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Fluorinated [2]Rotaxanes with Spirofluorene Motif: Non-symmetric Distribution of the Ring Component along the Axle Component

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Table S1. ^{1}H and ^{19}F NMR chemical shifts in rotaxanes (**10Aa-Ad**) with variable axle lengths.

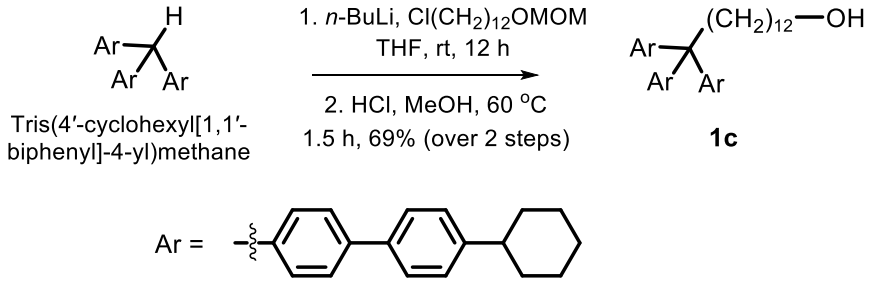
Entry	Length of alkylene group (n)	Rotaxane	δF		ΔδF[a]	δH[i], δH[j]		ΔδH[b]
1	3	**10Aa**	-127.12	-127.77	0.66	4.08	3.86	0.22
2	6	**10Ab**	-127.07	-127.84	0.77	4.01	3.79	0.22
3	12	**10Ac**	-127.12	-127.87	0.75	4.03	3.83	0.20
4	20	**10Ad**	-127.11	-127.85	0.74	4.05	3.85	0.20

[a] ΔδF = δF (low field) - δF (high field). [b] ΔδH = δH (low field) - δH (high field).

General Information:

All moisture and air-sensitive reactions were performed using standard syringe-septum technique under argon atmosphere. Low temperatures were maintained using dry ice-acetone combination. Oil bath was used as the heating source and the external temperature was reported. Commercially available reagents were used without further purification. All reactions were monitored by thin-layer chromatography (TLC, on Merck silica gel 60F-254 plates) and visualization of the spots was done under UV light or by dipping the plates in polymolybidic acid-ethanol or anisaldehyde-acid reagent and heating at ~120 °C. Column chromatography was performed using silica gel 60N (spherical, neutral 40−50 μm) from Kanto Chemicals. NMR spectra were recorded on a JEOL 300, 400 or 500 MHz spectrometer or a Bruker 400 MHz NMR spectrometer. Chemical shifts were reported in delta units (δ) relative to chloroform (7.24 ppm for ^{1}H NMR and 77.0 ppm for ^{13}C NMR) and dimethyl sulfoxide (DMSO, 2.50 ppm for ^{1}H NMR and 39.5 ppm for ^{13}C NMR) as internal reference standards or $BF_3{\cdot}Et_2O$ (0.00 ppm for ^{19}F) as external reference standard. Multiplicity is indicated by s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), or bs (broad singlet). Coupling constants (J) are reported in Hertz. IR spectra were recorded on a Fourier transform infrared spectrometer using a diamond ATR module. A YMC-GPC T30000 (21.2 mm ID × 600 mm L) column was used for GPC separation using $CHCl_3$ as the eluent. High-resolution mass spectra (HRMS) were obtained by using matrix-assisted laser desorption/ionization (MALDI) and a time-of-flight (TOF) mass analyzer.

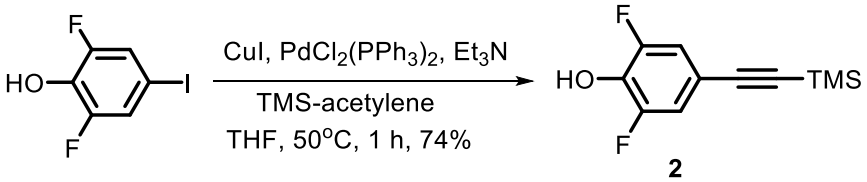
Synthesis of compound 1c:



n-BuLi (1.56 M solution in hexane, 1.78 mL, 2.78 mmol, 2 equiv) was added dropwise to the solution of tris(4-cyclohexylbiphenyl)methane[1] (1.00 g, 1.39 mmol, 1 equiv) in 15 mL of dry THF with stirring at room temperature. The color of the solution turned to dark blue. 1-Chloro-6-(methoxymethoxy)dodecane[2] (0.37 g, 1.39 mmol, 1 equiv) was added at rt, and the mixture was stirred for 12 h. MeOH (20 mL) and conc. HCl (7 mL) were added, and the mixture was heated at 60 °C for 1.5 h. Water was added, and extraction was done with CH_2Cl_2 (30 mL x 3). The combined organic layer was dried over $MgSO_4$, and the solvent was evaporated. The residue was purified by flash silica gel column chromatography using hexane:EtOAc (4:1 v/v) as eluent which resulted in compound **1c** as a colorless amorphous solid (0.82 g, 0.90 mmol, 65%):

Compound 1c: colourless amorphous solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (t, J = 8.0 Hz, 12H), 7.37 (d, J = 8.2 Hz, 6H), 7.26 (d, J = 8.2 Hz, 6H), 3.61 (t, J = 6.6 Hz, 2H), 2.62 (m, 2H), 2.56-2.50 (m, 3H), 1.88 (m, 12H), 1.75 (d, J = 12.8 Hz, 3H), 1.53 (m, 4H), 1.43 (m, 10H), 1.35-1.17 (m, 21H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 146.4, 138.4, 138.2, 129.6, 127.1, 126.8, 126.2, 63.1, 56.0, 44.2, 40.5, 34.4, 32.8, 30.5, 29.6, 29.6, 29.5, 29.5, 29.4, 26.9, 26.2, 25.7; Anal. Calcd for $C_{49}H_{52}O$: C, 89.59; H, 7.98. Found: C, 89.33; H, 8.03; IR(ATR): 3367, 3026, 2925, 2851, 2324, 1906, 1496, 1447 cm^{-1}.

Synthesis of compound 2:

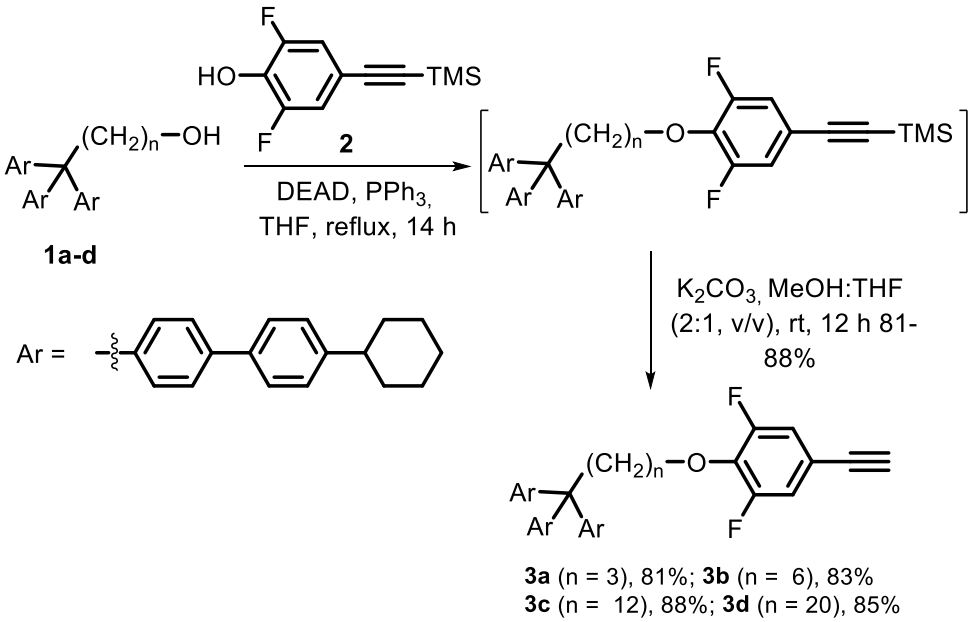


A mixture of 4-iodo-2,6-difluorophenol (2.30 g, 8.99 mmol, 1 equiv), CuI (0.086 g, 0.45 mmol, 0.05 equiv), $PdCl_2(PPh_3)_4$ (0.158 g, 0.225 mmol, 0.025 equiv) in anhydrous triethylamine (5 mL) and anhydrous THF (25 mL) was stirred at room temperature. After stirring for 10 minutes, trimethylsilylacetylene (1.55 mL, 10.8 mmol, 1.2 equiv) in anhydrous THF (5 mL) was added dropwise to the reaction mixture over 10 min and the resulting reaction mixture was heated to 50 °C. After stirring for 1 h, the mixture was

cooled to 0 °C and then 100 mL of hexane was added. Stirring was continued for 15 min after which the insoluble material was removed by filtration through a pad of celite and the residue was washed with hexane (3 × 50 mL). The filtrate was concentrated under vacuum and the crude product was purified by flash silica gel chromatography (EtOAc:Hexanes, 6:1 v/v) to afford the alkyne **2** (1.50 g, 6.6 mmol, 74%) as dark brown oil.

Compound 2: dark brown oil, ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.78 (s, 1H), 7.17 (dd, J = 7.4, 1.5 Hz, 2H), 0.21 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.0 (dd, J = 242.0, 7.1 Hz), 133.8 (t, J = 14.5 Hz), 115.5 (m), 114.6 (t, J = 10.8 Hz), 102.5, 94.6, -0.2; ^{19}F NMR (377 MHz, $CDCl_3$) δ -135.1; HRMS calcd. for $C_{11}H_{11}F_2OSi$ [M-H]$^-$: 225.0542, Found: 225.0580; IR(ATR): 3419, 2961, 2428, 2162, 1600, 1521, 1433, 1251 cm^{-1}.

General procedure for the synthesis of Axle precursors 3a-d:



To a solution of alcohol (**1a-d**, 1 equiv, **1a**,[3] **1b**,[4] and **1d**[5] were synthesized by the reported procedure), TMS-alkyne **2** (1.1 equiv) and PPh_3 (1.3 equiv) in dry THF (20 mL/mmol), was added DEAD (1.3 equiv). The mixture was stirred at room temperature for 16 h and after completion of reaction (as confirmed from TLC), water was added to the reaction mixture. Extraction was done with CH_2Cl_2 (×3), and the combined organic layer was washed with water, brine**,** dried over $MgSO_4$ and concentrated under reduced pressure. The crude products were directly used for next reaction without further purification. To a mixture of crude protected alkyne (1 equiv) in MeOH:THF (2:1, 20 mL/mmol), K_2CO_3 (5.0 equiv) was added and the mixture was stirred at room temperature for 14 h. After completion of reaction, the solvent was removed under vacuum and the residue was extracted with CH_2Cl_2 (× 3). The combined organic layer was washed with water and brine, dried over $MgSO_4$, and evaporated to dryness under reduced pressure. The crude product was

purified by flash silica gel chromatography (CH_2Cl_2:Hexanes, 3:7 v/v) to afford the terminal alkynes **3a-d** (81-88%).

Compound 3a: 0.800 g (1.03 mmol) of **1a** taken and 0.760 g (0.833 mmol) of **3a** obtained in 81% yield. Colorless needles, mp 86.4−87.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.49 (m, 12H), 7.40 (d, J = 7.6 Hz, 6H), 7.25 (d, J = 6.4 Hz, 6H), 7.01 (m, 2H), 4.16 (bs, 2H), 3.05 (s, 1H), 2.90-2.83 (bs, 2H), 2.56-2.46 (m, 3H), 1.87 (dd, J = 21.2, 9.2 Hz, 12H), 1.75 (d, J = 12.4 Hz, 3H), 1.63 (bs, 2H), 1.49-1.34 (m, 12H), 1.30-1.24 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.2 (dd, J = 247.8, 6.7 Hz), 147.1, 145.9, 138.6, 138.2, 137.1 (t, J = 14.4, 13.5 Hz), 129.5, 127.2, 126.8, 126.4, 116.2 (m), 81.3, 78.1, 75.1, 55.6, 44.2, 36.4, 34.4, 26.9, 26.6, 26.2; ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.529; HRMS calcd. for $C_{66}H_{70}F_2NO$ $[M+NH_4]^+$: 930.5420, Found: 930.5420; IR(ATR): 3295, 2923, 2849, 2120, 1567, 1509, 1341 cm^{-1}.

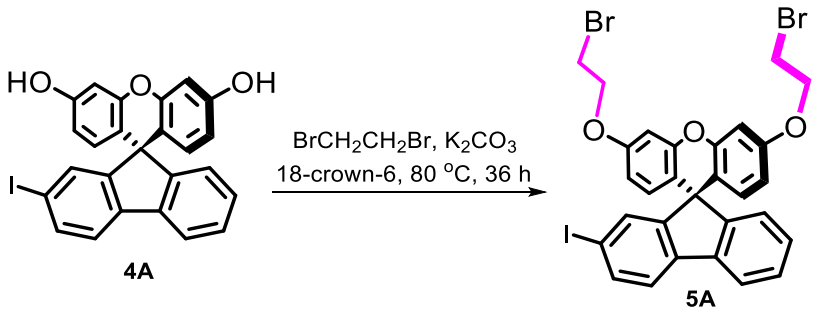
Compound 3b: 0.800 g (0.977 mmol) of **1b** taken and 0.775 g (0.812 mmol) of **3b** obtained in 83% yield. Colorless needles, mp 78.5−79.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.47 (m, 12H), 7.41-7.33 (m, 6H), 7.27 (d, J = 6.8 Hz, 6H), 7.03-6.96 (m, 2H), 4.11 (t, J = 6.2 Hz, 2H), 3.05 (s, 1H), 2.65 (m, 2H), 2.57-2.52 (m, 3H), 1.90 (dd, J = 22.2, 9.6 Hz, 12H), 1.80-1.66 (m, 5H), 1.52-1.35 (m, 15H), 1.32-1.17 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4 (dd, J = 248.8, 6.7 Hz), 147.0, 146.3, 138.4, 138.2, 137.0 (t, J = 13.5, 14.4 Hz), 129.5, 127.2, 126.8, 126.3, 116.2 (m), 81.3, 78.1, 74.7, 56.0, 44.2, 40.4, 34.4, 30.0, 29.9, 26.9, 26.2, 25.6, 25.4 (one signal is merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.574**;** HRMS calcd. for $C_{69}H_{76}F_2NO$ $[M+NH_4]^+$: 972.5889, Found: 972.5890**;** IR(ATR): 3295, 2924, 2850, 2363, 2116, 1571, 1509, 1496 cm^{-1}.

Compound 3c: 3.67 g (4.07 mmol) of **1c** taken and 3.72 g (3.58 mmol) of **3c** obtained in 88% yield. Colorless needles, mp 54.9−55.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (t, J = 8.0 Hz, 12H), 7.35 (d, J = 8.4 Hz, 6H), 7.26-7.22 (m, 6H), 7.02-6.96 (m, 2H), 4.11 (t, J = 6.8 Hz, 2H), 3.03 (s, 1H), 2.63-2.58 (m, 2H), 2.55-2.47 (m, 3H), 1.92-1.80 (m, 12H), 1.78-1.67 (m, 6H), 1.50-1.36 (m, 12H), 1.35-1.10 (m, 20H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4 (dd, J = 247.9, 6.8 Hz), 147.0, 146.4, 138.4, 138.2, 137.0 (t, J = 13.5, 14.4 Hz), 129.6, 127.1, 126.8, 126.2, 116.2 (m), 81.3 (t, J = 3.8, 2.9 Hz), 78.0, 74.8, 56.0, 44.2, 40.5, 34.4, 30.5, 29.9, 29.6, 29.55, 29.49, 29.2, 26.9, 26.2, 25.7, 25.5 (three signals are missing);^{19}F NMR (377 MHz, $CDCl_3$) δ -127.614; HRMS calcd. for $C_{75}H_{88}F_2NO$ $[M+NH_4]^+$: 1056.6828, Found: 1056.6826; IR(ATR): 3291, 2924, 2850, 2359, 2324, 1509 cm^{-1}.

Compound 3d: 1.00 g (0.985 mmol) of **1d** taken and 0.963 g (0.837 mmol) of **3d** obtained in 85% yield. Colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (t, J = 8.0 Hz, 12H), 7.36 (d, J = 8.4 Hz, 6H), 7.25 (d, J = 8.4 Hz, 6H), 7.00 (m, 2H), 4.14 (t, J = 6.8 Hz, 2H), 3.03 (s, 1H), 2.65-2.59 (m, 2H), 2.56-2.48 (m, 3H), 1.87 (dd, J = 22.0, 9.6 Hz, 12H), 1.78-1.70 (m, 5H), 1.50-1.36 (m, 14H), 1.35-1.12 (m, 35H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5 (dd,

J = 247.9, 6.7 Hz), 147.0, 146.4, 138.4, 138.2, 137.0 (t, J = 13.4 Hz), 129.6, 127.2, 126.8, 126.2, 116.2 (m), 81.3, 78.0, 74.9, 56.0, 44.2, 40.5, 34.4, 30.5, 29.9, 29.7, 29.65, 29.56, 29.5, 29.3, 26.9, 26.2, 25.7, 25.6 (some signals got merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.592; HRMS calcd. for $C_{83}H_{104}F_2NO$ $[M+NH_4]^+$: 1168.8080, Found 1168.8081; IR(ATR): 3304, 2921, 2849, 2362, 1906, 1568, 1509, 1495 cm^{-1}.

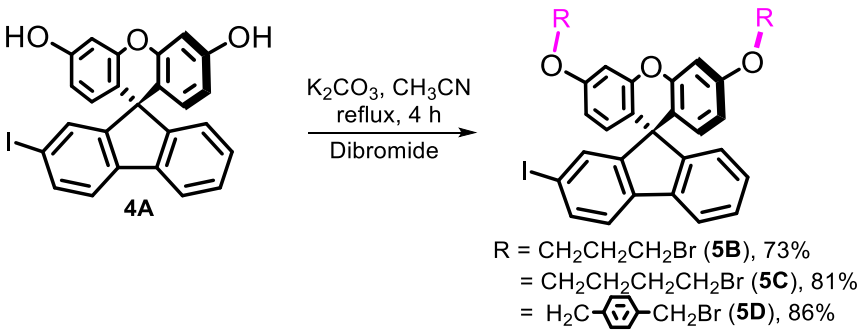
Procedure for the synthesis of 5A:



The procedure reported in the literature[6] was generally followed. A suspension of spirofluorenediol **4A**[7] (0.900 g, 1.84 mmol, 1 equiv), finely powdered K_2CO_3 (0.635 g, 4.59 mmol, 2.5 equiv) and 18-crown-6 (0.049 g, 0.184 mmol, 0.1 equiv) in 1, 2-dibromoethane (20 mL) was stirred at 80 °C for 36 h. After completion of reaction, the mixture was cooled, filtered, and washed with CH_2Cl_2. The filtrate was evaporated under vacuum to yield a residue that was purified by flash silica gel chromatography (EtOAc:Hexanes, 1:9 v/v) to yield pure dibromide **5A** (1.215 g, 1.73 mmol, 94%).

Compound 5A: White solid, mp 182.4−184.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.6 Hz, 1H), 7.70-7.65 (m, 1H), 7.54-7.47 (m, 1H), 7.43-7.33 (m, 2H), 7.27-7.22 (m, 1H), 7.10 (d, J = 8.0 Hz, 1H), 6.73 (d, J = 2.8 Hz, 2H), 6.43-6.38 (m, 2H), 6.31-6.27 (m, 2H), 4.30-4.24 (m, 4H), 3.65-3.59 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 157.3, 154.7, 151.8, 139.0, 138.6, 136.9, 134.6, 129.0, 128.9, 127.9, 125.6, 121.6, 120.0, 116.7, 111.2, 102.2, 93.4, 67.9, 53.2, 28.9; HRMS calcd. for $C_{29}H_{22}{}^{79}Br_2IO_3$ $[M+H]^+$: 702.8975, Found: 702.8989; IR(ATR): 3060, 2969, 2873, 2359, 1742, 1615, 1499, 1418, 1189 cm^{-1}.

General procedure for the synthesis of 5B-D:



A mixture of spirofluorenediol **4A** (1 equiv), dibromide (10 equiv) and K_2CO_3 (5 equiv) in CH_3CN (25 mL/mmol) was stirred under refluxing conditions for 4 h. After completion of

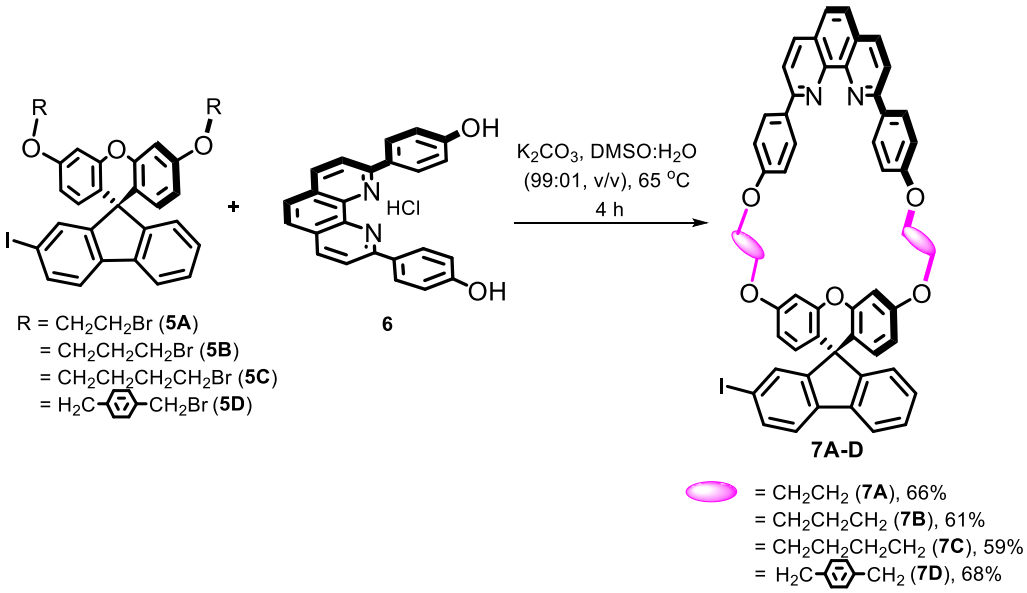
reaction, solvent was removed under vacuum and water was added to the residue. Extraction was done with CH_2Cl_2 (×3), combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The crude reaction mixture was purified using flash silica gel column chromatography (EtOAc:hexanes, 1:9 v/v) to yield the target compounds **5B** (73%), **5C** (81%) and **5D** (86%).

Compound 5B: 0.550 g (1.12 mmol) of **4A** and 1.14 mL (11.2 mmol) of 1,3-dibromopropane taken and 0.598 g (0.819 mmol) of **5B** obtained in 73% yield. White solid, mp 87.7−89.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 7.6 Hz, 1H), 7.68-7.64 (m, 1H), 7.52-7.46 (m, 1H), 7.43-7.39 (m, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.25-7.20 (m, 1H), 7.10 (d, J = 7.6 Hz, 1H), 6.73 (d, J = 2.0 Hz, 2H), 6.40-6.35 (m, 2H), 6.28-6.23 (m, 2H), 4.12-4.04 (t, J = 5.6 Hz, 4H), 3.60-3.53 (m, 4H), 2.33-2.25 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 157.4, 154.8, 151.9, 139.0, 138.6, 136.8, 134.6, 129.0, 128.8, 127.9, 125.6, 121.6, 119.9, 116.1, 111.0, 101.9, 93.4, 65.4, 53.3, 32.2, 29.9; HRMS calcd. for $C_{31}H_{26}{}^{79}Br_2IO_3$ $[M+H]^+$: 730.9288, Found: 730.9288; IR(ATR): 3060, 2973, 2359, 1749, 1610, 1497, 1252, 1181 cm^{-1}.

Compound 5C: 0.700 g (1.43 mmol) of **4A** and 1.70 mL (14.3 mmol) of 1,4-dibromobutane taken and 0.877 g (1.16 mmol) of **5C** obtained in 81% yield. White solid, mp 118.4−119.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 7.2 Hz, 1H), 7.65 (d, J = 8.4 Hz, 1H), 7.49 (d, J = 8.0 Hz, 1H), 7.42 (s, 1H), 7.34 (t, J = 7.2 Hz, 1H), 7.25-7.19 (m, 1H), 7.10 (d, J = 8.0 Hz, 1H), 6.71 (bs, 2H), 6.38-6.33 (m, 2H), 6.25 (d, J = 8.8 Hz, 2H), 3.96 (t, J = 6.0 Hz, 4H), 3.46 (t, J = 6.4 Hz, 4H), 2.08-2.00 (m, 4H), 1.96-1.88 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 157.5, 154.8, 151.9, 139.0, 138.6, 136.7, 134.6, 128.9, 128.7, 127.8, 125.6, 121.6, 119.9, 115.9, 111.0, 101.7, 93.4, 66.9, 53.3, 33.4, 29.4, 27.8; HRMS calcd. for $C_{33}H_{30}{}^{79}Br_2IO_3$ $[M+H]^+$: 758.9601, Found: 758.9605; IR(ATR): 3057, 2865, 1609, 1568, 1496, 1468, 1438, 1413 cm^{-1}.

Compound 5D: 0.600 g (1.22 mmol) of **4A** and 3.20 g (12.2 mmol) of 1,4-bis(bromomethyl)benzene taken and 0.900 g (1.05 mmol) of **5D** obtained in 86% yield. White solid, mp 203.1−204.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, J = 8.0 Hz, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.51-7.47 (m, 1H), 7.44-7.32 (m, 10H), 7.25-7.21 (m, 1H), 7.12-7.09 (m, 1H), 6.80-6.78 (m, 2H), 6.45-6.42 (m, 2H), 6.29-6.25 (m, 2H), 5.01 (s, 4H), 4.49 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 157.4, 154.8, 151.9, 139.0, 138.6, 137.6, 137.0, 136.8, 134.7, 129.3, 129.0, 128.8, 127.9, 127.8, 125.6, 121.6, 119.9, 116.3, 111.4, 102.2, 93.4, 69.7, 53.3, 33.1; HRMS calcd. for $C_{41}H_{33}{}^{79}Br_2INO_3$ $[M+NH_4]^+$: 871.9866, Found: 871.9863; IR(ATR): 3060, 2973, 2861, 2359, 1746, 1611, 1178 cm^{-1}.

General procedure for the Williamson's etherification (synthesis of 7A-D):



A mixture of 4,4'-(1,10-phenanthroline-2,9-diyl)diphenol hydrochloride **6**[8] (1 equiv), dibromospirofluorene (**5A-D**, 1 equiv) and powdered K_2CO_3 (10 equiv) in DMSO:H_2O (99:1, 250 mL/mmol) was stirred at 65 °C for 4 h. After completion of reaction, as confirmed from TLC, the solvent was removed under reduced pressure. Water was added to the crude reaction mixture and extraction was performed with CH_2Cl_2 (×3). The combined organic layer was washed with water and brine, dried over $MgSO_4$ and evaporated to dryness under vacuum. The residue was purified by flash silica gel column chromatography with CH_2Cl_2 as eluent to yield the corresponding macrocyclic phenanthroline complexes **7A-D**.

Compound 7A: 1.92 g (2.73 mmol) of **5A** taken and 1.64 g (1.80 mmol) of **7A** obtained in 66% yield. White solid, mp 339.4−342.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 7.2 Hz, 4H), 8.23 (d, J = 8.0 Hz, 2H), 8.03 (d, J = 8.0 Hz, 2H), 7.75-7.62 (m, 4H), 7.52-7.46 (d, J = 9.6 Hz, 2H), 7.32 (t, J = 6.8 Hz, 1H), 7.25-7.13 (m, 8H), 6.45-6.38 (m, 2H), 6.31-6.27 (m, 2H), 4.45 (d, J = 13.2 Hz, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.8, 157.0, 156.6, 154.4, 152.2, 146.1, 139.1, 138.6, 136.8, 136.7, 134.7, 133.3, 129.3, 129.0, 128.4, 127.8, 127.5, 125.8, 125.6, 121.5, 119.8, 119.5, 117.1, 115.8, 111.9, 103.7, 93.3, 68.2, 67.6, 53.6; HRMS calcd. for $C_{53}H_{36}IN_2O_5$ $[M+H]^+$: 907.1663, Found: 907.1663; IR(ATR): 3057, 2928, 2363, 1742, 1605, 1574, 1496, 1418, 1252, 1177 cm^{-1}.

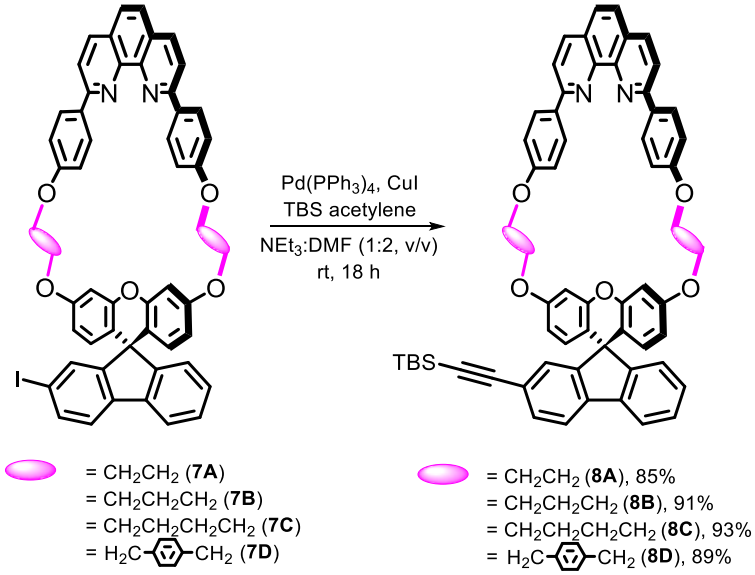
Compound 7B: 0.900 g (1.23 mmol) of **5B** taken and 0.703 g (0.752 mmol) of **7B** obtained in 61% yield. White solid, mp 348.5−350.2 °C (Decomp.); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, J = 7.6 Hz, 4H), 8.25 (d, J = 8.0 Hz, 2H), 8.07 (d, J = 8.2 Hz, 2H), 7.75-7.62 (m, 4H), 7.48 (d, J = 7.6 Hz, 2H), 7.32 (t, J = 6.8 Hz, 1H), 7.24 (s, 2H), 7.20-7.15 (m, 4H), 6.96 (s, 2H), 6.38 (d, J = 8.4 Hz, 2H), 6.26 (d, J = 8.4 Hz, 2H), 4.30-4.20 (m, 8H),

2.30-2.22 (t, *J* = 5.7 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.1, 158.8, 157.1, 156.5, 154.4, 152.3, 146.1, 139.1, 138.7, 136.7, 134.7, 132.7, 129.2, 129.0, 128.4, 127.8, 127.5, 125.8, 125.6, 121.5, 119.9, 119.4, 116.6, 115.2, 111.5, 102.3, 93.3, 64.7, 53.6, 28.6; HRMS calcd. for $C_{55}H_{40}IN_2O_5$ $[M+H]^+$: 935.1977, Found: 935.1961; IR(ATR): 3032, 2945, 2315, 1742, 1609, 1573, 1496, 1251, 1186 cm^{-1}.

Compound 7C: 0.750 g (0.989 mmol) of **5C** taken and 0.562 g (0.584 mmol) of **7C** obtained in 59% yield. White solid, mp 359.1−361.3 °C (Decomp.); ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.44 (d, *J* = 8.4 Hz, 4H), 8.24 (d, *J* = 8.4 Hz, 2H), 8.07 (d, *J* = 8.4 Hz, 2H), 7.75-7.62 (m, 4H), 7.48 (t, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.25-7.20 (m, 1H), 7.16-7.10 (m, 5H), 6.87-6.84 (m, 2H), 6.43-6.38 (m, 2H), 6.27-6.23 (m, 2H), 4.22-4.11 (m, 8H), 2.05 (bs, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.3, 159.0, 157.4, 156.3, 154.8, 152.0, 146.0, 139.1, 138.6, 136.8, 134.8, 132.2, 129.0, 128.7, 127.8, 127.5, 125.7, 125.6, 121.5, 119.9, 119.3, 115.7, 114.7, 112.7, 100.8, 93.4, 68.2, 67.9, 53.4, 26.9, 25.5; HRMS calcd. for $C_{57}H_{44}IN_2O_5$ $[M+H]^+$: 963.2289, Found: 963.2274; IR(ATR): 3040, 2920, 2869, 1604, 1570, 1488, 1395, 1245, 1175 cm^{-1}.

Compound 7D: 0.740 g (0.866 mmol) of **5D** taken and 0.623 g (0.589 mmol) of **7D** obtained in 68% yield. White solid, mp 243.6−246.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.33 (d, *J* = 8.8 Hz, 4H), 8.22 (d, *J* = 8.0 Hz, 2H), 8.03 (d, *J* = 8.0 Hz, 2H), 7.75-7.67 (m, 3H), 7.56-7.53 (m, 1H), 7.45-7.38 (m, 10H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.25-7.19 (m, 1H), 7.15-7.10 (m, 5H), 6.70 (d, *J* = 2.4 Hz, 2H), 6.45 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.27-6.22 (m, 2H), 5.20 (d, *J* = 16.8 Hz, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 160.1, 158.2, 156.6, 153.6, 152.2, 146.1, 138.9, 138.8, 136.8, 136.7, 136.7, 134.5, 132.8, 129.2, 128.8, 128.1, 127.8, 127.6, 127.1, 125.7, 121.5, 119.9, 119.7, 117.1, 115.4, 112.2, 102.8, 93.4, 70.1, 69.7, 53.6; HRMS calcd. for $C_{65}H_{44}IN_2O_5$ $[M+H]^+$: 1059.2289, Found: 1059.2288; IR(ATR): 3032, 2921, 2865, 2327, 1898, 1604, 1489, 1247, 1172 cm^{-1}.

General procedure for the synthesis of 8A-D:



A mixture of macrocyclic phenanthroline (**7A-D**, 1 equiv), $Pd(PPh_3)_4$ (10 mol%), CuI (25 mol%) and *tert*-butyldimethylsilylacetylene (3 equiv) in dry-NEt_3 and dry DMF (1:2 v/v, 40 mL/mmol) was stirred at room temperature and after completion of reaction (18 h), aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (1:1:2.5 v/v) were added for demetallation. After stirring at room temperature overnight, extraction was done with CH_2Cl_2 (× 3), the combined organic layer was washed with water and brine and dried over $MgSO_4$ before solvent removal under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with CH_2Cl_2/hexanes (6:4 v/v) as eluent to furnish the target compounds **8A-D** (85-93%).

Compound 8A: 1.40 g (1.54 mmol) of **7A** taken and 1.20 g (1.31 mmol) of **8A** obtained in 85% yield. White solid, mp 221.2−223.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (d, J = 8.5 Hz, 4H), 8.24 (d, J = 8.0 Hz, 2H), 8.03 (d, J = 8.0 Hz, 2H), 7.74-7.67 (m, 4H), 7.46 (d, J = 8.0 Hz, 1H), 7.31 (m, 2H), 7.22-7.15 (m, 8H), 6.41 (dd, J = 8.5, 2.5 Hz, 2H), 6.28 (d, J = 8.5 Hz, 2H), 4.50-4.40 (m, 8H), 0.88 (s, 9H), 0.06 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.7, 156.6, 155.6, 154.8, 152.2, 146.1, 140.1, 138.6, 136.7, 133.3, 132.0, 129.2, 129.1, 128.8, 128.5, 127.7, 127.5, 125.7, 125.6, 122.7, 120.0, 119.6, 119.5, 117.3, 115.8, 111.9, 106.0, 103.6, 92.8, 68.2, 67.6, 53.5, 26.1, 16.6, -4.7; HRMS calcd. for $C_{61}H_{51}N_2O_5Si$ $[M+H]^+$: 919.3562, Found: 919.3562; IR(ATR): 3036, 2928, 2853, 2359, 2141, 1742, 1605, 1575, 1488, 1417, 1247, 1176 cm^{-1}.

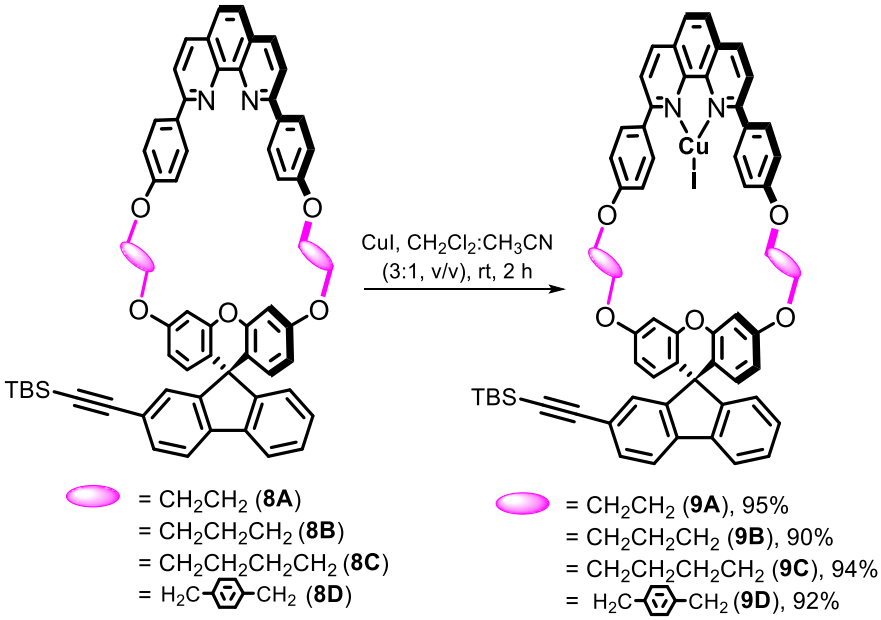
Compound 8B: 0.600 g (0.642 mmol) of **7B** taken and 0.553 g (0.584 mmol) of **8B** obtained in 91% yield. White solid, mp 222.6−225.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.42 (d, J = 8.5 Hz, 4H), 8.24 (d, J = 8.0 Hz, 2H), 8.06 (d, J = 8.0 Hz, 2H), 7.75-7.66 (m, 4H), 7.47-7.45 (m, 1H), 7.33-7.27 (m, 2H), 7.20-7.15 (m, 6H), 6.95 (d, J = 2.5 Hz, 2H), 6.37 (dd, J = 8.5, 2.5 Hz, 2H), 6.27 (d, J = 8.5 Hz, 2H), 4.30-4.20 (m, 8H), 2.29-2.23 (m, 4H), 0.88 (s, 9H), 0.06 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 158.7, 156.5, 155.6, 154.9,

152.3, 146.1, 140.0, 138.7, 136.7, 132.7, 132.0, 129.1, 128.8, 128.6, 127.7, 127.5, 125.7, 125.6, 122.7, 120.1, 119.6, 119.4, 116.7, 115.2, 111.5, 106.1, 102.2, 92.7, 64.7, 53.5, 28.5, 26.1, 16.6, -4.7; HRMS calcd. for $C_{63}H_{55}N_2O_5Si$ $[M+H]^+$: 947.3875, Found: 947.3873; IR(ATR): 3032, 2949, 2853, 2359, 2148, 1741, 1606, 1496, 1249, 1177 cm^{-1}.

Compound 8C: 0.500 g (0.520 mmol) of **7C** taken and 0.472 g (0.484 mmol) of **8C** obtained in 93% yield. White solid, mp 231.6−233.2 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.45 (d, J = 8.5 Hz, 4H), 8.22 (d, J = 8.0 Hz, 2H), 8.06 (d, J = 8.0 Hz, 2H), 7.75-7.68 (m, 4H), 7.49-7.46 (m, 1H), 7.34-7.28 (m, 2H), 7.22-7.15 (m, 2H), 7.13 (d, J = 8.5 Hz, 4H), 6.88 (d, J = 2.3 Hz, 2H), 6.41 (dt, J = 8.8, 2.0 Hz, 2H), 6.28 (dd, J = 9.0, 2.5 Hz, 2H), 4.16 (m, 8H), 2.04 (m, 8H), 0.92 (d, J = 1.0 Hz, 9H), 0.09 (d, J = 1.5 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.3, 158.9, 156.3, 155.9, 155.2, 152.0, 146.0, 140.0, 138.6, 136.7, 132.1, 132.0, 129.1, 128.9, 128.8, 127.7, 127.5, 125.7, 125.6, 122.7, 120.1, 119.6, 119.2, 115.9, 114.7, 112.6, 106.1, 100.7, 92.7, 68.1, 67.9, 53.3, 26.9, 26.1, 25.4, 16.6, -4.6; HRMS calcd. for $C_{65}H_{59}N_2O_5Si$ $[M+H]^+$: 975.4188, Found: 975.4187; IR(ATR): 3040, 2920, 2849, 2355, 2142, 1738, 1603, 1487, 1412, 1245, 1172 cm^{-1}.

Compound 8D: 0.450 g (0.425 mmol) of **7D** taken and 0.405 g (0.378 mmol) of **8D** obtained in 89% yield. White solid, mp 255.3−256.9 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, J = 8.5 Hz, 4H), 8.22 (d, J = 9.0 Hz, 2H), 8.04 (d, J = 8.5 Hz, 2H), 7.73-7.66 (m, 4H), 7.48-7.40 (m, 9H), 7.31 (t, J = 7.5 Hz, 1H), 7.27 (bs, 1H), 7.19 (t, J = 7.5 Hz, 1H), 7.16-7.12 (m, 5H), 6.71 (d, J = 3.0 Hz, 2H), 6.46 (dd, J = 8.5, 2.0 Hz, 2H), 6.28 (d, J = 8.5 Hz, 2H), 5.17 (d, J = 7.5 Hz, 8H), 0.94 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.2, 158.2, 156.6, 154.8, 154.3, 152.3, 146.1, 139.9, 138.8, 137.0, 136.7, 136.5, 132.8, 132.1, 129.2, 129.0, 128.7, 128.2, 127.8, 127.8, 127.6, 127.1, 125.7, 125.6, 122.6, 120.1, 119.7, 117.4, 115.4, 112.0, 106.1, 102.9, 92.8, 70.4, 69.9, 53.6, 26.1, 16.6, -4.6; HRMS calcd. for $C_{73}H_{59}N_2O_5Si$ $[M+H]^+$: 1071.4188, Found: 1071.4190; IR(ATR): 3036, 2928, 2853, 2363, 2148, 1738, 1605, 1574, 1495, 1248, 1173 cm^{-1}.

General procedure for the synthesis of 9A-D:



To the solution of phenanthroline macrocycle (**8A-D**, 1 equiv) in CH_2Cl_2 was added a suspension of CuI (1 equiv) in CH_3CN (ratio of CH_2Cl_2:CH_3CN = 3:1 v/v, 30 mL/mmol) and the mixture was stirred at room temperature for 2 h. After completion of reaction, the solvent was removed under vacuum, the residue was purified by flash silica gel column chromatography with CH_2Cl_2:MeOH (98:2 v/v) as eluent and finally recrystallized from CH_2Cl_2/hexane to furnish the phenanthroline-CuI complexes **9A-D** (90-95%).

Compound 9A: 0.850 g (0.926 mmol) of **8A** taken and 0.975 g (0.880 mmol) of **9A** obtained in 95% yield. Light brown solid, mp 248.7−250.2 °C (Decomp.); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, J = 7.6 Hz, 2H), 8.02-7.91 (m, 6H), 7.87 (bs, 2H), 7.70 (dd, J = 18.8, 7.2 Hz, 2H), 7.47-7.43 (m, 1H), 7.34-7.27 (m, 2H), 7.20-7.06 (m, 8H), 6.41 (dd, J = 8.8, 2.4 Hz, 2H), 6.24 (d, J = 8.8 Hz, 2H), 4.41 (s, 8H), 0.90 (s, 9H), 0.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.6, 155.7, 155.1, 152.3, 143.8, 139.9, 138.7, 137.9, 131.9, 131.4, 129.2, 128.8, 128.4, 127.7, 127.3, 125.9, 125.8, 124.6, 122.7, 120.0, 119.6, 116.8, 116.0, 112.7, 106.1, 103.3, 92.7, 67.2, 66.7, 53.5, 26.1, 16.6, -4.6; HRMS calcd. for $C_{61}H_{54}{}^{63}CuIN_3O_5Si$ $[M+NH_4]^+$: 1126.2168, Found: 1126.2169; IR(ATR): 3057, 2927, 2856, 2359, 2144, 1746, 1606, 1489, 1246, 1177 cm^{-1}.

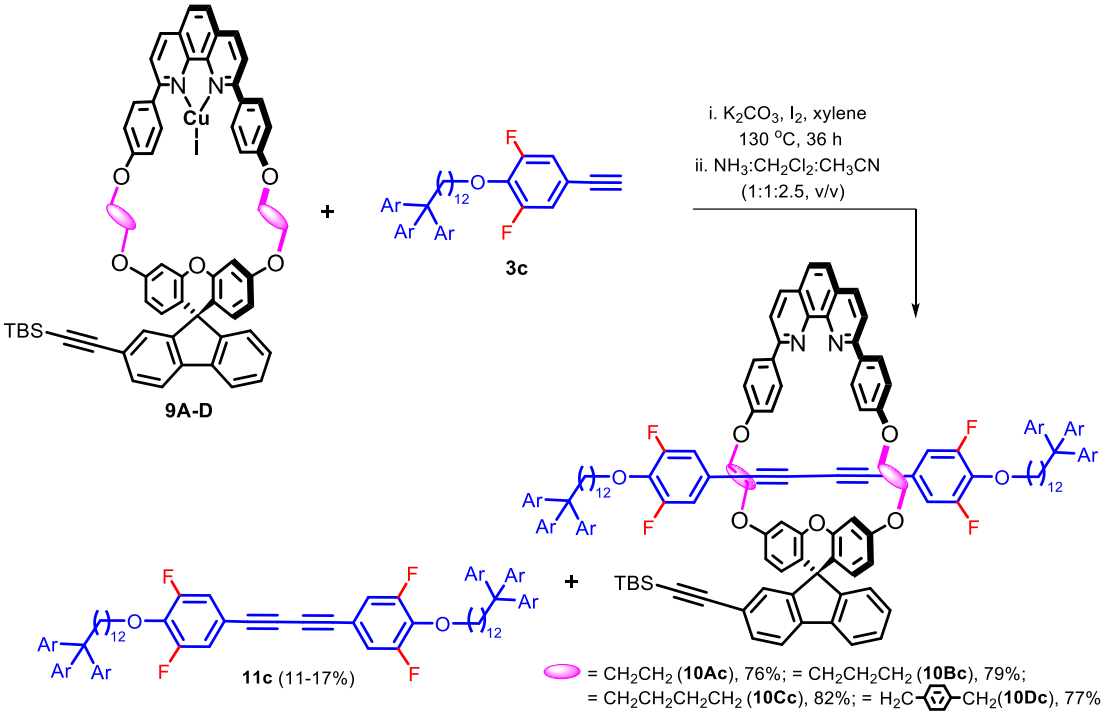
Compound 9B: 0.300 g (0.317 mmol) of **8B** taken and 0.325 g (0.286 mmol) of **9B** obtained in 90% yield. Light brown solid, mp 236.4−238.8 °C (Decomp.); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, J = 7.6 Hz, 2H), 8.08 (t, J = 8.0 Hz, 6H), 7.89 (s, 2H), 7.70 (dd, J = 19.6, 7.6 Hz, 2H), 7.45 (d, J = 7.6 Hz, 1H), 7.34-7.28 (m, 2H), 7.21-7.12 (m, 6H), 6.94 (s, 2H), 6.36 (d, J = 8.4 Hz, 2H), 6.24 (d, J = 8.4 Hz, 2H), 4.27-4.18 (m, 8H), 2.30-2.21 (m, 4H), 0.90 (s, 9H), 0.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.7, 158.7, 158.3, 155.6, 155.0, 152.4, 144.0, 139.9, 138.8, 137.8, 131.9, 131.1, 130.8, 129.2, 128.8, 128.3, 127.7, 127.3, 125.8, 124.3, 122.7, 120.0, 119.6, 116.7, 115.2, 111.5, 106.1, 102.6, 92.7,

65.1, 64.9, 53.5, 28.5, 26.1, 16.6, -4.6; HRMS calcd. for $C_{63}H_{58}{}^{63}CuIN_3O_5Si$ $[M+NH_4]^+$: 1154.2481, Found: 1154.2486; IR(ATR): 3060, 2949, 2857, 2357, 2148, 1607, 1496, 1415, 1255, 1180 cm^{-1}.

Compound 9C: 0.470 g (0.482 mmol) of **8C** taken and 0.527 g (0.453 mmol) of **9C** obtained in 94% yield. Yellow solid, mp 241.2−242.8 °C (Decomp.); ^1H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, J = 5.2 Hz, 2H), 8.15 (bs, 5H), 7.87 (bs, 2H), 7.70 (dd, J = 18.0, 7.2 Hz, 2H), 7.45 (d, J = 8.0 Hz, 1H), 7.35-7.26 (m, 2H), 7.22-7.08 (m, 6H), 6.86 (bs, 2H), 6.38 (d, J = 8.8 Hz, 2H), 6.25 (d, J = 8.4 Hz, 2H), 4.10 (bs, 8H), 2.02 (bs, 8H), 0.90 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.9, 158.8, 155.9, 155.1, 152.2, 140.0, 138.6, 137.8, 132.0, 130.9, 129.2, 128.8, 128.6, 127.7, 125.8, 122.7, 120.0, 119.6, 116.1, 114.7, 112.5, 106.1, 101.0, 92.7, 67.4, 53.4, 26.1, 25.8, 25.3, 16.6, -4.6 (some signals got merged); HRMS calcd. for $C_{65}H_{58}{}^{63}CuN_2O_5Si$ $[M-I]^+$: 1037.3406, Found: 1037.3405; IR(ATR): 3057, 2945, 2853, 2357, 2146, 1605, 1488, 1412, 1247, 1173 cm^{-1}.

Compound 9D: 0.250 g (0.233 mmol) of **8D** taken and 0.270 g (0.214 mmol) of **9D** obtained in 92% yield. Red solid, mp 256.6−258.8 °C (decomp.); ^1H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, J = 6.8 Hz, 2H), 8.10-7.93 (m, 6H), 7.77-7.65 (m, 4H), 7.47-7.41 (m, 9H), 7.30-7.23 (m, 2H), 7.18-7.08 (m, 6H), 6.69 (d, J = 1.6 Hz, 2H), 6.45 (dd, J = 8.8, 2.4 Hz, 2H), 6.25 (d, J = 8.8 Hz, 2H), 5.16 (d, J = 12.4 Hz, 8H), 0.92 (s, 9H), 0.09 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 158.3, 154.9, 154.4, 152.2, 143.8, 139.9, 138.7, 137.8, 136.8, 136.3, 132.0, 131.3, 130.7, 129.0, 128.7, 128.1, 127.7, 127.6, 127.2, 125.8, 125.6, 124.6, 122.6, 120.0, 119.6, 117.1, 115.3, 112.4, 106.1, 102.5, 92.7, 70.1, 70.0, 53.5, 26.1, 16.6, -4.6; HRMS calcd. for $C_{73}H_{58}{}^{63}CuN_2O_5Si$ $[M-I]^+$: 1133.3405, Found: 1133.3405; IR(ATR): 3032, 2926, 2861, 2367, 2141, 1746, 1605, 1490, 1416, 1249, 1173 cm^{-1}.

General procedure for the synthesis of rotaxanes 10Ac-Dc:



A sealed tube was charged with macrocyclic Cu-complex **9A-D**, (1 equiv), terminal alkyne **3c** (2.5 equiv), finely powdered K_2CO_3 (3.75 equiv), I_2 (1.25 equiv) and dry xylene (2 mL/0.01 mmol of **9A-D**) as solvent under argon. The reaction mixture was stirred for 36 h (12 h for **9D**) at 130 °C and after completion of reaction, aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (1:1:2.5 v/v, 2 mL: 2 mL: 5 mL/0.01 mmol of **9A-D**) were added for demetallation. After stirring the mixture at room temperature overnight, extraction was done with CH_2Cl_2 (× 3), the combined organic layer was washed with water and brine and dried over $MgSO_4$ before solvent was removed under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with EtOAc:hexanes (1:9 v/v) as eluent to furnish the target rotaxanes **10Ac-Dc** (76-82%) along with the dimerized axle component (**11c**) as side product. Further purification was done using GPC with $CHCl_3$ as eluent.

Compound 10Ac: 0.040 g (0.036 mmol) of **9A** and 0.094 g of **3c** (0.090 mmol) taken and 0.082 g (0.027 mmol) of **10Ac** and 0.032 g (0.015 mmol) of **11c** obtained in 76% and 17% (based upon **3c**) yield respectively. White solid, mp 171.2−173.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.20 (t, J = 8.0 Hz, 6H), 7.93 (d, J = 8.5 Hz, 2H), 7.73-7.70 (m, 3H), 7.64 (d, J = 8.0 Hz, 1H), 7.52-7.46 (m, 24H), 7.44-7.41 (m, 1H), 7.37-7.33 (m, 12H), 7.32-7.26 (m, 2H), 7.26-7.22 (m, 12H), 7.18 (d, J = 8.0 Hz, 2H), 7.12-7.07 (m, 6H), 7.04 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 8.0 Hz, 1H), 6.68 (m, 2H), 6.44 (m, 2H), 6.29 (m, 2H), 4.53-4.36 (m, 8H), 4.03 (t, J = 6.5 Hz, 2H), 3.83 (t, J = 6.5 Hz, 2H), 2.61 (m, 4H), 2.55-2.48 (m, 6H), 1.86 (dd, J = 26.0, 10.0 Hz, 24H), 1.74 (d, J = 12.0 Hz, 6H), 1.68-1.63 (m, 2H), 1.56-1.52 (m, 4H), 1.47-1.30 (m, 30H), 1.29-1.12 (m, 34H), 0.92 (s, 9H), 0.09 (s, 6H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 159.2, 158.0, 156.8, 155.2 (dd, J = 248.8 Hz, 6.7 Hz), 154.7 (dd, J = 247.9, 6.7 Hz), 154.4, 153.7, 152.7, 147.0, 146.4, 139.6, 139.2, 138.3, 138.2, 137.6 (t, J = 13.4 Hz), 137.2 (t, J = 14.4 Hz), 136.5, 133.5, 132.2, 129.6, 129.4, 128.6, 128.5, 128.3, 127.9, 127.4, 127.2, 126.8, 126.2, 125.8, 125.6, 122.7, 120.1, 119.8, 118.2, 117.0 (m), 116.8 (m), 115.3 (m), 113.2, 106.1, 102.9, 92.7, 81.3, 80.8, 75.6, 75.0, 74.8, 74.6, 66.6, 66.1, 56.0, 53.8, 44.2, 40.5, 34.4, 30.5, 29.9, 29.8, 29.7, 29.6, 29.5, 29.3, 29.2, 26.9, 26.2, 26.1, 25.7, 25.6, 25.5, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.121, -127.865; HRMS calcd. for $C_{211}H_{217}F_4N_2O_7Si$ $[M+H]^+$: 2994.6386, Found: 2994.6340; IR(ATR): 3025, 2924, 2850, 2324, 2144, 1740, 1506, 1340, 1242, 1172 cm^{-1}.

Compound 10Bc: 0.020 g (0.018 mmol) of **9B** and 0.046 g of **3c** (0.044 mmol) taken and 0.042 g (0.014 mmol) of **10Bc** and 0.013 g (0.0060 mmol) of **11c** obtained in 79 % and 14% (based upon **3c**) yield respectively. Light yellow solid, mp 179.1−181.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, J = 8.5 Hz, 4H), 8.20 (d, J = 8.5 Hz, 2H), 7.98 (d, J = 8.5 Hz, 2H), 7.72-7.68 (m, 3H), 7.64 (d, J = 8.0 Hz, 1H), 7.52-7.41 (m, 25H), 7.37-7.28 (m, 14H), 7.26-7.21 (m, 12H), 7.14 (d, J = 8.0 Hz, 4H), 7.07-7.03 (m, 3H), 6.98-6.94 (m, 2H), 6.87 (d, J = 7.5 Hz, 1H), 6.75-6.69 (m, 2H), 6.37-6.33 (m, 2H), 6.26-6.22 (m, 2H), 4.30-4.08 (m, 8H), 3.97 (t, J = 6.5 Hz, 2H), 3.85 (t, J = 6.5 Hz, 2H), 2.63-2.56 (m, 4H), 2.55-2.46 (s, 6H), 2.27-2.18 (m, 4H), 1.86 (dd, J = 25.5, 10.5 Hz, 24H), 1.74 (d, J = 12.5 Hz, 6H), 1.65-1.52 (m, 4H), 1.48-1.30(m, 32H), 1.29-1.10 (m, 34H), 0.91 (s, 9H), 0.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 158.9, 156.4, 155.1 (dd, J = 248.9, 6.8 Hz), 154.9 (dd, J = 247.9, 6.7 Hz), 154.4, 154.3, 152.7, 147.0, 146.4, 146.3, 139.5, 139.2, 138.3, 138.2, 137.4 (t, J = 13.4 Hz), 137.2 (t, J = 13.5 Hz), 136.5, 132.9, 132.1, 129.6, 129.1, 128.8, 128.6, 128.0, 127.8, 127.4, 127.1, 126.8, 126.2, 125.7, 125.6, 122.7, 120.0, 119.7, 119.3, 117.5, 116.8 (m), 116.6 (m), 115.6 (m), 115.1, 111.6, 106.1, 102.3, 92.7, 80.5, 75.3, 74.83, 74.76, 74.66, 65.0, 64.8, 56.0, 53.7, 44.2, 40.5, 34.4, 30.5, 29.9, 29.8, 29.7, 29.63, 29.58, 29.30, 29.26, 29.0, 26.9, 26.2, 26.1, 25.8, 25.6, 25.5, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.312, -127.647; HRMS calcd. for $C_{213}H_{221}F_4N_2O_7Si$ $[M+H]^+$: 3022.6699, Found: 3022.6685; IR(ATR): 3020, 2923, 2853, 2362, 2163, 1734, 1555, 1340, 1236 cm^{-1}.

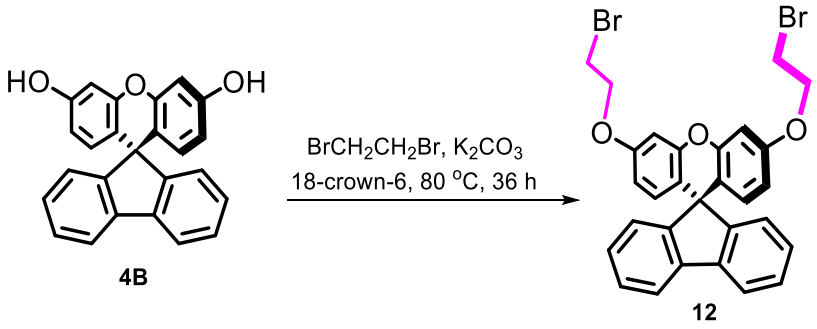
Compound 10Cc: 0.040 g (0.034 mmol) of **9C** and 0.089 g of **3c** (0.086 mmol) taken and 0.086 g (0.028 mmol) of **10Cc** and 0.019 g (0.0090 mmol) of **11c** obtained in 82% and 11% (based upon **3c**) yield respectively. Light yellow solid, mp 174.3−176.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.44 (d, J = 7.0 Hz, 4H), 8.22 (d, J = 8.5 Hz, 2H), 8.04 (d, J = 8.0 Hz, 2H), 7.74-7.66 (m, 4H), 7.53-7.45 (m, 26H), 7.39-7.33 (m, 12H), 7.29 (t, J = 7.5 Hz, 1H), 7.27-7.21 (m, 12H), 7.16-7.01 (m, 8H), 6.96 (d, J = 7.0 Hz, 2H), 6.86 (bs, 2H), 6.39-6.36 (m, 2H), 6.27-6.24 (m, 2H), 4.16-3.98 (m, 12H), 2.66-2.58 (m, 4H), 2.56-2.47 (m, 6H), 2.00 (bs, 8H), 1.88 (dd, J = 26.0, 10.5 Hz, 24H), 1.75 (d, J = 11.5 Hz, 6H), 1.70-1.60 (m, 4H), 1.50-1.32 (m, 32H), 1.31-1.14 (m, 34H), 0.93 (s, 9H), 0.10 (s, 6H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 160.2, 158.8, 156.4 (m), 156.3, 155.6, 155.1, 153.9 (m), 152.1, 147.0, 146.4, 146.1, 139.9, 138.7, 138.3, 138.2, 137.7 (m), 137.6 (m), 136.6, 132.3, 132.0, 129.6, 129.0, 128.7, 128.6, 127.7, 127.5, 127.1, 126.8, 126.2, 125.7, 125.6, 122.7, 120.0, 119.6, 119.3, 116.8 (m), 116.6 (m), 116.1, 115.4 (m), 114.6, 112.7, 106.1, 100.8, 92.7, 80.3, 74.9, 74.83, 74.79, 67.9, 67.6, 56.0, 53.4, 44.2, 40.6, 34.4, 30.6, 29.91, 29.87, 29.77, 29.63, 29.57, 29.3, 26.9, 26.6, 26.2, 26.1, 25.8, 25.6, 25.5, 25.3, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -126.999, -127.183; HRMS calcd. for $C_{215}H_{225}F_4N_2O_7Si$ $[M+H]^+$: 3050.7012, Found: 3050.6921; IR(ATR): 3025, 2924, 2850, 2320, 2144, 1742, 1606, 1506, 1335, 1245 cm^{-1}.

Compound 10Dc: 0.040 g (0.032 mmol) of **9D** and 0.082 g of **3c** (0.079 mmol) were taken and 0.077 g (0.024 mmol) of **10Dc** and 0.024 g (0.012 mmol) of **11c** was obtained in 77% and 15% (based upon **3c**) yields respectively. Off-white solid, mp 178.5−181.1 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.31 (d, J = 8.5 Hz, 4H), 8.20 (d, J = 8.5 Hz, 2H), 8.02 (d, J = 8.5 Hz, 2H), 7.70-7.67 (m, 3H), 7.64 (d, J = 8.0 Hz, 1H), 7.50-7.42 (m, 26H), 7.39-7.26 (m, 21H), 7.24-7.20 (m, 12H), 7.10 (t, J = 7.0 Hz, 1H), 7.05-6.99 (m, 5H), 6.90-8.84 (m, 2H), 6.82-6.75 (m, 2H), 6.55 (d, J = 2.5 Hz, 2H), 6.39 (dd, J = 8.5, 2.5 Hz, 2H), 6.22 (d, J = 8.5 Hz, 2H), 5.07 (d, J = 10.0 Hz, 8H), 4.02-3.95 (m, 4H), 2.69-2.46 (m, 10H), 1.85 (dd, J = 23.5, 10.5 Hz, 24H), 1.73 (d, J = 13.0 Hz, 6H), 1.63-1.56 (m, 4H), 1.47-1.33 (m, 24H), 1.31-1.22 (m, 12H), 1.20-1.06 (m, 30H), 0.91 (m, 9H), 0.08 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.3, 158.3, 156.5, 156.4 (m), 154.8, 154.6, 153.9 (m), 152.2, 147.0, 146.4, 146.2, 139.7, 139.0, 138.3, 138.2, 137.63 (m), 137.58 (m), 137.0, 136.7, 132.7, 132.1, 129.6, 129.1, 128.9, 128.7, 128.3, 127.8, 127.7, 127.6, 127.1, 127.0, 126.8, 126.2, 125.7, 122.7, 120.1, 119.7, 119.6, 117.2, 116.7 (m), 116.5 (m), 115.3, 111.9, 106.1, 102.7, 92.7, 79.9, 74.9, 74.4, 70.3, 69.8, 56.0, 53.6, 44.2, 40.5, 34.4, 30.5, 29.9, 29.8, 29.60, 29.56, 29.3, 26.9, 26.3, 26.1, 25.8, 25.5, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -126.977, -127.047; HRMS calcd. for $C_{223}H_{225}F_4N_2O_7Si$ $[M+H]^+$: 3146.7012, Found: 3146.7027; IR(ATR): 3020, 2925, 2851, 2320, 2144, 1730, 1603, 1506, 1341, 1246, 1172 cm^{-1}.

Compound 11c: Off-white solid, mp 131.2−133.6 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.52 (t, J = 8.5 Hz, 24H), 7.36 (d, J = 8.0 Hz, 12H), 7.25 (d, J = 8.0 Hz, 12H), 7.03 (m, 4H), 4.15 (t, J = 6.5 Hz, 4H), 2.64-2.58 (m, 4H), 2.55-2.47 (m, 6H), 1.87 (dd, J = 27.0, 10.5 Hz, 24H), 1.78-1.68 (m, 10H), 1.49-1.31 (m, 32H), 1.30-1.10 (m, 34H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4 (m, J = 248.8, 6.7 Hz), 147.0, 146.4, 138.4, 138.2, 137.8 (t, J = 14.4 Hz), 129.6, 127.2, 126.8, 126.2, 116.6 (m), 115.4 (t, J = 11.5 Hz), 79.8, 74.9, 74.2, 56.0, 44.2, 40.5, 34.4, 30.4, 29.9, 29.63, 29.55, 29.5, 29.2, 26.9, 26.2, 25.7, 25.5 (few signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.032; HRMS calcd. for $C_{150}H_{166}F_4NaO_2$ $[M+Na]^+$: 2098.2716, Found: 2098.2654; IR(ATR): 3020, 2924, 2851, 2331, 2154, 1901, 1742, 1506, 1341, 1244, cm^{-1}.

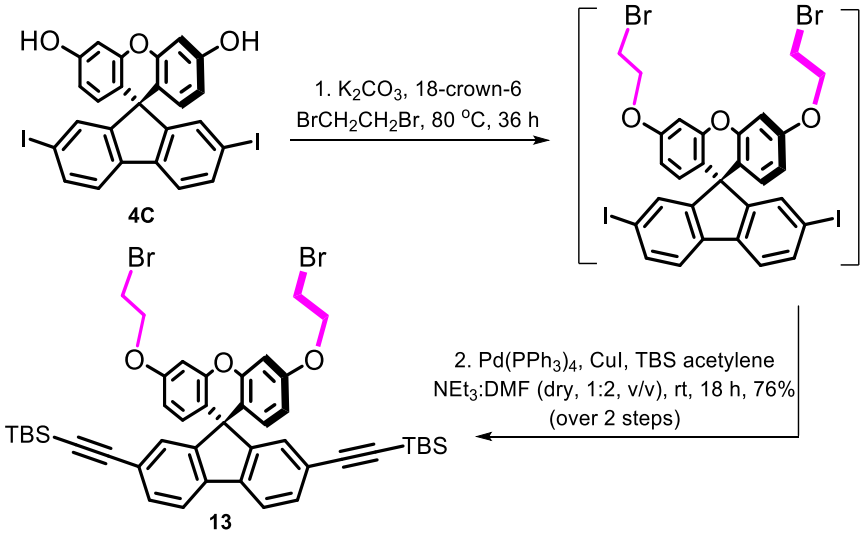
Synthesis of compound 12:



A stirred suspension of spirofluorenediol **4B**[9] (0.470 g, 1.29 mmol, 1 equiv), finely powdered K_2CO_3 (0.446 g, 3.22 mmol, 2.5 equiv) and 18-crown-6 (0.034 g, 0.129 mmol, 0.1 equiv) in 1,2-dibromoethane (15 mL) was heated at 80 °C for 36 h. After completion of reaction, the mixture was cooled, filtered, and washed with CH_2Cl_2. The filtrate was evaporated under vacuum to yield a residue that was purified by flash silica gel chromatography ($CHCl_3$) to yield pure dibromide **12** (0.675 g, 1.17 mmol, 91%).

Compound 12: White solid, mp 118.1−118.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, J = 8.0 Hz, 2H), 7.35 (t, J = 7.2 Hz, 2H), 7.20 (t, J = 7.6 Hz, 2H), 7.12 (d, J = 7.6 Hz, 2H), 6.73 (d, J = 2.8 Hz, 2H), 6.38 (dd, J = 8.8, 2.4 Hz, 2H), 6.30 (d, J = 8.4 Hz, 2H), 4.24 (t, J = 6.4 Hz, 4H), 3.60 (t, J = 6.4 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 155.2, 152.0, 139.5, 128.9, 128.4, 127.7, 125.5, 119.9, 117.6, 111.1, 102.1, 67.9, 53.3, 28.9; HRMS calcd. for $C_{29}H_{23}{}^{79}Br_2O_3$ $[M+H]^+$: 577.0008, Found: 577.0014; IR(ATR): 3032, 2853, 2367, 2143, 1738, 1615, 1498, 1414, 1259, 1191 cm^{-1}.

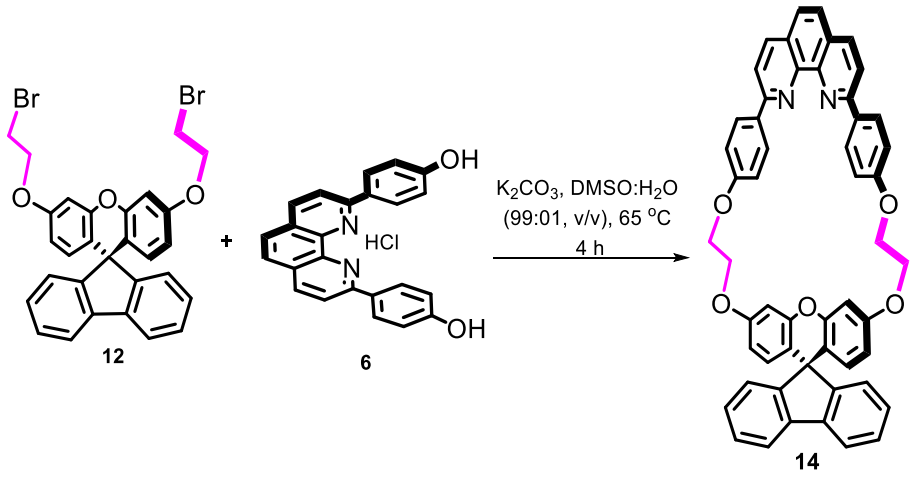
Synthesis of Compound 13:



A suspension of diiodo spirofluorenediol **4C**[10] (0.540 g, 0.877 mmol, 1 equiv), finely powdered K_2CO_3 (0.303 g, 2.19 mmol, 2.5 equiv) and 18-crown-6 (0.023 g, 0.088 mmol, 0.1 equiv) was stirred in 1,2-dibromoethane (15 mL) at 80 °C for 36 h. After completion of reaction, the mixture was cooled, filtered, and washed with CH_2Cl_2. The filtrate was evaporated under vacuum to yield a residue. Because of its low solubility, the residue was washed with solvent (EtOAc:hexane 3:7 v/v) and used for the next reaction without further purification. To the solid was added $Pd(PPh_3)_4$ (0.138 g, 0.119 mmol, 15 mol%), CuI (0.045 g, 0.239 mmol, 30 mol%), *tert*-butyldimethylsilyl acetylene (0.725 mL, 3.98 mmol, 5 equiv), anhydrous NEt_3 (8 mL) and anhydrous DMF (16 mL). The mixture was stirred at rt for 18 h, and aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (10 mL: 10 mL: 25 mL) were added for demetallation. After stirring at room temperature overnight, extraction was done with CH_2Cl_2 (3 × 30 mL), the combined organic layer was washed with water and brine and dried over $MgSO_4$ before solvent was removed under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with CH_2Cl_2/hexanes (6:4 v/v) as eluent to furnish the target compound **13** (0.570 g, 0.669 mmol, 76% over 2 steps).

Compound 13: White solid, mp 291.2−292.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, J = 7.5 Hz, 2H), 7.46 (d, J = 8.0 Hz, 2H), 7.16 (s, 2H), 6.72 (d, J = 2.5 Hz, 2H), 6.38 (dd, J = 9.0, 3.0 Hz, 2H), 6.24 (d, J = 8.5 Hz, 2H), 4.26 (t, J = 6.0 Hz, 4H), 3.61 (t, J = 6.0 Hz, 4H), 0.93 (s, 18H), 0.11 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.0, 155.6, 151.8, 139.0, 132.2, 129.1, 129.0, 123.3, 120.0, 116.2, 111.2, 105.8, 102.2, 93.4, 67.9, 53.1, 28.8, 26.1, 16.7, -4.6; HRMS calcd. for $C_{45}H_{51}{}^{79}Br_2O_3Si_2$ $[M+H]^+$: 853.1738, Found: 853.1745; IR(ATR): 3029, 2951, 2849, 2363, 2148, 1748, 1614, 1498, 1464, 1417, 1252, 1178 cm^{-1}.

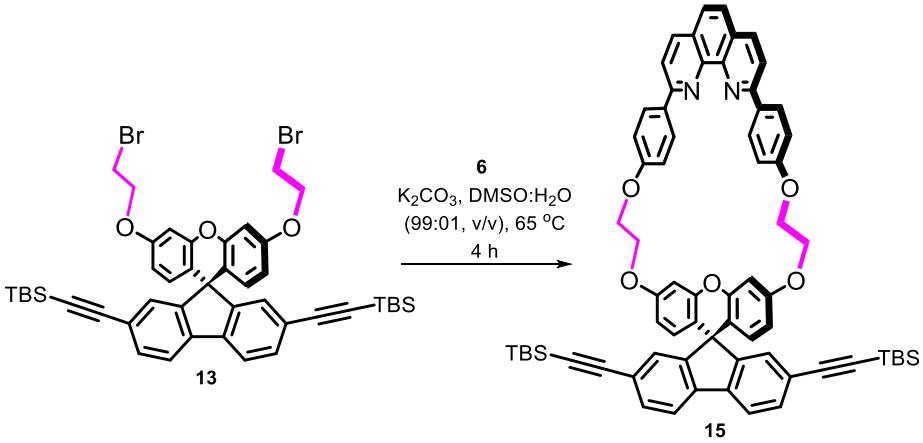
Synthesis of macrocycle 14:



A mixture of 4,4'-(1,10-phenanthroline-2,9-diyl)diphenol hydrochloride **6** (0.278 g, 0.694 mmol, 1 equiv), dibromospirofluorene **12** (0.400 g, 0.694 mmol, 1 equiv) and powdered K_2CO_3 (0.960 g, 6.94 mmol, 10 equiv) in DMSO:H_2O (297 mL: 3 mL) was stirred at 65 °C for 4 h. After completion of reaction, as confirmed from TLC, the solvent was removed under vacuum. Water was added to the crude reaction mixture and extraction was performed with CH_2Cl_2 (60 mL × 3). The combined organic layer was washed with water and brine, dried over $MgSO_4$ and evaporated to dryness under vacuum. The residue was purified by flash silica gel column chromatography with $CHCl_3$ as eluent to yield the macrocyclic phenanthroline complex **14** (0.320 g, 0.410 mmol, 59%).

Compound 14: White solid, mp 308.4−310.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 8.8 Hz, 4H), 8.26 (d, J = 8.8 Hz, 2H), 8.04 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 6.0 Hz, 4H), 7.34-7.28 (m, 2H), 7.21-7.16 (m, 8H), 7.15-7.11 (m, 2H), 6.40 (dd, J = 8.8, 2.4 Hz, 2H), 6.28 (d, J = 8.8 Hz, 2H), 4.49-4.39 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.6, 156.7, 155.0, 152.3, 146.1, 139.5, 136.7, 133.3, 129.3, 128.4, 128.3, 127.6, 127.5, 125.73, 125.68, 119.8, 119.6, 118.0, 115.9, 111.8, 103.6, 68.2, 67.6, 53.6; HRMS calcd. for $C_{53}H_{37}N_2O_5$ $[M+H]^+$: 781.2697, Found: 781.2689; IR(ATR): 3036, 2952, 2359, 2339, 1738, 1611, 1575, 1498, 1416, 1248, 1189 cm^{-1}.

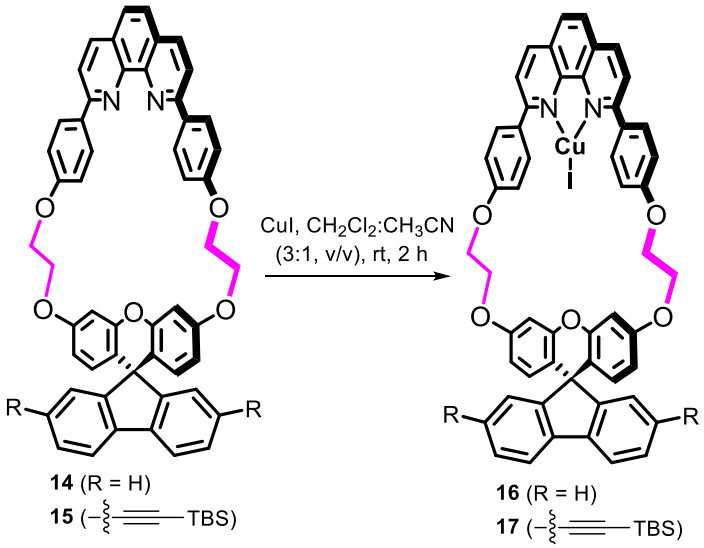
Synthesis of Compound 15:



A mixture of **6** (0.211 g, 0.528 mmol, 1 equiv), dibromospirofluorene **13** (0.450 g, 0.528 mmol, 1 equiv) and powdered K_2CO_3 (0.730 g, 5.28 mmol, 10 equiv) in DMSO:H_2O (297 mL:3 mL) was stirred at 65 °C for 4 h. After completion of reaction, as confirmed from TLC, the solvent was removed under vacuum. Water was added to the crude reaction mixture and extraction was performed with CH_2Cl_2 (3 × 50 mL). The combined organic layer was washed with water and brine, dried over $MgSO_4$, and evaporated to dryness under vacuum. The residue was purified by flash silica gel column chromatography with CH_2Cl_2 as eluent to yield the macrocyclic phenanthroline complex **15** (0.378 g, 0.358 mmol, 68%).

Compound 15 : White solid, mp 372.8−374.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.36 (d, J = 8.5 Hz, 4H), 8.25 (d, J = 8.5 Hz, 2H), 8.04 (d, J = 8.5 Hz, 2H), 7.74 (s, 2H), 7.64 (d, J = 8.0 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 3.0 Hz, 2H), 7.21 (d, J = 8.5 Hz, 4H), 7.15 (d, J = 2.5 Hz, 2H), 6.41 (dd, J = 8.5, 2.0 Hz, 2H), 6.25 (d, J = 9.0 Hz, 2H), 4.50-4.42 (m, 8H), 0.86 (s, 18H), 0.03 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 158.8, 156.7, 155.3, 152.2, 146.1, 139.0, 136.6, 133.4, 132.1, 129.3, 129.1, 128.5, 127.5, 125.6, 123.2, 119.9, 119.6, 116.6, 115.9, 112.0, 105.9, 103.6, 93.2, 68.1, 67.7, 53.4, 26.1, 16.6, -4.7; HRMS calcd. for $C_{69}H_{65}N_2O_5Si_2$ $[M+H]^+$: 1057.4427, Found: 1057.4426; IR(ATR): 3032, 2949, 2853, 2363, 2152, 1746, 1602, 1489, 1462, 1249, 1178 cm^{-1}.

General procedure for the synthesis of 16 and 17:

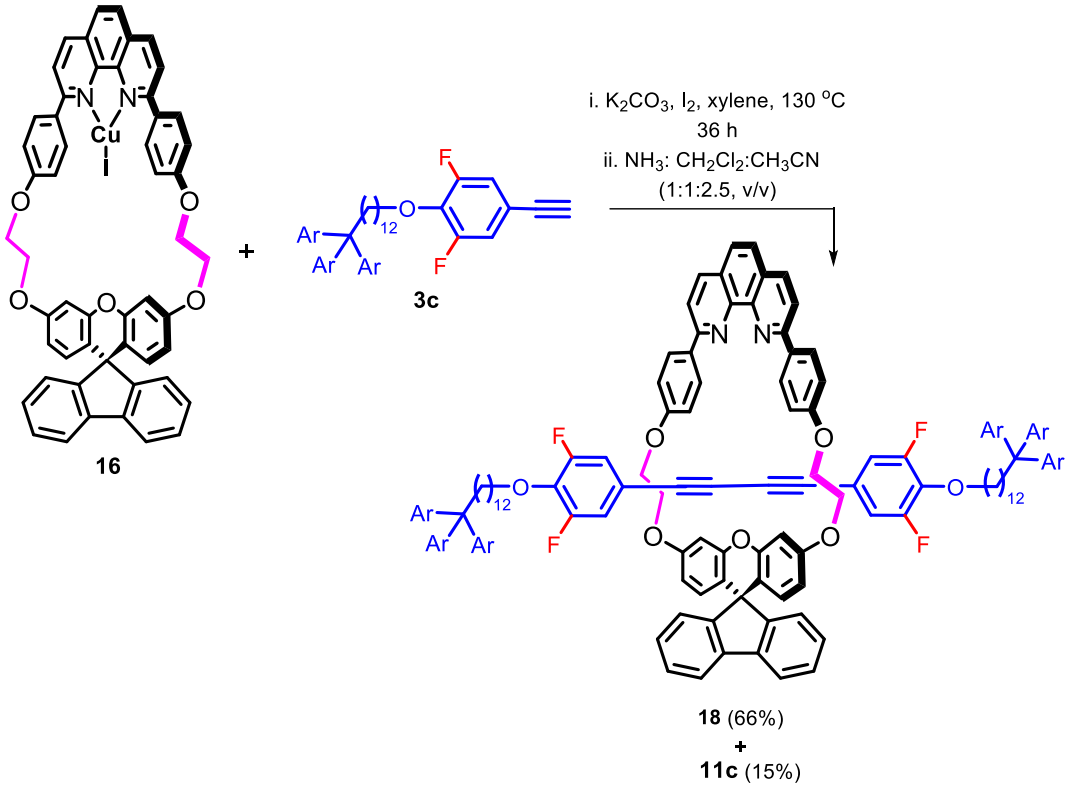


To a solution of phenanthroline macrocycle **14** or **15** (1 equiv) in CH_2Cl_2 was added CuI (1 equiv) in CH_3CN (ratio of CH_2Cl_2:CH_3CN = 3:1 v/v, 32 mL/mmol) and the mixture was stirred at room temperature for 2 h. After completion of reaction, the solvent was removed under vacuum, the residue was purified by flash silica gel column chromatography with CH_2Cl_2:MeOH (98:2 v/v) as eluent to furnish the phenanthroline-CuI complexes **16** and **17**.

Compound 16: 0.350 g (0.449 mmol) of **14** taken and 0.388 g (0.400 mmol) of **16** obtained in 89% yield. Brown solid, mp 330.1−332.7 °C (Decomp.); ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.83 (d, J = 8.0 Hz, 2H), 8.26-8.18 (m, 8H), 7.93 (d, J = 7.2 Hz, 2H), 7.36 (t, J = 7.2 Hz, 2H), 7.19-7.11 (m, 6H), 7.02-6.97 (d, J = 8.8 Hz, 2H), 6.91 (s, 2H), 6.50 (d, J = 8.0 Hz, 2H), 6.12 (d, J = 8.4 Hz, 2H), 4.46 (d, J = 30.0 Hz, 8H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 159.0, 158.0, 157.8, 153.8, 151.5, 142.8, 138.9, 138.4, 132.7, 131.0, 128.3, 127.8, 127.6, 126.3, 124.8, 120.2, 117.2, 115.2, 112.0, 102.9, 66.0, 52.8 (some signals are merged); HRMS calcd. for $C_{53}H_{36}{}^{63}CuN_2O_5$ $[M\text{-}I]^+$: 843.1915, Found: 843.1931; IR(ATR): 3032, 2928, 2855, 2356, 2142, 1742, 1605, 1490, 1416, 1249, 1177 cm^{-1}.

Compound 17: 0.200 g (0.189 mmol) of **15** taken and 0.217 g (0.174 mmol) of **17** obtained in 92% yield. Off-yellow solid, mp 252.3−254.7 °C (Decomp.); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, J = 8.4 Hz, 2H), 8.07-7.98 (m, 6H), 7.90 (s, 2H), 7.64 (d, J = 8.0 Hz, 2H), 7.43 (d, J = 8.0 Hz, 2H), 7.24 (s, 2H), 7.18-7.11 (m, 6H), 6.41 (dd, J = 8.4, 2.4 Hz, 2H), 6.21 (d, J = 8.8 Hz, 2H), 4.45 (m, 8H), 0.89 (s, 18H), 0.07 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 159.2, 158.7, 155.7, 152.2, 144.0, 139.1, 137.8, 132.0, 131.6, 129.3, 128.4, 127.4, 125.9, 124.8, 123.2, 119.8, 116.2, 116.0, 112.9, 106.0, 103.1, 93.1, 67.1, 66.5, 53.4, 26.2, 16.6, -4.6; HRMS calcd. for $C_{69}H_{68}{}^{63}CuIN_3O_5Si$ $[M+NH_4]^+$: 1264.3033, Found: 1264.3083; IR(ATR): 3060, 2927, 2858, 2359, 2144, 1734, 1607, 1495, 1416, 1249, 1179 cm^{-1}.

Synthesis of rotaxane 18:

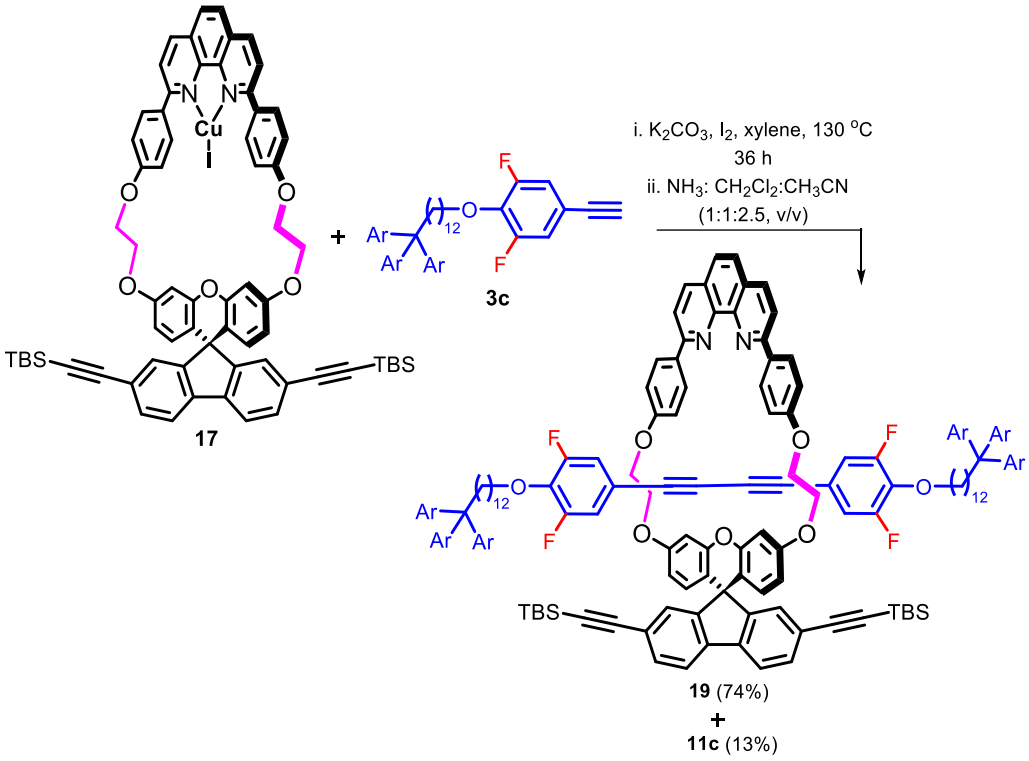


A sealed tube was charged with macrocyclic Cu-complex **16** (0.030 g, 0.031 mmol, 1 equiv), terminal alkyne **3c** (0.080 g, 0.077 mmol, 2.5 equiv), finely powdered K_2CO_3 (0.016 g, 0.12 mmol, 3.8 equiv), I_2 (0.010 g, 0.039 mmol, 1.3 equiv) and dry THF (7 mL) as solvent under argon. The reaction mixture was stirred for 36 h at 65 °C and after completion of reaction, aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (4mL: 4 mL: 10 mL) were added for demetallation. After stirring the mixture at room temperature overnight, extraction was done with CH_2Cl_2 (15 mL × 3), the combined organic layer was washed with water and brine and dried over $MgSO_4$ before solvent was removed under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with hexane:EtOAc (9:1 v/v) as eluent to furnish the target rotaxane **18** (0.058 g, 0.020 mmol, 66%) along with the dimerized axle component **11c** (0.024 g, 0.012 mmol, 15%, based upon **3c**) as side product. Further purification was carried out by GPC with $CHCl_3$ as mobile phase.

Compound 18: Light yellow solid, mp 167.6−169.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (t, J = 6.5 Hz, 6H), 7.94 (d, J = 8.0 Hz, 2H), 7.73-7.69 (m, 4H), 7.49 (t, J = 8.5 Hz, 24H), 7.35 (d, J = 8.5 Hz, 12H), 7.28 (t, J = 7.5 Hz, 1H), 7.26-7.22 (m, 13H), 7.12 (d, J = 8.0 Hz, 4H), 7.07-7.05 (m, 4H), 6.99 (d, J = 7.5 Hz, 2H), 6.90 (m, 4H), 6.44 (m, 2H), 6.31 (d, J = 8.5 Hz, 2H), 4.44 (m, 8H), 3.92 (t, J = 7.0 Hz, 4H), 2.63-2.57 (m, 4H), 2.54-2.47 (m, 6H), 1.86 (dd, J = 26.5, 11.0 Hz, 24H), 1.74 (d, J = 12.5 Hz, 6H), 1.62-1.55 (m, 4H), 1.48-1.27 (m, 32H), 1.26-1.10 (m, 34H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 157.8, 156.6, 154.9 (dd, J = 248.8, 6.7 Hz), 153.8, 152.9, 147.0, 146.4, 146.3, 139.6, 138.3, 138.2, 137.4 (t,

J = 14.4 Hz), 136.5, 133.4, 129.6, 129.3, 128.3, 128.1, 127.7, 127.4, 127.2, 126.8, 126.2, 125.6, 125.4, 119.9, 119.6, 119.0, 116.9 (m), 115.3 (m), 113.1, 103.0, 81.0, 75.3, 74.7, 66.7, 66.1, 56.0, 53.9, 44.2, 40.5, 34.4, 30.5, 29.8, 29.7, 29.6, 29.5, 29.2, 26.9, 26.2, 25.7, 25.5 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.504; HRMS calcd. for $C_{203}H_{203}F_4N_2O_7$ $[M+H]^+$: 2856.5521, Found: 2856.5533; IR(ATR): 3025, 2923, 2850, 2320, 2145, 1749, 1506, 1340, 1242, 1172 cm^{-1}.

Synthesis of rotaxane 19:

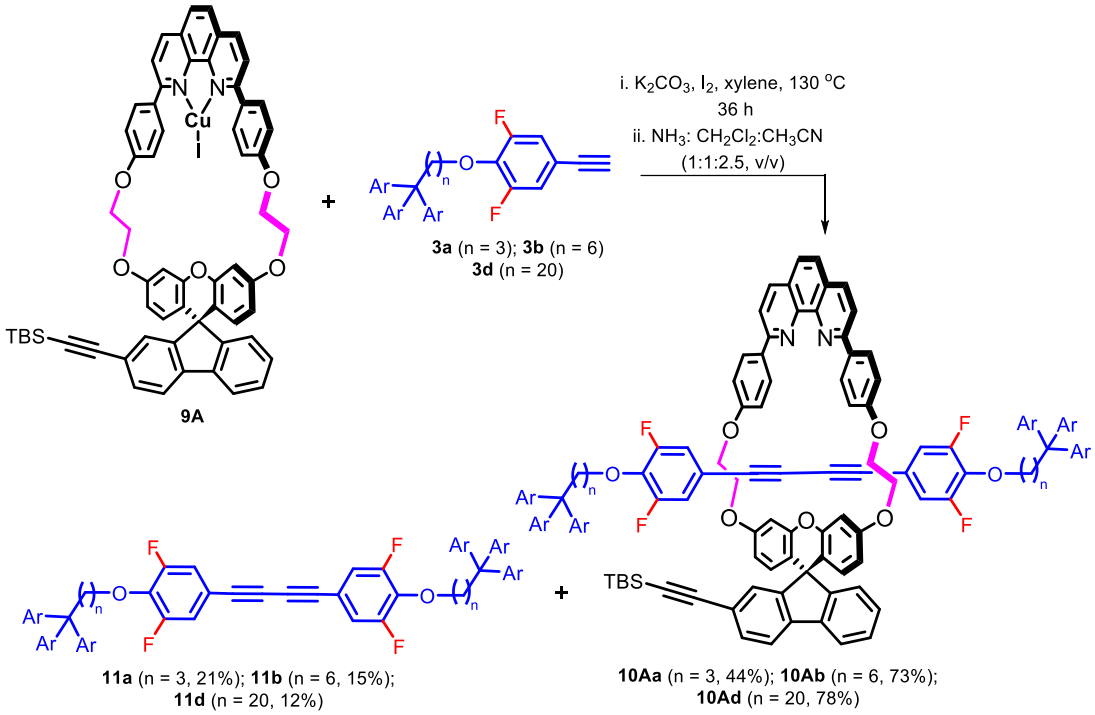


An oven dried sealed tube was charged with macrocyclic Cu-complex **17**, (0.020 g, 0.016 mmol, 1 equiv), terminal alkyne **3c** (0.042 g, 0.040 mmol, 2.5 equiv), finely powdered K_2CO_3 (0.0080 g, 0.060 mmol, 3.8 equiv), I_2 (0.0050 g, 0.020 mmol, 1.3 equiv) and dry xylene (4 mL) as solvent under argon. The reaction mixture was stirred for 36 h at 130 °C and after completion of reaction, aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (4 mL: 4 mL:10 v/v) were added for demetallation. After stirring the mixture at room temperature overnight, extraction was done with CH_2Cl_2 (15 mL × 3), the combined organic layer was washed with water, brine and dried over $MgSO_4$ before solvent was removed under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with EtOAc/hexanes (1:9 v/v) as eluent to furnish the target rotaxane **19** (0.037 g, 0.012 mmol, 74%) along with the dimerized axle component **11c** (0.001 g, 0.005 mmol, 13%, based upon **3c**). Further purification was done by GPC using $CHCl_3$ as mobile phase.

Compound 19: Light yellow solid, mp 187.2−189.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.5 Hz, 2H), 8.11 (d, *J* = 8.0 Hz, 4H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.74 (s, 2H), 7.64

(d, *J* = 8.0 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 12H), 7.45 (d, *J* = 8.5 Hz, 12H), 7.32 (d, *J* = 8.5 Hz, 12H), 7.23 (d, *J* = 9.0 Hz, 16H), 7.07-7.02 (m, 6H), 6.98 (m, 4H), 6.41 (dd, *J* = 9.0, 2.5 Hz, 2H), 6.30 (d, *J* = 8.5 Hz, 2H), 4.45-4.37 (m, 8H), 3.95 (t, *J* = 7.0 Hz, 4H), 2.61-2.56 (m, 4H), 2.53-2.47 (m, 6H), 1.91-1.79 (m, 24H), 1.73 (d, *J* = 12.5 Hz, 6H), 1.62-1.56 (m, 4H), 1.46-1.26 (m, 34H), 1.26-1.10 (m, 32H), 0.87 (s, 18H), 0.03 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) *δ* 159.0, 158.4, 157.3, 155.0 (dd, *J* = 247.9, 6.7 Hz), 154.2, 152.7, 147.0, 146.5, 146.4, 139.1, 138.3, 138.2, 137.4 (t, *J* = 14.4 Hz), 136.4, 133.8, 132.4, 129.6, 128.8, 128.2, 127.5, 127.1, 126.8, 126.2, 125.7, 123.3, 120.2, 120.0, 117.5, 116.9 (m), 115.3 (m), 113.4, 105.8, 102.6, 93.3, 81.1, 75.3, 74.7, 66.5, 66.1, 56.0, 53.8, 44.2, 40.4, 34.4, 30.5, 29.9, 29.8, 29.63, 29.57, 29.3, 26.9, 26.2, 26.1, 25.8, 25.5, 16.6, -4.7 (Some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) *δ* -127.419; HRMS calcd. for $C_{219}H_{231}F_4N_2O_7Si_2$ $[M+H]^+$: 3132.7251, Found: 3132.7286; IR(ATR): 3029, 2925, 2852, 2320, 2144, 1909, 1734, 1606, 1507, 1340, 1246, 1172 cm^{-1}.

General procedure for the synthesis of rotaxanes 10Aa, 10Ab and 10Ad:



A sealed tube was charged with macrocyclic Cu-complex **9A** (1 equiv), terminal alkyne **3** (2.5 equiv), finely powdered K_2CO_3 (3.75 equiv), I_2 (1.25 equiv) and dry xylene (2 mL/0.01 mmol of **9A**) as solvent under argon. The reaction mixture was stirred for 36 h at 130 °C and after completion of reaction, aqueous ammonia (30% solution), CH_2Cl_2 and CH_3CN (1:1:2.5 v/v, 2 mL: 2 mL: 5 mL/0.01 mmol of **9A**) were added for demetallation. After stirring the mixture at room temperature overnight, extraction was done with CH_2Cl_2 (× 3). The combined organic layer was washed with water, brine and dried over $MgSO_4$ before solvent was removed under reduced pressure. The crude reaction mixture was purified by flash silica gel column chromatography with EtOAc/hexanes (1:9 v/v) as eluent to

furnish the target rotaxanes **10Aa** (44%), **10Ab** (73%) and **10Ad** (78%) along with the dimerized axle components (**11a, 11b** and **11d**) as side products. Further purification by GPC using $CHCl_3$ as mobile phase resulted in separation of final compounds.

Compound 10Aa: 0.020 g (0.018 mmol) of **9A** and 0.041 g of **3a** (0.045 mmol) taken and 0.022 g (0.0080 mmol) of **10Aa** and 0.017 g (0.0090 mmol) of **11a** obtained in 44% and 21% (based upon **3a**) yields respectively. Light yellow solid, mp 218.3−220.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (d, J = 8.0 Hz, 2H), 8.10 (d, J = 8.5 Hz, 4H), 7.90 (d, J = 8.5 Hz, 2H), 7.72-7.68 (m, 3H), 7.64 (d, J = 8.0 Hz, 1H), 7.50-7.45 (m, 14H), 7.44-7.40 (m, 12H), 7.36-7.30 (m, 12H), 7.28-7.19 (m, 14H), 7.08-7.00 (m, 8H), 6.80 (d, J = 7.5 Hz, 1H), 6.75-6.68 (m, 2H), 6.38 (dd, J = 8.5, 2.0 Hz, 2H), 6.24 (d, J = 8.5 Hz, 2H), 4.53-4.32 (m, 8H), 4.08 (t, J = 5.5 Hz, 2H), 3.86 (d, J = 5.5 Hz, 2H), 2.82-2.75 (m, 2H), 2.74-2.67 (m, 2H), 2.54-2.46 (m, 6H), 1.86 (dd, J = 25.5, 10.0 Hz, 24H), 1.74 (d, J = 13.0 Hz, 6H), 1.66-1.56 (m, 2H), 1.52-1.32 (m, 26H), 1.29-1.19 (m, 6H), 0.87 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 157.9, 157.1, 155.1 (dd, J = 247.9, 6.7 Hz), 154.64, 154.63 (dd, J = 247.9, 6.8 Hz), 154.0, 152.6, 147.1, 147.0, 146.4, 145.9, 145.8, 139.5, 139.3, 138.5, 138.1, 137.9 (m), 137.6 (m), 136.4, 133.6, 132.1, 129.51, 129.48, 128.6, 128.4, 128.0, 127.4, 127.1, 126.8, 126.3, 125.8, 125.7, 122.7, 120.0, 119.7, 117.9, 117.0 (m), 116.8 (m), 115.1 (m), 113.2, 106.0, 102.5, 92.7, 81.1, 80.7, 75.5, 75.3, 75.2, 75.0, 66.3, 65.8, 55.6, 55.5, 53.7, 44.2, 36.2, 36.1, 34.4, 27.0, 26.9, 26.9, 26.8, 26.7, 26.2, 26.1, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.117 (s, 1F), -127.773 (s, 1F); HRMS calcd. for $C_{193}H_{181}F_4N_2O_7Si$ $[M+H]^+$: 2742.3569, Found: 2742.3562; IR(ATR): 3025, 2924, 2849, 2363, 2148, 1742, 1507, 1348, 1249, 1172 cm^{-1}.

Compound 10Ab: 0.020 g (0.018 mmol) of **9A** and 0.043 g of **3b** (0.045 mmol) taken and 0.037 g (0.013 mmol) of **10Ab** and 0.013 g (0.0070 mmol) of **11b** obtained in 73% and 15% (based upon **3b**) yield respectively. Off-white solid, mp 201.6−203.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19-8.14 (m, 6H), 7.90 (d, J = 8.5 Hz, 2H), 7.68-7.65 (m, 3H), 7.62 (d, J = 8.0 Hz, 1H), 7.52-7.44 (m, 24H), 7.41 (d, J = 8.0 Hz, 1H), 7.33 (t, J = 7.0 Hz, 12H), 7.27-7.22 (m, 14H), 7.16 (d, J = 8.0 Hz, 2H), 7.11-7.04 (m, 6H), 7.00 (t, J = 7.5 Hz, 1H), 6.78 (d, J = 8.0 Hz, 1H), 6.67-6.61 (m, 2H), 6.42 (dd, J = 8.5, 2.0 Hz, 2H), 6.26 (d, J = 9.0 Hz, 2H), 4.52-4.33 (m, 8H), 4.01 (t, J = 6.5 Hz, 2H), 3.79 (t, J = 6.5 Hz, 2H), 2.64-2.47 (m, 10H), 1.86 (dd, J = 26.0, 10.5 Hz, 24H), 1.74 (d, J = 13.0 Hz, 6H), 1.66-1.60 (m, 2H), 1.52-1.33 (m, 32H), 1.32-1.10 (m, 12H), 0.90 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 158.0, 156.8, 155.2 (dd, J = 248.8, 6.7 Hz), 154.7 (dd, J = 247.8, 6.7 Hz), 154.5, 153.7, 152.7, 147.0, 146.3, 139.6, 139.2, 138.4, 138.2, 137.6 (t, J = 14.4 Hz),137.2 (t, J = 14.4 Hz), 136.4, 133.5, 132.1, 129.5, 129.4, 128.6, 128.5, 128.3, 128.0, 127.4, 127.2, 126.8, 126.2, 125.8, 125.6, 122.7, 120.0, 119.8, 118.1, 117.0 (m), 116.8 (m), 115.2 (m), 113.2, 106.2, 102.8, 92.7, 81.3, 80.8, 75.6, 75.0, 74.7, 74.5, 66.6, 66.0, 55.9, 53.7, 44.2, 40.3, 34.4, 30.1, 30.0, 29.9, 29.8, 26.9, 26.2, 26.1, 25.6, 25.5, 25.4, 16.6, -4.6 (some

signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.073, -127.839; HRMS calcd. for $C_{199}H_{193}F_4N_2O_7Si$ $[M+H]^+$: 2826.4508, Found: 2826.4497; IR(ATR): 3029, 2923, 2856, 2320, 2146, 1734, 1603, 1559, 1495, 1335, 1241, 1169 cm^{-1}.

Compound 10Ad: 0.020 g (0.018 mmol) of **9A** and 0.052 g of **3d** (0.045 mmol) taken and 0.045 g (0.014 mmol) of **10Ad** and 0.012 g (0.005 mmol) of **11d** obtained in 78% and 12% (based upon **3d**) yield respectively. White solid, mp 152.5−154.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24-8.18 (m, 6H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.75-7.70 (m, 3H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.52-7.46 (m, 24H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.35 (d *J* = 8.0 Hz, 12H), 7.31-7.27 (m, 2H), 7.26-7.22 (m, 12H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.13-7.03 (m, 8H), 6.85 (d, *J* = 8.0 Hz, 1H), 6.69 (m, 2H), 6.47-6.43 (m, 2H), 6.30 (d, *J* = 9.0 Hz, 2H), 4.55-4.36 (m, 8H), 4.05 (t, *J* = 6.5 Hz, 2H), 3.85 (t, *J* = 6.5 Hz, 2H), 2.64-2.57 (m, 4H), 2.55-2.47 (m, 6H), 1.87 (dd, *J* = 26.5, 10.0 Hz, 24H), 1.74 (d, *J* = 12.5 Hz, 6H), 1.70-1.63 (m, 2H), 1.58-1.52 (m, 2H), 1.48-1.31 (m, 34H), 1.30-1.13 (m, 64H), 0.92 (s, 9H), 0.09 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.2, 158.0, 156.8, 155.2 (dd, *J* = 248.8, 6.7 Hz), 154.8 (dd, *J* = 248.8, 6.8 Hz), 154.4, 153.7, 152.8, 147.0, 146.4, 146.4, 139.6, 139.2, 138.3, 138.2, 137.6 (t, *J* = 14.4 Hz), 137.2 (t, *J* = 14.4 Hz), 136.5, 133.5, 132.2, 129.6, 129.4, 128.6, 128.5, 128.3, 127.9, 127.4, 127.2, 126.8, 126.2, 125.8, 125.7, 122.7, 120.1, 119.8, 118.2, 117.0 (m), 116.8 (m), 115.3 (m), 113.2, 106.0, 102.9, 92.7, 81.3, 80.8, 75.6, 75.0, 74.8, 74.6, 66.6, 66.1, 56.0, 53.8, 44.2, 40.5, 34.4, 30.5, 29.9, 29.7, 29.60, 29.55, 29.5, 29.3, 29.2, 26.9, 26.2, 26.1, 25.7, 25.6, 25.5, 16.6, -4.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.110, -127.846; HRMS calcd. for $C_{227}H_{249}F_4N_2O_7Si$ $[M+H]^+$: 3218.8890, Found: 3218.8854; IR(ATR): 3026, 2924, 2851, 2324, 2146, 1606, 1495, 1340, 1244, 1174 cm^{-1}.

Compound 11a: Light yellow solid, mp 186.5−188.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52-7.47 (m, 24H), 7.40 (d, *J* = 7.0 Hz, 12H), 7.25 (d, *J* = 8.0 Hz, 12H), 7.07-7.01 (m, 4H), 4.20 (t, *J* = 5.0 Hz, 4H), 2.90-2.82 (m, 4H), 2.55-2.46 (m, 6H), 1.86 (dd, *J* = 29.5, 11.0 Hz, 24H), 1.73 (d, *J* = 13.5 Hz, 6H), 1.66-1.58 (m, 4H), 1.48-1.33 (m, 24H), 1.29-1.17 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.2 (dd, *J* = 248.8, 6.7 Hz), 147.1, 145.8, 138.6, 138.1, 137.6 (t, *J* = 14.4 Hz), 129.5, 127.2, 126.8, 126.4, 116.6 (m), 115.3 (t, *J* = 11.5 Hz), 79.8, 75.0, 74.3, 55.6, 44.2, 36.3, 34.4, 26.9, 26.6, 26.2; ^{19}F NMR (377 MHz, $CDCl_3$) δ -126.852; HRMS calcd. for $C_{132}H_{130}F_4NaO_2$ $[M+Na]^+$: 1845.9899, Found: 1845.9834; IR(ATR): 3025, 2928, 2854, 2325, 1908, 1730, 1504, 1349, 1232 cm^{-1}.

Compound 11b: Light yellow solid, mp 166.5−168.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53-7.46 (m, 24H), 7.35 (d, *J* = 9.0 Hz, 12H), 7.27-7.22 (m, 12H), 7.03-6.96 (m, 4H), 4.12 (t, *J* = 6.0 Hz, 4H), 2.64-2.59 (m, 4H), 2.55-2.47 (m, 6H), 1.92-1.80 (m, 24H), 1.77-1.63 (m, 10H), 1.48-1.32 (m, 32H), 1.29-1.13 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4 (dd, *J* = 248.8, 6.7 Hz), 147.1, 146.3, 138.4, 138.2, 137.7 (t, *J* = 14.4 Hz), 129.5, 127.2, 126.8, 126.3, 116.6 (m), 115.4 (t, *J* = 11.5 Hz), 79.8, 74.8, 74.2, 56.0, 44.2, 40.4, 34.4,

30.0, 29.9, 26.9, 26.2, 25.5, 25.4; ^{19}F NMR (377 MHz, $CDCl_3$) δ -126.962; HRMS calcd. for $C_{138}H_{142}F_4NaO_2$ $[M+Na]^+$: 1930.0838, Found: 1930.0803; IR(ATR): 3025, 2924, 2850, 2320, 1909, 1734, 1506, 1341, 1240 cm^{-1}.

Compound 11d: Light yellow solid, mp 107.9−109.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52-7.47 (m, 24H), 7.35 (d, J = 8.5 Hz, 12H), 7.27-7.22 (m, 12H), 7.05-6.99 (m, 4H), 4.15 (t, J = 6.0 Hz, 4H), 2.64-2.57 (m, 4H), 2.55-2.47 (m, 6H), 1.93-1.80 (m, 24H), 1.77-1.69 (m, 10H), 1.49-1.37 (m, 28H), 1.36-1.10 (m, 70H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4 (dd, J = 248.8, 7.7 Hz), 147.0, 146.4, 138.4, 138.2, 137.8 (t, J = 14.4 Hz), 129.6, 127.2, 126.8, 126.2, 116.6 (m), 115.4 (t, J = 11.6 Hz), 79.8, 74.9, 74.2, 56.0, 44.2, 40.5, 34.5, 30.5, 29.9, 29.7, 29.64, 29.56, 29.5, 29.3, 26.9, 26.2, 25.7, 25.6 (some signals are merged); ^{19}F NMR (377 MHz, $CDCl_3$) δ -127.010; HRMS calcd. for $C_{166}H_{198}F_4NaO_2$ $[M+Na]^+$: 2322.5220, Found: 2322.5161; IR(ATR): 3025, 2920, 2848, 2328, 2148, 1905, 1505, 1447, 1340, 1242 cm^{-1}.

References:

1. S. Saito, E. Takahashi, K. Wakatsuki, K. Inoue, T. Orikasa, K. Sakai, R. Yamasaki, Y. Mutoh and T. Kasama, Synthesis of Large [2]Rotaxanes. The Relationship between the Size of the Blocking Group and the Stability of the Rotaxane, *J. Org. Chem.,* 2013, **78**, 3553−3560.
2. Y. Miyake, T. Kinsho, and Y. Nagae, Method for producing reduced halide compound having undergone reduction of carbon-carbon unsaturated bond. EP3556464, 2019.
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4. S. Saito, E. Takahashi, K. Wakatsuki, K. Inoue, T. Orikasa, K. Sakai, R. Yamasaki, Y. Mutoh and T. Kasama, Synthesis of Large [2]Rotaxanes. The Relationship between the Size of the Blocking Group and the Stability of the Rotaxane, *J. Org. Chem.*, 2013, **78**, 3553-3560.
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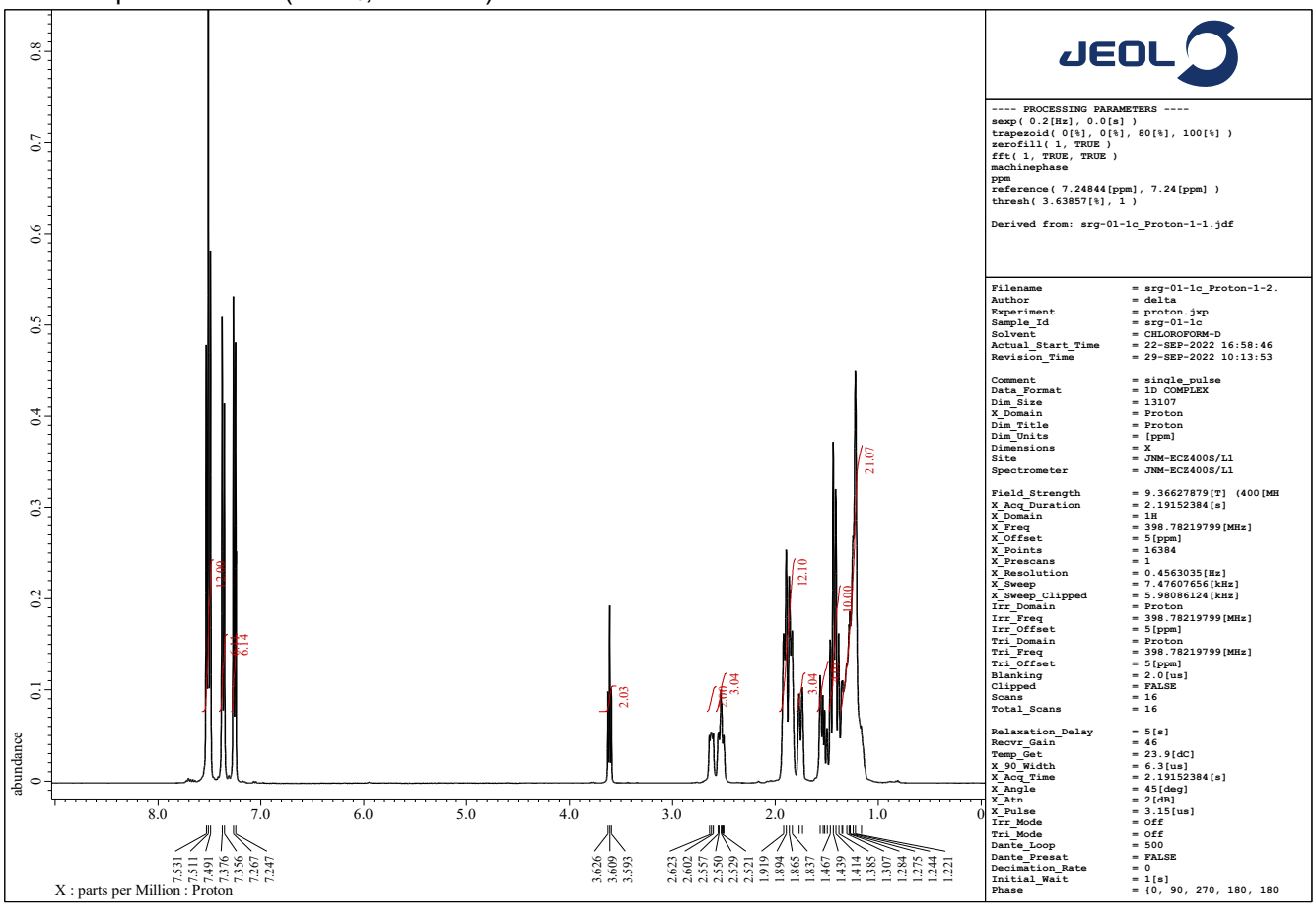
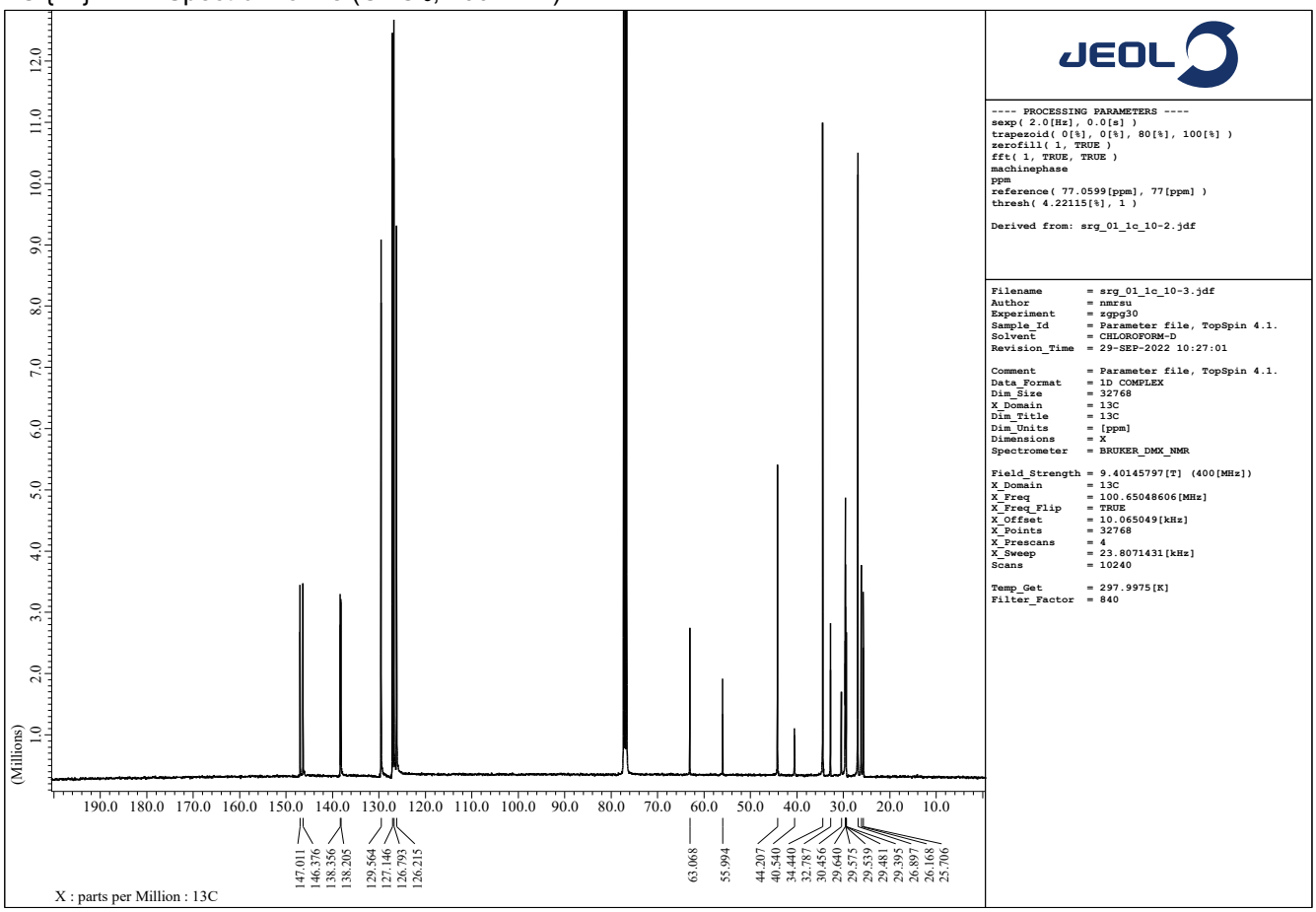
8. C. Dietrich-Buchecker, J. P. Sauvage, Templated synthesis of interlocked macrocyclic ligands, the catenands. Preparation and characterization of the prototypical bis-30 membered ring system, *Tetrahedron,* 1990, **46**, 503−51.
9. S. Zhang, Y. Li, T. Ma, J. Zhao, X. Xu, F. Yang and X.-Y. Xiang, Organosolubility and optical transparency of novel polyimides derived from 2′,7′-bis(4-aminophenoxy)-spiro(fluorene-9,9′-xanthene), *Polym. Chem.*, 2010, **1**, 485-493.
10. Z. Chu, D. Wang, C. Zhang, F. Wang, H. Wu, Z. Lv, S. Hou, X. Fan and D. Zou, Synthesis of spiro[fluorene-9,9'-xanthene] derivatives and their application as hole-transporting materials for organic light-emitting devices, *Synth. Met.*, 2012, **162**, 614-620.

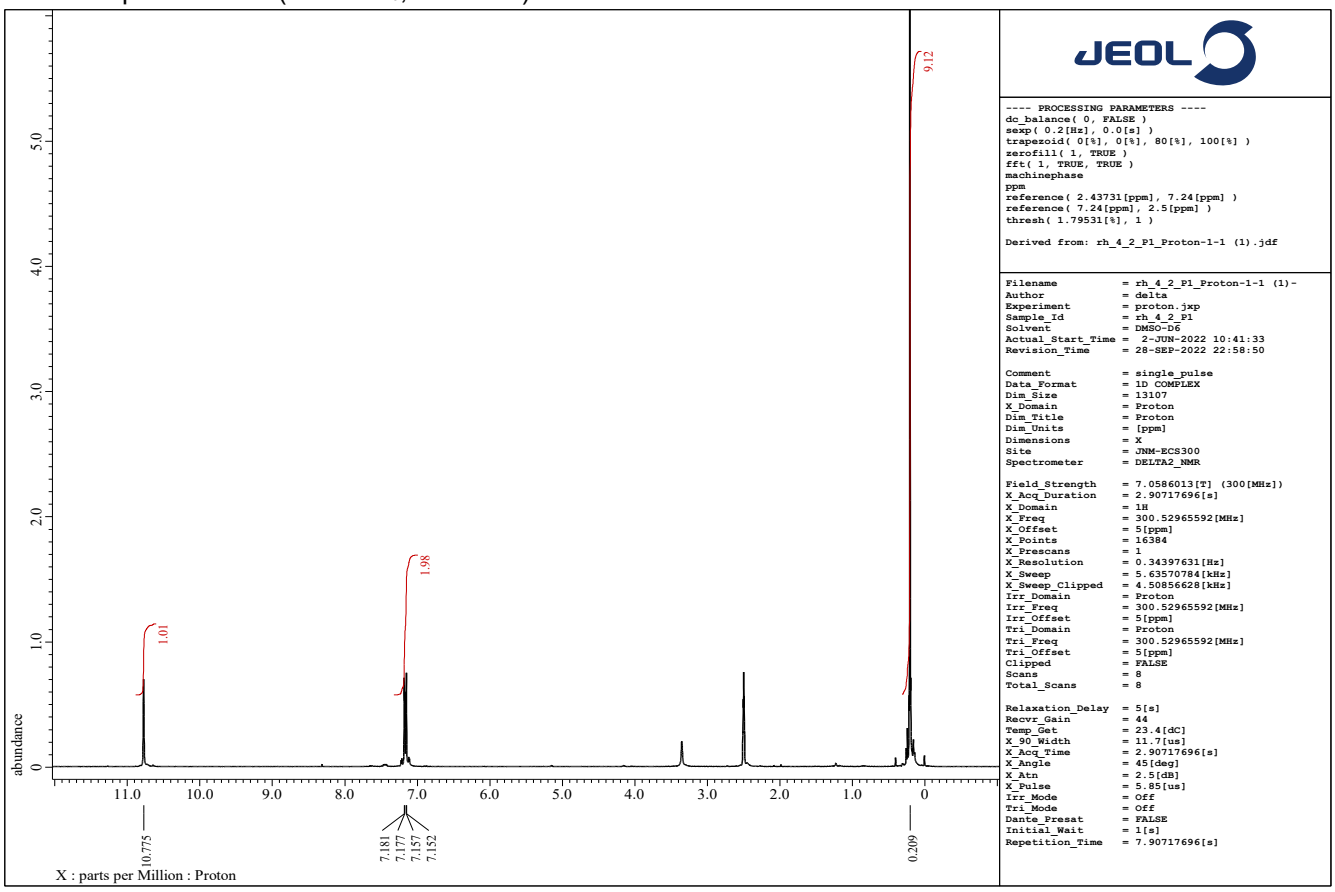
^{1}H NMR Spectrum of **1c** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **1c** ($CDCl_3$, 100 MHz).



^{1}H NMR Spectrum of **2** (DMSO-d_6, 300 MHz).

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trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 2.43731[ppm], 7.24[ppm] )
reference( 7.24[ppm], 2.5[ppm] )
thresh( 1.79531[%], 1 )

Derived from: rh_4_2_P1_Proton-1-1 (1).jdf

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Author              = delta
Experiment          = proton.jxp
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Solvent             = DMSO-D6
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Revision_Time       = 28-SEP-2022 22:58:50

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Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS300
Spectrometer        = DELTA2_NMR

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X_Acq_Duration      = 2.90717696[s]
X_Domain            = 1H
X_Freq              = 300.52965592[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.34397631[Hz]
X_Sweep             = 5.63570784[kHz]
X_Sweep_Clipped     = 4.50856628[kHz]
Irr_Domain          = Proton
Irr_Freq            = 300.52965592[MHz]
Irr_Offset          = 5[ppm]
Tri_Domain          = Proton
Tri_Freq            = 300.52965592[MHz]
Tri_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 8
Total_Scans         = 8

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 23.4[dC]
X_90_Width          = 11.7[us]
X_Acq_Time          = 2.90717696[s]
X_Angle             = 45[deg]
X_Atn               = 2.5[dB]
X_Pulse             = 5.85[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
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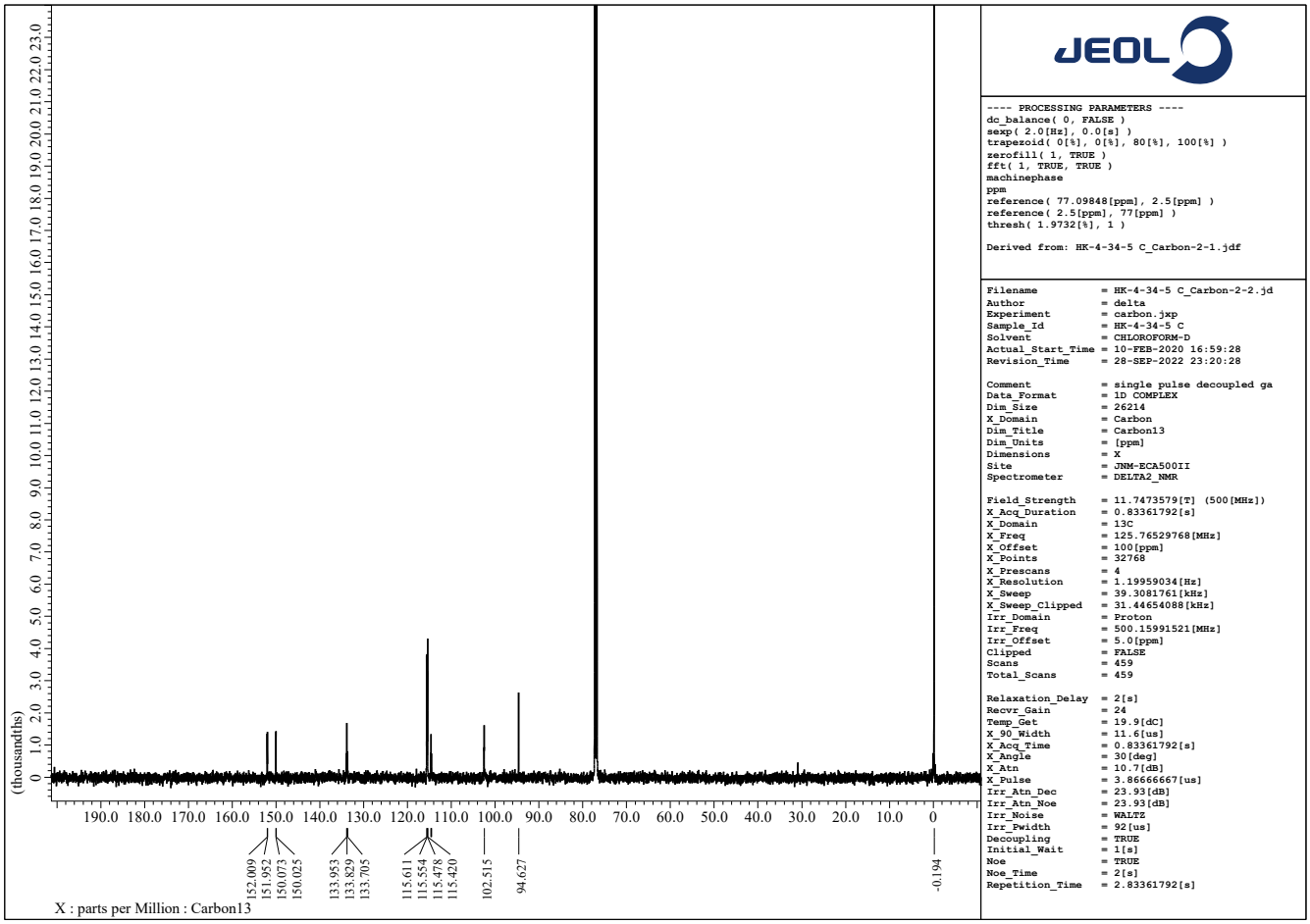
^{13}C {^{1}H} NMR Spectrum of **2** ($CDCl_3$, 125 MHz).

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sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.09848[ppm], 2.5[ppm] )
reference( 2.5[ppm], 77[ppm] )
thresh( 1.9732[%], 1 )

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Author              = delta
Experiment          = carbon.jxp
Sample_Id           = HK-4-34-5 C
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Actual_Start_Time   = 10-FEB-2020 16:59:28
Revision_Time       = 28-SEP-2022 23:20:28

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Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 459
Total_Scans         = 459

Relaxation_Delay    = 2[s]
Recvr_Gain          = 24
Temp_Get            = 19.9[dC]
X_90_Width          = 11.6[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.86666667[us]
Irr_Atn_Dec         = 23.93[dB]
Irr_Atn_Noe         = 23.93[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
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Repetition_Time     = 2.83361792[s]
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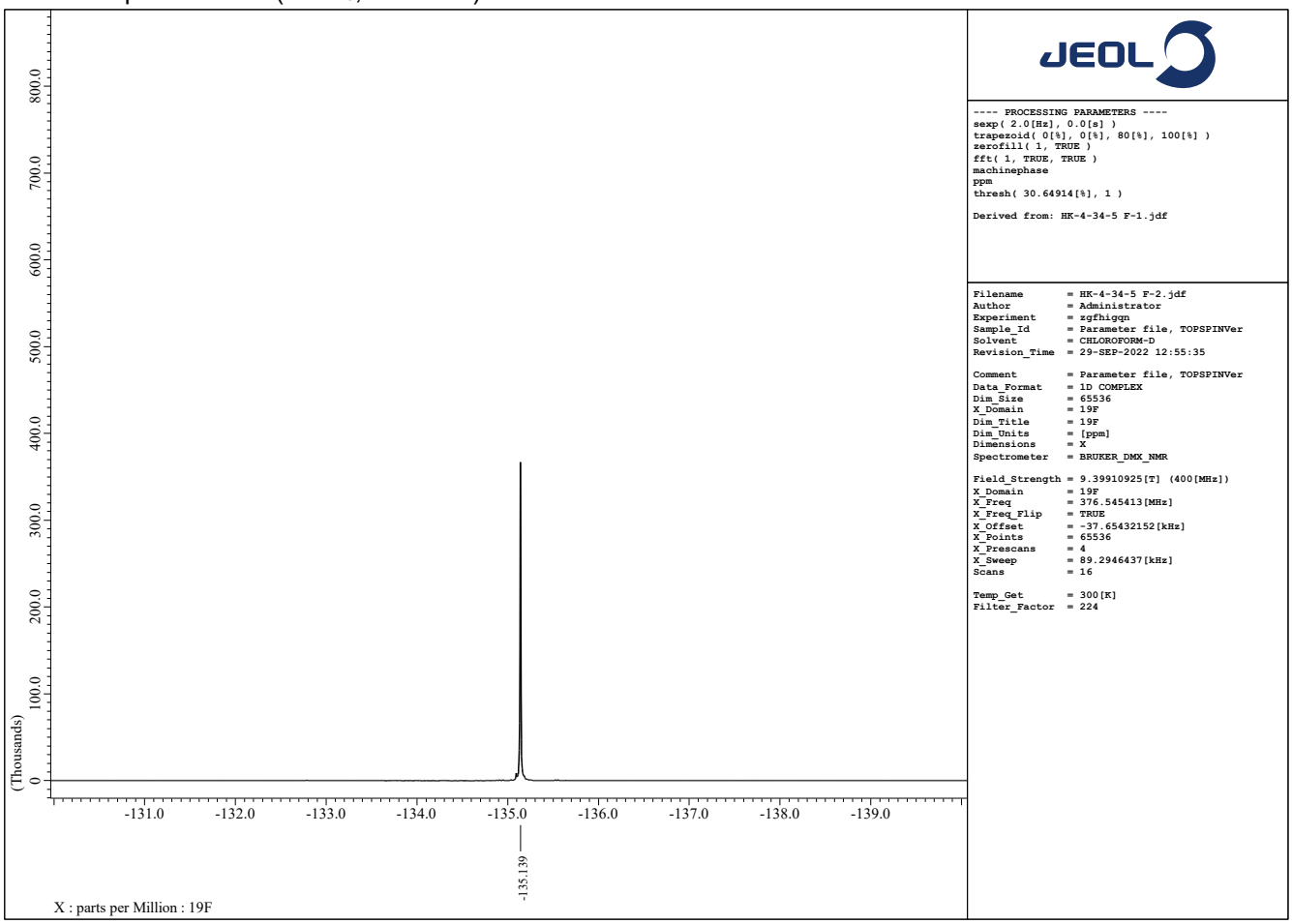
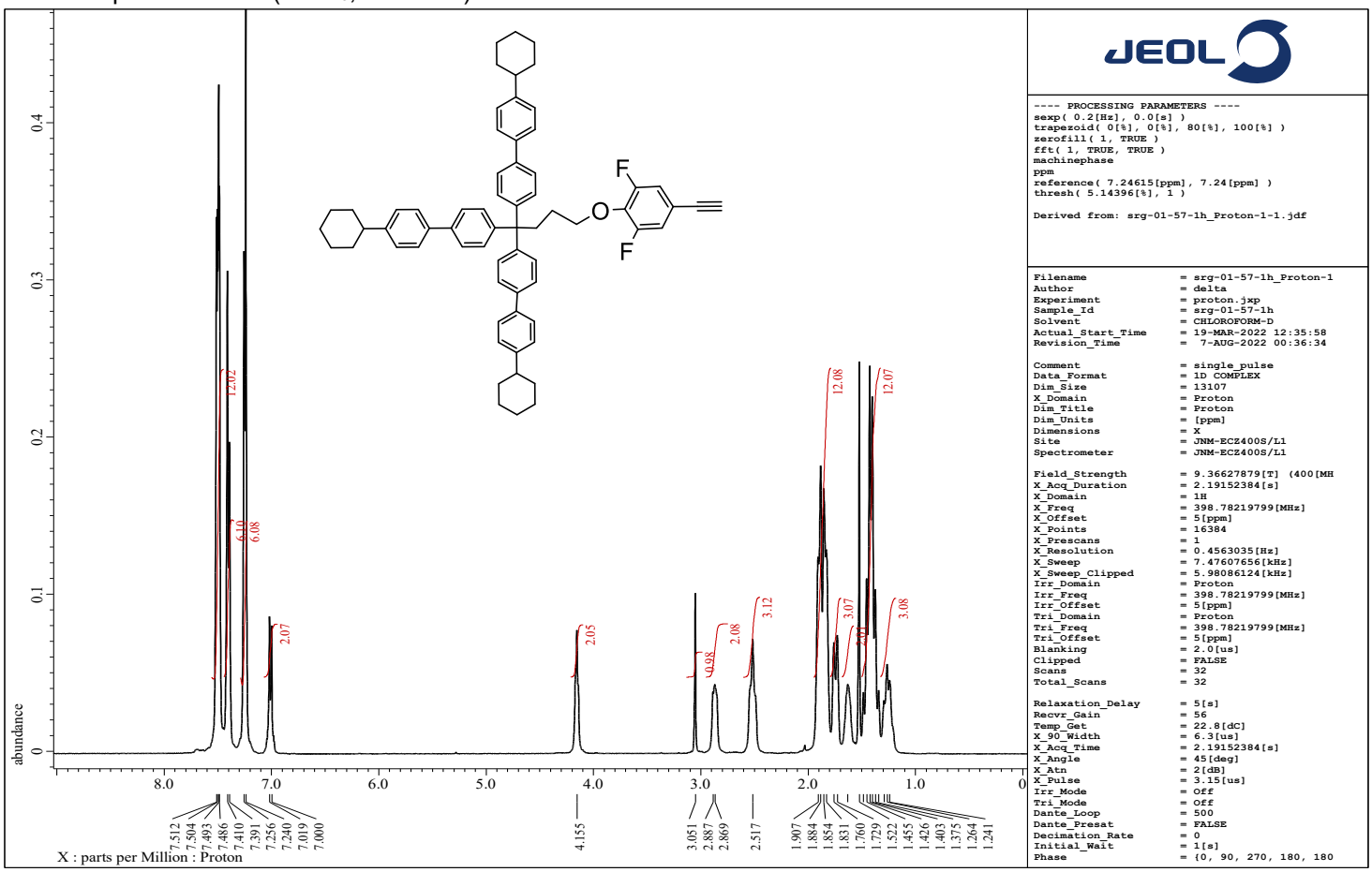
^{19}F NMR Spectrum of **2** ($CDCl_3$, 377 MHz).



^{1}H NMR Spectrum of **3a** ($CDCl_3$, 400 MHz).

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---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
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zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24615[ppm], 7.24[ppm] )
thresh( 5.14396[%], 1 )

Derived from: srg-01-57-1h_Proton-1-1.jdf
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Author	= delta
Experiment	= proton.jxp
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Solvent	= CHLOROFORM-D
Actual_Start_Time	= 19-MAR-2022 12:35:58
Revision_Time	= 7-AUG-2022 00:36:34
Comment	= single_pulse
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Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 22.8[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
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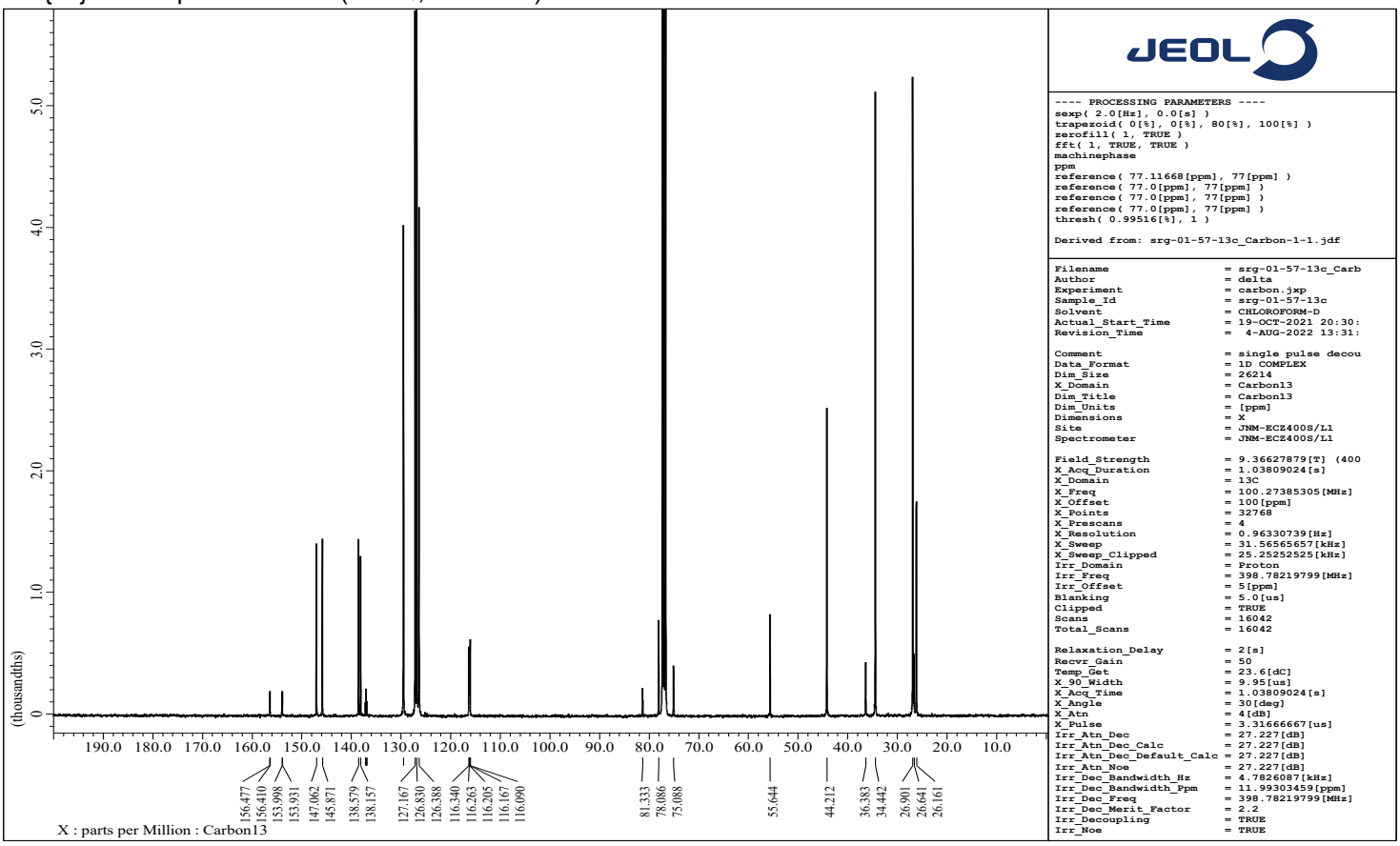
Phase	= {0, 90, 270, 180, 180

^{13}C {^{1}H} NMR Spectrum of **3a** ($CDCl_3$, 100 MHz).

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reference( 77.11668[ppm], 77[ppm] )
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thresh( 0.99516[%], 1 )

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Revision_Time	= 4-AUG-2022 13:31:
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Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
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Spectrometer	= JNM-ECZ400S/L1
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Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
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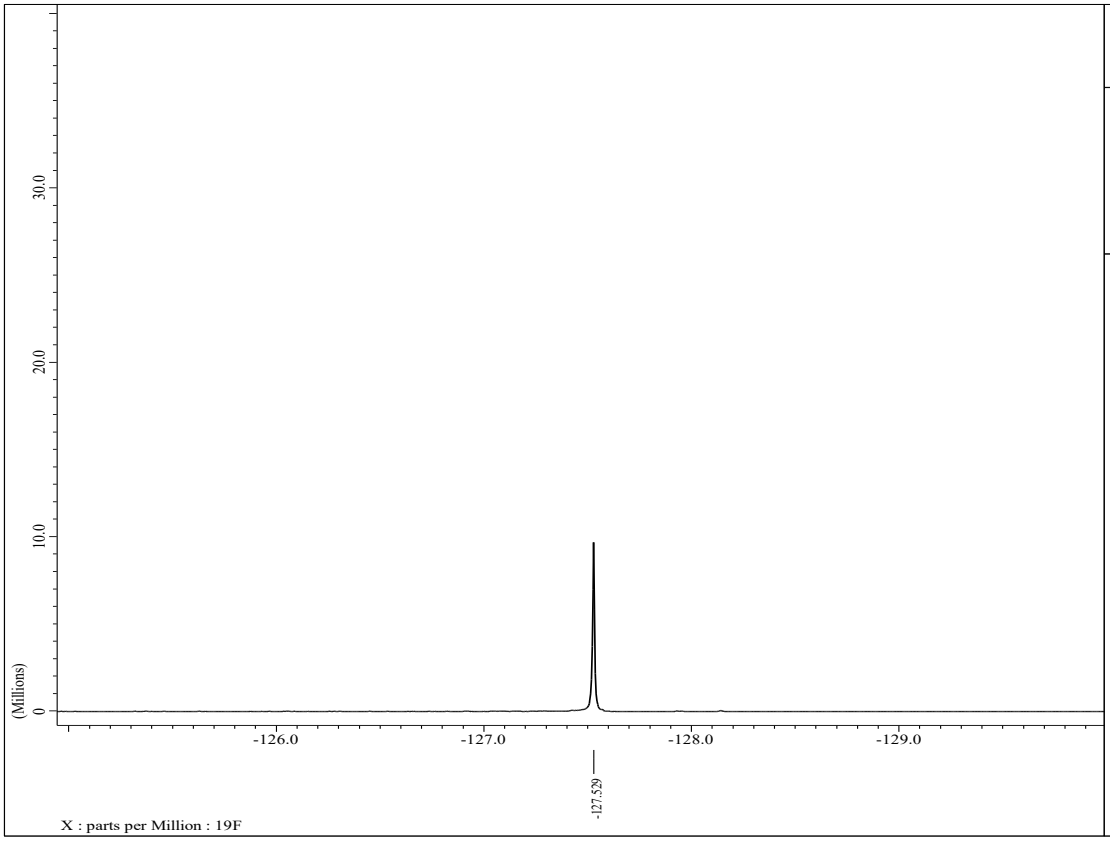
^{19}F NMR Spectrum of **3a** ($CDCl_3$, 377 MHz).

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Revision_Time   = 19-MAR-2022 16:20:30

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Filter_Factor   = 220
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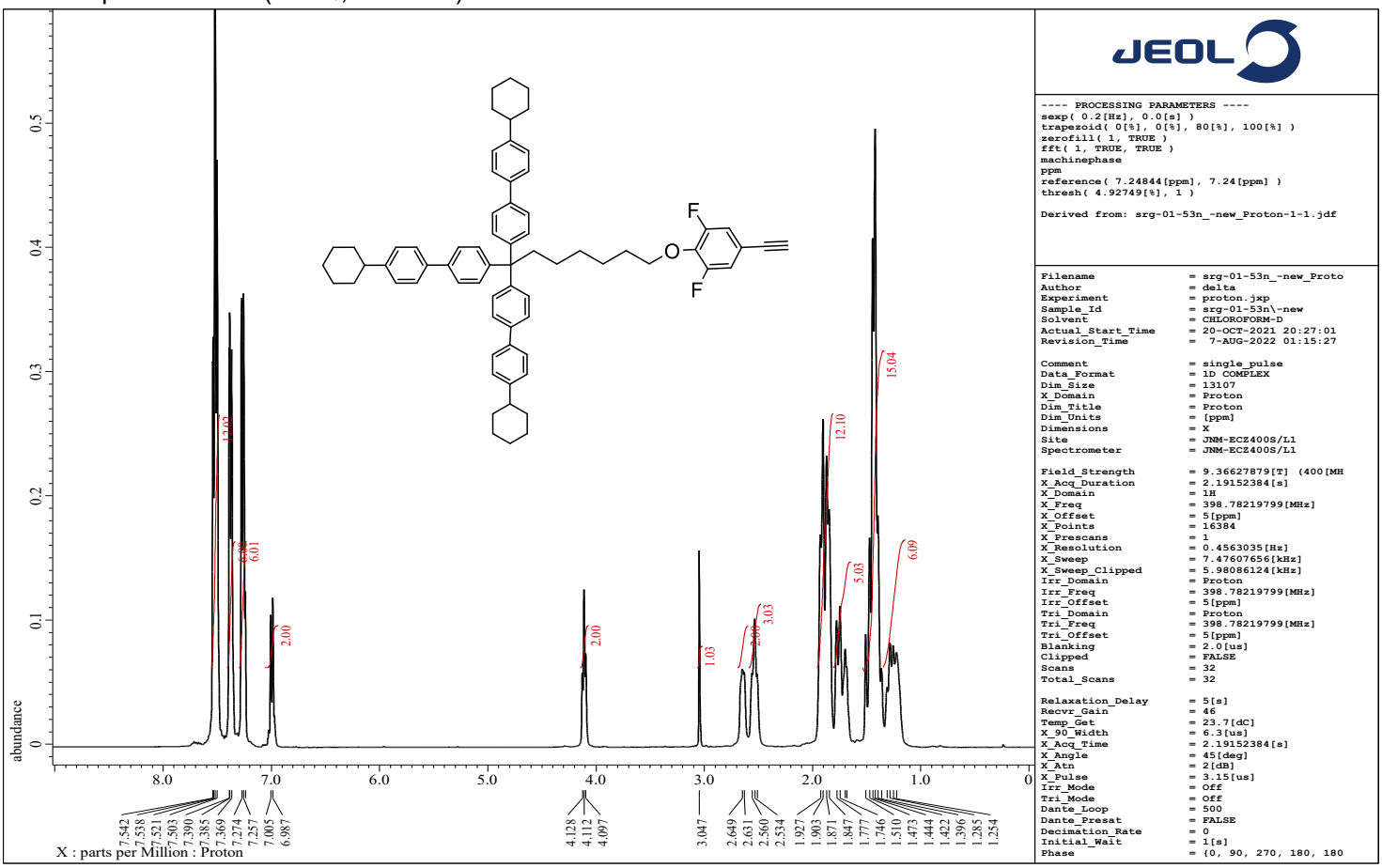
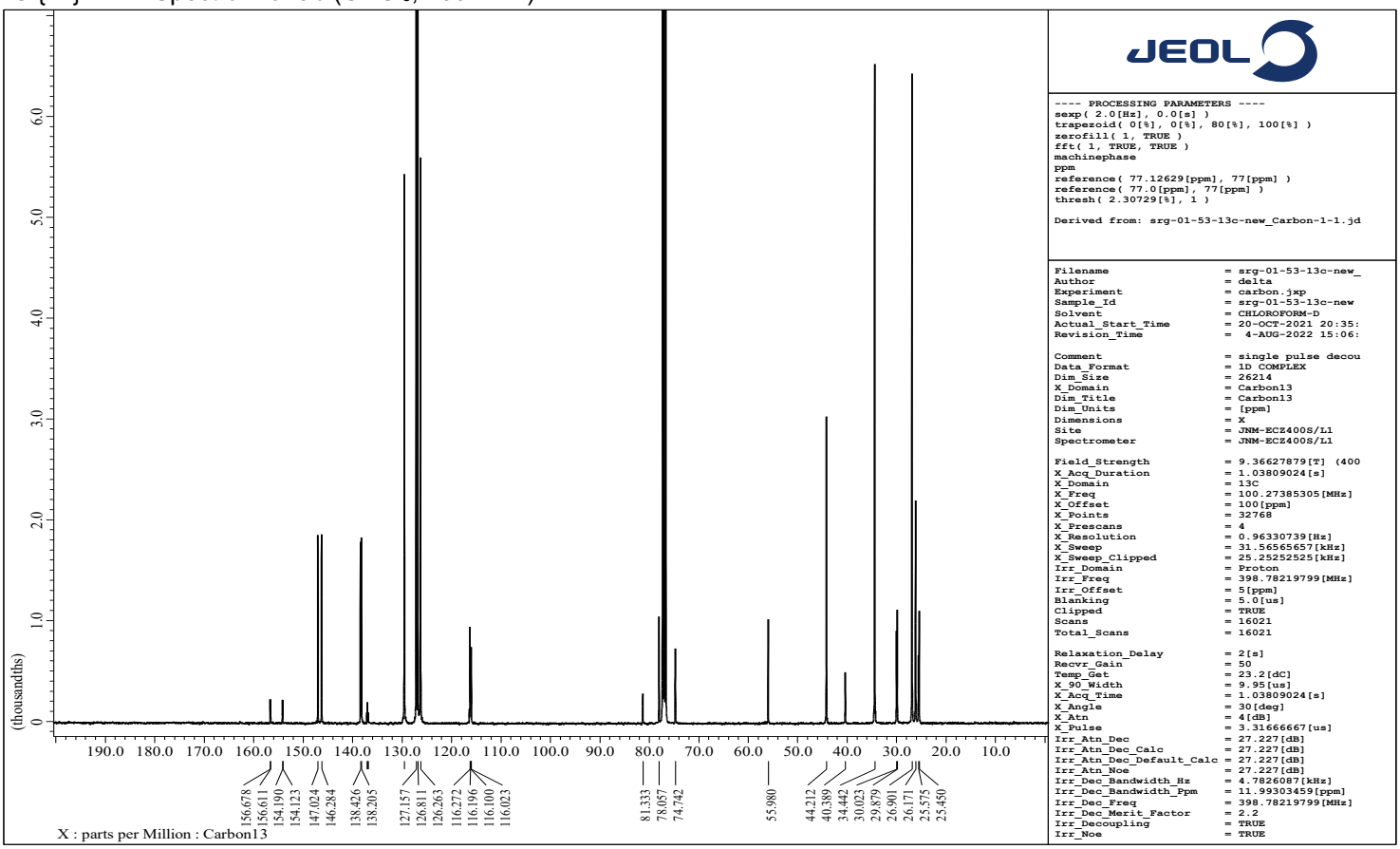
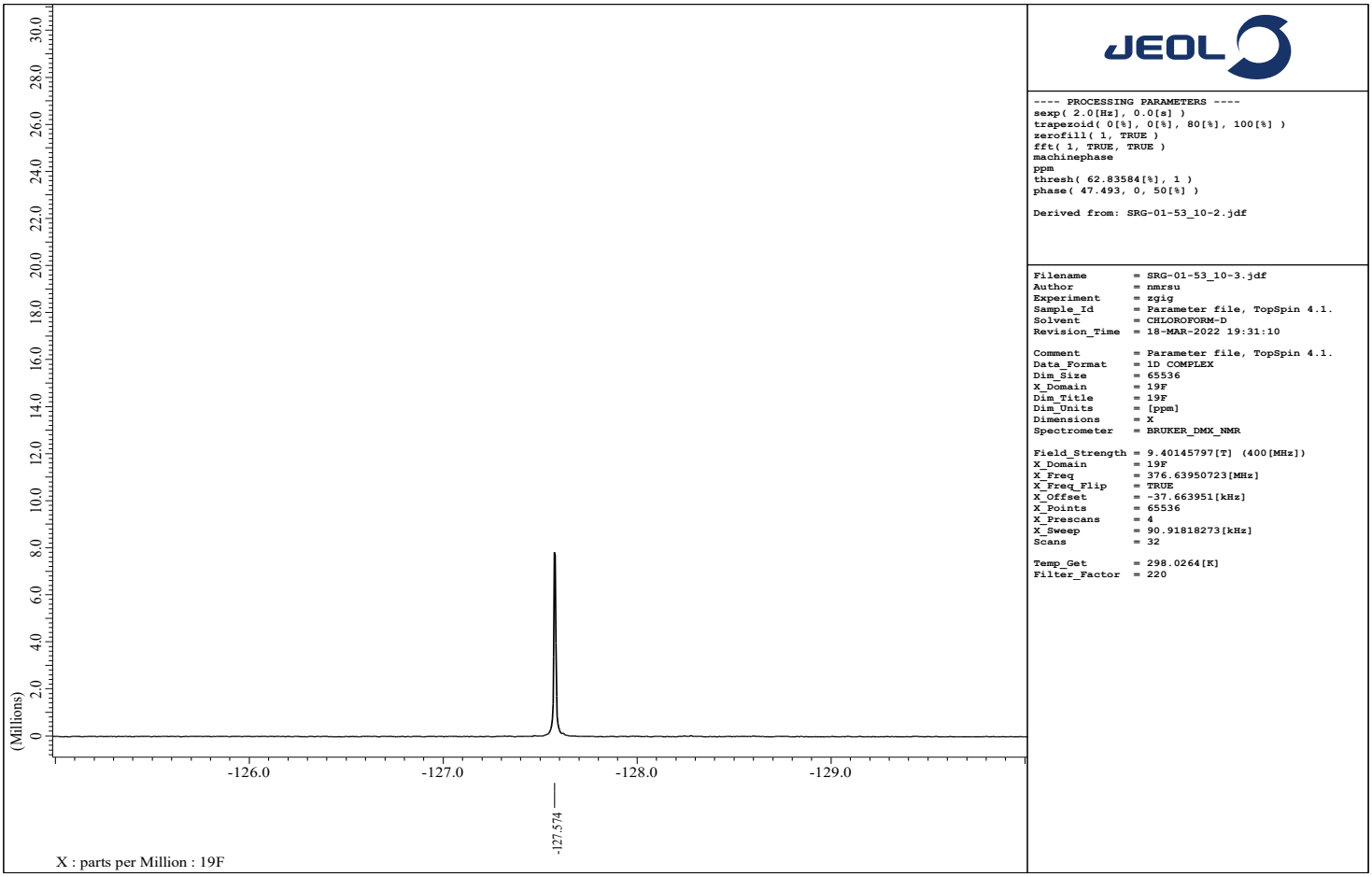
^{1}H NMR Spectrum of **3b** ($CDCl_3$, 400 MHz).



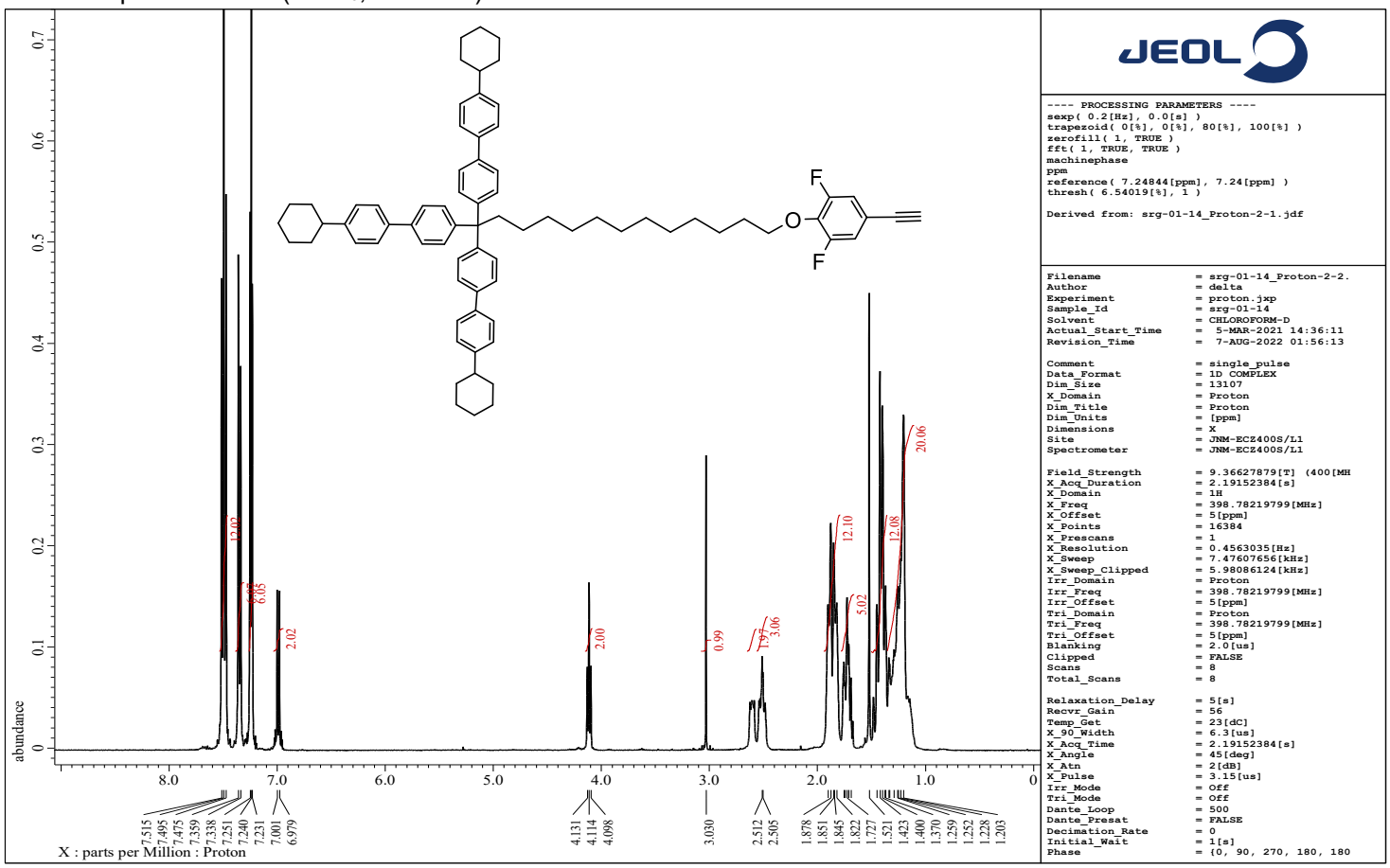
^{13}C {^{1}H} NMR Spectrum of **3b** ($CDCl_3$, 100 MHz).



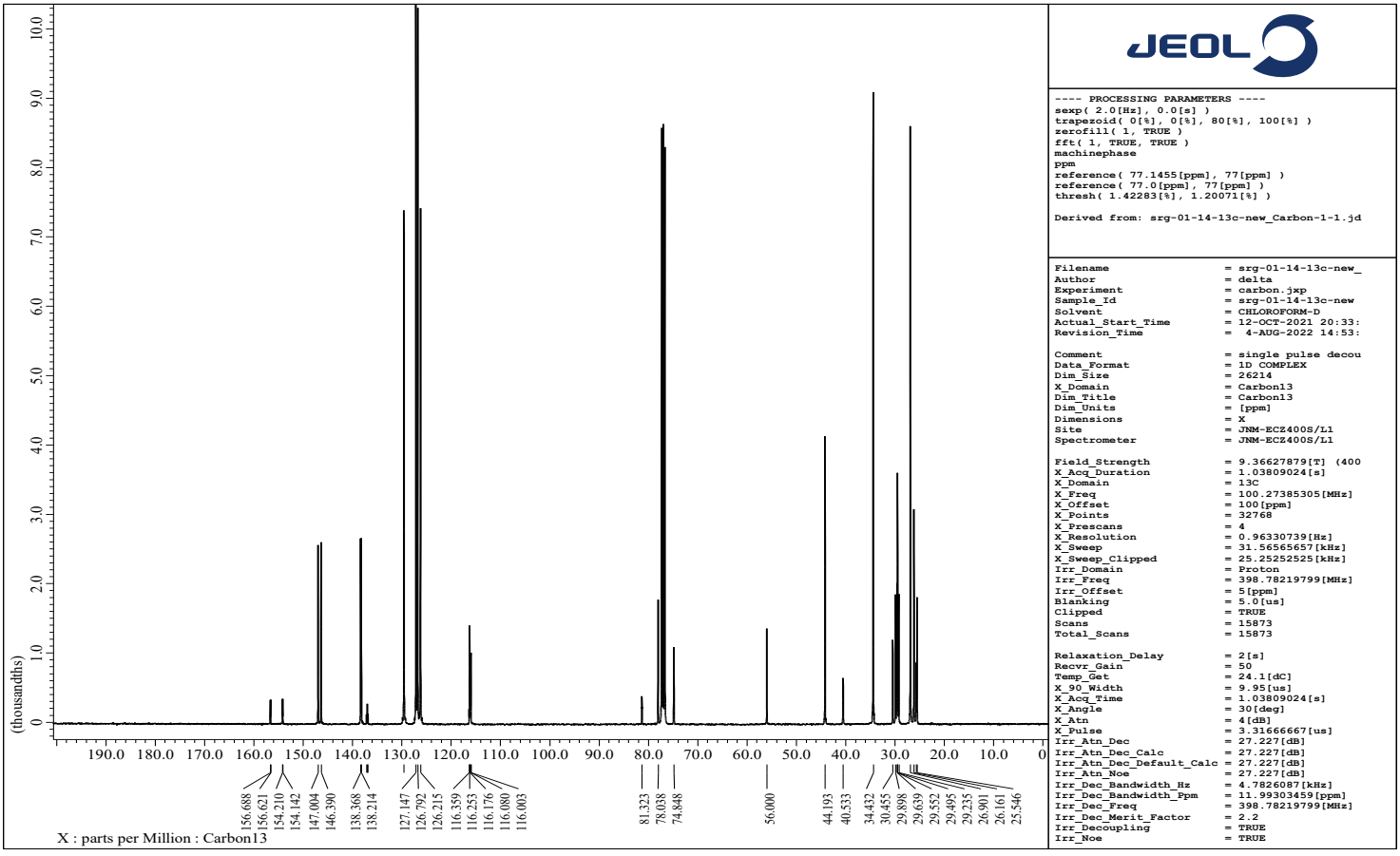
^{19}F NMR Spectrum of **3b** ($CDCl_3$, 377 MHz).



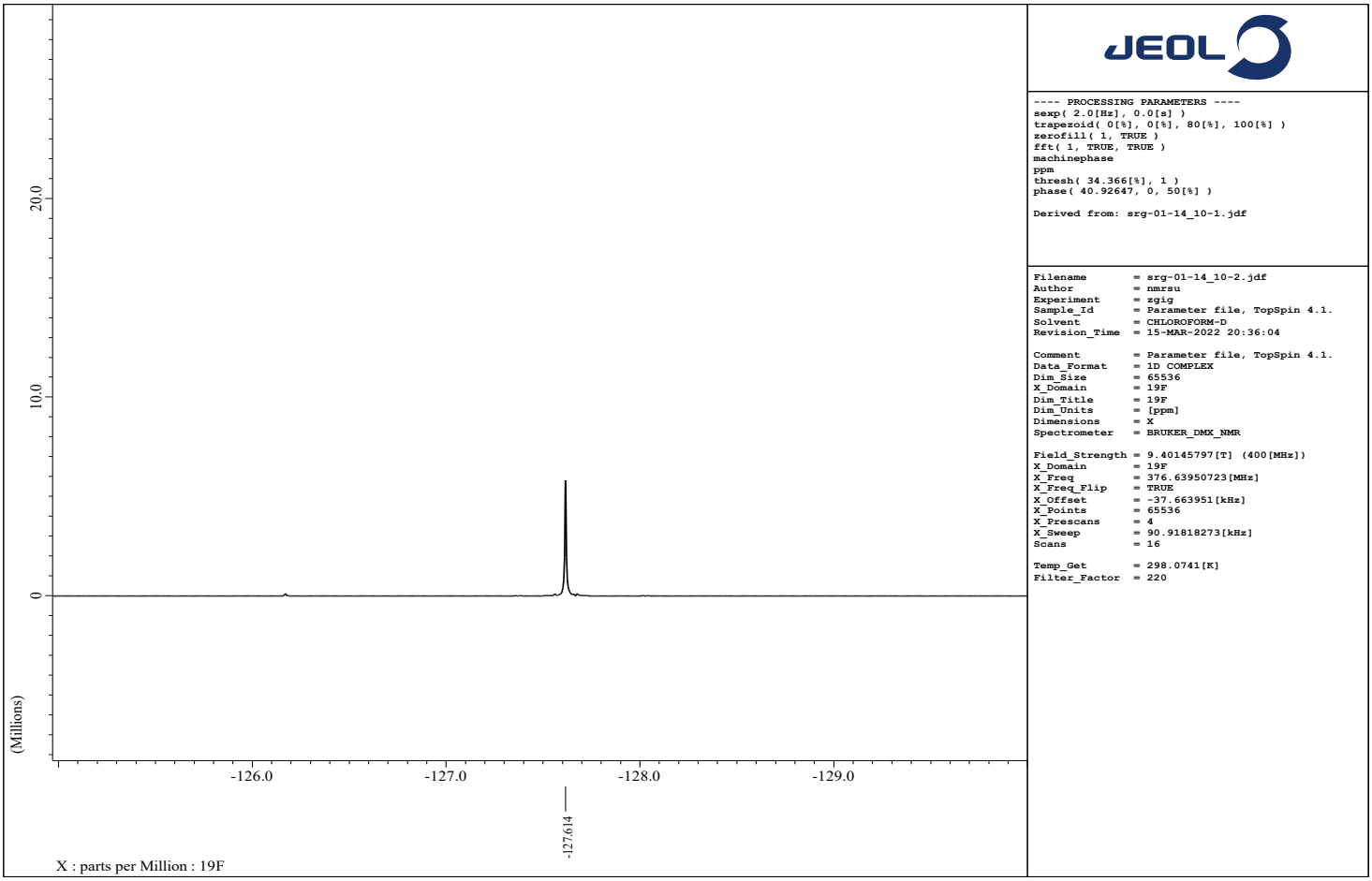
^{1}H NMR Spectrum of **3c** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **3c** ($CDCl_3$, 100 MHz).



^{19}F NMR Spectrum of **3c** ($CDCl_3$, 377 MHz).

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Dimensions      = X
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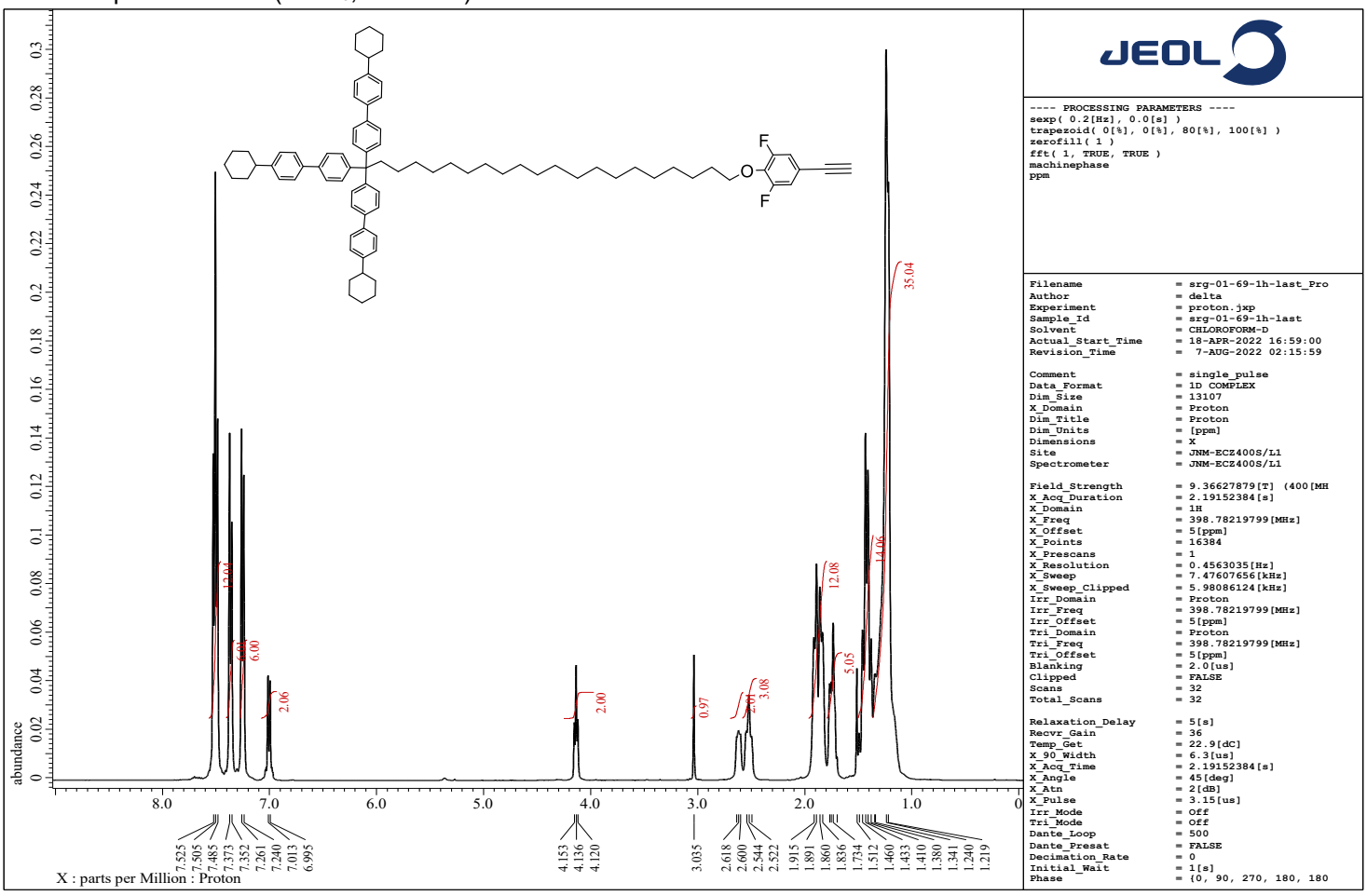
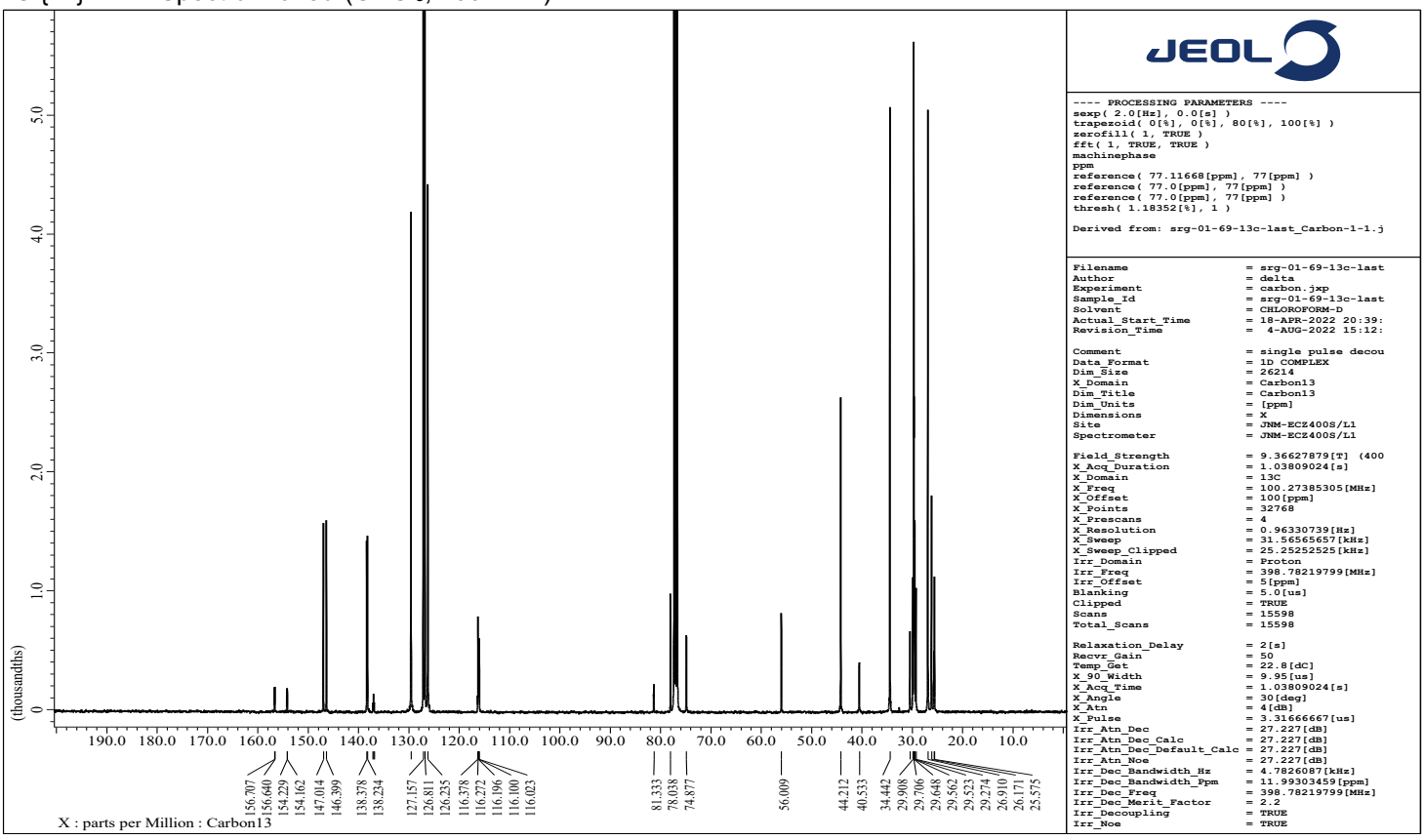
Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 16

Temp_Get        = 298.0741[K]
Filter_Factor   = 220
```

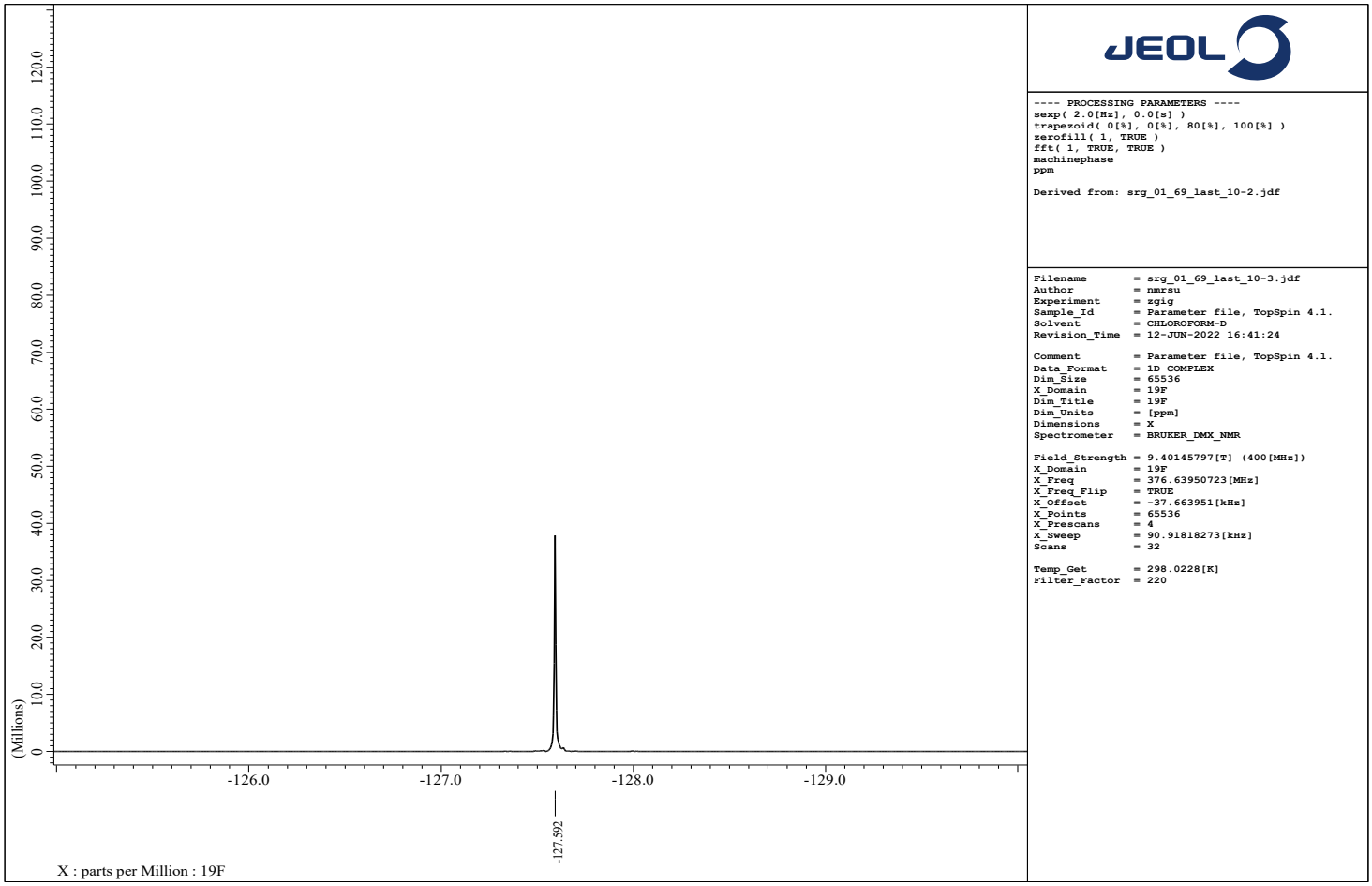
^{1}H NMR Spectrum of **3d** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **3d** ($CDCl_3$, 100 MHz).



^{19}F NMR Spectrum of **3d** ($CDCl_3$, 377 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: srg_01_69_last_10-2.jdf
```

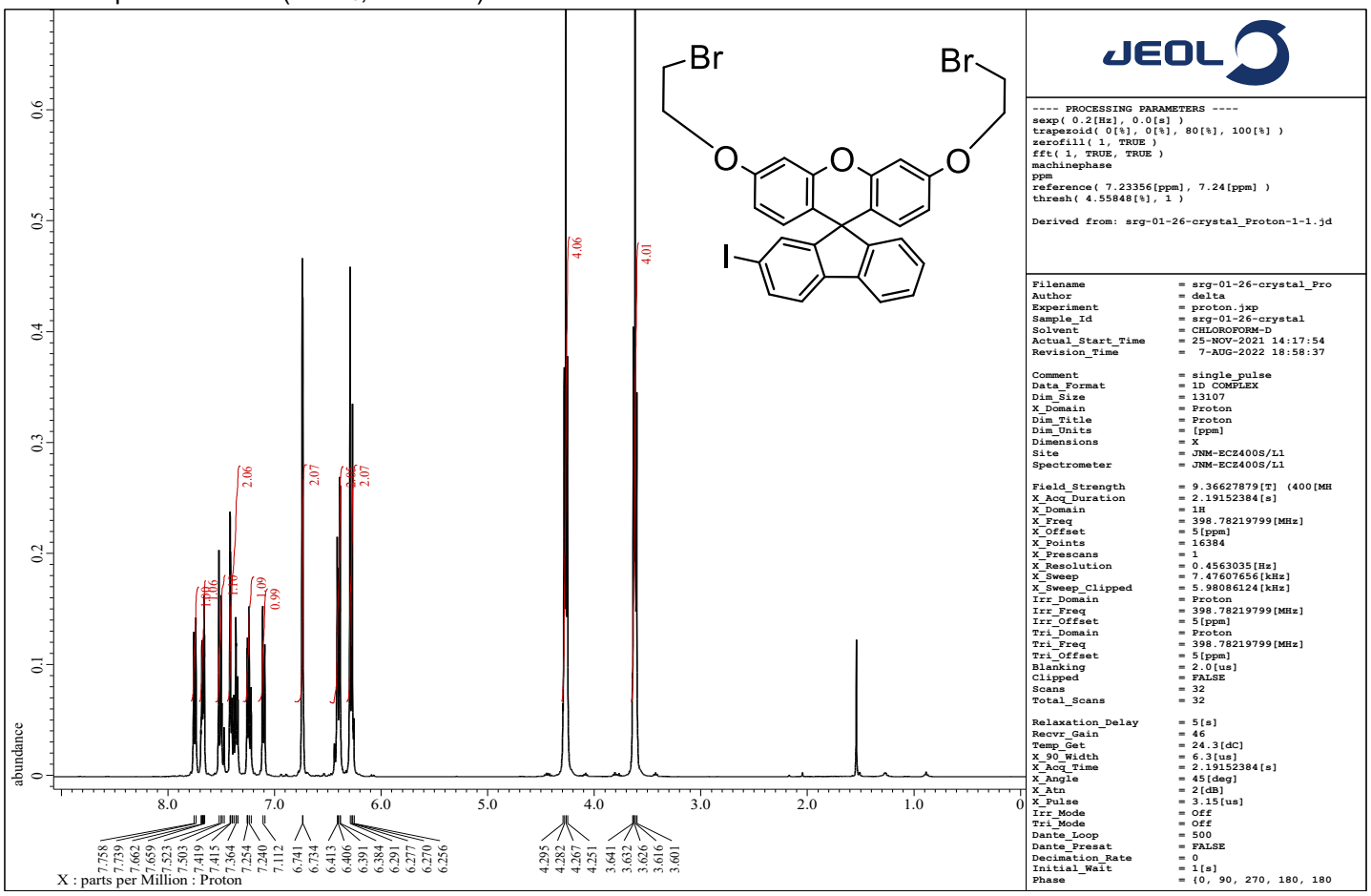
```
Filename        = srg_01_69_last_10-3.jdf
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 12-JUN-2022 16:41:24

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 32

Temp_Get        = 298.0228[K]
Filter_Factor   = 220
```

^{1}H NMR Spectrum of **5A** ($CDCl_3$, 400 MHz).

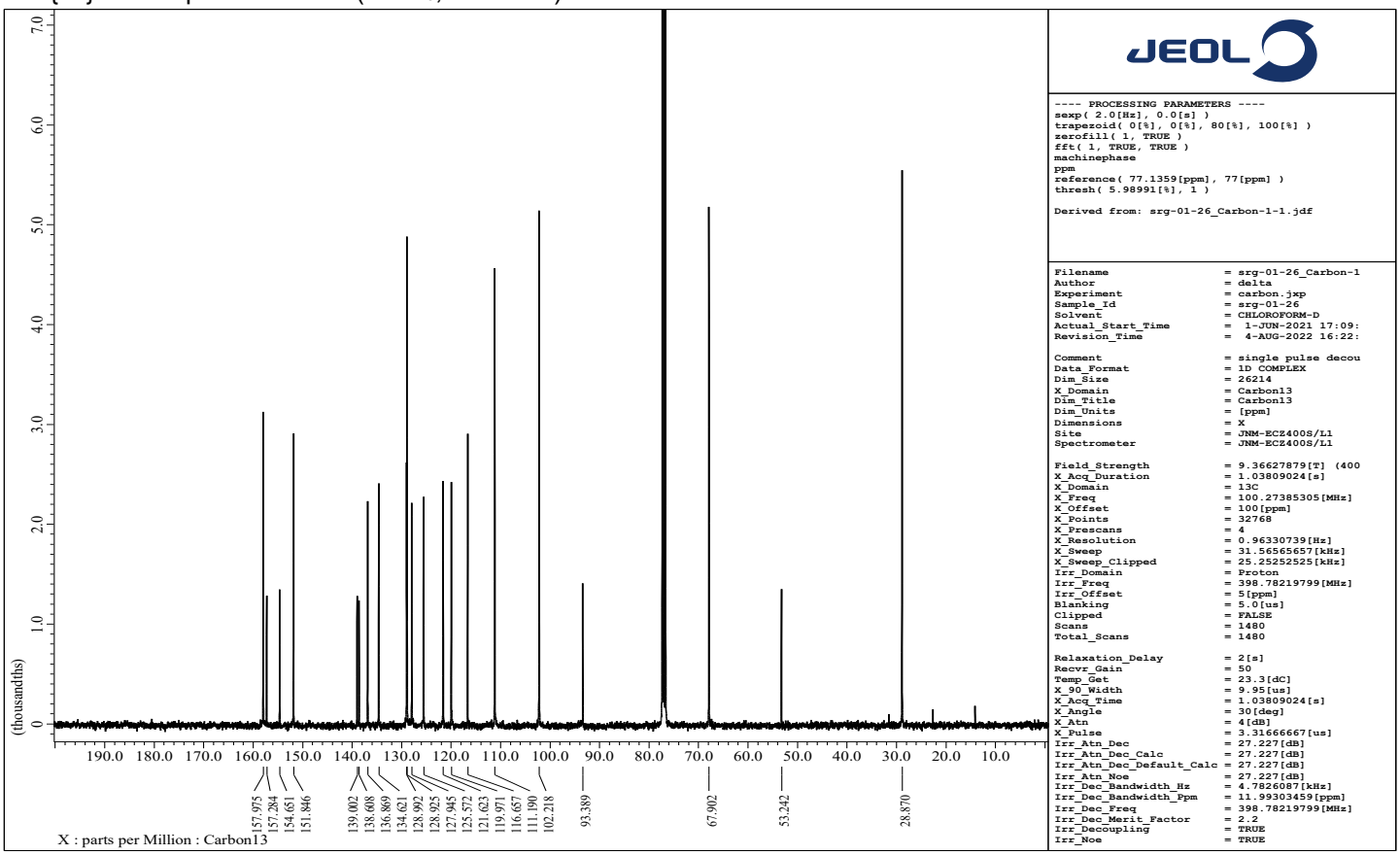



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.23356[ppm], 7.24[ppm] )
thresh( 4.55848[%], 1 )

Derived from: srg-01-26-crystal_Proton-1-1.jd
```

Filename	= srg-01-26-crystal_Pro
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-26-crystal
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-NOV-2021 14:17:54
Revision_Time	= 7-AUG-2022 18:58:37
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 24.3[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

^{13}C {^{1}H} NMR Spectrum of **5A** ($CDCl_3$, 100 MHz).

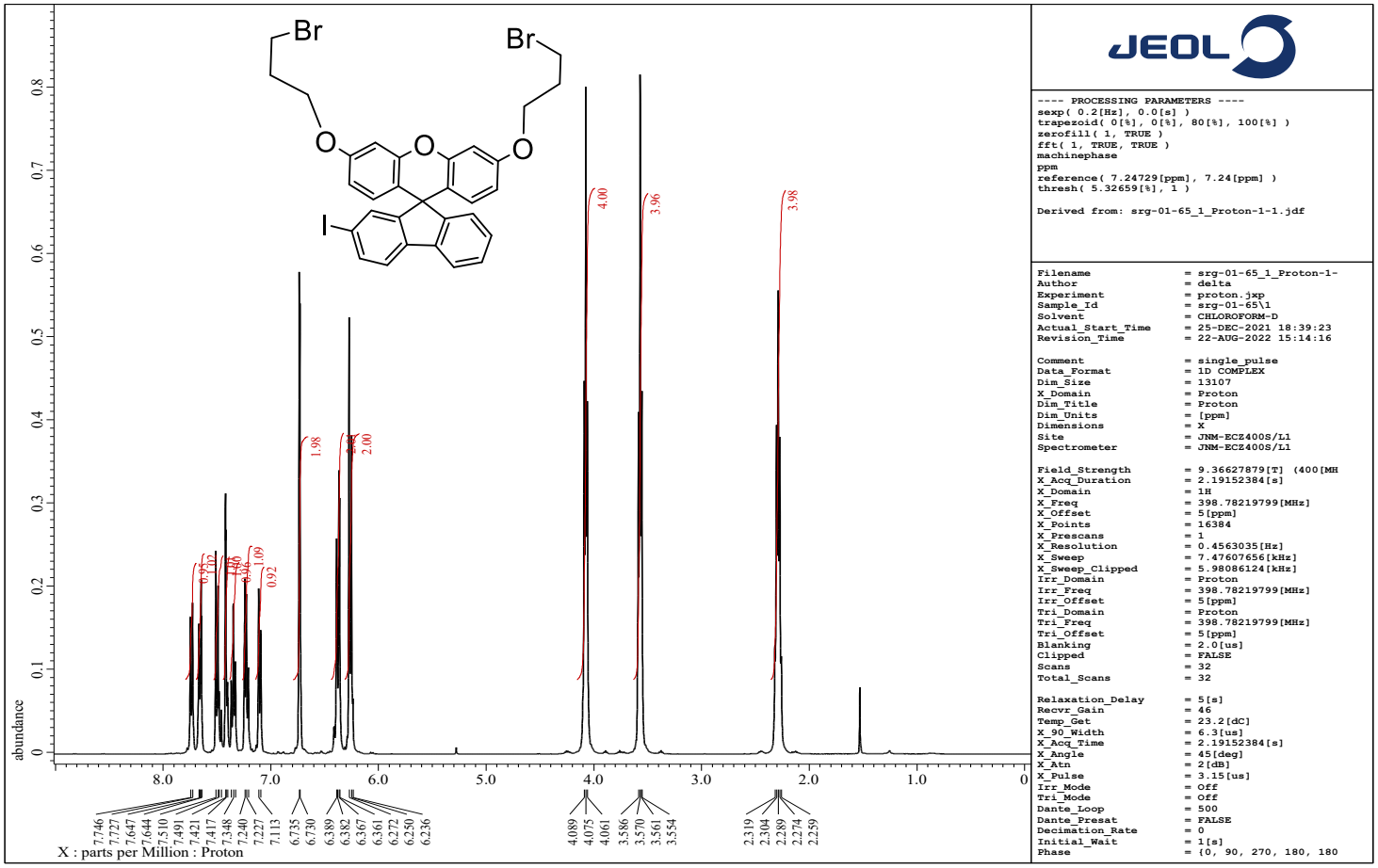



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.1359[ppm], 77[ppm] )
thresh( 5.98991[%], 1 )

Derived from: srg-01-26_Carbon-1-1.jdf
```

Filename	= srg-01-26_Carbon-1
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-26
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-JUN-2021 17:09:
Revision_Time	= 4-AUG-2022 16:22:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 1480
Total_Scans	= 1480
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.3[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

^{1}H NMR Spectrum of **5B** ($CDCl_3$, 400 MHz).

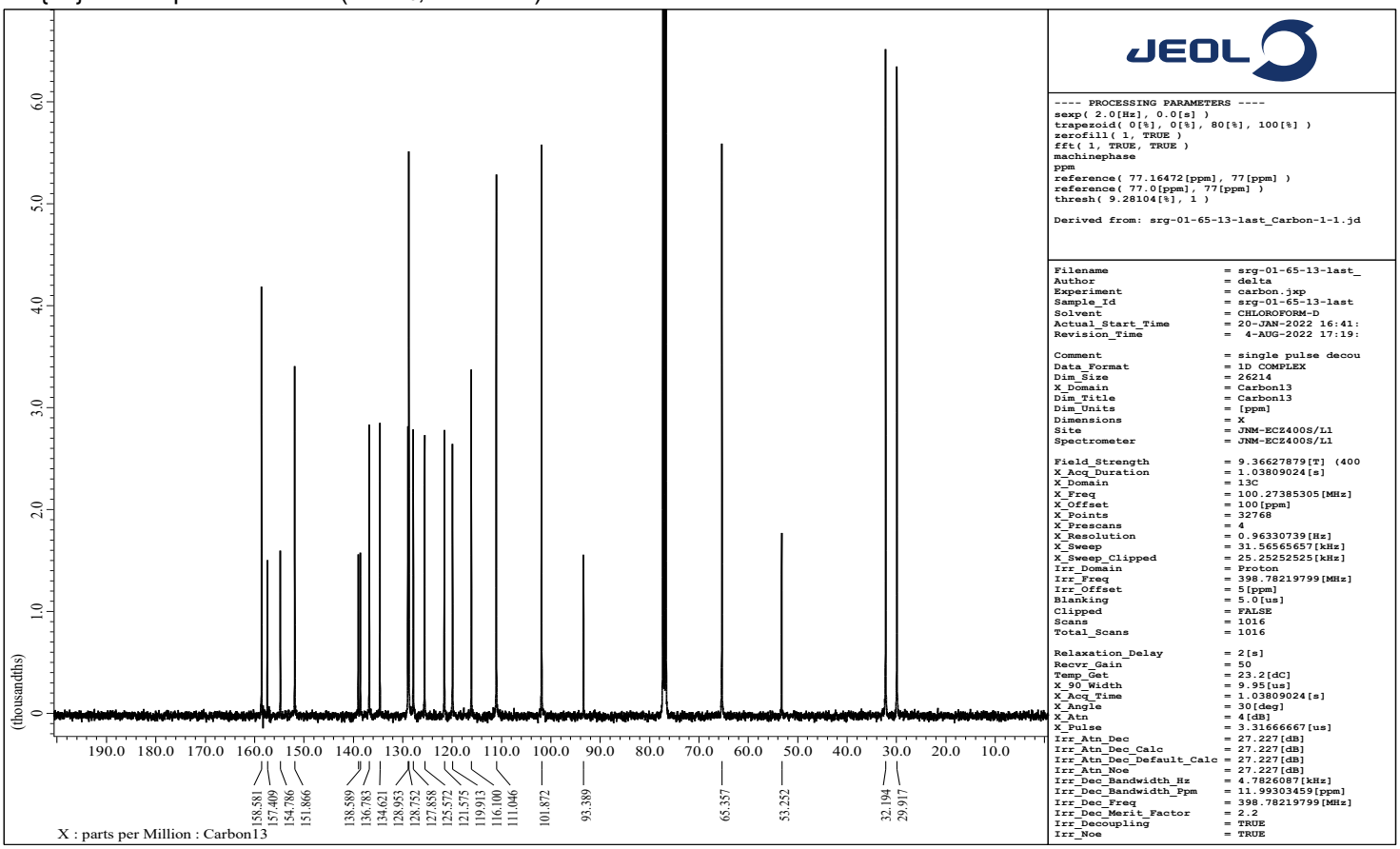



```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24729[ppm], 7.24[ppm] )
thresh( 5.32659[%], 1 )

Derived from: srg-01-65_1_Proton-1-1.jdf
```

Filename	= srg-01-65_1_Proton-1-
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-65\1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-DEC-2021 18:39:23
Revision_Time	= 22-AUG-2022 15:14:16
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 23.2[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

^{13}C {^{1}H} NMR Spectrum of **5B** ($CDCl_3$, 100 MHz).

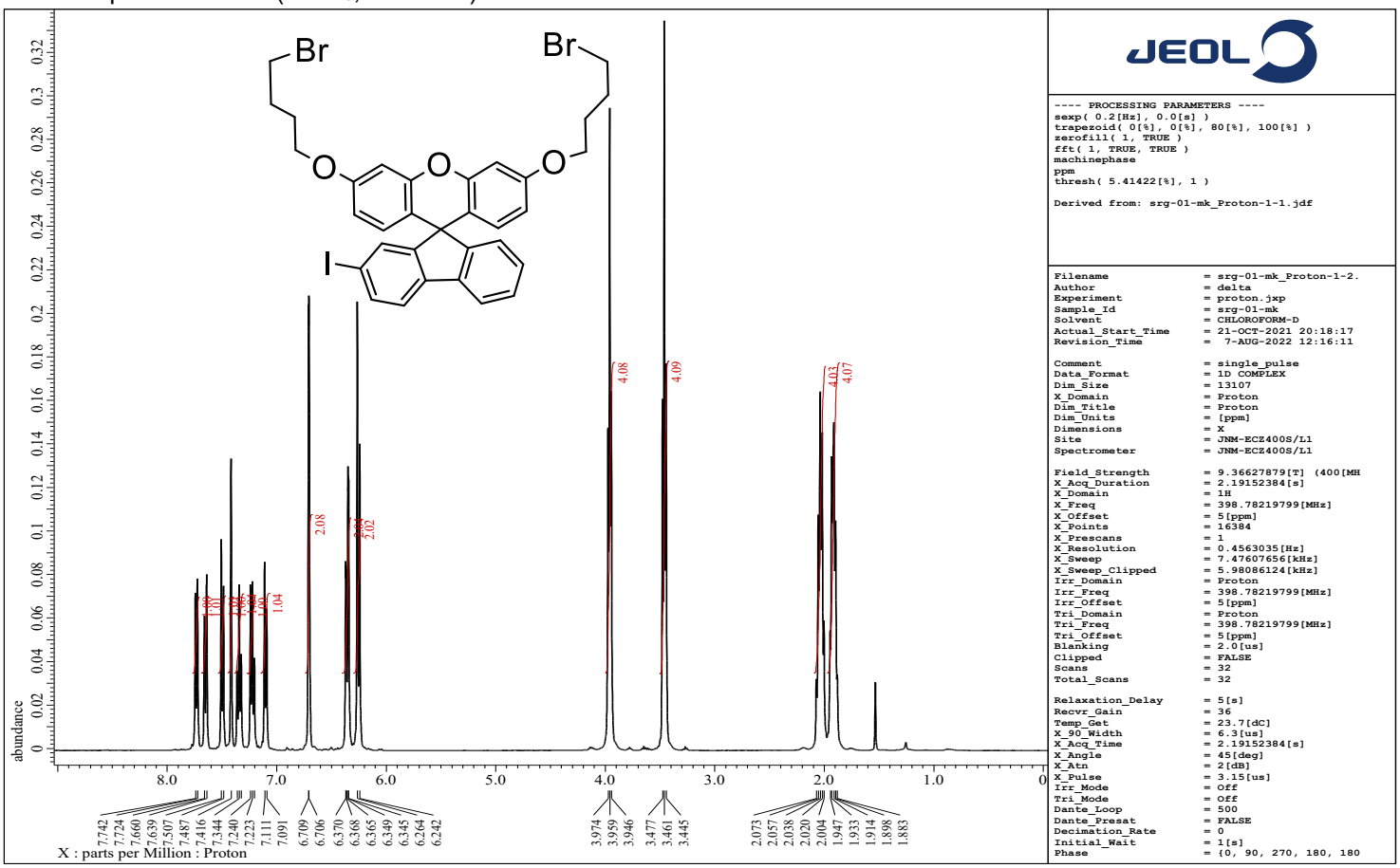



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.16472[ppm], 77[ppm] )
reference( 77.0[ppm], 77[ppm] )
thresh( 9.28104[%], 1 )

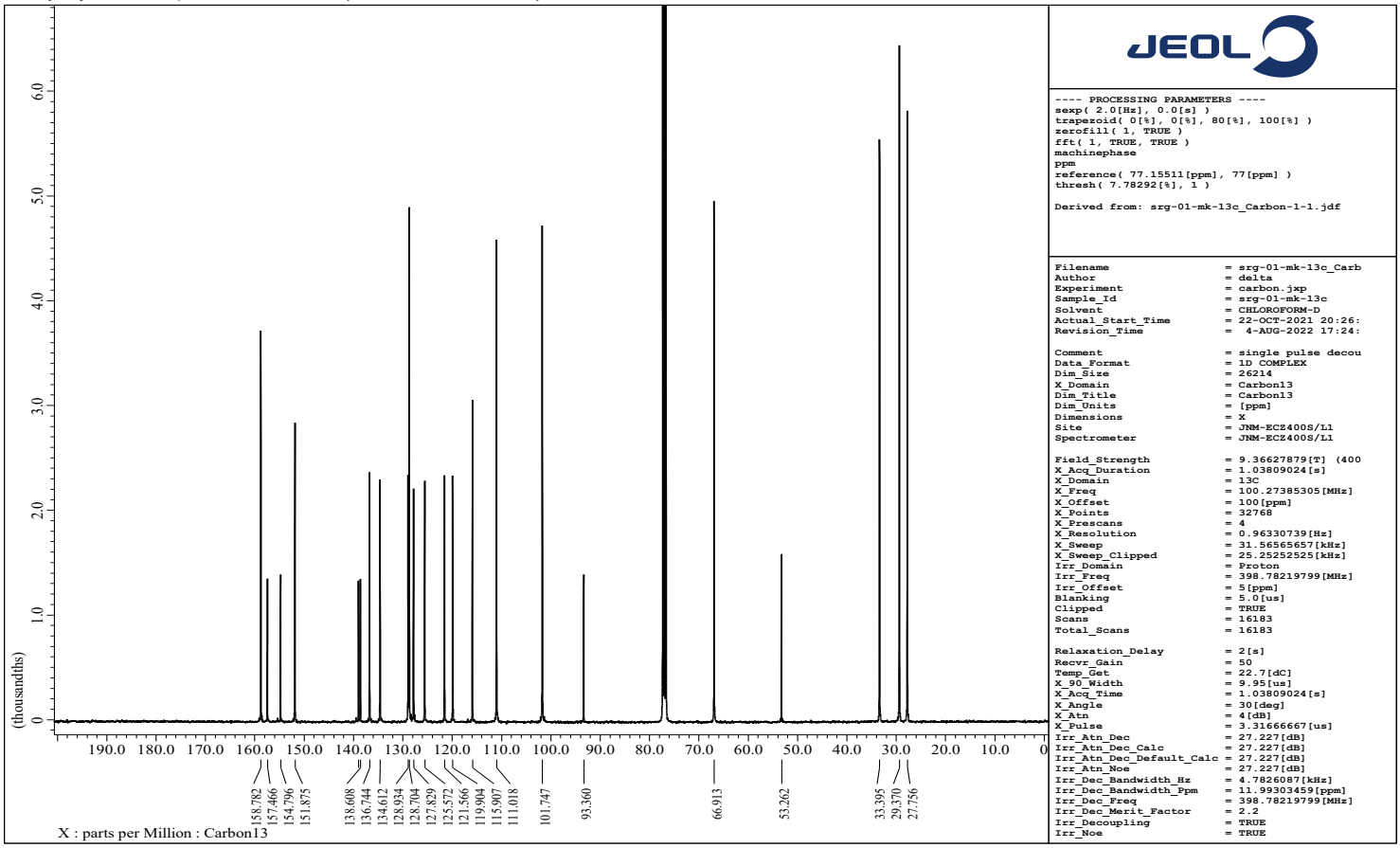
Derived from: srg-01-65-13-last_Carbon-1-1.jd
```

Filename	= srg-01-65-13-last_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-65-13-last
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JAN-2022 16:41:
Revision_Time	= 4-AUG-2022 17:19:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 1016
Total_Scans	= 1016
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.2[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

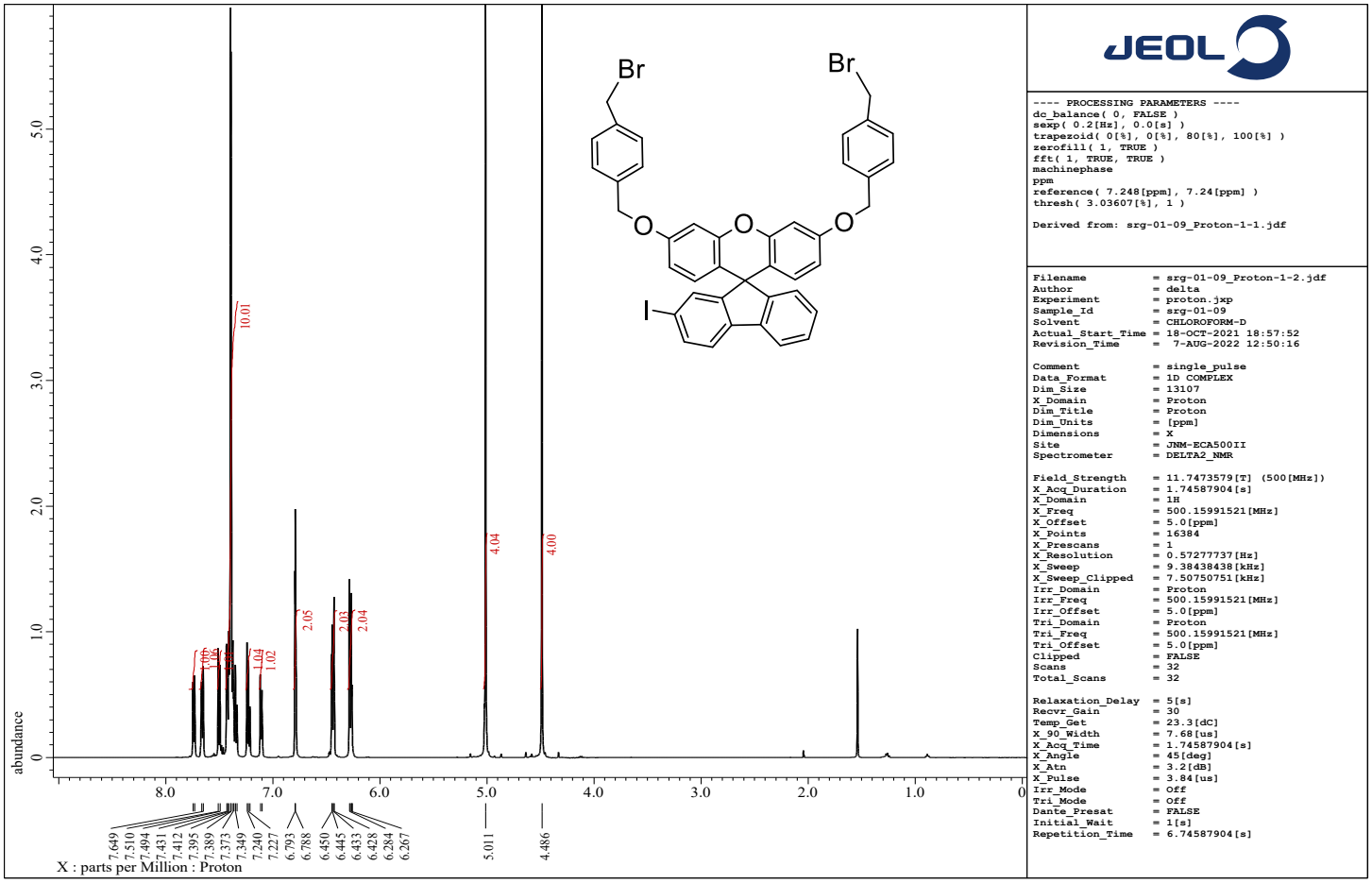
^{1}H NMR Spectrum of **5C** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **5C** ($CDCl_3$, 100 MHz).



^{1}H NMR Spectrum of **5D** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 3.03607[%], 1 )

Derived from: srg-01-09_Proton-1-1.jdf
```

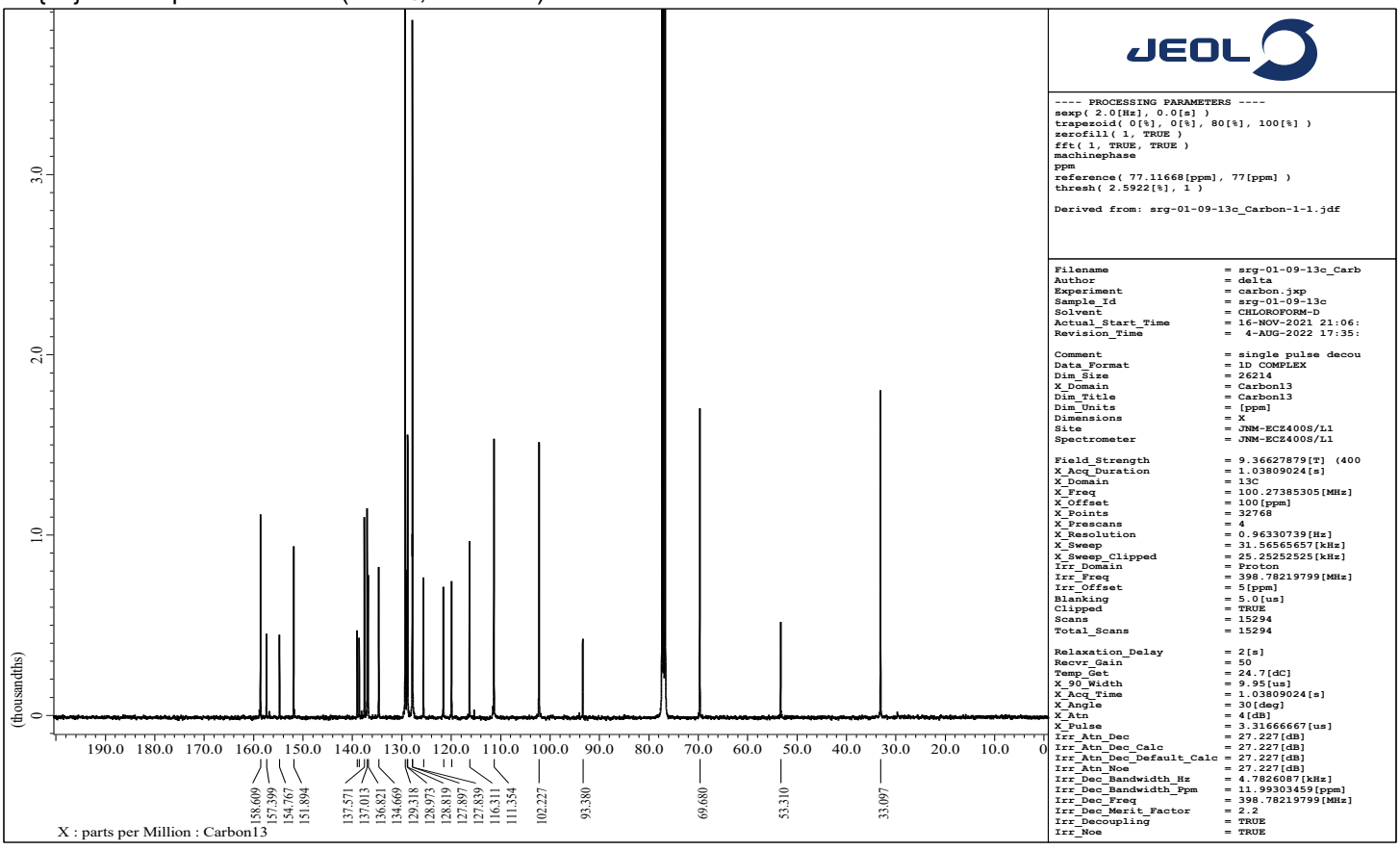
```
Filename              = srg-01-09_Proton-1-2.jdf
Author                = delta
Experiment            = proton.jxp
Sample_Id             = srg-01-09
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 18-OCT-2021 18:57:52
Revision_Time         =  7-AUG-2022 12:50:16

Comment               = single_pulse
Data_Format           = 1D COMPLEX
Dim_Size              = 13107
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Site                  = JNM-ECA500II
Spectrometer          = DELTA2_NMR

Field_Strength        = 11.7473579[T] (500[MHz])
X_Acq_Duration        = 1.74587904[s]
X_Domain              = 1H
X_Freq                = 500.15991521[MHz]
X_Offset              = 5.0[ppm]
X_Points              = 16384
X_Prescans            = 1
X_Resolution          = 0.57277737[Hz]
X_Sweep               = 9.38438438[kHz]
X_Sweep_Clipped       = 7.50750751[kHz]
Irr_Domain            = Proton
Irr_Freq              = 500.15991521[MHz]
Irr_Offset            = 5.0[ppm]
Tri_Domain            = Proton
Tri_Freq              = 500.15991521[MHz]
Tri_Offset            = 5.0[ppm]
Clipped               = FALSE
Scans                 = 32
Total_Scans           = 32

Relaxation_Delay      = 5[s]
Recvr_Gain            = 30
Temp_Get              = 23.3[dC]
X_90_Width            = 7.68[us]
X_Acq_Time            = 1.74587904[s]
X_Angle               = 45[deg]
X_Atn                 = 3.2[dB]
X_Pulse               = 3.84[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Presat          = FALSE
Initial_Wait          = 1[s]
Repetition_Time       = 6.74587904[s]
```

^{13}C {^{1}H} NMR Spectrum of **5D** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11668[ppm], 77[ppm] )
thresh( 2.5922[%], 1 )

Derived from: srg-01-09-13c_Carbon-1-1.jdf
```

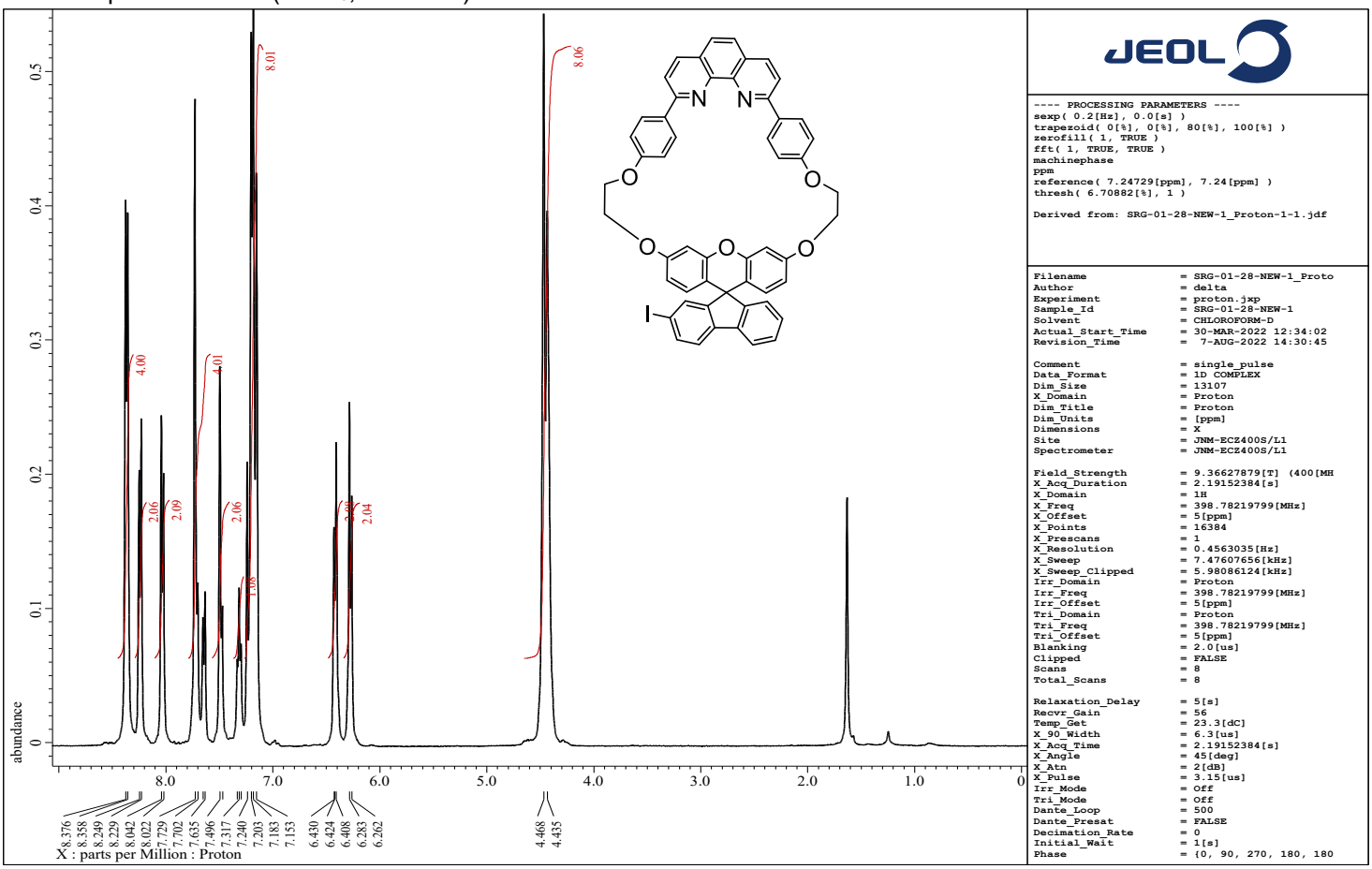
```
Filename              = srg-01-09-13c_Carb
Author                = delta
Experiment            = carbon.jxp
Sample_Id             = srg-01-09-13c
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 16-NOV-2021 21:06:
Revision_Time         =  4-AUG-2022 17:35:

Comment               = single pulse decou
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Carbon13
Dim_Title             = Carbon13
Dim_Units             = [ppm]
Dimensions            = X
Site                  = JNM-ECZ400S/L1
Spectrometer          = JNM-ECZ400S/L1

Field_Strength        = 9.36627879[T] (400
X_Acq_Duration        = 1.03809024[s]
X_Domain              = 13C
X_Freq                = 100.27385305[MHz]
X_Offset              = 100[ppm]
X_Points              = 32768
X_Prescans            = 4
X_Resolution          = 0.96330739[Hz]
X_Sweep               = 31.56565657[kHz]
X_Sweep_Clipped       = 25.25252525[kHz]
Irr_Domain            = Proton
Irr_Freq              = 398.78219799[MHz]
Irr_Offset            = 5[ppm]
Blanking              = 5.0[us]
Clipped               = TRUE
Scans                 = 15294
Total_Scans           = 15294

Relaxation_Delay      = 2[s]
Recvr_Gain            = 50
Temp_Get              = 24.7[dC]
X_90_Width            = 9.95[us]
X_Acq_Time            = 1.03809024[s]
X_Angle               = 30[deg]
X_Atn                 = 4[dB]
X_Pulse               = 3.31666667[us]
Irr_Atn_Dec           = 27.227[dB]
Irr_Atn_Dec_Calc      = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe           = 27.227[dB]
Irr_Dec_Bandwidth_Hz  = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.99303459[ppm]
Irr_Dec_Freq          = 398.78219799[MHz]
Irr_Dec_Merit_Factor  = 2.2
Irr_Decoupling        = TRUE
Irr_Noe               = TRUE
```

^{1}H NMR Spectrum of **7A** ($CDCl_3$, 400 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24729[ppm], 7.24[ppm] )
thresh( 6.70882[%], 1 )

Derived from: SRG-01-28-NEW-1_Proton-1-1.jdf

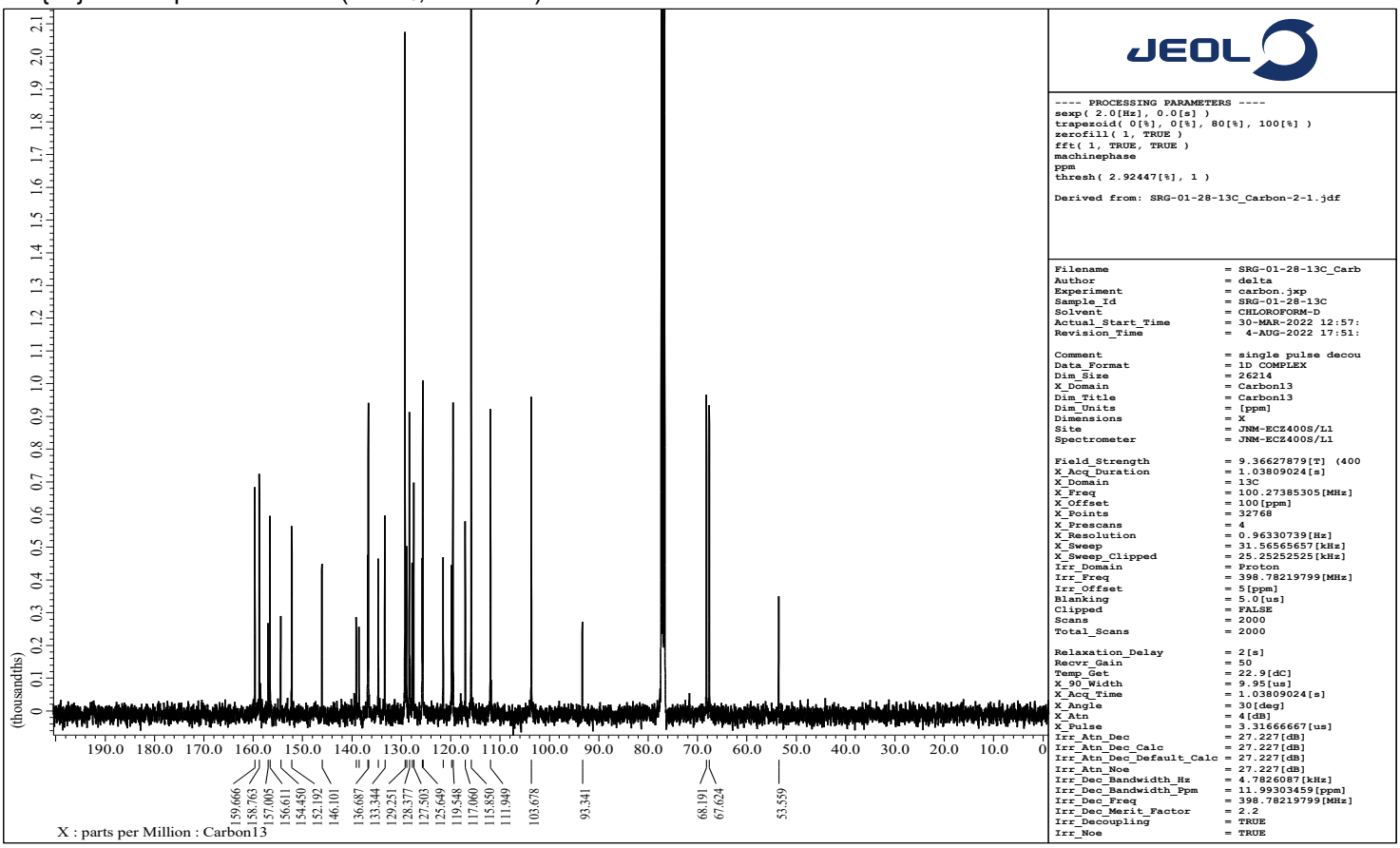
Filename                = SRG-01-28-NEW-1_Proto
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = SRG-01-28-NEW-1
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 30-MAR-2022 12:34:02
Revision_Time           =  7-AUG-2022 14:30:45

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400[MH
X_Acq_Duration          = 2.19152384[s]
X_Domain                = 1H
X_Freq                  = 398.78219799[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.4563035[Hz]
X_Sweep                 = 7.47607656[kHz]
X_Sweep_Clipped         = 5.98086124[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 398.78219799[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2.0[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 23.3[dC]
X_90_Width              = 6.3[us]
X_Acq_Time              = 2.19152384[s]
X_Angle                 = 45[deg]
X_Atn                   = 2[dB]
X_Pulse                 = 3.15[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
```

^{13}C {^{1}H} NMR Spectrum of **7A** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 2.92447[%], 1 )

Derived from: SRG-01-28-13C_Carbon-2-1.jdf

Filename                = SRG-01-28-13C_Carb
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = SRG-01-28-13C
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 30-MAR-2022 12:57:
Revision_Time           =  4-AUG-2022 17:51:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.27385305[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5.0[us]
Clipped                 = FALSE
Scans                   = 2000
Total_Scans             = 2000

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 22.9[dC]
X_90_Width              = 9.95[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 4[dB]
X_Pulse                 = 3.31666667[us]
Irr_Atn_Dec             = 27.227[dB]
Irr_Atn_Dec_Calc        = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe             = 27.227[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.99303459[ppm]
Irr_Dec_Freq            = 398.78219799[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
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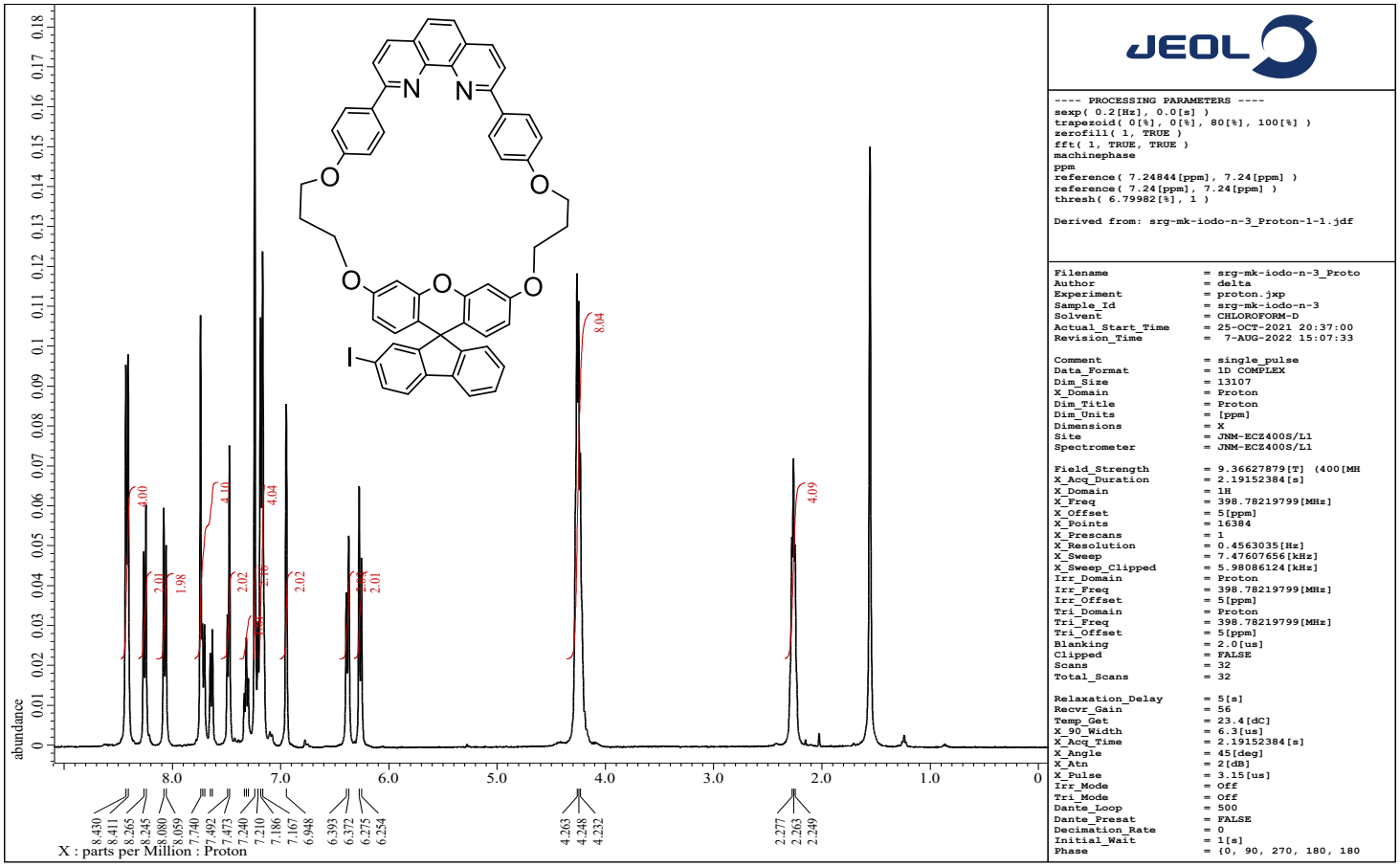
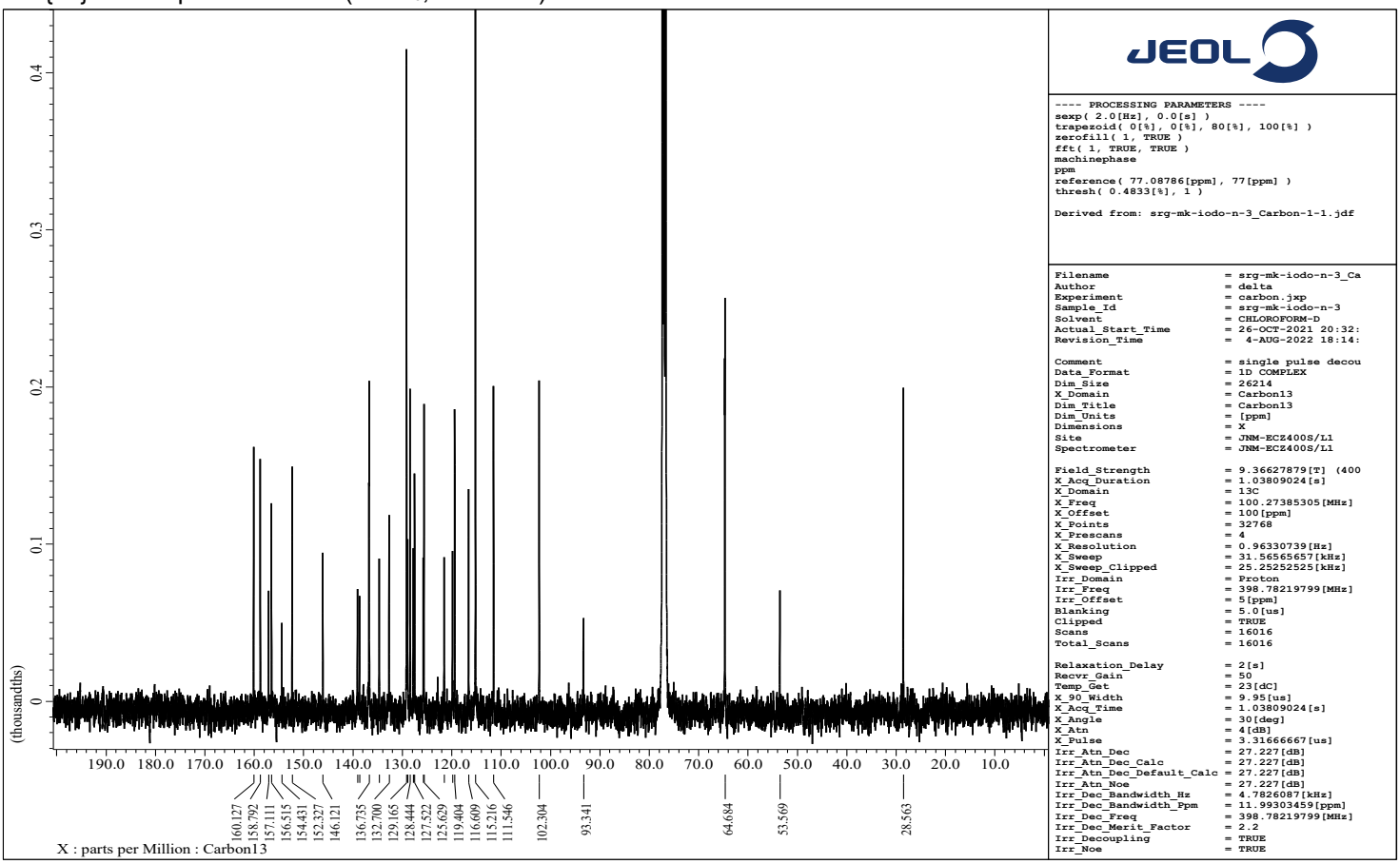
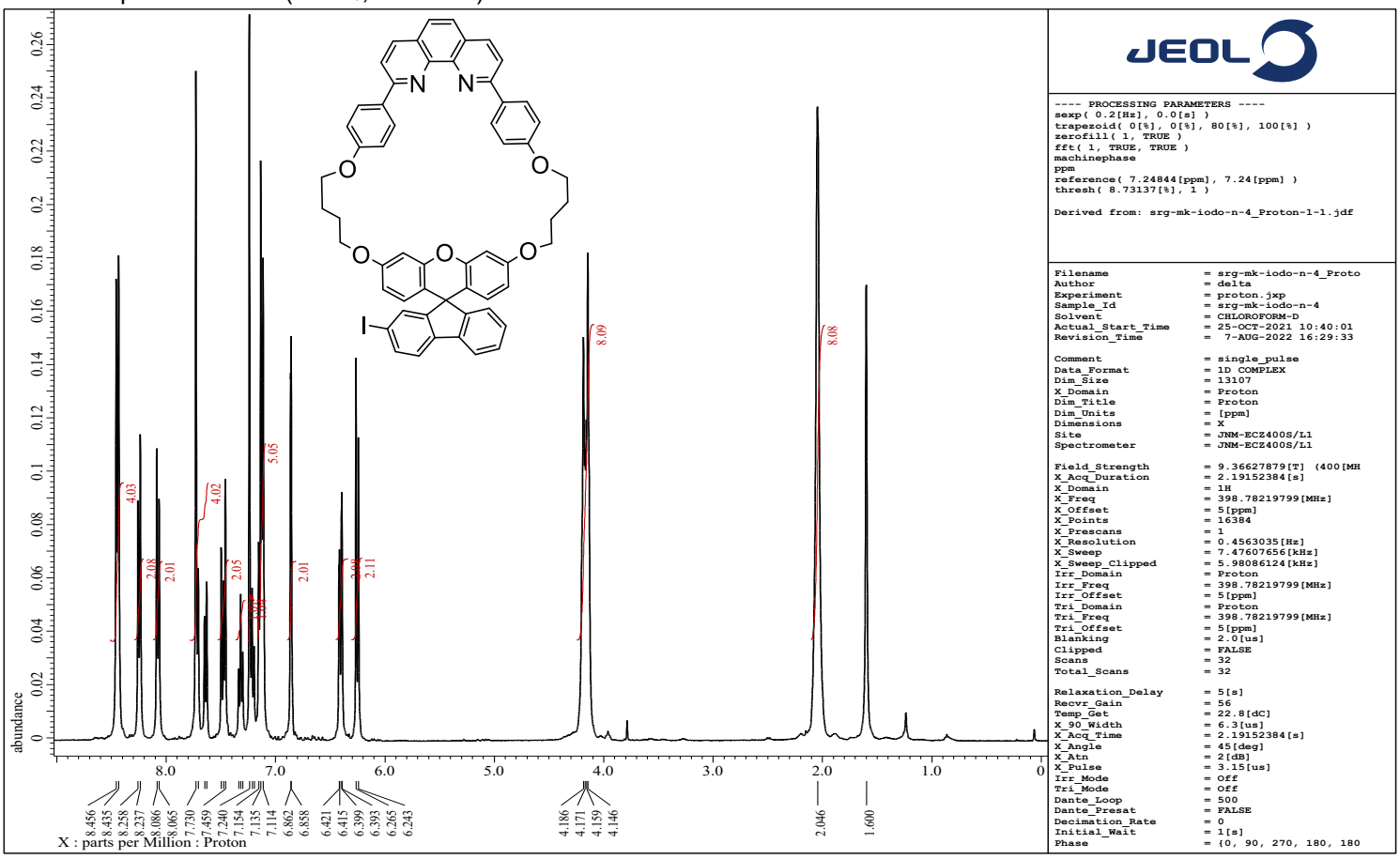
^{1}H NMR Spectrum of **7B** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **7B** ($CDCl_3$, 100 MHz).



^{1}H NMR Spectrum of **7C** ($CDCl_3$, 400 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24844[ppm], 7.24[ppm] )
thresh( 8.73137[%], 1 )

Derived from: srg-mk-iodo-n-4_Proton-1-1.jdf
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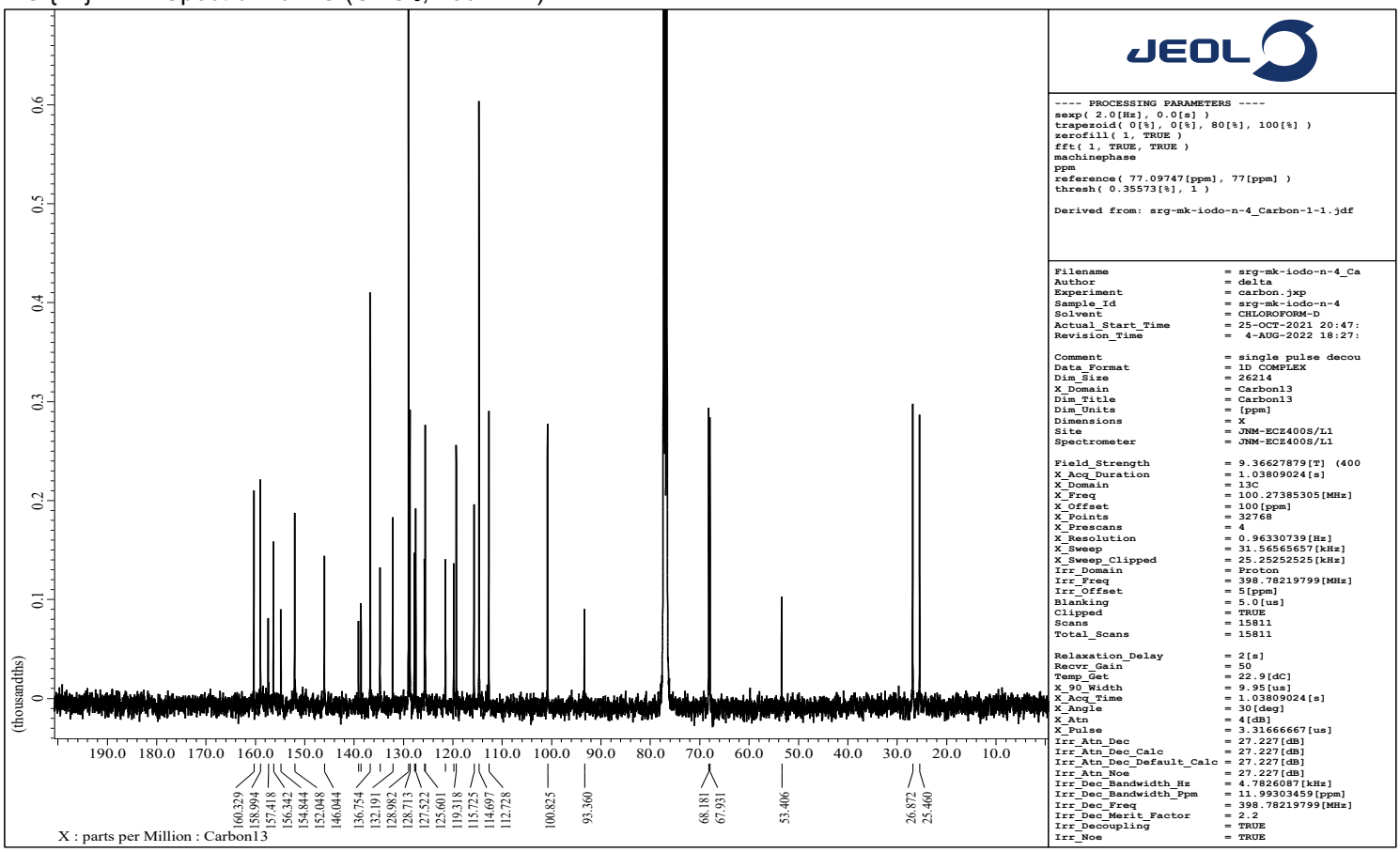
Filename	= srg-mk-iodo-n-4_Proto
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-mk-iodo-n-4
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2021 10:40:01
Revision_Time	= 7-AUG-2022 16:29:33
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 22.8[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

^{13}C {^{1}H} NMR Spectrum of **7C** ($CDCl_3$, 100 MHz).

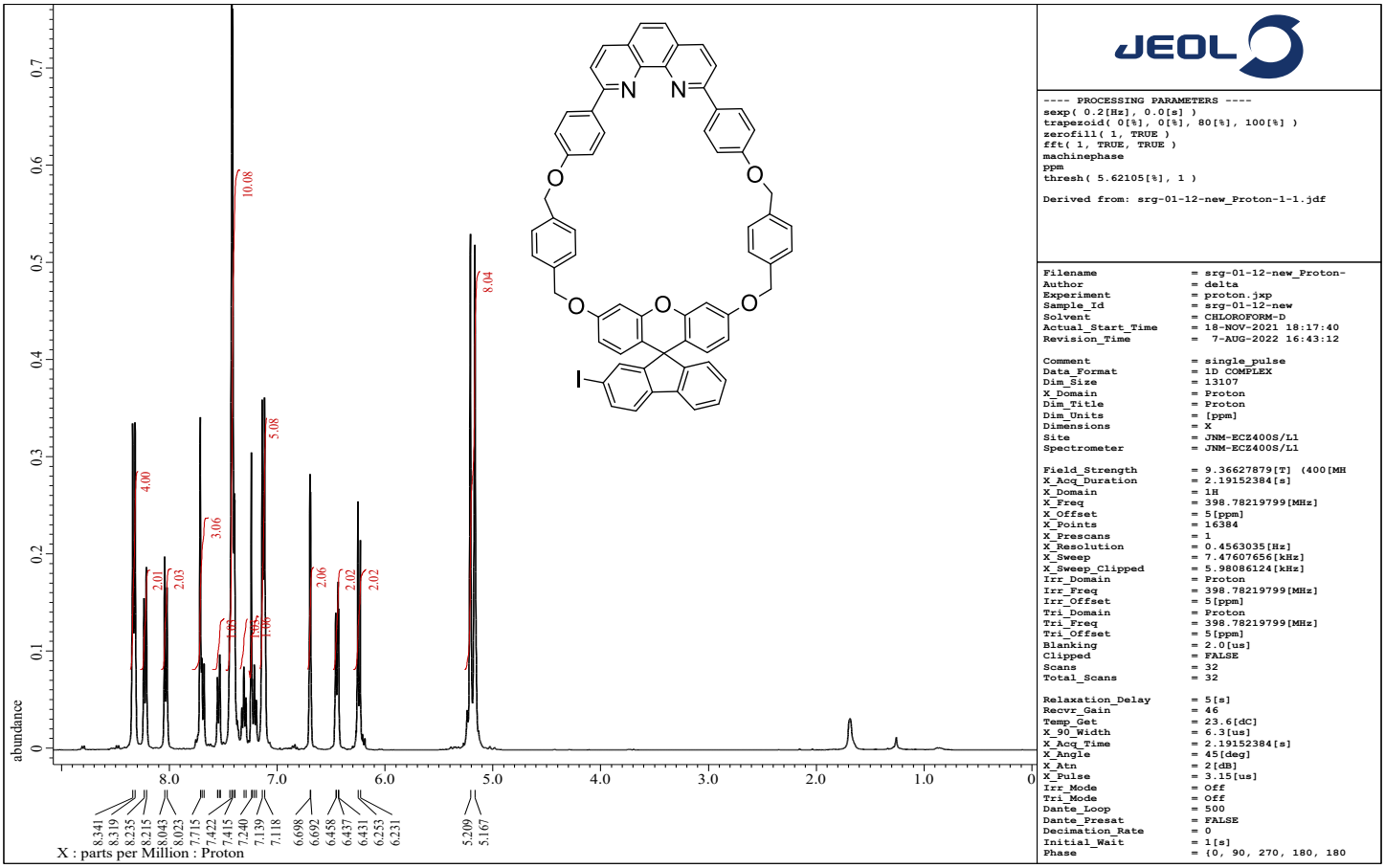



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.09747[ppm], 77[ppm] )
thresh( 0.35573[%], 1 )

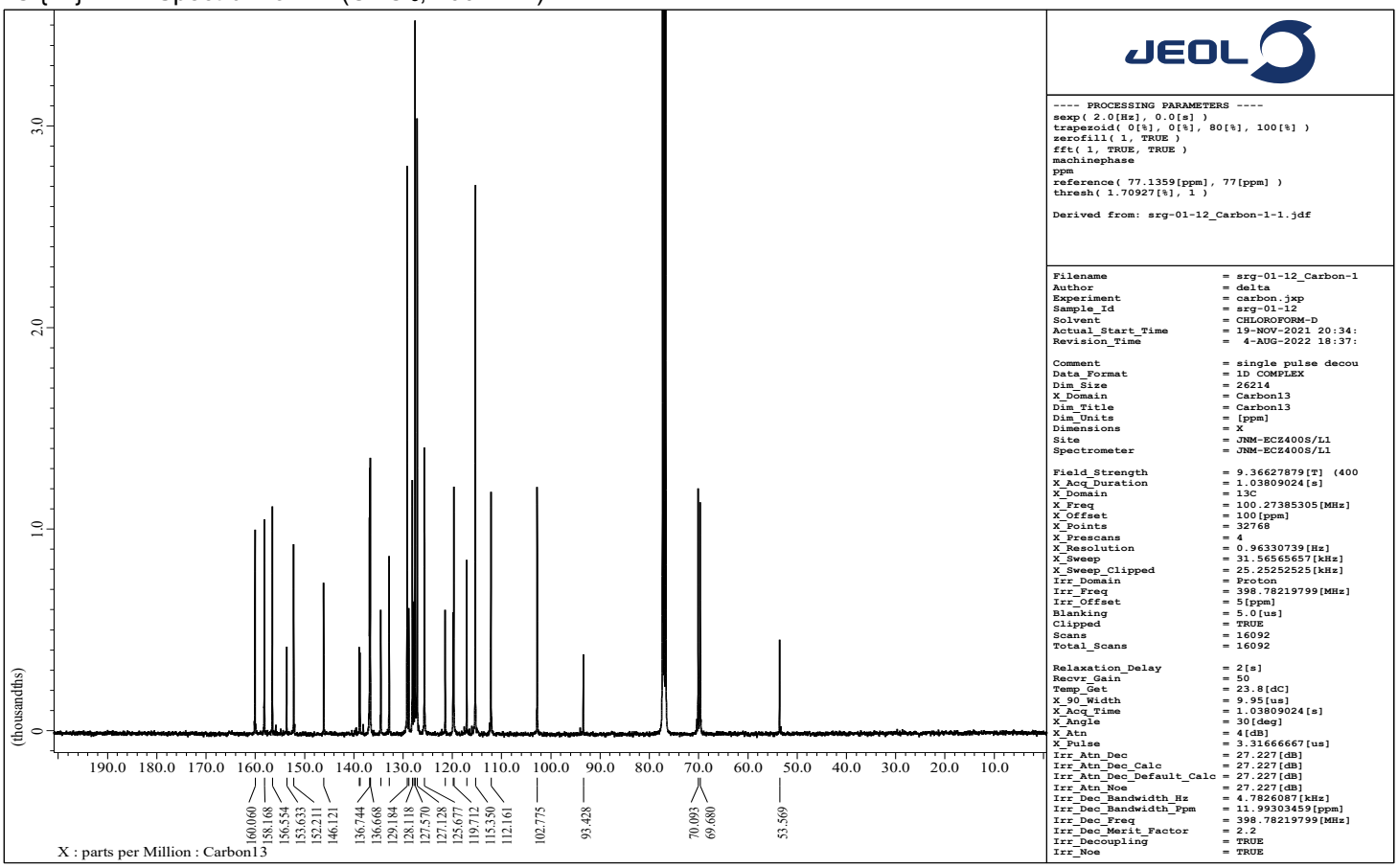
Derived from: srg-mk-iodo-n-4_Carbon-1-1.jdf
```

Filename	= srg-mk-iodo-n-4_Ca
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-mk-iodo-n-4
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-OCT-2021 20:47:
Revision_Time	= 4-AUG-2022 18:27:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 15811
Total_Scans	= 15811
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22.9[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

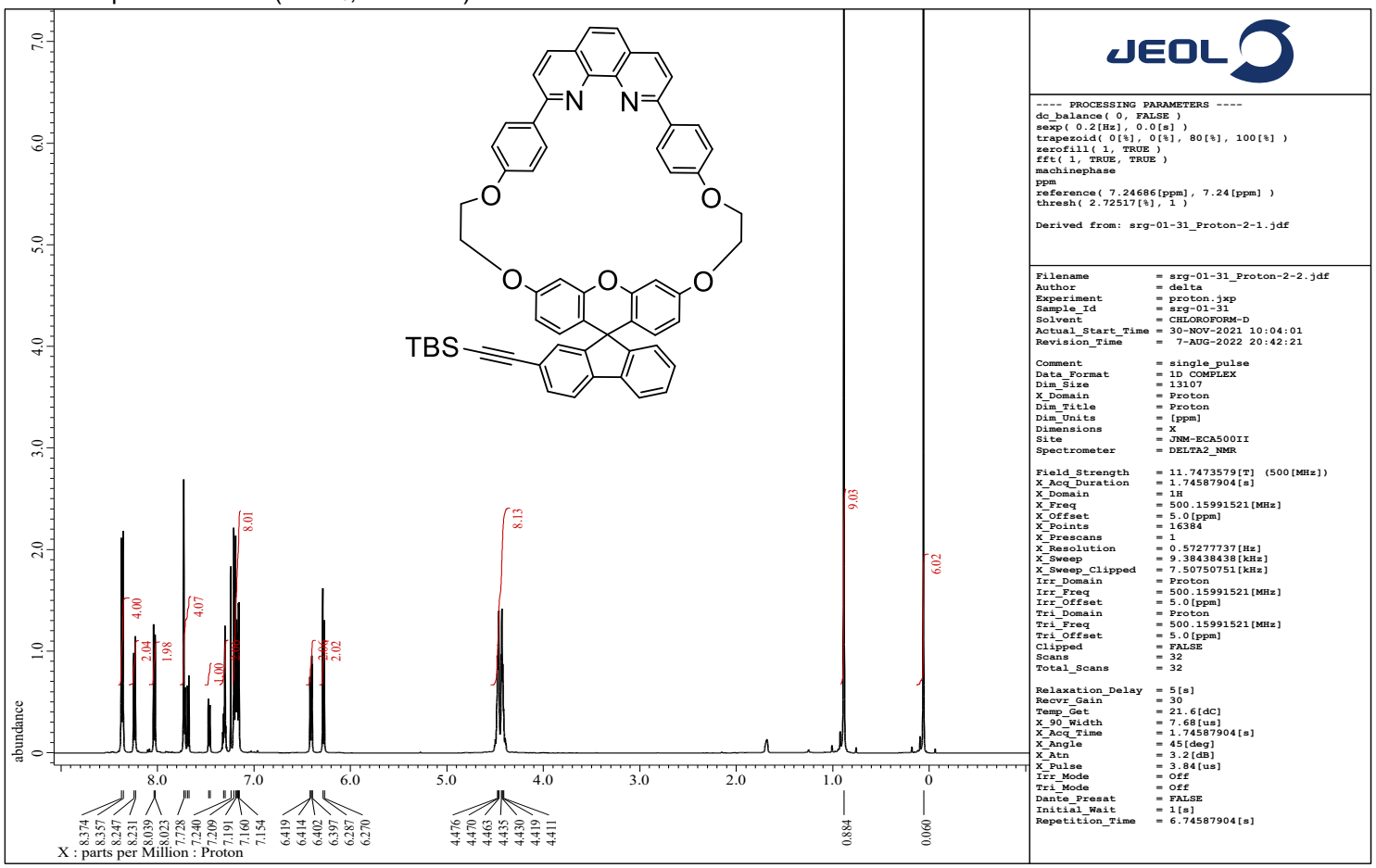
^{1}H NMR Spectrum of **7D** ($CDCl_3$, 400 MHz).



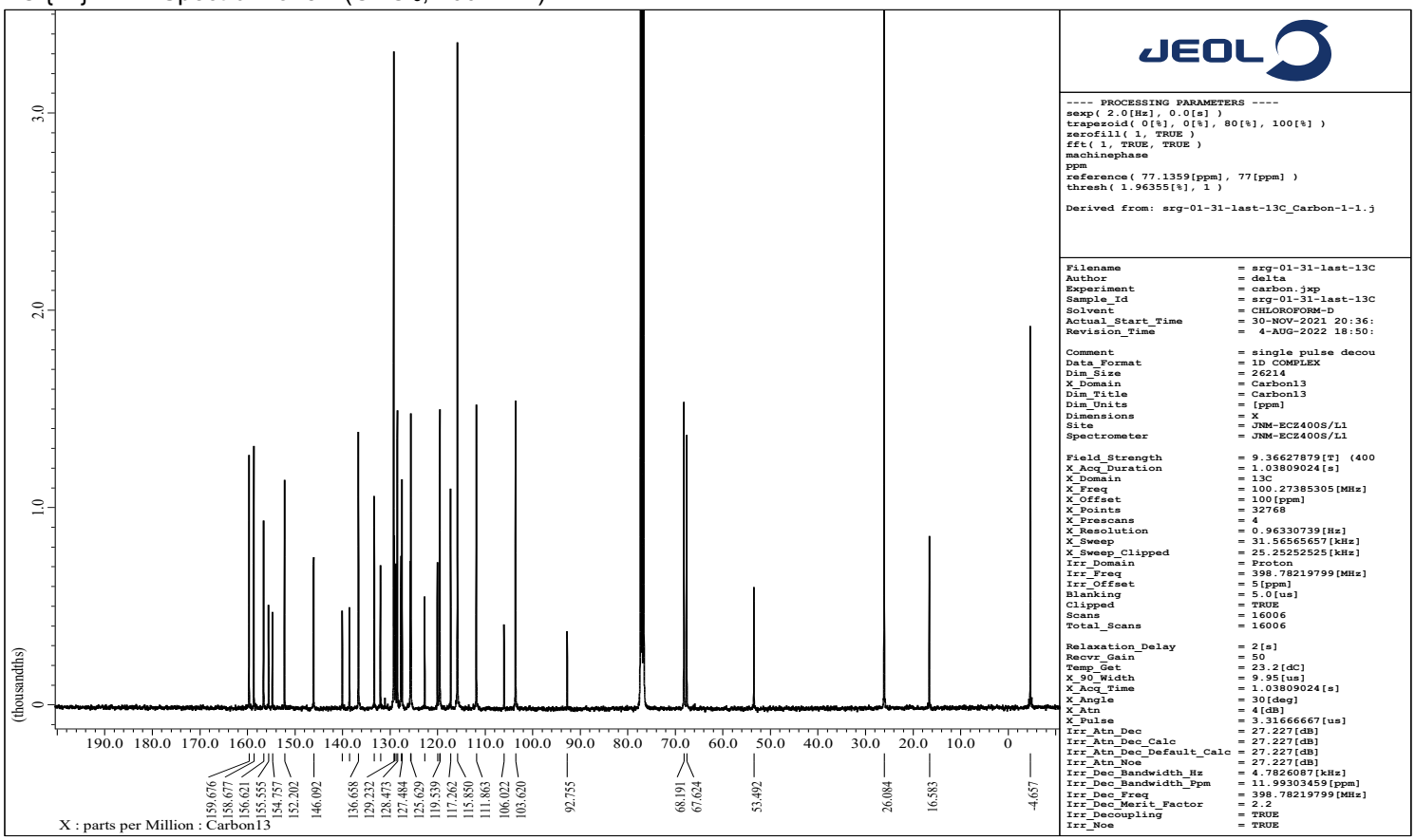
^{13}C {^{1}H} NMR Spectrum of **7D** ($CDCl_3$, 100 MHz).



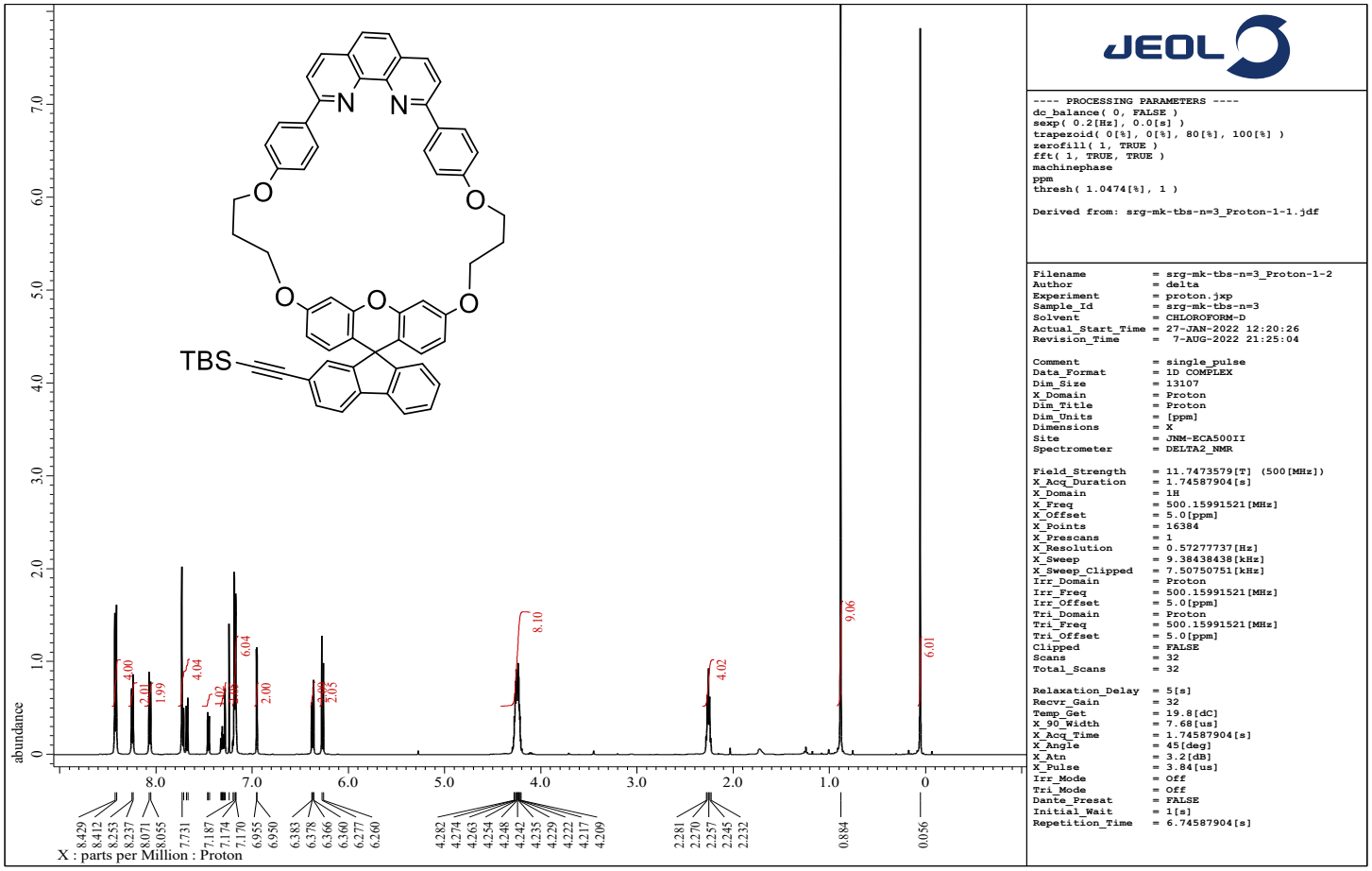
^{1}H NMR Spectrum of **8A** ($CDCl_3$, 500 MHz).



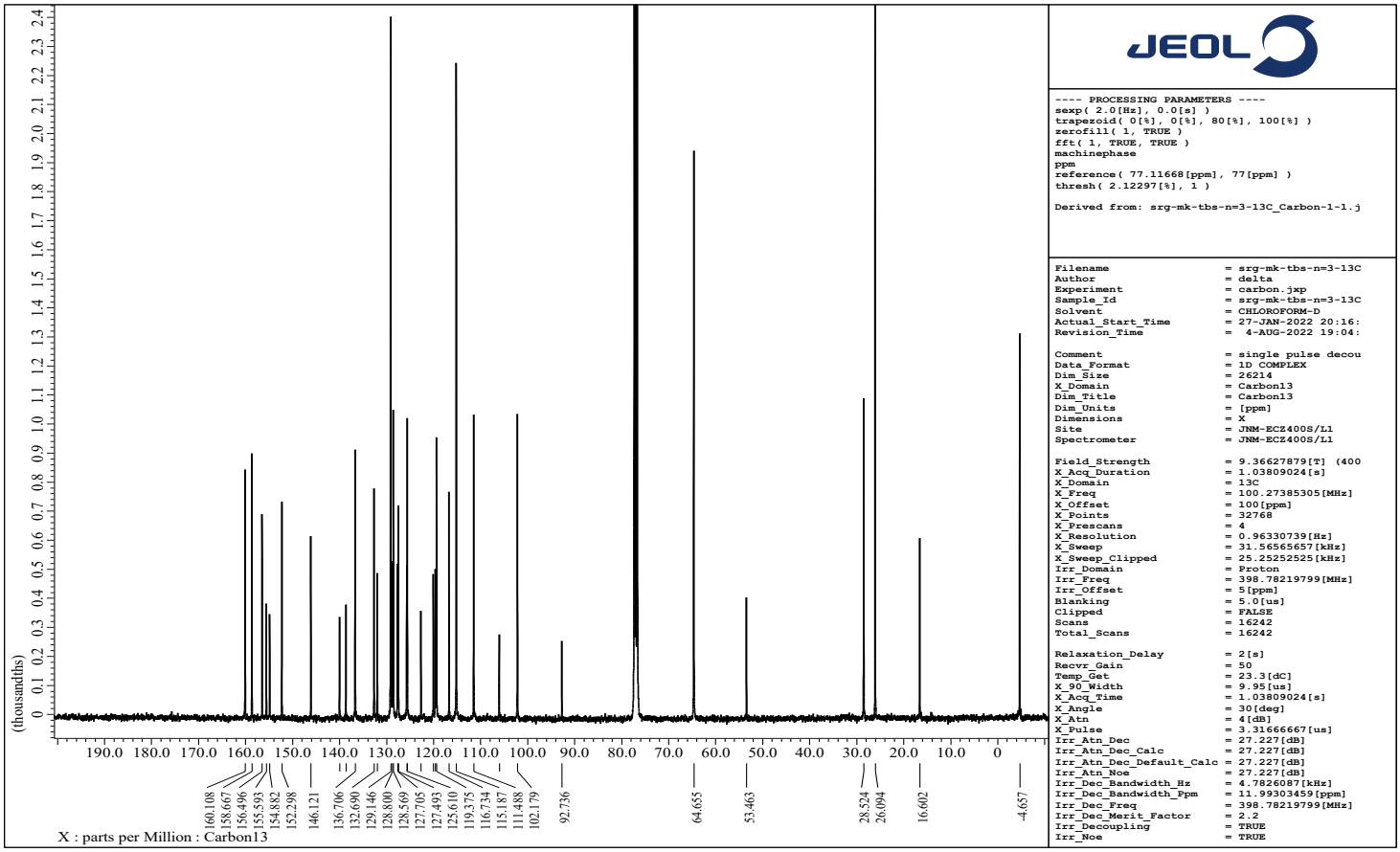
^{13}C {^{1}H} NMR Spectrum of **8A** ($CDCl_3$, 100 MHz).



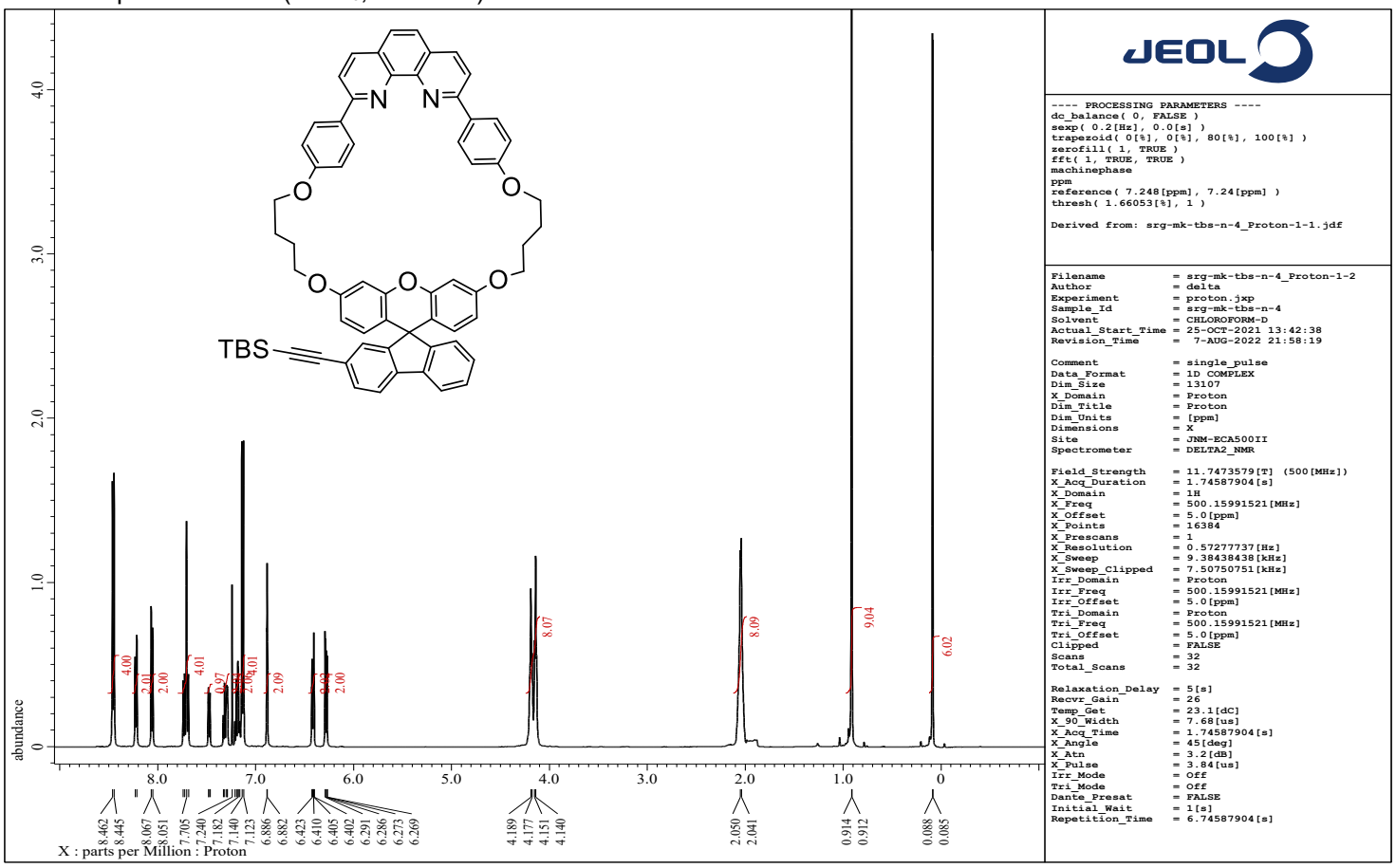
^{1}H NMR Spectrum of **8B** ($CDCl_3$, 500 MHz).



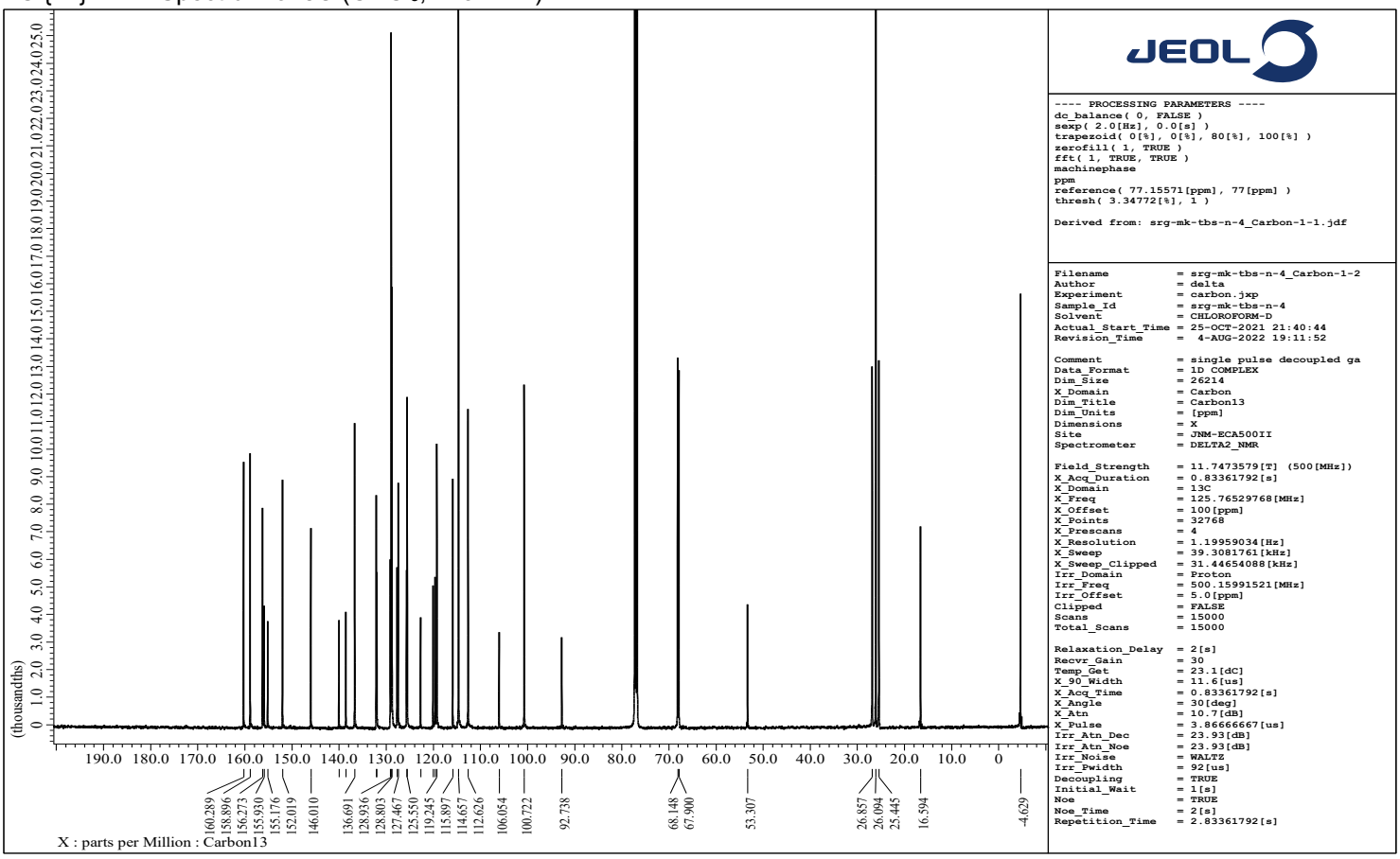
^{13}C {^{1}H} NMR Spectrum of **8B** ($CDCl_3$, 100 MHz).



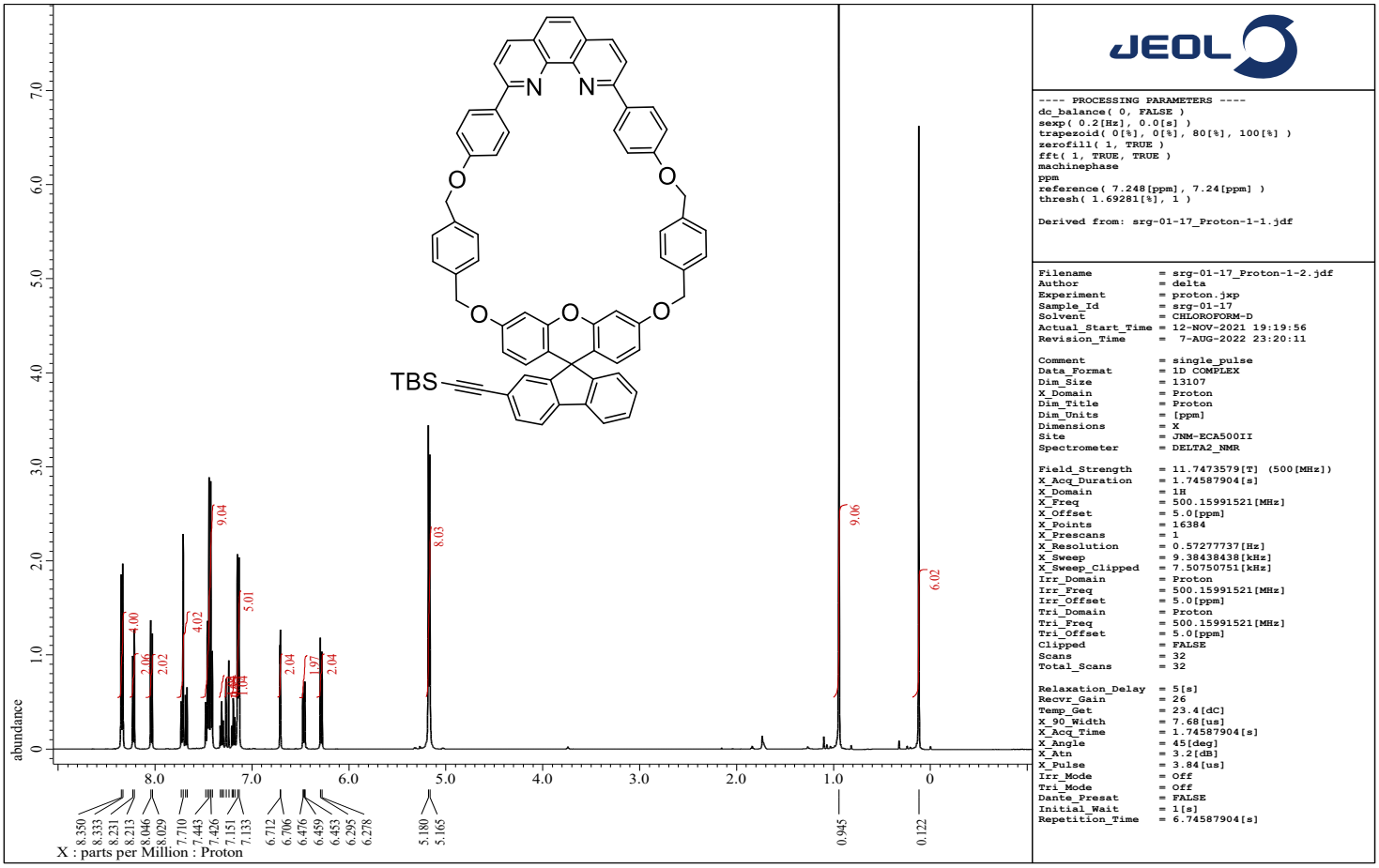
^{1}H NMR Spectrum of **8C** ($CDCl_3$, 500 MHz).



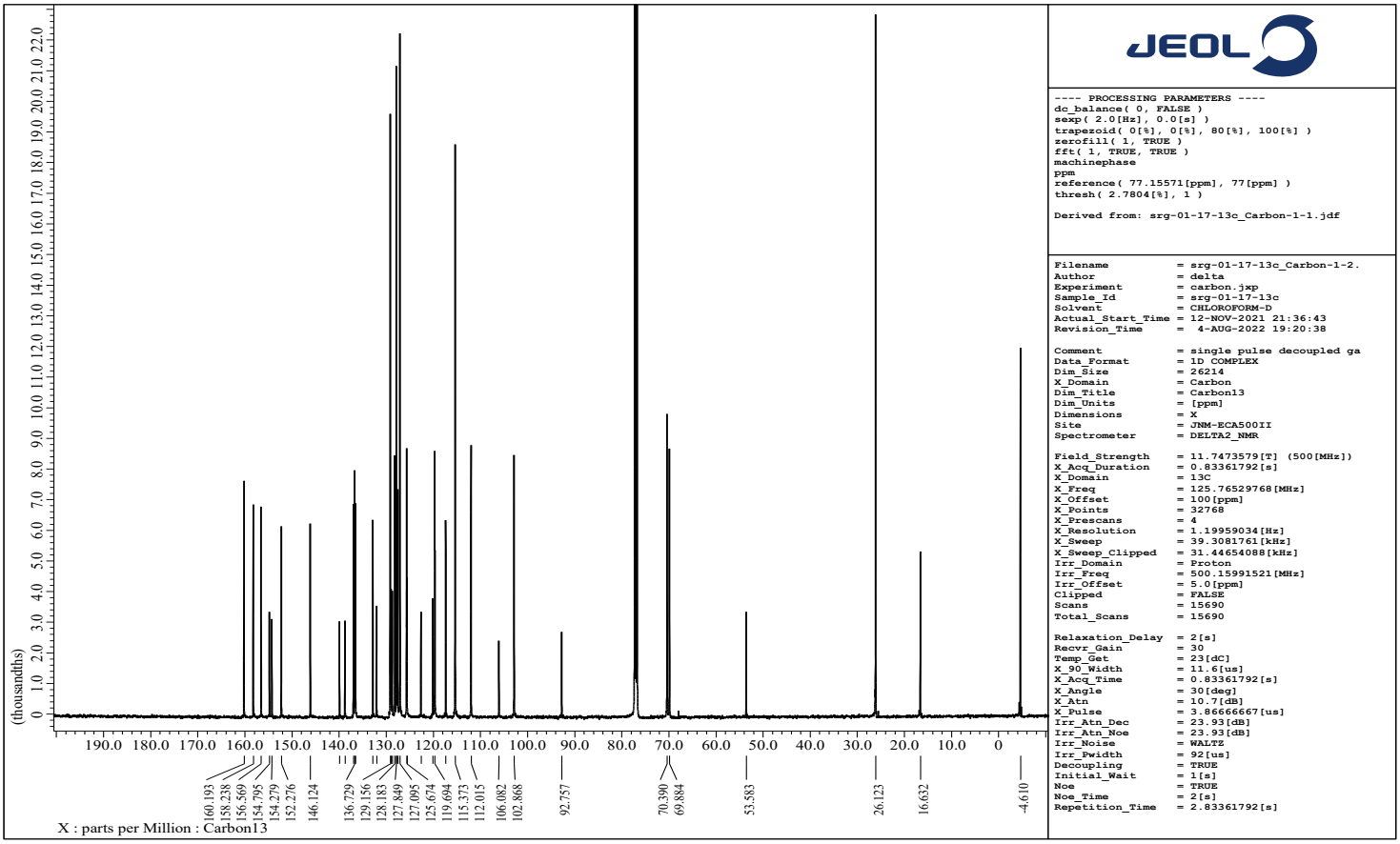
^{13}C {^{1}H} NMR Spectrum of **8C** ($CDCl_3$, 125 MHz).



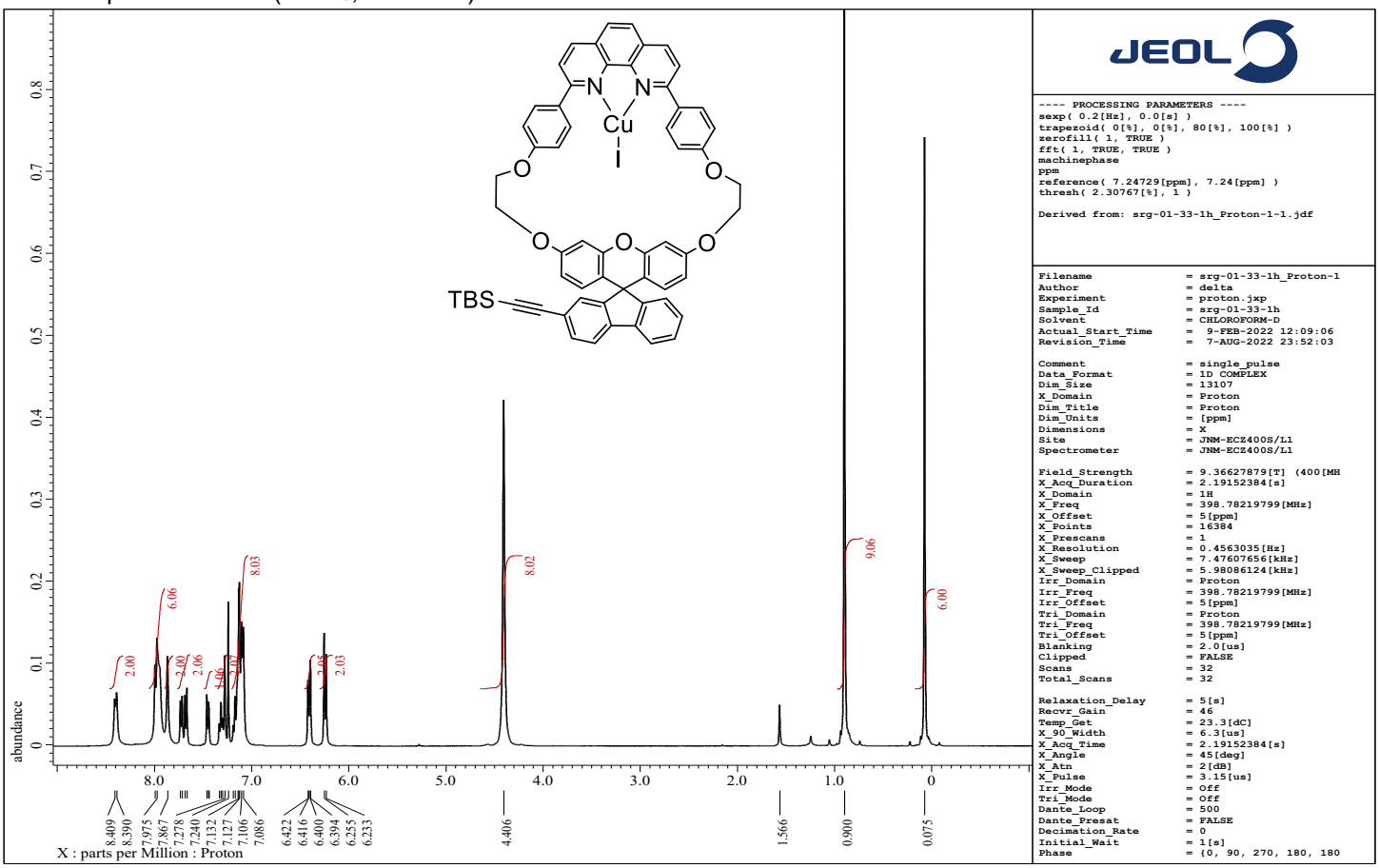
^{1}H NMR Spectrum of **8D** ($CDCl_3$, 500 MHz).



^{13}C {^{1}H} NMR Spectrum of **8D** ($CDCl_3$, 125 MHz).



^{1}H NMR Spectrum of **9A** ($CDCl_3$, 400 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24729[ppm], 7.24[ppm] )
thresh( 2.30767[%], 1 )

Derived from: srg-01-33-1h_Proton-1-1.jdf
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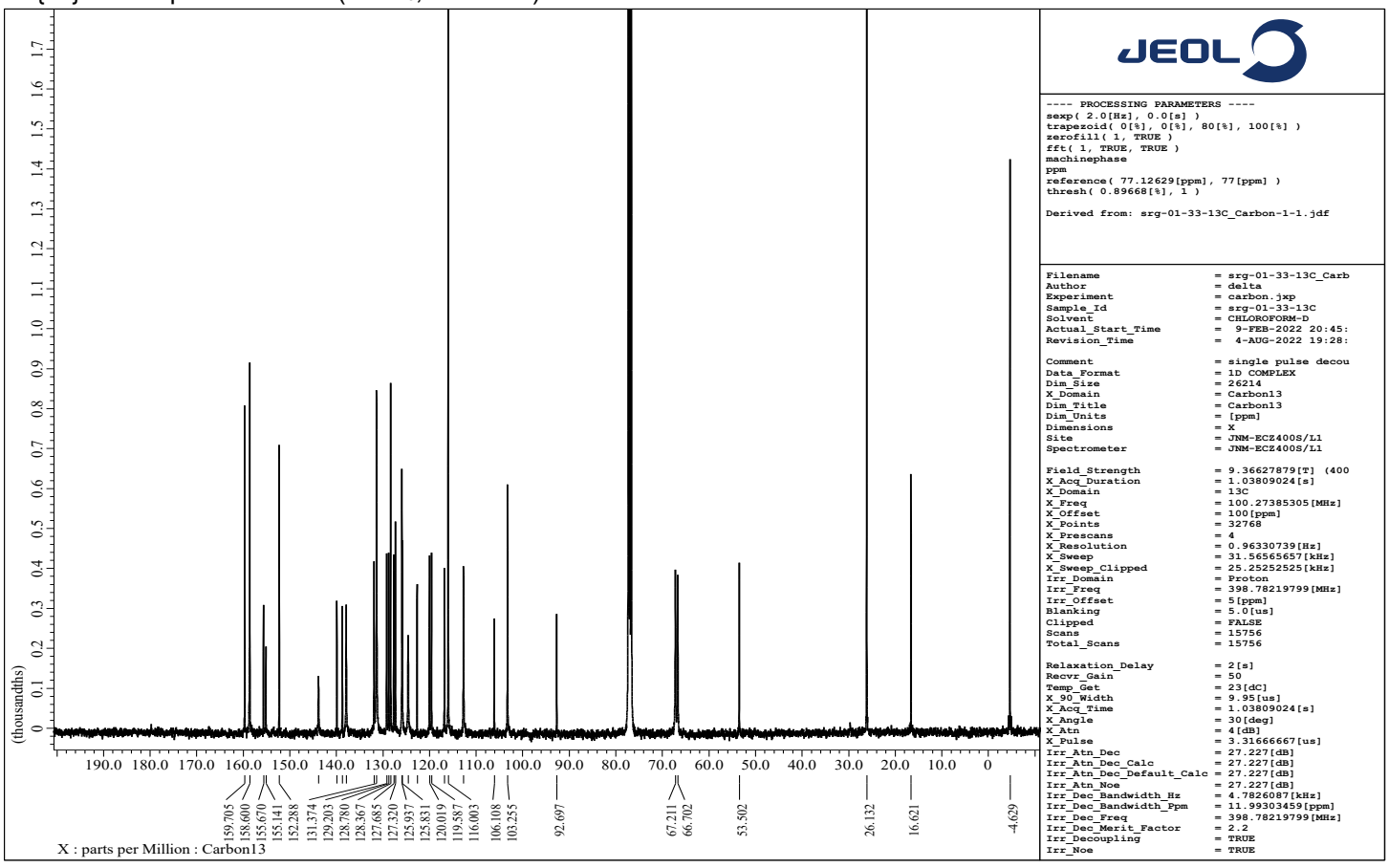
Filename	= srg-01-33-1h_Proton-1
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-33-1h
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-FEB-2022 12:09:06
Revision_Time	= 7-AUG-2022 23:52:03
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 23.3[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

^{13}C {^{1}H} NMR Spectrum of **9A** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.12629[ppm], 77[ppm] )
thresh( 0.89668[%], 1 )

Derived from: srg-01-33-13C_Carbon-1-1.jdf
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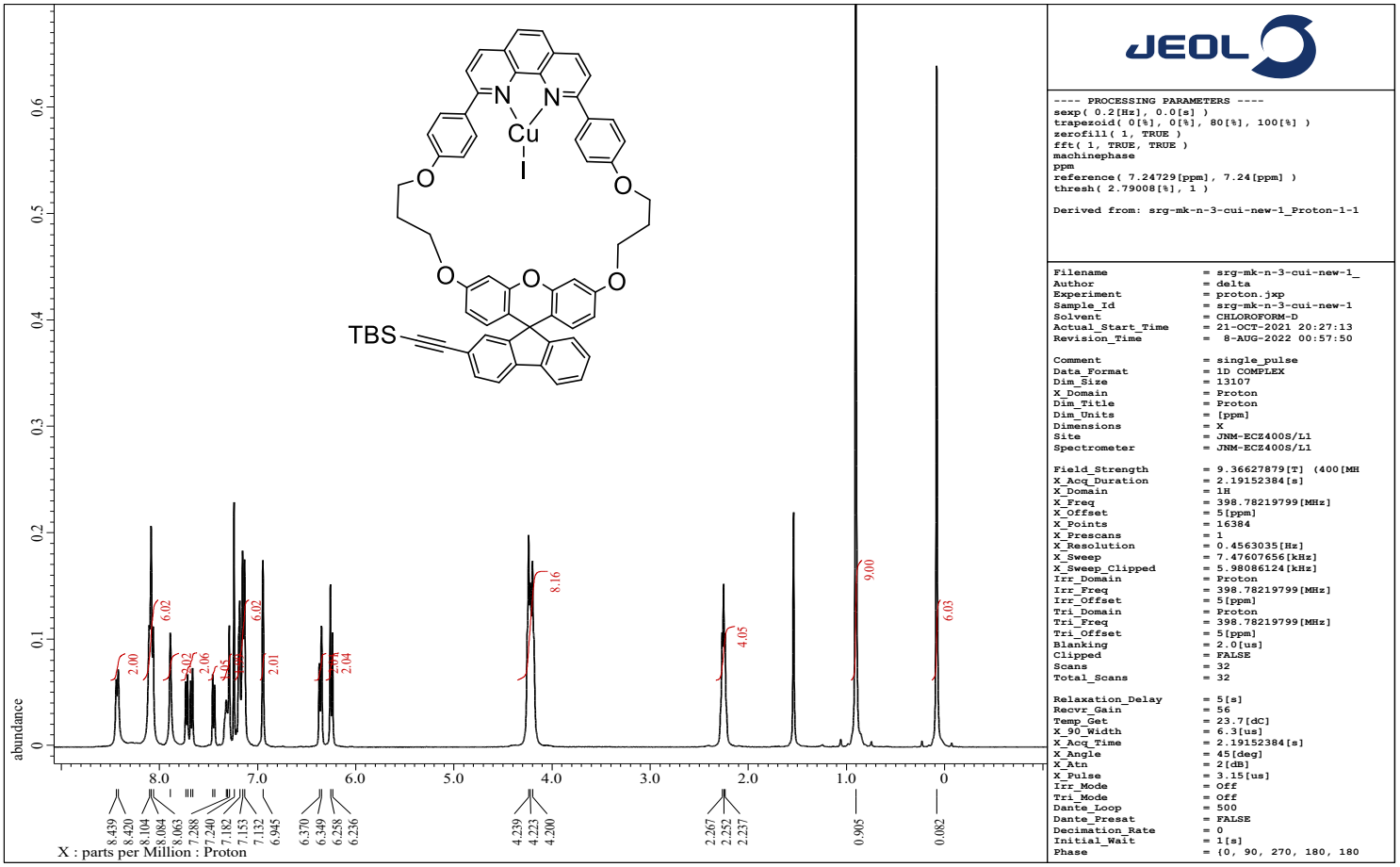
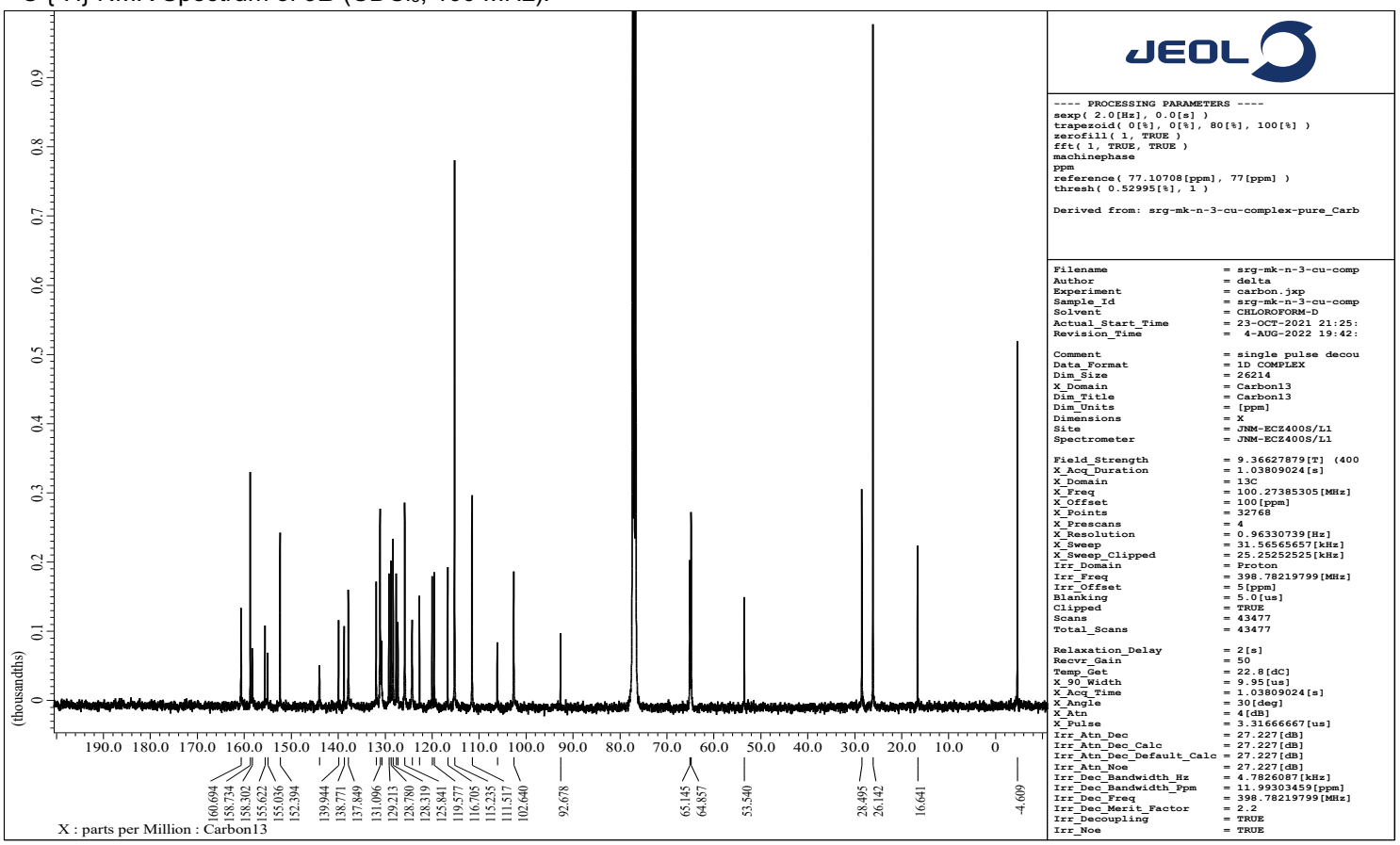
Filename	= srg-01-33-13C_Carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-33-13C
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-FEB-2022 20:45:
Revision_Time	= 4-AUG-2022 19:28:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 15756
Total_Scans	= 15756
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

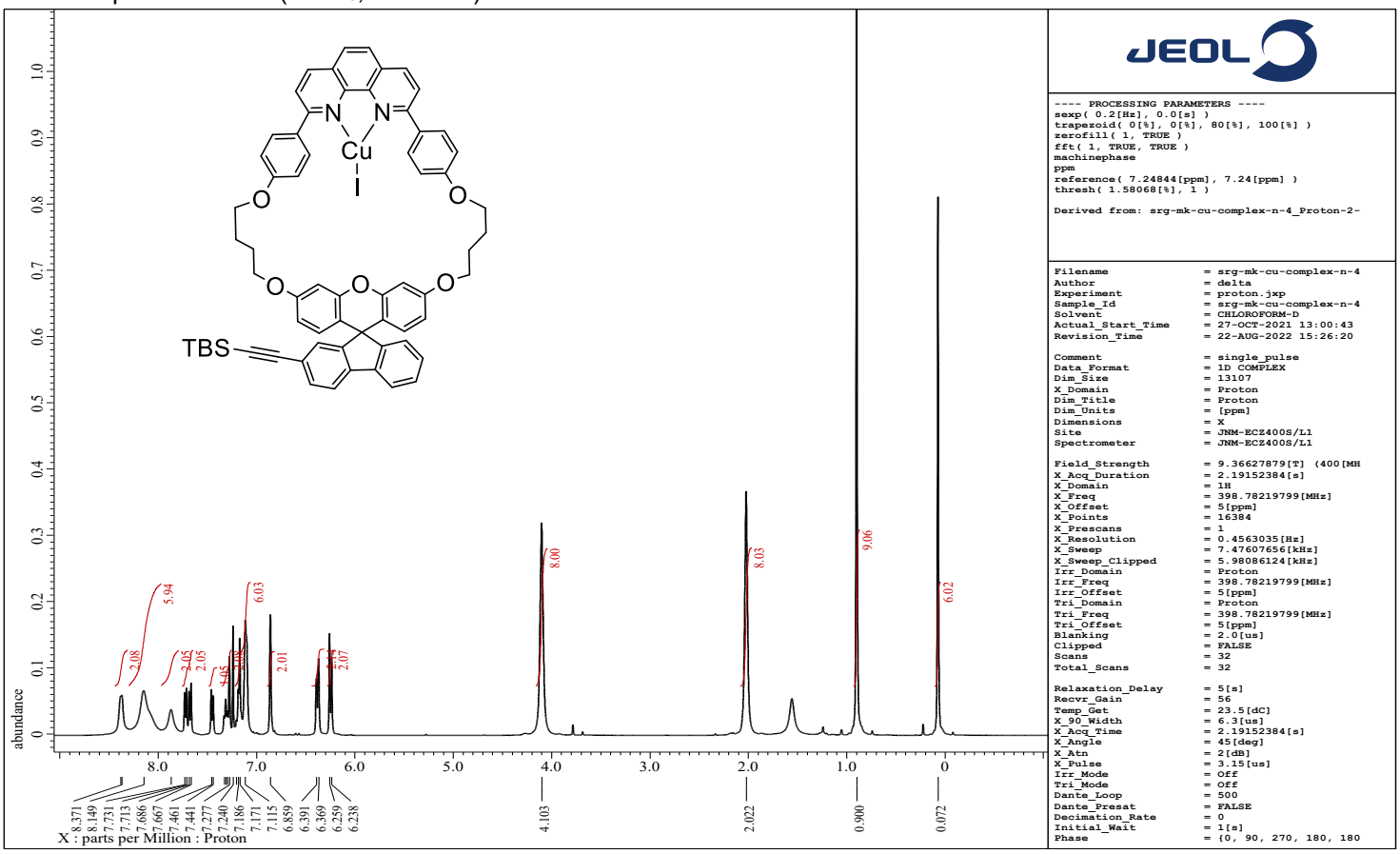
^{1}H NMR Spectrum of **9B** ($CDCl_3$, 400 MHz).



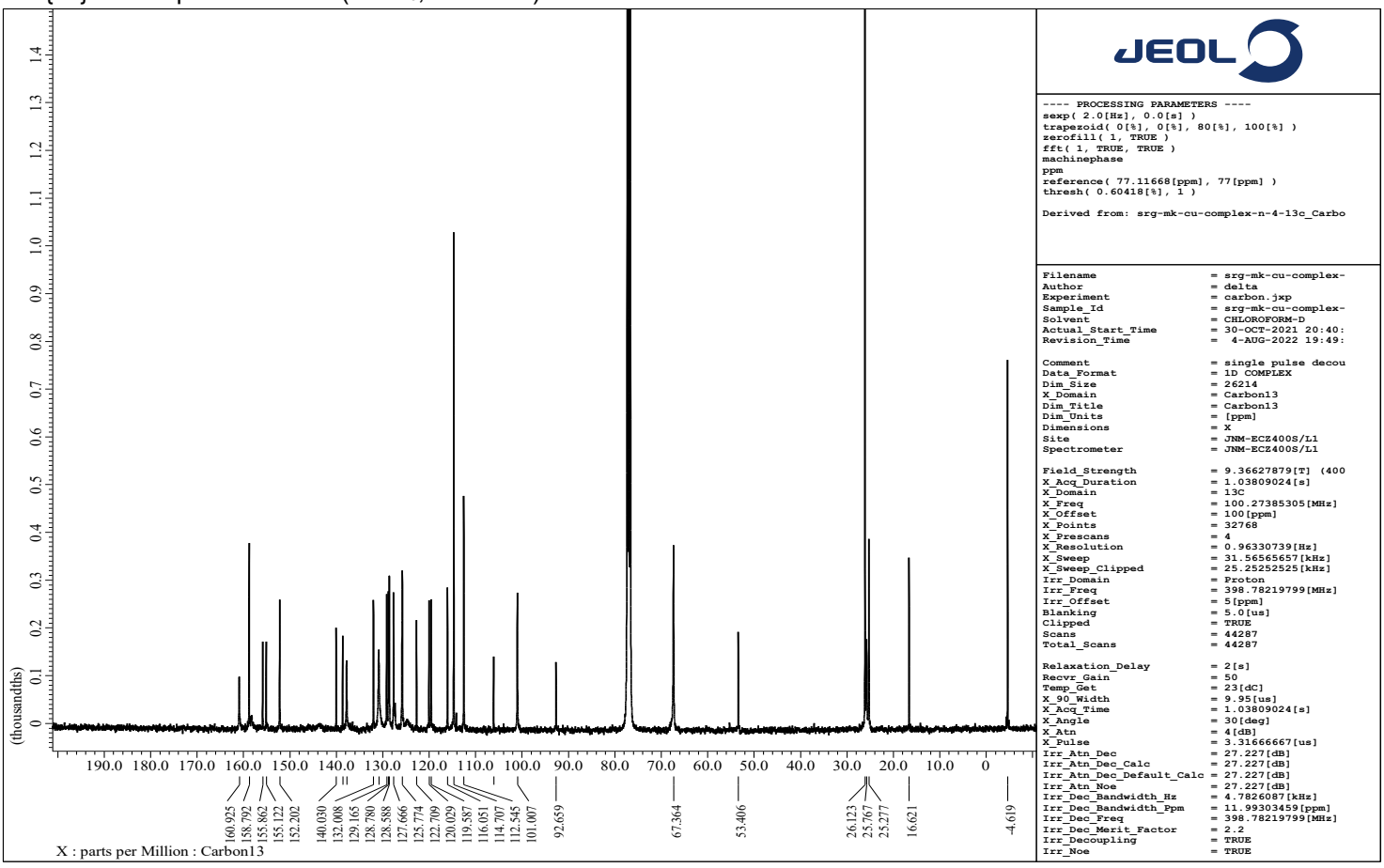
^{13}C {^{1}H} NMR Spectrum of **9B** ($CDCl_3$, 100 MHz).



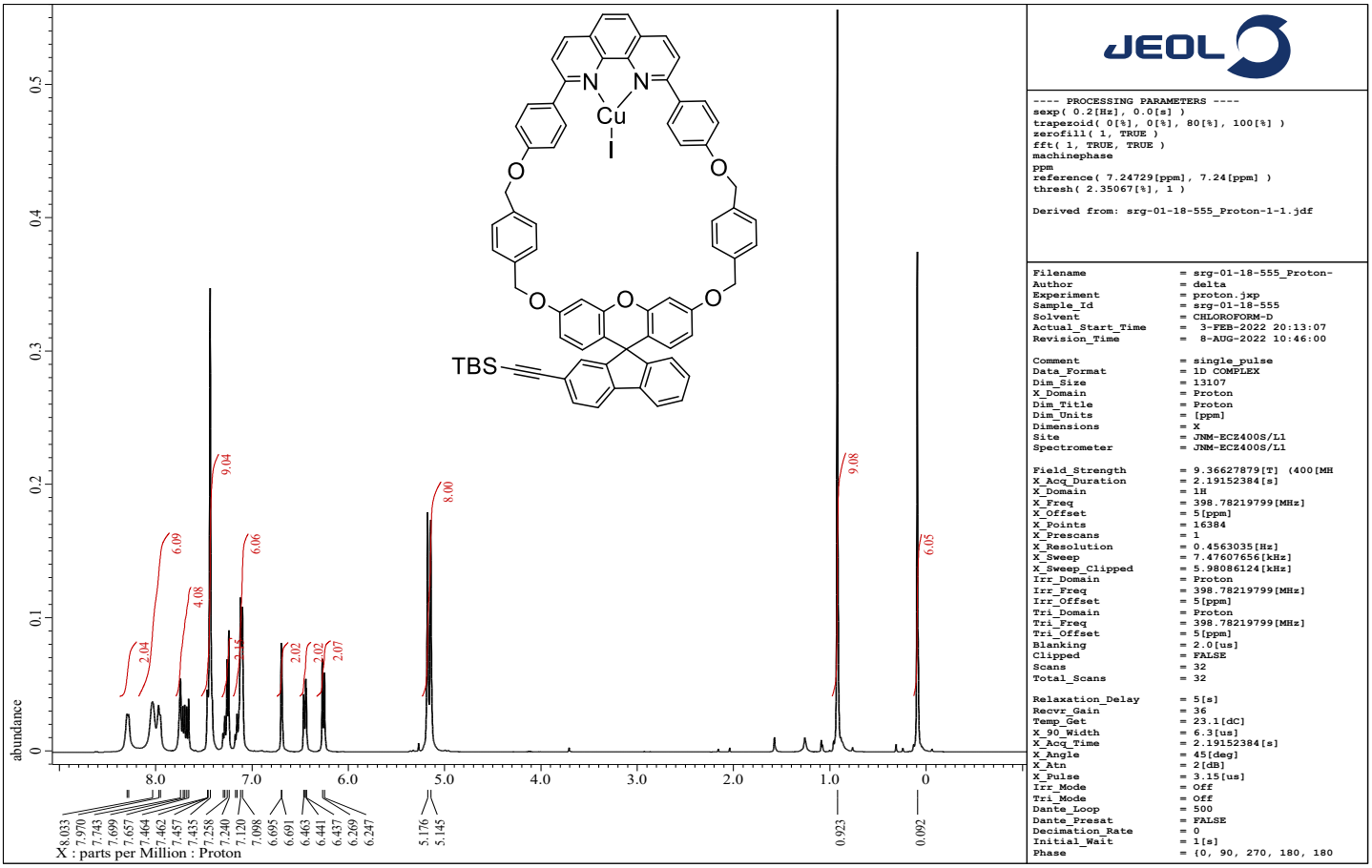
^{1}H NMR Spectrum of **9C** ($CDCl_3$, 400 MHz).



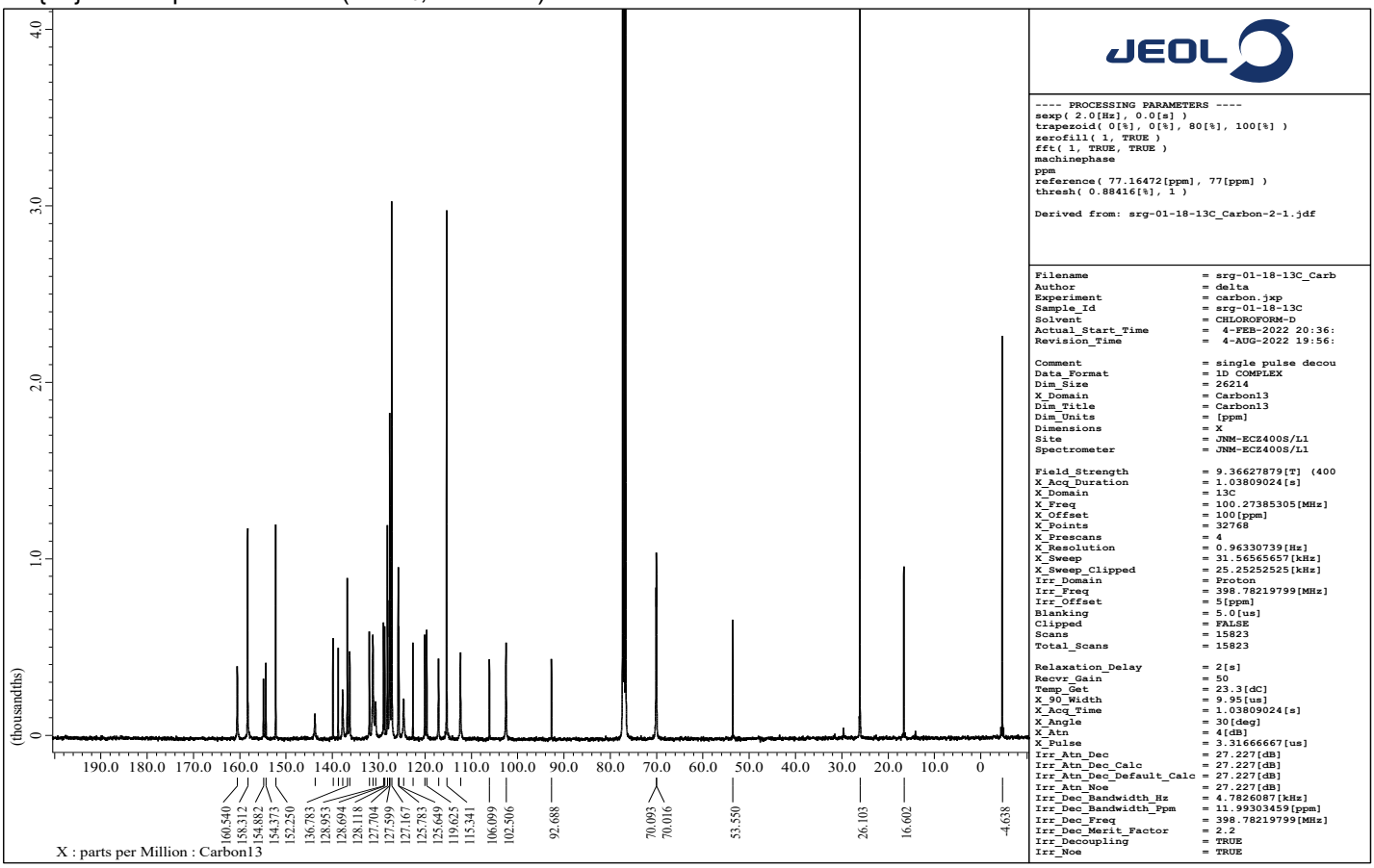
^{13}C {^{1}H} NMR Spectrum of **9C** ($CDCl_3$, 100 MHz).



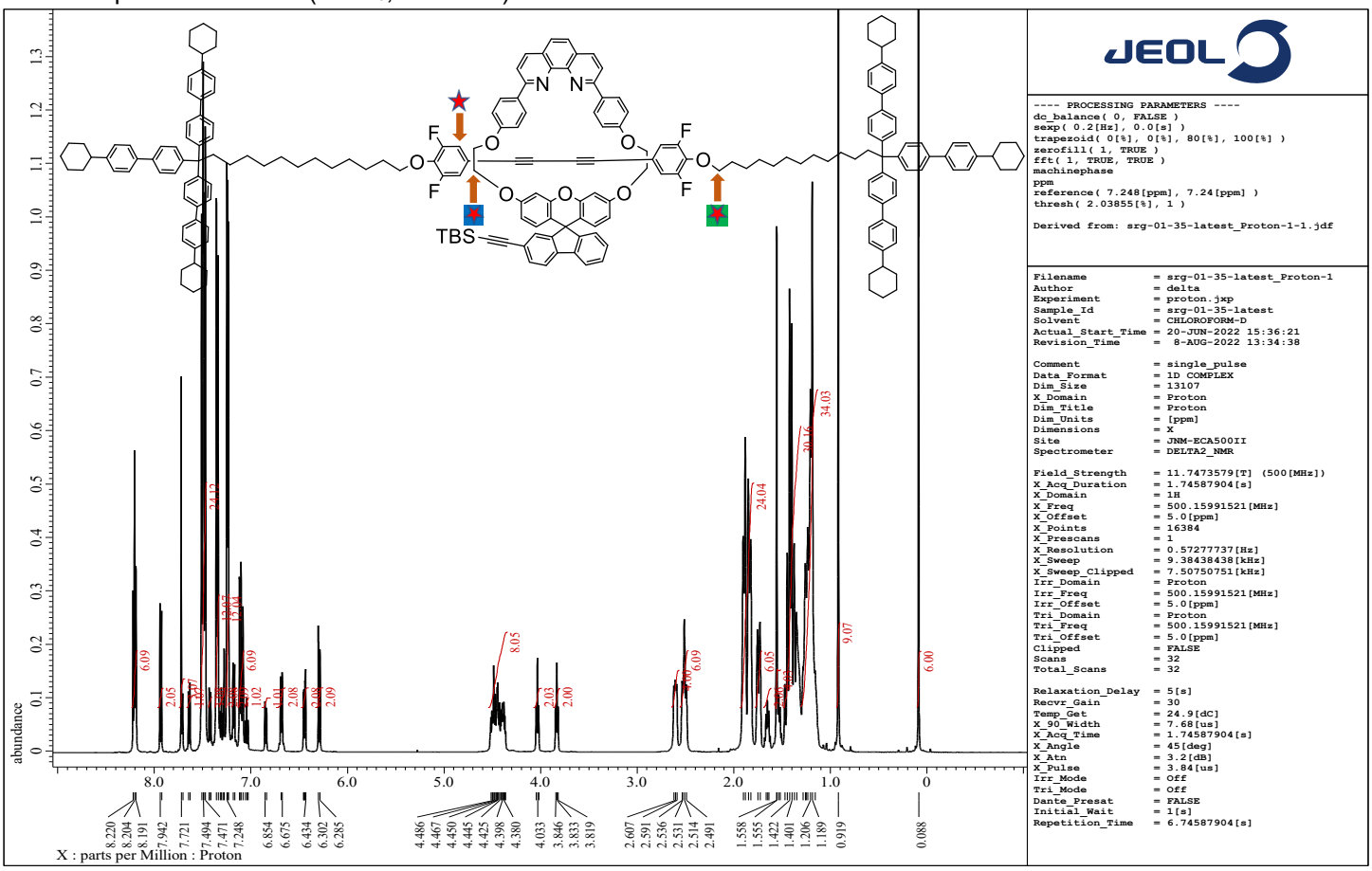
^{1}H NMR Spectrum of **9D** ($CDCl_3$, 400 MHz).



^{13}C {^{1}H} NMR Spectrum of **9D** ($CDCl_3$, 100 MHz).



^{1}H NMR Spectrum of **10Ac** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 2.03855[%], 1 )

Derived from: srg-01-35-latest_Proton-1-1.jdf
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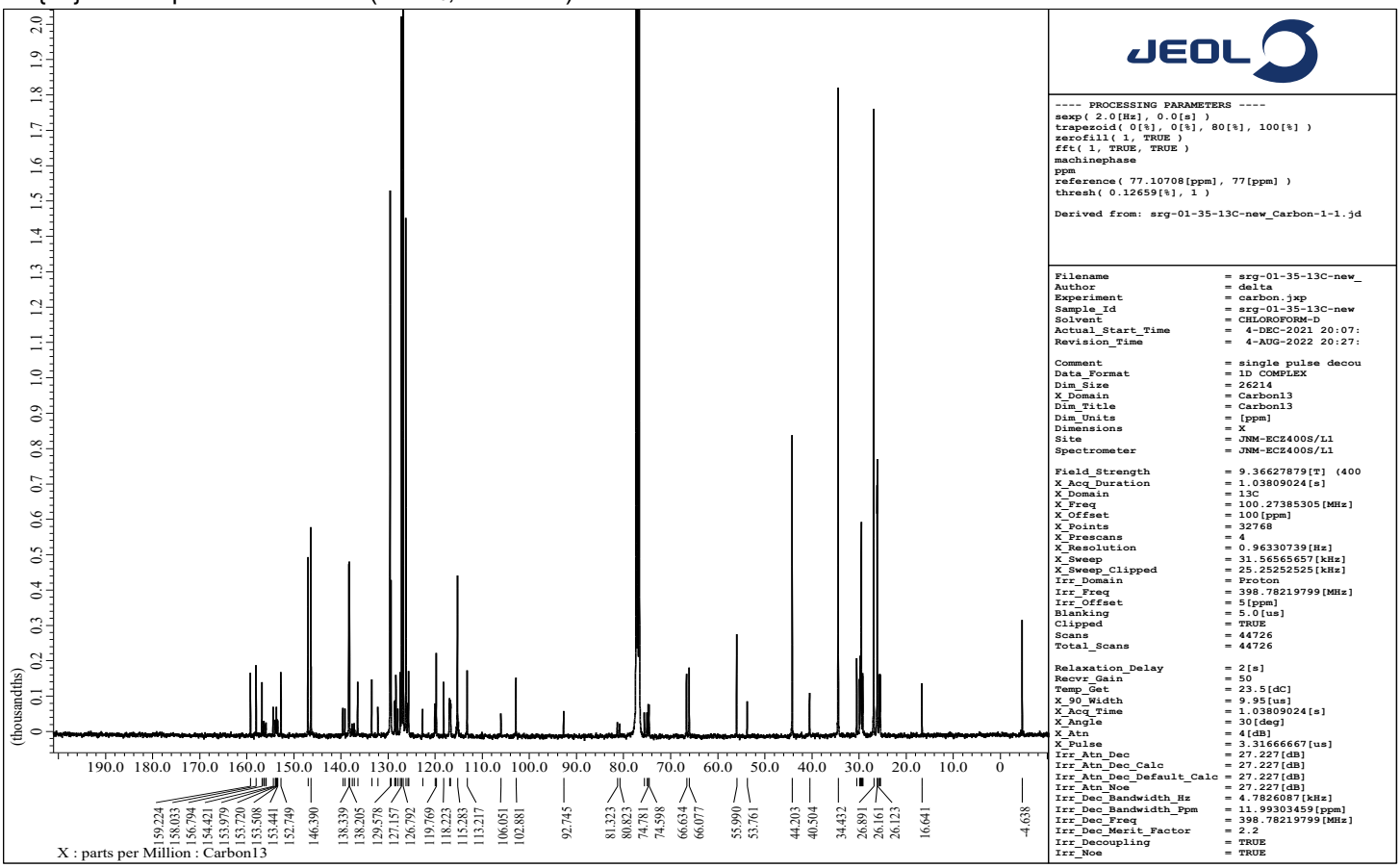
Filename	= srg-01-35-latest_Proton-1
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-35-latest
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JUN-2022 15:36:21
Revision_Time	= 8-AUG-2022 13:34:38
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 30
Temp_Get	= 24.9[dC]
X_90_Width	= 7.68[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 3.2[dB]
X_Pulse	= 3.84[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.74587904[s]

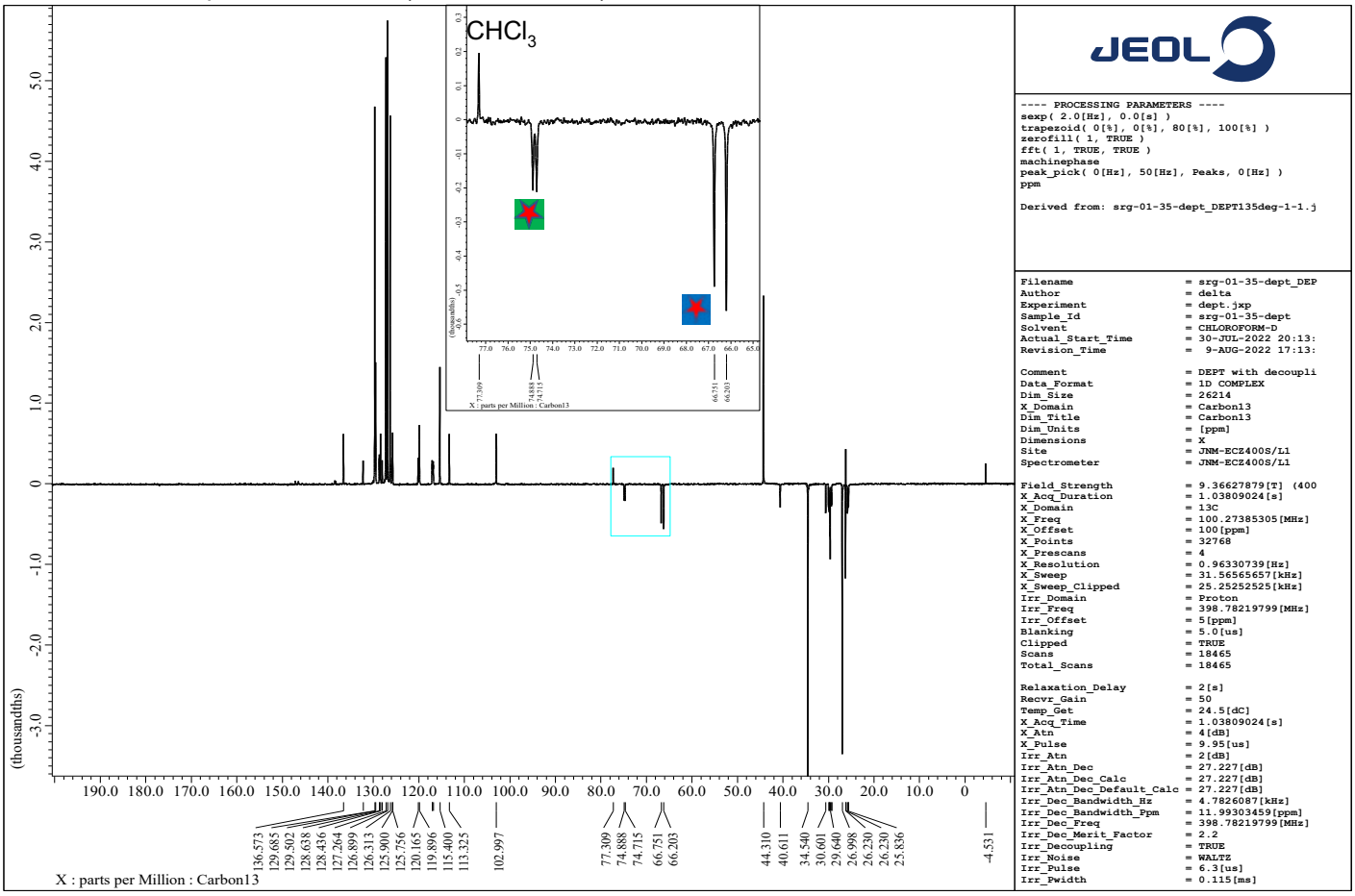
^{13}C {^{1}H} NMR Spectrum of **10Ac** ($CDCl_3$, 100 MHz).



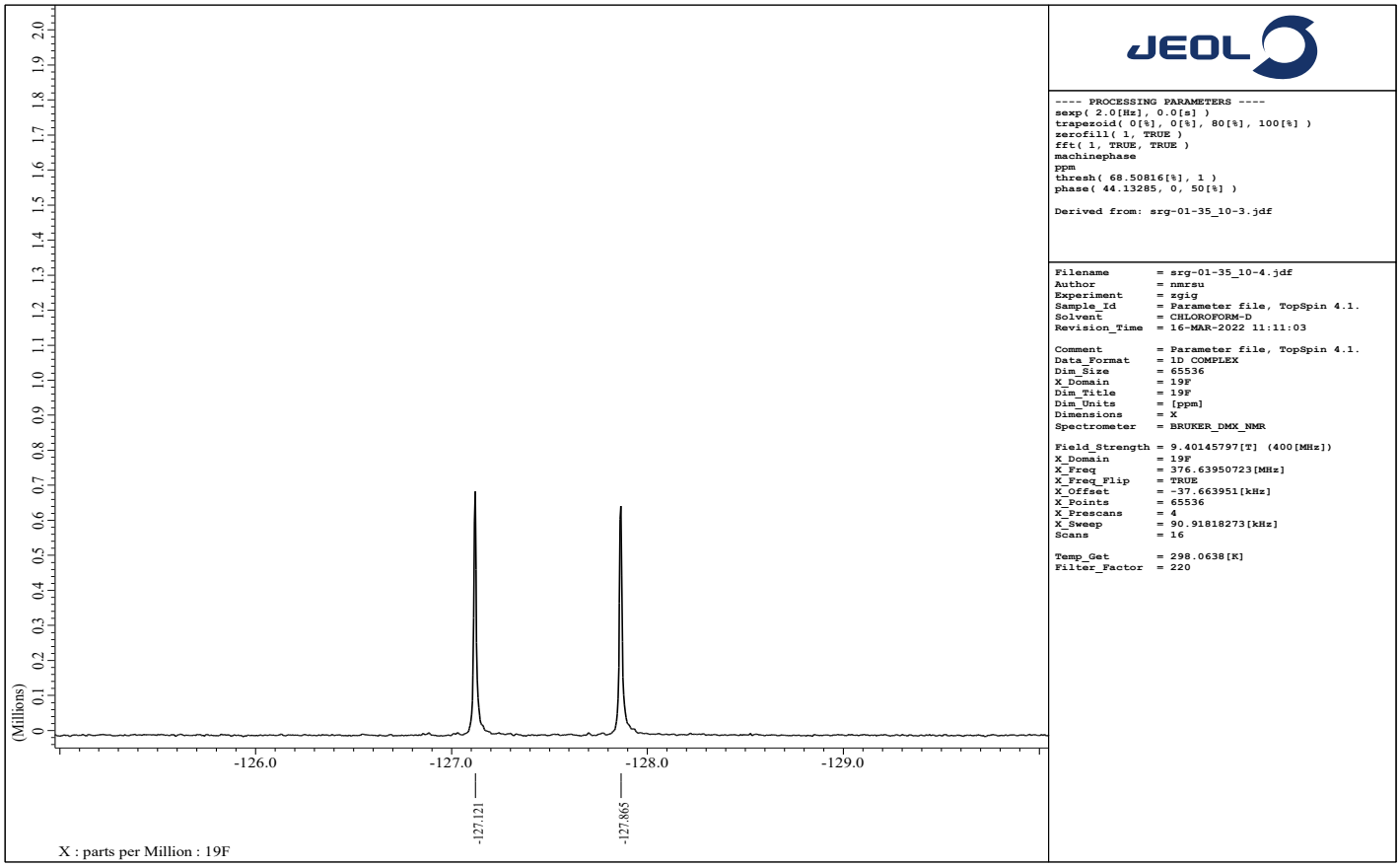

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.10708[ppm], 77[ppm] )
thresh( 0.12659[%], 1 )

Derived from: srg-01-35-13C-new_Carbon-1-1.jd
```

Filename	= srg-01-35-13C-new_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-35-13C-new
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 4-DEC-2021 20:07:
Revision_Time	= 4-AUG-2022 20:27:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 44726
Total_Scans	= 44726
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.5[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

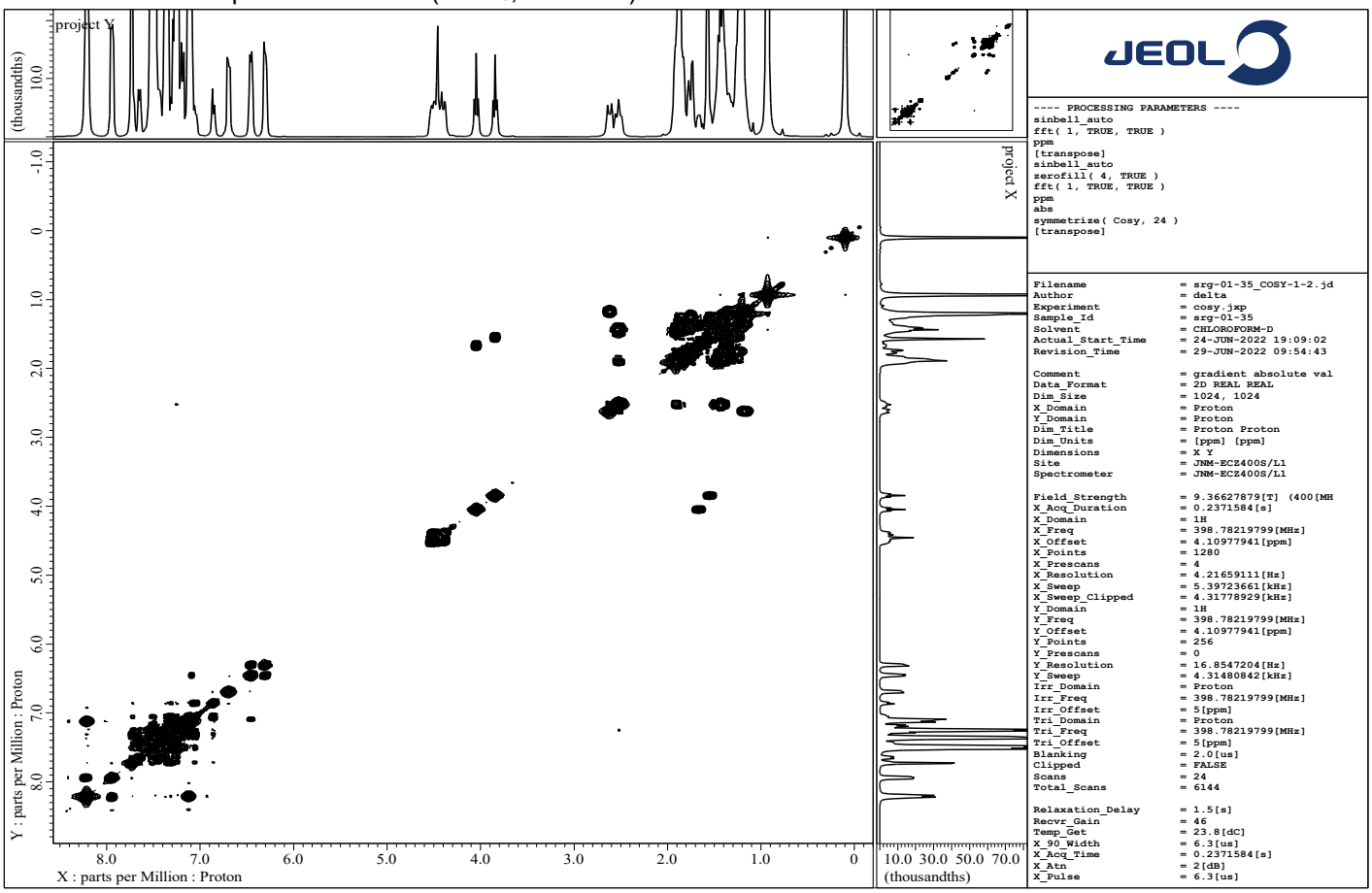
DEPT-135 NMR Spectrum of **10Ac** ($CDCl_3$, 100 MHz).



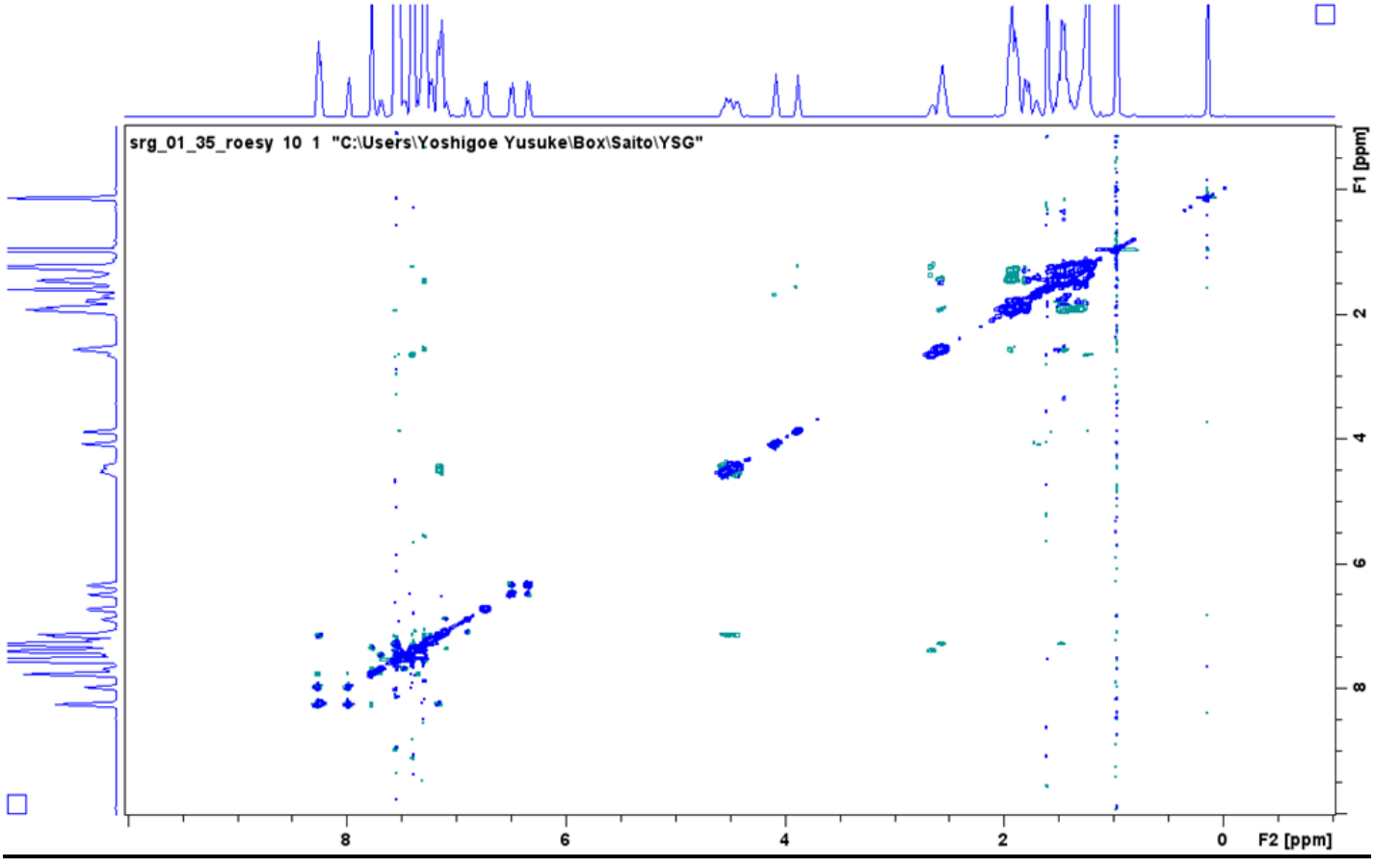
^{19}F NMR Spectrum of **10Ac** ($CDCl_3$, 377 MHz).



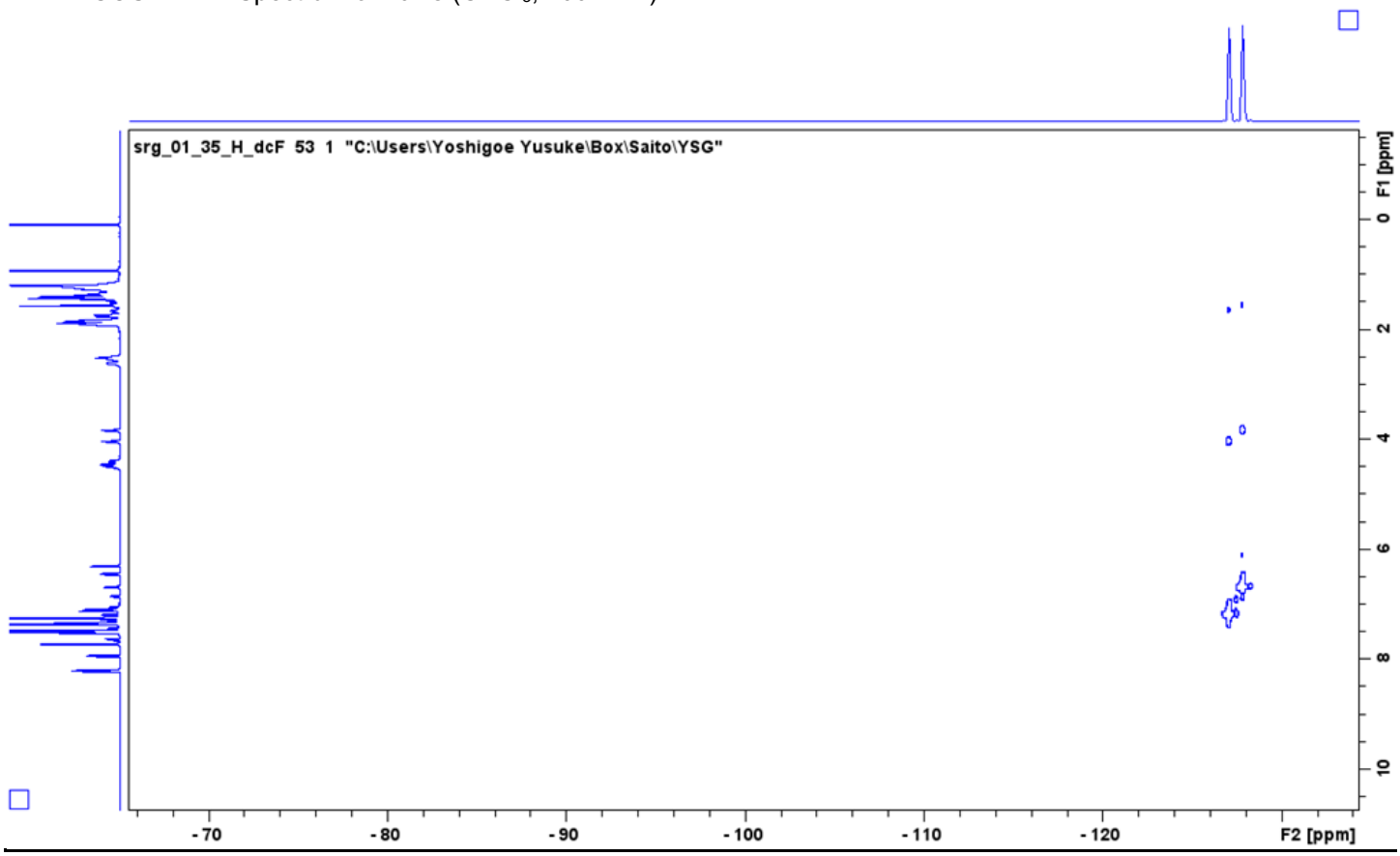
^{1}H-^{1}H COSY NMR Spectrum of **10Ac** ($CDCl_3$, 400 MHz).



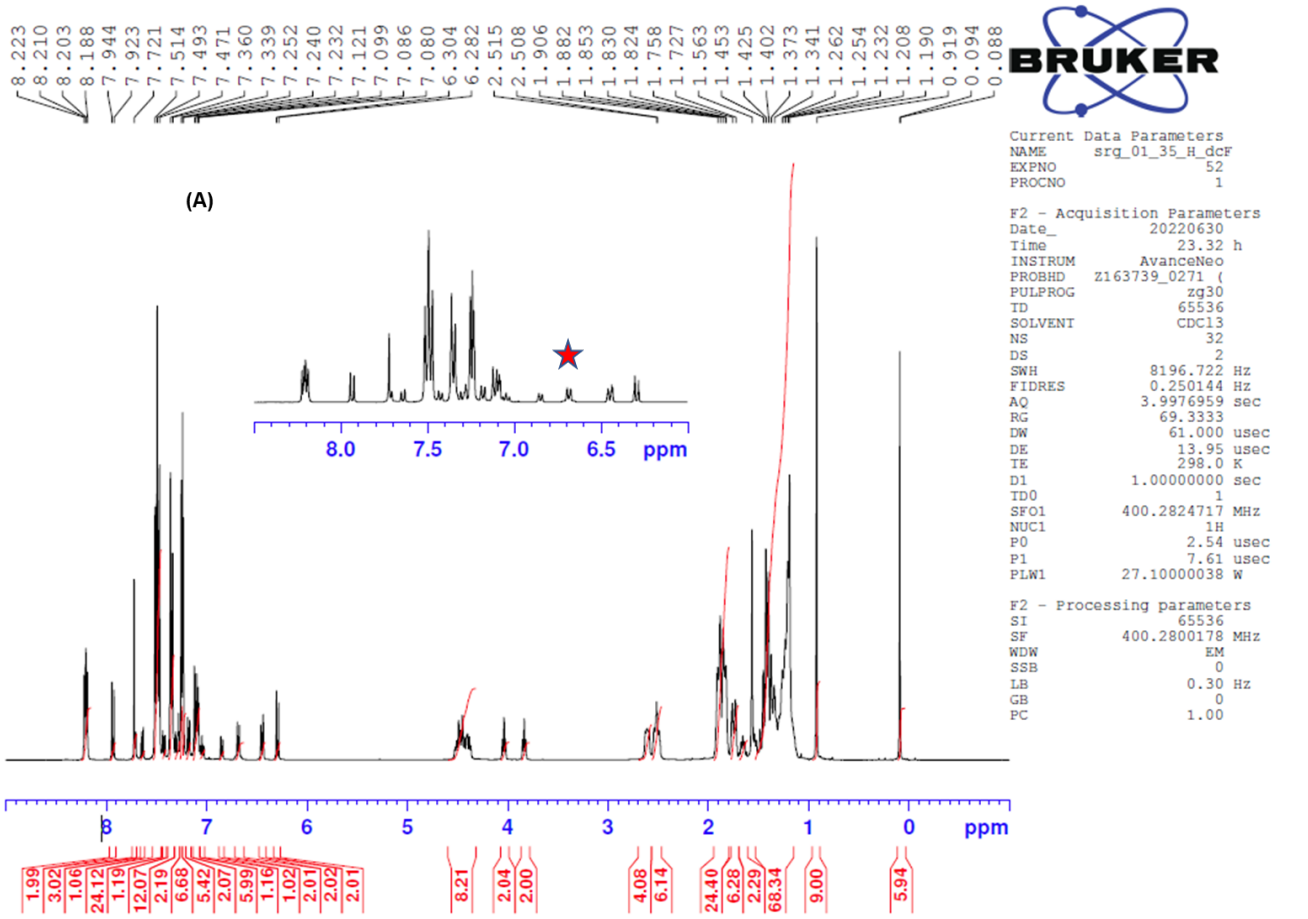
^{1}H-^{1}H ROESY NMR Spectrum of **10Ac** ($CDCl_3$, 400 MHz).

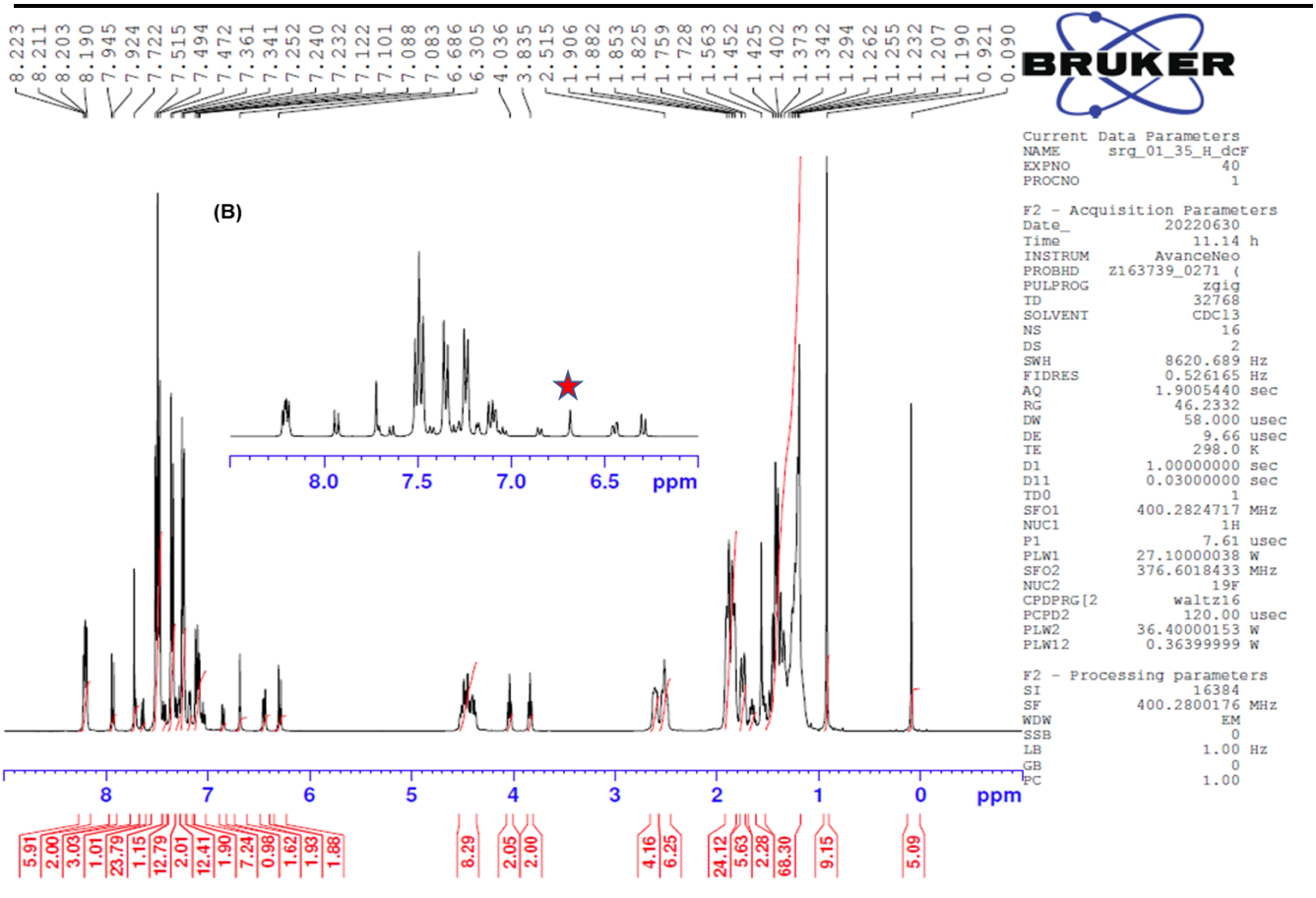


^{1}H-^{19}F-COSY NMR Spectrum of **10Ac** ($CDCl_3$, 400 MHz).

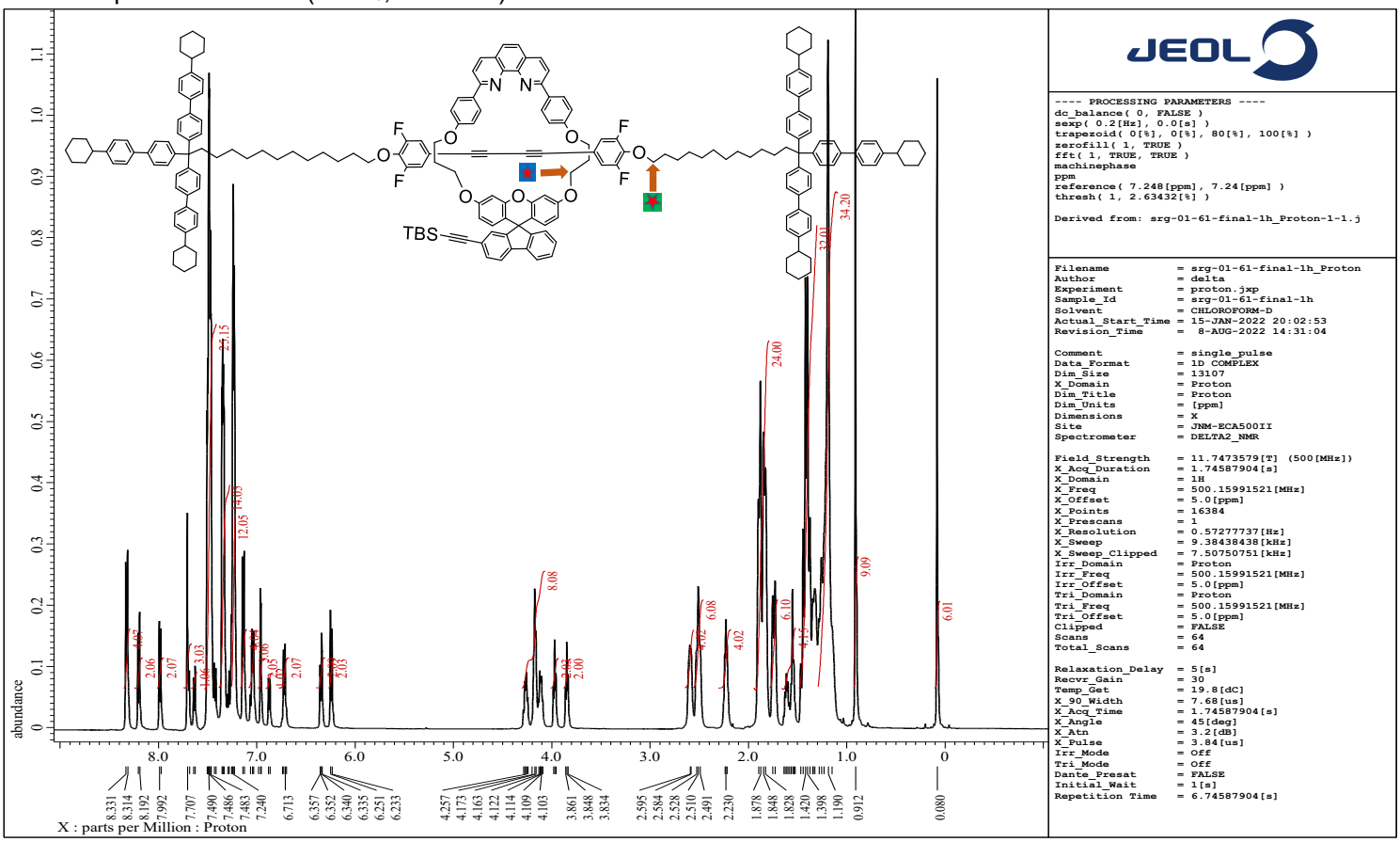


Comparative Normal **(A)** and ^{1}H and ^{1}H-^{19}F **(B)** decoupled NMR Spectrum of **10Ac** ($CDCl_3$, 400 MHz).





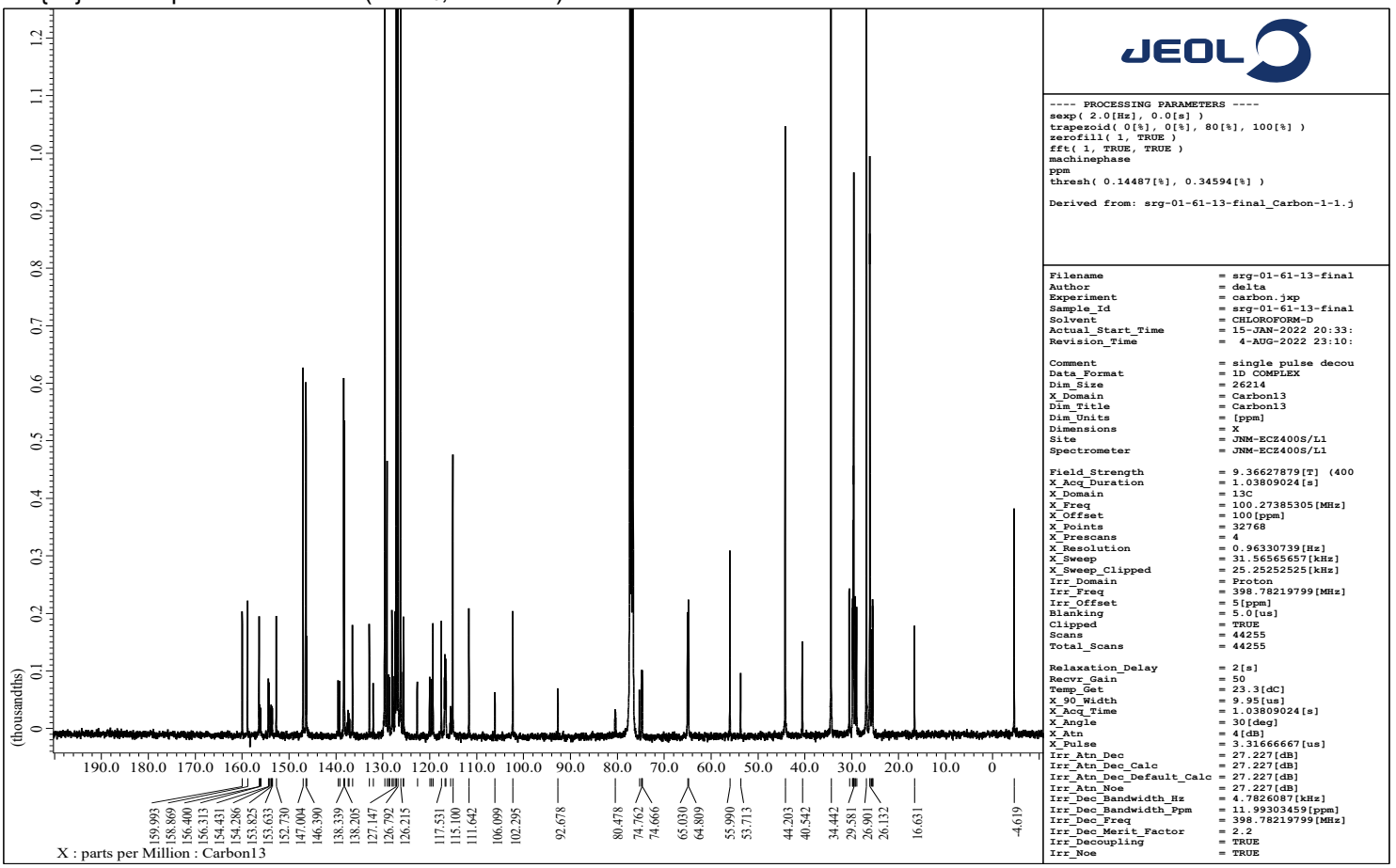
^{1}H NMR Spectrum of **10Bc** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 1, 2.63432[%] )

Derived from: srg-01-61-final-1h_Proton-1-1.j
```

Filename	= srg-01-61-final-1h_Proton
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-61-final-1h
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 15-JAN-2022 20:02:53
Revision_Time	= 8-AUG-2022 14:31:04
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 5[s]
Recvr_Gain	= 30
Temp_Get	= 19.8[dC]
X_90_Width	= 7.68[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 3.2[dB]
X_Pulse	= 3.84[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition Time	= 6.74587904[s]

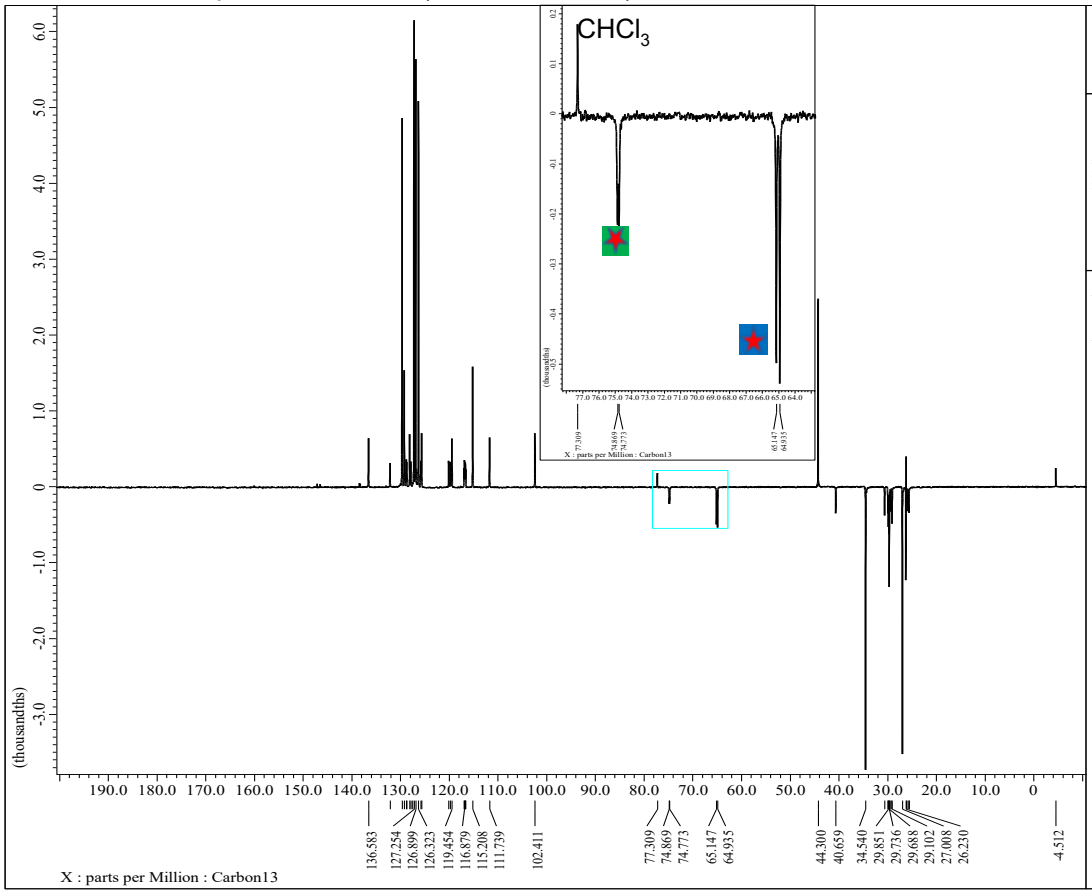
^{13}C {^{1}H} NMR Spectrum of **10Bc** ($CDCl_3$, 100 MHz).

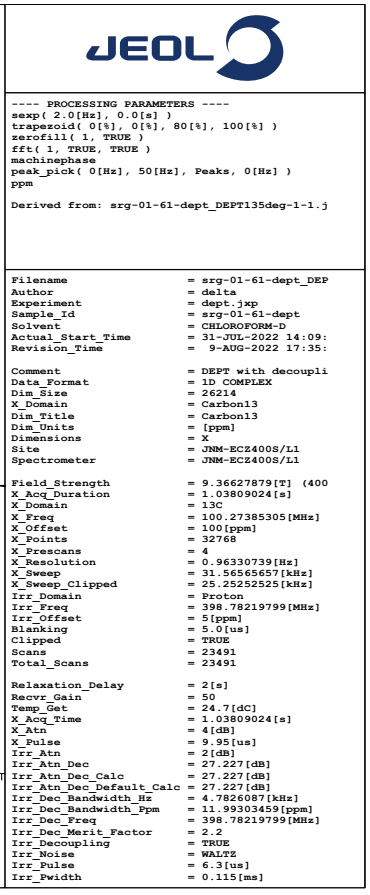



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 0.14487[%], 0.34594[%] )

Derived from: srg-01-61-13-final_Carbon-1-1.j
```

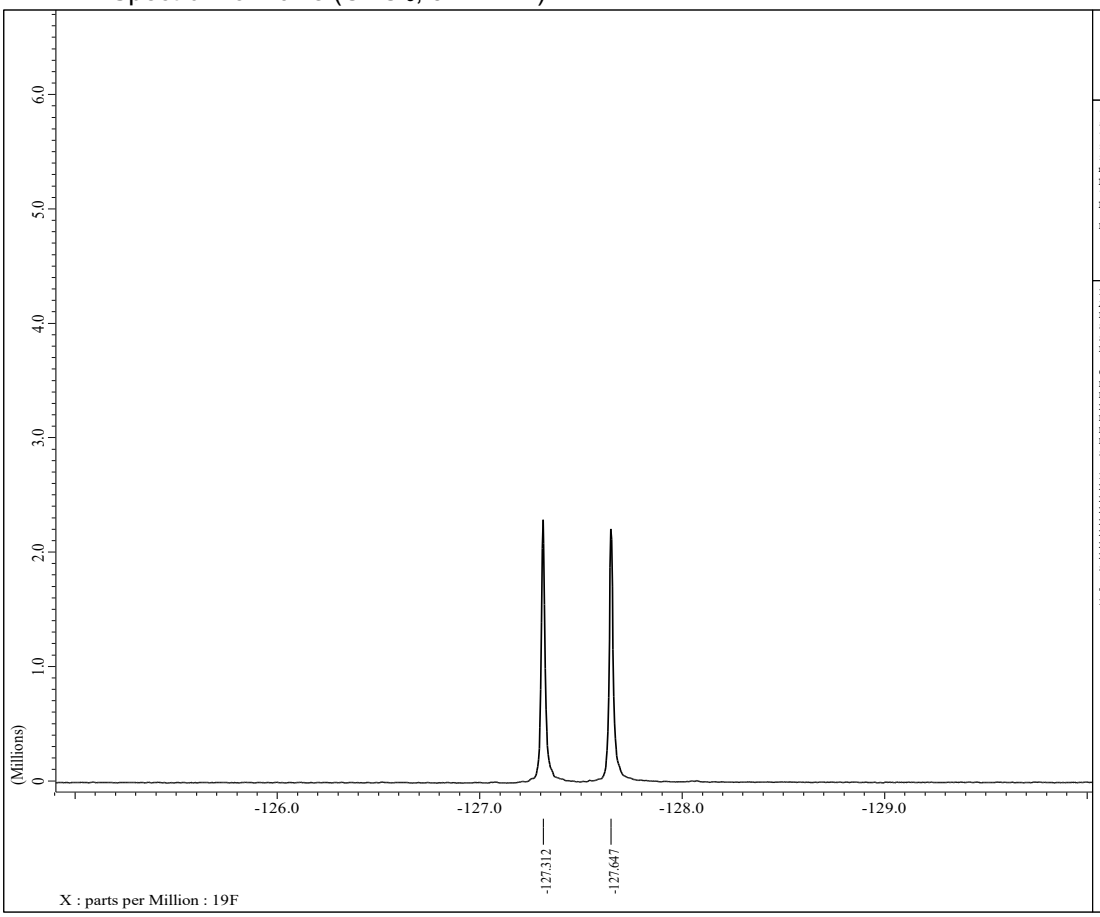
Filename	= srg-01-61-13-final
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-61-13-final
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 15-JAN-2022 20:33:
Revision_Time	= 4-AUG-2022 23:10:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 44255
Total_Scans	= 44255
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.3[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

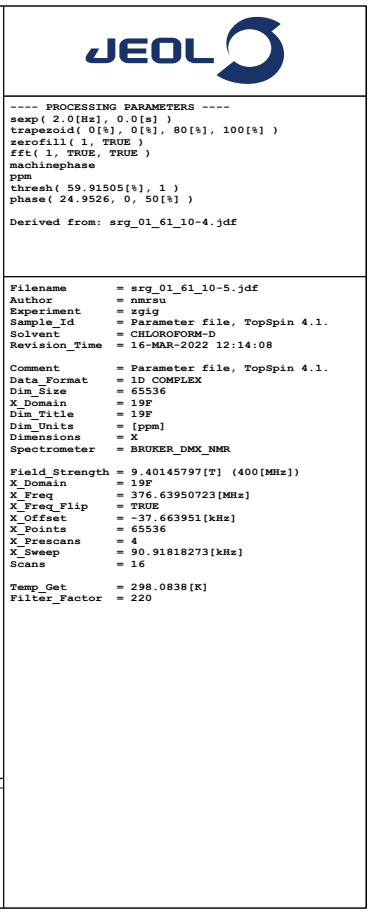
DEPT-135 NMR Spectrum of **10Bc** ($CDCl_3$, 100 MHz).



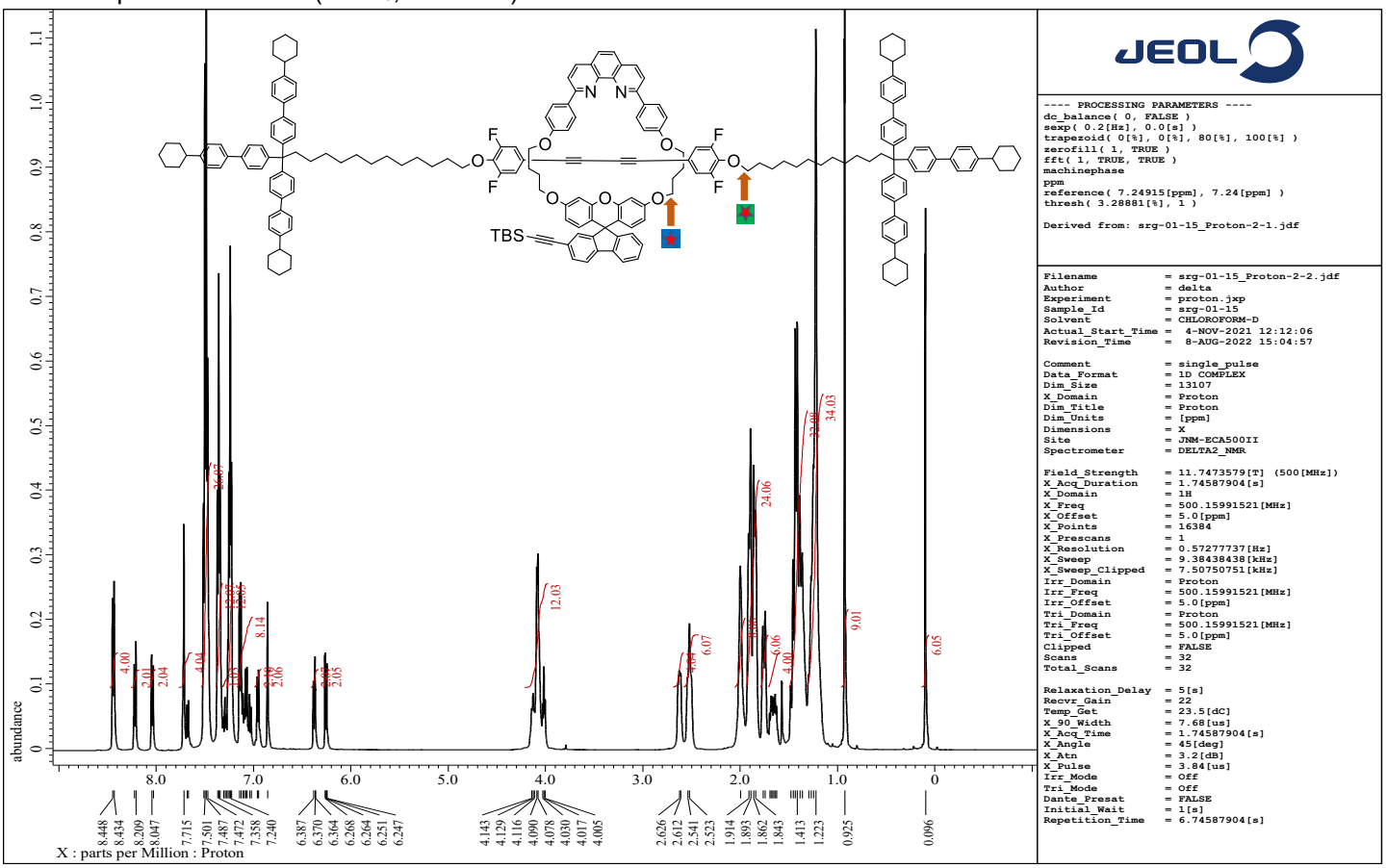


^{19}F NMR Spectrum of **10Bc** ($CDCl_3$, 377 MHz).





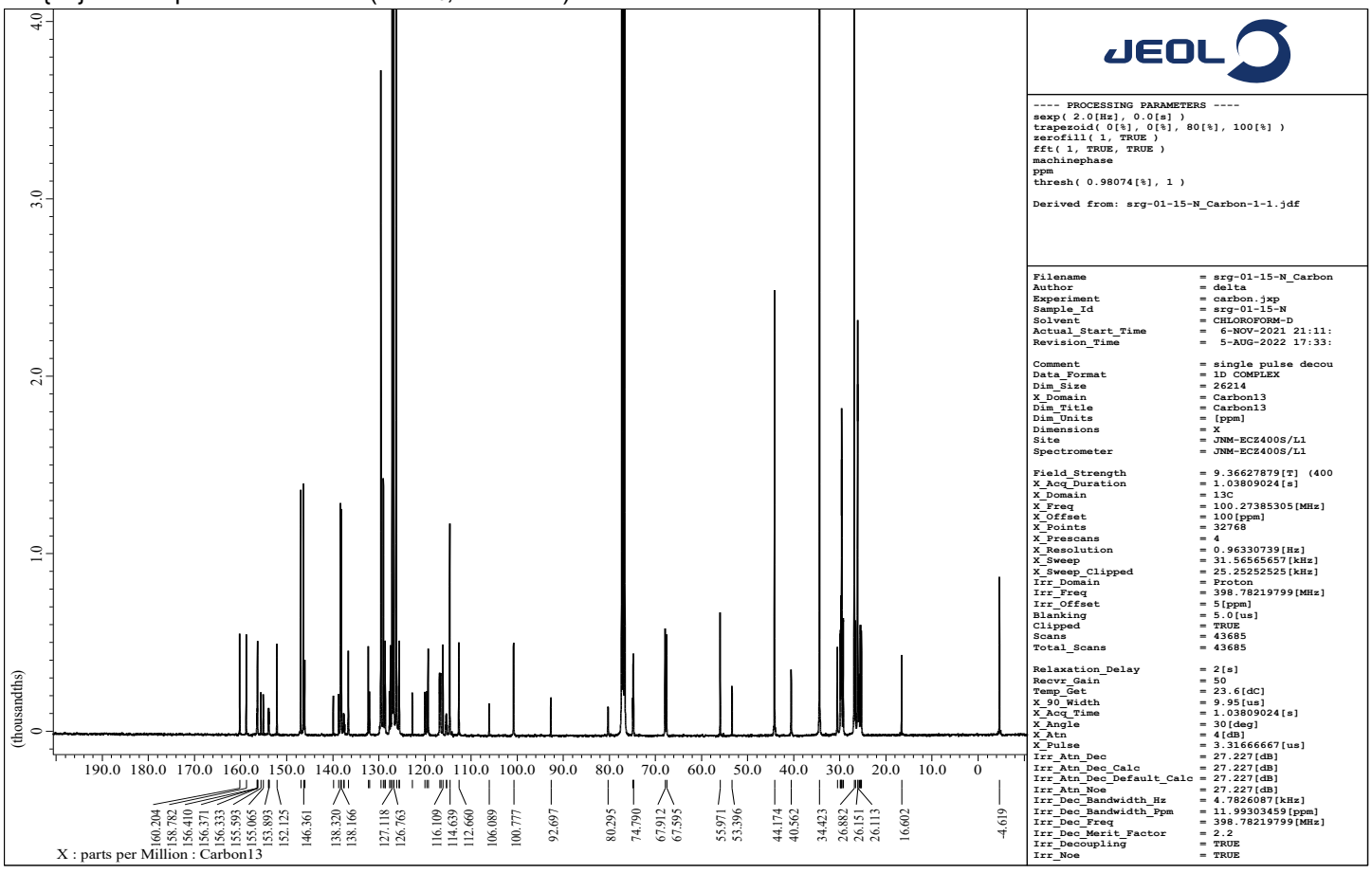
^{1}H NMR Spectrum of **10Cc** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24915[ppm], 7.24[ppm] )
thresh( 3.28881[%], 1 )

Derived from: srg-01-15_Proton-2-1.jdf
```

```
Filename            = srg-01-15_Proton-2-2.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = srg-01-15
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-NOV-2021 12:12:06
Revision_Time       =  8-AUG-2022 15:04:57

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 32
Total_Scans         = 32

Relaxation_Delay    = 5[s]
Recvr_Gain          = 22
Temp_Get            = 23.5[dC]
X_90_Width          = 7.68[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.2[dB]
X_Pulse             = 3.84[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

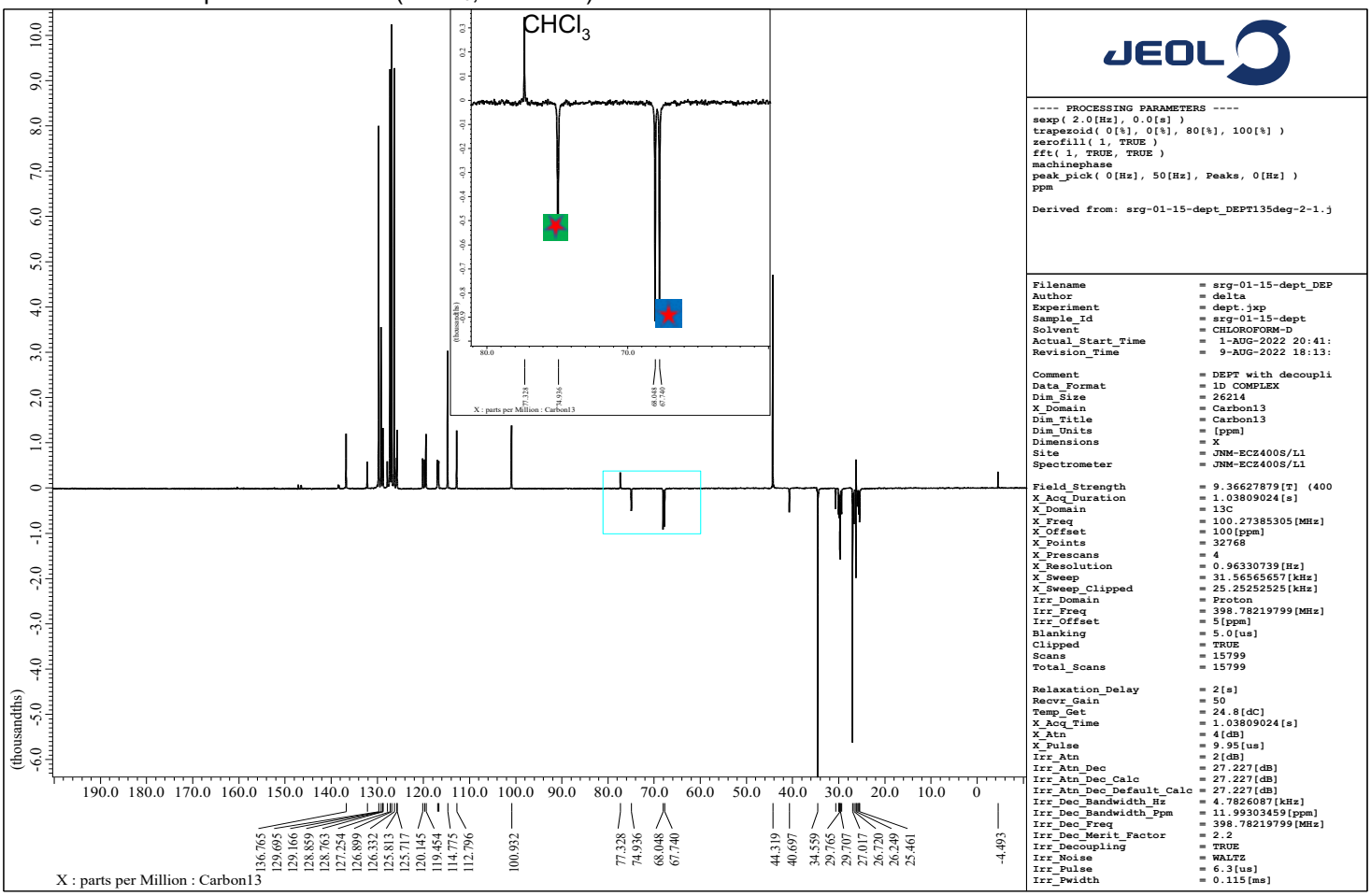
^{13}C {^{1}H} NMR Spectrum of **10Cc** ($CDCl_3$, 100 MHz).



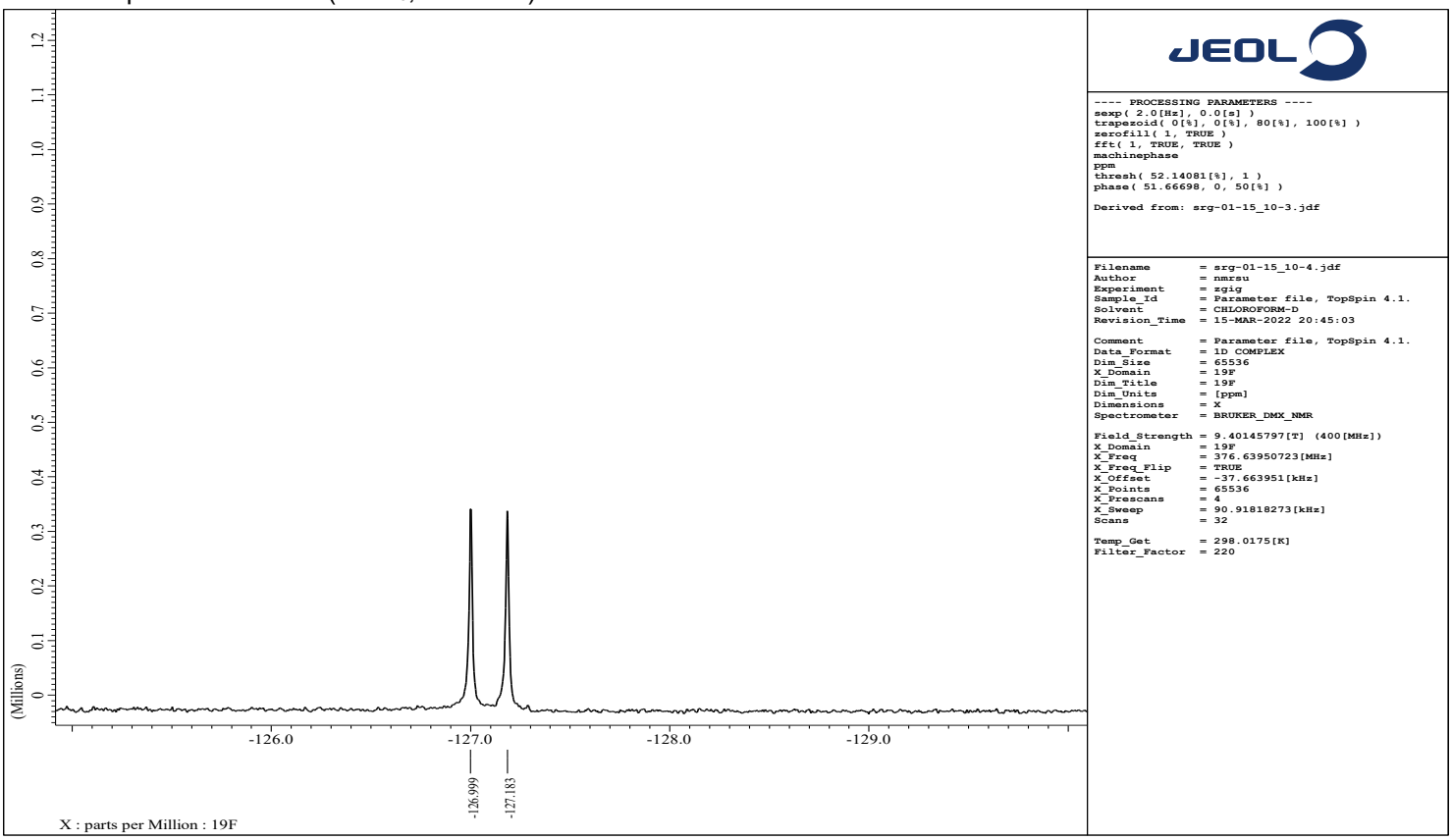

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 0.98074[%], 1 )

Derived from: srg-01-15-N_Carbon-1-1.jdf
```

```
Filename                = srg-01-15-N_Carbon
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = srg-01-15-N
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  6-NOV-2021 21:11:
Revision_Time           =  5-AUG-2022 17:33:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.27385305[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5.0[us]
Clipped                 = TRUE
Scans                   = 43685
Total_Scans             = 43685

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 23.6[dC]
X_90_Width              = 9.95[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 4[dB]
X_Pulse                 = 3.31666667[us]
Irr_Atn_Dec             = 27.227[dB]
Irr_Atn_Dec_Calc        = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe             = 27.227[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.99303459[ppm]
Irr_Dec_Freq            = 398.78219799[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
```

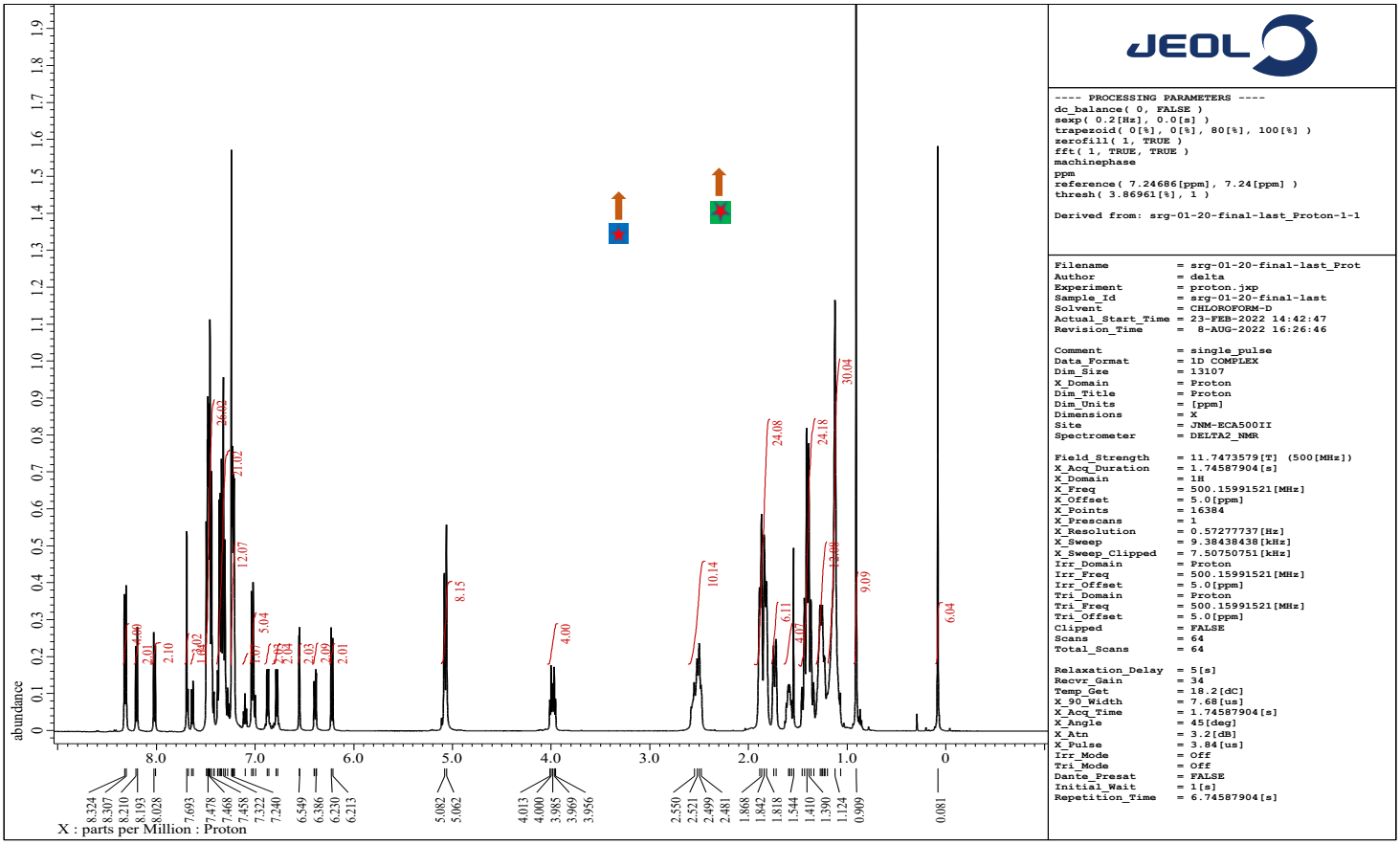
DEPT-135 NMR Spectrum of **10Cc** ($CDCl_3$, 100 MHz).



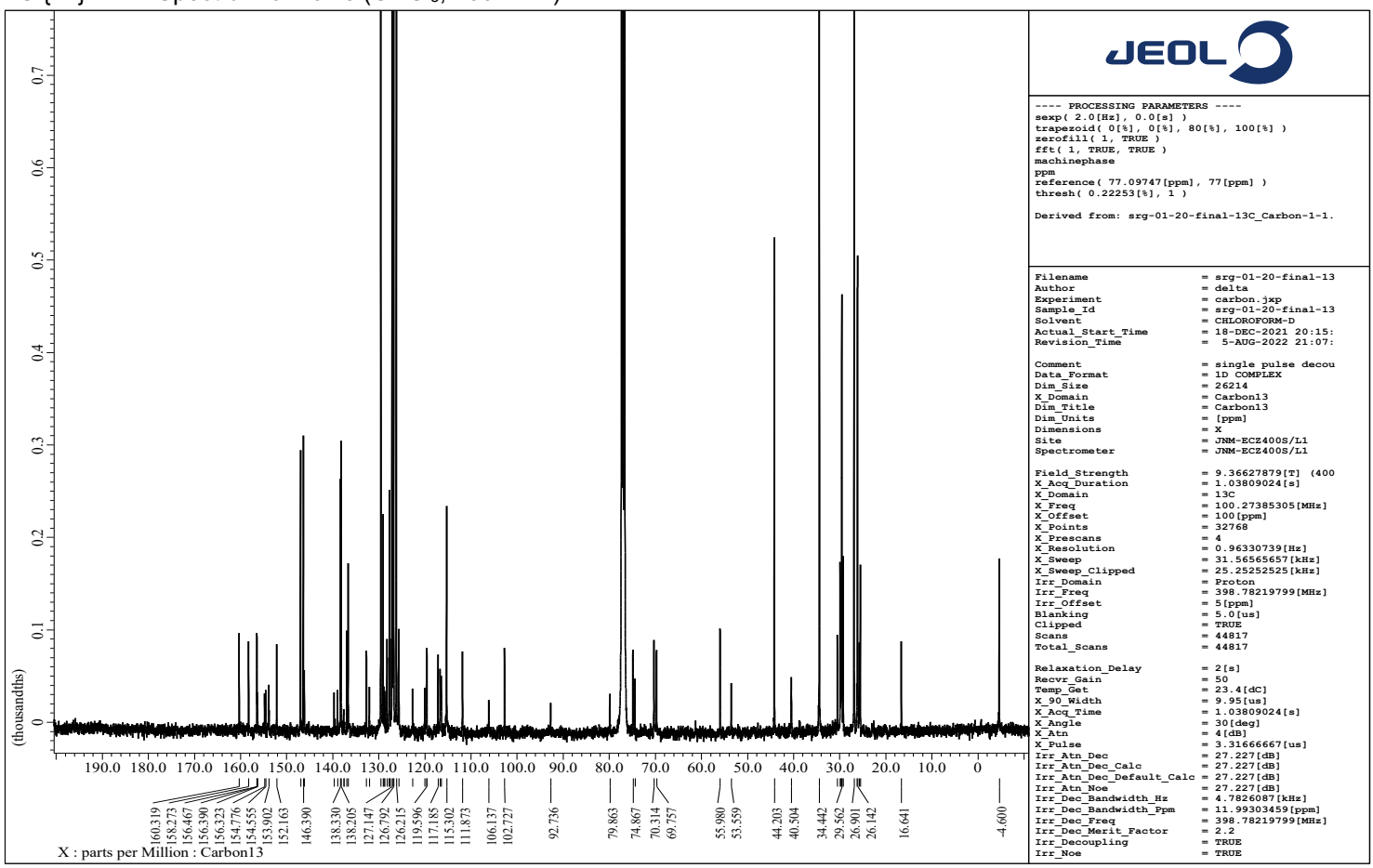
^{19}F NMR Spectrum of **10Cc** ($CDCl_3$, 377 MHz).



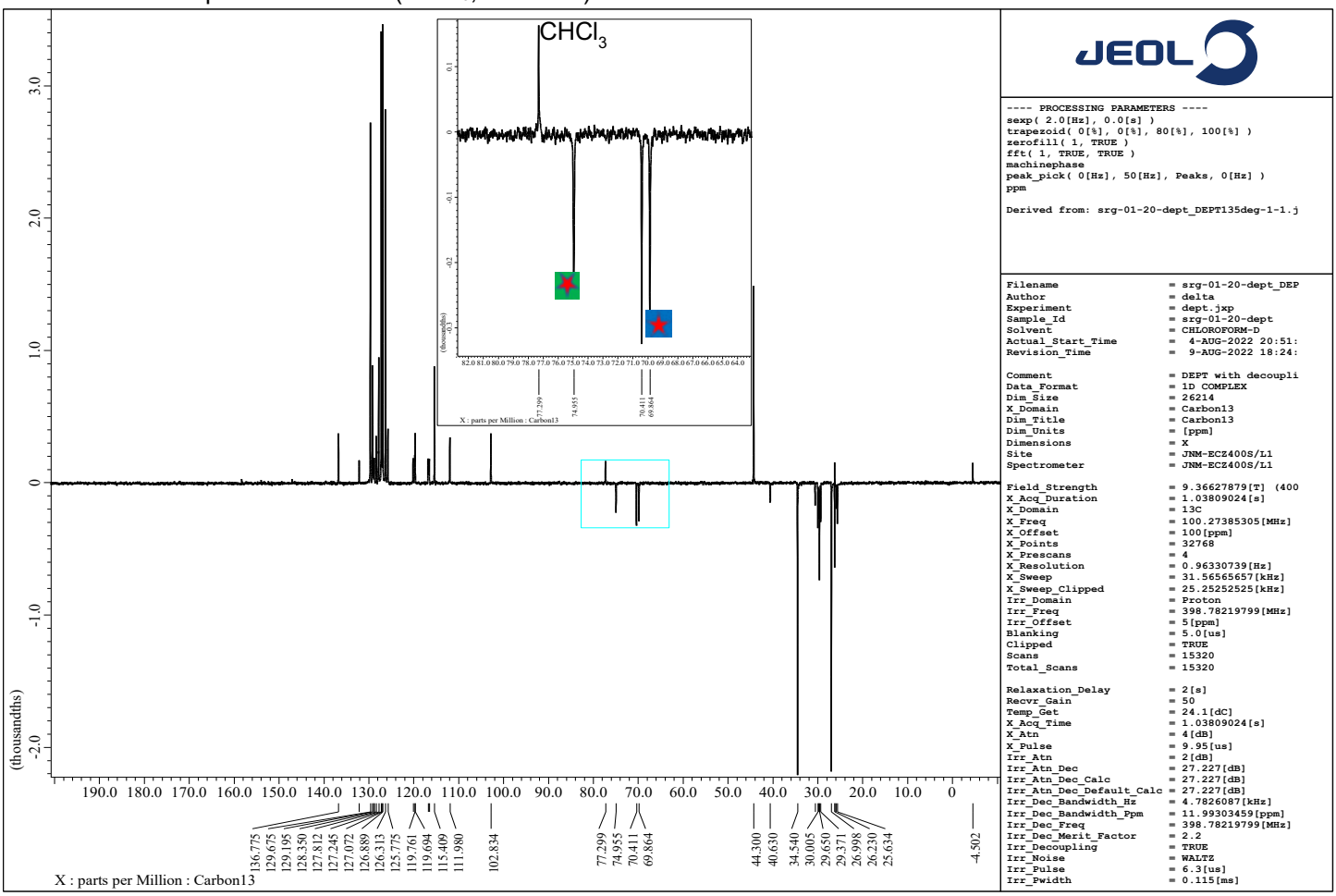
^{1}H NMR Spectrum of **10Dc** ($CDCl_3$, 500 MHz).



^{13}C {^{1}H} NMR Spectrum of **10Dc** ($CDCl_3$, 100 MHz).



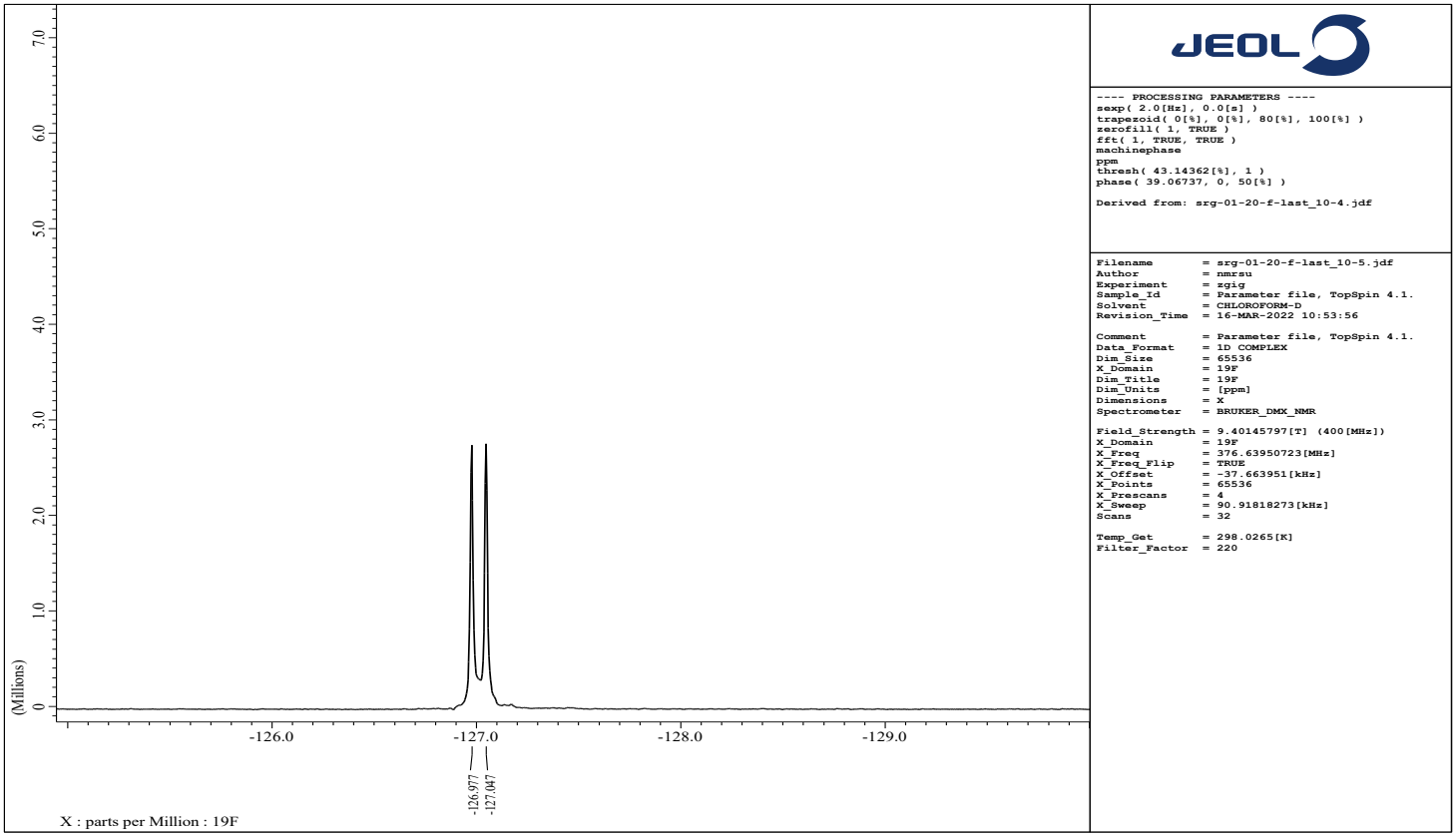
DEPT-135 NMR Spectrum of **10Dc** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
peak_pick( 0[Hz], 50[Hz], Peaks, 0[Hz] )
ppm

Derived from: srg-01-20-dept_DEPT135deg-1-1.j
```

Filename	= srg-01-20-dept_DEP
Author	= delta
Experiment	= dept.jxp
Sample_Id	= srg-01-20-dept
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 4-AUG-2022 20:51:
Revision_Time	= 9-AUG-2022 18:24:
Comment	= DEPT with decoupli
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 15320
Total_Scans	= 15320
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 24.1[dC]
X_Acq_Time	= 1.03809024[s]
X_Atn	= 4[dB]
X_Pulse	= 9.95[us]
Irr_Atn	= 2[dB]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noise	= WALTZ
Irr_Pulse	= 6.3[us]
Irr_Pwidth	= 0.115[ms]

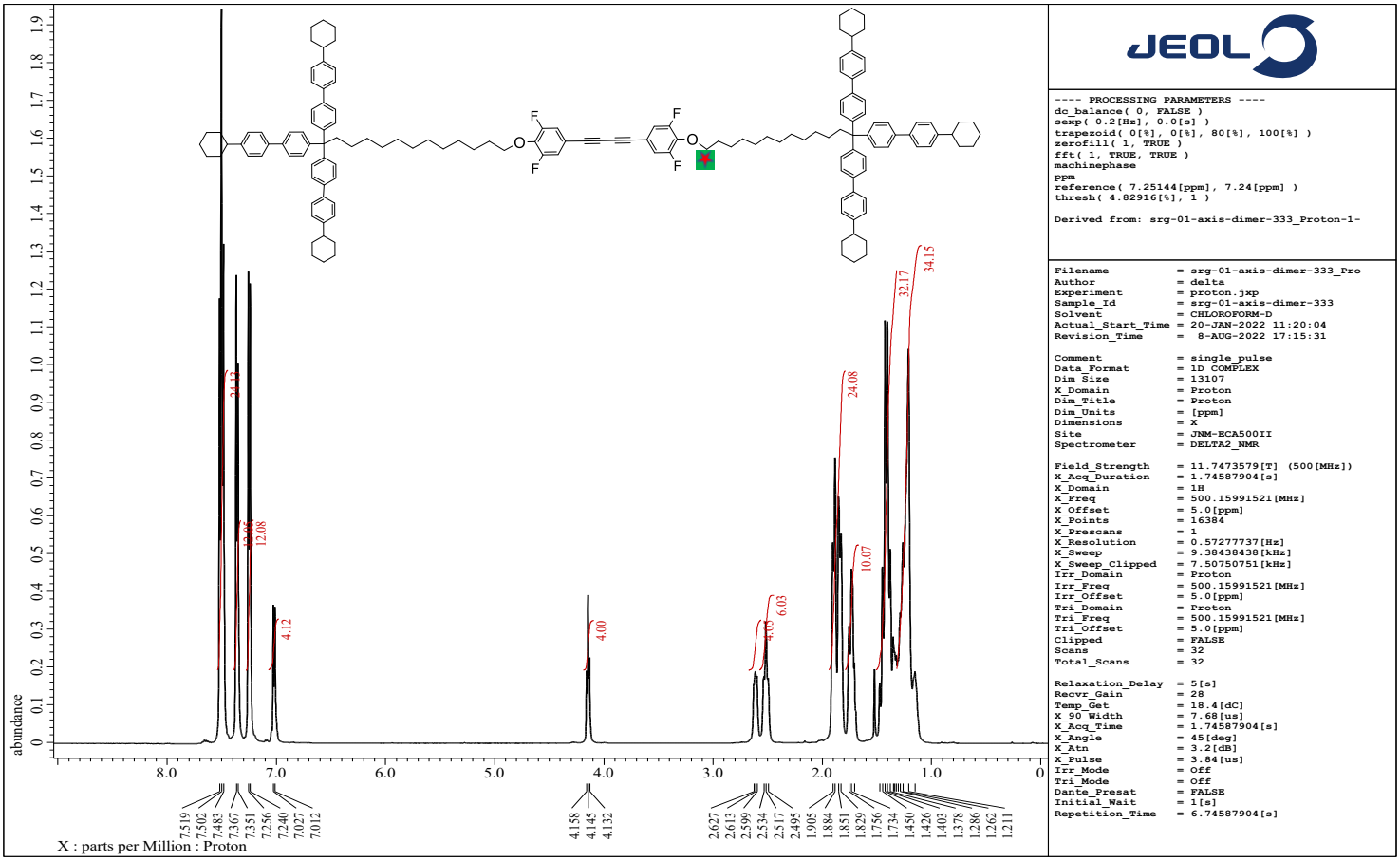
^{19}F NMR Spectrum of **10Dc** ($CDCl_3$, 377 MHz).



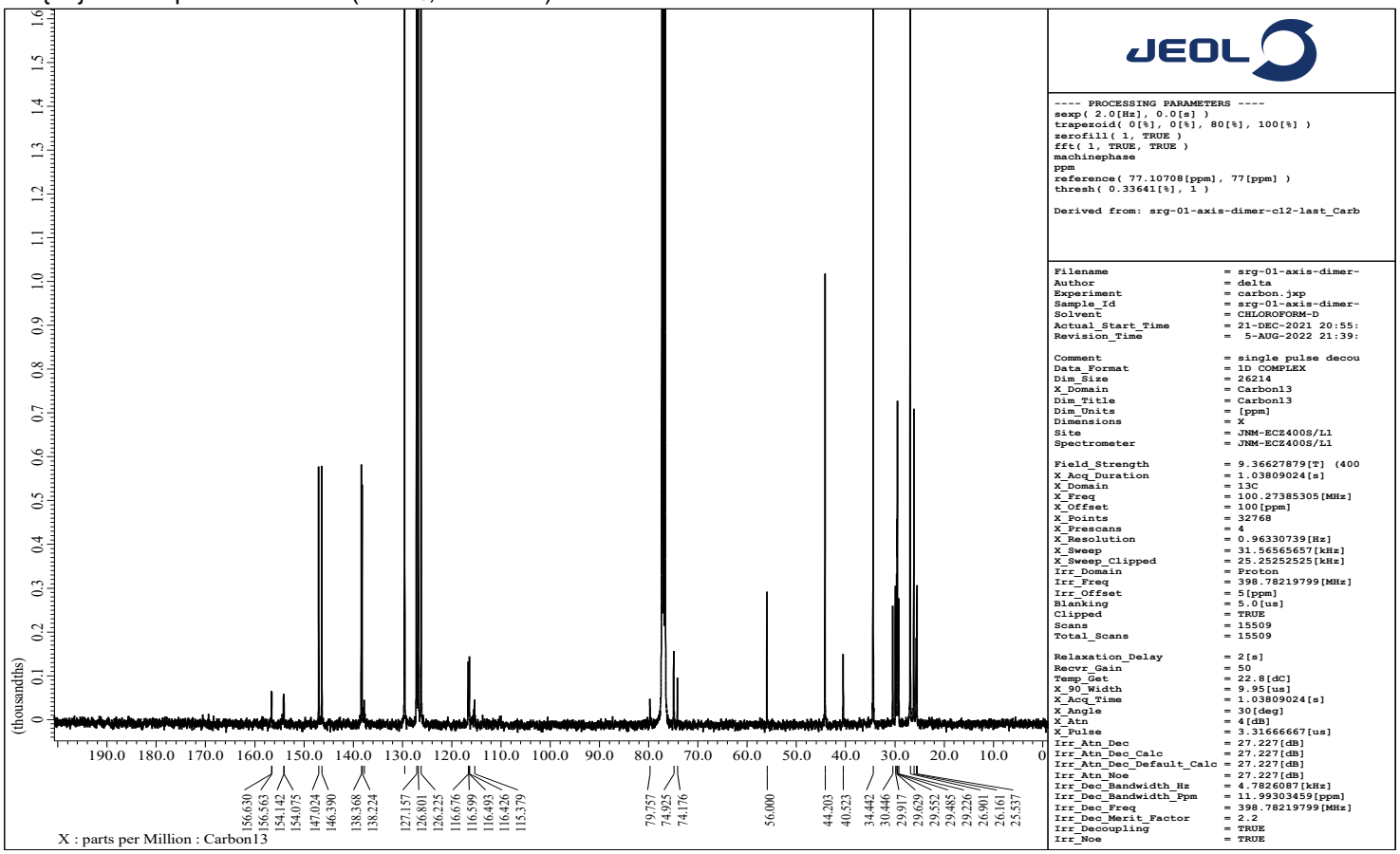

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 43.14362[%], 1 )
phase( 39.06737, 0, 50[%] )

Derived from: srg-01-20-f-last_10-4.jdf
```

Filename	= srg-01-20-f-last_10-5.jdf
Author	= nmrsu
Experiment	= zgig
Sample_Id	= Parameter file, TopSpin 4.1.
Solvent	= CHLOROFORM-D
Revision_Time	= 16-MAR-2022 10:53:56
Comment	= Parameter file, TopSpin 4.1.
Data_Format	= 1D COMPLEX
Dim_Size	= 65536
X_Domain	= 19F
Dim_Title	= 19F
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= BRUKER_DMX_NMR
Field_Strength	= 9.40145797[T] (400[MHz])
X_Domain	= 19F
X_Freq	= 376.63950723[MHz]
X_Freq_Flip	= TRUE
X_Offset	= -37.663951[kHz]
X_Points	= 65536
X_Prescans	= 4
X_Sweep	= 90.91818273[kHz]
Scans	= 32
Temp_Get	= 298.0265[K]
Filter_Factor	= 220

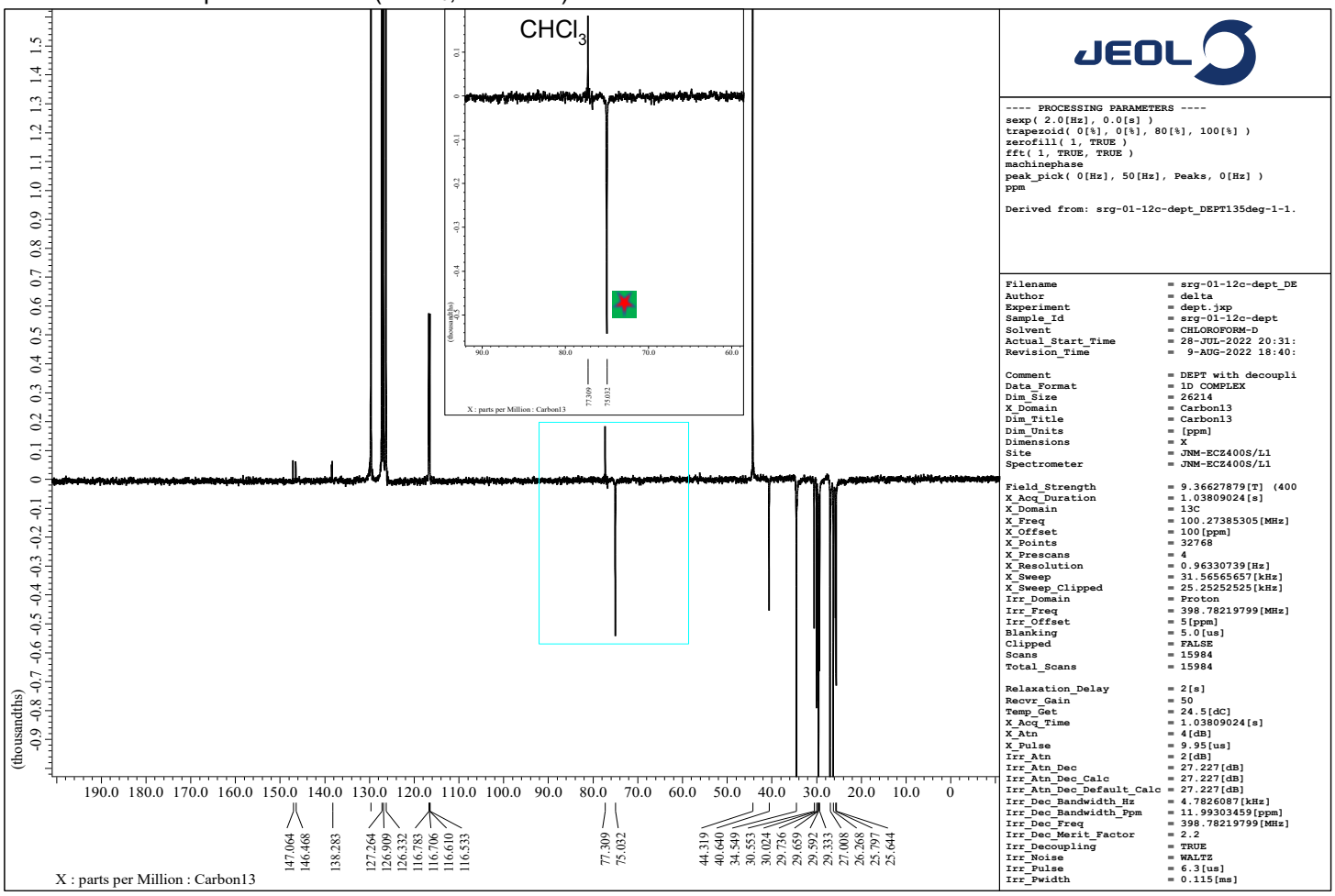
^{1}H NMR Spectrum of **11c** ($CDCl_3$, 500 MHz).



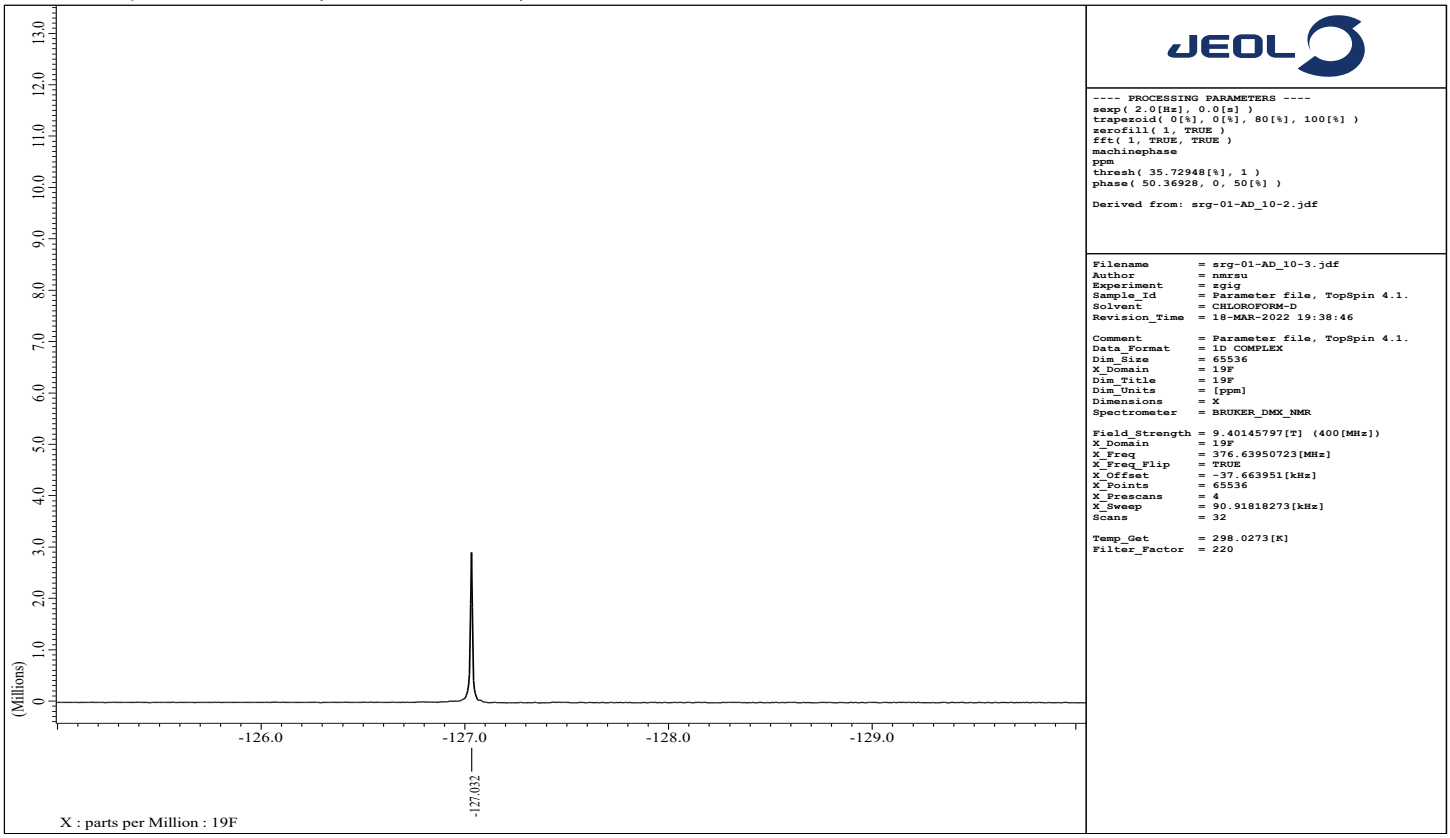
^{13}C {^{1}H} NMR Spectrum of **11c** ($CDCl_3$, 100 MHz).



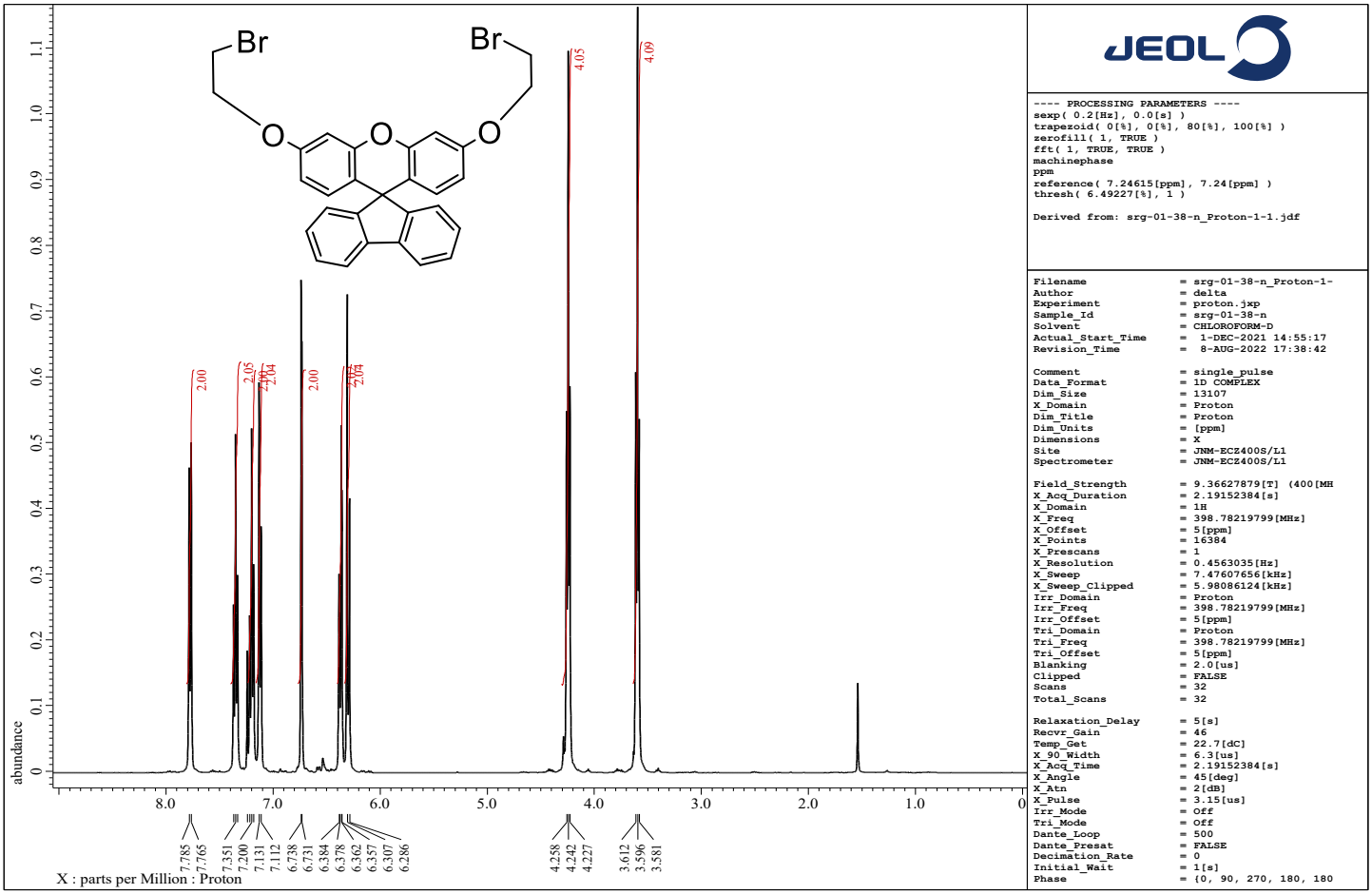
DEPT-135 NMR Spectrum of **11c** ($CDCl_3$, 100 MHz).



^{19}F NMR Spectrum of **11c** ($CDCl_3$, 377 MHz).



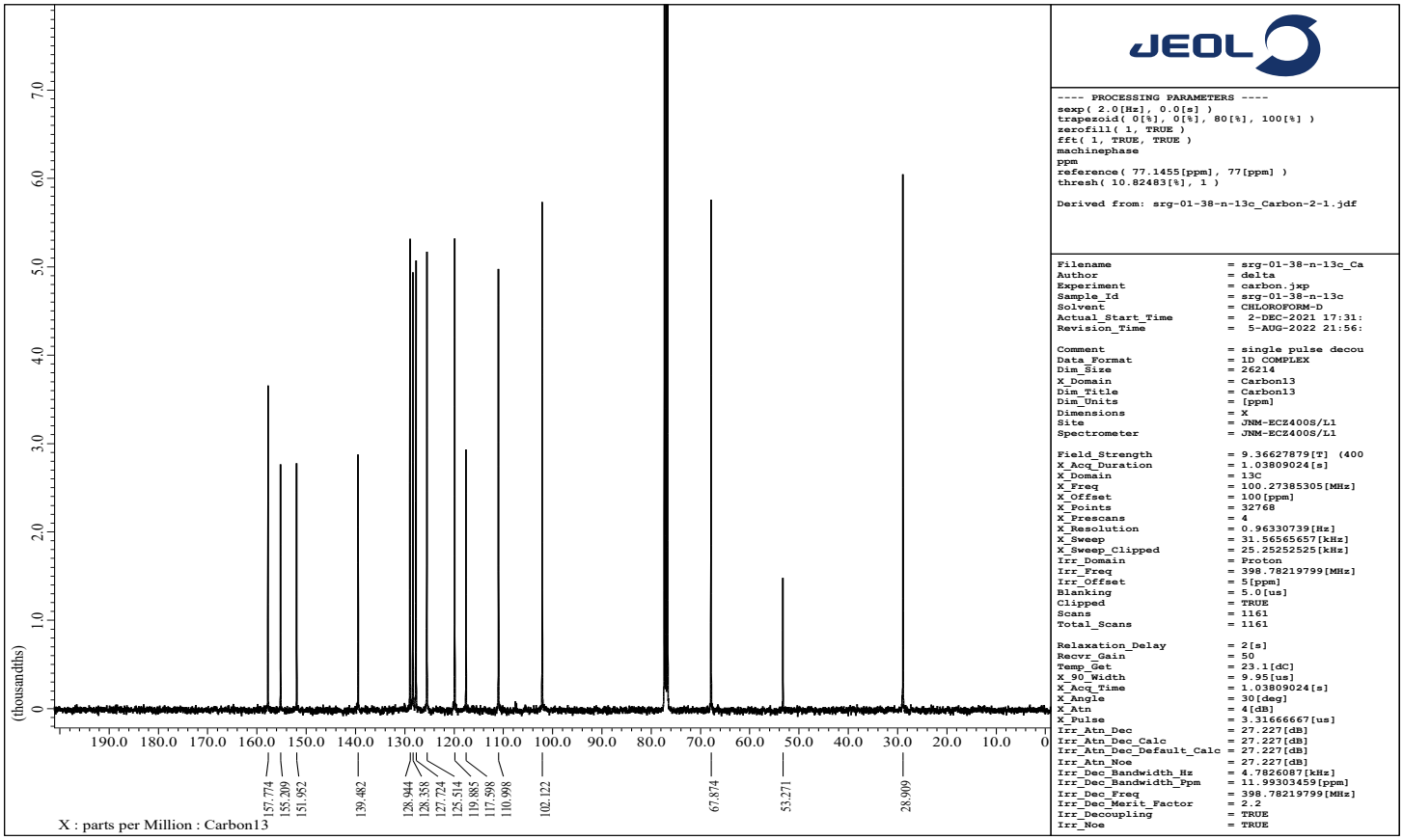
^{1}H NMR Spectrum of **12** ($CDCl_3$, 400 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24615[ppm], 7.24[ppm] )
thresh( 6.49227[%], 1 )

Derived from: srg-01-38-n_Proton-1-1.jdf
```

Filename	= srg-01-38-n_Proton-1-
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-38-n
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-DEC-2021 14:55:17
Revision_Time	= 8-AUG-2022 17:38:42
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 22.7[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

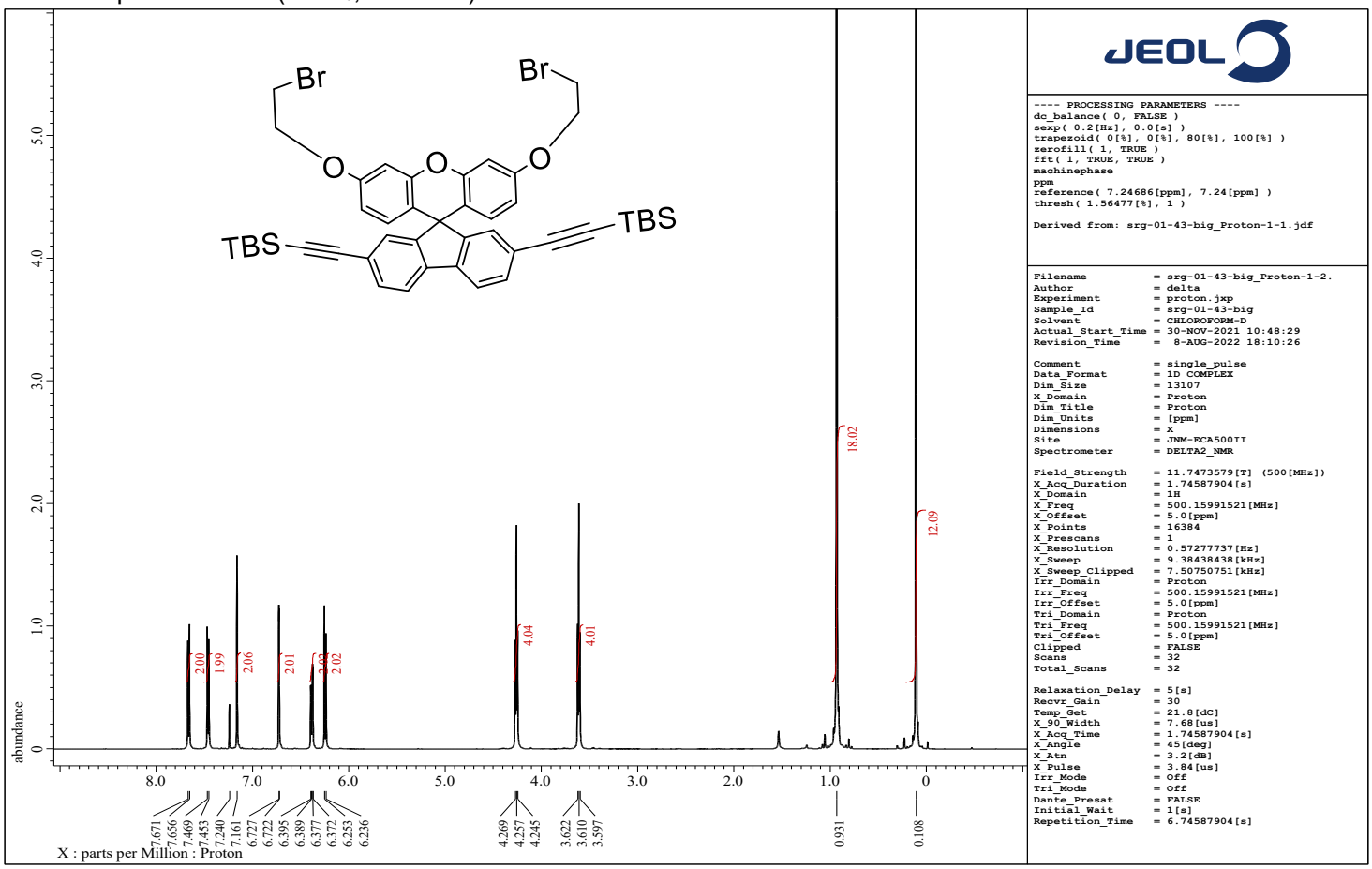
^{13}C {^{1}H} NMR Spectrum of **12** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.1455[ppm], 77[ppm] )
thresh( 10.82483[%], 1 )

Derived from: srg-01-38-n-13c_Carbon-2-1.jdf
```

Filename	= srg-01-38-n-13c_Ca
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-38-n-13c
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-DEC-2021 17:31:
Revision_Time	= 5-AUG-2022 21:56:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 1161
Total_Scans	= 1161
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.1[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

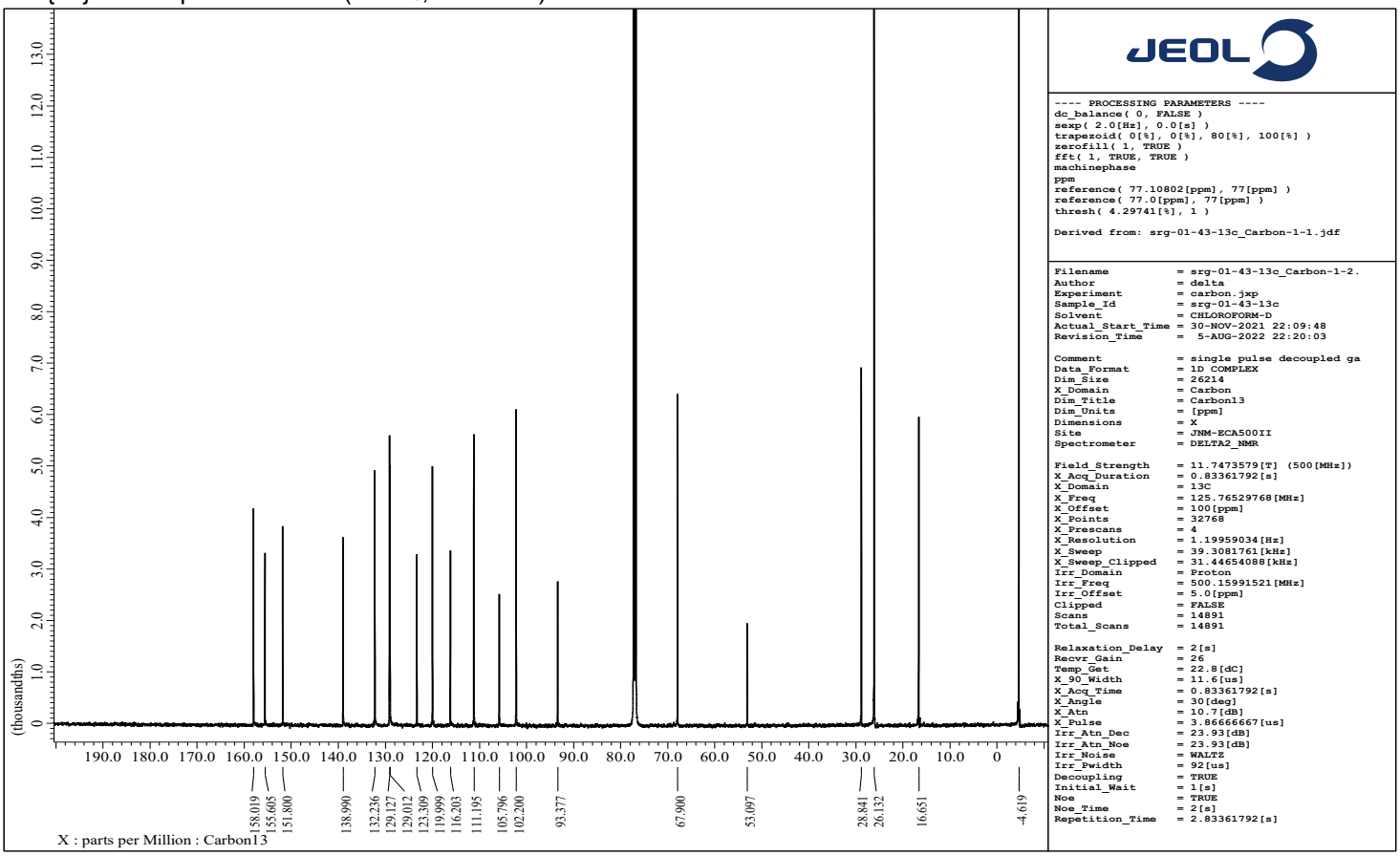
^{1}H NMR Spectrum of **13** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24686[ppm], 7.24[ppm] )
thresh( 1.56477[%], 1 )

Derived from: srg-01-43-big_Proton-1-1.jdf
```

```
Filename            = srg-01-43-big_Proton-1-2.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = srg-01-43-big
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-NOV-2021 10:48:29
Revision_Time       =  8-AUG-2022 18:10:26

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 32
Total_Scans         = 32

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 21.8[dC]
X_90_Width          = 7.68[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.2[dB]
X_Pulse             = 3.84[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

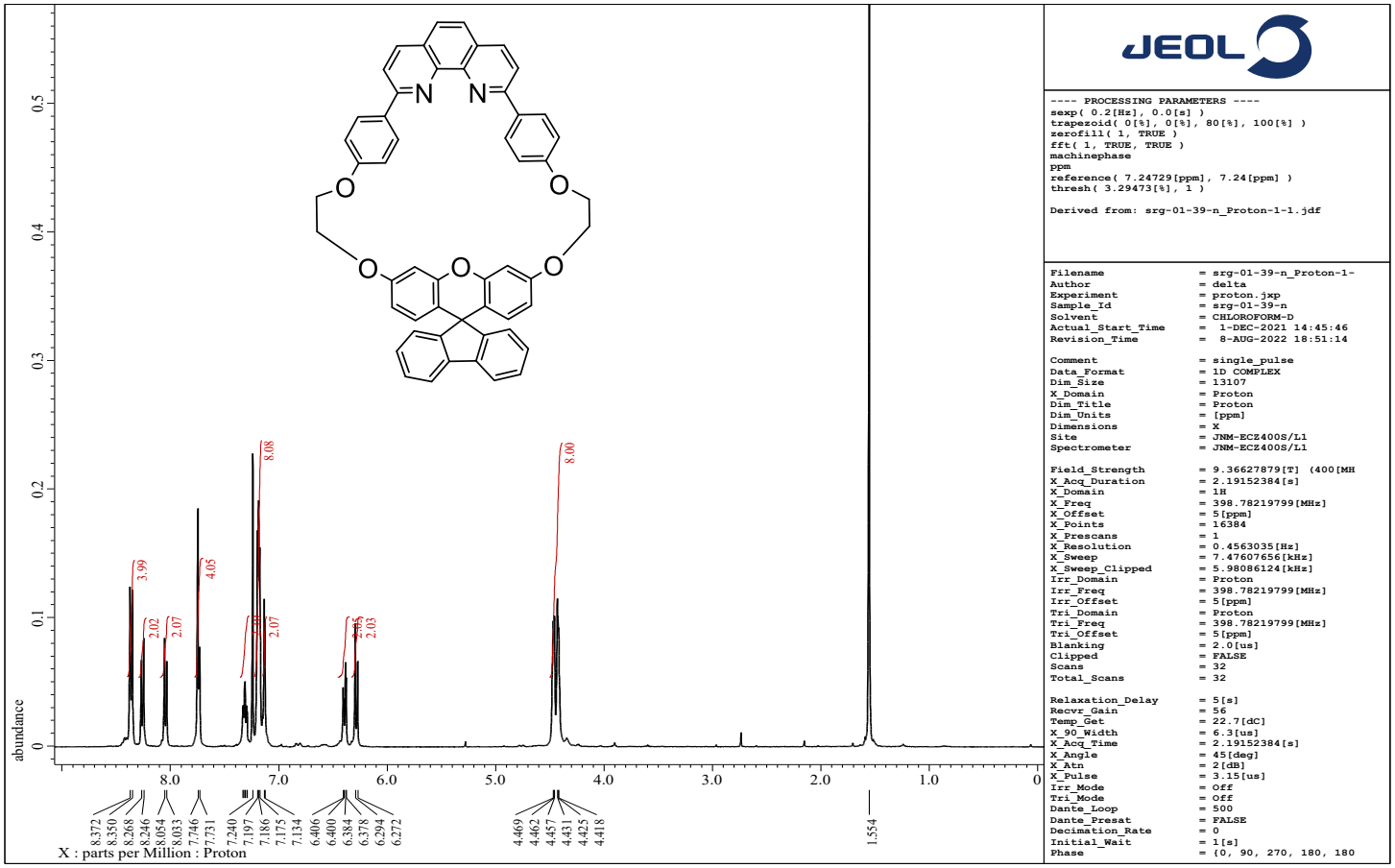
^{13}C {^{1}H} NMR Spectrum of **13** ($CDCl_3$, 125 MHz).



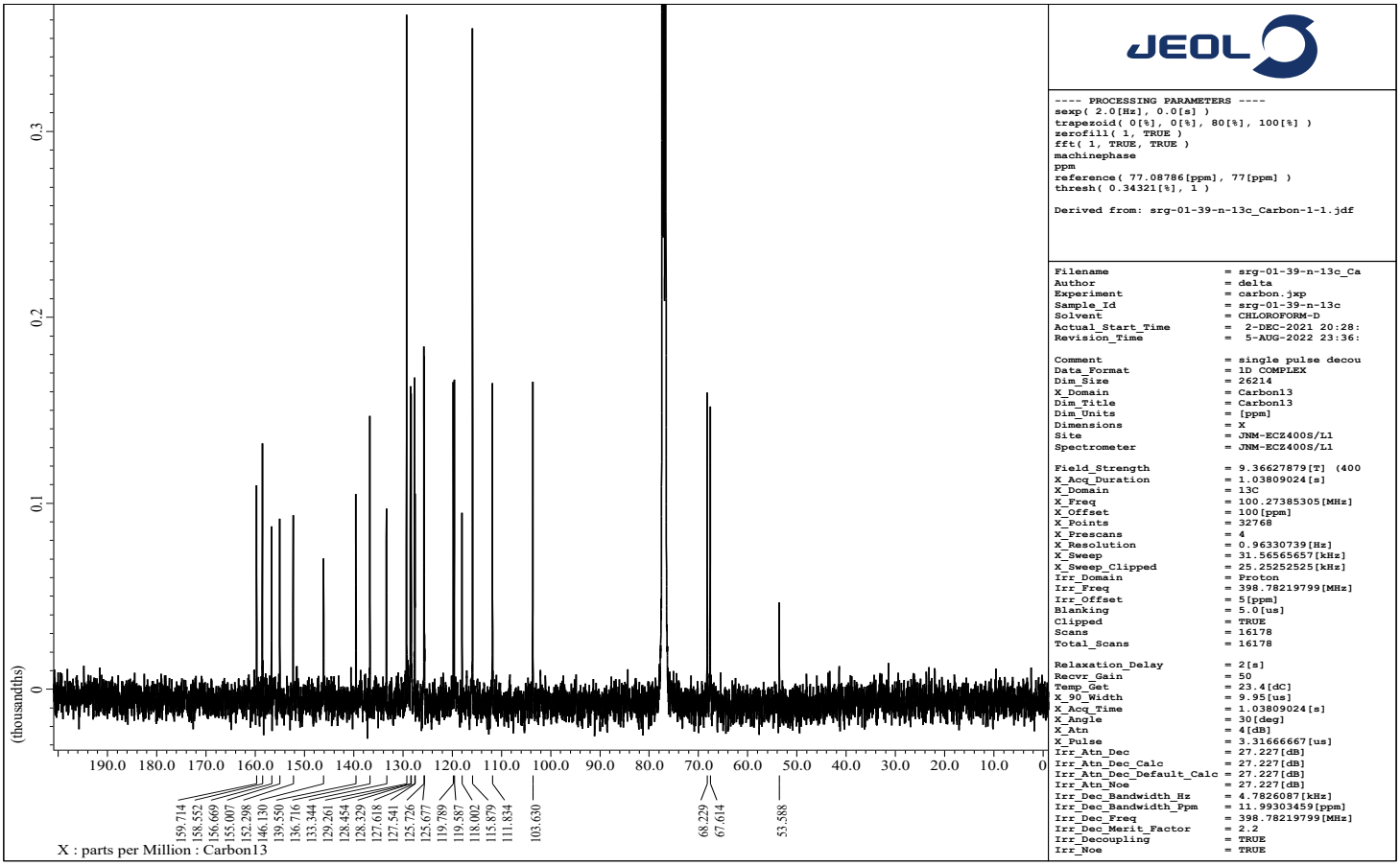

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.10802[ppm], 77[ppm] )
reference( 77.0[ppm], 77[ppm] )
thresh( 4.29741[%], 1 )

Derived from: srg-01-43-13c_Carbon-1-1.jdf
```

```
Filename            = srg-01-43-13c_Carbon-1-2.
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = srg-01-43-13c
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-NOV-2021 22:09:48
Revision_Time       =  5-AUG-2022 22:20:03

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 14891
Total_Scans         = 14891

Relaxation_Delay    = 2[s]
Recvr_Gain          = 26
Temp_Get            = 22.8[dC]
X_90_Width          = 11.6[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.86666667[us]
Irr_Atn_Dec         = 23.93[dB]
Irr_Atn_Noe         = 23.93[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

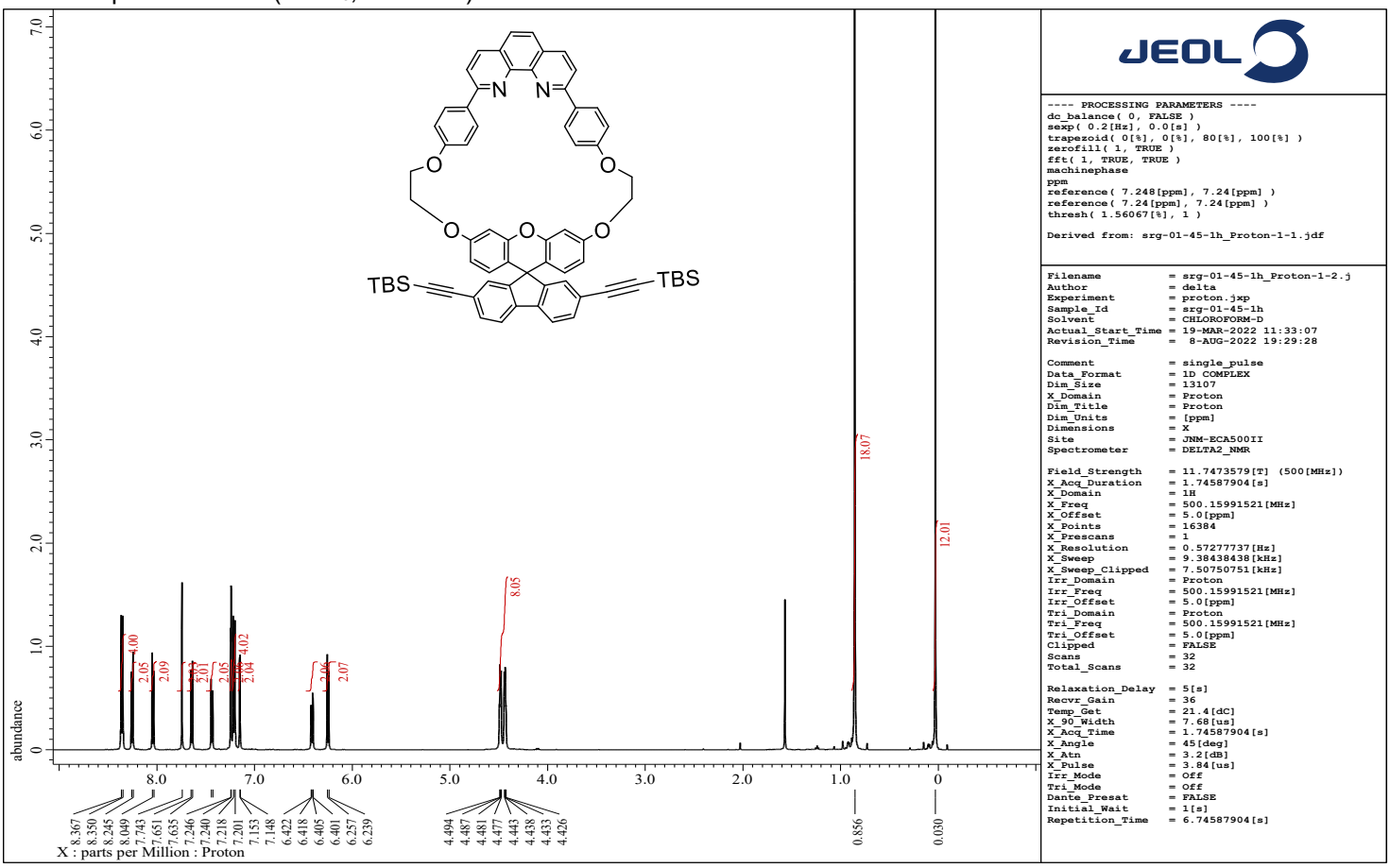
^{1}H NMR Spectrum of **14** ($CDCl_3$, 400 MHz).



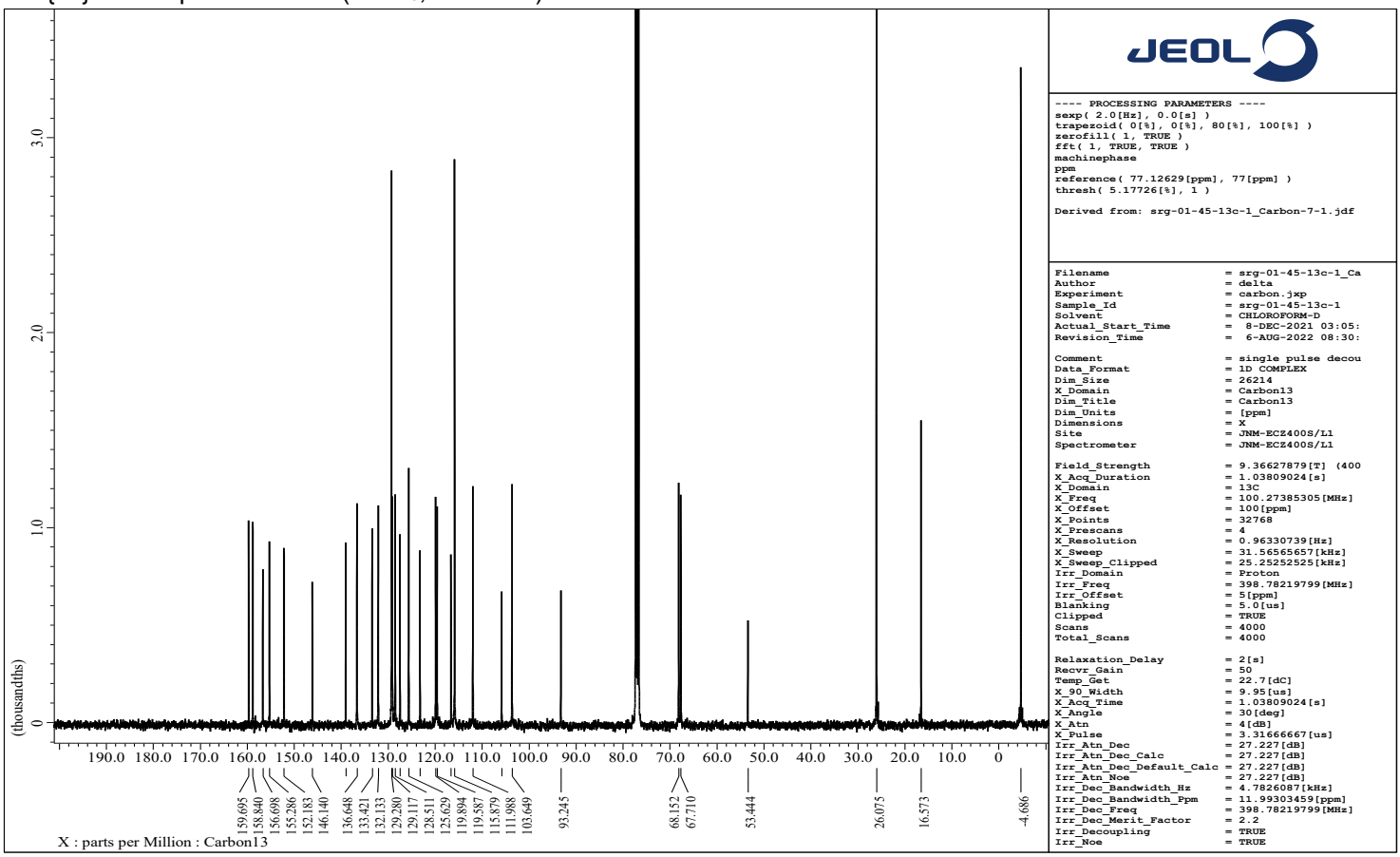
^{13}C {^{1}H} NMR Spectrum of **14** ($CDCl_3$, 100 MHz).



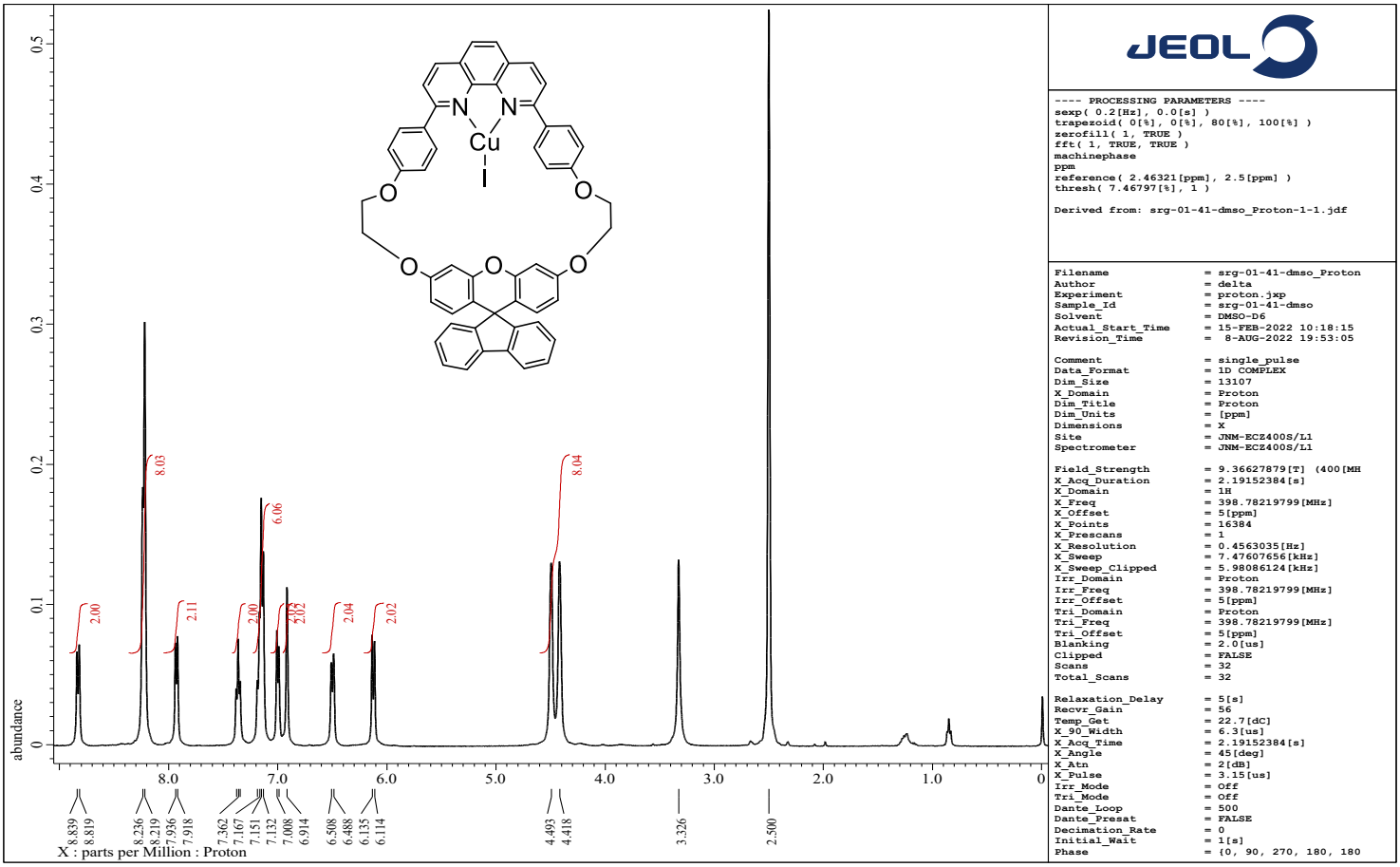
^{1}H NMR Spectrum of **15** ($CDCl_3$, 500 MHz).



^{13}C {^{1}H} NMR Spectrum of **15** ($CDCl_3$, 100 MHz).



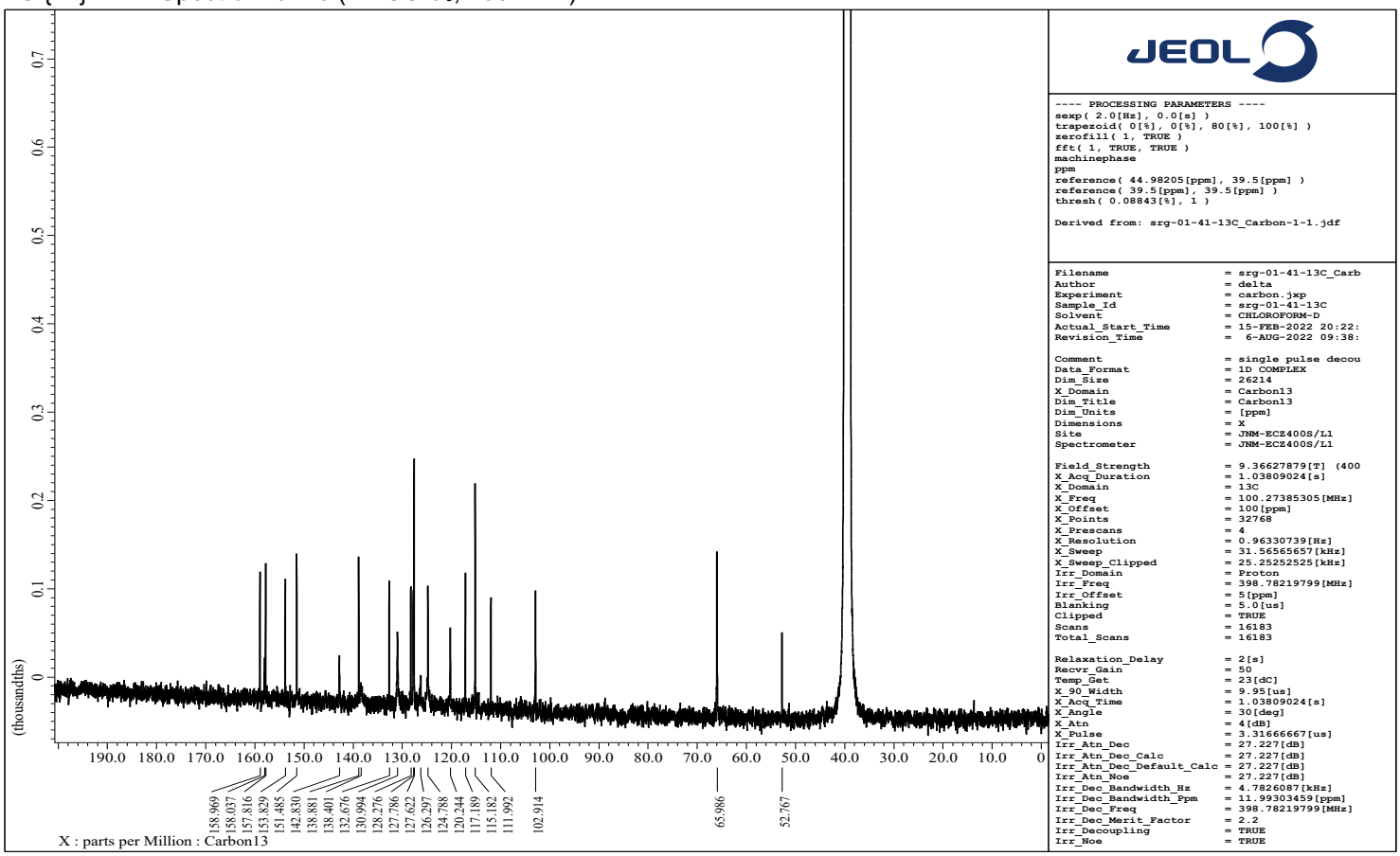
^{1}H NMR Spectrum of **16** (DMSO-d_6, 400 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 2.46321[ppm], 2.5[ppm] )
thresh( 7.46797[%], 1 )

Derived from: srg-01-41-dmso_Proton-1-1.jdf
```

Filename	= srg-01-41-dmso_Proton
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-41-dmso
Solvent	= DMSO-D6
Actual_Start_Time	= 15-FEB-2022 10:18:15
Revision_Time	= 8-AUG-2022 19:53:05
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400[MH
X_Acq_Duration	= 2.19152384[s]
X_Domain	= 1H
X_Freq	= 398.78219799[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.4563035[Hz]
X_Sweep	= 7.47607656[kHz]
X_Sweep_Clipped	= 5.98086124[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 398.78219799[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2.0[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 22.7[dC]
X_90_Width	= 6.3[us]
X_Acq_Time	= 2.19152384[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 3.15[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180

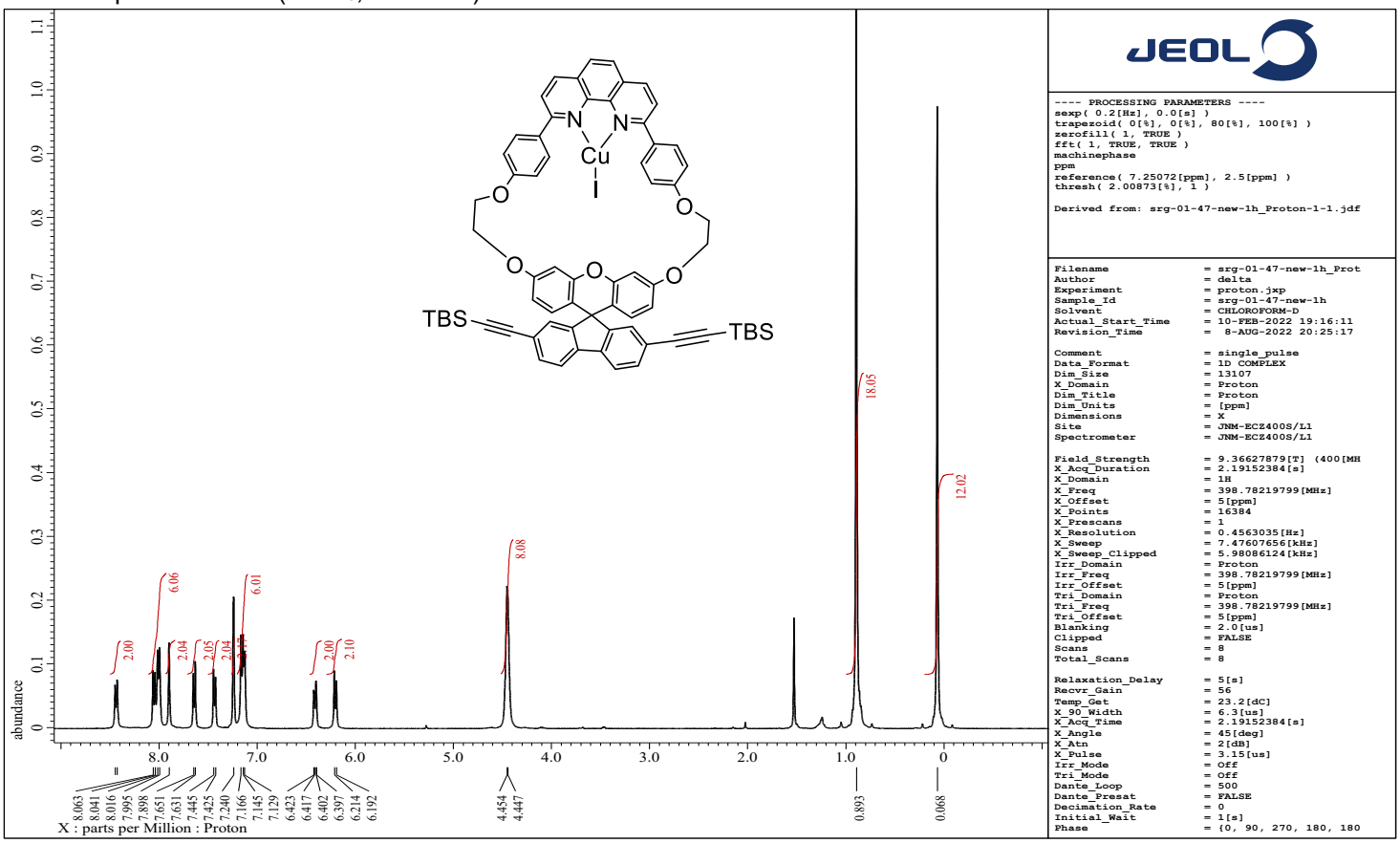
^{13}C {^{1}H} NMR Spectrum of **16** (DMSO-d_6, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 44.98205[ppm], 39.5[ppm] )
reference( 39.5[ppm], 39.5[ppm] )
thresh( 0.08843[%], 1 )

Derived from: srg-01-41-13C_Carbon-1-1.jdf
```

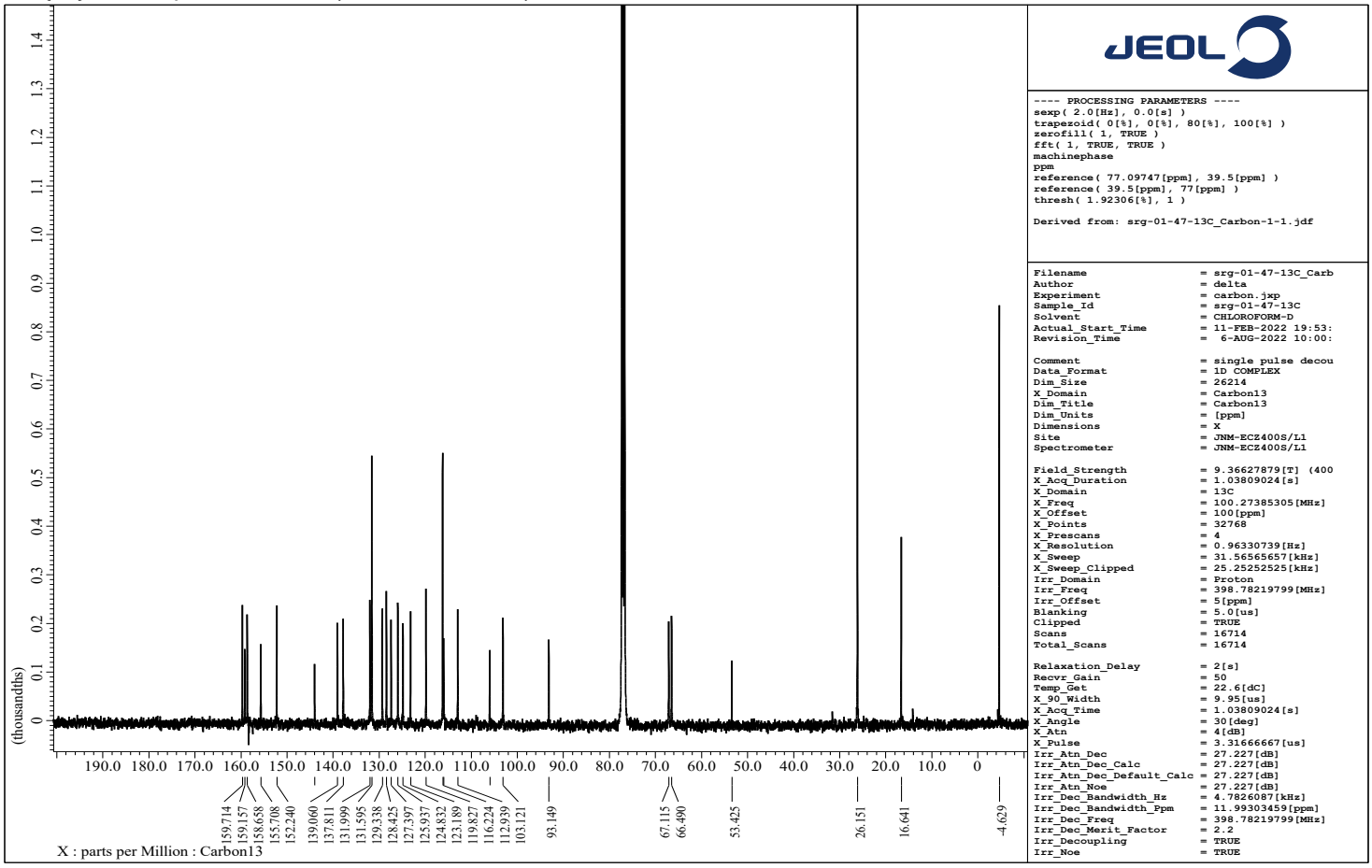
Filename	= srg-01-41-13C_Carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-41-13C
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 15-FEB-2022 20:22:
Revision_Time	= 6-AUG-2022 09:38:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 16183
Total_Scans	= 16183
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

^{1}H NMR Spectrum of **17** ($CDCl_3$, 400 MHz).



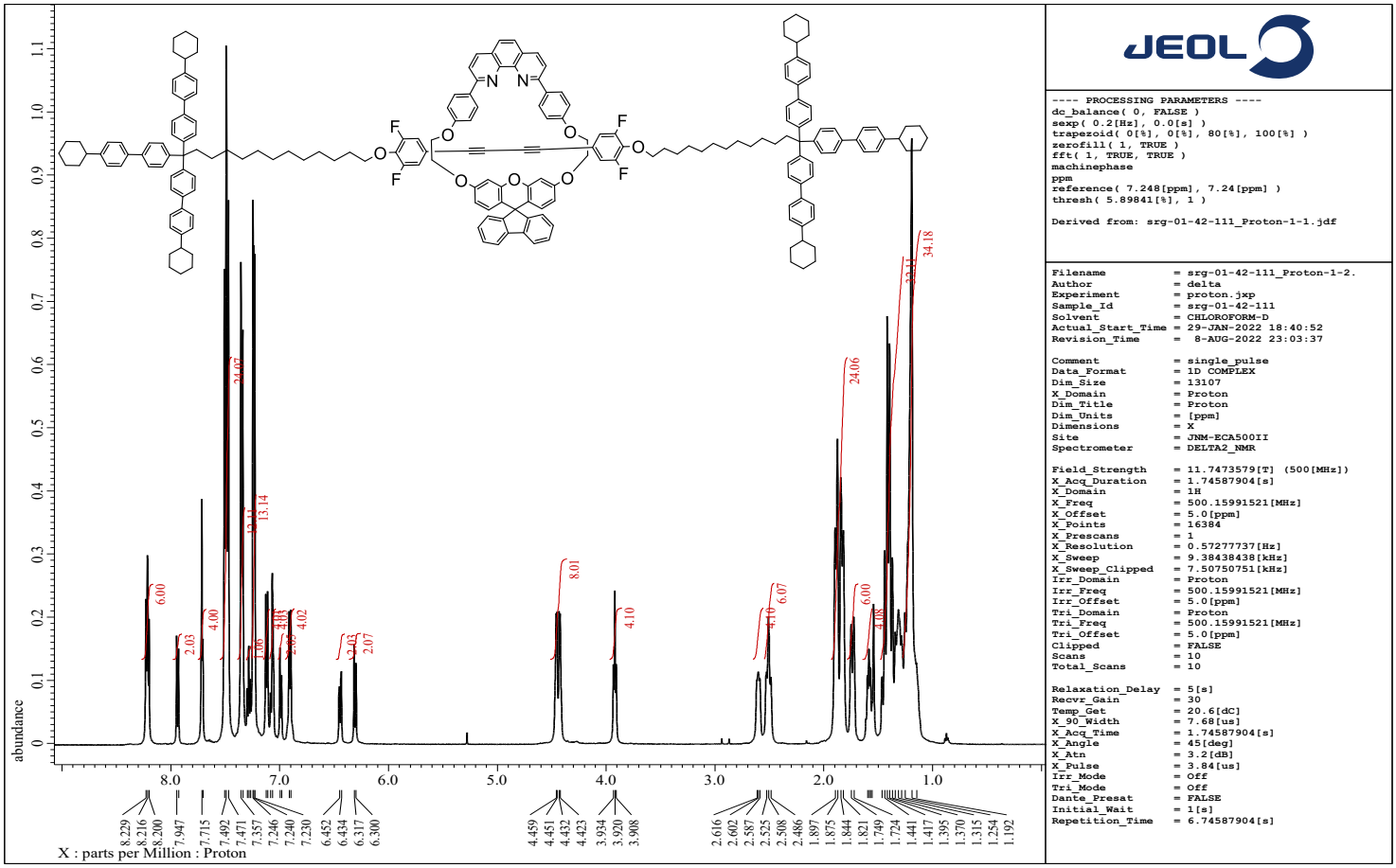

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.25072[ppm], 2.5[ppm] )
thresh( 2.00873[%], 1 )

Derived from: srg-01-47-new-1h_Proton-1-1.jdf

Filename                = srg-01-47-new-1h_Prot
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = srg-01-47-new-1h
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 10-FEB-2022 19:16:11
Revision_Time           =  8-AUG-2022 20:25:17

Comment                 = single_pulse
Data_Format             = 1D COMPLEX
Dim_Size                = 13107
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400[MH
X_Acq_Duration          = 2.19152384[s]
X_Domain                = 1H
X_Freq                  = 398.78219799[MHz]
X_Offset                = 5[ppm]
X_Points                = 16384
X_Prescans              = 1
X_Resolution            = 0.4563035[Hz]
X_Sweep                 = 7.47607656[kHz]
X_Sweep_Clipped         = 5.98086124[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 398.78219799[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2.0[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 5[s]
Recvr_Gain              = 56
Temp_Get                = 23.2[dC]
X_90_Width              = 6.3[us]
X_Acq_Time              = 2.19152384[s]
X_Angle                 = 45[deg]
X_Atn                   = 2[dB]
X_Pulse                 = 3.15[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 500
Dante_Presat            = FALSE
Decimation_Rate         = 0
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
```

^{13}C {^{1}H} NMR Spectrum of **17** ($CDCl_3$, 100 MHz).

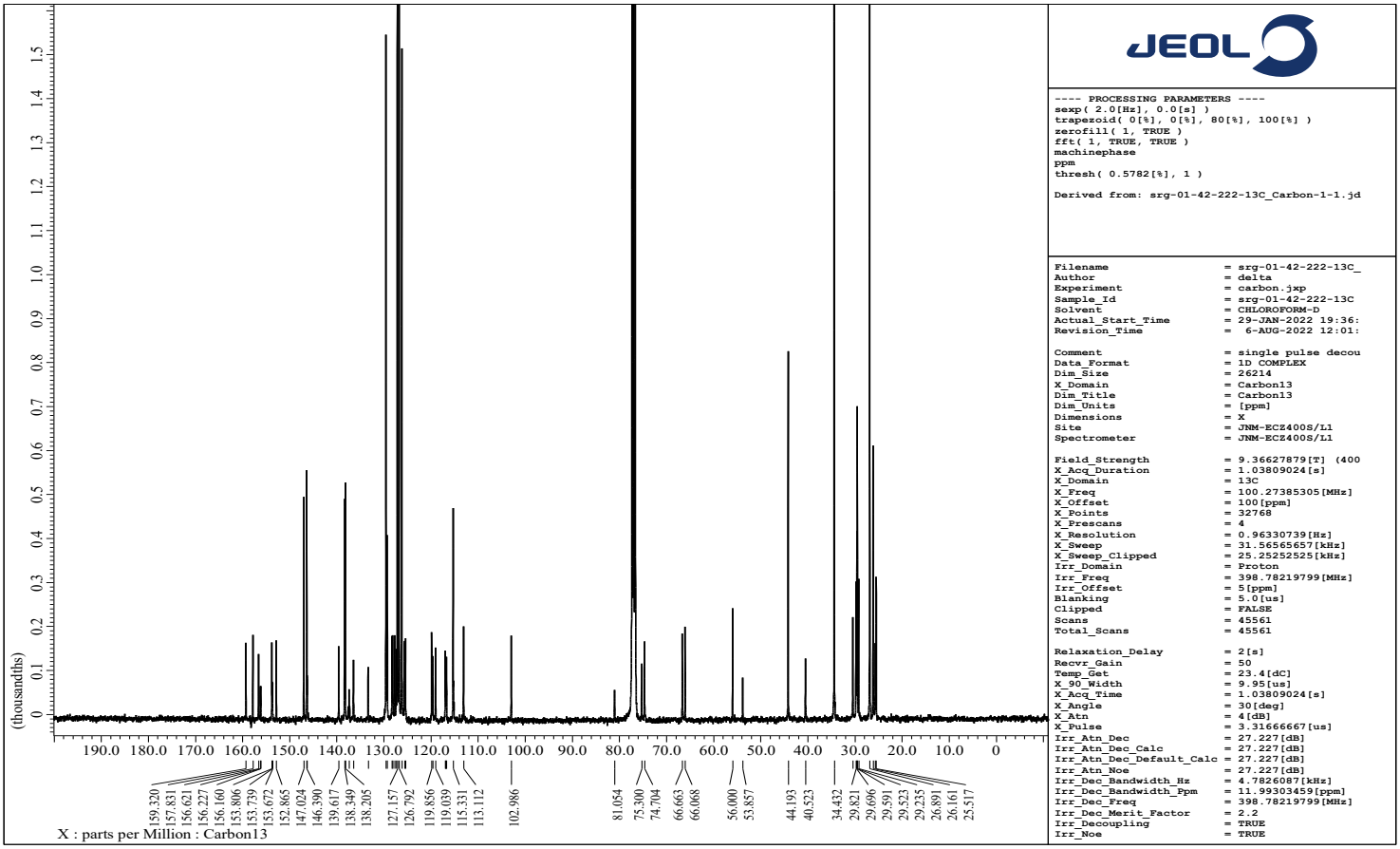



```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.09747[ppm], 39.5[ppm] )
reference( 39.5[ppm], 77[ppm] )
thresh( 1.92306[%], 1 )

Derived from: srg-01-47-13C_Carbon-1-1.jdf

Filename                = srg-01-47-13C_Carb
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = srg-01-47-13C
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2022 19:53:
Revision_Time           =  6-AUG-2022 10:00:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.27385305[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5.0[us]
Clipped                 = TRUE
Scans                   = 16714
Total_Scans             = 16714

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 22.6[dC]
X_90_Width              = 9.95[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 4[dB]
X_Pulse                 = 3.31666667[us]
Irr_Atn_Dec             = 27.227[dB]
Irr_Atn_Dec_Calc        = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe             = 27.227[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.99303459[ppm]
Irr_Dec_Freq            = 398.78219799[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
```

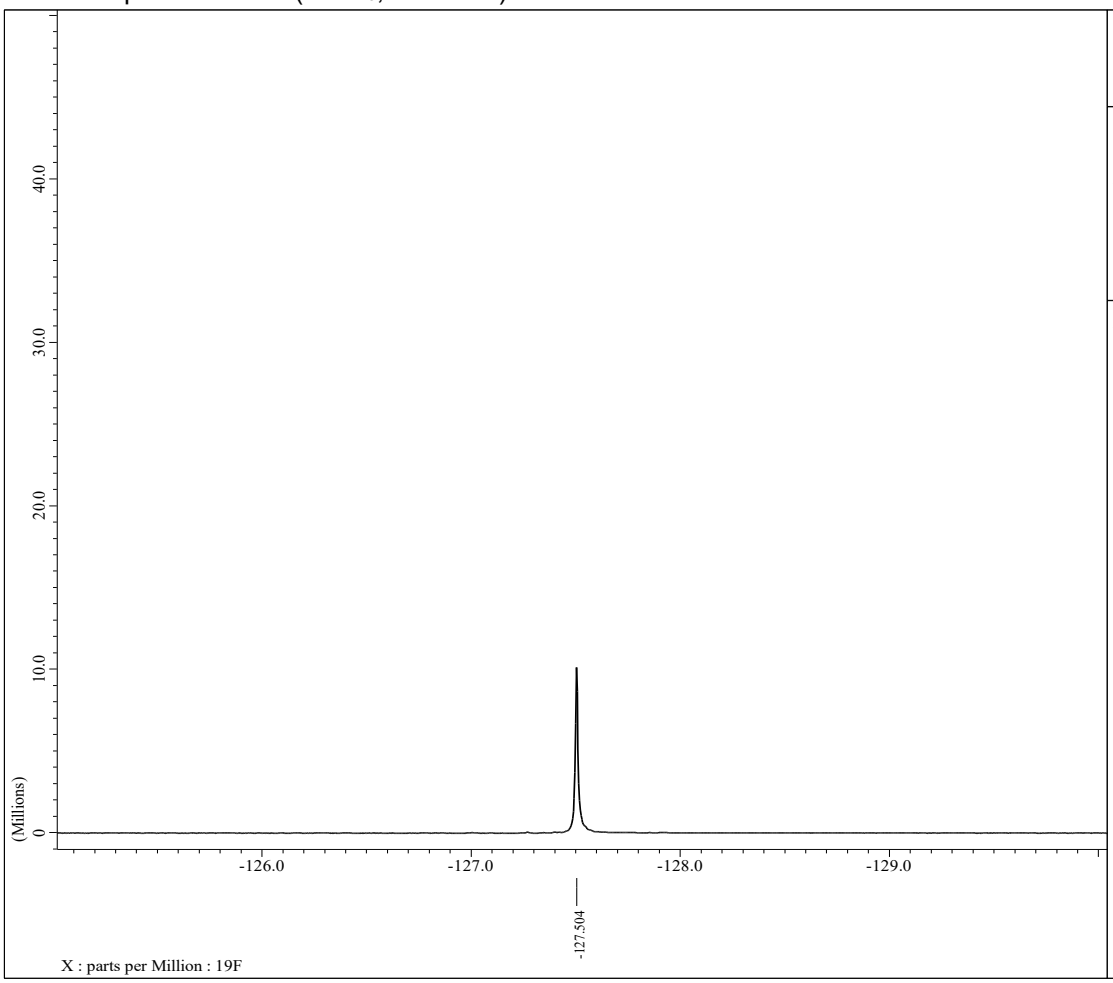
^{1}H NMR Spectrum of **18** ($CDCl_3$, 500 MHz).



^{13}C {^{1}H} NMR Spectrum of **18** ($CDCl_3$, 100 MHz).



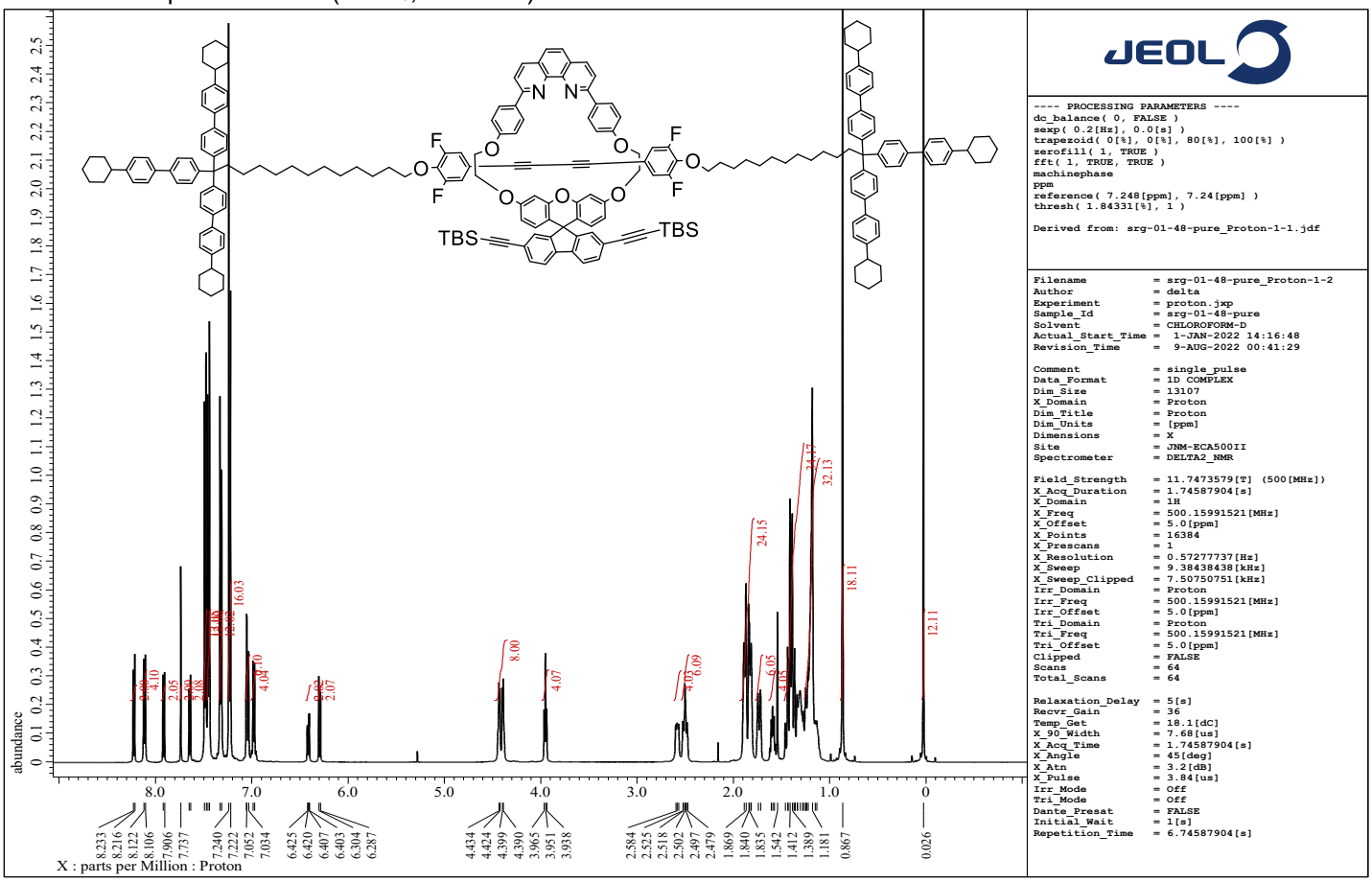
^{19}F NMR Spectrum of **18** ($CDCl_3$, 377 MHz).



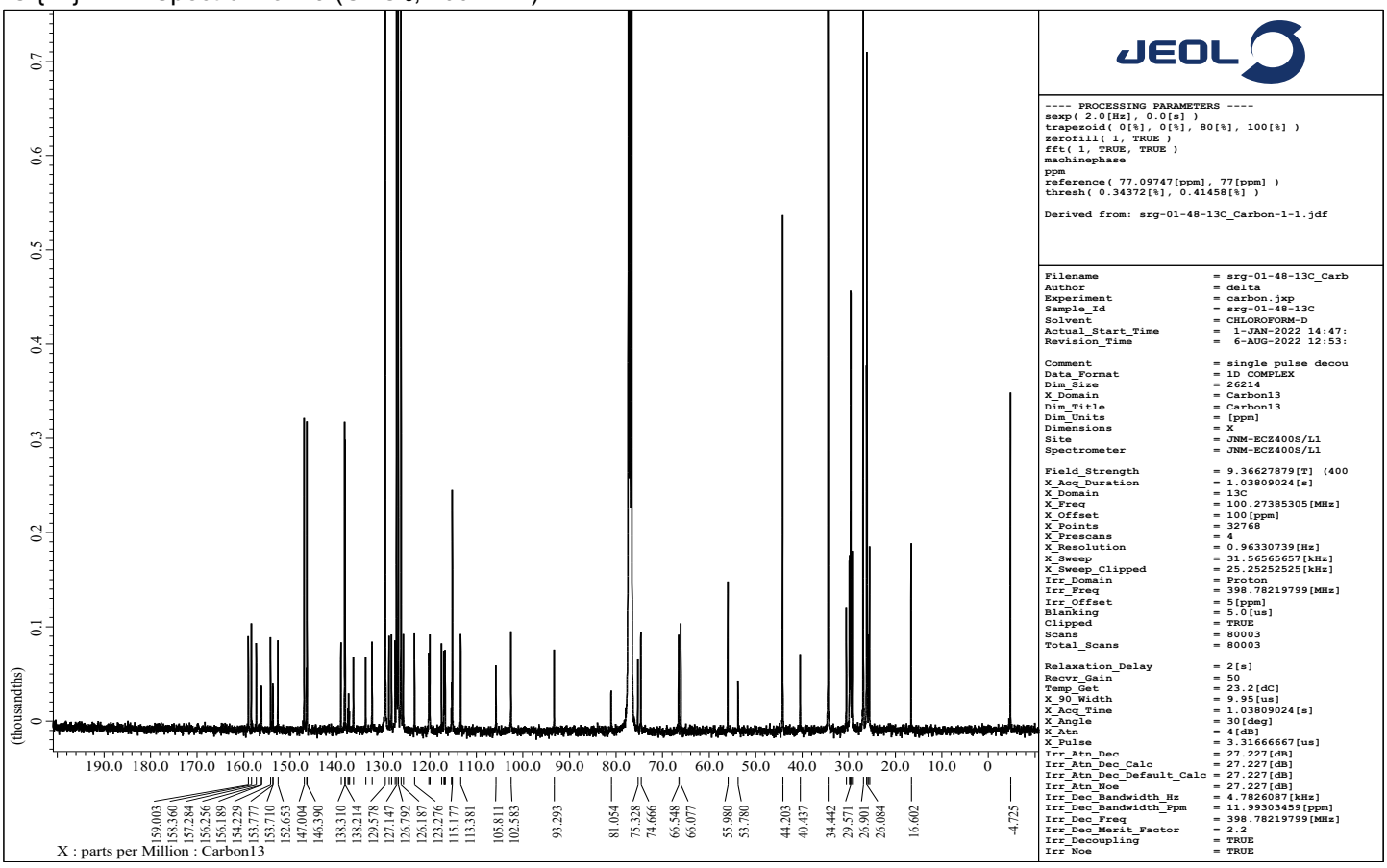

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 56.08808[%], 1 )
phase( 28.75979, 0, 50[%] )

Derived from: srg-01-42_20-1.jdf
```

```
Filename        = srg-01-42_20-2.jdf
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 18-MAR-2022 19:45:40

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 32

Temp_Get        = 298.0309[K]
Filter_Factor   = 220
```

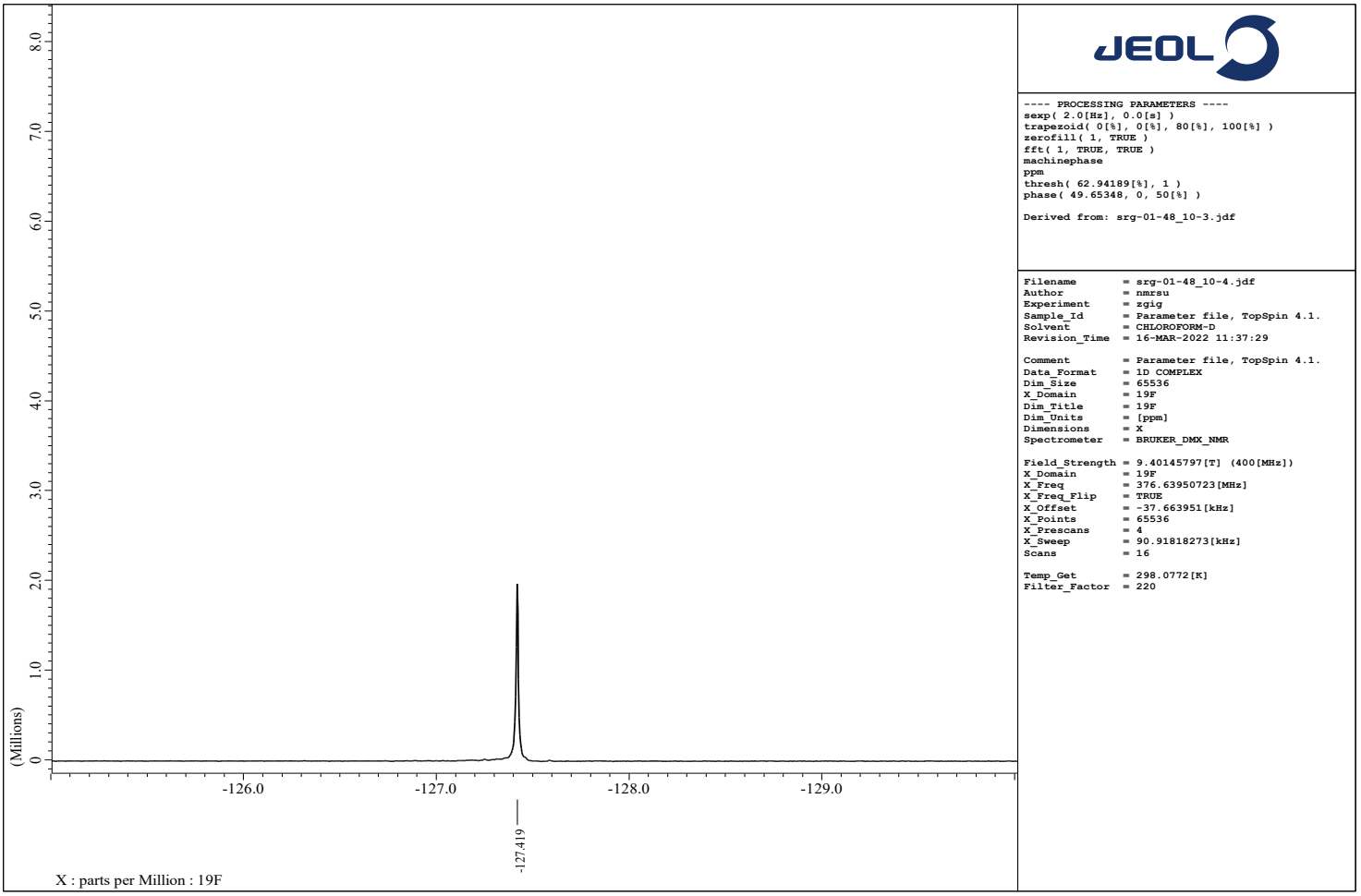
^{1}H NMR Spectrum of **19** ($CDCl_3$, 500 MHz).



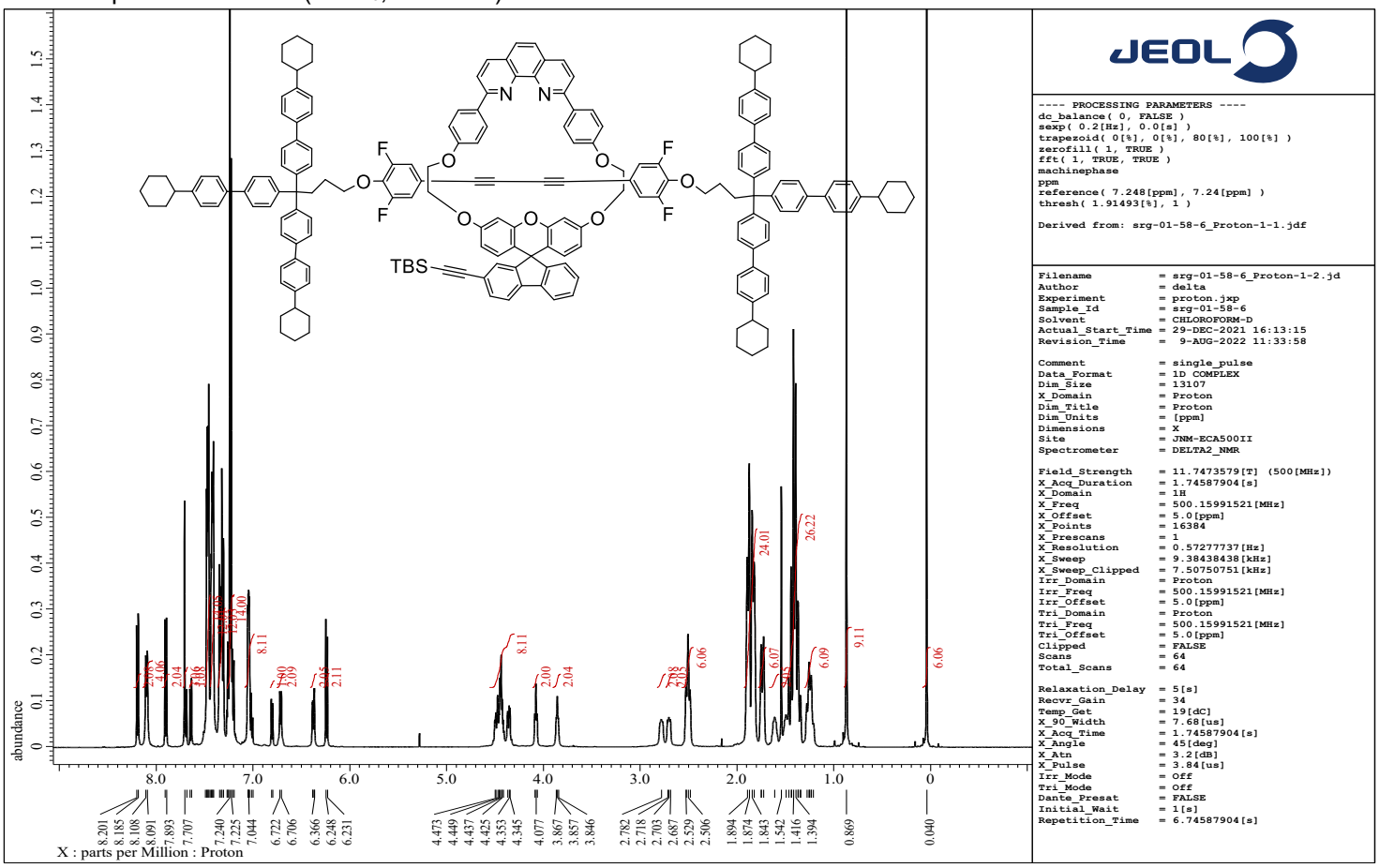
^{13}C {^{1}H} NMR Spectrum of **19** ($CDCl_3$, 100 MHz).



^{19}F NMR Spectrum of **19** ($CDCl_3$, 377 MHz).



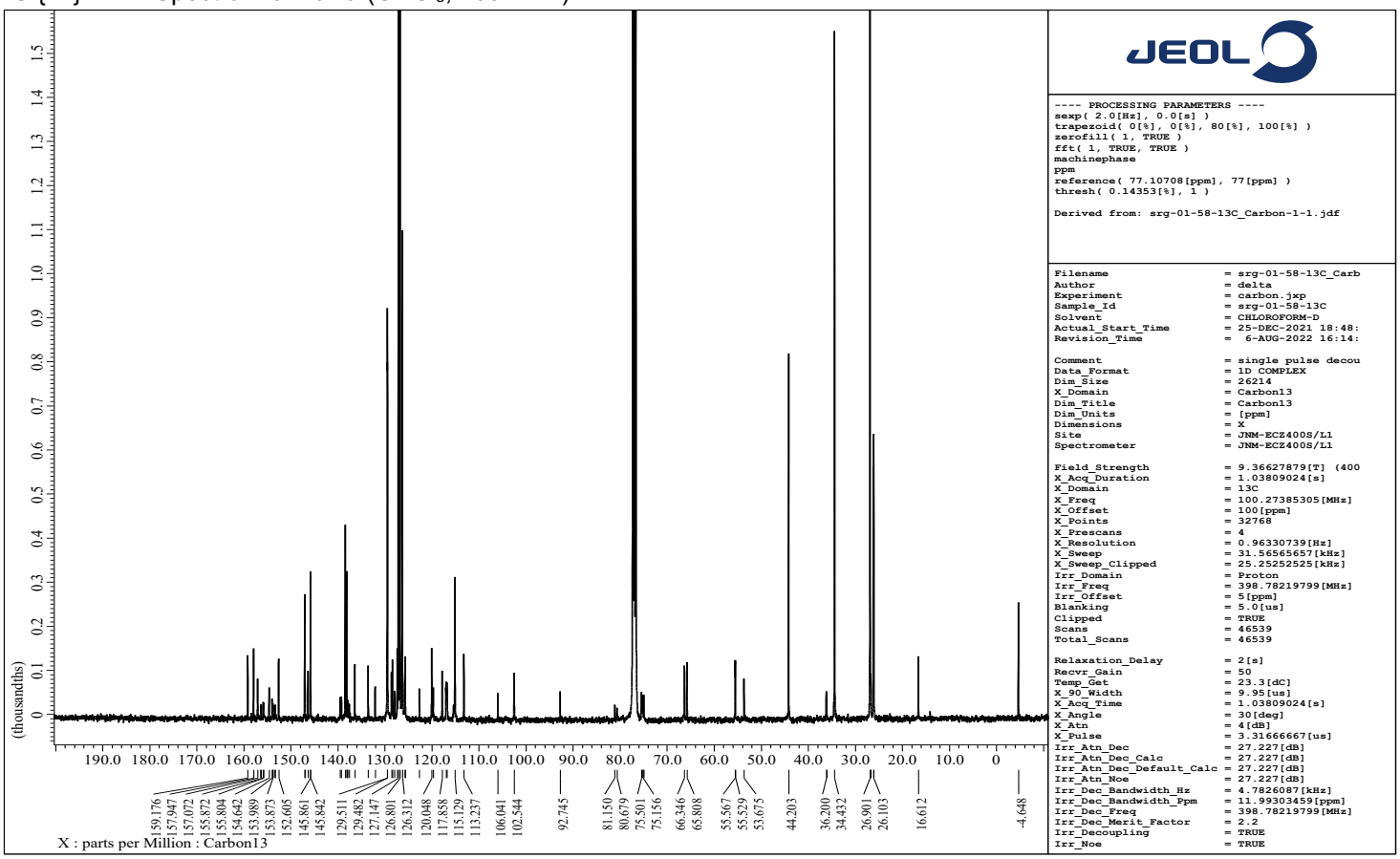
^{1}H NMR Spectrum of **10Aa** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 1.91493[%], 1 )

Derived from: srg-01-58-6_Proton-1-1.jdf
```

Filename	= srg-01-58-6_Proton-1-2.jd
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-58-6
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 29-DEC-2021 16:13:15
Revision_Time	= 9-AUG-2022 11:33:58
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 64
Total_Scans	= 64
Relaxation_Delay	= 5[s]
Recvr_Gain	= 34
Temp_Get	= 19[dC]
X_90_Width	= 7.68[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 3.2[dB]
X_Pulse	= 3.84[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.74587904[s]

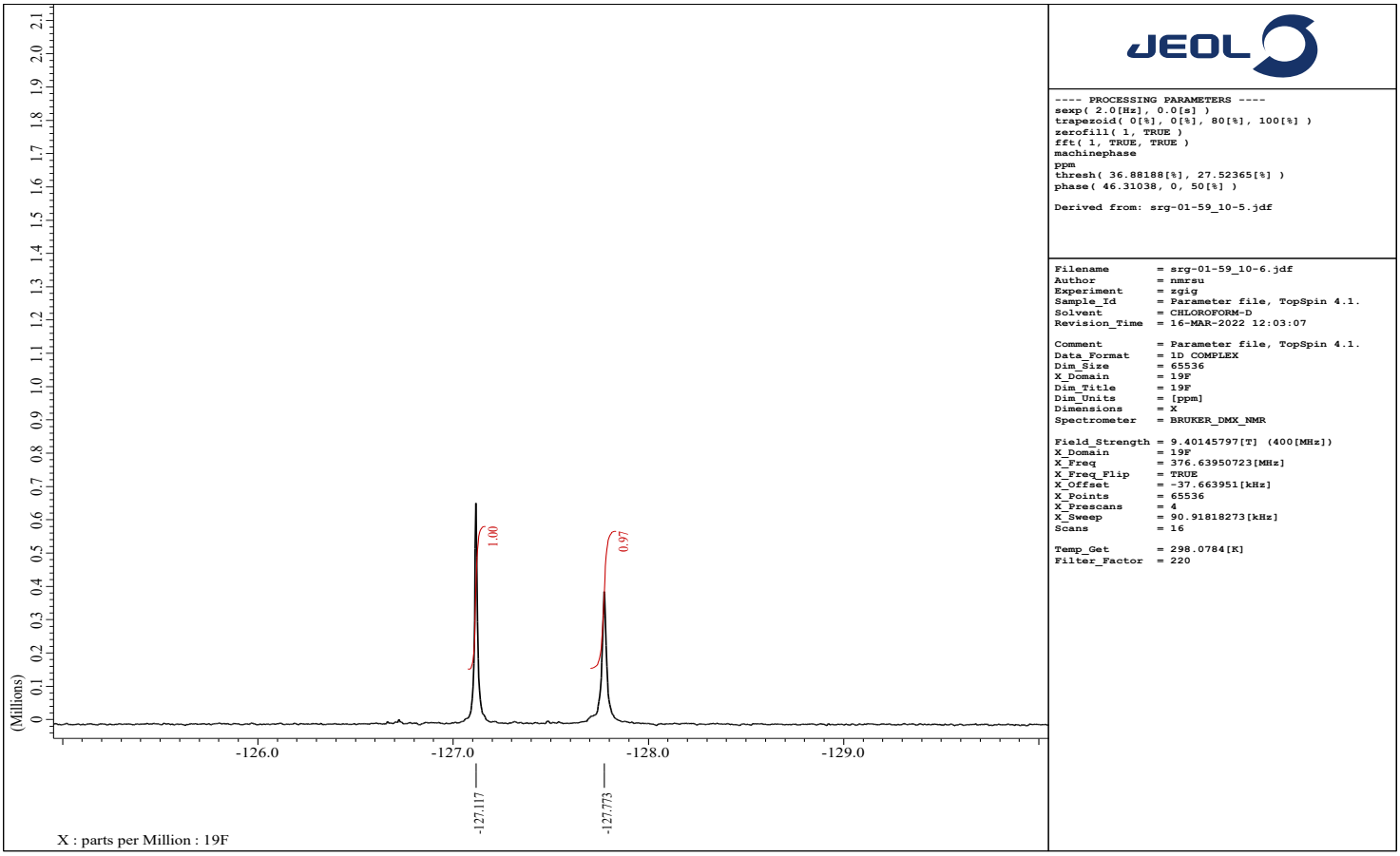
^{13}C {^{1}H} NMR Spectrum of **10Aa** ($CDCl_3$, 100 MHz).



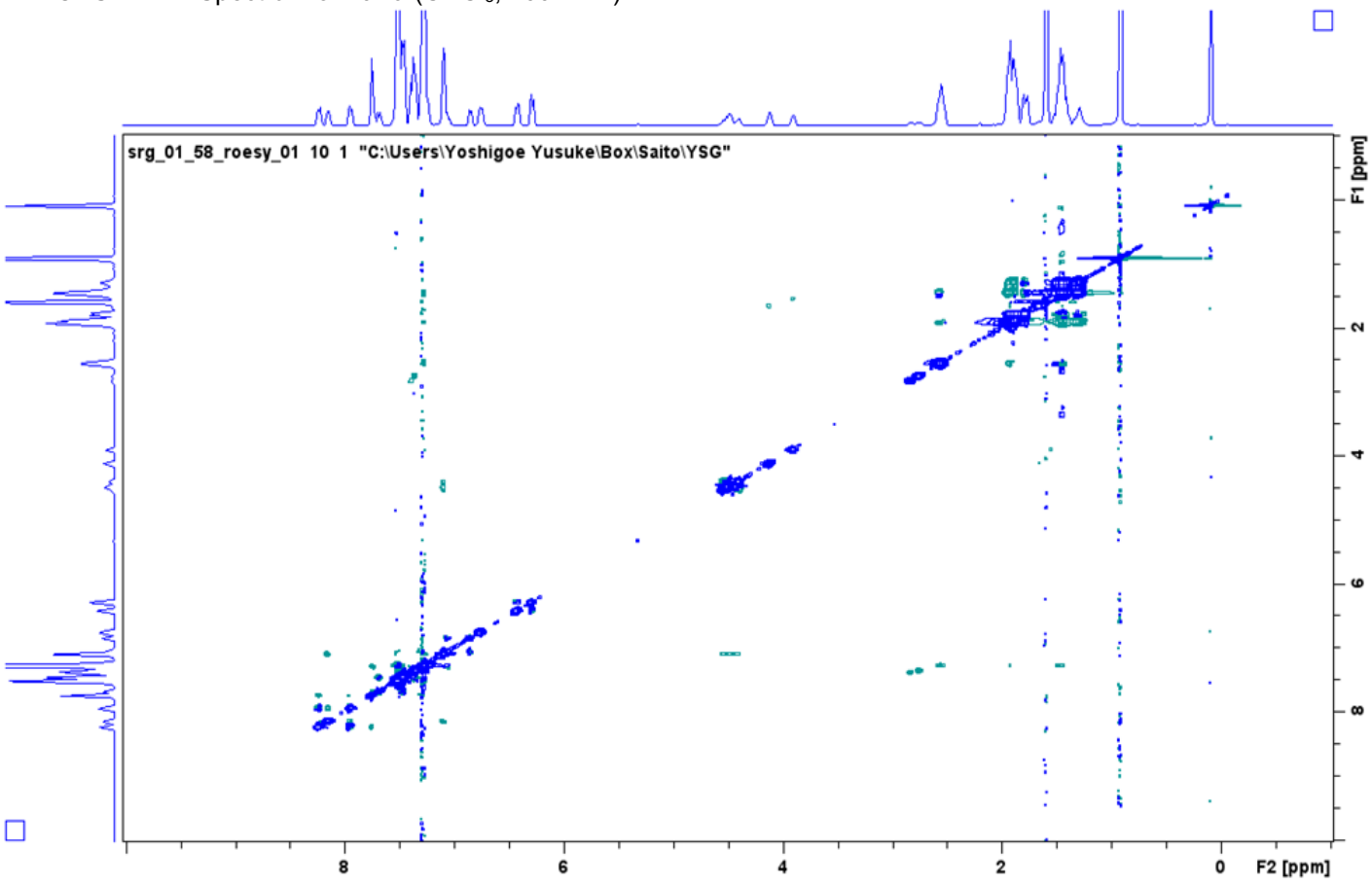

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.10708[ppm], 77[ppm] )
thresh( 0.14353[%], 1 )

Derived from: srg-01-58-13C_Carbon-1-1.jdf
```

Filename	= srg-01-58-13C_Carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-58-13C
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 25-DEC-2021 18:48:
Revision_Time	= 6-AUG-2022 16:14:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 46539
Total_Scans	= 46539
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 23.3[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

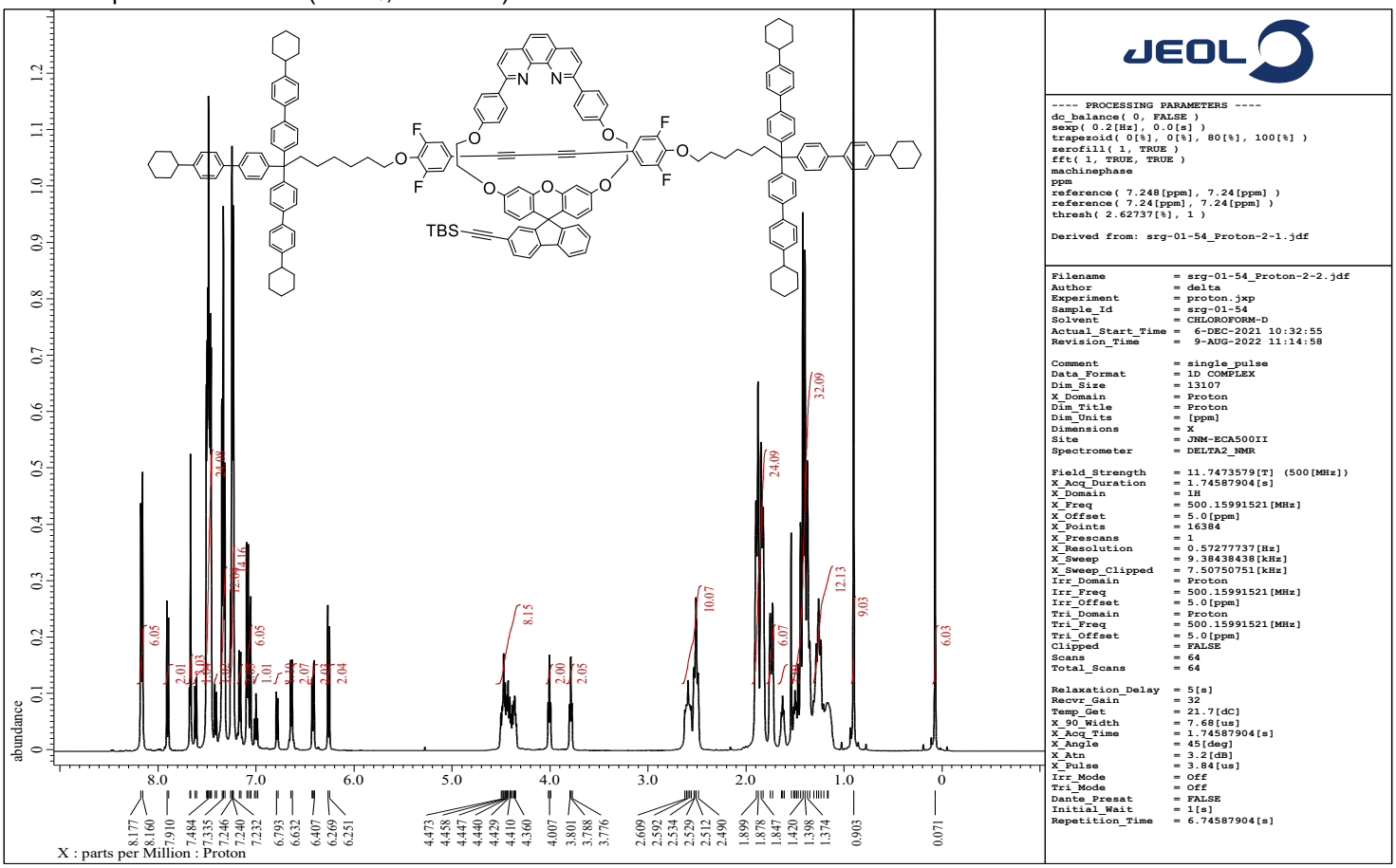
^{19}F NMR Spectrum of **10Aa** ($CDCl_3$, 377 MHz).



^{1}H-^{1}H ROESY NMR Spectrum of **10Aa** ($CDCl_3$, 400 MHz).

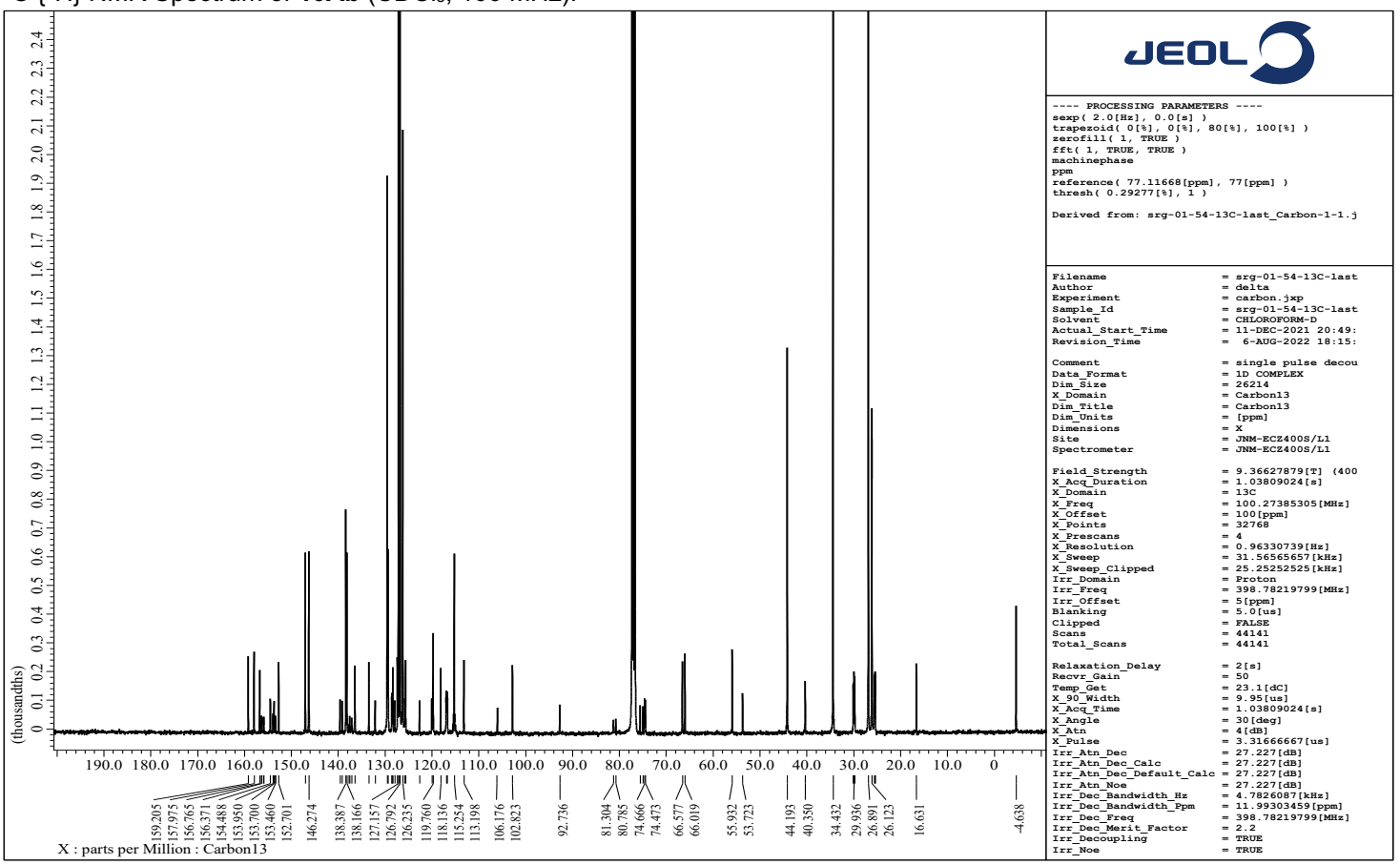


^{1}H NMR Spectrum of **10Ab** ($CDCl_3$, 500 MHz).



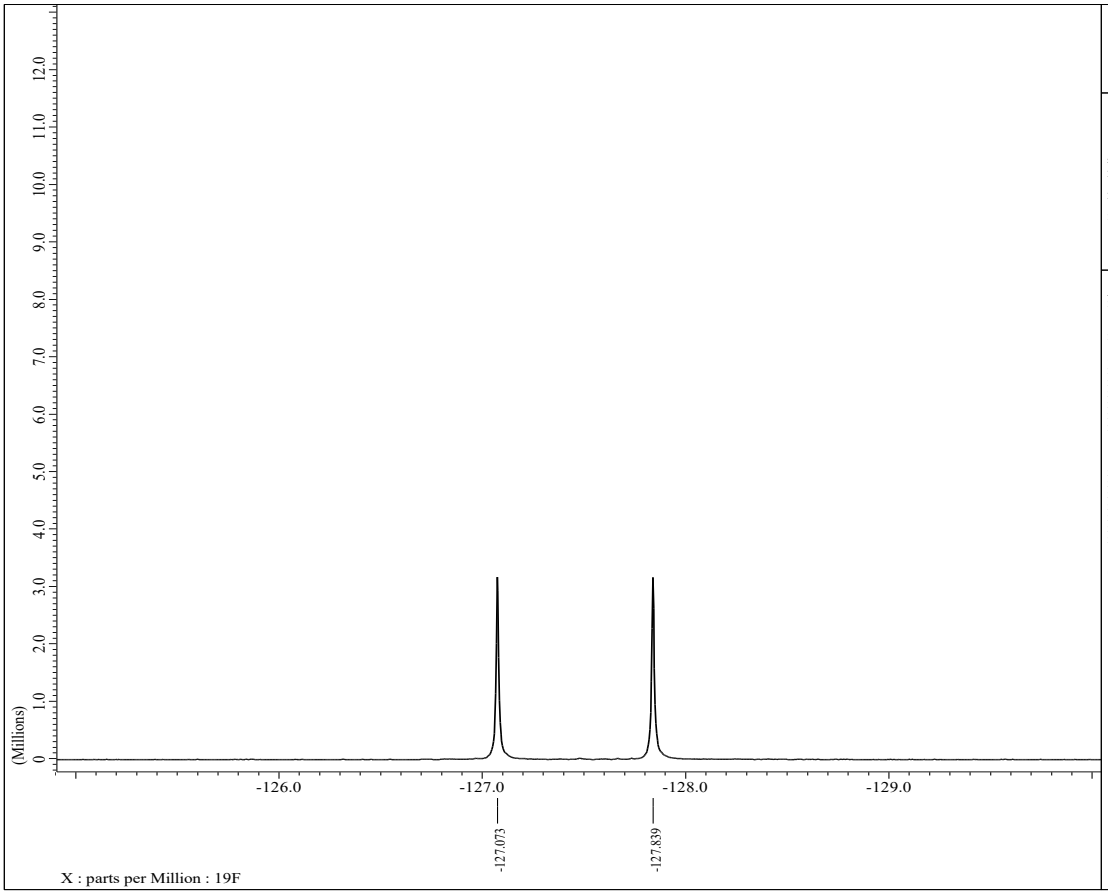

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
reference( 7.24[ppm], 7.24[ppm] )
thresh( 2.62737[%], 1 )

Derived from: srg-01-54_Proton-2-1.jdf

Filename              = srg-01-54_Proton-2-2.jdf
Author                = delta
Experiment            = proton.jxp
Sample_Id             = srg-01-54
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  6-DEC-2021 10:32:55
Revision_Time         =  9-AUG-2022 11:14:58

Comment               = single_pulse
Data_Format           = 1D COMPLEX
Dim_Size              = 13107
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Site                  = JNM-ECA500II
Spectrometer          = DELTA2_NMR

Field_Strength        = 11.7473579[T]  (500[MHz])
X_Acq_Duration        = 1.74587904[s]
X_Domain              = 1H
X_Freq                = 500.15991521[MHz]
X_Offset              = 5.0[ppm]
X_Points              = 16384
X_Prescans            = 1
X_Resolution          = 0.57277737[Hz]
X_Sweep               = 9.38438438[kHz]
X_Sweep_Clipped       = 7.50750751[kHz]
Irr_Domain            = Proton
Irr_Freq              = 500.15991521[MHz]
Irr_Offset            = 5.0[ppm]
Tri_Domain            = Proton
Tri_Freq              = 500.15991521[MHz]
Tri_Offset            = 5.0[ppm]
Clipped               = FALSE
Scans                 = 64
Total_Scans           = 64

Relaxation_Delay      = 5[s]
Recvr_Gain            = 32
Temp_Get              = 21.7[dC]
X_90_Width            = 7.68[us]
X_Acq_Time            = 1.74587904[s]
X_Angle               = 45[deg]
X_Atn                 = 3.2[dB]
X_Pulse               = 3.84[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Presat          = FALSE
Initial_Wait          = 1[s]
Repetition_Time       = 6.74587904[s]
```

^{13}C {^{1}H} NMR Spectrum of **10Ab** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11668[ppm], 77[ppm] )
thresh( 0.29277[%], 1 )

Derived from: srg-01-54-13C-last_Carbon-1-1.j

Filename                  = srg-01-54-13C-last
Author                    = delta
Experiment                = carbon.jxp
Sample_Id                 = srg-01-54-13C-last
Solvent                   = CHLOROFORM-D
Actual_Start_Time         = 11-DEC-2021 20:49:
Revision_Time             =  6-AUG-2022 18:15:

Comment                   = single pulse decou
Data_Format               = 1D COMPLEX
Dim_Size                  = 26214
X_Domain                  = Carbon13
Dim_Title                 = Carbon13
Dim_Units                 = [ppm]
Dimensions                = X
Site                      = JNM-ECZ400S/L1
Spectrometer              = JNM-ECZ400S/L1

Field_Strength            = 9.36627879[T]  (400
X_Acq_Duration            = 1.03809024[s]
X_Domain                  = 13C
X_Freq                    = 100.27385305[MHz]
X_Offset                  = 100[ppm]
X_Points                  = 32768
X_Prescans                = 4
X_Resolution              = 0.96330739[Hz]
X_Sweep                   = 31.56565657[kHz]
X_Sweep_Clipped           = 25.25252525[kHz]
Irr_Domain                = Proton
Irr_Freq                  = 398.78219799[MHz]
Irr_Offfset               = 5[ppm]
Blanking                  = 5.0[us]
Clipped                   = FALSE
Scans                     = 44141
Total_Scans               = 44141

Relaxation_Delay          = 2[s]
Recvr_Gain                = 50
Temp_Get                  = 23.1[dC]
X_90_Width                = 9.95[us]
X_Acq_Time                = 1.03809024[s]
X_Angle                   = 30[deg]
X_Atn                     = 4[dB]
X_Pulse                   = 3.31666667[us]
Irr_Atn_Dec               = 27.227[dB]
Irr_Atn_Dec_Calc          = 27.227[dB]
Irr_Atn_Dec_Default_Calc  = 27.227[dB]
Irr_Atn_Noe               = 27.227[dB]
Irr_Dec_Bandwidth_Hz      = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm     = 11.99303459[ppm]
Irr_Dec_Freq              = 398.78219799[MHz]
Irr_Dec_Merit_Factor      = 2.2
Irr_Decoupling            = TRUE
Irr_Noe                   = TRUE
```

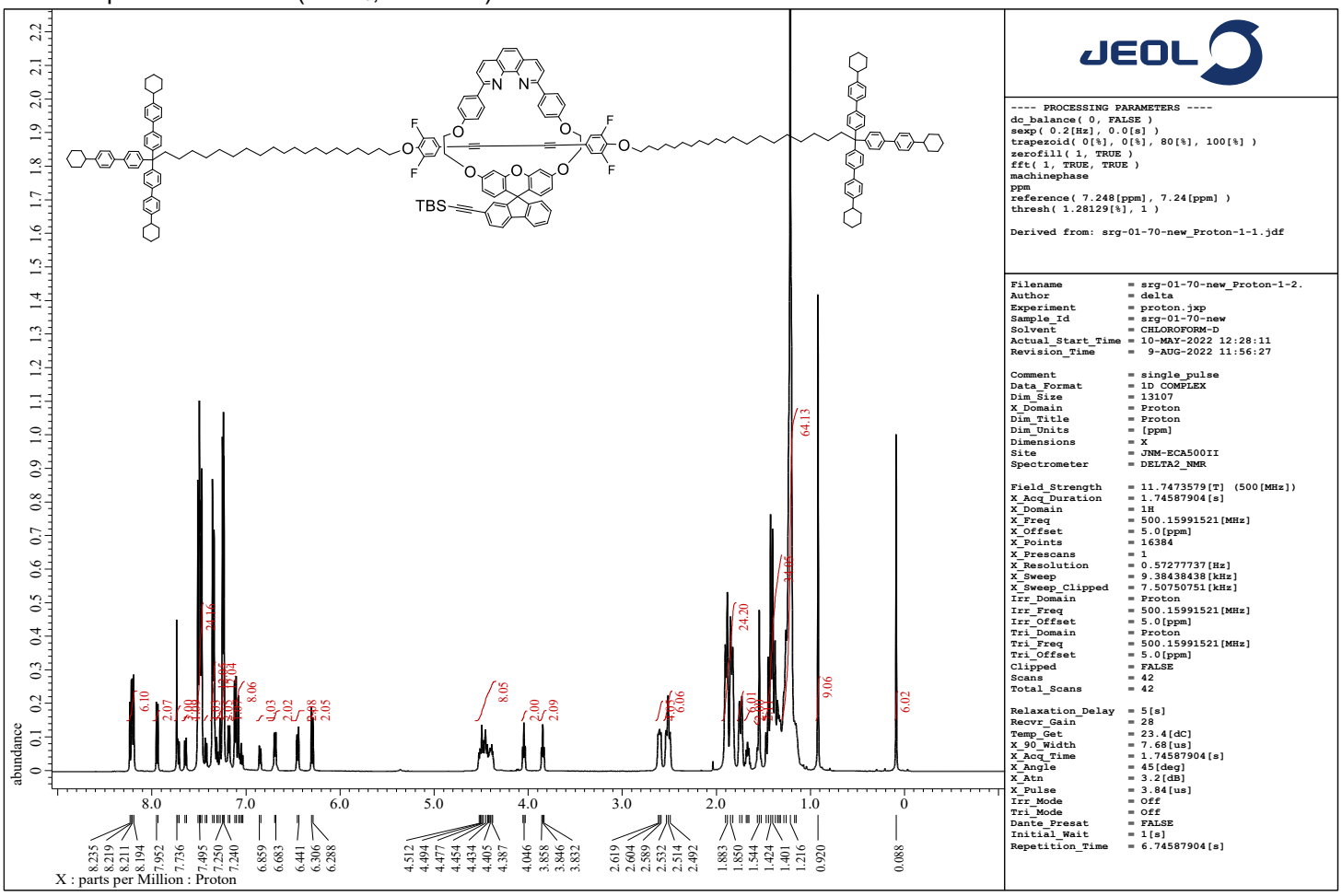
^{19}F NMR Spectrum of **10Ab** ($CDCl_3$, 377 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 57.99146[%], 1 )
phase( 40.71631, 0, 50[%] )

Derived from: srg-01-54_10-4.jdf
```

```
Filename        = srg-01-54_10-5.jdf
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 16-MAR-2022 11:48:40

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 16

Temp_Get        = 298.0736[K]
Filter_Factor   = 220
```

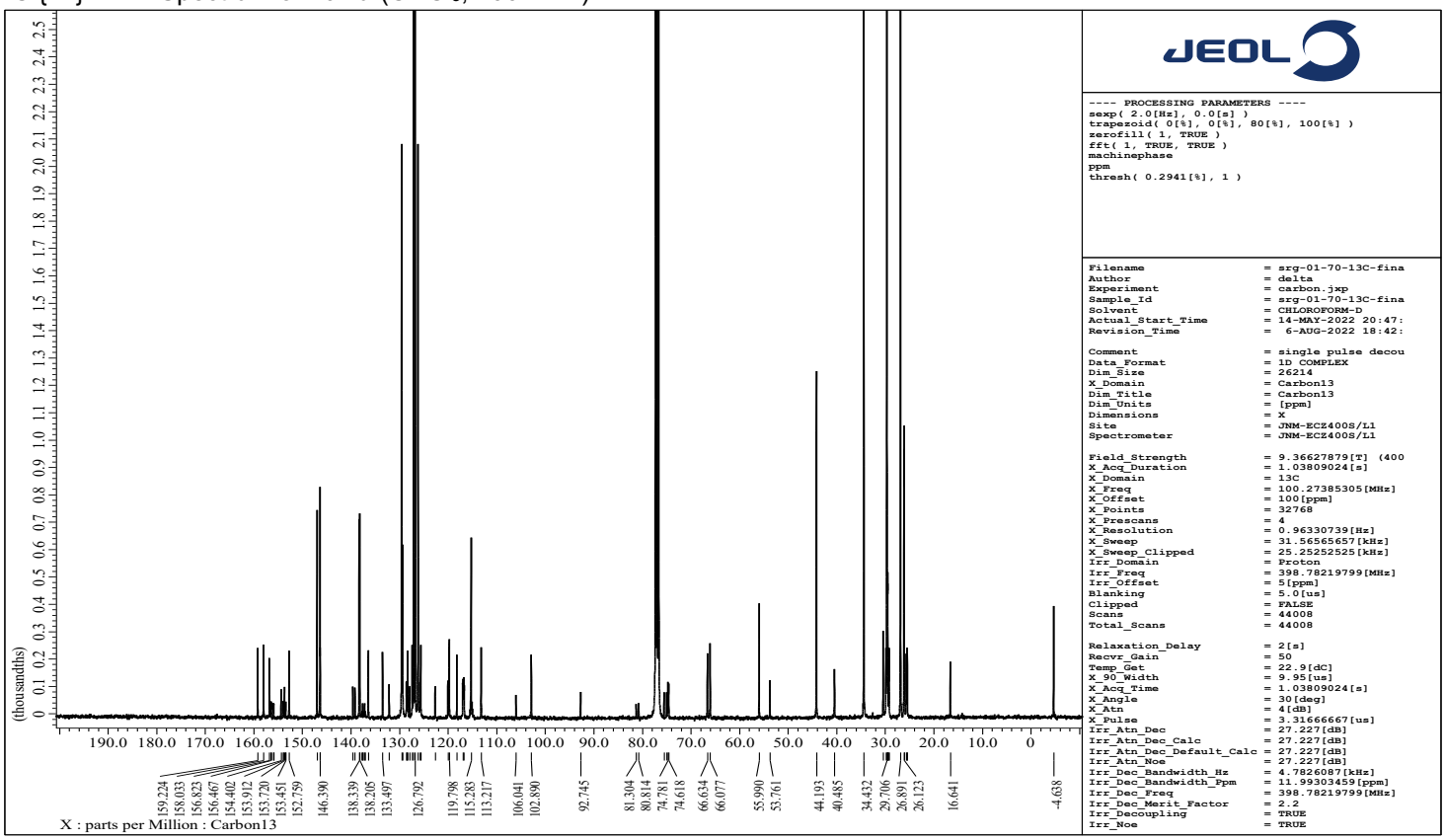
^{1}H NMR Spectrum of **10Ad** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 1.28129[%], 1 )

Derived from: srg-01-70-new_Proton-1-1.jdf
```

```
Filename            = srg-01-70-new_Proton-1-2.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = srg-01-70-new
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 10-MAY-2022 12:28:11
Revision_Time       =  9-AUG-2022 11:56:27

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 42
Total_Scans         = 42

Relaxation_Delay    = 5[s]
Recvr_Gain          = 28
Temp_Get            = 23.4[dC]
X_90_Width          = 7.68[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.2[dB]
X_Pulse             = 3.84[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

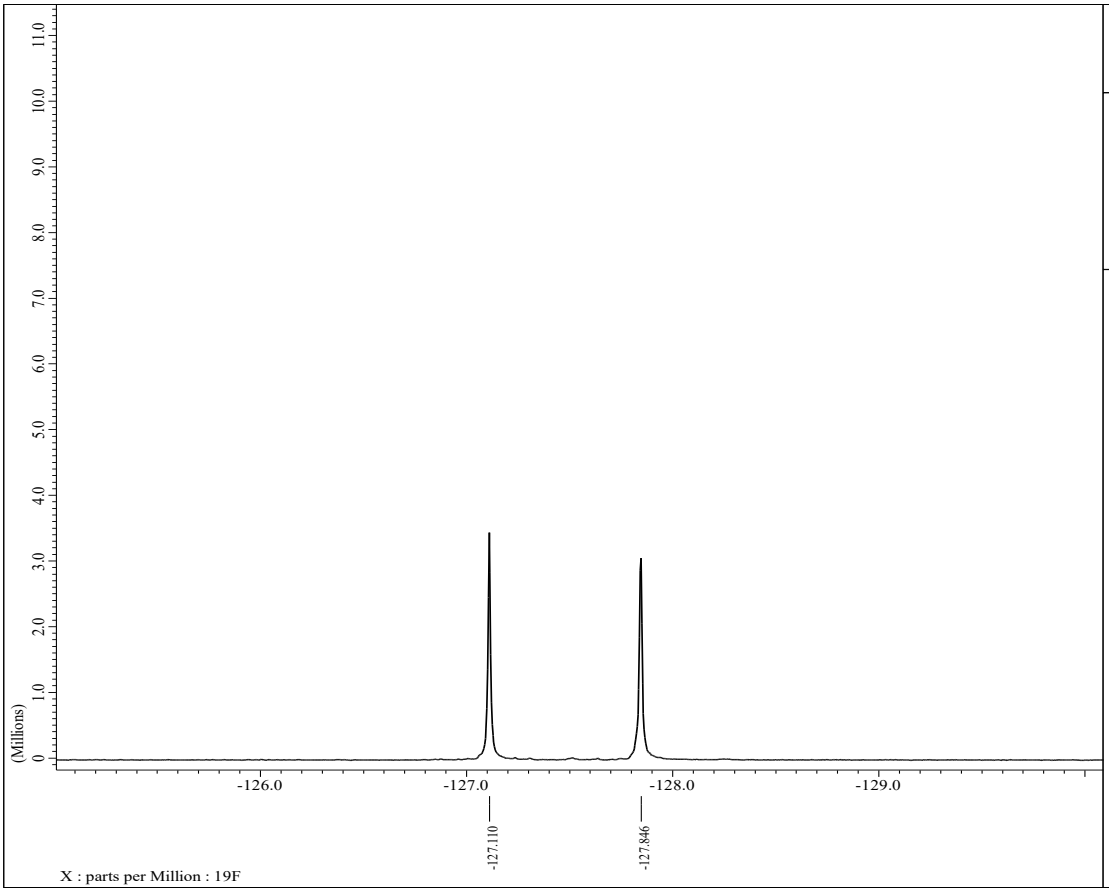
^{13}C {^{1}H} NMR Spectrum of **10Ad** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 0.2941[%], 1 )
```

```
Filename                = srg-01-70-13C-fina
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = srg-01-70-13C-fina
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 14-MAY-2022 20:47:
Revision_Time           =  6-AUG-2022 18:42:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.27385305[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5.0[us]
Clipped                 = FALSE
Scans                   = 44008
Total_Scans             = 44008

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 22.9[dC]
X_90_Width              = 9.95[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 4[dB]
X_Pulse                 = 3.31666667[us]
Irr_Atn_Dec             = 27.227[dB]
Irr_Atn_Dec_Calc        = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe             = 27.227[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.99303459[ppm]
Irr_Dec_Freq            = 398.78219799[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
```

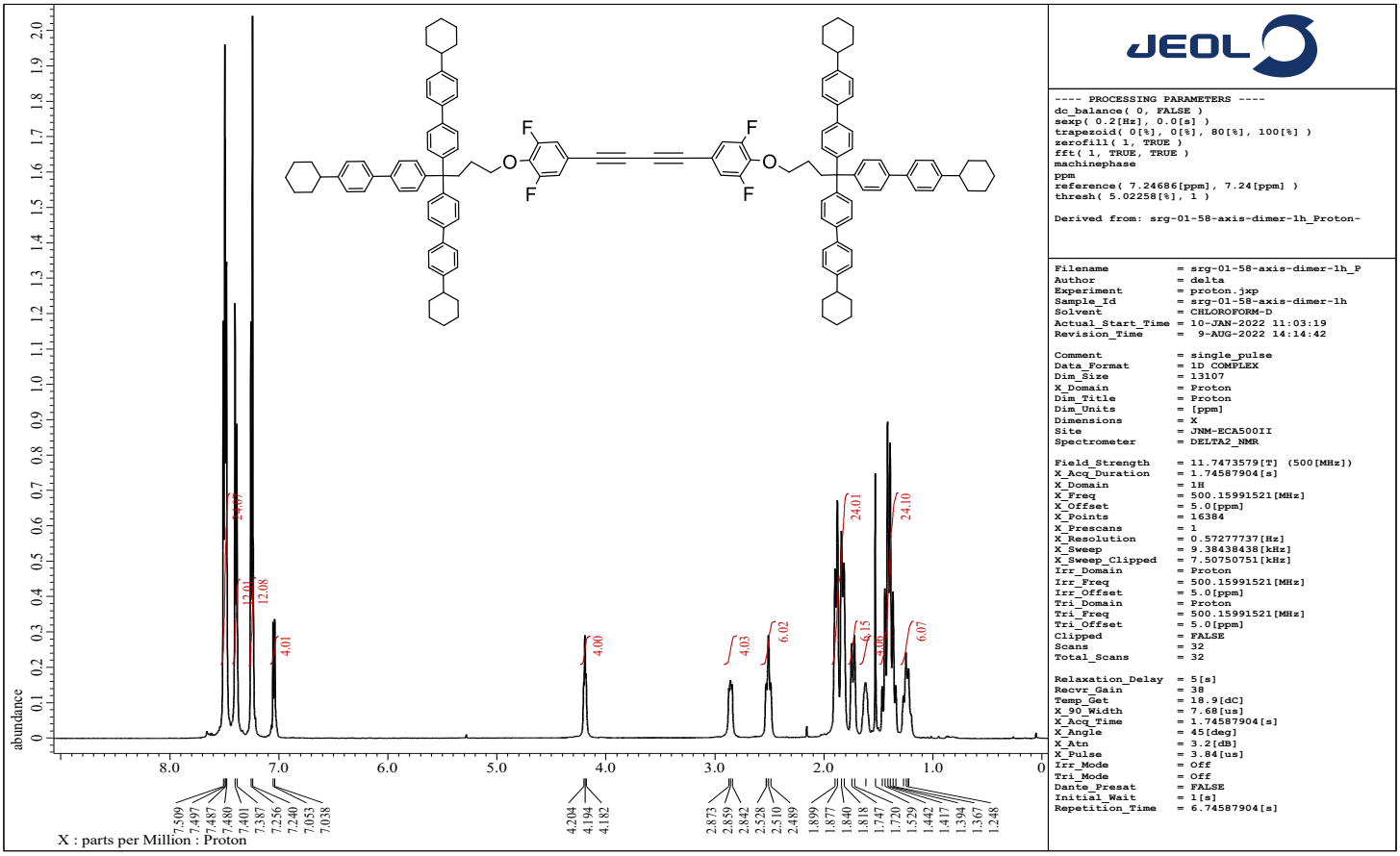
^{19}F NMR Spectrum of **10Ad** ($CDCl_3$, 377 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: srg_01_70_10-3.jdf
```

```
Filename        = srg_01_70_10-4.jdf
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 12-JUN-2022 14:35:22

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 32

Temp_Get        = 298.0269[K]
Filter_Factor   = 220
```

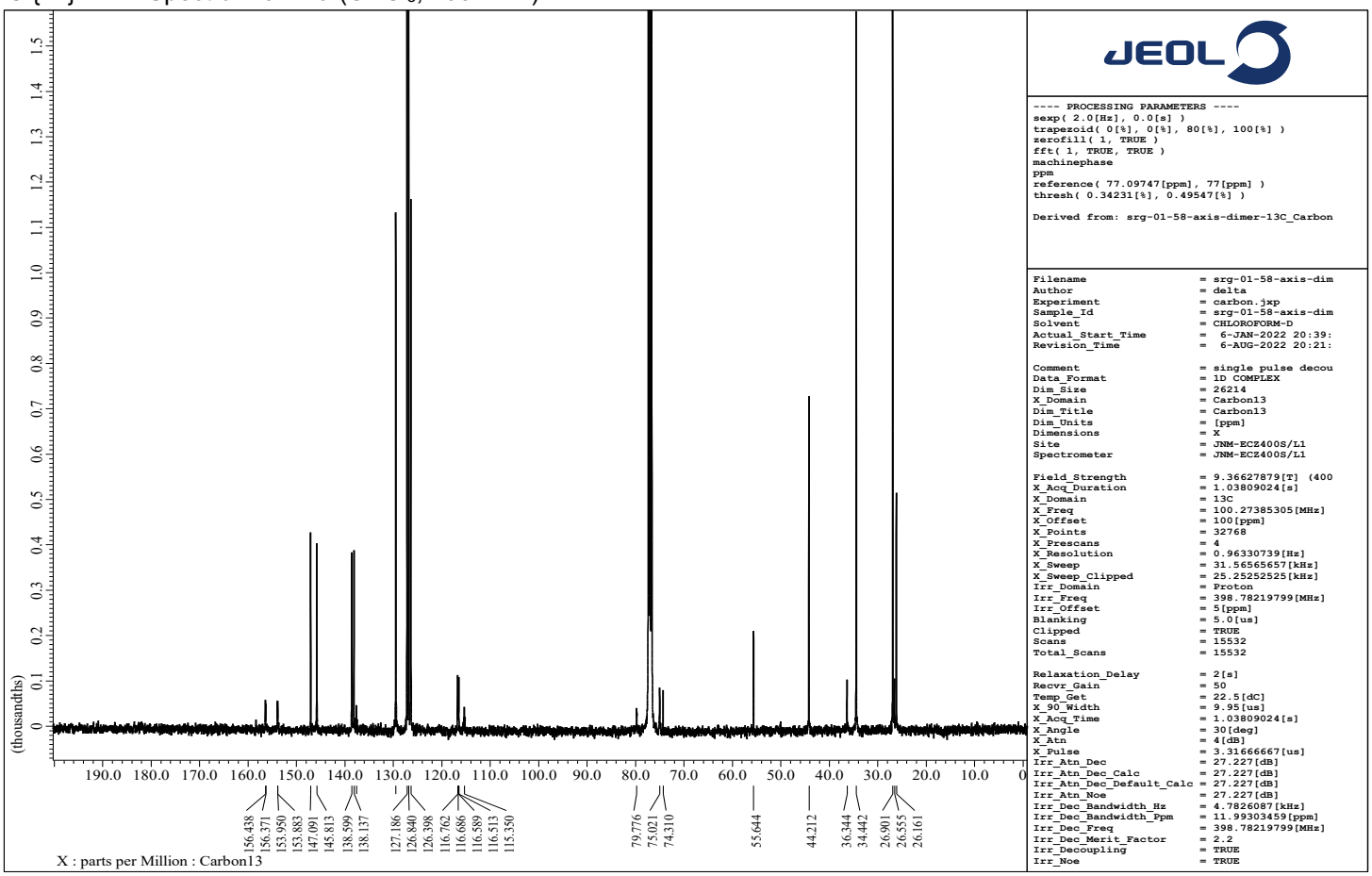
^{1}H NMR Spectrum of **11a** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.24686[ppm], 7.24[ppm] )
thresh( 5.02258[%], 1 )

Derived from: srg-01-58-axis-dimer-1h_Proton-
```

Filename	= srg-01-58-axis-dimer-1h_P
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-58-axis-dimer-1h
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 10-JAN-2022 11:03:19
Revision_Time	= 9-AUG-2022 14:14:42
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 18.9[dC]
X_90_Width	= 7.68[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 3.2[dB]
X_Pulse	= 3.84[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.74587904[s]

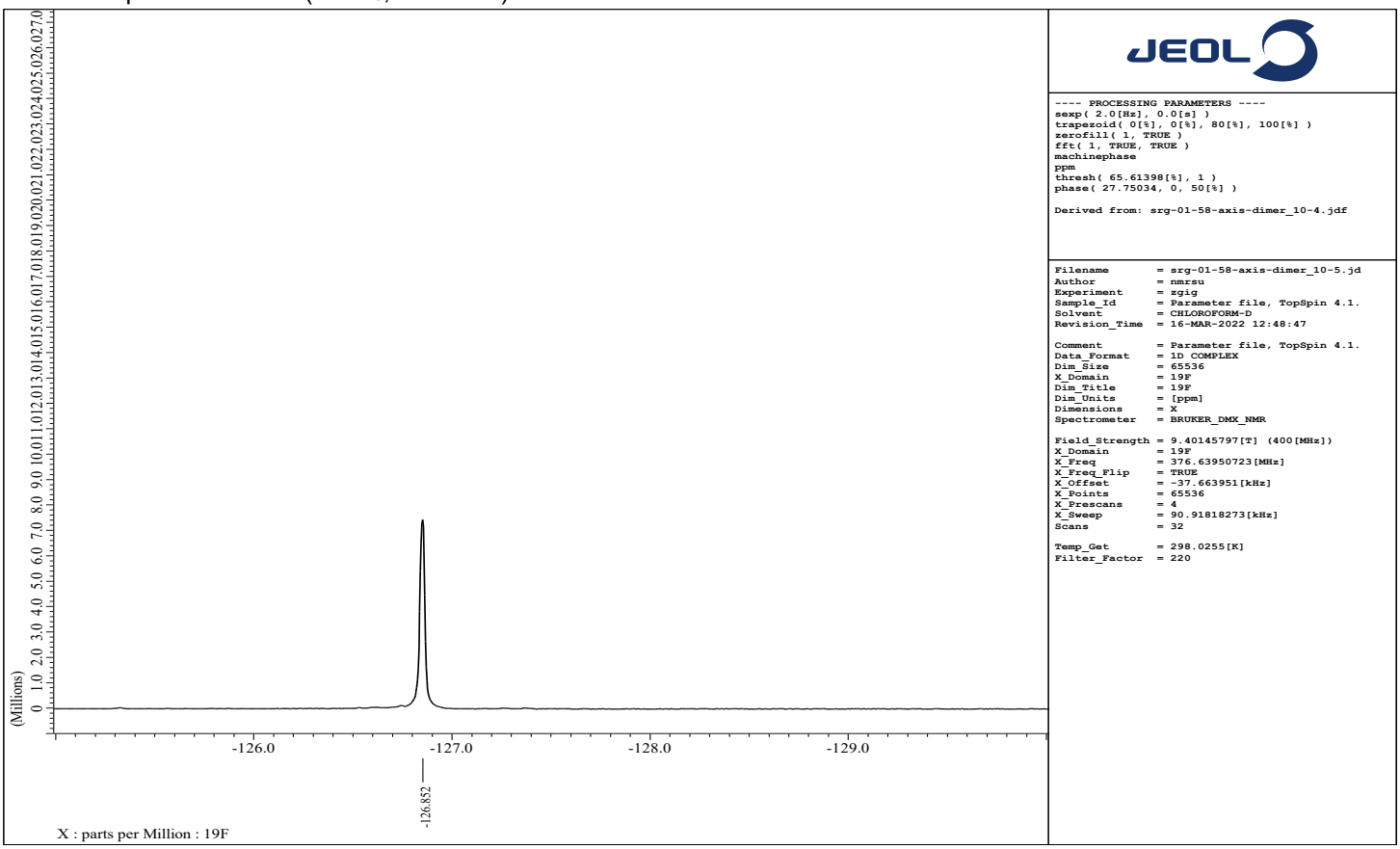
^{13}C {^{1}H} NMR Spectrum of **11a** ($CDCl_3$, 100 MHz).



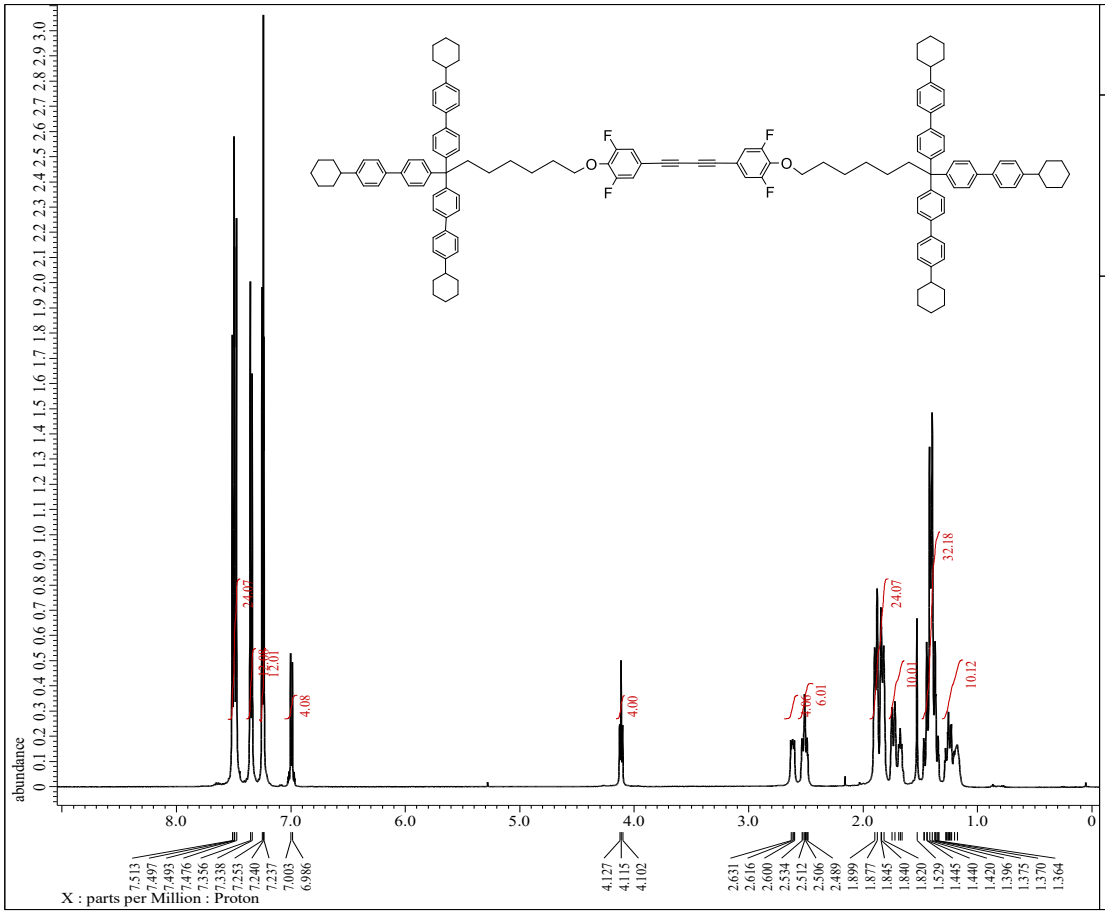

```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.09747[ppm], 77[ppm] )
thresh( 0.34231[%], 0.49547[%] )

Derived from: srg-01-58-axis-dimer-13C_Carbon
```

Filename	= srg-01-58-axis-dim
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-58-axis-dim
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-JAN-2022 20:39:
Revision_Time	= 6-AUG-2022 20:21:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= TRUE
Scans	= 15532
Total_Scans	= 15532
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22.5[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

^{19}F NMR Spectrum of **11a** ($CDCl_3$, 377 MHz).



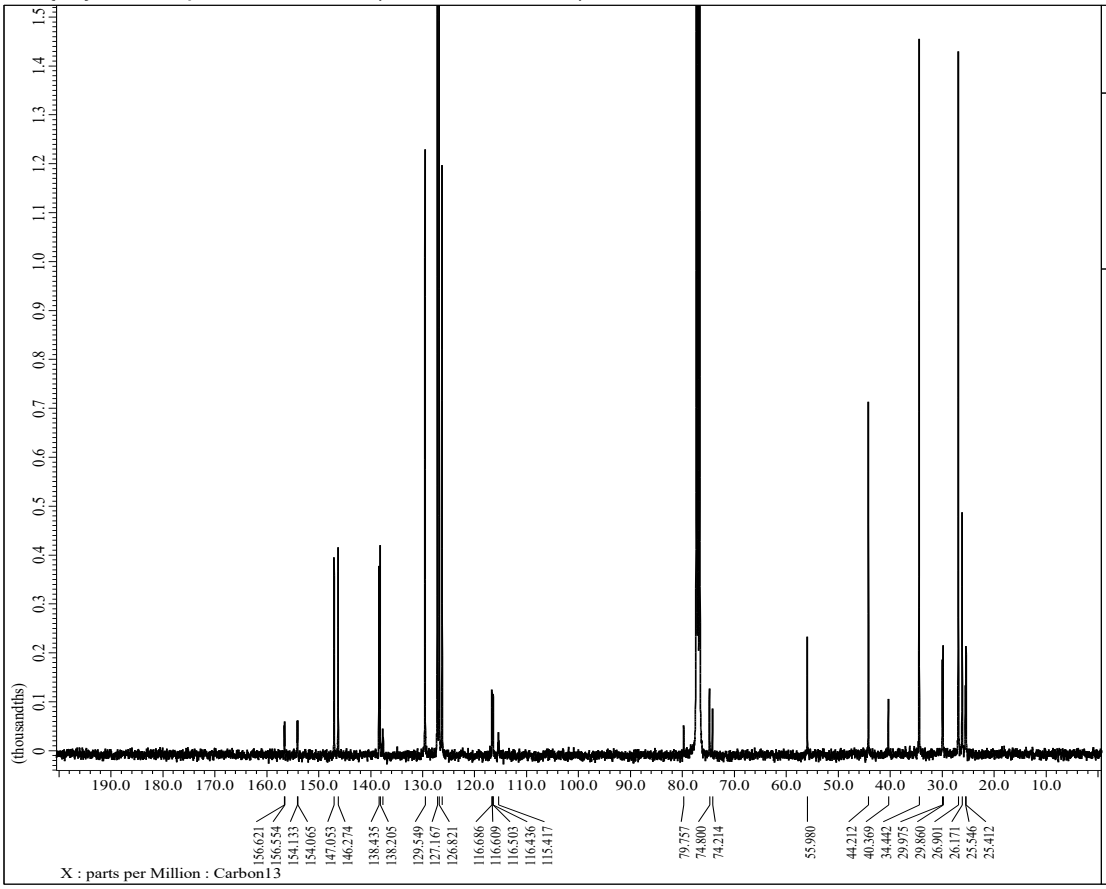
^{1}H NMR Spectrum of **11b** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
thresh( 3.62012[%], 1 )

Derived from: srg-0154-axis-dimer finaaal_Pro
```

```
Filename            = srg-0154-axis-dimer finaa
Author              = delta
Experiment          = proton.jxp
Sample_Id           = srg-0154-axis-dimer finaa
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 23-JAN-2022 16:34:25
Revision_Time       =  9-AUG-2022 14:41:58

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECA500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 32
Total_Scans         = 32

Relaxation_Delay    = 5[s]
Recvr_Gain          = 44
Temp_Get            = 18.6[dC]
X_90_Width          = 7.68[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.2[dB]
X_Pulse             = 3.84[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

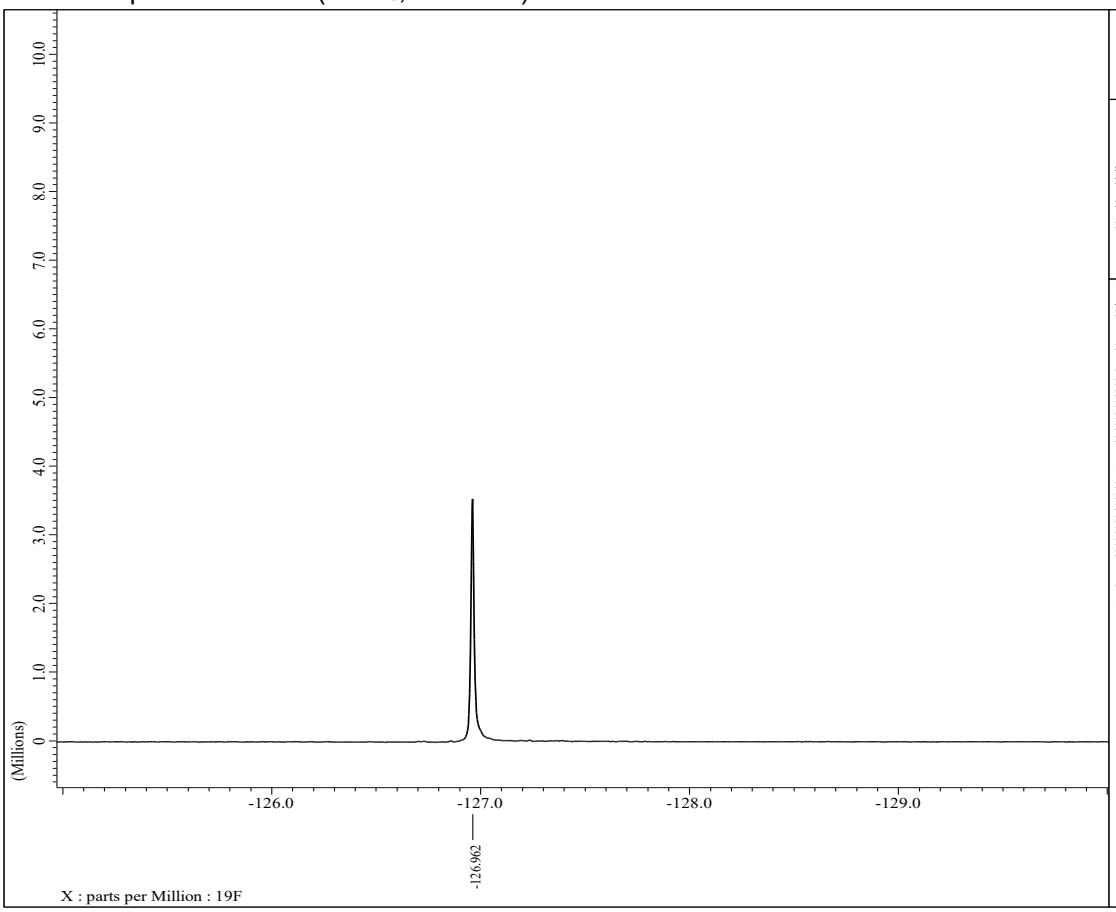
^{13}C {^{1}H} NMR Spectrum of **11b** ($CDCl_3$, 100 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 0.30951[%], 1 )

Derived from: srg-01-54-axis-dimer-13C_Carbon
```

```
Filename                = srg-01-54-axis-dim
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = srg-01-54-axis-dim
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 18-JAN-2022 20:24:
Revision_Time           =  6-AUG-2022 21:55:

Comment                 = single pulse decou
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Site                    = JNM-ECZ400S/L1
Spectrometer            = JNM-ECZ400S/L1

Field_Strength          = 9.36627879[T] (400
X_Acq_Duration          = 1.03809024[s]
X_Domain                = 13C
X_Freq                  = 100.27385305[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 0.96330739[Hz]
X_Sweep                 = 31.56565657[kHz]
X_Sweep_Clipped         = 25.25252525[kHz]
Irr_Domain              = Proton
Irr_Freq                = 398.78219799[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 5.0[us]
Clipped                 = FALSE
Scans                   = 16161
Total_Scans             = 16161

Relaxation_Delay        = 2[s]
Recvr_Gain              = 50
Temp_Get                = 23.3[dC]
X_90_Width              = 9.95[us]
X_Acq_Time              = 1.03809024[s]
X_Angle                 = 30[deg]
X_Atn                   = 4[dB]
X_Pulse                 = 3.31666667[us]
Irr_Atn_Dec             = 27.227[dB]
Irr_Atn_Dec_Calc        = 27.227[dB]
Irr_Atn_Dec_Default_Calc = 27.227[dB]
Irr_Atn_Noe             = 27.227[dB]
Irr_Dec_Bandwidth_Hz    = 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm   = 11.99303459[ppm]
Irr_Dec_Freq            = 398.78219799[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
```

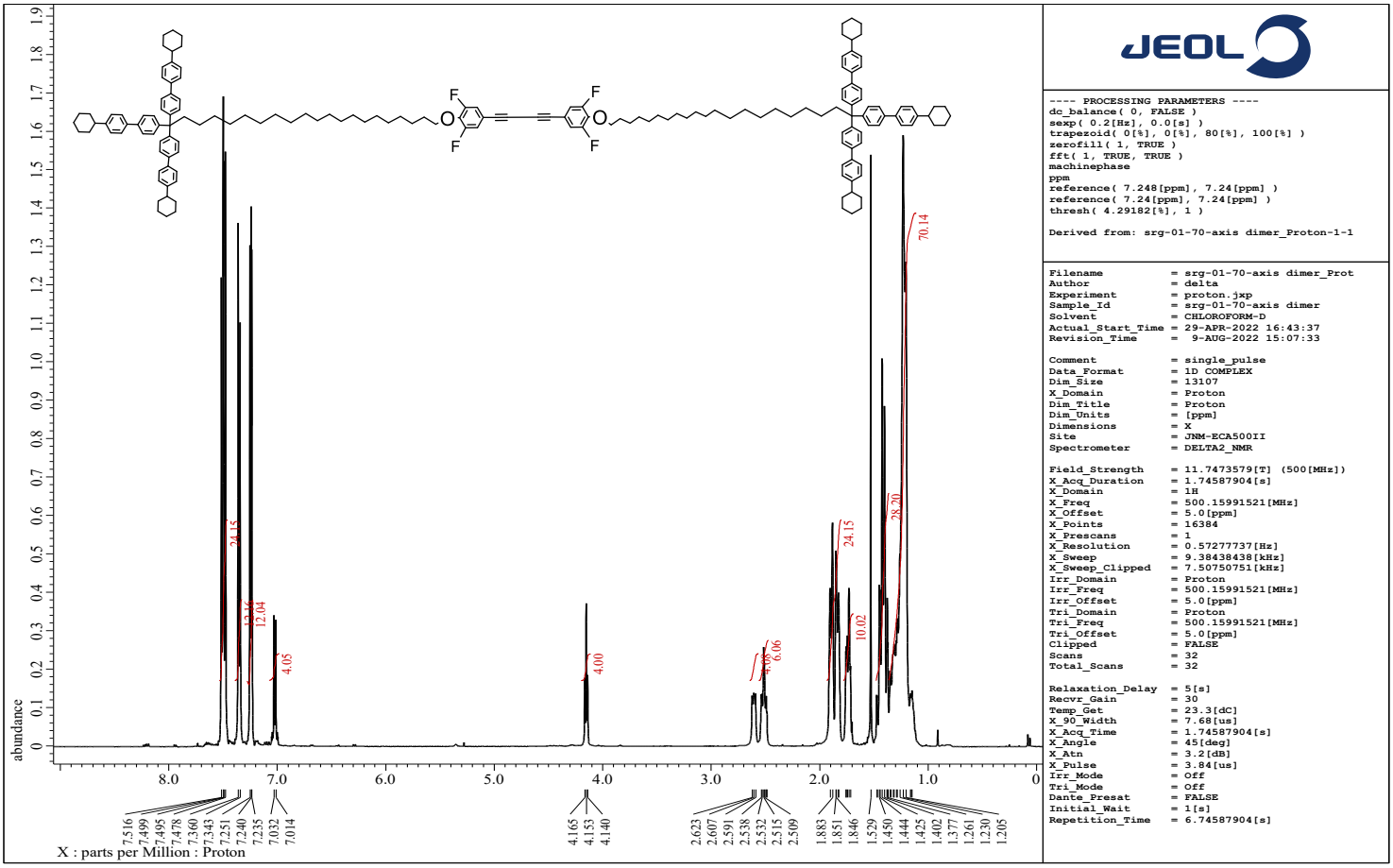
^{19}F NMR Spectrum of **11b** ($CDCl_3$, 377 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
thresh( 27.30328[%], 1 )
phase( 33.09089, 0, 50[%] )

Derived from: srg-01-54-axis dimer_10-4.jdf
```

```
Filename        = srg-01-54-axis dimer_10-5.jd
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 16-MAR-2022 12:37:43

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 16

Temp_Get        = 298.0773[K]
Filter_Factor   = 220
```

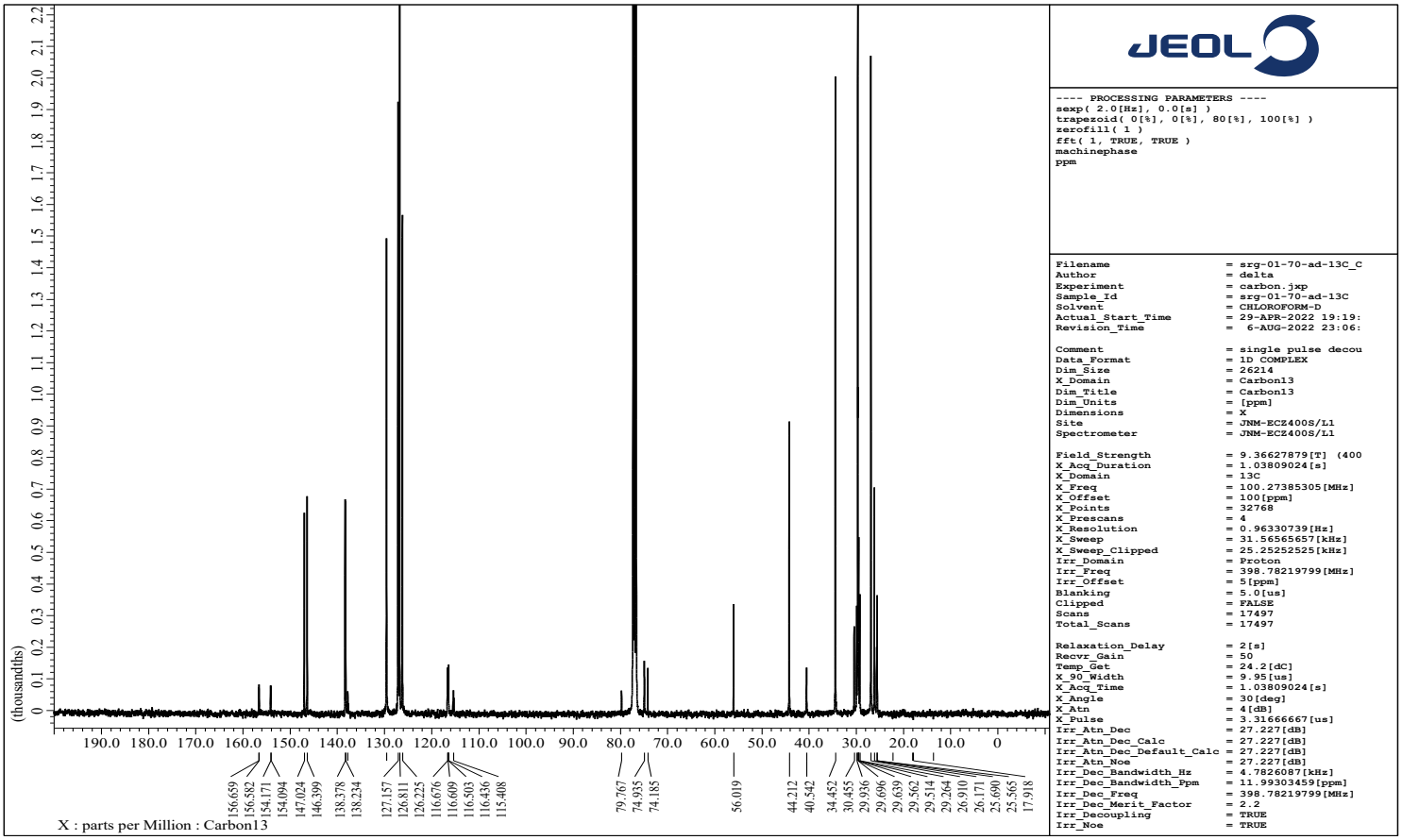
^{1}H NMR Spectrum of **11d** ($CDCl_3$, 500 MHz).




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 7.248[ppm], 7.24[ppm] )
reference( 7.24[ppm], 7.24[ppm] )
thresh( 4.29182[%], 1 )

Derived from: srg-01-70-axis dimer_Proton-1-1
```

Filename	= srg-01-70-axis dimer_Prot
Author	= delta
Experiment	= proton.jxp
Sample_Id	= srg-01-70-axis dimer
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 29-APR-2022 16:43:37
Revision_Time	= 9-AUG-2022 15:07:33
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECA500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 5[s]
Recvr_Gain	= 30
Temp_Get	= 23.3[dC]
X_90_Width	= 7.68[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 3.2[dB]
X_Pulse	= 3.84[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.74587904[s]

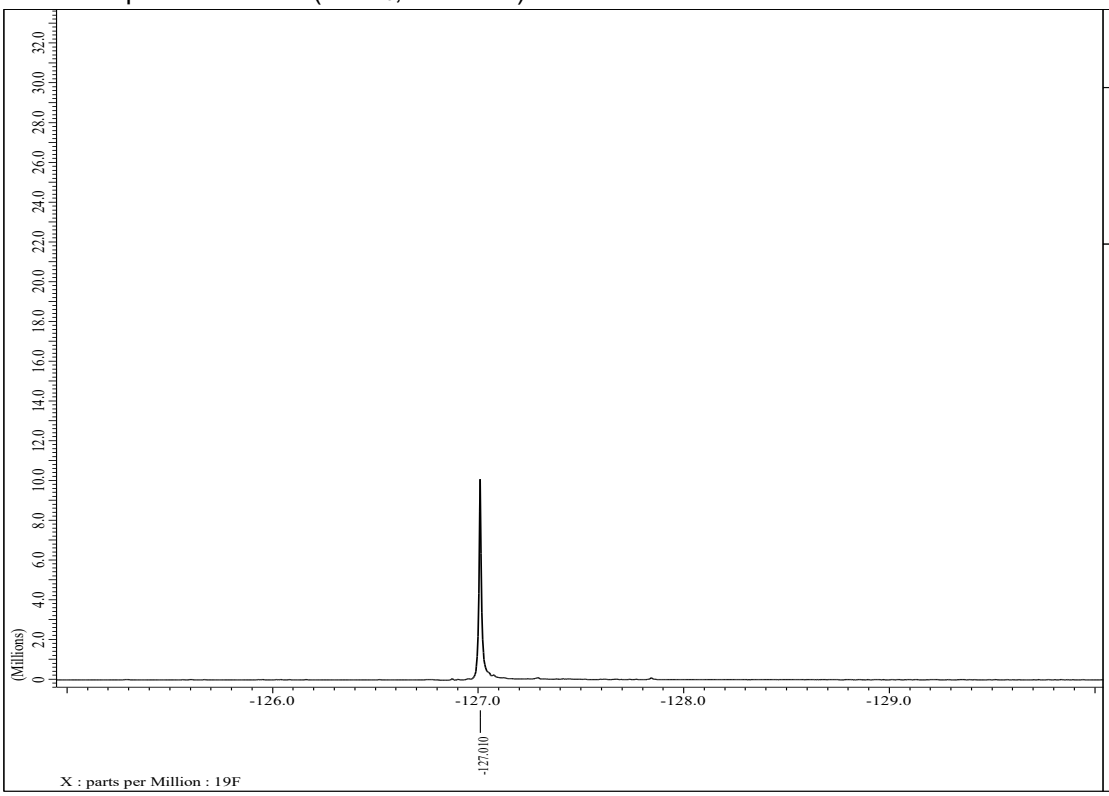
^{13}C {^{1}H} NMR Spectrum of **11d** ($CDCl_3$, 100 MHz)




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= srg-01-70-ad-13C_C
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= srg-01-70-ad-13C
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 29-APR-2022 19:19:
Revision_Time	= 6-AUG-2022 23:06:
Comment	= single pulse decou
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECZ400S/L1
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.36627879[T] (400
X_Acq_Duration	= 1.03809024[s]
X_Domain	= 13C
X_Freq	= 100.27385305[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.96330739[Hz]
X_Sweep	= 31.56565657[kHz]
X_Sweep_Clipped	= 25.25252525[kHz]
Irr_Domain	= Proton
Irr_Freq	= 398.78219799[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 5.0[us]
Clipped	= FALSE
Scans	= 17497
Total_Scans	= 17497
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 24.2[dC]
X_90_Width	= 9.95[us]
X_Acq_Time	= 1.03809024[s]
X_Angle	= 30[deg]
X_Atn	= 4[dB]
X_Pulse	= 3.31666667[us]
Irr_Atn_Dec	= 27.227[dB]
Irr_Atn_Dec_Calc	= 27.227[dB]
Irr_Atn_Dec_Default_Calc	= 27.227[dB]
Irr_Atn_Noe	= 27.227[dB]
Irr_Dec_Bandwidth_Hz	= 4.7826087[kHz]
Irr_Dec_Bandwidth_Ppm	= 11.99303459[ppm]
Irr_Dec_Freq	= 398.78219799[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE

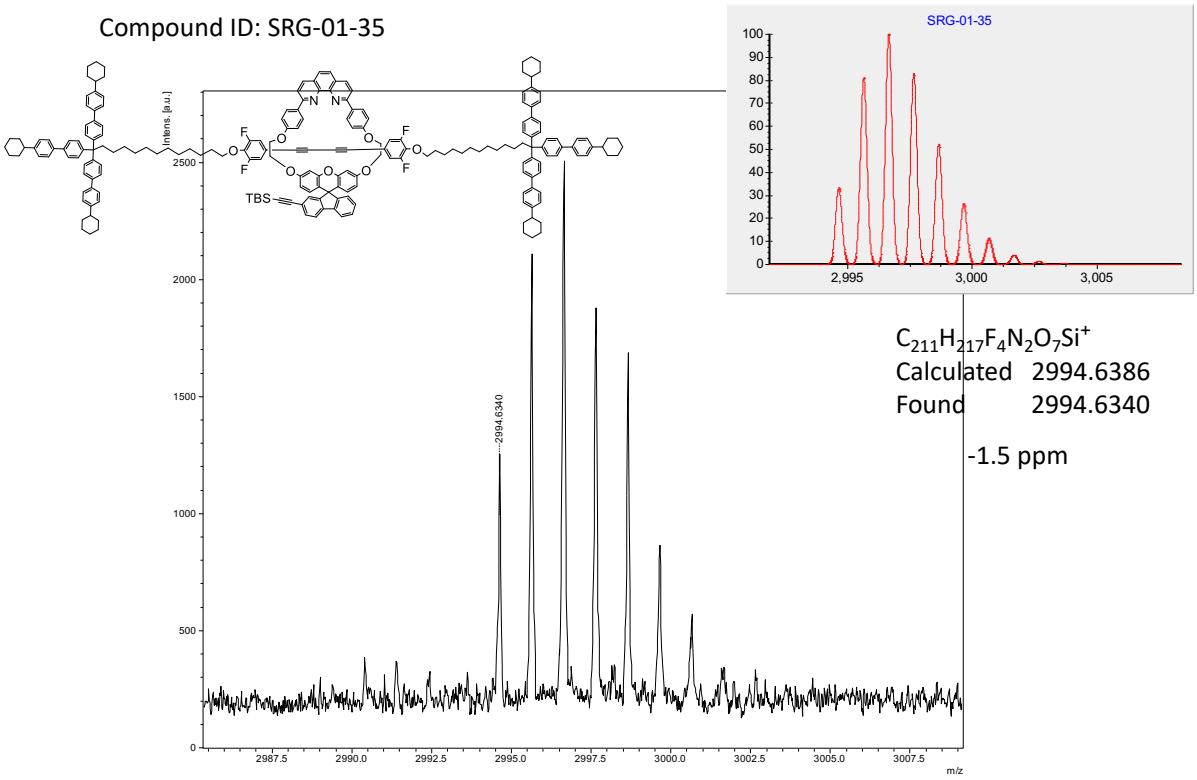
^{19}F NMR Spectrum of **11d** ($CDCl_3$, 377 MHz).




```
---- PROCESSING PARAMETERS ----
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: srg_01_70_AD_20-2.jdf
```

```
Filename        = srg_01_70_AD_20-3.jdf
Author          = nmrsu
Experiment      = zgig
Sample_Id       = Parameter file, TopSpin 4.1.
Solvent         = CHLOROFORM-D
Revision_Time   = 12-JUN-2022 14:28:14

Comment         = Parameter file, TopSpin 4.1.
Data_Format     = 1D COMPLEX
Dim_Size        = 65536
X_Domain        = 19F
Dim_Title       = 19F
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = BRUKER_DMX_NMR

Field_Strength = 9.40145797[T] (400[MHz])
X_Domain        = 19F
X_Freq          = 376.63950723[MHz]
X_Freq_Flip     = TRUE
X_Offset        = -37.663951[kHz]
X_Points        = 65536
X_Prescans      = 4
X_Sweep         = 90.91818273[kHz]
Scans           = 32

Temp_Get        = 293.027[K]
Filter_Factor   = 220
```

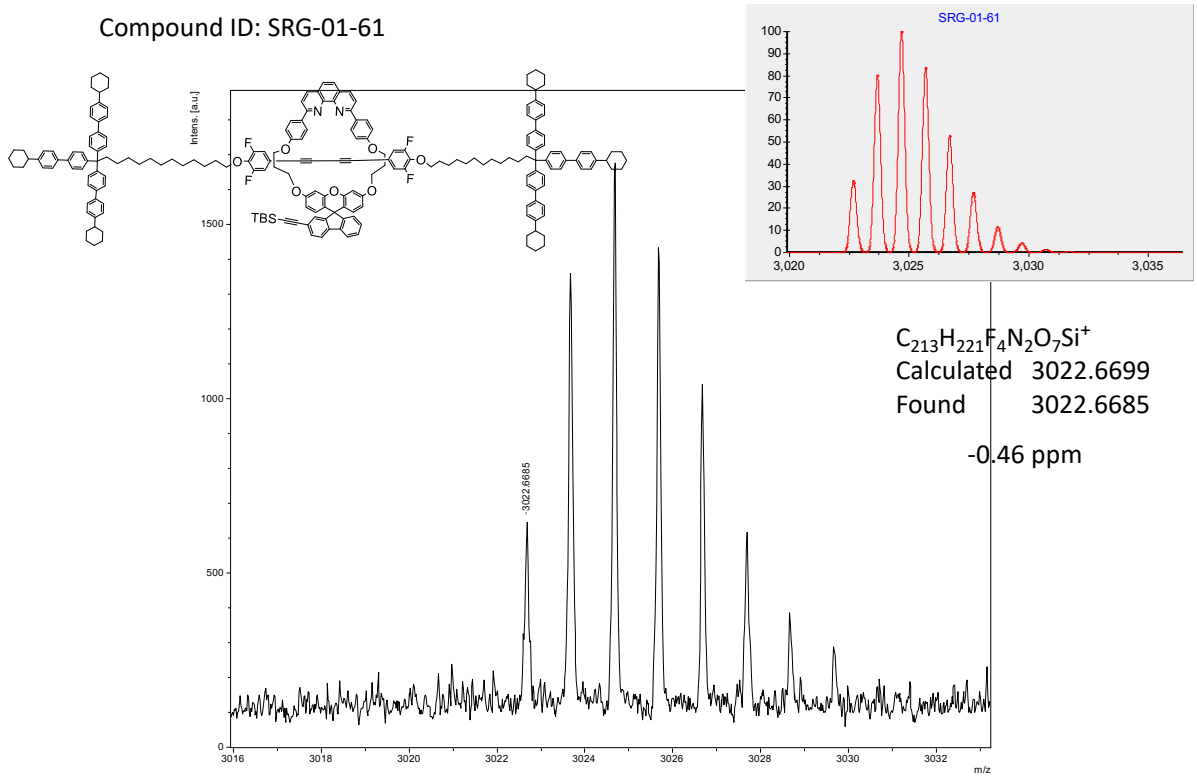
HRMS Spectrum of **10Ac**.



$C_{211}H_{217}F_4N_2O_7Si^+$
Calculated 2994.6386
Found 2994.6340

-1.5 ppm

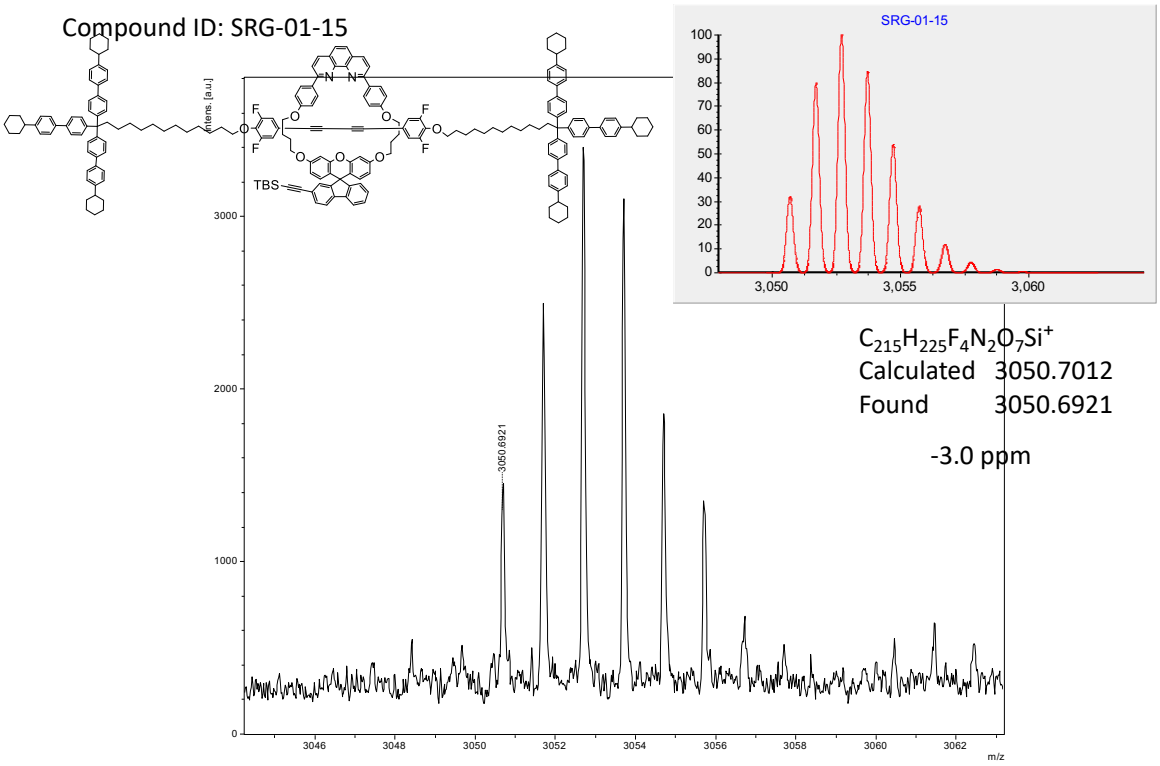
HRMS Spectrum of **10Bc**.



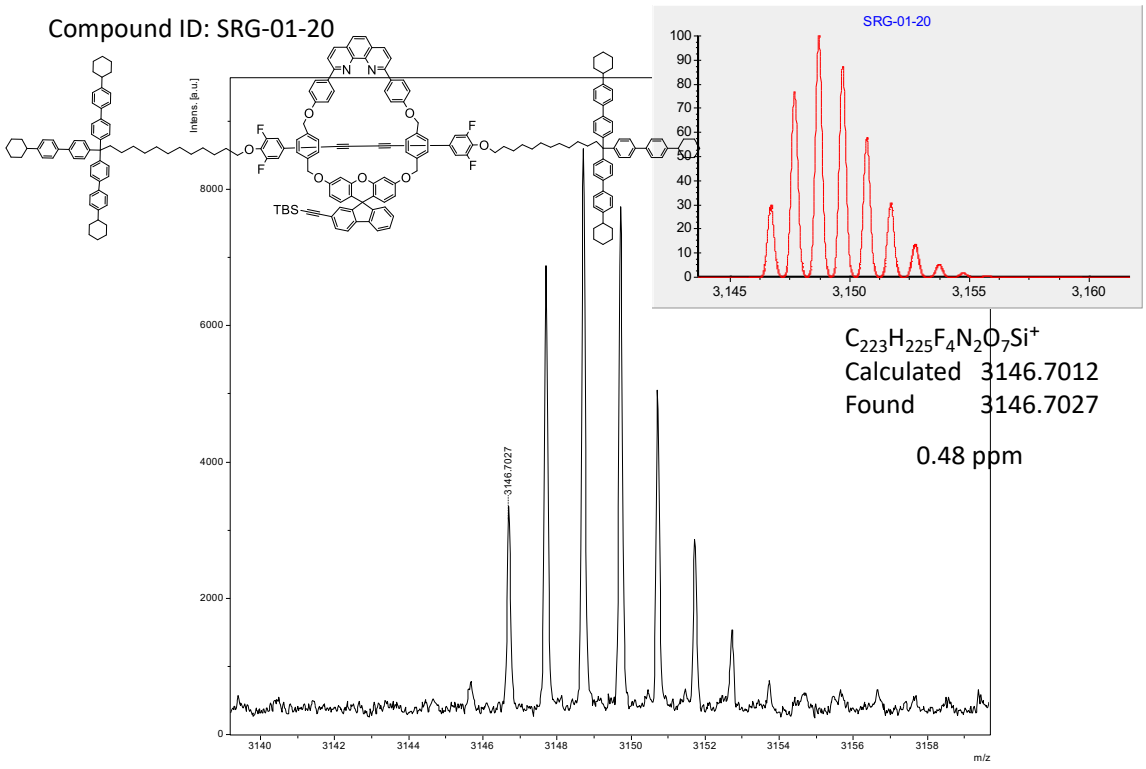
$C_{213}H_{221}F_4N_2O_7Si^+$
Calculated 3022.6699
Found 3022.6685

-0.46 ppm

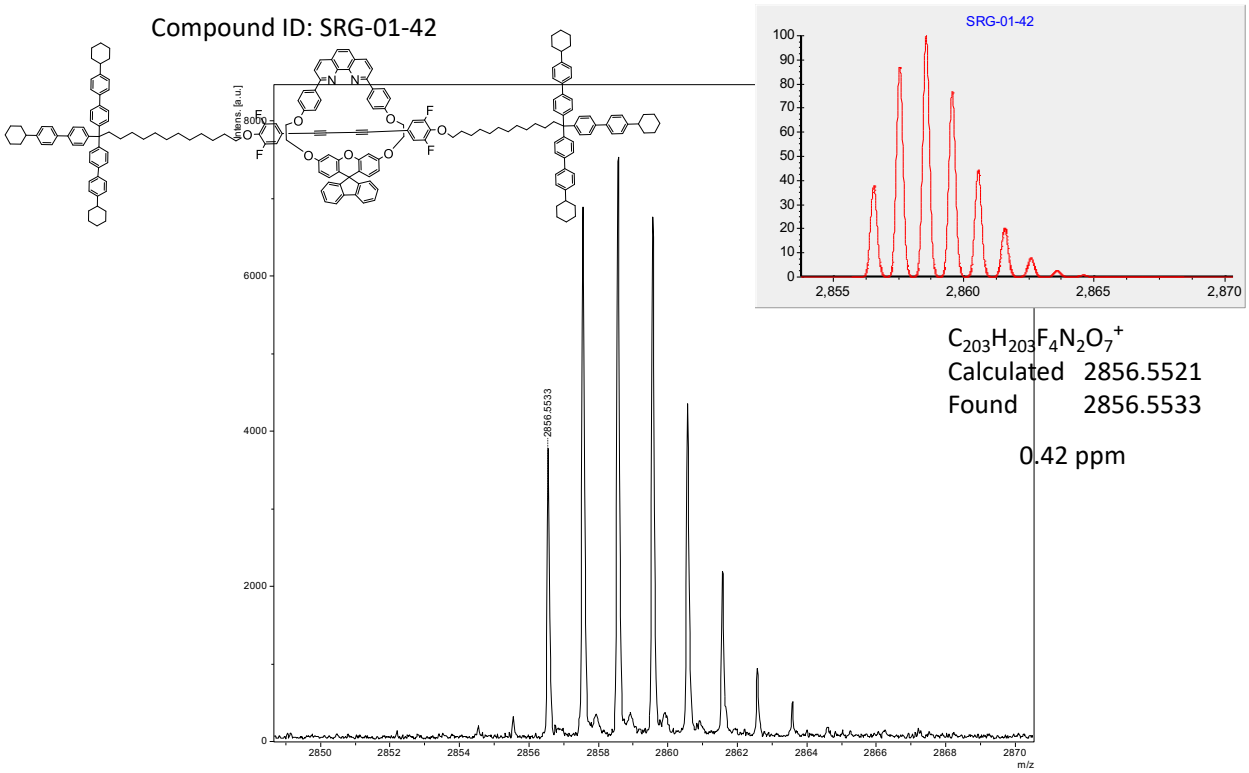
HRMS Spectrum of **10Cc**.



HRMS Spectrum of **10Dc**.



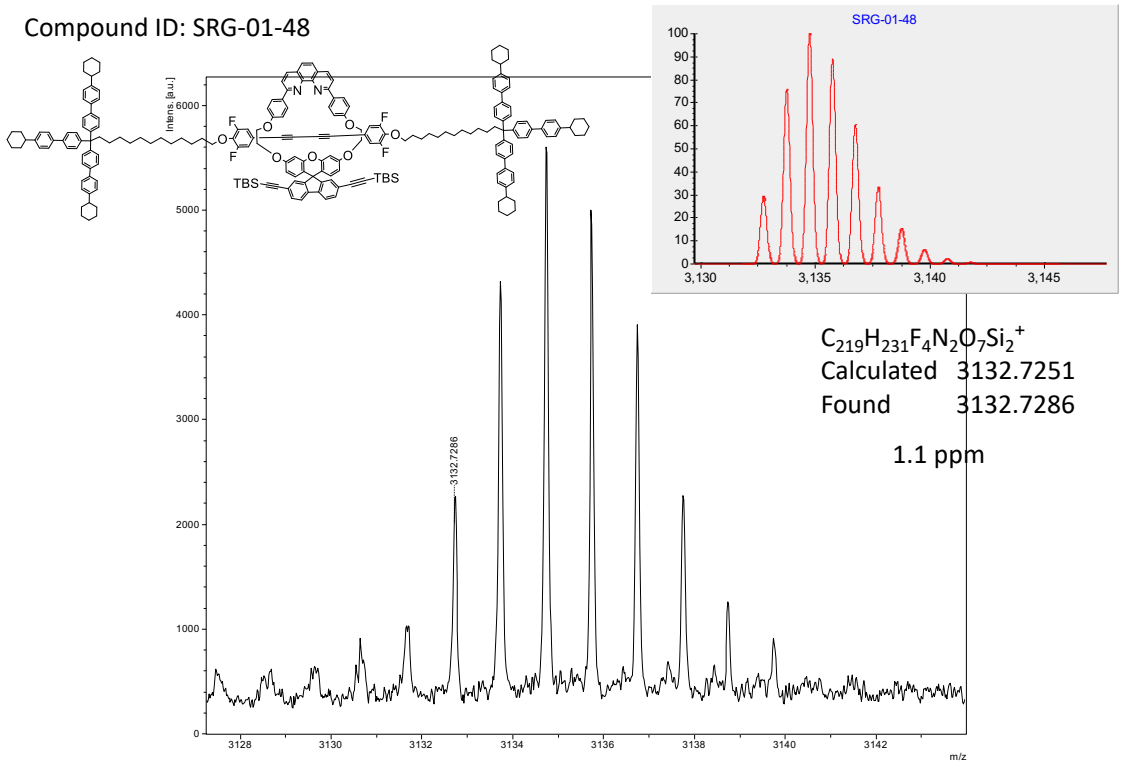
HRMS Spectrum of **18**.



$C_{203}H_{203}F_4N_2O_7^+$
Calculated 2856.5521
Found 2856.5533

0.42 ppm

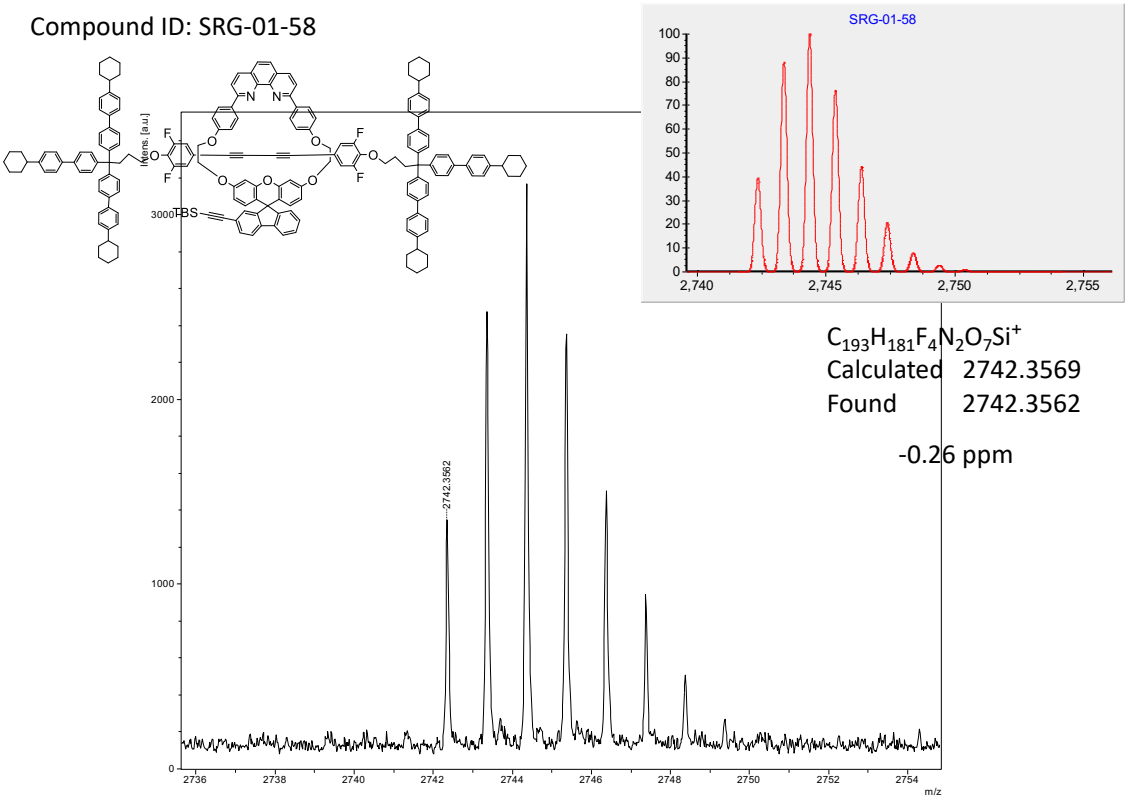
HRMS Spectrum of **19**.



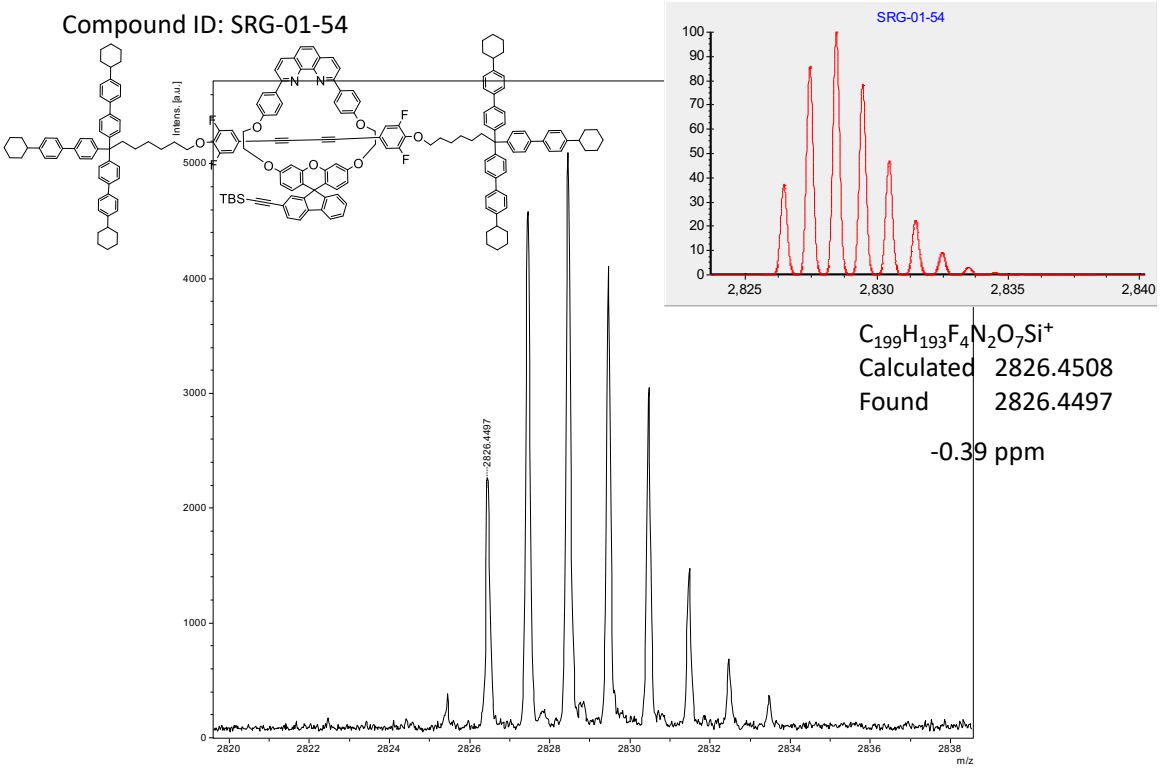
$C_{219}H_{231}F_4N_2O_7Si_2^+$
Calculated 3132.7251
Found 3132.7286

1.1 ppm

HRMS Spectrum of **10Aa**.



HRMS Spectrum of **10Ab**.



HRMS Spectrum of **10Ad**.