Supporting Information

K_2CO_3/TFAA-Activated Diarylation of α-Sulfonyl *o*-Hydroxyacetophenones with *o*-Nitroaryl Disulfides to Construct Sulfonyl Bis-*o*-nitroaryl Acetophenones

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Table of Contents

Experimental Section

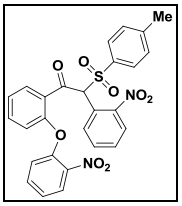
General. All reagents and solvents were obtained from commercial sources and used without further purification. Reactions were routinely carried out under an atmosphere of dry air with magnetic stirring. Products in organic solvents were dried with anhydrous magnesium sulfate before concentration in vacuo. Melting points were determined with a SMP3 melting apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Varian INOVA-400 spectrometer operating at 400 and at 100 MHz, respectively. Chemical shifts (δ) are reported in parts per million (ppm) and the coupling constants (*J*) are given in Hertz. High resolution mass spectra (HRMS) were measured with a mass spectrometer Finnigan/Thermo Quest MAT 95XL. X-ray crystal structures were obtained with an Enraf-Nonius FR-590 diffractometer (CAD4, Kappa CCD).

A representative synthetic procedure of skeleton 4 is as follows: $CuBr_2$ (470 mg, 2.1 mmol) was added to commercially available substituted *o*-hydroxyacetophenones (1.0 mmol) in EtOAc (20 mL) at 25 ^{o}C. The reaction mixture was stirred at reflux for 15 h. The reaction mixture was cooled to 25 ^{o}C and the solvent was filtered and washed with saturated $NaHCO_3$ (10 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Without further purification, sulfinic sodium salts (RSO_2Na, 1.2 mmol) was added to the resulting bromo *o*-hydroxyacetophenones in EtOH (10 mL) at 25 ^{o}C. The reaction mixture was stirred at reflux for 4 h. The reaction mixture was cooled to 25 ^{o}C and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 8/1~4/1) afforded **4**. For the starting substrates **4**, these materials were known compounds and the analytical data (e.g., HRMS, ^{1}H and $^{13}C\{^{1}H\}$ NMR) were consistent with those in the references ((a) M.-Y. Chang, Y.-L. Tsai and Y.-L. Chang, *J. Org. Chem.*, 2020, **85**, 1033-1043; b) N.-C. Hsueh, M.-C. Tsai, M.-Y. Chang and H.-Y. Chen, *J. Org. Chem.*, 2019, **84**, 15915-15925; c) M.-Y. Chang, H.-Y. Chen and Y.-L. Tsai, *J. Org. Chem.*, 2019, **84**, 326-337).

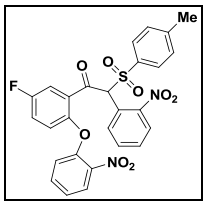
A representative synthetic procedure of skeleton 5 is as follows: H_2O_2 (30% in H_2O, 1 mL) was added to a solution of commercially available *o*-nitrothiophenol (1.0 mmol) in hexafluoroisopropanol (HFIP, 10 mL) at 25 ^{o}C. Then, the reaction mixture was stirred at 25 ^{o}C for 3 h. The reaction mixture was diluted with brine (5 mL) and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried,

filtered and evaporated to afford crude 3-arylacroliens under reduced pressure. Purification on silica gel (hexanes/EtOAc = 25/1~10/1) afforded **5**. For the starting substrates **5**, these materials were known compounds and the analytical data (e.g., HRMS, ^{1}H and $^{13}C\{^{1}H\}$ NMR) were consistent with those in the references (V. Kesavan, D. Bonnet-Delpon and J.-P. Bégué, *Synthesis*, 2000, 223-225).

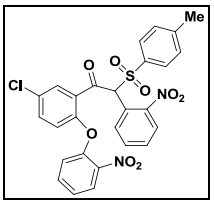
A representative synthetic procedure of skeleton 6 is as follows: Trifluoroacetic anhydride (TFAA, 210 mg, 1.0 mmol) was added to a solution of **5** (0.5 mmol) in MeCN (10 mL) at 25 ^{o}C. The reaction mixture was stirred at 25 ^{o}C for 10 min. K_2CO_3 (140 mg, 1.0 mmol) and **4** (0.5 mmol) were added to the reaction mixture at 25 ^{o}C in onestep. The reaction mixture was stirred at 25 ^{o}C for 20 h. Then, the solvent was concentrated to afford the crude products. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 8/1~4/1) afforded **6**.



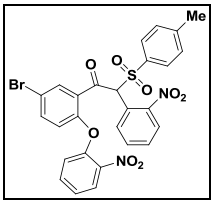
1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6a). Yield = 92% (245 mg); White solid; mp = 170-172 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{27}H_{21}N_2O_8S$ 533.1019, found 533.1021; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.95 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.80 (dd, *J* = 1.6, 8.0 Hz, 2H), 7.64 (s, 1H), 7.62-7.47 (m, 5H), 7.38 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.33 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.12 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.03 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.61 (dd, *J* = 0.4, 8.0 Hz, 1H), 2.34 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.9, 155.5, 149.6, 147.9, 145.3, 141.5, 134.8, 134.7, 134.5, 132.9, 132.6, 131.2, 130.1, 129.4 (2x), 129.4 (2x), 128.5, 126.1, 125.4, 125.2, 123.8, 123.2, 122.3, 116.7, 71.2, 21.5. Single-crystal X-Ray diagram: crystal of compound **6a** was grown by slow diffusion of EtOAc into a solution of compound **6a** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the triclinic crystal system, space group P-1, *a* = 8.08950(10) Å, *b* = 10.48040(10) Å, *c* = 14.8834(2) Å, *V* = 1185.61(3) Å^3, *Z* = 2, d_{calcd} = 1.492 g/cm^3, *F*(000) = 552.0, 2*θ* range 4.042~49.996^{o}, R indices (all data) R1 = 0.0372, wR2 = 0.0871.



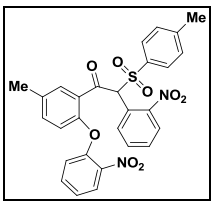
1-(5-Fluoro-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6b). Yield = 86% (237 mg); White solid; mp = 169-171 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{20}FN_2O_8S$ 551.0924, found 551.0929; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.96 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.81 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.61-7.57 (m, 2H), 7.62 (s, 1H), 7.53-7.48 (m, 4H), 7.35 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.10 (ddd, *J* = 3.6, 7.2, 9.2 Hz, 1H), 7.04 (dd, *J* = 0.8, 8.0 Hz, 1H), 6.62 (dd, *J* = 4.0, 9.2 Hz, 1H), 2.36 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 188.8, 158.2 (d, *J* = 244.1 Hz), 151.6 (d, *J* = 3.1 Hz), 149.6, 148.0, 145.6, 141.5, 135.0, 134.3, 133.0, 132.6, 130.2, 129.6 (d, *J* = 6.8 Hz), 129.5 (2x), 129.4 (2x), 126.2, 125.6, 125.3, 123.0, 122.1, 121.5 (d, *J* = 23.5 Hz), 118.5 (d, *J* = 7.6 Hz), 117.4 (d, *J* = 25.0 Hz), 71.1, 21.6; ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ -117.57~-117.62 (m, 1F).



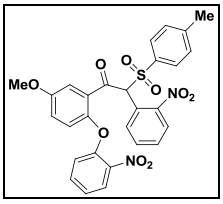
1-(5-Chloro-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6c). Yield = 87% (246 mg); White solid; mp = 165-167 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{20}ClN_2O_8S$ 567.0629, found 567.0633; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.81 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.70 (d, *J* = 2.8 Hz, 1H), 7.64-7.60 (m, 2H), 7.62 (s, 1H), 7.54-7.49 (m, 3H), 7.38 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.30 (dd, *J* = 2.8, 8.8 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 1H), 6.54 (d, *J* = 8.8 Hz, 1H), 2.36 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 188.9, 154.3, 149.6, 147.2, 145.6 (2x), 141.6, 135.1, 134.4, 132.9, 132.6, 130.8, 130.2, 129.43 (2x), 129.40 (2x), 129.3, 129.1, 126.3, 126.1, 125.3, 123.6, 122.0, 117.6, 71.2, 21.7.



1-(5-Bromo-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6d). Yield = 83% (253 mg); White solid; mp = 176-178 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{20}BrN_2O_8S$ 611.0124, found 611.0122; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (dd, *J* = 1.6, 8.0 Hz, 1H), 8.02 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.85 (d, *J* = 2.4 Hz, 1H), 7.82 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.66-7.63 (m, 2H), 7.62 (s, 1H), 7.56-7.50 (m, 3H), 7.45 (dt, *J* = 2.8, 8.4 Hz, 1H), 7.40 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.12 (dd, *J* = 1.6, 8.0 Hz, 1H), 6.47 (d, *J* = 8.8 Hz, 1H), 2.37 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 188.9, 154.9, 149.7, 147.2, 145.6, 141.7, 137.2, 135.1, 134.5, 133.9, 133.0, 132.7, 130.2, 129.8, 129.5 (4x), 126.4, 126.2, 125.3, 123.8, 122.1, 117.8, 116.4, 71.2, 21.7.

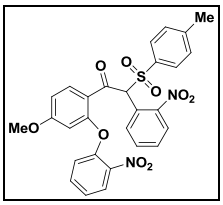


1-(5-Methyl-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6e). Yield = 86% (235 mg); White solid; mp = 196-198 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{28}H_{23}N_2O_8S$ 547.1175, found 547.1177; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.99 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.82 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.64-7.48 (m, 7H), 7.32 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.20 (dt, *J* = 2.4, 8.4 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.03 (dd, *J* = 1.2, 8.0 Hz, 1H), 6.54 (d, *J* = 8.4 Hz, 1H), 2.37 (s, 3H), 2.32 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 190.1, 153.3, 149.7, 148.6, 145.3, 135.3, 134.7, 134.6, 133.9, 132.9, 132.8, 131.4, 130.0, 129.6 (2x), 129.4 (2x), 128.7, 126.1, 125.2, 125.0, 124.8, 122.9, 122.5, 117.1, 71.2, 21.6, 20.5.

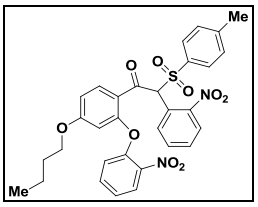


1-(5-Methoxy-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6f). Yield = 83% (233 mg); White solid; mp = 162-164 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{28}H_{23}N_2O_9S$ 563.1124, found 563.1127; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.94 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.83 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.61 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.58 (s, 1H), 7.54-7.48 (m, 4H), 7.31 (d, *J* = 3.2 Hz, 1H), 7.26 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.98-6.93 (m, 2H), 6.55 (d, *J* = 9.2 Hz, 1H), 3.81 (s, 3H), 2.37 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.5, 155.9, 149.7, 149.5, 148.7, 145.4, 141.3, 134.6, 134.4, 132.9, 132.7,

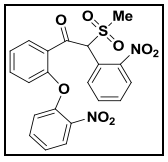
130.1, 129.6, 129.5 (2x), 129.4 (2x), 126.1, 125.3, 124.4, 122.4, 121.9, 121.6, 119.6, 114.1, 71.2, 55.9, 21.6.



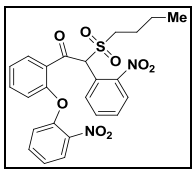
1-(4-Methoxy-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6g). Yield = 85% (239 mg); White solid; mp = 143-145 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{28}H_{23}N_2O_9S$ 563.1124, found 563.1130; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.89 (d, *J* = 5.2 Hz, 1H), 7.88 (dd, *J* = 4.8, 8.0 Hz, 1H), 7.79 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.63 (s, 1H), 7.59-7.45 (m, 5H), 7.33 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.01 (dd, *J* = 0.8, 8.0 Hz, 1H), 6.64 (dd, *J* = 4.0, 8.8 Hz, 1H), 6.03 (d, *J* = 2.0 Hz, 1H), 3.67 (s, 3H), 2.33 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 187.7, 165.1, 157.8, 149.6, 147.4, 145.1, 141.5, 134.9, 134.6, 133.7, 132.8, 132.4, 129.9, 129.4 (2x), 129.2 (2x), 126.1, 125.6, 125.2, 123.4, 122.7, 120.8, 108.9, 102.7, 71.0, 55.6, 21.5.



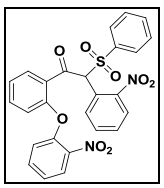
1-(4-*n*-Butoxy-2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6h). Yield = 83% (251 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{31}H_{29}N_2O_9S$ 605.1594, found 605.1596; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.91 (dd, *J* = 1.2, 7.6 Hz, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.81 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.63 (s, 1H), 7.61-7.46 (m, 5H), 7.35 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.05 (dd, *J* = 0.8, 8.0 Hz, 1H), 6.65 (dd, *J* = 2.4, 8.8 Hz, 1H), 6.03 (d, *J* = 2.4 Hz, 1H), 3.85 (t, *J* = 6.8 Hz, 2H), 2.35 (s, 3H), 1.68-1.61 (m, 2H), 1.42-1.33 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 187.7, 164.8, 157.9, 149.7, 147.6, 145.1, 141.6, 134.9, 134.7, 133.7, 132.8, 132.6, 129.9, 129.5 (2x), 129.3 (2x), 126.2, 125.6, 125.2, 123.5, 122.8, 120.7, 109.4, 103.2, 71.0, 68.3, 30.8, 21.6, 18.9, 13.6.



2-(Methylsulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6i). Yield = 80% (182 mg); White solid; mp = 185-187 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{21}H_{17}N_2O_8S$ 457.0706, found 457.0710; ^1H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 1.6, 8.4 Hz, 1H), 7.97 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.92 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.90 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.69-7.62 (m, 2H), 7.57 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.54 (s, 1H), 7.44 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.39 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.21 (dt, *J* = 0.8, 8.4 Hz, 1H), 7.13 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.64 (dd, *J* = 0.8, 8.4 Hz, 1H), 3.04 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 190.4, 156.0, 149.9, 147.5, 141.7, 135.3, 135.1, 133.3, 131.8, 131.7, 130.5, 127.7, 126.4, 125.9, 125.8, 124.0, 123.7, 122.3, 116.2, 70.9, 40.7. Single-crystal X-Ray diagram: crystal of compound **6i** was grown by slow diffusion of EtOAc into a solution of compound **6i** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the triclinic crystal system, space group P-1, *a* = 8.6503(4) Å, *b* = 9.5028(4) Å, *c* = 13.7219(5) Å, *V* = 73.673(3) $Å^3$, *Z* = 4, d_{calcd} = 1.538 g/cm^3, *F*(000) = 472.0, 2*θ* range 3.116~50°, R indices (all data) R1 = 0.0499, wR2 = 0.1149.

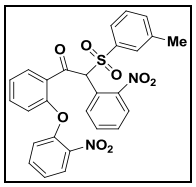


2-(*n*-Butylsulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6j). Yield = 80% (199 mg); White solid; mp = 130-132 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{24}H_{23}N_2O_8S$ 499.1175, found 499.1178; ^1H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.93-7.88 (m, 3H), 7.66-7.60 (m, 2H), 7.53 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.52 (s, 1H), 7.42 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.37 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.18 (dt, *J* = 0.8, 8.4 Hz, 1H), 7.06 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.62 (dd, *J* = 0.4, 8.4 Hz, 1H), 3.34-3.27 (m, 1H), 3.17-3.09 (m, 1H), 1.81-1.73 (m, 2H), 1.43-1.34 (m, 2H), 0.86 (t, *J* = 7.2 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 190.5, 155.7, 149.8, 147.4, 141.6, 135.1, 135.0, 133.1, 132.3, 131.5, 130.3, 127.8, 126.3, 125.8, 125.6, 123.9, 123.6, 122.0, 116.1, 69.8, 53.1, 23.0, 21.5, 13.4.

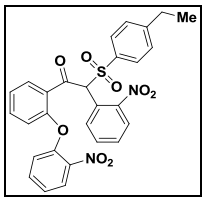


1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-(phenylsulfonyl)ethan-1-one (6k). Yield = 81% (210 mg); White solid; mp = 151-153 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{26}H_{19}N_2O_8S$ 519.0862, found 519.0865; ^1H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.99 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.82 (dt, *J* = 1.2, 8.0 Hz, 2H), 7.70-7.67 (m, 2H), 7.67 (s, 1H), 7.64-7.49 (m, 4H), 7.41-7.33

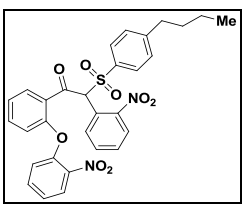
(m, 4H), 7.15 (dd, *J* = 0.8, 7.6 Hz, 1H), 7.06 (dd, *J* = 1.2, 8.0 Hz, 1H), 6.61 (dd, *J* = 0.8, 8.0 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.8, 155.7, 149.7, 147.9, 141.7, 137.6, 134.9, 134.8, 134.2, 133.0, 132.8, 131.4, 130.2, 129.5 (2x), 128.8 (2x), 128.6, 126.2, 125.5, 125.3, 124.0, 123.4, 122.3, 116.7, 71.3.



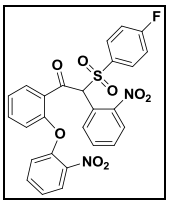
1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-(*m*-tolylsulfonyl)ethan-1-one (6l). Yield = 84% (223 mg); White solid; mp = 163-165 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{27}H_{21}N_2O_8S$ 533.1019, found 533.1024; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05-8.02 (m, 2H), 7.80 (dt, *J* = 1.2, 8.0 Hz, 2H), 7.65 (s, 1H), 7.64-7.57 (m, 2H), 7.52-7.45 (m, 3H), 7.40-7.31 (m, 3H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 7.04 (dd, *J* = 0.8, 8.4 Hz, 1H), 6.61 (d, *J* = 8.4 Hz, 1H), 2.28 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.8, 155.5, 149.7, 147.9, 141.5, 139.1, 137.4, 134.9, 134.8, 134.7, 132.9, 132.8, 131.3, 130.1, 129.6, 128.60, 128.55, 126.4, 126.2, 125.7, 125.2, 123.8, 123.2, 122.2, 116.6, 71.2, 21.0.



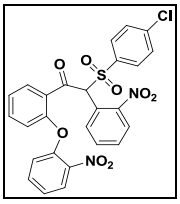
2-((4-Ethylphenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6m). Yield = 83% (227 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{28}H_{23}N_2O_8S$ 547.1175, found 547.1181; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.98 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.80 (dd, *J* = 1.6, 8.0 Hz, 2H), 7.65 (s, 1H), 7.62-7.55 (m, 4H), 7.49 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.37 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.33 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.14 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.03 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.61 (dd, *J* = 0.4, 8.0 Hz, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 1.19 (t, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.9, 155.4, 151.4, 149.7, 148.0, 141.6, 134.8, 134.7, 134.6, 132.9, 132.7, 131.2, 130.1, 129.5 (2x), 128.6, 128.2 (2x), 126.1, 125.4, 125.2, 123.9, 123.1, 122.4, 116.8, 71.3, 28.8, 14.9.



2-((4-*n*-Butylphenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6n). Yield = 86% (247 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{30}H_{27}N_2O_8S$ 575.1488, found 575.1492; ^1H NMR (400 MHz, $CDCl_3$): δ 8.04 (dd, *J* = 1.6, 8.0 Hz, 1H), 8.01 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.82 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.80 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.64 (s, 1H), 7.64-7.55 (m, 4H), 7.49 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.38 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.34 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.15 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.06 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.62 (dd, *J* = 0.4, 8.0 Hz, 1H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.57-1.53 (m, 2H), 1.33-1.25 (m, 2H), 0.90 (t, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 189.9, 155.5, 150.2, 149.7, 148.1, 141.7, 134.8, 134.7, 134.6, 132.9, 132.8, 131.3, 130.0, 129.5 (3x), 128.8 (2x), 126.2, 125.4, 125.2, 124.0, 123.2, 122.5, 116.8, 71.3, 35.5, 33.0, 22.1, 13.8.

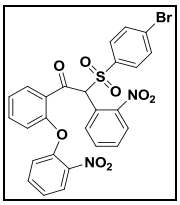


2-((4-Fluorophenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6o). Yield = 80% (214 mg); White solid; mp = 174-176 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{26}H_{18}FN_2O_8S$ 537.0768, found 537.0771; ^1H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.93 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.86-7.83 (m, 2H), 7.73-7.70 (m, 2H), 7.66 (s, 1H), 7.64-7.58 (m, 2H), 7.52 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.42-7.34 (m, 2H), 7.16 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.09-7.03 (m, 3H), 6.61 (dd, *J* = 0.8, 8.4 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 189.8, 166.1 (d, *J* = 256.2 Hz), 155.6, 149.7, 147.7, 141.6, 135.0, 134.9, 133.5 (d, *J* = 3.1 Hz), 133.0, 132.60, 132.57 (d, *J* = 9.1 Hz, 2x), 131.4, 130.3, 128.2, 126.3, 125.7, 125.4, 124.0, 123.4, 122.1, 116.6, 116.1 (d, *J* = 22.8 Hz, 2x), 71.5; $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$): δ -102.13~-102.20 (m, 1F).

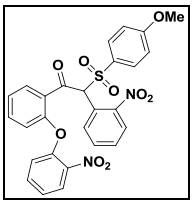


2-((4-Chlorophenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6p). Yield = 83% (229 mg); White solid; mp = 155-157 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{26}H_{18}ClN_2O_8S$ 553.0472, found 553.0475; ^1H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.92 dd, *J* = 1.6, 8.0 Hz, 1H), 7.86 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.83 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.67 (s, 1H),

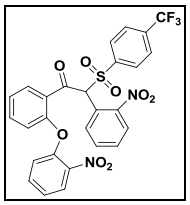
7.66-7.58 (m, 4H), 7.53 (dt, *J* = 1.6, 7.6 Hz, 1H), 7.42-7.35 (m, 4H), 7.16 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.03 (dd, *J* = 1.6, 8.4 Hz, 1H), 6.60 (dd, *J* = 1.2, 8.4 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.7, 155.7, 149.7, 147.7, 141.6, 141.0, 136.1, 135.0, 134.9, 133.1, 132.6, 131.5, 131.1 (2x), 130.4, 129.1 (2x), 128.2, 126.3, 125.7, 125.5, 124.0, 123.4, 122.0, 116.5, 71.5.



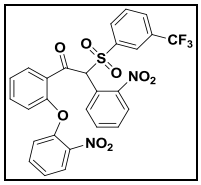
2-((4-Bromophenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6q). Yield = 82% (244 mg); White solid; mp = 135-137 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{26}H_{18}BrN_2O_8S$ 596.9967, found 596.9974; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.91 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.87 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.83 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.67 (s, 1H), 7.65-7.51 (m, 7H), 7.42-7.35 (m, 2H), 7.17 (dt, *J* = 0.8, 8.0 Hz, 1H), 7.04 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.60 (d, *J* = 8.4 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.7, 155.7, 149.7, 147.7, 141.6, 136.6, 135.0, 134.9, 133.1, 132.6, 132.1 (2x), 131.5, 131.1 (2x), 130.4, 129.8, 128.2, 126.3, 125.7, 125.5, 124.0, 123.4, 122.0, 116.5, 71.5.



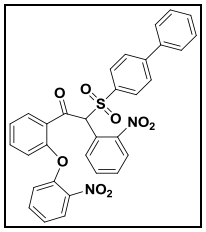
2-((4-Methoxyphenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6r). Yield = 86% (236 mg); White solid; mp = 197-199 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{27}H_{21}N_2O_9S$ 549.0968, found 549.0975; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 1.6, 8.4 Hz, 1H), 7.94 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.82 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.81 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.61 (s, 1H), 7.60-7.56 (m, 4H), 7.49 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.38 (dt, *J* = 1.6, 7.6 Hz, 1H), 7.33 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.14 (dt, *J* = 0.4, 7.6 Hz, 1H), 7.04 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.0 Hz, 1H), 3.79 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 190.0, 164.0, 155.4, 149.6, 148.0, 141.5, 134.8, 134.7, 132.8, 132.5, 131.7 (2x), 131.2, 130.0, 128.7, 128.6, 126.1, 125.4, 125.2, 123.9, 123.1, 122.5, 116.8, 113.9 (2x), 71.3, 55.6.



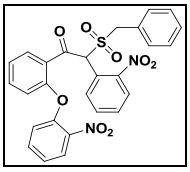
1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-((4-(trifluoromethyl)phenyl)sulfonyl)ethan-1-one (6s). Yield = 81% (237 mg); White solid; mp = 154-156 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{18}F_3N_2O_8S$ 587.0736, found 587.0741; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.92-7.87 (m, 4H), 7.82 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.73 (s, 1H), 7.68-7.53 (m, 5H), 7.42-7.36 (m, 2H), 7.16 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.02 (dd, *J* = 1.2, 8.0 Hz, 1H), 6.58 (dd, *J* = 1.2, 8.4 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.6, 155.7, 149.7, 147.6, 141.7, 141.3, 135.8, 135.6 (q, *J* = 32.8 Hz), 135.1, 135.0, 133.1, 132.7, 131.5, 130.5, 130.3 (2x), 127.9, 126.4, 125.9 (q, *J* = 3.8 Hz, 2x), 125.8, 124.0, 123.5, 123.0 (q, *J* = 263.0 Hz), 121.7,116.4, 71.7; ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ -63.15 (s, 3F).



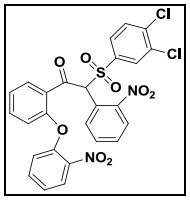
1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-((3-(trifluoromethyl)phenyl)sulfonyl)ethan-1-one (6t). Yield = 82% (240 mg); White solid; mp = 163-165 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{18}F_3N_2O_8S$ 587.0736, found 587.0741; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.4 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.90 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.87-7.84 (m, 2H), 7.82-7.79 (m, 2H), 7.72 (s, 1H), 7.65-7.58 (m, 3H), 7.54 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.42-7.36 (m, 2H), 7.17 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.08 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.59 (dd, *J* = 1.2, 8.4 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.5, 156.8, 149.7, 147.6, 141.6, 138.7, 135.1, 135.0, 133.11, 133.06, 132.6, 131.6, 131.4, 131.1, 130.7 (q, *J* = 3.4 Hz), 129.7, 127.9, 126.8 (q, *J* = 3.8 Hz), 126.4, 125.8, 125.5, 124.0, 123.9 (q, *J* = 271.1 Hz), 123.6, 121.9, 116.3, 71.7; ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ -62.78 (s, 3F). Single-crystal X-Ray diagram: crystal of compound **6t** was grown by slow diffusion of EtOAc into a solution of compound **6t** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the triclinic crystal system, space group P-1, *a* = 8.30070(10) Å, *b* = 9.92720(10) Å, *c* = 16.4260(2) Å, *V* = 1245.10(3) Å^3, *Z* = 5, d_{calcd} = 1.564 g/cm^3, *F*(000) = 600.0, 2θ range 4.46~54.254^{o}, R indices (all data) R1 = 0.0564, wR2 = 0.1309.



2-([1,1'-Biphenyl]-4-ylsulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6u). Yield = 84% (250 mg); White solid; mp = 210-212 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{32}H_{23}N_2O_8S$ 595.1175, found 595.1178; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (dt, *J* = 1.6, 8.0 Hz, 2H), 7.86-7.83 (m, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.72 (s, 1H), 7.65-7.31 (m, 12H), 7.16 (dt, *J* = 0.8, 7.6 Hz, 1H), 7.05 (dd, *J* = 1.2, 8.0 Hz, 1H), 6.62 (d, *J* = 8.4 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.8, 155.5, 149.7, 148.0, 147.0, 141.6, 138.9, 136.0, 134.82, 134.76, 133.0, 132.8, 131.4, 130.2, 130.0 (2x), 129.0 (2x), 128.64, 128.62, 127.4 (2x), 127.3 (2x), 126.2, 125.4, 125.3, 124.0, 123.2, 122.3, 116.8, 71.5.

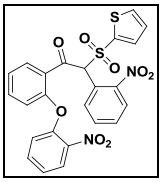


2-(Benzylsulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6v). Yield = 80% (213 mg); White solid; mp = 166-168 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{27}H_{21}N_2O_8S$ 533.1019, found 533.1023; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (dt, *J* = 1.6, 8.0 Hz, 1H), 7.93 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.85 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.70 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.61-7.49 (m, 3H), 7.42-7.29 (m, 7H), 7.25 (s, 1H), 7.17 (dt, *J* = 0.8, 8.0 Hz, 1H), 6.82 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.56 (dd, *J* = 0.4, 8.4 Hz, 1H), 4.73 (d, *J* = 13.2 Hz, 1H), 4.45 (d, *J* = 13.6 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 190.8, 155.5, 149.6, 147.6, 141.8, 135.0, 134.8, 133.2, 132.9, 131.3, 130.9 (2x), 130.4, 129.2, 128.9 (2x), 127.5, 126.6, 126.2, 125.8, 125.6, 123.9, 123.2, 121.6, 116.4, 68.9, 60.4.

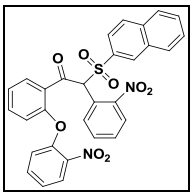


2-((3,4-Dichlorophenyl)sulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6w). Yield = 82% (240 mg); White solid; mp = 185-187 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{26}H_{17}Cl_2N_2O_8S$ 587.0083, found 587.0089; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.89 (dt, *J* = 1.6, 8.0 Hz, 2H), 7.84 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.73 (d, *J* = 2.0 Hz, 1H), 7.71 (s, 1H),

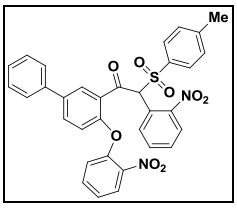
7.66-7.54 (m, 4H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.42-7.36 (m, 2H), 7.16 (dt, *J* = 0.8, 8.4 Hz, 1H), 7.04 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.58 (dd, *J* = 0.4, 8.4 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.5, 155.7, 149.7, 147.4, 141.5, 139.3, 137.3, 135.2, 135.0, 133.3, 133.1, 132.5, 131.53, 131.47, 130.8, 130.6, 128.7, 127.7, 126.4, 125.9, 125.6, 123.9, 123.5, 121.7, 116.2, 71.8.



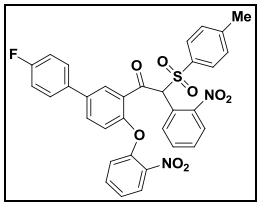
1-(2-(2-Nitrophenoxy)phenyl)-2-(2-nitrophenyl)-2-(thiophen-2-ylsulfonyl)ethan-1-one (6x). Yield = 84% (220 mg); White solid; mp = 175-177 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{24}H_{17}N_2O_8S_2$ 525.0426, found 525.0432; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.97 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.88 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.84 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.73 (s, 1H), 7.64-7.59 (m, 3H), 7.54-7.49 (m, 2H), 7.42 (ddt, *J* = 1.6, 3.6, 8.4 Hz, 1H), 7.36 (ddt, *J* = 1.6, 3.6, 8.4 Hz, 1H), 7.18 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.13 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.01 (dd, *J* = 3.6, 4.8 Hz, 1H), 6.65 (d, *J* = 8.4 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.7, 155.7, 149.7, 148.0, 141.7, 137.9, 136.5, 135.7, 134.93, 134.90, 133.0, 132.6, 131.5, 130.3, 128.4, 127.7, 126.2, 125.5, 125.4, 124.0, 123.4, 122.4, 116.8, 72.2. Single-crystal X-Ray diagram: crystal of compound **6x** was grown by slow diffusion of EtOAc into a solution of compound **6x** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the triclinic crystal system, space group P-1, *a* = 8.3670(2) Å, *b* = 9.8898(2) Å, *c* = 14.7637(2) Å, *V* = 1115.17(4) $Å^3$, *Z* = 2, d_{calcd} = 1.562 g/cm^3, *F*(000) = 540.0, 2*θ* range 4.482~49.998°, R indices (all data) R1 = 0.0387, wR2 = 0.0937.



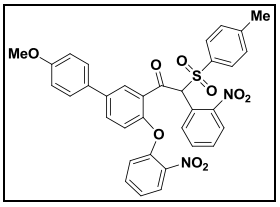
2-(Naphthalen-2-ylsulfonyl)-1-(2-(2-nitrophenoxy)phenyl)-2-(2-nitrophenyl)ethan-1-one (6y). Yield = 80% (227 mg); White solid; mp = 211-213 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{30}H_{21}N_2O_8S$ 569.1019, found 569.1014; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (d, *J* = 1.6 Hz, 1H), 8.04-8.01 (m, 2H), 7.86-7.78 (m, 5H), 7.76 (s, 1H), 7.68 (dd, *J* = 1.6, 8.4 Hz, 1H), 7.65-7.49 (m, 5H), 7.35-7.30 (m, 2H), 7.10 (dt, *J* = 0.8, 7.6 Hz, 1H), 7.01 (dd, *J* = 1.2, 8.4 Hz, 1H), 6.55 (dd, *J* = 0.4, 8.0 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 189.9, 155.5, 149.8, 147.9, 141.6, 135.4, 134.8, 134.7, 134.6, 133.0, 132.8, 131.9, 131.8, 131.4, 130.2, 129.5, 129.4, 128.9, 128.6, 127.8, 127.5, 126.2, 125.4, 125.3, 123.9, 123.7, 123.3, 122.3, 116.6, 71.5.



1-(4-(2-Nitrophenoxy)-[1,1'-biphenyl]-3-yl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6ab). Yield = 83% (252 mg); White solid; mp = 140-142 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{33}H_{25}N_2O_8S$ 609.1332, found 609.1335; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (dd, *J* = 1.6, 8.0 Hz, 1H), 8.02 (dd, *J* = 1.2, 8.0 Hz, 1H), 8.00 (d, *J* = 2.4 Hz, 1H), 7.84 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.69 (s, 1H), 7.66-7.34 (m, 12H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.12 (dd, *J* = 0.8, 8.4 Hz, 1H), 6.69 (d, *J* = 8.4 Hz, 1H), 2.34 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.9, 154.8, 149.7, 148.0, 145.4, 141.6, 138.7, 137.1, 134.9, 134.6, 133.03, 132.98, 132.7, 130.1, 129.7, 129.5 (2x), 129.4 (2x), 128.9 (2x), 128.8, 127.8, 126.8 (2x), 126.2, 125.5, 125.3, 123.3, 122.3, 117.2, 71.3, 21.6.

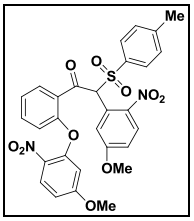


1-(4'-Fluoro-4-(2-nitrophenoxy)-[1,1'-biphenyl]-3-yl)-2-(2-nitrophenyl)-2-tosylethan-1-one (6ac). Yield = 90% (282 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for $C_{33}H_{24}FN_2O_8S$ 627.1238, found 627.1241; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (dd, *J* = 1.6, 8.0 Hz, 1H), 8.02 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.96 (d, *J* = 2.4 Hz, 1H), 7.83 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.69 (s, 1H), 7.65-7.47 (m, 8H), 7.37 (dt, *J* = 1.2, 8.0 Hz, 1H), 7.18-7.10 (m, 5H), 6.69 (d, *J* = 8.8 Hz, 1H), 2.34 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.9, 162.7 (d, *J* = 245.6 Hz), 154.9, 149.7, 147.9, 145.4 (2x), 141.7, 136.1, 134.92, 134.86 (d, *J* = 3.1 Hz), 134.5, 132.9 (d, *J* = 8.3 Hz, 2x), 132.7, 130.1, 129.5 (2x), 129.4 (2x), 128.8, 128.5, 128.4, 126.2, 125.6, 125.3, 123.4, 122.4, 117.2, 115.9 (d, *J* = 21.3 Hz, 2x), 71.3, 21.6; ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$): δ -114.39~-114.46 (m, 1F).

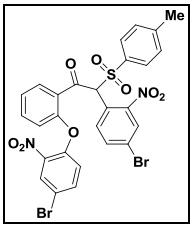


1-(4'-Methoxy-4-(2-nitrophenoxy)-[1,1'-biphenyl]-3-yl)-2-(2-nitrophenyl)-2-tosylethan -1-one (6ad). Yield = 90% (287 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: [M + H]$^{+}$ calcd for

$C_{34}H_{27}N_2O_9S$ 639.1437, found 639.1445; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.99 (dd, *J* = 0.8, 7.6 Hz, 1H), 7.95 (d, *J* = 2.4 Hz, 1H), 7.83 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.69 (s, 1H), 7.64-7.44 (m, 8H), 7.34 (dt, *J* = 1.2, 7.6 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 1H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.67 (d, *J* = 8.8 Hz, 1H), 3.83 (s, 3H), 2.33 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.9, 159.4, 154.2, 149.7, 148.1, 145.4, 141.5, 136.7, 134.8, 134.5, 132.9, 132.6, 132.5, 131.0, 130.1, 129.5 (2x), 129.4 (2x), 129.0, 128.7, 127.8 (2x), 126.1, 125.32, 125.27, 123.0, 122.3, 117.4, 114.3 (2x), 71.2, 55.3, 21.5.

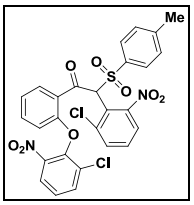


1-(2-(5-Methoxy-2-nitrophenoxy)phenyl)-2-(5-methoxy-2-nitrophenyl)-2-tosylethan-1-one (6ae). Yield = 78% (231 mg); White solid; mp = 206-208 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^+$ calcd for $C_{29}H_{25}N_2O_{10}S$ 593.1230, found 593.1233; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, *J* = 9.2 Hz, 1H), 7.92 (d, *J* = 9.2 Hz, 1H), 7.88 (s, 1H), 7.80 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 2.8 Hz, 1H), 7.38 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.14 (dt, *J* = 1.6, 8.0 Hz, 1H), 6.92 (dt, *J* = 2.8, 8.0 Hz, 1H), 6.81 (dd, *J* = 2.8, 7.6 Hz, 1H), 6.62 (dd, *J* = 0.8, 8.4 Hz, 1H), 6.54 (d, *J* = 2.8 Hz, 1H), 3.87 (s, 3H), 3.84 (s, 3H), 2.37 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 190.2, 164.8, 162.7, 155.5, 150.1, 145.3, 142.4, 134.8, 134.6, 131.4, 129.5 (2x), 129.4 (2x), 128.6, 128.4, 127.9, 125.2, 123.8, 117.1, 116.4, 115.5, 114.0, 111.2, 108.3, 70.9, 56.2, 56.1, 21.6.

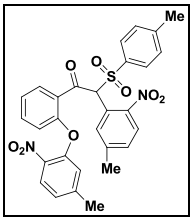


1-(2-(4-Bromo-2-nitrophenoxy)phenyl)-2-(4-bromo-2-nitrophenyl)-2-tosylethan-1-one (6af). Yield = 78% (268 mg); White solid; mp = 215-217 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: [M + H]$^+$ calcd for $C_{27}H_{19}Br_2N_2O_8S$ 688.9229, found 688.9234; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (d, *J* = 2.4 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 2.0 Hz, 1H), 7.81 (dd, *J* = 2.0, 8.0 Hz, 1H), 7.76 (dd, *J* = 2.0, 8.4 Hz, 1H), 7.70 (dd, *J* = 2.4, 8.8 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.49 (s, 1H), 7.46-7.42 (m, 1H), 7.23-7.18 (m, 3H), 6.98 (d, *J* = 8.8 Hz, 1H), 6.66 (dd, *J* = 0.8, 8.4 Hz, 1H), 2.38 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.3, 155.0, 150.0, 147.4, 145.7, 141.9, 137.8, 136.0,

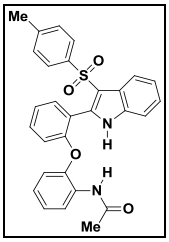
134.9, 134.23, 134.16, 131.5, 129.6 (2x), 129.4 (2x), 129.1, 128.4, 128.1, 124.54, 124.51, 124.0, 121.5, 117.7, 117.0, 70.6, 21.7.



1-(2-(2-Chloro-6-nitrophenoxy)phenyl)-2-(2-chloro-6-nitrophenyl)-2-tosylethan-1-one (6ag). Rotamer; Yield = 70% (210 mg); White solid; mp = 178-180 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{27}H_{19}Cl_2N_2O_8S$ 601.0239, found 601.0245; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, *J* = 8.4 Hz, 2H), 7.99-7.67 (m, 5H), 7.57 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.50 (br t, *J* = 8.0 Hz, 1H), 7.40-7.23 (m, 4H), 7.14-7.09 (m, 1H), 6.25 (br d, *J* = 8.4 Hz, 1H), 2.43 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 189.3, 162.0, 154.1, 144.9, 136.8, 136.0, 135.3, 134.4, 134.1, 133.3, 132.9, 130.7, 129.5 (4x), 127.7, 126.7, 126.8, 125.2, 125.1, 124.0, 123.7, 122.4, 114.3, 73.9, 21.7. Single-crystal X-Ray diagram: crystal of compound **6ag** was grown by slow diffusion of EtOAc into a solution of compound **6ag** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the triclinic crystal system, space group P-1, *a* = 9.5100(2) Å, *b* = 10.9393(3) Å, *c* = 14.4361(2) Å, *V* = 1466.83(6) Å^3, *Z* = 6, d_{calcd} = 1.362 g/cm^3, *F*(000) = 616.0, 2*θ* range 4.33~49.998^{o}, R indices (all data) R1 = 0.0443, wR2 = 0.1076.

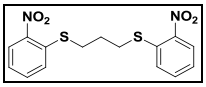


1-(2-(5-Methyl-2-nitrophenoxy)phenyl)-2-(5-methyl-2-nitrophenyl)-2-tosylethan-1-one (6ah). Yield = 73% (204 mg); White solid; mp = 197-199 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{29}H_{25}N_2O_8S$ 561.1332, found 561.1337; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.4 Hz, 1H), 7.80 (dd, *J* = 1.2, 8.4 Hz, 1H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.70 (s, 1H), 7.69 (d, *J* = 1.2 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.39-7.34 (m, 1H), 7.28-7.26 (m, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.13 (dt, *J* = 0.8, 7.6 Hz, 2H), 6.82 (br d, *J* = 0.8 Hz, 1H), 6.59 (d, *J* = 8.4 Hz, 1H), 2.40 (s, 3H), 2.38 (s, 3H), 2.36 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 190.1, 155.6, 147.9, 147.4, 146.8, 145.2, 144.5, 139.2, 134.8, 134.6, 133.1, 131.2, 130.5, 129.5 (2x), 129.3 (2x), 128.5, 126.1 (2x), 125.5, 123.7, 123.6, 122.3, 116.6, 71.2, 21.6, 21.5 (2x).



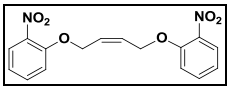
***N*-(2-(2-(3-Tosyl-1*H*-indol-2-yl)phenoxy)phenyl)acetamide (8a).** Pd/C (10%, 10 mg) was added to a solution of **6a** (160 mg, 0.3 mmol) in acetic acid (10 mL) at 25 ^{o}C Hydrogen gas was installed to the reaction mixture at 25 ^{o}C. The reaction mixture was stirred at 25 ^{o}C for 20 h. The reaction mixture was filtrated and the solvent was concentrated to afford the crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 10/1~2/1) afforded **8a**. Yield = 90% (134 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{29}H_{25}N_2O_4S$ 497.1535, found 497.1538; ^{1}H NMR (400 MHz, $CDCl_3$): δ 11.00 (br s, 1H), 8.95 (s, 1H), 8.18-8.16 (m, 1H), 7.92 (dd, *J* = 1.2, 8.4 Hz, 1H), 7.48-7.45 (m, 1H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.30-7.25 (m, 3H), 7.19 (dd, *J* = 1.2, 7.6 Hz, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.82-6.74 (m, 2H), 6.69 (dd, *J* = 1.2, 7.6 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 1H), 2.31 (s, 3H), 1.90 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 171.3, 156.1, 143.1, 142.4, 140.6, 136.7, 133.2, 132.8, 131.4, 130.5, 129.3 (2x), 126.2 (2x), 125.2, 124.2, 123.6, 122.6, 121.6, 121.4, 120.9, 120.7, 119.6, 116.9, 113.1, 109.4, 108.2, 23.9, 21.4.

A representative synthetic procedure of skeleton 9 is as follows: Trifluoroacetic anhydride (TFAA, 210 mg, 1.0 mmol) was added to a solution of **5a** (155 mg, 0.5 mmol) in MeCN (10 mL) at 25 ^{o}C. The reaction mixture was stirred at 25 ^{o}C for 10 min. K_2CO_3 (140 mg, 1.0 mmol) and 1,3-propanedithiol (55 mg, 0.5 mmol, for **9a**), *cis*-2-butene-1,4-diol (45 mg, 0.5 mmol, for **9b**) or water (9 mg, 0.5 mmol, for **9c**) was added to the reaction mixture at 25 ^{o}C in onestep. The reaction mixture was stirred at 25 ^{o}C for 20 h. Then, the solvent was concentrated to afford the crude products. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 8/1~4/1) afforded **9**.

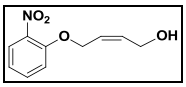


1,3-Bis((2-nitrophenyl)thio)propane (9a). Yield = 80% (140 mg); Yellowish solid; mp = 140-142 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{15}H_{15}N_2O_4S_2$ 351.0473, found 351.0478; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (dd, *J* = 1.6, 8.4 Hz, 2H), 7.55 (dt, *J* = 1.6, 8.0 Hz, 2H), 7.41 (dd, *J* = 0.8, 8.0 Hz, 2H), 7.27 (dt, *J* = 1.2, 8.0 Hz, 2H), 3.17 (t, *J* = 7.2 Hz, 4H), 2.21-2.13 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz,

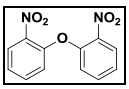
$CDCl_3$): δ 146.3 (2x), 136.7 (2x), 133.6 (2x), 126.6 (2x), 126.2 (2x), 124.8 (2x), 31.0 (2x), 26.0.



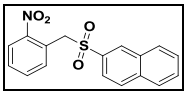
(*Z*)-1,4-Bis(2-nitrophenoxy)but-2-ene (9b). Yield = 83% (137 mg); White solid; mp = 132-134 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{16}H_{15}N_2O_6$ 331.0930, found 331.0933; ^1H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, *J* = 1.6, 8.0 Hz, 2H), 7.54 (dt, *J* = 1.6, 8.4 Hz, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.04 (dt, *J* = 1.2, 8.0 Hz, 2H), 5.98 (dt, *J* = 2.8, 3.6 Hz, 2H), 4.85 (d, *J* = 4.0 Hz, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 151.5 (2x), 140.0 (2x), 134.2 (2x), 127.9 (2x), 125.6 (2x), 120.7 (2x), 114.7 (2x), 65.9 (2x).



(*Z*)-4-(2-Nitrophenoxy)but-2-en-1-ol (9b'). Yield = 10% (11 mg); Viscous oil; HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{10}H_{12}NO_4$ 210.0766, found 210.0769; ^1H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.51 (dt, *J* = 1.6, 8.4 Hz, 1H), 7.08 (dd, *J* = 0.8, 8.4 Hz, 1H), 7.02 (dt, *J* = 0.8, 8.0 Hz, 1H), 5.93-5.87 (m, 1H), 5.85-5.79 (m, 1H), 4.78 (dd, *J* = 0.8, 6.8 Hz, 2H), 4.28 (dd, *J* = 1.2, 6.4 Hz, 2H), 2.08 (br s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 151.7, 134.1, 133.6, 127.8, 125.7, 125.5, 120.6, 114.8, 65.3, 58.9.



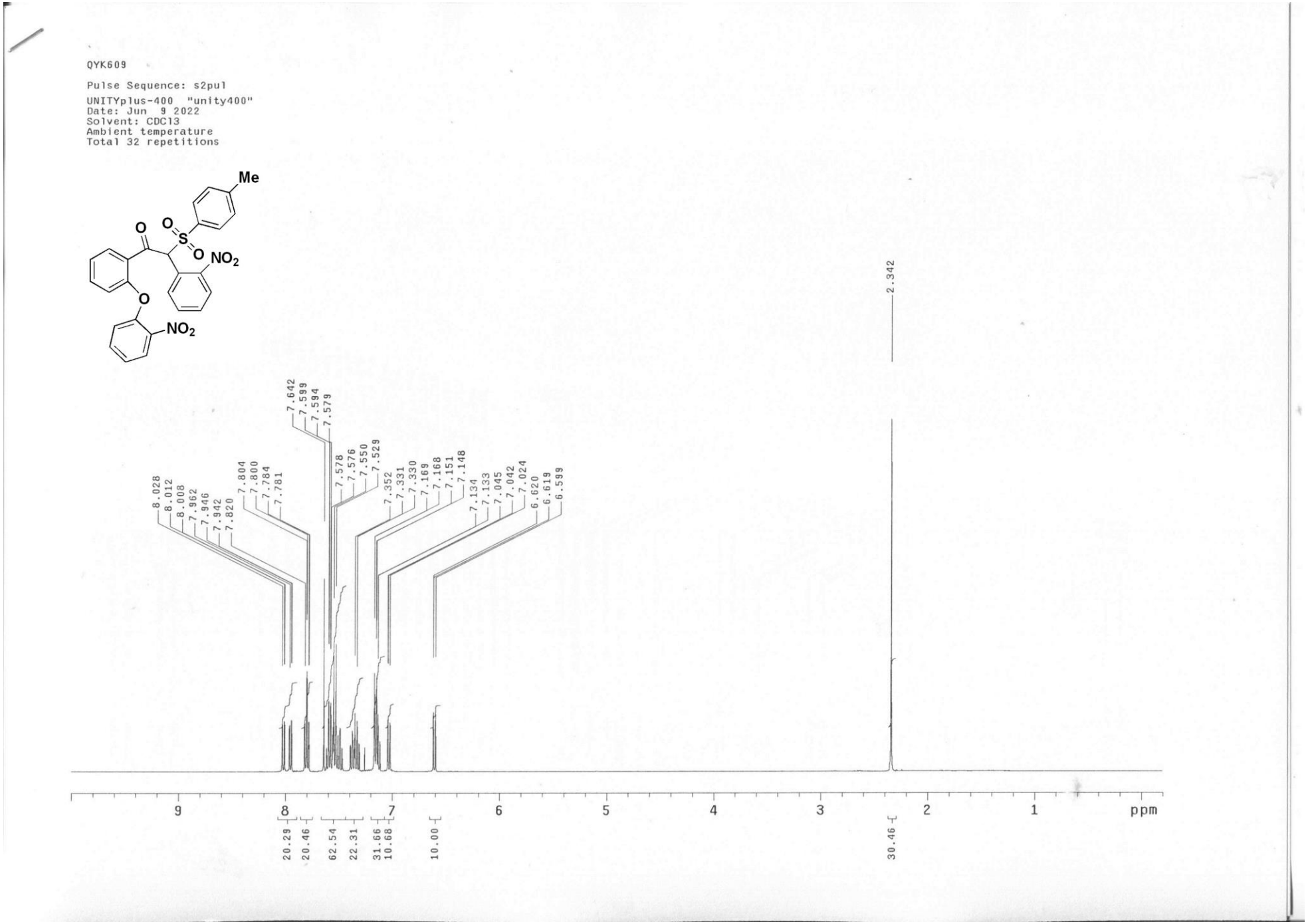
2,2'-Oxybis(nitrobenzene) (9c). Yield = 85% (111 mg); White solid; mp = 113-115 °C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{12}H_9N_2O_5$ 261.0512, found 261.0516; ^1H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 1.6, 8.0 Hz, 2H), 7.58 (dt, *J* = 1.6, 8.4 Hz, 2H), 7.31 (dt, *J* = 0.8, 8.4 Hz, 2H), 7.05 (dd, *J* = 1.2, 8.4 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 149.1 (2x), 141.1 (2x), 134.7 (2x), 126.1 (2x), 124.7 (2x), 120.9 (2x). Single-crystal X-Ray diagram: crystal of compound **9c** was grown by slow diffusion of EtOAc into a solution of compound **9c** in CH_2Cl_2 to yield colorless prisms. The compound crystallizes in the monoclinic crystal system, space group Cc, *a* = 7.16580(10) Å, *b* = 20.4831(4) Å, *c* = 7.7993(2) Å, *V* = 1143.55(4) $Å^3$, *Z* = 4, d_{calcd} = 1.511 g/cm^3, *F*(000) = 536.0, 2*θ* range 3.976~49.968°, R indices (all data) R1 = 0.0412, wR2 = 0.1130.



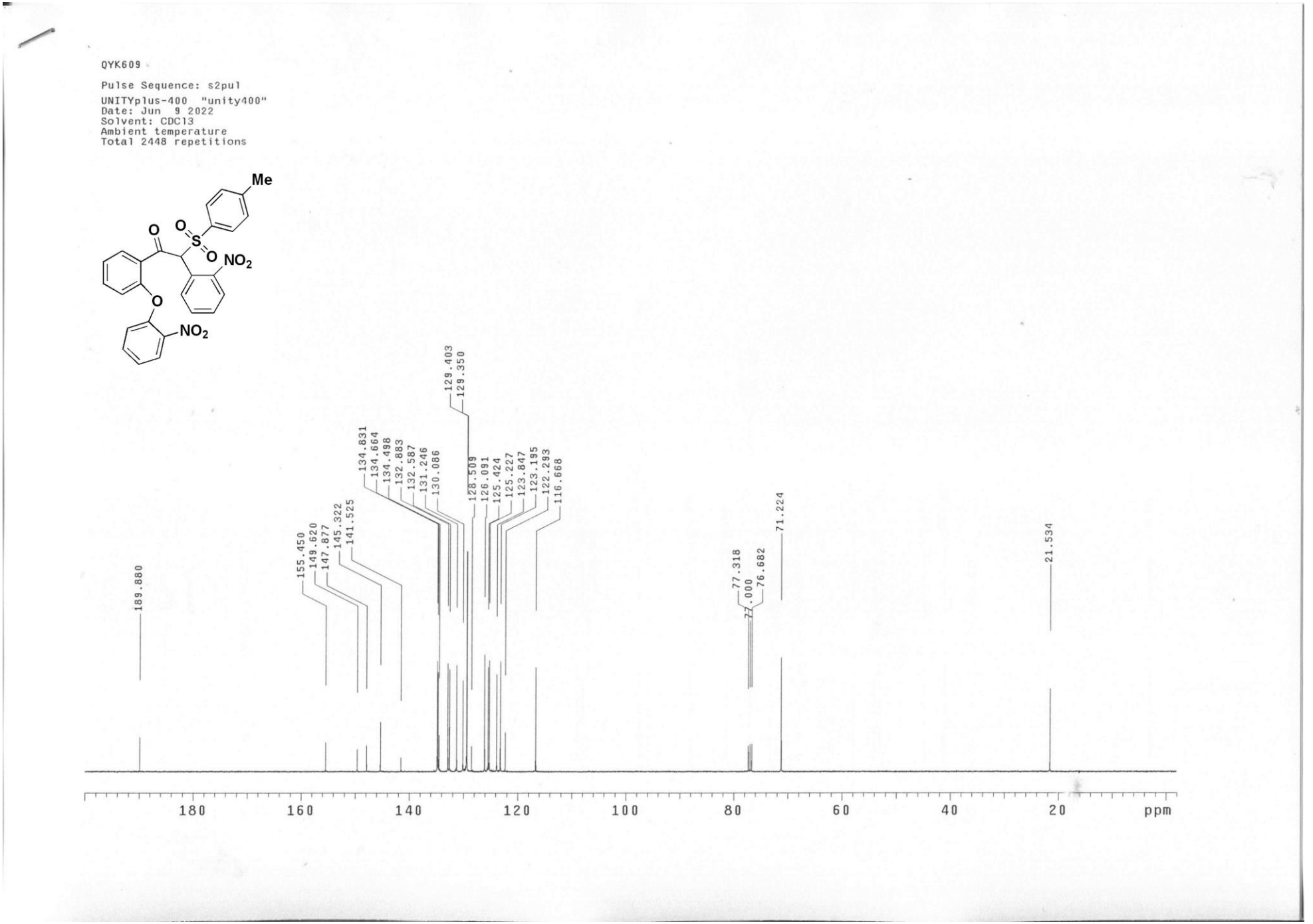
2-((2-Nitrobenzyl)sulfonyl)naphthalene (11). Trifluoroacetic anhydride (TFAA, 210 mg, 1.0 mmol) was added to a solution of **5a** (155 mg, 0.5 mmol) in MeCN (10 mL) at 25 ^{o}C. The reaction mixture was stirred at 25 ^{o}C for 10 min. K_2CO_3 (140 mg, 1.0 mmol) and compound **10** (264 mg, 1.0 mmol) was added to the reaction mixture at 25 ^{o}C in onestep. The reaction mixture was stirred at 25 ^{o}C for 20 h. Then, the solvent was concentrated to afford the crude products. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 8/1~4/1) afforded **11**. Yield = 43% (140 mg); White solid; mp = 138-140 ^{o}C (recrystallized from hexanes and EtOAc); HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ calcd for $C_{17}H_{14}NO_4S$ 328.0644, found 328.0648; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J* = 1.6 Hz, 1H), 7.95-7.89 (m, 4H), 7.71-7.66 (m, 2H), 7.64-7.57 (m, 2H), 7.53 (dt, *J* = 1.6, 7.6 Hz, 1H), 7.46 (dd, *J* = 1.6, 7.6 Hz, 1H), 5.01 (s, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 149.4, 135.4, 134.8, 134.3, 133.3, 132.0, 130.5, 130.1, 129.6, 129.5, 129.4, 128.0, 127.8, 125.5, 123.0, 122.7, 58.6.

Gram-scale synthesis of compound 6a. Trifluoroacetic anhydride (TFAA, 1.26 g, 6.0 mmol) was added to a solution of **5a** (920 mg, 3.0 mmol) in MeCN (60 mL) at 25 ^{o}C. The reaction mixture was stirred at 25 ^{o}C for 10 min. K_2CO_3 (830 mg, 6.0 mmol) and **4a** (870 mg, 3.0 mmol) were added to the reaction mixture at 25 ^{o}C in onestep. The reaction mixture was stirred at 25 ^{o}C for 20 h. Then, the solvent was concentrated to afford the crude products. The residue was diluted with water (10 mL) and the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product under reduced pressure. Purification on silica gel (hexanes/EtOAc = 8/1~4/1) afforded **6a** (1.2 g, 75%).

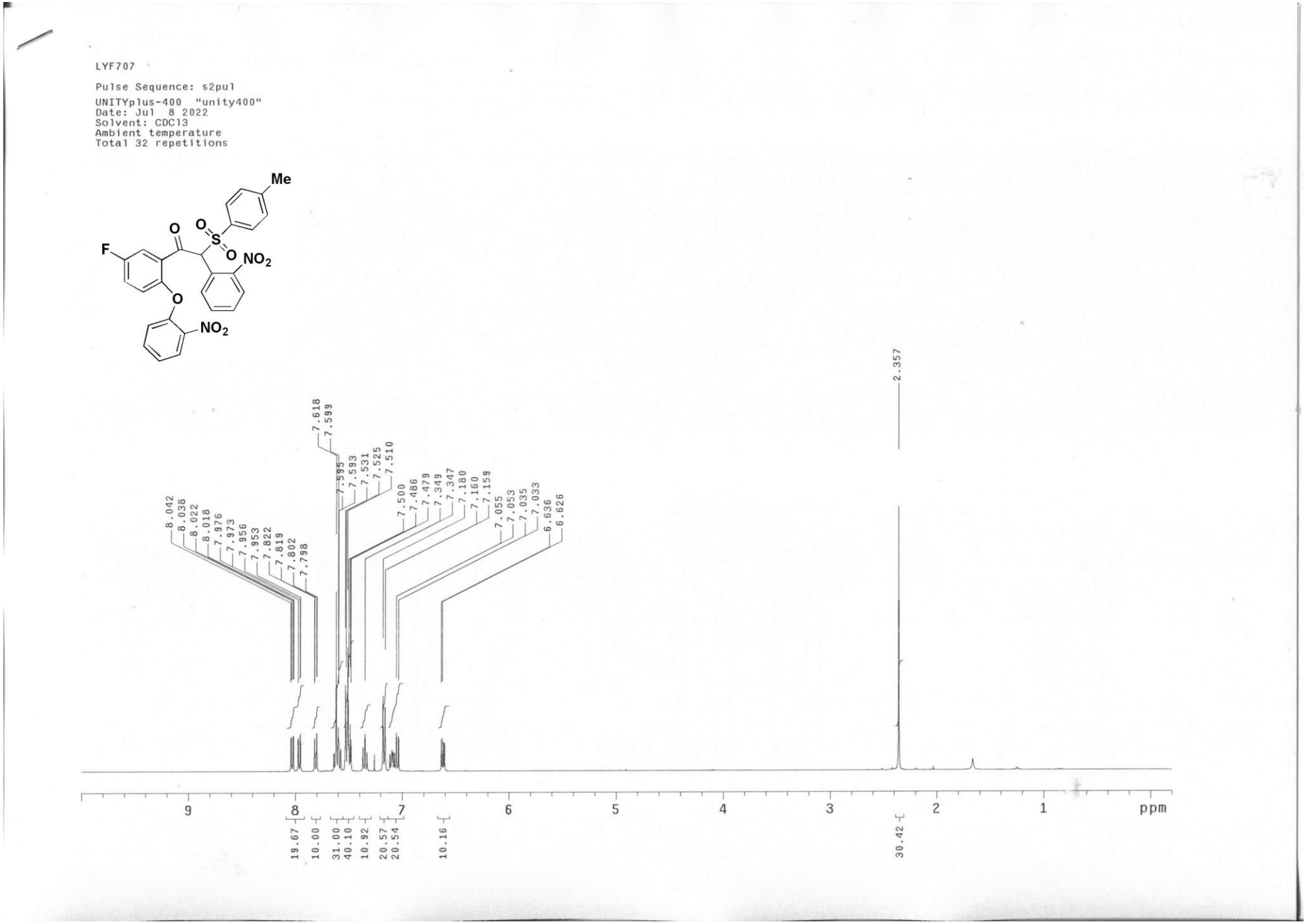
Compound 6a (^{1}H-NMR spectral data)



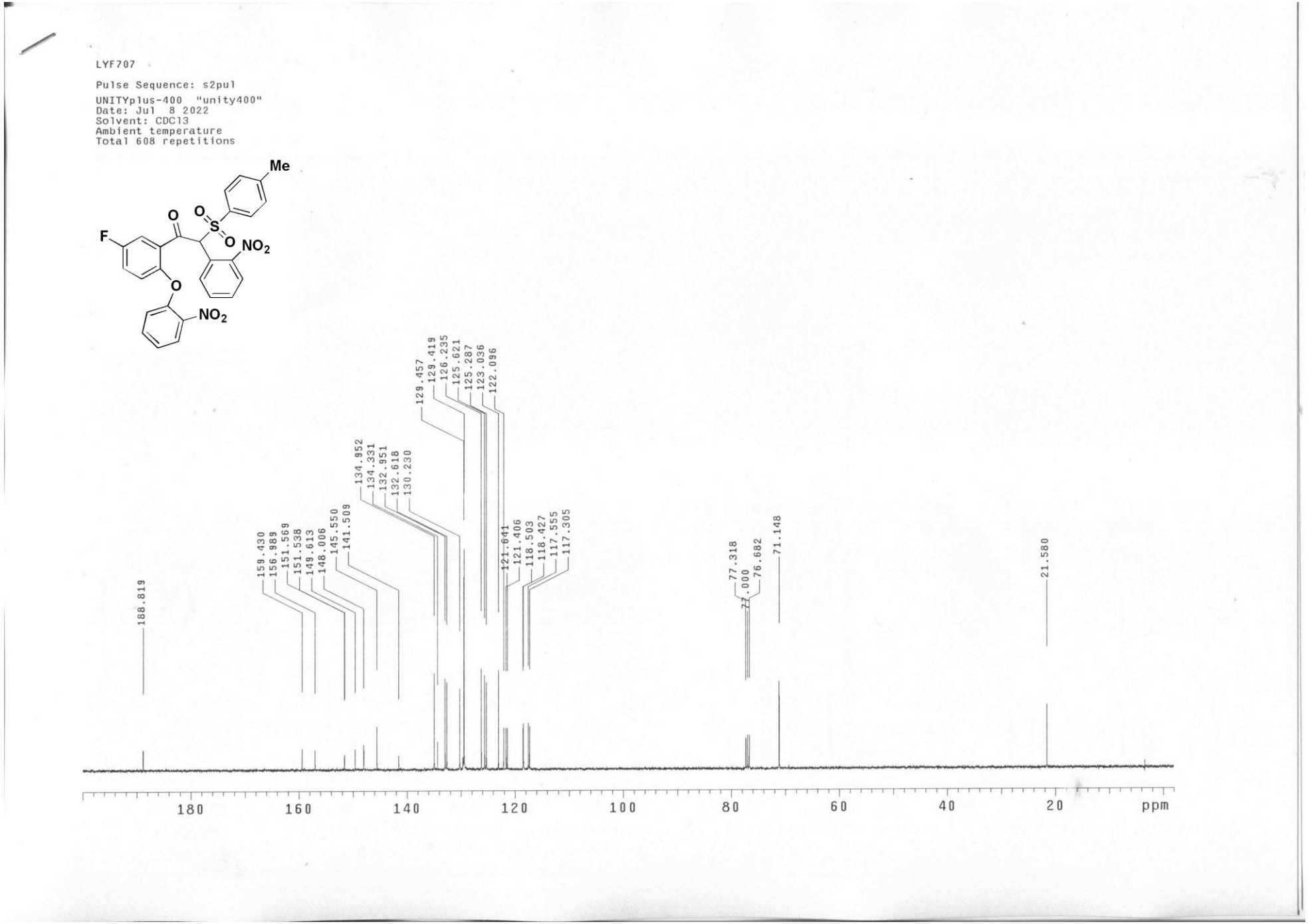
Compound 6a (^{13}C-NMR spectral data)



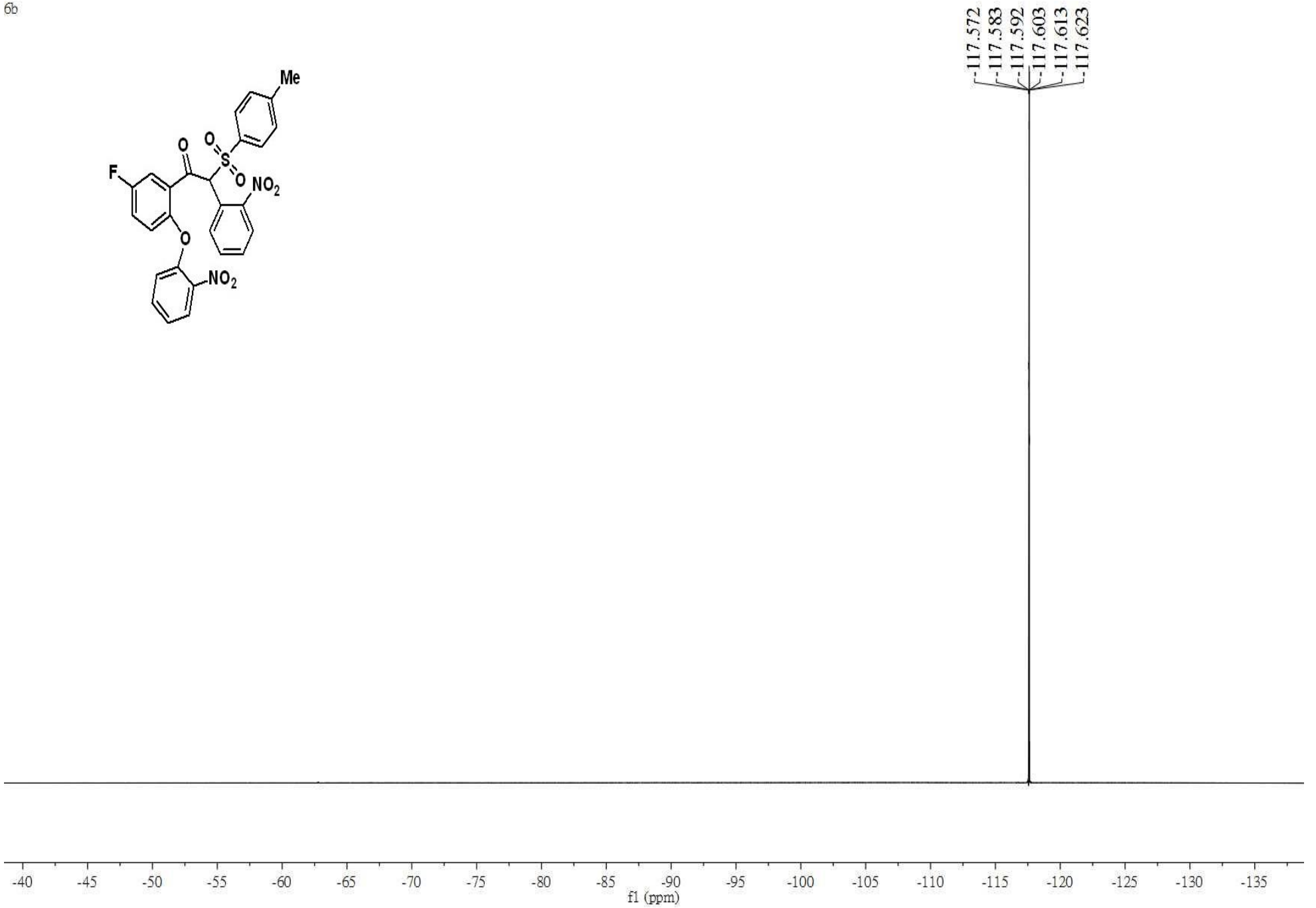
Compound 6b (^{1}H-NMR spectral data)



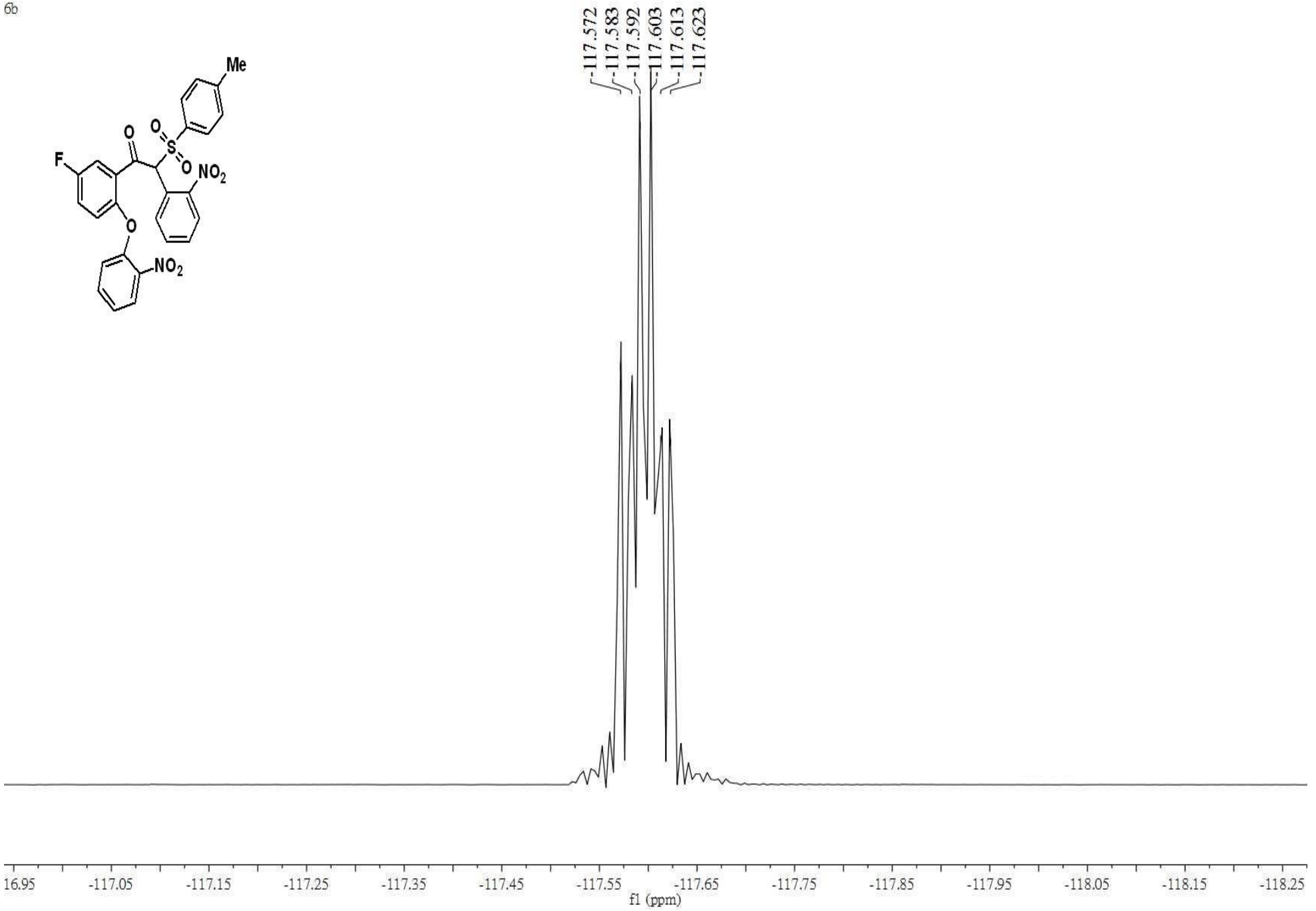
Compound 6b (^{13}C-NMR spectral data)



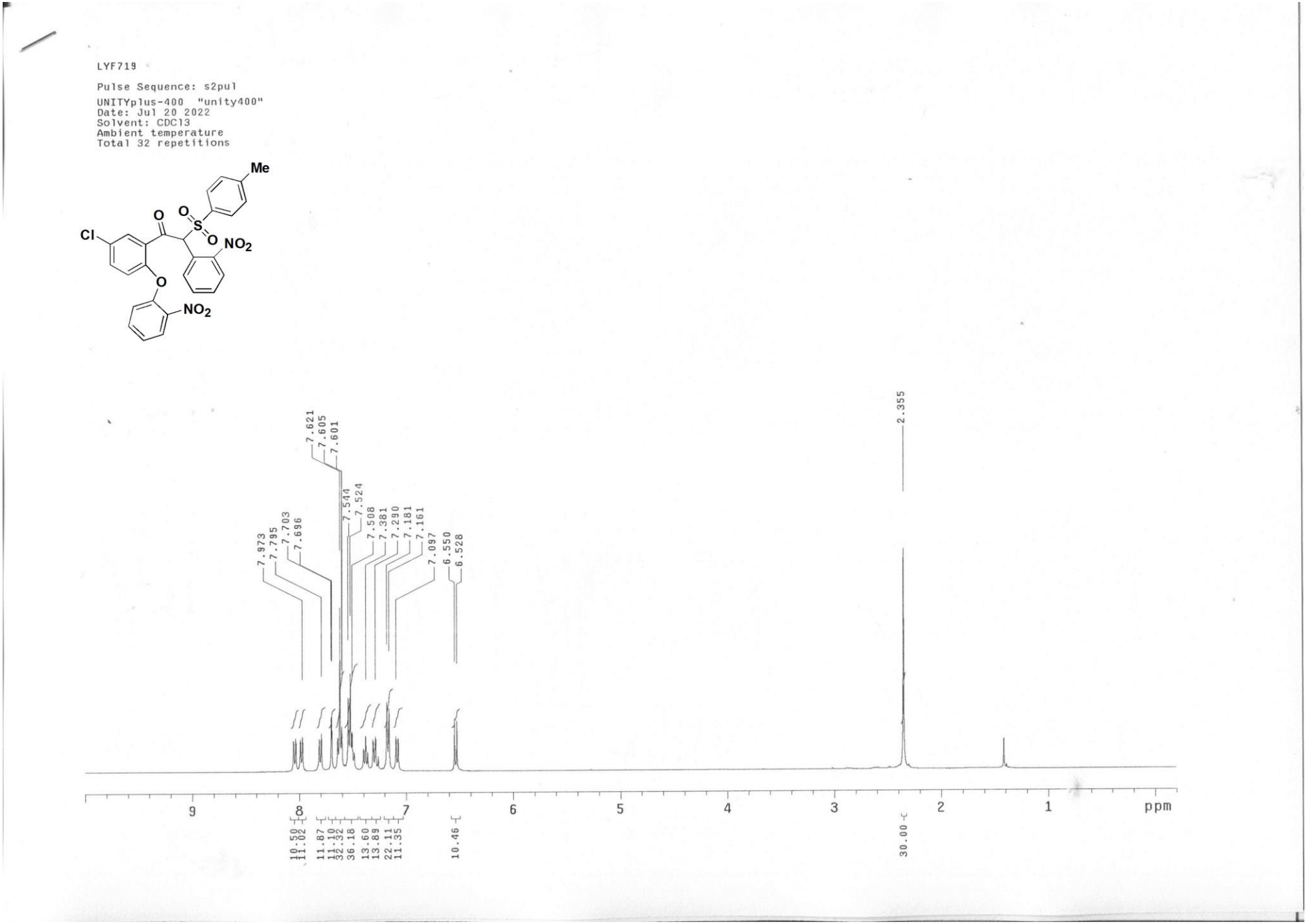
Compound 6b (^{19}F-NMR spectral data-1)



Compound 6b (^{19}F-NMR spectral data-2)



Compound 6c (^{1}H-NMR spectral data)

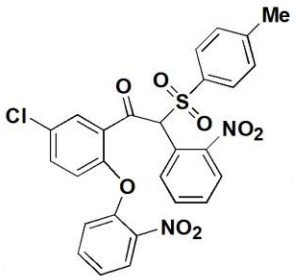


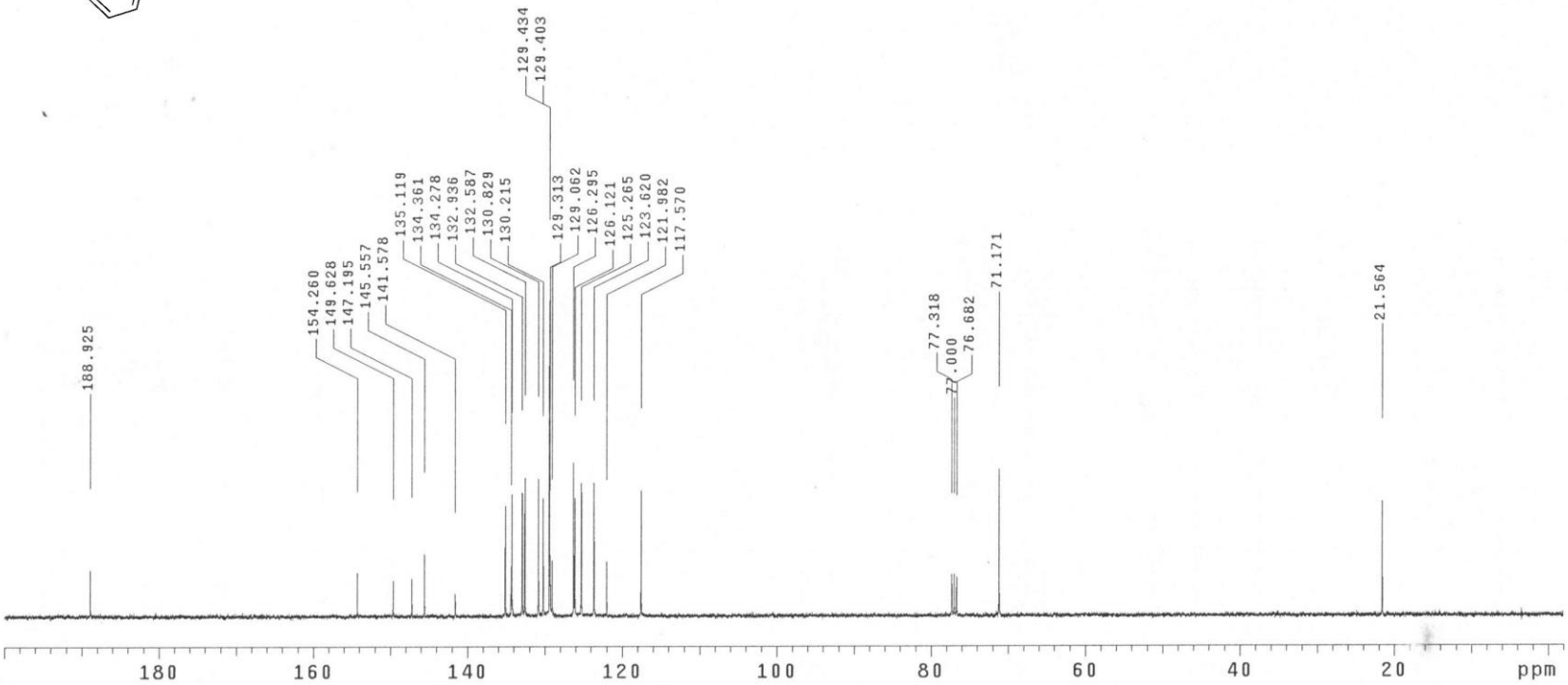
Compound 6c (^{13}C-NMR spectral data)

LYF719

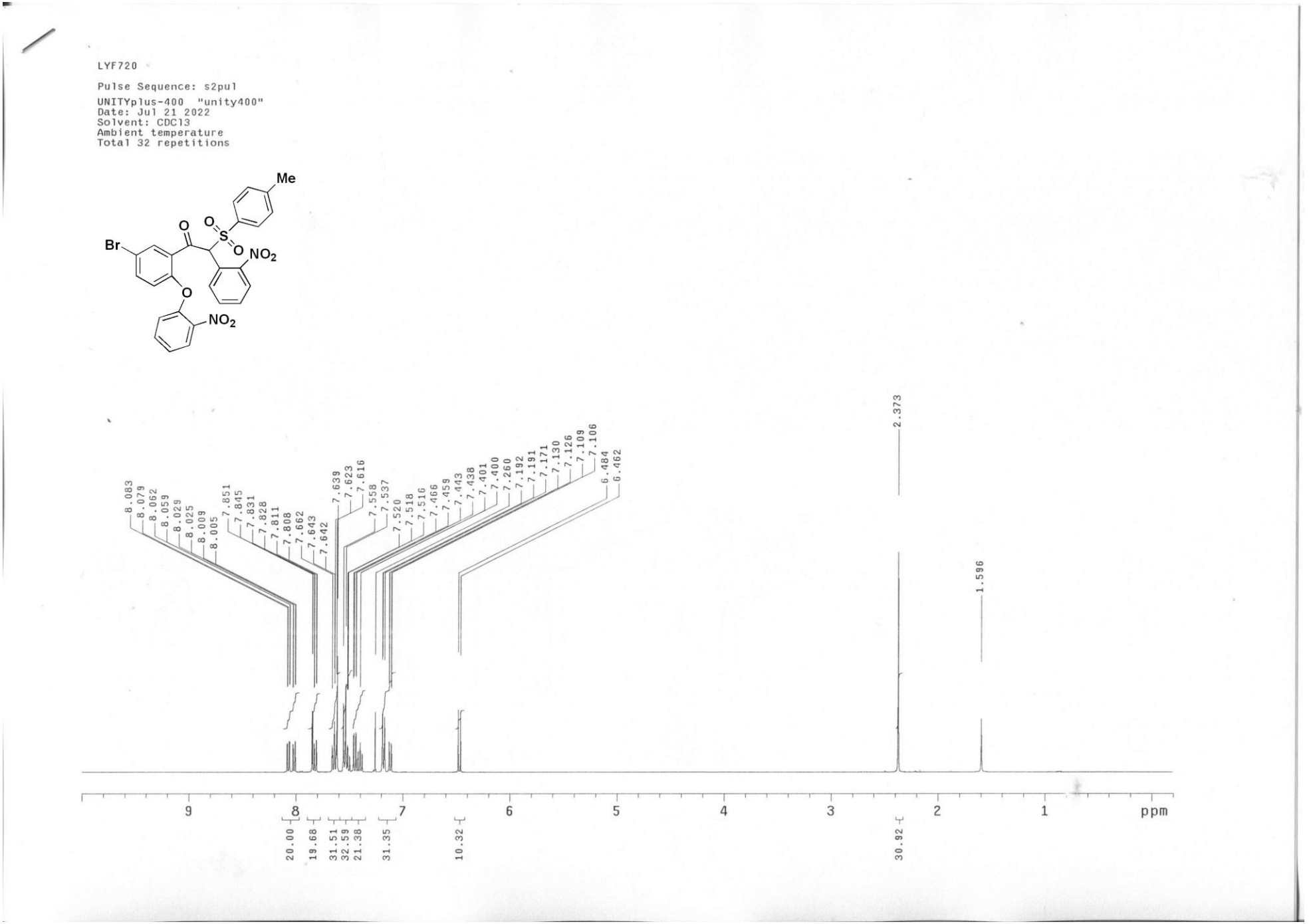
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 20 2022
Solvent: CDCl3
Ambient temperature
Total 672 repetitions

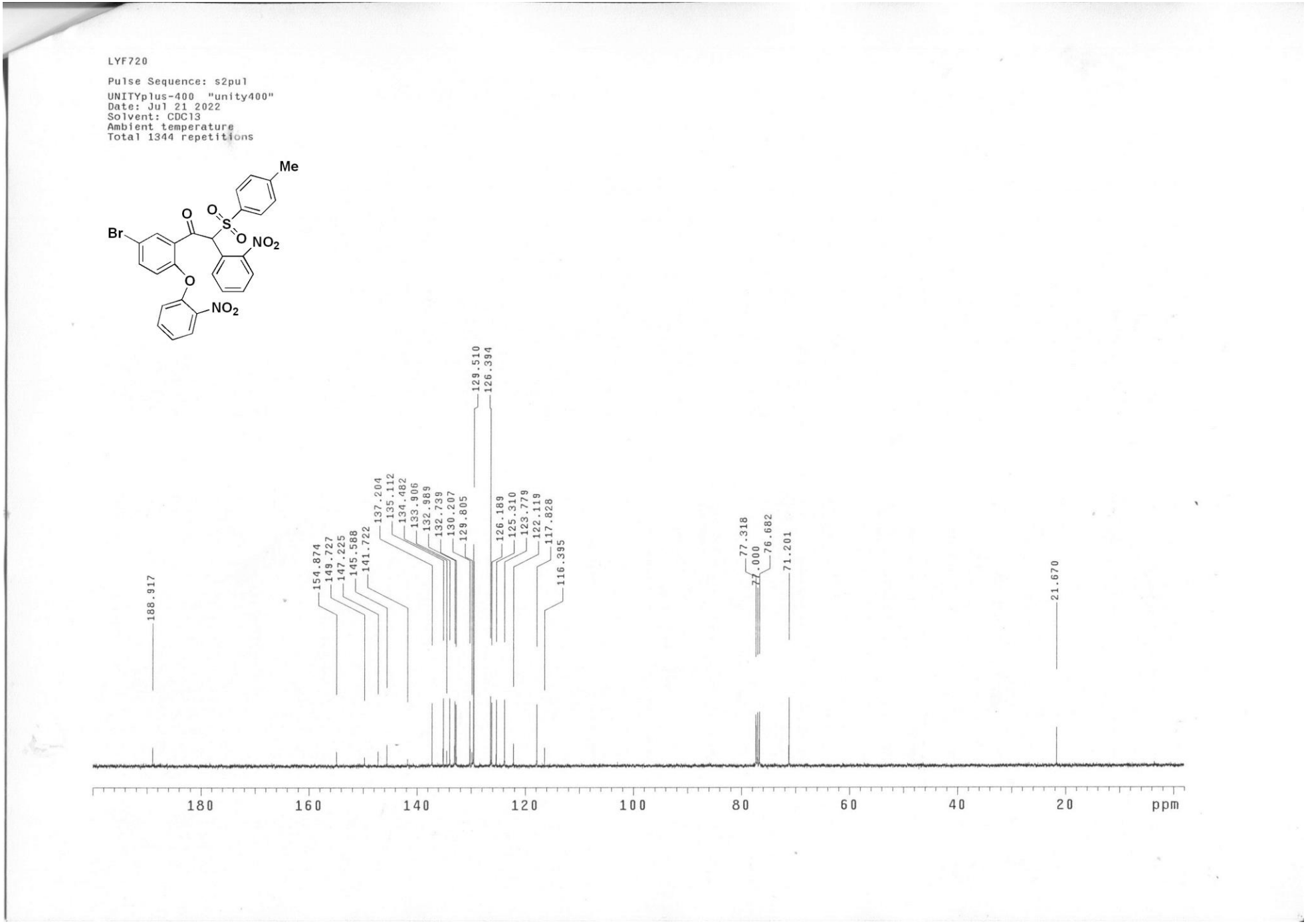




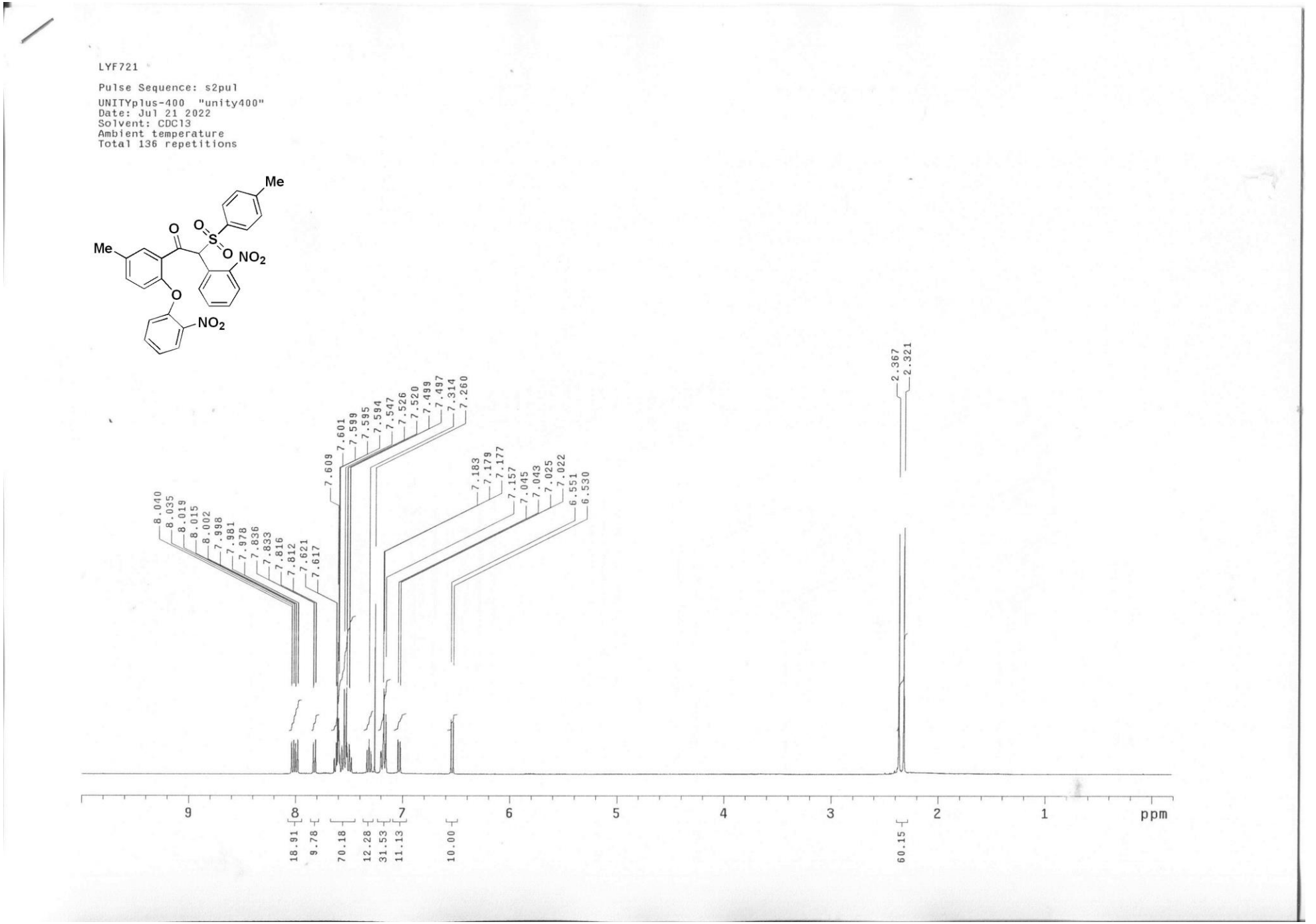
Compound 6d (^{1}H-NMR spectral data)



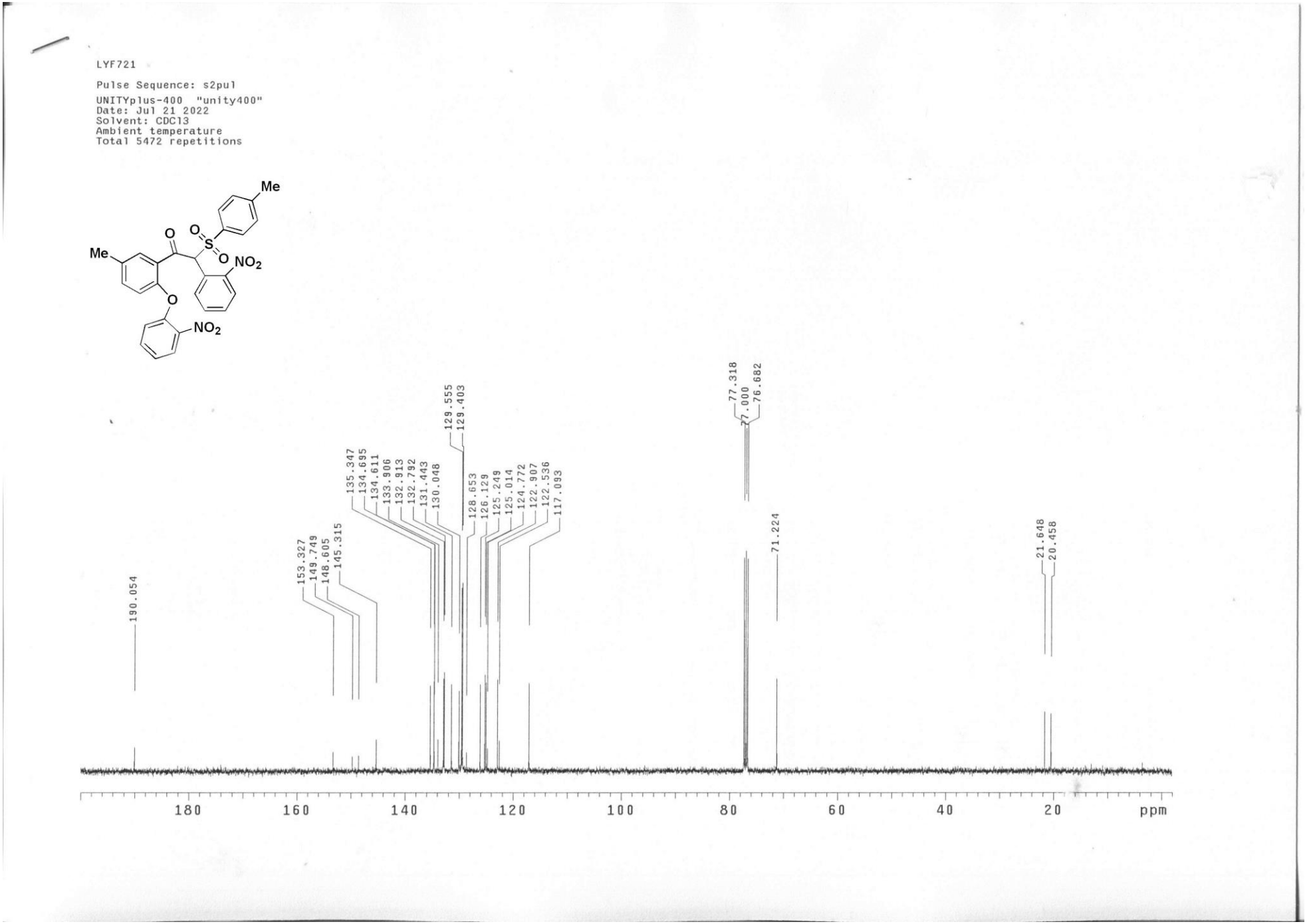
Compound 6d (^{13}C-NMR spectral data)



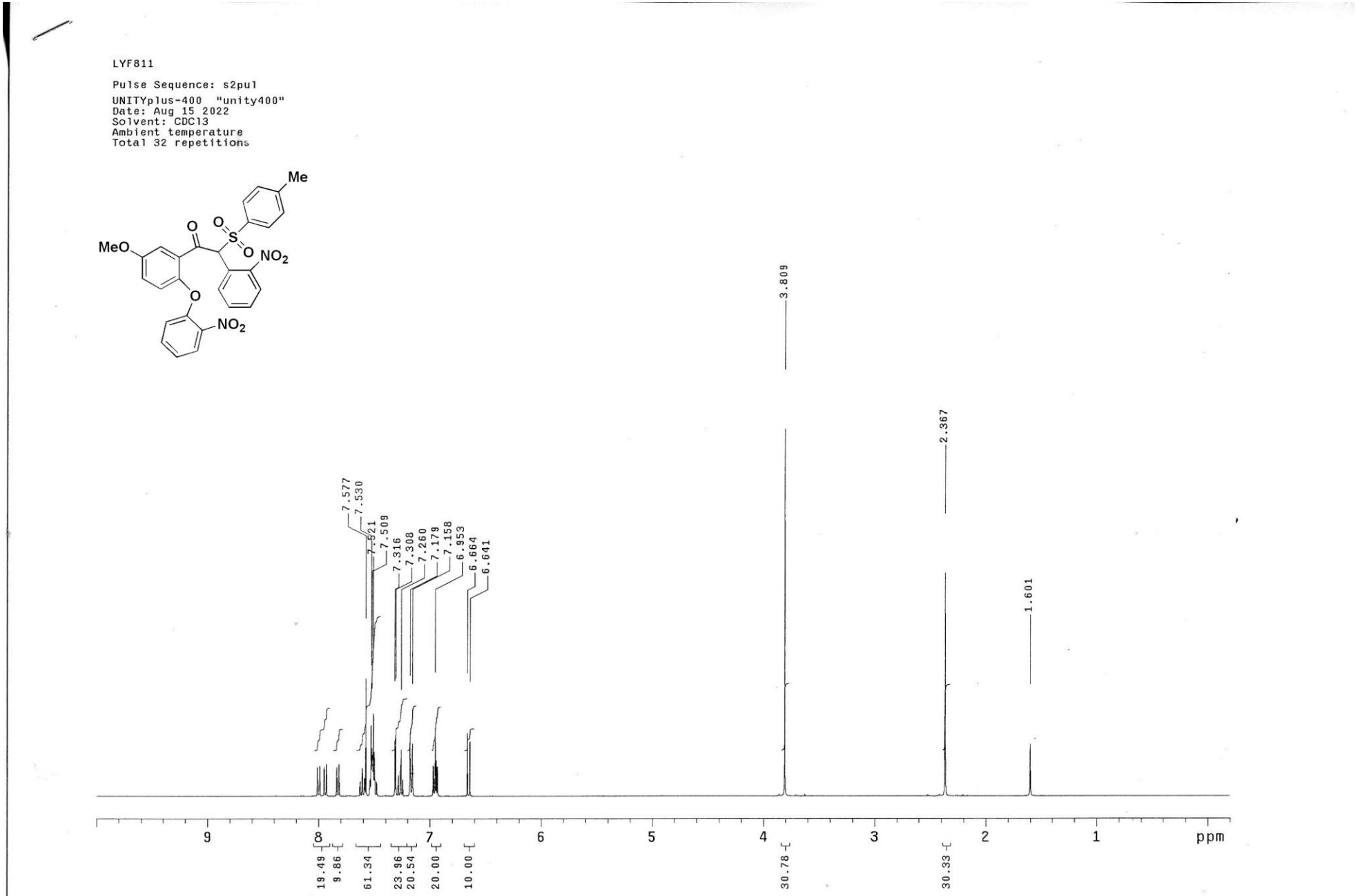
Compound 6e (^{1}H-NMR spectral data)



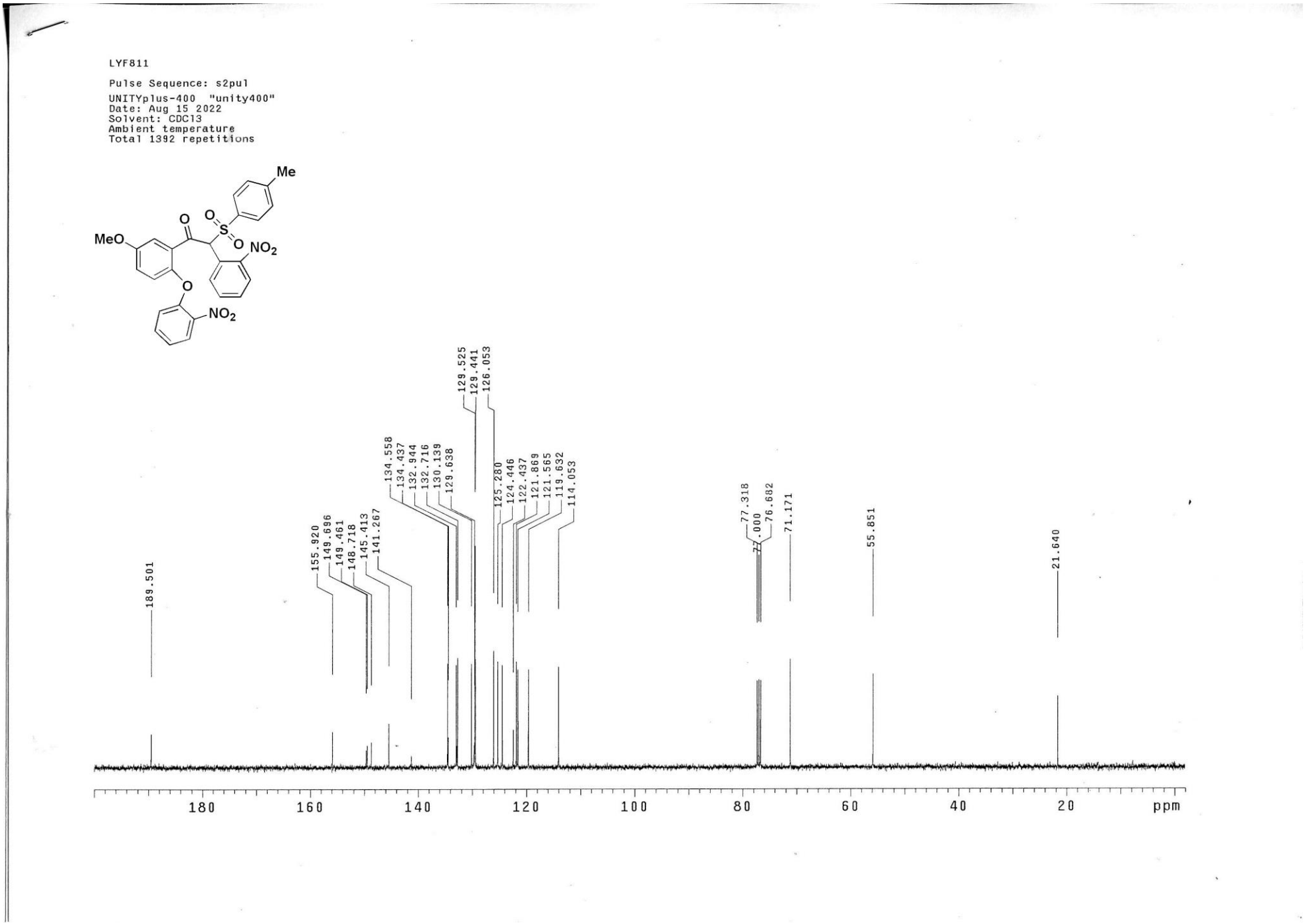
Compound 6e (^{13}C-NMR spectral data)



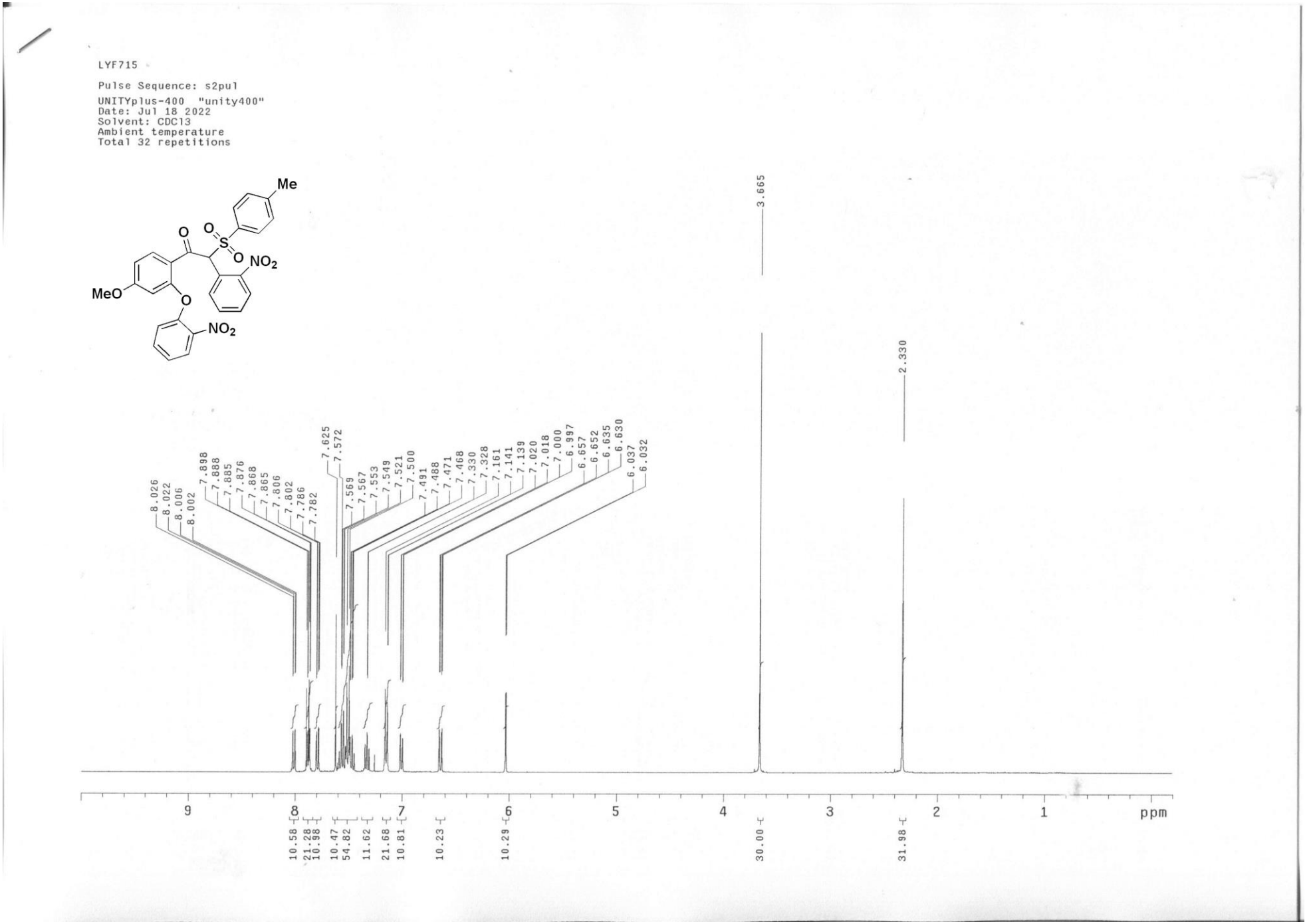
Compound 6f (^{1}H-NMR spectral data)



Compound 6f (^{13}C-NMR spectral data)



Compound 6g (^{1}H-NMR spectral data)

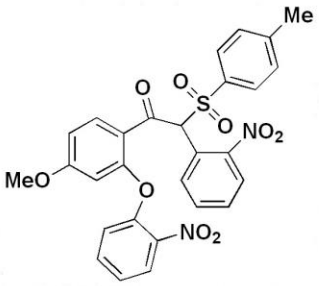


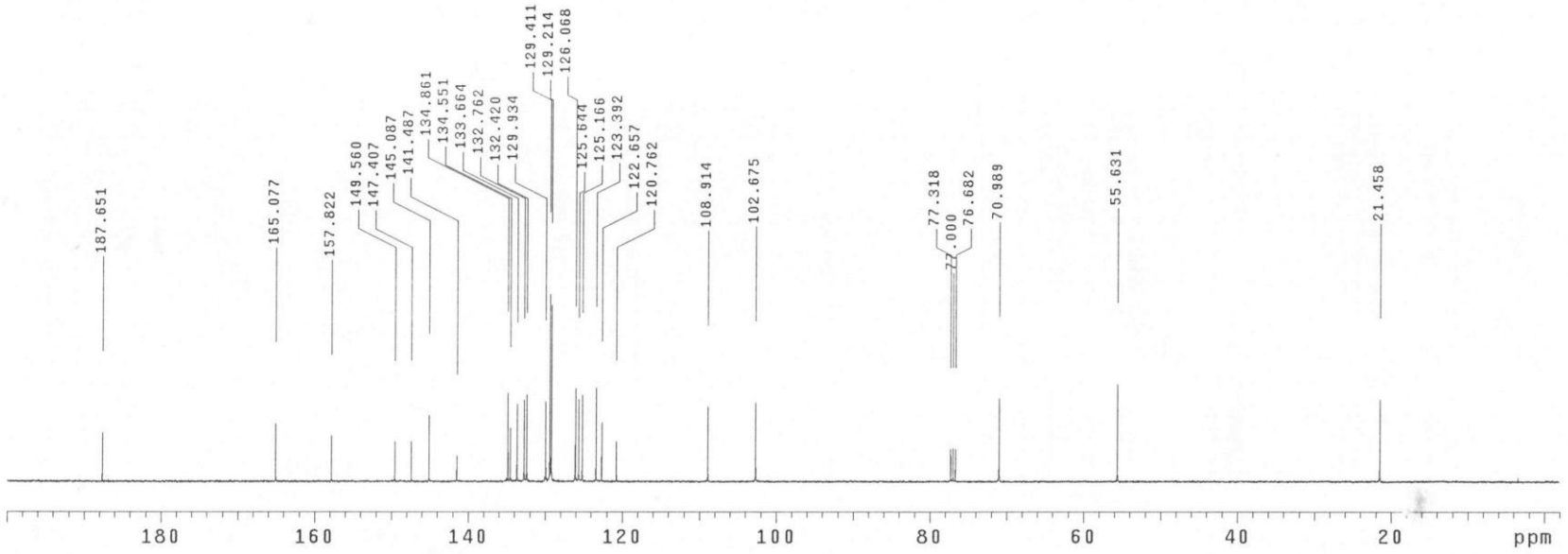
Compound 6g (^{13}C-NMR spectral data)

LYF715

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 18 2022
Solvent: CDCl3
Ambient temperature
Total 816 repetitions



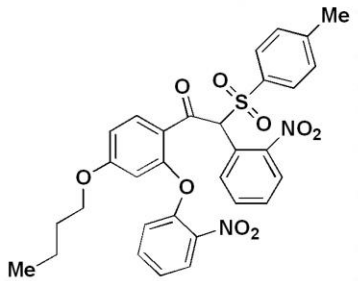


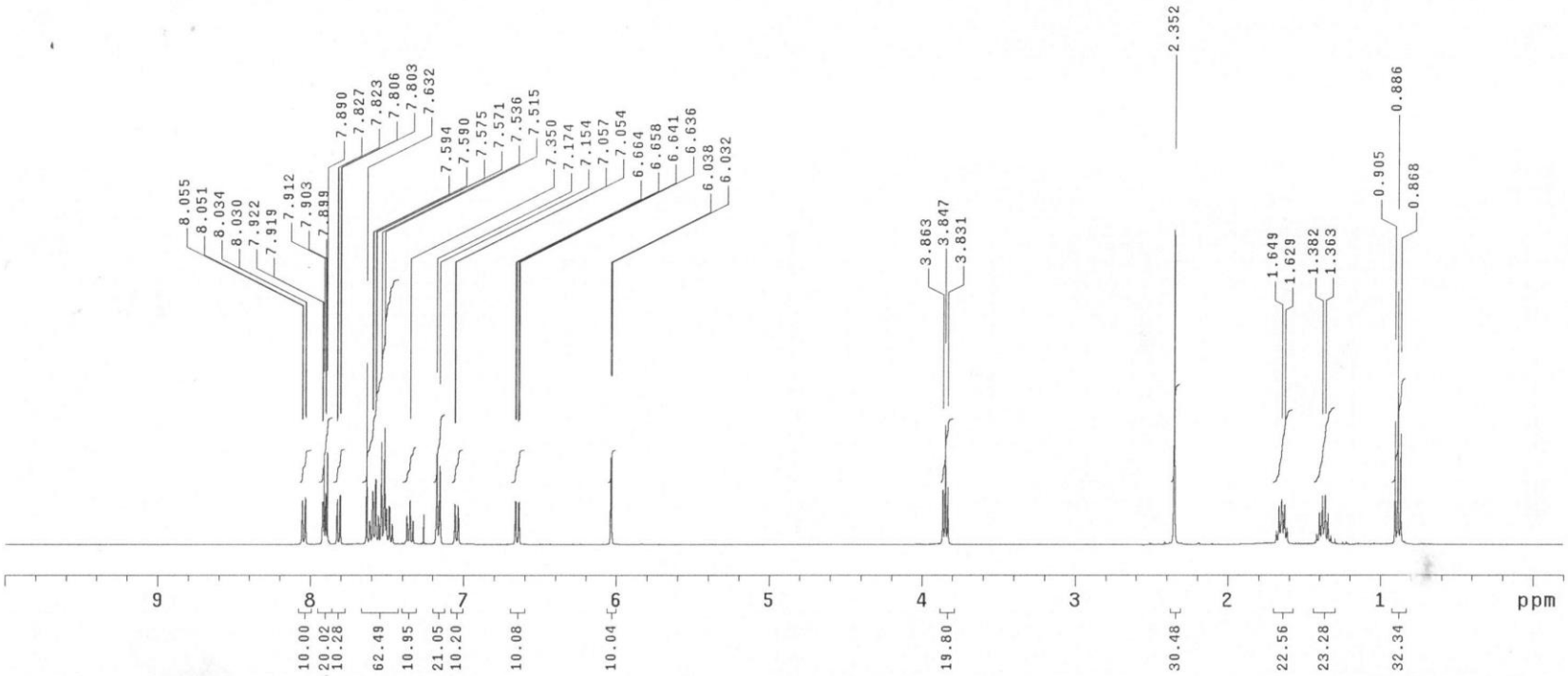
Compound 6h (^{1}H-NMR spectral data)

LYF727

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 28 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions





Compound 6h (^{13}C-NMR spectral data)

LYF727

Pulse Sequence: s2pul

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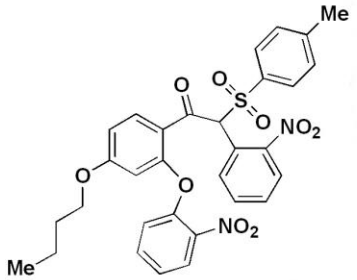
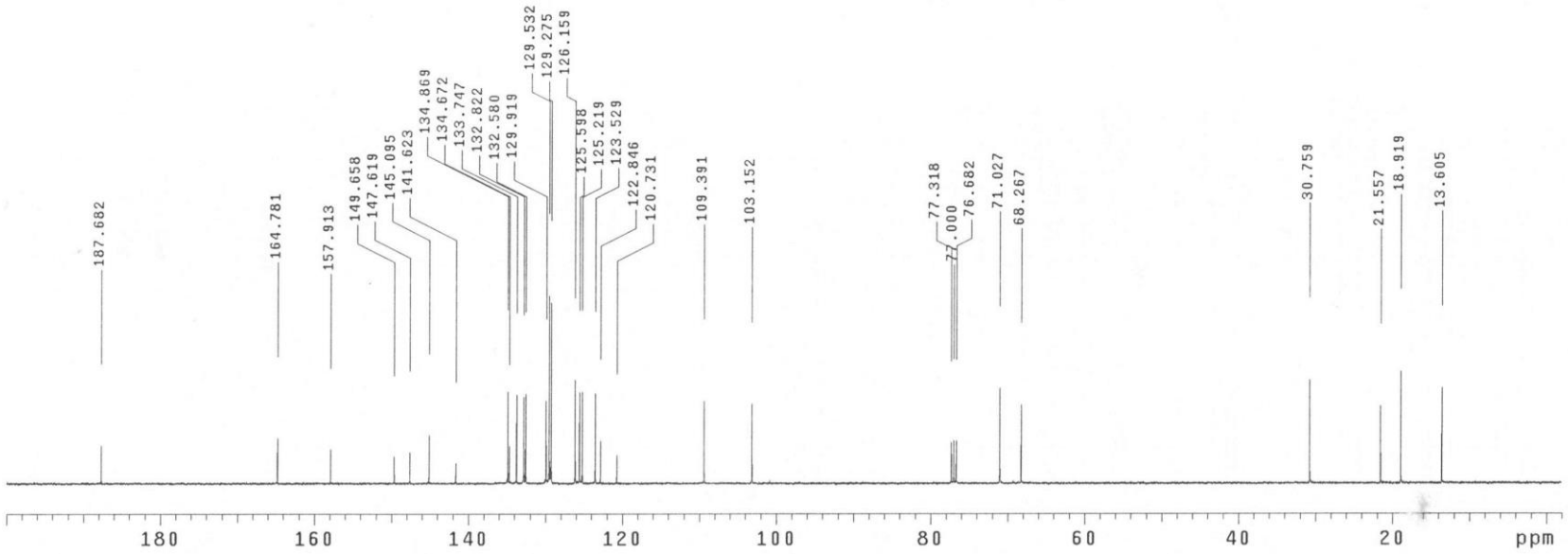
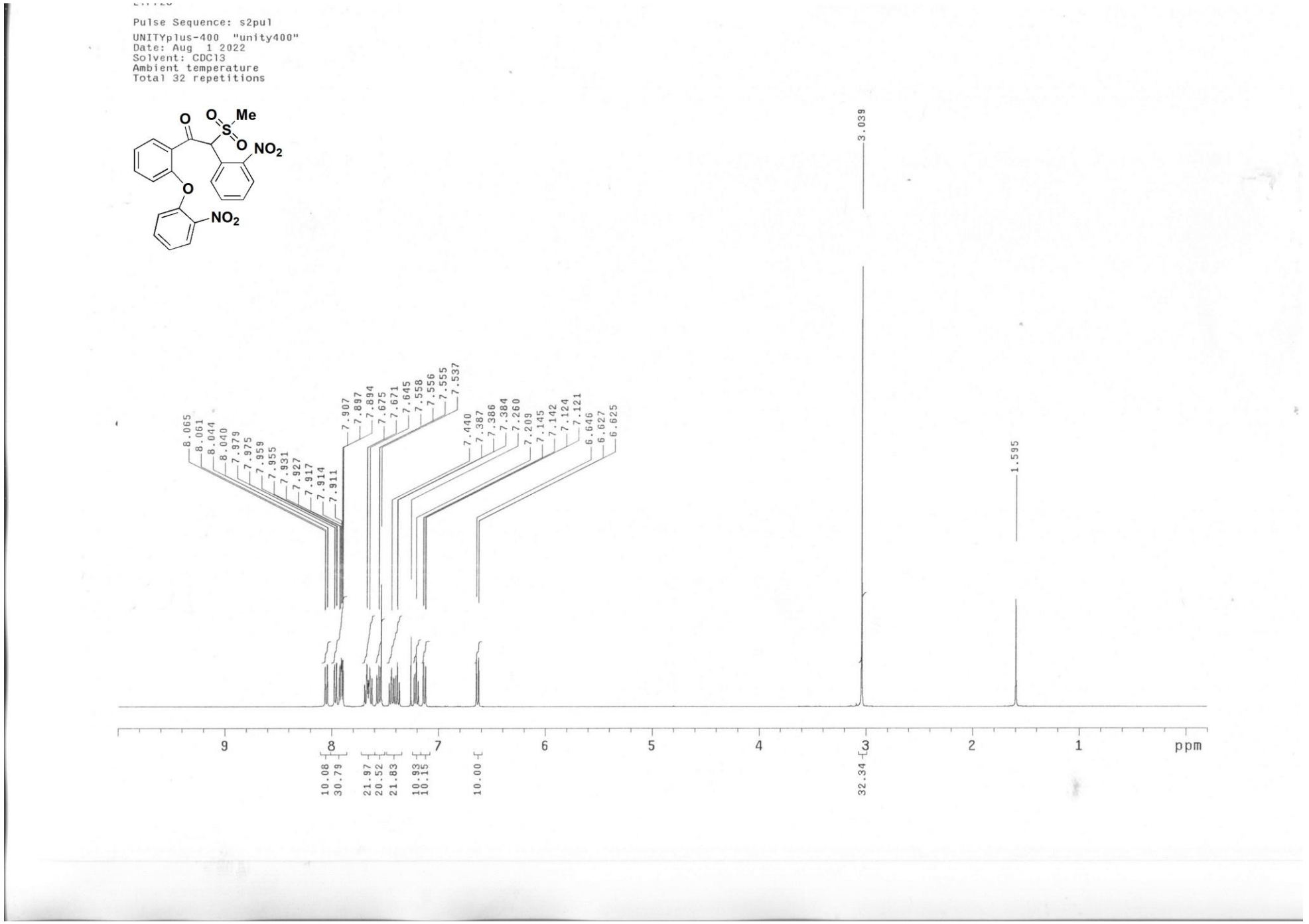
Date: Jul 28 2022
Solvent: CDCl3
Ambient temperature
Total 1552 repetitions

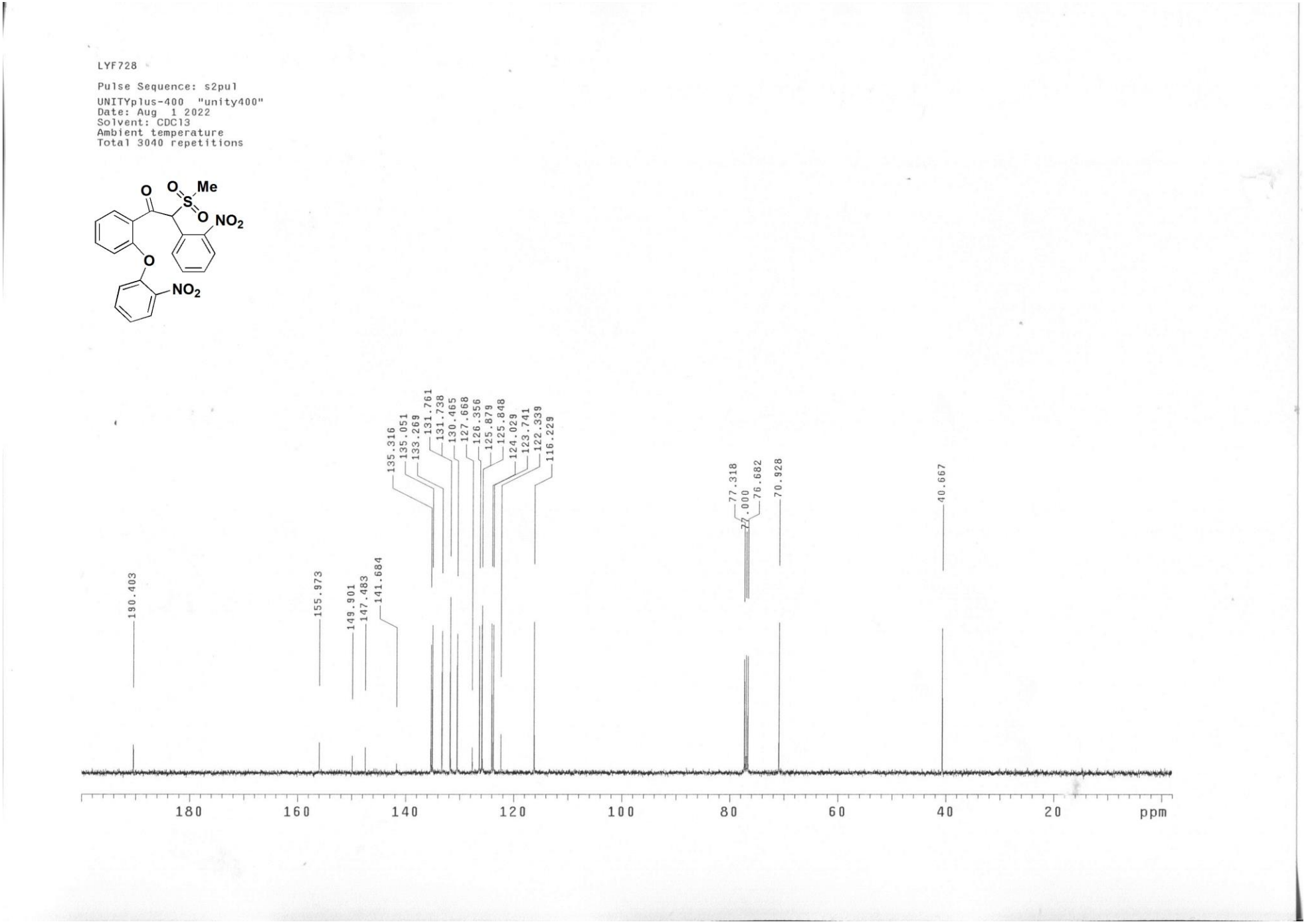




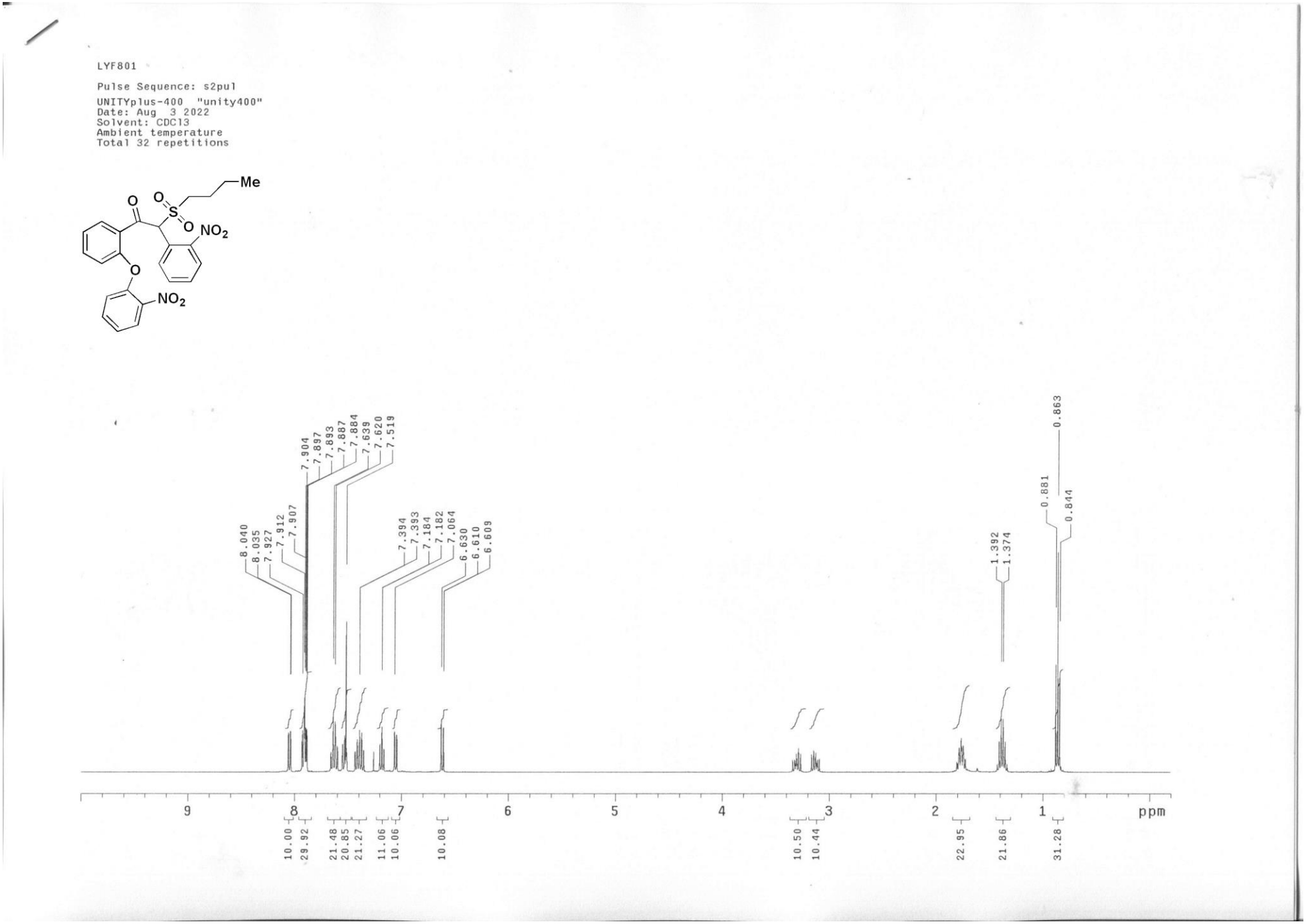
Compound 6i (^{1}H-NMR spectral data)



Compound 6i (^{13}C-NMR spectral data)



Compound 6j (^{1}H-NMR spectral data)

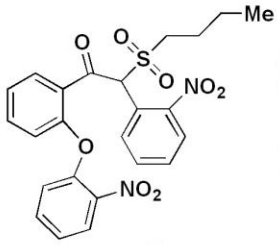


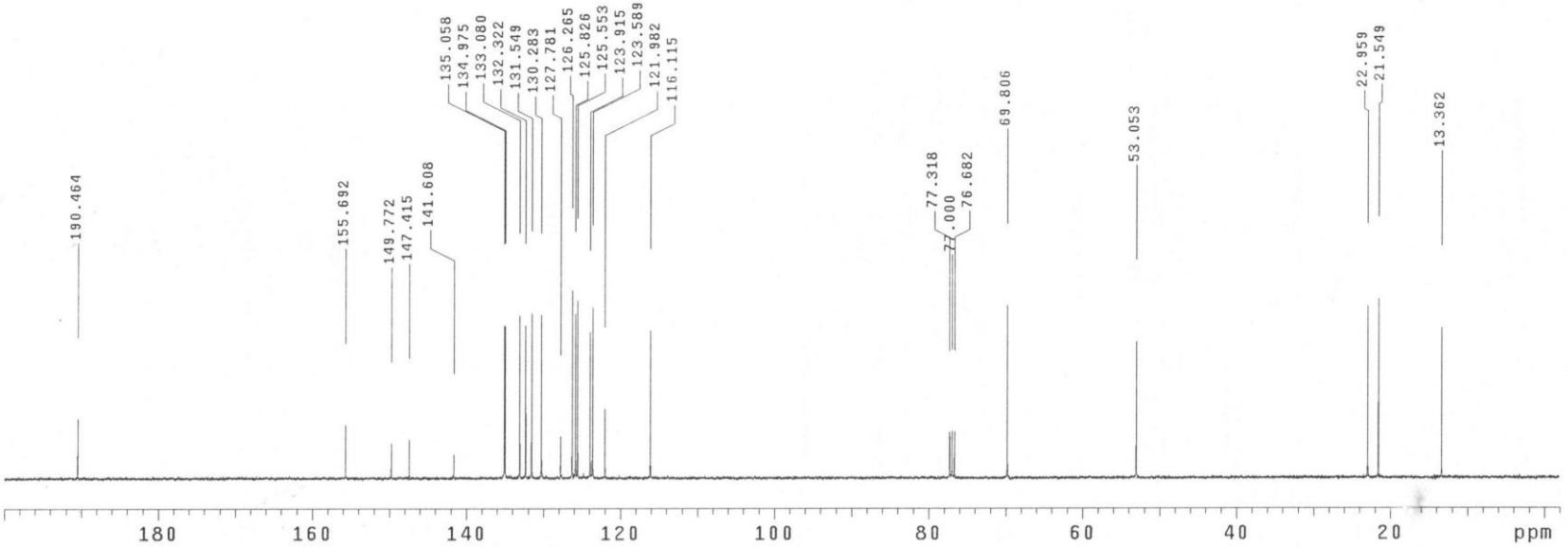
Compound 6j (^{13}C-NMR spectral data)

LYF801

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 3 2022
Solvent: CDCl3
Ambient temperature
Total 912 repetitions



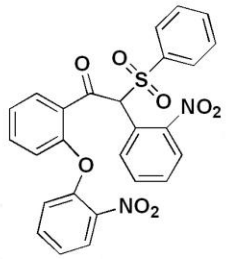


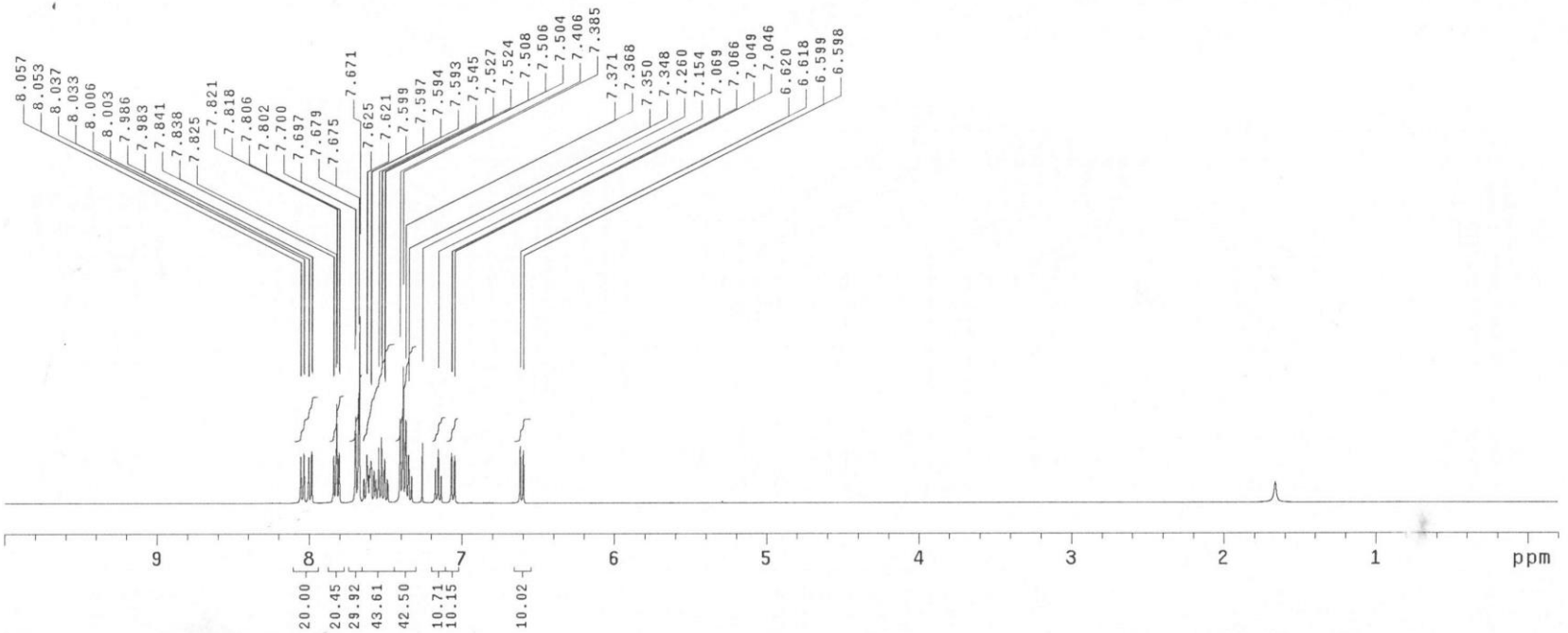
Compound 6k (^{1}H-NMR spectral data)

LYF802

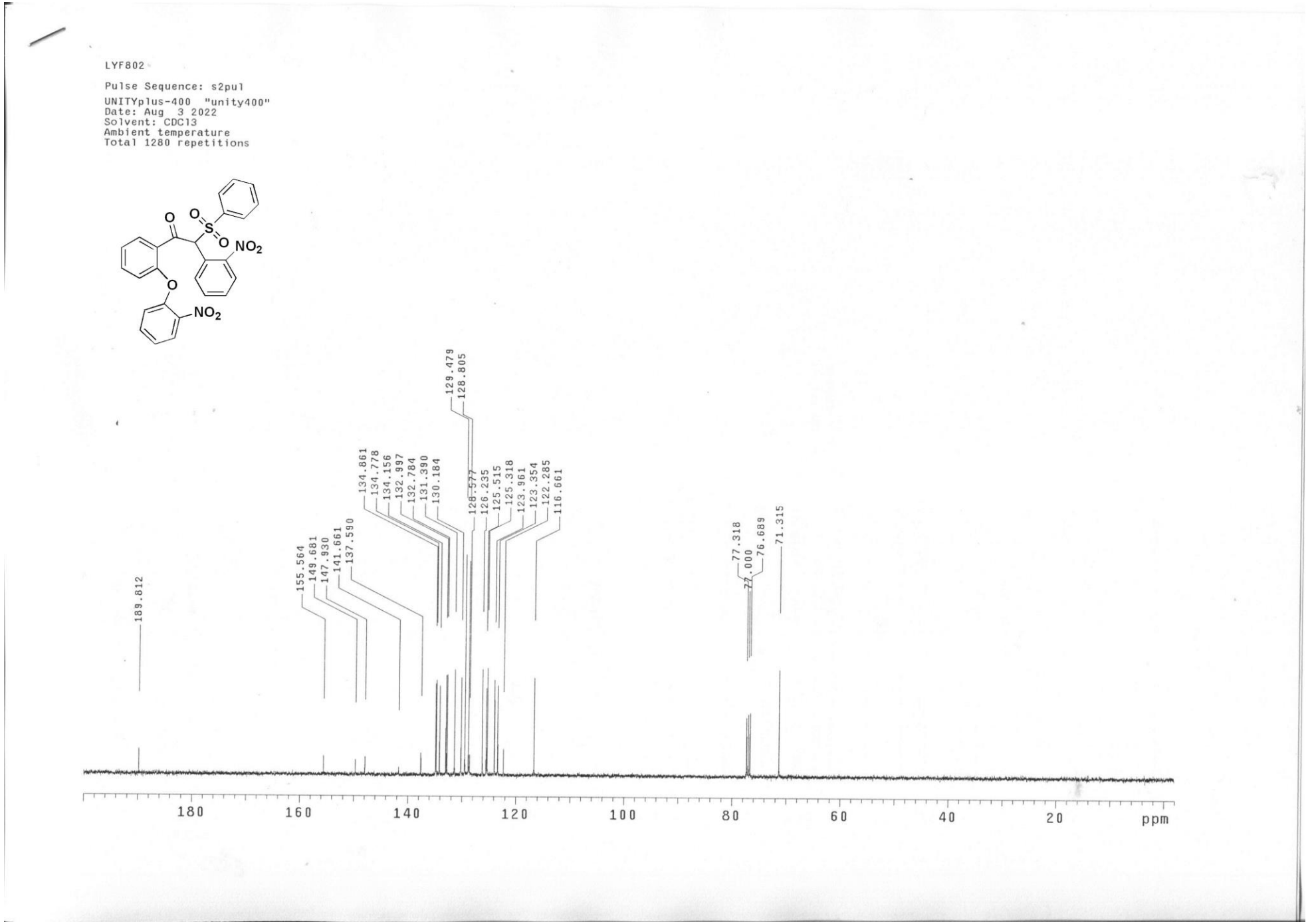
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 3 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

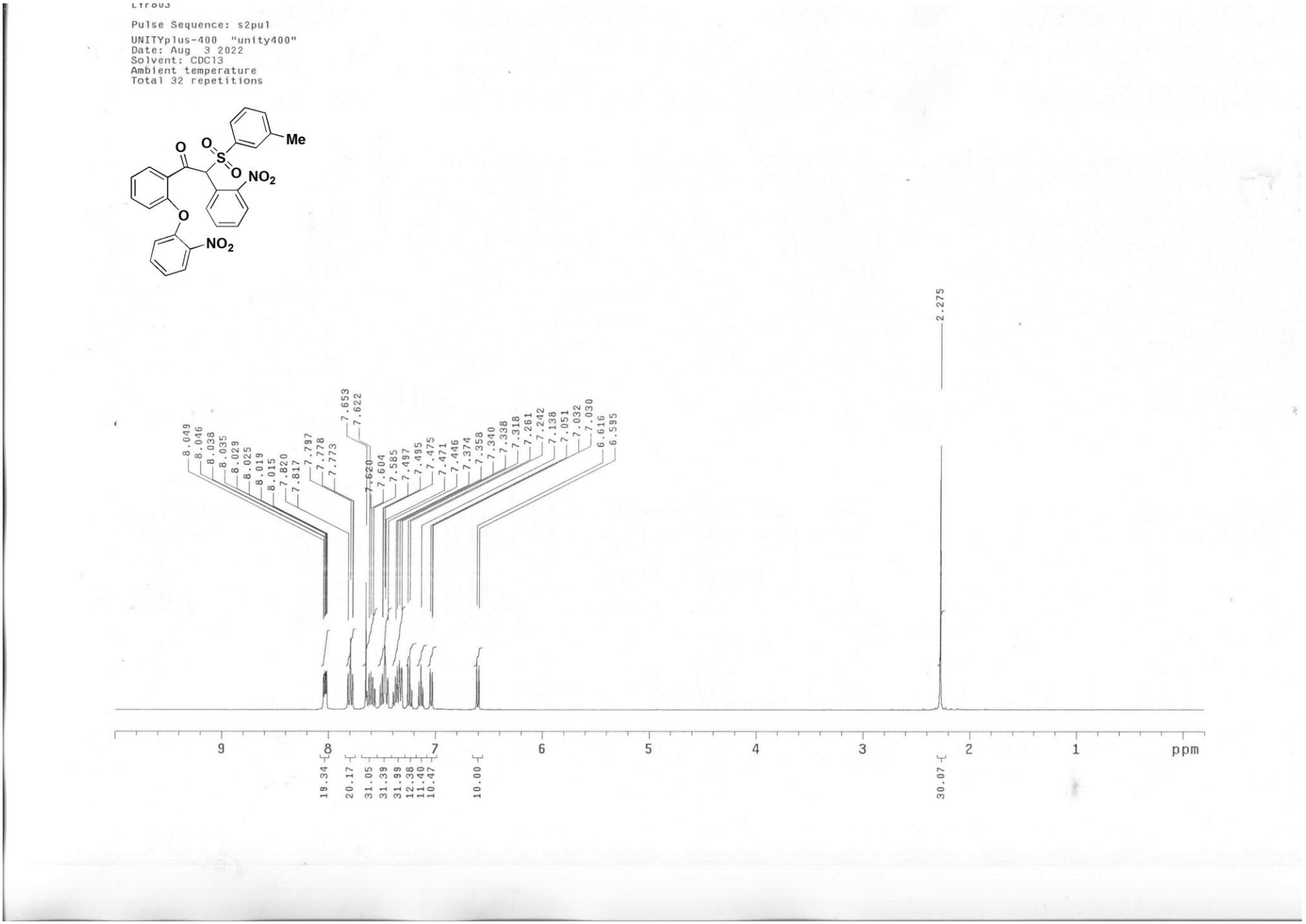




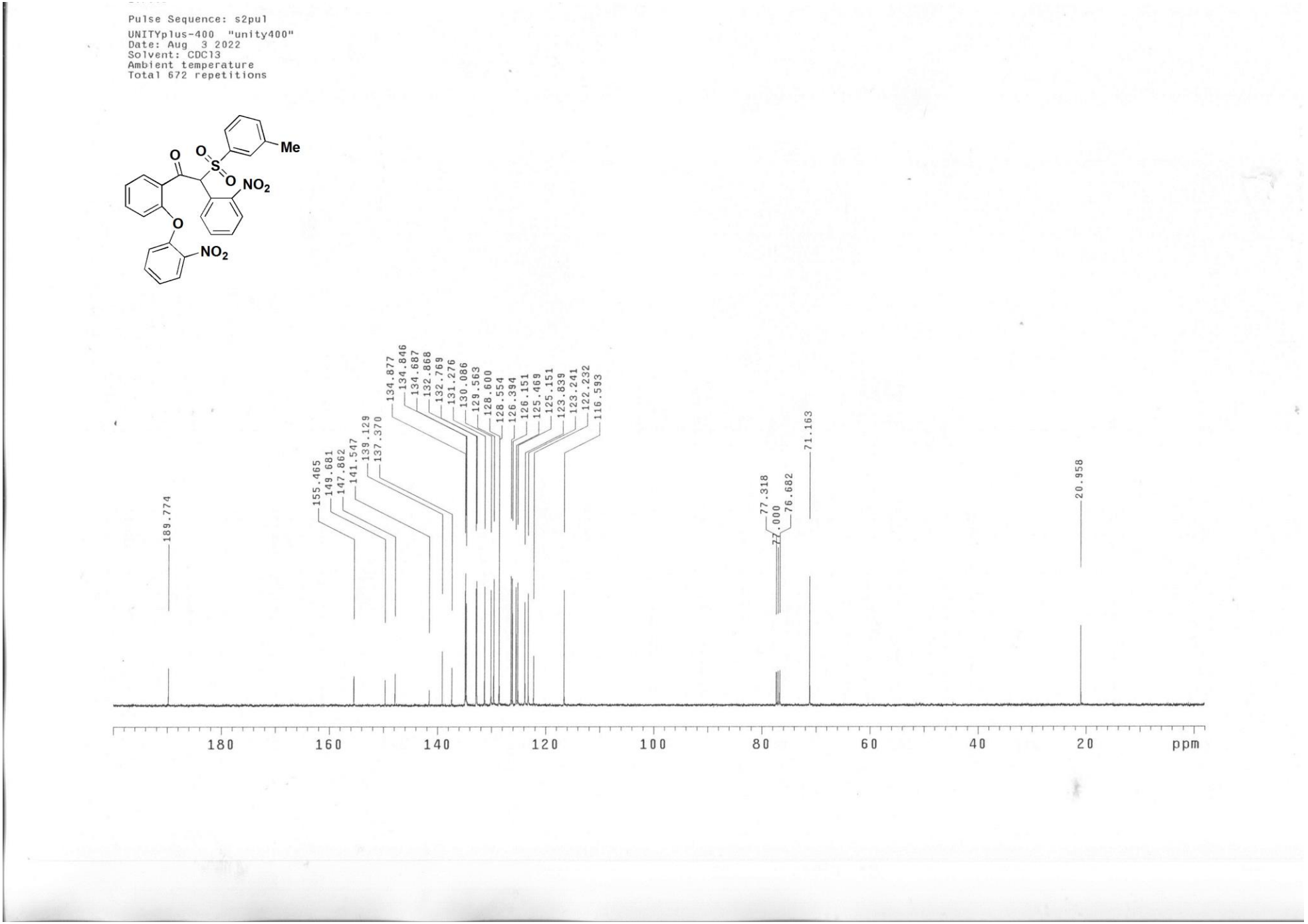
Compound 6k (^{13}C-NMR spectral data)



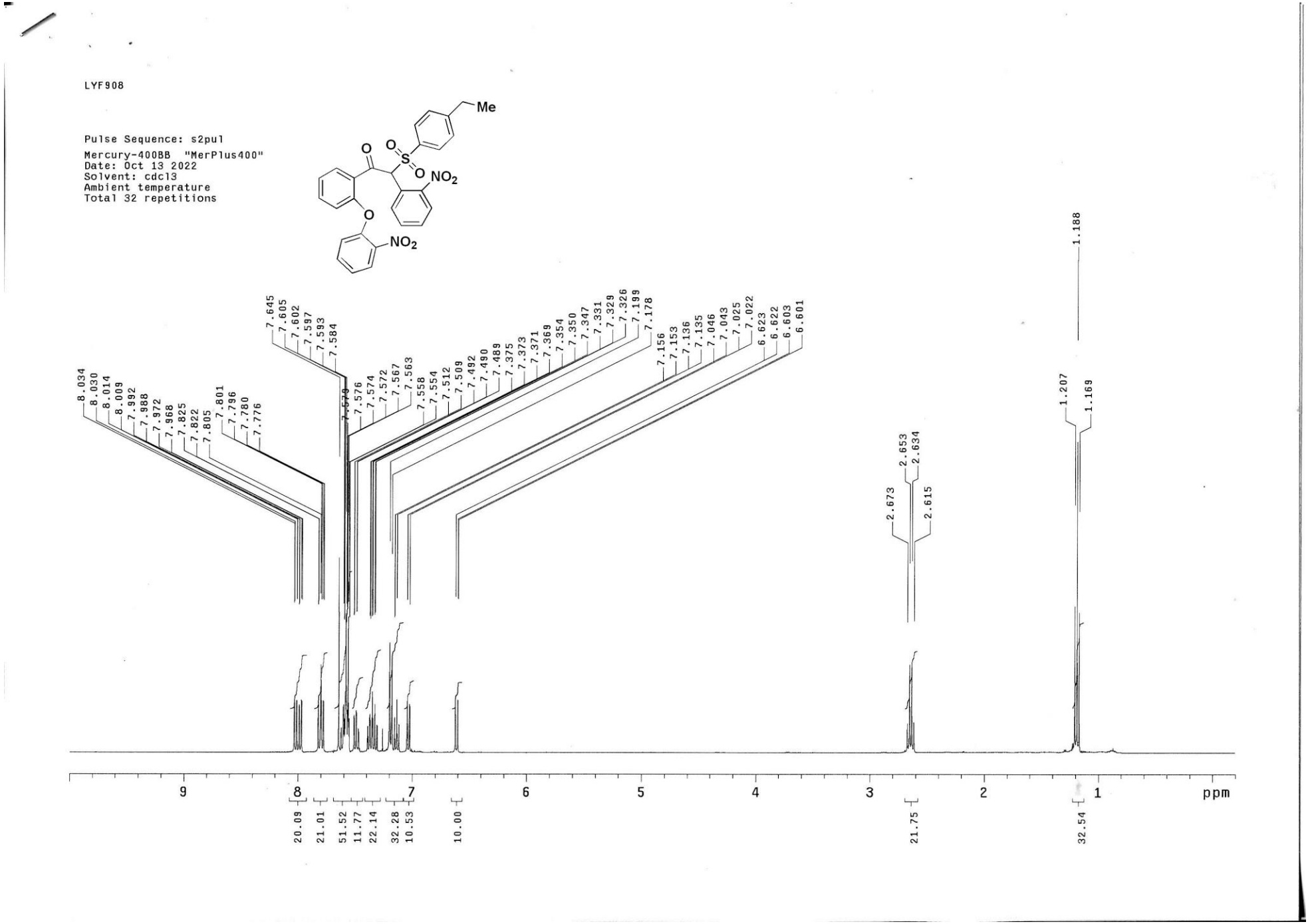
Compound 6l (^{1}H-NMR spectral data)



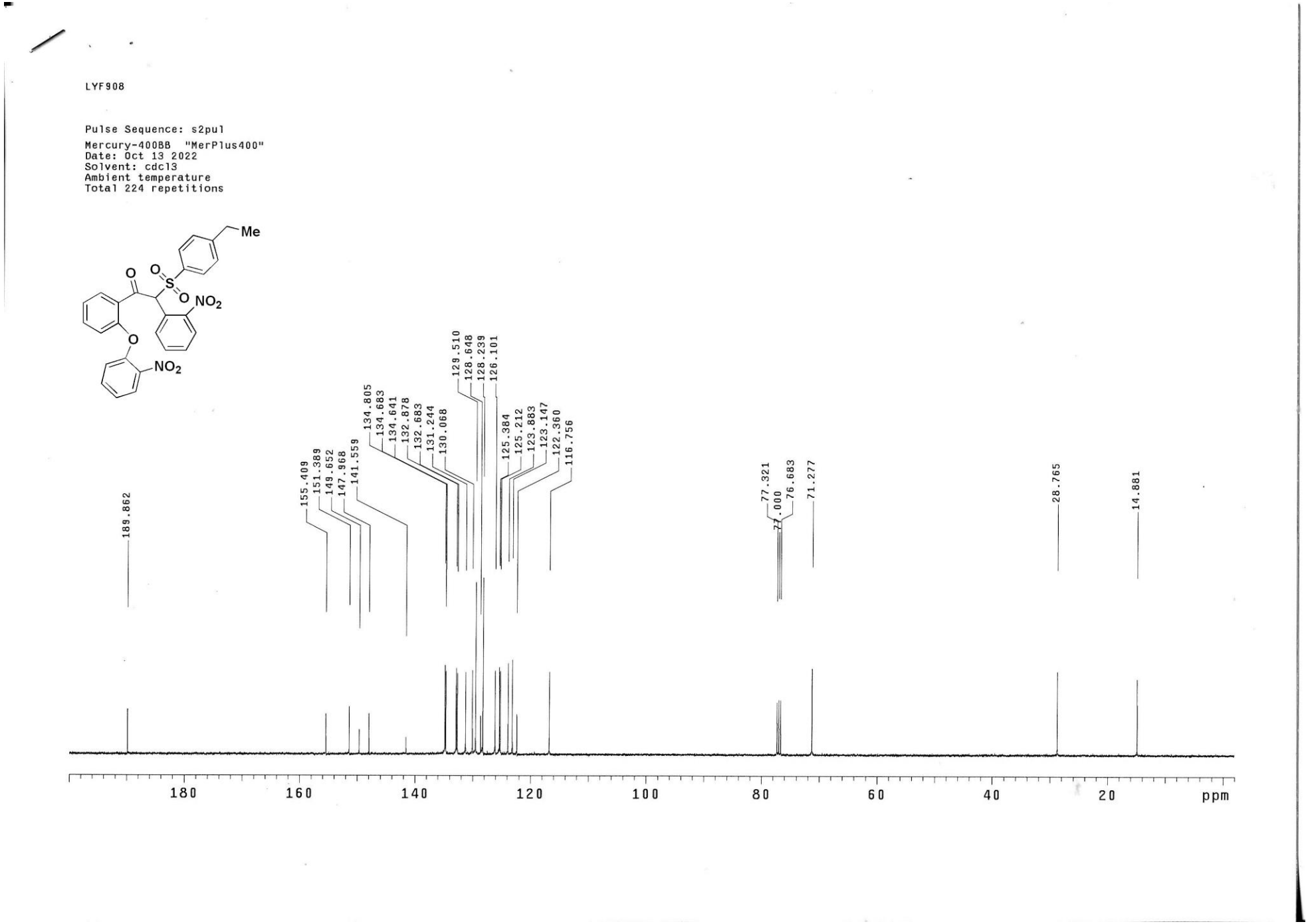
Compound 6l (^{13}C-NMR spectral data)



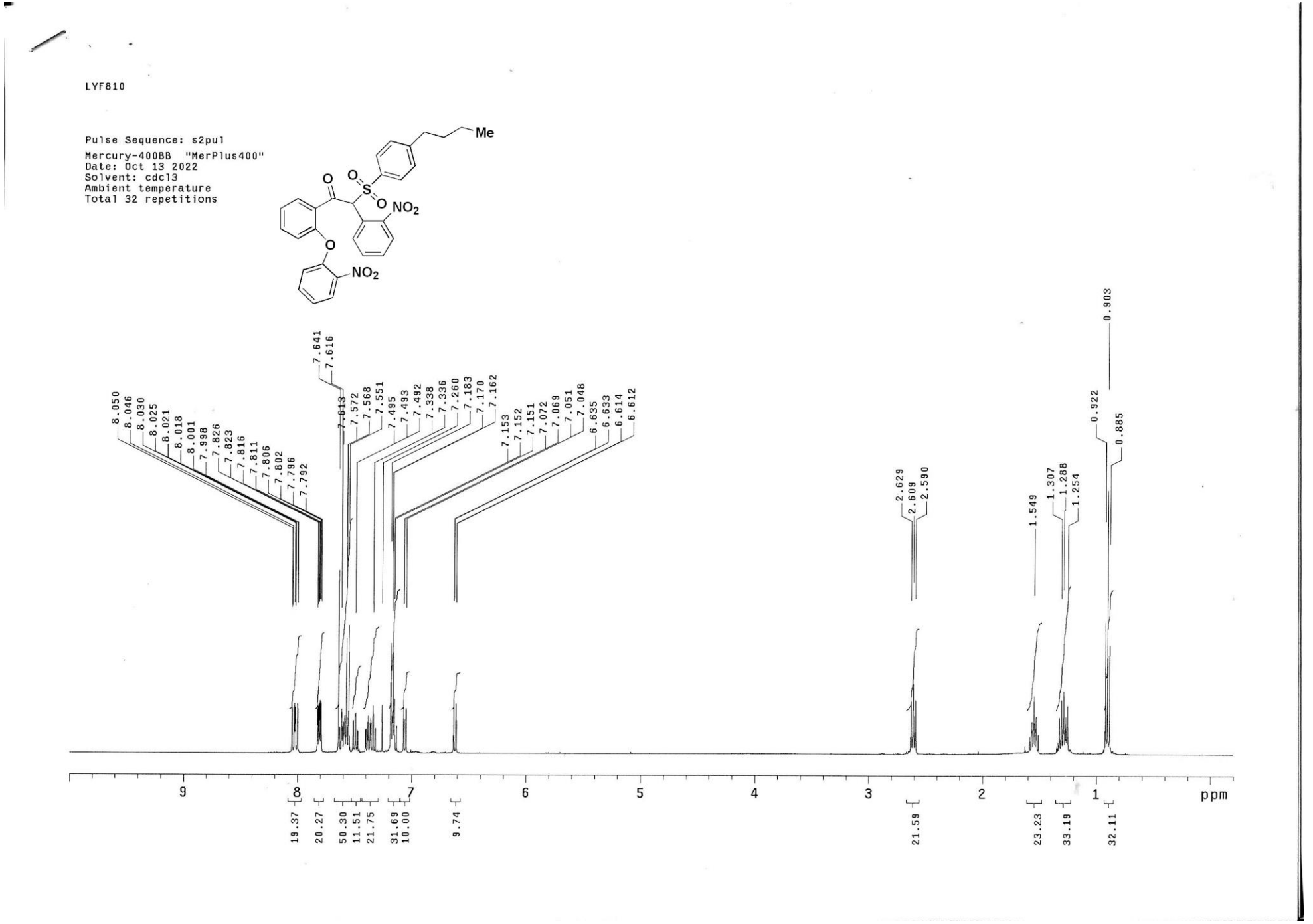
Compound 6m (^{1}H-NMR spectral data)



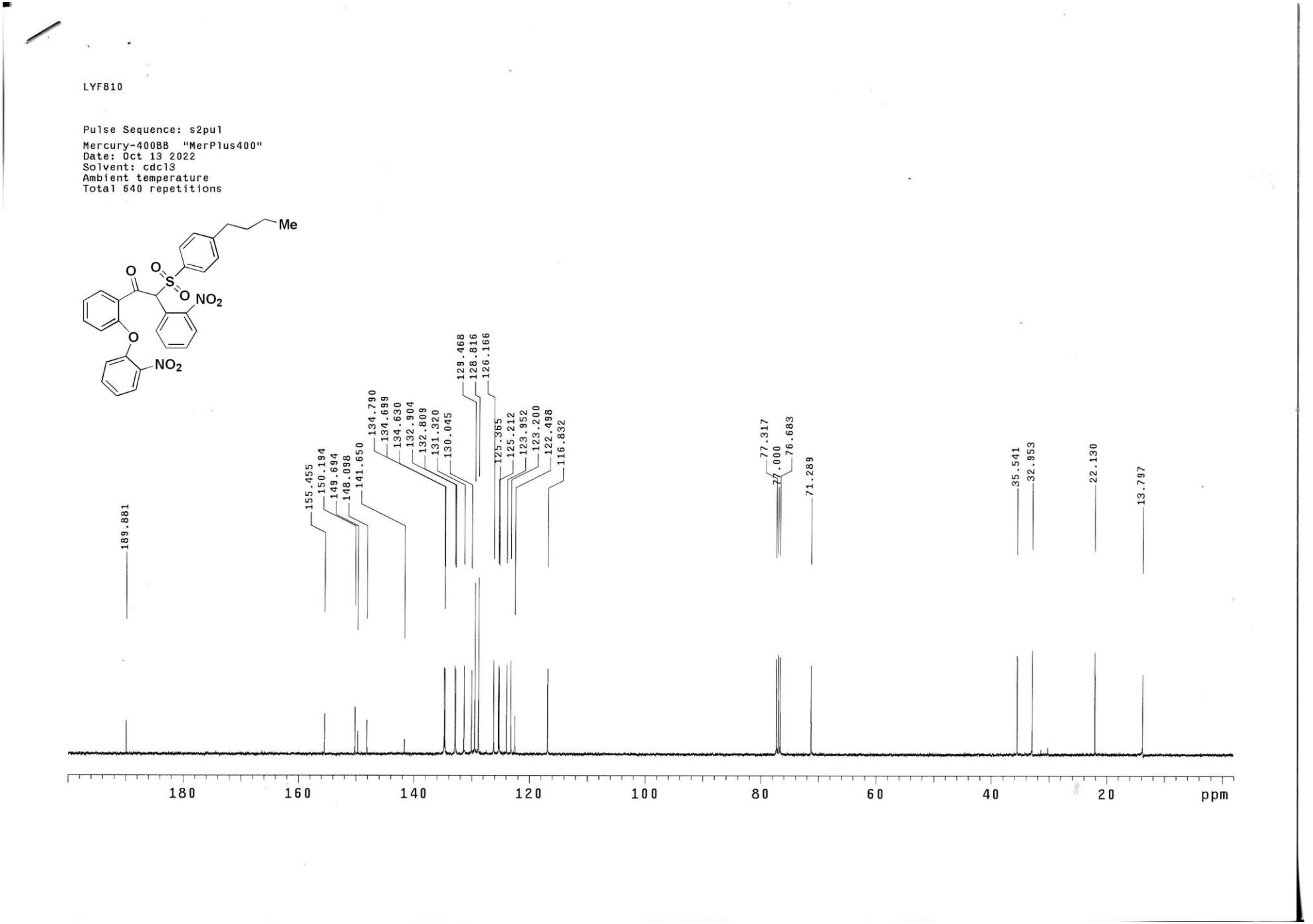
Compound 6m (^{13}C-NMR spectral data)



Compound 6n (^{1}H-NMR spectral data)



Compound 6n (^{13}C-NMR spectral data)

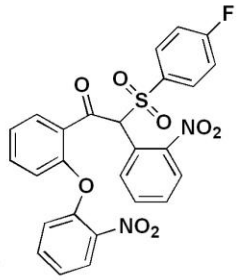


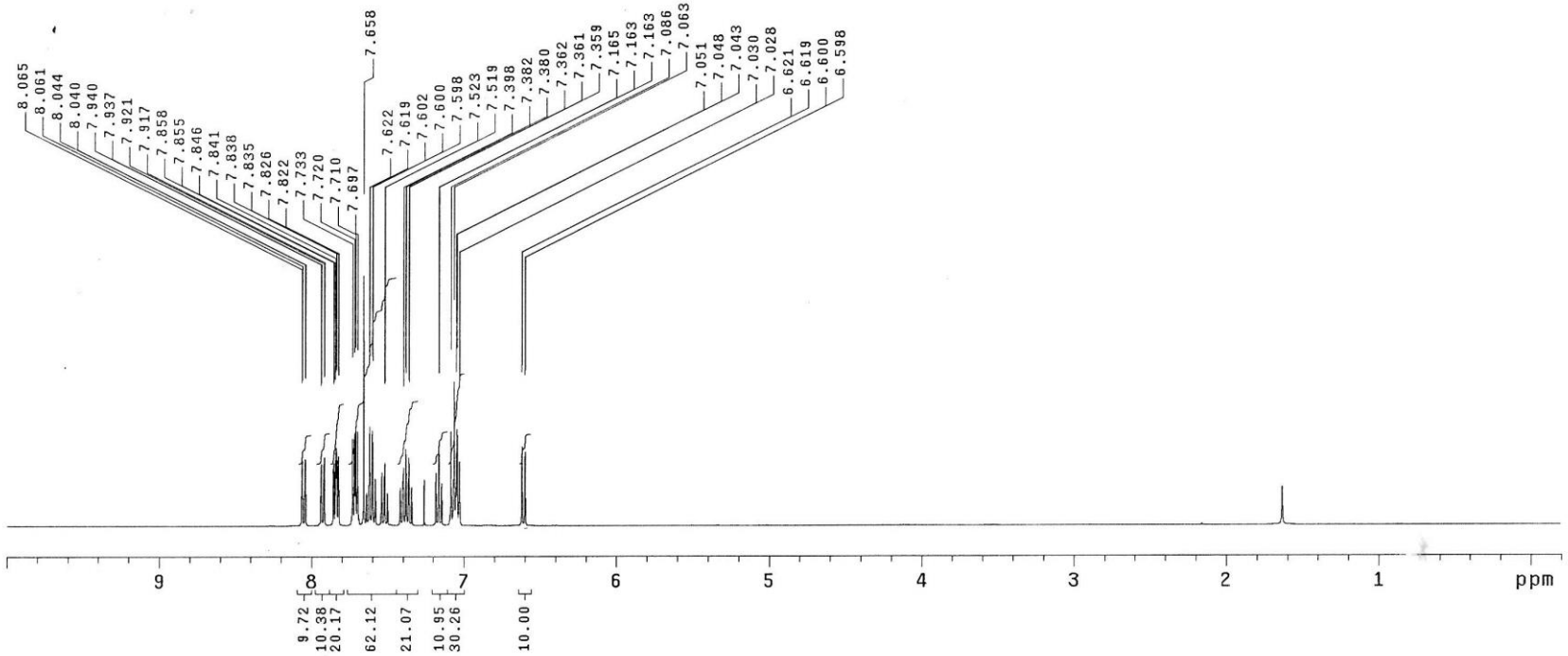
Compound 6o (^{1}H-NMR spectral data)

LYF809

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 10 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

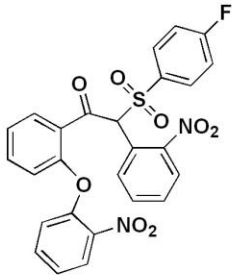


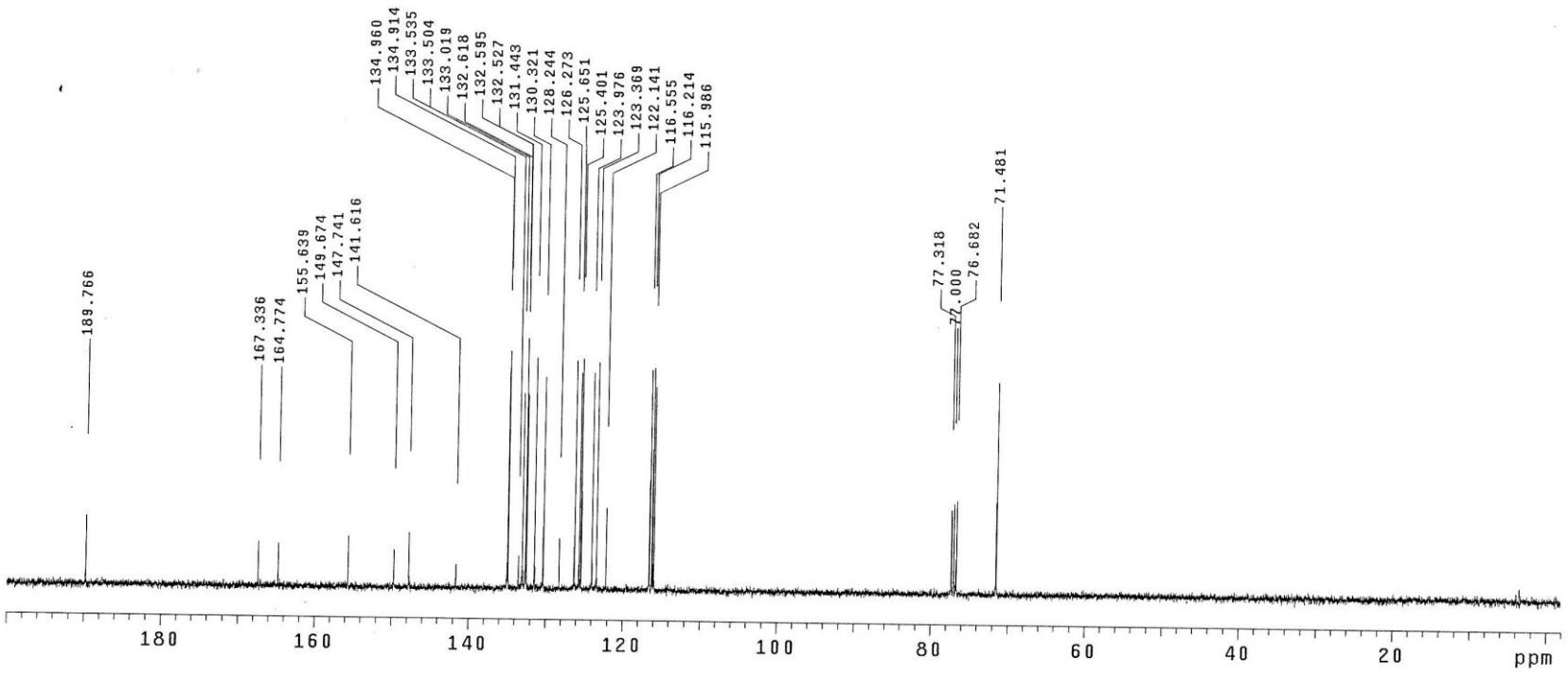


Compound 6o (^{13}C-NMR spectral data)

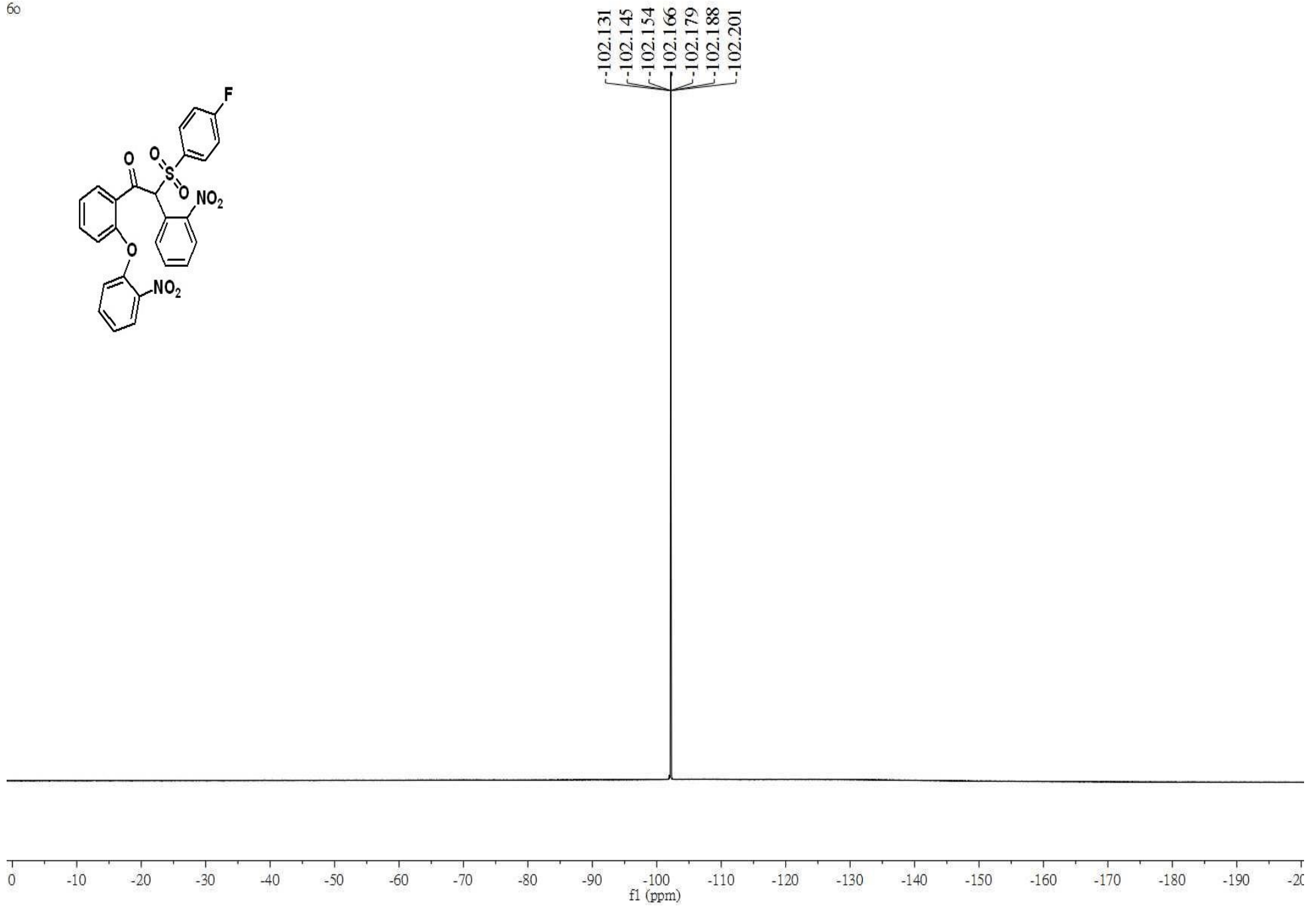
LYF809

Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 10 2022
Solvent: CDCl3
Ambient temperature
Total 720 repetitions

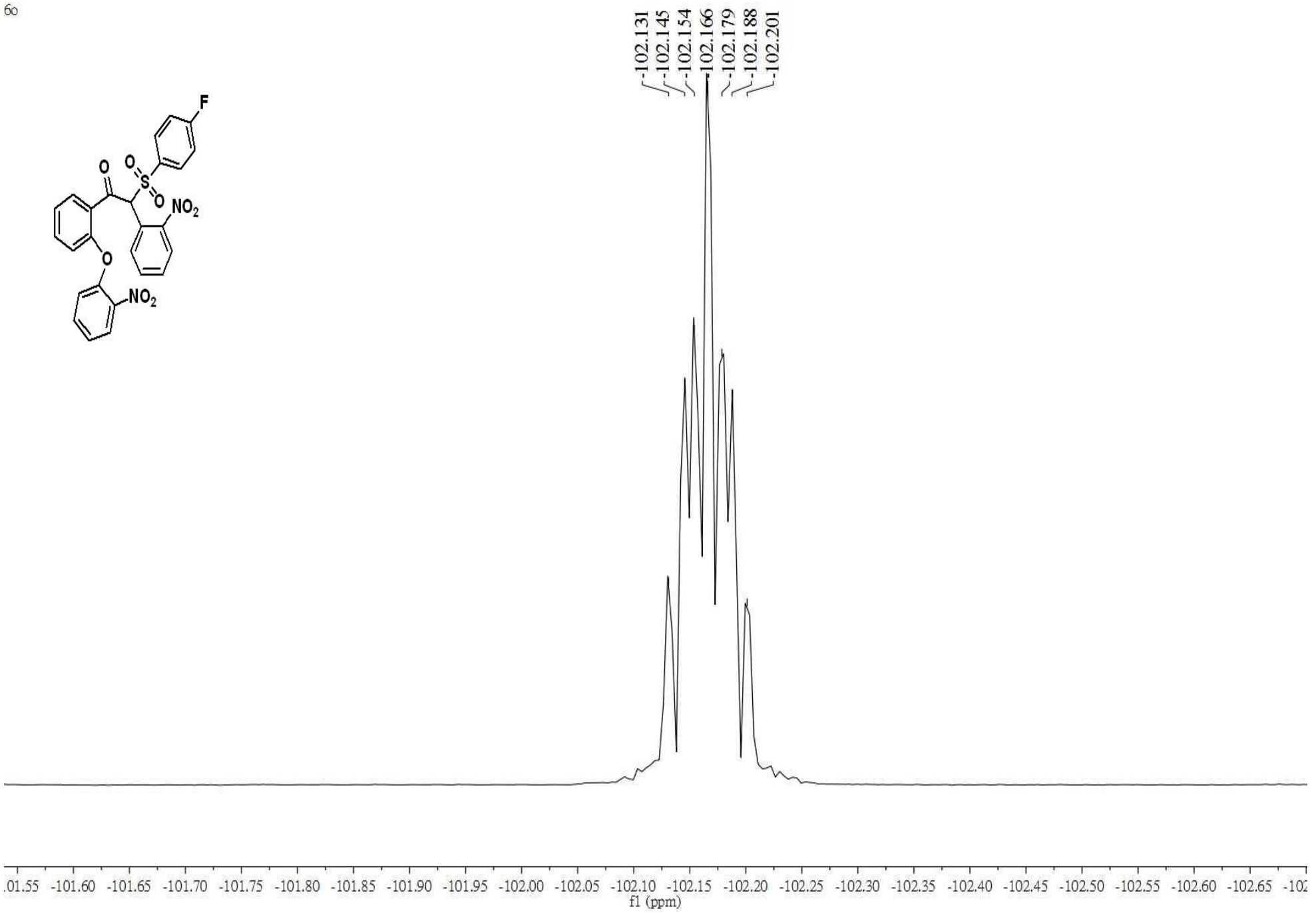




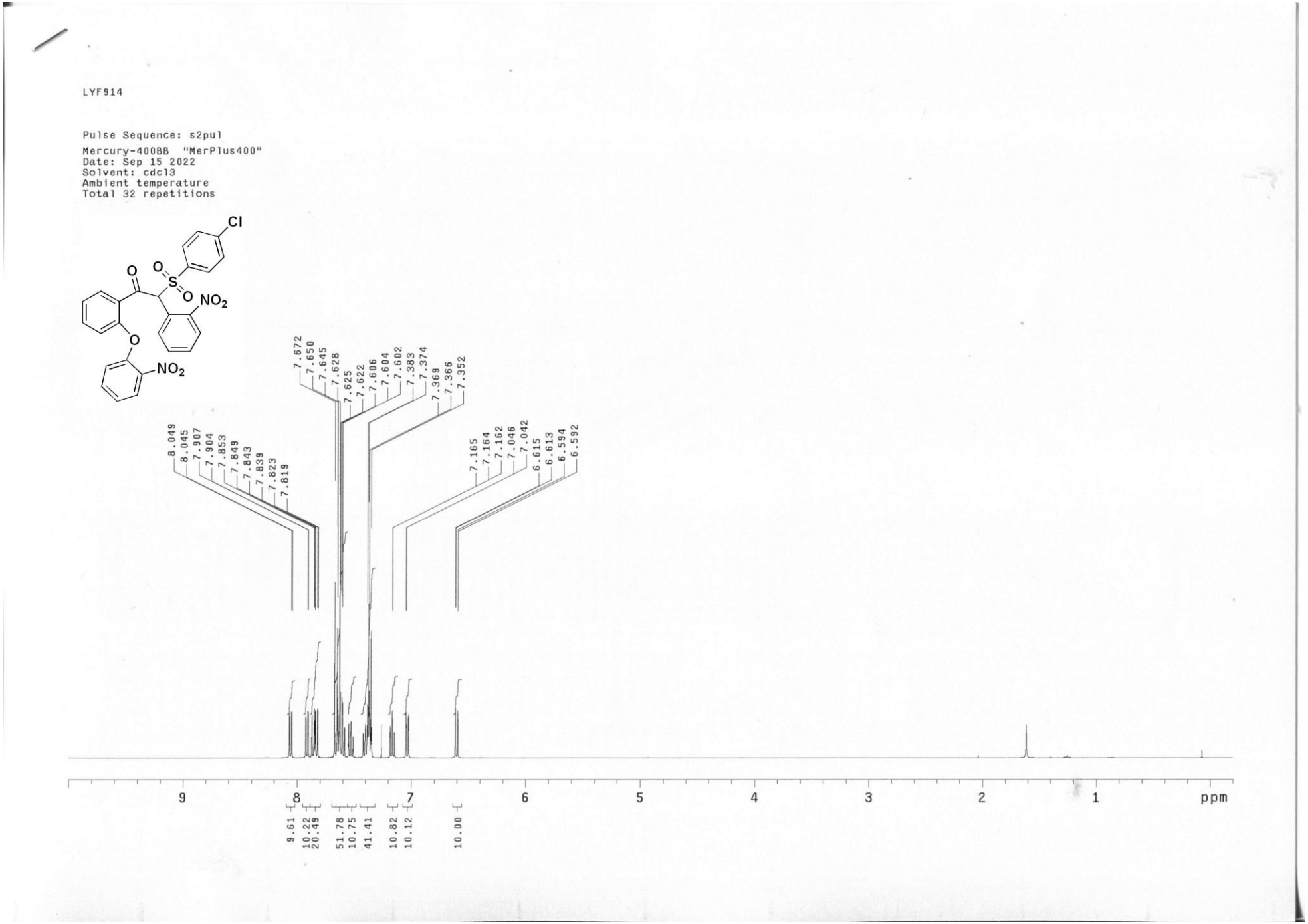
Compound 6o (^{19}F-NMR spectral data-1)



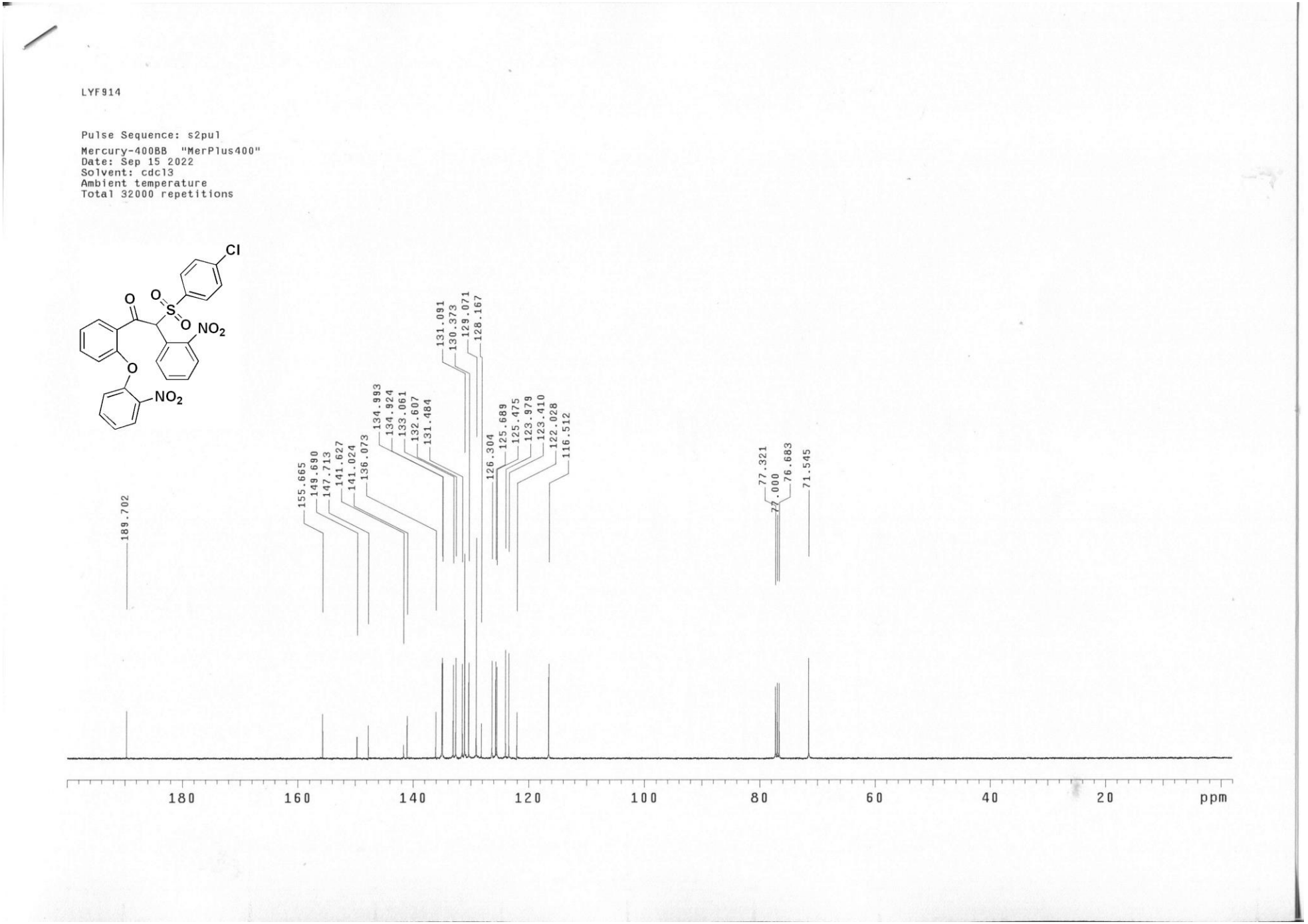
Compound 6o (^{19}F-NMR spectral data-2)



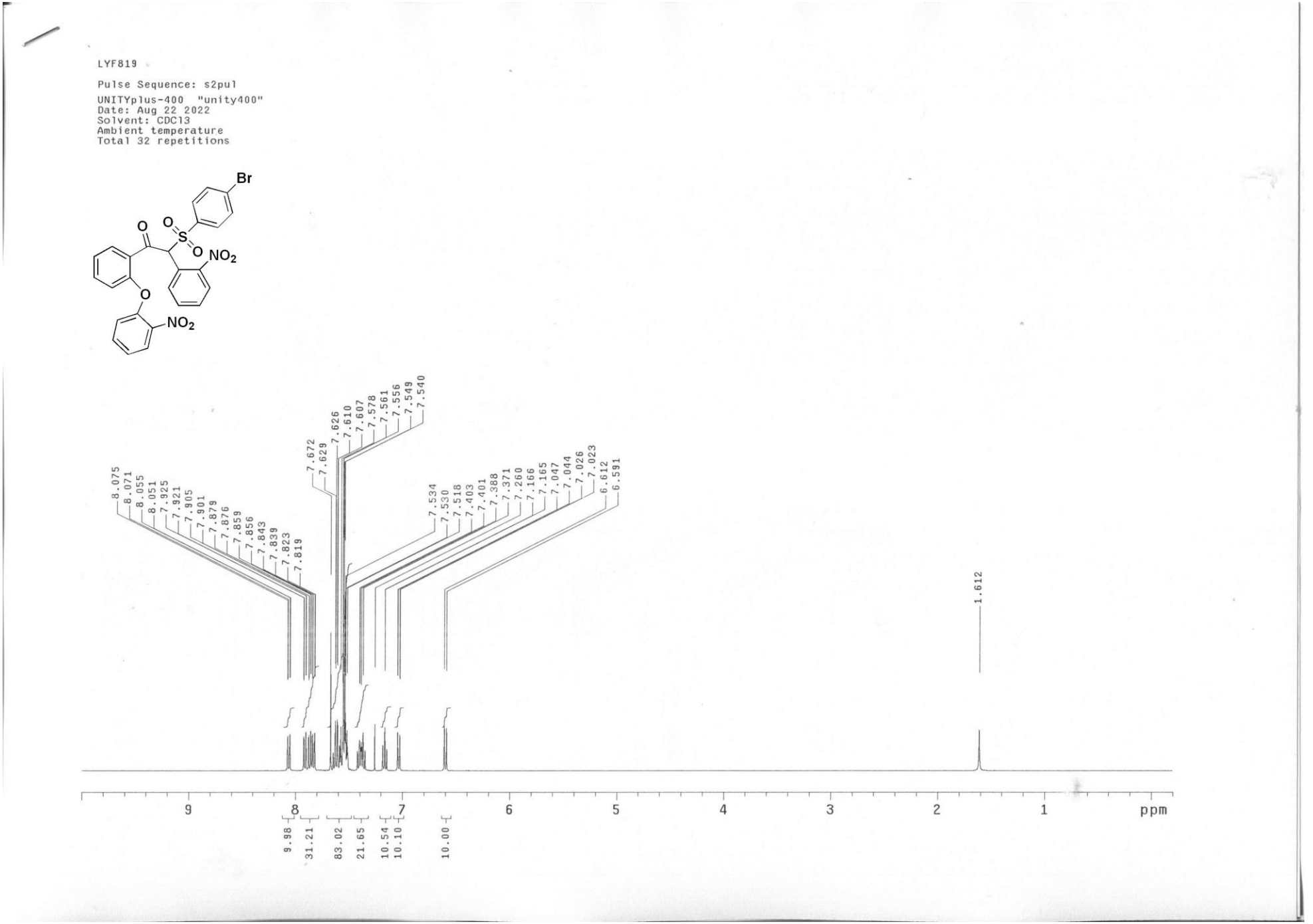
Compound 6p (^{1}H-NMR spectral data)



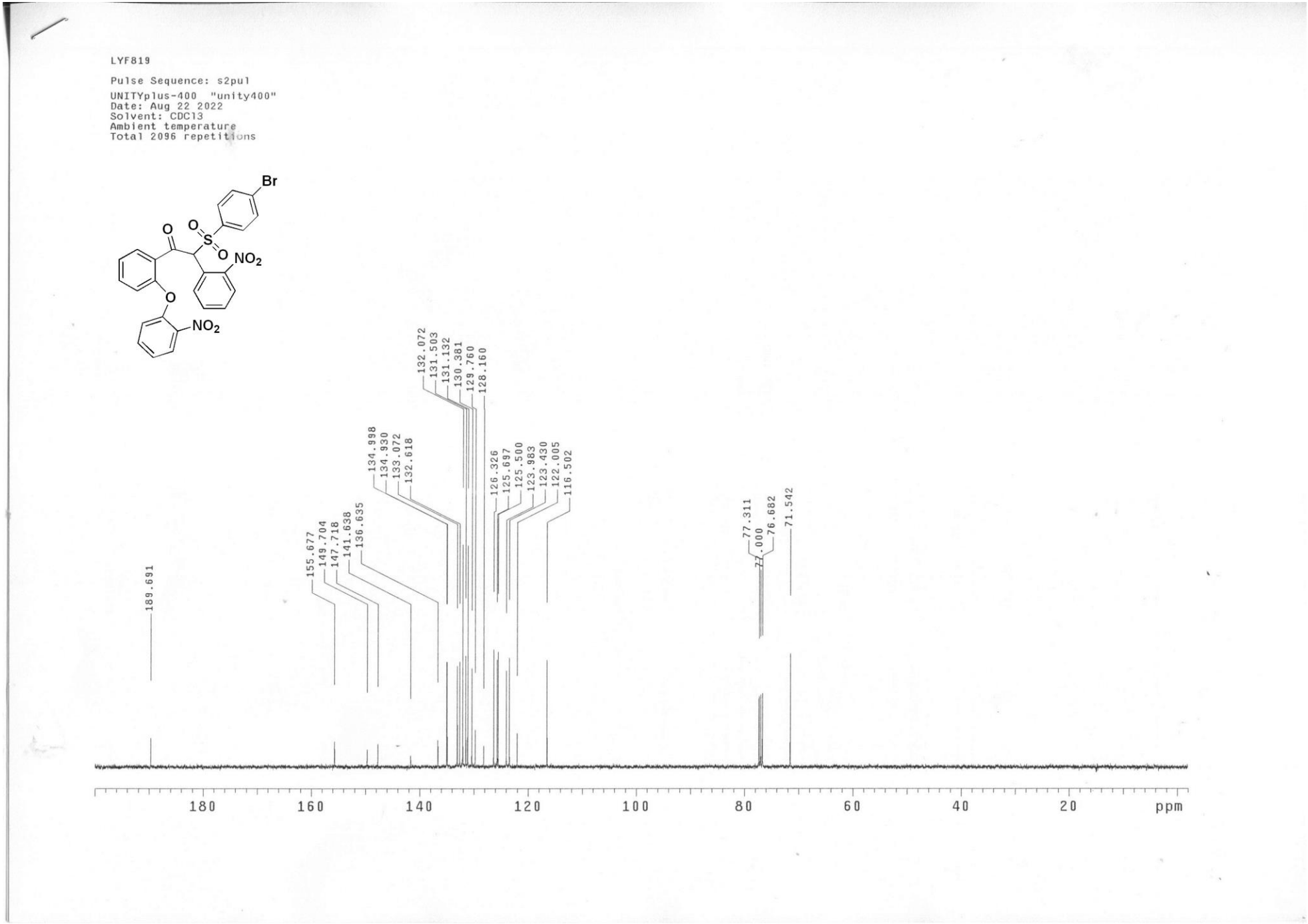
Compound 6p (^{13}C-NMR spectral data)



Compound 6q (^{1}H-NMR spectral data)



Compound 6q (^{13}C-NMR spectral data)

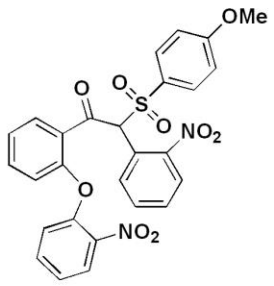


Compound 6r (^{1}H-NMR spectral data)

LYF906

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 13 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

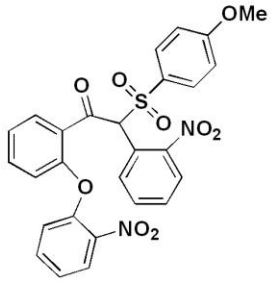


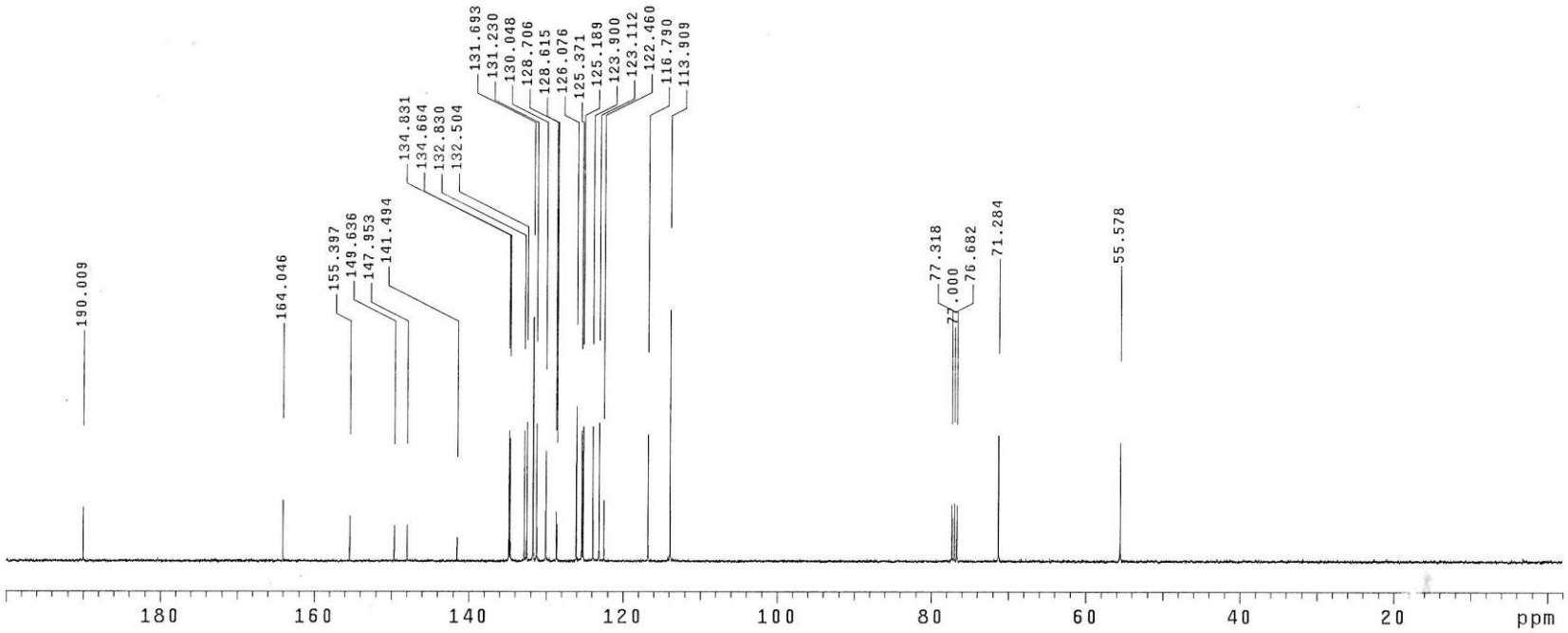
Compound 6r (^{13}C-NMR spectral data)

LYF906

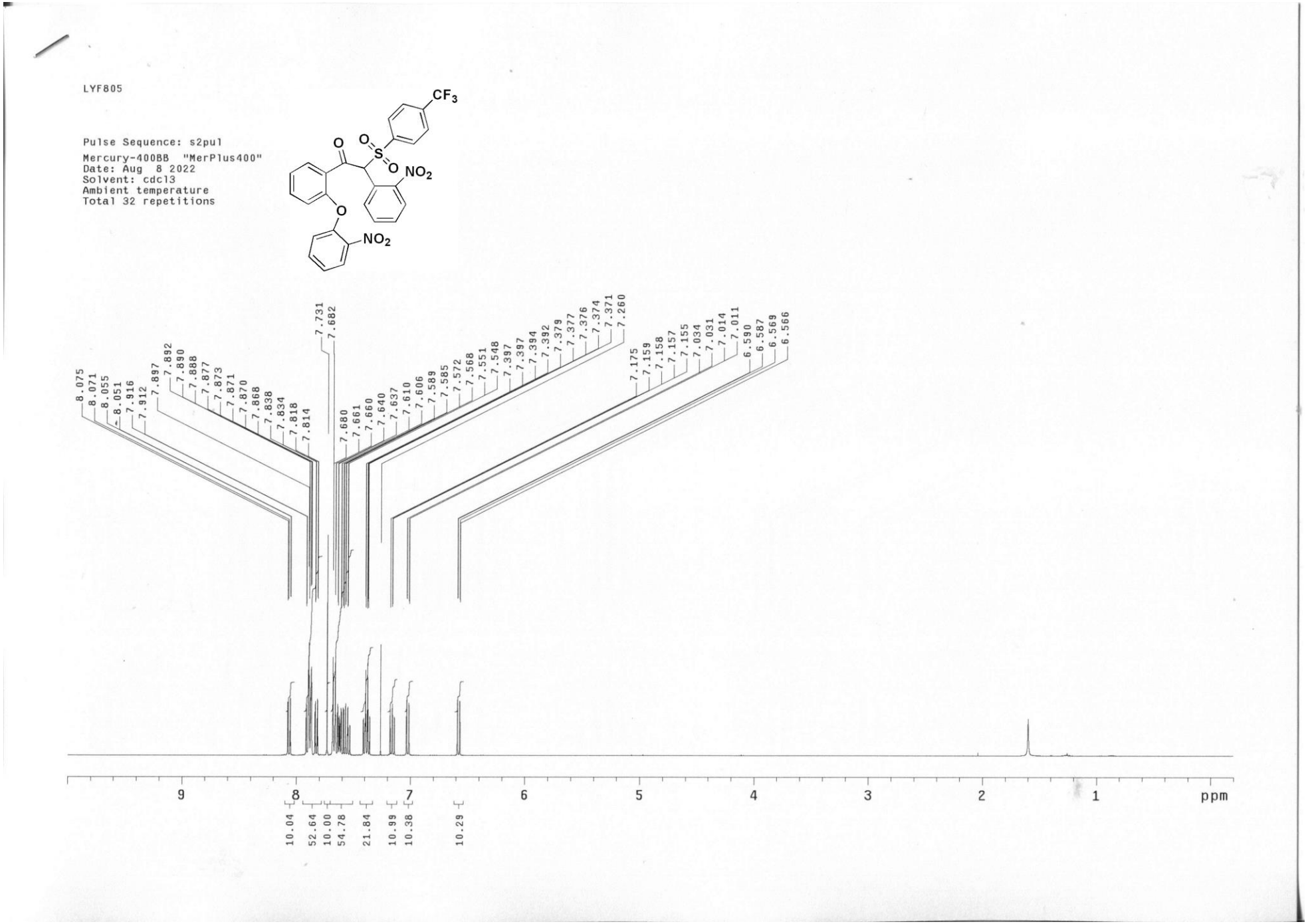
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 13 2022
Solvent: CDCl3
Ambient temperature
Total 640 repetitions

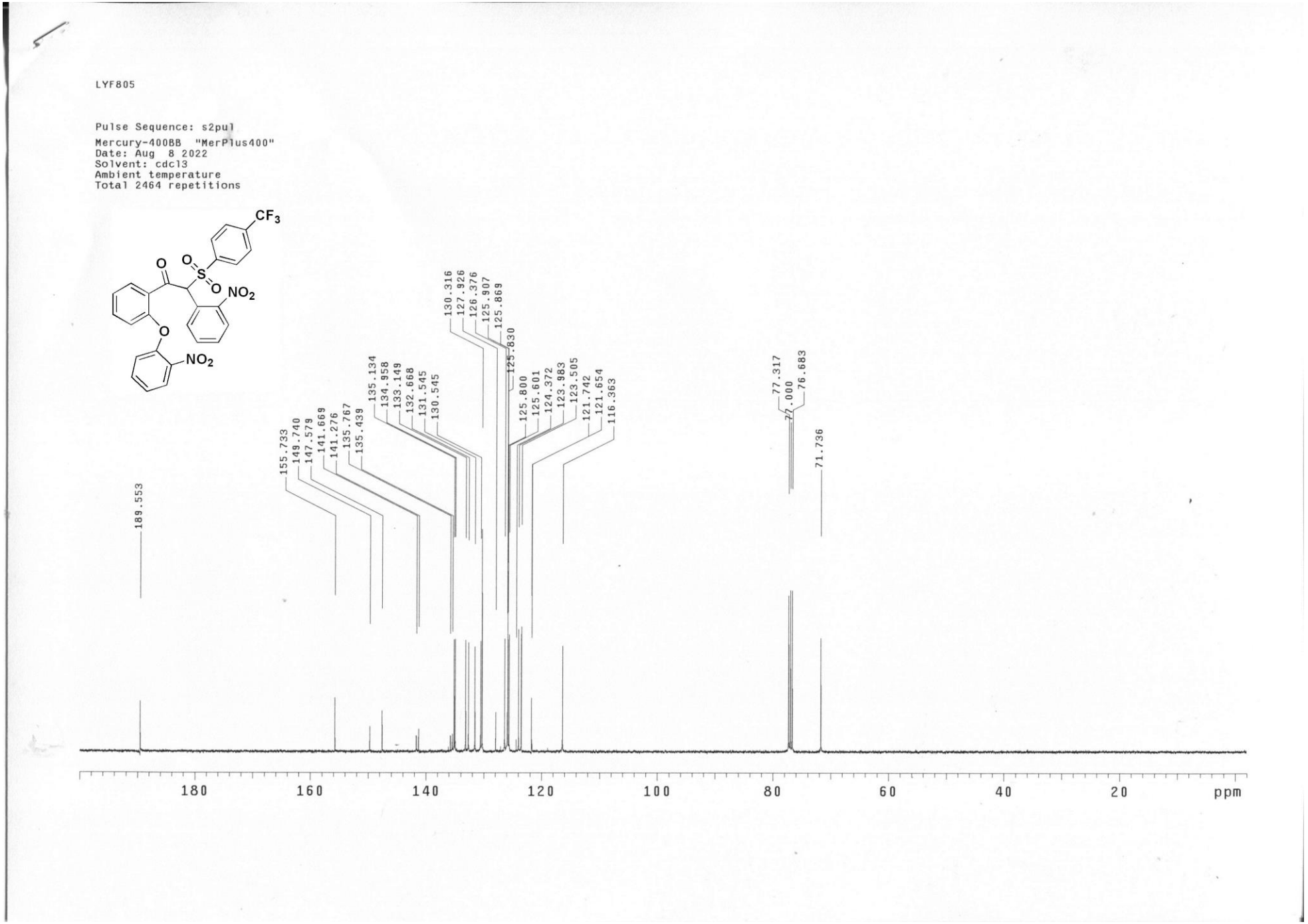




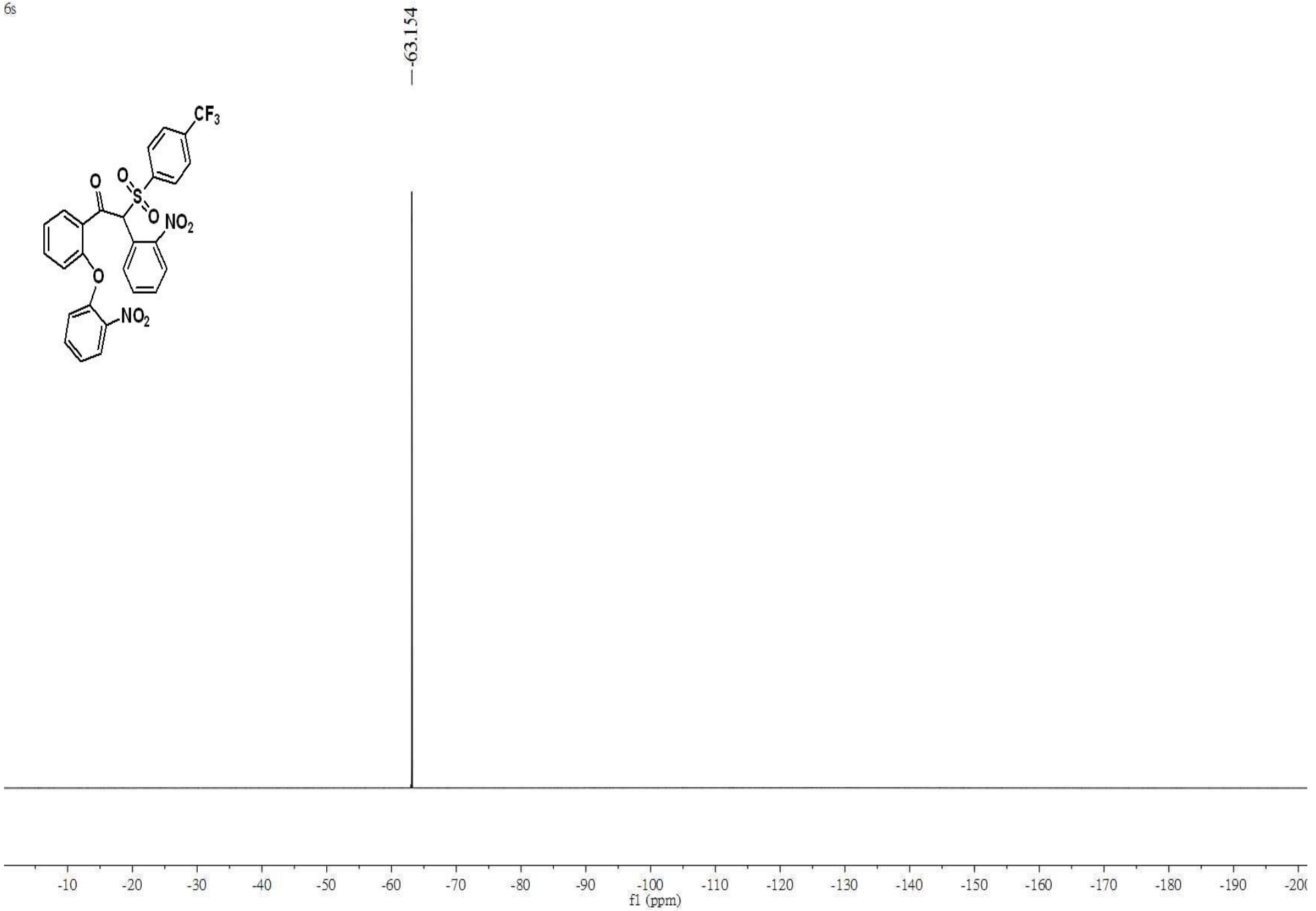
Compound 6s (^{1}H-NMR spectral data)



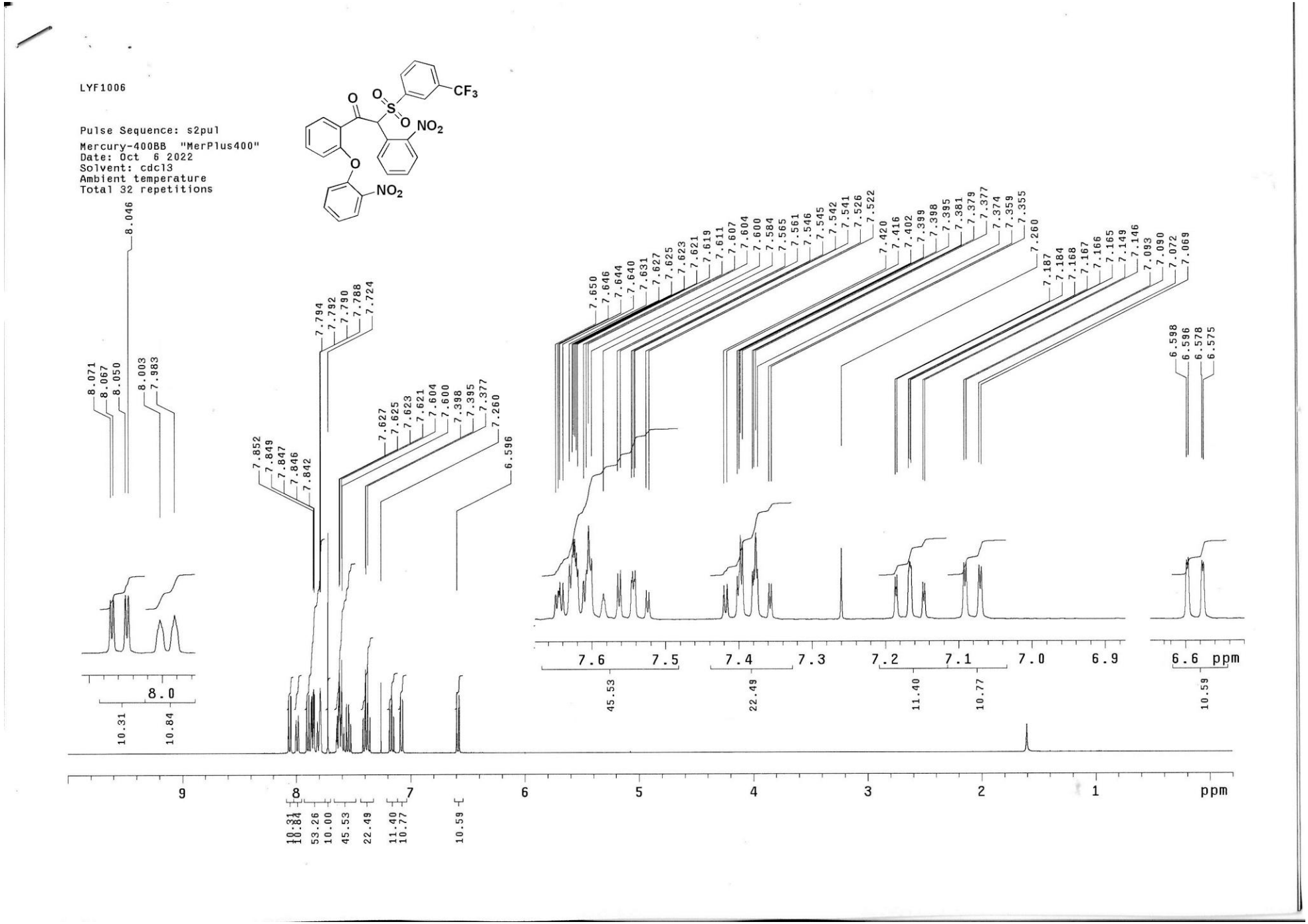
Compound 6s (^{13}C-NMR spectral data)



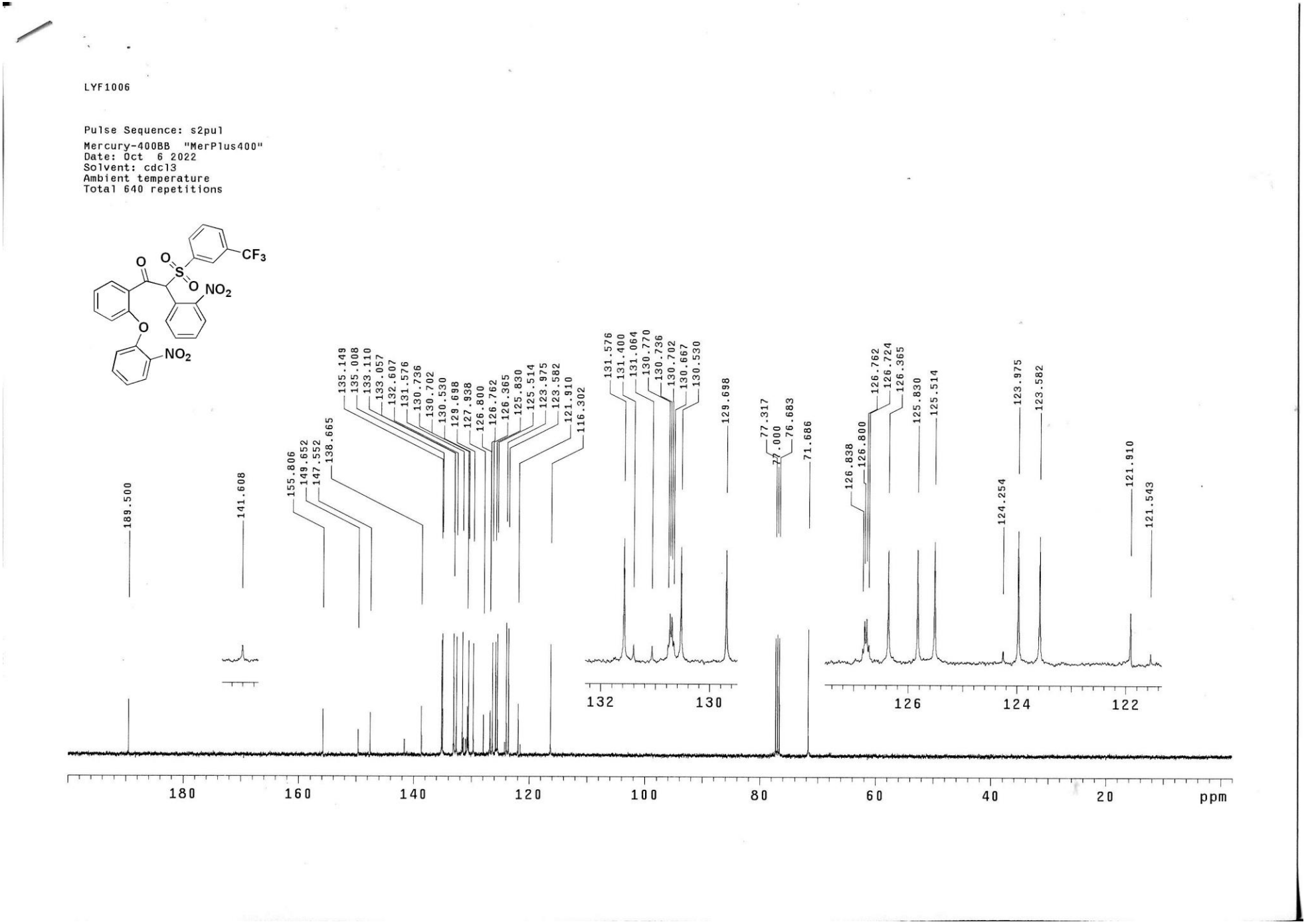
Compound 6s (^{19}F-NMR spectral data)



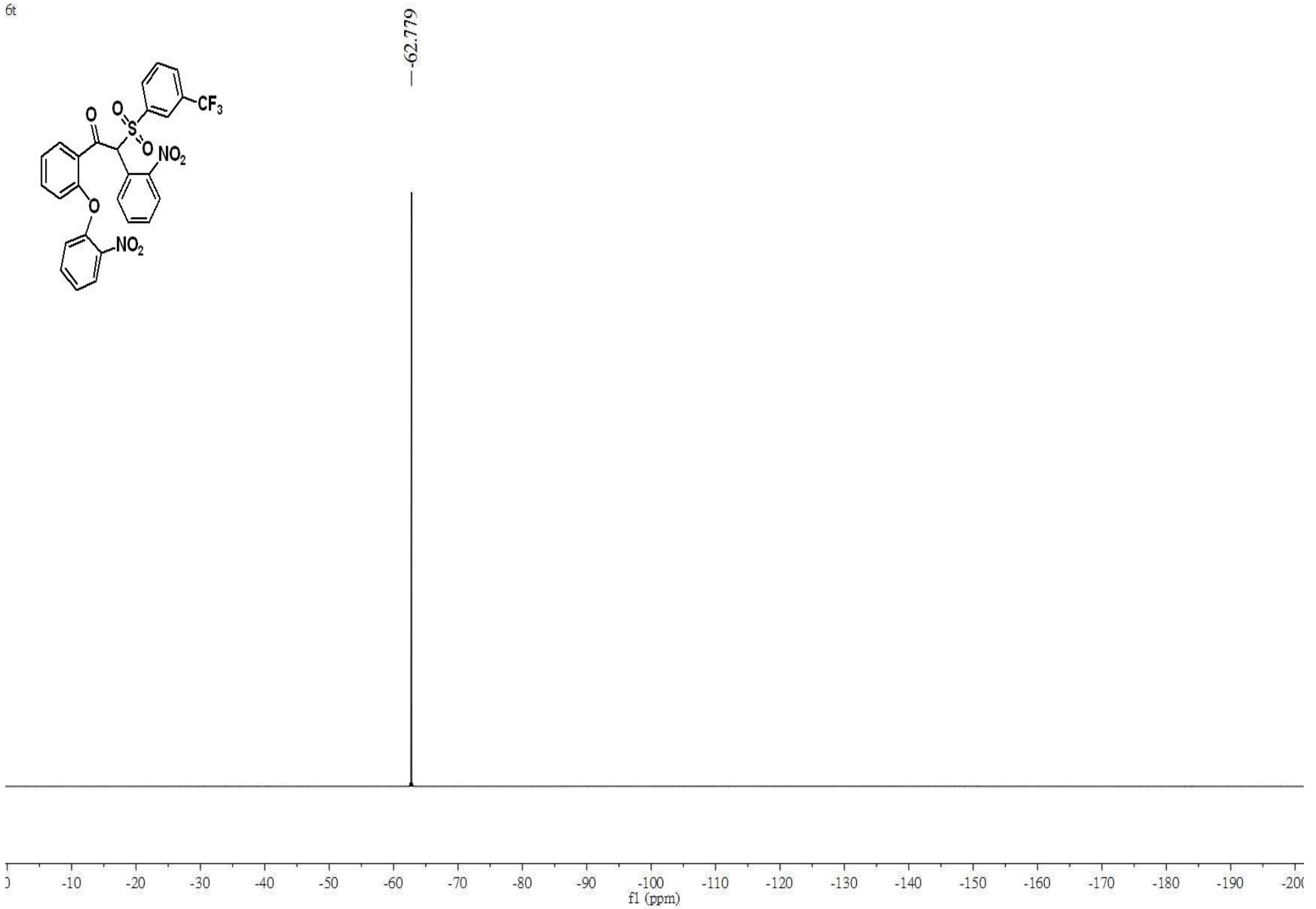
Compound 6t (^{1}H-NMR spectral data)



Compound 6t (^{13}C-NMR spectral data)



Compound 6t (^{19}F-NMR spectral data)

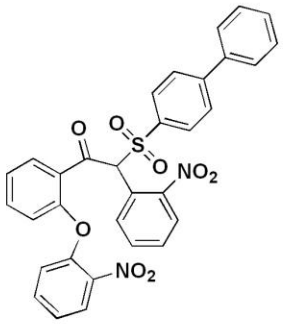


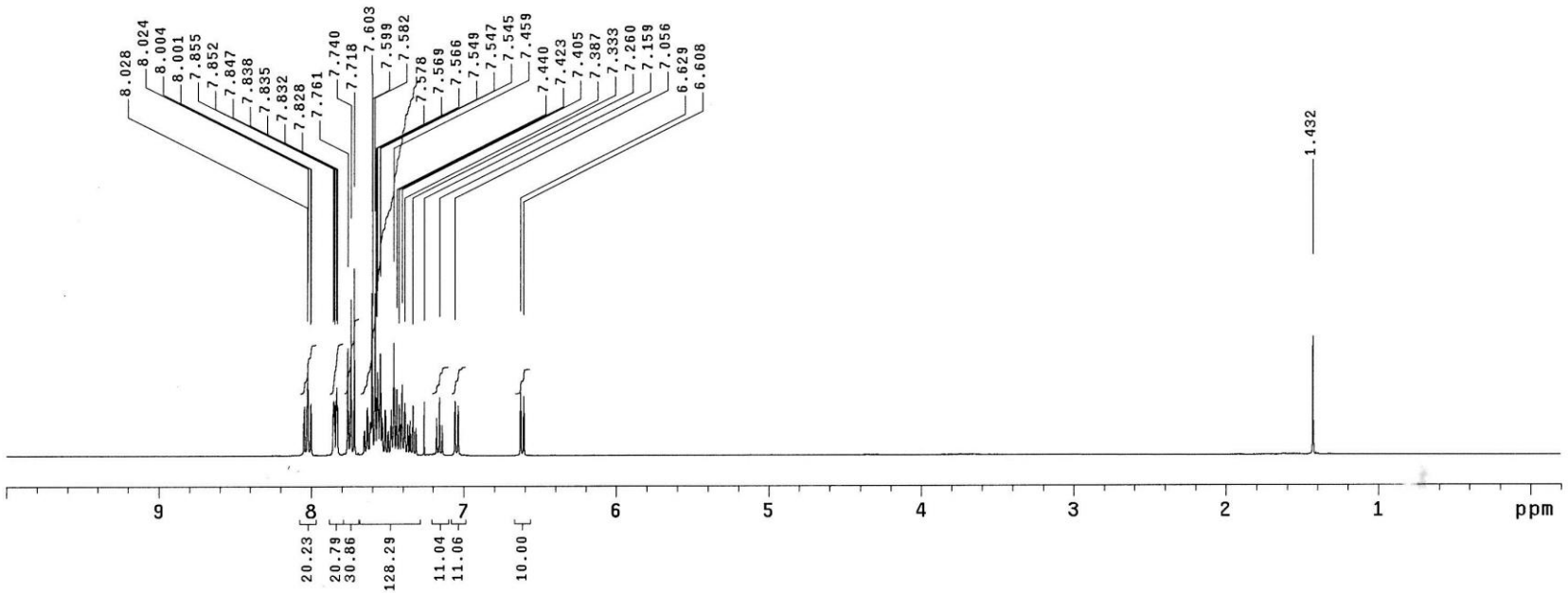
Compound 6u (^{1}H-NMR spectral data)

LYF915

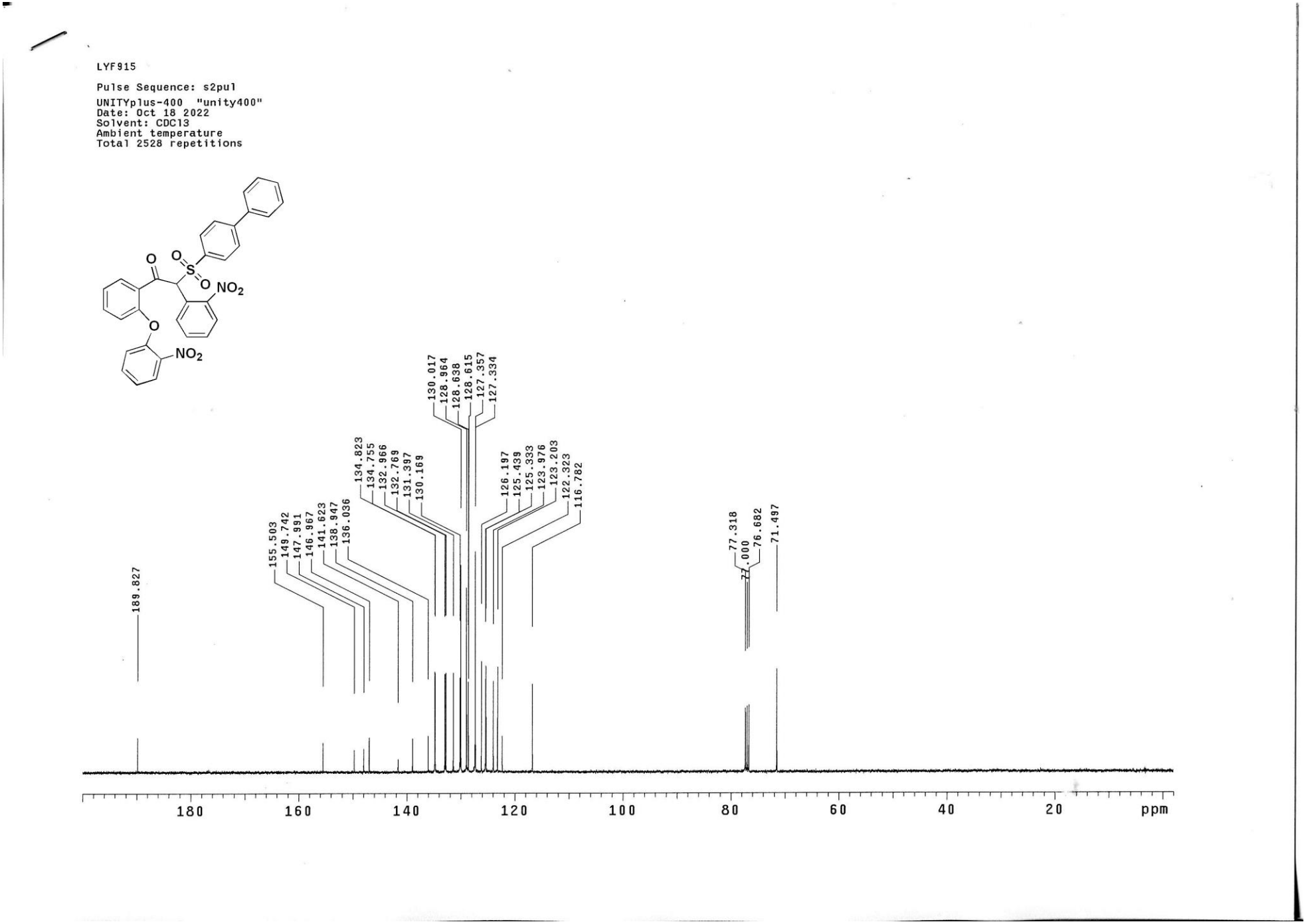
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 18 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

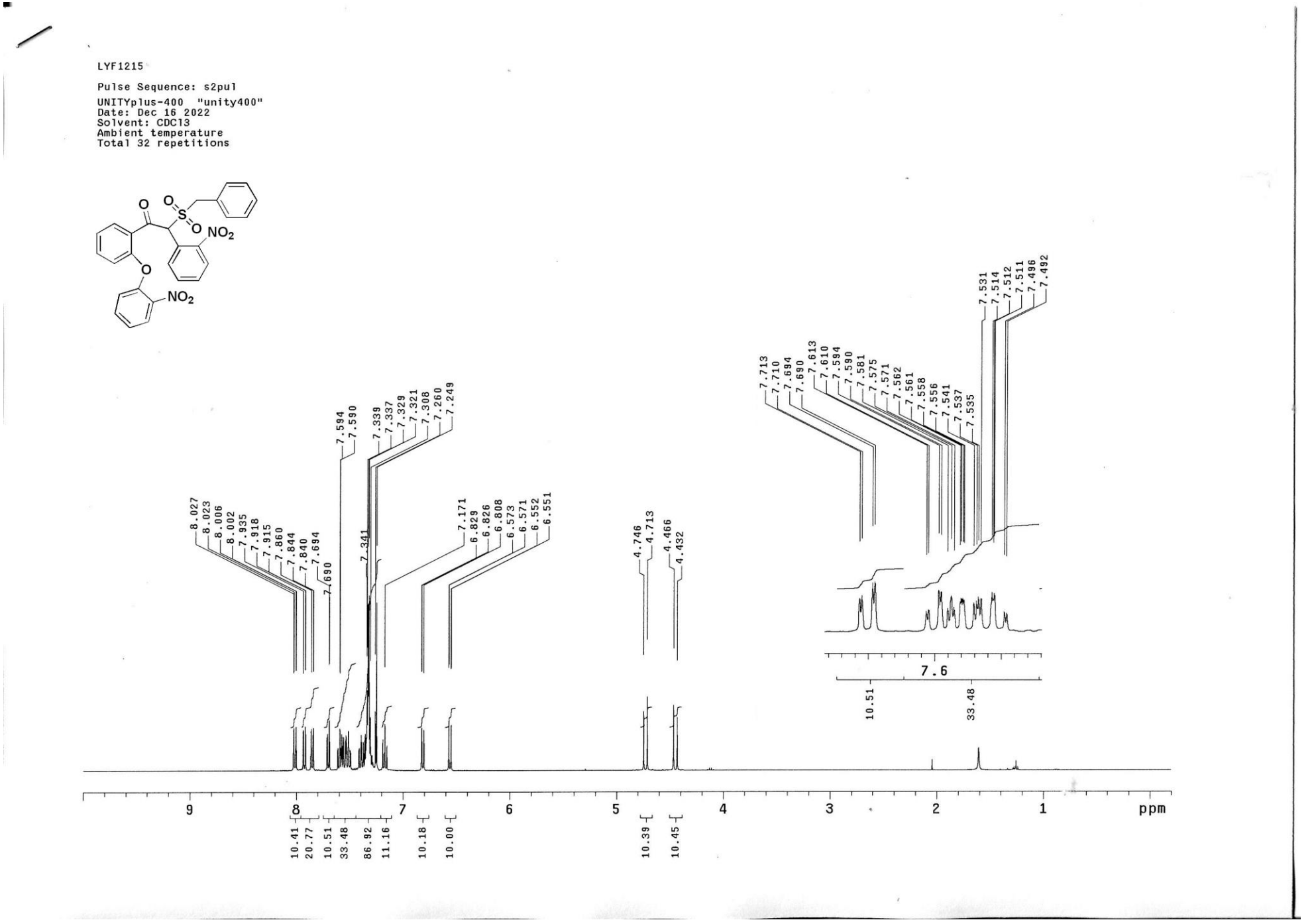




Compound 6u (^{13}C-NMR spectral data)



Compound 6v (^{1}H-NMR spectral data)

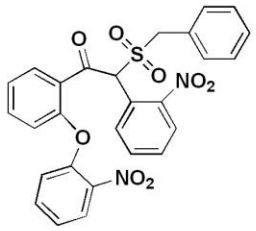


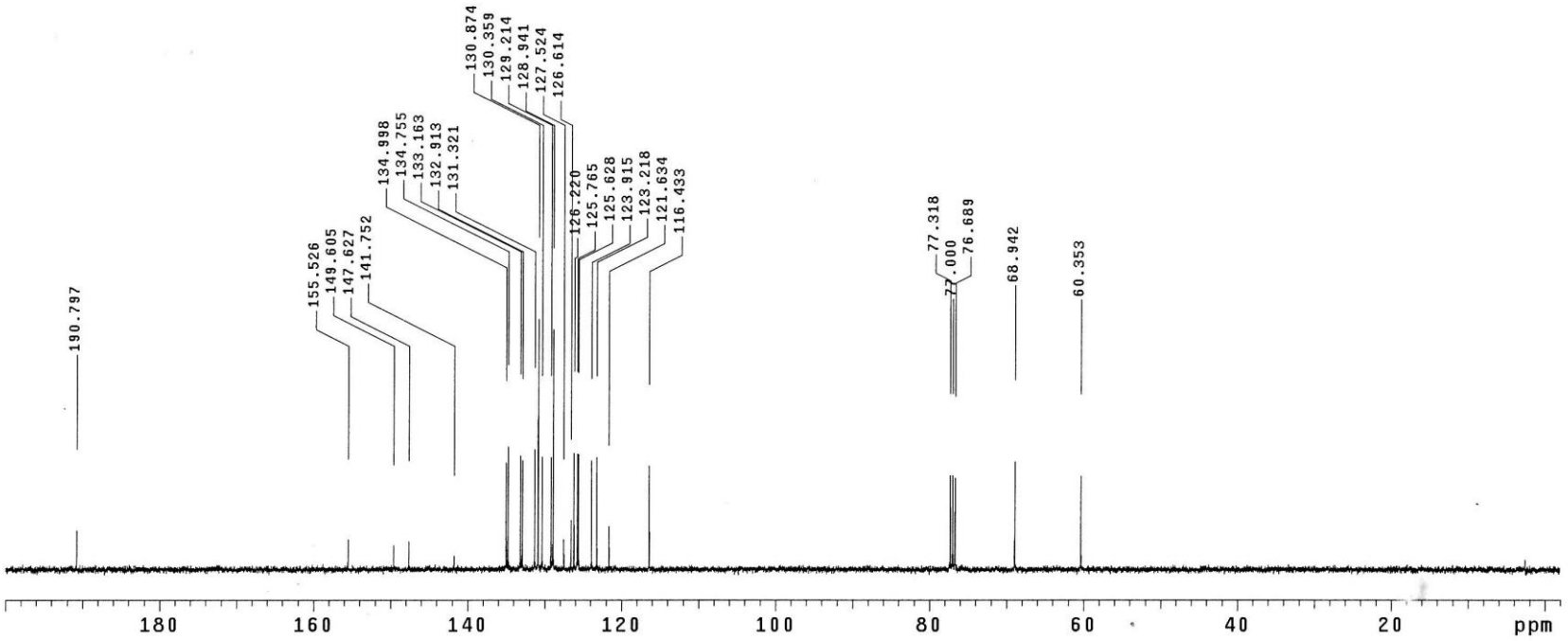
Compound 6v (^{13}C-NMR spectral data)

LYF1215

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 16 2022
Solvent: CDCl3
Ambient temperature
Total 1056 repetitions



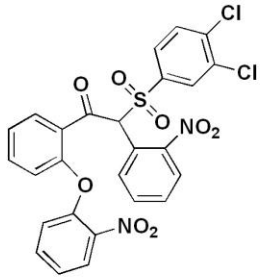


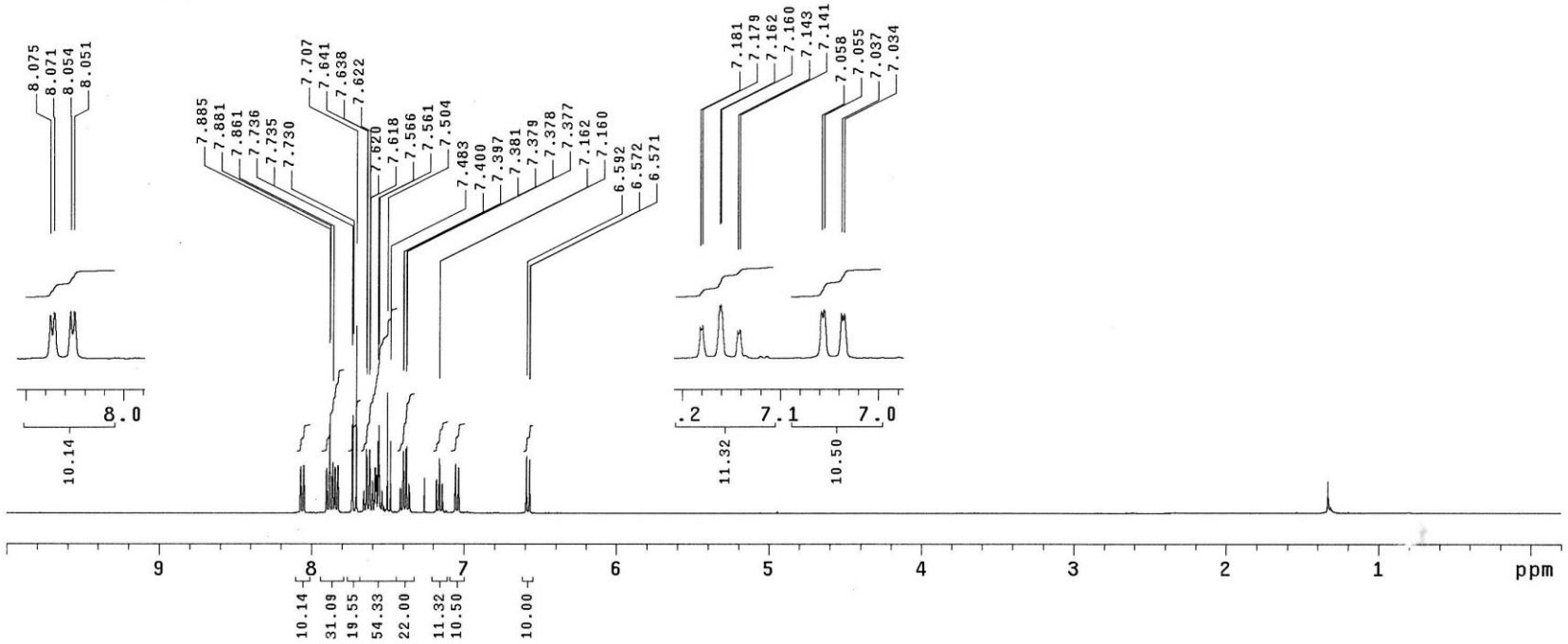
Compound 6w (^{1}H-NMR spectral data)

LYF1101

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Nov 2 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



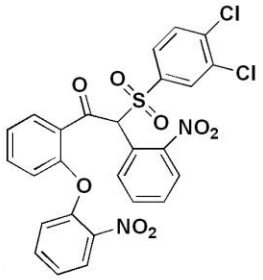


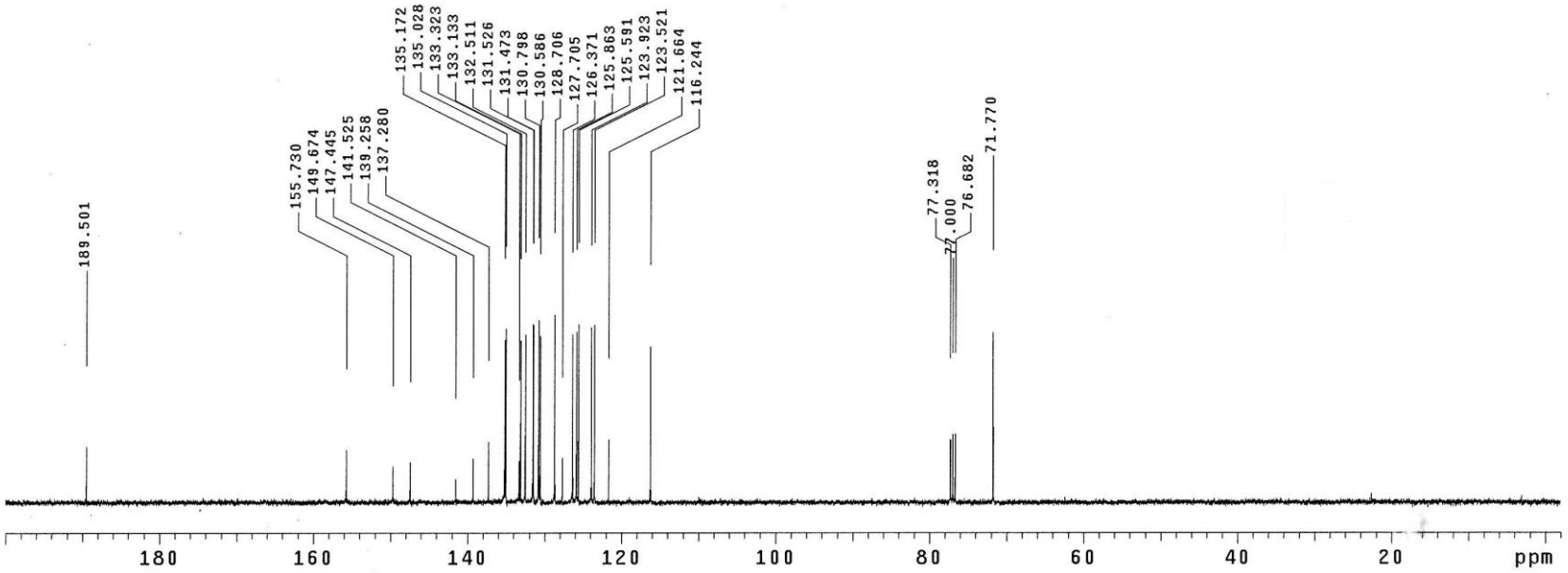
Compound 6w (^{13}C-NMR spectral data)

LYF1101

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Nov 2 2022
Solvent: CDCl3
Ambient temperature
Total 720 repetitions



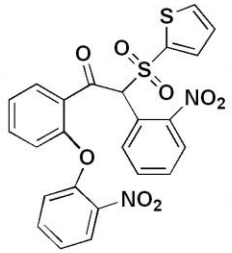


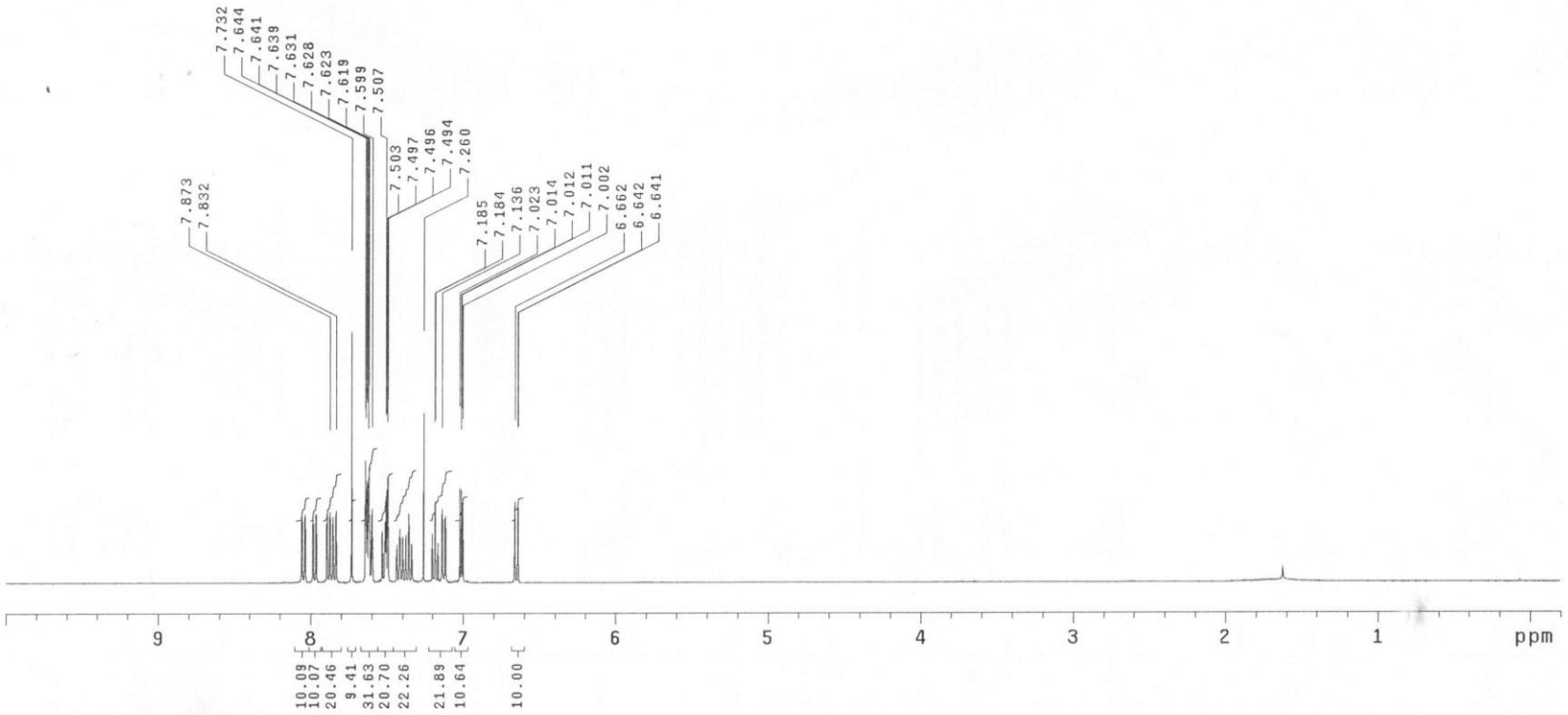
Compound 6x (^{1}H-NMR spectral data)

LYF726

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 27 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



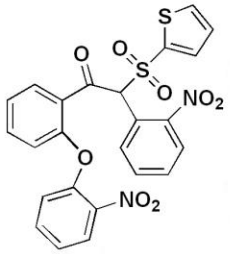


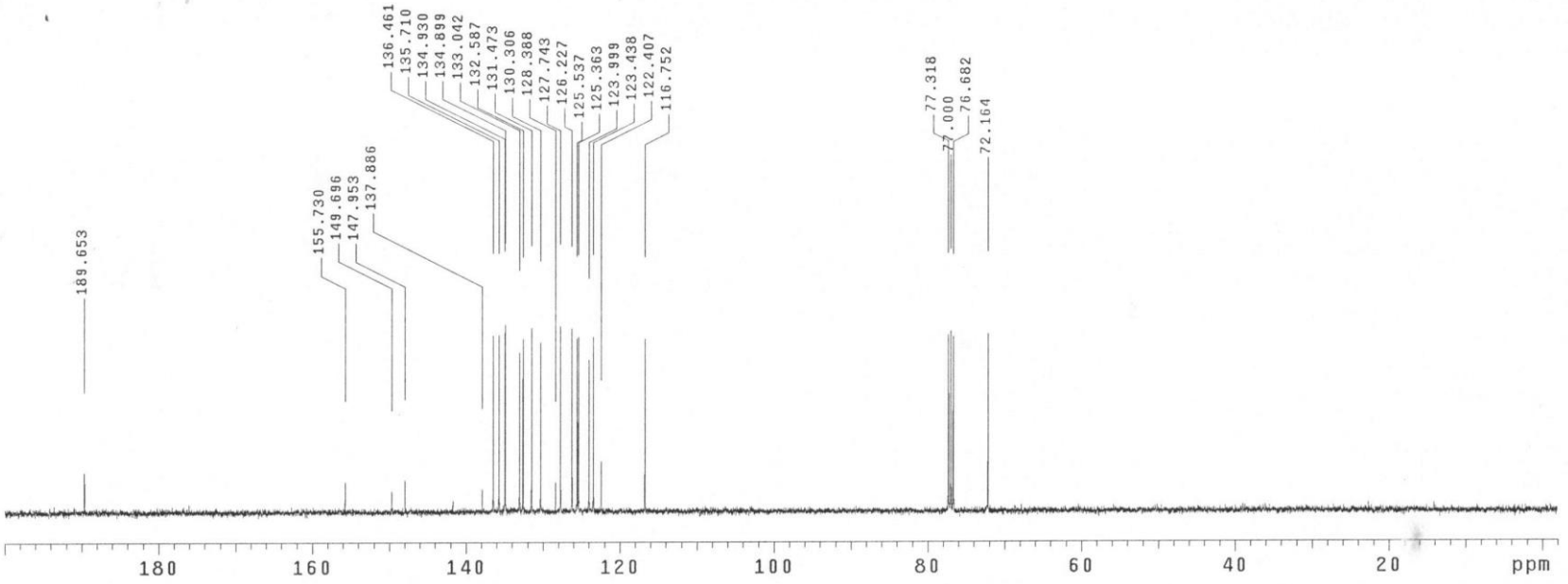
Compound 6x (^{13}C-NMR spectral data)

LYF726

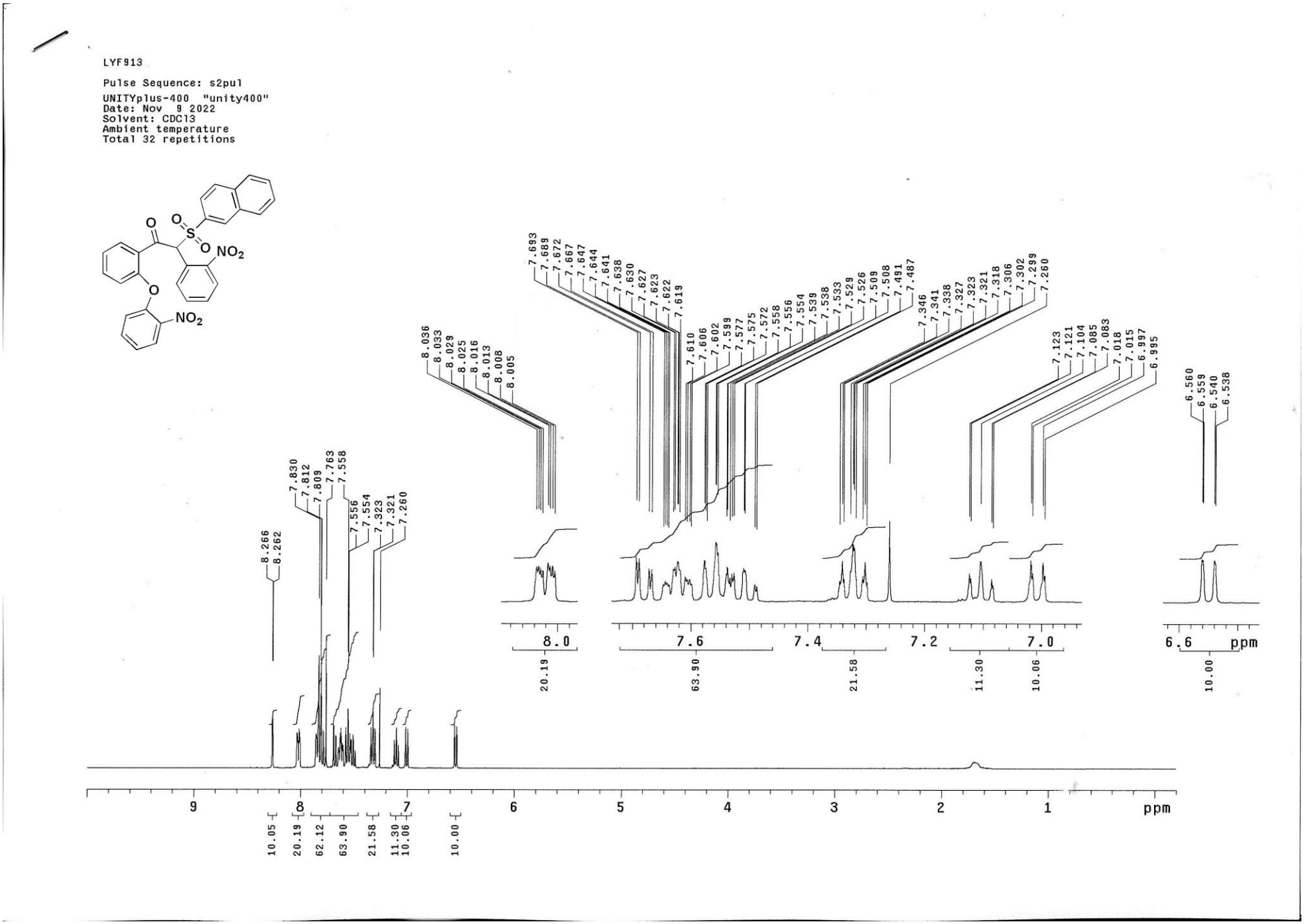
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 27 2022
Solvent: CDCl3
Ambient temperature
Total 3168 repetitions

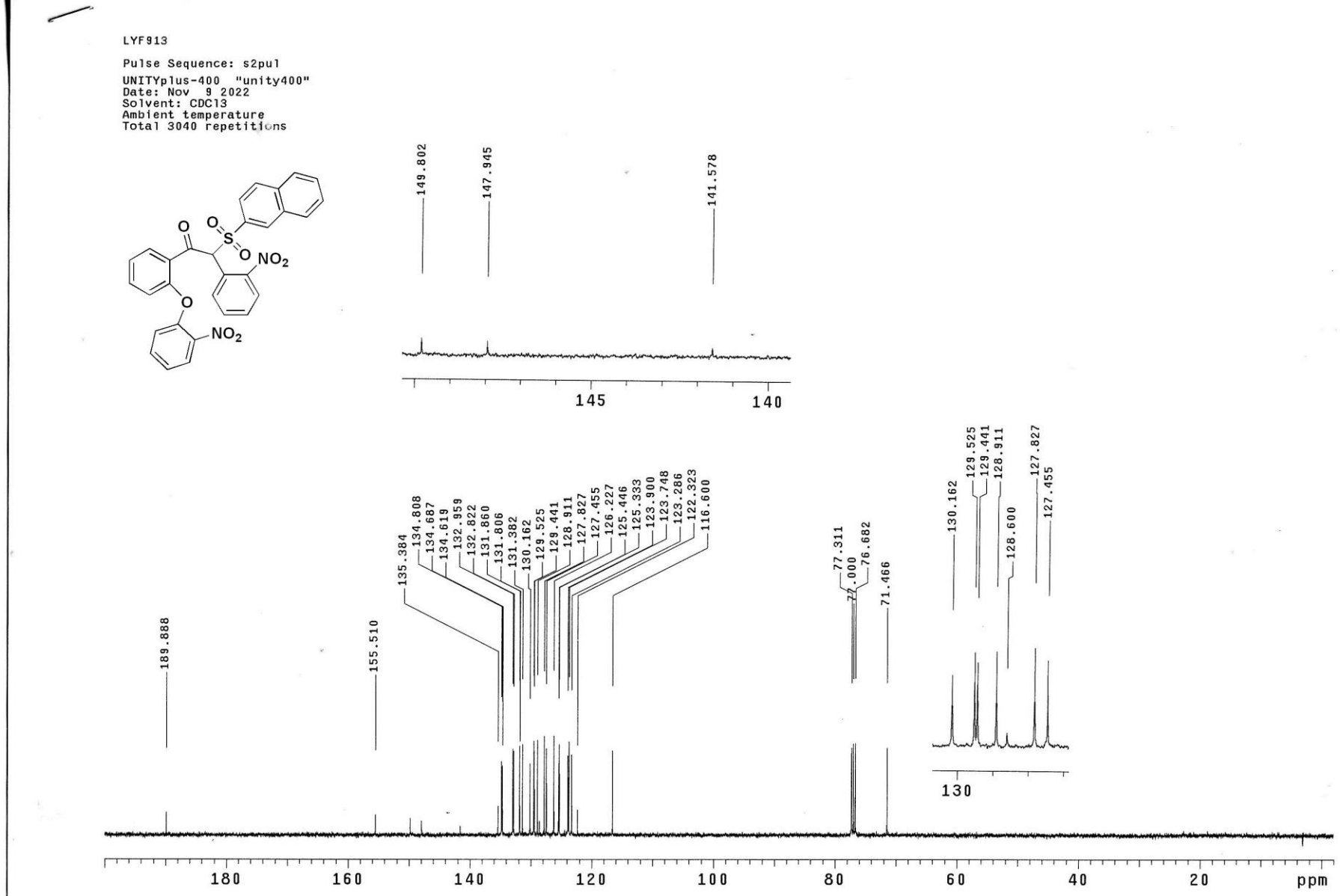




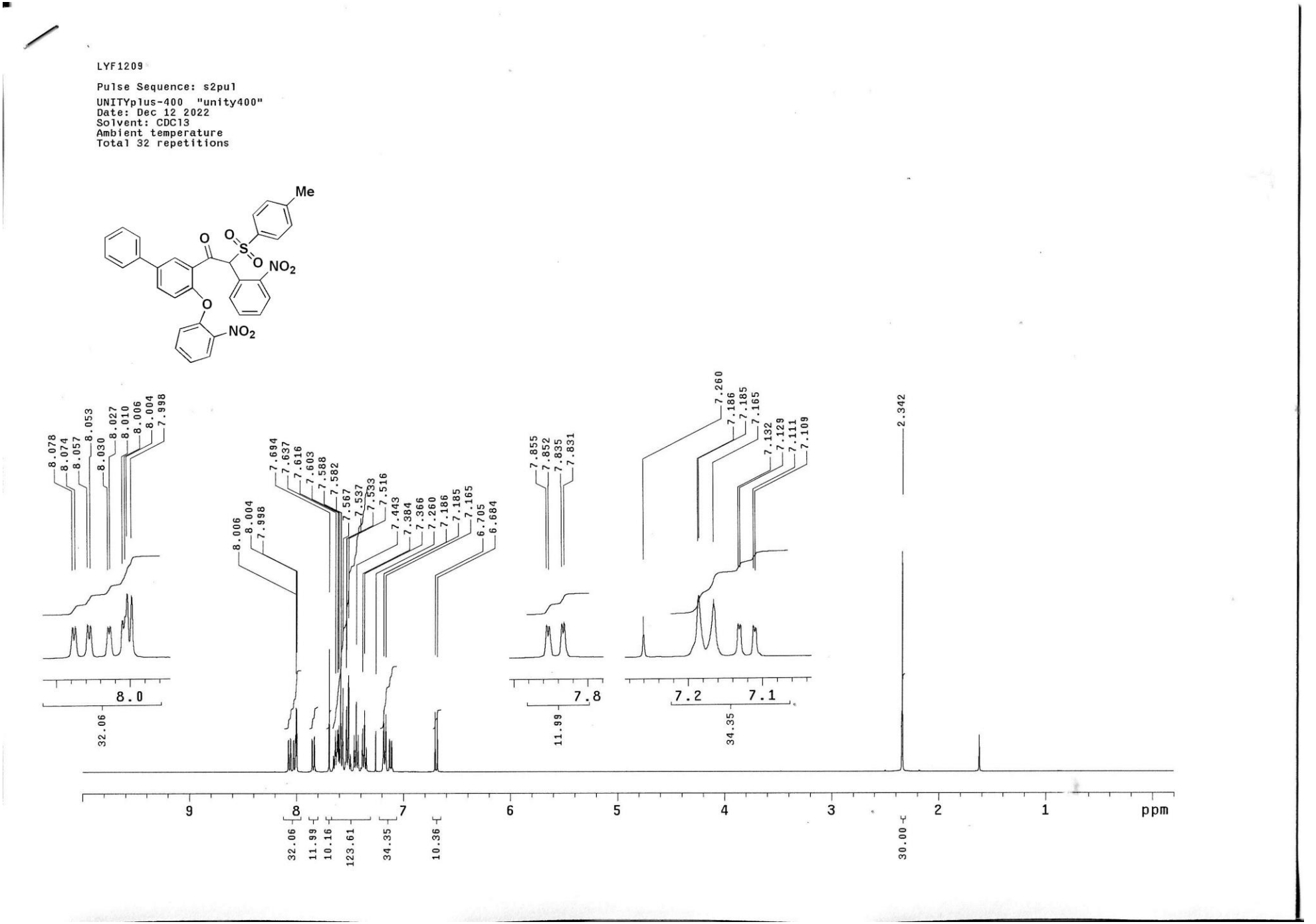
Compound 6y (^{1}H-NMR spectral data)



Compound 6y (^{13}C-NMR spectral data)



Compound 6ab (^{1}H-NMR spectral data)

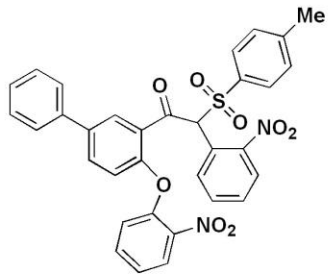


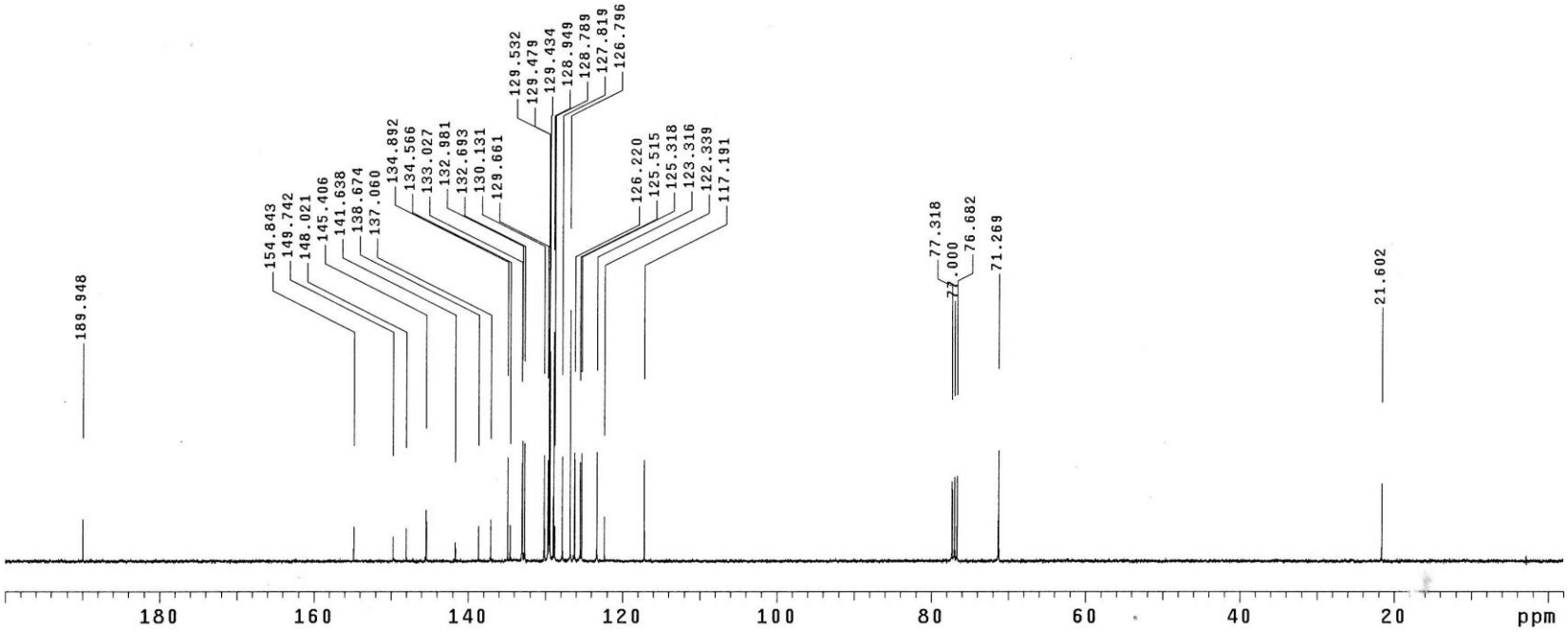
Compound 6ab (^{13}C-NMR spectral data)

LYF1209

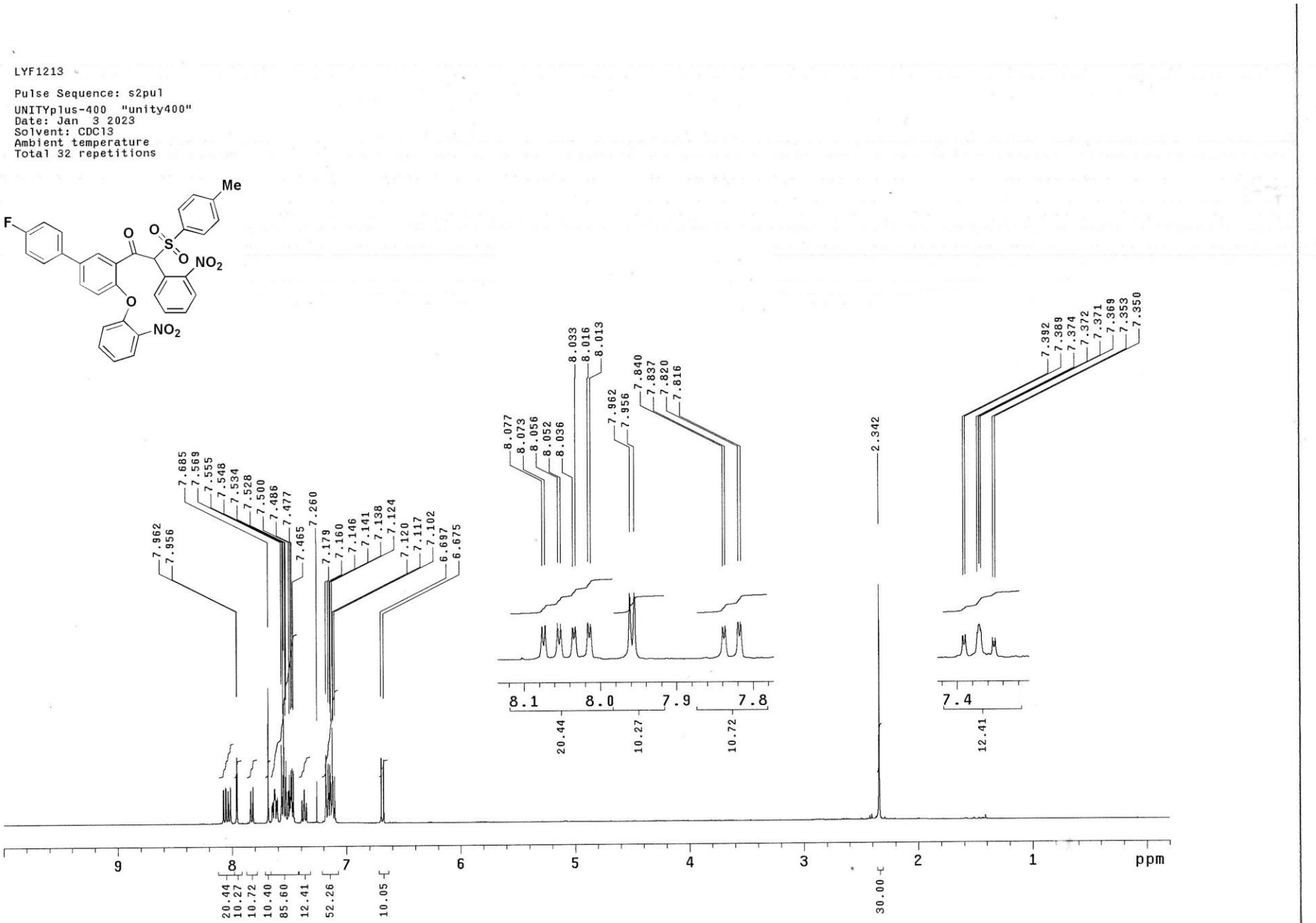
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 12 2022
Solvent: CDCl3
Ambient temperature
Total 3008 repetitions





Compound 6ac (^{1}H-NMR spectral data)

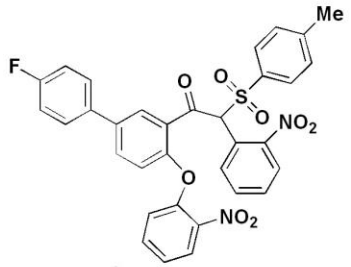


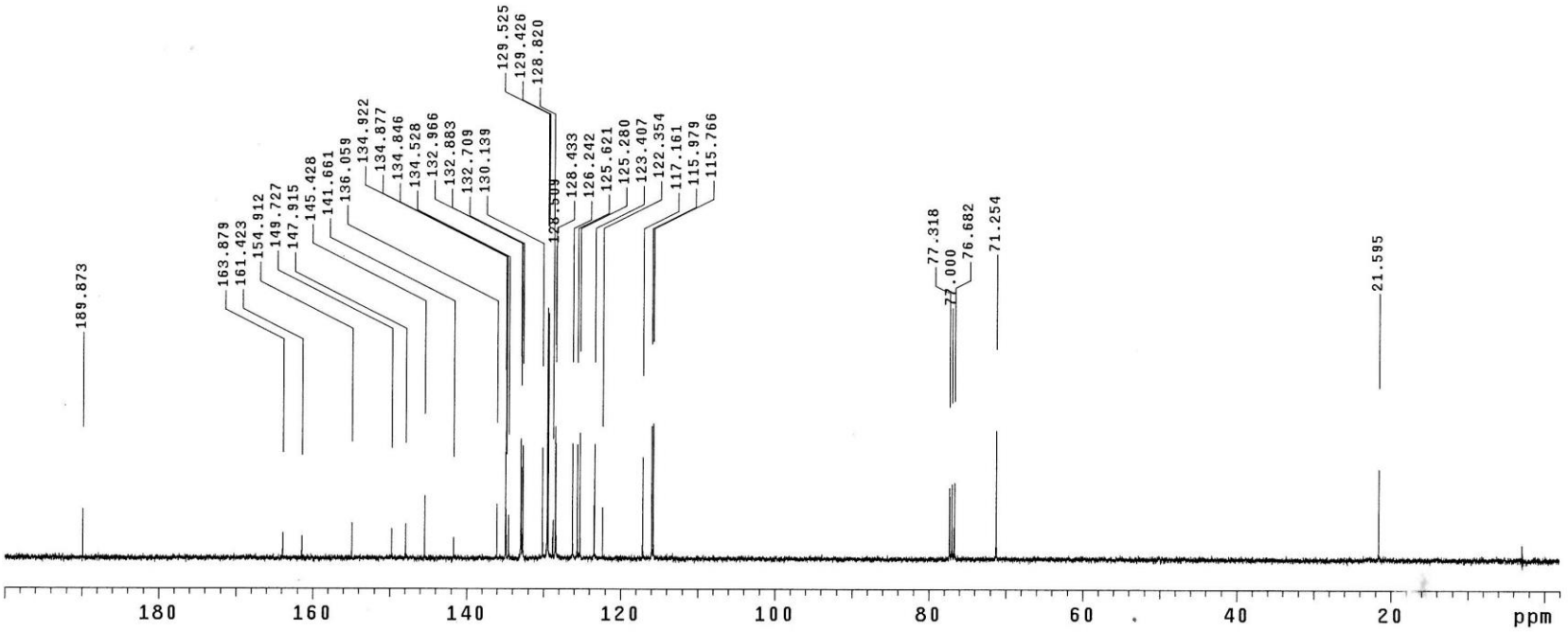
Compound 6ac (^{13}C-NMR spectral data)

LYF1213

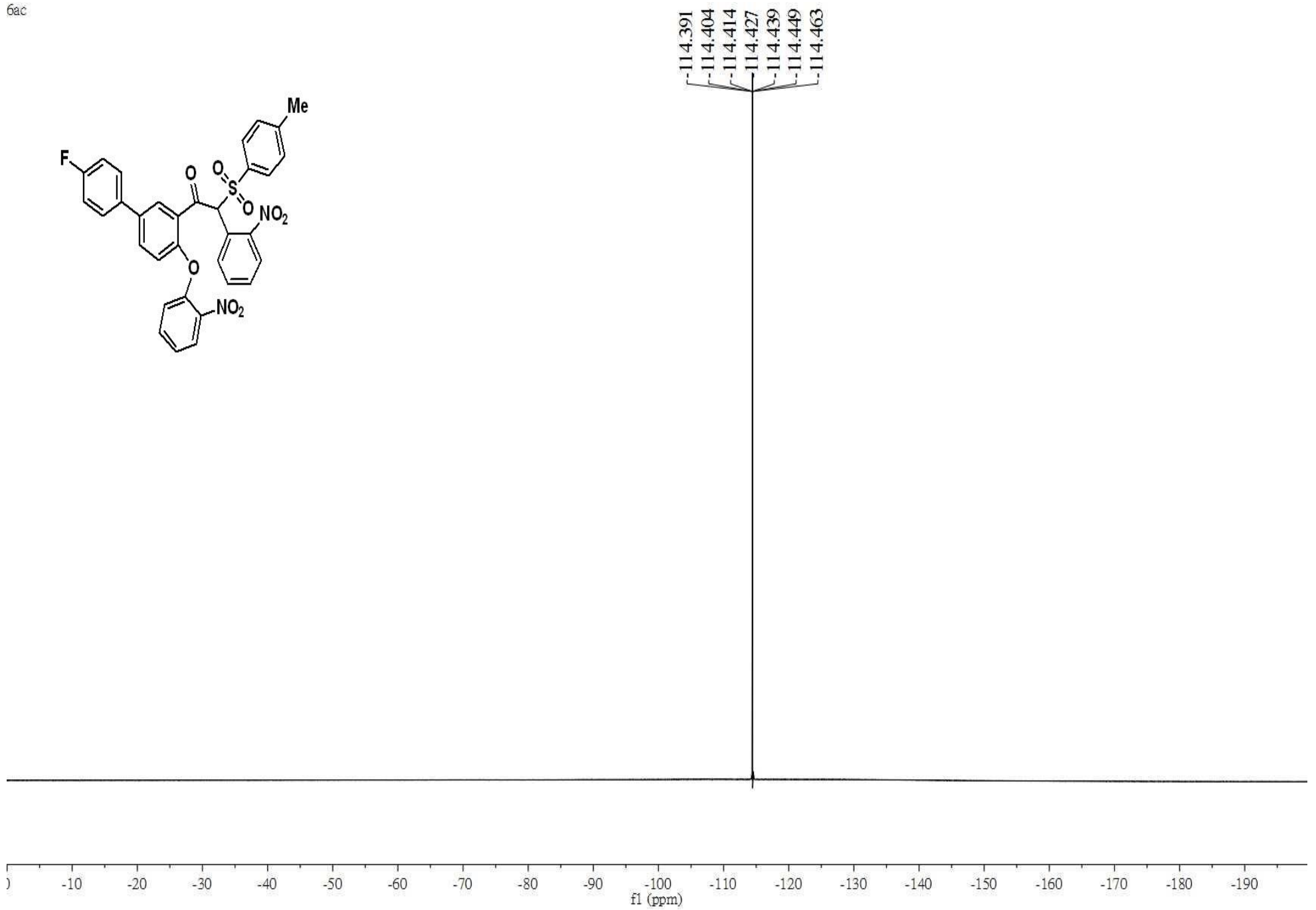
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jan 3 2023
Solvent: CDCl3
Ambient temperature
Total 1072 repetitions

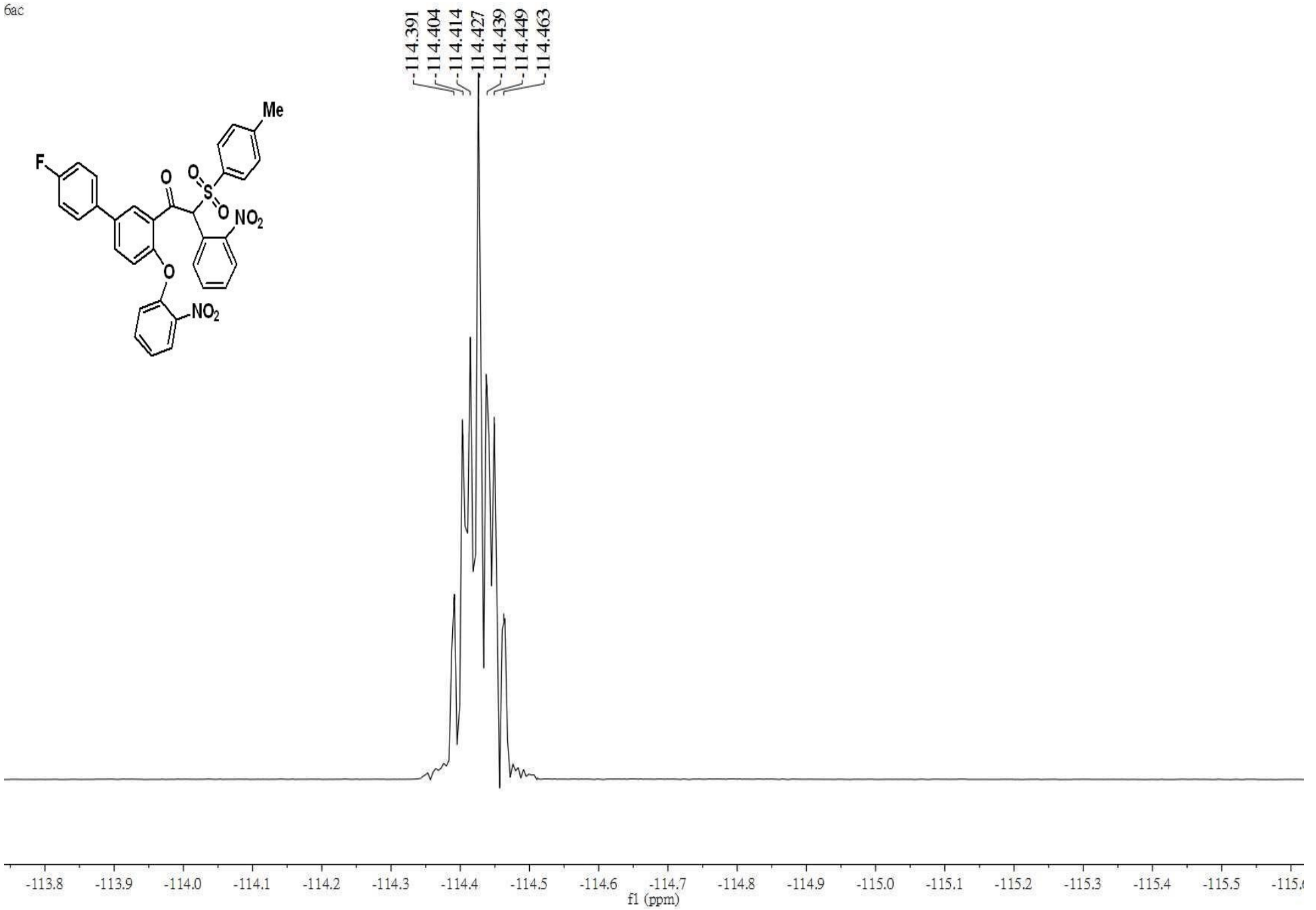




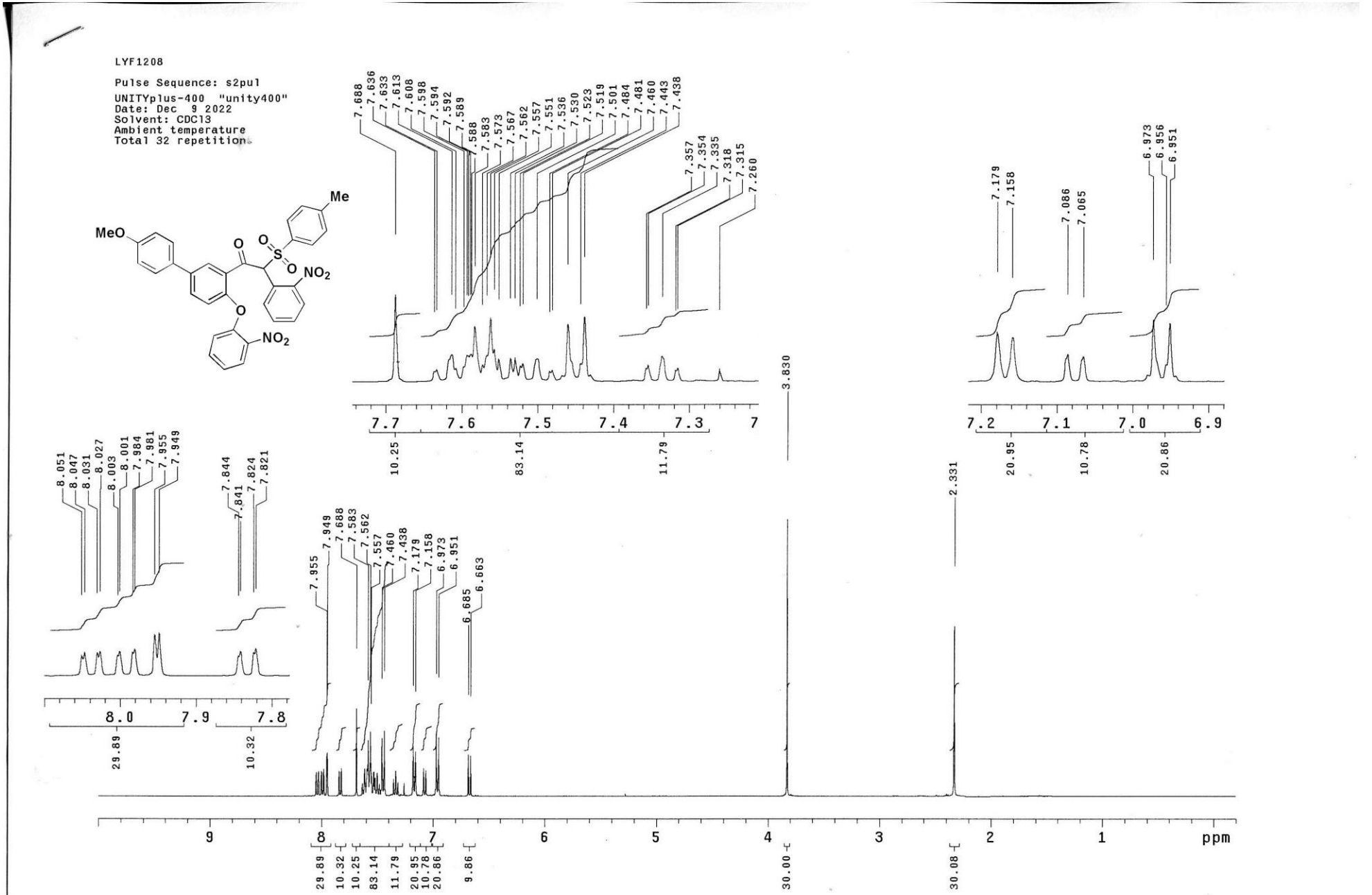
Compound 6ac (^{19}F-NMR spectral data-1)



Compound 6ac (^{19}F-NMR spectral data-2)



Compound 6ad (^{1}H-NMR spectral data)

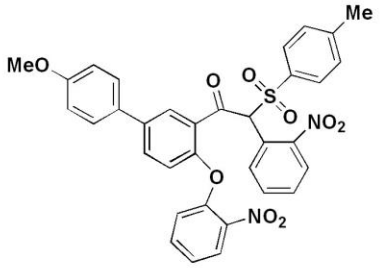


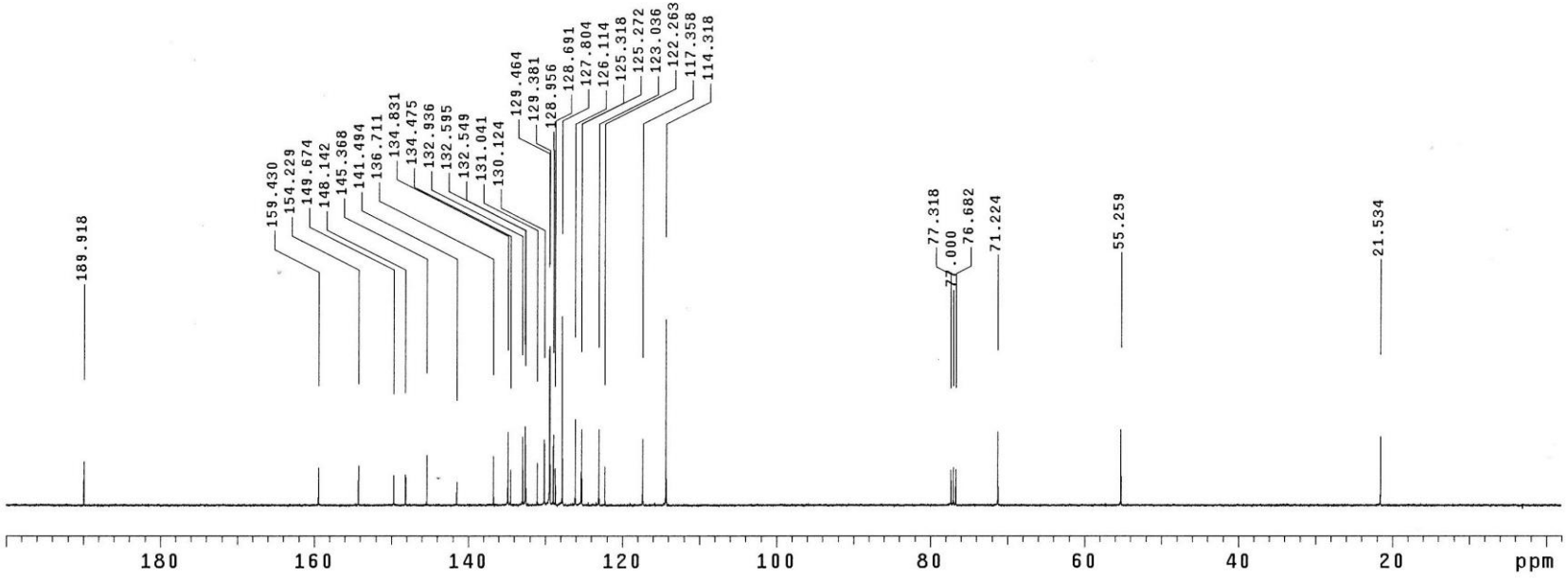
Compound 6ad (^{13}C-NMR spectral data)

LYF1208

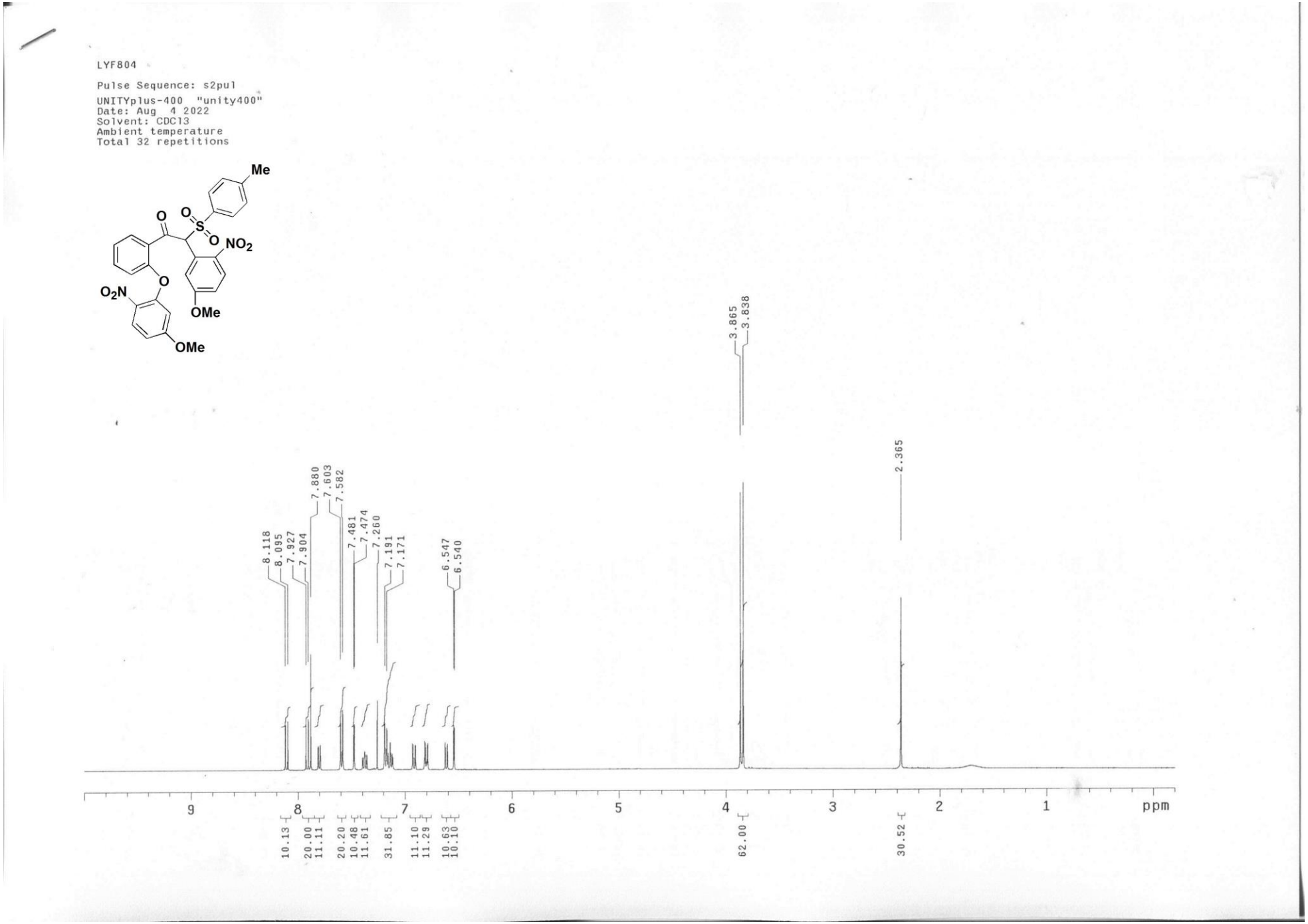
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 9 2022
Solvent: CDCl3
Ambient temperature
Total 1568 repetitions

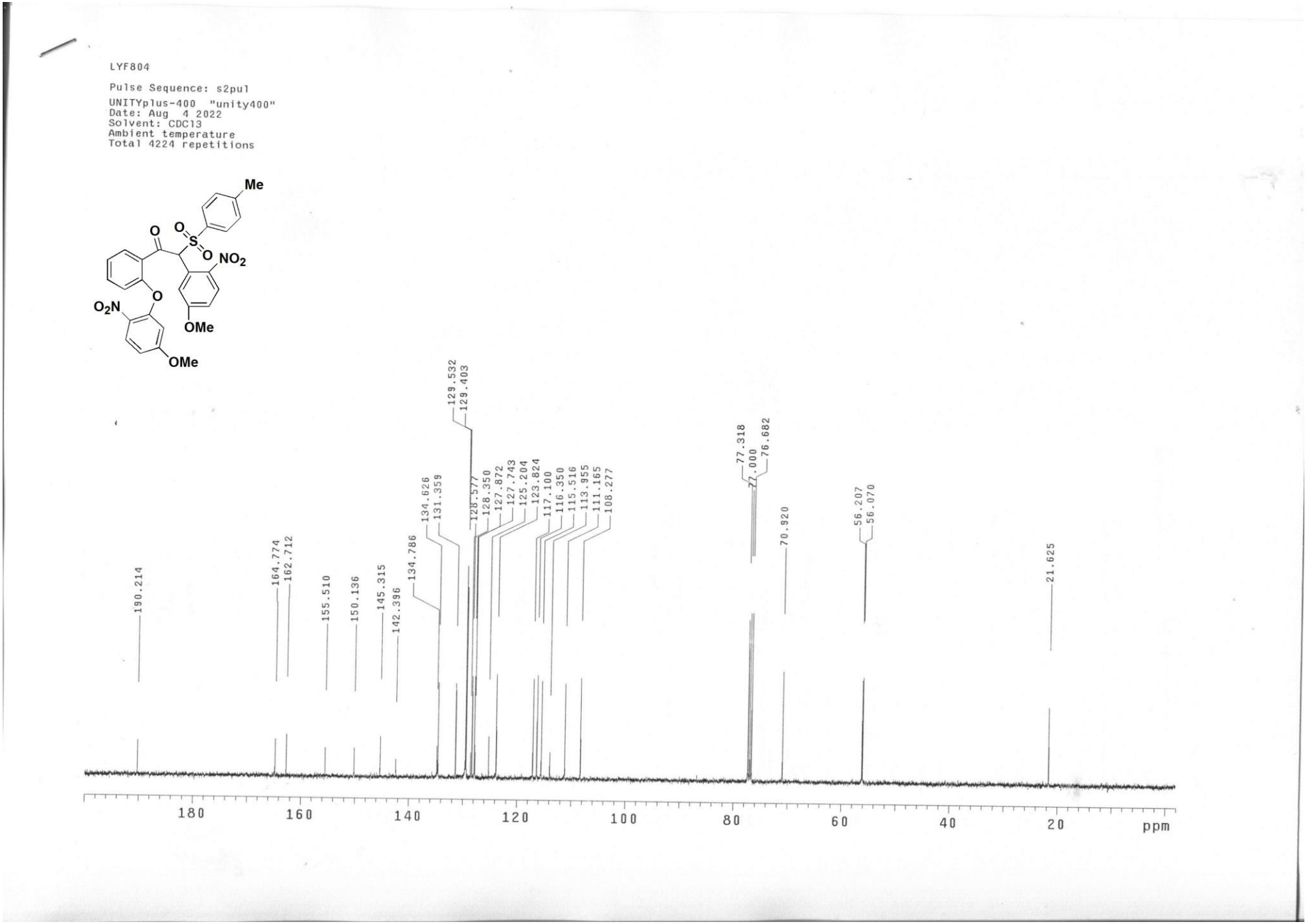




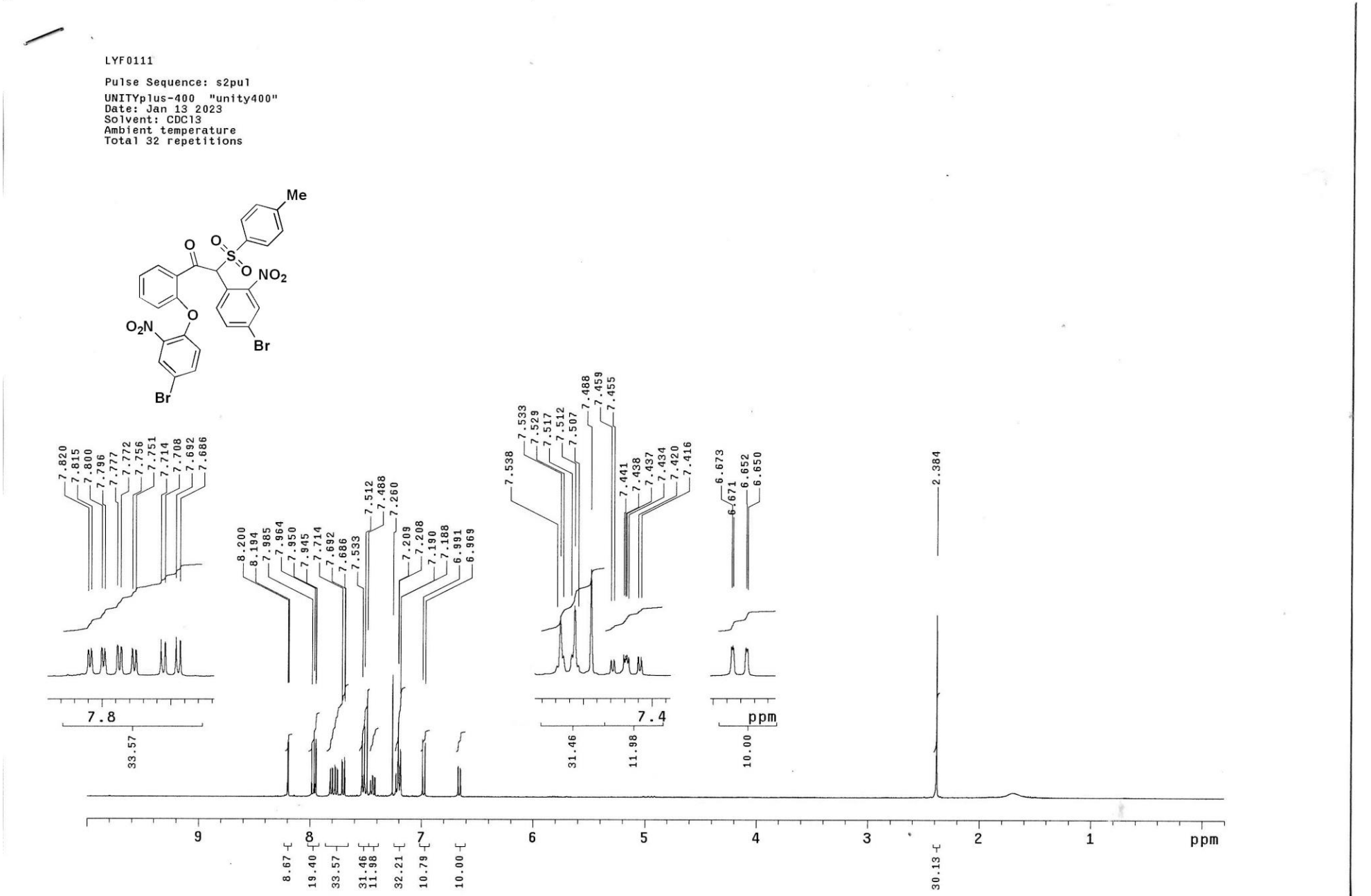
Compound 6ae (^{1}H-NMR spectral data)



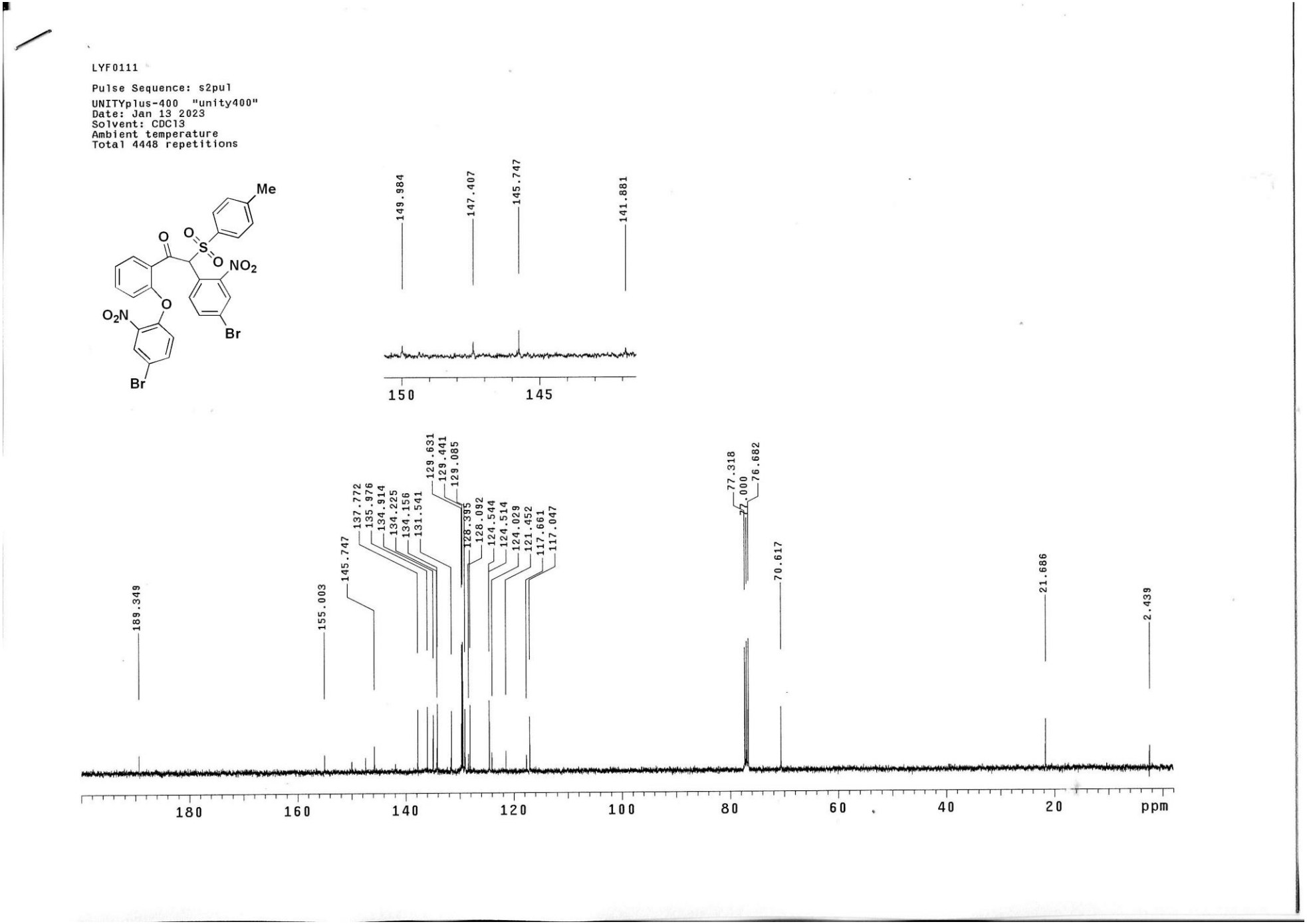
Compound 6ae (^{13}C-NMR spectral data)



Compound 6af (^{1}H-NMR spectral data)



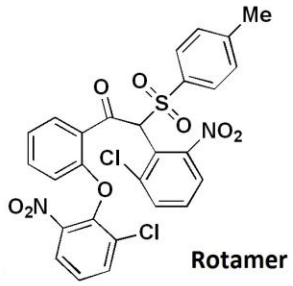
Compound 6af (^{13}C-NMR spectral data)



Compound 6ag (^{1}H-NMR spectral data)

LYF808

Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Aug 10 2022
Solvent: CDCl3
Ambient temperature
Total 48 repetitions



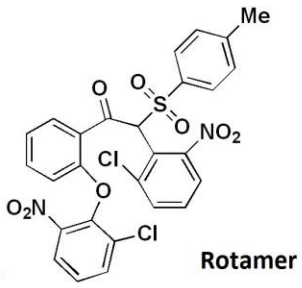
Rotamer

Compound 6ag (^{13}C-NMR spectral data)

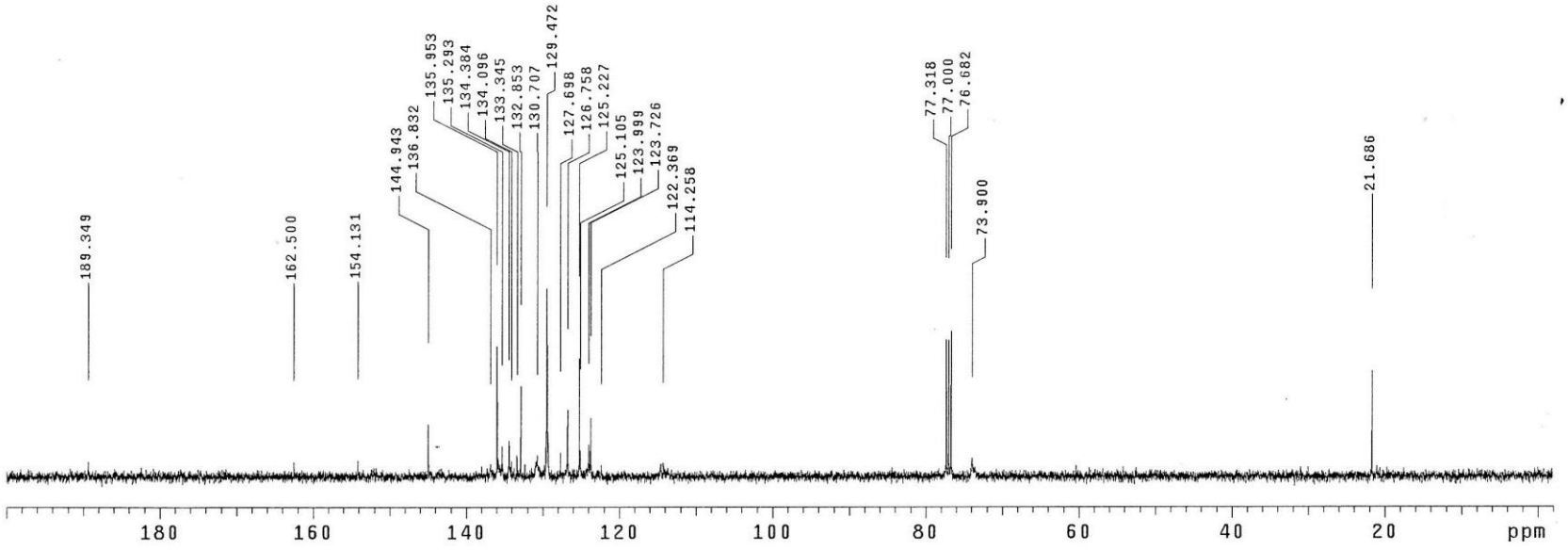
LYF808

Pulse Sequence: s2pul

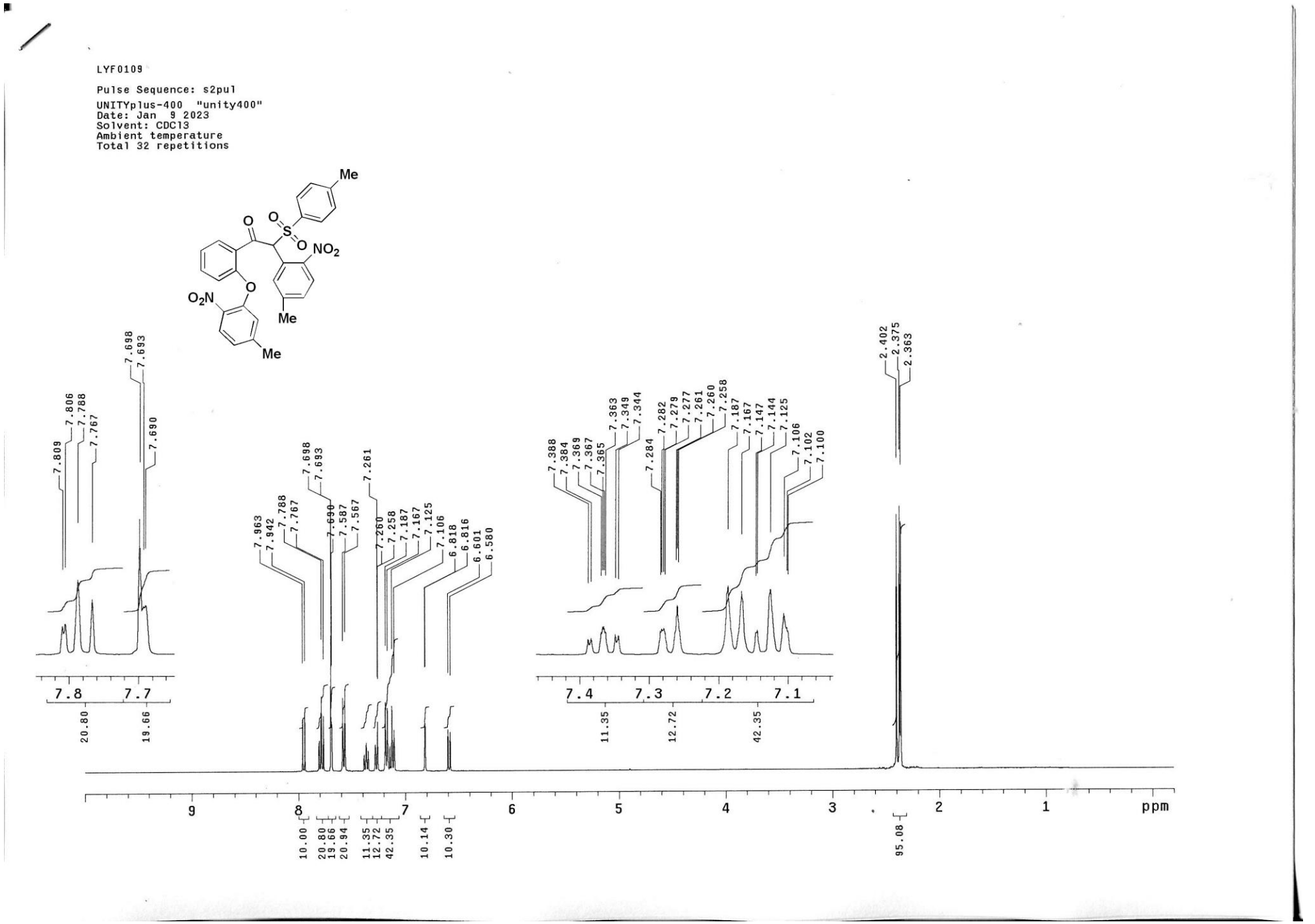
UNITYplus-400 "unity400"
Date: Aug 10 2022
Solvent: CDCl3
Ambient temperature
Total 1120 repetitions



Rotamer



Compound 6ah (^{1}H-NMR spectral data)

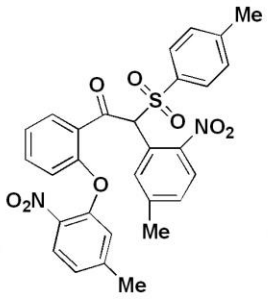


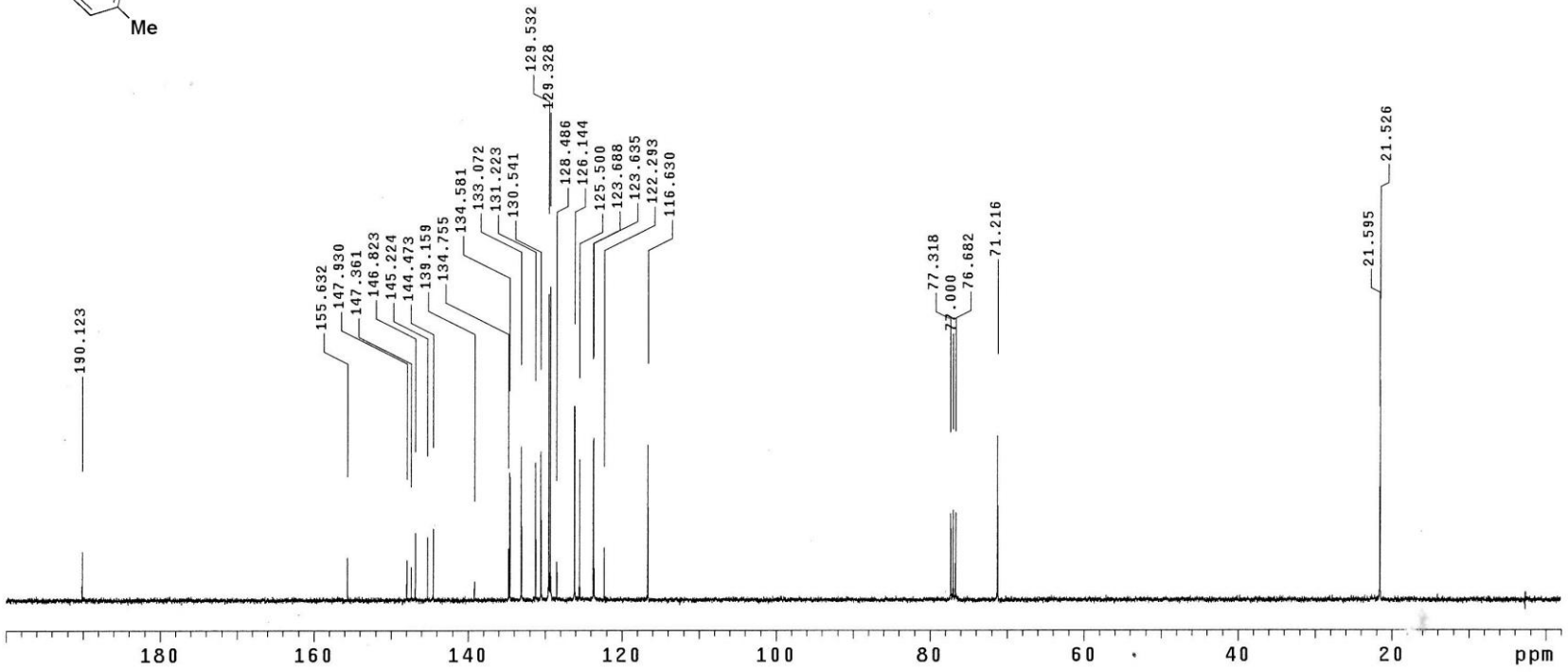
Compound 6ah (^{13}C-NMR spectral data)

LYF0109

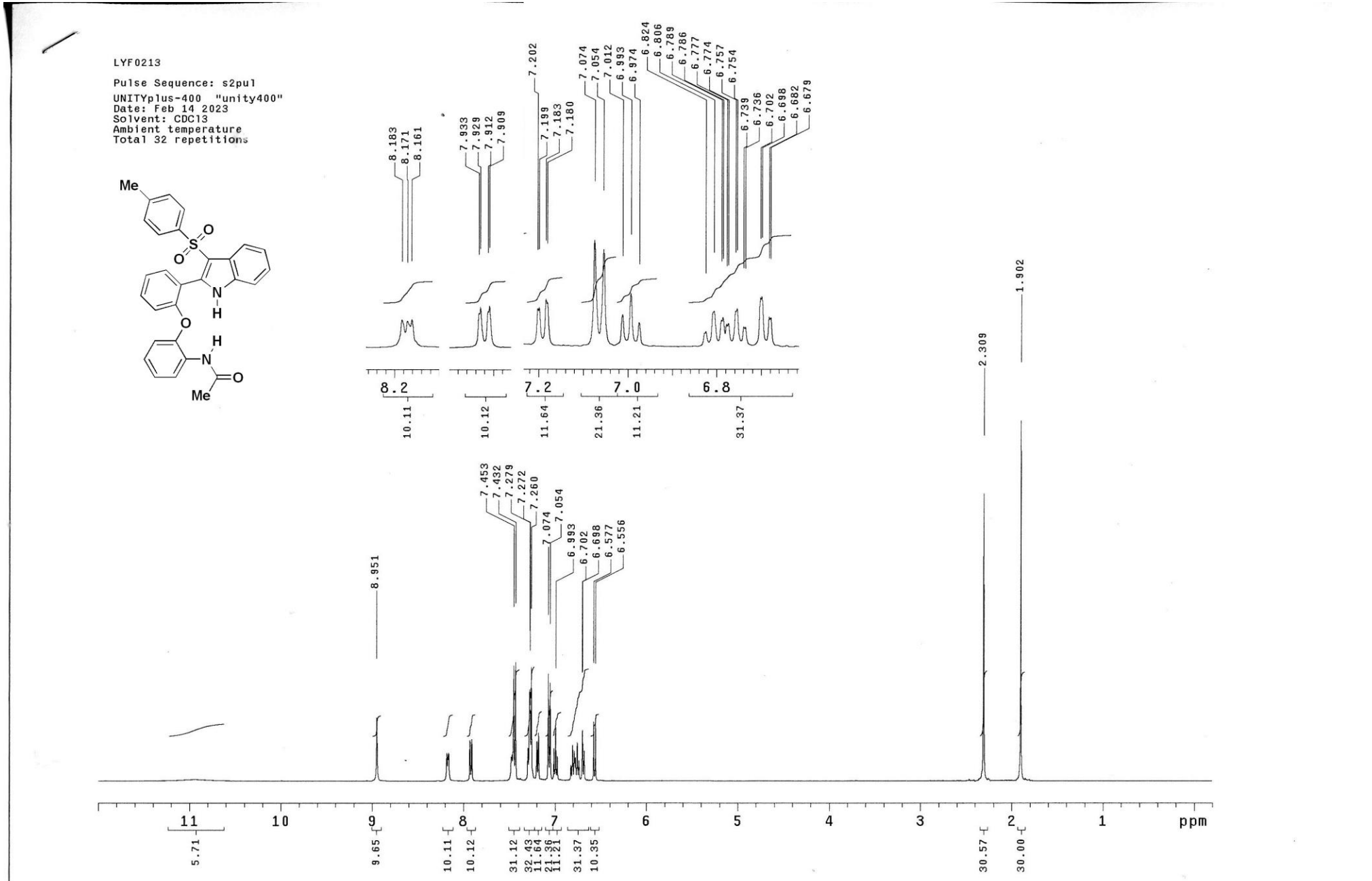
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jan 9 2023
Solvent: CDCl3
Ambient temperature
Total 1600 repetitions

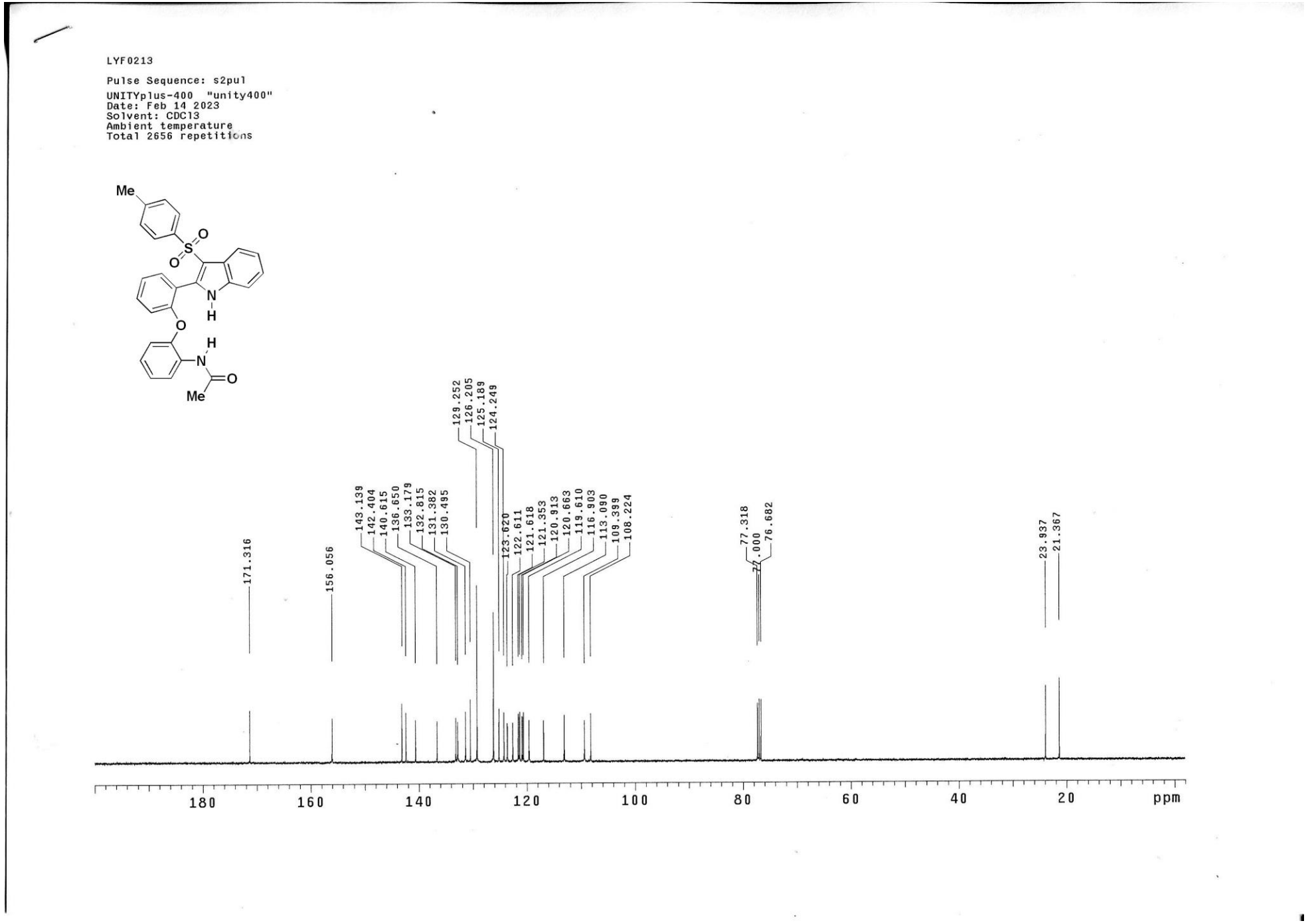




Compound 8a (^{1}H-NMR spectral data)



Compound 8a (^{13}C-NMR spectral data)

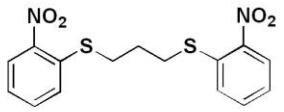


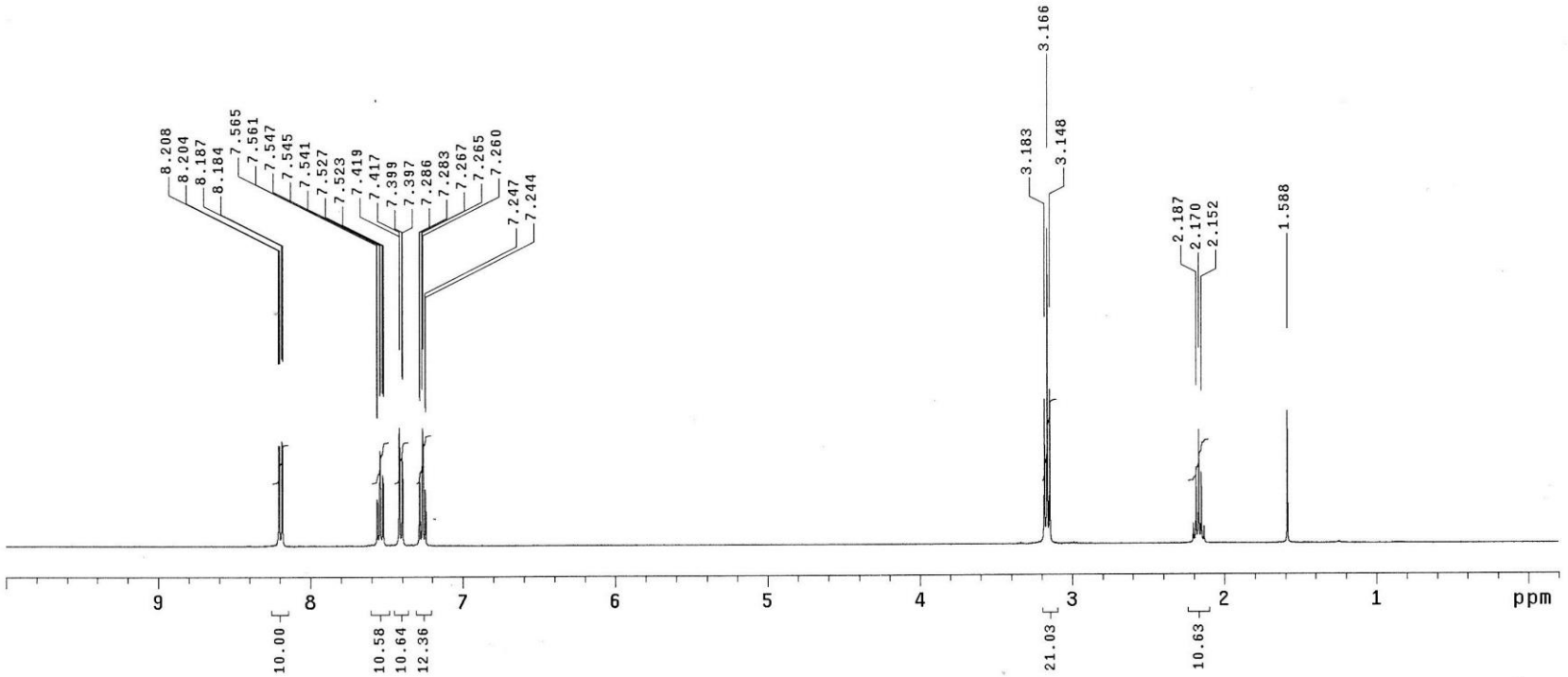
Compound 9a (^{1}H-NMR spectral data)

LYF0222

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Feb 23 2023
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



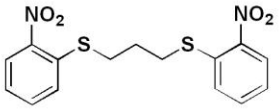


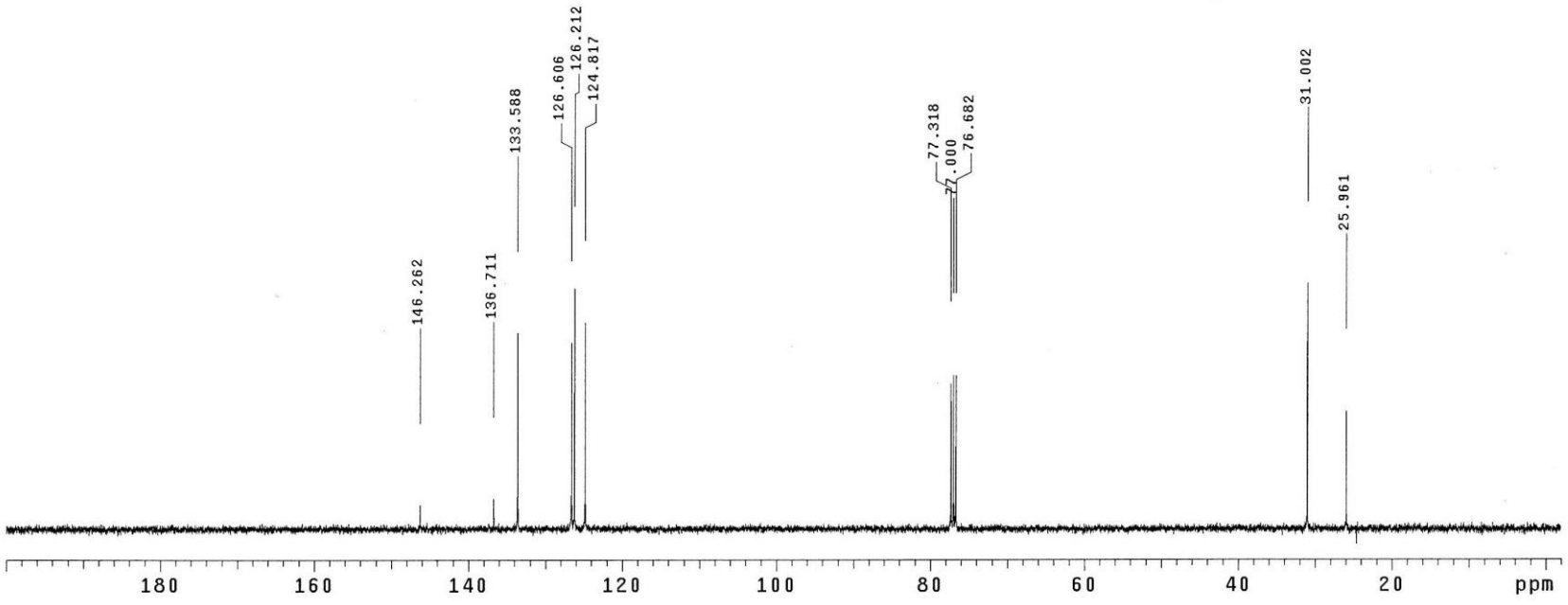
Compound 9a (^{13}C-NMR spectral data)

LYF0222

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Feb 23 2023
Solvent: CDCl3
Ambient temperature
Total 4000 repetitions



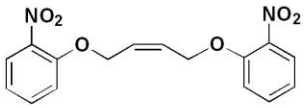


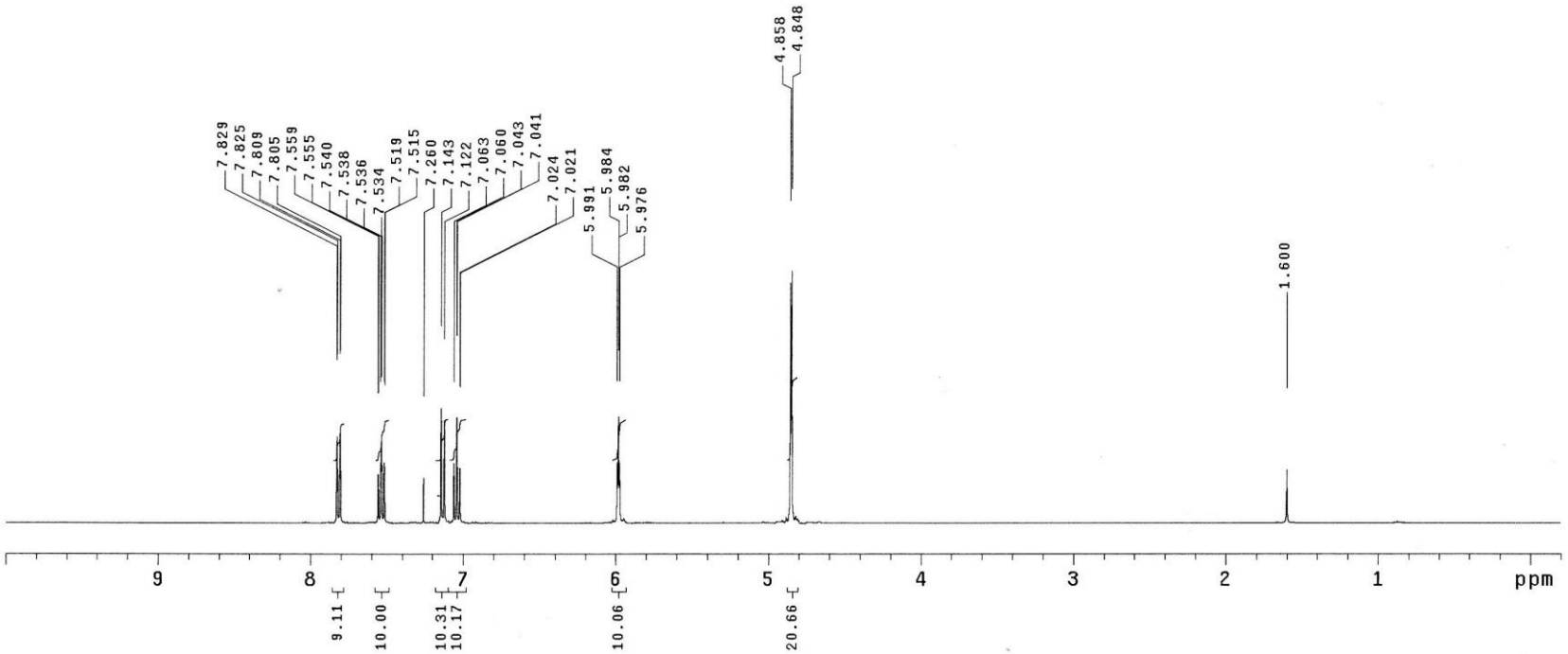
Compound 9b (^{1}H-NMR spectral data)

LYF0307

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Mar 7 2023
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



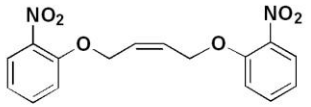


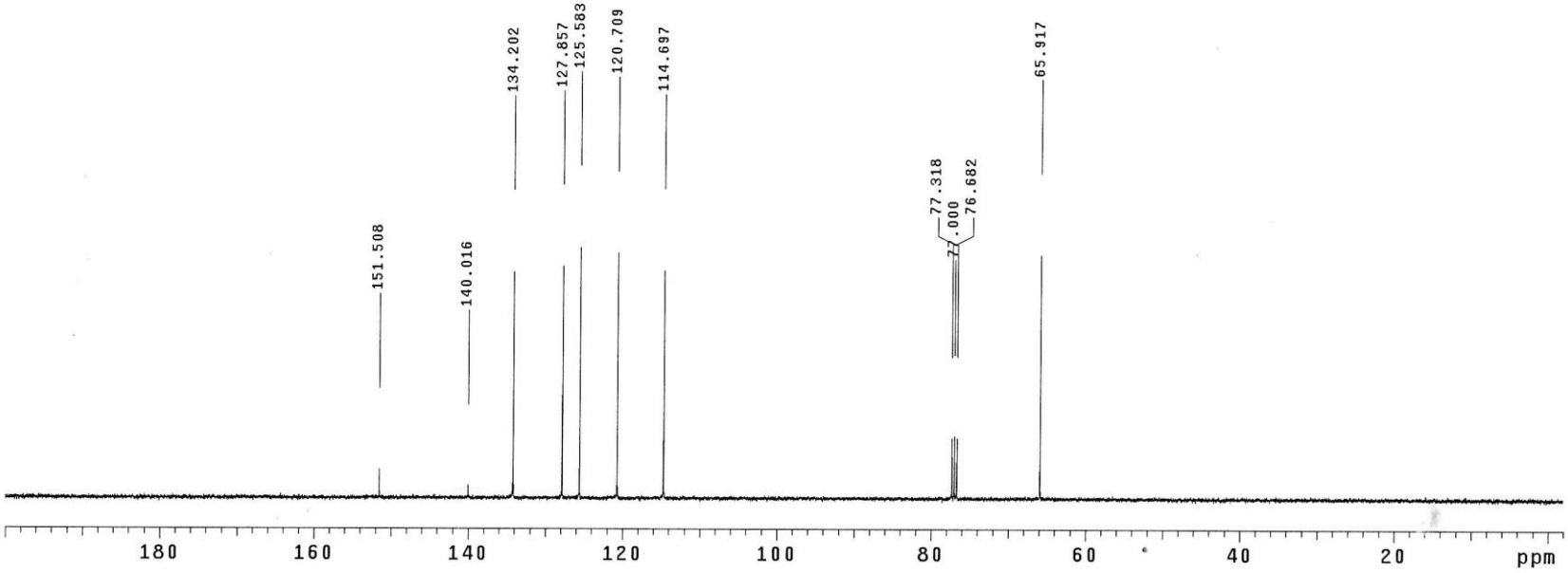
Compound 9b (^{13}C-NMR spectral data)

LYF0307

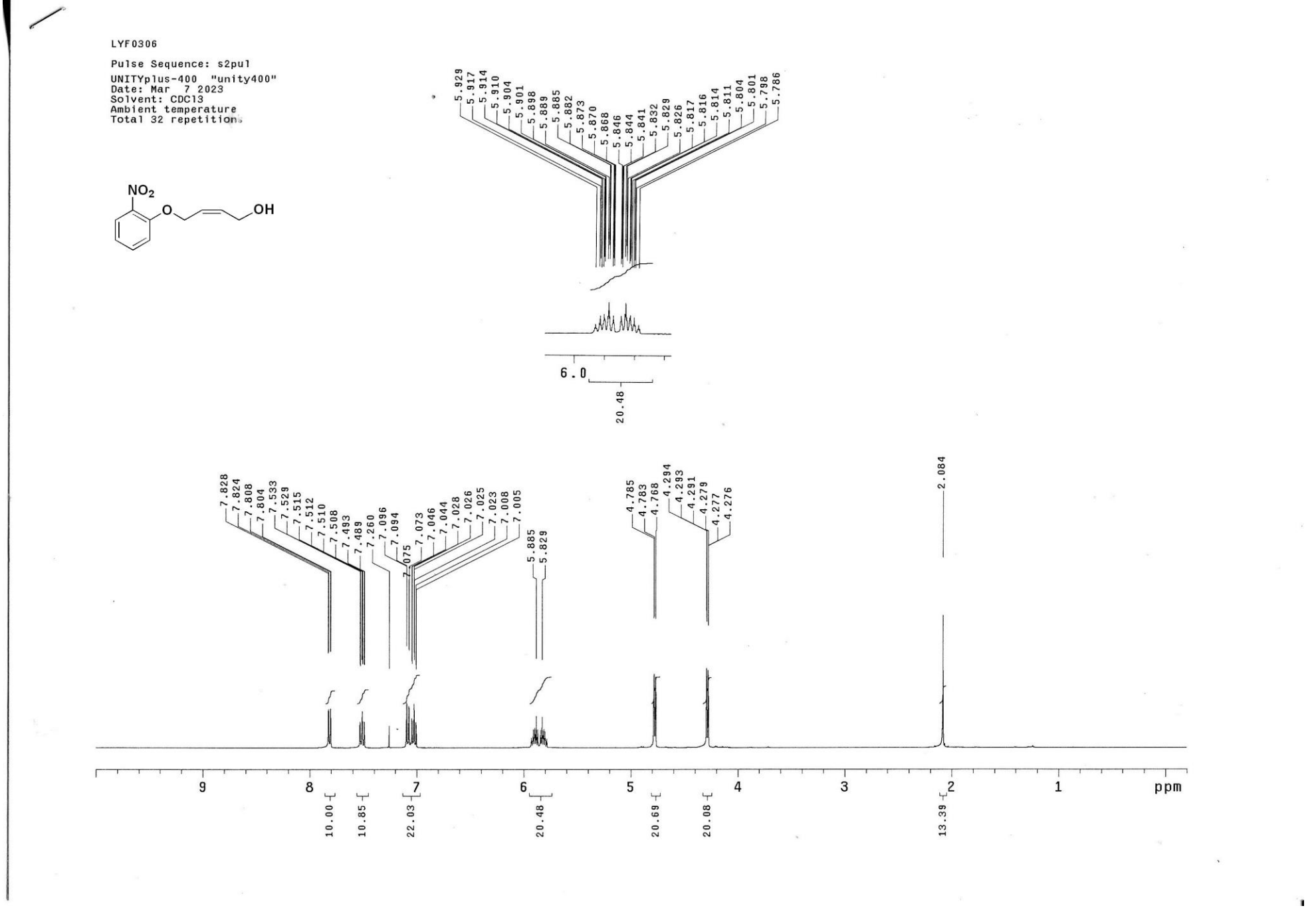
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Mar 7 2023
Solvent: CDCl3
Ambient temperature
Total 1840 repetitions





Compound 9b' (^{1}H-NMR spectral data)

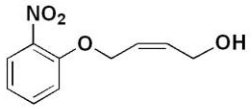


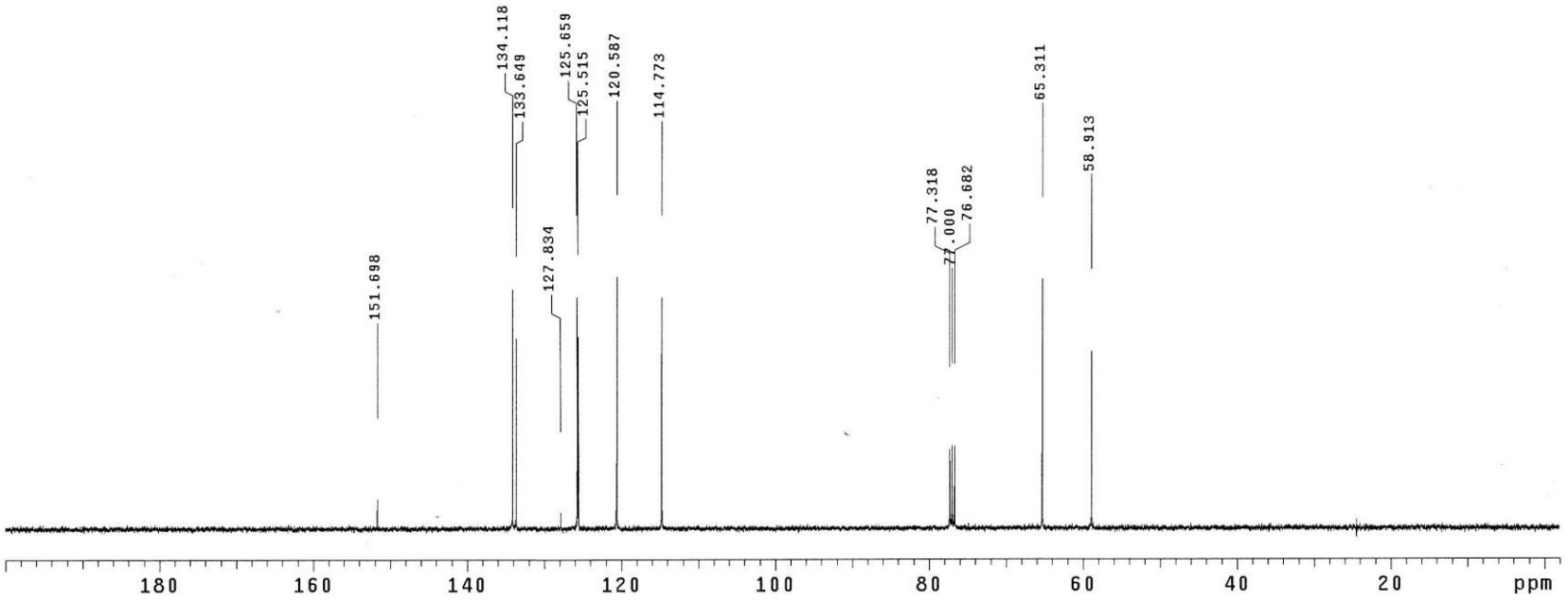
Compound 9b' (^{13}C-NMR spectral data)

LYF0306

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Mar 7 2023
Solvent: CDCl3
Ambient temperature
Total 2080 repetitions



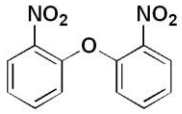


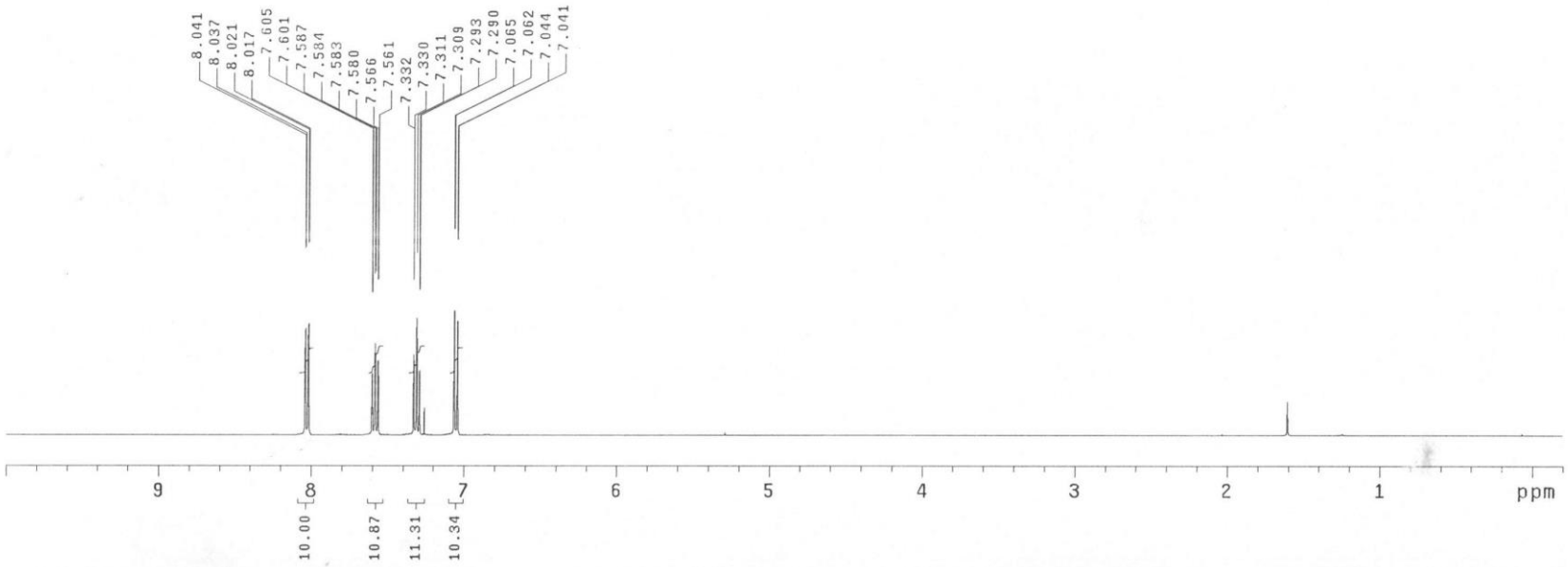
Compound 9c (^{1}H-NMR spectral data)

LYF1536

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 31 2022
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



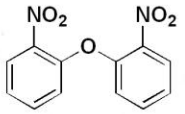


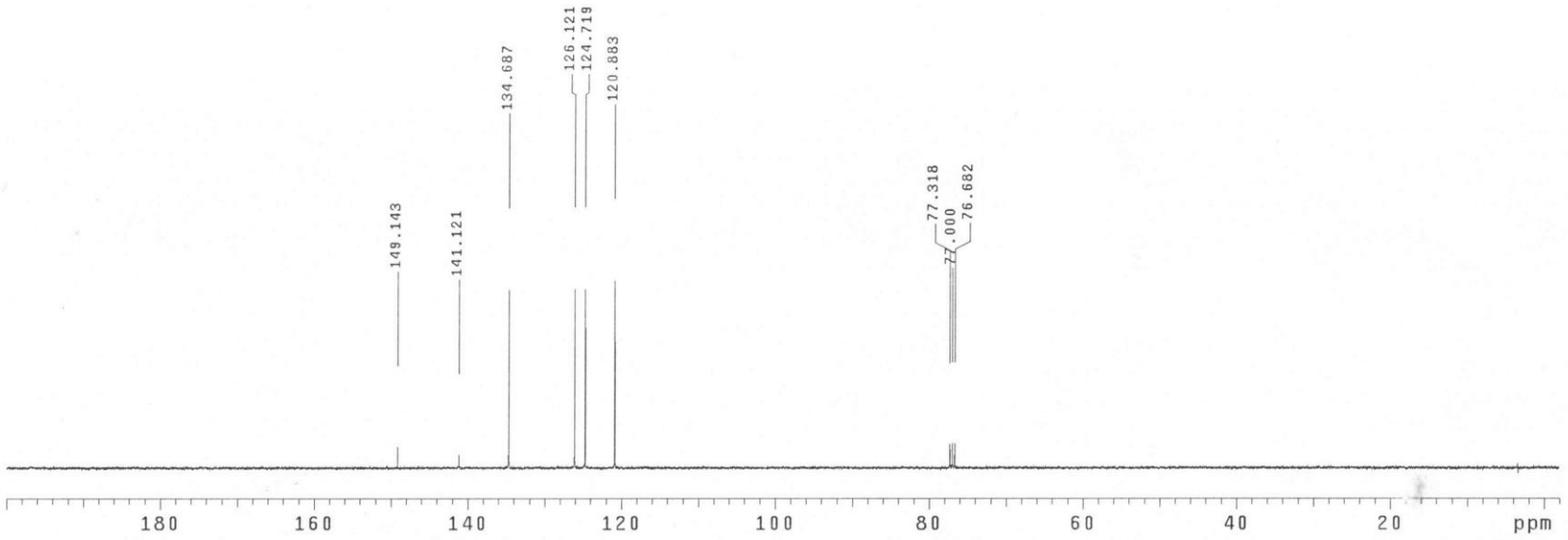
Compound 9c (^{13}C-NMR spectral data)

LYF1536

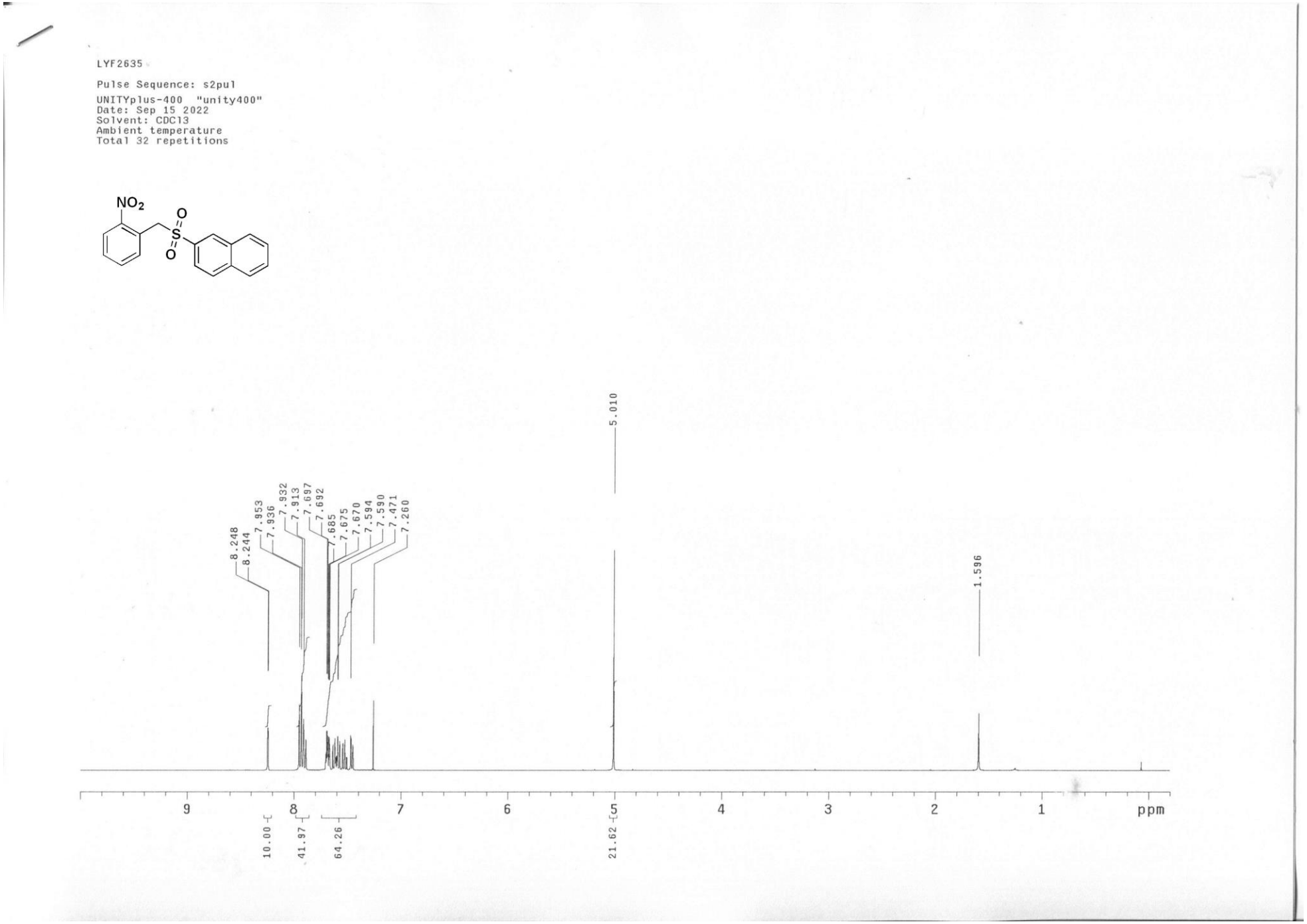
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 31 2022
Solvent: CDCl3
Ambient temperature
Total 432 repetitions





Compound 11 (^{1}H-NMR spectral data)

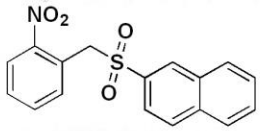


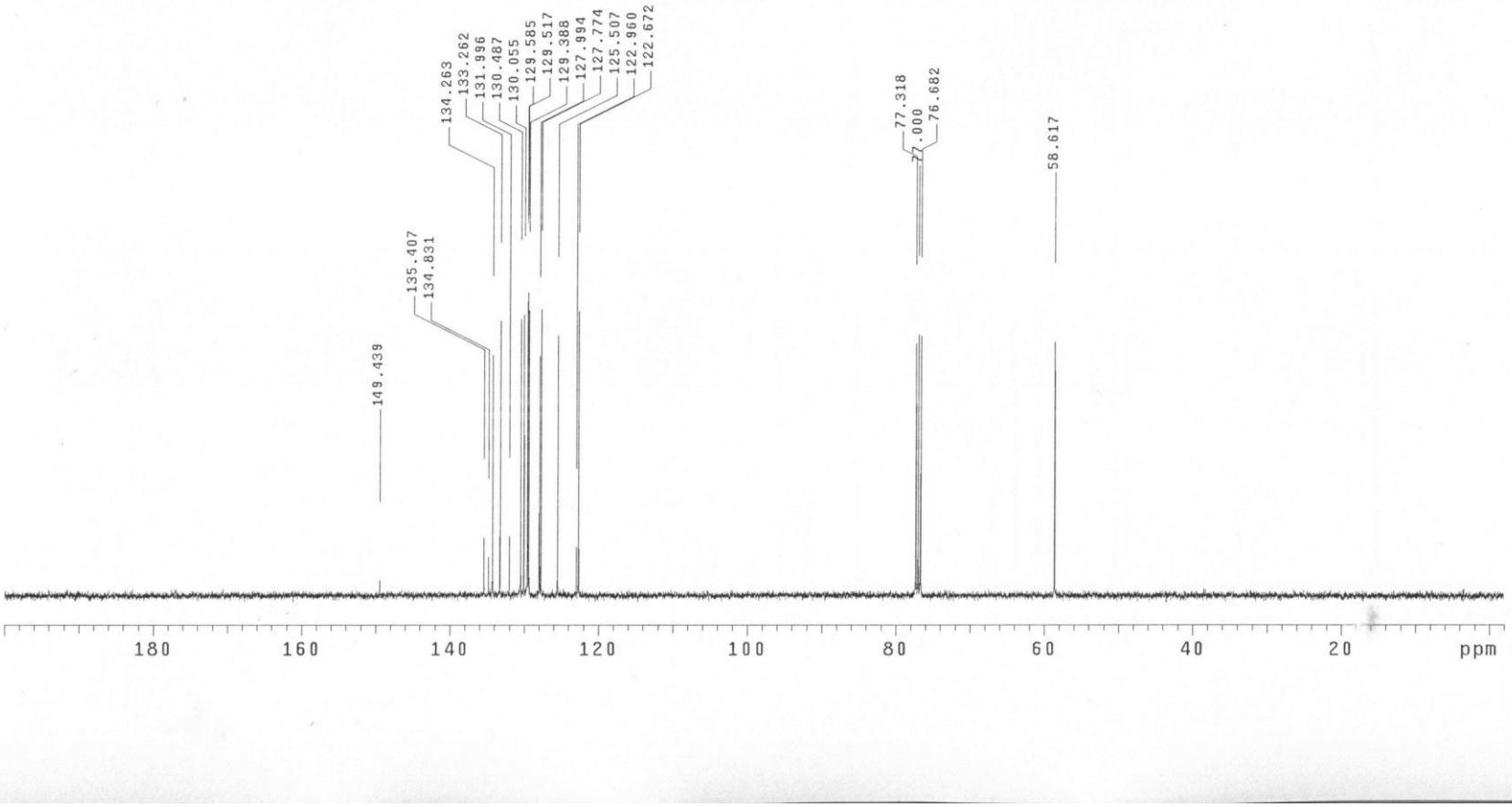
Compound 11 (^{13}C-NMR spectral data)

LYF2635

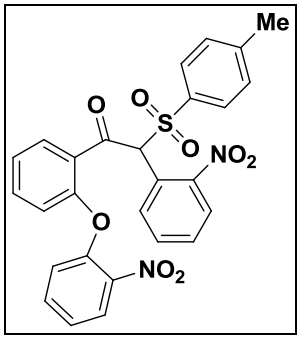
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Sep 15 2022
Solvent: CDCl3
Ambient temperature
Total 5520 repetitions



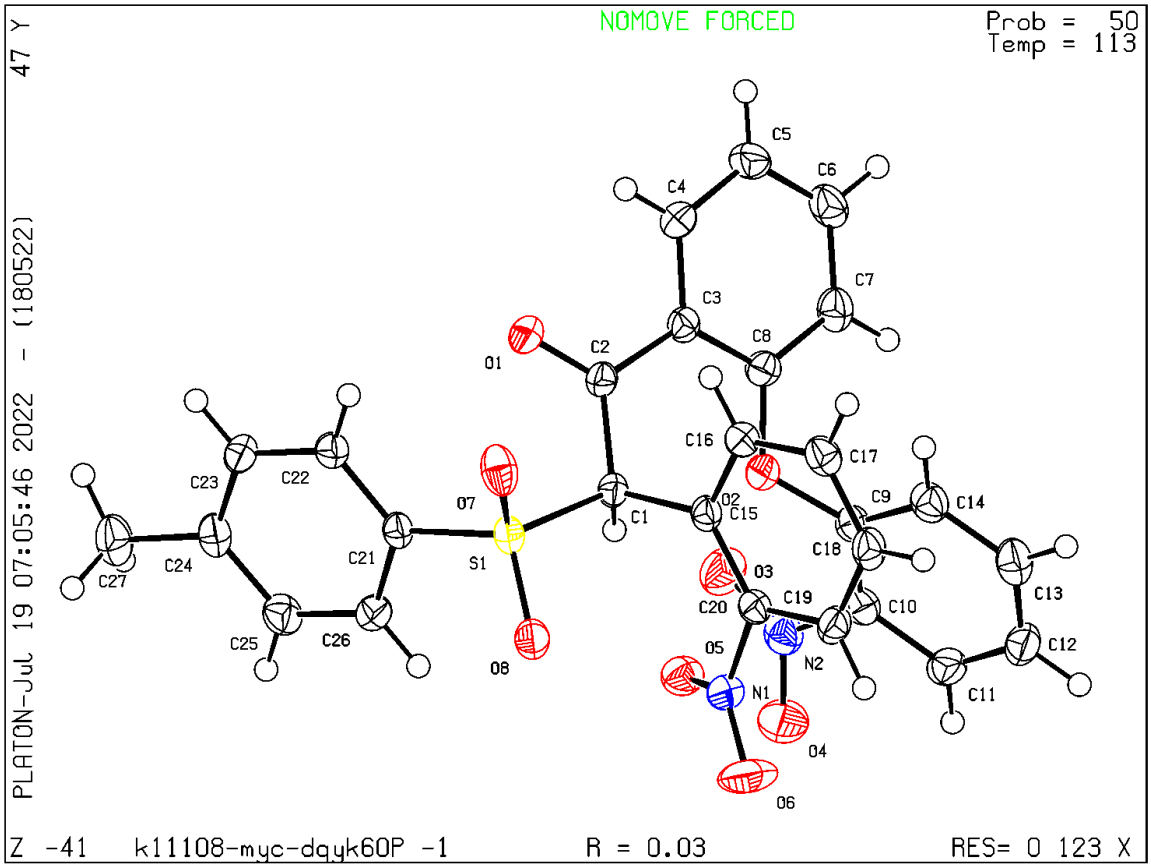


X-ray crystal data of compound 6a



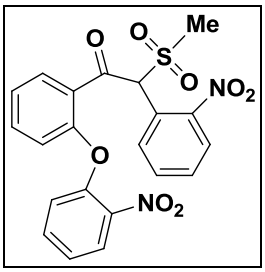
Sample preparation : A solution of compound **6a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



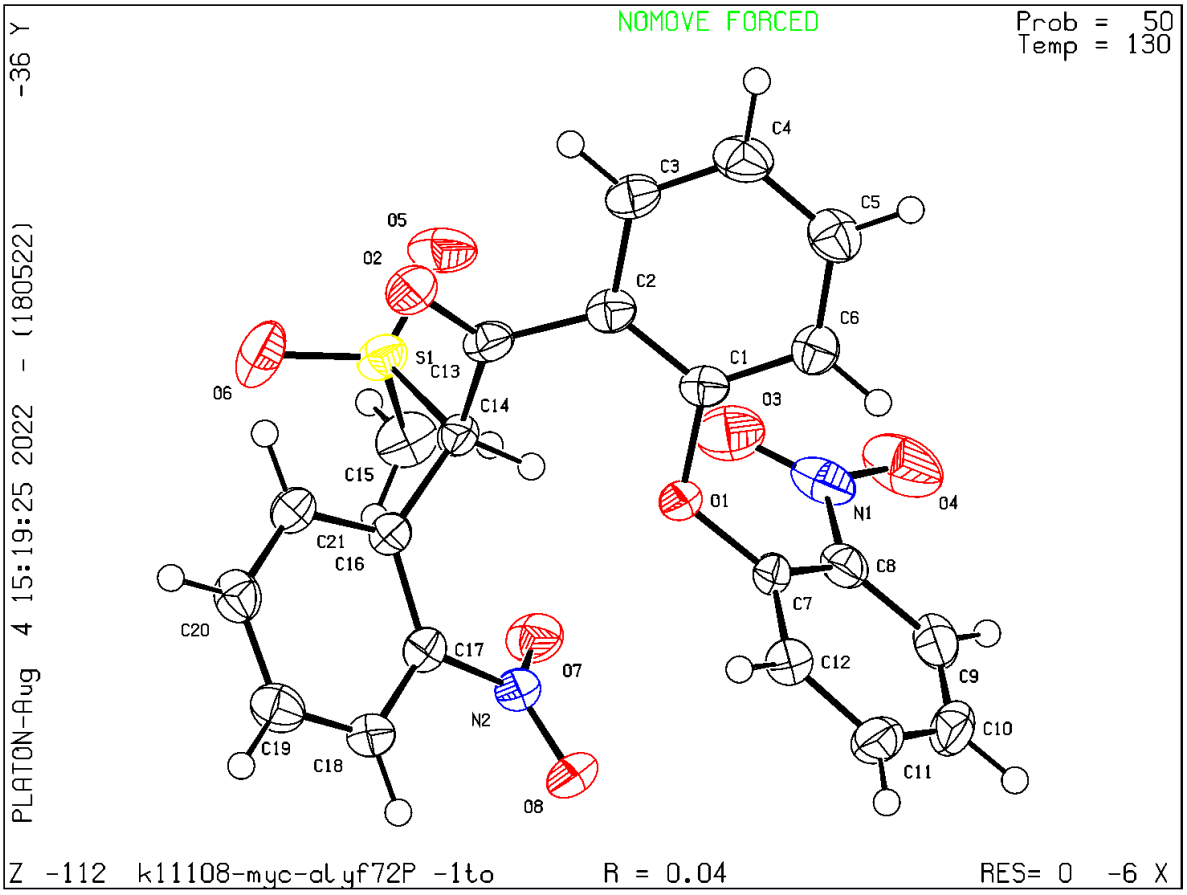
Empirical formula	$C_{27}H_{20}N_2O_8S$
Formula weight	532.51
Temperature/K	113(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.08950(10)
b/Å	10.48040(10)
c/Å	14.8834(2)
α/°	74.0530(10)
β/°	77.7610(10)
γ/°	86.0010(10)
Volume/Å ^{3}	1185.61(3)
Z	2
ρ_{calc}g/cm^3	1.492
μ/mm^{-1}	0.195
F(000)	552.0
Crystal size/mm^3	0.25 × 0.2 × 0.2
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.042 to 49.996
Index ranges	$-9 \leq h \leq 9$, $-12 \leq k \leq 12$, $-17 \leq l \leq 17$
Reflections collected	39857
Independent reflections	4183 [R_{int} = 0.0348, R_{sigma} = 0.0208]
Data/restraints/parameters	4183/138/344
Goodness-of-fit on F^2	1.077
Final R indexes [I>=2σ (I)]	R_1 = 0.0318, wR_2 = 0.0838
Final R indexes [all data]	R_1 = 0.0372, wR_2 = 0.0871
Largest diff. peak/hole / e Å $^{-3}$	0.30/-0.37

X-ray crystal data of compound 6i



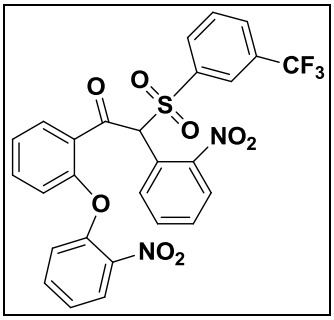
Sample preparation : A solution of compound **6i** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



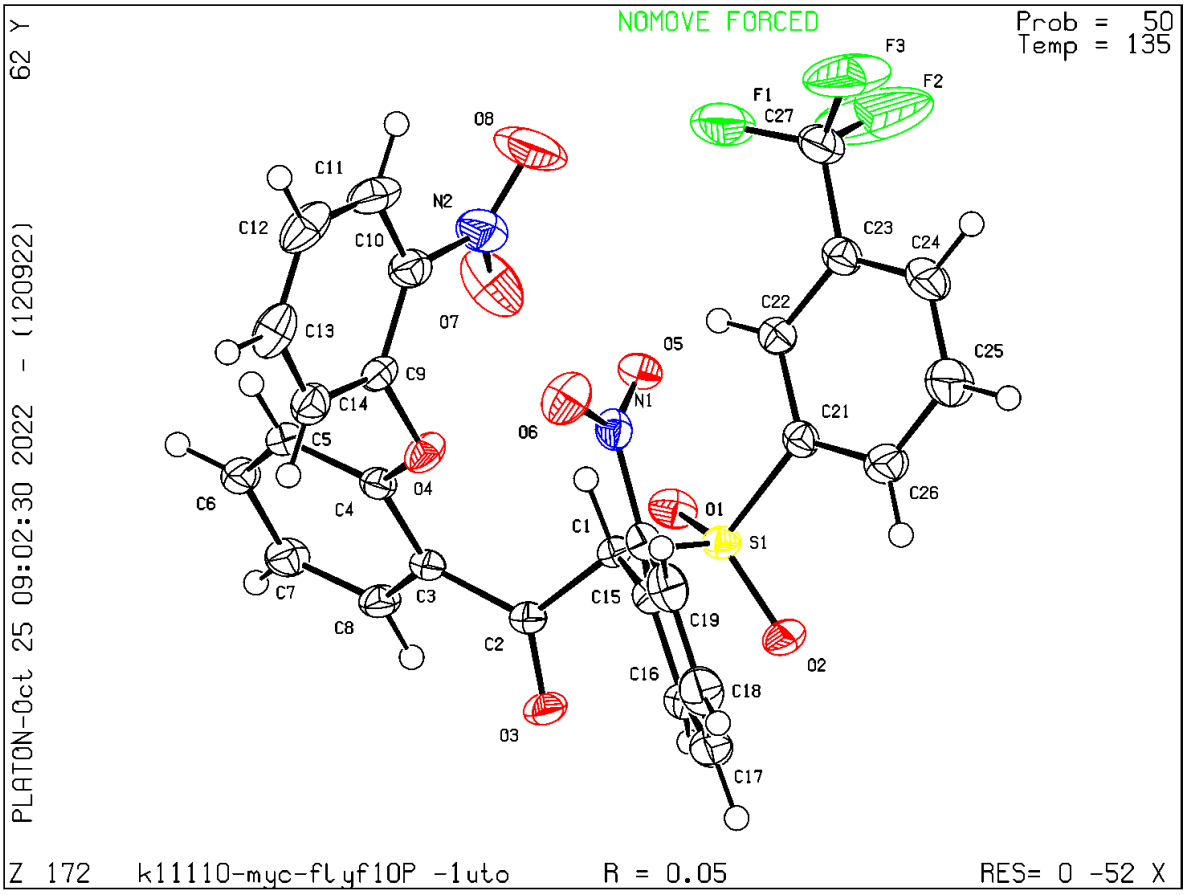
Empirical formula	$C_{10.5}H_8NO_4S_{0.5}$
Formula weight	228.21
Temperature/K	130(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.6503(4)
b/Å	9.5028(4)
c/Å	13.7219(5)
α/°	73.673(3)
β/°	77.253(3)
γ/°	66.575(4)
Volume/Å ^{3}	985.64(8)
Z	4
ρ_{calc}g/cm^3	1.538
μ/mm^{-1}	0.220
F(000)	472.0
Crystal size/mm^3	0.5 × 0.3 × 0.3
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	3.116 to 50
Index ranges	$-10 \leq h \leq 10$, $-11 \leq k \leq 11$, $-16 \leq l \leq 16$
Reflections collected	22256
Independent reflections	3455 [R_{int} = 0.0700, R_{sigma} = 0.0386]
Data/restraints/parameters	3455/0/290
Goodness-of-fit on F^2	1.065
Final R indexes [I>=2σ (I)]	R_1 = 0.0427, wR_2 = 0.1112
Final R indexes [all data]	R_1 = 0.0499, wR_2 = 0.1149
Largest diff. peak/hole / e Å $^{-3}$	0.50/-0.42

X-ray crystal data of compound 6t



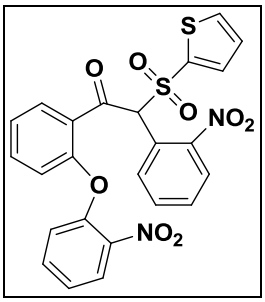
Sample preparation : A solution of compound **6t** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



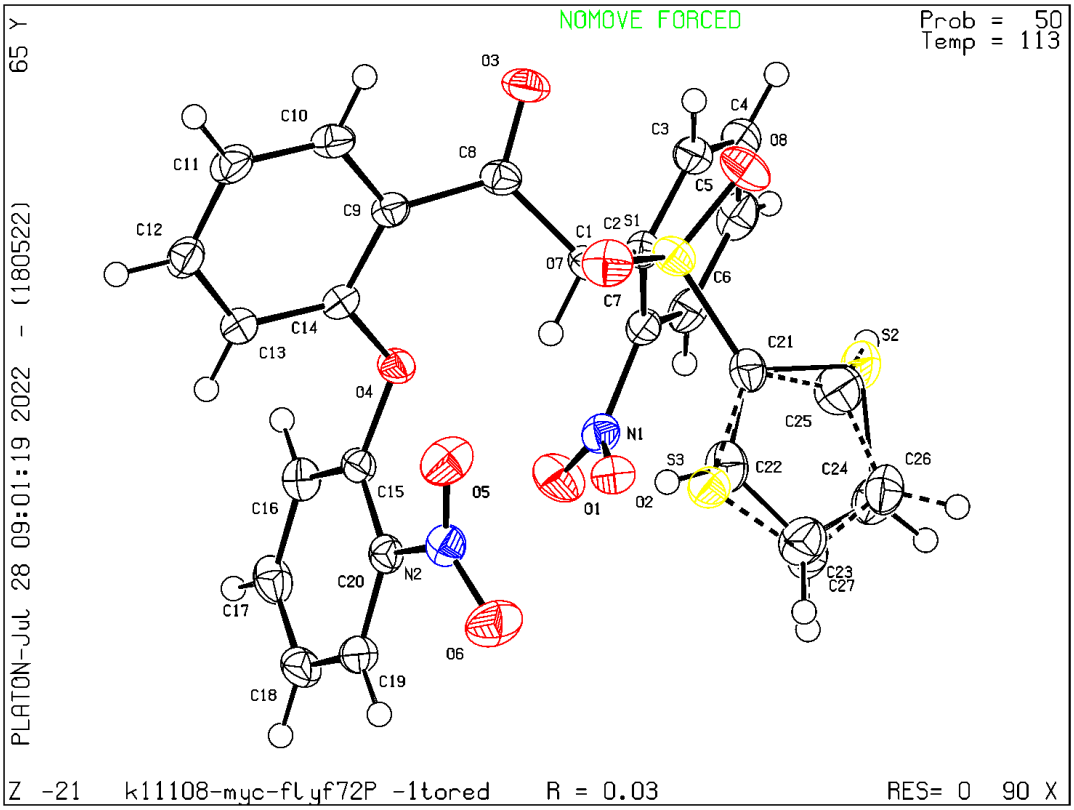
Formula weight	234.59
Temperature/K	135(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.30070(10)
b/Å	9.92720(10)
c/Å	16.4260(2)
α/°	104.0290(10)
β/°	91.0020(10)
γ/°	107.6750(10)
Volume/Å ^{3}	1245.10(3)
Z	5
ρ_{calc}g/cm^3	1.564
μ/mm^{-1}	0.210
F(000)	600.0
Crystal size/mm^3	0.03 × 0.02 × 0.02
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.46 to 54.254
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, -20 ≤ l ≤ 20
Reflections collected	54640
Independent reflections	5289 [R_{int} = 0.0621, R_{sigma} = 0.0274]
Data/restraints/parameters	5289/0/370
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0493, wR_2 = 0.1263
Final R indexes [all data]	R_1 = 0.0564, wR_2 = 0.1309
Largest diff. peak/hole / e Å $^{-3}$	0.95/-0.65

X-ray crystal data of compound 6x



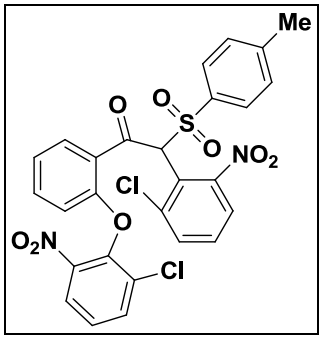
Sample preparation : A solution of compound **6x** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



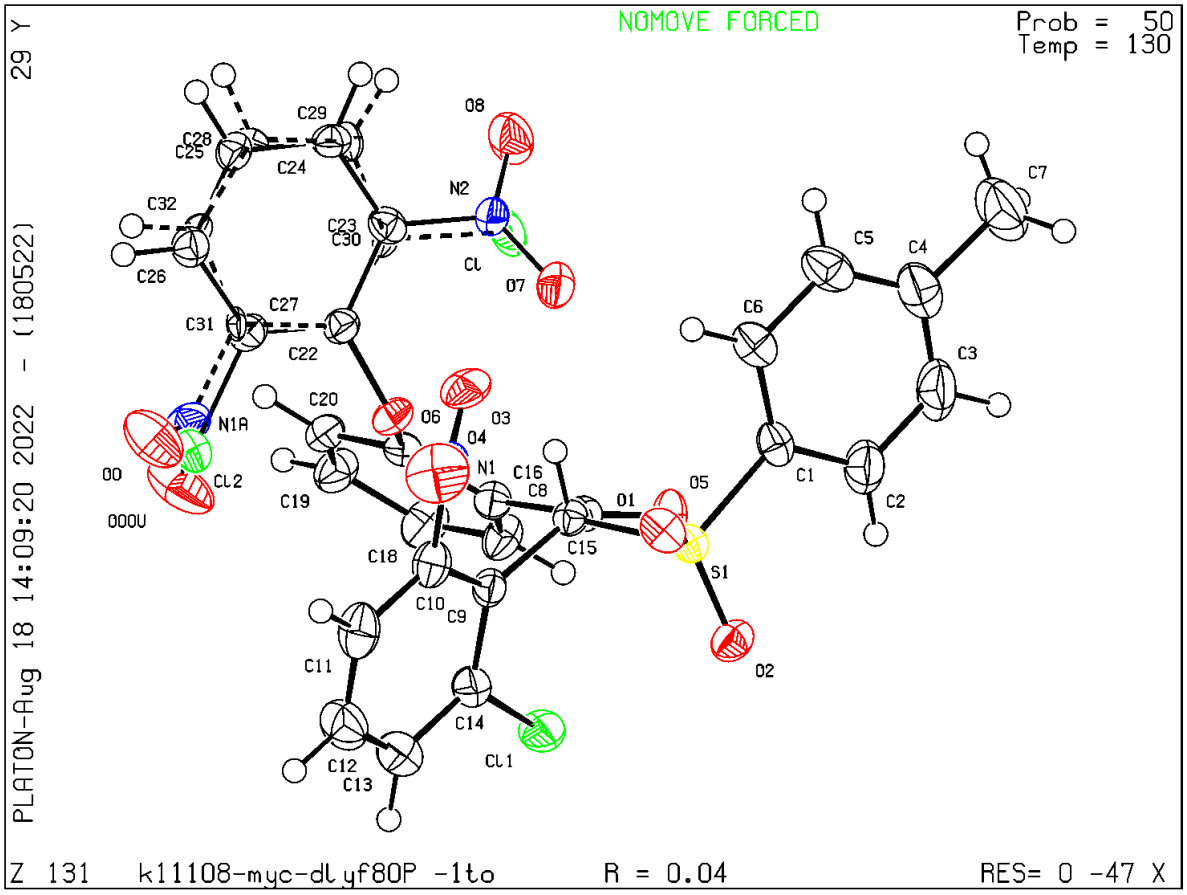
Empirical formula	$C_{24}H_{16}N_2O_8S_2$
Formula weight	524.51
Temperature/K	113(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.3670(2)
b/Å	9.8898(2)
c/Å	14.7637(2)
α/°	82.0990(10)
β/°	83.390(2)
γ/°	67.477(2)
Volume/Å ^{3}	1115.17(4)
Z	2
ρ_{calc}g/cm^3	1.562
μ/mm^{-1}	0.296
F(000)	540.0
Crystal size/mm^3	0.38 × 0.35 × 0.2
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.482 to 49.998
Index ranges	$-9 \leq h \leq 9$, $-11 \leq k \leq 11$, $-17 \leq l \leq 13$
Reflections collected	27573
Independent reflections	3913 [R_{int} = 0.0458, R_{sigma} = 0.0297]
Data/restraints/parameters	3913/1359/362
Goodness-of-fit on F^2	1.085
Final R indexes [I>=2σ (I)]	R_1 = 0.0348, wR_2 = 0.0910
Final R indexes [all data]	R_1 = 0.0387, wR_2 = 0.0937
Largest diff. peak/hole / e Å $^{-3}$	0.33/-0.39

X-ray crystal data of compound 6ag



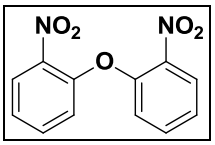
Sample preparation : A solution of compound **6ag** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



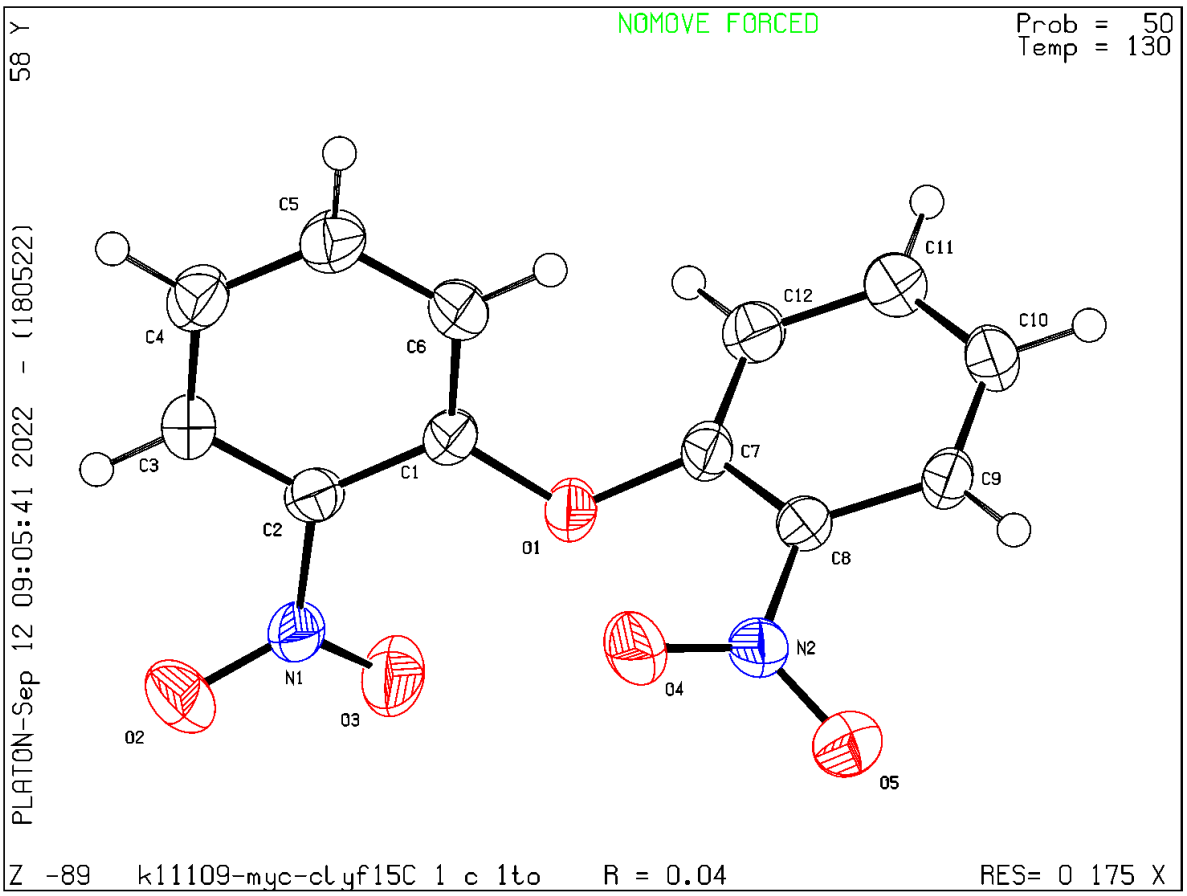
Formula weight	200.46
Temperature/K	130(2)
Crystal system	triclinic
Space group	P-1
a/Å	9.5100(2)
b/Å	10.9393(3)
c/Å	14.4361(2)
α/°	80.891(2)
β/°	88.429(2)
γ/°	81.563(2)
Volume/Å ^{3}	1466.83(6)
Z	6
ρ_{calc}g/cm^3	1.362
μ/mm^{-1}	0.342
F(000)	616.0
Crystal size/mm^3	0.5 × 0.4 × 0.4
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.33 to 49.998
Index ranges	-8 ≤ h ≤ 11, -12 ≤ k ≤ 13, -17 ≤ l ≤ 17
Reflections collected	30871
Independent reflections	5122 [R_{int} = 0.0281, R_{sigma} = 0.0215]
Data/restraints/parameters	5122/215/448
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0402, wR_2 = 0.1051
Final R indexes [all data]	R_1 = 0.0443, wR_2 = 0.1076
Largest diff. peak/hole / e Å $^{-3}$	0.51/-0.33

X-ray crystal data of compound 9c



Sample preparation : A solution of compound **9c** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



Empirical formula	$C_{12}H_8N_2O_5$
Formula weight	260.20
Temperature/K	130(2)
Crystal system	monoclinic
Space group	Cc
a/Å	7.16580(10)
b/Å	20.4831(4)
c/Å	7.7993(2)
α/°	90
β/°	92.643(2)
γ/°	90
Volume/Å ^{3}	1143.55(4)
Z	4
ρ_{calc}g/cm^3	1.511
μ/mm^{-1}	0.121
F(000)	536.0
Crystal size/mm^3	0.3 × 0.2 × 0.2
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	3.976 to 49.968
Index ranges	$-8 \leq h \leq 8$, $-24 \leq k \leq 24$, $-9 \leq l \leq 9$
Reflections collected	13291
Independent reflections	1944 [R_{int} = 0.0996, R_{sigma} = 0.0399]
Data/restraints/parameters	1944/2/172
Goodness-of-fit on F^2	0.906
Final R indexes [I>=2σ (I)]	R_1 = 0.0388, wR_2 = 0.1097
Final R indexes [all data]	R_1 = 0.0412, wR_2 = 0.1130
Largest diff. peak/hole / e Å $^{-3}$	0.15/-0.21
Flack parameter	-1.6(9)