Supplementary Information

Imidazole-derived Carbenes and Their Elusive Tetraazafulvalene Dimers

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General Information:

Infra-red spectra were recorded on Perkin Elmer Spectrum One FT-IR spectrometer. Proton NMR (^{1}H) and carbon NMR (^{13}C) spectra were recorded on a Bruker DPX400 spectrometer, Bruker AV400 spectrometer or Bruker DRX500 spectrometer. The chemical shifts (□) are quoted in parts per million (ppm). Multiplicities are abbreviated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet for the ^{1}H NMR spectra. The coupling constants (*J*) are reported in Hertz (Hz

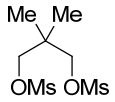
Proton NMR (^{1}H) spectra were recorded at 400.13 MHz on a Bruker® DPX 400™ spectrometer. Carbon NMR (^{13}C) spectra were recorded at 100.61 MHz, using a J-mod pulse program for resolving the carbon assignments, and standard decoupled carbon as a complement when needed. 2D experiment were recorded on a Bruker® AV500.™ The chemical shifts are quoted in parts per million (ppm), referenced to tetramethylsilane but calibrated on the solvent residual signal. Signal multiplicity is given by: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. The coupling constants are given in Hertz (Hz).

MS spectra were recorded using electron ionisation (EI), chemical ionisation (CI) or electrospray ionisation (ESI) techniques as stated for each compound. Column chromatography was performed using silica gel 60 (200-400 mesh). IR spectra were recorded on a Perkin Elmer Spectrum One FT-IR™ spectrometer, either pressed as disks in a potassium bromide matrix (KBr), or as films applied on NaCl plates or between two diamond plates (film).

All reagents were obtained from commercial suppliers. Tetrahydrofuran, dichloromethane, hexane, diethyl ether and toluene were dried and deoxygenated with a Pure-Solv 400 solvent purification system (Innovative Technology Inc., USA). Air-sensitive experiments were carried out in a glove-box (Innovative Technology Inc., USA). Dry DMF was obtained from commercial suppliers. Benzene was dried over molten potassium and under inert atmosphere.

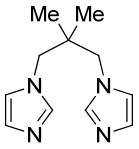
PREPARATION AND REACTIONS OF 30

Preparation of 2,2-dimethylpropane-1,3-diyl dimethanesulfonate[1]



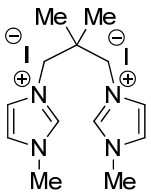
Neopentyl glycol (5.208 g, 50 mmol, 1.0 eq.) and triethylamine (13.94 mL, 100 mmol, 2.0 eq.) were dissolved in dry dichloromethane (100 mL) under an inert atmosphere and cooled with an ice-bath. Methanesulfonyl chloride (8.13 mL, 105 mmol, 2.1 eq.) was diluted in dichloromethane (40 mL) and the solution was then added dropwise by syringe to the neopentyl glycol/triethylamine mixture with stirring. When the addition was complete, the cooling bath was removed and the reaction mixture was allowed to reach room temperature and was stirred for an additional 4 h before working up with water (3 x 100 mL) and brine (100 mL). The organic layer was dried over sodium sulfate, filtered and concentrated *in vacuo* to afford 2,2-dimethylpropane-1,3-diyl dimethanesulfonate as a white solid (11.706, 44.0 mmol, 90%) mp 70-72 °C (Lit.[1] 69-71°C; ν_{max} (KBr)/cm^{-1} 2995, 2939, 1479, 1411, 1354, 1172, 981, 848, 760 and 527; Found: (ES)$^+$ (M+Na)$^+$ 283.0275. $C_7H_{16}O_6S_2Na$ (M+Na) requires 283.0285. ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 1.07 (6H, s, Me), 3.05 (6H, s, Me) and 4.02 ppm (4H, s, CH_2); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 21.5 (CH_3), 35.7 (C), 37.5 (CH_3), and 73.4 ppm (CH_2).

Preparation of 1,1'-(2,2-dimethylpropane-1,3-diyl)bis(1H-imidazole).



Sodium hydride (7.46 g, 60% in mineral oil, 186.4 mmol) was washed with dry hexane (2 x 100 mL) under argon. The residual hexane was removed. Dried dimethylformamide (40 mL) was added and the suspension was cooled in an ice-water bath. Imidazole (11.54 g, 169.5 mmol) in DMF (50mL) was added carefully. The clear solution obtained was stirred at room temp for 30 min and 2,2-dimethylpropane-1,3-diyl dimethanesulfonate (20.98 g, 80.7 mmol) in DMF (40 mL) was added. The mixture was heated at 125 °C for 24 h, cooled to room temperature, diluted with DCM (900 mL), filtered and concentrated. The product was purified by distillation (210 ˚C @ 0.01mmHg), to give the title product as yellow crystals (11.2 g, 68%). mp 80-82 °C; ν_{max} (KBr)/cm^{-1} 3124, 3104, 2970, 2936, 1666, 1636, 1508, 1232, 1110 and 1079; [Found (ES)$^+$: (M+H)$^+$ 205.1447. $C_{11}H_{17}N_4$ (M+H) requires 205.1448]; ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 0.97 (6H, s, Me), 3.82 (4H, s, CH_2), 6.86 (2H, s, ArH), 7.08 (2H, s, ArH) and 7.46 ppm (2H, s, ArH); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 23.9 (CH_3), 37.4 (C), 56.1 (CH_2), 120.9 (CH), 129.6 (CH) and 138.6 ppm (CH).

Preparation of 3,3'-(2,2-dimethylpropane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide ***30***



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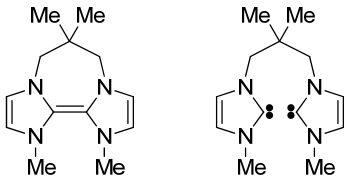
1,1'-(2,2-Dimethylpropane-1,3-diyl)bis(1H-imidazole) (10.214 g, 50.0 mmol, 1.0 eq.) was dissolved in acetonitrile (50 mL) under an inert atmosphere. Methyl iodide (3.42 mL, 105.0 mmol, 2.1 eq.) was added via a syringe and the reaction mixture was heated under reflux overnight. Dry diethyl ether was added to the cooled solution and the resultant precipitate was collected, rinsed with dry diethyl ether (3 x 100 mL) and solvent traces removed in a desiccator to afford *3,3'-(2,2-dimethylpropane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide* (22.801 g, 46.7 mmol, 93 %) (**30**) as an hygroscopic white powder; mp 200-202 °C; ν_{max} (KBr)/cm^{-1} 3077, 3056, 1578, 1562, 1474 and 1168; [Found: (HNES)$^+$: (M-I)$^+$ 361.0880. $C_{13}H_{22}IN_4$ (M-I) requires 361.0884]; ^{1}H-NMR (500 MHz, d^6-DMSO) δ = 0.96 (6H, s, CH_3), 3.94 (6H, s, CH_3), 4.25 (4H, s, CH_2), 7.79 (2H, apparent t, J 1.7 Hz, ArH), 7.82 (2H, apparent t, J 1.7 Hz, ArH) and 9.21 ppm (2H, s, ArH); ^{13}C-NMR (125 MHz, d^6-DMSO) δ = 22.9 (CH_3), 36.9 (C), 37.0 (CH_3), 56.9 (CH_2), 124.3 (CH), 123.8 (CH) and 138.4 ppm (CH).

Preparation of 1,6,6,11-tetramethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine ***31*** *and 3,3'-(2,2-dimethyl-propane-1,3-diyl)bis(1-methyl-1H-imidazol-2-ylidene)* ***32****.*

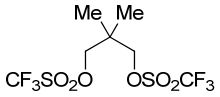


31 **32**

3,3'-(2,2-Dimethylpropane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide **30** (2.441 g, 5.0 mmol, 1.0 eq.) was weighed into a Schlenk flask (1 L) and sealed with a septum. Also in a glove-box, pre-washed sodium hydride (0.252 g, 10.5 mmol, 2.1 eq.) was weighed into a specialised dropping funnel equipped with a frit, before being sealed under inert atmosphere. The Schlenk flask and dropping funnel were removed to a fumehood, before the septum on the Schlenk flask was replaced with a dry-ice condenser under a stream of argon. Salt **25** was dissolved with stirring by the condensation of liquid ammonia (approx. 800 mL) into the Schlenk flask. Still under a stream of argon the dropping funnel was placed between the Schlenk flask and condenser. Liquid ammonia (100 mL) was condensed into the dropping funnel, where it dissolved some of the sodium hydride; the sodium hydride solution was added to the Schlenk flask; this was then repeated a further time. Over a 3 h period liquid ammonia was allowed to repeatedly reflux onto to the dry-ice condenser and collect in the dropping funnel to dissolve some sodium hydride before being re-added to the reaction mixture, ensuring that the majority of sodium hydride was transferred. The dropping funnel was then removed and the reaction was allowed to reflux. A colour change in the reaction mixture was observed during the course of the reaction, from colourless to a clear green and eventually a vibrant clear yellow. After 48 h the liquid ammonia was allowed to completely evaporate. At room temperature and under an atmosphere of argon, the Schlenk flask was sealed with a septum and transferred to a glove-box. The pink crystalline residue was extracted with diethyl ether (4 x 500 mL). The clear green-yellow diethyl ether extracts were concentrated to afford a yellow solid mixture of **31** and **32** in a ratio of 55:45.
1,6,6,11-tetramethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine **31**: ^{1}H-NMR (500 MHz, C_6D_6) δ = 0.79 (6H, s, CH_3), 2.32 (4H, s, CH_2), 2.59 (6H, s, CH_3), 5.41 (2H, d, J 2.4 Hz, ArH) and 5.48 ppm (2H, d, J 2.4 Hz, ArH); ^{13}C-NMR (125 MHz, C_6D_6) δ = 24.6 (CH_3), 35.5(C), 37.3 (CH_3), 57.7 (CH_2), 120.7 (CH), 123.1 (CH) and 127.3 ppm (C) and *3,3'-(2,2-dimethylpropane-1,3-diyl)bis(1-methyl-1H-imidazol-2-ylidene)* **32**; ^{1}H-NMR (500 MHz, C_6D_6) δ = 0.86 (6H, s, CH_3), 3.39 (6H, s, CH_2), 3.91 (4H, s, CH_3), 6.32 (2H, d, J 1.4 Hz, ArH) and 7.36 ppm (2H, d, J 1.4 Hz, ArH); ^{13}C-NMR (125 MHz, C_6D_6) δ = 25.3 (CH_3), 37.6 (C), 38.1 (CH_3), 64.7 (CH_2), 119.2 (CH), 122.8 (CH) and 216.7 (C) ppm.

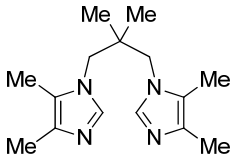
PREPARATION AND REACTIONS OF DISALT 33

Preparation of 2,2-dimethylpropane-1,3-diyl bis(trifluoromethanesulfonate)



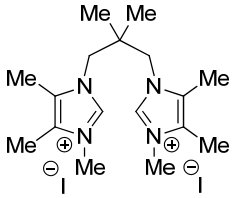
Trifluoromethanesulfonic anhydride (17.77 mL, 105 mmol, 2.1 eq.), dissolved in dichloromethane (80 mL) at -78°C, was added dropwise to a solution of neopentyl glycol (5.208 g, 50 mmol, 1.0 eq.) and pyridine (8.09 mL, 100 mmol, 2.0 eq.) in dry dichloromethane (100mL) under inert atmosphere at -78 °C with vigorous stirring. Then the cooling bath was removed and the reaction mixture was allowed to reach room temperature and to stir for an additional 30 min before working up with water (3 x 100 mL) and brine (100 mL). The organic layer was dried over sodium sulfate, filtered, concentrated and purified by flash filtration through silica. Elution with dichloromethane afforded 2,2-dimethylpropane-1,3-diyl bis(trifluoromethanesulfonate) (17.380 g, 47.2 mmol, 94 %) as a pink oil. ν_{max} (thin film)/cm^{-1} 2981, 1480, 1417, 1248, 1210, 1146, 946, 850 and 615; ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 1.14 [6H, s, $C(CH_3)_2$] and 4.32 ppm (4H, s, CH_2); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 20.8 (CH_3), 36.5 (C), 79.25 (CH_2) and 119.0 ppm (q, J = 319.5 Hz, CF_3).

Preparation of 1,1'-(2,2-dimethylpropane-1,3-diyl)bis(4,5-dimethyl-1H-imidazole)



4,5-Dimethyl-1H-imidazole[2] (1.971 g, 20.5 mmol, 2.05 eq.) was dissolved in anhydrous DMF (25 mL) within a glove box. Pre-washed sodium hydride (0.492g, 20.5 mmol, 2.05 eq.) was added portionwise to the vigorously stirred reaction mixture, allowing effervescence to subside between additions. On complete addition of sodium hydride, the reaction mixture was allowed to stir for an additional 10 min before 2,2-dimethylpropane-1,3-diyl bis(trifluoromethanesulfonate) (3.683 g, 10.0 mmol, 1.0 eq.) was added dropwise, at such a rate that minimised the exotherm. The round-bottomed flask was equipped with a condenser and heated at 105 °C overnight. The cooled reaction mixture was concentrated *in vacuo* before diluting in ethyl acetate (200 mL) and washing with 2M NaOH (3 x 75 mL), water (2 x 75 mL) and brine (50 mL). The organic layer was dried over sodium sulfate, filtered and concentrated to afford *1,1'-(2,2-dimethylpropane-1,3-diyl)bis(4,5-dimethyl-1H-imidazole)* (2.110 g, 8.1 mmol, 81%) as a viscous orange oil. ν_{max} (KBr)/cm^{-1} 2970, 2924, 1639, 1502, 1449, 1234 and 1176; [Found (HNES)$^{+}$: $(M+H)^{+}$ 261.2077. $C_{15}H_{25}N_4$ (M+H) requires 261.2074]; ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 0.67 (6H, s, CH_3), 1.81 (6H, s, CH_3), 1.87 (6H, s, CH_3), 3.44 (4H, s, CH_2) and 7.03 ppm (2H, s, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 8.7 (CH_3), 12.6 (CH_3), 23.3 (CH_3), 37.9 (C), 52.6 (CH_2), 122.3 (C), 133.3 (C) and 135.6 ppm (CH).

Preparation of *3,3'-(2,2-dimethylpropane-1,3-diyl)bis(1,4,5-trimethyl-1H-imidazol-3-ium) iodide* **33**.



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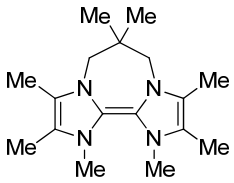
1,1'-(2,2-Dimethylpropane-1,3-diyl)bis(4,5-dimethyl-1H-imidazole) (2.08 g, 8.0 mmol, 1.0 eq.) was dissolved in acetonitrile (20 mL) in a flame-dried, round-bottomed flask equipped with a stirrer bar and condenser, which was then sealed with a septum under an inert atmosphere. Methyl iodide (1.05 mL, 16.8 mmol, 2.1 eq.) was added via a syringe and the reaction mixture was refluxed overnight. Dry diethyl ether was added to the cooled solution and the resultant precipitate was collected, rinsed with dry diethyl ether (3 x 50 mL) and solvent traces were removed in a desiccator to afford *3,3'-(2,2-dimethylpropane-1,3-diyl)bis(1,4,5-trimethyl-1H-imidazol-3-ium) iodide* **33** (4.09 g, 7.52 mmol, 94%) as a hygroscopic white powder; mp: 198-200 °C; ν_{max} (KBr)/cm^{-1} 3459, 3415, 3057, 2968, 2870, 1635, 1564, 1446 and 1202; [Found: (FTMS^{+}) (M-I)$^{+}$ 417.1504 $C_{17}H_{30}IN_4$ (M-I) requires 417.1510]; ^{1}H-NMR (500 MHz, d^{6}-DMSO) δ = 0.94 (6H, s, CH_3), 2.28 (6H, s, CH_3), 2.31 (6H, s, CH_3), 3.80 (6H, s, NCH_3), 4.18 (4H, s, CH_2) and 9.00 ppm (2H, s, ArH); ^{13}C-NMR (125 MHz, d^{6}-DMSO) δ = 8.84 (CH_3), 9.7 (CH_3), 23.1 (CH_3), 34.4 (CH_3), 38.3 (C), 53.9 (CH_2), 128.0 (C), 128.1 (C) and 136.7 ppm (CH).

Preparation of 1,2,3,6,6,9,10,11-octamethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]di-azepine ***34***



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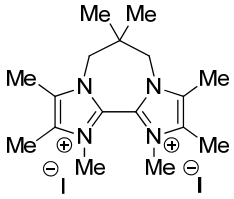
3,3'-(2,2-Dimethylpropane-1,3-diyl)bis(1,4,5-trimethyl-1H-imidazol-3-ium) iodide **33** (2.721 g, 5.0 mmol, 1.0 eq.) and pre-washed sodium hydride (0.600 g, 25.0 mmol, 5.0 eq.) were added to a Schlenk flask (500 mL) which was assembled with a dry-ice condenser under a stream of argon. Liquid ammonia (approx. 500 mL) was condensed onto the reactants forming a vivid yellow solution. The reaction was refluxed for 4 h before the liquid ammonia was allowed to evaporate overnight. At room temperature, the Schlenk flask was sealed under an atmosphere of argon with a septum and transferred to a glove-box. The solid vivid yellow product was extracted several times with diethyl ether, until extracts were colourless. The combined organic extracts were concentrated under vacuum to afford *1,2,3,6,6,9,10,11-octamethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine* **34** as a deep-yellow solid (1.39 g, 4.8 mmol, 96%); ^{1}H-NMR (500 MHz, C_6D_6) δ = 0.92 (6H, s, CH_3), 1.56 (6H, 2 x s, CH_3), 1.63 (6H, 2 x s, CH_3), 2.61 (4H, s, CH_2) and 2.78 ppm (6H, s, CH_3); ^{13}C-NMR (125 MHz, C_6D_6) δ = 10.5 (CH_3), 10.6 (CH_3), 25.4 (CH_3), 33.9 (CH_3), 35.0 (C), 60.7 (CH_2), 121.7 (C), 122.5 (C) and 127.8 ppm (C).

Preparation of 1,2,3,6,6,9,10,11-octamethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine-4,8-diium iodide ***37***



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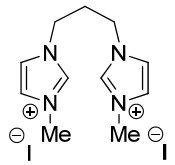
1,2,3,6,6,9,10,11-Octamethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine **34** (0.288 g, 1.0 mmol, 1.0 eq.) was dissolved in diethyl ether (10 mL). Iodine (0.305 g, 1.2 mmol, 1.2 eq.) was added to the vivid, transparent yellow solution causing precipitation, as well as a colour change to a transparent brown. The suspension was filtered; the brown precipitate was collected and washed with diethyl ether to afford *1,2,3,6,6,9,10,11-octamethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine-4,8-diium iodide* **37** (0.537 g, 0.99 mmol, 99 %) as a brown crystalline solid mp 180-182 °C. ν_{max} (KBr)/cm^{-1} 2957, 1612, 1450 and 1414; [Found (HNES)$^+$: (M-I)$^+$ 415.1354. $C_{17}H_{28}IN_4$ (M-I) requires 415.1353]; ^{1}H-NMR (500 MHz, CD_3CN) δ = 1.22 (6H, s, CH_3), 2.42 (6H, s, CH_3), 2.43 (6H, s, CH_3), 3.75 (2H, d, *J* 15.0 Hz, CH_2), 3.83 (6H, s, NCH_3) and 4.20 ppm (2H, d, *J* 15.0 Hz, CH_2); ^{13}C-NMR (125 MHz, CD_3CN) δ = 9.6 (CH_3), 9.8 (CH_3), 24.2 (CH_3), 36.1 (CH_3), 45.4 (C), 54.4 (CH_2), 126.9 (C), 132.7 (C) and 133.8 ppm (C).

PREPARATION AND REACTIONS OF 18.

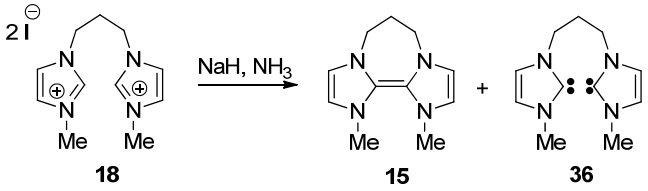
Preparation of 3,3'-(propane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide ***18***[3]



18

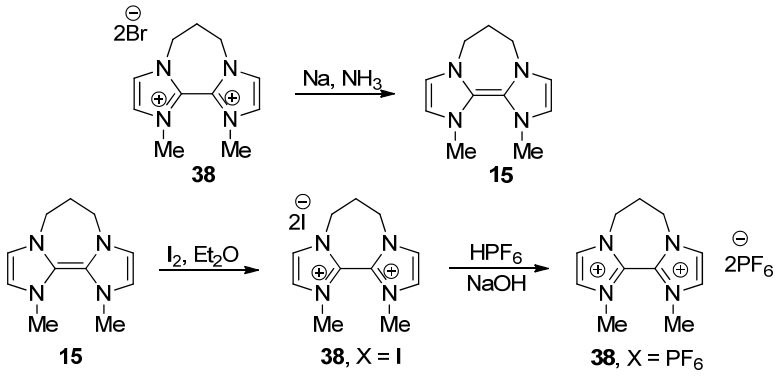
1-Methyl-1H-imidazole (3.42 mL, 50 mmol, 2.5 eq.) was dissolved in acetonitrile (20mL) under an inert atmosphere. 1,3-Diiodopropane (2.3 mL, 20 mmol, 1.0 eq.) was added via syringe and the reaction mixture was refluxed overnight. Dry diethyl ether was added to the cooled solution and the resultant precipitate was collected and rinsed with dry diethyl ether (3 x 100 mL). The white crystalline residue was placed in a desiccator to remove solvent traces, affording the pure hygroscopic 3,3'-(propane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide **18** (8.460 g, 18.4 mmol, 92 %). mp: 143-145 °C (lit.[3] 137 °C); ν_{max} (KBr)/cm^{-1}; 3075, 3020, 2988, 2834, 1568, 1557 and 1451; [Found: (HNES$^+$) (M-I)$^+$ 333.0571 $C_{11}H_{18}IN_4$ (M-I) requires 333.0571]; ^{1}H-NMR (500 MHz, d^6-DMSO) δ = 2.42 (2H, quin, *J* 6.7 Hz, CH_2), 3.90 (6H, s, CH_3), 4.26 (4H, t, *J* 6.7 Hz, CH_2), 7.78-7.80 (4H, m, ArH) and 9.14 ppm (2H, s, ArH); ^{13}C-NMR (125 MHz, d^6-DMSO) δ = 30.4 (CH_2), 36.8 (CH_2 or CH_3), 46.6 (CH_3 or CH_2), 123.1 (CH), 124.7 (CH) and 137.7 ppm (CH).

A deprotonation approach to the preparation of 1,11-dimethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine ***15***



In a glove-box 3,3'-(propane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide **18** (0.920 g, 2.0 mmol, 1.0 eq.) and pre-washed sodium hydride (0.480 g, 20.0 mmol, 10 eq.) were weighed into a Schlenk flask (1 L) and sealed with a septum, before removing to a fumehood. The Septum was replaced with a dry-ice condenser under a stream of argon. Liquid ammonia (1 L) was condensed into the Schlenk flask, the reaction was then refluxed for 24 h with the aid of cryocooler before the liquid ammonia was allowed to evaporate. The Schlenk flask containing the dry grey solid with a yellow/green hue was sealed with a septum under argon and transferred back into a glovebox. The solid (< 50 mg) was extracted with diethyl ether (4 x 250 mL), evaporation of the organic layer under reduced pressure afforded a yellow/orange residue that contained a number of components. ^{1}H NMR (C_6D_6) showed doublets at δ 5.3-5.5 suggestive of a tetraaza-alkene and a pair of doublets about δ = 6.5 suggestive of an imidazolylidene, but the very small amount led us to investigate formation of **15** by Birch reduction (see below).

Preparation of 1,11-dimethyl-5,6,7,11-tetrahydro-1H-diimidazo[1,2-a:2',1'-c][1,4]diazepine ***15*** *by Birch reduction.*



An air condenser was assembled on top of a Schlenk flask containing dibromide **38** (X = Br)[4] (500 mg, 1.37 mmol) in a glovebox. The flask was moved out of the glovebox and linked to an argon line, an ammonia line and a vacuum line with a three-way tap. The gas lines were put under vacuum and refilled with argon. The operation was repeated five times to make sure there was no oxygen in the line. Then a gentle flow of argon was passed through the system with a bubbler at the end. The Schlenk flask was cooled in a dry ice-acetone bath and ammonia gas was introduced, with liquid ammonia (~15 mL) being condensed into the flask (The ammonia had been dried under argon with sodium in another Schlenk flask and distilled at room temperature). The ammonia gas flow was stopped and the mixture was stirred in the cooling bath with a gentle argon flow passing through. Sodium (154 mg, 6.70 mmol, freshly cut in the glovebox) was added quickly from the top of the air condenser. The cooling bath was removed, the argon flow was stopped and the deep blue mixture was stirred at room temperature while the ammonia slowly evaporated. When the ammonia evaporation was finished, the reaction flask was moved into a glovebox and the mixture was extracted with ether (that had been freshly dried in the glovebox with sodium and benzophenone) [or benzene C_6D_6 (freshly dried by refluxing with molten potassium metal in glovebox for 6 h)]. The solvent was removed affording **15** as a yellow solid (228 mg,

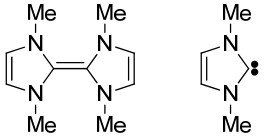
81%). NMR spectra were taken with specially dried C_6D_6 (freshly dried by refluxing with molten potassium metal in glovebox for 6 h). ^{1}HNMR (C_6D_6, 400MHz) δ = 1.31-1.37 (2H, m), 2.44-2.47 (4H, m), 2.56 (6H, s), 5.43 (2H, d, *J* 2.4 Hz) and 5.54 ppm (2H, d, *J* 2.4 Hz), ^{13}CNMR (C_6D_6, 100MHz) δ = 29.8, 35.7, 52.3, 119.8, 121.3 and 125.9 ppm.

Preparation of 1,11-dimethyl-6,7-dihydro-5H-diimidazo[1,2-a:2',1'-c][1,4]diazepine-1,11-diium hexafluorophosphate **38** (X = PF_6)

The above reaction was repeated and the neutral dimer **15** was extracted inside a glovebox with freshly dried benzene (30 mL) (dried by refluxing with potassium metal for 5 h inside a glovebox followed by distillation). The mixture was filtered and to the solution was added solution of I_2 (1.05g, 4.11 mmol) in dry benzene (20 mL). The mixture was stirred for 10 min and was transferred out of the glovebox. Water (40 mL) was added and the mixture was stirred vigorously at room temperature for 2 h. The water layer was separated and HPF_6 (3 mL, 60%) was added. The suspension was stirred at room temperature and then neutralized with NaOH. The solid was filtered, washed with water and dried under vacuum affording *1,11-dimethyl-6,7-dihydro-5H-diimidazo[1,2-a:2',1'-c][1,4]diazepine-1,11-diium hexafluorophosphate* **38** (X = PF_6) as a white solid, (312 mg, 46 %). m.p. 259 ºC (dec.). ν_{max} (KBr)/cm^{-1} 3161, 1560, 1544, 1508, 1459, 1384, 1242, 1180, 839. [Found: (ESI) $(M\text{-}PF_6)^+$ 349.1011, $C_{11}H_{16}F_6N_4P$ (M-PF_6) requires 349.1011; Found $(M\text{-}2PF_6\text{-}H)^+$ 203.1290, $C_{11}H_{15}N_4$ (M-2PF_6-H) requires 203.1291]; ^{1}H-NMR (DMSO-d^6, 400 MHz) δ = 2.54-2.62 (2H, m), 4.05 (6H, s), 4.25 (2H, ddd, *J* 14.8, 9.6, 9.6 Hz), 4.69 (2H, ddd, *J* 14.8, 4.3, 4.3 Hz), 8.17 (2H, d, *J* 1.8 Hz) and 8.22 ppm (2H, d, *J* 1.8 Hz); ^{13}C-NMR (DMSO-d^6, 100 MHz) δ = 29.7, 37.5, 45.4, 126.2, 127.1 and 128.0 ppm.

PREPARATION AND REACTIONS OF 3, R = Me

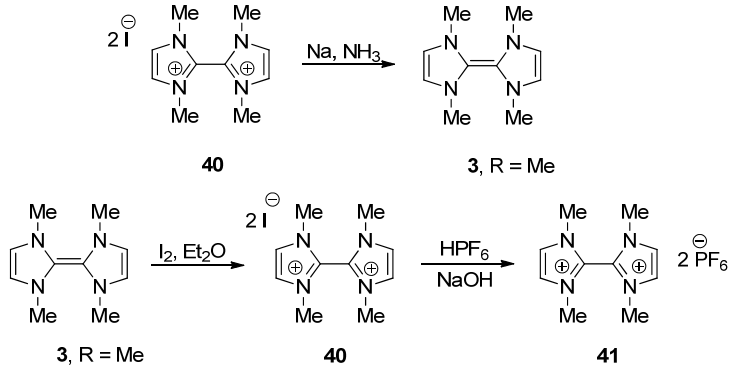
*A deprotonation approach to the preparation of 1,1',3,3'-tetramethyl-1,1',3,3'-tetrahydro-2,2'-biimidazolylidene **3** (R = Me) and 1,3-dimethyl-1H-imidazol-2-ylidene **39**.*



3 (R = Me) **39**

Within a glove-box, 1,3-dimethyl-1H-imidazol-3-ium iodide[3] **39** (0.672 g, 3.0 mmol, 1.5 eq.) and pre-washed sodium hydride (0.048 g, 2.0 mmol, 1.0 eq.) were weighed into a Schlenk flask (100 mL) that was then equipped with a dry-ice condenser under a stream of argon. The minimum volume of liquid ammonia required to fully dissolve the reactants was condensed into the Schlenk flask. Initially a pink coloration of the reaction mixture was observed and this gradually changed to yellow. The liquid ammonia was refluxed for 1 h and then allowed to evaporate overnight. At room temperature, the viscous yellow semi-solid was triturated and diethyl ether (50 mL) and the diethyl ether took on a yellow colour. This diethyl ether extract was concentrated to a pink residue (mass estimated at less than 50 mg) which was examined by NMR. A number of components were present but the presence of a singlet at δ = 5.3 ppm was suggestive of a tetraaza-alkene while a singlet at δ 6.2 was suggestive of an imidazolylidene. Because of the success in preparing **15** by Birch reduction, this route was now explored (see below).

*Preparation of **3** (R = Me) by Birch reduction.*



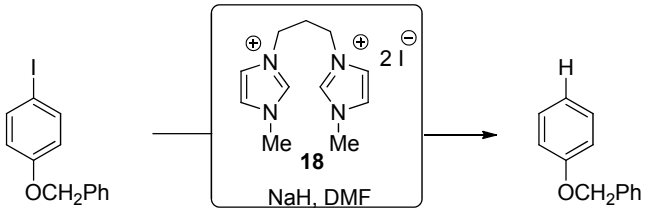
An air condenser was assembled on top of a Schlenk flask containing diiodide **40**[5] (500 mg, 1.12 mmol) in a glovebox. The flask was sealed, moved out of the glovebox, and linked to an argon line, an ammonia line and a vacuum line with a three-way tap. The gas lines were put under vacuum and refilled with argon. The operation was repeated five times to make sure there was no oxygen in the line. Then a gentle flow of argon was passed through the system with a bubbler attached to the end. The Schlenk flask was cooled in a dry ice-acetone bath, and ammonia gas was introduced with liquid ammonia (~15 mL) condensed in the flask (the ammonia had been dried in another Schlenk flask containing sodium and under argon and distilled at room temperature). The ammonia gas flow was stopped and the mixture was stirred in the cooling bath with a gentle argon flow passing through. Sodium (136 mg, 5.91 mmol, freshly cut in the glovebox) was added quickly from the top of the air condenser. The cooling bath was removed, the argon flow was stopped and the deep blue mixture was stirred at room temperature while the ammonia slowly evaporated. When the ammonia evaporation had finished, the reaction flask was moved into a glovebox and the mixture was extracted with diethyl ether (freshly dried in a glovebox with sodium and benzophenone). The ether was removed and a uniform yellow solid was obtained (157 mg, 73 %). The solid turned into an oil in a few minutes. To characterise the product, the reaction procedure was repeated and the reaction flask was transferred into a glovebox, where specially dried C_6D_6 (dried by refluxing with molten potassium metal in a glovebox for 5 h) was added into the reaction mixture, the clear yellow C_6D_6 solution was transferred into an NMR tube by pipette, and sealed with parafilm. The major component of this mixture as assigned as **3** (R = Me). ^{1}HNMR (C_6D_6, 400MHz) δ = 2.53 (12H, s), 5.45 ppm (4H, s). ^{13}C-NMR (C_6D_6, 100MHz) δ = 35.7, 121.7, 126.2 ppm. The ^{1}H NMR spectrum also contained signals at δ = 3.39 and 6.27 ppm ascribed to a trace of carbene **39**.

*Preparation of 1,1',3,3'-tetramethyl-1H,1'H-[2,2'-biimidazole]-3,3'-diium hexafluorophosphate **41**.*

The above reaction procedure was repeated and the neutral dimer was extracted inside a glovebox with benzene (30 mL, freshly dried by refluxing with molten potassium metal for 5 h in a glovebox and distilled). The clear solution was decanted into a flask and a solution of I_2 (1.05 g, 4.11 mmol) in dry benzene (20 mL) was added. The mixture was stirred for 10 min and was transferred out of the glovebox. Water (40mL) was added and the mixture was stirred vigorously at room temperature for 2 h. The water layer was separated and HPF_6 (3mL, 60%) was added. The suspension was stirred at room temperature and neutralized with NaOH. The solid was filtered, washed with water and dried under vacuum, affording *1,1',3,3'-tetramethyl-1H,1'H-[2,2'-biimidazole]-3,3'-diium hexafluorophosphate* **41** as a white solid, (167 mg, 31%), m.p. > 300ºC (dec.), ν_{max} (KBr)/cm^{-1} 3162, 1567, 1540, 1520, 1442, 1386, 1240, 832. [Found: (FTMS) $(M\text{-}PF_6)^+$ 337.1011, $C_{10}H_{16}F_6N_4P$ $(M\text{-}PF_6)$ requires 337.1011; Found

$(M\text{-}2PF_6\text{-}H)^+$ 191.1289, $C_{10}H_{15}N_4$ requires 191.1291]; ^{1}H-NMR (DMSO-d_6, 400MHz) δ = 3.87(12H, s), 8.27 ppm (4H, s); ^{13}C-NMR (DMSO-d_6, 100MHz) δ = 36.4, 124.7, 128.0 ppm.

*Reduction of **20** by a mixture of base and salt **18**.*



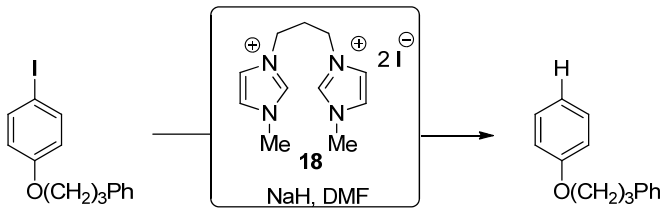
20 **22**, 79 %

Under inert atmosphere in a glove-box, 3,3'-(propane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) **18** (207 mg, 1.5 eq., 0.45 mmol) was added to a suspension of pre-washed sodium hydride (108 mg, 15.0 eq., 4.5 mmol) in anhydrous DMF (15 mL). This was stirred at room temperature for 3 h, and then 1-Iodo-4-benzyloxybenzene **20**[6] (101 mg, 1.0 eq., 0.3 mmol) was added and the reaction mixture was stirred for another 16 h. The reaction mixture was filtered, and the solid was washed with anhydrous DMF (4 mL). The filtrate was removed from the glove-box, quenched with distilled water (10 mL) and diluted with saturated brine (40 mL). The aqueous phase was extracted with diethyl ether (4 x 50 ml), the combined organic phases were washed with water (3 x 50 ml) and saturated brine (1 x 30 ml), dried over sodium sulfate, filtered and evaporated. The organic residue was purified by flash column chromatography (10% ethyl acetate in hexane) to afford (benzyloxy)benzene **22** as colourless crystals (47 mg, 0.237 mmol, 79%); m.p. 39-40 °C.(lit.[7] 39-41 °C). [Found: $(M)^+$ 184.0881. $C_{13}H_{12}O$ (M) requires 184.0883. IR (thin film) ν_{max} = 3056, 3034, 2907, 2866, 1599, 1585, 1497, 1468, 1455, 1377, 1300, 1246, 1172, 1078, 1029, 1012, 991, 916, 856, 801, 744, 696, 629, 515 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 5.08 (2H, s), 6.96-7.01 (m, 3H), 7.28-7.35 (m, 3H), 7.38-7.41 (m, 2H) and 7.44-7.46 ppm (m, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 70.3 (CH_2), 115.2 (CH), 121.3 (CH), 127.8 (CH), 128.3 (CH), 128.9 (CH), 129.8 (CH), 137.4 (C) and 159.2 ppm (C).

Preparation of 1-iodo-4-(3-phenylpropoxy) benzene ***21.***

Potassium carbonate (27.4 g, 20 mmol, 10 eq.) was added to a solution of 4-iodophenol (5.28 g, 24 mmol, 1.2 eq.) and 1-bromo-3-phenylpropane (4.0 g, 3.04 ml, 20 mmol, 1.0 eq.) in DMF (100 ml). The suspension was stirred overnight, and then the DMF was distilled under reduced pressure. The solid residue was then dissolved in water (200 ml) and extracted with diethyl ether (200 ml and 2 x 150 ml). The combined organic layers were then washed with sodium hydroxide (2M, 3 x 200 ml), water (2 x 200 ml), brine (200 ml) and dried over magnesium sulfate. The crude oil obtained after evaporation of the solvents under reduced pressure was purified by distillation in a kügelrohr (250 °C @ 0.11 mm Hg) providing 1-iodo-4-(3-phenylpropoxy) benzene)[1] **21** as a white solid (6.63 g, 98 %); m.p.: 53-55 °C; [Found: (ESI^+) $(M+NH_4)^+$ 356.0504. $C_{15}H_{20}INO$ $(M+NH_4)$ requires 356.0506]; ν_{max}(film)/cm^{-1} 3025, 2944, 2869, 1586, 1486, 1244, 1174, 1033, 818; ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 2.09-2.16 (2H, m, OCH_2CH_2), 2.82 (2H, t, *J* 7.6 Hz, $ArCH_2$), 3.94 (2H, t, *J* 6.2 Hz, OCH_2), 6.68-6.71 (2H, m, ArH), 7.22-7.24 (3H, m, ArH), 7.30-7.34 (2H, m, ArH), 7.56-7.59 (2H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 30.9 (CH_2), 32.3 (CH_2), 67.2 (CH_2), 82.8 (C), 117.2 (CH), 126.2 (CH), 128.7 (CH), 128.7 (CH), 138.4 (CH), 141.5 (C), 159.1 (C); *m/z* (EI^+) 338 [$(M)^+$, 10 %], 220 (10), 91 (100).

*Reduction of **21** by a mixture of sodium hydride and salt **18**.*

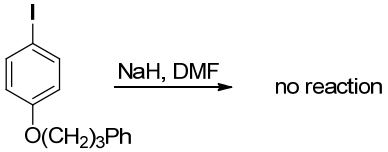


21 **23**, 79 %

Under an inert atmosphere in a glove-box, 3,3'-(propane-1,3-diyl)bis(1-methyl-1H-imidazol-3-ium) iodide **18** (207 mg, 1.5 eq., 0.45 mmol) was added to a suspension of pre-washed sodium hydride (108 mg, 15.0 eq., 4.5 mmol) in anhydrous DMF (15 mL). This was stirred at room temperature for 3 h, and then 1-iodo-4-(3-phenylpropoxy)benzene **21** (101 mg, 1.0 eq., 0.3 mmol was added to the reaction mixture and stirred for a further 16 h. The reaction mixture was filtered, and the solid washed with dry DMF (4 mL). The filtrate was removed from the glove-box, quenched with distilled water (10 mL) and diluted with saturated brine (40 mL). Following extraction with diethyl ether (4 x 50 mL), the combined organic phases were washed with water (3 x 50 mL) and saturated brine (1 x 30 mL), dried over sodium sulfate, filtered and evaporated. The organic residue was redissolved in the minimum volume of solvent (3:2 hexane/dichloromethane) and adsorbed onto a silica column packed in neat hexane, before being eluted with 20 ml portions of solvent (3:2 hexane/dichloromethane → 1:1 hexane/dichloromethane → neat dichloromethane) to afford (3-phenoxypropyl)benzene **23** (50 mg, 0.236 mmol, 79%) as a colourless oil.[8]

Found: (ESI$^+$) $(M+NH_4)^+$, 230.1538. $C_{15}H_{20}NO$ $(M+NH_4)$, requires 230.1539]; ν_{max}(film)/cm^{-1} 3062, 3027, 2946, 2870, 1600, 1497, 1245, 1038, 751; ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 2.15 (2H, m, CH_2), 2.86 (2H, t, *J* 7.5 Hz, $PhCH_2$), 4.01 (2H, t, *J* 6.3 Hz, OCH_2), 6.93-7.00 (3H, m, ArH) and 7.22-7.35 ppm (7H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 31.1 (CH_2), 32.4 (CH_2), 67.0 (CH_2), 114.8 (CH), 120.8 (CH), 126.1 (CH), 128.6 (CH), 128.7 (CH), 129.6 (CH), 141.8 (C) and 159.3 ppm (C); *m/z* (CI$^+$) 230 [$(M+NH_4)^+$, 100 %], 212 (M^+, 20 %), 118 (10), 108 (13), 91 (22).

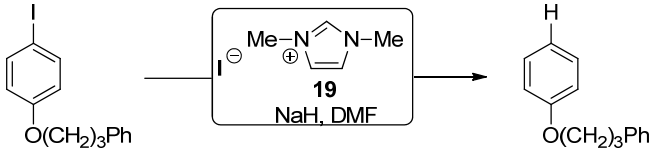
*Blank experiment in the absence of **18***



21

When the above experiment was repeated, but in the absence of salt **31**, starting material **21** (101 mg, 100 %) was recovered.

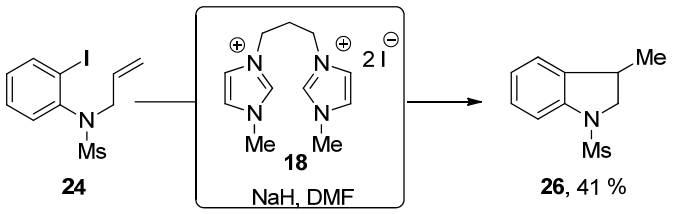
*Reduction of **21** by a mixture of sodium hydride and salt **19***



21 **23**, 61 %

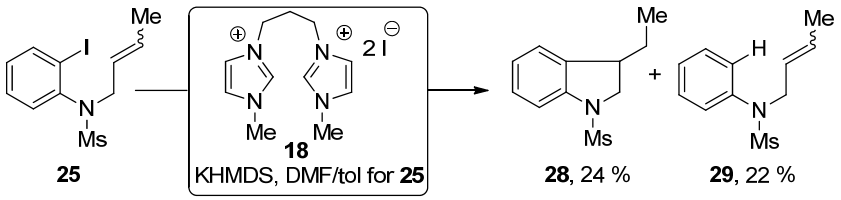
Application of the same procedure as in the previous experiment, to 1-iodo-4-(3-phenylpropoxy)benzene **21** (100 mg, 0.30 mmol, 1.0 eq.) using *N,N'*-dimethylimidazolium iodide **35** (224 mg, 1 mmol, 3.3 equiv) and pre-washed sodium hydride (24 mg, 1 mmol, 3.3 equiv) in an overnight reaction (16 h) provided (3-phenylpropoxy)benzene **23** as a colourless oil (39 mg, 61 %). Spectra of **23** were identical to those reported above.

*Reductive cyclisation of iodoarene **24***



A suspension of 60 % sodium hydride in oil (162 mg, 4.05 mmol, 8.1 equiv.) in *N,N*-dimethylformamide (50 mL) was purged with argon. 1,3-*Bis*[3-methyl-3*H*-imidazolium]propane diiodide, **18** (756 mg, 1.64 mmol, 3.3 equiv.) was added to the suspension and this was stirred for 1 h. This was then added to a purged solution of *N*-allyl-*N*-(2-iodophenyl)methanesulfonamide **24** (168 mg, 0.50 mmol, 1.0 equiv.)[9] in *N,N*-dimethylformamide (5 mL). The reaction mixture was heated at 100 °C for 18 h. The reaction mixture was poured into water (50 mL) and diethyl ether (50 mL). The aqueous phase was extracted with further diethyl ether (2 x 50 mL) and the combined organic phases were washed with water (3 x 100 mL) and then saturated brine solution (100 mL). The organic phase was dried over sodium sulfate, filtered and evaporated. The residue was purified by column chromatography (80:20 petroleum ether-ethyl acetate) to afford 1-methanesulfonyl-3-methyl-2,3-dihydro-1*H*-indole[9] **26** as a colourless oil (43 mg, 41%), (Found: (ESI) $[M+NH_4]^+$, 229.1004. $C_{10}H_{17}N_2O_2S$ $[M+NH_4]$ requires 229.1005); ν_{max} (NaCl)/cm^{-1} 2932 (C-H), 1492 (C-H), 1461 (C-H), 1344 and 1157 (SO_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 1.43 (3H, d, *J* 5.2 Hz, CH_3) 2.96 (3H, s, SO_2CH_3), 3.52-3.59 (2H, m, CH_2), 4.19-4.25 (1H, m, CH), 7.12-7.16 (1H, m, ArH), 7.27-7.35 (2H, m, ArH) and 7.45-7.52 ppm (1H, m, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 19.7 (CH_3), 26.9 (CH), 35.0 (CH_3), 55.2 (CH_2), 113.7 (CH) 124.4 (CH), 128.3 (CH), 135.7 (CH) 136.5 (C) and 143.2 ppm (C); *m/z* (EI) 211 (M^+, 25%), 199 (30), 130 (52), 86 (60) and 84 (100).

*Reductive cyclisation of iodoarene **25***



A solution of 1,3-*bis*[3-methyl-3*H*-imidazolium]propane diiodide, **18** (276 mg, 0.60 mmol, 2.0 equiv.) in *N,N*-dimethylformamide (10 mL) was purged with argon and cooled to –20 °C. A 0.5 M solution of potassium *bis*(trimethylsilyl)amide (1.8 mL, 0.89 mmol, 3.0 equiv.) in toluene, was added dropwise and the solution was stirred at this temperature for 30 min. A solution of *N*-but-2-enyl-*N*-(2-iodophenyl)methanesulfonamide **25** (105 mg, 0.3 mmol, 1.0 equiv.)[9] in *N,N*-dimethylformamide (5 mL), under argon was cooled to –20 °C and then added to the solution of the electron donor. The resulting solution was allowed to slowly warm to 0 °C and was stirred at this temperature for 4 h and was then slowly warmed to room temperature and stirred for a further 18 h. The

reaction mixture was poured into water (30 mL) and diethyl ether (30 mL). The aqueous phase was extracted with further diethyl ether (3 x 50 mL). The combined organic phases were washed with water (3 x 50 mL) and saturated brine solution (50 mL). The organic phase was dried over sodium sulfate, filtered and evaporated. The residue was purified by column chromatography (90 : 10, 80 : 20 petroleum ether - ethyl acetate) to afford 1-methanesulfonyl-3-methyl-2,3-dihydro-1*H*-indole[9] **28** as a colourless oil (16 mg, 24%).

The reaction also afforded *N-(but-2-enyl)-N-phenyl methansulfonamide* **29** as a colourless oil (15 mg, 22%).

28:[9] (Found: (ESI) $[M+NH_4]^+$, 243.1169. $C_{11}H_{19}N_2O_2S$ $[M+NH_4]$, requires 243.1167); ν_{max} (NaCl)/cm^{-1} 3016 (Ar-H), 2963 (C-H), 2930 (C-H), 1599 (Ar), 1478 (C-H), 1342 and 1161 (SO_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 1.07 (3H, t, *J* 7.3, CH_3) 1.66 (1H, m, CH_2), 1.90 (1H, m, CH_2), 2.93 (3H, s, SO_2CH_3), 3.38 (1H, m, CH), 3.69 (1H, dd, *J* 10.2, 6.4, CH_2), 4.13 (1H, dd, *J* 10.2, 9.2, CH_2), 7.11 (1H, dd, *J* 7.5, 7.5, ArH), 7.27 (2H, m, ArH) and 7.46 ppm (1H, d, *J* 7.9, ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 11.3 (CH_3), 27.5 (CH_2), 34.3 (CH_3), 41.4 (CH), 55.9 (CH_2), 113.4 (CH), 123.6 (CH), 124.7 (CH), 128.1 (CH), 135.0 (C) and 141.8 ppm (C); *m/z* (EI) 225 (M^+, 45%), 196 (50), 146 (78), 130 (79), 118 (100) and 91 (35).

29[9] (Found: (ESI) $[M+NH_4]^+$, 243.1161. $C_{11}H_{19}N_2O_2S$ $[M+NH_4]$ requires 243.1162); ν_{max} (NaCl)/cm^{-1} 3016 (C-H), 2928 (Ar-H), 2852 (Ar-H), 1598 (Ar), 1495 (C-H), 1335 and 1153 (SO_2); ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 1.56 (3H, d, *J* 6.9, CH_3), 2.91 (3H, s, SO_2CH_3), 4.22 (2H, d, *J* 6.4, CH_2), 5.45-5.53 (1H, m, =CH), 5.56-5.64 (1H, m, =CH), 7.23-7.33 (3H, m, 3 x ArH) and 7.35-7.42 ppm (2H, m, 2 x ArH); ^{13}C-NMR (100 MHz, $CDCl_3$) δ = 18.1 (CH_3), 38.7 (CH_3), 53.6 (CH_2), 125.8 (CH), 128.1 (CH), 128.8 (CH), 129.5 (CH), 129.6 (CH), 130.9 (CH) and 139.7 ppm (C); *m/z* (EI) 225 (M^+, 15%), 171 (80), 104 (60), 92 (100), 77 (80) and 55 (75).

*Determination of the molecular mass of **15** prepared by Birch reduction by DOSY NMR.*

^{1}H NMR data were acquired on a Bruker AVANCE 400 MHz NMR spectrometer operating at a magnetic field strength of 9.4 T, equipped with a 5 mm BBFO-z-atm probehead equilibrated at a constant temperature of 298 K by virtue of a BCU-05 cooling unit and operating under Topspin (version 2.1, Bruker, Karlsruhe, Germany) on a HP-XW3300 workstation within a Windows XP operating environment.

One-dimensional (1D) ^{1}H NMR data were acquired with 4 transients over a frequency width of 12 ppm (acquisition time aq = 3.42 s) and centred at 5 ppm using a single pulse-acquire pulse programme. Diffusion Ordered NMR data were acquired with 4 or 8 transients over the same observation frequency window using a double stimulated echo pulse sequence with bipolar gradients for diffusion encoding (dstebpgp3s) according to the method of Jerschow and Müller[10,11] to compensate for the effects of laminar flow caused by convection. Diffusion encoding gradients (16 values) were distributed between values of 5% and 95% of maximum according to the square of the gradient value. Diffusion coefficients were calculated directly by fitting the experimental data to the standard Stejskal-Tanner expression relating diffusion coefficient to signal intensity and diffusion encoding gradient strength. Diffusion coefficient data were determined for all reference compounds in an identical manner. Alignment of data sets was carried out by virtue of the common presence of benzene as both solvent and internal reference point for chemical shift and diffusion coefficient, *D* (average value measured over seven reference data sets $\overline{D}$ = 23.2 ± 0.08 × 10^{-10} m^2/s).

Results

Diffusion coefficient values were determined for reference compounds over the molecular weight range 78 – 1203 g/mol to establish a calibration curve against which NMR diffusion data from **15** and **13** could be assessed (**Table S1**). The calibration curve (**Fig S1**) plotted as log *D vs* log MW showed a linear fit to the expression log D = -0.559 log MW – 7.609 (r^2 = 0.981) over a molecular weight range covering two orders of magnitude (blue diamonds). Diffusion coefficients measured for the mono-bridged species **15** (MW = 204, D_{meas} = 12.9 × 10^{-10} m^2/s) and for the di-bridged species **13** (MW = 216, D_{meas} = 11.7 × 10^{-10} m^2/s) were consistent with their molecular weights. The di-bridged species, **13**, is a known compound, whereas this test was devised to see whether the mono-bridged species, **15**, was present or whether a higher molecular mass version e.g. the dimer **42** (MW = 408 g/mol, calculated diffusion coefficient, D_{calc} = 8.54 × 10^{-10} m^2/s, see **Fig S1**) or higher oligomers might be present. The diffusion coefficients measured for **15** and **13** were similar to one another and corresponded to values expected for their respective molecular weights of 204 and 216 g/mol.

Table S1: Molecular Weight (MW, g/mol) and self-diffusion coefficient (D, m^2/s) data for seven reference compounds used to establish a calibration curve and for the two keynote compounds **15** and **13.**

Compound§	MW (g/mol)	log MW	10^{-10} D (m^2/s)	log D
i	78	1.892	23.20	-8.694
ii	220	2.342	11.60	-8.936
iii	330	2.519	9.00	-9.046
iv	422	2.625	7.92	-9.102
v	426	2.629	7.87	-9.104
vi	554	2.743	7.54	-9.122
vii	1203	3.080	5.01	-9.300
15	204	2.310	12.90	-8.889
13	216	2.338	11.70	-8.931

§Reference compounds used: **i** – benzene; **ii** – 4-methyl-2,6-di-tertiarybutylphenol; **iii** – 9,10-diphenylanthracene; **iv** – squalane; **v** - 1,4-bis(diphenylphosphinyl)-butane; **vi** – 1,1'-bis(diphenylphospinyl)ferrocene; **vii** – Cyclosporine A. Diffusion NMR data were measured for **ii**-**vii** individually as solutions in C_6D_6 and for a mixture of **ii**, **iii**, **iv** and **vi** in C_6D_6 in order to compare results.

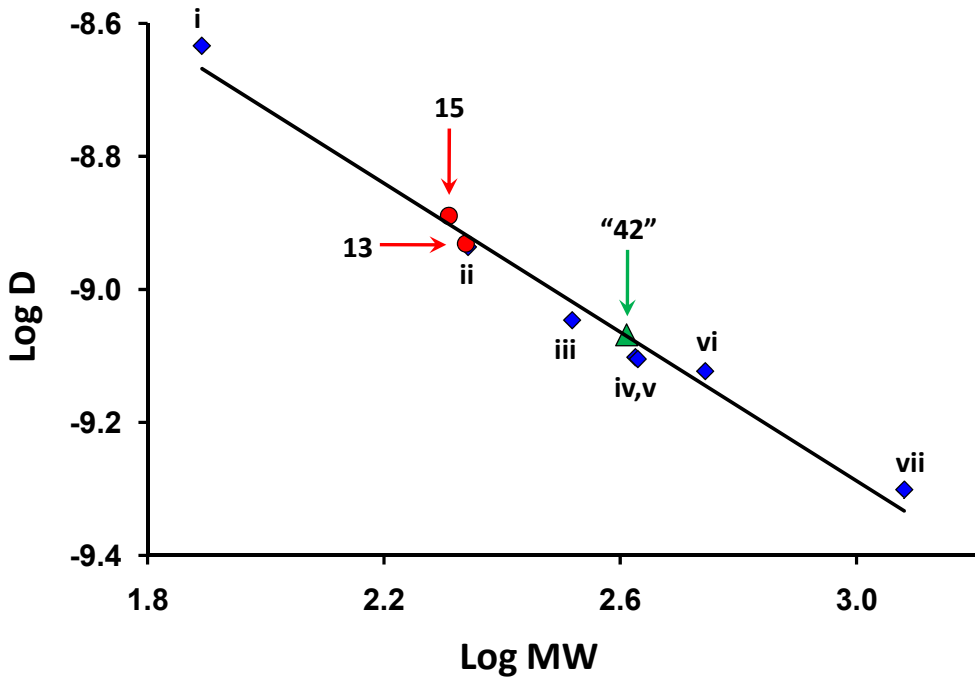


Figure S1: Plot of log *D* vs log MW for calibration and keynote synthetic compounds. Calibration curve (black line) associated with experimental data from reference compounds **i**-**vii** (blue diamonds, **Table S1**). Experimental data points for mono- and di-bridged keynote compounds, **15** and **13** respectively (red filled circles). Reference compounds used: **i** – benzene; **ii** – 4-methyl-2,6-di-tertiarybutylphenol; **iii** – 9,10-diphenylanthracene; **iv** – squalane; **v** - 1,4-bis(diphenylphosphino)-butane; **vi** – 1,1'-bis(diphenylphospino)ferrocene; **vii** – Cyclosporine A. A linear fit of the calibration data corresponds to log *D* = -0.559 log MW – 7.609 (r^2 = 0.981). Calculated log *D* value (D_{calc} = 8.54 × 10^{-10} m^2/s) for the theoretical dimer **42** based on a proposed molecule weight MW = 408 g/mol (green triangle).

Diffusion NMR spectroscopy is increasingly being used to determine Formula Weight (FW) or Molecular Weight (MW) for species that are difficult or impossible to isolate from the solution phase thereby precluding other forms of analysis capable of determining molecular weight and thus identity. The approach has been successfully applied in our laboratory in a number of recent applications: NMR measured diffusion coefficients have been related to (molecular weight)$^{0.33}$ as a way of proving the size of small biomolecules[i]; by means of log *D* vs log MW plots these type of data have been used as a way of determining the solution character of a number of so-called organometallic reagents which, whilst readily isolable in crystal form, bear little resemblance to their determined crystal structures in solution.[12-14] In the current instance, the range of molecular weights selected for calibration purposes varied from MW = 78 at the lower end to MW = 1203 at the upper end. It was particularly important to use consistent solvent conditions in order to eliminate the effects on diffusion coefficient that could be caused by differences in viscosity, hydrophobicity, hydrogen bonding and other factors that might contribute to a variable and inconsistent baseline for data calibration purposes. The choice of benzene as solvent was ideal and allowed the peptide cyclosporine A to be used as an upper molecular weight marker. On a scale of distributed molecular weights the other reference molecules were chosen to have a reasonable distribution of values across the range between those of benzene and cyclosporine A.

Use of the log *D* vs log MW approach in this instance proves to be robust and provides a linear calibration curve against which data from the keynote compounds could be assessed. Whilst the ^{1}H NMR spectrum and measured diffusion

coefficient of the di-bridged compound, **13**, were consistent with what could only be a species with MW = 216 g/mol, the ^{1}H NMR spectrum of the "mono-bridged" species was consistent with both monomer (**15**) or dimer (**42**) forms. A solution phase NMR-based diffusion method provides the only reliable method for distinguishing the two. The measured diffusion coefficient determined for **15** was very similar to that for **13**, indicating that their masses were very similar and confirming successful formation of **15** for the very first time.

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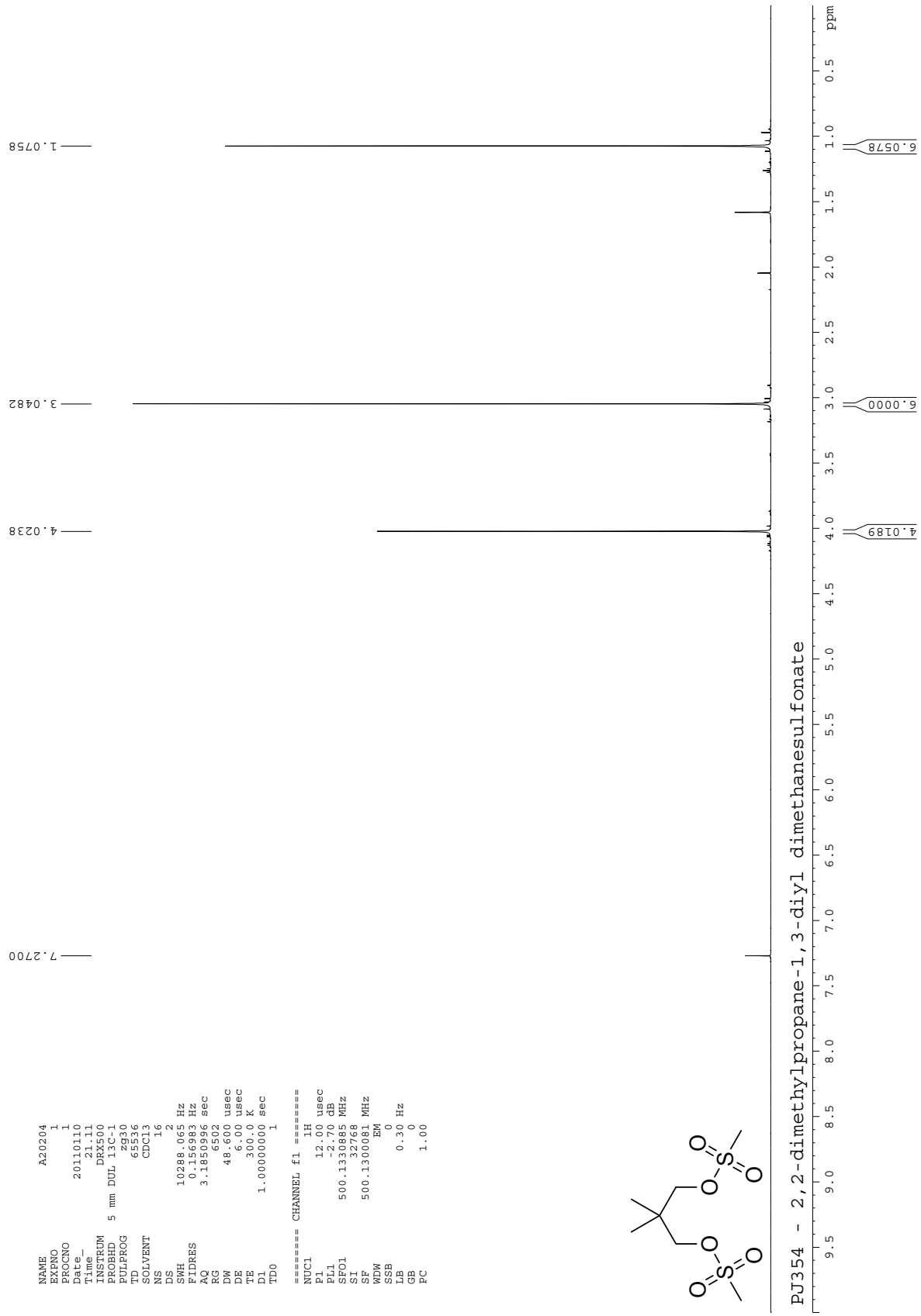
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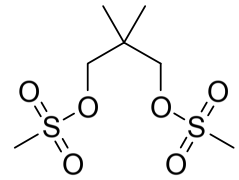

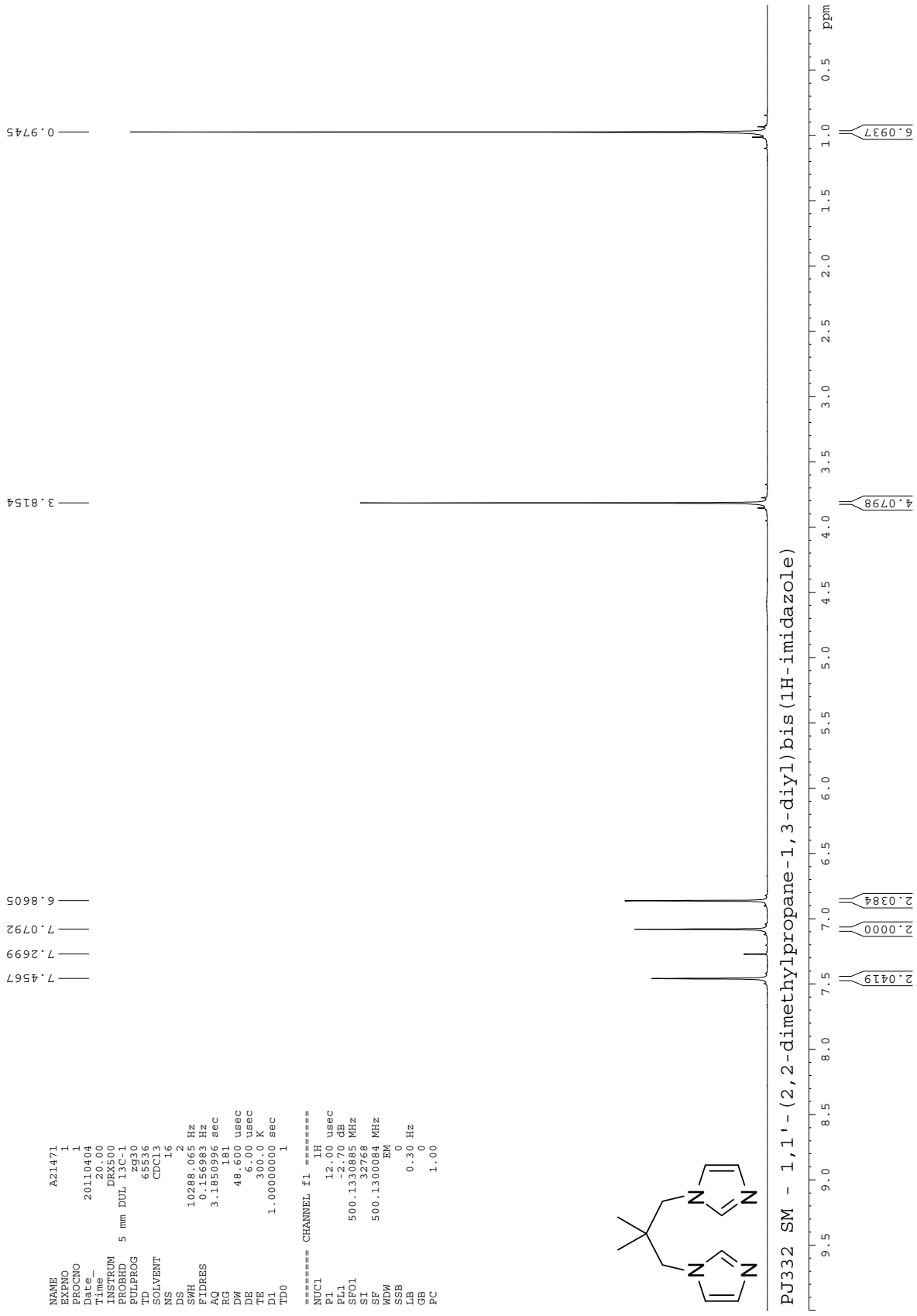
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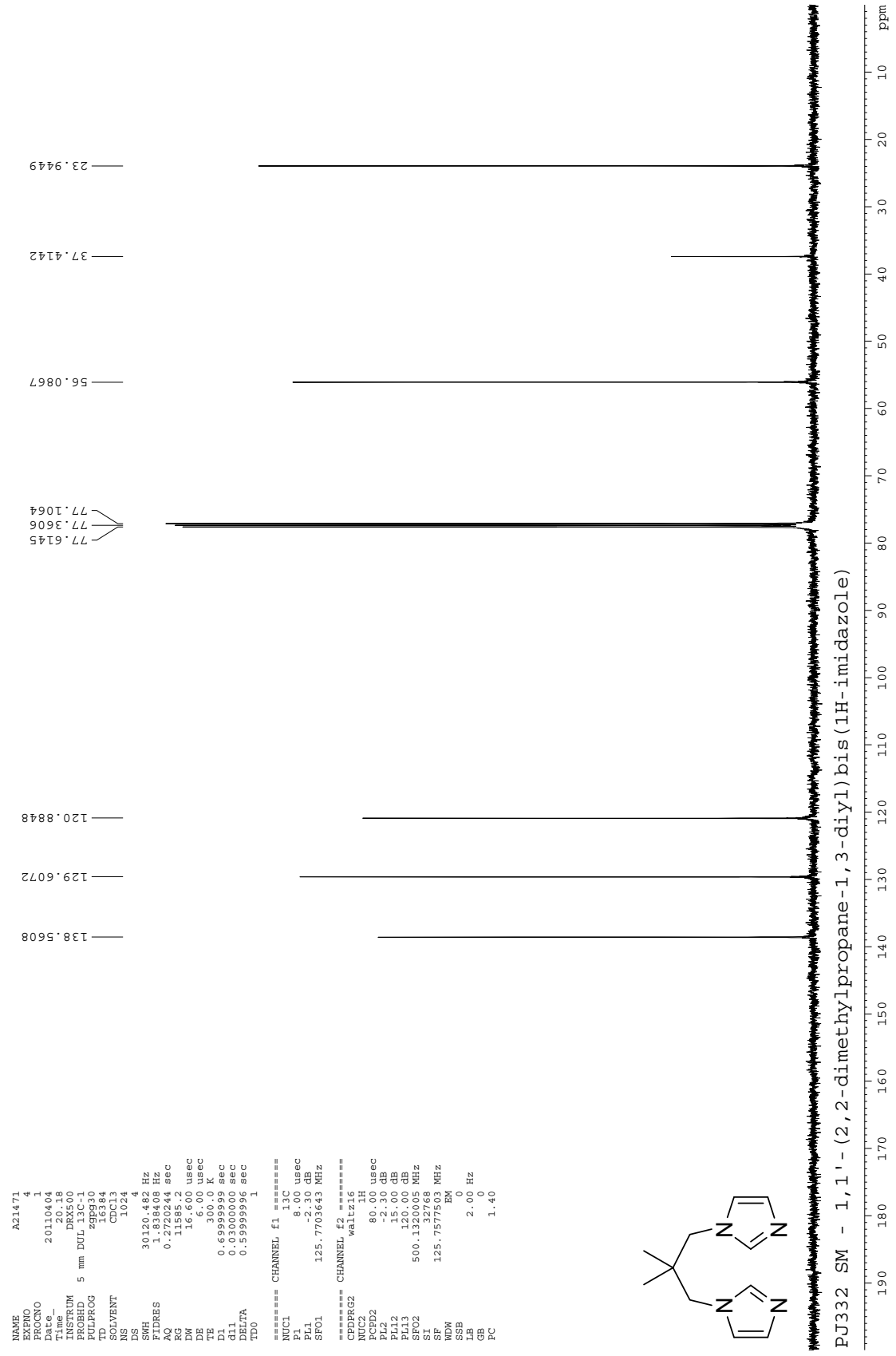
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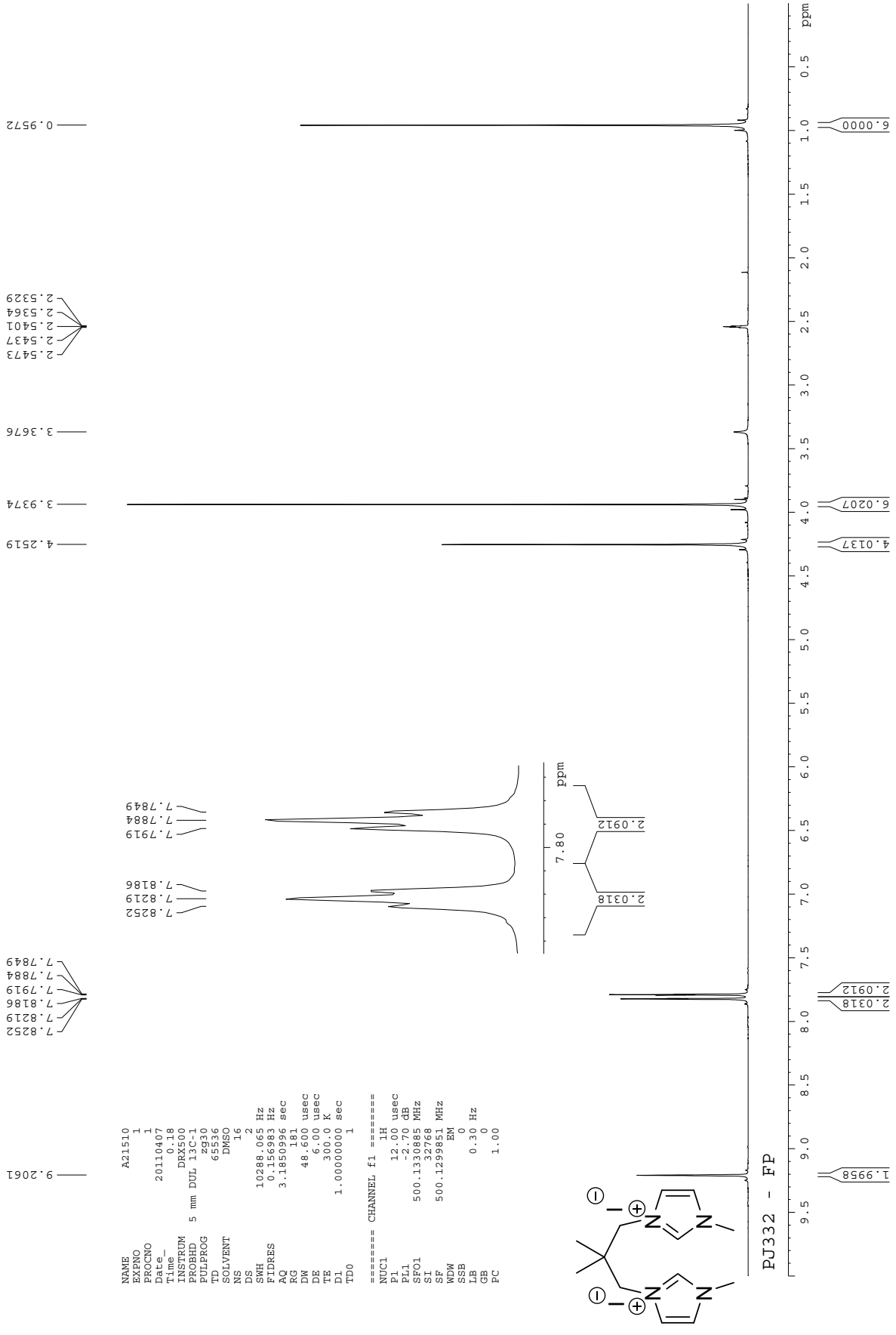

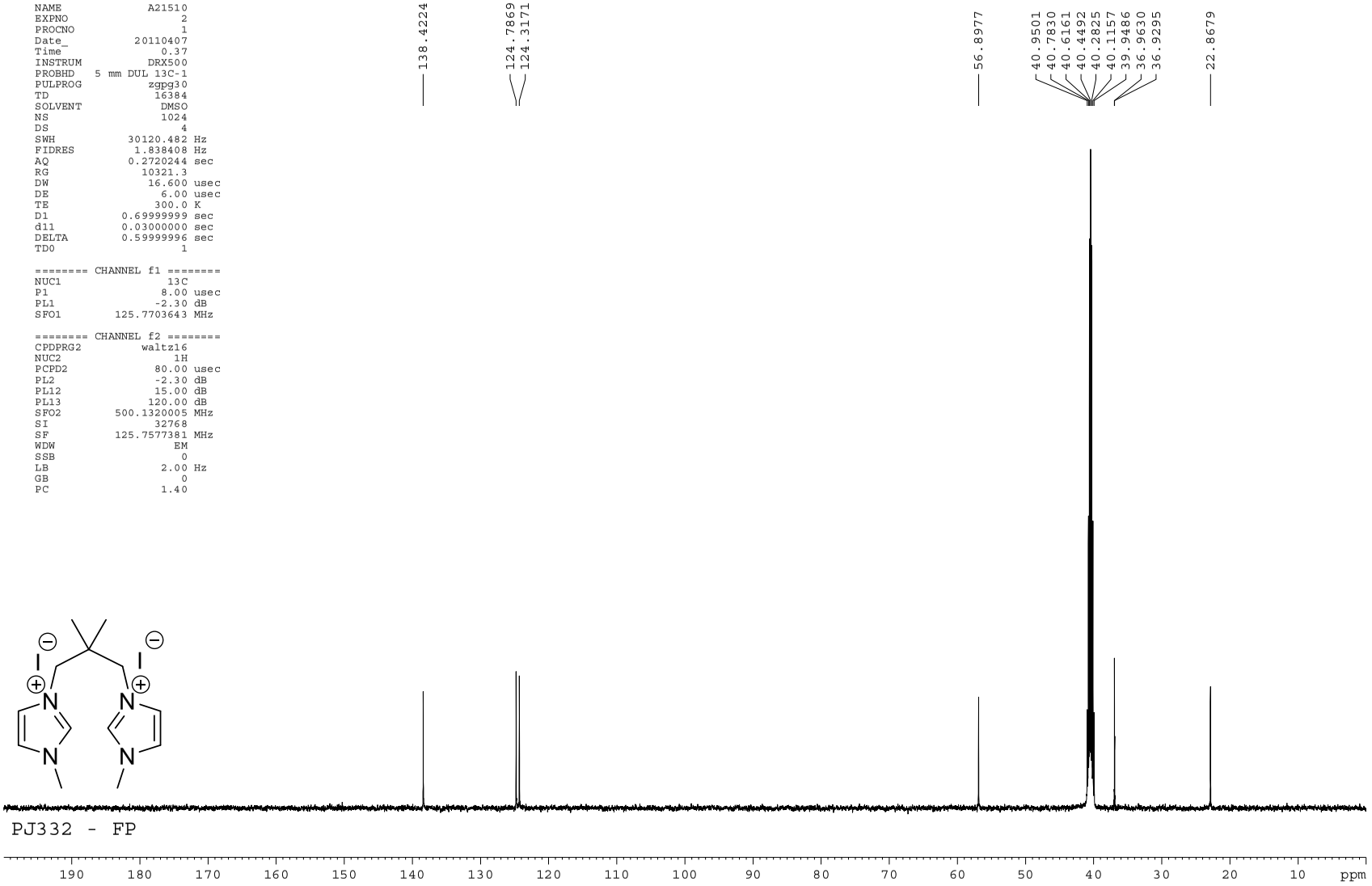

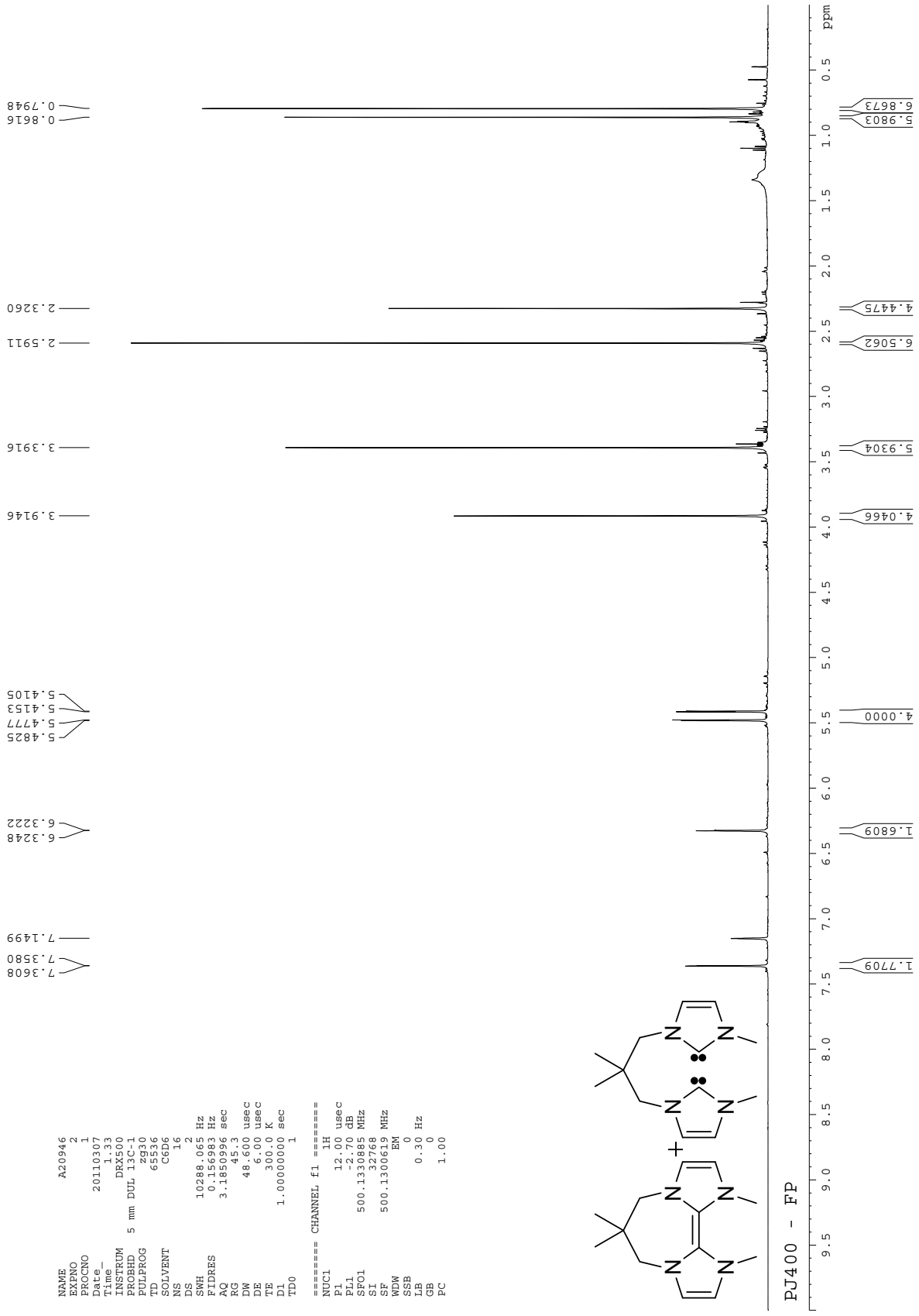

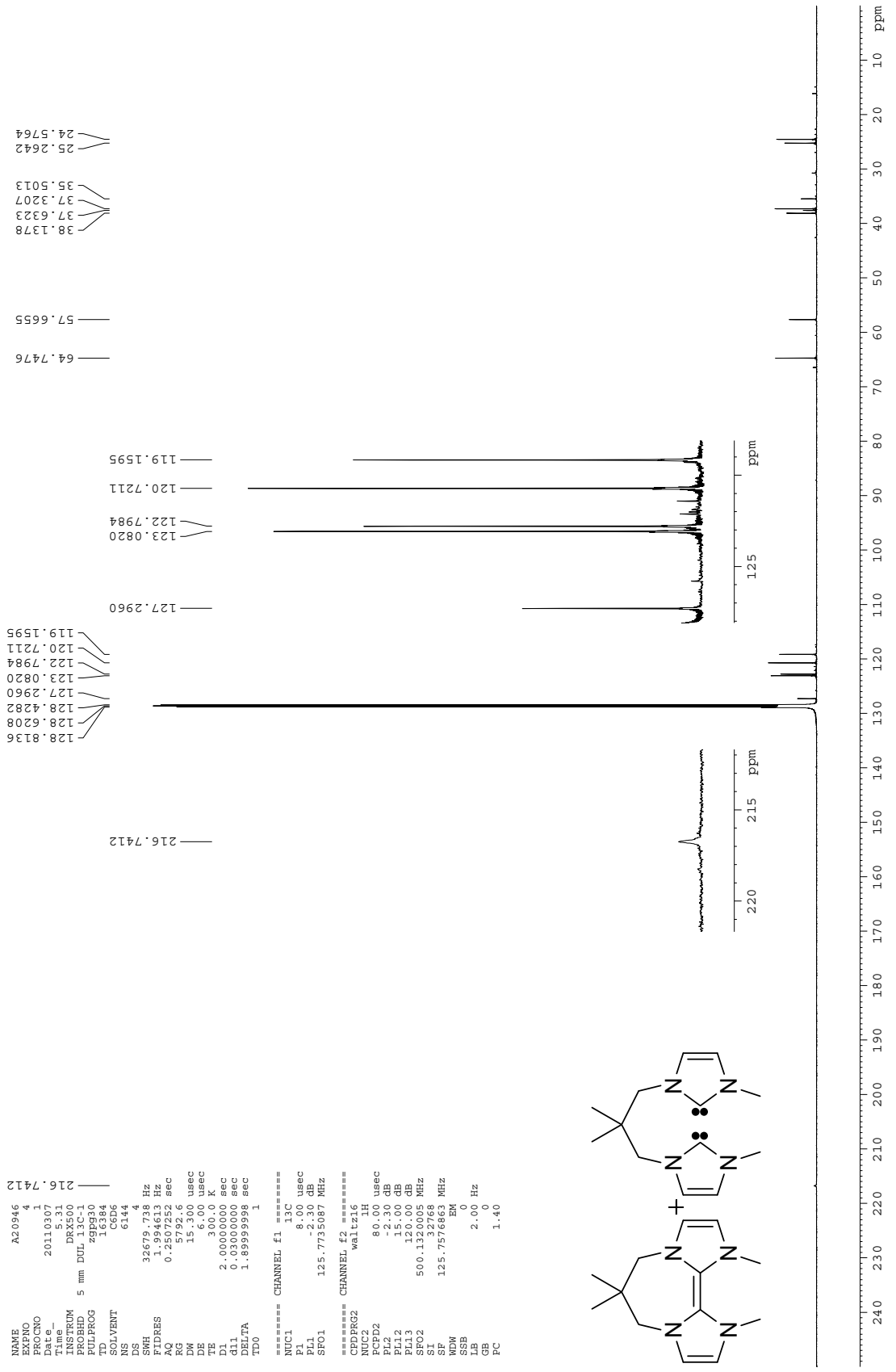
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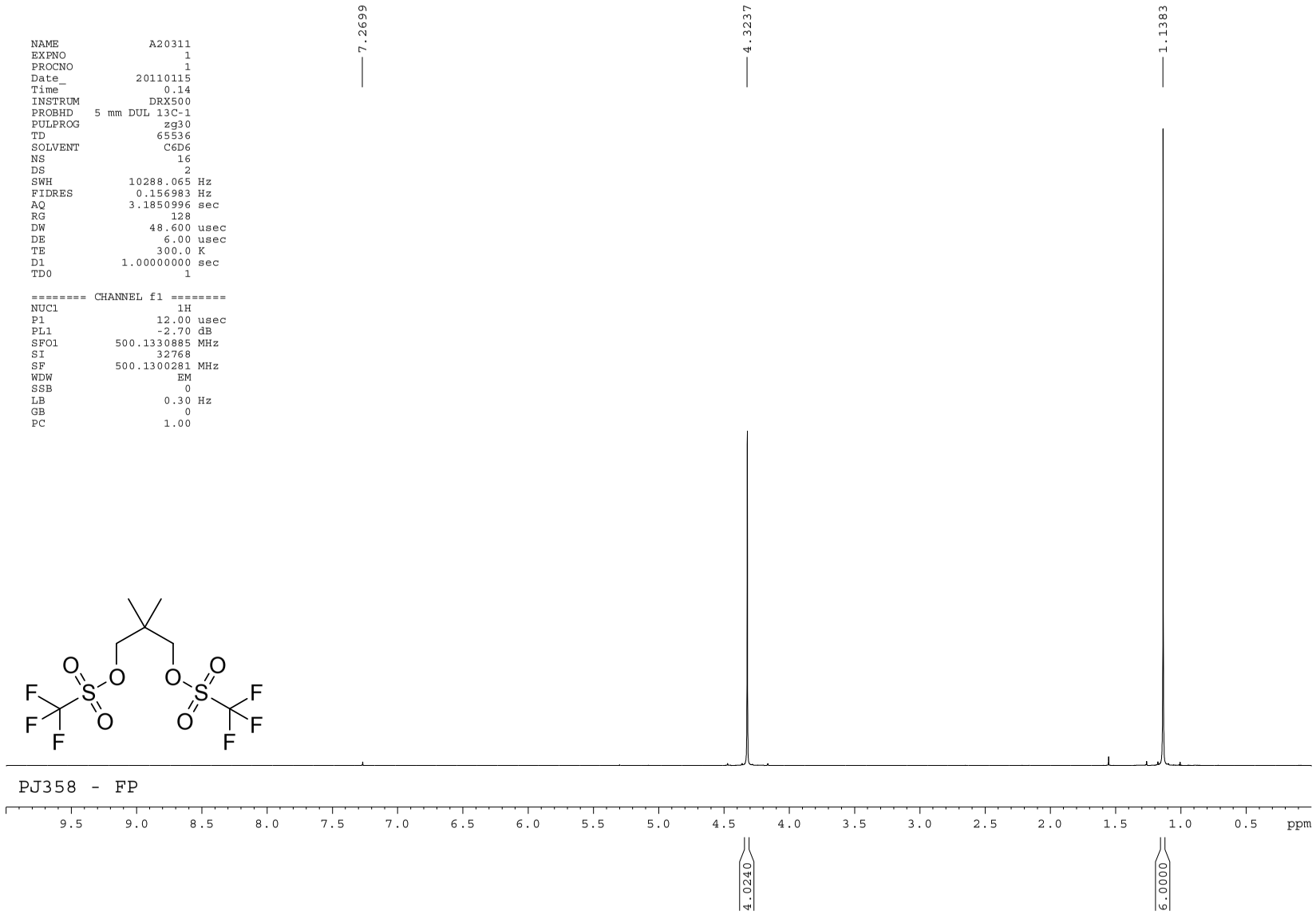

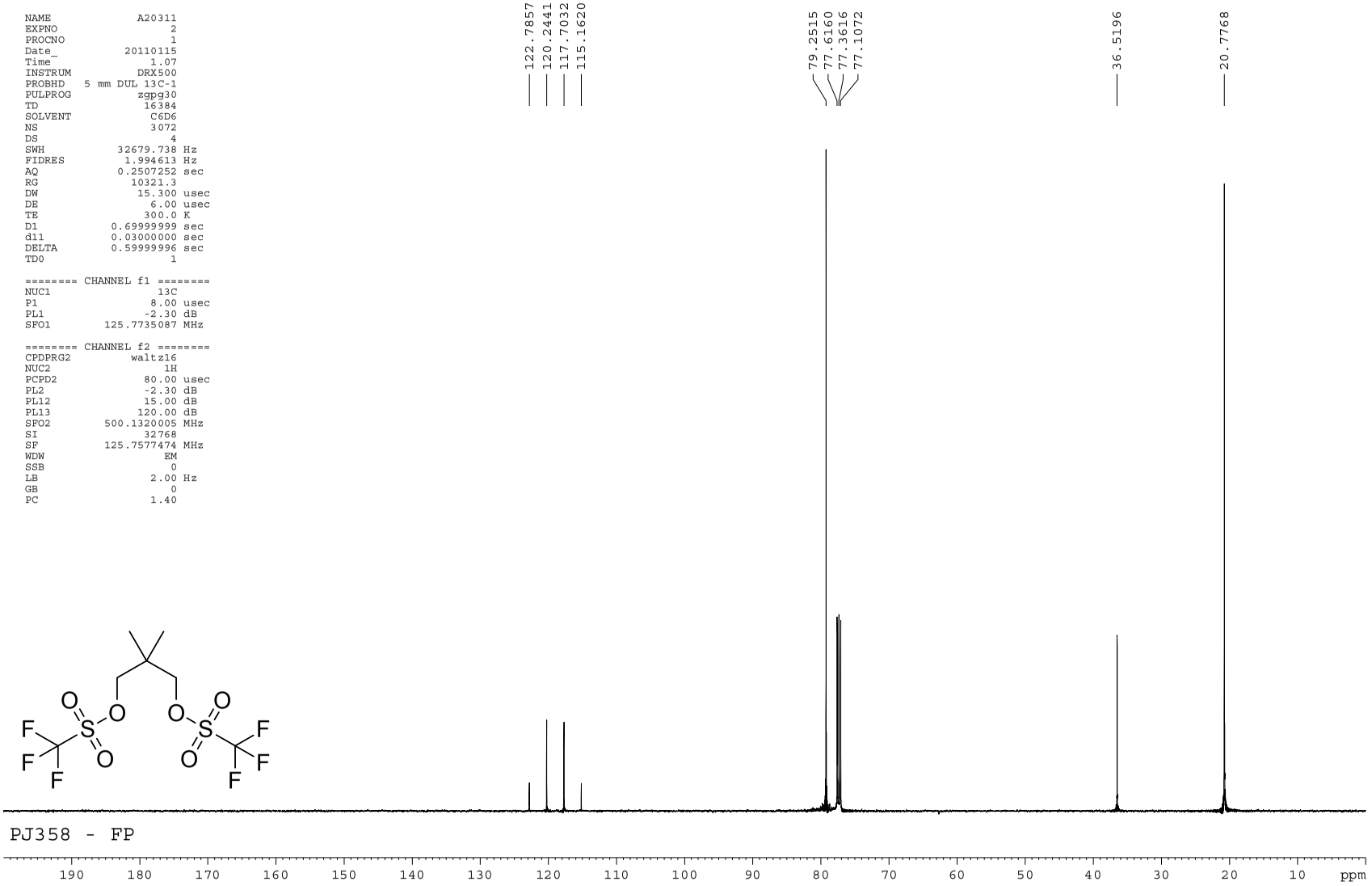

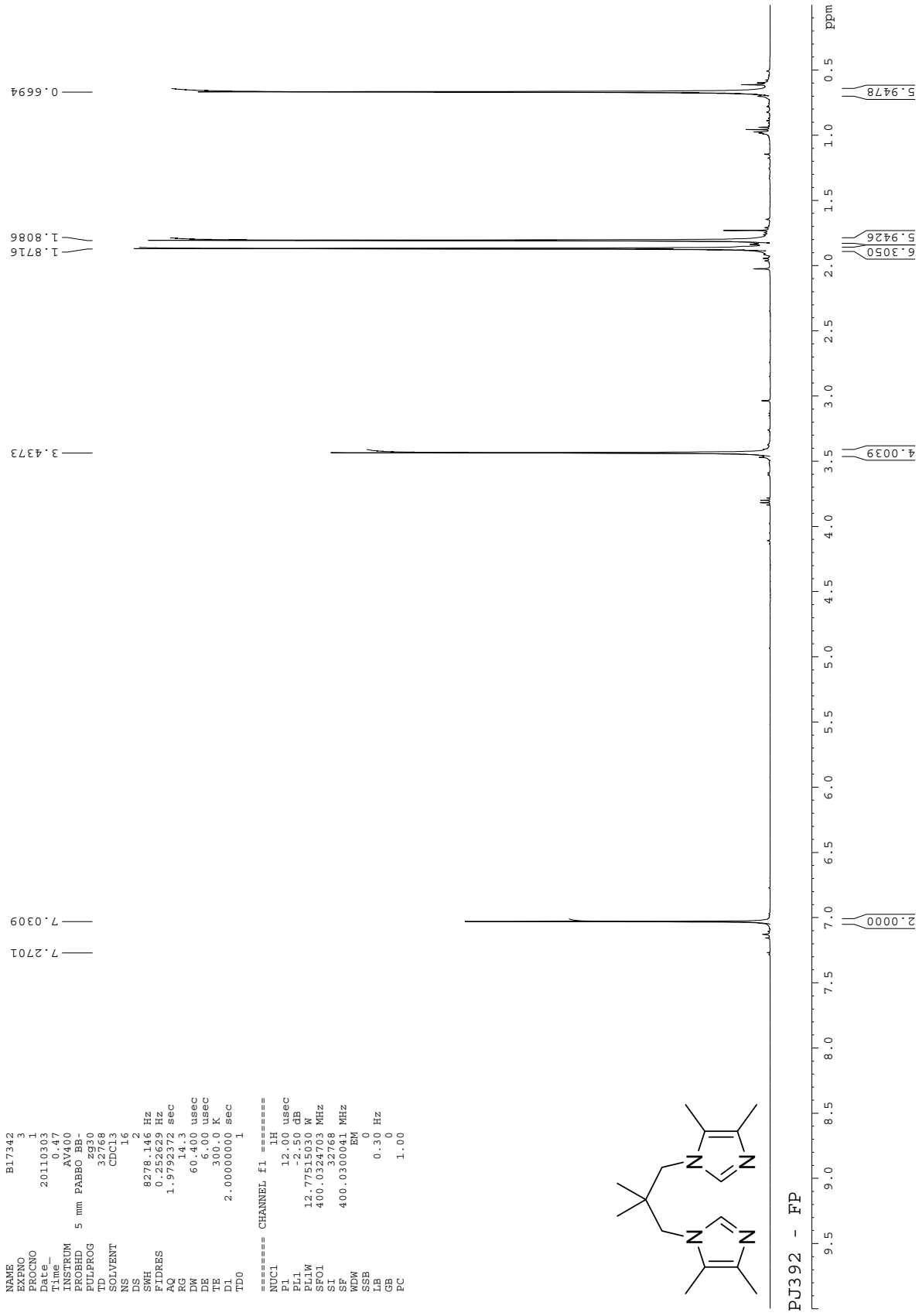
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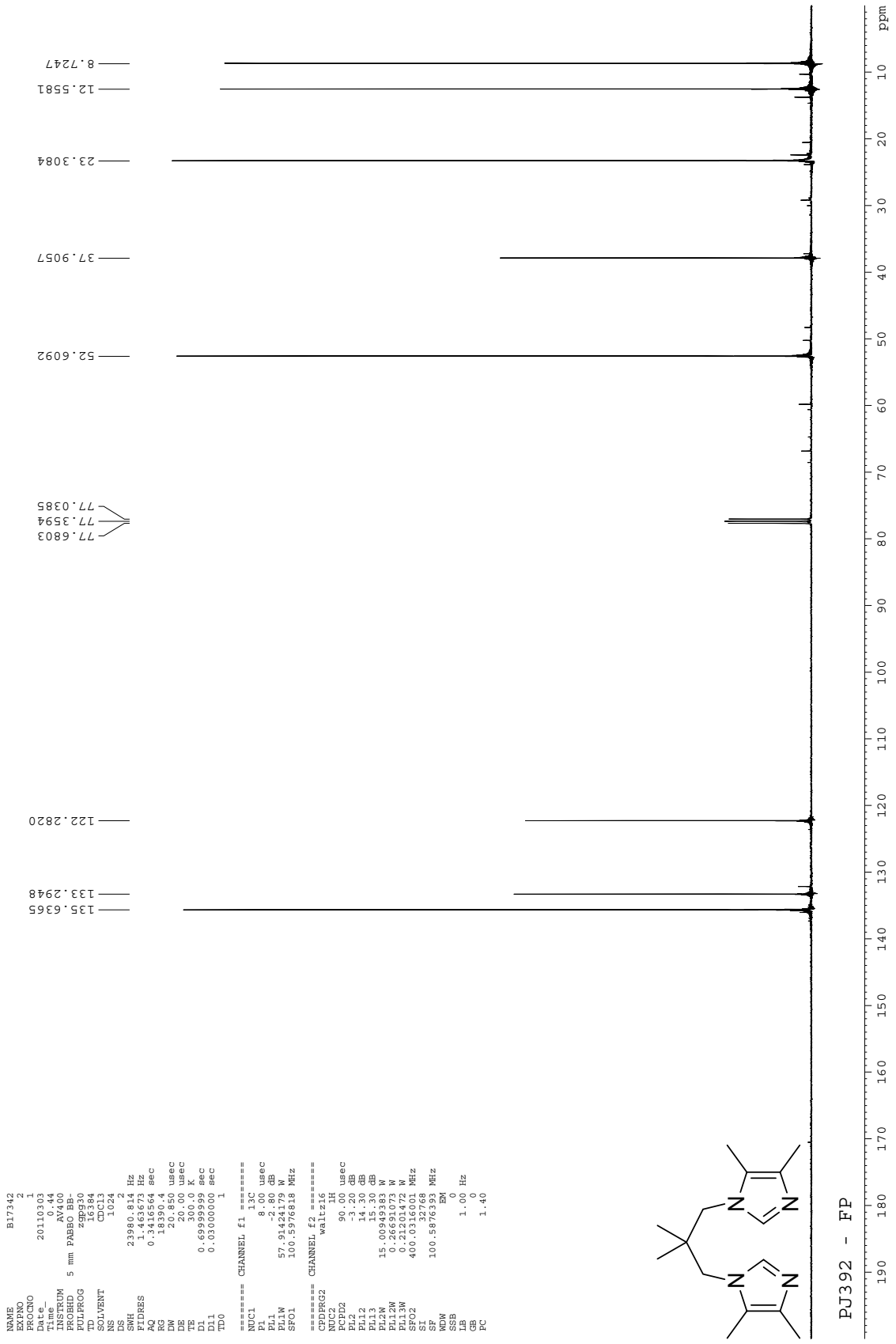
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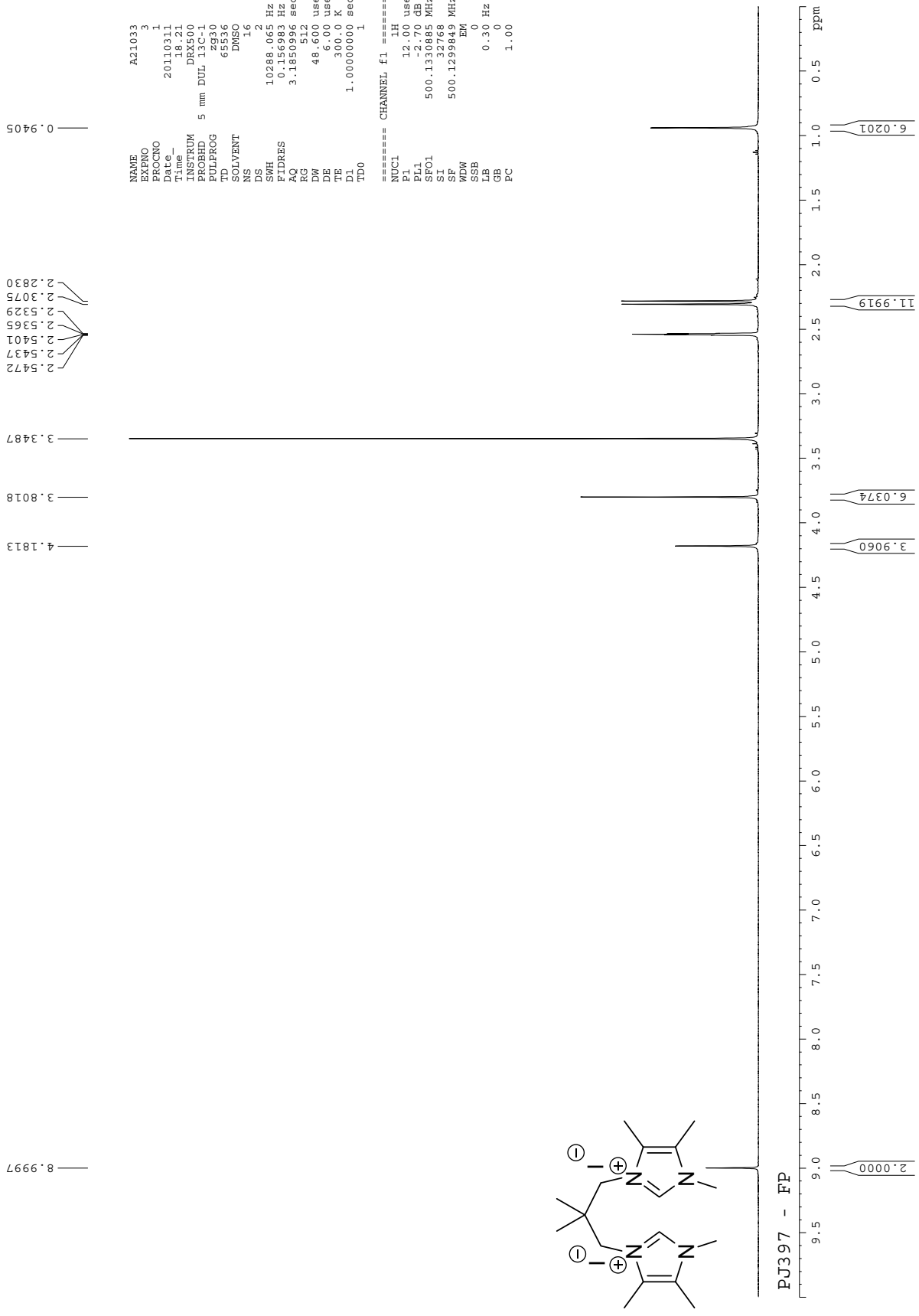
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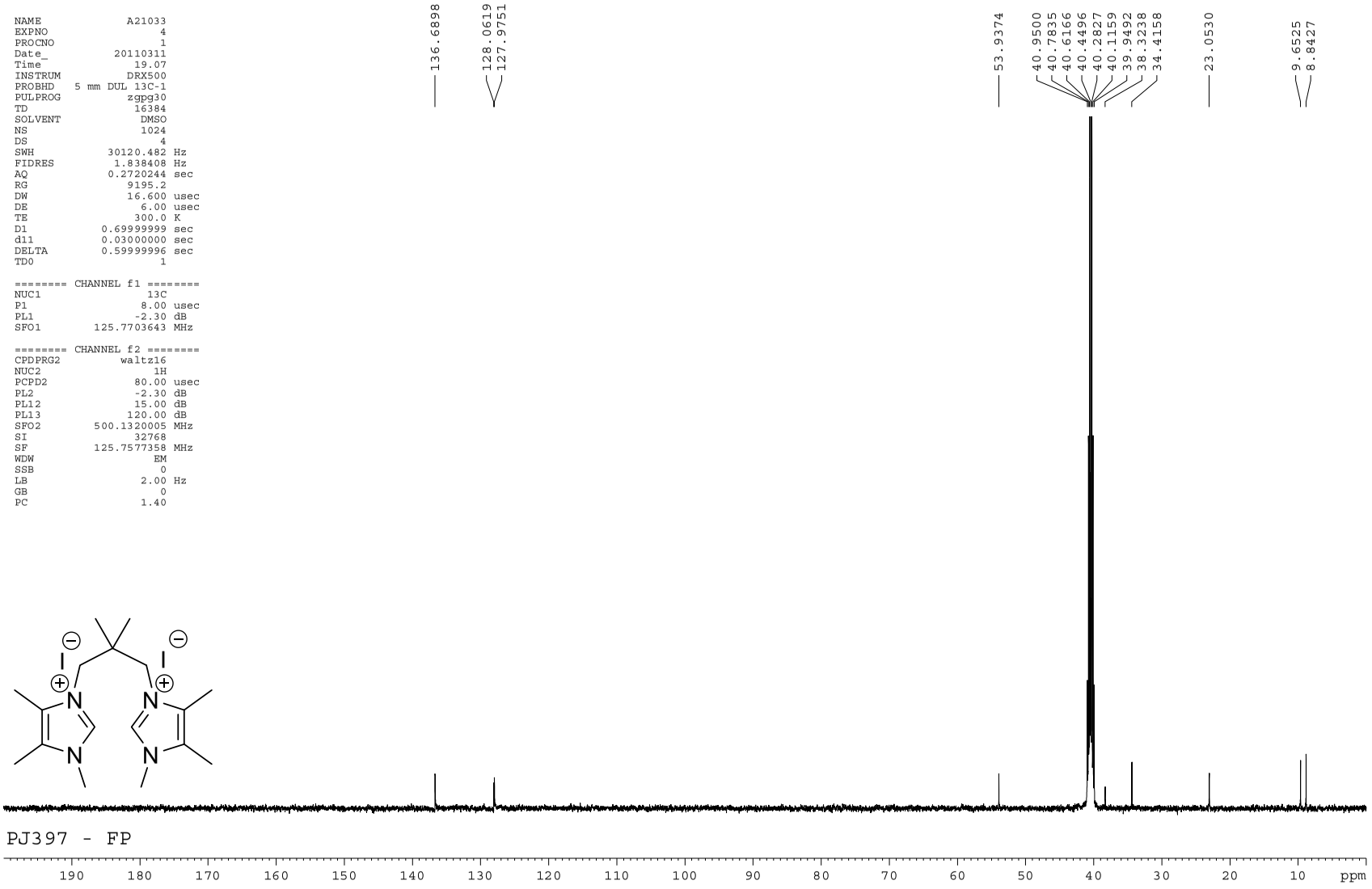
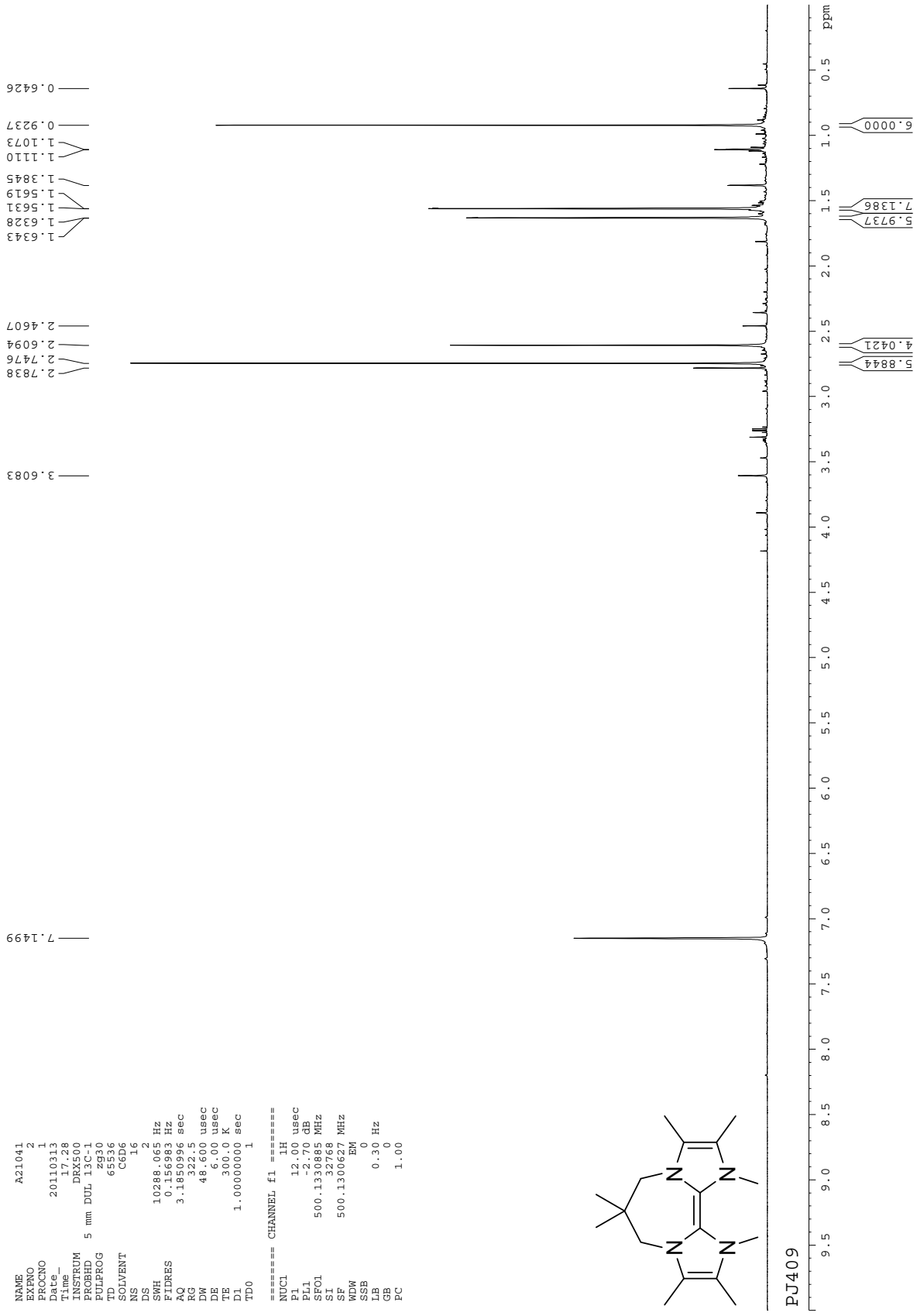
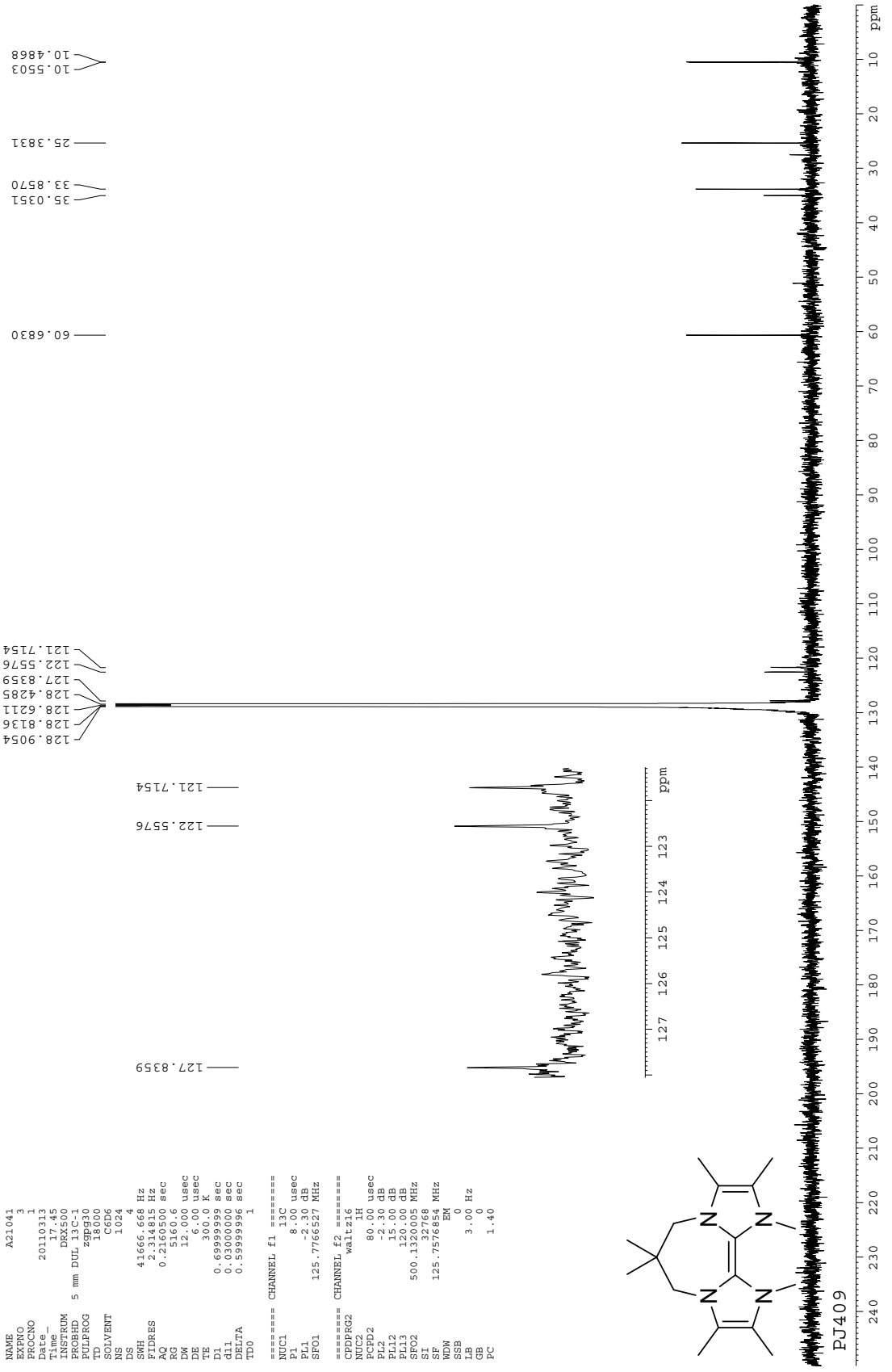
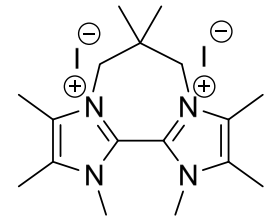
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1299849 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

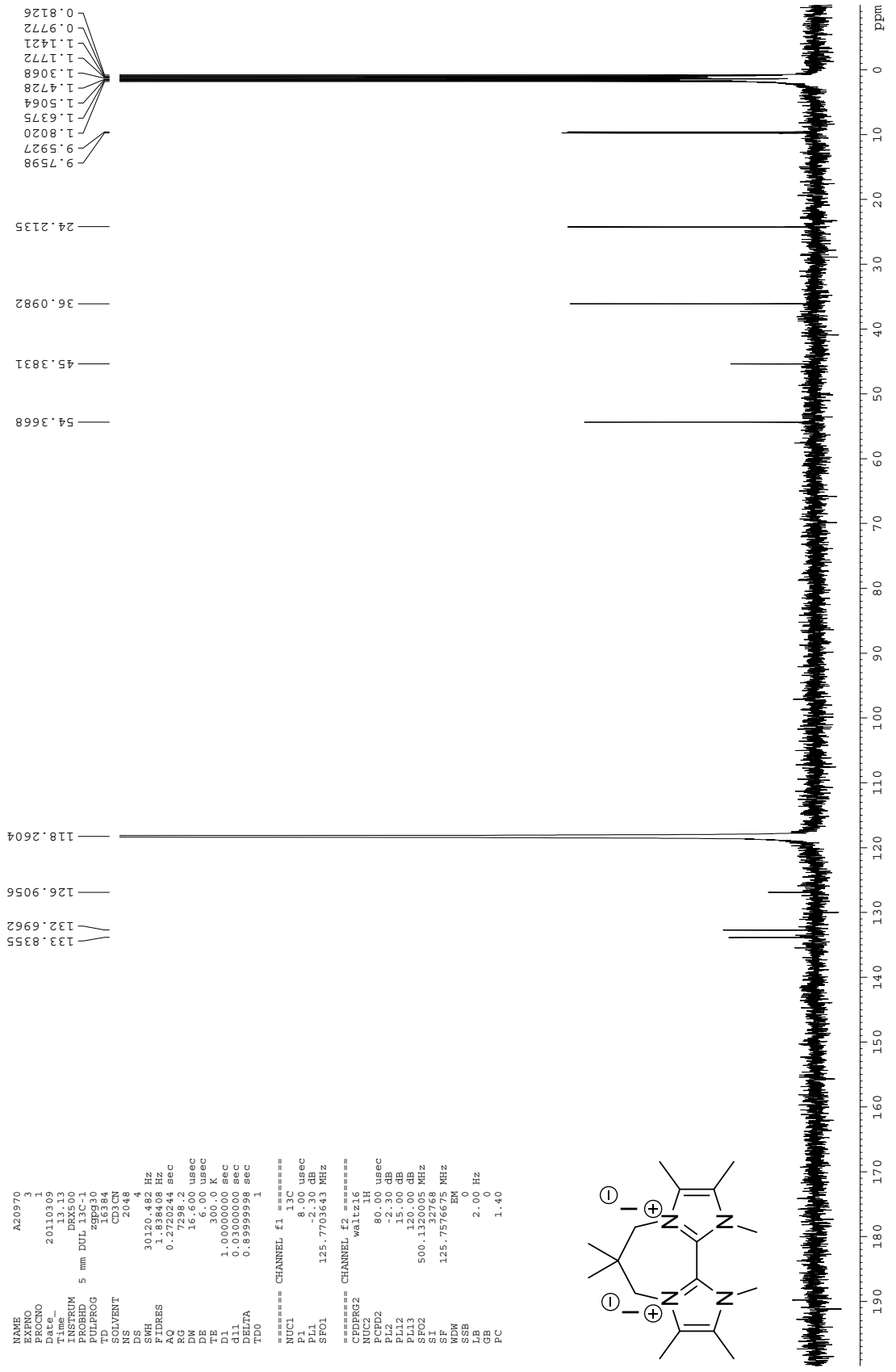


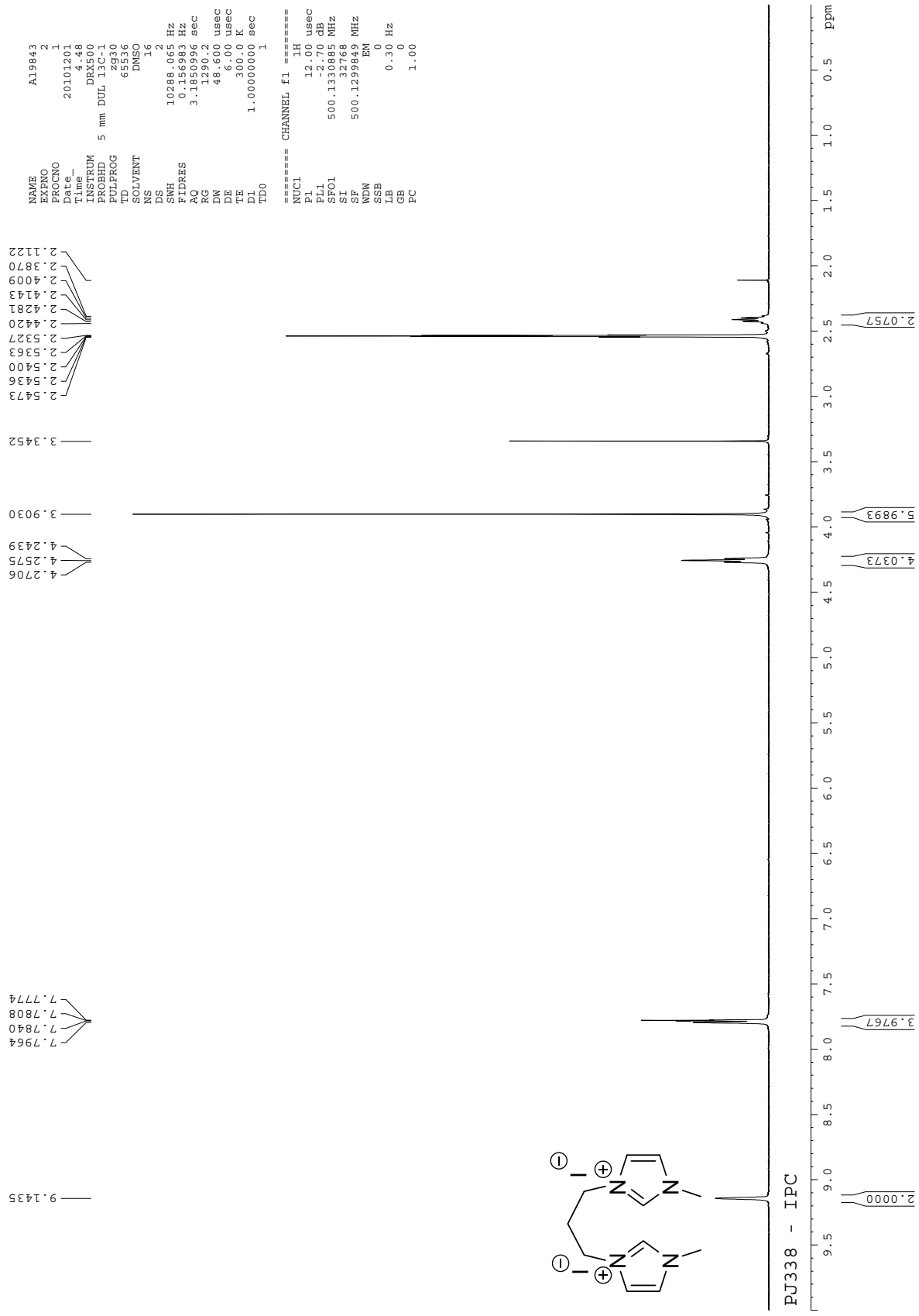

NAME A21041
EXPNO 2
PROCNO 1
Date_ 20110313
Time_ 17.28
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850996 sec
RG 322.5
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300627 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.1499
3.6083
2.7838
2.7476
2.6094
2.4607
1.6343
1.6328
1.5631
1.5619
1.3845
1.1110
1.1073
0.9237
0.6426
6.0000
5.9737
7.1386
4.0421
5.8844
PJ409
ppm

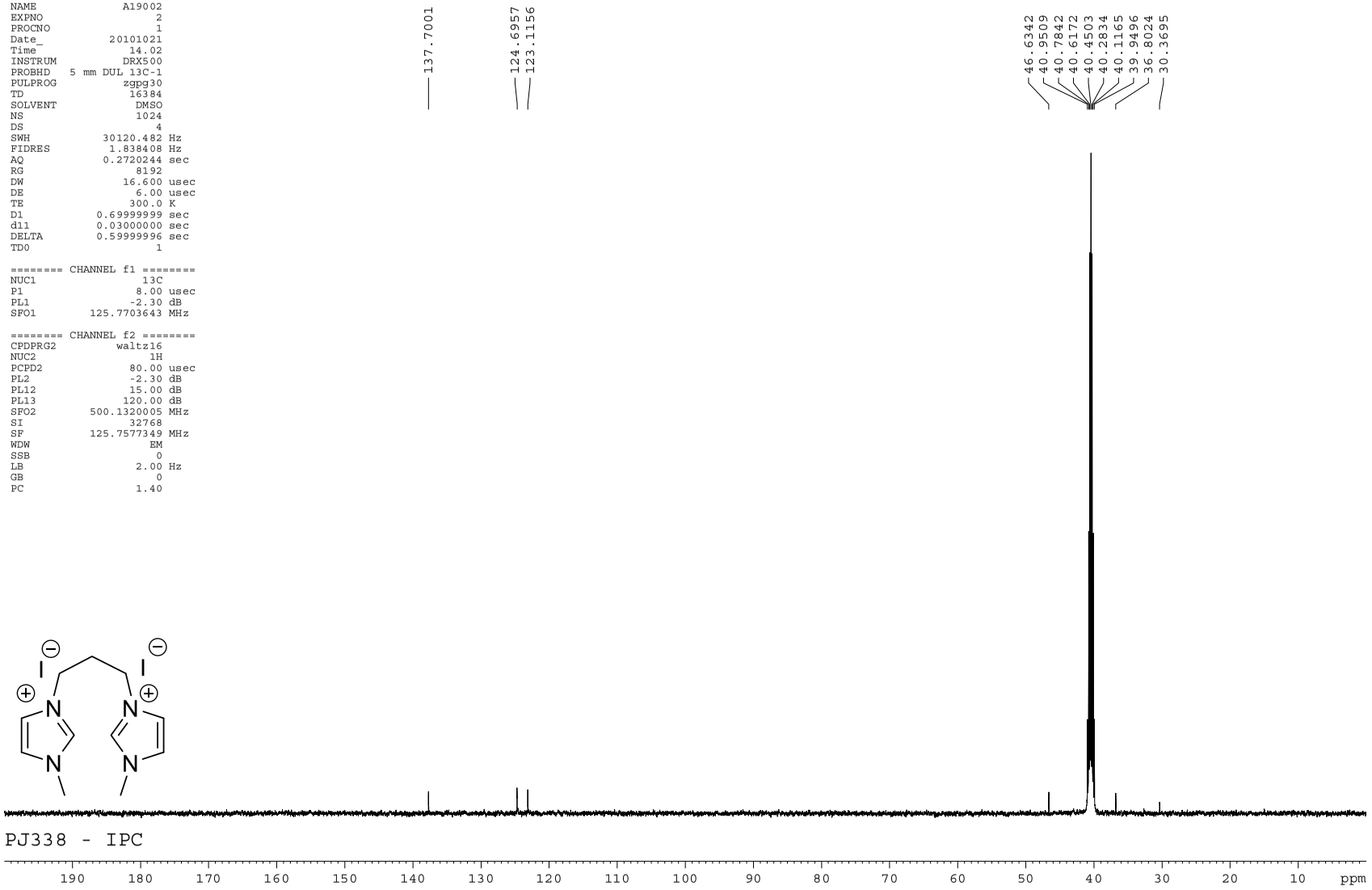

10.4868
10.5503
25.3831
33.8570
35.0351
60.6830
121.7154
122.5576
127.8359
128.4285
128.6211
128.8136
128.9054
PJ409

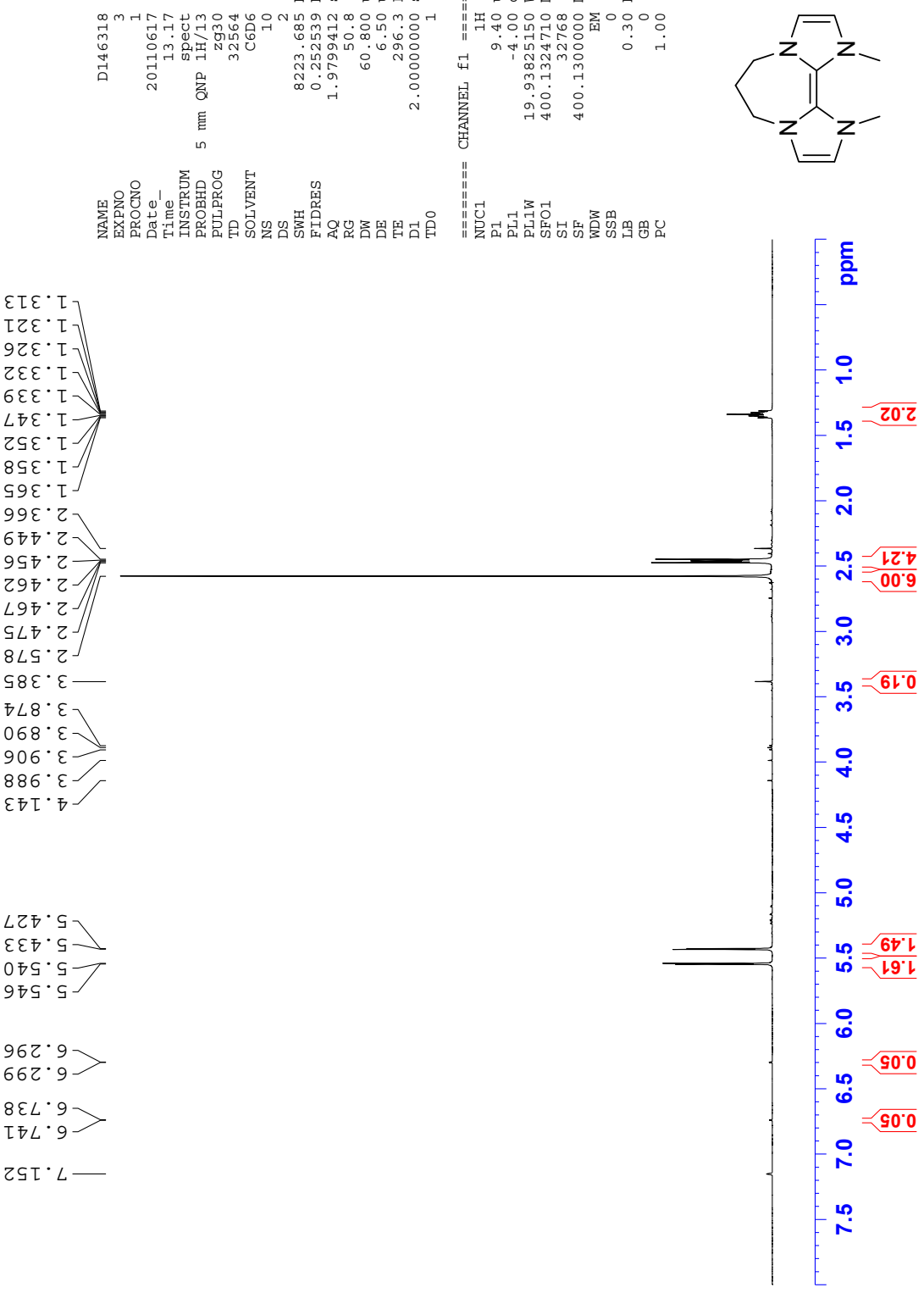
NAME	A20970
EXPNO	4
PROCNO	1
Date_	20110309
Time	14.03
INSTRUM	DRX500
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CD3CN
NS	16
DS	2
SWH	10288.065 Hz
FIDRES	0.156983 Hz
AQ	3.1850996 sec
RG	322.5
DW	48.600 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.00 usec
PL1	-2.70 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

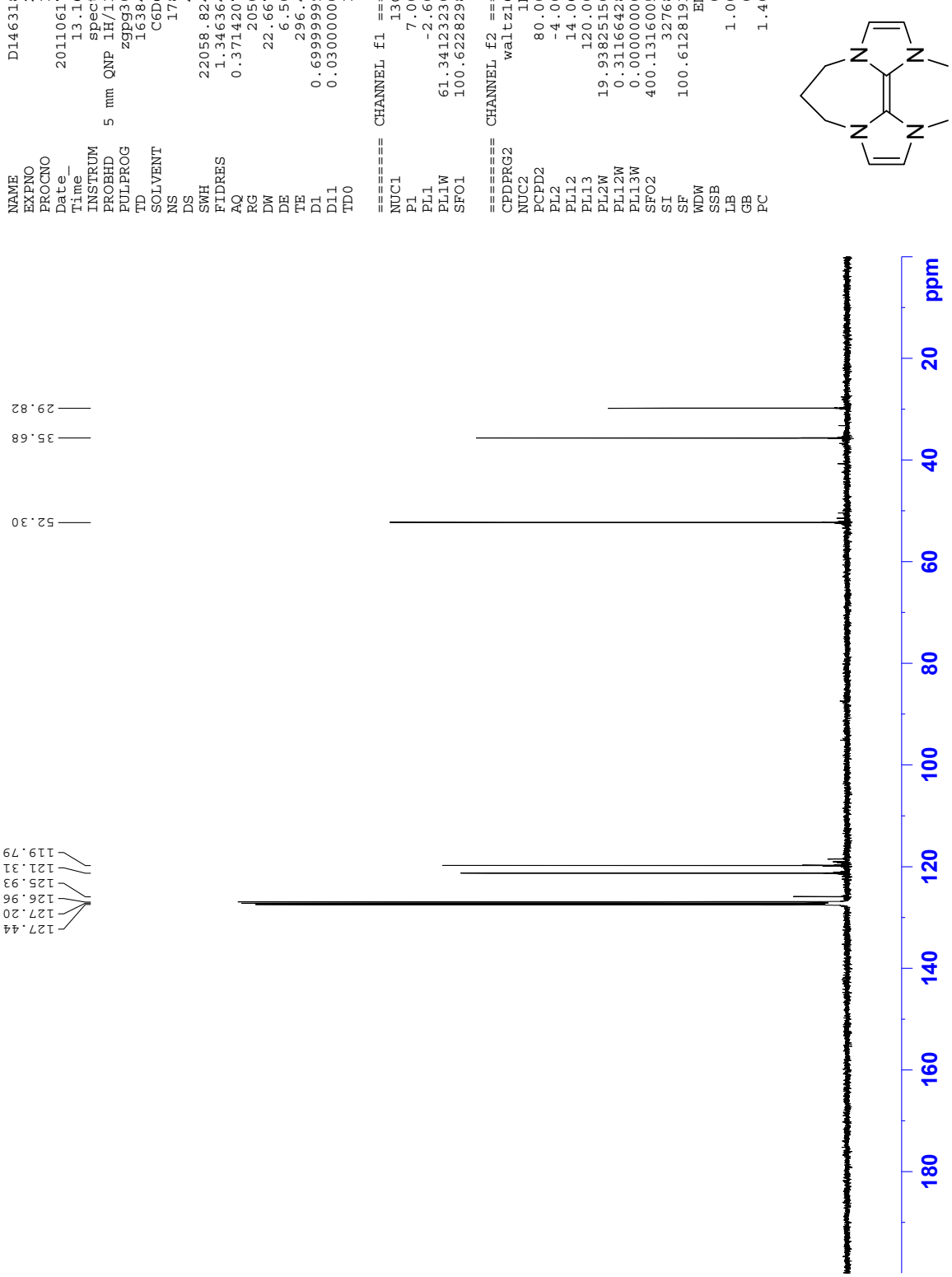


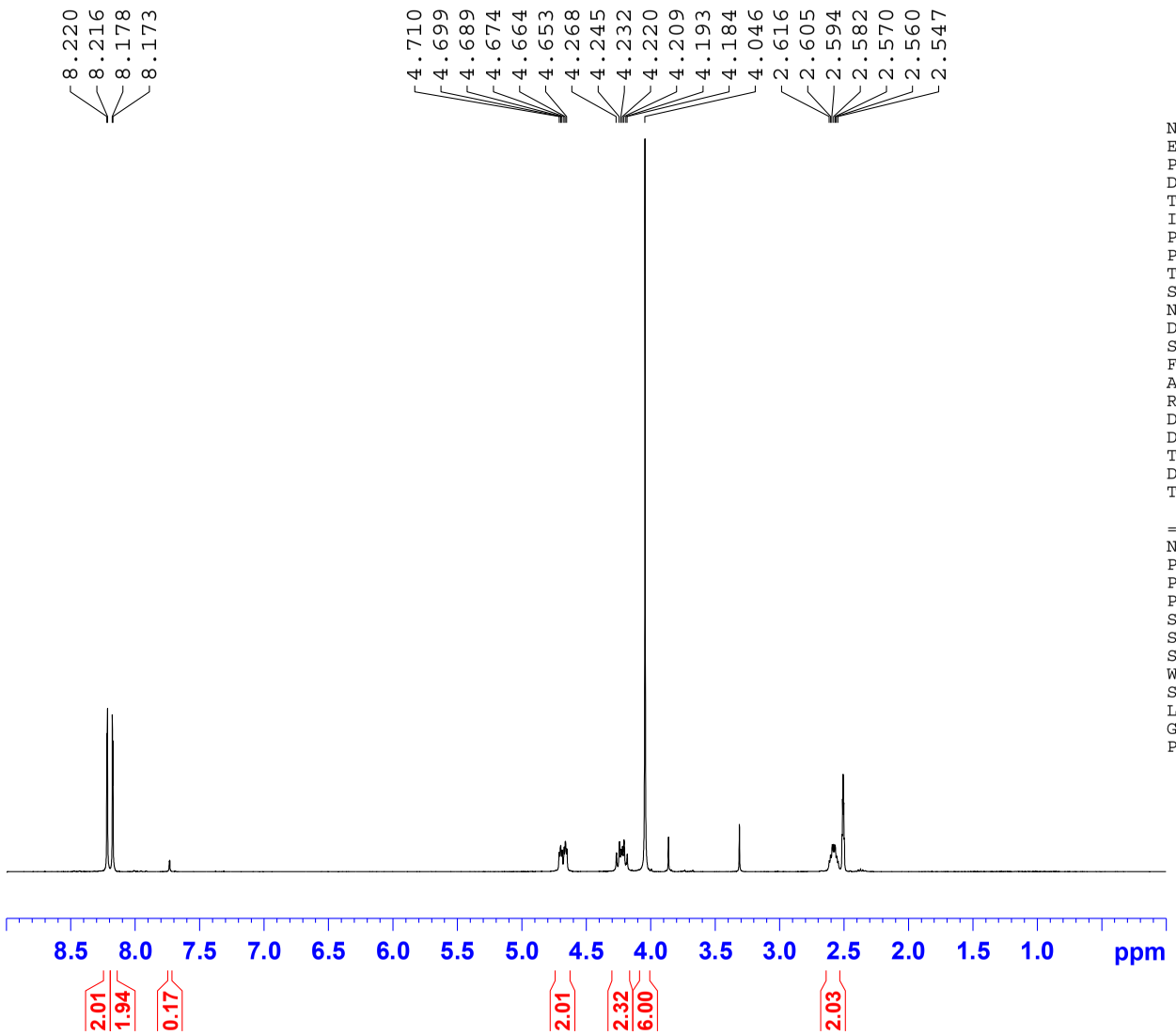



PJ338 - IPC
ppm
NAME A19843
EXPNO 2
PROCNO 1
Date_ 20101201
Time_ 4.48
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850996 sec
RG 1290.2
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1299849 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.1122
2.3870
2.4009
2.4143
2.4281
2.4420
2.5327
2.5363
2.5400
2.5436
2.5473
3.3452
3.9030
4.2439
4.2575
4.2706
7.7764
7.7808
7.7840
7.7964
9.1435
2.0757
5.9893
4.0373
3.9767
2.0000

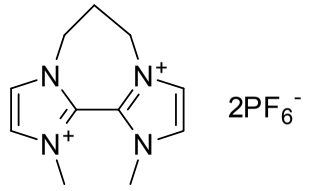


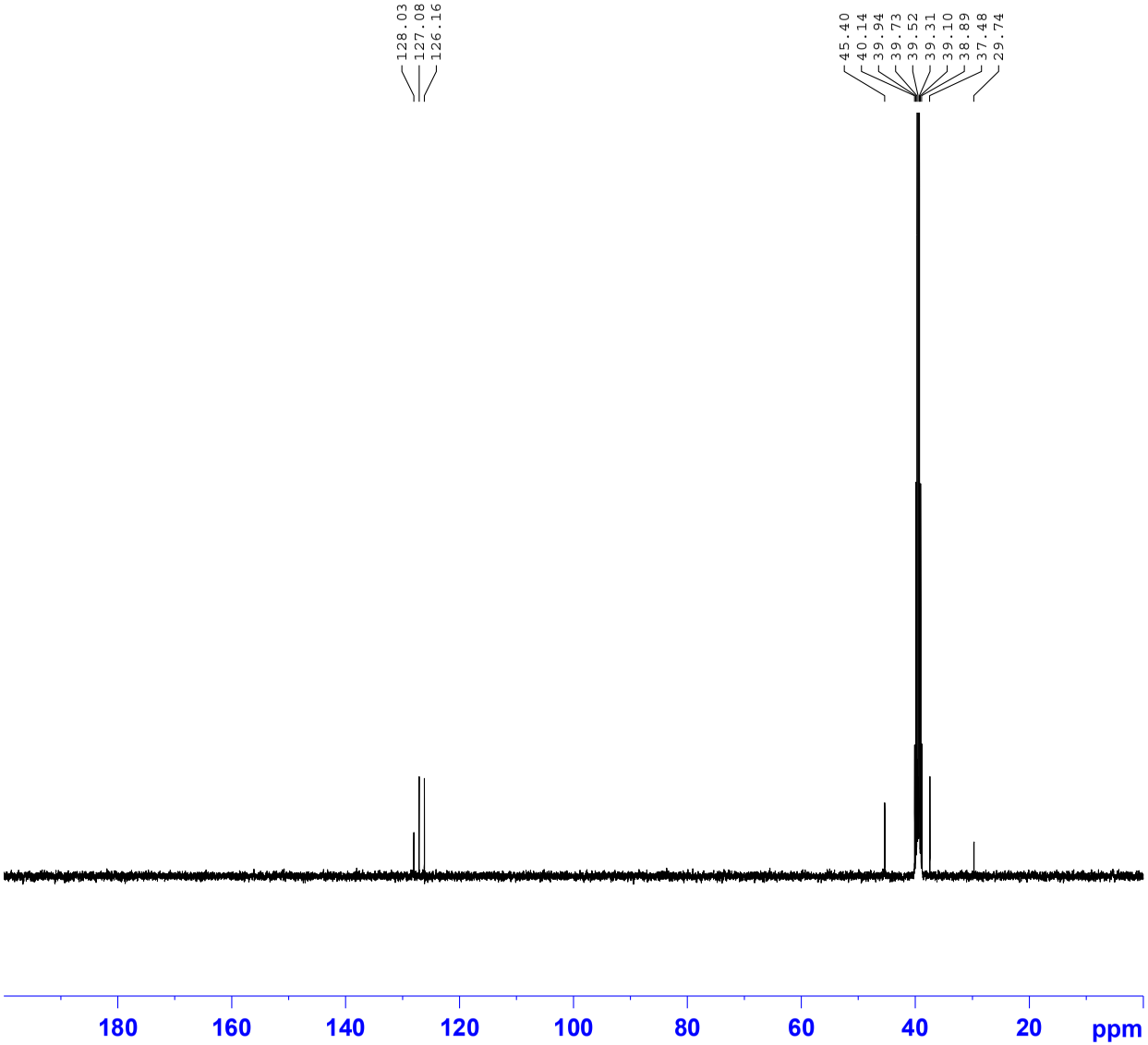

NAME D146318
EXPNO 3
PROCNO 1
Date_ 20110617
Time 13.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32564
SOLVENT C6D6
NS 10
DS 2
SWH 8223.685 Hz
FIDRES 0.252539 Hz
AQ 1.9799412 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.40 usec
PL1 -4.00 dB
PL1W 19.93825150 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
1.313
1.321
1.326
1.332
1.339
1.347
1.352
1.358
1.365
2.366
2.449
2.456
2.462
2.467
2.475
2.578
3.385
3.874
3.890
3.906
3.988
4.143
5.427
5.433
5.540
5.546
6.296
6.299
6.738
6.741
7.152
2.02
4.21
6.00
0.19
1.61
1.49
0.05
0.05


NAME D146318
EXPNO 2
PROCNO 1
Date_ 20110617
Time_ 13.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 16384
SOLVENT C6D6
NS 178
DS 4
SWH 22058.824 Hz
FIDRES 1.346364 Hz
AQ 0.3714207 sec
RG 2050
DW 22.667 usec
DE 6.50 usec
TE 296.4 K
D1 0.69999999 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -2.60 dB
PL1W 61.34123230 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.06 dB
PL13 120.00 dB
PL2W 19.93825150 W
PL12W 0.31166428 W
PL13W 0.00000000 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
29.82
35.68
52.30
119.79
121.31
125.93
126.96
127.20
127.44
ppm
20
40
60
80
100
120
140
160
180

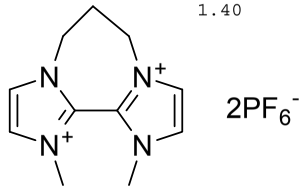


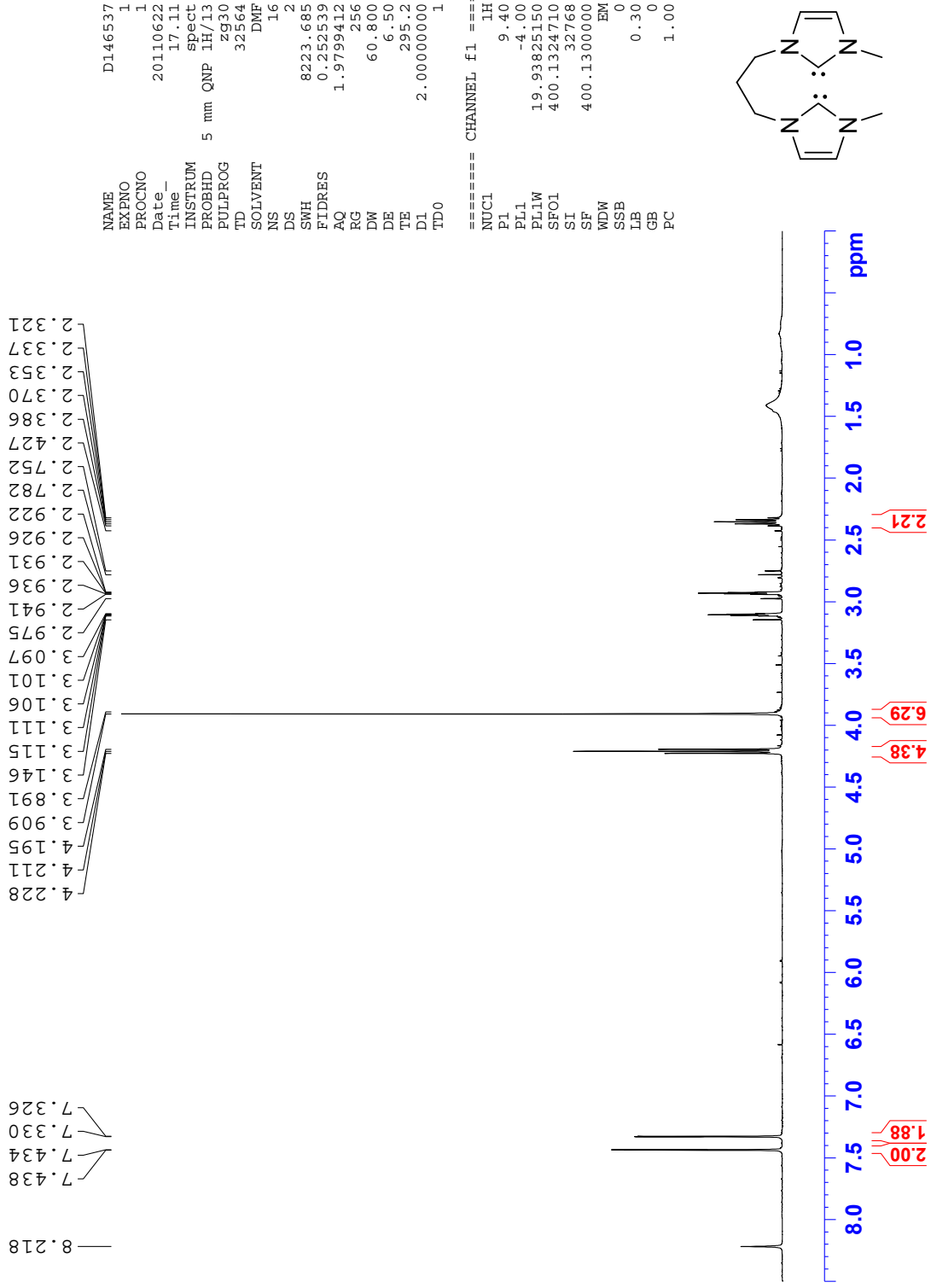
NAME	D150549
EXPNO	1
PROCNO	1
Date_	20111003
Time	18.22
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32564
SOLVENT	DMSO
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.252539 Hz
AQ	1.9799412 sec
RG	406
DW	60.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-4.00 dB
PL1W	19.93825150 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

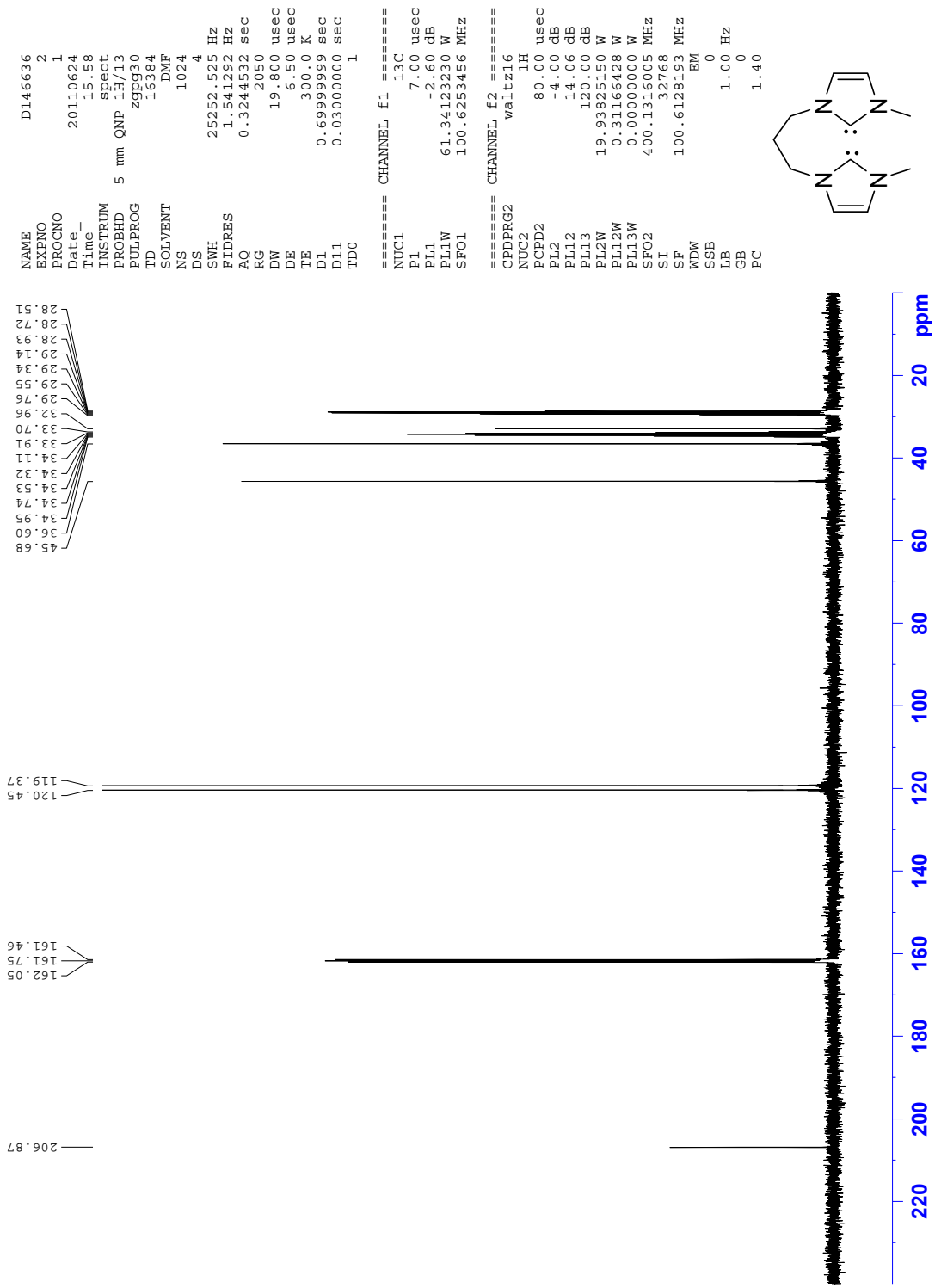


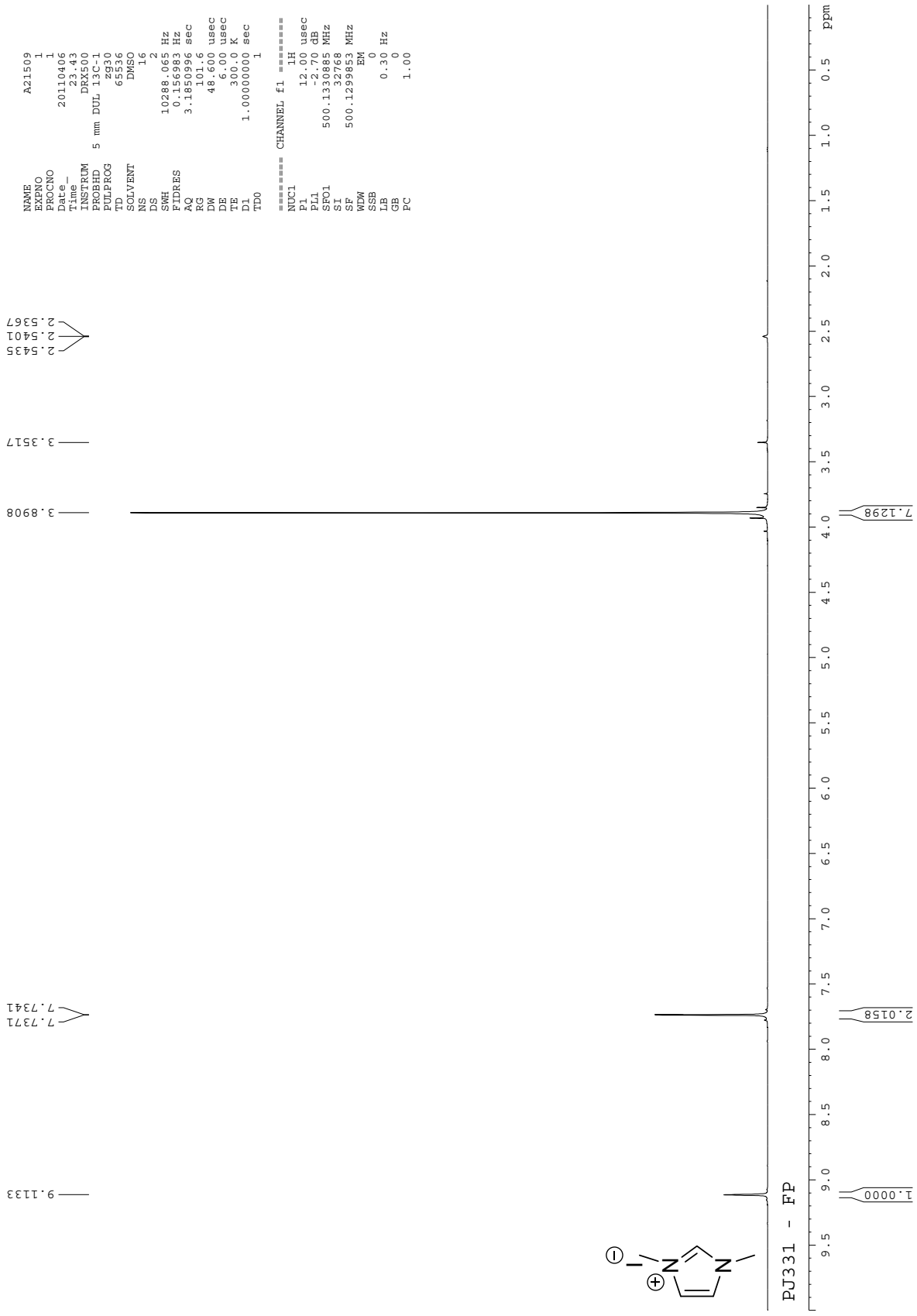


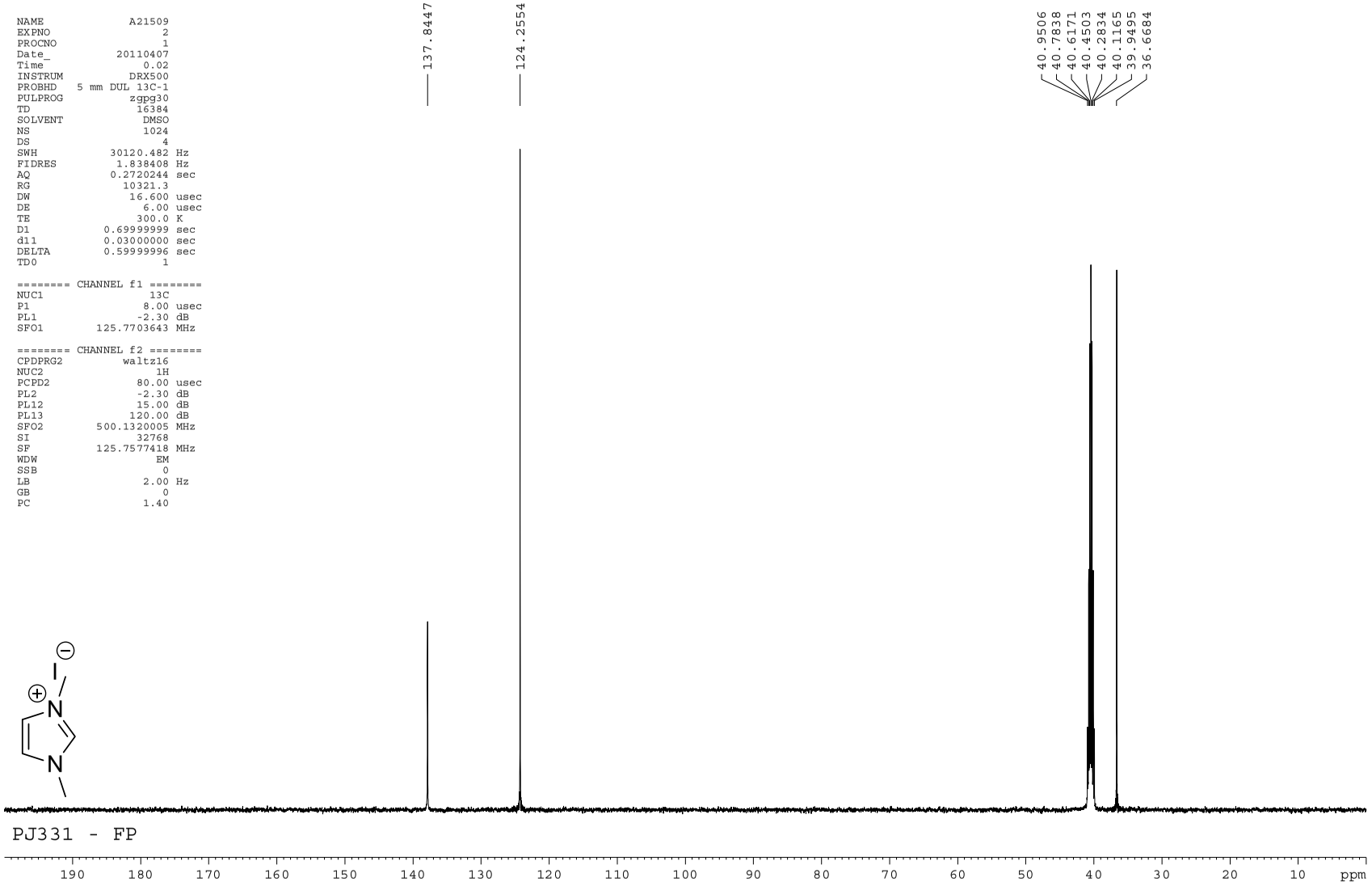
NAME	D150549
EXPNO	2
PROCNO	1
Date_	20111003
Time	18.42
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	16384
SOLVENT	DMSO
NS	1024
DS	4
SWH	22058.824 Hz
FIDRES	1.346364 Hz
AQ	0.3714207 sec
RG	2050
DW	22.667 usec
DE	6.50 usec
TE	300.0 K
D1	0.69999999 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.00 usec
PL1	-2.60 dB
PL1W	61.34123230 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.00 dB
PL12	14.06 dB
PL13	120.00 dB
PL2W	19.93825150 W
PL12W	0.31166428 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6128193 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

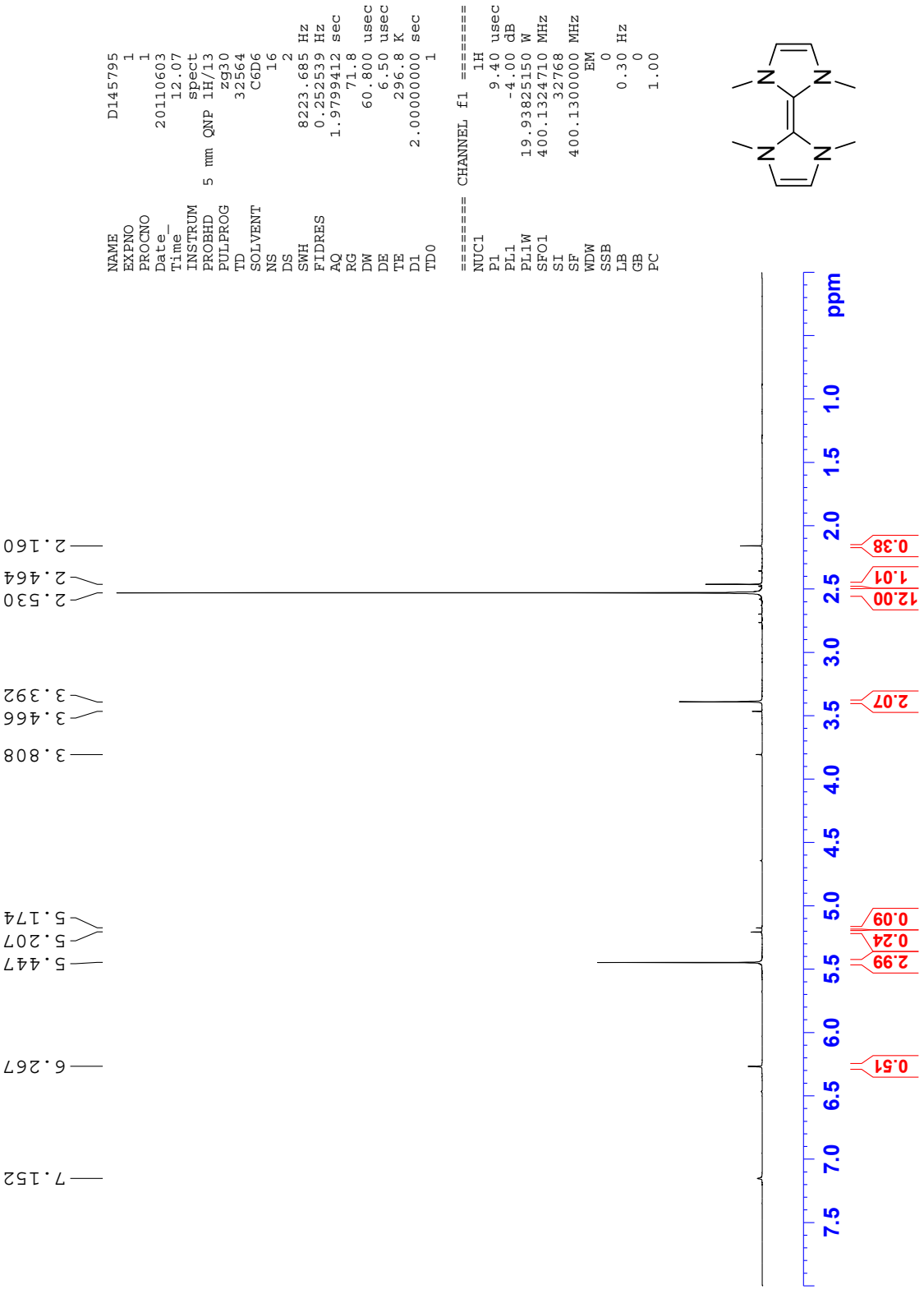


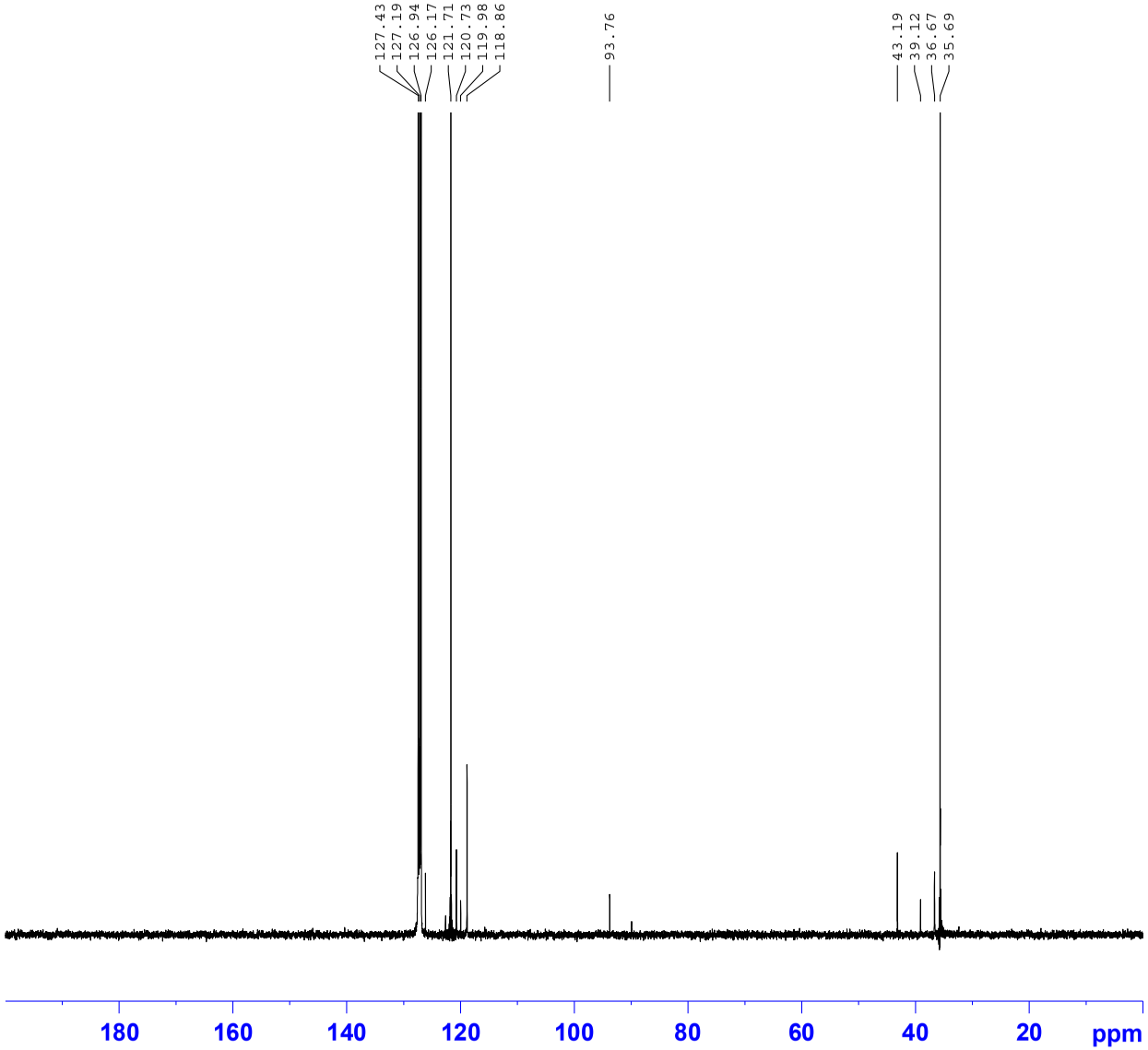

NAME D146537
EXPNO 1
PROCNO 1
Date_ 20110622
Time_ 17.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32564
SOLVENT DMF
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.252539 Hz
AQ 1.97999412 sec
RG 256
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.40 usec
PL1 -4.00 dB
PL1W 19.93825150 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
2.21
6.29
4.38
2.00
1.88


NAME D146636
EXPNO 2
PROCNO 1
Date_ 20110624
Time_ 15.58
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 16384
SOLVENT DMF
NS 1024
DS 4
SWH 25252.525 Hz
FIDRES 1.541292 Hz
AQ 0.32444532 sec
RG 2050
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 0.69999999 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -2.60 dB
PL1W 61.34123230 W
SFO1 100.6253456 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.06 dB
PL13 120.00 dB
PL2W 19.93825150 W
PL12W 0.31166428 W
PL13W 0.00000000 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
206.87
162.05
161.75
161.46
120.45
119.37
45.68
36.60
34.95
34.74
34.53
34.32
34.11
33.91
33.70
32.96
29.76
29.55
29.34
29.14
28.93
28.72
28.51
ppm
220
200
180
160
140
120
100
80
60
40
20

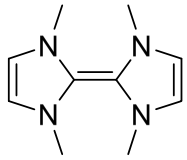

NAME A21509
EXPNO 1
PROCNO 1
Date_ 20110406
Time_ 23.43
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850996 sec
RG 101.6
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1299853 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.1133
7.7741
7.7371
3.8908
3.3517
2.5435
2.5401
2.5367
7.1298
2.0158
1.0000
PJ331 - FP
ppm

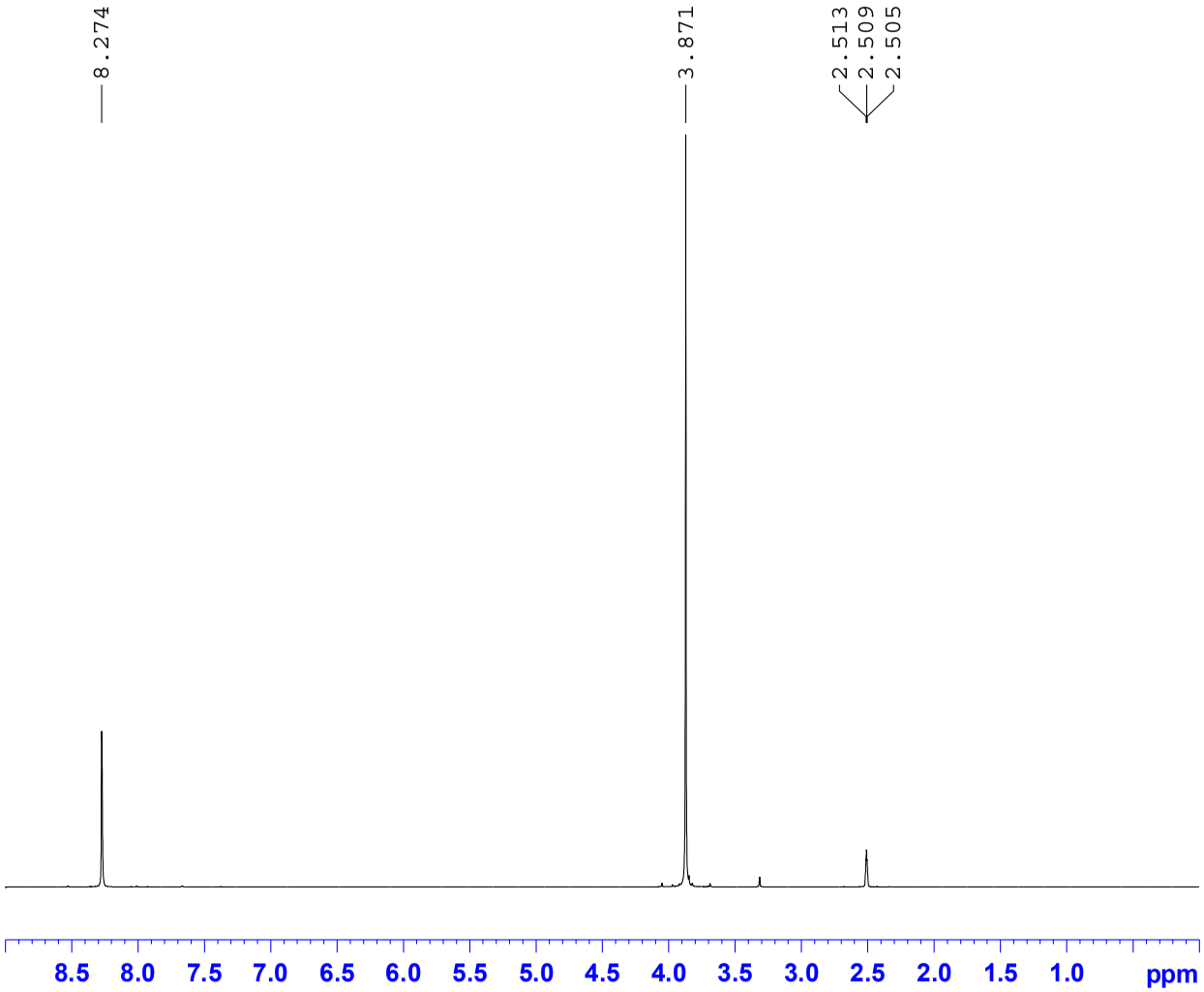



NAME D145795
EXPNO 1
PROCNO 1
Date_ 20110603
Time 12.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32564
SOLVENT C6D6
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.252539 Hz
AQ 1.9799412 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.40 usec
PL1 -4.00 dB
PL1W 19.93825150 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
N
N
7.152
6.267
5.447
5.207
5.174
3.808
3.466
3.392
2.530
2.464
2.160
0.51
2.99
0.24
0.09
2.07
12.00
1.01
0.38
ppm
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

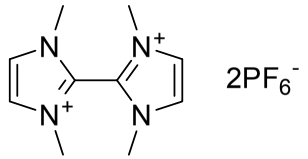


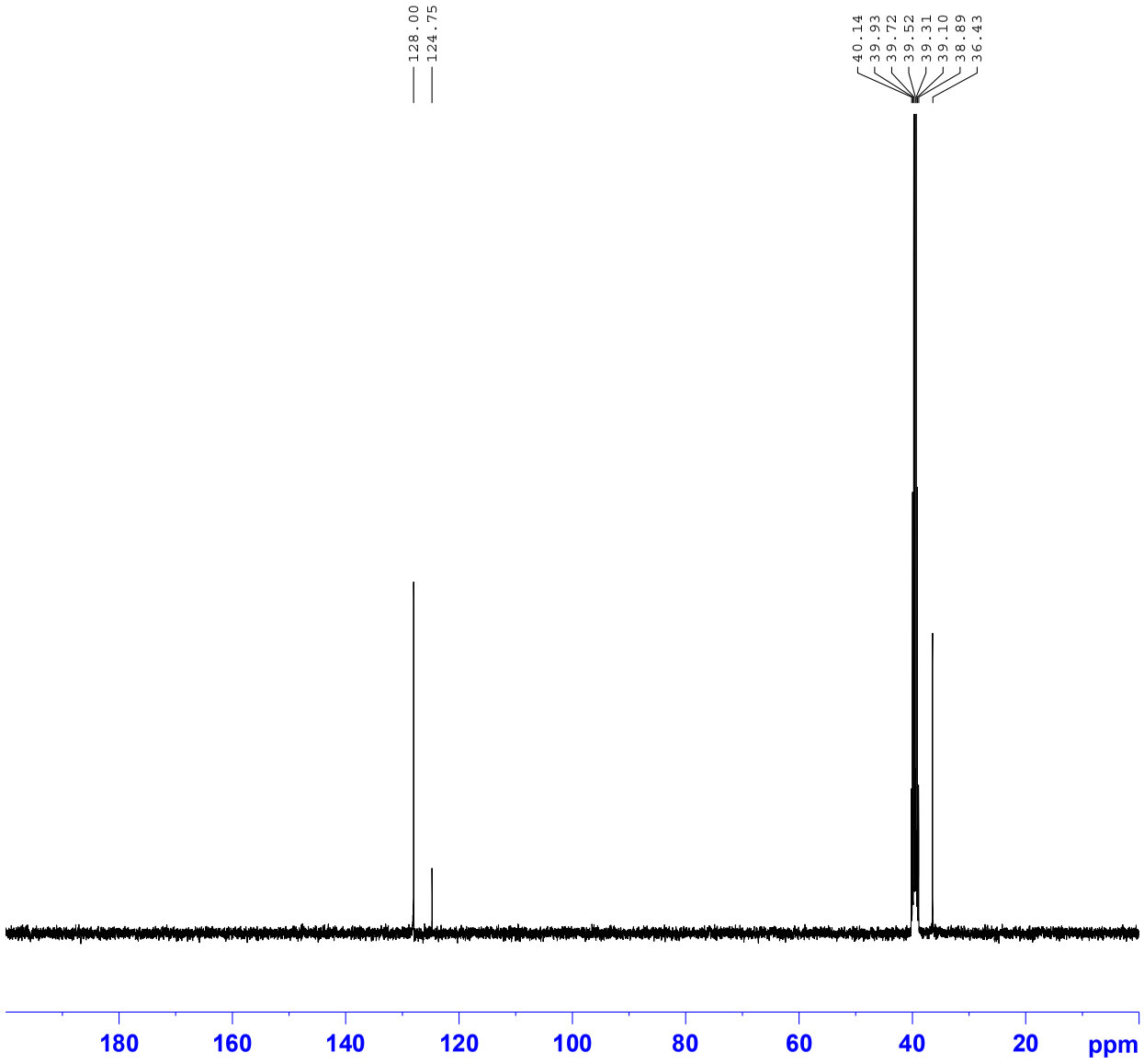
NAME	D145795
EXPNO	2
PROCNO	1
Date_	20110603
Time	12.30
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	16384
SOLVENT	C6D6
NS	1024
DS	4
SWH	22058.824 Hz
FIDRES	1.346364 Hz
AQ	0.3714207 sec
RG	2050
DW	22.667 usec
DE	6.50 usec
TE	297.4 K
D1	0.69999999 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.00 usec
PL1	-2.60 dB
PL1W	61.34123230 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.00 dB
PL12	14.06 dB
PL13	120.00 dB
PL2W	19.93825150 W
PL12W	0.31166428 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6128193 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



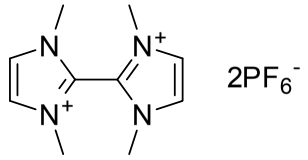


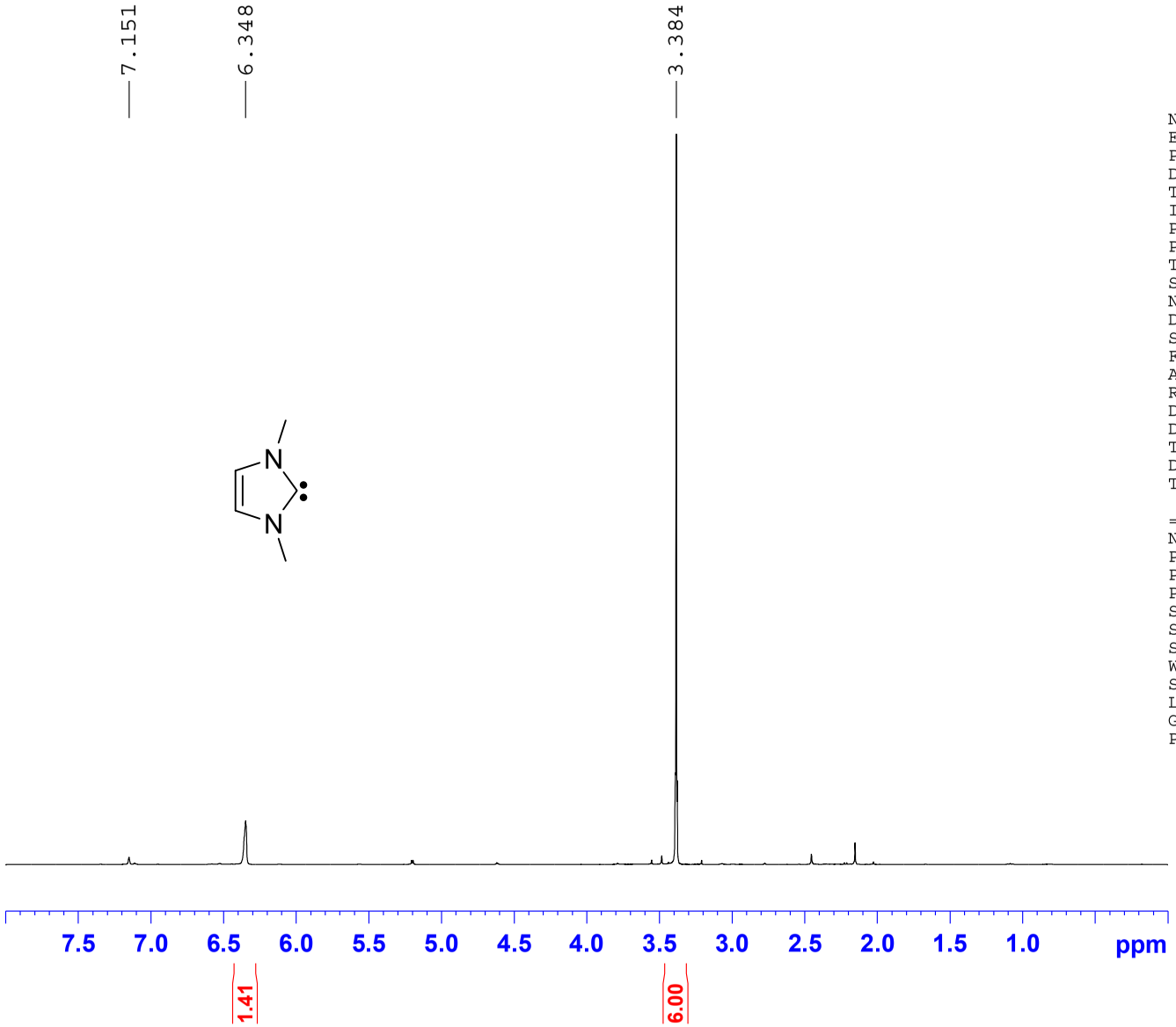
NAME	D150550
EXPNO	1
PROCNO	1
Date_	20111003
Time	18.46
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32564
SOLVENT	DMSO
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.252539 Hz
AQ	1.9799412 sec
RG	228
DW	60.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-4.00 dB
PL1W	19.93825150 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



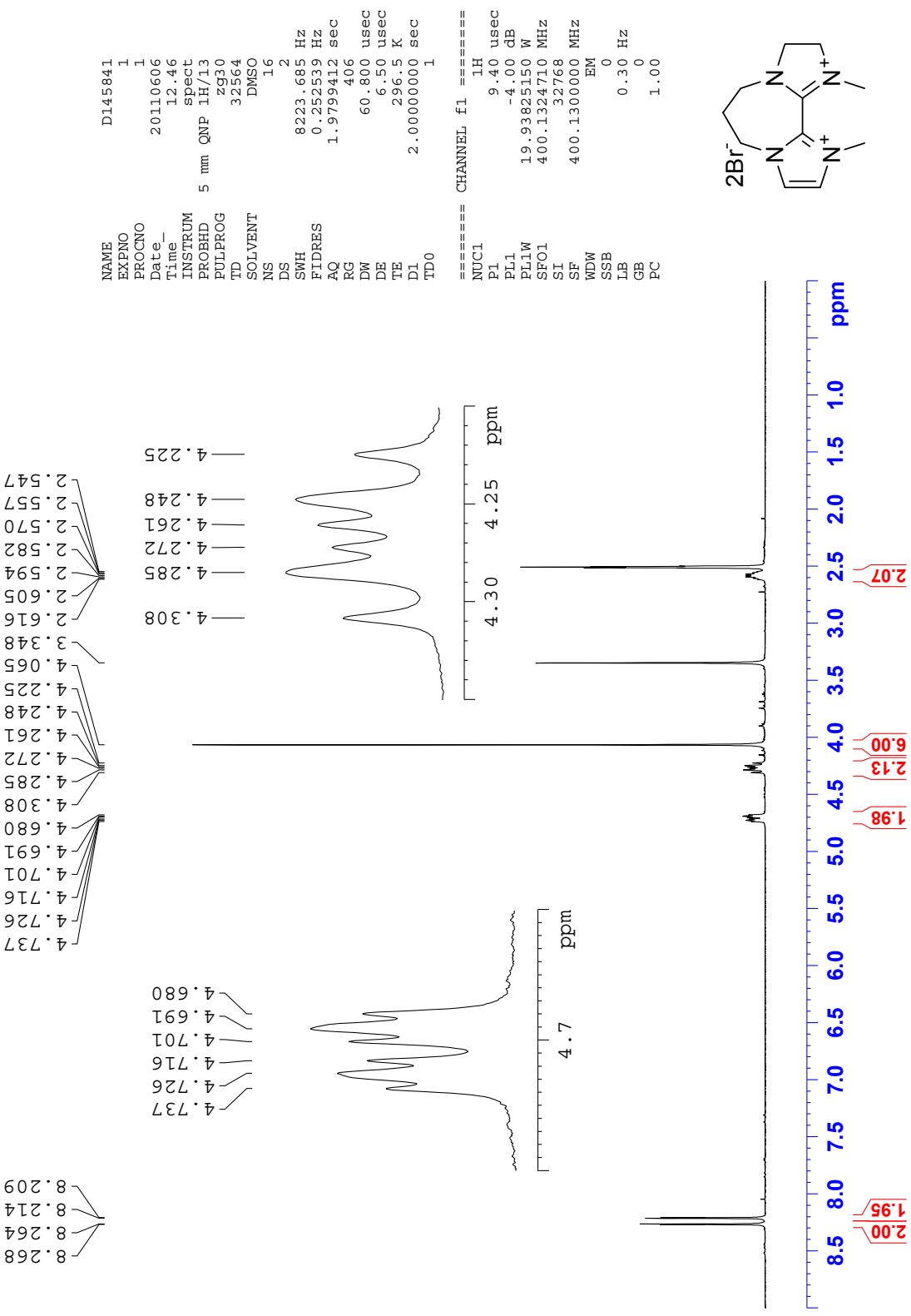


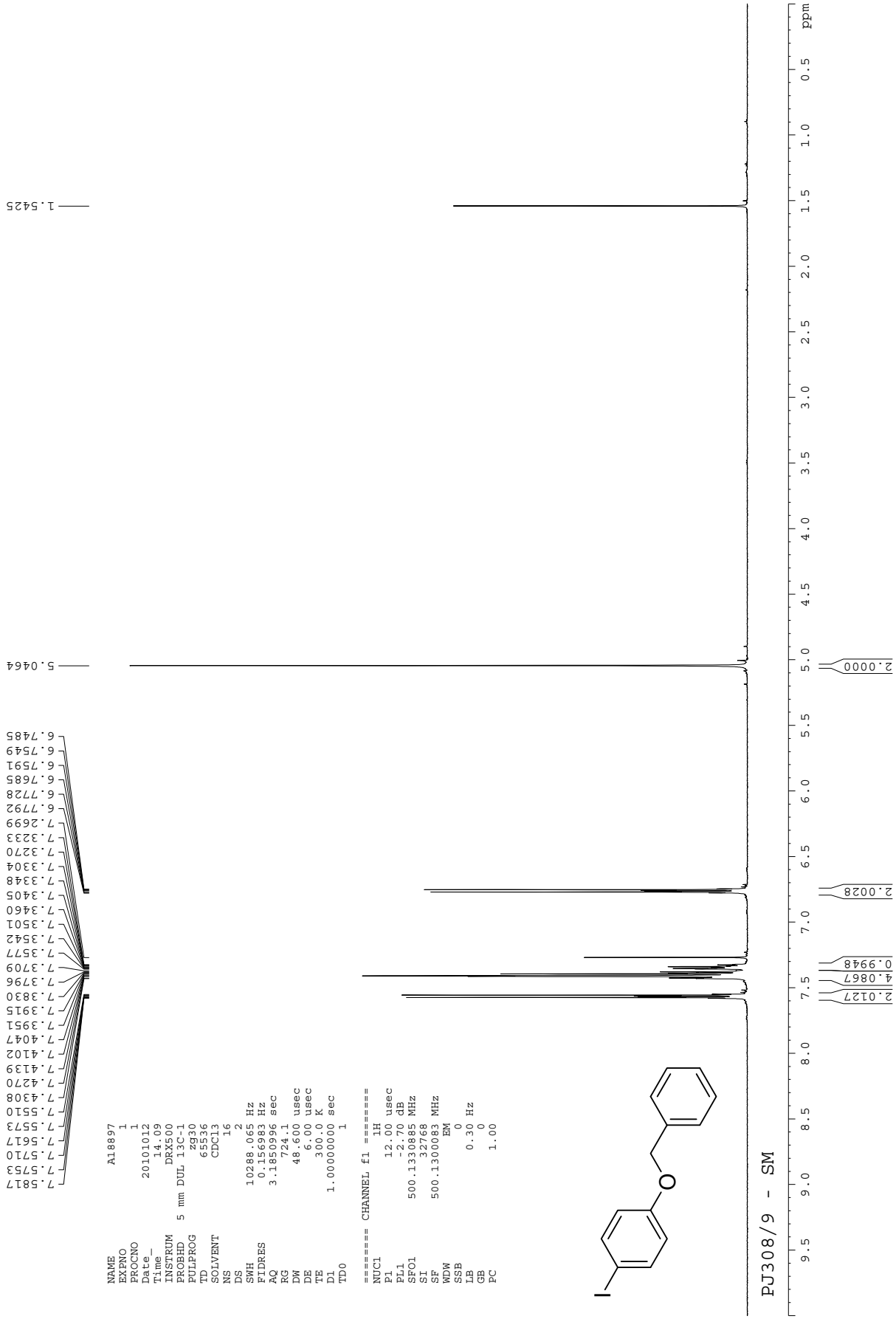
NAME	D150550
EXPNO	2
PROCNO	1
Date_	20111003
Time	19.06
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	16384
SOLVENT	DMSO
NS	1024
DS	4
SWH	22058.824 Hz
FIDRES	1.346364 Hz
AQ	0.3714207 sec
RG	2050
DW	22.667 usec
DE	6.50 usec
TE	300.0 K
D1	0.69999999 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	7.00 usec
PL1	-2.60 dB
PL1W	61.34123230 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.00 dB
PL12	14.06 dB
PL13	120.00 dB
PL2W	19.93825150 W
PL12W	0.31166428 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6128193 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

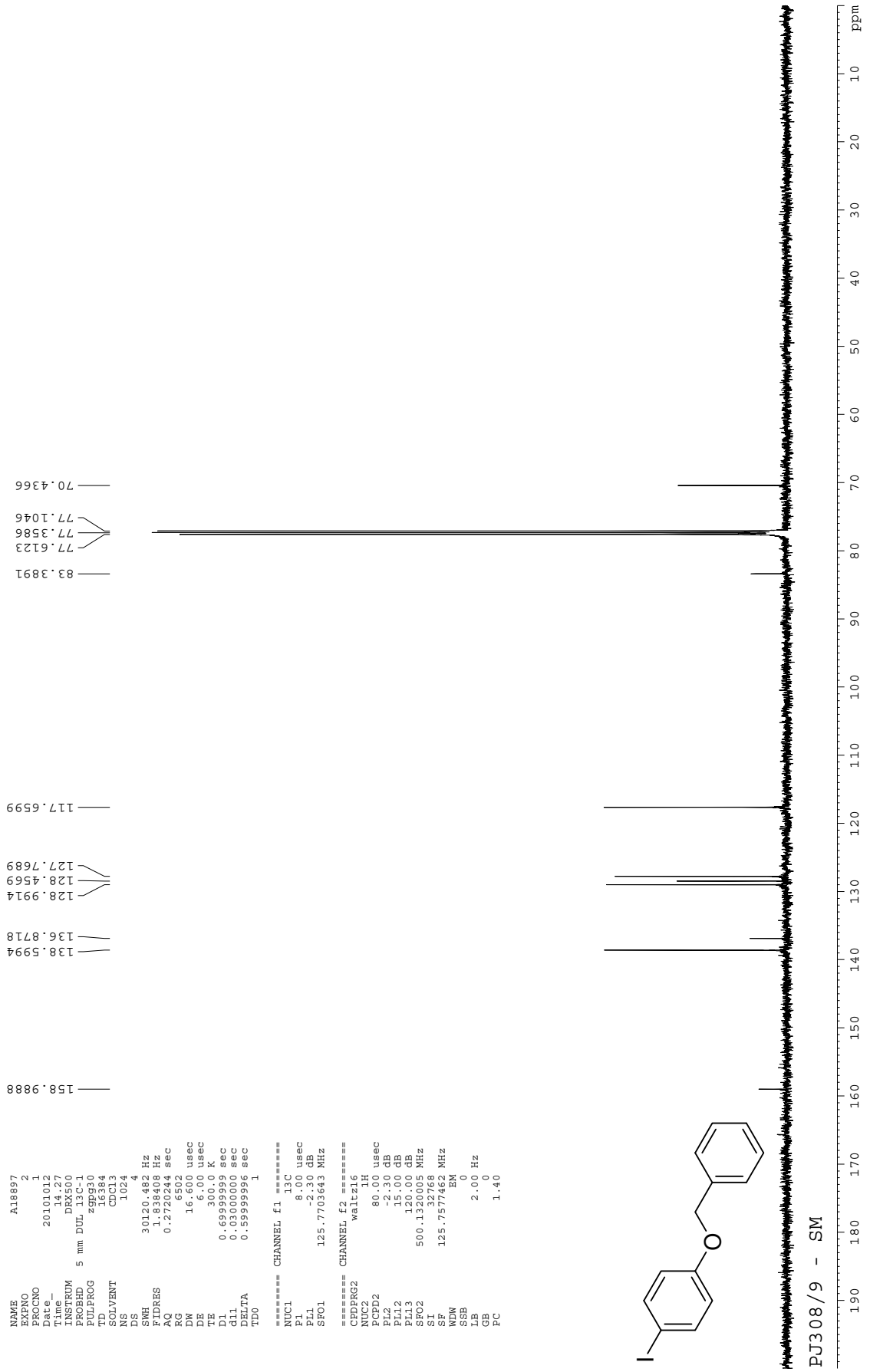


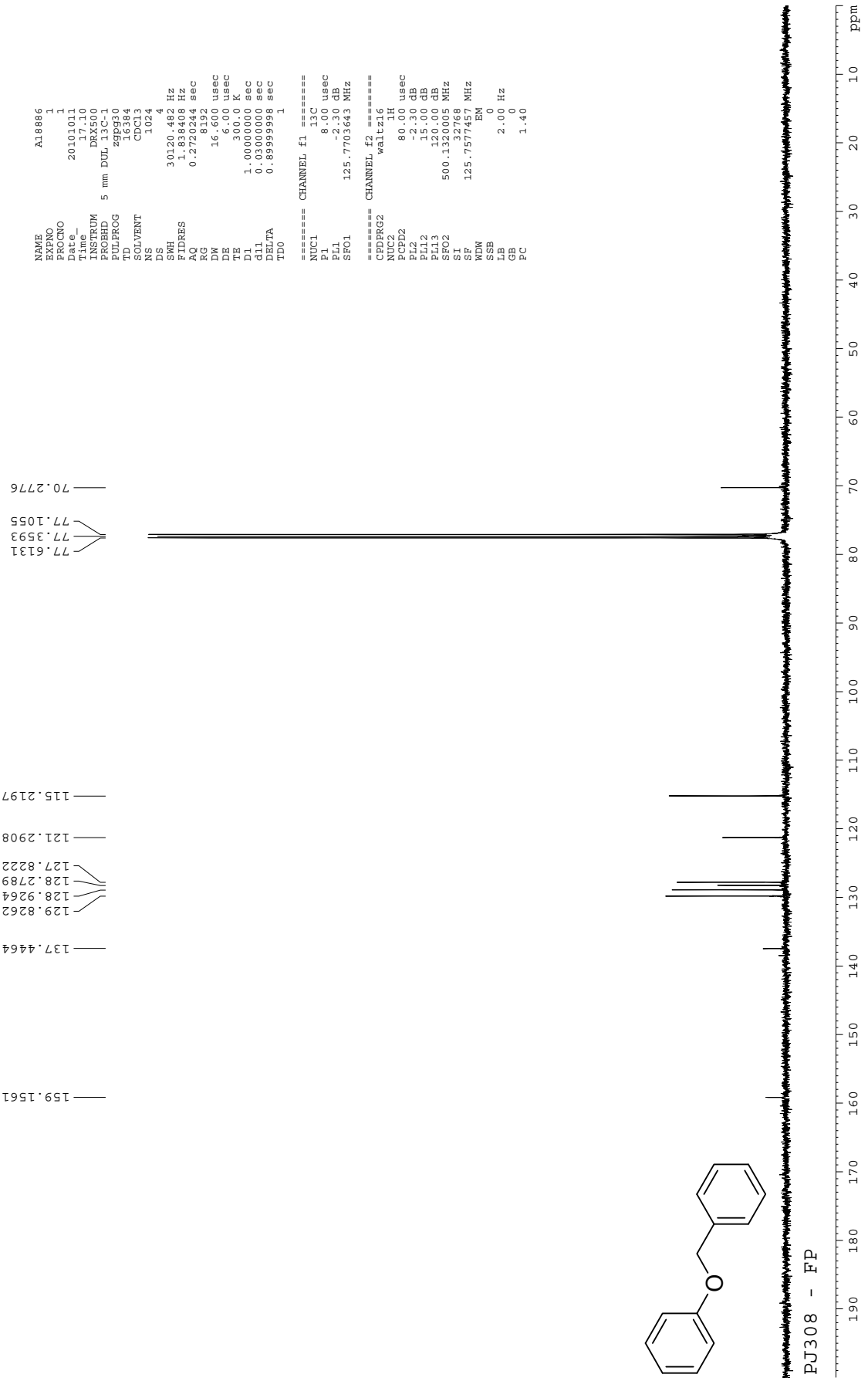


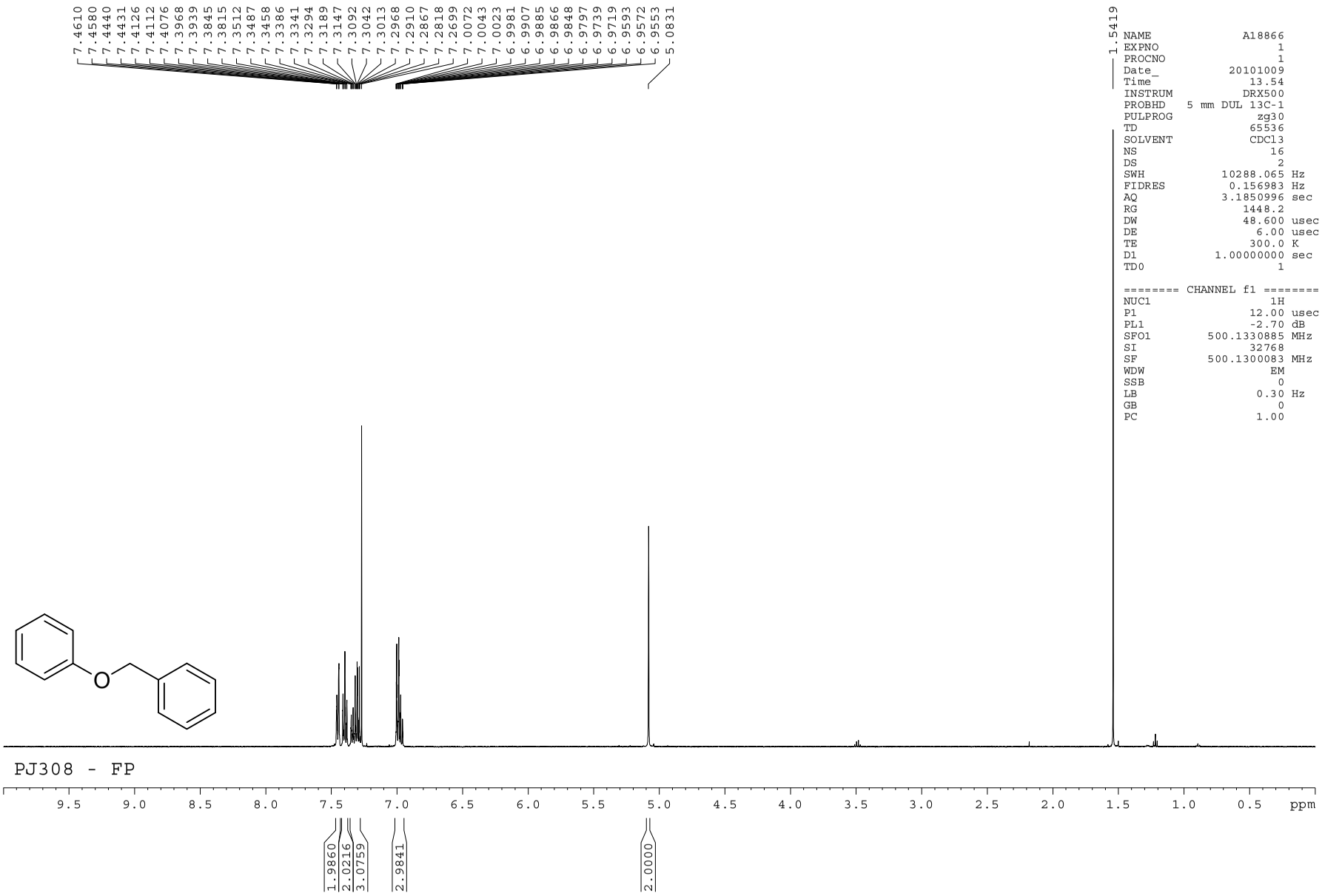
NAME	D145225	
EXPNO	1	
PROCNO	1	
Date_	20110518	
Time	15.04	
INSTRUM	spect	
PROBHD	5 mm QNP 1H/13	
PULPROG	zg30	
TD	32564	
SOLVENT	C6D6	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.252539	Hz
AQ	1.9799412	sec
RG	80.6	
DW	60.800	usec
DE	6.50	usec
TE	300.0	K
D1	2.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.40	usec
PL1	-4.00	dB
PL1W	19.93825150	W
SFO1	400.1324710	MHz
SI	32768	
SF	400.1300000	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

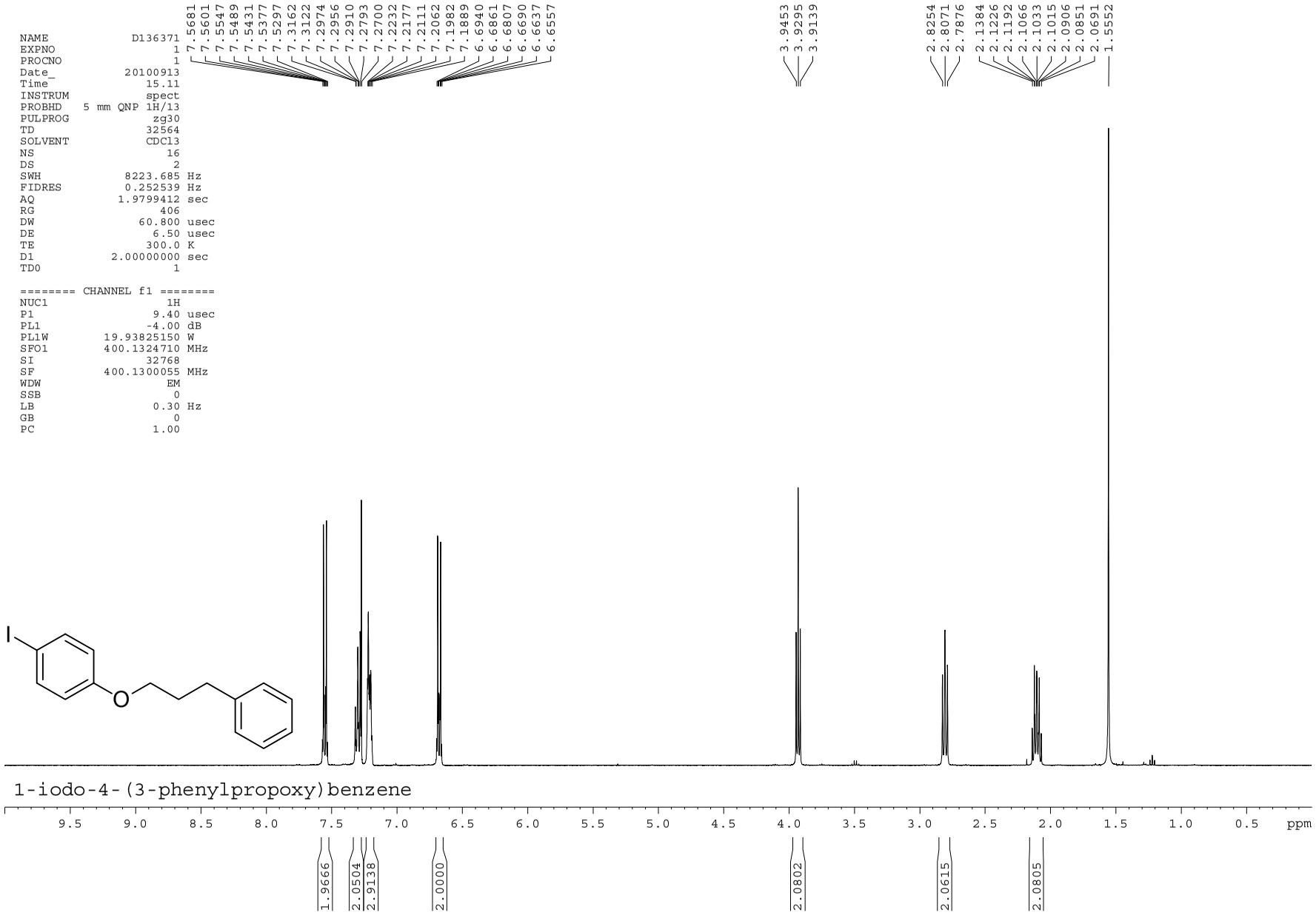

NAME D145841
EXPNO 1
PROCNO 1
Date_ 20110606
Time_ 12.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32564
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.252539 Hz
AQ 1.9799412 sec
RG 406
DW 60.800 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.40 usec
PL1 -4.00 dB
PL1W 19.93825150 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2Br-
ppm
2.07
6.00
2.13
1.98
2.00
1.95
4.225
4.248
4.261
4.272
4.285
4.308
4.30
4.25
ppm
4.737
4.726
4.716
4.701
4.691
4.680
4.7
ppm
2.547
2.557
2.570
2.582
2.594
2.605
2.616
3.348
4.065
4.225
4.248
4.261
4.272
4.285
4.308
4.680
4.691
4.701
4.716
4.726
4.737
8.209
8.214
8.264
8.268

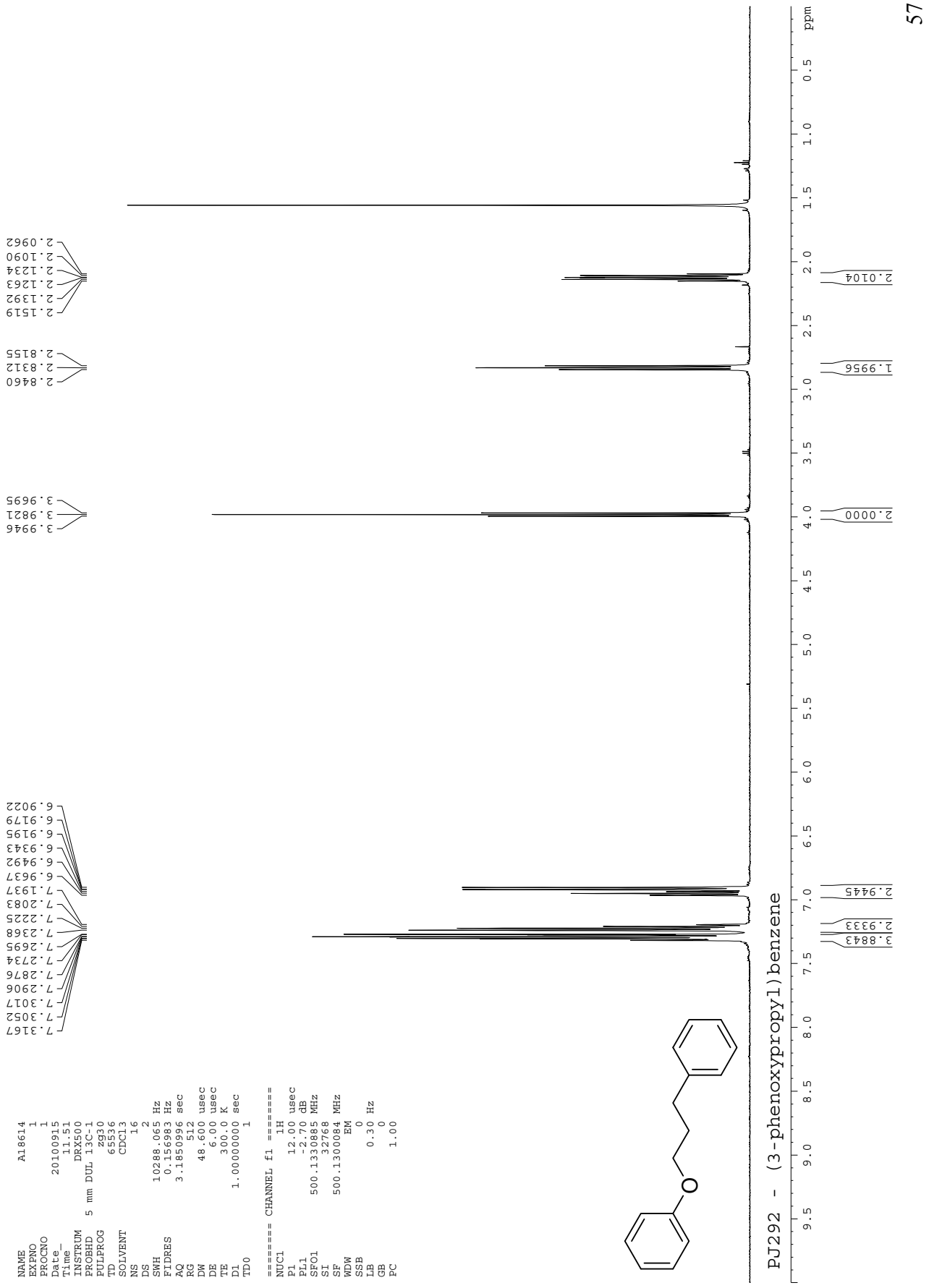


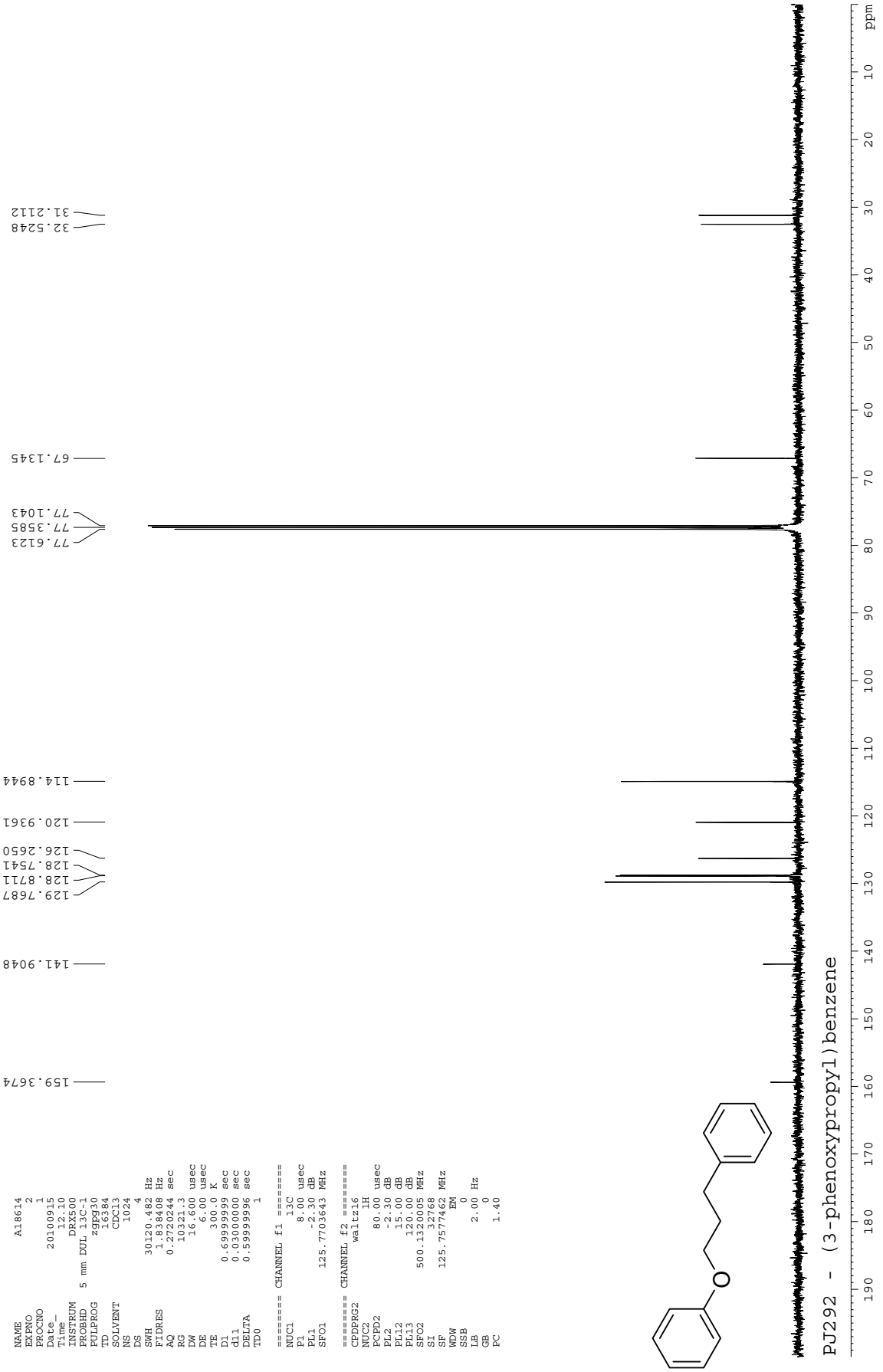

158.9888
138.5994
136.8718
128.9914
128.4569
127.7689
117.6599
83.3891
77.6123
77.3586
77.1046
70.4366
PJ308/9 - SM
ppm


NAME A18886
EXPNO 1
PROCNO 1
Date_ 20101011
Time_ 17.10
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 15384
SOLVENT CDCl3
NS 1024
DS 4
SWH 30120.482 Hz
FIDRES 1.834408 Hz
AQ 0.2720244 sec
RG 8192
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -2.30 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 15.00 dB
PL13 120.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577457 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
159.1561
137.4464
129.8262
128.9264
128.2789
127.8222
121.2908
115.2197
77.6131
77.3593
77.1055
70.2776
PJ308 - FP
ppm








PJ292 - (3-phenoxypropyl)benzene
159.3674
141.9048
129.7687
128.8711
128.7541
126.2650
120.9361
114.8944
77.6123
77.3585
77.1043
67.1345
32.5248
31.2112
ppm