Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

for

The intramolecular amination of allenes

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(1) Details for the single crystal X-ray diffraction experiments

(2) Experimental procedures and characterisation data for experiments in Schemes 1, 3 and 4 and Table 1

(3) ^{1}H NMR and ^{13}C NMR spectra [spectra are the machine-generated original PDFs, except that the spectra for **7**, **8**, **14**, **15**, **28**, and **29** (obtained at GSK, Harlow, UK) were generated in MestReNova for Mac OS X]

X-ray details and data summary

Single crystal X-ray diffraction data were obtained using a Nonius Kappa-CCD area detector diffractometer, with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å) at 150 K. Cell parameters and intensity data were processed using the DENZO-SMN package[1] and reflection intensities were corrected for absorption effects by the multi-scan method, based on multiple scans of identical and Laue equivalent reflections. The structures were solved by direct methods using SIR92[2] and refined by full-matrix least squares on F^2 using the CRYSTALS suite[3] as per the details in the CIF.

Crystal data for Compound **5**: (clear colourless, 0.14 × 0.30 × 0.34 mm): $C_{10}H_{17}NO_5S$ M_r = 263.31; monoclinic, $P2_1/n$; a = 6.5055(2) Å, b = 22.4224(6) Å, c = 8.9905(3) Å, β = 108.6921(10)°, V = 1019.55(6) Å^3; Z = 4; μ = 0.270 mm^{-1}; D_{calc} = 1.408 gcm^{-3}; reflections collected = 12353; independent reflections = 2829 (R_{int} = 0.035); R values [$I > 2\sigma(I)$, 2141 reflections]: R_1 = 0.0391, wR_2 = 0.0864; $\rho_{min/max}$ = –0.47/0.41 e Å^{-3}; CCDC 757478. Crystal data for Compound **12**: (clear colourless, 0.34 × 0.62 × 0.64 mm): $C_{10}H_{17}NO_5S$ M_r = 263.31; triclinic, $P\bar{1}$; a = 7.6562(2) Å, b = 8.7683(2) Å, c = 10.0293(2) Å, α = 77.2922(12)°, β = 78.9430(12)°, γ = 69.3572(10)°, V = 609.79(2) Å^3; Z = 2; μ = 0.275 mm^{-1}; D_{calc} = 1.434 gcm^{-3}; Reflections collected = 8663; independent reflections = 2766 (R_{int} = 0.021); R values [$I > 2\sigma(I)$, 2569 reflections]: R_1 = 0.0330, wR_2 = 0.0837; $\rho_{min/max}$ = –0.40/0.34 e Å^{-3}; CCDC 757479. Crystal data for Compound **17** were published by Feast *et al.*[4] but are included in the CIF for completeness (CCDC 763606).

Crystallographic data (excluding structure factors) for the structures of **5**, **12** and **17** have been deposited with the Cambridge Crystallographic Data Centre (CCDC 757478-9 & 763606). Copies of these data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

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General Procedure A

To a solution of sulfamate (1.0 equiv.) in dichloromethane (20 mL/mmol) was added MgO (2.3 equiv.), rhodium(II) acetate dimer (0.05 equiv.) and iodobenzene diacetate (1.3 equiv.) at RT. After stirring for 18 h, the reaction mixture was filtered through Celite and concentrated *in vacuo* to give the crude product.

4-Oxo-1-(sulfamoyloxy)pentan-3-yl acetate 2

The reaction of penta-3,4-dienyl sulfamate (**1**) according to General Procedure A and purification by column chromatography (ether) afforded the *title compound* (63 mg, 35%) as a colourless oil. R_f 0.14 (ether); ν_{max} (thin film)/cm^{-1} 3627 br, 1726 br, 1564 s, 1374 s, 1244 s; δ_H (400 MHz, $CDCl_3$) 2.19 (3 H, s, C*H*$_3$COCH), 2.21 (3 H, s, CH_3CO_2), 2.26–2.34 (2 H, m, C*H*$_2$CHO), 4.22–4.33 (2 H, m, CH_2O), 5.19 (1 H, dd, *J* 8.3, 4.2, C*H*OAc); δ_C (100 MHz, $CDCl_3$) 20.5, 26.1, 29.5, 66.3, 74.8, 170.7, 205.3; HRMS (ESI$^+$) found 262.0354, $C_7H_{13}NNaO_6S$ (MNa^+) requires 262.0356.

(*E*)-4-(2-Methylpropylidene)-2,2-dioxido-1,2,3-oxathiazepan-5-yl acetate 5

The reaction of 6-methylhepta-3,4-dienyl sulfamate (**4**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (382 mg, 52%) as a white crystalline solid. R_f 0.52 (ether); m.p. 87 °C; ν_{max} (thin film)/cm^{-1} 3272 br, 2965 s, 2872 w, 1743 s, 1467 s, 1418 s, 1375 s; δ_H (400 MHz, $CDCl_3$) 1.04 (3 H, d, *J* 6.6) and 1.05 (3 H, d, *J* 6.7, *Me*$_2$CH), 2.10–2.16 (1 H, m, C*H*H′CH$_2$O) overlays 2.13 (3 H, s, CH_3CO), 2.20 (1 H, dt, *J* 11.6, 3.3, CH*H*′CH$_2$O), 2.59–2.68 (1 H, m, Me$_2$C*H*), 4.25 (1 H, dt, *J* 12.3, 3.3) and 4.64 (1 H, dt, *J* 12.3, 1.3, CH_2OS), 5.77 (1 H, d, *J* 10.5, CH=), 5.92 (1 H, t, *J* 3.4, C*H*OAc), 6.28 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 21.1, 22.4, 22.6, 27.1, 34.7, 65.1, 66.7, 125.6, 143.0, 169.3; HRMS (ESI$^+$) found 286.0719, $C_{10}H_{17}NNaO_5S$ (MNa^+) requires 286.0720.

4-Methyl-2,2-dioxido-5-phenyl-6,7-dihydro-5H-1,2,3-oxathiazepin-5-yl acetate 7

The reaction of 3-phenylpenta-3,4-dienyl sulfamate (**6**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (135 mg, 24%) as a white crystalline solid. R_f 0.32 (ether); m.p. 118 °C; ν_{max} (thin film)/cm^{-1} 3068 s, 1745 s, 1638 s, 1448 m, 1368 s, 1258 w, 1234 s, 1180 s; δ_H (500 MHz, $CDCl_3$) 2.07 (1 H, ddd, *J* 14.8, 4.1, 1.1, C*H*H′CH$_2$O), 2.20 (3 H, s, CH_3CO), 2.24 (3 H, s, CH_3C=N), 3.43–3.51 (1 H, m, C*H*H′O), 4.06–4.15 (1 H, m, CH*H*′CH$_2$O), 4.30 (1 H, dd, *J* 11.0, 5.9, C*H*H′O), 7.37 (2 H, dd, *J* 7.7, 1.9) and 7.45–7.48 (3 H, m, Ph); δ_C (100 MHz, $CDCl_3$) 21.3, 25.2, 36.7, 65.2, 88.4, 125.9, 129.3, 129.8, 133.6, 169.8, 181.4; HRMS (ESI$^+$) found 320.0558, $C_{13}H_{15}NNaO_5S$ (MNa^+) requires 320.0563. Also obtained was **(2,2-dioxido-5-phenyl-6,7-dihydro-3H-1,2,3-oxathiazepin-4-yl)methyl acetate** (**8**) (26 mg, 5%) as a yellow solid. R_f 0.49 (ether); ν_{max} (thin film)/cm^{-1} 3377 br, 2919s, 2850 m, 1738 s, 1659 w, 1443 m, 1411 s; δ_H (400 MHz, $CDCl_3$) 2.11 (3 H, s, CH_3), 3.00 (2 H, t, *J* 5.0, C*H*$_2$CH$_2$O), 4.51 (2 H, app. tt, *J* 3.5, 1.5, CH_2O), 4.55 (2 H, s, CH_2OAc), 7.20–7.22 (2 H, m) and 7.34–7.41 (3 H, m, Ph); δ_C (100 MHz, $CDCl_3$) 20.8, 36.4, 63.1, 70.5, 128.0, 128.3, 128.6, 128.8, 135.9, 139.4, 171.4; HRMS (ESI$^+$) found 320.0560, $C_{13}H_{15}NNaO_5S$ (MNa^+) requires 320.0563.

7-Methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]oct-1-yl acetate 10

The reaction of 3-methylpenta-3,4-dienyl sulfamate (**9**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (236 mg, 49%) as a white crystalline solid. R_f 0.21 (petrol/ether, 2:1); m.p. 91 °C; ν_{max} (thin film)/cm^{-1} 3303 br, 2973 s, 2922 s, 2865 m, 1741 m, 1455 m, 1423 s, 1345 s; δ_H (400 MHz, $CDCl_3$) 1.03 (1 H, d, *J* 6.6, C*H*H′C(OAc)), 1.35 (3 H, s, C*H*$_3$C(CH$_2$)), 1.38 (1 H, d, *J* 6.6, CH*H*′C(OAc)), 1.93 (1 H, ddd, *J* 16.0, 12.0, 2.0, C*H*H′CH$_2$O), 2.10 (3 H, s, CH_3CO), 2.29 (1 H, dd, *J* 16.0, 4.6, CH*H*′CH$_2$O), 4.26 (1 H, ddd, *J* 12.0, 4.6, 2.0) and 4.67 (1 H, app. t, *J* 12.0, , CH_2O), 6.75 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 17.7, 21.2, 27.6, 31.1, 38.7, 69.2, 77.2, 171.6; HRMS (ESI$^+$) found 258.0407, $C_8H_{13}NNaO_5S$ (MNa^+) requires 258.0407.

7-Propyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]oct-1-yl acetate 12

The reaction of 3-(vinylidene)hexyl sulfamate (**11**) according to General Procedure A and purification by column chromatography (petrol/ether, 1:1) afforded the *title compound* (167 mg, 40%) as a white crystalline solid. R_f 0.42 (ether); m.p. 79 °C; ν_{max} (thin film)/cm^{-1} 3260 br, 2962 s, 1740 s, 1510 m, 1363 m, 1211 s, 1191 s; δ_H (400 MHz, $CDCl_3$) 0.95 (3 H, t, *J* 7.3 C*H*$_3$CH$_2$), 1.02 (1 H, d, *J* 6.6) and 1.34 (1 H, d, *J* 6.6, C*H*H′C(OAc)), 1.30–1.48 (2 H, m, C*H*H′Et overlays C*H*H′CH$_3$), 1.52–1.63 (1 H, m, CH*H*′CH$_3$), 1.70 (1 H, ddd, *J* 11.1, 4.3, 3.0, CH*H*′Et), 1.80 (1 H, dd, *J* 16.0, 11.2, C*H*H′CH$_2$O), 2.11 (3 H, s, CH_3CO), 2.42 (1 H, dd, *J* 16.0, 4.8, CH*H*′CH$_2$O), 4.24 (1 H, ddd, *J* 12.0, 4.8, 2.0) and 4.64 (1 H, app. t, *J* 12.0, CH_2O), 6.72 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 14.2, 19.8, 21.2, 30.2, 31.7, 32.4, 35.4, 69.5, 69.6, 171.6; HRMS (FI) found 263.0820, $C_{10}H_{17}NO_5S$ (M^+) requires 263.0827.

7-(1-Methylethyl)-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 14

The reaction of 3-isopropylpenta-3,4-dienyl sulfamate (**13**) according to General Procedure A and purification by column chromatography (dichloromethane/ethyl acetate, 20:1) afforded the *title compound* (184 mg, 20%) as a white crystalline solid. R_f 0.20 (petrol/ethyl acetate, 2:1); m.p. 167 °C; ν_{max} (thin film)/cm^{-1} 3455 s, 3211 br, 2958 m, 1466 w, 1416 s, 1366 s, 1321 m, 1262 s, 1202 s, 1169 s; δ_H (400 MHz, $CDCl_3$) 0.88 (1 H, d, *J* 5.7) and 0.92 (1 H, d, *J* 5.7, C*H*H′C(OAc)), 1.01

(3 H, d, *J* 7.0) and 1.14 (3 H, d, *J* 7.0, *Me*$_2$CH), 1.68 (1 H, dddd, *J* 16.5, 11.6, 2.0, 0.4, C*H*H′CH$_2$O), 1.79 (1 H, dsept, *J* 7.0, 0.4, Me$_2$C*H*), 2.48 (1 H, dd, *J* 16.5, 4.6, CH*H*′CH$_2$O), 3.62 (1 H, br s, OH), 4.25 (1 H, ddd, *J* 12.3, 4.6, 2.0) and 4.69 (1 H, ddd, *J* 12.3, 11.6, 0.4, CH$_2$O), 6.05 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 19.5, 20.5, 30.5, 31.0, 32.0, 37.8, 70.6, 71.6; HRMS (ESI$^+$) found 244.0612, $C_8H_{15}NNaO_4S$ (MNa^+) requires 244.0614. Also obtained was **[5-(1-methyl)-2,2-dioxido-6,7-dihydro-3H-1,2,3-oxathiazepin-4-yl]methyl acetate** (**15**) (137 mg, 12%) as a white crystalline solid. R_f 0.24 (petrol/ethyl acetate, 2:1); ν_{max} (thin film)/cm^{-1} 3248 br, 2965 s, 1737 s, 1420 m, 1364 s, 1173 s; δ_H (400 MHz, $CDCl_3$) 1.02 (6 H, d, *J* 6.8, *Me*$_2$CH), 2.09 (3 H, s, CH_3CO), 2.56–2.59 (2 H, m, C*H*$_2$CH$_2$O), 2.96 (1 H, sept, *J* 6.8, Me$_2$C*H*), 4.33–4.36 (2 H, m, CH$_2$O), 4.68 (2 H, s, CH$_2$OAc), 6.39 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 20.1, 20.8, 27.8, 30.2, 62.2, 70.8, 125.8, 142.6, 171.4; HRMS (ESI$^-$) found 262.0748, $C_{10}H_{16}NO_5S$ $(M-H)^-$ requires 262.0755.

6-(*tert*-Butyl)-3-oxa-2-thia-1-azabicyclo[5.1.0]oct-6-ene 2,2-dioxide 17

The reaction of 3-*tert*-butylpenta-3,4-dienyl sulfamate (**16**) according to General Procedure A and purification by column chromatography (petrol/ether, 4:1) afforded the *title compound* (160 mg, 68%) as a white crystalline solid; R_f 0.26 (petrol/ether, 2:1); m.p. 58 °C; ν_{max} (thin film)/cm^{-1} 3075m, 2968s, 1468m, 1359s, 1296w, 1261w, 1183s; δ_H (500 MHz, CD_2Cl_2, 228K) 1.11 (9H, s, $(CH_3)_3C$), 2.39 (1H, d, *J* 14.3, OCH$_2$CH*H*′), 2.90 (1H, app. t, 14.3, (OCH$_2$C*H*H′), 3.45 (1H, br s, NCH*H*′), 3.53 (1H, br s, NC*H*H′), 4.48-4.57 (2H, m, OCH$_2$); δ_C (100 MHz, $CDCl_3$) 27.9, 28.0, 35.3, 40.2, 75.3, 114.9, 126.4; HRMS (ESI$^+$) found 240.0670, $C_9H_{15}NNaO_3S$ (MNa^+) requires 240.0665. Also obtained was **6-(*tert*-butyl)-7-methylidene-3-oxa-2-thia-1-azabicyclo[4.1.0]heptane 2,2-dioxide** (**18**) (31 mg, 13%). R_f 0.26 (petrol/ether, 2:1); ν_{max} (thin film)/cm^{-1} 3298 br, 2972 s, 2875 m, 1754 s, 1626 m, 1468 s, 1371 s, 1187 s; δ_H (500 MHz, $CDCl_3$) 1.06 (9 H, s, $(CH_3)_3C$), 2.11 (1 H, dt, *J* 15.2, 3.4) and 2.49 (1 H, ddd, 15.2, 10.8, 5.3, CH$_2$CH$_2$O), 4.36 (1 H, ddd, *J* 12.0, 10.8, 3.4) and 4.43 (1 H, ddd, *J* 12.0, 5.3, 3.4, CH$_2$O), 5.04 and 5.36 (2 × 1 H, 2 × d, *J* 2.8, CH$_2$=); δ_C (125 MHz, $CDCl_3$) 21.3, 25.5, 34.9, 66.7, 67.3, 90.0, 133.2; HRMS (ESI$^+$) found 240.0661, $C_9H_{15}NNaO_3S$ (MNa^+) requires 240.0665.

(1*S,7*S**,8*S**)-7-Ethyl-8-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl acetate 20**

The reaction of 3-ethylhexa-3,4-dienyl sulfamate (**19**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (170 mg, 26%) as a white crystalline solid. R_f 0.38 (ether); m.p. 104 °C; ν_{max} (thin film)/cm^{-1} 3299 br, 2975 s, 1741 s, 1411 s, 1357 s, 1256 m, 1189 s; δ_H (500 MHz, $CDCl_3$) 0.99 (3 H, d, *J* 6.5, C*H*$_3$CH), 1.00 (3 H, t, *J* 7.6 C*H*$_3$CH$_2$), 1.40 (1 H, q, *J* 6.5, C*H*C(OAc)), 1.50–1.58 (1 H, m, C*H*H′CH$_3$), 1.65–1.75 (2 H, m, C*H*H′CH$_2$O overlays CH*H*′CH$_3$), 2.13 (3 H, s, CH_3CO), 2.50 (1 H, dd, *J* 16.2, 4.6, CH*H*′CH$_2$O), 4.23 (1 H, ddd, *J* 12.0, 4.6, 2.0) and 4.63 (1 H, app. t, *J* 12.0, CH$_2$O), 6.64 (1 H, br s, NH); δ_C (125 MHz, $CDCl_3$) 8.1, 10.2, 18.4, 20.9, 33.2, 33.4, 36.0, 69.2, 71.8, 171.5; HRMS (ESI$^+$) found 286.0716, $C_{10}H_{17}NNaO_5S$ (MNa^+) requires 286.0720. Also obtained was **1-(5-ethyl-2,2-dioxido-6,7-dihydro-3*H*-1,2,3-oxathiazepin-4-yl)ethyl acetate** (**21**) (180 mg, 28%) as a white crystalline solid. R_f 0.30 (ether); ν_{max} (thin film)/cm^{-1} 3281 br, 2975 s, 2259 s, 1734 s, 1371 s; δ_H (500 MHz, $CDCl_3$) 1.04 (3 H, t, *J* 7.6, C*H*$_3$CH$_2$), 1.40 (3 H, d, *J* 6.6, C*H*$_3$CH), 2.05 (3 H, s, C*H*$_3$CO), 2.13–2.19 (1 H, m) and 2.25–2.32 (1 H, m, C*H*$_2$CH$_3$), 2.48 (1 H, ddd, *J* 16.2, 6.8, 1.7) and 2.74 (1 H, ddd, *J* 16.2, 8.4, 2.7, C*H*$_2$CH$_2$O), 4.34–4.42 (2 H, m, CH$_2$O), 5.74 (1 H, q, *J* 6.7, C*H*OAc), 6.23 (1 H, br s, NH); δ_C (125 MHz, $CDCl_3$) 12.3, 18.4, 21.1, 26.9, 33.7, 36.4, 70.5, 128.6, 136.2, 169.9; HRMS (ESI$^+$) found 286.0717, $C_{10}H_{17}NNaO_5S$ (MNa^+) requires 286.0720.

7-(*tert*-Butyl)-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 26

To a stirred solution of methylene aziridine **17** (47 mg, 0.210 mmol) in DMF (1.5 mL) was added NaI (32 mg, 0.210 mmol) at RT. After 20 h the reaction was quenched with sat. aq. NH_4Cl solution (5 mL) and the mixture was extracted with ether (3 × 20 mL). The combined extracts were washed with brine (10 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Column chromatography (petrol/ether, 2:1) afforded the *title compound* as a white crystalline solid (20 mg, 41%). R_f 0.12 (petrol/ethyl acetate, 2:1); m.p. 132 °C; ν_{max} (thin film)/cm^{-1} 3460 s, 3180 br, 2958 s, 1457 s, 1265 s, 1144 s; δ_H (400 MHz, $CDCl_3$) 0.75 (1 H, d, *J* 6.2, C*H*H′C(OH)), 1.12 (9 H, s, $(CH_3)_3C$), 1.47 (1 H, d, *J* 6.2, CH*H*′C(OH)), 1.67 (1 H, ddd, *J* 17.0, 11.2, 1.6) and 2.69 (1 H, dd, *J* 17.0, 5.3, C*H*$_2$CH$_2$O), 3.61 (1 H, br s, OH), 4.27 (1 H, ddd, *J* 12.3, 5.3, 1.6) and 4.76 (1 H, app. t, *J* 11.8, CH$_2$O), 6.05 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 26.7, 29.8, 34.1, 36.3, 40.1, 71.1, 71.8; HRMS (ESI$^+$) found 258.0769, $C_9H_{17}NNaO_4S$ (MNa^+) requires 258.0770.

7-Methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 27

To a stirred solution of cyclopropane **10** (78 mg, 0.33 mmol) in isopropanol (2 mL) was added $NaBH_4$ (50 mg, 1.32 mmol) at 0 °C and the reaction mixture was allowed to warm to RT. After 20 h the reaction was quenched with water (5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic extracts were washed with brine (10 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Column chromatography (petrol/ethyl acetate, 4:1) afforded the *title compound* as a white solid (36 mg, 62%). R_f 0.28 (petrol/ethyl acetate, 2:1); m.p. 91 °C; ν_{max} (thin film)/cm^{-1} 3300 br, 2961 s, 1338 s, 1163 s; δ_H (500 MHz, $CDCl_3$) 0.83 (1 H, dd, *J* 5.5, 3.7) and 0.93 (1 H, dd, *J* 7.3, 5.5, C*H*$_2$CH), 1.17 (3 H, s, CH_3), 2.00 (1 H, ddd, *J* 16.2, 11.2, 1.4) and 2.21 (1 H, dd, *J* 16.2, 4.7, C*H*$_2$CH$_2$O), 2.45 (1 H, ddd, *J* 7.3, 3.7, 2.0, C*H*NH), 4.20 (1 H, ddd, *J* 12.3, 4.7, 1.4) and 4.69 (1 H, app. t, *J* 11.7, CH$_2$O), 5.16 (1 H, br s, NH); δ_C (125 MHz, $CDCl_3$) 22.2, 24.2, 24.7, 34.9, 37.8, 69.8; *m/z* (CI) 195 (MNH_4^+, 100%); HRMS (CI) found 195.0795, $C_6H_{15}NO_3S$ (MNH_4^+) requires 195.0803.

7-Methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 28

To a solution of cylopropane **10** (45 mg, 0.191 mmol) in ether (6 mL) at RT was added $LiAlH_4$ (22 mg, 0.574 mmol) and the mixture was stirred for 1 h. The reaction was quenched by the addition of water (10 mL) and acidified with 1 M hydrochloric acid (5 mL). The mixture was extracted with ether (3 × 20 mL) and the combined organic extracts were washed with brine (10 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 3:1) afforded the *title compound* as a white solid (28 mg, 76%). R_f 0.34 (petrol/ethyl acetate, 2:1); m.p. 92 °C; ν_{max} (thin film)/cm^{-1} 3259 br, 2961 s, 1710 w, 1421 s, 1266 s, 1120 s, 1160 s; δ_H (400 MHz, $CDCl_3$) 0.99 (1 H, d, *J* 6.6) and 1.06 (1 H, d, *J* 6.6, C*H*$_2$C(OH)), 1.30 (3 H, s, CH_3), 1.86 (1 H, ddd, *J* 16.2, 11.6, 1.5) and 2.26 (1 H, dd, *J* 16.2, 4.6, C*H*$_2$CH$_2$O), 3.72 (1 H, br s, OH), 4.25 (1 H, ddd, *J* 11.8, 4.6, 1.5) and 4.63 (1 H, app. t, *J* 11.8, CH_2O), 6.14 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 17.2, 29.2, 29.4, 38.7, 68.5, 69.8; HRMS (ESI$^+$) found 216.0300, $C_6H_{11}NNaO_4S$ (MNa$^+$) requires 216.0301.

1-(Ethoxy)-7-methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 29

To a stirred solution of cyclopropane **10** (43 mg, 0.183 mmol) in ethanol (15 mL) at RT was added NaOH (146 mg, 3.66 mmol); after 30 min TLC analysis showed the reaction to be complete. The reaction mixture was diluted with water (20 mL), acidified with 1 M hydrochloric acid and extracted with dichloromethane (3 × 30 mL). The combined extracts were dried over Na_2SO_4 and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 2:1) afforded the *title compound* as a white crystalline solid (33 mg, 82%). R_f 0.15 (petrol/ethyl acetate, 2:1); m.p. 126 °C; ν_{max} (thin film)/cm^{-1} 3708 br, 2981 s, 2844 m, 1417 m, 1370 s, 1213 m, 1166 m; δ_H (400 MHz, $CDCl_3$) 0.89 (1 H, d, *J* 5.7) and 0.92 (1 H, d, *J* 5.7, C*H*$_2$C(OEt)), 1.23 (3 H, t, *J* 7.0, C*H*$_3$CH$_2$), 1.32 (3 H, s, CH_3C), 1.87 (1 H, ddd, *J* 16.2, 11.0, 2.0) and 2.29 (1 H, ddd, *J* 16.2, 5.0, 1.1, C*H*$_2$CH$_2$O), 3.41 (1 H, dq, *J* 9.0, 7.0) and 3.84 (1 H, dq, *J* 9.0, 7.0, C*H*$_2$CH$_3$), 4.21 (1 H, ddd, *J* 12.3, 5.0, 2.0) and 4.64 (1 H, ddd, *J* 12.0, 11.0, 1.1, CH_2O), 5.93 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 15.1, 18.0, 29.2, 30.9, 39.1, 61.9, 69.2, 72.8; HRMS (ESI$^+$) found 244.0615, $C_8H_{15}NNaO_4S$ (MNa$^+$) requires 244.0614.

1-Ethyl-7-methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 30

To a stirred solution of cyclopropane **10** (70 mg, 0.297 mmol) in THF (10 mL) at 0 °C was added ethylmagnesium bromide (0.89 mL of a 1.0 M solution in THF, 0.89 mmol). The reaction mixture was allowed to warm to RT and stirring was continued for 20 h. The reaction was quenched with sat. aq. NH_4Cl solution (10 mL) and extracted with ether (3 × 20 mL). The combined organic extracts were washed with brine (10 mL), dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 5:1) afforded the *title compound* as a white crystalline solid (42 mg, 68%). R_f 0.48 (petrol/ethyl acetate, 2:1); m.p. 88 °C; ν_{max} (thin film)/cm^{-1} 3299 br, 2962 s, 1421 s, 1332 s, 1164 s, 1105 m; δ_H (500 MHz, $CDCl_3$) 0.57 (1 H, d, *J* 5.5) and 0.80 (1 H, dd, *J* 5.5, 1.9, C*H*$_2$C(Et)), 0.94–1.01 (1 H, m, C*H*H′CH$_3$), 1.04 (3 H, t, *J* 6.9, C*H*$_3$CH$_2$), 1.24 (3 H, s, CH_3C), 2.03 (1 H, ddd, *J* 16.1, 11.0, 1.6) and 2.27 (1 H, dd, *J* 16.1, 4.7, C*H*$_2$CH$_2$O), 2.34–2.42 (1 H, m, CH*H*′CH$_3$), 4.19 (1 H, ddd, *J* 12.0, 4.7, 1.6) and 4.57 (1 H, dd, *J* 12.0, 11.0, CH_2O), 5.13 (1 H, br s, NH); δ_C (125 MHz, $CDCl_3$) 10.0, 19.3, 25.7, 29.3, 29.8, 40.5, 44.0, 69.0; HRMS (ESI$^+$) found 228.0667, $C_8H_{15}NNaO_3S$ (MNa$^+$) requires 228.0665.

Ethyl 2-(7-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl)-3-oxobutanoate 31

To a stirred solution of ethyl acetoacetate (28 mg, 0.212 mmol) in THF (2 mL) at 0 °C was added NaH (9 mg of a 60% suspension in mineral oil, 0.212 mmol). After 15 min cyclopropane **10** (30 mg, 0.106 mmol) was added and the solution was allowed to warm to RT and stirred for 18 h. The reaction was quenched with water (2 mL), acidified with 1 M hydrochloric acid (0.5 mL) and extracted with ether (3 × 5 mL). The combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (petrol/ether, 1:1) afforded the *title compound* as a mixture of inseparable diastereomers A and B (26 mg, 77%, A:B = 2.4:1). R_f 0.16 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 3301 br, 2983 s, 1716 s, 1619 m, 1414 s, 1178 s; δ_H (400 MHz, $CDCl_3$) 0.69 (1 H, d, *J* 6.9, C*H*H′C(EAA), A), 0.88 (1 H, d, *J* 6.6, C*H*H′C(EAA), B), 1.29 (3 H, s, CH_3C, B), 1.29 (3 H, s, CH_3C, A), 1.30 (3 H, t, *J* 7.1, C*H*$_3$CH$_2$, B), 1.31 (3 H, t, *J* 7.1, C*H*$_3$CH$_2$, A), 1.47 (1 H, d, *J* 6.6, CH*H*′C(EAA), B), 1.74 (1 H, d, *J* 6.9, CH*H*′C(EAA), A), 2.06–2.13 (1 H, m, C*H*H′CH$_2$O, A & B), 2.29 (3 H, d, *J* 1.0, CH_3CO, A), 2.33 (3 H, d, *J* 1.0, CH_3CO, B), 2.41 (1 H, dd, *J* 16.4, 8.7, CH*H*′CH$_2$O, A & B), 3.26 (1 H, d, *J* 1.0, C*H*COCH$_3$, A), 3.64 (1 H, br s, C*H*COCH$_3$, B), 4.07–4.15 (1 H, m, C*H*H′O, A & B), 4.19–4.28 (2 H, m, C*H*$_2$CH$_3$, A & B), 4.33–4.38 (1 H, m, CH*H*′O, A & B), 6.08 (1 H, br s, NH, B), 6.12 (1 H, br s, NH, A); δ_C (100 MHz, $CDCl_3$) data for A: 13.9, 22.0, 24.9, 28.7, 29.4, 37.1, 41.9, 62.1, 63.5, 67.2, 168.6, 202.4; HRMS (ESI$^+$) found 328.0822, $C_{12}H_{19}NNaO_6S$ (MNa$^+$) requires 328.0825.

Diethyl 2-(7-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl)malonate 32

To a stirred solution of diethyl malonate (37 mg, 0.230 mmol) in THF (2 mL) at 0 °C was added NaH (9 mg of a 60% suspension in mineral oil, 0.230 mmol). After 15 min cyclopropane **10** (27 mg, 0.114 mmol) was added and the solution was allowed to warm to RT and stirred for 18 h. The reaction was quenched with water (2 mL), acidified with 1M hydrochloric acid (0.5 mL) and extracted with ether (3 × 5 mL). The combined extracts were dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* as a

colourless oil (25 mg, 65%). R_f 0.20 (petrol/ether, 1:1); ν_{max} (thin film)/cm^{-1} 3313 br, 2984 s, 1729 s, 1391 s, 1369 s, 1313 s, 1238 s, 1175 s; δ_H (500 MHz, $CDCl_3$) 0.71 (1 H, d, *J* 6.3, C*H*H′C(DEM)), 1.30 (6 H, 2 × d, *J* 7.1, 2 × C*H*$_3$CH$_2$), 1.36 (1 H, app. s, CH*H*′C(DEM)), 1.37 (3 H, s, CH_3C), 2.11 (1 H, ddd, *J* 16.2, 8.8, 1.1) and 2.43 (1 H, ddd, *J* 16.2, 7.6, 1.0 C*H*$_2$CH$_2$O), 3.24 (1 H, s, C*H*(CO$_2$Et)$_2$), 4.12 (1 H, ddd, *J* 12.3, 7.6, 1.3, C*H*H′O), 4.19–4.42 (2 H, m) and 4.26–4.31 (2 H, m, 2 × C*H*$_2$CH$_3$), 4.43 (1 H, ddd, *J* 12.3, 8.8, 1.1, CH*H*′O), 6.24 (1 H, br s, NH); δ_C (100 MHz, $CDCl_3$) 13.8, 21.5, 27.2, 29.4, 38.5, 41.9, 56.1, 61.8, 67.7, 167.1, 168.7; HRMS (ESI$^+$) found 358.0925, $C_{13}H_{21}NNaO_7S$ (MNa$^+$) requires 358.0931.

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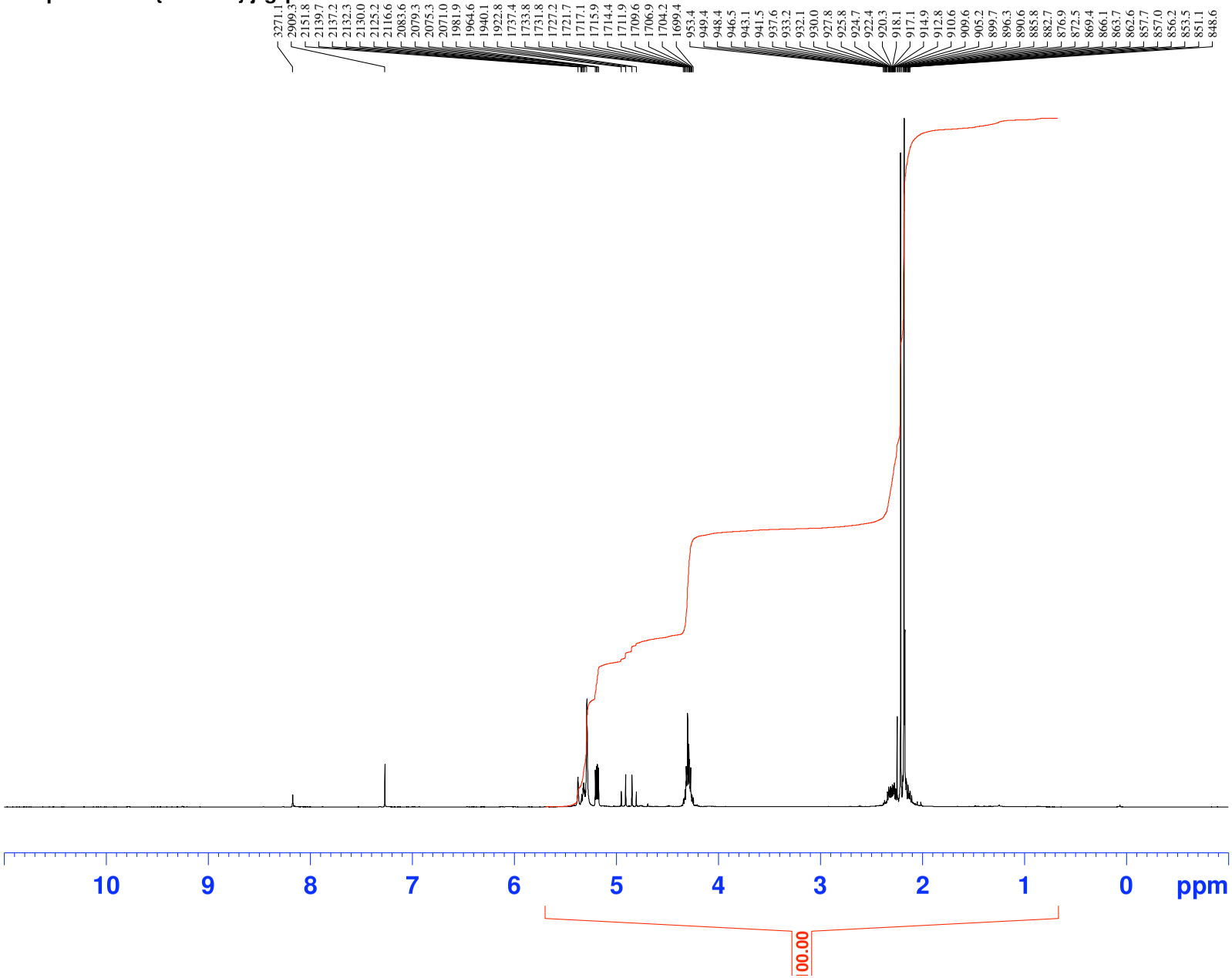


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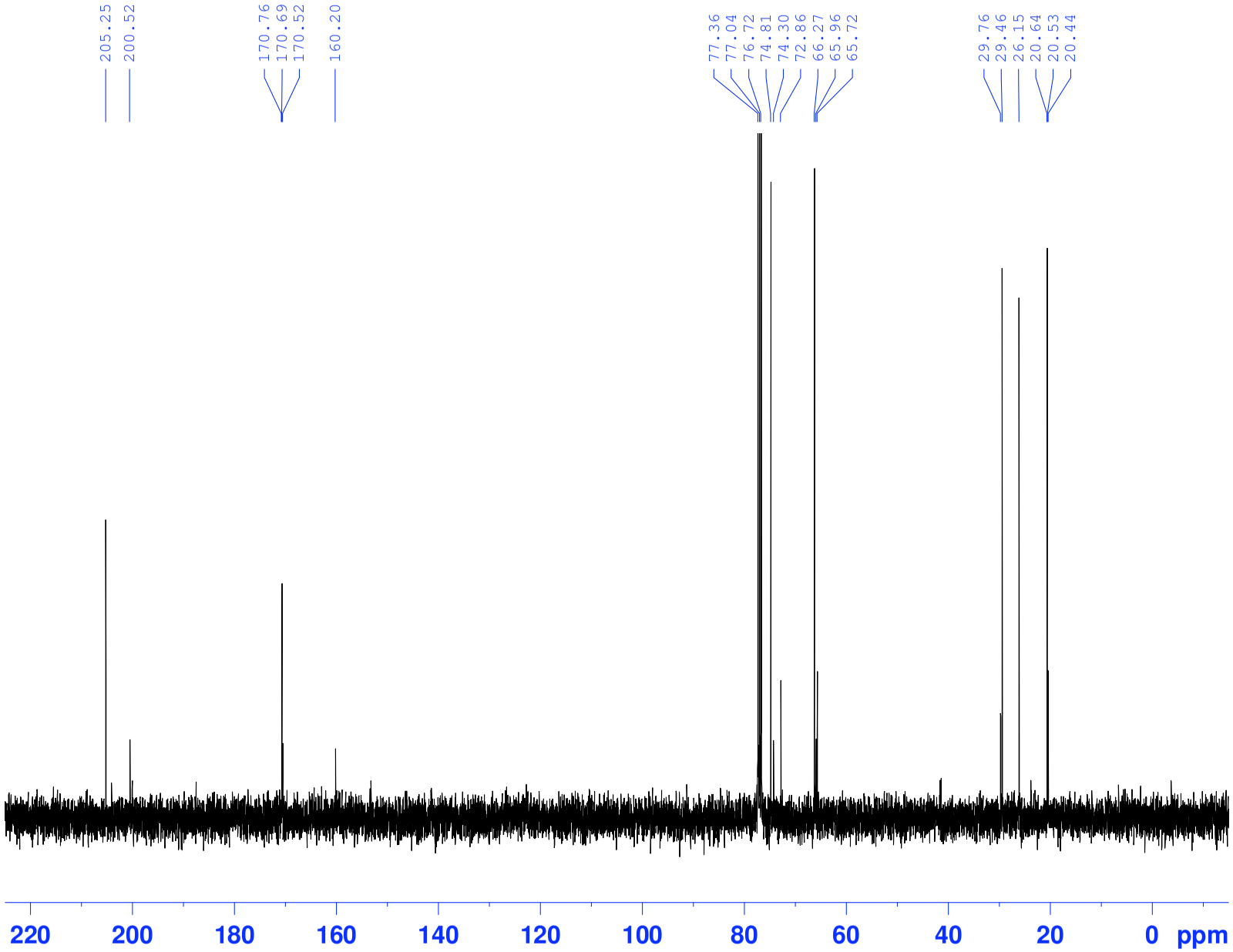

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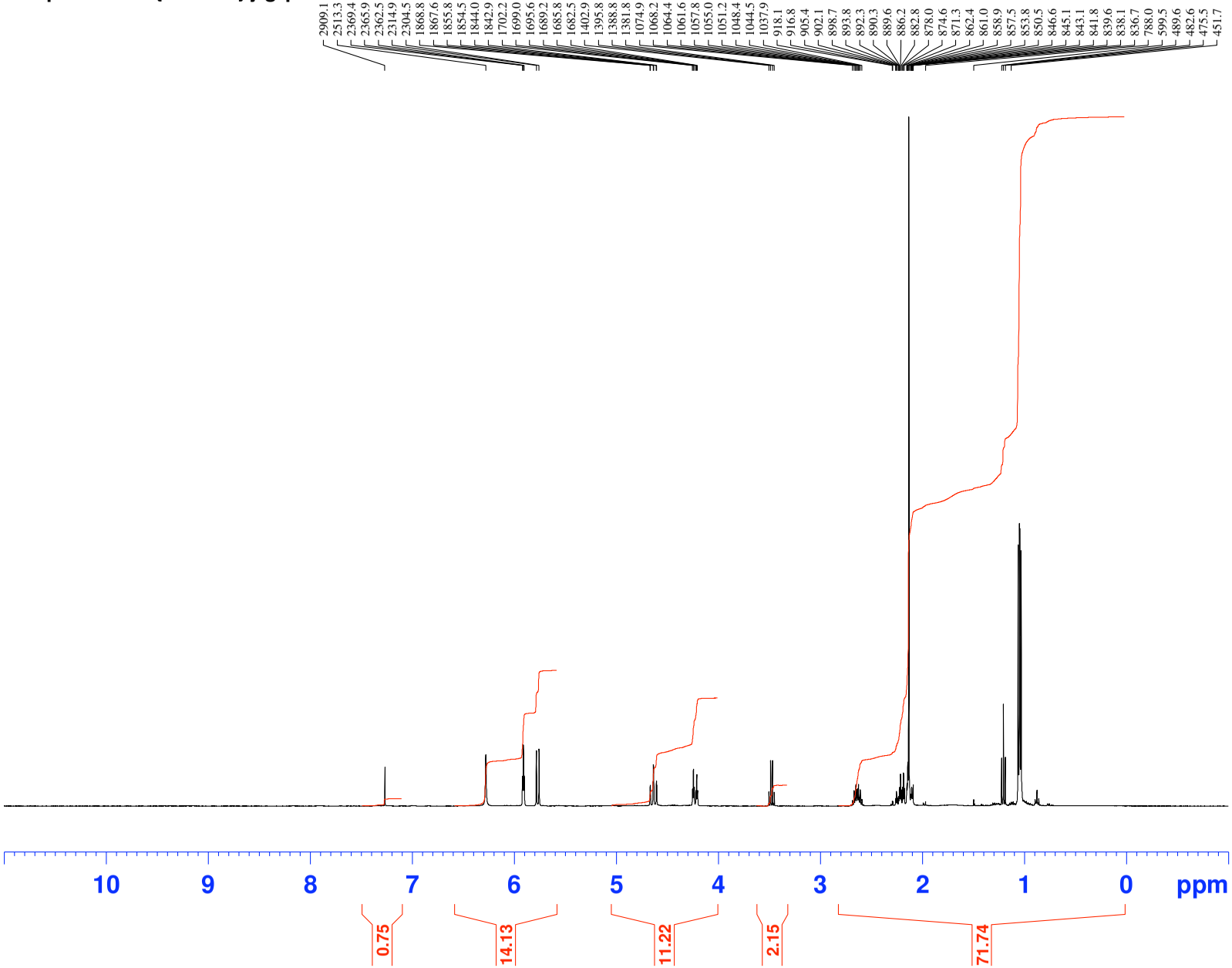

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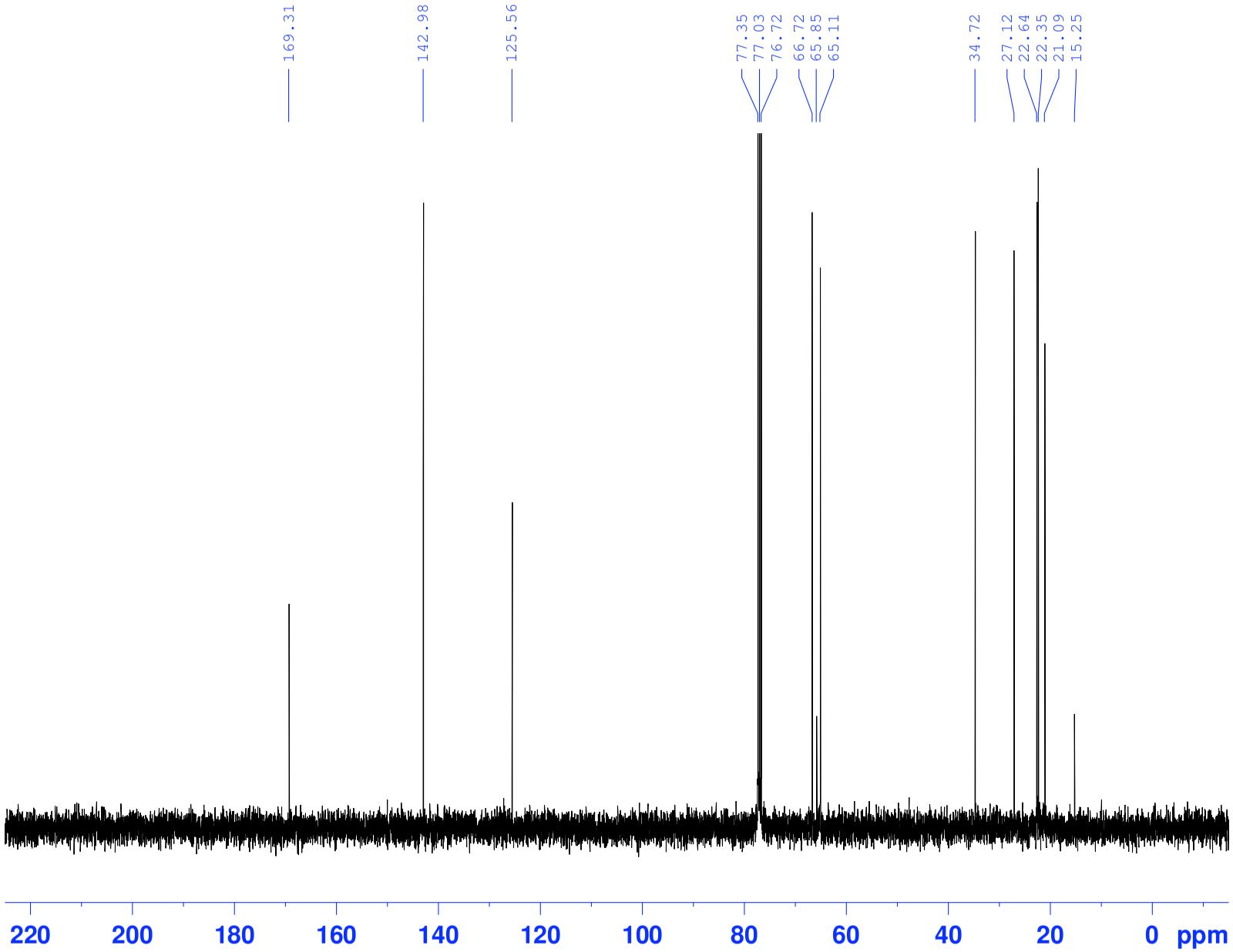

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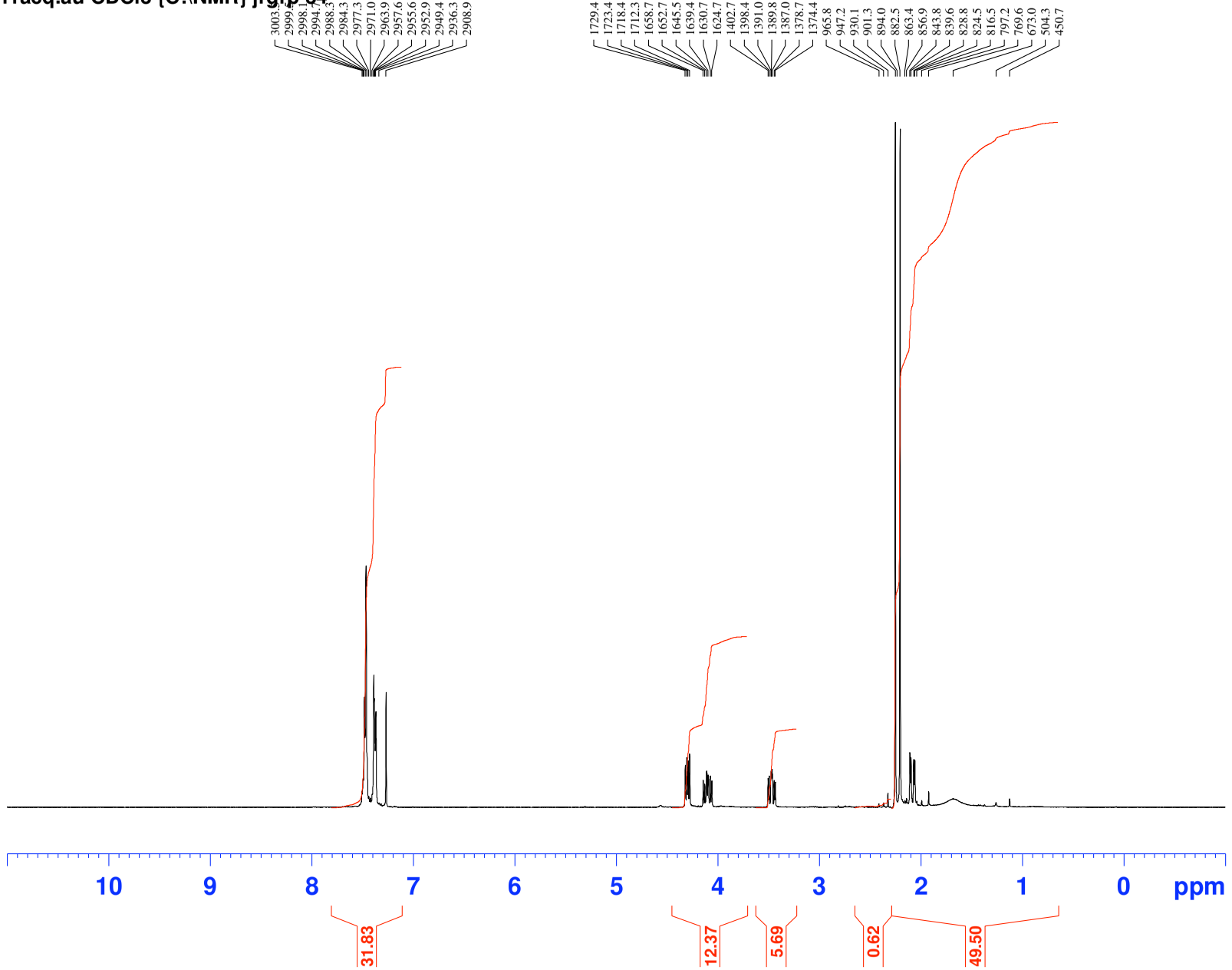

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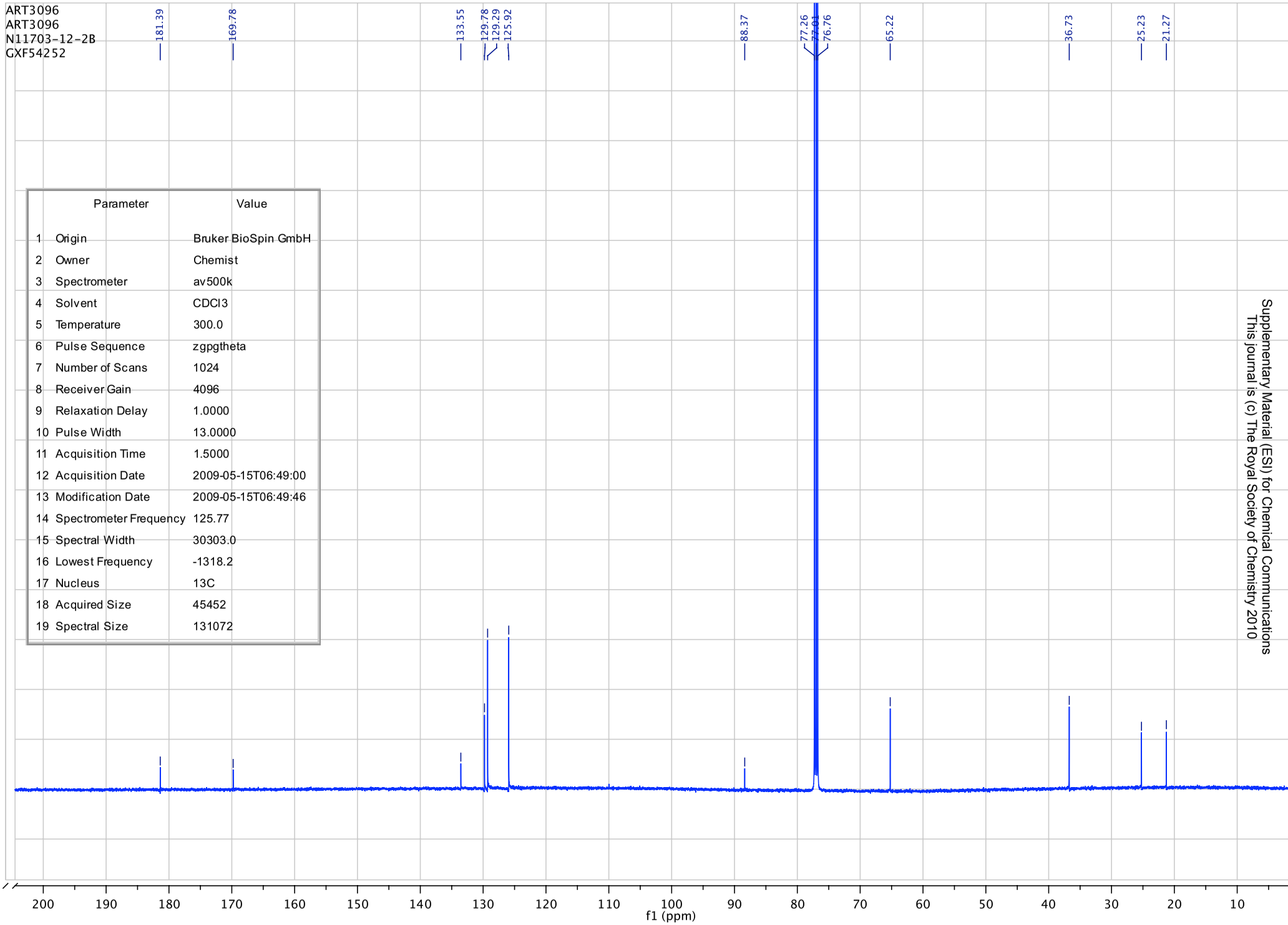

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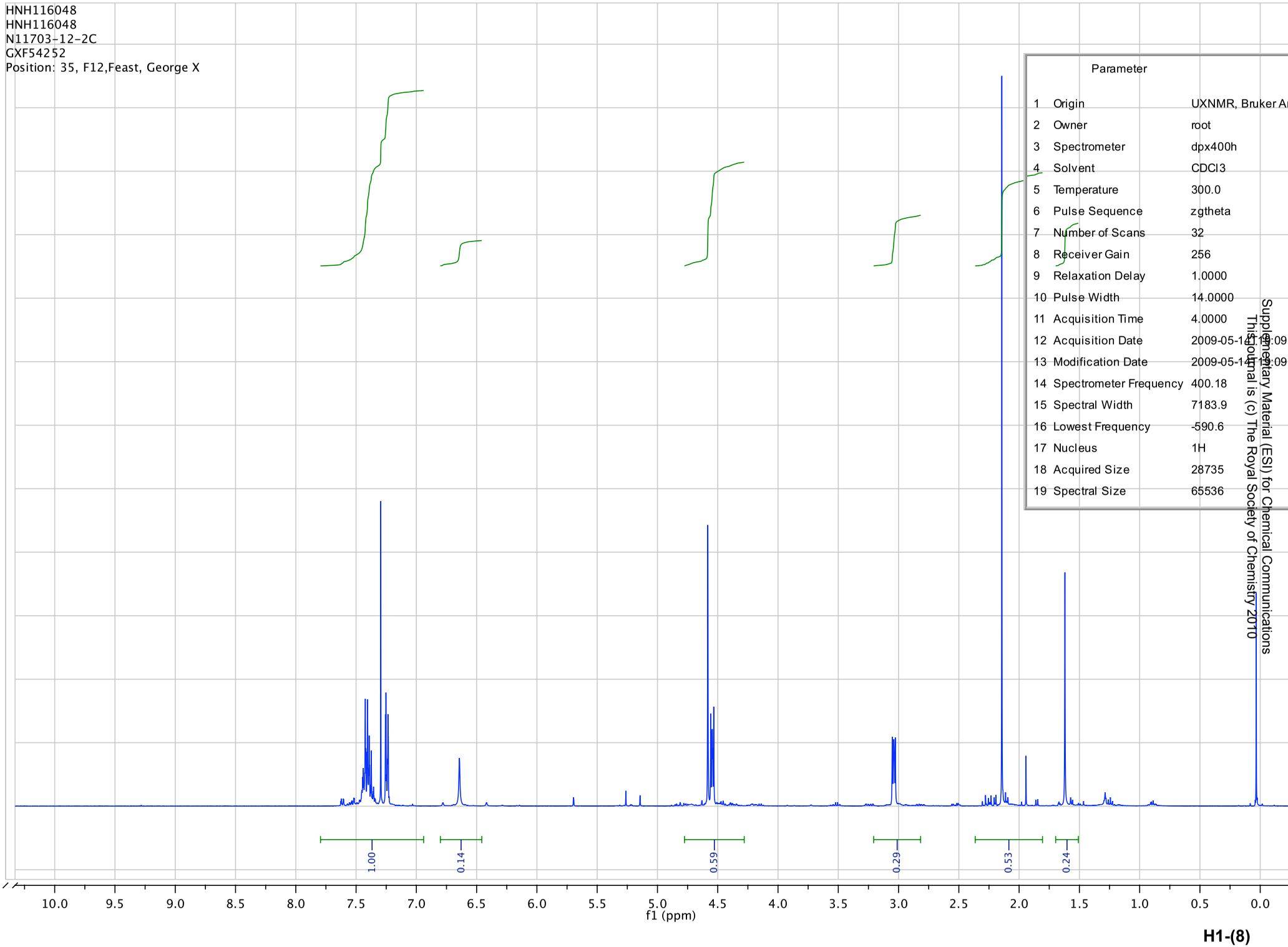

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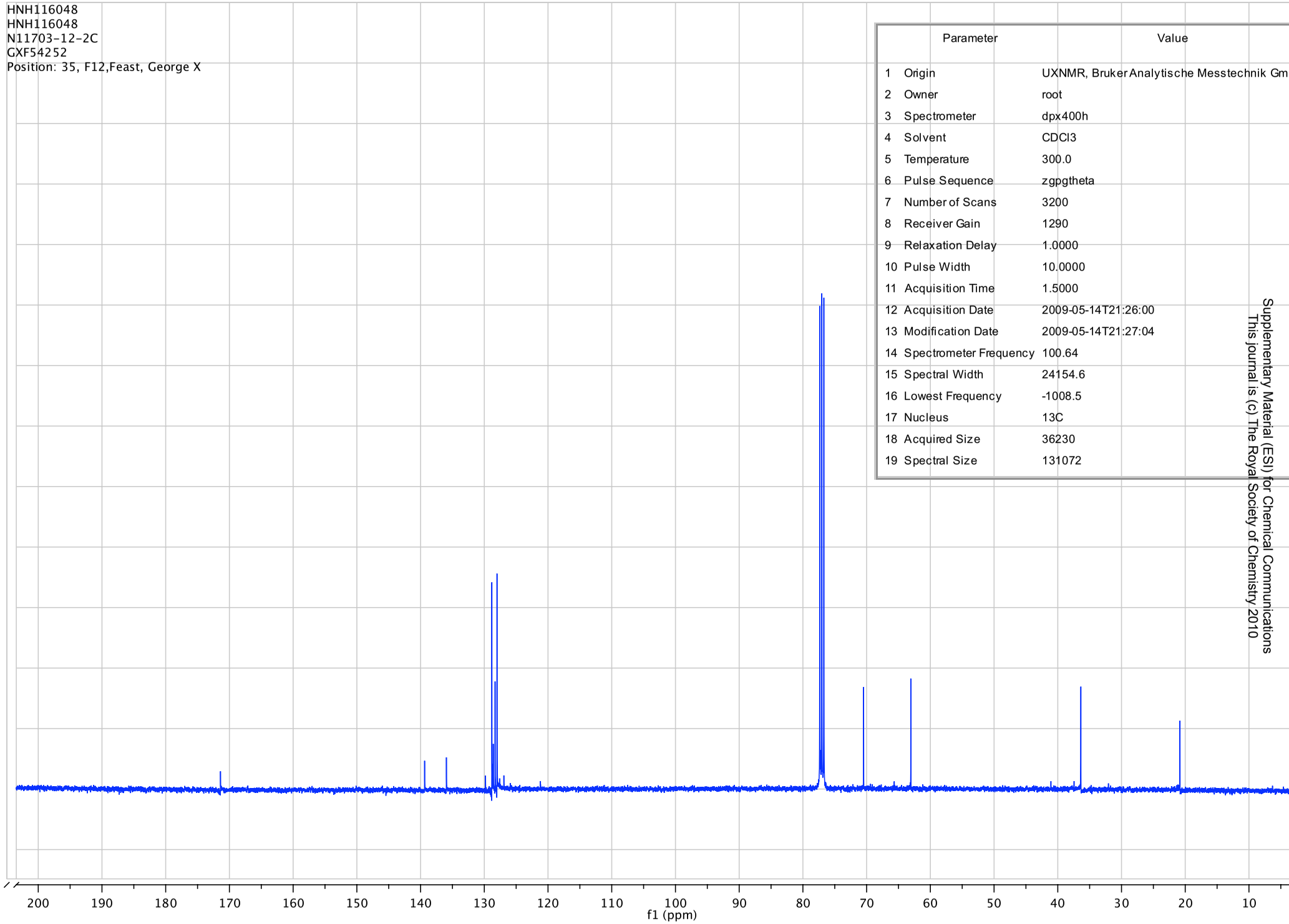

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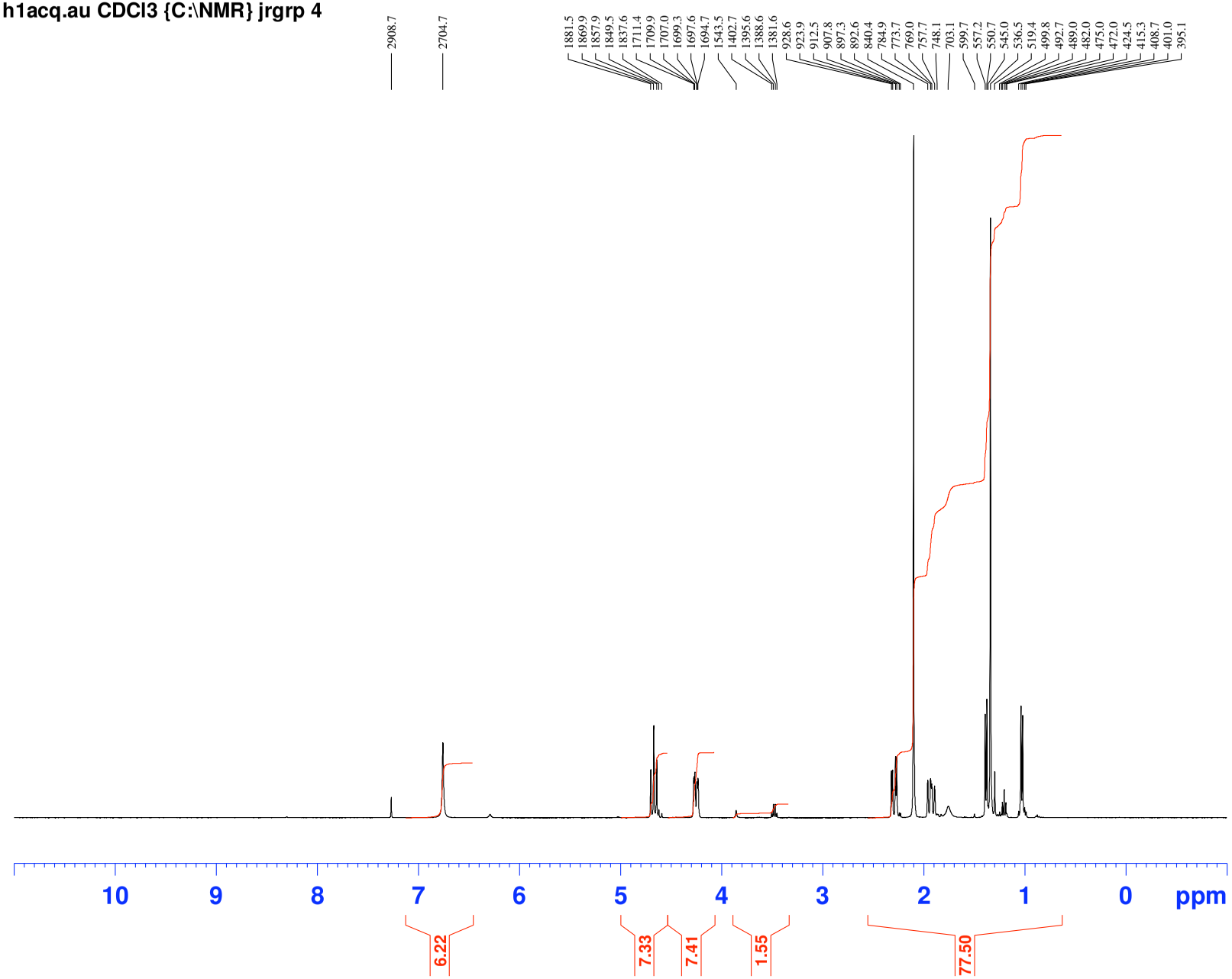

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Group JR
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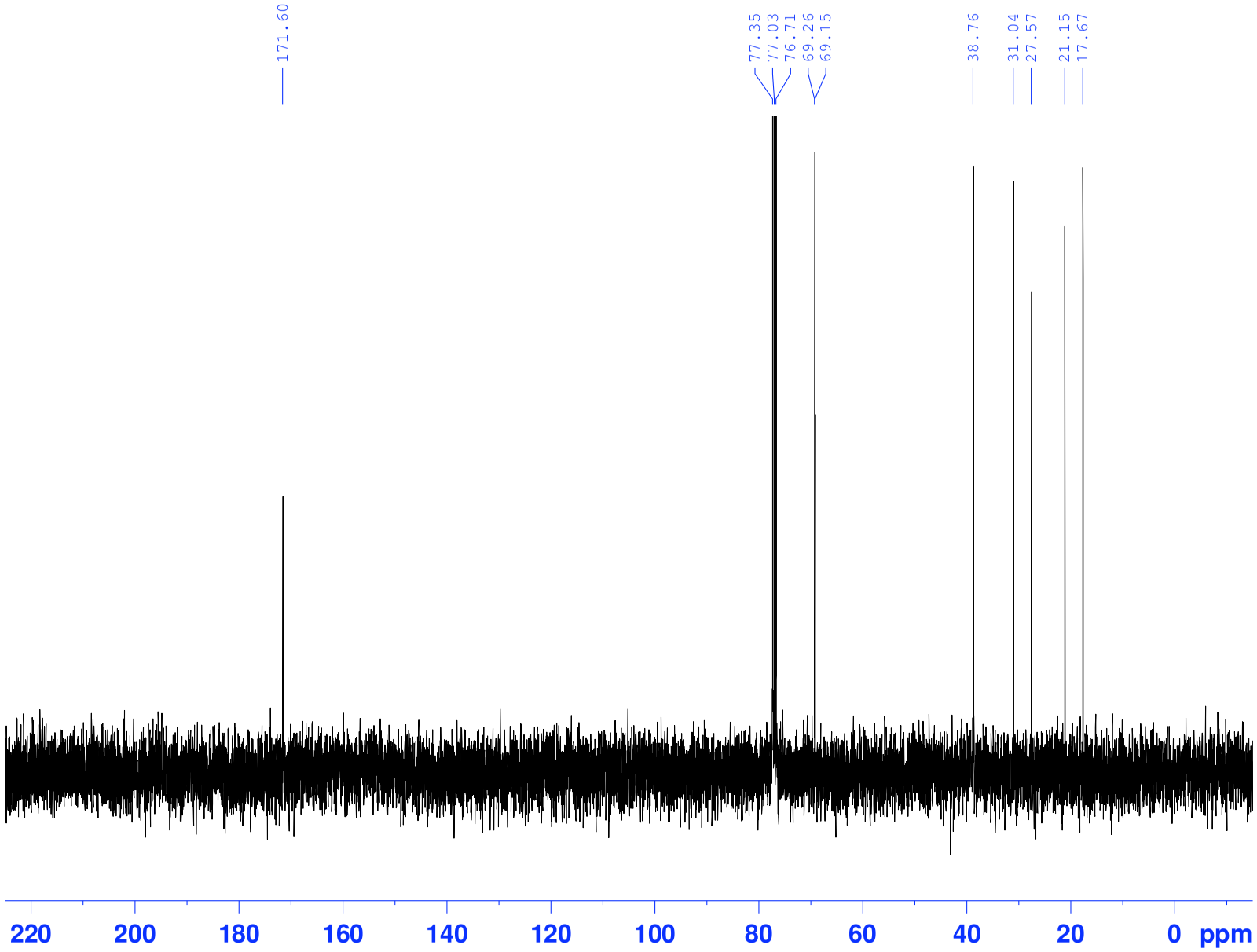

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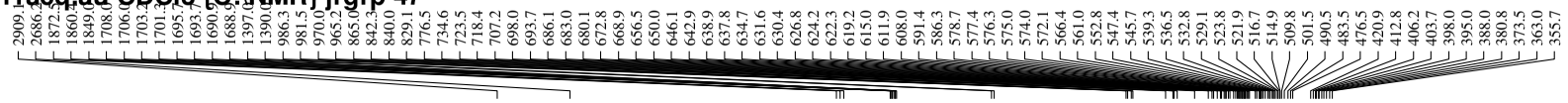
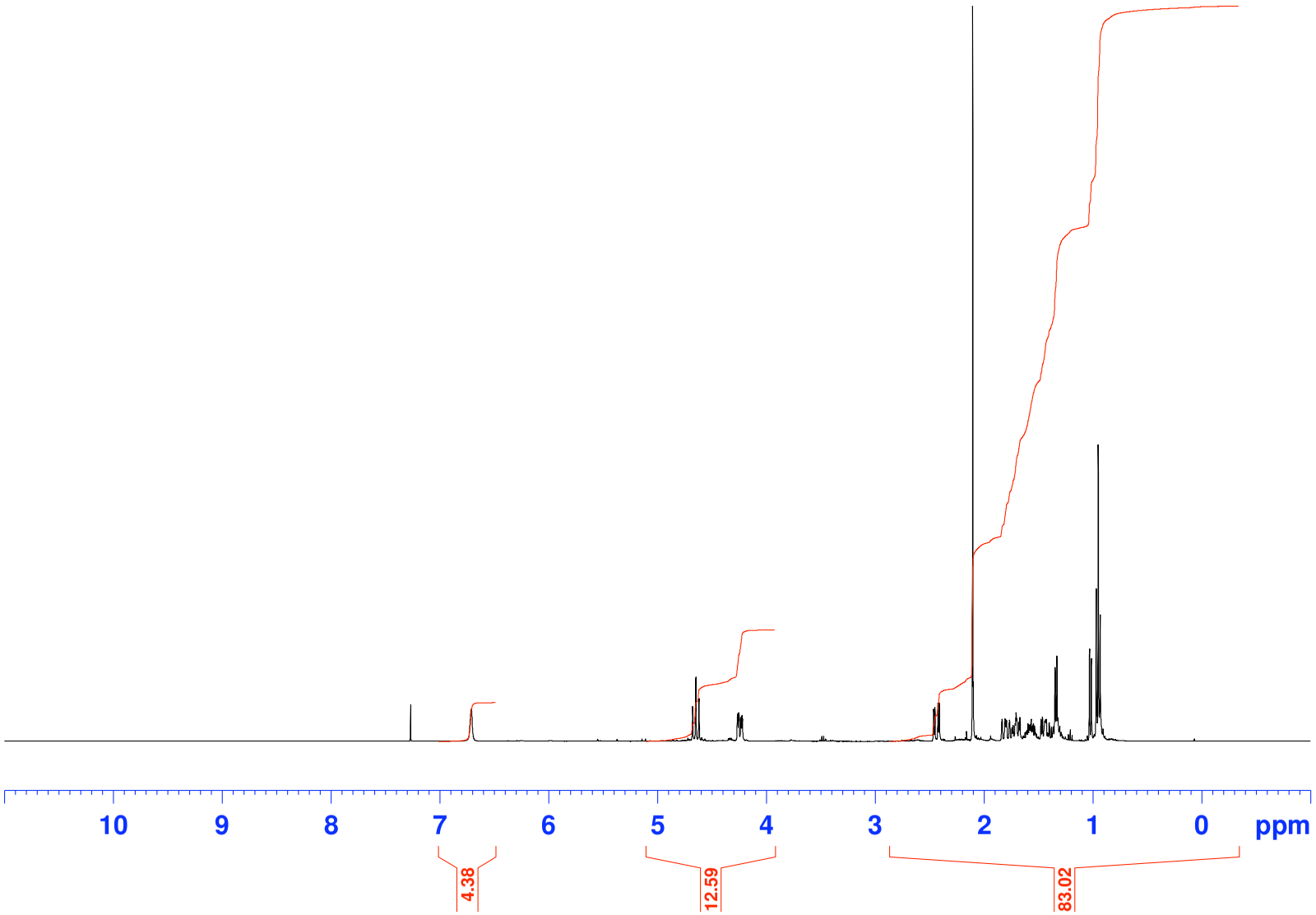
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	0.00 dB
SFO1	100.6403931 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	25.00 dB
SFO2	400.2016008 MHz
SI	32768
SF	100.6303718 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Instrument DQX400
Chemist GCF
Group JR
3–96
h1acq.au CDCl3 {C:\NMR} jrgrp 47

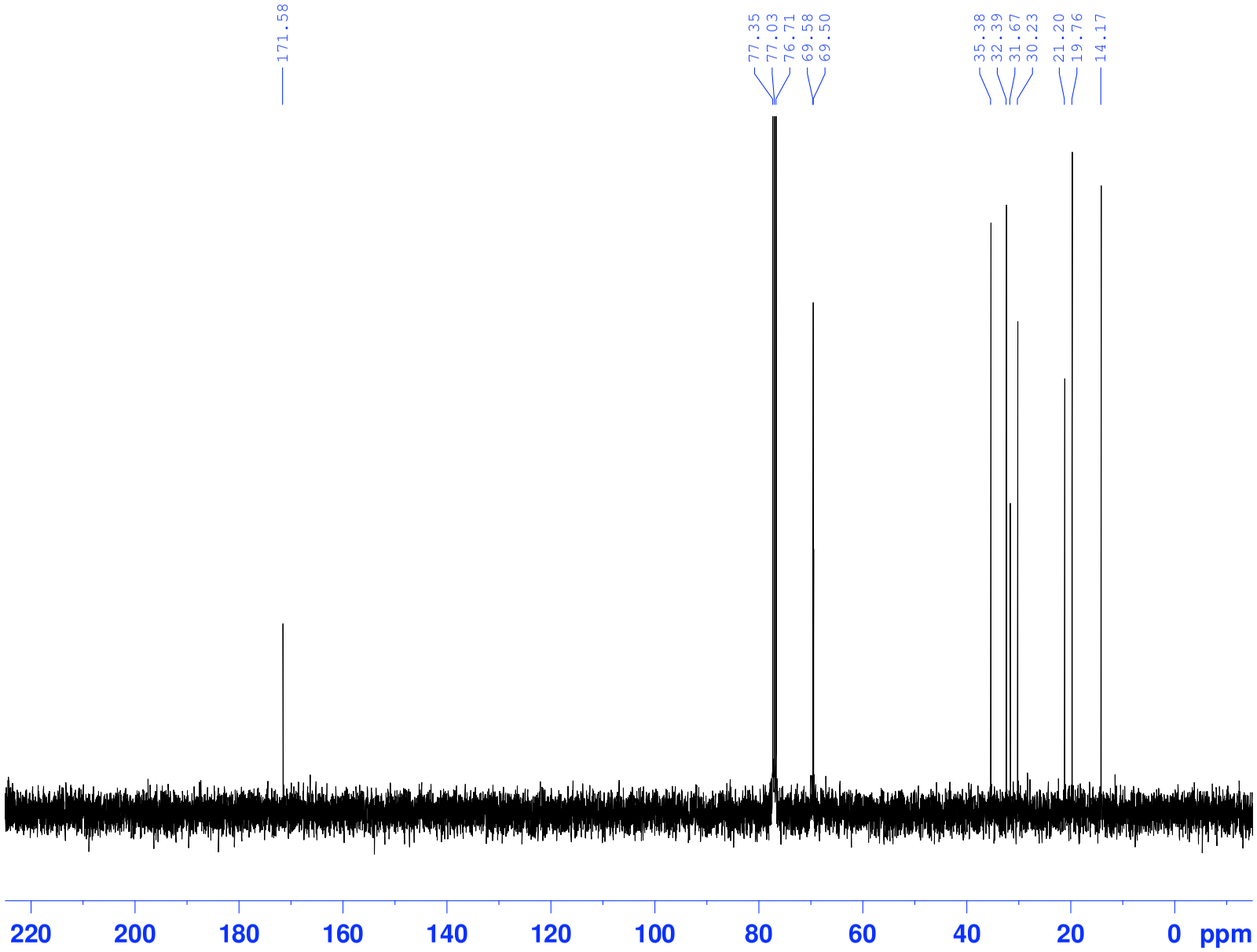




NMR@CHEM.OX

NAME	Feb11-2009-47	
EXPNO	1	
PROCNO	1	
Date_	20090211	
Time	16.39	
INSTRUM	av400	
PROBHD	5 mm QNP 1H/13	
PULPROG	zg60	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	90.5	
DW	60.400	usec
DE	7.50	usec
TE	294.7	K
D1	1.00000000	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.00	usec
PL1	0.00	dB
SFO1	400.2024714	MHz
SI	32768	
SF	400.2000028	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

Instrument DQX400
Chemist GCF
Group JR
3–96
c13acq.au CDCl3 {C:\NMR} jrgrp 47

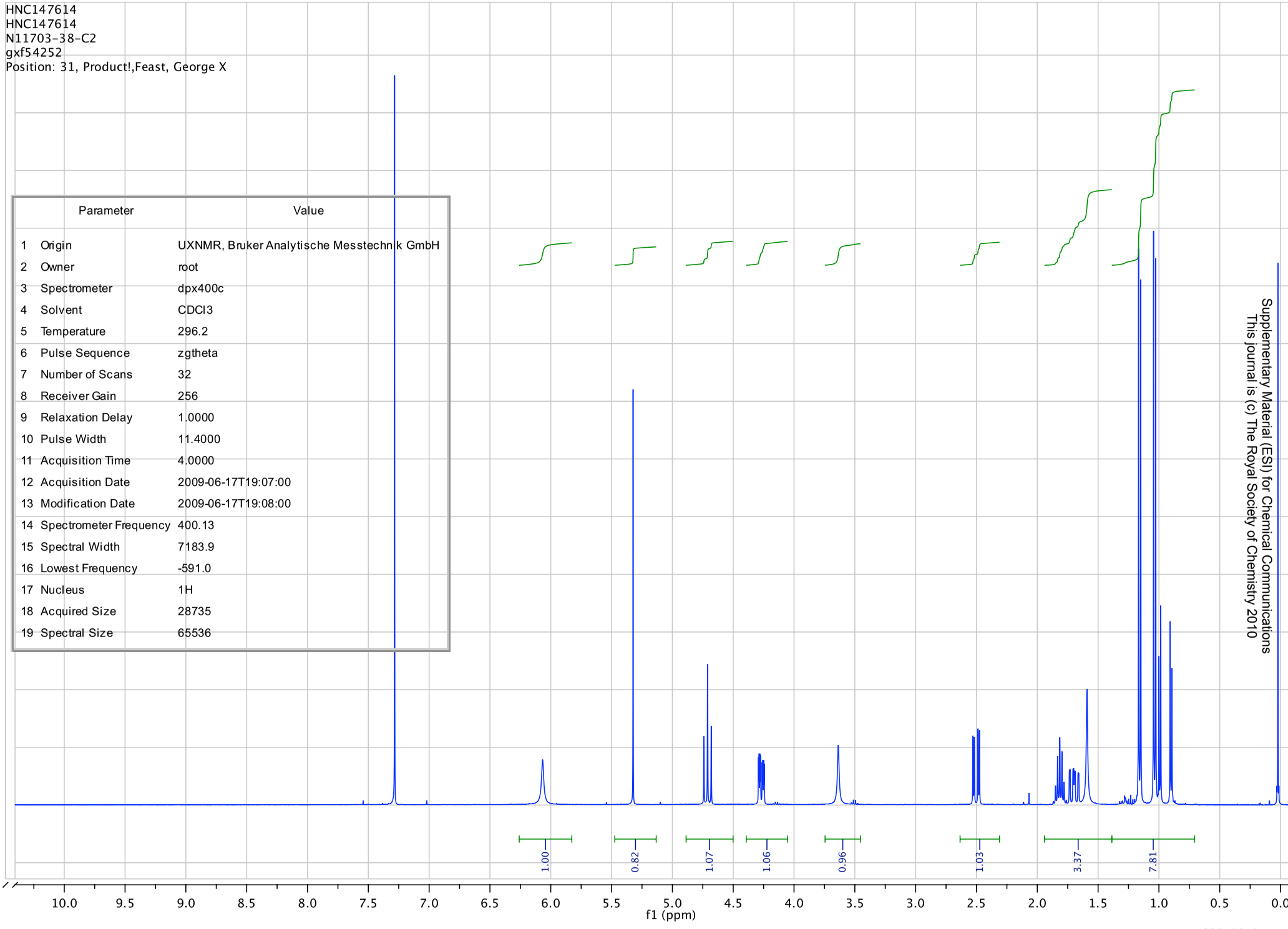


NMR@CHEM.OX

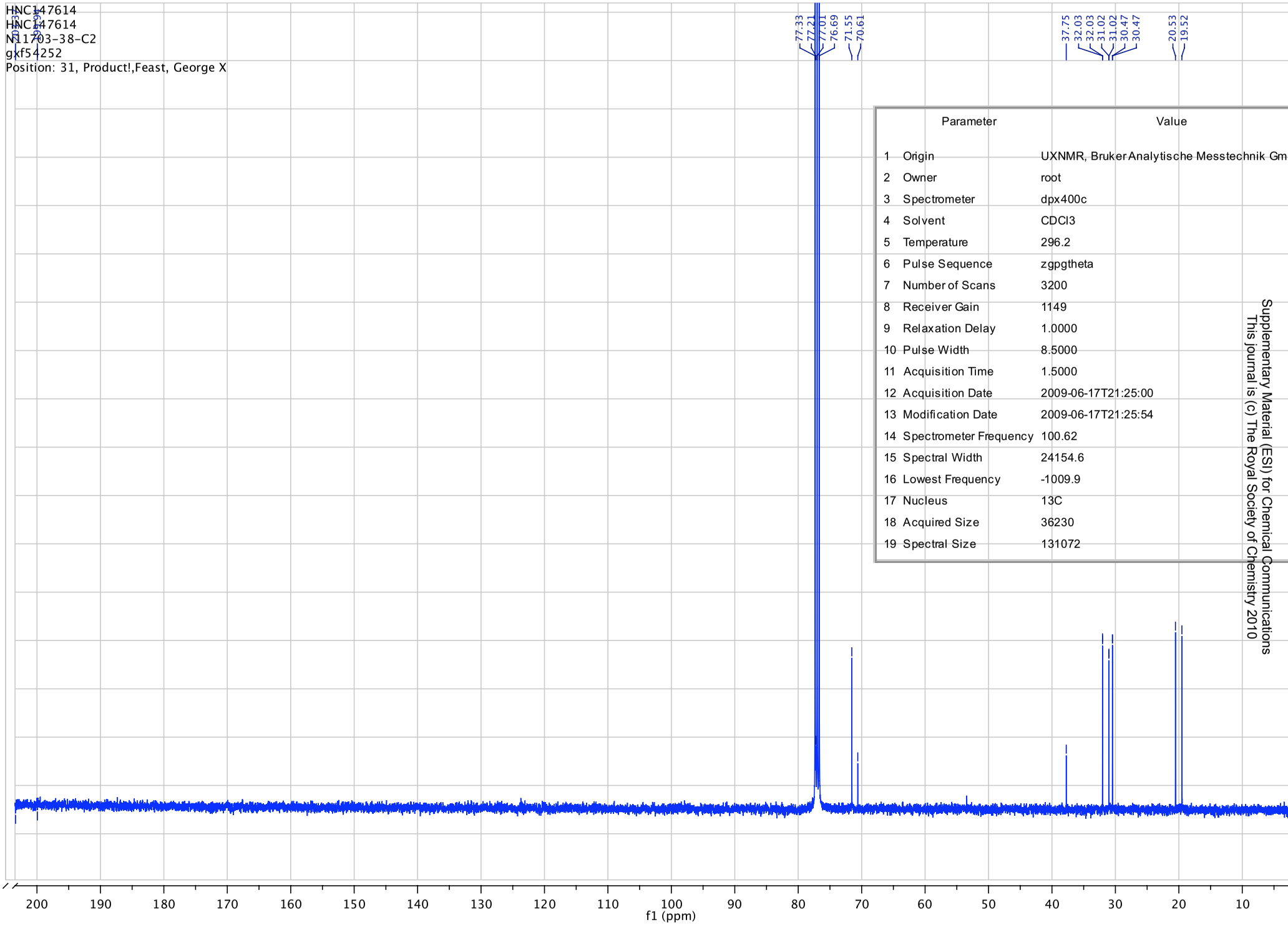
NAME	Feb11-2009-47
EXPNO	2
PROCNO	1
Date_	20090211
Time	16.47
INSTRUM	av400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	256
DS	4
SWH	26178.010 Hz
FIDRES	0.798889 Hz
AQ	0.6259188 sec
RG	32768
DW	19.100 usec
DE	7.50 usec
TE	295.1 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	0.00 dB
SFO1	100.6403931 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	25.00 dB
SFO2	400.2016008 MHz
SI	32768
SF	100.6303718 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

HNC147614
HNC147614
N11703-38-C2
gxf54252
Position: 31, Product!,Feast, George X

	Parameter	Value
1	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
2	Owner	root
3	Spectrometer	dpx400c
4	Solvent	CDCl3
5	Temperature	296.2
6	Pulse Sequence	zgtheta
7	Number of Scans	32
8	Receiver Gain	256
9	Relaxation Delay	1.0000
10	Pulse Width	11.4000
11	Acquisition Time	4.0000
12	Acquisition Date	2009-06-17T19:07:00
13	Modification Date	2009-06-17T19:08:00
14	Spectrometer Frequency	400.13
15	Spectral Width	7183.9
16	Lowest Frequency	-591.0
17	Nucleus	1H
18	Acquired Size	28735
19	Spectral Size	65536



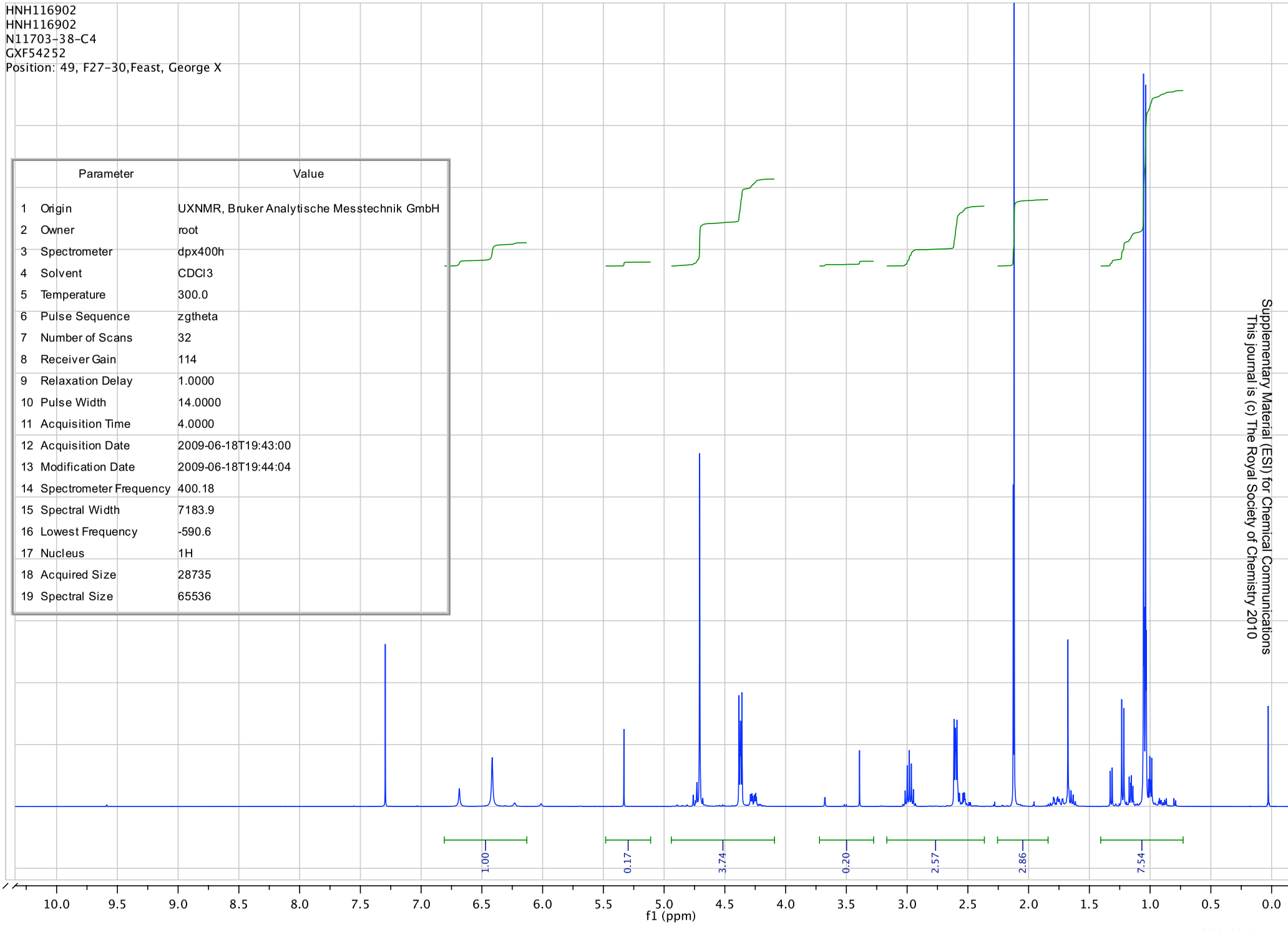
H1-(14)



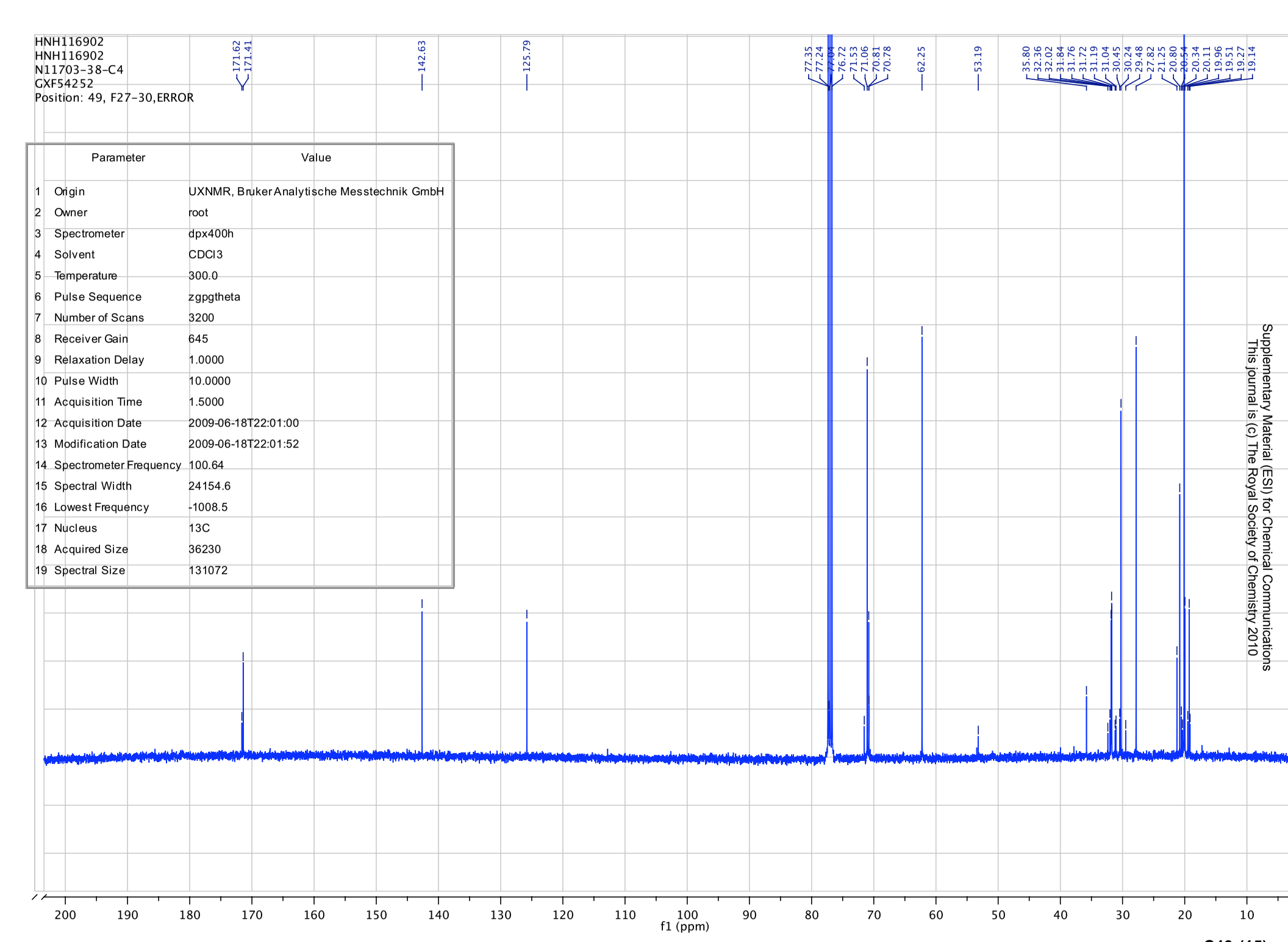
C13-(14)

HNH116902
HNH116902
N11703-38-C4
GXF54252
Position: 49, F27-30,Feast, George X

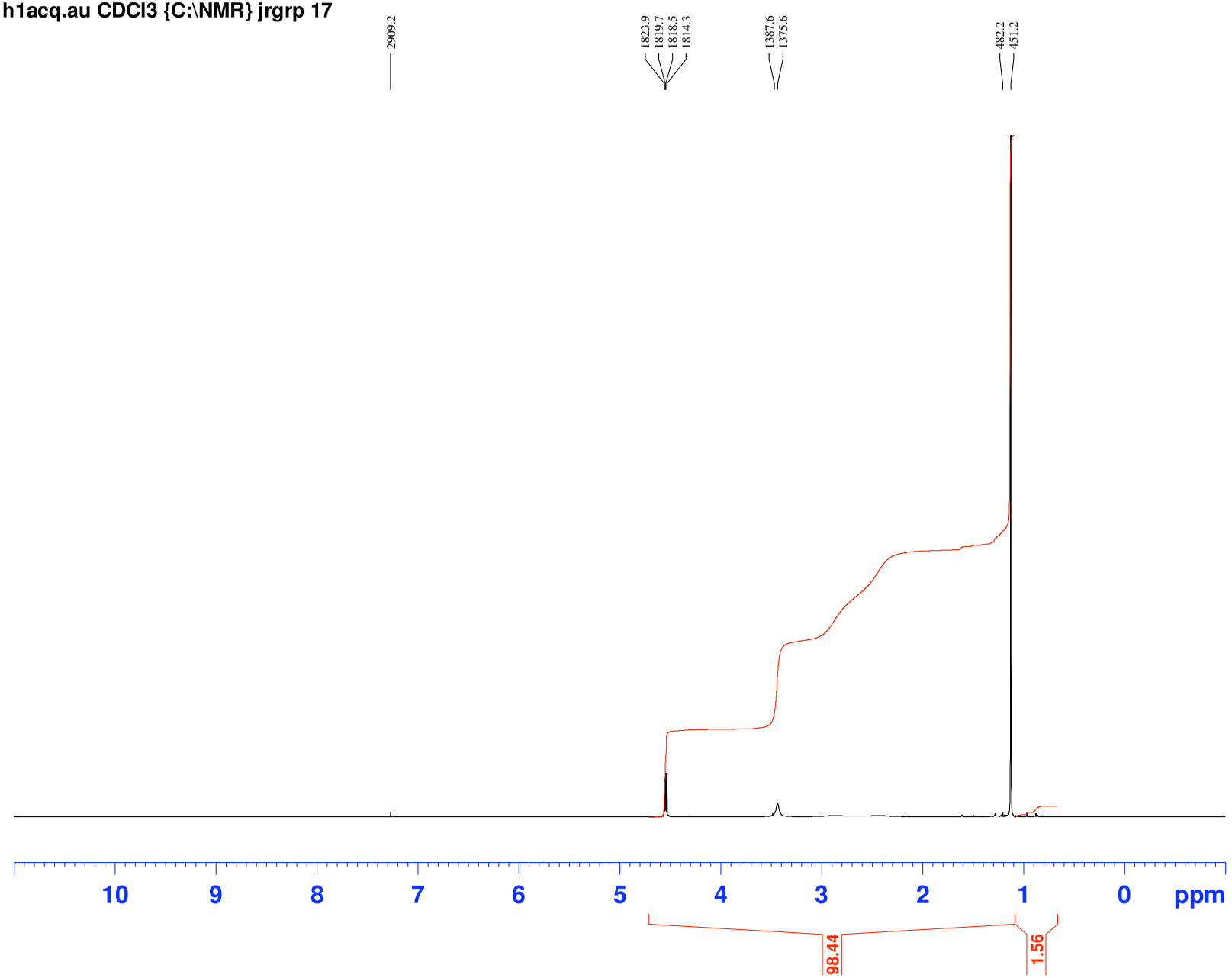
	Parameter	Value
1	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
2	Owner	root
3	Spectrometer	dpx400h
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgtheta
7	Number of Scans	32
8	Receiver Gain	114
9	Relaxation Delay	1.0000
10	Pulse Width	14.0000
11	Acquisition Time	4.0000
12	Acquisition Date	2009-06-18T19:43:00
13	Modification Date	2009-06-18T19:44:04
14	Spectrometer Frequency	400.18
15	Spectral Width	7183.9
16	Lowest Frequency	-590.6
17	Nucleus	1H
18	Acquired Size	28735
19	Spectral Size	65536



H1-(15)



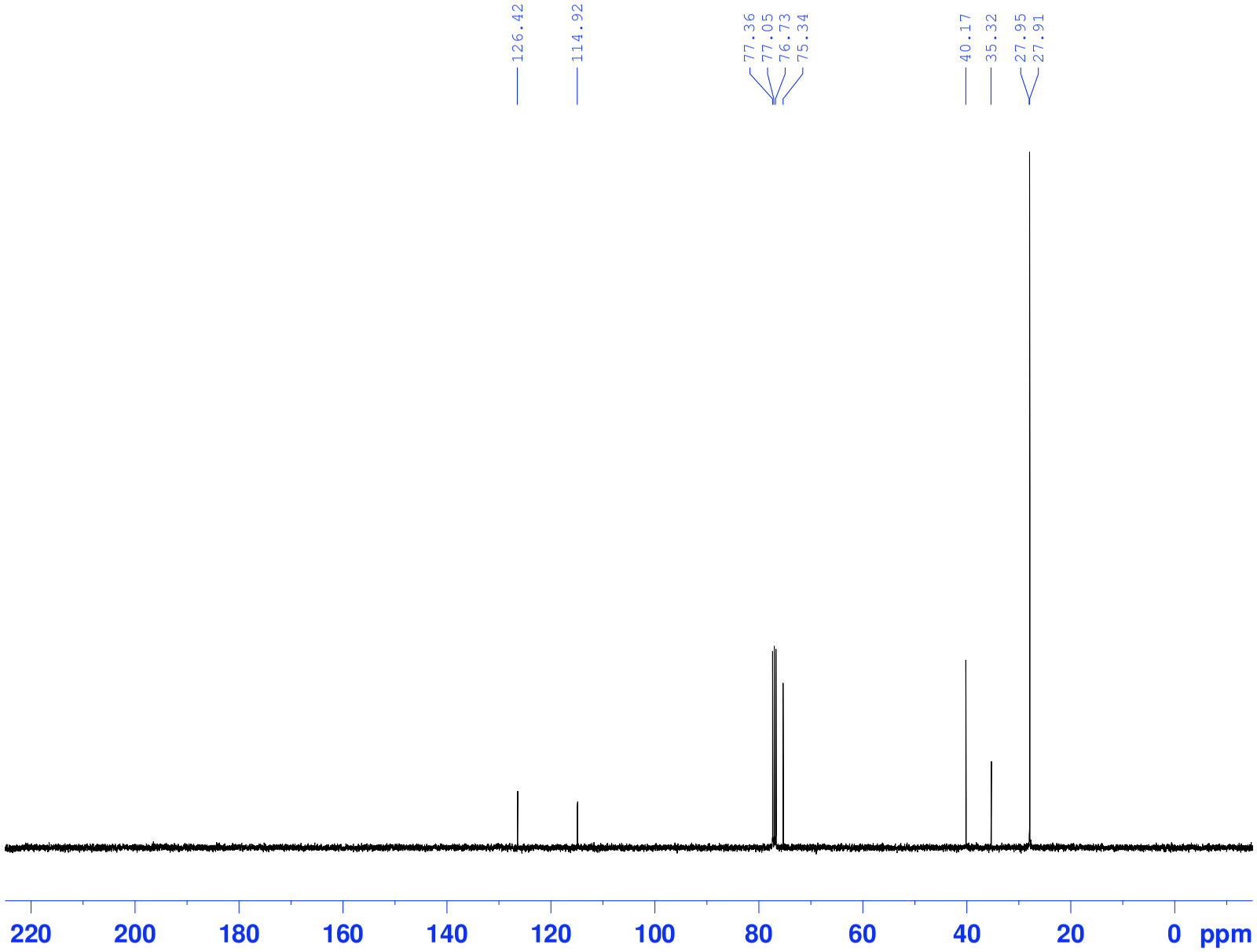
Instrument DQX400
Chemist GCF
Group JR
3–78 F12
h1acq.au CDCl3 {C:\NMR} jrgrp 17



NMR@CHEM.OX

NAME	Jan09-2009-17	
EXPNO	6	
PROCNO	1	
Date_	20090110	
Time	6.42	
INSTRUM	av400	
PROBHD	5 mm QNP 1H/13	
PULPROG	zg60	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8278.146	Hz
FIDRES	0.126314	Hz
AQ	3.9584243	sec
RG	90.5	
DW	60.400	usec
DE	7.50	usec
TE	294.4	K
D1	1.00000000	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.00	usec
PL1	0.00	dB
SFO1	400.2024714	MHz
SI	32768	
SF	400.2000028	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

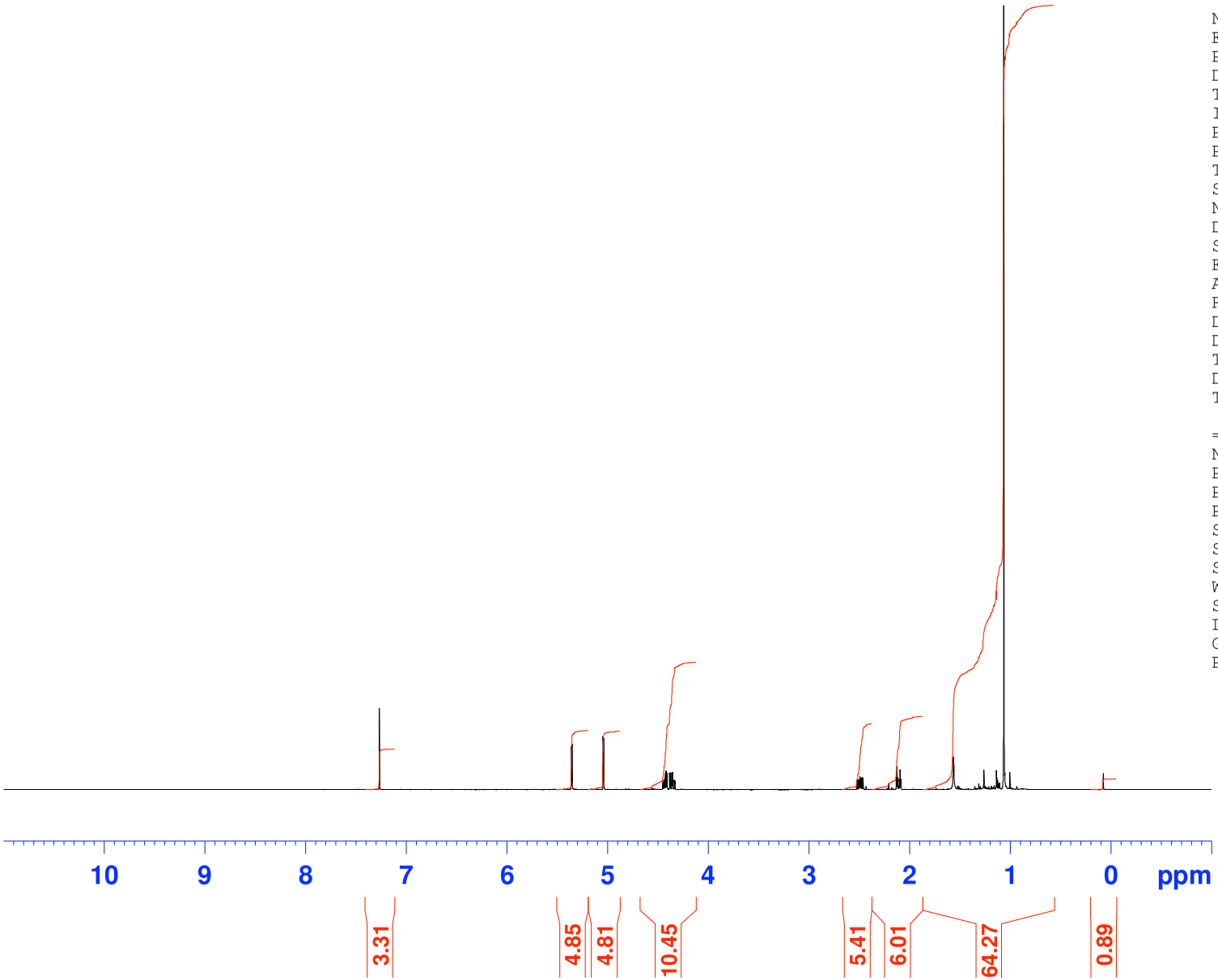
Instrument DQX400
Chemist GCF
Group JR
3–78 F12
c13acq.au CDCl3 {C:\NMR} jrgrp 17



NMR@CHEM.OX

NAME	Jan09-2009-17
EXPNO	7
PROCNO	1
Date_	20090110
Time	6.50
INSTRUM	av400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	256
DS	4
SWH	26178.010 Hz
FIDRES	0.798889 Hz
AQ	0.6259188 sec
RG	32768
DW	19.100 usec
DE	7.50 usec
TE	294.7 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	0.00 dB
SFO1	100.6403931 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	25.00 dB
SFO2	400.2016008 MHz
SI	32768
SF	100.6303718 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

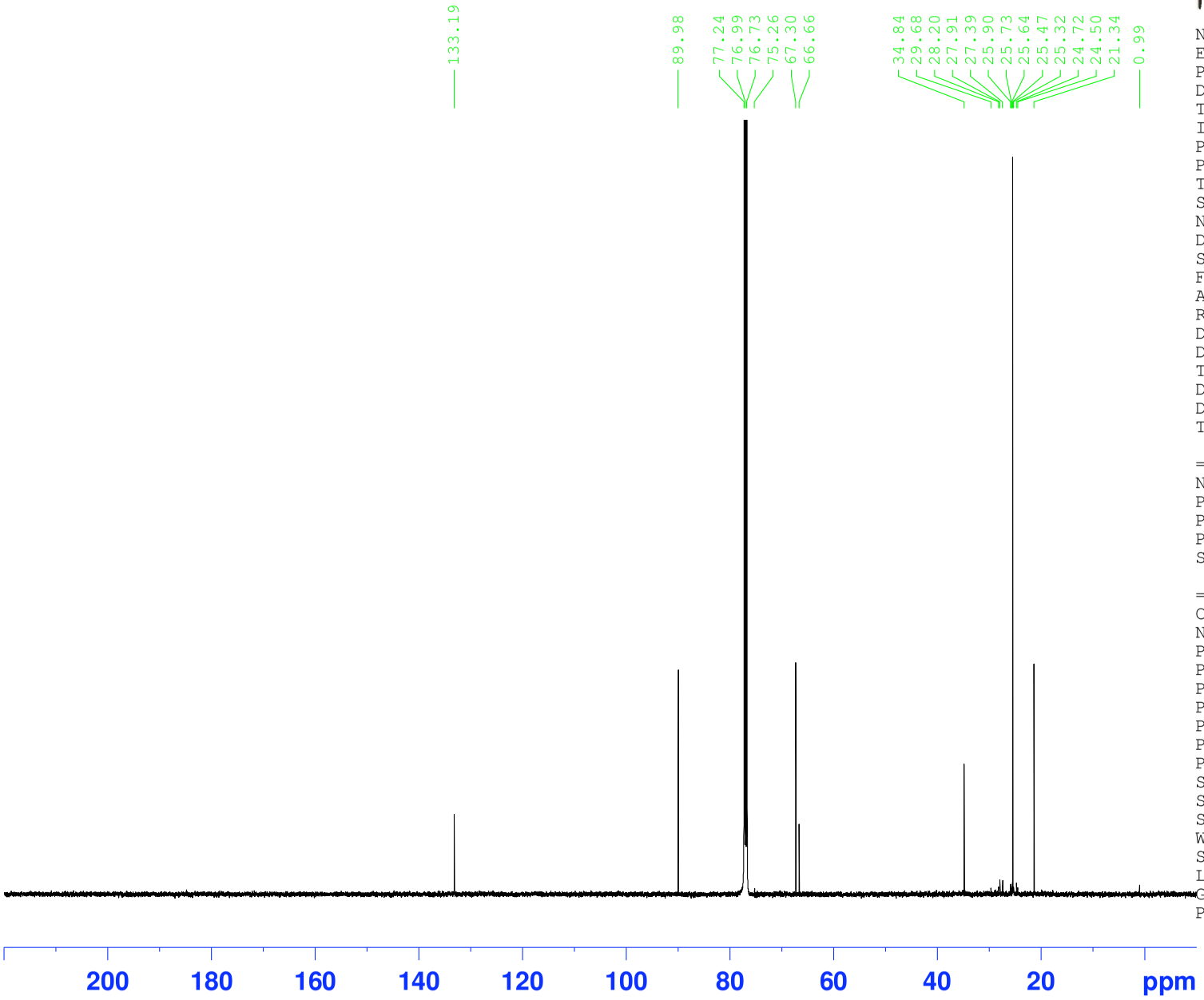
Instrument AVC500
2606 George Feast 12/1/09



NMR@CHEM.OX

NAME	gf26061201
EXPNO	1
PROCNO	1
Date_	20090113
Time	15.23
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

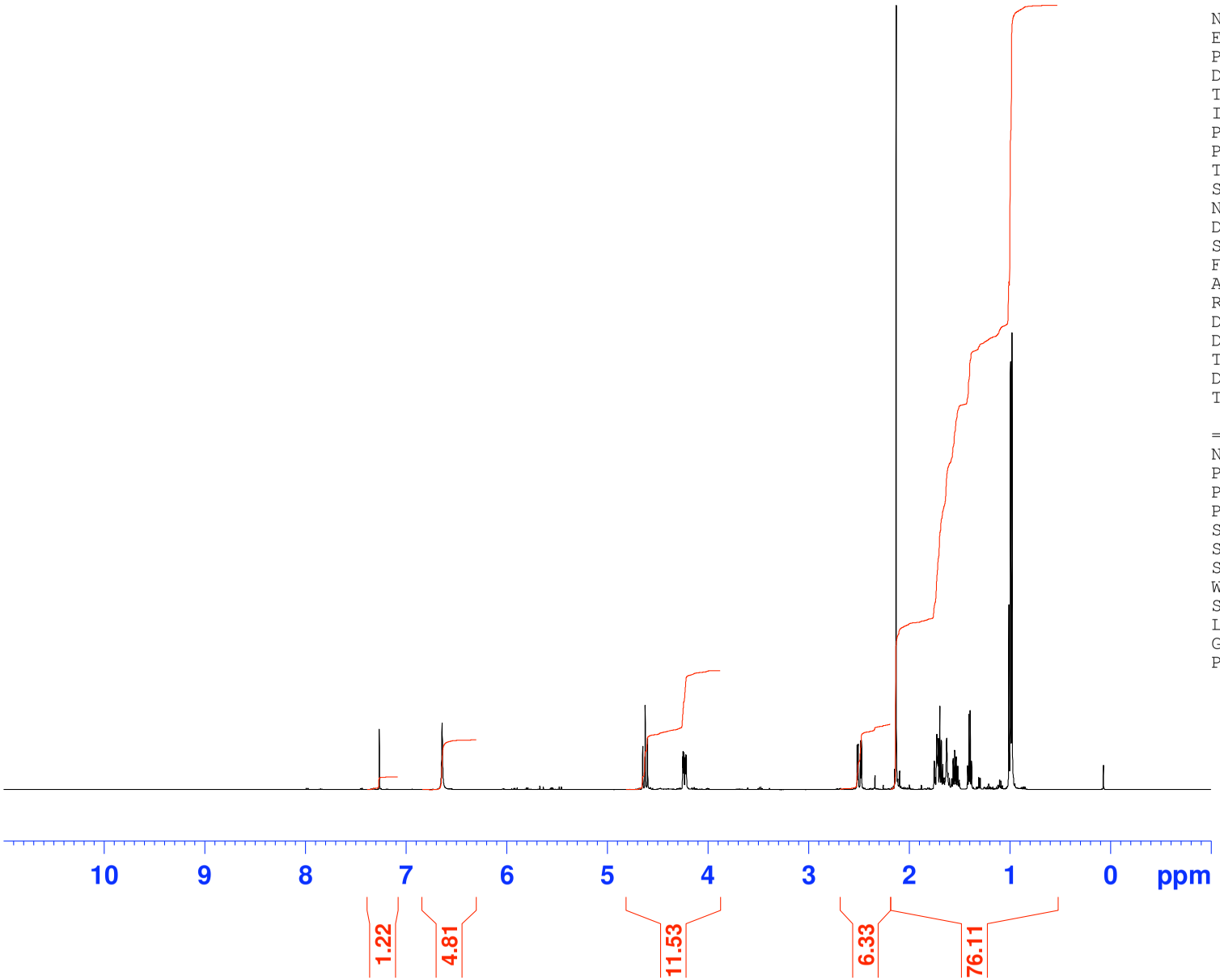
Instrument AVC500
2606 George Feast 12/1/09



NMR@CHEM.OX

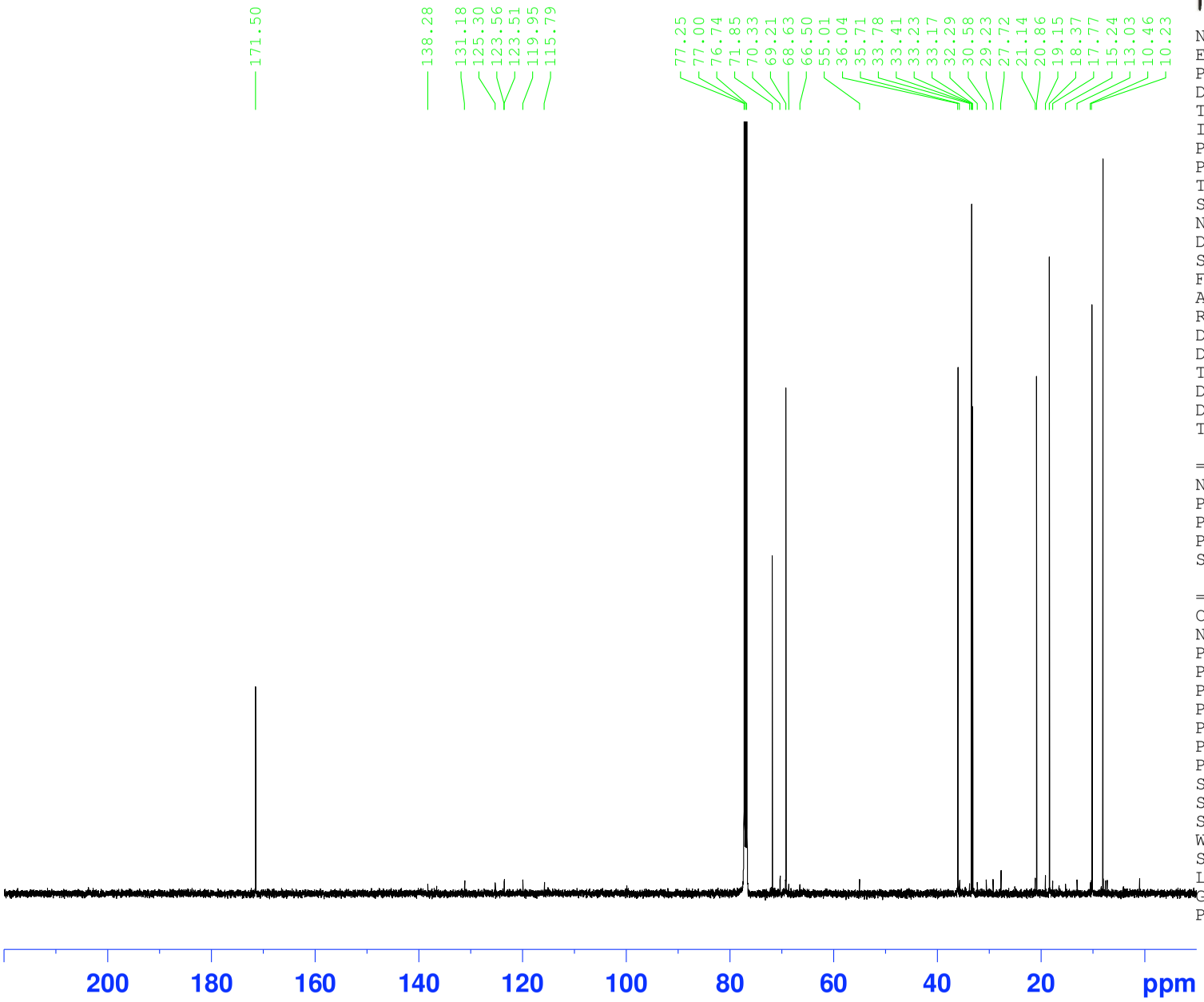
NAME	gf26061201
EXPNO	4
PROCNO	1
Date_	20090113
Time	16.30
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Instrument AVC500
6328 George Feast 21/10/09



NAME	gf63282110
EXPNO	1
PROCNO	1
Date_	20091022
Time	3.09
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

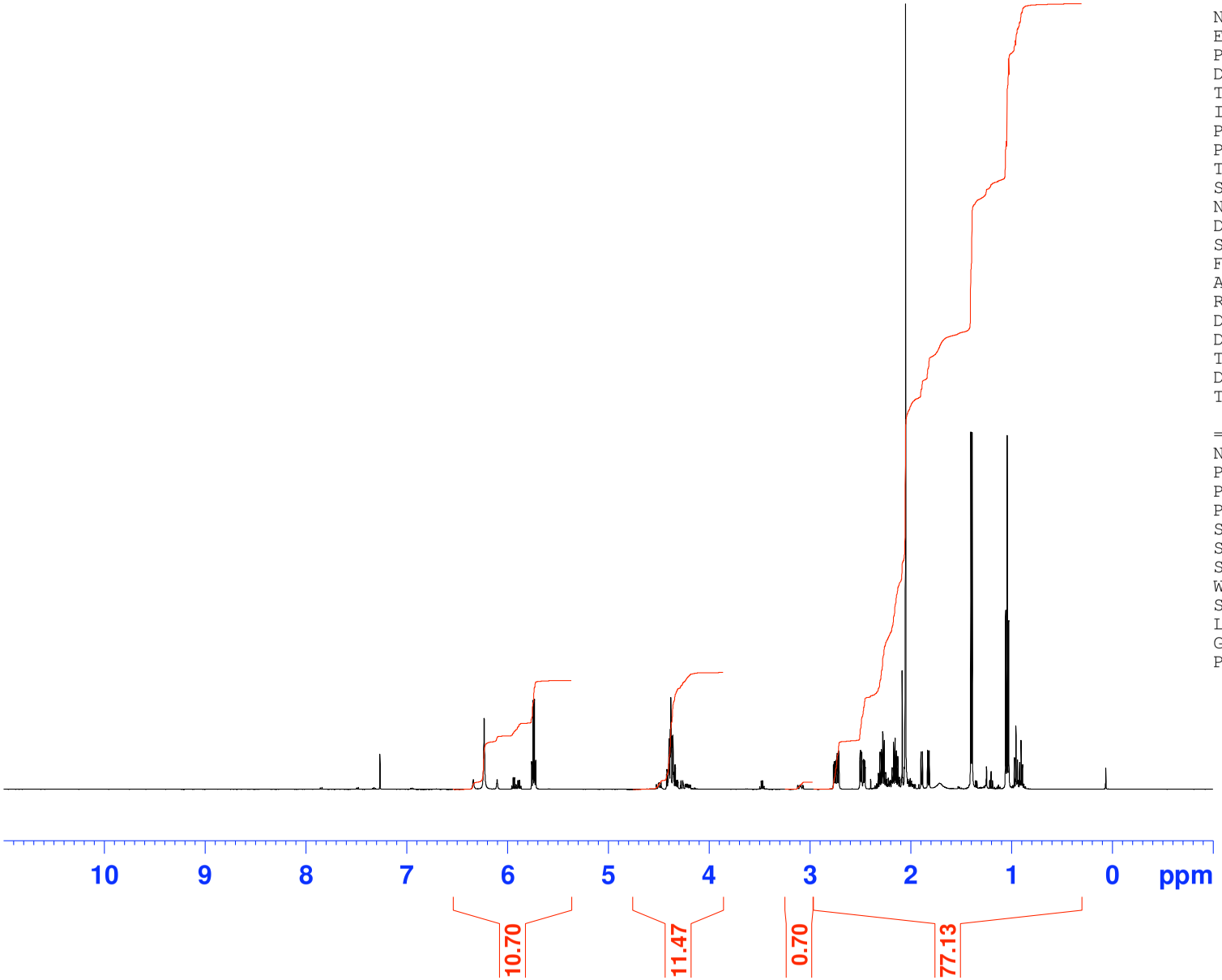
Instrument AVC500
6328 George Feast 21/10/09



NMR@CHEM.OX

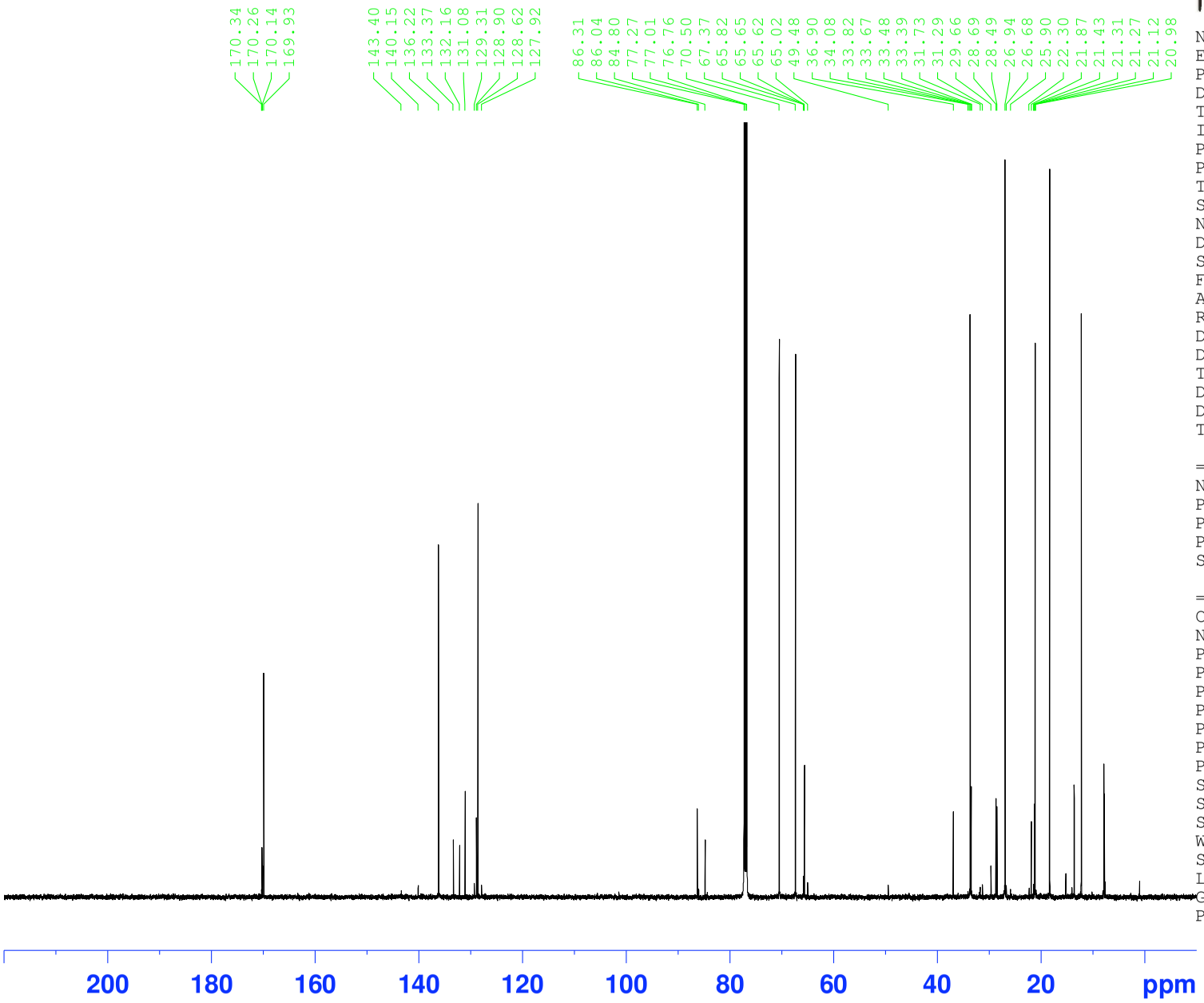
NAME	gf63282110
EXPNO	4
PROCNO	1
Date_	20091022
Time	3.39
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Instrument AVC500
6329 George Feast 21/10/09



NAME	gf63292110
EXPNO	1
PROCNO	1
Date_	20091022
Time	3.43
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

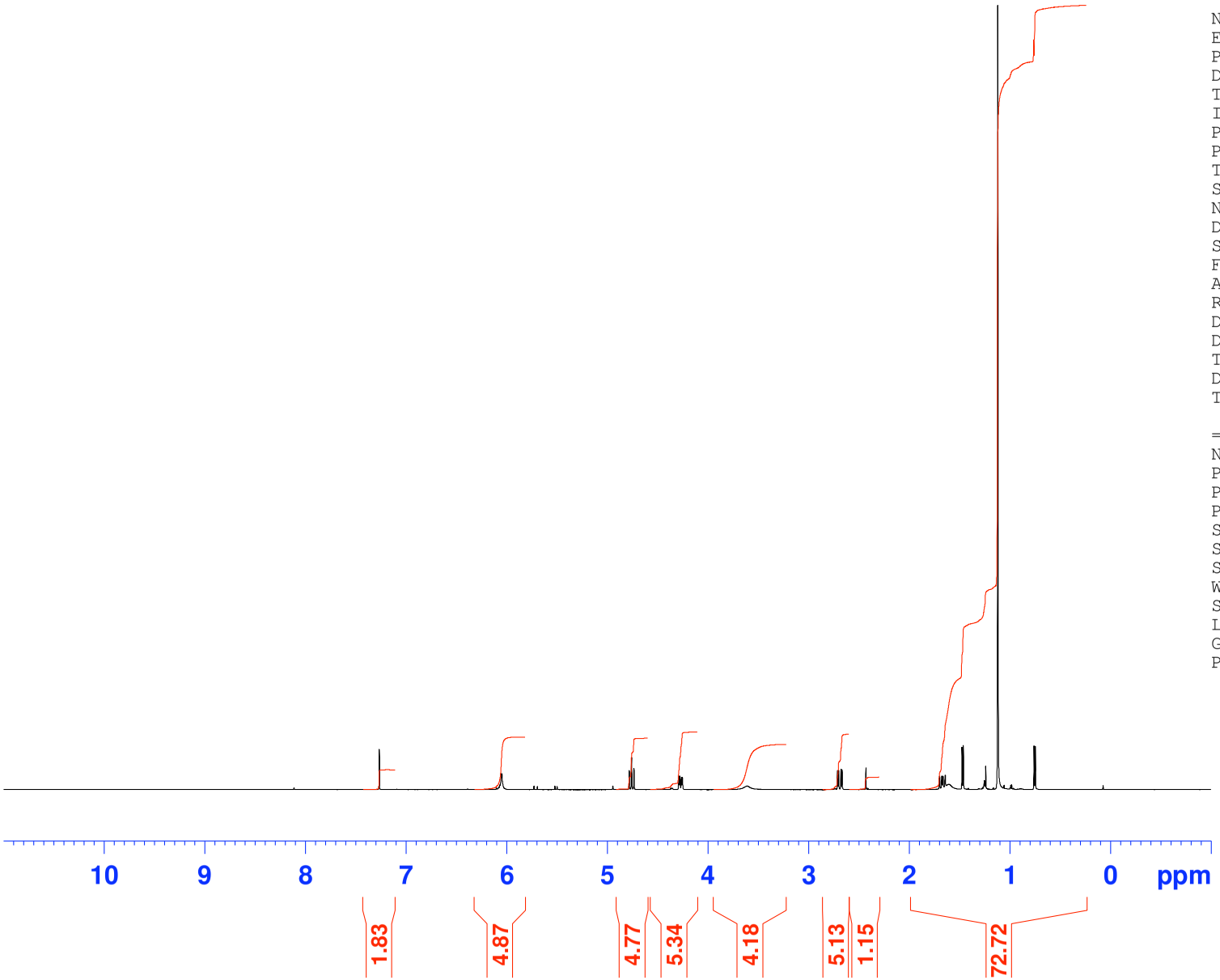
Instrument AVC500
6329 George Feast 21/10/09



NMR@CHEM.OX

NAME	gf63292110
EXPNO	4
PROCNO	1
Date_	20091022
Time	4.13
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

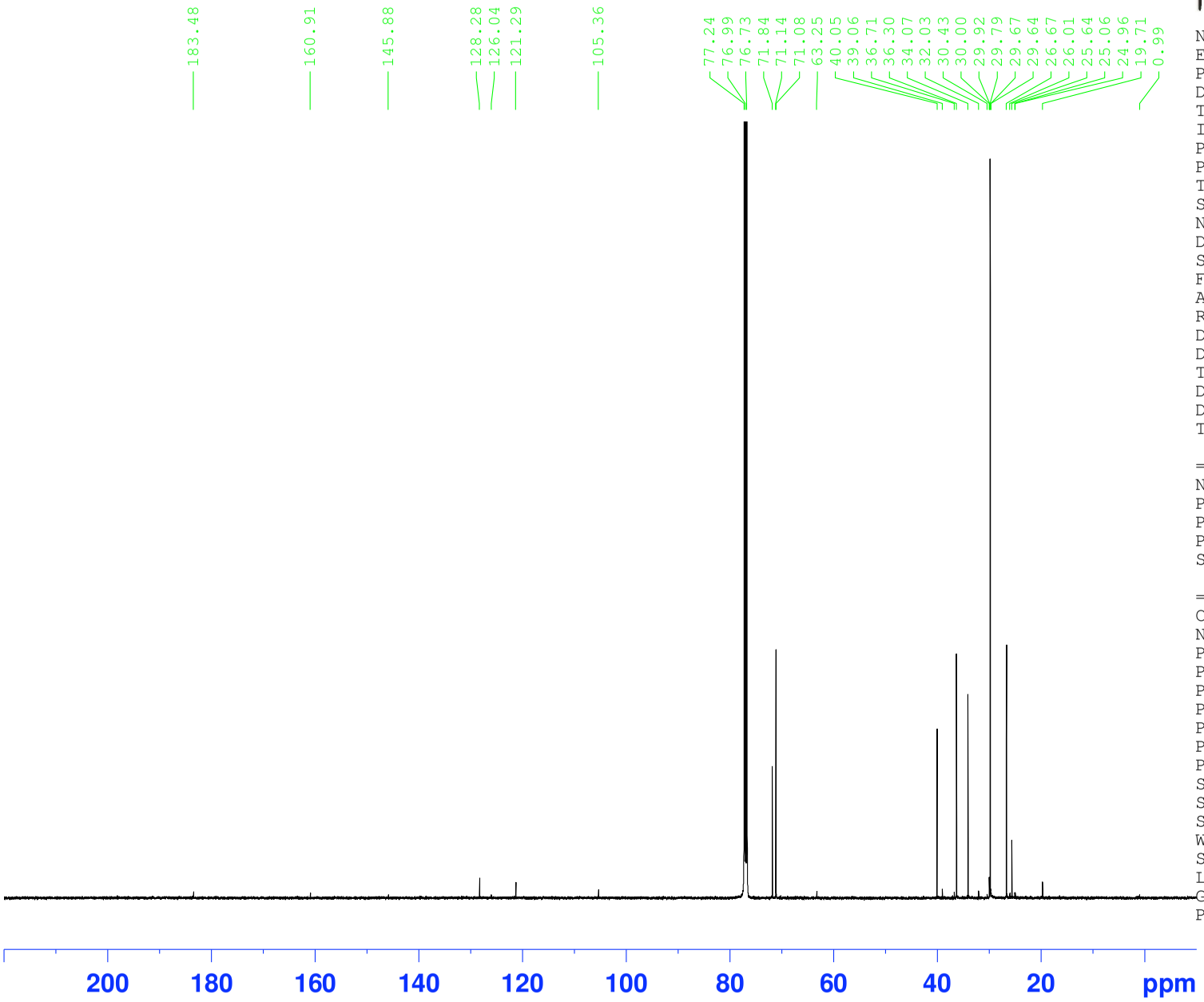
Instrument AVC500
5629 George Feast 20/8/09



NMR@CHEM.OX

NAME	gf56292008
EXPNO	1
PROCNO	1
Date_	20090820
Time	10.33
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

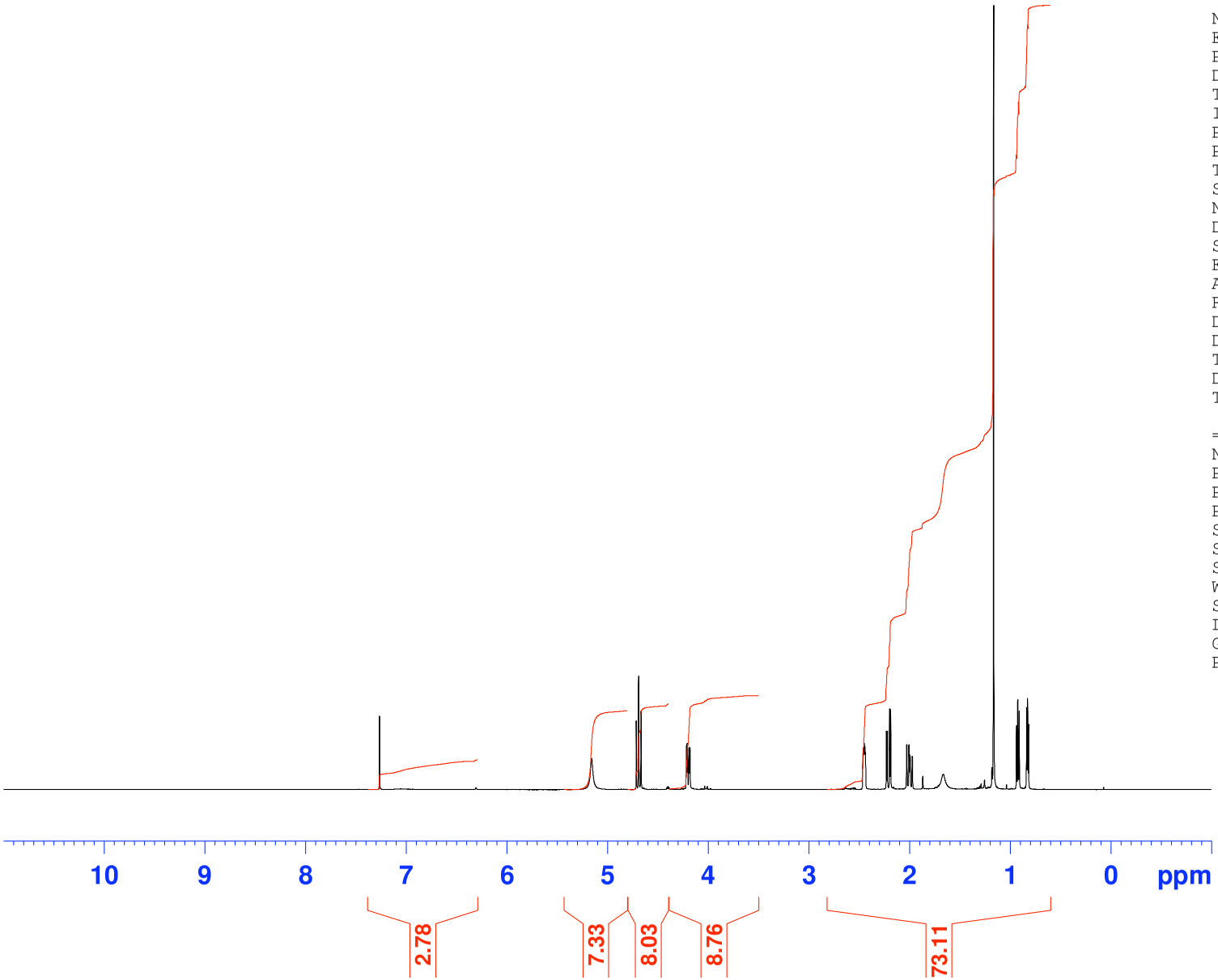
Instrument AVC500
5629 George Feast 20/8/09



NMR@CHEM.OX

NAME	gf56292008
EXPNO	4
PROCNO	1
Date_	20090820
Time	13.22
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1678
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	912
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

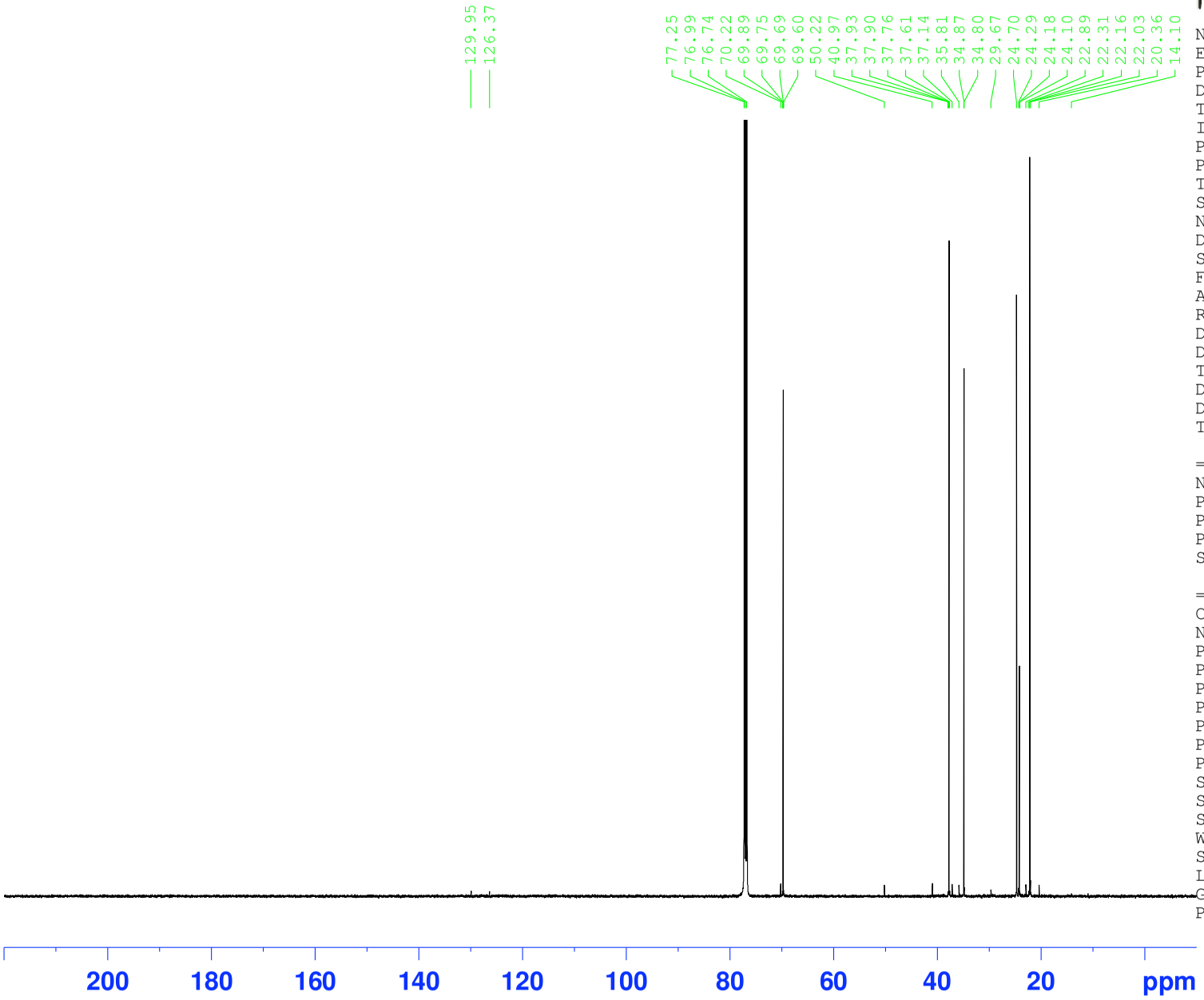
Instrument AVC500
GEORGE FEAST 5477 6/8/09



NMR@CHEM.OX

NAME	gf54770608	
EXPNO	1	
PROCNO	1	
Date_	20090806	
Time	22.33	
INSTRUM	avc500	
PROBHD	5 mm CPDUL 13C	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	4	
DW	48.400	usec
DE	6.00	usec
TE	298.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.60	usec
PL1	-6.00	dB
PL1W	15.19999981	W
SFO1	500.3030896	MHz
SI	32768	
SF	500.3000240	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

Instrument AVC500
GEORGE FEAST 5477 6/8/09

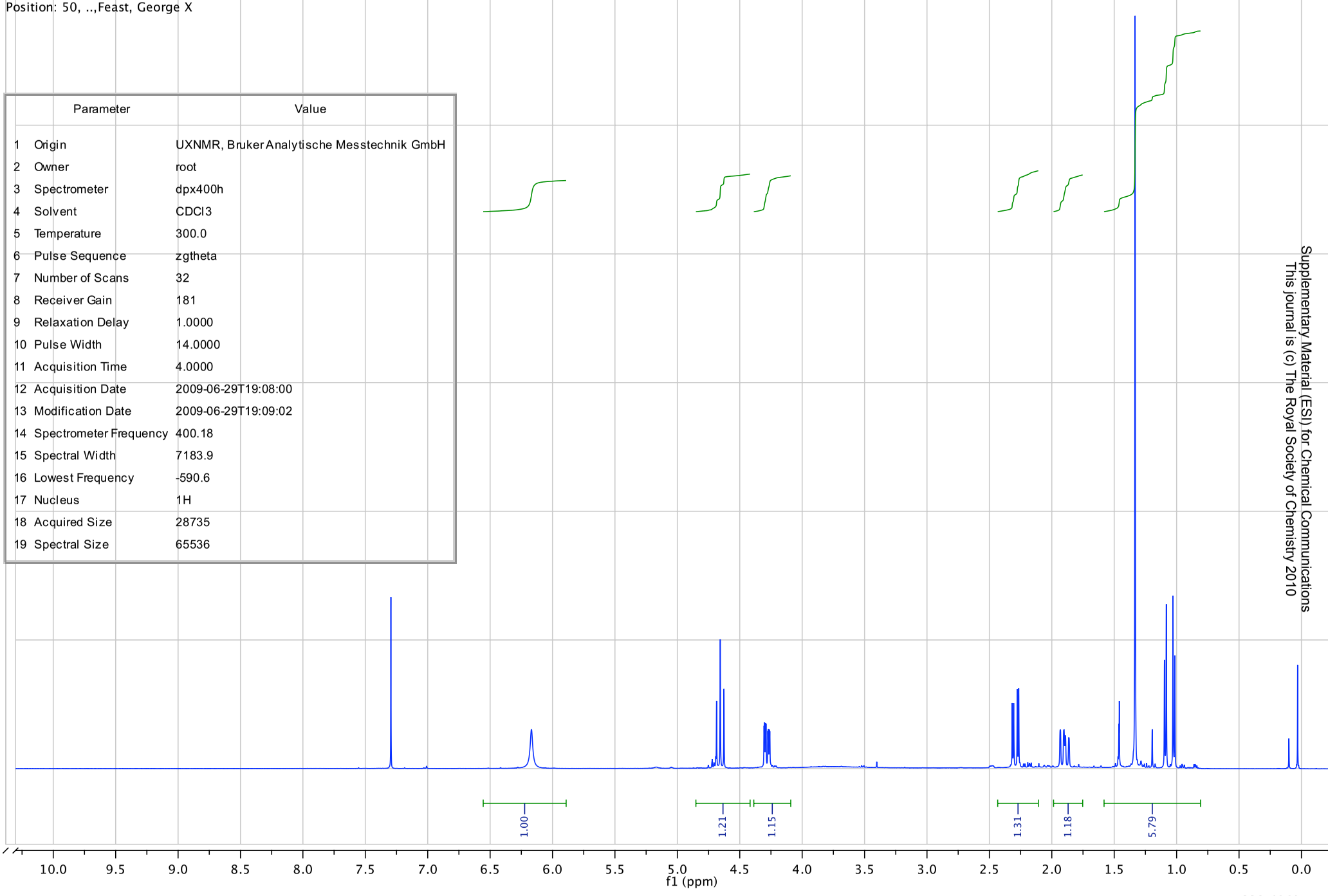


NMR@CHEM.OX

NAME	gf54770608
EXPNO	4
PROCNO	1
Date_	20090807
Time	0.36
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

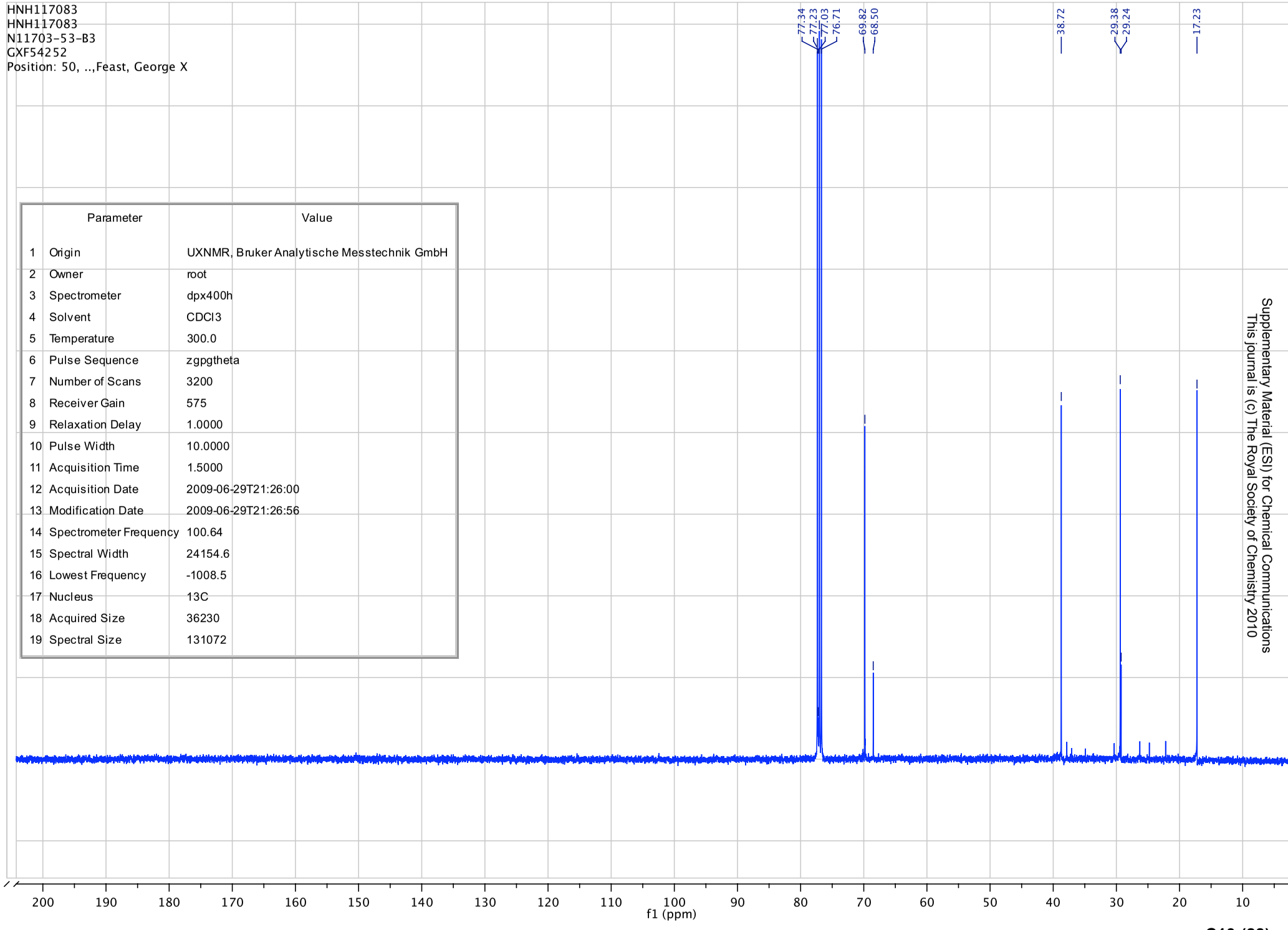
HNH117083
HNH117083
N11703-53-B3
GXF54252
Position: 50, ..,Feast, George X

	Parameter	Value
1	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
2	Owner	root
3	Spectrometer	dpx400h
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgtheta
7	Number of Scans	32
8	Receiver Gain	181
9	Relaxation Delay	1.0000
10	Pulse Width	14.0000
11	Acquisition Time	4.0000
12	Acquisition Date	2009-06-29T19:08:00
13	Modification Date	2009-06-29T19:09:02
14	Spectrometer Frequency	400.18
15	Spectral Width	7183.9
16	Lowest Frequency	-590.6
17	Nucleus	1H
18	Acquired Size	28735
19	Spectral Size	65536



H1-(28)

HNH117083
HNH117083
N11703-53-B3
GXF54252
Position: 50, ..,Feast, George X

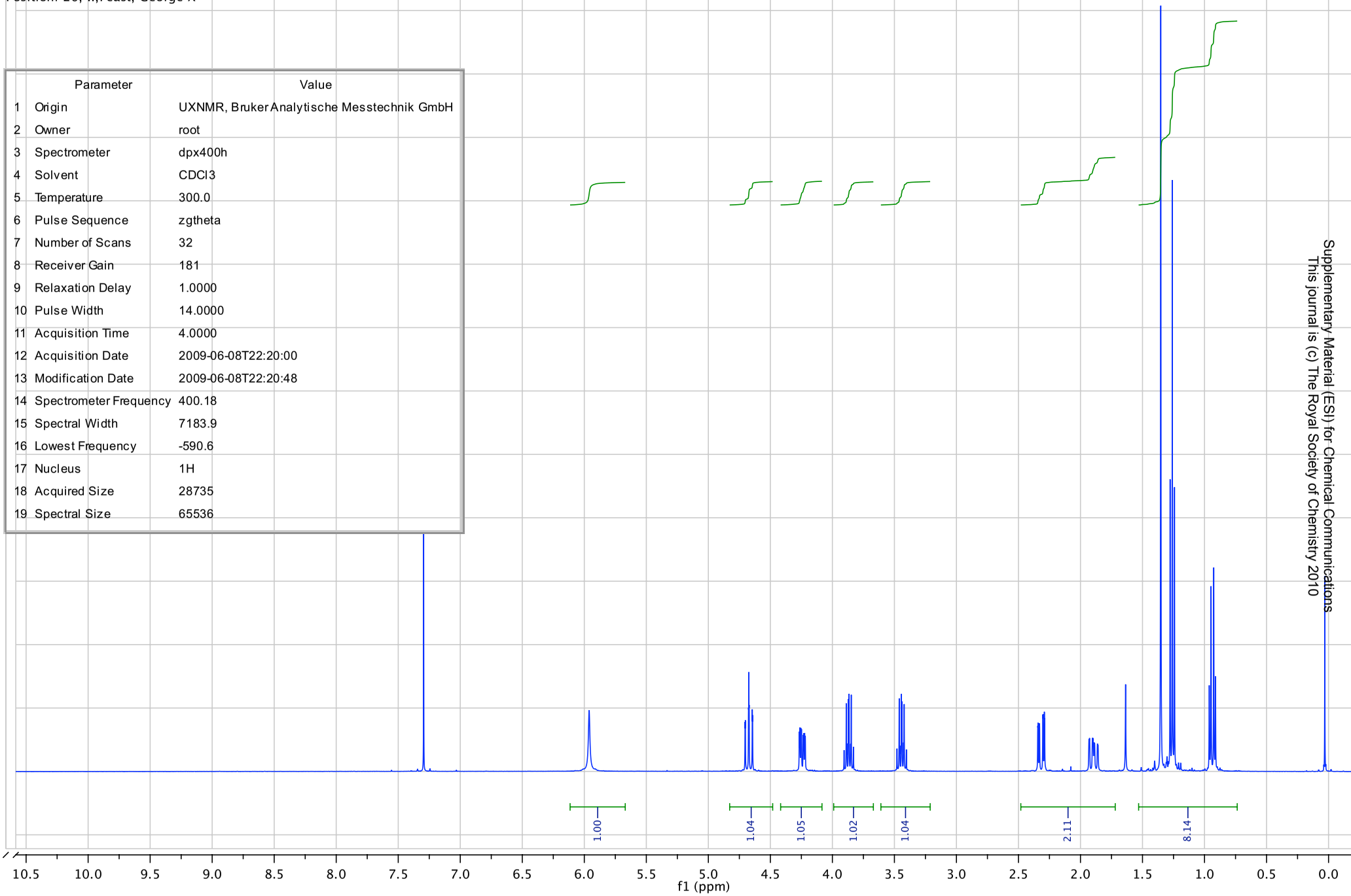


	Parameter	Value
1	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
2	Owner	root
3	Spectrometer	dpx400h
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgpgtheta
7	Number of Scans	3200
8	Receiver Gain	575
9	Relaxation Delay	1.0000
10	Pulse Width	10.0000
11	Acquisition Time	1.5000
12	Acquisition Date	2009-06-29T21:26:00
13	Modification Date	2009-06-29T21:26:56
14	Spectrometer Frequency	100.64
15	Spectral Width	24154.6
16	Lowest Frequency	-1008.5
17	Nucleus	13C
18	Acquired Size	36230
19	Spectral Size	131072

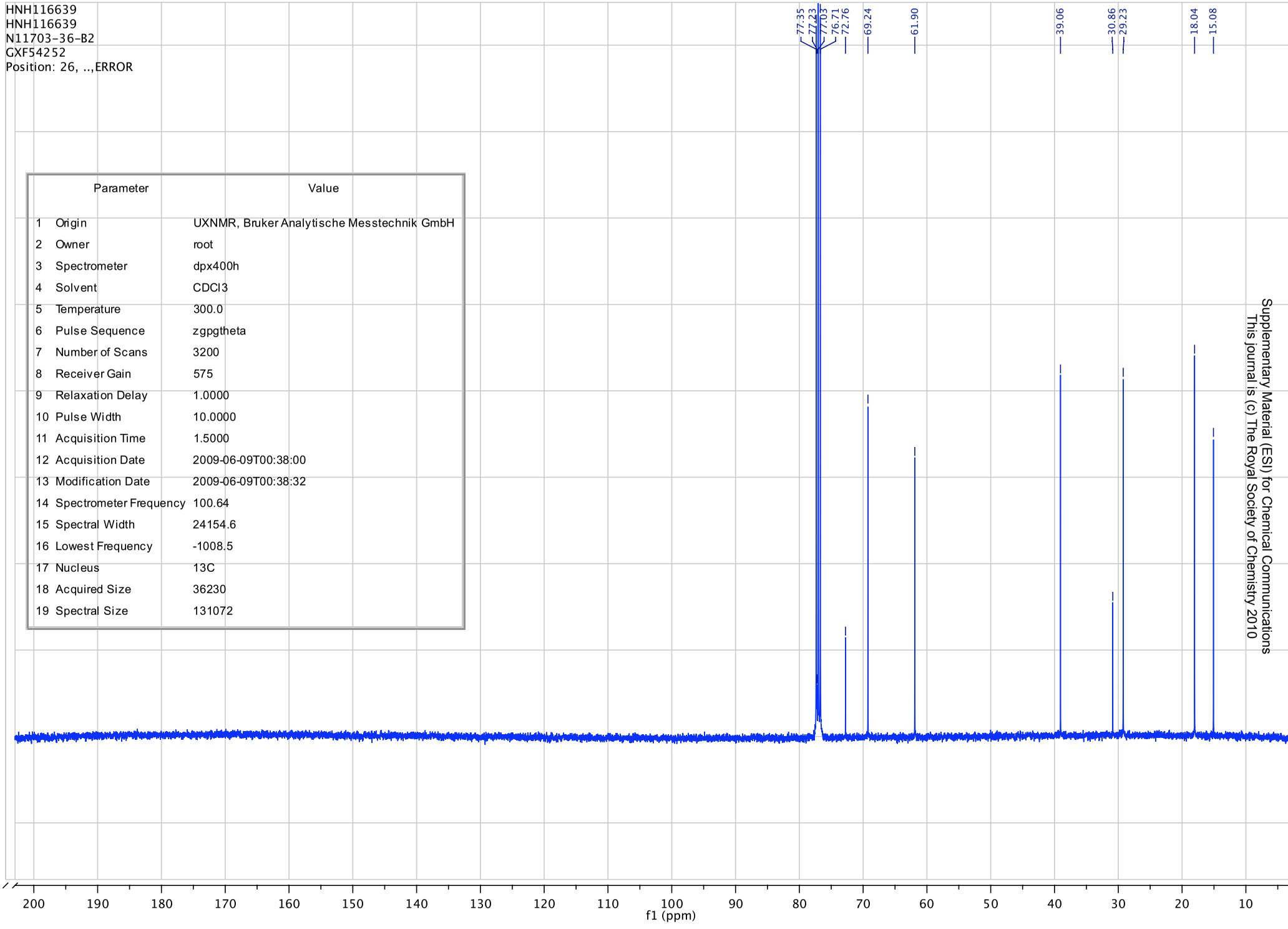
C13-(28)

HNH116639
HNH116639
N11703-36-B2
GXF54252
Position: 26, ..,Feast, George X

	Parameter	Value
1	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
2	Owner	root
3	Spectrometer	dpx400h
4	Solvent	CDCl3
5	Temperature	300.0
6	Pulse Sequence	zgtheta
7	Number of Scans	32
8	Receiver Gain	181
9	Relaxation Delay	1.0000
10	Pulse Width	14.0000
11	Acquisition Time	4.0000
12	Acquisition Date	2009-06-08T22:20:00
13	Modification Date	2009-06-08T22:20:48
14	Spectrometer Frequency	400.18
15	Spectral Width	7183.9
16	Lowest Frequency	-590.6
17	Nucleus	1H
18	Acquired Size	28735
19	Spectral Size	65536

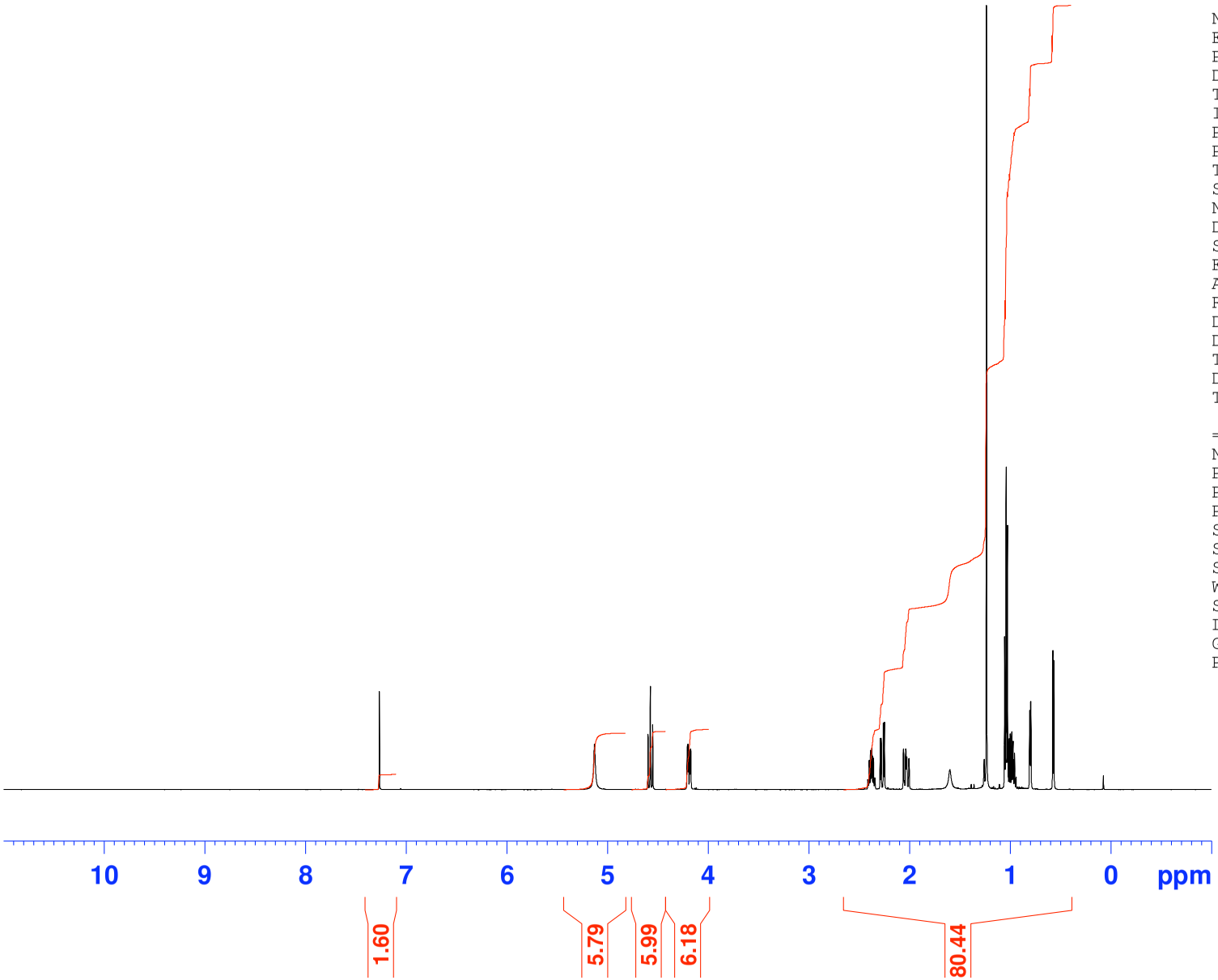


H1-(29)



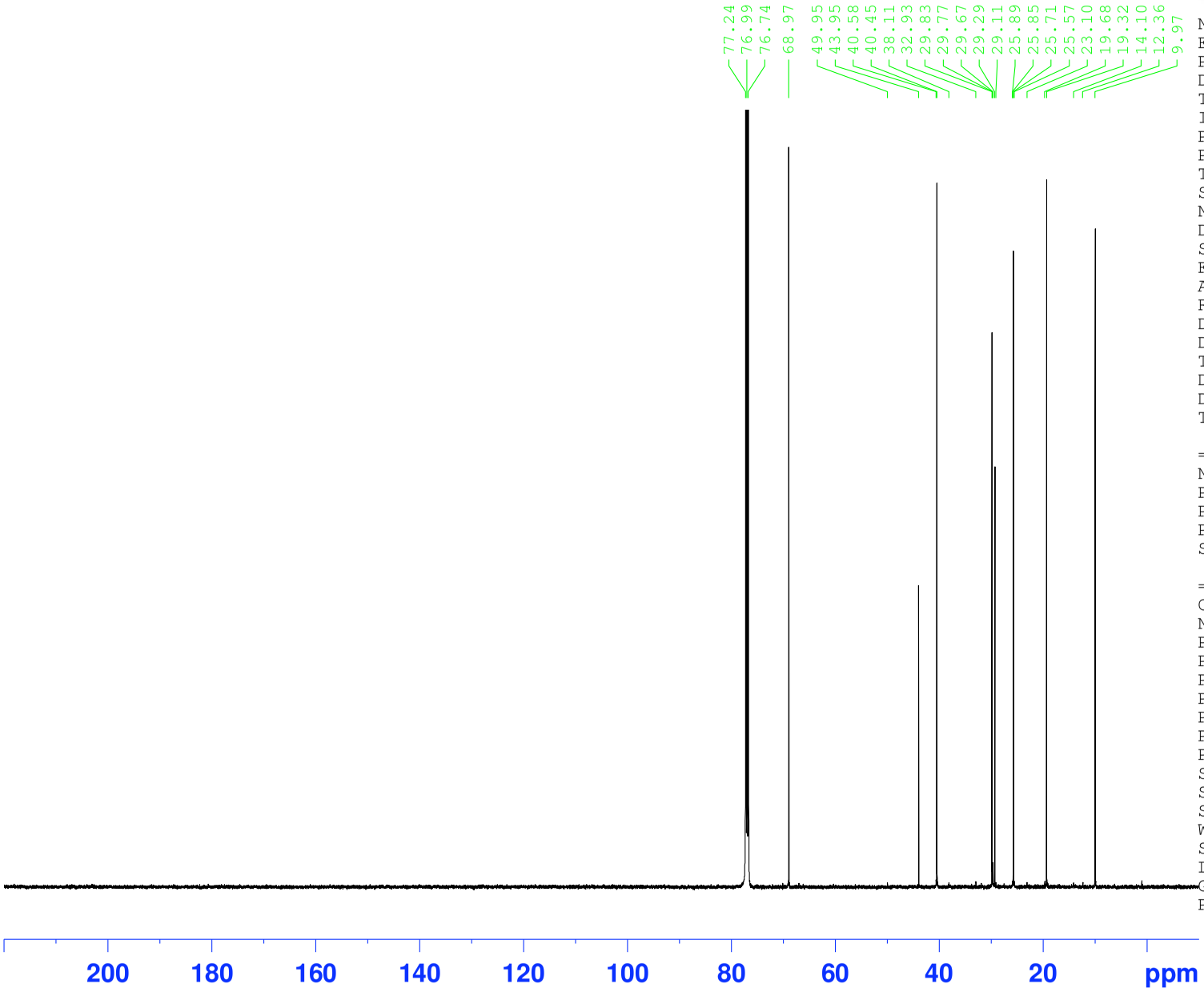
C13-(29)

Instrument AVC500
GEORGE FEAST 5574 13/8/09



NAME	gf555741308	
EXPNO	1	
PROCNO	1	
Date_	20090813	
Time	21.49	
INSTRUM	avc500	
PROBHD	5 mm CPDUL 13C	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	4	
DW	48.400	usec
DE	6.00	usec
TE	298.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	9.60	usec
PL1	-6.00	dB
PL1W	15.19999981	W
SFO1	500.3030896	MHz
SI	32768	
SF	500.3000240	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

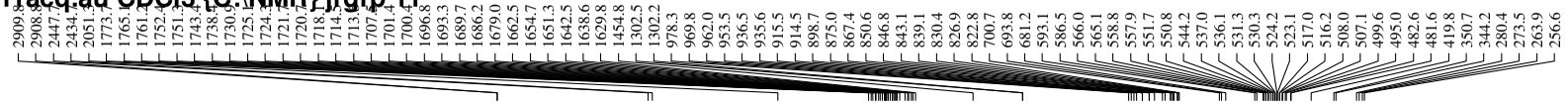
Instrument AVC500
GEORGE FEAST 5574 13/8/09

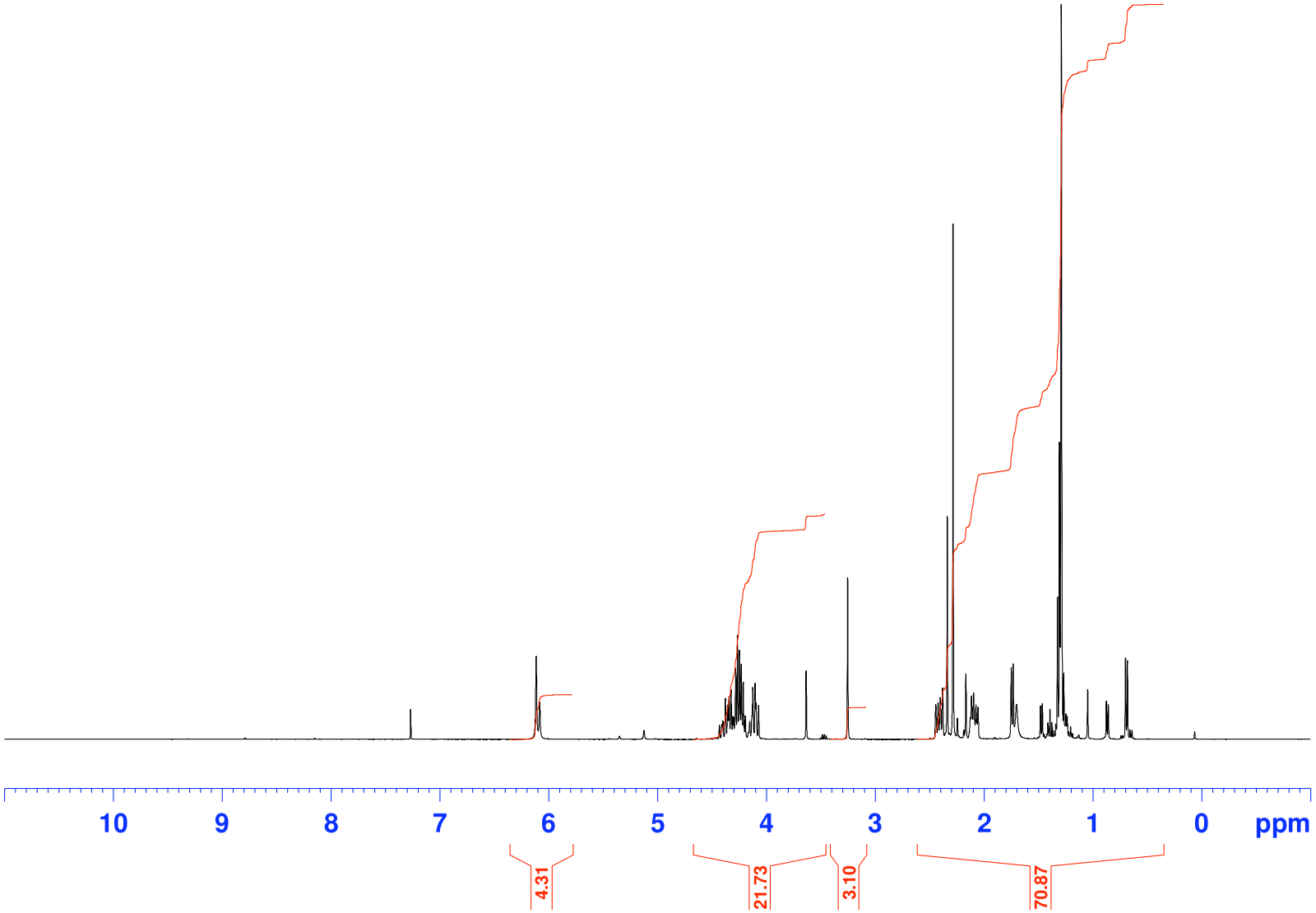


NMR@CHEM.OX

NAME	gf555741308
EXPNO	4
PROCNO	1
Date_	20090813
Time	23.52
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	912
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Instrument DQX400
Chemist GCF
Group JR
4–66 F25–35
h1acq.au CDCl3 {C:\NMR} jrgrp 11

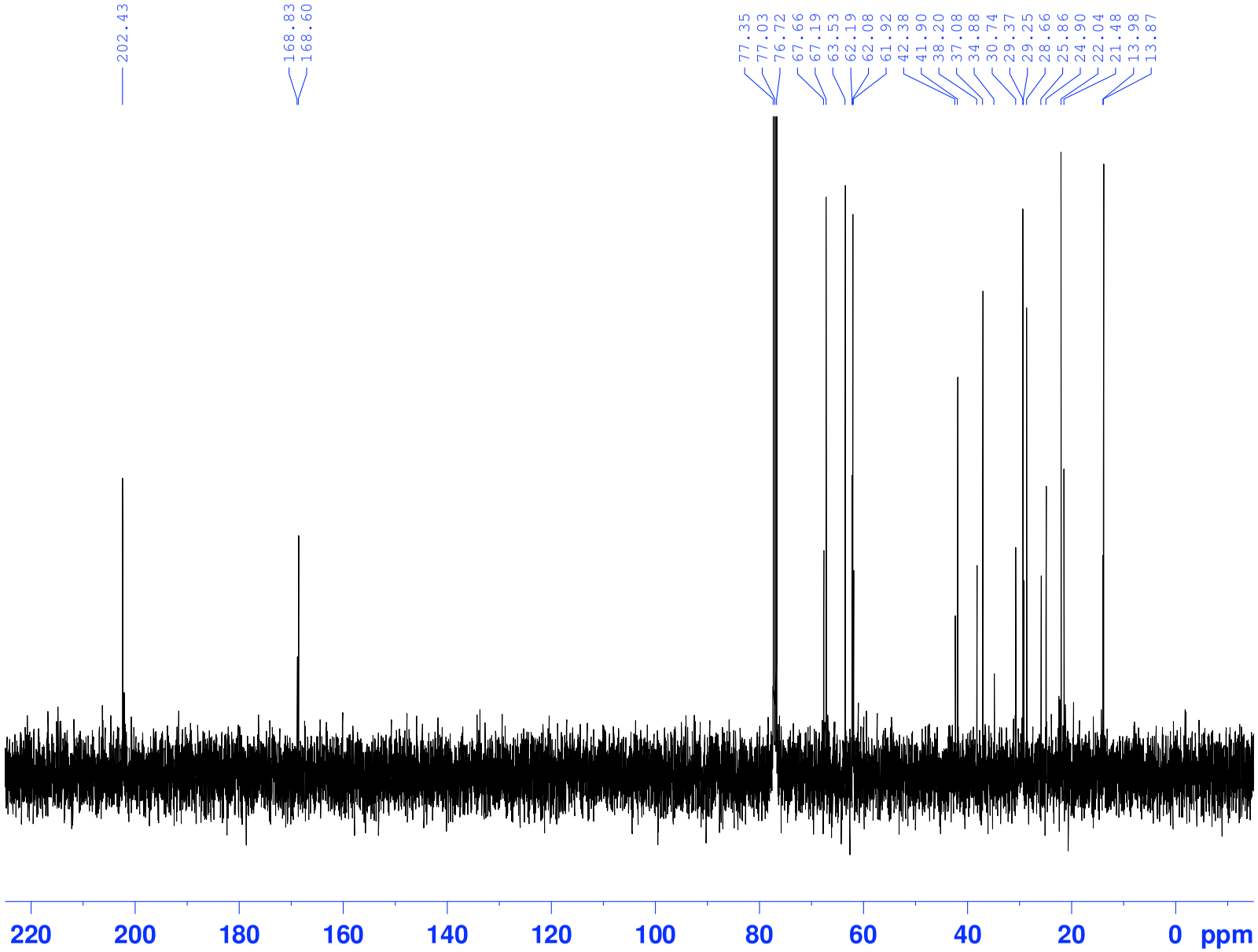




NMR@CHEM.OX

NAME	Sep15-2009-11
EXPNO	1
PROCNO	1
Date_	20090915
Time	9.50
INSTRUM	av400
PROBHD	5 mm QNP 1H/13
PULPROG	zg60
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	90.5
DW	60.400 usec
DE	7.50 usec
TE	300.0 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.00 usec
PL1	0.00 dB
SFO1	400.2024714 MHz
SI	32768
SF	400.2000028 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

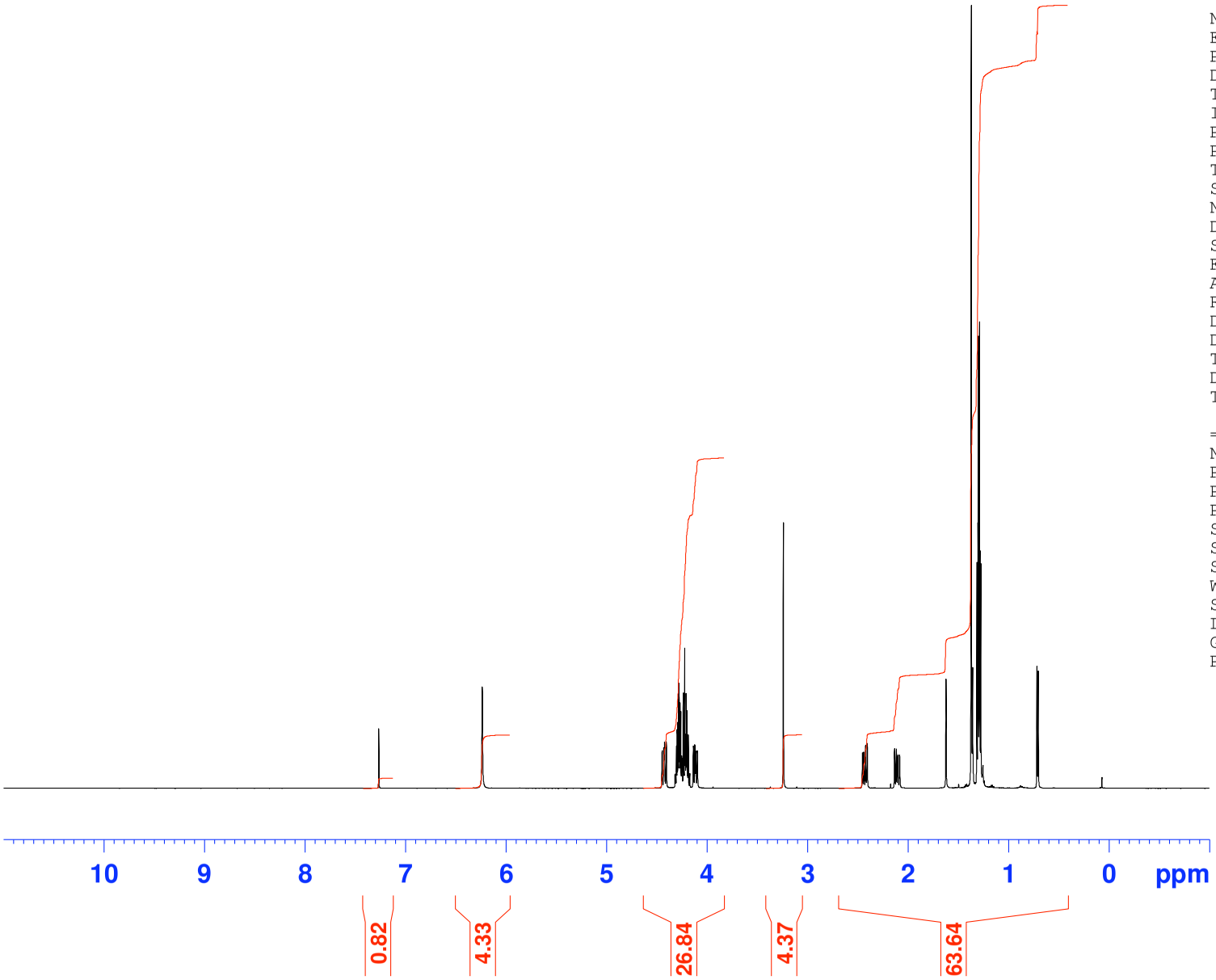
Instrument DQX400
Chemist GCF
Group JR
4–66 F25–35
c13acq.au CDCl3 {C:\NMR} jrgrp 11



NMR@CHEM.OX

NAME	Sep15-2009-11
EXPNO	2
PROCNO	1
Date_	20090915
Time	9.58
INSTRUM	av400
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	256
DS	4
SWH	26178.010 Hz
FIDRES	0.798889 Hz
AQ	0.6259188 sec
RG	32768
DW	19.100 usec
DE	7.50 usec
TE	300.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.50 usec
PL1	0.00 dB
SFO1	100.6403931 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	19.00 dB
PL13	25.00 dB
SFO2	400.2016008 MHz
SI	32768
SF	100.6303718 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

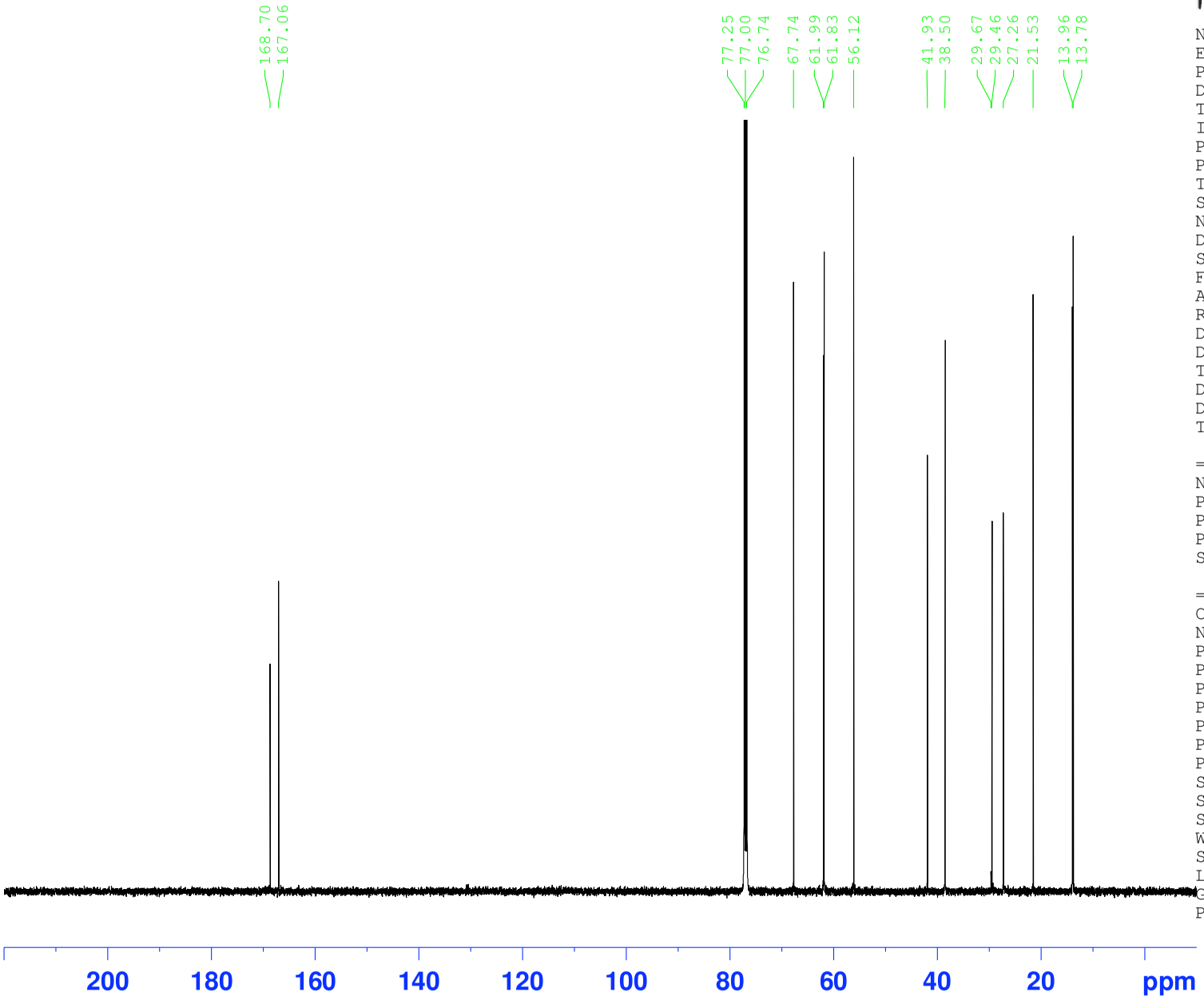
Instrument AVC500
6394 George Feast 27/10/09



NMR@CHEM.OX

NAME	gf63942710
EXPNO	1
PROCNO	1
Date_	20091027
Time	8.42
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	4
DW	48.400 usec
DE	6.00 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.60 usec
PL1	-6.00 dB
PL1W	15.19999981 W
SFO1	500.3030896 MHz
SI	32768
SF	500.3000240 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Instrument AVC500
6394 George Feast 27/10/09



NMR@CHEM.OX

NAME	gf63942710
EXPNO	4
PROCNO	1
Date_	20091027
Time	9.08
INSTRUM	avc500
PROBHD	5 mm CPDUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	31250.000 Hz
FIDRES	0.476837 Hz
AQ	1.0486259 sec
RG	1820
DW	16.000 usec
DE	20.00 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	8.00 usec
PL1	-4.40 dB
PL1W	28.15752029 W
SFO1	125.8131151 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	12.40 dB
PL13	17.00 dB
PL2W	15.19999981 W
PL12W	0.21970686 W
PL13W	0.07618046 W
SFO2	500.3020012 MHz
SI	32768
SF	125.8005438 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40