

Electronic Supplementary Information for:

Synthesis and Properties of the Ivyanes: The Parent 1,1-Oligocyclopropanes

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General Methods

Reactions were conducted under a positive pressure of dry argon or dry nitrogen in oven-dried glassware. Dichloromethane was dried by distillation from calcium hydride. Magnesium sulfate was dried at 140 °C for 12 h prior to use. Commercially available reagents were used as purchased. Analytical thin layer chromatography was performed using plates precoated with silica gel 60 F₂₅₄ (0.2 mm). Flash chromatography employed 230–400 mesh silica gel.

¹H NMR spectra, unless otherwise stated, were recorded at 500 MHz and 300 MHz using a Varian INOVA 500 or a Varian Unity INOVA 300 spectrometer at 298 K. Data is expressed in parts per million (ppm) downfield shift from tetramethylsilane with residual solvent as an internal reference (δ 7.26 ppm for chloroform) and is reported as position (δ in ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (J in Hz) and integration (number of protons).

¹³C NMR spectra, unless otherwise stated, were recorded at 125 MHz or 75 MHz using a Varian INOVA 500 or a Varian Unity Inova 300 spectrometer at 298 K with complete proton decoupling. Data is expressed in parts per million (ppm) downfield shift relative to the internal reference (δ 77.2 ppm for the central peak of deuterated chloroform) and is reported as position (δ in ppm). Assignment of C, CH, CH₂ and CH₃ was aided with ¹³C-DEPT experiments.

IR spectra were collected using a Perkin–Elmer Spectrum One spectrophotometer as neat thin films on NaCl plates for oils and KBr pellets for solids.

Low resolution mass spectra (LRMS) were recorded on a Finnigan PolarisQ ion trap mass spectrometer using electron impact (EI⁺) ionization mode at 40 or 70 eV. High resolution mass spectra (HRMS) were recorded on a VG Autospec mass spectrometer operating at 70 eV.

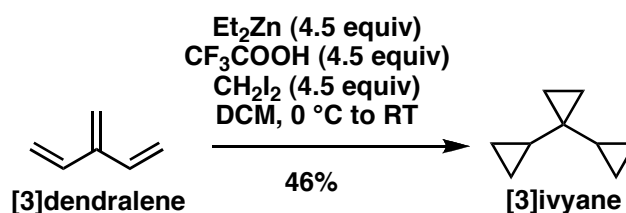
Experimental Procedures and Characterization Data of [n]Ivyanes

General Experimental Procedure for Cyclopropanation of [n]Dendralenes:

To a two-neck round bottom flask containing dry dichloromethane (1 volume equiv of diethyl zinc solution in hexane) at 0 °C under a nitrogen atmosphere was added a 1 M solution of diethyl zinc in hexane (1.2 mol equiv per alkene*). A solution of trifluoroacetic acid (1.2 mol equiv per alkene*) in dry dichloromethane (0.5 volume equiv of diethyl zinc solution in hexanes) was added dropwise and the resulting mixture was vigorously stirred for 20 min. A solution of diiodomethane (1.2 mol equiv per alkene*) in dry dichloromethane (0.5 volume equiv of diethyl zinc solution in hexanes) was added dropwise and the resulting mixture was vigorously stirred for another 20 min. The reaction mixture was kept at 0 °C during all this time. A solution of [n]dendralene (1.0 mol equiv) in dry dichloromethane (0.5 volume equiv of diethyl zinc solution in hexanes) was then added and the reaction mixture was slowly warmed to RT and stirred for 2–18 h. The progress of the reaction was followed by TLC. Once complete, the reaction mixture was quenched with a solution of sat. aq. NH₄Cl (2 volume equiv of diethyl zinc solution in hexanes) and the organic and aqueous layers were separated. The aqueous layer was extracted with more dichloromethane (× 2) and the combined organic extracts were washed with water, sat. aq. NaCl then dried over anhydrous MgSO₄ and concentrated in vacuo (20 mbar, 0 °C) to give an oil. The oil was purified by flash chromatography eluting with pentane to give the [n]ivyane.

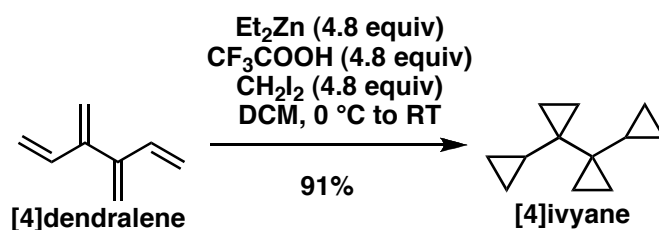
(*1.5 mol equiv was employed in the synthesis of [3]ivyane)

Cyclopropanation of [3]dendralene



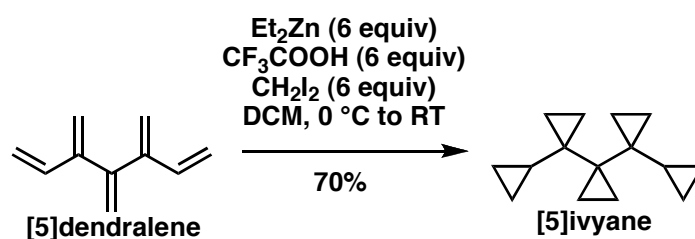
[3]Ivyane has been synthesized previously using different routes.^{S1} [3]Dendralene^{S2} (80.1 mg, 1.00 mmol), diethyl zinc (1 M solution in hexane, 4.5 mL, 4.5 mol equiv), diiodomethane (363 μL, 4.5 mol equiv) and trifluoroacetic acid (335 μL, 4.5 mol equiv) were employed according to the general procedure with a 2 h reaction time. **[3]Ivyane** (56.2 mg, 46%) was isolated as a colorless oil: *R_f* = 0.66 (pentane); ¹H NMR (500 MHz, CDCl₃) δ 1.07 (2H, tt, *J* = 8.5, 3.5 Hz), 0.30 (4H, m), 0.03 (8H, m) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 21.0 (2 × C), 16.5 (2 × CH), 6.9 (2 × CH₂), 1.8 (2 × CH₂) ppm; IR (thin film) $\tilde{\nu}$ = 3078, 3003, 2923, 1466, 1013 cm⁻¹ MS (40 eV, EI) *m/z*: 122 ([*M*]⁺, 5%), 94 ([*M*]⁺ - C₂H₄, 85), 91 (100), 79 (60). The ¹H NMR data reported here match those previously reported.^{S1}

Cyclopropanation of [4]dendralene



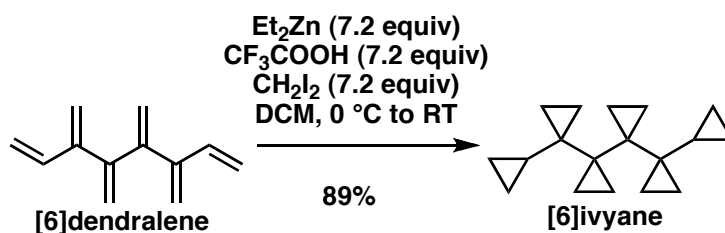
[4]Dendralene^{S3} (106 mg, 1.00 mmol), diethyl zinc (1 M solution in hexane, 4.8 mL, 4.8 mol equiv), diiodomethane (387 μL , 4.8 mol equiv) and trifluoroacetic acid (357 μL , 4.8 mol equiv) were employed according to the general procedure with a 2 h reaction time. **[4]Ivyane** (148 mg, 91%) was isolated as a colorless oil: $R_f = 0.64$ (pentane); ^1H NMR (500 MHz, CDCl_3) δ 1.32 (2H, tt, $J = 8.0, 5.5$ Hz), 0.32 (4H, m), 0.10 (4H, m), 0.04 (4H, m), -0.02 (4H, m) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 25.2 ($2 \times \text{C}$), 15.1 ($2 \times \text{CH}$), 7.0 ($4 \times \text{CH}_2$), 2.2 ($4 \times \text{CH}_2$) ppm; IR (thin film) $\tilde{\nu} = 3077, 3005, 1465, 1014$ cm^{-1} ; MS (40 eV, EI) m/z : 161 ($[\text{M}]^+ - \text{H}$, 1%), 134 ($[\text{M}]^+ - \text{C}_2\text{H}_4$, 97), 119 (87), 91 (100), 79 (50); HRMS: calcd for $\text{C}_{12}\text{H}_{18}$ $[\text{M}]^+$: 162.1409; found 162.1415.

Cyclopropanation of [5]dendralene



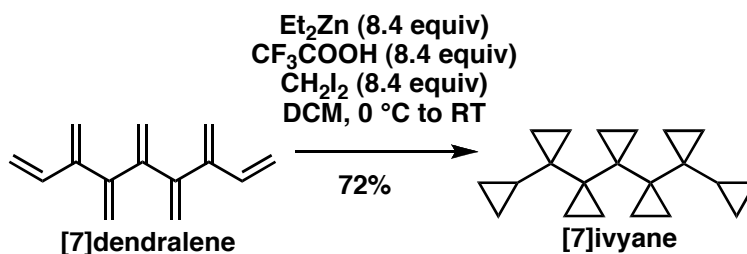
[5]Dendralene^{S3} (132 mg, 1 mmol), diethyl zinc (1 M solution in hexane, 6.0 mL, 6 mol equiv), diiodomethane (483 μL , 6 mol equiv) and trifluoroacetic acid (445 μL , 6 mol equiv) were employed according to the general procedure with a 2 h reaction time. **[5]Ivyane** (142 mg, 70%) was isolated as a colorless oil: $R_f = 0.63$ (pentane); ^1H NMR (500 MHz, CDCl_3) δ 1.47 (2H, tt, $J = 8.5, 5.5$ Hz), 0.30 (4H, m), 0.18 (4H, m), 0.07 (4H, bs), 0.06 (4H, m), -0.02 (4H, m) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 27.5 (C), 23.3 ($2 \times \text{C}$), 15.3 ($2 \times \text{CH}$), 7.6 ($2 \times \text{CH}_2$), 7.3 ($4 \times \text{CH}_2$), 2.3 ($4 \times \text{CH}_2$) ppm; IR (thin film) $\tilde{\nu} = 3078, 3007, 1467, 1016$ cm^{-1} ; MS (40 eV, EI) m/z : 202 ($[\text{M}]^+$, 2%), 174 ($[\text{M}]^+ - \text{C}_2\text{H}_4$, 75), 159 (100), 131 (73), 91 (98); HRMS: calcd for $\text{C}_{15}\text{H}_{22}$ $[\text{M}]^+$: 202.1722; found 202.1715.

Cyclopropanation of [6]dendralene



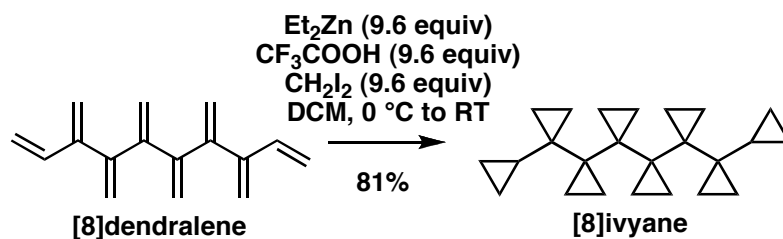
[6]Dendralene^{S3} (1.04 g, 6.6 mmol), diethyl zinc (1 M solution in hexane, 47.6 mL, 7.2 mol equiv), diiodomethane (3.83 mL, 7.2 mol equiv) and trifluoroacetic acid (3.67 mL, 7.2 mol equiv) were employed according to the general procedure with a reaction time of 5 h. **[6]Ivyane** (1.42 g, 89%) was isolated as a colorless oil: $R_f = 0.60$ (pentane); ¹H NMR (500 MHz, CDCl₃) δ 1.56 (2H, tt, $J = 8.0, 6.0$ Hz), 0.33 (4H, m), 0.24 (8H, m), 0.19 (4H, bs), 0.02 (4H, bs), -0.09 (4H, m) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 25.4 (2 × C), 23.5 (2 × C), 15.3 (2 × CH), 8.9 (4 × CH₂), 7.4 (4 × CH₂), 2.1 (4 × CH₂) ppm; IR (thin film) $\tilde{\nu} = 3079, 3008, 1469, 1018$ cm⁻¹; MS (40 eV, EI) m/z : 242 ([M]⁺, 1%), 214 ([M]⁺-C₂H₄, 72), 185 (70), 91 (100), 79 (92); HRMS: calcd for C₁₈H₂₆ [M]⁺: 242.2035; found 242.2021.

Cyclopropanation of [7]dendralene



[7]Dendralene^{S3} (92.1 mg, 0.5 mmol), diethyl zinc (1 M solution in hexane, 4.2 mL, 8.4 mol equiv), diiodomethane (338 μ L, 8.4 mol equiv) and trifluoroacetic acid (311 μ L, 8.4 mol equiv) were employed according to the general procedure with a reaction time of 18 h. **[7]Ivyane** (102 mg, 72%) was isolated as a colorless oil: $R_f = 0.58$ (pentane). Recrystallization from *n*-pentane gave the title compound as colorless needles: mp 48–50 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.41 (2H, tt, $J = 8.5, 5.5$ Hz), 0.57 (4H, s), 0.51 (4H, m), 0.20–0.18 (12H, m), -0.04 (4H, m), -0.08 (4H, m) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 25.8 (2 × C), 24.0 (4 × C), 16.4 (2 × CH), 11.4 (2 × CH₂), 10.4 (4 × CH₂), 8.1 (2 × CH₂), 2.7 (4 × CH₂) ppm; IR (thin film) $\tilde{\nu} = 3079, 3010, 1019$ cm⁻¹; MS (40 eV, EI) m/z : 282 ([M]⁺, 1%), 254 (M -C₂H₄, 28), 225 (50), 91 (100); HRMS: calcd for C₁₉H₂₆ [M -C₂H₄]⁺: 254.2035; found 254.2034.

Cyclopropanation of [8]dendralene



[8]Dendralene^{S3} (53 mg, 0.25 mmol), diethyl zinc (1 M solution in hexanes, 2.4 mL, 9.6 mol equiv), diiodomethane (194 μ L, 9.6 mol equiv) and trifluoroacetic acid (179 μ L, 9.6 mol equiv) were employed according to the general procedure with a reaction time of 18 h. **[8]Ivyane** (65.3 mg, 81%) was isolated as a colorless oil: $R_f = 0.57$ (pentane). Recrystallization from *n*-pentane gave the title compound as colorless prisms: mp 93–95 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.46 (2H, tt, $J = 8.5, 5.5$ Hz), 0.75 (4H, bs), 0.54 (4H, bs), 0.39 (4H, bt, $J = 5.5$ Hz), 0.24 (4H, m), 0.16 (4H, bs), 0.11 (4H, bt, $J = 5.0$ Hz), -0.04 (4H, bs), -0.10 (4H, dd, $J = 10.0, 5.5$ Hz) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 26.0 (2 \times C), 24.3 (2 \times C), 23.7 (2 \times C), 15.8 (2 \times CH), 11.9 (4 \times CH₂), 10.2 (4 \times CH₂), 8.8 (4 \times CH₂), 2.6 (4 \times CH₂) ppm; IR (thin film) $\tilde{\nu} = 3079, 3011, 1020$ cm⁻¹; MS (30 eV, EI) m/z : 322 ($[M]^+$, 1%), 294 ($M - C_2H_4$, 12), 159 (98), 105 (100); HRMS: calcd for C₂₄H₃₄ $[M]^+$: 322.2661; found 322.2664.

X-ray Crystal Structures of [7]- and [8]Ivyane

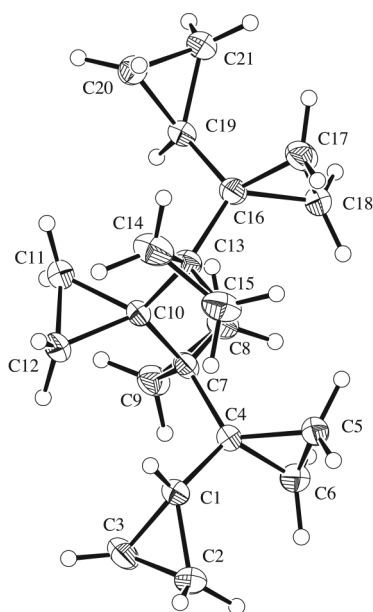


Figure S1: Anisotropic displacement ellipsoid plot of [7]ivyane (CCDC 766962) with labelling of selected atoms. Ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii.

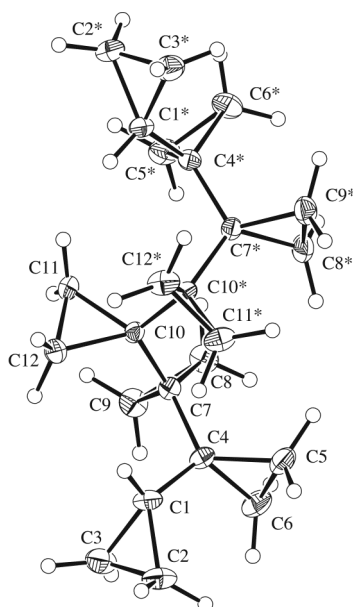


Figure S2: Anisotropic displacement ellipsoid plot of [8]ivyane (CCDC 766963) with labelling of selected atoms. Asterisks indicate atoms generated by the crystallographic two-fold symmetry operation. Ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii.

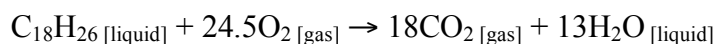
Calorimetric Studies on [6]Ivyane

[6]Ivyane was selected as a representative of the ivyane family for Combustion Analysis and Differential Scanning Calorimetric Analysis.

Combustion Analysis

These experiments were carried out using a Gallenkamp Bomb Calorimeter at the University of New South Wales campus at Australian Defence Force Academy, Canberra. The heat of combustion of several organic compounds – including benzoic acid, heptane and toluene – were analyzed using this equipment before the [6]ivyane analyses. The experimental results fit well with literature values for heat of combustion of these compounds, with less than 4% discrepancy. Based on these experiments, the heat capacity of the Bomb Calorimeter was determined as $10816 \pm 100 \text{ J K}^{-1}$. The empirical value for heat of combustion of igniting material (cotton thread and fuse wire) was 70 J.

[6]Ivyane was made according to the experimental procedure described above. The sample was degassed before use. The experiments were carried out in triplicate at 25 atm of oxygen and 25 °C. A known mass (ca. 0.5 g) of [6]ivyane was used each time.



$$\text{Heat of Combustion} = \frac{(\text{10816} \times \text{Temperature increase} - 70)}{\text{Mass of [6]ivyane}} \quad (\text{J g}^{-1})$$

Analysis No	Mass of [6]Ivyane (g)	Temperature Increase (°C)	Calculated Heat of Combustion (J g ⁻¹)	Average Heat of Combustion (J g ⁻¹)
1	0.4903	2.3	50593.5	
2	0.5096	2.4	50799.7	50760.9
3	0.5087	2.4	50889.6	

Note: the Bomb Calorimeter was saturated with water vapor, so any water formed in the combustion will be in the liquid form. Total systematic error for this analysis ~5%.

Hence, the heat of combustion for [6]ivyane = $50.8 \pm 2.5 \text{ (MJ kg}^{-1}\text{)}$ or $12.3 \pm 0.6 \text{ (MJ mol}^{-1}\text{)}$

Differential Scanning Calorimetric (DSC) Analysis

DSC analysis was carried out using a DSC 2920 Differential Scanning Calorimeter (TA Instrument). [6]Ivyane samples of 2–4 mg were encapsulated in aluminium sample pans. The samples were slowly heated from 30 °C to 400 °C while monitoring the heat flow through the sample pan. Each heating profile was tested in duplicate to ensure the reproducibility of the experiment.

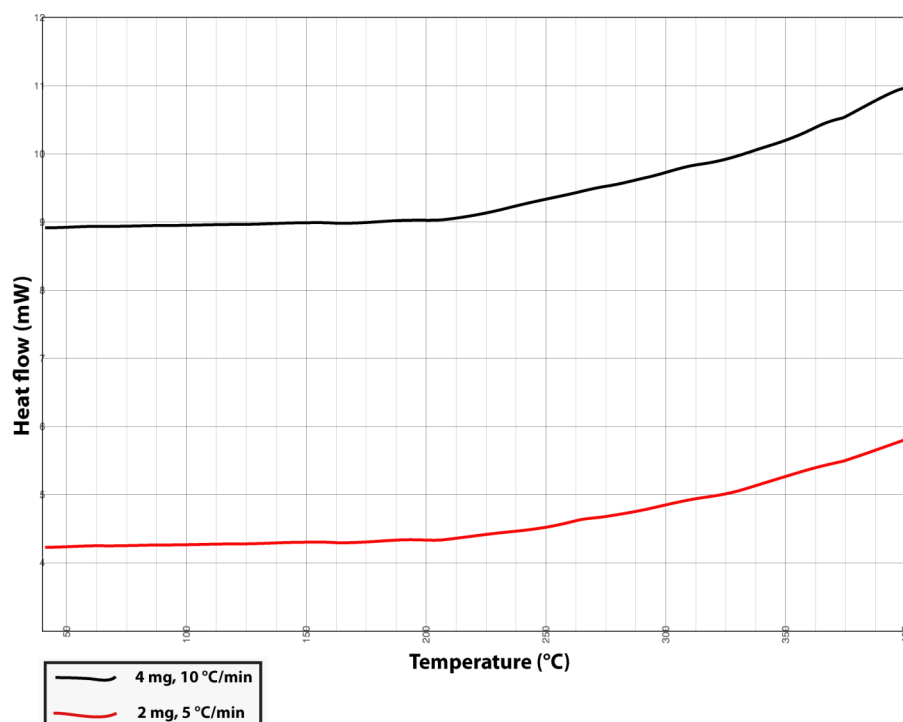



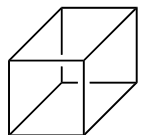
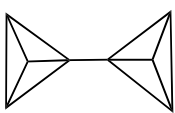
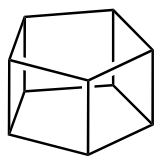
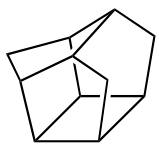
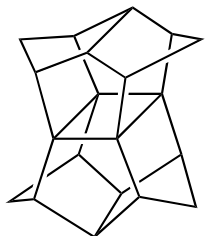
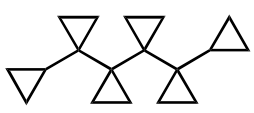
Figure S3: Differential Scanning Calorimetric Studies of [6]Ivyane

The DSC analyses at various heating rates revealed that [6]ivyane is kinetically stable up to ~200 °C. After this temperature, there was evidence of a phase change, which is ascribed to boiling and/or decomposition of the material.

Heat of Combustion for Related Strained Hydrocarbons

Heats of combustion (calculated or experimental value, as specified) for several related strained hydrocarbons are quoted here for comparison purposes:

Table S1: Heats of combustion for selected strained hydrocarbons

Name	Structure	Heat of combustion (MJ mol ⁻¹) at 298 K	Heat of combustion (MJ kg ⁻¹) at 298 K
Cyclopropane		2.1 ^{S4} (experimental)	49.7 ^{S4} (experimental)
Cubane		4.7 ^{S5} (experimental)	46.5 ^{S5} (experimental)
Ditetrahedryl		4.9 ^{S6} (estimated)	47.5 ^{S6} (estimated)
Pentaprismane		5.6 ^{S6} (estimated)	43.0 ^{S6} (estimated)
Pentacyclo[5.4.0.0 ^{2,6} .0 ^{3,10} .0 ^{5,9}]-undecane		6.1 ^{S6} (estimated)	41.7 ^{S6} (estimated)
Pagodane		10.8 ^{S7} (experimental)	41.6 ^{S7} (experimental)
[6]Ivyane		12.3 (experimental)	50.8 (experimental)

The molar heat of combustion of [6]ivyane is roughly 6 times higher than that of cyclopropane. This is an expected result, since [6]ivyane is essentially six cyclopropanes connected in a linear chain. Furthermore, the heats of combustion per kg for these two compounds are almost identical, indicating that the strain energies in ‘stand-alone’ and ‘ivyane connected’ cyclopropane rings are comparable.

Estimation of ΔG^\ddagger for the Interconversion of [6]Ivyane Helices by VT-NMR

The ^1H NMR spectra of [6]ivyane at various temperatures, though more complicated than the ^{13}C NMR spectra (Figure 3, main manuscript), display a similar behavior with more signals appearing at low temperature (**Figure S4**).

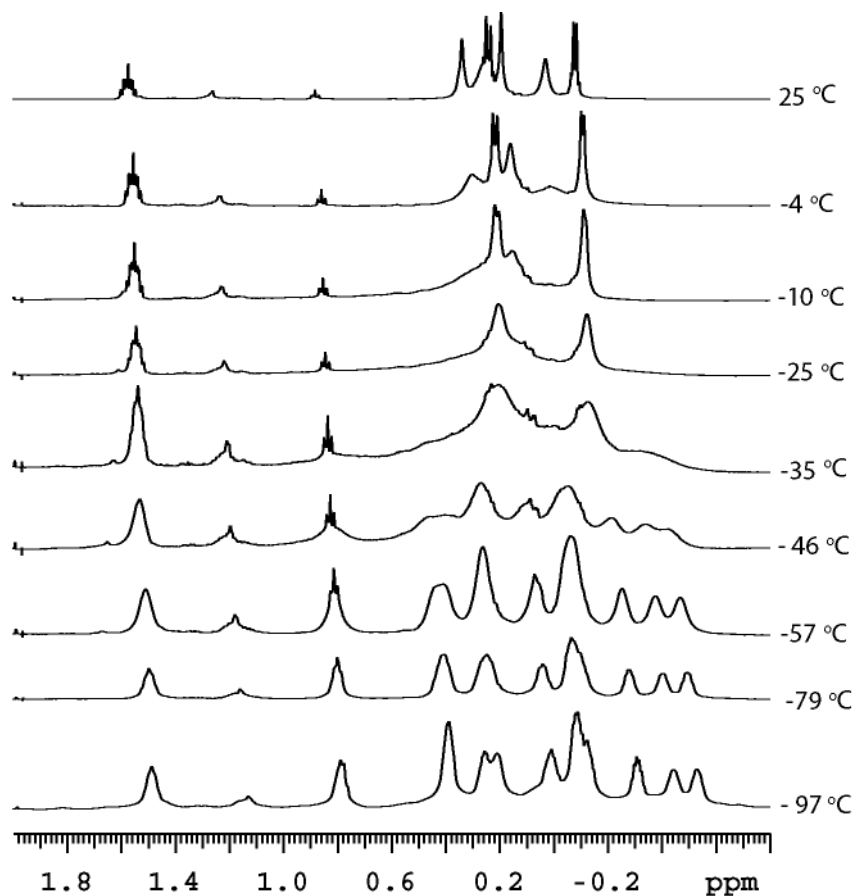


Figure S4: ^1H VT-NMR spectra of [6]ivyane (500 MHz, CD_2Cl_2)

From the ^1H VT-NMR spectra it is evident that the coalescence point occurs at ca. -35 ± 4 °C. Thus, the free energy of activation for the interconversion of [6]iviyane helical enantiomeric conformers *in solution* can be estimated as follows:

Assuming [6]iviyane rotation follows a first order rate law, then equation (1)^{S8} affords the rate constant k_r at the coalescence point T_c :

$$\begin{aligned}k_r &= \pi(\nu_B - \nu_A)/\sqrt{2} & (1) \\ &= \pi\Delta\nu/\sqrt{2} \\ &= 2.22\Delta\nu\end{aligned}$$

where $\Delta\nu$ is the full width at half-maximum of the signal at coalescence point T_c and it corresponds to the difference in chemical shift observed during slow exchange.

In this case $\Delta\nu$ is 0.083 ppm i.e. 41.5 Hz (at 500 MHz)

Therefore

$$\begin{aligned}k_r &= 2.22 \times 41.5 \text{ Hz} \\ &= 92.1 \text{ Hz}\end{aligned}$$

The exchange frequency k_r decreases exponentially with the free energy of activation ΔG^\ddagger as shown in equation 2:

$$k_r = [kT_c/h] e^{-G^\ddagger/RT_c} \quad (2)$$

where R = gas constant, k = Boltzmann constant, and h is Planck's constant.

Hence the free energy of activation for first order exchange process:

$$\Delta G^\ddagger = 19.1T_c[10.32 + \log(T_c/k_r)] \times 10^{-3} \text{ kJ mol}^{-1} \quad (3)$$

We know $k_r = 92.1$ Hz, $T_c = 238$ K (-35 °C), hence,

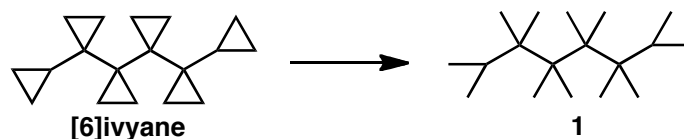
$$\begin{aligned}\Delta G^\ddagger_{234} &= 4545.8 [10.32 + \log 2.584] \times 10^{-3} \text{ kJ mol}^{-1} \\ &= 48.8 \text{ kJ mol}^{-1}\end{aligned}$$

Total systematic error $\sim 15\%$. Hence, the free energy of activation ΔG^\ddagger for the interconversion between the two helical enantiomeric conformers was obtained as 48 ± 7 kJ mol $^{-1}$.

Chemistry of Ivyanes

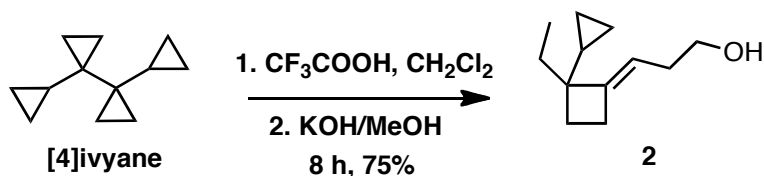
Preliminary investigations into ivyane chemistry were carried out on [6] and [4]ivyanes.

Ring-Opening Hydrogenolysis of [6]Ivyane



A suspension of $\text{PtO}_2 \cdot \text{H}_2\text{O}$ (49 mg, 0.20 mmol, 40 mol%) in acetic acid (5 mL) was exposed to an atmosphere of hydrogen (1.6 atm) for 30 min at 40 °C. A solution of [6]ivyane (141 mg, 0.50 mmol) in hexane (1 mL) was added in one portion and the resulting mixture was stirred at 40 °C under H_2 (1.6 atm) for 12 h, then cooled to RT and diluted with hexane (30 mL). The resulting mixture was washed successively with water (2 × 30 mL), sat. aq. NaHCO_3 (2 × 20 mL) and brine (30 mL). The organic extract was dried over anhydrous MgSO_4 then concentrated at reduced pressure (20 mbar, 0 °C) to give an oil. This oil was purified by column chromatography (SiO_2 , pentane) to give pure 2,3,3,4,4,5,5,6,6,7-decamethyloctane **1** (112 mg, 88%) as a colorless oil: $R_f = 0.83$ (pentane); ^1H NMR (300 MHz, CDCl_3 , recorded at 50 °C since the signals at 25 °C were broad, presumably due to slow conformational interchange on the NMR timescale) δ 2.67 (2H, sept, $J = 6.9$ Hz), 0.74 (12H, d, $J = 6.9$ Hz), 0.45–0.04 (24H, m) ppm; ^{13}C NMR (75 MHz, CDCl_3 , 25 °C) δ 27.0, 26.5, 23.8, 20.0 (br), 8.1, 1.9 (br) ppm; IR (thin film) $\tilde{\nu} = 3081, 3010, 2961, 2905, 2870, 1474, 1109, 1016 \text{ cm}^{-1}$; MS (40 eV, EI) m/z : 254 ($[M]^+$, 1%), 124 (100), 92 (65). Anal. Calcd. for $\text{C}_{18}\text{H}_{38}$: C, 84.95; H, 15.05; found C, 84.69; H, 15.31.

Acid Induced Rearrangement of [4]Ivyane



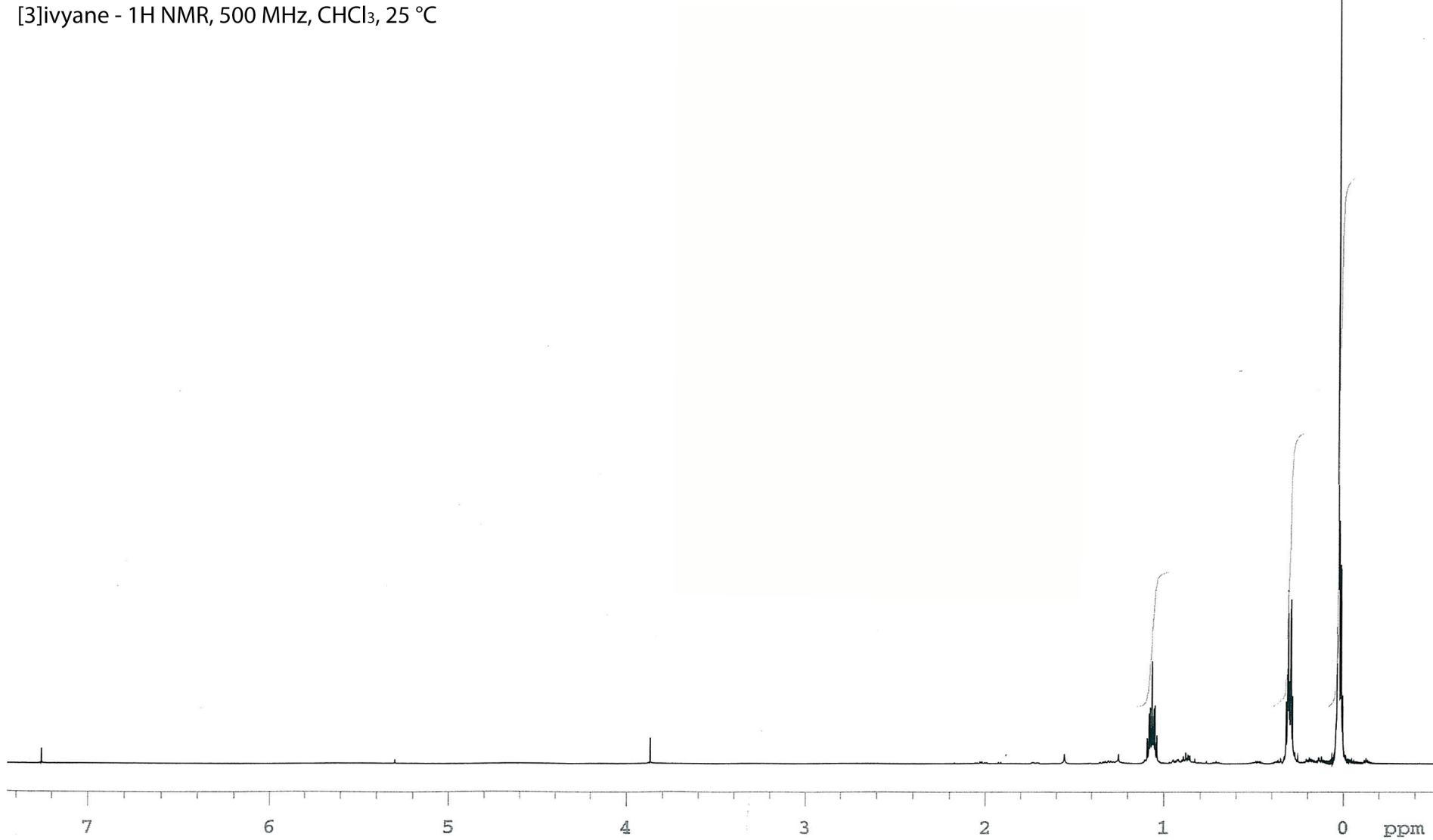
To a solution of [4]ivyane (100 mg, 0.62 mmol) in dichloromethane (5.0 mL) under nitrogen was added trifluoroacetic acid (300 μL , 3.89 mmol). After stirring at RT for 6 h, a solution of sat. aq. NaHCO_3 (20 mL) was added with vigorous stirring. The organic layer was collected, washed with brine (20 mL) and dried over anhydrous MgSO_4 before being concentrated in vacuo to give a yellow oil (145 mg). This oil was then added to a solution of KOH (100 mg, 1.78 mmol) in

methanol (3.0 mL) and the resulting mixture was stirred for 2 h at RT. The reaction mixture was partitioned between dichloromethane (10 mL) and sat. aq. NH₄Cl (20 mL), the organic phase was dried over anhydrous MgSO₄ and the solvent was removed in vacuo to give an oil. The oil was purified by column chromatography (SiO₂, 20:80 ethyl acetate/hexanes) to afford alcohol **2** (77 mg, 0.43 mmol, 75%) as a colorless oil: *R_f* = 0.26 (20:80 ethyl acetate/hexanes); ¹H NMR (500 MHz, CDCl₃) δ 4.99 (1H, tt, *J* = 2.0, 8.0 Hz), 3.57 (2H, t, *J* = 6.5 Hz), 2.41 (2H, m), 2.14 (2H, q, *J* = 7.5 Hz), 1.69 (1H, m), 1.60 (1H, m), 1.55 (2H, q, *J* = 7.5 Hz), 0.90 (3H, t, *J* = 7.0 Hz), 0.83 (1H, m), 0.34 (2H, m), 0.19 (2H, m) ppm; ¹³C NMR (75 MHz, CDCl₃) δ 149.1 (C), 115.0 (CH), 62.4 (CH₂), 50.9 (C), 32.7 (CH₂), 31.4 (CH₂), 25.5 (CH₂), 24.7 (CH₂), 16.9 (CH), 8.9 (CH₃), 1.1 (CH₂), 0.7 (CH₂) ppm; IR (thin film) $\tilde{\nu}$ = 3327, 3077, 2965, 1049, 1016 cm⁻¹; MS (40 eV, EI) *m/z*: 180 ([*M*]⁺, 4%), 152 (*M*-C₂H₄, 47), 121 (100), 93 (78); HRMS: calcd for C₁₂H₂₀O [*M*]⁺: 180.1514; found 180.1510. All attempts to form crystalline derivatives of alcohol **2** suitable for single crystal X-ray analyses were unsuccessful. The structure of **2** is assigned on the basis of COSY, HMBC and HSQC experiments.

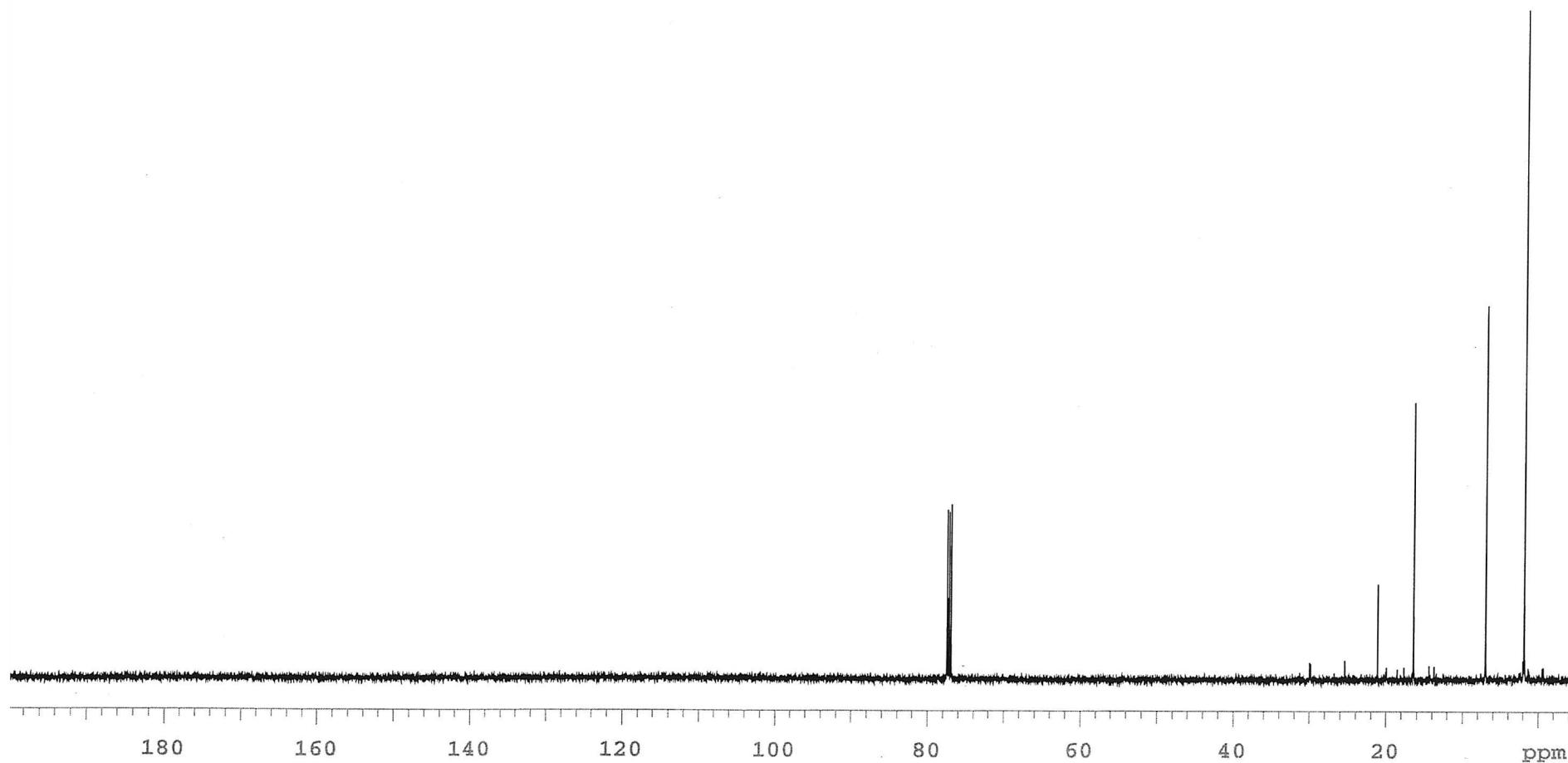
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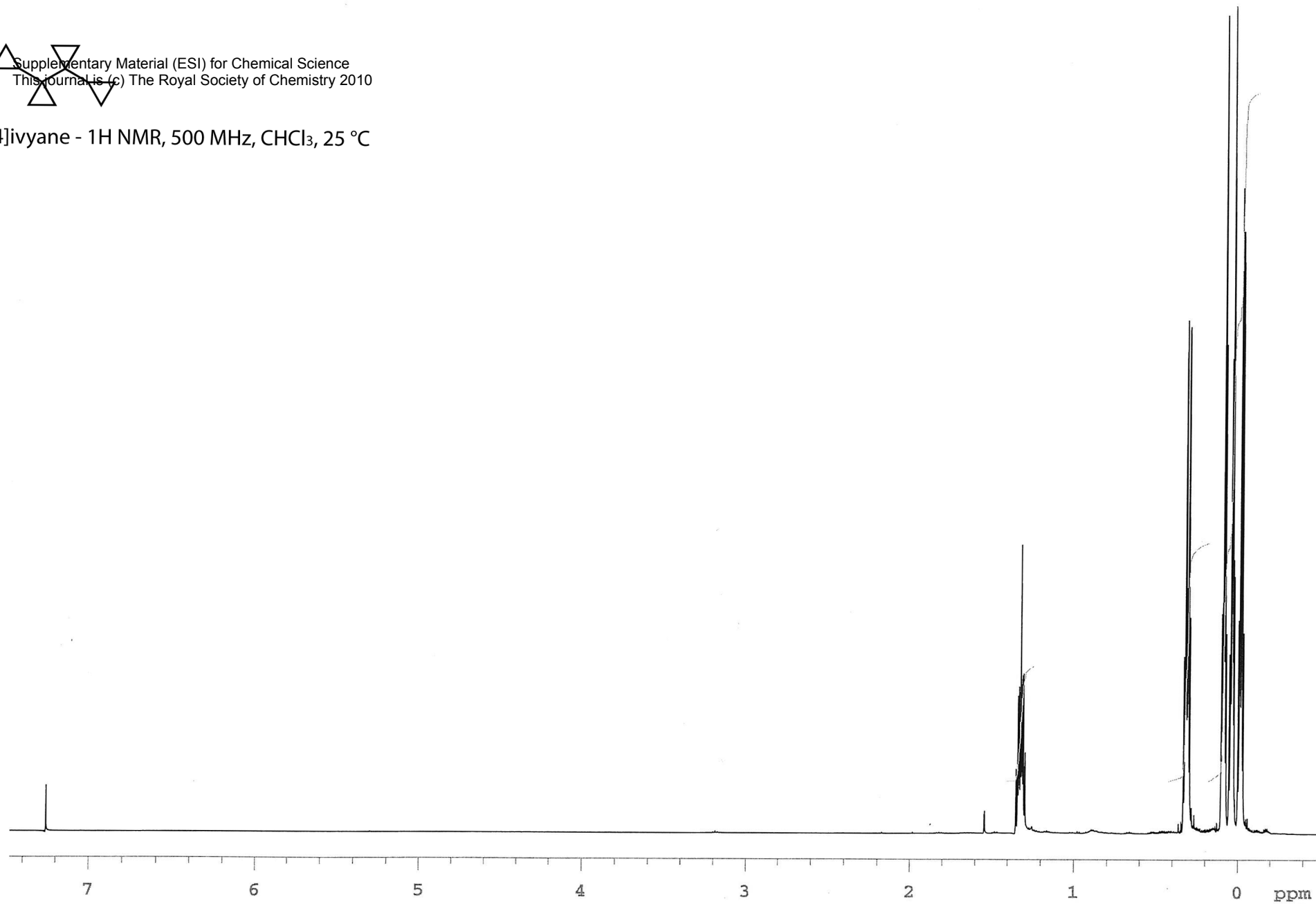
[3]ivyane - ¹H NMR, 500 MHz, CHCl₃, 25 °C



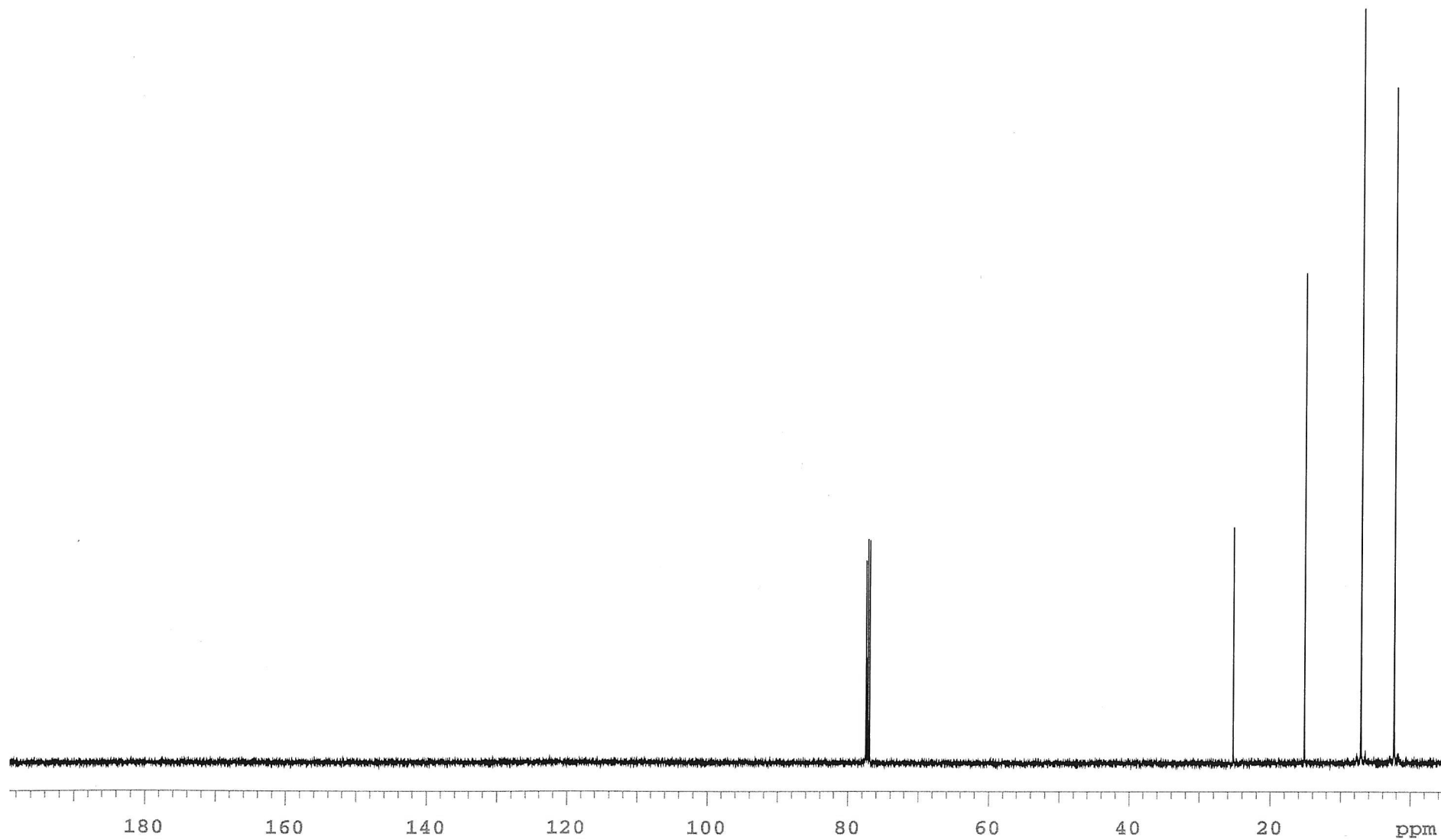
[3]ivyane - ^{13}C NMR, 125 MHz, CHCl_3 , 25 °C



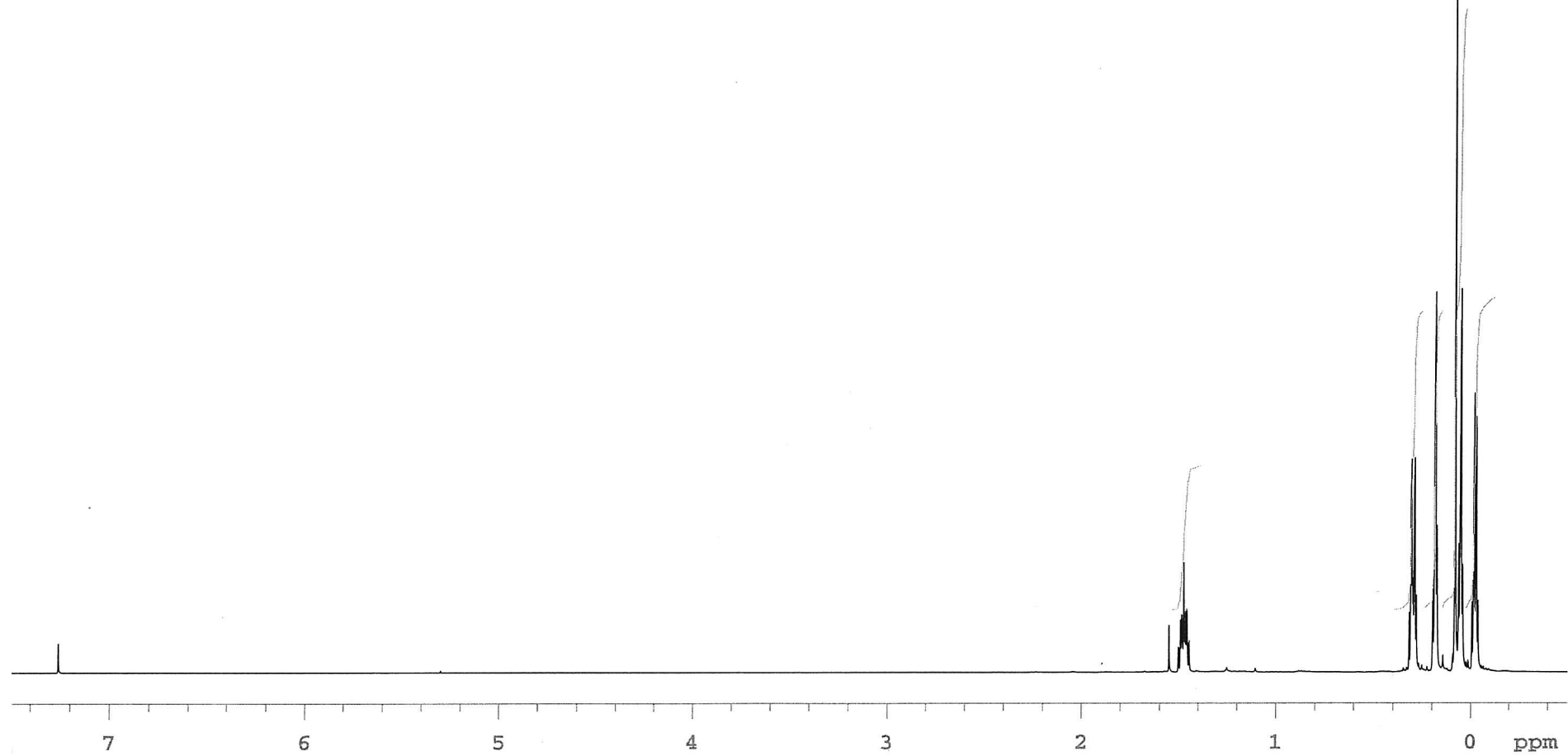
[4]ivane - ^1H NMR, 500 MHz, CHCl_3 , 25 °C



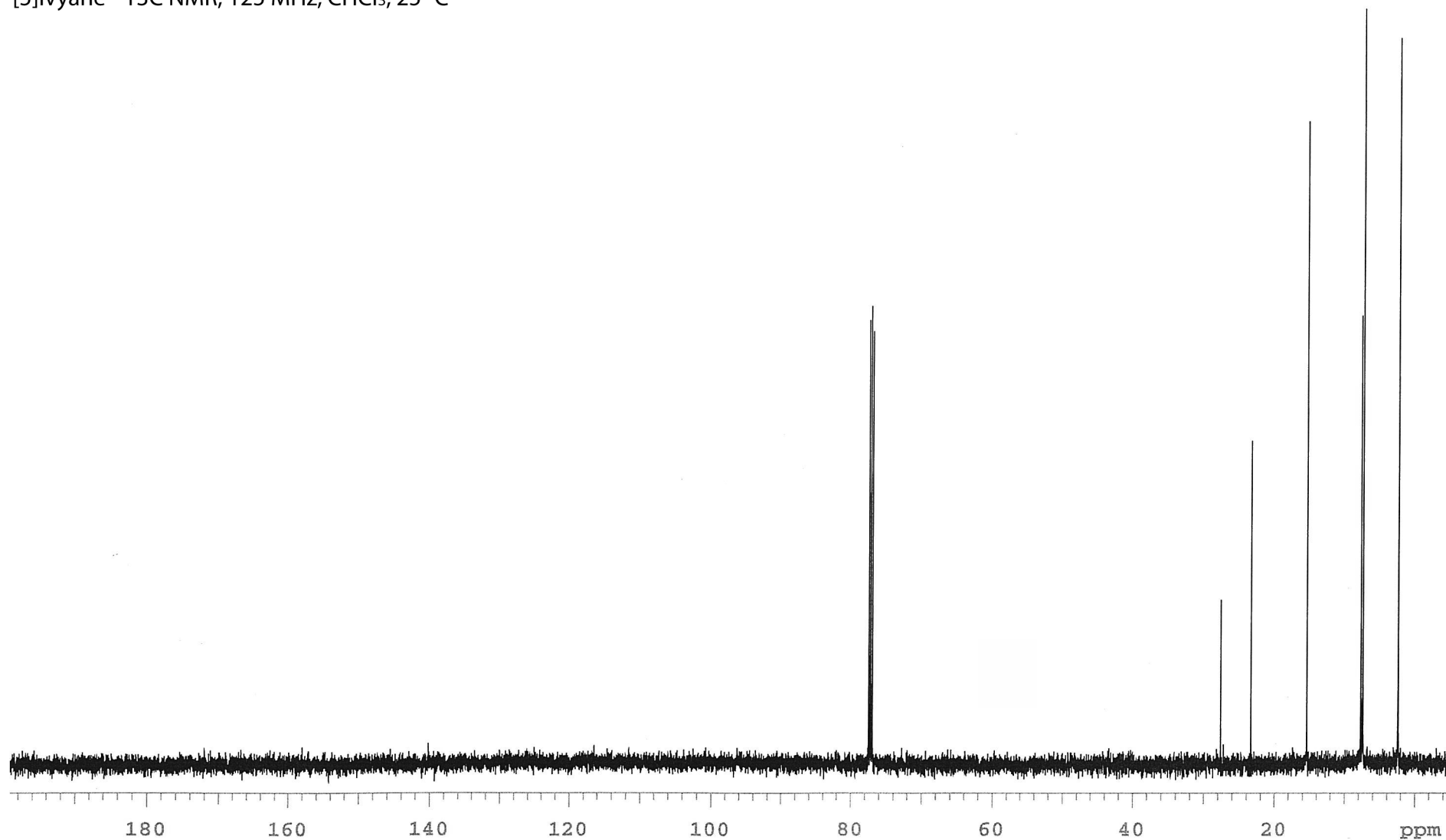
[4]ivylene - ^{13}C NMR, 125 MHz, CHCl_3 , 25 °C



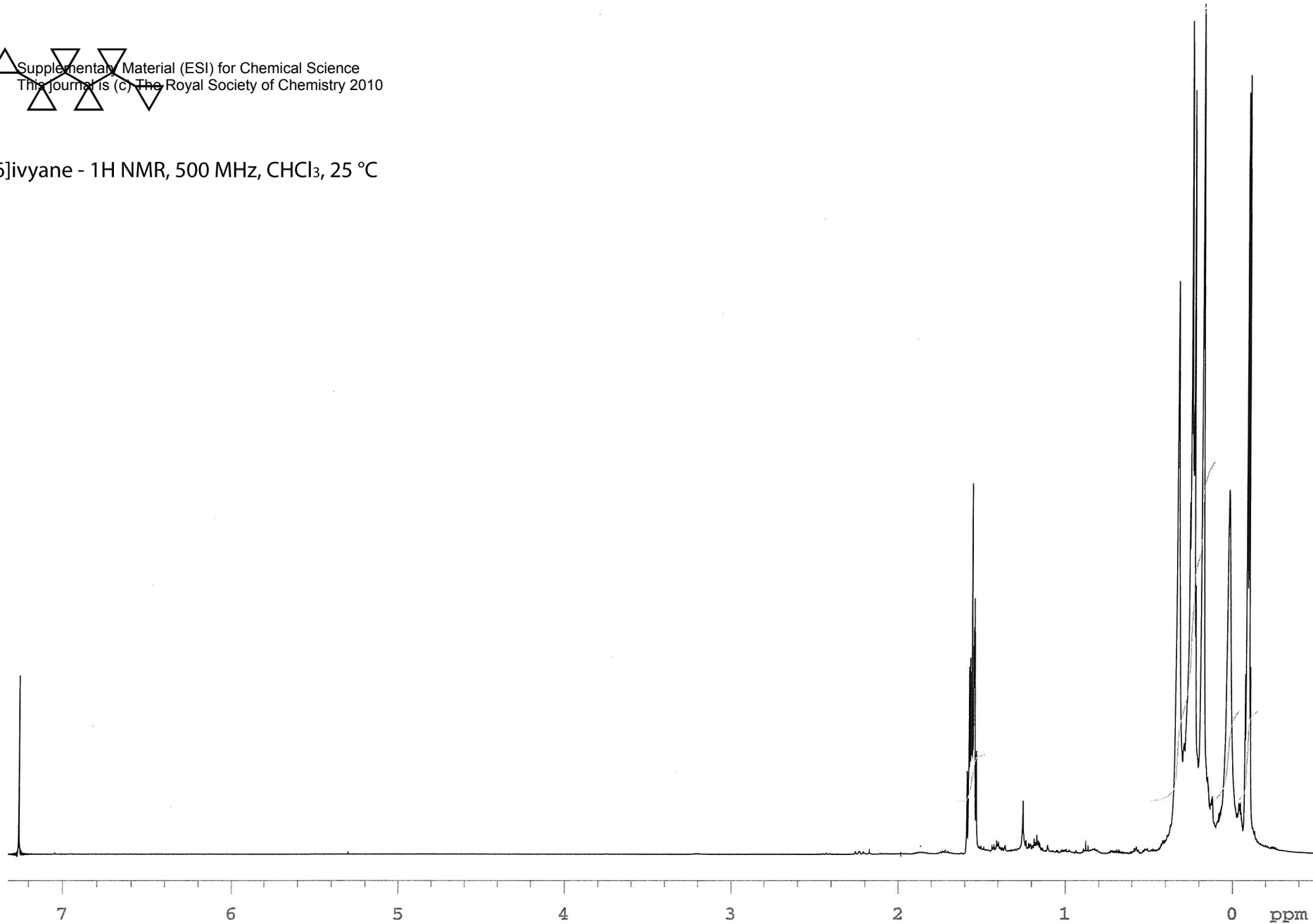
[5]ivyane - ^1H NMR, 500 MHz, CHCl_3 , 25 °C



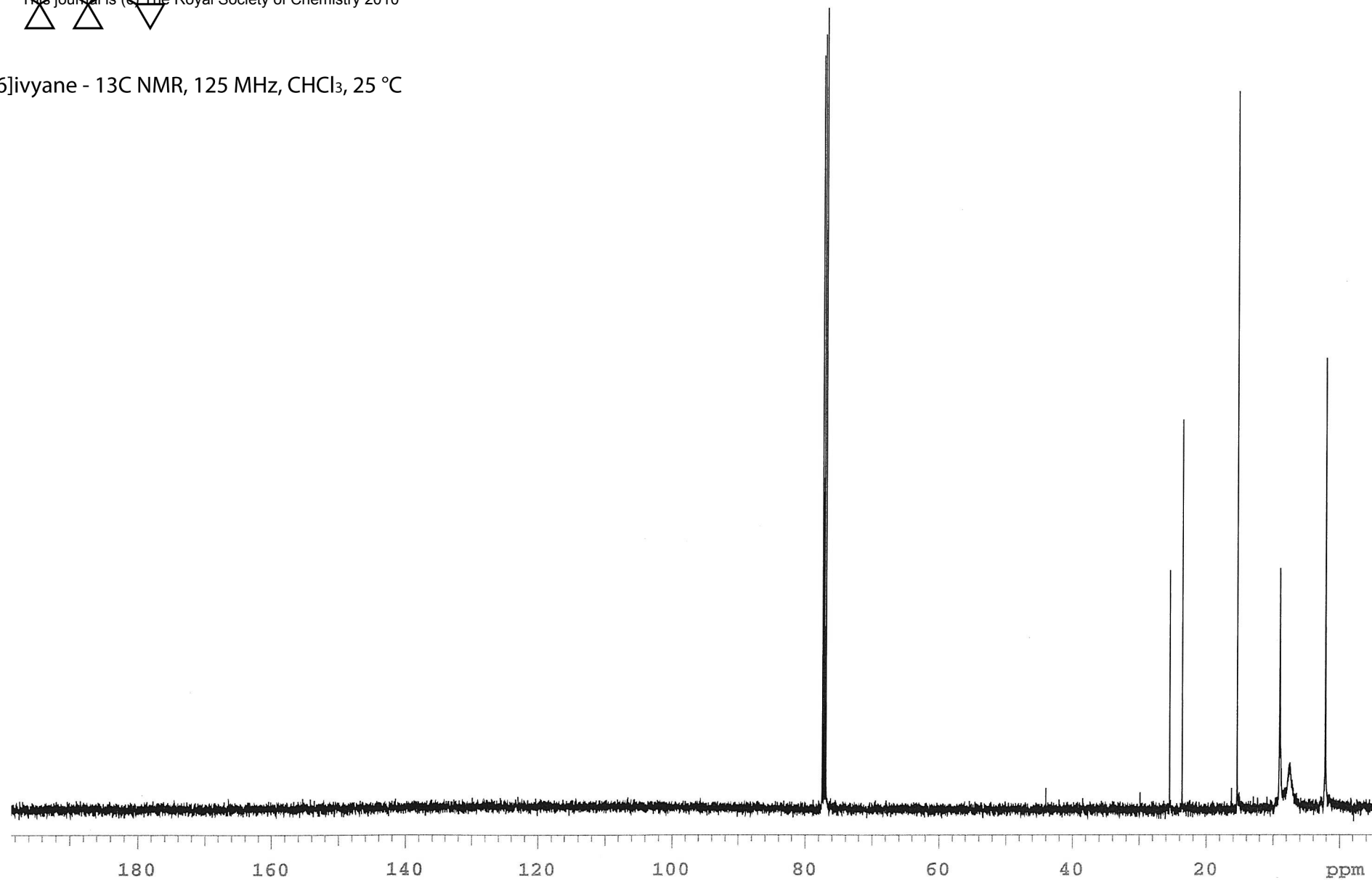
[5]ivyane - ^{13}C NMR, 125 MHz, CHCl_3 , 25 °C



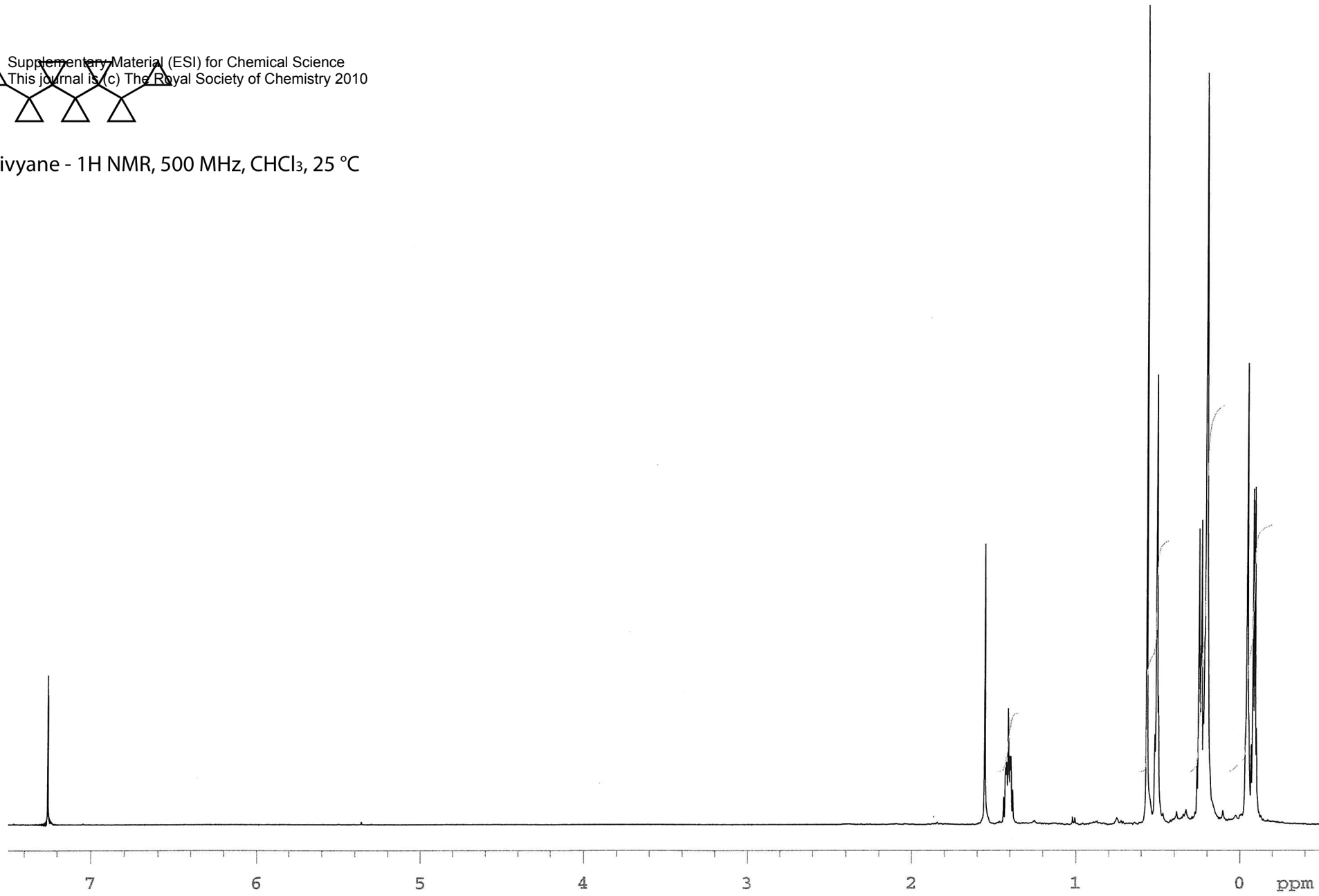
[6]ivylene - ^1H NMR, 500 MHz, CHCl_3 , 25 °C

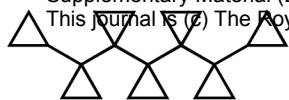


[6]ivyane - ^{13}C NMR, 125 MHz, CHCl_3 , 25 °C

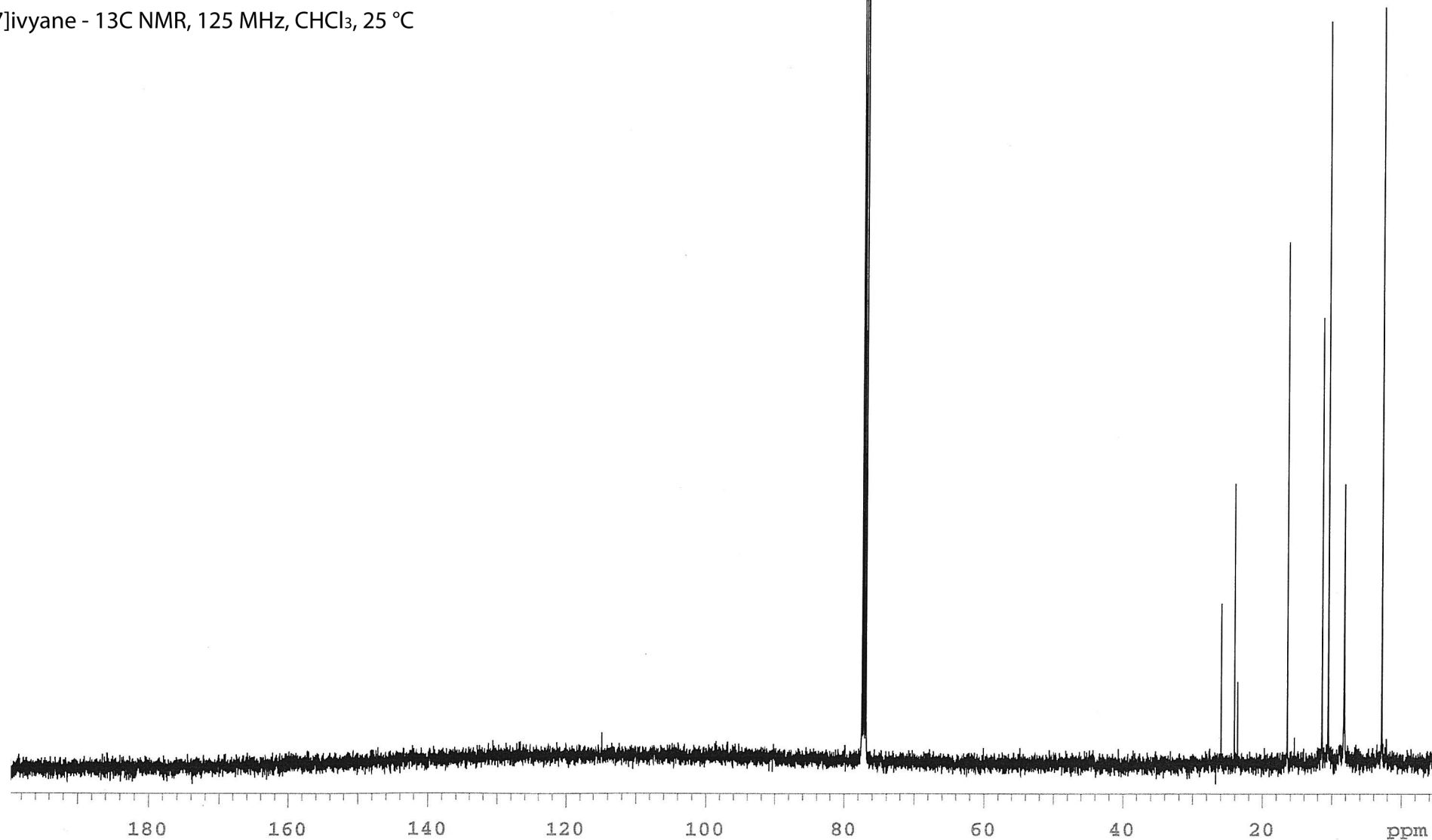


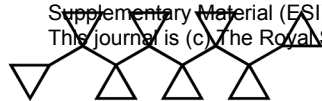
[7]ivylene - ¹H NMR, 500 MHz, CHCl₃, 25 °C



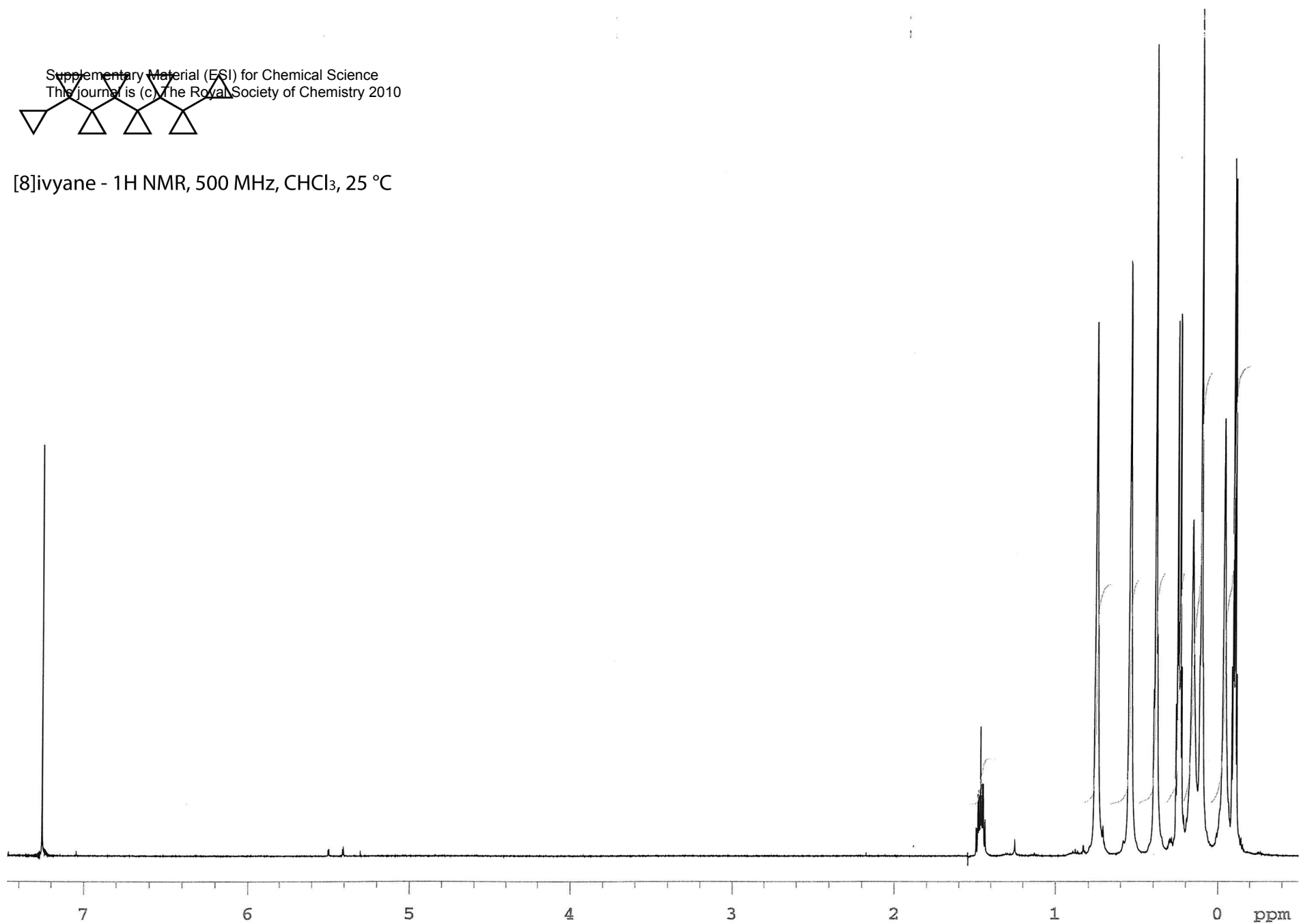


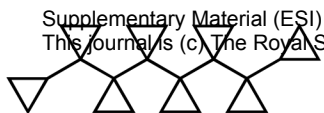
[7]ivane - ^{13}C NMR, 125 MHz, CHCl_3 , 25 °C



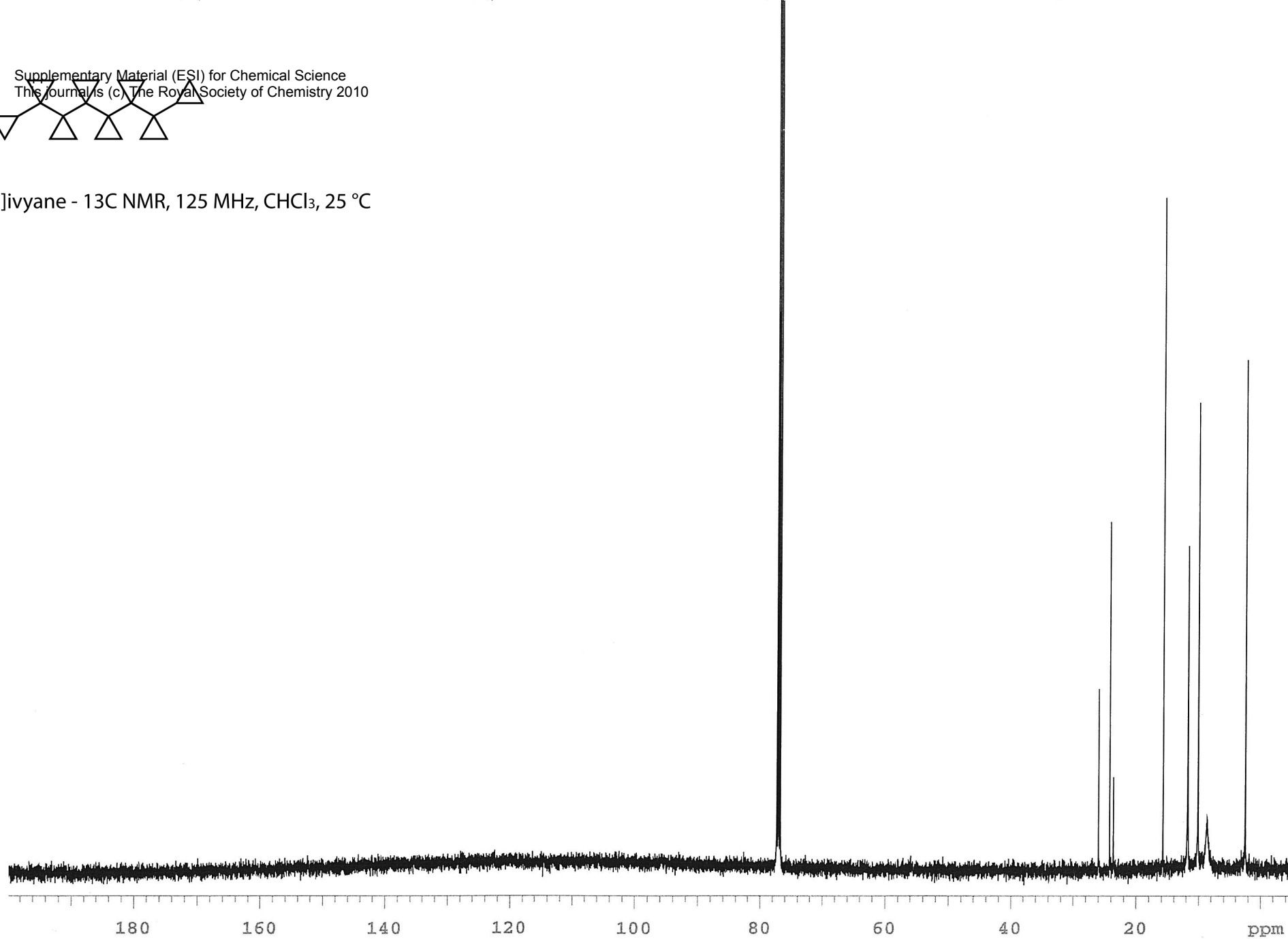


[8]ivylene - ^1H NMR, 500 MHz, CHCl_3 , 25 °C



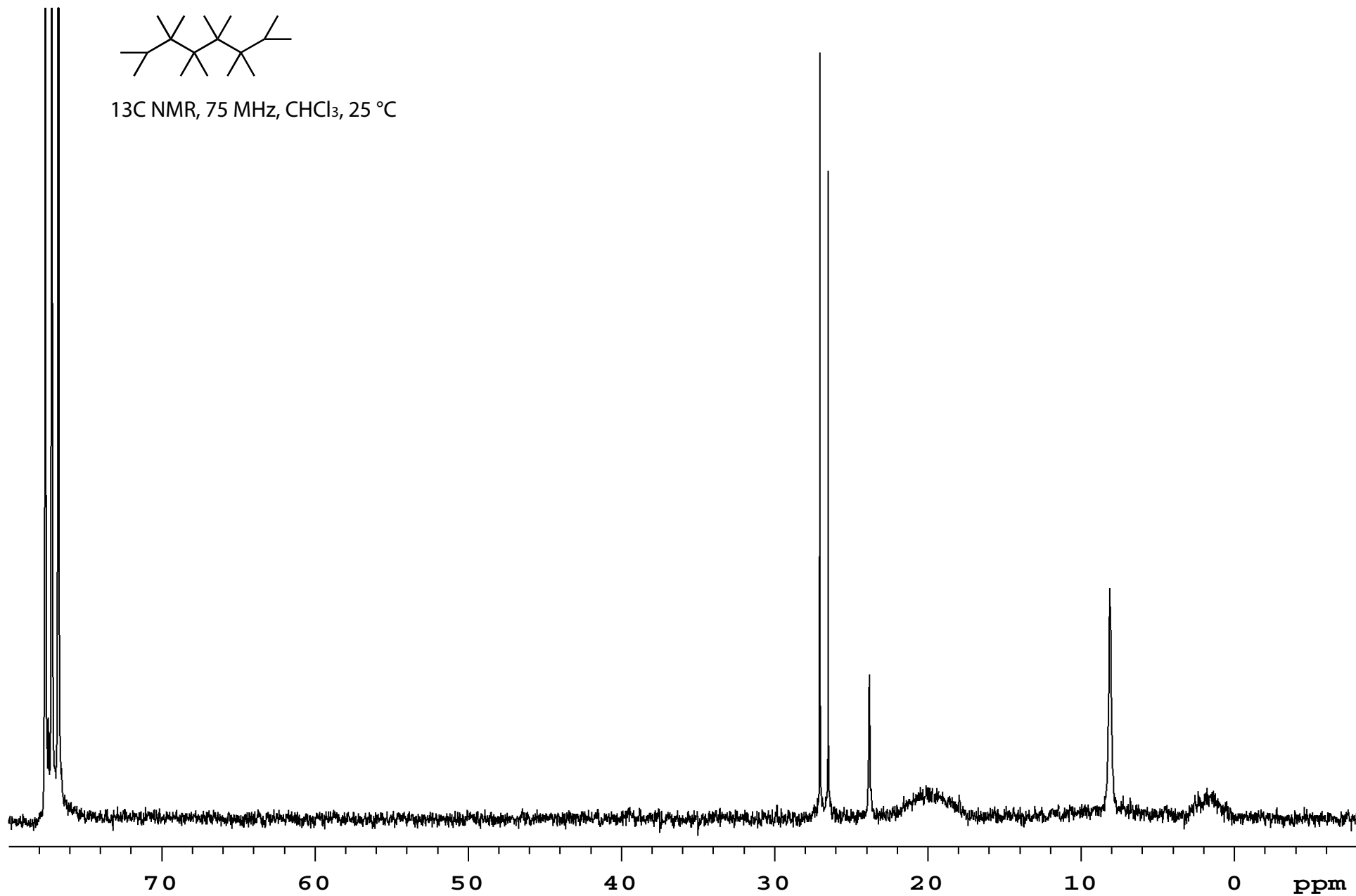


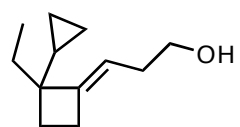
[8]ivylene - ^{13}C NMR, 125 MHz, CHCl_3 , 25 $^\circ\text{C}$



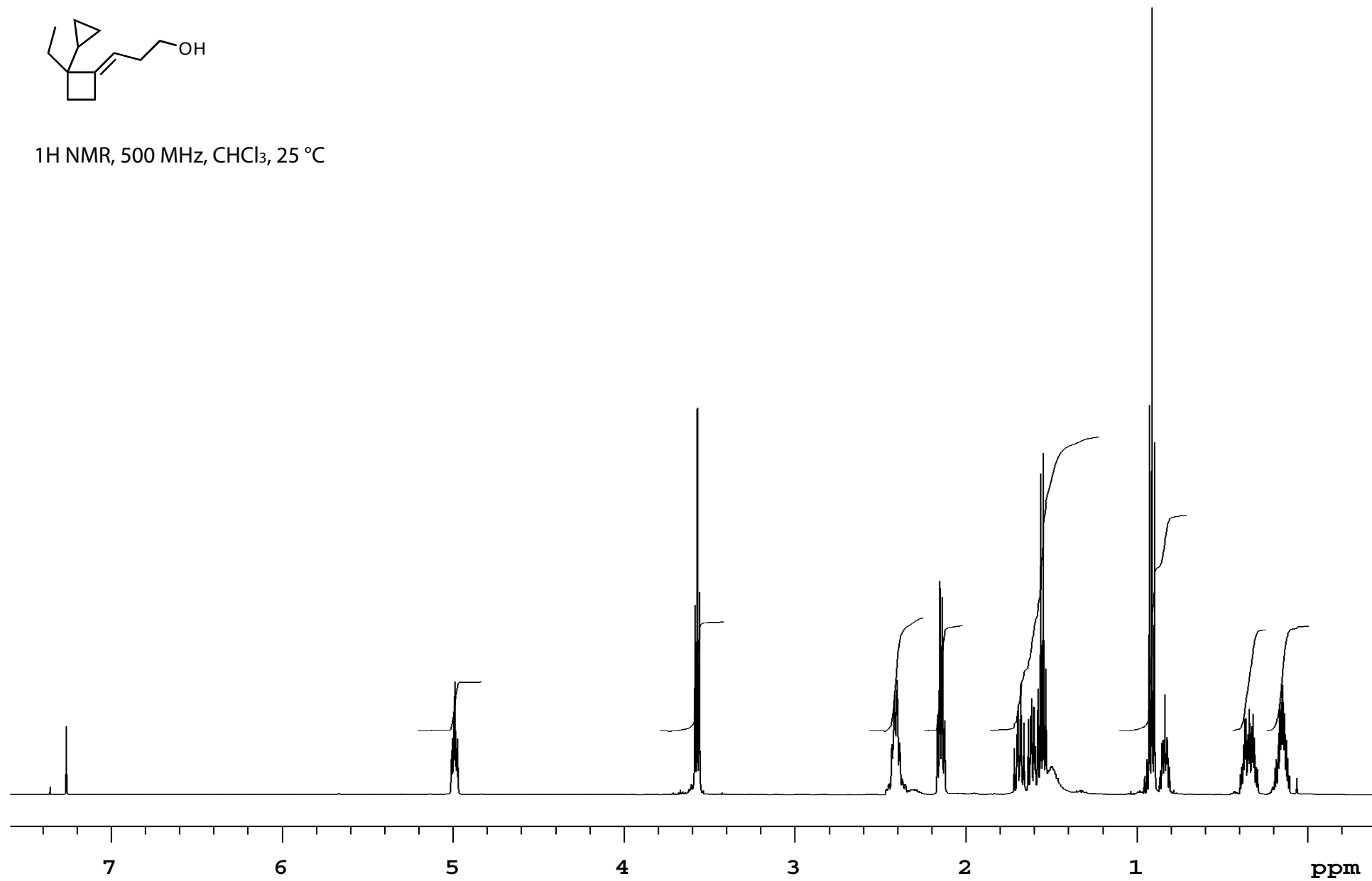


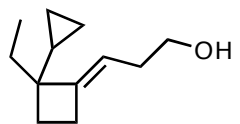
^{13}C NMR, 75 MHz, CHCl_3 , 25 °C





$^1\text{H NMR}$, 500 MHz, CHCl_3 , 25 $^\circ\text{C}$





^{13}C NMR, 75 MHz, CHCl_3 , 25 °C

