General experimental

Infra-red spectra were recorded on a Perkin-Elmer Paragon 1000 Fourier transform I.R. spectrometer.

NMR spectra were recorded using a Bruker AM360 or AM400 spectrometer in deuterochloroform, unless otherwise stated, referenced to TMS (δ 0). Chemical shifts are in parts per million (δ ppm). Coupling constants are in Hertz (J Hz). The following abbreviations are used: s-singlet, d-doublet, dd-double doublet, t-triplet, q-quartet, m-multiplet.

Mass spectra were recorded on a Jeol AX505W spectrometer (EI) and Kratos MS890 (FAB).

Products were isolated by flash chromatography using Merck silica gel 60 (4063 μm).[1] Analytical t.l.c was carried out on Merck (aluminium sheets) silica gel 60 F_{254} plates using short wave (254 nm) UV light, $KMnO_4$ or anisaldehyde to visualise components.

Irradiations were carried out under an argon atmosphere using an immersion well reactor (model RQ125 and RQ400, Photochemical Reactors Limited) and monitored by t.l.c.

Microwave experiments were carried out using sealed tubes in a CEM Discover microwave reactor.

Dichloromethane was freshly distilled from calcium hydride. Tetrahydrofuran was freshly distilled from sodium and benzophenone. Pyrrolidine was distilled from calcium hydride and stored over pellets of potassium hydroxide. *m*-CPBA was purified by dissolving in diethyl ether and then washing three times with a phosphate buffer solution. The solvent was carefully removed *in-vacuo* to give pure *m*-CPBA. All other reagents were used as received.

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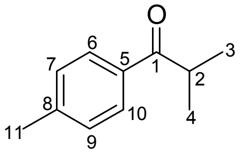
[1] W. C. Still, M. Kahn, A. Mitra, *J. Org. Chem*., 1978, **43**, 2923.

2-Methyl-1-*p*-tolyl-propan-1-one (5).



5

To powdered $AlCl_3$ (30.8 g, 224.99 mmol) in a 500 mL, three neck round bottom flask equipped with an argon inlet, magnetic stirrer, double face condenser, pressure equalising dropping funnel and a HCl gas outlet bubbling into water, was added toluene (67 mL, 629.96 mmol). Isobutyryl chloride (18.8 mL, 179.99 mmol) was then added dropwise to the yellow solution over a period of 30 minutes. Once the addition was complete the reaction mixture was heated to $60^{\circ}C$ for 3 hours. The resulting dark green/black mixture was then poured into water (400 mL) and ice. The organic phase was separated from the aqueous and the aqueous was extracted with ether (x3). The combined organic layers were washed with NaOH (x2, 10%), water (x1), brine (x1), dried over magnesium sulphate and concentrated under reduced pressure to give the crude product as an orange/brown oil. Fractional distillation through a vigreux column under water pump pressure afforded the title compound as a colourless oil (28.7g, 98%); ν_{max} (neat)/cm^{-1} 3029 (m), 2972 (m), 2932 (m), 2872 (m), 2360 (m), 1678 (s), 1607 (s), 1223 (s), 1161 (m), 977 (m), 830 (m); δ_H (360 MHz, $CDCl_3$) 7.87 (2H, d, *J* 8.0, 6, 10-H), 7.27 (2H, d, *J* 8.0, 7, 9-H), 3.56 (1H, m, 2-H), 2.20 (3H, s, 11-H), 1.21 (6H, d, *J* 6.9, 3, 4-H); δ_C (90 MHz, $CDCl_3$) 204.1 (1-C), 143.5 (5-C), 133.7 (8-C), 129.3 (6, 10-C), 128.4 (7, 9-C), 35.2 (2-C), 21.6 (11-C), 19.2 (3, 4-C); *m/z* (EI) 162 (M^+; 12), 119 (100), 105 (14.9), 91 (22%). All spectral data matched that reported in the literature.[2]

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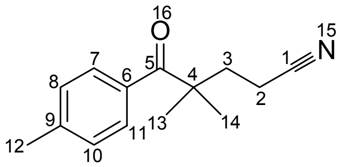
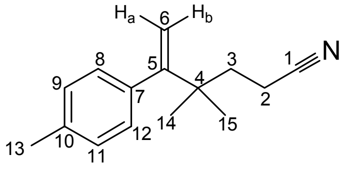
[2] A. R. Katritzky, W. Kuzmierkiewicz, *J. Chem. Soc., Perkin Trans. 1.*, 1987, 819-823.

4,4-Dimethyl-5-oxo-5-*p*-tolyl-pentane nitrile.



Acrylonitrile (1.72 g, 32.4 mmol) was added dropwise over 5 minutes to a stirred mixture of 2-methyl-1-*p*-tolyl-propan-1-one (4.8 g, 29.63 mmol), 1,4 dioxane (5 mL) and benzyltrimethylammonium hydroxide (40% w/w, 1.25 g, 2.99 mmol) kept at 30-35°C. The mixture was stirred for a further 18 hours, then acidified using hydrochloric acid, diluted with water and extracted with chloroform (x4). The combined organic extract were freed from dioxane by washing with water, and then dried over magnesium sulphate. The organic phase was concentrated under reduced pressure to give the crude product as a yellow oil. The residue was purified by column chromatography (hexane/diethyl ether 4:1) to afford the title compound as a colourless oil (4.33 g, 68%); R$_f$ 0.27 (hexane/diethyl ether 4:1); ν_{max} (neat)/cm^{-1} 2971 (s), 2875 (m), 2246 (s, nitrile), 1669 (s, C=O), 961 (s), 835 (m); δ_H (360 MHz, $CDCl_3$) 7.65 (2H, d, *J* 8.2, 7, 11-H), 7.24 (2H, d, *J* 8.2, 8, 10-H), 2.39 (3H, s, 12-H), 2.31 (2H, dd, *J* 9.0 and 8.9, 2-H), 2.14 (2H, dd, *J* 9.0 and 8.9, 3-H), 1.39 (3H, s, 13, 14-H); δ_C (90 MHz, $CDCl_3$) 206.4 (5-C), 142.8 (6-C), 135.2 (9-C), 129.4 (7, 11-C), 128.6 (8, 10-C), 120.3 (1-C), 47.3 (4-C), 37.0 (3-C), 26.3 (13, 14-C), 21.9 (12-C), 13.5 (3-C); *m/z* (FAB) 216 ([M+H]$^+$; 46), 161 (5), 119 (100%); HRMS *m/z* (FAB) calculated for $C_{14}H_{18}NO$, 216.1388 ([M+H]$^+$), found 216.1394.

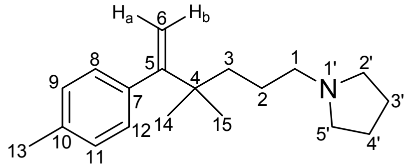
4,4-Dimethyl-5-*p*-tolyl-hex-5-enenitrile (7).



7

Potassium tert-butoxide (0.77 g, 6.89 mmol), methyltriphenylphosphonium bromide (2.46 g, 6.89 mmol) and toluene (30 mL) were placed in a 100 mL two neck round bottom flask, fitted with a condenser and under an argon atmosphere. The mixture was refluxed with stirring for 2 hours at which point it became bright yellow. To this was added a solution of 4,4-dimethyl-5-oxo-5-*p*-tolyl-pentane nitrile (1.14 g, 5.3 mmol) in toluene (5 mL) dropwise at ~35°C. The resulting brown/orange mixture was then refluxed for a further 6 hours and then left to stir overnight at 40°C for 18 hours. The reaction mixture was diluted with diethyl ether (20 mL) and water (10 mL), stirred for a further 5 minutes and the organic layer was separated from the aqueous. The aqueous layer was washed with diethyl ether (10 mL x2) and the combined organic layers were dried over magnesium sulphate and rotary evaporated to give a yellow oil. The crude product was purified by flash column chromatography (diethyl ether/hexane 1:4) to afford the title compound as a colourless oil (0.7 g, 62%); R_f 0.23 (diethyl ether/hexane 1:4); ν_{max} (neat)/cm^{-1} 2969 (s), 2873 (m), 2246 (s, nitrile), 1626 (m, C=C), 1512 (s), 1384 (s), 911 (s), 826 (s); δ_H (360 MHz, $CDCl_3$) 7.12 (2H, d, *J* 8.0, 8, 12-H), 6.98 (2H, d, *J* 8.0, 9, 11-H), 5.16 (1H, d, *J* 1.2, 6-H_a), 4.96 (1H, d, *J* 1.1, 6-H_b), 2.35 (3H, s, 13-H), 2.33 (2H, dd, *J* 9.8 and 8.3, 2-H), 1.78 (2H, dd, *J* 8.3 and 9.8, 3-H), 1.13 (6-H, s, 14, 15-H); δ_C (90 MHz, $CDCl_3$) 155.8 (5-C), 139.5 (7-C), 136.9 (10-C), 128.9 (8, 12-C), 128.8 (9, 11-C), 120.8 (1-C), 115.5 (6-C), 39.5 (4-C), 36.5 (2-C), 27.6 (14, 15-C), 21.5 (13-C), 13.3 (3-C); *m/z* (FAB) 214 ($[M+H]^+$; 100), 159 (25), 119 (79), 105 (85%); HRMS *m/z* (FAB) calculated for $C_{15}H_{20}NO$, 214.1596 ($[M+H]^+$), found 214.1604.

1-(4,4-Dimethyl-5-*p*-tolyl-hex-5-enyl)-pyrrolidine (8).



8

Method 1: To a solution of alkyl bromide **6**[3] (1 g, 3.56 mmol) in absolute ethanol (10 mL) was sequentially added pyrrolidine (386 μL, 4.62 mmol) and anhydrous sodium carbonate (3.77 g, 35.6 mmol). The resulting suspension was refluxed for 4 days. The reaction mixture was diluted with EtOAc, filtered, concentrated in vacuo and finally purified by flash column chromatography on silica gel (triethylamine/hexane 1:95) to afford compound **8** (0.91 g, 95%) as a colourless oil.

Method 2: In an oven dried, 25 mL single necked round bottom flask fitted with a condenser and under an argon atmosphere was added 4,4-dimethyl-5-*p*-tolyl-hex-5-enenitrile **7** (0.523 g, 2.46 mmol) and copper (I) chloride (0.27 g, 2.73 mmol). To this was then added a solution of pyrrolidine (306 μL, 3.69 mmol) in ethanol (10 mL) and the resulting green/brown mixture was then refluxed for 24 hours. The dark brown/red mixture was then cooled down to room temperature and poured with vigorous stirring into a Erlenmeyer flask (100 mL) containing aqueous NaOH (10 mL, 30%) and diethyl ether (20 mL). The mixture was stirred vigorously for 5 minutes. The organic layer was then separated and the aqueous layer was extracted with diethyl ether (x3). The combined organic extracts were dried over magnesium sulphate and rotary evaporated to afford the crude amidine - 4,4-dimethyl-1-pyrrolidine-1-yl-5-*p*-tolyl-hex-5-enylideneamine as a brown oil (0.69 g).

The crude amidine (0.69 g, 2.43 mmol) was then transferred to an oven dried 25 mL two neck round bottom flask, equipped with a magnetic stirrer bar and under an argon atmosphere. To this was then added ethanol (10 mL) via syringe and the reaction mixture cooled to 0°C. Sodium borohydride (0.11 g, 2.95 mmol) was then added with stirring in small portions. The solution was then left to stir at room temperature for 12 hours. Upon completion the mixture was poured into an Erlenmeyer flask (100 mL) containing NaOH (10 mL, 30%) and diethyl ether (20 mL). The organic layer was separated and the aqueous layer was extracted with diethyl ether (x3). The combined organic extracts were dried over magnesium sulphate, filtered and solvent removed under reduced pressure. The residue (dark brown oil) was purified by column chromatography (hexane/triethylamine 95:5) to afford the title compound **8** (0.56 g, 84%) as a colourless oil; R_f 0.45 (hexane/triethylamine 95:5); ν_{max} (neat)/cm^{-1} 2964 (s), 2873 (m), 2785 (s), 1625 (m, C=C), 1511 (m), 1449 (s), 1380 (s), 904 (s), 835 (s); δ_H (360 MHz, $CDCl_3$) 7.09 (2H, d, *J* 7.9, 8, 12-H), 7.02 (2H, d, *J* 8.1, 9, 11-H), 5.11 (1H, d, *J* 1.7, 6-H_a), 4.84 (1H, d, *J* 1.7, 6-H_b), 2.51-2.46 (4H, m, 2', 5'-H), 2.38 (2H, d, *J* 7.8, 1-H), 2.34 (3H, s, 13-H), 1.79-1.75 (4H, m, 3', 4'-H), 1.58-1.49 (2H, m, 2-H), 1.35-1.30 (2H, m, 3-H), 1.08 (6H, s, 14, 15-H); δ_C (90 MHz, $CDCl_3$) 157.9 (5-C), 140.9 (7-C), 136.2 (10-C), 129.1 (8, 12-C), 128.4 (9, 11-C), 113.8 (6-C), 57.6 (1-C), 54.7 (2', 5'-C), 39.5 (4-C), 39.1 (2-C), 28.2 (14, 15-C), 24.9 (3-C), 23.8 (3', 4'-C), 21.5 (13-C); *m/z* (EI) 271 (M^+, 30), 256 (13), 200 (16), 152 (20), 143 (23), 110 (80), 84 (100%); HRMS *m/z* (EI) calculated for $C_{19}H_{29}N$, 271.22999 (M^+), found 271.2295.

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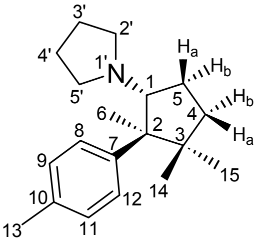
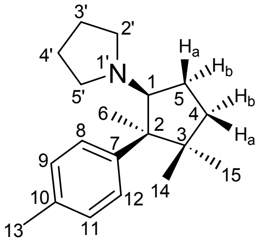
[3] A. Srikrishna, G. Sundarababu, *Tetrahedron*, 1991, **47**, 481-496.

(1*RS*, 2*SR*)-1-(2,3,3-Trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine (9) and (1*SR*, 2*SR*)-1-(2,3,3-Trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine (10).



9



10

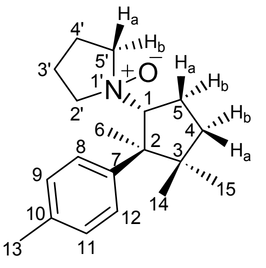
A hexane solution (280 mL, 0.005M) of styryl amine 1-(4,4-dimethyl-5-ρ-tolyl-hex-5-enyl)-pyrrolidine **8** (350 mg, 1.29 mmol) was placed in a quartz vessel and purged with argon for 1 hour. The solution was then irradiated with a medium pressure 400W mercury lamp for 50 minutes. NMR analysis of the irradiated sample showed 100% conversion and the formation of two products **9** and **10** in a 6:1 ratio respectively. After removal of the solvent under reduced pressure, the residue, a deep yellow oil, was chromatographed on silica gel (hexane/diethyl ether 1:1 or hexane/diethyl amine 19:1) to afford diastereomers **9** (215 mg, 62%) and **10** (25 mg, 7%) as pale yellow oils.

Data for **9**: R_f 0.50 (hexane/diethyl amine 19:1); ν_{max} (neat)/cm^{-1} 2957 (s, C-H), 2786 (m), 1515 (m), 1469 (m), 1112 (w), 811 (m); δ_H (360 MHz, $CDCl_3$) 7.26 (2H, d, *J* 8.3, 8, 12-H), 6.99 (2H, d, *J* 8.1, 9, 11-H), 3.39 (1H, t, *J* 8.1, 1-H), 2.27-2.20 (2H, m, 2' or 5'-H), 2.25 (3H, s, 13-H), 2.19-2.06 (2H, m, 2' or 5'-H), 2.05-1.98 (1H, m, 5-H_a or 5-H_b), 1.78-1.68 (2H, m, 5-H_a or 5-H_b and 4H_a or 4-H_b), 1.60-1.49 (4H, m, 3', 4'-H), 1.47-1.43 (1H, m, 4-H_a or 4-H_b), 1.29 (3H, s, 6-H), 0.70 (3H, s, 15-H), 0.62 (3H, s, 14-H); δ_C (90 MHz, $CDCl_3$) 141.9 (7-C), 135.7 (10-C), 128.6 (8, 12-C), 128.4 (9, 11-C), 70.2 (1-C), 55.1 (2', 5'-C), 53.8 (2-C), 46.2 (3-C), 38.4 (4-C), 29.3 (5-C), 28.9 (14-C), 24.4 (15-C), 23.9 (3', 4'-C), 21.8 (13-C), 17.3 (6-C); *m/z* (EI) 271 (M^+, 12), 205 (10), 159 (5), 129 (30), 111 (12), 110 (100), 96 (11%); HRMS *m/z* (EI) calculated for $C_{19}H_{29}N$, 271.23001 (M^+), found 271.22939.

Data for **10**: R_f 0.52 (hexane/diethyl amine 19:1); ν_{max} (neat)/cm^{-1} 2968 (s, C-H), 2873 (w), 2778 (m), 1515 (m), 1458 (m), 1085 (m); δ_H (360 MHz, $CDCl_3$) 7.34 (2H, d, *J*

8.3, 8, 12-H), 7.01 (2H, d, *J* 8.1, 9, 11-H), 2.75 (1H, dd, *J* 6.7 and 10.2, 1-H), 2.49-2.36 (2H, m, 2' or 5'-H), 2.30 (3H, s, 13-H), 2.24-2.18 (1H, m, 5-H_a or 5-H_b), 2.17-2.07 (2H, m, 2' or 5'-H), 1.95-1.89 (1H, m, 4-H_a or 4-H_b or 5-H_a or 5-H_b), 1.78-1.69 (1H, m, 4-H_a or 4-H_b or 5-H_a or 5-H_b), 1.59-1.47 (5H, m, 3', 4'-H and 4-H_a or 4-H_b or 5-H_a or 5-H_b), 1.01 (3H, s, 15-H), 0.40 (3H, s, 14-H); δ_C (90 MHz, $CDCl_3$) 139.3 (7-C), 132.4 (10-C), 127.3 (8, 12-C), 125.1 (9, 11-C), 74.9 (1-C), 51.8 (2', 5'-C), 51.3 (2-C), 44.7 (3-C), 35.8 (4 or 5-C), 29.5 (5 or 4-C), 23.9 (15-C), 23.1 (14-C), 22.7 (6-C), 21.3 (3', 4'-C), 19.1 (13-C); *m/z* (EI) 271 (M^+, 11), 111 (11), 110 (100), 96 (11%); HRMS *m/z* (EI), calculated for $C_{19}H_{29}N$, 271.23001 (M^+), found 271.22944.

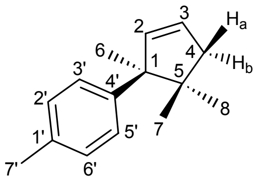
(1*RS*, 2*SR*)-1-(2,3,3-Trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine-1'-oxide (11).



11

To (1*RS*, 2*SR*)-1-(2,3,3-trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine **9** (213 mg, 0.79 mmol) dissolved in dichloromethane (5 mL) at 0°C was added dropwise a solution of *m*-chloroperbenzoic acid (0.16 g, 0.94 mmol) in dichloromethane (3 mL). Once the addition was complete the reaction mixture was brought to room temperature and stired for 3 hours. The reaction was then quenched with $Na_2S_2O_3$ (10 mL, 10%) and washed with saturated aqueous $NaHCO_3$ (3 x 15 mL). The combined organic extracts were dried over magnesium sulphate and the solvent removed in vacuo. The resulting pale brown sticky solid/oil was purified by flash column chromatography (methanol/ethyl acetate 1:1) to yield the pure amine oxide **11** (188 mg, 83%) as a white fluffy sticky solid/oil; R_f 0.17 (methanol/ethyl acetate 1:1); ν_{max} (neat)/cm^{-1} 2924 (s), 2854 (m), 1466 (m, N=O), 1381 (w), 812 (w); δ_H (360 MHz, $CDCl_3$) 7.30 (2H, d, *J* 8.5, 8, 12-H), 7.12 (2H, d, *J* 7.9, 9, 11-H), 4.45 (1H, t, *J* 8.9, 1-H), 3.48 (1H, q, *J* 8.5, 5'-H_a or H_b), 3.13-3.09 (1H, m, 5'-H_a or H_b), 3.01-2.98 (1H, m, 5-H_b), 2.93-2.88 (2H, m, 3'-H), 2.36-2.29 (2H, m, 3' or 4'-H), 2.33 (3H,s, 13-H), 2.19-2.13 (1H, m, 5-H_a), 2.02-1.96 (1H, m, 4-H_b), 1.80-1.74 (2H, m, 3' or 4'-H), 1.74 (3H, s, 6-H), 1.65-1.61 (1H, m, 4-H_a), 0.79 (3H, s, 15-H), 0.76 (3H, s, 14-H); δ_C (90 MHz, $CDCl_3$) 139.8 (7-C), 136.1 (10-C), 128.0 (8, 9, 11, 12-C), 79.9 (1-C), 71.8 (5'-C), 67.2 (3'-C), 53.8 (2-C), 46.8 (3-C), 37.0 (4-C), 28.1 (14-C), 23.4 (15-C), 22.5 (3' or 4'-C), 22.2 (5-C), 21.2 (13-C), 21.0 (3' or 4'-C), 18.1 (6-C); *m/z* (FAB) 288 ($[M+H]^+$, 100), 270 (11), (13), 165 (12), 154 (12), 145 (69%); HRMS *m/z* (FAB) calculated for $C_{19}H_{30}NO$, 288.2327 ($[M+H]^+$), found 288.2320.

(1*SR*)-1'-Methyl-4'-(1,5,5-trimethyl-cyclopent-2-enyl)-benzene (12).

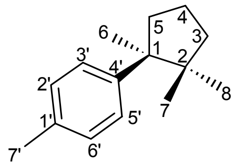


12

Method 1: To a 25ml flame dried two neck round bottom flask, under an argon atmosphere was added (1*RS*, 2*SR*)-1-(2,3,3-trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine-1'-oxide **11** (28.5 mg, 0.1 mmol) and THF (4 mL). The colourless solution was then heated to 60°C for 18 hours. The resulting pale yellow solution was cooled to 25°C, concentrated in vacuo and chromatographed over silica gel (hexane) to afford the title compound **12** as a colourless oil (8 mg, 40%).

Method 2: (1*RS*, 2*SR*)-1-(2,3,3-Trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine-1'-oxide **11** (8.6 mg, 0.03 mmol) in deuterated DMSO (0.3 mL) was placed in a microwave tube. The colourless solution was then irradiated (100W) in the chamber for 1 minute which raised the temperature from 25°C to 200°C. The resulting dark brown solution was then cooled, diluted with diethyl ether (10 mL) and washed with water (2 x 10 mL). The organic layer was removed and the aqueous was extracted with diethyl ether (3 x 5 mL). The combined organic extracts were washed with brine, dried over magnesium sulphate, filtered and rotary evaporated to give the crude product. Purification by silica gel chromatography (hexane) afforded **12** as a colourless oil (4.3 mg, 72%); R_f 0.79 (hexane); ν_{max} (thin film)/cm^{-1} 3052 (m, C-H), 3024 (m), 2969 (s), 2925 (s), 1514 (s), 1456 (s), 810 (m), 722 (m); δ_H (360 MHz, DMSO) 7.14 (2H, d, J 8.3, 3', 5'-H), 7.08 (2H, d, J 8.1, 2', 6'-H), 5.86-5.84 (1H, m, 2 or 3-H), 5.81-5.79 (1H, m, 3 or 2-H), 2.34-2.29 (1H, m, 4-H$_a$ or 4-H$_b$), 2.26 (3H, s, 7'-H), 2.18-2.07 (1H, m, 4-H$_a$ or 4-H$_b$), 1.22 (3H, s, 6-H), 1.09 (3H, s, 8-H), 0.38 (3H, s, 7-H); δ_C (90 MHz, $CDCl_3$) 143.0 (4'-C), 140.6 (2 or 3-C), 135.6 (1'-C), 128.8 (3', 5'-C), 128.8 (2 or 3-C), 127.1 (2', 6'-C), 56.4 (1-C), 47.8 (4-C), 44.8 (5-C), 28.4 (6 or 7 or 8-C), 24.3 (6 or 7 or 8-C), 23.5 (6 or 7 or 8-C), 21.3 (7'-C); m/z (EI) 201 ([M+H]$^+$, 9), 200 (M$^+$, 49), 186 (7), 185 (36), 157 (100), 143 (23), 128 (12), 105 (11), 84 (20%); HRMS *m/z* (FAB) calculated for $C_{15}H_{20}$, 200.15660 (M$^+$), found 200.15627.

(±)-Cuparene.



(±)-Cuparene

To a solution of 1'-methyl-4'-(1,5,5-trimethyl-cyclopent-2-enyl)-benzene **12** (56 mg, 0.28 mmol) in EtOAc (15 mL) was added 5% Pd on charcoal (20 mg). The mixture was stirred under atmospheric hydrogen for 3 hours. The catalyst was removed by filtration through celite and washed well with EtOAc. The combined filtrate and washings were concentrated in vacuo. The resulting yellow oil was purified by flash column chromatography (hexane) to afford the title compound as a colourless oil (46.5 mg, 82%); R_f 0.66 (hexane); ν_{max} (neat)/cm^{-1} 3094 (w), 3061 (w), 3025 (w), 2969 (s), 2874 (s), 2727 (w), 1898 (w), 1790 (w), 1516 (s), 1459 (s), 1374 (s), 1193 (m), 1135 (w), 1108 (w), 1020 (m), 812 (s), 724 (m), 547 (s); δ_H (360 MHz, $CDCl_3$) 7.23 (2H, d, *J* 8.2, 3', 5'-H), 7.08 (2H, d, *J* 8.1, 2', 6'-H), 2.53-2.45 (1H, m, 5-H_a or H_b), 2.31 (3H, s, 7'-H), 1.83-1.64 (4H, m, 3,4-H), 1.61-1.43 (1H, m, 5-H_a or H_b), 1.25 (3H, s, 6-H), 1.06 (3H, s, 8-H), 0.56 (3H, s, 7-H); δ_C (90 MHz, $CDCl_3$) 144.9 (4'-C), 135.1 (1'-C), 128.6 (3', 5'-C), 127.3 (2', 6'-C), 50.7 (1-C), 44.6 (2-C), 40.1 (3 or 4 or 5-C), 37.2 (3 or 4 or 5-C), 26.9 (6-C), 24.8 (7 or 8-C), 24.7 (7 or 8-C), 21.3 (7'-C), 20.2 (3 or 4 or 5-C); m/z (EI) 203 ([M+H]$^+$, 7), 202 (M$^+$, 42), 145 (35), 133 (27), 132 (100), 131 (33), 120 (15), 119 (29), 117 (11), 105 (17%). All spectroscopic data matched that reported in the literature.[4]

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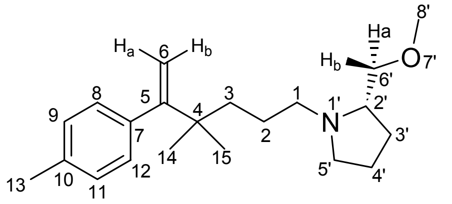
[4] C. a) Enzell, H. Erdtman, *Tetrahedron*, 1958, **4**, 361-368. b) B. R. Aavula, Q. Cui, E. A. Mash, *Tetrahedron: Asymmetry*, 2000, **11**, 4681-4686.

(2*S*)-1-(4,4-Dimethyl-5-*p*-tolyl-hex-5-enyl)-2'-methoxymethyl-pyrrolidine.



14

To a solution of alkyl bromide **6**[3] (123 mg, 0.44 mmol) in absolute ethanol (3 mL) was sequentially added (*S*)-(+)-2-methoxymethyl-pyrrolidine[5] (74 μL, 0.60 mmol) and anhydrous sodium carbonate (0.47 g, 4.4 mmol). The resulting suspension was heated at 150°C in a sealed tube for 48 hours. The reaction mixture was diluted with EtOAc, filtered, concentrated in vacuo and finally purified by flash column chromatography on silica gel (triethylamine/hexane 1:95) to afford the alkylated product **14** (107 mg, 77%) as a colourless oil; R_f 0.15 (triethylamine/hexane 1:95); ν_{max} (neat)/cm^{-1} 3086 (w), 3023 (w), 2964 (s), 2871 (s), 2806 (m), 1625 (w), 1512 (m), 1459 (m), 1112 (s), 904 (m), 824 (s); δ_H (360 MHz, $CDCl_3$) 7.07 (2H, d, *J* 8.0, 9, 11-H), 7.01 (2H, d, *J* 8.1, 8, 12-H), 5.10 (1H, d, *J* 1.7, 6-H_b), 4.83 (1H, d, *J* 1.7, 6-H_a), 3.40 (1H, dd, *J* 4.7 and 9.3, 6'-H_a or H_b), 3.35 (3H, s, 8'-H), 3.27 (1H, dd, *J* 4.7 and 9.3, 6'-H_a or H_b), 3.17-3.13 (1H, m, 5'-H_a or H_b), 2.78-2.71 (1H, m, 1-H_a or H_b), 2.56-2.49 (1H, m, 2'-H), 2.34 (3H, s, 13-H), 2.25-2.17 (1H, m, 1-H_a or H_b), 2.16-2.11 (1H, m, 5'-H_a or H_b), 1.94-1.60 (4H, m, 3', 4'-H), 1.59-1.43 (2H, m, 2 or 3-H), 1.39-1.26 (2H, m, 2 or 3-H), 1.08 (6H, s, 14-15-H); δ_C (90 MHz, $CDCl_3$) 157.9 (5-C), 140.8 (7-C), 136.1 (10-C), 129.0 (9, 11-C), 128.4 (8, 12-C), 113.8 (6-C), 76.5 (6'-C), 64.1 (2'-C), 59.4 (8'-C), 56.6 (1-C), 54.9 (5'-C), 39.5 (4-C), 38.9 (2 or 3-C), 28.7 (3' or 4'-C), 28.2 (14 or 15-C), 28.0 (15 or 14-C), 24.5 (2 or 3-C), 23.2 (3' or 4'-C), 21.4 (13-C); *m/z* (FAB) 316 ($[M+H]^+$, 96), 315 (M^+, 7), 314 (55), 282 (10), 271 (16), 270 (100), 154 (29), 133 (10), 128 (22), 119 (16), 110 (12), 105 (22%); HRMS *m/z* (FAB calculated for $C_{21}H_{34}NO$, 316.2640 ($[M+H]^+$), found 316.2650; $[\alpha]^{20}_D$ –64.7 (*c* 1.31, $CHCl_3$).

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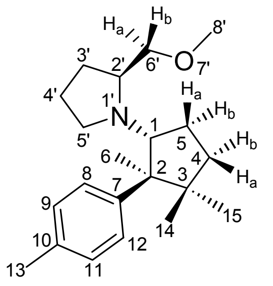
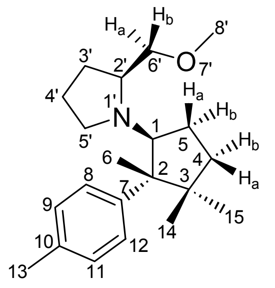
[5] D. Enders, M. Klatt, *Synthesis*, 1996, 1403-1418.

(2’*S*, 1*R*, 2*S*)- 2’-Methoxymethyl–1-(2,3,3-trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine (15) and (2’*S*, 1*S*, 2*R*)- 2’-Methoxymethyl-1-(2,3,3-trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine (16).



15



16

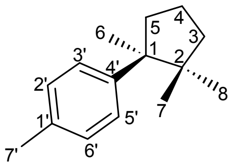
A hexane solution (280 mL, 0.01M) of styryl amine **14** (1 g, 3.17 mmol) was placed in a quartz vessel and purged with argon for 1 hour. This solution was then irradiated with a medium pressure 400W mercury lamp for 3 hours. NMR analysis of the irradiated sample showed 100% conversion to a mixture of diastereomers in a 10:5:2:1 ratio. After removal of the solvent under reduced pressure, the residue a yellow oil was chromatographed on silica gel (hexane-hexane/diethyl ether 1:1). Two major diastereomers were isolated **16** (84 mg, 8%) and **15** (322 mg, 36%) as colourless oils.

Data for **16**: R_f 0.33 (hexane/diethyl amine 19:1); ν_{max} (neat)/cm^{-1} 3060 (w), 2958 (s), 2873 (s), 2822 (s), 2360 (w), 2340 (w), 1515 (m), 1463 (m), 1196 (m), 1113 (s), 811 (m); δ_H (360 MHz, $CDCl_3$) 7.31 (2H, d, *J* 8.2, 8, 12-H), 7.05 (2H, d, *J* 8.1, 9, 11-H), 3.96 (1H, t, *J* 8.6, 1-H), 3.36-3.29 (1H, m, 6’-H_a or H_b), 3.33 (3H, s, 8’-H), 3.06-3.00 (2H, m, 6’-H_a or H_b and 2’-H), 2.35-2.26 (1H, m, 5’-H_a or H_b), 2.31 (3H, s, 13-H), 2.19-2.14 (1H, m, 5’-H_a or H_b), 2.11-2.03 (1H, m, 5-H_b), 1.93-1.89 (1H, m, 5-H_a), 1.78-1.74 (1H, m, 4-H_a or H_b), 1.70-1.65 (2H, m, 3’-H), 1.55-1.40 (3H, m, 4-H_a or H_b and 4’-H), 1.30 (3H, s, 6-H), 0.80 (3H, s, 14-H), 0.67 (3H, s, 15-H); δ_C (90 MHz, $CDCl_3$) 142.0 (10-C), 135.0 (7-C), 128.2 (8, 12-C), 127.9 (9, 11-C), 78.2 (6’-C), 68.4 (1-C), 61.9 (2’-C), 59.3 (8’-C), 54.8 (5’-C), 54.0 (2-C), 45.0 (3-C), 38.0 (4-C), 29.0 (3’-C), 28.7 (15-C), 24.5 (4’-C), 24.3 (14-C), 21.3 (13-C), 16.9 (6-C); *m/z* (EI) 315

(M^+, 8), 270 (37), 155 (17), 154 (100), 110 (10%); HRMS *m/z* (EI) calculated for $C_{21}H_{33}NO$, 315.25623 (M^+), found 315.25714; $[\alpha]^{20}_{D}$ +37.4 (*c* 1.2, $CHCl_3$).

Data for **15**: R_f 0.30 (hexane/diethyl amine 19:1); ν_{max} (neat)/cm^{-1} 2956 (s), 2872 (s), 2824 (m), 2360 (m), 2341 (m), 1515 (m), 1467 (m), 1376 (w), 1196 (w), 1112 (s), 1017 (w), 812 (m); δ_H (360 MHz, $CDCl_3$) 7.34 (2H, d, *J* 8.2, 8, 12-H), 7.08 (2H, d, *J* 8.1, 9, 11-H), 4.14 (1H, t, *J* 8.7, 1-H), 3.32 (1H, dd, *J* 8.5 and 4.0, 6'-H_a or H_b), 3.23 (3H, s, 8'-H), 3.15-3.09 (2H, m, 6'-H_a or H_b and 2'-H), 2.60-2.50 (2H, m, 5'-H), 2.31 (3H, s, 13-H), 2.12-2.06 (1H, m, 5-H_a), 1.86-1.77 (1H, m, 5-H_b), 1.75-1.71 (2H, m, 4-H_b and 3' or 4'-H), 1.63-1.49 (4H, m, 4-H_a and 3', 4'-H), 1.30 (3H, s, 6-H), 0.82 (3H, s, 15-H), 0.64 (3H, s, 14-H); δ_C (90 MHz, $CDCl_3$) 141.9 (7-C), 135.1 (10-C), 128.2 (8, 12-C), 128.0 (9, 11-C), 74.8 (6'-C), 65.1 (1-C), 61.8 (2'-C), 59.2 (8'-C), 53.4 (2 or 3-C), 51.1 (5'-C), 44.8 (3 or 2-C), 37.3 (3' or 4'-C), 28.5 (14-C), 27.9 (4-C), 24.5 (5-C), 24.4 (15-C), 24.0 (3' or 4'-C), 21.3 (13-C), 17.4 (6-C); *m/z* (EI) 315 (M^+, 8), 270 (37), 155 (17), 154 (100), 110 (10%); HRMS *m/z* (EI) calculated for $C_{21}H_{33}NO$, 315.25623 (M^+), found 315.25728; $[\alpha]^{20}_{D}$–83.9 (*c* 1.22, $CHCl_3$).

(*S*)-(-)- Cuparene.



(*S*)-(-)- Cuparene

To (2'*S*, 1*R*, 2*S*)-2'-Methoxymethyl-1-(2,3,3-trimethyl-2-*p*-tolyl-cyclopentyl)-pyrrolidine **15** (30 mg, 0.095 mmol) dissolved in dichloromethane (1 mL) at 0°C was added dropwise a solution of *m*-chloroperbenzoic acid (20 mg, 0.11 mmol) in dichloromethane (1 mL). Once the addition was complete the reaction was brought to room temperature and stirred for 3 hours. The reaction was then quenched with $Na_2S_2O_3$ and washed with saturated aqueous $NaHCO_3$. The combined organic extracts were dried over magnesium sulphate and the solvent removed in vacuo to give the crude amine oxide as a pale brown sticky oil (30 mg). The crude amine oxide (30 mg) was then transferred to a microwave tube using deuterated DMSO (0.8 mL). The pale yellow solution was then irradiated (100W) in the chamber for 1 minute which raised the temperature to 200°C. The resulting dark brown solution was then cooled, diluted with diethyl ether and washed with water. The organic layer was removed and the aqueous was extracted with diethyl ether. The combined organic extracts were washed with brine, dried over magnesium sulphate, filtered and rotary evaporated to give the crude alkene as a orange/brown oil (29.8 mg) which was used in the next step without further purification. To a solution of the crude alkene (29.8 mg) in EtOAc (10 mL) was added 5% Pd on charcoal (12 mg). The mixture was stirred under atmospheric hydrogen for 3 hours. The catalyst was removed by filtration through celite. The combined filtrate and washings were concentrated in vacuo to afford a pale yellow oil, which was purified by flash column chromatography (hexane) to give the title compound as a colourless oil (4.6 mg, 24% over 3 steps); R_f 0.66 (hexane); ν_{max} (neat)/cm^{-1} 3094 (m), 3061 (m), 3025 (m), 2969 (s), 2874 (s), 2727 (m), 1898 (m), 1790 (m), 1516 (s), 1459 (s), 1374 (s), 1193 (m), 1135 (w), 1108 (w), 1020 (m); δ_H (360 MHz, $CDCl_3$) 7.23 (2H, d, J 8.2, 3', 5'-H), 7.08 (2H, d, J 8.1, 2', 6'-H), 2.53-2.45 (1H, m, 5-H_a or H_b), 2.31 (3H, s, 7'-H), 1.83-

1.64 (4H, m, 3, 4-H), 1.61-1.43 (1H, m, 5-H_a or H_b), 1.25 (3H, s, 6-H), 1.06 (3H, s, 8-H), 0.56 (3H, s, 7-H); δ_C (90 MHz, $CDCl_3$) 144.9 (4'-C), 135.1 (1'-C), 128.6 (3', 5'-C), 127.3 (2', 6'-C), 50.7 (1-C), 44.6 (2-C), 40.1 (3 or 4 or 5-C), 37.2 (3 or 4 or 5-C), 26.9 (6-C), 24.8 (7 or 8-C), 24.7 (7 or 8-C), 21.3 (7'-C), 20.2 (3 or 4 or 5-C); m/z (EI) 203 ($[M+H]^+$, 7), 202 (M^+, 42), 145 (35), 133 (27), 132 (100), 131 (33), 120 (15), 119 (29), 117 (11), 105 (17%); $[\alpha]^{20}_D$ –62.2 (*c* 0.23, $CDCl_3$), lit.[6] $[\alpha]^{20}_D$ –63 (*c* 1.6, $CHCl_3$). All spectroscopic data matched that reported in the literature.[4b]

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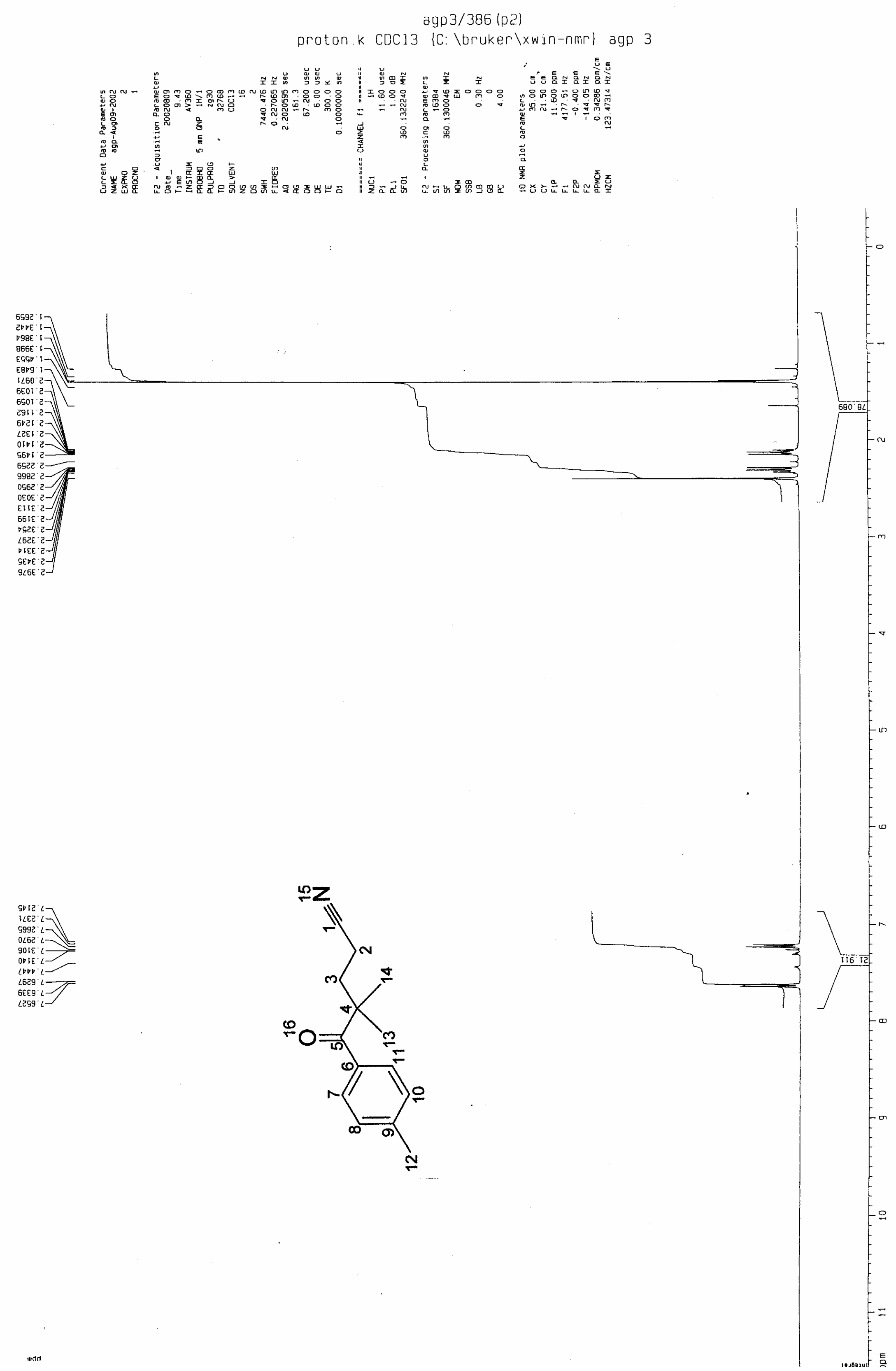
[6] A. Matsuo, N. Nakayama, M. Nakayama, *Phytochemistry*, 1985, **24**, 777-781.



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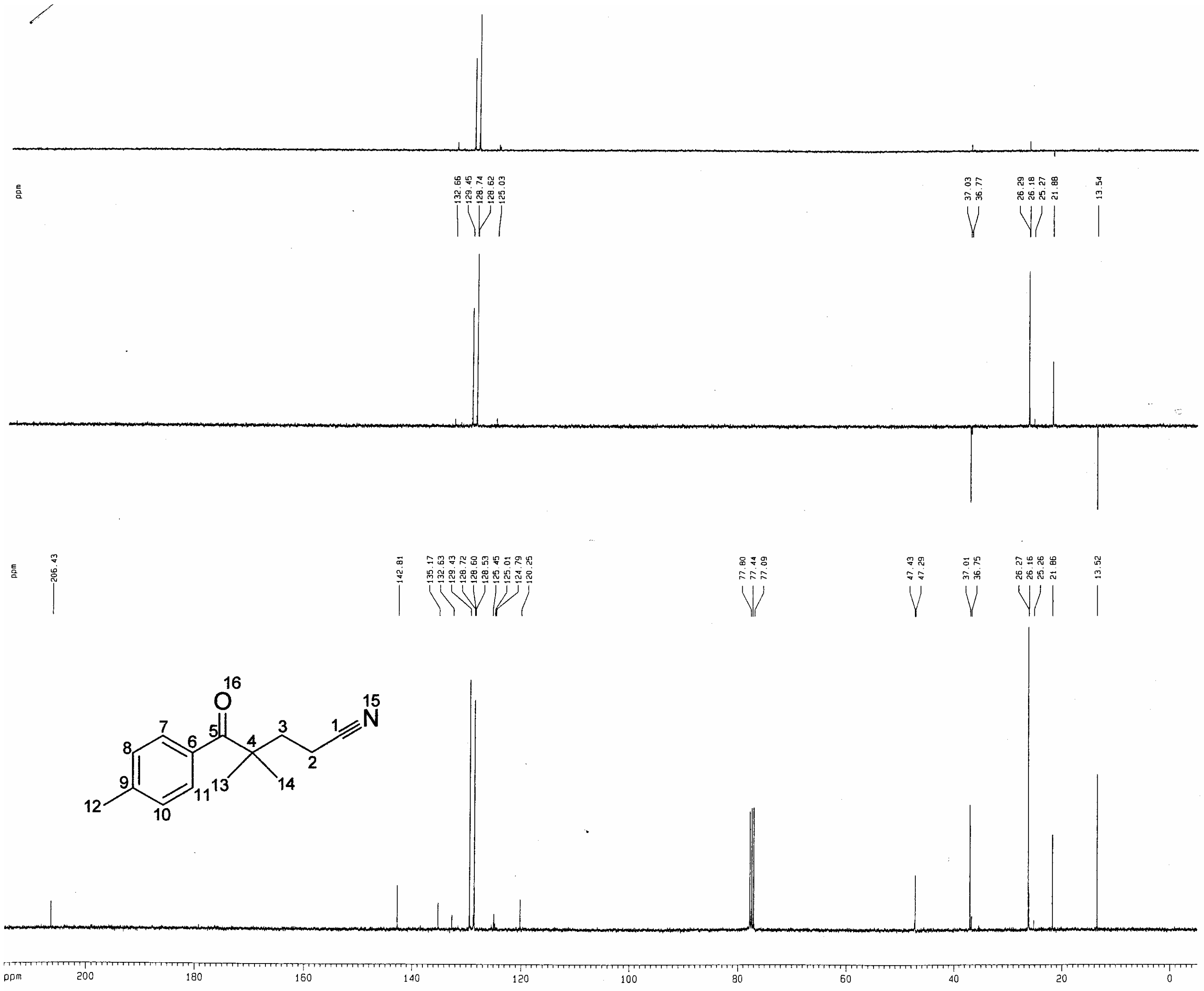

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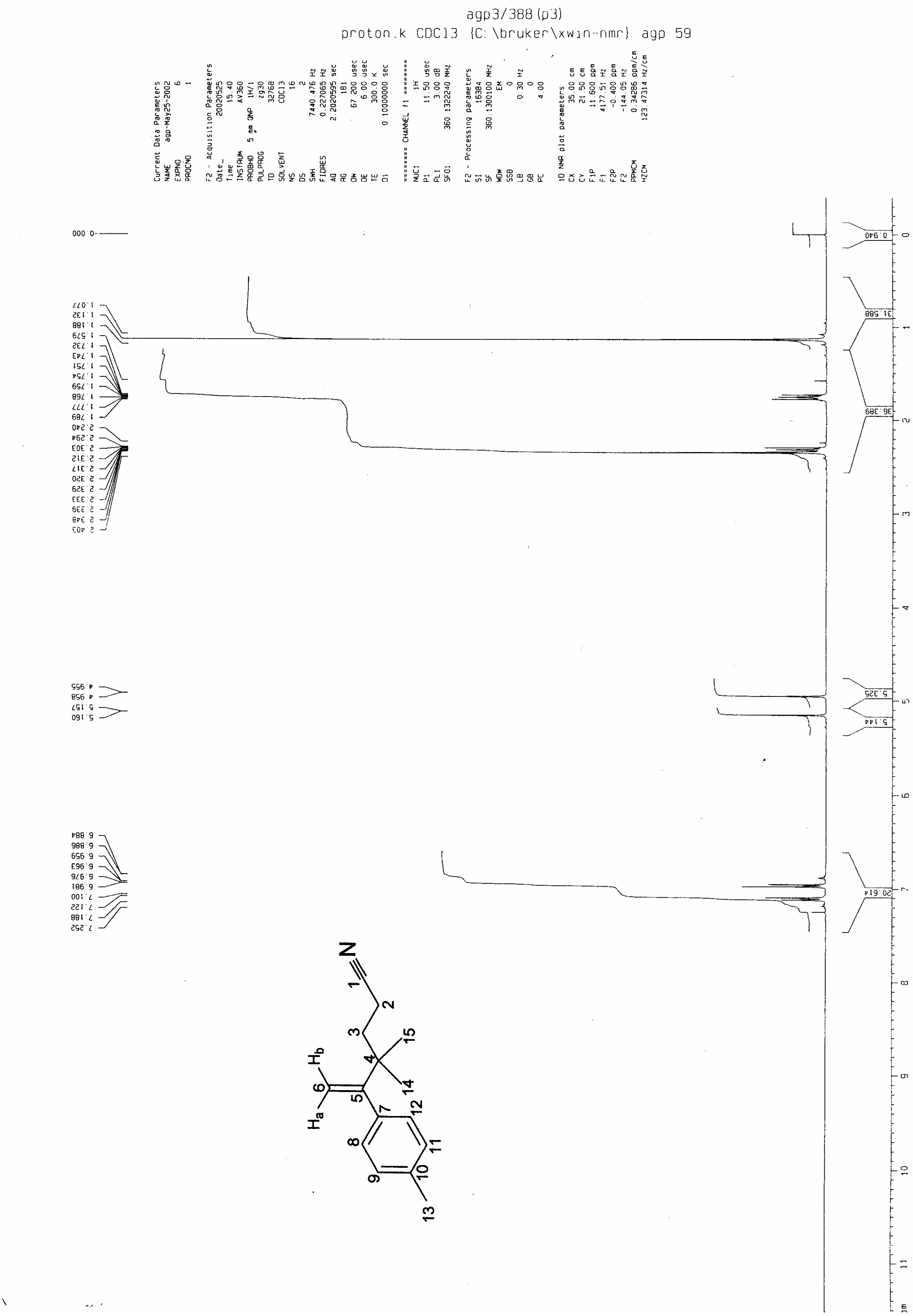
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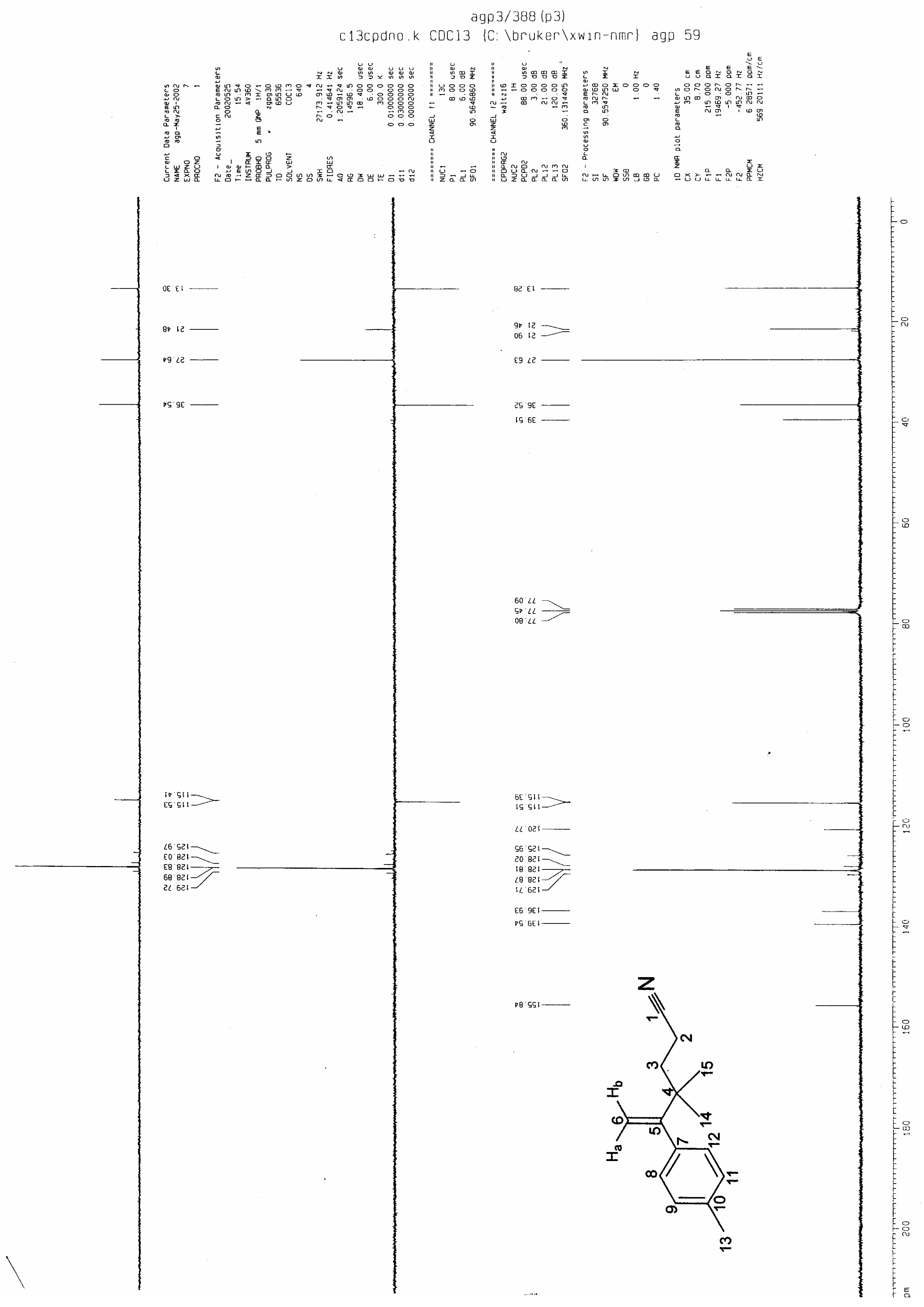
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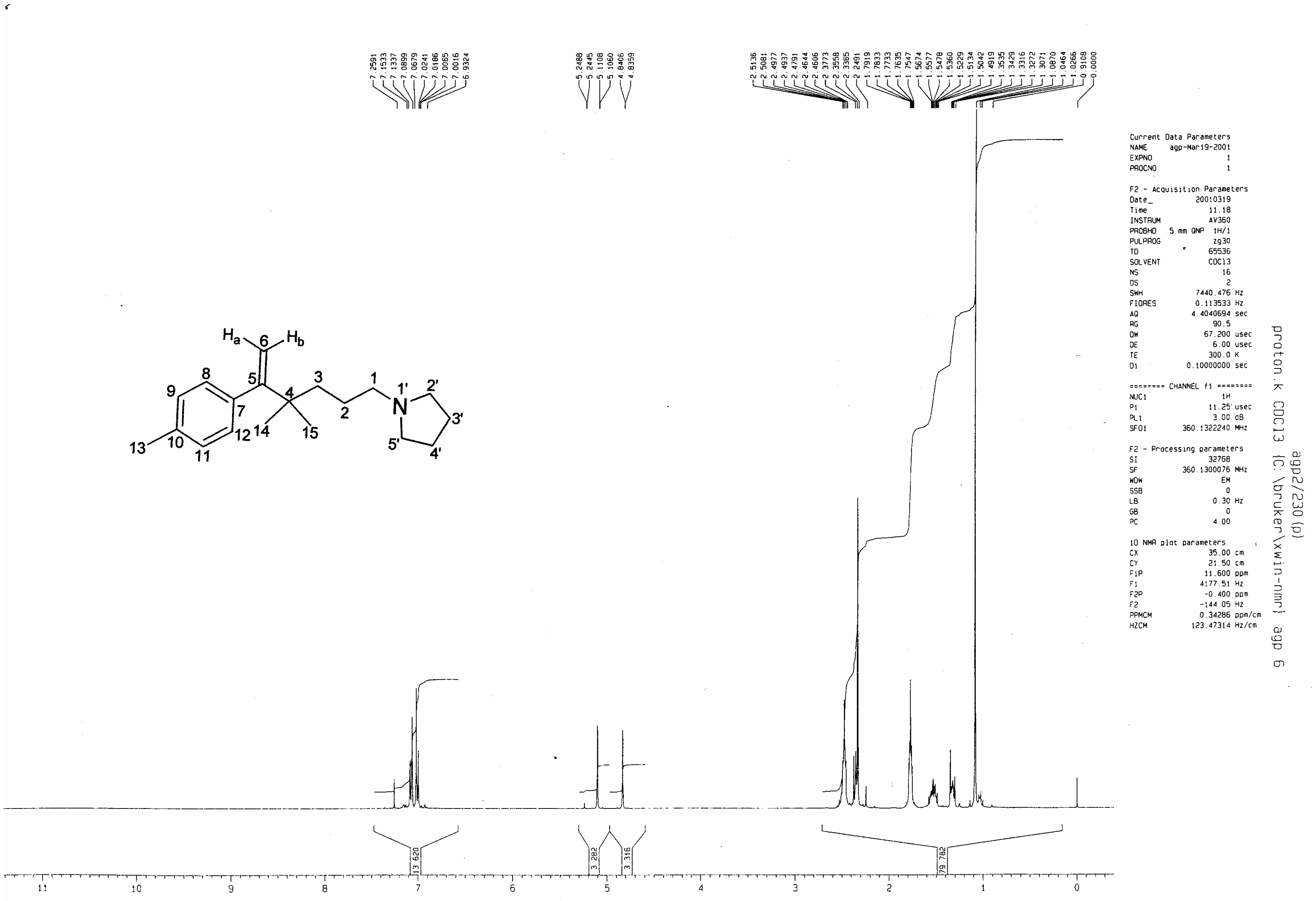
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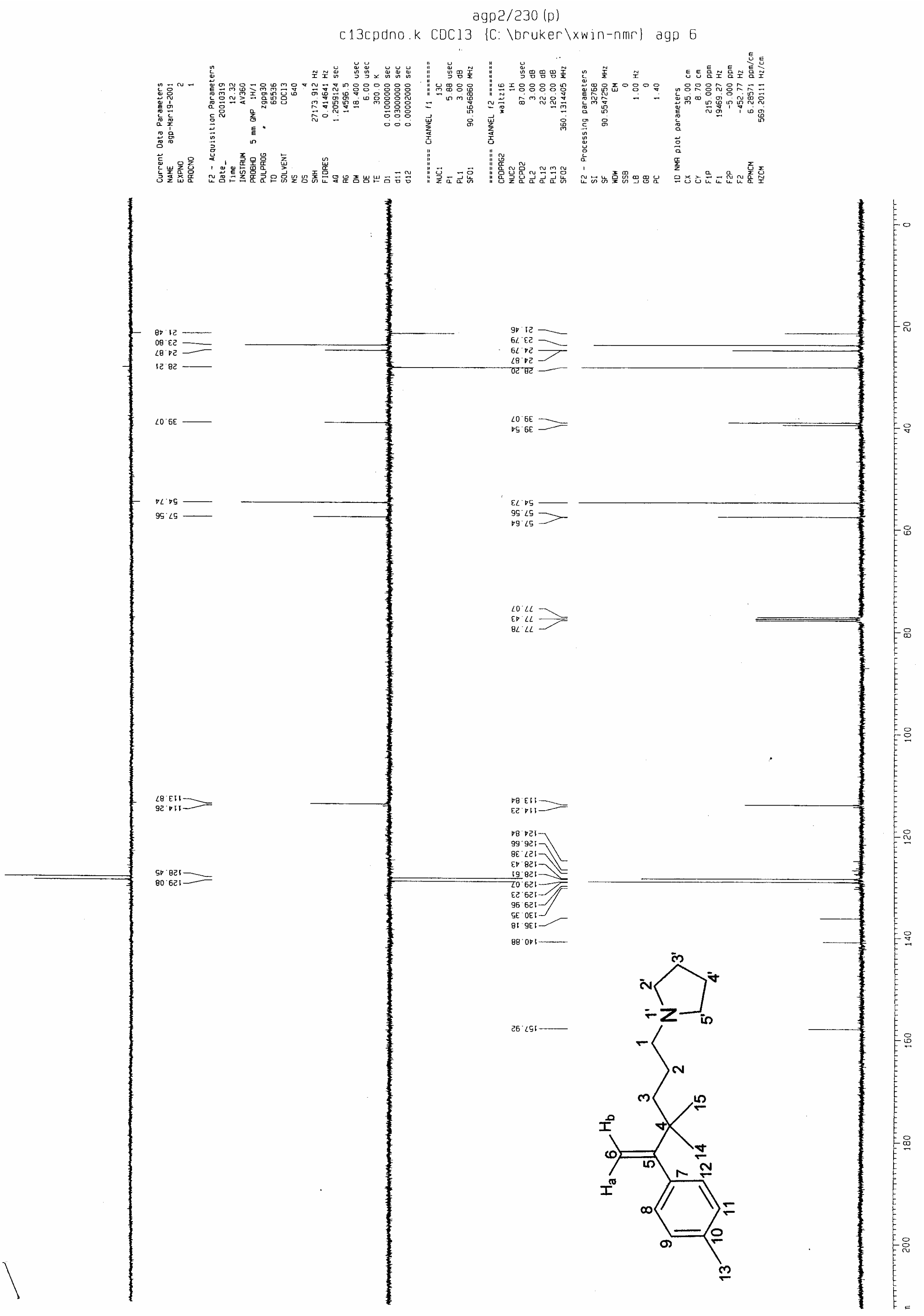
5'


agp2/230 (p)
c13cpdno.k CDCl3 {C:\bruker\xwin-nmr} agp 6
Current Data Parameters
NAME agp-Mar19-2001
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010319
Time 12.32
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 14596.5
DW 18.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.88 usec
PL1 3.00 dB
SFO1 90.5646860 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 87.00 usec
PL2 3.00 dB
PL12 22.00 dB
PL13 120.00 dB
SFO2 360.1314405 MHz
F2 - Processing parameters
SI 32768
SF 90.5547250 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 35.00 cm
CY 8.70 cm
F1P 215.000 ppm
F1 19469.27 Hz
F2P -5.000 ppm
F2 -452.77 Hz
PPMCM 6.28571 ppm/cm
HZCM 569.20111 Hz/cm
21.48
23.80
24.87
28.21
39.07
54.74
57.56
113.87
114.26
128.45
129.08
21.46
23.79
24.79
24.87
28.20
39.07
39.54
54.73
57.56
57.64
77.07
77.43
77.78
113.84
114.23
124.84
126.66
127.38
128.43
128.61
129.07
129.23
129.96
130.35
136.18
140.88
157.92
0
20
40
60
80
100
120
140
160
180
200
H_a
H_b
6
5
7
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9
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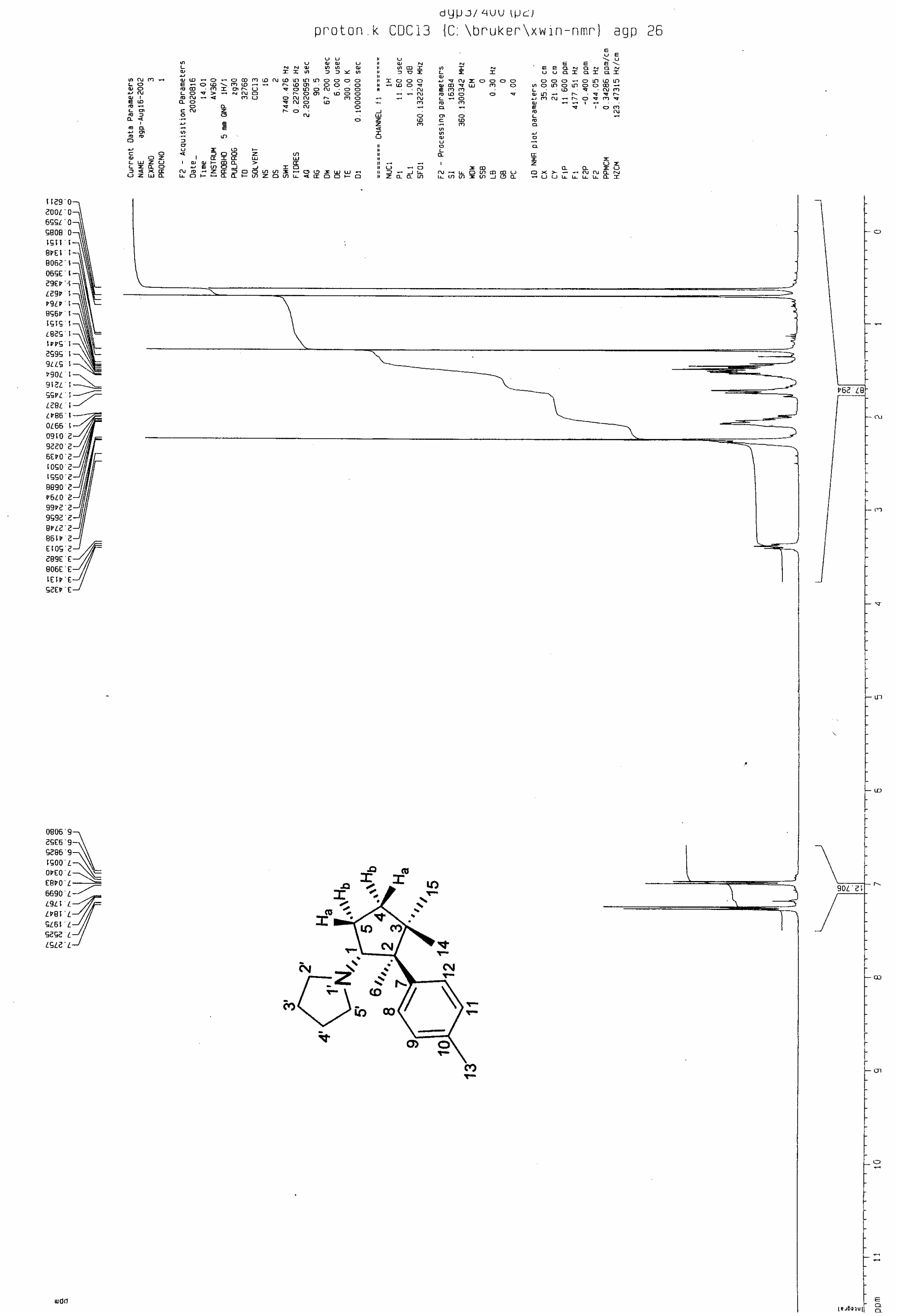
12
13
14
15
4
3
2
1
1'
N
2'
3'
4'
5'


agp3/400 (p2)
proton.k CDCl3 {C:\bruker\xwin-nmr} agp 26
Current Data Parameters
NAME agp-Aug16-2002
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020816
Time 14.01
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 7440.476 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 90.5
DW 67.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 1.00 dB
SFO1 360.1322240 MHz
F2 - Processing parameters
SI 16384
SF 360.1300342 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 35.00 cm
CY 21.50 cm
F1P 11.600 ppm
F1 4177.51 Hz
F2P -0.400 ppm
F2 -144.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 123.47315 Hz/cm
0.6211
0.7002
0.7559
0.8085
1.1151
1.1348
1.2908
1.3590
1.4362
1.4627
1.4764
1.4958
1.5151
1.5287
1.5441
1.5652
1.5776
1.7064
1.7216
1.7455
1.7827
1.9847
1.9970
2.0160
2.0226
2.0439
2.0501
2.0551
2.0688
2.0794
2.2466
2.2656
2.2748
2.4198
2.5013
3.3682
3.3908
3.4131
3.4325
6.9080
6.9352
6.9825
7.0051
7.0340
7.0483
7.0699
7.1767
7.1847
7.1975
7.2525
7.2757
ppm
87.294
12.706
Integral
0
1
2
3
4
5
6
7
8
9
10
11
ppm
Ha
Hb
Hb
Ha
1
2
3
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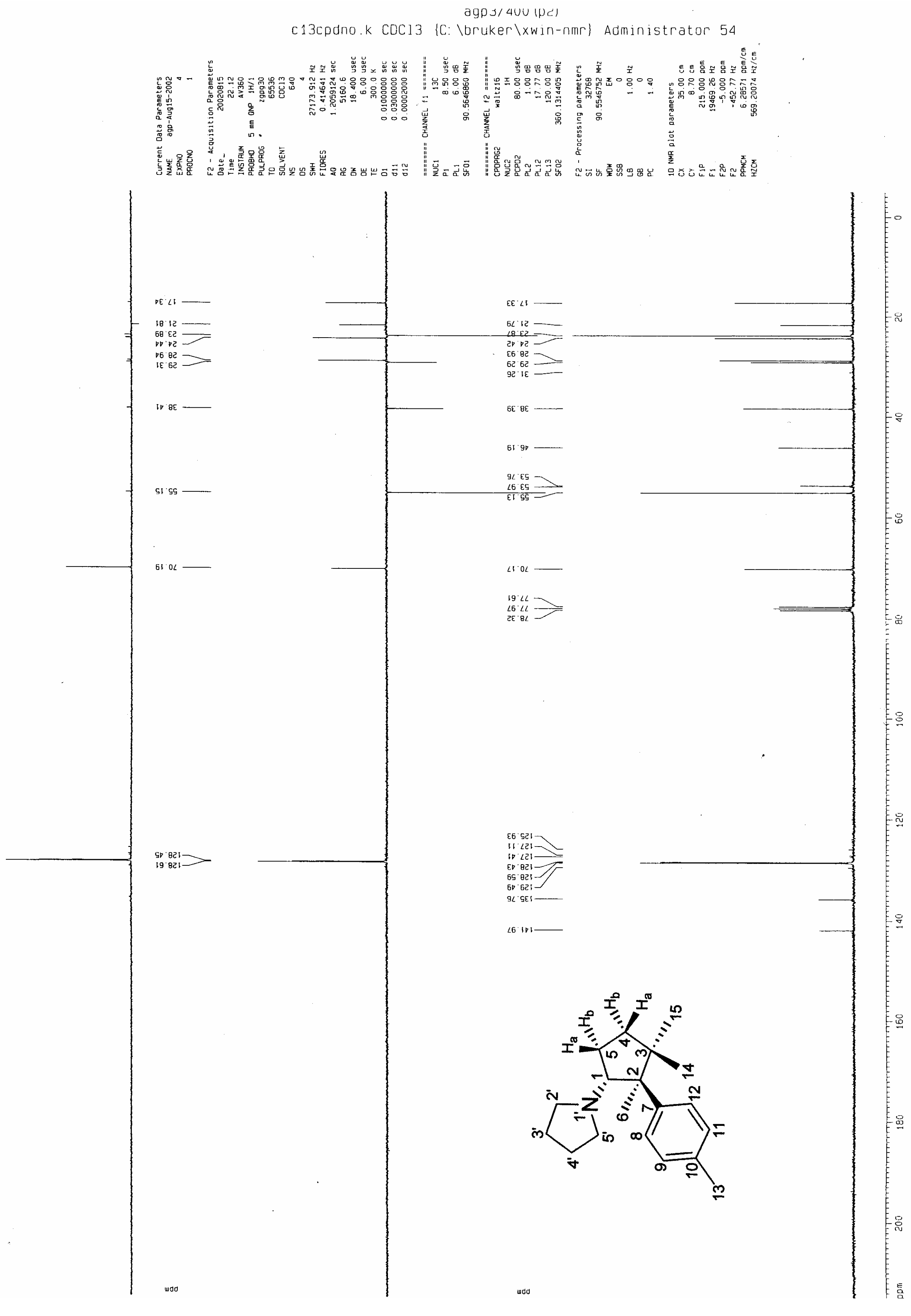
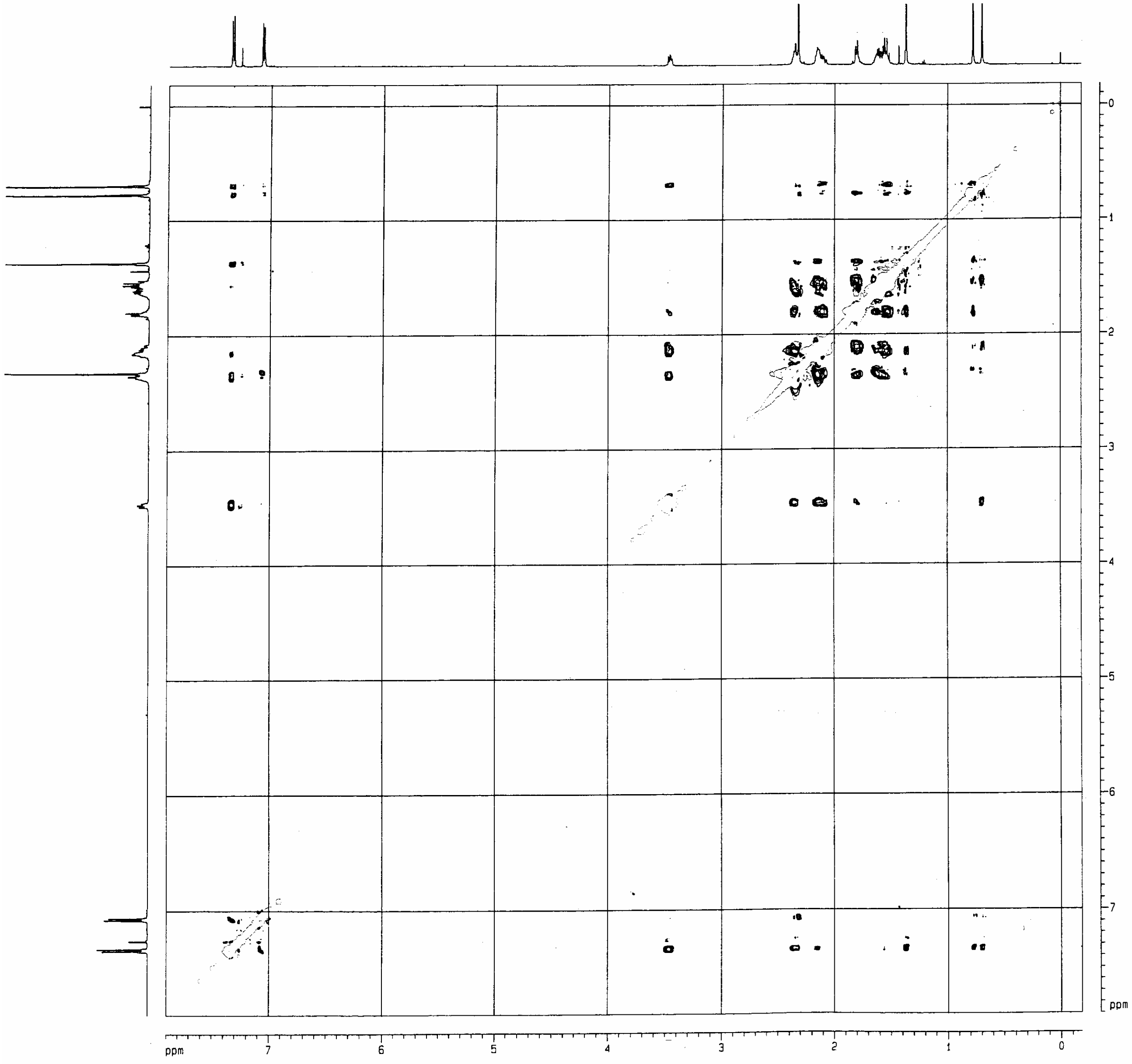
9
10
11
12
13
14
15
N
1'
2'
3'
4'
5'

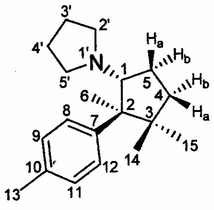


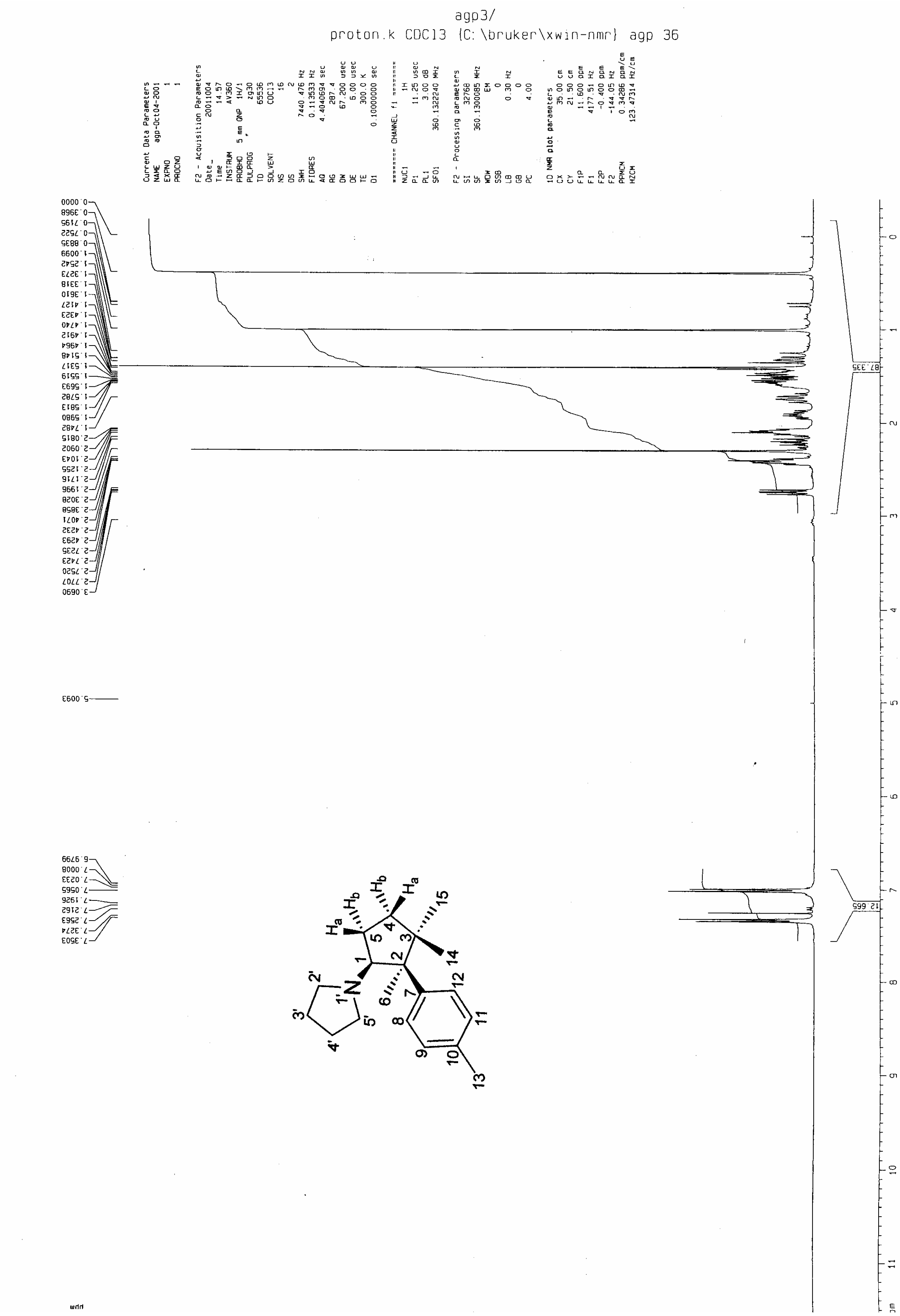
agp3/400 (p2)
c13cpdno.k CDCl3 {C:\bruker\xwin-nmr} Administrator 54

```
Current Data Parameters
NAME      agp-Aug15-2002
EXPNO              4
PROCNO             1

F2 - Acquisition Parameters
Date_           20020815
Time               22.12
INSTRUM            AV360
PROBHD   5 mm QNP 1H/1
PULPROG  *        zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   640
DS                     4
SWH          27173.912 Hz
FIDRES        0.414641 Hz
AQ           1.2059124 sec
RG                5160.6
DW              18.400 usec
DE                6.00 usec
TE               300.0 K
D1          0.01000000 sec
d11         0.03000000 sec
d12         0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 6.00 dB
SFO1         90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.77 dB
PL13              120.00 dB
SFO2        360.1314405 MHz

F2 - Processing parameters
SI                 32768
SF            90.5546752 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 35.00 cm
CY                  8.70 cm
F1P              215.000 ppm
F1              19469.26 Hz
F2P               -5.000 ppm
F2               -452.77 Hz
PPMCM            6.28571 ppm/cm
HZCM           569.20074 Hz/cm
```



```
AGP27
noesytpsw_a3 CDCl3 C:/u jeh 2

        Current Data Parameters
NAME        Jul2501-5.002
EXPNO                  11
PROCNO                  1

      F2 - Acquisition Parameters
Date_            20010725
Time                20.02
INSTRUM            drx500
PROBHD     5 mm BBO BB-
PULPROG           noesytp
TD                   2048
SOLVENT             CDCl3
NS                     16
DS                      4
SWH              4045.307 Hz
FIDRES           1.975248 Hz
AQ              0.2531828 sec
RG                     32
DW                123.600 usec
DE                   6.00 usec
TE                  300.0 K
d0             0.00000300 sec
D1             1.44798100 sec
D8             0.80000001 sec
IN0            0.00012360 sec

============ CHANNEL f1 ============
NUC1                   1H
P1                  11.75 usec
PL1                  0.00 dB
SFO1          500.1319428 MHz

      F1 - Acquisition parameters
ND0                     2
TD                    256
SFO1             500.1319 MHz
FIDRES          15.801982 Hz
SW                  8.088 ppm

      F2 - Processing parameters
SI                   1024
SF            500.1300137 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.00

      F1 - Processing parameters
SI                   1024
MC2                  TPPI
SF            500.1300137 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0

        2D NMR plot parameters
CX2                 25.00 cm
CX1                 25.00 cm
F2PLO               7.901 ppm
F2LO              3951.76 Hz
F2PHI              -0.187 ppm
F2HI               -93.55 Hz
F1PLO               7.901 ppm
F1LO              3951.76 Hz
F1PHI              -0.187 ppm
F1HI               -93.55 Hz
F2PPMCM           0.32354 ppm/cm
F2HZCM          161.81230 Hz/cm
F1PPMCM           0.32354 ppm/cm
F1HZCM          161.81230 Hz/cm
```




agp3/
proton.k CDCl3 {C:\bruker\xwin-nmr} agp 36
Current Data Parameters
NAME agp-Oct04-2001
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20011004
Time 14.57
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7440.476 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 287.4
DW 67.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 3.00 dB
SFO1 360.1322240 MHz
F2 - Processing parameters
SI 32768
SF 360.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 35.00 cm
CY 21.50 cm
F1P 11.600 ppm
F1 4177.51 Hz
F2P -0.400 ppm
F2 -144.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 123.47314 Hz/cm
0.0000
0.3968
0.7195
0.7522
0.8835
1.0099
1.2542
1.3273
1.3318
1.3610
1.4127
1.4323
1.4740
1.4912
1.4964
1.5148
1.5317
1.5519
1.5693
1.5782
1.5813
1.5980
1.7482
2.0815
2.0902
2.1043
2.1255
2.1716
2.1996
2.3028
2.3858
2.4071
2.4232
2.4293
2.7235
2.7423
2.7520
2.7707
3.0690
5.0093
6.9799
7.0008
7.0233
7.0565
7.1926
7.2162
7.2563
7.3274
7.3503
87.335
12.665
0
1
2
3
4
5
6
7
8
9
10
11
ppm
Ha
Hb
Hb
Ha
5
4
1
2
3
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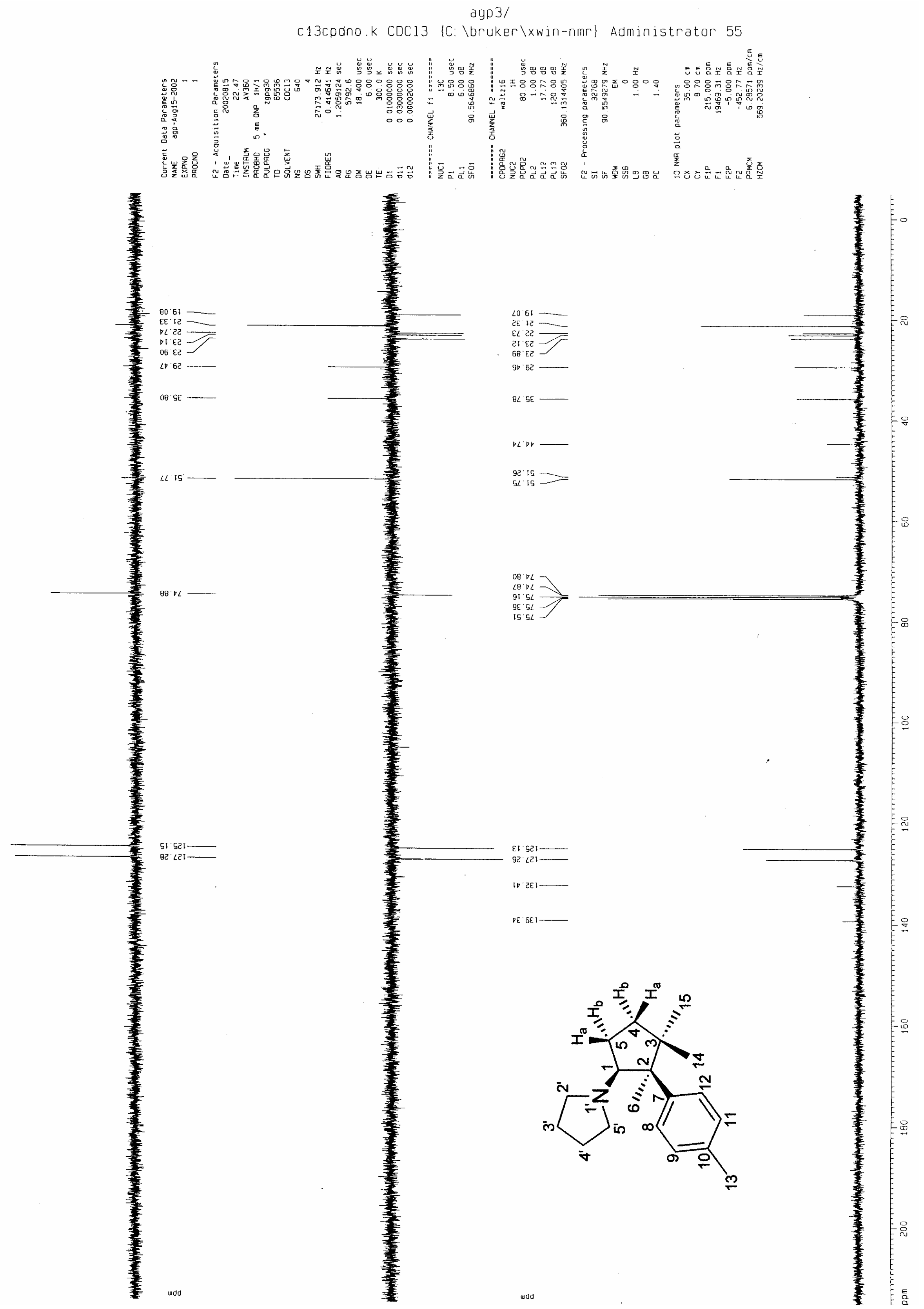
8
9
10
11
12
13


agp3/
c13cpdno.k CDCl3 {C:\bruker\xwin-nmr} Administrator 55
Current Data Parameters
NAME agp-Aug15-2002
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020815
Time 22.47
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 5792.6
DW 18.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.01000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 6.00 dB
SFO1 90.5546860 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 17.77 dB
PL13 120.00 dB
SFO2 360.1314405 MHz
F2 - Processing parameters
SI 32768
SF 90.5549279 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 35.00 cm
CY 8.70 cm
F1P 215.000 ppm
F1 19469.31 Hz
F2P -5.000 ppm
F2 -452.77 Hz
PPMCM 6.28571 ppm/cm
HZCM 569.20239 Hz/cm
19.08
21.33
22.74
23.14
23.90
29.47
35.80
51.77
74.88
125.15
127.28
ppm
19.07
21.32
22.73
23.12
23.89
29.46
35.78
44.74
51.26
51.75
74.80
74.87
75.16
75.36
75.51
125.13
127.26
132.41
139.34
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm
Ha
Hb
Hb
Ha
15
14
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3
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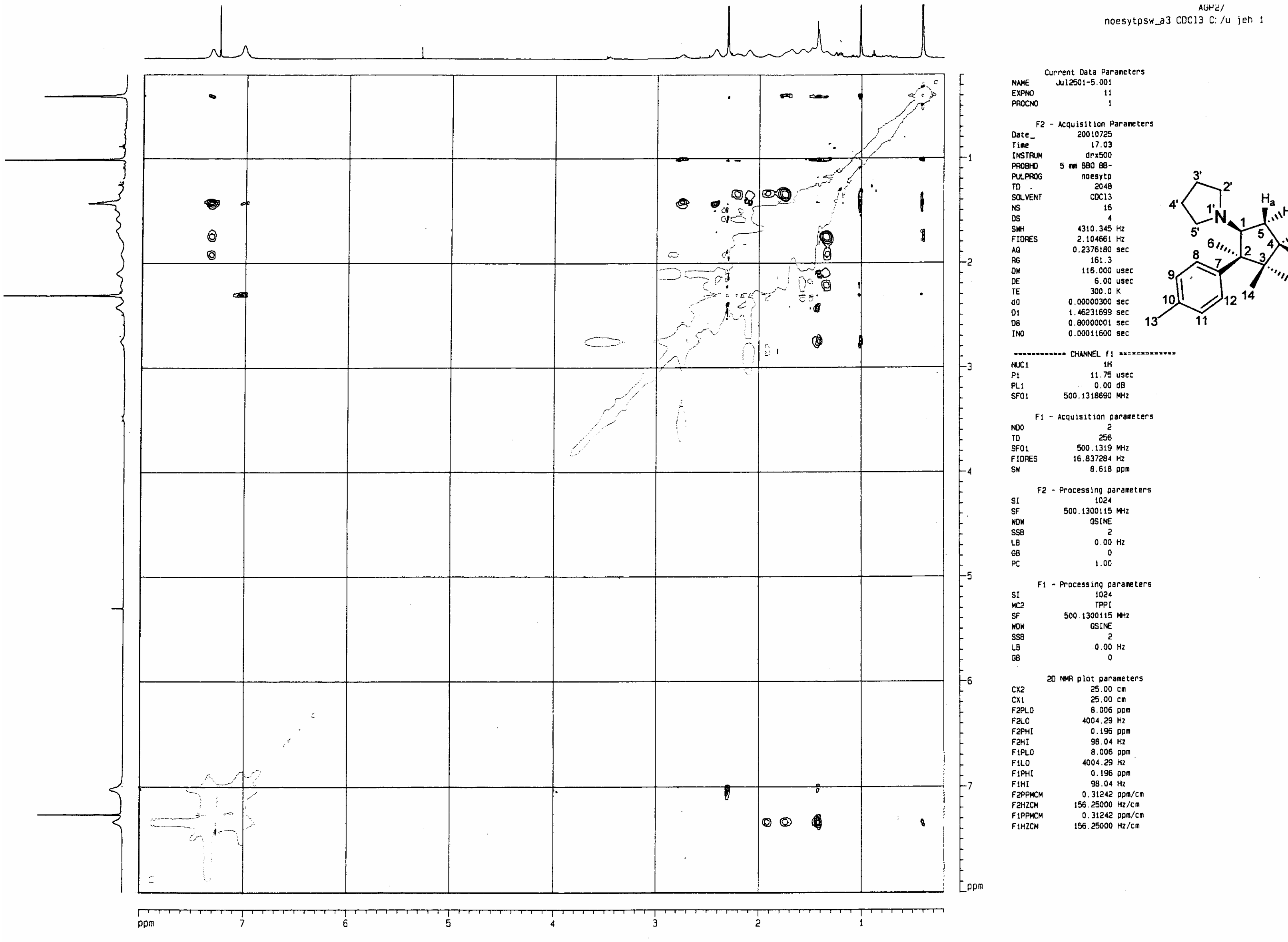
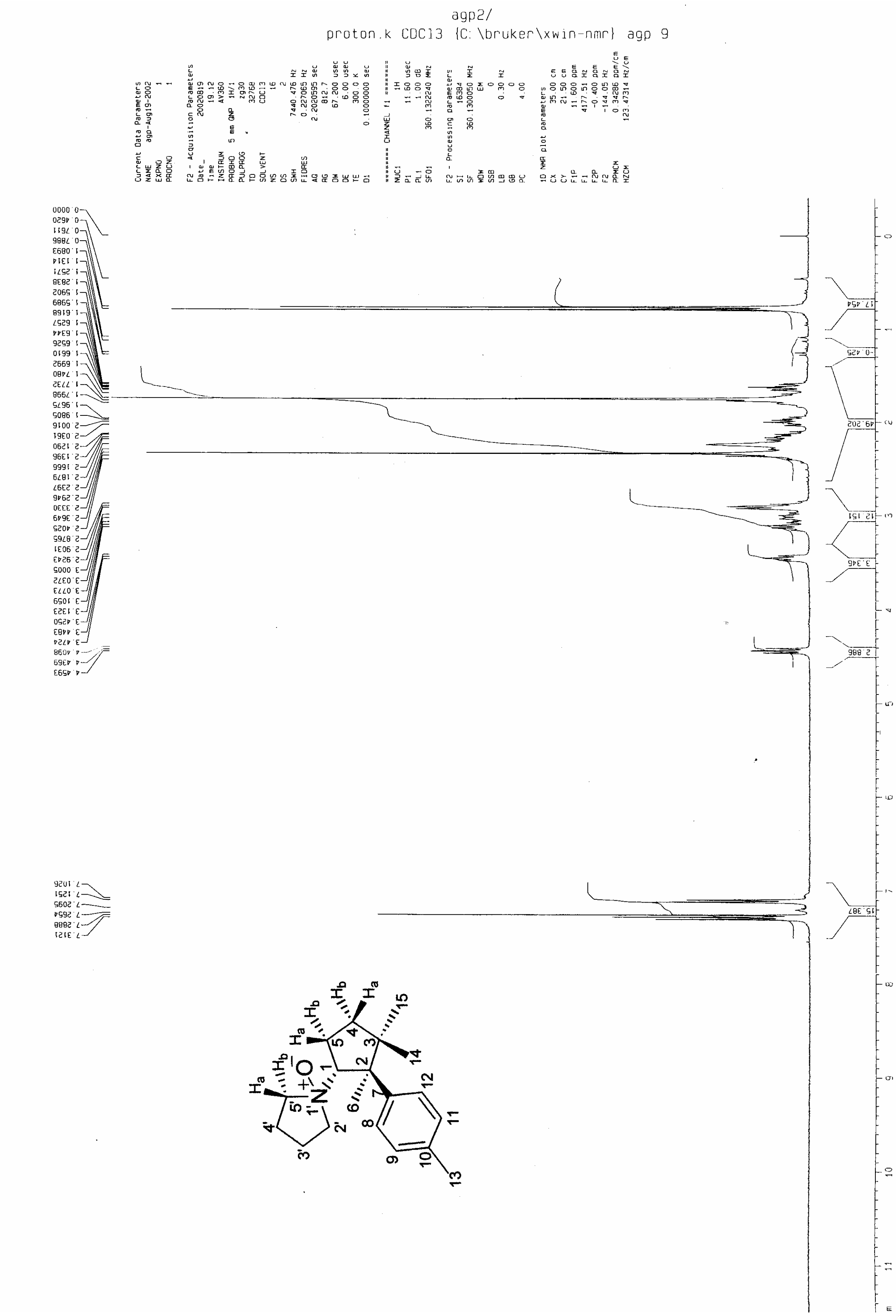
7
8
9
10
11
12
13
N
1'
2'
3'
4'
5'



```
                                        AGP2/
                          noesytpsw_a3 CDC13 C:/u jeh 1

      Current Data Parameters
NAME       Jul2501-5.001
EXPNO                 11
PROCNO                 1

     F2 - Acquisition Parameters
Date_           20010725
Time               17.03
INSTRUM           drx500
PROBHD     5 mm BBO BB-
PULPROG          noesytp
TD                  2048
SOLVENT            CDC13
NS                    16
DS                     4
SWH             4310.345 Hz
FIDRES          2.104661 Hz
AQ             0.2376180 sec
RG                 161.3
DW               116.000 usec
DE                  6.00 usec
TE                 300.0 K
d0            0.00000300 sec
D1            1.46231699 sec
D8            0.80000001 sec
IN0           0.00011600 sec

============ CHANNEL f1 ============
NUC1                  1H
P1                 11.75 usec
PL1                 0.00 dB
SF01         500.1318690 MHz

     F1 - Acquisition parameters
ND0                    2
TD                   256
SF01            500.1319 MHz
FIDRES         16.837284 Hz
SW                 8.618 ppm

     F2 - Processing parameters
SI                  1024
SF           500.1300115 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00

     F1 - Processing parameters
SI                  1024
MC2                 TPPI
SF           500.1300115 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0

       2D NMR plot parameters
CX2                25.00 cm
CX1                25.00 cm
F2PLO              8.006 ppm
F2LO             4004.29 Hz
F2PHI              0.196 ppm
F2HI               98.04 Hz
F1PLO              8.006 ppm
F1LO             4004.29 Hz
F1PHI              0.196 ppm
F1HI               98.04 Hz
F2PPMCM          0.31242 ppm/cm
F2HZCM         156.25000 Hz/cm
F1PPMCM          0.31242 ppm/cm
F1HZCM         156.25000 Hz/cm
```


agp2/
proton.k CDCl3 {C:\bruker\xwin-nmr} agp 9
Current Data Parameters
NAME agp-Aug19-2002
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020819
Time 19.12
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 7440.476 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 812.7
DW 67.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 1.00 dB
SFO1 360.1322240 MHz
F2 - Processing parameters
SI 16384
SF 360.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 35.00 cm
CY 21.50 cm
F1P 11.600 ppm
F1 4177.51 Hz
F2P -0.400 ppm
F2 -144.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 123.47314 Hz/cm
17.454
-0.425
49.202
12.151
3.346
2.886
15.387
0
1
2
3
4
5
6
7
8
9
10
11
ppm
Ha
Hb
O
N
1'
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4'
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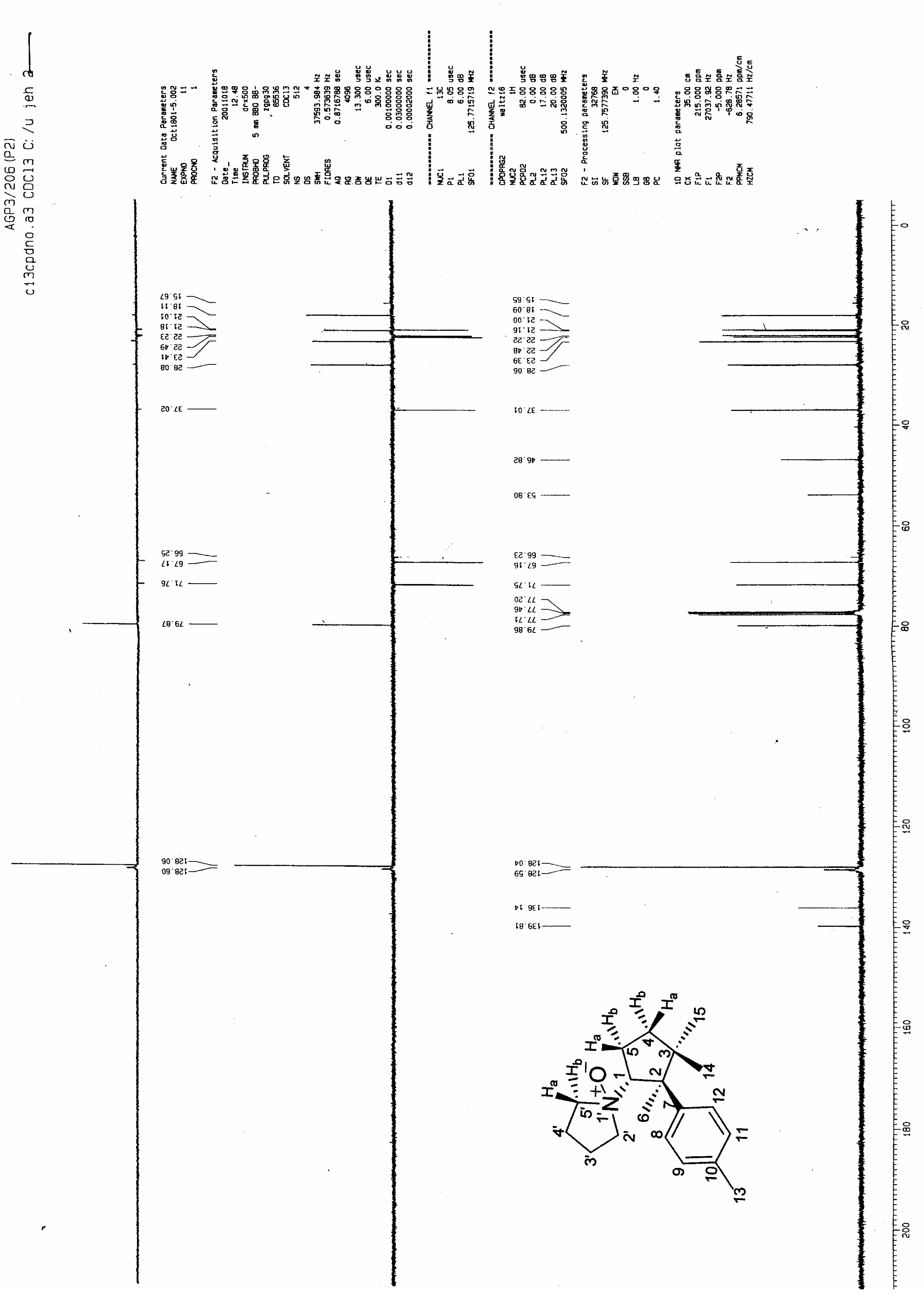
15


AGP3/206 (P2)
c13cpdno.a3 CDCl3 C:/u jeh 2
Current Data Parameters
NAME Oct1801-5.002
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20011018
Time 12.48
INSTRUM drx500
PROBHD 5 mm BBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716788 sec
RG 4096
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.00100000 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 8.05 usec
PL1 6.00 dB
SFO1 125.7715719 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 35.00 cm
F1P 215.000 ppm
F1 27037.92 Hz
F2P -5.000 ppm
F2 -628.78 Hz
PPMCM 6.28571 ppm/cm
HZCM 790.47711 Hz/cm
15.67
18.11
21.01
21.18
22.23
22.49
23.41
28.08
37.02
66.25
67.17
71.76
79.87
128.06
128.60
15.65
18.09
21.00
21.16
22.22
22.48
23.39
28.06
37.01
46.82
53.80
66.23
67.16
71.75
77.20
77.46
77.71
79.86
128.04
128.59
136.14
139.81
0
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40
60
80
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140
160
180
200
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Hb
Ha
Hb
Hb
Ha
O
+
N
1
1'
2'
3'
4'
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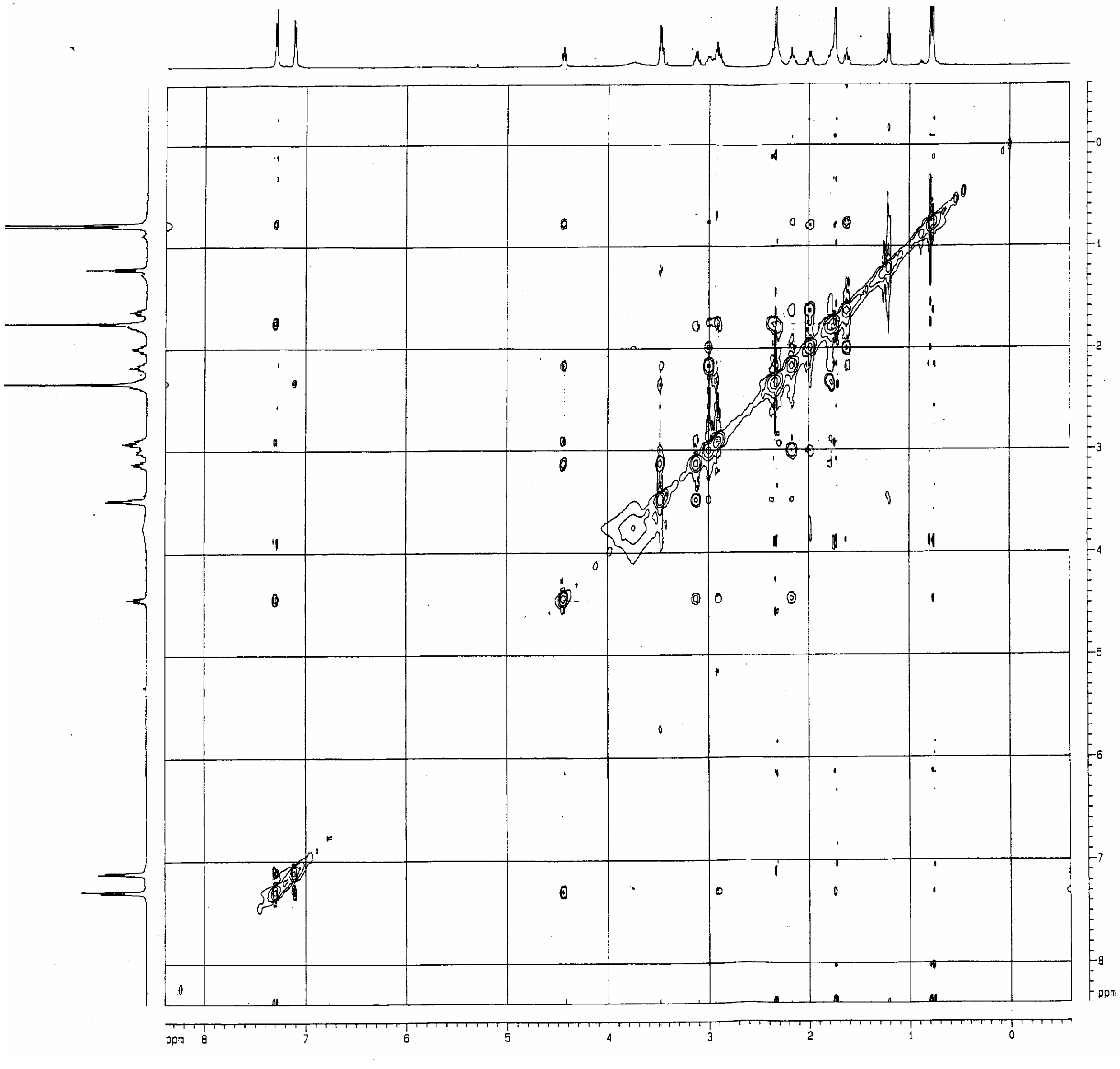
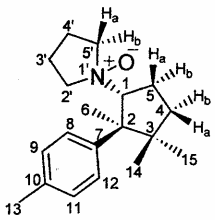
15

AGP3/306 (P2)
noesytpsw_a3 CDCl3 C:/u jeh 2



```
          Current Data Parameters
NAME       Oct1801-5.002
EXPNO                 23
PROCNO                 1

       F2 - Acquisition Parameters
Date_           20011018
Time               15.53
INSTRUM           drx500
PROBHD     5 mm BBO BB-
PULPROG          noesytp
TD                  2048
SOLVENT            CDCl3
NS                     8
DS                     4
SWH             4496.403 Hz
FIDRES          2.195509 Hz
AQ             0.2277876 sec
RG                    32
DW               111.200 usec
DE                  6.00 usec
TE                 300.0 K
d0            0.00000300 sec
D1            0.97255647 sec
D8            0.80000001 sec
IN0           0.00011120 sec

============ CHANNEL f1 =============
NUC1                  1H
P1                 11.75 usec
PL1                 0.00 dB
SFO1         500.1319415 MHz

       F1 - Acquisition parameters
ND0                    2
TD                   256
SFO1            500.1319 MHz
FIDRES         17.564074 Hz
SW                 8.990 ppm

       F2 - Processing parameters
SI                  1024
SF           500.1299980 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  1.00

       F1 - Processing parameters
SI                  1024
MC2                 TPPI
SF           500.1299980 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0

          2D NMR plot parameters
CX2                25.00 cm
CX1                25.00 cm
F2PLO              8.389 ppm
F2LO             4195.76 Hz
F2PHI             -0.601 ppm
F2HI             -300.63 Hz
F1PLO              8.389 ppm
F1LO             4195.76 Hz
F1PHI             -0.601 ppm
F1HI             -300.63 Hz
F2PPMCM          0.35962 ppm/cm
F2HZCM         179.85542 Hz/cm
F1PPMCM          0.35962 ppm/cm
F1HZCM         179.85542 Hz/cm
```



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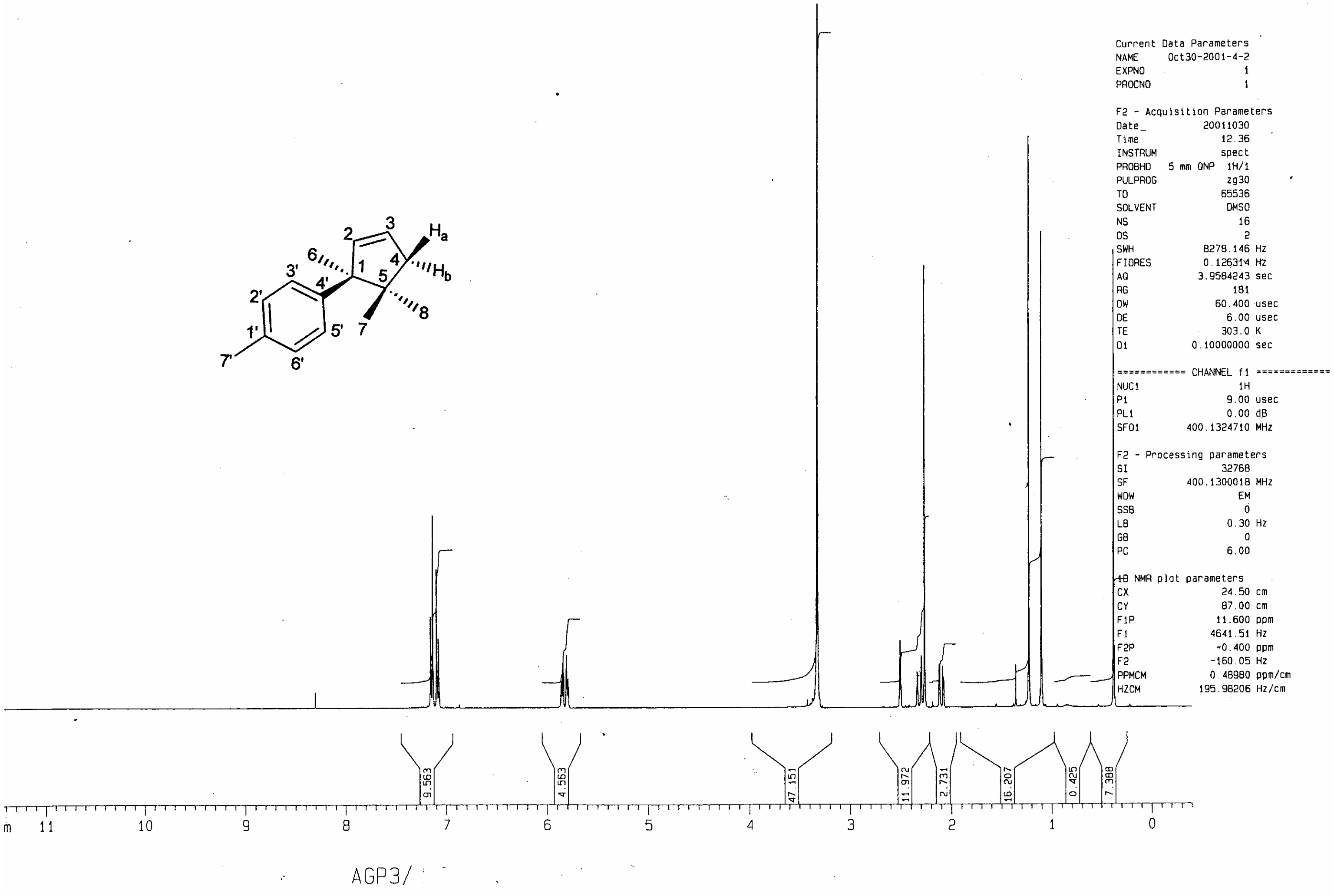


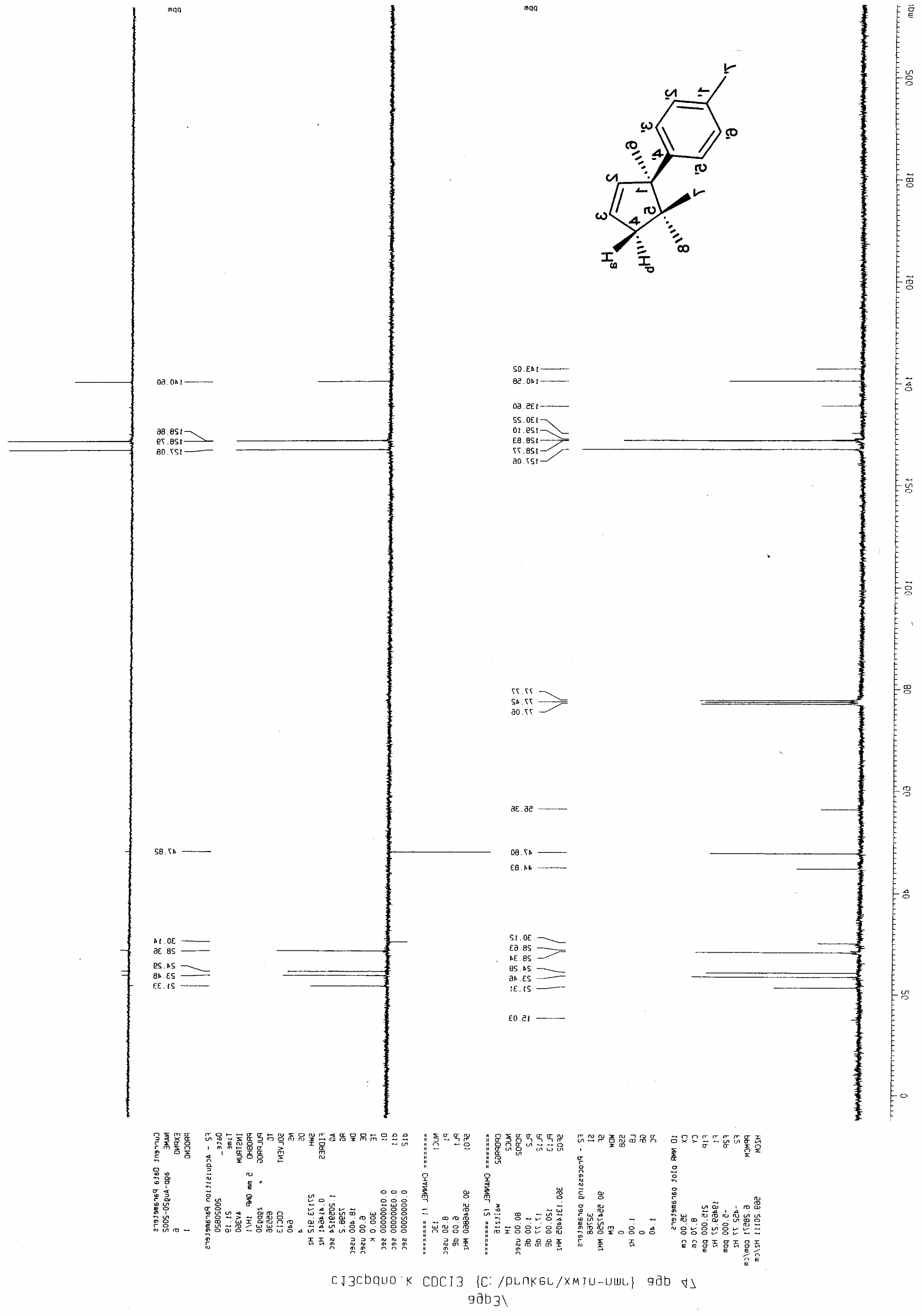
```
Current Data Parameters
NAME      Oct30-2001-4-2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20011030
Time               12.36
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ             3.9584243 sec
RG                   181
DW                60.400 usec
DE                  6.00 usec
TE                 303.0 K
D1            0.10000000 sec

============ CHANNEL f1 ============
NUC1                  1H
P1                  9.00 usec
PL1                 0.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300018 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  6.00

1D NMR plot parameters
CX                 24.50 cm
CY                 87.00 cm
F1P               11.600 ppm
F1               4641.51 Hz
F2P               -0.400 ppm
F2               -160.05 Hz
PPMCM            0.48980 ppm/cm
HZCM           195.98206 Hz/cm
```


ppm
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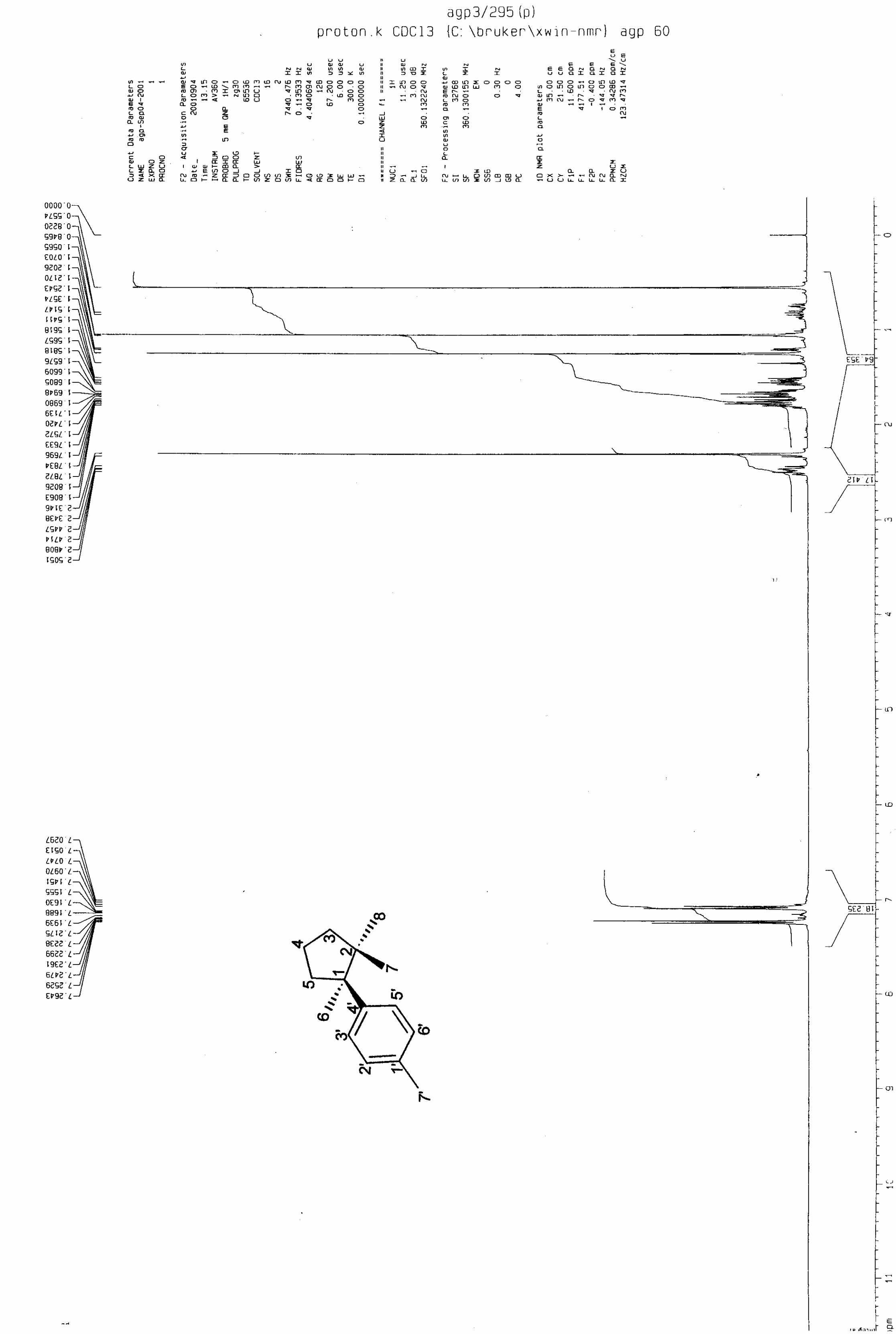
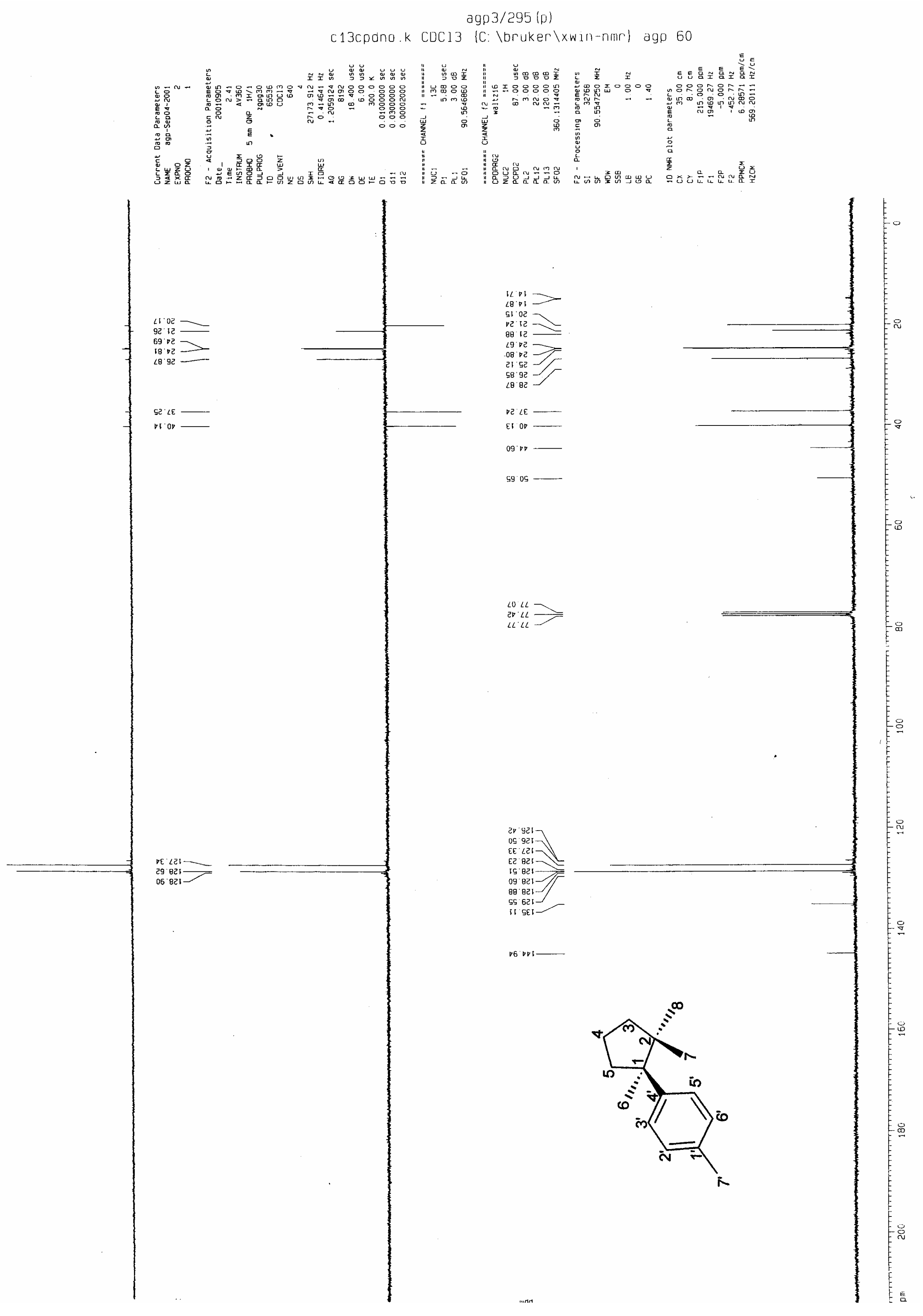
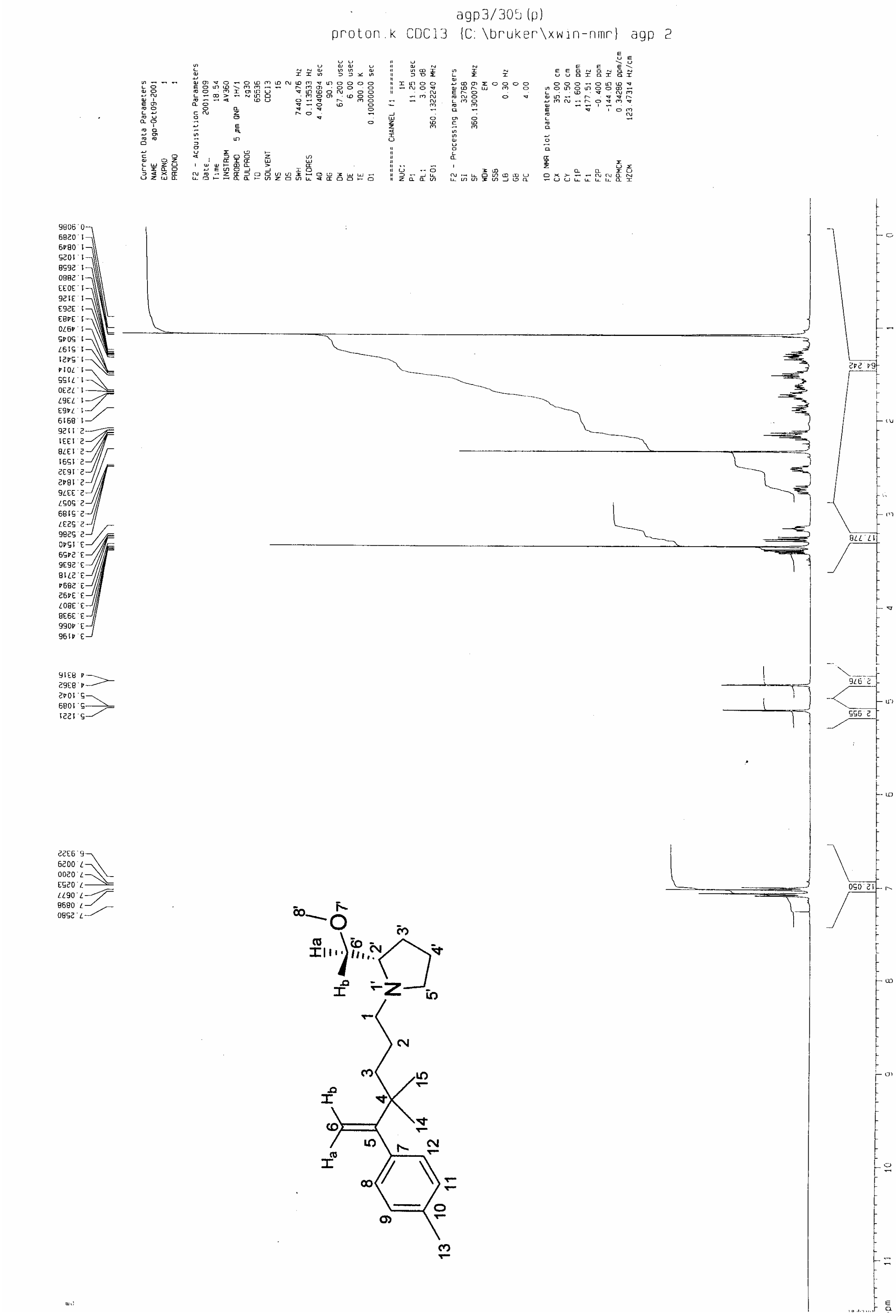
128.86
128.79
127.08
47.82
30.14
28.38
24.29
23.48
21.33
ppm
143.05
140.58
136.60
130.22
129.10
128.83
128.77
127.06
77.77
77.45
77.06
56.36
47.80
44.83
30.15
28.63
28.34
24.28
23.46
21.31
15.03
200
180
160
140
120
100
80
60
40
20
0
ppm
7'
1'
2'
3'
4'
5'
6'
1
2
3
4
5
6
7
8
9
Ha
Hb

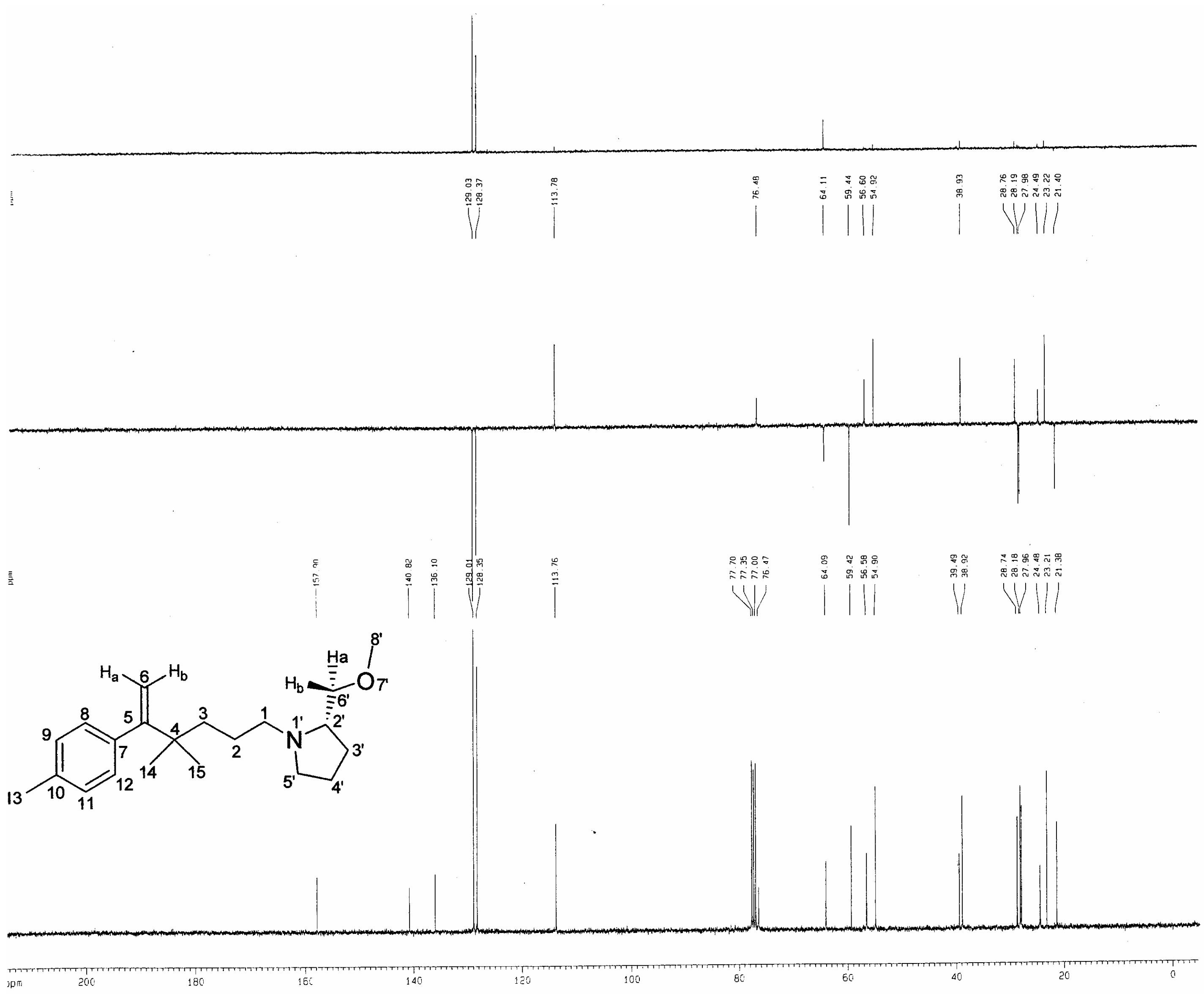

agp3/295 (p)
proton.k CDCl3 {C:\bruker\xwin-nmr} agp 60
Current Data Parameters
NAME agp-Sep04-2001
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010904
Time 13.15
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7440.476 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 128
DW 67.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 3.00 dB
SFO1 360.1322240 MHz
F2 - Processing parameters
SI 32768
SF 360.1300155 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 35.00 cm
CY 21.50 cm
F1P 11.600 ppm
F1 4177.51 Hz
F2P -0.400 ppm
F2 -144.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 123.47314 Hz/cm
64.353
17.412
18.235
ppm
0
1
2
3
4
5
6
7
8
9
10
11
1
2
3
4
5
6
7
8
1'
2'
3'
4'
5'
6'
7'



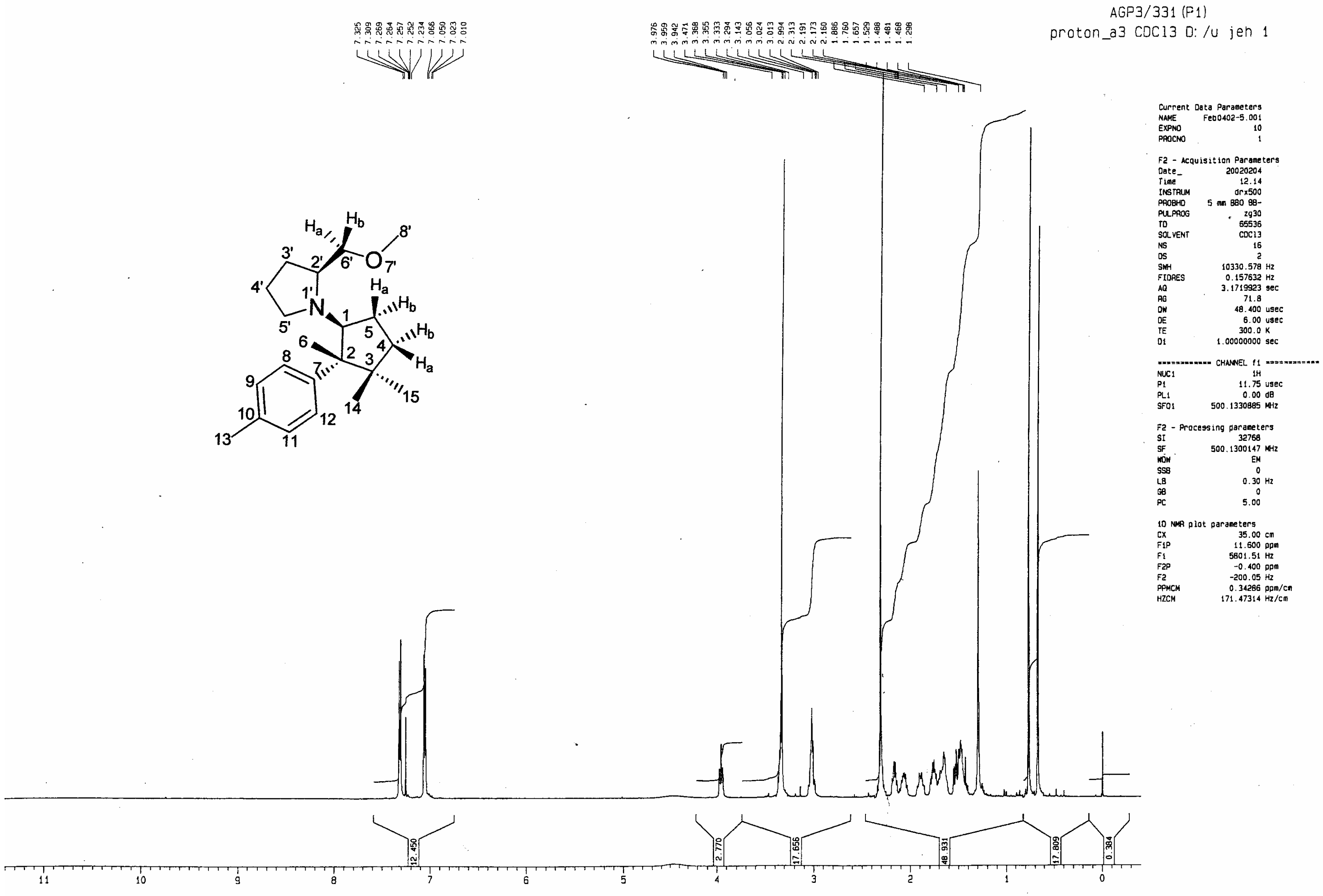
```
agp3/295 (p)
c13cpdno.k CDC13 {C:\bruker\xwin-nmr} agp 60

Current Data Parameters
NAME     agp-Sep04-2001
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20010905
Time               2.41
INSTRUM           AV360
PROBHD   5 mm QNP 1H/1
PULPROG         zgpg30
TD                65536
SOLVENT           CDC13
NS                  640
DS                    4
SWH          27173.912 Hz
FIDRES        0.414641 Hz
AQ           1.2059124 sec
RG                 8192
DW               18.400 usec
DE                 6.00 usec
TE                300.0 K
D1           0.01000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 5.88 usec
PL1                3.00 dB
SFO1         90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             87.00 usec
PL2                3.00 dB
PL12              22.00 dB
PL13             120.00 dB
SFO2        360.1314405 MHz

F2 - Processing parameters
SI                32768
SF           90.5547250 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                35.00 cm
CY                 8.70 cm
F1P             215.000 ppm
F1             19469.27 Hz
F2P              -5.000 ppm
F2              -452.77 Hz
PPMCM           6.28571 ppm/cm
HZCM          569.20111 Hz/cm
```


agp3/305 (p)
proton.k CDCl3 {C:\bruker\xwin-nmr} agp 2
Current Data Parameters
NAME agp-Oct09-2001
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20011009
Time 18.54
INSTRUM AV360
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7440.476 Hz
FIDRES 0.113533 Hz
AQ 4.4040694 sec
RG 90.5
DW 67.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 3.00 dB
SFO1 360.1322240 MHz
F2 - Processing parameters
SI 32768
SF 360.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 35.00 cm
CY 21.50 cm
F1P 11.600 ppm
F1 4177.51 Hz
F2P -0.400 ppm
F2 -144.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 123.47314 Hz/cm
0.9086
1.0289
1.0849
1.1025
1.2658
1.2880
1.3033
1.3126
1.3263
1.3483
1.4970
1.5045
1.5197
1.5421
1.7014
1.7155
1.7230
1.7367
1.7463
1.8919
2.1126
2.1331
2.1378
2.1591
2.1632
2.1842
2.3376
2.5057
2.5189
2.5237
2.5286
3.1540
3.2459
3.2636
3.2718
3.2894
3.3492
3.3807
3.3938
3.4066
3.4196
4.8316
4.8362
5.1042
5.1089
5.1221
6.9322
7.0029
7.0200
7.0253
7.0427
7.0898
7.2580
64.242
17.778
2.976
2.955
12.050
0
1
2
3
4
5
6
7
8
9
10
11
ppm
Ha
Hb
6
5
7
8
9
10
11
12
13
4
14
15
3
2
1
N
1'
2'
3'
4'
5'
6'
O
7'
8'
Ha
Hb

agp3/305 (p)
c13cpdno.k CDC13 {C:\bruker\xwin-nmr} agp 57



```
Current Data Parameters
NAME      agp-Sep05-2002
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20020906
Time                0.36
INSTRUM            AV360
PROBHD   5 mm QNP  1H/1
PULPROG    ,      zgpg30
TD                 65536
SOLVENT            CDC13
NS                   640
DS                     4
SWH            27173.912 Hz
FIDRES          0.414641 Hz
AG             1.2059124 sec
RG                5792.6
DW                18.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            0.01000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.50 usec
PL1                 6.00 dB
SFO1          90.5646860 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               17.77 dB
PL13              120.00 dB
SFO2         360.1314405 MHz

F2 - Processing parameters
SI                 32768
SF            90.5547315 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 35.00 cm
CY                  8.70 cm
F1P              215.000 ppm
F1              19469.27 Hz
F2P               -5.000 ppm
F2               -452.77 Hz
PPMCM            6.28571 ppm/cm
HZCM           569.20111 Hz/cm
```


AGP3/331 (P1)
proton_a3 CDCl3 D:/u jeh 1
Current Data Parameters
NAME Feb0402-5.001
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020204
Time 12.14
INSTRUM drx500
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 71.8
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
============ CHANNEL f1 ============
NUC1 1H
P1 11.75 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 5.00
1D NMR plot parameters
CX 35.00 cm
F1P 11.600 ppm
F1 5801.51 Hz
F2P -0.400 ppm
F2 -200.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 171.47314 Hz/cm
7.325
7.309
7.269
7.264
7.257
7.252
7.234
7.066
7.050
7.023
7.010
3.976
3.959
3.942
3.471
3.368
3.355
3.333
3.294
3.143
3.056
3.024
3.013
2.994
2.313
2.191
2.173
2.160
1.886
1.760
1.657
1.529
1.488
1.481
1.468
1.298
12.450
2.770
17.656
48.931
17.809
0.384
11
10
9
8
7
6
5
4
3
2
1
0
Ha
Hb
8'
O
7'
6'
3'
2'
4'
1'
N
1
5
Ha
Hb
Hb
4
Ha
5'
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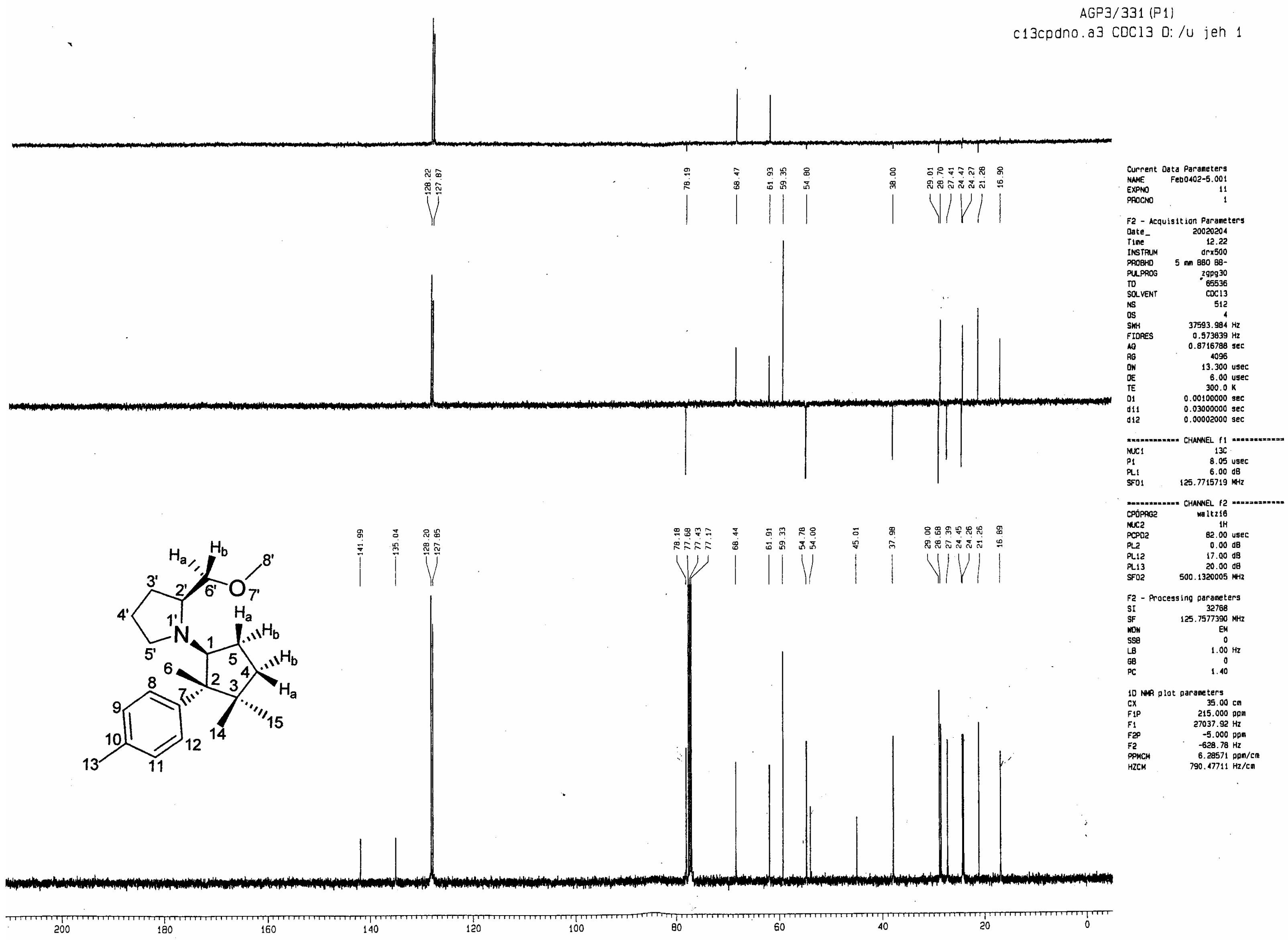
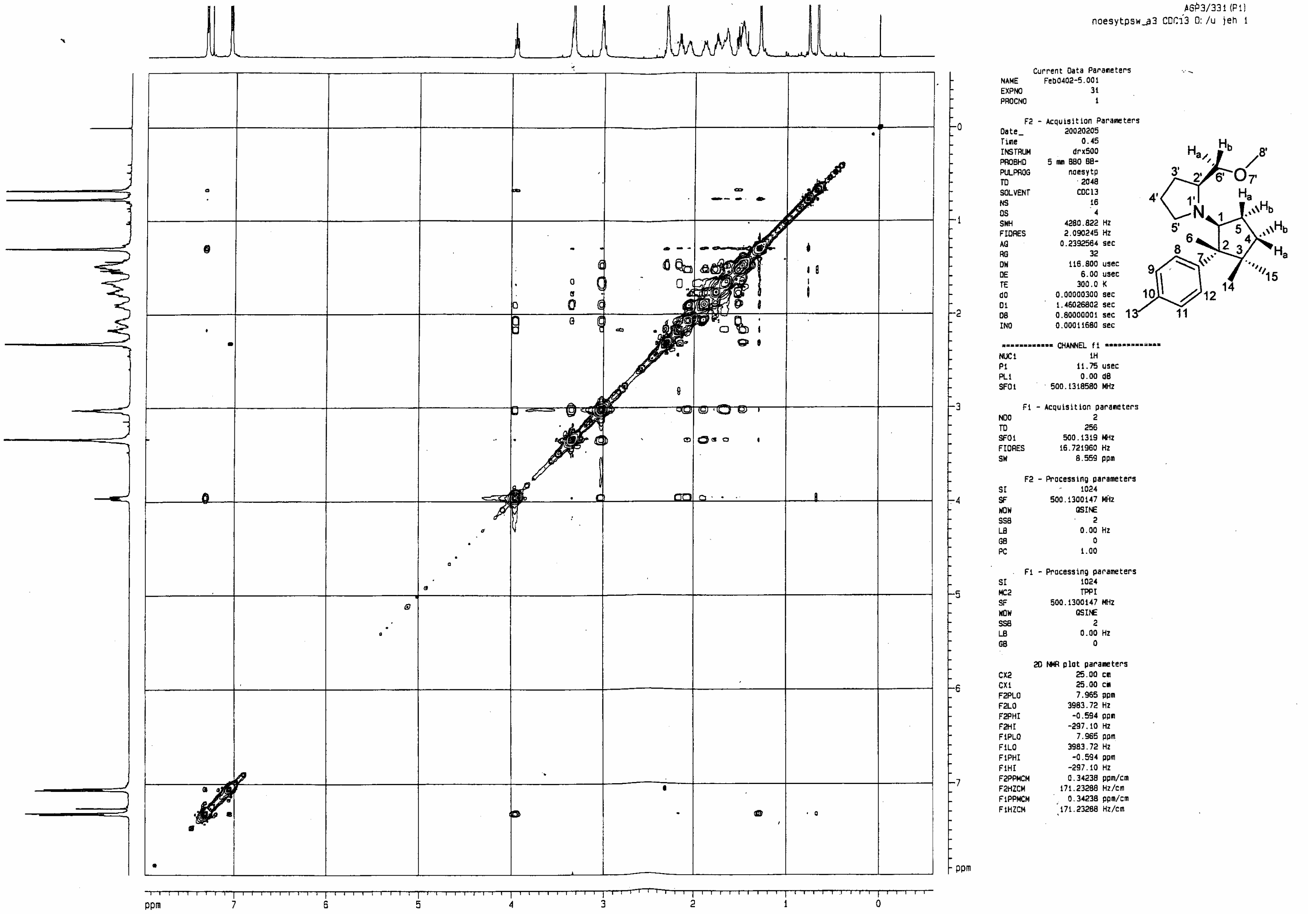
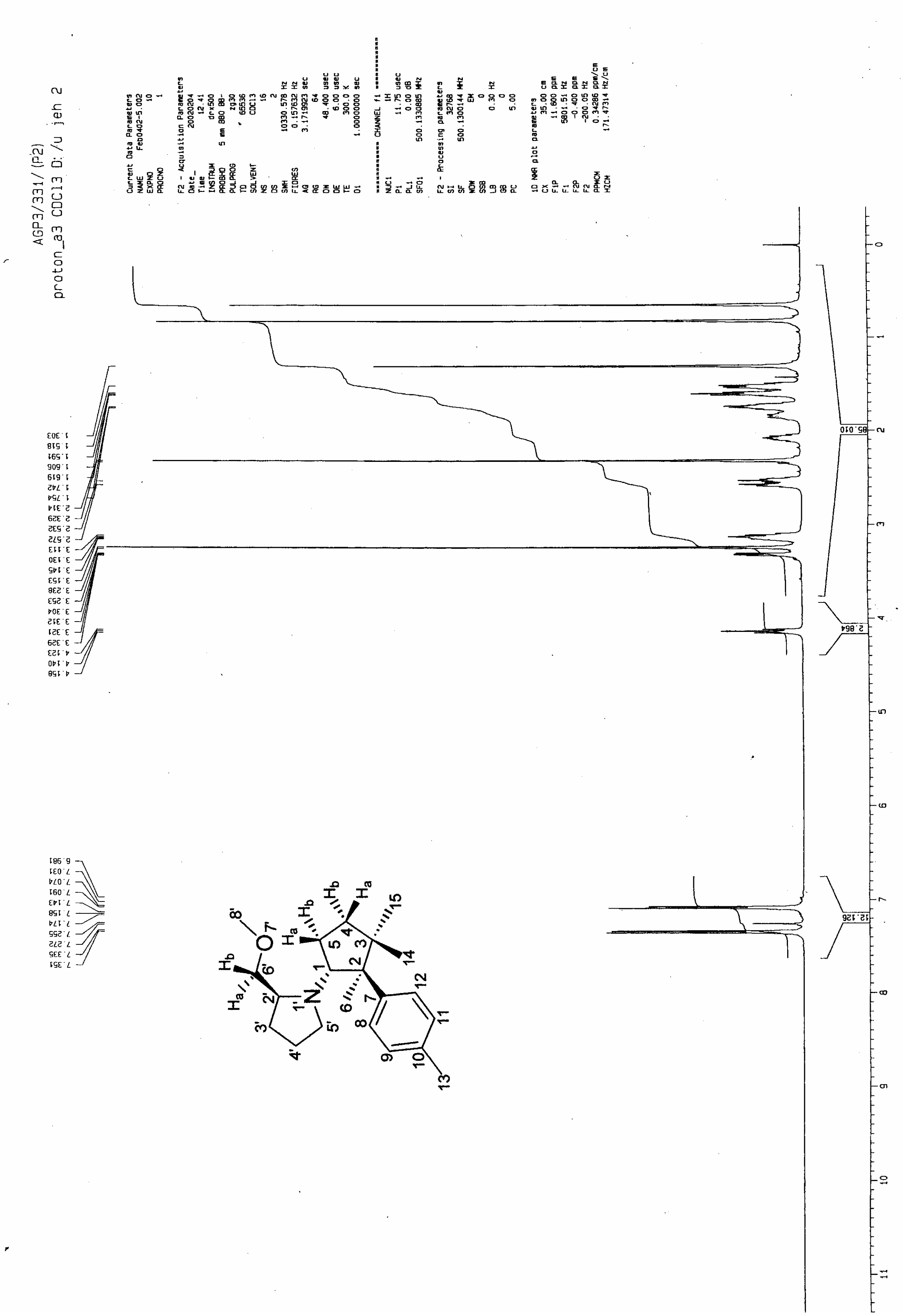
10
12
13
11
14
15



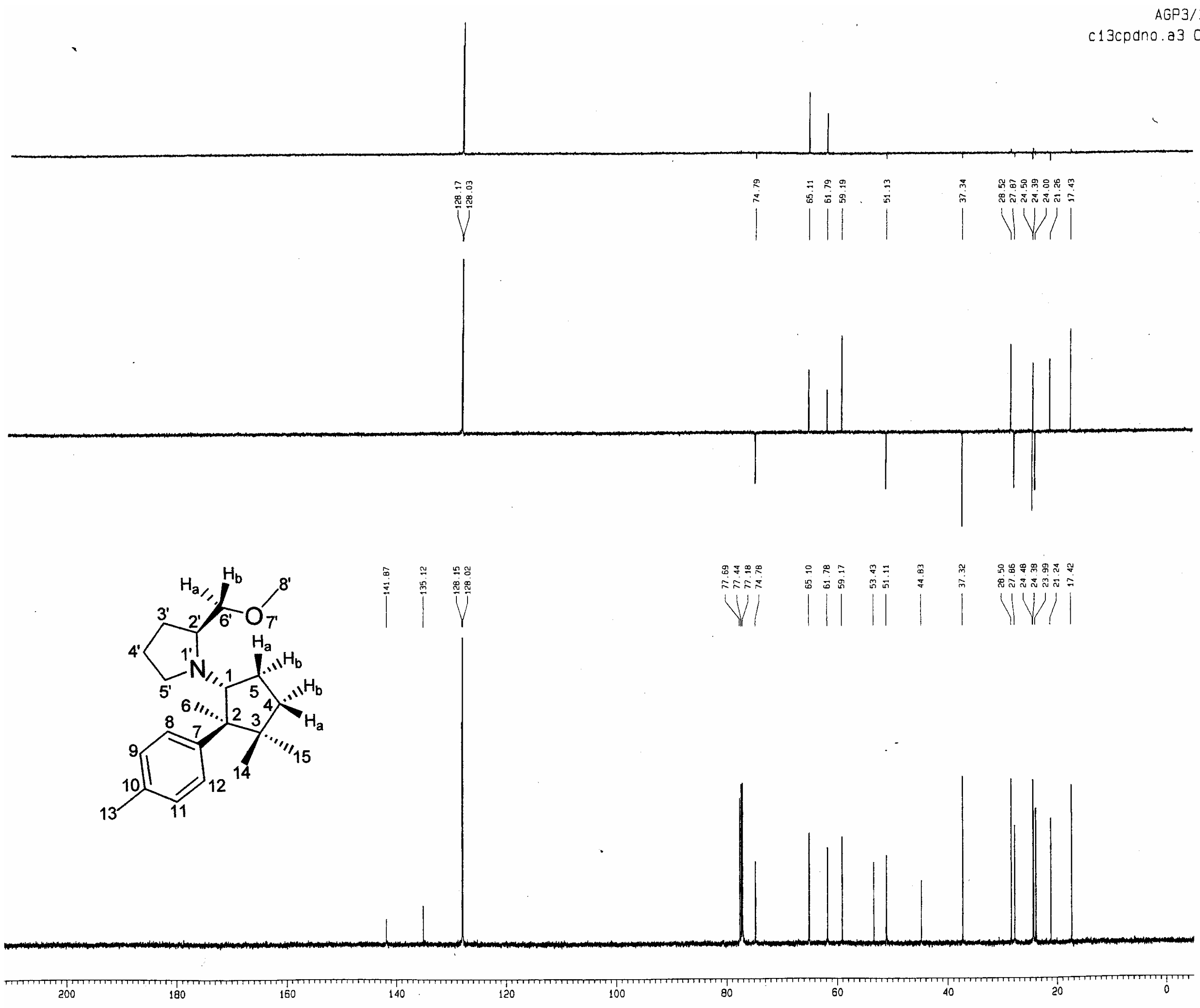
```
Current Data Parameters
NAME       Feb0402-5.001
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20020204
Time               12.22
INSTRUM           drx500
PROBHD      5 mm BBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH           37593.984 Hz
FIDRES         0.573839 Hz
AQ            0.8716788 sec
RG                  4096
DW               13.300 usec
DE                 6.00 usec
TE                300.0 K
D1           0.00100000 sec
d11          0.03000000 sec
d12          0.00002000 sec

============ CHANNEL f1 ============
NUC1                 13C
P1                  8.05 usec
PL1                 6.00 dB
SFO1         125.7715719 MHz

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                  1H
PCPD2              82.00 usec
PL2                 0.00 dB
PL12               17.00 dB
PL13               20.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF           125.7577390 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 35.00 cm
F1P              215.000 ppm
F1              27037.92 Hz
F2P               -5.000 ppm
F2               -628.78 Hz
PPMCM            6.28571 ppm/cm
HZCM           790.47711 Hz/cm
```



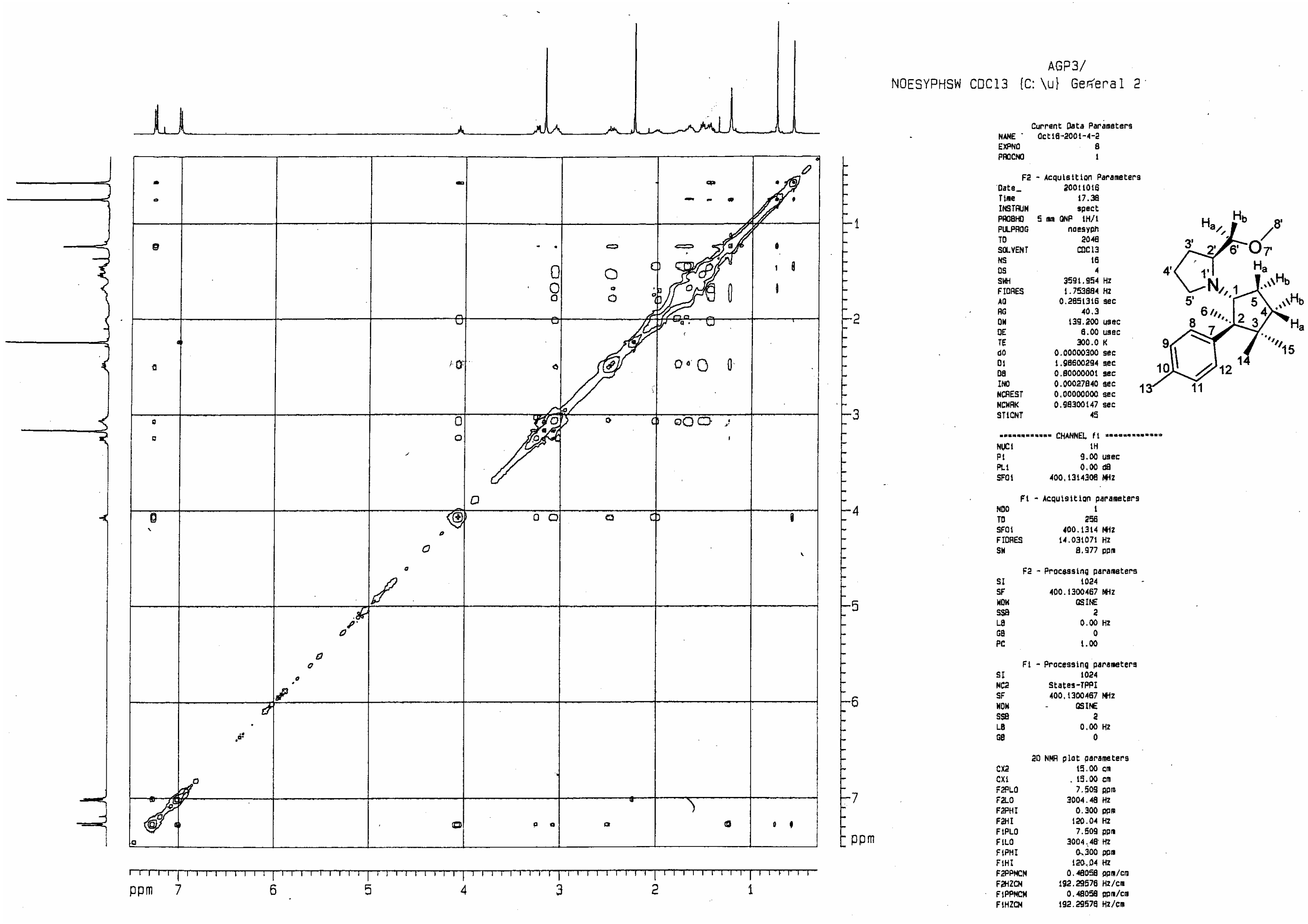
```
                                          AGP3/331(P1)
                          noesytpsw_a3 CDCl3 D:/u jeh 1

        Current Data Parameters
NAME          Feb0402-5.001
EXPNO                    31
PROCNO                    1

      F2 - Acquisition Parameters
Date_              20020205
Time                   0.45
INSTRUM              drx500
PROBHD       5 mm BBO BB-
PULPROG             noesytp
TD                     2048
SOLVENT               CDCl3
NS                       16
DS                        4
SWH                4280.822 Hz
FIDRES             2.090245 Hz
AQ                0.2392564 sec
RG                       32
DW                  116.800 usec
DE                     6.00 usec
TE                    300.0 K
d0               0.00000300 sec
D1               1.46026802 sec
D8               0.80000001 sec
IN0              0.00011680 sec

============ CHANNEL f1 =============
NUC1                     1H
P1                    11.75 usec
PL1                    0.00 dB
SFO1           500.1318580 MHz

      F1 - Acquisition parameters
ND0                       2
TD                      256
SFO1               500.1319 MHz
FIDRES            16.721960 Hz
SW                    8.559 ppm

      F2 - Processing parameters
SI                     1024
SF              500.1300147 MHz
WDW                   QSINE
SSB                       2
LB                     0.00 Hz
GB                        0
PC                     1.00

      F1 - Processing parameters
SI                     1024
MC2                    TPPI
SF              500.1300147 MHz
WDW                   QSINE
SSB                       2
LB                     0.00 Hz
GB                        0

        2D NMR plot parameters
CX2                   25.00 cm
CX1                   25.00 cm
F2PLO                 7.965 ppm
F2LO                3983.72 Hz
F2PHI                -0.594 ppm
F2HI                -297.10 Hz
F1PLO                 7.965 ppm
F1LO                3983.72 Hz
F1PHI                -0.594 ppm
F1HI                -297.10 Hz
F2PPMCM             0.34238 ppm/cm
F2HZCM            171.23288 Hz/cm
F1PPMCM             0.34238 ppm/cm
F1HZCM            171.23288 Hz/cm
```


AGP3/331/(P2)
proton_a3 CDCl3 D:/u jeh 2
Current Data Parameters
NAME Feb0402-5.002
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020204
Time 12.41
INSTRUM drx500
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
============ CHANNEL f1 ============
NUC1 1H
P1 11.75 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 5.00
1D NMR plot parameters
CX 35.00 cm
F1P 11.600 ppm
F1 5801.51 Hz
F2P -0.400 ppm
F2 -200.05 Hz
PPMCM 0.34286 ppm/cm
HZCM 171.47314 Hz/cm
1.303
1.518
1.591
1.606
1.619
1.742
1.754
2.314
2.329
2.532
2.572
3.113
3.130
3.145
3.153
3.238
3.253
3.304
3.312
3.321
3.329
4.123
4.140
4.158
6.981
7.031
7.074
7.091
7.143
7.158
7.174
7.255
7.272
7.335
7.351
Ha
Hb
6'
O
7'
8'
2'
3'
4'
5'
1'
N
1
5
4
3
2
Ha
Hb
Hb
Ha
15
14
6
7
8
9
10
11
12
13
85.010
2.864
12.126
0
1
2
3
4
5
6
7
8
9
10
11

AGP3/331/(P2)
c13cpdno.a3 CDCl3 D:/u jeh 2



```
Current Data Parameters
NAME        Feb0402-5.002
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20020204
Time                12.49
INSTRUM            drx500
PROBHD       5 mm 880 88-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             37593.984 Hz
FIDRES           0.573639 Hz
AQ              0.8716788 sec
RG                  724.1
DW                 13.300 usec
DE                   6.00 usec
TE                  300.0 K
D1             0.00100000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                  13C
P1                   8.05 usec
PL1                  6.00 dB
SFO1          125.7715719 MHz

============ CHANNEL f2 ============
CPDPRG2           waltz16
NUC2                   1H
PCPD2               82.00 usec
PL2                  0.00 dB
PL12                17.00 dB
PL13                20.00 dB
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                  32768
SF            125.7577390 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  35.00 cm
F1P               215.000 ppm
F1               27037.92 Hz
F2P                -5.000 ppm
F2                -628.78 Hz
PPMCM             6.28571 ppm/cm
HZCM            790.47711 Hz/cm
```



```
                AGP3/
NOESYPHSW CDC13 {C:\u} Gerferal 2

          Current Data Parameters
NAME      Oct16-2001-4-2
EXPNO                  8
PROCNO                 1

       F2 - Acquisition Parameters
Date_             20011016
Time                 17.38
INSTRUM              spect
PROBHD    5 mm QNP   1H/1
PULPROG            noesyph
TD                    2048
SOLVENT              CDC13
NS                      16
DS                       4
SWH             3591.954 Hz
FIDRES          1.753884 Hz
AQ             0.2851316 sec
RG                    40.3
DW               139.200 usec
DE                  6.00 usec
TE                 300.0 K
d0            0.00000300 sec
D1            1.98600294 sec
D8            0.80000001 sec
IN0           0.00027840 sec
NCREST        0.00000000 sec
NCWRK         0.98300147 sec
ST1CNT                  45

============ CHANNEL f1 =============
NUC1                    1H
P1                    9.00 usec
PL1                   0.00 dB
SF01           400.1314308 MHz

       F1 - Acquisition parameters
ND0                      1
TD                     256
SF01              400.1314 MHz
FIDRES           14.031071 Hz
SW                   8.977 ppm

       F2 - Processing parameters
SI                    1024
SF             400.1300467 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0
PC                    1.00

       F1 - Processing parameters
SI                    1024
MC2            States-TPPI
SF             400.1300467 MHz
WDW                  QSINE
SSB                      2
LB                    0.00 Hz
GB                       0

        2D NMR plot parameters
CX2                  15.00 cm
CX1                  15.00 cm
F2PLO                7.509 ppm
F2LO               3004.48 Hz
F2PHI                0.300 ppm
F2HI                120.04 Hz
F1PLO                7.509 ppm
F1LO               3004.48 Hz
F1PHI                0.300 ppm
F1HI                120.04 Hz
F2PPMCM            0.48058 ppm/cm
F2HZCM           192.29576 Hz/cm
F1PPMCM            0.48058 ppm/cm
F1HZCM           192.29576 Hz/cm
```