Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Generation and alkylation of 2-boryl allylic sulfone anions

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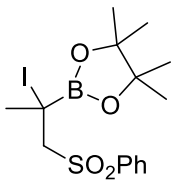
General

All glassware and needles were oven-dried and allowed to cool in a desiccator prior to use. Tetrahydrofuran (THF) was purchased from Fisher Scientific and distilled from sodium-benzophenone prior to use. Diisopropylamine and dichloromethane were distilled from CaH_2. Organolithium bases *n*-butyl lithium (*n*BuLi) and lithium hexamethyldisilazid (LiHMDS) were purchased from Aldrich. *n*BuLi was titrated with freshly recrystallized diphenylacetic acid prior to use. The starting material, 2-isopropenyl boronic acid pinacol ester, was donated from Frontier Scientific and used as received. Alkyl halides were purchased from commercial sources or prepared from corresponding alcohols and distilled prior to use. Alkylation products were purified by flash chromatography using 230-400 mesh silica purchased from Aldrich and, if crystalline, were recrystallized from hexane-diethyl ether until a constant melting point was observed. All compounds were characterized by ^{1}H, and ^{13}C Nuclear Magnetic Resonance (NMR) spectroscopy using a 500 MHz Bruker instrument. Proton spectra were reported in δ units, parts per million (ppm), relative to trimethylsilane internal standard (0.00 ppm). Carbon spectra were recorded in ppm relative to deuterated chloroform peak (77.16 ppm) or (39.52 ppm) where DMSO was used. Samples were further characterized using mass spectroscopy on a Bruker Apex-Qe instrument. Infrared spectra were recorded on a Thermo Nicolet NEXUS 670 FT-IR instrument.

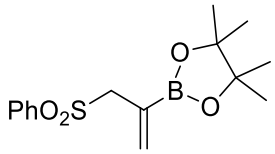
Experimental Procedures

Synthesis of Borylated Allylic Sulfone



10

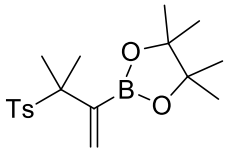
2-(2-iodo-1-(phenylsulfonyl)propan-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (10): Benzenesulfinic acid (6.44 g, 39.2 mmol) was dissolved in 200 mL water and treated with a saturated solution of iodine (10.0 g, 39.3 mmol) dropwise. Once the addition was complete, the organic layer was separated and dried over $MgSO_4$ for 2 h in the dark. The red solution of sulfonyl iodide was added dropwise to a solution of 2-isopropenyl boronic acid pinacol ester (6.0 g, 35.7 mmol) dissolved in 100 mL dry dichloromethane and stirred for 12 h at room temperature. The reaction mixture was washed with $Na_2S_2O_3$, brine and the organic layer was dried over $MgSO_4$. The solvent was removed under rotary evaporator and the remaining solid was recrystallized from hexanes-diethyl ether (13.2 g, 77%). mp = 106 – 107 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, J = 8 Hz, 2H), 7.65 (t, J = 7.5, 1H), 7.56 (t, J = 7.5, 2H), 4.10 (d, J = 13 Hz, 1H), 3.84 (d, J = 13 Hz, 1H), 2.26 (s, 3H), 1.36 (d, J = 8.5 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 141.16, 133.88, 129.46, 127.81, 84.90, 69.56, 29.48, 24.57, 24.50; HRMS calcd for $(C_{15}H_{22}BIO_4S)Na^+$ 459.0268; Found: 459.0266.



7

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)prop-1-en-2-yl)-1,3,2-dioxaborolane (7): A solution **15** (13.6 g, 31.3 mmol) in dry chloroform (125 mL) was treated with triethylamine (8.7 mL, 62.5 mmol) and heated to reflux for four days. The reaction mixture washed with 0.5 M HCl, water and brine. The organic layer was dried over MgSO4 and concentrated to give a solid which was recrystallized from hexanes-diethyl ether (7.3 g, 76%). mp = 105- 107 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, J = 8 Hz, 2H), 7.61 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 7.5 Hz, 2H), 6.09 (d, J = 2.5 Hz, 1H), 5.78 (s, 1H), 3.93 (s, 2H), 1.15 (s, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.90, 138.68, 133.54, 129.14, 128.97, 84.26, 60.76, 24.78; IR (film) ν_{max} = 2996, 2983, 1613, 1446, 1440, 1392, 1383, 1332, 1304, 1245, 1168, 1139, 1123, 1084, 979, 861, 768, 702, 690, 651 cm^{-1}; HRMS calcd for $(C_{15}H_{21}BO_4S)Na^+$ 331.1145; Found: 331.1140.

Alkylation of 3



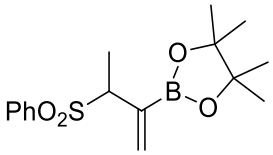
6

4,4,5,5-tetramethyl-2-(3-methyl-3-tosylbut-1-en-2-yl)-1,3,2-dioxaborolane (**6**): Borylated allylic sulfone **3** (100 mg, 0.310 mmol) was added to a 10 mL flask under argon atmosphere and dissolved in 3.0 mL dry THF. The solution was cooled to -78°C in a dry-ice/acetone bath and treated with 4.2 equivalents of LiHMDS (~1 M in hexanes, 1.30 mL, and 1.30 mmol). The solution was allowed to stir for 1 h before addition of methyl iodide (184 mg, 1.30 mmol). Once

the addition was complete, the reaction as allowed to warm to room temperature over 3 hours. The reaction was quenched with a saturated solution of aqueous NH_4Cl and the mixture was extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was purified on a flash column (20% EtOAc in Hexanes) affording **6** as a colorless semisolid (60 mg, 55% yield). mp = 102 – 103 ^oC ; ^1H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, J = 7.5 Hz, 2H), 7.27 (d, J = 7.5 Hz, 2H), 5.81 (d, J = 1.5 Hz, 1H), 5. 48 (s, 1H), 2.41 (s, 3H), 1.54 (s, 6H), 1.29 (s, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 144.21, 132.43, 131.38, 130.99, 128.98, 83.79, 67.13, 24.86, 21.74, 21.52 ; IR (film) ν_{max} = 2977, 2935, 1488, 1415, 1380, 1297, 1233, 1214, 1009 cm^{-1}; HRMS calcd for $(C_{18}H_{27}BO_4S)Na+$ 373.1615; Found: 373.1610.

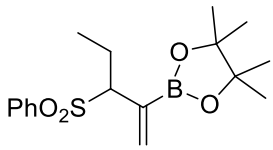
General Procedure for the Alkylation of 7

To a solution of diisopropylamine (60 µL , 0.42 mmol) in THF (2.2 mL) was added *n*butyl lithium (2.04 M in THF, 174 µL, 0.35 mmol) at -78 ^oC and stirred for one hour. The solution of lithium diisopropylamine was then treated with a solution of **7** (100 mg, 0.324mmol) in 1.0 mL THF and stirred for an additional hour before addition of electrophile (0.356 mmol). After dropwise addition of electrophile, the reaction was removed from cold bath and allowed to warm to room temperature over 3 h. The reaction was quenched with a saturated solution of aqueous NH_4Cl and the mixture was extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was absorbed onto silica and purified via flash chromatography (20% EtOAc in Hexanes) affording the corresponding mono-alkylated product.



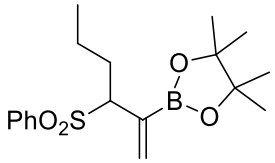
8a

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)but-1-en-2-yl)-1,3,2-dioxaborolane (**8a**): mp = 104 – 105 °C ; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8 Hz, 2H), 7.60 (t, *J* = 7 Hz, 1H), 7.50 (t, *J* = 8 Hz, 2H), 6.09 (d, *J* = 2 Hz, 1H), 5.85 (s, 1H), 4.11 (q, *J* = 7 Hz, 1H), 1.49 (d, *J* = 7.5 Hz, 3H), 1.19 (d, *J* = 3 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 137.70, 135.03, 133.44, 129.86, 18.74, 84.11, 62.62, 24.87, 24.77, 13.30 ; IR (film) ν_{max} = 2963, 2929, 1490, 1423, 1383, 1373, 1300, 1249, 1215, 1002 ,975 cm^{-1}; HRMS calcd for $(C_{16}H_{23}BO_4S)Na^+$ 345.1302 ; Found: 345.1297.



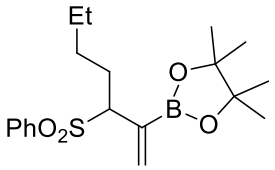
8b

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)pent-1-en-2-yl)-1,3,2-dioxaborolane (8b): mp = 145 – 146 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8 Hz, 2H), 7.58 (t, *J* = 7 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 6.14 (s, 1H), 5.87 (s, 1H), 3.84 (dd, *J* = 3.5, 11.5 Hz, 1H), 2.22 (dq, *J* = 7, 3.5 Hz, 1H), 1.96 (dq, *J* = 11.5, 7 Hz, 1H), 1.15 (s, 12H), 0.915 (t, *J* = 7 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.41, 136.33, 133.30, 129.75, 128.71, 83.98, 24.77, 24.73, 20.57, 11.55; IR (film) ν_{max} = 2977, 2936, 1447, 1423, 1380, 1372, 1317, 1305, 1249, 1215, 1081,969, 862, 840,755, 720, 689, 671, 651 cm^{-1}; HRMS calcd for $(C_{17}H_{25}BO_4S)Na^+$ 359.1458; Found: 359.1454.



8c

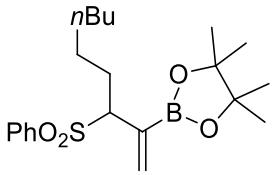
4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)hex-1-en-2-yl)-1,3,2-dioxaborolane (8c): mp = 84 – 85 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7 Hz, 2H), 7.58 (t, *J* = 7 Hz, 1H), 7.48 (t, *J* = 8 Hz), 6.14 (d, *J* = 2 Hz, 1H), 5.90 (d, *J* = 1.5 Hz, 1H), 2.12 (m, 1H), 1.97 (m, 1H), 1.38 (m, 1H), 1.25 (m, 2H), 1.3 (d, *J* = 3.5 Hz, 12H), 0.86 (t, *J* = 7 Hz); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.47, 136.51, 133.28, 129.79, 128.70, 83.96, 67.63, 28.93, 24.79, 24.68, 20.07, 13.73; IR (film) ν_{max} = 2976, 2933, 1447, 1422, 1380, 1372, 1316, 1306, 1141, 1083, 968, 852, 719, 689, 650 cm^{-1}; HRMS calcd for $(C_{18}H_{27}BO_4S)Na+$ 373.1615; Found: 373.1611.



8d

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)hept-1-en-2-yl)-1,3,2-dioxaborolane (8d): mp = 51 – 52 °C**.** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 6.14 (d, *J* = 2.5 Hz, 1H), 5.89 (d, *J* = 1.5 Hz, 1H), 3.91 (dd, *J* = 12 Hz, *J* = 3.5 Hz, 1H), 2.16 (m, 1H), 1.98 (m, 1H), 1.27 (m, 4H), 1.13 (d, *J* = 3.5 Hz, 12H), 0.86 (t, *J* = 7 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.50, 136.51, 133.27, 129.79, 128.70, 83.97, 67.96, 28.92, 26.62, 24.81,24.69, 22.40, 13.92; IR (film) ν_{max} = 2977, 2957, 2931, 2860, 1447, 1423, 1372, 1306, 1268, 1213, 1145, 1084, 967, 848, 756, 720, 689, 650 cm^{-1}; HRMS calcd for

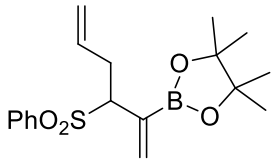
($C_{19}H_{29}BO_4S$)Na+ 387.1778; Found: 387.1766.



8e

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)non-1-en-2-yl)-1,3,2-dioxaborolane (8e):

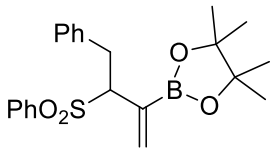
mp = 40 - 43°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8 Hz, 2H), 6.14 (d, *J* = 2 Hz, 1H), 5.89 (d, *J* = 1 Hz, 1H), 3.91 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.17 – 2.12 (m, 1H), 2.02 – 1.95 (m, 1H), 1.29 – 1.23 (m, 8H), 1.13 (d, *J* = 3 Hz, 12H), 0.85 (t, *J* = 7 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.51, 136.51, 133.26, 129.78, 128.70, 83.96, 67.95, 31.62, 28.90, 26.87, 26.70, 24.81, 24.69, 22.66, 14.17; IR (film) ν_{max} = 2955, 2927, 2857, 1722, 1467, 1447, 1423, 1380, 1372, 1306, 1144, 1084, 967, 720, 689 cm^{-1}; HRMS calcd for ($C_{21}H_{33}BO_4S$)Na+ 415.2084; Found: 415.2079.



8f

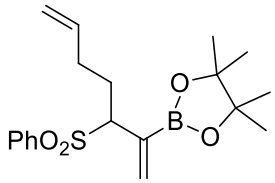
4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)hexa-1,5-dien-2-yl)-1,3,2-dioxaborolane (8f): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.5 Hz, 2H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 2H), 6.14 (d, *J* = 2 Hz, 1H), 5.86 (d, *J* = 1 Hz, 1H), 5.63 (dddd, *J* = 7 Hz, J = 7 Hz, *J* = 10

Hz, *J* = 13.5 Hz, 1H), 5.06 (dd, *J* = 1.5 Hz, *J* = 17.5 Hz, 1H), 5.01 (dd, *J* = 1.5 Hz, *J* = 10 Hz, 1H), 3.99 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.96 – 2.91 (m, 1H), 2.79 – 2.72 (m, 1H), 1.15 (d, *J* = 3 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.15, 136.80, 133.73, 133.45, 129.78, 128.78, 117.90, 84.02, 67.68, 31.42, 24.80, 24.70; IR (film) ν_{max} = 3077, 2978, 2931, 1639, 1480, 1447, 1419, 1379, 1372, 1355, 1305, 1259, 1144, 1082, 1025, 998, 917, 690 cm^{-1}; HRMS calcd for $(C_{18}H_{25}BO_4S)Na^+$ 371.1458; Found: 371.1455.



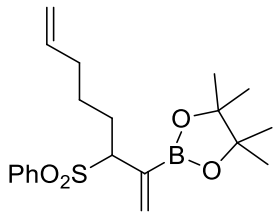
8g

4,4,5,5-tetramethyl-2-(4-phenyl-3-(phenylsulfonyl)but-1-en-2-yl)-1,3,2-dioxaborolane (8g): mp =112 – 113 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.5 Hz, 2H), 7.58 (t, *J* = 7 Hz, 1H), 7.49 (t, *J* = 8 Hz, 2H), 7.21 - 7.18 (m, 2H), 7.15 – 7.12 (m, 3H), 6.06 (d, *J* = 2 Hz, 1H), 5.94 (s, 1H), 4.24 (dd, *J* = 3.5 Hz, *J* = 12 Hz, 1H), 3.63 (dd, *J* = 3.5 Hz, *J* = 14 Hz, 1H), 3.29 (dd, *J* = 12 Hz, *J* = 14 Hz, 1H), 1.06 (d, *J* = 17.5 Hz, 12H) ; ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.49, 137.41, 137.24, 133.40, 129.63, 129.36, 128.80, 128.39, 126.52, 83.97, 68.97, 33.03, 24.77, 24.49 ; IR (film) ν_{max} = 3060, 3015, 2922, 1620, 1506, 1473, 1450, 1421, 1382, 1301, 1275, 1244, 1145, 1112, 1082 cm^{-1}; HRMS calcd for $(C_{22}H_{27}BO_4S)Na^+$ 421.1615; Found: 421.1610.



8h

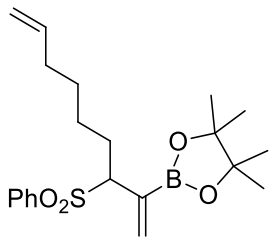
4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)hepta-1,6-dien-2-yl)-1,3,2-dioxaborolane (8h): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 6.16 (d, *J* = 2 Hz, 1H), 5.89 (s, 1H), 5.76 – 5.68 (m, 1H), 4.99 (d, *J* = 6 Hz, 1H), 4.96 (s, 1H), 3.93 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.90 – 2.24 (m, 1H), 2.18 – 2.07 (m, 2H), 2.00 – 1.95 (m, 1H), 1.13 (d, *J* = 2.5 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.32, 136.96, 136.79, 133.31, 129.71, 128.68, 115.98, 83.91, 67.32, 30.71, 26.13, 24.76, 24.70, 24.61; IR (film) ν_{max} = 3077, 2978, 2931, 1639, 1480, 1447, 1419, 1390, 1379, 1372, 1355, 1305, 1259, 1214, 1144, 1082, 1025, 998, 967, 917, 867, 845, 753, 818, 690 cm^{-1}; HRMS calcd for $(C_{19}H_{27}BO_4S)Na^+$ 385.1615; Found: 385.1610.



8i

4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)octa-1,7-dien-2-yl)-1,3,2-dioxaborolane (8i): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8 Hz, 2H), 6.14 (d, *J* = 2 Hz, 1H), 5.90 (s, IH), 5.73 (dddd, *J* = 6.5 Hz, *J* = 6.5 Hz, *J* = 10 Hz, *J* = 17 Hz, 1H), 4.97 (dd, *J* = 1.5 Hz, *J* = 17 Hz, 1H), 4.93 (dd, *J* = 1 Hz, *J* = 10 Hz, 1H), 3.92 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.18 (dddd, J = 3.5 Hz, J = 6.5 Hz, J = 10 Hz, J = 13.5 Hz, 1H), 2.09 – 1.96 (m, 3H), 1.44 – 1.39 (m, 1H), 1.36 – 1.30 (m, 1H), 1.13 (d, *J* = 2.5 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.46, 138.12, 136.63, 133.31, 129.78, 128.73, 115.14, 84.01, 67.77, 33.37, 26.47, 26.11, 24.82, 24.70; IR (film) ν_{max} = 3072, 2977, 2931, 1640, 1447, 1423, 1380,

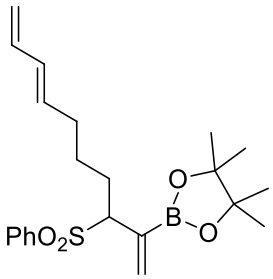
1372, 1306, 1213, 1143, 1085, 1024, 968, 913, 849, 720, 689 cm^{-1}; HRMS calcd for $(C_{20}H_{29}BO_4S)Na+$ 399.1771; Found: 399.1767.



8j

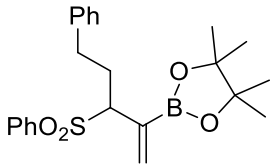
4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)nona-1,8-dien-2-yl)-1,3,2-dioxaborolane (8j):

^1H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8 Hz, 2H), 6.14 (d, *J* = 2 Hz, 1H), 5.90 (s, IH), 5.73 (dddd, *J* = 6.5 Hz, *J* = 6.5 Hz, *J* = 10 Hz, *J* = 17 Hz, 1H), 4.96 (dd, *J* = 1.5 Hz, *J* = 17 Hz, 1H), 4.91 (dd, *J* = 1 Hz, *J* = 10 Hz, 1H), 3.91 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.19 – 2.13 (m, 1H), 2.03 – 1.95 (m, 3H), 1.41 – 1.31 (m, 3H), 1.28 – 1.22 (m, 1H), 1.13 (d, *J* = 4 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.66, 138.42, 136.54, 133.28, 130.62, 130.04, 129.71, 128.69, 114.63, 83.95, 67.83, 33.47, 28.44, 26.72, 26.19, 24.77, 24.64, 22.79; IR (film) ν_{max} = 3072, 2977, 2931, 1640, 1447, 1423, 1380, 1372, 1306, 1213, 1143, 1085, 1024, 968, 913, 849, 720, 689 cm^{-1}; HRMS calcd for $(C_{21}H_{31}BO_4S)Na+$ 413.1928 ; Found: 413.1923.



8k

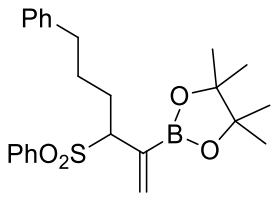
(E)-4,4,5,5-tetramethyl-2-(3-(phenylsulfonyl)deca-1,7,9-trien-2-yl)-1,3,2-dioxaborolane (8k): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8 Hz, 2H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 8 Hz, 2H), 6.26 (ddd, *J* = 10.5, *J* = 10.5, *J* = 17 Hz, 1H), 6.14 (d, *J* = 2 Hz, IH), 6.01 (dd, *J* = 10.5 Hz, *J* = 15.5 Hz, 1H), 5.88 (s, 1H), 5.62 (ddd, *J* = 7 Hz, J = 7 Hz, *J* = 15 Hz, 1H), 5.07 (d, *J* = 17 Hz, 1H), 4.95 (d, *J* = 10 Hz, 1H), 3.91 (dd, *J* = 3.5 Hz , *J* = 11.5, 1H), 2.04 – 2.13 (m, 1H), 2.10 – 1.97 (m, 3H), 1.45 – 1.40 (m, 1H), 1.39 – 1.31 (m, 1H), 1.13 (s, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 138.36, 137.17, 136.60, 134.23, 133.31, 131.63, 129.40, 128.70, 115.21, 83.97, 67.81, 32.14, 26.50, 26.41, 24.78, 24.67; IR (film) ν_{max} = 3072, 2977, 2931, 1640, 1447, 1423, 1380, 1372, 1306, 1213, 1143, 1085, 1024, 968, 913, 849, 720, 689 cm^{-1}; HRMS calcd for $(C_{22}H_{31}BO_4S)Na^+$ 425.1925; Found: 425.1925.



8l

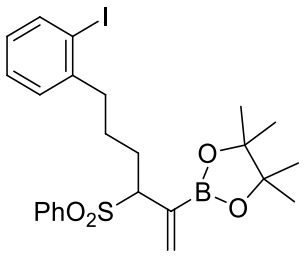
4,4,5,5-tetramethyl-2-(5-phenyl-3-(phenylsulfonyl)pent-1-en-2-yl)-1,3,2-dioxaborolane (8l): mp = 98 - 99°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5, 2H), 7.57 (t, *J* = 7.5, 1 H), 7.47 (t, *J* = 8 Hz, 2H), 7.25 (t, *J* = 8 Hz, 2H), 7.18 (t, *J* =7 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 2H), 6.18 (d, *J* = 2 Hz, 1H), 5.86 (s, 1H), 3.90 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.72 – 2.67 (m, 1H), 2.53 – 2.44 (m, 2H), 2.38 – 2.30 (m, 1H), 1.16 (d, *J* = 4 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 140.85, 138.20, 136.96, 133.36, 129.77, 128.74, 128.60, 128.56, 126.28, 84.03, 68.03, 32.92, 28.62, 24.87, 24.74; IR (film) ν_{max} = 3063, 3027, 2933, 1604, 1495, 1479, 1447, 1422,

1372, 1305, 1267, 1242, 1146, 1112, 1085, 720, 689 cm^{-1}; HRMS calcd for $(C_{23}H_{29}BO_4S)Na^+$ 435.1771; Found: 435.1770.



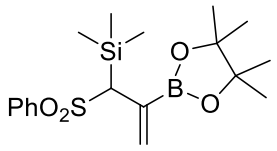
8m

4,4,5,5-tetramethyl-2-(6-phenyl-3-(phenylsulfonyl)hex-1-en-2-yl)-1,3,2-dioxaborolane (8m): mp = 65 - 66°C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.5, 2H), 7.58 (t, *J* = 7 Hz, 1H), 7.48 (t, *J* = 8 Hz, 2H), 7.24 (t, *J* = 8 Hz, 2H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.11 (d, *J* = 7 Hz), 6.12 (d, *J* = 2 Hz, 1H), 5.83 (d, *J* = 1.5 Hz, 1H), 3.94 (dd, *J* = 3.5 Hz, *J* = 11.5 Hz, 1H), 2.65 – 2.52 (m, 2H), 2.34 – 2.17 (m, 1H), 2.08 – 2.01 (m, 1H), 1.68 – 1.61 (m, 1H), 1.60 – 1.52 (m, 1H), 1.13 (s, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 141.66, 138.10, 136.39, 133.09, 129.53, 128.49, 128.40, 128.23, 128.15, 125.74, 83.77, 67.68, 35.36, 29.60, 28.49, 26.38, 24.56, 24.50; IR (film) ν_{max} = 3062, 3025, 2975, 2932, 2860, 1603, 1496, 1453, 1446, 1416, 1390, 1372, 1355, 1304, 1141, 1078, 1029, 966, 849, 749, 732, 700, 691 cm^{-1}; HRMS calcd for $(C_{24}H_{31}BO_4S)Na^+$ 449.1928; Found: 449.1928.



8n

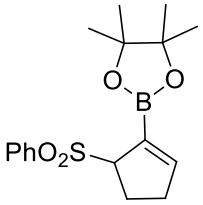
2-(6-(2-iodophenyl)-3-(phenylsulfonyl)hex-1-en-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (8n): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8 Hz, 2H), 7.77 (d, *J* = 8 Hz, 1H) 7.58 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 8 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 8 Hz, 1H), 7.85 (t, *J* = 8 Hz, 1H), 6.12 (d, *J* = 2 Hz, 1H), 5.80 (s, 1H), 3.95 (dd, *J* = 4 Hz, *J* = 11.5 Hz, 1H), 2.75 – 2.63 (m, 2H), 2.27 – 2.16 (m, 1H), 2.13 – 2.05 (m, 1H), 1.66 – 1.61 (m, 1H), 1.55 – 1.49 (m, 1H), 1.14 (s, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 144.42, 139.52, 138.11, 136.56, 133.36, 129.76, 129.46, 128.71, 128.43, 127.89, 100.53, 83.98, 68.22, 40.47, 27.40, 26.38, 24.80, 24.75; IR (film) ν_{max} = 3062, 3025, 2975, 2932, 2860, 1603, 1496, 1453, 1446, 1416, 1390, 1372, 1355, 1304, 1141, 1078, 1029, 966, 849, 749, 732, 700, 691 cm^{-1}; HRMS calcd for $(C_{25}H_{30}BIO_4S)Na^+$ 575.0894; Found: 575.0894.



8o

trimethyl(1-(phenylsulfonyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)allyl)silane (8o): mp = 122 – 123 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8 Hz, 2H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 6.19 (s, 1H), 6.01 (s, 1H), 3.90 (s, 1H), 1.05 (d, *J* = 3 Hz, 12H), 0.31 (s, 9H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 141.75, 134.95, 132.38, 128.47, 128.35, 84.11, 24.61, 24.54; IR (film) ν_{max} = 2977, 2926, 1446, 1422, 1380, 1372, 1359, 1316, 1305, 1250, 1137, 1112, 1085, 958, 848, 757, 714, 689, 646 cm^{-1}; HRMS calcd for $(C_{18}H_{29}BO_4SSi)Na^+$ 403.1541; Found: 403.1538.

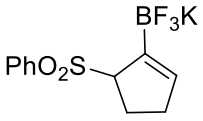
Ring Closing Metathesis of 8h



11

4,4,5,5-tetramethyl-2-(5-(phenylsulfonyl)cyclopent-1-en-1-yl)-1,3,2-dioxaborolane (11): Alkylation product **8h** (64 mg, 0.176 mmol) was dissolved in 880 µL dry dichloromethane (0.2M) that was degassed with argon prior to use. The solution was added to a sealed tube under argon atmosphere and treated with Grubbs 2nd generation catalyst (7.5 mg, 5 mol %). The tube was sealed with Teflon coated cap and heated to 45 ^{o}C for 2 h. Solvent was evaporated and the crude black residue was purified on a flash column (20% EtOAc/Hexanes) affording **11** as a solid (53 mg, 92%). mp = 92 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8 Hz, 2H), 7.61 (t, *J* = 7.5, 1H), 7.50 (t, *J* = 8, 2H), 6.72 (s, 1H), 4.45 (dd, *J* = 1 Hz, *J* = 8.5 Hz, 1H), 244. (dd, *J* = 8 Hz, *J* = 14.5 Hz, 1H), 2.19 – 2.11 (m, 1H), 2.29 – 2.23 (m, 1H), 2.04 – 1.97 (m, 1H), 1.24 (d, *J* = 7 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 3060, 2996, 2919, 1957, 1700, 1613, 1447, 1426, 1392, 1365, 1331, 1266, 1168, 1085, 980, 897 cm^{-1}; HRMS calcd for $(C_{17}H_{23}BO_4S)Na^+$ 357.1302; Found: 357.1297.

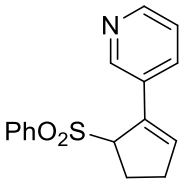
Conversion of 11 to potassium trifluoroborate



17

Potassium (5-(phenylsulfonyl)cyclopent-1-en-1-yl)trifluoroborate (17): Boronic ester **11** (126 mg, 0.376 mmol) was dissolved in 3.75 mL methanol (0.1M) and treated with an aqueous solution of potassium bifluoride (206 mg, 2.63 mmol). After stirring for 3 h at 25 ^{o}C, the aqueous solution was removed under vacuum and the resulting white residue was dissolved in hot acetonitrile and hot filtered. The acetonitrile was removed under vacuum and the crude solid was washed with EtOAc to remove any remaining impurities (100 mg, 85%). mp = 105 °C; ^{1}H MR (500 MHz, DMSO) δ 7.87 (d, *J* = 7.5 Hz, 2H), 7.62 (t, *J* = 7.5, 1H), 7.50 (t, *J* = 8 Hz, 2H), 5.67 (s, 1H), 4.14 (d, *J* = 9.5 Hz), 2.13 (dd, *J* = 8.5 Hz, *J* = 14.5 Hz, 1H), 1.97 – 1.93 (m, 1H), 1.90 – 1.84 (m, 1H), 1.45 – 1.39 (m, 1H); ^{13}C NMR (500 MHz, DMSO) δ 138.26, 138.10, 132.83, 129.31, 128.04, 75.30, 31.32, 27.56; HRMS calcd for $(C_{11}H_{11}BF_3KO_2S)Na^+$ 337.0054; Found: 337.0054.

SM cross-coupling of 17 and 2-iodopyridine

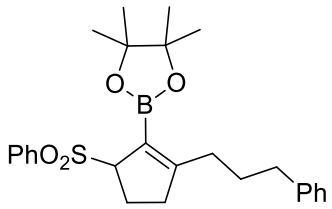


16

3-(5-(phenylsulfonyl)cyclopent-1-en-1-yl)pyridine (16): A sealed tube was purged with argon and charged with 2.0 mL of a (3:1) toluene/water. To the solvent was added 3-iodopyridine (47 mg, 0.228 mmol), $PdCl_2$(dppf) (15.5 mg, 19 μmol), **17** (60 mg, 0.190 mmol) and cesium carbonate (185 mg, 0.570 mmol). The vessel was purged with argon balloon for an additional five minutes before being sealed with a teflon cap and heated to 80 ^{o}C for 9 h. The reaction mixture was diluted with EtOAc and the organic layer was separated and dried over $MgSO_4$. The $MgSO_4$ was and the solvent was removed in *vacuo*. The residue was absorbed onto silica

and purified via flash chromatography (100% EtOAc) affording the coupled product **16** (38 mg, 70%). mp = 91 °C; ^{1}H MR (500 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.41 (d, *J* = 4, 1H), 7.69 (d, *J* = 7 Hz, 2H), 7.60 (d, *J* = 8 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.39 (t, J = 7.5 Hz, 2H), 7.14 (dd, J = 5 Hz, J = 8 Hz, 1H), 6.43 (s, 1H), 4.72 (d, J = 8.5 Hz, 1H), 2.76 (dd, J = 8.5 Hz, J = 13 Hz, 1H), 2.48 – 2.43 (m, 2H), 2.34- 2.44 (m, 1H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 148.64, 147.80, 138.28, 137.70, 134.87, 133.90, 133.77, 130.49, 129.27, 128.93, 123.18, 71.69, 31.74, 27.32; IR (film) ν_{max} = 3095, 3060, 2975, 2931, 1868, 1623, 1446, 1399, 1317, 1138, 1081, 934 cm^{-1}; HRMS calcd for $(C_{16}H_{15}NO_2S)Na+$ 308.0715; Found: 308.0714.

Alkylation of 11 with 3-phenylpropyl triflate



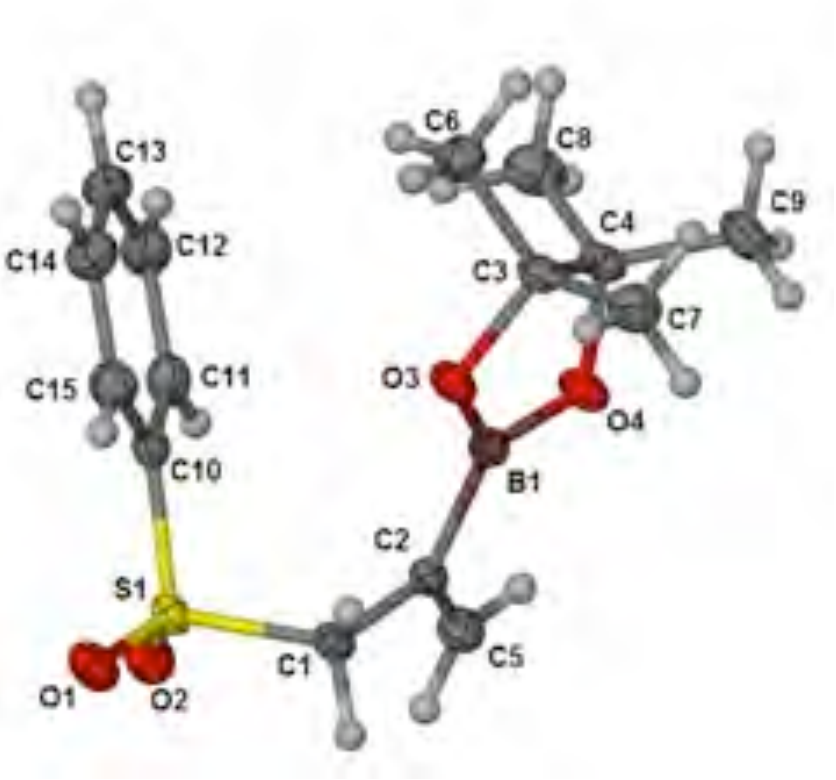
14

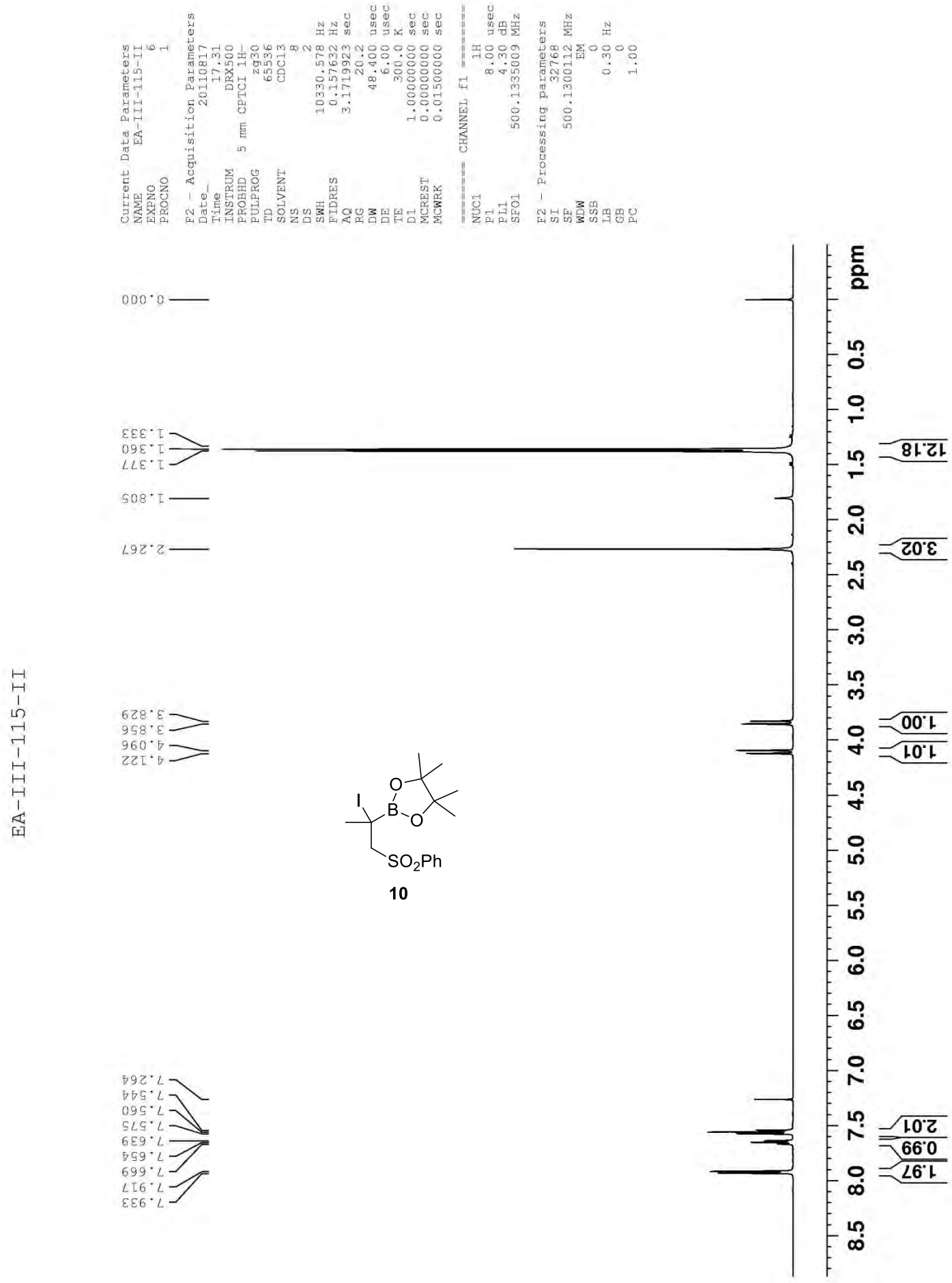
4,4,5,5-tetramethyl-2-(2-(3-phenylpropyl)-5-(phenylsulfonyl)cyclopent-1-en-1-yl)-1,3,2-dioxaborolane (14): To a solution of diisopropylamine (176 µL , 1.25 mmol) in THF (7.6 mL) was added *n*butyl lithium (2.04 M in THF, 566 µL, 1.15 mmol) at -78 °C and stirred for one hour. To the lithium diisopropylamine was added a solution of **11** (322 mg, 0.963mmol) in 2.0 mL THF which was stirred for an hour before addition of 3-phenylpropyl triflate (0.963 mmol). After dropwise addition of electrophile, the reaction was removed from cold bath and allowed to warm to room temperature over 3 h. The reaction was quenched with a saturated solution of aqueous NH_4Cl and the mixture was extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated in *vacuo*. The residue was absorbed onto silica and purified via flash chromatography (15% EtOAc in Hexanes) affording the

corresponding alkylated product **13**/**14** as a 1:1.73 mixture of isomers (274 mg, 63%). In a flask containing isomers **13**/**14** (82 mg, 0.181 mmol) and 2.0 mL AcOH/water (3:1) was added benzenesulfinate (207 mg, 1.26 mmol). The flask was heated to 95 ^{o}C at which point the mixture became homogenous. After stirring for 7 h at 95 ^{o}C, the reaction was diluted with water and extracted with EtOAc. The organic layer was separated, dried over MgSO4 and concentrated in *vacuo*. The resulting solid was recrystallized from hexanes/Et2O (3:1) affording **14** as a white solid (60 mg, 73%).mp = 130 ^{o}C ; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, J = 8.5 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.47 (t, J = 8 Hz, 2H), 7.27 (t, J = 7.5 Hz, 2H), 7.17 (t, J = 7.5 Hz, 1H), 7.14 (d, J = 7 Hz, 2H), 4.44 (d, J = 8 Hz, 1H), 2.54 (t, J = 8 Hz, 2H), 2.48 – 2.42 (m, 1H), 2.36 – 2.29 (m, 2H), 2.16 – 2.09 (m, 2H), 1.91 – 1.86 (m, 1H), 1.69 – 1.63 (m, 1H), 1.58 – 1.51 (m, 1H), 1.25 (d, J = 12 Hz, 12H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 169.18, 142.30, 137.68, 133.33, 129.80, 128.71, 128.52, 128.41, 125.88, 83.57, 76.01, 36.30, 35.89, 31.14, 30.02, 26.33, 25.16, 24.69; IR (film) ν_{max} = 3062, 3027, 2977, 2931, 1636, 1601, 1447, 1372, 1305, 1144, 1085 cm^{-1}; HRMS calcd for $(C_{26}H_{33}BO_4S)Na^+$ 475.2084; Found: 475.2079.

Crystallographic Data

CCDC Deposit Number: CCDC 914468

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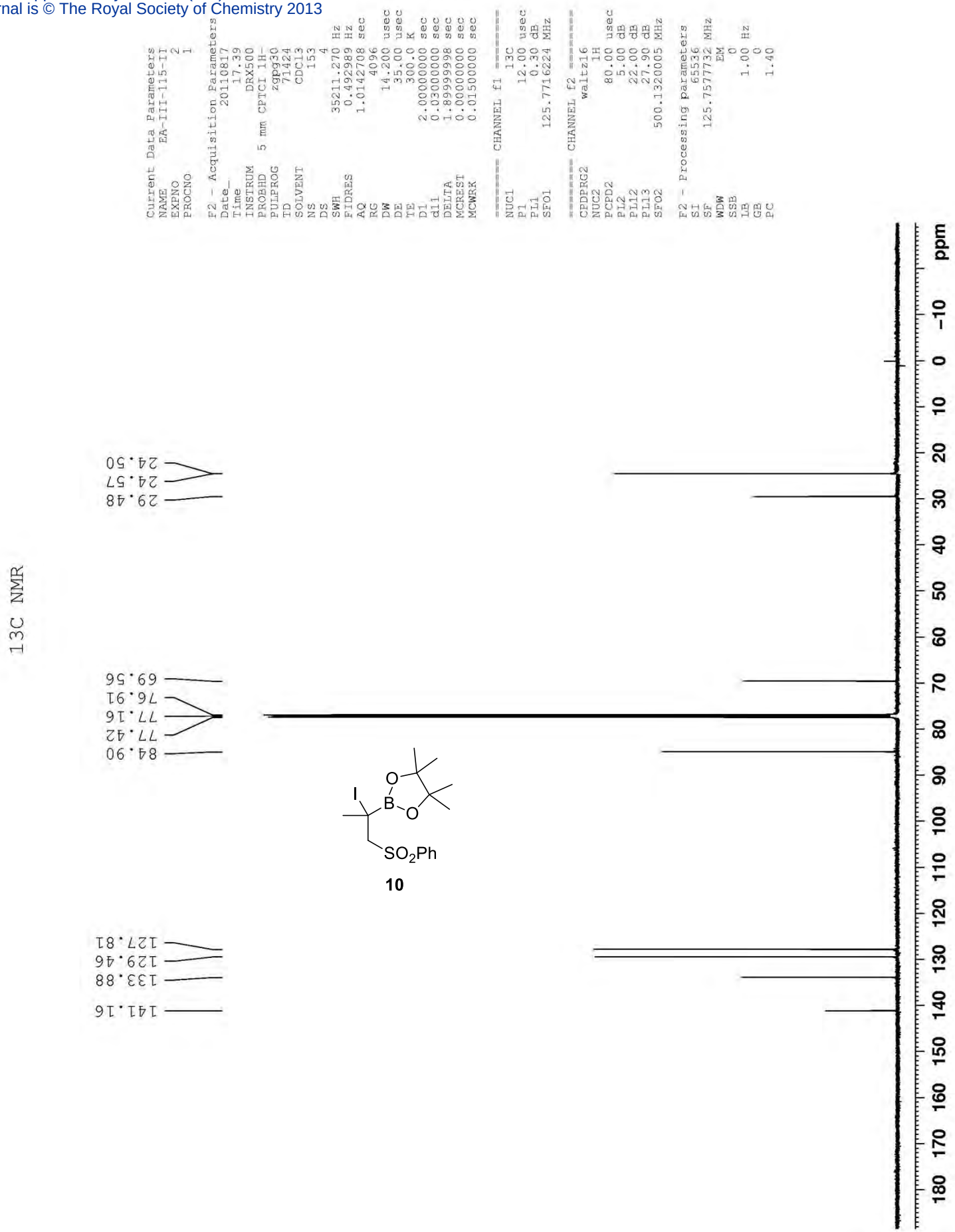
10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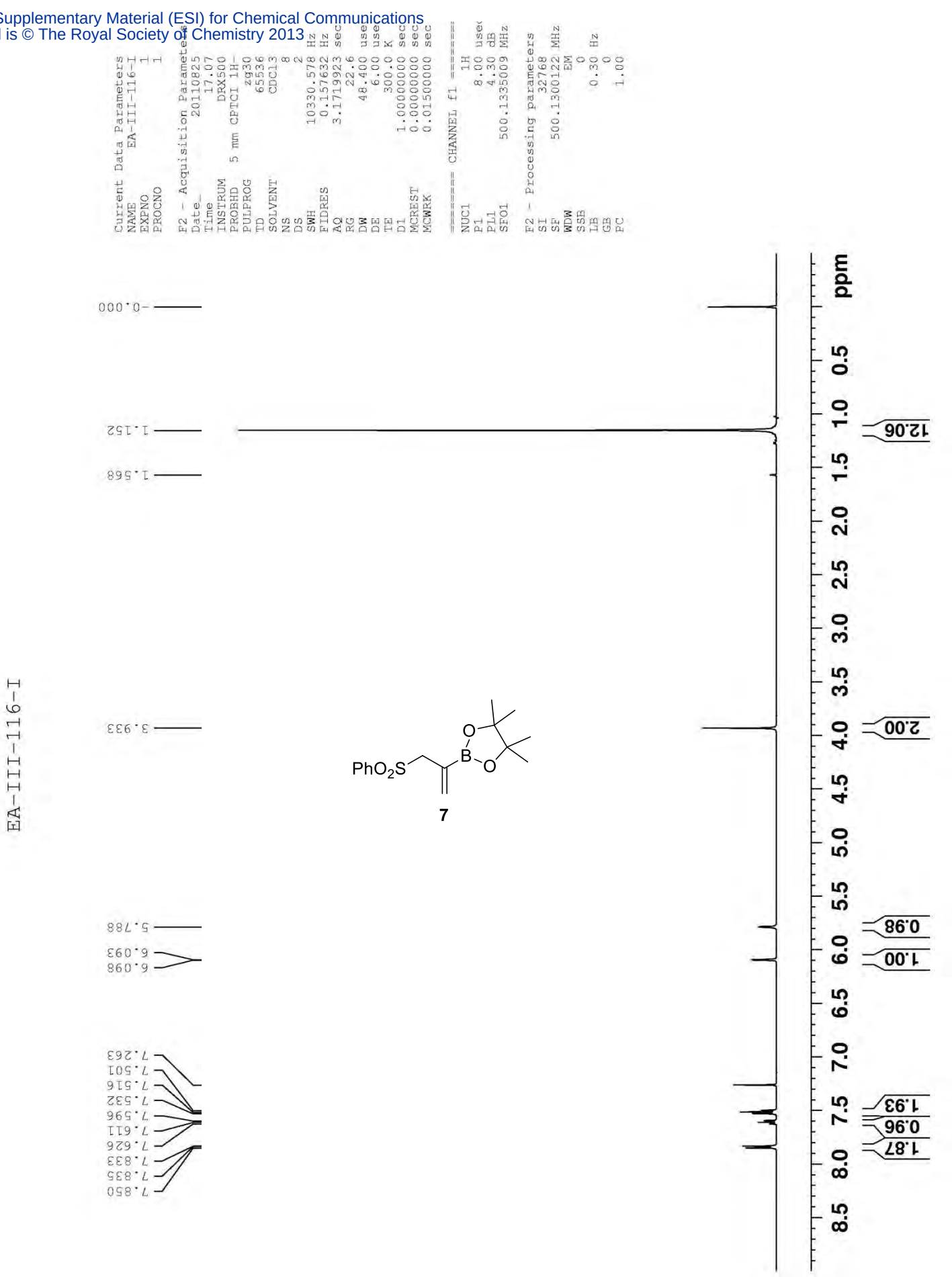
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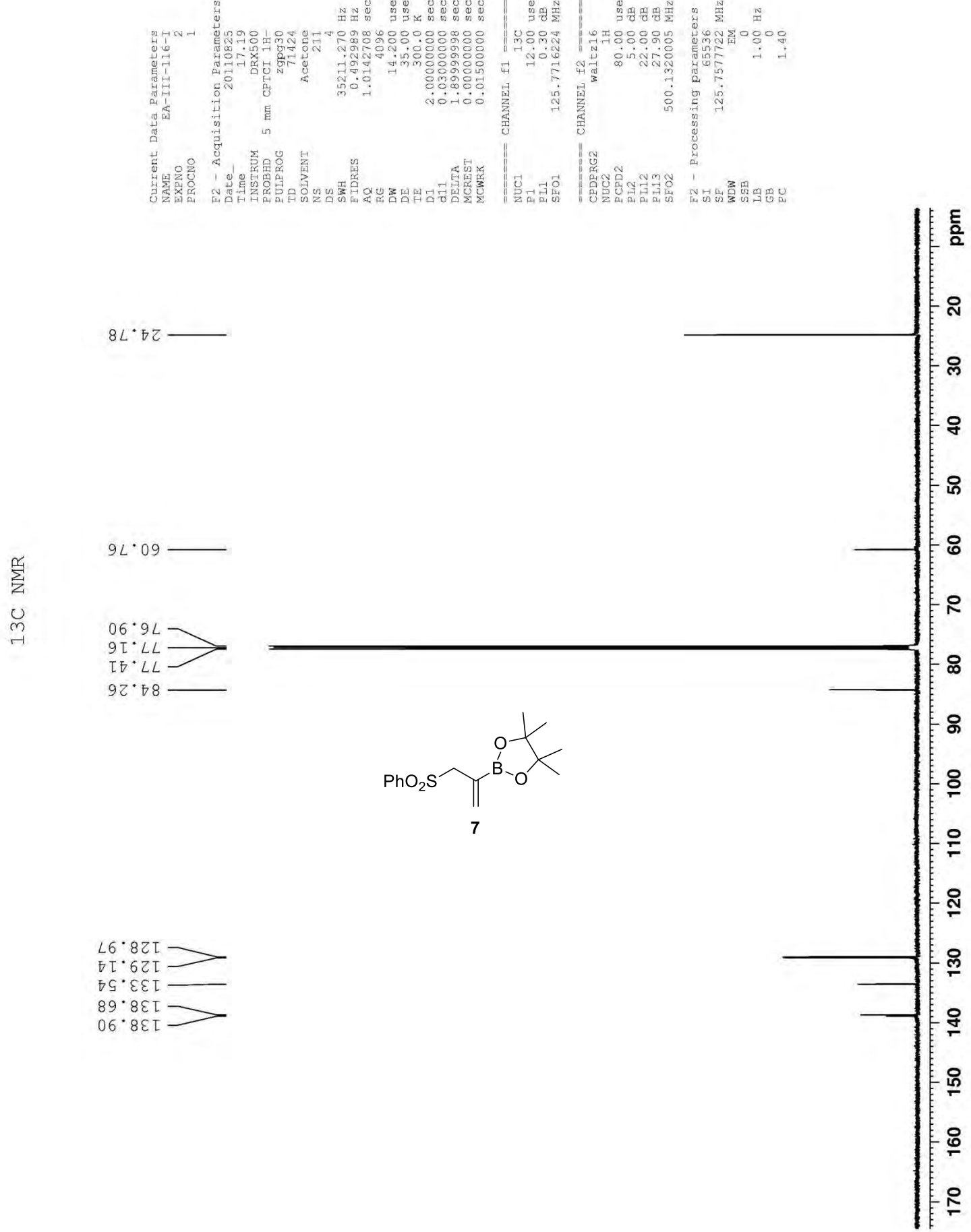
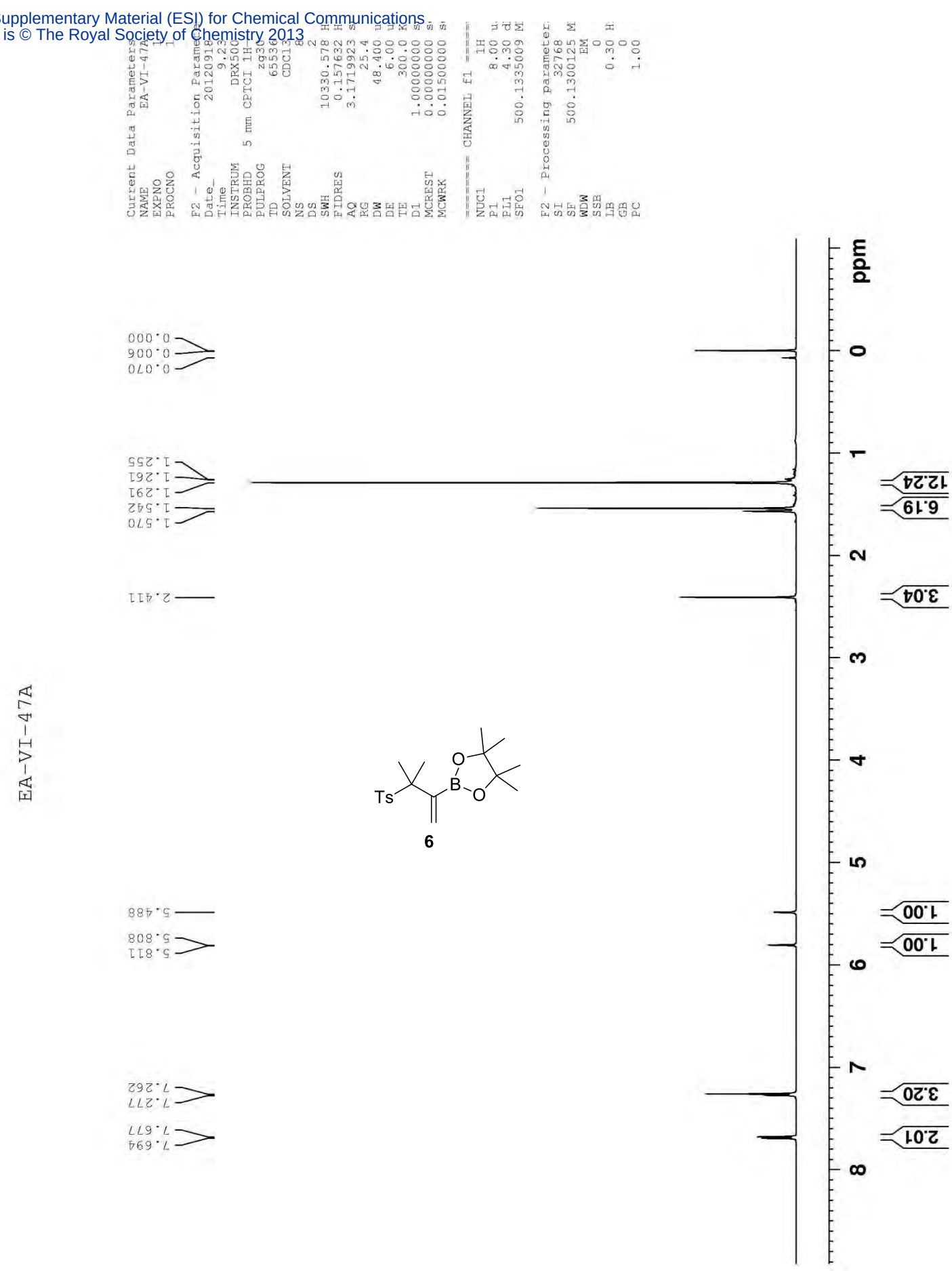
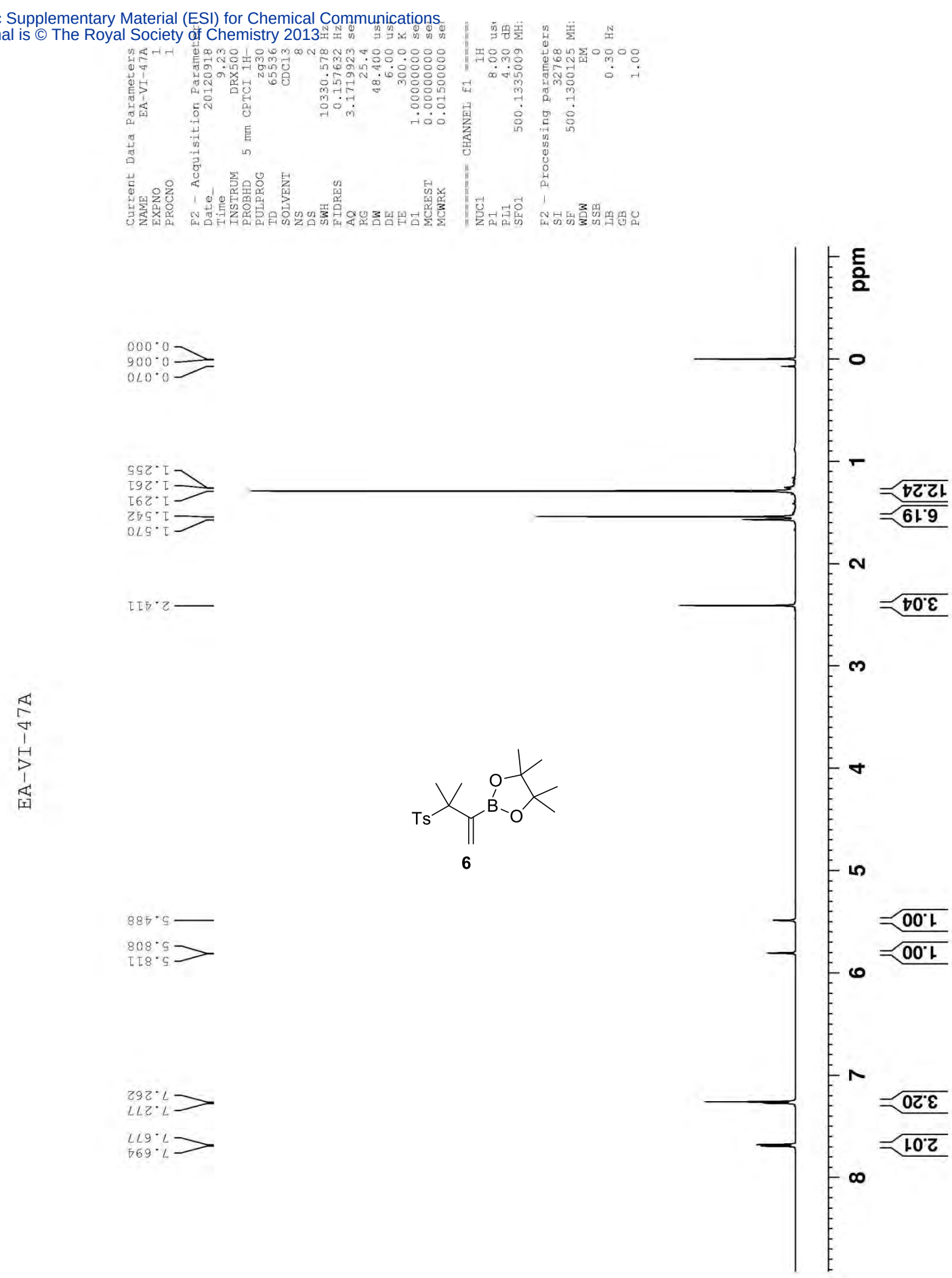
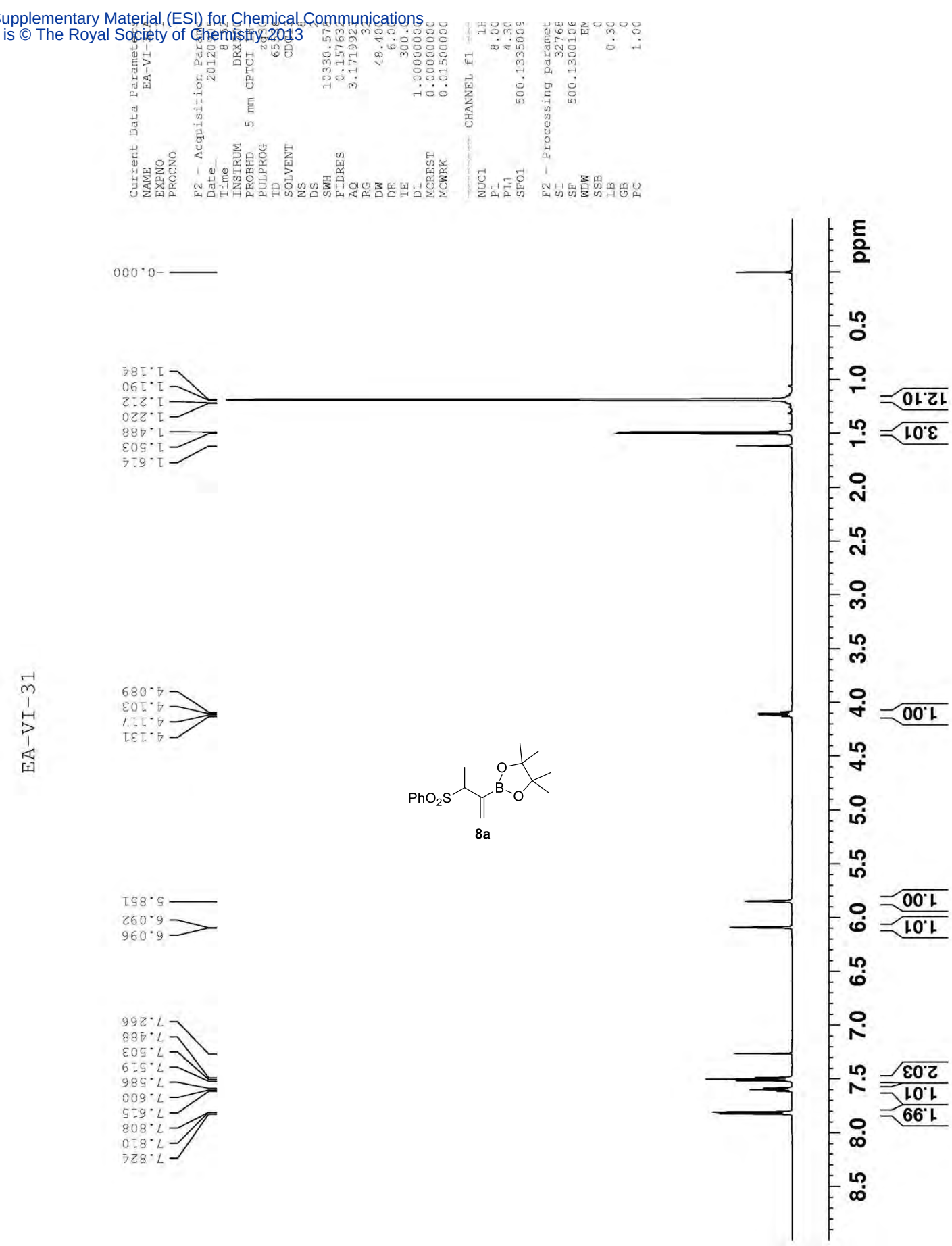
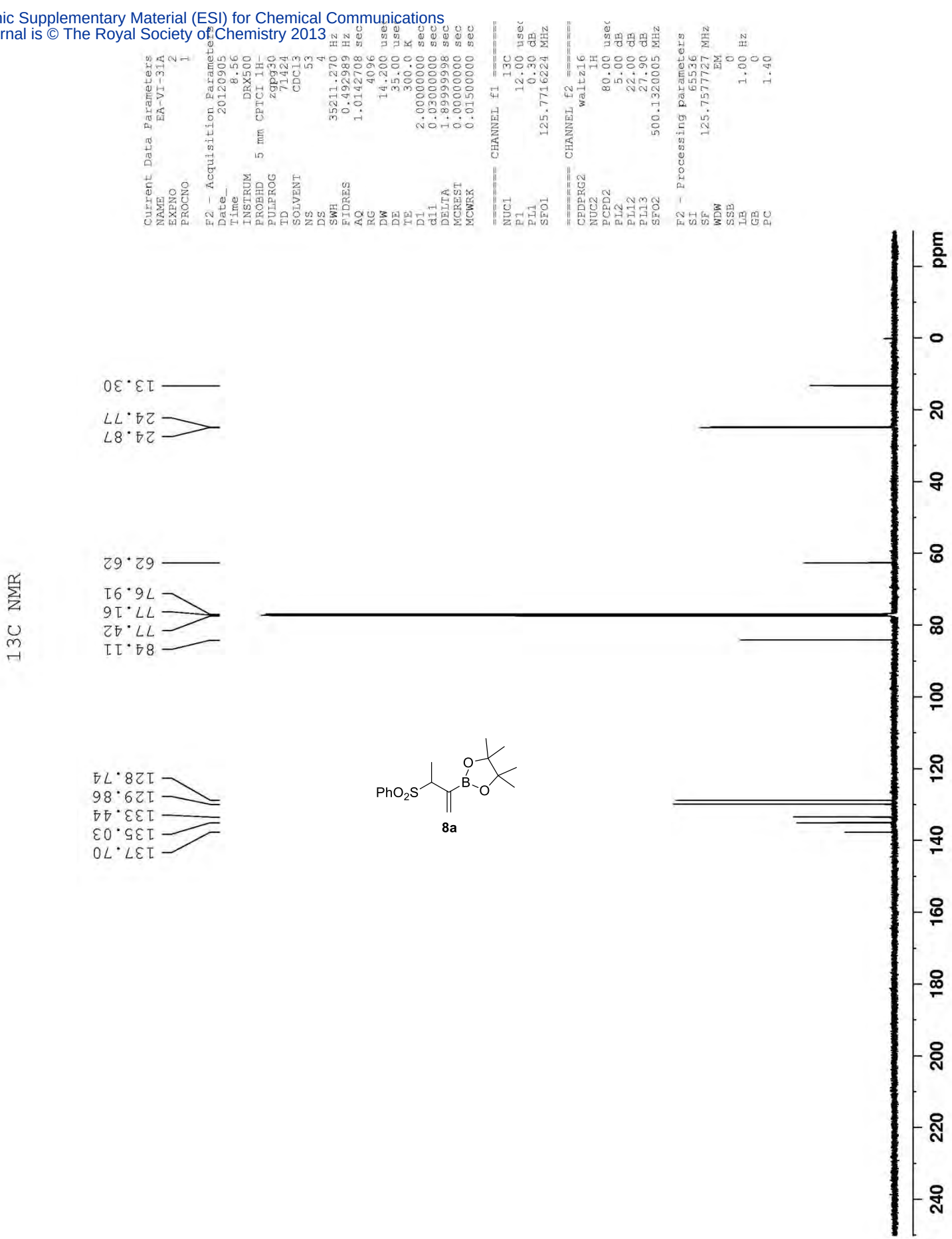
LB 0.30 Hz
GB 0
PC 1.00
PhO2S
O
B
O
7
7.850
7.835
7.833
7.626
7.611
7.596
7.532
7.516
7.501
7.263
6.098
6.093
5.788
3.933
1.568
1.152
0.000
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
1.87
0.96
1.93
1.00
0.98
2.00
12.06

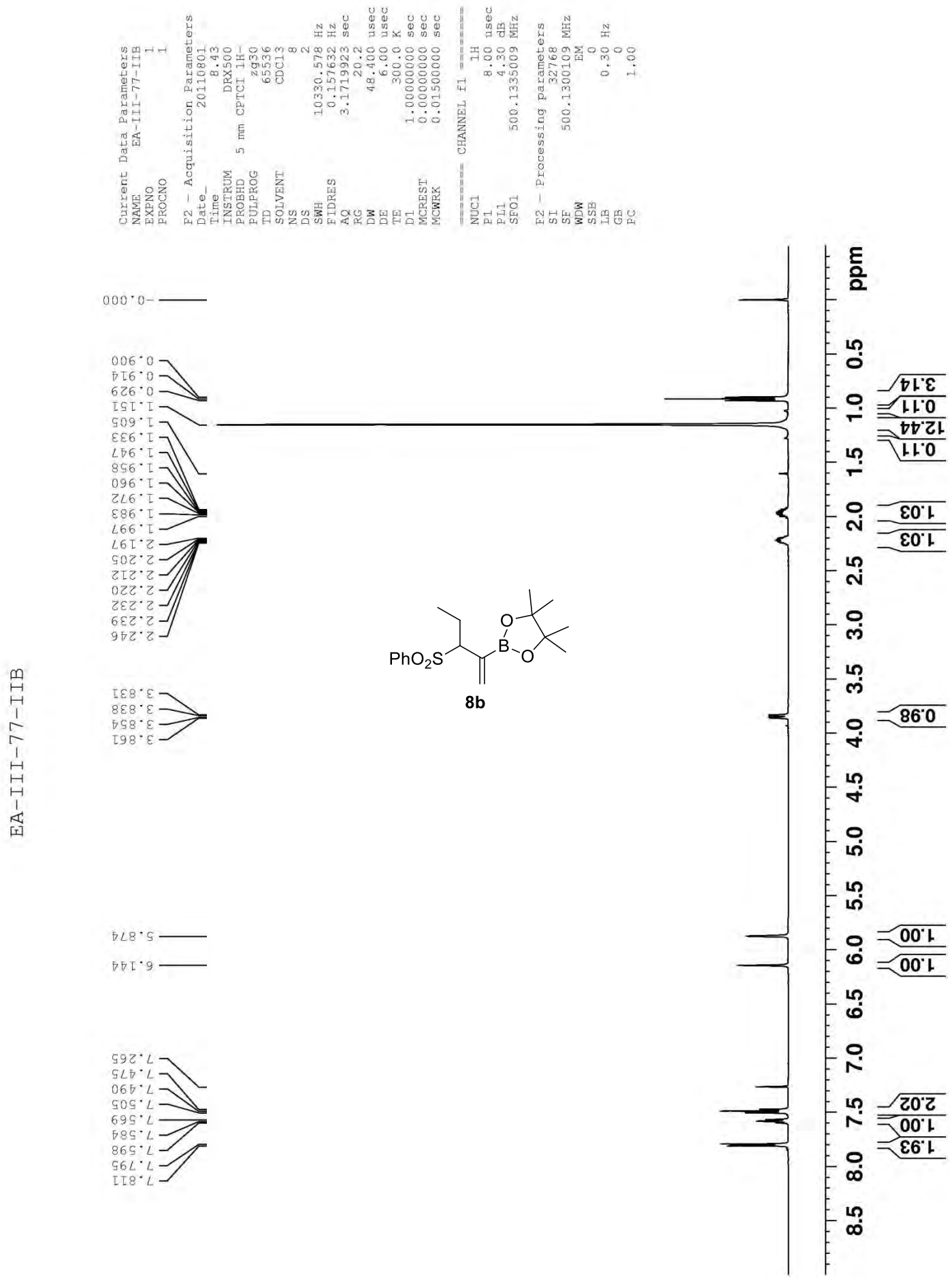

13C NMR
Current Data Parameters
NAME EA-III-116-I
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110825
Time 17.19
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT Acetone
NS 211
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
24.78
60.76
76.90
77.16
77.41
84.26
128.97
129.14
133.54
138.68
138.90
PhO2S
O
B
O
O
7
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

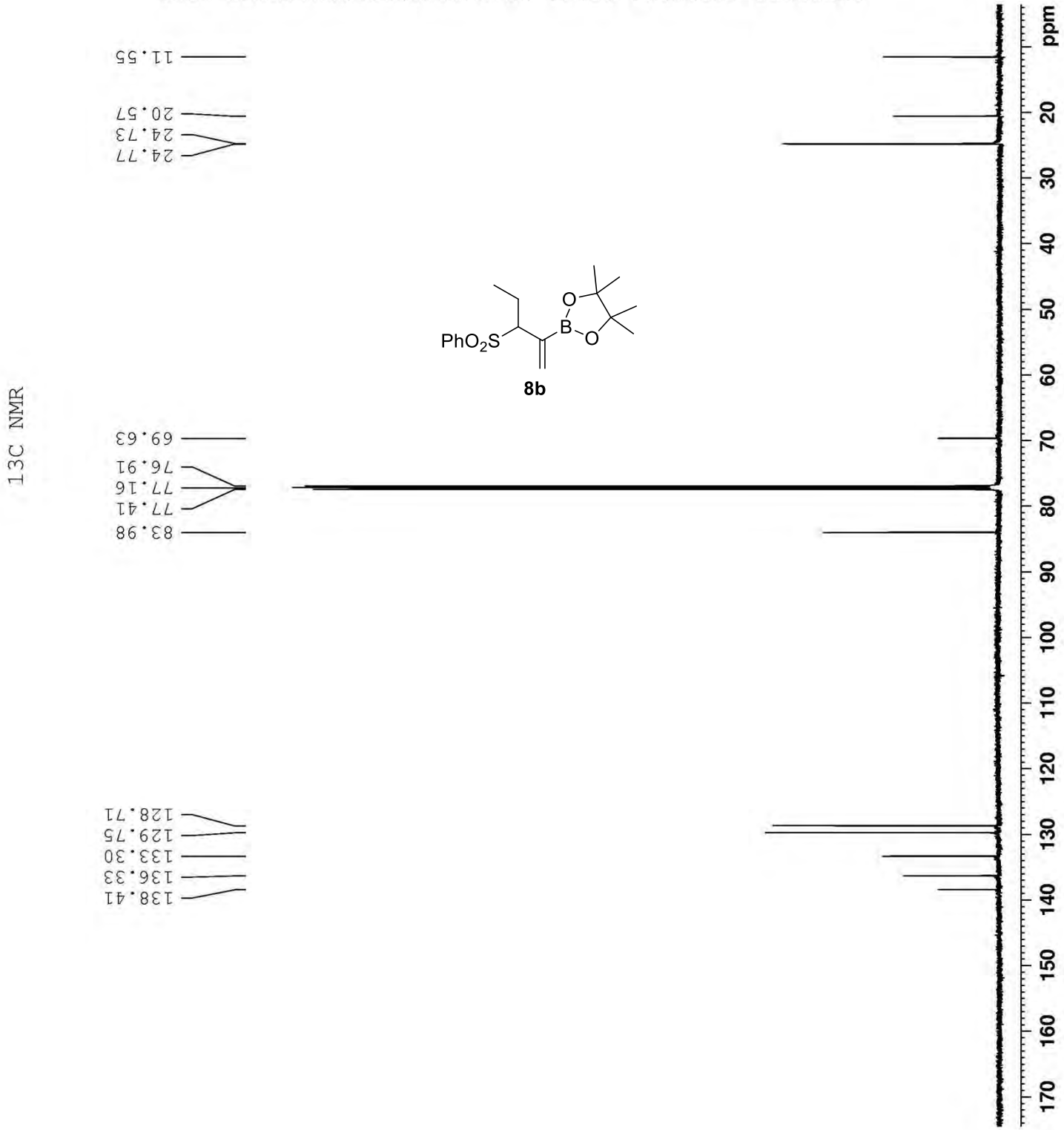


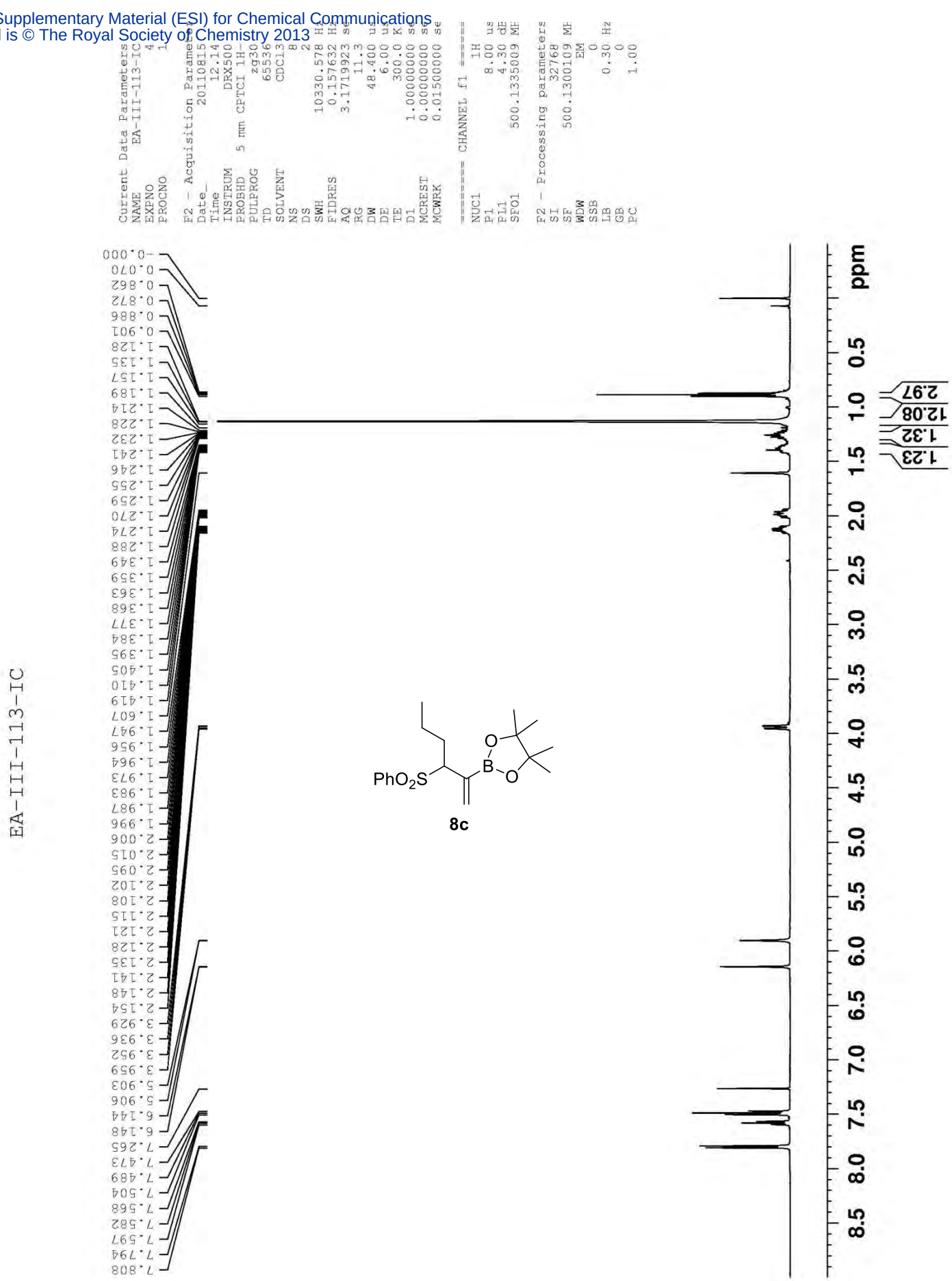


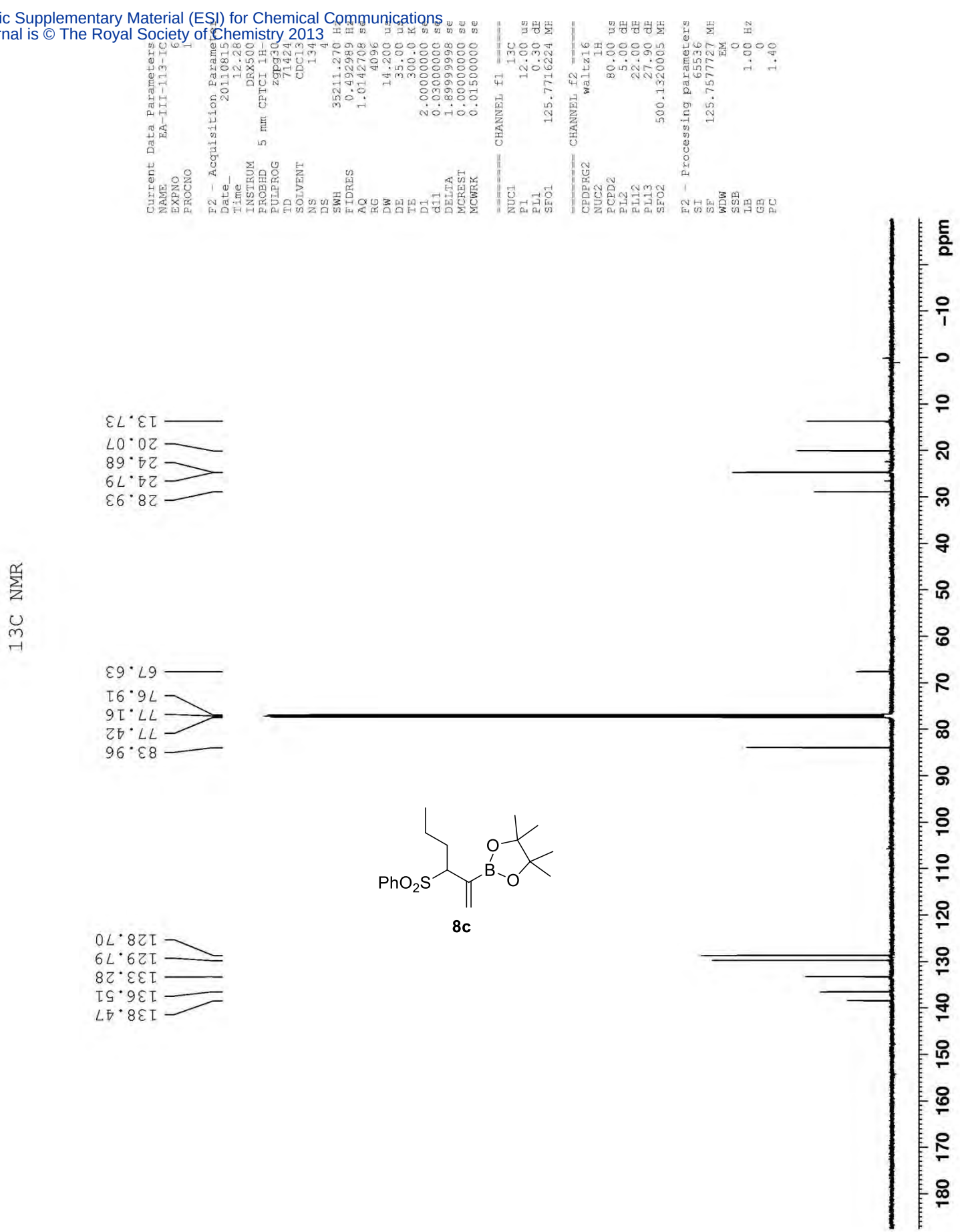


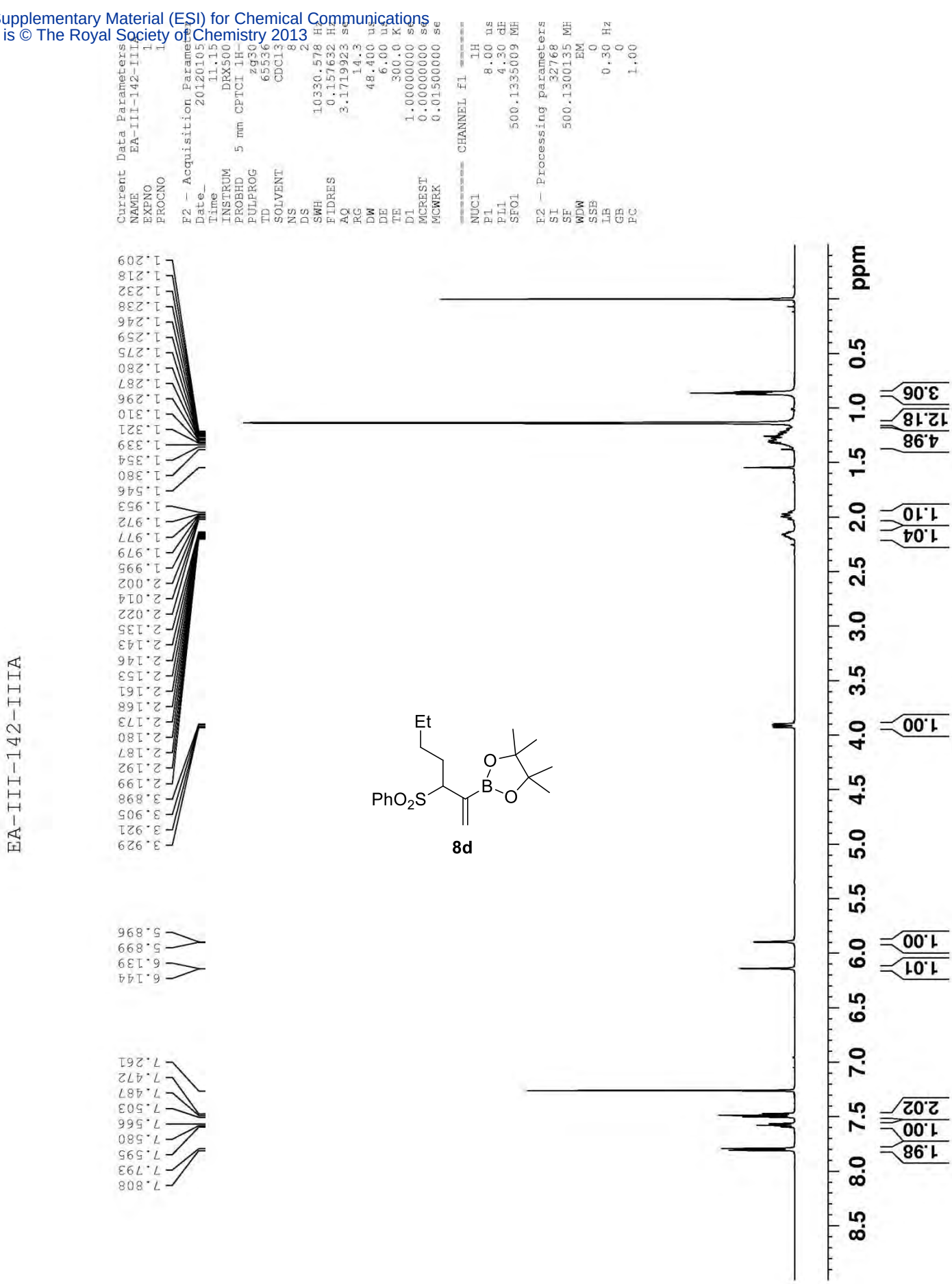


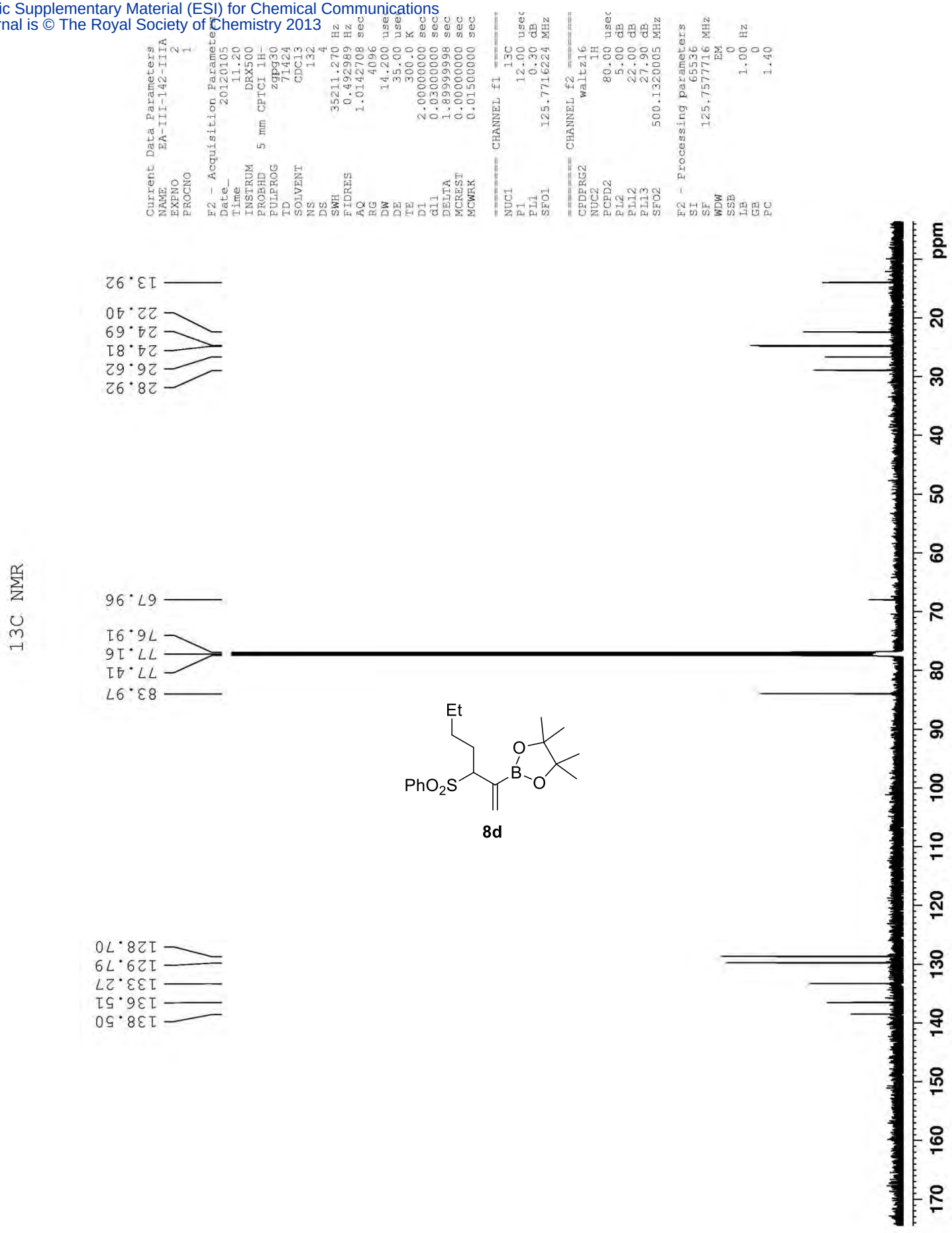

EA-III-77-IIB
Current Data Parameters
NAME EA-III-77-IIB
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110801
Time 8.43
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 20.2
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz
F2 - Processing parameters
SI 32768
SF 500.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
PhO2S
O
B
O
8b
ppm
0.98
1.00
1.00
1.93
2.02
1.03
1.03
0.11
12.44
0.11
3.14

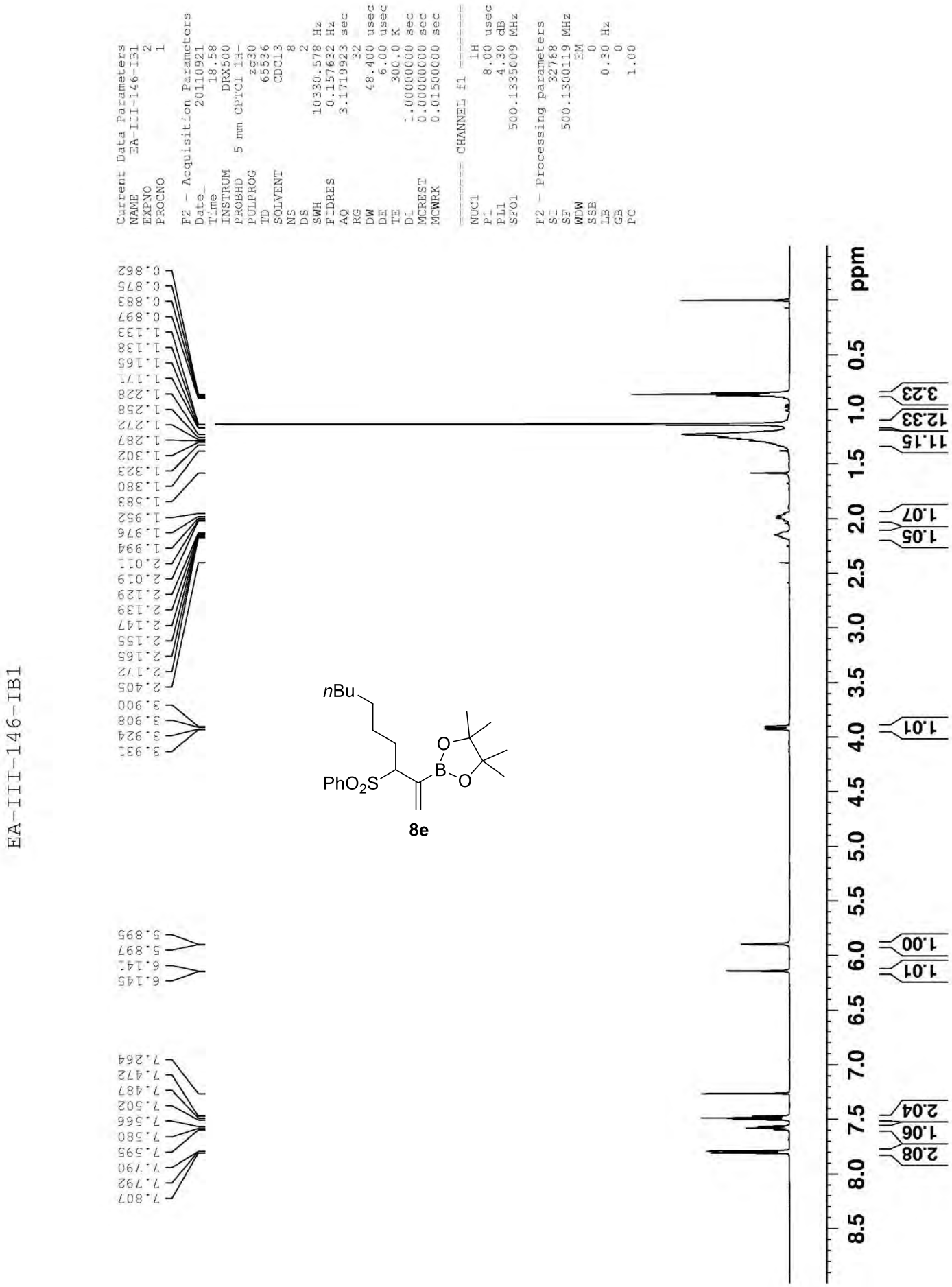

13C NMR
Current Data Parameters
NAME EA-III-77-IIB
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110801
Time 8.49
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 111
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
11.55
20.57
24.73
24.77
69.63
76.91
77.16
77.41
83.98
128.71
129.75
133.30
136.33
138.41
PhO2S
O
B
O
8b
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

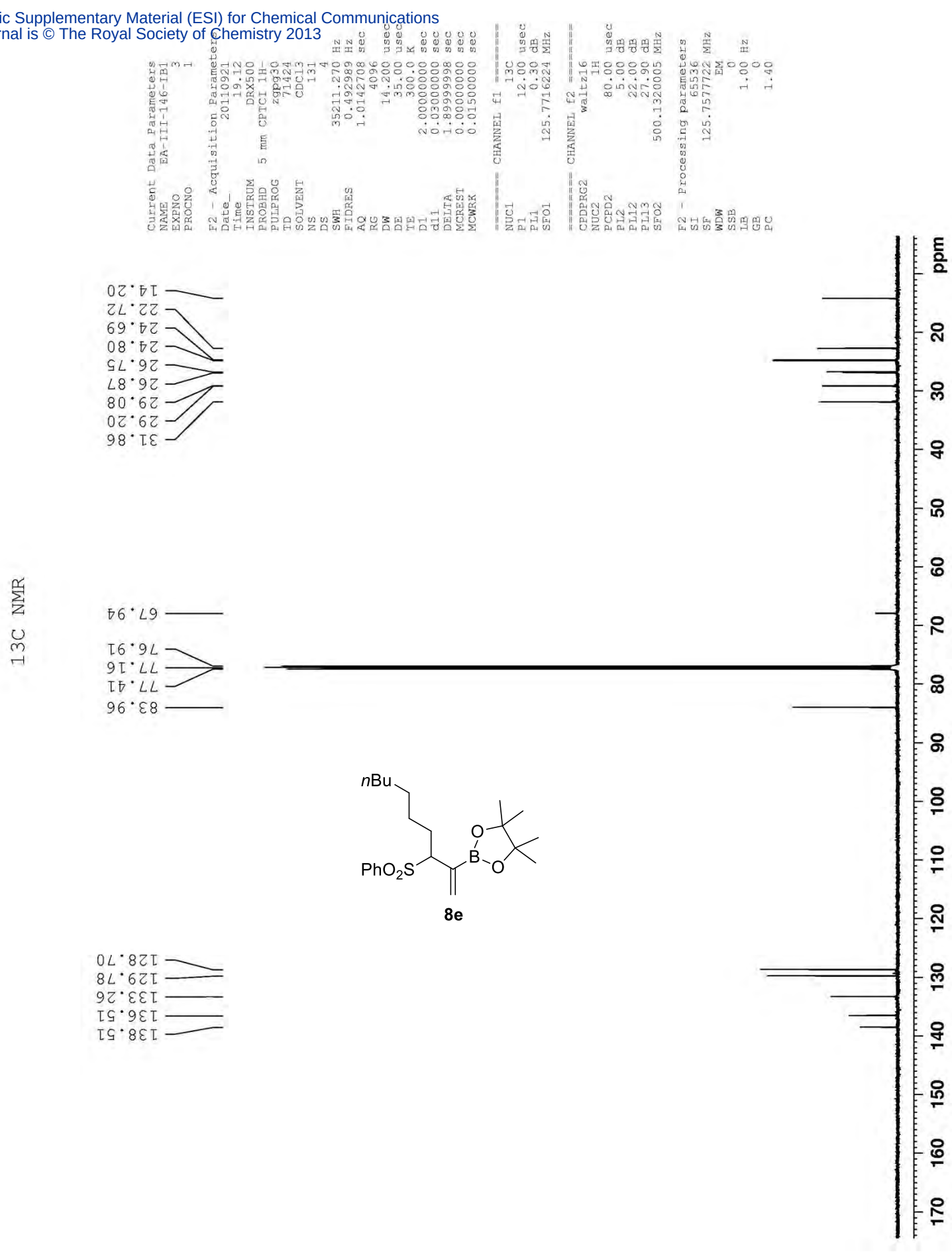

EA-III-113-IC
Current Data Parameters
NAME EA-III-113-IC
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110815
Time 12.14
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 11.3
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D0 0.00000000 sec
MCREST 0.01500000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz
F2 - Processing parameters
SI 32768
SF 500.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
PhO2S
O
B
O
8c
1.23
1.32
12.08
2.97
ppm

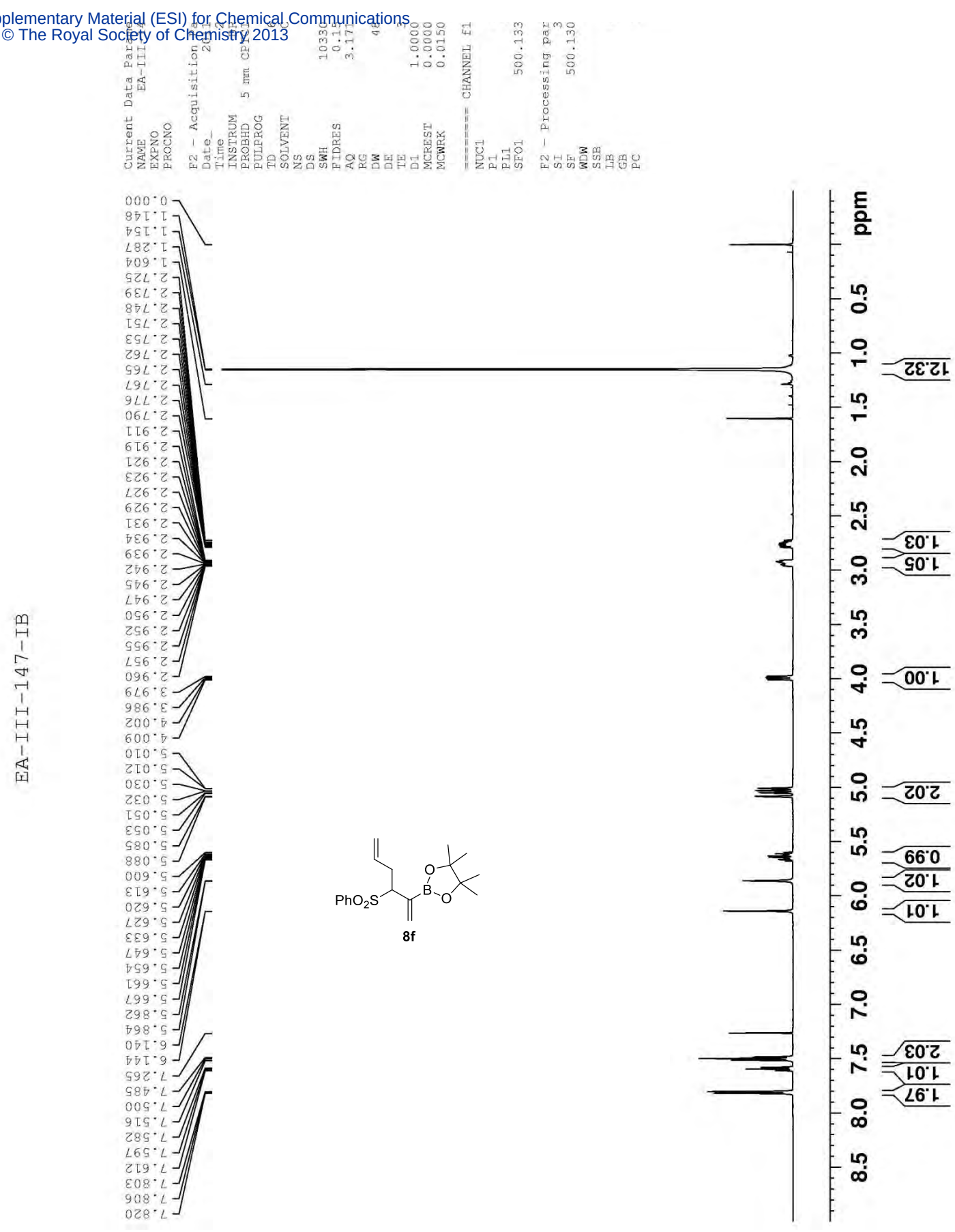

13C NMR
13.73
20.07
24.68
24.79
28.93
67.63
76.91
77.16
77.42
83.96
128.70
129.79
133.28
136.51
138.47
PhO2S
O
B
O
8c
ppm

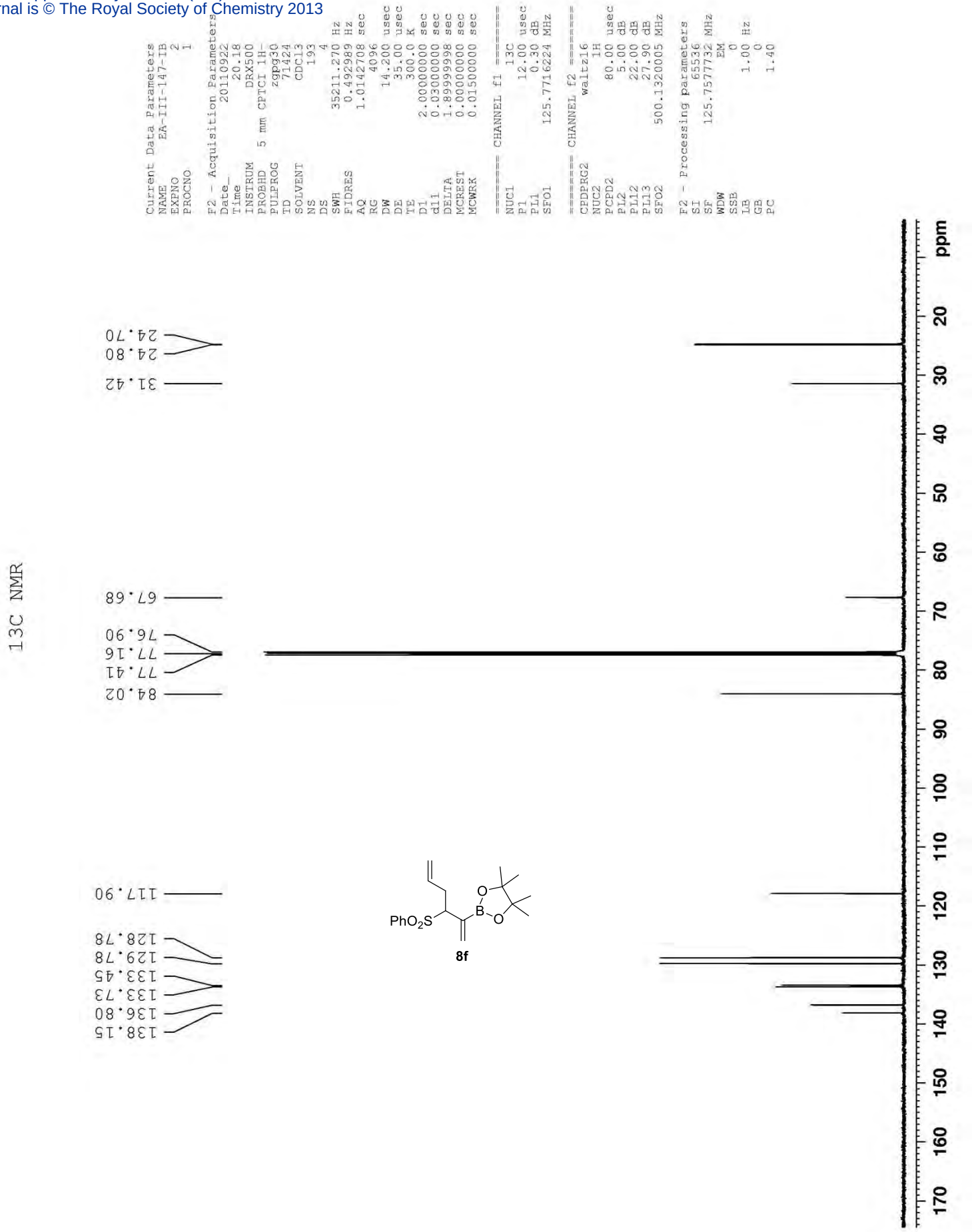

EA-III-142-IIIA
Current Data Parameters
NAME EA-III-142-IIIA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120105
Time 11.15
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.3
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz
F2 - Processing parameters
SI 32768
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Et
PhO2S
O
B
O
8d
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
1.98
2.02
1.00
1.00
1.01
1.00
1.00
1.04
1.10
4.98
12.18
3.06

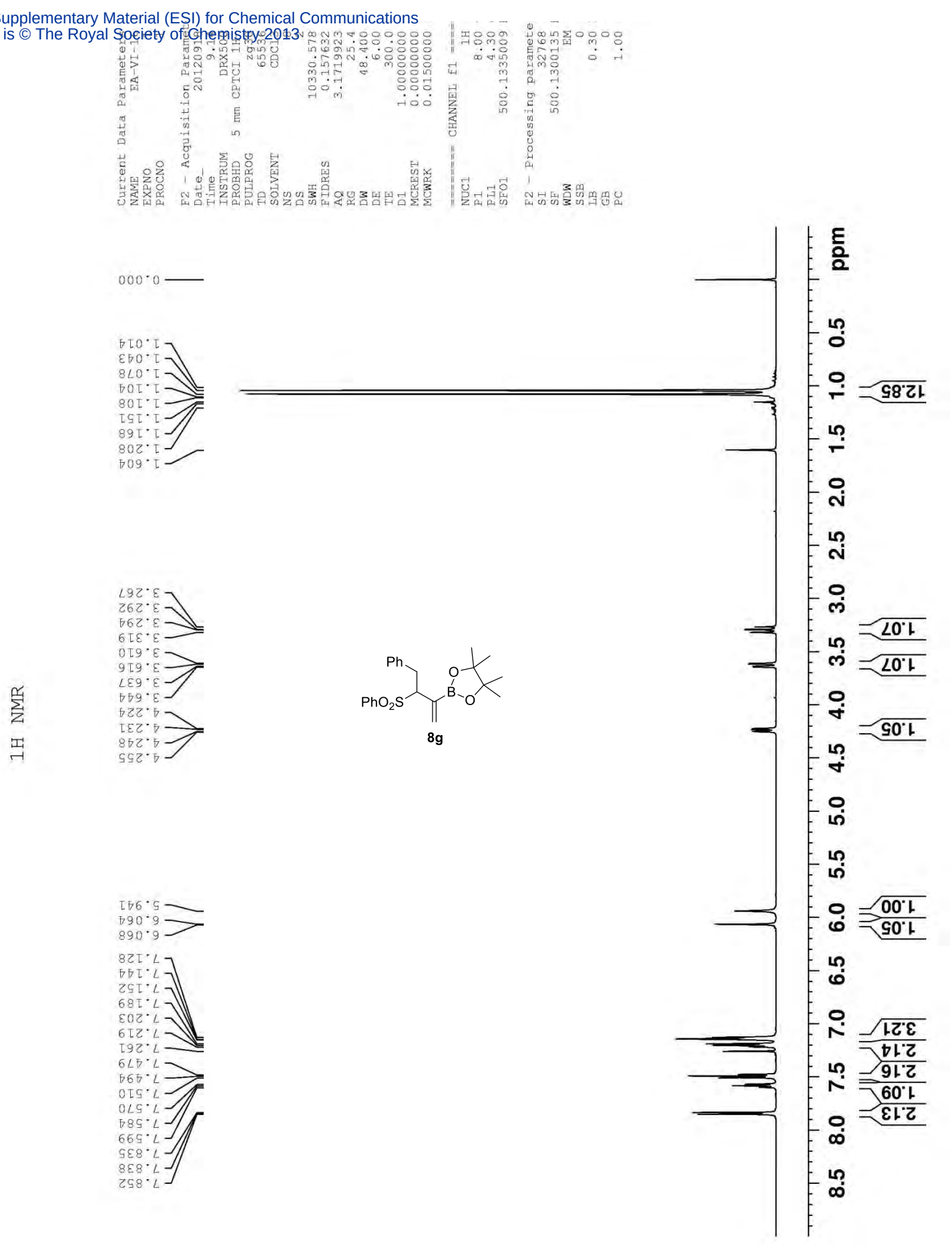


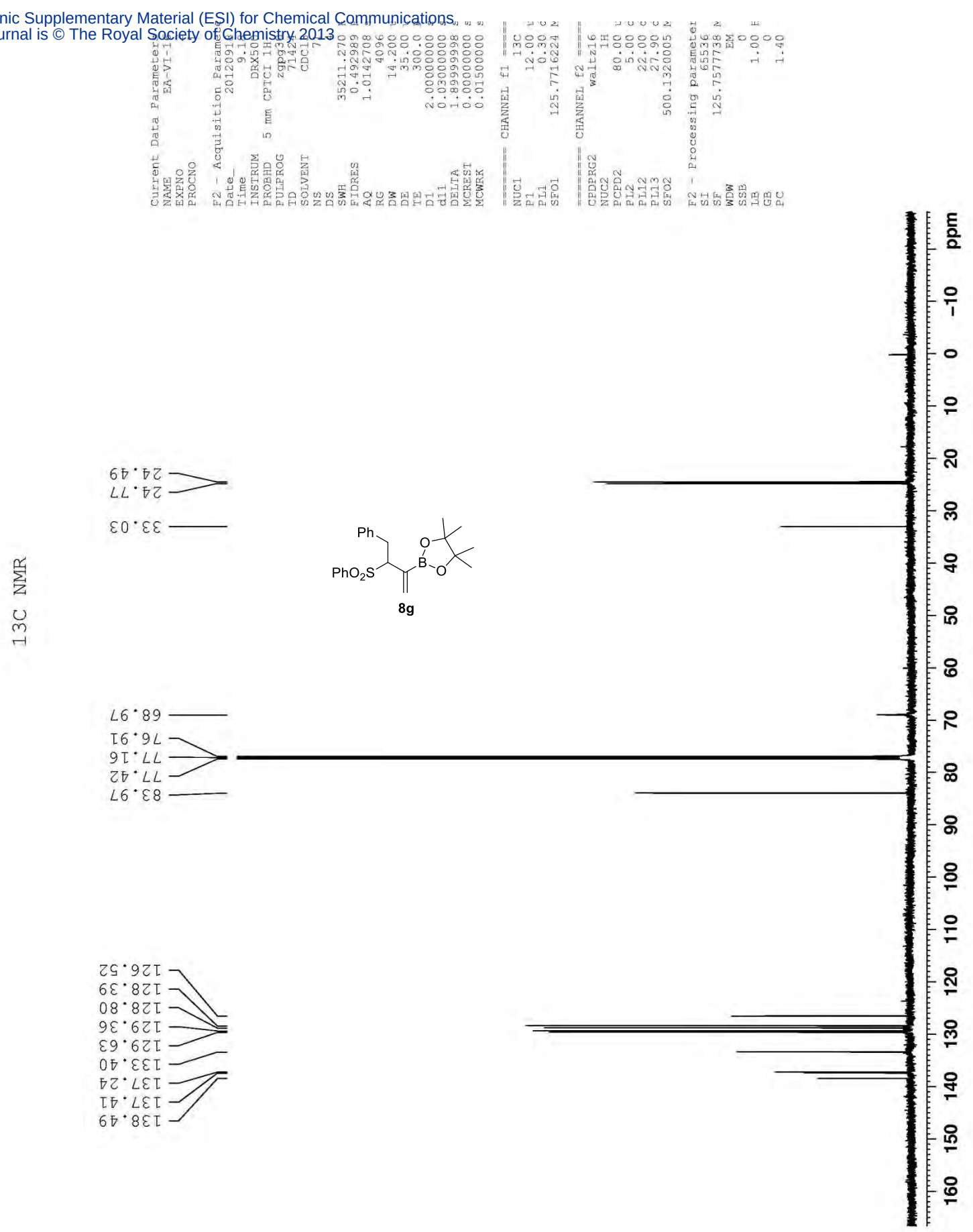

EA-III-146-IB1
Current Data Parameters
NAME EA-III-146-IB1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110921
Time 18.58
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 32
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.30 dB
SFO1 500.1335009 MHz
F2 - Processing parameters
SI 32768
SF 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
nBu
PhO2S
O
B
O
8e
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
2.08
1.06
2.04
1.01
1.00
1.01
1.07
1.05
11.15
12.33
3.23

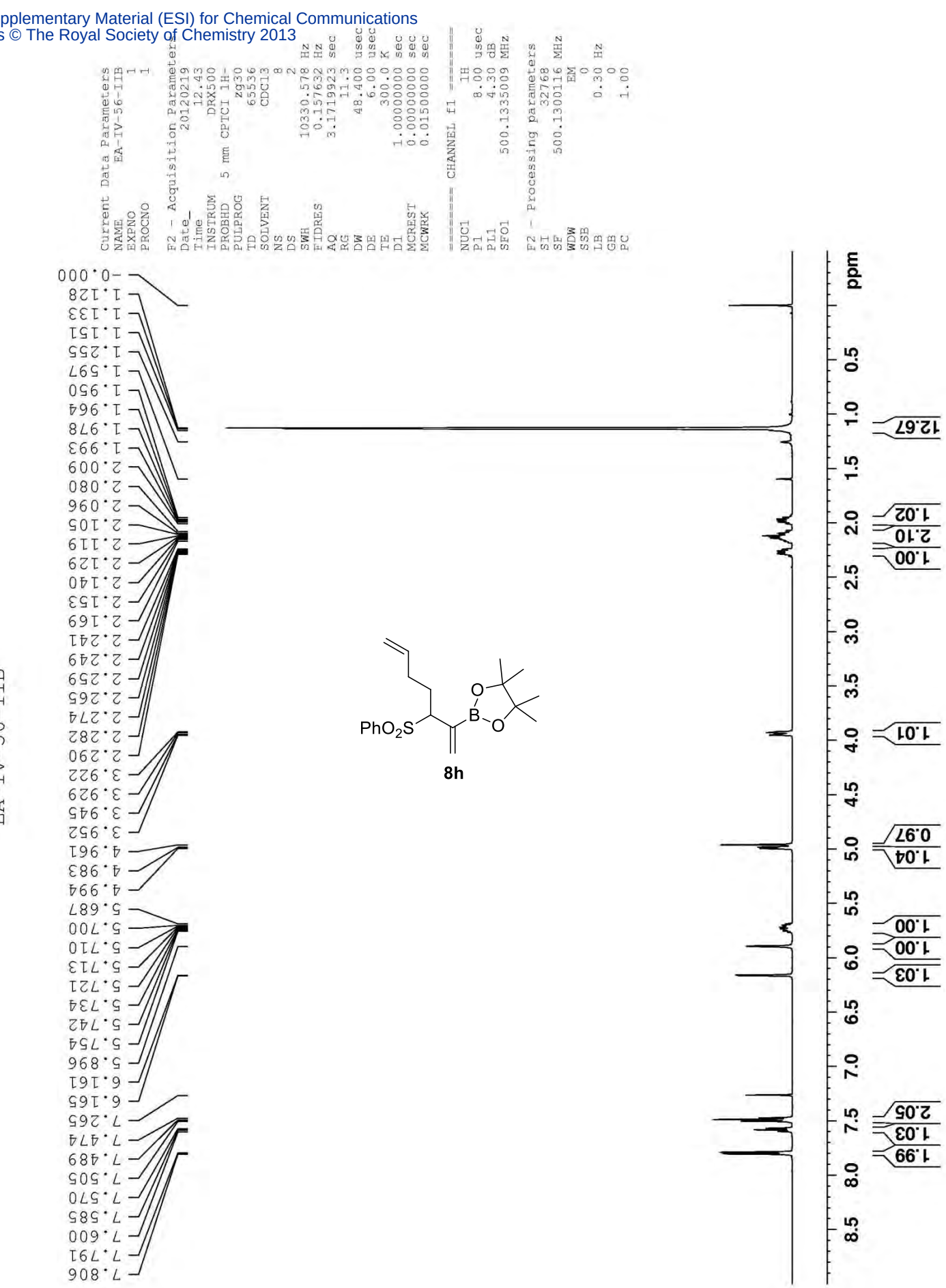

13C NMR
Current Data Parameters
NAME EA-III-146-IB1
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110921
Time 19.12
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 131
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
14.20
22.72
24.69
24.80
26.75
26.87
29.08
29.20
31.86
67.94
76.91
77.16
77.41
83.96
128.70
129.78
133.26
136.51
138.51
nBu
PhO2S
O
B
O
8e
ppm

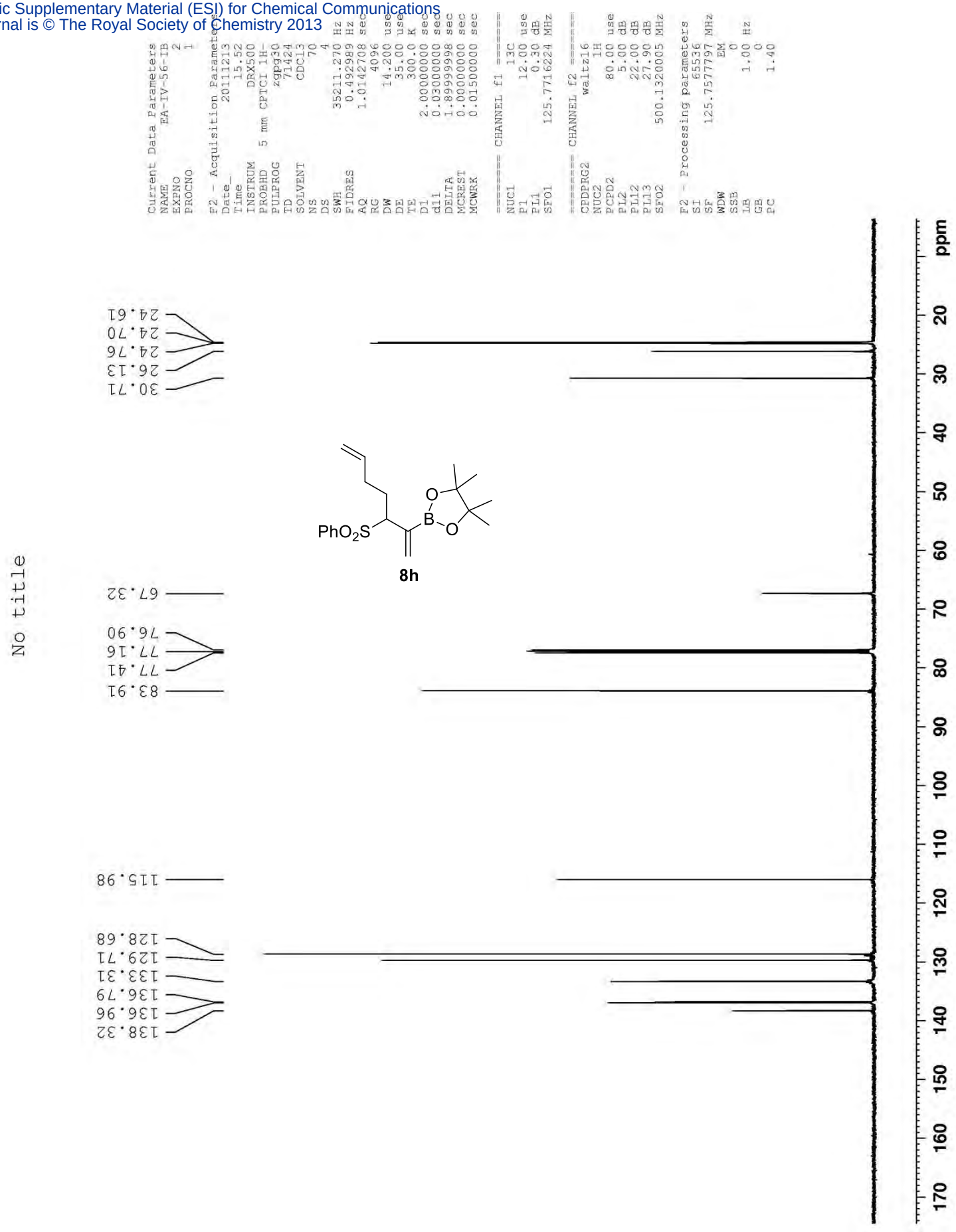


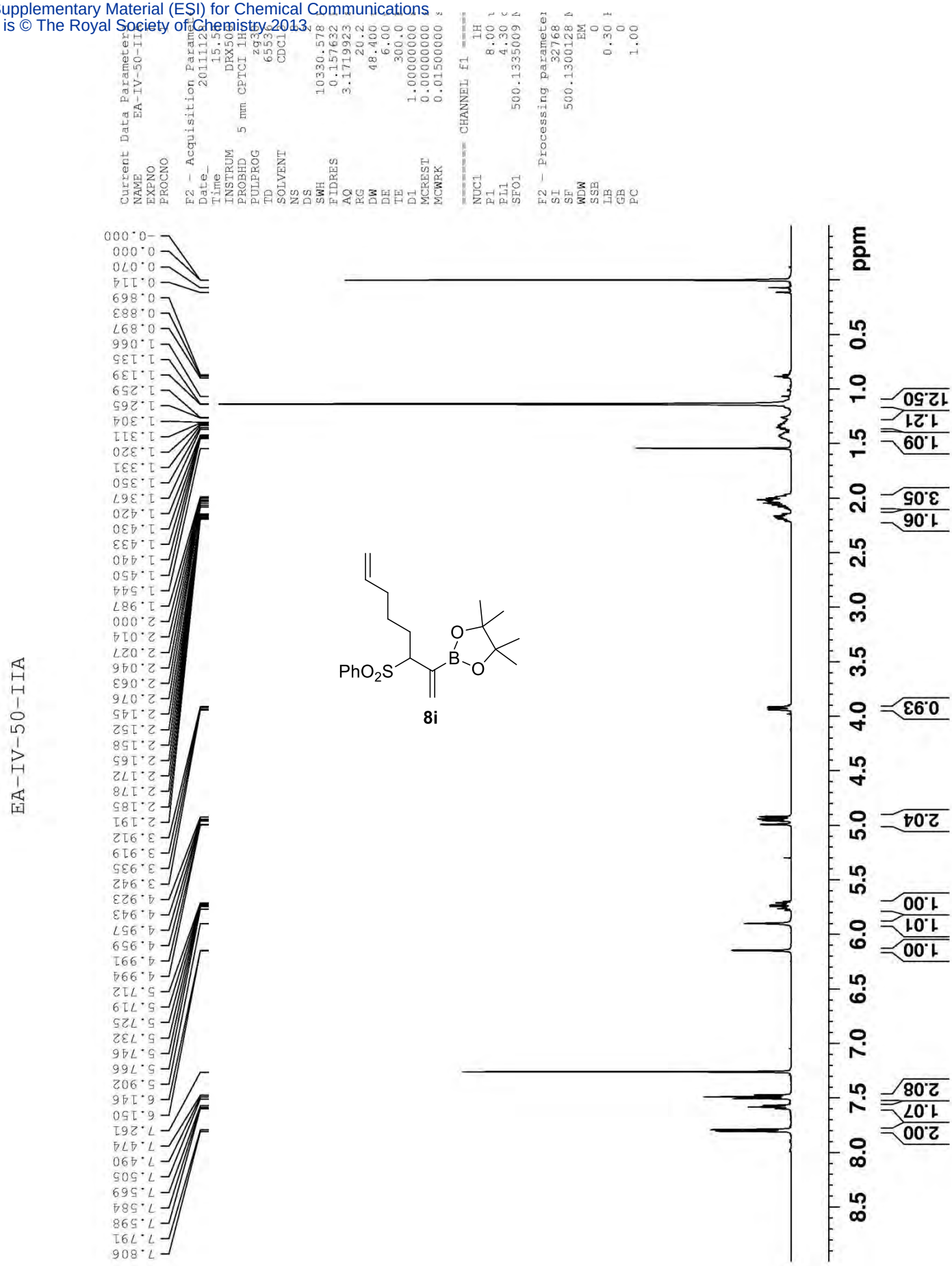

13C NMR
Current Data Parameters
NAME EA-III-147-IB
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110922
Time 20.18
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 193
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
24.70
24.80
31.42
67.68
76.90
77.16
77.41
84.02
117.90
128.78
129.78
133.45
133.73
136.80
138.15
PhO2S
O
B
O
8f
ppm

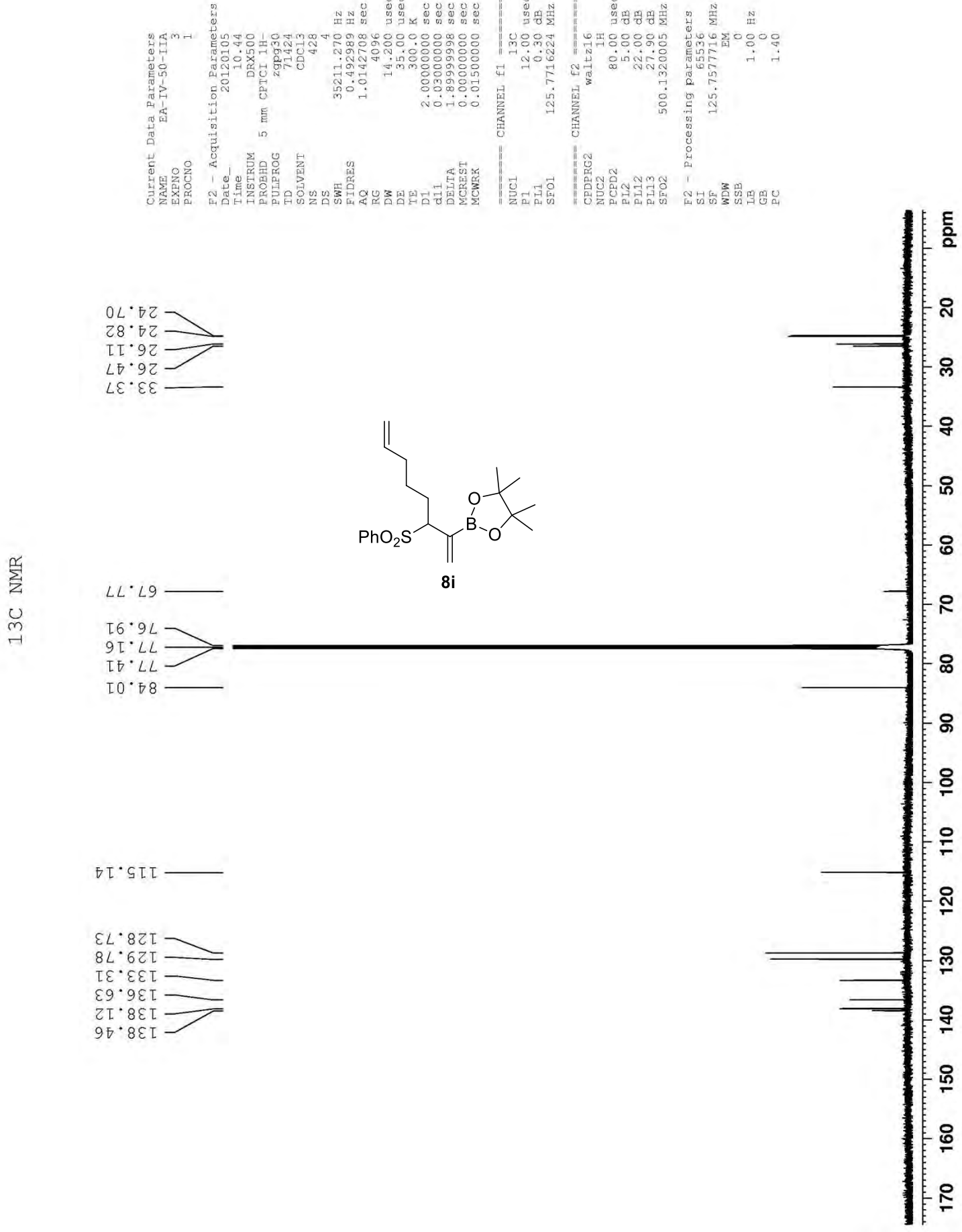


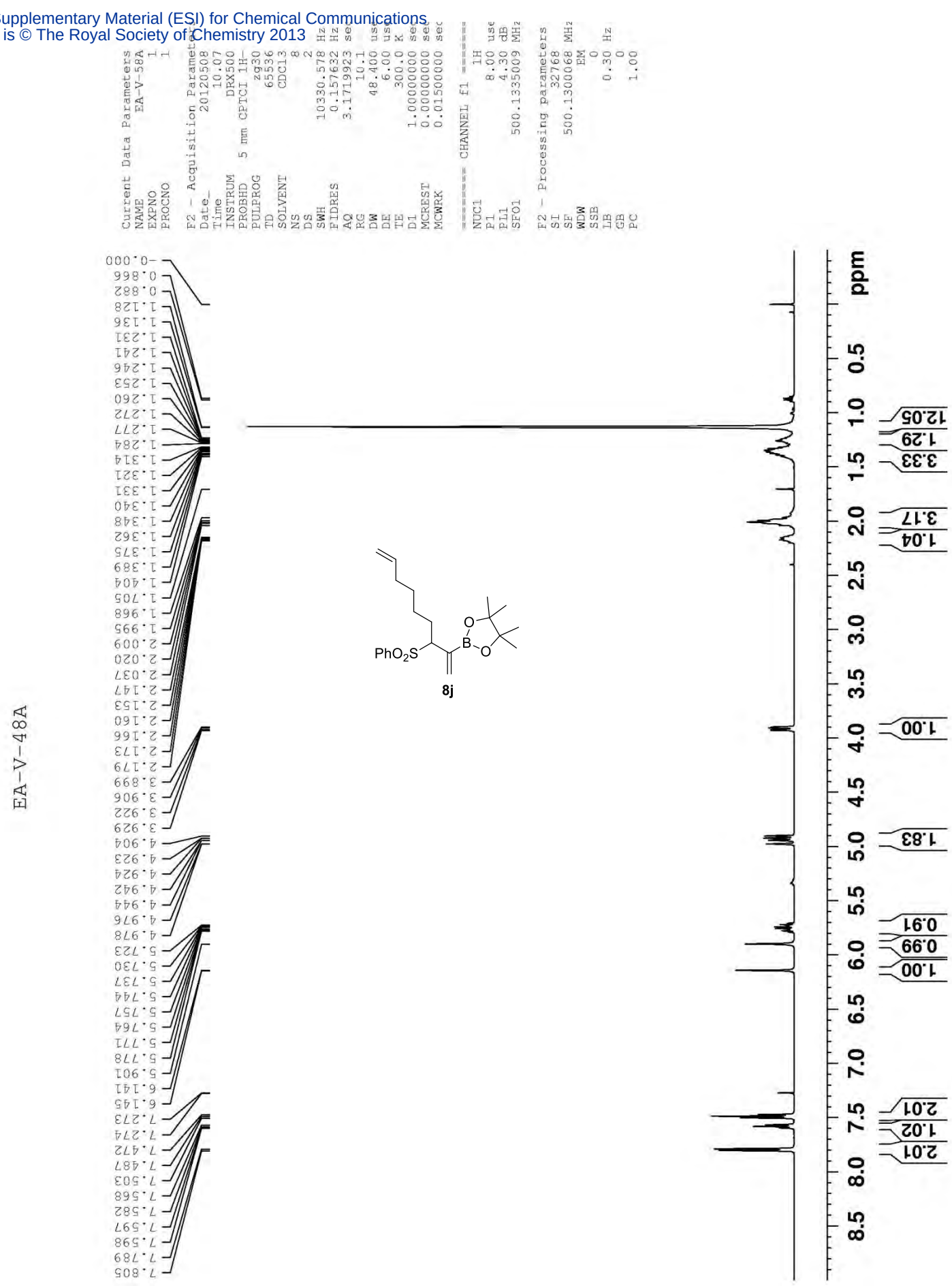

13C NMR
Ph
PhO2S
O
B
O
8g
24.49
24.77
33.03
68.97
76.91
77.16
77.42
83.97
126.52
128.39
128.80
129.36
129.63
133.40
137.24
137.41
138.49
ppm



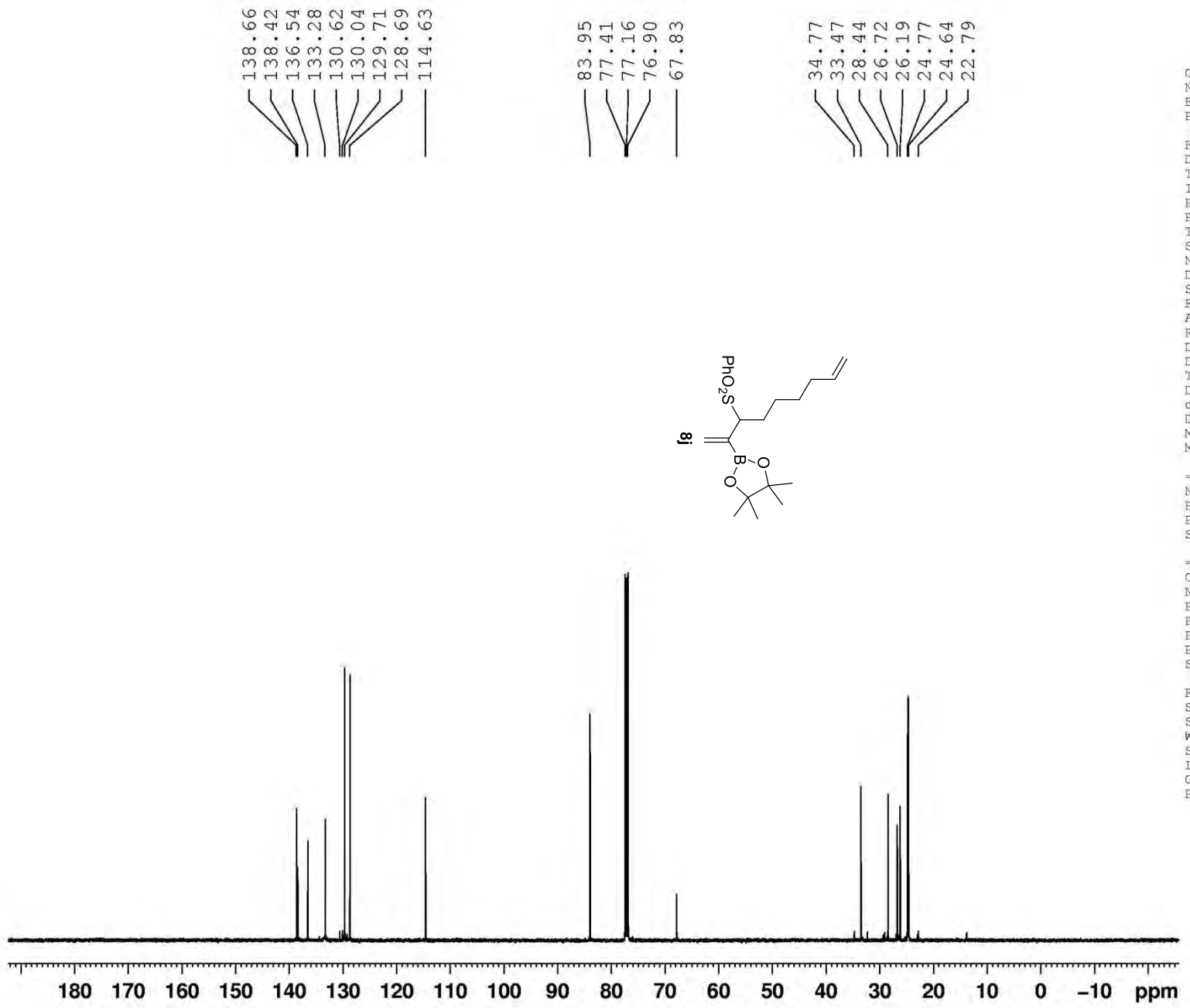

No title
Current Data Parameters
NAME EA-IV-56-IB
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111213
Time 15.52
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 70
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 use
DE 35.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 use
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577797 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
PhO2S
O
B
O
8h
24.61
24.70
24.76
26.13
30.71
67.32
76.90
77.16
77.41
83.91
115.98
128.68
129.71
133.31
136.79
136.96
138.32
ppm
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170



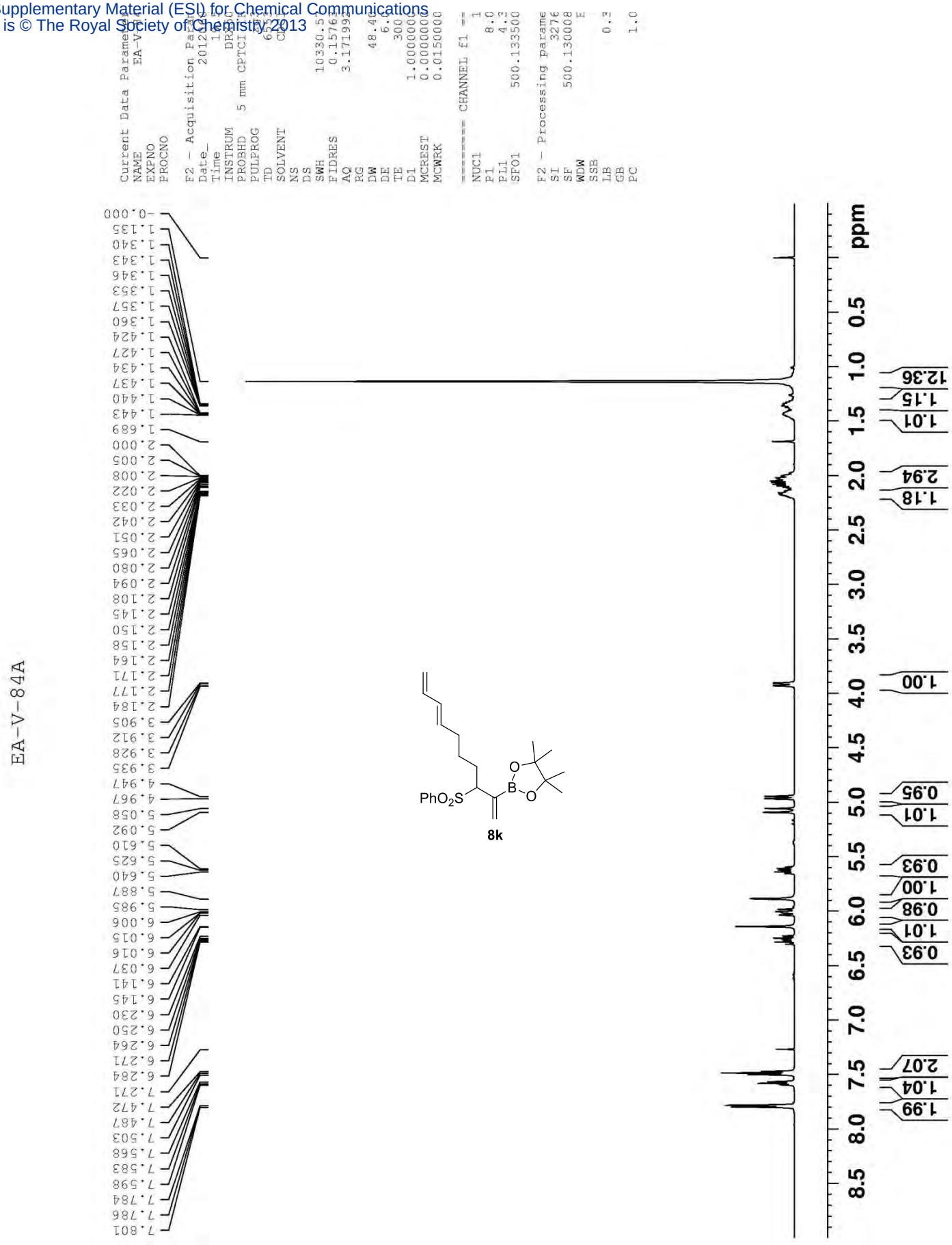


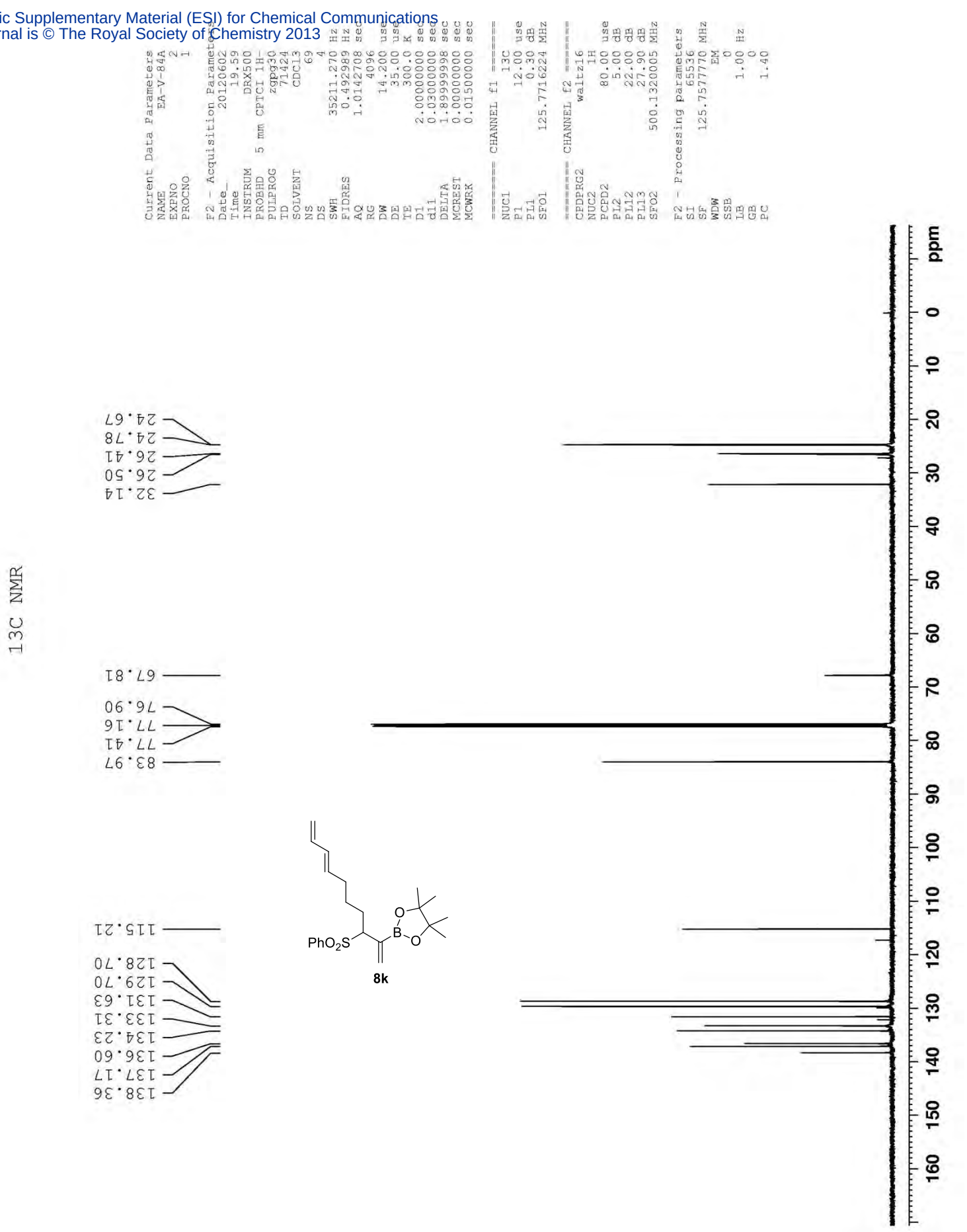

EA-V-48A
Current Data Parameters
NAME EA-V-58A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120508
Time 10.07
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 se
RG 10.1
DW 48.400 use
DE 6.00 use
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 use
PL1 4.30 dB
SF01 500.1335009 MHz
F2 - Processing parameters
SI 32768
SF 500.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
PhO2S
O
B
O
8j
ppm
12.05
1.29
3.33
3.17
1.04
1.00
1.83
0.91
0.99
1.00
2.01
1.02
2.01

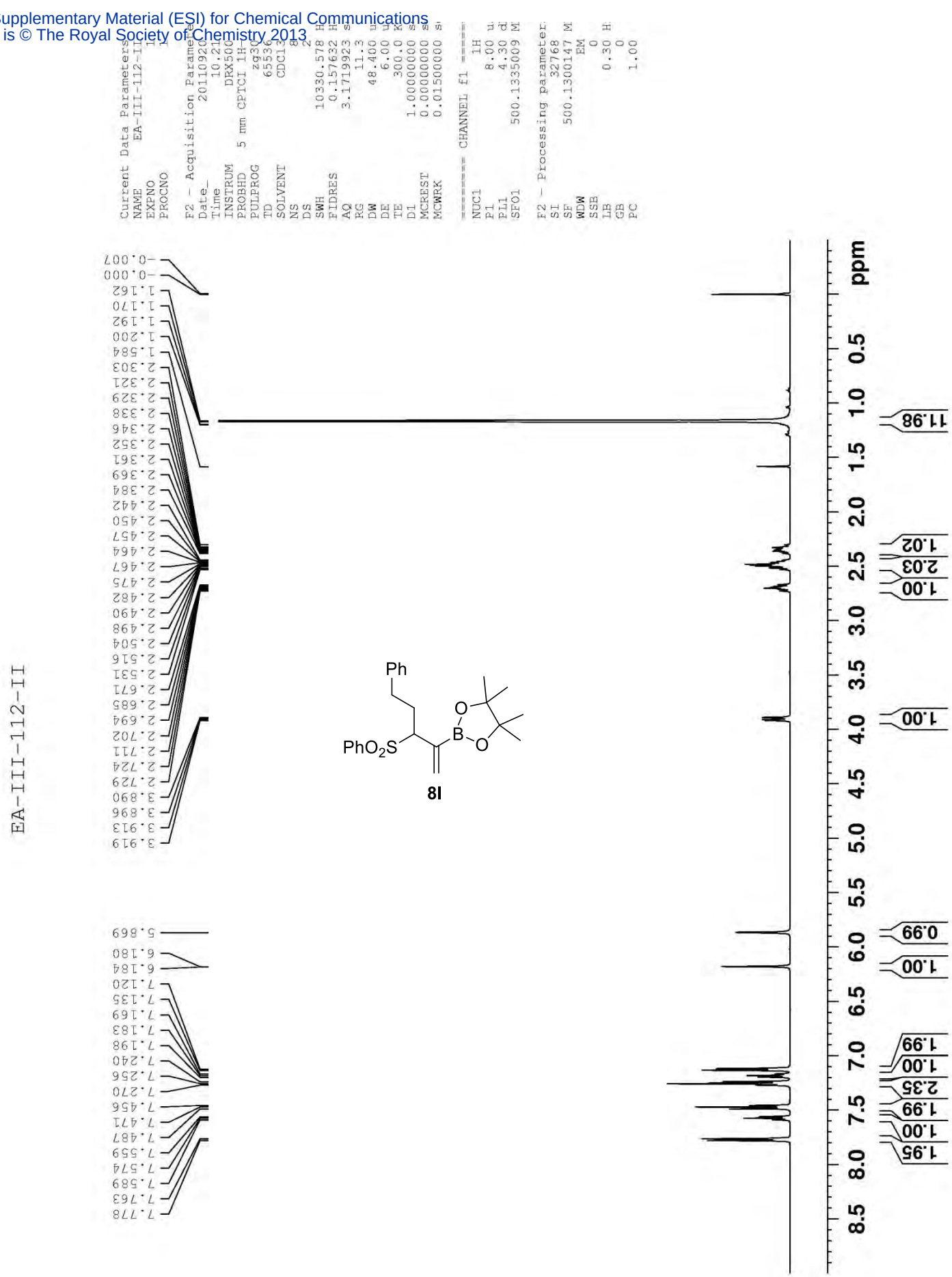
13C NMR

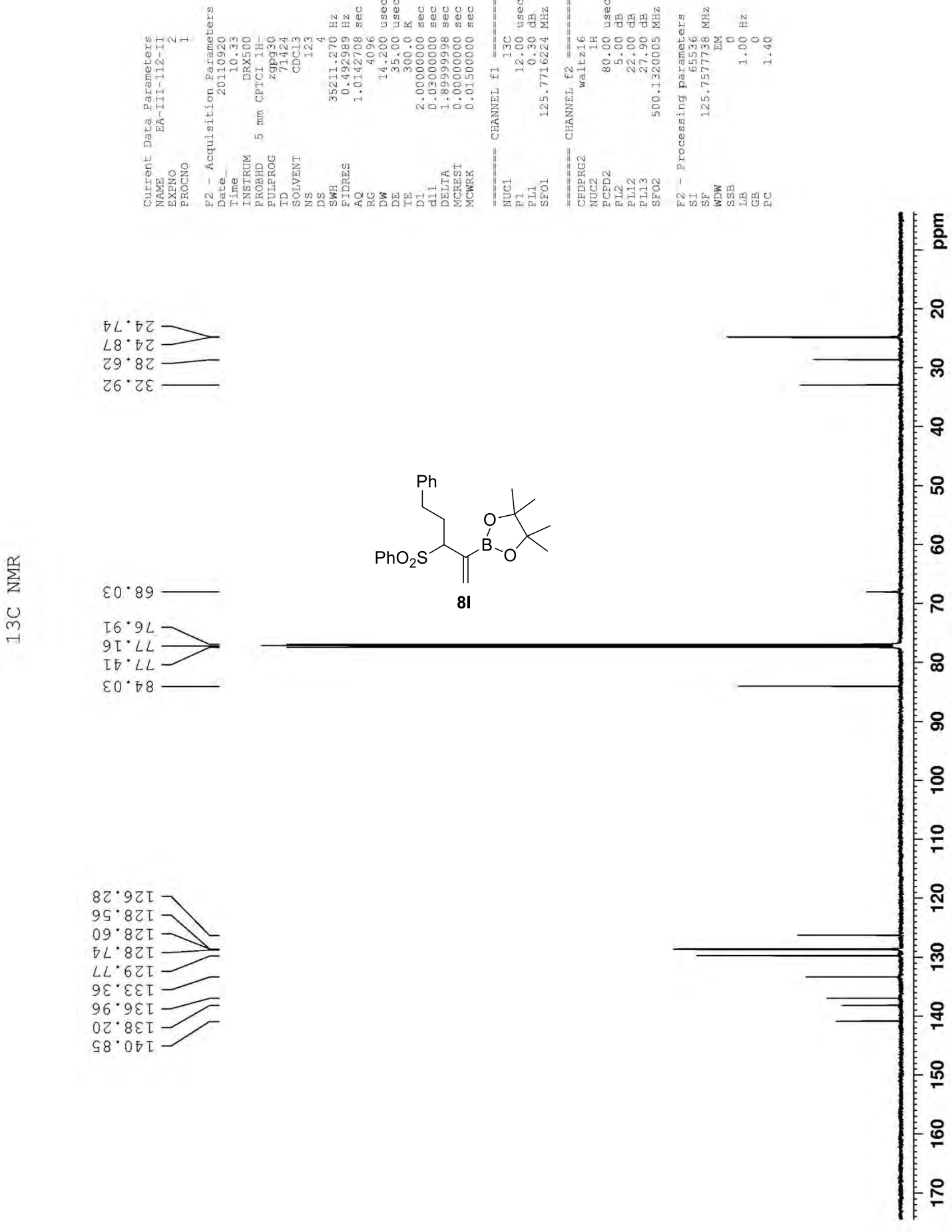


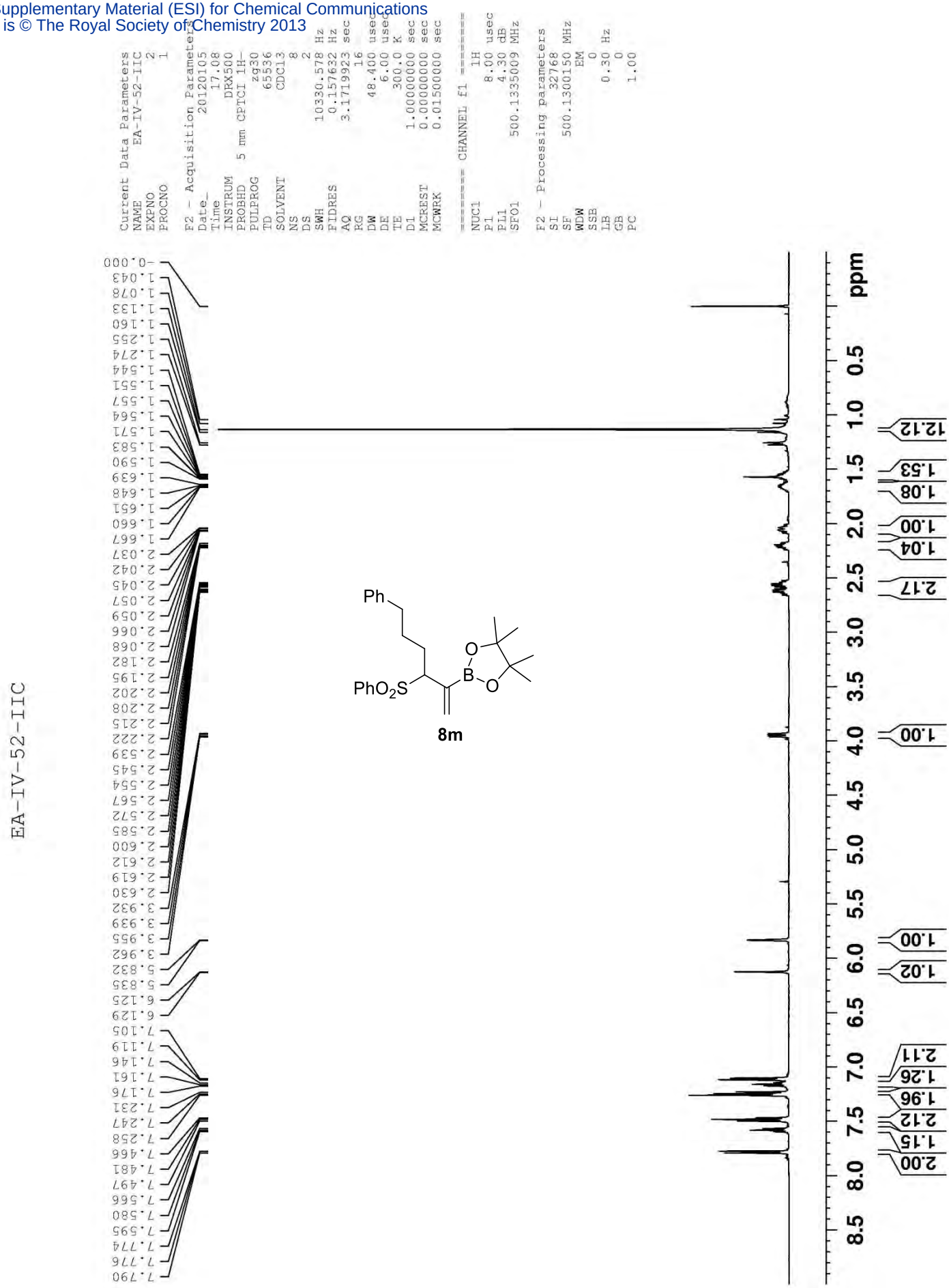

```
Current Data Parameters
NAME                  EA-V-50B
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120501
Time                     20.22
INSTRUM                DRX500
PROBHD    5 mm CPTCI 1H-
PULPROG                zgpg30
TD                      71424
SOLVENT                 CDCl3
NS                         61
DS                          4
SWH                35211.270 Hz
FIDRES             0.492989 Hz
AQ                 1.0142708 sec
RG                       4096
DW                     14.200 usec
DE                     35.00 usec
TE                     300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     12.00 usec
PL1                     0.30 dB
SFO1           125.7716224 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     5.00 dB
PL12                   22.00 dB
PL13                   27.90 dB
SFO2           500.1320005 MHz

F2 - Processing parameters
SI                        65536
SF             125.7577765 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40
```

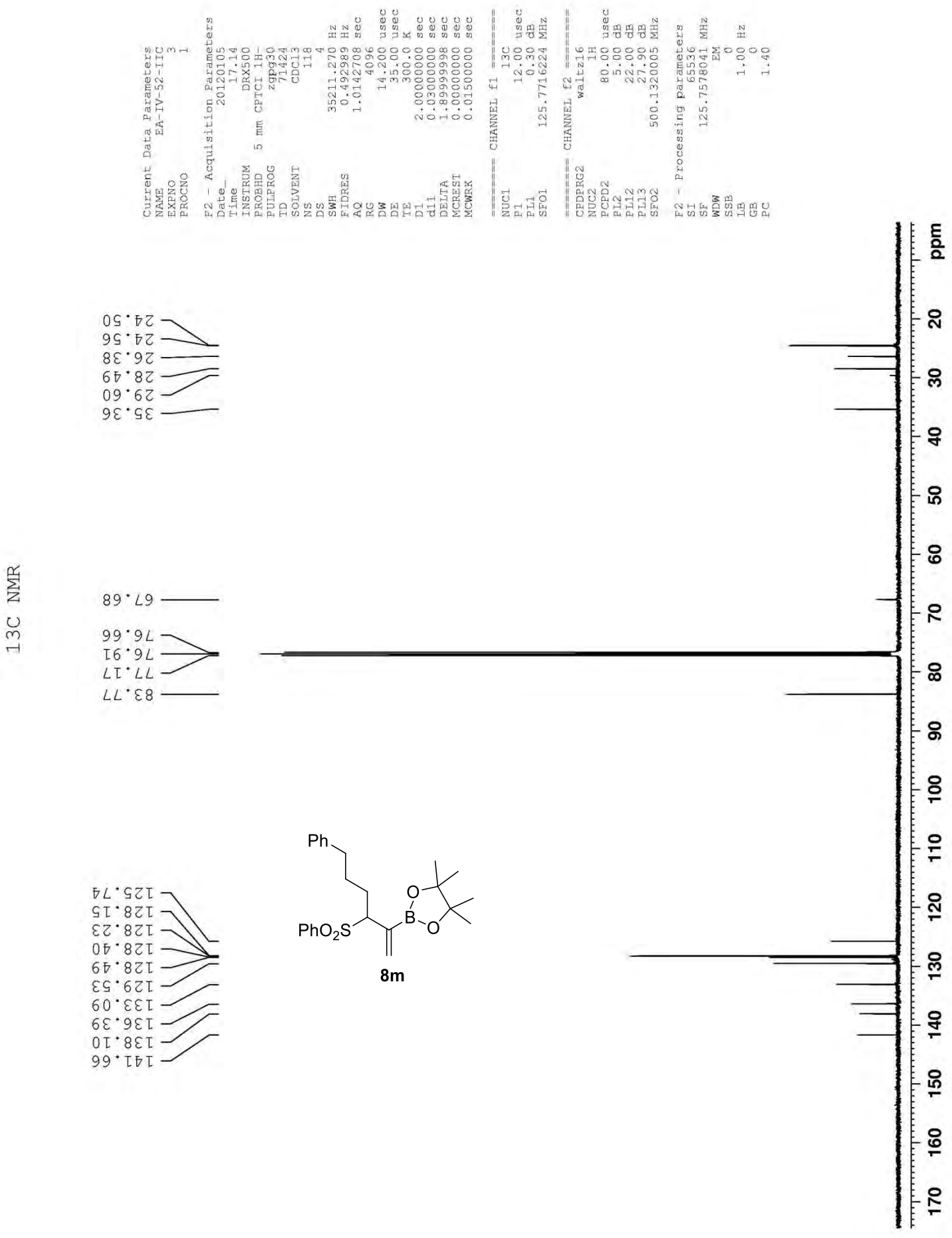


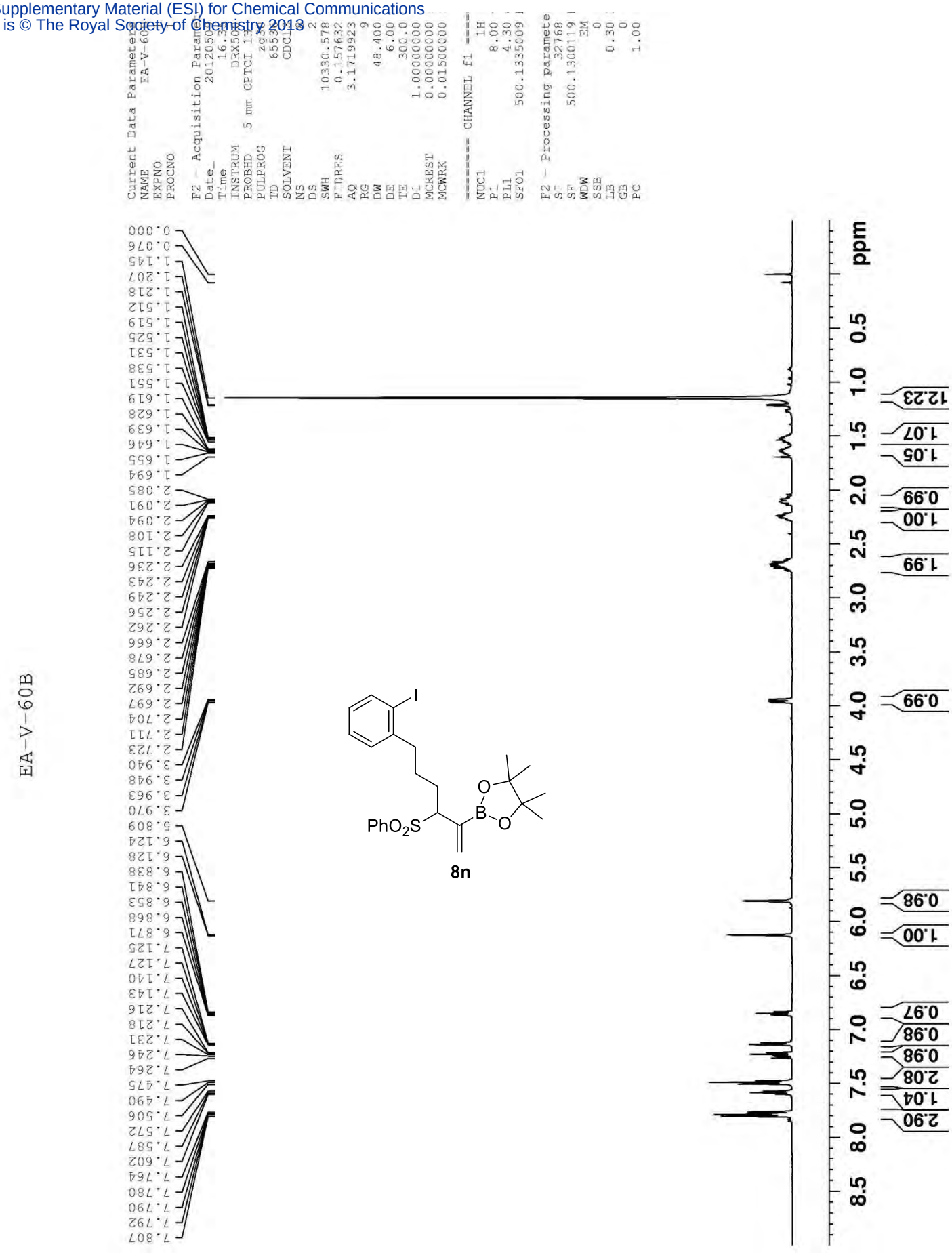


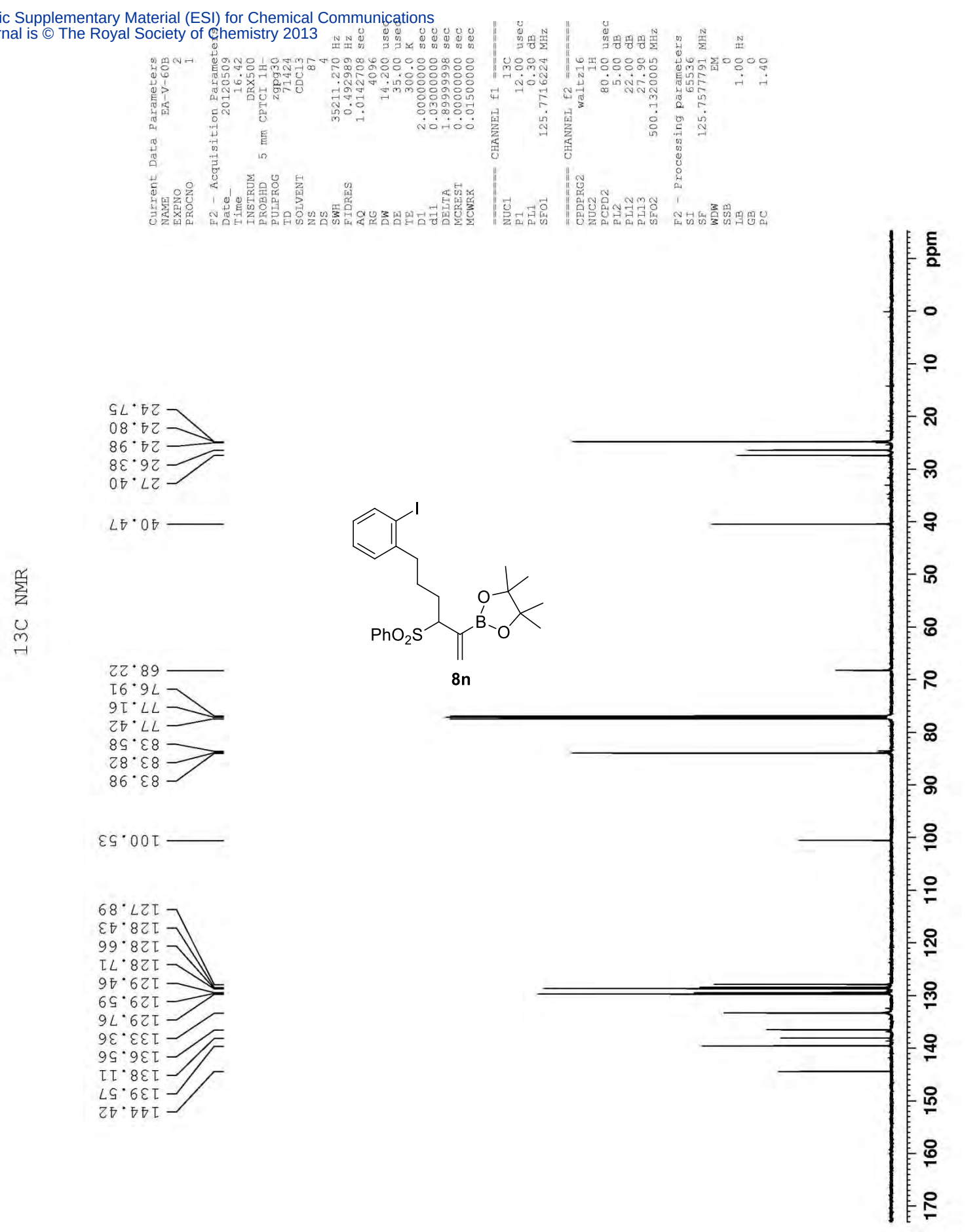

EA-III-112-II
Current Data Parameters
NAME EA-III-112-II
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110926
Time 10.21
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 H
FIDRES 0.157632 H
AQ 3.1719923 s
RG 11.3
DW 48.400 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
MCREST 0.00000000 s
MCWRK 0.01500000 s
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 u
PL1 4.30 d
SFO1 500.1335009 M
F2 - Processing parameters
SI 32768
SF 500.1300147 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
Ph
PhO2S
O
B
O
8l
ppm
11.98
1.02
2.03
1.00
1.00
0.99
1.00
1.99
1.00
2.35
1.99
1.00
1.95

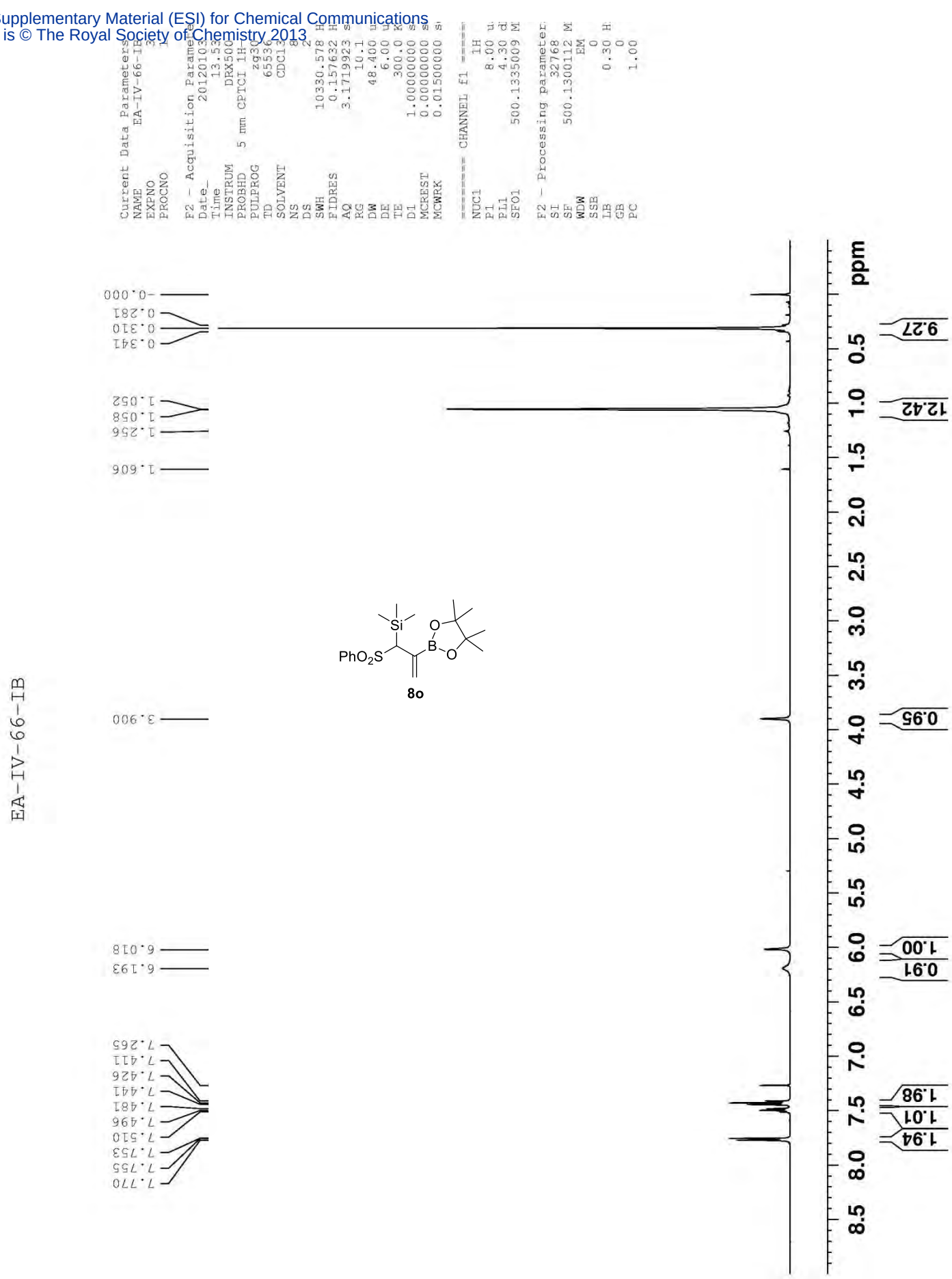

13C NMR
Current Data Parameters
NAME EA-III-112-II
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110920
Time 10.33
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 123
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577738 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Ph
PhO2S
O
B
O
8l
32.92
28.62
24.87
24.74
68.03
77.41
77.16
76.91
84.03
126.28
128.56
128.60
128.74
129.77
133.36
136.96
138.20
140.85
ppm

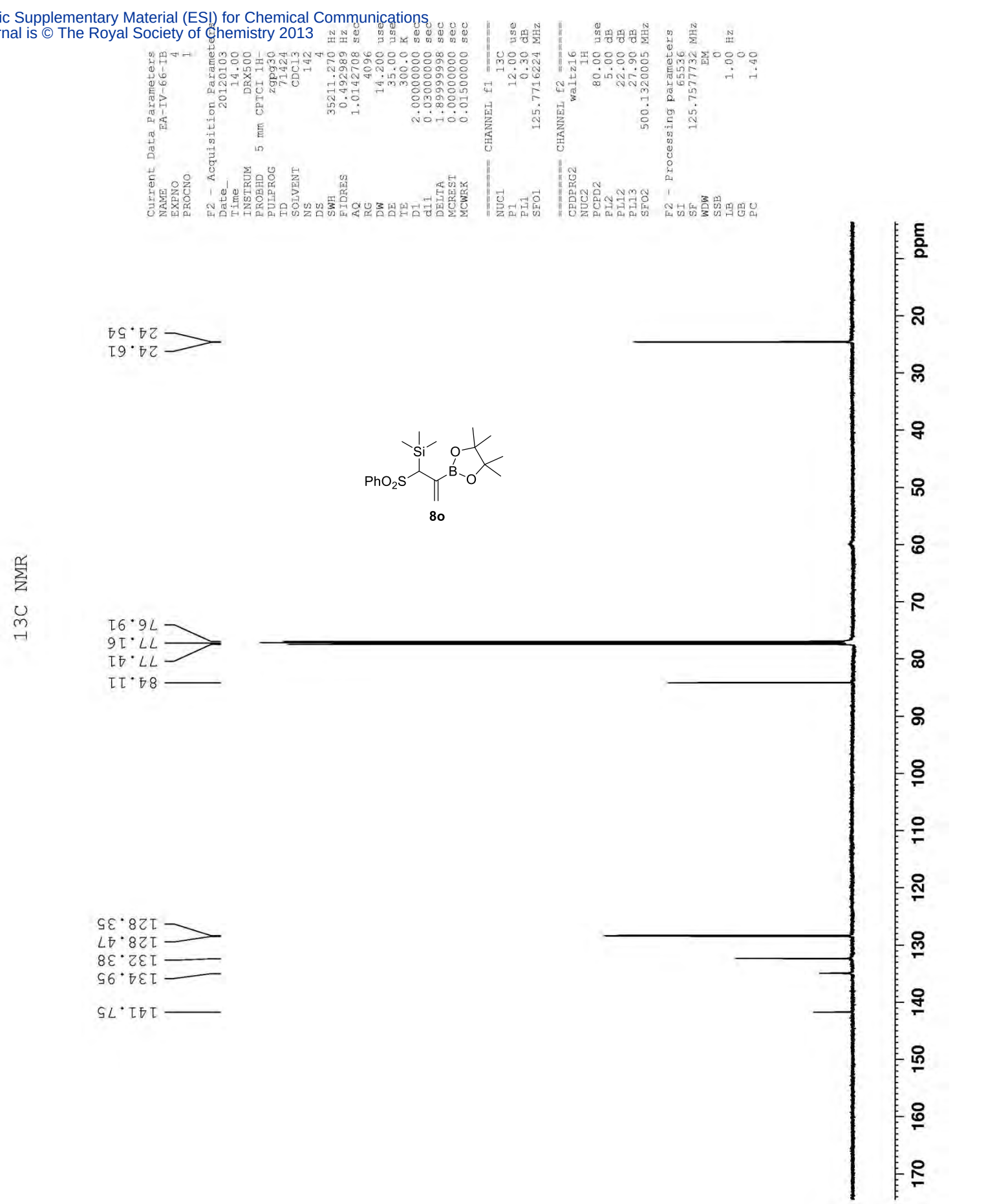


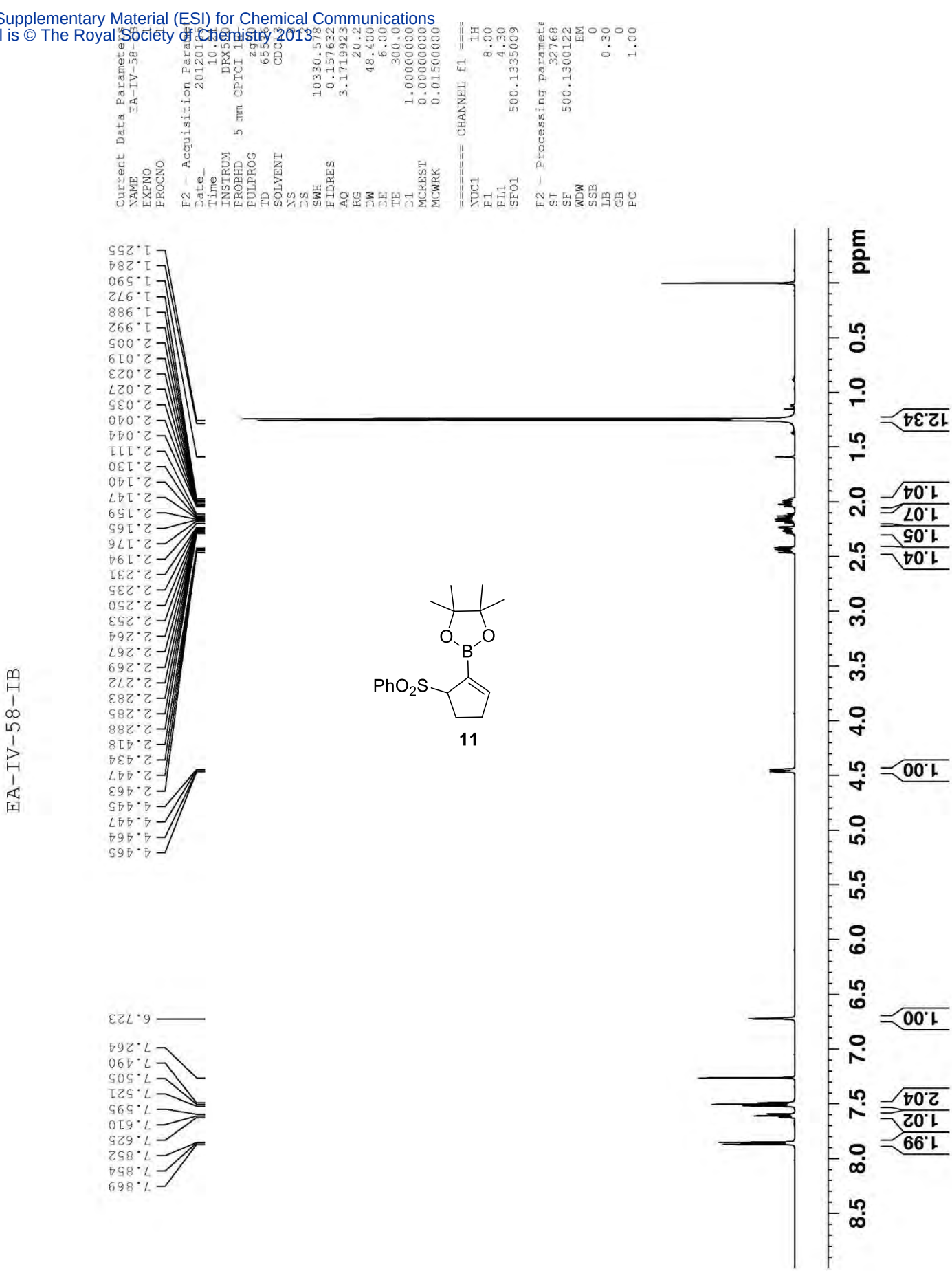


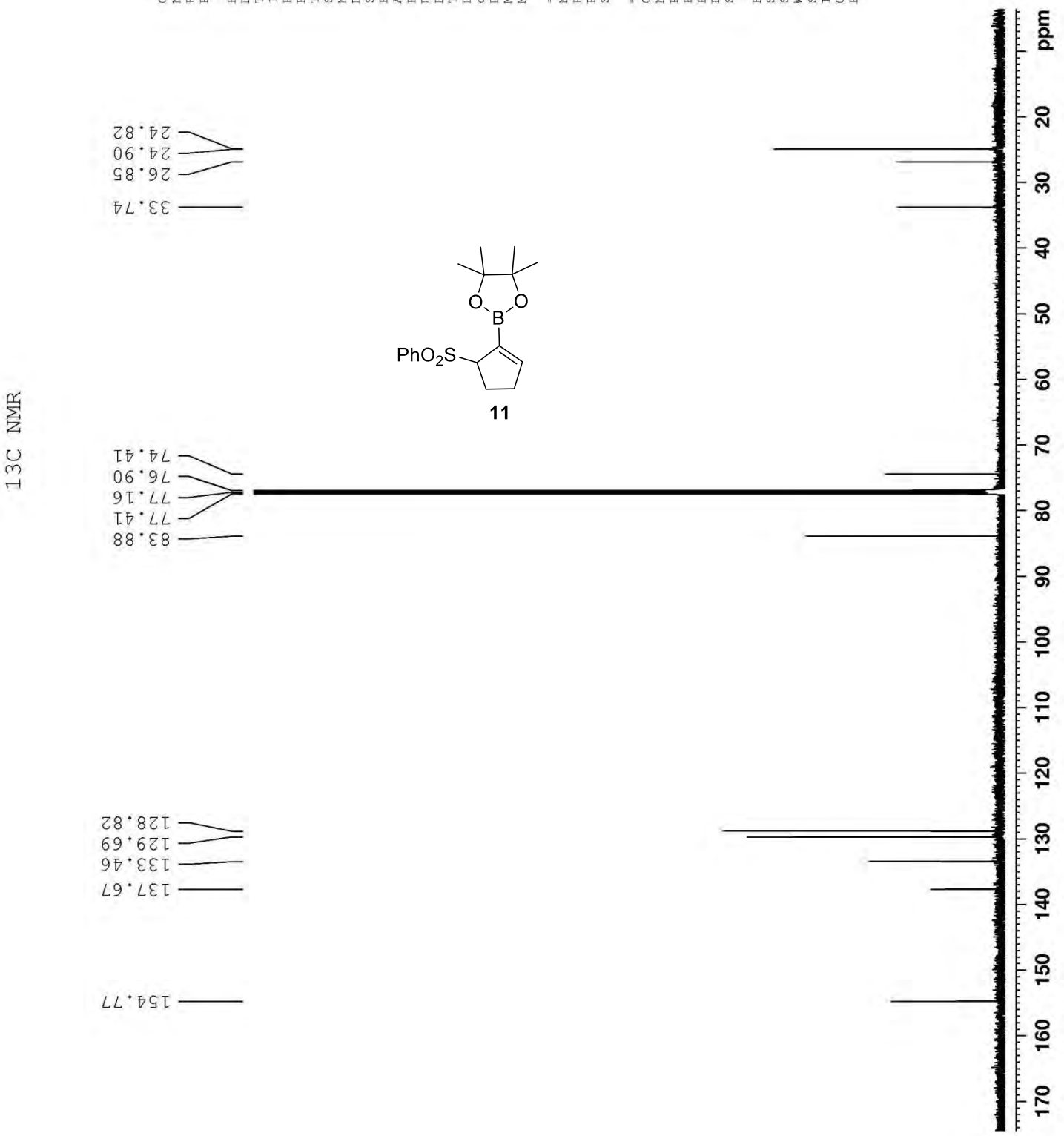

EA-V-60B
8n
ppm

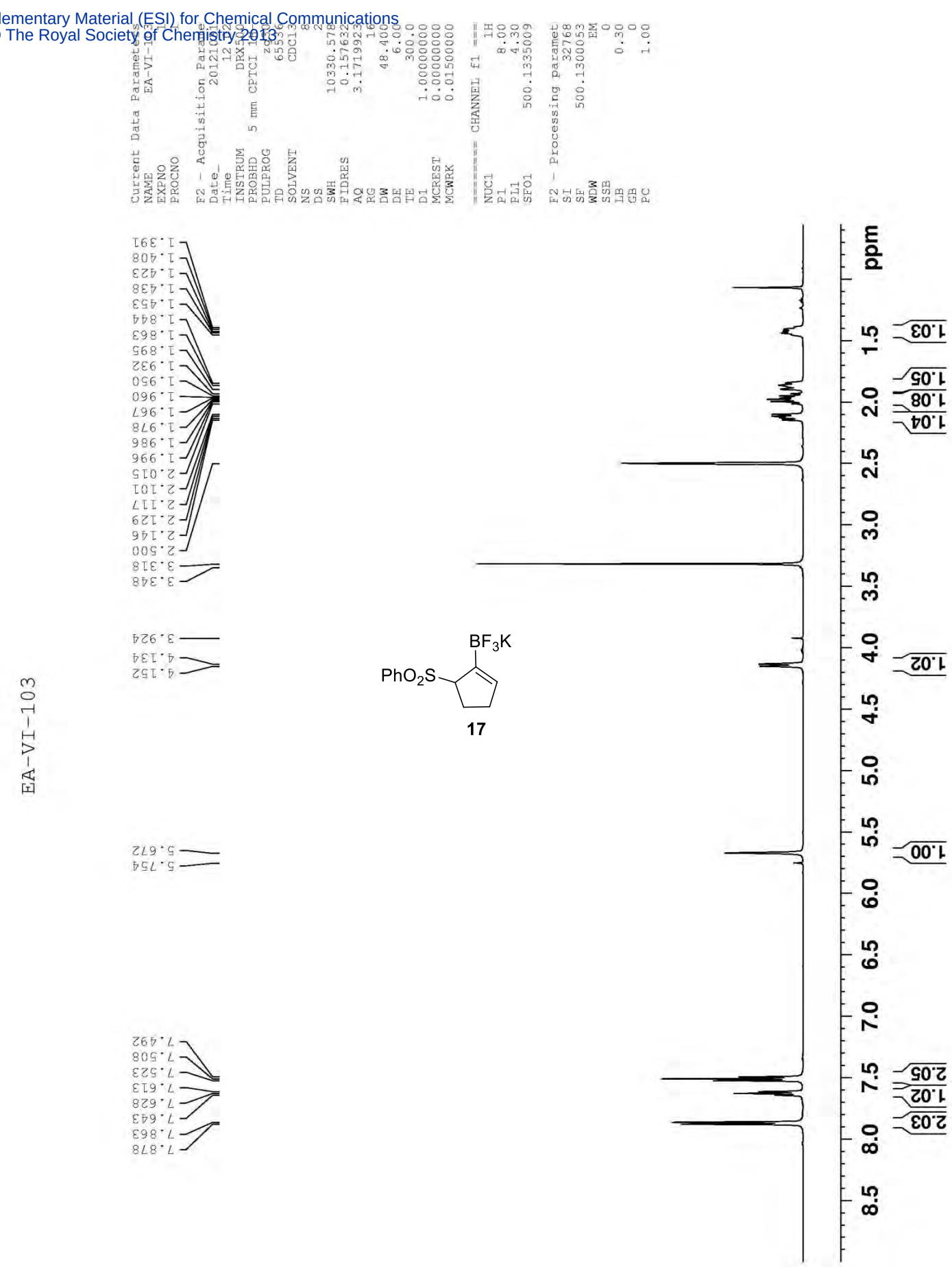


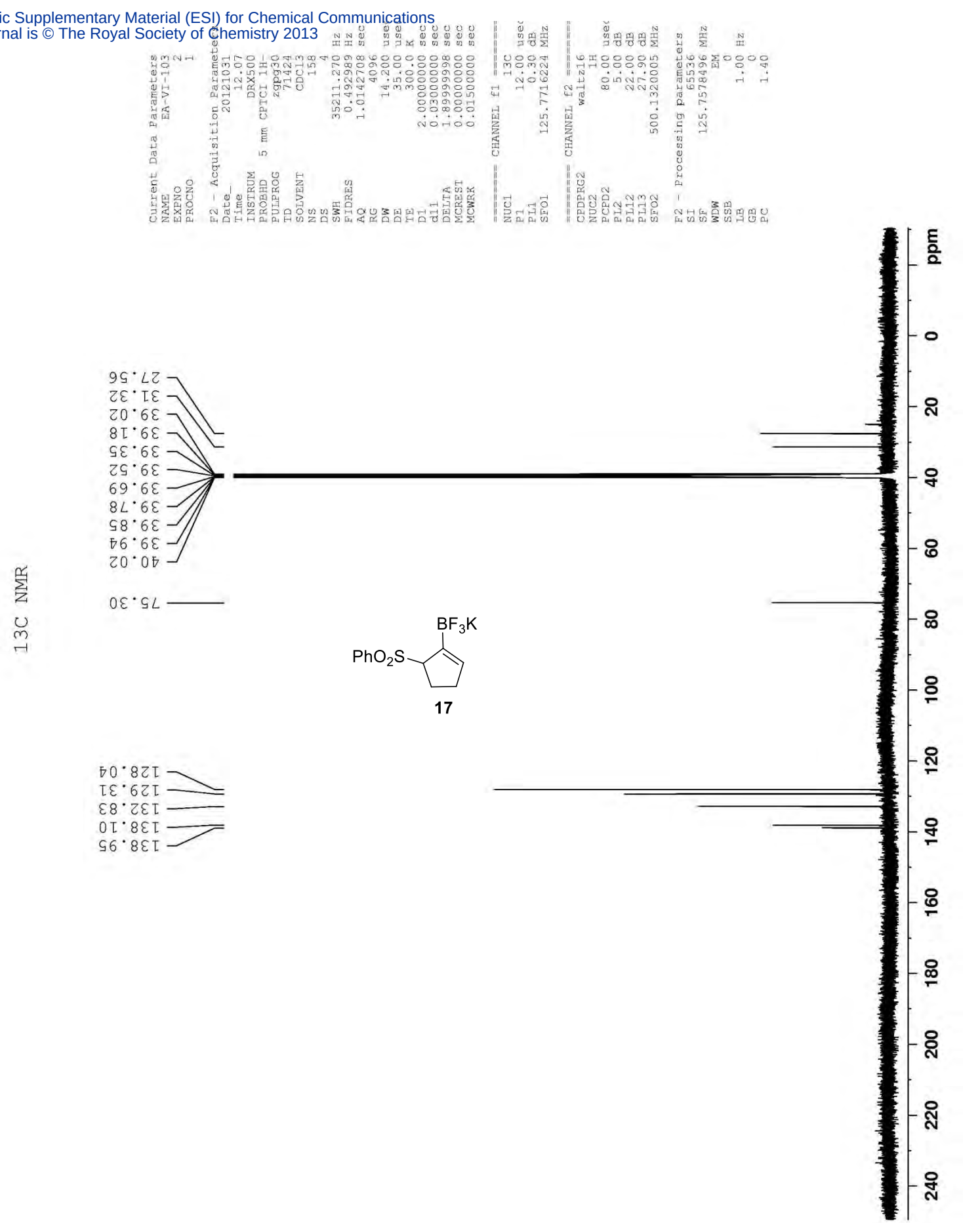


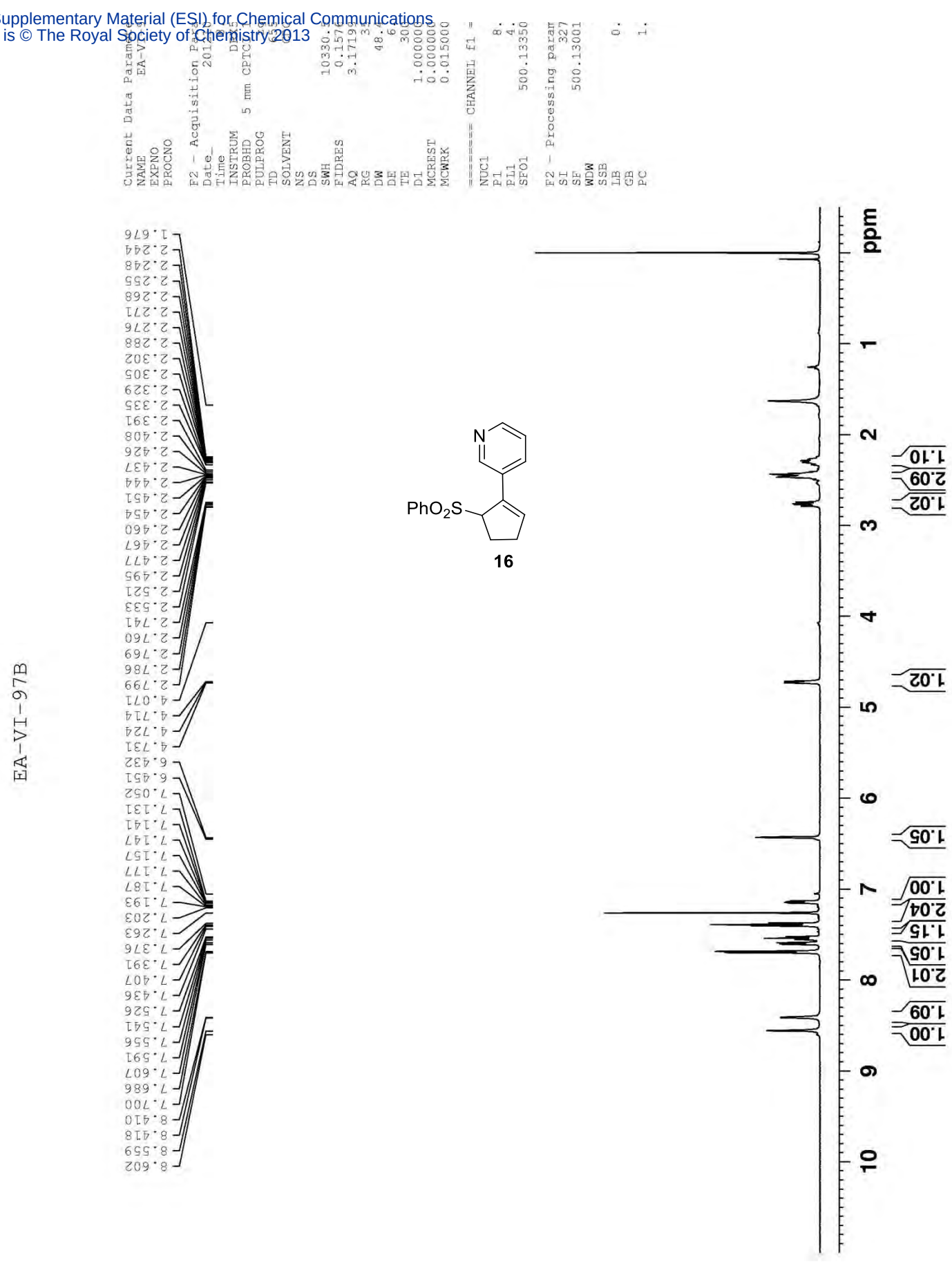

13C NMR
Current Data Parameters
NAME EA-IV-66-IB
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120103
Time 14.00
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 142
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 use
DE 35.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 use
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577732 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
24.54
24.61
76.91
77.16
77.41
84.11
128.35
128.47
132.38
134.95
141.75
Si
PhO2S
O
B
O
8o
ppm

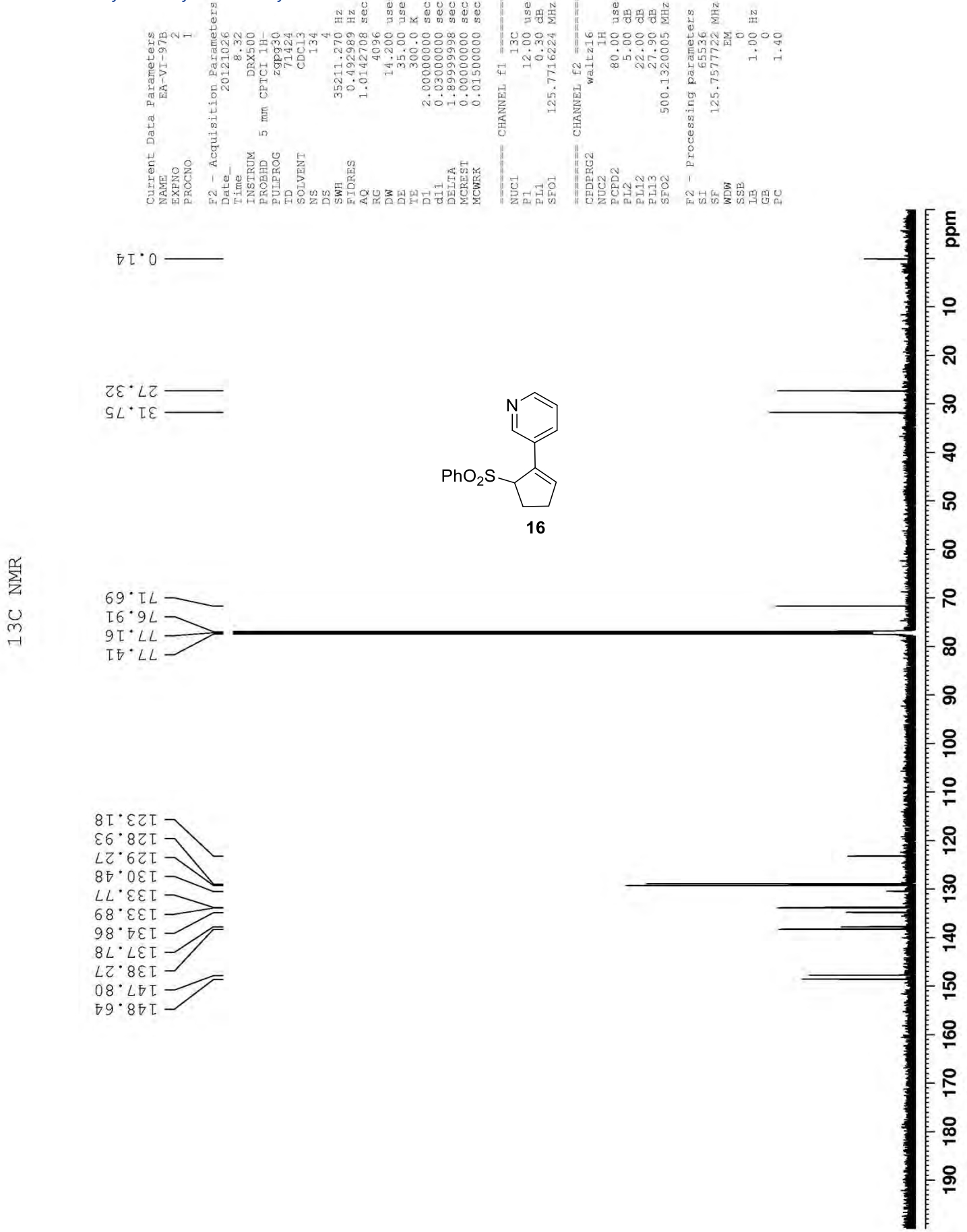


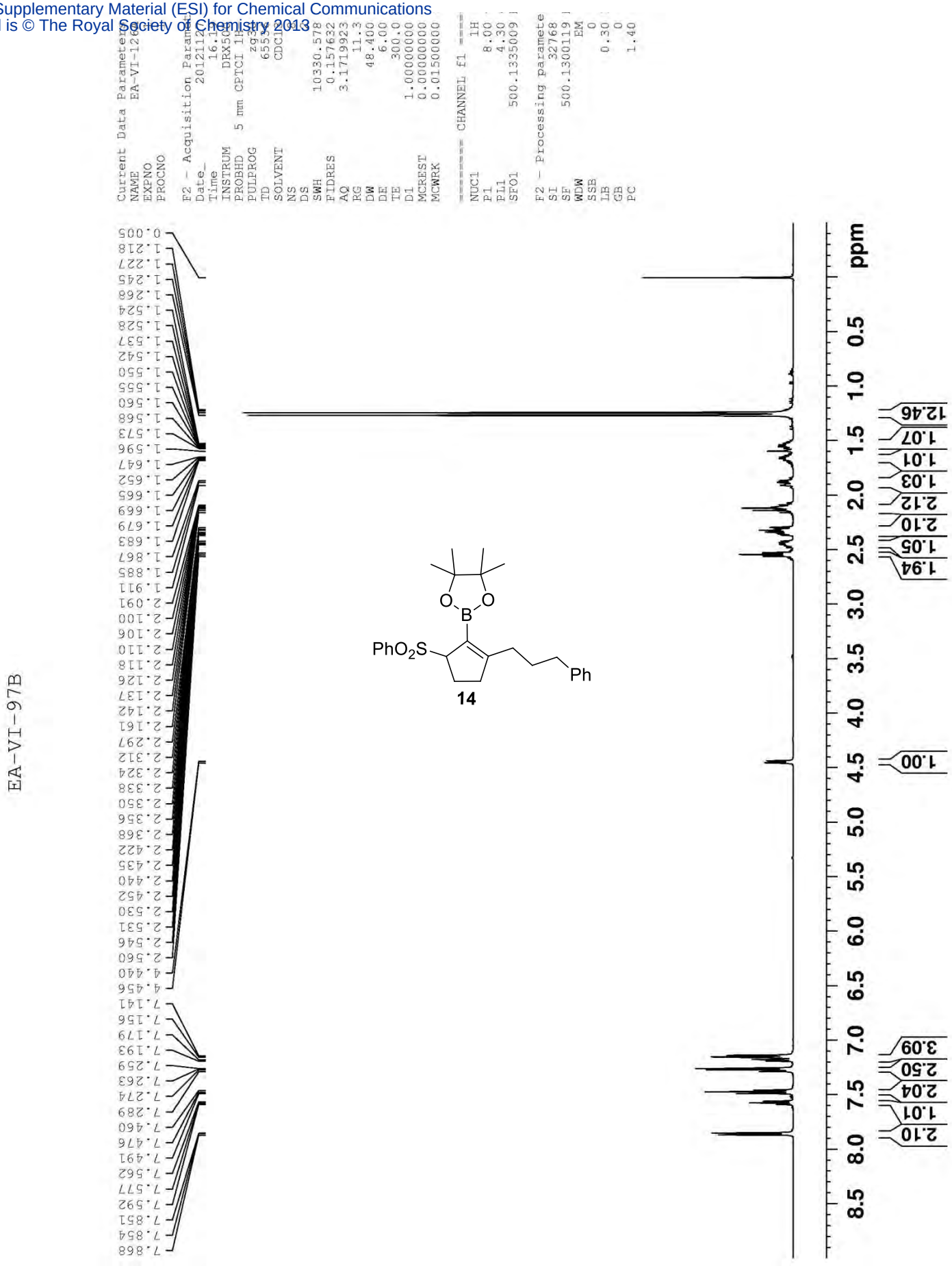

13C NMR
Current Data Parameters
NAME EA-IV-58-IB
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120105
Time 10.28
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 54
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 use
DE 35.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 use
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577727 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
24.82
24.90
26.85
33.74
74.41
76.90
77.16
77.41
83.88
128.82
129.69
133.46
137.67
154.77
O
B
O
PhO2S
11
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20




13C NMR
Current Data Parameters
NAME EA-VI-103
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121031
Time 12.07
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 158
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 use
DE 35.00 use
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 12.00 use
PL1 0.30 dB
SFO1 125.7716224 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7578496 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
27.56
31.32
39.02
39.18
39.35
39.52
39.69
39.78
39.85
39.94
40.02
75.30
128.04
129.31
132.83
138.10
138.95
BF3K
PhO2S
17
ppm
0
20
40
60
80
100
120
140
160
180
200
220
240




13C NMR
Current Data Parameters
NAME EA-VI-97B
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121026
Time_ 8.32
INSTRUM DRX500
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 71424
SOLVENT CDCl3
NS 134
DS 4
SWH 35211.270 Hz
FIDRES 0.492989 Hz
AQ 1.0142708 sec
RG 4096
DW 14.200 usec
DE 35.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 0.30 dB
SFO1 125.7716224 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 5.00 dB
PL12 22.00 dB
PL13 27.90 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
0.14
27.32
31.75
71.69
76.91
77.16
77.41
123.18
128.93
129.27
130.48
133.77
133.89
134.86
137.78
138.27
147.80
148.64
PhO2S
N
16
ppm


EA-VI-97B
14
ppm