Red-light-activatable ruthenium phthalocyanine catalysts

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Supporting information

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General Comments

Instrumentation

Photoreactions were carried out in a reaction vessel wherein the reaction mixtures were irradiated using red (λ_{max} = 634 nm) or blue (λ_{max} = 470 nm) LED light. LDL2-119X16RD2 (nominal wavelength: 634 nm, fwhm: 15.0 nm) and ALDKIT001 (nominal wavelength: 470 nm, fwhm: 24.7 nm) were purchased from CCS Inc. and Aldrich Inc., respectively (Fig. S10). The output power was 12 W, and the LED light was placed 5.0 cm from the reaction vessel. NMR spectra were obtained using JEOL ECA-500 or Bruker AVANCE 400 spectrometer. Chemical shifts are expressed in δ (ppm) values, and coupling constants are expressed in hertz (Hz). ^{1}H and ^{13}C NMR spectra were referenced to the tetramethylsilane (TMS) or the residual solvent as an internal standard. ^{19}F NMR spectra were referenced to the trifluoroacetic acid (δ = –79.0 ppm) as an internal standard. The following abbreviations are used: s = singlet, d = doublet, m = multiplet, and brs = broad singlet. High-resolution mass spectra (HRMS) were recorded using a Bruker Daltonics solariX spectrometer (MALDI). Electronic absorption spectra were recorded on a JASCO V-770 spectrophotometer. A photonic multichannel analyzer (Hamamatsu, PMA-12) was used for the measurement of phosphorescence spectra under N_2 atmosphere, in which the wavelength of excitation light was 632 nm. Cyclic voltammetry (CV) measurements were recorded using a Hokuto Denko HZ5000 potentiostat under a nitrogen atmosphere with 0.1 M of tetrabutylammonium perchlorate (TBAP) as the supporting electrolyte. Measurements were made using a glassy carbon electrode (area = 0.07 cm^2), an Ag/AgCl reference electrode, and a Pt wire counter electrode. The concentration of the solution was fixed at 0.5 mM, and the sweep rate was set to 100 mV/s. The ferrocenium/ferrocene (Fc^+/Fc) couple was used as an internal standard.

Materials

Unless otherwise noted, materials were purchased from Tokyo Kasei Co., Aldrich Inc., and other commercial suppliers and were used after appropriate purification (distillation or recrystallization). **ZnPc**,[1] **Ru(py)$_2$TAP**,[2] **2a**,[3] **2c**,[4] **2d**,[5] **2e**,[6] **2f**,[7] **2g**,[8] **2j**,[9] and **2n**[10] were synthesized according to published procedures.

Crystallographic data collection

Data collection for **1b** was carried out on a Bruker APEXIII CCD diffractometer with Bruker Helios multilayered confocal mirror monochromatized Cu*K*α radiation (λ = 1.54178 Å) at –183°C. The structures were solved by a direct method (SIR2004)[11] and refined using a full-matrix least square technique (SHELXL-2014).[12] Yadokari-XG 2009 software was used as a GUI for SHELXL-2014.[13] All non-hydrogen atoms were refined anisotropically. Positions of all hydrogen atoms were calculated geometrically, and refined by applying riding models. Some large electron peaks due to a solvent molecule(s) were found in the unit cell. As we failed to model them properly, the rest molecules were refined without the effect of the solvent molecule(s) by the Platon squeeze technique.[14] CCDC-2108084 contains the supplementary crystallographic data. Their data can be obtained free of charge from Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Additional Experimental Results

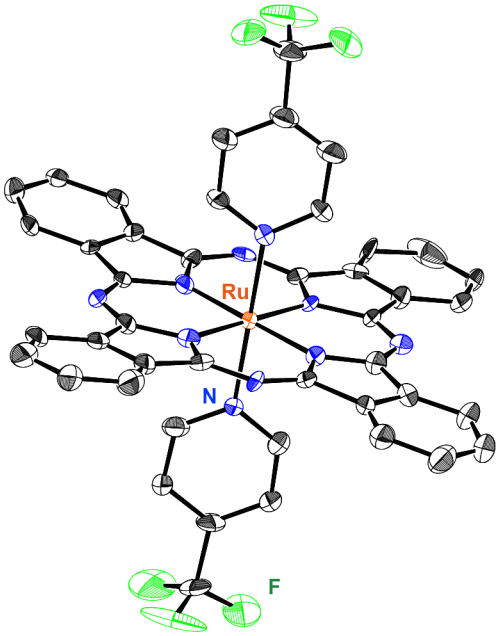


Fig. S1 Molecular structure of **1b** with thermal ellipsoids at 50% probability. Hydrogen atoms have been omitted for clarity and only selected atoms have been labeled.

Table S1 Crystal data and structure refinement for **1b**.

Empirical formula	$C_{44}H_{24}F_6N_{10}Ru$	
Formula weight	907.80	
Temperature	90(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	*Cc*	
Unit cell dimensions	a = 20.1094(18) Å	α = 90°
	b = 22.559(2) Å	β = 108.370(2)°
	c = 10.5878(10) Å	γ = 90°
Volume	4558.4(7) Å^3	
Z	4	
Density (Calcd.)	1.323 Mg/m^3	
Absorption coefficient	3.351 mm^{-1}	
F(000)	1824	
Crystal size	0.200 × 0.200 × 0.100 mm^3	
Theta range for data collection	5.920 to 66.497°	
Index ranges	-23<=h<=23, -26<=k<=25, -12<=l<=12	
Reflections collected	14260	
Independent reflections	6274 [R(int) = 0.0395]	
Completeness to theta = 66.497°	98.1%	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints /parameters	6274 / 2 / 550	
Goodness-of-fit on F^2	1.042	
Final R indices [I > 2sigma(I)]	R_1 = 0.0431, wR_2 = 0.1125	
R indices (all data)	R_1 = 0.0435, wR_2 = 0.1133	
Largest diff. peak and hole	1.635 and -0.861 e.Å^{-3}	
CCDC No.	2108084	

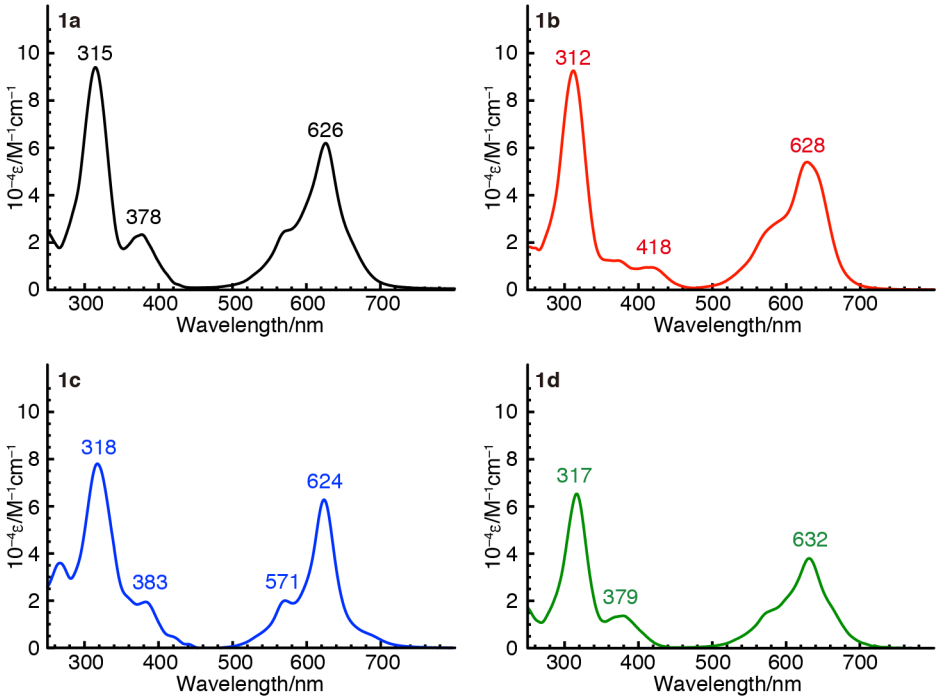


Fig. S2 Absorption spectra of RuPcs **1a-d** in $CHCl_3$.

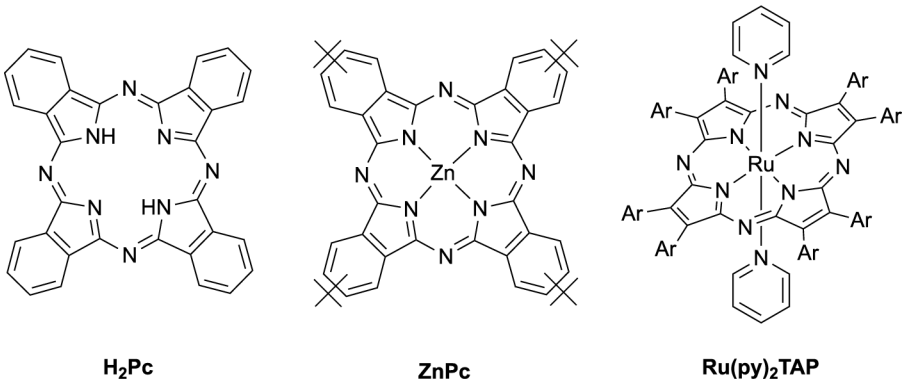


Fig. S3 Structures of **H_2Pc**, **ZnPc**, and **Ru(py)$_2$TAP**.

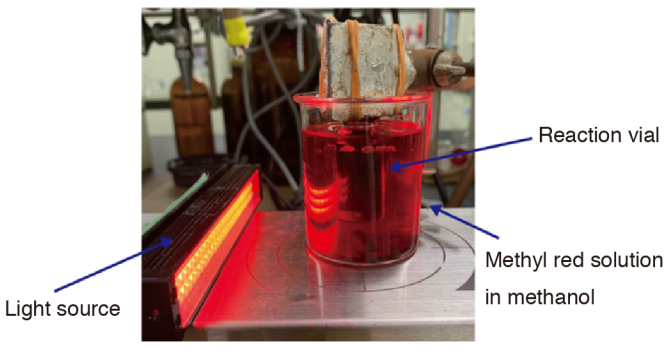


Fig. S4 Reaction setup for the shielded condition. The photo shows the reaction vessel immersed in a

methanol solution of methyl red (Fig. 3a, entry 2). The entire reaction system was shielded from natural light during the photoreaction.

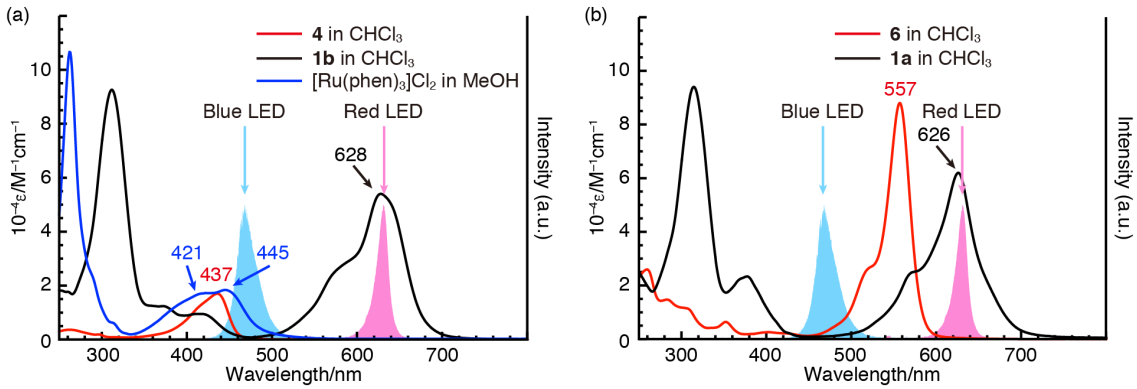


Fig. S5 Absorption spectra of (a) **1b**, **4**, and $[Ru(phen)_3]Cl_2$ and (b) **1a** and **6**. The emission spectra of the LEDs overlapped.

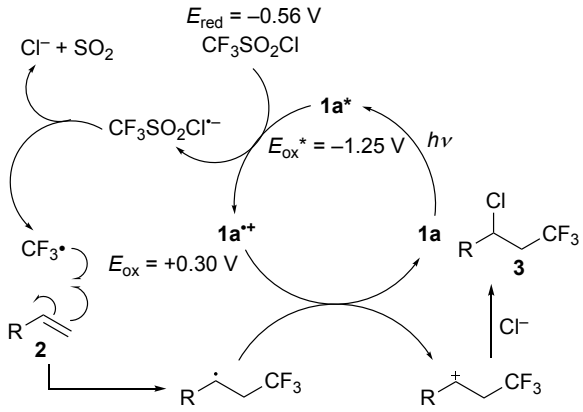


Fig. S6 Plausible reaction mechanism.

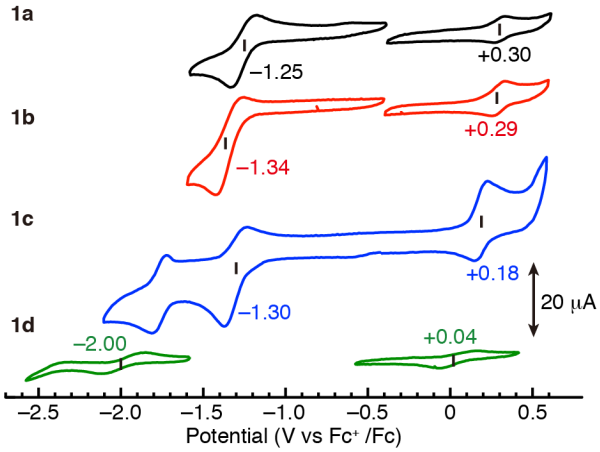


Fig. S7 Cyclic voltammograms of RuPcs **1a-d** recorded using 0.5 mM solutions of the analytes in [^{n}Bu$_4$N]ClO$_4$/DMF. Ferrocene was used as the internal standard and the Fc/Fc$^+$ couple was set to 0 V.

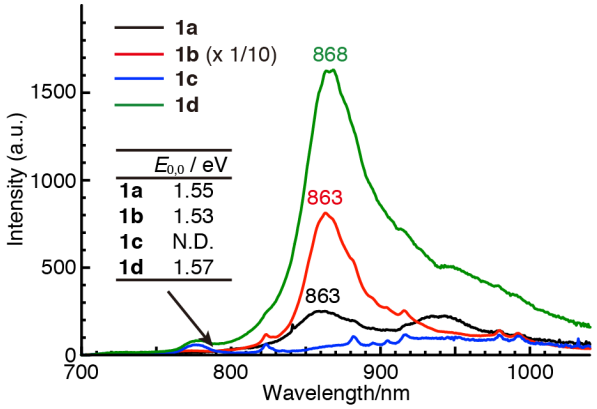


Fig. S8 Phosphorescence spectra of RuPcs **1a-d** (2 μM) in acetone under an N$_2$ atmosphere (λ_{ex} = 632 nm).

Table S2 Summary of optical and redox parameters for the photocatalysts

Catalyst	E_{ox} [V] [a] (cat/cat$^{\bullet+}$)	E_{red} [V] [a] (cat/cat$^{\bullet-}$)	$\lambda_{phos,\ max}$ [nm]	$E_{0,0}$ [eV]	E_{ox}* [V] [a] (cat*/cat$^{\bullet+}$)	E_{red}* [V] [a] (cat*/cat$^{\bullet-}$)
1a	+0.30	–1.25	863	1.55	–1.25	+0.30
1b	+0.29	–1.34	863	1.53	–1.24	+0.19
1c	+0.18	–1.30	N.D. [b]	N.D. [b]	N.D. [b]	N.D. [b]
1d	+0.04	–2.00	868	1.57	–1.53	–0.43
Ru(phen)$_3$Cl$_2$ [c]	+0.88	–1.74	610	2.15	–1.25	+0.44

[a] vs Fc/Fc$^+$. [b] Not determined. [c] The data was taken from ref. 15.

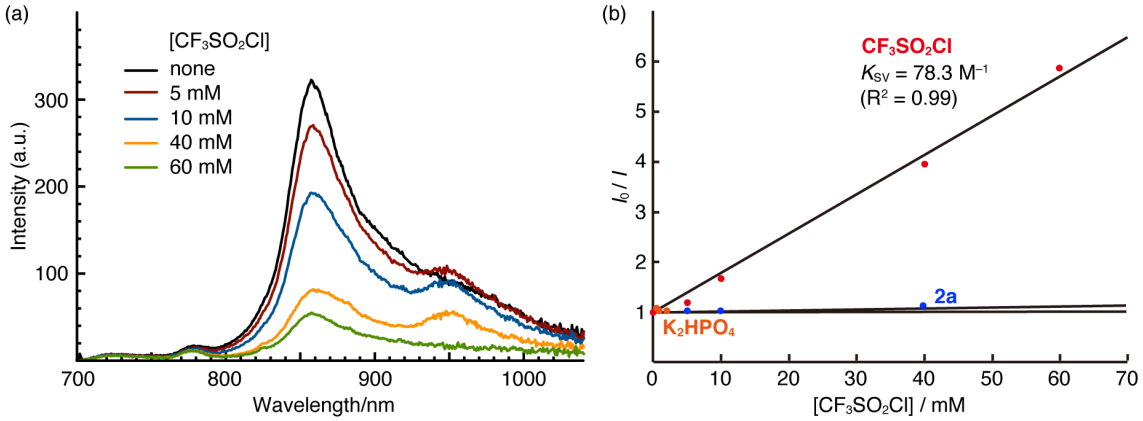


Fig. S9 (a) Phosphorescence spectra of **1a** (16 μM, in acetone) with CF_3SO_2Cl (0~60 mM). (b) The Stern-Volmer plot for **1a**/CF_3SO_2Cl, **2a**, and K_2HPO_4. When the lifetime τ_0 of **1a** was 135 ns (in CH_2Cl_2)[16], the quencher rate coefficient k_q of CF_3SO_2Cl was calculated as 5.8 x 10^8 $M^{-1}s^{-1}$.

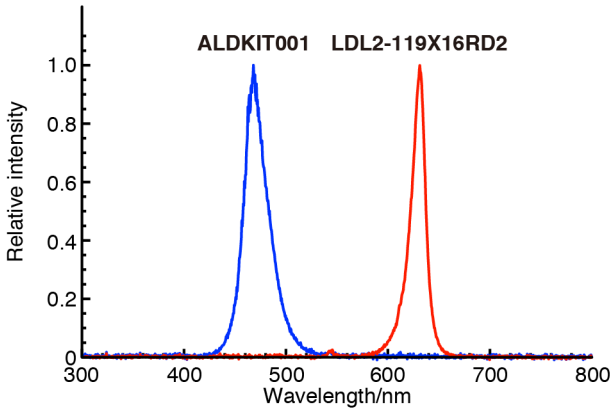
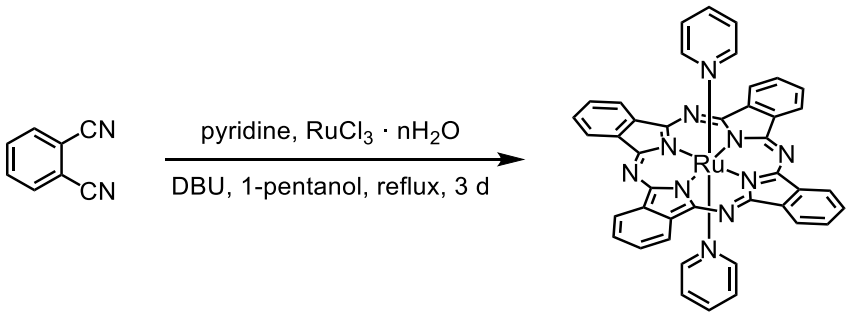


Fig. S10 Emission spectra of the NIR and blue LEDs.

Full Experimental Procedures

Preparation of catalysts

Bis(pyridyl) ruthenium(II) phthalocyanine (1a)[17]



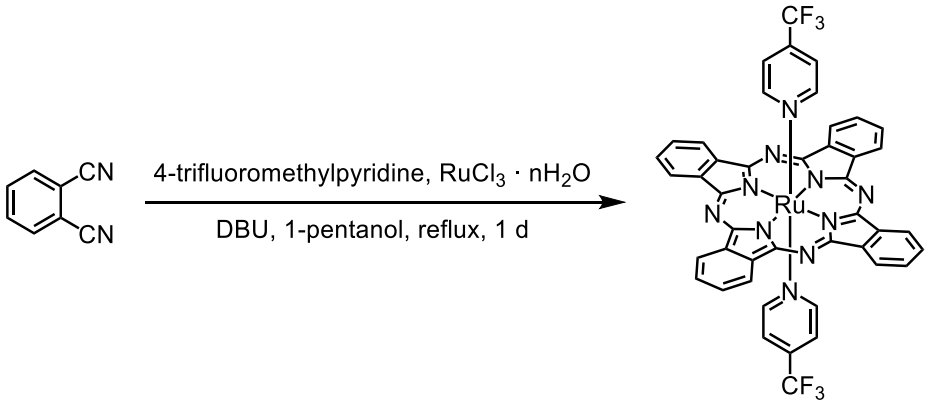
A mixture of phthalonitrile (201 mg, 1.6 mmol), pyridine (0.15 mL, 1.9 mmol), and DBU (0.05 mL, 0.33 mmol) in 1-pentanol (4.0 mL) was refluxed. At the same time, $RuCl_3 \cdot nH_2O$ (108 mg, 521 μmol) was boiled in 1-pentanol (2.0 mL) until a blue color formed. The $RuCl_3$ blue solution was added over 5 min to the phthalonitrile/pyridine/DBU mixture, and resulting solution was refluxed for 3 d. After the 1-pentanol was removed by evaporation, the crude product was purified by silica gel column chromatography ($CHCl_3$). The blue band was collected and concentrated. Then, MeOH was added to the residue, and precipitate was collected by filtration. The desired complex was obtained as a blue solid. (61.9 mg, 20%)

400 MHz ^{1}H NMR($CDCl_3$) δ(ppm): 9.16-9.14 (m, 8H, Pc-H), 7.90-7.88 (m, 8H, Pc-H), 6.04 (t, J = 7.6 Hz, 2H, py), 5.23 (dd, J = 7.6, 6.8 Hz, 4H, py), 2.45 (d, J = 5.2 Hz, 4H, py).

UV-vis($CHCl_3$) ($\varepsilon \times 10^{-4}$) λ_{max} nm: 315(9.4), 378(2.3), 626(6.2).

HR-MALDI-FT-ICR-MS calcd for $C_{37}H_{21}N_9Ru$ [M-pyridine]$^+$: 693.0968. Found: 693.0959.

Bis(4-trifluoromethylpyridyl) ruthenium(II) phthalocyanine (1b)



Synthesized according to the procedure for **1a**. Blue solid. (14%)

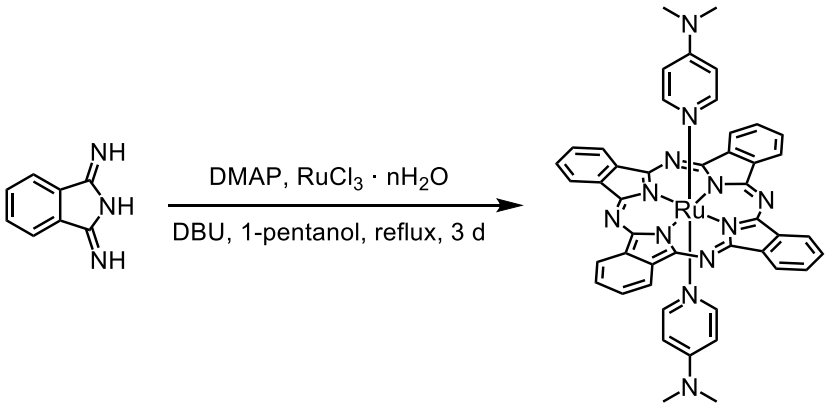
500 MHz ^{1}H NMR($CDCl_3$) δ(ppm): 9.21-9.17 (m, 8H, Pc-H), 7.96-7.92 (m, 8H, Pc-H), 5.47 (d, *J* = 7.4 Hz, 4H, 4-CF_3-py), 2.53 (d, *J* = 6.4 Hz, 4H, 4-CF_3-py).

470MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (s, CF_3).

UV-vis($CHCl_3$) ($\varepsilon\times10^{-4}$) λ_{max} nm: 312(9.3), 418(0.95), 628(5.4).

HR-MALDI-FT-ICR-MS calcd for $C_{44}H_{24}F_6N_{10}Ru$ $[M]^+$: 908.1139. Found: 908.1131.

Bis(*N, N*-dimethyl-4-aminopyridyl) ruthenium(II) phthalocyanine (1c)



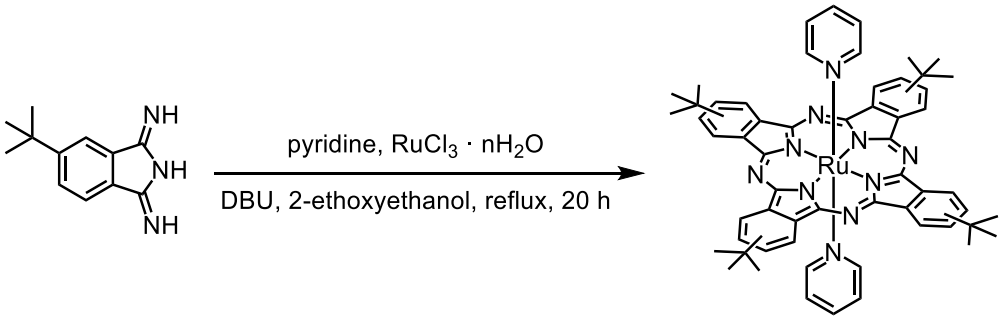
Synthesized according to the procedure for **1a**. Blue solid. (7%)

400 MHz ^{1}H NMR($CDCl_3$) δ(ppm): 9.09-9.07 (m, 8H, Pc-H), 7.83-7.81 (m, 8H, Pc-H), 4.37 (d, *J* = 7.6 Hz, 4H, DMAP), 2.21 (d, *J* = 7.6 Hz, 4H, DMAP), 2.01 (s, 12H, DMAP).

UV-vis($CHCl_3$) ($\varepsilon\times10^{-4}$) λ_{max} nm: 318(7.8), 383(1.9), 571(2.0), 624(6.3).

HR-MALDI-FT-ICR-MS calcd for $C_{46}H_{36}N_{12}Ru$ $[M]^+$: 858.2236. Found: 858.2239.

β-tetrakis(^{t}Bu) bis(pyridyl) ruthenium(II) phthalocyanine (1d)[18]



A mixture of 6-^{t}Bu-1,3-diiminoindoline (500 mg, 2.5 mmol), pyridine (1.3 mL, 17 mmol), $RuCl_3 \cdot nH_2O$ (150 mg, 750 μmol), and DBU (1.0 mL, 6.70 mmol) in 2-ethoxyethanol (5.0 mL) was refluxed for 20 h under Ar atmosphere. After the reaction mixture was cooled, MeOH was added. Then, the precipitate was collected by filtration. After the residue was dried under reduced pressure, the product was purified by silica gel column chromatography ($CHCl_3$). The desired complex was obtained as a blue solid. (5.1 mg, 8%)

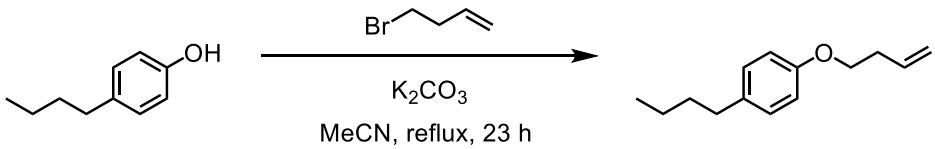
400 MHz ^{1}H NMR($CDCl_3$) δ(ppm): 9.21-9.16 (m, 4H, Pc-H), 9.09-9.03 (m, 4H, Pc-H), 7.97-7.26 (m, 4H, Pc-H), 6.01 (t, *J* = 7.8 Hz, 2H, py), 5.21 (dd, *J* = 7.52, 6.64 Hz, 4H, py), 2.47 (d, *J* = 5.2 Hz, 4H, py), 1.74-1.23 (m, 36H, ^{t}Bu)

UV-vis($CHCl_3$) ($\varepsilon \times 10^{-4}$) λ_{max} nm: 317(6.6), 379(1.4), 632(3.8).

HR-MALDI-FT-ICR-MS calcd for $C_{58}H_{58}N_{10}Ru$ $[M]^+$: 996.3900. Found: 996.3901.

Preparation of substrates

Compound 2i



To a solution of 4-butylphenol (316 mg, 2.1 mmol) and K_2CO_3 (733 mg, 5.3 mmol) in 20 mL of MeCN, 4-bromo-1-butene (549 mg, 4.1 mmol) was added, and the mixture was refluxed for 23 h. The reaction was quenched with water, extracted with EtOAc and washed with water and brine. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo.* The product was purified by silica gel column chromatography (hexane : EtOAc = 9 : 1). The desired compound was obtained as colorless oil. (165 mg,

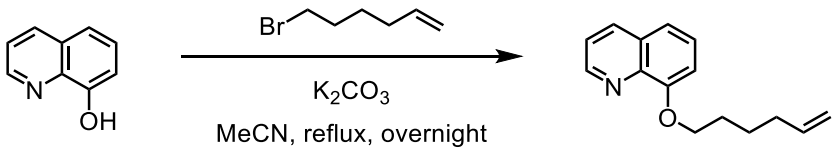
38%)

400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.08 (d, *J* = 8.7 Hz, 2H, Ar), 6.82 (d, *J* = 8.7 Hz, 2H, Ar), 5.94-5.87 (m, 1H, CH), 5.19-5.08 (m, 2H, CH_2), 3.99 (t, *J* = 6.7 Hz, 2H, CH_2), 2.56-2.51 (m, 4H, CH_2×2), 1.59-1.52 (m, 2H, CH_2), 1.37-1.31 (m, 2H, CH_2), 0.92 (t, *J* = 7.3 Hz, 3H, CH_3).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 157.05, 135.19, 134.73, 129.36, 117.01, 114.51, 67.38, 34.87, 34.04, 33.87, 22.44, 14.10.

HR-FAB-MS calcd for $C_{14}H_{21}O$ $[M+H]^+$: 205.1587. Found: 205.1588.

Compound 2k



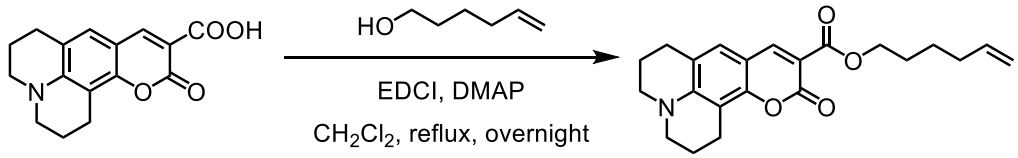
To a solution of 8-hydroxyquinoline (218 mg, 1.5 mmol) and K_2CO_3 (525 mg, 3.8 mmol) in 5 mL of MeCN, 6-bromo-1-hexene (294 mg, 1.8 mmol) was added, and the mixture was refluxed overnight. The reaction was quenched with water, extracted with EtOAc and washed with water. The organic layer was dried by Na_2SO_4 and concentrated in *vacuo.* The product was purified by silica gel column chromatography (hexane : EtOAc = 9 : 1). The desired compound was obtained as colorless oil. (332 mg, 99%)

400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.95 (dd, *J* = 4.2, 1.8 Hz, 1H, Ar), 8.12 (dd, *J* = 8.3, 1.7 Hz, 1H, Ar), 7.46-7.36 (m, 3H, Ar), 7.06 (dd, *J* = 7.7, 1.3 Hz, 1H, Ar), 5.85-5.81 (m, 1H, CH), 5.06-4.95 (m, 2H, CH_2), 4.25 (t, *J* = 7.0 Hz, 2H, CH_2), 2.18-2.14 (m, 2H, CH_2), 2.07-2.03 (m, 2H, CH_2), 1.67-1.63 (m, 2H, CH_2).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 154.92, 149.37, 140.50, 138.58, 135.94, 129.58, 126.74, 121.59, 119.47, 114.87, 108.68, 68.84, 33.59, 28.53, 25.46.

HR-MALDI-FT-ICR-MS calcd for $C_{15}H_{17}NO$ $[M]^+$: 228.1383. Found: 228.1384.

Compound 4



A mixture of Coumarin 343 (77 mg, 0.20 mmol), 5-hexen-1-ol (64 mg, 0.64 mmol), *N, N*-dimethylaminopyridine (DMAP) (25 mg, 0.20 mmol), 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI) (78 mg, 0.41 mmol), in 20 mL of CH_2Cl_2 was refluxed overnight. The reaction was quenched with water, extracted with CH_2Cl_2 and washed with 1N HCl (×2), sat. $NaHCO_3$aq and brine. The organic layer was dried over Na_2SO_4 and concentrated in *vacuo.* The product was purified by silica gel column chromatography ($CHCl_3$: MeOH = 30 : 1). The desired compound was obtained as a yellow solid. (64 mg, 87%).

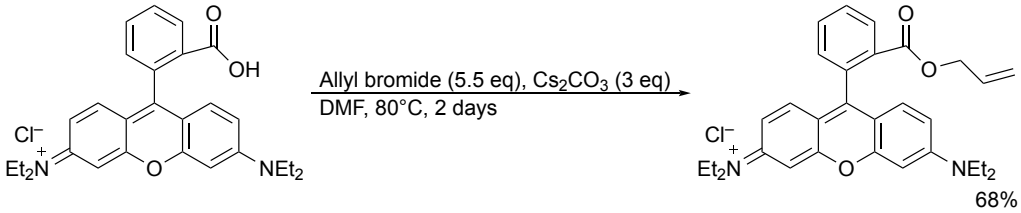
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.30 (s, 1H, Ar), 6.94 (s, 1H, Ar), 5.91-5.73 (m, 1H, CH), 5.10-4.90 (m, 2H, CH_2), 4.29 (t, *J* = 6.7 Hz, 2H, CH_2), 3.33 (q, *J* = 6.5 Hz, 4H, CH_2×2), 2.87 (t, *J* = 6.4 Hz 2H, CH_2), 2.75 (t, *J* = 6.2 Hz 2H, CH_2), 2.19-2.04 (m, 2H, CH_2), 2.01-1.93 (m, 4H, CH_2×2), 1.84-1.71 (m, 2H, CH_2), 1.64-1.46 (m, 2H, CH_2)

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 164.76, 158.74, 153.61, 149.20, 148.58, 138.65, 127.06, 119.23, 114.90, 107.81, 107.66, 105.93, 65.00, 50.39, 50.01, 33.49, 28.33, 27.56, 25.41, 21.32, 20.34, 20.22.

UV-vis($CHCl_3$) ($\varepsilon\times10^{-4}$) λ_{max} nm: 437(1.7).

HR-MALDI-FT-ICR-MS calcd for $C_{22}H_{25}NO_4$ $[M]^+$: 367.1778. Found: 367.1792.

Compound 6[19]



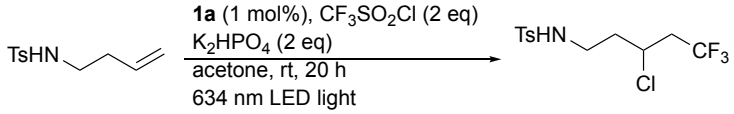
To a flask, rhodamine B (479 mg, 1.0 mmol), allyl bromide (658 mg, 5.4 mmol), and Cs_2CO_3 (990 mg, 3.0 mmol) were dissolved in 10 mL of dry DMF and the mixture was heated at 80°C for 2 days. Then, the solvent was removed *in vacuo*. The product was purified by silica gel column chromatography ($CHCl_3$: MeOH = 100 : 1). The desired compound was obtained as a gold solid. (352 mg, 68%).

500 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.30 (dd, *J* = 7.9, 0.9 Hz, 1H, Ar), 7.82 (td, *J* = 7.5, 1.3 Hz, 1H, Ar), 7.73 (td, *J* = 7.7, 1.3 Hz, 1H, Ar), 7.31 (dd, *J* = 7.6, 0.9 Hz, 1H, Ar), 7.06 (d, *J* = 9.5 Hz, 2H, Ar), 6.91 (dd, *J* = 9.5, 2.5 Hz, 2H, Ar), 6.80 (d, *J* = 2.5 Hz, 2H, Ar), 5.71-5.66 (m, 1H, CH), 5.21-5.10 (m, 2H, CH_2), 4.50 (dt, *J* = 5.9, 1.3 Hz 2H, CH_2), 3.65 (q, *J* = 7.2 Hz 8H, CH_2×4), 1.32 (t, *J* = 7.1 Hz 12H, CH_3×4).

100MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 164.86, 158.83, 157.91, 155.69, 133.80, 133.35, 131.46, 131.40, 131.25, 130.53, 130.47, 130.05, 119.24, 114.48, 113.73, 96.57, 66.26, 46.37, 12.85.

UV-vis($CHCl_3$) ($\varepsilon\times10^{-4}$) λ_{max} nm: 260(2.6), 352(0.64), 557(8.8).

General procedure for the red-light-mediated chlorotrifluoromethylation of alkenes: Compound 3a



2a **3a** 94%

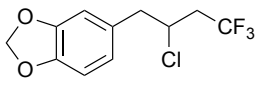
In a 4 mL glass-vial, **1a** (3.9 mg, 5 μmol), K_2HPO_4 (174.1 mg, 1 mmol), and **2a** (59 mg, 0.5 mmol) were suspended in acetone (3 mL) and the solution was degassed by argon. After CF_3SO_2Cl (169.0 mg, 1 mmol) was added, the reaction mixture was stirred for 20 h and irradiation with 634 nm red-LED light, then the reaction was quenched with water. The mixture was extracted with ethyl acetate, and then washed with water and brine. The organic layer was dried over Na_2SO_4. The solvent was removed and purified using flash column chromatography on silica gel (hexane:ethyl acetate = 9:1 v/v). Compound **3a**[20] was obtained (155 mg, 94%) as a white solid.

500 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.75 (d, *J* = 8.3 Hz, 2H, Ar), 7.33 (d, *J* = 8.0 Hz, 2H, Ar), 4.61 (br t, *J* = 6.5 Hz, 1H, NH), 4.18-4.16 (m, 1H, CH), 3.23-3.12 (m, 2H, CH_2), 2.67-2.57 (m, 1H, CH_2), 2.54-2.46 (m, 1H, CH_2), 2.44 (s, 3H, CH_3), 2.13-2.07 (m, 1H, CH_2), 1.87-1.80 (m, 1H, CH_2).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 143.95, 136.65, 130.00, 127.21 (q, $J_{C,F}$ = 277.7 Hz), 51.32 (q, $J_{C,F}$ = 3.3 Hz), 42.44 (q, $J_{C,F}$ = 28.7 Hz), 40.11, 37.88, 21.63.

470 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 9.7 Hz, CF_3).

Compound 3b[21]

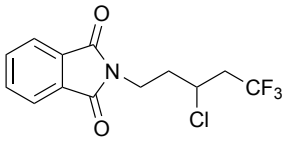


400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 6.77 (d, J = 7.9 Hz, 1H, Ar), 6.71 (d, J = 1.5 Hz, 1H, Ar), 6.66 (dd, J = 7.9, 1.7 Hz, 1H, Ar), 5.96 (s, 2H, CH_2), 4.28-4.21 (m, 1H, CH), 3.02 (d, J = 6.9 Hz, 2H, CH_2), 2.62-2.49 (m, 2H, CH_2).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 148.00, 147.01, 130.03, 125.48 (q, $J_{C,F}$ = 277.5 Hz), 122.75, 109.81, 108.53, 101.25, 54.32 (q, $J_{C,F}$ = 3.0 Hz), 44.25, 41.34 (q, $J_{C,F}$ = 28.7 Hz).

376 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -66 (t, $J_{F,H}$ = 10.1 Hz, CF_3).

Compound 3c[22]



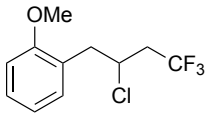
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.87-7.84 (m, 2H, Ar), 7.76-7.71 (m, 2H, Ar), 4.18-4.15 (m, 1H, CH), 3.96-3.85 (m, 2H, CH_2), 2.69-2.59 (m, 2H, CH_2), 2.29-2.25 (m, 1H, CH_2), 2.17-2.11 (m, 1H, CH_2).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 168.29, 134.29, 132.11, 123.78 (q, $J_{C,F}$ = 277.7 Hz), 123.56, 51.49 (q, $J_{C,F}$ = 3.3 Hz), 42.40 (q, $J_{C,F}$ = 28.9 Hz), 36.59, 35.18.

376 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.2 Hz, CF_3).

HR-APCI-FT-ICR-MS calcd for $C_{13}H_{11}ClF_3NO_2$ $[M]^+$: 306.0503. Found: 306.0502.

Compound 3d[21]

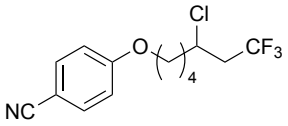


500 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.30-7.26 (m, 1H, Ar), 7.15 (d, J = 7.3 Hz, 1H, Ar), 6.92 (tt, J = 7.5, 1.4 Hz, 1H, Ar), 6.88 (d, J = 8.1 Hz, 1H, Ar), 4.45 (p, J = 6.6 Hz, 1H, CH), 3.84 (d, J = 1.6 Hz, 3H, CH_3), 3.15 (dd, J = 13.6, 6.5 Hz, 1H, CH_2) 3.07 (dd, J = 13.6, 6.5 Hz, 1H, CH_2), 2.59-2.52 (m, 2H, CH_2).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 157.64, 131.66, 128.93, 125.66 (q, $J_{C,F}$ = 277.5 Hz), 124.96, 120.66, 110.61, 55.37, 53.07 (q, $J_{C,F}$ = 3.1 Hz), 41.65 (q, $J_{C,F}$ = 28.6 Hz), 40.13.

470 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.2 Hz, CF_3).

Compound 3e[23]



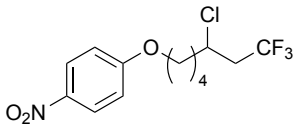
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.58 (d, J = 8.9 Hz, 2H, Ar), 6.93 (d, J = 8.9 Hz, 2H, Ar), 4.16-4.14 (m, 1H, CH), 4.02 (t, J = 6.1 Hz, 2H, CH_2), 2.66-2.53 (m, 2H, CH_2), 1.94-1.64 (m, 6H, CH_2×3).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 162.32, 134.10, 125.32(q, $J_{C,F}$ = 277.6 Hz), 119.34, 115.26, 104.01, 67.99, 54.01 (q, $J_{C,F}$ = 3.2 Hz), 42.53 (q, $J_{C,F}$ = 28.5 Hz), 37.70, 28.36, 22.76.

376 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.3 Hz, CF_3).

HR-MALDI-FT-ICR-MS calcd for $C_{14}H_{15}ClF_3NO$ $[M]^+$: 306.0867. Found: 306.0871.

Compound 3f



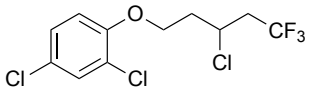
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.20 (d, J = 9.3 Hz, 2H, Ar), 6.94 (d, J = 9.3 Hz, 2H, Ar), 4.16-4.14 (m, 1H, CH), 4.08 (t, J = 6.1 Hz, 2H, CH_2), 2.67-2.54 (m, 2H, CH_2), 1.95-1.65 (m, 6H, CH_2×3).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 164.10, 141.60, 126.05, 125.34 (q, $J_{C,F}$ = 277.6 Hz), 114.51, 68.48, 54.02 (q, $J_{C,F}$ = 3.3 Hz), 42.56(q, $J_{C,F}$ = 28.5 Hz), 37.71, 28.39, 22.77.

376 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.1 Hz, CF_3).

HR-MALDI-FT-ICR-MS calcd for $C_{13}H_{15}ClF_3NO_3$ $[M]^+$: 326.0765. Found: 326.0769.

Compound 3g



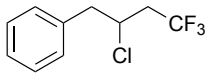
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.40 (d, J = 2.5 Hz, 1H, Ar), 7.22 (dd, J = 8.8, 2.5 Hz, 1H, Ar), 6.89 (d, J = 8.8 Hz, 1H, Ar) 4.70-4.42 (m, 1H, CH), 4.37-4.07 (m, 2H, CH_2), 2.97-2.62 (m, 2H, CH_2), 2.56-2.36 (m, 1H, CH_2), 2.29-2.00 (m, 1H, CH_2).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 153.01, 130.23, 127.79, 126.45, 125.30 (q, $J_{C,F}$ = 277.6 Hz), 124.05, 114.36, 65.60, 50.98 (q, $J_{C,F}$ = 28.7 Hz), 37.52.

376MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.2 Hz, CF_3).

HR-APCI-FT-ICR-MS calcd for $C_{11}H_{10}Cl_3F_3O$ $[M]^+$: 319.9744. Found: 319.9744.

Compound 3h[21]

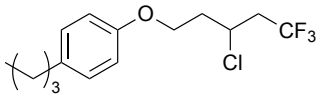


400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.37-7.21 (m, 5H, Ar), 4.36-4.30 (m, 1H, CH), 3.11 (d, 2H, CH_2), 2.62-2.53 (m, 2H, CH_2).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 136.40, 129.55, 128.83, 127.50, 125.47 (q, $J_{C,F}$ = 278.76 Hz), 54.16 (q, $J_{C,F}$ = 3.03 Hz), 44.58, 41.49 (q, $J_{C,F}$ = 28.28 Hz).

376 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 11.3 Hz, CF_3).

Compound 3i



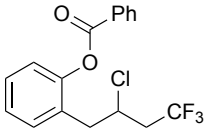
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 7.10 (d, J = 8.7 Hz, 2H, Ar), 6.82 (d, J = 8.6 Hz, 2H, Ar), 4.45-4.43 (m, 1H, CH), 4.18-4.12 (m, 2H, CH_2), 2.72-2.63 (m, 2H, CH_2), 2.55 (t, J = 7.7 Hz, 2H, CH_2), 2.37-2.34 (m, 1H, CH_2), 2.15-2.12 (m, 1H, CH_2), 1.60-1.53 (m, 2H, CH_2) , 1.37-1.32 (m, 2H, CH_2), 0.92 (t, J = 7.3 Hz, 3H, CH_3).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 156.63, 135.72, 129.49, 126.74 (q, $J_{C,F}$ = 277.7 Hz), 114.48, 64.05, 51.16 (q, $J_{C,F}$ = 3.3 Hz), 42.69 (q, $J_{C,F}$ = 28.6 Hz), 37.88, 34.88, 34.03, 22.44, 14.10.

376 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.1 Hz, CF_3).

HR-MALDI-FT-ICR-MS calcd for $C_{15}H_{20}ClF_3O$ $[M]^+$: 308.1149. Found: 308.1151.

Compound 3j



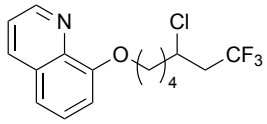
500 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.21-8.19 (m, 2H, Ar), 7.69-7.66 (m, 1H, Ar), 7.56-7.53 (m, 1H, Ar), 7.40-7.34 (m, 2H, Ar), 7.30-7.26 (m, 1H, Ar), 7.23-7.21 (m, 1H, Ar), 4.41-4.35 (m, 1H, CH), 3.16 (dd, J = 14.4, 5.9 Hz, 1H, CH_2), 3.04 (dd, J = 14.3, 8.3 Hz, 1H, CH_2), 2.60-2.56 (m, 2H, CH_2).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 164.95, 149.49, 133.98, 131.57, 130.16, 128.99, 128.84, 128.80, 128.79, 126.34, 125.20 (q, $J_{C, F}$ = 277.6 Hz), 122.94, 53.10 (q, $J_{C, F}$ = 3.1 Hz), 41.86 (q, $J_{C, F}$ = 28.7 Hz), 39.38.

470 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F, H}$ = 9.5 Hz, CF_3).

HR-MALDI-FT-ICR-MS calcd for $C_{17}H_{14}ClF_3O_2$ $[M]^+$: 343.0707. Found: 343.0711.

Compound 3k



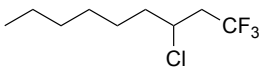
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.95 (dd, J = 4.2, 1.8 Hz, 1H, Ar), 8.13 (dd, J = 8.3, 1.8 Hz, 1H, Ar), 7.50-7.36 (m, 3H, Ar), 7.06 (dd, J = 7.6, 1.4 Hz, 1H, Ar), 4.27 (t, J = 6.7 Hz, 2H, CH_2), 4.22-4.11 (m, 1H, CH), 2.71-2.49 (m, 2H, CH_2), 2.17-1.66 (m, 6H, CH_2×3).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 154.75, 149.40, 140.39, 136.11, 129.64, 126.79, 125.40 (q, $J_{C, F}$ = 277.7 Hz), 121.70, 119.76, 108.87, 68.66, 54.09 (q, $J_{C, F}$ = 3.3 Hz), 42.53 (q, $J_{C, F}$ = 28.4 Hz), 37.93, 28.33, 22.98.

376 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F, H}$ = 10.4 Hz, CF_3).

HR-MALDI-FT-ICR-MS calcd for $C_{16}H_{17}ClF_3NO$ $[M]^+$: 332.1024. Found: 332.1025.

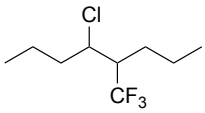
Compound 3l[24]



400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 4.14-4.08 (m, 1H, CH), 2.62-2.53 (m, 2H, CH_2), 1.83-1.73 (m, 2H, CH_2), 1.55-1.42 (m, 8H, CH_2×4) 0.91-0.88 (m, 3H, CH_3).

376 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F, H}$ = 10.2 Hz, CF_3).

Compound 3m



(*dr* 1 : 1.7)

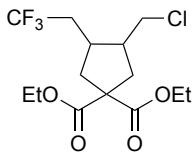
400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 4.24-4.14 (m, 1H), 2.62-2.46 (m, 0.40H), 2.42-2.27 (m, 0.67H), 1.89-1.32 (m, 8H), 0.98-0.92 (m, 6H).

100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 127.30 (q, $J_{C, F}$ = 281.5 Hz), 125.68 (q, $J_{C, F}$ = 281.7 Hz), 59.87 (q, $J_{C, F}$ = 3.0 Hz), 59.08 (q, $J_{C, F}$ = 3.3 Hz), 49.46 (q, $J_{C, F}$ = 24.2 Hz), 48.45 (q, $J_{C, F}$ = 24.7 Hz), 38.59, 36.14 (d, $J_{C, F}$ = 1.6 Hz), 26.79 (q, $J_{C, F}$ = 1.8 Hz), 26.54 (q, $J_{C, F}$ = 2.0 Hz), 21.37, 21.10, 20.52, 20.25, 14.21, 14.05, 13.45, 13.44.

376 MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -69 (d, $J_{F, H}$ = 9.6 Hz, CF_3), -70 (d, $J_{F, H}$ = 9.1 Hz, CF_3).

HR-APCI-FT-ICR-MS calcd for $C_9H_{16}ClF_3$ $[M+H]^+$: 217.0965. Found: 217.0990.

Compound 3n[21]

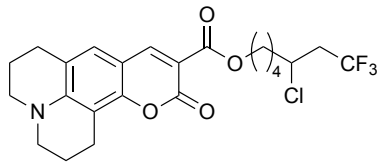


500 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 4.20 (m, *J* = 7.2 Hz, 2H, CH_2×2), 3.57-3.38 (m, 2H, CH×2), 2.72-2.42 (m, 4H, CH_2×2), 2.41-1.95 (m, 1H, CH_2+CH×2), 1.25 (t, *J* = 7.1 Hz, 3H, CH_3), 1.25 (t, *J* = 7.1 Hz, 3H, CH_3).

100 MHz ^{13}C-NMR($CDCl_3$) δ(ppm): 172.20, 172.11, 126.94 (q, $J_{C, F}$ = 277.1 Hz), 61.96, 61.92, 58.62, 44.43, 43.98, 38.61, 37.08, 35.56 (q, $J_{C, F}$ = 2.5 Hz), 33.37 (q, $J_{C, F}$ = 28.7 Hz), 14.10.

470 MHz ^{19}F-NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.5 Hz, CF_3).

Compound 5



400 MHz ^{1}H NMR ($CDCl_3$) δ(ppm): 8.31 (s, 1H, Ar), 6.93 (s, 1H, Ar), 4.31 (t, J = 6.3 Hz, 2H, CH_2), 4.19-4.08 (m, 1H, CH), 3.33 (q, J = 6.3 Hz, 4H, CH_2×2), 2.87 (t, J = 6.4 Hz, 2H, CH_2), 2.81-2.71 (m, 2H, CH_2), 2.70-2.50 (m, 2H, CH_2), 2.02-1.92 (m, 4H, CH_2×2), 1.88-1.69 (m, 4H, CH_2×2), 1.67-1.51 (m, 2H, CH_2).

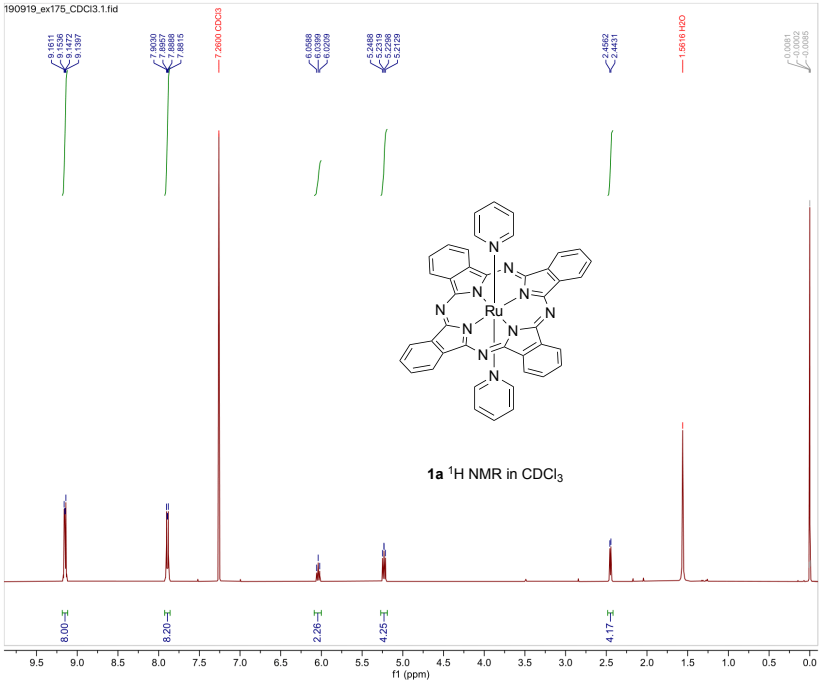
100 MHz ^{13}C NMR($CDCl_3$) δ(ppm): 164.77, 158.72, 153.61, 149.32, 148.69, 127.08, 125.40 (q, $J_{C,F}$ = 277.6 Hz), 119.31, 107.62, 107.36, 105.84, 64.53, 54.11 (q, $J_{C,F}$ = 3.2 Hz), 50.37, 49.98, 42.49 (q, $J_{C,F}$ = 28.4 Hz), 37.73, 28.07, 27.52, 22.73, 21.25, 20.27, 20.17.

470MHz ^{19}F NMR ($CDCl_3$) δ(ppm): -67 (t, $J_{F,H}$ = 10.3 Hz, CF_3).

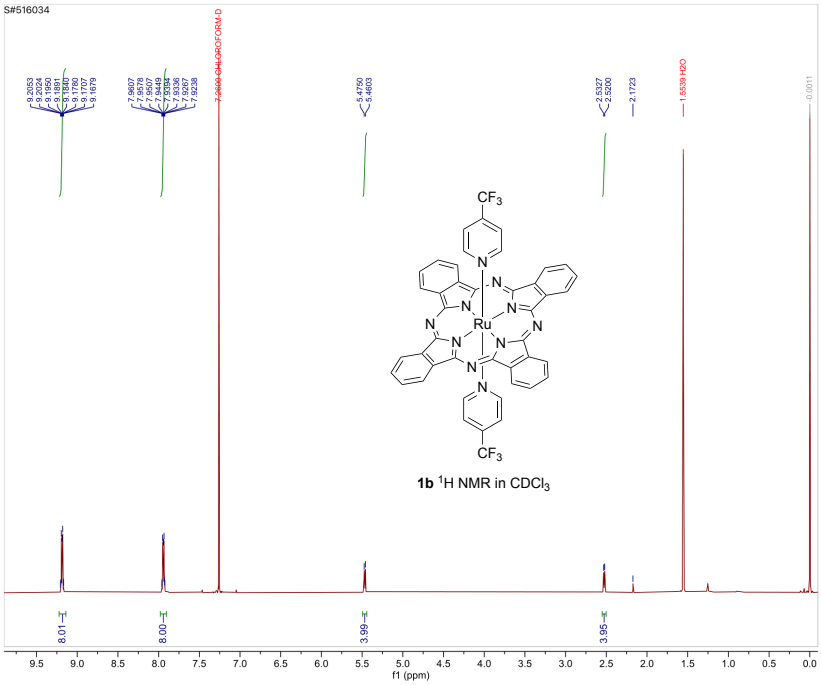
UV-vis($CHCl_3$) ($\varepsilon\times10^{-4}$) λ_{max} nm: 438(3.8).

HR-FAB-MS calcd for $C_{23}H_{25}ClF_3NO_4$ $[M]^+$: 471.1419. Found: 471.1415.

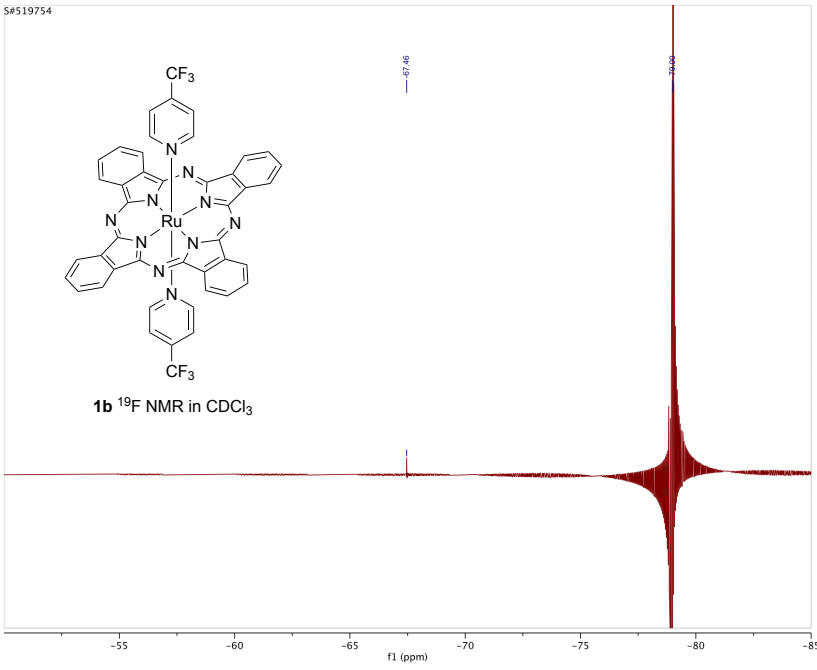
Copies of the NMR Spectra of Studied Compounds



	パラメータ	値
1	データファイル名	C:/ Users/ furuyama/ Documents/ Personaldata/ ishikawa/ NMR/ 生data/ 190919_ex175_CDCl3/ 1/ fid
2	タイトル	190919_ex175_CDCl3.1.fid
3	コメント	
4	オリジン	Bruker BioSpin GmbH
5	オーナー	nmrsu
6	サイト	
7	装置	Avance
8	作成者	
9	溶媒	CDCl3
10	温度	298.1
11	パルスシーケンス	zg30
12	実験	1D
13	プローブ	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	スキャン数	16
15	レシーバーゲイン	101.0

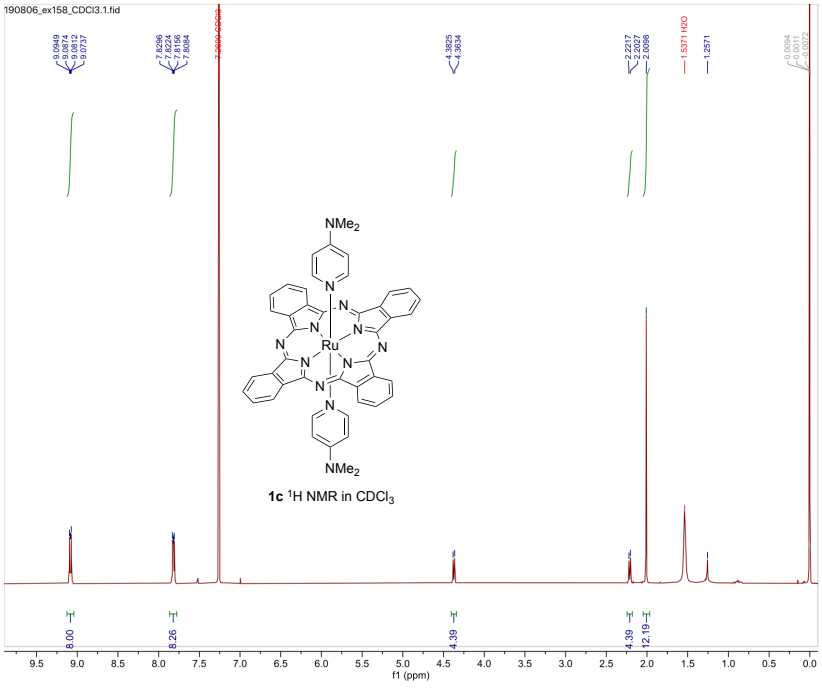


	パラメータ	値
1	データファイル名	C:/ Users/ furuyama/ Documents/ Personaldata/ ishikawa/ NMR/ 生data/ 191002_ex176_CDCl3-1.jdf
2	タイトル	S#516034
3	コメント	
4	オリジン	JEOL
5	オーナー	
6	サイト	
7	装置	ECA 500
8	作成者	delta
9	溶媒	CHLOROFORM-D
10	温度	21.5
11	パルスシーケンス	single_pulse.ex2
12	実験	1D
13	プローブ	2756
14	スキャン数	8
15	レシーバーゲイン	58.0



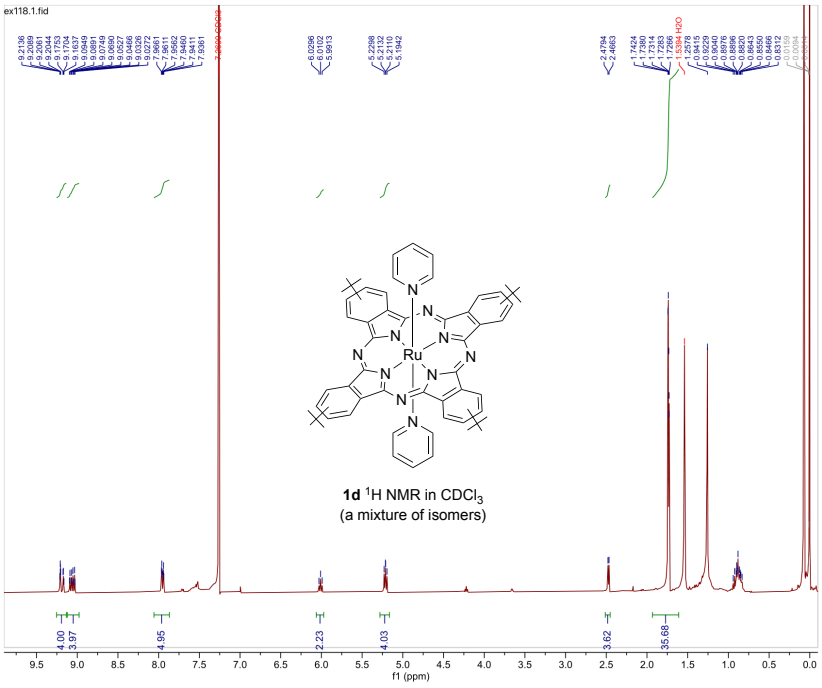
1b ^{19}F NMR in $CDCl_3$

	パラメータ	値
1	データファイル名	C:/ Users/ furuyama/ Documents/ Personaldata/ ishikawa/ NMR/ 生data/ 191002_ex176_CDCl3_19F-1.jdf
2	タイトル	S#519754
3	コメント	
4	オリジン	JEOL
5	オーナー	
6	サイト	
7	装置	ECA 500
8	作成者	delta
9	溶媒	CHLOROFORM-D
10	温度	21.4
11	パルスシーケンス	single_pulse.ex2
12	実験	1D
13	プ	2756

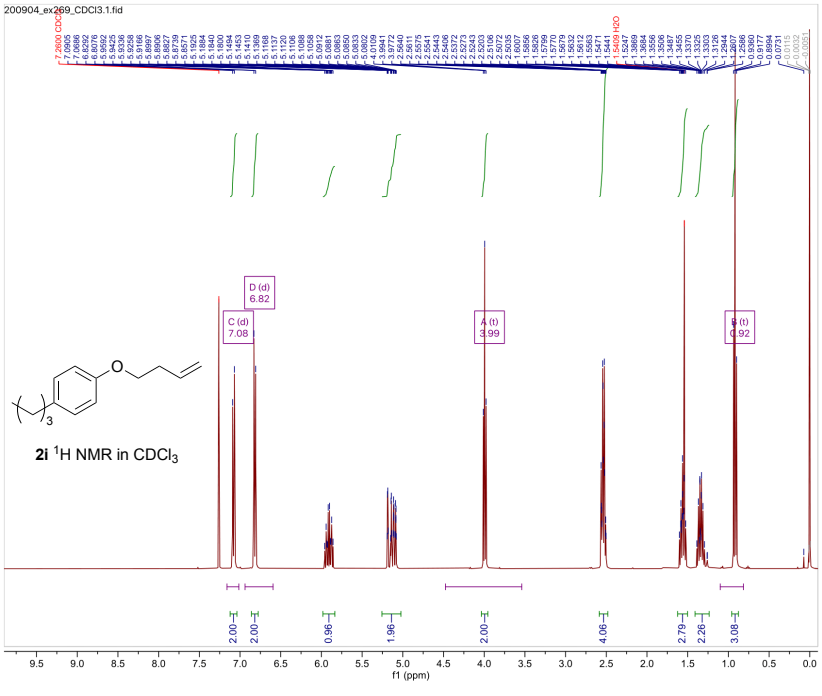


1c ^{1}H NMR in $CDCl_3$

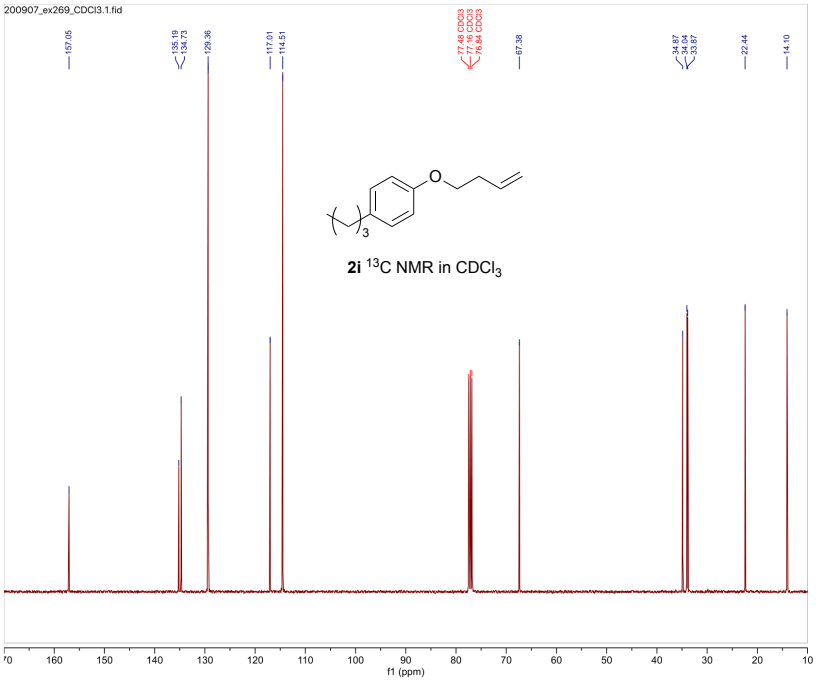
	パラメータ	値
1	データファイル名	E:/ NMR/ 190806_ex158_CDCl3/ 1/ fid
2	タイトル	190806_ex158_CDCl3.1.fid
3	コメント	
4	オリジン	Bruker BioSpin GmbH
5	オーナー	nmrsu
6	サイト	
7	装置	Avance
8	作成者	
9	溶媒	CDCl3
10	温度	301.1
11	パルスシーケンス	zg30
12	実験	1D
13	プローブ	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	スキャン数	16
15	レシーバーゲイン	101.0



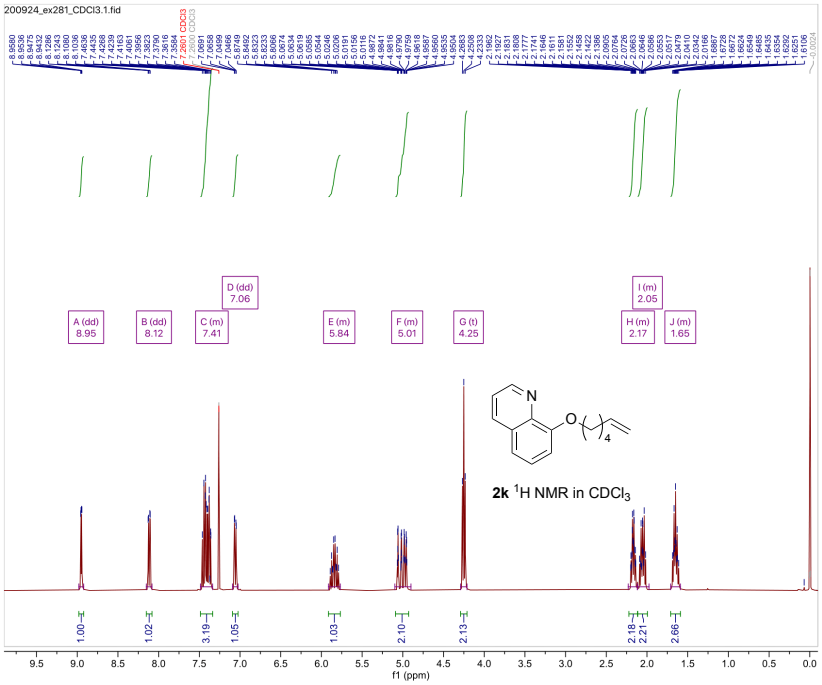
	パラメータ	値
1	データファイル名	E:/ NMR/ ex118/ 1/ fid
2	タイトル	ex118.1.fid
3	コメント	
4	オリジン	Bruker BioSpin GmbH
5	オーナー	nmrsu
6	サイト	
7	装置	Avance
8	作成者	
9	溶媒	CDCl3
10	温度	301.1
11	パルスシーケンス	zg30
12	実験	1D
13	プローブ	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	スキャン数	32
15	レシーバーゲイン	101.0
16	緩和時間	1.0000
17	パルス幅	10.0000
18	固定周波数	



	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200904_ex269_CDCl3/ 1/ fid
2	Title	200904_ex269_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-04T17:53:59
21	Modification Date	2020-09-04T17:48:46

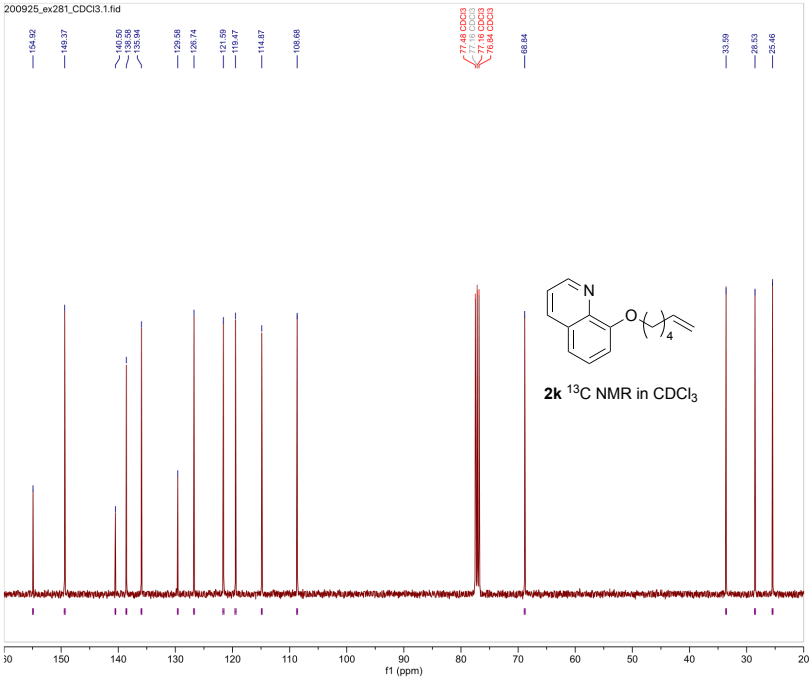


	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200907_ex269_CDCl3/ 1/ fid
2	Title	200907_ex269_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	1024
15	Receiver Gain	36.2
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-07T15:55:18



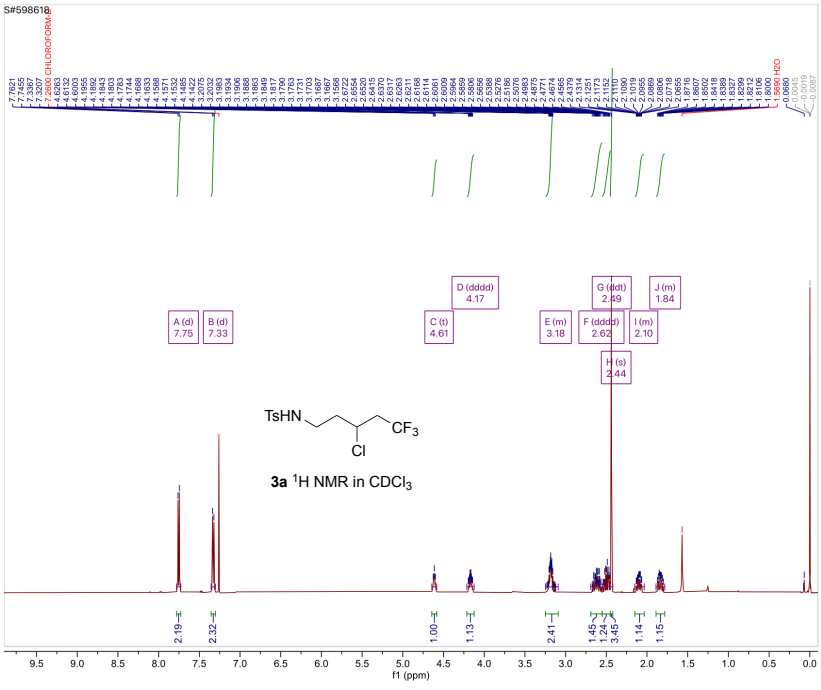
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.95 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.12 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.49 – 7.34 (m, 3H), 7.06 (dd, *J* = 7.7, 1.3 Hz, 1H), 5.91 – 5.77 (m, 1H), 5.10 – 4.90 (m, 2H), 4.25 (t, *J* = 7.0 Hz, 2H), 2.23 – 2.12 (m, 2H), 2.12 – 1.97 (m, 2H), 1.71 – 1.59 (m, 2H).

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200924_ex281_CDCl3/ 1/ fid
2	Title	200924_ex281_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-24T15:25:08
21	Modification Date	2020-09-24T15:23:56



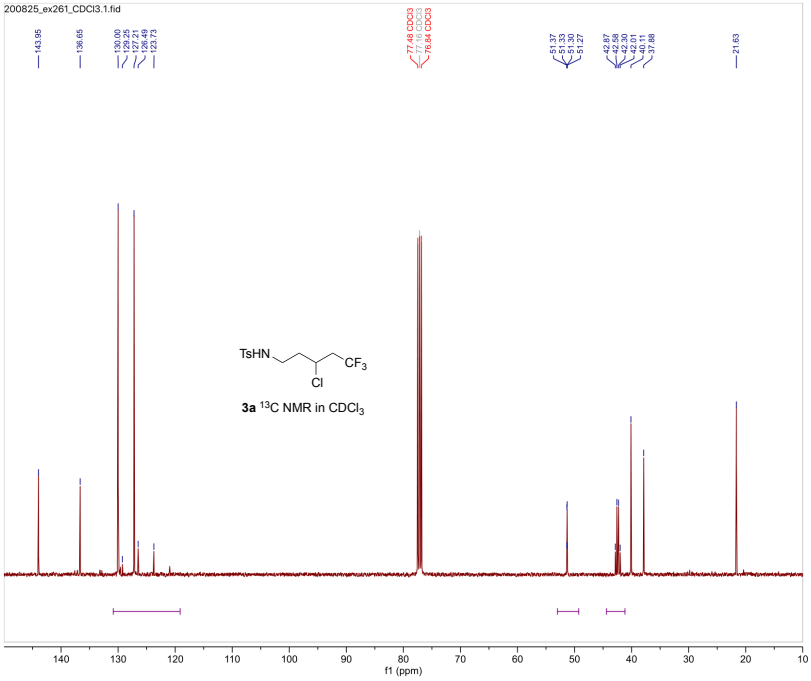
^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.92, 149.37, 140.50, 138.58, 135.94, 129.58, 126.74, 121.59, 119.47, 114.87, 108.68, 68.84, 33.59,

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200925_ex281_CDCl3/ 1/ fid
2	Title	200925_ex281_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	329
15	Receiver Gain	36.5
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-25T15:56:47



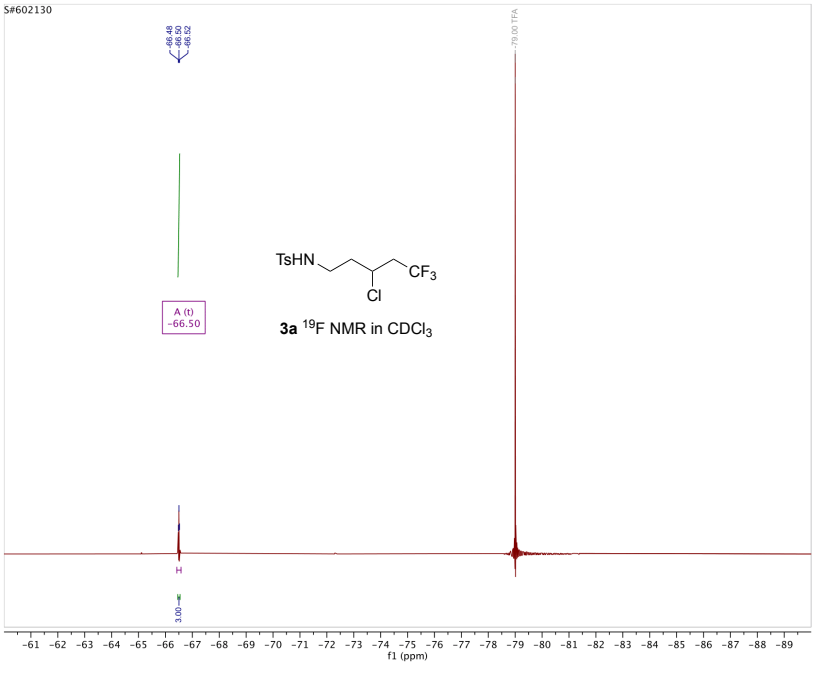
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 4.61 (t, *J* = 6.5 Hz, 1H), 4.17 (dddd, *J* = 10.6, 7.4, 5.5, 3.2 Hz, 1H), 3.25 – 3.09 (m, 2H), 2.62 (dddd, *J* = 20.3, 15.4, 10.1, 7.9 Hz, 1H), 2.49 (ddt, *J* = 15.5, 10.3, 5.0 Hz, 1H), 2.44 (s, 3H), 2.15 – 2.03 (m, 1H), 1.89 – 1.78 (m, 1H).

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200824_ex261_CDCl3-1.jdf
2	Title	S#598618
3	Comment	
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	22.0
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	50.0
16	Relaxation Delay	5.0000
17	Pulse Width	5.7000
18	Presaturation Frequency	
19	Acquisition Time	1.7459
20	Acquisition Date	2020-08-24T16:19:50
21	Modification Date	2020-08-24T16:39:01
22	Class	
23	Spectrometer Frequency	500.16



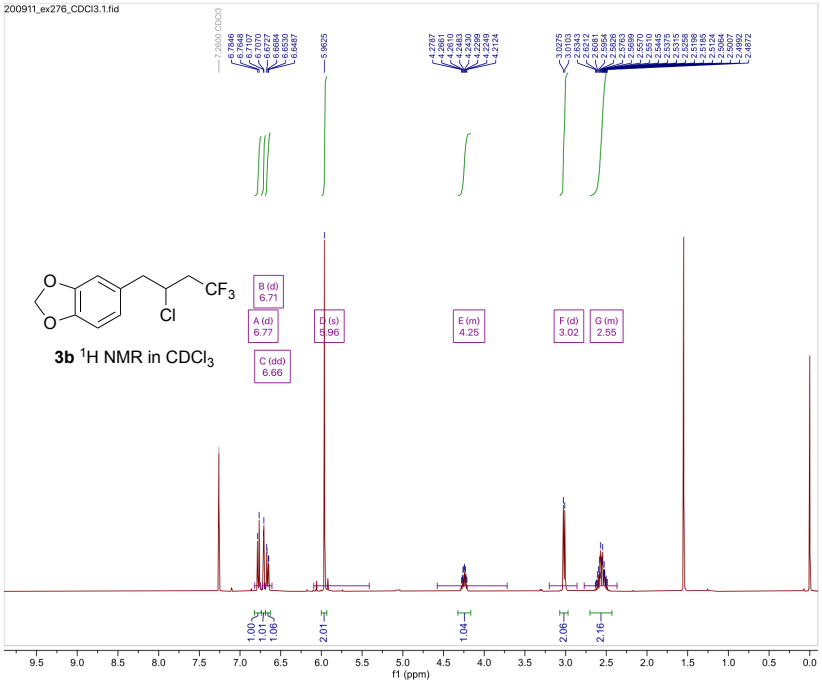
^{13}C NMR (101 MHz, Chloroform-*d*) δ 125.11 (q, J = 277.7 Hz), 51.32 (q, J = 3.3 Hz), 42.44 (q, J = 28.7 Hz).

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200825_ex261_CDCl3/ 1/ fid
2	Title	200825_ex261_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	1024
15	Receiver Gain	36.2
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-08-25T10:30:47



^{19}F NMR (471 MHz, Chloroform-*d*) δ -66.50 (t, J = 9.7 Hz).

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200824_ex261_CDCl3-2.jdf
2	Title	S#602130
3	Comment	
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	22.0
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	4
15	Receiver Gain	44.0
16	Relaxation Delay	5.0000
17	Pulse Width	6.5500
18	Presaturation Frequency	
19	Acquisition Time	0.1520
20	Acquisition Date	2020-08-24T16:25:54
21	Modification Date	2020-08-24T16:45:07
22	Class	
23	Spectrometer	470.62



	Parameter	Value
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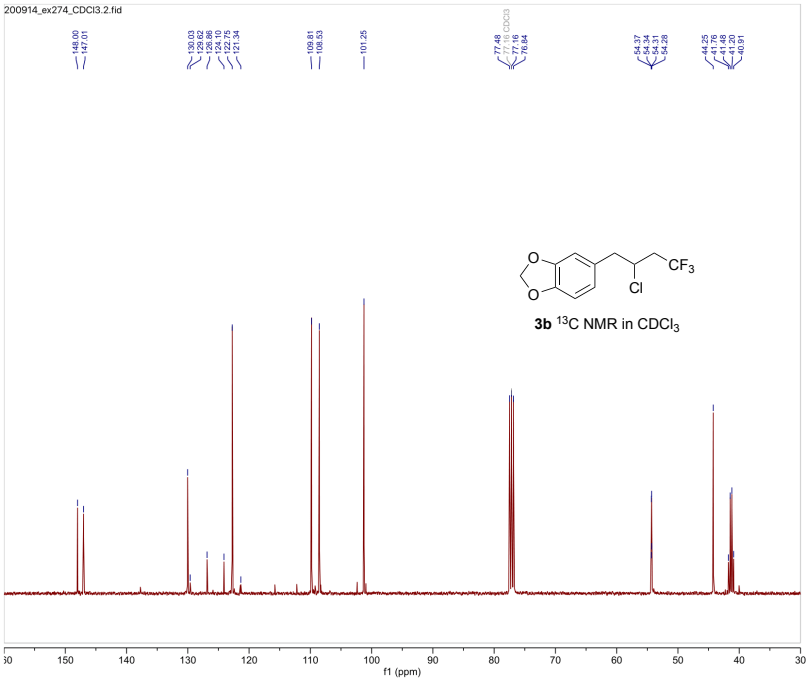
2	Title	200911_ex276_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-11T09:36:20
21	Modification Date	2020-09-11T09:30:28

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.77 (d, *J* = 7.9 Hz, 1H), 6.71 (d, *J* = 1.5 Hz, 1H), 6.66 (dd, *J* = 7.9, 1.7 Hz, 1H), 5.96 (s, 2H), 4.58 – 3.72 (m, 1H), 3.02 (d, *J* = 6.9 Hz, 2H), 2.77 – 2.37 (m, 2H).



	Parameter	Value
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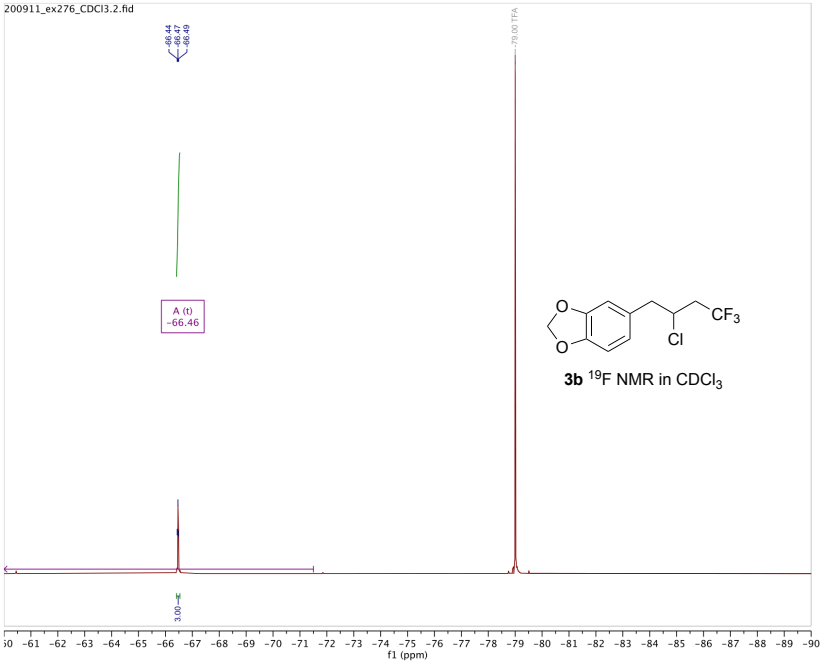
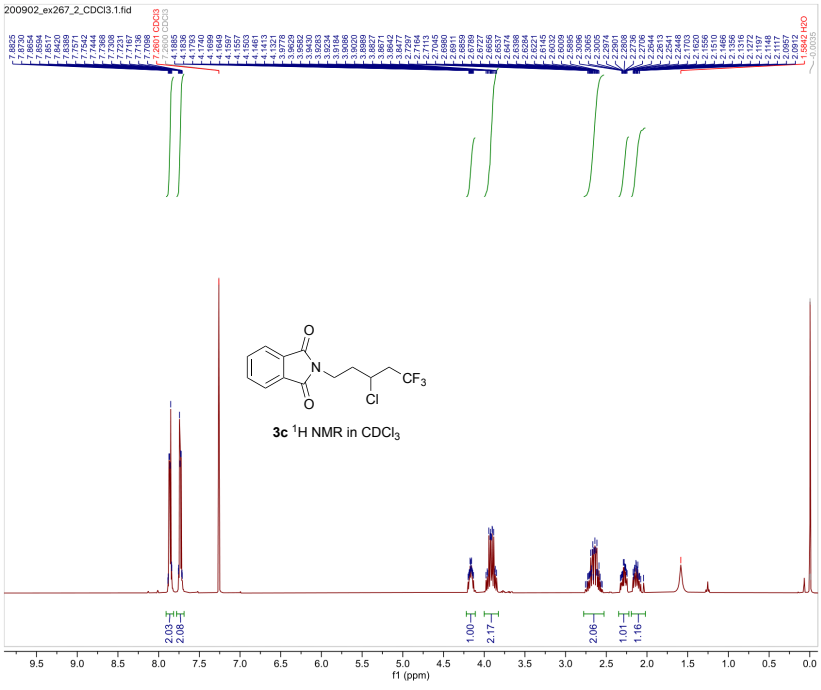
2	Title	200914_ex274_CDCl3.2.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	297.9
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	748
15	Receiver Gain	39.1
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-14T09:50:32

^{13}C NMR (101 MHz, Chloroform-*d*) δ 125.48 (q, *J* = 277.5 Hz), 54.32 (q, *J* = 3.0 Hz), 41.34 (q, *J* = 28.7 Hz).

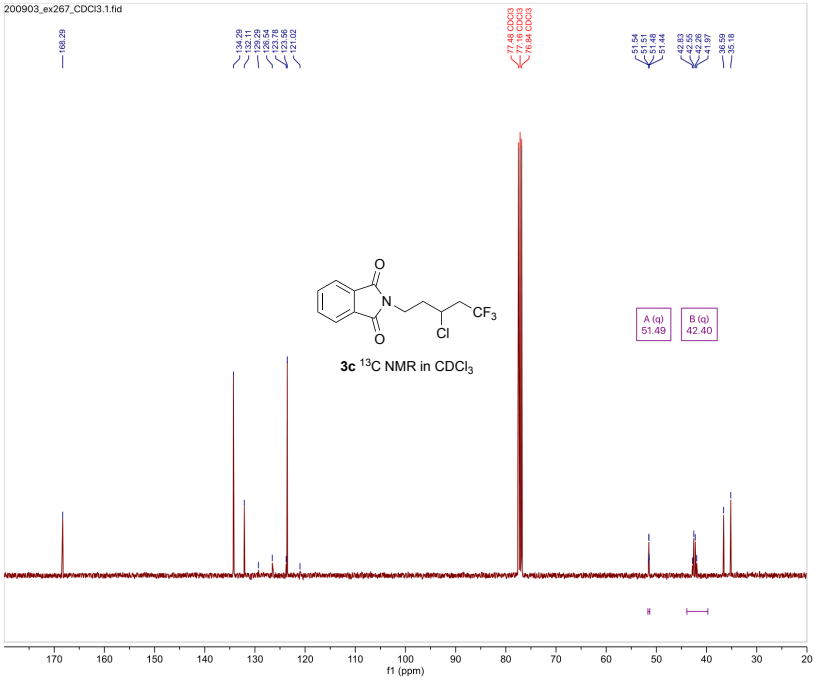


^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.46 (t, *J* = 10.1 Hz).

	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200911_ex276_CDCl3/ 2/ fid
2	Title	200911_ex276_CDCl3.2.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-11T09:40:45
21	Modification Date	2020-09-11T09:3

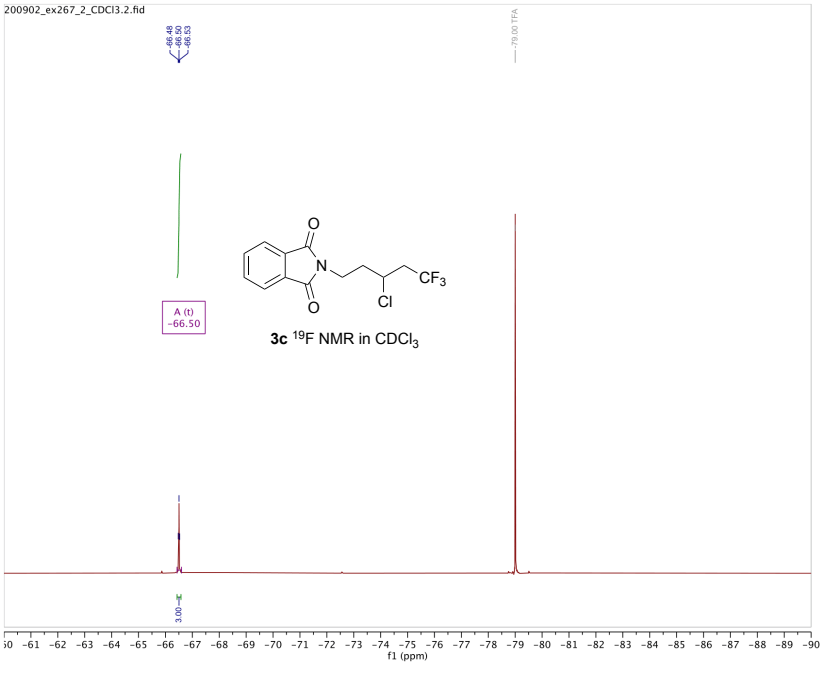


	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200902_ex267_2_CDCl3/ 1/ fid
2	Title	200902_ex267_2_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-02T14:06:19
21	Modification Date	2020-09-02T14:01:18



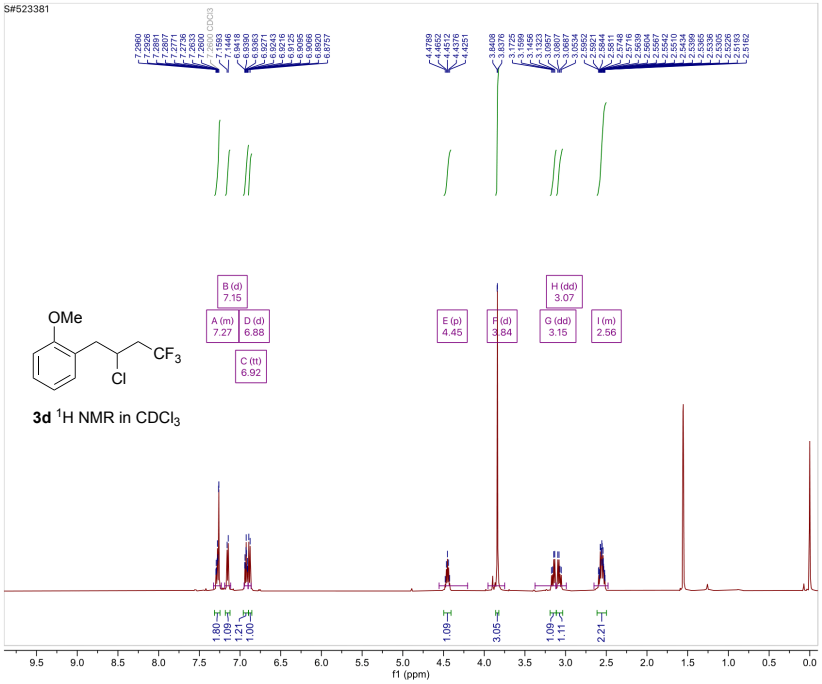
	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200903_ex267_CDCl3/ 1/ fid
2	Title	200903_ex267_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	855
15	Receiver Gain	36.2
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-03T14:52:53

^{13}C NMR (101 MHz, Chloroform-*d*) δ 51.49 (q, *J* = 3.3 Hz), 42.40 (q, *J* = 28.9 Hz).



	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200902_ex267_2_CDCl3/ 2/ fid
2	Title	200902_ex267_2_CDCl3.2.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-02T14:14:44
21	Modification Date	2020-09-02T14:0

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.50 (t, *J* = 10.2 Hz).



	Parameter	Value
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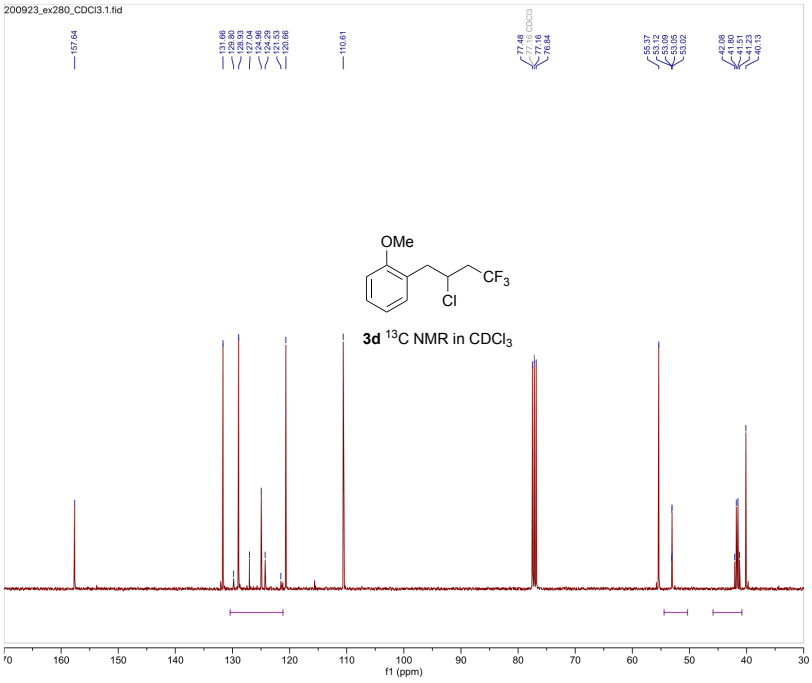
2	Title	S#523381
3	Comment	
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.9
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	58.0
16	Relaxation Delay	5.0000
17	Pulse Width	5.7000
18	Presaturation Frequency	
19	Acquisition Time	1.7459
20	Acquisition Date	2020-09-18T14:14:41
21	Modification Date	2020-09-18T14:33:51
22	Class	
23	Spectrometer Frequency	500.16

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 – 7.23 (m, 1H), 7.15 (d, *J* = 7.3 Hz, 1H), 6.92 (tt, *J* = 7.5, 1.4 Hz, 1H), 6.88 (d, *J* = 8.1 Hz, 1H), 4.45 (p, *J* = 6.6 Hz, 1H), 3.84 (d, *J* = 1.6 Hz, 2H), 3.15 (dd, *J* = 13.6, 6.5 Hz, 1H), 3.07 (dd, *J* = 13.6, 7.6 Hz, 1H), 2.65 – 2.48 (m, 1H).



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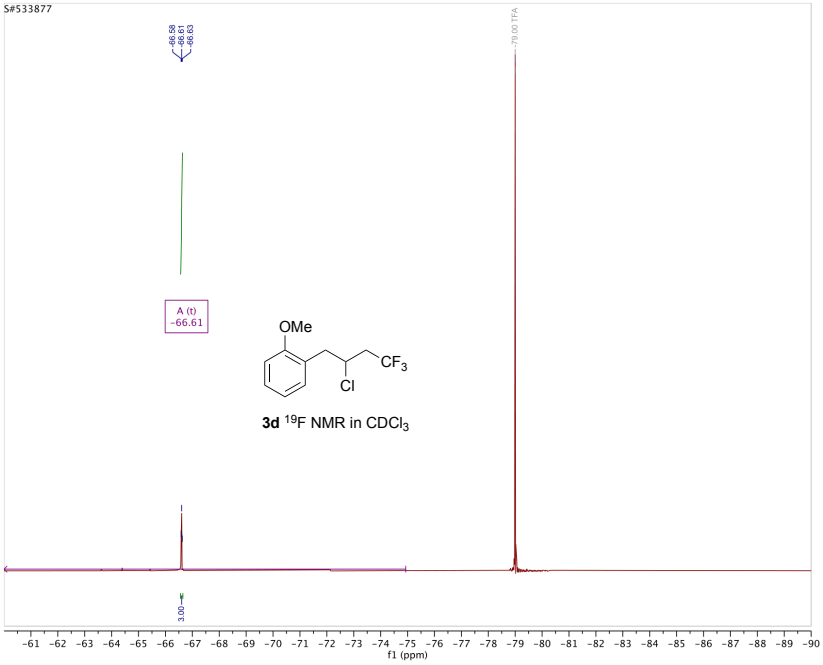
2	Title	200923_ex280_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	720
15	Receiver Gain	33.7
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-23T10:58:12

^{13}C NMR (101 MHz, Chloroform-*d*) δ 125.66 (q, *J* = 277.5 Hz), 53.07 (q, *J* = 3.1 Hz), 41.65 (q, *J* = 28.6 Hz).



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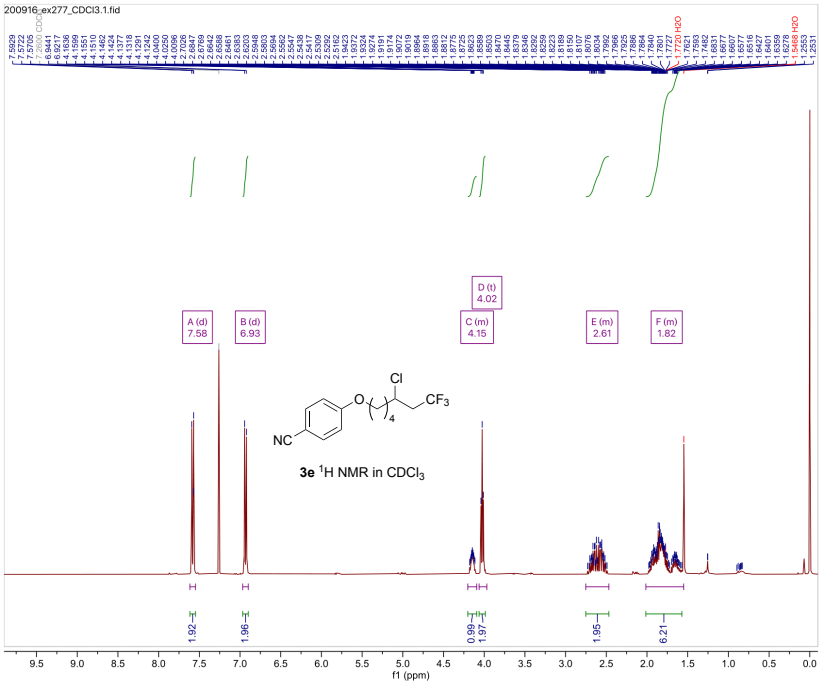
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200918_ex280_CDCl3_19F-1.jdf
2	Title	S#533877
3	Comment	
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.7
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	44.0
16	Relaxation Delay	5.0000
17	Pulse Width	6.5500
18	Presaturation Frequency	
19	Acquisition Time	0.1520
20	Acquisition Date	2020-09-18T14:32:21
21	Modification Date	2020-09-18T14:51:32
22	Class	
23	Spectrometer	470.62

[19]F NMR (471 MHz, Chloroform-*d*) δ -66.61 (t, *J* = 10.2 Hz).



	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200916_ex277_CDCl3/ 1/ fid
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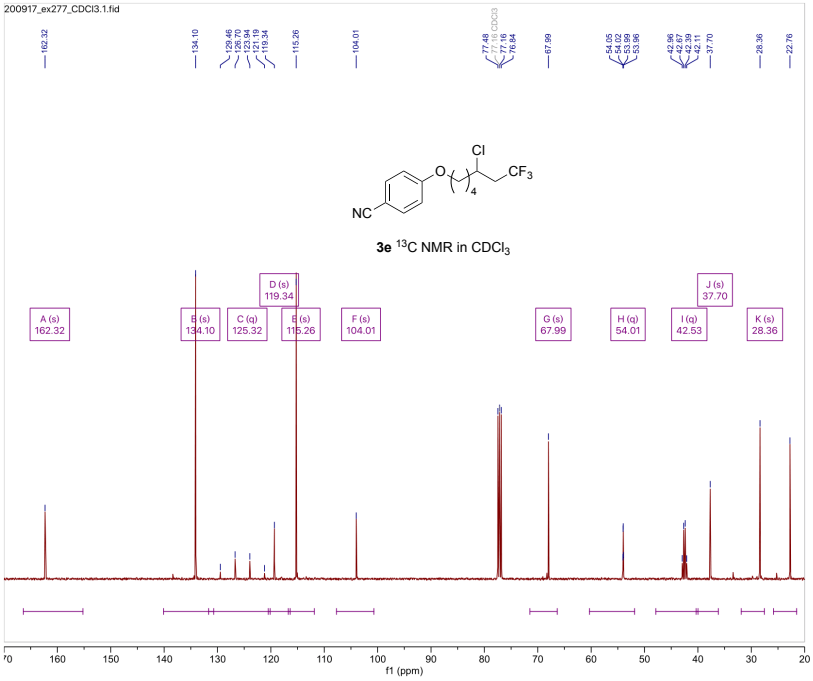
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-16T09:27:14
21	Modification Date	2020-09-16T09:26:48

[1]H NMR (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 4.20 – 4.09 (m, 1H), 4.02 (t, *J* = 6.1 Hz, 2H), 2.75 – 2.47 (m, 2H), 2.01 – 1.55 (m, 6H).



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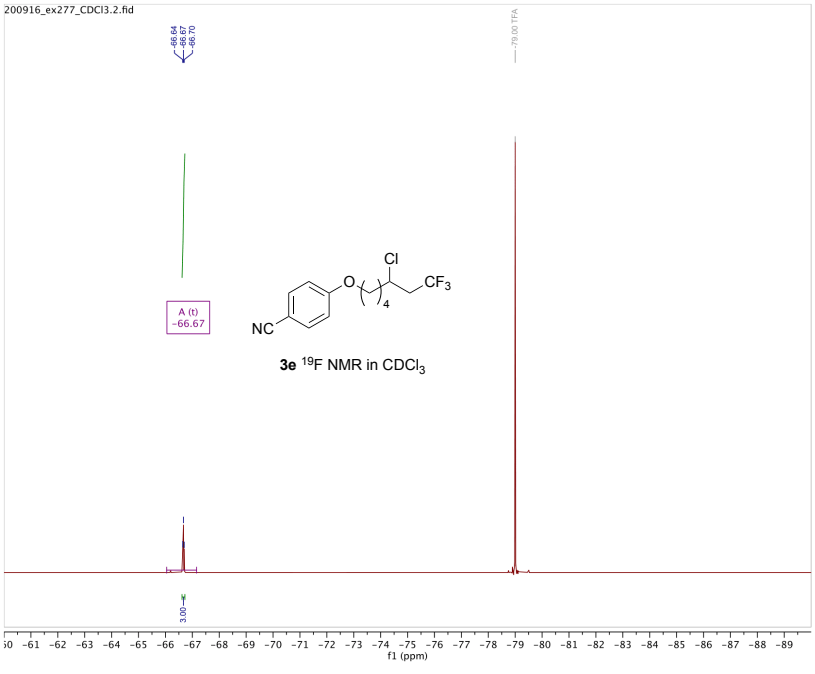
2	Title	200917_ex277_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	729
15	Receiver Gain	34.9
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-17T13:35:44

^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.32, 134.10, 125.32 (q, J = 277.6 Hz), 119.34, 115.26, 104.01, 67.99, 54.01 (q, J = 3.2 Hz), 42.53 (q, J = 28.5 Hz), 37.70, 28.36, 22.76.



	Parameter	Value
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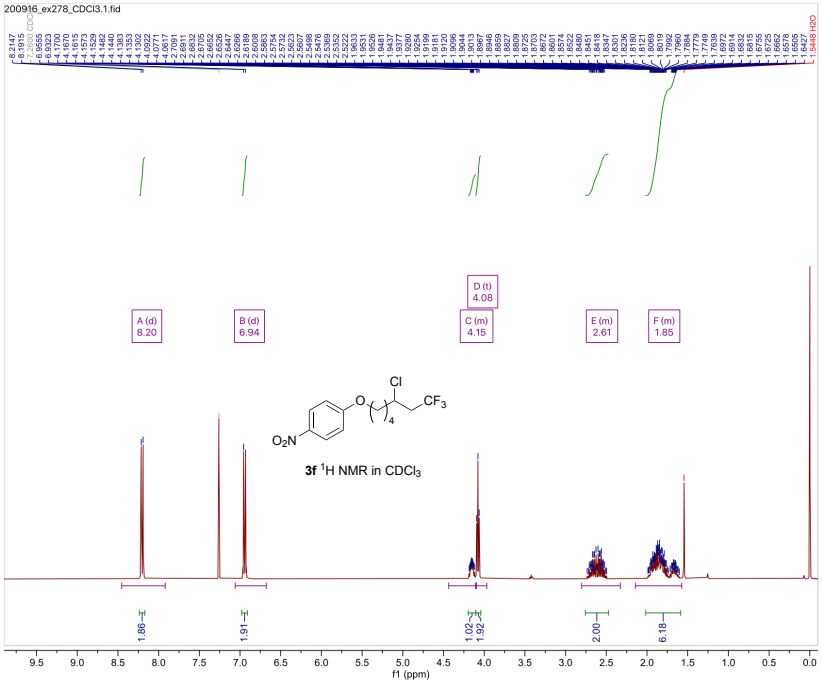
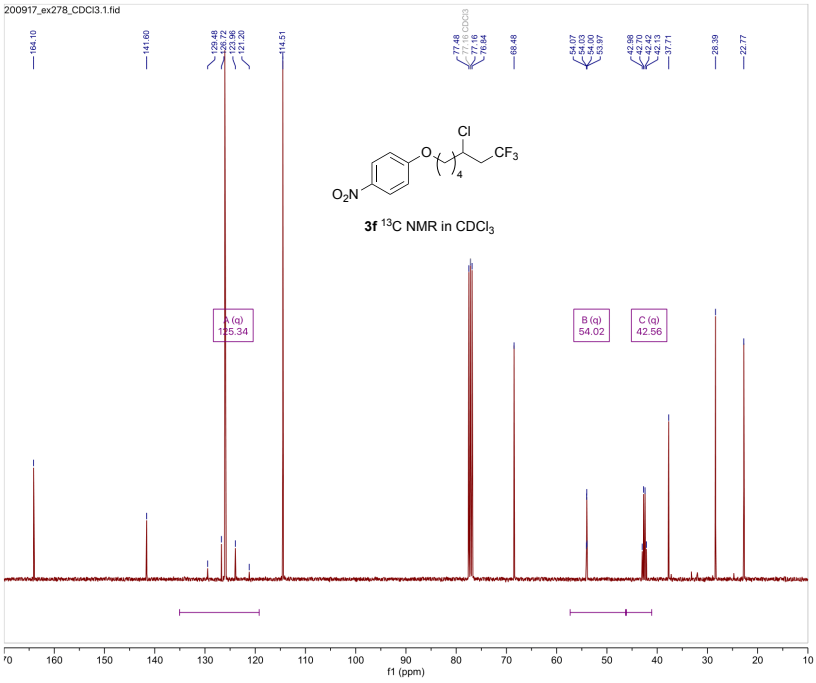
2	Title	200916_ex277_CDCl3.2.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-16T09:31:25
21	Modification Date	2020-09-16T09:3

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.67 (t, J = 10.3 Hz).



	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200916_ex278_CDCl3/ 1/ fid
2	Title	200916_ex278_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-16T09:49:28
21	Modification Date	2020-09-16T09:49:02

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 9.3 Hz, 1H), 6.94 (d, *J* = 9.3 Hz, 1H), 4.44 – 4.10 (m, 1H), 4.08 (t, *J* = 6.1 Hz, 1H), 2.80 – 2.33 (m, 1H), 2.14 – 1.57 (m, 6H).



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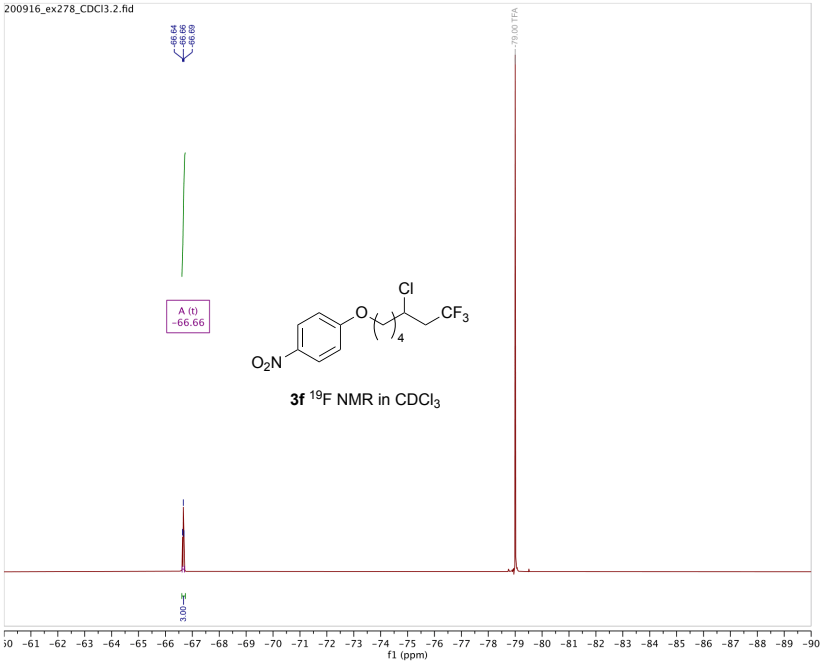
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	837
15	Receiver Gain	34.9
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-17T14:27:06

^{13}C NMR (101 MHz, Chloroform-*d*) δ 125.34 (q, *J* = 277.6 Hz), 54.02 (q, *J* = 3.3 Hz), 42.56 (q, *J* = 28.5 Hz).



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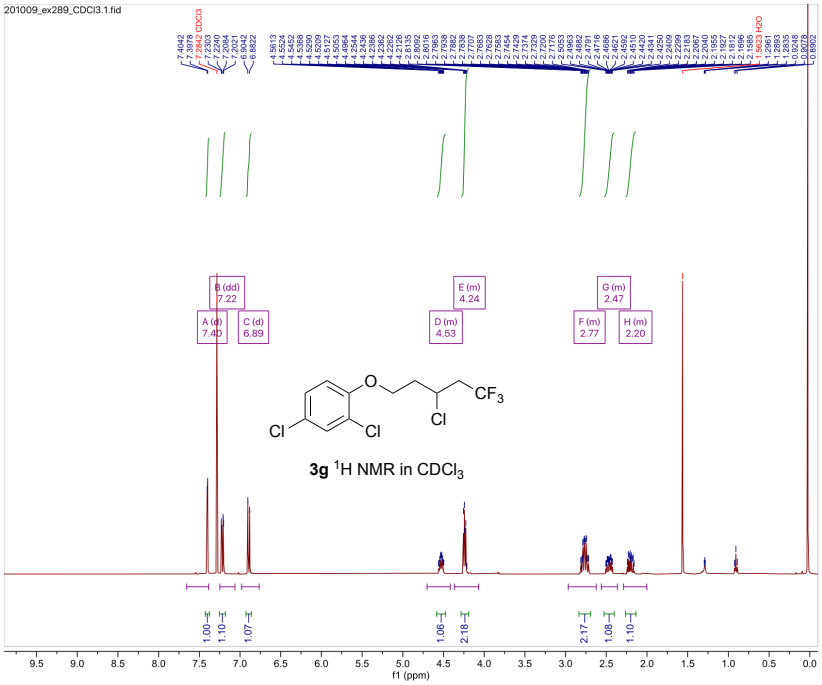
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-16T09:53:50
21	Modification Date	2020-09-16T09:5

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.66 (t, *J* = 10.1 Hz).



	Parameter	Value
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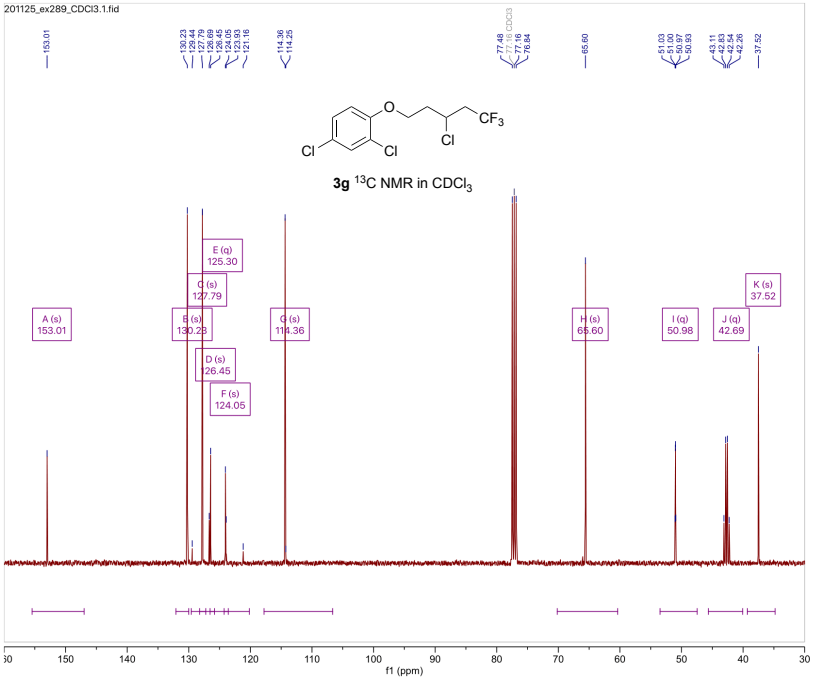
2	Title	201009_ex289_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-10-09T14:32:12
21	Modification Date	2020-10-09T14:2

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 2.5 Hz, 1H), 7.22 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 1H), 4.70 – 4.42 (m, 1H), 4.37 – 4.07 (m, 2H), 2.97 – 2.62 (m, 2H), 2.56 – 2.36 (m, 1H), 2.29 – 2.00 (m, 1H).



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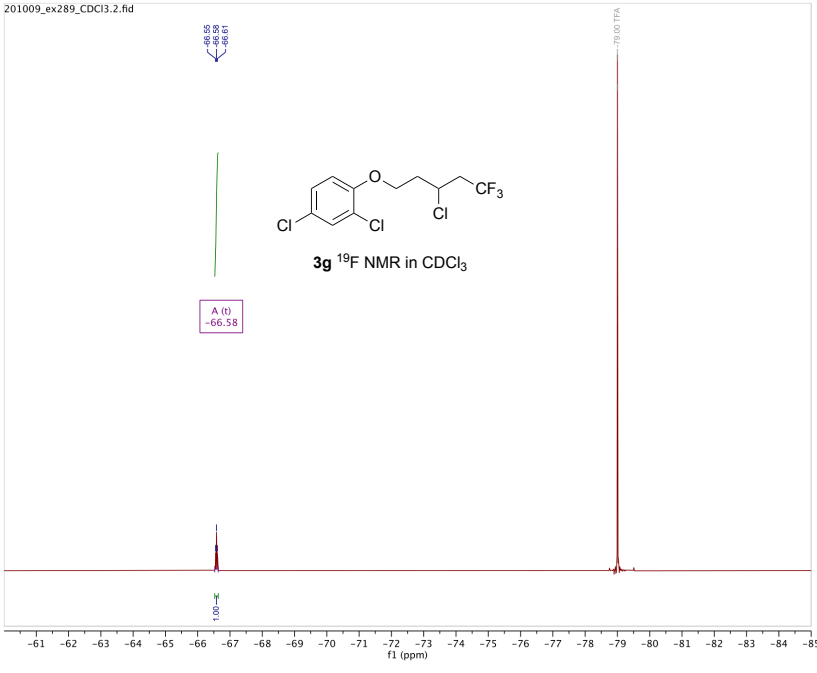
2	Title	201125_ex289_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.3
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	927
15	Receiver Gain	26.4
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-11-25T14:51:55

^{13}C NMR (101 MHz, Chloroform-*d*) δ 153.01, 130.23, 127.79, 126.45, 125.30 (q, J = 277.7 Hz), 124.05, 114.36, 65.60, 50.98 (q, J = 3.4 Hz), 42.69 (q, J = 28.7 Hz), 37.52.



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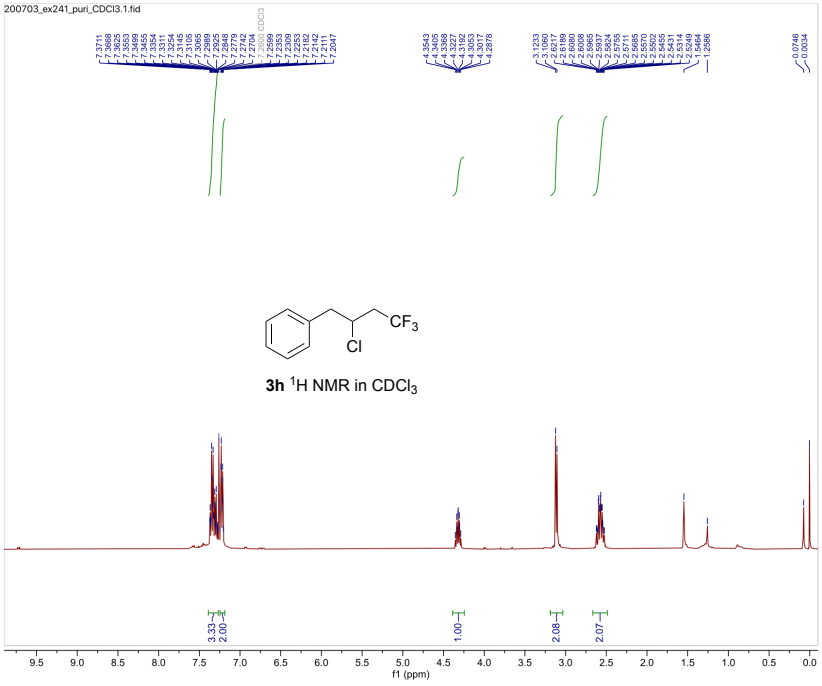
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-10-09T14:36:28
21	Modification Date	2020-10-09T14:3

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.58 (t, J = 10.2 Hz).



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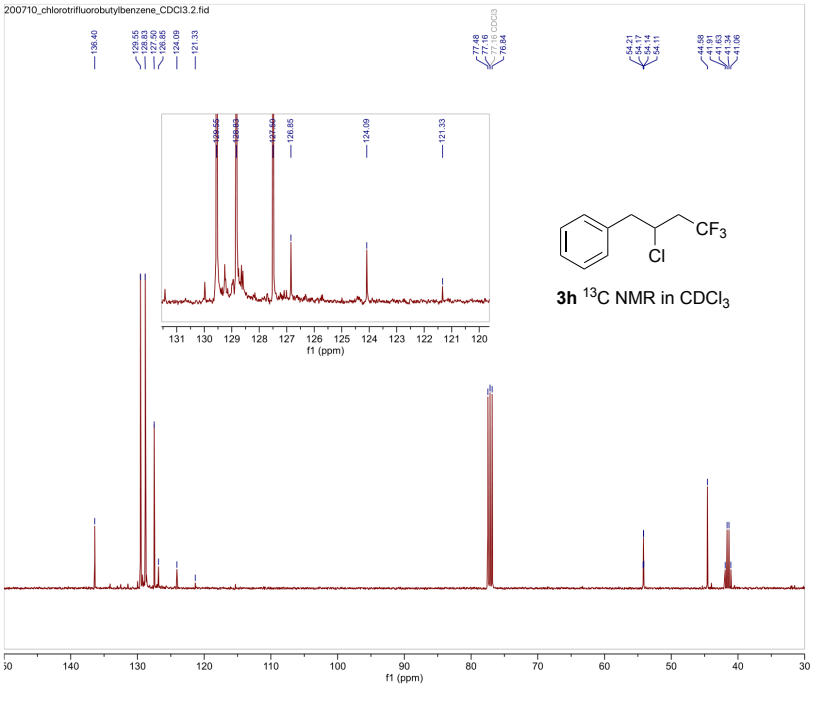
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-07-03T14:52:58
21	Modification Date	2020-07-03T14:50:06



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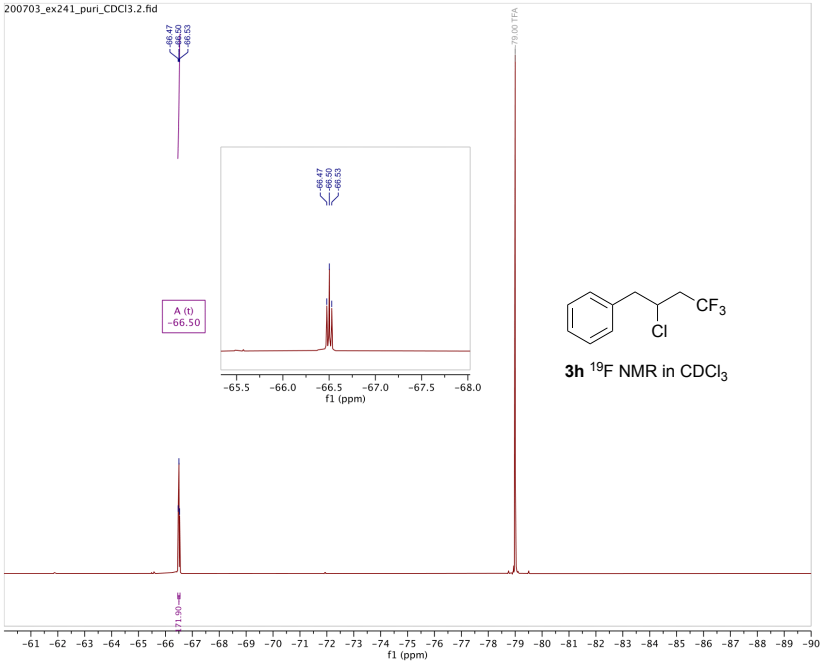
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	1024
15	Receiver Gain	32.6
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763



^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.50 (t, *J* = 10.1 Hz).

	Parameter	Value
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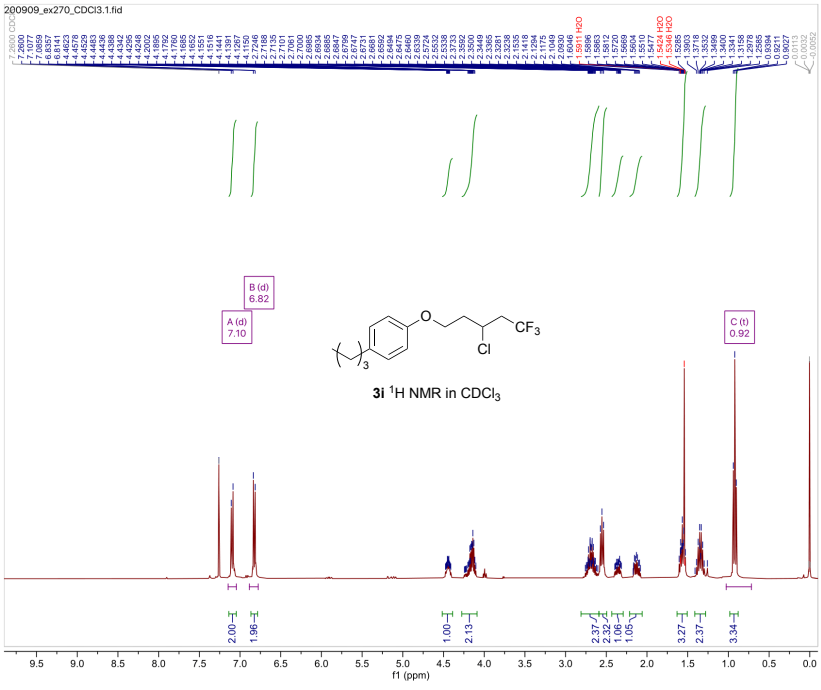
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-07-03T14:57:13
21	Modification Date	2020-07-03T14:5



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1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 200909_ex270_CDCl3/ 1/ fid
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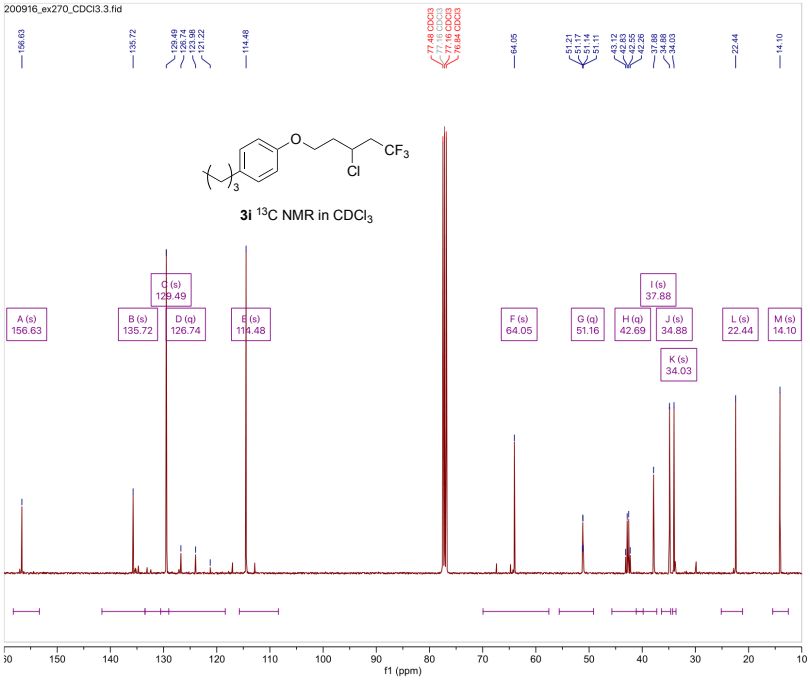
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-09T12:59:30
21	Modification Date	2020-09-09T12:53:48



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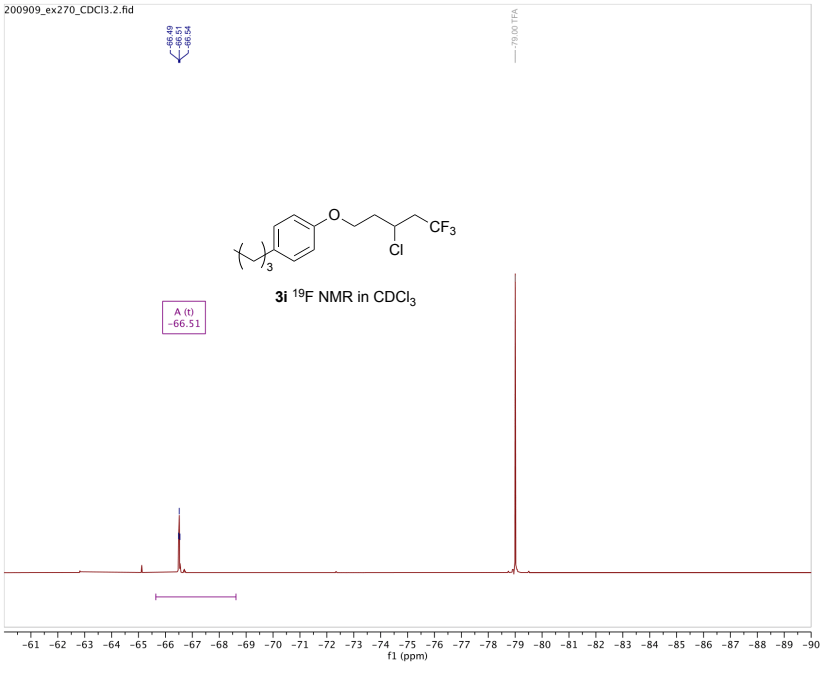
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	10000
15	Receiver Gain	34.9
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-09-17T05:39:18

^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.63, 135.72, 129.49, 126.74 (q, J = 277.7 Hz), 114.48, 64.05, 51.16 (q, J = 3.3 Hz), 42.69 (q, J = 28.6 Hz), 37.88, 34.88, 34.03, 22.44, 14.10.



	Parameter	Value
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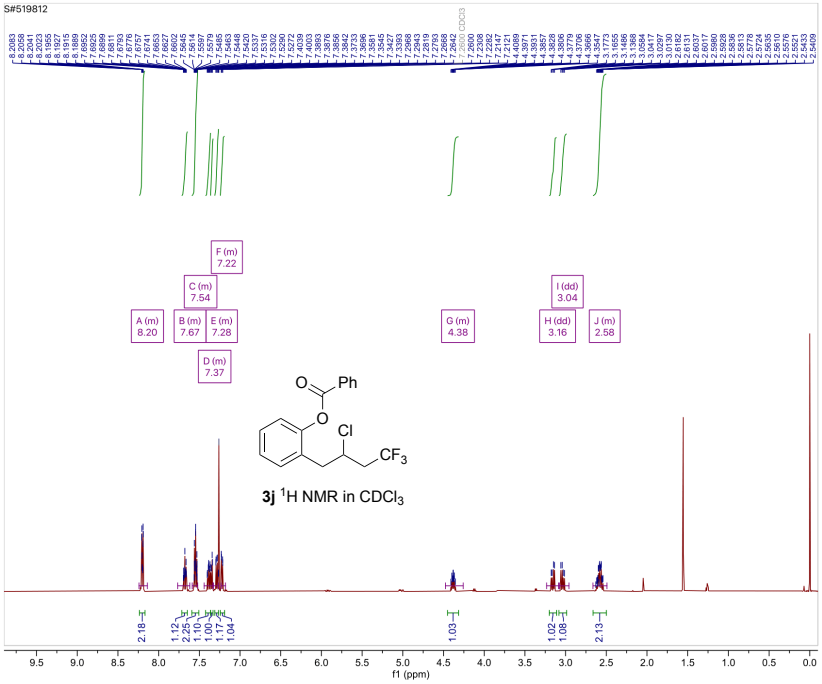
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-09T13:05:52
21	Modification Date	2020-09-09T13:0

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.51 (t, J = 10.1 Hz).



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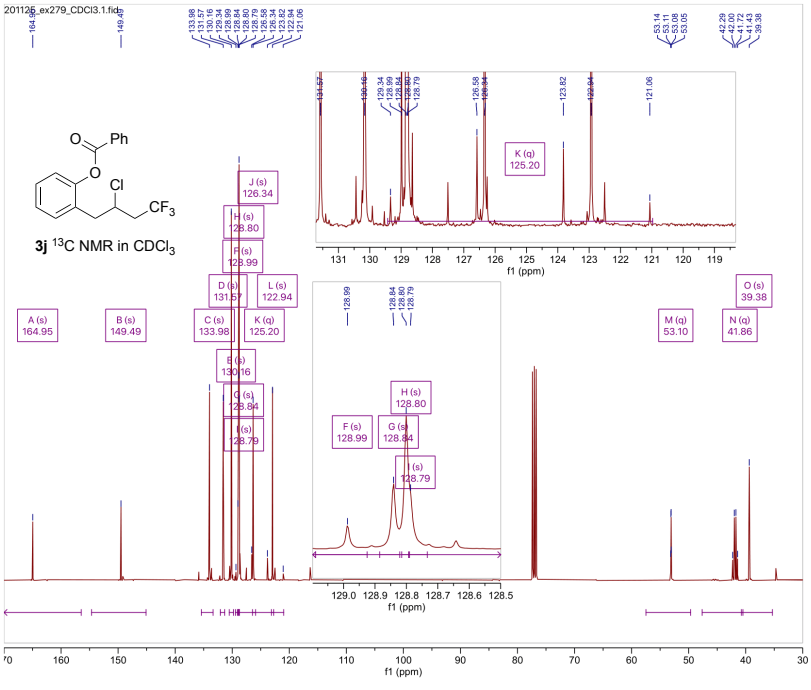
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.8
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	56.0
16	Relaxation Delay	5.0000
17	Pulse Width	5.7000
18	Presaturation Frequency	
19	Acquisition Time	1.7459
20	Acquisition Date	2020-09-18T14:08:45
21	Modification Date	2020-09-18T14:27:56
22	Class	
23	Spectrometer Frequency	500.16

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.24 – 8.14 (m, 1H), 7.77 – 7.62 (m, 1H), 7.59 – 7.52 (m, 1H), 7.44 – 7.33 (m, 1H), 7.33 – 7.27 (m, 1H), 7.23 – 7.18 (m, 1H), 4.48 – 4.26 (m, 1H), 3.16 (dd, *J* = 14.4, 5.9 Hz, 1H), 3.04 (dd, *J* = 14.3, 8.3 Hz, 1H), 2.66 – 2.49 (m, 1H).



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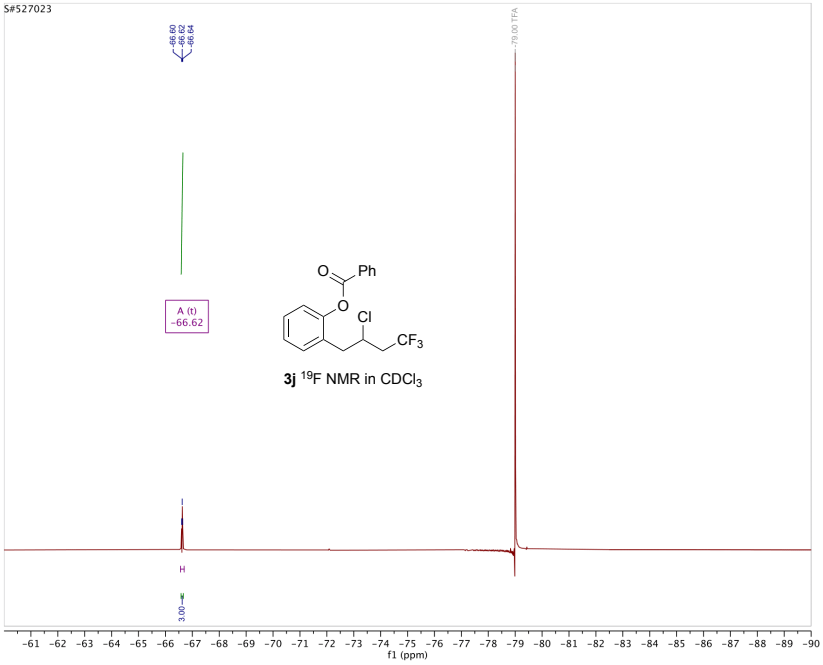
2	Title	201125_ex279_CDCl3.1.fid
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	10000
15	Receiver Gain	28.7
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-11-26T05:39:08

^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.95, 149.49, 133.98, 131.57, 130.16, 128.99, 128.84, 128.80, 128.79, 126.34, 125.20 (q, *J* = 277.6 Hz), 122.94, 53.10 (q, *J* = 3.1 Hz), 41.86 (q, *J* = 28.7 Hz), 39.38.



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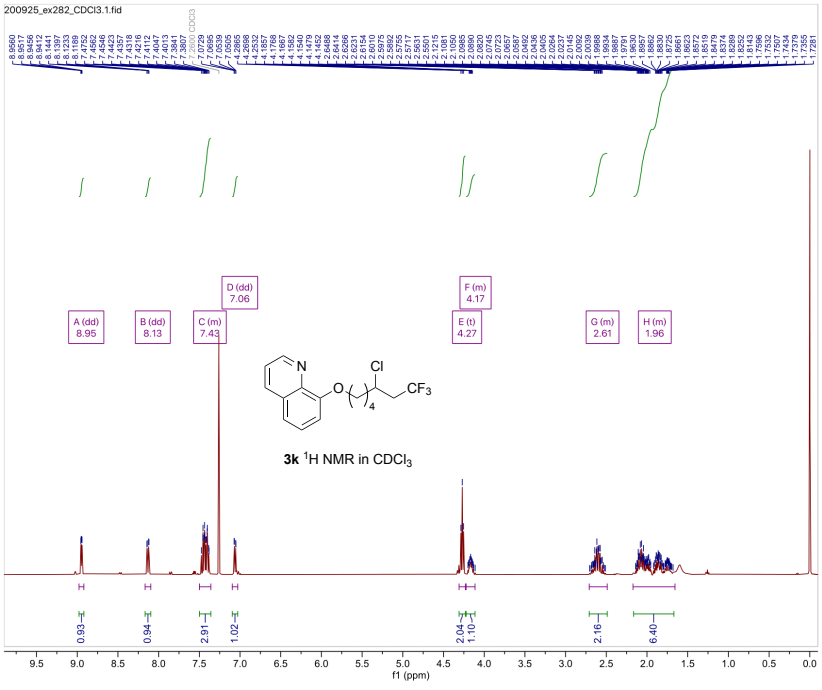
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.9
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	44.0
16	Relaxation Delay	5.0000
17	Pulse Width	6.5500
18	Presaturation Frequency	
19	Acquisition Time	0.1520
20	Acquisition Date	2020-09-18T14:21:10
21	Modification Date	2020-09-18T14:40:20
22	Class	
23	Spectrometer	470.62

^{19}F NMR (471 MHz, Chloroform-*d*) δ -66.62 (t, *J* = 9.5 Hz).



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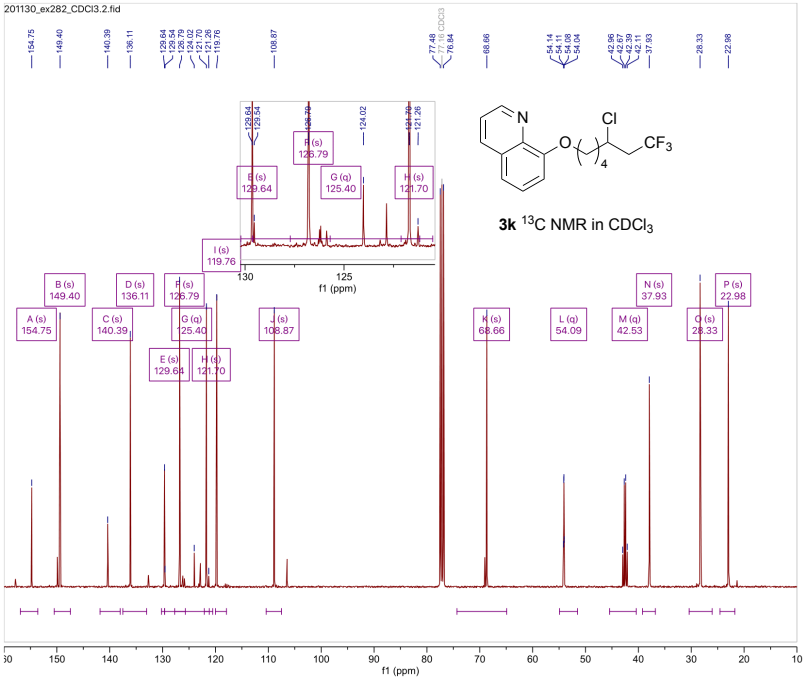
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-09-25T15:23:54
21	Modification Date	2020-09-25T15:22:36

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.95 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.13 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.50 – 7.36 (m, 3H), 7.06 (dd, *J* = 7.6, 1.4 Hz, 1H), 4.27 (t, *J* = 6.7 Hz, 2H), 4.22 – 4.11 (m, 1H), 2.71 – 2.49 (m, 2H), 2.17 – 1.66 (m, 6H).



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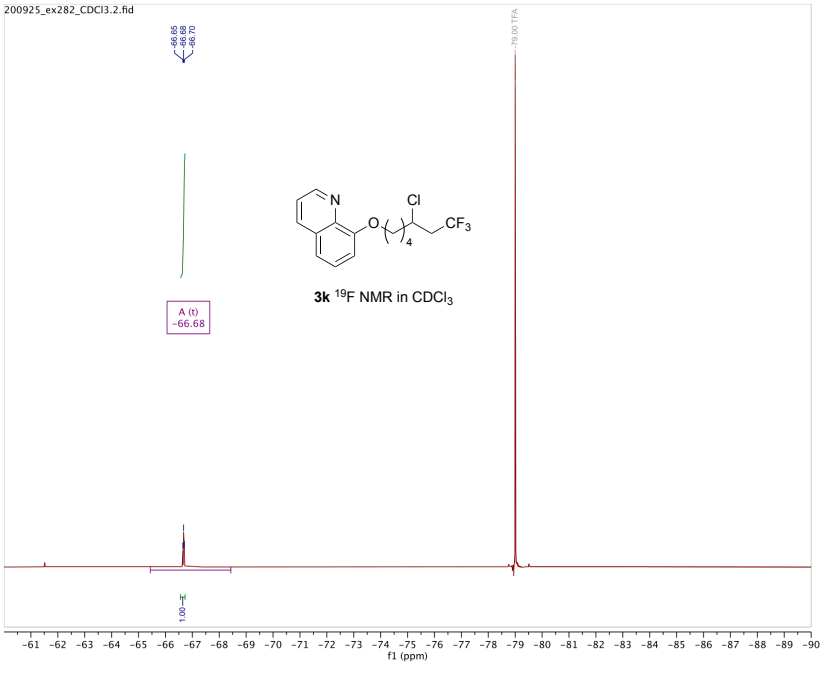
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	13585
15	Receiver Gain	27.1
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-12-01T08:48:57

^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.75, 149.40, 140.39, 136.11, 129.64, 126.79, 125.40 (q, J = 277.7Hz), 121.70, 119.76, 108.87, 68.66, 54.09 (q, J = 3.3 Hz), 42.53 (q, J = 28.4 Hz), 37.93, 28.33, 22.98.



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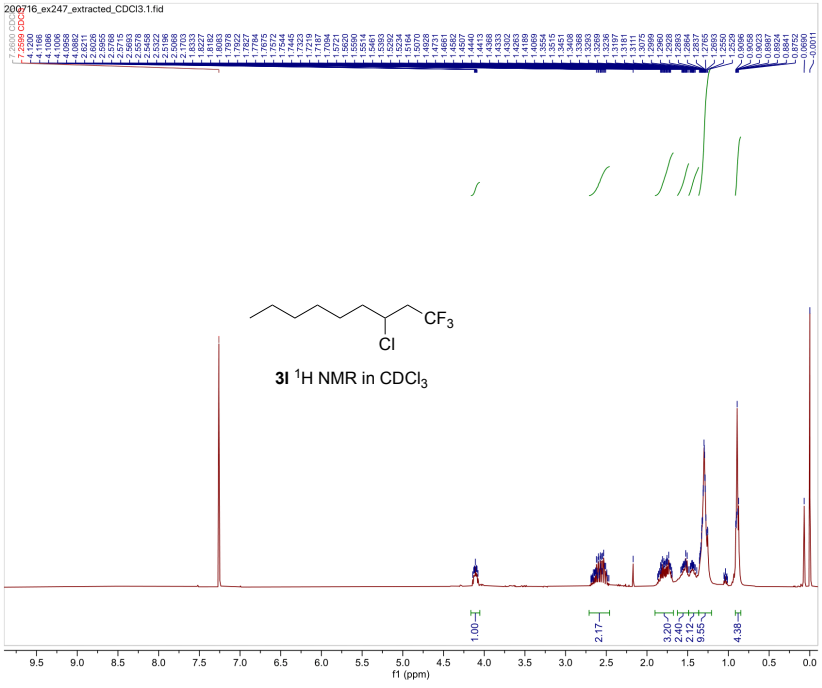
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-09-25T15:28:22
21	Modification Date	2020-09-25T15:2

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.68 (t, J = 10.4 Hz).



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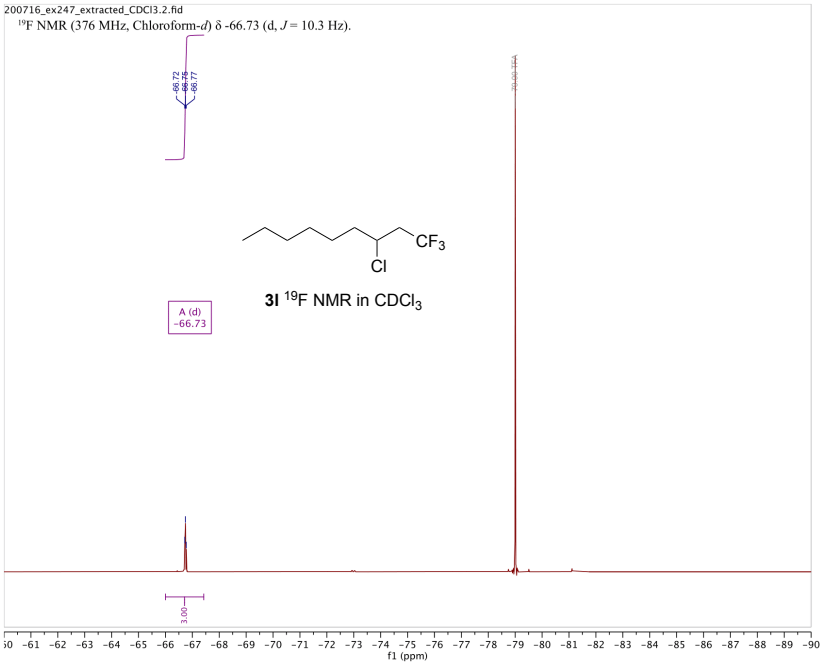
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-07-16T19:06:06



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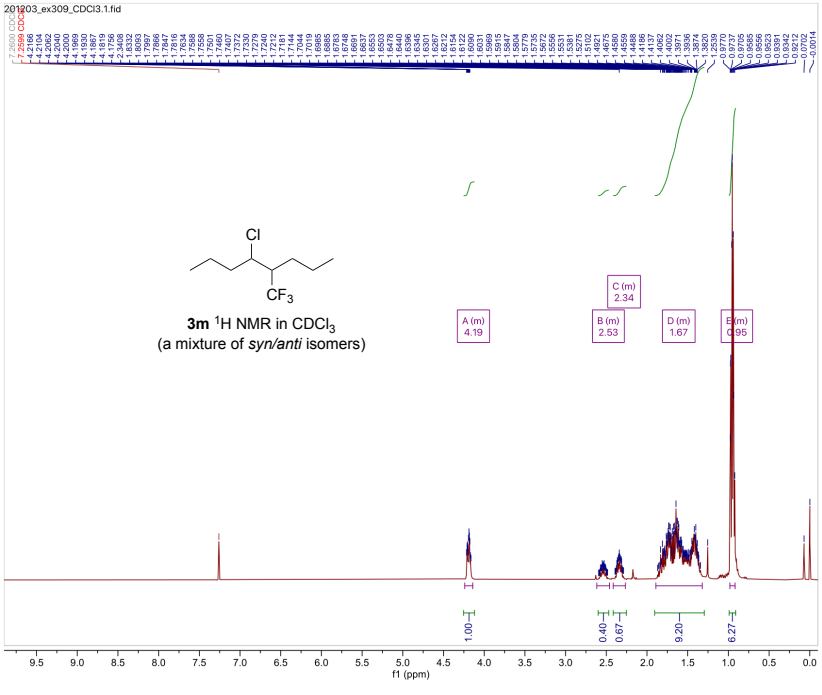
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-07-16T19:10:34



^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.24 – 4.14 (m, 1H), 2.62 – 2.46 (m, 0.40H), 2.42 – 2.27 (m, 0.67H), 1.89 – 1.32 (m, 8H), 0.98 – 0.92 (m, 6H).

	Parameter	Value
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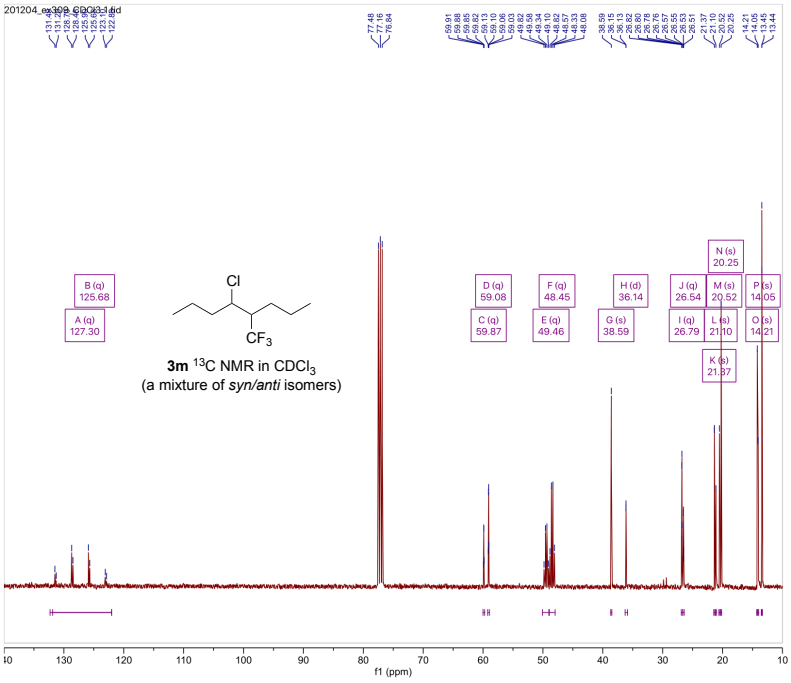
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	32.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2020-12-03T09:19:06
21	Modification Date	2020-12-03T09:16:34



^{13}C NMR (101 MHz, Chloroform-*d*) δ 127.30 (q, *J* = 281.5 Hz), 125.68 (q, *J* = 281.7 Hz), 59.87 (q, *J* = 3.0 Hz), 59.08 (q, *J* = 3.3 Hz), 49.46 (q, *J* = 24.2 Hz), 48.45 (q, *J* = 24.7 Hz), 38.59, 36.14 (d, *J* = 1.6 Hz), 26.79 (q, *J* = 1.8 Hz), 26.54 (q, *J* = 2.0 Hz), 21.37, 21.10, 20.52, 20.25, 14.21, 14.05, 13.45, 13.44.

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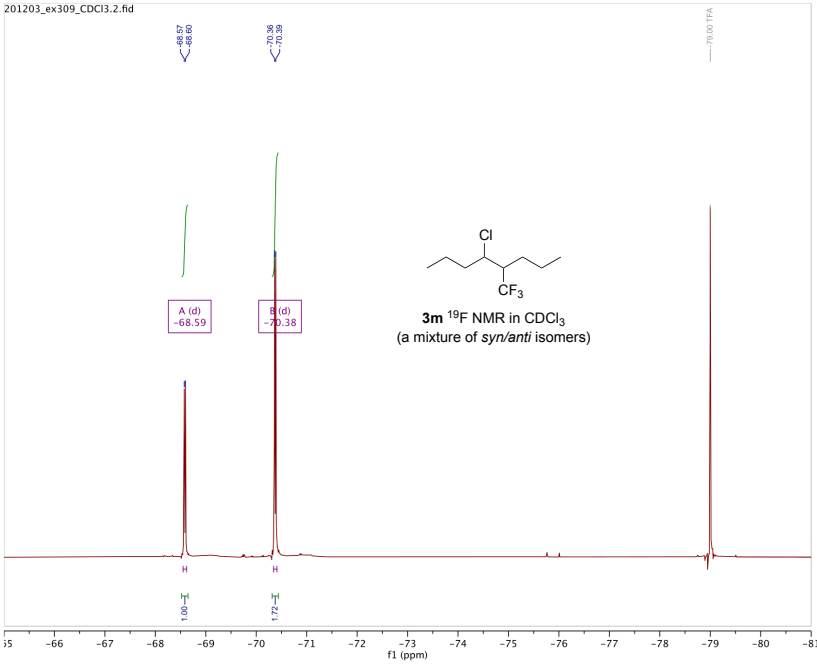
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	839
15	Receiver Gain	26.4
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-12-04T10:51:10



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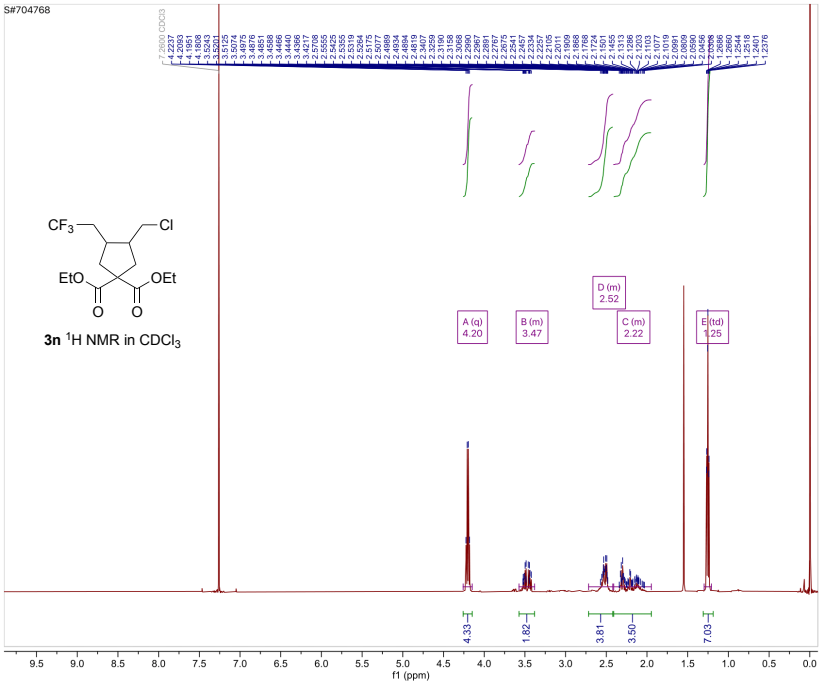
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PABBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2020-12-03T09:24:08
21	Modification Date	2020-12-03T09:2

^{19}F NMR (376 MHz, Chloroform-*d*) δ -68.59 (d, *J* = 9.6 Hz), -70.38 (d, *J* = 9.1 Hz).



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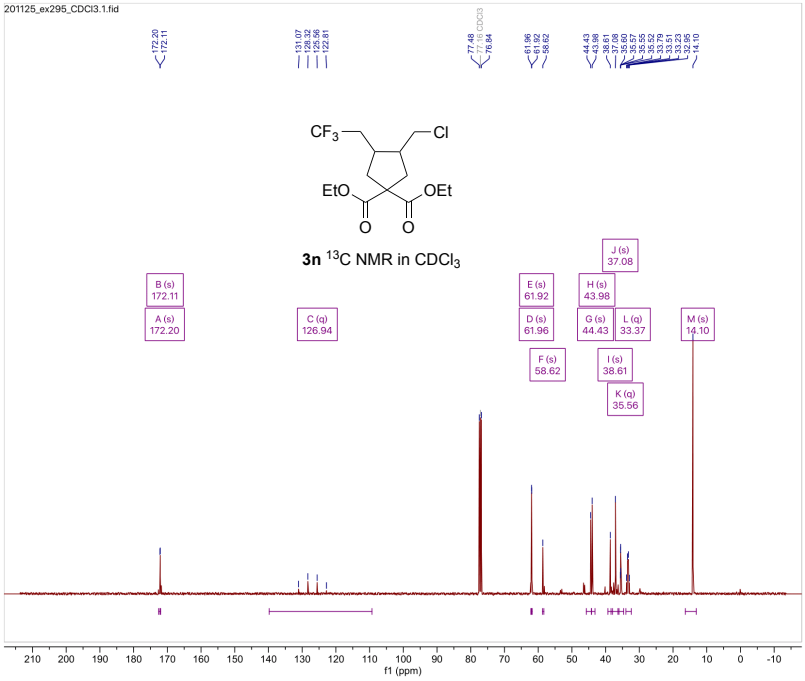
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.5
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	52.0
16	Relaxation Delay	5.0000
17	Pulse Width	5.7000
18	Presaturation Frequency	
19	Acquisition Time	1.7459
20	Acquisition Date	2020-10-22T19:11:27
21	Modification Date	2020-10-22T19:36:10
22	Class	
23	Spectrometer Frequency	500.16



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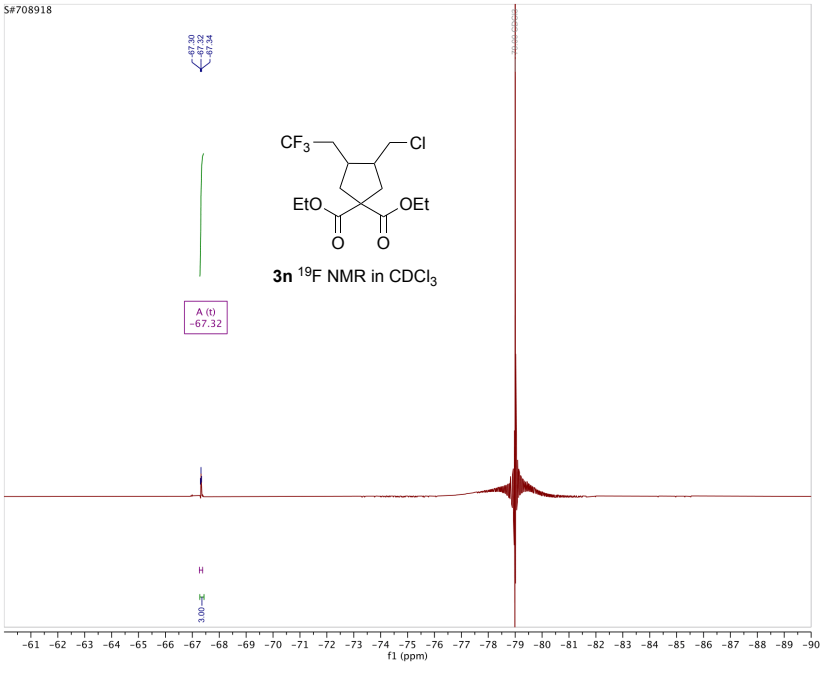
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.5
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	1024
15	Receiver Gain	27.1
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2020-11-25T20:02:48

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.20, 172.11, 126.94 (q, *J* = 277.1 Hz), 61.96, 61.92, 58.62, 44.43, 43.98, 38.61, 37.08, 35.56 (q, *J* = 2.5 Hz), 33.37 (q, *J* = 2.5 Hz), 14.10.



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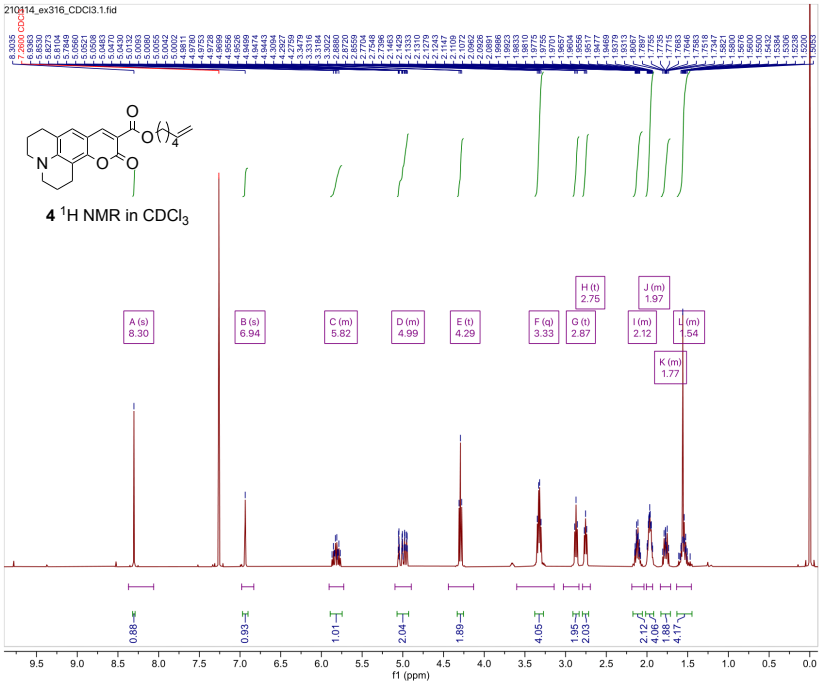
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.8
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	4
15	Receiver Gain	46.0
16	Relaxation Delay	5.0000
17	Pulse Width	6.5500
18	Presaturation Frequency	
19	Acquisition Time	0.1520
20	Acquisition Date	2020-10-22T19:18:30
21	Modification Date	2020-10-22T19:43:13
22	Class	
23	Spectrometer	470.62



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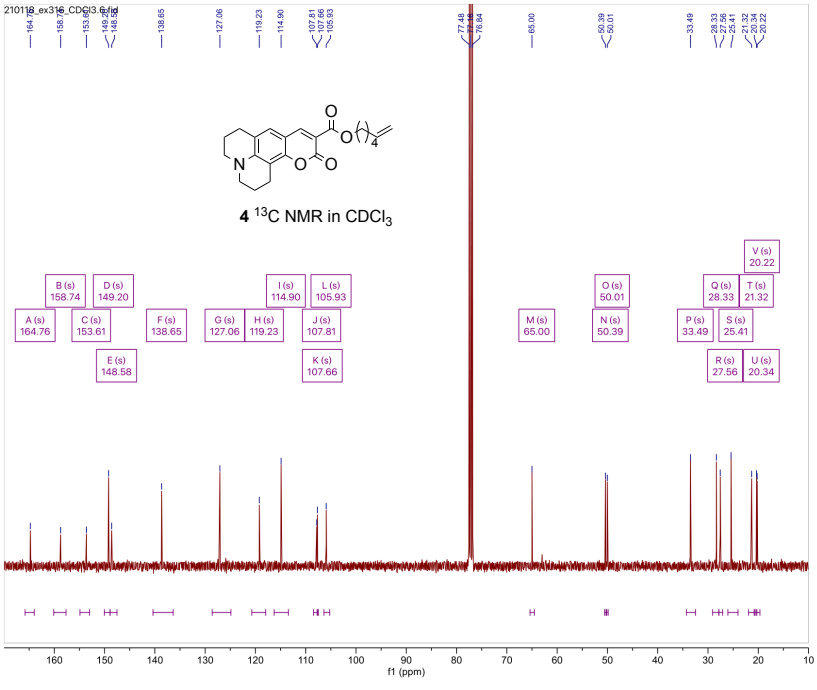
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2021-01-14T16:02:10
21	Modification Date	2021-01-14T16:01:24

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (s, 1H), 6.94 (s, 1H), 5.91 – 5.73 (m, 1H), 5.10 – 4.90 (m, 2H), 4.29 (t, *J* = 6.7 Hz, 2H), 3.33 (q, *J* = 6.5 Hz, 4H), 2.87 (t, *J* = 6.4 Hz, 2H), 2.75 (t, *J* = 6.2 Hz, 2H), 2.19 – 2.04 (m, 2H), 2.01 – 1.93 (m, 4H), 1.84 – 1.71 (m, 2H), 1.64 – 1.46 (m, 4H).



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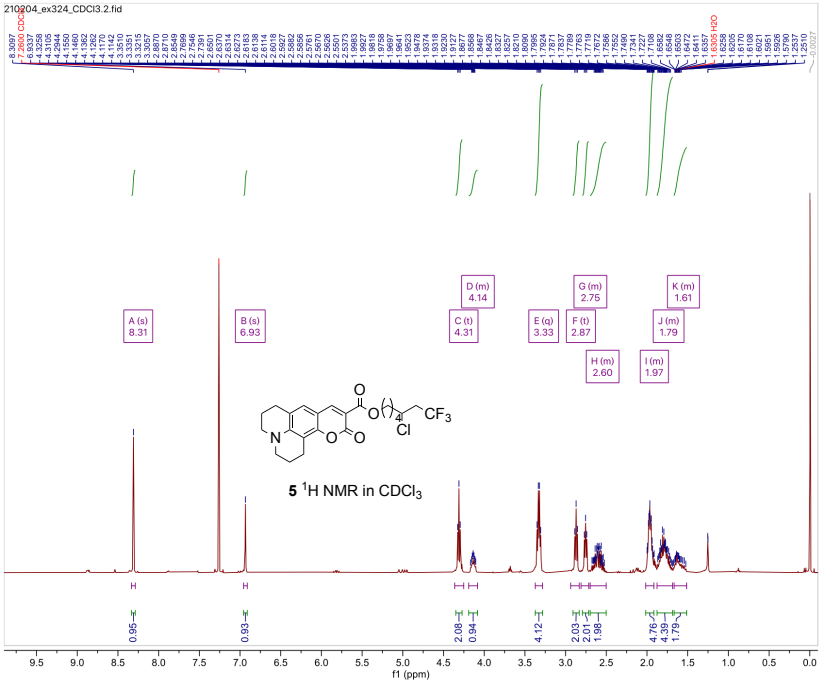
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	792
15	Receiver Gain	21.7
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2021-01-18T14:50:39

^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.76, 158.74, 153.61, 149.20, 148.58, 138.65, 127.06, 119.23, 114.90, 107.81, 107.66, 105.93, 65.00, 50.39, 50.01, 33.49, 28.33, 27.56, 25.41, 21.32, 20.34, 20.22.



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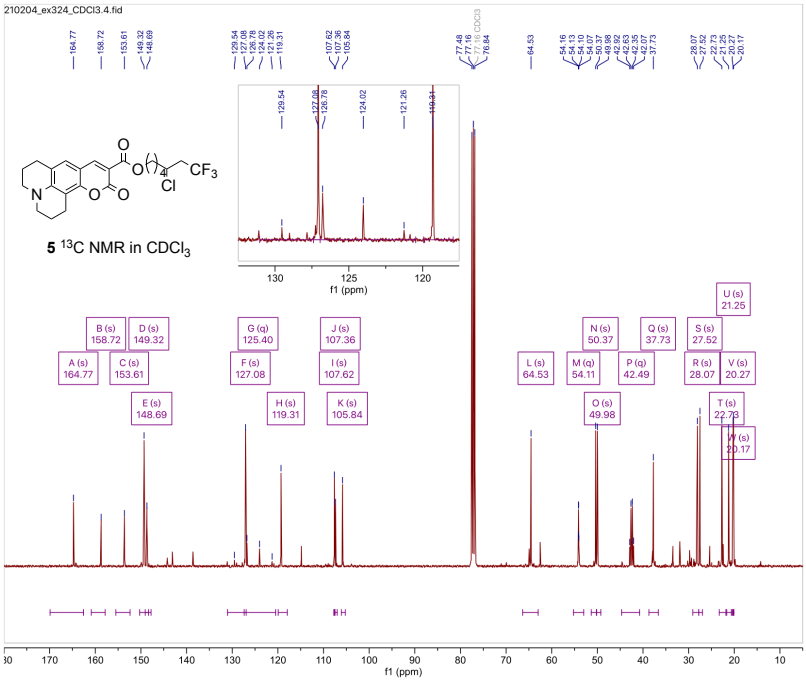
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.1
11	Pulse Sequence	zg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	3.9977
20	Acquisition Date	2021-02-04T16:44:32
21	Modification Date	2021-02-04T16:44:18

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.31 (s, 1H), 6.93 (s, 1H), 4.31 (t, *J* = 6.3 Hz, 2H), 4.19 – 4.08 (m, 1H), 3.33 (q, *J* = 6.3 Hz, 4H), 2.87 (t, *J* = 6.4 Hz, 2H), 2.81 – 2.71 (m, 2H), 2.70 – 2.50 (m, 2H), 2.02 – 1.92 (m, 4H), 1.88 – 1.69 (m, 4H), 1.67 – 1.51 (m, 2H).



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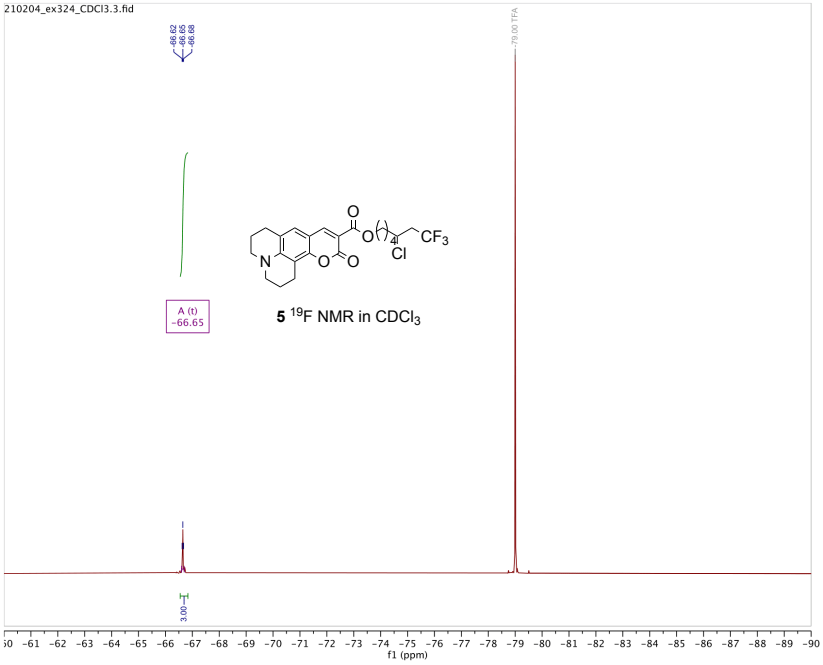
3	Comment	
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Probe	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	13054
15	Receiver Gain	23.3
16	Relaxation Delay	2.0000
17	Pulse Width	10.0000
18	Presaturation Frequency	
19	Acquisition Time	1.3763
20	Acquisition Date	2021-02-05T08:39:44

^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.77, 158.72, 153.61, 149.32, 148.69, 127.08, 125.40 (q, *J* = 277.6 Hz), 119.31, 107.62, 107.36, 105.84, 64.53, 54.11 (q, *J* = 3.2 Hz), 50.37, 49.98, 42.49 (q, *J* = 28.4 Hz), 37.73, 28.07, 27.52, 22.73, 21.25, 20.27, 20.17.



	Parameter	Value
1	Data File Name	C:/ Users/ furuyama/ Documents/ Personaldata/ IshikawaYuta/ NMR/ 生data/ 210204_ex324_CDCl3/ 3/ fid
2	Title	210204_ex324_CDCl3.3.fid
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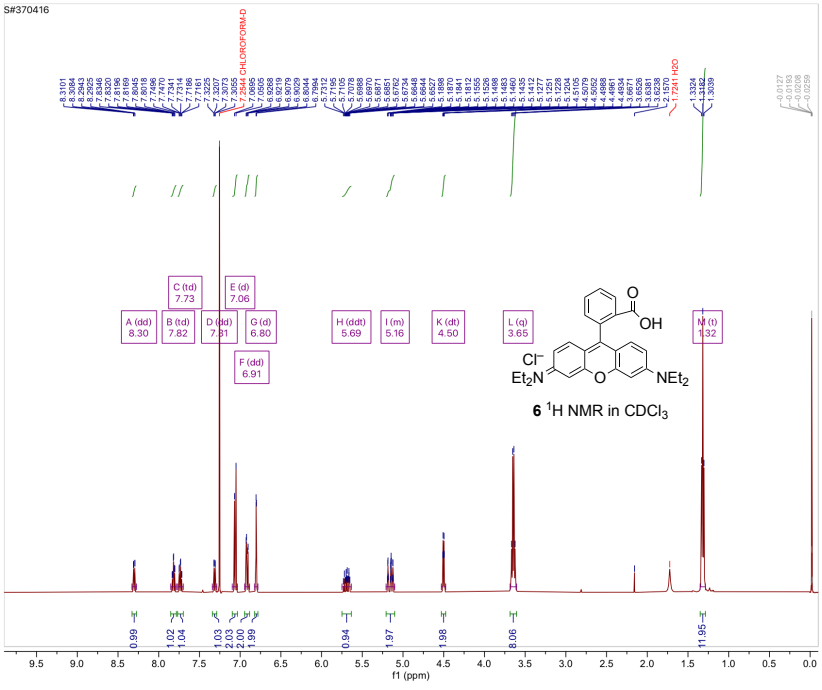
5	Owner	nmrsu
6	Site	
7	Instrument	Avance
8	Author	
9	Solvent	CDCl3
10	Temperature	298.2
11	Pulse Sequence	zg
12	Experiment	1D
13	Probe	Z116098_0842 (PABBO 400S1 BBF-H-D-05 Z SP)
14	Number of Scans	16
15	Receiver Gain	101.0
16	Relaxation Delay	1.0000
17	Pulse Width	18.0000
18	Presaturation Frequency	
19	Acquisition Time	0.7209
20	Acquisition Date	2021-02-04T16:52:53
21	Modification Date	2021-02-04T16:5

^{19}F NMR (376 MHz, Chloroform-*d*) δ -66.65 (t, *J* = 10.3 Hz).



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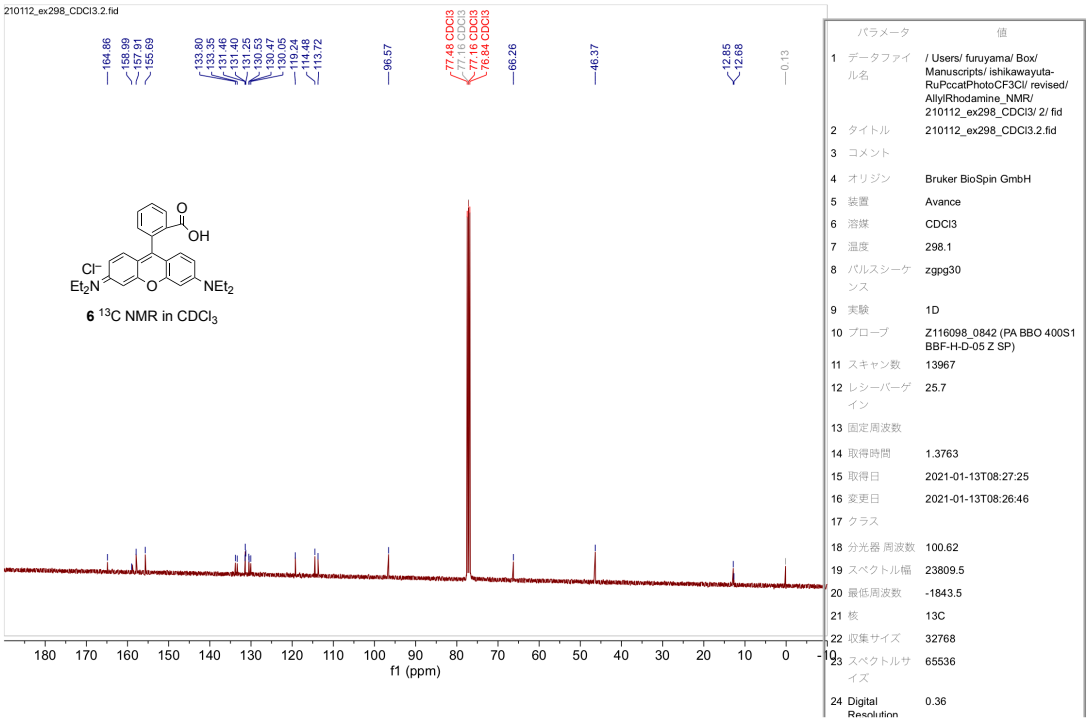
3	Comment	
4	Origin	JEOL
5	Owner	
6	Site	
7	Instrument	ECA 500
8	Author	delta
9	Solvent	CHLOROFORM-D
10	Temperature	20.2
11	Pulse Sequence	single_pulse.ex2
12	Experiment	1D
13	Probe	2756
14	Number of Scans	8
15	Receiver Gain	46.0
16	Relaxation Delay	5.0000
17	Pulse Width	5.7000
18	Presaturation Frequency	
19	Acquisition Time	1.7459
20	Acquisition Date	2020-11-02T09:54:13
21	Modification Date	2020-11-02T10:18:55
22	Class	
23	Spectrometer Frequency	500.16

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.82 (td, *J* = 7.5, 1.3 Hz, 1H), 7.73 (td, *J* = 7.7, 1.3 Hz, 1H), 7.31 (dd, *J* = 7.6, 0.9 Hz, 1H), 7.06 (d, *J* = 9.5 Hz, 2H), 6.91 (dd, *J* = 9.5, 2.5 Hz, 2H), 6.80 (d, *J* = 2.5 Hz, 2H), 5.69 (ddt, *J* = 17.2, 10.4, 5.9 Hz, 1H), 5.21 – 5.10 (m, 2H), 4.50 (dt, *J* = 5.9, 1.3 Hz, 2H), 3.65 (q, *J* = 7.2 Hz, 8H), 1.32 (t, *J* = 7.1 Hz, 12H).



	パラメータ	値
1	データファイル名	/ Users/ furuyama/ Box/ Manuscripts/ ishikawayuta-RuPccatPhotoCF3Cl/ revised/ AllylRhodamine_NMR/ 210112_ex298_CDCl3/ 2/ fid
2	タイトル	210112_ex298_CDCl3.2.fid
3	コメント	
4	オリジン	Bruker BioSpin GmbH
5	装置	Avance
6	溶媒	CDCl3
7	温度	298.1
8	パルスシーケンス	zgpg30
9	実験	1D
10	プローブ	Z116098_0842 (PA BBO 400S1 BBF-H-D-05 Z SP)
11	スキャン数	13967
12	レシーバーゲイン	25.7
13	固定周波数	
14	取得時間	1.3763
15	取得日	2021-01-13T08:27:25
16	変更日	2021-01-13T08:26:46
17	クラス	
18	分光器 周波数	100.62
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20	最低周波数	-1843.5
21	核	13C
22	収集サイズ	32768
23	スペクトルサイズ	65536
24	Digital Resolution	0.36

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