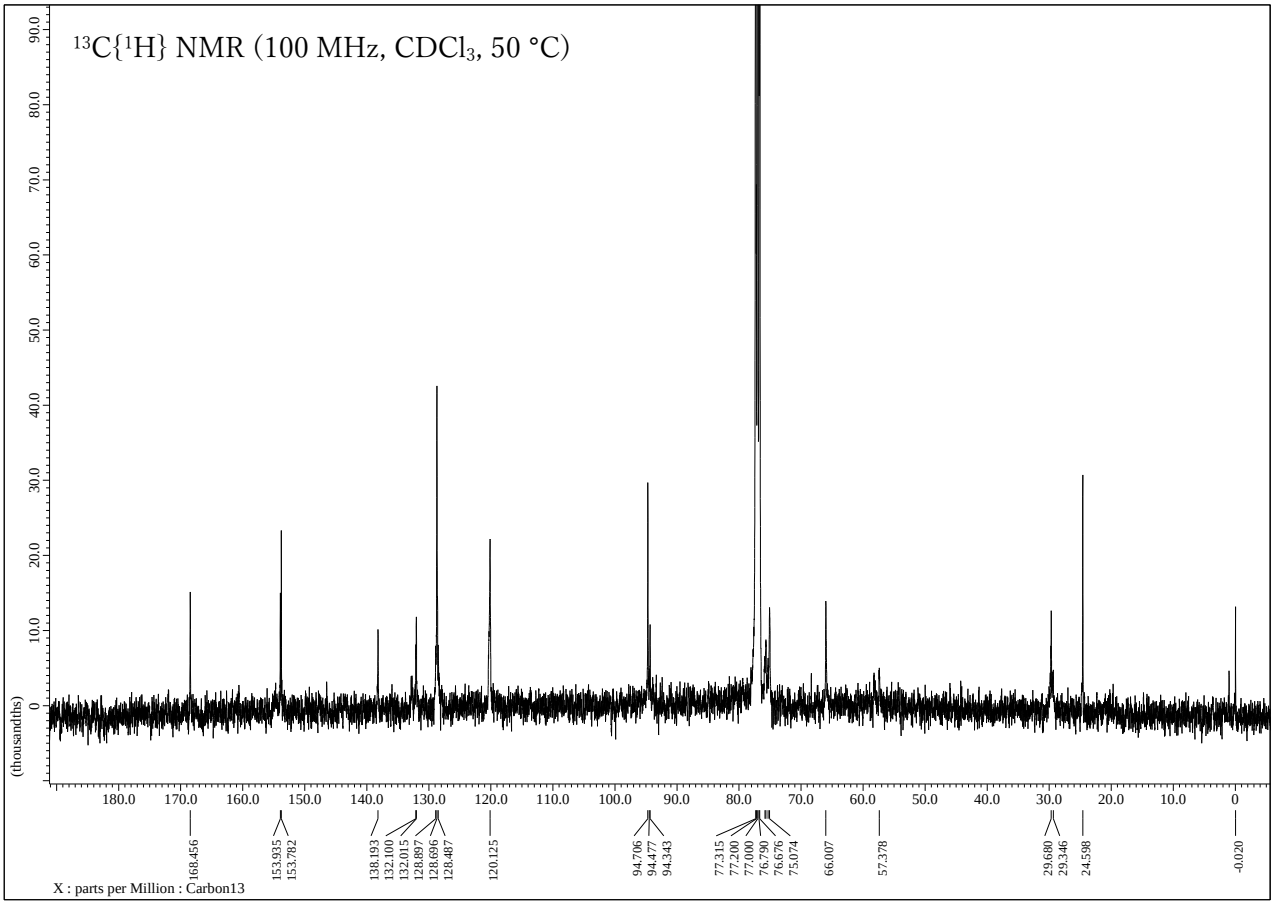

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N–Troc
MeO
MeO
OH
2q
9.71
4.29
3.19
1.00
0.59
0.78
1.16
abundance
X : parts per Million : Proton

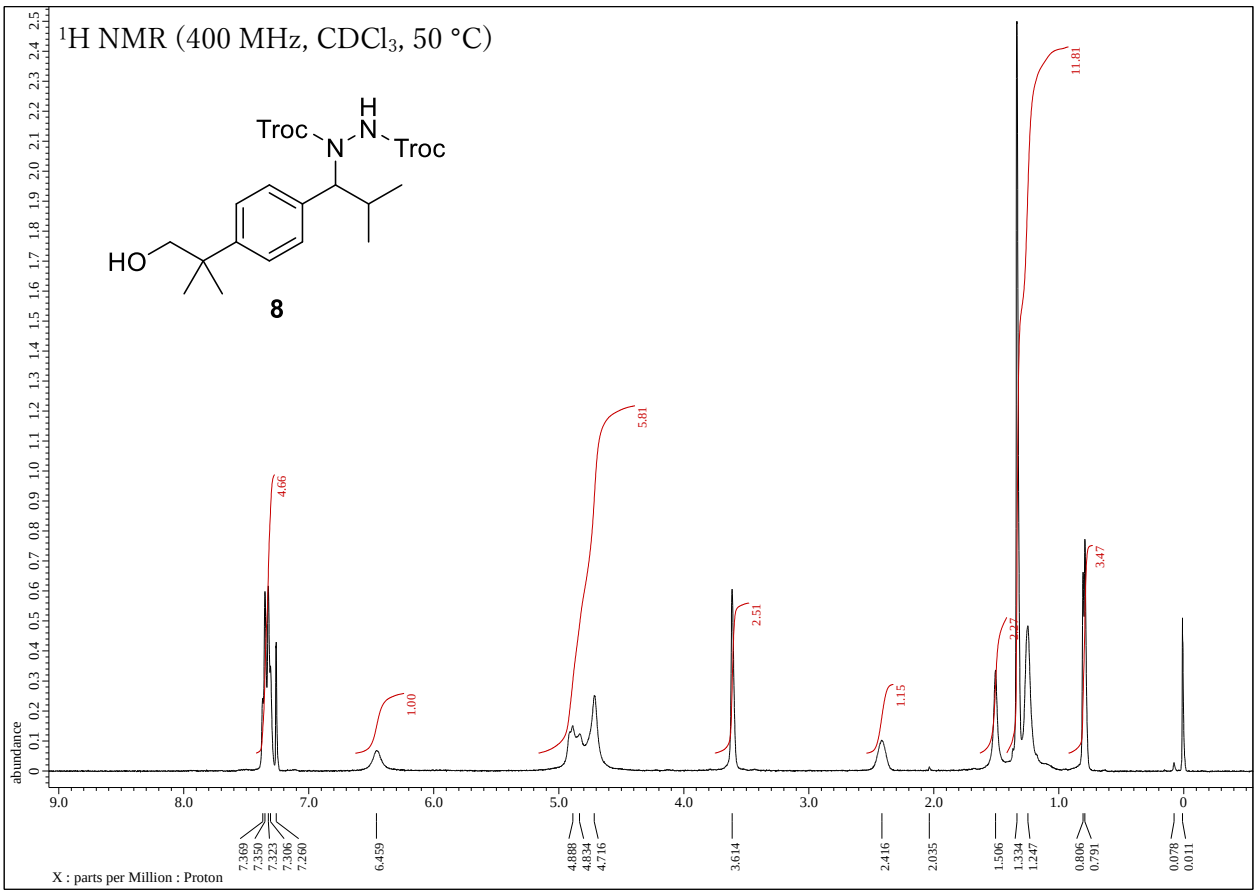

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

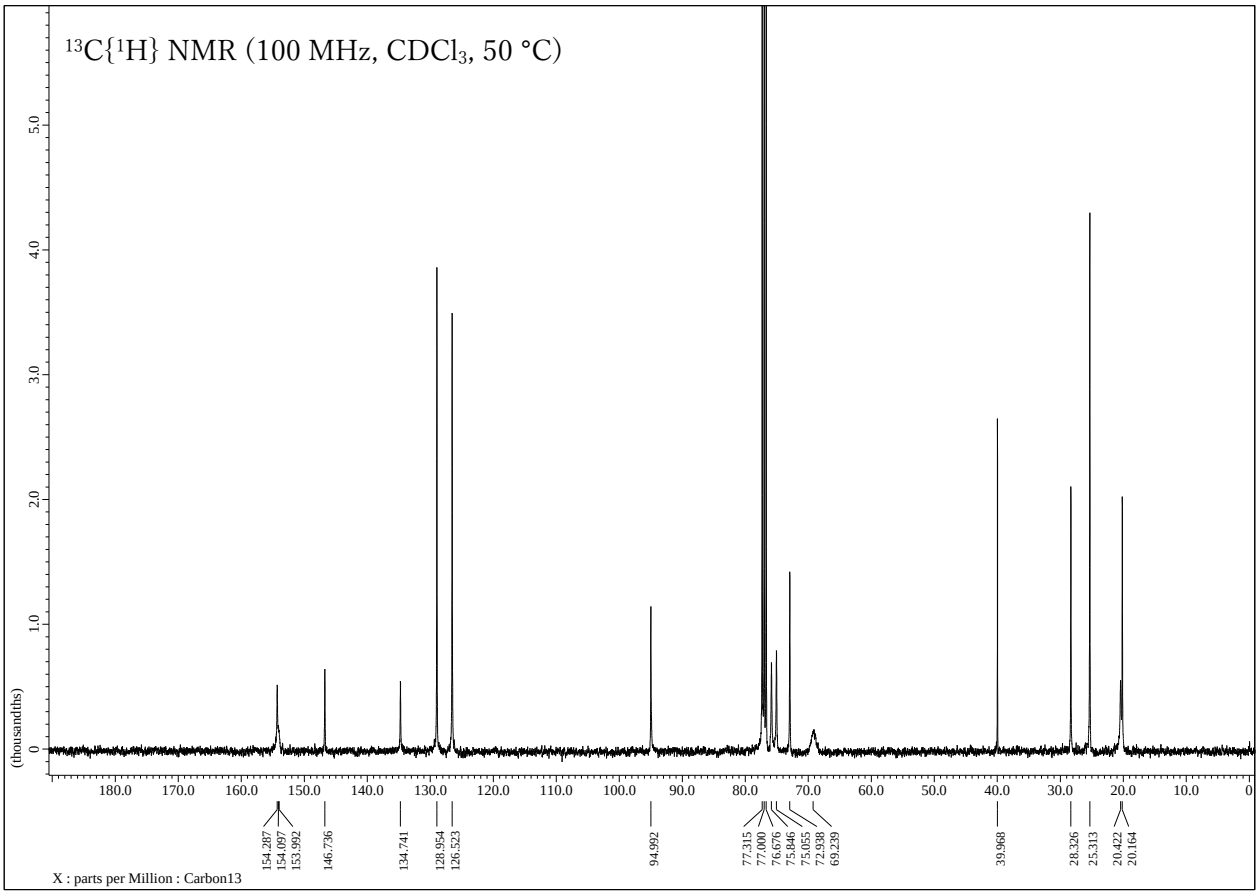

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N
Troc
OH
AcHN
2r
abundance
X : parts per Million : Proton

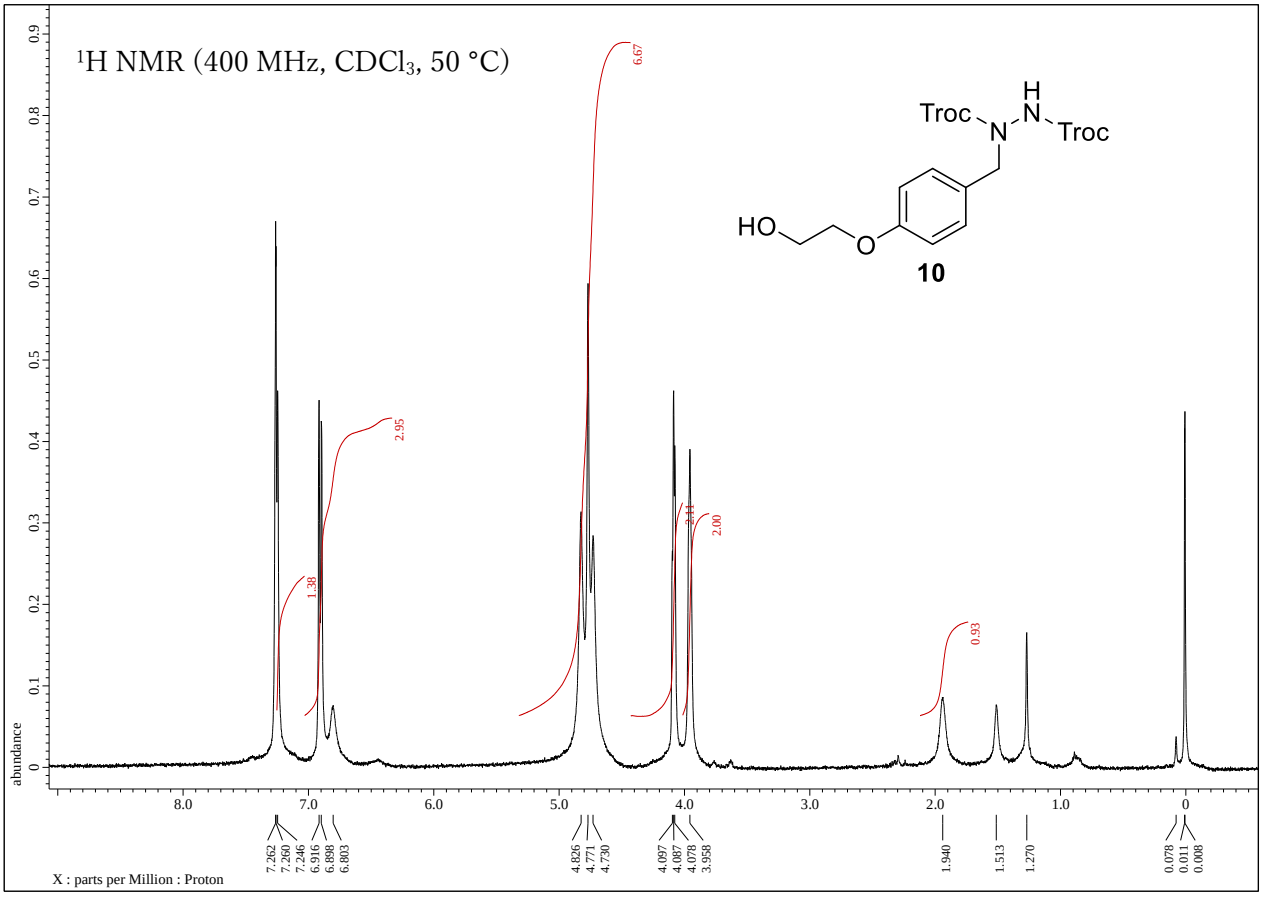

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

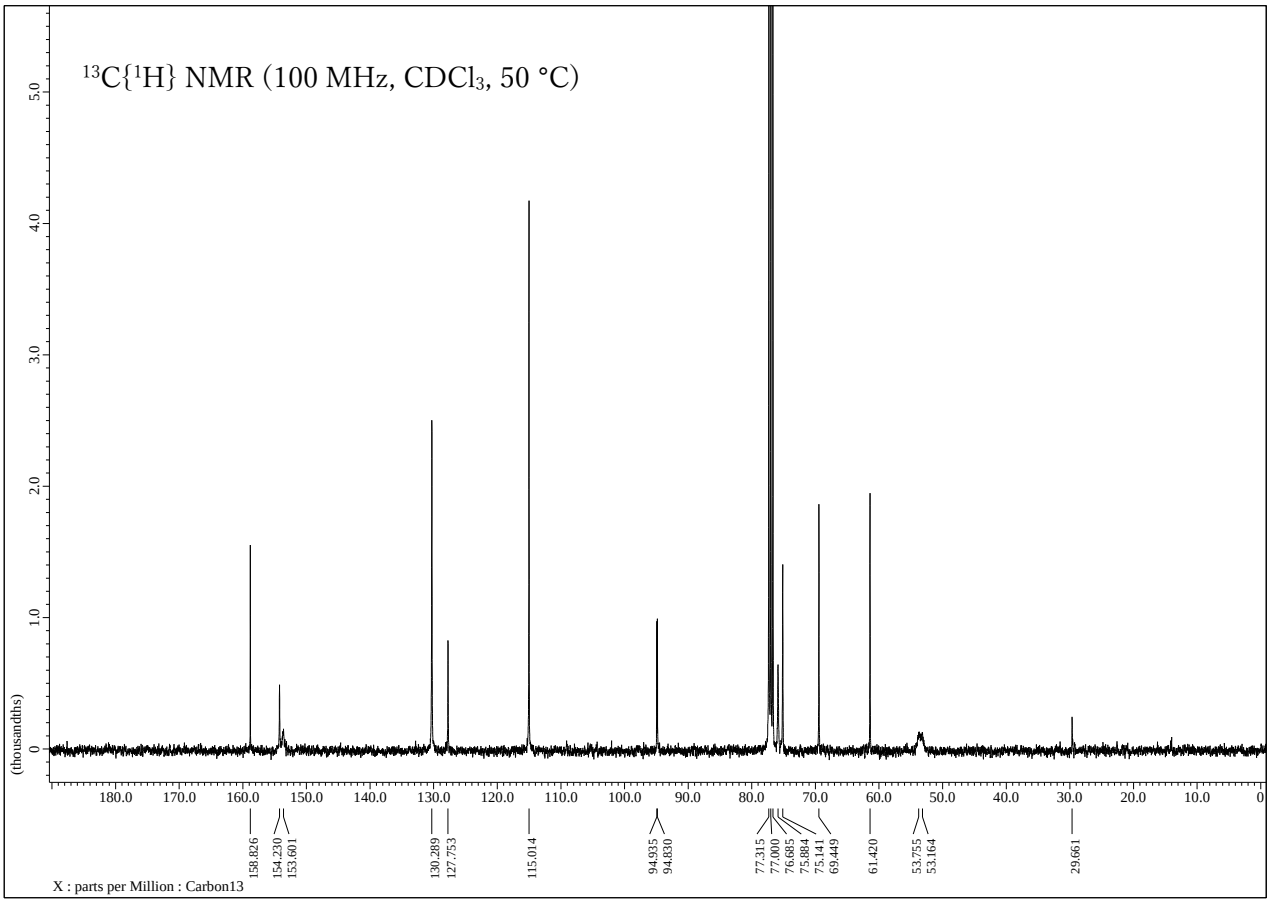

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N–Troc
O
O
OCH_2CCl_3
AcHN
4r
mesitylene
(internal standard)
mesitylene
(internal standard)
9.82
6.62
6.56
3.39
1.81
1.01
1.00
1.00
abundance
X : parts per Million : Proton

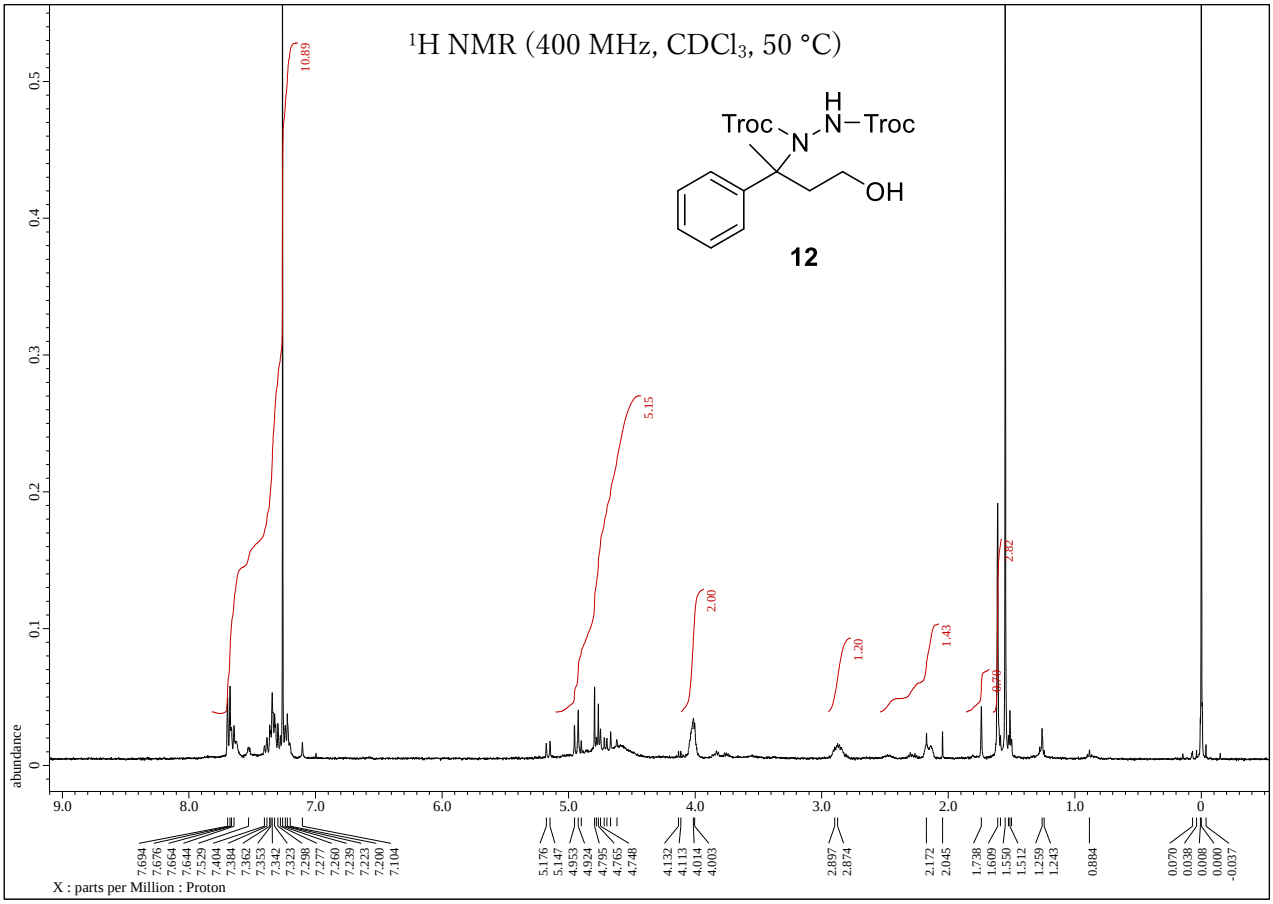

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

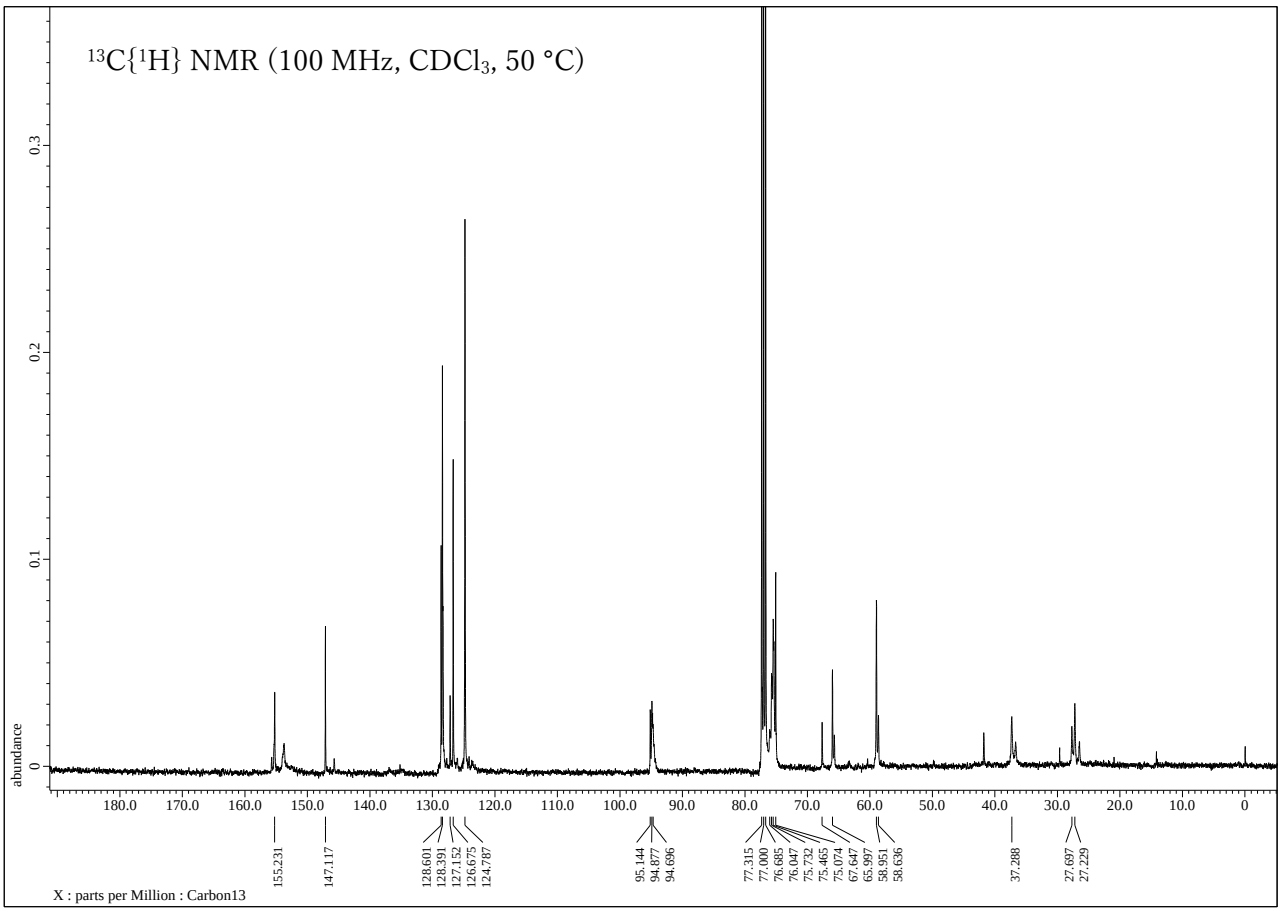

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
HO
8
abundance
X : parts per Million : Proton

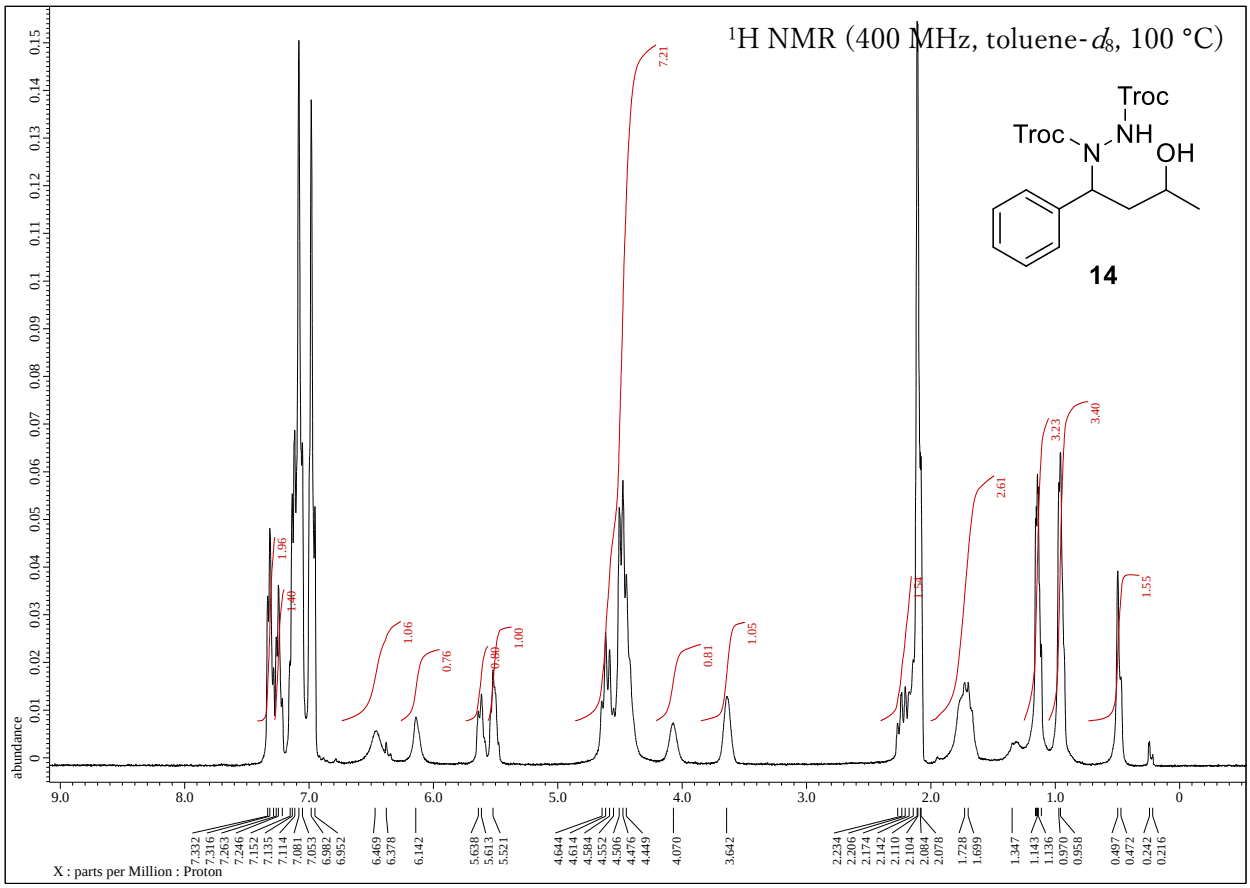

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13

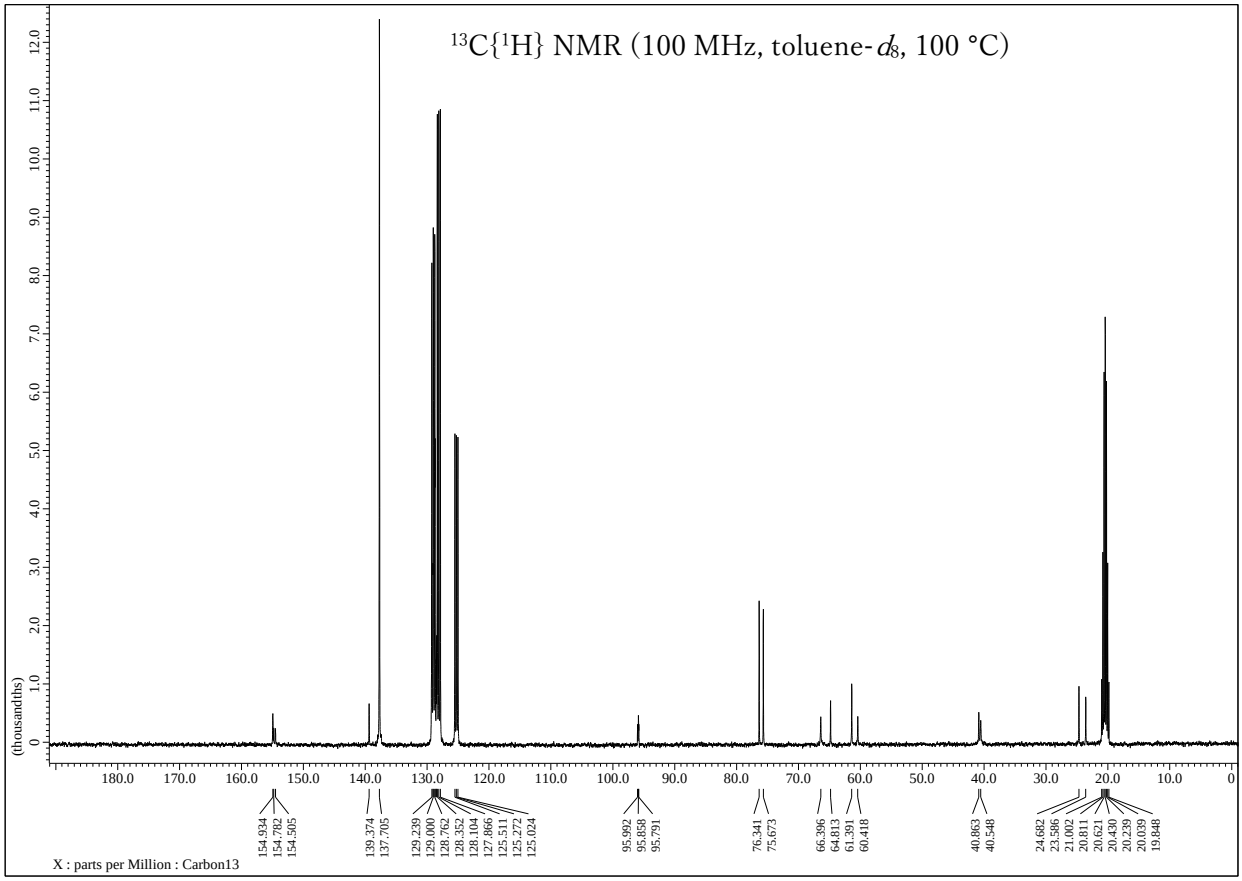

^{1}H NMR (400 MHz, CDCl$_3$, 50 °C)
Troc
N
H
N
Troc
HO
O
10
abundance
6.67
2.95
1.38
2.11
2.00
0.93
7.262
7.260
7.246
6.916
6.898
6.803
4.826
4.771
4.730
4.097
4.087
4.078
3.958
1.940
1.513
1.270
0.078
0.011
0.008
X : parts per Million : Proton

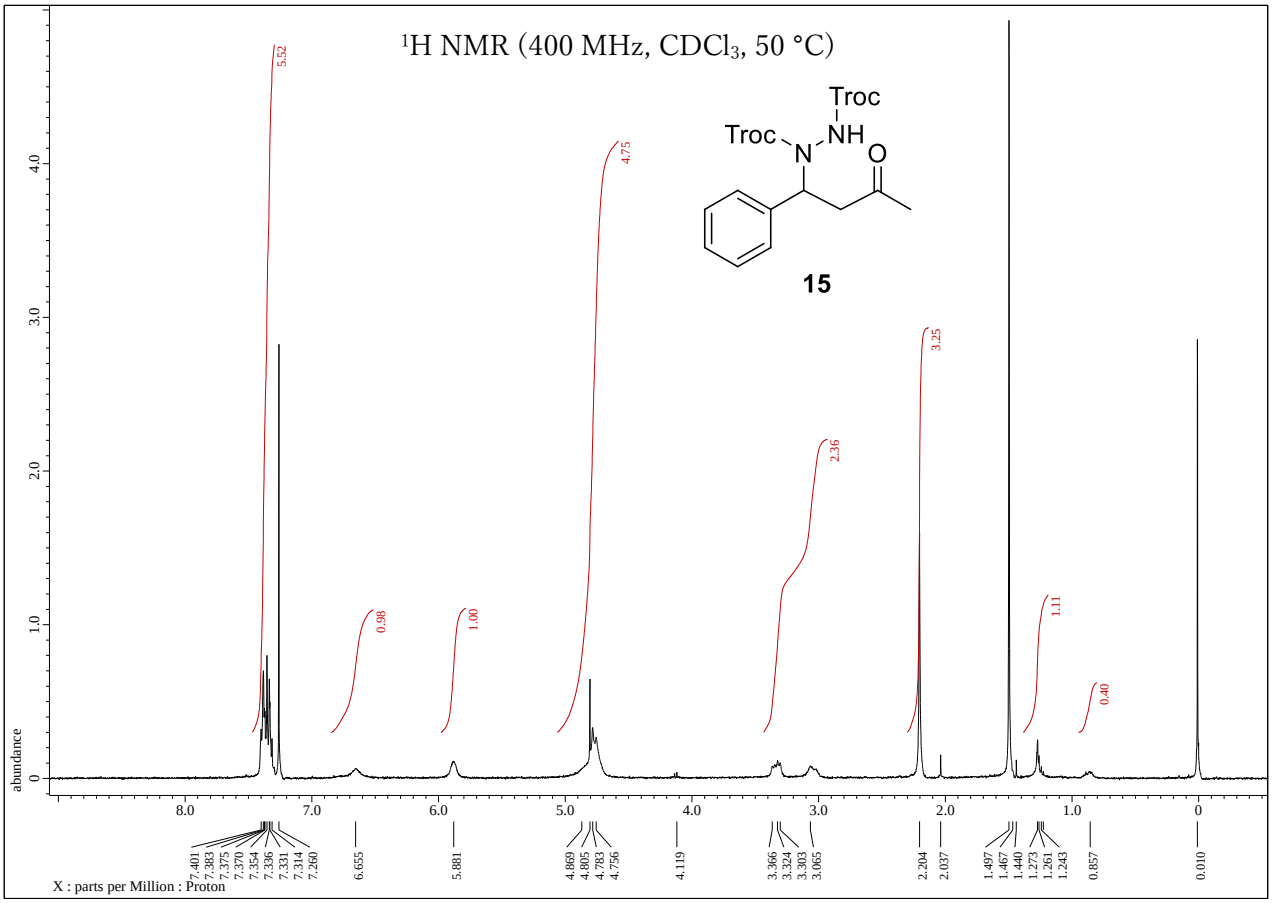

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$, 50 °C)
(thousandths)
158.826
154.230
153.601
130.289
127.753
115.014
94.935
94.830
77.315
77.000
76.685
75.884
75.141
69.449
61.420
53.755
53.164
29.661
X : parts per Million : Carbon13

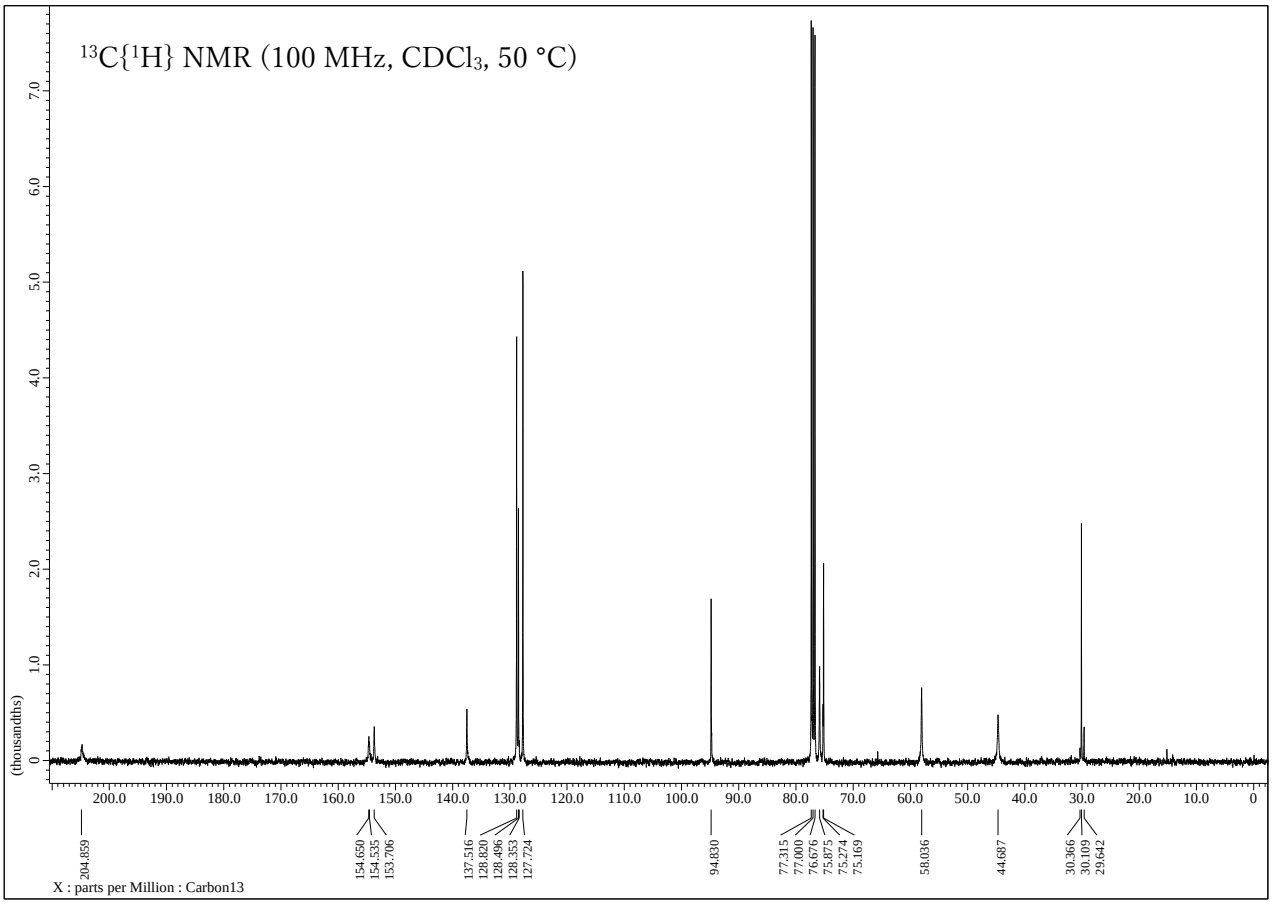

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
N
H
N
Troc
OH
12
10.89
5.15
2.00
1.20
1.43
2.82
0.74
abundance
X : parts per Million : Proton

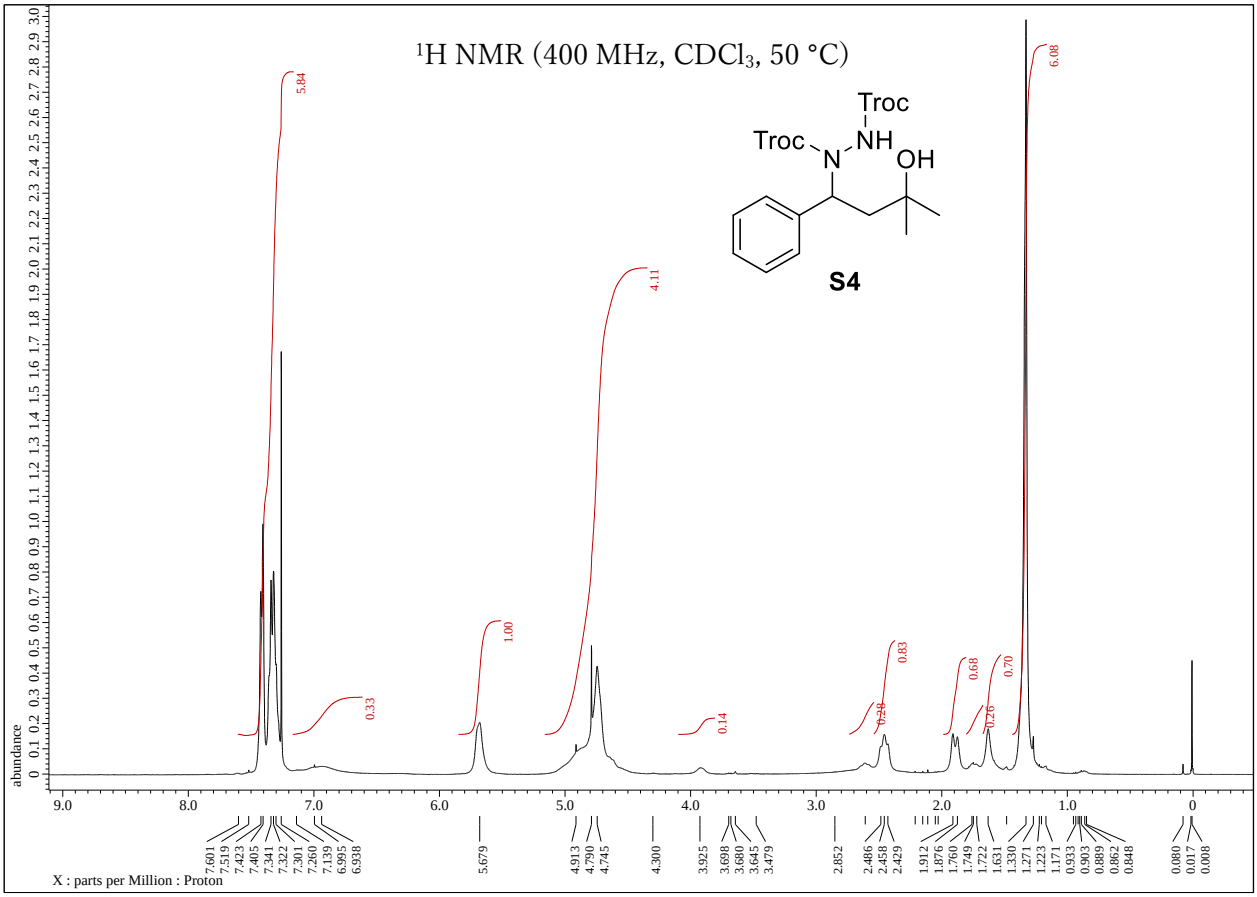

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
abundance
X : parts per Million : Carbon13

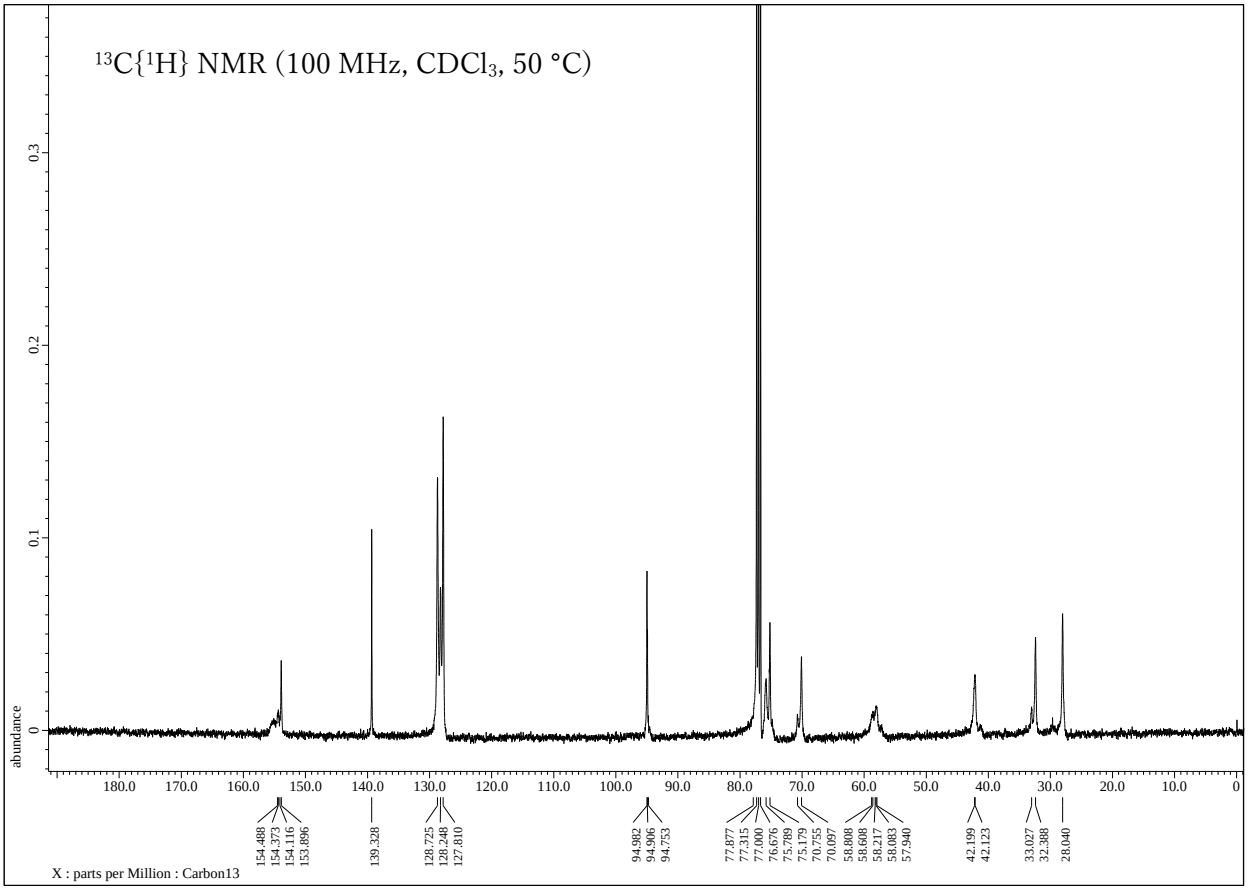

^{1}H NMR (400 MHz, toluene-d_8, 100 °C)
Troc
Troc
N
NH
OH
14
abundance
X : parts per Million : Proton

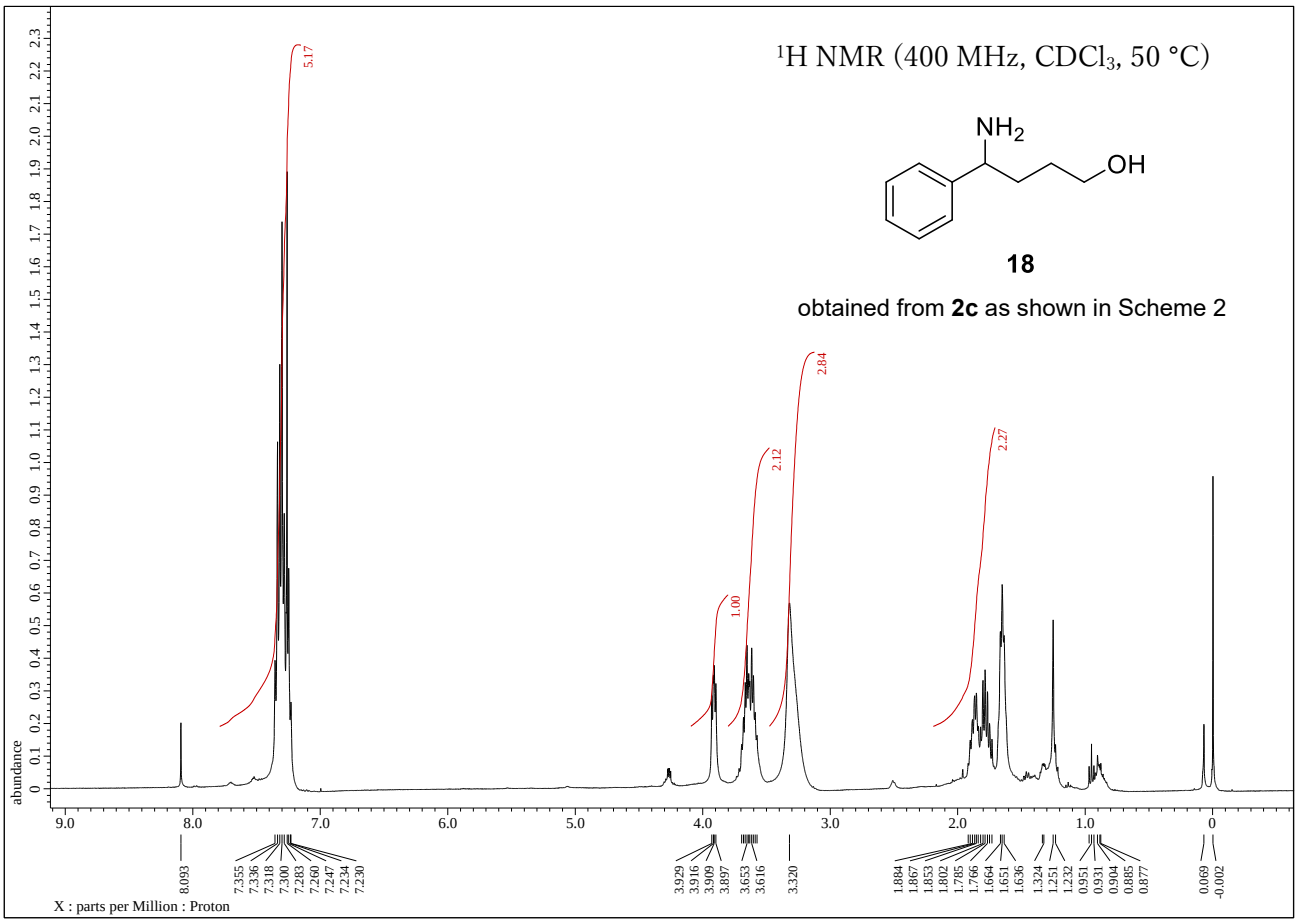

^{13}C{^{1}H} NMR (100 MHz, toluene-d_8, 100 °C)
(thousandths)
X : parts per Million : Carbon13

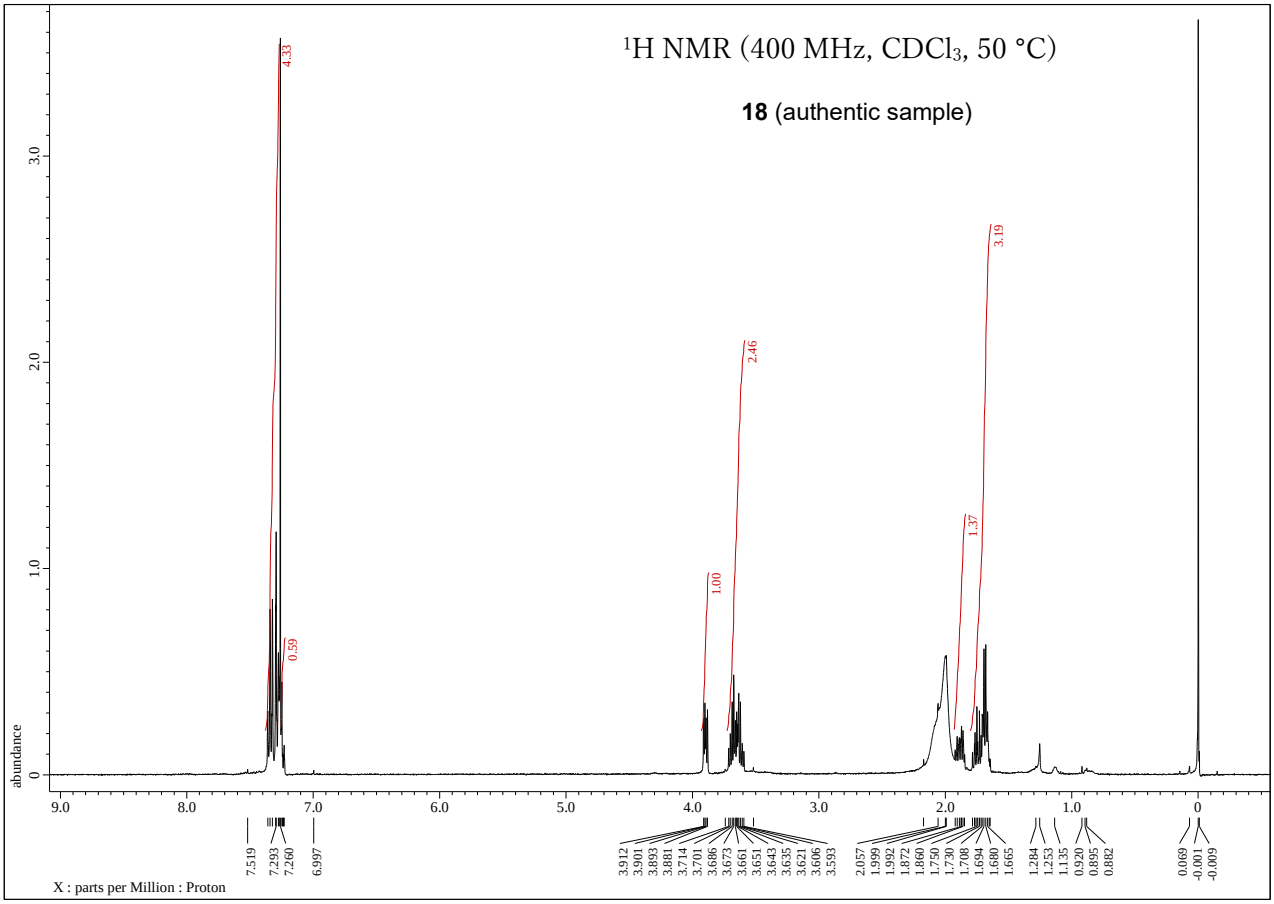

^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
Troc
N
NH
O
15
abundance
X : parts per Million : Proton

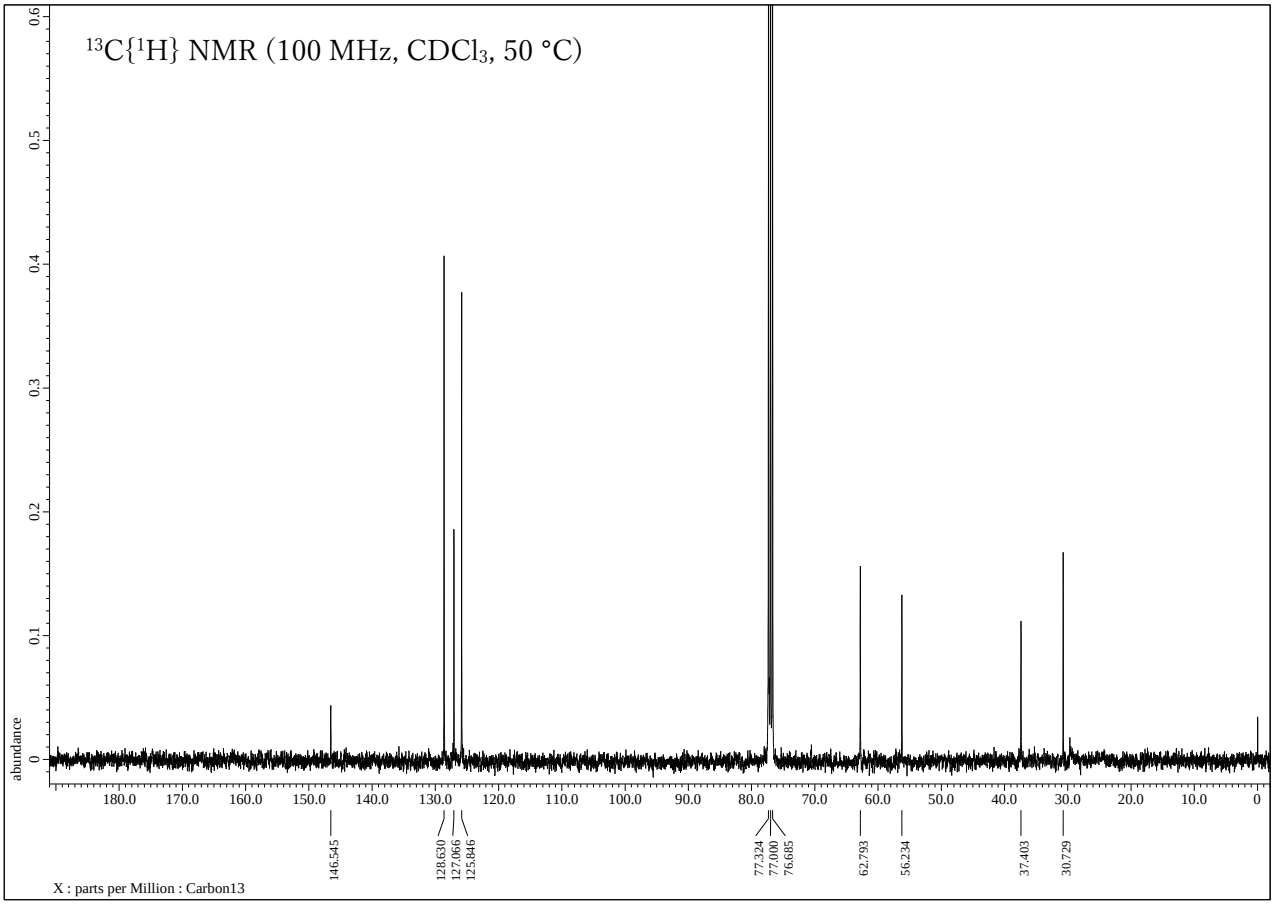

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
(thousandths)
X : parts per Million : Carbon13


^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
Troc
Troc
N
NH
OH
S4
abundance
X : parts per Million : Proton


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
abundance
X : parts per Million : Carbon13


^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
NH2
OH
18
obtained from 2c as shown in Scheme 2
abundance
X : parts per Million : Proton


^{1}H NMR (400 MHz, $CDCl_3$, 50 °C)
18 (authentic sample)
4.33
2.46
3.19
1.37
1.00
0.59
abundance
X : parts per Million : Proton


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, 50 °C)
abundance
X : parts per Million : Carbon13