Supporting Information

Selective Indium-Mediated 1,2,4-Pentatrien-3-ylation of Carbonyl Compounds for the Efficient Synthesis of Vinyl Allenols

Jisu Park,[†] Sung Hong Kim,[‡] and Phil Ho Lee[*,†]

[†]National Research Laboratory for Catalytic Organic Reaction, Department of Chemistry, and Institute for Molecular Science and Fusion Technology, Kangwon National University, Chuncheon 200-701, Republic of Korea and [‡]Analysis Research Division, Daegu Center, Korea Basic Science Institute, Daegu 702-701, Republic of Korea

phlee@kangwon.ac.kr

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Experimental Section

General: Reactions were carried out in oven-dried glassware under nitrogen atmosphere. All commercial reagents were used without purification, and all solvents were reaction grade. DMF and CH_2Cl_2 were freshly distilled from CaH_2. THF was freshly distilled from sodium/benzophenone under nitrogen. Indium powder (-100 mesh, 99.99%, Cat. No. 26,403-2) was purchased from Aldrich Chem Co. All reaction mixtures were magnetically stirred and were monitored by thin-layer chromatography using Merck silica gel 60 F_{254} precoated glass plates, which were visualized with UV light and then, developed using either iodine or a solution of anisaldehyde. Flash column chromatography was carried out using Merck silica gel 60 (0.040-0.063 mm, 230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Brucker DPX FT (400 MHz) spectrometer. Deuterated chloroform was used as the solvent, and chemical shift values (δ) are reported in parts per million relative to the residual signals of this solvent (δ 7.24 for ^{1}H and δ 77.0 for ^{13}C). Infrared spectra were recorded on a JASCO FT/IR-460 plus FT-IR spectrometer as either a thin film pressed between two sodium chloride plates or as a solid suspended in a potassium bromide disk. High-resolution mass spectra were recorded on a Jeol JMS 700 high resolution mass spectrometer.

Preparation of pent-4-en-2-yn-1-ol[1]: To a solution of $PdCl_2(PPh_3)_2$ (281.0 mg, 0.4 mmol), CuI (152.4 mg, 0.8 mmol), disopropylamine (5.6 mL, 40 mmol), and vinyl bromide (20 mL of a 1.0 M solution in THF, 20 mmol) in THF (60 mL) was added propargyl alcohol (20 mmol) at room temperature. The reaction mixture was kept at room temperature for overnight and then, quenched with saturated NH_4Cl (30 mL). The aqueous layer was extracted with Et_2O (3 x 30 mL). The combined organic layers

were successively washed with aqueous HCl (0.2 M, 20 mL), $NaHCO_3$ (20 mL), and H_2O (2 x 30 mL). The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:5) to give pent-4-en-2-yn-1-ol (90%, 1.48 g)

Preparation of 4-methylpent-4-en-2-yn-1-ol: To a solution of $PdCl_2(PPh_3)_2$ (79.2 mg, 0.1 mmol), CuI (38.1 mg, 0.2 mmol), disopropylamine (1.4 mL, 10 mmol), and 2-bromopropene (445 μL, 5.0 mmol) in THF (30 mL) was added propargyl alcohol (292 μL, 5.0 mmol) at room temperature. The reaction mixture was kept at 60 ℃ for a 16 h and then, quenched with saturated NH_4Cl (10 mL). The aqueous layer was extracted with Et_2O (3 x 15 mL). The combined organic layers were successively washed with aqueous HCl (0.2 M, 10 mL), $NaHCO_3$ (10 mL), and H_2O (2 x 10 mL). The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:5) to give 4-methylpent-4-en-2-yn-1-ol (83%, 400.0 mg)

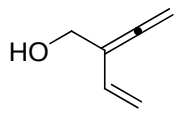
Preparation of (5-methoxy-3,4-dihydro-naphthalen-1-yl)-prop-2-yn-1-ol[2]: To a solution of $PdCl_2(PPh_3)_2$ (106.0 mg, 0.15 mmol), CuI (9.6 mg, 0.3 mmol), and 1-trifluoromethanesulfony-5-methoxy-3,4-dihydronaphthalene (925.0 mg, 3.0 mmol) in a 1:1 solution of THF:diethyl amine (30 mL) was added propargyl alcohol (192 μL, 3.3 mmol) at room temperature. The reaction mixture was kept at 60 ℃ for a 16 h and then, quenched with saturated NH_4Cl (10 mL). The aqueous layer was extracted with Et_2O (3

x 20 mL). The combined organic layers were successively washed with aqueous HCl (0.2 M, 10 mL), $NaHCO_3$ (10 mL), and H_2O (2 x 10 mL). The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:10) to give **(**5-methoxy-3,4-dihydro-naphthalen-1-yl)-prop-2-yn-1-ol (53%, 340.0 mg)

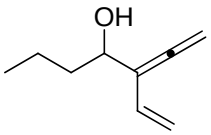
Bromination of propargylic alcohols[3]: Pyridine (8.1 μL, 0.1 mmol) was added to a solution of propargylic alcohol (1.0 mmol) in Et_2O (7 mL) at 0 ^oC. After being stirred for 10 min, phosphorus tribromide (32 μL, 0.34 mmol) in Et_2O (3 mL) was added and reaction mixture was stirred for 2 h at room temperature. The reaction mixture was quenched with ice $NaHCO_3$ (10 mL). The aqueous layer was extracted with Et_2O (3 x 10 mL), and combined organic layers were washed with water (10 mL) and brine (10 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography using hexane to give propargylic bromides (**2, 10** and **12**).

Experimental procedure of synthesis of 1-phenyl-2-vinyl-2,3-butadien-1-ol (3g)[4]**:** 1-Bromopent-4-en-2-yne (72.5 mg, 0.5 mmol) was added to suspension of indium (57.4 mg, 0.5 mmol) and lithium iodide (66.9 mg, 0.5 mmol) in dry THF (2 mL). After being stirred for 30 min at room temperature under nitrogen atmosphere, benzaldehyde (53.1 mg, 0.5 mmol) was added. After 2 h, the reaction mixture was quenched with saturated $NaHCO_3$. The aqueous layer was extracted with Et_2O (3 x 20 mL) and the combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated

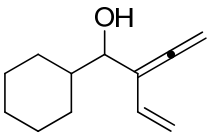
under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:10) to give 1-phenyl-2-vinyl-2,3-butadien-1-ol (75.8 mg, 88%).



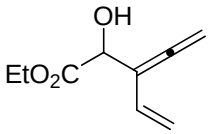
2-Vinyl-2,3-butadien-1-ol (3a): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.24 (dd, J = 17.6 Hz, 11.8 Hz, 1H), 5.23 (d, J = 17.6 Hz, 1H), 5.10 (d, J = 11.8 Hz, 1H), 5.08 (s, 2H), 4.32 (s, 2H), 1.56, (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.3, 131.7, 113.7, 105.3, 78.7; IR (film) 3348, 2927, 2871, 1938, 1615, 904, 852 cm^{-1}; HRMS (EI): calcd for C_6H_8O 96.0575, found 96.0572.



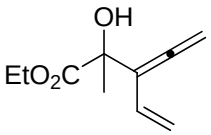
3-Vinyl-1,2-heptadien-4-ol (3b): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.18 (dd, J = 17.6 Hz, 10.8 Hz, 1H), 5.32 (d, J = 17.6 Hz, 1H), 5.14 (d, J = 10.8 Hz,1H), 5.08 (s, 1H), 4.36-4.31 (m, 1H), 1.72-1.35 (m, 5H), 0.94 (t, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.7, 132.0, 114.8, 109.1, 79.7, 69.3, 38.7, 19.3, 14.3; IR (film) 3396, 2959, 2932, 2872, 1936, 1681, 903, 849 cm^{-1} HRMS (EI): calcd for $C_9H_{14}O$ M^+ 138.1045, found 138.1040.



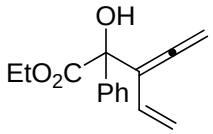
1-Cyclohexyl-2-vinyl-2,3-butadien-1-ol (3c): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.15 (dd, J = 17.6 Hz, 10.9 Hz, 1H), 5.32 (d, J = 17.6 Hz, 1H), 5.13 (d, J = 10.9 Hz,1H), 5.11-5.03 (m, 2H), 4.05 (dd, J = 11.1 Hz, 5.9 Hz, 1H), 1.90 (d, J = 11.1 Hz, 1H), 1.79-1.53 (m, 6H), 1.26-1.01 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.4, 131.4, 114.8, 107.5, 79.1, 74.1, 42.3, 29.9, 27.7, 26.5, 26.2, 26.0; IR (film) 3396, 2924, 2852, 1936, 1612, 1015, 906, 845 cm^{-1}; HRMS (EI): calcd for $C_{12}H_{18}O$ 178.1358, found 178.1355.



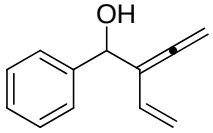
1-Ethoxylcarbonyl-2-vinyl-2,3-butadien-1-ol (3d): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.16 (dd, J = 17.6 Hz, 11.0 Hz, 1H), 5.40 (d, J = 17.6 Hz, 1H), 5.17 (d, J = 11.0 Hz, 1H), 5.15-5.07 (m, 2H), 4.84 (d, J = 5.1 Hz, 1H), 4.26 (q, J = 7.1 Hz, 2H), 3.13 (d, J = 5.1 Hz, 1H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 210.0, 173.4, 130.7, 115.8, 104.8, 79.8, 70.2, 62.4, 14.6; IR (film) 3461, 3095, 2982, 1939, 1737, 1615, 1082, 911, 862 cm^{-1}; HRMS (EI): calcd for $C_9H_{12}O_3$ 168.0786, found 168.0784.



2-Ethoxylcarbonyl-3-vinyl-3,4-pentadien-2-ol (3e): yellow oil;[1]H NMR (400 MHz, $CDCl_3$): δ 6.06 (dd, J = 17.5 Hz, 10.7 Hz, 1H), 5.38 (d, J = 17.5 Hz, 1H), 5.20-5.12 (m, 2H), 5.19 (d, J = 10.7 Hz, 1H), 4.25 (q, J = 7.1 Hz, 2H), 3.49 (s, 1H), 1.55 (s, 3H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.8, 176.1, 129.6, 117.1, 108.4, 80.9, 74.3, 62.7, 25.3, 14.5; IR (film) 3499, 2983, 1941, 1729, 1448, 1248, 1139, 936, 859 cm^{-1} HRMS (EI): calcd for $C_{10}H_{14}O_3$ 182.0943, found 182.0945.

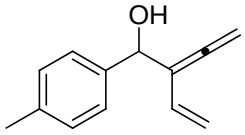


1-Ethoxylcarbonyl-1-phenyl-2-vinyl-2,3-butadien-1-ol (3f): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J = 8.7 Hz, 2H), 7.36-7.27 (m, 3H), 5.92 (dd, J = 17.3 Hz, 10.7 Hz, 1H), 5.35 (d, J = 17.3 Hz, 1H), 5.15 (d, J = 10.7 Hz, 1H), 5.07 (s, 2H), 4.29-4.13 (m, 2H), 4.08 (s, 1H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.3, 174.0, 139.7, 130.2, 128.4, 128.3, 127.6, 117.4, 108.2, 80.6, 80.2, 63.2, 14.4; IR (film) 3487, 3060, 2981, 1940, 1726, 1448, 1248, 1065, 910, 860 cm^{-1} HRMS (EI): calcd for $C_{15}H_{16}O_3$ 244.1099, found 244.1103.

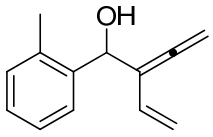


1-Phenyl-2-vinyl-2,3-butadien-1-ol (3g): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.27 (m, 5H), 6.11 (dd, J = 17.6 Hz, 10.8 Hz, 1H), 5.40 (s, 1H), 5.22 (d, J = 17.6

Hz, 1H), 5.18-5.10 (m, 2H), 5.08, (d, J = 10.8 Hz, 1H), 2.23 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.6, 141.9, 130.9, 128.4, 127.9, 126.8, 115.5, 108.7, 80.0, 71.9; IR (film) 3396, 3029, 2903, 1937, 1810, 1682, 1022, 908, 853 cm^{-1}; HRMS (EI): calcd for $C_{12}H_{12}O$ 172.0888, found 172.0888.

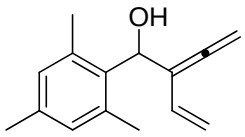


1-(*p*-Tolyl)-2-vinyl-2,3-butadien-1-ol (3h): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, J = 8.0 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 6.10 (dd, J = 17.7 Hz, 11.0 Hz, 1H), 5.35 (d, J = 5.1 Hz, 1H), 5.20 (d, J = 17.7 Hz, 1H), 5.19-5.10 (m, 2H), 5.06 (d, J = 11.0 Hz, 1H), 2.34 (s, 3H), 2.18 (d, J = 5.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.6, 139.0, 137.6, 131.0, 129.1, 126.7, 115.4, 108.8, 80.0, 71.6, 21.2; IR (film) 3388, 3013, 2920, 1937, 1801, 1615, 1038, 905, 853 cm^{-1}

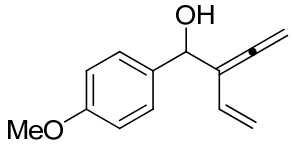


1-(*o*-Tolyl)-2-vinyl-2,3-butadien-1-ol (3i): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.44 (m, 1H), 7.22-7.14 (m, 3H), 6.15 (dd, J = 17.7 Hz, 10.8 Hz, 1H), 5.58 (s, 1H), 5.20 (d, J = 17.7 Hz, 1H), 5.09 (d, J = 10.8 Hz, 1H), 5.08-4.99 (m, 2H), 2.35 (s, 3H), 2.10 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.4, 140.0, 136.2, 131.7, 130.9, 128.1,

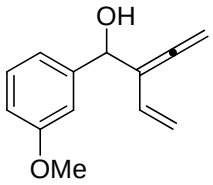
126.6, 126.4, 115.6, 108.5, 80.0, 69.4, 19.5; IR (film) 3366, 3021, 2922, 1937, 1812, 1614, 1026, 905, 850 cm^{-1}; HRMS (EI): calcd for $C_{13}H_{14}O$ 186.1045, found 186.1042.



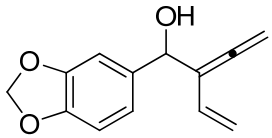
1-Mesityl-2-vinyl-2,3-butadien-1-ol (3j): white solid; mp: 55 ℃; ^1H NMR (400 MHz, $CDCl_3$): δ 6.81 (s, 2H), 5.97 (dd, J = 17.3 Hz, 11.1 Hz, 1H), 5.79 (d, J = 3.6 Hz, 1H), 5.24 (d, 17.3 Hz, 1H), 5.08-5.05 (m, 3H), 2.36 (s, 6H), 2.24 (s, 3H), 2.07 (d, 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.6, 137.1, 137.0, 133.8, 131.0, 130.0, 115.4, 108.1, 80.2, 68.0, 20.8, 20.5; IR (film) 3416, 2919, 1938, 1612, 1448, 1201, 1045, 1012, 904, 849 cm^{-1}; HRMS (EI): calcd for $C_{15}H_{18}O$ 214.1358, found 214.1358.



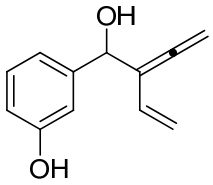
1-(4-Methoxyphenyl)-2-vinyl-2,3-butadien-1-ol (3k): yellow oil; ^1H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 6.10 (dd, J = 17.7 Hz, 11.1 Hz, 1H), 5.35 (d, J = 5.1 Hz, 1H), 5.18 (d, J = 17.7 Hz, 1H), 5.20-5.12 (m, 2H), 5.07 (d, J = 11.1 Hz, 1H), 3.80 (s, 3H), 2.12 (d, J = 5.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.5, 159.3, 134.1, 131.1, 128.1, 115.4, 113.8, 108.9, 80.2, 71.3, 55.3; IR (film) 3419, 3004, 2835, 1937, 1890, 1611, 906, 853 cm^{-1}; HRMS (EI): calcd for $C_{13}H_{14}O_2$ 202.0994, found 202.0998.



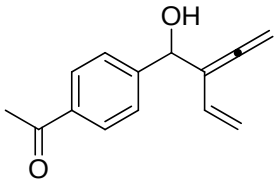
1-(3-Methoxyphenyl)-2-vinyl-2,3-butadien-1-ol (3l): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26 (dd, J = 8.1, 7.8 Hz, 1H), 6.99 (d, J = 7.8 Hz, 1H), 6.98(s, 1H), 6.83 (d, J = 8.1 Hz, 1H), 6.11 (dd, J = 17.6 Hz, 10.9 Hz, 1H), 5.37 (s, 1H), 5.23 (d, J = 17.6 Hz, 1H), 5.18-5.09 (m, 2H), 5.08 (d, J = 10.9 Hz, 1H), 3.80 (s, 3H), 2.27 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.6, 159.7, 143.6, 130.9, 129.4, 119.1, 115.5, 113.2, 112.4, 108.6, 80.0, 71.8, 55.2; IR (film) 3424, 3004, 2835, 1937, 1684, 1040, 907, 856 cm^{-1}; HRMS (EI): calcd for $C_{13}H_{14}O_2$ 202.0994, found 202.0996.



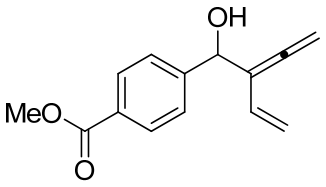
1-[3,4-(methylenedioxy)phenyl]-2-vinyl-2,3-butadien-1-ol (3m): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.90 (s, 1H), 6.88 (d, J = 7.9 Hz, 1H), 6.77 (d, J = 7.9 Hz, 1H), 6.09 (dd, J = 17.6, 10.9 Hz, 1H), 5.94 (s, 2H), 5.29 (d, J = 4.8 Hz, 1H), 5.22-5.12 (m, 2H), 5.19 (d, J = 17.6 Hz, 1H), 5.07 (d, J = 10.9 Hz, 1H), 2.25 (d, J = 4.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.8, 148.2, 147.6, 136.4, 131.3, 120.9, 115.9, 109.2, 115.9, 109.2, 108.5, 107.7, 101.5, 80.7, 71.9; IR (film) 3397, 3011, 2892, 1937. 1850, 1611, 1092, 927, 858 cm^{-1}; HRMS (EI): calcd for $C_{13}H_{12}O_3$ 216.0786, found 216.0786.



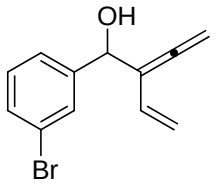
1-(3-Hydroxyphenyl)-2-vinyl-2,3-butadien-1-ol (3n): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16 (dd, J = 8.0, 7.8 Hz, 1H), 6.90 (d, J = 7.8 Hz, 1H), 6.88 (s, 1H), 6.72 (d, J = 8.0 Hz, 1H), 6.51 (bs, 1H), 6.07 (dd, J = 17.7 Hz, 11.0 Hz, 1H), 5.31 (s, 1H), 5.18 (d, J = 17.7 Hz, 1H), 5.13-5.04 (m, 2H), 5.05 (d, J = 11.0 Hz, 1H), 2.91 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.6, 155.8, 143.3, 130.8, 129.6, 119.1, 115.5, 115.2, 113.9, 108.2, 80.2, 71.7; IR (film) 3348, 2899, 1937, 1846, 1600, 1021, 906, 858 cm^{-1}; HRMS (EI): calcd for $C_{12}H_{12}O_2$ 188.0837, found 188.0837.



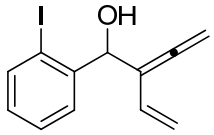
1-(4-Acetophenyl)-2-vinyl-2,3-butadien-1-ol (3o): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 8.4 Hz, 2H), 6.11 (dd, J = 18.5 Hz, 11.3 Hz, 1H) 5.49 (s, 1H), 5.26 (d, J = 18.5 Hz, 1H), 5.15-5.08 (m, 3H), 2.60 (s, 3H), 2.43 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.8, 197.9, 147.2, 136.5, 130.6, 128.5, 126.8, 115.8, 108.3, 80.1, 71.6, 26.7; IR (film) 3423, 3054, 2921, 1937, 1810, 1677, 1607, 1014, 910, 856 cm^{-1} HRMS (EI): calcd for $C_{14}H_{14}O_2$ 214.0994, found 214.0993



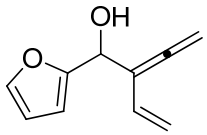
1-(4-Methoxycarbonylphenyl)-2-vinyl-2,3-butadien-1-ol (3p): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 8.3 Hz, 2H), 7.47 (d, J = 8.3 Hz 2H), 6.10 (dd, J = 17.6 Hz, 11.1 Hz, 1H), 5.47 (s, 1H), 5.25 (d, J = 17.6 Hz, 1H), 5.13-5.04 (m, 3H), 3.90 (s, 3H), 2.58 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.3, 167.4, 147.4, 131.1, 130.1, 129.8, 127.0, 116.1, 108.7, 80.4, 72.1, 52.5 ; IR (film) 3459, 3055, 2952, 1937, 1720, 1610, 1017, 908, 864 cm^{-1} HRMS (EI): calcd for $C_{14}H_{14}O_3$ 230.0943, found 230.0942



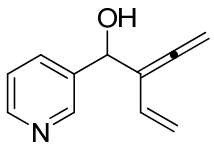
1-(3-Bromophenyl)-2-vinyl-2,3-butadien-1-ol (3q): brown oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (s, 1H), 7.41 (d, J = 7.9 Hz, 1H), 7.33 (d, J = 7.7 Hz, 1H), 7.21 (dd, J = 7.9 Hz, 7.7 Hz, 1H), 6.10 (dd, J = 17.7 Hz, 10.9 Hz, 1H), 5.38 (s, 1H), 5.23 (d, J = 17.6 Hz, 1H), 5.19-5.10 (m, 2H), 5.11 (d, J = 10.9 Hz, 1H), 2.23 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.1, 144.6, 131.3, 131.0, 130.3, 130.2, 125.8, 122.9, 116.2, 108.7, 80.7, 71.7; IR (film) 3388, 3060, 2888, 1936, 1807, 1682, 1071, 906, 855, 1024, 689 cm^{-1} HRMS (EI): calcd for $C_{12}H_{11}BrO$ M^+ 249.9993, found 249.9988



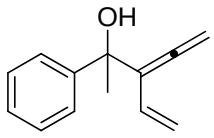
1-(2-Iodophenyl)-2-vinyl-2,3-butadien-1-ol (3r): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 7.9, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.35 (dd, J = 7.8 Hz, 7.6 Hz, 1H), 6.98 (dd, J = 7.9 Hz, 7.6 Hz, 1H), 6.19 (dd, J = 17.7 Hz, 11.2 Hz, 1H), 5.61 (d, J = 4.9 Hz, 1H), 5.28 (d, J = 17.7Hz, 1H), 5.13 (d, J = 11.2 Hz, 1H), 4.99 (s, 2H), 2.31 (d, J = 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.9, 144.0, 139.8, 131.7, 129.9, 128.7, 128.4, 115.7, 108.4, 99.3, 80.2, 75.5; IR (film) 3356, 3057, 2911, 1936, 1811, 1686, 1009, 903, 851, 748 cm^{-1} HRMS (EI): calcd for $C_{12}H_{11}IO$ 297.9855, found 297.9859



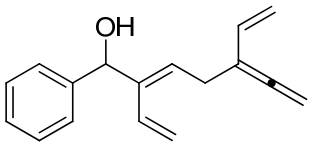
1-(2-Furanyl)-2-vinyl-2,3-butadien-1-ol (3s): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (dd, J = 1.8, 1.0 Hz, 1H), 6.33 (dd, J = 3.2 Hz, 1.8 Hz, 1H), 6.31 (dd, J = 3.2 Hz, 1.0 Hz, 1H), 6.17 (dd, J = 17.7 Hz, 10.9 Hz, 1H), 5.41 (s, 1H), 5.23 (d, J = 17.7 Hz, 1H), 5.24-5.16 (m, 2H), 5.11 (d, J = 10.9 Hz, 1H), 2.29 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.4, 154.5, 142.4, 130.7, 115.1, 110.3, 107.5, 106.8, 80.8, 65.4; IR (film) cm^{-1}; 3387, 3091, 2890, 1939, 1616, 1010, 906, 856 cm^{-1} HRMS (EI): calcd for $C_{10}H_{10}O_2$ 162.0681, found 162.0677.



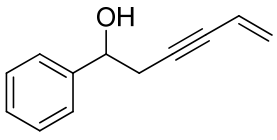
1-(3-Pyridinyl)-2-vinyl-2,3-butadien-1-ol (3t): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 8.41 (d, J = 4.9 Hz, 1H), 7.77 (d, J = 7.6 Hz, 1H), 7.26 (dd J = 7.6 Hz, 4.9 Hz, 1H), 6.13 (dd J = 17.7 Hz, 10.9 Hz, 1H), 5.48 (s, 1H), 5.28 (d, J = 17.7 Hz, 1H), 5.11 (d, J = 10.9 Hz, 1H), 5.09-5.00 (m, 2H), 4.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 209.6, 148.8, 148.5, 138.6, 135.1, 131.2, 123.7, 116.1, 108.6, 80.1, 70.1; IR (film) cm^{-1}; 3159, 2850, 1936, 1614, 1188, 1027, 908, 853 cm^{-1} HRMS (EI): calcd for $C_{11}H_{11}NO$ 173.0841, found 173.0840.



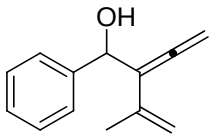
2-Phenyl-3-vinyl-3,4-pentadien-2-ol (3u): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, J = 7.2 Hz, 2H), 7.43 (t, J = 7.2 Hz, 2H), 7.25 (t, J = 7.2 Hz, 1H), 5.88 (dd J = 17.5 Hz, 10.7 Hz, 1H), 5.20 (s, 2H), 5.10 (d, J = 10.7 Hz, 1H), 2.15 (s, 1H), 1.66 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.8, 146.2, 130.5, 128.2, 126.9, 125.3, 117.0, 111.6, 80.0, 74.6, 31.1; IR (film) 3434, 3058, 2977, 1937, 1613, 1446, 1068, 909, 850 cm^{-1} HRMS (EI): calcd for $C_{13}H_{14}O$ 186.1045, found 186.1043.



Experimental procedure of synthesis of 1-phenyl-2,5-divinyl-2,5,6-heptatrien-1-ol (4): 1-bromopent-4-en-2-yne (145.0 mg, 1.0 mmol) was added to suspension of indium (114.82 mg, 1.0 mmol) in dry THF (2 mL). After stirring the mixture for 30 min at room temperature under nitrogen atmosphere, benzaldehyde (53.1 mg, 0.5 mmol) was added and stirred for 1 h. The reaction mixture was quenched with saturated $NaHCO_3$. The aqueous layer was extracted with Et_2O (3 x 20 mL) and the combined organic phase was washed with brine. The resulting organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:10) to give 1-phenyl-2,5-divinyl-2,5,6-heptatrien-1-ol (88.0 mg, 74%). yellow oil; ^1H NMR (400 MHz, $CDCl_3$): δ 7.42-7.25 (m, 5H), 6.52 (dd, J = 17.9 Hz, 11.5 Hz, 1H), 6.31 (dd, J = 17.5 Hz, 10.7 Hz, 1H), 5.91 (t, J = 7.4 Hz, 1H), 5.50 (s, 1H), 5.23 (d, J = 17.9 Hz, 1H), 5.16 (d, J = 11.5 Hz, 1H), 5.15 (d, J = 17.5 Hz, 1H), 5.05 (d, J = 10.7 Hz, 1H), 4.92(d, J = 3.0 Hz, 2H), 3.09 (dd, J = 7.4, 3.0 Hz, 2H), 1.94(s, 1H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 211.3, 142.5, 139.3, 134.3, 130.8, 128.3, 128.1, 127.5, 126.7, 116.8, 112.8, 103.5, 76.9, 75.1, 26.3; IR (film) 3406, 3027, 2923, 1937, 1811, 1614, 900, 851 cm^{-1}; HRMS (EI): calcd for $C_{15}H_{16}O_3$ 238.1352, found 238.1355.

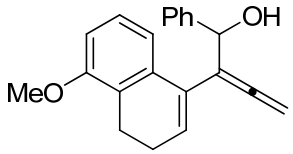


1-Phenylhex-5-en-3-yn-1-ol (5): yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.27 (m, 5H), 5.77 (ddt, J = 17.5 Hz, 11.0 Hz, 2.1 Hz, 1H), 5.59 (dd, J = 17.5 Hz, 2.1 Hz, 1H), 5.43 (dd, J = 11.0 Hz, 2.1 Hz, 1H), 4.86 (td, J = 6.2, 2.8 Hz, 1H), 2.75 (d, J = 6.2 Hz, 2H), 2.47 (d, J = 2.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.6, 128.4, 127.9, 126.6, 125.7, 117.1, 86.7, 81.8, 72.5, 30.5; IR (film) 3369, 3030, 2228, 1834, 1607, 1047, 916cm^{-1} HRMS (EI): calcd for $C_{12}H_{12}O$ 172.0888, found 172.0888.



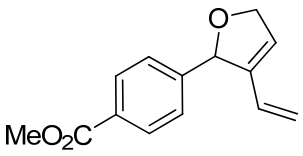
Experimental procedure of synthesis of 1-phenyl-2-(prop-1-en-2-yl)-2,3-butadien-1-ol (11)[5]: 1-bromo-4-methylpent-4-en-2-yne (80.0 mg, 0.5 mmol) was added to suspension of indium (57.4 mg, 0.5 mmol) and lithium iodide (66.9 mg, 0.5 mmol) in dry THF (2 mL). After being stirred for 30 min at room temperature under nitrogen atmosphere, benzaldehyde (53.1 mg, 0.5 mmol) was added. After 2 h, the reaction mixture was quenched with saturated $NaHCO_3$. The aqueous layer was extracted with Et_2O (3 x 20 mL) and the combined organic layers was washed with brine. The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:10) to give 1-phenyl-2-(prop-1-en-2-yl)-2,3-butadien-1-ol (83.6 mg, 90%). colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.24 (m, 5H), 5.47 (s, 1H), 5.14 (q,d, J = 12.5 Hz, 1.2 Hz, 2H), 4.91 (d, J = 5.8 Hz, 2H), 2.35 (bs, 1H), 1.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.9, 142.9, 137.5, 128.7, 128.2, 127.3, 113.0,

112.1, 81.7, 71.8, 22.9; IR (film) 3397, 3028, 2919, 1934, 1771, 1618, 1375, 1187, 1020, 850, 748. 699 cm^{-1}.

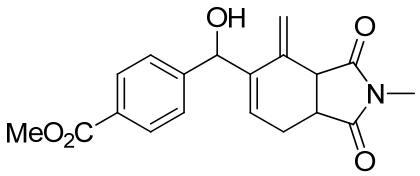


Experimental procedure of synthesis of 2-(5-methoxy-3,4-dihydronaphthalen-1-yl)-1-phenyl-2,3-butadien-1-ol (13): 3-(3,4-dihydro-5-methoxy-1-naphthalenyl)-2-propyn-1-bromide (111.0 mg, 0.4 mmol) was added to a suspension of indium (45.9 mg, 0.4 mmol) and lithium iodide (53.5 mg, 0.4 mmol) in dry THF (2 mL). After being stirred for 30 min at room temperature under nitrogen atmosphere, benzaldehyde (42.5 mg, 0.4 mmol) was added. After 1 h, the reaction mixture was quenched with saturated $NaHCO_3$. The aqueous layer was extracted with Et_2O (3 x 20 mL) and the combined organic layers were washed with brine. The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silicagel column chromatography (ethyl acetate:hexane=1:10) to give 2-(5-methoxy-3,4-dihydronaphthalen-1-yl)-1-phenyl-2,3-butadien-1-ol (91.0 mg, 75 %). yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.22 (m, 5H), 7.15 (dd, J = 7.7 Hz, 8.2 Hz, 1H), 7.00 (d, J = 7.7 Hz, 1H), 6.79 (d, J = 8.2 Hz, 1H), 5.87 (t, J = 4.7 Hz, 1H), 5.33 (ds, 1H), 5.02 (m, 2H), 3.82 (s, 3H), 2.80 (dt, J= 15.4 Hz, 7.2 Hz, 1H), 2.56 (dt, J= 16.1 Hz, 8.1 Hz, 1H), 2.39 (d, J= 4.0 Hz, 1H), 2.19-2.14 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.8, 155.0, 141.1, 133.6, 131.4, 128.2, 127.1, 126.6, 125.6, 125.3, 123.5, 116.6, 108.9, 107.8, 78.2, 72.0, 54.5, 21.7, 18.5; IR (film) 3425, 3028, 2936, 2833, 1951, 1571, 1468,

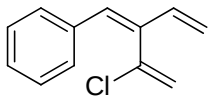
1340, 1260, 1074, 915, 842, 768, 721 cm^{-1}; HRMS (EI): calcd for $C_{21}H_{20}O_2$ 304.1463, found 304.1461.



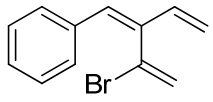
Experimental procedure of synthesis of 2-(4-methoxycarbonylphenyl)-3-vinyl-2,5-dihydrofuran (14): 1-(4-Methoxycarbonylphenyl)-2-vinyl-2,3-butadien-1-ol (69.1 mg, 0.3 mmol) was added to a suspension of $AuCl_3$ (4.6 mg, 5 mol%) in dry CH_2Cl_2 (1.2 mL). After being stirred for 10 min at room temperature under nitrogen atmosphere, methylene chloride was removed under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:10) to give 2-(4-methoxycarbonylphenyl)-3-vinyl-2,5-dihydrofuran (65.9 mg, 95%). yellow oil; ^1H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.2 Hz, 2H), 6.40 (dd, J = 17.8 Hz, 11.1 Hz, 1H), 6.11 (s, 1H), 5.84 (dd, J = 2.6, 2.5 Hz, 1H), 5.02 (d, J = 11.1 Hz, 1H), 4.92-4.75 (m, 2H), 4.82 (d, J = 17.8 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 146.5, 140.5, 130.4, 130.3, 129.1, 128.0, 127.2, 118.1, 87.3, 75.6, 52.5; IR (film) 3091, 2951, 2846, 1933, 1722, 1435, 1281, 1192, 1112, 1064, 914, 854 cm^{-1} HRMS (EI): calcd for $C_{14}H_{14}O_3$ 230.0943, found 230.0946.



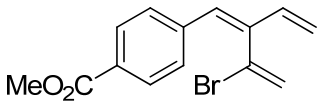
Experimental procedure of synthesis of methyl 4-(hydroxy(2-methyl-4-methylene-1,3-dioxo-2,3,3a,4,7,7a-hexahydro-1H-isoindol-5-yl)methyl)benzoate (15): *N*-Methyl maleimide (33.4 mg, 0.3 mmol) was added to a suspension of 1-(4-Methoxy-carbonylphenyl)-2-vinyl-2,3-butadien-1-ol (69.1 mg, 0.3 mmol) in dry methylene chloride (1.2 mL). After being stirred for 3 h at room temperature under nitrogen atmosphere, the methylene chloride was removed under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate:hexane=1:2) to give methyl 4-(hydroxy(2-methyl-4-methylene-1,3-dioxo-2,3,3a,4,7,7a-hexahydro-1*H*-isoi ndol-5-yl)-methyl)benzoate (86.7 mg, 85%). White solide; mp: 66 ℃; **Isomer A**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 5.92-5.90 (m, 1H), 5.45 (s, 1H), 5.37 (s, 1H), 5.35 (s, 1H), 3.90 (s, 3H), 3.63 (d, J = 9.1 Hz, 1H), 3.18-3.12 (m, 1H), 2.90 (s, 3H), 2.85-2.78 (m, 2H), 2.42-2.34 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.7, 176.8, 167.3, 147.1, 140.8, 134.3, 130.0, 127.0, 126.7, 116.3, 74.8, 52.5, 47.4, 39.0, 25.8, 23.5, 14.6; **Isomer B**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.3 Hz, 2H), 6.17-6.16 (m, 1H), 5.47 (s, 1H), 5.32 (s, 1H), 5.26 (s, 1H), 3.88 (s, 3H), 3.60 (d, J = 9.1 Hz, 1H), 3.18-3.12 (m, 1H), 2.77-2.71 (m, 2H), 2.73 (s, 3H), 2.34-2.28 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.4, 171.7, 167.2, 147.6, 141.2, 133.8, 129.8, 126.9, 124.2, 116.3, 73.9, 60.8, 47.9, 39.4, 25.6, 23.2, 21.4; IR (film) 3458, 2952, 1611, 1437, 1283, 1190, 1114, 914, 870 cm^{-1} HRMS (EI): calcd for $C_{19}H_{19}NO_5$ 341.1263, found 341.1262.



Experimental procedure of synthesis of 2-Chloro-4-phenyl-3-vinyl-1,3-butadiene (16)[6]: 1-Phenyl-2-vinyl-2,3-butadien-1-ol (51.7 mg, 0.3 mmol) was added to a suspension of $InCl_3$ (66.4 mg, 0.3 mmol) in dry CH_2Cl_2 (1.2 mL). After being stirred for 10 min at room temperature under nitrogen atmosphere, the reaction mixture was quenched with saturated H_2O. The aqueous layer was extracted with CH_2Cl_2 (3 x 20 mL) and the combined organic layers were washed with brine. The resulting organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to give 2-Chloro-4-phenyl-3-vinyl-1,3-butadiene (41.4 mg, 72%).[6]**:** yellow oil; **Isomer A**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, J = 7.6 Hz, 2H), 7.36-7.31 (m, 3H), 6.48 (s, 1H), 6.45 (dd, J = 17.1 Hz, 10.6 Hz, 1H), 5.63 (d, J = 1.0 Hz, 1H), 5.46 (d, J = 17.1 Hz, 1H), 5.30 (d, J = 1.0 Hz, 1H), 5.27 (d, J = 10.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 137.8, 135.9, 133.0, 132.3, 129.8, 128.7, 128.4, 118.5, 116.6; **Isomer B**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.31 (m, 3H), 7.29-7.24 (m, 2H), 6.88 (s, 1H), 6.63 (dd, J = 17.6 Hz, 11.1 Hz, 1H), 5.57 (d, J = 0.7 Hz, 1H), 5.54 (d, J = 0.7 Hz, 1H), 5.51 (d, J = 17.6 Hz, 1H), 5.45 (d, J = 11.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 138.0, 136.5, 135.7, 131.9, 130.1, 128.6, 128.1, 121.0, 116.8; IR (film) 2925, 1734, 1644, 1492, 983, 893, 694 cm^{-1} HRMS (EI): calcd for $C_{12}H_{11}Cl$ 190.0549, found 190.0548.



2-Bromo-4-phenyl-3-vinyl-1,3-butadiene (17): yellow oil; **Isomer A**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, J = 7.5 Hz, 2H), 7.34 (m, 3H), 6.44 (dd, J = 17.1 Hz, 10.6 Hz, 1H), 6.43 (s, 1H), 5.88 (d, J = 1.6 Hz, 1H), 5.73 (d, J = 1.6 Hz, 1H), 5.48 (d, J = 17.1 Hz, 1H), 5.28 (d, J = 10.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.9, 137.7, 132.6, 131.5, 130.1, 128.6, 128.2, 125.8, 122.8, 116.8; **Isomer B**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (m, 3H), 7.28 (m, 2H), 6.82 (s, 1H), 6.65 (dd, J = 17.5 Hz, 11.1 Hz, 1H), 5.94 (d, J = 1.2 Hz, 1H), 5.78 (d, J = 1.2 Hz, 1H), 5.54 (d, J = 17.5 Hz, 1H), 5.45 (d, J = 11.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.9, 136.4, 135.9, 132.4, 131.1, 130.1, 128.8, 128.4, 121.3, 120.9; IR (film) 2923, 1638, 1492, 981, 896, 693cm^{-1} HRMS (EI): calcd for $C_{12}H_{11}Br$ 234.0044, found 234.0042.



2-Bromo-4-(4-methoxycarbonylphenyl)-3-vinyl-1,3-butadiene (18): yellow oil; **Isomer A**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-7.99 (m, 2H), 7.63 (d, J = 8.5 Hz, 2H), 6.45 (s, 1H), 6.44 (dd, J = 17.1 Hz, 10.5Hz, 1H), 5.89 (d, J = 1.6 Hz, 1H), 5.73 (d, J = 1.6 Hz, 1H), 5.54 (d, J = 17.1 Hz, 1H), 5.35 (d, J = 10.5 Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 141.5, 140.0, 136.9, 131.0, 130.7, 129.6, 129.4, 129.1, 122.8, 117.8, 52.1; **Isomer B**: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-7.99 (m, 2H), 7.41 (d, J = 7.4 Hz, 2H), 6.84 (s, 1H), 6.60 (dd, J = 17.6 Hz, 11.5 Hz, 1H), 5.97 (d, J =

1.4 Hz, 1H), 5.82 (d, J = 1.4 Hz, 1H), 5.57 (d, J = 17.6 Hz, 1H), 5.50 (d, J = 10.5 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.8, 141.0, 140.6, 130.8, 130.2, 129.6, 129.5, 129.1, 124.7, 121.7, 121.5, 52.1; IR (film) 2950, 1721, 1607, 1434, 1278, 1182, 1109, 899, 700 cm^{-1} HRMS (EI): calcd for $C_{14}H_{13}BrO_2$ 292.0099, found 292.0103.

References

(1) Hamze, A.; Provot, O.; Brion, J.-D.; Alami, M. *J. Org. Chem.* **2007,** *72,* 3868.

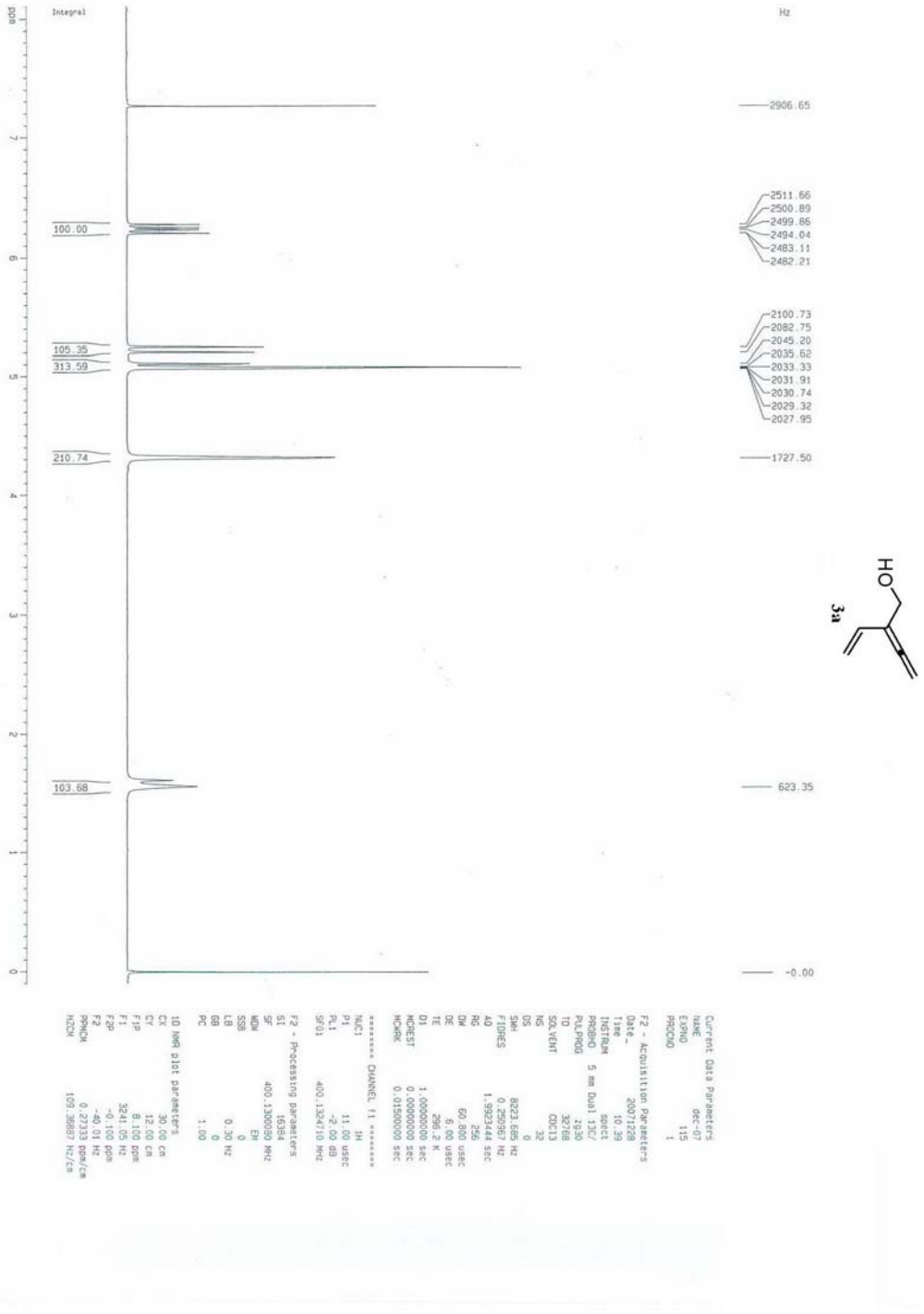
(2) Tessier, P. E.; Nguyen, N.; Clay, M. D.; Fallis, A. G. *Org. Lett.* **2005,** *7,* 767.

(3) Clardy, J.; Forsyth, C. J. *J. Am. Chem. Soc.* **1990**, *112*, 3497. (b) Laird, T.; Ollis, D. W.; Ian, O. *J. Chem. Soc. Perkin Trans. 1*, **1980,** *9,* 2033.

(4) (a) Delbecq, F.; Baudouy, R.; Goré, J. *Nou. J. Chim.* **1979,** *3,* 321.

(5) (a) Khrimyan, A. P.; Makaryan, G. M.; Badanyan, S. O. *Zh. Org. Khim,* **1988,** *24,* 94. (b) Doutheau, A.; Gore, J.; Diab, J. *Tetrahedron* **1985,** *41,* 329.

(6) (a) Horváth, A.; Bäckvall, J. E. *J. Org. Chem.* **2001,** *66,* 8120. (b) Wang, G.; Ma, S. *Tetrahedron Lett.* **2002,** *43,* 5723.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2
3
4
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6
7
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MCWRK 0.01500000 sec
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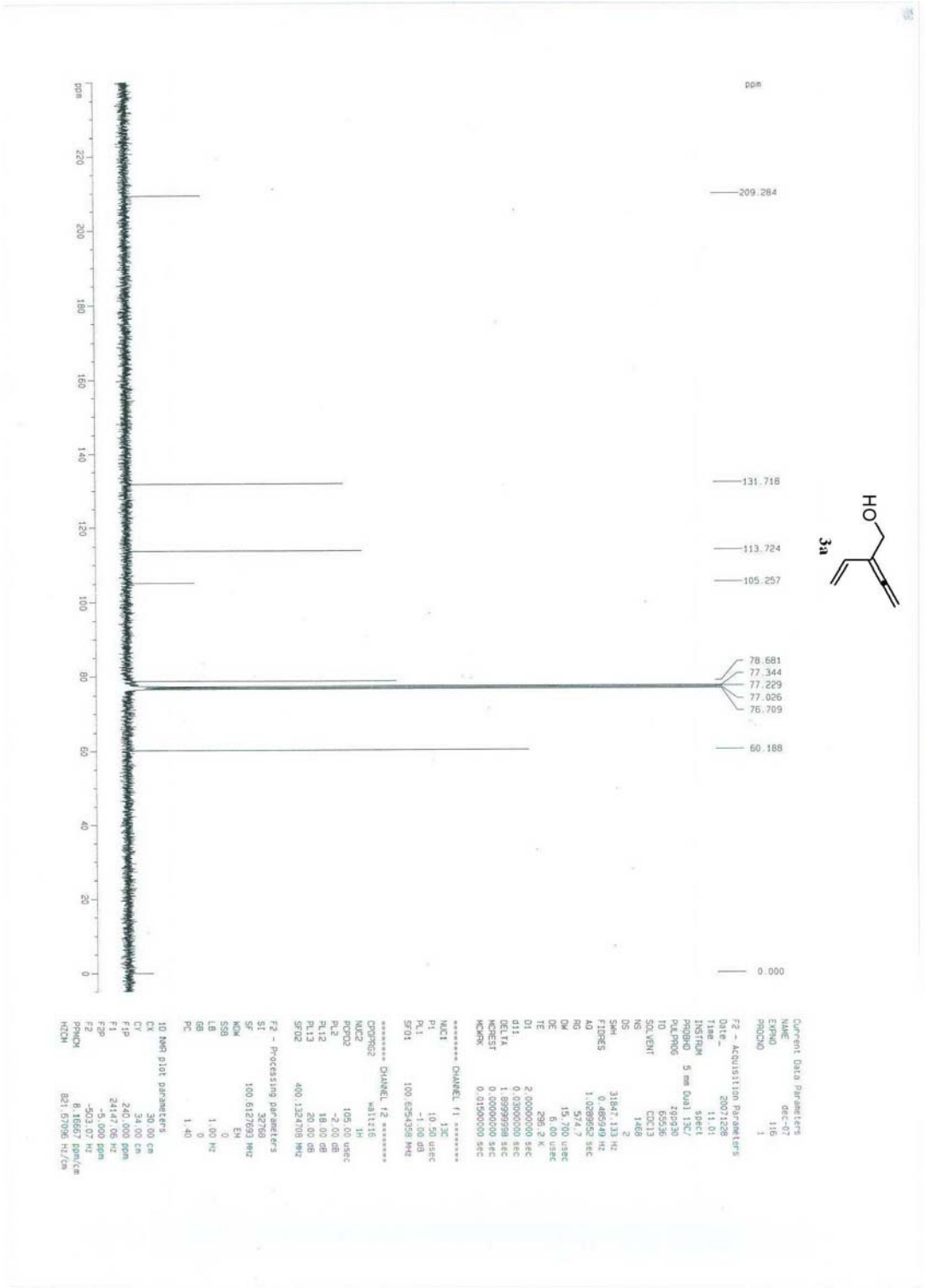
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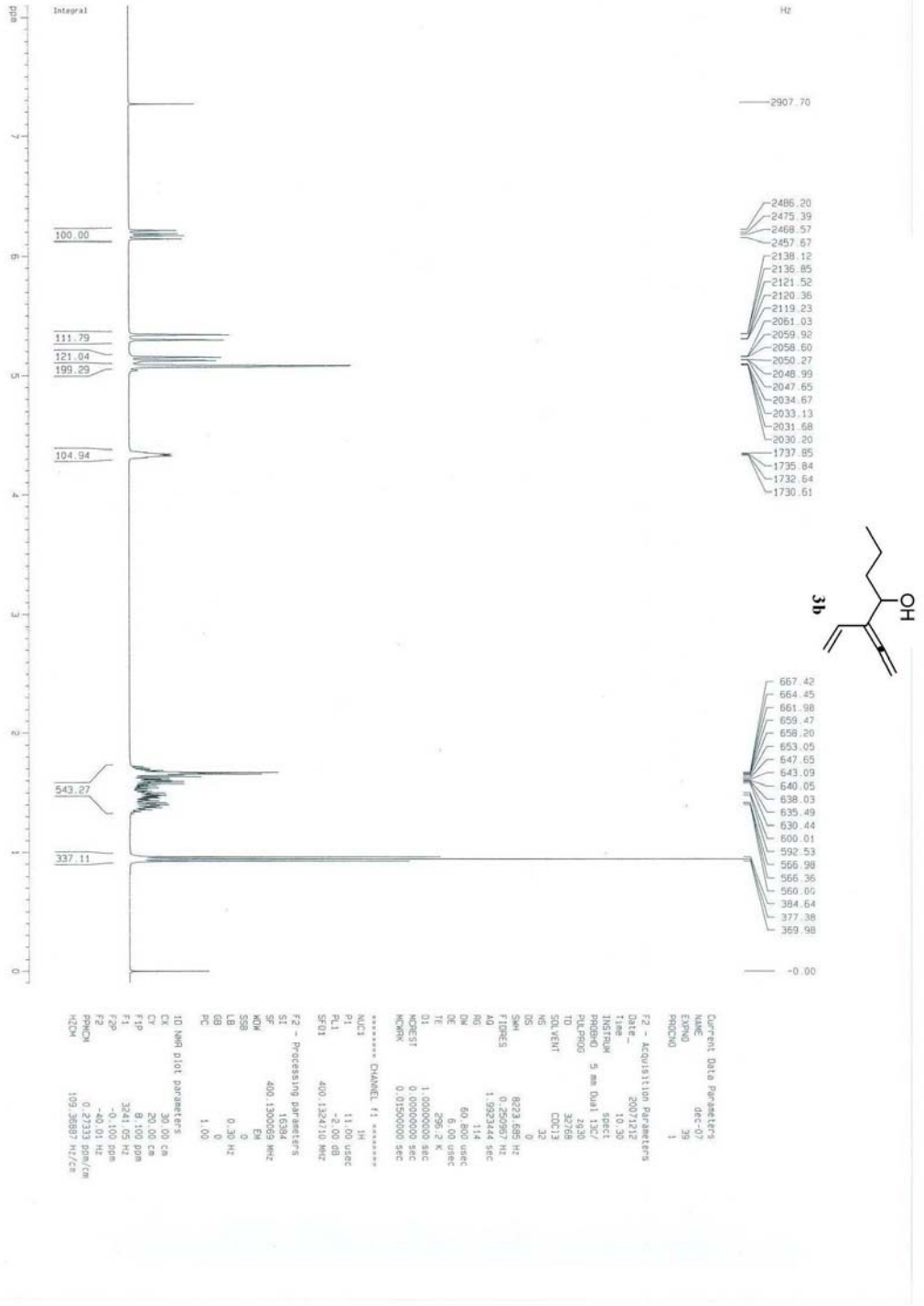
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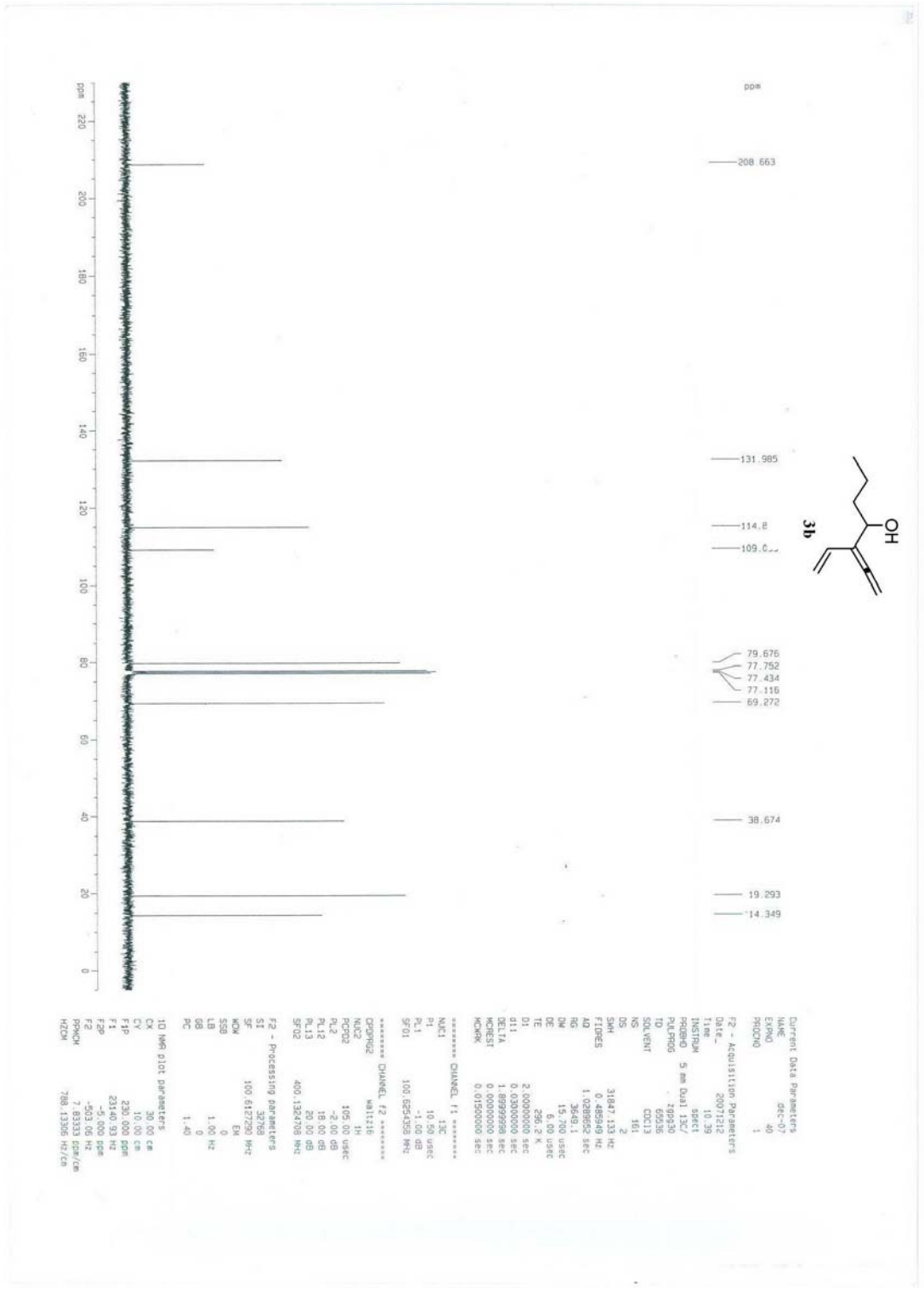
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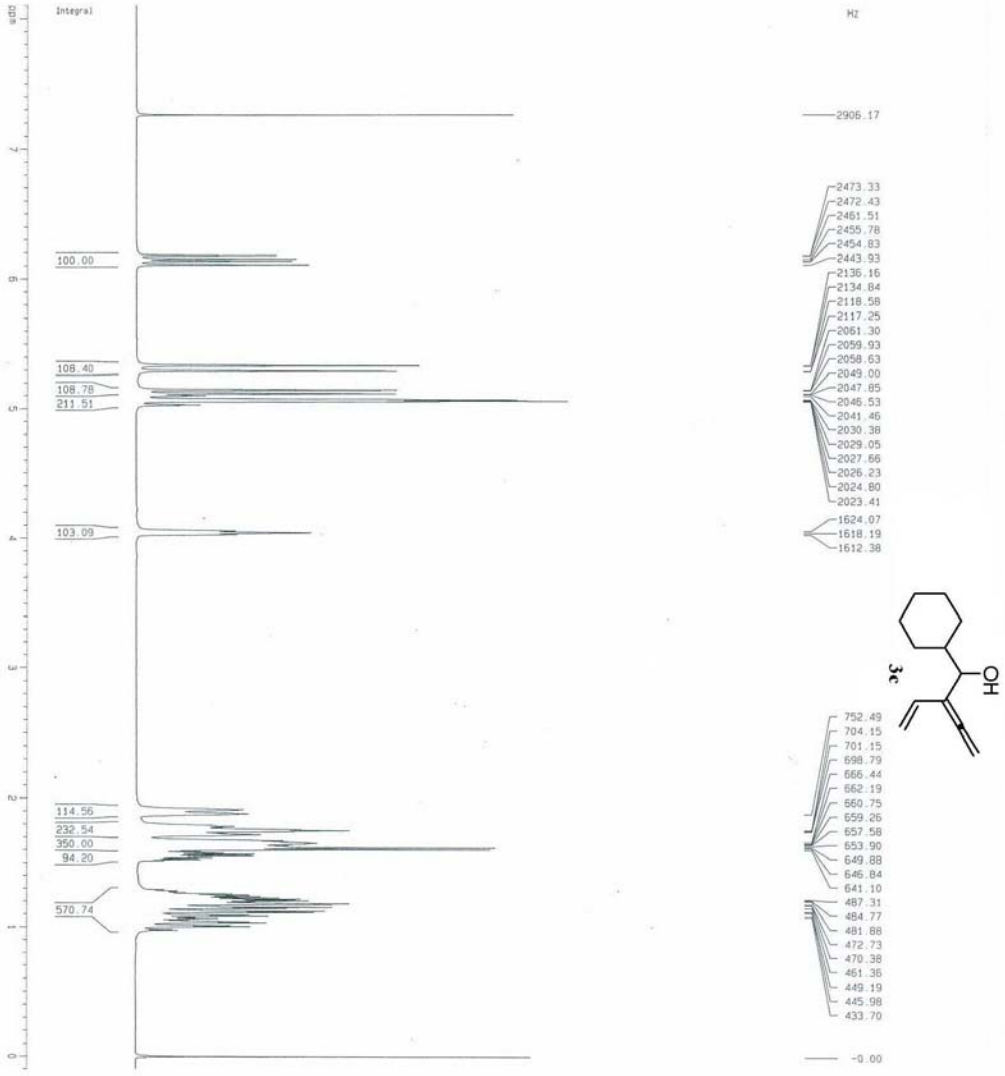

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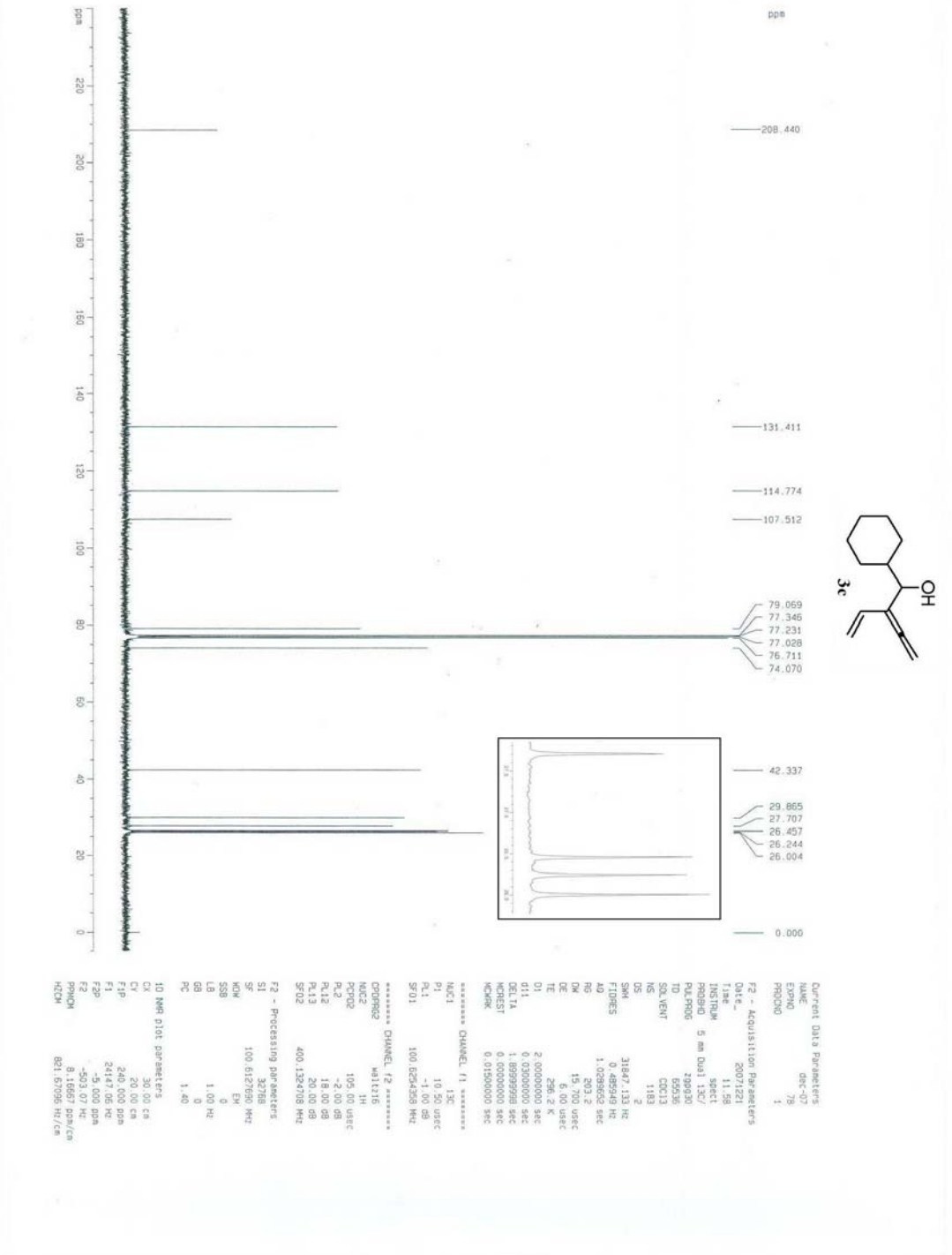
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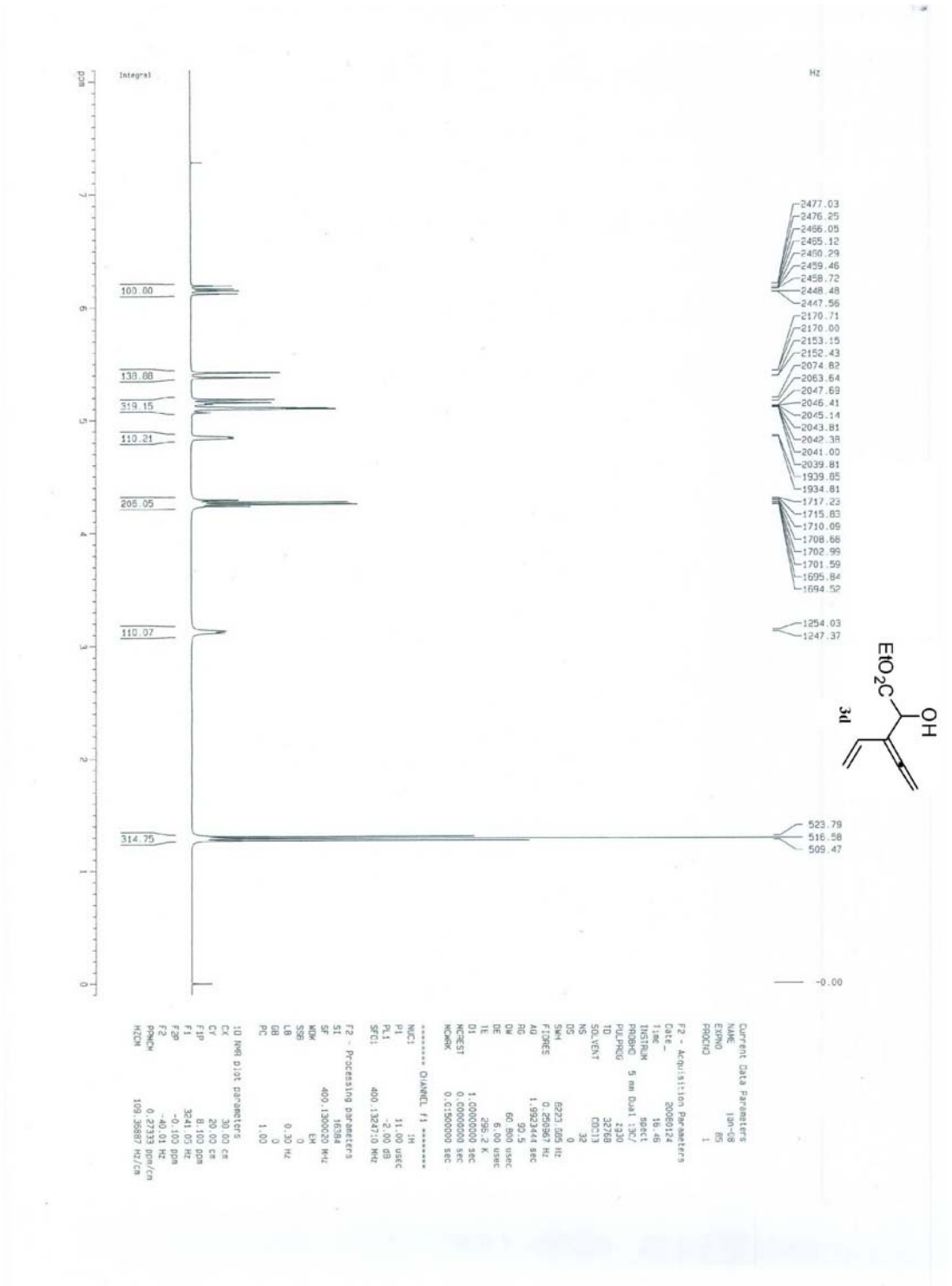
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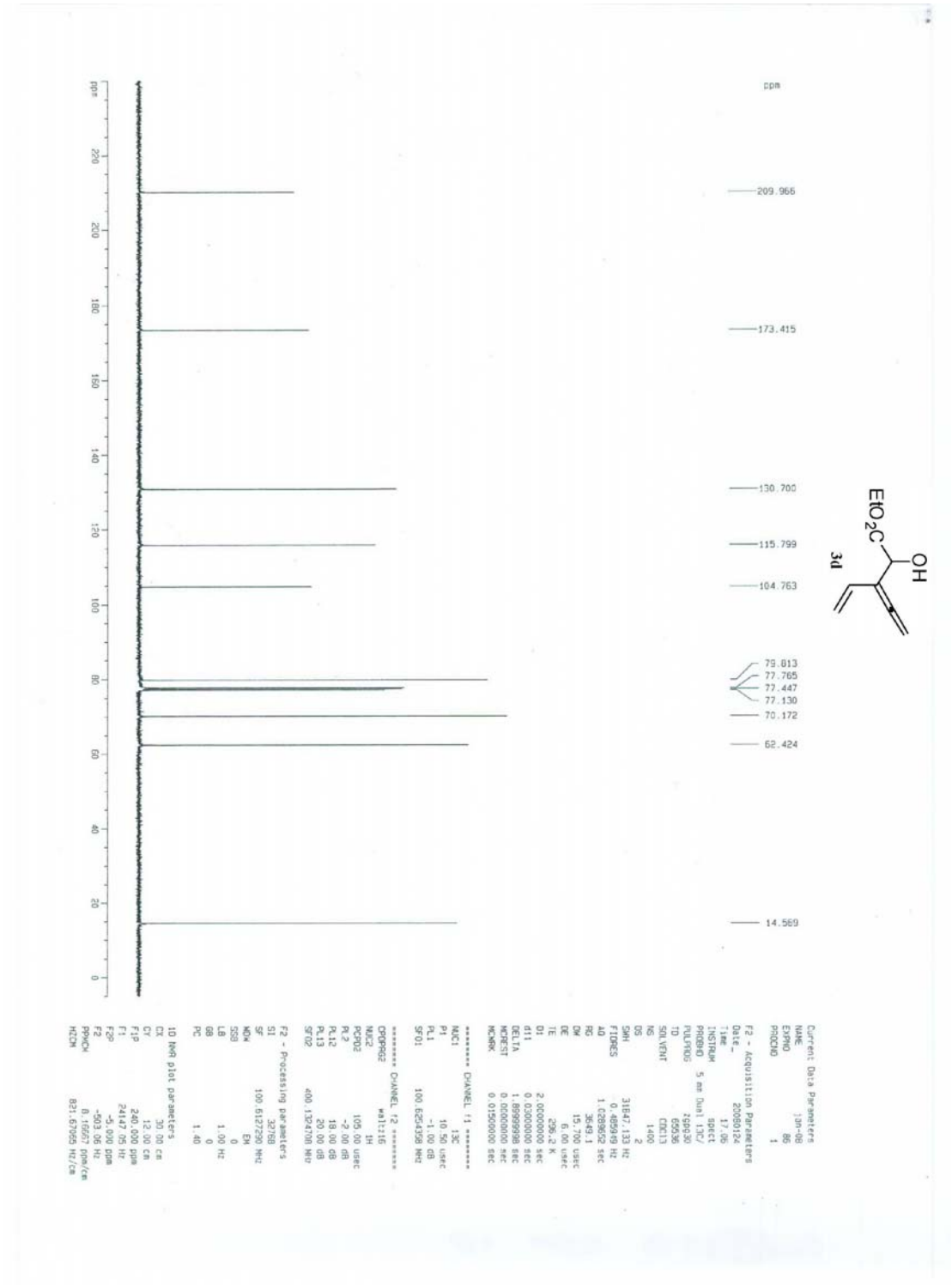
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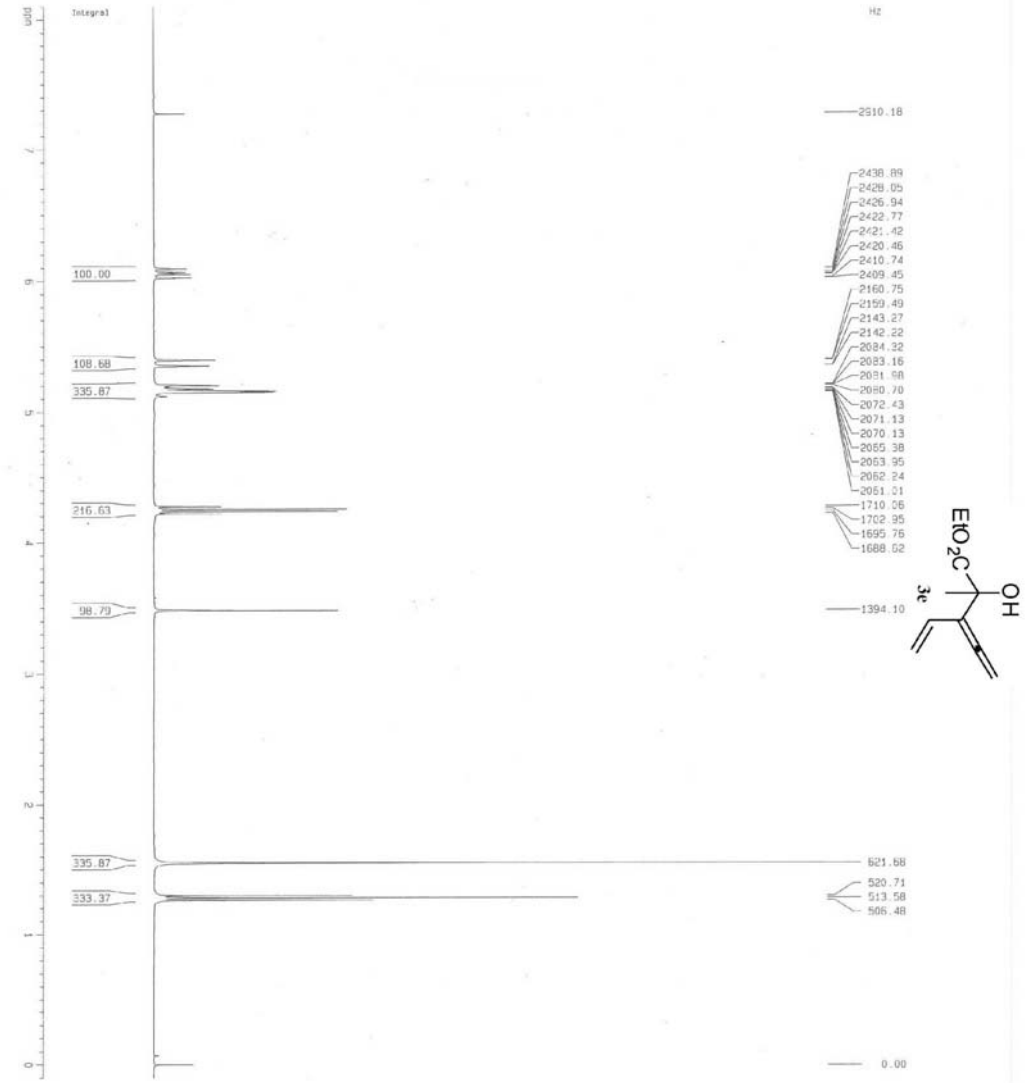

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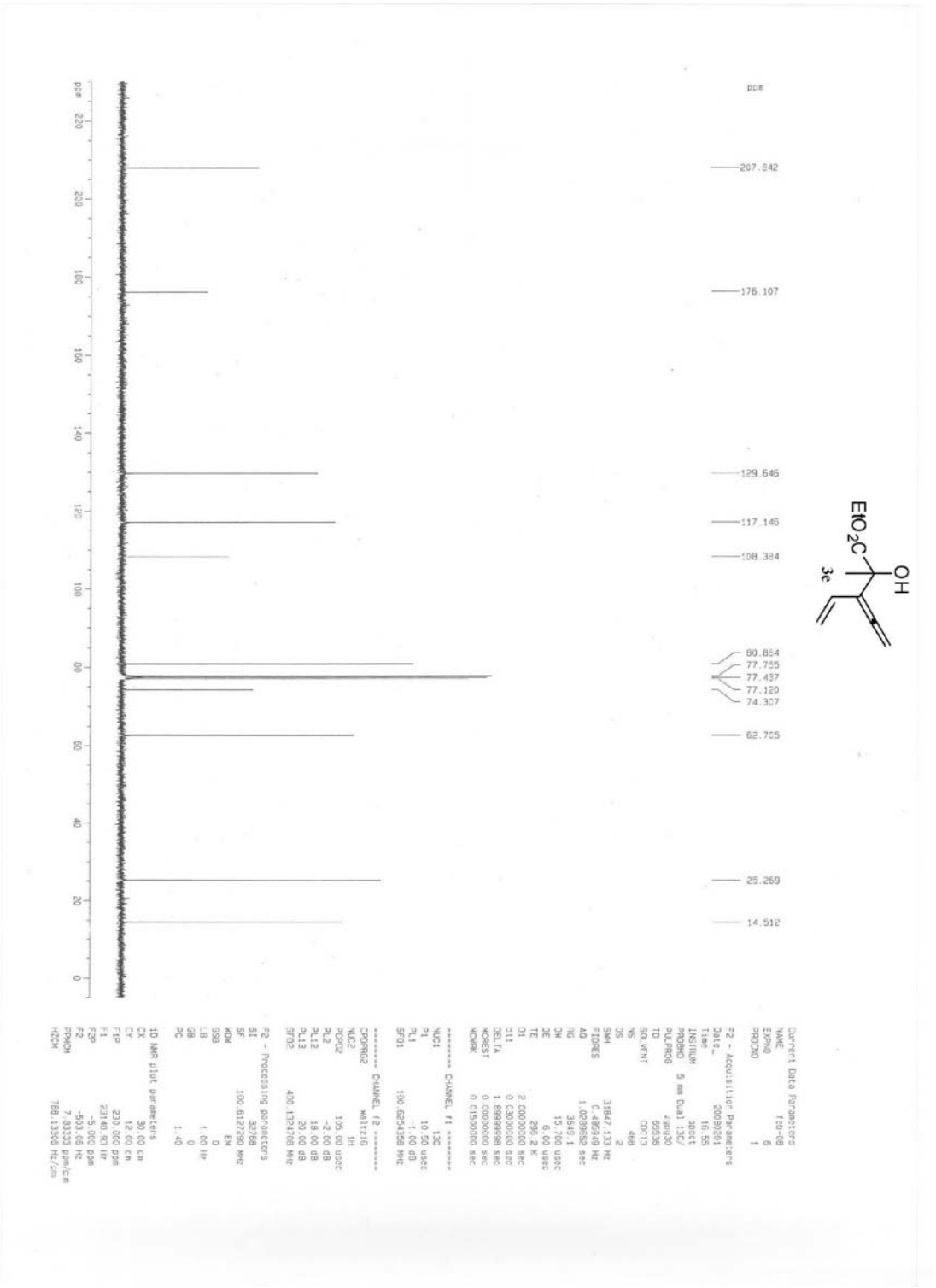
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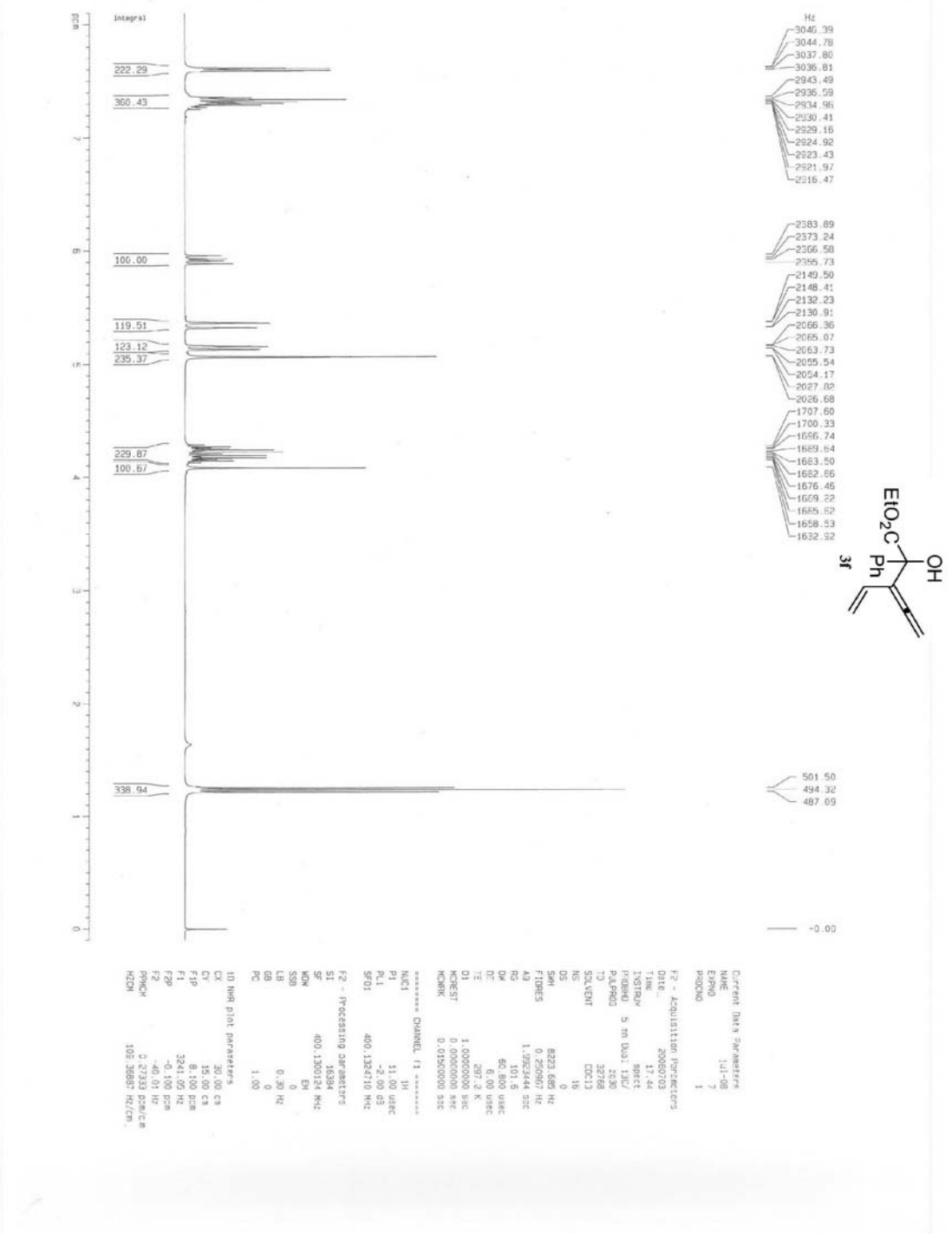
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P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
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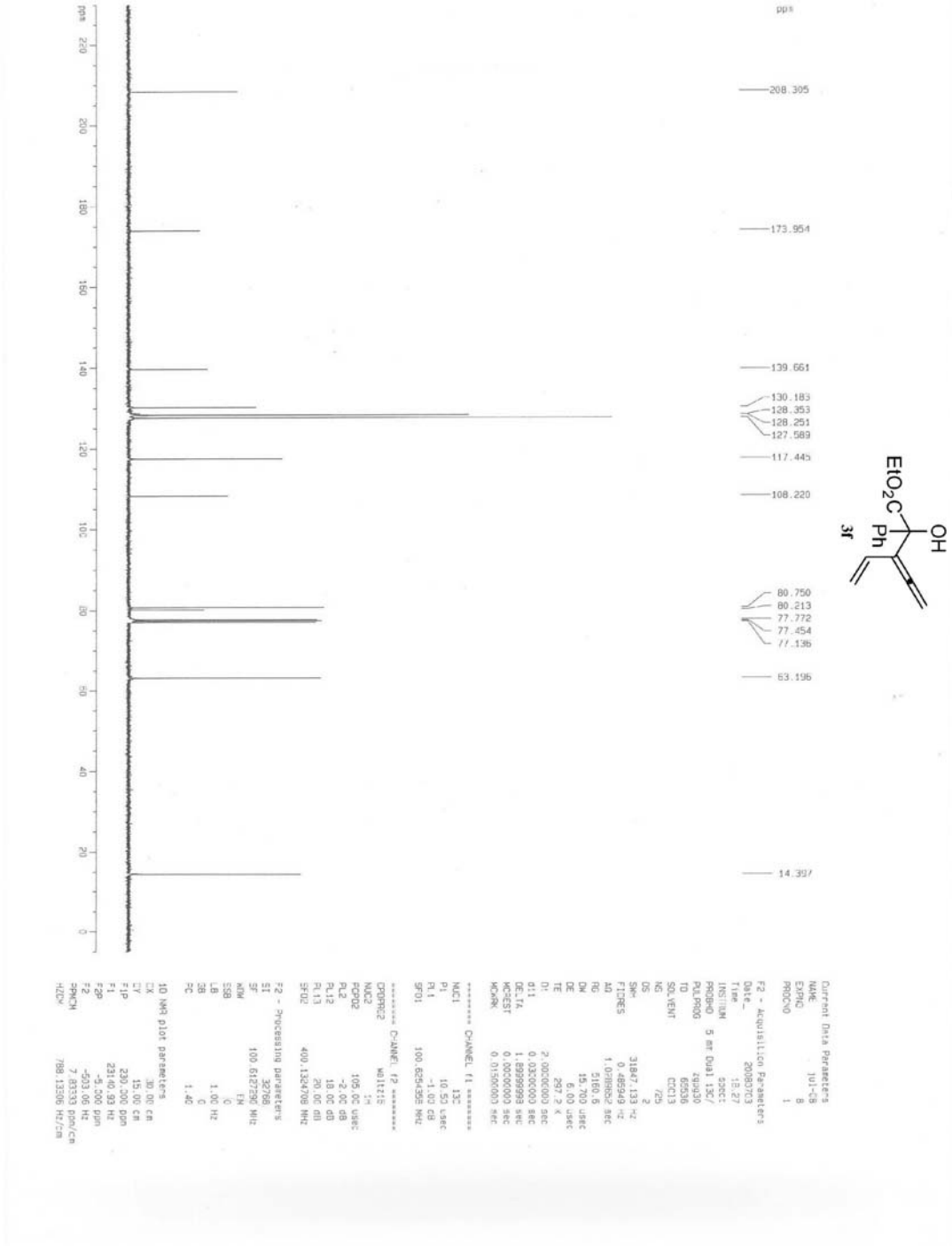
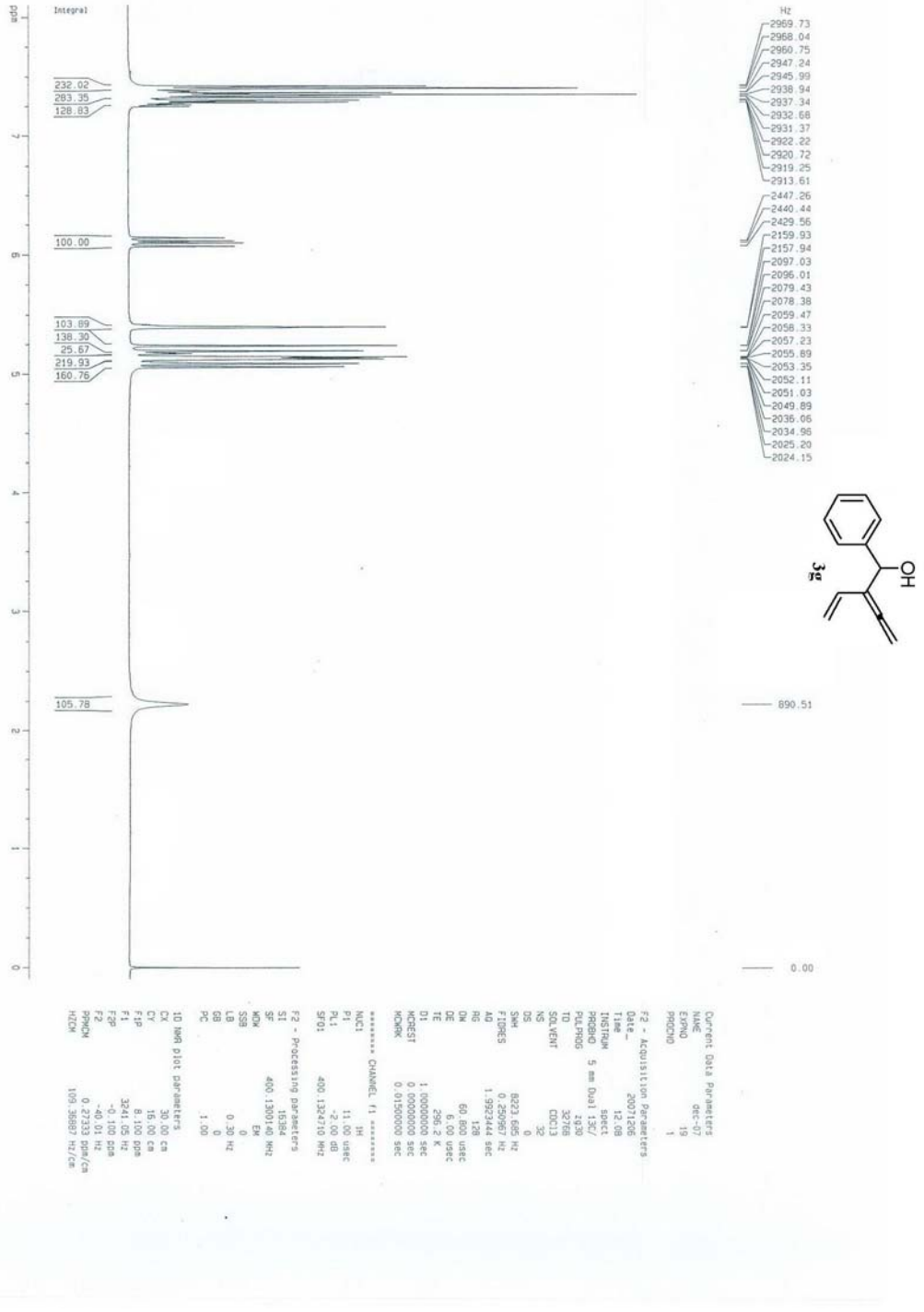
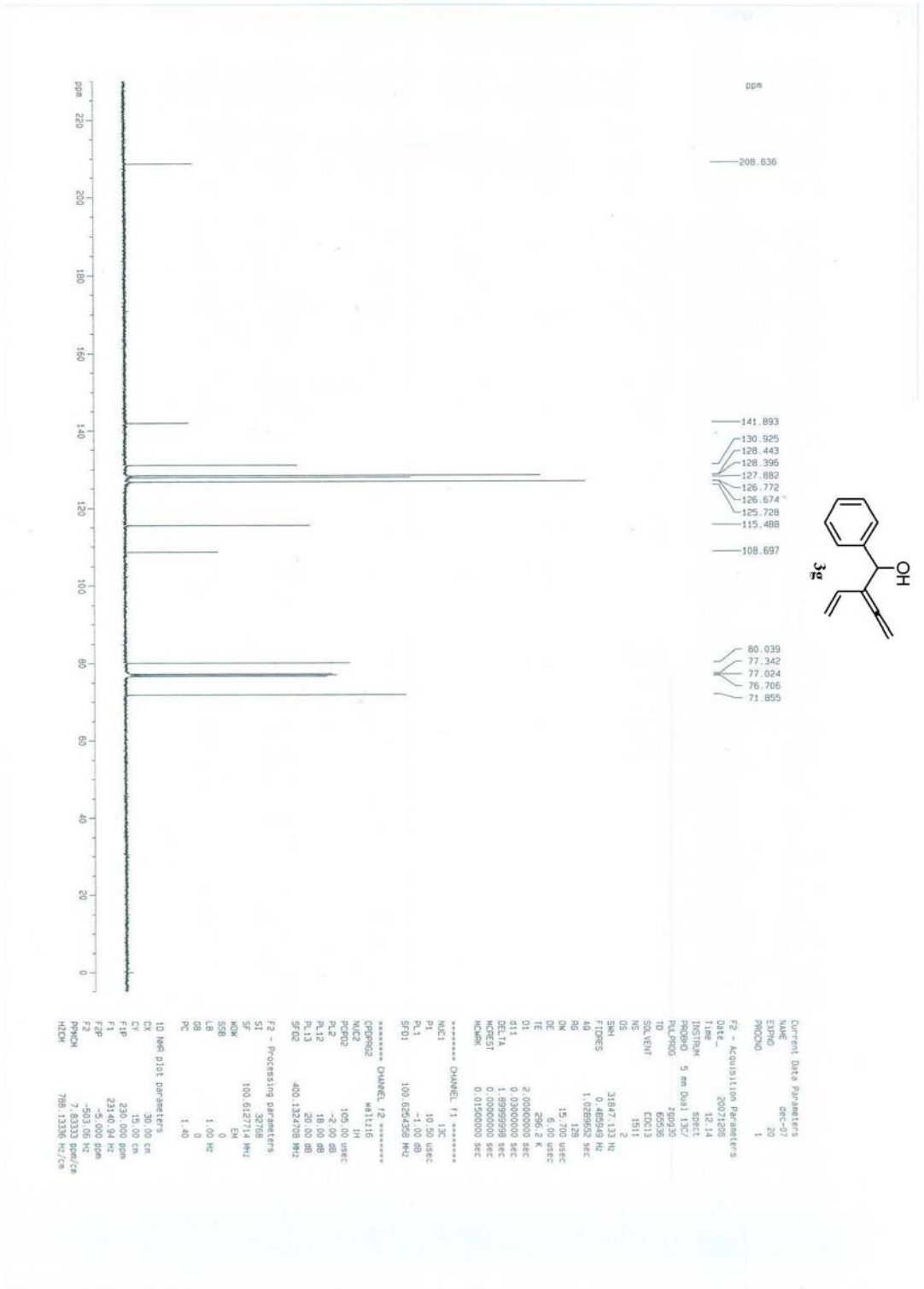
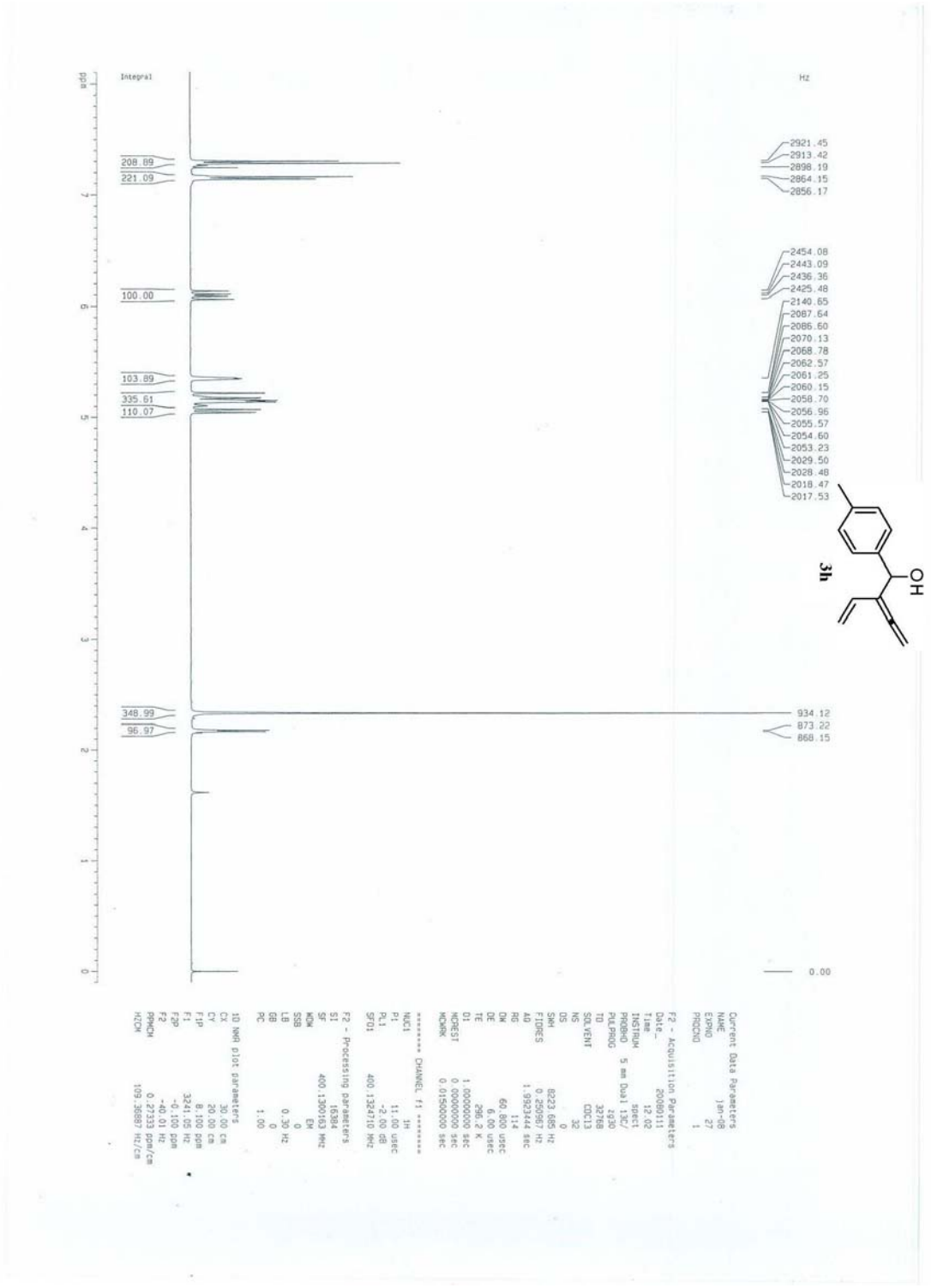
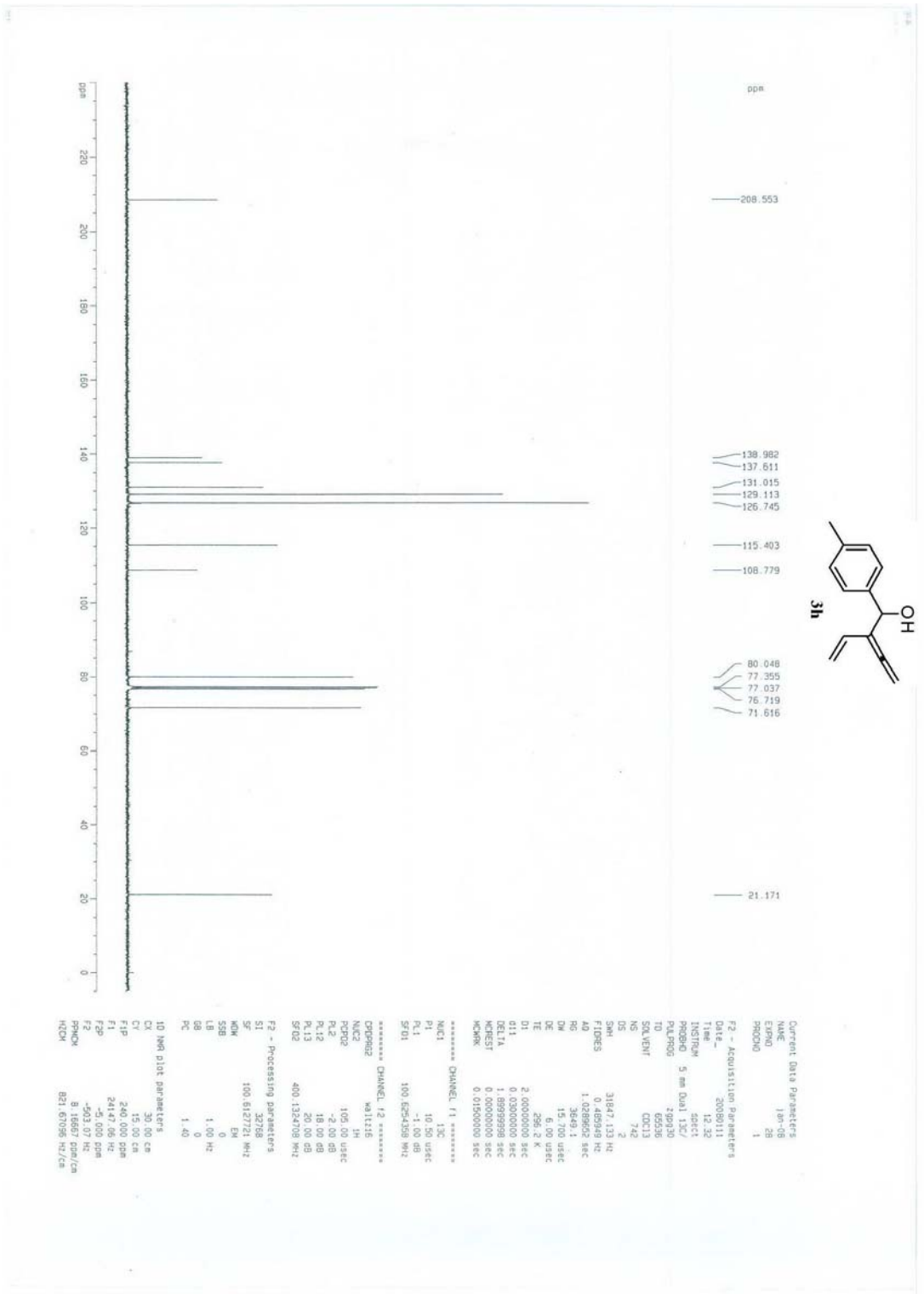
PPMCM
HZCM

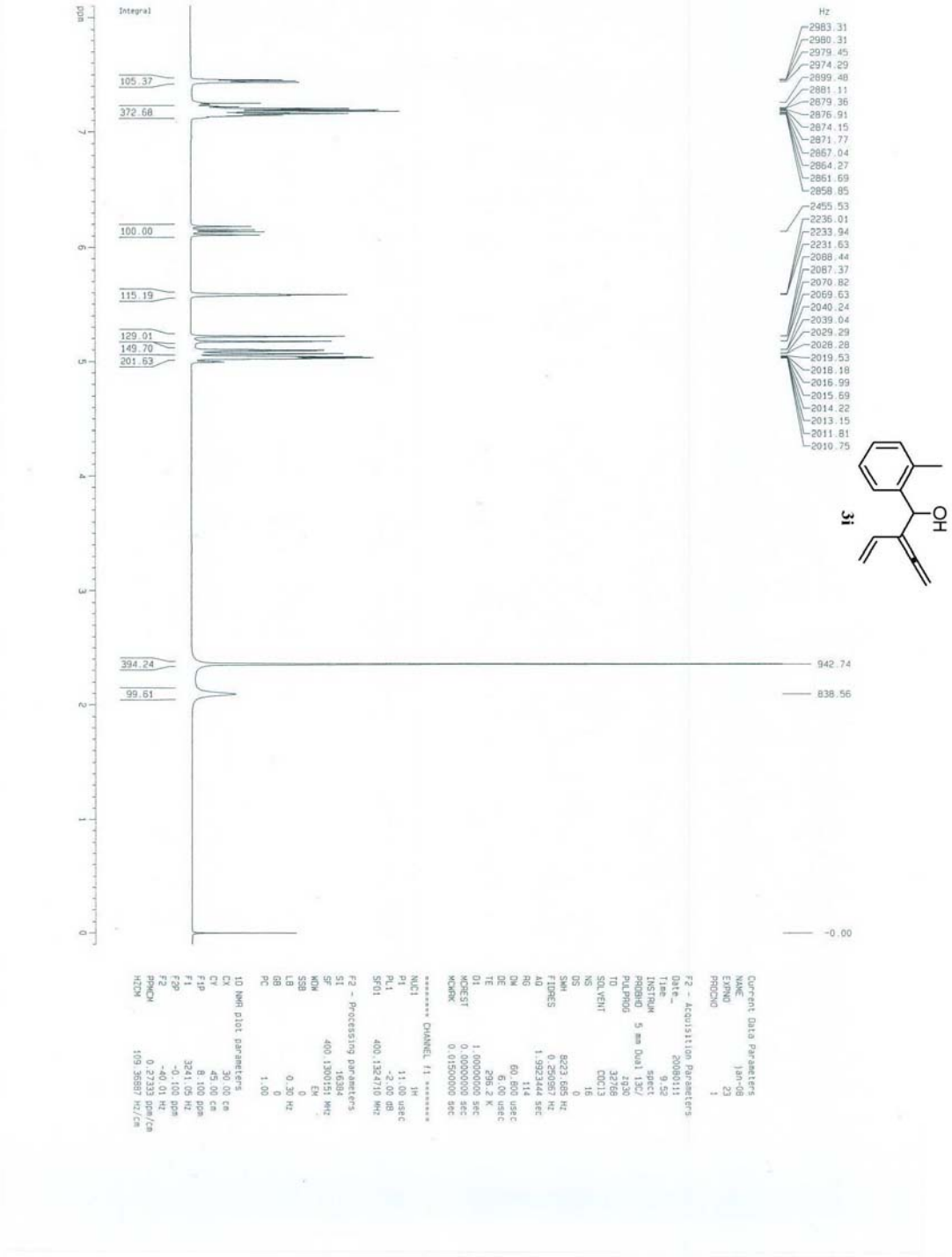

ppm
208.305
173.954
139.661
130.183
128.353
128.251
127.589
117.445
108.220
80.750
80.213
77.772
77.454
77.136
63.196
14.397
OH
EtO2C
Ph
3f


Integral
232.02
283.35
128.83
100.00
103.89
138.30
25.67
219.93
160.76
105.78
Hz
2969.73
2968.04
2960.75
2947.24
2945.99
2938.94
2937.34
2932.66
2931.37
2922.22
2920.72
2919.25
2913.61
2447.26
2440.44
2429.56
2159.93
2157.94
2097.03
2096.01
2079.43
2078.38
2059.47
2058.33
2057.23
2055.89
2053.35
2052.11
2051.03
2049.89
2036.06
2034.96
2025.20
2024.15
890.51
0.00
3ga
OH
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD 5 mm Dual 13C/
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
MCREST
MCWRK
******** CHANNEL f1 ********
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F2P
F1
F2
PPMCM
HZCM


ppm
208.636
141.893
130.925
128.443
128.396
127.882
126.772
126.674
125.728
115.488
108.697
80.039
77.342
77.024
76.706
71.855
OH
3g


Integral
Hz
208.89
221.09
100.00
103.89
335.61
110.07
348.99
96.97
2921.45
2913.42
2898.19
2864.15
2856.17
2454.08
2443.09
2436.36
2425.48
2140.65
2087.64
2086.60
2070.13
2068.78
2062.57
2061.25
2060.15
2058.70
2056.96
2055.57
2054.60
2053.23
2029.50
2028.48
2018.47
2017.53
934.12
873.22
868.15
0.00
OH
3h


ppm
208.553
138.982
137.611
131.015
129.113
126.745
115.403
108.779
80.048
77.355
77.037
76.719
71.616
21.171
3h
OH



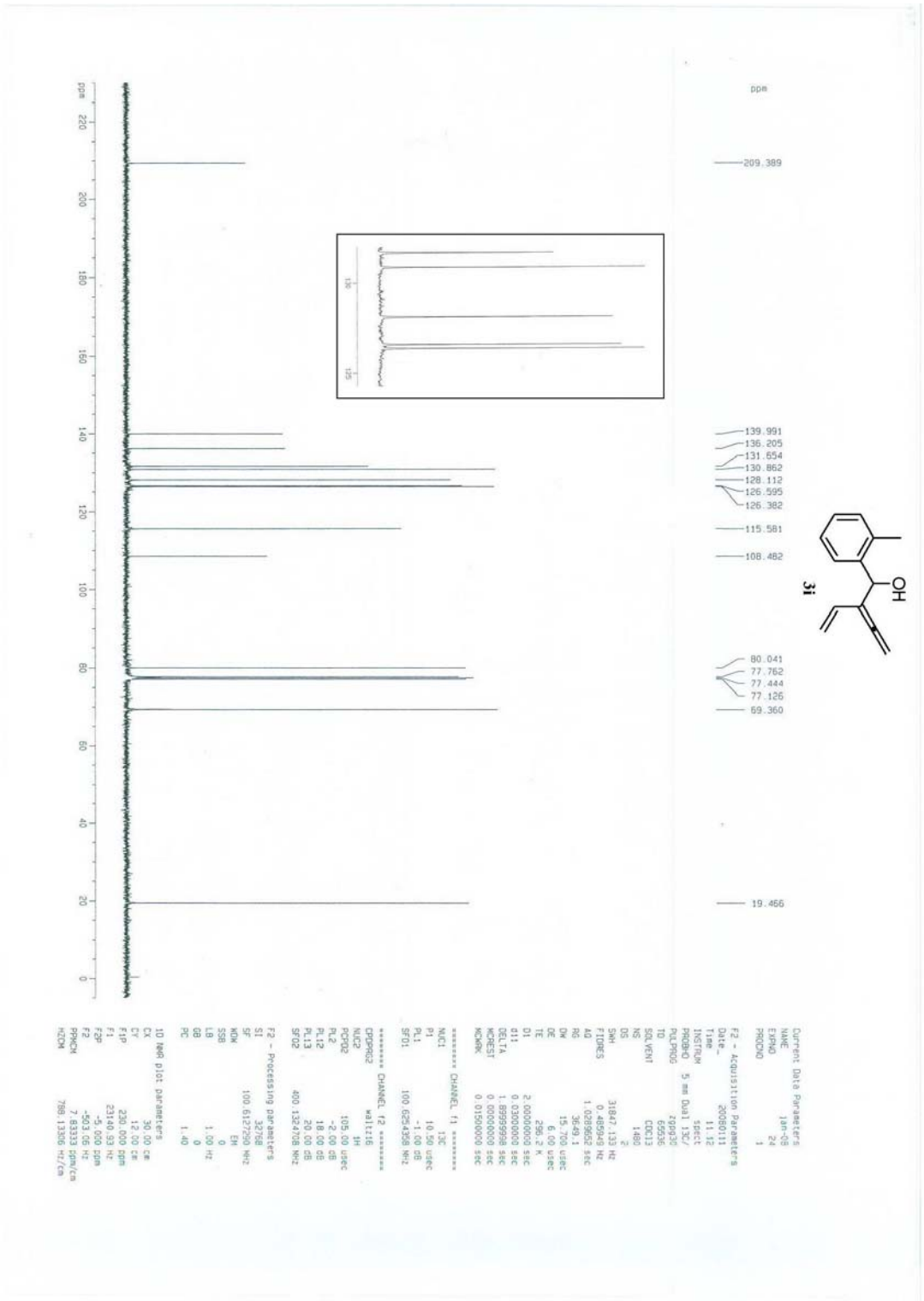
Current Data Parameters
NAME jan-08
EXPNO 23
PROCNO 1

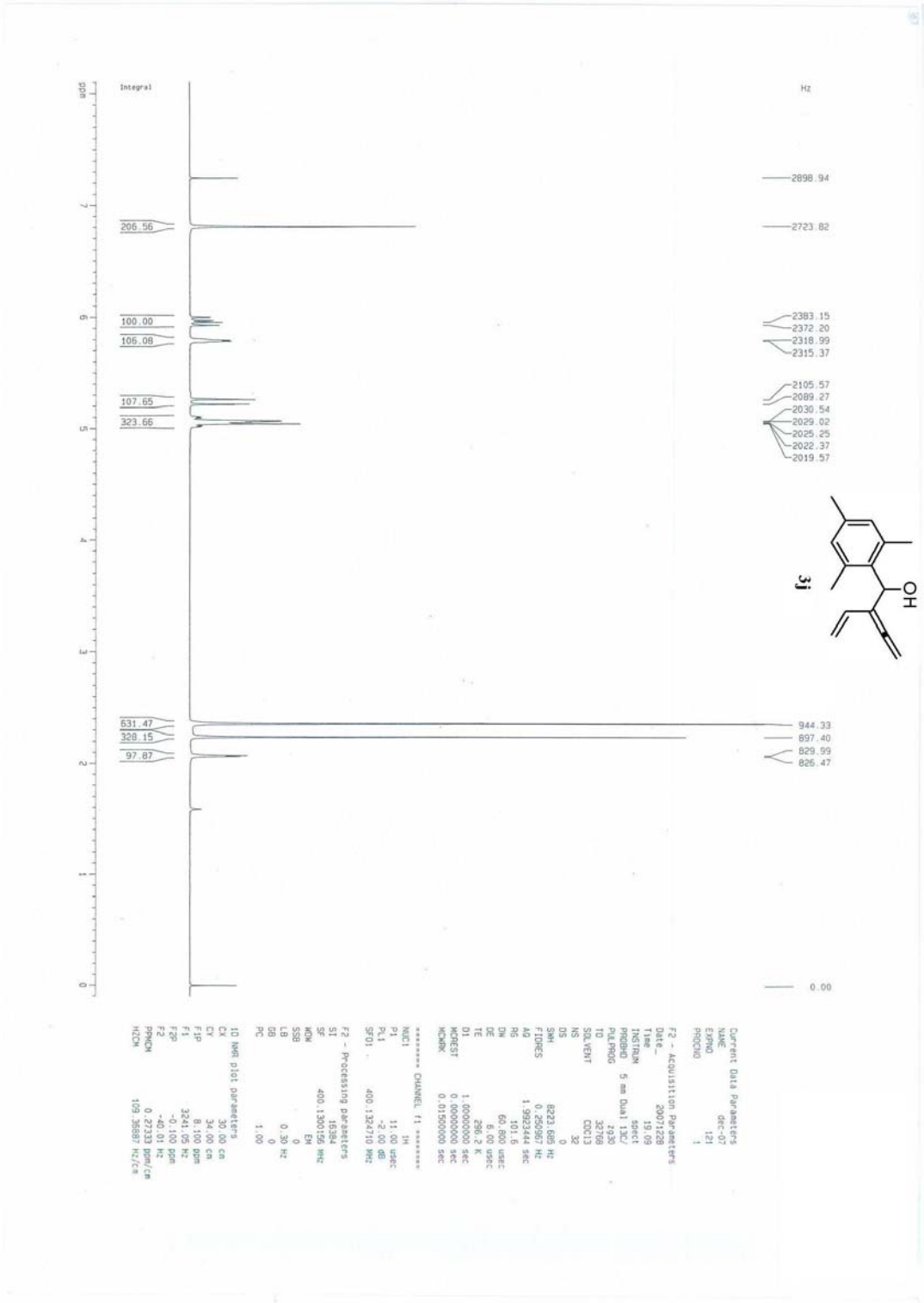
F2 - Acquisition Parameters
Date_ 20080111
Time 9.52
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8823.686 Hz
FIDRES 0.269267 Hz
AQ 1.9923414 sec
RG 114
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

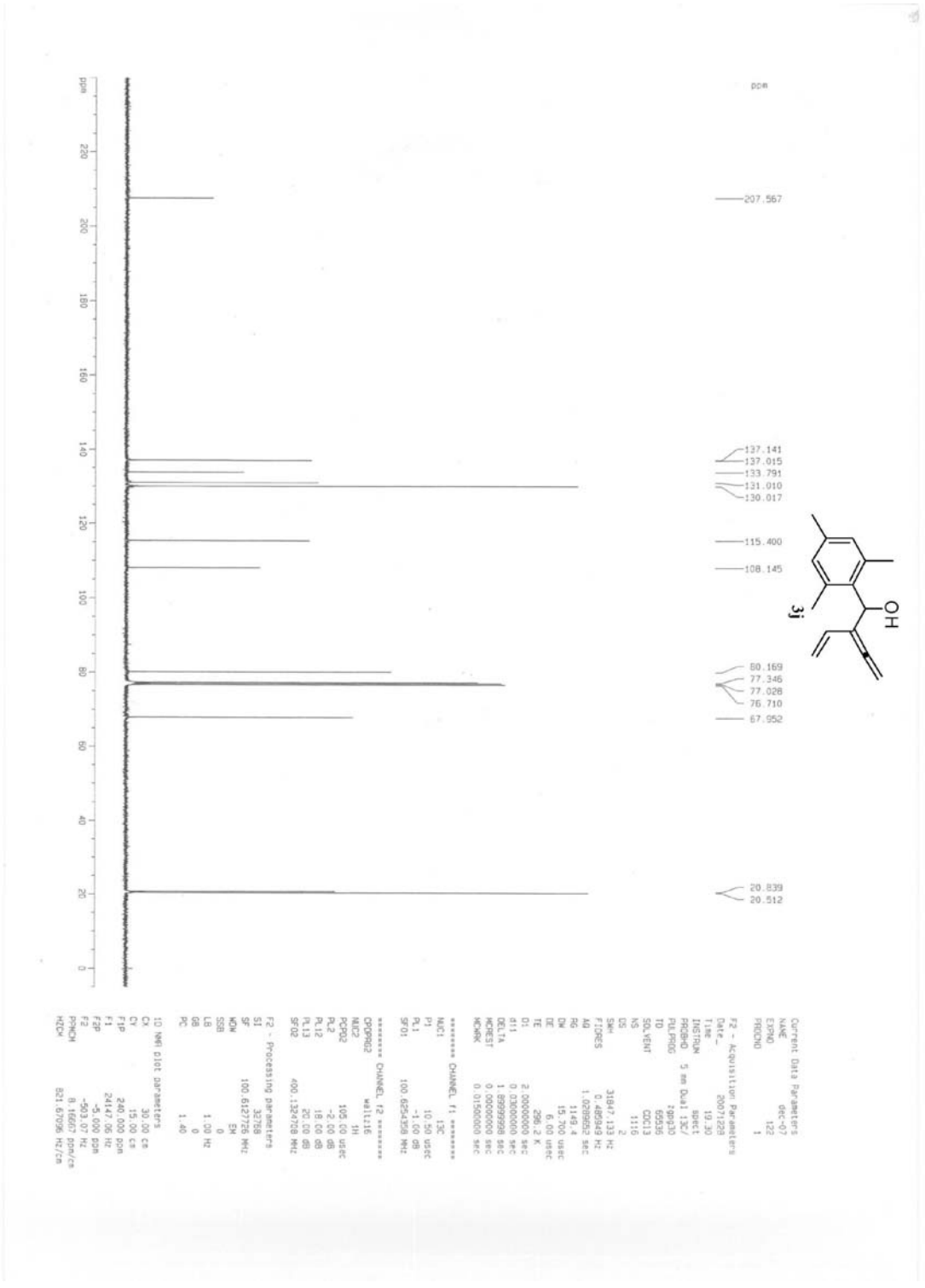
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

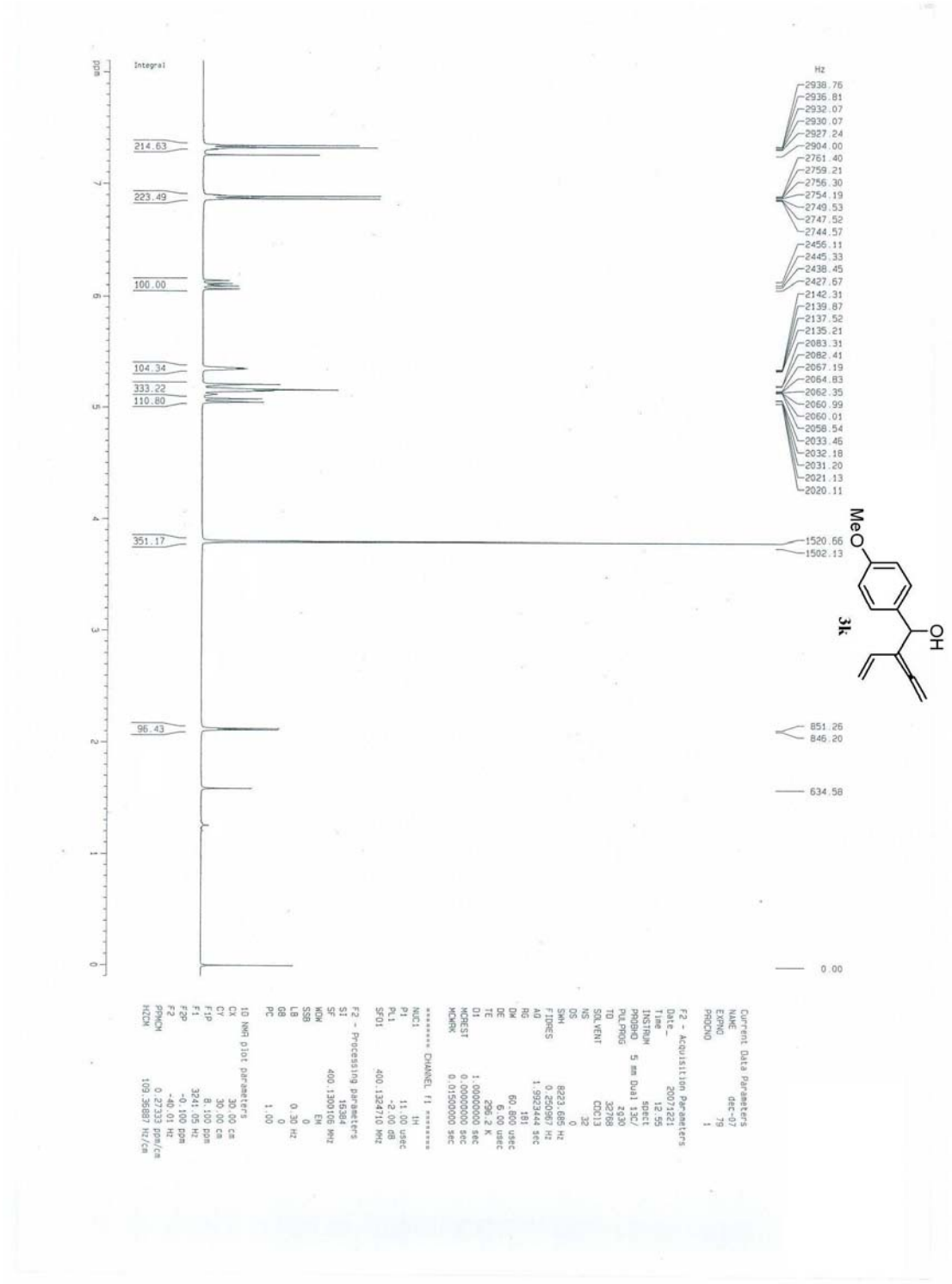
F2 - Processing parameters
SI 16384
SF 400.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

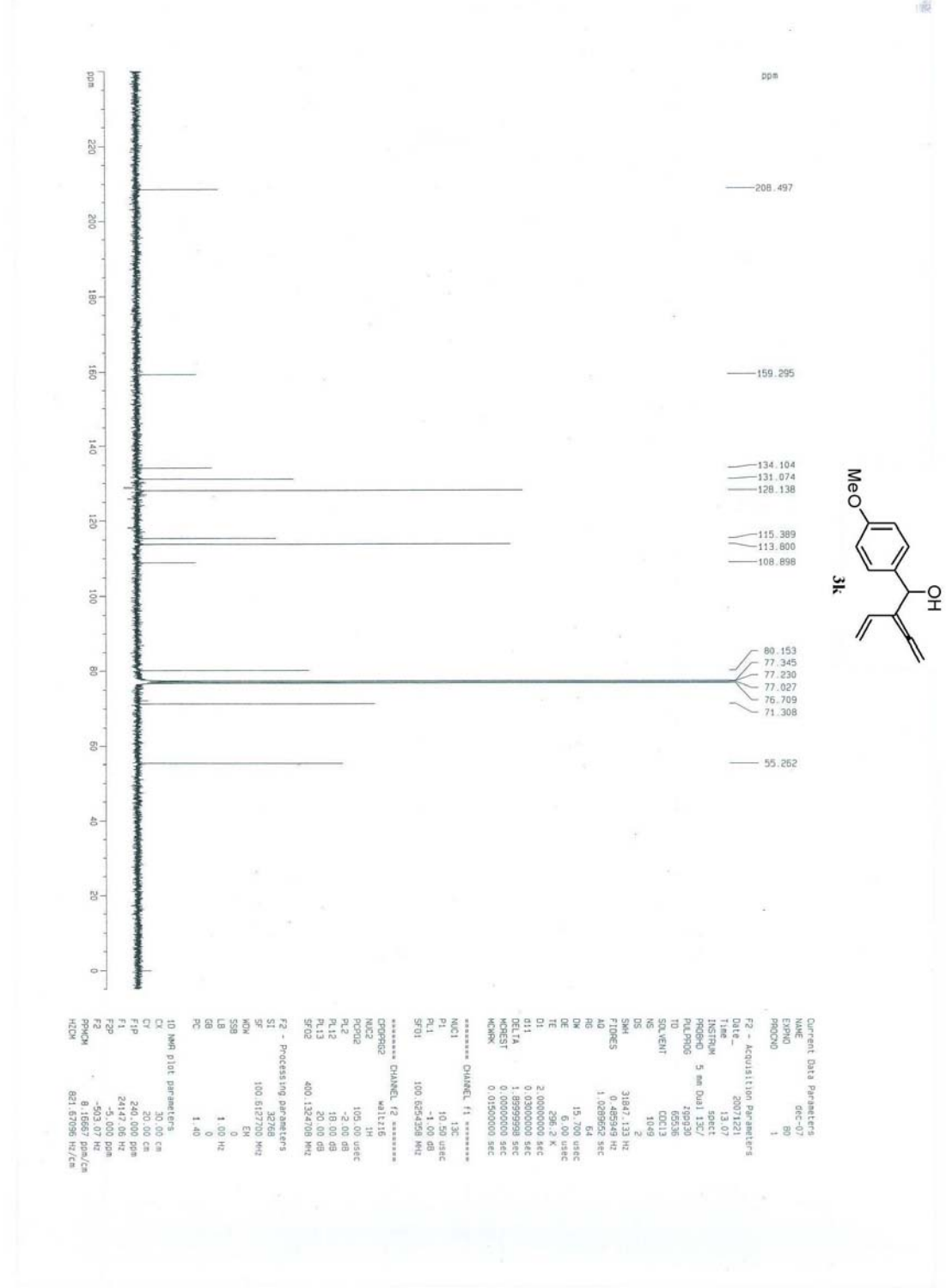
1D NMR plot parameters
CX 30.00 cm
CY 45.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36887 Hz/cm

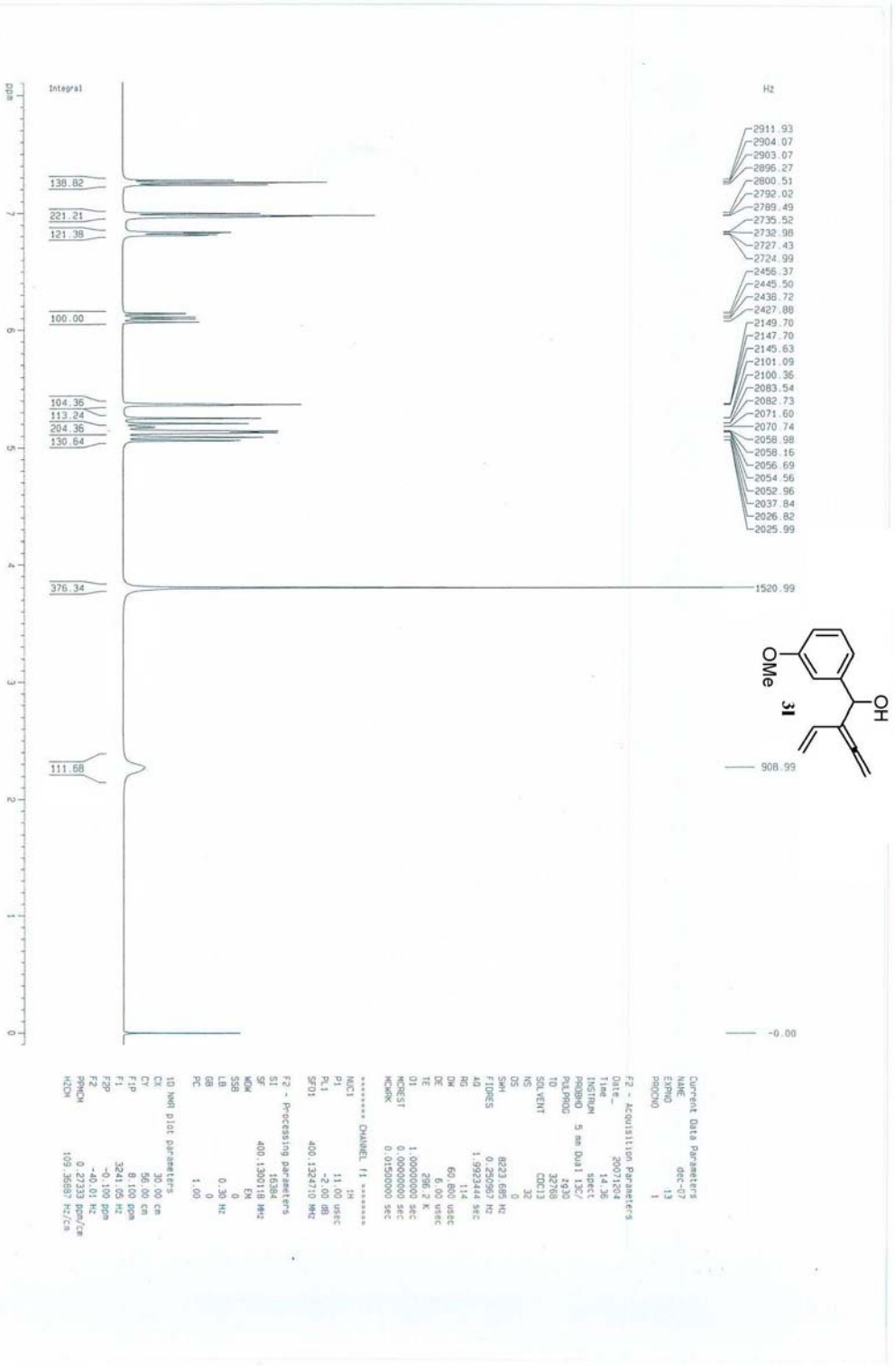

ppm
209.389
139.991
136.205
131.654
130.862
128.112
126.595
126.382
115.581
108.482
80.041
77.762
77.444
77.126
69.360
19.466
OH
3i

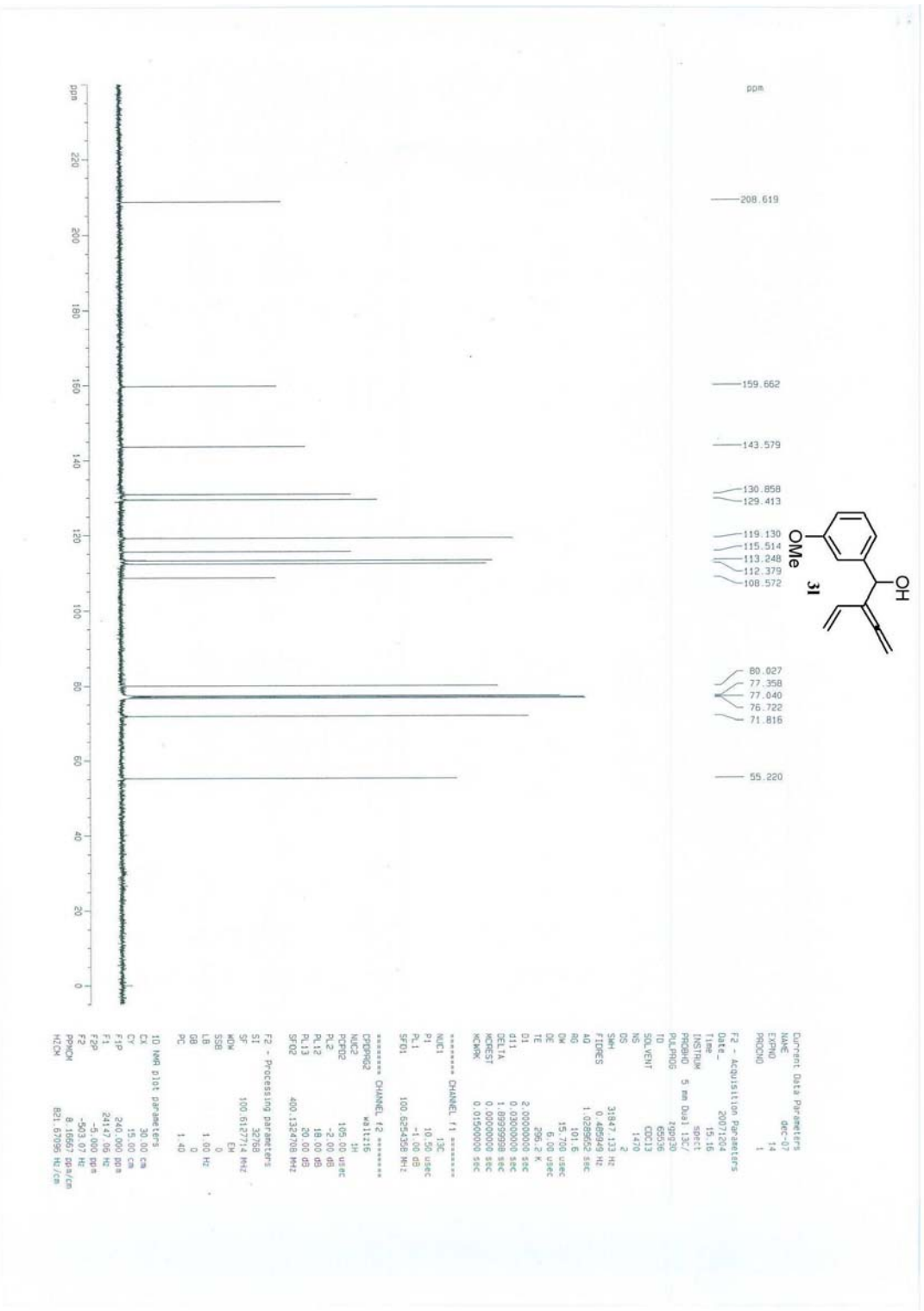

Integral
Hz
206.56
100.00
106.08
107.65
323.66
631.47
328.15
97.87
2898.94
2723.82
2383.15
2372.20
2318.99
2315.37
2105.57
2089.27
2030.54
2029.02
2025.25
2022.37
2019.57
944.33
897.40
829.99
826.47
0.00
3j
OH

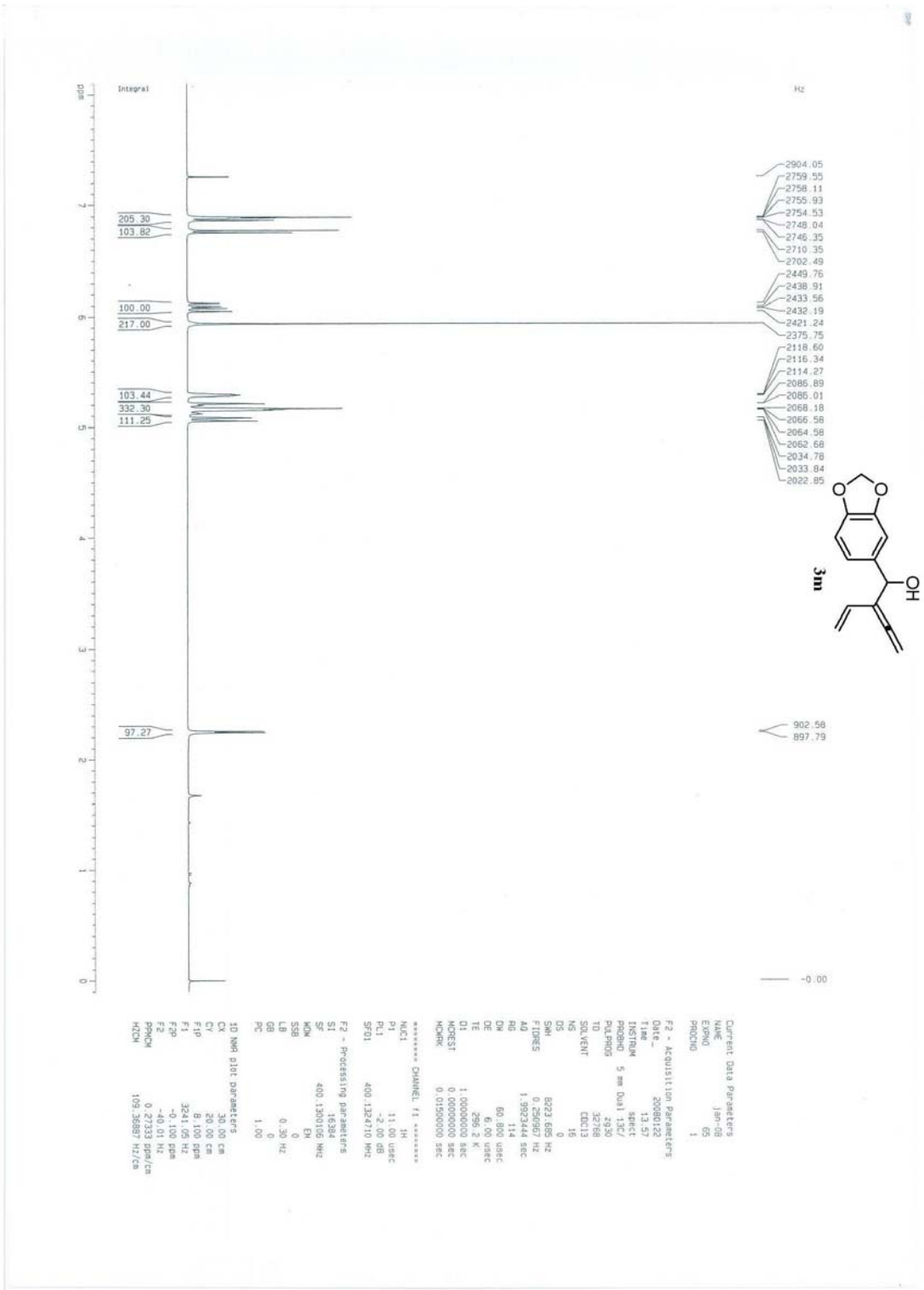

ppm
207.567
137.141
137.015
133.791
131.010
130.017
115.400
108.145
80.169
77.346
77.028
76.710
67.952
20.839
20.512
3j
OH

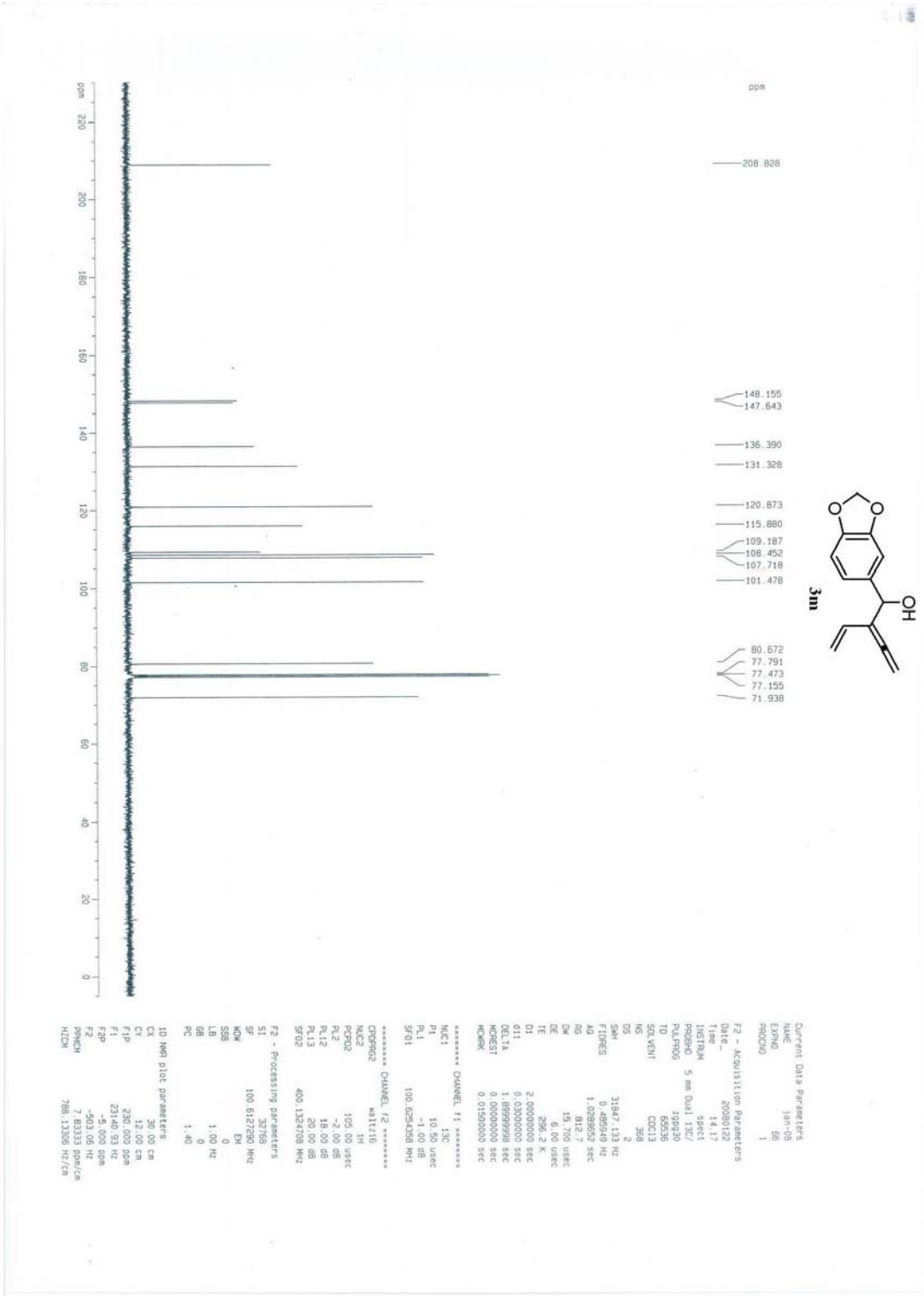

Integral
214.63
223.49
100.00
104.34
333.22
110.80
351.17
96.43
Hz
2938.76
2936.81
2932.07
2930.07
2927.24
2904.00
2761.40
2759.21
2756.30
2754.19
2749.53
2747.52
2744.57
2456.11
2445.33
2438.45
2427.67
2142.31
2139.87
2137.52
2135.21
2083.31
2082.41
2067.19
2064.83
2062.35
2060.99
2060.01
2058.54
2033.46
2032.18
2031.20
2021.13
2020.11
1520.66
1502.13
851.26
846.20
634.58
0.00
MeO
OH
3k
Current Data Parameters
NAME
EXPNO 79
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071221
Time 12.55
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250987 Hz
AQ 1.9923444 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 30.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36687 Hz/cm

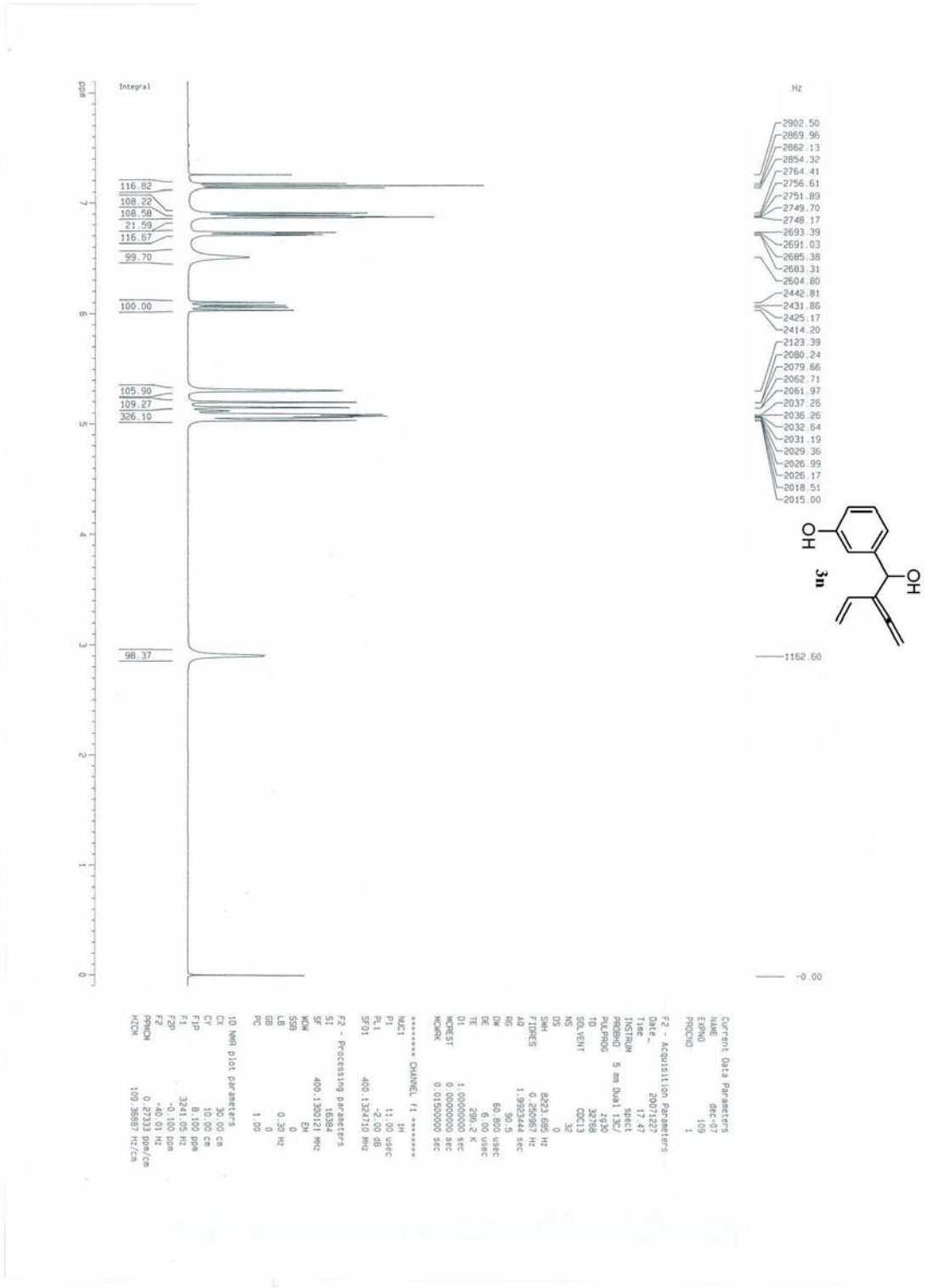

ppm
208.497
159.295
134.104
131.074
128.138
115.389
113.800
108.898
80.153
77.345
77.230
77.027
76.709
71.308
55.262
MeO
OH
3k

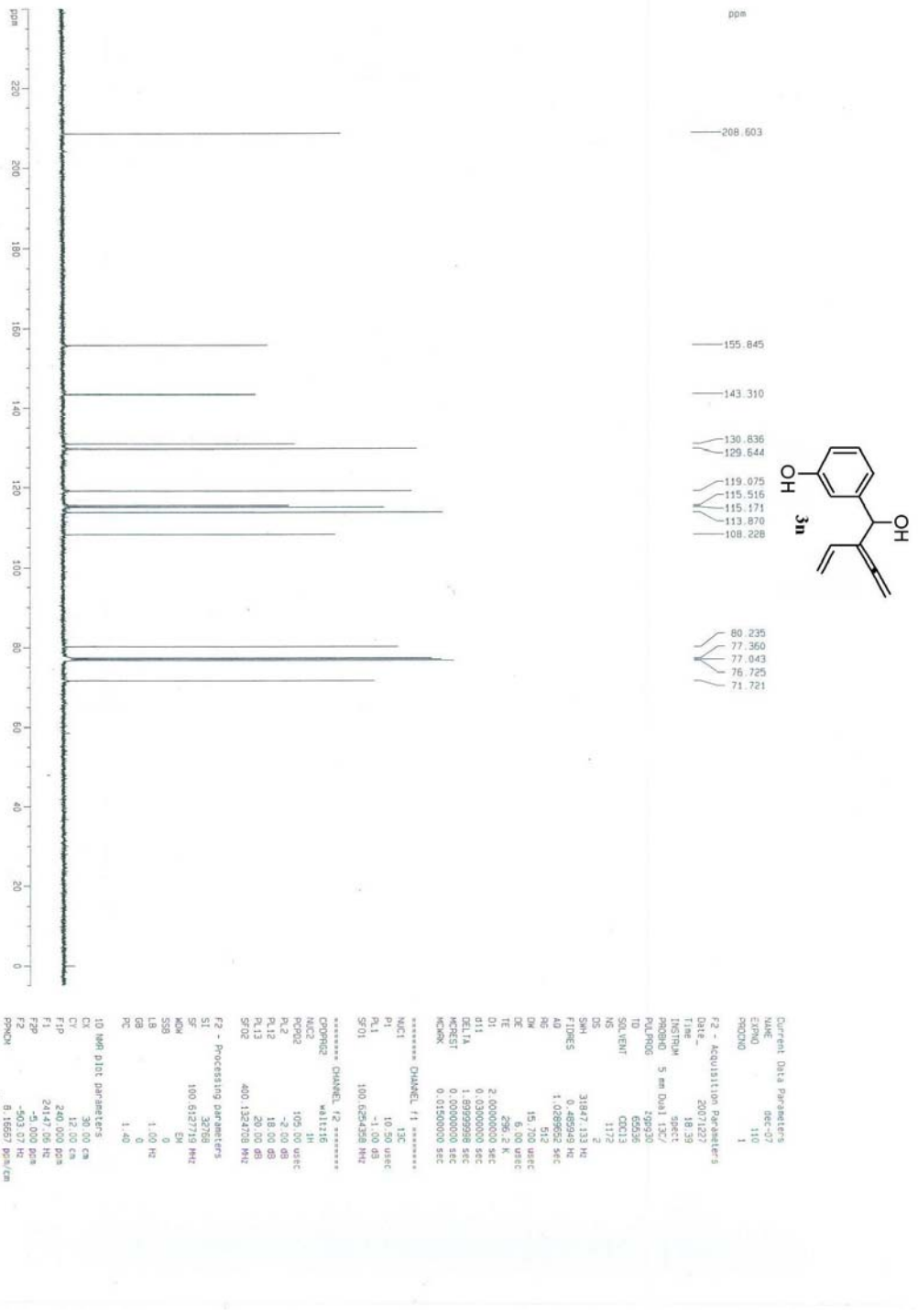

Integral
Hz
2911.93
2904.07
2903.07
2896.27
2800.51
2792.02
2789.49
2735.52
2732.98
2727.43
2724.99
2456.37
2445.50
2438.72
2427.88
2149.70
2147.70
2145.63
2101.09
2100.36
2083.54
2082.73
2071.60
2070.74
2058.98
2058.16
2056.69
2054.56
2052.96
2037.84
2026.82
2025.99
138.82
221.21
121.38
100.00
104.36
113.24
204.36
130.64
376.34
111.68
1520.99
908.99
-0.00
OMe
OH
31
F2 - Acquisition Parameters
Current Data Parameters

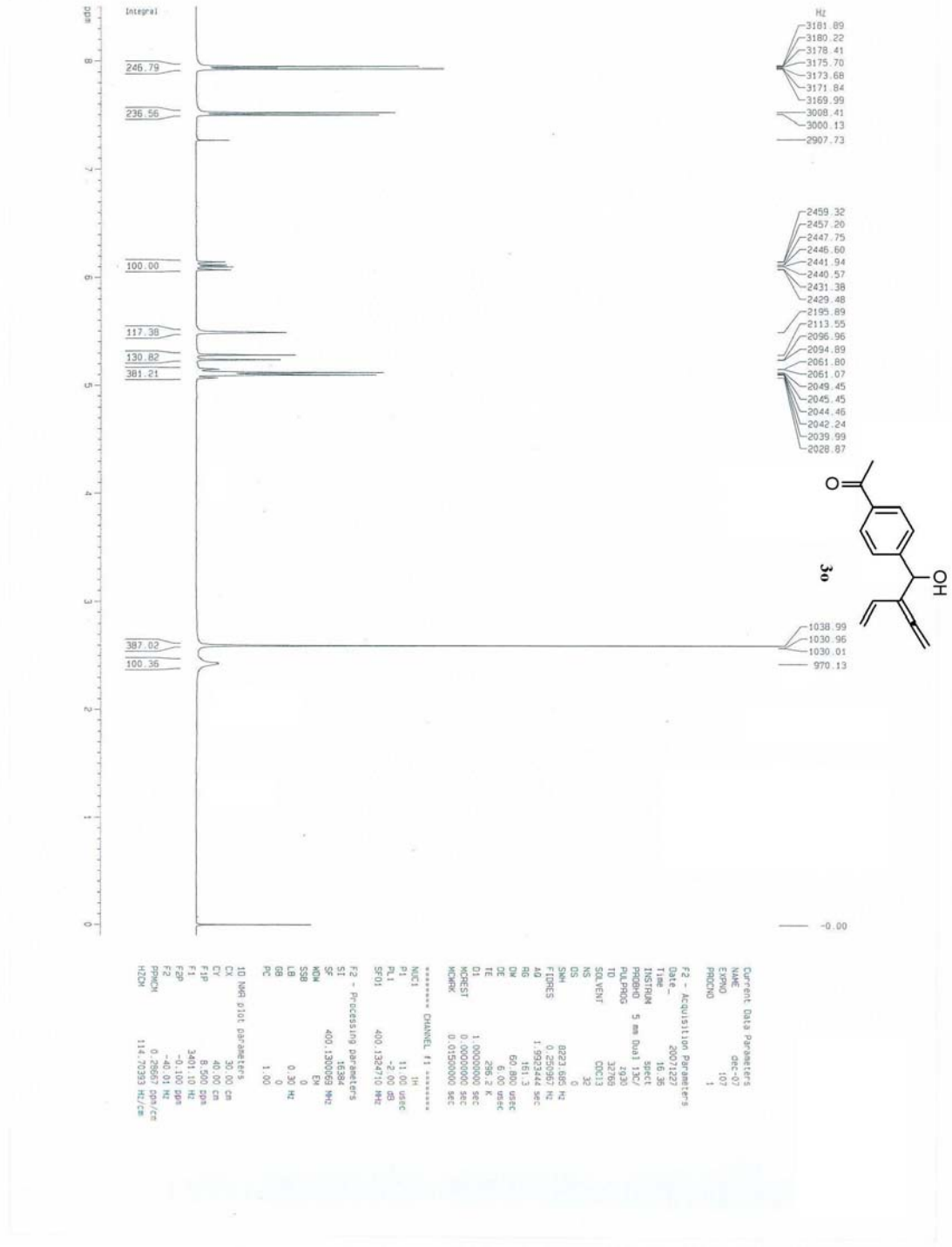

ppm
208.619
159.662
143.579
130.858
129.413
119.130
115.514
113.248
112.379
108.572
80.027
77.358
77.040
76.722
71.816
55.220
OMe
OH
31
Current Data Parameters
NAME qec-07
EXPNO 14
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071204
Time 15.16
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1470
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 101.6
DW 15.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324708 MHz
F2 - Processing parameters
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 240.000 ppm
F1 24147.06 Hz
F2P -5.000 ppm
F2 -503.07 Hz
PPMCM 8.16667 ppm/cm
HZCM 821.67096 Hz/cm

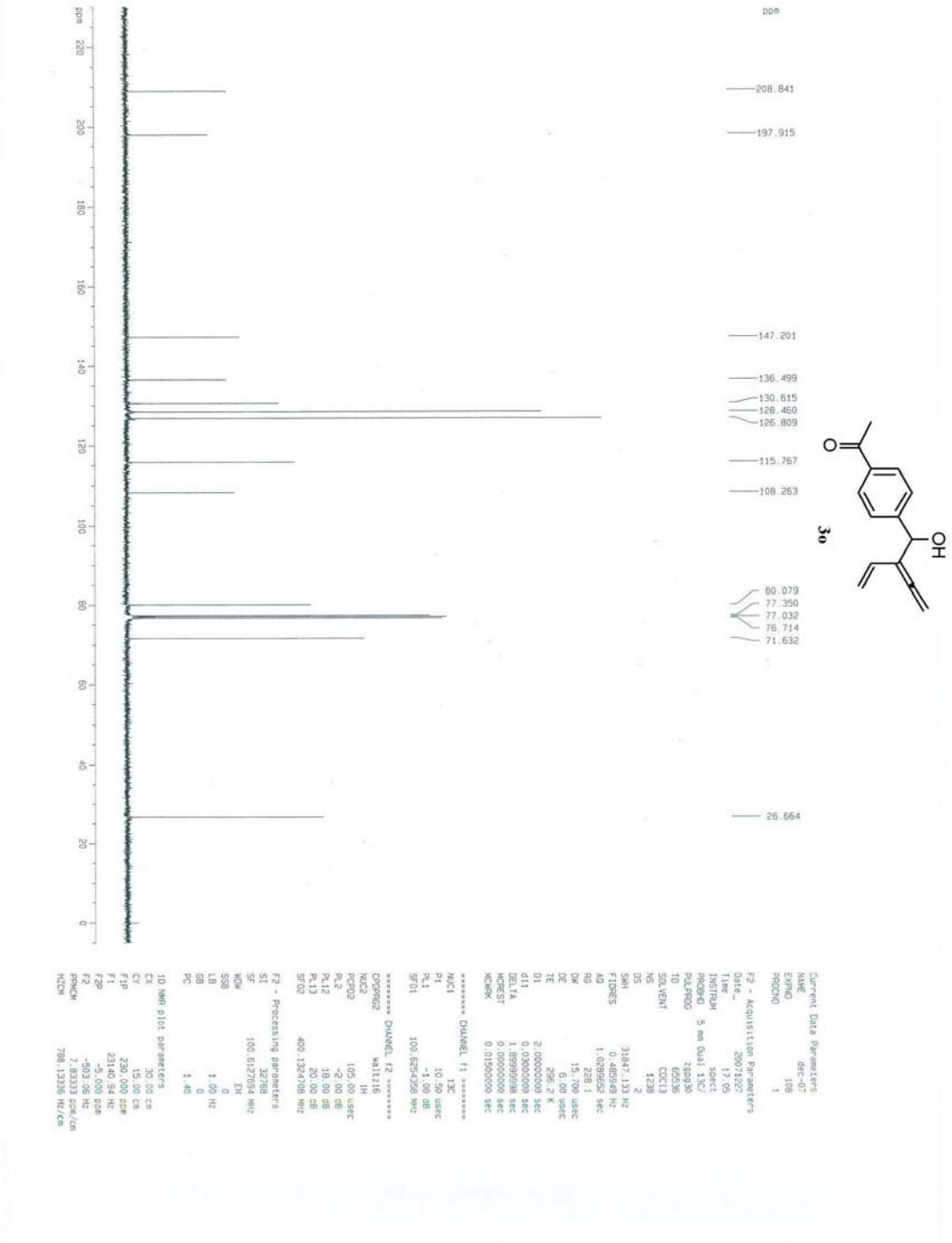

Integral
Hz
ppm
205.30
103.82
100.00
217.00
103.44
332.30
111.25
97.27
2904.05
2759.55
2758.11
2755.93
2754.53
2748.04
2746.35
2710.35
2702.49
2449.76
2438.91
2433.56
2432.19
2421.24
2375.75
2118.60
2116.34
2114.27
2086.89
2086.01
2068.18
2066.58
2064.58
2062.68
2034.78
2033.84
2022.85
902.58
897.79
-0.00
3m
OH
Current Data Parameters
NAME jan-08
EXPNO 65
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080122
Time 13.57
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 114
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
******** CHANNEL f1 ********
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36687 Hz/cm

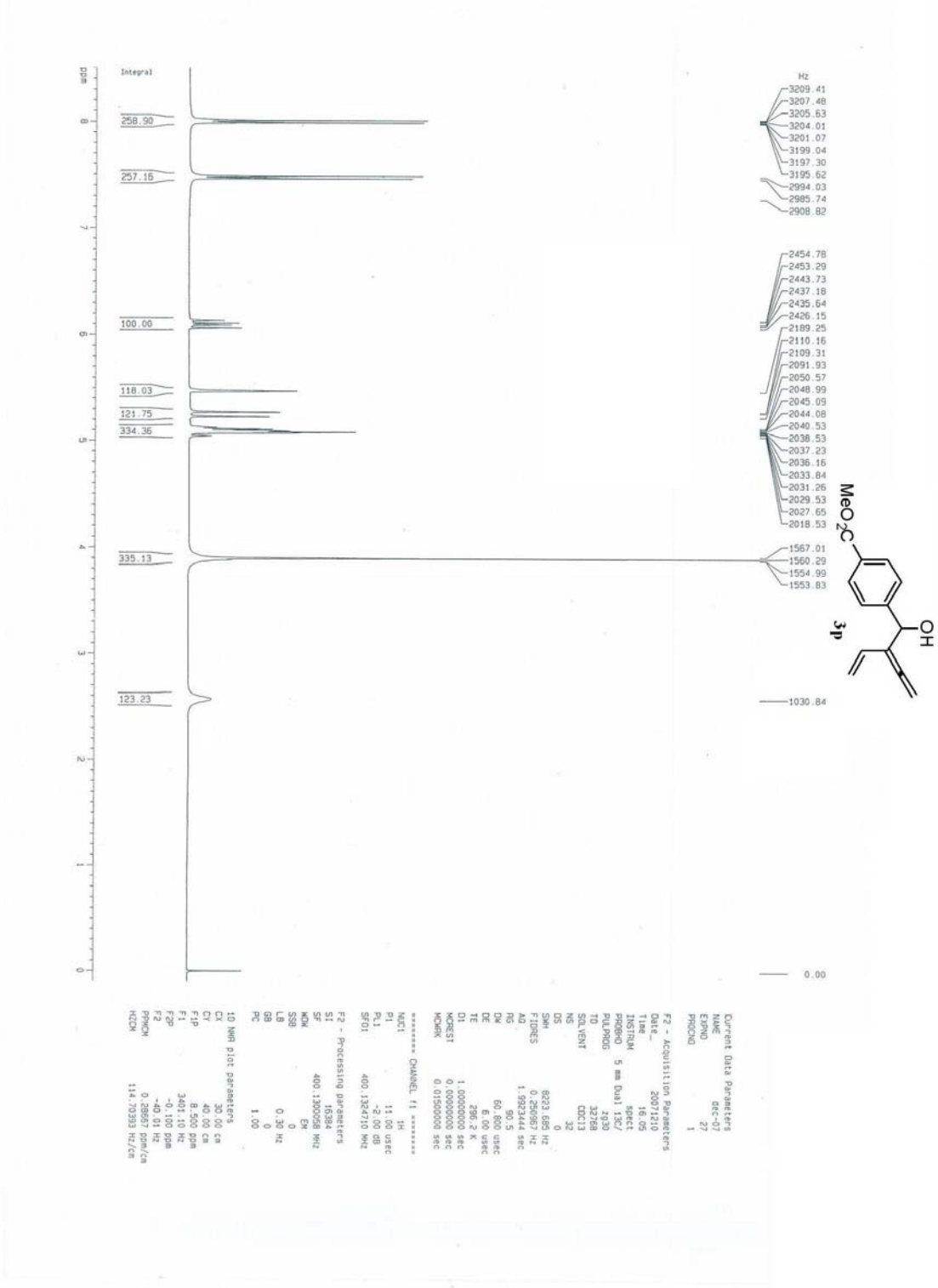

ppm
208.828
148.155
147.643
136.390
131.326
120.873
115.880
109.187
108.452
107.718
101.478
80.672
77.791
77.473
77.155
71.938
3m
OH
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
DELTA
MCREST
MCWRK
======== CHANNEL f1 ========
NUC1
P1
PL1
SF01
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SF02
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM

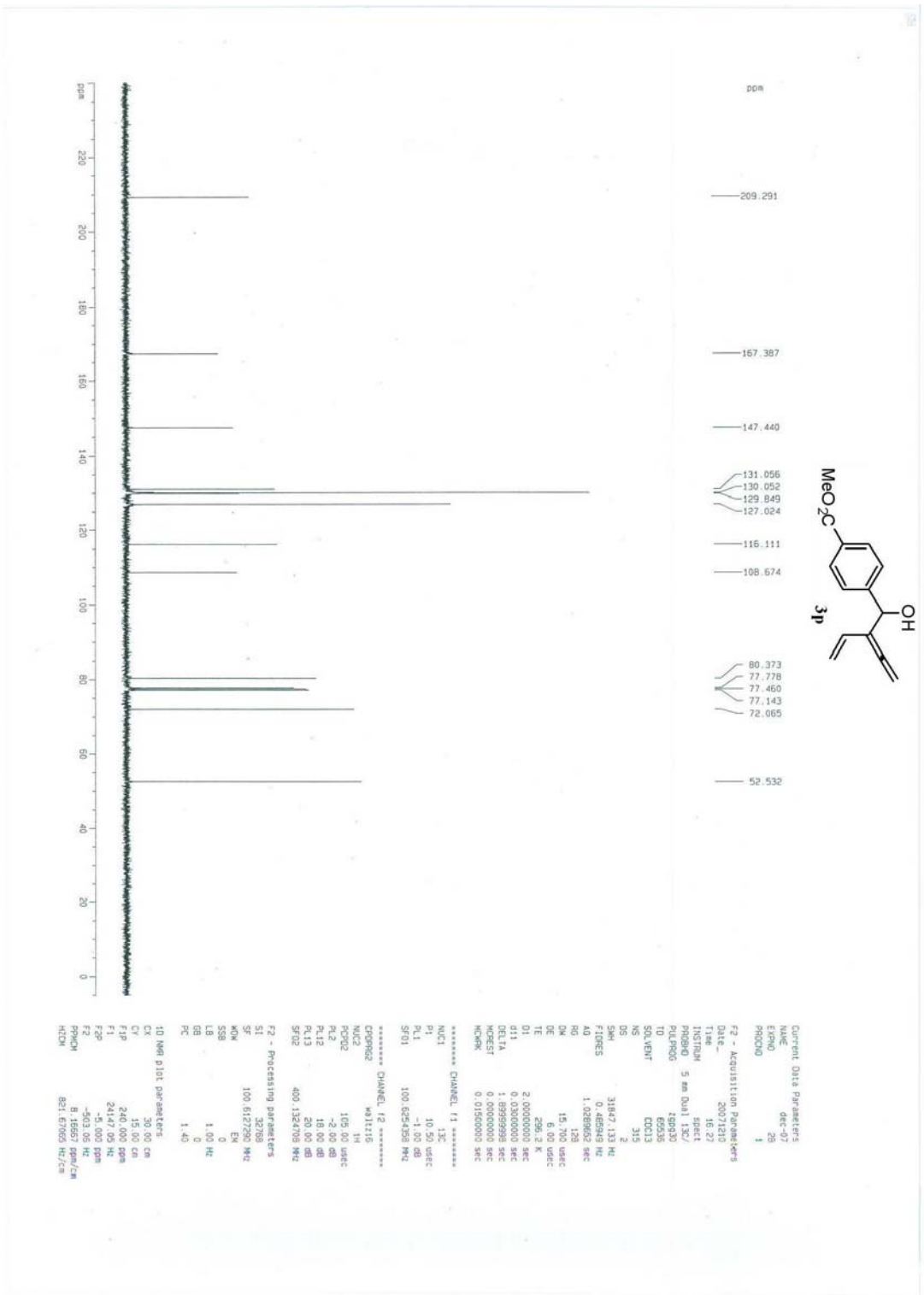

Integral
116.82
108.22
108.58
21.59
116.67
99.70
100.00
105.90
109.27
326.10
98.37
Hz
2902.50
2869.96
2862.13
2854.32
2764.41
2756.61
2751.89
2749.70
2748.17
2693.39
2691.03
2685.38
2683.31
2604.80
2442.81
2431.86
2425.17
2414.20
2123.39
2080.24
2079.66
2062.71
2061.97
2037.26
2036.26
2032.64
2031.19
2029.36
2026.99
2026.17
2018.51
2015.00
OH
OH
3n
1162.60
-0.00
Current Data Parameters
NAME dec-07
EXPNO 109
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071227
Time 17.47
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8823.695 Hz
FIDRES 0.269967 Hz
AQ 1.9923444 sec
RG 90.5
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
D11 0.00000000 sec
MCREST 0.01500000 sec
MCWRK
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 10.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36887 Hz/cm

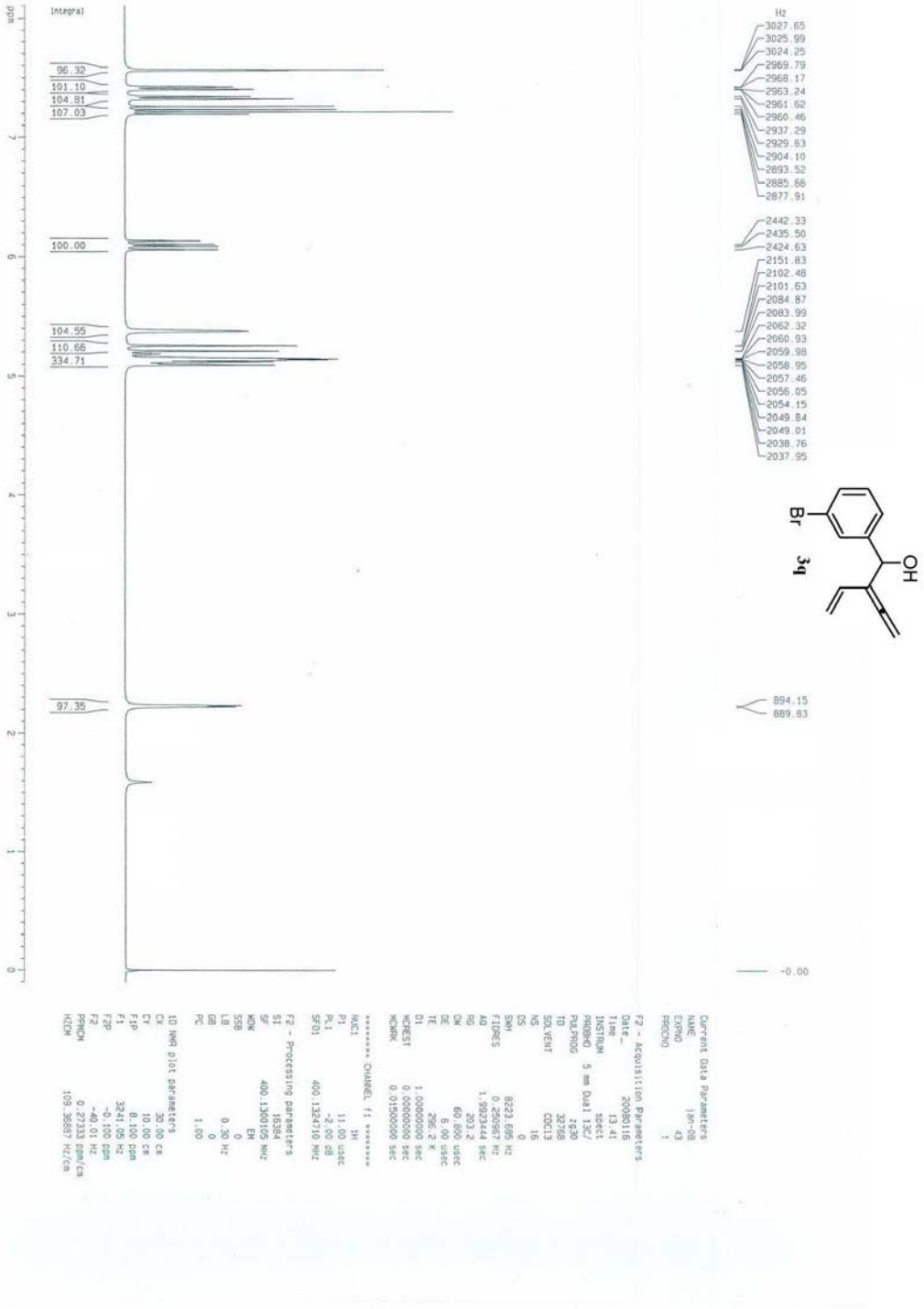

ppm
208.603
155.845
143.310
130.836
129.644
119.075
115.516
115.171
113.870
108.228
80.235
77.360
77.043
76.725
71.721
OH
OH
3n

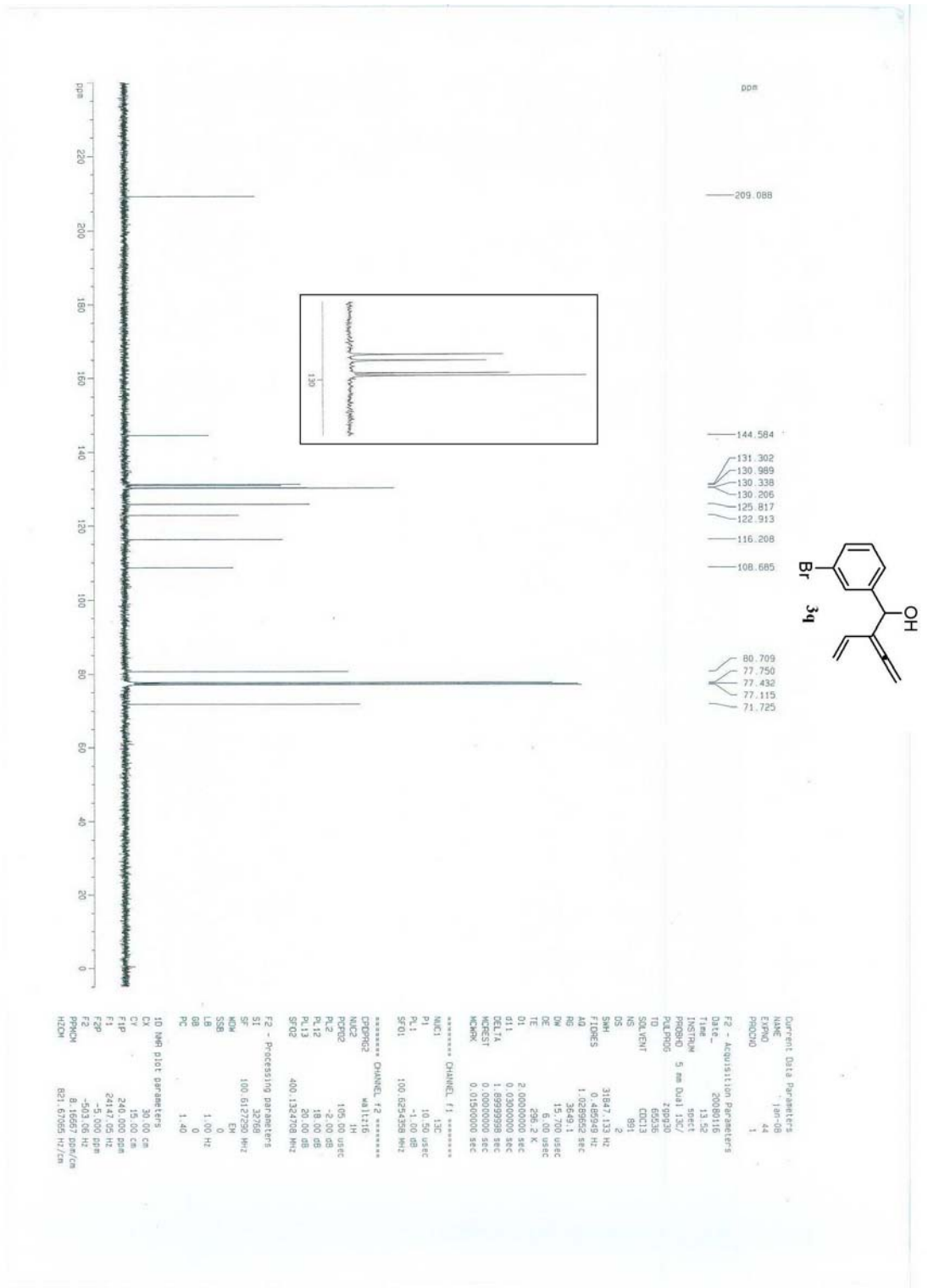

3o
OH
Integral
246.79
236.56
100.00
117.38
130.82
381.21
387.02
100.36
Hz
3181.89
3180.22
3178.41
3175.70
3173.68
3171.84
3169.99
3008.41
3000.13
2907.73
2459.32
2457.20
2447.75
2446.60
2441.94
2440.57
2431.38
2429.48
2195.89
2113.55
2096.96
2094.89
2061.80
2061.07
2049.45
2045.45
2044.46
2042.24
2039.99
2028.87
1038.99
1030.96
1030.01
970.13
-0.00
Current Data Parameters
NAME
EXPNO 107
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071227
Time 16.36
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 161.3
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
******** CHANNEL f1 ********
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 40.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.28667 ppm/cm
HZCM 114.70393 Hz/cm

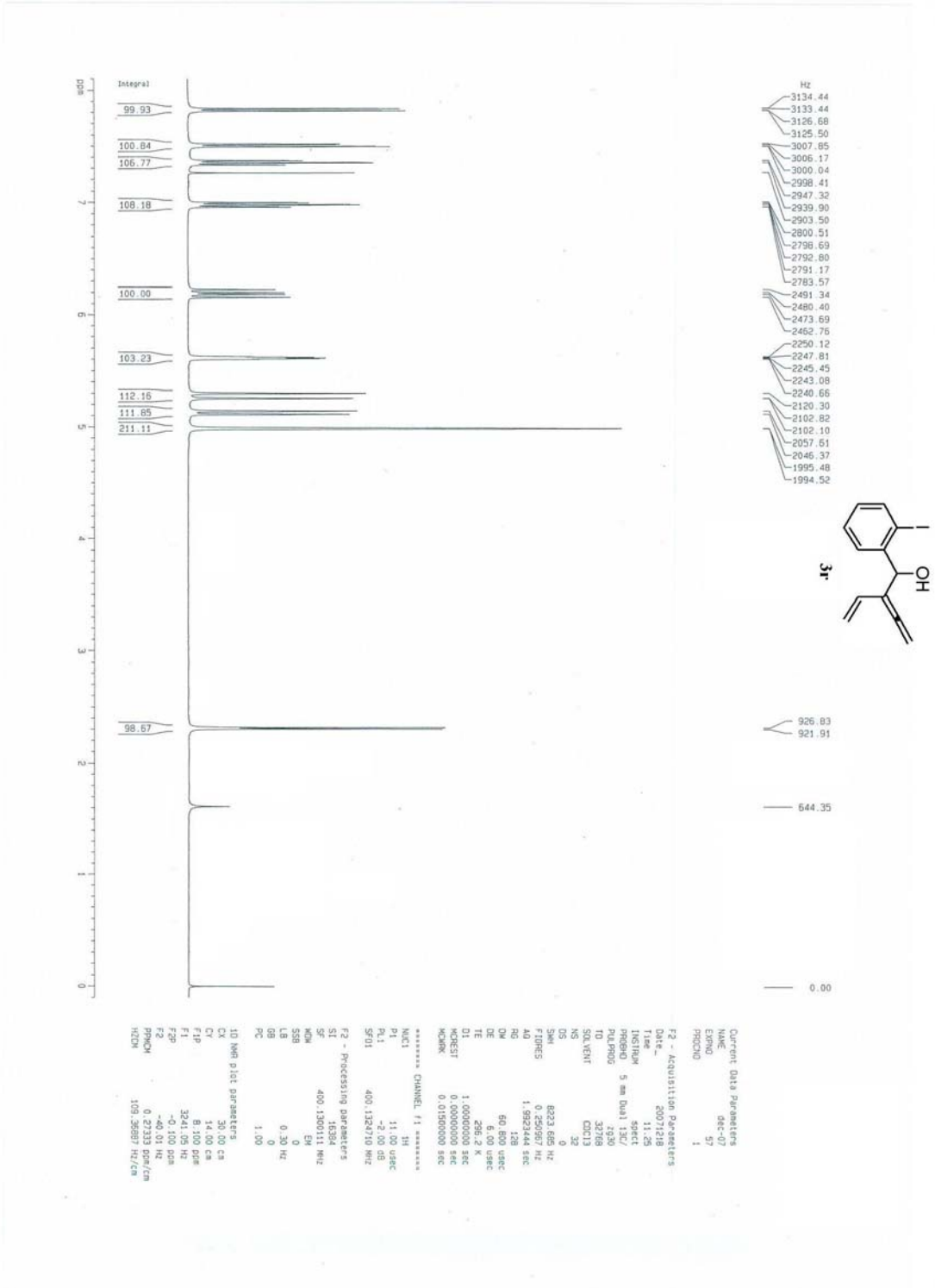

ppm
208.841
197.915
147.201
136.499
130.615
128.460
126.809
115.767
108.263
80.079
77.350
77.032
76.714
71.632
26.664
O
OH
3o

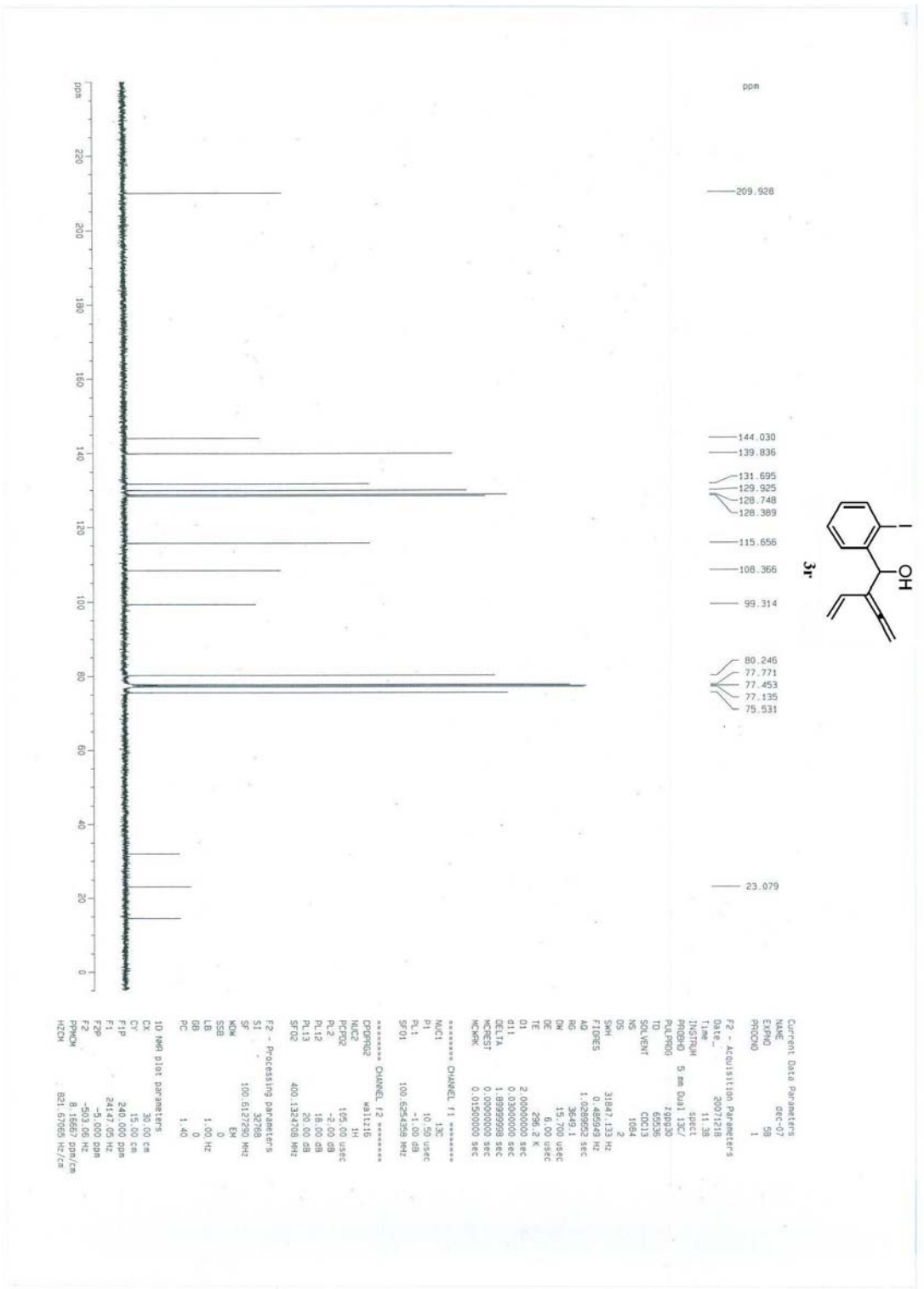

Integral
ppm
258.90
257.15
100.00
118.03
121.75
334.36
335.13
123.23
Hz
3209.41
3207.48
3205.63
3204.01
3201.07
3199.04
3197.30
3195.62
2994.03
2985.74
2908.82
2454.78
2453.29
2443.73
2437.18
2435.64
2426.15
2189.25
2110.16
2109.31
2091.93
2050.57
2048.99
2045.09
2044.08
2040.53
2038.53
2037.23
2036.16
2033.84
2031.26
2029.53
2027.65
2018.53
1567.01
1560.29
1554.99
1553.83
1030.84
0.00
MeO2C
OH
3p
F2 - Acquisition Parameters
Date_ 20071210
Time 16:05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250957 Hz
AQ 1.9923444 sec
RG 90.5
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 33.00 cm
CY 40.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.29967 ppm/cm
HZCM 114.70393 Hz/cm

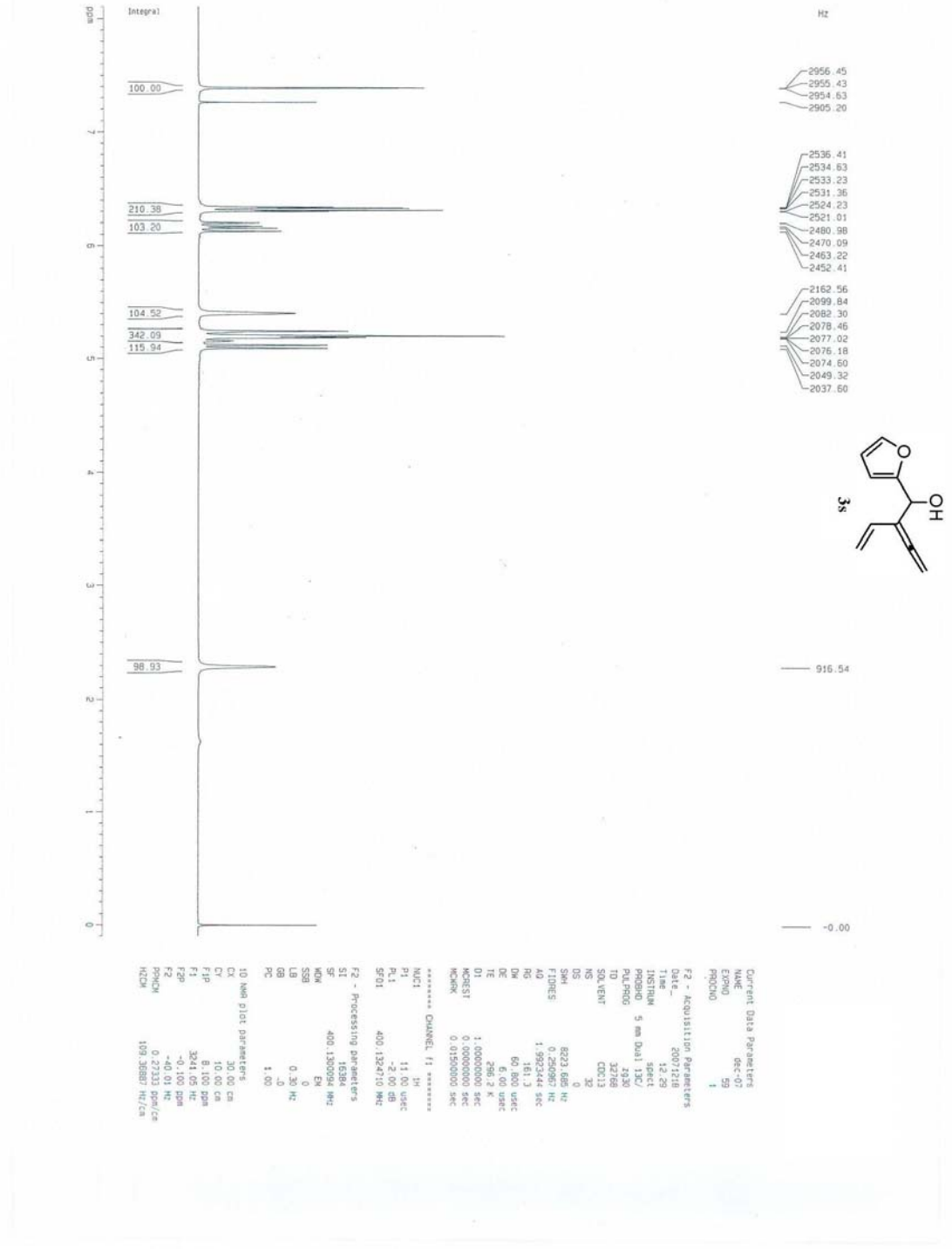

ppm
209.291
167.387
147.440
131.056
130.052
129.849
127.024
116.111
108.674
80.373
77.778
77.460
77.143
72.065
52.532
MeO2C
OH
3p

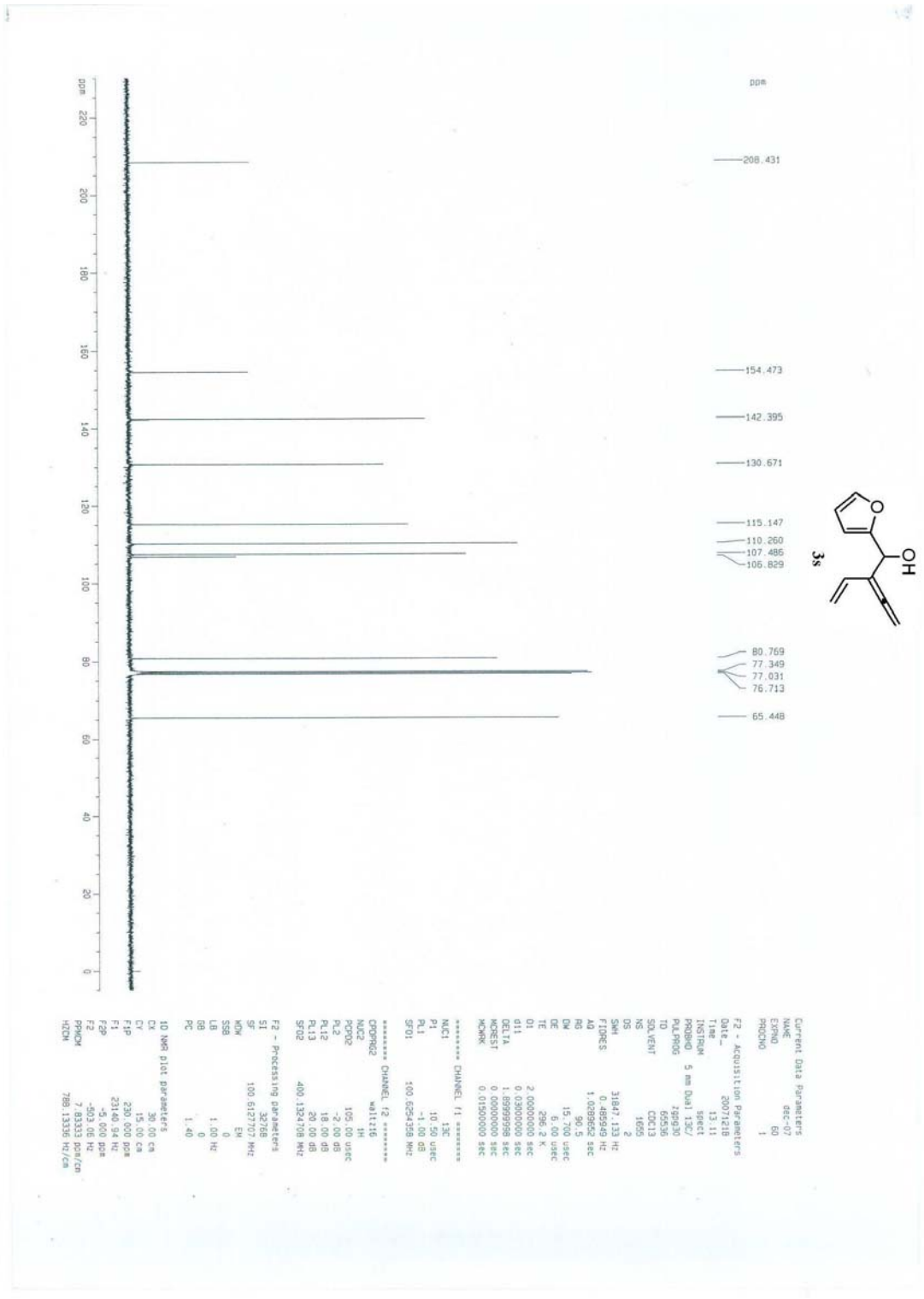

Br
3q
OH

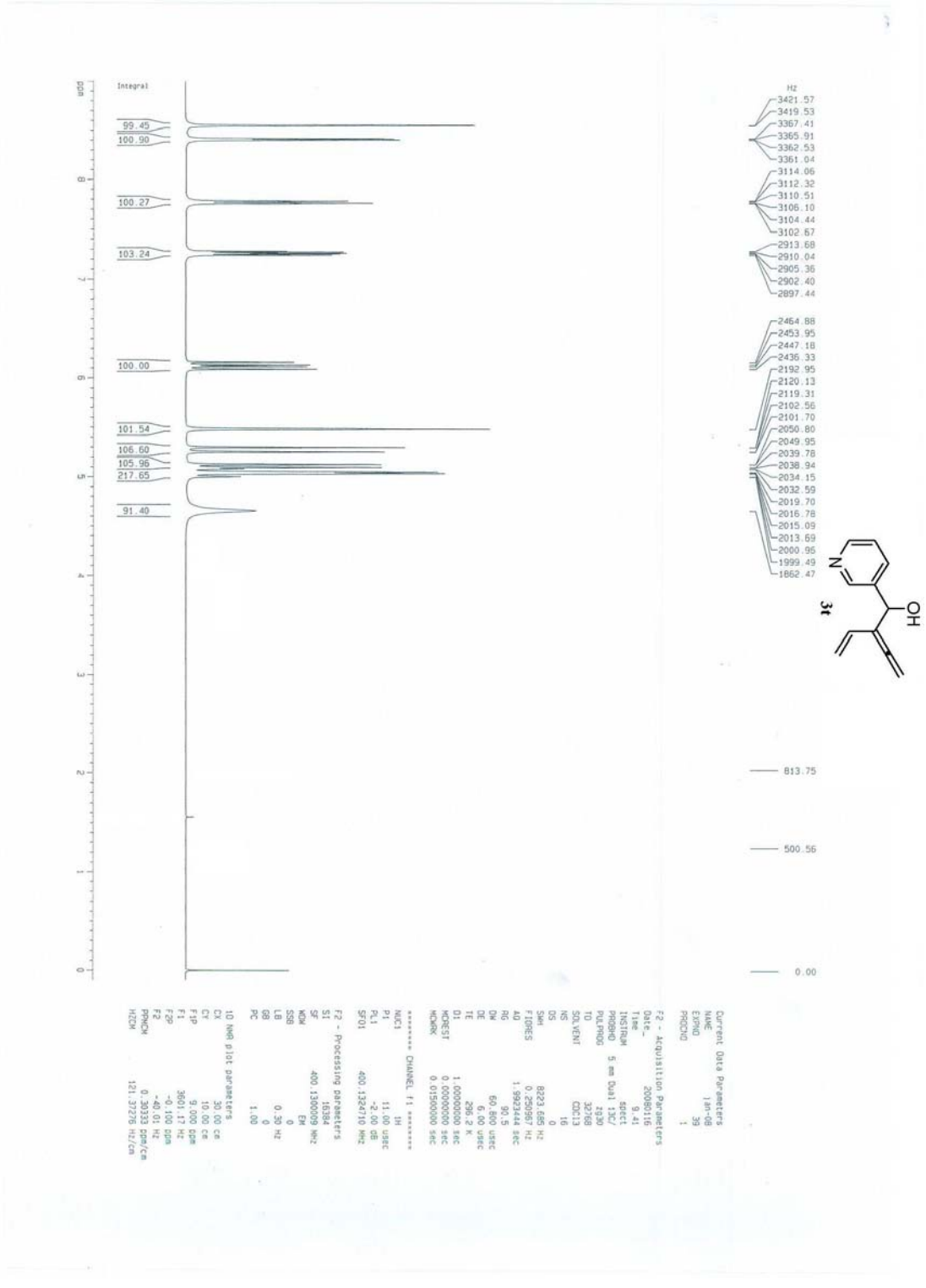

ppm
209.088
144.584
131.302
130.989
130.338
130.206
125.817
122.913
116.208
108.685
80.709
77.750
77.432
77.115
71.725
Br
OH
3q

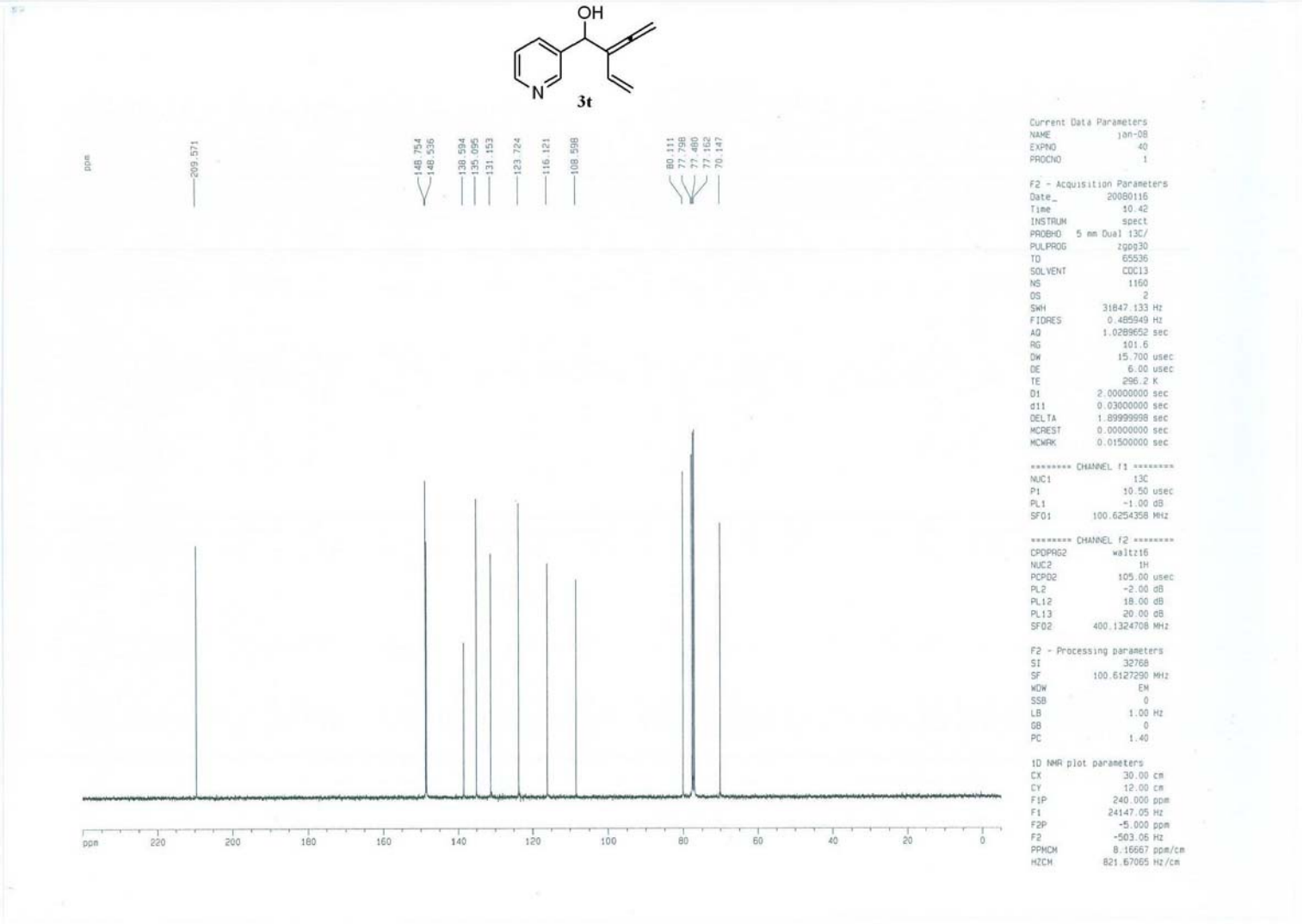

Integral
99.93
100.84
106.77
108.18
100.00
103.23
112.16
111.85
211.11
98.67
Hz
3134.44
3133.44
3126.68
3125.50
3007.85
3006.17
3000.04
2998.41
2947.32
2939.90
2903.50
2800.51
2798.69
2792.80
2791.17
2783.57
2491.34
2480.40
2473.69
2462.76
2250.12
2247.81
2245.45
2243.08
2240.66
2120.30
2102.82
2102.10
2057.61
2046.37
1995.48
1994.52
926.83
921.91
644.35
0.00
3r
I
OH
Current Data Parameters
NAME dec-07
EXPNO 57
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071218
Time 11.25
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250897 Hz
AQ 1.9923444 sec
RG 128
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 14.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36087 Hz/cm


ppm
209.928
144.030
139.836
131.695
129.925
128.748
128.389
115.656
108.366
99.314
80.246
77.771
77.453
77.135
75.531
23.079
I
OH
3r
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
d11
DELTA
MCREST
MCWRK
======== CHANNEL f1 ========
NUC1
P1
PL1
SF01
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM
ppm
220
200
180
160
140
120
100
80
60
40
20
0

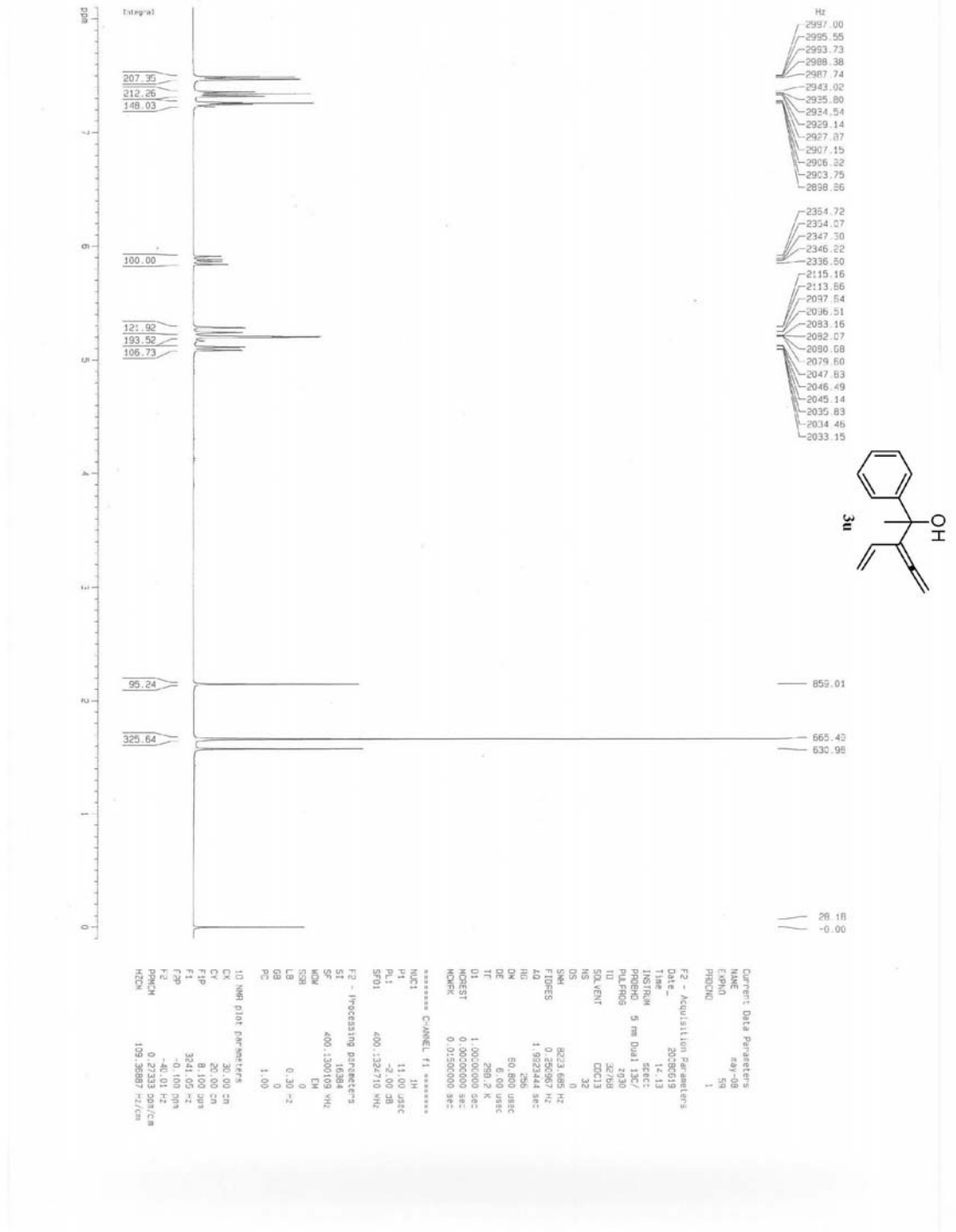

Integral
100.00
210.38
103.20
104.52
342.09
115.94
98.93
Hz
2956.45
2955.43
2954.63
2905.20
2536.41
2534.63
2533.23
2531.36
2524.23
2521.01
2480.98
2470.09
2463.22
2452.41
2162.56
2099.84
2082.30
2078.46
2077.02
2076.18
2074.60
2049.32
2037.60
916.54
-0.00
3s
OH

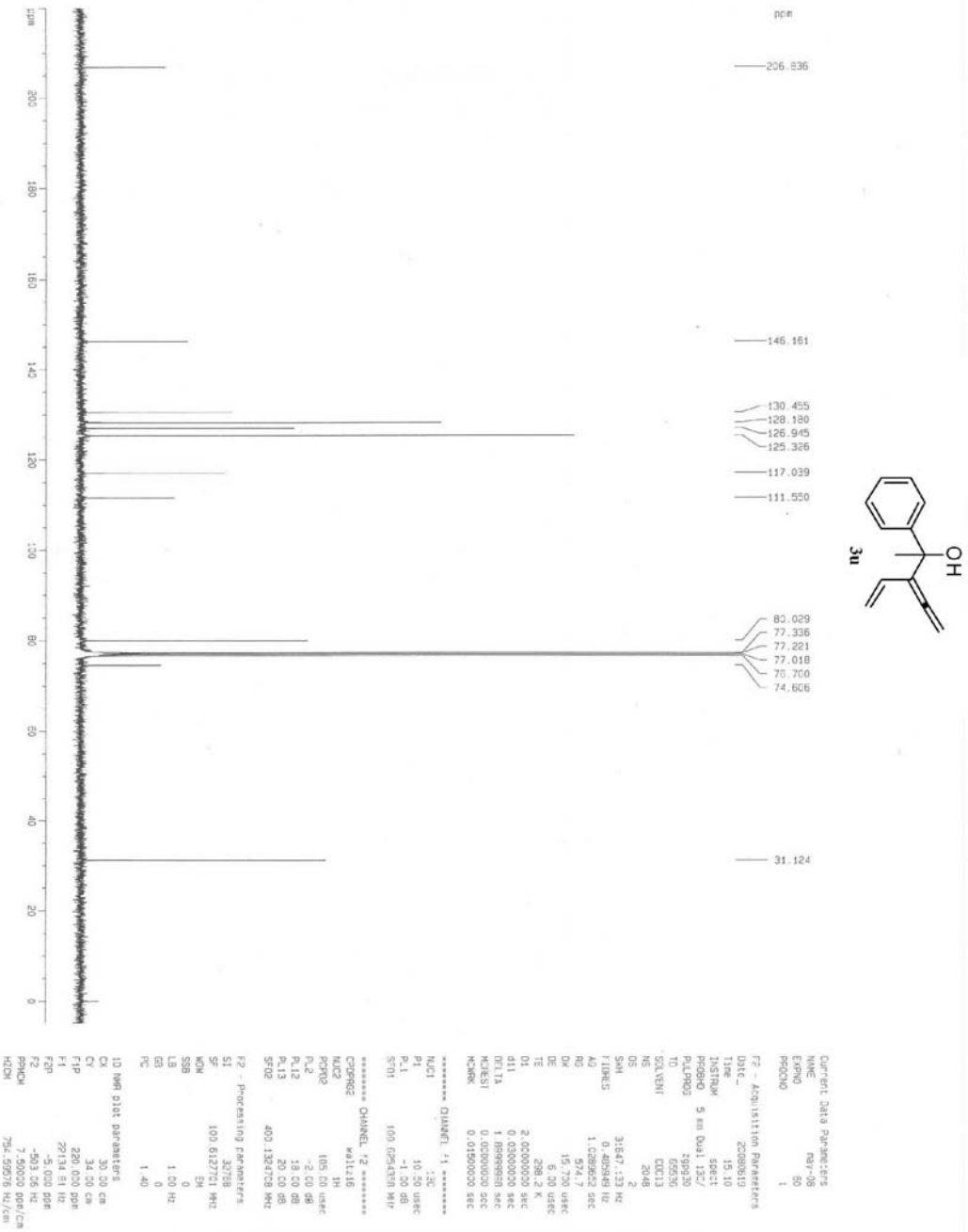

ppm
208.431
154.473
142.395
130.671
115.147
110.260
107.485
105.829
80.769
77.349
77.031
76.713
65.448
3s
OH
O
Current Data Parameters
NAME dec-07
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071218
Time 13.11
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1605
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 90.5
DW 15.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324708 MHz
F2 - Processing parameters
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 230.000 ppm
F1 23140.94 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.83333 ppm/cm
HZCM 788.13336 Hz/cm

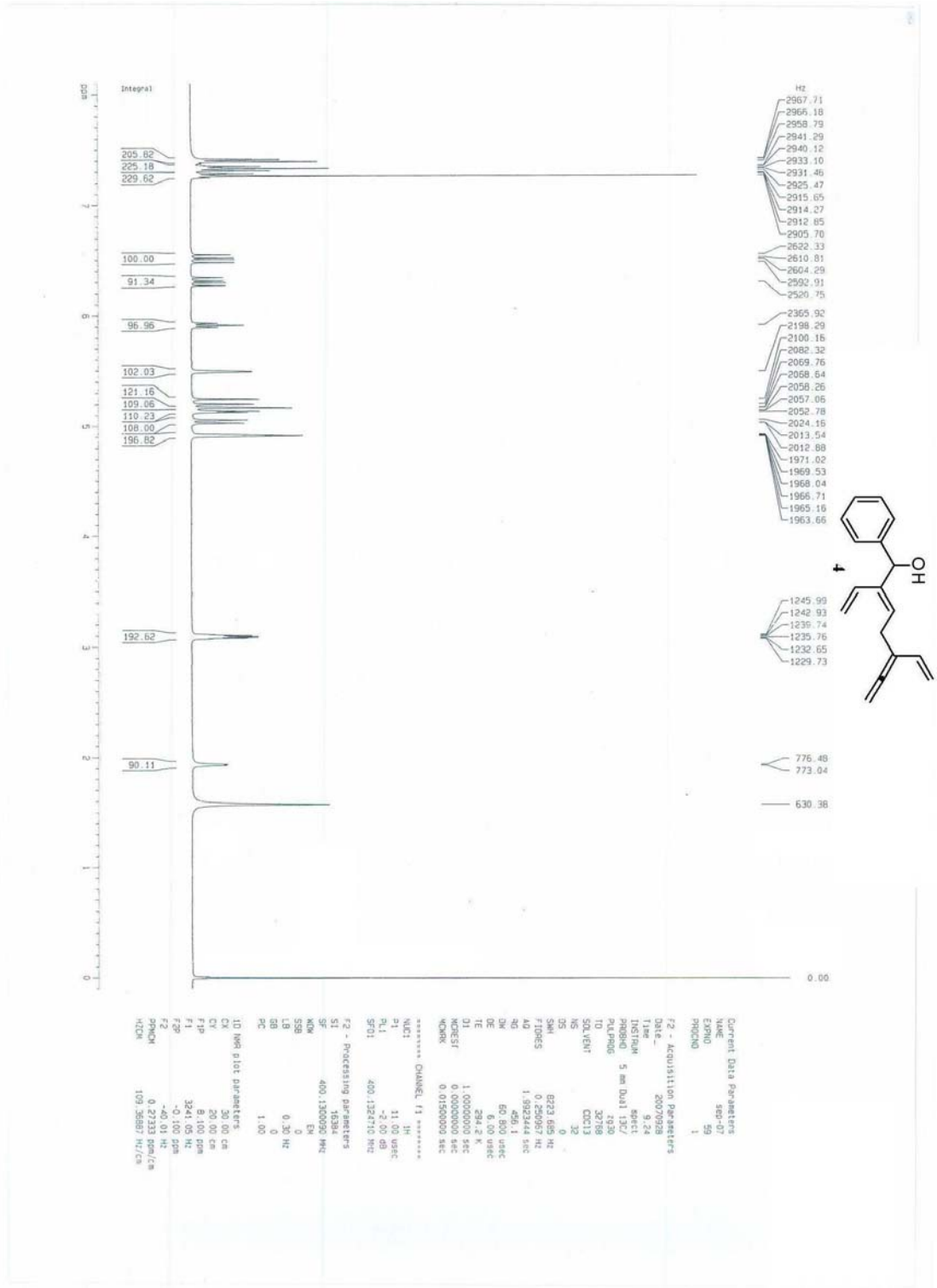

Integral
99.45
100.90
100.27
103.24
100.00
101.54
106.60
105.96
217.65
91.40
Hz
3421.57
3419.53
3367.41
3365.91
3362.53
3361.04
3114.06
3112.32
3110.51
3106.10
3104.44
3102.67
2913.68
2910.04
2905.36
2902.40
2897.44
2464.88
2453.95
2447.18
2436.33
2192.95
2120.13
2119.31
2102.56
2101.70
2050.80
2049.95
2039.78
2038.94
2034.15
2032.59
2019.70
2016.78
2015.09
2013.69
2000.95
1999.49
1862.47
N
OH
3t
813.75
500.56
0.00
Current Data Parameters
NAME 1an-08
EXPNO 39
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080116
Time 9.41
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.250987 Hz
AQ 1.9923444 sec
RG 90.5
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
D11 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300009 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.30333 ppm/cm
HZCM 121.37276 Hz/cm

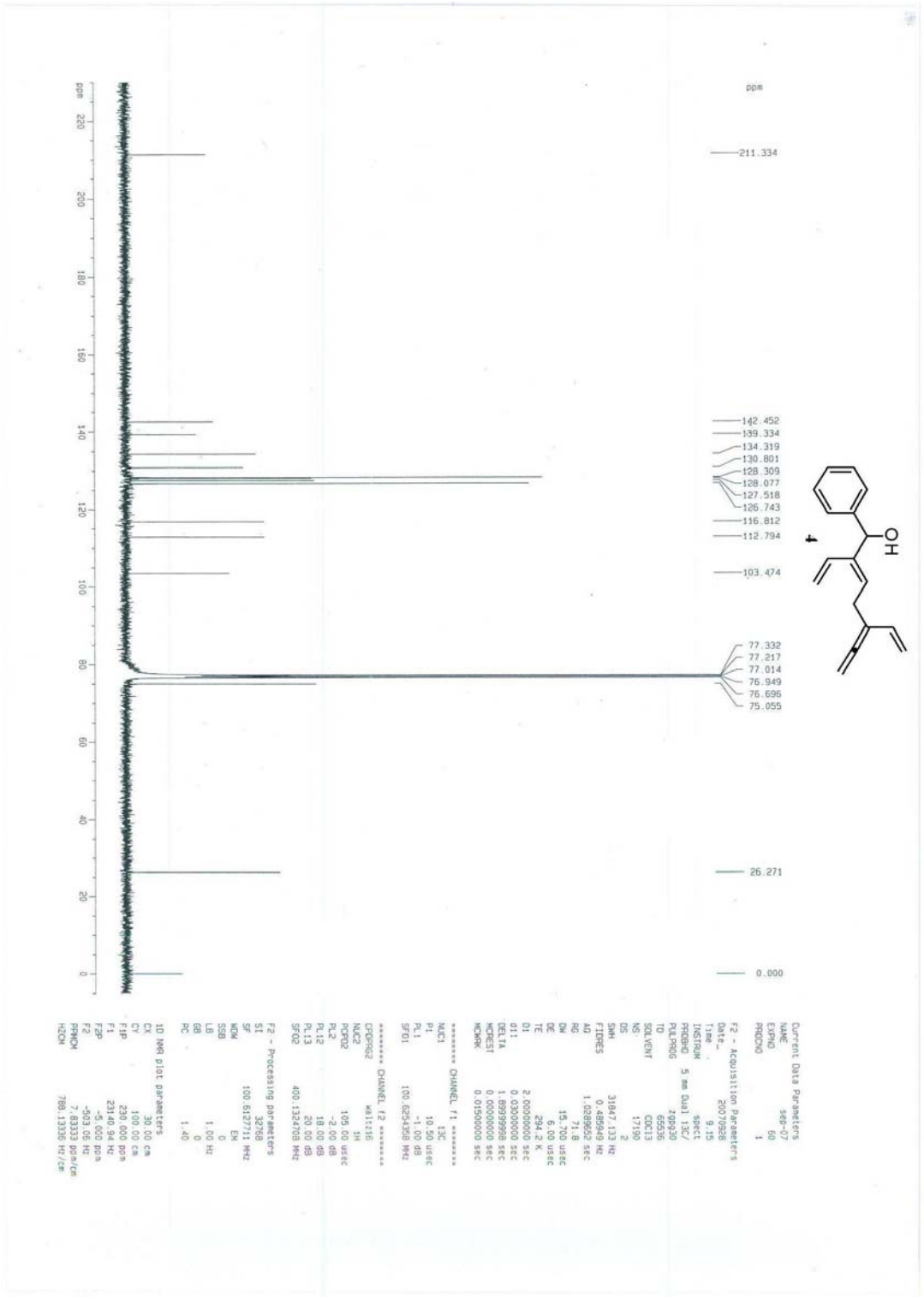


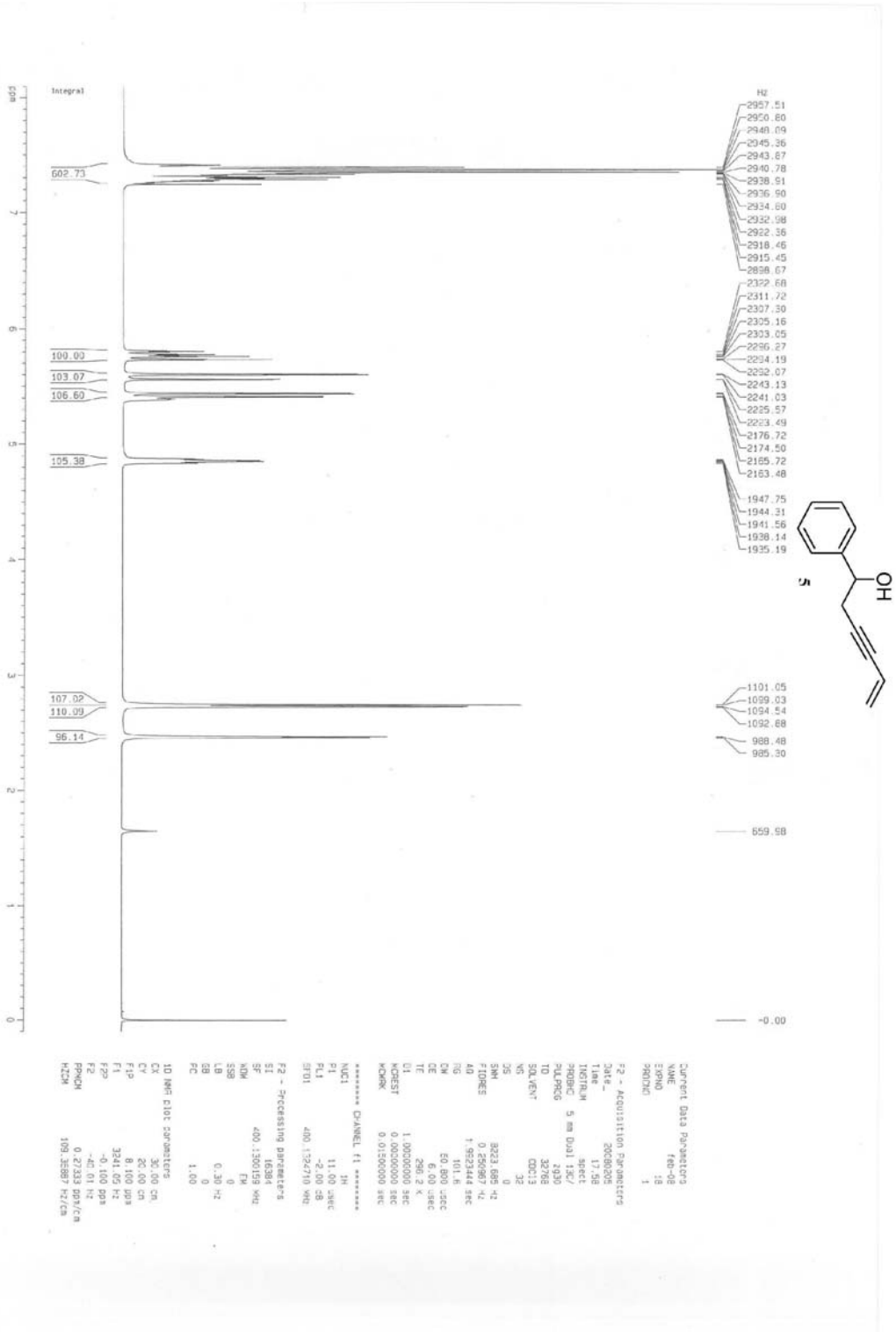

```
Current Data Parameters
NAME                  jan-08
EXPNO                     40
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080116
Time                   10.42
INSTRUM                spect
PROBHD    5 mm Dual 13C/
PULPROG              zgpg30
TD                      65536
SOLVENT                CDCl3
NS                       1160
DS                          2
SWH                31847.133 Hz
FIDRES             0.485949 Hz
AQ                 1.0289652 sec
RG                      101.6
DW                     15.700 usec
DE                      6.00 usec
TE                     296.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.50 usec
PL1                     -1.00 dB
SFO1              100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  105.00 usec
PL2                     -2.00 dB
PL12                    18.00 dB
PL13                    20.00 dB
SFO2              400.1324708 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127290 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        30.00 cm
CY                        12.00 cm
F1P                      240.000 ppm
F1                     24147.05 Hz
F2P                       -5.000 ppm
F2                      -503.06 Hz
PPMCM                  8.16667 ppm/cm
HZCM                821.67065 Hz/cm
```

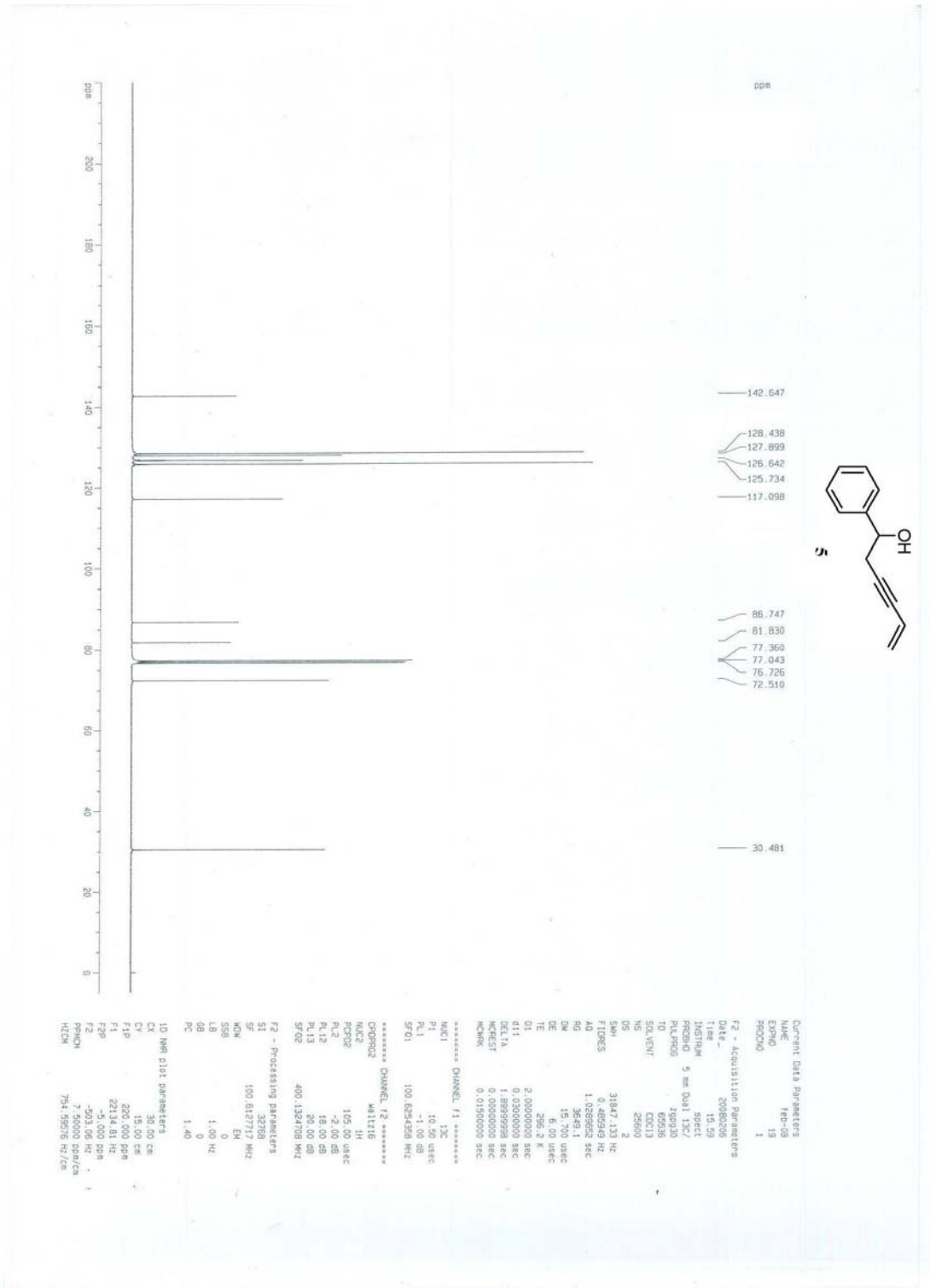

Hz
2997.00
2995.55
2993.73
2988.38
2987.74
2943.02
2935.80
2934.54
2929.14
2927.87
2907.15
2906.32
2903.75
2898.86
2354.72
2354.07
2347.30
2346.22
2336.50
2115.16
2113.86
2097.54
2096.51
2083.16
2082.07
2080.08
2079.60
2047.83
2046.49
2045.14
2035.83
2034.46
2033.15
Integral
207.35
212.26
148.03
100.00
121.92
193.52
106.73
95.24
325.64
859.01
665.40
630.98
28.18
-0.00
ppm
3u
OH
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F2P
F1
F2
PPMCM
HZCM

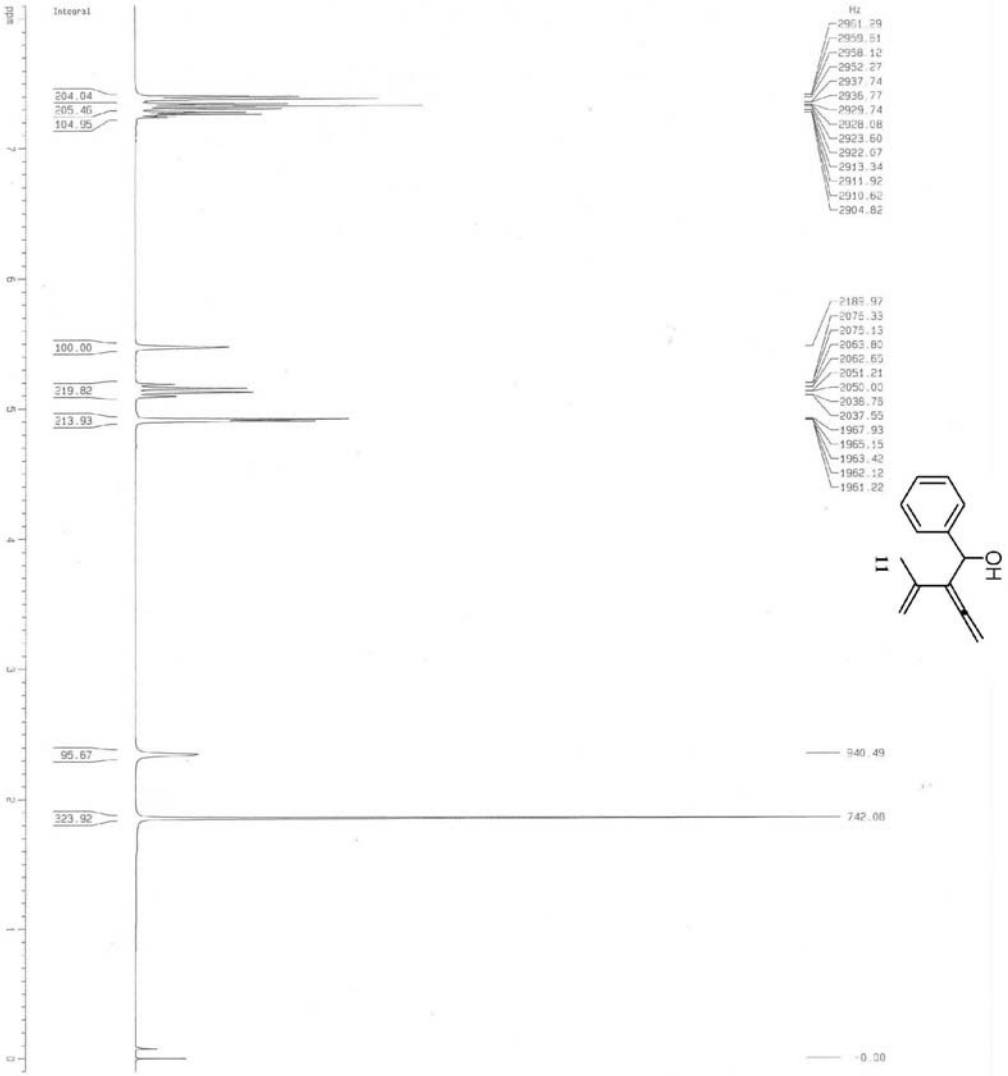

ppm
206.836
146.161
130.455
128.180
126.945
125.326
117.039
111.550
80.029
77.336
77.221
77.018
76.700
74.606
31.124
OH
3u


Integral
205.82
225.18
229.62
100.00
91.34
96.96
102.03
121.16
109.06
110.23
108.00
196.82
192.62
90.11
Hz
2967.71
2966.18
2958.79
2941.29
2940.12
2933.10
2931.46
2925.47
2915.65
2914.27
2912.85
2905.70
2622.33
2610.81
2604.29
2592.91
2520.75
2365.92
2198.29
2100.16
2082.32
2069.76
2068.64
2058.26
2057.06
2052.78
2024.16
2013.54
2012.88
1971.02
1969.53
1968.04
1966.71
1965.16
1963.66
1245.99
1242.93
1239.74
1235.76
1232.65
1229.73
776.48
773.04
630.38
0.00
OH
4


ppm
211.334
142.452
139.334
134.319
130.801
128.309
128.077
127.518
126.743
116.812
112.794
103.474
77.332
77.217
77.014
76.949
76.696
75.055
26.271
0.000
4
OH


ppm
Integral
602.73
100.00
103.07
106.60
105.38
107.02
110.09
96.14
Hz
2957.51
2950.80
2948.09
2945.36
2943.87
2940.78
2938.91
2936.90
2934.60
2932.98
2922.36
2918.46
2915.45
2898.67
2322.68
2311.72
2307.30
2305.16
2303.05
2296.27
2294.19
2292.07
2243.13
2241.03
2225.57
2223.49
2176.72
2174.50
2165.72
2163.48
1947.75
1944.31
1941.56
1938.14
1935.19
1101.05
1099.03
1094.54
1092.88
988.48
985.30
659.98
-0.00
OH
5
Current Data Parameters
NAME feb-08
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080305
TIME 17.58
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250867 Hz
AQ 1.9923444 sec
RG 101.6
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
HDREST 0.00000000 sec
HDWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300159 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.35887 Hz/cm


ppm
142.647
128.438
127.899
126.642
125.734
117.098
86.747
81.830
77.360
77.043
76.726
72.510
30.481
OH
5
Current Data Parameters
NAME feb-08
EXPNO 19
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080206
Time 15.59
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0288652 sec
RG 3649.1
DW 15.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6284358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324708 MHz
F2 - Processing parameters
SI 32768
SF 100.6127717 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.50000 ppm/cm
HZCM 754.59576 Hz/cm



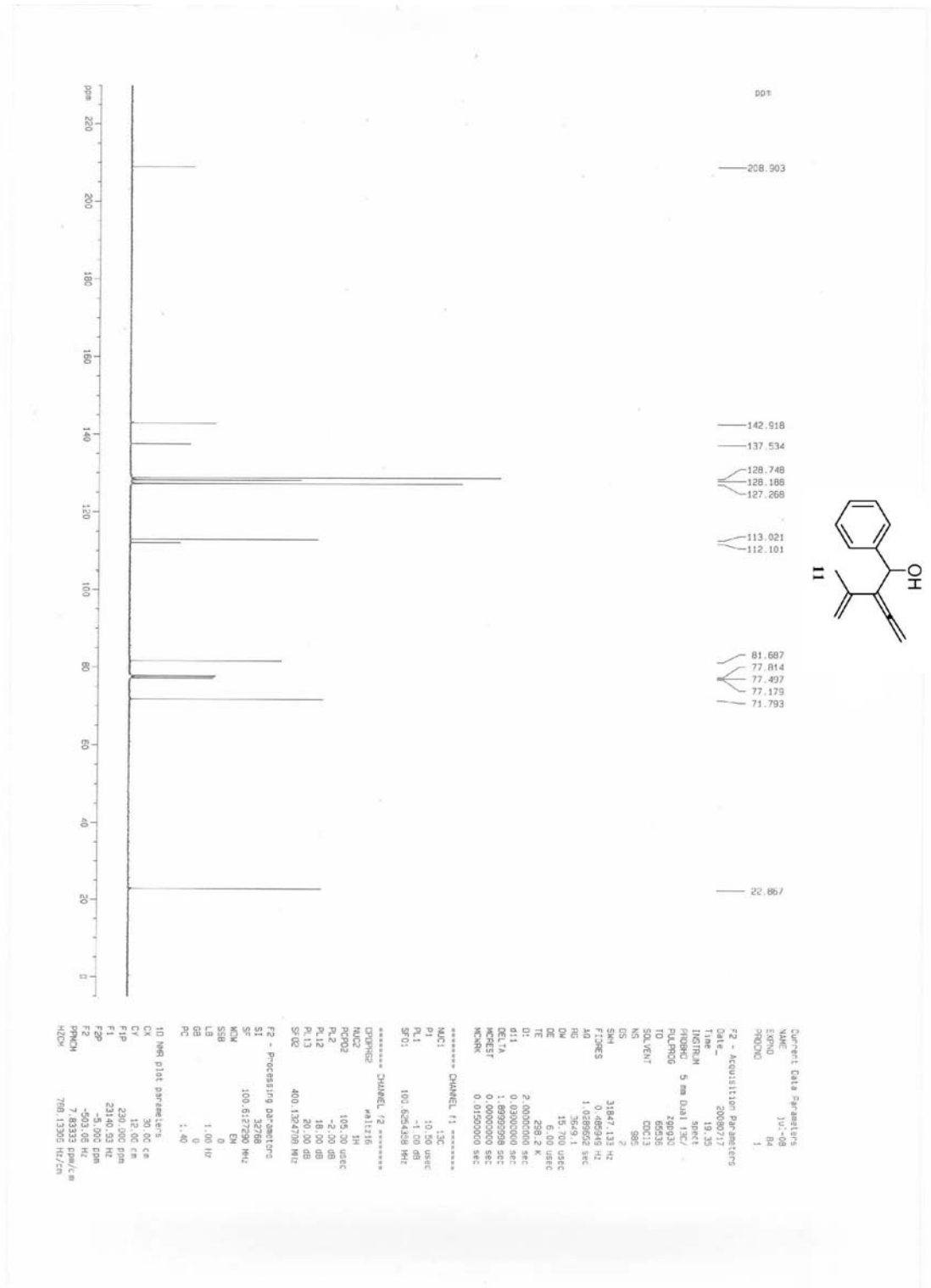
Current Data Parameters
NAME 11
EXPNO 83
PROCNO 1

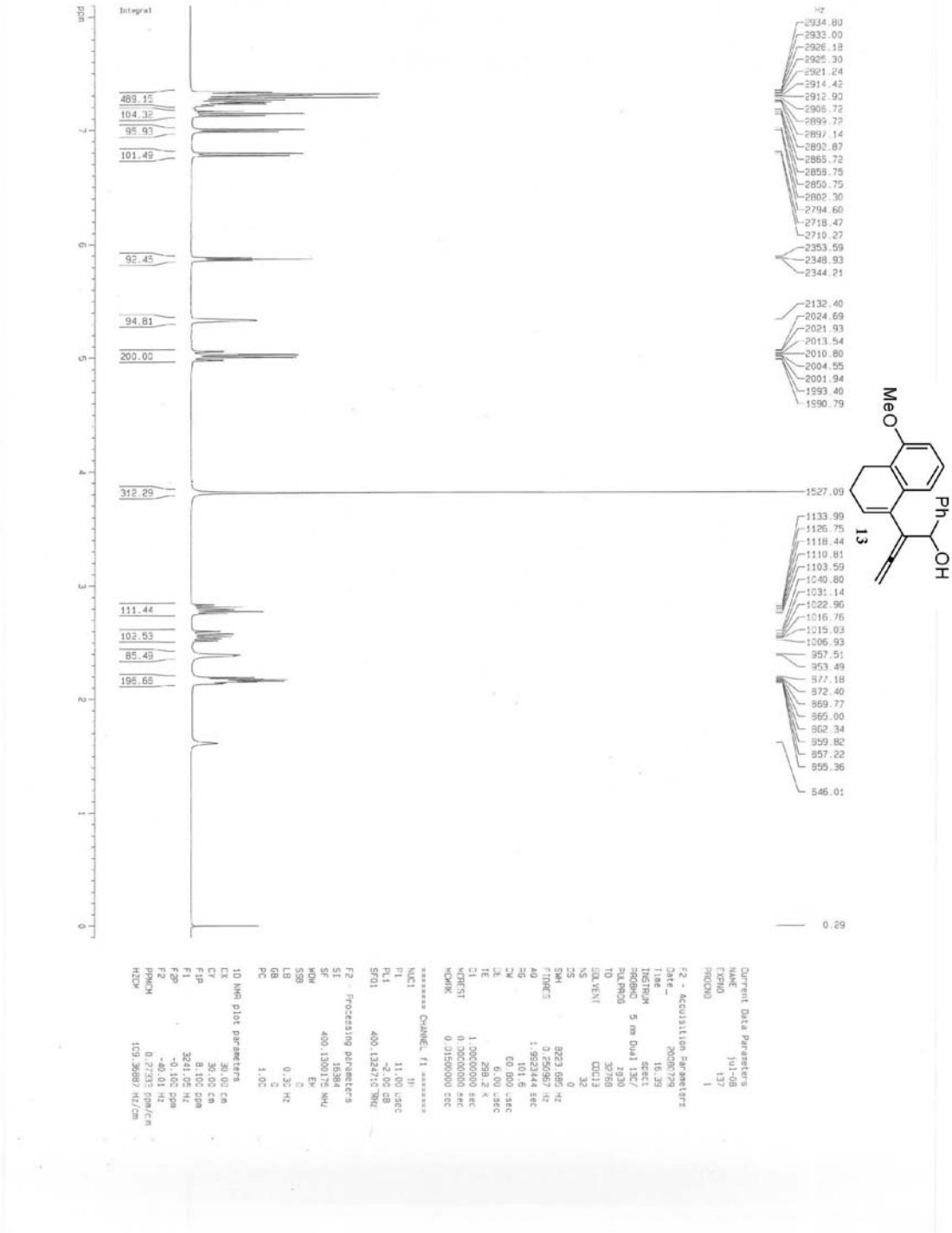
F2 - Acquisition Parameters
Date_ 20080717
Time 18.42
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250867 Hz
AQ 1.9923444 sec
RG 71.0
DW 60.800 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

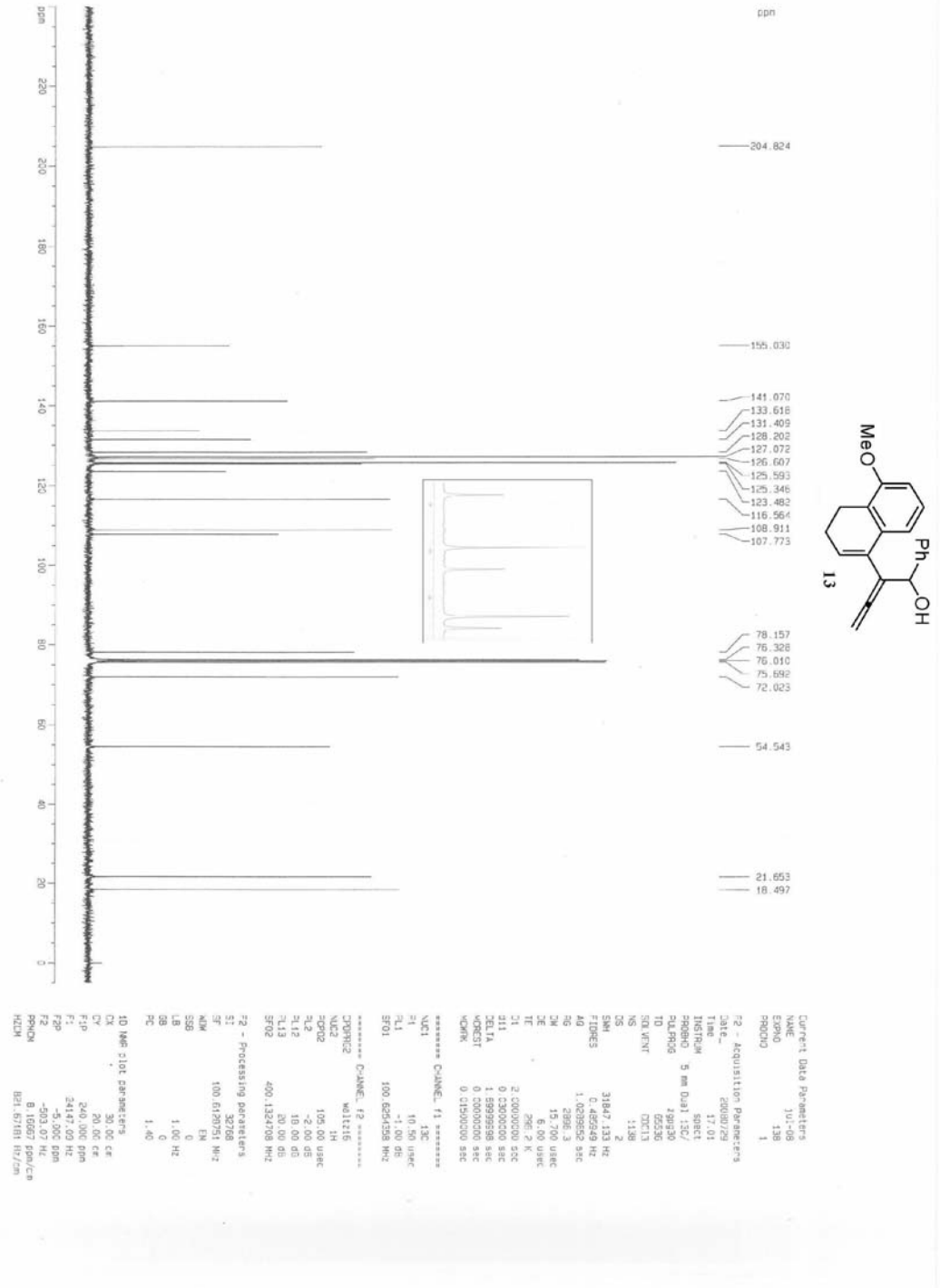
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

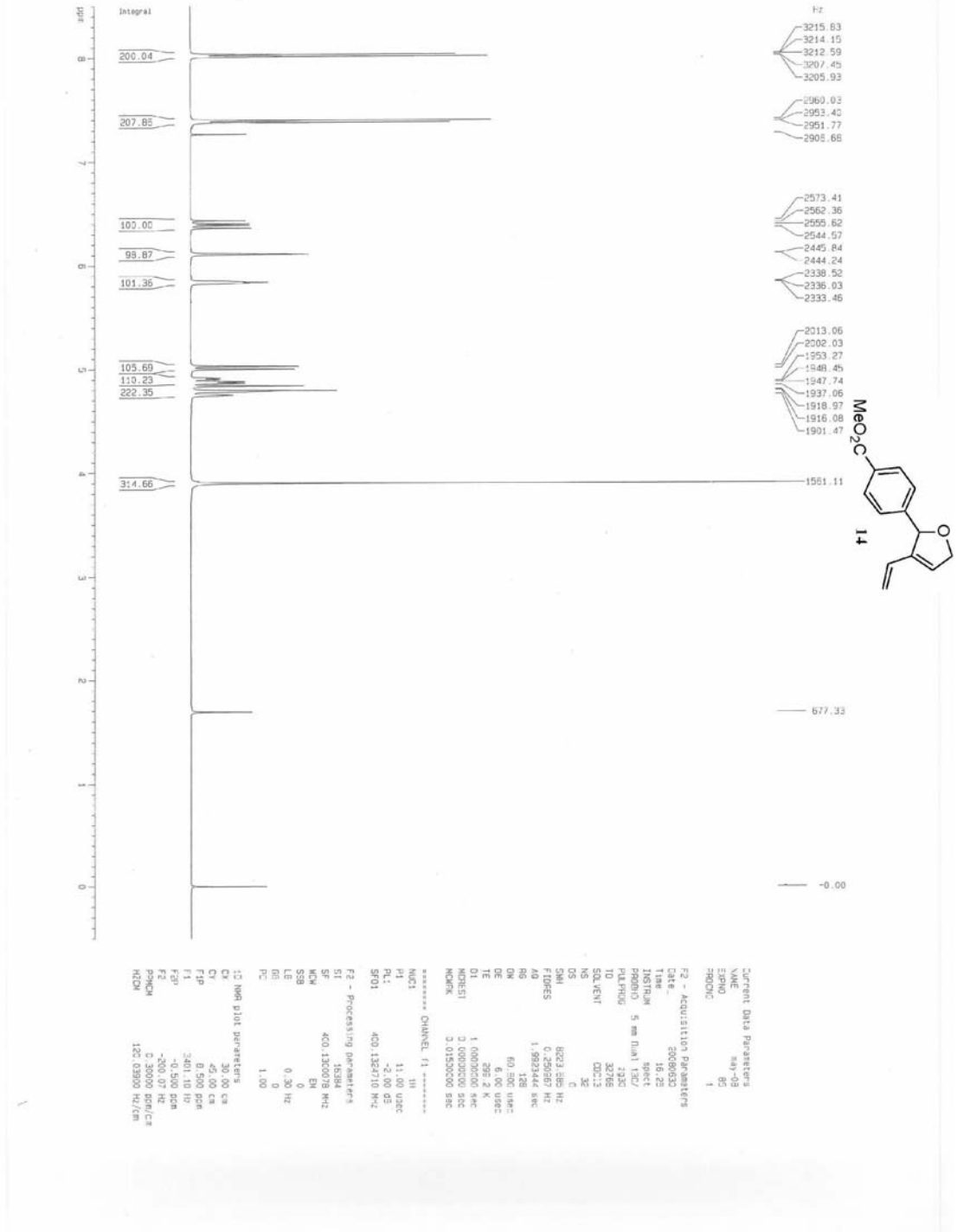
F2 - Processing parameters
SI 16384
SF 400.1300195 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36887 Hz/cm


ppm
208.903
142.918
137.534
128.748
128.188
127.268
113.021
112.101
81.687
77.814
77.497
77.179
71.793
22.867
OH
11
Current Data Parameters
NAME jw-08
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080717
Time 19.35
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 985
DS 2
SWH 31847.133 Hz
FIDRES 0.485946 Hz
AQ 1.0286652 sec
RG 3649.1
DW 15.700 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89995998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254388 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324700 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 12.00 cm
F1P 230.000 ppm
F1 23140.03 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.83333 ppm/cm
HZCM 788.13306 Hz/cm


MeO
Ph
OH
13


ppm
204.824
155.030
141.070
133.618
131.409
128.202
127.072
126.607
125.593
125.346
123.482
116.564
108.911
107.773
78.157
76.328
76.010
75.692
72.023
54.543
21.653
18.497
MeO
Ph
OH
13



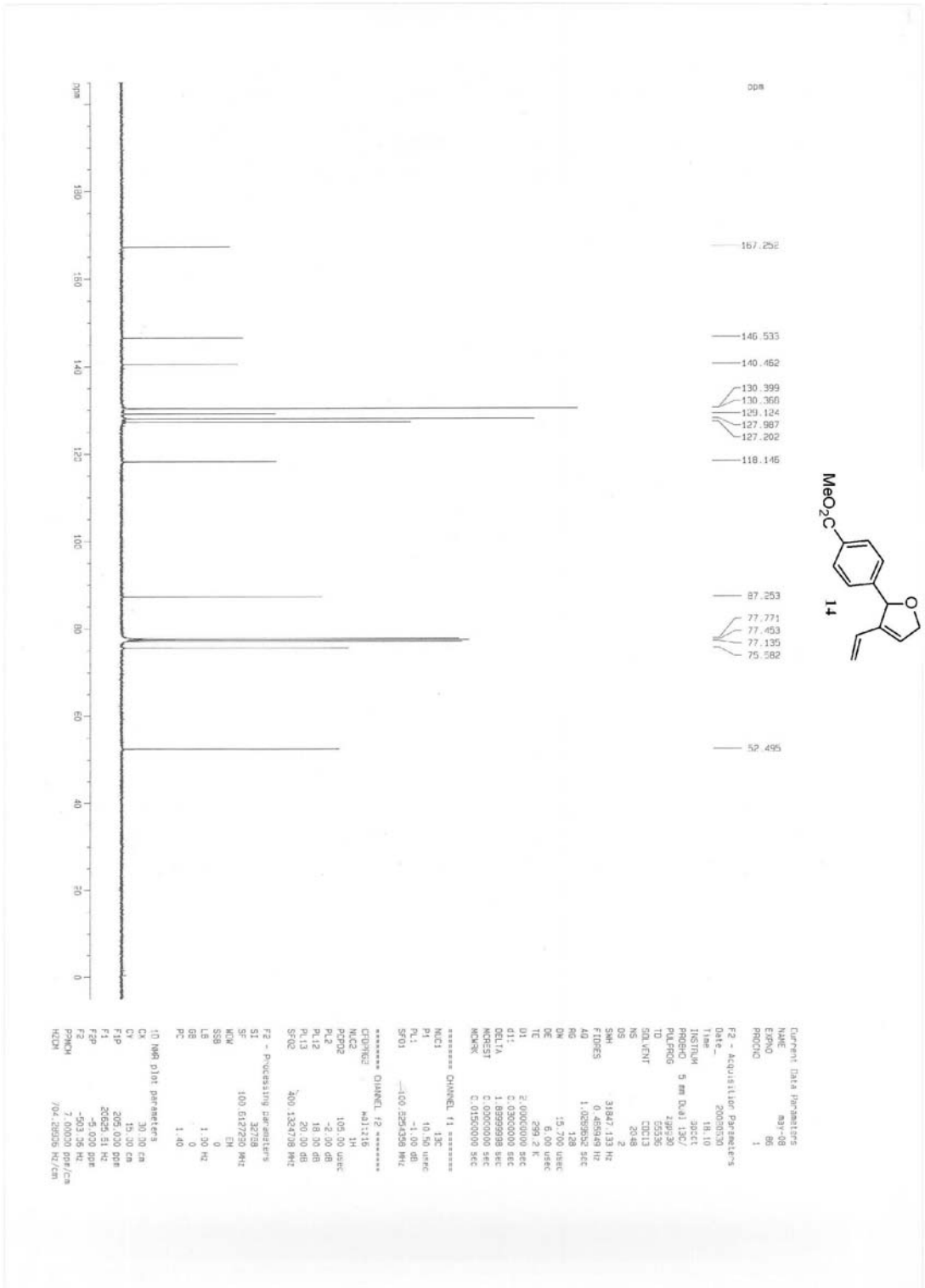
Current Data Parameters
NAME
EXPNO 85
PROCNO 1

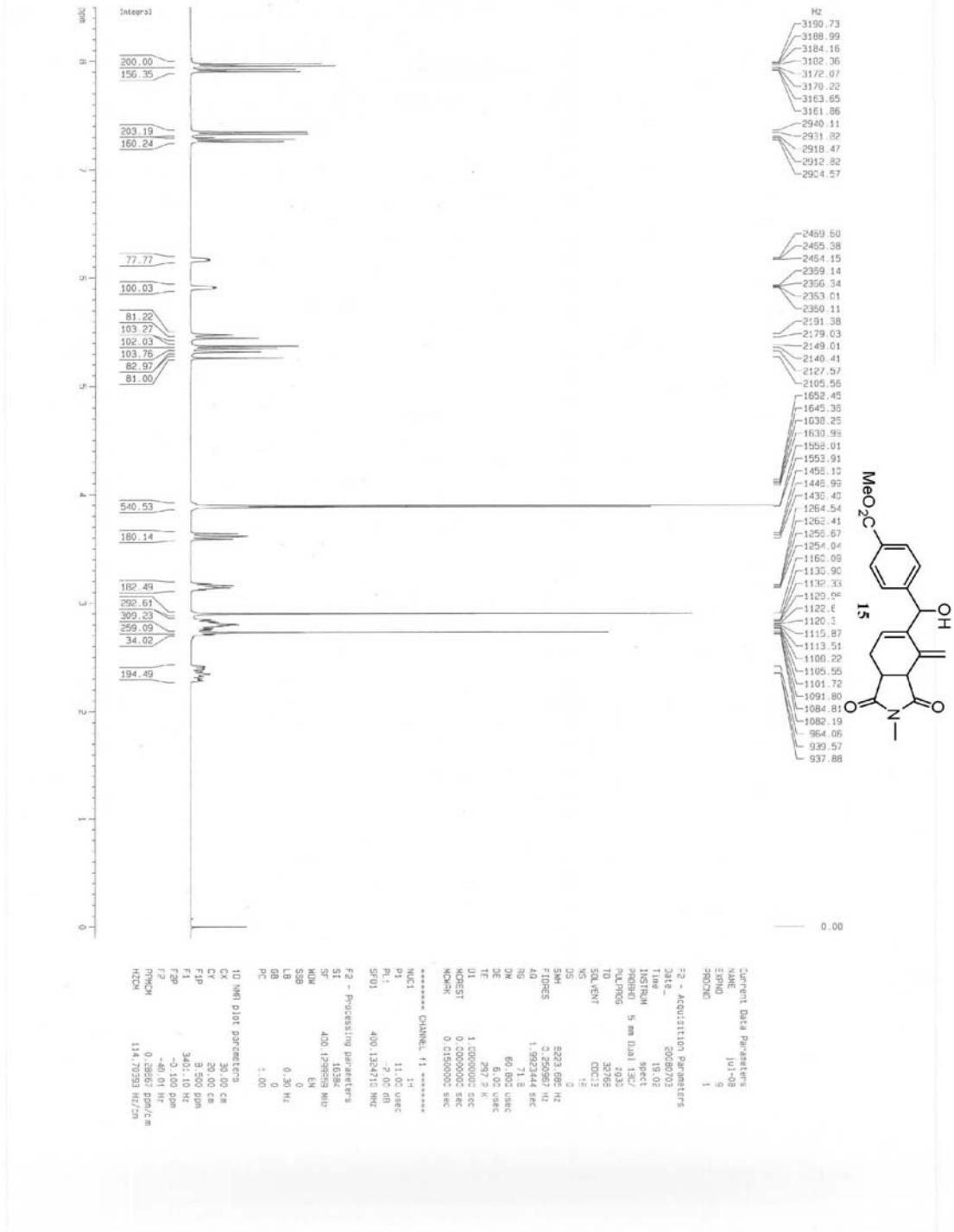
F2 - Acquisition Parameters
Date_ 20080833
Time 16.25
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250987 Hz
AQ 1.9923444 sec
RG 128
DW 60.500 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
D11 0.03000000 sec
MCWRK 0.01500000 sec

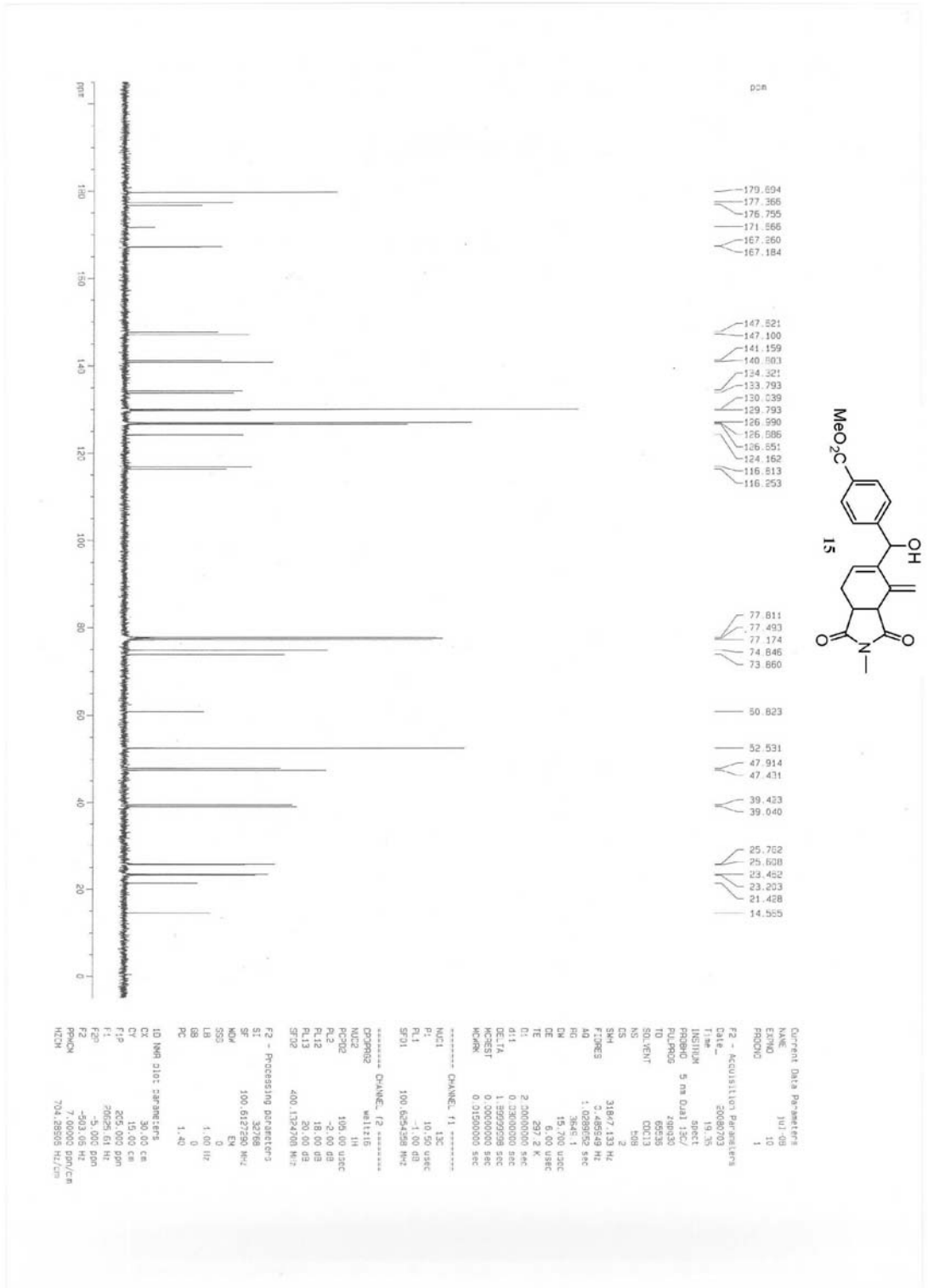
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1322710 MHz

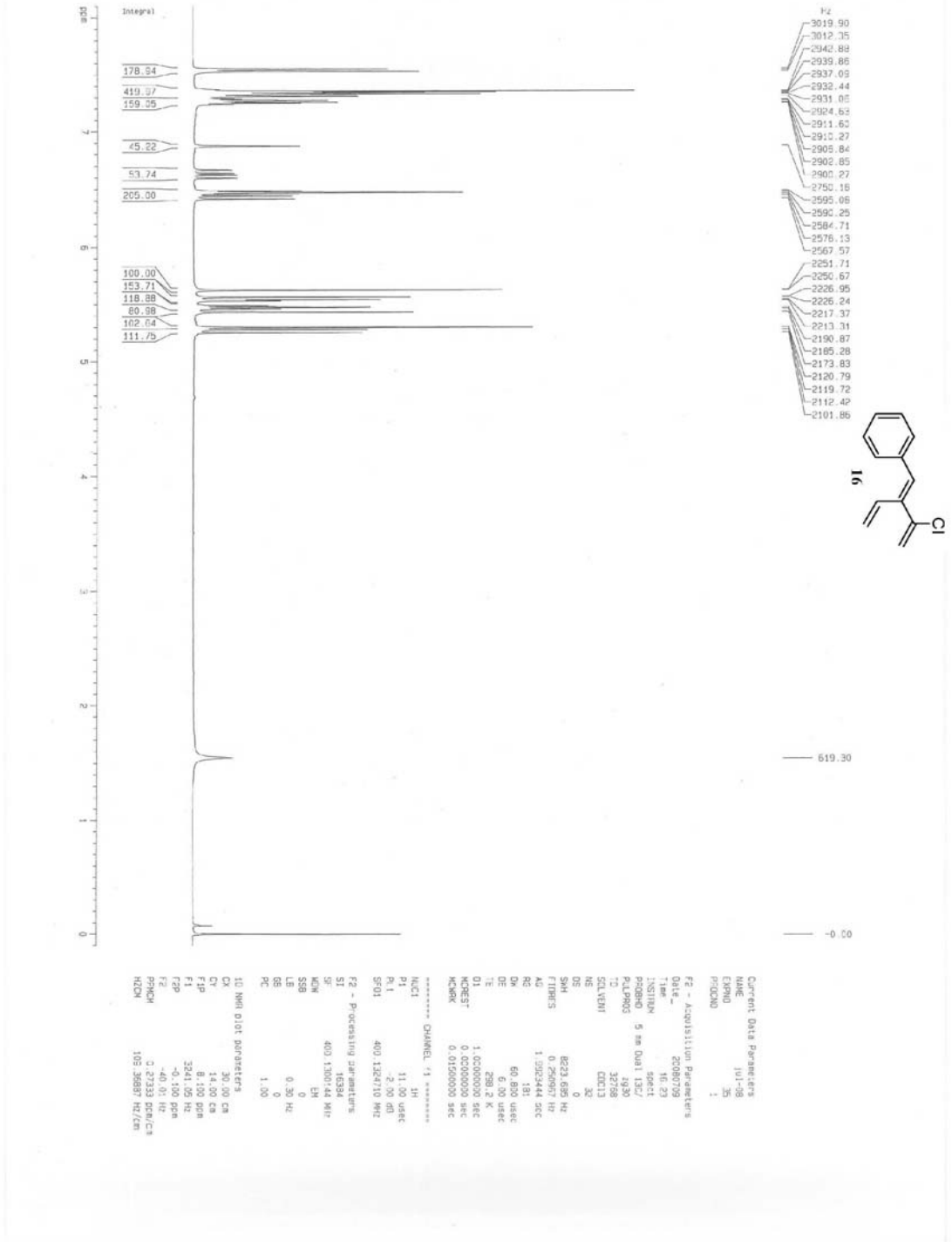
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

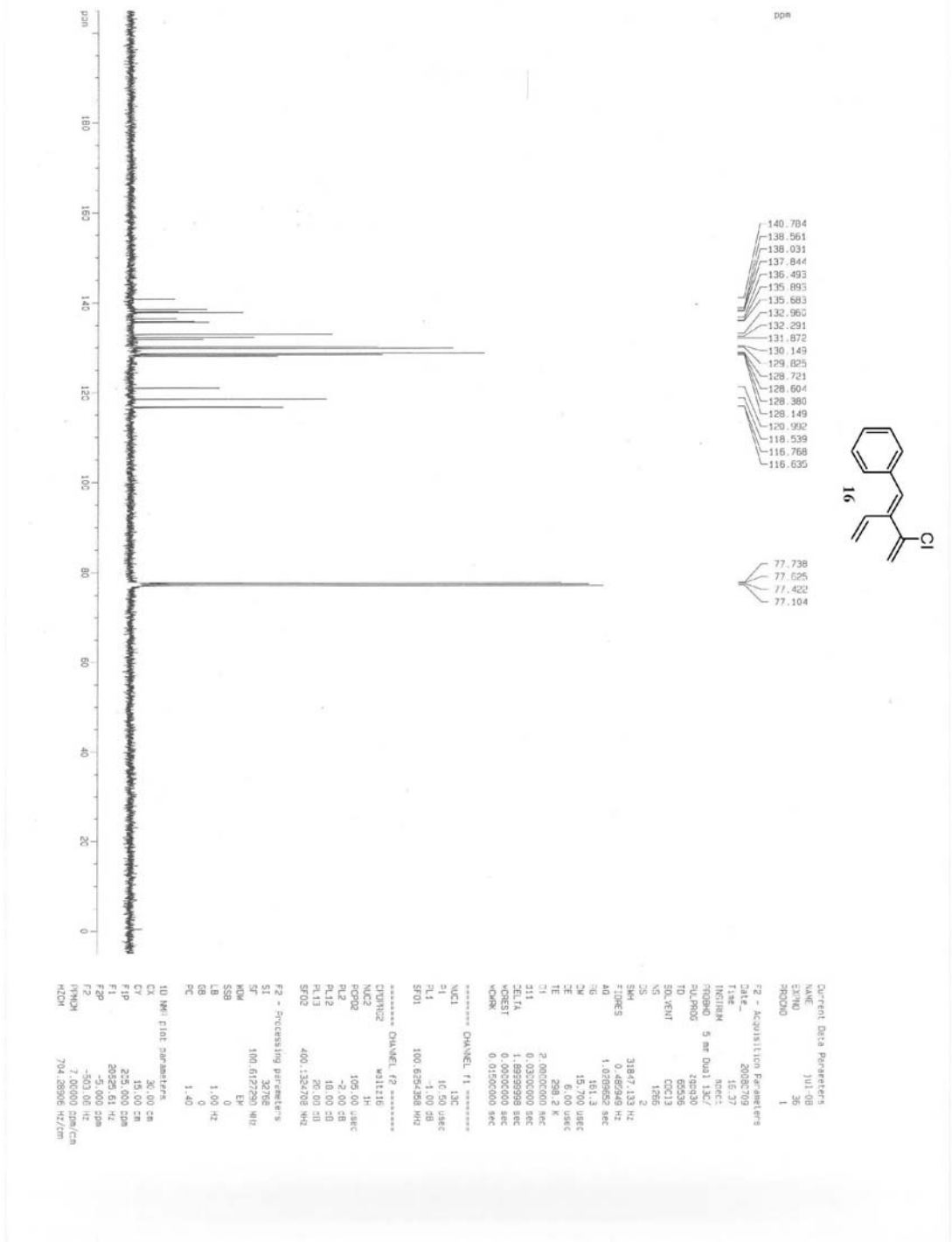
1D NMR plot parameters
CX 30.00 cm
CY 45.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm

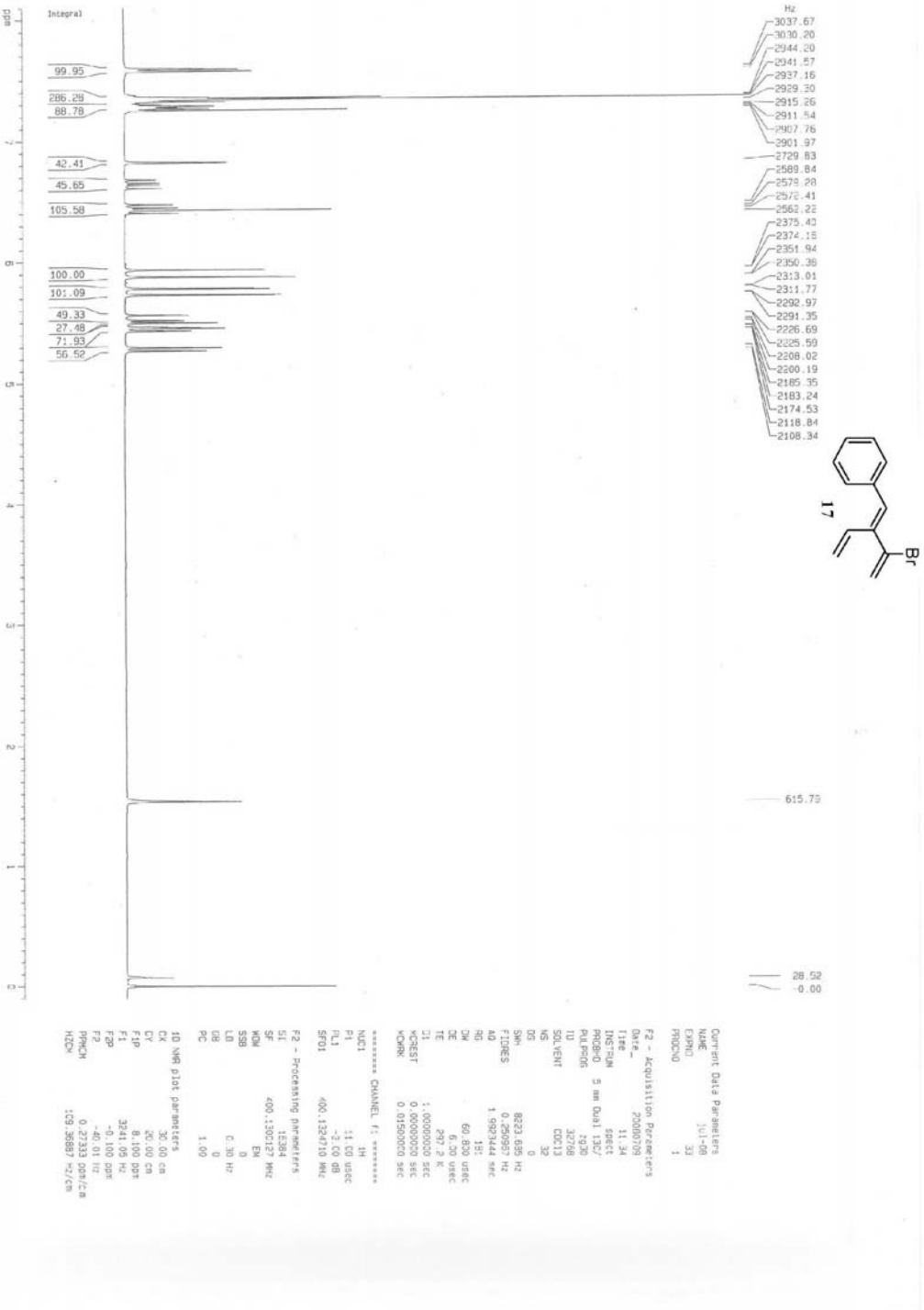

MeO2C
14
167.252
146.533
140.462
130.399
130.366
129.124
127.987
127.202
118.146
87.253
77.771
77.453
77.135
75.582
52.495
ppm

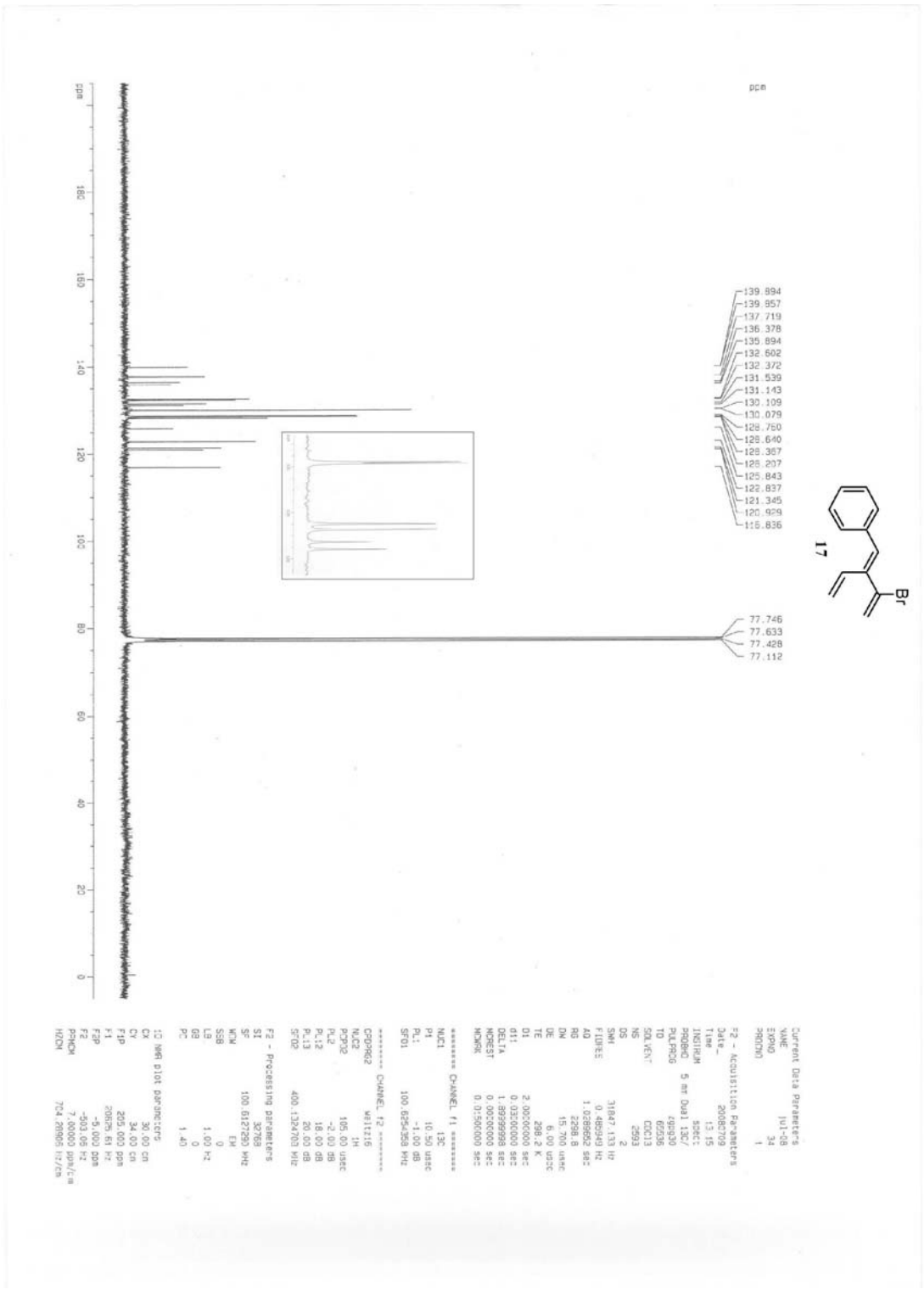

MeO2C
OH
O
O
N
15

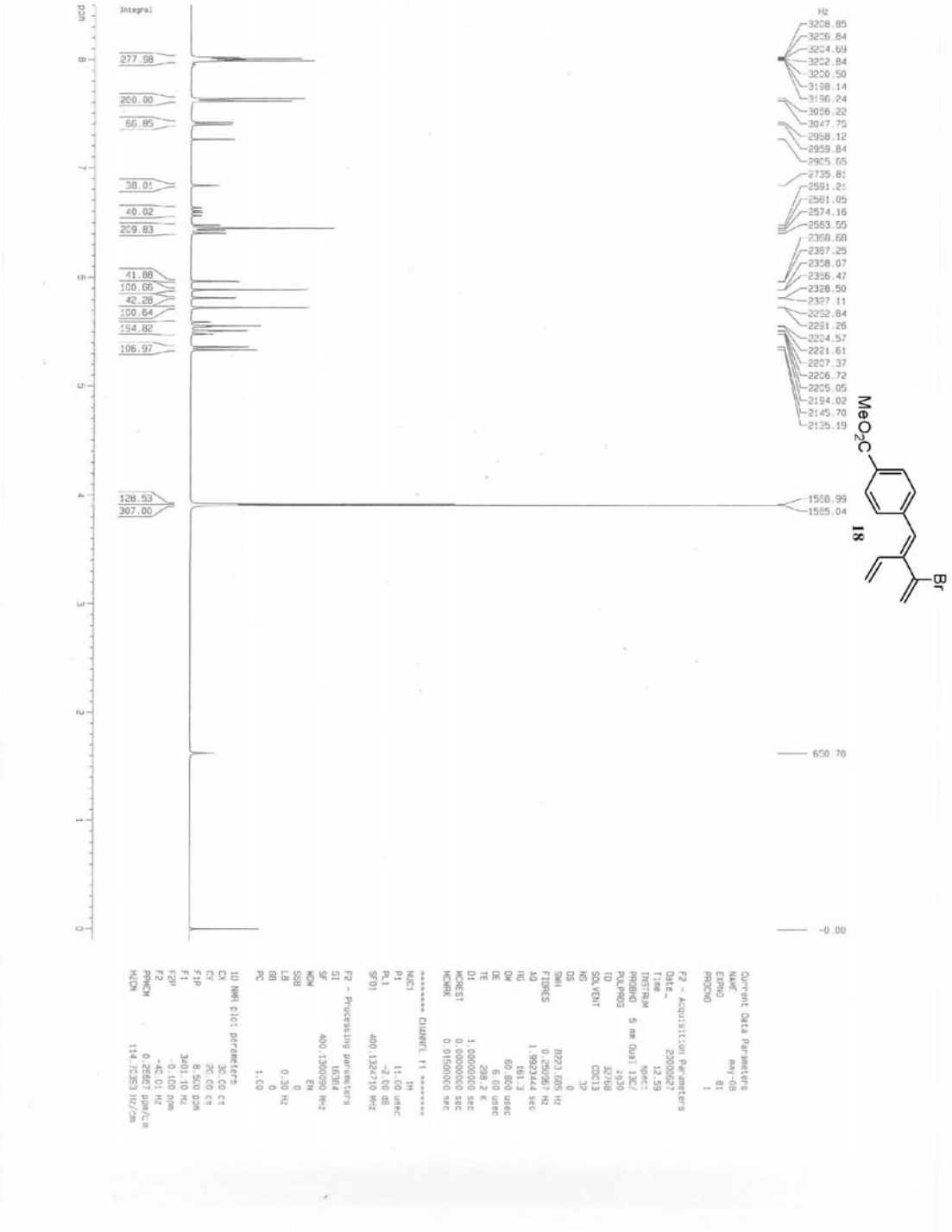

ppm
179.694
177.366
176.755
171.565
167.260
167.184
147.821
147.100
141.159
140.603
134.321
133.793
130.039
129.793
126.990
126.886
126.651
124.162
116.813
116.253
77.811
77.493
77.174
74.846
73.860
50.823
52.531
47.914
47.431
39.423
39.040
25.762
25.608
23.462
23.203
21.428
14.565
MeO2C
OH
15
O
O
N
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
d11
DELTA
MCREST
MCWRK
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM

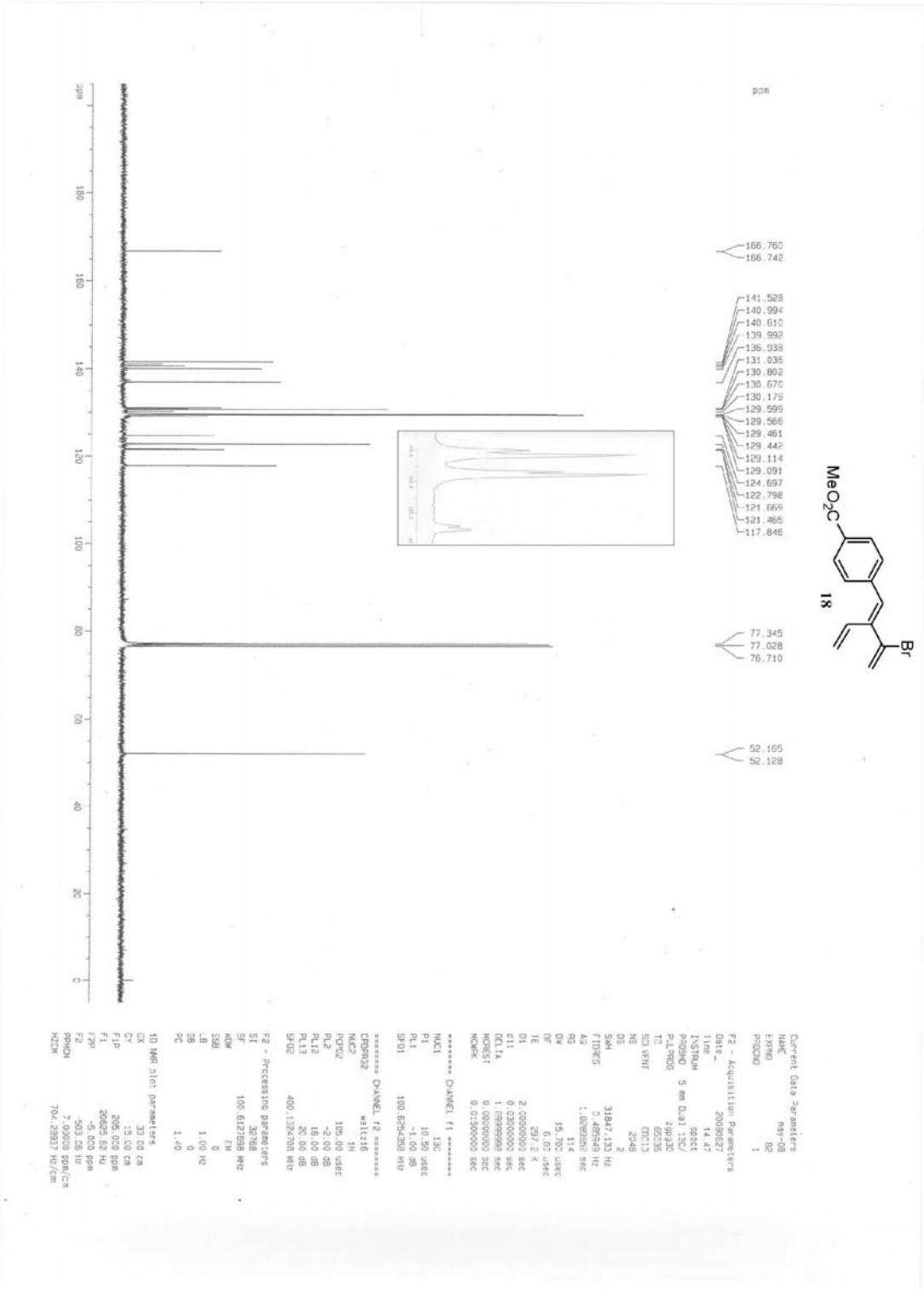

16
Cl


ppm
140.784
138.561
138.031
137.844
136.493
135.893
135.683
132.960
132.291
131.872
130.149
129.825
128.721
128.604
128.380
128.149
120.992
118.539
116.768
116.635
77.738
77.625
77.422
77.104
16
Cl
Current Data Parameters
NAME jul08
EXPNO 36
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080709
Time 16.37
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1266
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289152 sec
RG 161.3
DW 15.700 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
D0REST 0.00000000 sec
D0WAIT 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 16.50 usec
PL1 -1.00 dB
SFO1 100.6254868 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324708 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 225.000 ppm
F1 20525.61 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.00000 ppm/cm
HZCM 704.28906 Hz/cm


Integral
99.95
286.28
88.78
42.41
45.65
105.58
100.00
101.09
49.33
27.48
71.93
56.52
Hz
3037.67
3030.20
2944.20
2941.57
2937.16
2929.30
2915.26
2911.54
2907.76
2901.97
2729.83
2589.84
2579.28
2572.41
2562.22
2375.40
2374.15
2351.94
2350.38
2313.01
2311.77
2292.97
2291.35
2226.69
2225.59
2208.02
2200.19
2185.35
2183.24
2174.53
2118.84
2108.34
17
Br
615.79
28.52
0.00
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
HCREST
HCHARK
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM


ppm
139.894
139.957
137.719
136.378
135.894
132.602
132.372
131.539
131.143
130.109
130.079
128.760
128.640
128.367
128.207
125.843
122.837
121.345
120.929
115.836
77.746
77.633
77.428
77.112
17
Br
ppm
Current Data Parameters
NAME jul-08
EXPNO 34
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080709
Time 13.15
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2593
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0288682 sec
RG 2288.8
DW 15.700 usec
DE 6.00 usec
TE 298.2 K
D1 2.00200000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00200000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254358 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324700 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 30.00 cm
CY 34.00 cm
F1P 205.000 ppm
F2P -5.000 ppm
PPMCM 7.00000 ppm/cm
HZCM 704.28906 Hz/cm


Integral
277.98
200.00
66.85
38.01
40.02
209.83
41.88
100.66
42.28
100.64
194.82
106.97
128.53
307.00
Hz
3208.85
3206.84
3204.69
3202.84
3200.50
3198.14
3196.24
3056.22
3047.75
2958.12
2959.84
2905.55
2735.81
2591.25
2581.05
2574.16
2563.55
2388.60
2387.25
2358.07
2356.47
2328.50
2327.11
2292.84
2291.26
2224.57
2221.61
2207.37
2206.72
2205.05
2194.02
2145.70
2135.19
1568.99
1565.04
650.70
-0.00
MeO2C
Br
18
Current Data Parameters
NAME may-08
EXPNO 81
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080527
Time 12.59
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 161.3
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SF01 400.1324710 MHz
F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.60
1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.25667 ppm/cm
HZCM 114.72653 Hz/cm



Current Data Parameters
NAME may-08
EXPNO 82
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080827
Time 14.47
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 31847.133 Hz
FIDRES 0.485549 Hz
AQ 1.0289856 sec
RG 114
DW 15.700 usec
DE 6.00 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.8999980 sec
D0 0.00000030 sec
MCREST 0.01500000 sec
MCWRK

======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254350 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324700 MHz

F2 - Processing parameters
SI 32768
SF 100.6127698 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 15.00 cm
F1P 205.000 ppm
F1 20625.62 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.00000 ppm/cm
HZCM 704.22937 Hz/cm