

Supplementary Information

Readily available alkylbenzenes as precursors for one-pot preparation of buta-1,3-dienes under DDQ visible-light photocatalysis in benzotrifluoride

Palani Natarajan,* Deachen Chuskit and Priya

Department of Chemistry & Centre for Advanced Studies in Chemistry, Panjab University, Chandigarh - 160014, India.

pnataraj@pu.ac.in

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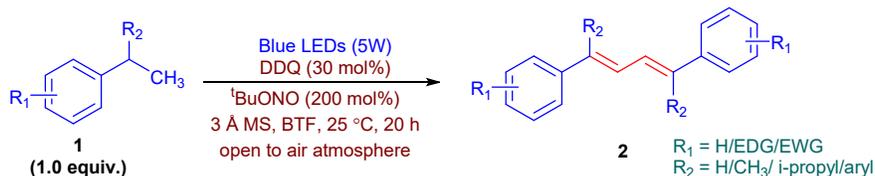
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Experimental Section

1. General Aspects

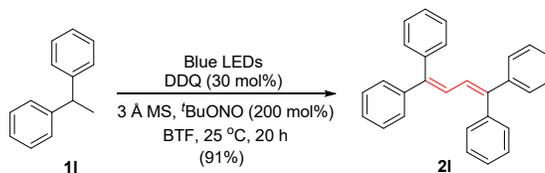
Unless otherwise noted, all the reactions were performed at 25 °C. All chemicals, reagents and precursors were purchased from commercial sources with the best quality and they were used without further purification. All solvents were dried over 3 Å / 4 Å molecular sieves and distilled prior to use.¹ All reactions were carried out in an open-air atmosphere. The column chromatography was performed using silica gel with 100-200 mesh size. Reactions were monitored by analytical thin layer chromatography on silica gel and visualization was accomplished by irradiation with short wave UV light at 254 nm and near UV 366 nm lights. All NMR spectra were recorded on a Bruker Avance (300 MHz) spectrometer in DMSO-d₆ as a non-chlorinated solvent. All products are known and were characterized by their ¹H- and ¹³C NMR followed by a comparison with authentic samples spectra.²⁻⁹ Chemical shifts are expressed as δ -value in parts per million (ppm) and were calibrated using the residual protonated solvent as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; and so on. The coupling constants, J, are reported in Hertz (Hz). High resolution mass spectra were collected by positive mode electrospray ionization (ESI) using Waters-Q-TOF-Premier mass spectrometer. IR spectra were recorded on a Perkin Elmer Spectrum 1000 FT-IR spectrometer. Photochemical reactions were performed with 455 nm (OSRAM Oslon SSL 80 royal-blue LEDs ($\lambda = 455 \text{ nm} (\pm 15 \text{ nm})$), 3.5 V, 700 mA).

2.1 General procedure for the synthesis of substituted buta-1,4-dienes from alkylbenzenes



To a vial (20 mL) equipped with a magnetic stir bar was charged under open-air atmosphere with alkylbenzenes (1.0 mmol, 1.0 equiv.), ^tBuONO (2.0 mmol, 2.0 equiv.), DDQ (0.3 mmol, 30 mol%), 3 Å molecular sieve (100 mg) and a freshly distilled BTF (5 mL). Under open-air atmosphere, the mixture was stirred few minutes to mix well and then vial was irradiated through the plane bottom side of the vial using a 5W blue LED at a distance of 2 cm at ambient conditions. After the completion (≈20 h, as indicated by disappearance of precursor monitored by TLC), volatiles were evaporated under reduced pressure and then admixed with aqueous NaCl solution (10 mL). The organic matters were extracted with diethyl ether (3 × 10 mL), dried over Na₂SO₄ and evaporated under reduced pressure to yield pale-yellow materials, which was purified by a filtration through short-pad of silica-gel column chromatography using 0 to 10% of ethyl acetate in hexane as eluent. The identity and purity of the product was confirmed by spectroscopic analysis as well as by a comparison with authentic samples spectra, *vide infra*.

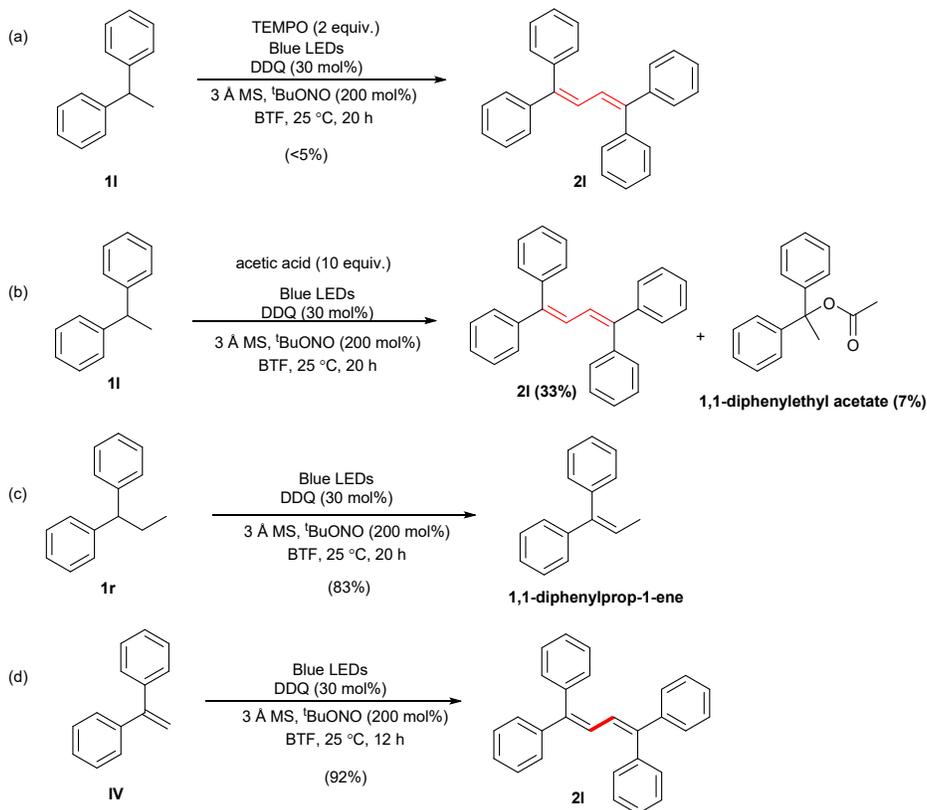
2.2 Procedure for the large-scale synthesis of **2I**



An oven-dried round bottom flask equipped with a magnetic stir bar was charged with 1,1-diphenylethane (**1I**, 1.28g, 7.0 mmol, 1.0 equiv.), ^tBuONO (0.83 mL, 14.0 mmol, 2.0 equiv.), DDQ (0.48g, 0.3 mmol, 30 mol%) and 3 Å molecular sieve (250 mg). To this mixture, a freshly distilled BTF (20 mL) was added and then the flask was covered with a CaCl₂ guard tube. Resultant mixture was stirred few minutes to mix well and then round bottom was irradiated using 5W blue LEDs at a distance of 2 cm at ambient conditions for 20 h. Afterwards, solvent was removed under reduced pressure and the residue was then admixed with cold brine solution (30 mL). The solid precipitate formed was collected by filtration and washed with cold water (10

mL). Finally, product was purified by recrystallization using ethyl acetate and methanol mixture to afford **2I** as a colorless solid in 91% (1.14 g) yield.

2.3 Procedure for control experiments



a) TEMPO (2 equiv. 1.0 mmol, 156 mg) was added to the standard reaction conditions. Resultant mixture was stirred few minutes to mix well and then round bottom was irradiated using 5W blue LEDs at a distance of 2 cm at ambient conditions for 20 h. Solvent was afterwards removed under reduced pressure. TLC, GC and NMR analysis of the residue showed no desired product (**2I**); instead, 36% TEMPO-adduct was formed.

b) Acetic acid (10 equiv. 5.0 mmol, 0.3 mL) was added to the standard reaction conditions. Resultant mixture was stirred few minutes to mix well and then round bottom was irradiated using 5W blue LEDs at a distance of 2 cm at ambient conditions for 20 h. Then it was quenched by diluted NaHCO₃ solution and extracted with ethyl acetate (3 x 10 mL). Combined organic phase were dried over anhydrous Na₂SO₄ and concentrated. The residue was then purified by recrystallization at low-temperature to afford **2I** (colorless solid, 33%) and 1,1-diphenylethyl acetate (pale-yellow liquid, 7%). The partial decomposition of 1,1-diphenylethyl acetate during

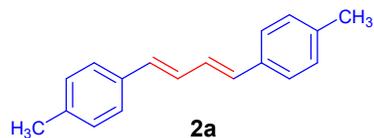
distillation or chromatography resulted in diphenylethylene. **1,1-Diphenylethyl acetate**: ^1H NMR (300MHz, DMSO- d_6): 7.26-7.18 (m, 10H), 2.04 (s, 3H), 1.96 (s, 3H); ^{13}C NMR (75MHz, DMSO- d_6): δ 170.3, 140.4, 128.7, 127.8, 126.1, 80.1, 34.3, 21.5.²

c) Under standard reaction conditions **1r** was used as a precursor. Resultant mixture was stirred few minutes to mix well and then round bottom was irradiated using 5W blue LEDs at a distance of 2 cm at ambient conditions for 20 h. Afterwards, solvent was removed under reduced pressure, treated with brine, extracted and purified by column chromatography on silica gel. 1,1-Diphenylprop-1-ene obtained as a colorless solid (m.p. 49-50 °C) in 83% yield.

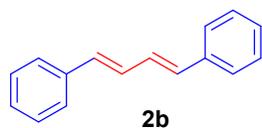
1,1-Diphenylprop-1-ene : ^1H NMR (300MHz, DMSO- d_6) δ 7.35-7.16 (m, 10H), 6.18 (q, $J = 7.2$ Hz, 1H), 1.74 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (75MHz, DMSO- d_6) δ 142.8, 142.4, 140.1, 130.0, 128.2, 128.0, 127.1, 126.9, 126.7, 124.1, 15.6.8.³

d) Under standard reaction conditions 1,1-diphenylethene (**IV**) was used as a precursor. Resultant mixture was stirred few minutes to mix well and then round bottom was irradiated using 5W blue LEDs at a distance of 2 cm at ambient conditions for 12 h. Afterwards, solvent was removed under reduced pressure and the residue was then admixed with cold brine solution (10 mL). The solid precipitate formed was collected by filtration and washed with cold water (10 mL). Finally, product was purified by recrystallization using ethyl acetate and methanol mixture to afford **2l** as a colorless solid in 92% yield.

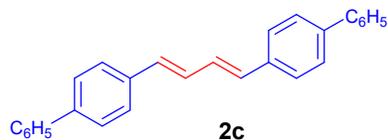
3. Experimental characterization data for products



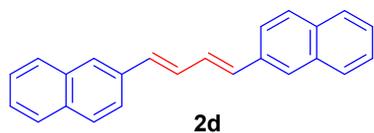
(1E,3E)-1,4-di-*p*-tolylbuta-1,3-diene (2a)⁴: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 90 mg, 76% yield; R_f 0.49 in pet. ether; **IR (KBr, cm^{-1})**: ν 3064, 3035, 2997, 1458, 1374, 1313, 1168, 759; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.36 (d, $J = 8.4$ Hz, 4H), 7.16 (d, $J = 8.4$ Hz, 4H), 6.91 (AA' of AA'BB' pattern, 2H), 6.65 (BB' of AA'BB' pattern, 2H), 2.37(s, 6H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 137.4, 134.7, 132.3, 129.2, 128.6, 126.3, 21.2. **HRMS (ESI)** calcd for $\text{C}_{18}\text{H}_{18}$: $[\text{M} + \text{H}]^+$ 235.1487, found 235.1492.



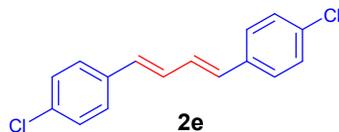
(1E,3E)-1,4-diphenylbuta-1,3-diene (2b)⁴: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 83 mg, 80% yield; R_f 0.53 in pet. ether; **IR (KBr, cm^{-1})**: ν 3066, 2939, 1464, 1349, 1108, 757; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.47-7.43 (m, 4H), 7.36-7.29 (m, 4H), 7.26-7.21 (m, 2H), 6.98 (AA' of AA'BB' pattern, 2H), 6.67 (BB' of AA'BB' pattern, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 137.4, 132.9, 129.4, 128.6, 127.7, 126.4. **HRMS (ESI)** calcd for $\text{C}_{16}\text{H}_{14}$: $[\text{M} + \text{H}]^+$ 207.1174, found 207.1180.



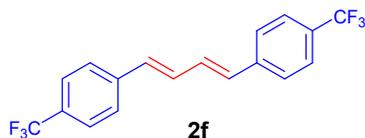
(1E,3E)-1,4-di([1,1'-biphenyl]-4-yl)buta-1,3-diene (2c)⁵: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 131 mg, 73% yield; R_f 0.45 in pet. ether; **IR (KBr, cm^{-1})**: ν 3080, 3067, 1464, 1448, 1433, 786, 734, 689; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.59-7.55 (m, 4H), 7.51-7.40 (m, 10H) 7.39-7.30 (m, 4H), 6.76 (AA' of AA'BB' pattern, 2H), 5.81 (BB' of AA'BB' pattern, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 141.1, 140.9, 136.9, 133.1, 129.7, 129.1, 128.7, 127.9, 127.7, 126.9. **HRMS (ESI)** calcd for $\text{C}_{28}\text{H}_{22}$: $[\text{M}+\text{H}]^+$ 359.1800, found 359.1809.



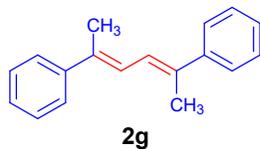
(1E,3E)-1,4-di(naphthalen-2-yl)buta-1,3-diene (2d)⁶: Compound was prepared according to the general procedure. Because of the product's very poor solubility, it was easy to isolate **2d** by filtration. The collected solid was washed with ethyl acetate and isopropanol and dried. Pale-yellow solid; 122 mg, 79% yield; **IR (KBr, cm^{-1})**: ν 3084, 3068, 1466, 1454, 1431, 786, 735, 686; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.86-7.80 (m, 8H), 7.71 (d, $J = 8.8$ Hz, 2H), 7.50-7.43 (m, 4H), 7.20 (AA' of AA'BB', 2H), 6.91 (BB' of AA'BB', 2H). Anal. calcd. for $\text{C}_{24}\text{H}_{18}$: C, 94.08; H, 5.92; found: C, 94.06; H, 5.87. In common organic solvents **2d** was sparingly soluble that precluded collection of $^{13}\text{C NMR}$ spectra.



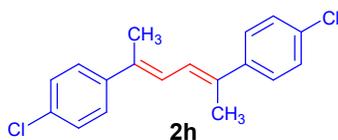
(1E,3E)-1,4-bis(4-chlorophenyl)buta-1,3-diene (2e)⁷: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 94 mg, 68% yield; R_f 0.50 in pet. ether; **IR (KBr, cm^{-1})**: ν 3037, 2994, 2967, 1457, 1433, 1377, 1167, 731, 648; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.36 (d, $J = 7.8$ Hz, 4H), 7.31 (d, $J = 7.8$ Hz, 4H), 6.93 (AA' of AA'BB' pattern, 2H), 6.63 (BB' of AA'BB' pattern, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 135.8, 133.4, 132.0, 130.1, 128.9, 127.7. **HRMS (ESI)** calcd for $\text{C}_{16}\text{H}_{12}\text{Cl}_2$: $[\text{M} + \text{H}]^+$ 275.0394, found 275.0399.



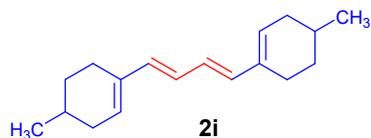
(1E,3E)-1,4-bis(4-(trifluoromethyl)phenyl)buta-1,3-diene (2f)⁶: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 120 mg, 70% yield; R_f 0.44 in pet. ether; **IR (KBr, cm^{-1})**: ν 3039, 2993, 2967, 1458, 1434, 1376, 1295, 1250, 1189, 1165, 733, 648; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.58 (d, $J = 8.2$ Hz, 4H), 7.52 (d, $J = 8.2$ Hz, 4H) 7.01 (AA' of AA'BB' pattern, 2H), 6.73 (BB' of AA'BB' pattern, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 140.4 (q, $J = 1.4$ Hz), 132.8, 131.0, 129.6 (q, $J = 32.4$ Hz), 126.6, 125.7 (q, $J = 3.8$ Hz), 124.1 (q, $J = 272.6$ Hz). **HRMS (ESI)** calcd for $\text{C}_{18}\text{H}_{12}\text{F}_6$: $[\text{M} + \text{H}]^+$ 343.0921, found 343.0927.



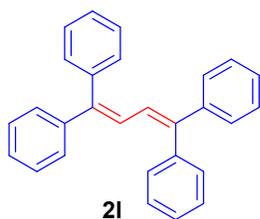
(2E,4E)-hexa-2,4-diene-2,5-diyl dibenzene (2g)⁸: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 103 mg, 87% yield; R_f 0.49 in pet. ether; **IR (KBr, cm^{-1})**: ν 3045, 2932, 2854, 1454, 1072, 827, 749; **$^1\text{H NMR}$ (DMSO- d_6 , 300 MHz)** δ 7.38-7.35 (m, 10H), 6.72 (s, 2H), 2.22 (s, 6H); **$^{13}\text{C NMR}$ (DMSO- d_6 , 75 MHz)** δ 141.1, 133.2, 129.5, 128.6, 127.8, 126.3, 21.8. **HRMS (ESI)** calcd for $\text{C}_{18}\text{H}_{18}$: $[\text{M} + \text{H}]^+$ 235.1487, found 235.1491.



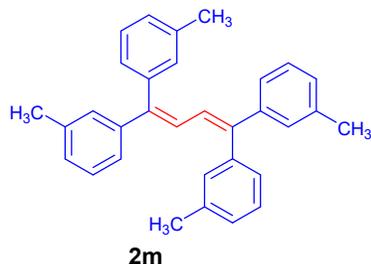
4,4'-((2E,4E)-hexa-2,4-diene-2,5-diyl)bis(chlorobenzene) (2h): Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 130 mg, 86% yield; R_f 0.50 in pet. ether; **IR (KBr, cm^{-1})**: ν 3047, 2933, 2856, 1454, 1308, 1073, 829; **$^1\text{H NMR}$ (DMSO- d_6 , 300 MHz)** δ 7.39 (d, $J = 7.8$ Hz, 4H), 7.32 (d, $J = 7.8$ Hz, 4H), 6.71 (s, 2H), 2.18 (s, 6H); **$^{13}\text{C NMR}$ (DMSO- d_6 , 75 MHz)** δ 140.9, 133.3, 131.9, 130.1, 128.7, 127.8, 21.9. **HRMS (ESI)** calcd for $\text{C}_{18}\text{H}_{16}\text{Cl}_2$: $[\text{M} + \text{H}]^+$ 303.0707, found 303.0713.



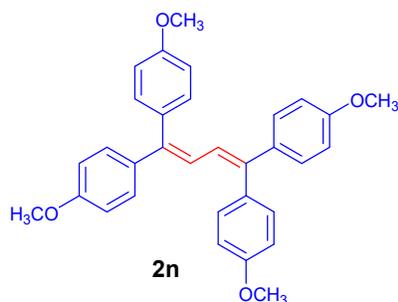
(1E,3E)-1,4-bis(4-methylcyclohex-1-en-1-yl)buta-1,3-diene (2i): Compound was prepared according to the general procedure. The crude product was purified over a silica-gel column chromatography. Pale-yellow liquid; 96 mg, 79% yield; R_f 0.61 in pet. ether; $^1\text{H NMR}$ (DMSO- d_6 , 300 MHz) δ 6.78-6.73 (m, 1H), 5.65-5.62 (m, 1H), 5.23-5.21 (m, 1H), 5.19-5.16 (m, 1H), 2.29-2.24 (m, 4H), 2.18-2.08 (m, 2H), 1.86-1.78 (m, 4H), 1.72-1.69 (m, 2H), 1.30-1.26 (m, 4H), 0.89 (d, $J = 6.6$ Hz, 6H); $^{13}\text{C NMR}$ (DMSO- d_6 , 75 MHz) δ 148.1, 138.4, 134.5, 114.4, 34.9, 30.4, 28.3, 24.2, 21.5. **HRMS (ESI)** calcd for $\text{C}_{18}\text{H}_{26}$: $[\text{M} + \text{H}]^+ 243.2113$, found 243.2122.



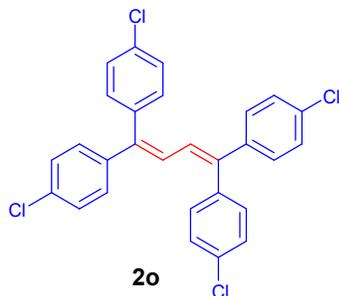
1,1,4,4-tetraphenylbuta-1,3-diene (2l)⁸: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 160 mg, 89% yield; R_f 0.31 in pet. ether; **IR (KBr, cm^{-1}):** ν 3054, 2923, 2852, 1484, 1449, 756; $^1\text{H NMR}$ (DMSO- d_6 , 300 MHz) δ 7.47-7.37 (m, 6H), 7.34-7.29 (m, 4H), 7.26-7.19 (m, 6H), 7.18-7.11 (m, 4H), 6.76 (s, 2H); $^{13}\text{C NMR}$ (DMSO- d_6 , 75 MHz) δ 144.5, 142.9, 140.3, 131.1, 128.7, 128.5, 128.0, 127.9, 127.8, 126.4. **HRMS (ESI)** calcd for $\text{C}_{28}\text{H}_{22}$: $[\text{M} + \text{H}]^+ 359.1800$, found 359.1807.



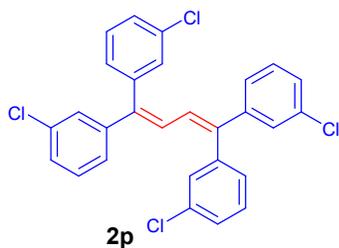
1,1,4,4-tetra-*m*-tolylbuta-1,3-diene (2m)⁸: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless solid; 189 mg, 91% yield; R_f 0.24 in pet. ether; **IR (KBr, cm^{-1})**: ν 3053, 2928, 2854, 1457, 1082, 835; **^1H NMR** (DMSO- d_6 , 300 MHz) δ 7.48-7.44 (m, 4H), 7.31-7.20 (m, 8H), 7.11-7.02 (m, 4H), 6.78 (s, 2H), 1.57 (s, 6H), 1.56 (s, 6H); **^{13}C NMR** (DMSO- d_6 , 75 MHz) δ 144.1, 143.0, 140.6, 139.6, 138.2, 130.7, 130.3, 129.0, 128.2, 128.1, 127.2, 126.3, 126.1, 125.6, 21.4, 21.3. **HRMS (ESI)** calcd for $\text{C}_{32}\text{H}_{30}$: $[\text{M} + \text{H}]^+ 415.2426$, found 415.2431.



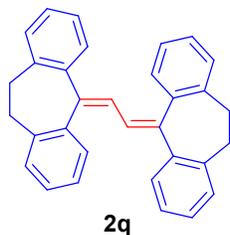
1,1,4,4-tetrakis(4-methoxyphenyl)buta-1,3-diene (2n)⁹: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 173 mg, 72% yield; R_f 0.29 in EtOAc-pet. ether (5:95); **IR (KBr, cm^{-1})**: ν 2926, 2856, 1451, 1433, 833; **^1H NMR** (DMSO- d_6 , 300 MHz) δ 7.29 (d, $J = 9.4$ Hz, 4H), 7.13 (d, $J = 8.6$ Hz, 4H), 6.96 (d, $J = 8.6$ Hz, 4H), 6.81 (d, $J = 9.4$ Hz, 4H), 6.67 (s, 2H), 3.86 (s, 6H), 3.81 (s, 6H); **^{13}C NMR** (DMSO- d_6 , 75 MHz) δ 158.9, 158.5, 142.0, 135.6, 132.4, 131.8, 128.7, 124.7, 113.4, 113.3, 55.38, 55.36. **HRMS (ESI)** calcd for $\text{C}_{32}\text{H}_{30}\text{O}_4$: $[\text{M} + \text{H}]^+ 479.2222$, found 479.2228.



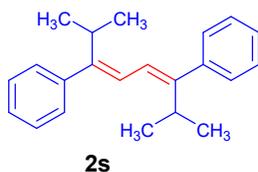
1,1,4,4-tetrakis(4-chlorophenyl)buta-1,3-diene (2o)⁸: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 216 mg, 87% yield, R_f 0.78 in EtOAc-pet. ether (5:95); **IR (KBr, cm^{-1})**: ν 2922, 2853, 2384, 1453, 1432, 836; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.34 (d, $J = 8.8$ Hz, 4H), 7.27-7.20 (m, 8H), 7.18 (d, $J = 8.8$ Hz, 4H), 6.69 (s, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 144.4, 140.6, 137.7, 135.9, 135.6, 132.4, 130.2, 128.8, 128.6, 126.3. **HRMS (ESI)** calcd for $\text{C}_{28}\text{H}_{18}\text{Cl}_4$: $[\text{M} + \text{H}]^+ 495.0241$, found 495.0247.



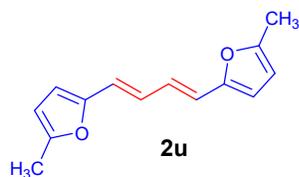
1,1,4,4-tetrakis(3-chlorophenyl)buta-1,3-diene (2p)⁸: Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow solid; 206 mg, 83% yield; R_f 0.26 in pet. ether; **IR (KBr, cm^{-1})**: ν 2926, 2856, 1455, 1429, 1074, 1025, 968; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 7.37 (d, $J = 8.8$ Hz, 2H), 7.31-7.27 (m, 4H), 7.25-7.21 (m, 2H), 7.19-7.13 (m, 8H), 6.76 (s, 2H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 144.2, 143.1, 140.7, 134.2, 134.1, 132.1, 130.7, 129.2, 128.7, 128.6, 127.1, 126.3, 126.2. **HRMS (ESI)** calcd for $\text{C}_{28}\text{H}_{18}\text{Cl}_4$: $[\text{M} + \text{H}]^+ 495.0241$, found 495.0249.



1,2-bis(10,11-dihydro-5H-dibenzo[a,d][7]annulen-5-ylidene)ethane (2q): Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Yellow solid; 182 mg, 88% yield; R_f 0.18 in pet. ether; **IR (KBr, cm^{-1}):** ν 3214, 3125, 3056, 3031, 2928, 2945, 2876, 2839, 1561, 1487, 1451, 897, 758; **^1H NMR** (CD_2Cl_2 , 300 MHz) δ 7.36-7.33 (m, 2H), 7.29-7.15 (m, 12H), 7.09 (d, $J = 7.4$ Hz, 2H), 6.65 (s, 2H), 3.40-3.27 (m, 4H), 3.11-2.83 (m, 4H); **^{13}C NMR** (CD_2Cl_2 , 75 MHz) δ 147.4, 139.2, 138.8, 138.4, 137.6, 130.3, 128.7, 128.5, 128.4, 128.3, 128.2, 126.4, 125.9, 106.2, 33.4, 31.8. **HRMS (ESI)** calcd for $\text{C}_{32}\text{H}_{26}$: $[\text{M} + \text{H}]^+ 411.2113$, found 411.2118.



((3E,5E)-2,7-dimethylocta-3,5-diene-3,6-diyl)dibenzene (2s): Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Colourless gum; 76 mg, 52% yield; R_f 0.49 in pet. ether; **IR (KBr, cm^{-1}):** ν 3062, 2941, 1461, 1355, 1107, 759; **^1H NMR** ($\text{DMSO}-d_6$, 300 MHz) δ 7.46-7.42 (m, 4H), 7.35-7.29 (m, 4H), 7.27-7.21 (m, 2H), 6.69 (s, 2H), 2.54-2.49 (m, 2H), 1.09 (d, $J = 6.8$ Hz, 12H); **^{13}C NMR** ($\text{DMSO}-d_6$, 75 MHz) δ 140.2, 132.8, 129.4, 128.5, 127.7, 126.2, 31.4, 18.7. **HRMS (ESI)** calcd for $\text{C}_{22}\text{H}_{26}$: $[\text{M} + \text{H}]^+ 291.2113$, found 291.2119.



(1E,3E)-1,4-bis(5-methylfuran-2-yl)buta-1,3-diene (2u): Compound was prepared according to the general procedure. The crude product was purified by a filtration through short-pad of silica-gel column chromatography. Pale-yellow oil; 81 mg, 75% yield; R_f 0.46 in pet. ether; **IR (KBr, cm^{-1}):** ν 2964, 2926, 1604, 1510, 1461, 1440, 1366, 1234, 1215, 1146, 1083, 1015, 917, 728; **$^1\text{H NMR}$** (DMSO- d_6 , 300 MHz) δ 6.94 (AA' of AA'BB' pattern, 2H), 6.65 (BB' of AA'BB' pattern, 2H), 6.24-6.19 (m, 2H), 5.95-5.91 (m, 2H), 2.26 (s, 6H); **$^{13}\text{C NMR}$** (DMSO- d_6 , 75 MHz) δ 152.2, 149.1, 129.6, 114.2, 110.4, 105.5, 13.5. **HRMS (ESI)** calcd for $\text{C}_{14}\text{H}_{14}\text{O}_2$: $[\text{M} + \text{H}]^+ 215.1072$, found 215.1078.

4. References:

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5. Copies of ^1H and ^{13}C NMR spectra

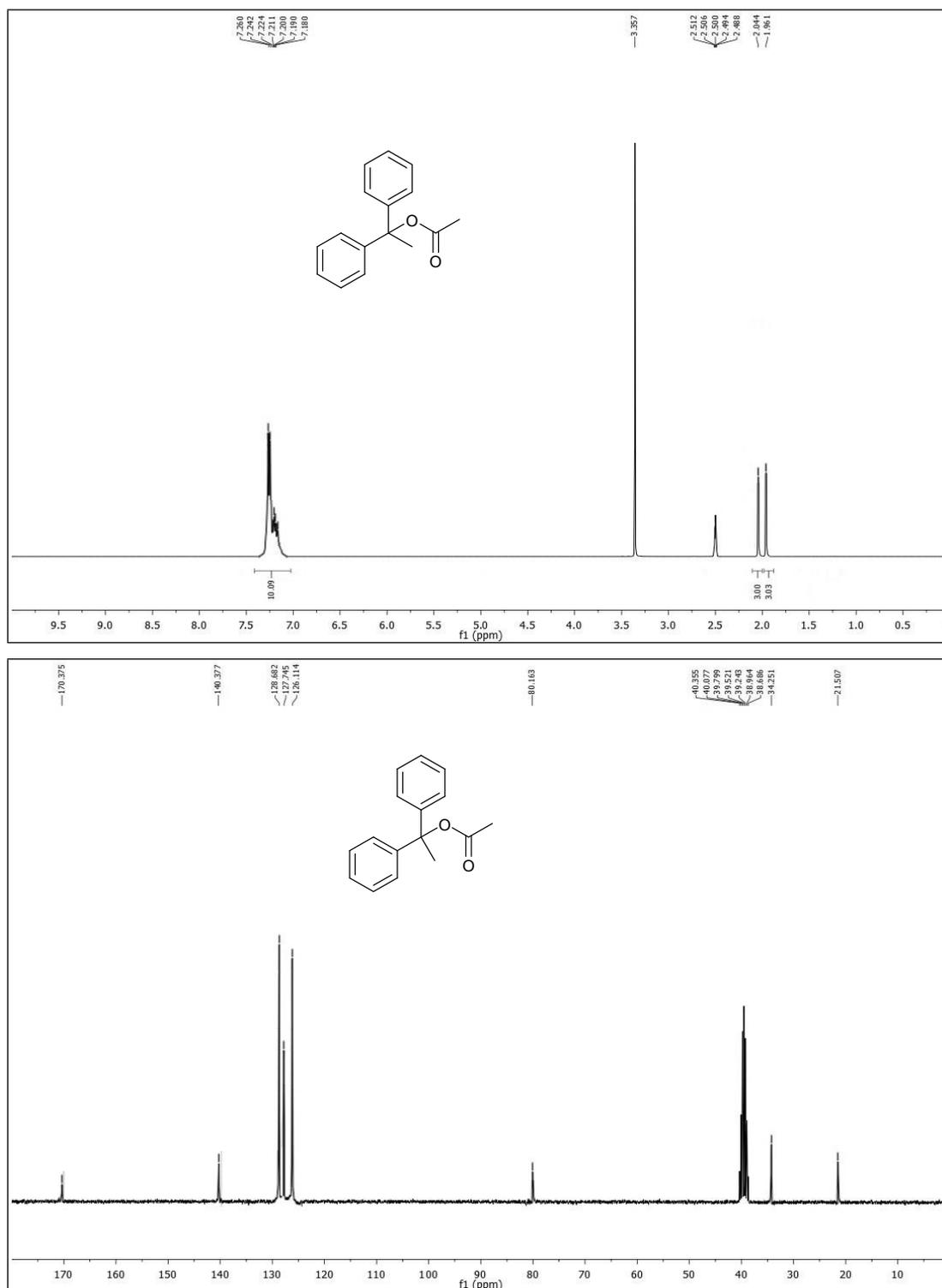


Figure S1. ^1H (top) and ^{13}C (bottom, 75 MHz) NMR spectra of 1,1-diphenylethyl acetate in DMSO-d_6 .

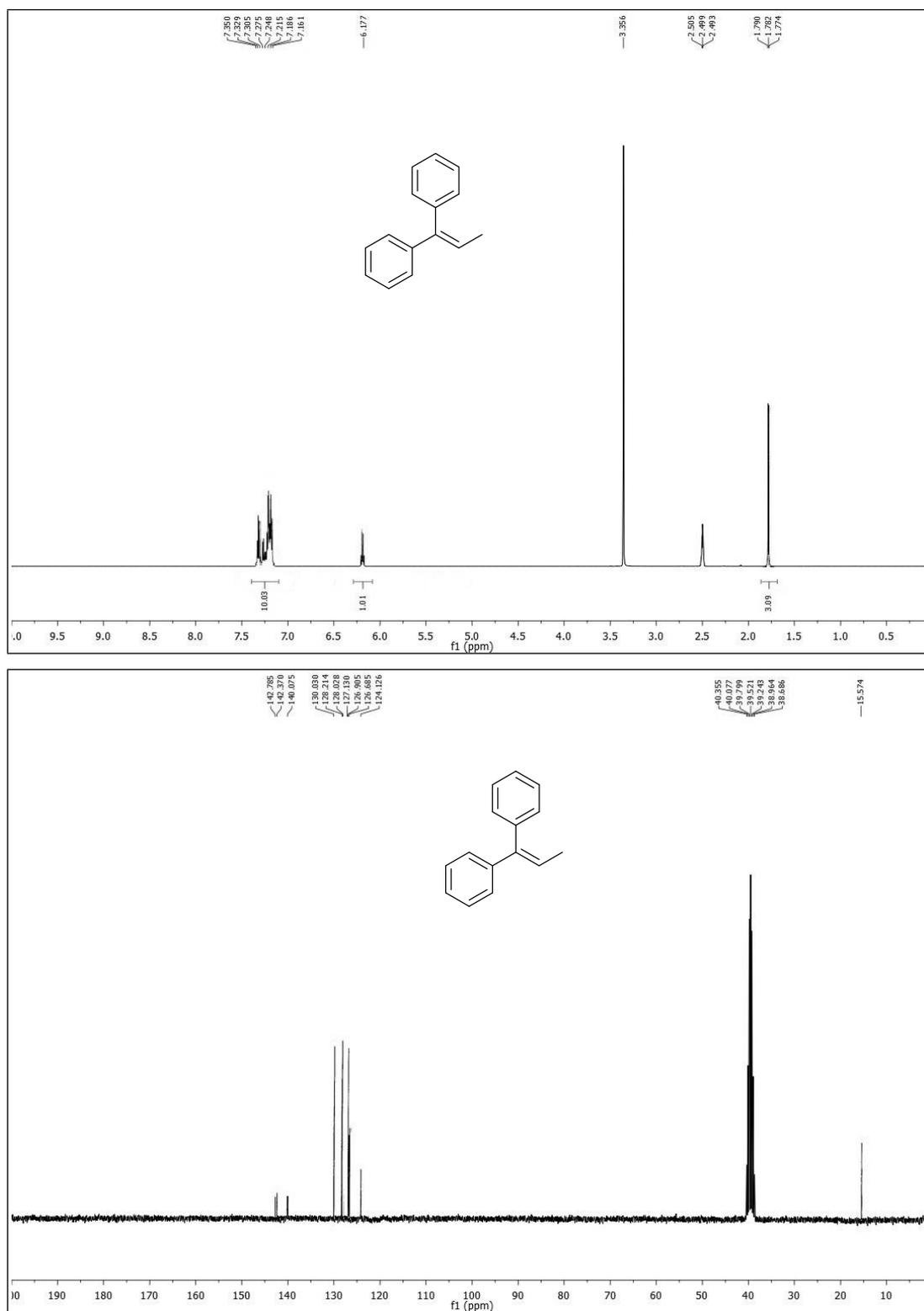


Figure S2. ¹H (top) and ¹³C (bottom, 75 MHz) NMR spectra of 1,1-diphenylprop-1-ene in DMSO-d₆.

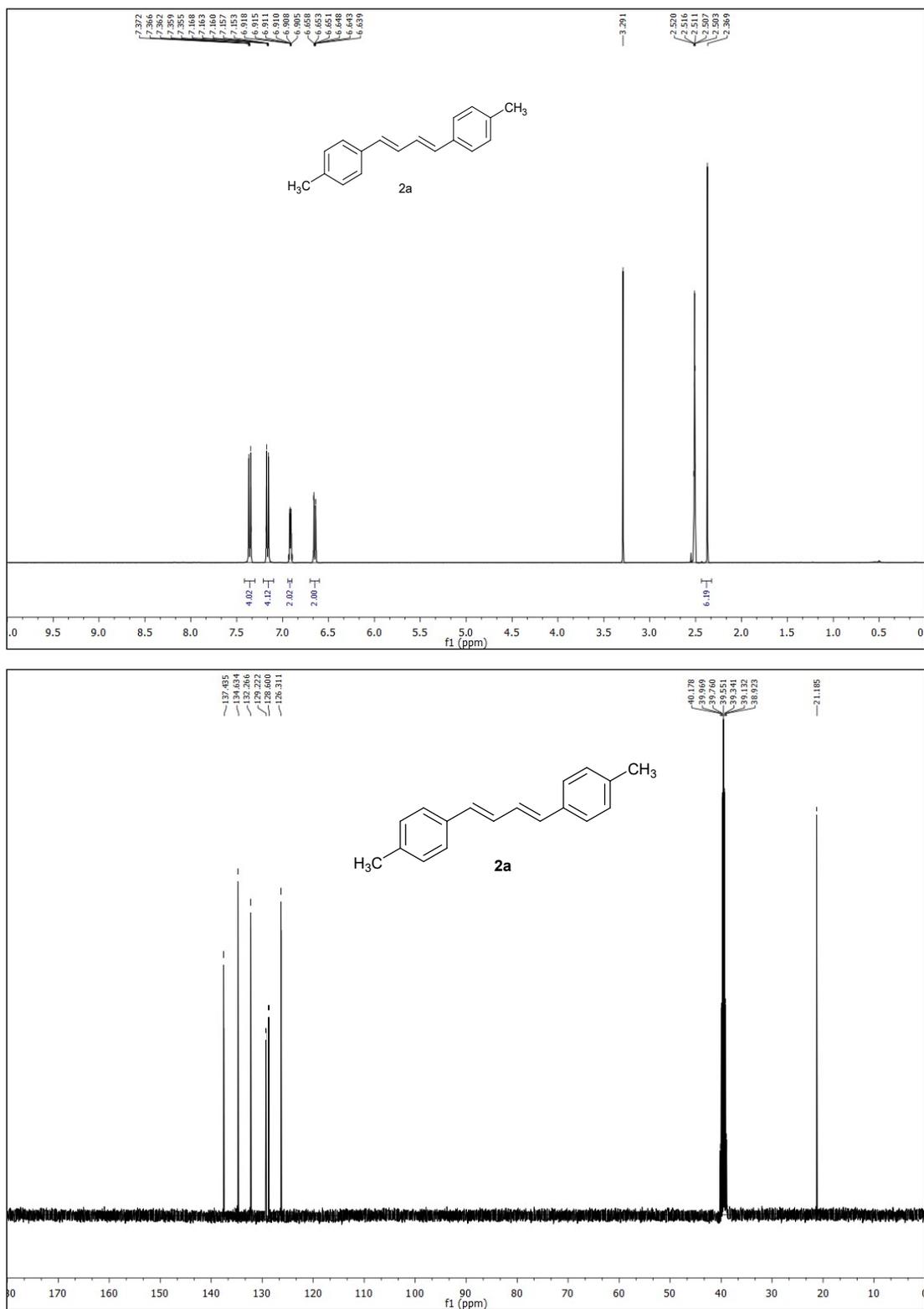


Figure S3. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2a** in DMSO-d₆.

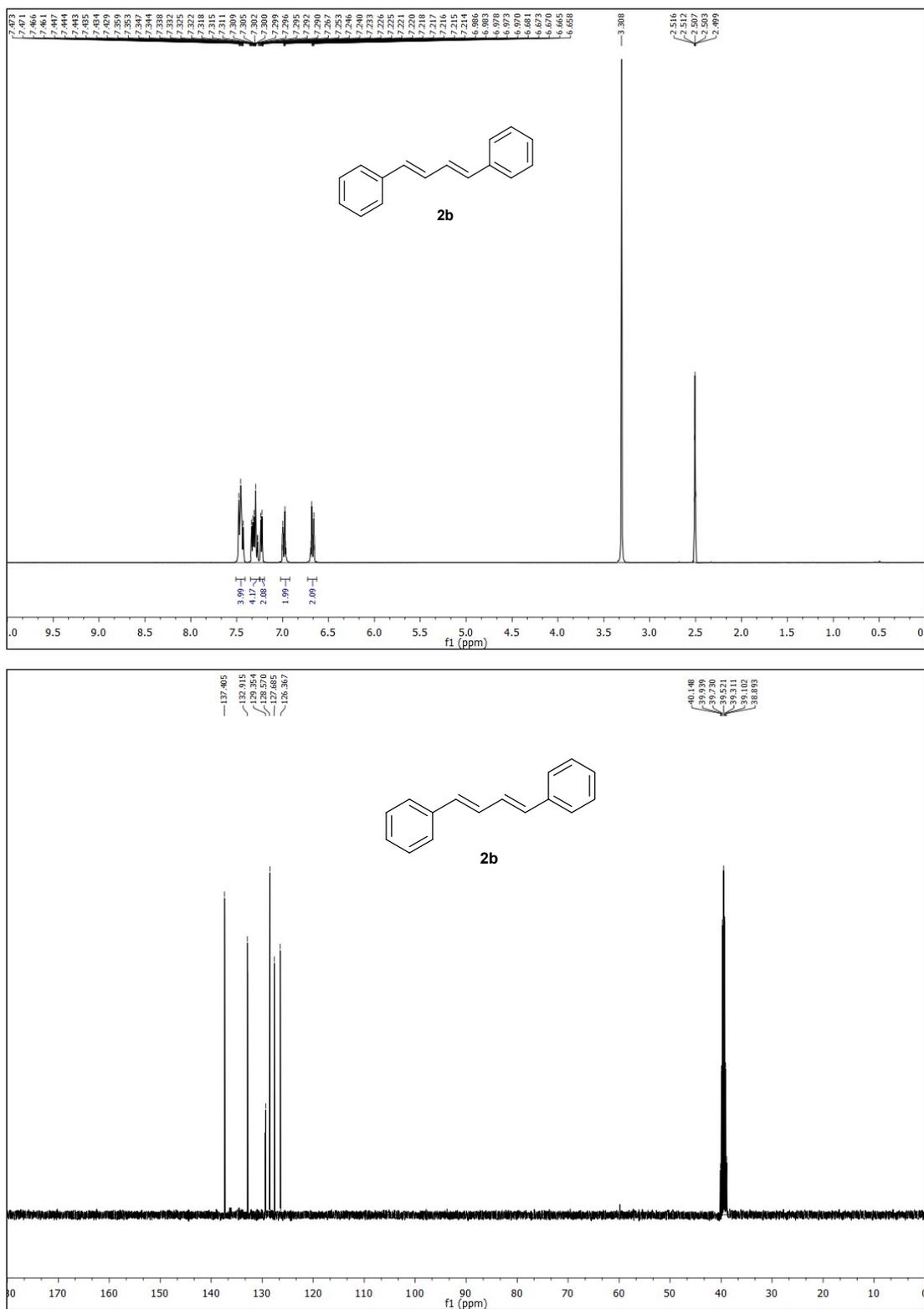


Figure S4. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2b** in DMSO-d₆.

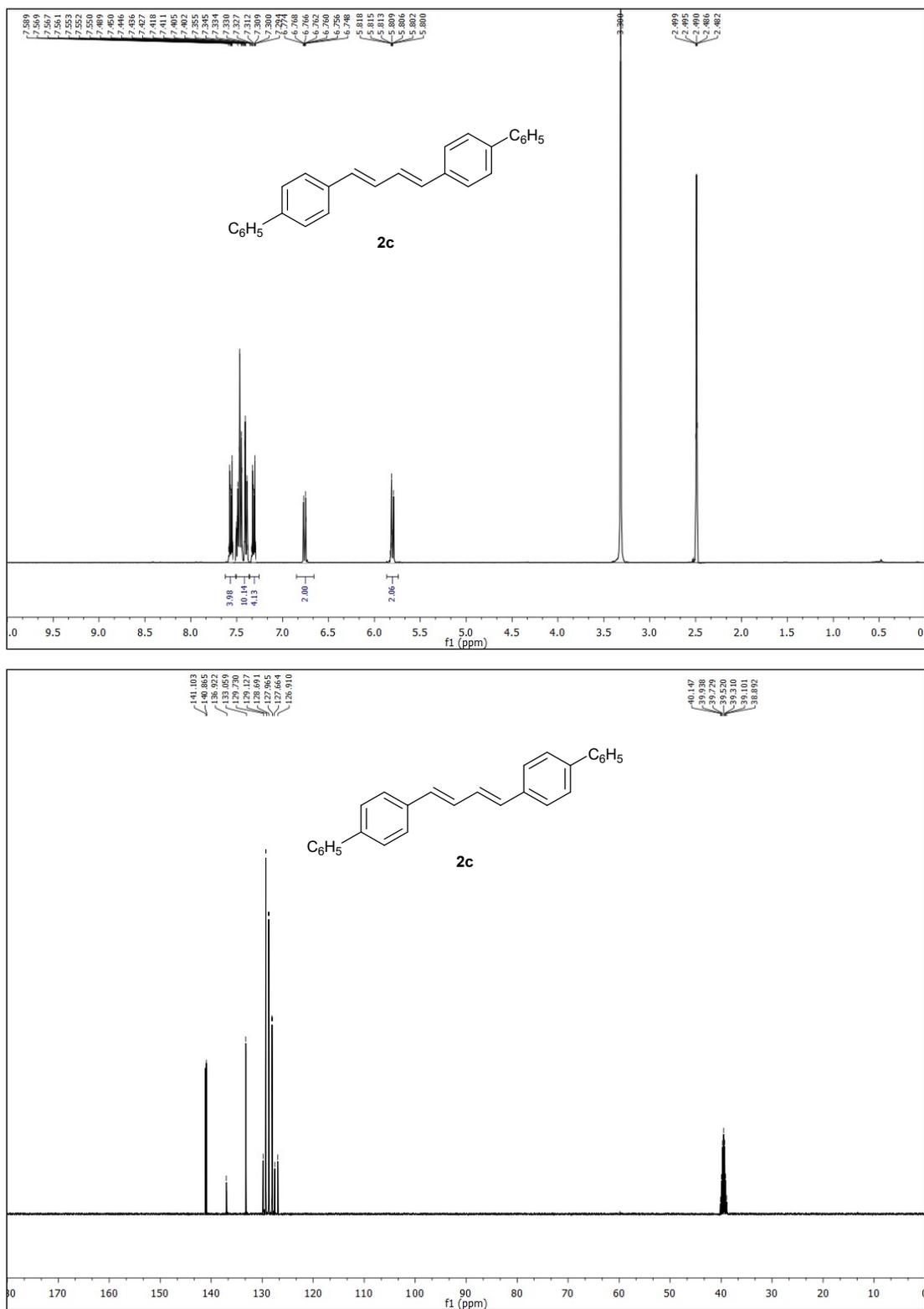


Figure S5. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2c** in DMSO-d₆.

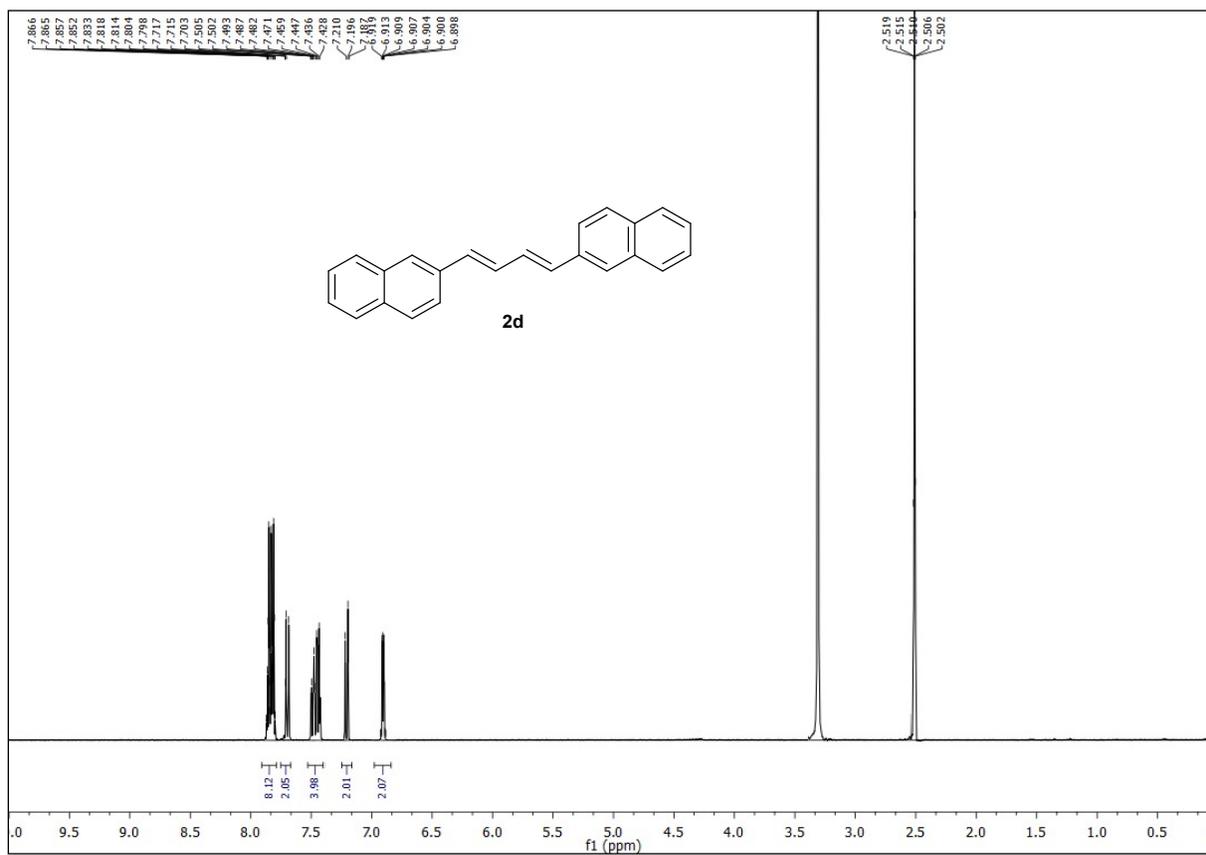


Figure S6. ^1H (300 MHz) NMR spectra of **2d** in DMSO-d_6 .

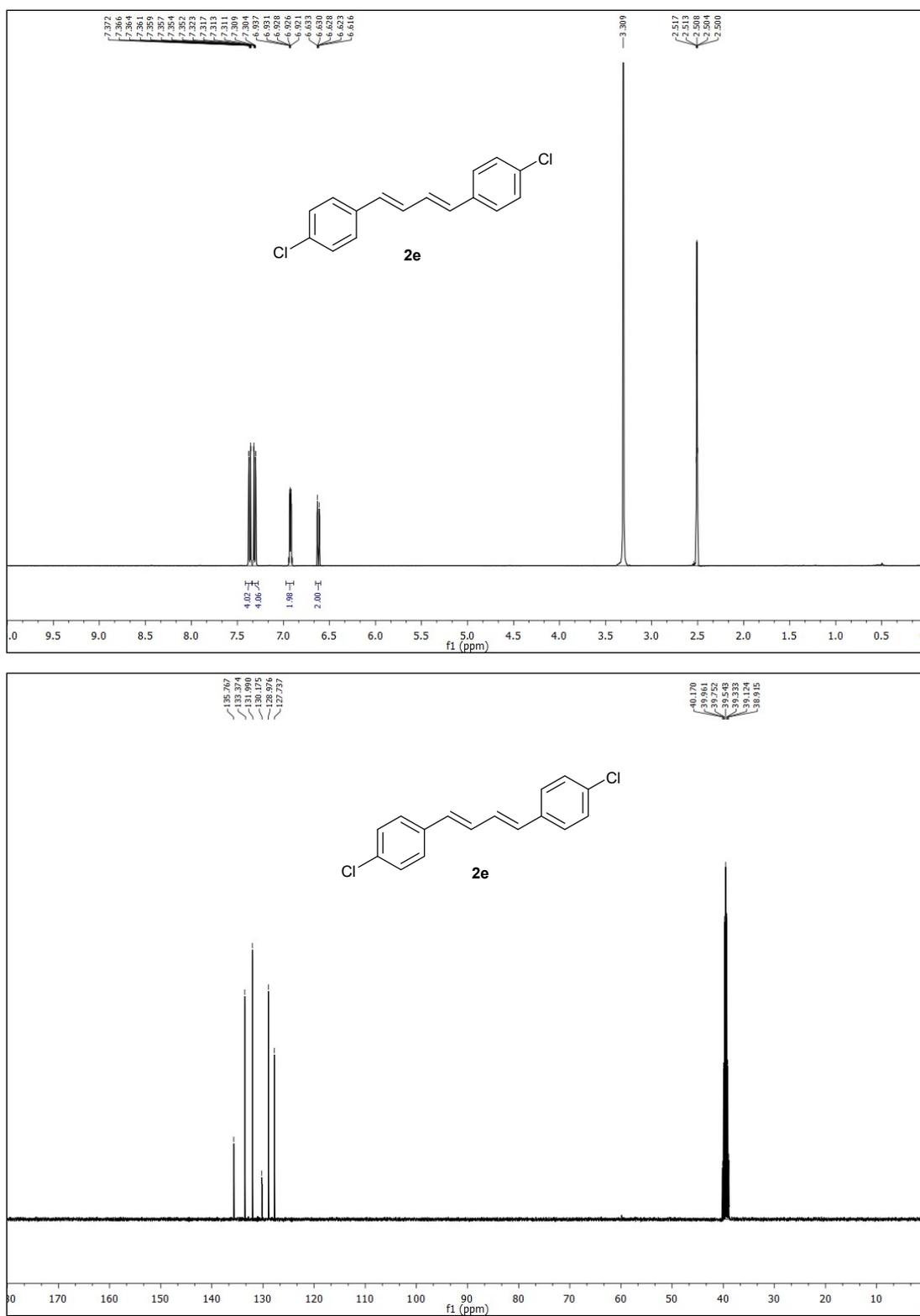


Figure S7. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2e** in DMSO-d₆.

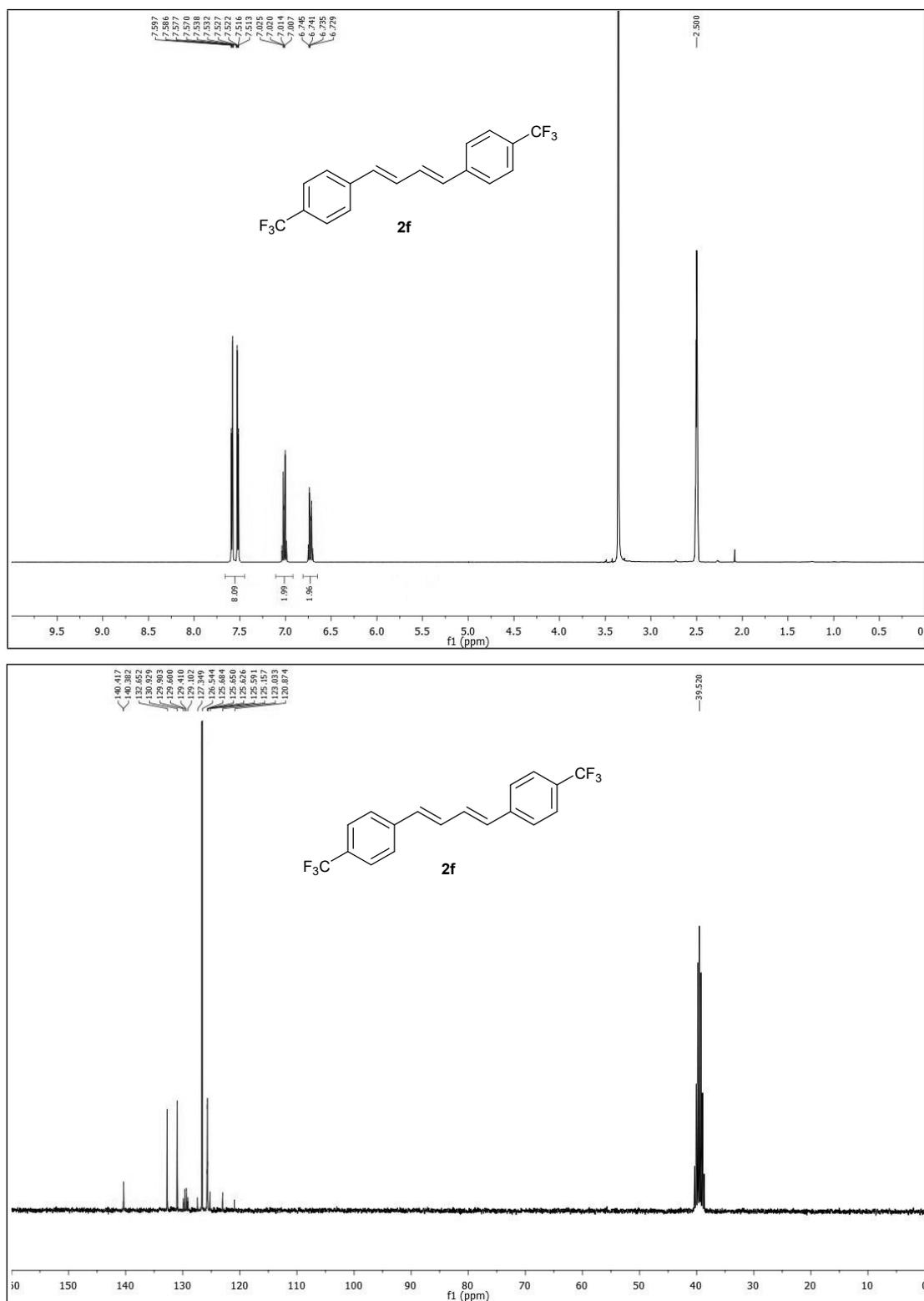


Figure S8. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2f** in DMSO-d₆.

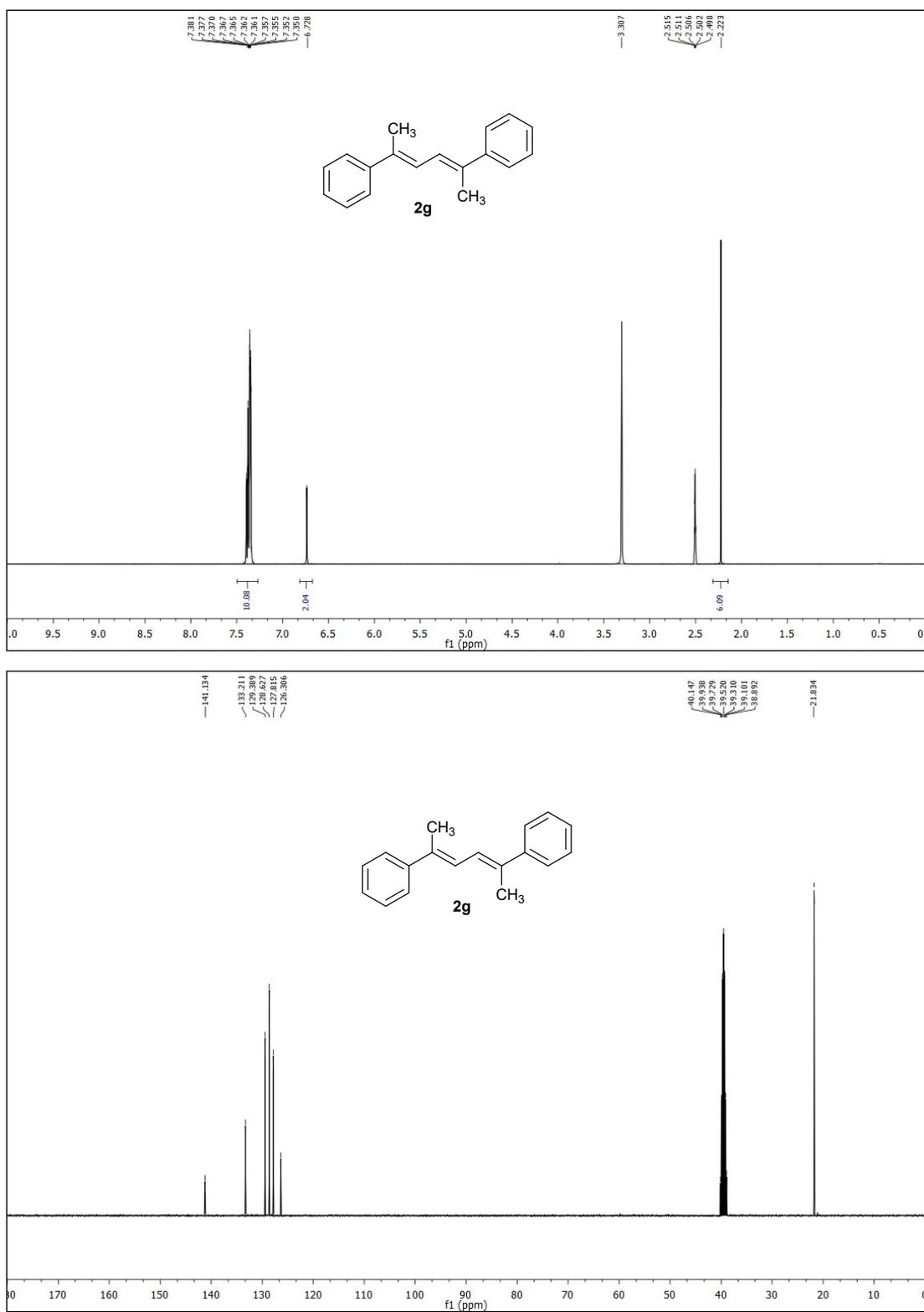


Figure S9. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2g** in DMSO-d₆.

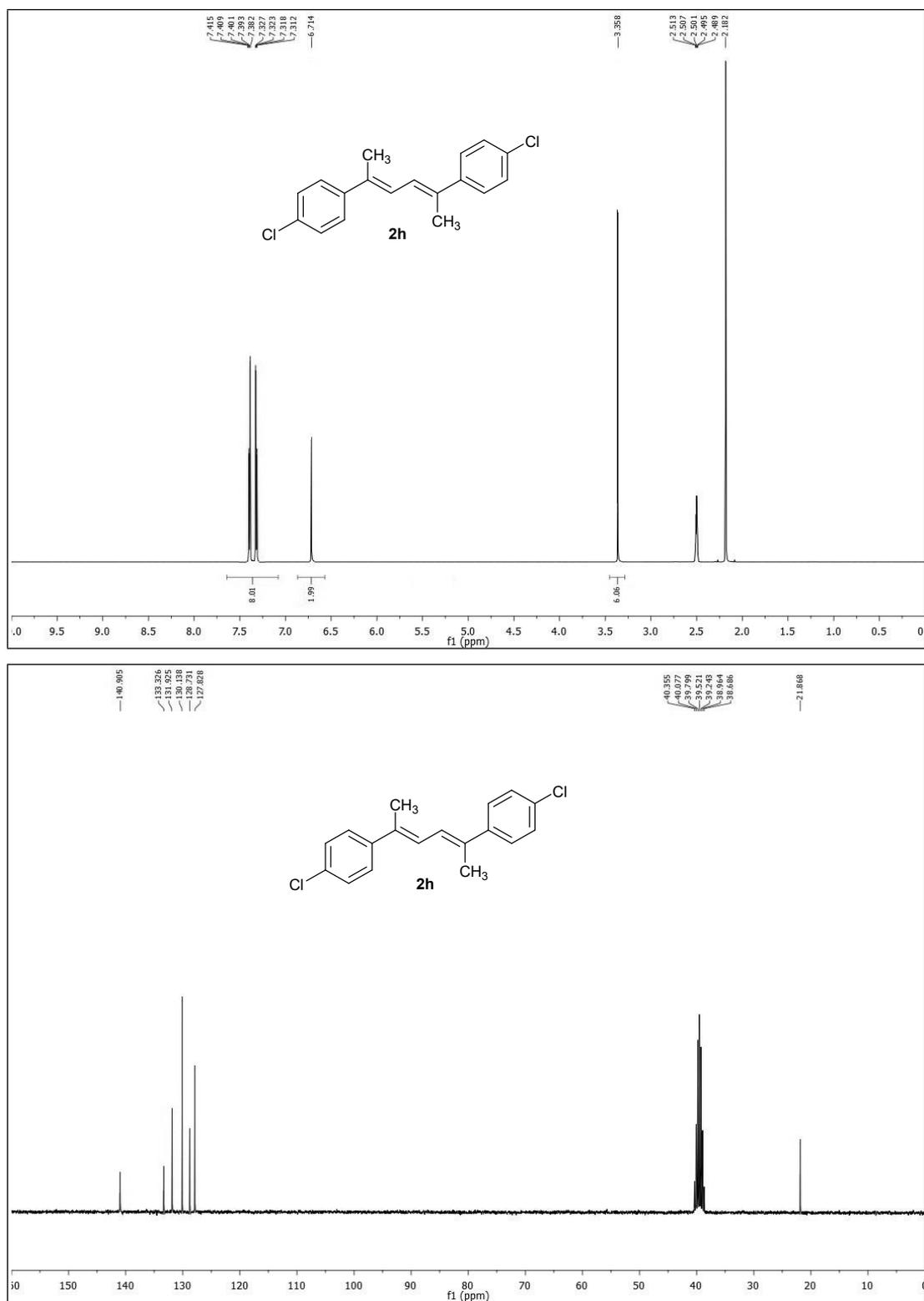


Figure S10. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2h** in DMSO-d₆.

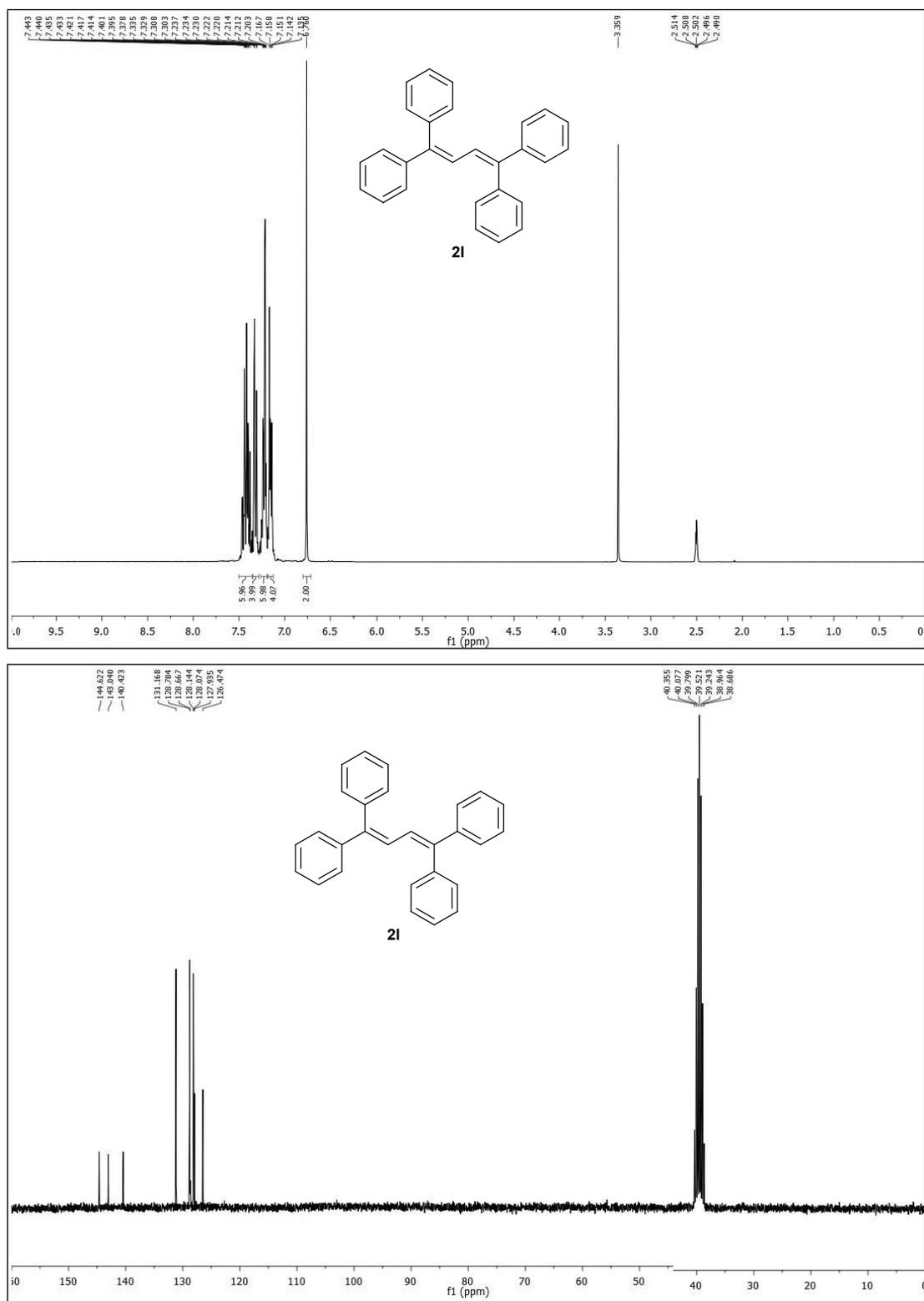


Figure S11. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2i** in DMSO-d₆.

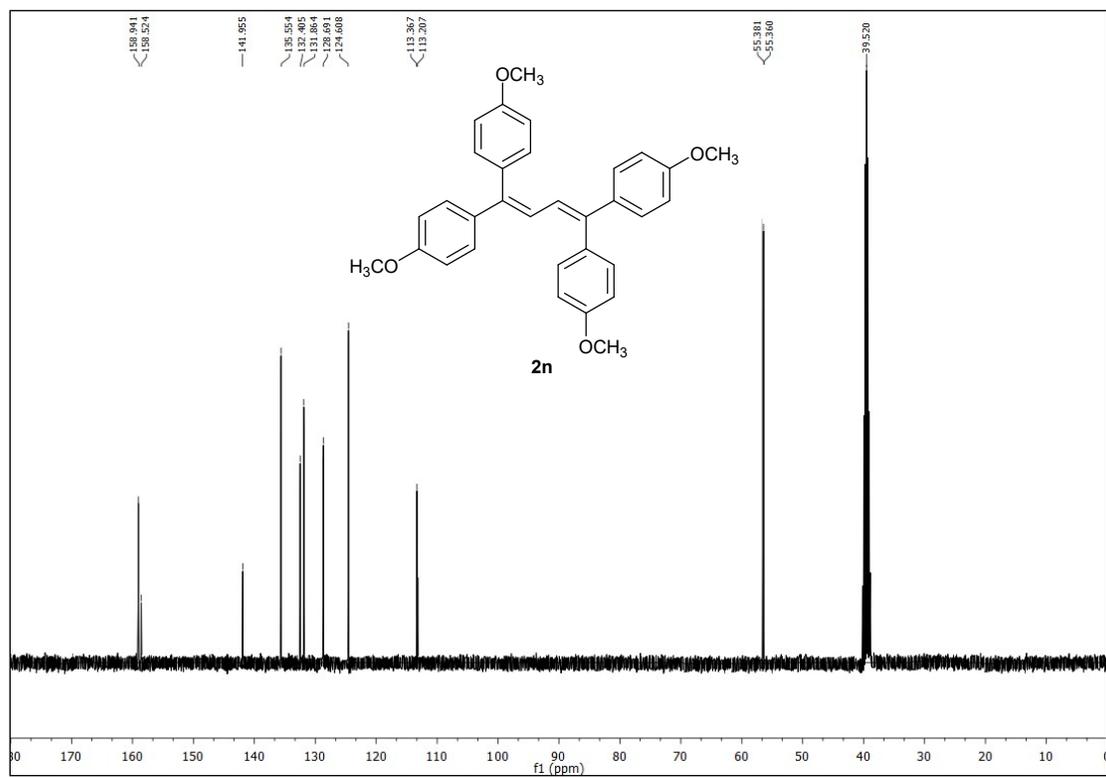
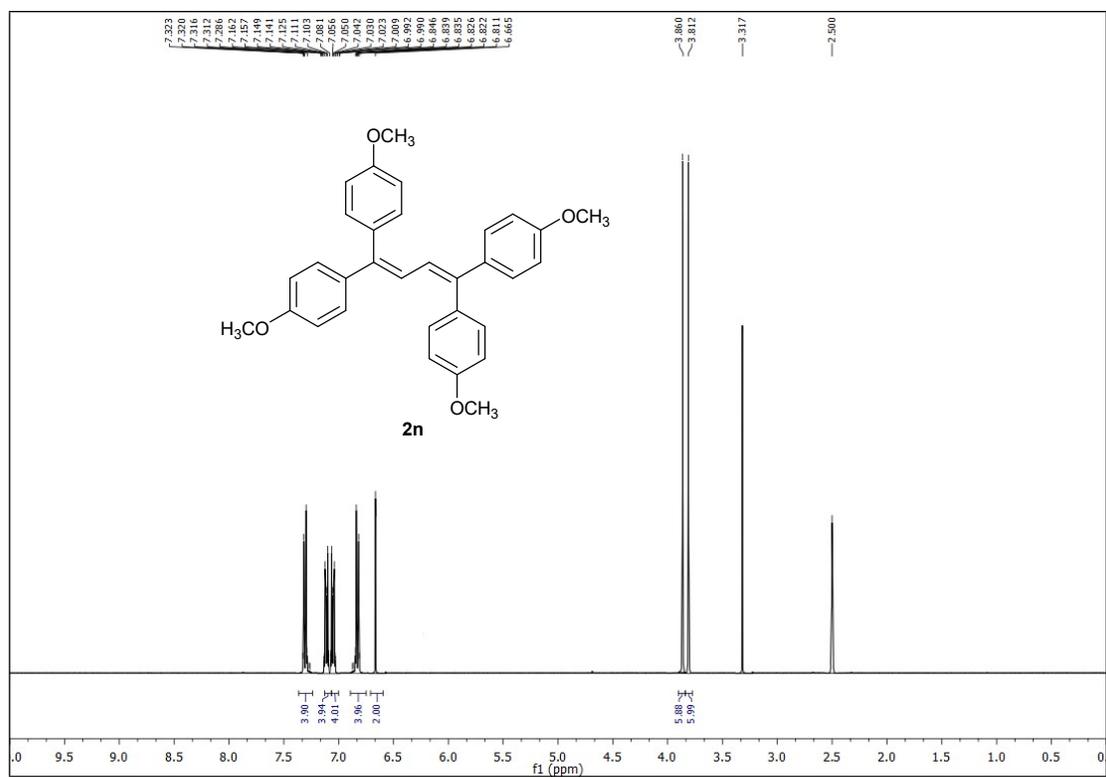


Figure S13. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2n** in DMSO-d₆.

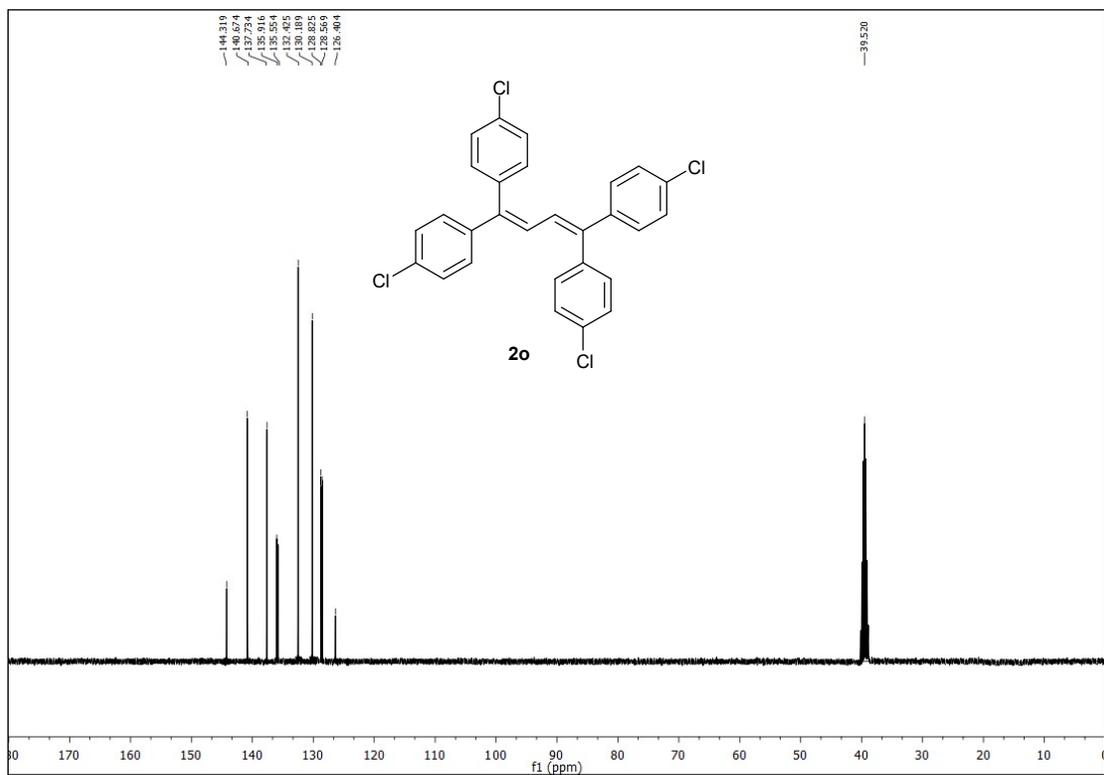
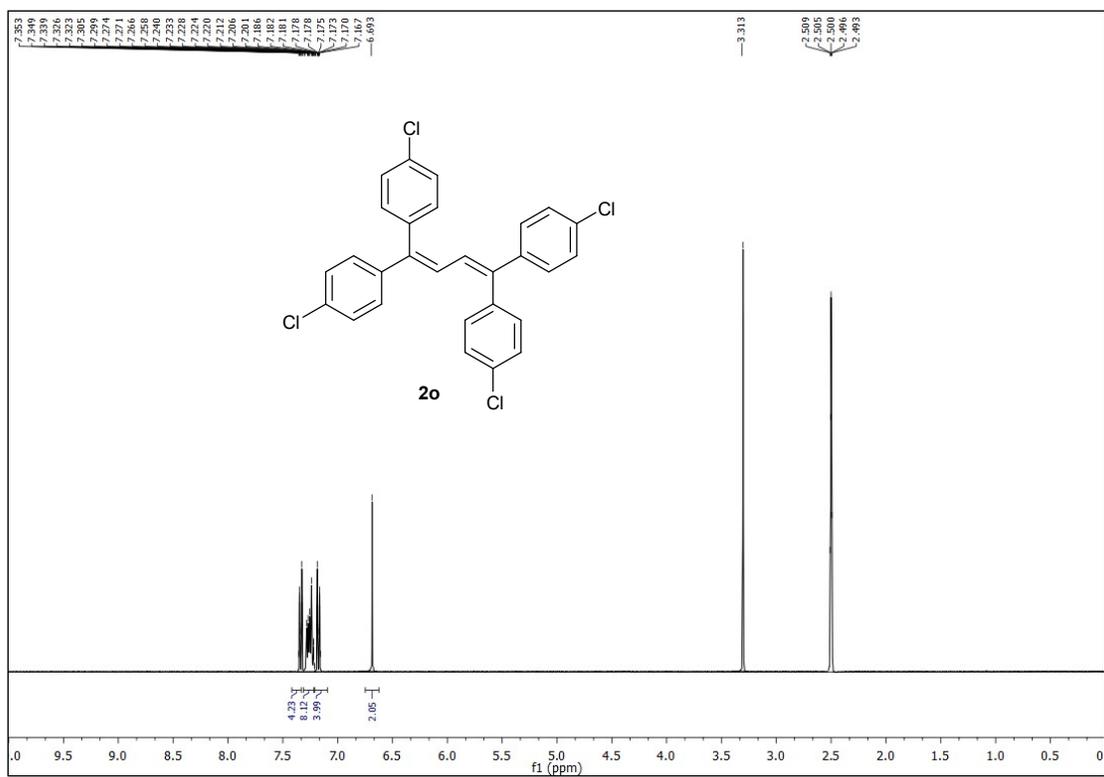


Figure S14. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2o** in DMSO-d₆.

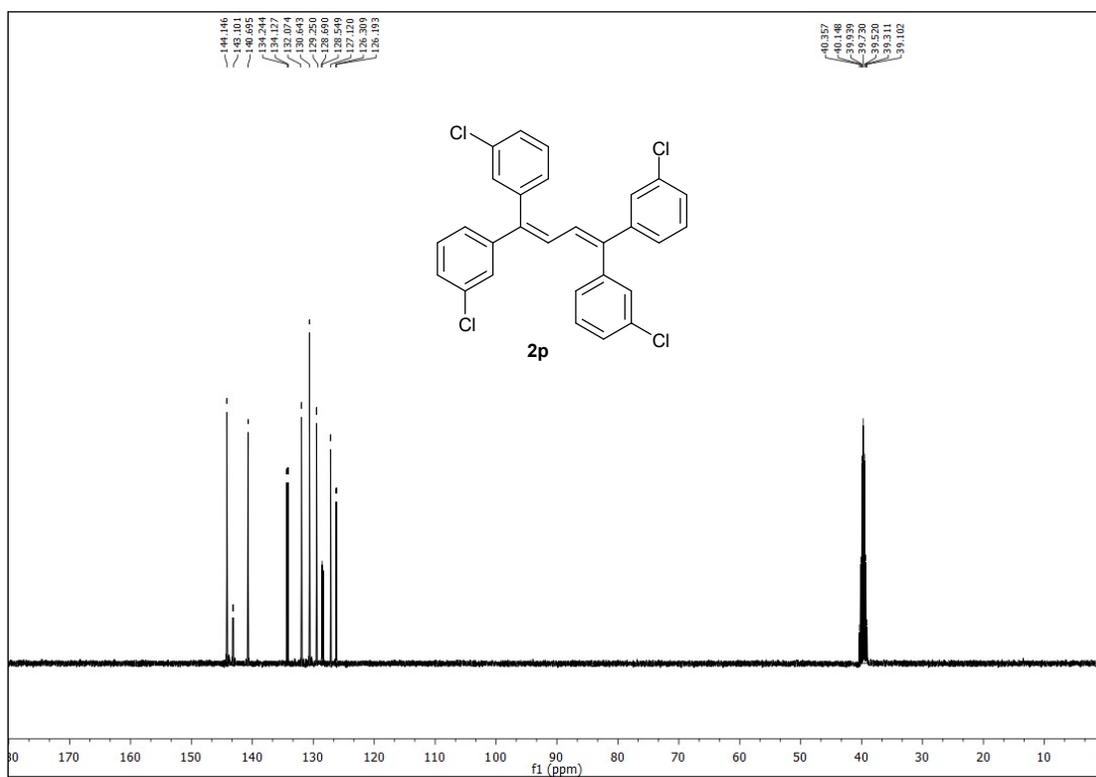
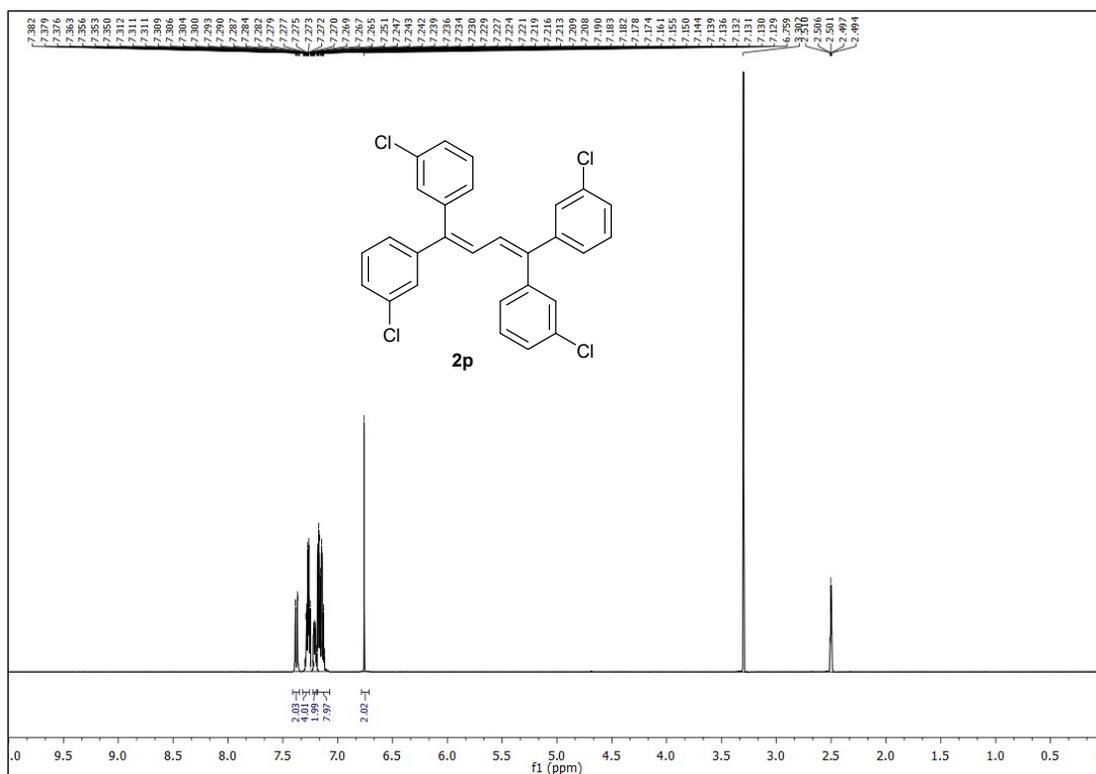


Figure S15. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2p** in DMSO-d₆.

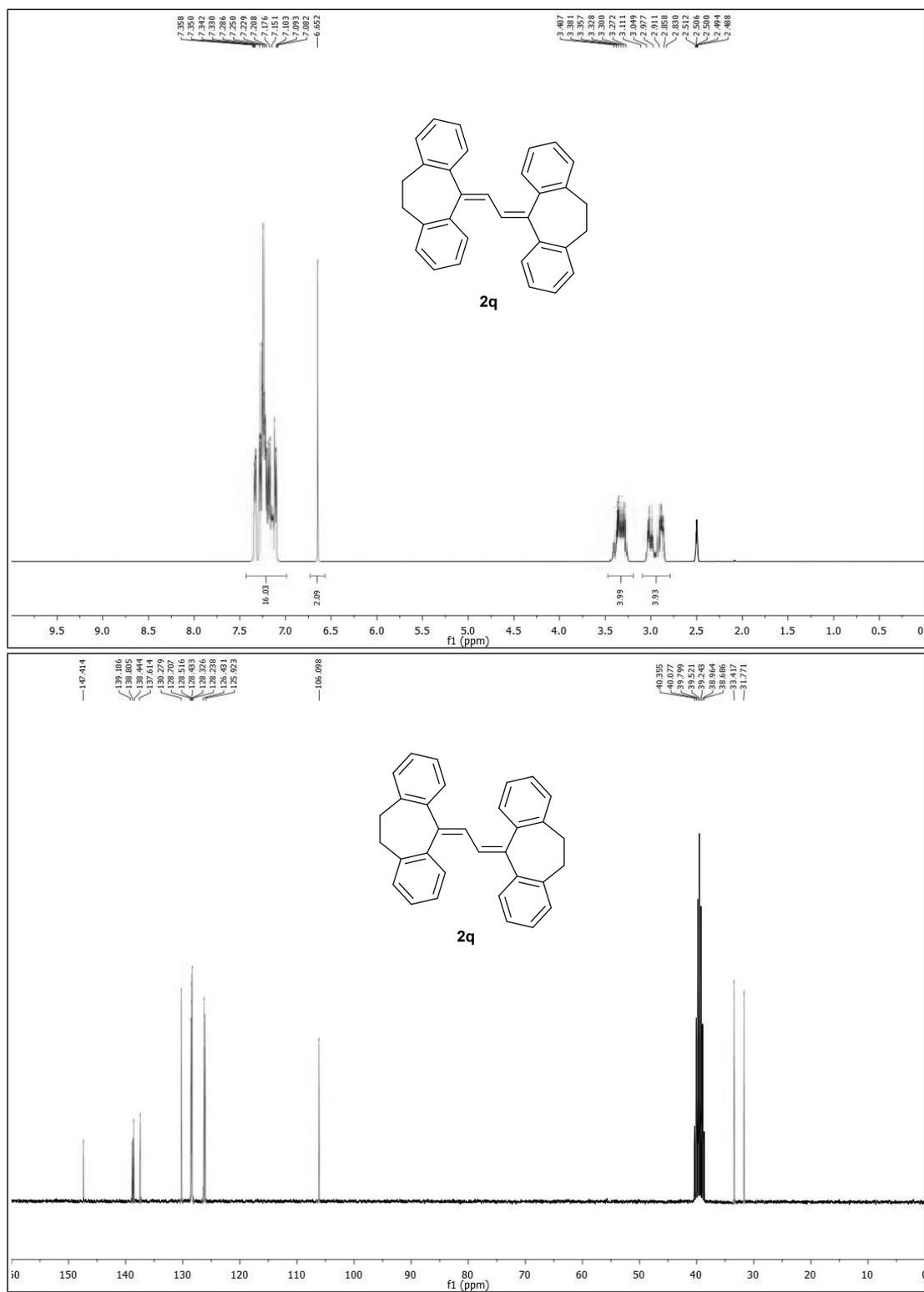


Figure S16. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2q** in DMSO-d₆.

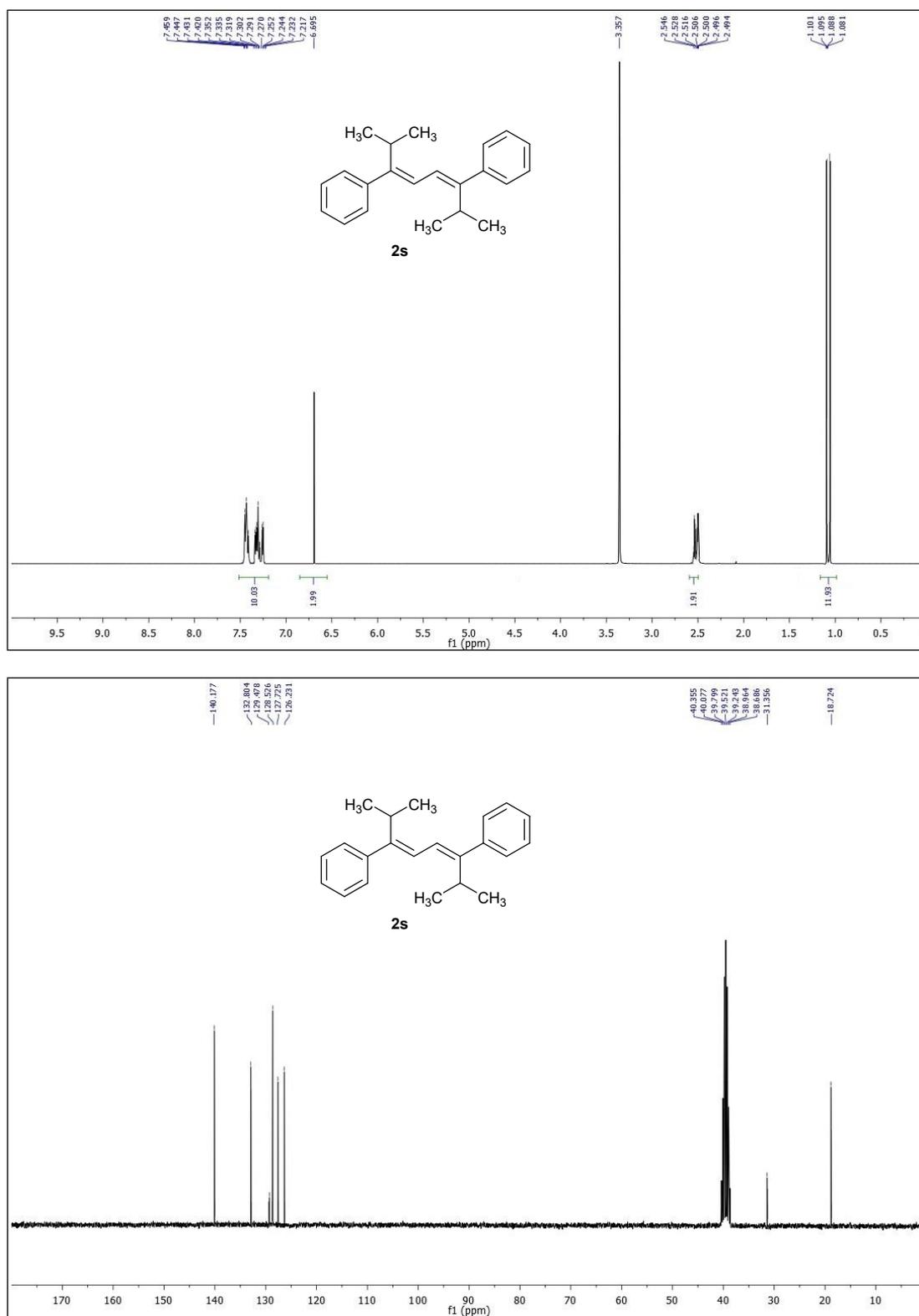


Figure S17. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2s** in DMSO-d₆.

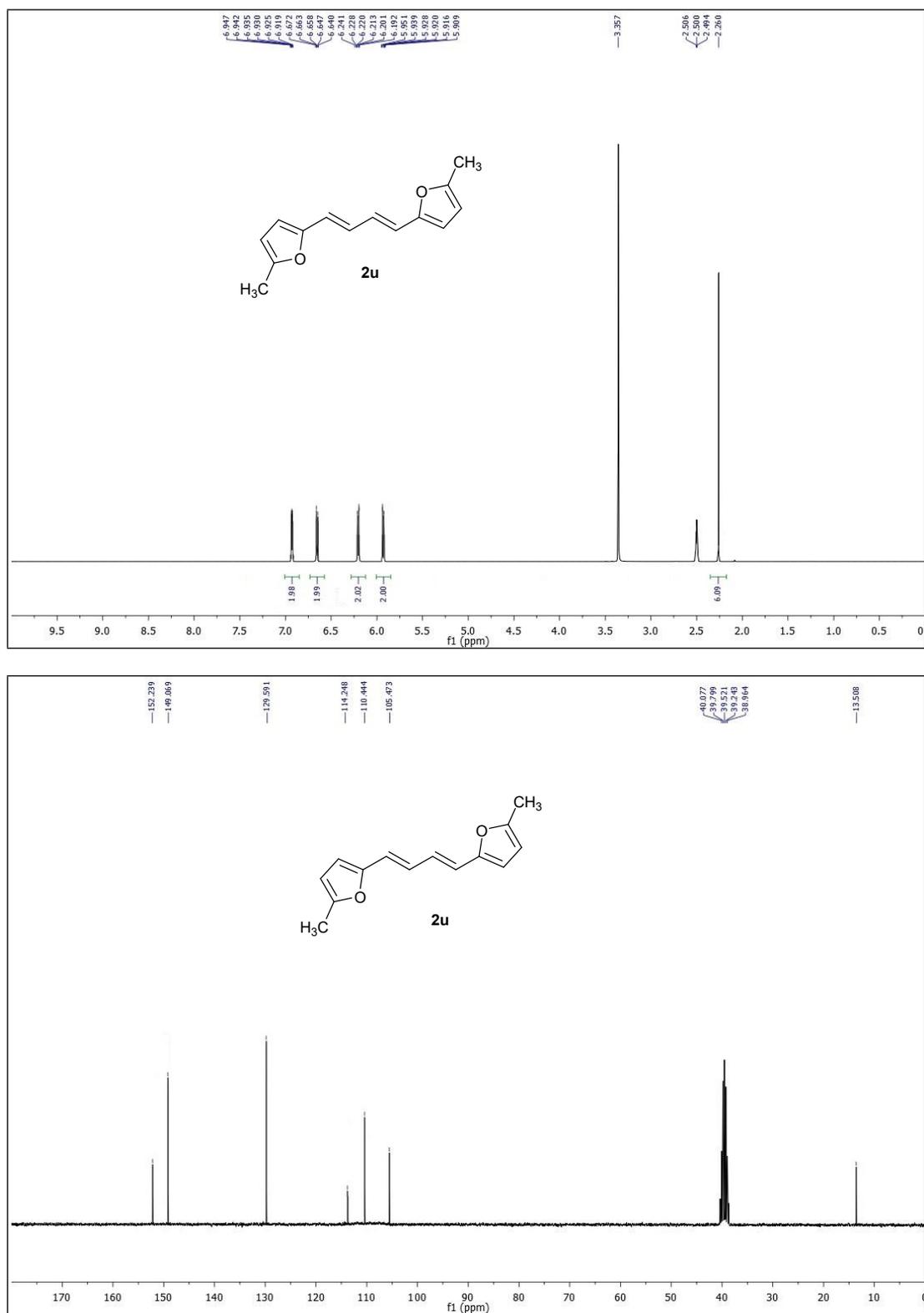


Figure S18. ¹H (top, 300 MHz) and ¹³C (bottom, 75 MHz) NMR spectra of **2u** in DMSO-d₆.

HRMS data of products

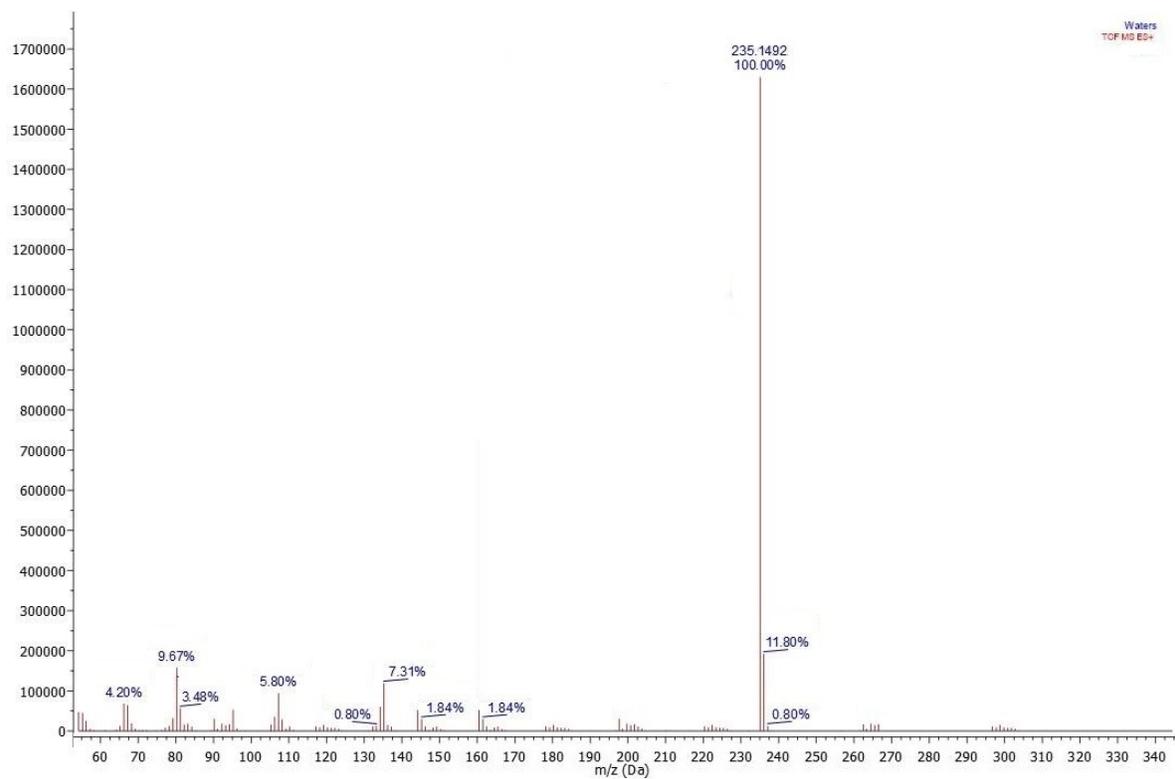


Figure S19. HRMS spectra of compound **2a**

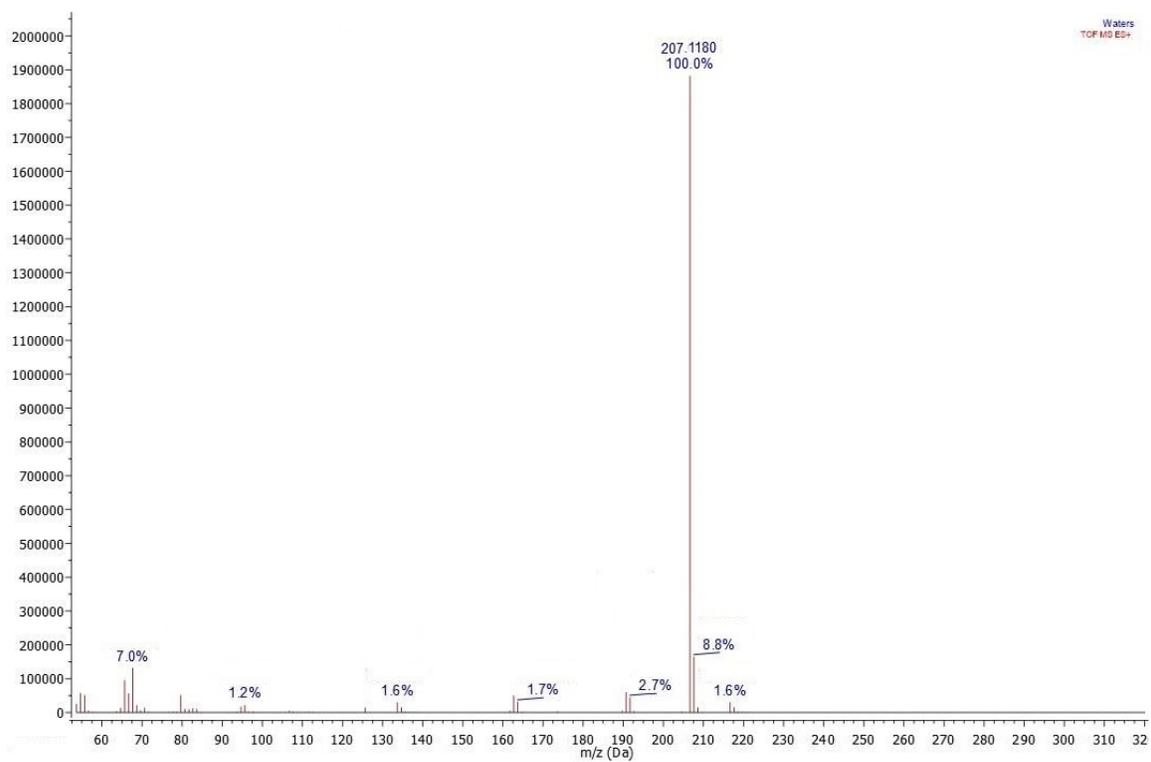


Figure S20. HRMS spectra of compound **2b**

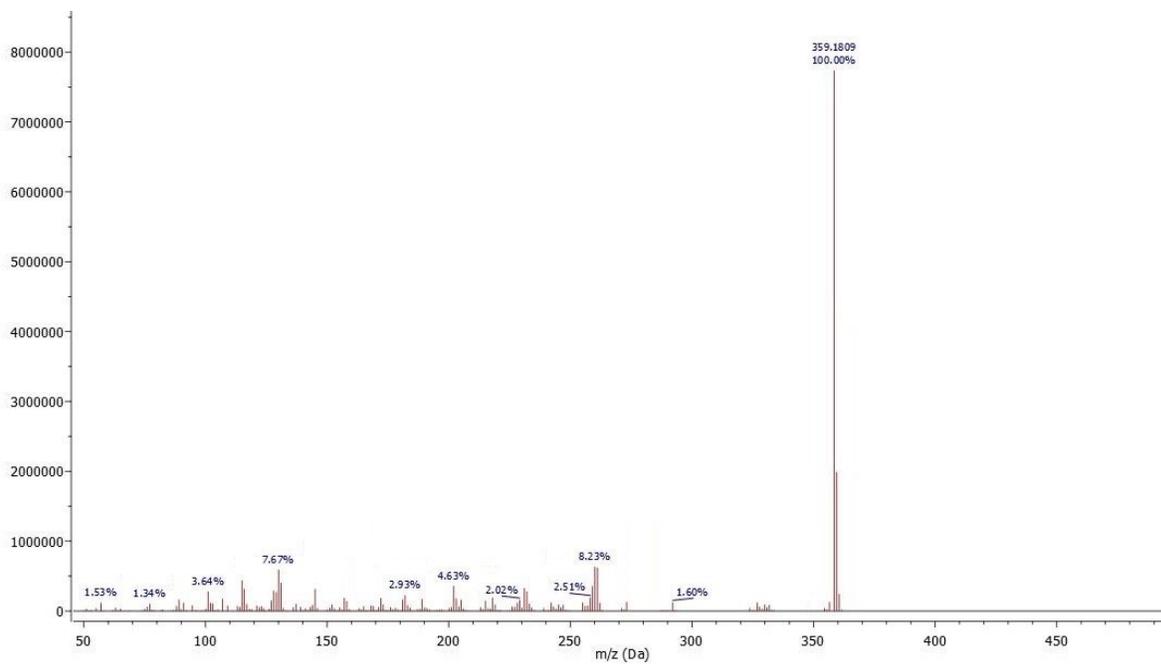


Figure S21. HRMS spectra of compound **2c**

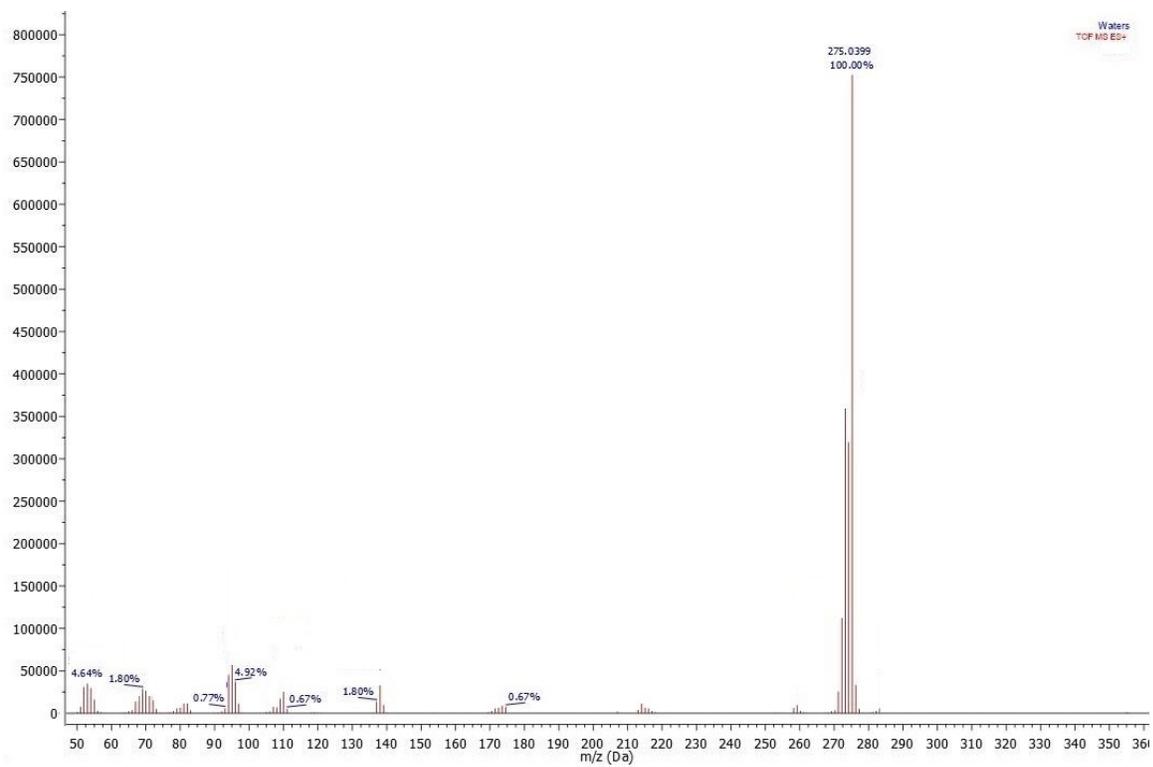


Figure S22. HRMS spectra of compound **2e**

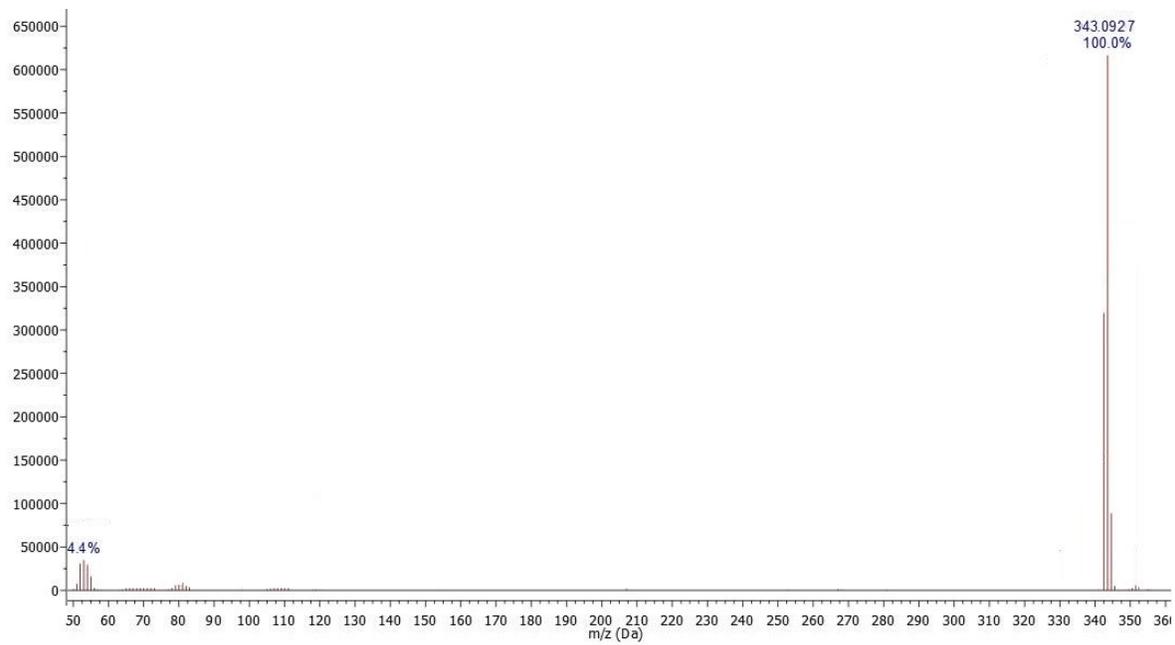


Figure S23. HRMS spectra of compound **2f**

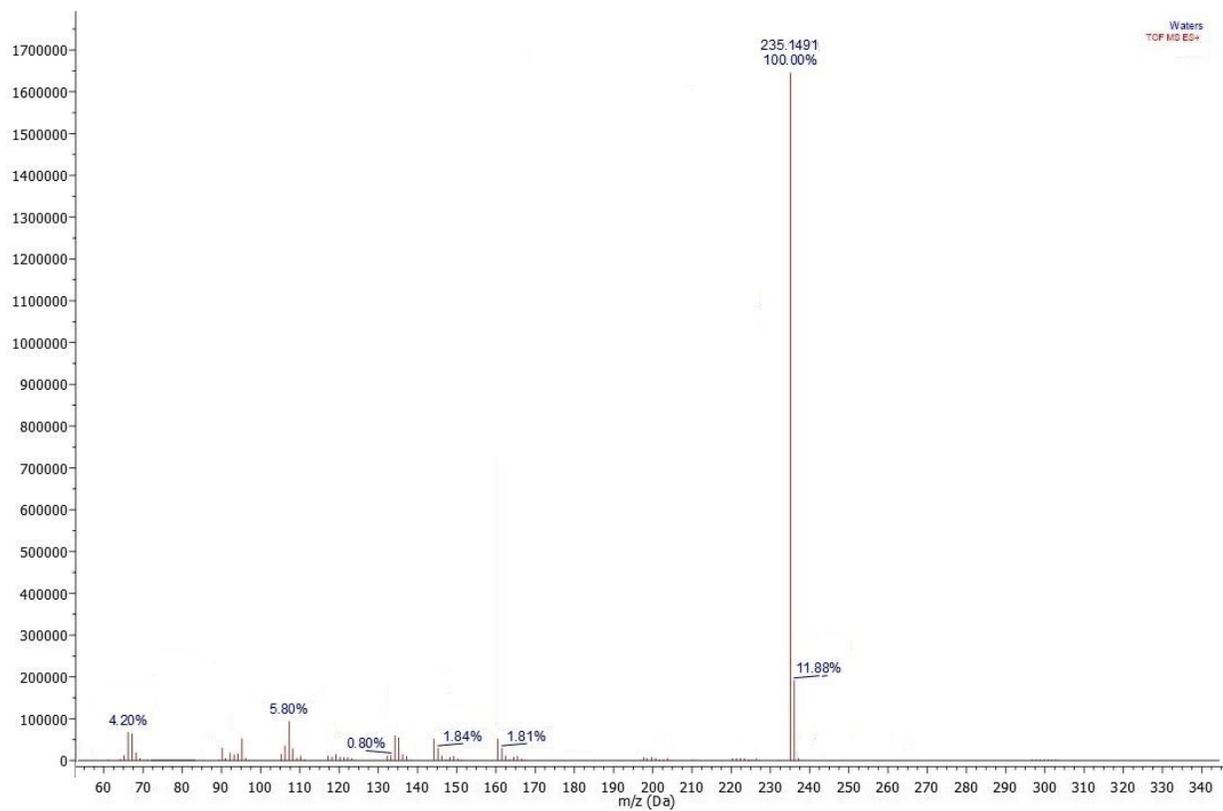


Figure S24. HRMS spectra of compound **2g**

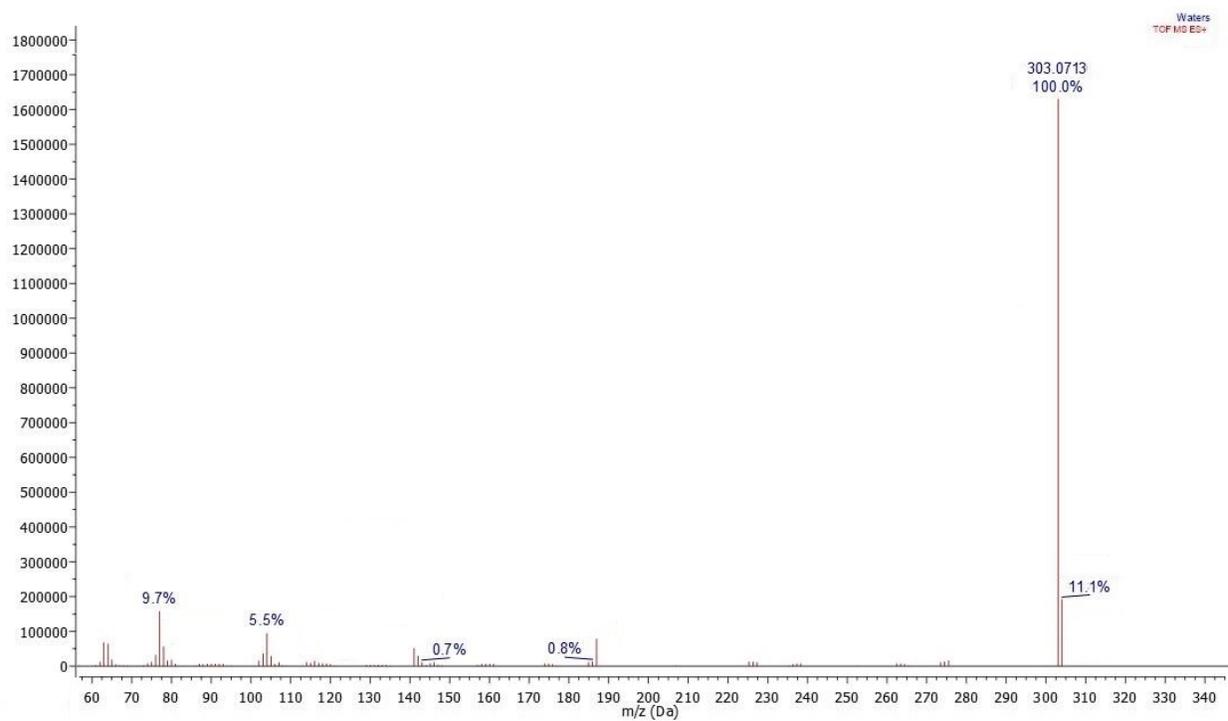


Figure S25. HRMS spectra of compound **2h**

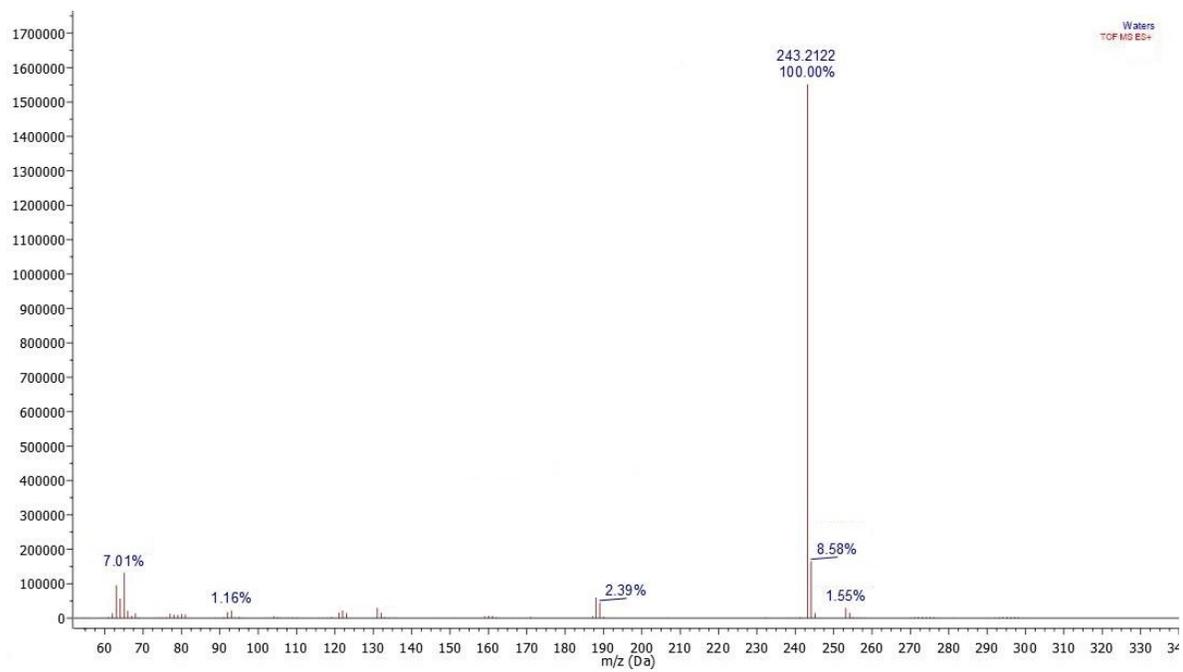


Figure S27. HRMS spectra of compound **2i**

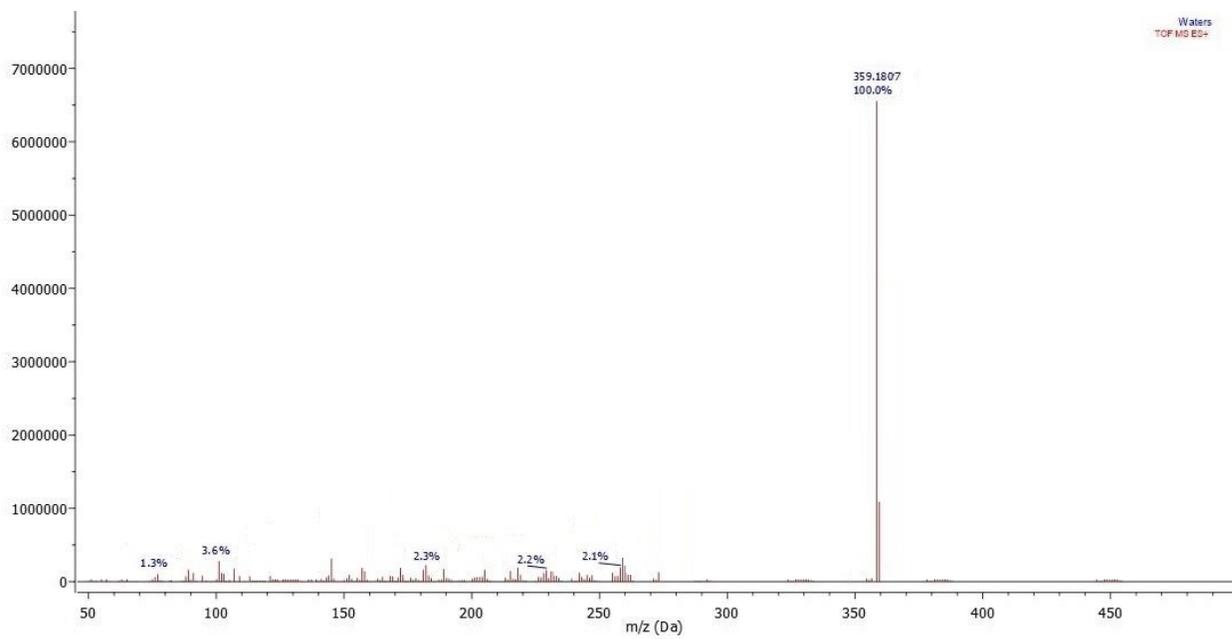


Figure S28. HRMS spectra of compound 2I

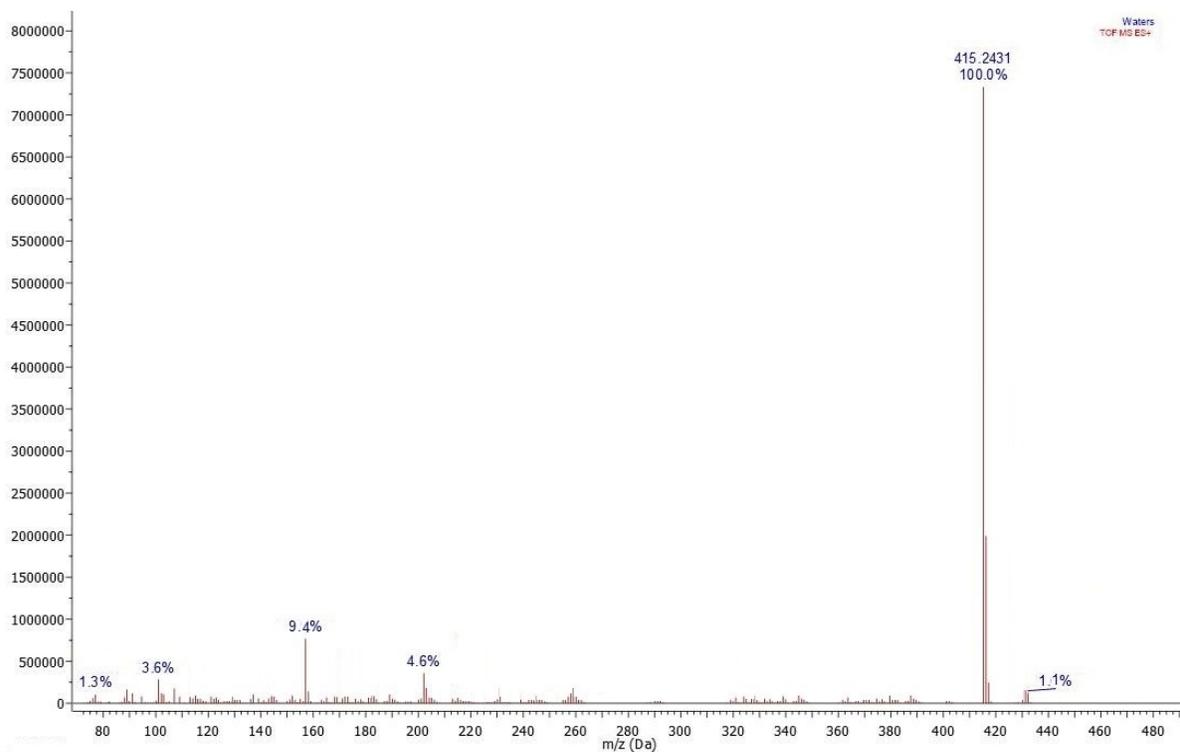


Figure S29. HRMS spectra of compound **2m**

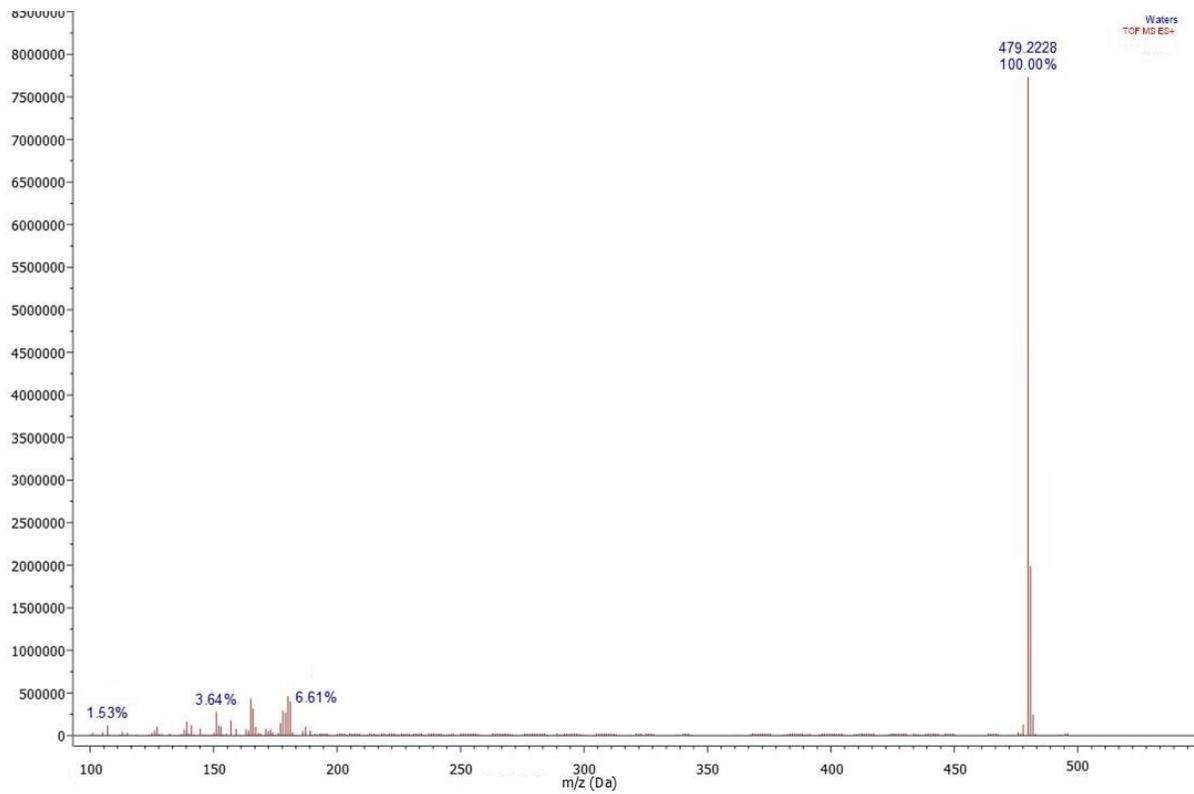


Figure S30. HRMS spectra of compound **2n**

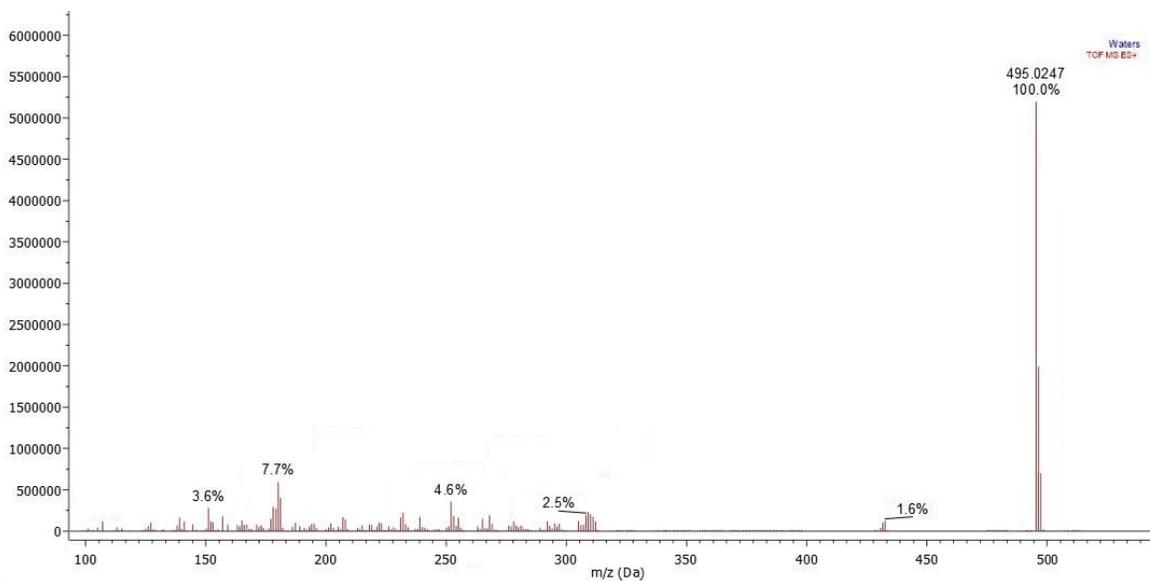


Figure S31. HRMS spectra of compound **2o**

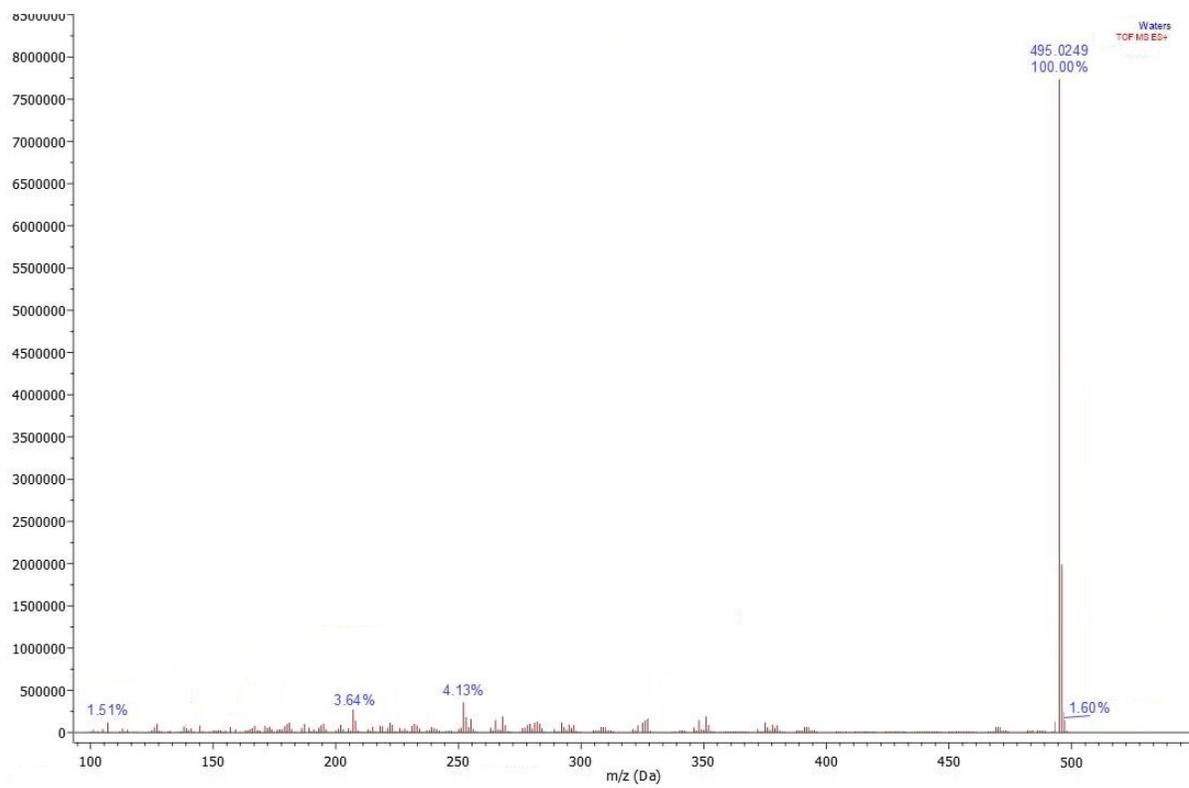


Figure S32. HRMS spectra of compound **2p**

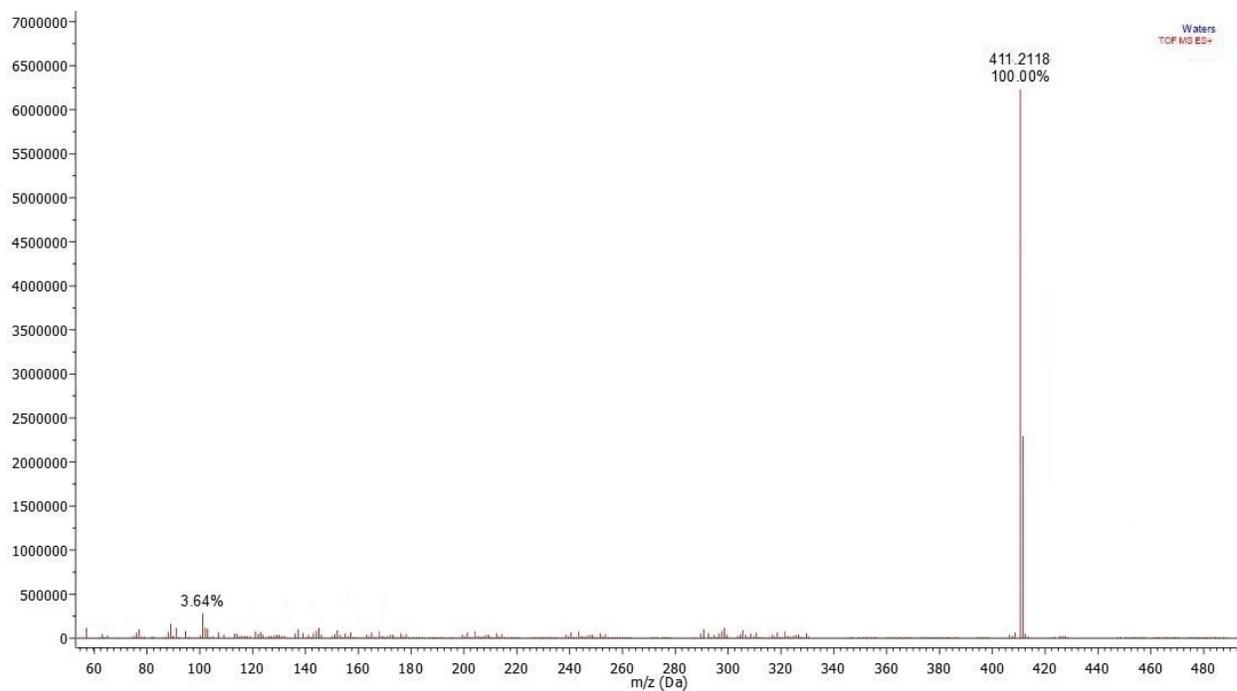


Figure S33. HRMS spectra of compound **2q**

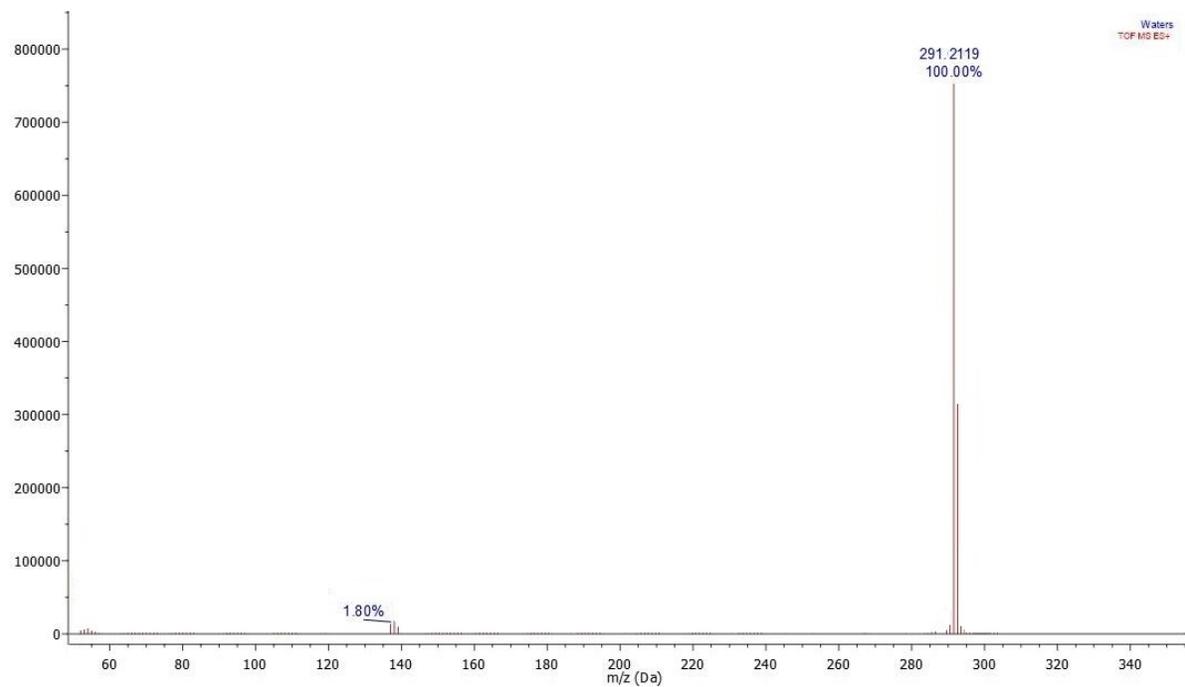


Figure S34. HRMS spectra of compound **2s**

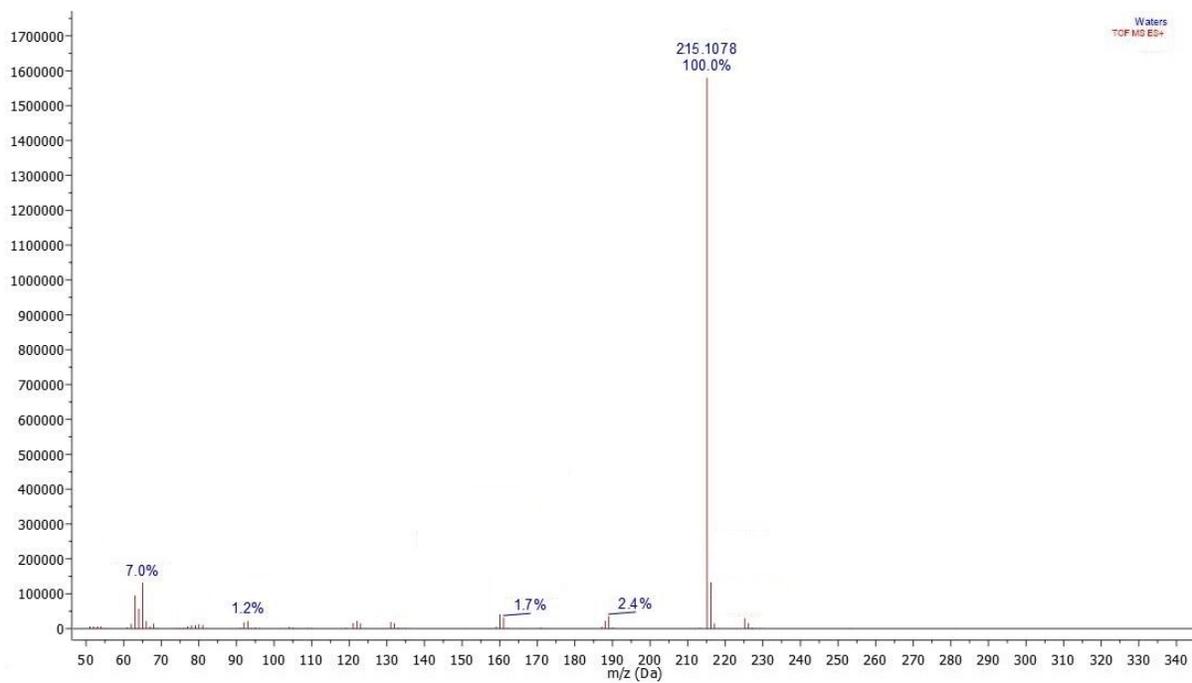


Figure S35. HRMS spectra of compound **2u**