Supplementary Material (ESI) for Chemical Communications

Supplementary Information of

Enantioselective Prévost and Woodward reactions using chiral hypervalent iodine(III): switchover of stereochemical course of optically active 1,3-dioxolan-2-yl cation

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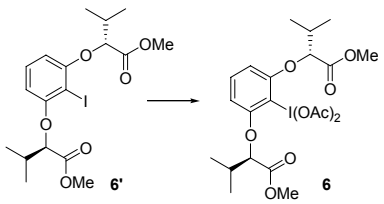
Experimental Section

General. Proton and ^{13}C NMR spectra were measured on a JEOL ECA-600 spectrometer as solutions in $CDCl_3$. Proton NMR spectra were recorded using the residual $CHCl_3$ as an internal reference (7.24 ppm) and ^{13}C NMR using $CDCl_3$ as an internal reference (77.00 ppm). For mass spectra measurements was used JEOL JMS-T100LC. Optical rotations were measured on a Perkin-Elmer 241 polarimeter. X-ray diffraction data were recorded using a Quantum-1 CCD/AFC-7R diffractometer. Styrene substrates **1f–j** were purchased from TCI and used without further purification. Dichloromethane was purified by distillation over CaH_2. Reaction temperature of the dioxyacetylation was controlled using a low temperature bath with magnetic stirrer (EYELA, PSL-1800).

Preparation of (diacetoxyiodo)arene. Optically active aryl-λ^3-iodanes **3**–**5** were prepared as reported previously.[S1] An optically active aryl-λ^3-iodane **6** was also prepared by similar procedures for preparation of **3**–**5**. The corresponding iodoarene **6'** was stereospecifically prepared by the Mitsunobu reaction of 2-iodobenzene-1,3-diol and an α-hydroxycarboxylate as follows.



A solution of diisopropyl azodicarboxylate (1.9 M in toluene × 28 mL, 53 mmol) was added dropwise to a solution of Ph_3P (14.3 g, 54.5 mmol), (*S*)-methyl 2-hydroxy-3-methylbutanoate (7.20 g, 54.5 mmol), and 2-iodobenzene-1,3-diol (3.20 g, 13.6 mmol) in THF (60 mL). The mixture was stirred overnight at ambient temperature, and then concentrated in vacuo. Purification of the residue by chromatography (SiO_2; eluent; 20% EtOAc in hexane) gave **6'** (2.77 g, 5.97 mmol, 44% yield). Selected data for **6'**: $[\alpha]_D^{20}$ +5.9 (*c* 0.94 in $CHCl_3$); δ_H(600 MHz; $CDCl_3$) 7.09 (1H, t, *J* 8.2), 6.22 (2H, d, *J* 8.2), 4.42 (2H, d, *J* 4.8), 3.71 (6H, s), 2.33 (2H, sept, d, *J* 6.9 and 4.8), 1.15 (6H, d, *J* 6.9) and 1.11 (6H, d, *J* 6.9); δ_C(150 MHz; $CDCl_3$) 171.25, 158.22, 129.53, 105.16, 82.24, 78.96, 52.02, 31.92, 18.97 and 17.67; *m/z* (ESI+) 487.0617 (M + Na. $C_{18}H_{25}O_6INa$ requires 487.0594) Oxidation of **6'** (2.34 g, 5.0 mmol) by acetic peracid gave **6** (0.84 g, 1.44 mmol, 28% yield) as solid. Selected data for **6**: $[\alpha]_D^{20}$ –73.5 (*c* 1.02 in $CHCl_3$); δ_H(600 MHz; $CDCl_3$) 7.35 (1H, t, *J* 8.2), 6.47 (2H, d, *J* 8.2), 4.55 (2H, d, *J* 4.8), 3.70 (6H, s), 2.34 (2H, m), 1.94 (6H, s) and 1.07 (12H, d, *J* 6.9); δ_C(150 MHz; $CDCl_3$) 176.95, 170.34, 156.76, 135.20, 105.71, 105.07, 82.21, 52.18, 31.70, 20.38, 18.74 and 17.44; *m/z* (ESI+ in MeOH) 495.0872 (M – 2×OAc + OMe. $C_{19}H_{28}O_7I$ requires 495.0880). Recrystallization for X-ray crystallographic analysis was carried out using CH_2Cl_2/hexane/ethyl acetate. X-ray crystallographic data for **6**·0.5H_2O have been deposited at the Cambridge Crystallographic Centre: Deposition number: CCDC 798662. The data were also summarized in Table S1 and Figure S1.

Table S1. Crystal data and structure refinement for optically active hypervalent iodine(III) **6**.

CCDC[a]	798662	γ (°)	120.000
Formula	$C_{22}H_{32}O_{10.5}I$	V (Å^3)	4062.0(3)
	($C_{22}H_{31}O_{10}I$ +0.5H_2O)	Z	6
Fw	591.39	D_{calcd} (g/cm^{-3})	1.450
Crystal system	hexagonal	μ (cm^{-1})	12.33
Space group	$P6_5$	$2\theta_{max}$ (°)	55.0
a (Å)	23.3688(11)	no. of data collected	5061
b (Å)	23.3688(11)	no. of parameters	342
c (Å)	8.5888(2)	R	0.0517
α (°)	90.000	Rw	0.0577
β (°)	90.000	Flack parameter	0.07(3)

[a] The crystallographic data have been deposited at the Cambridge Crystallographic Data Centre (CCDC) and the deposition numbers are shown here. These data can be obtained free of charge from CCDC via www.ccdc.cam.ac.uk/data_request/cif.

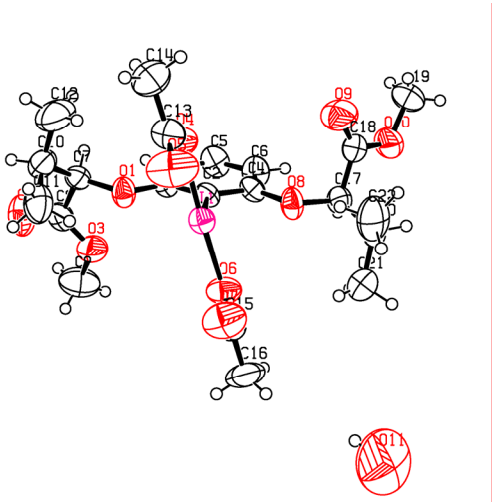
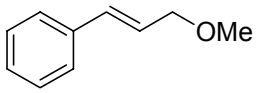
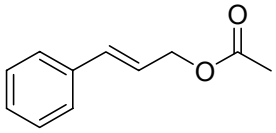


Figure S1. Crystal structure of **6**·0.5H_2O.

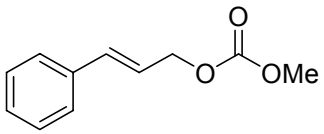
Substrates 1a-e were prepared from the corresponding cinnamyl alchol. Methyl ether substrates, **1a**, **1d**, and **1e**, were obtained by methylation with iodomethane in the presence of sodium hydride in DMF. The acetate **1b** and carbonate **1c** was prepared with acetic anhydride and methyl chloroformate, respectively.



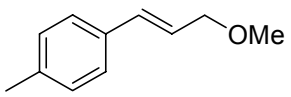
(*E*)-(3-Methoxyprop-1-enyl)benzene (1a):[S2] δ_H(600 MHz; $CDCl_3$) 7.38 (2H, d, *J* 7.6), 7.30 (2H, t, *J* 7.6), 7.23 (1H, t, *J* 7.6), 6.60 (1H, d, *J* 15.8), 6.27 (1H, dt, *J* 15.8 and 6.2), 4.08 (2H, d, *J* 6.2) and 3.38 (3H, s); δ_C(150 MHz; $CDCl_3$) 136.74, 132.43, 128.54, 127.64, 126.46, 125.98, 73.08 and 57.97.



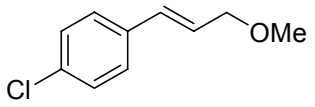
Cinnamyl acetate (1b):[S3] δ_H(600 MHz; $CDCl_3$) 7.37 (2H, d, *J* 7.6), 7.31 (2H, t, *J* 7.6), 7.25 (1H, t, *J* 7.6), 6.64 (1H, d, *J* 15.8), 6.27 (1H, dt, *J* 15.8 and 6.2), 4.71 (2H, d, *J* 6.2) and 2.08 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.81, 136.23, 134.21, 128.60, 128.06, 126.61, 123.20, 65.05 and 20.98.



Cinnamyl methyl carbonate (1c):[S4] δ_H(600 MHz; $CDCl_3$) 7.38 (2H, d, *J* 7.6), 7.31 (2H, t, *J* 7.6), 7.25 (1H, t, *J* 7.6), 6.67 (1H, d, *J* 15.8), 6.28 (1H, dt, *J* 15.8, 6.2), 4.78 (2H, d, *J* 6.2) and 3.79 (3H, s); δ_C(150 MHz; $CDCl_3$) 155.64, 136.00, 134.77, 128.58, 128.17, 126.66, 122.38, 68.38 and 54.81.



1-((*E*)-3-Methoxyprop-1-enyl)-4-methylbenzene (1d):[S5] δ_H(600 MHz; $CDCl_3$) 7.27 (2H, d, *J* 7.6), 7.11 (2H, d, *J* 7.6), 6.56 (1H, d, *J* 15.8), 6.21 (1H, dt, *J* 15.8 and 6.2), 4.06 (2H, d, *J* 6.2), 3.37 (3H, s) and 2.32 (3H, s); δ_C(150 MHz; $CDCl_3$) 137.50, 133.93, 132.50, 129.24, 126.39, 124.87, 73.20, 57.89 and 21.17.



1-Chloro-4-((*E*)-3-methoxyprop-1-enyl)benzene (1e):[S6] δ_H(600 MHz; $CDCl_3$) 7.29 (2H, d, *J* 8.2), 7.26 (2H, d, *J* 8.2), 6.55 (1H, d, *J* 16.5), 6.24 (1H, dt, *J* 16.5 and 6.2), 4.06 (2H, d, *J* 6.2) and 3.37 (3H, s); δ_C(150 MHz; $CDCl_3$) 135.22, 133.26, 131.00, 128.70, 127.64, 126.68, 72.87 and 58.10.

Typical procedures for the dioxyacetylation under conditions A. (Diacetoxyiodo)arene (0.5 mmol) and substrate **1** (0.4 mmol) were dissolved in dichloromethane (4 mL) in the presence of acetic acid (0.2 mL). The solution was cooled at –80 °C using a low temperature bath with magnetic stirrer (EYELA, PSL-1800). Boron trifluoride diethyl etherate (0.1 mL) was added to the solution at –80 °C. The solution was gradually warmed up to –40 °C over 2 h, and additionally stirred at –40 °C for 1 h. The reaction mixture was then quenched by adding water, and extracted with dichloromethane. The organic phase was dried with Na_2SO_4 and concentrated in vacuo. The

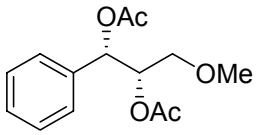
crude mixture was checked by ^{1}H NMR, and then acetylated with acetic anhydride in dichloromethane containing pyridine and 4-(dimethylamino)pyridine. The mixture was worked up and purified by column chromatography (SiO_2, eluent: 10%–20% ethyl acetate in hexane). Yields were calculated from weight of the isolated product. The diastereomers, *syn*-**2** and *anti*-**2**, were not separated under the conditions.

Enantiomeric ratio of the product was determined by GC analysis. Most of the GC analyses were carried out after hydrolysis of the acetate product **2** under basic conditions. The alcohol obtained by hydrolysis was treated with trimethysilylimidazole, and then injected into a chiral GC column (CP-Chirasil-DEX CB, i.d. 0.25 mm × 25 m).

Typical procedures for the dioxyacetylation under conditions B. (Diacetoxyiodo)arene (0.5 mmol) and substrate **1** (0.4 mmol) were dissolved in dichloromethane (4 mL) in the presence of acetic acid (0.2 mL) and trimethylsilyl acetate (0.2 mL). The solution was cooled at –80 °C using a low temperature bath with magnetic stirrer (EYELA, PSL-1800). Boron trifluoride diethyl etherate (0.1 mL) was added to the solution at –80 °C. The solution was gradually warmed up to room temperature over 10 h. The reaction mixture was then quenched by adding water, and extracted with dichloromethane. The organic phase was dried with Na_2SO_4 and concentrated in vacuo. The mixture was purified by column chromatography (SiO_2, eluent: 10%–20% ethyl acetate in hexane). Yields were calculated from weight of the isolated product.

Enantiomeric ratio of the product was determined by GC or HPLC analyses. Most of the GC analyses were carried out after hydrolysis of the acetate product **2** under basic conditions. The alcohol obtained by hydrolysis was treated with trimethysilylimidazole, and then injected into a chiral GC column (CP-Chirasil-DEX CB, i.d. 0.25 mm × 25 m). HPLC analysis was carried out on a chiral stationary phase (Daicel Chiralpak AD (0.46 cm ϕ × 25 cm)).

(1*S*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-phenylpropyl ester ((1*S*,2*S*)-*syn*-**2a**): δ_H(600 MHz; $CDCl_3$) 7.38–7.28 (5H, m), 5.99 (1H, d, *J* 8.2), 5.32 (1H, ddd, *J* 8.2, 4.8 and 3.4), 3.38 (1H, dd, *J* 11.0 and 3.4), 3.24 (3H, s), 3.17 (1H, dd, *J* 11.0 and 4.8), 2.06 (3H, s) and 2.05 (3H, s);



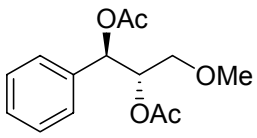
δ_C(150 MHz; $CDCl_3$) 170.28, 169.71, 136.70, 128.63, 128.57, 127.26, 74.16, 73.78, 70.65, 59.14, 20.96 and 20.92; *m/z* (ESI+) 289.1049 (M + Na. $C_{14}H_{18}O_5Na$ requires 289.1052); $[\alpha]_D^{20}$ +55.2 (*c* 0.52 in $CHCl_3$) for 95% ee of (1*S*,2*S*)-**2a**.

The NMR data agree well with the reported values.[S7] The reported values: δ_H(300 MHz, $CDCl_3$) 7.40–7.30 (5H, m), 6.01 (1H, d, *J* 8.0), 5.33 (1H, ddd, *J* 7.6, 4.8 and 3.2), 3.39 (1H, dd, *J* 10.8 and 3.2), 3.25 (3H, s), 3.18 (1H, dd, *J* 10.8 and 4.8), 2.08 (3H, s) and 2.06 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.53, 169.97, 136.90, 128.86, 128.80, 127.47, 74.37, 73.99, 70.85, 59.37, 21.21 and 21.17.

Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained

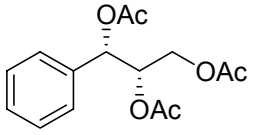
by hydrolysis of *syn*-**2a** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 31 min (1*R*,2*R*) and 32 min (1*S*,2*S*), when temperature of the column (DEX-CB) was maintained at 120 °C. Under the same conditions, the retention time of the *anti* isomer was 30 min (the enantiomers were not separated.). Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.37–7.26 (5H, m), 4.70 (1H, d, *J* 6.2), 3.80 (1H, m), 3.38 (1H, dd, *J* 9.6 and 3.4), 3.34 (3H, s), 3.32 (1H, dd, *J* 9.6 and 4.8), 2.96 (1H, br) and 2.66 (1H, br); δ_C(150 MHz; $CDCl_3$) 140.49, 128.45, 128.00, 126.64, 74.83, 74.62, 73.46 and 59.18; *m/z* (ESI+) 205.0837 (M + Na. $C_{10}H_{14}O_3Na$ requires 205.0841).

(1*R*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-phenylpropyl ester ((1*R*,2*S*)-*anti*-**2a**): δ_H(600 MHz; $CDCl_3$) 7.35–7.27 (5H, m), 6.00 (1H, d, *J* 5.5), 5.37 (1H, ddd, *J* 6.9, 5.5 and 3.4), 3.50 (1H, dd, *J* 11.0 and 6.9), 3.42 (1H, dd, *J* 11.0 and 3.4), 3.30 (3H, s), 2.09 (3H, s) and 1.97 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.13, 169.56, 136.20, 128.35, 127.11, 73.60, 73.09, 70.19, 59.10, 20.99 and 20.85; *m/z* (ESI+) 289.1069 (M + Na. $C_{14}H_{18}O_5Na$ requires 289.1052); $[\alpha]_D^{20}$ –49.3 (*c* 1.20 in $CHCl_3$) for 96% ee of (1*R*,2*S*)-**2a**.



Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of *anti*-**2a** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 87 (1*R*,2*S*) and 89 (1*S*,2*R*) min, when temperature of the column (DEX-CB) was maintained at 100 °C. Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.37–7.27 (5H, m), 4.86 (1H, m), 3.93 (1H, m), 3.45 (1H, dd, *J* 9.6 and 6.2), 3.35 (s, 3H), 3.34 (1H, dd, *J* 9.6 and 3.4), 2.99 (1H, br) and 2.56 (1H, br); δ_C(150 MHz; $CDCl_3$) 140.26, 128.43, 127.74, 126.13, 75.35, 73.23, 72.99 and 59.19; *m/z* (ESI+) 205.0844 (M + Na. $C_{10}H_{14}O_3Na$ requires 205.0841).

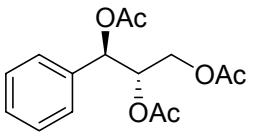
(1*S*,2*S*)-1-Phenyl-1,2,3-triacetoxypropane ((1*S*,2*S*)-*syn*-**2b**): δ_H(600 MHz; $CDCl_3$) 7.35–7.29 (5H, m), 5.94 (1H, d, *J* 7.6), 5.41 (1H, ddd, *J* 7.6, 5.5 and 3.4), 4.24 (1H, dd, *J* 11.7 and 3.4), 3.78 (1H, dd, *J* 11.7 and 5.5), 2.06 (3H, s), 2.03 (3H, s) and 2.03 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.40, 169.99, 169.69, 135.99, 128.93, 128.75, 127.19, 73.93, 72.35, 62.21, 20.93, 20.74 and 20.64; *m/z* (ESI+) 317.1001 (M + Na. $C_{15}H_{18}O_6Na$ requires 317.1001); $[\alpha]_D^{20}$ +26.2 (*c* 0.60 in $CHCl_3$) for 92% ee of (1*S*,2*S*)-**2b**. The NMR data agree well with the reported values.[S8] Enantiomeric ratio of the product was determined by chiral GC analysis of *syn*-**2b** itself. Retention times of (1*R*,2*R*)-**2b** and (1*S*,2*S*)-**2b** were 60 and 61 min, when temperature of the column (Chirasil-DEX-CB) was



maintained at 140 °C.

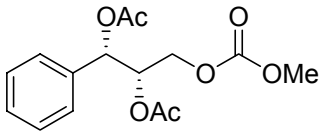
The sample was hydrolyzed under basic conditions, and the triol was obtained. Proton nmr of the triol obtained agrees with that of the *syn* isomer reported. Optically rotation of the triol obtained ($[\alpha]_D^{20}$ +33.8 (*c* 0.31 in $CHCl_3$)) indicates that the sample has (1*S*,2*S*) configuration; reported value for (1*S*,2*S*)-1-phenylpropane-1,2,3-triol:[S9] $[\alpha]_D$ +20.92 (*c* 3.68 in $CHCl_3$).

(1*R*,2*S*)-1-Phenyl-1,2,3-triacetoxypropane ((1*R*,2*S*)-*anti*-**2b**): Reaction of **1b** with (diacetoxyiodo)benzene under the conditions B gave a mixture of *anti*-**2b** and a byproduct (87/13).[S10] The combined yield of the mixture was 79% yield, but these two products were not separated by column chromatography (SiO_2, eluent: ethyl acetate in hexane). Thus, pure *anti*-**2b** was obtained by conversion from *anti*-**2c**, which formed in the reaction of **1c** under the conditions B. Selected data for *anti*-**2b**: δ_H(600 MHz; $CDCl_3$) 7.35–7.28 (5H, m), 5.98 (1H, d, *J* 5.5), 5.38 (1H, q, *J* 4.1), 4.22–4.18 (2H, m), 2.11 (3H, s), 2.00 (3H, s) and 1.97 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.60, 169.93, 169.52, 135.89, 128.55, 128.52, 126.84, 73.21, 72.52, 61.70, 20.93, 20.73 and 20.65; *m/z* (ESI+) 317.1005 (M + Na. $C_{15}H_{18}O_6Na$ requires 317.1001). The NMR data agree well with the reported values.[S11] $[\alpha]_D^{20}$ –25.9 (*c* 0.45 in $CHCl_3$) for 84% ee of (1*R*,2*S*)-**2b**; For reported value of (1*S*,2*R*)-**2b**: $[\alpha]_D^{25}$ +34 (*c* 9.0 in $CHCl_3$).[S12]



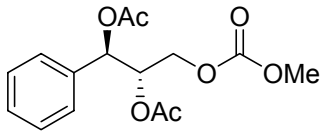
Enantiomeric ratio of the product was determined by chiral GC analysis of (1*R**,2S*)-1-phenylpropan-1,2,3-triol. After treatment of the 1,2,3-triol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 46 min (1*R*,2*S*) and 47 min (1*S*,2*R*), when temperature of the column (DEX-CB) was maintained at 120 °C.

(1*S*,2*S*)-1,2-Diacetoxy-3-methoxycarbonyloxy-1-phenylpropan e ((1*S*,2*S*)-*syn*-**2c**): δ_H(600 MHz; $CDCl_3$) 7.36–7.29 (5H, m), 5.96 (1H, d, *J* 7.6), 5.41 (1H, m), 4.28 (1H, dd, *J* 11.7 and 3.4), 3.85 (1H, dd, *J* 11.7 and 5.5), 3.75 (3H, s), 2.06 (3H, s) and 2.04 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.00, 169.63, 155.32, 135.82, 128.93, 128.75, 127.13, 73.68, 72.19, 65.52, 55.03, 20.90 and 20.72; *m/z* (ESI+) 333.0960 (M + Na. $C_{15}H_{18}O_7Na$ requires 333.0950); $[\alpha]_D^{20}$ +16.9 (*c* 1.09 in $CHCl_3$) for 89% ee of (1*S*,2*S*)-**2c**.



Enantiomeric ratio of the product was determined by chiral GC analysis of the 1-phenyl-1,2,3-triacetoxypropane (*syn*-**2b**) obtained by transformation of hydrolysis of *syn*-**2c** under basic conditions and acetylation with acetic anhydride.

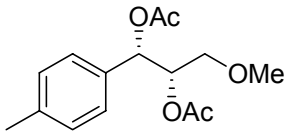
(1*R*,2*S*)-1,2-Diacetoxy-3-methoxycarbonyloxy-1-phenylpropan



e ((1*R*,2*S*)-*anti*-**2c**): δ_H(600 MHz; $CDCl_3$) 7.35–7.28 (5H, m), 6.00 (1H, d, *J* 6.2), 5.38 (1H, td, *J* 6.2 and 3.4), 4.29 (1H, dd, *J* 11.7 and 6.2), 4.27 (1H, dd, *J* 11.7 and 3.4), 3.75 (3H, s), 2.12 (3H, s) and 1.97 (3H, s); δ_C(150 MHz; $CDCl_3$) 169.94, 169.48, 155.44, 135.76, 128.61, 128.57, 126.75, 73.08, 72.46, 65.05, 55.05, 20.95 and 20.73; *m/z* (ESI+) 333.0968 (M + Na. $C_{15}H_{18}O_7Na$ requires 333.0950); $[\alpha]_D^{20}$ –26.2 (*c* 1.17 in $CHCl_3$) for 84% ee of (1*R*,2*S*)-**2c**.

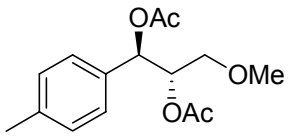
Enantiomeric ratio of the product was determined by chiral GC analysis of the 1-phenylpropane-1,2,3-triol obtained by hydrolysis of *anti*-**2c** under basic conditions. After treatment of the 1,2,3-triol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 46 min (1*R*,2*S*) and 47 min (1*S*,2*R*), when temperature of the column (DEX-CB) was maintained at 120 °C.

(1*S*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-(4-methylphenyl)-propyl ester ((1*S*,2*S*)-*syn*-**2d**): δ_H(600 MHz; $CDCl_3$) 7.25 (2H, d, *J* 7.6), 7.14 (2H, d, *J* 7.6), 5.95 (1H, d, *J* 8.2), 5.31 (1H, m), 3.37 (1H, dd, *J* 11.0 and 2.7), 3.23 (3H, s), 3.17 (1H, dd, *J* 11.0 and 4.8), 2.32 (3H, s), 2.07 (3H, s) and 2.03 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.34, 169.75, 138.48, 133.68, 129.28, 127.23, 74.08, 73.81, 70.71, 59.13, 21.18, 20.98 and 20.96; *m/z* (ESI+) 303.1228 (M + Na. $C_{15}H_{20}O_5Na$ requires 303.1208); $[\alpha]_D^{20}$ +50.7 (*c* 0.90 in $CHCl_3$) for 88% ee of (1*S*,2*S*)-**2d**.



Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of *syn*-**2d** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 48 (1*R*,2*R*) and 49 (1*S*,2*S*) min, when temperature of the column (DEX-CB) was maintained at 120 °C. Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.24 (2H, d, *J* 8.2), 7.15 (2H, d, *J* 8.2), 4.66 (1H, d, *J* 6.2), 3.78 (1H, m), 3.36 (1H, dd, *J* 10.3 and 3.4), 3.34 (3H, s), 3.31 (1H, dd, *J* 10.3 and 5.5), 2.88 (1H, br), 2.65 (1H, br) and 2.33 (3H, s); δ_C(150 MHz; $CDCl_3$) 137.69,137.49, 129.13, 126.56, 74.65, 73.46, 59.14 and 21.10; *m/z* (ESI+) 219.1009 (M + Na. $C_{11}H_{16}O_3Na$ requires 219.0997).

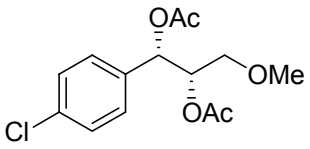
(1*R*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-(4-methylphenyl)-propyl ester ((1*R*,2*S*)-*anti*-**2d**): δ_H(600 MHz; $CDCl_3$) 7.22 (2H, d, *J* 7.6), 7.13 (2H, d, *J* 7.6), 5.95 (1H, d, *J* 4.8), 5.36 (1H, m), 3.48 (1H, dd, *J* 10.3 and 6.9), 3.41 (1H, dd, *J* 10.3 and 4.1), 3.29 (3H, s), 2.31 (3H, s), 2.08 (3H, s) and 1.98 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.16, 169.59, 138.11, 133.14, 129.02, 127.05, 73.52, 73.05, 70.23, 59.06, 21.14, 20.99 and 20.87; *m/z* (ESI+) 303.1214 (M + Na.



$C_{15}H_{20}O_5Na$ requires 303.1208); $[\alpha]_D^{20}$ –36.7 (*c* 1.32 in $CHCl_3$) for a sample of 88% ee of (1*R*,2*S*)-**2d**. The sample included 18% *syn*-**2d** (88% ee of (1*S*,2*S*)-**2d**).

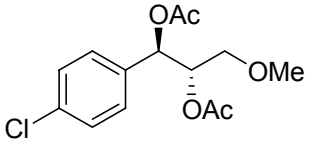
Enantiomeric ratio of the product was determined by chiral HPLC analysis with Chiralpak AD. Retention times of (1*S*,2*R*)-**2d** and (1*R*,2*S*)-**2d** were 10 min and 14 min, respectively, when 97/3 of hexane/EtOAc was used as an eluent. Under the conditions, the *syn* product, (1*R**,2*R**)-**2d**, appeared at 9 min. Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.23 (2H, d, *J* 7.6), 7.16 (2H, d, *J* 7.6), 4.80 (1H, d, *J* 4.8), 3.90 (1H, m), 3.45 (1H, dd, *J* 9.6 and 6.2), 3.37 (1H, dd, *J* 9.6 and 4.1), 3.33 (3H, s) and 2.33 (3H, s); δ_C(150 MHz; $CDCl_3$) 137.51,137.26, 129.17, 126.08, 75.32, 73.23, 73.11, 59.23 and 21.11.

(1*S*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-(4-chlorophenyl)-propyl ester ((1*S*,2*S*)-*syn*-**2e**): δ_H(600 MHz; $CDCl_3$) 7.30 (4H, s), 5.96 (1H, d, *J* 7.6), 5.26 (1H, m), 3.39 (1H, dd, *J* 11.0 and 3.4), 3.24 (3H, s), 3.16 (1H, dd, *J* 11.0 and 4.8), 2.06 (3H, s) and 2.04 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.18, 169.64, 135.27, 134.52, 128.81, 128.61, 73.50, 73.47, 70.50, 59.17 and 20.89; *m/z* (ESI+) 323.0670 (M + Na. $C_{14}H_{17}O_5{}^{35}ClNa$ requires 323.0662); $[\alpha]_D^{20}$ +51.6 (*c* 0.71 in $CHCl_3$) for 96% ee of (1*S*,2*S*)-**2e**.

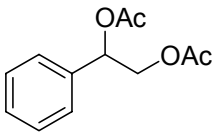


Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of *syn*-**2e** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 49 (1*R*,2*R*) and 50 (1*S*,2*S*) min, when temperature of the column (DEX-CB) was maintained at 130 °C. Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.30 (2H, d, *J* 8.2), 7.28 (2H, d, *J* 8.2), 4.66 (1H, d, *J* 6.2), 3.72 (1H, m), 3.36 (1H, dd, *J* 9.6 and 3.4), 3.32 (3H, s) and 3.28 (1H, dd, *J* 9.6 and 5.5); δ_C(150 MHz; $CDCl_3$) 139.04, 133.72, 128.61, 128.01, 74.47, 74.21, 73.46 and 59.25; *m/z* (ESI+) 239.0471 (M + Na. $C_{10}H_{13}O_3{}^{35}ClNa$ requires 239.0451).

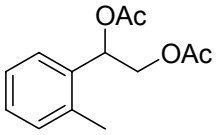
(1*R*,2*S*)-Acetic acid 2-acetoxy-3-methoxy-1-(4-chlorophenyl)-propyl ester ((1*R*,2*S*)-*anti*-**2e**): δ_H(600 MHz; $CDCl_3$) 7.30 (2H, d, *J* 8.2), 7.28 (2H, d, *J* 8.2), 5.95 (1H, d, *J* 5.5), 5.33 (1H, q, *J* 5.5), 3.48 (1H, dd, *J* 10.3 and 6.2), 3.38 (1H, dd, *J* 10.3 and 4.1), 3.30 (3H, s), 2.08 (3H, s) and 1.97 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.00, 169.44, 134.77, 134.28, 128.62, 128.56, 72.87, 72.71, 70.11, 59.14, 20.92 and 20.83; *m/z* (ESI+) 323.0672 (M + Na. $C_{14}H_{17}O_5{}^{35}ClNa$ requires 323.0662); $[\alpha]_D^{20}$ –51.8 (*c* 0.62 in $CHCl_3$) for 92% ee of (1*R*,2*S*)-**2e**. Enantiomeric ratio of the product was determined by chiral HPLC analysis with Chiralpak AD. Retention times of the enantiomers were 9 min ((1*S*,2*R*)-(+)-**2e**) and 12 min ((1*R*,2*S*)-(–)-**2e**), when 97/3 of hexane/EtOAc was used as an eluent.



1,2-Diacetoxy-1-phenylethane (**2f**):[S7] δ_H(600 MHz; $CDCl_3$) 7.36–7.30 (5H, m), 5.99 (1H, dd, *J* 8.2 and 3.4), 4.31 (1H, dd, *J* 11.7 and 3.4), 4.27 (1H, dd, *J* 11.7 and 8.2), 2.10 (3H, s) and 2.04 (3H, s). Enantiomeric ratio of the product was determined by chiral GC analysis (Chirasil-DEX-CB). Retention times of (*R*)-**2f** and (*S*)-**2f** were 49 and 50 min, respectively, when temperature of the column (Chirasil-DEX-CB) was maintained at 115 °C. Authentic sample of (*R*)-**2f** was prepared by acetylation of (*R*)-(–)-phenylethan-1,2-diol (TCI).

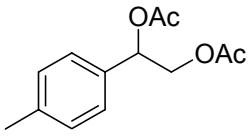


1,2-Diacetoxy-1-(2-methylphenyl)ethane (**2g**): δ_H(600 MHz; $CDCl_3$) 7.34 (1H, m), 7.20–7.14 (3H, m), 6.21 (1H, dd, *J* 8.2 and 3.4), 4.27 (1H, dd, *J* 11.7 and 3.4), 4.20 (1H, dd, *J* 11.7 and 8.2), 2.42 (3H, s), 2.09 (3H, s) and 2.05 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.70, 170.06, 135.54, 134.81, 130.56, 128.38, 126.22, 126.03, 70.40, 65.46, 21.06, 20.78 and 19.08; $[\alpha]_D^{20}$ +13.4 (*c* 0.99 in $CHCl_3$) for 34% ee of (*S*)-**2g**; $[\alpha]_D^{20}$ –4.3 (c 0.73 in $CHCl_3$) for 11% ee of (*R*)- **2g**; *m/z* (ESI+) 259.0967 (M + Na. $C_{13}H_{16}O_4Na$ requires 259.0946).



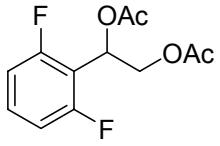
Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of **2g** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 35 min (*R*) and 36 min (*S*), when temperature of the column (DEX-CB) was maintained at 110 °C. Selected data for the diol: δ_H(600 MHz; $CDCl_3$) 7.48 (1H, d, *J* 7.6), 7.2–7.1 (3H, m), 5.06 (1H, m), 3.72 (1H, m), 3.61 (1H, m) and 2.33 (3H, s); δ_C(150 MHz; $CDCl_3$) 138.43, 134.76, 130.44, 127.74, 126.30, 125.64, 71.42, 66.89 and 18.99; *m/z* (ESI+) 175.0765 (M + Na. $C_9H_{12}O_2Na$ requires 175.0735). The data for the diol agree with the reported values.[S13]

1,2-Diacetoxy-1-(4-methylphenyl)ethane (**2h**):[S14] δ_H(600 MHz; $CDCl_3$) 7.23 (2H, d, *J* 7.6), 7.15 (2H, d, *J* 7.6), 5.96 (1H, dd, *J* 7.6 and 4.1), 4.28 (1H, dd, *J* 11.7 and 4.1), 4.27 (1H, dd, *J* 11.7 and 7.6), 2.32 (3H, s), 2.08 (3H, s) and 2.04 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.64, 170.07, 138.45, 133.47, 129.29, 126.66, 73.20, 66.05, 21.14, 21.08 and 20.76; *m/z* (ESI+) 259.0953 (M + Na. $C_{13}H_{16}O_4Na$ requires 259.0946); $[\alpha]_D^{20}$ +6.9 (*c* 0.63 in $CHCl_3$) for 11% ee of (*S*)-**2h**. Lit. ($[\alpha]_D^{25}$ +70.2 (*c* 1.5 in $CHCl_3$) for (*S*)-**2h**[S14]

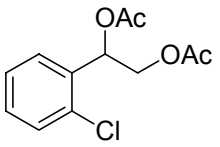


Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of **2h** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB).

Retention times of the enantiomers were 59 min (*R*) and 60 min (*S*), when temperature of the column was maintained at 105 °C. Selected data for the diol:[S15] δ_H(600 MHz; $CDCl_3$) 7.25 (2H, d, *J* 7.6), 7.16 (2H, d, *J* 7.6), 4.78 (1H, m), 3.72 (1H, m), 3.65 (1H, m) and 2.33 (3H, s); δ_C(150 MHz; $CDCl_3$) 137.75, 137.51, 129.21, 125.99, 74.53, 68.06 and 21.09; *m/z* (ESI+) 175.0765 (M + Na. $C_9H_{12}O_2Na$ requires 175.0735).



1,2-diacetoxy-1-(2,6-Difluorophenyl)ethane (**2i**): δ_H(600 MHz; $CDCl_3$) 7.27 (1H, m), 6.88 (2H, t, J_H 8.2, J_F 8.2), 6.30 (1H, dd, *J* 8.2 and 4.1), 4.57 (1H, dd, *J* 11.7 and 8.2), 4.38 (1H, dd, *J* 11.7 and 4.1), 2.08 (3H, s) and 2.04 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.44, 169.92, 161.25 (dd, $J_{C\text{-}F}$ 251 and 8), 130.71 (t, $J_{C\text{-}F}$ 11), 112.60 (t, $J_{C\text{-}F}$ 17), 111.81 (dd, $J_{C\text{-}F}$ 22 and 4), 65.18, 63.80, 20.76 and 20.64; *m/z* (ESI+) 281.0639 (M + Na. $C_{12}H_{12}O_4F_2Na$ requires 281.0601); $[\alpha]_D^{20}$ +12 (*c* 1.1 in $CHCl_3$) for 30% ee of (*S*)-**2i**. Enantiomeric ratio of the product was determined by chiral GC analysis (Chirasil-DEX-CB). Retention times of (*S*)-**2i** and (*R*)-**2i** were 36 min and 37 min, respectively, when temperature of the column (Chirasil-DEX-CB) was maintained at 120 °C.



1-(2-Chlorophenyl)-1,2-diacetoxyethane (**2j**):[S7] δ_H(600 MHz; $CDCl_3$) 7.40 (1H, d, *J* 7.6), 7.34 (1H, d, *J* 7.6), 7.25 (1H, t, *J* 7.6), 7.23 (1H, t, *J* 7.6), 6.36 (1H, dd, *J* 7.6 and 4.1), 4.32 (1H, dd, *J* 12.4 and 4.1), 4.29 (1H, dd, *J* 12.4 and 7.6), 2.11 (3H, s) and 2.02 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.41, 169.54, 134.33, 132.44, 129.67, 129.52, 127.61, 126.97, 70.28, 64.52, 20.86 and 20.61; *m/z* (ESI+) 279.0377 (M + Na. $C_{12}H_{13}O_4{}^{35}ClNa$ requires 279.0400); $[\alpha]_D^{20}$ +25.2 (c 0.45 in $CHCl_3$) for 92% ee of (*S*)-**2j**.

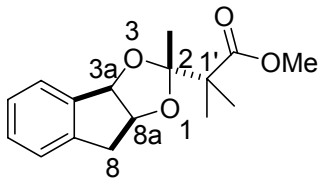
The absolute stereochemistry was determined by comparison with optical rotation of the 1,2-diol hydrolyzed the sample of 88% ee. $([\alpha]_D^{20}$ +63.9 (*c* 0.17 in $CHCl_3$), lit. $[\alpha]_D^{20}$ +70 (*c* 0.2 in $CHCl_3$) for (*S*)-1-(2-chlorophenyl)-1,2-ethanediol)[S16] Selected data for the diol: HRMS (ESI+) calcd for $C_8H_9O_2{}^{35}ClNa$ (M+Na) 195.0189 found 195.0230.

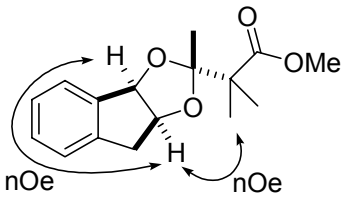
Enantiomeric ratio of the product was determined by chiral GC analysis of the 1,2-diol obtained by hydrolysis of **2j** under basic conditions. After treatment of the 1,2-diol with trimethylsilylimidazole, the sample was injected into a chiral column (Chirasil-DEX-CB). Retention times of the enantiomers were 28 min (*R*) and 29 min (*S*), when temperature of the column (DEX-CB) was maintained at 120 °C.

Assignment of Relative and Absolute Stereochemistries of 2. Relative stereochemistries of *syn*-**2a**,[S7] *syn*-**2b**,[S8] and *anti*-**2b**[S11] were determined by comparison with the reported value of NMR. Absolute stereochemistry of *anti*-**2b** was determined by comparison with the optically rotation

reported.[S12] In the case of *syn*-**2b**, hydrolysis of *syn*-**2b** to the triol was carried out under basic conditions for comparison with the reported value of optically rotation of the triol.[S9] The carbonate products, *syn*-**2c** and *anti*-**2c**, were converted to the triacetate products, *syn*-**2a** and *syn*-**2b**, respectively, and then their relative and absolute configurations were assigned. Absolute stereochemistries of **2f**, **2h**, and **2j** were determined as follows: Authentic sample of (*R*)-**2f** was prepared from commercially available (*R*)-(–)-phenylethan-1,2-diol. Optically rotation of **2h** was compared with the reported value.[S14] The product **2j** was hydrolyzed to the corresponding diol, and optically rotation of the diol was compared with the reported value.[S16] Assignment of stereochemistries of the other products **2** rests on similarity of the spectral data and the reasonable assumption that the oxidation with hypervalent iodine reagent should follow the same stereochemical course.

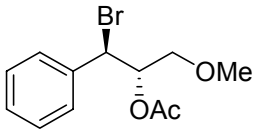
(2*R,3a*R**,8a*S**)-2-(1'-(Methoxycarbonyl)-1'-methylethyl)-2-methyl-8,8a-dihydro-3a*H*-indeno[1,2-*d*][1,3]-dioxole (7)**:





(Diacetoxyiodo)benzene (144 mg, 0.45 mmol) and indene (50 μL, 0.43 mmol) were dissolved in dichloromethane (4 mL) in the presence of acetic acid (30 μL). The solution was cooled at –80 °C using a low temperature bath with magnetic stirrer (EYELA, PSL-1800). Boron trifluoride diethyl etherate (0.1 mL) was added to the solution at –80 °C. The solution was warmed up to –40 °C over 1 h. Dimethylketene methyl trimethylsilyl acetal (90 μL, 0.44 mmol) was added to the solution at the temperature, and then the mixture was allowed to warm up to room temperature over 1 h. The reaction mixture was quenched by addition of water, and extracted with dichloromethane. The organic phase was dried with $MgSO_4$ and concentrated in vacuo. The crude mixture was purified by column chromatography (SiO_2, eluent: 20% ethyl acetate in hexane) to give the titled product **7** (61.5 mg, 0.223 mmol, 52% yield). Selected data for **7**: δ_H(600 MHz; $CDCl_3$) 7.46 (1H, d, *J* 7.6), 7.28–7.19 (3H, m), 5.64 (1H, d, *J* 5.5, 3a-*H*), 4.88 (1H, t, *J* 5.5, 8a-*H*), 3.69 (3H, s), 3.17 (1H, d, *J* 17.2), 3.08 (1H, dd, *J* 17.2 and 5.5), 1.25 (6H, s, 1'-*Me*) and 0.97 (3H, s, 2-*Me*); δ_C(150 MHz; $CDCl_3$) 175.88, 141.80, 140.69, 128.95, 127.27, 125.75, 125.45, 114.38, 85.36, 81.52, 51.99, 51.82, 38.57, 23.86 (2-*Me*), 21.45 (1'-*Me*) and 21.32 (1'-*Me*); *m/z* (ESI+) 299.1280 (M + Na. $C_{16}H_{20}O_4Na$ requires 299.1259). Relative configuration of **7** was determined by NOESY spectrum, where nOe was observed as illustrated. Assignments of NMR signals were carried out using HMQC and HMBC spectra.

(1*R*,2*S*)-2-Acetoxy-1-bromo-3-methoxy-1-phenylpropane (**8**):



Substrate **1a** (53 mg, 0.36 mmol) and **3** (175 mg, 0.42 mmol) and were dissolved in dichloromethane (4 mL) in the presence of acetic acid (20 μL). The solution was cooled at –80 °C using a low temperature bath with magnetic stirrer (EYELA, PSL-1800). Boron trifluoride diethyl etherate (0.10 mL, 0.79 mmol) was added to the solution at –80 °C. The solution was warmed up to –40 °C over 1 h. Trimethylsilyl bromide (0.10 mL, 0.76 mmol) was added to the solution at the temperature, and then the mixture was allowed to warm up to room temperature over 1 h. The reaction mixture was quenched by addition of water, and extracted with dichloromethane. The organic phase was dried with $MgSO_4$ and concentrated in vacuo. The crude mixture was purified by column chromatography (SiO_2, eluent: 10% ethyl acetate in hexane) to give the titled product **8** (55 mg, 0.19 mmol, 54% yield). δ_H(600 MHz; $CDCl_3$) 7.39 (2H, d, *J* 7.6), 7.30 (2H, t, *J* 7.6), 7.26 (1H, t, *J* 7.6), 5.54 (1H, m), 5.16 (1H, d, *J* 7.6), 3.82 (1H, dd, *J* 11.0 and 4.8), 3.58 (1H, dd, *J* 11.0 and 2.7), 3.38 (3H, s) and 1.84 (3H, s); δ_C(150 MHz; $CDCl_3$) 169.68, 137.89, 128.63, 128.46, 128.44, 74.35, 71.46, 59.37, 50.40 and 20.59; $[\alpha]_D^{20}$ –73.6 (*c* 0.64 in $CHCl_3$) for 88% ee of (1*R*,2*S*)-**8**; *m/z* (ESI+) 309.0090 (M + Na. $C_{12}H_{15}O_3{}^{79}BrNa$ requires 309.0102). Retention times of (1*S*,2*R*)-**8** and (1*R*,2*S*)-**8** were 12 and 13 min, respectively, for HPLC analyses using a chiral column Chiralpak IA (0.46 cm ϕ × 25 cm) (eluent: CH_2Cl_2/hexane = 10/90, the flow rate = 0.7 mL/min).

Stereochemistry of **8** was determined as follows. A sample of **8** with 88% ee (30 mg, 0.1 mmol) was hydrolyzed in aqueous tetrahydrofuran solution containing $NaHCO_3$ under reflux. Extraction with ether gave a mixture containing alcohols, which were employed for acetylation to give *syn*-**2a** with 88% ee of (1*S*,2*S*)-**2a** (18.5 mg, 0.070 mmol, 67% yield). No anti-**2a** was detected. The hydrolysis may proceed via a 1,3-dioxolan-2-yl cation, which reacts with water at the 2-position to give the mixture of alcohols.

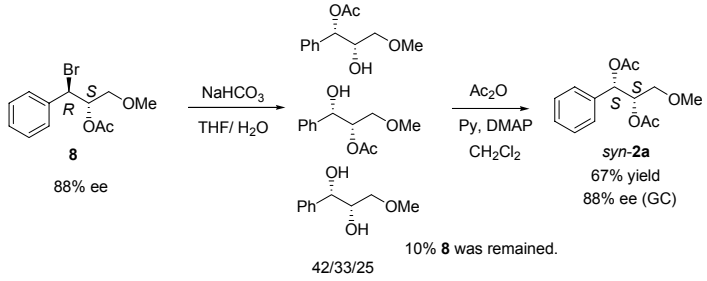
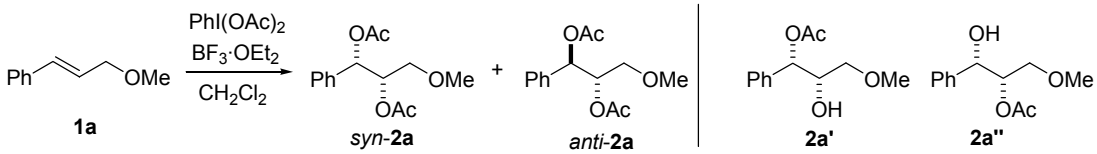


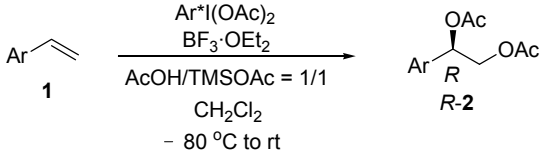
Table S2 Optimization of reaction conditions for diastereoselective formation of dioxyacetylation of **1a** with (diacetoxyiodo)benzene[a]



Entry	Conditions[b]	additive[c]	Yield of **2a** (%)	*syn*-**2a**/*anti*-**2a**
1	A	AcOH	58[d]	>98/2
2	A	AcOH+TMSOAc	57[d]	>98/2
3	B	AcOH	52	19/81
4	B	TMSOAc	48	11/89
5	B	AcOH+TMSOAc	68	<2/98
6	B	AcOH+TMSOAc[e]	57	<2/98
7	C	AcOH	40	20/80
8	C	AcOH+TMSOAc	70	<2/98

[a] **1a** = 0.25 mmol, $PhI(OAc)_2$ = 0.3 mmol, $BF_3{\cdot}OEt_2$ = 0.5 mmol in CH_2Cl_2 (4 mL). [b] Conditions A: Reaction was started at –80 °C, and terminated at –40 °C. The crude products obtained were treated with acetic anhydride and pyridine. Conditions B: Reaction was started at –80 °C, and terminated at rt. Conditions C: Reaction was carried out at 0 °C. [c] In the presence of acetic acid (0.2 mL) and/or trimethylsilyl acetate (0.2 mL). [d] Isolated yield of **2a** after acetylation of the crude products, which contained **2a'** and **2a''** in the ratio of 6/4. [e] Reaction was started in the presence of acetic acid, and trimethylsilyl acetate was added at –40 °C.

Table S3 Additional data for asymmetric Prévost reaction of styrenes under the conditions B[a]



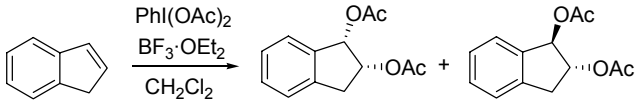
f: Ar = C_6H_5 **g**: Ar = 2-MeC_6H_4

1	Ar*I(OAc)$_2$	Yield (%)	*ee*(%)
1f	**3**	77	52 (*R*)
1f	**4**	79	57 (*R*)
1f	**5**	84	50 (*R*)
1g	**3**	47	11 (*R*)

[a] Reaction was carried out in dichloromethane (4 mL) containing (diacetoxyiodo)arene (0.5 mmol), substrate **1** (0.4 mmol), acetic acid (0.2 mL), trimethylsilyl acetate (0.2 mL), and $BF_3{\cdot}OEt_2$ (0.9 mmol). The reaction was started at –80 °C, and terminated at rt.

Reaction of indene.

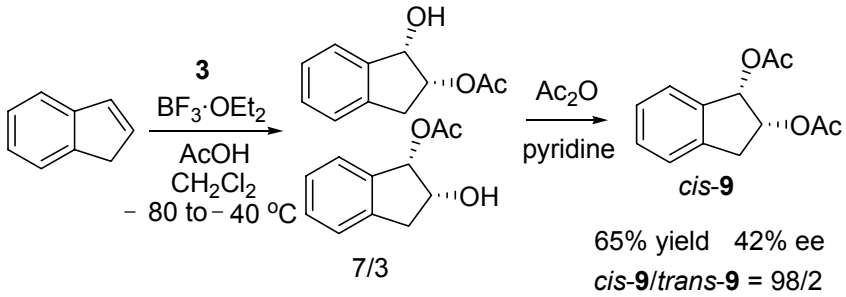
Table S4 Diastereoselective dioxyacetylation of indene

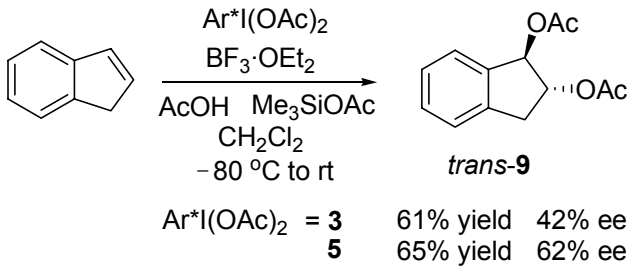


cis-**9** *trans*-**9**

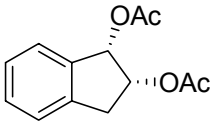
Entry	Conditions[a]	Additive[b]	Yield of **9** (%)	*cis*/*trans*
1	A	AcOH	72[c]	98/2
2	C	AcOH	66	15/85
3	B	AcOH	51	18/82
4	B	AcOH+TMSOAc	64	<2/98

[a] Conditions A: Reaction was started at –80 °C, and quenched at –40 °C. The crude products obtained were treated with acetic anhydride and pyridine. Conditions B: Reaction was started at –80 °C, and quenched at rt. Conditions C: Reaction was carried out at rt. [b] The reaction was carried out in dichloromethane (4 mL) containing acetic acid (0.4 mL) and/or trimethylsilyl acetate (0.4 mL). [c] Isolated yield of **9** after acetylation of the crude products, which contained 7/3 mixture of 2-acetoxy-1-hydroxyindane and 1-acetoxy-2-hydroxyindane.

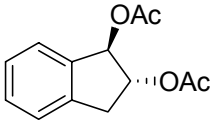




(1*S*,2*R*)-1,2-Diacetoxyindane (*cis*-9): δ_H(600 MHz; $CDCl_3$) 7.38 (1H, d, *J* 7.6), 7.32 (1H, t, *J* 7.6), 7.27–7.24 (2H, m), 6.21 (1H, d, *J* 5.5), 5.53 (1H, q, *J* 5.5), 3.22 (1H, dd, *J* 15.8 and 6.9), 3.12 (1H, dd, *J* 15.8 and 5.5), 2.08 (3H, s) and 2.06 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.35, 140.14, 138.08, 129.54, 127.35, 125.72, 124.92, 74.99, 73.19, 35.84, 20.80 and 20.75; The NMR data agree well with the reported value.[S17] Retention times of (1*S*,2*R*)-**9** and (1*R*,2*S*)-**9** were 15.5 and 16.0 min, when temperature of the column (Chirasil-DEX-CB) was maintained at 160 °C. $[\alpha]_D^{20}$ +23 (*c* 0.92 in $CHCl_3$) for 42% ee of (1*S*,2*R*)-**9** (1% of *trans*-**9** was contained.) Lit.[S18] ($[\alpha]_D$ +69 (*c* 1.0 in $CHCl_3$) for (1*S*,2*R*)-**9**)



(1*R*,2*R*)-1,2-Diacetoxyindane (*trans*-9): δ_H(600 MHz; $CDCl_3$) 7.34 (1H, d, *J* 7.6), 7.31 (1H, t, *J* 7.6), 7.26–7.21 (2H, m), 6.23 (1H, d, *J* 3.4), 5.43 (1H, m), 3.50 (1H, dd, *J* 16.5 and 6.9), 2.88 (1H, dd, *J* 16.5 and 4.8), 2.09 (3H, s) and 2.06 (3H, s); δ_C(150 MHz; $CDCl_3$) 170.63, 170.59, 140.61, 138.33, 129.51, 127.43, 125.65, 124.92, 80.74, 78.79, 36.85, 21.06 and 21.04. The NMR data agree well with the reported value.[S17c] $[\alpha]_D^{20}$ –36 (*c* 0.40 in $CHCl_3$) for 29% ee of (1*R*,2*R*)-**9** (2% of *cis*-isomer was contained.) The sample of 29% ee of (1*R*,2*R*)-**9** was obtained in the reaction in the presence of only acetic acid (without trimethylsily acetate) under the conditions B. The reaction gave an 8:2 mixture of *trans*-**9** and *cis*-**9** in 55% isolated yield. Separation of *trans*-**9** and *cis*-**9** was carried out by SiO_2 column chromatography. Retention times of (1*R*,2*R*)-**9** and (1*S*,2*S*)-**9** were 13.9 and 14.2 min, when temperature of the column (Chirasil-DEX-CB) was maintained at 160 °C. The sample of 29% ee of (1*R*,2*R*)-**9** was hydrolyzed under basic conditions. The diol obtained has negative value of optically rotation ($[\alpha]_D^{20}$ –9 (*c* 0.19 in EtOH)). Lit.[S19] ($[\alpha]_D$ –29.4 (*c* 1.0 in EtOH) for (1*R*,2*R*)-**9**).



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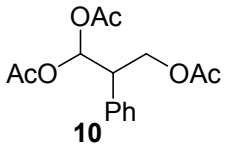

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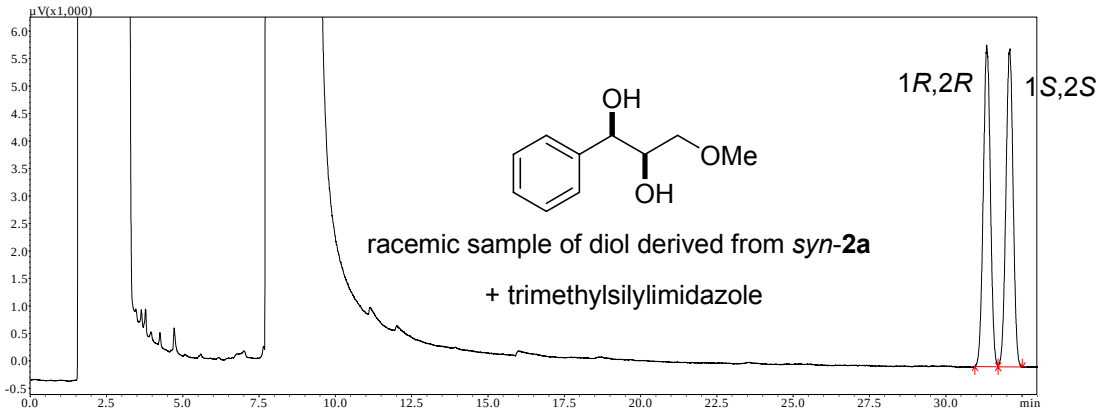
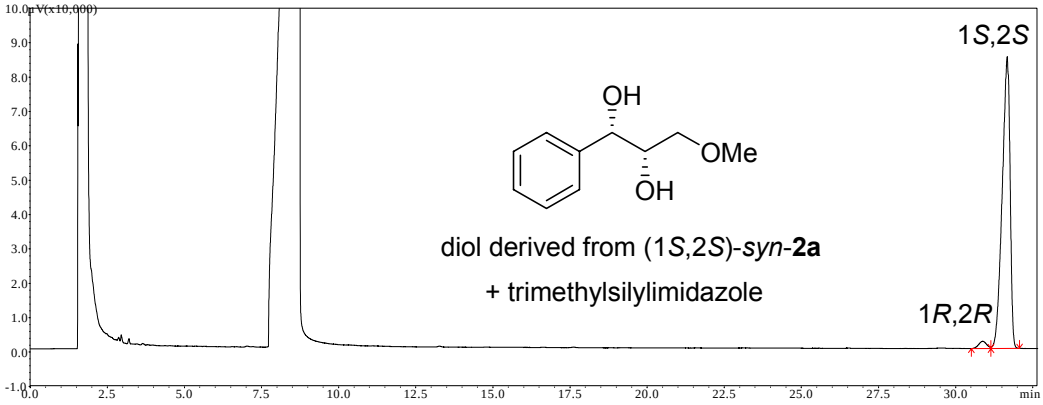
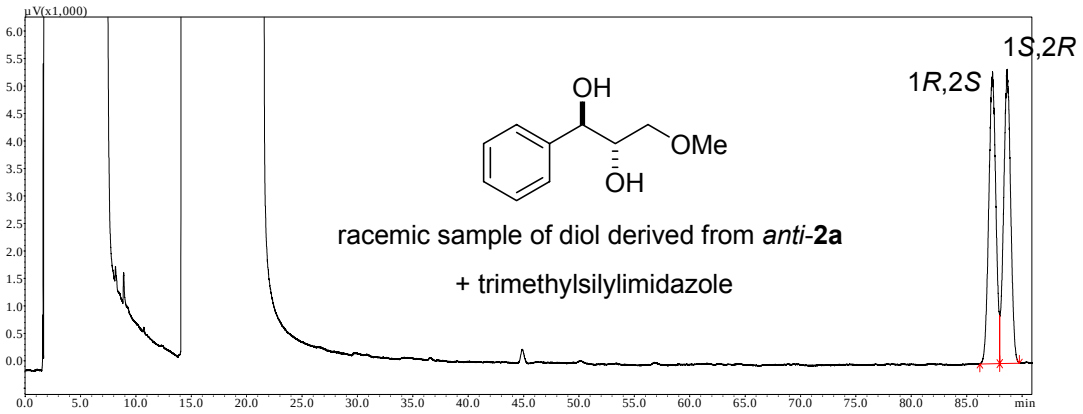


GC analysis of racemic sample of *syn*-**2a** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



GC analysis of optically active *syn*-**2a** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



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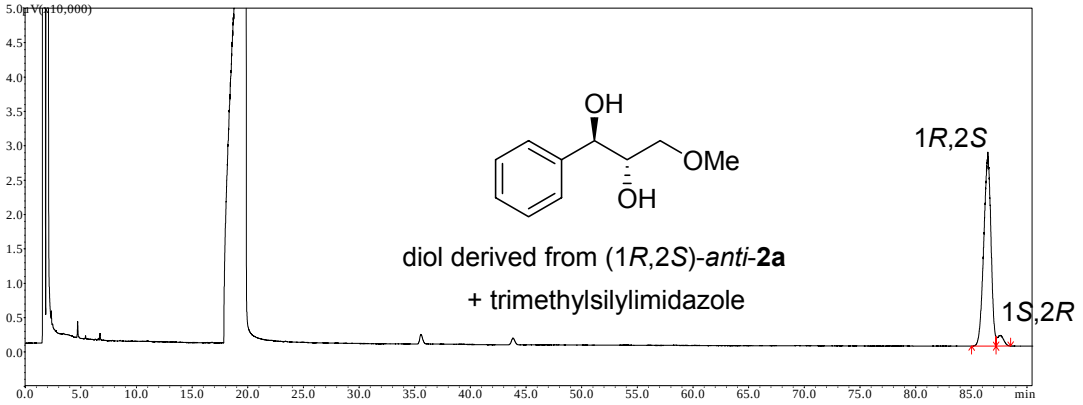
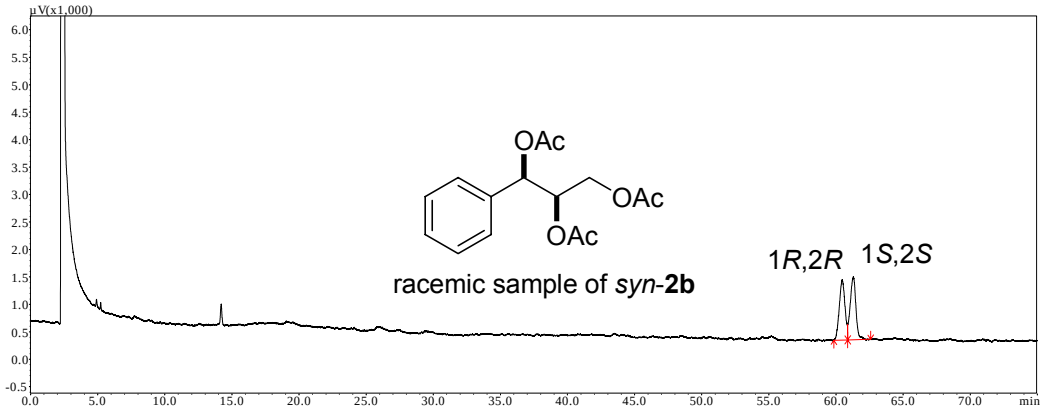
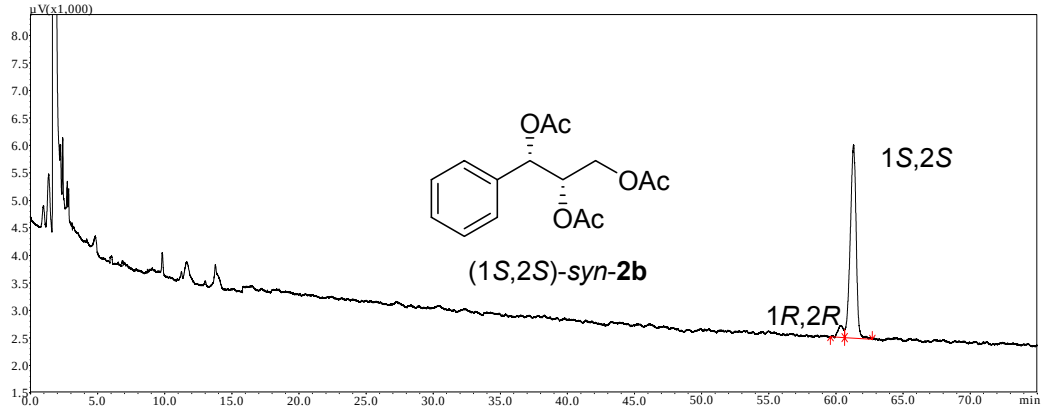


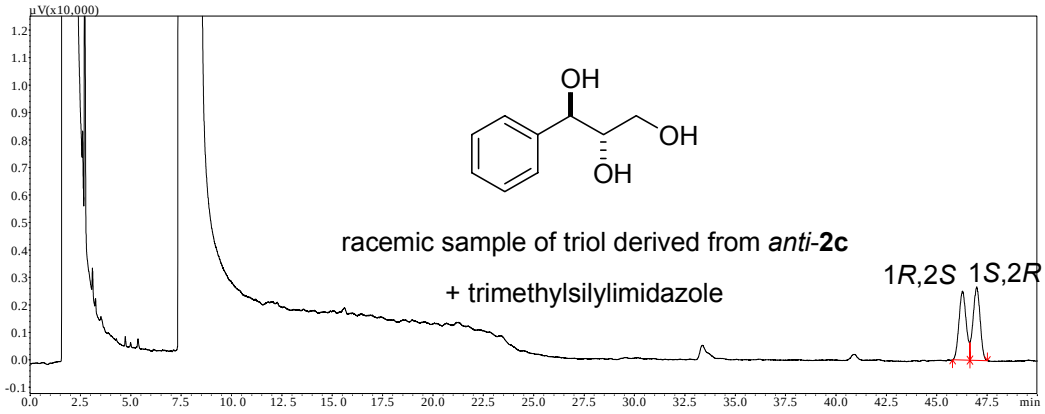
GC analysis of optically active *anti*-**2a** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 100 °C and a linear velocity of 31.7 cm/s of He carrier gas



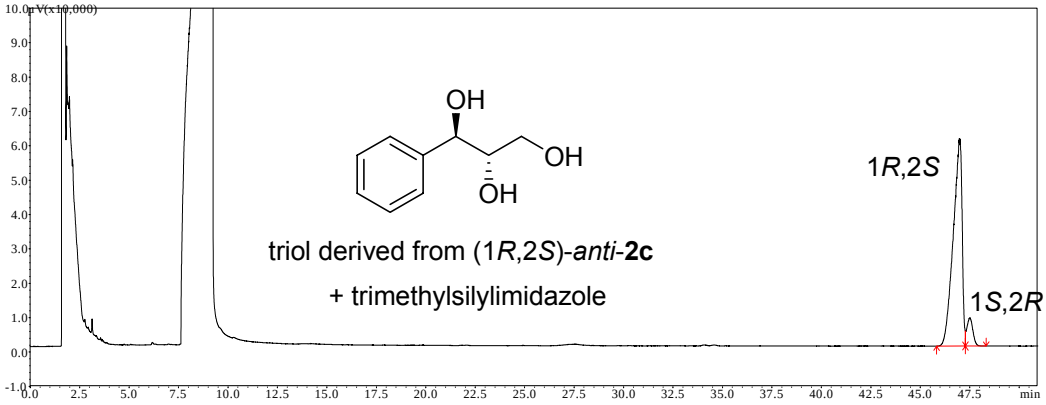
GC trace of racemic sample of *syn*-**2b** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 140 °C and a linear velocity of 30.1 cm/s of He carrier gas



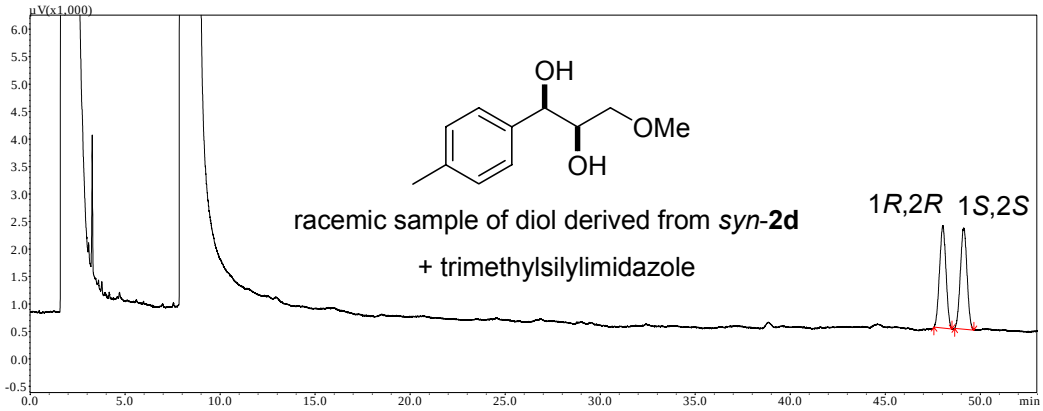
GC trace of optically active *syn*-**2b** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 140 °C and a linear velocity of 30.1 cm/s of He carrier gas



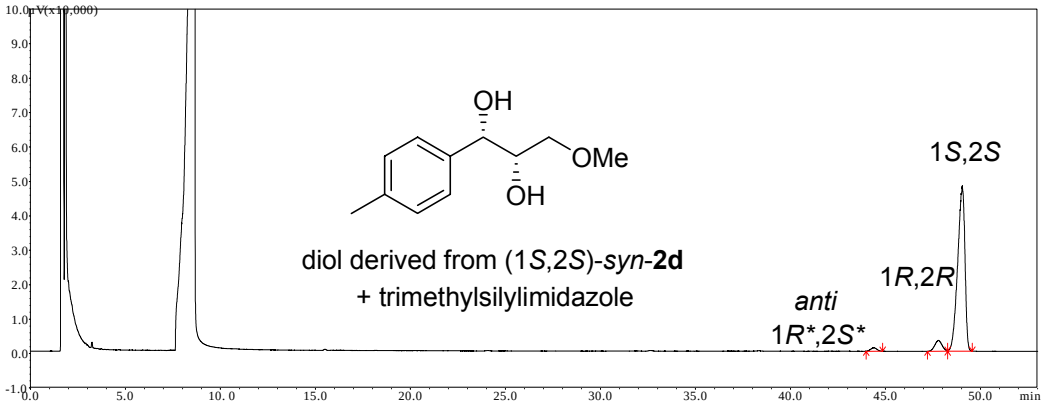
GC analysis of racemic sample of *anti*-**2c** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



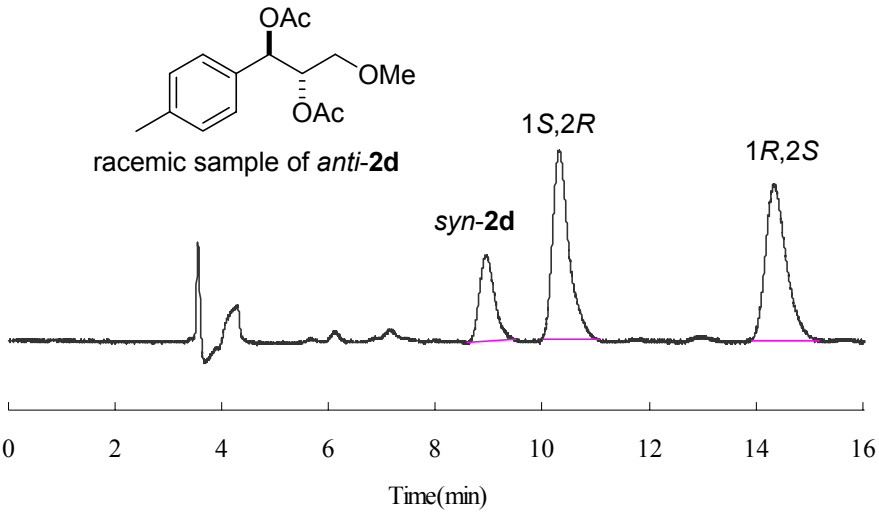
GC analysis of optically active *anti*-**2c** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



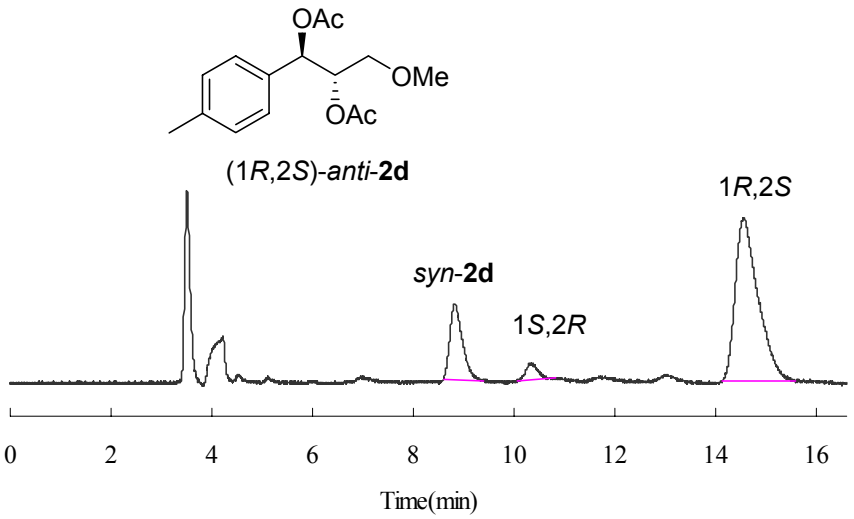
GC analysis of racemic sample of *syn*-**2d** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



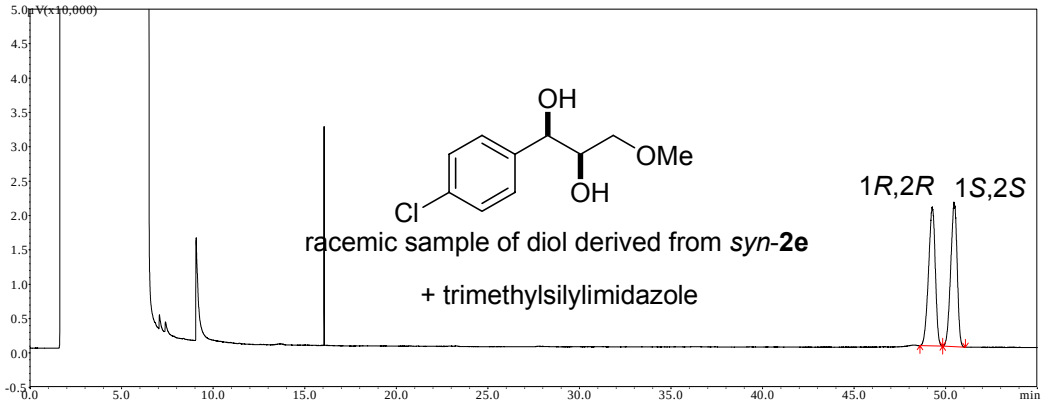
GC analysis of optically active *syn*-**2d** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



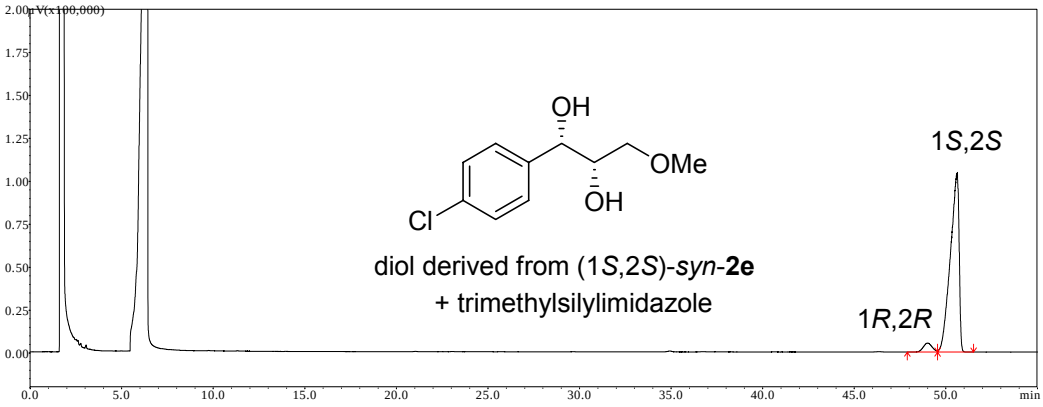
HPLC trace of racemic sample of *anti*-**2d** (Chiralpak AD (0.46 cm ϕ × 25 cm), eluent: hexane/*i*PrOH = 97/3 at a low rate of 1.0 mL/min, detected at 260 nm)



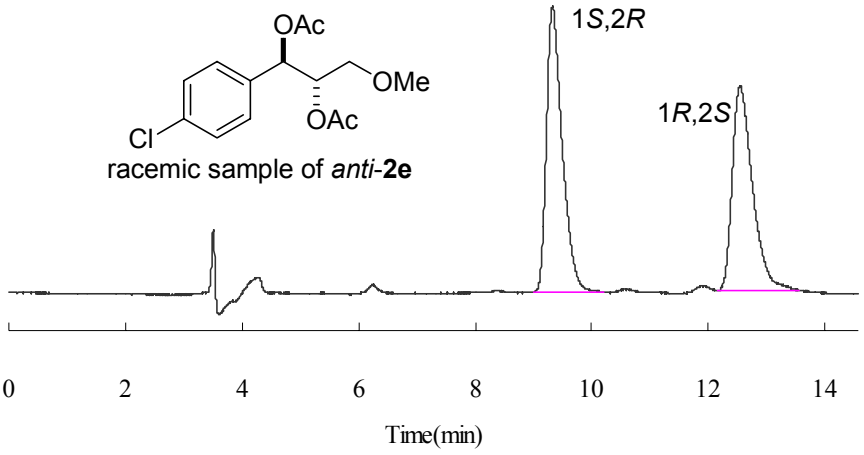
HPLC trace of optically active *anti*-**2d** (Chiralpak AD (0.46 cm ϕ × 25 cm), eluent: hexane/*i*PrOH = 97/3 at a low rate of 1.0 mL/min, detected at 260 nm)



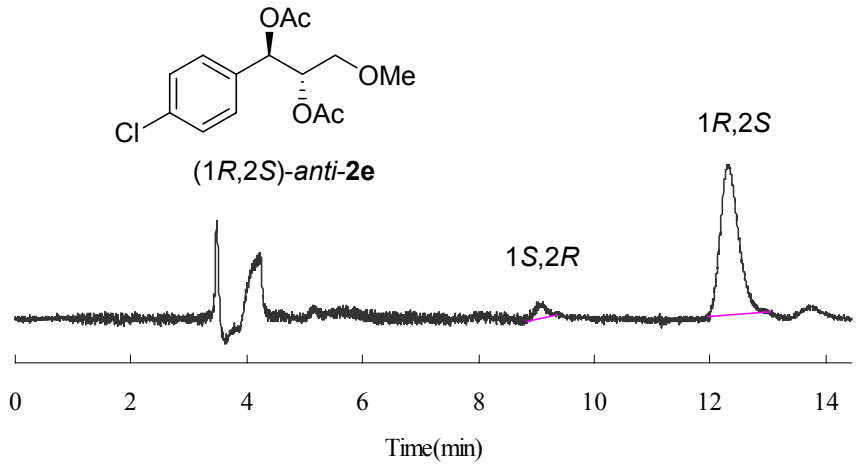
GC analysis of racemic sample of *syn*-**2e** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 130 °C and a linear velocity of 30.6 cm/s of He carrier gas



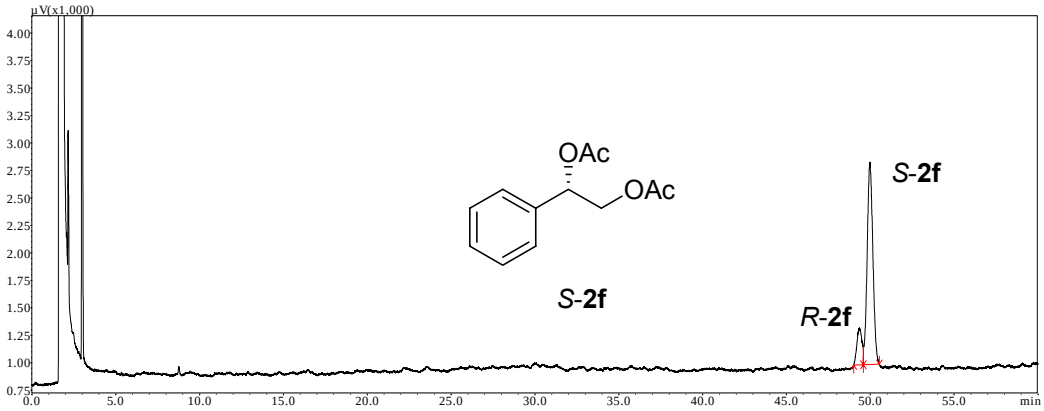
GC analysis of optically active *syn*-**2e** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 130 °C and a linear velocity of 30.6 cm/s of He carrier gas



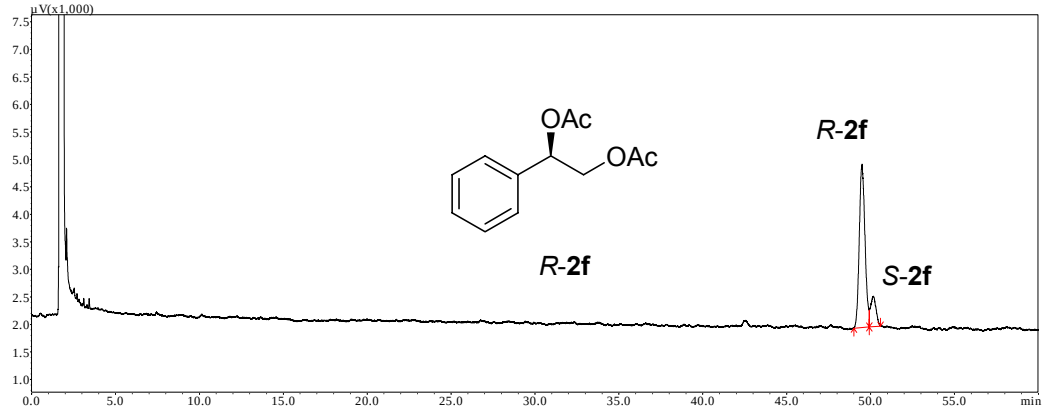
HPLC trace of racemic sample of *anti*-**2e** (Chiralpak AD (0.46 cm ϕ × 25 cm), eluent: hexane/*i*PrOH = 97/3 at a low rate of 1.0 mL/min, detected at 260 nm)



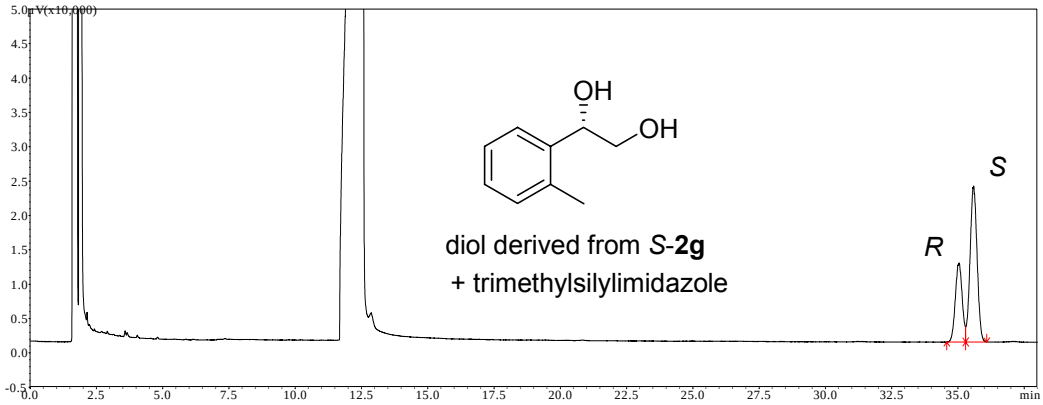
HPLC trace of optically active *anti*-**2e** (Chiralpak AD (0.46 cm ϕ × 25 cm), eluent: hexane/*i*PrOH = 97/3 at a low rate of 1.0 mL/min, detected at 260 nm)



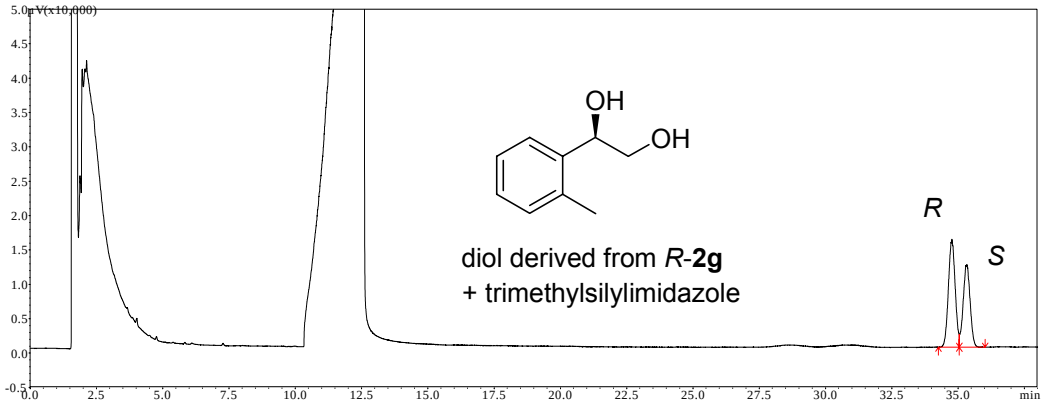
GC trace of optically active *S*-**2f** obtained under the conditions A (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 115 °C and a linear velocity of 31.4 cm/s of He carrier gas



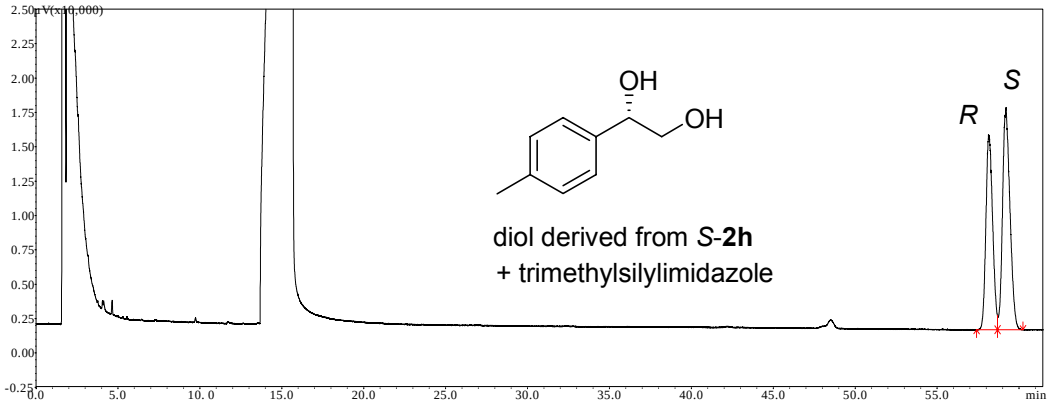
GC trace of optically active *R*-**2f** obtained under the conditions B (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 115 °C and a linear velocity of 31.4 cm/s of He carrier gas



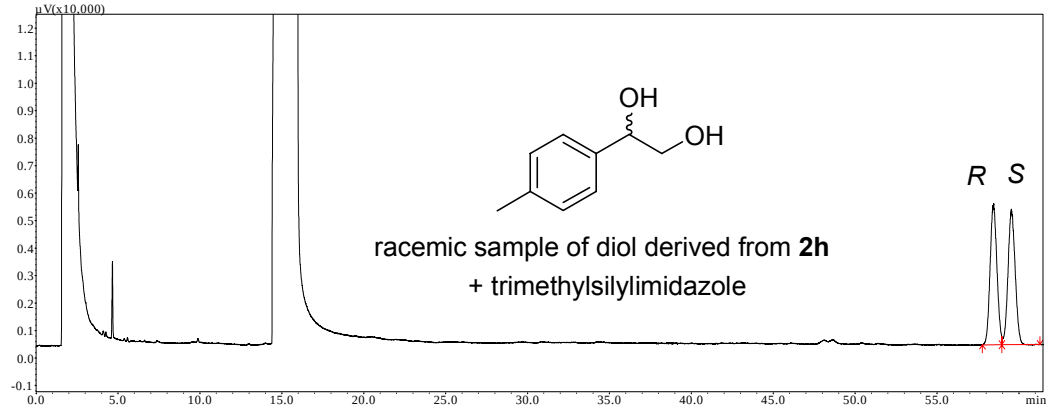
GC analysis of optically active *S*-**2g** obtained under the conditions A (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 110 °C and a linear velocity of 31.7 cm/s of He carrier gas



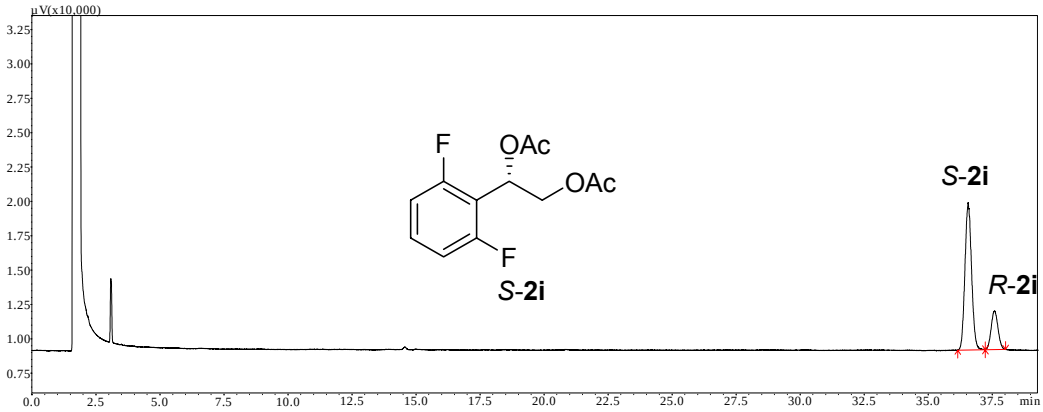
GC analysis of optically active *R*-**2g** obtained under the conditions B (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 110 °C and a linear velocity of 31.7 cm/s of He carrier gas



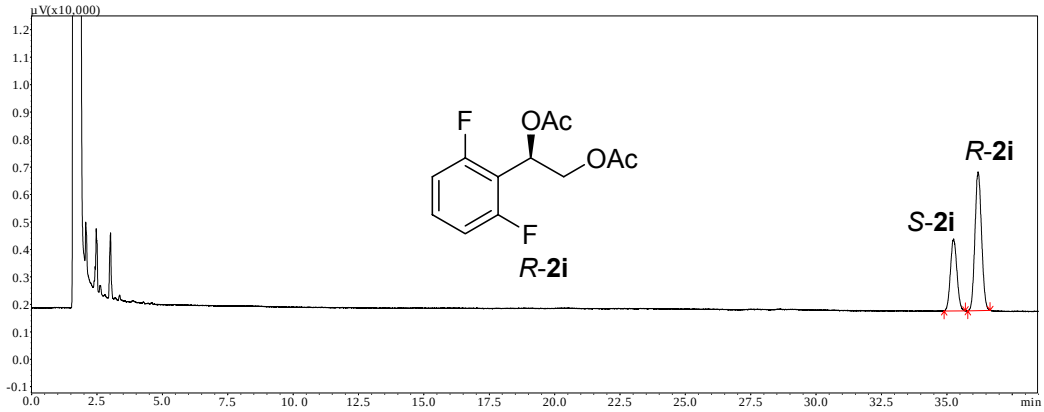
GC analysis of optically active *S*-**2h** obtained under the conditions A (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 105 °C and a linear velocity of 32.0 cm/s of He carrier gas



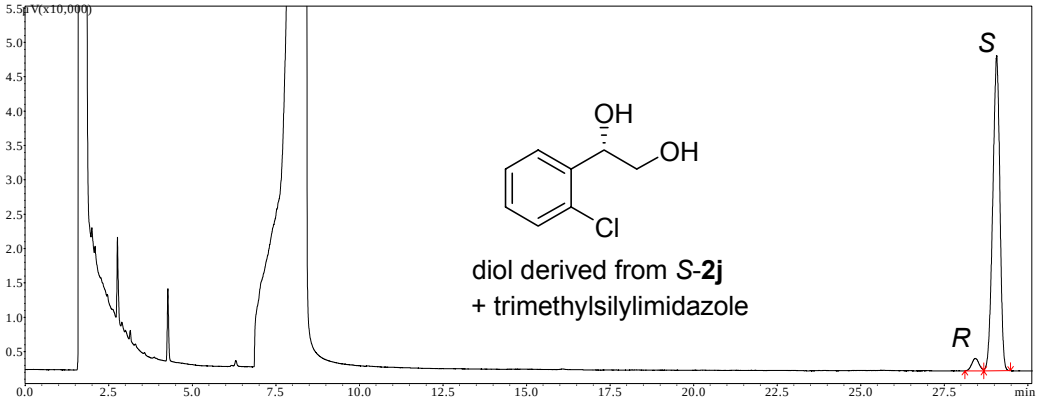
GC analysis of racemic **2h** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 105 °C and a linear velocity of 32.0 cm/s of He carrier gas



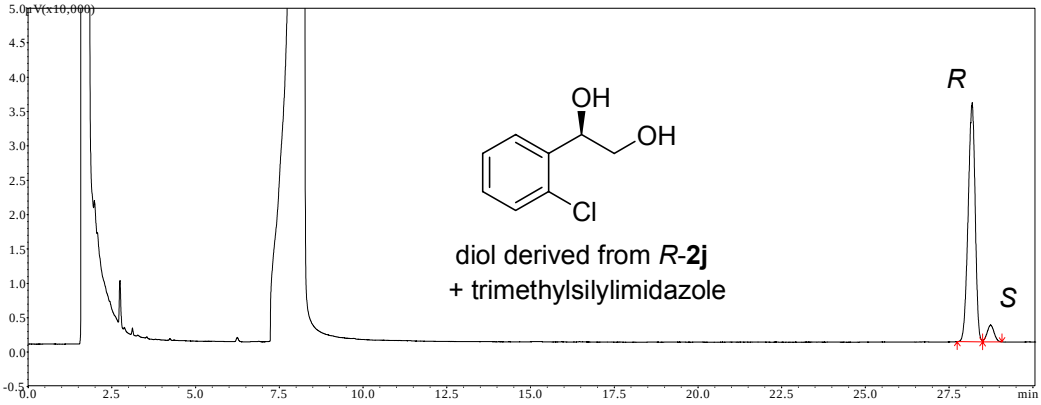
GC trace of optically active **2i** obtained under the conditions A (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



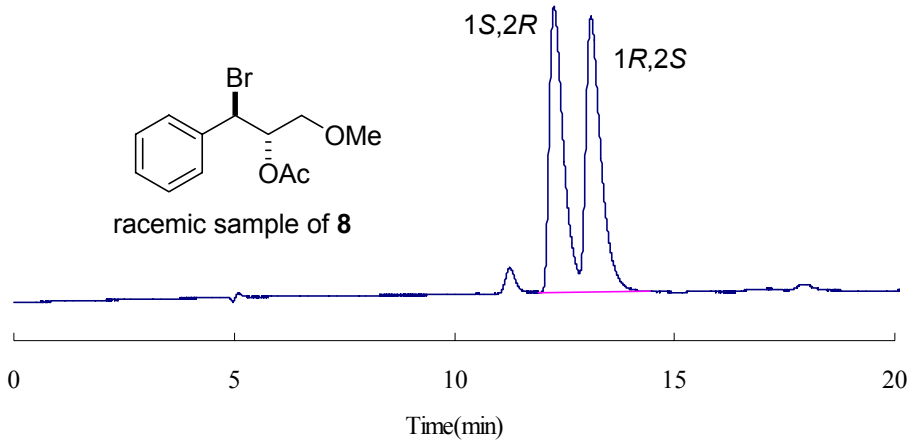
GC trace of optically active **2i** obtained under the conditions B (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



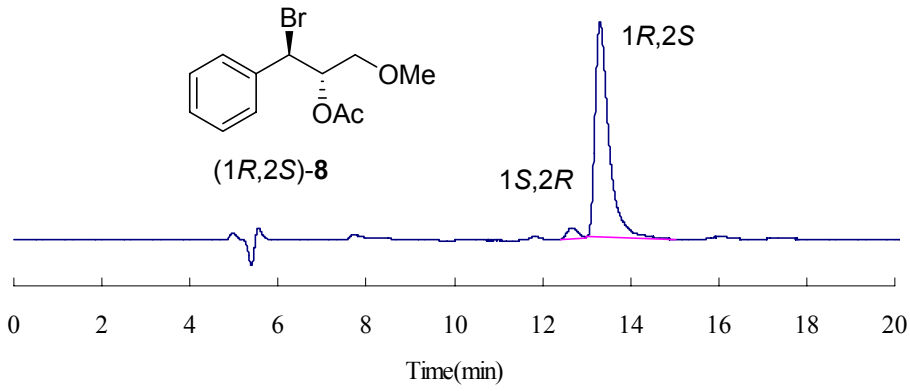
GC analysis of optically active *S*-**2j** obtained under the conditions A (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



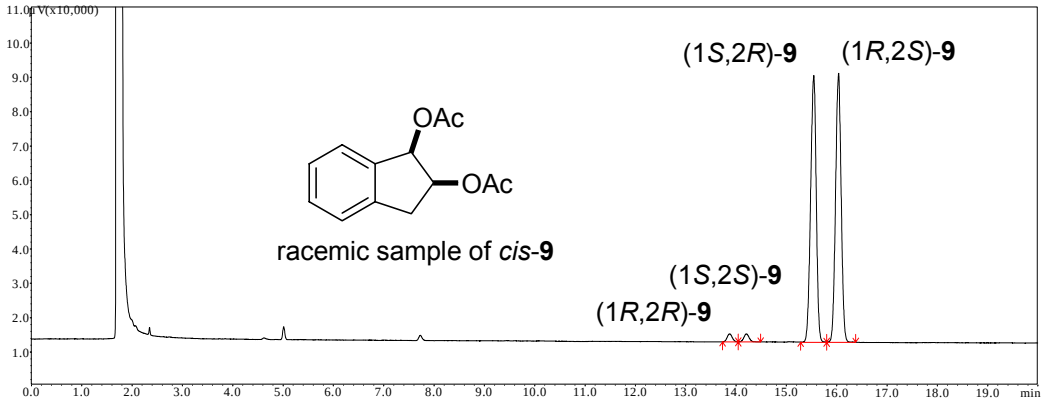
GC analysis of optically active *R*-**2j** obtained under the conditions B (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 120 °C and a linear velocity of 31.2 cm/s of He carrier gas



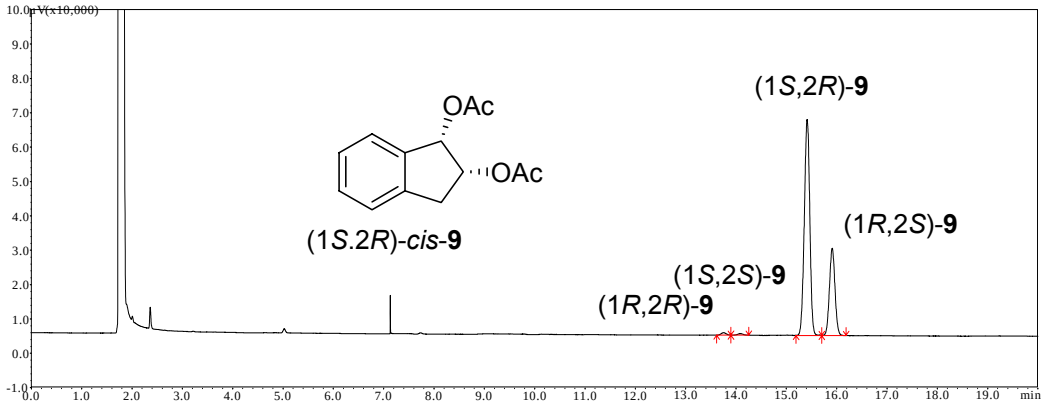
HPLC trace of racemic sample of **8** (Chiralpak IA (0.46 cm ϕ × 25 cm), eluent: hexane/CH_2Cl_2 = 9/1 at a low rate of 0.7 mL/min, detected at 260 nm)



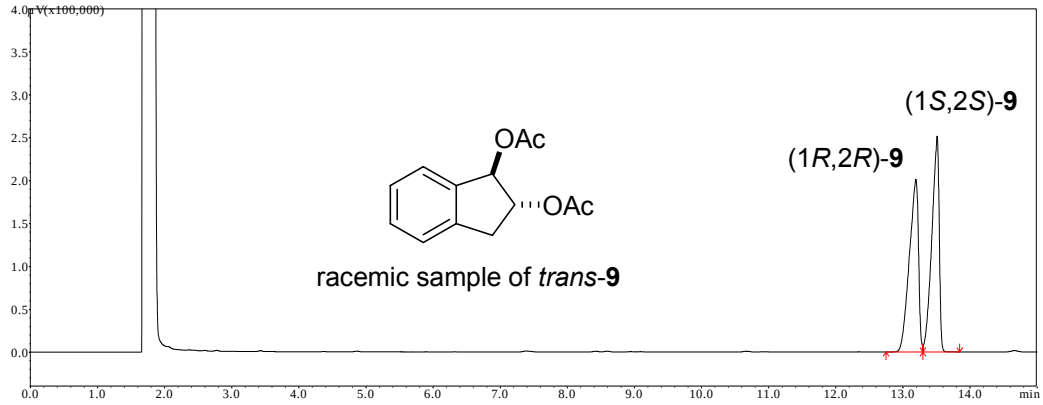
HPLC trace of optically active **8** (Chiralpak IA (0.46 cm ϕ × 25 cm), eluent: hexane/CH_2Cl_2 = 9/1 at a low rate of 0.7 mL/min, detected at 260 nm)



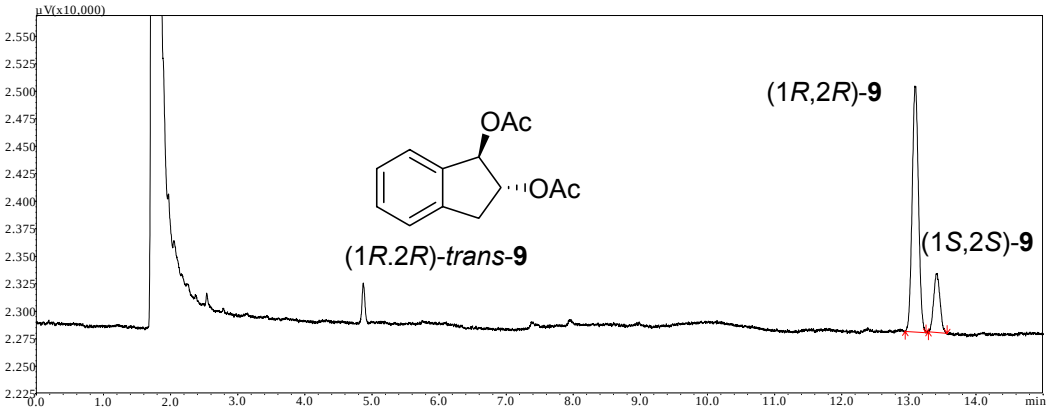
GC trace of racemic sample of *cis*-**9** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 160 °C and a linear velocity of 29.2 cm/s of He carrier gas



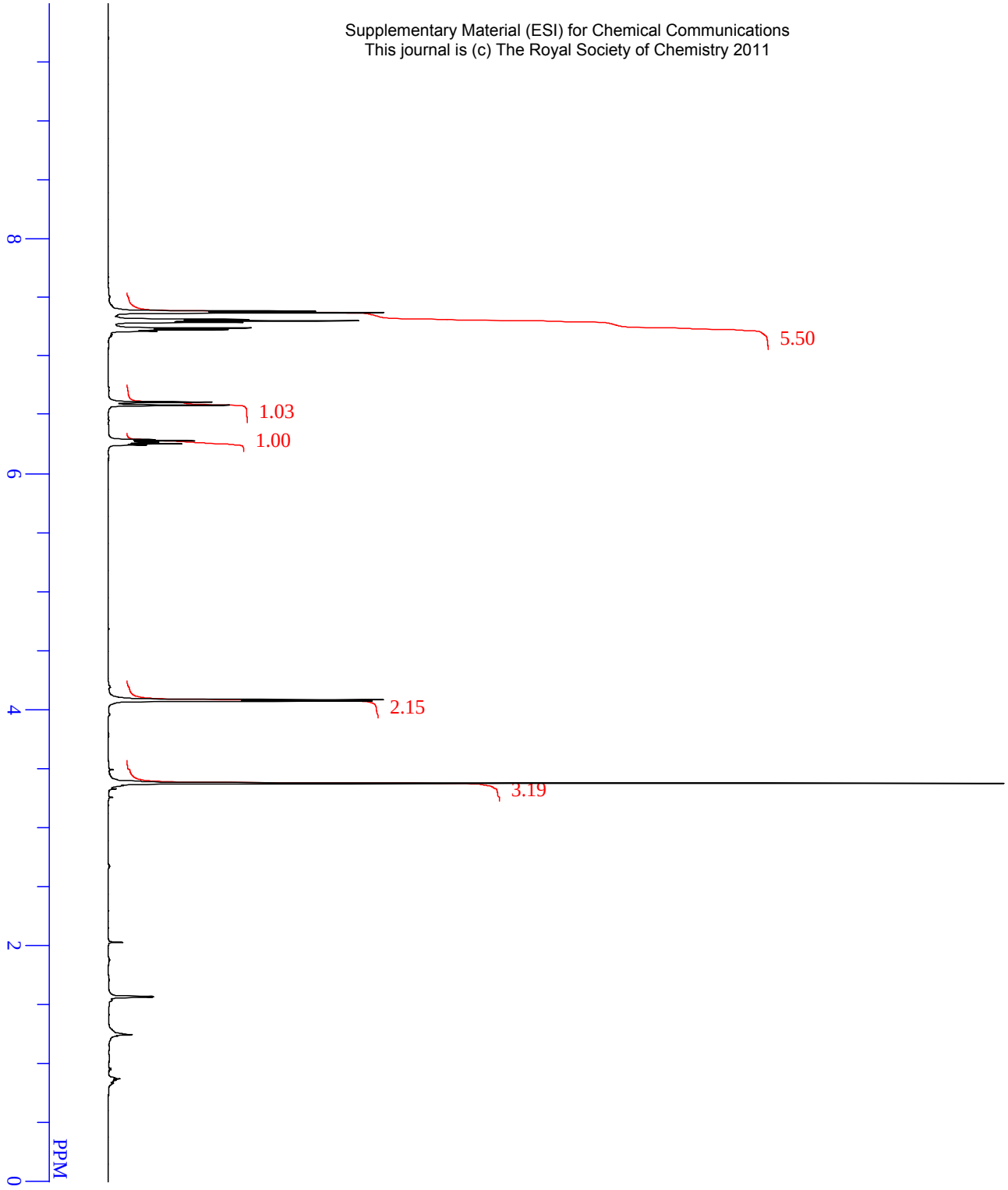
GC trace of optically active *cis*-**9** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 160 °C and a linear velocity of 29.2 cm/s of He carrier gas



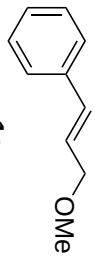
GC trace of racemic sample of *trans*-**9** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 160 °C and a linear velocity of 29.2 cm/s of He carrier gas



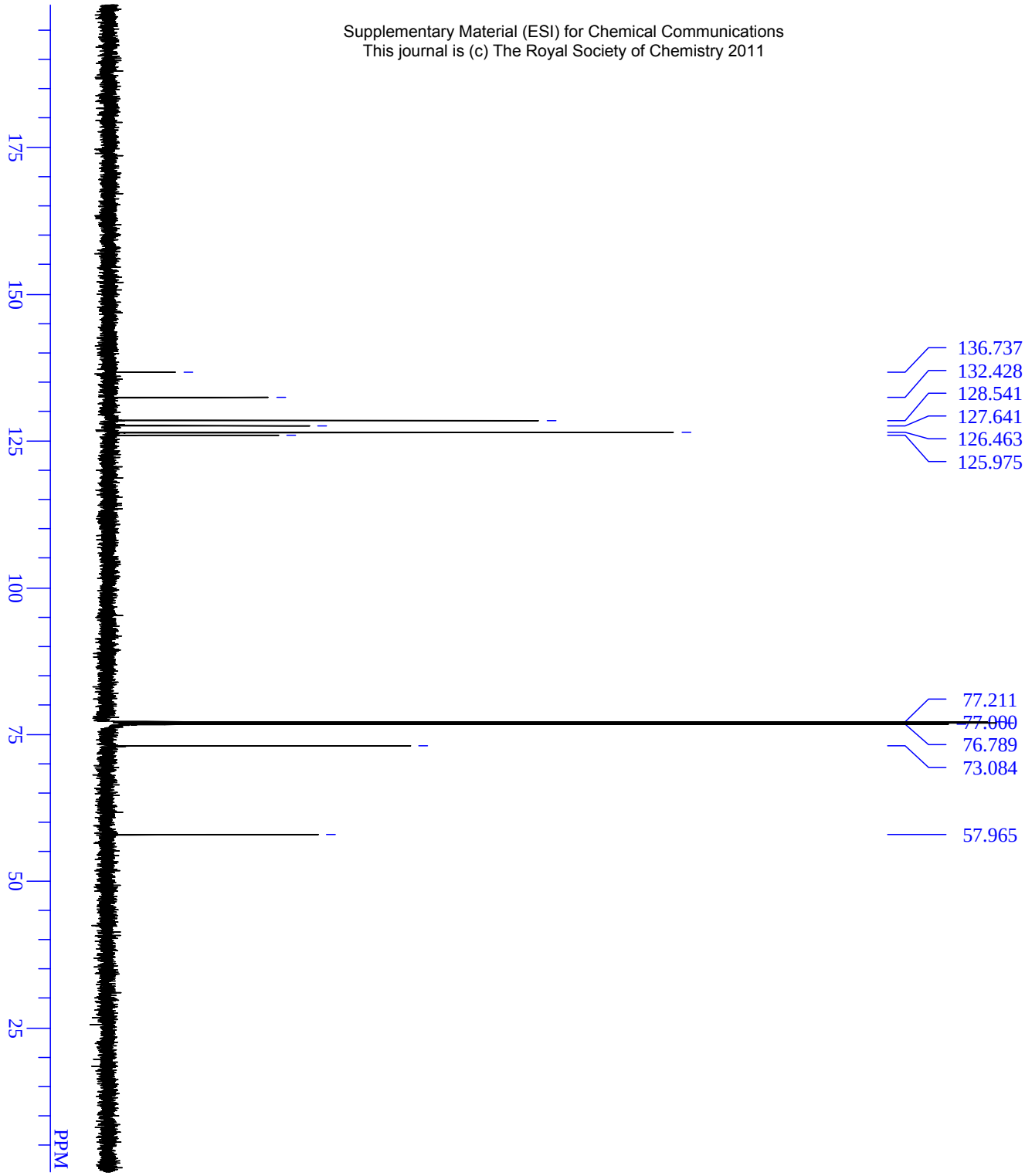
GC trace of optically active *trans*-**9** (DEX-CB, i.d. 0.25 mm × 25 m) at column temperature of 160 °C and a linear velocity of 29.2 cm/s of He carrier gas



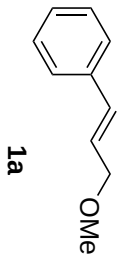
^{1}H NMR (600 MHz, $CDCl_3$)



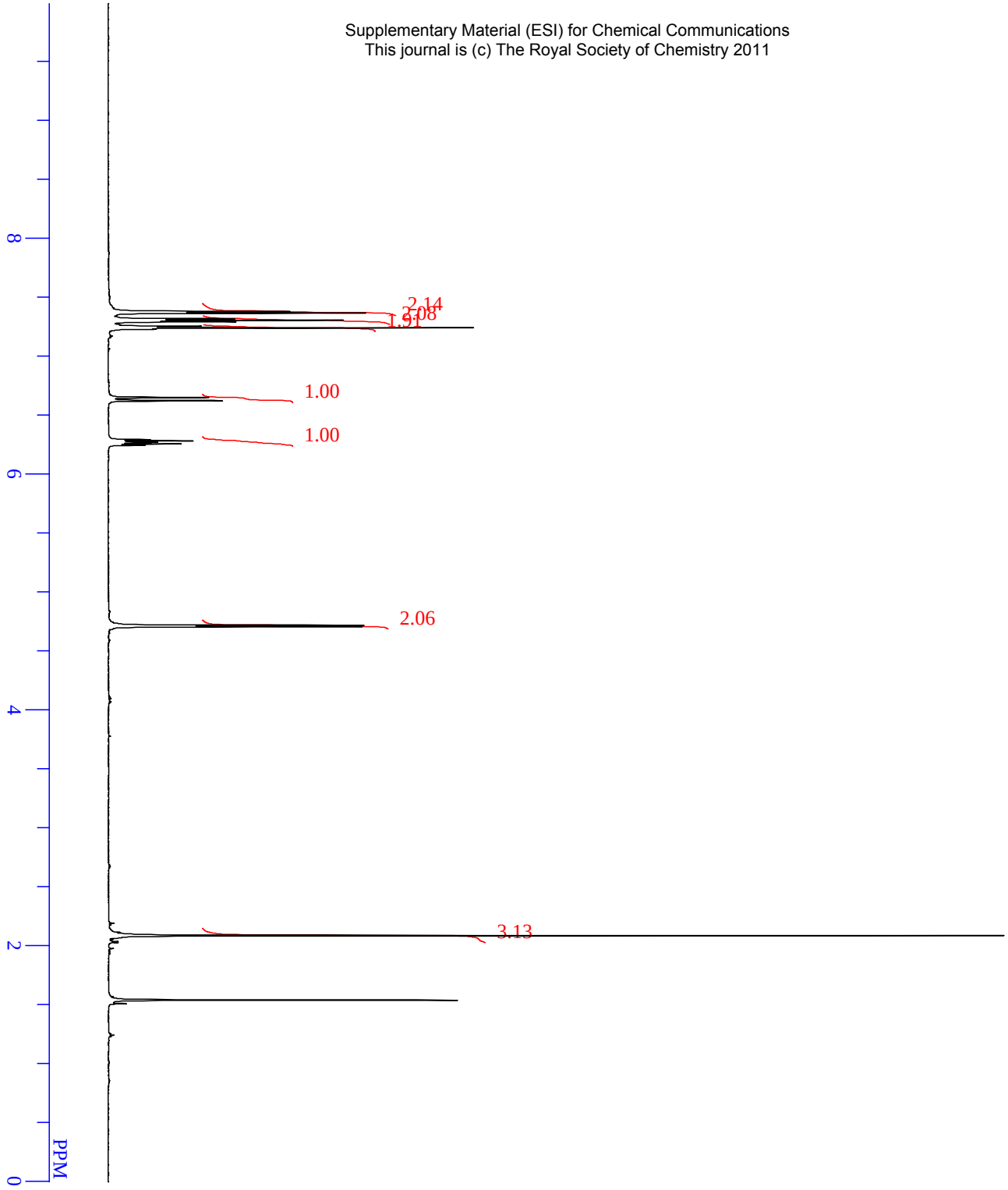
1a



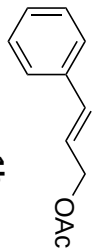
^{13}C NMR (150 MHz, $CDCl_3$)



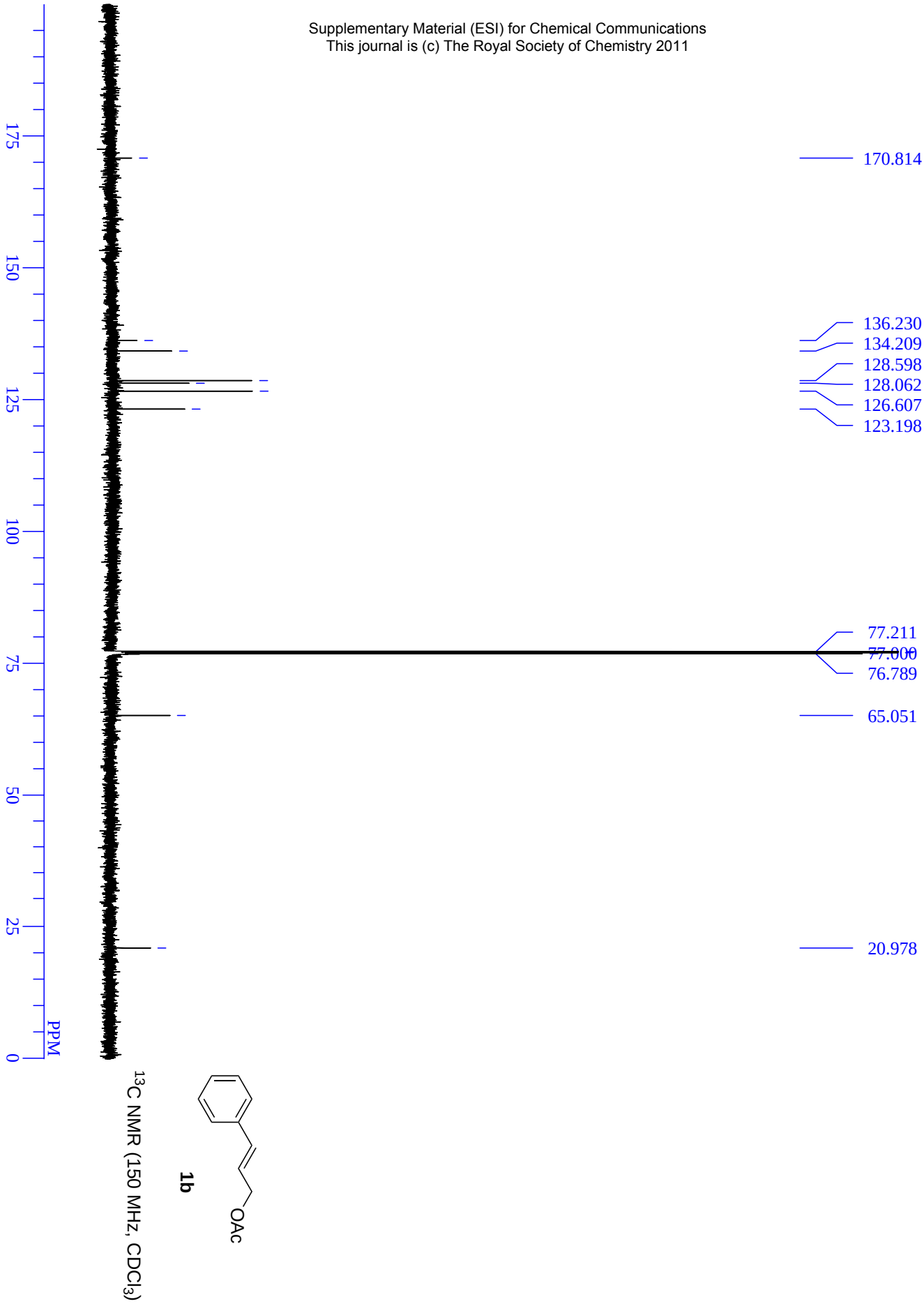
1a

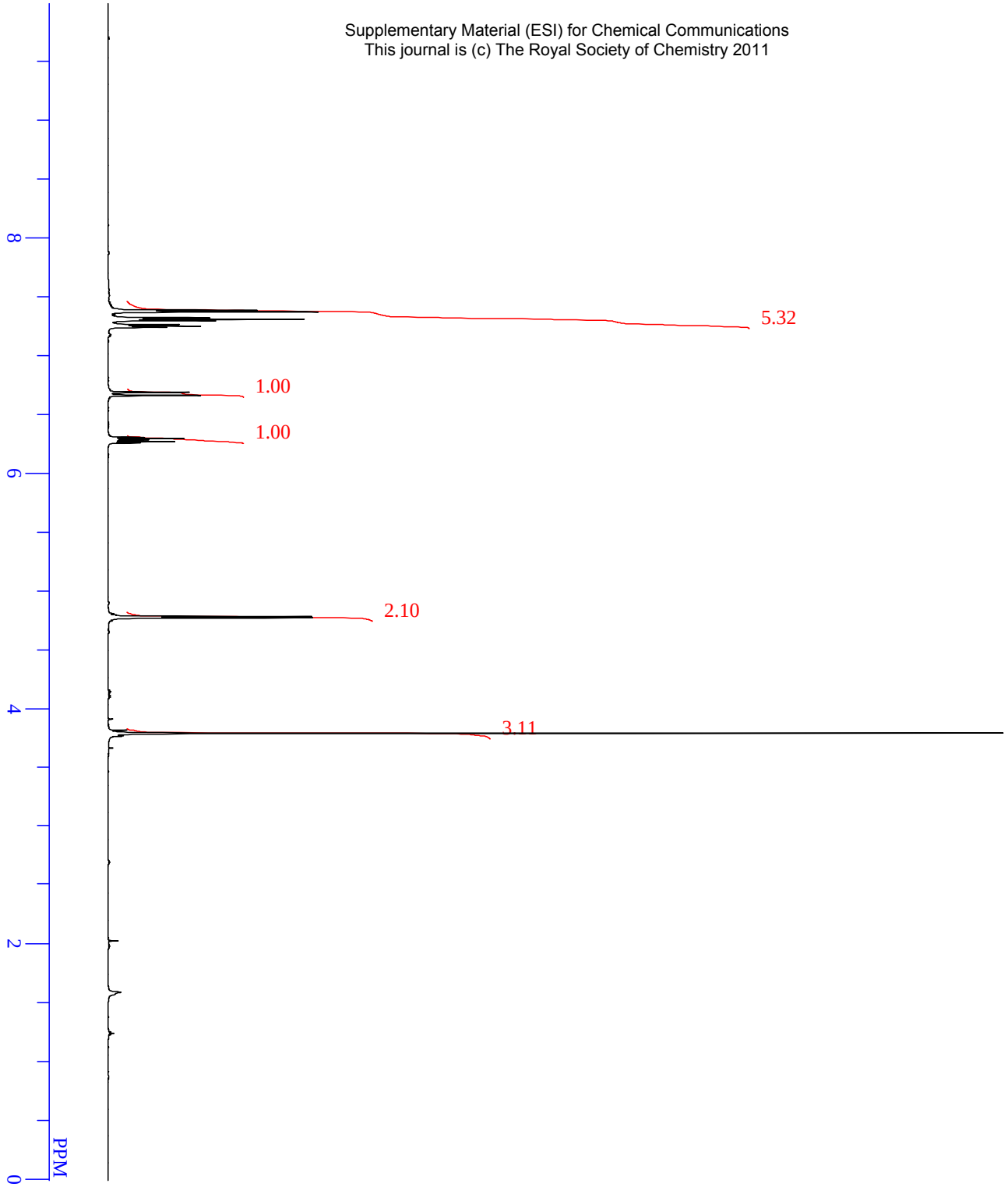


^{1}H NMR (600 MHz, $CDCl_3$)

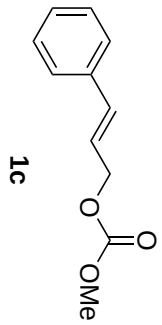


1b

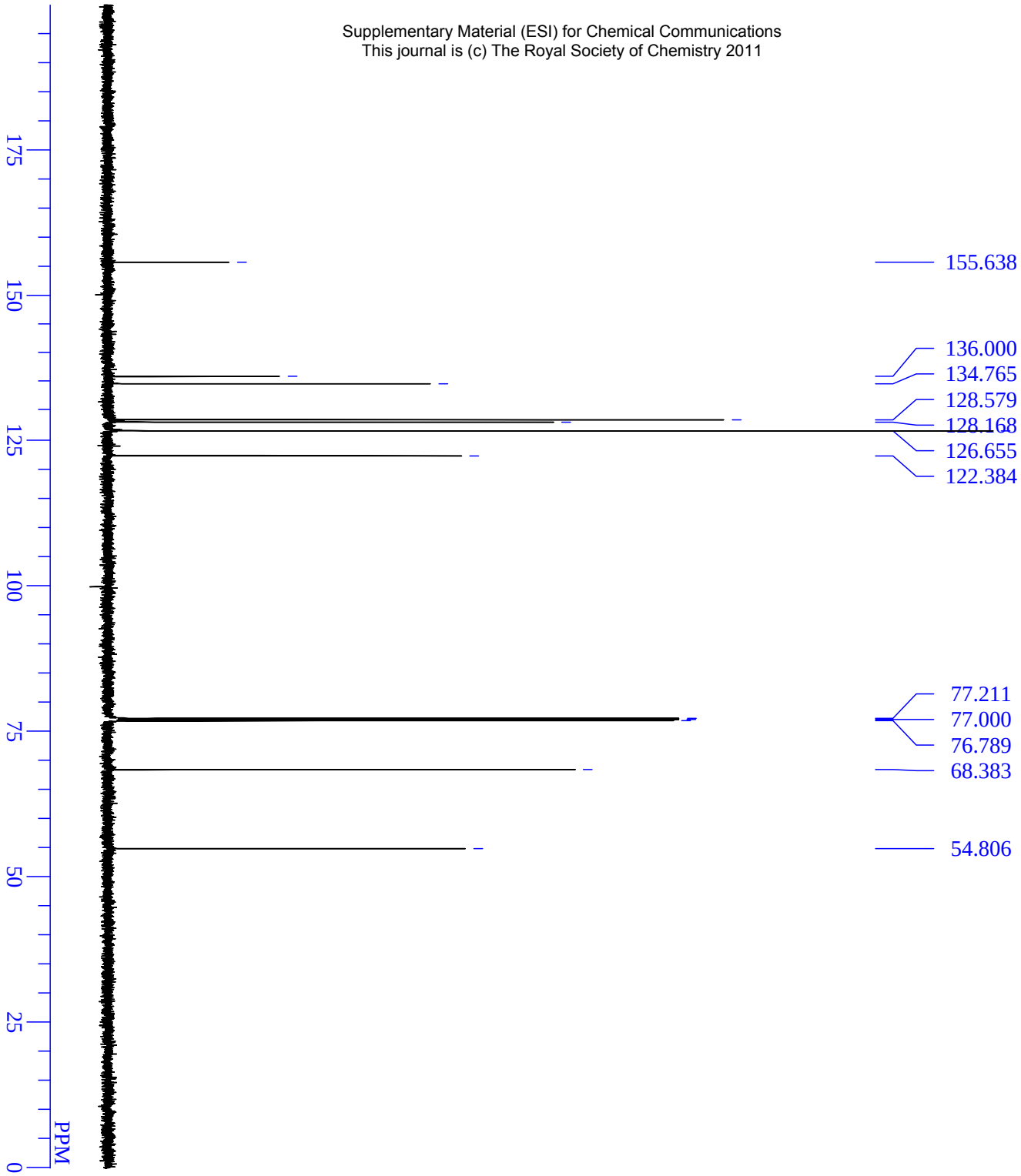

170.814
136.230
134.209
128.598
128.062
126.607
123.198
77.211
77.000
76.789
65.051
20.978
PPM
175
150
125
100
75
50
25
0
^{13}C NMR (150 MHz, CDCl$_3$)
1b
OAc



^{1}H NMR (600 MHz, $CDCl_3$)

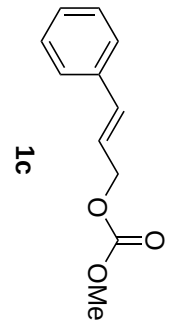


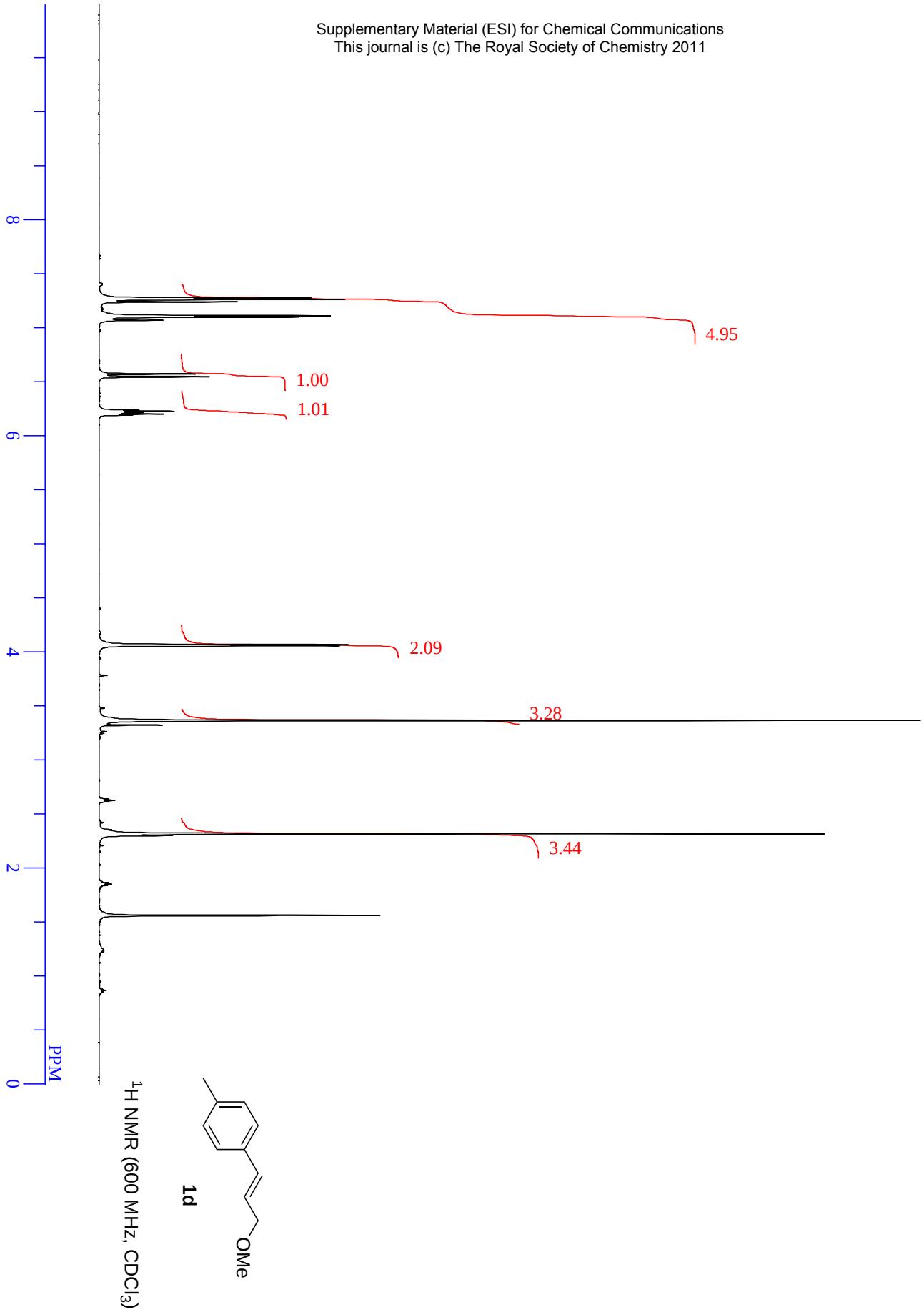
1c



^{13}C NMR (150 MHz, $CDCl_3$)

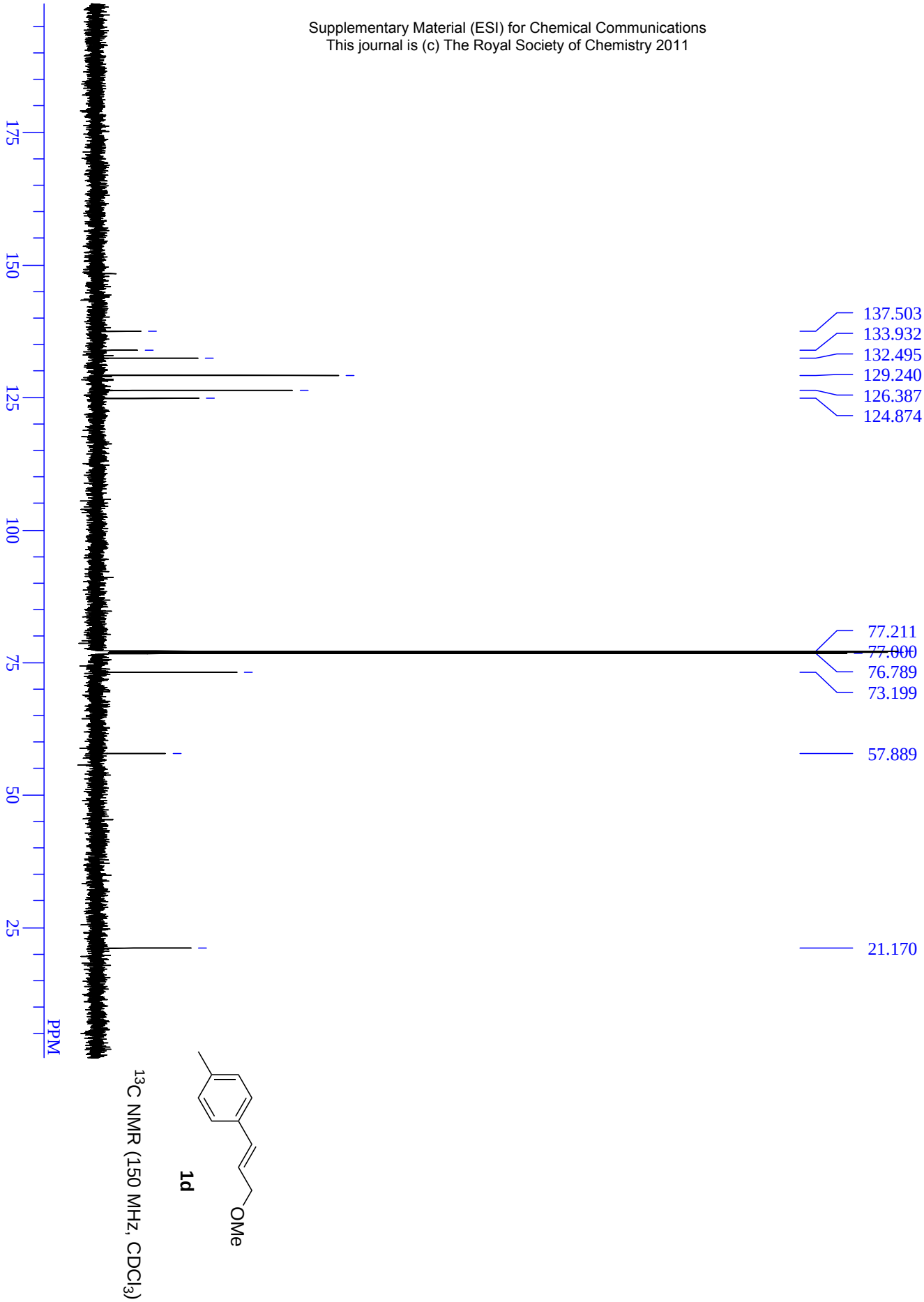
1c

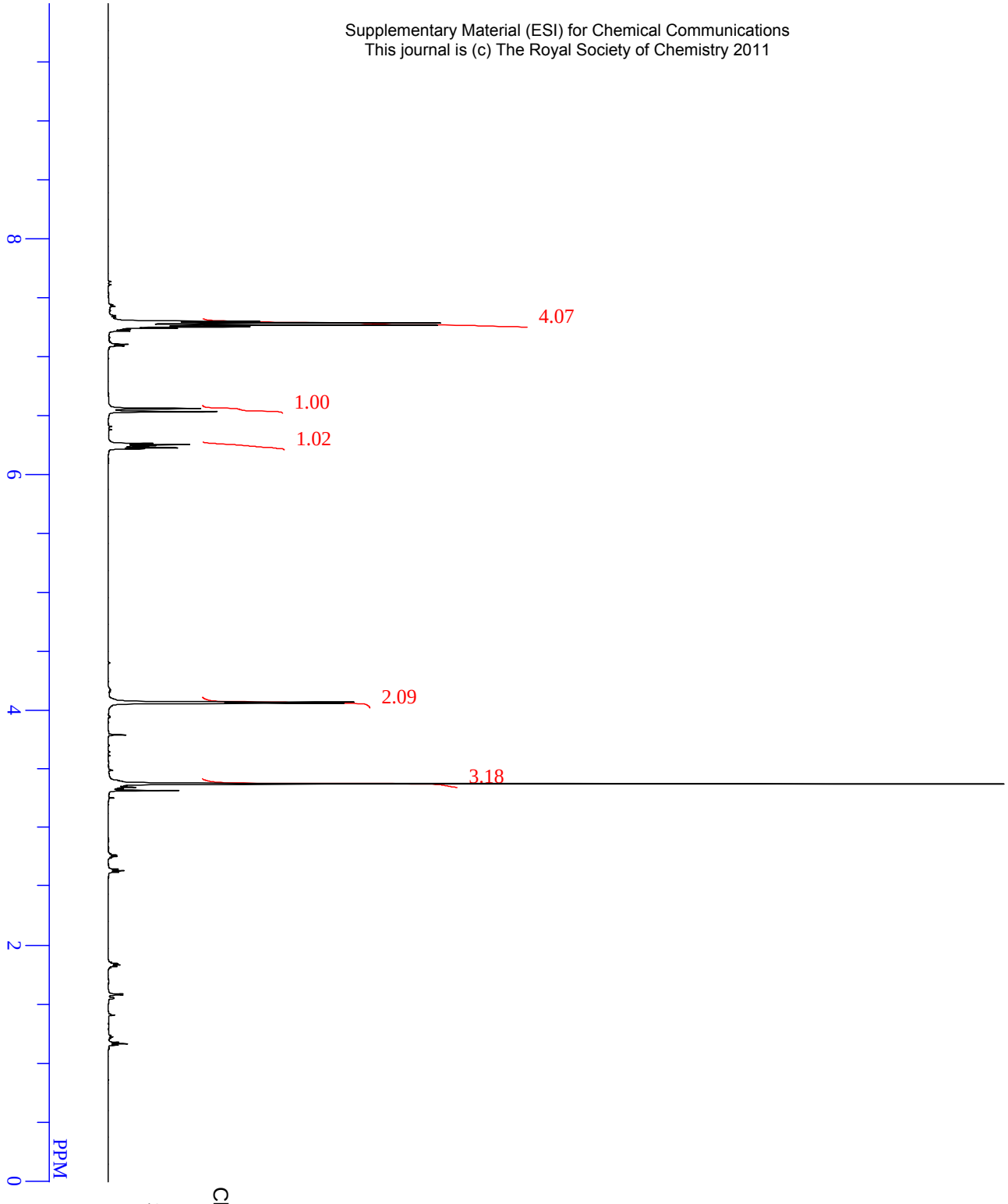




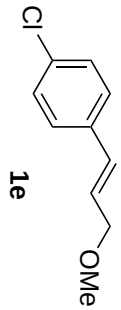
^{1}H NMR (600 MHz, $CDCl_3$)

1d

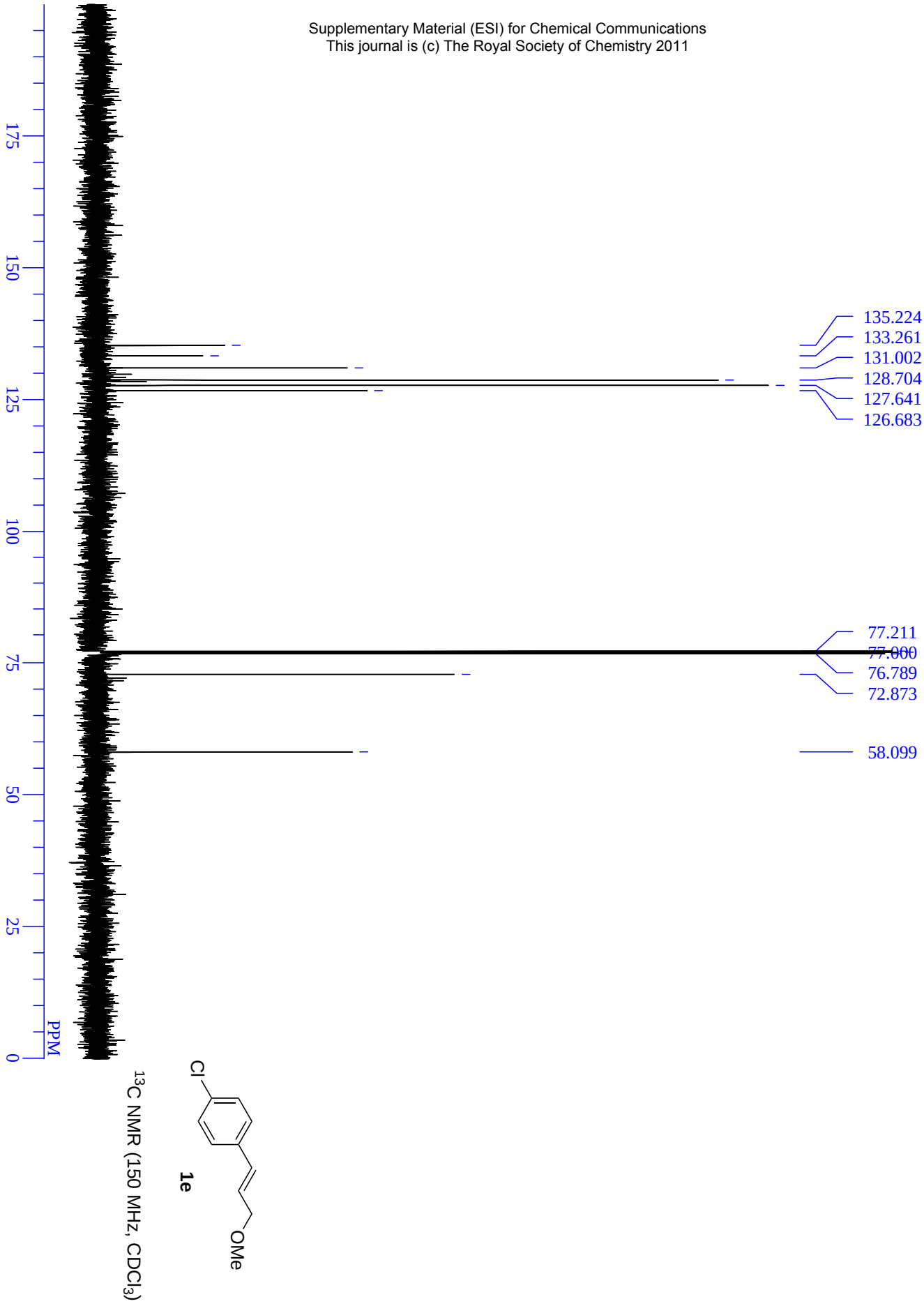

137.503
133.932
132.495
129.240
126.387
124.874
77.211
77.000
76.789
73.199
57.889
21.170
175
150
125
100
75
50
25
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
1d
OMe

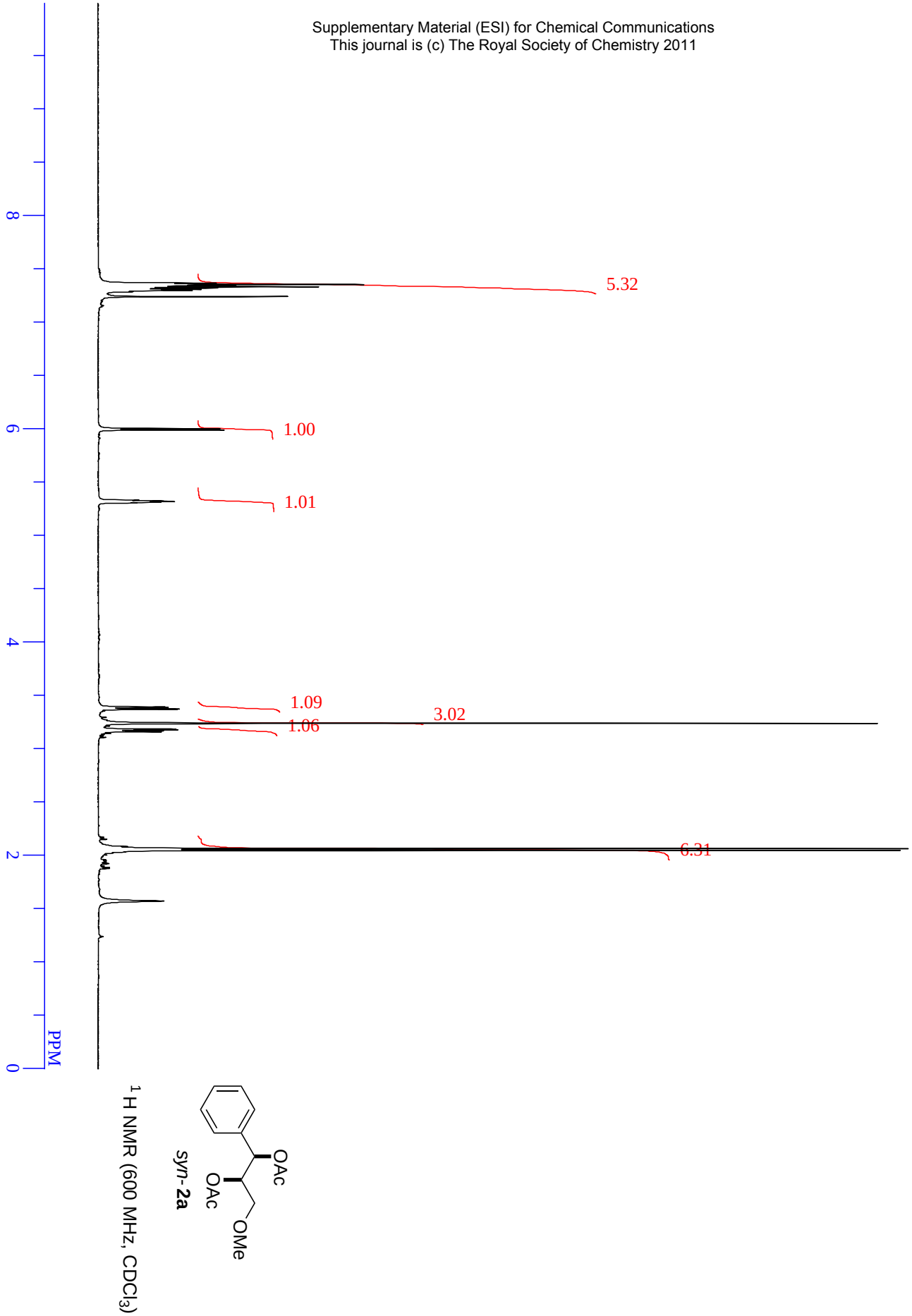


^{1}H NMR (600 MHz, $CDCl_3$)



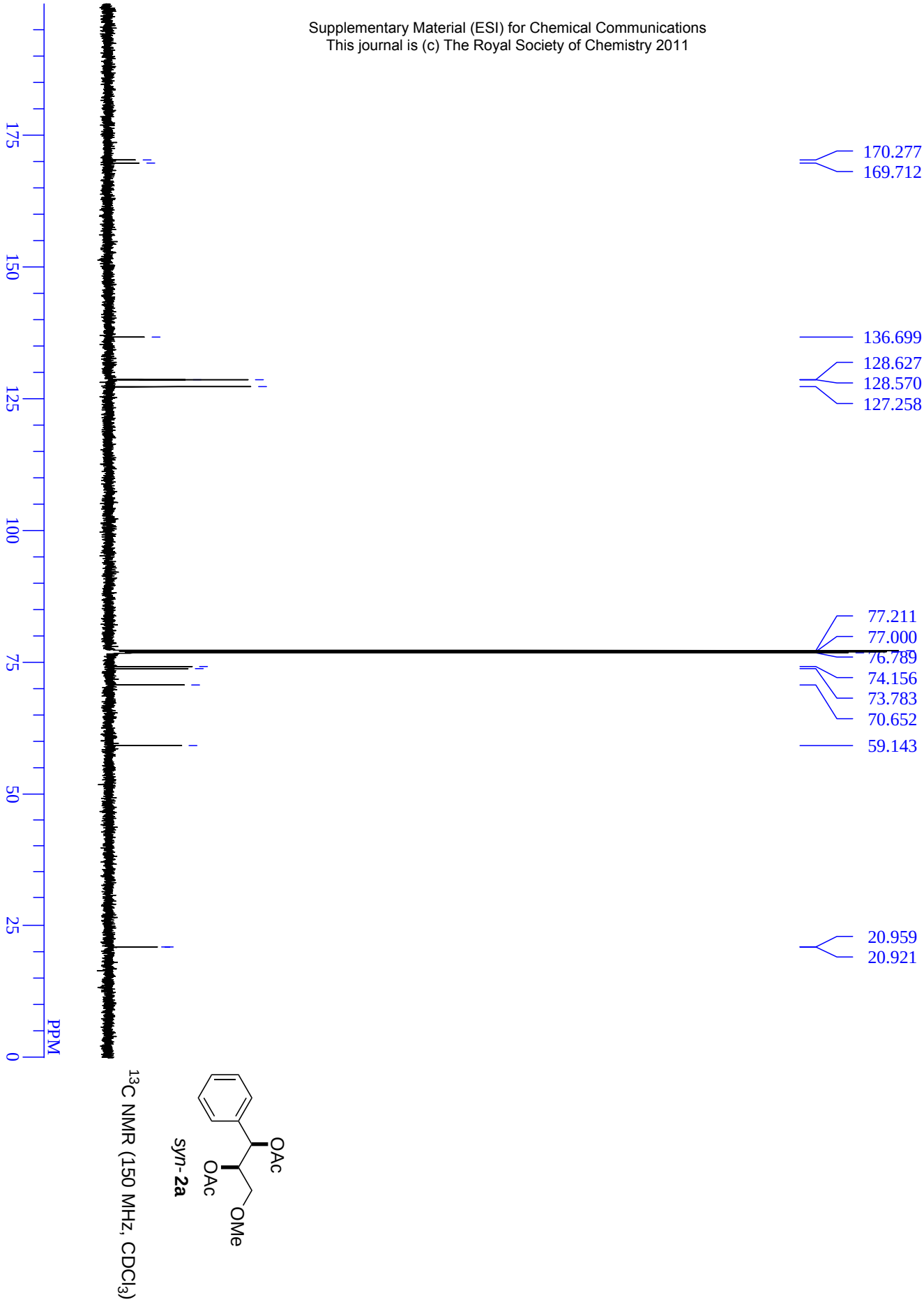
1e

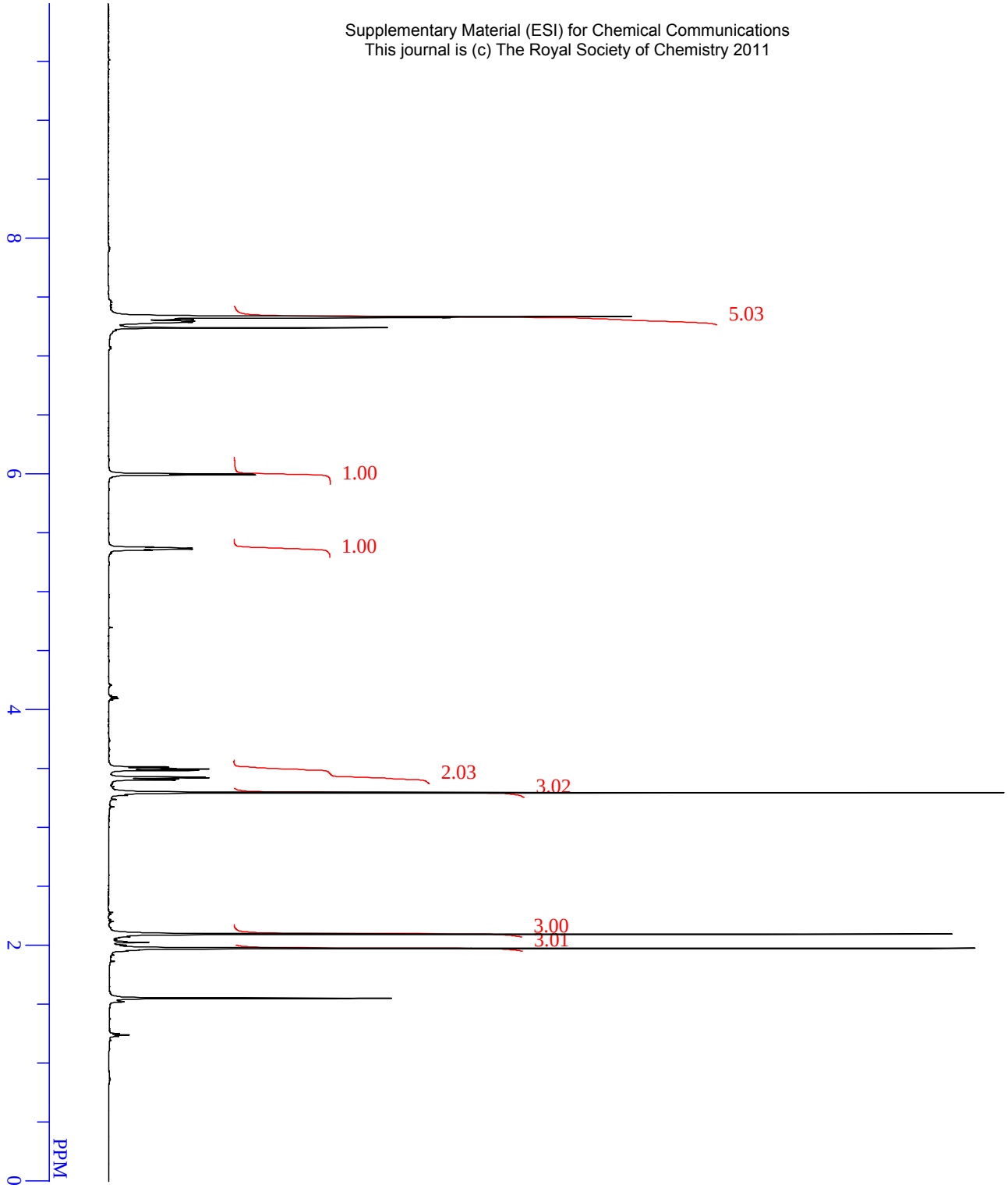

135.224
133.261
131.002
128.704
127.641
126.683
77.211
77.000
76.789
72.873
58.099
175
150
125
100
75
50
25
0
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
Cl
OMe
1e



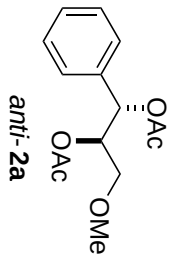
^{1}H NMR (600 MHz, $CDCl_3$)

syn-**2a**

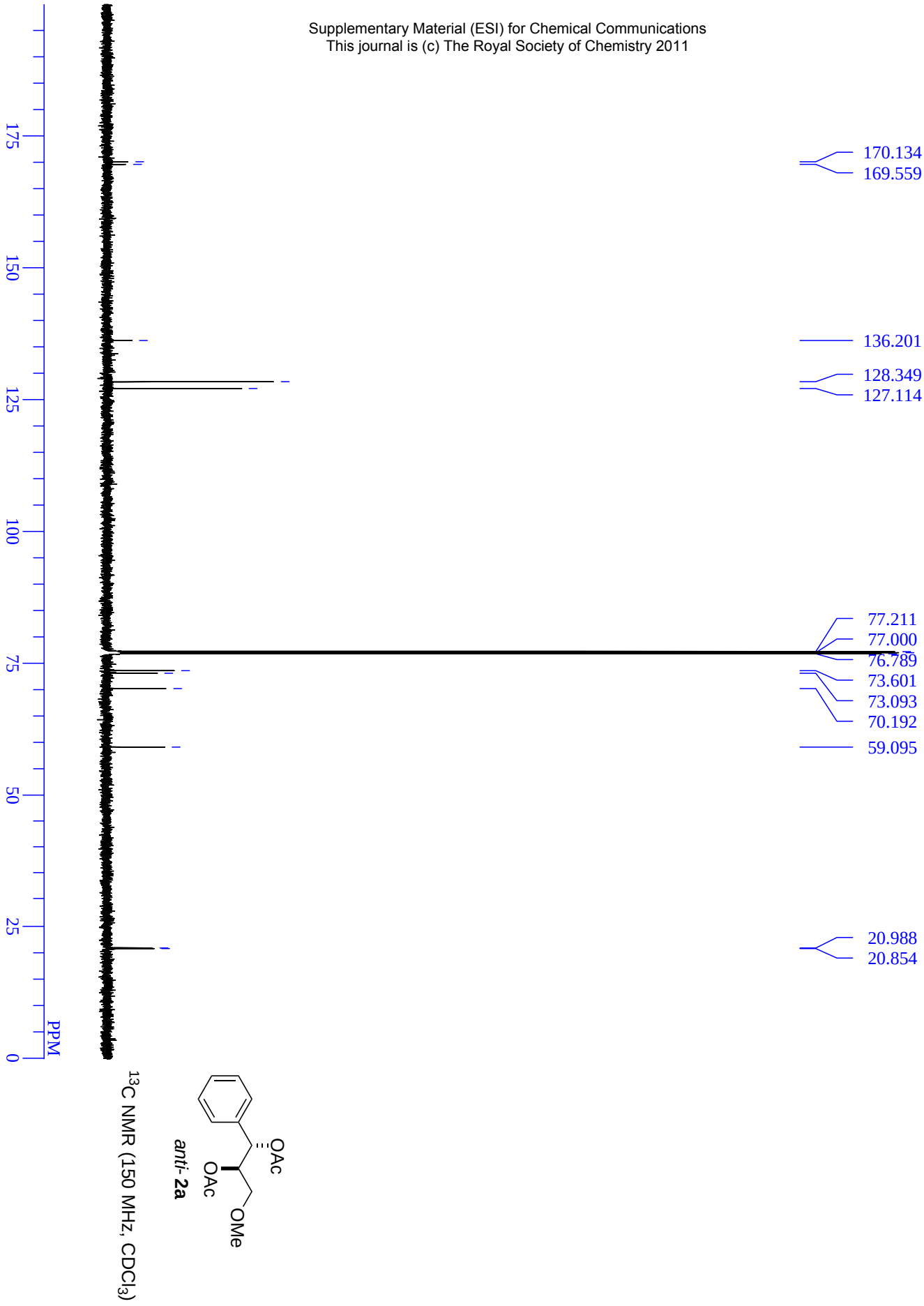

170.277
169.712
136.699
128.627
128.570
127.258
77.211
77.000
76.789
74.156
73.783
70.652
59.143
20.959
20.921
175
150
125
100
75
50
25
0
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
OAc
OAc
OMe
syn-**2a**

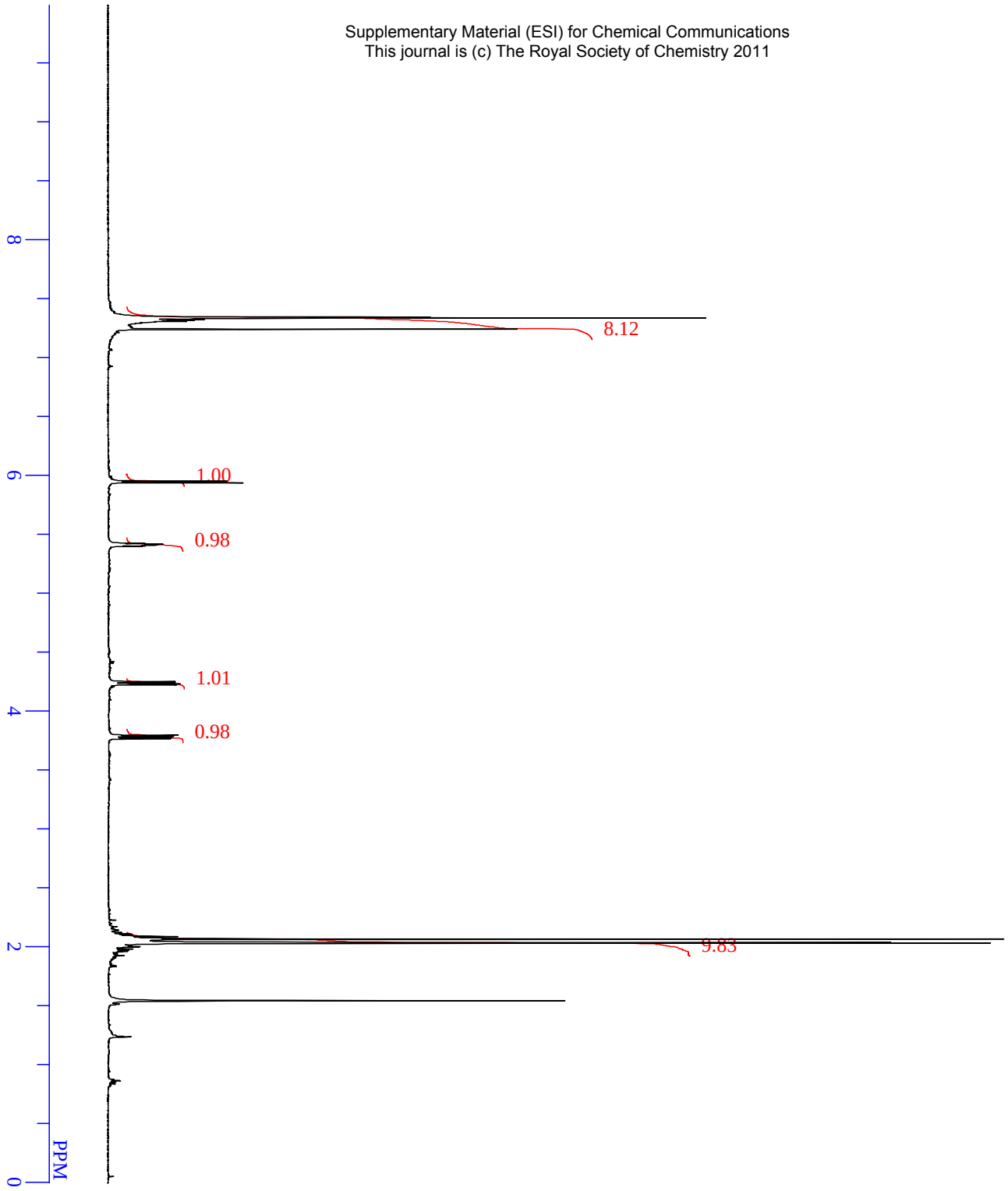


^{1}H NMR (600 MHz, $CDCl_3$)

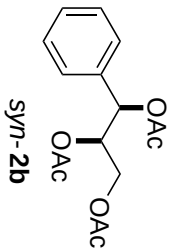


anti-**2a**

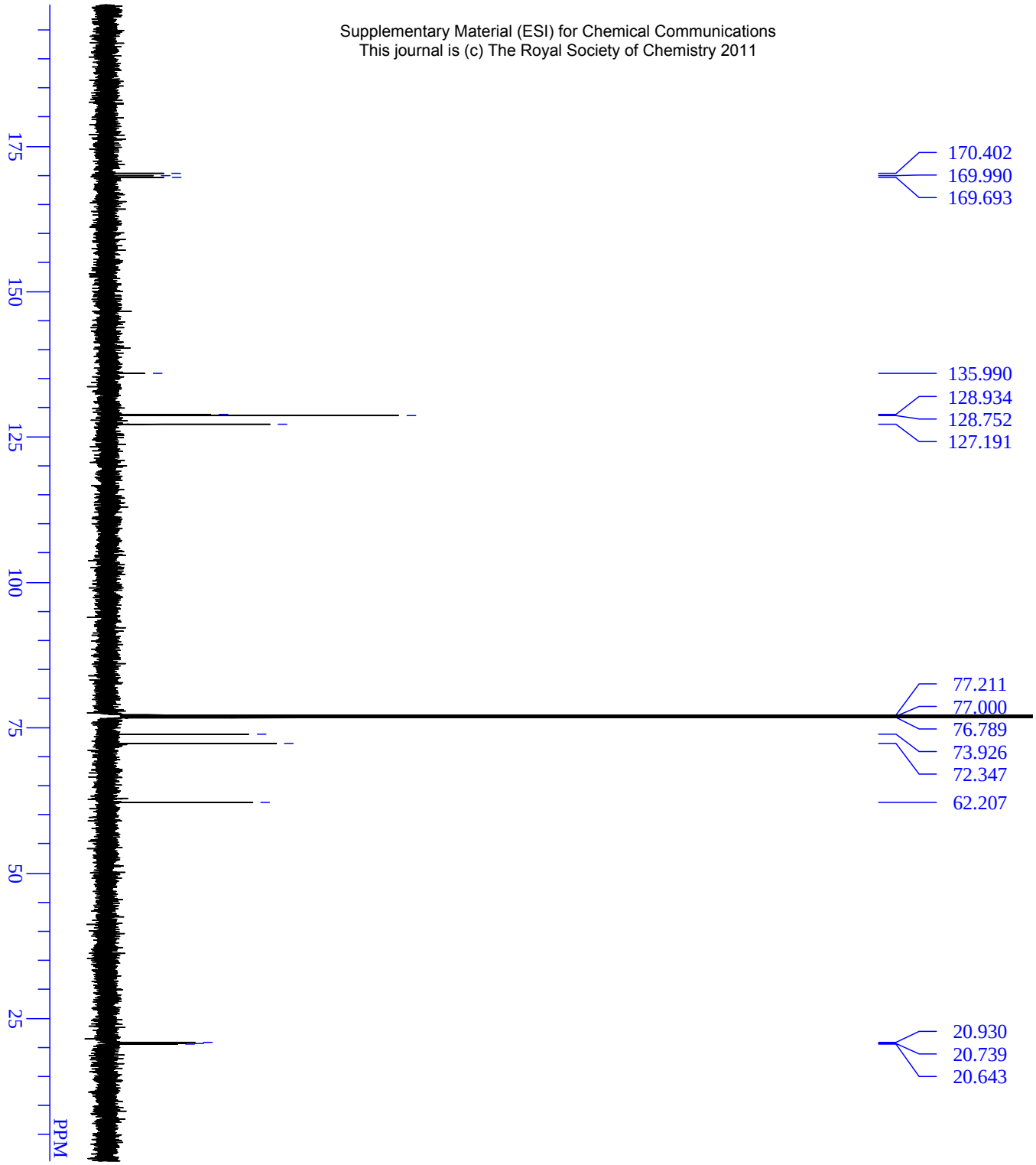

170.134
169.559
136.201
128.349
127.114
77.211
77.000
76.789
73.601
73.093
70.192
59.095
20.988
20.854
175
150
125
100
75
50
25
0
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
OAc
OAc
OMe
anti- 2a



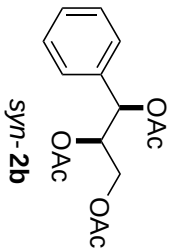
^{1}H NMR (600 MHz, $CDCl_3$)



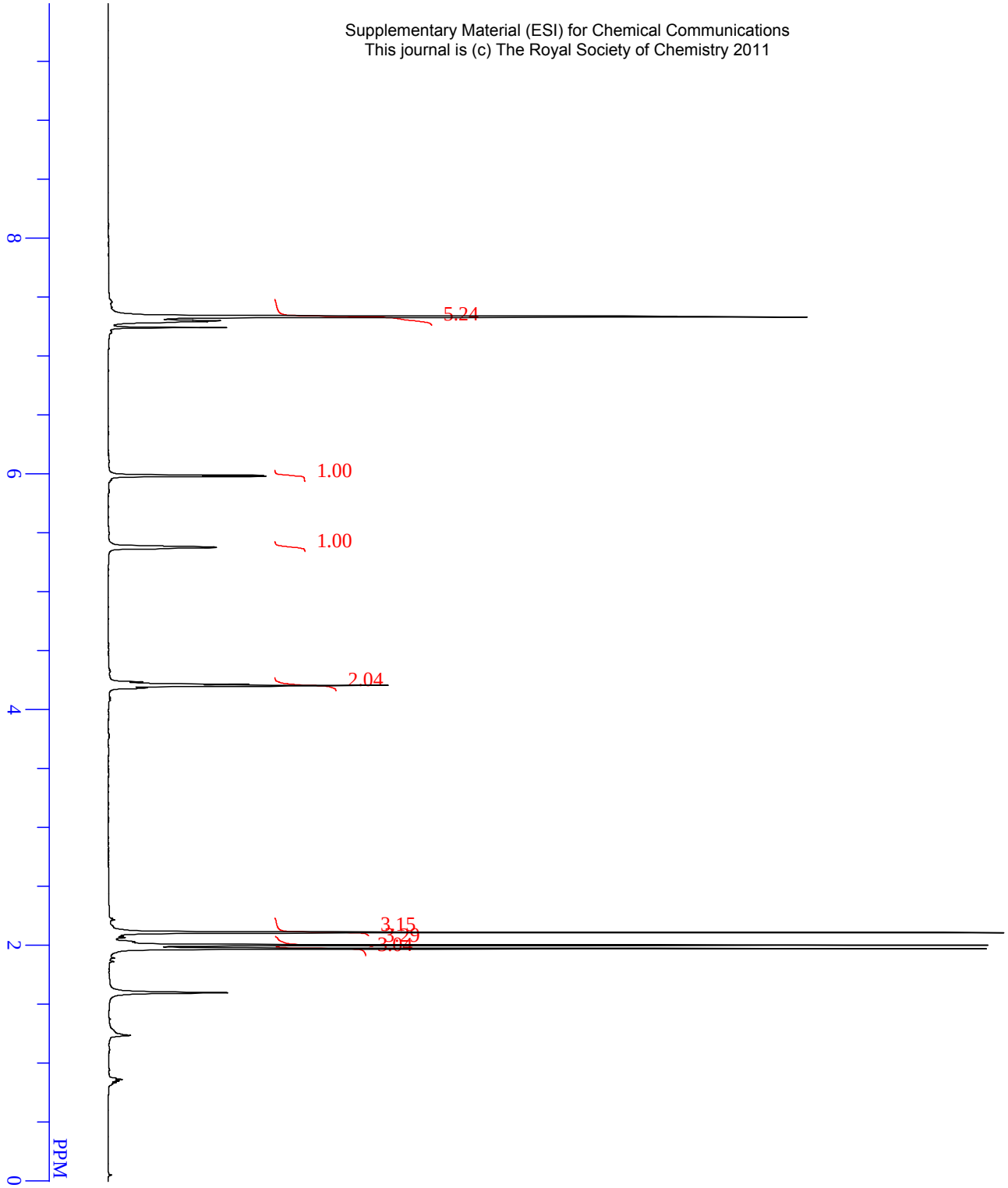
syn-**2b**



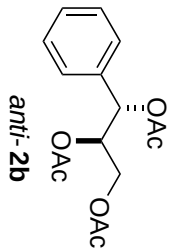
^{13}C NMR (150 MHz, $CDCl_3$)



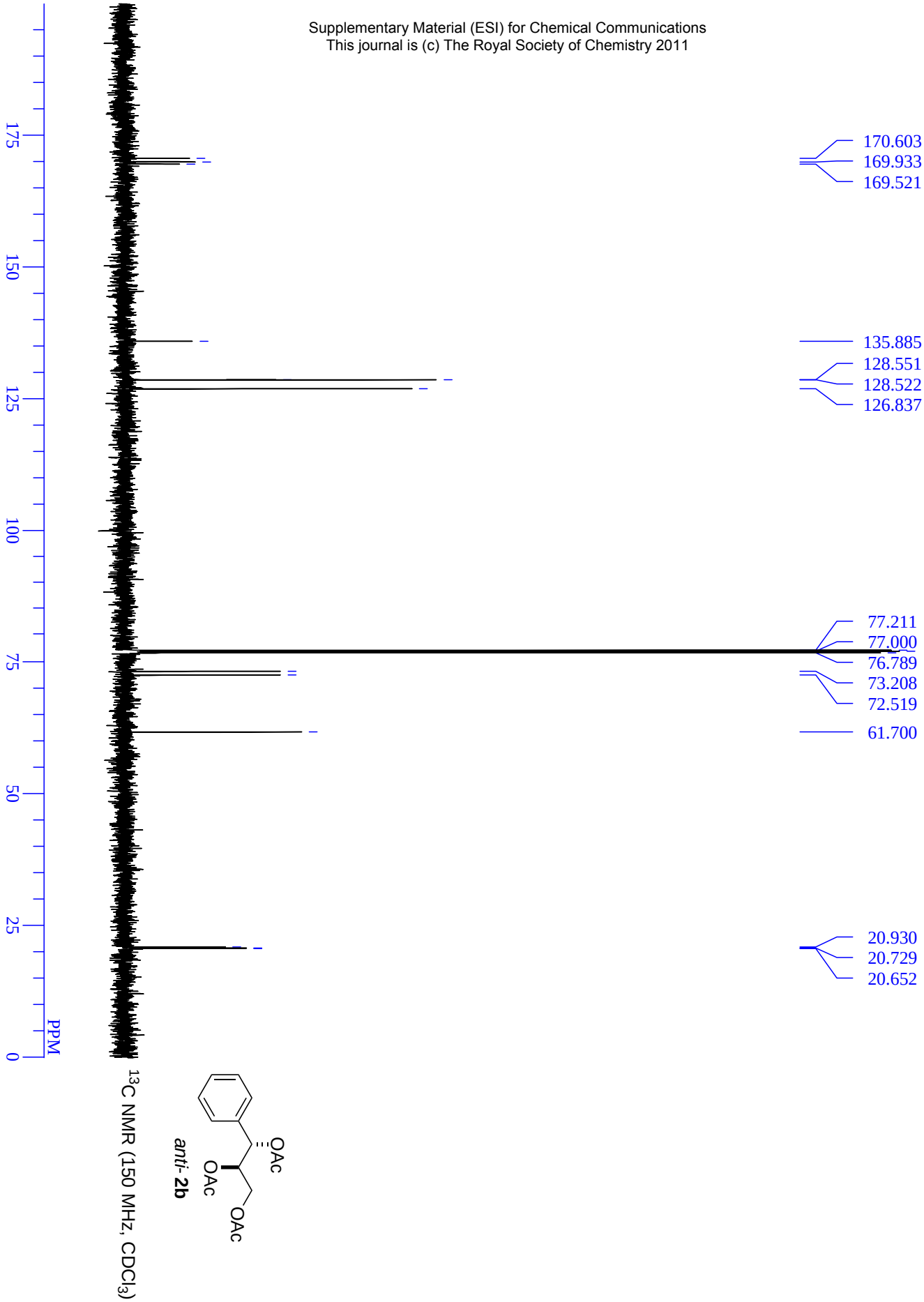
syn-**2b**

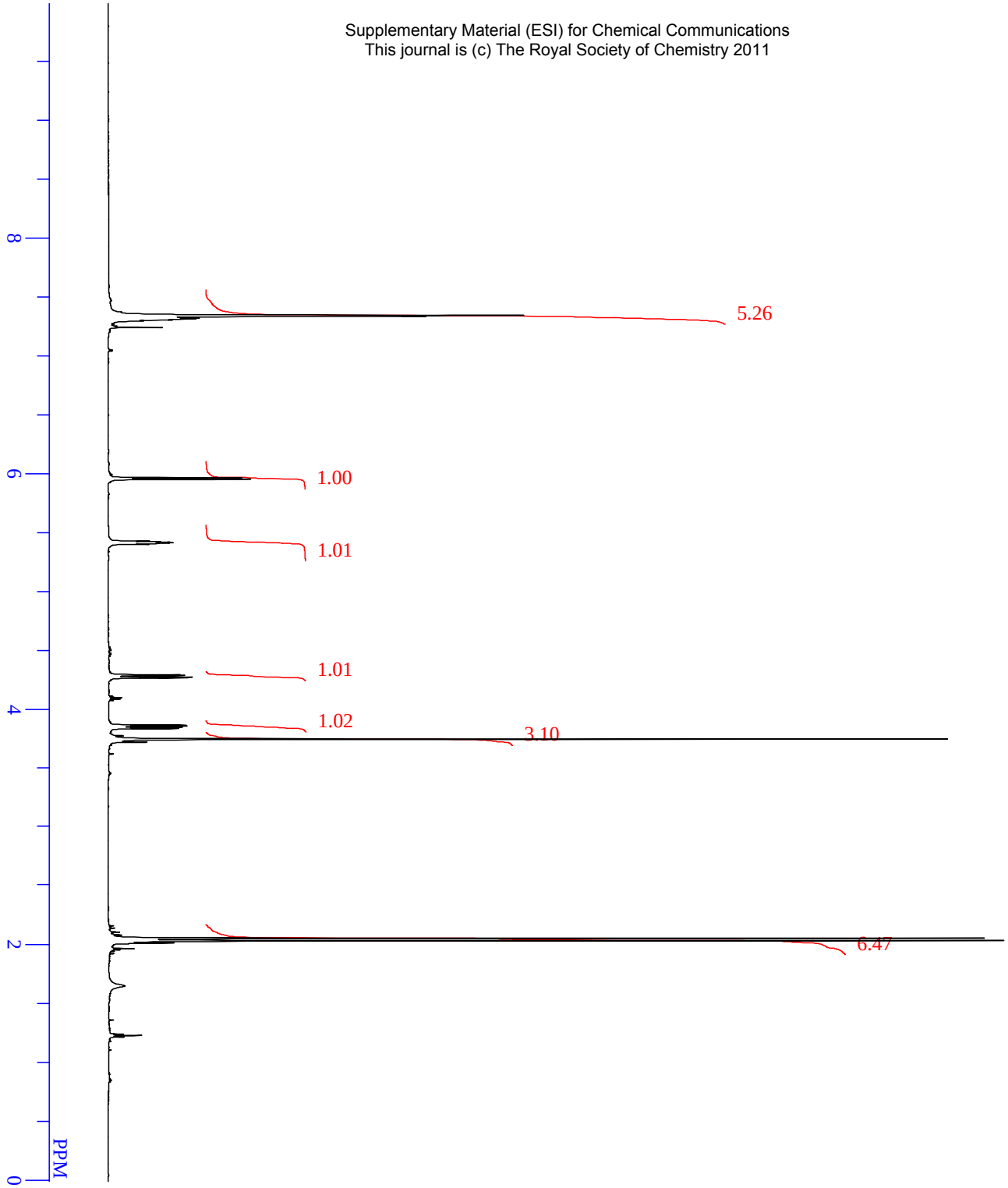


^{1}H NMR (600 MHz, $CDCl_3$)

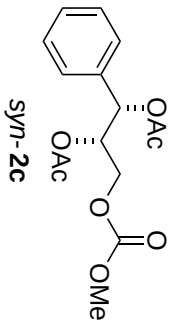


anti-**2b**

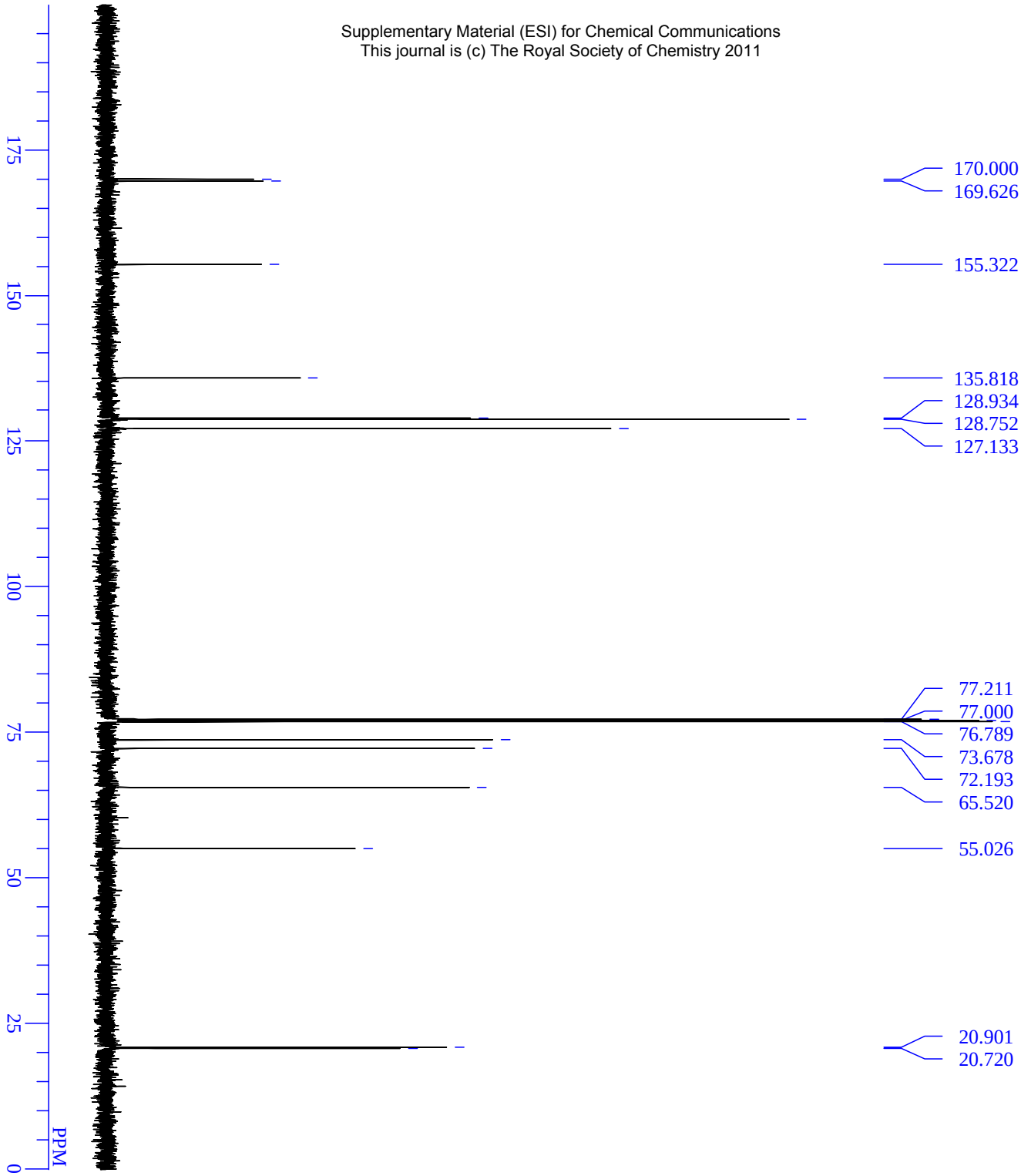

170.603
169.933
169.521
135.885
128.551
128.522
126.837
77.211
77.000
76.789
73.208
72.519
61.700
20.930
20.729
20.652
175
150
125
100
75
50
25
0
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
OAc
OAc
OAc
anti- 2b



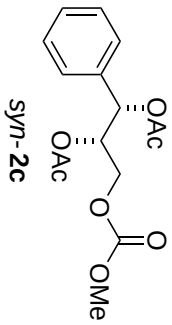
^{1}H NMR (600 MHz, $CDCl_3$)



syn-**2c**



^{13}C NMR (150 MHz, $CDCl_3$)



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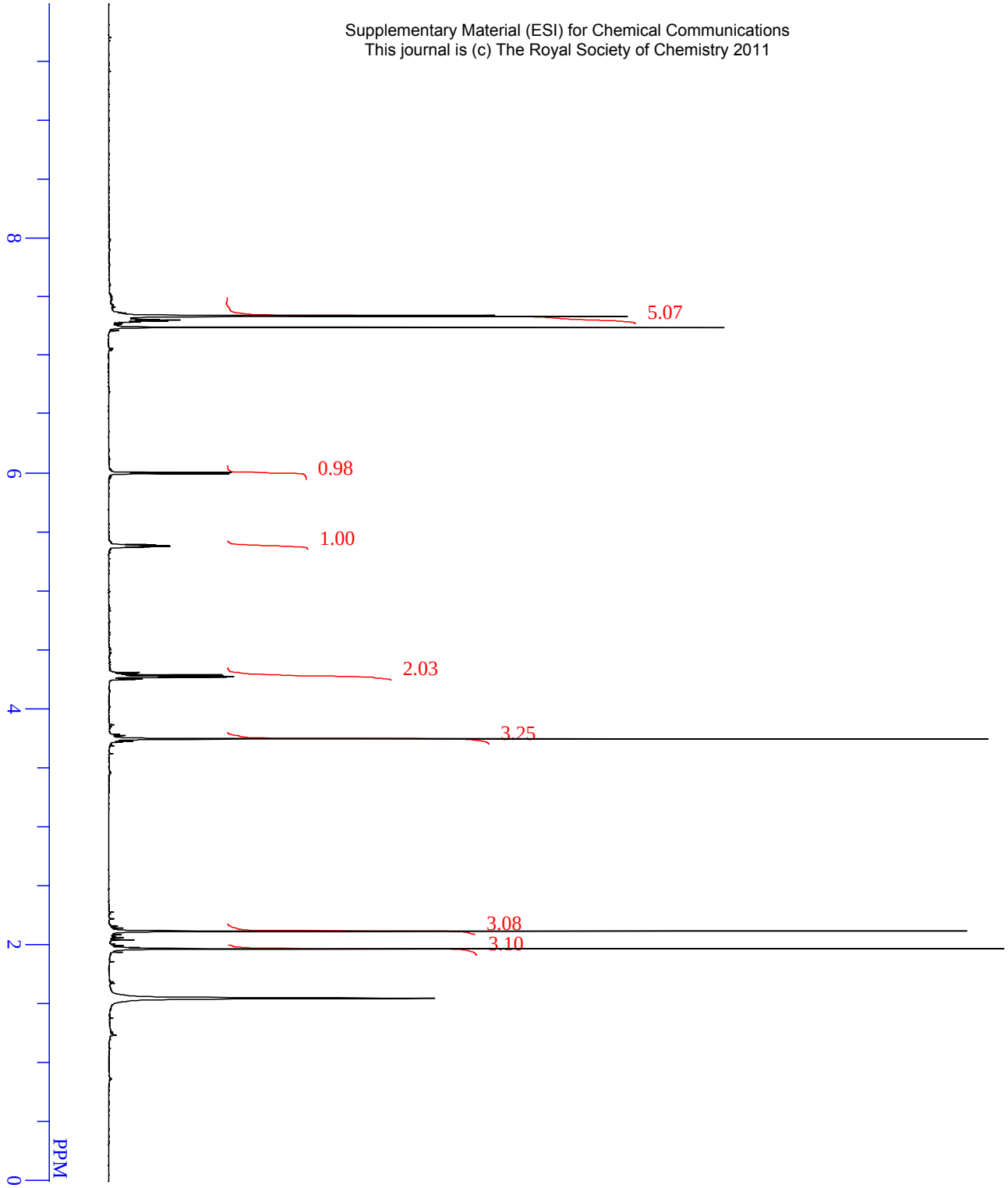
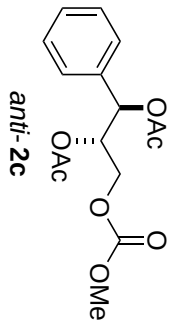


^{1}H NMR (600 MHz, $CDCl_3$)



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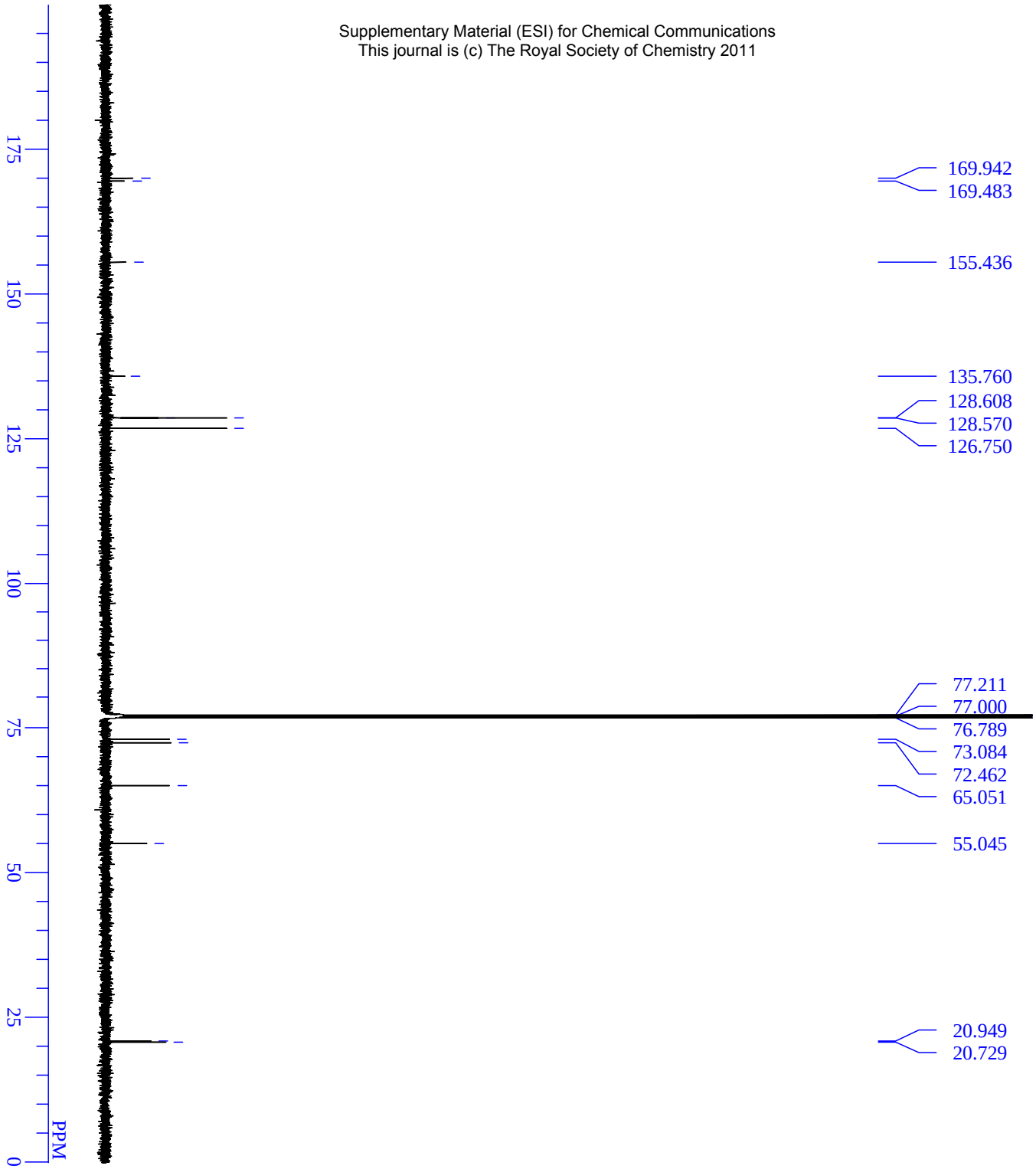
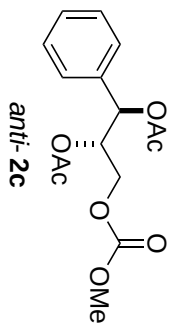


^{13}C NMR (150 MHz, $CDCl_3$)



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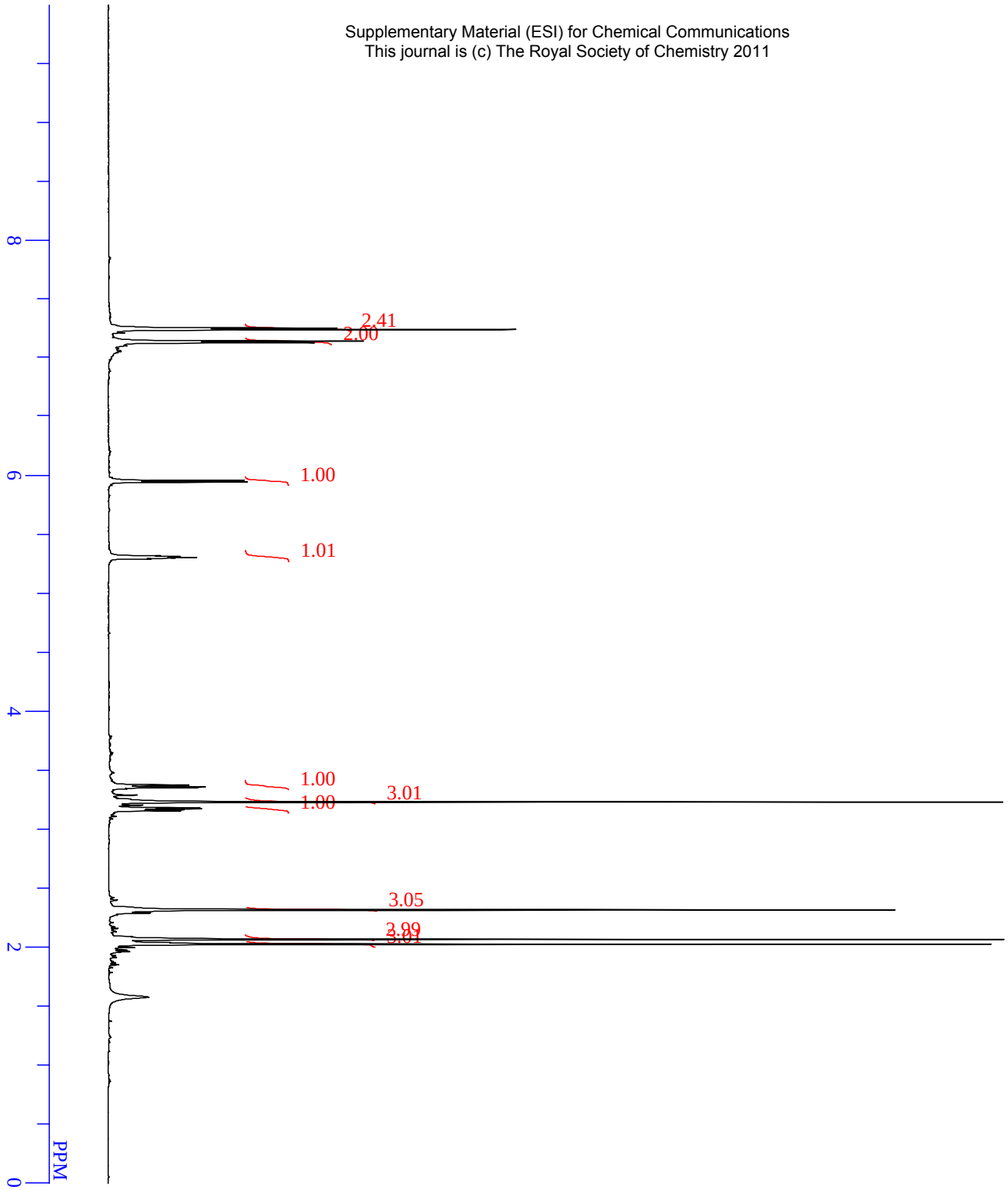
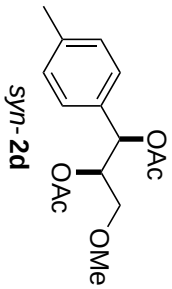


^{1}H NMR (600 MHz, $CDCl_3$)



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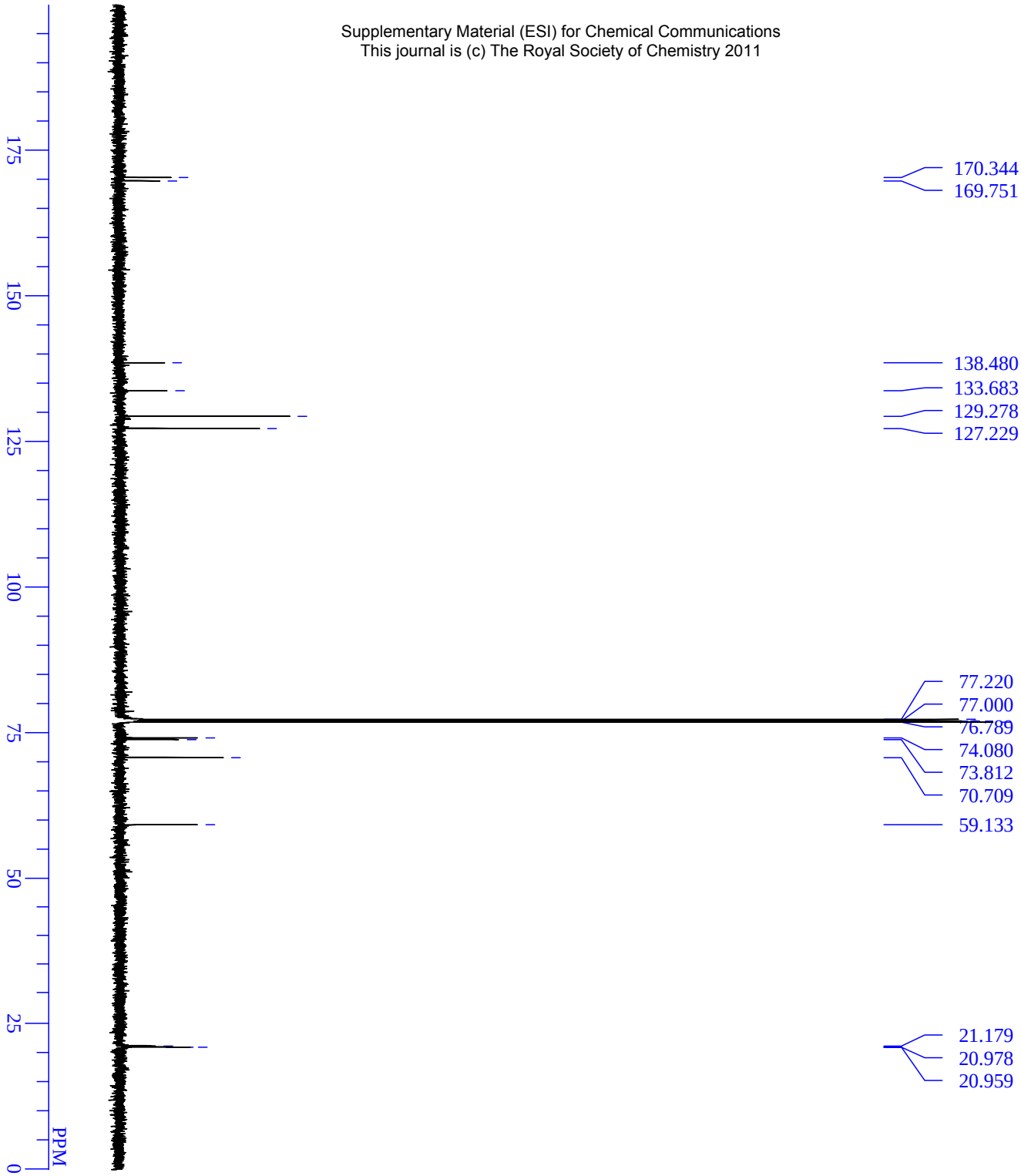
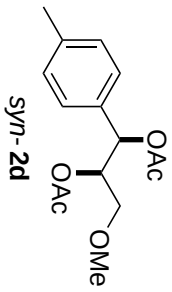


^{13}C NMR (150 MHz, $CDCl_3$)



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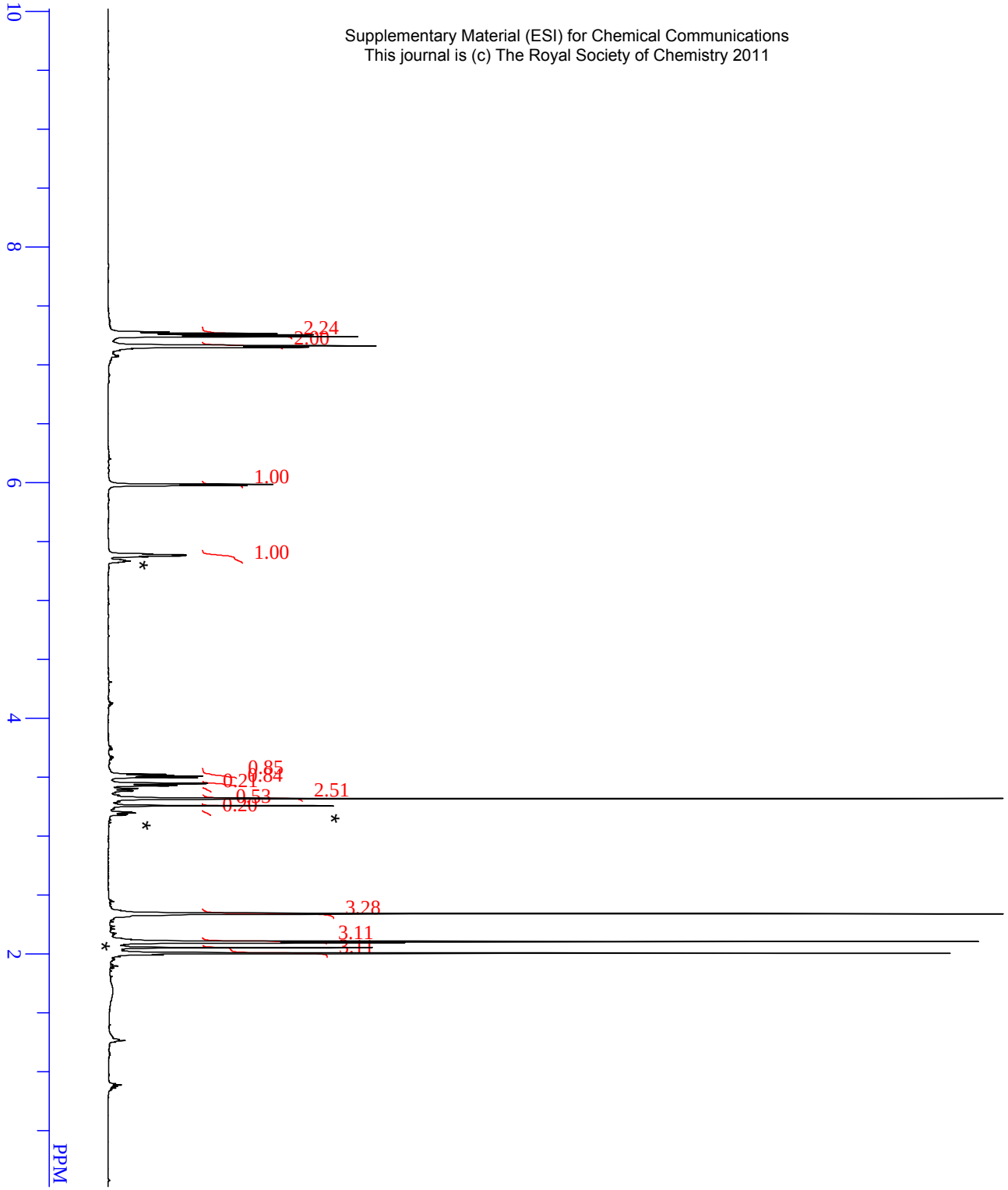
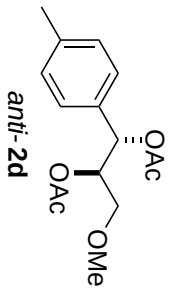
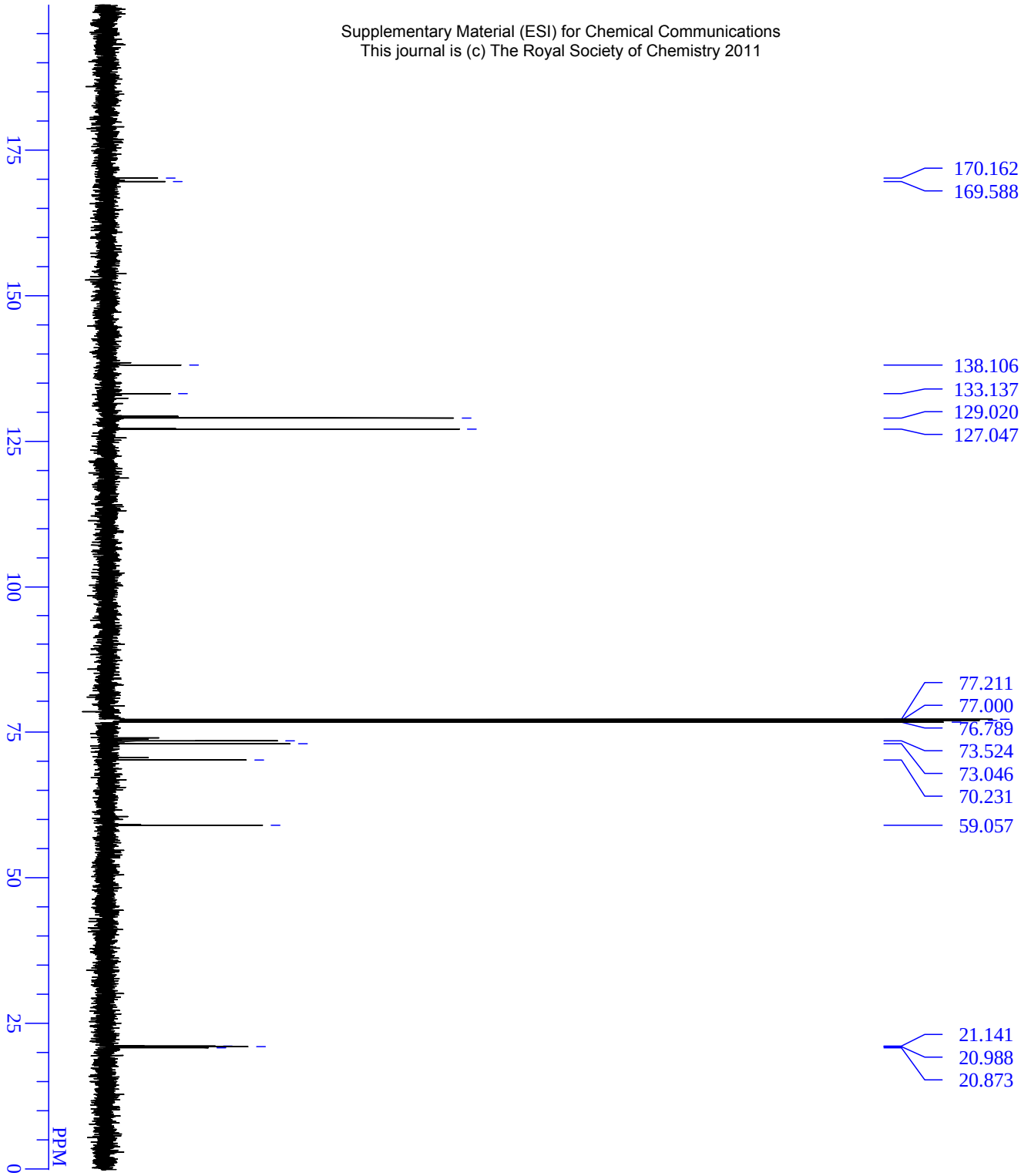


^{1}H NMR (600 MHz, $CDCl_3$)

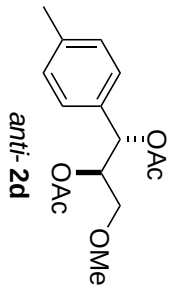


anti-**2d**

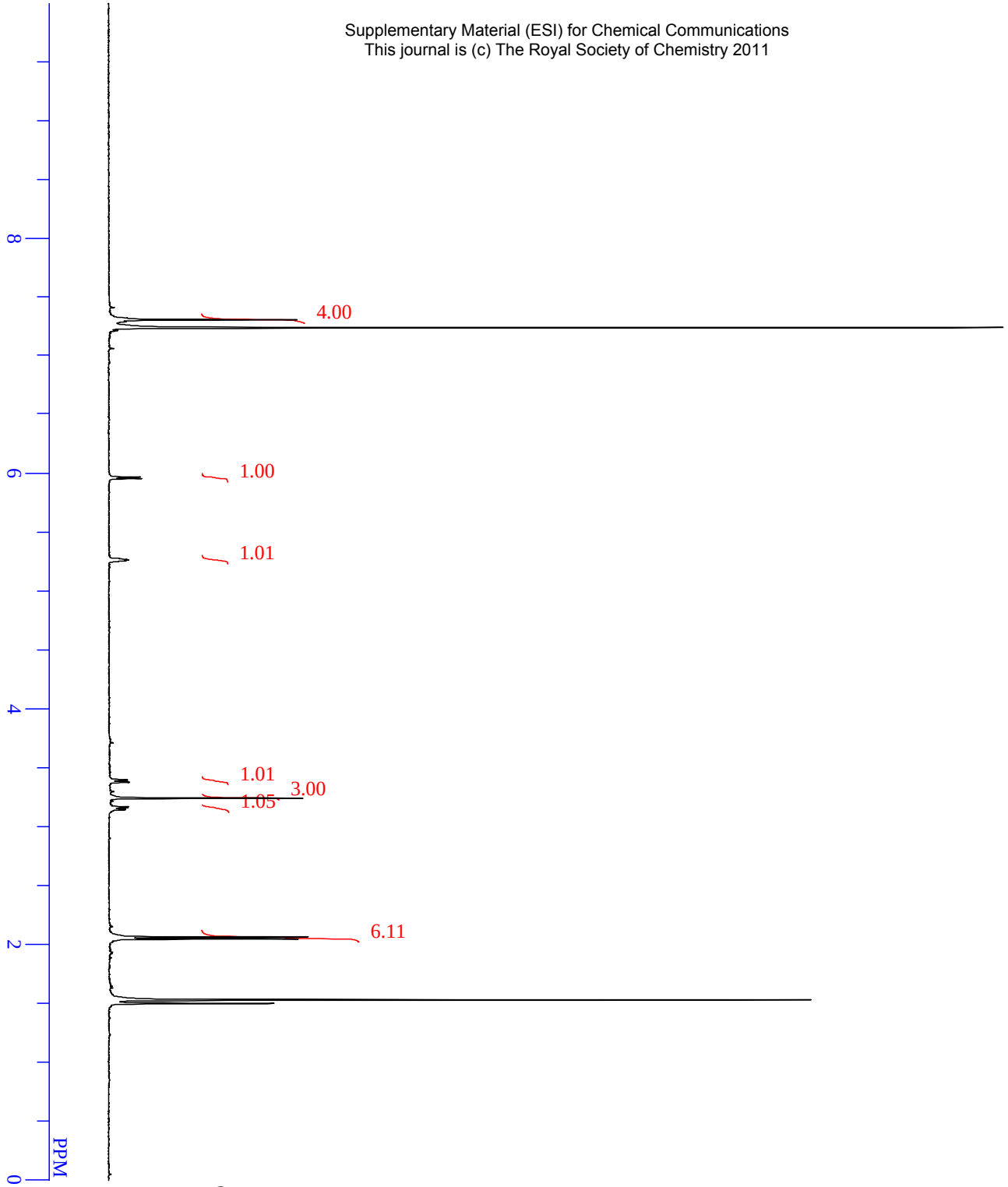
* *syn*-**2d**



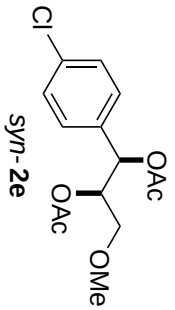
^{13}C NMR (150 MHz, $CDCl_3$)



anti- **2d**



^{1}H NMR (600 MHz, $CDCl_3$)



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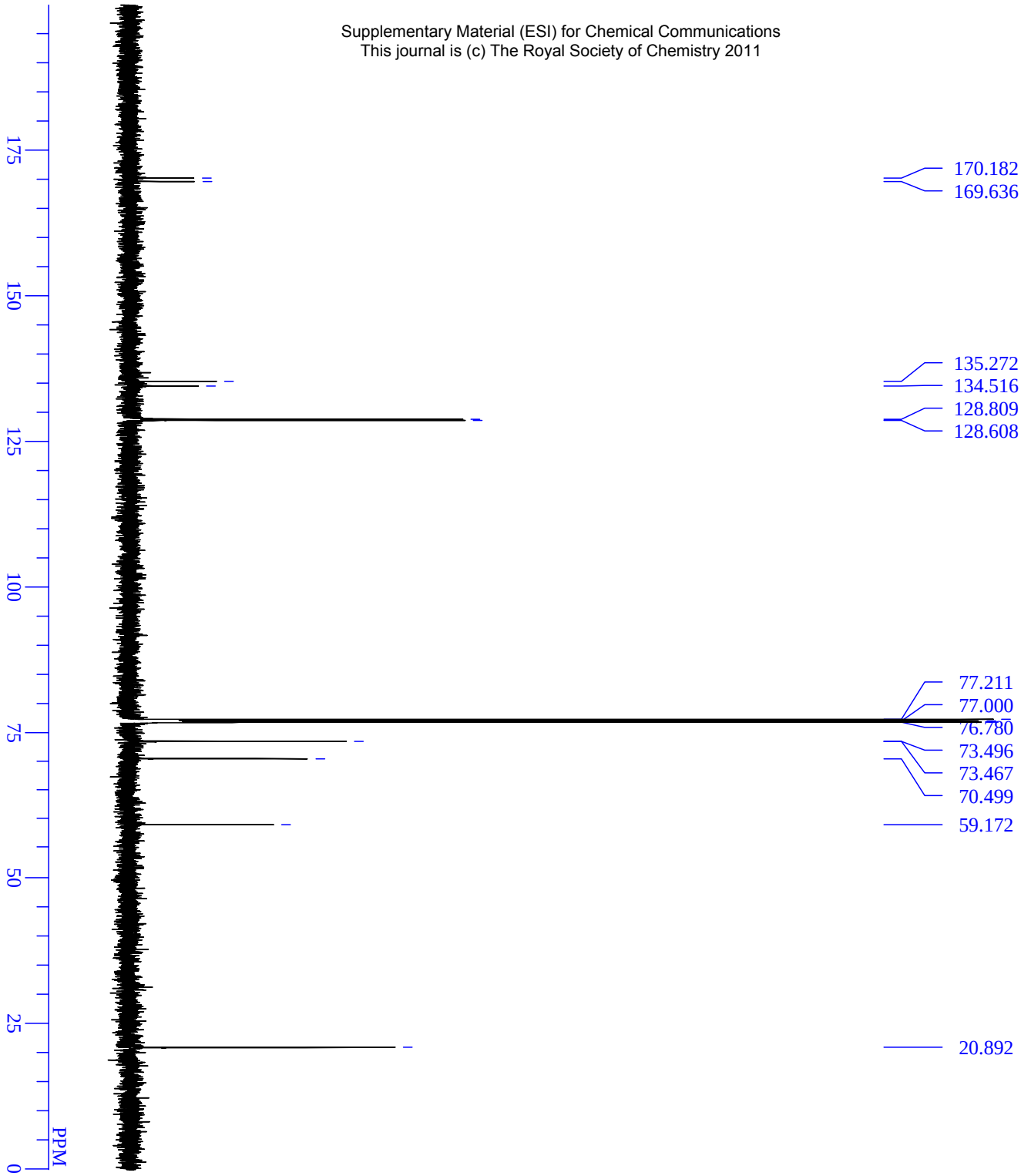
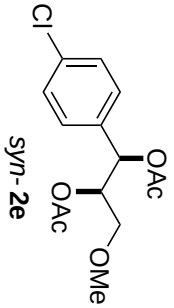


^{13}C NMR (150 MHz, $CDCl_3$)



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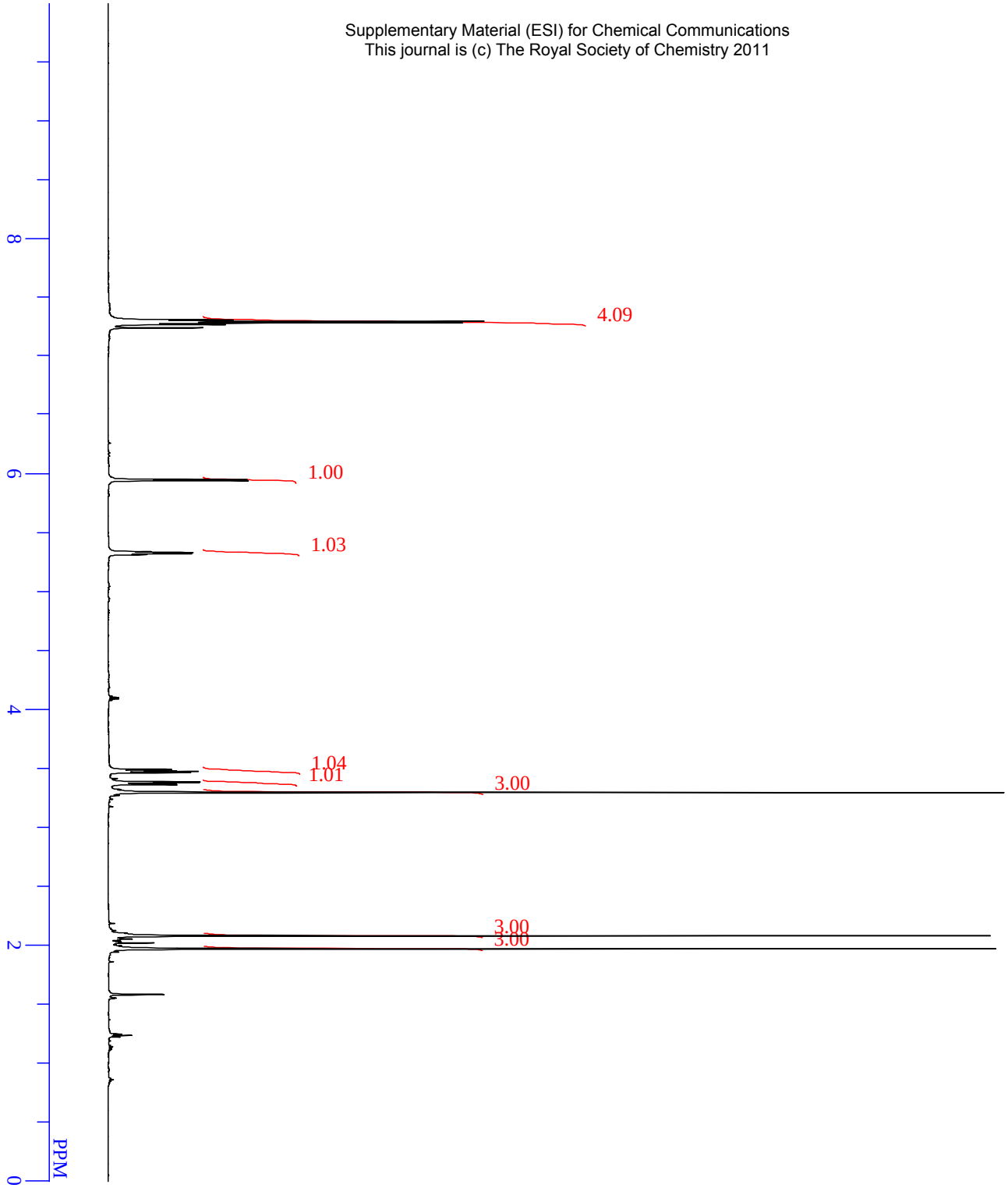

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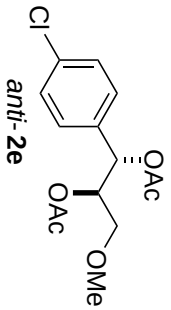

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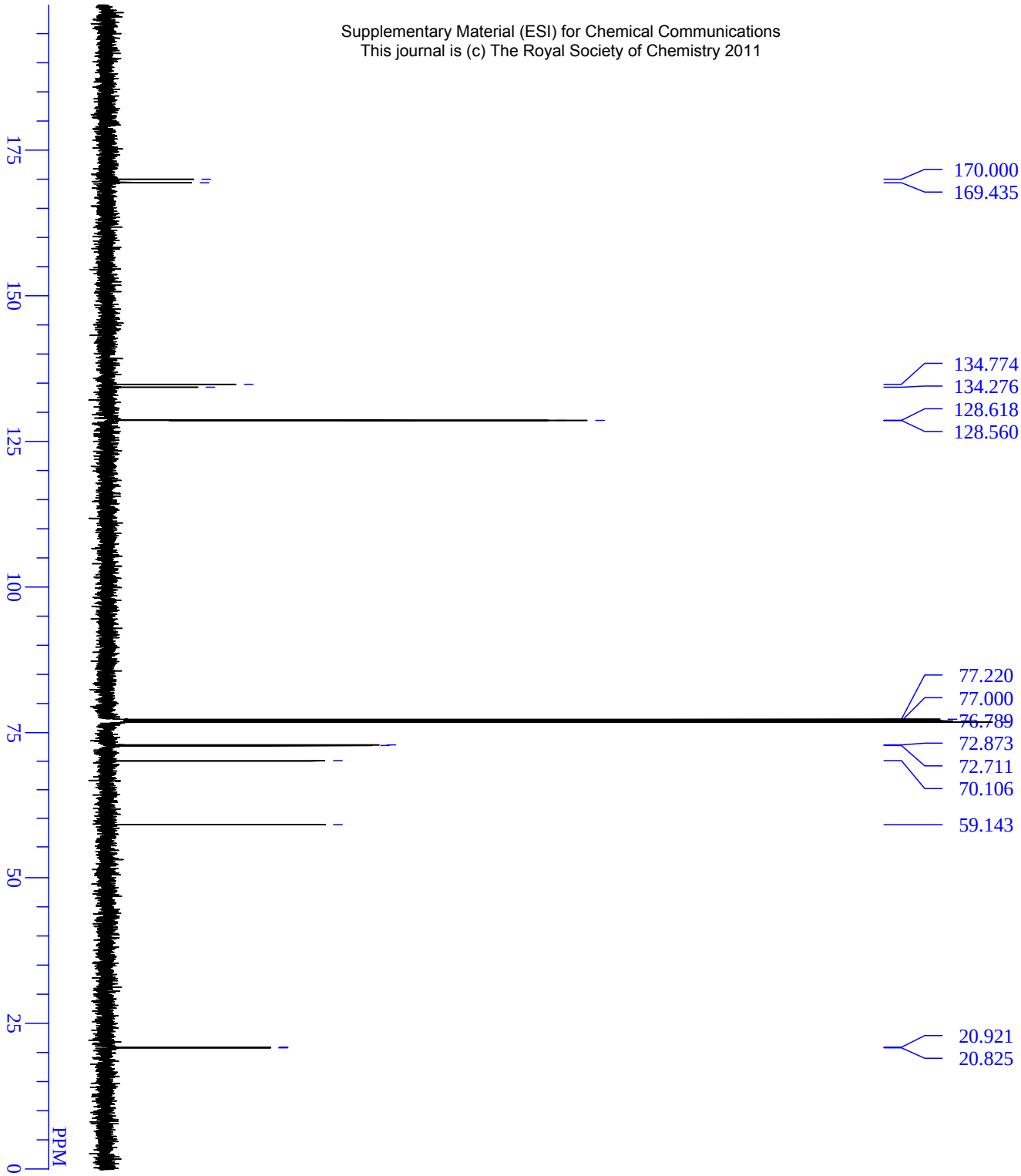

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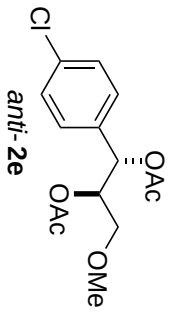

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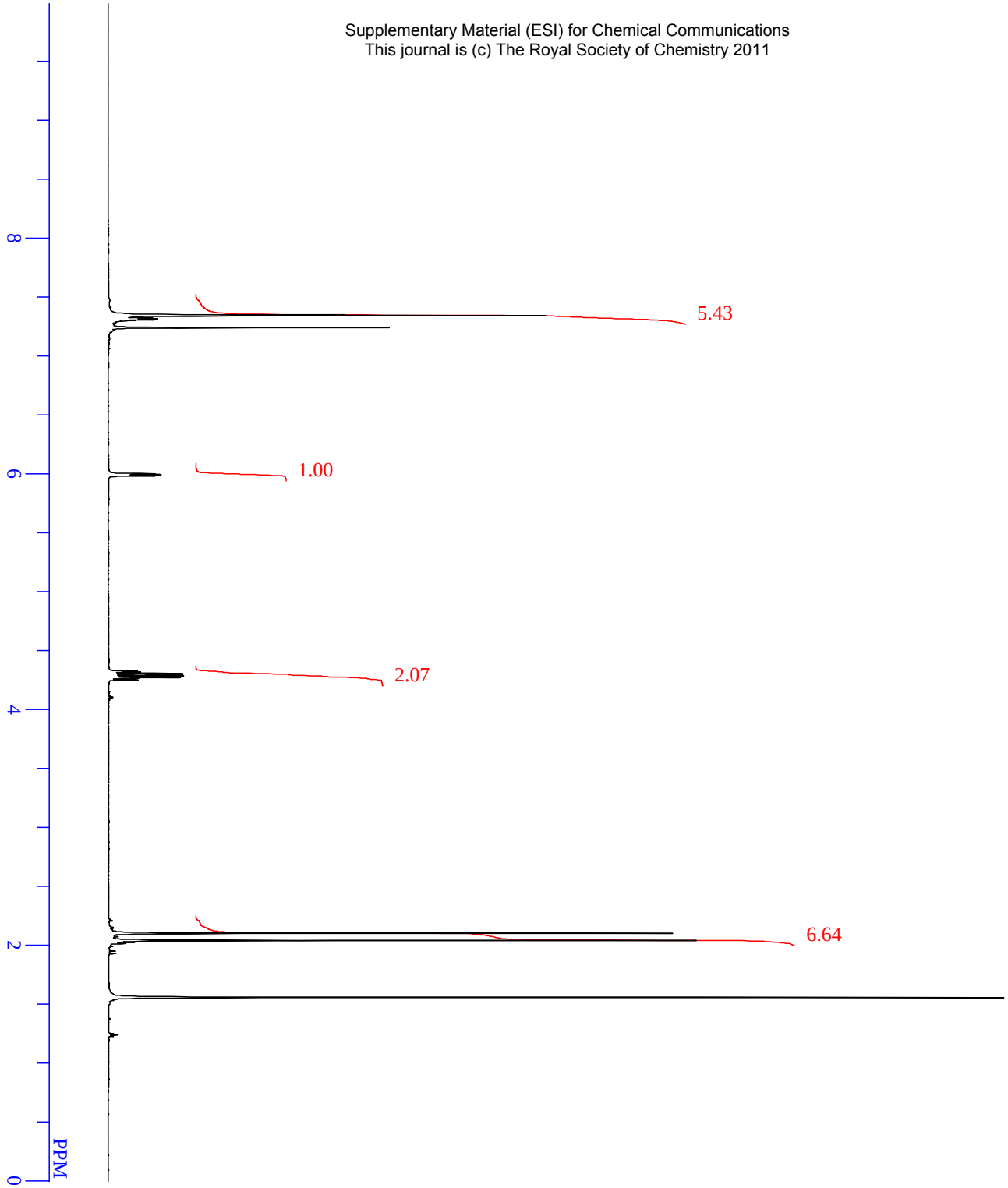
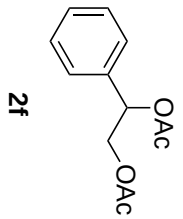


^{1}H NMR (600 MHz, $CDCl_3$)



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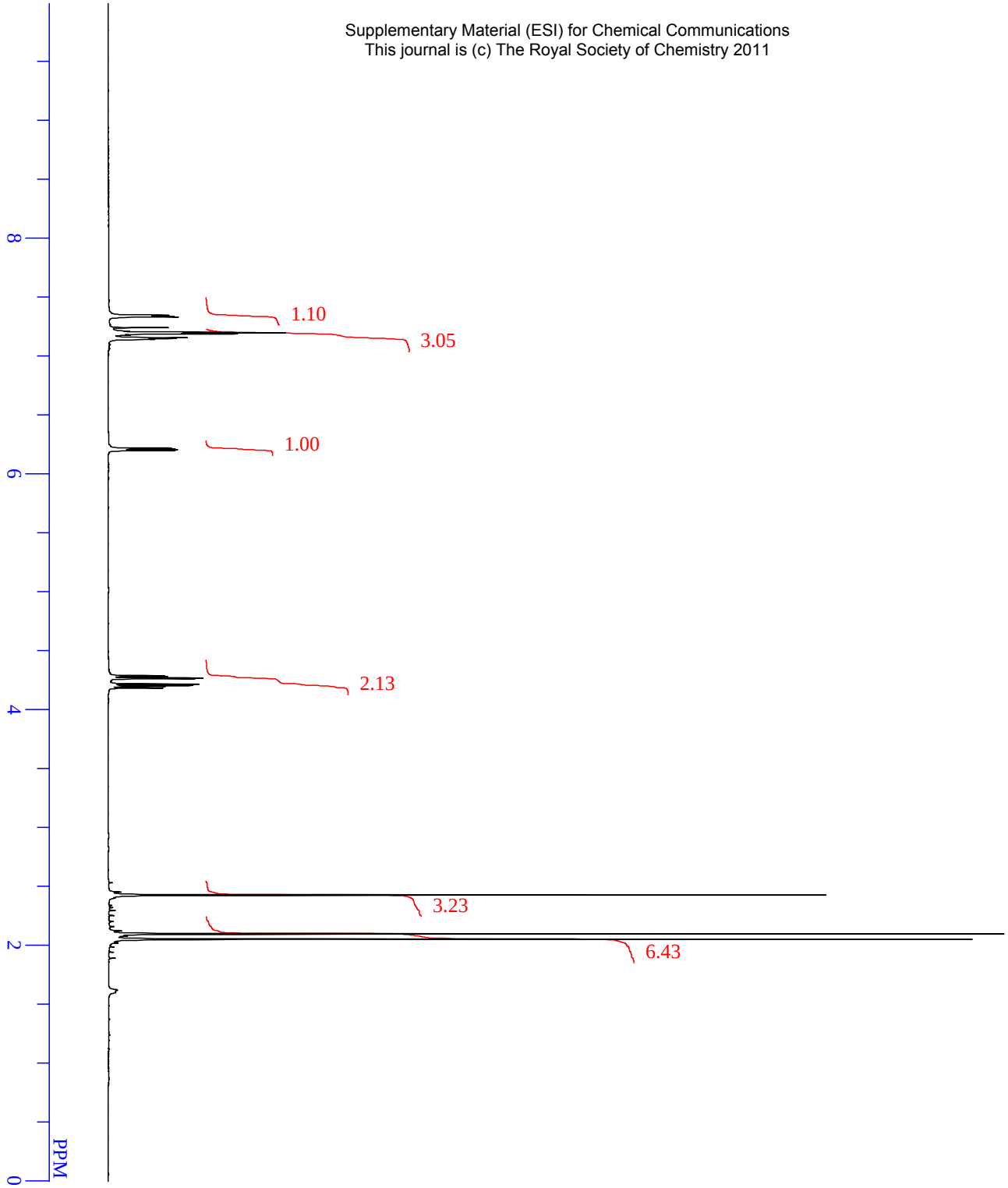

^{1}H NMR (600 MHz, $CDCl_3$)

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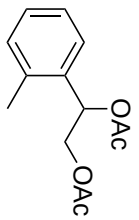

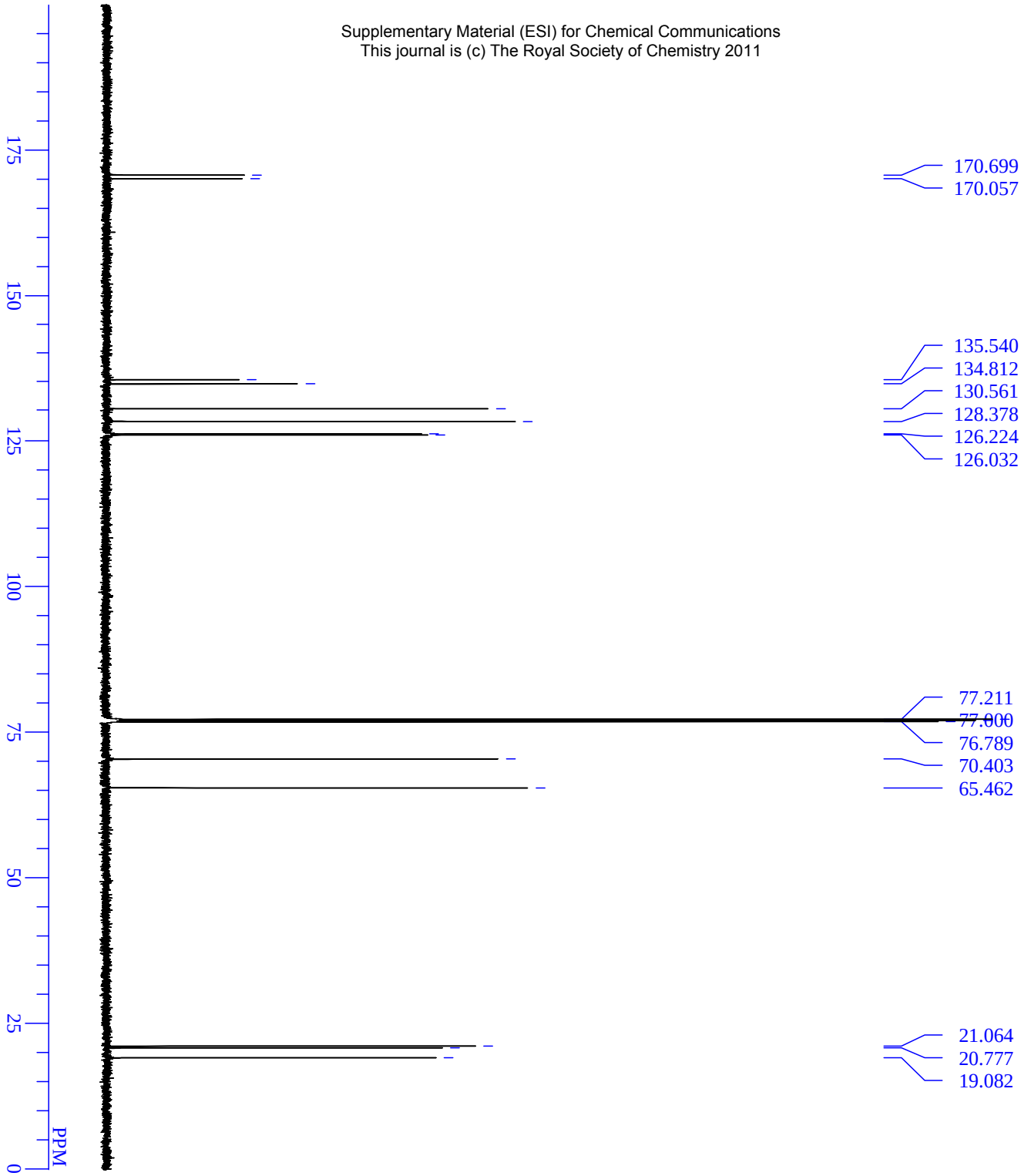

^{13}C NMR (150 MHz, $CDCl_3$)

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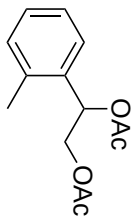
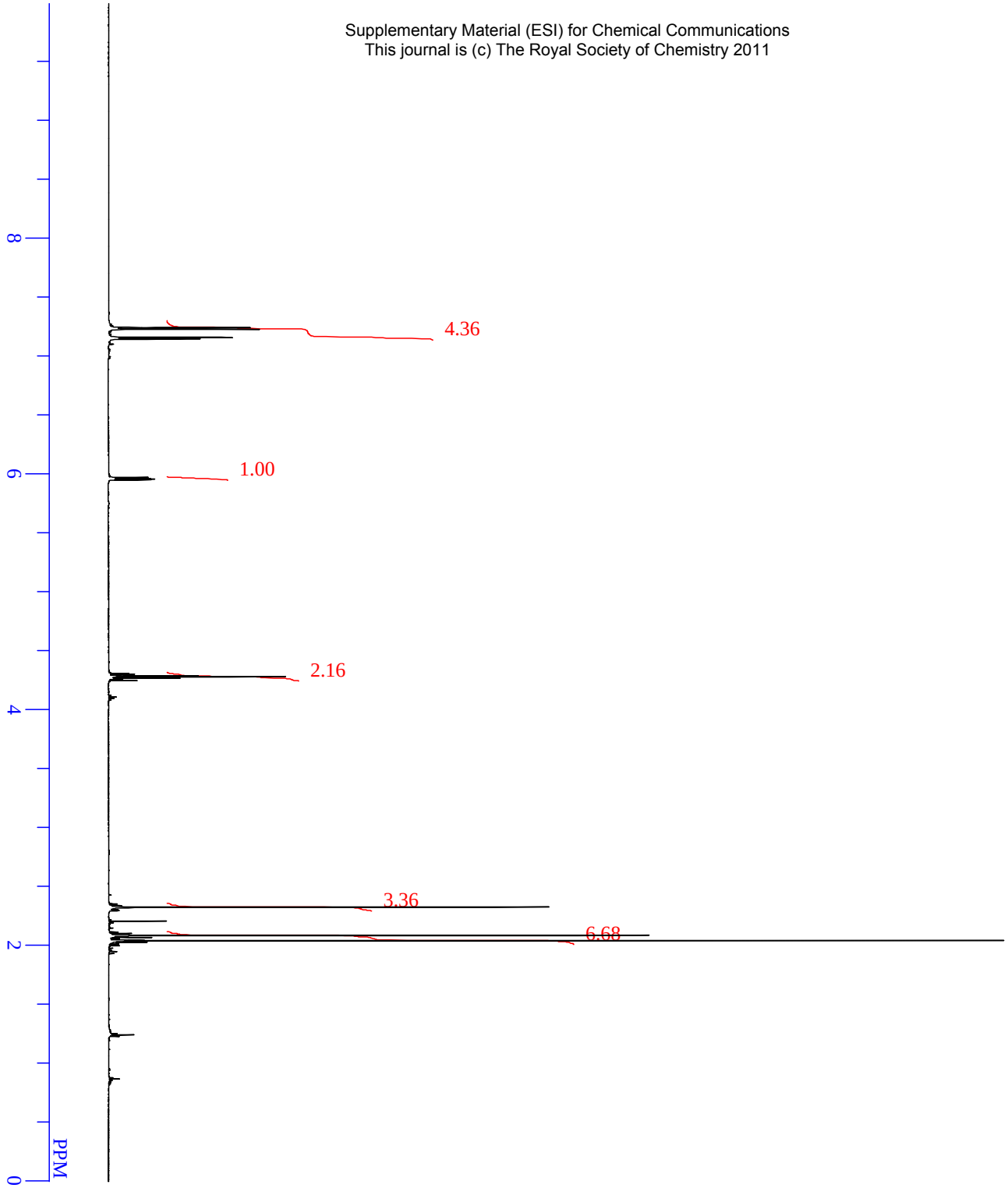
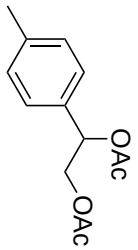
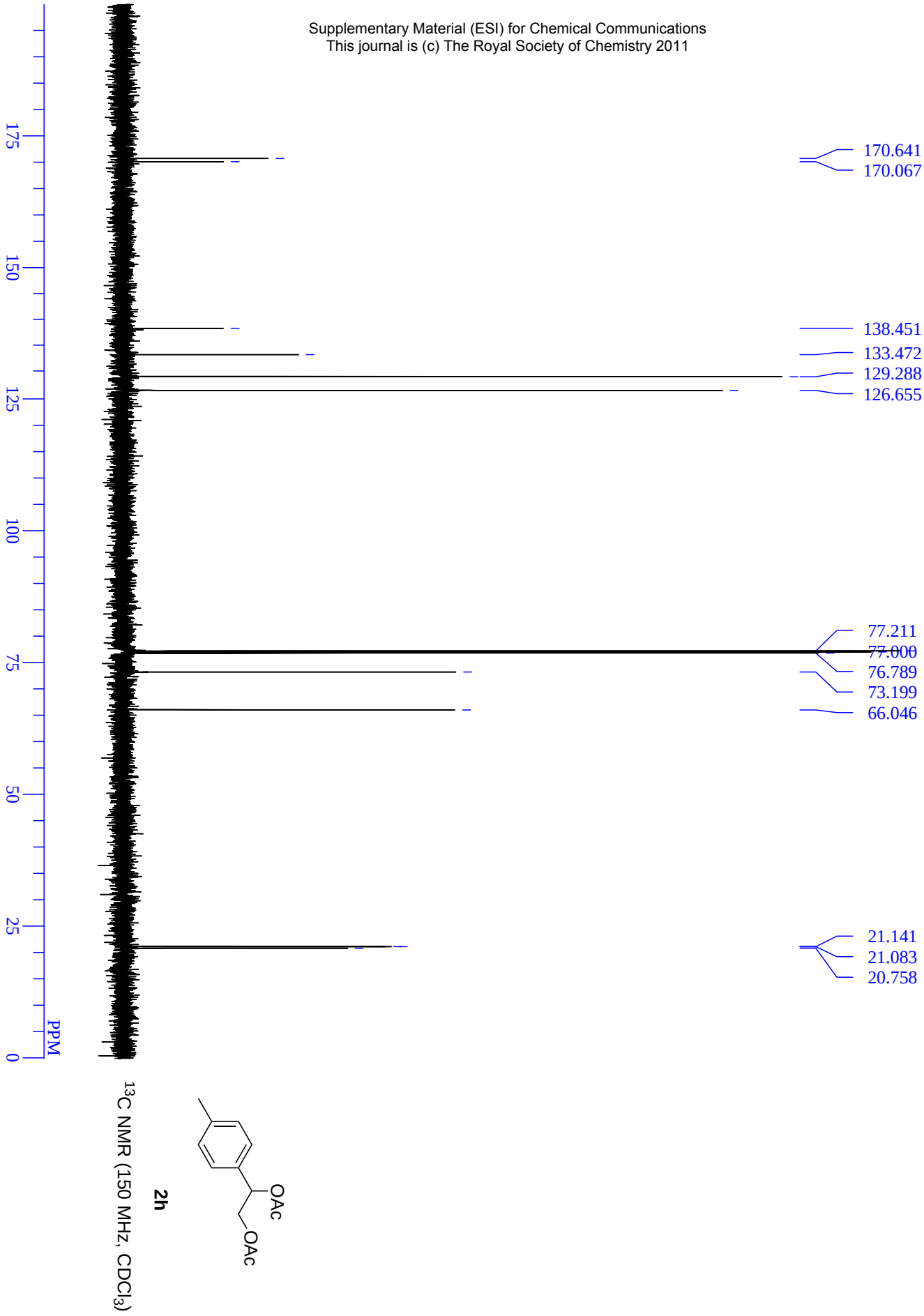




^{1}H NMR (600 MHz, $CDCl_3$)

2h

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170.067
138.451
133.472
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126.655
77.211
77.000
76.789
73.199
66.046
21.141
21.083
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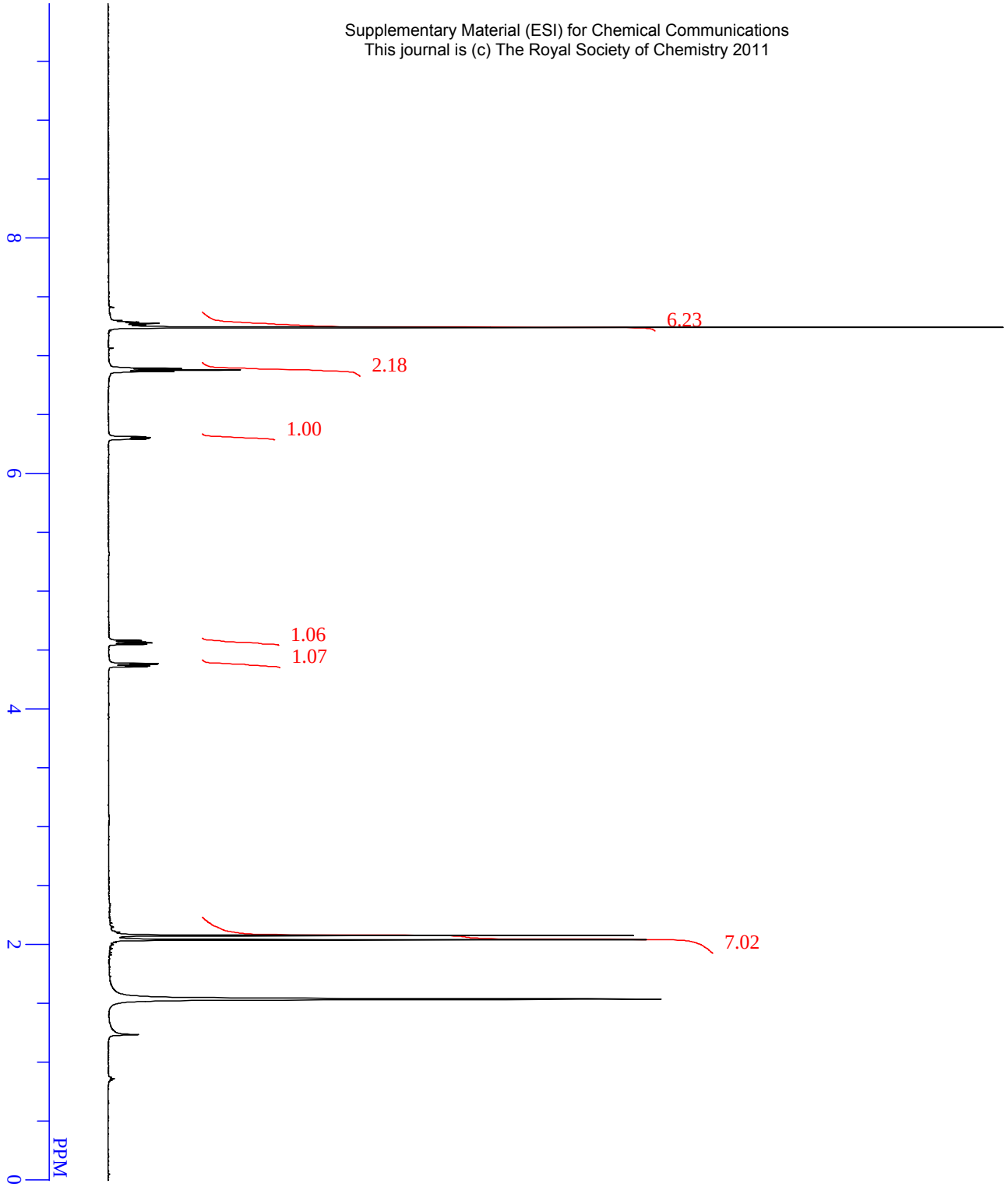
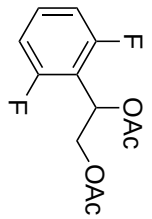
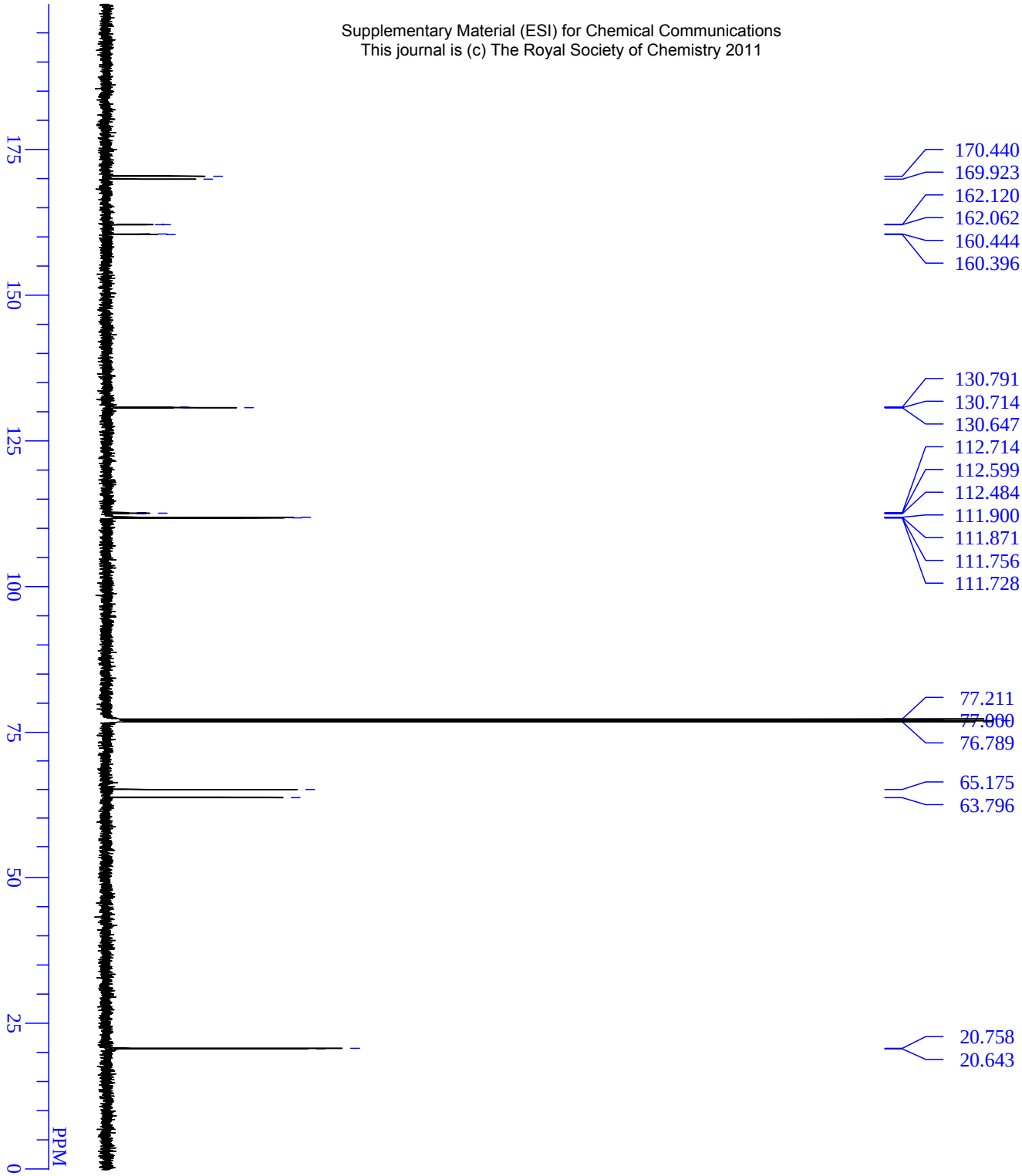
175
150
125
100
75
50
25
0
PPM
^{13}C NMR (150 MHz, CDCl$_3$)
2h
OAc
OAc



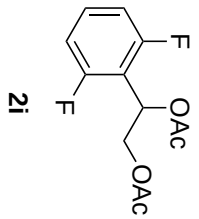
^{1}H NMR (600 MHz, $CDCl_3$)

2i





^{13}C NMR (150 MHz, $CDCl_3$)



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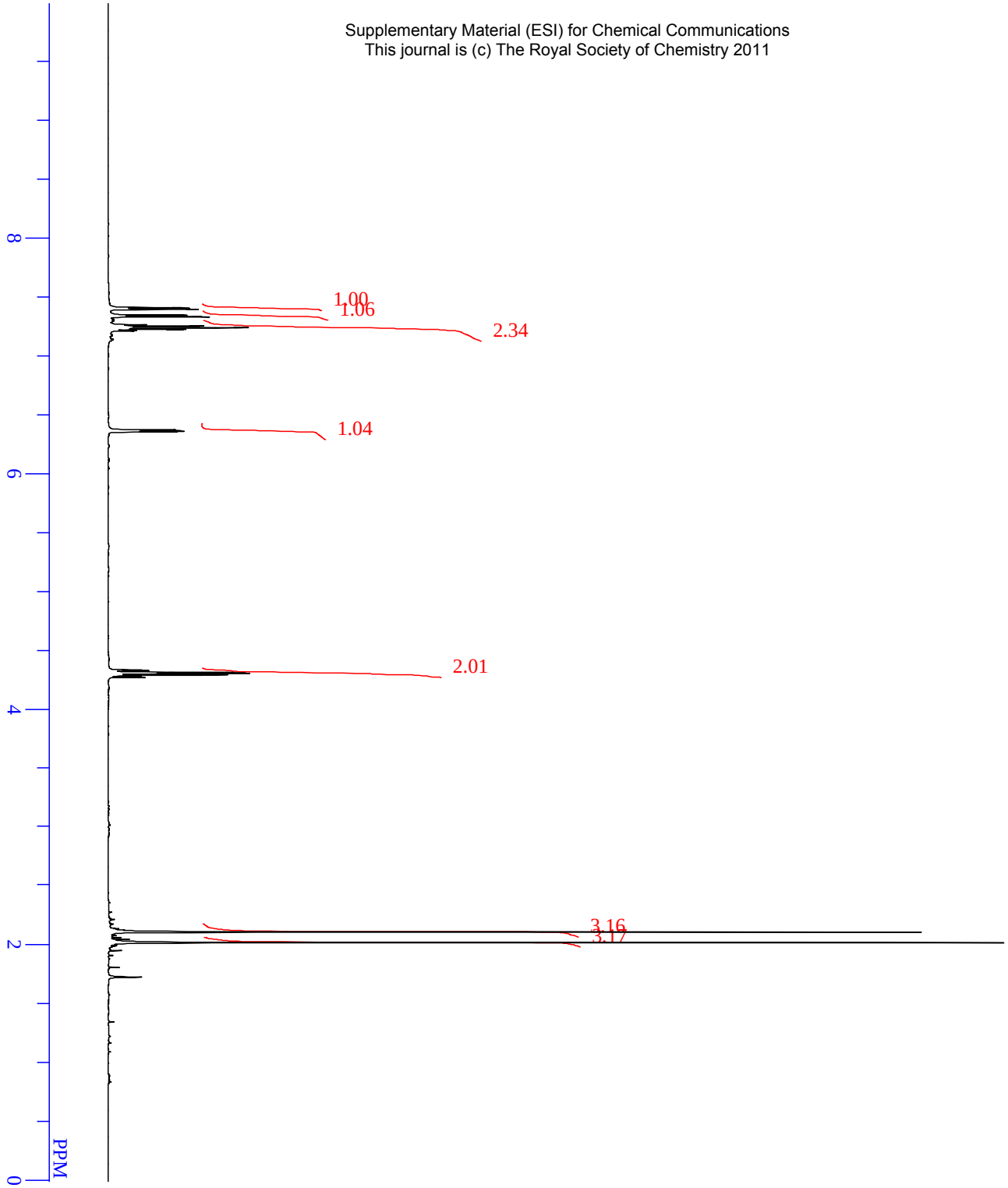

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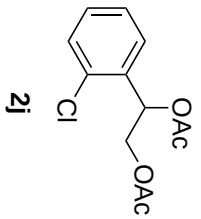

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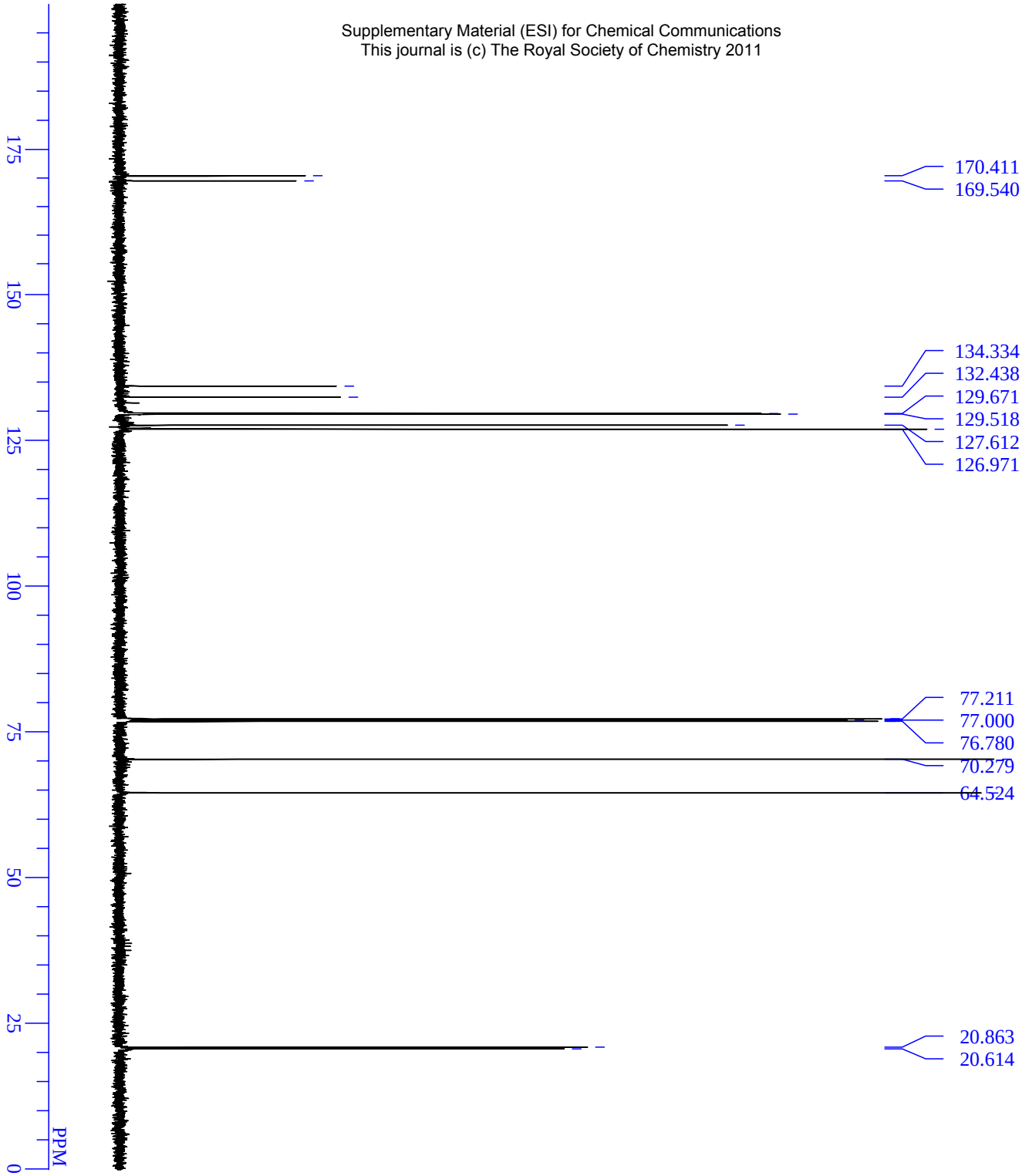
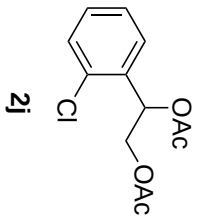


^{13}C NMR (150 MHz, $CDCl_3$)



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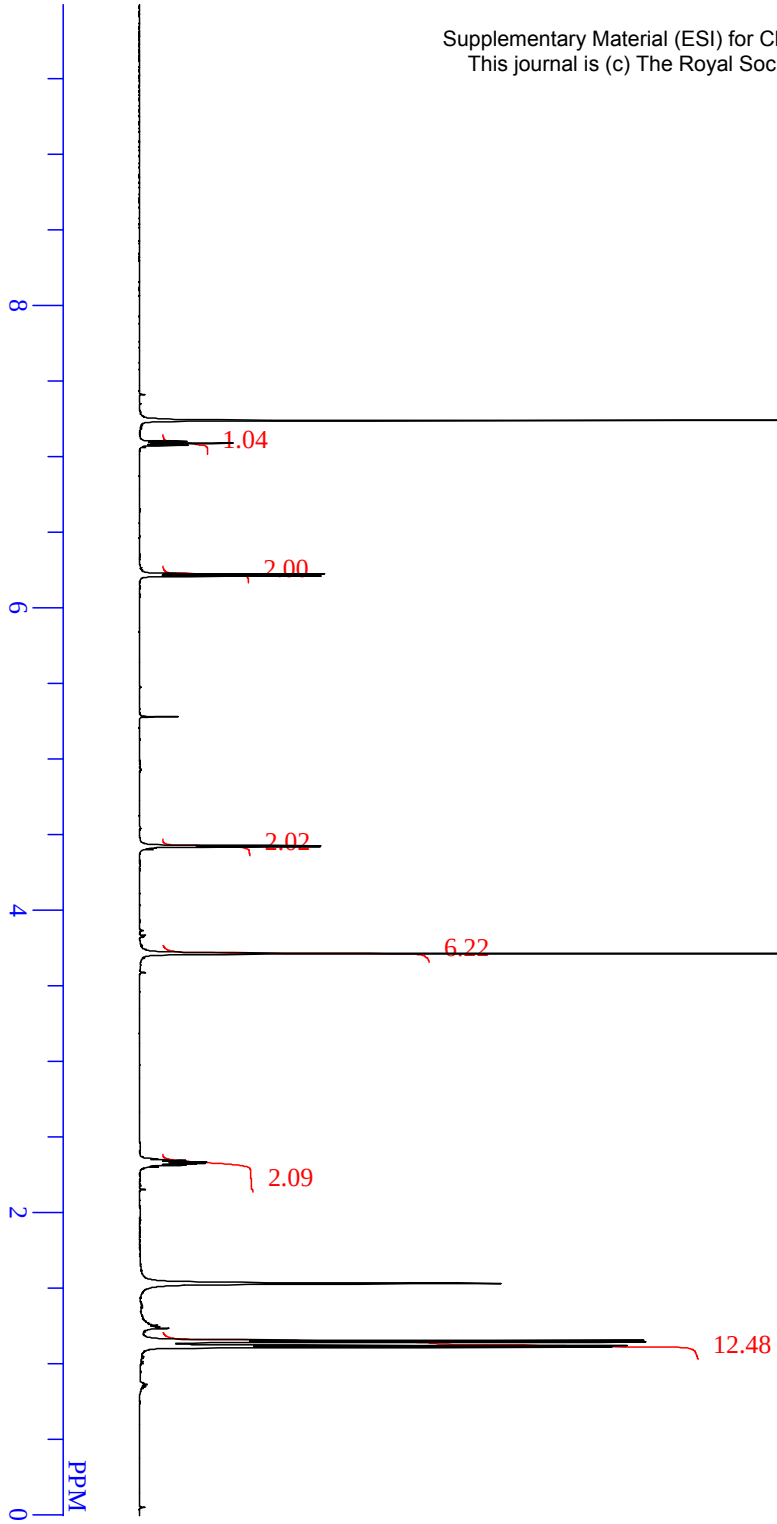
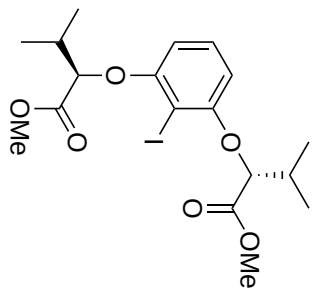
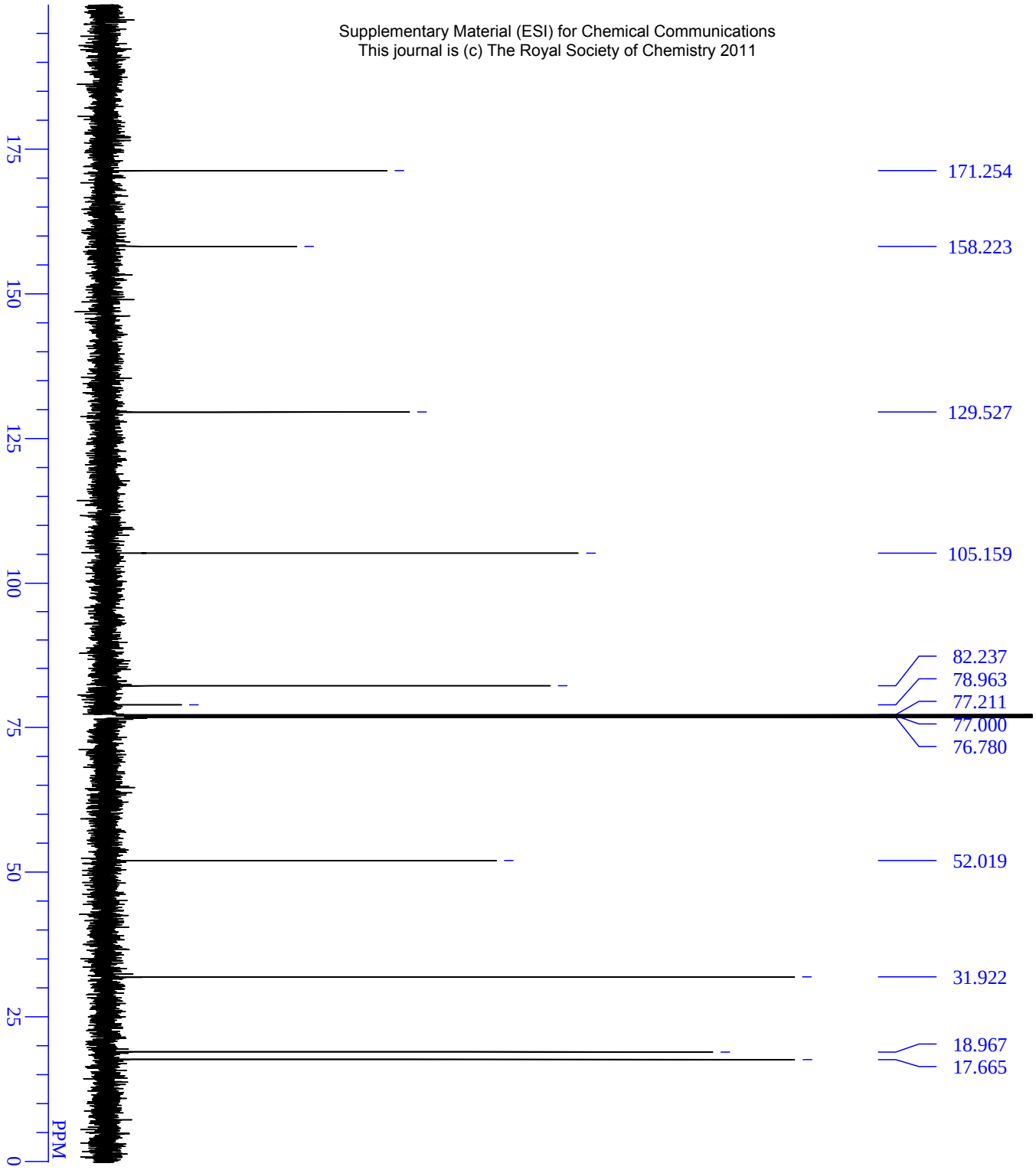


^{1}H NMR (600 MHz, $CDCl_3$)

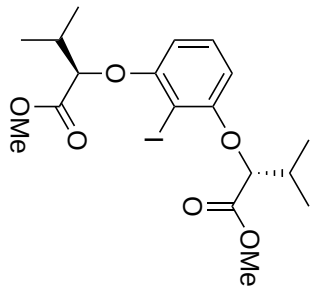
6'

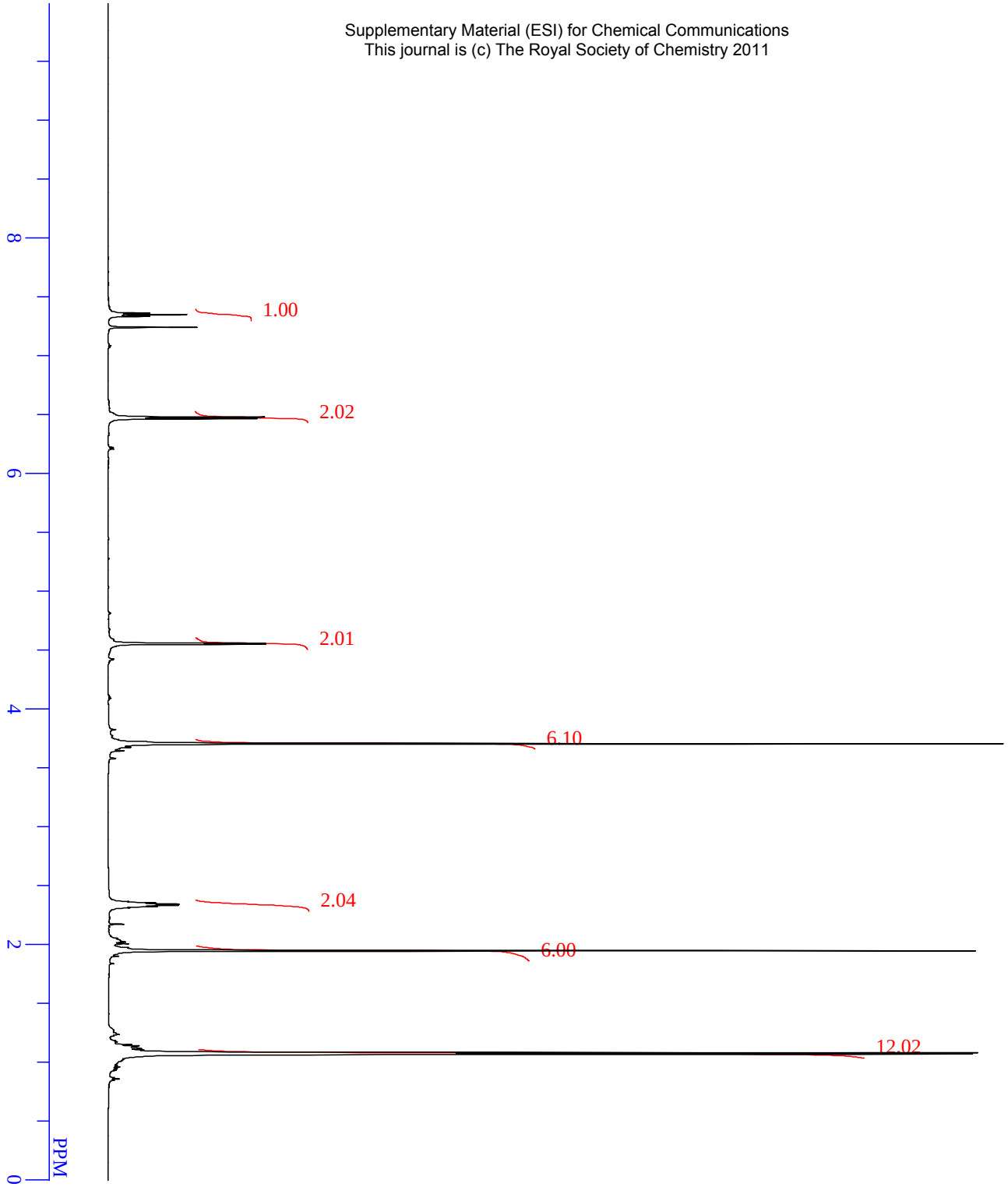




^{13}C NMR (150 MHz, $CDCl_3$)

6'





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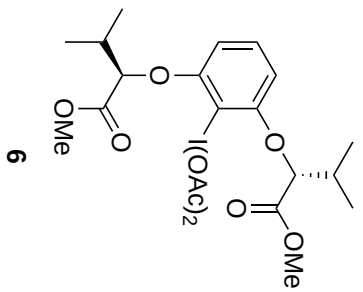

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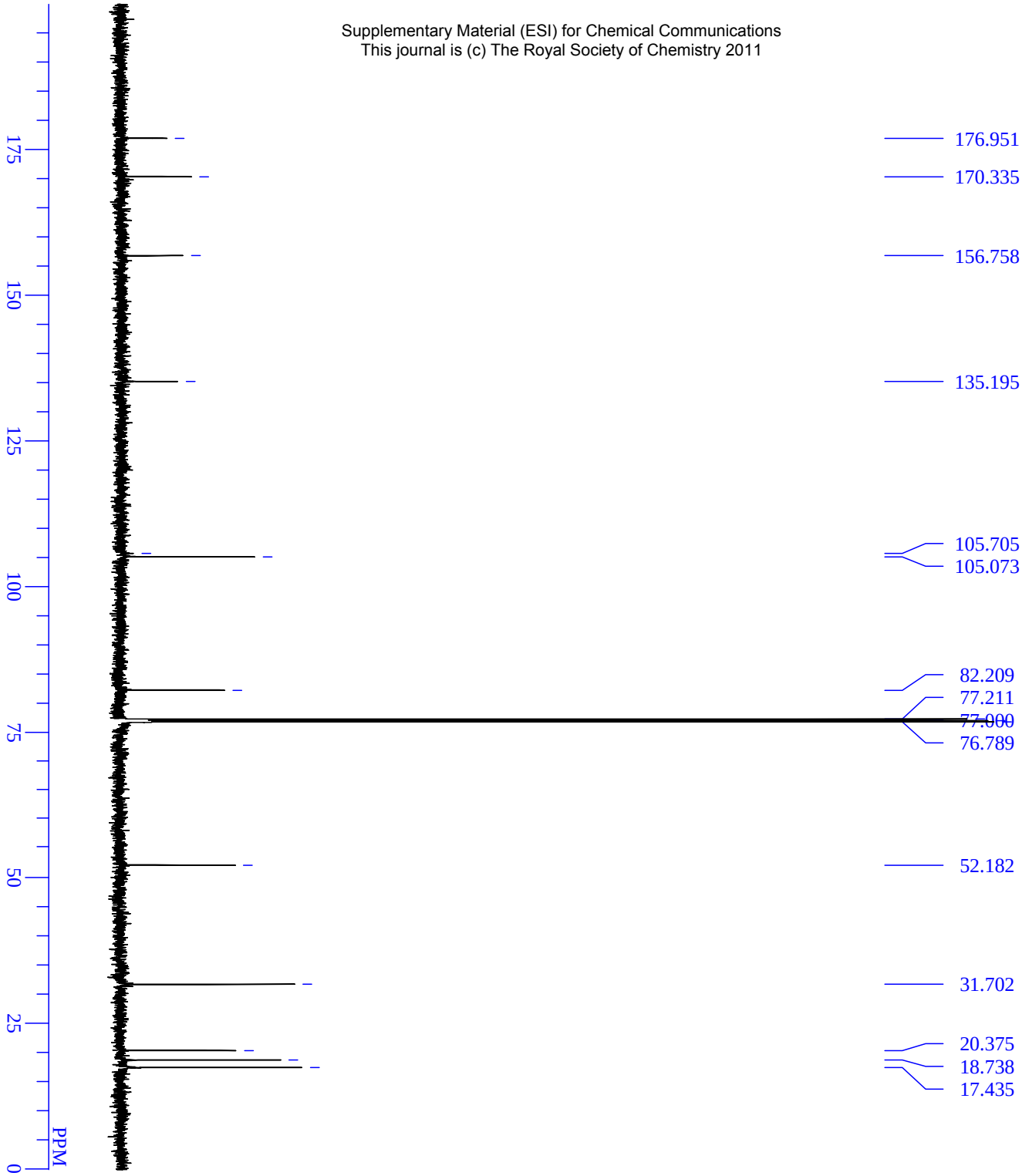
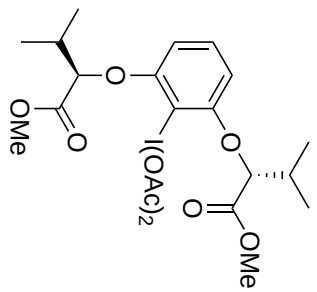


^{13}C NMR (150 MHz, $CDCl_3$)



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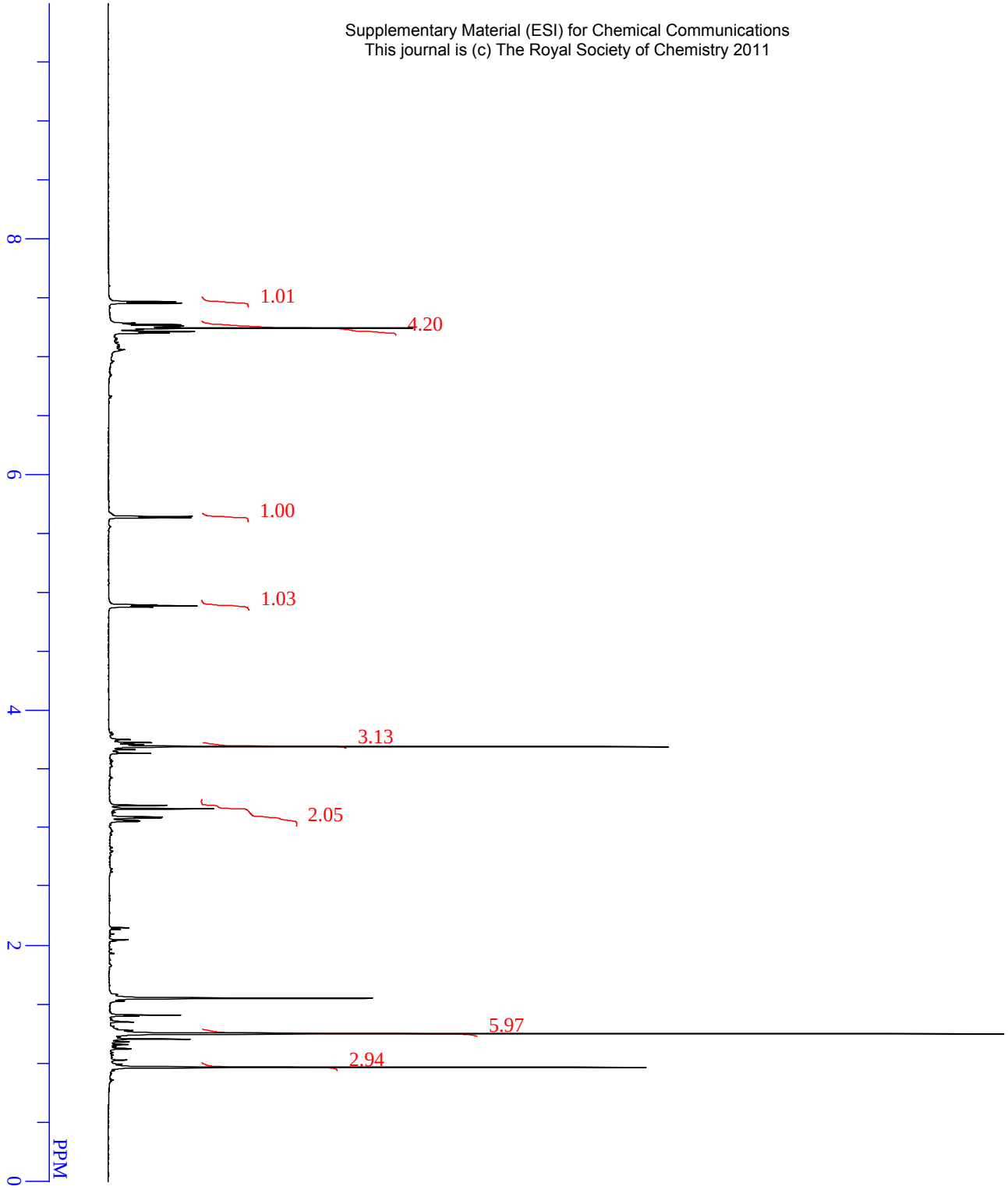
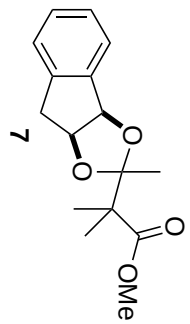
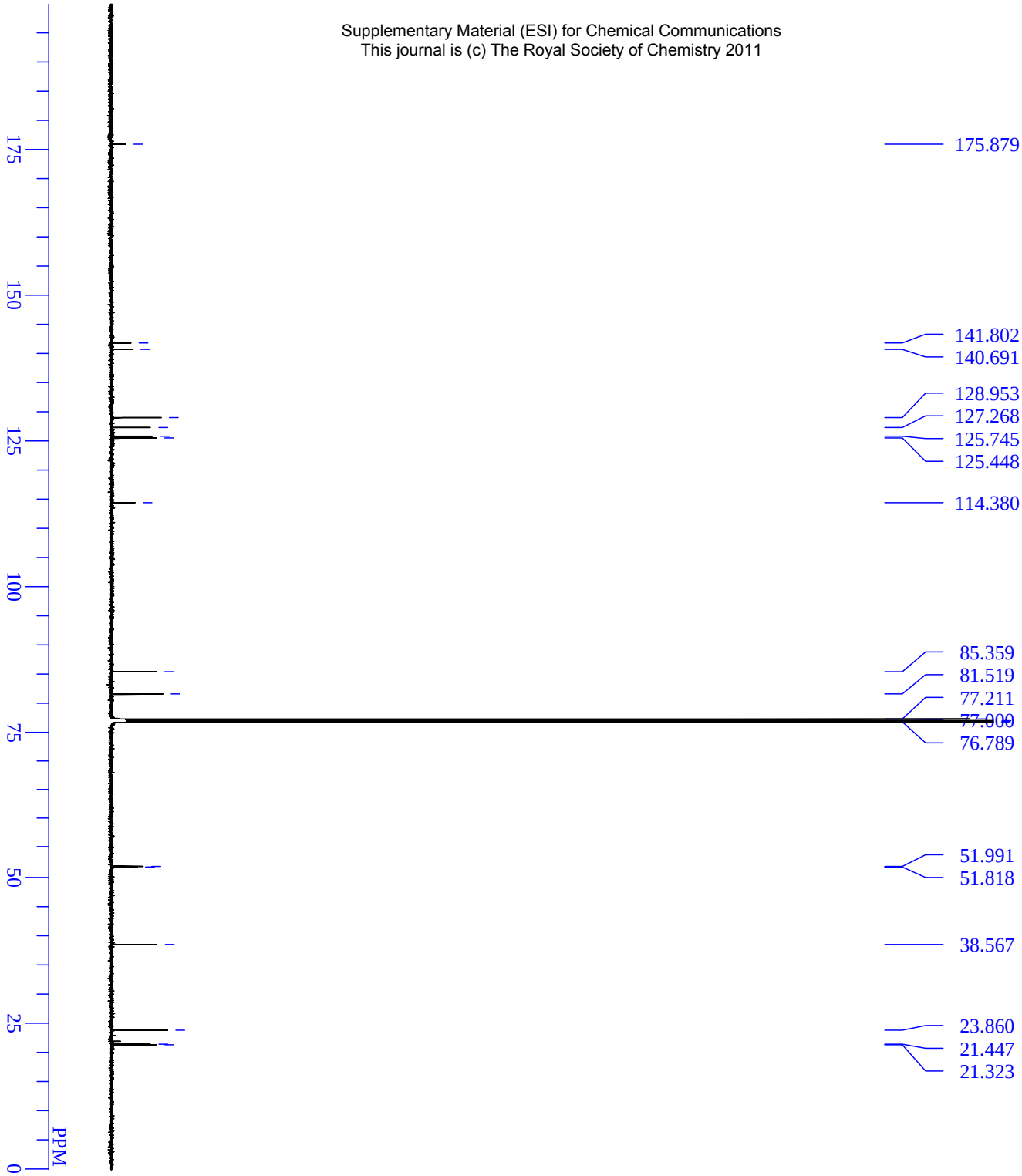


^{1}H NMR (600 MHz, $CDCl_3$)





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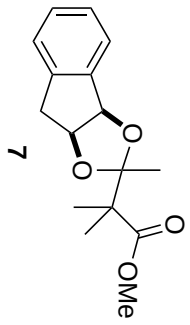
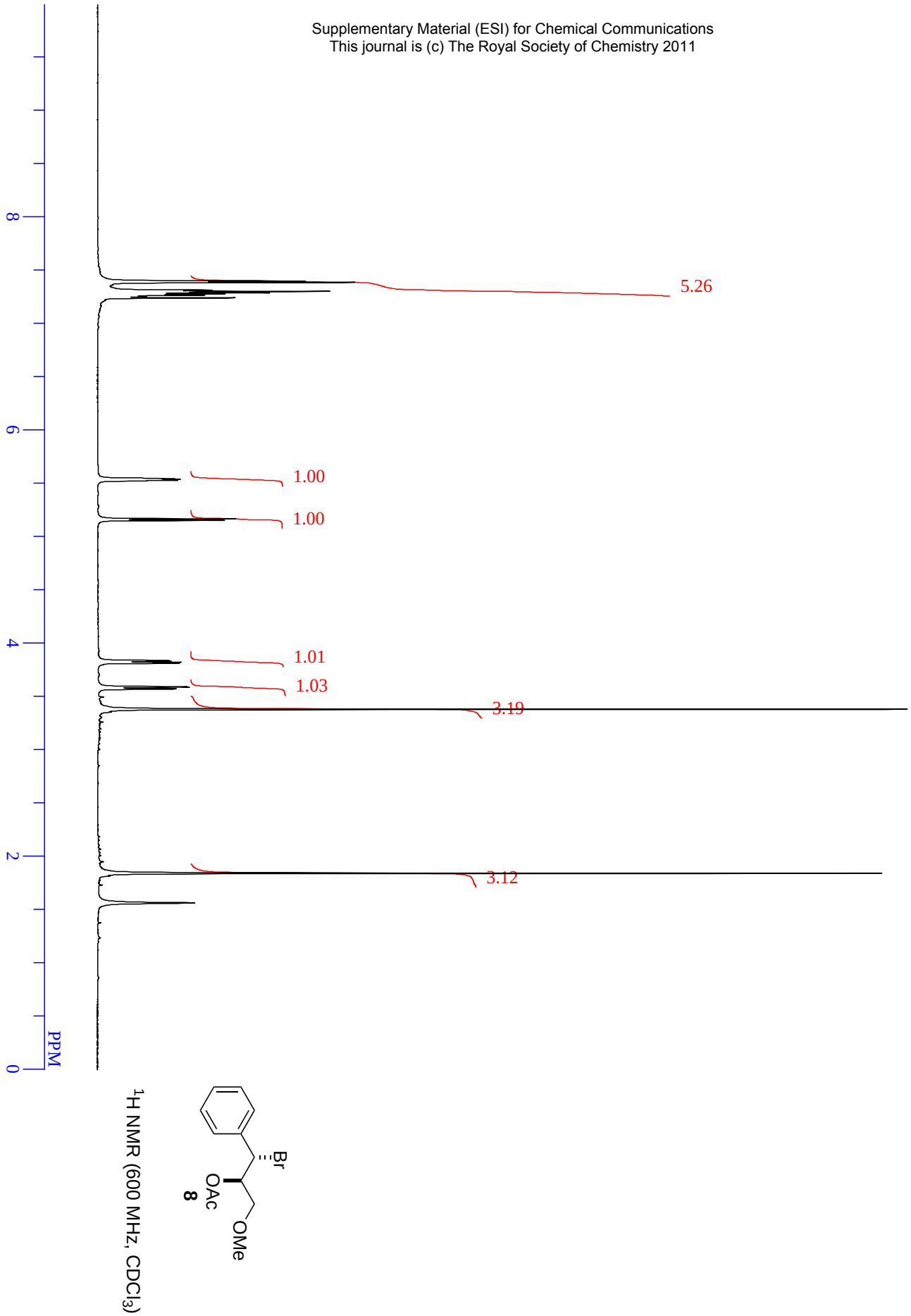
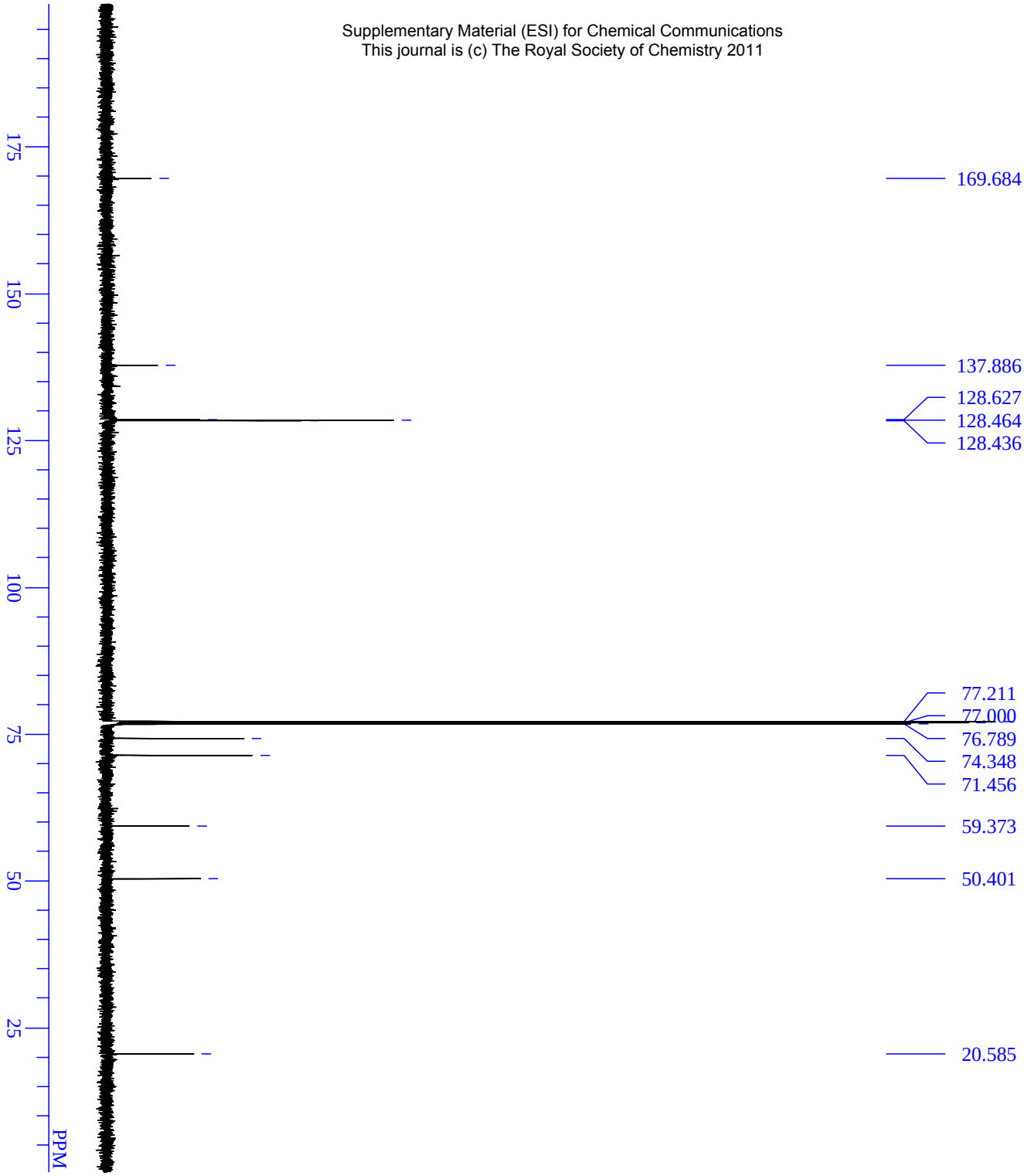




^{1}H NMR (600 MHz, $CDCl_3$)

8



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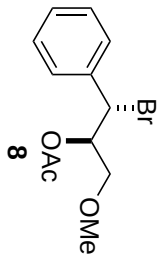
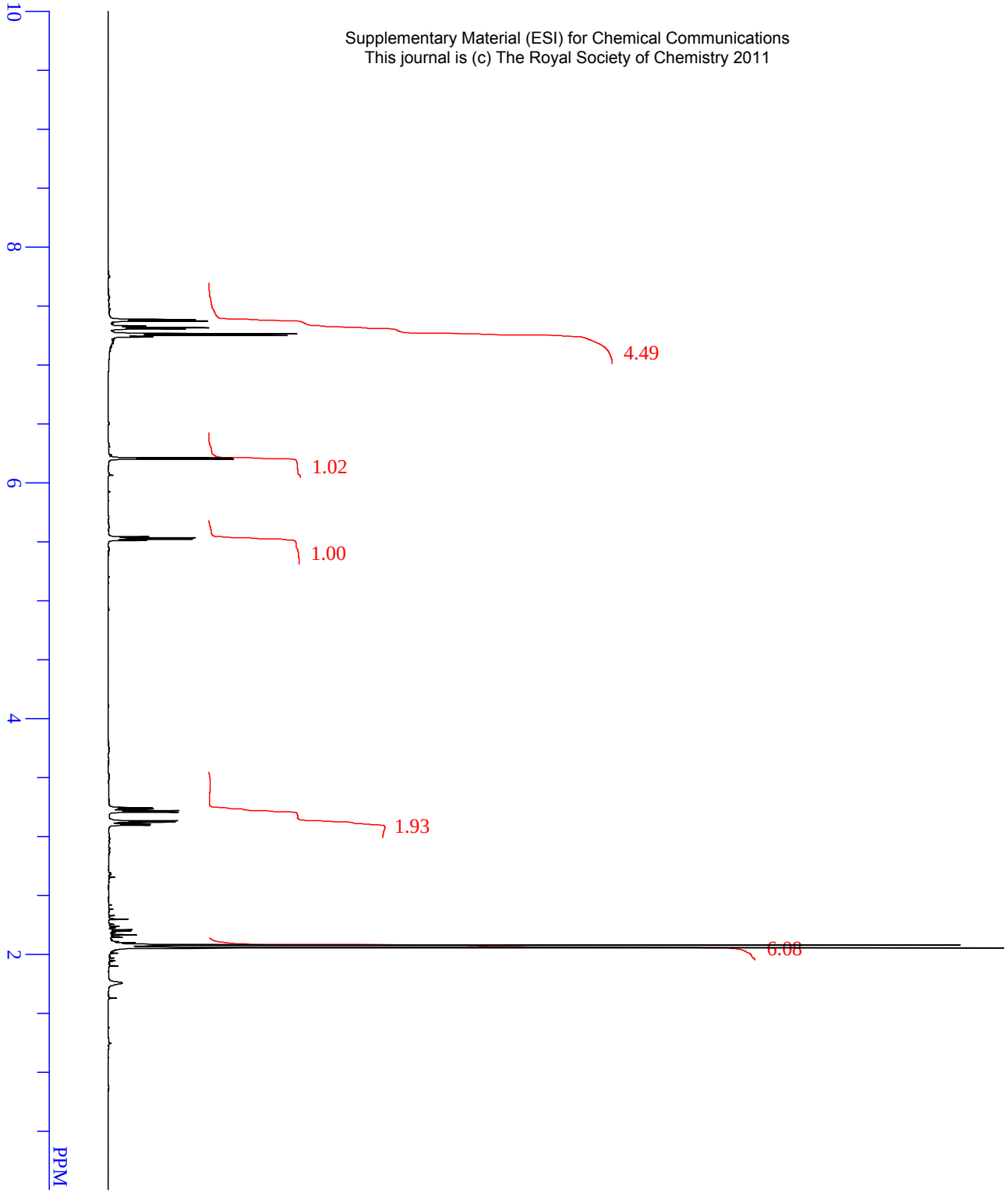
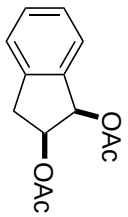


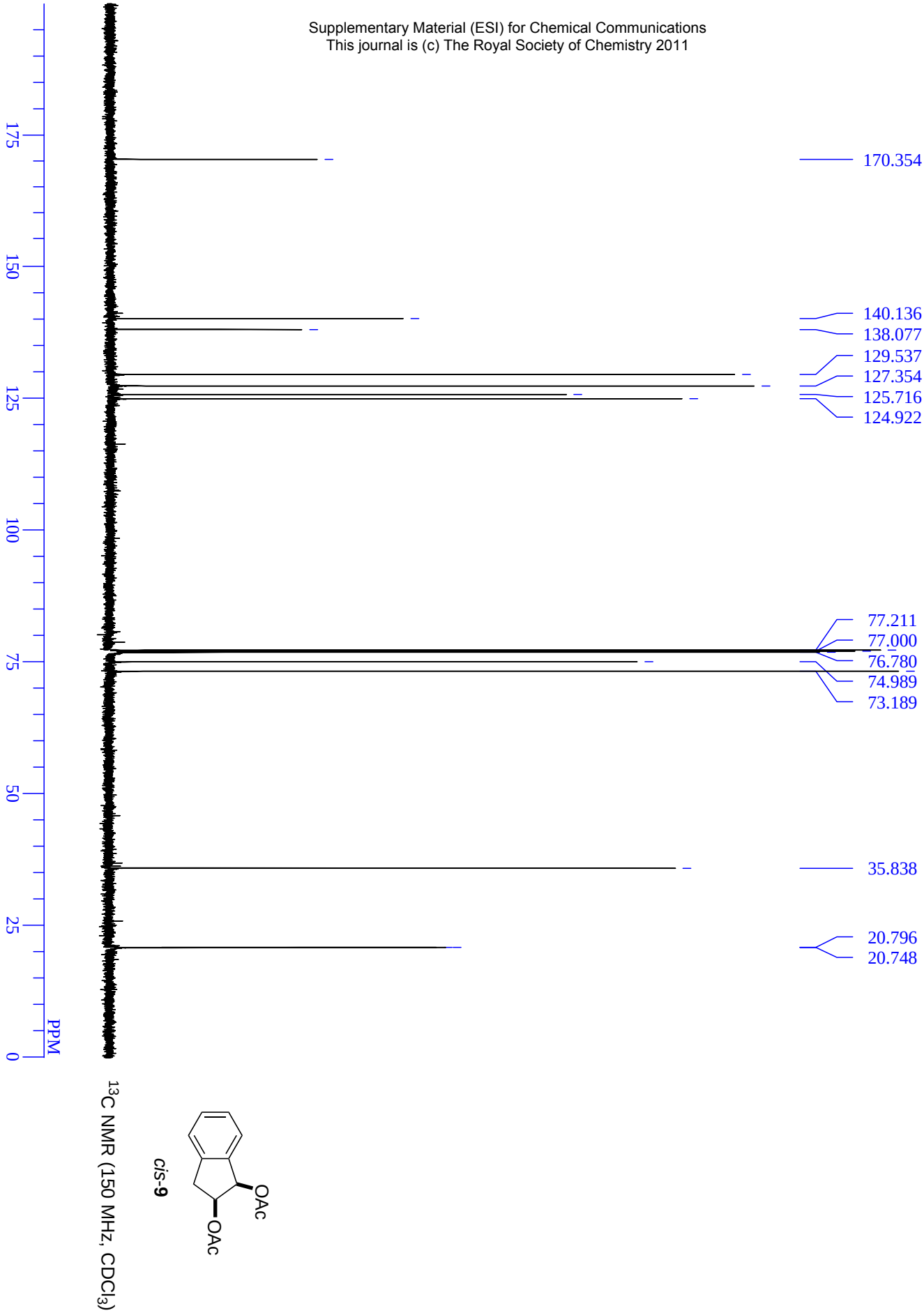
8



^{1}H NMR (600 MHz, $CDCl_3$)

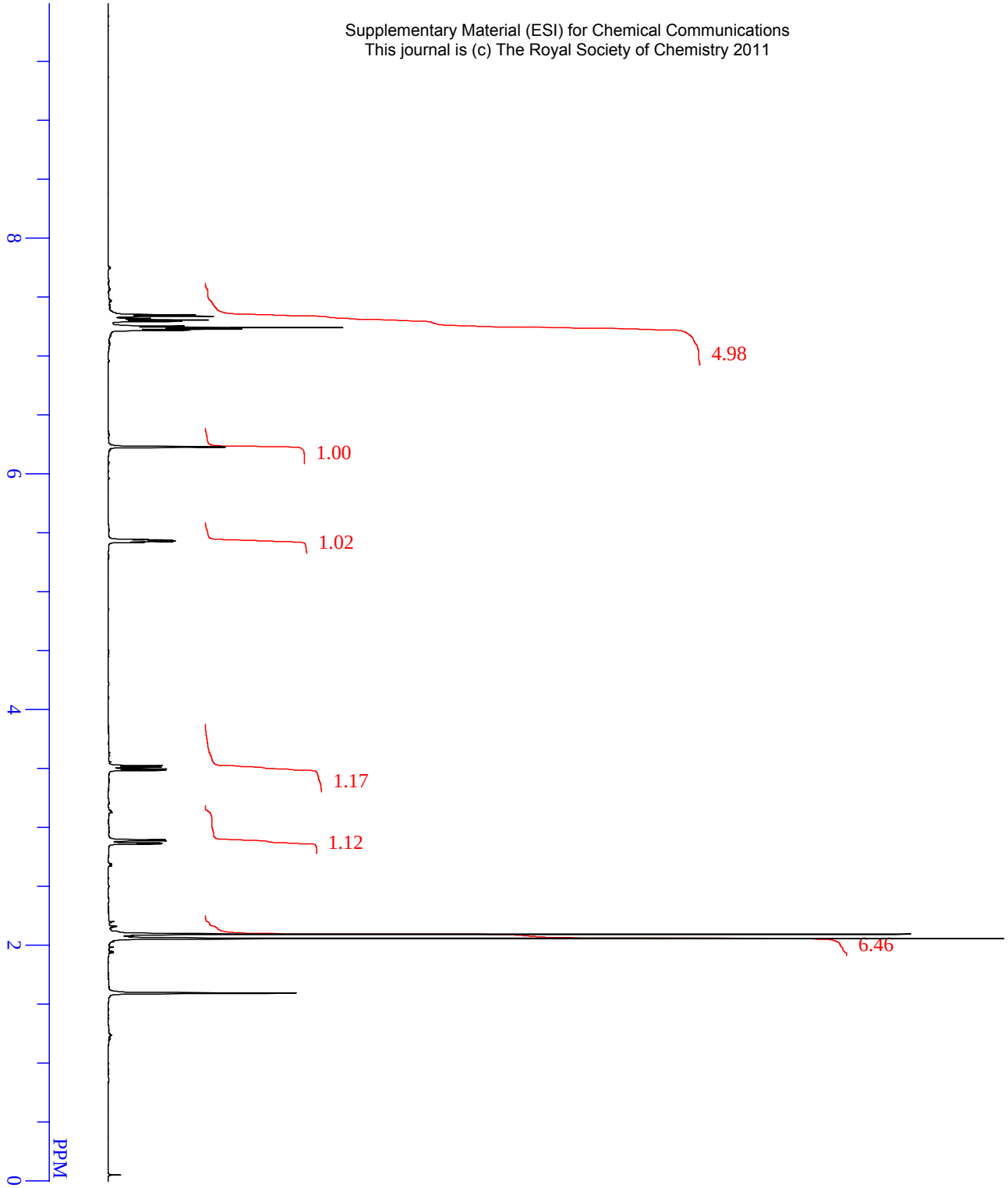
cis-**9**





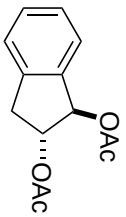
^{13}C NMR (150 MHz, $CDCl_3$)

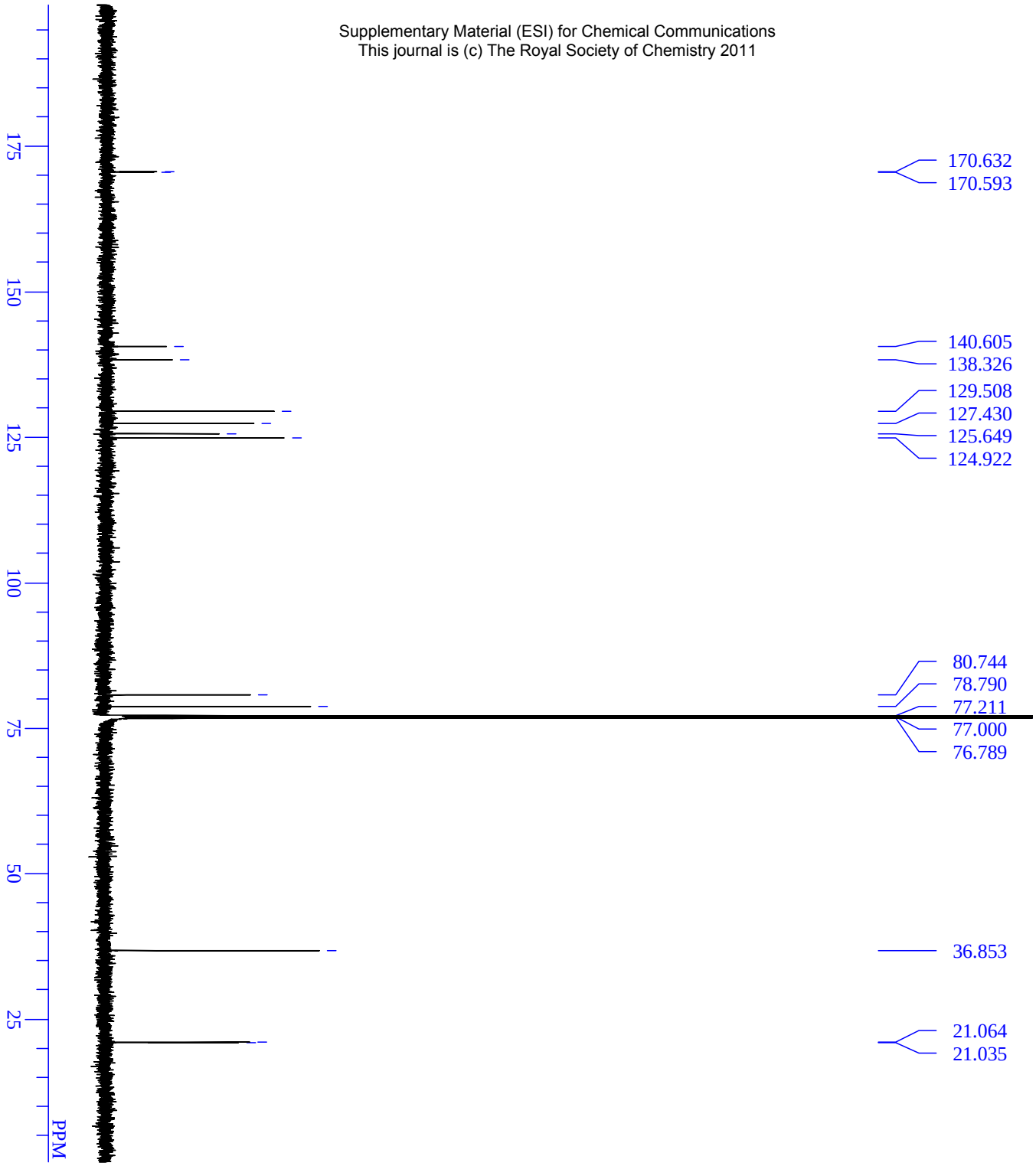
cis-**9**



^{1}H NMR (600 MHz, $CDCl_3$)

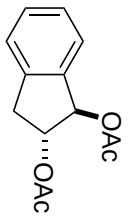
trans-**9**

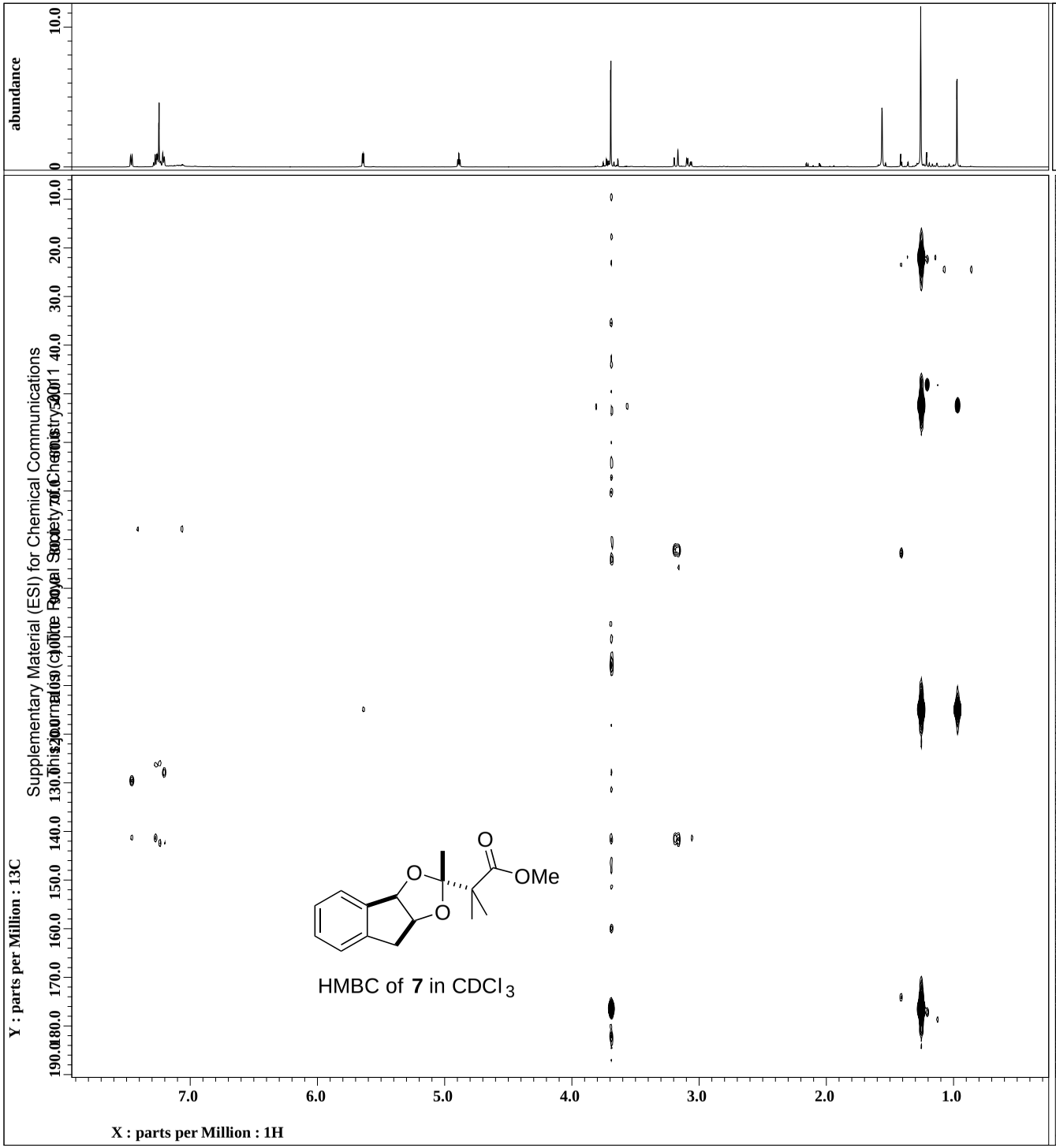




^{13}C NMR (150 MHz, $CDCl_3$)

trans-**9**



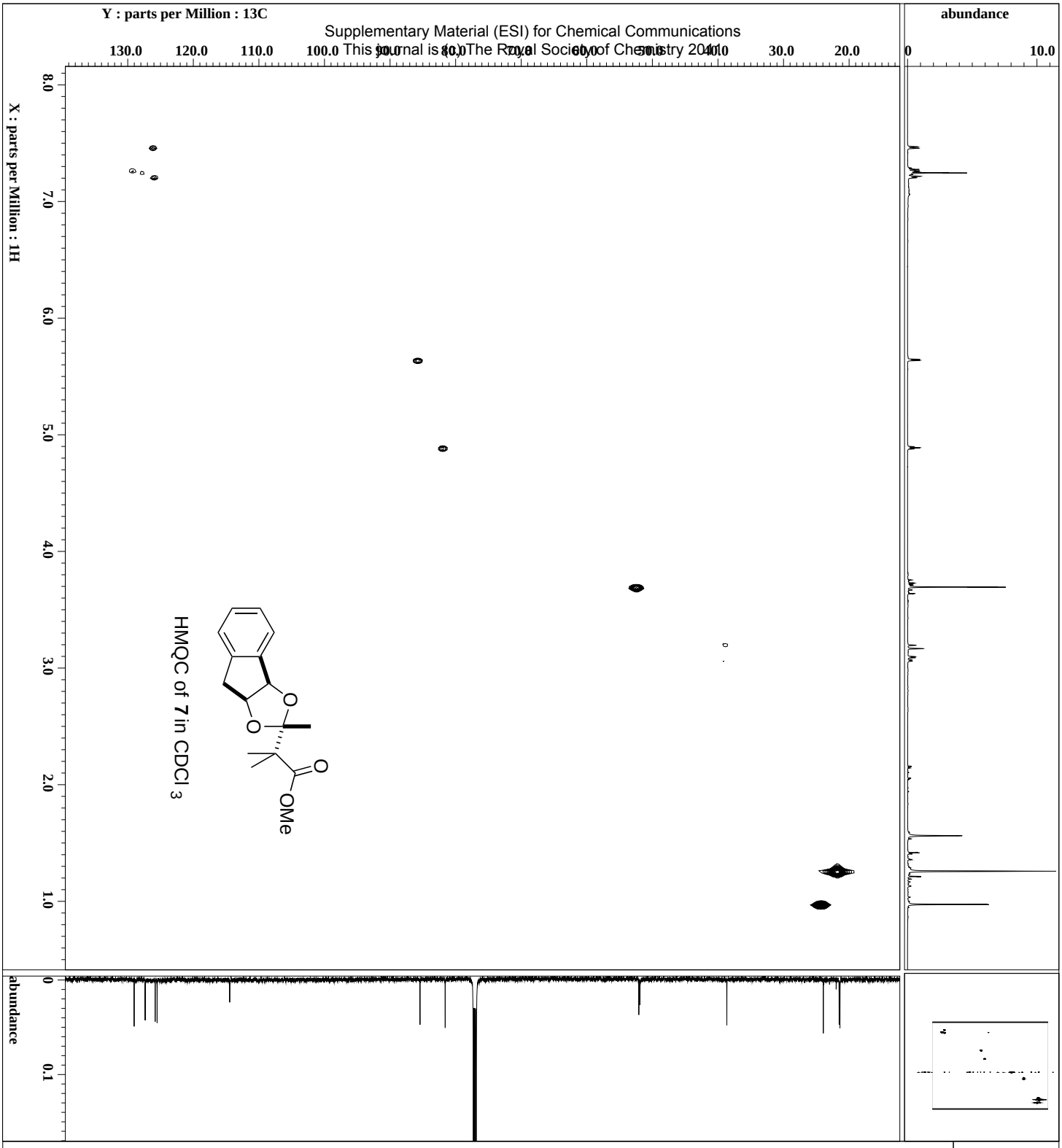



```
Filename          = 2010-mf-026-hmbc-2.jd
Author            = delta
Experiment        = hmbc_pfg.ex2
Sample_id         = 2010-mf-026-hmbc
Solvent           = CHLOROFORM-D
Creation_time     = 22-MAY-2010 11:33:37
Revision_time     = 22-MAY-2010 13:24:25
Current_time      = 22-MAY-2010 13:27:21

Comment           = gradient enhanced HMB
Data_format       = 2D REAL REAL
Dim_size          = 1638, 512
Dim_title         = 1H 13C
Dim_units         = [ppm] [ppm]
Dimensions        = X Y
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.1818624[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 2048
X_prescans        = 4
X_resolution      = 5.49866273[Hz]
X_sweep           = 11.26126126[kHz]
Y_domain          = 13C
Y_freq            = 150.91343039[MHz]
Y_offset          = 100[ppm]
Y_points          = 256
Y_prescans        = 0
Y_resolution      = 147.51699396[Hz]
Y_sweep           = 37.76435045[kHz]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 4
Total_scans       = 1024

X_acq_time        = 0.1818624[s]
X_atn             = 3.5[dB]
X_pulse           = 7.8[us]
Y_acq_time        = 6.77888[ms]
Y_atn             = 4[dB]
Y_pulse           = 21.2[us]
Tri_mode          = Off
Dante_presat      = FALSE
Delta             = 62.5[ms]
Grad_1            = 1[ms]
Grad_1_amp        = 0.18[T/m]
Grad_2            = 1[ms]
Grad_2_amp        = 0.18[T/m]
Grad_3            = 1[ms]
Grad_3_amp        = 90.54325956[mT/m]
Grad_recover      = 0.1[ms]
Grad_selection    = 13C = 1.988:1.98
Grad_shape        = SINE
Initial_wait      = 1[s]
J_constant        = 140[Hz]
Long_range_j      = 8[Hz]
Recvr_gain        = 90
Refocus_comp      = 2.57142857[ms]
Relaxation_delay  = 1.5[s]
Repetition_time   = 1.6818624[s]
T1                = 1[us]
```

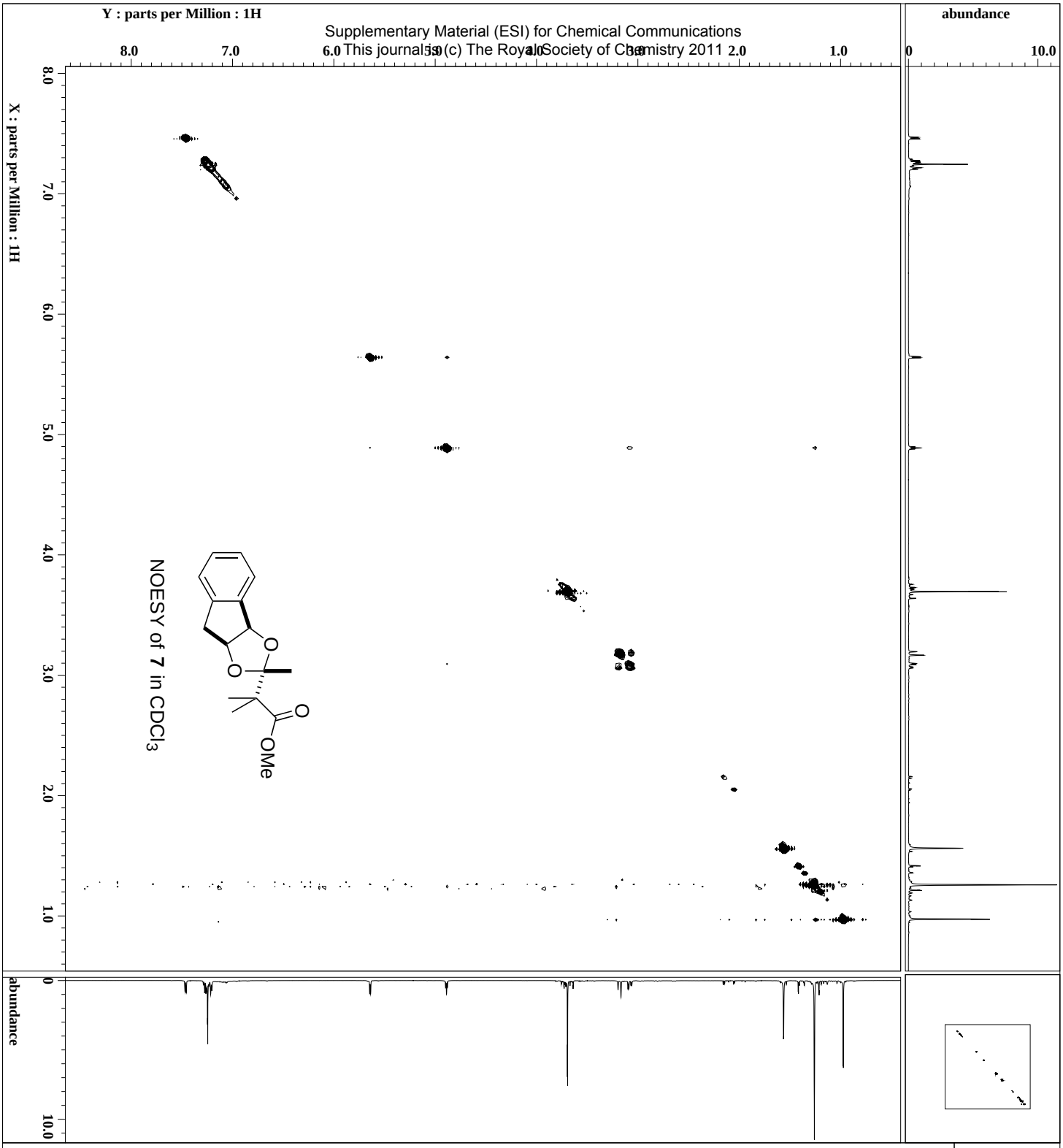



```
Filename                = 2010-mf-026-hmqc-2.jd
Author                  = delta
Experiment              = hmqc_pfg.ex2
Sample_id               = 200-ī0-mf-026-hmqc
Solvent                 = CHLOROFORM-D
Creation_time           = 22-MAY-2010 13:35:25
Revision_time           = 22-MAY-2010 13:51:54
Current_time            = 22-MAY-2010 13:55:06

Comment                 = gradient enhanced HMQ
Data_format             = 2D REAL REAL
Dim_size                = 819, 512
Dim_title               = 1H 13C
Dim_units               = [ppm] [ppm]
Dimensions              = X Y
Site                    = ECA600
Spectrometer            = DELTA2_NMR

Field_strength          = 14.09636928[T] (600[M
X_acq_duration          = 90.9312[ms]
X_domain                = 1H
X_freq                  = 600.1723046[MHz]
X_offset                = 5[ppm]
X_points                = 1024
X_prescans              = 4
X_resolution            = 10.99732545[Hz]
X_sweep                 = 11.26126126[kHz]
Y_domain                = 13C
Y_freq                  = 150.91343039[MHz]
Y_offset                = 85[ppm]
Y_points                = 256
Y_prescans              = 0
Y_resolution            = 100.26309035[Hz]
Y_sweep                 = 25.66735113[kHz]
Tri_domain              = 1H
Tri_freq                = 600.1723046[MHz]
Tri_offset              = 5[ppm]
Clipped                 = TRUE
Mod_return              = 1
Scans                   = 4
Total_scans             = 1024

X_acq_time              = 90.9312[ms]
X_atn                   = 3.5[dB]
X_pulse                 = 7.8[us]
Y_acq_time              = 9.97376[ms]
Y_atn                   = 4[dB]
Y_pulse                 = 21.2[us]
Irr_atn_dec             = 13[dB]
Irr_noise               = MPF8
Tri_mode                = Off
Dante_presat            = FALSE
Decoupling              = TRUE
Grad_1                  = 1[ms]
Grad_1_amp              = 0.18[T/m]
Grad_2                  = 1[ms]
Grad_2_amp              = 0.18[T/m]
Grad_3                  = 1[ms]
Grad_3_amp              = 90.54325956[mT/m]
Grad_recover            = 0.1[ms]
Grad_selection          = 13C = 1.988:1.98
Grad_shape              = SINE
Initial_wait            = 1[s]
J_constant              = 140[Hz]
Recvr_gain              = 90
Refocus_comp            = 2.57142857[ms]
Relaxation_delay        = 1.5[s]
Repetition_time         = 1.5909312[s]
```




```
Filename                = 2010-mf-026-noesy-2.j
Author                  = delta
Experiment              = noesy_phase_pfgz
Sample_id               = 2010-mf-026-fra8-noes
Solvent                 = CHLOROFORM-D
Creation_time           = 21-MAY-2010 20:06:18
Revision_time           = 22-MAY-2010 13:31:19
Current_time            = 22-MAY-2010 13:33:16

Comment                 = phase sensitive noesy
Data_format             = 2D COMPLEX COMPLEX
Dim_size                = 819, 512
Dim_title               = 1H 1H
Dim_units               = [ppm] [ppm]
Dimensions              = X Y
Site                    = ECA600
Spectrometer            = DELTA2_NMR

Field_strength          = 14.09636928[T] (600[M
X_acq_duration          = 90.9312[ms]
X_domain                = 1H
X_freq                  = 600.1723046[MHz]
X_offset                = 5[ppm]
X_points                = 1024
X_prescans              = 4
X_resolution            = 10.9973254[Hz]
X_sweep                 = 11.2612612[kHz]
Y_domain                = 1H
Y_freq                  = 600.1723046[MHz]
Y_offset                = 5[ppm]
Y_points                = 256
Y_prescans              = 0
Y_resolution            = 35.17242932[Hz]
Y_sweep                 = 9.00414191[kHz]
Irr_domain              = 1H
Irr_freq                = 600.1723046[MHz]
Irr_offset              = 5[ppm]
Tri_domain              = 1H
Tri_freq                = 600.1723046[MHz]
Tri_offset              = 5[ppm]
Clipped                 = FALSE
Mod_return              = 1
Scans                   = 4
Total_scans             = 1024

X_acq_time              = 90.9312[ms]
X_atn                   = 3.5[dB]
X_pulse                 = 7.8[us]
X_acq_time              = 28.43136[ms]
Y_pl_correction         = 180
Irr_mode                = Off
Tri_mode                = Off
Dante_presat            = FALSE
Grad_1                  = 1[ms]
Grad_1_amp              = 45[mT/m]
Initial_wait            = 1[s]
Mix_time                = 1.5[s]
Recvr_gain              = 52
Relaxation_delay        = 1.5[s]
Repetition_time         = 1.5909312[s]
Scramble                = 1[ms]
T1                      = 45.546[us]
T1_cal1                 = 45.546[us]
T1_cal2                 = 0.101076[ms]
T1_cal3                 = 0.156606[ms]
T1_cal4                 = 0.212136[ms]
T1_cal5                 = 0.267666[ms]
T1_cal6                 = 0.323196[ms]
```