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Isoxazole to Oxazole: a Mild and Unexpected Transformation

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Supporting Information

Contents

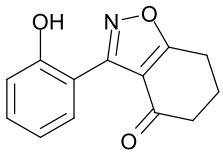
General directions

Commercially available reagents and solvents were used throughout without further purification, except tetrahydrofuran (THF) (distilled from benzophenone/Na). Light petroleum refers to the fraction with b.p 40-60 °C. Thin layer chromatography was carried out on Merck Kieselgel 60 GF254 coated onto aluminium foil-backed plates. The plates were visualized under UV light. Flash column chromatography was carried out using Merck Kieselgel 60 H silica or Matrix silica 60, with eluent as specified. IR spectra were recorded using a Perkin Elmer FTIR Spectrometer (Paragon 100) either neat, or as solutions using dichloromethane as solvent or using attenuated total reflectance (ATR) for solids. ^{1}H and ^{13}C NMR spectra were recorded using a Bruker 400 MHz NMR spectrometer (frequencies ^{1}H 400 MHz and ^{13}C 100 MHz). Chemical shifts δ are quoted in ppm and coupling constants *J* are quoted in Hz; d-chloroform was used as solvent throughout unless otherwise stated. In the ^{13}C spectra, signals corresponding to C, CH, CH_2 or CH_3 groups are noted, as assigned from either DEPT or HMQC experiments. Quaternary carbons were assigned using a combination of NMR techniques including COSY & HMQC unless otherwise stated. Spectra were calibrated to residual solvent peaks. High resolution mass spectra were recorded on a Thermofisher Exactive (Orbi) high resolution mass spectrometer. Melting points were recorded on a Stuart Scientific apparatus and are uncorrected. Unless otherwise stated all air sensitive reactions were carried out under an atmosphere of nitrogen.

General Procedure for formation of Benzisoxazoles 4

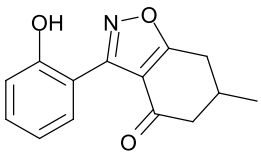
Sodium isopropoxide (2 mMol) was added to a solution of dione (2 mMol) in isopropanol (10 mL) at room temperature and left to stir for 10 minutes. Afterwards this solution was added very slowly to a solution of benzimidoyl chloride (1mMol) in isopropanol (10 mL) at 0°C and left to stir for 4 hours at room temperature under an atmosphere of nitrogen. Following this, the reaction mixture was concentrated under reduced pressure. The crude mixture was then dissolved in either ethyl acetate or dichloromethane (20 mL) and washed with water (2 x 20 mL) and once with saturated sodium chloride solution (20 mL), dried over magnesium sulfate and concentrated under reduced pressure to yield crude benzisoxazole **4**. This was used without purification, recrystallized from a mixture of ethyl acetate and petrol or purified via column chromatography using light petroleum: ethyl acetate (1:1 v/v) as a solvent.

3-(2-Hydroxyphenyl)-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4a



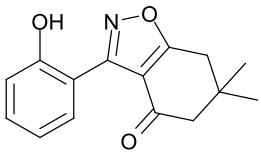
Pale yellow solid (94%), m.p. 108-110 °C; HRMS (EI$^+$) [M+Na]$^+$ 252.0623, $C_{13}H_{11}NO_3$ requires [M+Na]$^+$ 252.0631; ν_{max} (ATR)/cm^{-1} 1679 (C=O), 3136 (OH); δ_H (400MHz; $CDCl_3$) 2.28 (2H, dt, J = 6.4, 12.4, CH_2), 2.67 (2H, t, *J* = 6.4, $OCCH_2$), 3.10 (2H, t, *J* = 12.4, $O{=}CCH_2$), 7.01-7.08 (2H, m, Ar-H), 7.36-7.41 (1H, m, Ar-H,) 8.60 (1H, dd, *J* = 1.60, 8.00, Ar-H), OH not observed; δ_C (100MHz; $CDCl_3$) 21.6, 23.4 (CH_2), 39.0 ($O{=}C\mathit{C}H_2$), 112.9, 114.8 (C), 117.5, 119.9, 131.9, 132.5 (Ar-CH), 156.6, 159.5, 181.6 (C), 192.2 (CO).

3-(2-Hydroxyphenyl)-6-methyl-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4b



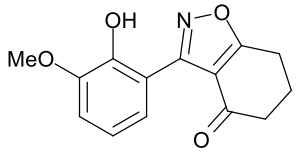
Pale yellow solid (83%), m.p. 121-122 °C; HRMS (EI$^+$) [M+Na]$^+$ 266.0782, $C_{14}H_{13}NO_3$ requires [M+Na]$^+$ 266.0788; ν_{max} (ATR)/cm^{-1} 1689 (C=O), 3078 (OH); δ_H (400MHz; $CDCl_3$) 1.24-1.26 (3H, d, *J* = 6.8, CH_3), 2.38-2.42 (1H, m, C*H*H), 2.45-2.59 (1H, m, CH), 2.70-2.76 (2H, m, CH_2), 3.20-3.26 (1H, dd, *J* = 5.2, 17.2, C*H*H), 6.99-7.08 (2H, m, Ar-H), 7.35-7.41 (1H, m, Ar-H), 8.62-8.65 (1H, dd, *J* = 2.0, 8.0, Ar-H), 9.49 (1H, s, Ar-OH); δ_C (100MHz; $CDCl_3$) 20.7 (CH_3), 29.9 (CH), 31.1, 47.4 (CH_2), 112.8, 114.6 (C), 117.5, 119.8, 131.9, 132.5 (Ar-CH), 156.6, 159.5, 181.5 (C), 191.7 (CO).

3-(2-Hydroxyphenyl)-6,6-dimethyl-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4c



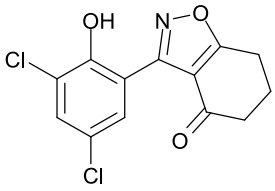
Clear crystals (78%), m.p. 111-113 °C; HRMS (EI$^+$) [M+Na]$^+$ 280.0946, $C_{15}H_{15}NO_3$ requires [M+Na]$^+$ 280.0944; ν_{max} (ATR)/cm^{-1} 1685 (C=O), 3103 (OH); δ_H (400MHz; $CDCl_3$) 1.25 (6H, s, 2 x CH_3), 2.58 (2H, s, $OCCH_2$), 2.98 (2H, s, $O{=}CCH_2$), 7.04-7.12 (2H, m, Ar-H), 7.39-7.43 (1H, m, Ar-H,) 8.67-8.69 (1H, dd, *J* = 1.6, 8.0, Ar-H), OH not observed; δ_C (100MHz; $CDCl_3$) 28.1 (2 x CH_3), 34.8 ($\mathit{C}CH_3$), 36.9, 53.5 (CH_2), 112.8, 113.8 (C), 117.5, 119.8, 131.9, 132.5 (Ar-CH), 156.7, 159.5, 181.1 (C), 191.5 (CO).

3-(2-Hydroxy-3-methoxyphenyl)-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4d



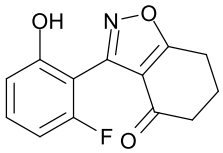
Beige solid (50%), m.p. 112-114 °C; HRMS (EI^+) $[M+Na]^+$ 282.0732, $C_{14}H_{13}NO_4$ requires $[M+Na]^+$ 282.0737; ν_{max} (DCM)/cm^{-1} 1692 (C=O), 3521 (OH); δ_H (400MHz; $CDCl_3$) 2.29-2.32 (2H, m, CH_2), 2.66 (2H, t, *J* = 6.0, $OCCH_2$), 3.13 (2H, t, *J* = 6.4, $O{=}CCH_2$), 3.95 (3H, s, OCH_3), 6.99-7.03 (2H, m, Ar-H), 7.88 (1H, d, *J* = 6.0, Ar-H), 8.55 (1H, br s, Ar-OH); δ_C (100MHz; $CDCl_3$) 21.7, 23.3, 38.7 (CH_2), 56.1 (OCH_3), 113.6 (C), 113.8, 119.6, 123.2 (Ar-CH), 145.9, 148.2, 158.6, 181.6 (C), 192.2 (CO).

3-(3,5-dichloro-2-hydroxyphenyl)-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4g



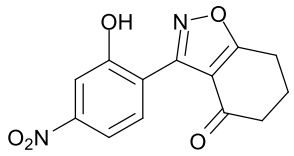
Brown solid (19%), decomp. 133-135 °C; ν_{max} (DCM)/cm^{-1} 1692 (C=O), 3690 (OH); δ_H (400MHz; d_6-DMSO) 2.16-2.19 (2H, m, CH_2), 2.47-2.52 (2H, m, $OCCH_2$), 3.14 (2H, t, *J* = 6.0, $O{=}CCH_2$), 7.41, 7.71 (each 1H, d, *J* = 2.4, Ar-H,) 10.08 (1H, br s, Ar-OH); δ_C (100MHz; $CDCl_3$) 21.6, 23.4, 38.9 (CH_2), 114.7, 115.8, 123.1, 124.6 (C), 130.0 (Ar-CH), 132.4 (Ar-CH), 151.4, 158.3, 182.1 (C), 192.4 (CO).

3-(2-Fluoro-6-hydroxyphenyl)-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one 4h



White crystals (47%), m.p. 133-135 °C; HRMS (EI^+) MH^+ 248.0716, $C_{13}H_{10}NO_3F$ requires MH^+ 248.0717; ν_{max} (DCM)/cm^{-1} 1692 (C=O), 3690 (OH); δ_H (400MHz; $CDCl_3$) 2.32 (2H, quin, *J* = 6.4, CH_2), 2.70 (2H, t, *J* = 6.4, $OCCH_2$), 3.15 (2H, t, *J* = 6.4, $O{=}CCH_2$), 6.85 (1H, td, *J* = 8.4, 5.2, Ar-H), 7.21-7.26 (1H, m, Ar-H), 8.39 (1H, dt, *J* = 8.0, 1.6, Ar-H), 9.49 (1H, br s, Ar-OH); δ_C (100MHz; $CDCl_3$) 21.6, 23.3, 38.9 (CH_2), 114.9 (C), 115.3 (d, $^4J_{CF}$ = 3, C), 118.7 (d, $^2J_{CF}$ = 18, Ar-CH), 119.6 (d, $^3J_{CF}$ = 7, Ar-CH), 126.9 (d, $^4J_{CF}$ = 4, Ar-CH), 145.2 (d, $^2J_{CF}$= 13, Ar-C), 152.1 (d, $^1J_{CF}$ = 240, Ar-CF), 158.1 (d, $^3J_{CF}$ = 3, C), 181.9 (C), 192.4 (CO).

3-(2-Hydroxy-4-nitrophenyl)-6,7-dihydrobenzo[d]isoxazol-4(5*H*)-one 4i

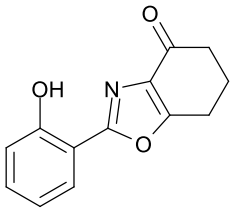


Pale yellow solid (23%), m.p. 159-161 °C; HRMS (EI^+) MH^+ 275.0659, $C_{13}H_{10}N_2O_5$ requires MH^+ 275.0662; ν_{max} (DCM)/cm^{-1} 1342 (NO_2) 1691 (C=O), 3689 (OH); δ_H (400MHz; $CDCl_3$) 2.33-2.40 (2H, m, CH_2), 2.77 (2H, t, *J* = 6.0 Hz, $OCCH_2$), 3.19 (2H, t, *J* = 6.4 $O=CCH_2$), 7.17 (1H, d, *J* = 8.8, Ar-H), 8.30 (1H, dd, *J* = 2.8, 8.8, Ar-H,) 10.01 (1H, d, *J* = 2.80, Ar-H), 10.50 (1H, br s, Ar-OH); δ_C (100MHz; $CDCl_3$) 21.6, 23.3, 38.8 (CH_2), 112.6, 113.5 (C), 118.1, 127.8, 128.9 (Ar-CH), 139.8, 158.7, 162.1, 182.1 (C), 192.8 (CO).

General Procedure for rearrangement to Benzoxazoles 6

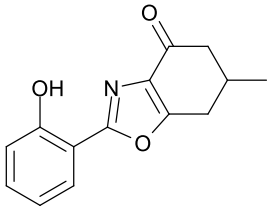
Benzisoxazole **4** (4.37 mMol) and caesium carbonate (4.37 mMol) in dry THF (30 mL) were heated under reflux for the stated period of time (see Table 2). The reaction mixture was then cooled to room temperature and hydrochloric acid (2M; 10 mL) and dichloromethane (25 mL) or ethyl acetate (25 mL) was added. The mixture was then separated and the organic layer washed with water (25 mL) and saturated sodium chloride solution (25 mL), dried over magnesium sulfate and concentrated under reduced pressure to yield crude benzoxazole **6** which was used without purification, or purified via column chromatography eluting with light petroleum:ethyl acetate (1:1 v/v).

2-(2-Hydroxyphenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6a



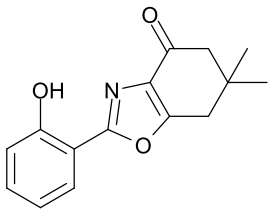
Beige solid (87%), decomp. 202-204 °C; HRMS (EI^+) MH^+ 230.0809, $C_{13}H_{11}O_3N$ requires MH^+ 230.0812; ν_{max} (DCM)/cm^{-1}) 1694 (C=O), 3408 (OH); δ_H (400MHz; $CDCl_3$) 2.20-2.27 (2H, m, CH_2), 2.57 (2H, t, *J* = 5.6, $OCCH_2$), 3.00 (2H, t, *J* = 6.0, $O=CCH_2$), 6.86-6.90 (1H, m, Ar-H), 7.10 (1H, dd, *J* = 0.8, 8.4, Ar-H), 7.30-7.34 (1H, m, Ar-H), 7.74 (1H, dd, *J* = 1.6, 8.0, Ar-H), 10.58 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 22.2 (2 x CH_2), 37.9 (CH_2), 110 (C), 117.6, 119.5, 126.3, 133.2 (Ar-CH), 133.7, 157.7, 161.2, 163.0 (C), 190.7 (CO).

2-(2-Hydroxyphenyl)-6-methyl-6,7-dihydrobenzo[d]oxazol-4(5H)-one 6b



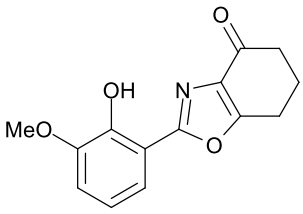
Beige solid (88%), m.p. 154-156 °C; HRMS (EI$^+$) MH$^+$ 244.0968, $C_{14}H_{13}O_3N$ requires MH$^+$ 244.09737; ν_{max} (ATR)/cm^{-1}) 1680 (C=O), 3187 (OH); δ_H (400MHz; $CDCl_3$) 1.20 (3H, d, *J* = 6.4, CH_3), 2.32 (1H, dd, *J* = 16.0, 11.2, CH), 2.49-2.70 (3H, m, 3 x CH), 3.07 (1H, dd, *J* = 4.8, 17.2, CH), 6.87 (1H, dd, *J* = 8.0, Ar-H), 7.00 (1H, d, 8.4, Ar-H), 7.30-7.34 (1H, m, Ar-H), 7.69 (1H, dd, *J* = 1.6, 8.0, Ar-H), 10.59 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 21.1 (CH_2), 29.9 (CH_3), 30.6 (CH), 46.4 (CH_2) 109.9 (C), 117.3, 119.5, 126.3, 133.1 (Ar-CH), 133.2, 157.4, 161.2, 162.8 (C), 190.3 (CO).

2-(2-Hydroxyphenyl)-6,6-dimethyl-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6c



Beige solid (82%), m.p. 125-127 °C; HRMS (EI$^+$) MH$^+$ 258.1125, $C_{15}H_{15}O_3N$ requires MH$^+$ 258.1125; ν_{max} (DCM)/cm^{-1}) 1694 (C=O), 3410 (OH); δ_H (400MHz; $CDCl_3$) 1.08 (6H, s, 2 x CH_3), 2.41 (2H, s, $OCCH_2$), 2.83 (2H, s, O=CCH_2) 6.85 (1H, t, *J* = 8.0, Ar-H), 6.97 (1H, t, *J* = 8.0, Ar-H), 7.29 (1H, t, *J* = 8.0, Ar-H), 7.69 (1H, d, *J* = 8.0, Ar-H), 10.54 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 27.6 (2 x CH_3), 34.5 (C) 34.9, 51.2 (CH_2) 109 (C), 116.4, 118.5, 125.3 (Ar-CH), 131.9 (C), 132.1 (Ar-CH), 156.5, 160.6, 161.2 (C), 189.2 (CO).

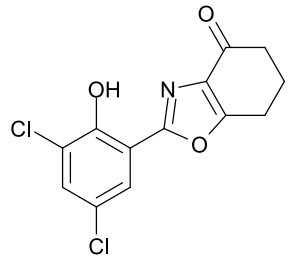
2-(2-Hydroxy-3-methoxyphenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6d



White solid (70%), m.p. 114-115 °C; HRMS (EI$^+$) [M+Na]$^+$ 282.0731, $C_{14}H_{13}NO_4$ requires [M+Na]$^+$ 282.0737; ν_{max} (DCM)/cm^{-1} 1693 (C=O), 3580 (OH); δ_H (400MHz; $CDCl_3$) 2.30 (2H, quin, *J* =6.4. CH_2), 2.63 (2H, t, *J* = 6.4, $OCCH_2$), 3.06 (2H, t, *J* = 6.4, O=CCH_2), 3.92 (3H, s, OCH_3), 6.89 (1H, t, *J* = 8.0, Ar-H), 6.98 (1H, d, *J* = 8.0, Ar-H), 7.41 (1H, d, *J* = 8.0, Ar-H), 10.79 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 21.1,

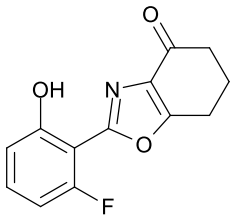
21.2, 36.9 (CH_2), 55.4 (OCH_3), 109.2 (C), 113.7, 116.8, 118.3 (Ar-CH), 132.6, 146.8, 147.6, 160.2, 161.9 (C), 189.6 (CO).

2-(3,5-Dichloro-2-hydroxyphenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6g



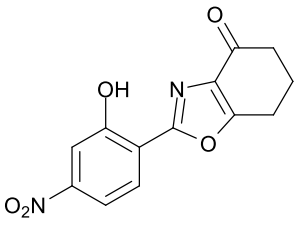
Brown solid (58%), decomp. 165-167 °C; HRMS (EI^+) MH^+ 298.0027, $C_{13}H_9NO_3Cl_2$ requires MH^+ 298.0032; ν_{max} (DCM)/cm^{-1} 1698 (C=O), 3691 (OH); δ_H (400MHz; $CDCl_3$) 2.32-.235 (2H, m, CH_2), 2.66 (2H, t, *J* = 6.4, $OCCH_2$), 3.11 (2H, t, *J* = 6.0, $O{=}CCH_2$), 7.46 (1H, d, *J* = 2.4, Ar-H), 7.70 (1H, d, *J* = 2.4, Ar-H), 11.20 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 22.1, 22.2, 37.9 (CH_2), 111.7, 123.4 (C), 124.2 (Ar-CH), 124.3 (C), 132.9 (Ar-CH), 133.7, 152.2, 159.5, 163.6 (C), 190.4 (CO).

2-(2-Fluoro-6-hydroxyphenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6h



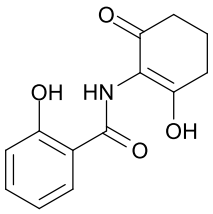
White solid (75%), m.p. 200-202 °C; HRMS (EI^+) MH^+ 248.0714, $C_{13}H_{10}O_3NF$ requires MH^+ 248.0717; ν_{max} (DCM)/cm^{-1}) 1684 (C=O), 3396 (OH); δ_H (400MHz; $CDCl_3$) 2.23-.236 (2H, m. CH_2), 2.67 (2H, t, *J* = 7.2, $OCCH_2$), 3.11 (2H, t, *J* = 6.4, $O{=}CCH_2$), 6.90 (1H, td, *J* = 8.0, 4.8 Hz, Ar-H), 7.18-7.23 (1H, m, Ar-H), 7.29 (1H, dt, *J* = 8.0, 1.20, Ar-H), 10.54 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 21.1, 21.2, 36.9 (CH_2), 111.3 (d, *J* = 4, C), 118.1 (d, *J* = 2, Ar-CH) 118.3 (d, *J* = 13, Ar-CH), 120.3 (d, *J* = 3, Ar-CH), 145.2 (d, *J* = 13, C), 150.6 (d, *J* = 244, Ar-CF), 159.4 (d, *J* = 4, C), 162.1 (C), 189.5 (CO).

2-(2-Hydroxy-4-nitrophenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 6i



Yellow solid (77%), m.p. 184-186 °C; HRMS (EI$^+$) $[M+Na]^+$ 275.0659, $C_{13}H_{10}N_2O_5$ requires $[M+Na]^+$ 275.0662; ν_{max} (DCM)/cm^{-1} 1344 (NO_2) 1698 (C=O), 3584 (OH); δ_H (400MHz; $CDCl_3$) 2.35 (2H, quin, J = 6.4, CH_2), 2.69 (2H, t, J = 6.4, $OCCH_2$), 3.14 (2H, t, J = 6.4, $O{=}CCH_2$), 7.19 (1H, d, J = 9.2, Ar-H), 8.28 (1H, dd, J = 9.2, 2.8, Ar-H), 8.78 (1H, d, J = 2.8, Ar-H), 11.50 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 21.1, 21.2, 36.9 (CH_2), 109.2 (C), 117.4, 121.8, 127.3 (Ar-CH), 132.7, 139.4, 158.4, 161.3, 162.6 (C), 189.3 (CO).

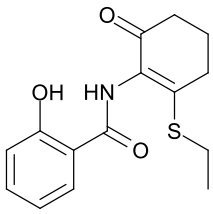
2-Hydroxy-N-(2-hydroxy-6-oxocyclohex-1-en-1-yl)benzamide 7a



3-(2-Hydroxyphenyl)-6,7-dihydrobenzo[*d*]isoxazol-4(5*H*)-one **4a** (2.18 mMol, 0.50 g) and caesium carbonate (4.36 mMol, 1.42 g) in ethanol (30 mL) was heated under reflux for 4 h. Hydrochloric acid (1M; 20 mL) and dichloromethane (50 mL) were added to the reaction mixture after it had cooled to 20 °C. The mixture was separated and the organic layer washed with water (50 mL) and saturated sodium chloride solution (50 mL), dried over magnesium sulfate and concentrated under reduced pressure to yield 2-hydroxy-N-(2-hydroxy-6-oxocyclohex-1-en-1-yl)benzamide **7a** as a beige solid (0.49 g, 91%), m.p. 172-174 °C; HRMS (EI$^+$) $[M+Na]^+$ 270.0729, $C_{13}H_{13}NO_4$ requires $[M+Na]^+$ 270.0737; ν_{max} (ATR)/cm^{-1} 1587 (CONH), 3177 (OH); δ_H (400MHz; $CDCl_3$) 2.06 (2H, quin, J = 6.4, CH_2), 2.56 (2H, t, J = 6.4, $OCCH_2$), 2.68 (2H, t, J = 6.4, $O{=}CCH_2$), 6.98 (1H, t, J = 7.6, Ar-H), 7.05 (1H, d, J = 8.4, Ar-H), 7.46-7.50 (1H, m, Ar-H), 7.67 (1H, d, J = 8.4, Ar-H), 9.48, 10.93 (each 1H, br s, OH), 12.88 (1H, br s, NH); δ_C (100MHz; $CDCl_3$) 20.3 (CH_2), 30.2(CH), 35.0 (CH_2), 113.4, 113.7 (C), 118.9, 119.8, 127.1, 135.2 (Ar-CH), 160.8, 165.9, 167.4 (C), 192.9 (CO). A sample was submitted for X-ray crystal structure determination, see Fig. 3 page 9.

Crystal data for **7a**: $C_{13}H_{13}NO_4$, M = 247.24, monoclinic, $P2_1/n$, a = 5.5001(6), b = 15.2463(18), c = 13.6680 (16) Å, β = 97.7388(18)°, V = 1135.7(2) Å^3, Z = 4, μ(Mo-Kα) = 0.11 mm^{-1}, 12997 reflections measured, 3446 unique, R_{int} = 0.026, R_1[for 2970 data with $F^2 > 2\sigma(F^2)$] = 0.045, $wR2$ (all data) = 0.131. Two-fold disorder at atom C(4); major component 85.5(4)%. CCDC 962974.

N-(2-(Ethylthio)-6-oxocyclohex-1-en-1-yl)-2-hydroxybenzamide 7b



3-(2-Hydroxyphenyl)-6,7-dihydrobenzo[d]isoxazol-4(5H)-one **4a** (2.18 mMol, 0.50 g), ethanethiol (2.18 mMol, 0.135 g) and caesium carbonate (2.18 mMol, 0.72 g) in dry THF (10 mL) was heated under reflux for 4 h. Hydrochloric acid (2M; 10 mL) and dichloromethane (10 mL) were added to the reaction mixture after it had cooled to 20 °C. The mixture was separated and the organic layer washed with water (10 mL) and saturated sodium chloride solution (10 mL), dried over magnesium sulfate and concentrated under reduced pressure to yield N-(2-(ethylthio)-6-oxocyclohex-1-en-1-yl)-2-hydroxybenzamide **7b** as colourless crystals (38.1 mg, 6%), decomp. 159-161 °C; HRMS (EI^+) $[M+Na]^+$ 314.0819, $C_{15}H_{17}O_3NS$ requires $[M+Na]^+$ 314.0821; ν_{max} (ATR)/cm^{-1} 1564 (CONH) 1628 (C=O), 3327 (OH); δ_H (400MHz; $CDCl_3$) 1.31 (3H, t, J = 7.2, CH_3), 2.14 (2H, quin, J = 6.4, CH_2), 2.55 (2H, t, J = 6.4, O=CCH_2), 2.82 (2H, t, J = 6.4, O=CCH_2), 2.90 (2H, q, J = 7.2, SCH_2), 6.84-6.89 (1H, m, Ar-H), 6.95 (1H, dd, J = 8.4, 1.2, Ar-H), 7.38-7.40 (1H, m, Ar-H), 7.59 (1H, dd, J = 8.0, 1.6, Ar-H), 8.06 (1H, br s, NH), 11.85 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 14.3 (CH_3), 22.0, 25.4, 29.5, 36.3 (CH_2), 114.1 (C), 118.5, 118.9 (Ar-CH), 126.3 (Ar-CH, C), 134.7 (Ar-CH), 159.0, 161.7, 168.0 (C), 191.2 (CO). A sample was submitted for X-ray crystal structure determination, see Fig. 4 below.

Crystal data for **7b**: $C_{15}H_{17}NO_3S$, M = 291.35, monoclinic, $P2_1/c$, a = 9.7309(7), b = 7.3003(5), c = 20.4979(14) Å, β = 91.2676(10)°, V = 1455.78(18) $Å^3$, Z = 4, μ(Mo-Kα) = 0.23 mm^{-1}, 16546 reflections measured, 4425 unique, R_{int} = 0.024, R_1[for 3740 data with $F^2 > 2\sigma(F^2)$] = 0.042, wR_2 (all data) = 0.121. CCDC 962975.

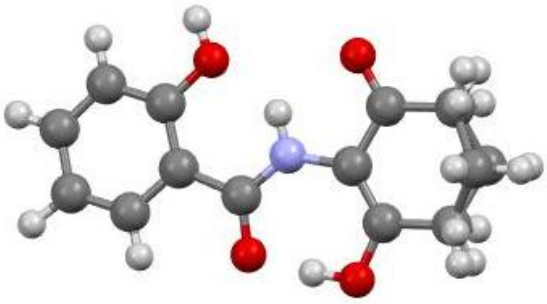


Figure 3: X-ray crystal structure of amide **7a**

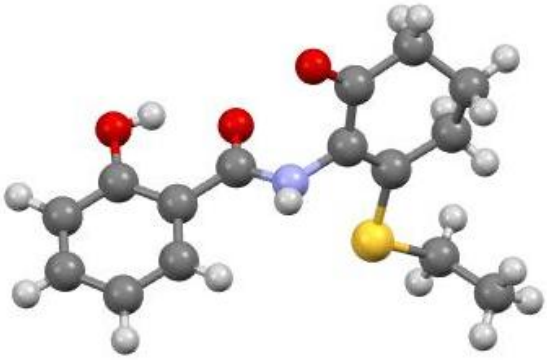
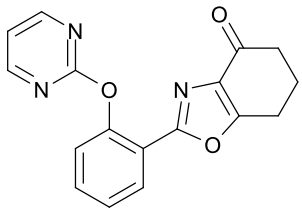


Figure 4: X-ray crystal structure of amide **7b**

2-(2-(Pyrimidin-2-yloxy)phenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one 8



2-(2-Hydroxyphenyl)-6,7-dihydrobenzo[d]oxazol-4(5*H*)-one **6a** (2.18 mMol, 0.50 g), 2-chloropyrimidine (6.54 mMol, 0.75 g), copper powder (2.18 mMol, 0.14 g) and caesium carbonate (2.18 mMol, 0.72 g) in dry DMF (25 mL) was heated under reflux for 18 h. Dichloromethane (50 mL) was added to the reaction mixture after it had cooled to 20 °C. The mixture was separated and the organic layer washed with water (2 x 25mL) and saturated sodium chloride solution (25 mL), dried over magnesium sulfate and concentrated under reduced pressure. The residue was purified via flash column chromatography (eluent light petroleum:ethyl acetate, 1:1 v/v) to yield 2-(2-(pyrimidin-2-yloxy)phenyl)-6,7-dihydrobenzo[*d*]oxazol-4(5*H*)-one as intense yellow solid (0.315 g, 47%), m.p. 178-180 °C; HRMS (EI$^+$) $[M+Na]^+$ 330.0846, $C_{17}H_{13}O_3N_3$ requires $[M+Na]^+$ 330.0849; ν_{max} (DCM)/cm^{-1} 1692 (C=O); δ_H (400MHz; $CDCl_3$) 2.17 (2H, quin, *J* = 6.4, CH_2), 2.55 (2H, t, *J* = 6.4, O=CCH_2), 2.80 (2H, t, *J* = 6.4, O=CCH_2), 7.04 (1H, t, *J* = 4.8, Ar-H), 7.31 (1H, dd, *J* = 8.4, 1.2, Ar-H), 7.38-7.42 (1H, m, Ar-H), 7.56-7.60 (1H, m, Ar-H), 8.30 (1H, dd, *J* = 8.0, 1.6, Ar-H), 8.55 (1H, d, *J* = 4.8 Ar-H), 11.85 (1H, br s, OH); δ_C (100MHz; $CDCl_3$) 22.1, 22.2, 37.9 (CH_2), 116.1 (Ar-CH), 120.0 (C), 124.0, 126.2, 130.5, 132.5 (Ar-CH, C), 134.8, 159.3, 159.7 (C), 164.0 (Ar-CH), 165.6 (C), 191.4 (CO).

AJC_ACOPI3
(Jones) Alex Chatterley

Current Data Parameters
NAME Nov12-2013
EXPNO 200

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7.080
7.062
7.059
7.055
7.040
7.038
7.035
7.020
7.017
3.080
3.064
3.048
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2.650
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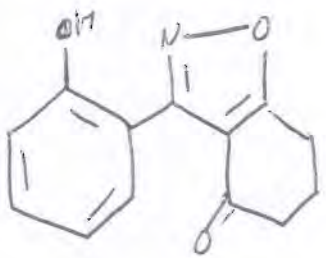
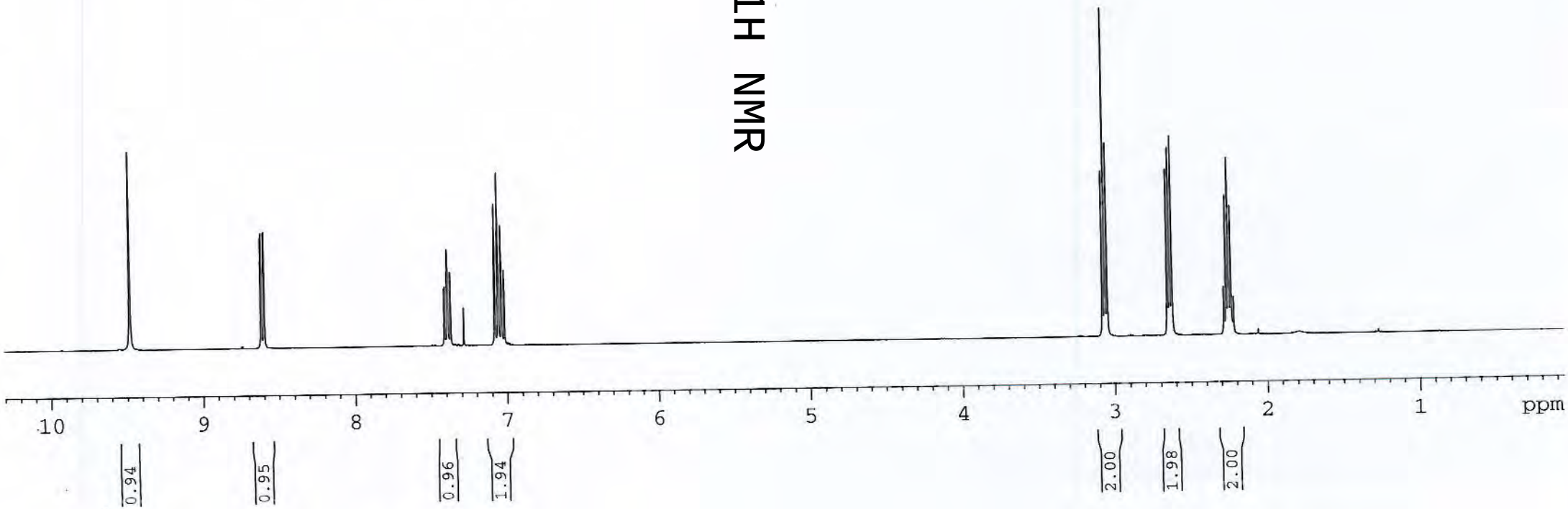
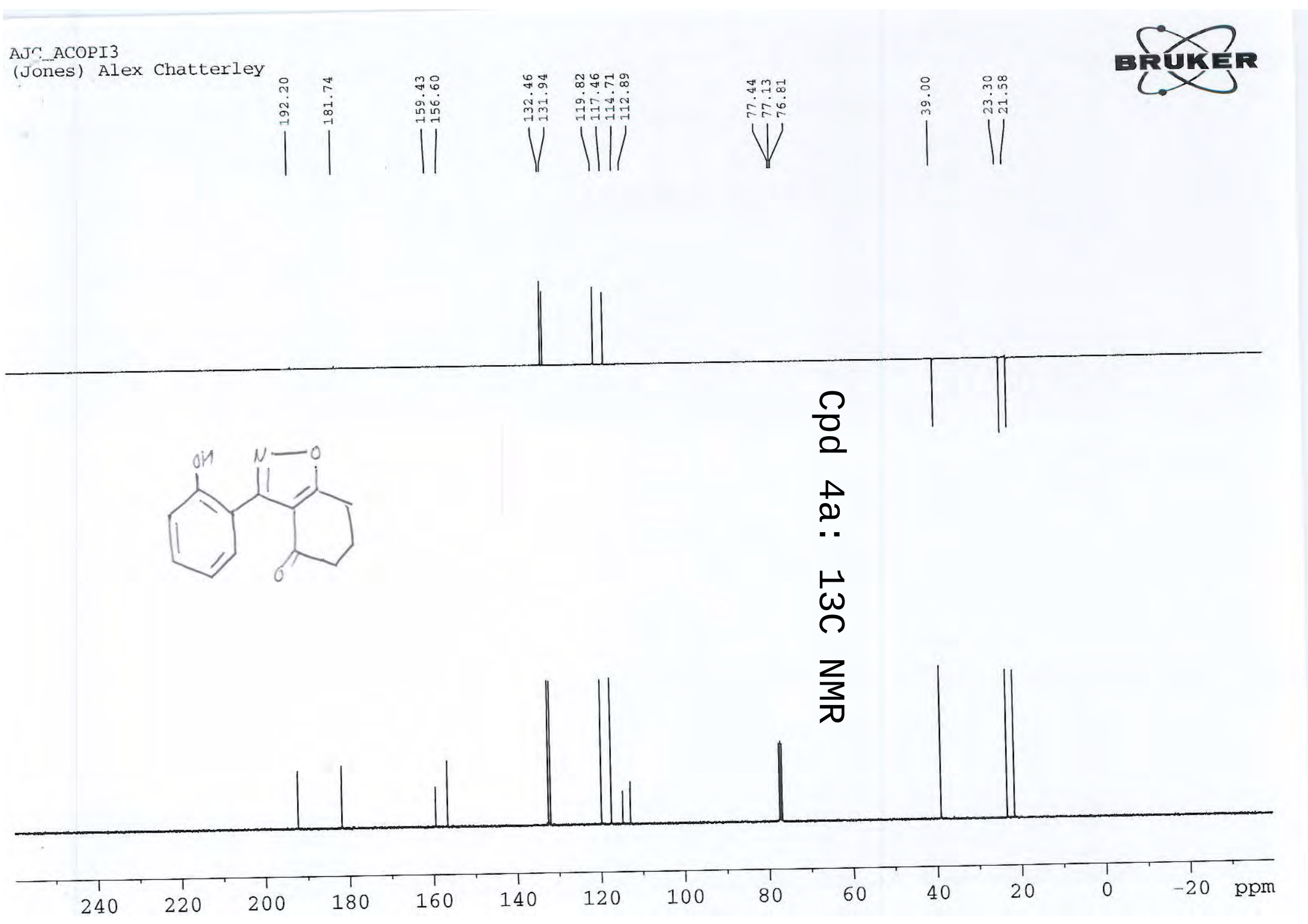
2.220



Cpd 4a: 1H NMR

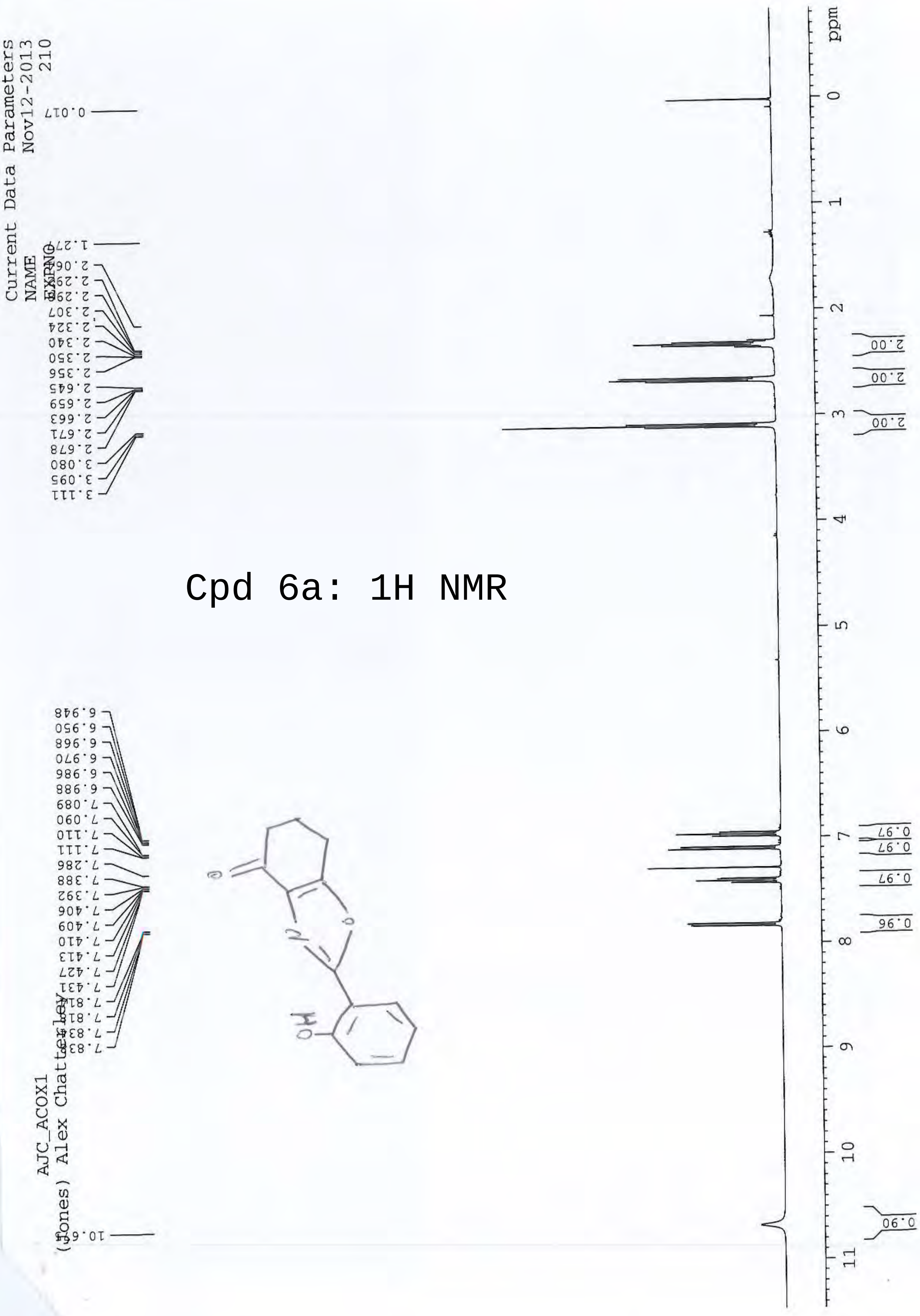


AJC_ACOPI3
(Jones) Alex Chatterley



Cpd 4a: 13C NMR

Cpd 6a: 1H NMR



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EXPNO 210
Nov12-2013
(Jones) Alex Chatterley
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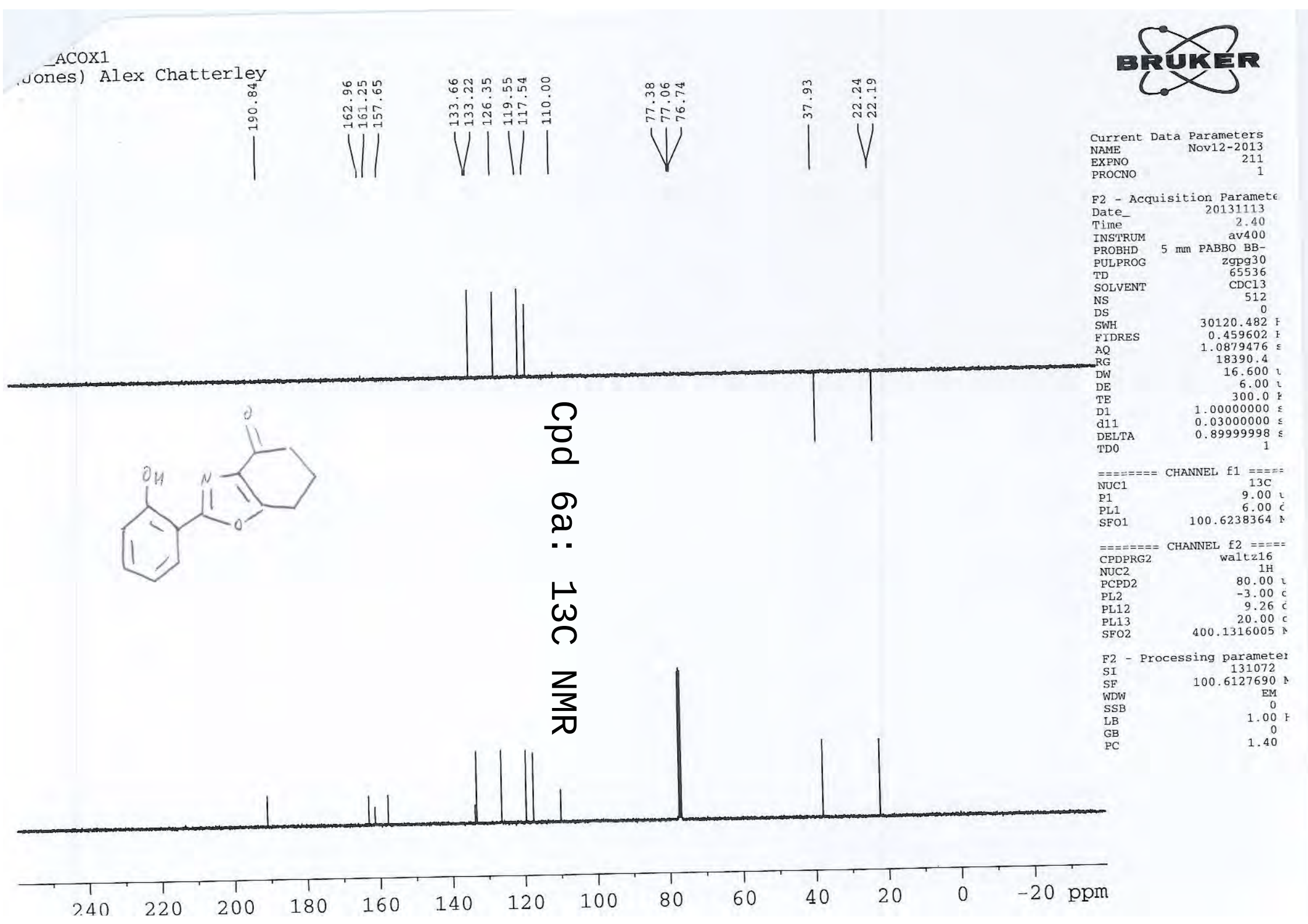
Jones) Alex Chatterley



BRUKER

Current Data Parameters
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PROCNO 1

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SOLVENT CDCl3
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SWH 30120.482 H
FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

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P1 9.00 u
PL1 6.00 d
SFO1 100.6238364 M

======== CHANNEL f2 =====
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PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127690 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

Cpd 4b: 1H NMR

AJC_AC431PFA_7 JUN 2013_Proton_ft-1-4.jdf
X : parts per Million : Proton
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8.606
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7.388
7.371
7.354
7.349
7.247
7.065
7.062
7.041
7.021
7.002
7.000
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3.230
3.220
3.218
3.189
3.176
3.174
2.741
2.716
2.698
2.426
2.386
1.598
1.239
1.223
-0.014
1.04
1.00
1.03
2.06
1.07
2.15
1.06
1.05
0.64
3.26

Cpd 4b: 13C NMR
AJC_AC431PFA_ 7 JUN 2013_DEPT135deg_ft-1-1.jdf
abundance
X : parts per Million : Carbon13
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117.572
119.918
131.979
132.580
AJC_AC431PFA_ 7 JUN 2013_Carbon_ft-1-2.jdf
abundance
X : parts per Million : Carbon13
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31.170
47.512
76.793
77.117
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117.572
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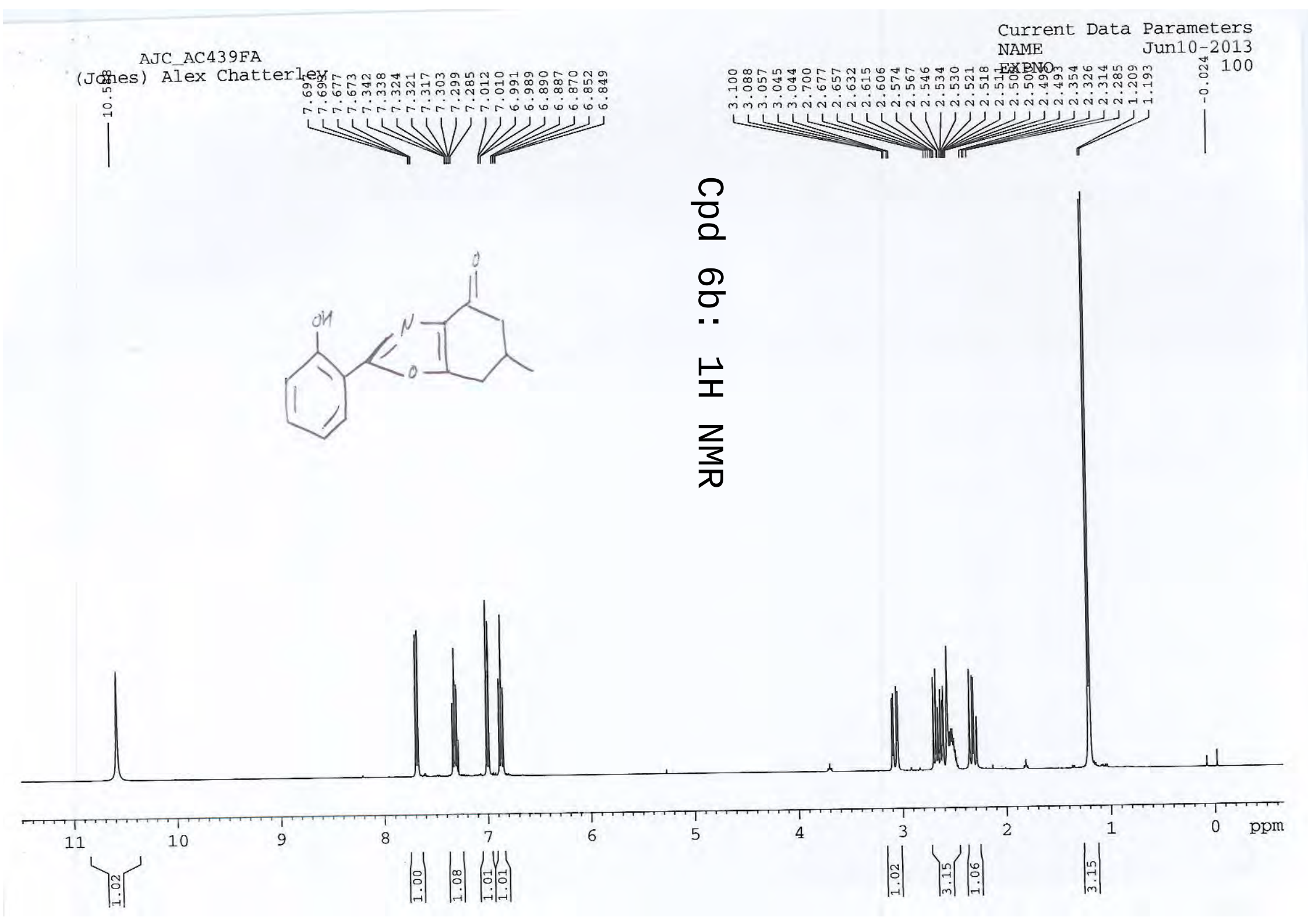
159.553
181.597
191.789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(Jones) Alex Chatterley
Current Data Parameters
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1.209
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O
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Cpd 6b: 1H NMR
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10
9
8
7
6
5
4
3
2
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0
ppm
1.02
1.00
1.08
1.01
1.01
1.02
3.15
1.06
3.15

AJC_AC439FA
(Jones) Alex Chatterley

190.33
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133.09
132.85
132.79
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126.02
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110.01
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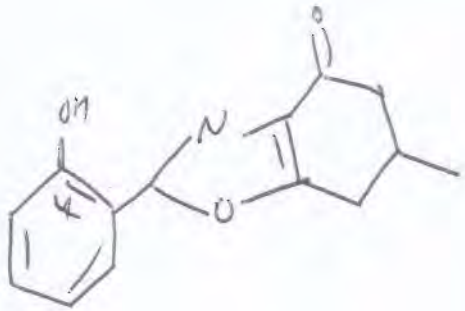

Cpd 6b: 13C NMR

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Current Data Parameters
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FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

======== CHANNEL f1 =====
NUC1 13C
P1 9.00 u
PL1 6.00 d
SFO1 100.6238364 M

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127690 M
WDW EM
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LB 1.00 H
GB 0
PC 1.40

AJC_ACOPI2
(Jones) Alex Chatterley

Current Data Parameters
NAME Nov12-2013
EXPNO 220

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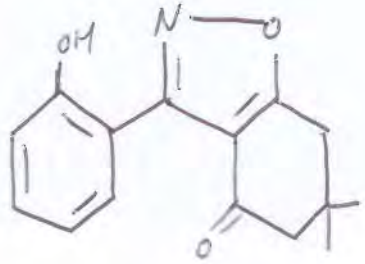

Cpd 4c: 1H NMR

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1.97
1.96
2.00
6.02

AJC_ACOPI2
(Jones) Alex Chatterley

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159.45
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118.90
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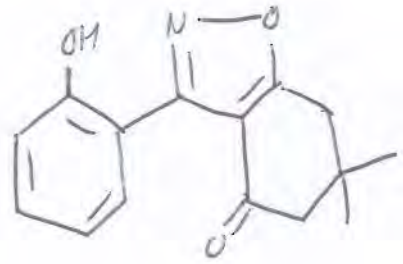

Cpd 4c: 13C NMR

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PROCNO 1

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FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

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PL1 6.00 d
SFO1 100.6238364 M

======== CHANNEL f2 =====
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NUC2 1H
PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
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SF 100.6127690 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

Current Data Parameters
NAME Nov08-2012
EXPNO 160

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1.075

AJC_AC300C
(Jones) Alex Chatterley

Left to right

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3 7.2736 8.4027 2 J(3, 0)
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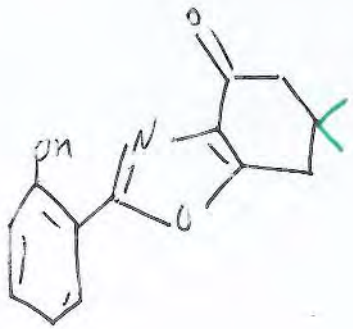

Cpd 6c: 1H NMR

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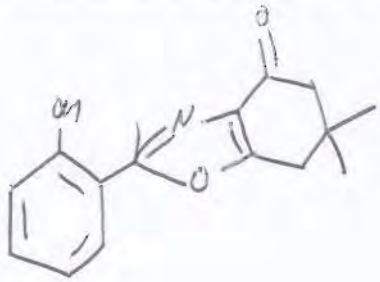
0.90 1.00 1.03 1.02 1.05 2.05 2.09 6.70

AJC_AC300C
(Jones) Alex Chatterley



Cpd 6c:: 13C NMR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PROCNO                        1

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AQ                 1.0879476 s
RG                   18390.4
DW                    16.600 u
DE                     6.00 u
TE                    300.0 K
D1                 1.00000000 s
d11                0.03000000 s
DELTA              0.89999999 s
TD0                           1

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PL1                       3.00 d
SFO1              100.6238364 M

======== CHANNEL f2 =====
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PL12                    3.96 d
PL13                   20.00 d
SFO2              400.1316005 M

F2 - Processing parameter
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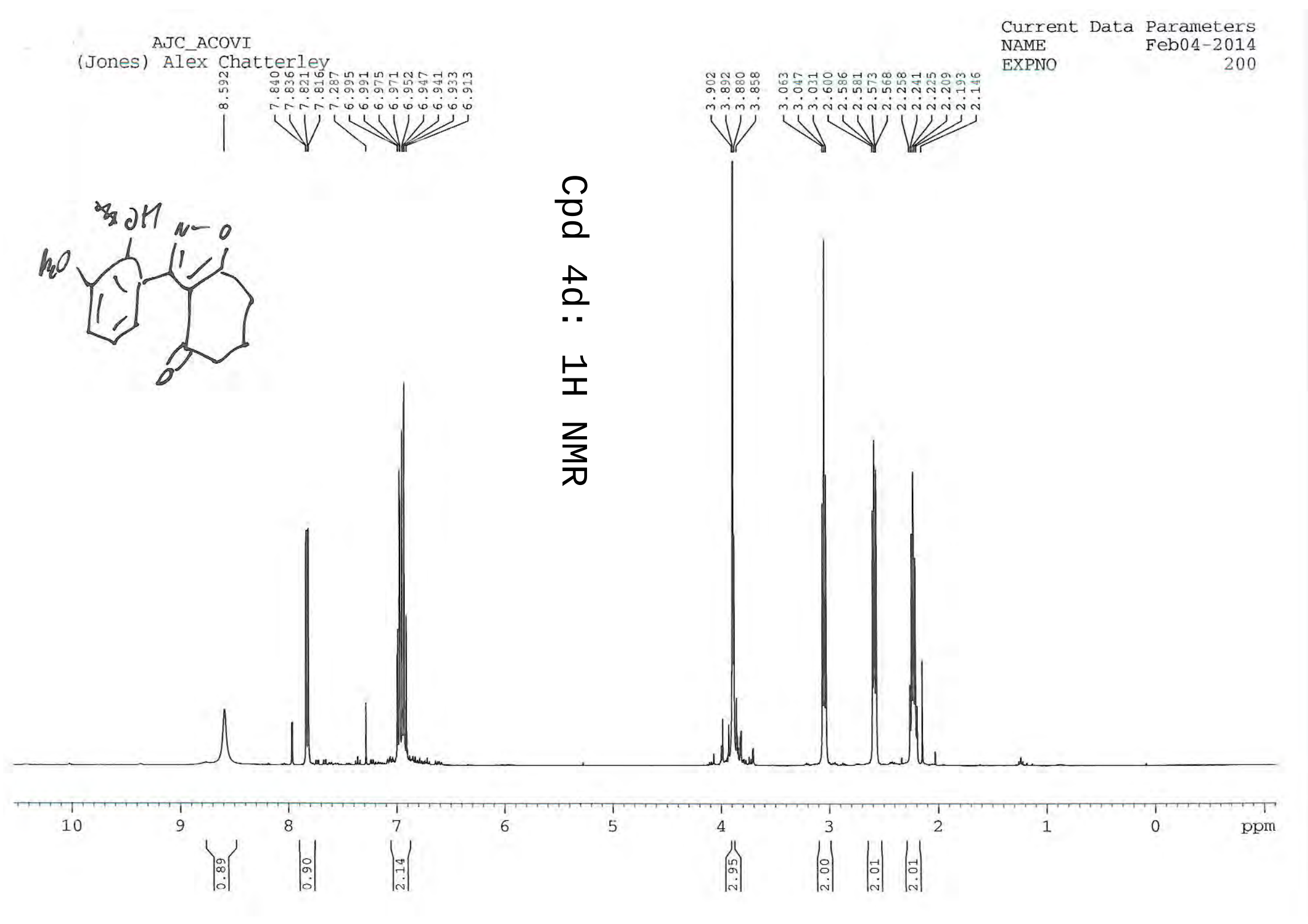
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(Jones) Alex Chatterley
Current Data Parameters
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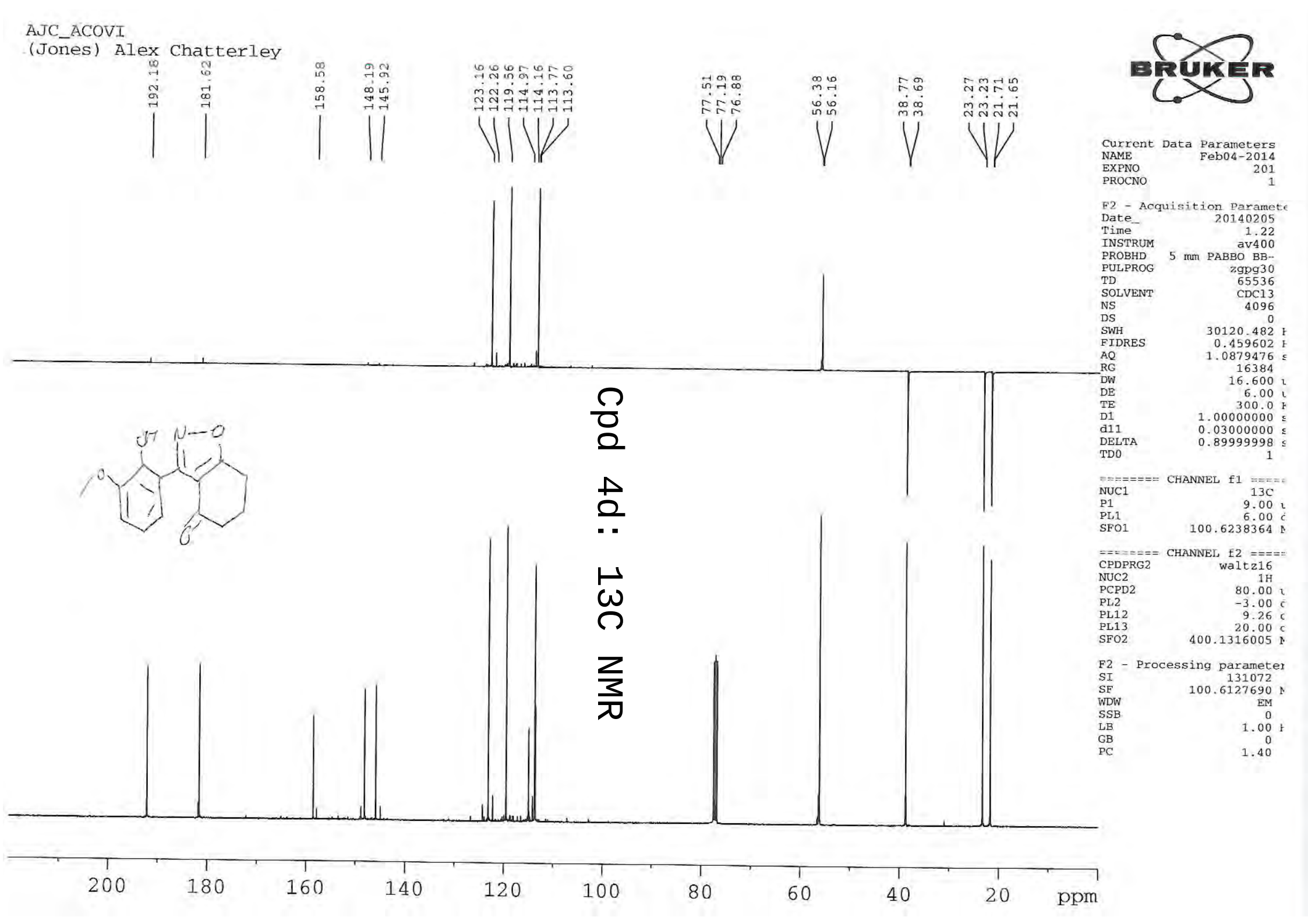
7.836
7.821
7.816
7.287
6.995
6.991
6.975
6.971
6.952
6.947
6.941
6.933
6.913
3.902
3.892
3.880
3.858
3.063
3.047
3.031
2.600
2.586
2.581
2.573
2.568
2.258
2.241
2.225
2.209
2.193
2.146
Cpd 4d: 1H NMR
10
9
8
7
6
5
4
3
2
1
0
ppm
0.89
0.90
2.14
2.95
2.00
2.01
2.01

AJC_ACOVI
(Jones) Alex Chatterley



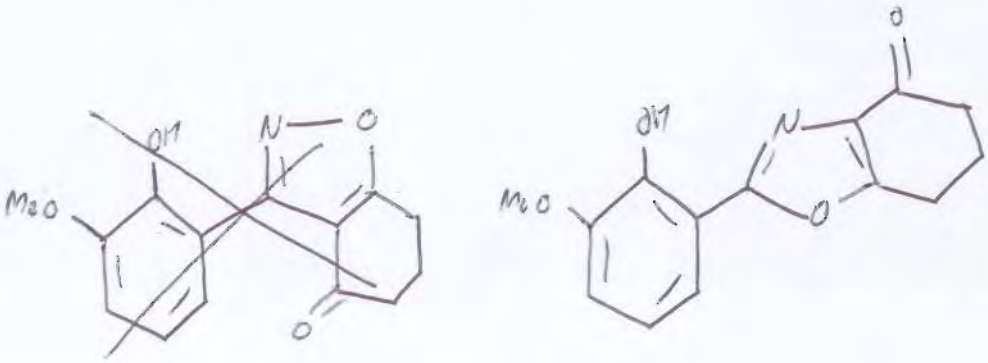
Current Data Parameters

NAME	Feb04-2014
EXPNO	201
PROCNO	1
F2 - Acquisition Paramete	
Date_	20140205
Time	1.22
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4096
DS	0
SWH	30120.482 H
FIDRES	0.459602 H
AQ	1.0879476 s
RG	16384
DW	16.600 u
DE	6.00 u
TE	300.0 K
D1	1.00000000 s
d11	0.03000000 s
DELTA	0.89999998 s
TD0	1
======== CHANNEL f1 =====	
NUC1	13C
P1	9.00 u
PL1	6.00 d
SFO1	100.6238364 M
======== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	-3.00 d
PL12	9.26 d
PL13	20.00 d
SFO2	400.1316005 M
F2 - Processing parameter	
SI	131072
SF	100.6127690 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40

AJC_ACTJ4FA 4096 SCANS PLEASE
(Jones) Alex Chatterley

Current Data Parameters
NAME Jun19-2013
EXPNO 180

10.787
7.418
7.414
7.398
7.394
7.276
6.993
6.975
6.973
6.908
6.888
3.923
3.080
3.065
3.049
2.644
2.629
2.612
2.311
2.295
2.279

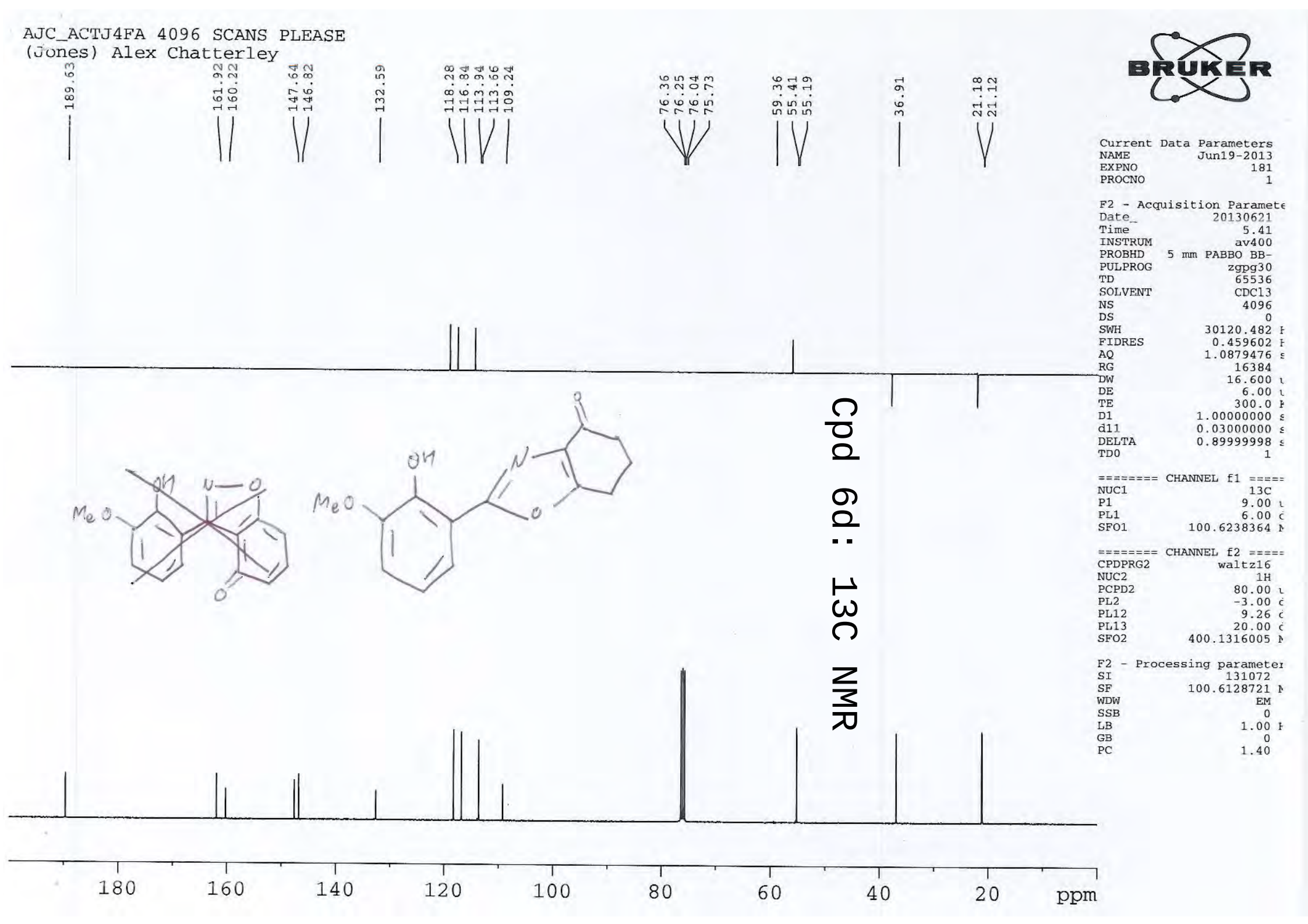


Cpd 6d: 1H NMR

11 10 9 8 7 6 5 4 3 2 1 0 ppm

0.98 0.95 0.98 0.98 3.03 2.00 2.00 2.01

AJC_ACTJ4FA 4096 SCANS PLEASE
(Jones) Alex Chatterley



Current Data Parameters

NAME	Jun19-2013
EXPNO	181
PROCNO	1

F2 - Acquisition Paramete

Date_	20130621
Time	5.41
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4096
DS	0
SWH	30120.482 H
FIDRES	0.459602 H
AQ	1.0879476 s
RG	16384
DW	16.600 u
DE	6.00 u
TE	300.0 K
D1	1.00000000 s
d11	0.03000000 s
DELTA	0.89999998 s
TD0	1

======== CHANNEL f1 =====

NUC1	13C
P1	9.00 u
PL1	6.00 d
SFO1	100.6238364 M

======== CHANNEL f2 =====

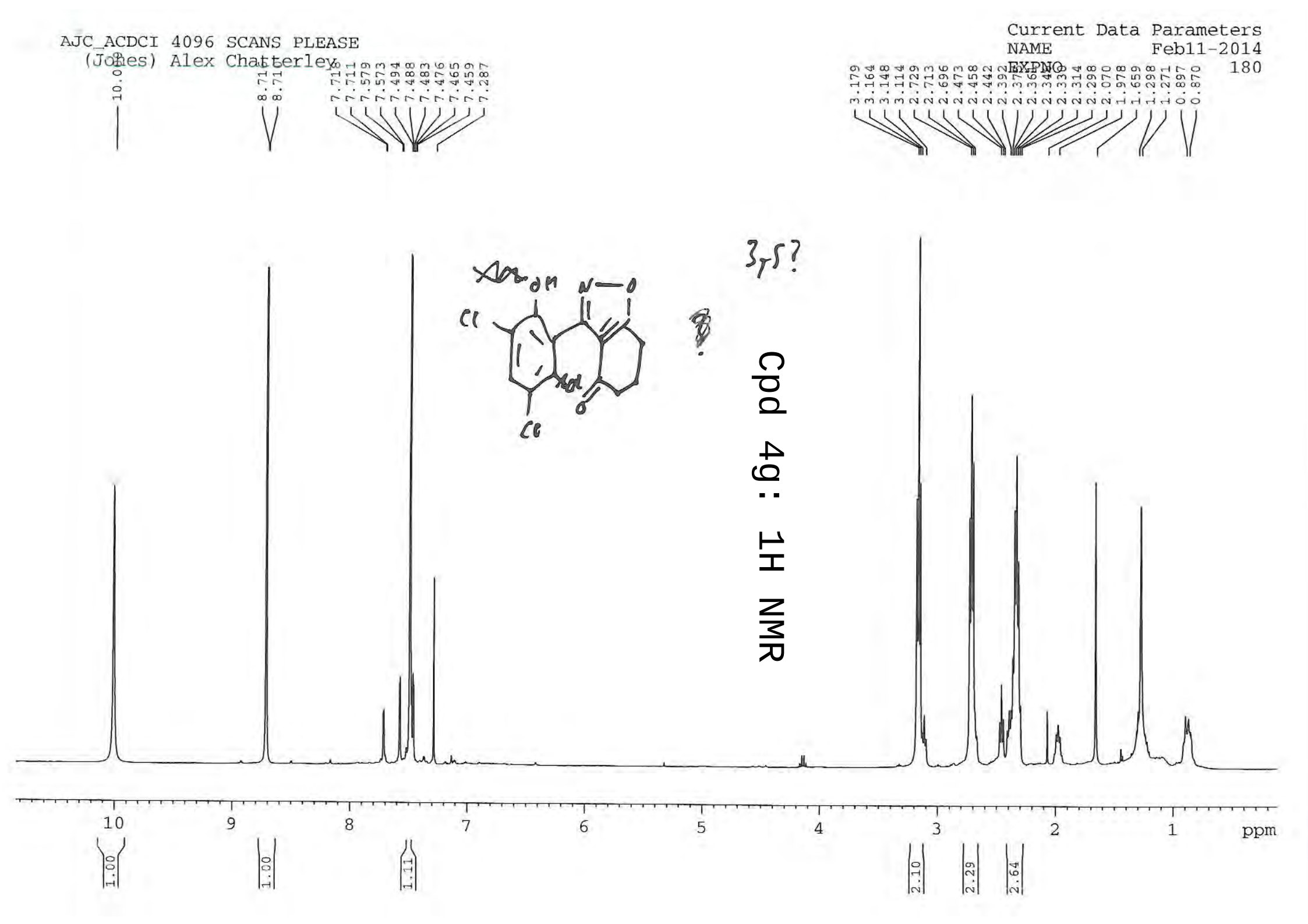
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	-3.00 d
PL12	9.26 d
PL13	20.00 d
SFO2	400.1316005 M

F2 - Processing parameter

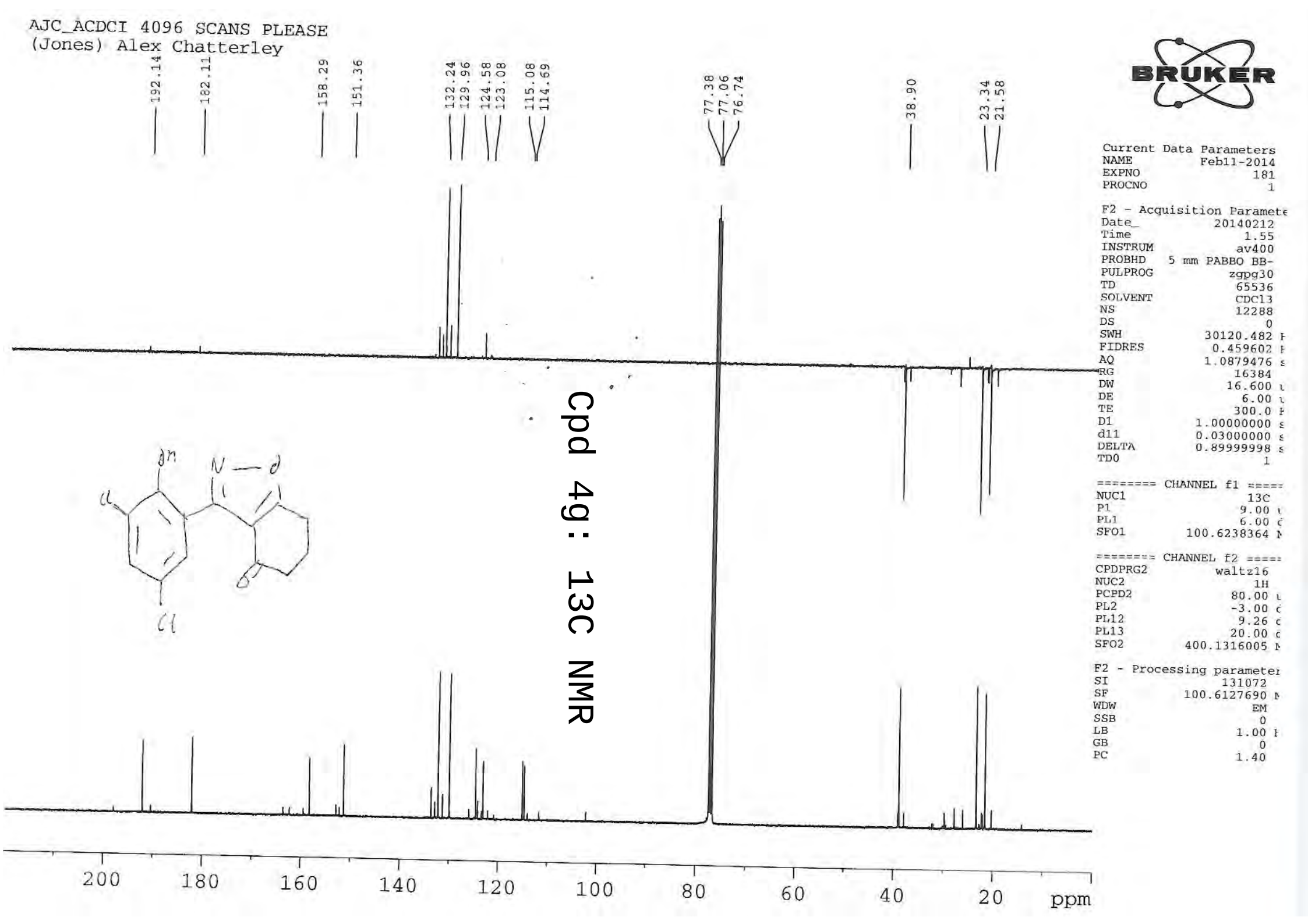
SI	131072
SF	100.6128721 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40

AJC_ACDCI 4096 SCANS PLEASE
(Jones) Alex Chatterley

Current Data Parameters
NAME Feb11-2014
EXPNO 180



AJC_ACDCI 4096 SCANS PLEASE
(Jones) Alex Chatterley



Current Data Parameters

NAME	Feb11-2014
EXPNO	181
PROCNO	1

F2 - Acquisition Parameter

Date_	20140212
Time	1.55
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	12288
DS	0
SWH	30120.482 H
FIDRES	0.459602 H
AQ	1.0879476 s
RG	16384
DW	16.600 u
DE	6.00 u
TE	300.0 K
D1	1.00000000 s
d11	0.03000000 s
DELTA	0.89999998 s
TD0	1

======== CHANNEL f1 =====

NUC1	13C
P1	9.00 u
PL1	6.00 d
SFO1	100.6238364 M

======== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	-3.00 d
PL12	9.26 d
PL13	20.00 d
SFO2	400.1316005 M

F2 - Processing parameter

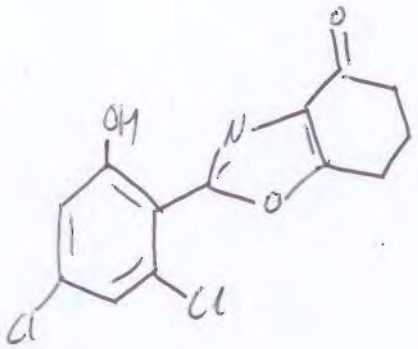
SI	131072
SF	100.6127690 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40

AJC_AC379F3FA
(Jones) Alex Chatterley

Current Data Parameters
NAME Mar13-2013
EXPNO 510

7.703
7.690
7.446
7.277

3.120
3.089
2.680
2.664
2.648
2.345
2.331
2.317

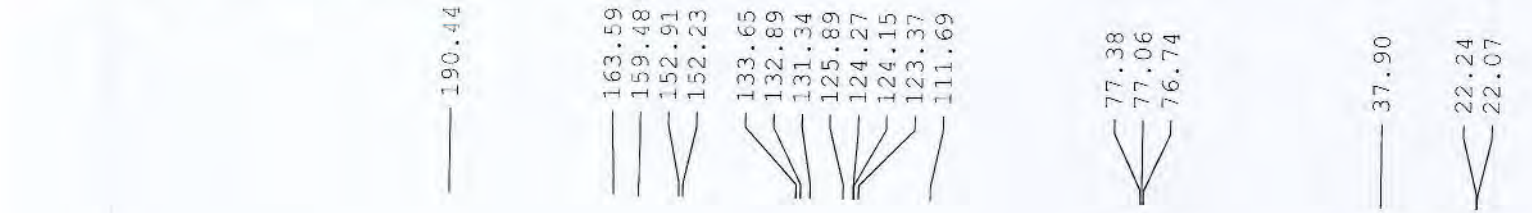


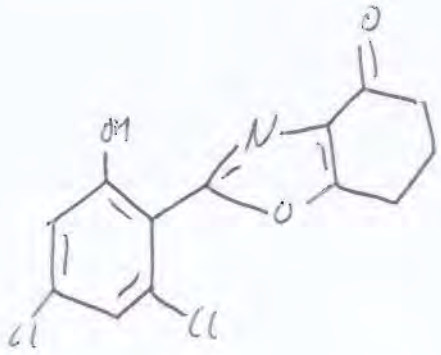
Cpd 6g: 1H NMR

9.0 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 ppm

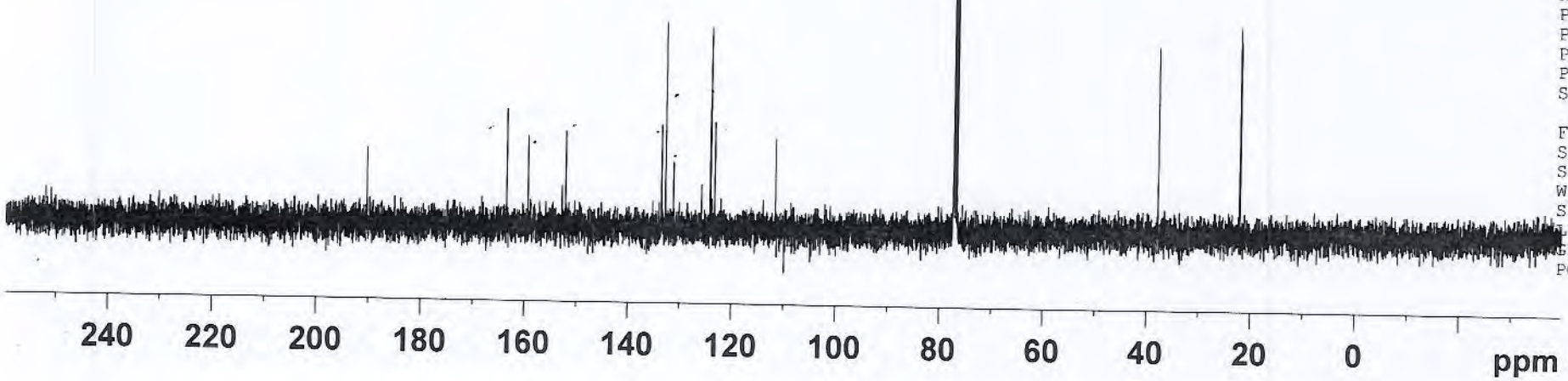
0.98
1.38
2.00
1.99
2.00

AJC_AC379F3FA
(Jones) Alex Chatterley





Cpd 6g: 13C NMR



BRUKER

Current Data Parameters
NAME Mar13-2013
EXPNO 511
PROCNO 1

F2 - Acquisition Paramete
Date_ 20130314
Time 6.00
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 30120.482 H
FIDRES 0.459602 H
AQ 1.0879476 s
RG 41285.1
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

======== CHANNEL f1 =====
NUC1 13C
P1 9.00 u
PL1 -3.00 d
SFO1 100.6238364 M

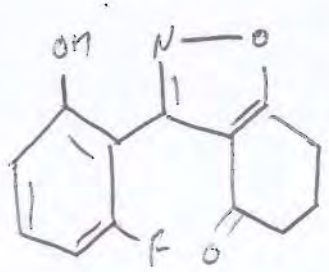
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127690 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

AJC_AC419F2FA
(Jones) Alex Chatterley

Current Data Parameters
NAME May13-2013
EXPNO 60

9.487
8.401 8.397 8.393 8.380 8.377 8.373
7.285 7.261 7.257 7.241 7.237 7.234 7.231 7.214 7.210 7.011 6.998 6.990 6.978 6.970 6.958
3.166 3.150 3.134 2.717 2.711 2.702 2.701 2.698 2.690 2.684 2.351 2.345 2.334 2.329 2.318 2.309 2.302 2.292 2.286 2.192 1.643 1.275
0.094 0.020



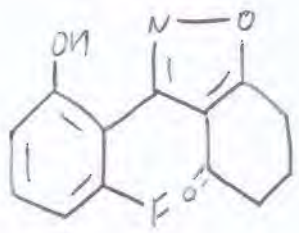
Cpd 4h: 1H NMR

10 9 8 7 6 5 4 3 2 1 ppm

0.93 0.95 0.96 1.02 2.01 2.00 2.01

AJC_AC419F2FA
(Jones) Alex Chatterley

192.35
181.88
158.83
158.80
153.33
150.91
145.31
145.18
126.89
126.85
119.61
119.54
118.82
118.64
115.35
115.32
114.85
77.35
77.24
77.04
76.72
38.94
23.34
21.60



Cpd 4h: 13C NMR

240 220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm

BRUKER

Current Data Parameters
NAME May13-2013
EXPNO 61
PROCNO 1

F2 - Acquisition Paramete
Date_ 20130513
Time 18.24
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 30120.482 H
FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

======== CHANNEL f1 =====
NUC1 13C
P1 9.00 u
PL1 6.00 d
SFO1 100.6238364 M

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127696 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

AJC_AC445
(Jones) Alex Chatterley

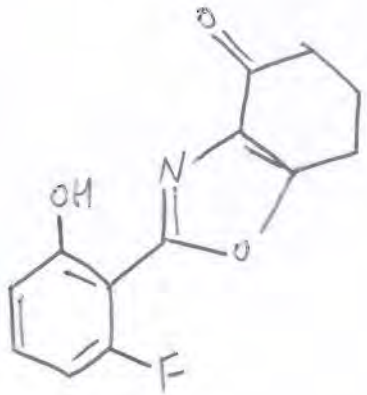
Current Data Parameters
NAME
EXPNO 150
Nov15-2013

10.774

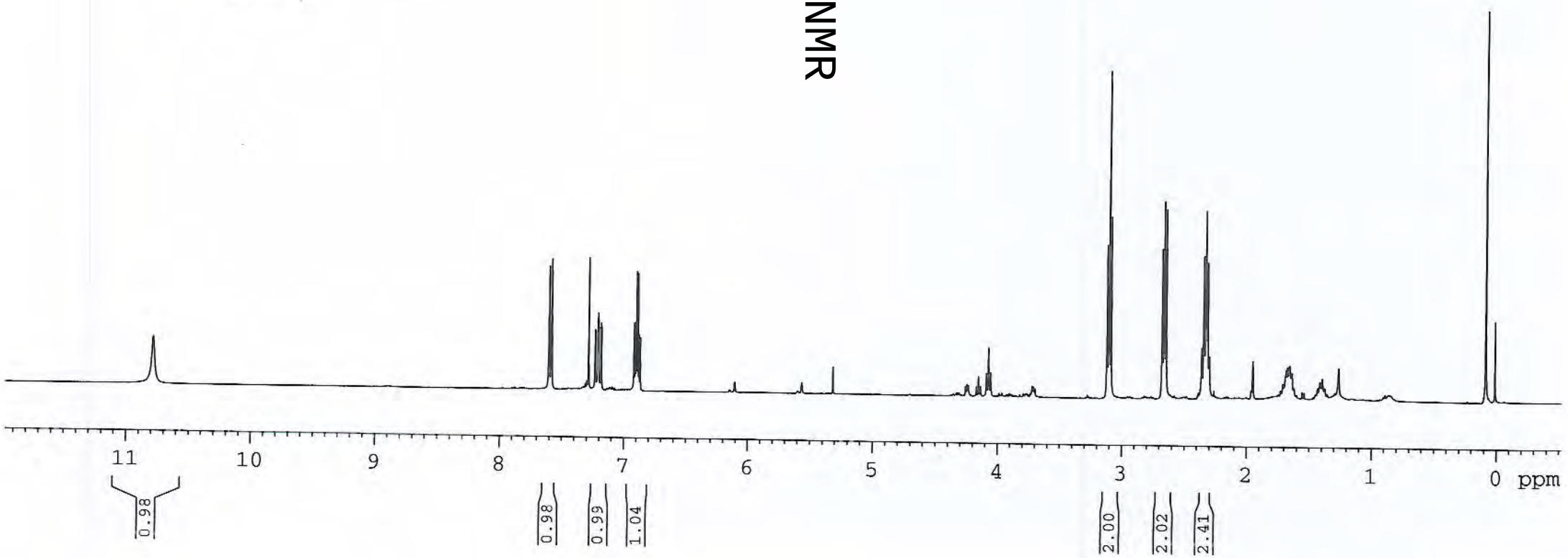
7.608 7.604 7.601 7.588 7.584 7.581 7.286 7.234 7.230 7.214 7.210 7.207 7.203 7.187 7.183 6.922 6.910 6.902 6.890 6.881 6.870

4.073 3.121 3.105 3.090 2.681 2.675 2.666 2.648 2.363 2.347 2.331 2.324 2.315 2.305 2.295 1.955 1.949 1.685 1.676 1.665 1.658 1.265

0.087 0.012

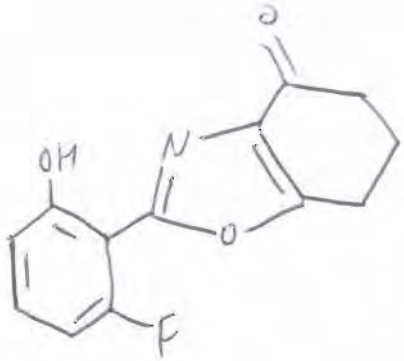


Cpd 6h: 1H NMR



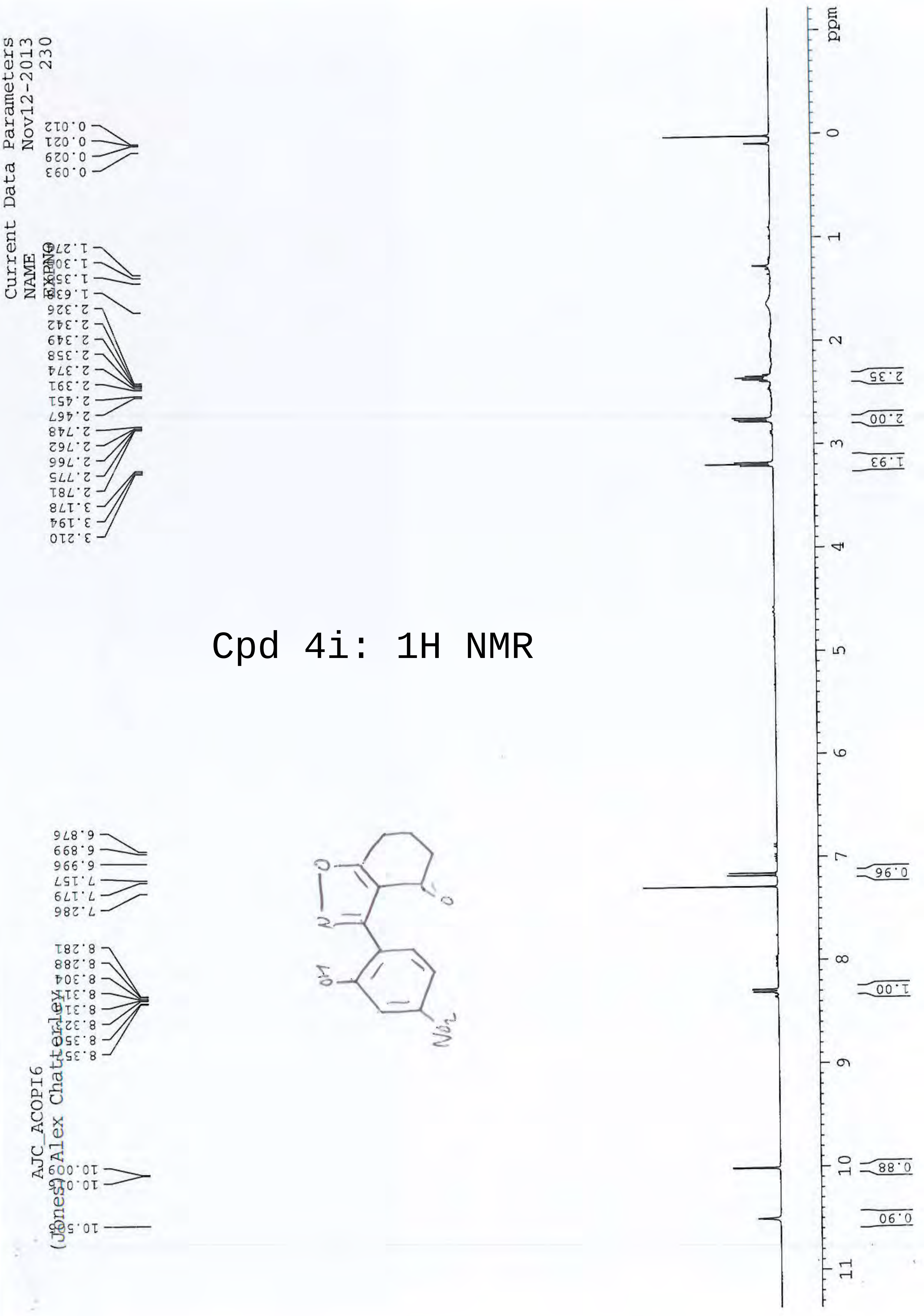
AJC_AC445
(Jones) Alex Chatterley

189.60
162.13
159.49
159.45
151.81
149.37
145.32
145.19
132.63
120.36
120.33
118.33
118.20
118.15
118.13
111.05
111.01
76.36
76.04
75.72
36.90
27.32
21.19
21.10



Cpd 6h: 13C NMR

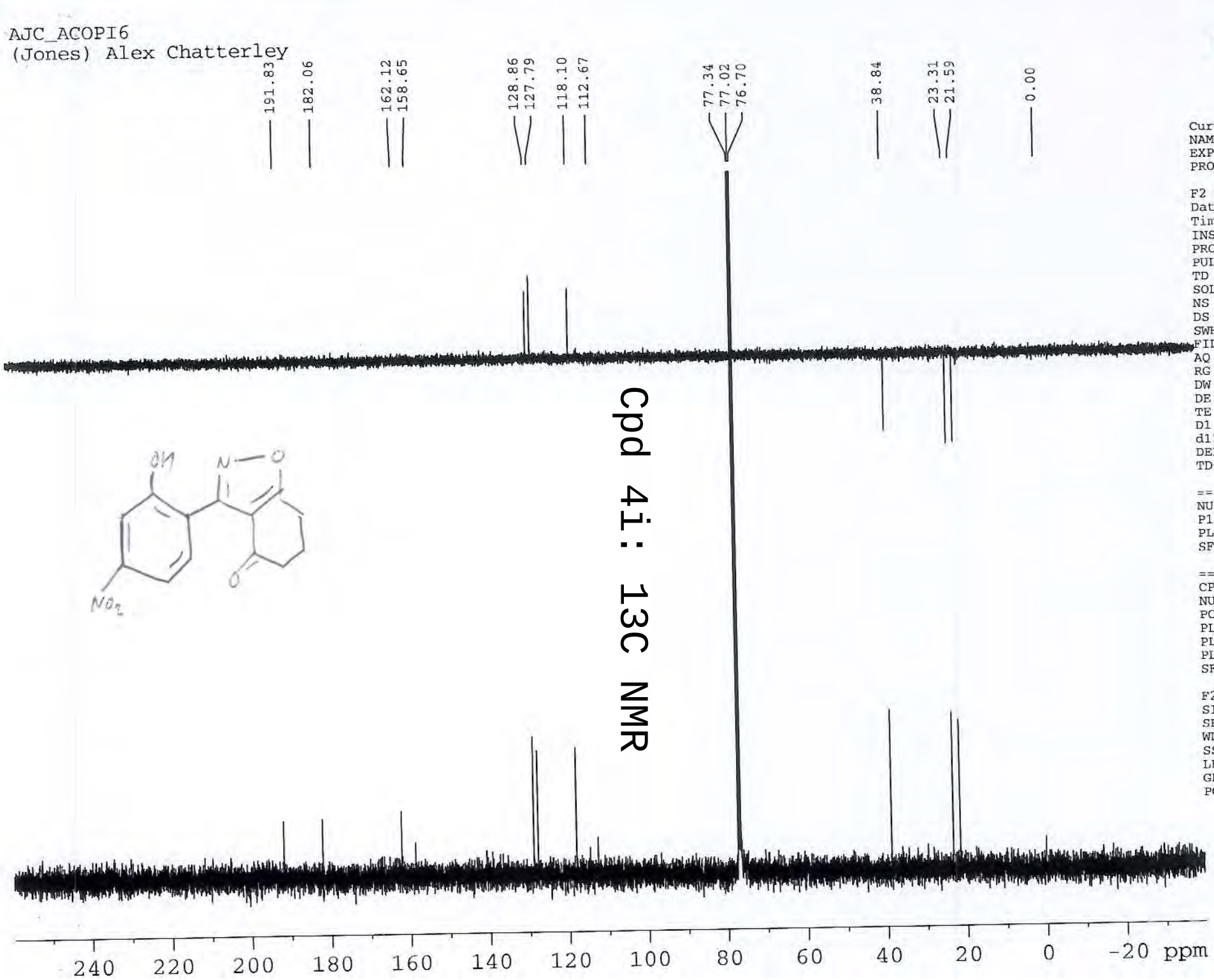
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



Current Data Parameters
NAME AJC_ACOPI6
EXPNO 230
Nov12-2013
(Jones) Alex Chatterley
ppm

Cpd 4i: 1H NMR

AJC_ACOPI6
(Jones) Alex Chatterley



BRUKER

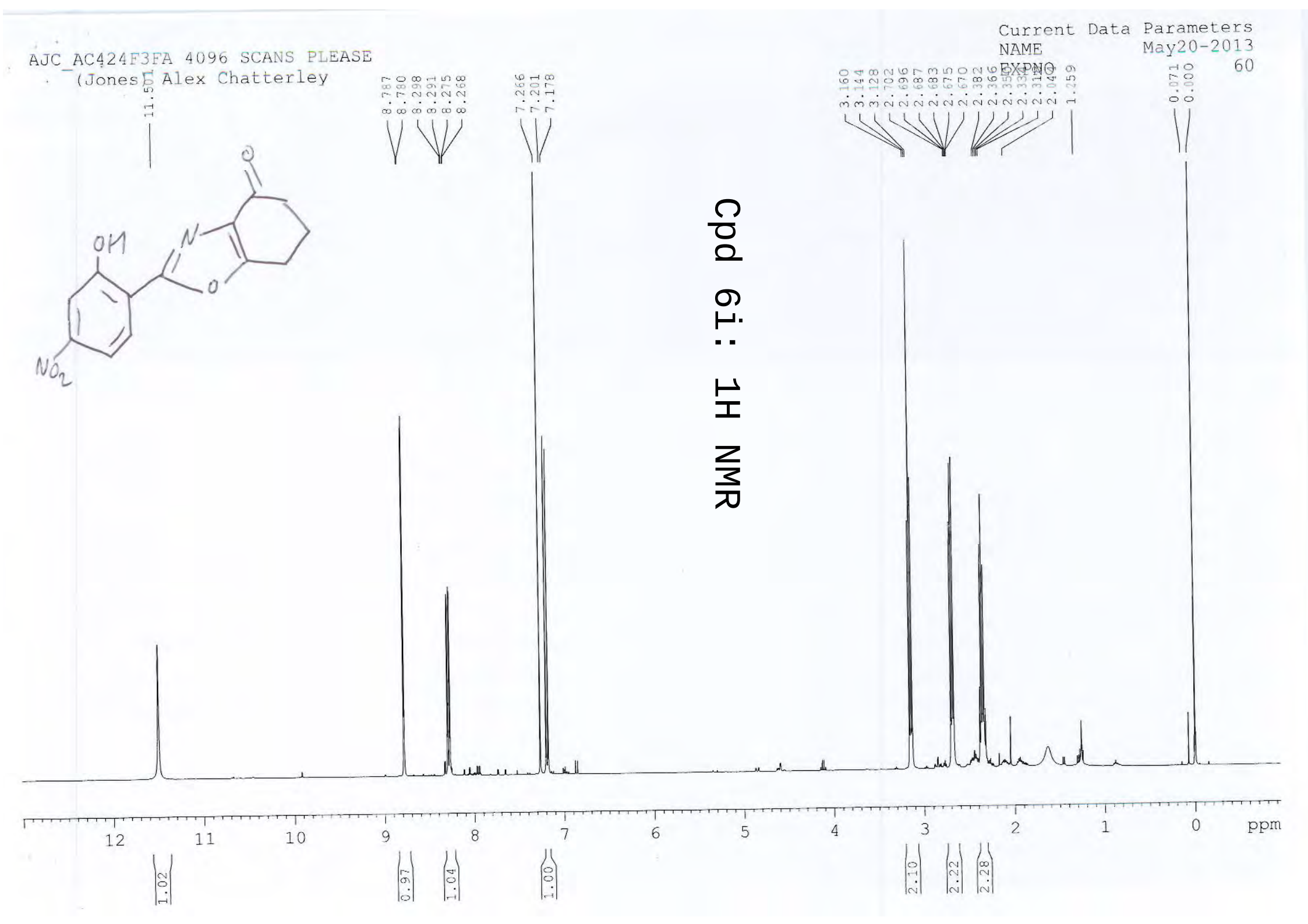
Current Data Parameters
NAME Nov12-2013
EXPNO 231
PROCNO 1

F2 - Acquisition Paramete
Date_ 20131113
Time 4.37
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 30120.482 H
FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

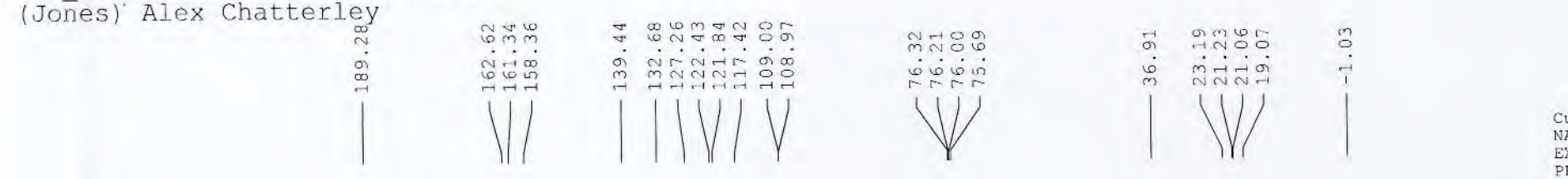
======== CHANNEL f1 =====
NUC1 13C
P1 9.00 u
PL1 6.00 d
SFO1 100.6238364 M

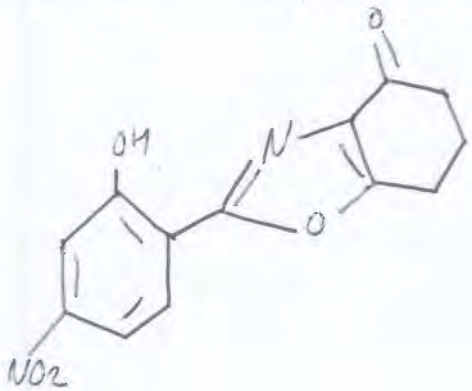
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -3.00 d
PL12 9.26 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127709 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

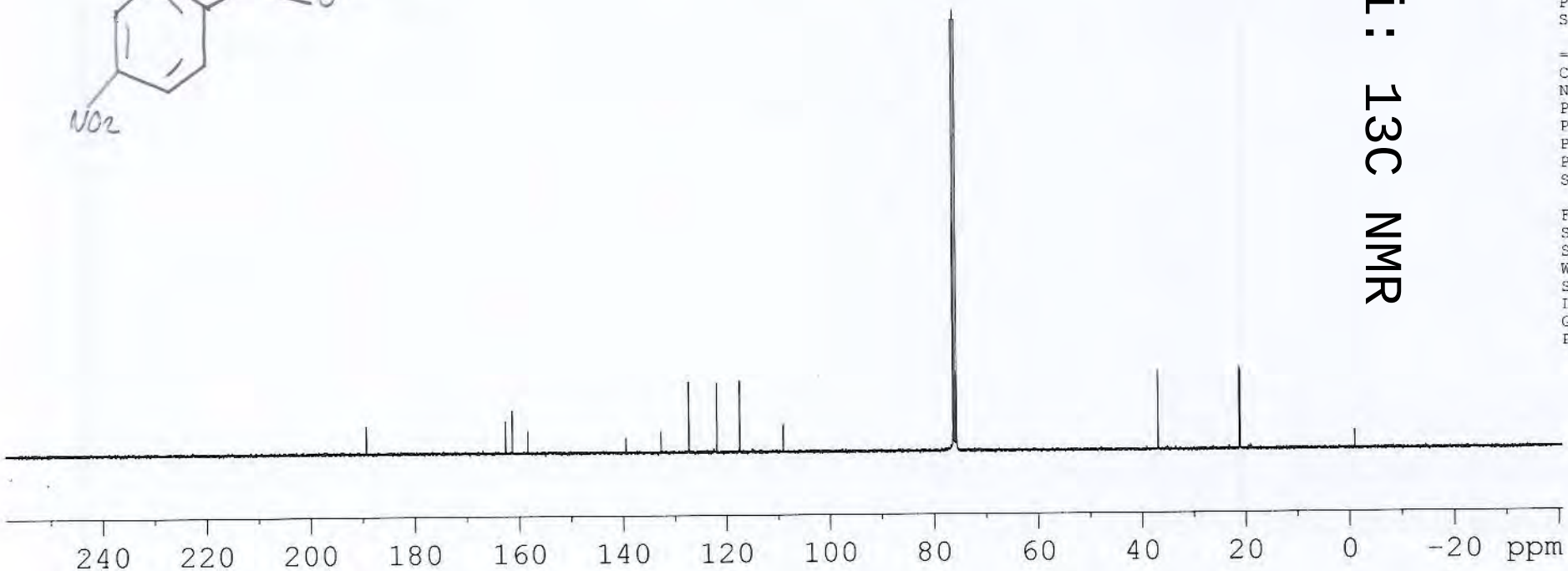

AJC_AC424F3FA 4096 SCANS PLEASE
(Jones) Alex Chatterley
Current Data Parameters
NAME
EXPNO 60
May20-2013
Cpd 6i: 1H NMR
11.501
8.787
8.780
8.298
8.291
8.275
8.268
7.266
7.201
7.178
3.160
3.144
3.128
2.702
2.696
2.687
2.683
2.675
2.670
2.382
2.366
2.350
2.334
2.318
2.044
1.259
0.071
0.000
OH
N
O
NO2
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
0.97
1.04
1.00
2.10
2.22
2.28

AJC_AC424F3FA 4096 SCANS PLEASE
(Jones) Alex Chatterley





Cpd 6i: 13C NMR



Current Data Parameters

NAME	May20-2013
EXPNO	61
PROCNO	1

F2 - Acquisition Paramete

Date_	20130520
Time	20.38
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	4096
DS	0
SWH	30120.482 H
FIDRES	0.459602 H
AQ	1.0879476 s
RG	16384
DW	16.600 u
DE	6.00 u
TE	300.0 K
D1	1.00000000 s
d11	0.03000000 s
DELTA	0.89999998 s
TD0	1

======== CHANNEL f1 =====

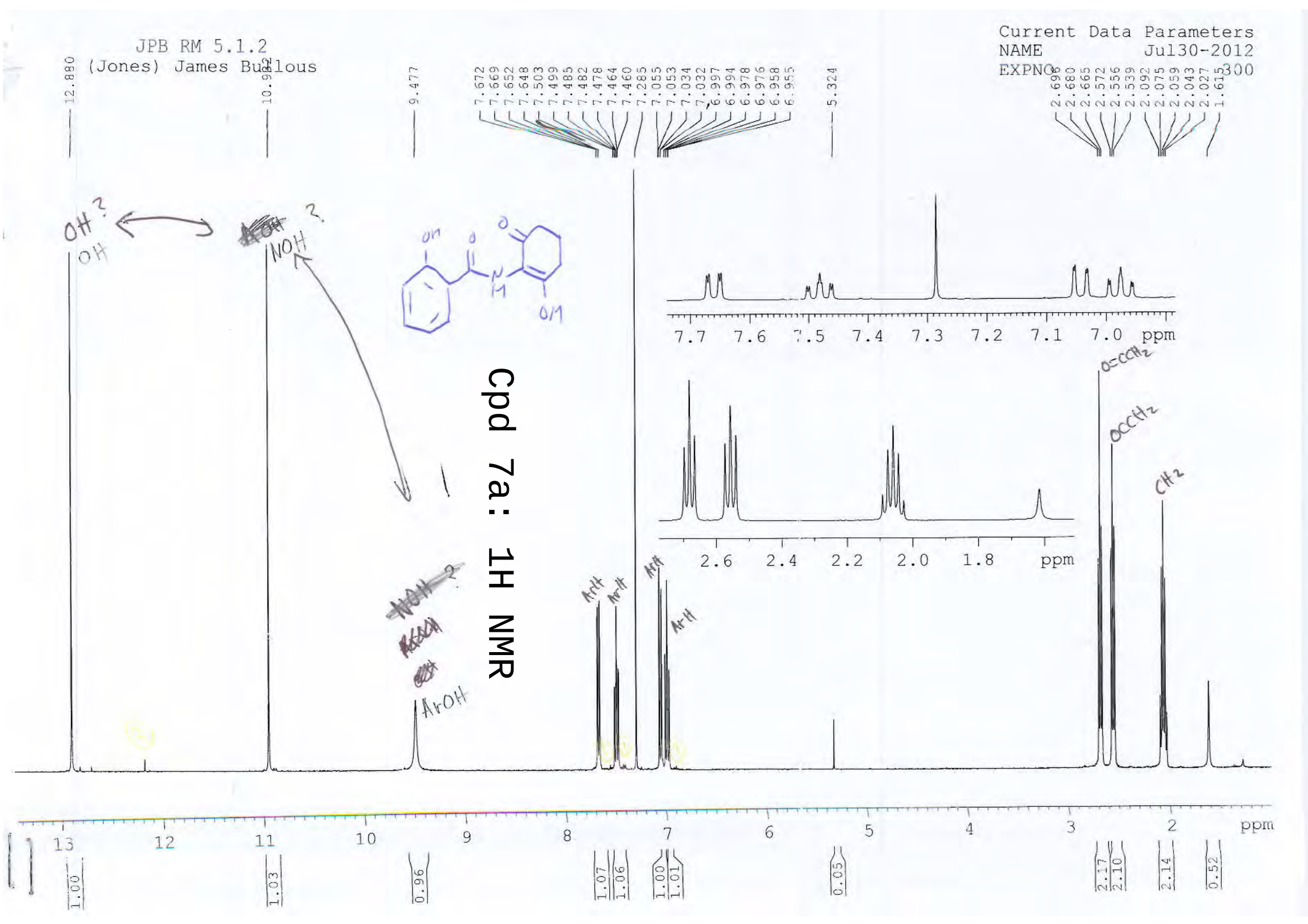
NUC1	13C
P1	9.00 u
PL1	6.00 d
SFO1	100.6238364 M

======== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	-3.00 d
PL12	9.26 d
PL13	20.00 d
SFO2	400.1316005 M

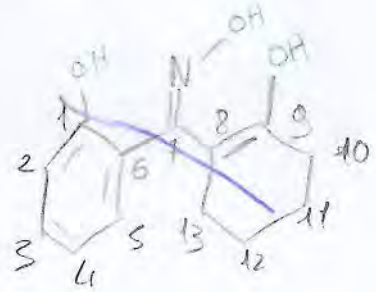
F2 - Processing parameter

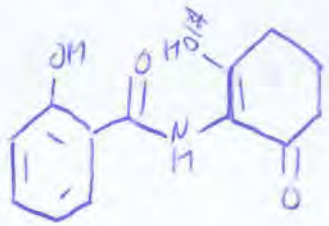
SI	131072
SF	100.6128721 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40


JPB RM 5.1.2
(Jones) James Bullous
Current Data Parameters
NAME Jul30-2012
EXPNO 300
12.880
10.992
9.477
7.672
7.669
7.652
7.648
7.503
7.499
7.485
7.482
7.478
7.464
7.460
7.285
7.055
7.053
7.034
7.032
6.997
6.994
6.978
6.976
6.958
6.955
5.324
2.698
2.680
2.665
2.572
2.556
2.539
2.092
2.075
2.059
2.043
2.027
1.615
OH ?
OH
NOH ?
NOH
ArH
ArH
ArH
ArH
ArOH
O=CCH2
OCCH2
CH2
Cpd 7a: 1H NMR
7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 ppm
2.6 2.4 2.2 2.0 1.8 ppm
13 12 11 10 9 8 7 6 5 4 3 2 ppm
1.00
1.03
0.96
1.07
1.06
1.00
1.01
0.05
2.17
2.10
2.14
0.52

JPB RM 5.1.2
(Jones) James Bullous

192.91
167.36
165.94
160.81
135.24
127.06
119.75
118.86
113.67
113.38
77.34
77.03
76.71
34.97
30.19
20.32





Cpd 7a: 13C NMR

Current Data Parameters
NAME Aug03-2012
EXPNO 351
PROCNO 1

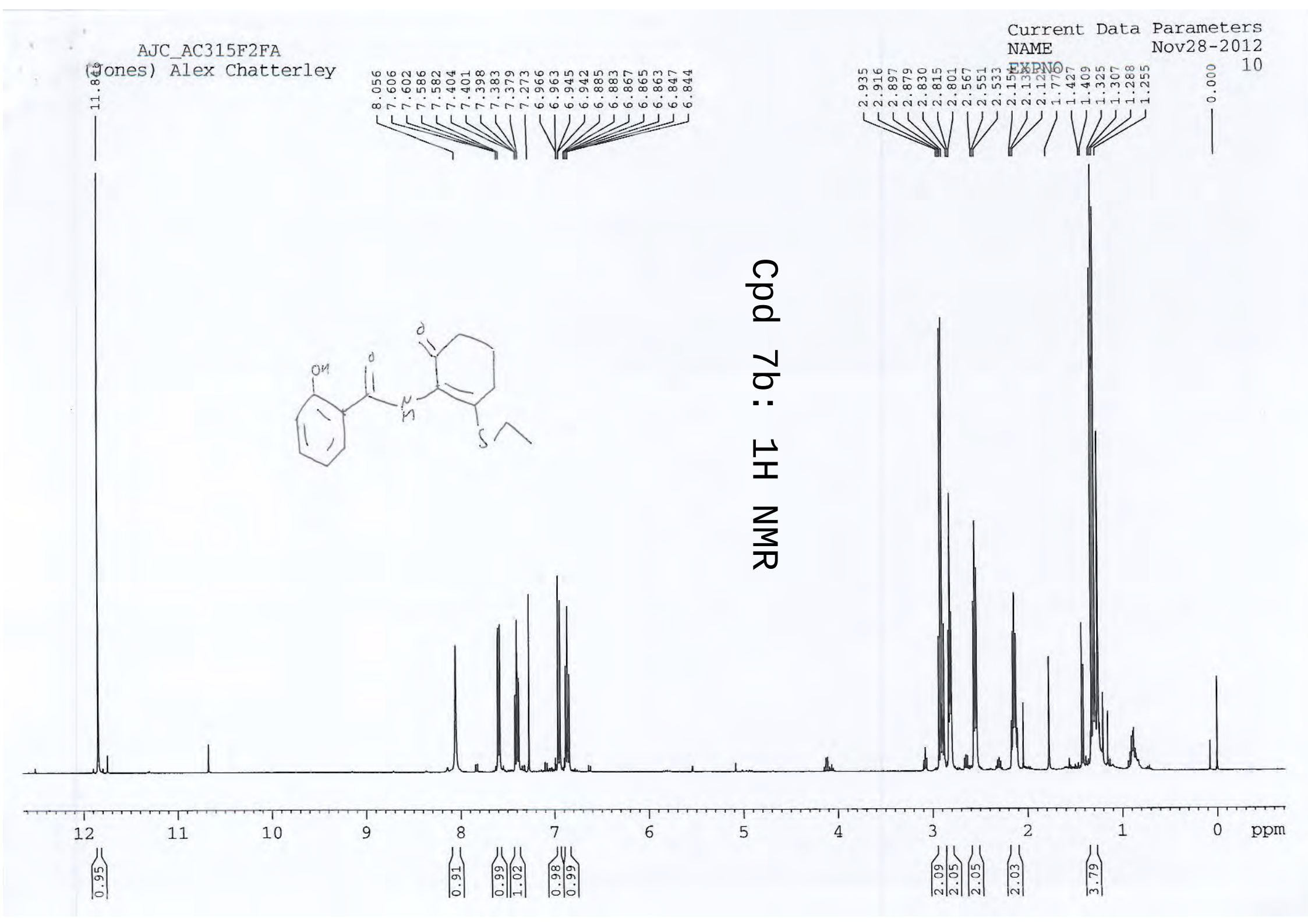
F2 - Acquisition Paramete
Date_ 20120806
Time 18.24
INSTRUM av400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 0
SWH 30120.482 H
FIDRES 0.459602 H
AQ 1.0879476 s
RG 18390.4
DW 16.600 u
DE 6.00 u
TE 300.0 K
D1 1.00000000 s
d11 0.03000000 s
DELTA 0.89999998 s
TD0 1

======== CHANNEL f1 =====
NUC1 13C
P1 6.75 u
PL1 3.00 d
SFO1 100.6238364 M

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 u
PL2 -4.00 d
PL12 10.54 d
PL13 20.00 d
SFO2 400.1316005 M

F2 - Processing parameter
SI 131072
SF 100.6127690 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


AJC_AC315F2FA
(Jones) Alex Chatterley
Current Data Parameters
NAME
Nov28-2012
EXPNO
10
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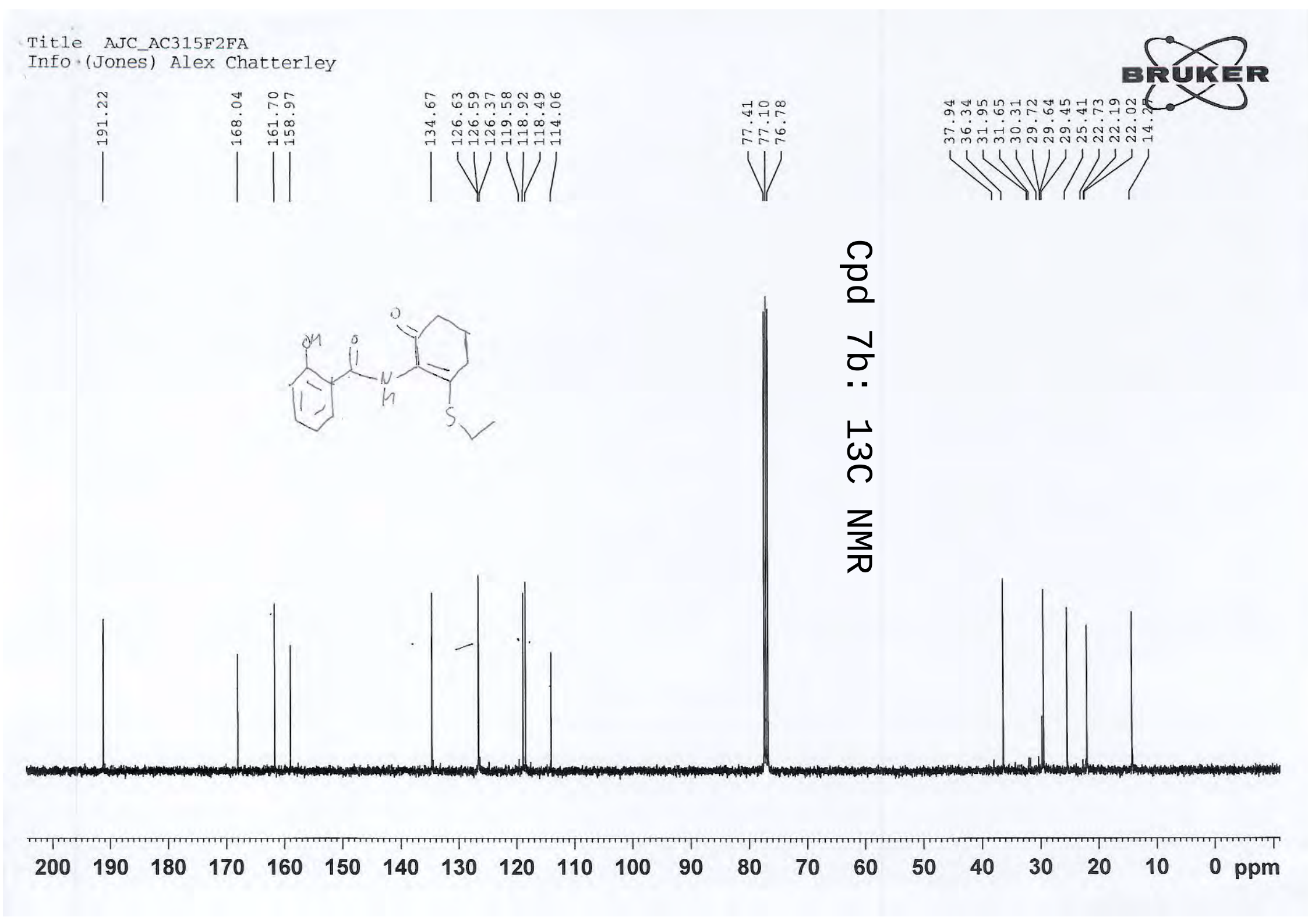
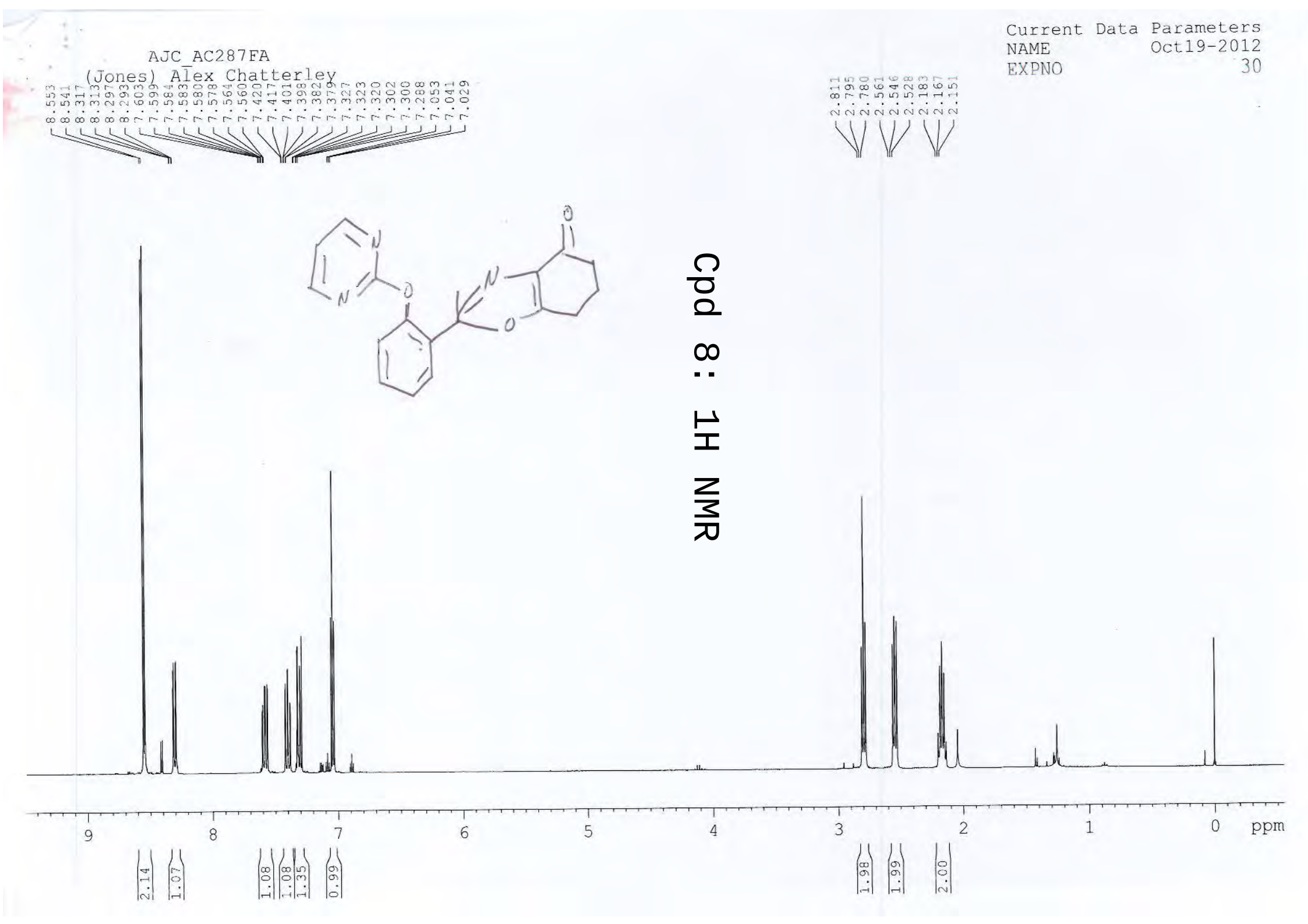
7.606
7.602
7.586
7.582
7.404
7.401
7.398
7.383
7.379
7.273
6.966
6.963
6.945
6.942
6.885
6.883
6.867
6.865
6.863
6.847
6.844
2.935
2.916
2.897
2.879
2.830
2.815
2.801
2.567
2.551
2.533
1.770
1.427
1.409
1.325
1.307
1.288
1.255
0.000
Cpd 7b: 1H NMR
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
0.91
0.99
1.02
0.98
0.99
2.09
2.05
2.05
2.03
3.79

Title AJC_AC315F2FA
Info (Jones) Alex Chatterley




AJC_AC287FA
(Jones) Alex Chatterley
Current Data Parameters
NAME Oct19-2012
EXPNO 30
8.553
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8.317
8.313
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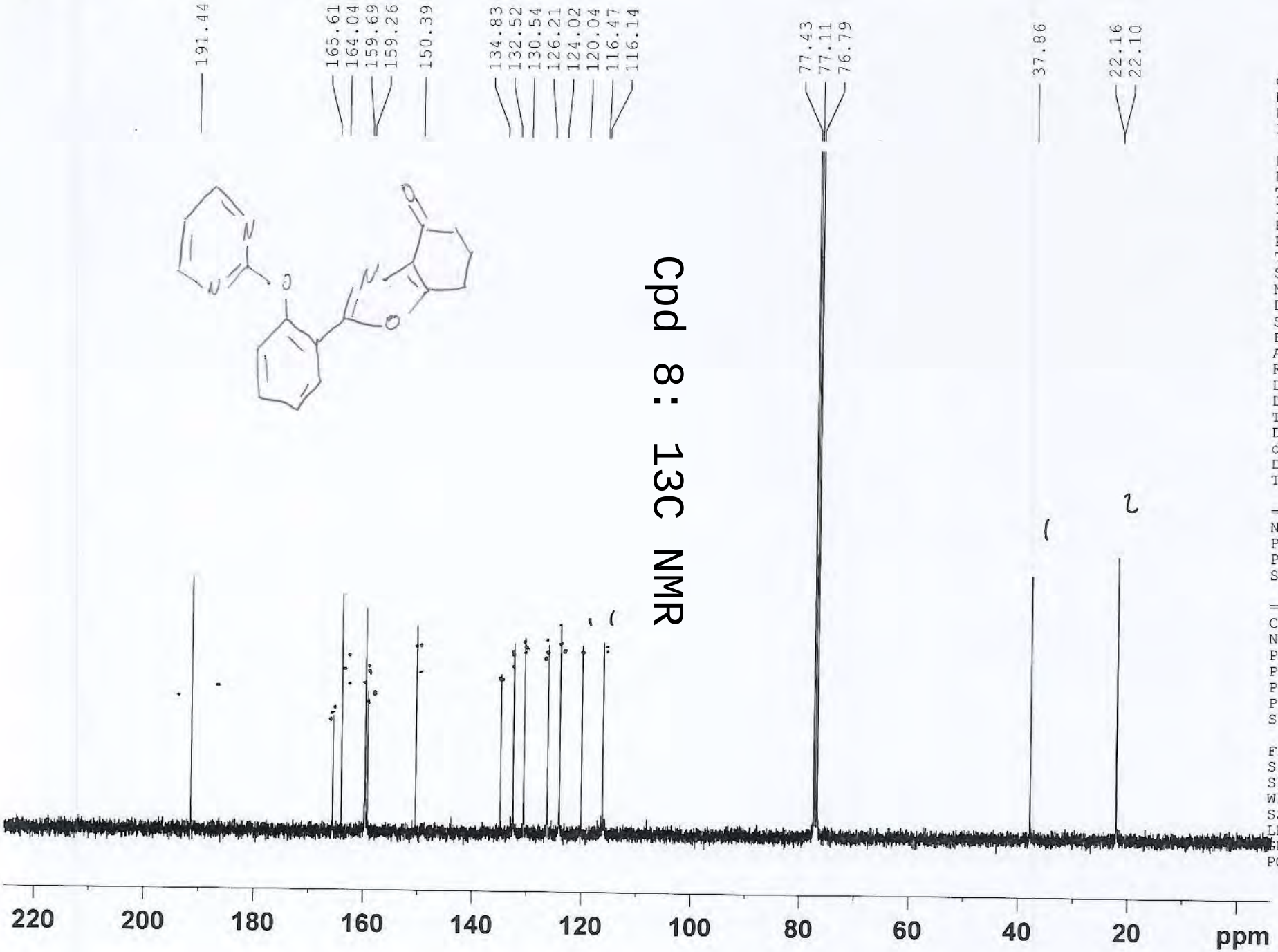
8.293
7.603
7.599
7.584
7.583
7.580
7.578
7.564
7.560
7.420
7.417
7.401
7.398
7.382
7.379
7.327
7.323
7.320
7.302
7.300
7.288
7.053
7.041
7.029
2.811
2.795
2.780
2.561
2.546
2.528
2.183
2.167
2.151
Cpd 8: 1H NMR
9
8
7
6
5
4
3
2
1
0
ppm
2.14
1.07
1.08
1.08
1.35
0.99
1.98
1.99
2.00

AJC_AC287FA
(Jones) Alex Chatterley



BRUKER

Current Data Parameters	
NAME	Oct19-2012
EXPNO	31
PROCNO	1

F2 - Acquisition Paramete	
Date_	20121019
Time	19.49
INSTRUM	av400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	30120.482 H
FIDRES	0.459602 H
AQ	1.0879476 s
RG	18390.4
DW	16.600 u
DE	6.00 u
TE	300.0 K
D1	1.00000000 s
d11	0.03000000 s
DELTA	0.89999998 s
TD0	1

======== CHANNEL f1 =====	
NUC1	13C
P1	6.75 u
PL1	3.00 d
SFO1	100.6238364 M

======== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 u
PL2	-4.00 d
PL12	10.54 d
PL13	20.00 d
SFO2	400.1316005 M

F2 - Processing parameter	
SI	131072
SF	100.6127690 M
WDW	EM
SSB	0
LB	1.00 H
GB	0
PC	1.40