Supplementary Material (ESI) for Chemical Communications

Supporting Information

Stereocontrolled Synthesis of Carbocycles via Four Successive Pericyclic Reactions

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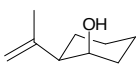
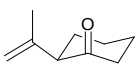
General

All reactions were performed under nitrogen or argon atmosphere in flame-dried glassware equipped with a magnetic stir bar and a rubber septum, unless otherwise indicated. All solvents were freshly distilled prior to use; diethyl ether and THF over sodium and benzophenone; toluene, DMF, triethylamine, DMSO, DME, and DCM over calcium hydride. $MgBr_2 \cdot OEt_2$ was prepared in our laboratory and stored in the glove box. All other commercial reagents were used without purification, unless otherwise noted. Reactions were monitored by thin layer chromatography (TLC) analysis of aliquots using glass sheets pre-coated (0.2 mm layer thickness) with silica gel 60 F_{254} (E. Merck). Thin layer chromatography plates were viewed under UV light and stained with phosphomolybdic acid or *p*-anisaldehyde staining solution. Column chromatographies were carried out with silica gel 60 (230-400 mesh, Merck). ^{1}H and ^{13}C NMR spectra were recorded in deuterated solvents, on Bruker AMX 300 MHz and Bruker AMX 500 MHz spectrometers. IR spectra were recorded with a Bomem Michaelson 100 FTIR spectrometer. HRMS were obtained on a Kratos Analytical Concept instrument (University of Ottawa Mass Spectrum Centre). Microwave reactions were conducted in a CEM Model ESP-1500 Plus oven, equipped with a EST-300 Plus temperature probe. All experiments were effected in a quartz tube in the presence of two carboflons.™

2-Isopropenyl-cyclohexanol and 2-Isopropenyl-cyclohexanone (A)

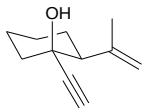


and


A dry round bottom flask was charged with CuBr·DMS (4.810 g, 23.4 mmol) and THF (300 mL). The solution was cooled to -30 °C, followed by the addition of isopropenylmagnesium bromide (608.0 mL, 0.304 mol). The mixture was stirred for 20 minutes, at which point cyclohexene oxide (22.96 g, 0.234 mol) was added and the solution was stirred to room temperature. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion.

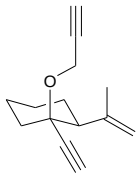
The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. A dry round bottom flask was charged with oxalyl chloride (9.69 mL, 0.111 mol) and DCM (200 mL). The solution was cooled to -78 °C, and DMSO (15.8 mL, 0.222 mol) was slowly added. After 20 minutes of stirring, the crude alcohol in DCM (20 mL) was added to give an opaque white mixture. The latter was stirred at -78 °C for 1.5 hours, followed by the addition of triethylamine (64.4 mL, 0.463 mol). The solution was again stirred at 0 °C for an additional hour and quenched with NH_4Cl (sat. aq.). The mixture was extracted with DCM (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (3-10% ethyl acetate in hexanes) afforded **A** (9.690 g, 76% over two steps) as a yellow oil. Characterization data is available through the literature.[1]

1-Ethynyl-2-isopropenyl-cyclohexanol (B)



To a solution of ketone **A** (1.055 g, 15.9 mmol) in THF (40 mL) at 0 °C was added dropwise ethynylmagnesium bromide (94.8 mL, 47.4 mmol). The solution was warmed to room temperature and allowed to stir overnight. The reaction was quenched with NH_4Cl (sat. aq.) and the mixture was extracted with diethyl ether (3x). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (5% ethyl acetate in hexanes) afforded the major diastereoisomer (*60:40*) **B** as a yellow oil (2.421 g, 59%). IR (neat, cm^{-1}) 3548 (m), 3468 (br), 3307 (s), 3079 (w), 2938 (s), 2856 (s), 1639 (m), 1447 (m), 1071 (m), 972 (s); ^1H NMR ($CDCl_3$, 300 MHz) δ 4.95 (s, 1H), 4.79 (s, 1H), 2.37 (s, 1H), 2.23 (s, 1H), 2.18-2.08 (m, 2 H), 1.92 (s, 3H), 1.72-1.37 (m, 6H), 1.28-1.12 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 148.4 (C), 112.8 (CH_2), 89.0 (C), 71.7 (CH), 67.4 (C), 52.8 (CH), 40.1 (CH_2), 27.0 (CH_2), 26.1 (CH_3), 26.1 (CH_2), 20.8 (CH_2); HRMS (EI) m/z calcd for $C_{11}H_{14}$ [$(M-H_2O)^+$] 146.1095, found 146.1095.

1-Ethynyl-2-isopropenyl-1-prop-2-ynyloxy-cyclohexane (7)

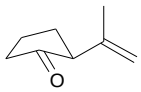


To a solution of **B** (2.545 g, 15.5 mmol) and propargyl bromide (5.16 mL, 46.0 mmol) in THF/DMF (9:3 mL) was added sodium hydride 60% in oil (1.860 g, 46.0 mmol) slowly at room temperature. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (5% ethyl acetate in hexanes) afforded **7** (2.568 g, 81%) as a yellow oil. IR (neat, cm^{-1}) 3303 (s), 3073 (m), 2934 (s), 2858 (s), 1641 (m), 1448 (m), 1376 (m), 1143 (m), 1094 (s), 1063 (s), 892 (s); ^1H NMR ($CDCl_3$) δ 4.81 (d, J = 1.2 Hz, 2H), 4.24 (dd, J_{AB} = 15.2 Hz, J_{AX} = 2.5 Hz, 1H), 4.13 (dd, J_{AB} = 15.2 Hz, J_{BX} = 2.5 Hz, 1H), 2.48 (s, 1H), 2.34 (t, J = 2.5 Hz, 1H), 2.25-2.12 (m, 2H), 1.86 (qd, J = 12.4, 3.7 Hz, 1H), 1.78 (s, 3H), 1.75-1.67 (m, 1H), 1.60-1.38 (m, 4H), 1.33-1.16 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 146.6 (C), 113.5 (CH_2), 84.2 (C), 80.9 (C), 75.9 (C), 74.9 (CH), 73.1 (CH), 54.3 (CH), 51.9 (CH_2), 35.8 (CH_2),

[1] (a) Onishi, T.; Nishida, T. *J. Chem. Soc.* **1978**, 651. (b) Fujita, Y.; Onishi, T.; Nishida, T. *Synthesis* **1978**, *12*, 934. (c) Utagawa, A.; Hirota, H.; Ohno, S.; Takahashi, T. *Bull. Chem. Soc. Jpn.* **1988**, *61*, 1207.

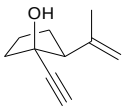
26.2 (CH_2), 25.7 (CH_2), 22.1 (CH_3), 20.6 (CH_2); HRMS (EI) m/z calcd for $C_{14}H_{18}O$ (M^+) 202.1358, found 202.1348.

2-Isopropenyl-cyclopentanone (C)



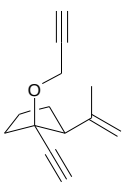
A dry round bottom flask was charged with CuBr·DMS (0.733 g, 3.57 mmoles) and THF (100 mL). The solution was cooled to -30 °C, followed by the addition of isopropenylmagnesium bromide (92.7 mL, 46.4 mmoles). The mixture was stirred for 20 minutes, at which point cyclopentene oxide (3.11 mL, 36.0 mmoles) was added and the solution was stirred to room temperature. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. The residue was re-dissolved in diethyl ether (50 mL) and Jones' reagent (20.6 mL, 53.5 mmoles) was slowly added at 0 °C. The reaction was allowed to reach room temperature. Upon completion, as observed by TLC analysis, the reaction was quenched with NH_4Cl (sat. aq.). The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (15% ethyl acetate in hexanes) afforded **C** (2.145 g, 48%) as a yellow oil. Characterization data is available through the literature.[2]

1-Ethynyl-2-isopropenyl-cyclopentanol (D)



To a solution of ketone **C** (2.145 g, 17.3 mmol) in THF (40 mL) at 0 °C was added dropwise ethynylmagnesium bromide (69.1 mL, 34.6 mmol). The reaction was warmed to room temperature and stirred until completion by TLC, at which point it was quenched with NH_4Cl (sat. aq.). The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (10% ethyl acetate in hexanes) afforded the major diastereoisomer (*66:34*) **D** as a yellow oil (0.989 g, 38%). IR (neat, cm^{-1}) 3496 (br), 3305 (s), 3085 (w), 2969 (s), 2923 (m), 2875 (m), 1640 (m), 1450 (m), 1376 (m), 1019 (m), 896 (m), 649 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 5.03 (s, 1H), 4.89 (s, 1H), 2.60 (m, 1H), 2.41 (s, 1H), 2.19-1.97 (m, 3H), 1.92 (s, 3H), 1.85-1.53 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 143.9 (C), 113.5 (CH), 87.9 (C), 73.2 (C), 71.8 (CH), 57.9 (CH), 42.2 (CH_2), 28.4 (CH_2), 25.2 (CH_3), 21.8 (CH_2); HRMS (EI) m/z calcd for $C_{10}H_{14}O$ (M^+) 150.1045, found 150.1016.

1-Ethynyl-2-isopropenyl-1-prop-2-ynyloxy-cyclopentane (14)

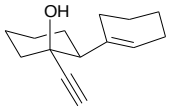


To a solution of **D** (2.070 g, 13.8 mmol) and propargyl bromide (4.61 mL, 41.4 mmol) in THF/DMF (30:10 mL) was added sodium hydride 60% in oil (1.656 g, 41.4 mmol) slowly at room temperature. The reaction was followed by TLC and quenched with a saturated aqueous solution of NH_4Cl upon

[2] Sate, T.; Takezoe, K. *Tetrahedron Lett.* **1991**, *32*, 4003.

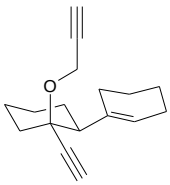
completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (5% ethyl acetate in hexanes) afforded **14** (2.234 g, 86%) as a yellow oil. IR (neat, cm^{-1}) 3297 (s), 3076 (w), 2970 (s), 2875 (m), 1640 (m), 1451 (m), 1375 (m), 1185 (m), 1065 (s), 894 (s); ^1H NMR ($CDCl_3$, 300 MHz) 4.88-4.86 (m, 2H), 4.25 (dd, J_{AB} = 15.5 Hz, J_{AX} = 25 Hz, 1H), 4.16 (dd, J_{AB} = 15.5 Hz, J_{BX} = 2.5 Hz, 1H), 2.62-2.54 (m, 1H), 2.54 (s, 1H), 2.33 (t, J = 2.5 Hz, 1H), 2.32-2.24 (m, 1H), 2.03-1.86 (m, 2H), 1.85 (s, 3H), 1.83-1.56 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 143.7 (C), 113.7 (CH_2), 84.2 (C), 82.6 (C), 81.4 (C), 75.2 (CH), 73.7 (CH), 58.3 (CH), 53.3 (CH_2), 38.9 (CH_2), 28.1 (CH_2), 23.0 (CH_3), 21.6 (CH_2); HRMS (EI) m/z calcd for $C_{13}H_{16}O$ (M^+) 188.1201, found 188.1117.

2-Ethynyl-bicyclohexyl-1'-en-2-ol (E)



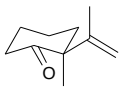
To a solution of 2-(1-cyclohexenyl)-cyclohexanone (3.99 g, 22.4 mmol) in THF (60 mL) at 0 °C was added dropwise ethynylmagnesium bromide (67.2 mL, 33.6 mmol). The reaction was warmed to room temperature and stirred until completion by TLC, at which point it was quenched with NH_4Cl (sat. aq.). The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (5% ethyl acetate in hexanes) afforded the major diastereoisomer (*60:40*) **E** as a white solid (2.454 g, 54%). IR (neat, cm^{-1}) 3439 (br), 3253 (m), 2939 (w), 1642 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 5.53 (s, 1H), 2.51-2.40 (m, 1H), 2.34 (s, 1H), 2.25 (s, 1H), 2.13-2.06 (m, 1H), 2.04-1.89 (m, 4H), 1.71-1.32 (m, 10H), 1.26-1.11 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) 140.8 (C), 123.8 (CH), 89.4 (C), 71.4 (CH), 67.6 (C), 53.0 (CH), 39.9 (CH_2), 32.3 (CH_2), 26.8 (CH_2), 26.3 (CH_2), 25.7 (CH_2), 23.4 (CH_2), 22.8 (CH_2), 20.9 (CH_2); HRMS (EI) m/z calcd for $C_{14}H_{20}O$ (M^+) 204.1514, found 204.1498; m.p. 77.5-78.3 °C.

2'-Ethynyl-2'-prop-2-ynyloxy-bicyclohexyl-1-ene (20)



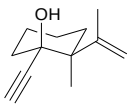
To a solution of **E** (0.800 g, 3.92 mmol) and propargyl bromide (1.53 mL, 13.7 mmol) in THF/DMF (9:3 mL) was added sodium hydride 60% in oil (0.548 g, 13.7 mmol) slowly at room temperature. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (5% ethyl acetate in hexanes) afforded **20** (0.938 g, 99%) as a colourless oil. IR (neat, cm^{-1}) 3307 (s), 2930 (s), 2856 (s), 1447 (m), 1137 (m), 1091 (m), 1072 (m), 1056 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 5.54 (s, 1H), 4.26 (dd, J_{AB} = 15.2 Hz, J_{AX} = 2.5 Hz, 1H), 4.15 (dd, J_{AB} = 15.2 Hz, J_{BX} = 2.5 Hz, 1H), 2.44 (s, 1H), 2.34 (t, J = 2.4 Hz, 1H), 2.17 (m, 2H), 1.98 (m, 4H), 1.85 (qd, J = 12.6, 3.5 Hz, 1H), 1.73-1.69 (m, 1H), 1.58-1.36 (m, 8H), 1.32-1.21 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 138.9 (C), 124.7 (CH), 84.8 (C), 81.5 (C), 76.7 (C), 75.1 (CH), 73.4 (CH), 55.3 (CH), 52.3 (CH_2), 36.5 (CH_2), 28.1 (CH_2), 26.5 (CH_2), 26.3 (CH_2), 25.8 (CH_2), 23.7 (CH_2), 23.0 (CH_2), 20.1 (CH_2); HRMS (EI) m/z calcd for $C_{17}H_{22}O$ (M^+) 242.1671, found 242.1625.

2-Isopropenyl-2-methyl-cyclohexanone (F)



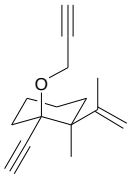
To a solution of sodium methoxide (5.330 g, 38.6 mmoles) in THF (100 mL) at 0 °C was added ketone **A** (2.633 g, 46.3 mmol) in THF (20 mL) *via* canula. The solution was stirred for 20 minutes and freshly distilled methyl iodide (2.40 mL, 38.6 mmoles) was added. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (3% hexanes in benzene) afforded **F** as a yellow oil. (3.73 g, 64%). IR (neat, cm^{-1}) 2967 (m), 2937 (s), 2865 (m), 1710 (s), 1638 (m), 1449 (m), 898 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 4.90 (s, 1H), 4.69 (s, 1H), 2.52-2.42 (m, 1H), 2.27-2.17 (m, 2H), 1.97-1.87 (m, 1H), 1.72-1.53 (m, 3H), 1.63 (s, 3H), 1.44-1.33 (m, 1H), 1.06 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 214.5 (C), 146.7 (C), 112.3 (CH_2), 55.2 (C), 39.8 (CH_2), 37.9 (CH_2), 27.9 (CH_2), 24.5 (CH_3), 21.8 (CH_2), 19.5 (CH_3); HRMS (EI) m/z calcd for $C_{10}H_{16}O$ (M^+) 152.1201, found 152.1329

1-Ethynyl-2-isopropenyl-2-methyl-cyclohexanol (G)



To a solution of ketone **F** (3.736 g, 24.6 mmol) in THF (100 mL) at room temperature was added dropwise ethynylmagnesium bromide (73.7 mL, 36.9 mmol). The reaction was warmed to room temperature and allowed to stir until completion by TLC, at which point it was quenched with NH_4Cl (sat. aq.). The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (10% ethyl acetate in hexanes) afforded the major diastereoisomer (*63:37*) **G** as a yellow oil (1.712 g, 39%). IR (neat, cm^{-1}) 2537 (br m), 2206 (m), 2933 (s), 2867 (m), 1625 (m), 1445 (m), 981 (s); ^1H NMR ($CDCl_3$, 300 MHz) δ 5.08 (s, 1H), 5.03 (s, 1H), 2.47 (s, 1H), 2.40 (s, 1H), 1.98 (s, 3H), 1.99-1.78 (m, 3H), 1.63-1.38 (m, 4H), 1.27 (s, 3H), 1.28-1.17 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 150.6 (C), 114.5 (CH_2), 88.3 (C), 72.4 (CH), 70.1 (C), 45.5 (C), 35.3 (CH_2), 31.1 (CH_2), 23.0 (CH_3), 20.9 (CH_2), 20.1 (CH_2), 20.0 (CH_3); HRMS (EI) m/z calcd for $C_{11}H_{15}O$ [$(M\text{-}CH_3)^+$] 163.1123, found 163.1130.

1-Ethynyl-2-isopropenyl-2-methyl-1-prop-2-ynyloxy-cyclohexane (17)

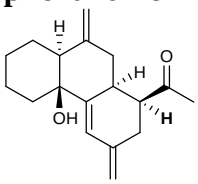


To a solution of **G** (1.712 g, 9.62 mmol) and propargyl bromide (5.36 mL, 48.1 mmol) in THF/DMF (18:6 mL) was added sodium hydride 60% in oil (1.924 g, 48.1 mmol) slowly at room temperature. The reaction was followed by TLC and quenched with NH_4Cl (sat. aq.) upon completion. The mixture was extracted with diethyl ether (3x) and the combined organic layers were dried over $MgSO_4$, filtered, and concentrated. Flash chromatography (10% hexanes in benzene) afforded **17** (1.89 g, 91%) as a yellow oil. IR (neat, cm^{-1}) 3302 (s), 3095 (w), 2933 (s), 2886 (m), 1630 (m), 1445 (m), 1374 (m), 1087 (s), 1066 (s), 895 (m); ^1H NMR ($CDCl_3$; 300 MHz) δ 5.01 (s, 1H), 4.94-4.93 (m, 1H), 4.26 (dd, J_{AB} = 15.5 Hz, J_{AX} = 2.5 Hz, 1H), 4.17 (dd, J_{AB} = 15.5 Hz, J_{BX} = 2.5 Hz, 1H), 2.54 (s, 1H), 2.33 (t, J = 2.5 Hz, 1H), 2.12-1.98 (m, 2H), 1.89 (s, 3H), 1.81-1.70 (m, 1H), 1.61-1.32 (m, 5H), 1.24

(s, 3H); ^{13}C NMR ($CDCl_3$; 75 MHz) δ 150.3 (C), 113.2 (CH_2), 83.7 (C), 81.1 (C), 80.5 (C), 76.5 (CH), 73.0 (CH), 52.0 (CH_2), 45.3 (C), 32.1 (CH_2), 31.4 (CH_2), 23.1 (CH_3), 22.3 (CH_3), 21.0 (CH_2), 20.8 (CH_2); HRMS (EI) m/z calcd for $C_{14}H_{17}O$ [$(M-CH_3)^+$] 201.1279, found 201.1272.

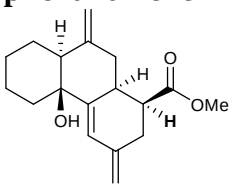
Typical procedure :

1-(4b-Hydroxy-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthren-1-yl)-ethanone (11)



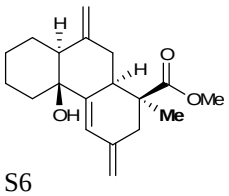
To a microwave quartz cell (100 mL) containing a glass-coated CARBOFLON^{TM} (2 cm) was added a solution of **7** (47 mg, 0.23 mmol) in toluene (10 mL) and triethylamine (2.3 mmol). After bubbling argon for 20 minutes, the solution was heated at 207ºC for 60 minutes using microwaves (600 W). The solution was cooled to room temperature and concentrated. The residue was dissolved in dichloromethane (2 mL) and added to a solution of 2,6-lutidine (0.11 mL, 0.92 mmol) and $MgBr_2$-OEt_2 (118.8 mg, 0.46 mmol) in dichloromethane (4 mL) previously stirred for 30 minutes. After stirring for 2 hours at room temperature, methyl vinyl ketone (40.3 mg, 0.575 mmol) was added. The solution was stirred for overnight and quenched with a saturated solution of ammonium chloride. The mixture was extracted with dichloromethane (3X) and the combined organic phases were dried over $MgSO_4$, filtered and concentrated. The residue was purified by flash chromatography (10% ethyl acetate in hexanes) to afford **11** (31 mg, 49%) as a yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.43 (s, 1H), 4.88 (s, 1H), 4.85-4.83 (m, 2H), 4.71 (s, 1H), 2.99 (quint., J = 6.2 Hz, 1H), 2.89 (quint., J = 5.6 Hz, 1H), 2.44 (d, J = 9.9 Hz, 1H) 2.39-2.08 (m, 4H), 2.18 (s, 3H), 1.91-1.25 (m, 9 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 209.4 (C), 149.7 (C), 145.7 (C), 141.3 (C), 125.2 (CH), 112.6 (CH_2), 109.9 (CH_2), 72.6 (C), 49.9 (CH), 46.6 (CH), 37.0 (CH_2), 32.9 (CH_2), 32.5 (CH), 28.8 (CH_3), 27.6 (CH_2), 25.7 (CH_2), 22.6 (CH_2), 21.4 (CH_2); IR (neat, cm^{-1}) 3498 (br), 3078 (w), 2931 (s), 2856 (m), 1709 (s), 1644 (m), 1608 (w), 1436 (m), 1355 (m), 971 (m), 888 (s); HRMS (EI) m/z calcd for $C_{18}H_{24}O_2$ (M^+) 272.1776, found 272.1813.

4b-Hydroxy-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthrene-1-carboxylic acid methyl ester (9)



IR (neat, cm^{-1}) 3517 (br), 3079 (w), 2931 (s), 2856 (m), 1737 (s), 1644 (m), 1608 (w), 1437 (m), 1276 (m), 1203 (m), 1165 (m), 891 (m); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.42 (s, 1H), 4.89 (s, 1H), 4.85 (s, 2H), 4.71 (s, 1H), 3.69 (s, 3H), 2.96 (quint., J = 6.2 Hz, 1H), 2.84 (quint., J = 5.6 Hz, 1H), 2.48-2.11 (m, 6H), 1.90-1.40 (m, 8H); ^{13}C NMR ($CDCl_3$, 75 Hz) δ 174.4 (C), 150.2 (C), 146.4 (C), 141.5 (C), 125.3 (CH), 113.0 (CH_2), 110.1 (CH_2), 73.0 (C), 52.0 (CH_3), 46.9 (CH), 42.4 (CH), 37.3 (CH_2), 33.5 (CH_2), 33.0 (CH), 28.5 (CH_2), 26.1 (CH_2), 23.0 (CH_2), 21.8 (CH_2); HRMS (EI) m/z calcd for $C_{18}H_{22}O_2$ [$(M-H_2O)^+$] 270.1620, found 270.1565.

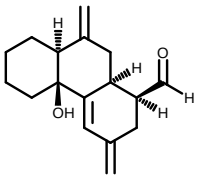
4b-Hydroxy-1-methyl-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthrene-1-carboxylic acid methyl ester (10)



IR (neat, cm^{-1}) 3524 (br), 3078 (w), 2933 (s), 2857 (m), 1732 (s), 1644 (m), 1435 (m), 1255 (m), 1236 (m), 1109 (m), 891 (m); ^{1}H NMR

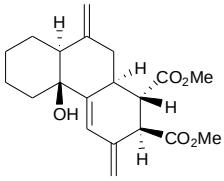
($CDCl_3$, 300 MHz) δ 6.38 (s, 1H), 4.93 (s, 1H), 4.88 (s, 1H), 4.82 (s, 1H), 4.68 (s, 1H), 3.67 (s, 3H), 2.58-2.50 (m, 2H), 2.46-2.36 (m, 2H), 2.19 (d, J = 14.9 Hz, 1H), 2.15-2.04 (m, 1H), 1.86-1.25 (m, 9H), 1.16 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 148.3 (C), 146.1 (C), 140.3 (C), 123.6 (CH), 113.5 (CH_2), 109.1 (CH_2), 76.4 (C), 72.4 (C), 51.5 (CH_3), 46.4 (CH), 43.7 (C), 39.7 (CH), 36.5 (CH_2), 34.8 (CH_2), 34.2 (CH_2), 25.5 (CH_2), 23.1 (CH_3), 22.4 (CH_2), 21.2 (CH_2); HRMS (EI) m/z calcd for $C_{19}H_{26}O_3$ (M^+) 302.1882, found 302.1863.

4b-Hydroxy-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthrene-1-carbaldehyde (12)



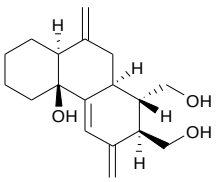
IR (neat, cm^{-1}) 3476 (br), 2931 (s), 2854 (m), 2718 (w), 1721 (s), 1642 (m), 1445 (m), 1073 (m), 890 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 9.75 (d, J = 1.2 Hz, 1H), 6.44 (d, J = 1.9 Hz, 1H), 4.93 (s, 1H), 4.91 (s, 1H), 4.88-4.87 (m, 1H), 4.78-4.77 (m, 1H), 3.02 (quint., J = 6.8 Hz, 1H), 2.76-2.69 (m, 1H), 2.55-2.29 (m, 4H), 1.99 (d, J = 13.0 Hz, 1H), 1.84-1.41 (m, 8H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 203.6 (CH), 148.1 (C), 145.8 (C), 139.9 (C), 125.1 (CH), 113.4 (CH_2), 109.7 (CH_2), 71.8 (C), 49.1 (CH), 45.4 (CH), 36.7 (CH_2), 33.1 (CH_2), 32.1 (CH), 27.8 (CH_2), 25.3 (CH_2), 23.2 (CH_2), 21.2 (CH_2); HRMS (EI) m/z calcd for $C_{17}H_{22}O_2$ (M^+) 258.1620, found 258.1634.

4b-Hydroxy-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthrene-1,2-dicarboxylic acid dimethyl ester (13)



IR (neat, cm^{-1}) 3535 (br), 3084 (w), 2931 (m), 2855 (w), 1739 (s), 1643 (w), 1436 (m), 1280 (m), 1260 (m), 1225 (m), 1165 (m), 894 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 6.41 (s, 1H), 5.03 (d, J = 2.5 Hz, 1H), 4.91 (s, 1H), 4.81 (s, 2H), 3.72 (s, 3H), 3.70 (s, 3H), 3.59-3.53 (m, 1H), 2.78-2.64 (m, 3H), 2.30-2.17 (m, 2H), 2.10-2.02 (m, 1H), 1.84-1.69 (m, 2H), 1.66-1.29 (m, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.8 (C), 173.1 (C), 147.4 (C), 145.8 (C), 139.6 (C), 125.3 (CH), 113.6 (CH_2), 110.3 (CH_2), 71.3 (C), 52.5 (CH_3), 52.4 (CH_3), 50.0 (CH), 48.5 (CH), 44.9 (CH), 38.6 (CH_2), 35.8 (CH_2), 35.5 (CH), 25.6 (CH_2), 24.1 (CH_2), 22.0 (CH_2); HRMS (EI) m/z calcd for $C_{20}H_{24}O_4$ [$(M- H_2O)^+$] 328.1675, found 328.1668.

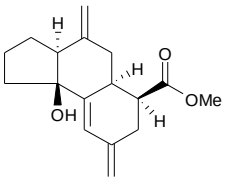
7,8-Bis-hydroxymethyl-6,10-dimethylene-1,3,4,6,7,8,8a,9,10,10a-decahydro-2H-phenanthren-4a-ol.



IR (neat, cm^{-1}) 3390 (br), 2929 (s), 2850 (m), 1707 (m), 1651 (m), 1447 (m), 1034 (m), 890 (w); ^1H NMR ($CDCl_3$; 300 MHz) δ 6.37 (d, J = 1.9 Hz, 1H), 4.99 (s, 1H), 4.93 (s, 1H), 4.87 (s, 1H), 4.74 (s, 1H), 3.43-3.57 (m, 4H), 2.84-2.74 (m, 1H), 2.58 (quint., J = 6.8 Hz, 1H), 2.44 (q, J = 6.2 Hz, 1H), 2.36 (t, J = 7.4 Hz, 1H), 2.15-1.22 (m, 13H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 149.0 (C), 146.5 (C), 142.6 (C), 124.6

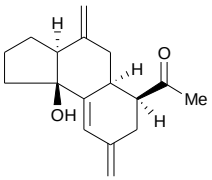
(CH), 112.3 (CH_2), 109.2 (CH_2), 71.9 (C), 63.9 (CH_2), 63.1 (CH_2), 45.7 (CH), 43.3 (CH), 43.3 (CH), 38.0 (CH_2), 37.4 (CH_2), 32.3 (CH), 25.5 (CH_2), 22.8 (CH_2), 21.6 (CH_2); HRMS (EI) m/z calcd for $C_{18}H_{24}O_2$ [$(M-H_2O)^+$] 272.1776, found 272.1738.

9b-Hydroxy-4,8-dimethylene-2,3,3a,4,5,5a,6,7,8,9b-decahydro-1H-cyclopenta[a]naphthalene-6-carboxylic acid methyl ester (15)



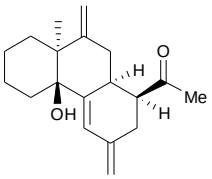
IR (neat, cm^{-1}) 3490 (m, br), 3080 (w), 2952 (s), 2870 (m), 1729 (s), 1647 (m), 1429 (m), 1196 (s), 1172 (s), 882 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 6.21 (s, 1H), 4.93 (s, 1H), 4.90 (s, 1H), 4.85 (s, 1H), 4.77 (s, 1H), 3.61 (s, 3H), 2.93-2.89 (m, 1H), 2.78-2.60 (m, 4H), 2.46-2.37 (m, 2H), 2.18 (br. s, 1H), 2.10-1.71 (m, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.6 (C), 146.7 (C), 143.3 (C), 140.0 (C), 124.3 (CH), 113.4 (CH_2), 109.5 (CH_2), 80.5 (C), 51.5 (CH_3), 49.4 (CH), 43.1 (CH), 36.0 (CH), 35.9 (CH_2), 34.4 (CH_2), 33.4 (CH_2), 25.6 (CH_2), 21.3 (CH_2); HRMS (EI) m/z calcd for $C_{17}H_{22}O_3$ (M^+) 274.1569, found 274.1583.

1-(9b-Hydroxy-4,8-dimethylene-2,3,3a,4,5,5a,6,7,8,9b-decahydro-1H-cyclopenta[a]naphthalen-6-yl)-ethanone (16)



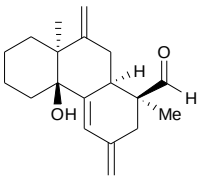
IR (neat, cm^{-1}) 3486 (br, m), 2986 (w), 2945 (m), 2859 (m), 1700 (s), 1644 (m), 1361 (m), 1160 (m), 884 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 6.14 (s, 1H), 4.91 (s, 1H), 4.87 (s, 1H), 4.84 (s, 1H), 4.77 (s, 1H), 2.98-2.94 (m, 1H), 2.79-2.74 (m, 4H), 2.58 (s, 1H), 2.45-2.36 (m, 1H), 2.31 (d, J = 11.2 Hz, 1H), 2.11 (s, 3H), 2.06-1.91 (m, 3H), 1.84-1.69 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 209.6 (C), 146.8 (C), 144.3 (C), 139.7 (C), 123.7 (CH), 113.5 (CH_2), 109.5 (CH_2), 80.5 (C), 50.9 (CH), 49.4 (CH), 36.0 (CH), 35.8 (CH_2), 32.9 (CH_2), 28.8 (CH_2), 25.6 (CH_3), 21.3 (CH_2); HRMS (EI) m/z cald for $C_{17}H_{22}O_2$ (M^+) 258.1620, found 258.1620.

1-(4b-Hydroxy-8a-methyl-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthren-1-yl)-ethanone (18)



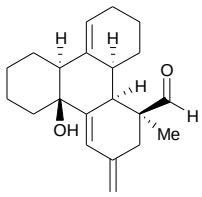
IR (neat, cm^{-1}) 3461 (br w), 2927 (s), 2859 (m), 1707 (m), 1445 (w) 1336 (w), 885 (m); ^1H NMR ($(CD_3)_2CO$, 500 MHz) δ 6.26 (s, 1H), 5.01 (s, 1H), 4.86 (s, 1H), 4.83 (s, 1H), 4.68 (s, 1H), 3.38 (d, J = 11.6 Hz, 1H), 2.71-2.65 (m, 1H), 2.55-2.48 (m, 1H), 2.41-2.37 (m, 1H), 2.23-2.11 (m, 1H), 2.15 (s, 3H) 1.98-1.90 (m, 2H), 1.78-1.46 (m, 8H), 1.02 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 210.2 (C), 154.9 (C), 147.7 (C), 140.7 (C), 123.6 (CH), 113.8 (CH_2), 108.5 (CH_2), 73.8 (C), 54.9 (CH), 41.6 (C), 36.2 (CH_2), 34.5 (CH_2), 33.6 (CH), 32.6 (CH_2), 30.4 (CH_2), 26.7 (CH_3), 25.9 (CH_3), 20.8 (CH_2 x 2); HRMS (EI) m/z calcd for $C_{19}H_{26}O_2$ (M^+) 286.1933, found 286.1919.

4b-Hydroxy-1,8a-dimethyl-3,9-dimethylene-1,2,3,4b,5,6,7,8,8a,9,10,10a-dodecahydro-phenanthrene-1-carbaldehyde (19)



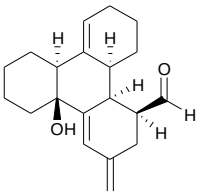
IR (neat, cm^{-1}) 3463 (br w), 2930 (s), 2861 (m), 1718 (s), 1634 (w), 1458 (w), 1444 (w), 1372 (w), 888 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 9.48 (s, 1H), 6.24 (d, J = 2.5 Hz, 1H), 4.92 (s, 1H), 4.89-4.85 (m, 3H), 2.95 (dddd, J = 14.3, 14.3, 2.5, 2.5 Hz, 1H), 2.69-2.35 (m, 5H), 2.07 (br. s, 1H), 1.95-1.89 (m, 1H), 1.78-1.52 (m, 6H), 1.18 (s, 3H), 1.01 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 205.3 (CH), 155.3 (C), 143.9 (C), 139.7 (C), 124.7 (CH), 113.9 (CH_2), 108.8 (CH_2), 73.7 (C), 48.8 (C), 41.6 (C), 40.9 (CH, CH_2), 32.9 (CH_2), 30.8 (CH_2), 30.3 (CH_2), 26.2 (CH_3), 21.0 (CH_2), 20.9 (CH_2), 20.3 (CH_3); HRMS (EI) m/z calcd for $C_{19}H_{26}O_2$ (M^+) 286.1933, found 286.1928.

4b-Hydroxy-1-methyl-3-methylene-1,2,3,4b,5,6,7,8,8a,10,11,12,12a,12b-tetradecahydro-triphenylene-1-carbaldehyde (21)



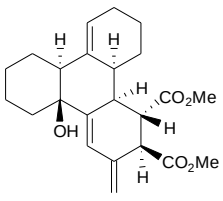
IR (neat, cm^{-1}) 3454 (br w), 2929 (s), 2858 (m), 1720 (s), 1449 (w); ^1H NMR ($CDCl_3$, 300 MHz) δ 9.74 (s, 1H), 6.11 (s, 1H), 5.53 (s, 1H), 4.97 (s, 1H), 4.93 (s, 1H), 2.66 (d, J = 11.2 Hz, 1H), 2.36 (q, J = 14.7 Hz, 1H), 2.18-2.08 (m, 1H), 2.03-1.97 (m, 2H), 1.91 (d, J = 11.8 Hz, 1H), 1.84-1.06 (m, 14H), 1.01 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 205.0 (CH), 146.9 (C), 140.2 (C), 139.2 (C), 123.2 (CH), 120.6 (CH), 114.2 (CH_2), 72.5 (C), 51.7 (CH), 48.9 (C), 47.1 (CH), 43.4 (CH), 34.0 (CH_2), 33.2 (CH_2), 29.7 (CH_2), 25.7 (CH_2), 25.6 (CH_2), 24.2 (CH_2), 21.4 (CH_2), 21.3 (CH_3), 20.7 (CH_2); HRMS (EI) m/z calcd for $C_{21}H_{28}O_2$ (M^+) 312.2089, found 312.2077.

4b-Hydroxy-1,8a-dimethyl-3-methylene-1,2,3,4b,5,6,7,8,8a,10,11,12,12a,12b-tetradecahydro-triphenylene-1-carbaldehyde (22)



IR (neat, cm^{-1}) 3423 (br w), 2930 (s), 2856 (m), 1718 (m), 1447 (w), 896 (w); ^1H NMR ($CDCl_3$; 300 MHz) δ 9.69 (d, J = 4.3 Hz, 1H), 6.32 (d, 1.9 Hz, 1H), 5.64-5.59 (m, 1H), 4.96 (s, 1H), 4.90 (s, 1H), 2.84 (quint., J = 4.3 Hz, 1H), 2.69-2.62 (m, 1H), 2.54-2.50 (m, 2H), 2.12-1.17 (m, 17H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 205.5 (CH), 144.6 (C), 139.3 (C), 138.3 (C), 123.4 (CH), 123.3 (CH), 114.2 (CH_2), 72.0 (C), 48.9 (CH), 47.2 (CH), 42.3 (CH), 39.6 (CH), 34.2 (CH_2), 31.3 (CH_2), 27.8 (CH_2), 25.6 (CH_2), 25.5 (CH_2), 24.1 (CH_2), 21.5 (CH_2), 20.7 (CH_2); HRMS (EI) m/z calcd for $C_{20}H_{24}O$ [$(M-H_2O)^+$] 280.1827, found 280.1824.

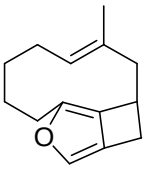
4b-Hydroxy-3-methylene-1,2,3,4b,5,6,7,8,8a,10,11,12,12a,12b-tetradecahydro-triphenylene-1,2-dicarboxylic acid dimethyl ester (23)



IR (neat, cm^{-1}) 3533 (br w), 2931 (m), 2858 (m), 1739 (s), 1436 (m), 1265 (m), 1167 (m); ^1H NMR ($CDCl_3$, 300 MHz) δ 6.47 (s, 1H), 5.36 (s, 1H), 4.97 (s, 1H), 4.71 (s, 1H), 3.74 (s, 3H), 3.69 (s, 3H), 3.47 (d, J = 11.2 Hz, 1H), 3.18-3.04 (m, 2H), 2.61-2.50 (m, 1H), 2.32 (d, J = 11.8 Hz, 1H), 2.19-1.99 (m, 3H), 1.88-1.17 (m, 11H), 0.97-0.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.7 (C), 173.0 (C), 147.3 (C), 139.6 (C), 139.5 (C), 127.0 (CH), 121.3 (CH), 111.8 (CH_2), 71.3 (C), 52.1 (CH_3), 51.7 (CH_3), 48.2 (CH), 44.9 (CH), 43.5 (CH), 39.1 (CH_2), 38.5 (CH), 38.0 (CH), 25.4 (CH_2),

24.2 (CH_2), 24.1 (CH_2), 22.1 (CH_2 x 2), 21.5 (CH_2); HRMS (EI) m/z calcd for $C_{23}H_{30}O_5$ (M^+) 386.2093, found 386.2071.

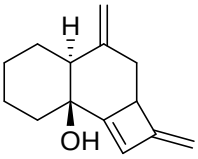
7-Methyl-13-oxa-tricyclo[7.4.1.0^{11,14}]tetradeca-1(14),6,11-triene (24)



To a solution of **7** (51.5 mg, 0.255 mmol) in toluene (12 mL) in a microwave cell was added Et_3N (0.355 mL, 2.55 mmol). The solution was degassed with argon (30 minutes) and subsequently heated with microwaves at 200 °C for 35 minutes. The solution was concentrated and once it was cooled to room temperature, it was put under the pump (very important as the compound is somewhat volatile). Purification by flash chromatography (5 % ethyl acetate/ 95 % hexanes) gave the tandem product **8** (yield not calculated since it is unstable on silica gel) along with the tricyclic compound **24** (13.9 mg, 27% yield) and tricyclic compound **25** (2.2 mg, 4 % yield).

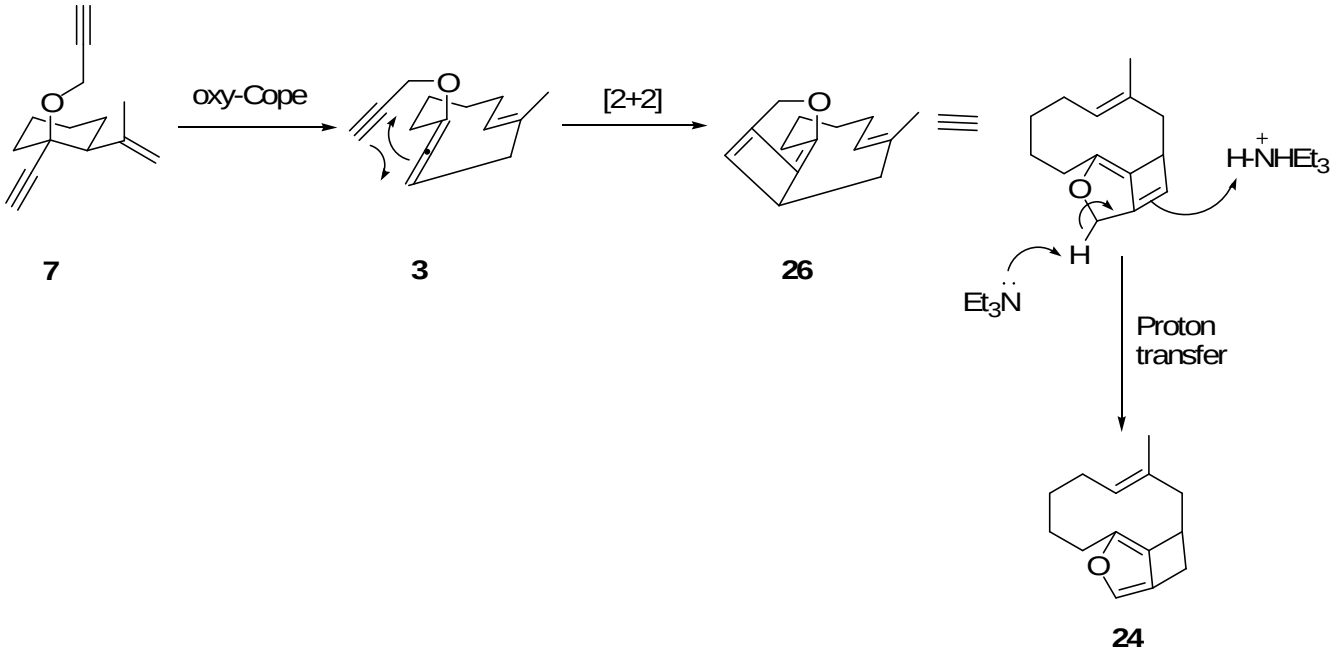
24 : IR (neat, cm^{-1}) 2928 (s), 2856 (m), 1570 (w), 1436 (m), 1222 (w), 976 (w), 727 (w); ^1H NMR ($CDCl_3$, 400 MHz) δ 6.88 (s, 1H), 4.99 (t, J=7.5 Hz, 1H), 3.72-3.66 (m, 1H), 3.25 (ddd, J=13.7, 5.7, 1.2 Hz, 1H), 2.66 (ddd, J=13.7, 3.1, 1.1 Hz, 1H), 2.62-2.49 (m, 3H), 2.03-1.87 (m, 4H), 1.82-1.73 (m, 1H), 1.69 (s, 3H), 1.41-1.32 (m, 1H), 1.11-1.01 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 146.7 (C), 133.9 (C), 129. 6 (CH), 128.7 (CH), 124.8 (C), 124.5 (C), 45.0 (CH_2), 41.2 (CH), 32.1 (CH_2), 28.7 (CH_2), 28.5 (CH_2), 26.7 (CH_2), 22.6 (CH_2), 16.3 (CH_3); HRMS (EI) m/z calcd for $C_{14}H_{17}O$ $(M-H)^+$ 201.1274, found 201.1275.

2,4-Dimethylene-2a,3,4,4a,5,6,7,8-octahydro-2H-cyclobuta[a]naphthalen-8a-ol (25)



IR (neat, cm^{-1}) 3462 (br), 3079 (w), 2932 (s), 2854 (m), 1718 (w), 1667 (w), 1446 (w), 1276 (w), 890 (w); ^1H NMR ($CDCl_3$, 500 MHz) δ 5.93 (s, 1H), 5.00 (d, J=1.6 Hz, 1H), 4.75 (d, J=1.0Hz, 1H), 4.60 (d, J=1.2 Hz, 1H), 4.50 (s, 1H), 3.12 (dd, J=10.4, 6.7 Hz, 1H), 2.68 (dd, J=12.4, 6.8 Hz, 1H), 2.05-1.96 (m, 4H), 1.83-1.79 (m, 1H), 1.71-1.56 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 161.3 (C), 150.3 (C), 147.6 (C), 123.3 (CH), 110.9 (CH_2), 97.2 (CH_2), 70.1 (C), 51.3 (CH), 46.3 (CH), 41.3 (CH_2), 31.2 (CH_2), 25.7 (CH_2), 23.4 (CH_2), 20.6 (CH_2); HRMS (EI) m/z calcd for $C_{14}H_{18}O$ $(M)^+$ 202.1358, found 202.1358.

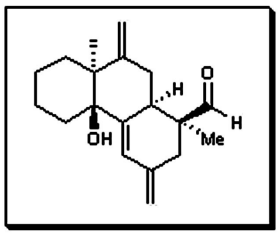
The proposed mechanism for **24 :**



7 **3** **26** **24**

The proposed mechanism for **25:**

8 **25**



13C with proton decoupling

ppm
205.32
155.343
143.878
139.741
124.694
113.924
108.750
77.424
77.001
76.577
73.740
48.846
41.618
40.899
32.931
30.765
30.295
26.208
20.975
20.929
20.278

ppm 200 175 150 125 100 75 50 25

Current Data Parameters
NAME rc_668p
EXPNO 2
PROCNO 1

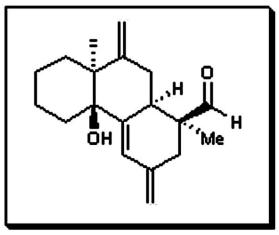
F2 - Acquisition Parameters
Date_ 20040317
Time 14.41
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 252
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 5160.6
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

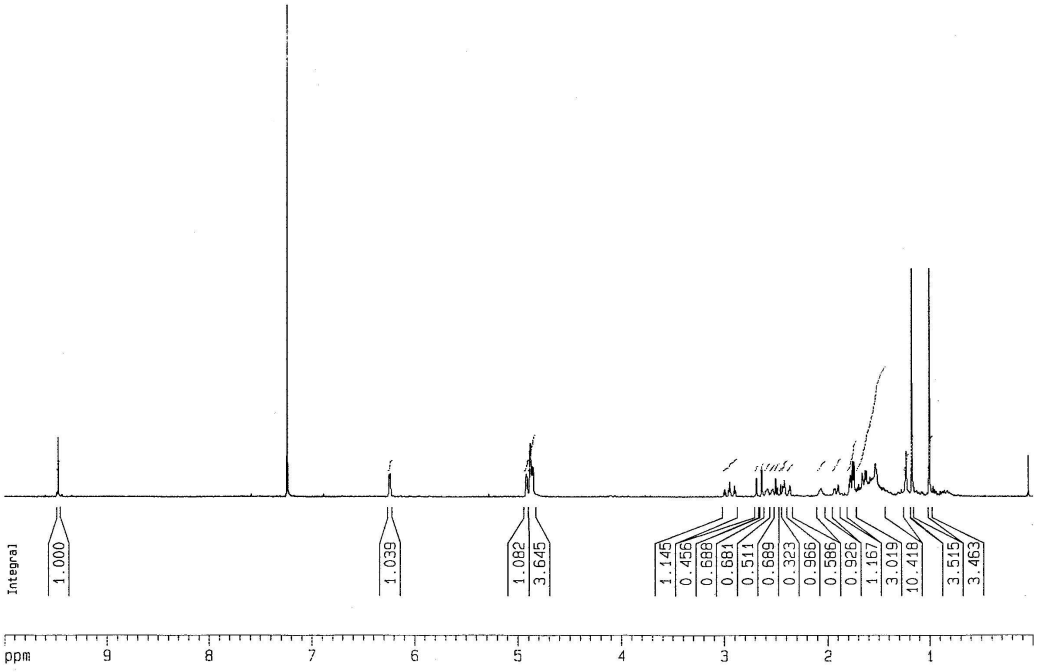
========== CHANNEL f1 ==========
NUC1 13C
P1 5.00 usec
PL1 -6.00 dB
SFO1 75.4752653 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SFO2 300.1314860 MHz

F2 - Processing parameters
SI 65536
SF 75.4677481 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm



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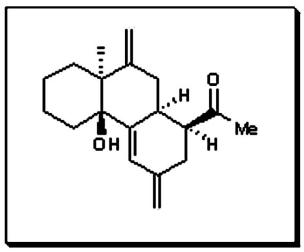
Current Data Parameters
NAME                  rc_668pb
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20040401
Time                      9.49
INSTRUM                 av300
PROBHD    5 mm QNP  1H/1
PULPROG                  zg30
TD                      30720
SOLVENT                 CDCl3
NS                          32
DS                           0
SWH                  5081.301 Hz
FIDRES               0.165407 Hz
AQ                3.0228980 sec
RG                       912.3
DW                     98.400 usec
DE                      6.00 usec
TE                      300.0 K
D1                 1.00000000 sec

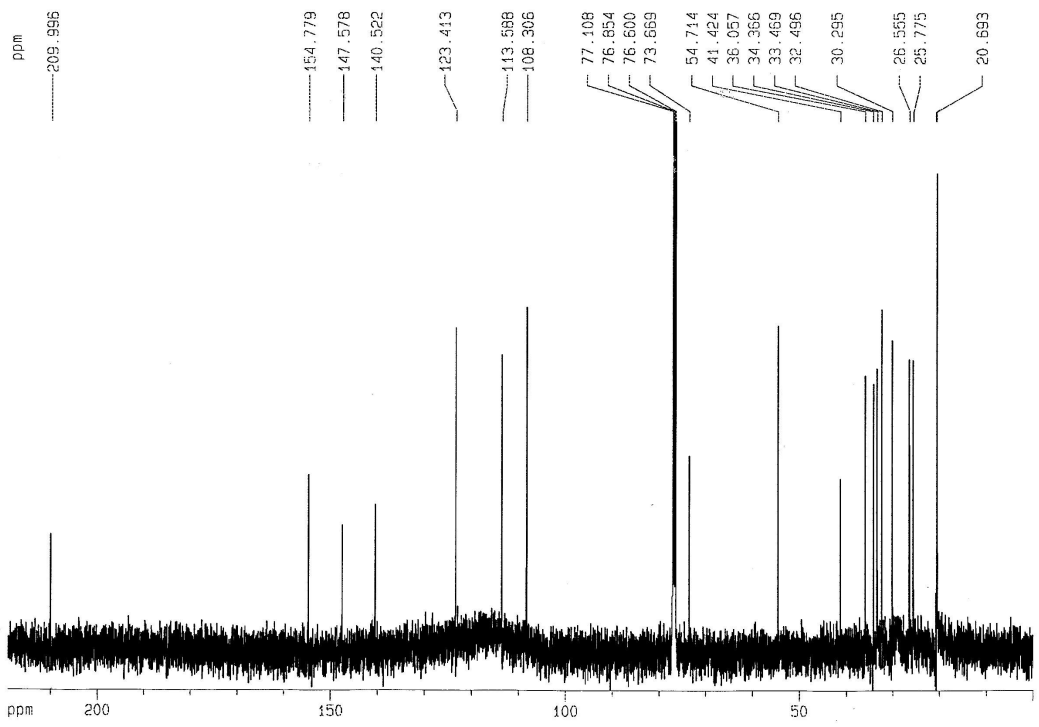
============ CHANNEL f1 =============
NUC1                        1H
P1                       10.50 usec
PL1                      -3.00 dB
SFO1               300.1319477 MHz

F2 - Processing parameters
SI                          65536
SF                 300.1300000 MHz
WDW                            EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                     10.000 ppm
F1                     3001.30 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.50000 ppm/cm
HZCM                150.06500 Hz/cm
```



13C with proton decoupling



Current Data Parameters
NAME rc_670c13
EXPNO 1
PROCNO 1

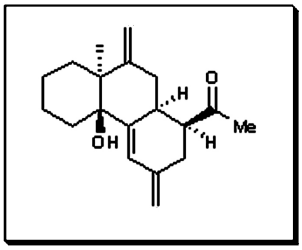
F2 - Acquisition Parameters
Date_ 20040331
Time 15.27
INSTRUM AV500WB
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2661
DS 0
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 3251
DW 16.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

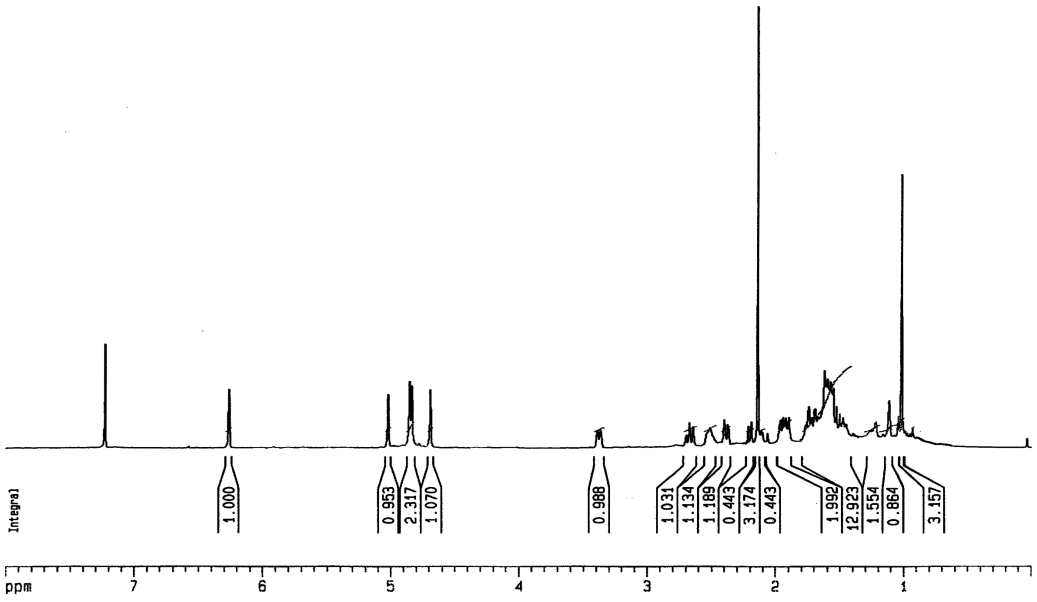
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -3.00 dB
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 20.64 dB
PL13 23.56 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578051 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 80.00 cm
F1P 220.000 ppm
F1 27666.72 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1383.33582 Hz/cm





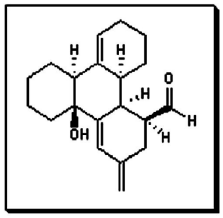

```
Current Data Parameters
NAME                  rc_670p1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20040322
Time                      9.24
INSTRUM                AV500MB
PROBHD   5 mm BBI 1H-BB
PULPROG                   zg30
TD                       65536
SOLVENT                Acetone
NS                            32
DS                             0
SWH                   7440.476 Hz
FIDRES                0.113533 Hz
AQ                    4.4040694 sec
RG                        45.25
DW                       67.200 usec
DE                        6.00 usec
TE                       300.0 K
D1                  0.01000000 sec

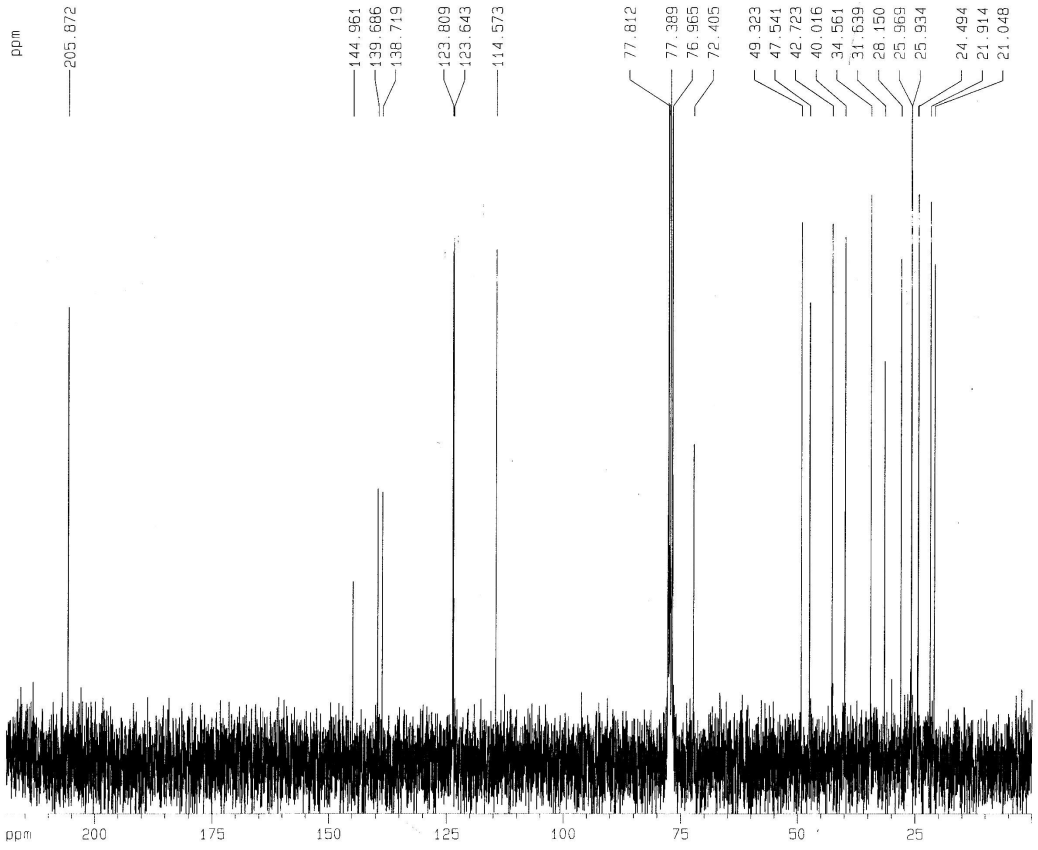
======== CHANNEL f1 ========
NUC1                         1H
P1                        10.50 usec
PL1                        3.00 dB
SFO1                500.1327766 MHz

F2 - Processing parameters
SI                           65536
SF                  500.1300049 MHz
WDW                             EM
SSB                             0
LB                         0.00 Hz
GB                              0
PC                         1.00

1D NMR plot parameters
CX                        20.00 cm
CY                        10.00 cm
F1P                        8.000 ppm
F1                      4001.04 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                  0.40000 ppm/cm
HZCM                 200.05200 Hz/cm
```



13C with proton decoupling




```
Current Data Parameters
NAME                 rc_651p
EXPNO                      2
PROCNO                     1

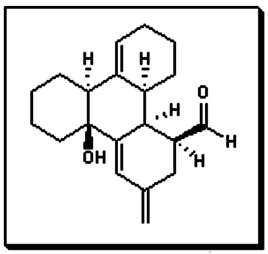
F2 - Acquisition Parameters
Date_              20040302
Time                  16.51
INSTRUM                av300
PROBHD   5 mm QNP  1H/1
PULPROG               zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       561
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110004 sec
RG                    2298.8
DW                    27.800 usec
DE                     6.00 usec
TE                     300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

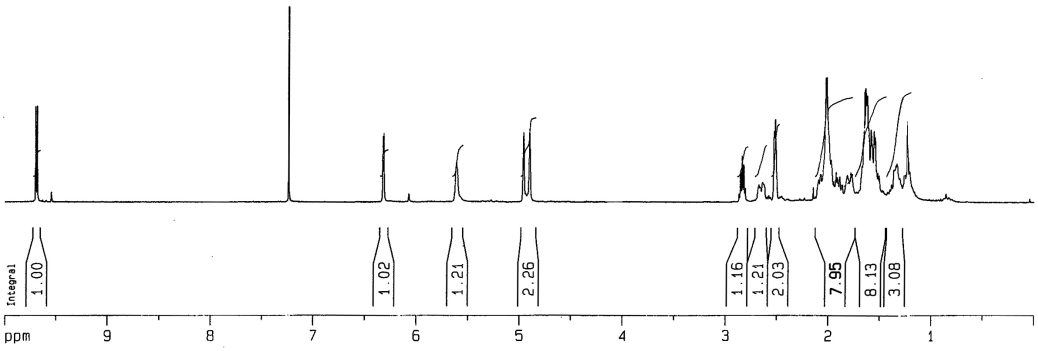
============ CHANNEL f1 =============
NUC1                     13C
P1                     5.00 usec
PL1                   -6.00 dB
SFO1              75.4752653 MHz

============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 70.00 usec
PL2                   -3.00 dB
PL12                  13.48 dB
PL13                  15.63 dB
SFO2             300.1314860 MHz

F2 - Processing parameters
SI                       65536
SF                75.4677190 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.50 cm
F1P                220.000 ppm
F1                16602.90 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             11.00000 ppm/cm
HZCM             830.14490 Hz/cm
```





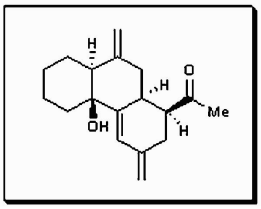

```
Current Data Parameters
NAME                  rc_651_b
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20040301
Time                     11.28
INSTRUM                  av300
PROBHD   5 mm QNP  1H/1
PULPROG                  zg30
TD                      30720
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  5081.301 Hz
FIDRES               0.165407 Hz
AQ                   3.0228980 sec
RG                       287.4
DW                     98.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                1.00000000 sec

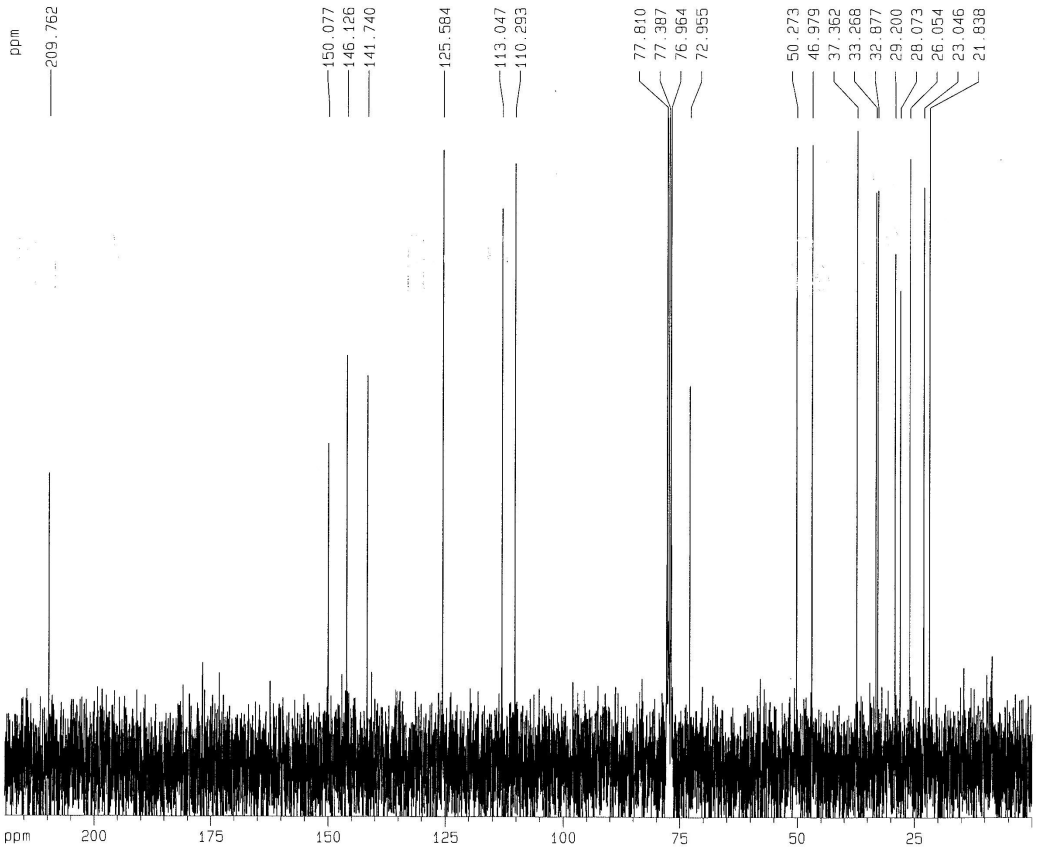
============ CHANNEL f1 =============
NUC1                        1H
P1                       10.50 usec
PL1                      -3.00 dB
SFO1              300.1319477 MHz

F2 - Processing parameters
SI                       65536
SF                300.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                     10.000 ppm
F1                    3001.30 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.50000 ppm/cm
HZCM                150.06500 Hz/cm
```



13C with proton decoupling




```
Current Data Parameters
NAME                 rc_505
EXPNO                     2
PROCNO                    1

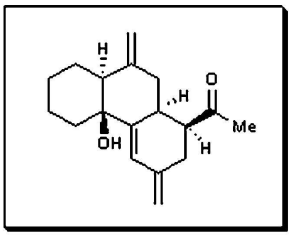
F2 - Acquisition Parameters
Date_              20031031
Time                  14.10
INSTRUM               av300
PROBHD   5 mm QNP  1H/1
PULPROG               zgpg30
TD                     32768
SOLVENT               CDCl3
NS                       217
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110004 sec
RG                    3649.1
DW                    27.800 usec
DE                     6.00 usec
TE                    300.0 K
D1              1.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

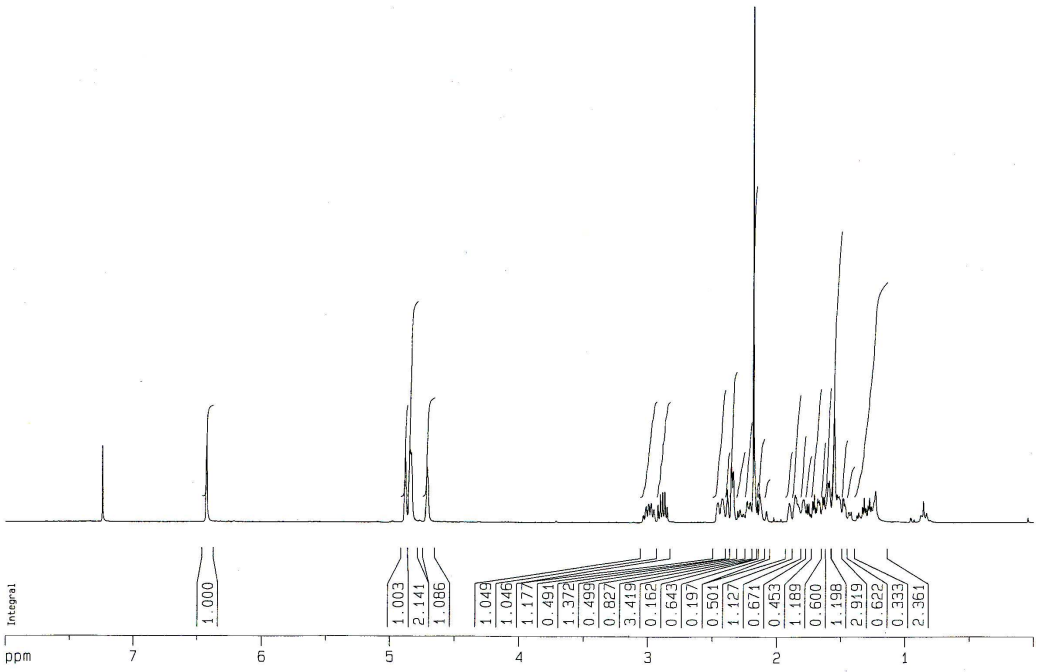
============ CHANNEL f1 ============
NUC1                    13C
P1                     5.00 usec
PL1                   -6.00 dB
SFO1             75.4752653 MHz

============ CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 70.00 usec
PL2                   -3.00 dB
PL12                  13.48 dB
PL13                  15.63 dB
SFO2            300.1314860 MHz

F2 - Processing parameters
SI                     65536
SF               75.4677190 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     12.50 cm
F1P                  220.000 ppm
F1                  16602.90 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM               11.00000 ppm/cm
HZCM               830.14490 Hz/cm
```





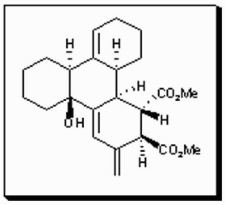

```
Current Data Parameters
NAME                  rc_505
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20031031
Time                  14.06
INSTRUM               av300
PROBHD   5 mm QNP  1H/1
PULPROG                zg30
TD                    30720
SOLVENT               CDCl3
NS                       64
DS                        0
SWH                5081.301 Hz
FIDRES            0.165407 Hz
AQ                3.0228980 sec
RG                    287.4
DW                   98.400 usec
DE                    6.00 usec
TE                   300.0 K
D1              1.00000000 sec

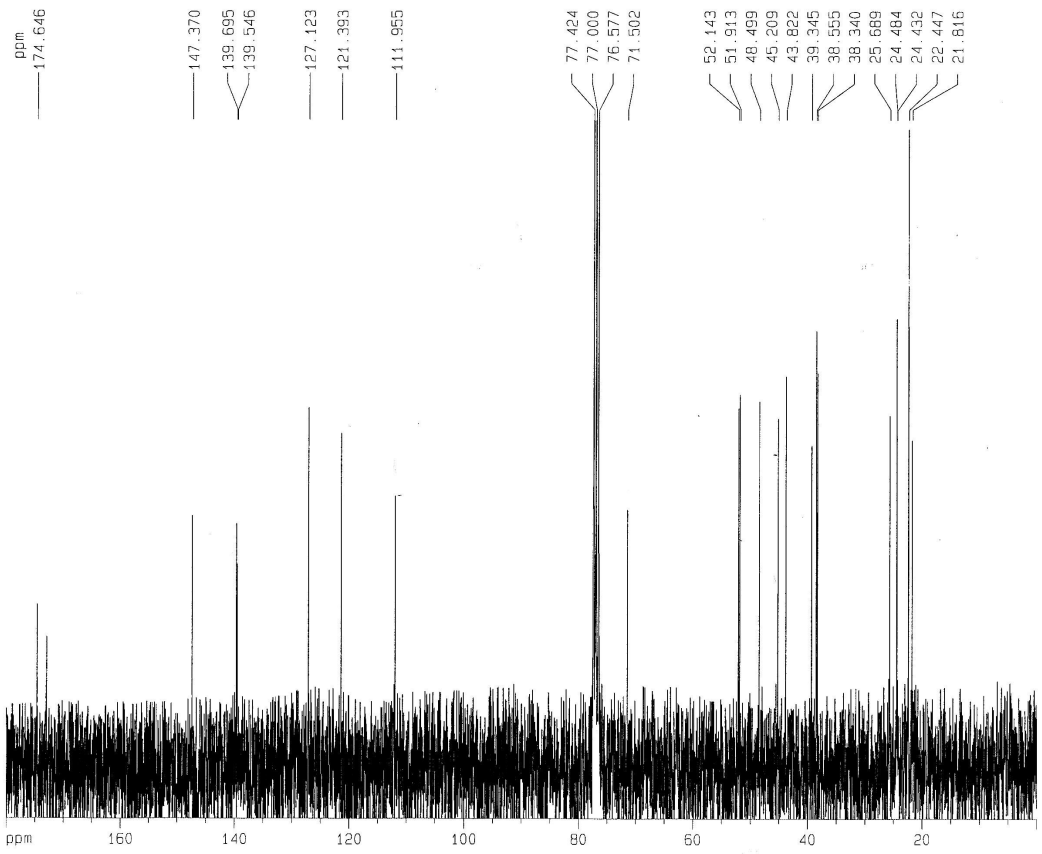
============ CHANNEL f1 =============
NUC1                      1H
P1                    10.50 usec
PL1                   -3.00 dB
SFO1           300.1319477 MHz

F2 - Processing parameters
SI                        65536
SF              300.1300000 MHz
WDW                          EM
SSB                       0
LB                     0.10 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    10.00 cm
F1P                    8.000 ppm
F1                  2401.04 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM              0.40000 ppm/cm
HZCM             120.05200 Hz/cm
```



13C with proton decoupling




```
Current Data Parameters
NAME                  rc_630_2
EXPNO                        1
PROCNO                       1

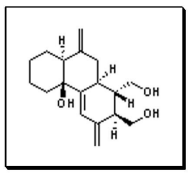
F2 - Acquisition Parameters
Date_              20040211
Time                    16.23
INSTRUM                 av300
PROBHD   5 mm QNP  1H/1
PULPROG                 zgpg30
TD                       32768
SOLVENT                  CDCl3
NS                         256
DS                           0
SWH                  17985.611 Hz
FIDRES                0.548877 Hz
AQ                    0.9110004 sec
RG                        3251
DW                      27.800 usec
DE                       6.00 usec
TE                      300.0 K
D1                 1.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

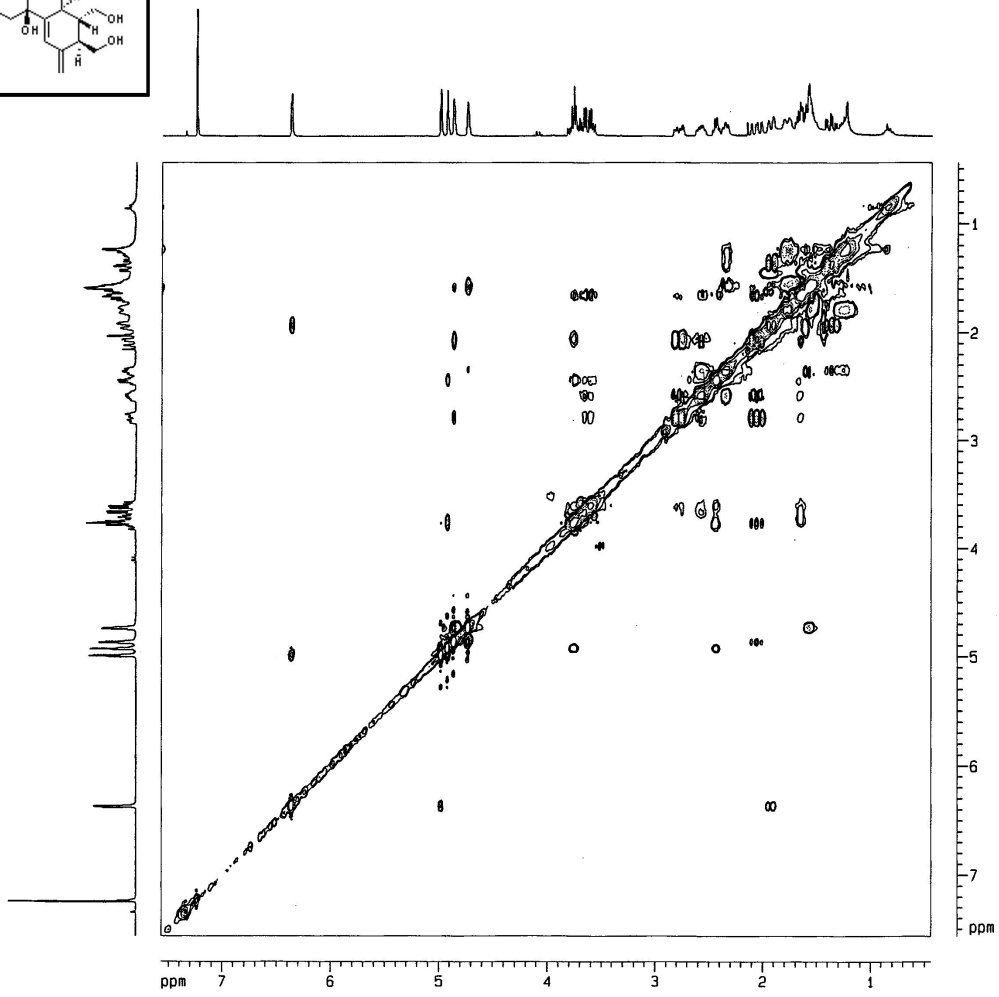
========== CHANNEL f1 ==========
NUC1                       13C
P1                        5.00 usec
PL1                      -6.00 dB
SFO1                75.4752653 MHz

========== CHANNEL f2 ==========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    70.00 usec
PL2                      -3.00 dB
PL12                     13.48 dB
PL13                     15.63 dB
SFO2               300.1314860 MHz

F2 - Processing parameters
SI                         65536
SF                  75.4677478 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
CY                         12.50 cm
F1P                       180.000 ppm
F1                      13564.20 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                   9.00000 ppm/cm
HZCM                  679.20978 Hz/cm
```





1H NOESY

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Current Data Parameters
NAME            rc_434_2d
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_         20030923
Time               0.42
INSTRUM          av300
PROBHD     5 mm QNP 1H/1
PULPROG       noesyetgp
TD                 1024
SOLVENT           CDCl3
NS                   48
DS                    8
SWH           2140.411 Hz
FIDRES        2.090245 Hz
AQ            0.239256s sec
RG                7298.2
DW              233.500 usec
DE                6.00 usec
TE                300.0 K
d0            0.00000300 sec
D1            1.00000000 sec
D8            1.00000000 sec
d11           0.03000000 sec
D16           0.00010000 sec
DELTA         0.00110300 sec
DELTA1        0.00110850 sec
IN0           0.00046720 sec
NOREST        0.00000000 sec
NCHAN         0.00750000 sec
STSIGNT             128
TAU           0.99890000 sec

========== CHANNEL f1 ==========
NUC1                 1H
P1                10.50 usec
p2                21.00 usec
PL1               -3.00 dB
SFO1        300.1312017 MHz

========== GRADIENT CHANNEL ========
GPNAM1          sine.100
GPNAM2          sine.100
GPNAM3          sine.100
GPX1              0.00 %
GPX2              0.00 %
GPX3              0.00 %
GPY1              0.00 %
GPY2              0.00 %
GPY3              0.00 %
GPZ1             30.00 %
GPZ2             50.00 %
GPZ3             30.00 %
P16            1000.00 usec

F1 - Acquisition parameters
NDD                  1
TD                 256
SFO1        300.1312 MHz
FIDRES        8.390980 Hz
SW                7.132 ppm

F2 - Processing parameters
SI                1024
SF          300.1300000 MHz
WDW               QSINE
SSB                  2
LB                0.00 Hz
GB                   0
PC                1.00

F1 - Processing parameters
SI                1024
MC2       echo-antiecho
SF          300.1300000 MHz
WDW               QSINE
SSB                  2
LB                0.00 Hz
GB                   0

2D NMR plot parameters
CX2              15.00 cm
CX1              15.00 cm
F2PLO             7.570 ppm
F2LO           2271.91 Hz
F2PHI             0.438 ppm
F2HI            131.50 Hz
F1PLO             7.570 ppm
F1LO           2271.91 Hz
F1PHI             0.438 ppm
F1HI            131.50 Hz
F2PPMCM       0.47544 ppm/cm
F2HZCM      142.89498 Hz/cm
F1PPMCM       0.47544 ppm/cm
```
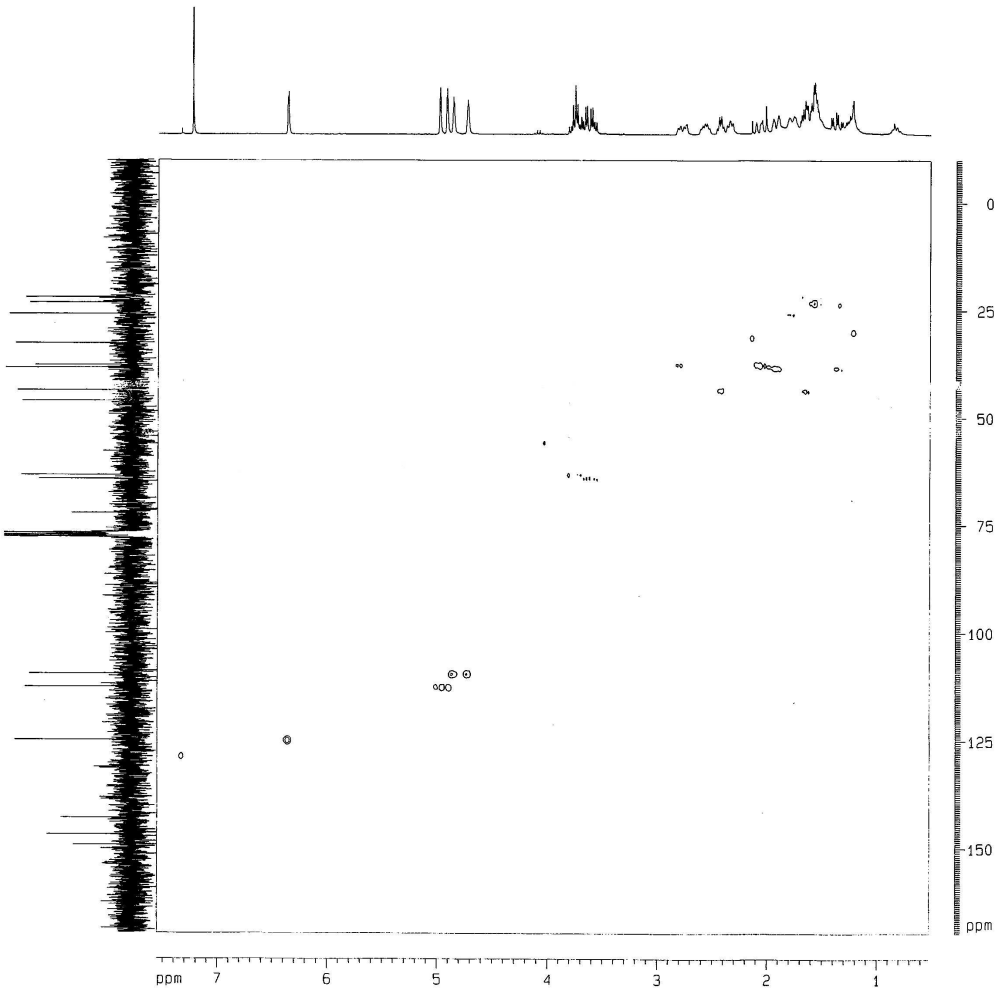
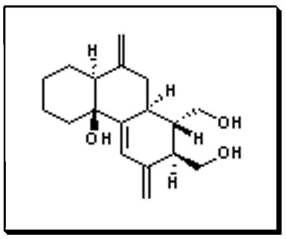





DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.00000000 sec
d2 0.00344828 sec
d12 0.00002000 sec
d13 0.00000400 sec
D16 0.00016000 sec
d20 0.00242428 sec
IN0 0.00003580 sec
MCREST 0.00000000 sec
MCWRK 1.00000000 sec

********** CHANNEL f1 **********
NUC1 1H
P1 10.50 usec
p2 21.00 usec
PL1 -3.00 dB
SFO1 300.1312123 MHz

********** CHANNEL f2 **********
CPDPRG2 garp
NUC2 13C
P3 5.00 usec
PCPD2 70.00 usec
PL2 -6.00 dB
PL12 15.32 dB
SFO2 75.4737564 MHz

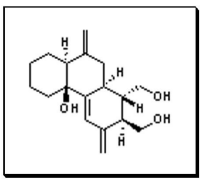
********** GRADIENT CHANNEL **********
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPZ1 0.00 %
GPZ2 0.00 %
GPZ3 0.00 %
GPT1 0.00 %
GPT2 0.00 %
GPT3 0.00 %
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
P16 1000.00 usec

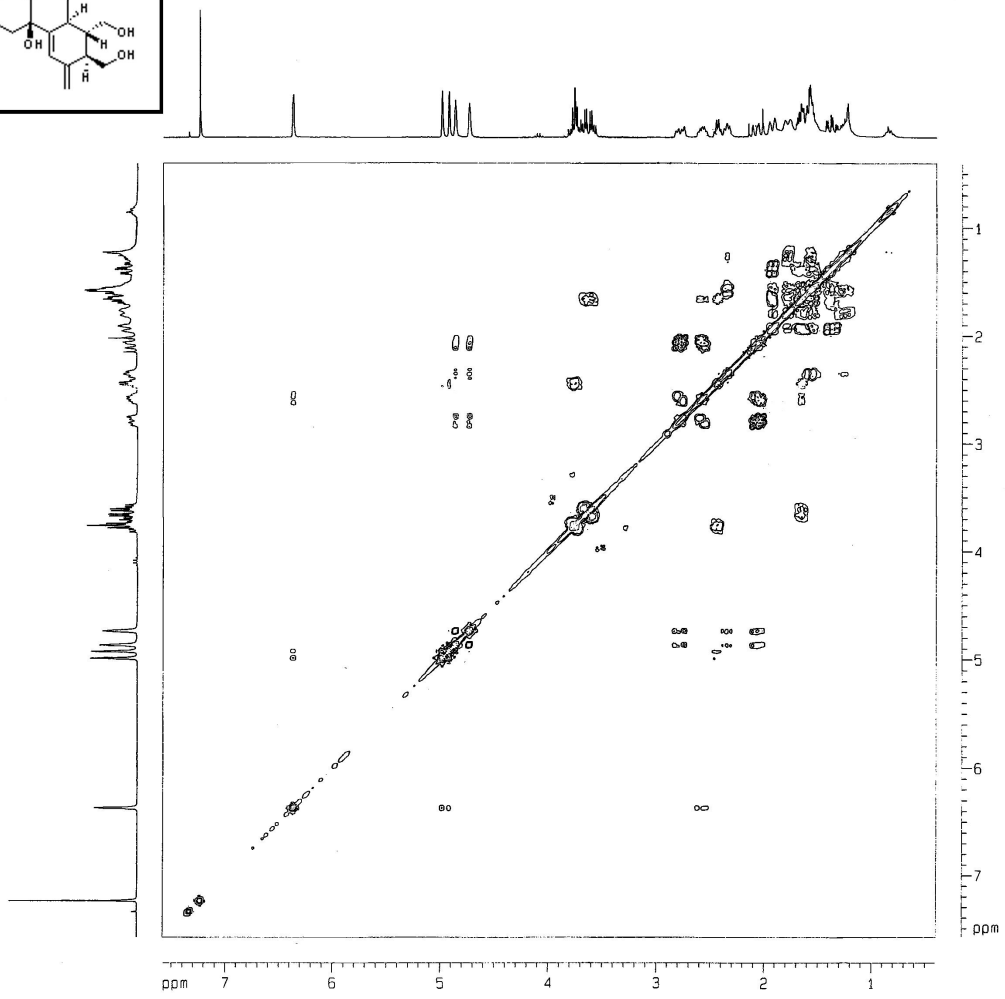
F1 - Acquisition parameters
NO0 2
TD 256
SFO1 75.47376 MHz
FIDRES 53.074047 Hz
SW 199.022 ppm

F2 - Processing parameters
SI 512
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 512
MC2 QF
SF 75.4677193 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 7.557 ppm
F2LO 2268.01 Hz
F2PHI 0.522 ppm
F2HI 156.52 Hz
F1PLO 170.019 ppm
F1LO 12830.88 Hz
F1PHI -10.019 ppm
F1HI -756.88 Hz
F2PPMCM 0.48902 ppm/cm
F2HZCM 146.76578 Hz/cm
F1PPMCM 12.00244 ppm/cm
F1HZCM 905.79712 Hz/cm





1H COSY

Current Data Parameters
NAME rc_434(2d)
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030822
Time 21.54
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG cosy45gpqf
TD 1024
SOLVENT CDCl3
NS 1
DS 0
SWH 2155.172 Hz
FIDRES 2.104661 Hz
AQ 0.2376180 sec
RG 2580.3
DW 232.000 usec
DE 6.00 usec
TE 300.0 K
d0 0.00000300 sec
D1 1.00000000 sec
d13 0.00000400 sec
D16 0.00010000 sec
IN0 0.00046400 sec
MCREST 0.00000000 sec
MCWRK 1.00000000 sec

---------- CHANNEL f1 ----------
NUC1 1H
p0 5.25 usec
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.1311982 MHz

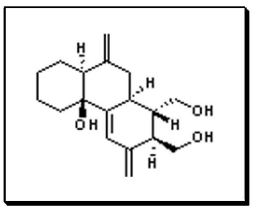
---------- GRADIENT CHANNEL --------
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec

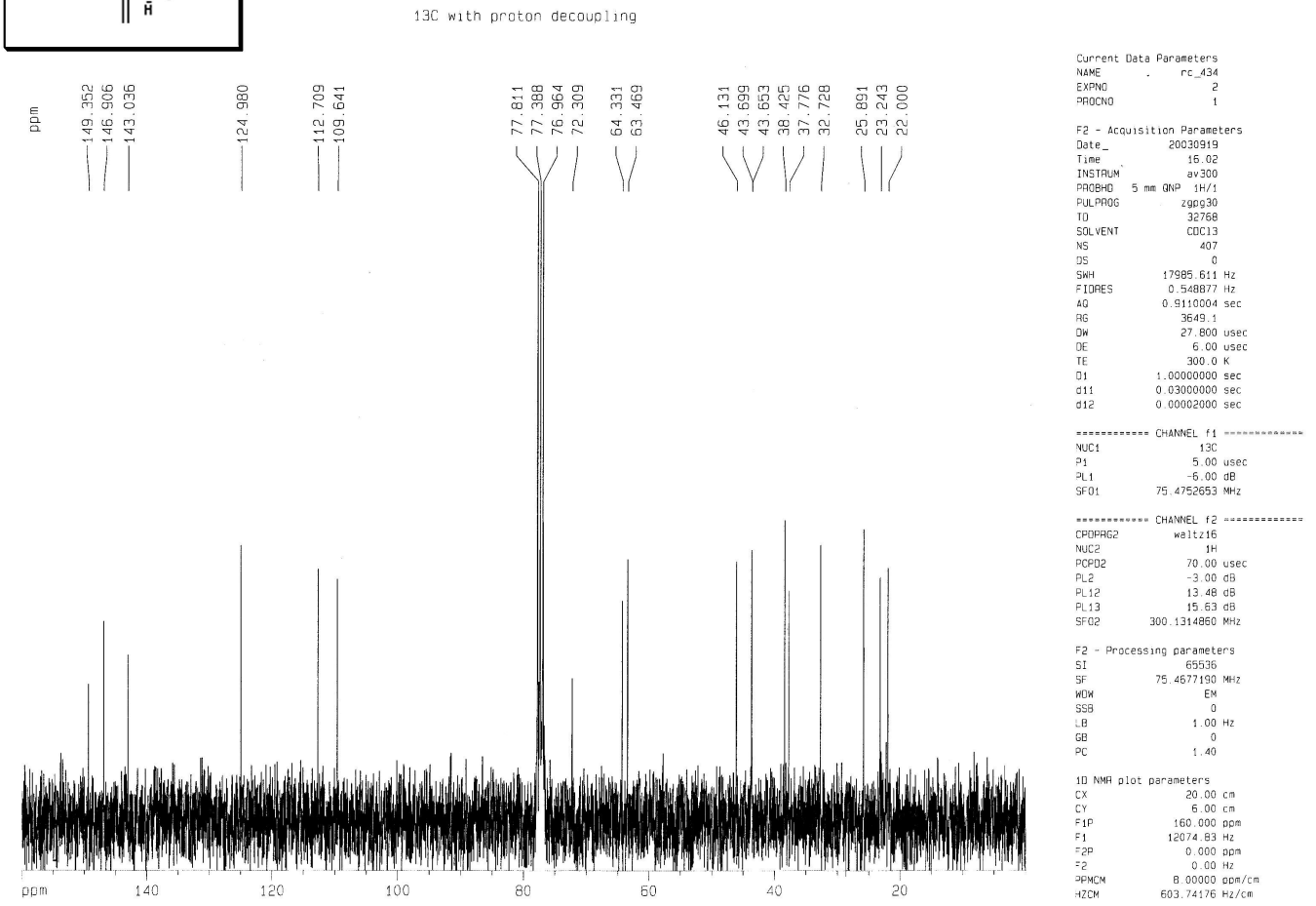
F1 - Acquisition parameters
ND0 1
TD 256
SFO1 300.1312 MHz
FIDRES 8.418642 Hz
SW 7.181 ppm

F2 - Processing parameters
SI 1024
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

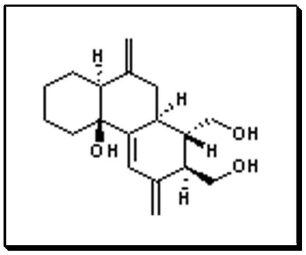
F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1300000 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

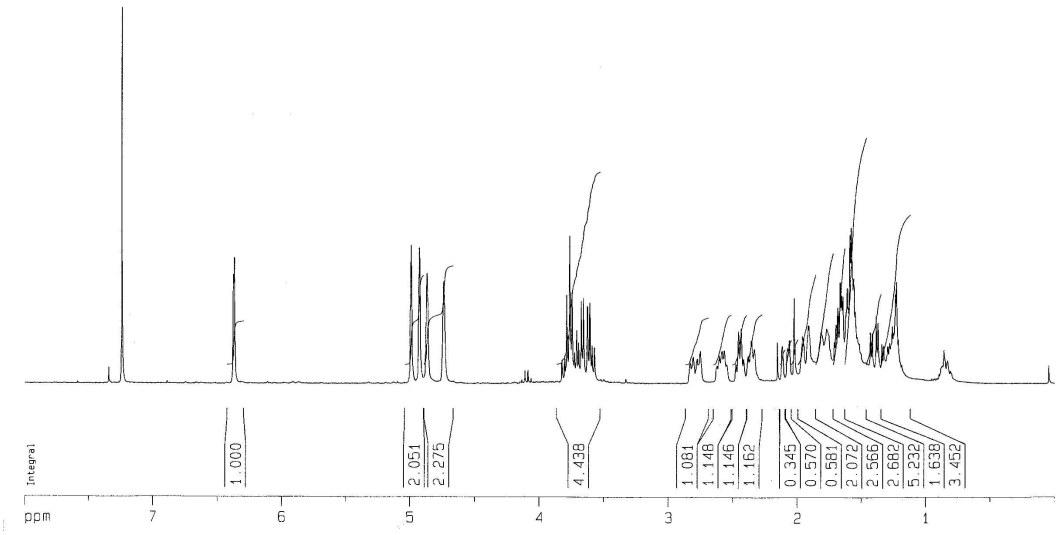
2D NMR plot parameters
CX2 15.00 cm
CX1 15.00 cm
F2PLO 7.583 ppm
F2LO 2275.77 Hz
F2PHI 0.402 ppm
F2HI 120.60 Hz
F1PLO 7.583 ppm
F1LO 2275.77 Hz
F1PHI 0.402 ppm
F1HI 120.60 Hz
F2PPMCM 0.47872 ppm/cm
F2HZCM 143.67816 Hz/cm
F1PPMCM 0.47872 ppm/cm
F1HZCM 143.67816 Hz/cm



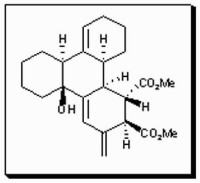


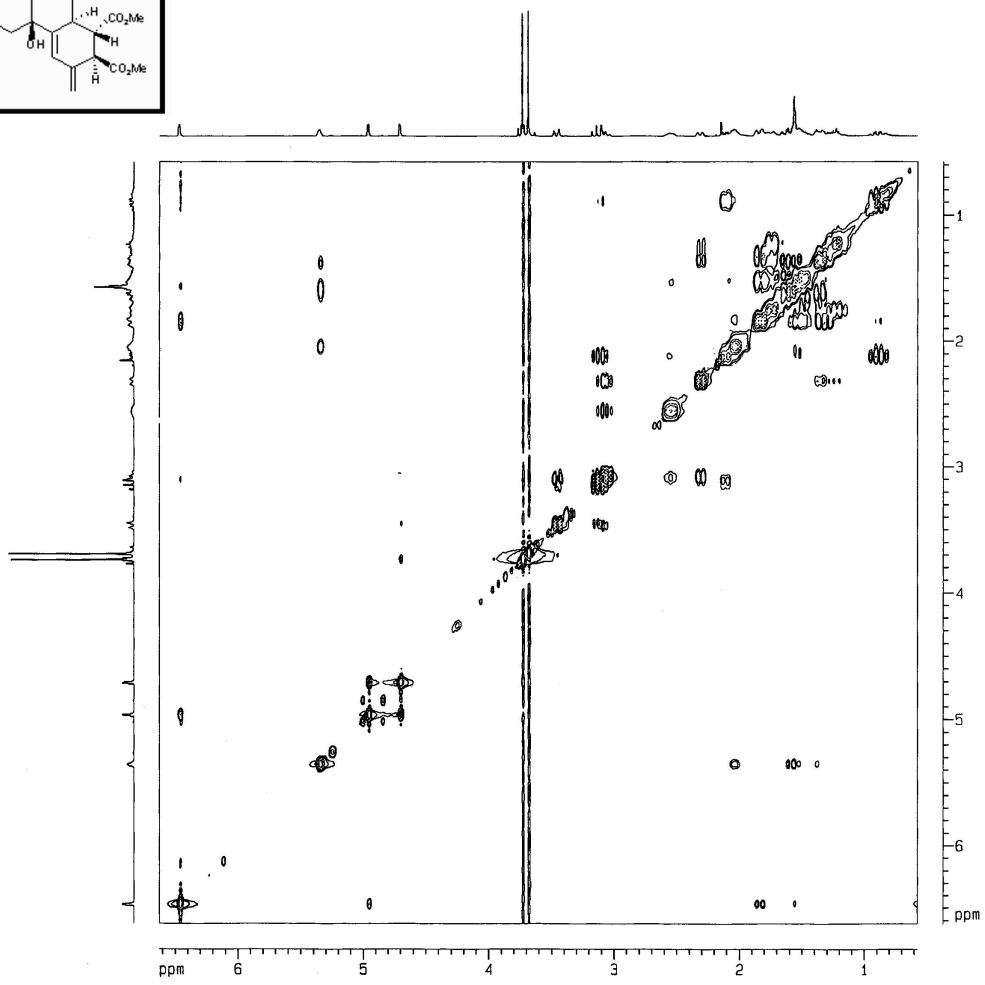

```
Current Data Parameters
NAME                 rc_434
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20030919
Time                 16.02
INSTRUM                av300
PROBHD   5 mm QNP  1H/1
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      407
DS                        0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                0.9110004 sec
RG                   3649.1
DW                   27.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                    5.00 usec
PL1                  -6.00 dB
SF01             75.4752653 MHz

============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                70.00 usec
PL2                   -3.00 dB
PL12                  13.48 dB
PL13                  15.63 dB
SF02            300.1314860 MHz

F2 - Processing parameters
SI                      65536
SF               75.4677190 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                       6.00 cm
F1P                    160.000 ppm
F1                   12074.83 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 8.00000 ppm/cm
HZCM                603.74176 Hz/cm
```





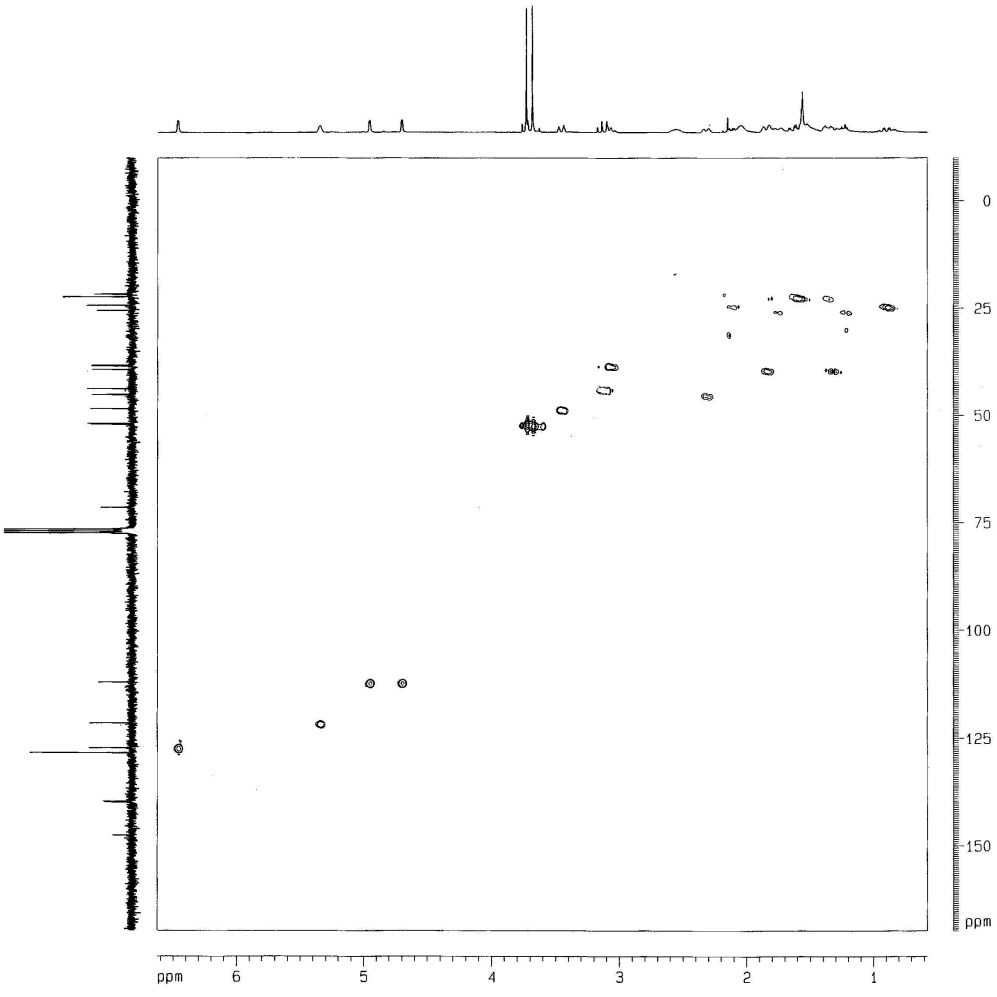

```
Current Data Parameters
NAME                  rc_434
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20030919
Time                   15.49
INSTRUM                av300
PROBHD   5 mm GNP  1H/1
PULPROG                zg30
TD                     30720
SOLVENT                CDCl3
NS                        64
DS                         0
SWH                5081.301 Hz
FIDRES             0.165407 Hz
AQ                 3.0228980 sec
RG                       512
DW                  98.400 usec
DE                   6.00 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =============
NUC1                       1H
P1                    10.50 usec
PL1                   -3.00 dB
SFO1             300.1319477 MHz

F2 - Processing parameters
SI                         65536
SF                300.1300000 MHz
WDW                           EM
SSB                            0
LB                          0.10 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
CY                         10.00 cm
F1P                        8.000 ppm
F1                       2401.04 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                  0.40000 ppm/cm
HZCM                 120.05200 Hz/cm
```

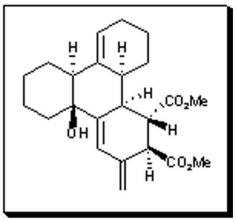


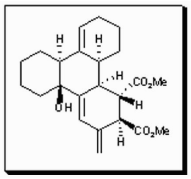


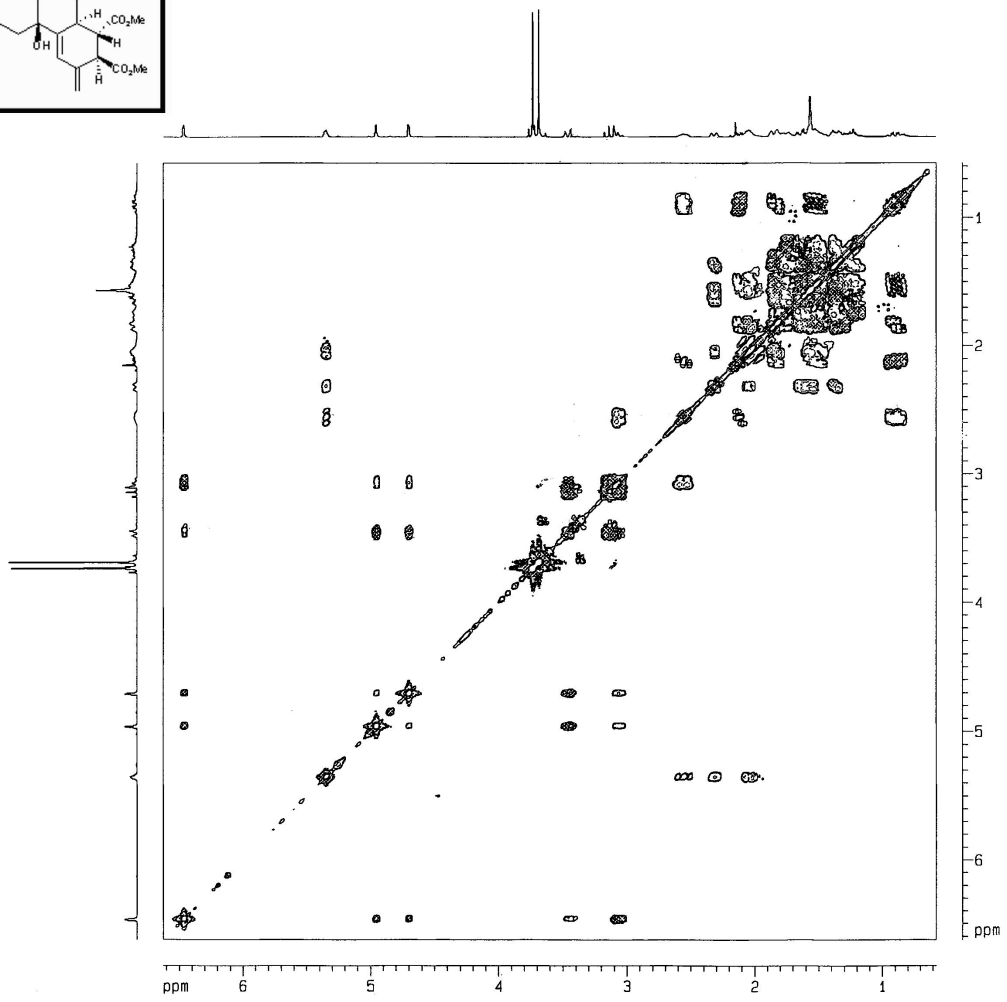
1H NOESY

```
Current Data Parameters
NAME                  nc_830_2H
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_              20040611
Time                   2.16
INSTRUM                av300
PROBHD    5 mm BBF  UV/I
PULPROG          noesytp
TD                     4624
SOLVENT               CDCl3
NS                       34
DS                        8
SWH                 1816.860 Hz
FIDRES              1.774278 Hz
AQ                  0.2818548 sec
RG                       8192
DW                   275.200 usec
DE                     6.00 usec
TE                     300.0 K
d0                  0.00000300 sec
D1                  1.00000000 sec
D8                  1.00000000 sec
d11                 0.03000000 sec
D16                 0.00010000 sec
DELTA               0.00110300 sec
DELTA1              0.00110800 sec
D9                  0.00059040 sec
MCREST              0.00000000 sec
MCWRK               0.00750000 sec
STSIGNT                    126
TAU                 0.99890000 sec

========== CHANNEL f1 ==========
NUC1                       1H
P1                      10.50 usec
p2                      21.00 usec
PL1                     -3.00 dB
SFO1               300.1310812 MHz

========== GRADIENT CHANNEL ========
GPNAM1                sine.100
GPNAM2                sine.100
GPNAM3                sine.100
GPX1                     0.00 %
GPX2                     0.00 %
GPX3                     0.00 %
GPY1                     0.00 %
GPY2                     0.00 %
GPY3                     0.00 %
GPZ1                    30.00 %
GPZ2                    50.00 %
GPZ3                    30.00 %
P16                   1000.00 usec

F1 - Acquisition parameters
NDO                          1
TD                         253
SFO1               300.1311 MHz
FIDRES              7.181267 Hz
SW                     8.054 ppm

F2 - Processing parameters
SI                        1024
SF                 300.1300000 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
SI                        1024
MC2                echo-antiecho
SF                 300.1300000 MHz
WDW                      QSINE
SSB                          2
LB                        0.00 Hz
GB                           0

2D NMR plot parameters
CX2                      15.00 cm
CX1                      15.00 cm
F2PLO                    6.629 ppm
F2LO                  1989.60 Hz
F2PHI                    0.576 ppm
F2HI                   172.74 Hz
F1PLO                    6.629 ppm
F1LO                  1989.60 Hz
F1PHI                    0.576 ppm
F1HI                   172.74 Hz
F2PPMCM                0.40357 ppm/cm
F2HZCM               121.12403 Hz/cm
F1PPMCM                0.40357 ppm/cm
```

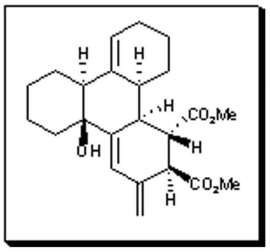

0
25
50
75
100
125
150
ppm
ppm
6
5
4
3
2
1

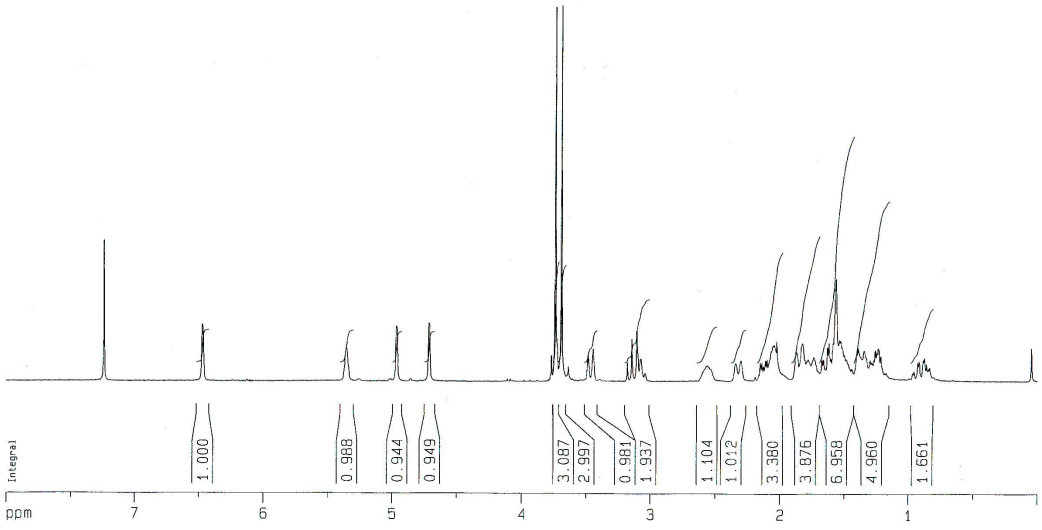

H
H
H
CO2Me
H
OH
CO2Me
H



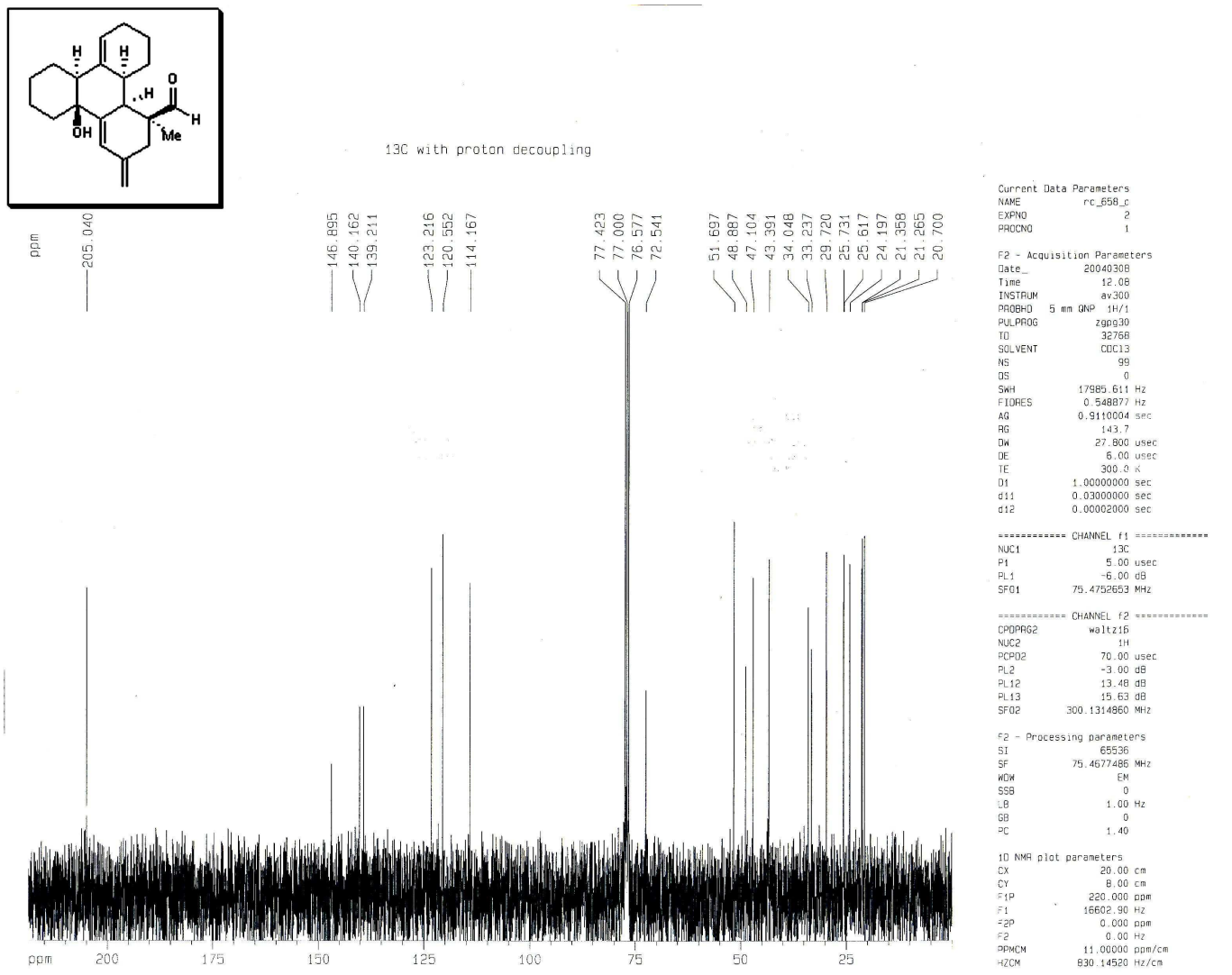


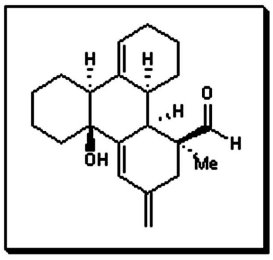

```
                Current Data Parameters
NAME                  rc_630_2d
EXPNO                         2
PROCNO                        1

        F2 - Acquisition Parameters
Date_               20080840
Time                      21.31
INSTRUM                   av400
PROBHD     5 mm QNP  4nuc
PULPROG             cosy45gpqf
TD                        1024
SOLVENT                   CDCl3
NS                          20
DS                           0
SWH                  1616.060 Hz
FIDRES               1.774078 Hz
AQ                   0.2818548 sec
RG                       2896.3
DW                     275.200 usec
DE                       6.00 usec
TE                      300.0 K
d0                   0.00000300 sec
D1                   1.00000000 sec
d13                  0.00000400 sec
D16                  0.00010000 sec
IN0                  0.00055040 sec
MCREST               0.00000000 sec
MCWRK                0.00000000 sec

------------ CHANNEL f1 ------------
NUC1                        1H
p0                        5.25 usec
P1                       10.50 usec
PL1                      -3.00 dB
SFO1                300.1310812 MHz

------------ GRADIENT CHANNEL -------
GPNAM1                  sine.100
GPNAM2                  sine.100
GPX1                        0.00 %
GPY1                        0.00 %
GPZ1                       10.00 %
GPZ2                       10.00 %
P16                      1000.00 usec

        F1 - Acquisition parameters
ND0                           1
TD                          256
SFO1                300.1311 MHz
FIDRES               7.097111 Hz
SW                       6.054 ppm

        F2 - Processing parameters
SI                          1024
SF                  300.1300000 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

        F1 - Processing parameters
SI                          1024
MC2                           QF
SF                  300.1300000 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0

        2D NMR plot parameters
CX2                        15.00 cm
CX1                        15.00 cm
F2PLO                      6.629 ppm
F2LO                    1989.60 Hz
F2PHI                      0.578 ppm
F2HI                     172.74 Hz
F1PLO                      6.629 ppm
F1LO                    1989.60 Hz
F1PHI                      0.578 ppm
F1HI                     172.74 Hz
F2PPMCM                 0.40357 ppm/cm
F2HZCM                121.12403 Hz/cm
F1PPMCM                 0.40357 ppm/cm
F1HZCM                121.12403 Hz/cm
```

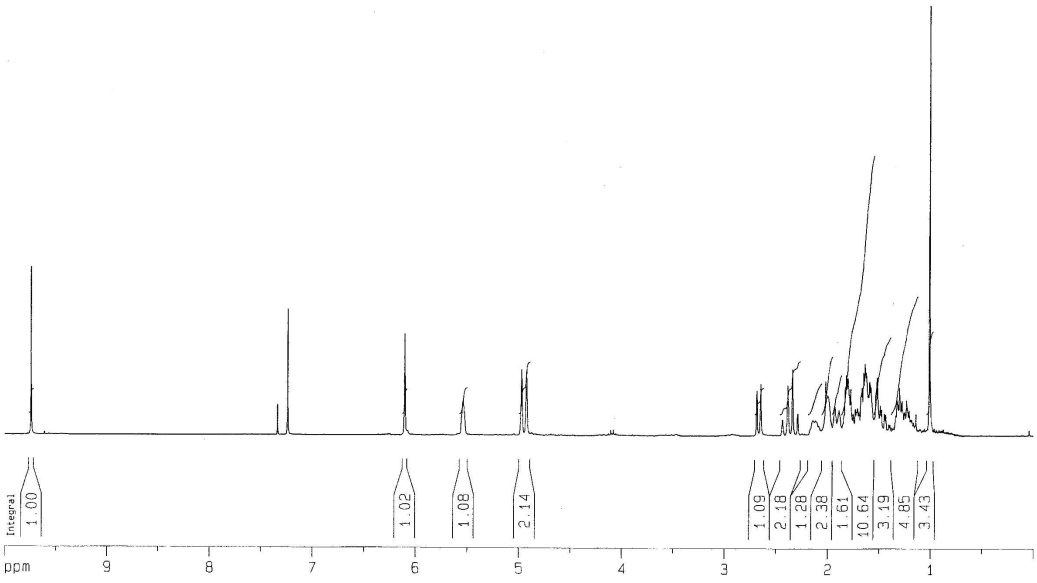




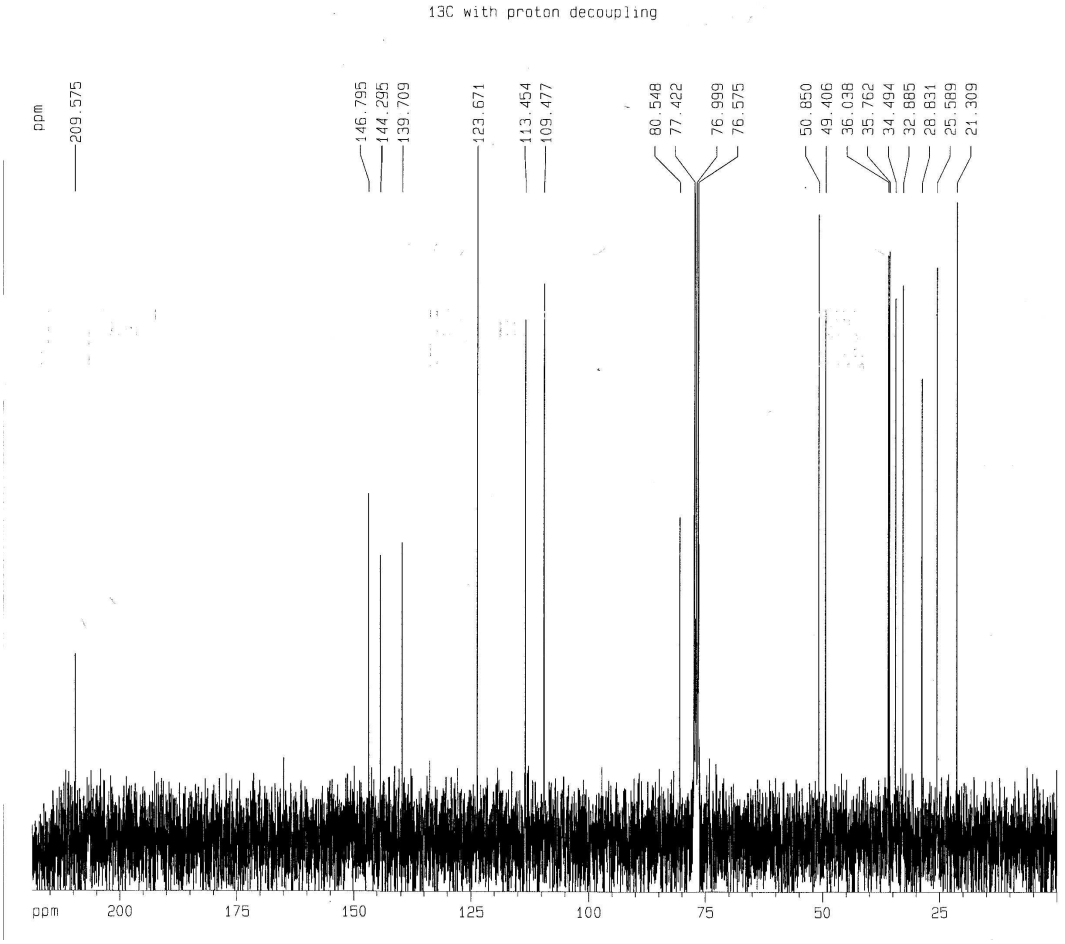

```
Current Data Parameters
NAME                  rc_630
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040211
Time                  16.20
INSTRUM               av300
PROBHD    5 mm QNP  1H/1
PULPROG                zg30
TD                    30720
SOLVENT               CDCl3
NS                       16
DS                        0
SWH               5081.301 Hz
FIDRES            0.165407 Hz
AQ                3.0228980 sec
RG                    406.4
DW                  98.400 usec
DE                    6.00 usec
TE                   300.0 K
D1              1.00000000 sec

============ CHANNEL f1 =============
NUC1                      1H
P1                    10.50 usec
PL1                   -3.00 dB
SFO1           300.1319477 MHz

F2 - Processing parameters
SI                    65536
SF             300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.10 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    10.00 cm
F1P                    8.000 ppm
F1                  2401.04 Hz
F2P                   0.000 ppm
F2                     0.00 Hz
PPMCM              0.40000 ppm/cm
HZCM             120.05200 Hz/cm
```


13C with proton decoupling
ppm
205.040
146.895
140.162
139.211
123.216
120.552
114.167
77.423
77.000
76.577
72.541
51.697
48.887
47.104
43.391
34.048
33.237
29.720
25.731
25.617
24.197
21.358
21.265
20.700
ppm
200
175
150
125
100
75
50
25
Current Data Parameters
NAME rc_658_c
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040308
Time 12.08
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 99
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 143.7
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
============ CHANNEL f1 ============
NUC1 13C
P1 5.00 usec
PL1 -6.00 dB
SFO1 75.4752653 MHz
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SFO2 300.1314860 MHz
F2 - Processing parameters
SI 65536
SF 75.4677486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14520 Hz/cm

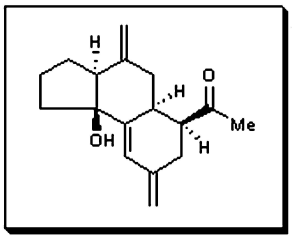


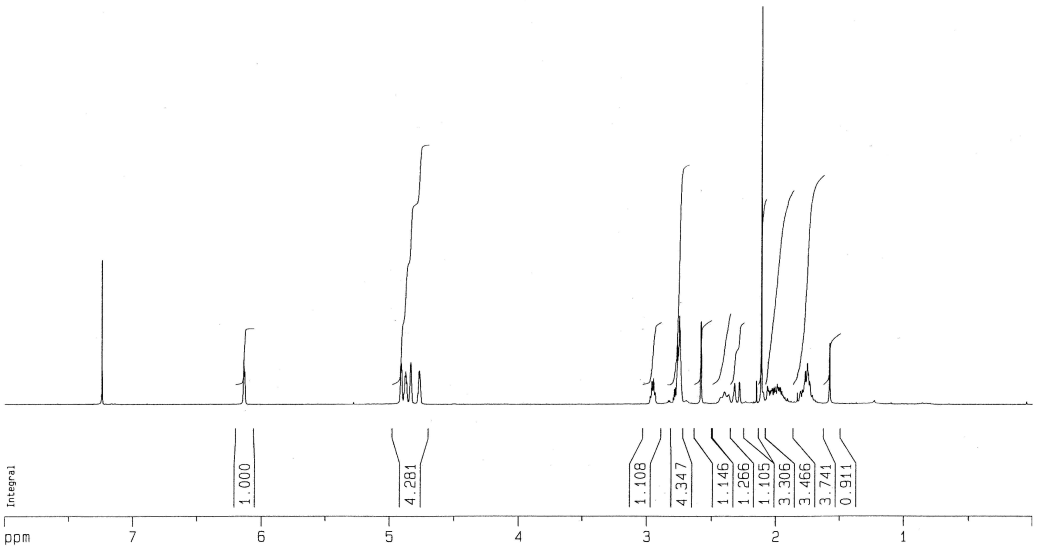



```
Current Data Parameters
NAME                  rc_658_c
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20040308
Time                     12.05
INSTRUM                  av300
PROBHD    5 mm QNP  1H/1
PULPROG                  zg30
TD                       30720
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 5081.301 Hz
FIDRES              0.165407 Hz
AQ                  3.0228980 sec
RG                         256
DW                     98.400 usec
DE                      6.00 usec
TE                      300.0 K
D1                 1.00000000 sec

============ CHANNEL f1 =============
NUC1                        1H
P1                       10.50 usec
PL1                      -3.00 dB
SFO1               300.1319477 MHz

F2 - Processing parameters
SI                       65536
SF                 300.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                     10.000 ppm
F1                    3001.30 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.50000 ppm/cm
HZCM                150.06500 Hz/cm
```

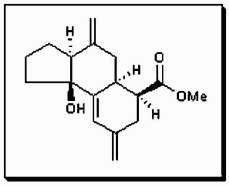



```
Current Data Parameters
NAME            rc_550_a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20031125
Time              17.50
INSTRUM           av300
PROBHD   5 mm QNP 1H/1
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  547
DS                    0
SWH           17985.611 Hz
FIDRES        0.548877 Hz
AQ            0.9110004 sec
RG              10321.3
DW               27.800 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                 5.00 usec
PL1               -6.00 dB
SFO1         75.4752653 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             70.00 usec
PL2               -3.00 dB
PL12              13.48 dB
PL13              15.63 dB
SFO2        300.1314860 MHz

F2 - Processing parameters
SI                 65536
SF           75.4677484 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P              220.000 ppm
F1              16602.90 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM          830.14520 Hz/cm
```

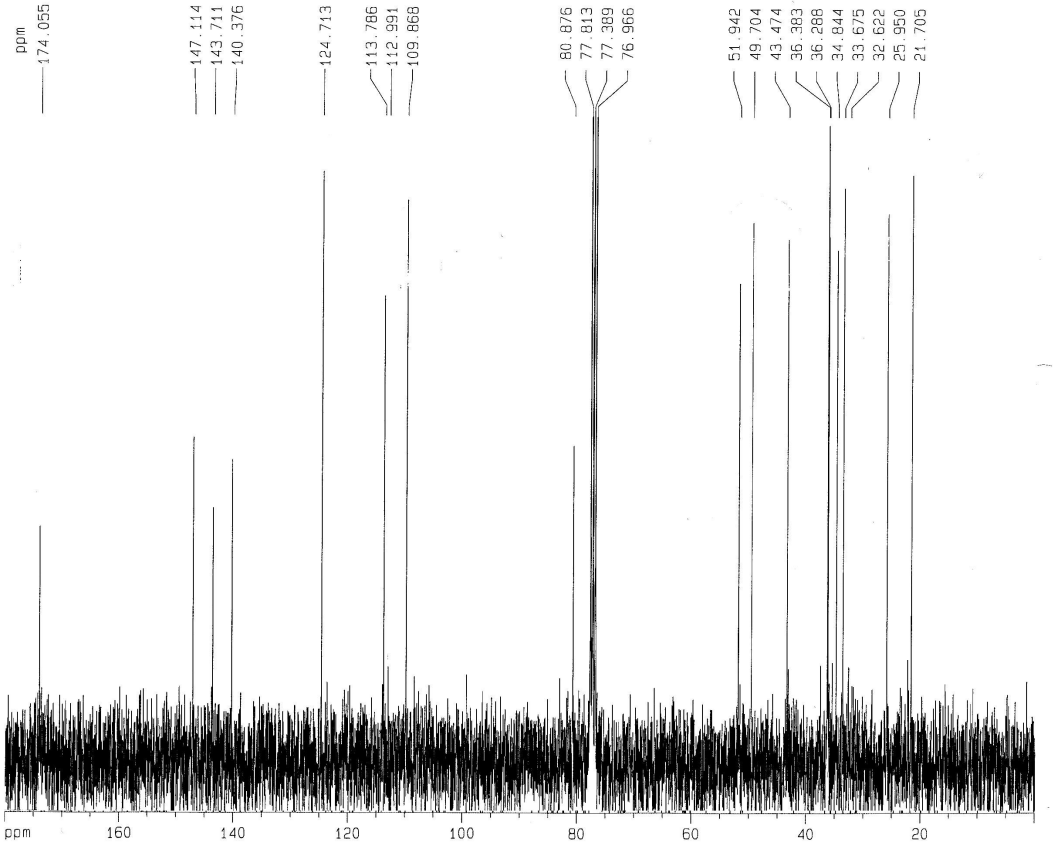




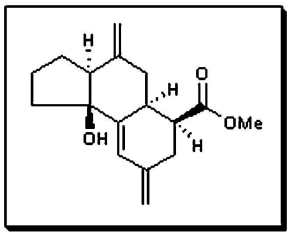

```
Current Data Parameters
NAME                  rc_550_a
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20031125
Time                    17.48
INSTRUM                 av300
PROBHD   5 mm GNP  1H/1
PULPROG                 zg30
TD                      30720
SOLVENT                 CDCl3
NS                         32
DS                          0
SWH                 5081.301 Hz
FIDRES              0.165407 Hz
AQ                  3.0228980 sec
RG                      406.4
DW                    98.400 usec
DE                     6.00 usec
TE                     300.0 K
D1                 1.00000000 sec

============ CHANNEL f1 =============
NUC1                       1H
P1                      10.50 usec
PL1                     -3.00 dB
SFO1               300.1319477 MHz

F2 - Processing parameters
SI                       65536
SF                 300.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                      8.000 ppm
F1                     2401.04 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.40000 ppm/cm
HZCM                120.05200 Hz/cm
```

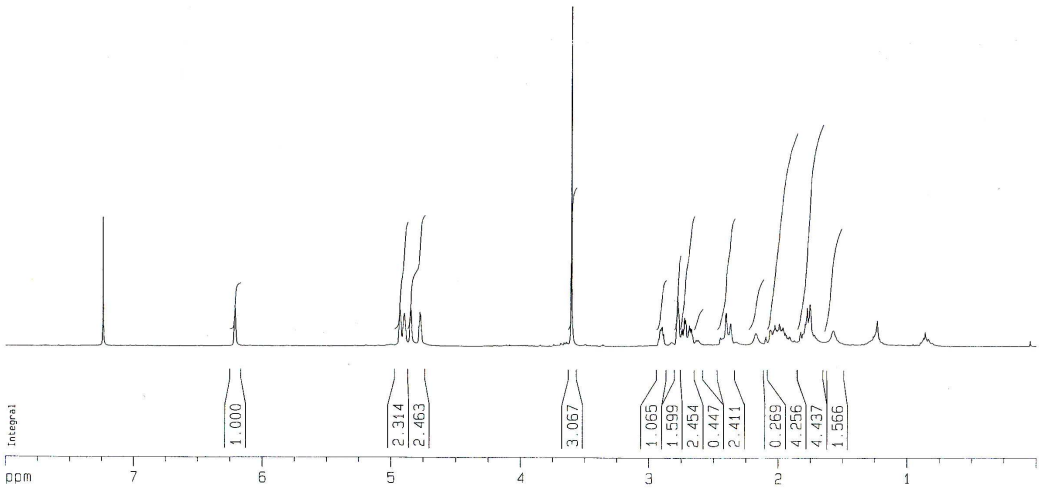


13C with proton decoupling

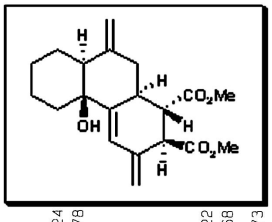



```
Current Data Parameters
NAME                  rc_437
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20030916
Time                  16.23
INSTRUM                av300
PROBHD    5 mm QNP ¯1H/1
PULPROG              zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       318
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110004 sec
RG                    5792.6
DW                   27.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                      5.00 usec
PL1                    -6.00 dB
SFO1              75.4752653 MHz

============ CHANNEL f2 =============
CPDPRG2               waltz16
NUC2                      1H
PCPD2                  70.00 usec
PL2                    -3.00 dB
PL12                   13.48 dB
PL13                   15.63 dB
SFO2             300.1314860 MHz

F2 - Processing parameters
SI                       65536
SF                75.4677190 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
CY                       12.50 cm
F1P                     180.000 ppm
F1                    13584.19 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 9.00000 ppm/cm
HZCM                679.20947 Hz/cm
```

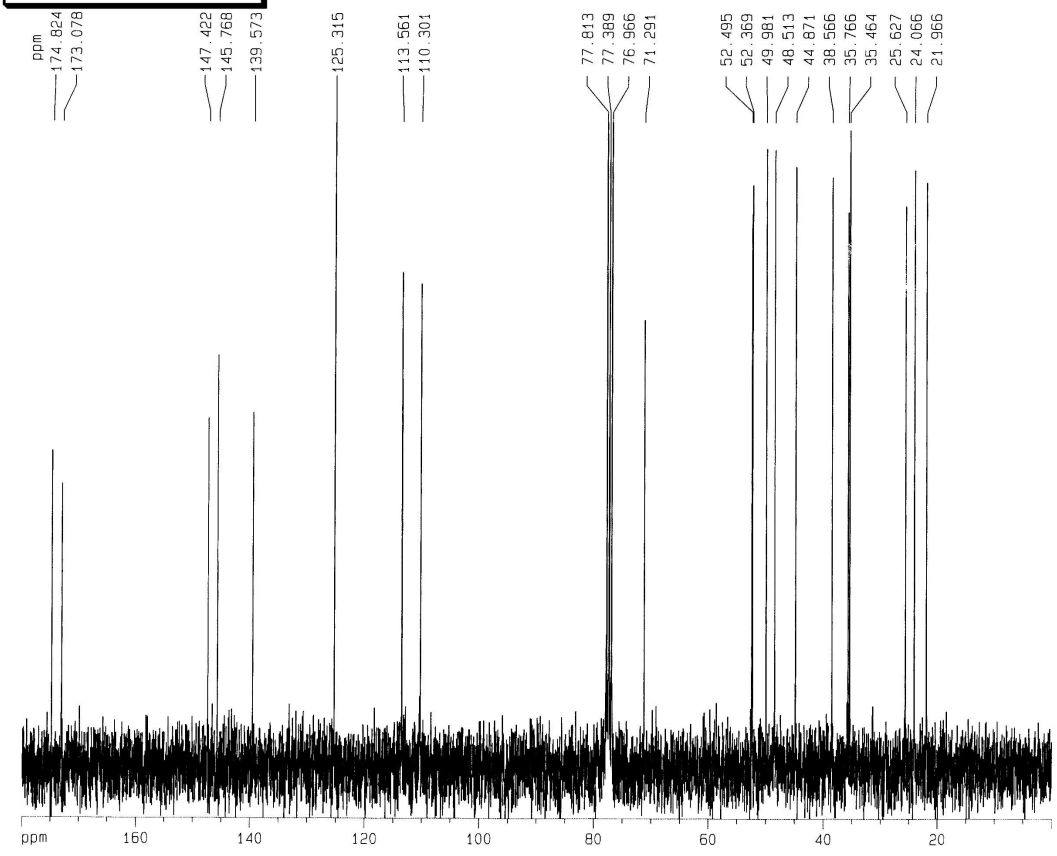




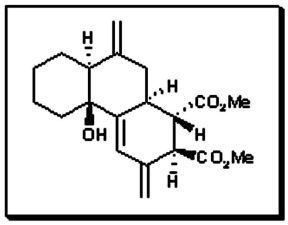

```
Current Data Parameters
NAME          rc_437(_2)
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20030918
Time               15.49
INSTRUM            av300
PROBHD   5 mm GNP  1H/1
PULPROG            zg30
TD                30720
SOLVENT           CDCl3
NS                  121
DS                    0
SWH           5081.301 Hz
FIDRES        0.165407 Hz
AQ            3.0228980 sec
RG                  512
DW               98.400 usec
DE                6.00 usec
TE               300.0 K
D1            1.00000000 sec

============ CHANNEL f1 ============
NUC1                1H
P1               10.50 usec
PL1              -3.00 dB
SF01        300.1319477 MHz

F2 - Processing parameters
SI                65536
SF          300.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               20.00 cm
CY               10.00 cm
F1P               8.000 ppm
F1             2401.04 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          0.40000 ppm/cm
HZCM         120.05200 Hz/cm
```

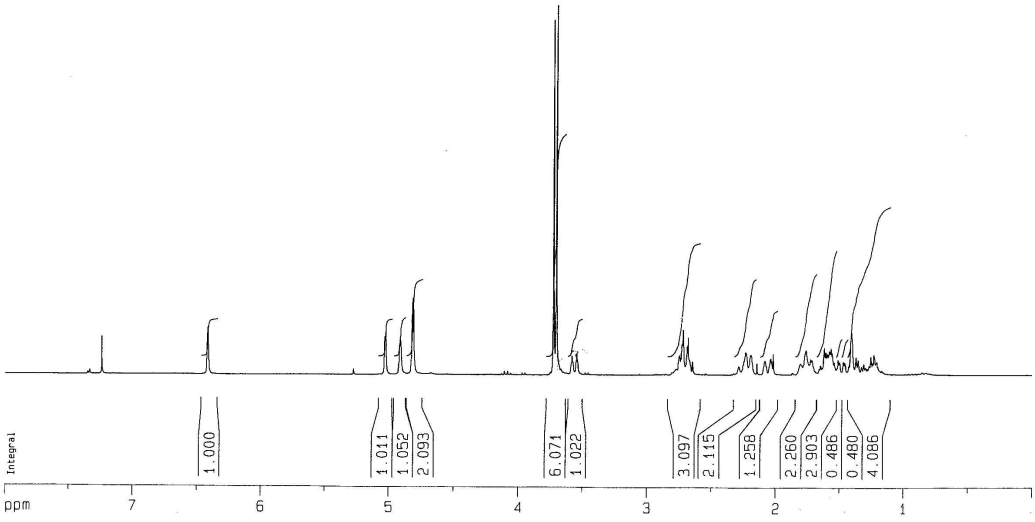


13C with proton decoupling

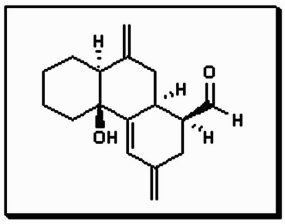



```
Current Data Parameters
NAME                 rc_398
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_             20030821
Time                 16.10
INSTRUM              av300
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   213
DS                     0
SWH              17985.611 Hz
FIDRES           0.548877 Hz
AQ               0.9110004 sec
RG                3649.1
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                 13C
P1                  5.00 usec
PL1                -6.00 dB
SFO1           75.4752653 MHz

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -3.00 dB
PL12               13.48 dB
PL13               15.63 dB
SFO2          300.1314860 MHz

F2 - Processing parameters
SI                 65536
SF             75.4677190 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P               180.000 ppm
F1              13584.19 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM              9.00000 ppm/cm
HZCM             679.20947 Hz/cm
```

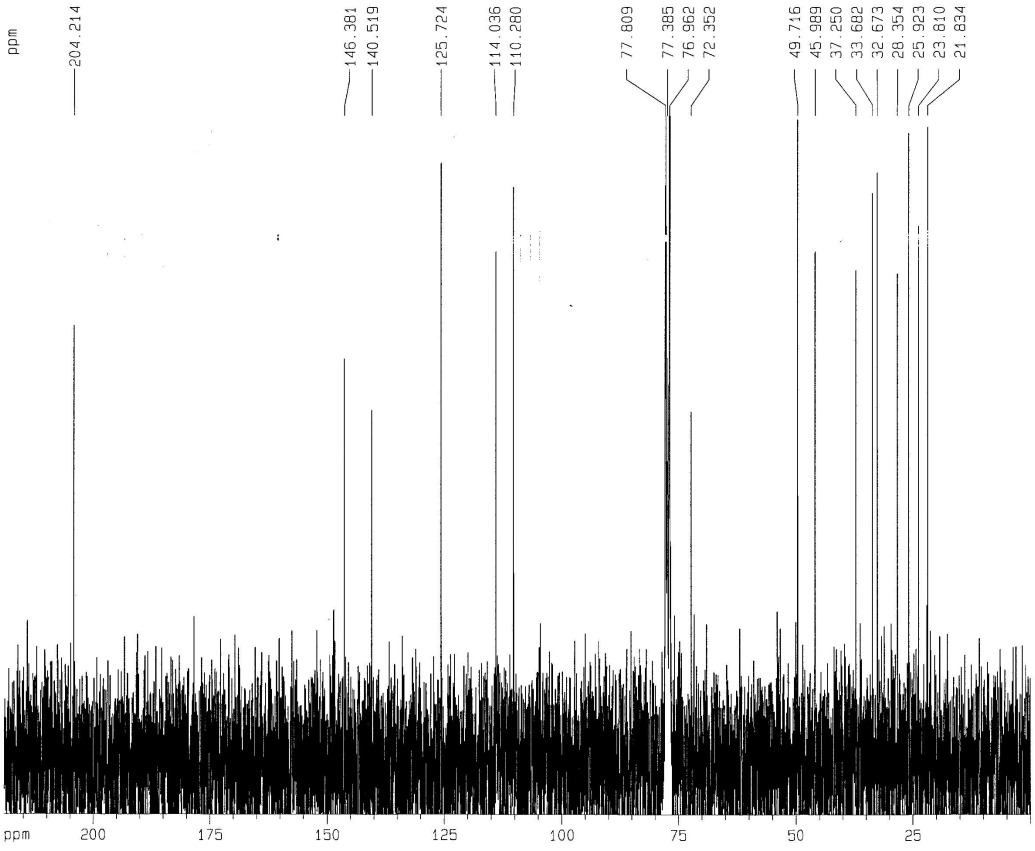




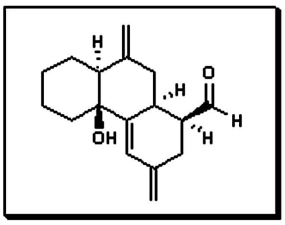

```
Current Data Parameters
NAME                 rc_398
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20030821
Time                  16.05
INSTRUM               av300
PROBHD   5 mm QNP  1H/1
PULPROG               zg30
TD                   30720
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               5081.301 Hz
FIDRES            0.165407 Hz
AQ                3.0228980 sec
RG                   203.2
DW                  98.400 usec
DE                   6.00 usec
TE                  300.0 K
D1              1.00000000 sec

============ CHANNEL f1 =============
NUC1                    1H
P1                   10.50 usec
PL1                  -3.00 dB
SFO1           300.1319477 MHz

F2 - Processing parameters
SI                      65536
SF             300.1300000 MHz
WDW                        EM
SSB                         0
LB                   0.10 Hz
GB                          0
PC                   1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                   8.000 ppm
F1                 2401.04 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             0.40000 ppm/cm
HZCM            120.05200 Hz/cm
```

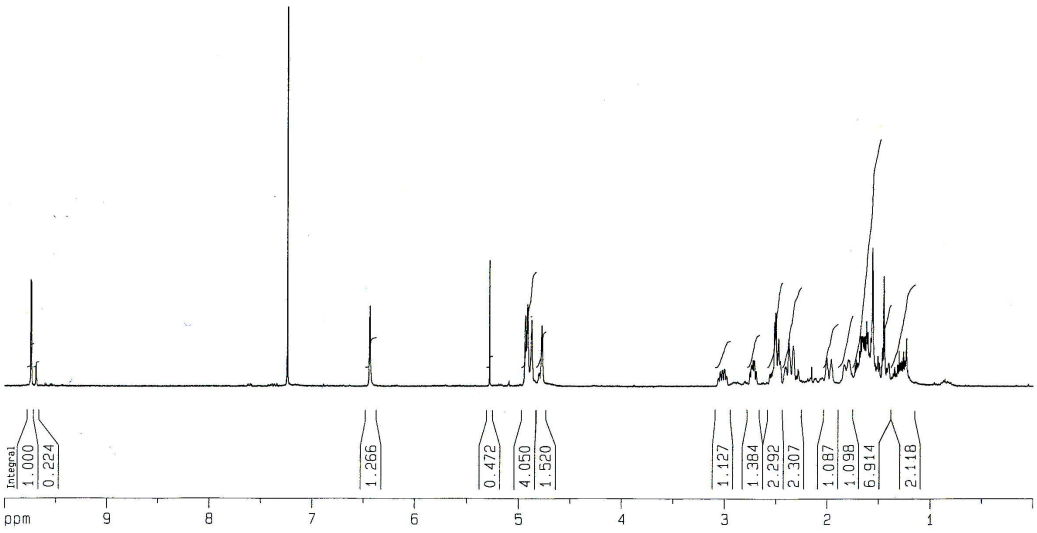


13C with proton decoupling

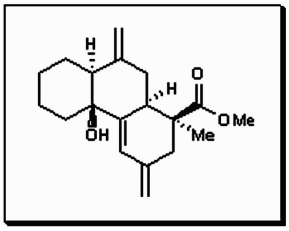



```
Current Data Parameters
NAME            rc_544_1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20031121
Time               17.03
INSTRUM            av300
PROBHD   5 mm QNP  1H/1
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   627
DS                     0
SWH            17985.611 Hz
FIDRES          0.548877 Hz
AQ              0.9110004 sec
RG                  6502
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                 5.00 usec
PL1               -6.00 dB
SFO1          75.4752653 MHz

============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2             70.00 usec
PL2               -3.00 dB
PL12              13.48 dB
PL13              15.63 dB
SFO2         300.1314860 MHz

F2 - Processing parameters
SI                 65536
SF            75.4677190 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 12.50 cm
F1P               220.000 ppm
F1              16602.90 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          11.00000 ppm/cm
HZCM          830.14490 Hz/cm
```

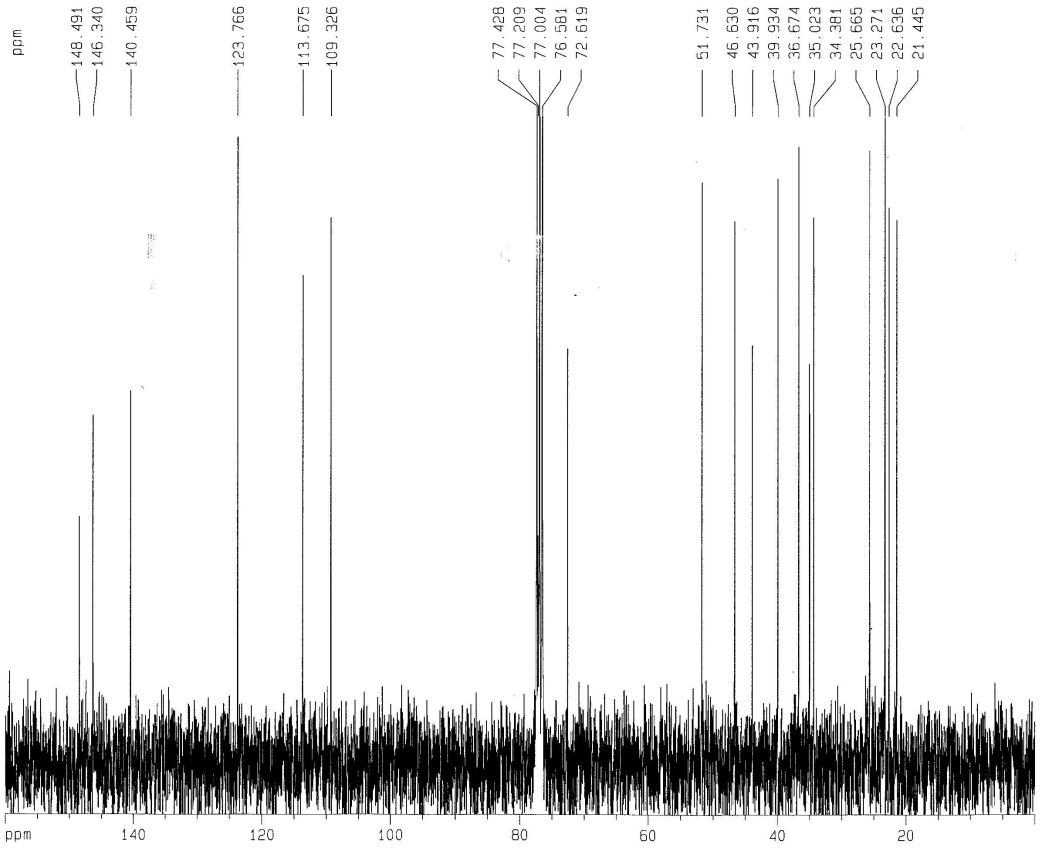




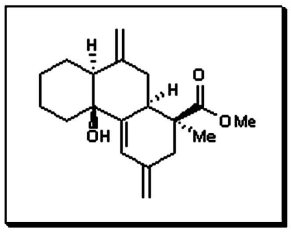

```
Current Data Parameters
NAME                  rc_544_1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20031121
Time                     16.57
INSTRUM                  av300
PROBHD   5 mm QNP  1H/1
PULPROG                  zg30
TD                       30720
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                  5081.301 Hz
FIDRES               0.165407 Hz
AQ                   3.0228980 sec
RG                       456.1
DW                     98.400 usec
DE                      6.00 usec
TE                      300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =============
NUC1                        1H
P1                       10.50 usec
PL1                      -3.00 dB
SFO1              300.1319477 MHz

F2 - Processing parameters
SI                       65536
SF                300.1300000 MHz
WDW                         EM
SSB                          0
LB                        0.10 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                     10.000 ppm
F1                    3001.30 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                 0.50000 ppm/cm
HZCM                150.06500 Hz/cm
```

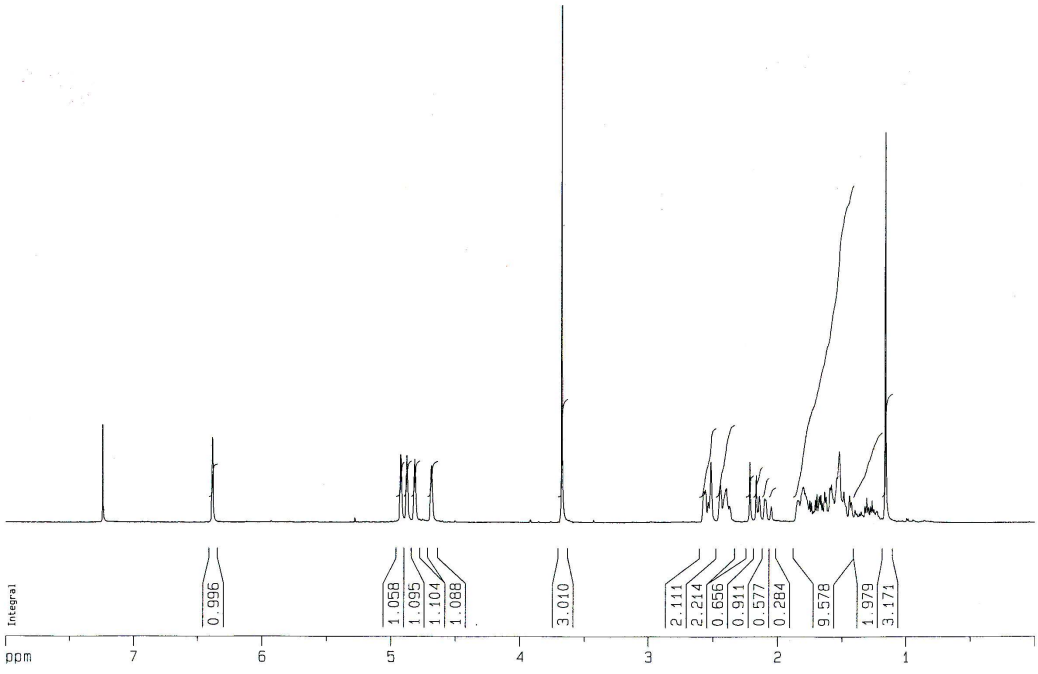


13C with proton decoupling

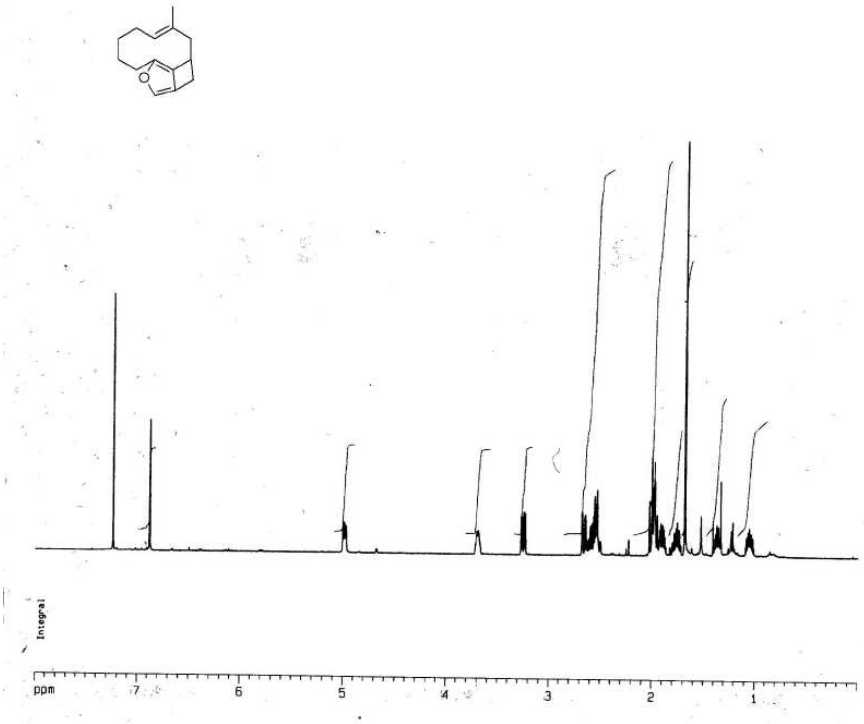



```
Current Data Parameters
NAME                  rc_508
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20031104
Time                  17.08
INSTRUM                av300
PROBHD   5 mm QNP  1H/1
PULPROG               zgpg30
TD                     32768
SOLVENT                CDCl3
NS                       398
DS                         0
SWH               17985.611 Hz
FIDRES             0.548877 Hz
AQ                 0.9110004 sec
RG                    7298.2
DW                    27.800 usec
DE                     6.00 usec
TE                    300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

========== CHANNEL f1 ==========
NUC1                      13C
P1                       5.00 usec
PL1                     -6.00 dB
SFO1              75.4752653 MHz

========== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   70.00 usec
PL2                     -3.00 dB
PL12                    13.48 dB
PL13                    15.63 dB
SFO2             300.1314860 MHz

F2 - Processing parameters
SI                        65536
SF                75.4677478 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
CY                        12.50 cm
F1P                      160.000 ppm
F1                     12074.84 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                  8.00000 ppm/cm
HZCM                603.74200 Hz/cm
```

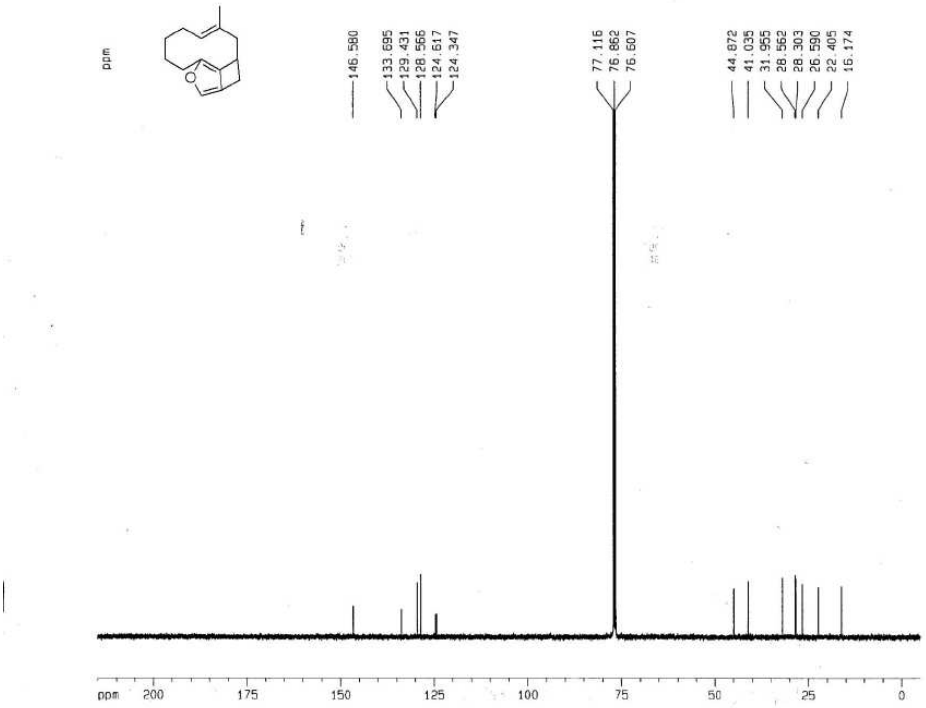




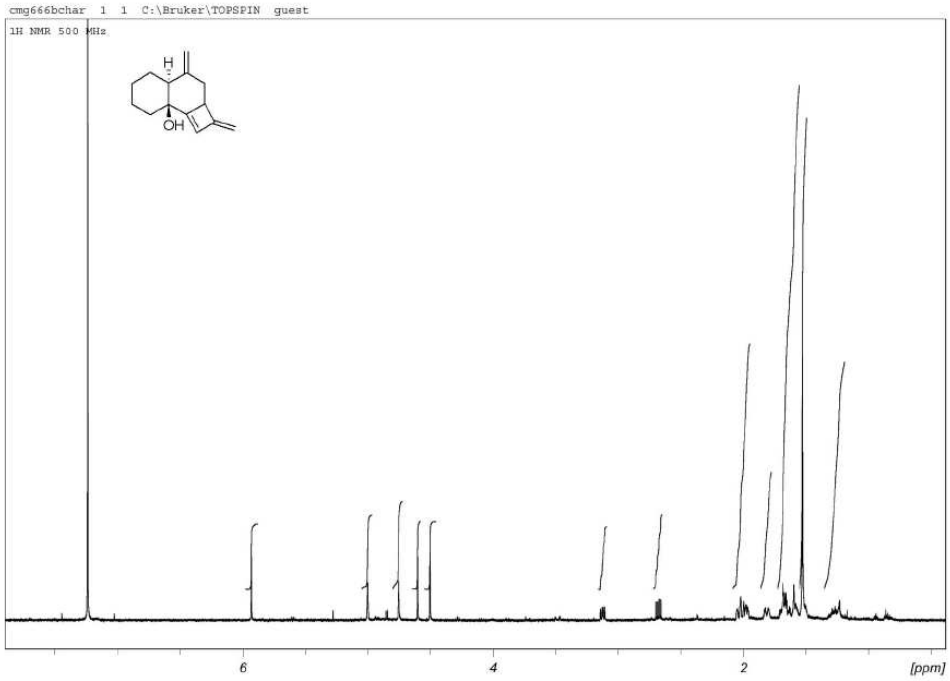

```
Current Data Parameters
NAME                 rc_508
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_             20031104
Time                 17.04
INSTRUM              av300
PROBHD   5 mm QNP  1H/1
PULPROG              zg30
TD                   30720
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              5081.301 Hz
FIDRES           0.165407 Hz
AQ               3.0228980 sec
RG                     362
DW                98.400 usec
DE                 6.00 usec
TE                 300.0 K
D1              1.00000000 sec

============ CHANNEL f1 =============
NUC1                   1H
P1                 10.50 usec
PL1                -3.00 dB
SFO1          300.1319477 MHz

F2 - Processing parameters
SI                    65536
SF            300.1300000 MHz
WDW                      EM
SSB                       0
LB                  0.10 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                  8.000 ppm
F1                2401.04 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             0.40000 ppm/cm
HZCM            120.05200 Hz/cm
```

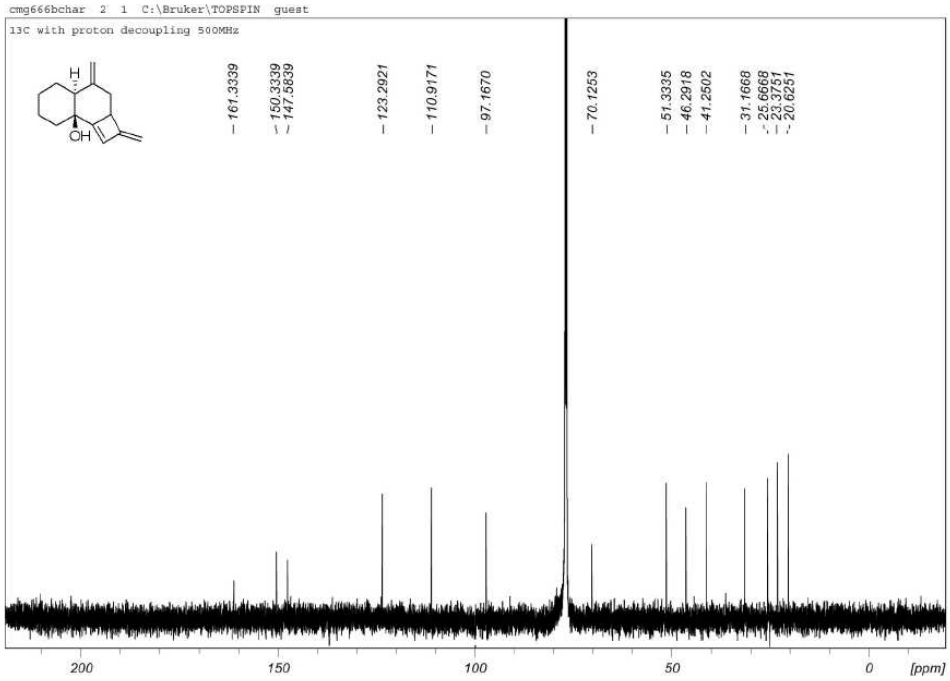



```
Current Data Parameters
NAME                 cmg672top
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20060420
Time                   18.19
INSTRUM                AV500WB
PROBHD     5 mm TBO BB/1H
PULPROG                 zg30
TD                     65536
SOLVENT              Acetone
NS                          16
DS                           0
SWH                 7440.476 Hz
FIDRES              0.113533 Hz
AQ                  4.4040694 sec
RG                      203.2
DW                     67.200 usec
DE                      6.00 usec
TE                     300.0 K
D1                0.01000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.50 usec
PL1                     0.00 dB
SFO1              500.1327766 MHz

F2 - Processing parameters
SI                         65536
SF                  500.1300049 MHz
WDW                           EM
SSB                            0
LB                      0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     10.00 cm
F1P                     8.000 ppm
F1                   4001.04 Hz
F2P                     0.000 ppm
F2                      0.00 Hz
PPMCM                0.40000 ppm/cm
HZCM               200.05200 Hz/cm
```




```
Current Data Parameters
NAME                 cmg672top
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20060420
Time                   18.25
INSTRUM                AV500WB
PROBHD     5 mm TBO BB/1H
PULPROG              zgpg30
TD                     65536
SOLVENT              CDCl3
NS                          1024
DS                           0
SWH                30030.029 Hz
FIDRES              0.458222 Hz
AQ                  1.0912244 sec
RG                      14600
DW                     16.650 usec
DE                      40.00 usec
TE                     300.0 K
D1                1.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

-------- CHANNEL f1 --------
NUC1                        13C
P1                     13.25 usec
PL1                     -3.00 dB
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  80.00 usec
PL2                     0.00 dB
PL12                   15.46 dB
PL13                   14.90 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                         32768
SF                  125.7578051 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     20.00 cm
F1P                   215.000 ppm
F1                  27037.93 Hz
F2P                    -5.000 ppm
F2                   -628.79 Hz
PPMCM               11.00000 ppm/cm
HZCM              1383.33582 Hz/cm
```


cmg666bchar 1 1 C:\Bruker\TOPSPIN guest
1H NMR 500 MHz
H
OH
6
4
2
[ppm]


cmg666bchar 2 1 C:\Bruker\TOPSPIN guest
13C with proton decoupling 500MHz
H
OH
161.3339
150.3339
147.5839
123.2921
110.9171
97.1670
70.1253
51.3335
46.2918
41.2502
31.1668
25.6668
23.3751
20.6251
200
150
100
50
0
[ppm]