Electronic Supplementary Material (ESI) for Chemical Science.

Electrochemical Radical Reactions of Alkyl Iodides: A Highly Efficient, Clean, Green Alternative to Tin Reagents

Supporting Information

Experimental Procedures, ^{1}H and ^{13}C NMR Spectra

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General Experimental Techniques

All reagents and solvents were used directly without further purification unless otherwise stated. Reaction progress was monitored by analytical thin layer chromatography (TLC) on silica gel coated aluminum oxide F254 plates (Merck KGaA). Developed TLC were visualized under UV light (254 nm). Flash column chromatography was performed using Biotage automatic column system (IS11579109). Reactions are terminated when the developed TLC plates showed complete consumption of reaction substrate. Mass spectra were measured on Thermo Finnigan MAT900 XE and Waters LCT Premier XE machines operating in ESI modes with the use of TOF analyzer. All ^{1}H and ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz respectively on Bruker Avance spectrometers at ambient temperature in $CDCl_3$ unless otherwise noted. NMR spectra were referenced to residual solvent peaks ($CDCl_3$: δ = 7.26 for ^{1}H NMR and δ = 77.0 for ^{13}C NMR) and chemical shifts were reported in ppm. In ^{1}H NMR, the multiplicity of the signal is indicated as *s* (singlet), *d* (doublet), *t* (triplet) and m (multiplet), defined as all multipeak signals where overlap or complex coupling of signals makes definitive descriptions of peaks difficult. Coupling constants are defined as *J* and quoted in Hz to one decimal place. IR spectra were obtained on a Bruker Alpha FTIR Spectrometer operating in ATR mode. Melting points were measured with a Gallenkamp apparatus and are uncorrected. Electrochemical reactions were carried out using an Ivium Technologies Vertex model potentiostat operating in chronoamperometry mode. CV plots were measured using the same machine with a glassy carbon working electrode, silver wire reference electrode and Pt wire counter electrode.

Electrochemical Setup

For all reactions we used a divided 'H' cell as our reaction vessel (dimensions shown in **Figure S1**) with each chamber having a size B19 ground-glass neck and a total volume of 30 mL. A semiporous sintered glass divider sits between each chamber. All reactions were carried out using 15 mL of electrolyte solution in each. Where graphite electrodes were used for the working-electrode and counter electrode, rods of 5 mm diameter were used at a depth of 25 mm giving an effective area of 412 mm^2. A silver wire, which was 1 mm thick, was used as a quasi-reference electrode and was likewise placed into solution to a depth of 25 mm giving an effective area of 79 mm^2 . One graphite and the silver wire were placed into the same chamber to minimise the potential drop deriving from resistance and kept 10 mm apart. Another graphite was used as the counter-electrode and placed in the other chamber of the H cell. Reactions were run using an Ivium Technologies Vertex model potentiostat operating in chronoamperometry mode. This mode provides real-time charge over time and current over time graphs for measuring the total charges passed over the course of reaction.

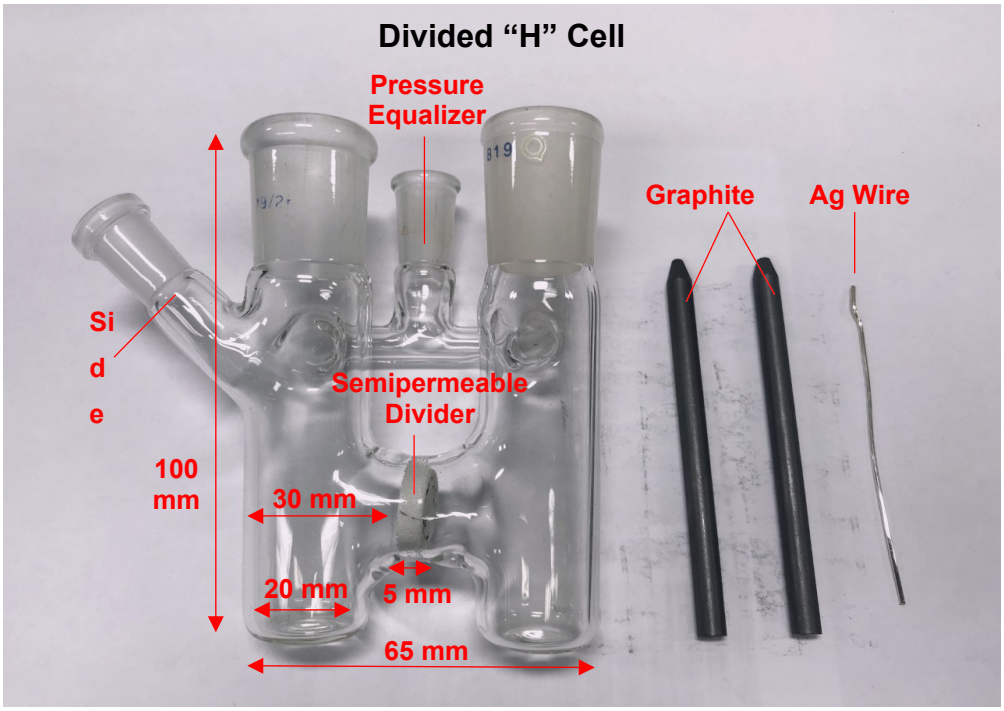
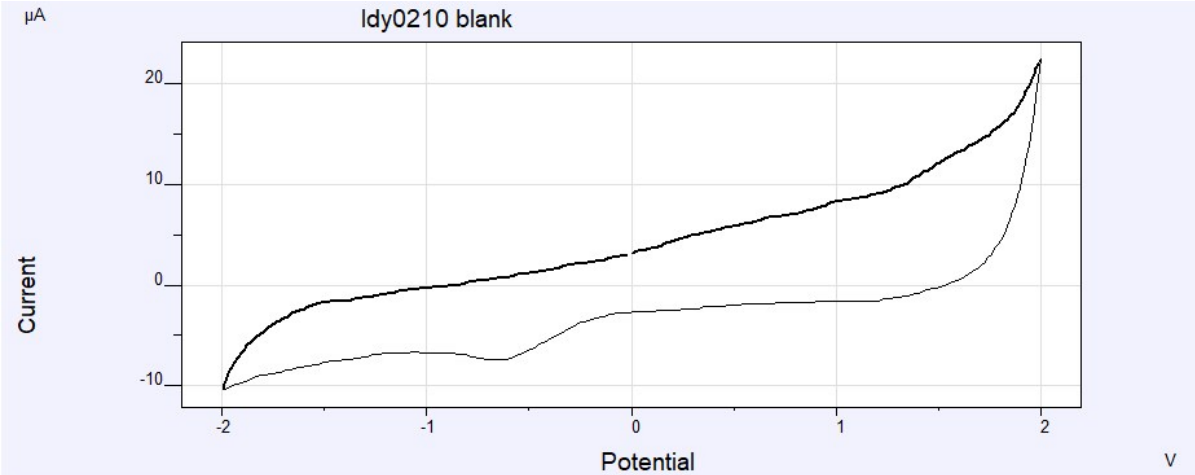
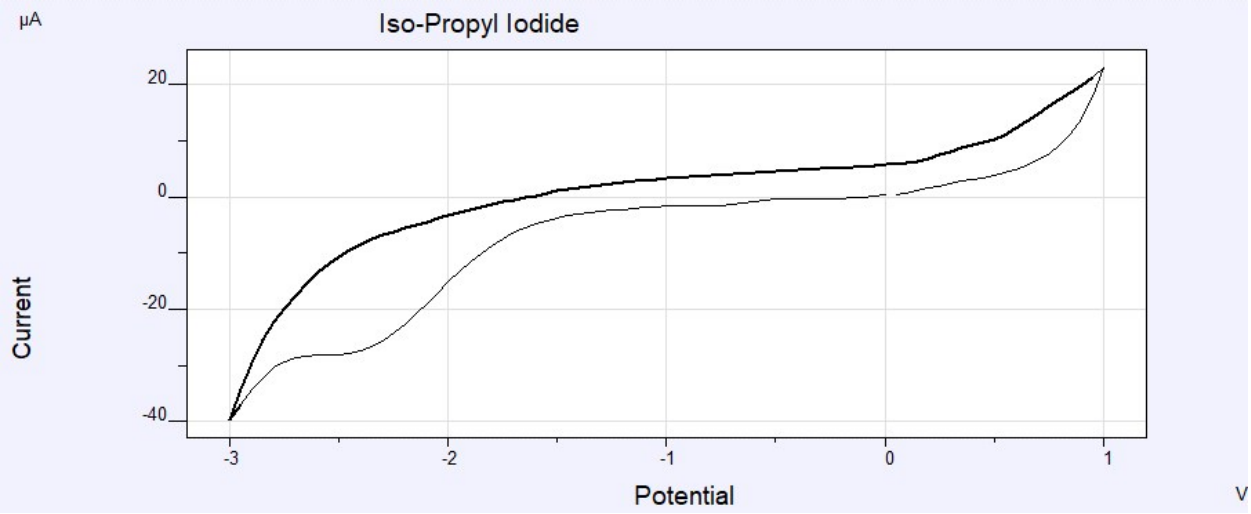


Figure S1. Image of divided H-cell and electrodes used with dimensions.

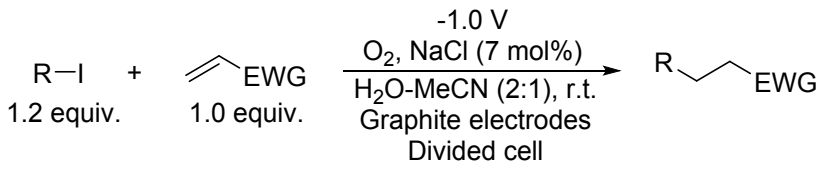
Cyclic Voltammetry of Molecular Oxygen



Cyclic Voltammetry of Isopropyl Iodide

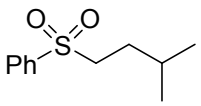


General Procedure for the Electrochemical Radical Alkene Addition Reactions



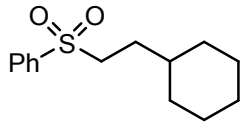
Two graphite electrodes (4.12 cm^2 area each) were placed into each chamber of the divided H cell and $HCl_{(aq)}$ (pH = 2, 10 mL), MeCN (5 mL) and NaCl (7 mol%) were added to each chamber. Alkene and alkyl halide were added to the cathodic chamber and the reaction mixture was stirred at room temperature under a constant reductive potential (-1.0 V) applied to the cathode for 20 to 45 h. The reaction mixture was then diluted with water (10 mL) and phases were separated. The aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layer was dried ($MgSO_4$), filtered, concentrated under reduced pressure and purified by column chromatography (cyclohexane : EtOAc 100 : 0 to 9 : 1) to yield the addition product.

3-Methylbutyl phenyl sulfone



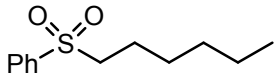
3-Methylbutyl phenyl sulfone (250 mg, 1.18 mmol, 99%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 2-iodopropane (0.142 mL, 1.43 mmol, 1.2 equiv.), was obtained as a colourless oil in 20 h: $\mathbf{R_f}$ 0.30 (petroleum ether : EtOAc 9 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 – 7.86 (m, 2H), 7.70 – 7.63 (m, 1H), 7.60 – 7.54 (m, 2H), 3.11 – 3.05 (m, 2H), 1.66 – 1.54 (m, 3H), 0.86 (d, *J* = 6.3 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3 (2C), 128.1 (2C), 54.8, 31.1, 27.3, 22.1 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{11}H_{17}O_2S$ 213.0949; found 213.0949. The analytical data were in excellent agreement with those reported in the literature.[1]

2-(Cyclohexyl)ethyl phenyl sulfone



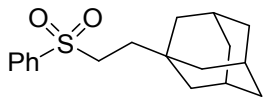
2-(Cyclohexyl)ethyl phenyl sulfone (300 mg, 1.19 mmol, 100%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and iodocyclohexane (0.185 mL, 1.43 mmol, 1.2 equiv.), was obtained as a colourless oil in 20 h: $\mathbf{R_f}$ 0.30 (petroleum ether : EtOAc 9 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.65 – 7.57 (m, 1H), 7.55 – 7.49 (m, 2H), 3.13 – 2.97 (m, 2H), 1.67 – 1.52 (m, 7H), 1.32 – 0.96 (m, 4H), 0.90 – 0.74 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3 (2C), 128.1 (2C), 54.4, 36.7, 32.8 (2C), 29.7, 26.3, 26.0 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{14}H_{21}O_2S$ 253.1257; found 253.1257. The analytical data were in excellent agreement with those reported in the literature.[2]

(Hexylsulfonyl)benzene



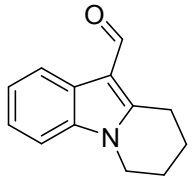
(Hexylsulfonyl)benzene (220 mg, 0.98 mmol, 82%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 1-iodobutane (0.163 mL, 1.43 mmol, 1.2 equiv.), was obtained as a colourless oil in 20 h: $\mathbf{R_f}$ 0.30 (petroleum ether : EtOAc 9 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 – 7.86 (m, 2H), 7.68 – 7.61 (m, 1H), 7.60 – 7.52 (m, 2H), 3.13 – 3.01 (m, 2H), 1.77 – 1.60 (m, 2H), 1.38 – 1.29 (m, 2H), 1.29 – 1.18 (m, 4H), 0.84 (t, J = 6.9 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3 (2C), 128.1 (2C), 56.4, 31.2, 28.0, 22.7, 22.3, 14.0. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{19}O_2S$ 227.1106; found 227.1111. The analytical data were in excellent agreement with those reported in the literature.[1]

(3r,5r,7r)-1-[2-(Phenylsulfonyl)ethyl]adamantane



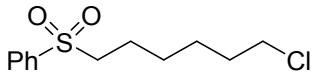
(3r,5r,7r)-1-[2-(Phenylsulfonyl)ethyl]adamantane (217 mg, 0.71 mmol, 60%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 1-iodoadamantane (0.44 mL, 1.43 mmol, 1.2 equiv.), was obtained as a white solid in 45 h: $\mathbf{R_f}$ 0.40 (petroleum ether : EtOAc 9 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 – 7.86 (m, 2H), 7.67 – 7.60 (m, 1H), 7.58 – 7.52 (m, 2H), 3.07 – 3.00 (m, 2H), 1.95 – 1.85 (m, 3H), 1.69 – 1.61 (m, 3H), 1.58 – 1.50 (m, 3H), 1.48 – 1.41 (m, 2H), 1.37 (d, *J* = 2.9 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.2, 133.5, 129.2 (2C), 127.9 (2C), 51.2, 41.8 (3C), 36.7 (3C), 35.8, 31.8, 28.3 (3C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{18}H_{25}O_2S$ 305.1570; found 305.1570. The analytical data were in excellent agreement with those reported in the literature.[3]

6,7,8,9-Tetrahydropyrido[1,2-a]indole-10-carbaldehyde



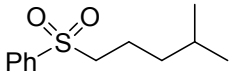
6,7,8,9-Tetrahydropyrido[1,2-a]indole-10-carbaldehyde (206 mg, 1.03 mmol, 89%), prepared from 1-(4-iodobutyl)-1H-indole-3-carbaldehyde (0.380 g, 1.16 mmol), was obtained as a white solid in 66 h: $\mathbf{R_f}$ 0.50 (petroleum ether : EtOAc 9 : 1). **m.p.** 121 – 123 °C (lit. 124 °C)[4]. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.20 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.37 – 7.18 (m, 3H), 4.10 (t, *J* = 6.2 Hz, 2H), 3.32 (t, *J* = 6.4 Hz, 2H), 2.22 – 2.09 (m, 2H), 2.05 – 1.91 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 183.6, 148.2, 136.5, 126.0, 123.2, 122.8, 120.6, 113.0, 109.2, 42.5, 22.8, 22.5, 19.7. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{13}H_{14}NO$ 200.1070; found 200.1070. The analytical data were in excellent agreement with those reported in the literature.[4]

((6-Chlorohexyl)sulfonyl)benzene



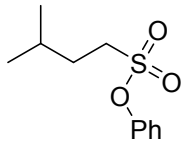
((6-Chlorohexyl)sulfonyl)benzene (295 mg, 1.13 mmol, 95%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 1-chloro-4-iodobutane (0.44 mL, 3.57 mmol, 3.0 equiv.), was obtained as a yellow oil in 32 h: $\mathbf{R_f}$ 0.30 (petroleum ether : EtOAc 4 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 – 7.88 (m, 2H), 7.69 – 7.63 (m, 1H), 7.61 – 7.54 (m, 2H), 3.49 (t, *J* = 6.6 Hz, 2H), 3.14 – 3.03 (m, 2H), 1.81 – 1.65 (m, 4H), 1.49 – 1.34 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.1, 133.7, 129.3 (2C), 128.0 (2C), 56.1, 44.7, 32.1, 27.5, 26.2, 22.5. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{18}ClO_2S$ 261.0711; found 261.0712. The analytical data were in excellent agreement with those reported in the literature.[5]

4-Methylpentyl phenyl sulfone



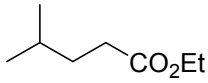
4-Methylpentyl phenyl sulfone (263 mg, 1.17 mmol, 98%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 1-iodo-2-methylpropane (0.16 mL, 3.57 mmol, 3.0 equiv.), was obtained as a colourless oil in 40 h: $\mathbf{R_f}$ 0.30 (petroleum ether : EtOAc 9 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 – 7.88 (m, 2H), 7.68 – 7.62 (m, 1H), 7.60 – 7.53 (m, 2H), 3.11 – 2.98 (m, 2H), 1.78 – 1.65 (m, 2H), 1.49 (dh, *J* = 13.3, 6.7 Hz, 1H), 1.30 – 1.15 (m, 2H), 0.84 (d, *J* = 6.6 Hz, 5H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.3, 133.7, 129.3 (2C), 128.1 (2C), 56.6, 37.4, 27.7, 22.3 (2C), 20.6. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{19}O_2S$ 227.1100; found 227.1100. The analytical data were in excellent agreement with those reported in the literature.[6]

Phenyl 3-Methylbutane-1-sulfonate



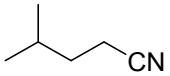
Phenyl 3-Methylbutane-1-sulfonate (245 mg, 1.08 mmol, 99%), prepared from phenyl vinyl sulfone (200 mg, 1.19 mmol, 1.0 equiv.) and 2-iodopropane (0.13 mL, 1.30 mmol, 1.2 equiv.), was obtained as a yellow oil in 40 h: $\mathbf{R_f}$ 0.40 (petroleum ether : EtOAc 1 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45 – 7.37 (m, 2H), 7.35 – 7.30 (m, 1H), 7.30 – 7.25 (m, 2H), 3.28 – 3.20 (m, 2H), 1.92 – 1.83 (m, 2H), 1.75 (dp, *J* = 13.4, 6.6 Hz, 1H), 0.97 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.2, 130.0 (2C), 127.26, 122.1 (2C), 48.9, 32.0, 27.3, 22.1 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{11}H_{17}O_3S$ 229.0893; found 229.0892.

Ethyl 4-methylpentanoate



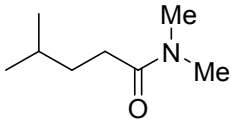
Ethyl 4-methylpentanoate (256 mg, 1.78 mmol, 89%), prepared from ethyl acrylate (0.22 mL, 1.99 mmol, 1.0 equiv.) and 2-iodopropane (0.239 mL, 2.39 mmol, 1.2 equiv.), was obtained as a red oil in 20 h without further purification: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.12 (q, *J* = 7.1 Hz, 2H), 2.34 – 2.26 (m, 2H), 1.66 – 1.45 (m, 3H), 1.25 (t, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.3 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.1, 60.2, 33.8, 32.5, 27.7, 22.2 (2C), 14.2. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_8O_2H_{17}$ 145.1223; found 145.1223.

4-Methylpentanenitrile



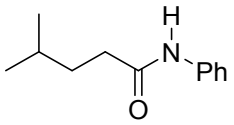
4-Methylpentanenitrile (359 mg, 3.69 mmol, 98%), prepared from acrylonitrile (0.25 mL, 3.77 mmol, 1.0 equiv.) and 2-iodopropane (0.45 mL, 4.52 mmol, 1.2 equiv.), was obtained as a yellow oil in 30 h without further purification: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.33 (t, *J* = 7.4 Hz, 2H), 1.73 (dp, *J* = 13.3, 6.7 Hz, 1H), 1.55 (q, *J* = 7.4 Hz, 2H), 0.93 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 120.0, 34.0, 27.3, 21.9 (2C), 15.2. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_6H_{12}N$ 227.1100; found 227.1100.

***N*,*N*,4-Trimethylpentanamide**



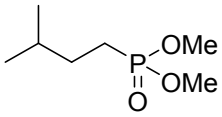
N,*N*,4-Trimethylpentanamide (277 mg, 1.94 mmol, 96%), prepared from *N*, *N*-dimethylacrylamide (0.21 mL, 2.02 mmol, 1.0 equiv.) and 2-iodopropane (0.24 mL, 2.42 mmol, 1.2 equiv.), was obtained as an orange oil in 32 h: **R_f** 0.3 (petroleum ether : EtOAc 1 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.00 (s, 3H), 2.93 (s, 3H), 2.35 – 2.26 (m, 2H), 1.65 – 1.47 (m, 3H), 0.90 (d, *J* = 6.4 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.42, 37.29, 35.36, 34.02, 31.41, 27.89, 22.36 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_8H_{18}NO$ 144.1383; found 144.1384.

4-Methyl-*N*-phenylpentanamide



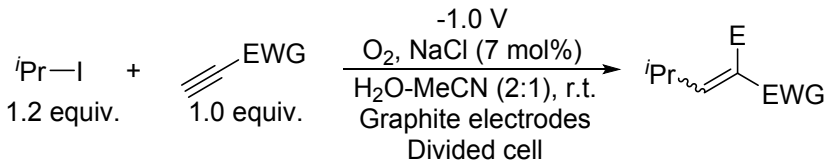
4-Methyl-*N*-phenylpentanamide (255 mg, 1.33 mmol, 98%), prepared from *N*-methyl-*N*-phenylacrylamide (200 mg, 1.36 mmol, 1.0 equiv.) and 2-iodopropane (0.16 mL, 1.63 mmol, 1.2 equiv.), was obtained as a white foam in 32 h: $\mathbf{R_f}$ 0.4 (petroleum ether : EtOAc 4 : 1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.31 (dd, *J* = 8.5, 7.4 Hz, 2H), 7.10 (t, *J* = 7.5 Hz, 1H), 2.44 – 2.31 (m, 2H), 1.63 (dd, *J* = 7.0, 4.6 Hz, 3H), 0.94 (d, *J* = 6.0 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.5, 137.9, 129.0 (2C), 124.2, 119.7 (2C), 35.9, 34.4, 27.8, 22.3 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{18}NO$ 192.1383; found 192.1383. The analytical data were in excellent agreement with those reported in the literature.[7]

Dimethyl isopentylphosphonate



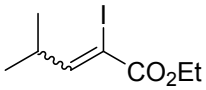
Dimethyl isopentylphosphonate (252 mg, 1.40 mmol, 95%), prepared from dimethyl vinylphosphonate (200 mg, 1.47 mmol, 1.0 equiv.) and 2-iodopropane (0.22 mL, 2.21 mmol, 1.5 equiv.), was obtained as a yellow oil in 32 h: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.74 (s, 3H), 3.71 (s, 3H), 1.78 – 1.66 (m, 2H), 1.64 – 1.52 (m, 1H), 1.52 – 1.42 (m, 2H), 0.89 (d, *J* = 6.5 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 52.3, 30.9, 28.8, 28.6, 23.2, 21.9 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_7H_{18}O_3P$ 181.0988; found 181.0988.

General Procedure for the Electrochemical Radical Alkyne Addition Reactions



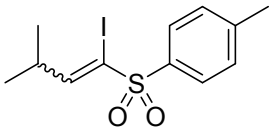
Two graphite electrodes (4.12 cm^2 area each) were placed into each chamber of the divided H cell and $HCl_{(aq)}$ (pH = 2, 10 mL), MeCN (5 mL) and NaCl (7 mol%) were added to each chamber. Alkyne and alkyl halide were added to the cathodic chamber and the reaction mixture was stirred at room temperature under a constant reductive potential (-1.0 V) applied to the cathode for 20 h. The reaction mixture was then diluted with water (10 mL) and phases were separated. The aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layer was dried ($MgSO_4$), filtered, concentrated under reduced pressure and purified by column chromatography (cyclohexane : EtOAc 100 : 0 to 9 : 1) to yield the addition product.

Ethyl 2-iodo-4-methylpent-2-enoate



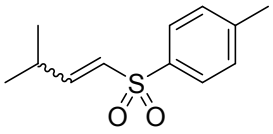
Ethyl 2-iodo-4-methylpent-2-enoate (140 mg, 0.552 mmol, 25%), prepared from ethyl propiolate (0.21 mL, 2.07 mmol, 1.0 equiv.) and 2-iodopropane (0.25 mL, 2.49 mmol, 1.2 equiv.), was obtained as a colourless oil in 20 h: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 9.1 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 2.71 (dp, *J* = 9.1, 6.7 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.08 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 163.1, 158.6, 92.5, 62.6, 36.5, 20.7 (2C), 14.2. **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_8H_{14}IO_2$ 269.0033; found 269.0033.

1-((1-Iodo-3-methylbut-1-en-1-yl)sulfonyl)-4-methylbenzene



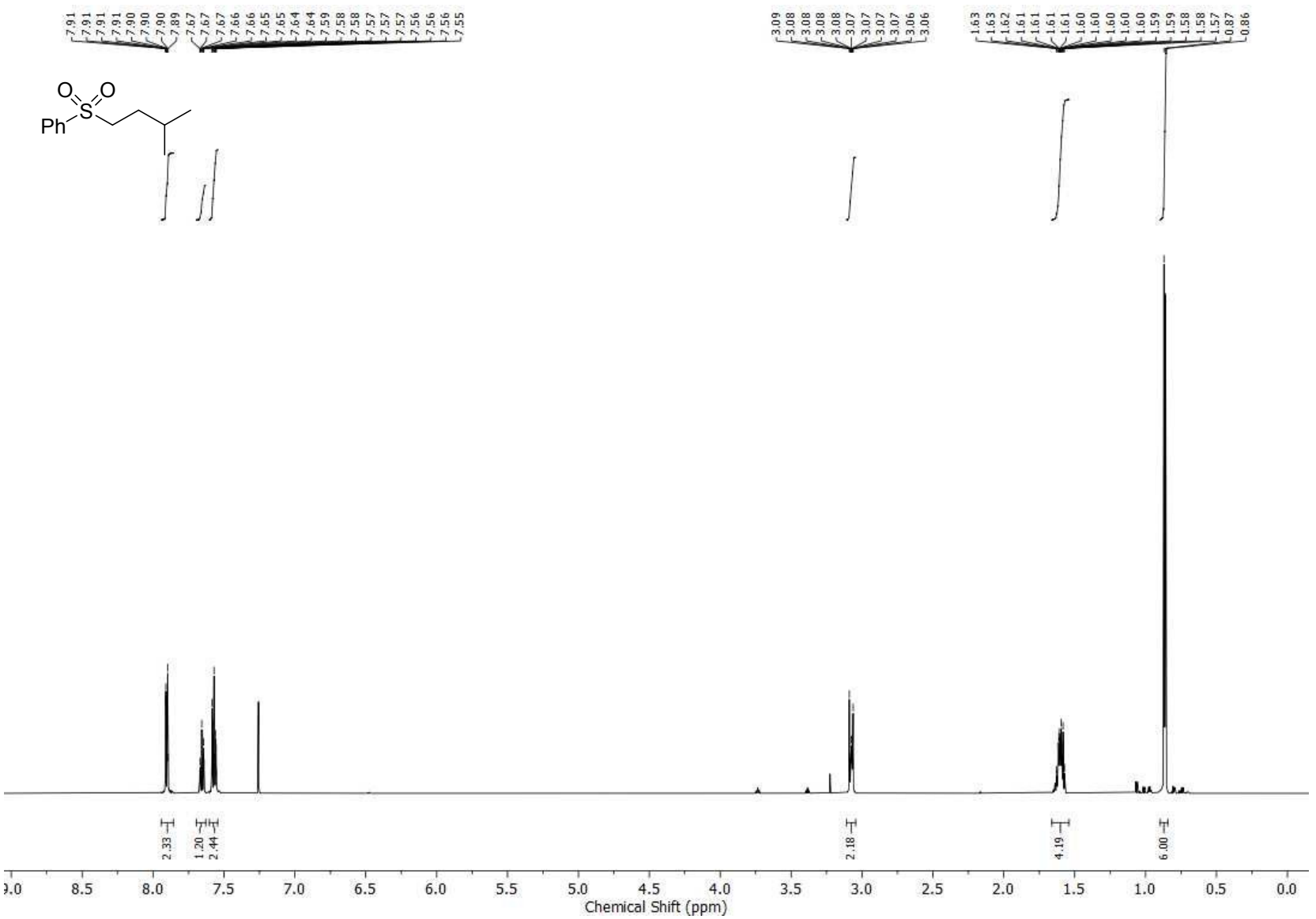
1-((1-Iodo-3-methylbut-1-en-1-yl)sulfonyl)-4-methylbenzene (54 mg, 0.155 mmol, 14%, cis : trans 1 : 4), prepared from 1-(ethynylsulfonyl)-4-methylbenzene (0.200 g, 1.11 mmol, 1.0 equiv.) and 2-iodopropane (0.11 mL, 1.33 mmol, 1.2 equiv.), was obtained as a white foam in 24 h: Major isomer: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.10 (d, *J* = 9.1 Hz, 1H), 2.56 (hept, *J* = 9.2, 6.7 Hz, 1H), 2.44 (s, 3H), 1.09 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 156.5, 144.9, 134.5, 129.8 (2C), 129.3 (2C), 99.9, 35.8, 21.8, 20.7 (2C). Minor isomer: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.33 (d, *J* = 7.9 Hz, 2H), 3.03 (hept, *J* = 7.4 Hz, 1H), 2.44 (s, 3H), 1.14 (d, *J* = 7.2 Hz, 6H). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{16}IO_2S_1$ 350.9910; found 350.9910.

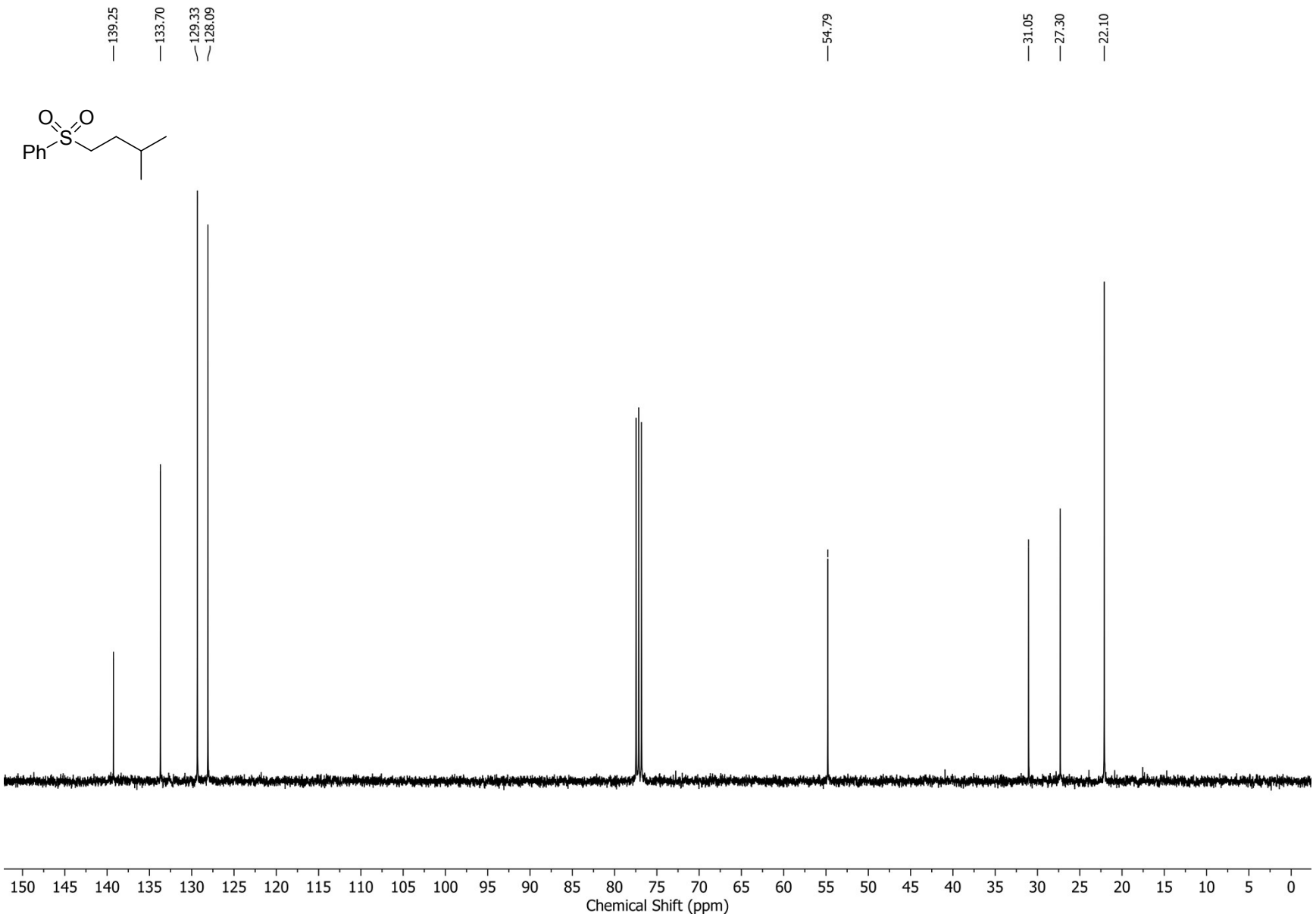
1-Methyl-4-((3-methylbut-1-en-1-yl)sulfonyl)benzene



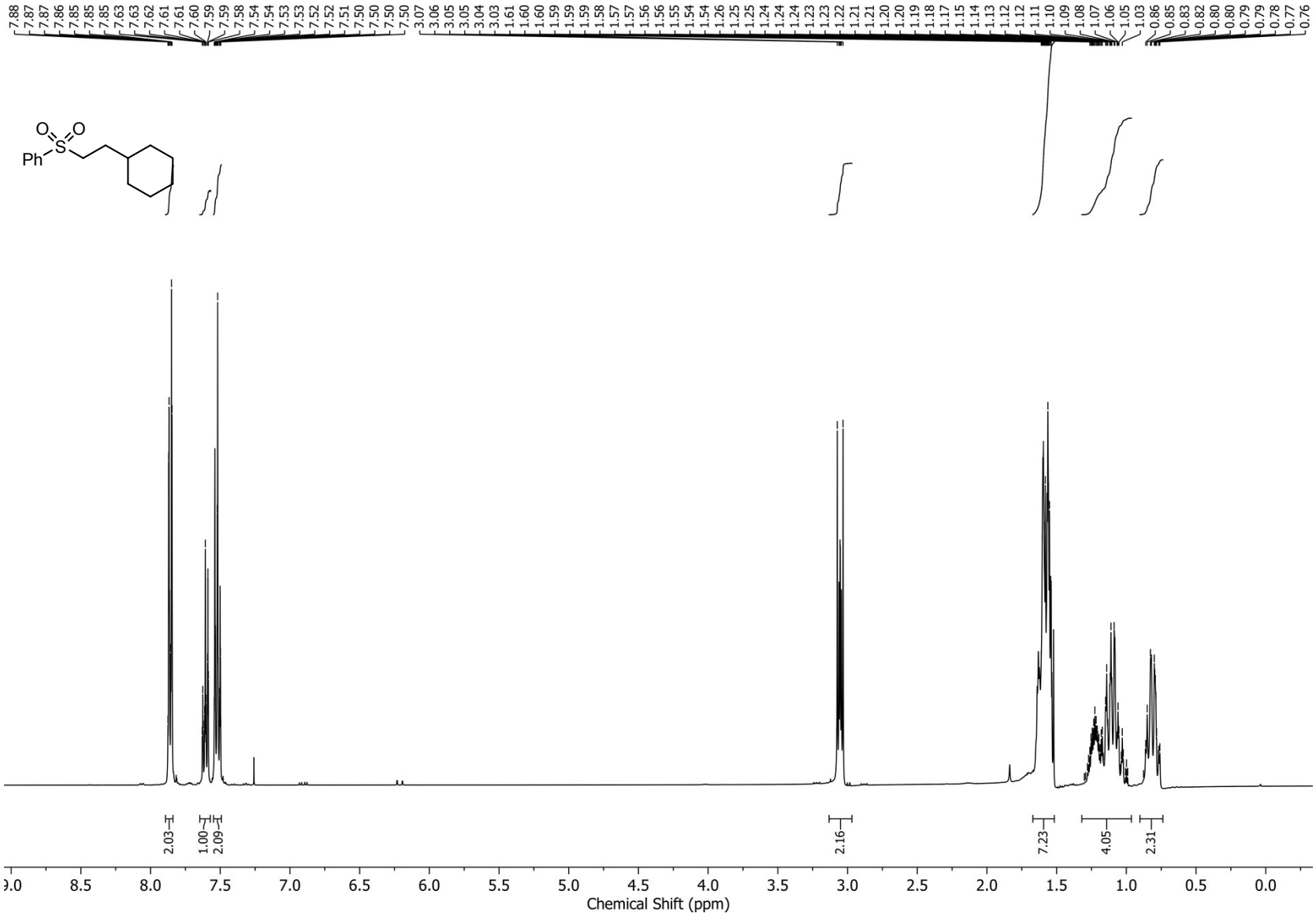
1-Methyl-4-((3-methylbut-1-en-1-yl)sulfonyl)benzen (167 mg, 0.744 mmol, 67%, cis : trans 1 : 5), prepared from 1-(ethynylsulfonyl)-4-methylbenzene (0.200 g, 1.11 mmol, 1.0 equiv.) and 2-iodopropane (0.11 mL, 1.33 mmol, 1.2 equiv.), was obtained as a colourless oil in 24 h: *trans*-Isomer **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 6.94 (dd, *J* = 15.1, 6.3 Hz, 1H), 6.23 (dd, *J* = 15.2, 1.5 Hz, 1H), 2.57 – 2.45 (m, 1H), 2.43 (s, 3H), 1.05 (d, *J* = 6.8 Hz, 6H). *cis*-Isomer **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 6.16 (dd, *J* = 11.0, 0.8 Hz, 1H), 5.99 (d, *J* = 10.9 Hz,1H), 2.57 – 2.45 (m, 1H), 2.43 (s, 3H), 0.98 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 152.4, 144.2, 137.9, 137.5, 129.9 (2C), 127.7 (2C), 30.6, 21.7, 20.9 (2C). **HRMS (ESI)** *m/z* $[M + H]^+$ calcd for $C_{12}H_{17}SO_2$ 225.0944; found 225.0943.

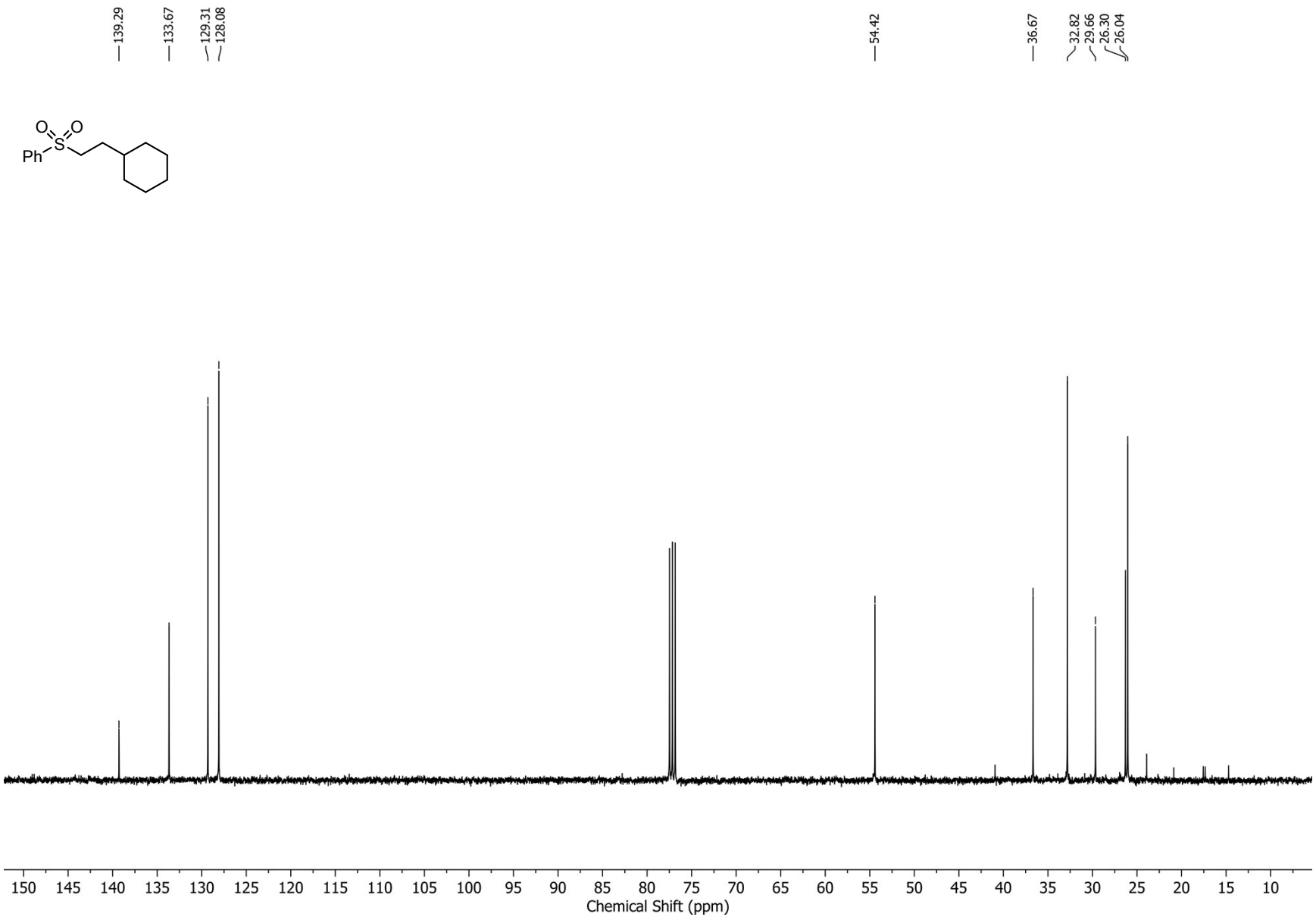
3-Methylbutyl phenyl sulfone ^{1}H and ^{13}C NMR



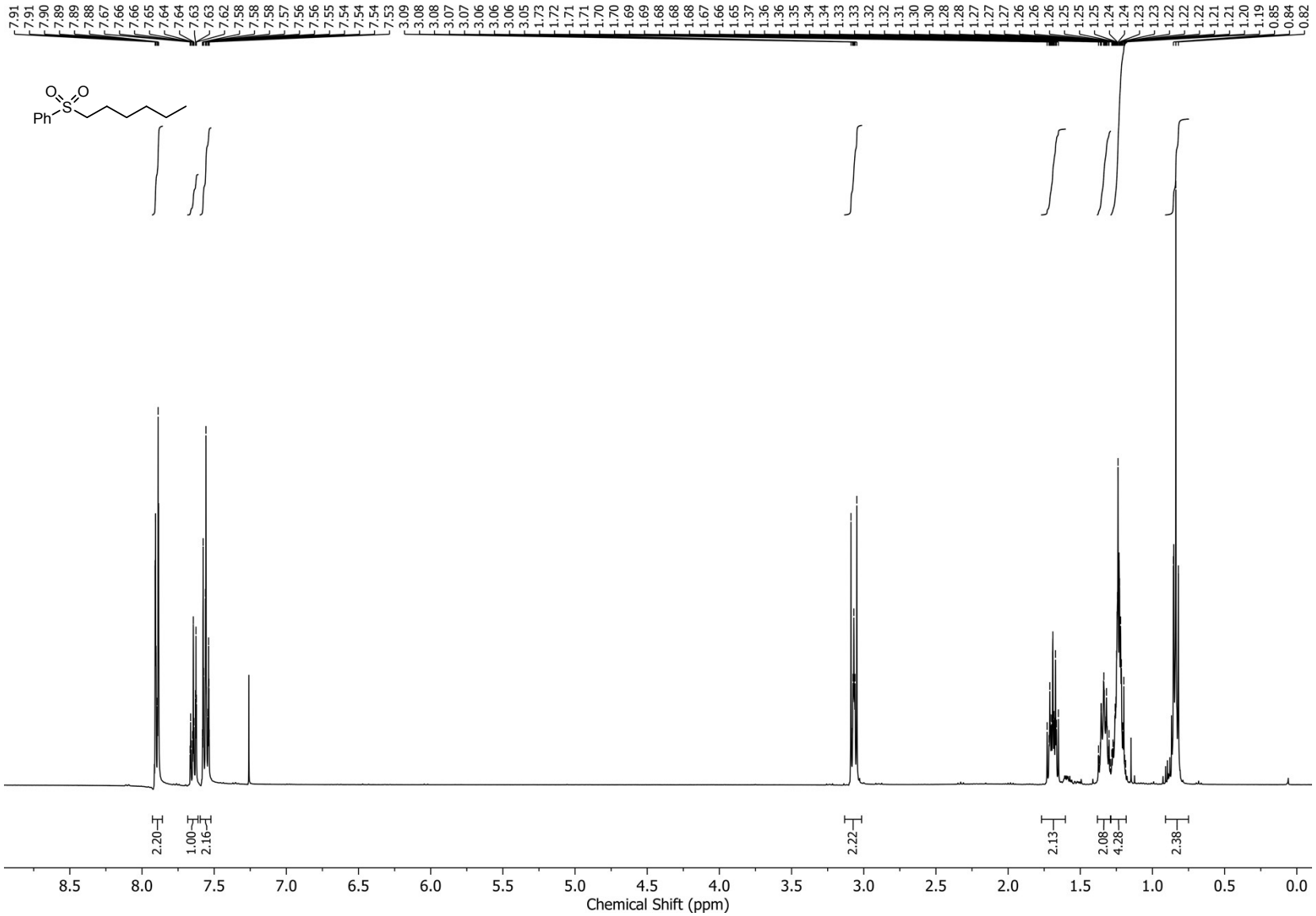

139.25
133.70
129.33
128.09
54.79
31.05
27.30
22.10
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
Chemical Shift (ppm)

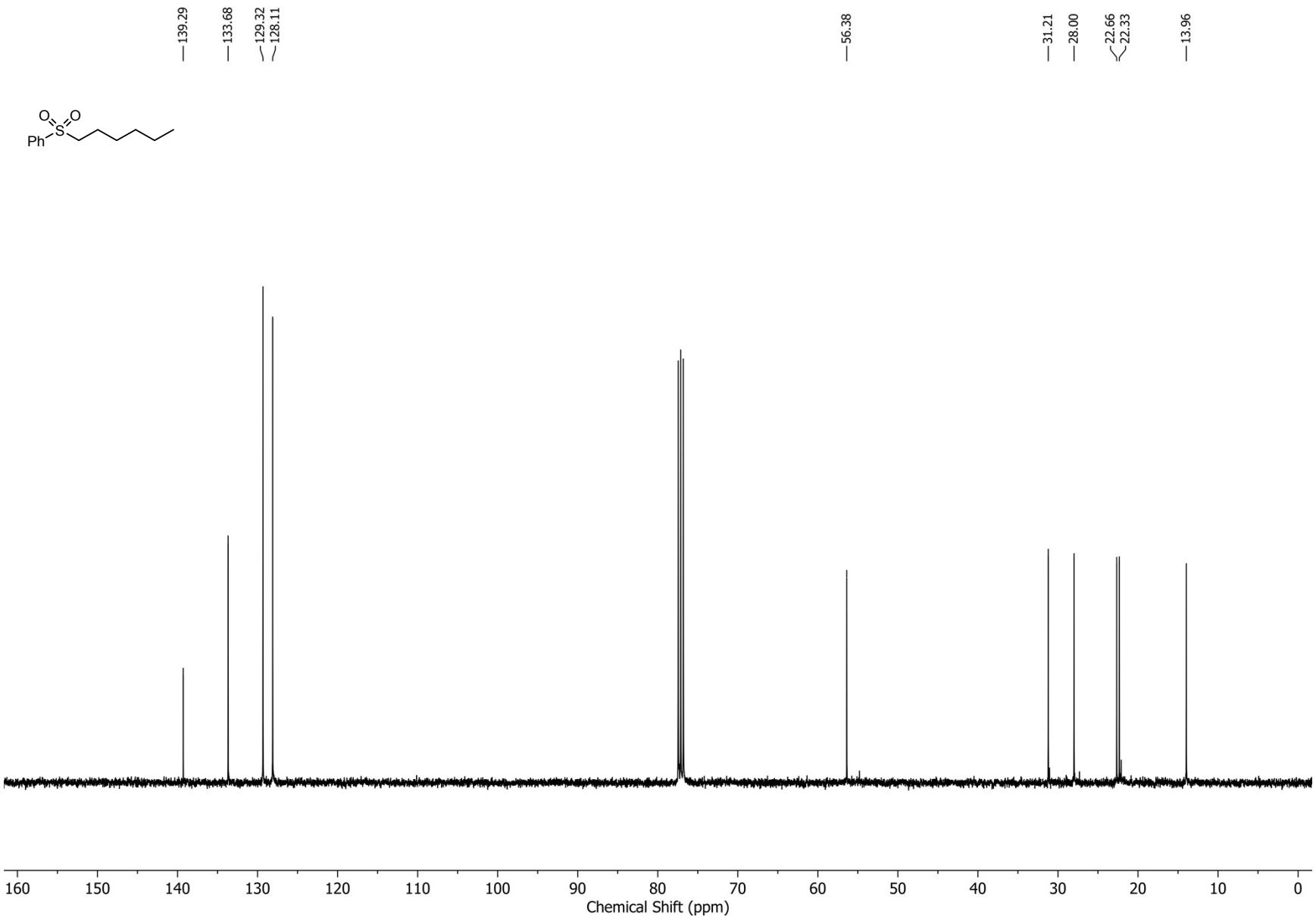
2-(Cyclohexyl)ethyl phenyl sulfone ^{1}H and ^{13}C NMR



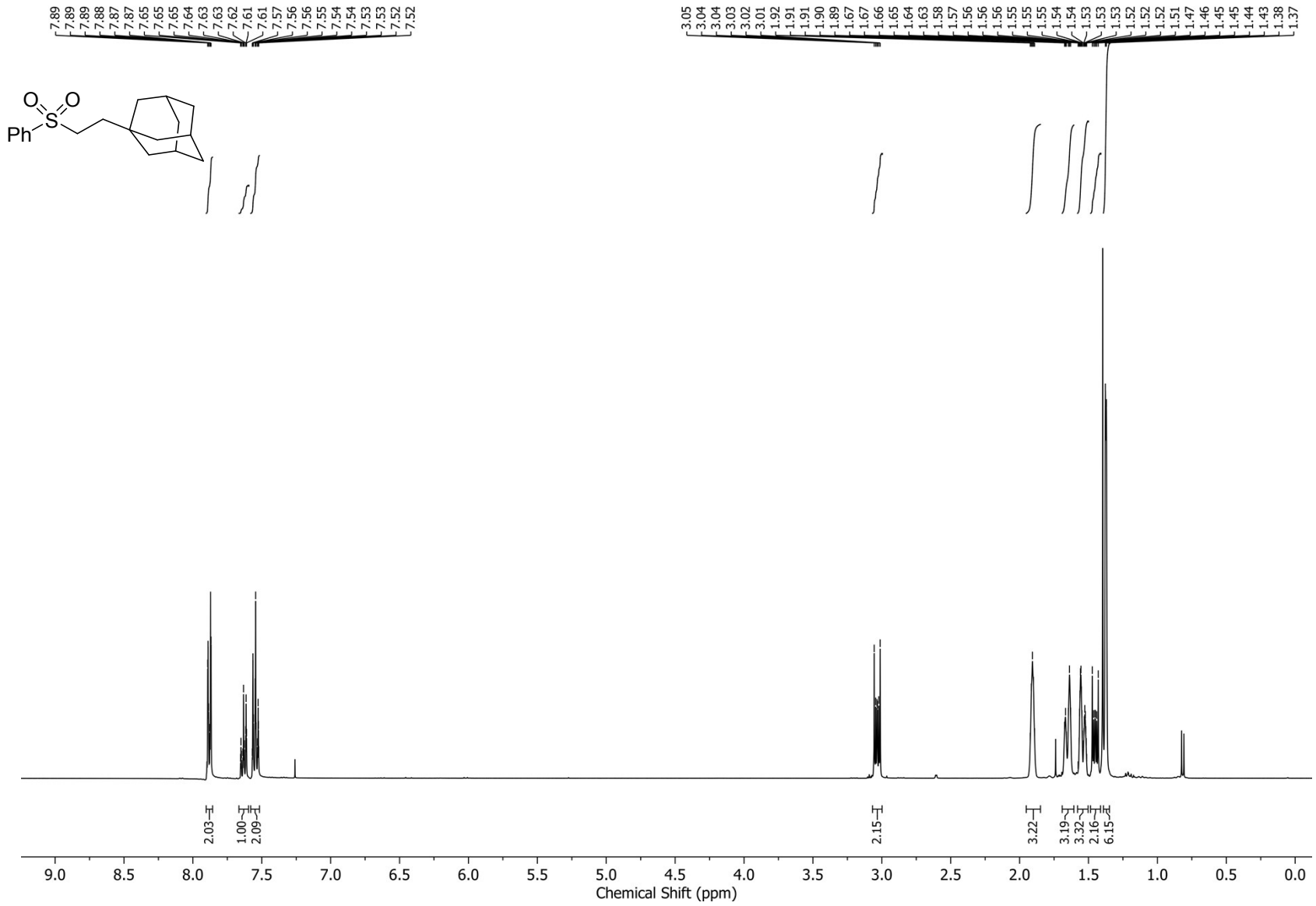

139.29
133.67
129.31
128.08
54.42
36.67
32.82
29.66
26.30
26.04
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
Chemical Shift (ppm)

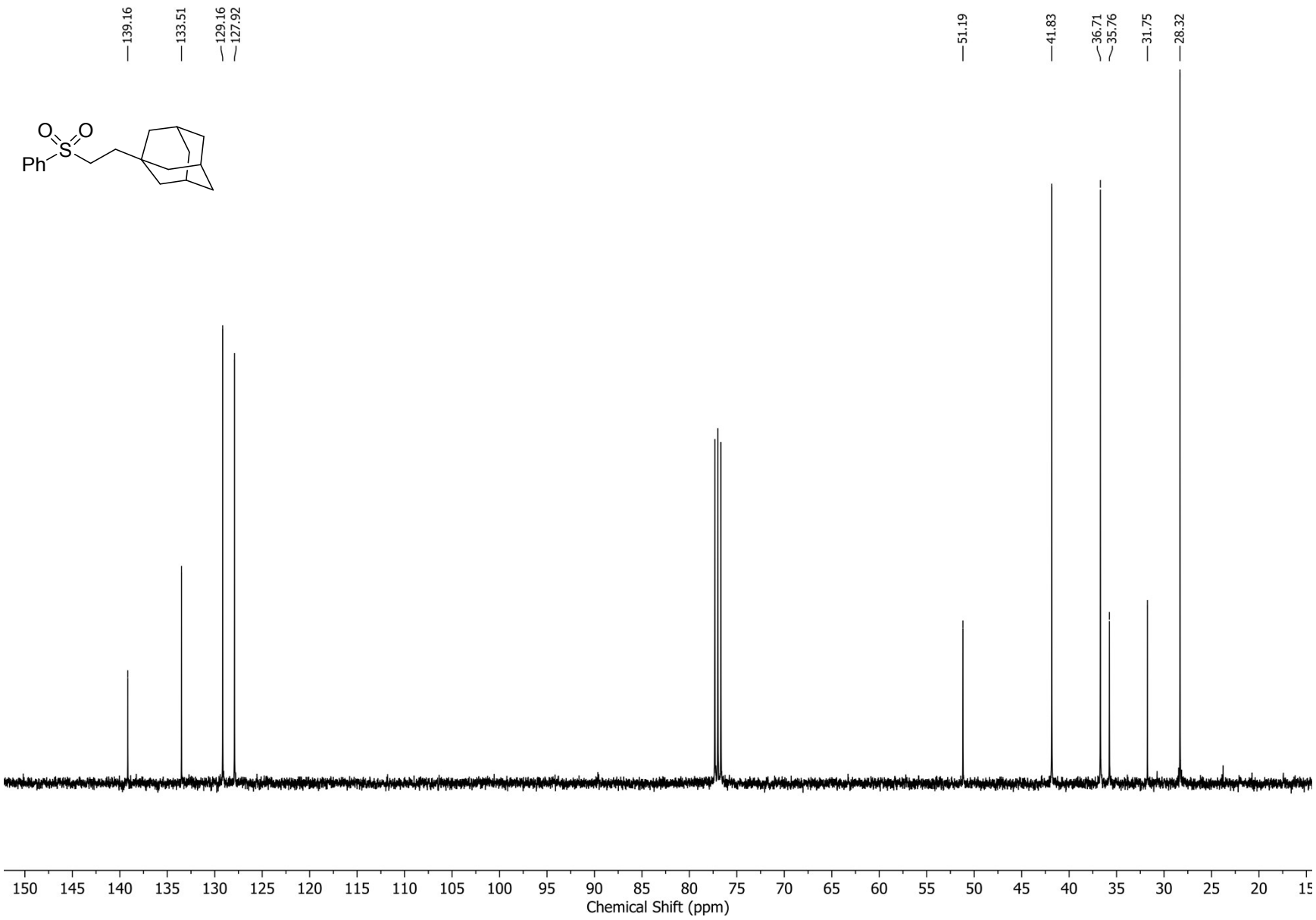
(Hexylsulfonyl)benzene ^{1}H and ^{13}C NMR



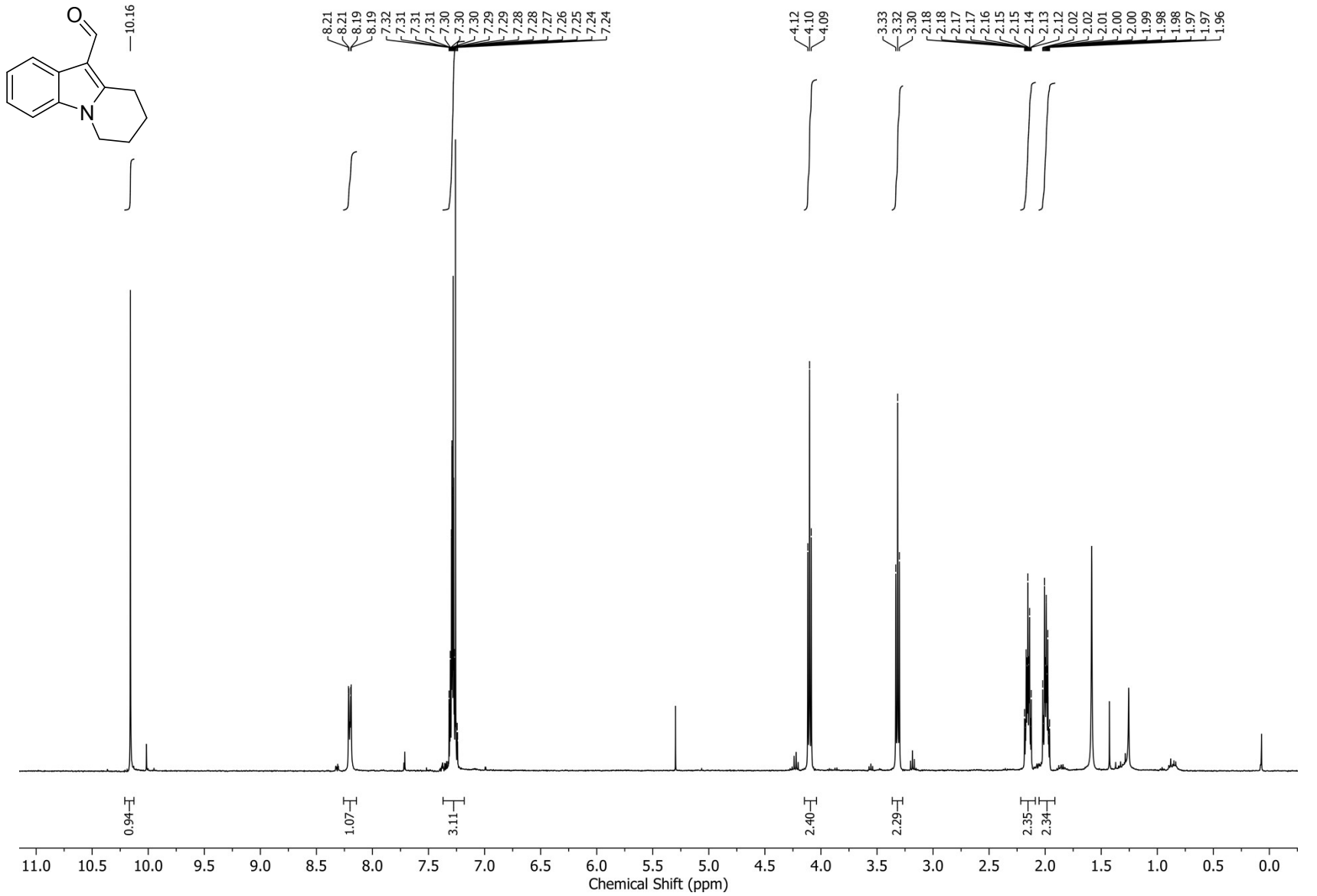

139.29
133.68
129.32
128.11
56.38
31.21
28.00
22.66
22.33
13.96
O
O
S
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

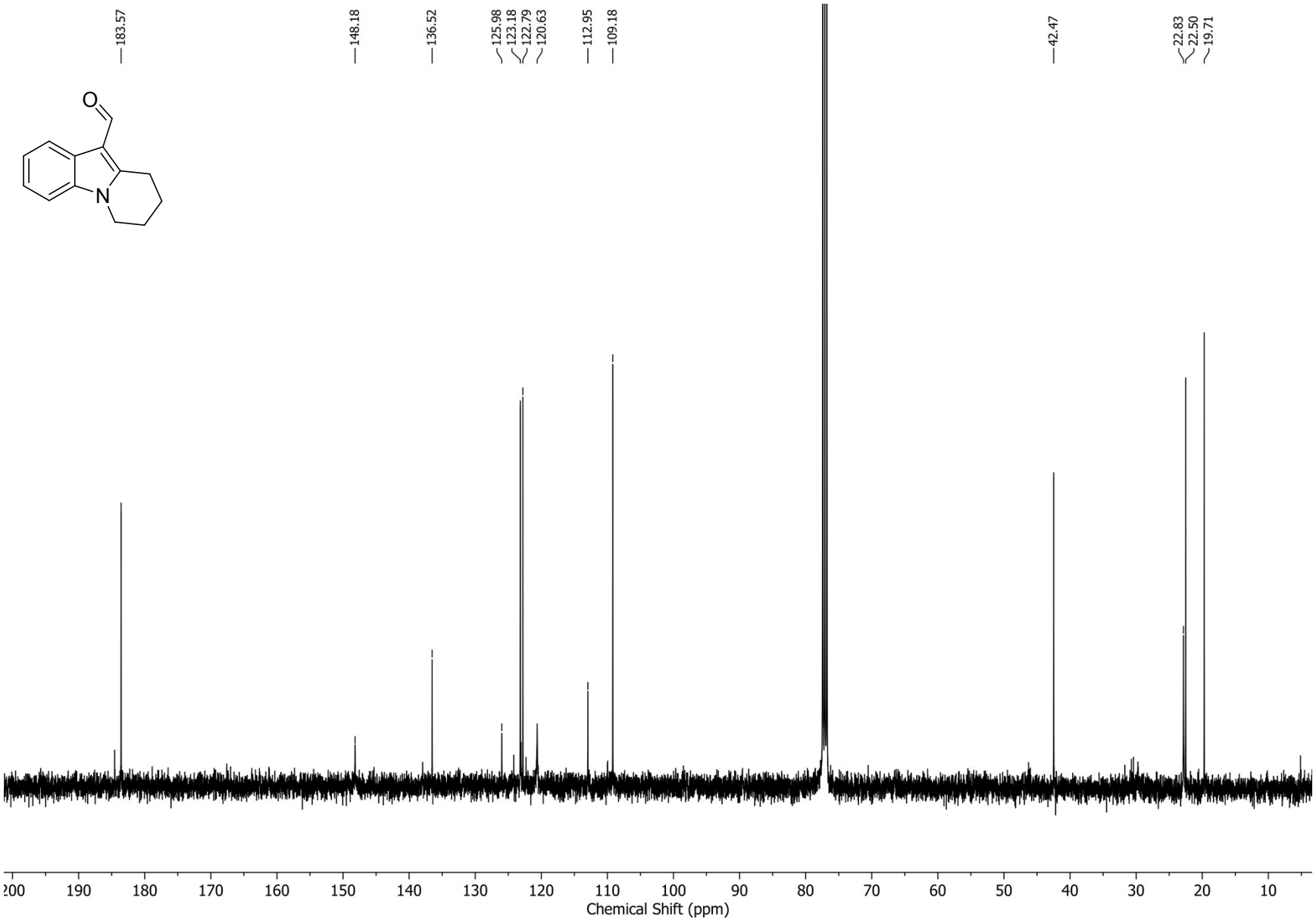
(3r,5r,7r)-1-[2-(Phenylsulfonyl)ethyl]adamantane ^{1}H and ^{13}C NMR



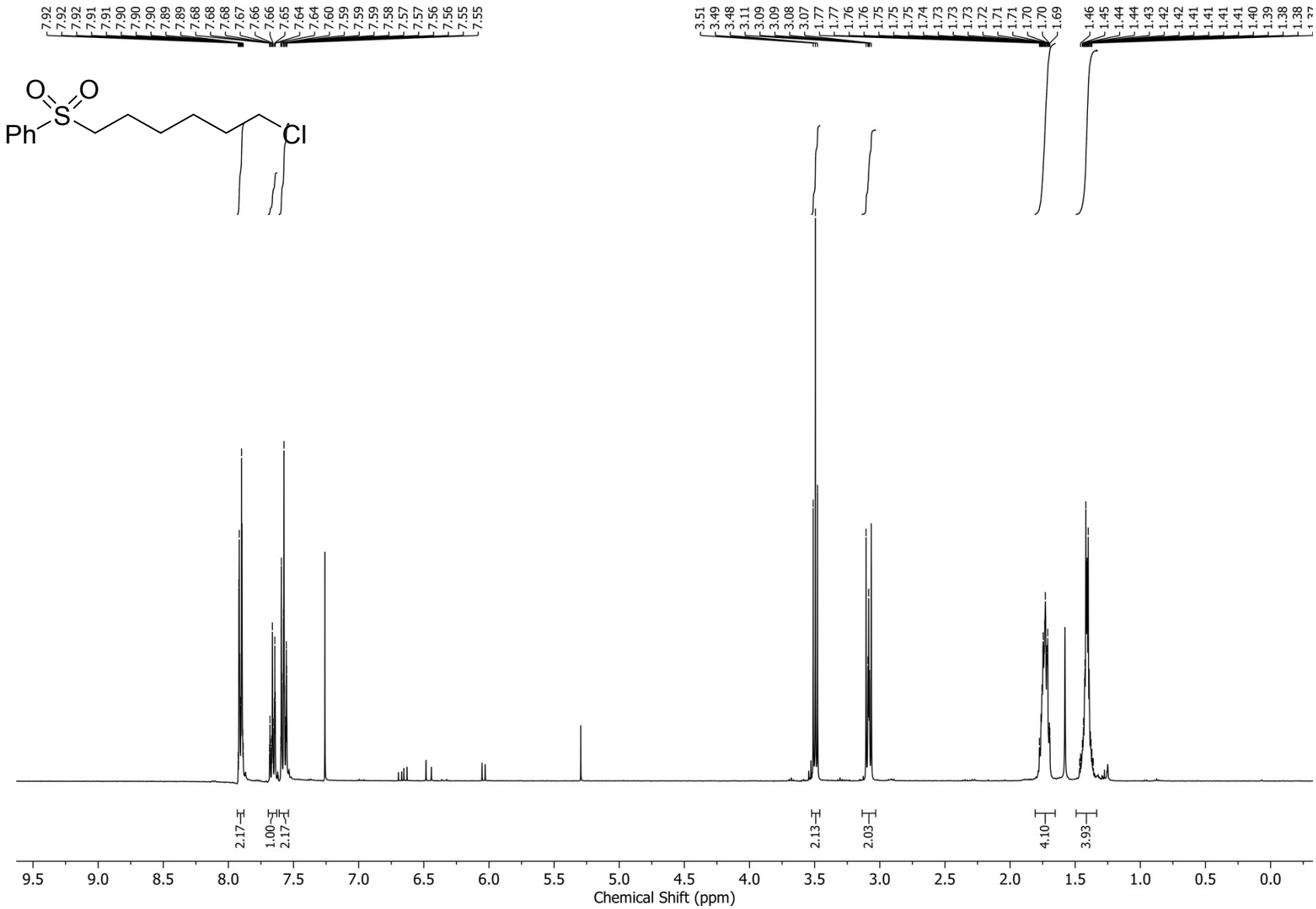

139.16
133.51
129.16
127.92
51.19
41.83
36.71
35.76
31.75
28.32
O
O
S
Ph
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15
Chemical Shift (ppm)

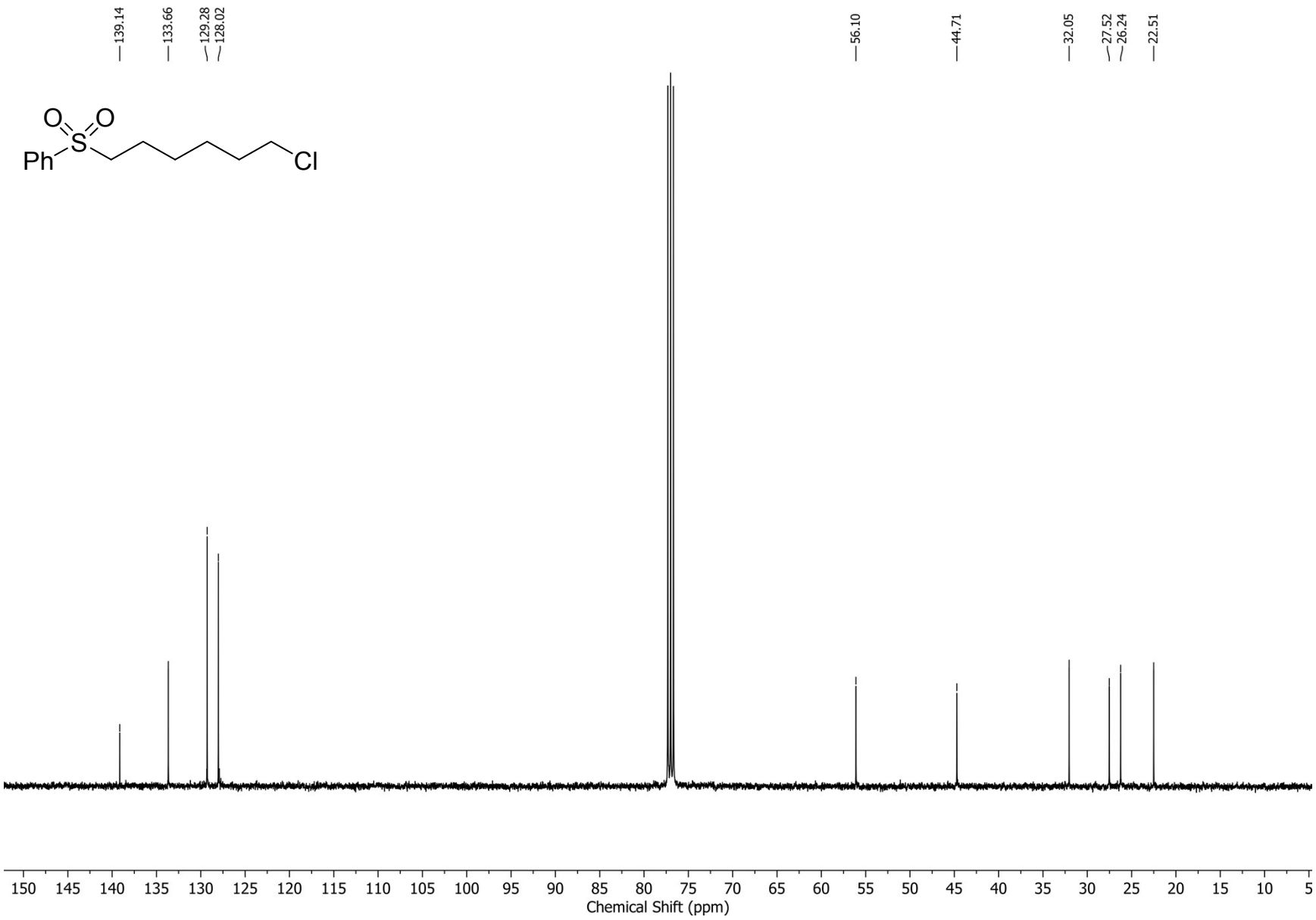
6,7,8,9-Tetrahydropyrido[1,2-*a*]indole-10-carbaldehyde ^{1}H and ^{13}C NMR



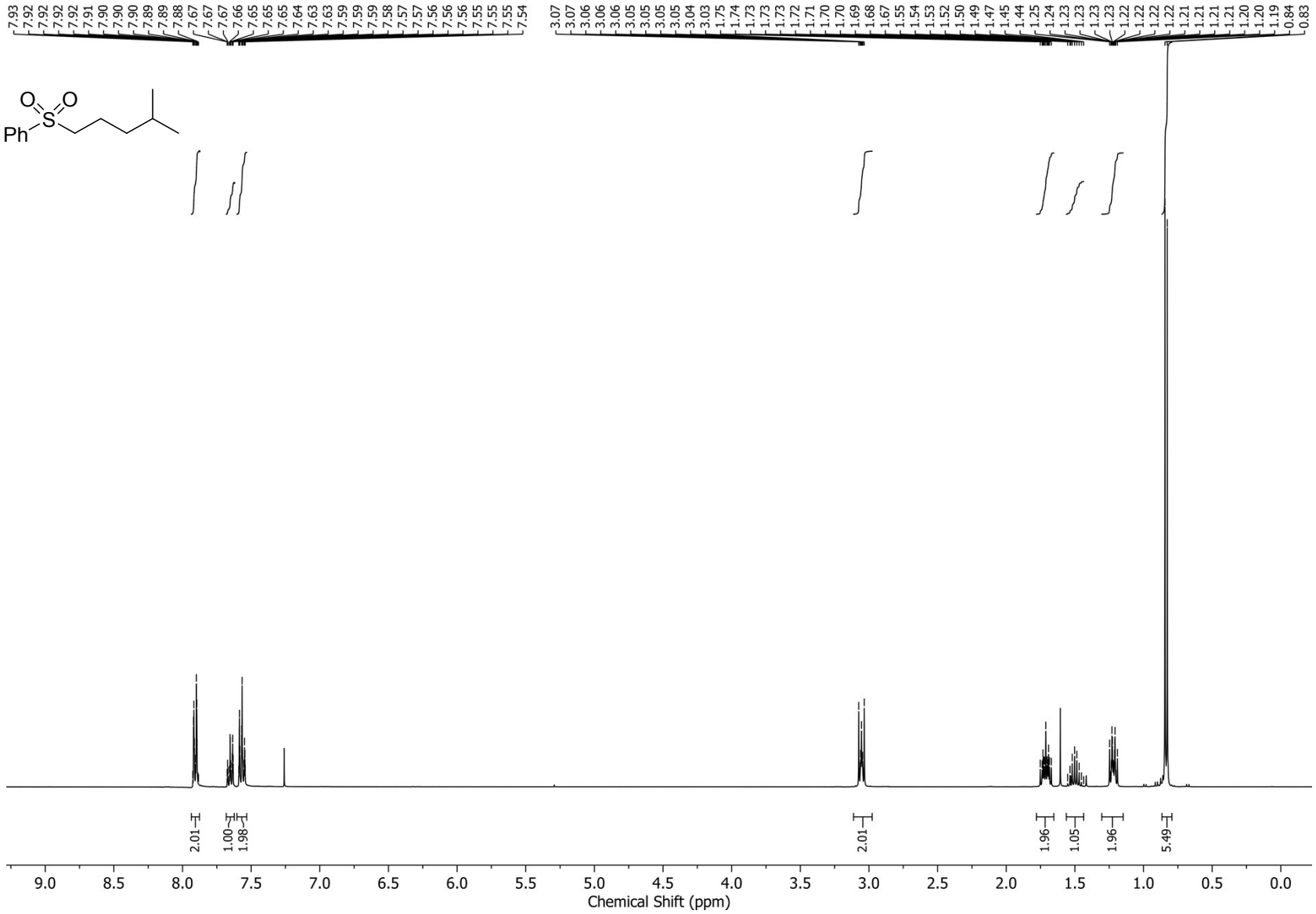

183.57
148.18
136.52
125.98
123.18
122.79
120.63
112.95
109.18
42.47
22.83
22.50
19.71
O
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

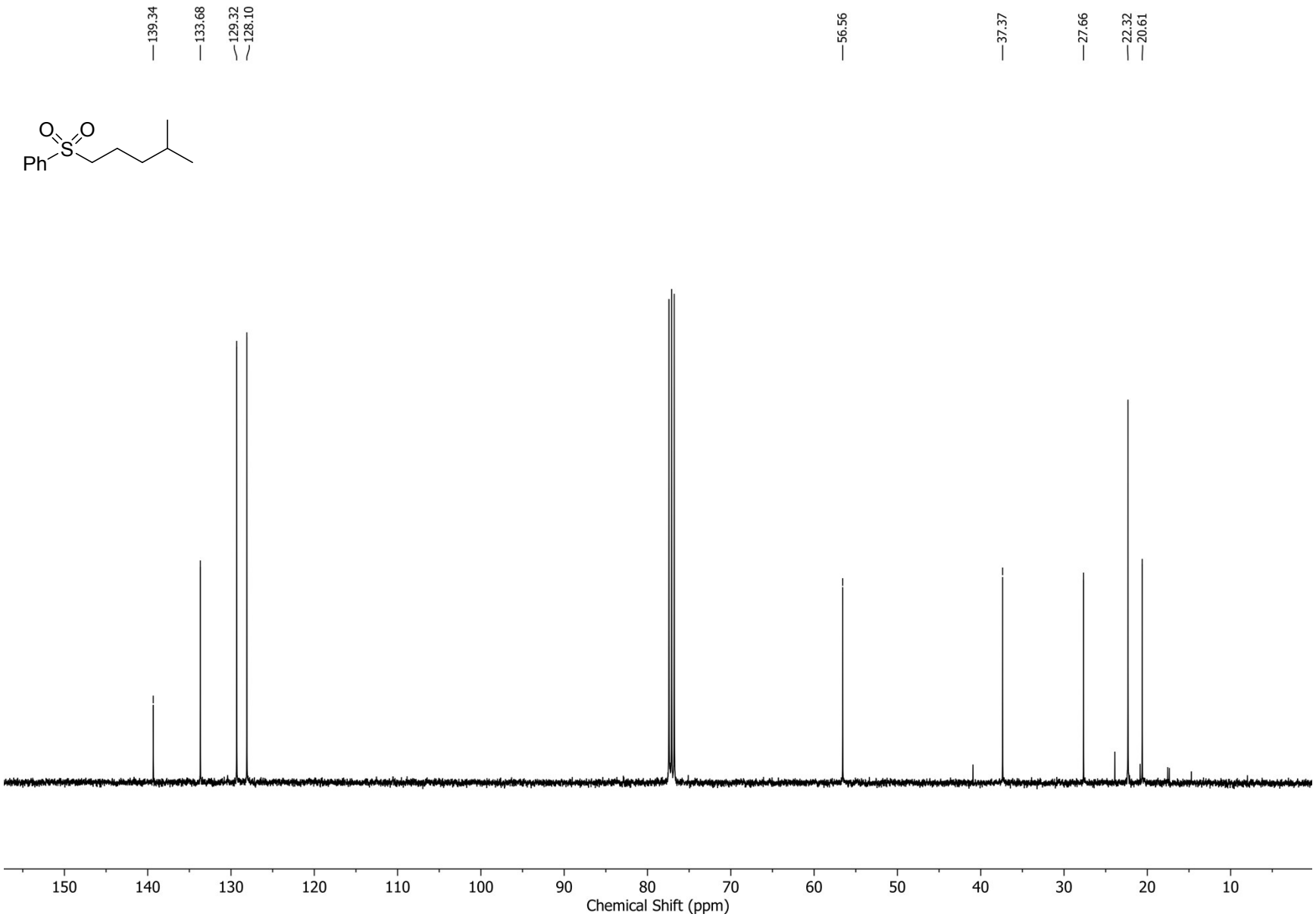
((6-Chlorohexyl)sulfonyl)benzene ^{1}H and ^{13}C NMR



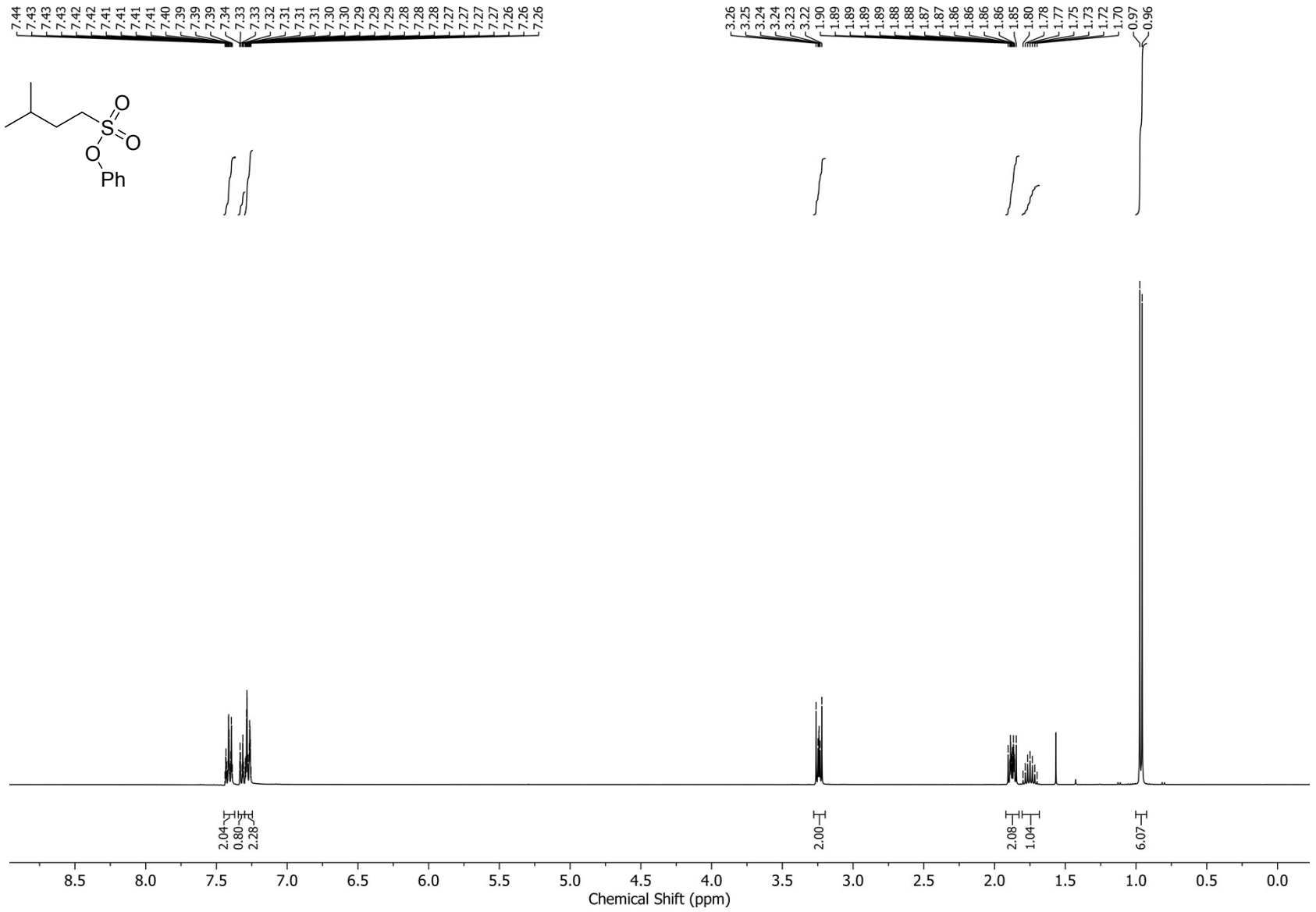

139.14
133.66
129.28
128.02
56.10
44.71
32.05
27.52
26.24
22.51
O
O
S
Ph
Cl
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
Chemical Shift (ppm)

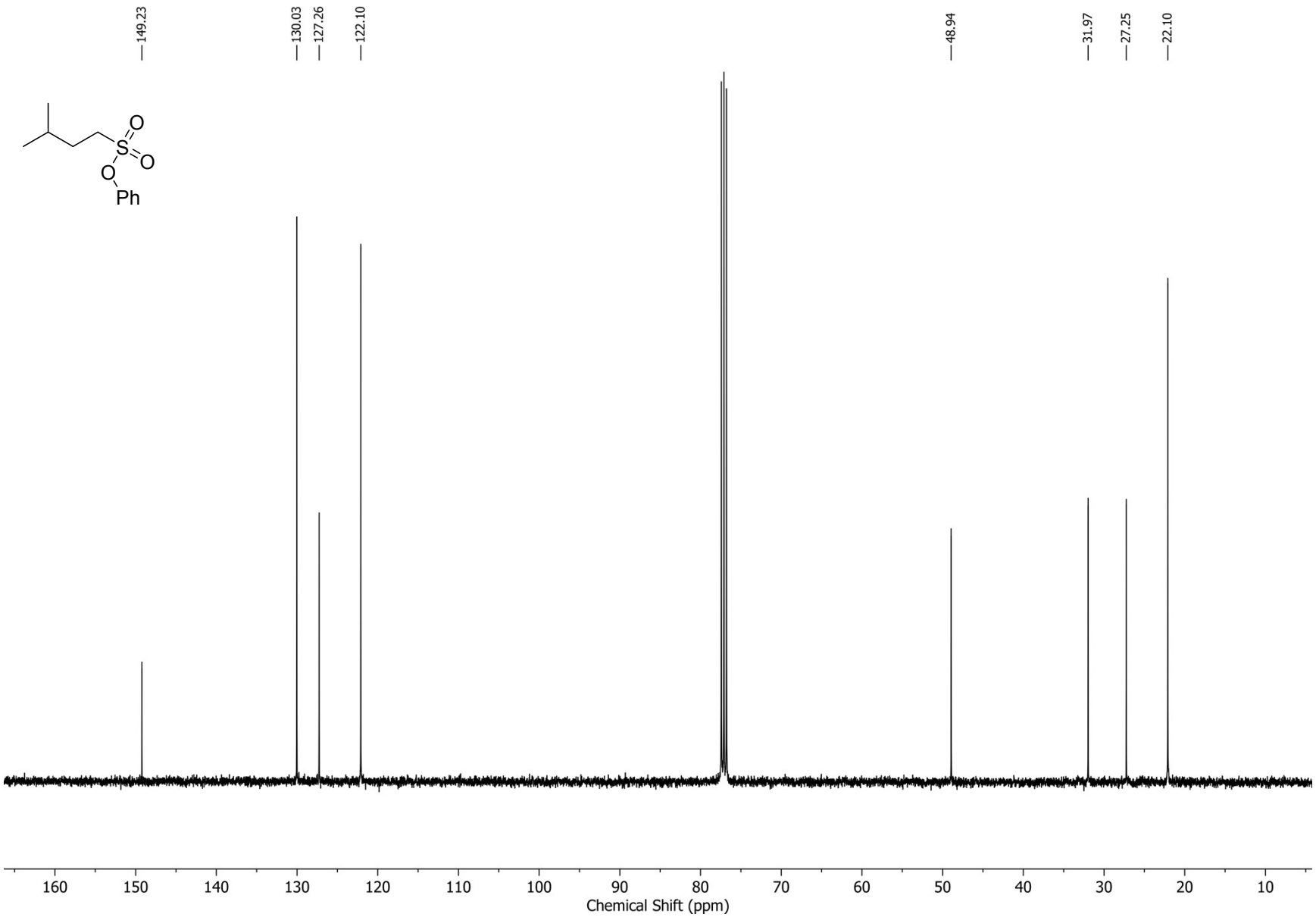
4-Methylpentyl phenyl sulfone ^{1}H and ^{13}C NMR



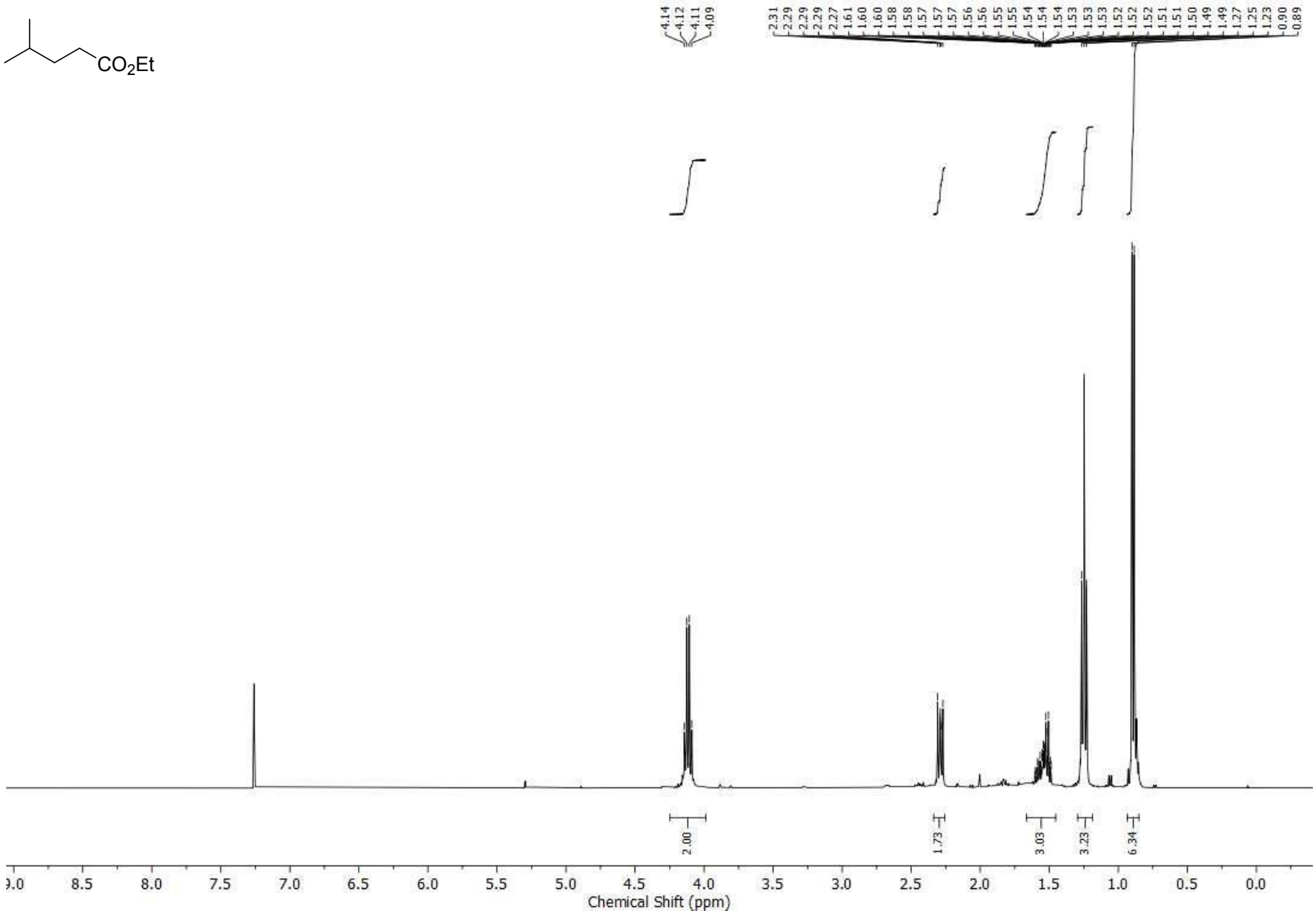

139.34
133.68
129.32
128.10
56.56
37.37
27.66
22.32
20.61
O
O
S
Ph
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

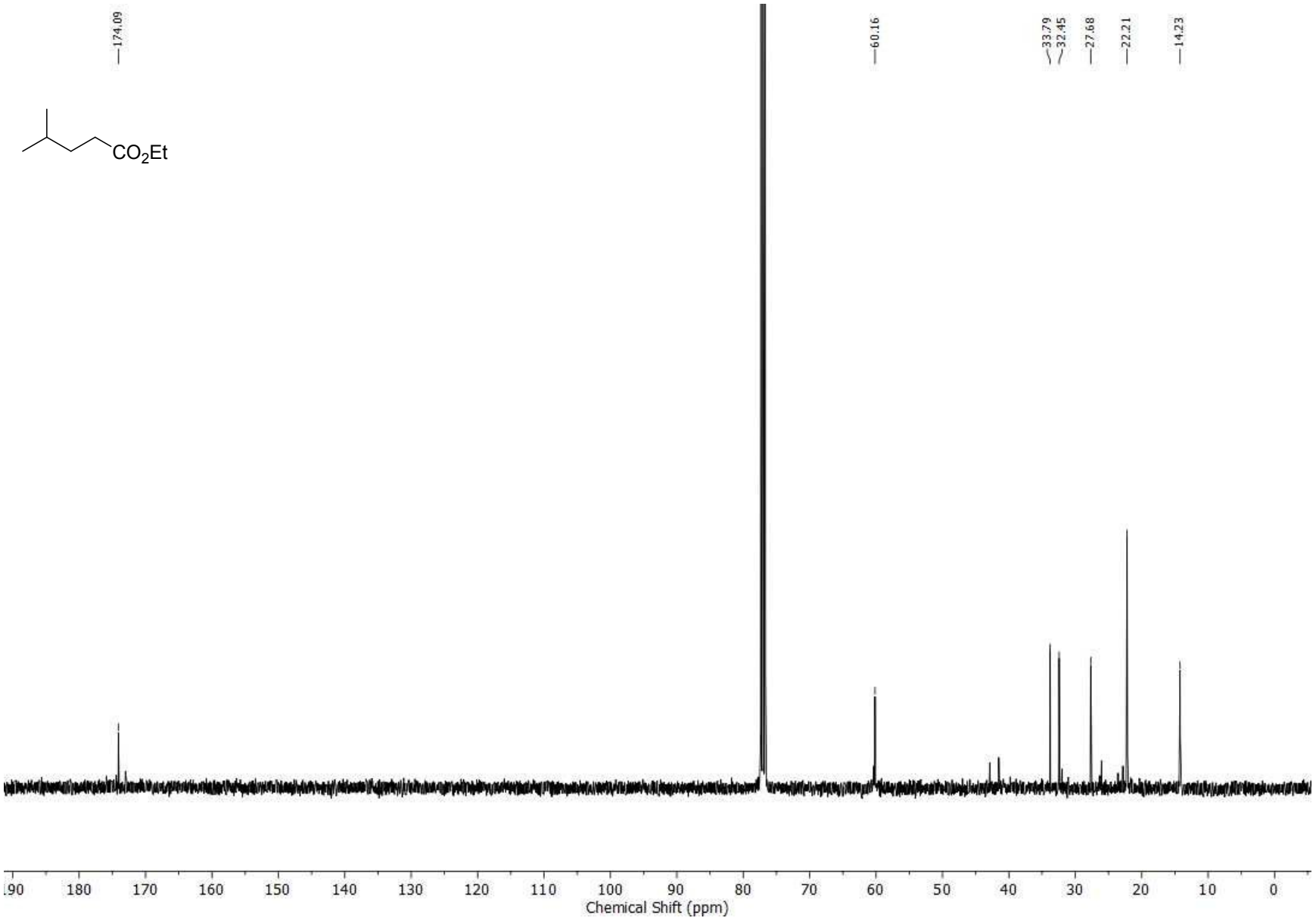
Phenyl 3-Methylbutane-1-sulfonate ^{1}H and ^{13}C NMR



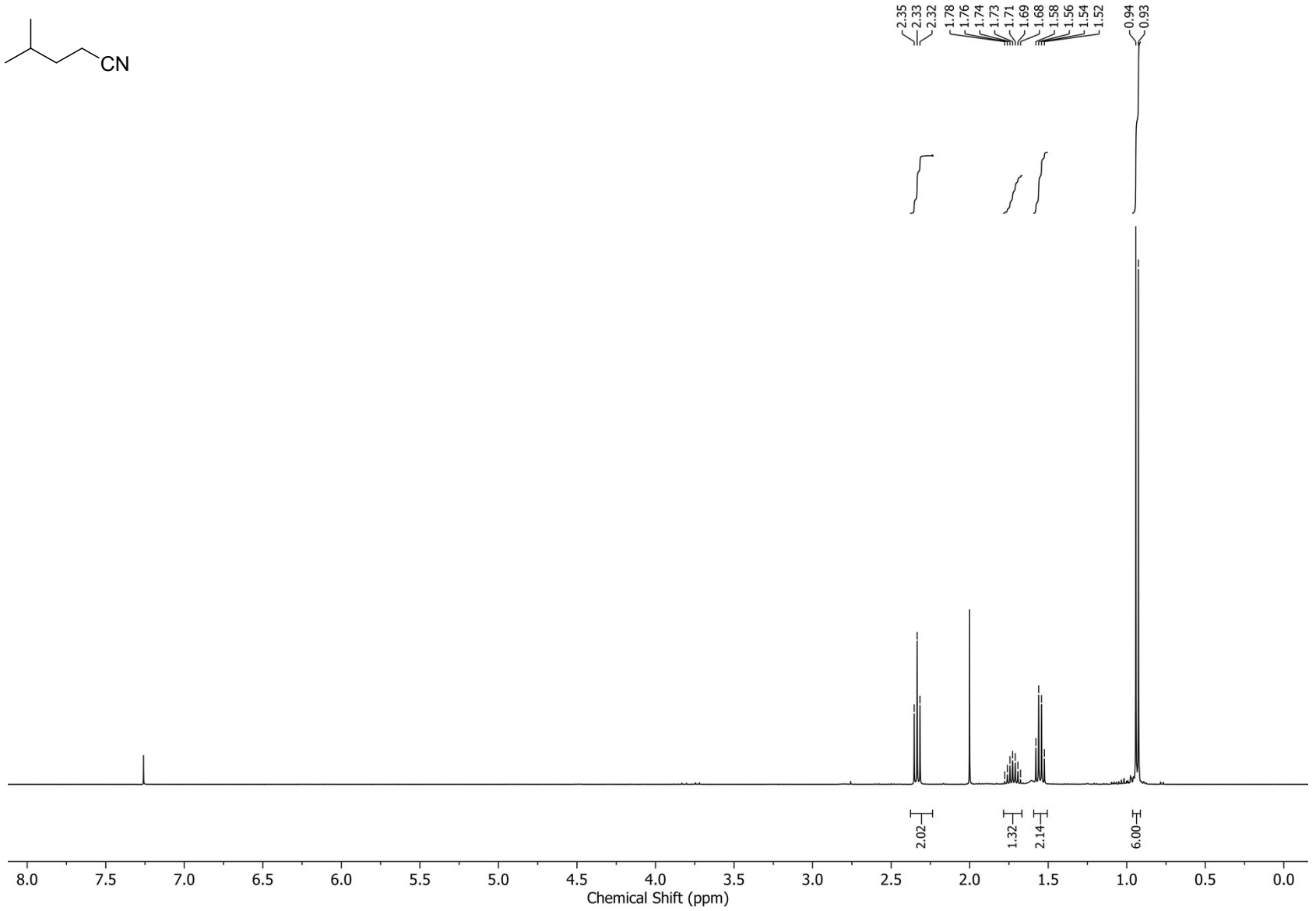

149.23
130.03
127.26
122.10
48.94
31.97
27.25
22.10
O
S
O
O
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

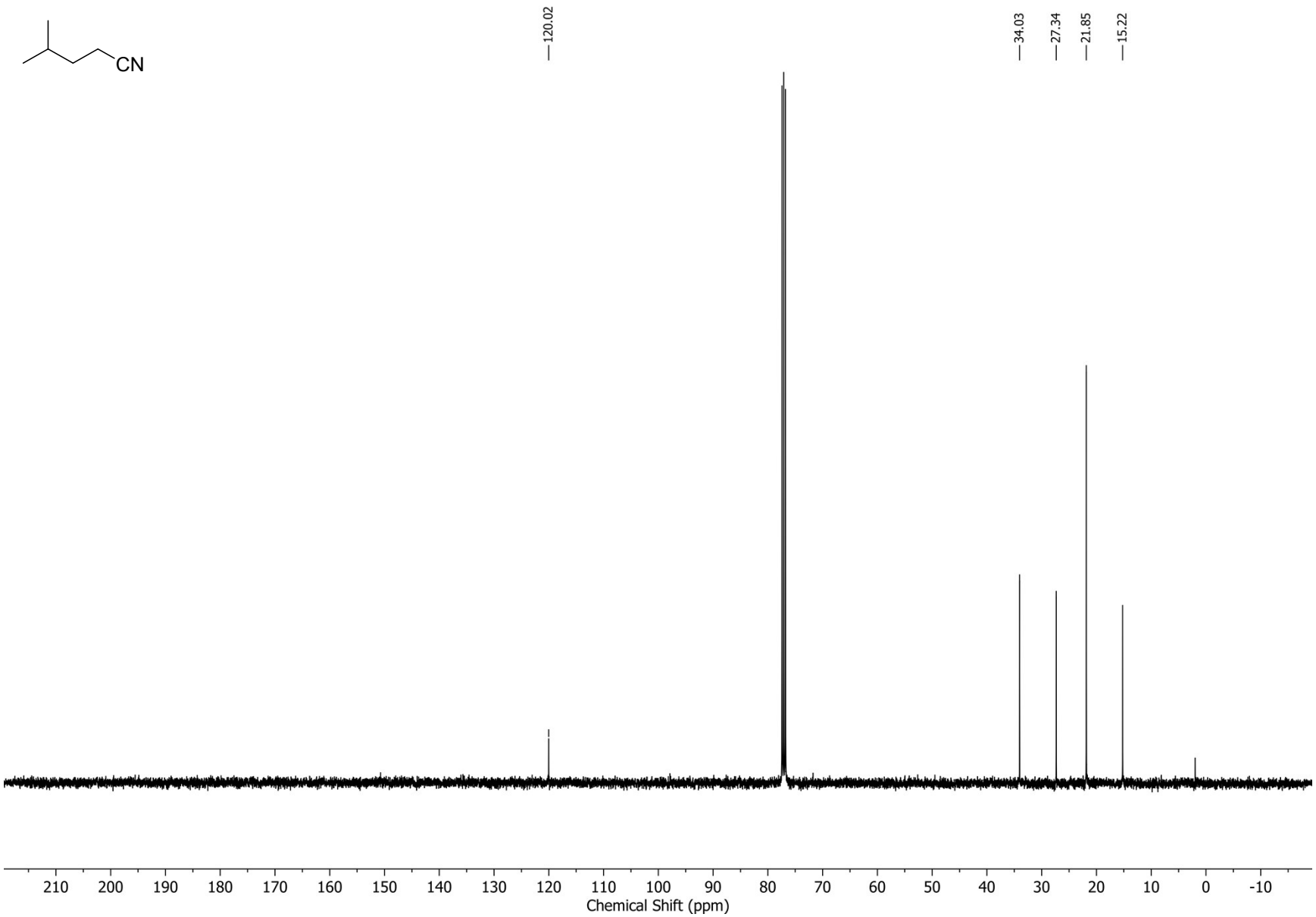
Ethyl 4-methylpentanoate ^{1}H and ^{13}C NMR



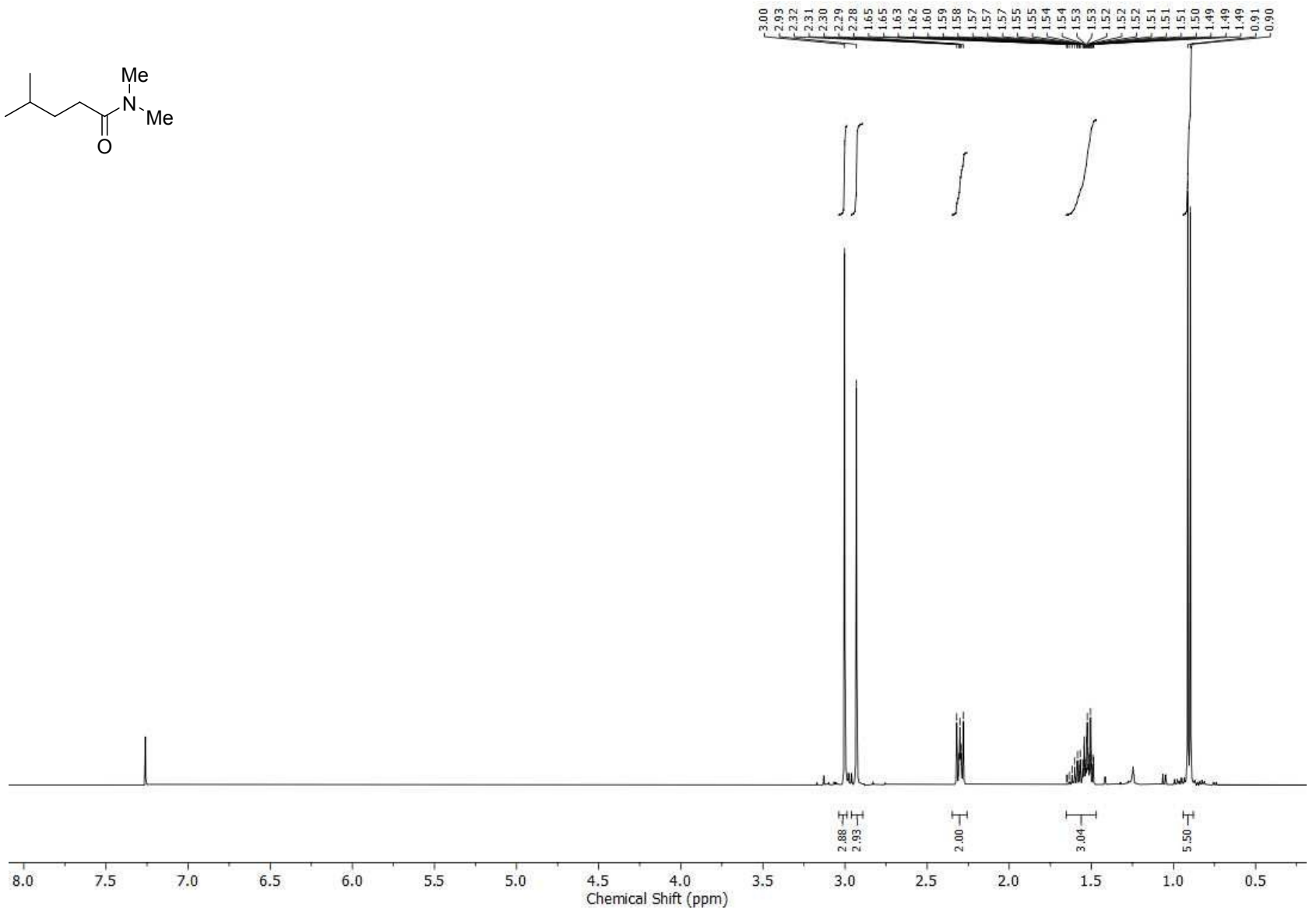

174.09
60.16
33.79
32.45
27.68
22.21
14.23
CO2Et
Chemical Shift (ppm)

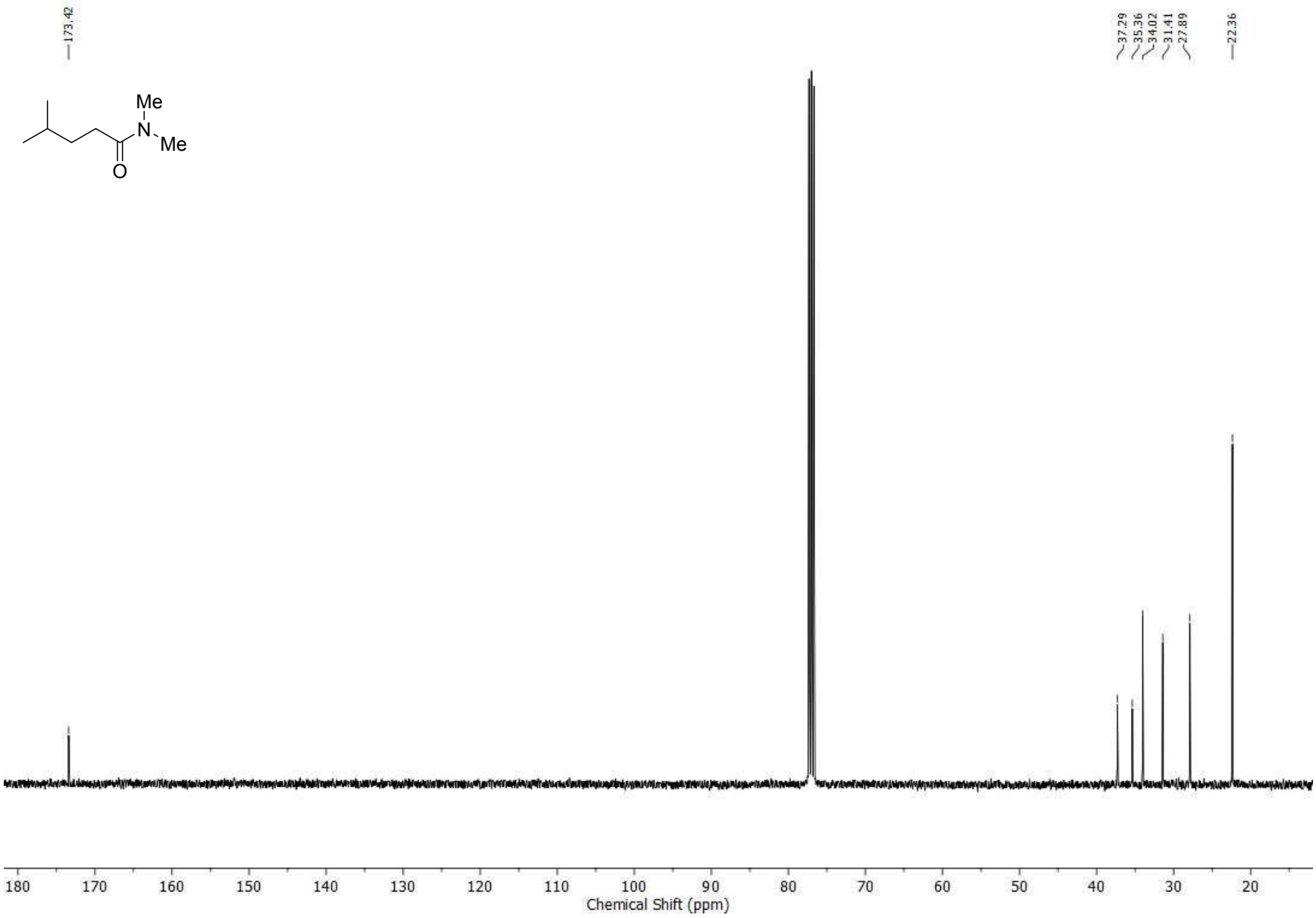
4-Methylpentanenitrile ^{1}H and ^{13}C NMR



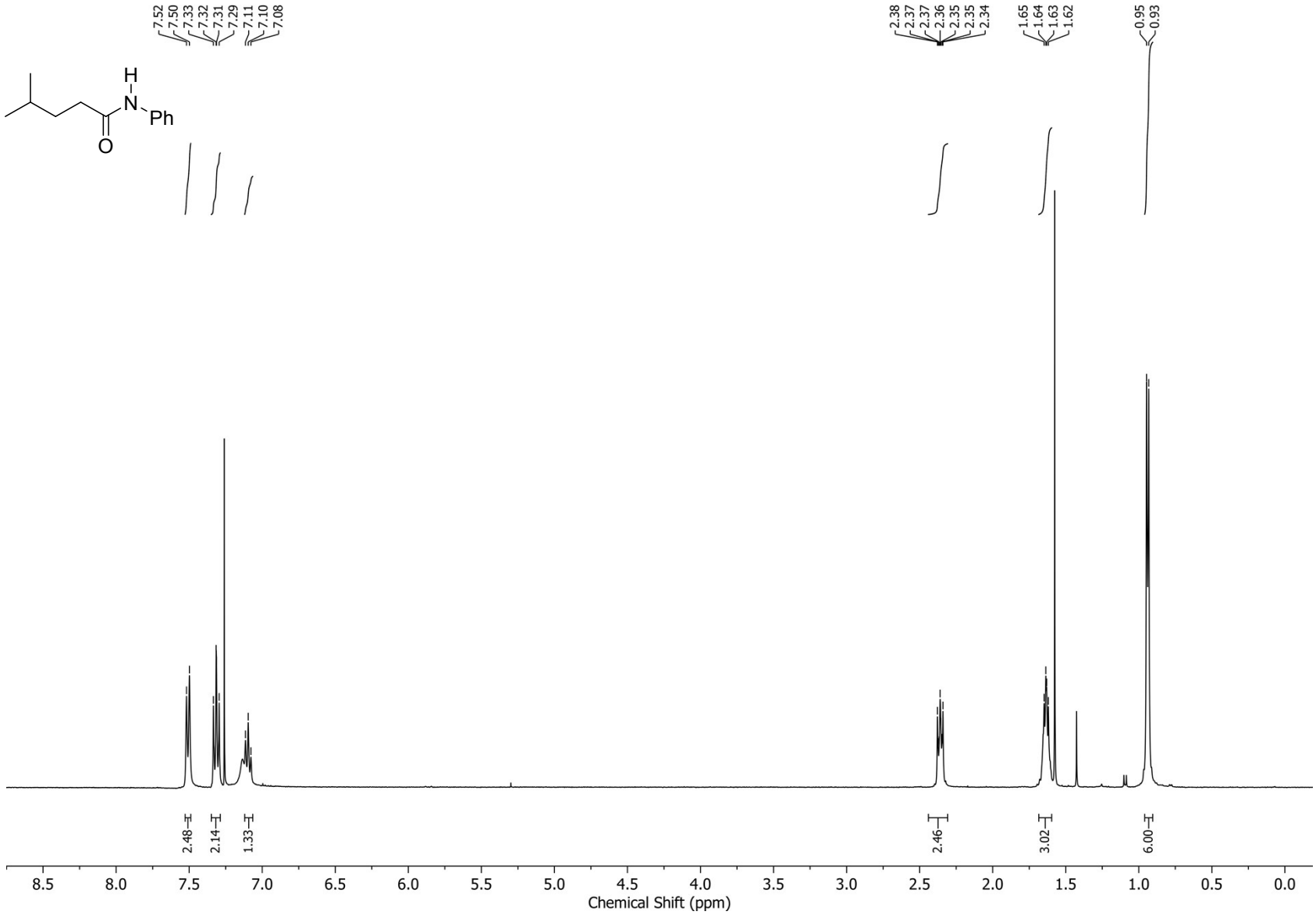

CN
120.02
34.03
27.34
21.85
15.22
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
Chemical Shift (ppm)

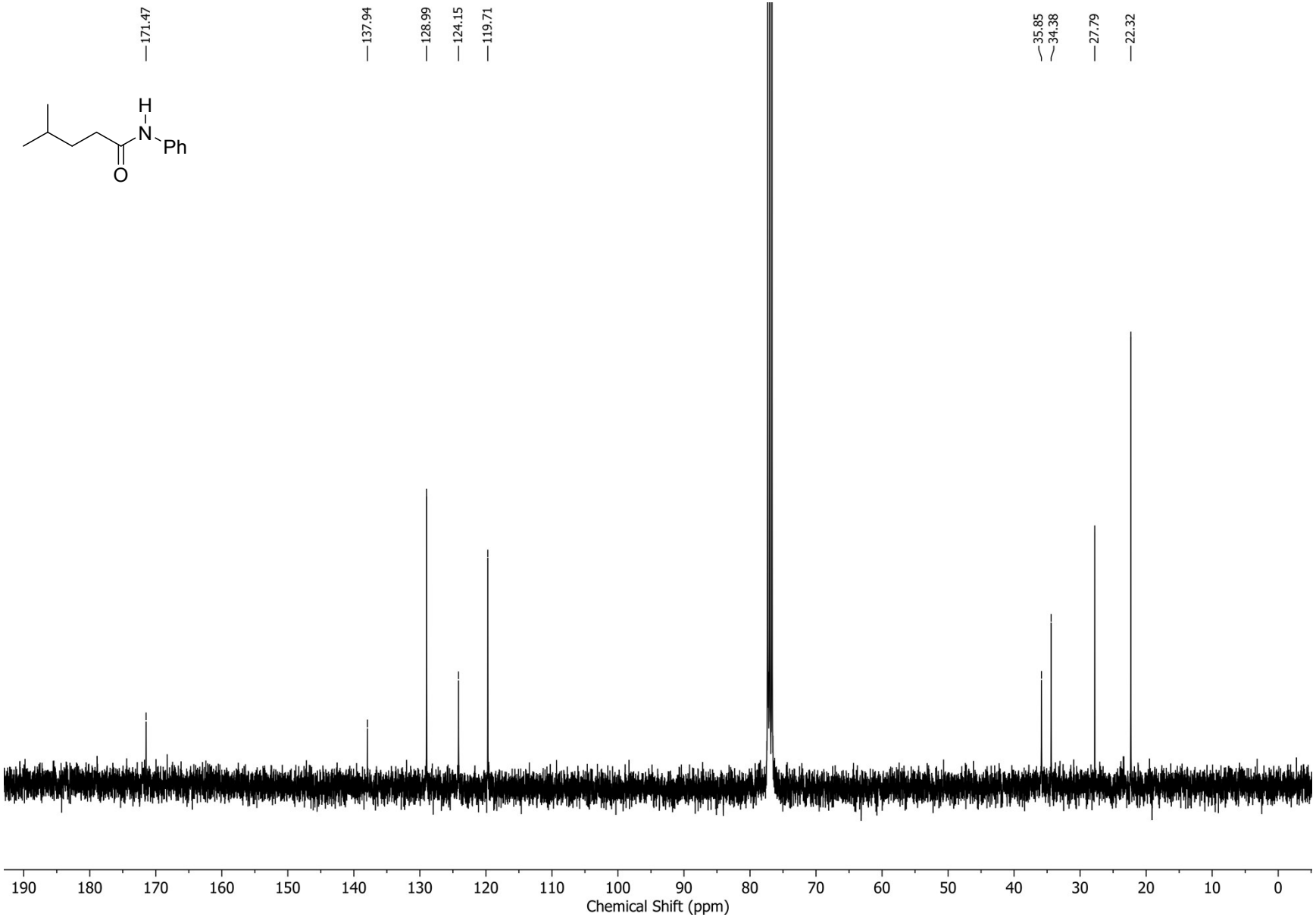
***N*,*N*,4-Trimethylpentanamide ^{1}H and ^{13}C NMR**



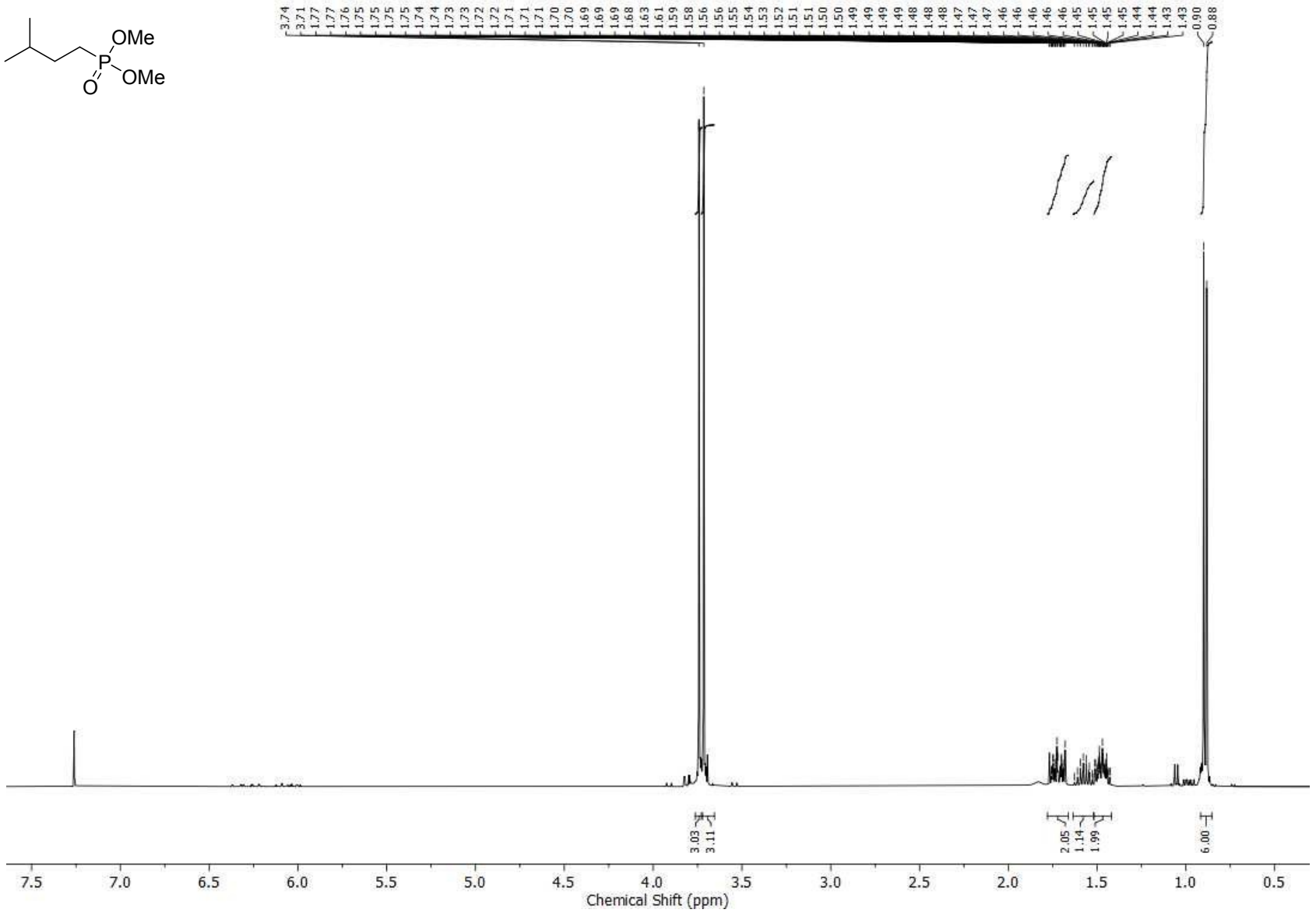

173.42
37.29
35.36
34.02
31.41
27.89
22.36
Me
N
Me
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
Chemical Shift (ppm)

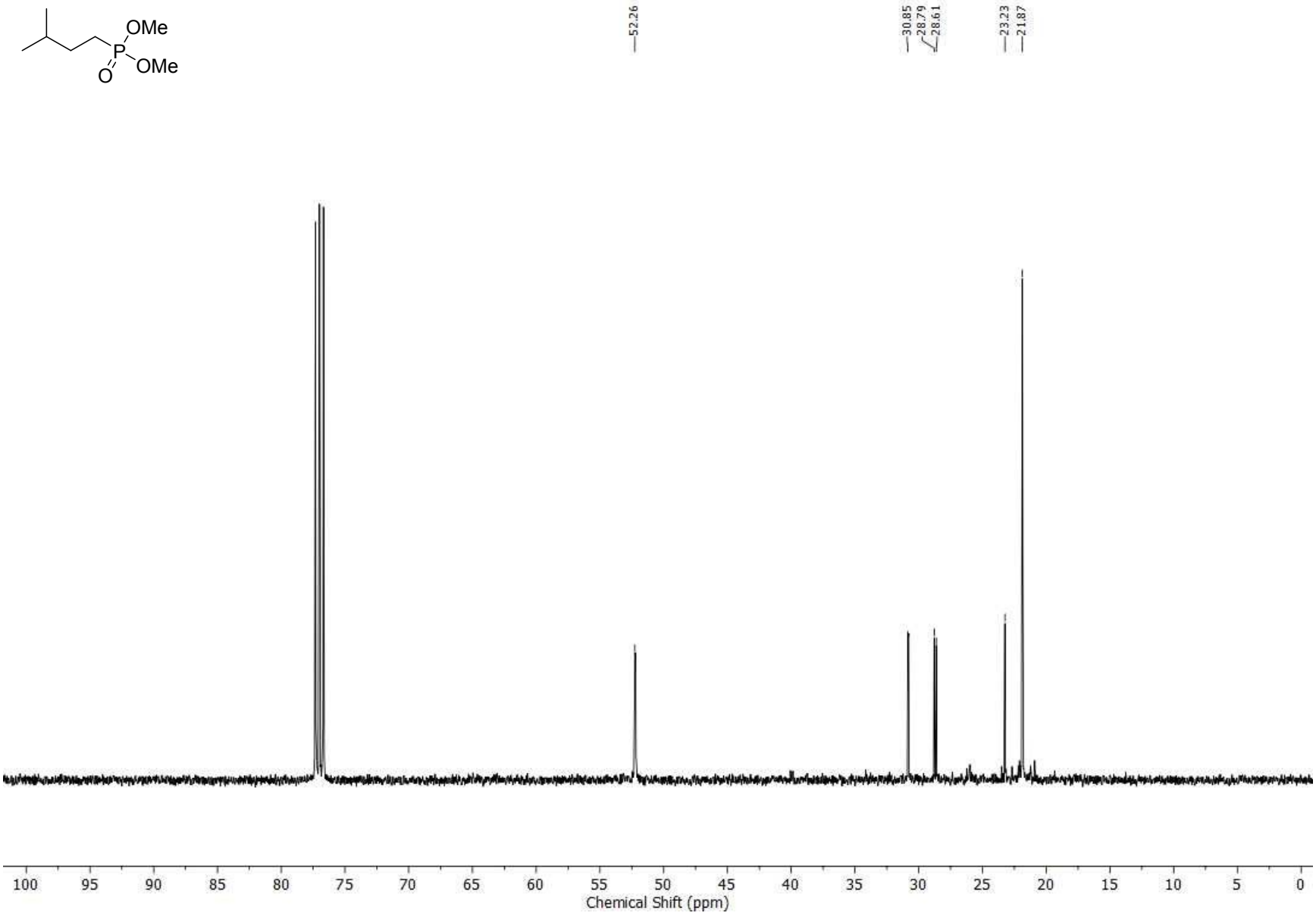
4-Methyl-*N*-phenylpentanamide ^{1}H and ^{13}C NMR



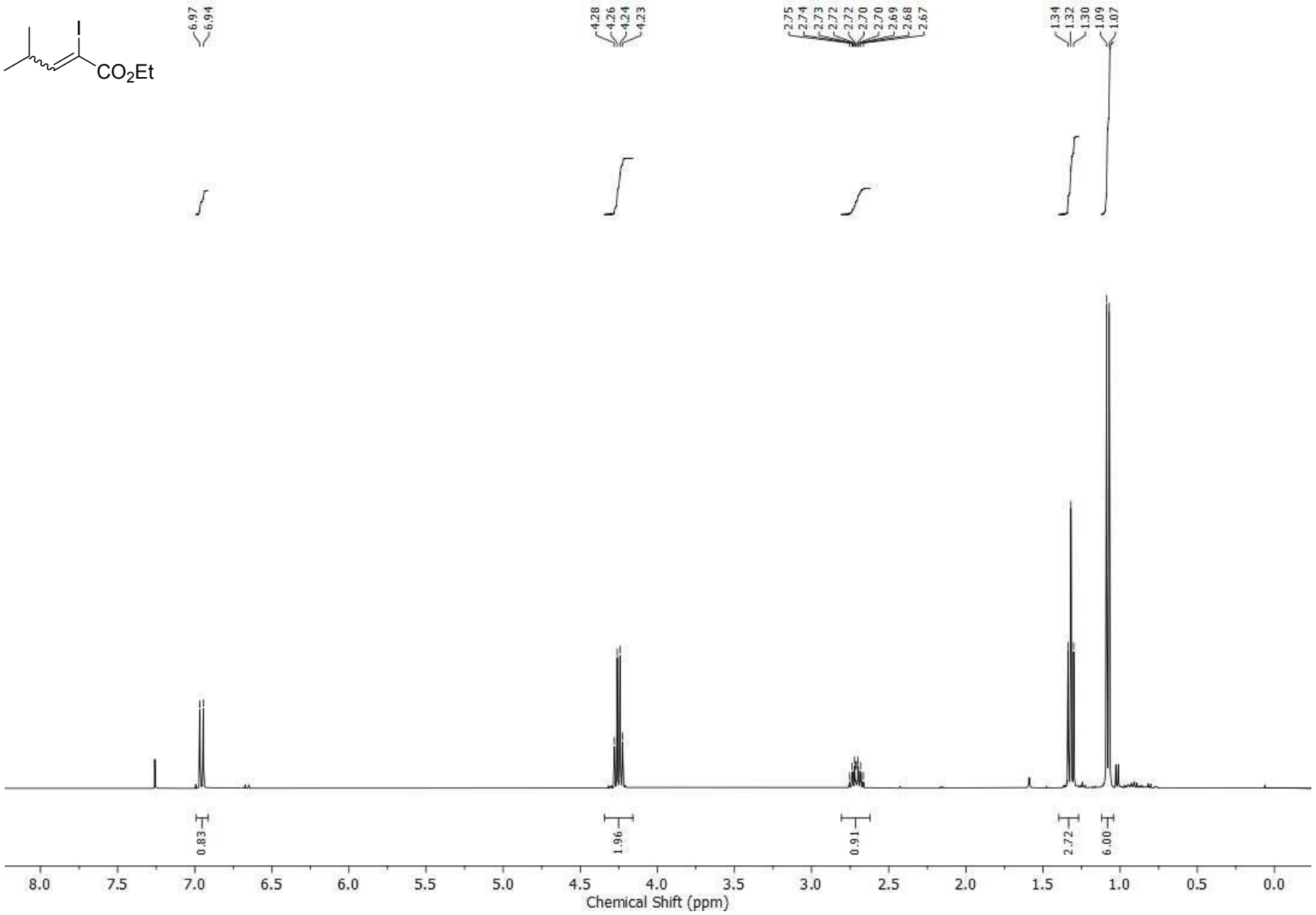

171.47
137.94
128.99
124.15
119.71
35.85
34.38
27.79
22.32
H
N
Ph
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

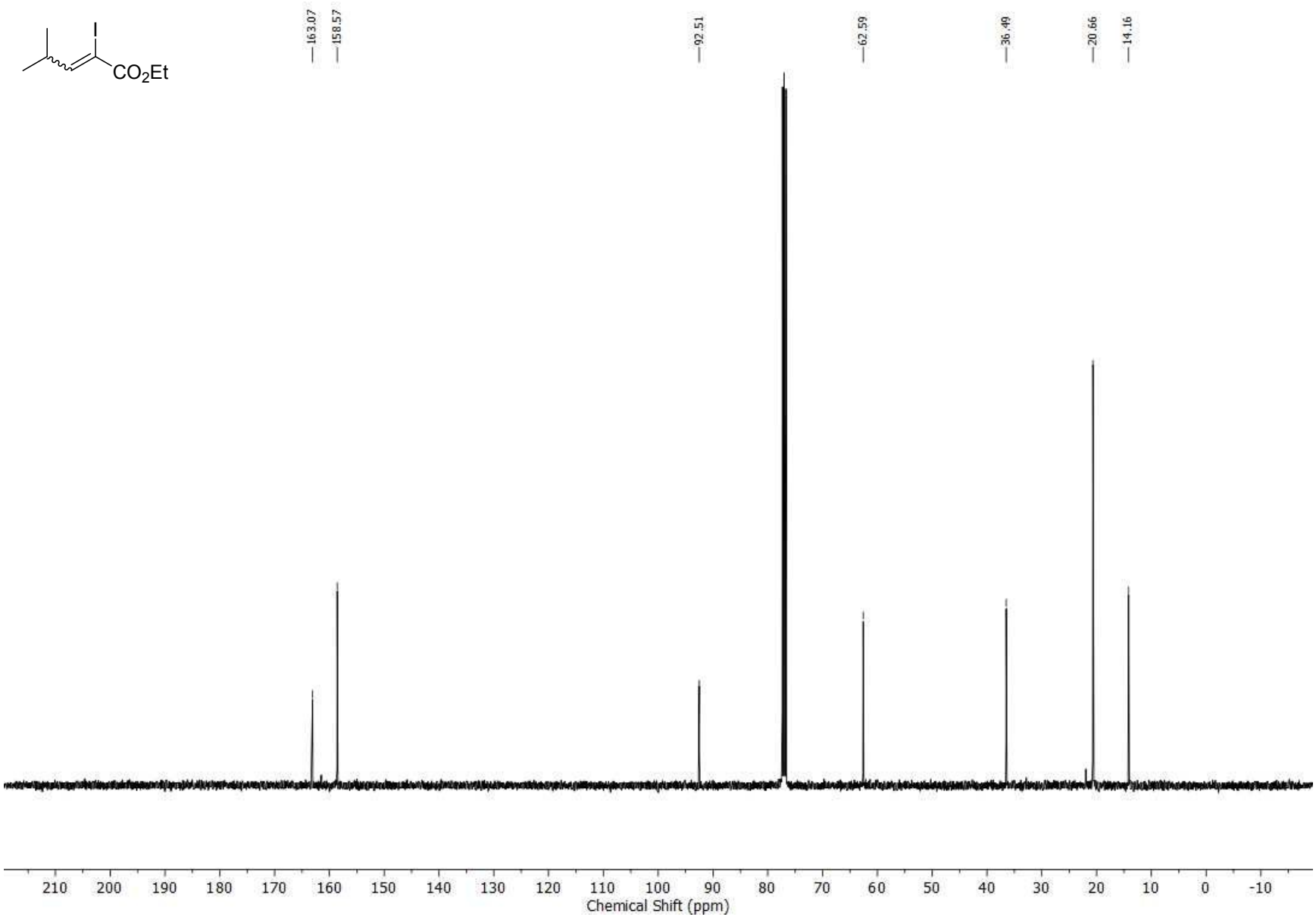
Dimethyl isopentylphosphonate ^{1}H and ^{13}C NMR



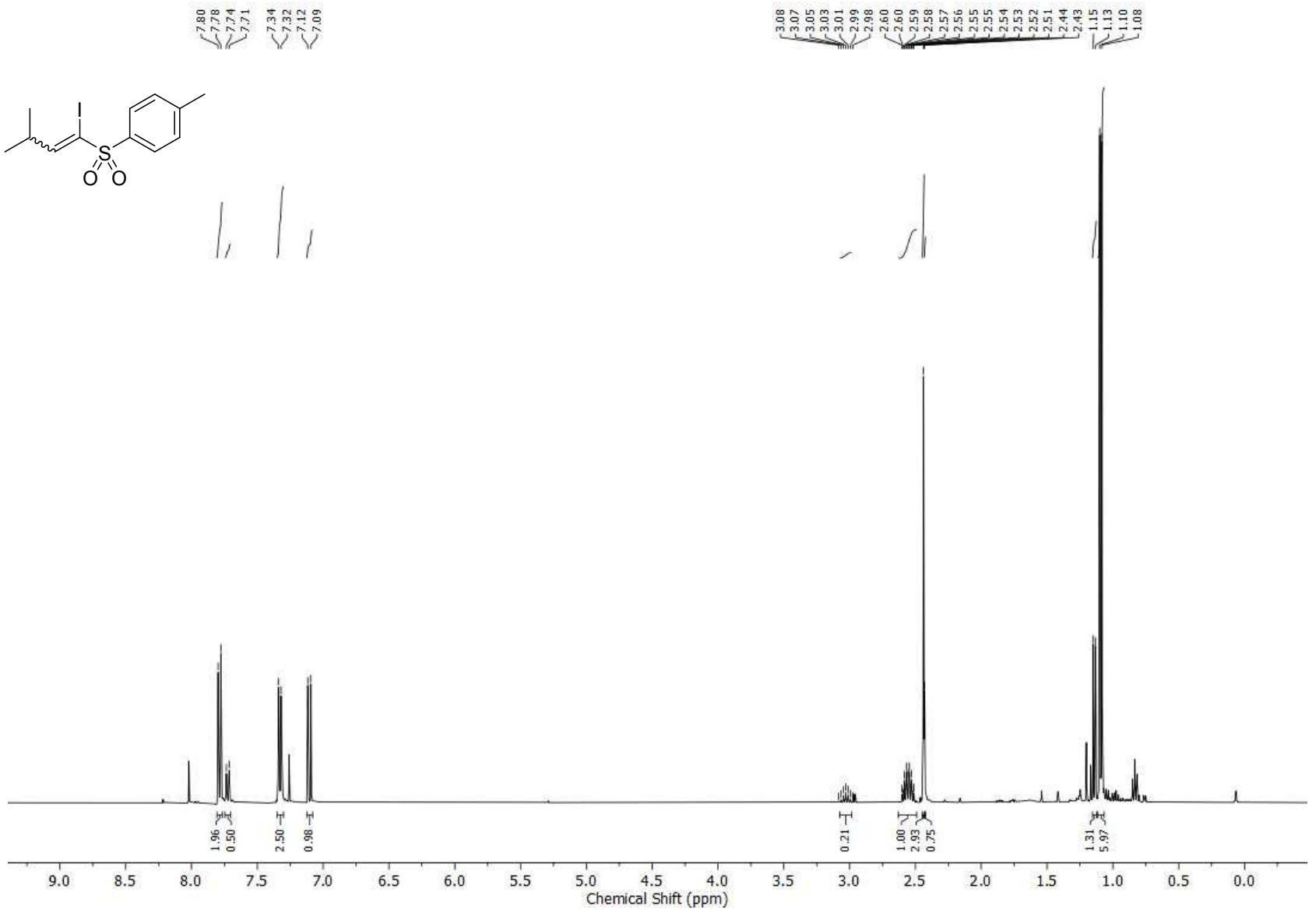

OMe
P
O
OMe
52.26
30.85
28.79
28.61
23.23
21.87
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
Chemical Shift (ppm)

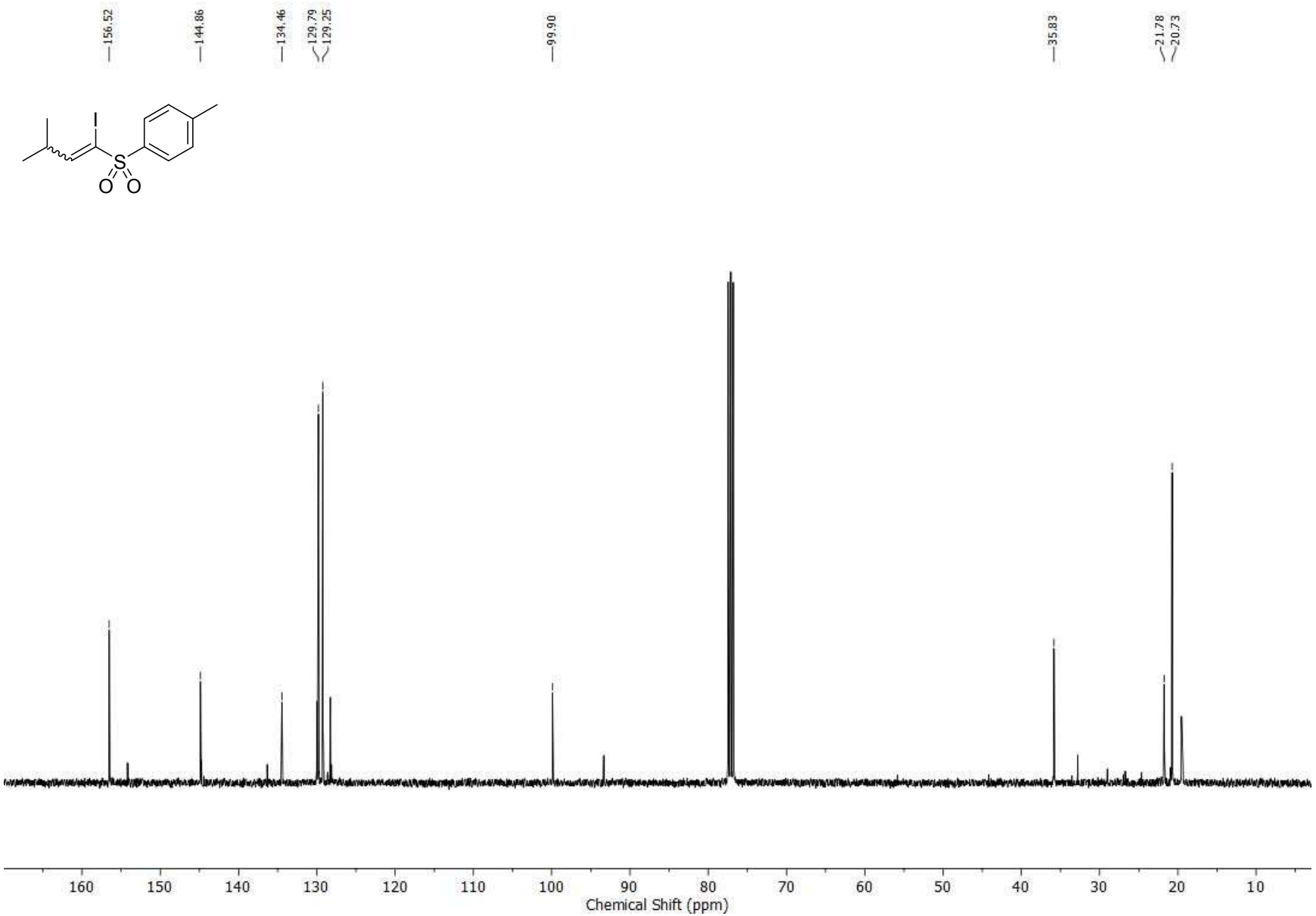
Ethyl 2-iodo-4-methylpent-2-enoate ^{1}H and ^{13}C NMR



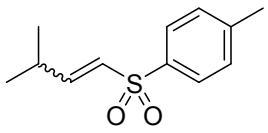

I
CO2Et
163.07
158.57
92.51
62.59
36.49
20.66
14.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
Chemical Shift (ppm)

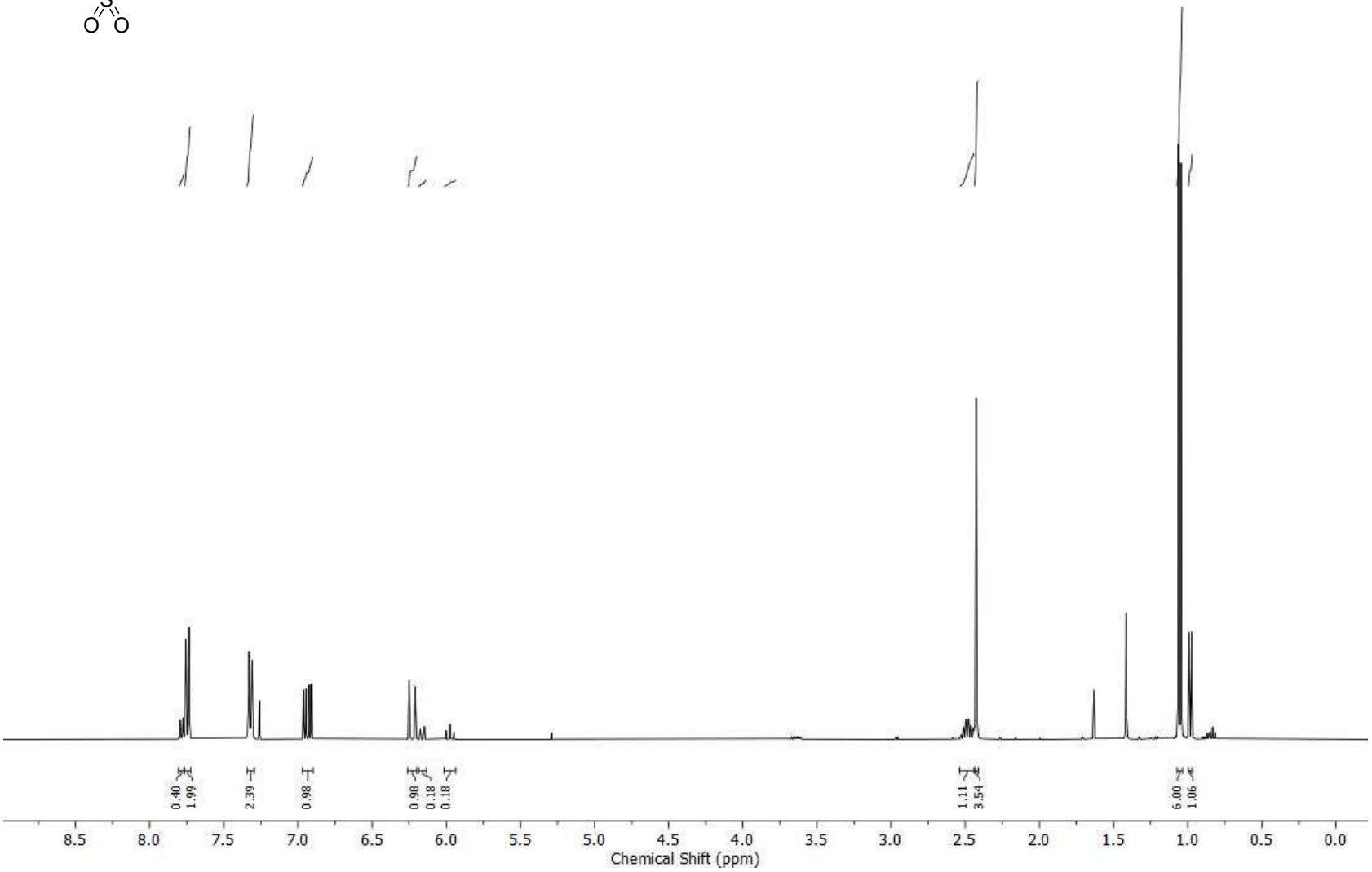
1-((1-Iodo-3-methylbut-1-en-1-yl)sulfonyl)-4-methylbenzene ^{1}H and ^{13}C NMR

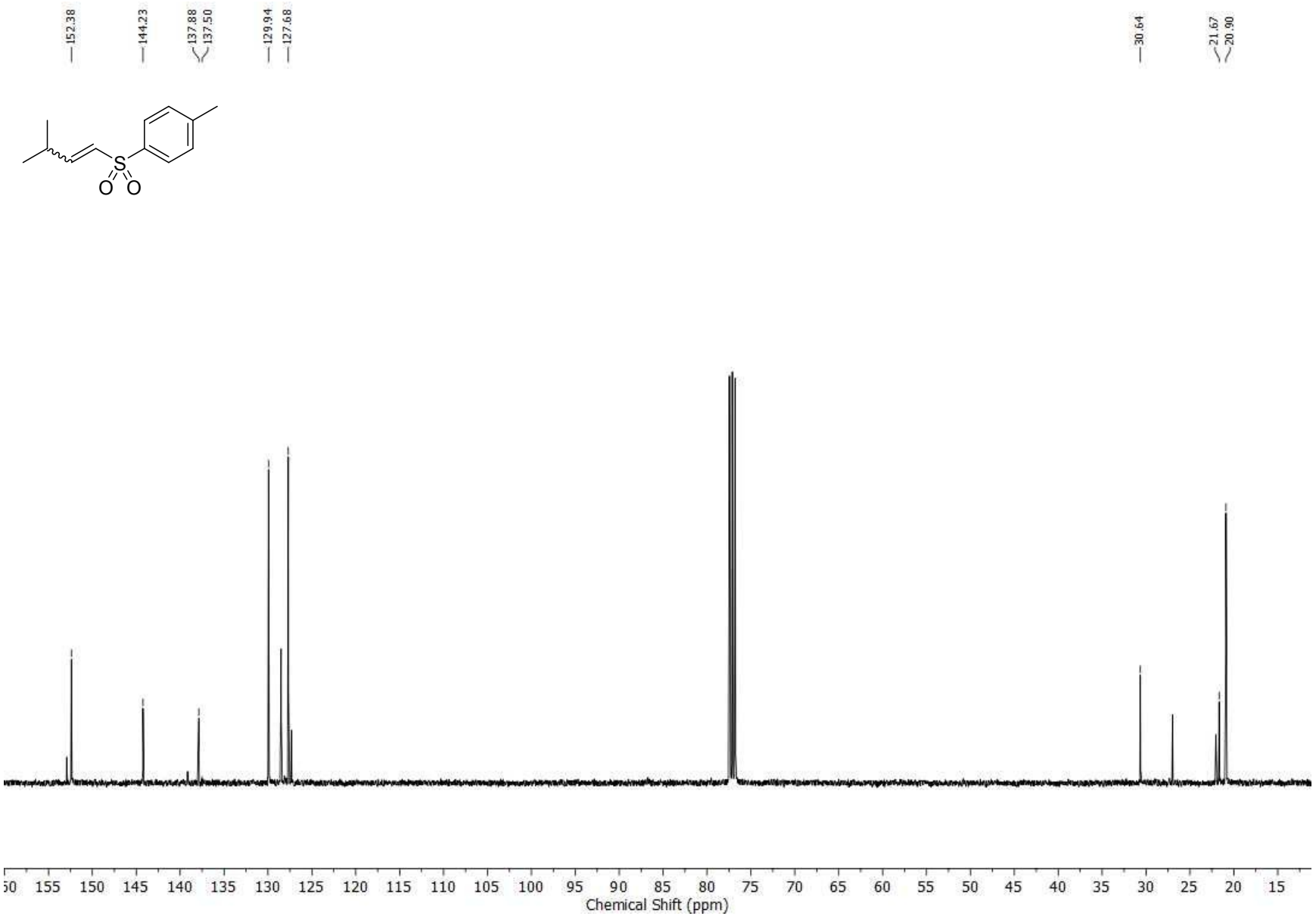



156.52
144.86
134.46
129.79
129.25
99.90
35.83
21.78
20.73
I
S
O
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
Chemical Shift (ppm)

1-Methyl-4-((3-methylbut-1-en-1-yl)sulfonyl)benzene ^{1}H and ^{13}C NMR






152.38
144.23
137.88
137.50
129.94
127.68
30.64
21.67
20.90
S
O
O
Chemical Shift (ppm)

References

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