Gold(I)-Catalyzed Synthesis of γ-Hydroxyketones from 5-Allyloxy-1-ynes

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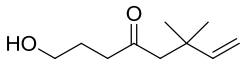
I. General Information

All commercially available chemicals were used without further purification. All solvents were dried and distilled according to the standard methods before use. $Au(PPh_3)Cl$ and $AgSbF_6$ were purchased from Aldrich Chemicals and stored in a dry-keeper. Au[*t*-Bu_2P(*o*-biphenyl)]Cl and Au[$P(C_6F_5)_3$]Cl were prepared according to the literature procedures.[1] Experiments were performed in flame-dried glasswares with rubber septa under a positive pressure of nitrogen. Reactions were monitored by thin-layer chromatography using UV light as a visualizing agent and acidic *p*-anisaldehyde, PMA/EtOH and heat as developing agent. ^{1}H and ^{13}C NMR spectra were recorded with 300 MHz spectrometer. ^{1}H NMR spectra were referenced to $CDCl_3$ (7.26 ppm) and reported as follows; chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, quint = quintet, m = multiplet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, ddt = doublet of doublet of triplets). Chemical shifts of the ^{13}C NMR spectra were measured relative to $CDCl_3$ (77.23 ppm).

II. General procedure for the gold(I)-catalyzed reaction

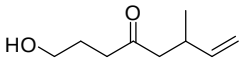
Compound 10



10

To a solution of $AgSbF_6$ (2.5 mg, 0.0073 mmol) in dry CH_2Cl_2 (1 mL) was added a solution of **11a** (5.6 mg, 0.0073 mmol) in dry CH_2Cl_2 (1 mL). The solution was stirred for 10 min. The resulting solution was filtered though a pad of celite and concentrated. The residue was dried over high vacuum for 2 hours and then cooled to 0°C. To this residue was added the solution of **8** (56 mg, 0.37mmol) in CH_2Cl_2 : H_2O = 10 : 1 (7.4 mL, pre-cooled to 0°C). The resulting green solution was stirred for 30 min. Triethylamine (1 mL) was added and the solution was stirred for 5 min. The resulting solution was filtered through a pad of silica and concentrated. The crude oil was purified by flash chromatography on silica gel (eluted with hexane : ethyl acetate = 70 : 30) to give the compound **10** as a colorless oil (60mg, 0.35mmol, 95% yield). R_f = 0.08 (hexane : ethyl acetate = 80 : 20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.10 (s, 6H), 1.77 (quint, *J* = 6.3 Hz, 2H), 2.03 (br s, 1H), 2.42 (s, 2H), 3.60 (t, *J* = 6.1 Hz, 2H), 4.90 - 4.96 (m, 2H), 5.89 (dd, *J* = 17.6 Hz, *J* = 10.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 26.5, 27.2, 36.6, 41.8, 54.5, 62.4, 111.1, 147.3, 210.8; IR: (cm^{-1}) ν 3397, 2966, 1640, 1456, 1371; HRMS calcd for $C_{10}H_{18}O_2$: 170.1307. found: 170. 1308.

Compound 13

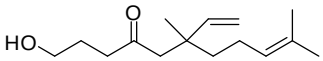


13

Using the representative procedure (performed at rt), a mixture of **12** (25 mg, 0.18 mmol), gold complex **11a** (6.7 mg, 0.0087 mmol) and $AgSbF_6$ (3.0 mg, 0.0087 mmol) were reacted to give **13** as a colorless oil. (25 mg, 0.16 mmol, 90 % yield). R_f = 0.17 (hexane: ethyl acetate = 70:30); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.00 (d, *J* = 6.7 Hz, 3H), 1.81 (quint, *J* = 6.5 Hz, 2H), 2.03 (s, 1H), 2.32 - 2.55 (m, 4H), 2.67 - 2.76 (m, 1H), 3.62 (t, *J* = 6.1 Hz, 2H), 4.90 – 5.00 (m, 2H), 5.73 (ddd, *J* = 17.2 Hz, *J* = 10.3 Hz, *J* = 6.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 20.0, 26.5, 33.6, 40.4, 49.7, 62.4, 113.3, 143.0, 210.7;

IR: (cm^{-1}) ν 3420, 3081, 2958, 1717, 1641, 1419, 1373, 915; HRMS calcd for $C_9H_{16}O_2$: 156.1150. found: 156.1153.

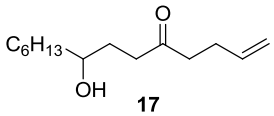
Compound 15



15

Using the representative procedure (performed at -15°C), a mixture of **14** (38 mg, 0.17 mmol), gold complex **11a** (6.7 mg, 0.0087 mmol) and $AgSbF_6$ (3.0 mg, 0.0087 mmol) were reacted to give **15** as a colorless oil. (30 mg, 0.13 mmol, 75 % yield). R_f = 0.09 (hexane: ethyl acetate = 80:20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.09 (s, 3H), 1.32-1.48 (m, 2H), 1.56 (s, 3H), 1.65 (s, 3H), 1.77 (quint, *J* = 6.5 Hz, 2H), 1.81 - 1.90 (m, 2H), 2.03 (s, 1H), 2.37 - 2.57 (m, 4H), 3.59 (t, *J* = 6.1, 2H), 4.92 (dd, *J* = 17.5 Hz, *J* = 1.0 Hz, 1H), 5.02 (dd, *J* = 10.8 Hz, *J* = 1.1 Hz, 1H), 5.02 - 5.08 (m, 1H), 5.82 (dd, *J* = 17.5, 10.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 17.8, 23.0, 23.0, 25.8, 26.5, 39.7, 41.0, 41.9, 52.9, 62.4, 112.4, 124.6, 131.6, 146.0, 210.8; IR: (cm^{-1}) ν 3415, 3083, 3020, 2965, 2926, 1700, 1375, 1060; HRMS calcd for $C_{15}H_{26}O_2$: 238.1933. found: 238.1930.

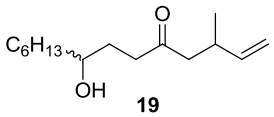
Compound 17



17

Using the representative procedure (performed at rt), a mixture of **16** (50 mg, 0.24 mmol), gold complex **11a** (18.4 mg, 0.024 mmol) and $AgSbF_6$ (8.2 mg, 0.024 mmol) were reacted to give **17** (mixed with cyclized form in $CDCl_3$ solution) as a white solid. (25 mg, 0.11 mmol, 46 % yield). R_f = 0.30 (hexane: ethyl acetate = 80:20), m.p = 39 – 41°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.8 Hz, 3H), 1.27 - 1.42 (m, 12H), 1.52 - 2.00 (m, 5H), 2.09 - 2.24 (m, 1H), 2.28 - 2.51 (m, 2H), 2.51 - 2.59 (m, 4H), 3.53 - 3.58 (m, 1H), 4.93 - 5.08 (m, 2H), 5.73 - 5.93 (m, 1H); {distinctive signals for cyclized form} δ = 3.92 - 4.02 (m, 1H), 4.13 - 4.22 (m, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 22.8, 25.8, 28.0, 29.5, 31.1, 32.0, 38.0, 39.4, 42.1, 71.6, 115.4, 137.3, 211.2; {distinctive signals for cyclized form} δ = 26.1, 26.3, 29.1, 29.1, 29.6, 30.4, 30.8, 35.9, 35.9, 37.0, 37.8, 39.9, 40.0, 78.9, 81.3, 106.6, 106.8, 114.7, 114.8, 138.7; IR: (cm^{-1}) ν 3419, 3079, 2928, 2857, 1714, 1456, 1377; HRMS calcd for $C_{14}H_{27}O_2$ (FAB^+): 227.2011. found: 227.2014.

Compound 19

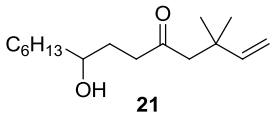


19

Using the representative procedure, a mixture of **18** (50 mg, 0.23 mmol), gold complex **11a** (3.4 mg, 0.0044 mmol) and $AgSbF_6$ (1.5 mg, 0.0044 mmol) were reacted to give **19** as a colorless oil. (45 mg, 0.19 mmol, the mixture of diastereomers = 1 : 1, 83 % yield). R_f = 0.12 (hexane: ether = 70:30); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.7 Hz, 3H), 1.01 (d, *J* = 6.8 Hz, 3H), 1.21 - 1.43 (m, 10H),

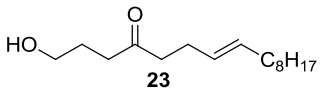
1.55 - 1.68 (m, 1H), 1.73 - 1.84 (m, 2H), 2.33 - 2.50 (m, 2H), 2.55 (t, J = 7.0 Hz, 2H), 2.68 - 2.77 (m, 1H), 3.50 - 3.60 (m, 1H), 4.91 - 5.02 (m, 1H), 5.69 - 5.80 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 20.0, 20.0, 22.8, 25.8, 29.5, 31.0, 32.0, 33.6, 38.0, 40.0, 40.0, 49.8, 71.7, 113.3, 113.3, 143.1, 143.1, 211.0; IR: (cm^{-1}) ν 3422, 3082, 2957, 2929, 2858, 1712; HRMS calcd for $C_{15}H_{28}O_2$: 240.2089. found: 240.2087.

Compound 21



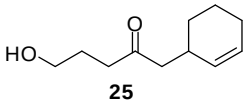
Using the representative procedure, a mixture of **20** (87 mg, 0.37 mmol), gold complex **11a** (5.6 mg, 0.0073 mmol) and $AgSbF_6$ (2.5 mg, 0.0073 mmol) were reacted to give **21** as a colorless oil. (83 mg, 0.33 mmol, 89 % yield). R_f = 0.32 (hexane: ethyl acetate = 80:20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, J = 6.6 Hz, 3H), 1.10 (s, 6H), 1.20 - 1.41 (m, 10H), 1.51 - 1.64 (m, 1H), 1.70 - 1.80 (m, 2H), 2.42 (s, 2H), 2.53 (t, J = 6.9 Hz, 2H), 3.50 - 3.60 (m, 1H), 4.91 - 4.97 (m, 2H), 5.90 (dd, J = 17.6 Hz, J = 10.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 22.8, 25.8, 27.2, 27.3, 29.5, 30.9, 32.0, 36.7, 38.0, 41.4, 54.5, 71.6, 111.0, 147.4, 211.0; IR: (cm^{-1}) ν 3418, 2958, 2929, 2858, 1710, 1468, 1363, 913; HRMS calcd for $C_{16}H_{30}O_2$: 254.2246. found: 254.2247.

Compound 23



Using the representative procedure (performed at rt), a mixture of **22** (33 mg, 0.14 mmol), gold complex **11a** (5.3 mg, 0.0069 mmol) and $AgSbF_6$ (2.4 mg, 0.0069 mmol) were reacted to give **23** as a white solid. (28 mg, 0.11 mmol, 77 % yield). R_f = 0.13 (hexane: ethyl acetate = 80:20), m.p = 46 – 47°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, J = 6.9 Hz, 3H), 1.25 - 1.30 (m, 12H), 1.78 - 1.98 (m, 5H), 2.48 - 2.57 (m, 4H), 3.64 (t, J = 6.0 Hz, 2H), 5.31 - 5.46 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 22.9, 26.7, 27.1, 29.4, 29.5, 29.7, 32.1, 32.7, 39.8, 43.0, 62.6, 128.3, 131.9, 211.3; IR: (cm^{-1}) ν 3341, 2954, 2918, 2849, 1704, 1463, 1437, 1056, 969; HRMS calcd for $C_{16}H_{30}O_2$: 254.2246. found: 254.2248.

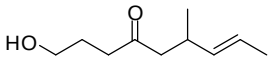
Compound 25



Using the representative procedure, a mixture of **24** (24 mg, 0.15 mmol), gold complex **11a** (5.6 mg, 0.0073 mmol) and $AgSbF_6$ (2.5 mg, 0.0073 mmol) were reacted to give **25** as a colorless oil. (25 mg, 0.14 mmol, 93 % yield). R_f = 0.12 (hexane: ethyl acetate = 80:20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.12 – 1.22 (m, 1H), 1.47 - 1.93 (m, 7H), 2.32 - 2.40 (m, 3H), 2.50 - 2.60 (m, 3H), 3.60 (t, J = 6.1 Hz,

2H), 5.42 - 5.46 (m, 1H), 5.64 - 5.67 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.2, 25.2, 26.6, 29.1, 31.4, 40.4, 49.4, 62.4, 128.2, 130.6, 211.2; IR: (cm^{-1}) ν 3397, 2925, 1700, 1448, 1371, 1058; HRMS calcd for $C_{11}H_{18}O_2$: 182.1307. found: 182.1309.

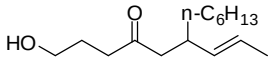
Compound 27



27

Using the representative procedure, a mixture of **26** (29 mg, 0.19 mmol), gold complex **11a** (6.9 mg, 0.0090 mmol) and $AgSbF_6$ (3.1 mg, 0.0090 mmol) were reacted to give **27** as a colorless oil. (28 mg, 0.16 mmol, 85 % yield). R_f = 0.07 (hexane: ethyl acetate = 80:20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.97 (d, *J* = 6.7 Hz, 3H), 1.61 (d, *J* = 5.9 Hz, 3H), 1.80 (quint, *J* = 6.5 Hz, 2H), 2.06 (s, 1H), 2.32 (dd, *J* = 15.5 Hz, *J* = 7.2 Hz, 1H), 2.42 (dd, *J* = 15.6 Hz, *J* = 7.0 Hz, 1H), 2.51 (t, *J* = 6.9 Hz, 2H), 2.60 - 2.69 (m, 1H), 3.61 (t, *J* = 6.1, 2H), 5.26 - 5.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 18.0, 20.7, 26.6, 33.0, 40.3, 50.3, 62.4, 124.0, 135.7, 211.1; IR: (cm^{-1}) ν 3419, 3027, 2958, 2881, 1710, 1410, 1373, 1059; HRMS calcd for $C_{10}H_{18}O_2$: 170.1307. found: 170.1310.

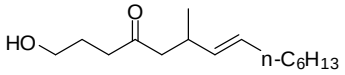
Compound 29



29

Using the representative procedure, a mixture of **28** (83 mg, 0.37 mmol), gold complex **11a** (5.6 mg, 0.0073 mmol) and $AgSbF_6$ (2.5 mg, 0.0073 mmol) were reacted to give **29** as a colorless oil. (77 mg, 0.32 mmol, 85 % yield). R_f = 0.13 (hexane: ethyl acetate = 80:20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.86 (t, *J* = 6.3 Hz, 3 H), 1.10 - 1.30 (m, 10H), 1.62 (dd, *J* = 6.3, *J* = 1.4 Hz, 3H), 1.80 (quint, *J* = 6.5 Hz, 2H), 1.92 (s, 1H), 2.36 - 2.53 (m, 5H), 3.61 (t, *J* = 6.1 Hz, 2H), 5.12 - 5.20 (m, 1H), 5.35 - 5.43 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 18.1, 22.8, 26.5, 27.2, 29.4, 32.0, 35.5, 39.0, 40.5, 49.1, 62.5, 125.6, 134.3, 211.4; IR: (cm^{-1}) ν 3423, 2930, 1645, 1456, 967; HRMS calcd for $C_{15}H_{28}O_2$: 240.2089. found: 240.2088.

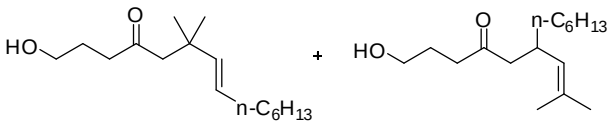
Compound 31



31

Using the representative procedure, a mixture of **30** (83 mg, 0.37 mmol), gold complex **11a** (5.6 mg, 0.0073 mmol) and $AgSbF_6$ (2.5 mg, 0.0073 mmol) were reacted to give **31** as a colorless oil. (74 mg, 0.31 mmol, 83 % yield). R_f = 0.22 (hexane: ethyl acetate = 70:30); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.5 Hz, 2H), 0.98 (d, *J* = 6.7 Hz, 3H), 1.20 – 1.35 (m, 8H), 1.77 – 1.86 (m, 3H), 1.91 – 1.97 (m, 2H), 2.30 – 2.47 (m, 2H), 2.52 (t, *J* = 6.9 Hz, 2H), 2.59 – 2.70 (m, 1H), 3.63 (t, *J* = 6.0 Hz, 2H), 5.25 – 5.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 20.9, 22.8, 26.6, 29.0, 29.7, 31.9, 32.7, 33.1, 40.4, 50.5, 62.6, 129.7, 134.5, 211.1; IR: (cm^{-1}) ν 3415, 2926, 2856, 1714, 1456, 1377, 1106, 969; HRMS calcd for $C_{15}H_{28}O_2$: 240.2089. found: 240.2086.

Mixture of 33 and 34

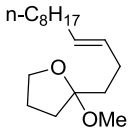


33 **34**

Using the representative procedure (performed at rt), a mixture of **32** (41 mg, 0.17 mmol), gold complex **11a** (6.7 mg, 0.0087 mmol) and $AgSbF_6$ (3.0 mg, 0.0087 mmol) were reacted to give mixture of **33** and **34** as a colorless oil. (32 mg, 0.13 mmol, the mixture of constitutional isomers = 1.3 : 1, 72 % yield). R_f = 0.32 (hexane: ethyl acetate = 70:30); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.83 – 0.88 (m, 6H), 1.07 (s, 6H), 1.24 - 1.35 (m, 16H), 1.69 - 1.82 (m, 4H), 1.93 - 2.00 (m, 4H), 2.26 - 2.41 (m, 4H), 2.47 - 2.52 (m, 4H), 2.72 - 2.75 (m, 1H), 3.60 (t, *J* = 6.1 Hz, 2H), 5.27 - 5.36 (m, 1H), 5.44 - 5.49 (m, 1H); {distinctive signals for [1,3]-product **34**} 1.60 (d, *J* = 1.0, 3H), 1.66 (d, *J* = 1.0, 3H), 4.77 - 4.80 (m, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 18.4, 22.8, 26.0, 26.6, 26.6, 27.3, 27.9, 29.0, 29.6, 29.8, 31.9, 32.0, 32.8, 34.8, 36.2, 40.6, 42.0, 49.4, 55.1, 62.5, 127.1, 128.4, 132.3, 138.9, 211.2, 211.6; IR: (cm^{-1}) ν 3419, 2957, 2926, 2856, 1710, 1456, 1377, 1059, 973; HRMS calcd for $C_{16}H_{30}O_2$: 254.2246. found: 254.2247.

III. Representative procedure for the gold(I)-catalyzed reaction with methanol

Compound 35

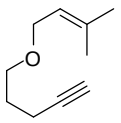


35

To a solution of $AgSbF_6$ (2.5 mg, 0.0073 mmol) in dry CH_2Cl_2 (1 mL) was added a solution of $Au[P(C_6F_5)_3]Cl$ (5.6 mg, 0.0073 mmol) in dry CH_2Cl_2 (1 mL). The solution was stirred for 10 min. The resulting solution was filtered though a pad of celite and concentrated. The residue was dried over high vacuum for 2 - 3 hours. To this residue was added the solution of **22** (34 mg, 0.14 mmol) and methanol (30 μL, 0.74 mmol) in dry CH_2Cl_2 (3.0 mL) at rt. The reaction mixture was stirred for 20 min. Triethylamine (1 mL) was added and the solution was stirred for 5 min. The resulting solution was concentrated under reduced pressure. The crude oil was purified by flash chromatography on silica gel (deactivated by triethylamine before use, eluted with hexane : ethyl acetate = 95 : 5) to give the compound **35** as a colorless oil (28 mg, 0.10mmol, 71% yield). R_f = 0.63 (hexane : ethyl acetate = 80 : 20); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.7 Hz, 3H), 1.26 - 1.32 (m, 12H), 1.53 - 2.07 (m, 10H), 3.17 (s, 3H), 3.84 - 3.90 (m, 2H), 5.37 - 5.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 22.9, 24.5, 28.1, 29.4, 29.5, 29.7, 29.8, 32.1, 32.8, 34.3, 35.7, 48.3, 67.5, 109.5, 129.7, 130.9; IR: (cm^{-1}) ν 2925, 2854, 1457, 1092, 1043, 966; HRMS calcd for $C_{17}H_{32}O_2$: 268.2402. found: 268.2404.

IV. Representative procedure for the preparation of substrates

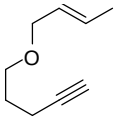
Procedure A (substrate 8)



8

The literature procedure was modified.[2] A solution of 4-pentyn-1-ol (800 mg, 9.51 mmol) in THF (4mL) was added dropwise to a suspension of sodium hydride (60% dispersion in mineral oil, 418 mg, 11 mmol) in THF (15 mL) at 0°C. After stirring for 30 min this temperature, prenyl bromide (1.29 mL, 9.99 mmol) was added. The reaction mixture was stirred for 15 hr at rt, then quenched with water. THF was removed under reduced pressure. The crude oil was extracted with Et_2O, dried over Na_2SO_4, concentrated and purified by flash chromatography on silica gel (pentane : ether = 90 : 10) to give the compound **8** as a colorless oil (1.20 g, 7.88 mmol, 83% yield). R_f = 0.69 (pentane : ether = 90 : 10). This spectral data was consistent with the literature value.[2]; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.68 (s, 3H), 1.75 (s, 3H), 1.80 (quint, *J* = 6.5 Hz, 2H), 1.94 (t, *J* = 2.6 Hz, 1H), 2.29 (td, *J* = 7.1 Hz, *J* = 2.6 Hz, 2H), 3.51 (t, *J* = 6.2 Hz, 2H), 3.95 (d, *J* = 6.8 Hz, 2H), 5.33 - 5.37 (m, 1H).

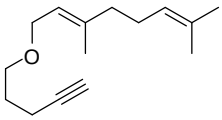
Substrate 12



12

Procedure A : Yield = 76% (mixture of olefin isomers); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.66 - 1.84 (m, 5H), 1.94 (t, *J* = 2.6 Hz, 1H), 2.26 - 2.31 (m, 2H), 3.50 (t, *J* = 6.3 Hz, 2H), 3.89 (d, *J* = 6.2 Hz, 2H), 5.51 - 5.62 (m, 1H), 5.66 - 5.77 (m, 1H); {distinctive signals for Z-isomer} δ = 4.03 (d, *J* = 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.4, 15.5, 18.0, 28.9, 66.4, 68.6, 68.6, 68.7, 71.8, 84.2, 127.2, 127.9, 128.0, 129.6; IR: (cm^{-1}) ν 3306, 3020, 2938, 2857, 2119, 1446, 1363, 1109, 967; HRMS calcd for $C_9H_{14}O$: 138.1045. found: 138.1045.

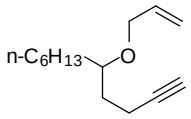
Substrate 14



14

Procedure A : Yield = 72%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.60 (s, 3H), 1.67 - 1.71 (m, 6H), 1.80 (quint, *J* = 6.7 Hz, 2H), 1.94 (t, *J* = 2.7 Hz, 1H), 2.00 - 2.14 (m, 4H), 2.29 (td, *J* = 7.1 Hz, *J* = 2.6 Hz, 2H), 3.51 (t, *J* = 6.2 Hz, 2H), 3.98 (d, *J* = 6.7 Hz, 2H), 5.07 - 5.12 (m, 1H), 5.32 - 5.37 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.5, 16.7, 17.9, 25.9, 26.6, 28.9, 39.8, 67.6, 68.6, 68.6, 84.3, 121.1, 124.2, 131.8, 140.3; IR: (cm^{-1}) ν 3311, 2926, 2857, 1669, 1436, 1377, 1107; HRMS calcd for $C_{15}H_{24}O$: 220.1827. found: 220.1826.

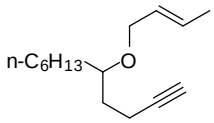
Substrate 16



16

Procedure A : Yield = 76%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.28 - 1.54 (m, 10H), 1.65 - 1.72 (m, 2H), 1.92 (t, *J* = 2.6 Hz, 1H), 2.49 (td, *J* = 7.2 Hz, *J* = 2.6 Hz, 2H), 3.42 (quint, *J* = 5.9 Hz, 1H), 3.92 - 4.04 (m, 2H), 5.11 - 5.15 (m, 1H), 5.22 - 5.29 (m, 1H), 5.85 - 5.98 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 14.8, 22.8, 25.4, 29.6, 32.0, 33.1, 33.9, 68.4, 70.4, 77.7, 84.7, 116.7, 135.5; IR: (cm^{-1}) ν 3313, 2930, 2859, 2120, 1647, 1459, 1342, 1134; HRMS calcd for $C_{15}H_{24}O(MH^+)$: 209.1905. found: 209.1902.

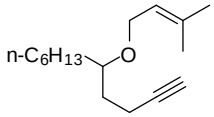
Substrate 18



18

Procedure A : Yield = 84% (mixture of olefin isomers); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.6 Hz, 3H), 1.25 - 1.51 (m, 10H), 1.60 - 1.71 (m, 5H), 1.92 (t, *J* = 2.6 Hz, 1H), 2.24 - 2.29 (m, 2H), 3.35 - 3.43 (m, 1H), 3.58 - 4.07 (m, 2H), 5.52 - 5.73 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.4, 14.3, 14.8, 18.0, 22.8, 25.5, 29.7, 32.0, 33.2, 34.1, 64.5, 68.3, 68.4, 70.2, 77.4, 84.8, 127.6, 127.6, 128.3, 129.3; IR: (cm^{-1}) ν 3314, 2930, 2858, 1456, 1094, 966; HRMS calcd for $C_{15}H_{26}O$: 222.1984. found: 222.1982.

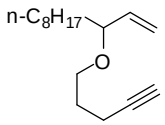
Substrate 20



20

Procedure A : Yield = 68%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.6 Hz, 3H), 1.25 - 1.54 (m, 10H), 1.65 - 1.72 (m, 5H), 1.74 (s, 3H), 1.93 (t, *J* = 2.6 Hz, 1H), 2.25 - 2.31 (m, 2H), 3.39 (quint, *J* = 5.9 Hz, 1H), 3.93 - 3.99 (m, 2H), 5.33 - 5.38 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 14.9, 18.2, 22.8, 25.5, 26.0, 29.7, 32.1, 33.3, 34.1, 65.8, 68.3, 77.4, 84.8, 121.8, 136.8; IR: (cm^{-1}) ν 3314, 2930, 2858, 1447, 1377, 1069; HRMS calcd for $C_{16}H_{28}O$: 236.2140. found: 236.2137.

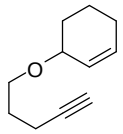
Procedure B (substrate 22)



22

A solution of undec-1-en-3-ol (380 mg, 2.23 mmol) in DMF (1 mL) was added dropwise to a suspension of sodium hydride (60 w% in mineral oil, 107 mg, 2.7 mmol) in DMF (1 mL) at 0°C. After stirring for 30 min this temperature, a solution of homopropargyl bromide (295 mg, 2.01 mmol) in DMF (0.5 mL) was added. The reaction mixture was stirred for 20 hr at rt, then diluted with water and extracted with Et_2O. The organic layer was dried over Na_2SO_4, concentrated and purified by flash chromatography on silica gel (hexane : ethyl acetate = 95 : 5) to give the compound **22** as a colorless oil (192 mg, 0.812 mmol, 40% yield, 50% of starting material was recovered). R_f = 0.63 (hexane : ethyl acetate = 90 : 10); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.6 Hz, 3H), 1.26 - 1.59 (m, 14H), 1.77 (quint, *J* = 6.6 Hz, 2H), 1.93 (t, *J* = 2.6 Hz, 1H), 2.29 (td, *J* = 7.2 Hz, *J* = 2.6 Hz, 2H), 3.31 - 3.38 (m, 1H), 3.54 - 3.63 (m, 2H), 5.13 - 5.19 (m, 2H), 5.61 - 5.72 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 15.6, 22.9, 25.6, 29.1, 29.5, 29.8, 29.8, 32.1, 35.7, 66.9, 68.5, 81.7, 84.4, 116.5, 139.7; IR: (cm^{-1}) ν 3314, 3078, 2926, 2856, 1467, 1323, 1102, 993, 924; HRMS calcd for $C_{16}H_{29}O$ (MH^+): 237.2218. found: 237.2221.

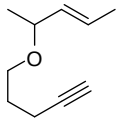
Substrate 24



24

Procedure B : Yield = 20%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.53 - 1.83 (m, 6H), 1.93 (t, *J* = 2.6 Hz, 1H), 1.95 - 2.03 (m, 2H), 2.30 (td, *J* = 7.0 Hz, *J* = 2.6 Hz, 2H), 3.52 - 3.65 (m, 1H), 3.83 - 3.85 (m, 1H), 5.74 - 5.78 (m, 1H), 5.82 - 5.87 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.5, 19.5, 25.5, 28.5, 29.3, 66.7, 68.5, 73.1, 84.4, 128.1, 131.0; IR: (cm^{-1}) ν 3306, 3027, 2937, 2862, 1436, 1319, 1100; HRMS calcd for $C_{11}H_{17}O$ (MH^+): 165.1279. found: 165.1281.

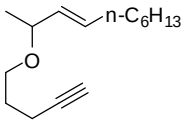
Substrate 26



26

Procedure B : Yield = 30%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.21 (d, *J* = 6.3 Hz, 3H), 1.67 (dd, *J* = 9.1 Hz, *J* = 1.5 Hz, 3H), 1.76 (quint, *J* = 6.7 Hz, 2H), 1.93 (t, *J* = 2.7 Hz, 1H), 2.25 - 2.30 (m, 2H), 3.33 - 3.43 (m, 1H), 3.47 - 3.55 (m, 1H), 3.76 (quint, *J* = 6.7 Hz, 1H), 5.36 - 5.39 (m, 1H), 5.57 - 5.64 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.5, 17.8, 21.7, 29.1, 66.4, 68.4, 76.7, 84.4, 127.3, 133.7; IR: (cm^{-1}) ν 3311, 2930, 2858, 1446, 1371, 1102, 967; HRMS calcd for $C_{10}H_{16}O$: 152.1201. found: 152.1201.

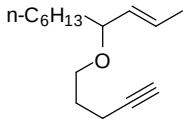
Substrate 28



28

Procedure B : Yield = 42%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.7 Hz, 3H), 1.21 (d, *J* = 6.3 Hz, 3H), 1.23 - 1.39 (m, 9H), 1.76 (quint, *J* = 6.7 Hz, 2H), 1.92 (t, *J* = 2.6 Hz, 1H), 1.99 - 2.05 (m, 2H), 2.25 - 2.31 (m, 2H), 3.33 - 3.40 (m, 1H), 3.47 - 3.56 (m, 1H), 3.77 (quint, *J* = 6.7 Hz, 1H), 5.31 (ddt, *J* = 15.4 Hz, *J* = 7.7 Hz, *J* = 1.4 Hz, 1H), 5.58 (dt, *J* = 15.4 Hz, *J* = 6.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 15.6, 21.9, 22.8, 29.0, 29.1, 29.4, 31.9, 32.4, 66.4, 68.4, 76.8, 84.4, 132.3, 133.0; IR: (cm^{-1}) ν 3314, 2928, 2856, 1456, 1370, 1102, 969; HRMS calcd for $C_{15}H_{26}O$: 222.1984. found: 222.1981.

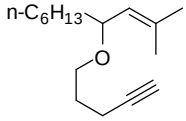
Substrate 30



30

Procedure B : Yield = 37%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.6 Hz, 3H), 1.26 - 1.59 (m, 10H), 1.65 - 1.79 (m, 5H), 1.92 (t, *J* = 2.6 Hz, 1H), 2.27 (td, *J* = 7.2 Hz, *J* = 2.5 Hz, 2H), 3.27 - 3.36 (m, 1H), 3.50 - 3.58 (m, 2H), 5.24 - 5.32 (m, 1H), 5.52 - 5.64 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 15.6, 17.9, 22.8, 25.7, 29.1, 29.5, 32.1, 35.9, 66.5, 68.4, 81.2, 84.5, 128.1, 132.7; IR: (cm^{-1}) ν 3314, 2930, 2858, 2121, 1671, 1456, 1100, 968; HRMS calcd for $C_{15}H_{27}O$ (MH^+): 223.2062. found: 223.2057.

Substrate 32



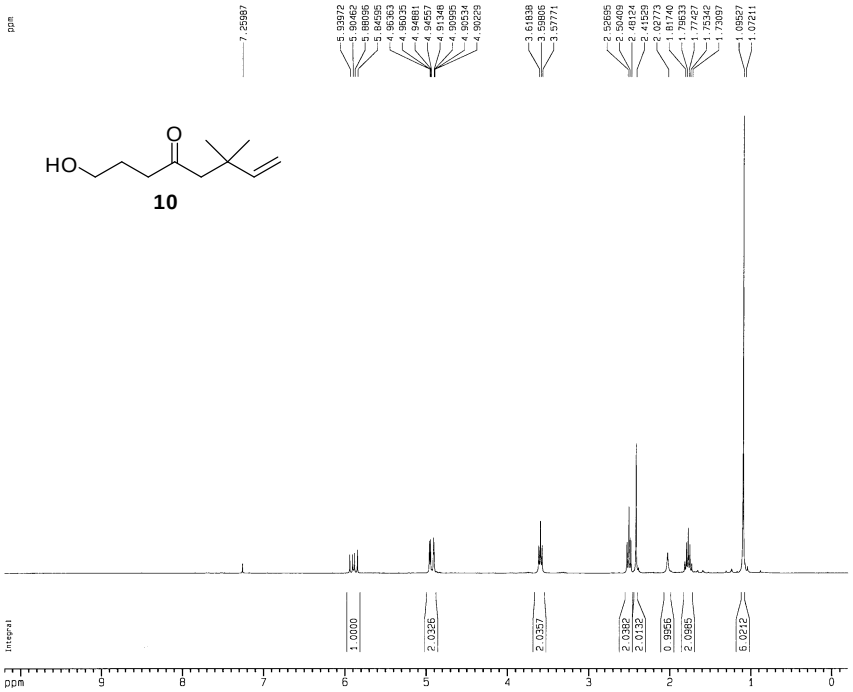
32

Procedure B : Yield = 29%; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.4 Hz, 3H), 1.20 - 1.58 (m, 10H), 1.65 (s, 3H), 1.69 - 1.78 (m, 5H), 1.91 (t, *J* = 2.6 Hz, 1H), 2.24 - 2.29 (m, 2H), 3.28 - 3.36 (m, 1H), 3.47 - 3.54 (m, 1H), 3.87 - 3.94 (m, 1H), 4.99 - 5.02 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.3, 15.6, 18.5, 22.9, 25.6, 26.1, 29.2, 29.6, 32.1, 36.0, 66.3, 68.4, 76.2, 84.5, 127.0, 135.3; IR: (cm^{-1}) ν 3314, 2930, 2858, 1446, 1377, 1100; HRMS calcd for $C_{16}H_{28}O$: 236.2140. found: 236.2137.

References:

1. Shin, S. *Bull. Kor. Chem. Soc.* **2005**, *26*, 1925.
2. Ito, H.; Ito, S.; Sasaki, Y.; Matsuura, K.; Sawamura, M. *J. Am. Chem. Soc.* **2007**, *129*, 14856.

MR-01-111-D 1 1 v yhr 20100418



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FIDRES 0.145322 Hz
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DE 4.50 usec
TE 300.0 K
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P1 11.40 usec
DE 4.50 usec
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PL1 -4.00 dB

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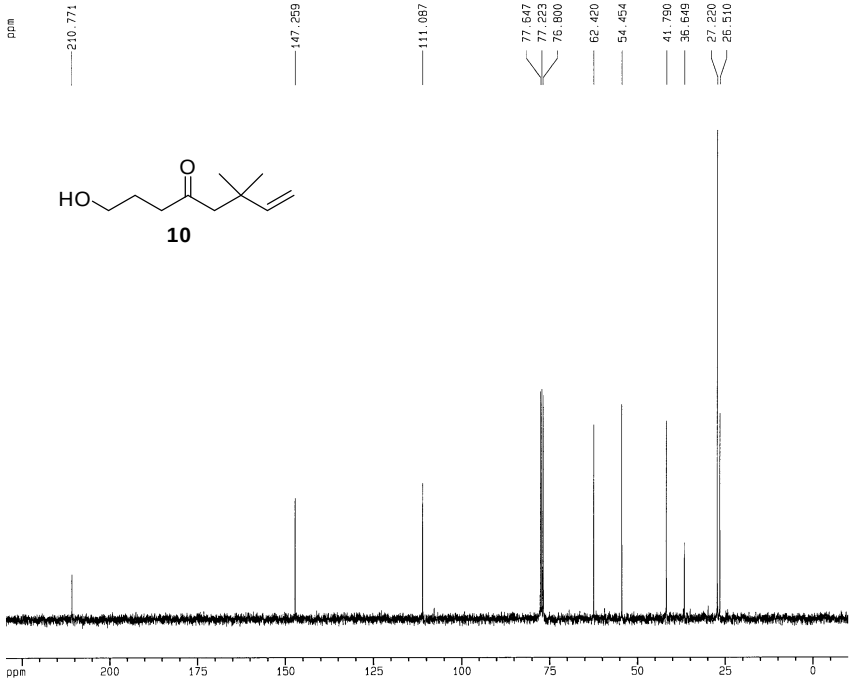
MR-01-111-D 13 1 v yhr 20100418



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AQ 1.4103156 sec
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NUC2 1H
PL2 -4.00 dB
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DE 4.50 usec
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NUC1 13C
PL1 -2.00 dB
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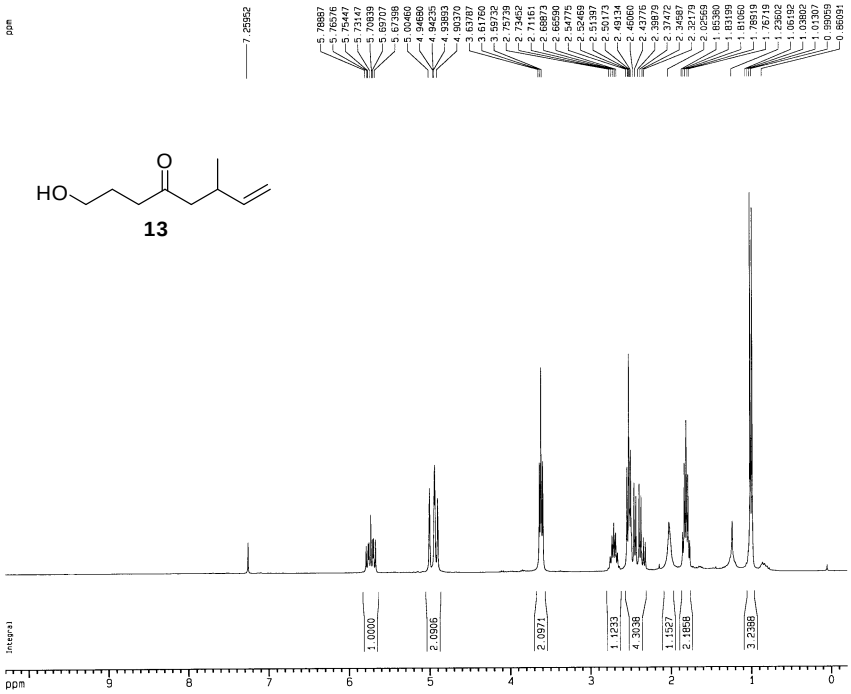
MR-01-132-P 1 1 v yhr 20100322

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PROCNO            1

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FIDRES       0.145322 Hz
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RG               128
DW           105.000 usec
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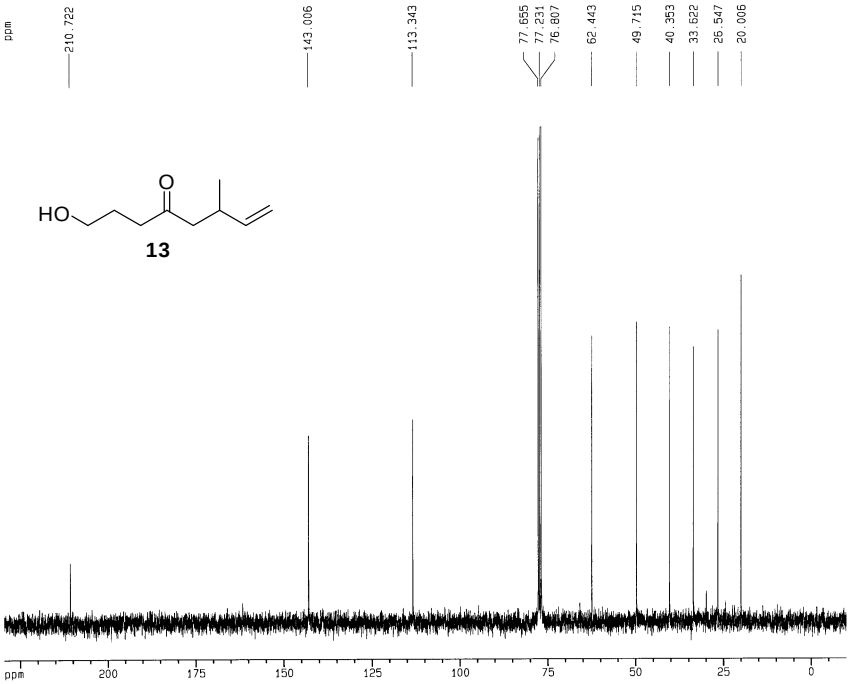
MR-01-132-D 13 1 v yhr 20100418

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NUC2              1H
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PL12           15.80 dB
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NUC1              13C
PL1            -2.00 dB
D11         0.03000000 sec

F2 - Processing parameters
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WDW               EM
SSB                0
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GB                 0
PC              1.00

1D NMR plot parameters
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F2P          -10.000 ppm
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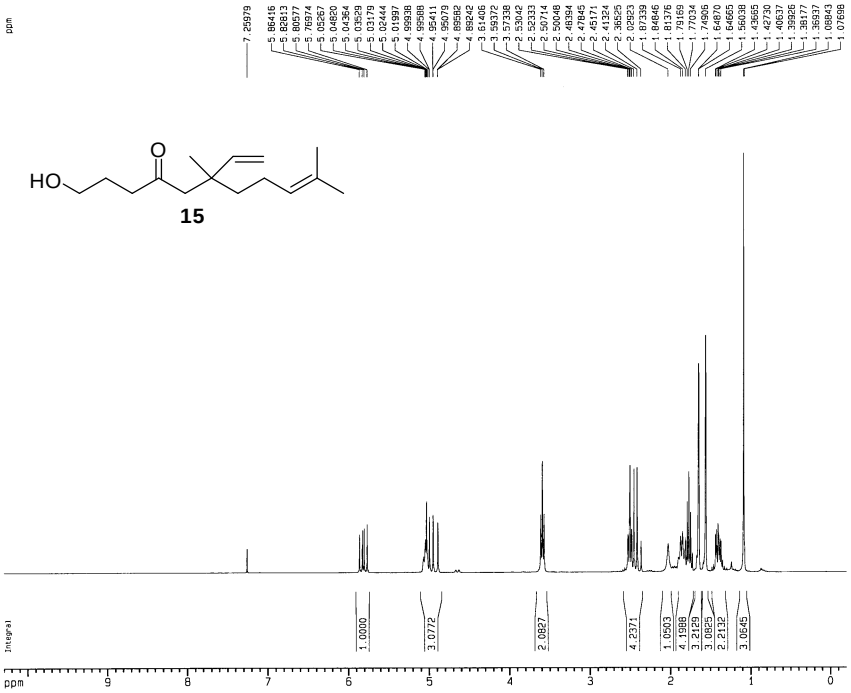
cjy-4-147-1 1 1 v yhr 20100420

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PROCNO              1

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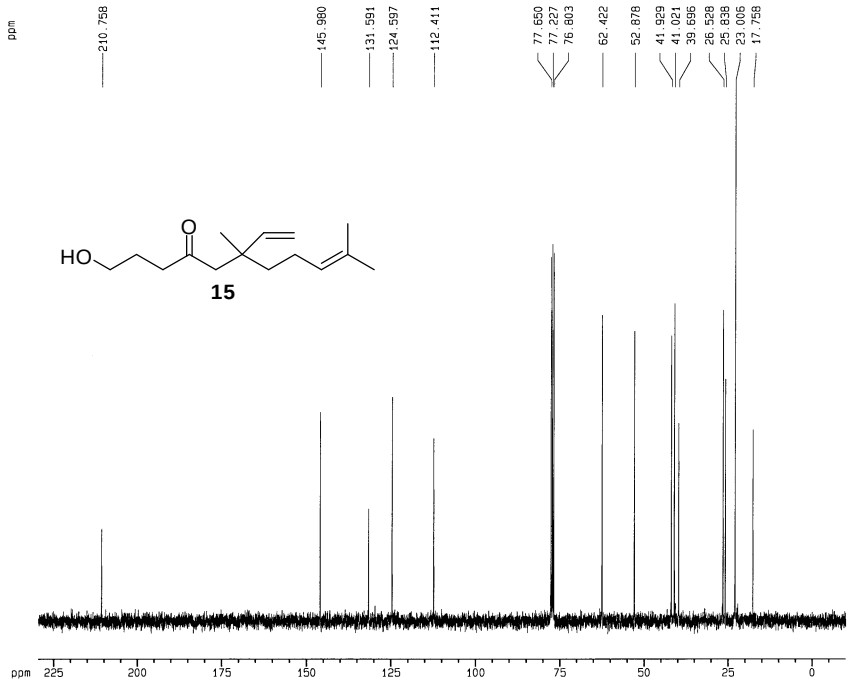
FIDRES         0.145322 Hz
AQ            3.4406900 sec
RG                  128
DW              105.000 usec
DE                4.50 usec
TE               300.0 K
D1            2.50000000 sec
P1               11.40 usec
DE                4.50 usec
SFO1         300.1318365 MHz
NUC1                1H
PL1               -4.00 dB

F2 - Processing parameters
SI                 16384
SF           300.1300069 MHz
WDW                  EM
SSB                   0
LB                 0.20 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
F1P               10.300 ppm
F1               3091.34 Hz
F2P              -0.200 ppm
F2               -60.03 Hz
PPMCM           0.47727 ppm/cm
HZCM          143.24385 Hz/cm
```

cjy-4-147-1 13 1 v yhr 20100420

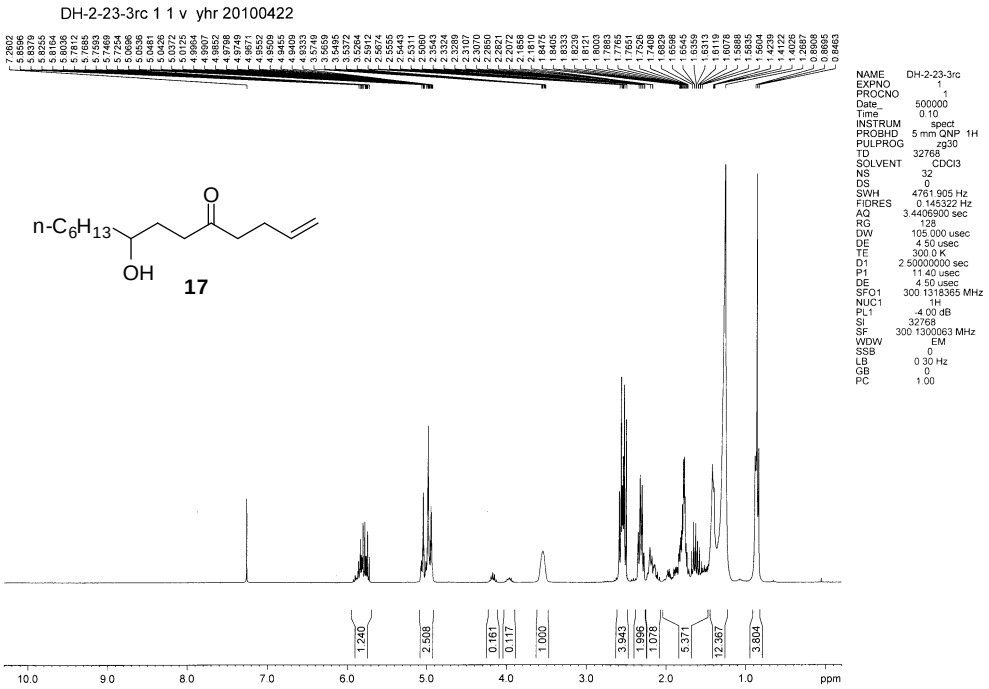


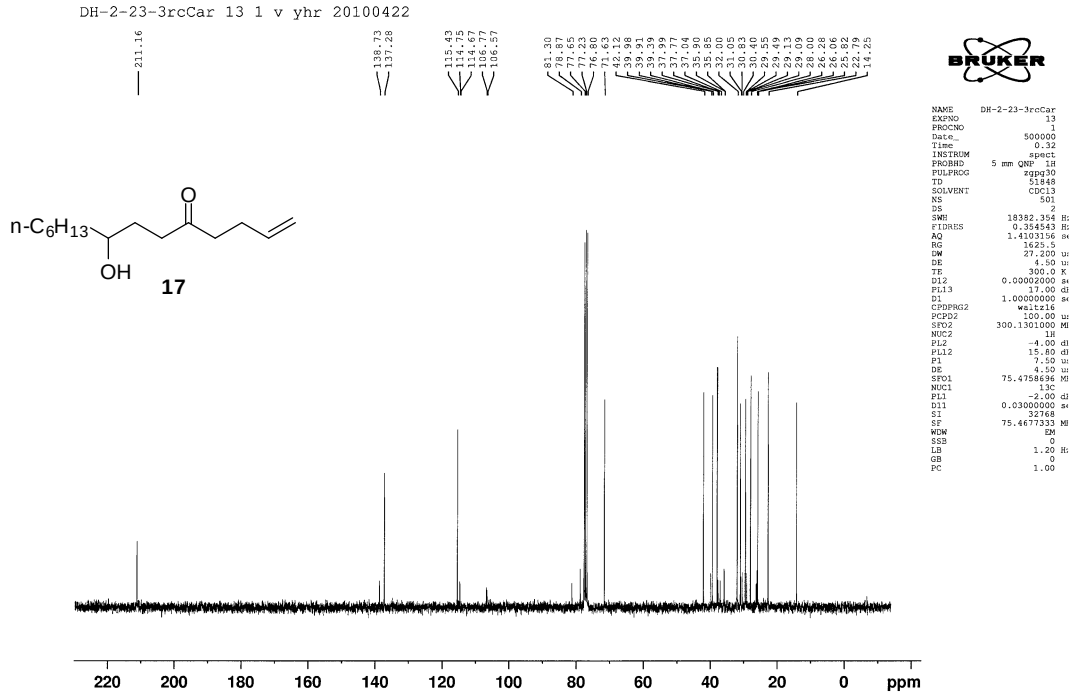

```
Current Data Parameters
NAME          cjy-4-147-1
EXPNO              13
PROCNO              1

F2 - Acquisition Parameters
Date_             500000
Time               21.00
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG           zgpg30
TD                51848
SOLVENT           CDCl3
NS                  301
DS                    0
SWH           18382.354 Hz
FIDRES         0.354543 Hz
AQ            1.4103156 sec
RG                 1625.5
DW              27.200 usec
DE                4.50 usec
TE               300.0 K
D12           0.00002000 sec
PL13             17.00 dB
D1            1.00000000 sec
CPDPRG2          waltz16
PCPD2           100.00 usec
SFO2         300.1301000 MHz
NUC2                1H
PL2               -4.00 dB
PL12              15.60 dB
P1                 7.50 usec
DE                4.50 usec
SFO1          75.4758696 MHz
NUC1                13C
PL1               -2.00 dB
D11           0.03000000 sec

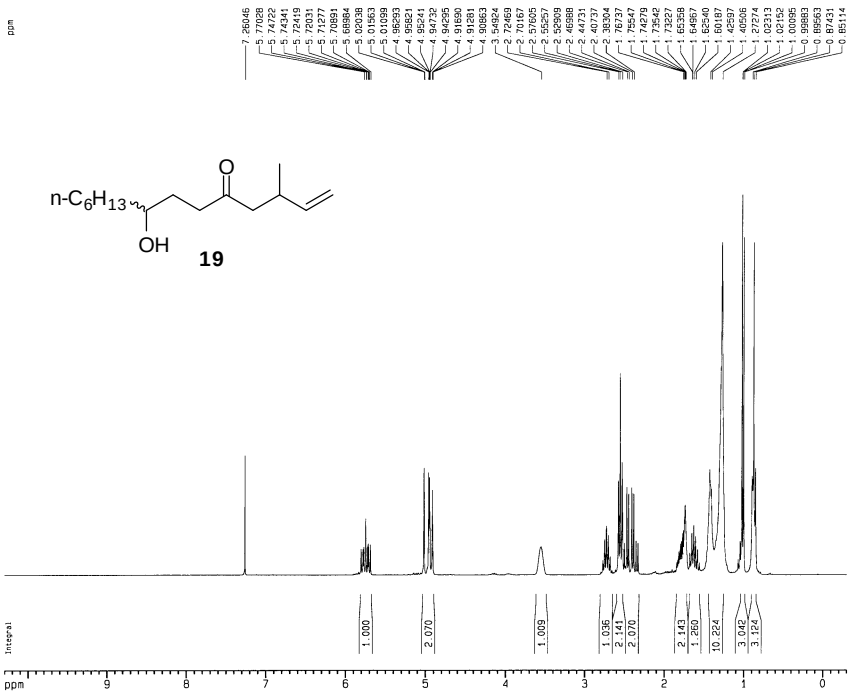
F2 - Processing parameters
SI                 32768
SF           75.4677350 MHz
WDW                  EM
SSB                   0
LB                 1.20 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
F1P              240.000 ppm
F1              18112.26 Hz
F2P              -10.000 ppm
F2              -754.68 Hz
PPMCM          11.36364 ppm/cm
HZCM          857.58789 Hz/cm
```


DH-2-23-3rc 1 1 v yhr 20100422
n-C6H13
OH
17
NAME DH-2-23-3rc
EXPNO 1
PROCNO 1
Date_ 500000
Time 0.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 128
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB
SI 32768
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.240
2.508
0.161
0.117
1.000
3.943
1.996
1.078
5.371
12.367
3.804
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm


DH-2-23-3rcCar 13 1 v yhr 20100422
211.16
138.73
137.28
115.43
114.75
114.67
106.77
106.57
81.30
78.87
77.65
77.23
76.80
73.63
42.12
39.98
39.91
39.39
37.99
37.77
37.04
35.90
35.85
32.00
31.65
31.03
30.40
29.55
29.49
29.13
29.00
28.00
26.28
26.06
25.82
25.79
14.25
n-C6H13
OH
17
BRUKER
NAME DH-2-23-3rcCar
EXPNO 13
PROCNO 1
Date_ 500000
Time 0.32
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 501
DS 2
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758696 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec
SI 32768
SF 75.4677333 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00
220
200
180
160
140
120
100
80
60
40
20
0
ppm

DH-2-15-1 1 1 v yhr 20100413



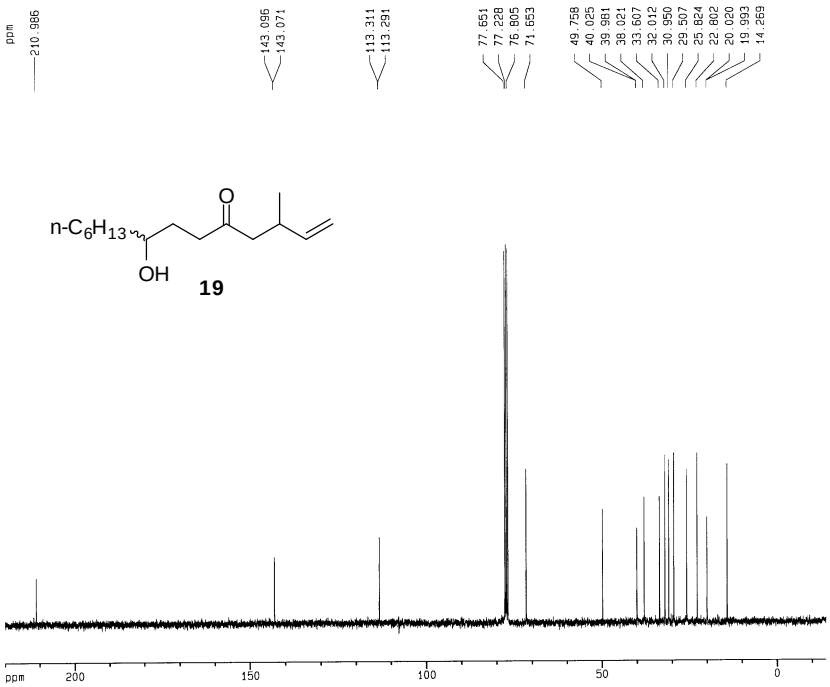

```
Current Data Parameters
NAME          DH-2-15-1
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_           500000
Time             22.32
INSTRUM          spect
PROBHD     5 mm QNP  1H
PULPROG          zg30
TD               32768
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           4761.905 Hz
FIDRES        0.145322 Hz
AQ           3.4406900 sec
RG               228.1
DW            105.000 usec
DE              4.50 usec
TE             300.0 K
D1           2.50000000 sec
P1              11.40 usec
DE              4.50 usec
SFO1       300.1318365 MHz
NUC1              1H
PL1            -4.00 dB

F2 - Processing parameters
SI              16384
SF         300.1300066 MHz
WDW               EM
SSB                0
LB              0.20 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX             22.00 cm
F1P            10.300 ppm
F1            3091.34 Hz
F2P            -0.300 ppm
F2            -90.04 Hz
PPMCM         0.48182 ppm/cm
HZCM        144.60809 Hz/cm
```

DH-2-15-1carb 13 1 v yhr 20100414



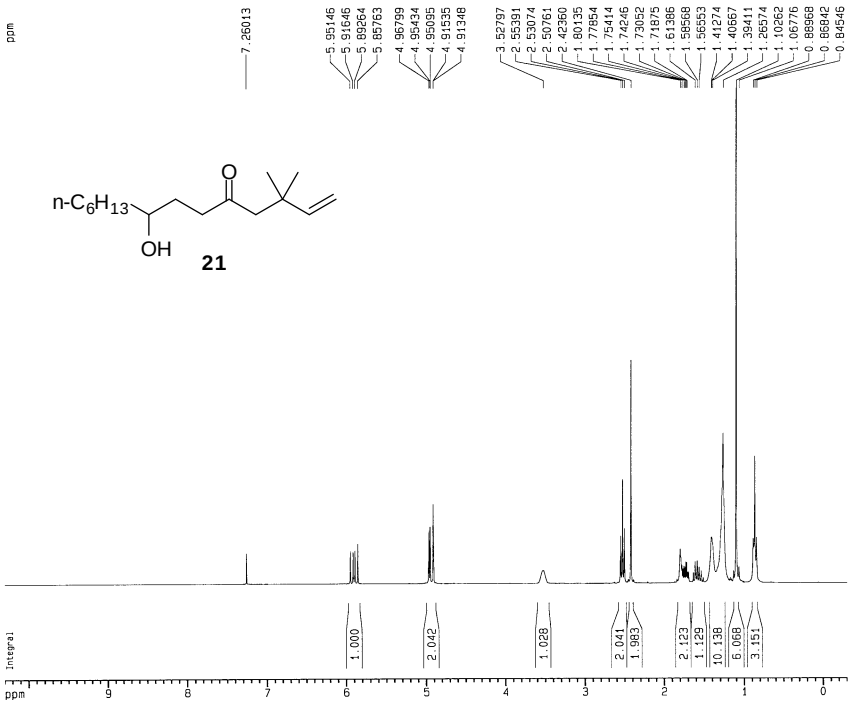

```
Current Data Parameters
NAME      DH-2-15-1carb
EXPNO              13
PROCNO              1

F2 - Acquisition Parameters
Date_           500000
Time             14.13
INSTRUM          spect
PROBHD     5 mm QNP  1H
PULPROG        zgpg30
TD               51648
SOLVENT          CDCl3
NS                 901
DS                   2
SWH          18382.354 Hz
FIDRES        0.354543 Hz
AQ           1.4103156 sec
RG              1625.5
DW             27.200 usec
DE              4.50 usec
TE             300.0 K
D12          0.00002000 sec
PL13            17.00 dB
D1           1.00000000 sec
CPDPRG2        waltz16
PCPD2          100.00 usec
SFO2       300.1301000 MHz
NUC2              1H
PL2             -4.00 dB
PL12            15.80 dB
P1               7.50 usec
DE              4.50 usec
SFO1        75.4758696 MHz
NUC1              13C
PL1             -2.00 dB
D11          0.03000000 sec

F2 - Processing parameters
SI              32768
SF         75.4677328 MHz
WDW               EM
SSB                0
LB              1.20 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX             22.00 cm
F1P           220.000 ppm
F1           16602.90 Hz
F2P           -20.000 ppm
F2           -1509.36 Hz
PPMCM        10.90909 ppm/cm
HZCM        823.28436 Hz/cm
```

DH-1-295-1 1 1 v yhr 20100409



21

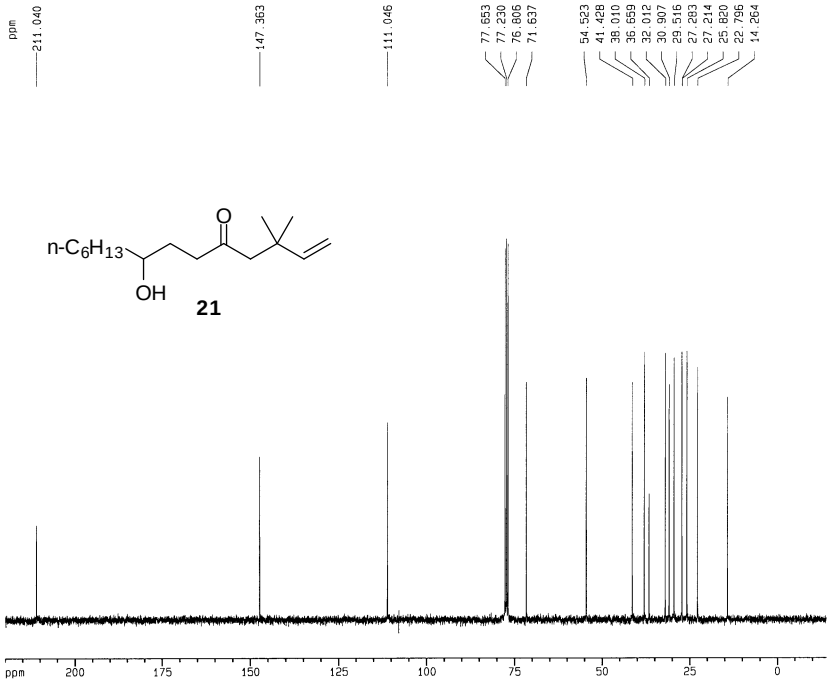
```
Current Data Parameters
NAME          DH-1-295-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_            500000
Time              16.46
INSTRUM           spect
PROBHD     5 mm  QNP Pr
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            5411.255 Hz
FIDRES         0.082569 Hz
AQ           6.0555763 sec
RG                161.3
DW               92.400 usec
DE                 4.50 usec
TE                300.0 K
D1           2.00000000 sec
P1                 9.50 usec
DE                 4.50 usec
SFO1        300.1317887 MHz
NUC1                  1H
PL1                1.00 dB

F2 - Processing parameters
SI                 32768
SF          300.1300063 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               10.300 ppm
F1               3091.34 Hz
F2P               -0.300 ppm
F2                -90.04 Hz
PPMCM           0.48182 ppm/cm
HZCM          144.60809 Hz/cm
```

DH-1-295-1carb 13 1 v yhr 20100410



21

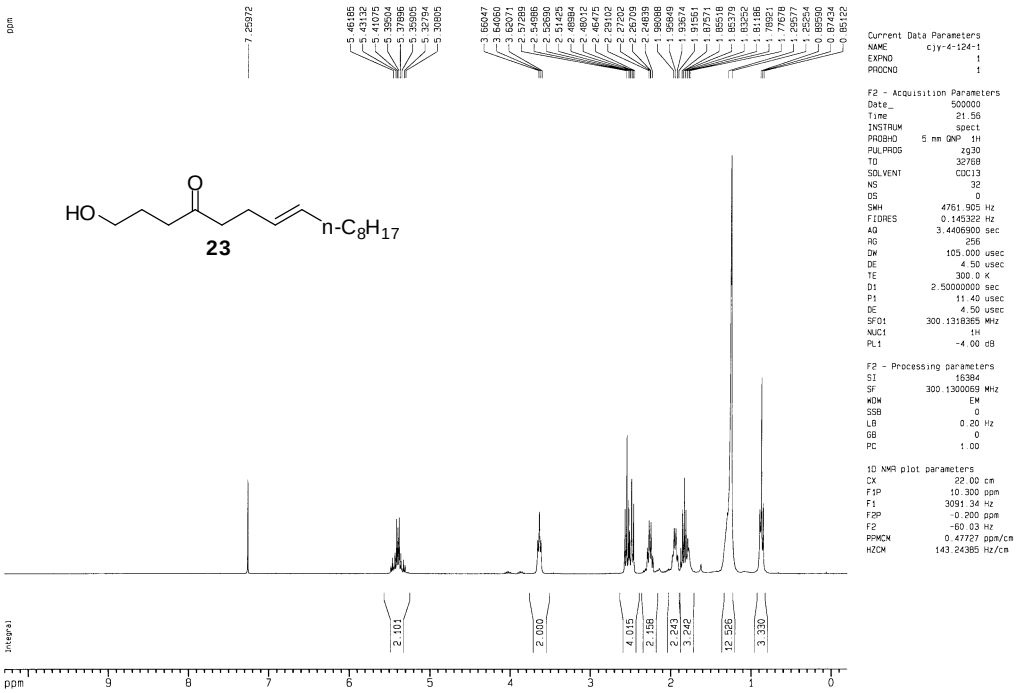
```
Current Data Parameters
NAME     DH-1-295-1carb
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_            500000
Time              13.38
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG         zgpg30
TD                51848
SOLVENT           CDCl3
NS                 1000
DS                    2
SWH           18382.354 Hz
FIDRES         0.354543 Hz
AQ            1.4103156 sec
RG                2580.3
DW               27.200 usec
DE                 4.50 usec
TE                300.0 K
D12           0.00002000 sec
PL13             17.00 dB
D1            1.00000000 sec
CPDPRG2          waltz16
PCPD2            100.00 usec
SFO2         300.1301000 MHz
NUC2                  1H
PL2               -4.00 dB
PL12              15.80 dB
P1                 7.50 usec
DE                 4.50 usec
SFO1          75.4758696 MHz
NUC1                 13C
PL1               -2.00 dB
D11           0.03000000 sec

F2 - Processing parameters
SI                 32768
SF          75.4677333 MHz
WDW                   EM
SSB                    0
LB                  1.20 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               220.000 ppm
F1               16602.90 Hz
F2P               -20.000 ppm
F2               -1509.36 Hz
PPMCM           10.90909 ppm/cm
HZCM           823.28436 Hz/cm
```

cjy-4-124-1 1 1 v yhr 20100401



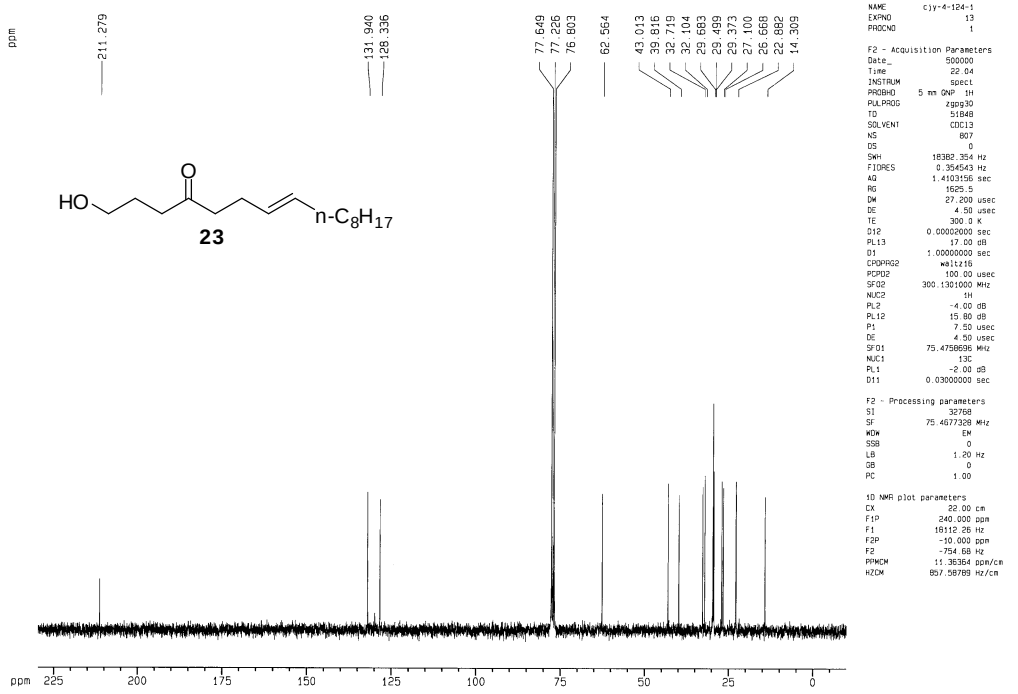
Current Data Parameters
NAME cjy-4-124-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 21.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 256
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm

cjy-4-124-1 13 1 v yhr 20100401



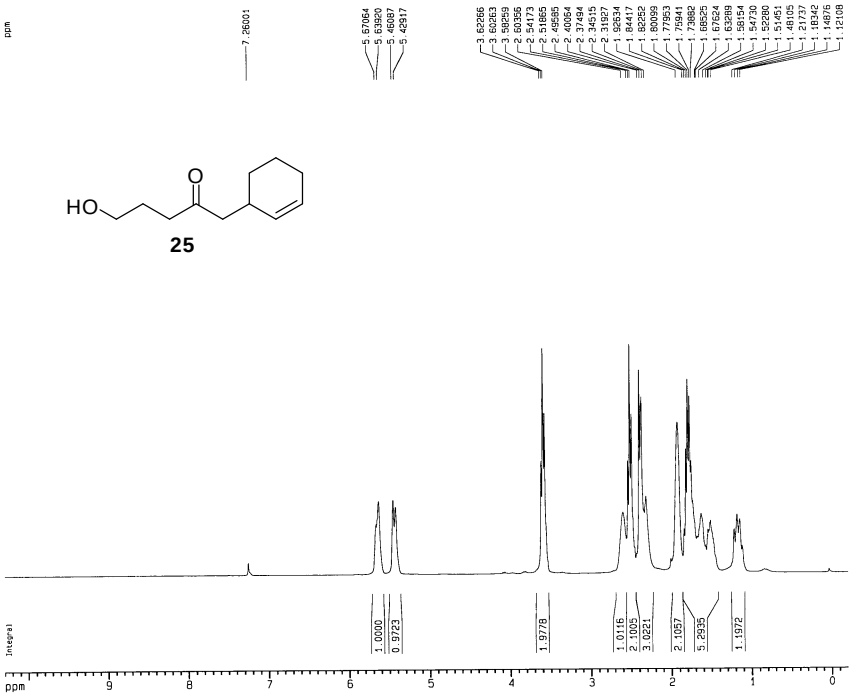
Current Data Parameters
NAME cjy-4-124-1
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 22.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 807
DS 0
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758696 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677328 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 240.000 ppm
F1 18112.26 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.36364 ppm/cm
HZCM 857.58789 Hz/cm

MR-01-163-P 1 1 v yhr 20100513



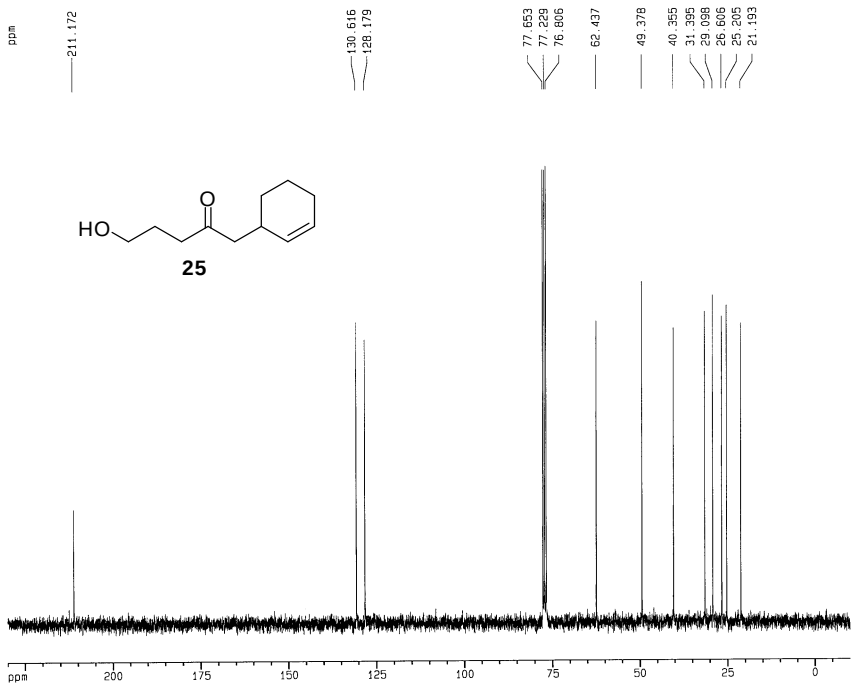

```
Current Data Parameters
NAME          MR-01-163-P
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_             500000
Time               13.45
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG            zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            4761.905 Hz
FIDRES        0.145322 Hz
AQ            3.4406900 sec
RG                    64
DW             105.000 usec
DE                4.50 usec
TE                300.0 K
D1            2.50000000 sec
P1                11.40 usec
DE                4.50 usec
SFO1        300.1318365 MHz
NUC1                  1H
PL1                -4.00 dB

F2 - Processing parameters
SI                 16384
SF          300.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P             10.300 ppm
F1              3091.34 Hz
F2P             -0.200 ppm
F2               -60.03 Hz
PPMCM           0.47727 ppm/cm
HZCM          143.24385 Hz/cm
```

MR-01-161-P 13 1 v yhr 20100510



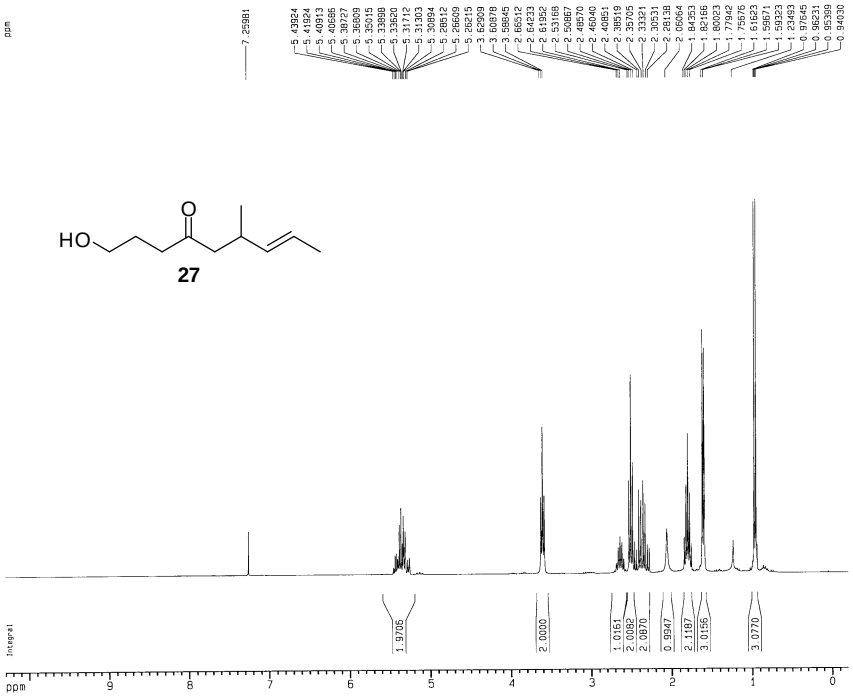

```
Current Data Parameters
NAME          MR-01-161-P
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_             500000
Time               23.29
INSTRUM            spect
PROBHD     5 mm  QNP Pr
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    367
DS                      2
SWH           18382.354 Hz
FIDRES        0.280492 Hz
AQ            1.7826293 sec
RG                  4096
DW             27.200 usec
DE                4.50 usec
TE                300.0 K
d11            0.0300000 sec
d12            0.0000200 sec
PL13              25.00 dB
D1            1.00000000 sec
CPDPRG2          waltz16
PCPD2            100.00 usec
SFO2        300.1312005 MHz
NUC2                  1H
PL2                1.00 dB
PL12              23.00 dB
P1                 7.10 usec
DE                4.50 usec
SFO1         75.4759022 MHz
NUC1                 13C
PL1                2.00 dB

F2 - Processing parameters
SI                 32768
SF          75.4677351 MHz
WDW                   EM
SSB                    0
LB                  1.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P            230.000 ppm
F1             17357.58 Hz
F2P            -10.000 ppm
F2              -754.68 Hz
PPMCM          10.90909 ppm/cm
HZCM          823.28436 Hz/cm
```

cjy-4-111-1 1 1 v yhr 20100318



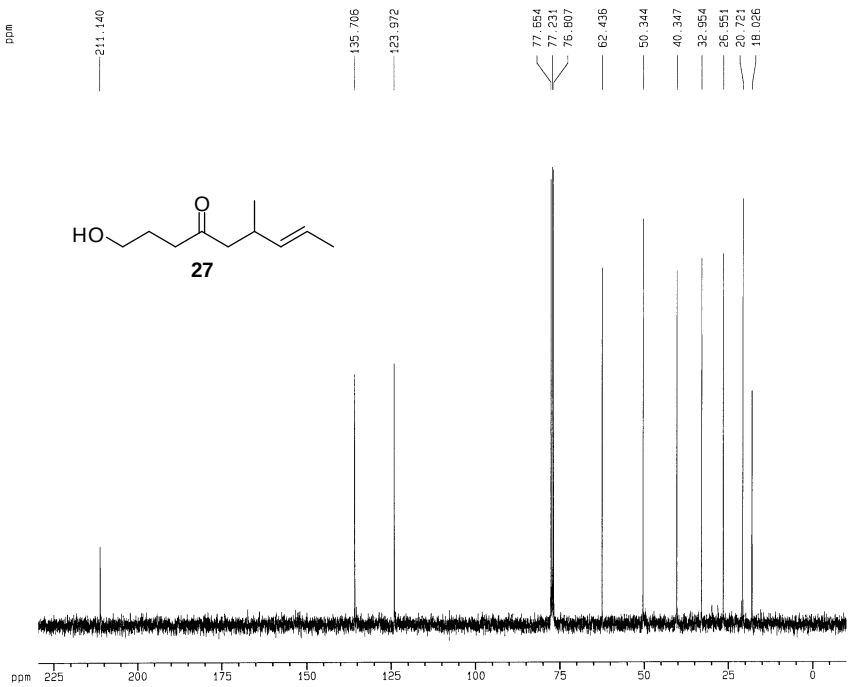
Current Data Parameters
NAME cjy-4-111-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 21.35
INSTRUM spect
PROBHD 5 mm DNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 128
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm

cjy-4-111-1 13 1 v yhr 20100318



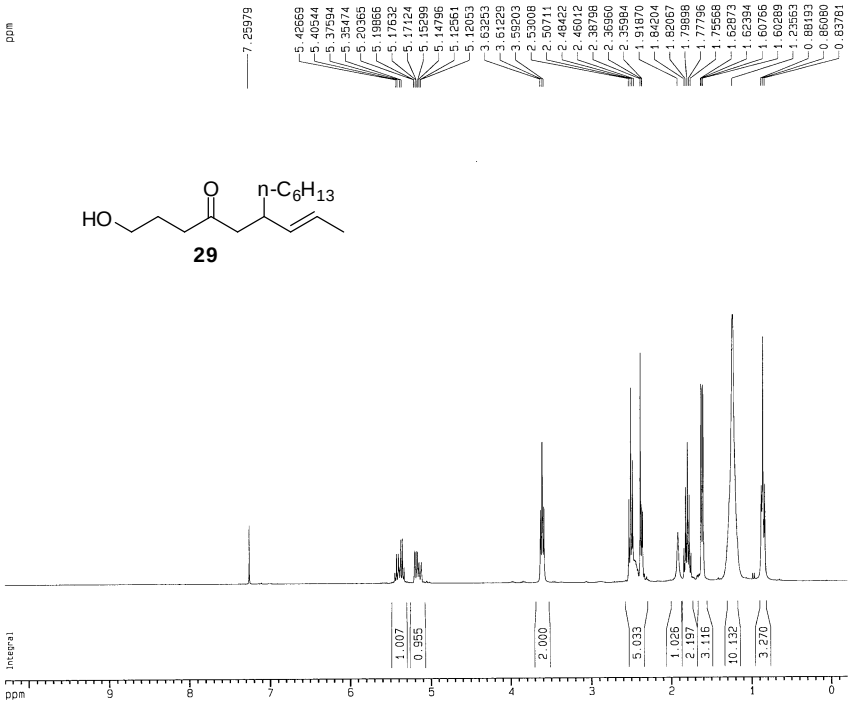
Current Data Parameters
NAME cjy-4-111-1
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 21.46
INSTRUM spect
PROBHD 5 mm DNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 337
DS 0
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103155 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758695 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677339 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 240.000 ppm
F1 18112.26 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.36364 ppm/cm
HZCM 857.58789 Hz/cm

cjy-4-121-1 1 1 v yhr 20100329



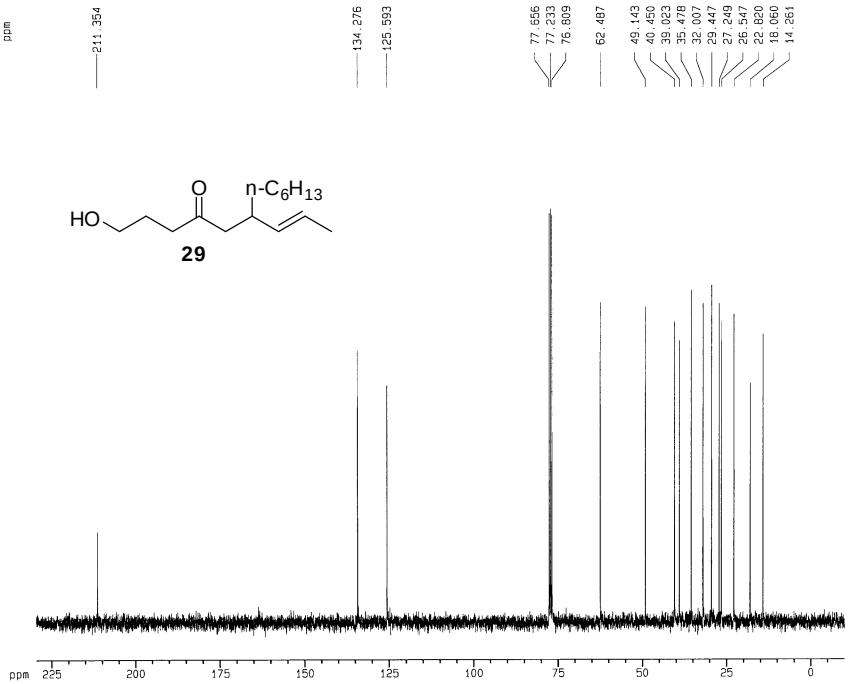

```
Current Data Parameters
NAME          cjy-4-121-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              500000
Time                20.36
INSTRUM             spect
PROBHD     5 mm  GNP Pr
PULPROG             zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      0
SWH             5411.255 Hz
FIDRES          0.082569 Hz
AQ             6.0555763 sec
RG                 161.3
DW                 92.400 usec
DE                  4.50 usec
TE                 300.0 K
D1             3.00000000 sec
P1                  9.50 usec
DE                  4.50 usec
SFO1          300.1317087 MHz
NUC1                   1H
PL1                 1.00 dB

F2 - Processing parameters
SI                  32768
SF            300.1300064 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
F1P                10.300 ppm
F1                 3091.34 Hz
F2P                -0.200 ppm
F2                  -60.03 Hz
PPMCM             0.47727 ppm/cm
HZCM            143.24385 Hz/cm
```

cjy-4-120-1 13 1 v yhr 20100328



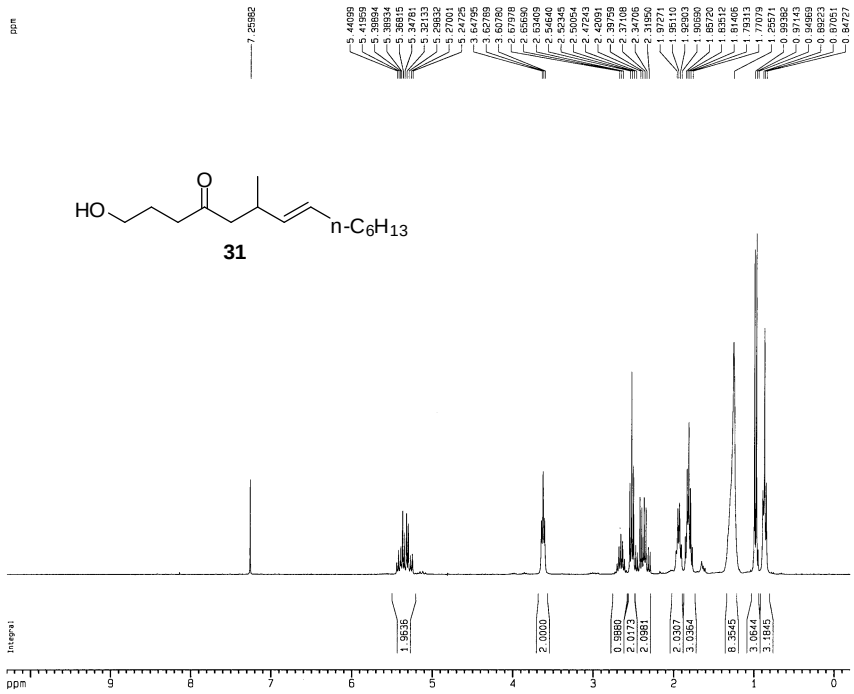

```
Current Data Parameters
NAME          cjy-4-120-1
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_              500000
Time                21.11
INSTRUM             spect
PROBHD     5 mm GNP 1H
PULPROG          zgpg30
TD                  51846
SOLVENT             CDCl3
NS                    304
DS                      0
SWH            18382.354 Hz
FIDRES          0.354543 Hz
AQ             1.4103156 sec
RG                 1625.5
DW                 27.200 usec
DE                  4.50 usec
TE                 300.0 K
D12            0.00002000 sec
PL13                17.00 dB
D1             1.00000000 sec
CPDPRG2            waltz16
PCPD2             100.00 usec
SFO2          300.1301000 MHz
NUC2                   1H
PL2                 -4.00 dB
PL12                15.80 dB
P1                  7.50 usec
DE                  4.50 usec
SFO1          75.4758696 MHz
NUC1                   13C
PL1                 -2.00 dB
D11            0.03000000 sec

F2 - Processing parameters
SI                  32768
SF            75.4677333 MHz
WDW                    EM
SSB                     0
LB                   1.20 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
F1P                240.000 ppm
F1                18112.26 Hz
F2P                -10.000 ppm
F2                  -754.68 Hz
PPMCM             11.36354 ppm/cm
HZCM            857.58789 Hz/cm
```

cjy-MR-1 1 1 v yhr 20100525

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Current Data Parameters
NAME          cjy-MR-1
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           500000
Time              1.46
INSTRUM          spect
PROBHD     5 mm QNP  1H
PULPROG          zg30
TD               32768
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           4761.905 Hz
FIDRES        0.145322 Hz
AQ           3.4406900 sec
RG               228.1
DW             105.000 usec
DE               4.50 usec
TE              300.0 K
D1           2.50000000 sec
P1              11.40 usec
DE               4.50 usec
SFO1        300.1318365 MHz
NUC1                 1H
PL1              -4.00 dB

F2 - Processing parameters
SI                16384
SF          300.1300063 MHz
WDW                 EM
SSB                  0
LB                0.20 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
F1P            10.300 ppm
F1             3091.34 Hz
F2P            -0.200 ppm
F2             -60.03 Hz
PPMCM          0.47727 ppm/cm
HZCM         143.24385 Hz/cm
```
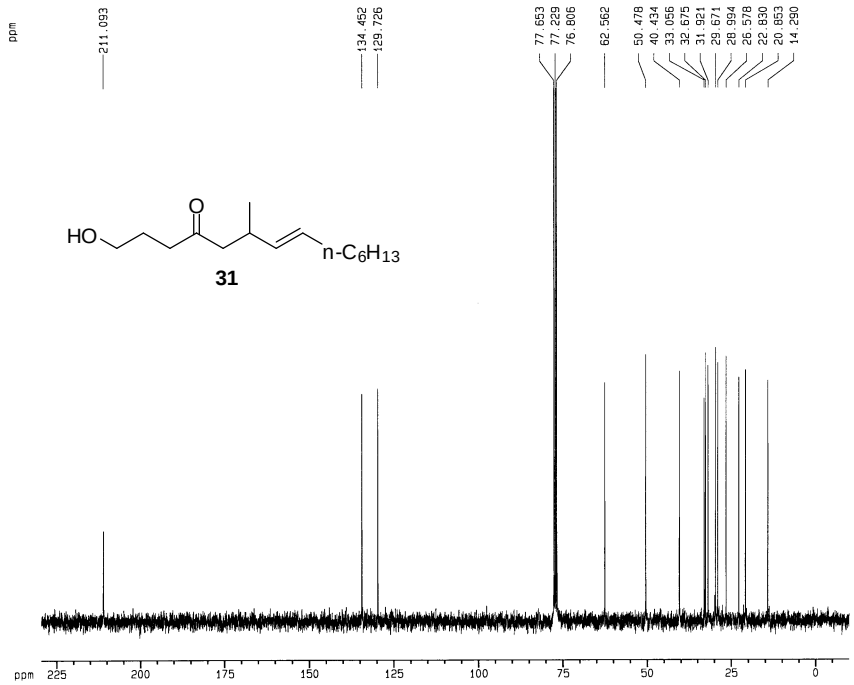

cjy-Mr-1 13 1 v yhr 20100525

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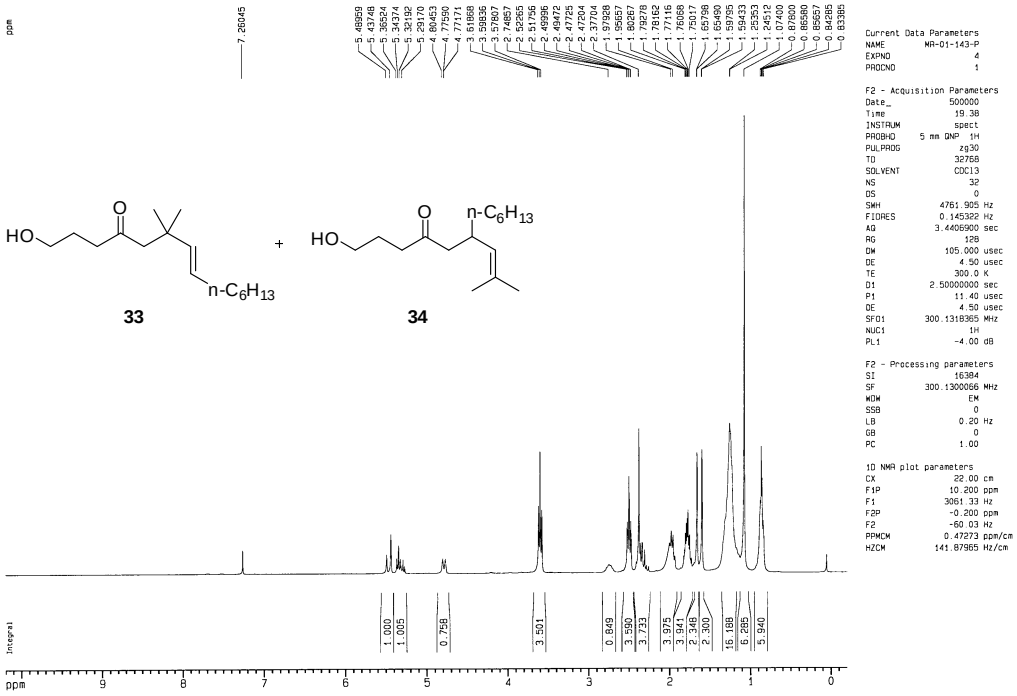
Current Data Parameters
NAME        cjy-4-122-1
EXPNO                13
PROCNO                1

F2 - Acquisition Parameters
Date_           500000
Time              2.05
INSTRUM          spect
PROBHD     5 mm QNP  1H
PULPROG        zgpg30
TD               51848
SOLVENT          CDCl3
NS                 879
DS                   0
SWH          18382.354 Hz
FIDRES        0.354543 Hz
AQ           1.4103156 sec
RG              2580.3
DW              27.200 usec
DE               4.50 usec
TE              300.0 K
D12          0.00002000 sec
PL13            17.00 dB
D1           1.00000000 sec
CPDPRG2        waltz16
PCPD2          100.00 usec
SFO2        300.1301000 MHz
NUC2                 1H
PL2              -4.00 dB
PL12            15.80 dB
P1               7.50 usec
DE               4.50 usec
SFO1         75.4758696 MHz
NUC1                13C
PL1              -2.00 dB
D11          0.03000000 sec

F2 - Processing parameters
SI                32768
SF          75.4677322 MHz
WDW                 EM
SSB                  0
LB                1.20 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX               22.00 cm
F1P            240.000 ppm
F1             18112.26 Hz
F2P            -10.000 ppm
F2             -754.68 Hz
PPMCM          11.36364 ppm/cm
HZCM         857.58789 Hz/cm
```

MR-01-143-P 4 1 v yhr 20100513



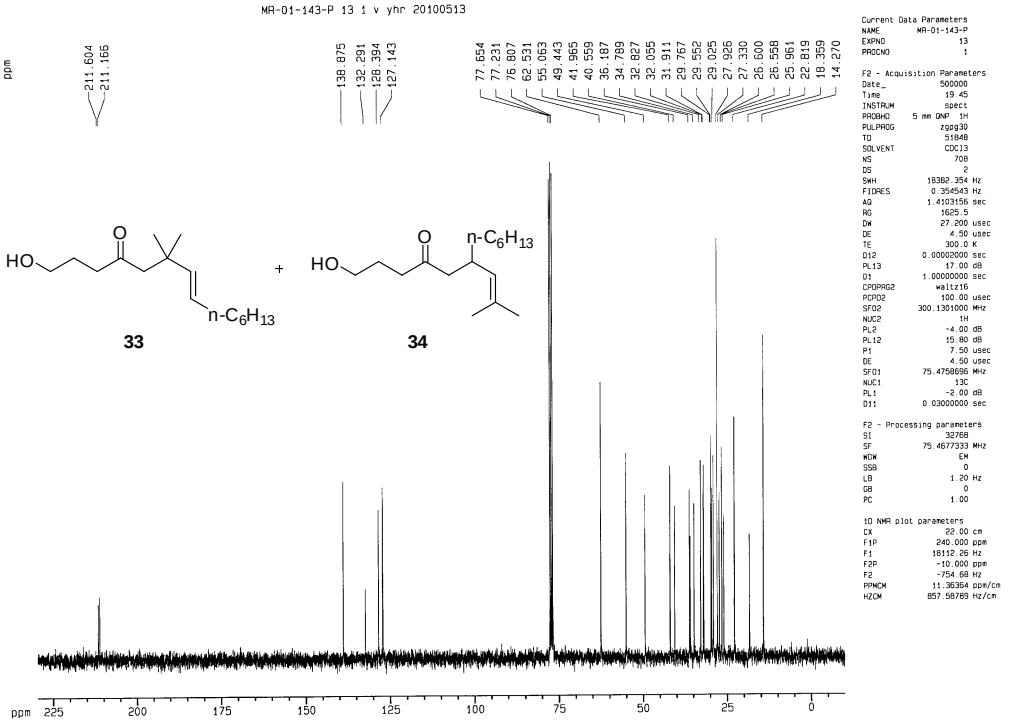
Current Data Parameters
NAME MR-01-143-P
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 19.38
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 128
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.200 ppm
F1 3061.33 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47273 ppm/cm
HZCM 141.87965 Hz/cm

MR-01-143-P 13 1 v yhr 20100513



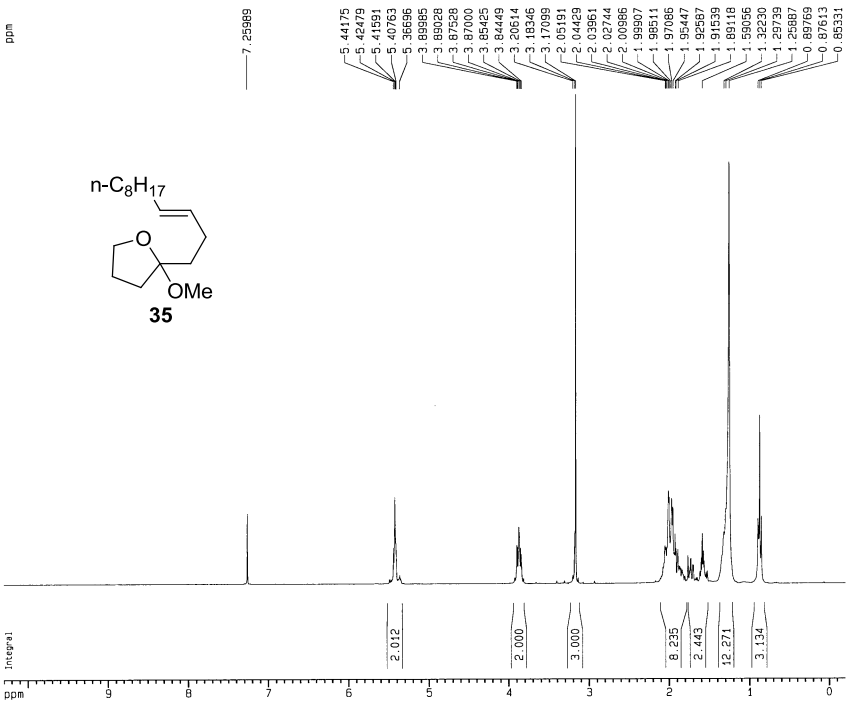
Current Data Parameters
NAME MR-01-143-P
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 19.45
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 708
DS 2
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758698 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677333 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 240.000 ppm
F1 18112.26 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.36354 ppm/cm
HZCM 857.58769 Hz/cm

cjy-4-195-1 1 1 v yhr 20100603



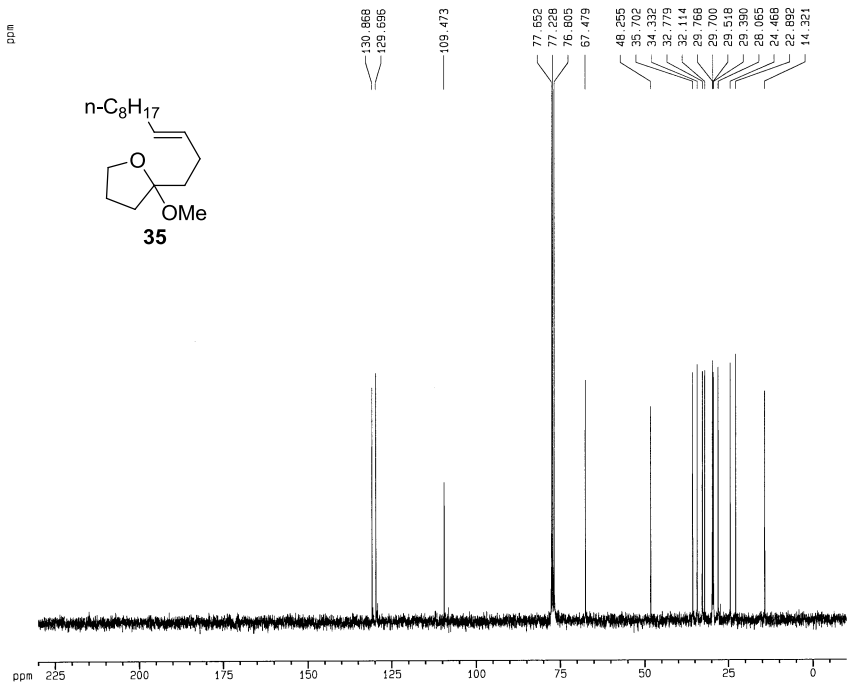

```
Current Data Parameters
NAME          cjy-4-195-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_               500000
Time                  0.08
INSTRUM              spect
PROBHD     5 mm  QNP Pr
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              5411.255 Hz
FIDRES           0.082569 Hz
AQ             6.0555763 sec
RG                     256
DW                92.400 usec
DE                 4.50 usec
TE                 300.0 K
D1             3.00000000 sec
P1                  9.50 usec
DE                  4.50 usec
SFO1          300.1317887 MHz
NUC1                    1H
PL1                  1.00 dB

F2 - Processing parameters
SI                   32768
SF            300.1300063 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
F1P                 10.300 ppm
F1                 3091.34 Hz
F2P                 -0.200 ppm
F2                   -60.03 Hz
PPMCM              0.47727 ppm/cm
HZCM             143.24385 Hz/cm
```

cjy-4-194-1 13 1 v yhr 20100603



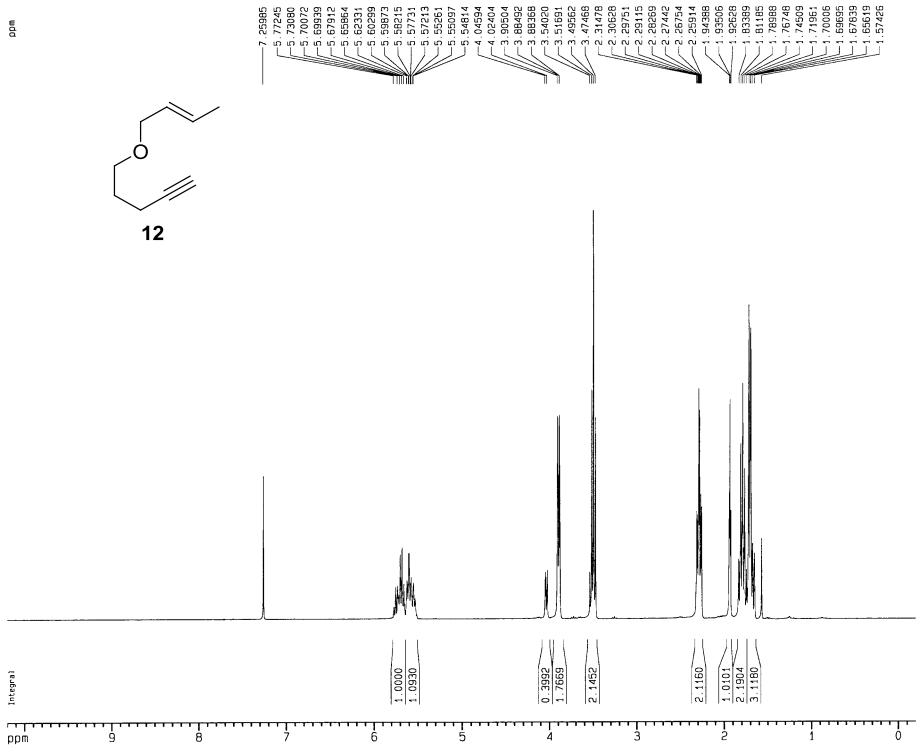

```
Current Data Parameters
NAME          cjy-4-194-1
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_               500000
Time                  1.09
INSTRUM              spect
PROBHD     5 mm  QNP Pr
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     592
DS                       0
SWH             18382.354 Hz
FIDRES           0.280492 Hz
AQ             1.7826293 sec
RG                    2048
DW                27.200 usec
DE                 4.50 usec
TE                 300.0 K
d11             0.0300000 sec
d12             0.0000200 sec
PL13                25.00 dB
D1             1.00000000 sec
CPDPRG2            waltz16
PCPD2             100.00 usec
SFO2          300.1312005 MHz
NUC2                    1H
PL2                  1.00 dB
PL12                23.00 dB
P1                   7.10 usec
DE                  4.50 usec
SFO1           75.4759022 MHz
NUC1                   13C
PL1                  2.00 dB

F2 - Processing parameters
SI                   32768
SF             75.4677328 MHz
WDW                     EM
SSB                      0
LB                    1.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.00 cm
F1P                 240.000 ppm
F1                18112.26 Hz
F2P                -10.000 ppm
F2                  -754.68 Hz
PPMCM             11.36364 ppm/cm
HZCM            857.58789 Hz/cm
```

MR-01-113-D 1 1 v yhr 20100516



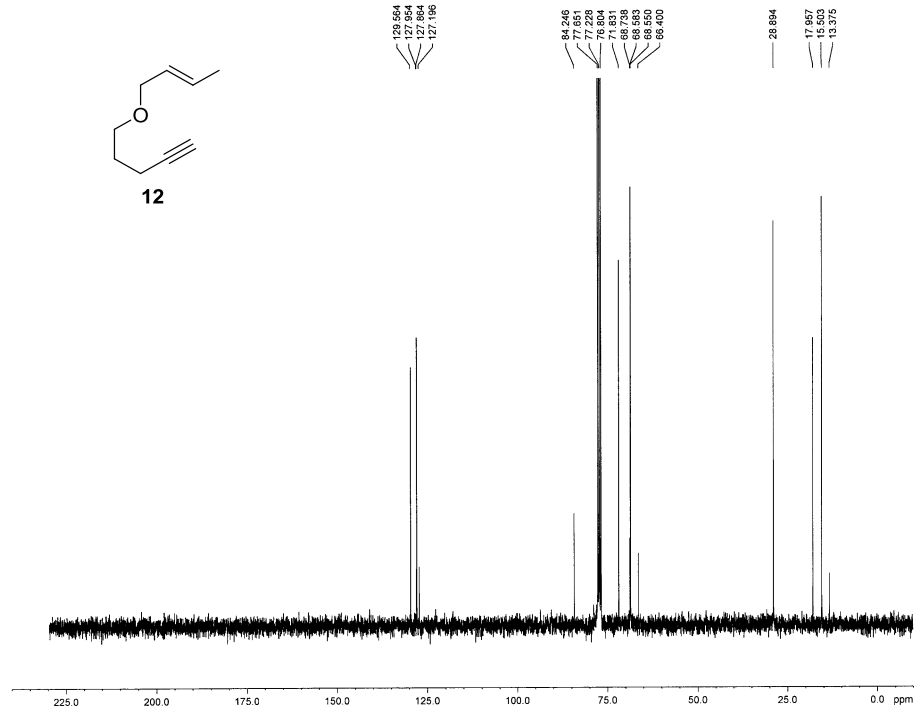
Current Data Parameters
NAME MR-01-113-D
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 16.51
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 362
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

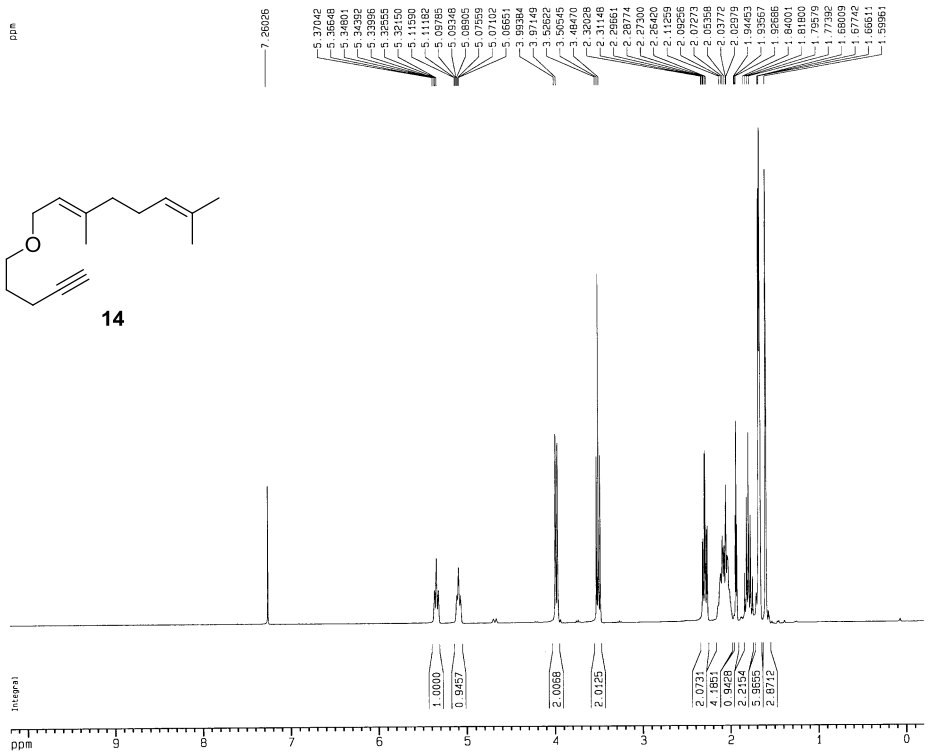
1D NMR plot parameters
CX 22.00 cm
F1P 10.200 ppm
F1 3061.33 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47273 ppm/cm
HZCM 141.87965 Hz/cm

MR-01-113-D 13 1 v yhr 20100516



NAME MR-01-113-D
EXPNO 13
PROCNO 1
Date_ 500000
Time 17.12
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 1661
DS 2
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758696 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec
SI 32768
SF 75.4677322 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

MR-01-133-D 2 1 v yhr 20100418



14

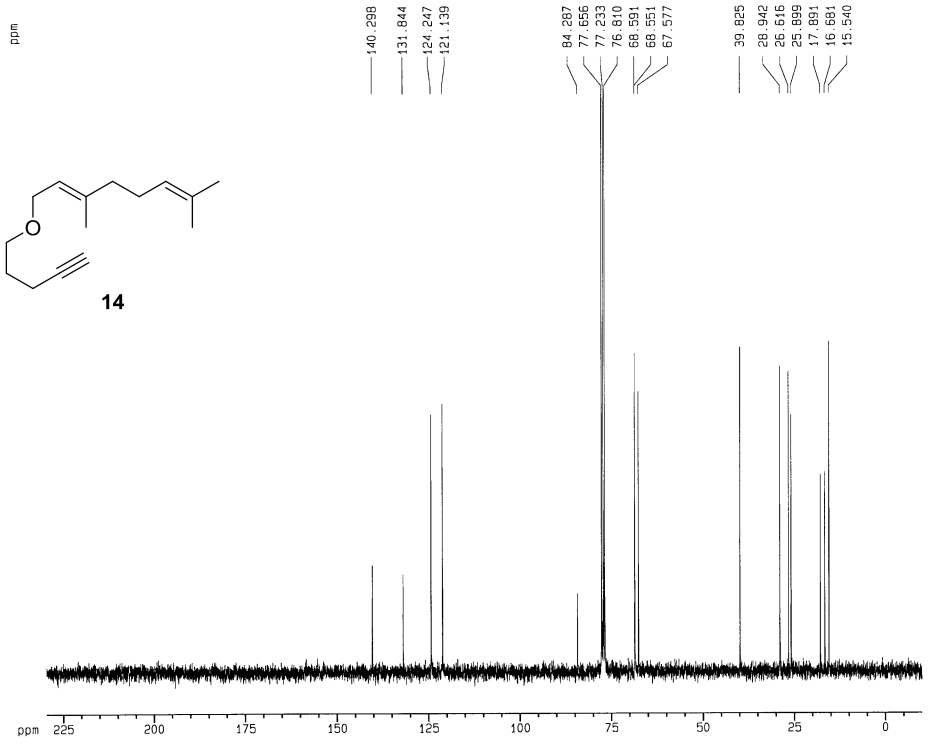
```
Current Data Parameters
NAME          MR-01-133-D
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              500000
Time                22.23
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG             zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH             4761.905 Hz
FIDRES          0.145322 Hz
AQ             3.4406900 sec
RG                    256
DW                105.000 usec
DE                   4.50 usec
TE                  300.0 K
D1             2.50000000 sec
P1                  11.40 usec
DE                   4.50 usec
SFO1          300.1318365 MHz
NUC1                   1H
PL1                 -4.00 dB

F2 - Processing parameters
SI                  16384
SF            300.1300060 MHz
WDW                    EM
SSB                     0
LB                   0.20 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
F1P                10.200 ppm
F1               3061.33 Hz
F2P                -0.200 ppm
F2                 -60.03 Hz
PPMCM            0.47273 ppm/cm
HZCM           141.87965 Hz/cm
```

MR-01-133-D 13 1 v yhr 20100418



14

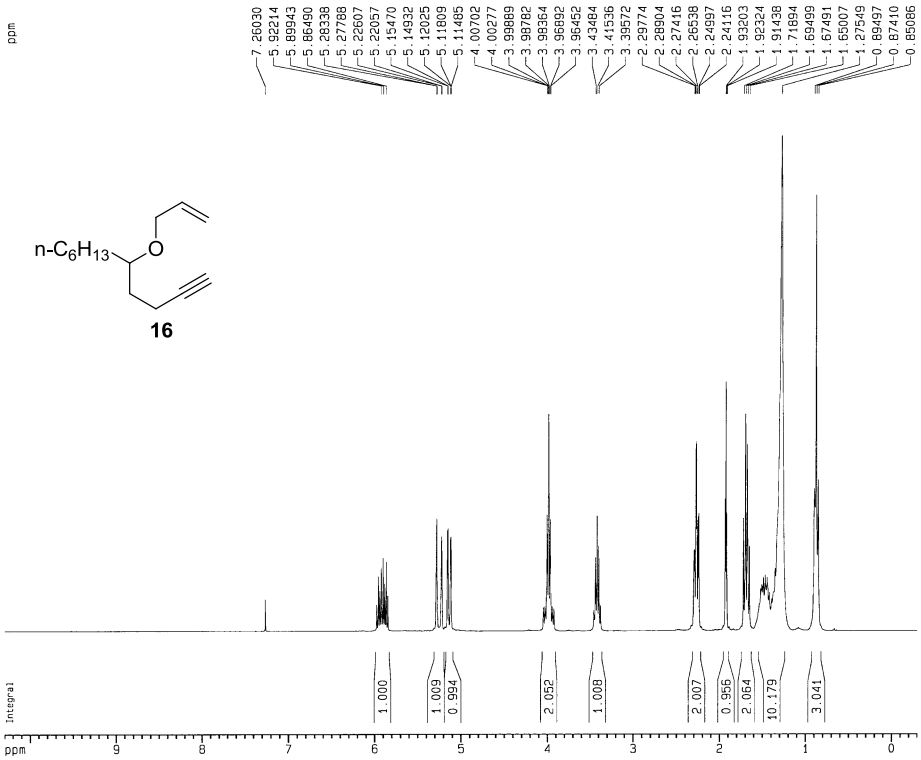
```
Current Data Parameters
NAME          MR-01-133-D
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_              500000
Time                23.21
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG            zgpg30
TD                  51848
SOLVENT             CDCl3
NS                    1235
DS                       2
SWH            18382.354 Hz
FIDRES          0.354543 Hz
AQ             1.4103156 sec
RG                  1625.5
DW                27.200 usec
DE                   4.50 usec
TE                  300.0 K
D12            0.00002000 sec
PL13                17.00 dB
D1             1.00000000 sec
CPDPRG2            waltz16
PCPD2             100.00 usec
SFO2          300.1301000 MHz
NUC2                   1H
PL2                 -4.00 dB
PL12                15.80 dB
P1                   7.50 usec
DE                   4.50 usec
SFO1           75.4758695 MHz
NUC1                  13C
PL1                 -2.00 dB
D11            0.03000000 sec

F2 - Processing parameters
SI                  32768
SF             75.4677316 MHz
WDW                    EM
SSB                     0
LB                   1.20 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.00 cm
F1P                240.000 ppm
F1               18112.26 Hz
F2P                -10.000 ppm
F2                 -754.68 Hz
PPMCM            11.36364 ppm/cm
HZCM           857.58789 Hz/cm
```

DH-1-207-1rc 1 1 v yhr 20100419



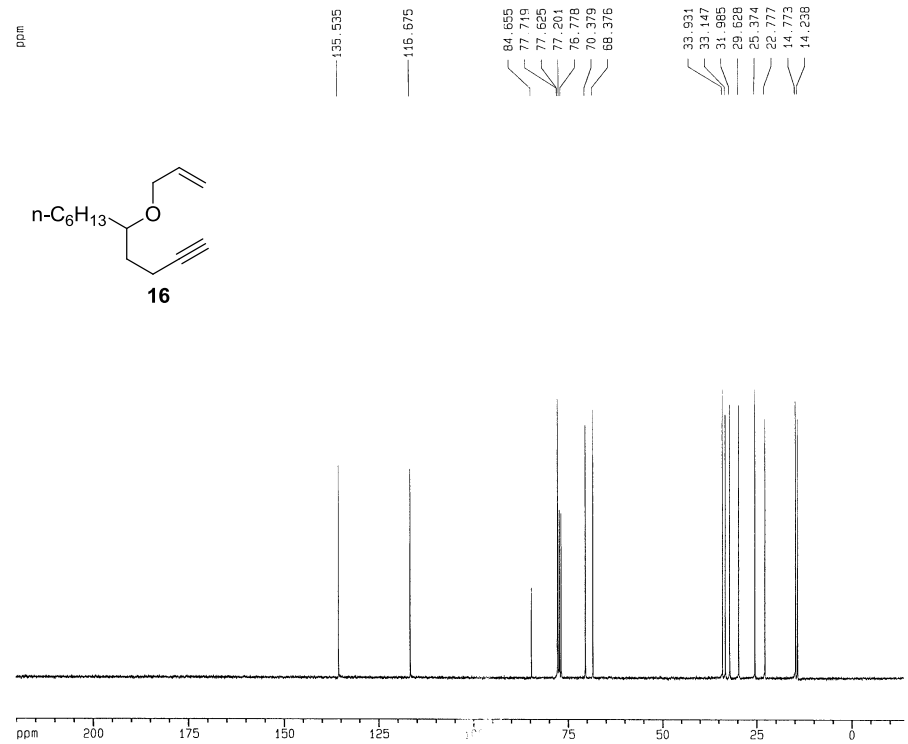

```
Current Data Parameters
NAME        DH-1-207-1rc
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_             500000
Time               23.47
INSTRUM            spect
PROBHD     5 mm  QNP Pr
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            5411.255 Hz
FIDRES         0.082569 Hz
AQ            6.0555763 sec
RG                  71.8
DW                92.400 usec
DE                 4.50 usec
TE                300.0 K
D1           2.00000000 sec
P1                  9.50 usec
DE                  4.50 usec
SFO1        300.1317887 MHz
NUC1                  1H
PL1                1.00 dB

F2 - Processing parameters
SI                 32768
SF          300.1300063 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               10.300 ppm
F1               3091.34 Hz
F2P               -0.300 ppm
F2                -90.04 Hz
PPMCM           0.48182 ppm/cm
HZCM          144.60809 Hz/cm
```

DH-1-207-1rcCa 13 1 v yhr 20100419



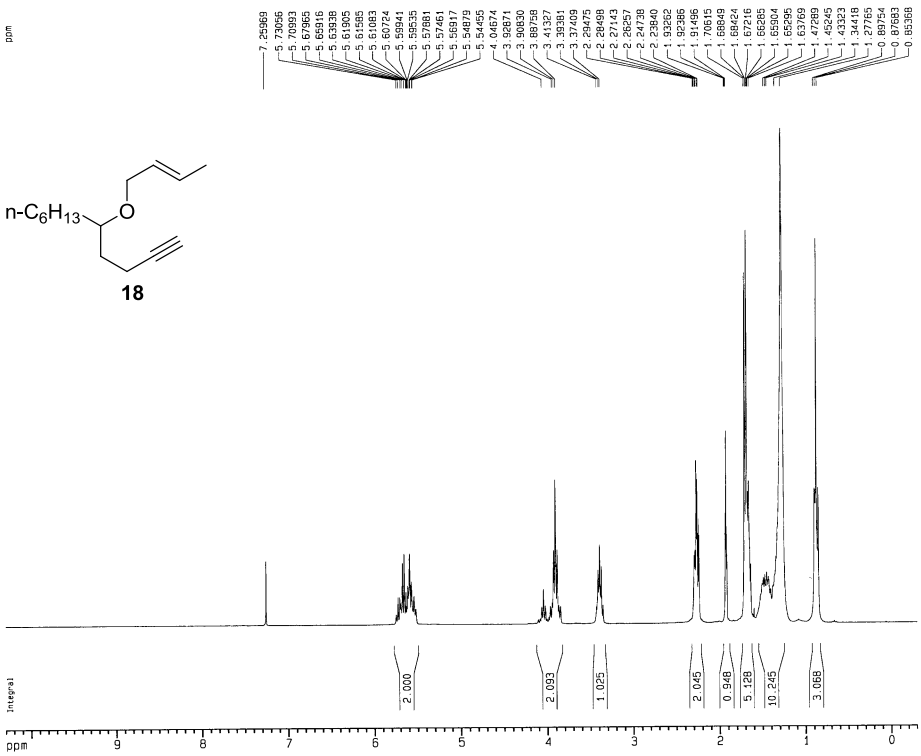

```
Current Data Parameters
NAME      DH-1-207-1rcCa
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_             500000
Time               0.41
INSTRUM            spect
PROBHD     5 mm  QNP Pr
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     2
SWH           18382.354 Hz
FIDRES         0.280492 Hz
AQ            1.7826293 sec
RG                  1024
DW                27.200 usec
DE                 4.50 usec
TE                300.0 K
d11            0.0300000 sec
d12            0.0000200 sec
PL13              25.00 dB
D1            1.00000000 sec
CPDPRG2          waltz16
PCPD2            100.00 usec
SFO2        300.1312005 MHz
NUC2                  1H
PL2                1.00 dB
PL12              23.00 dB
P1                 7.10 usec
DE                 4.50 usec
SFO1         75.4759022 MHz
NUC1                 13C
PL1                2.00 dB

F2 - Processing parameters
SI                 32768
SF          75.4677362 MHz
WDW                   EM
SSB                    0
LB                  1.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               220.000 ppm
F1              16602.90 Hz
F2P               -20.000 ppm
F2              -1509.36 Hz
PPMCM           10.90909 ppm/cm
HZCM           823.28436 Hz/cm
```

DH-1-209-1rc 1 1 v yhr 20100218



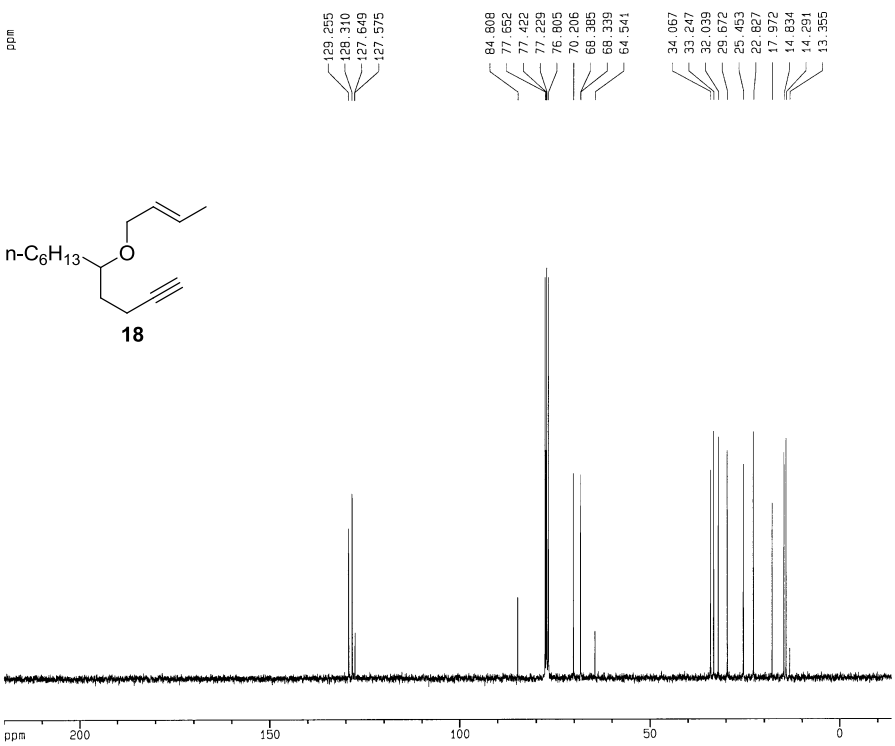
Current Data Parameters
NAME DH-1-209-1rc
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 22.03
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 64
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.48182 ppm/cm
HZCM 144.60809 Hz/cm

DH-1-209-1rcCa 13 1 v yhr 20100421



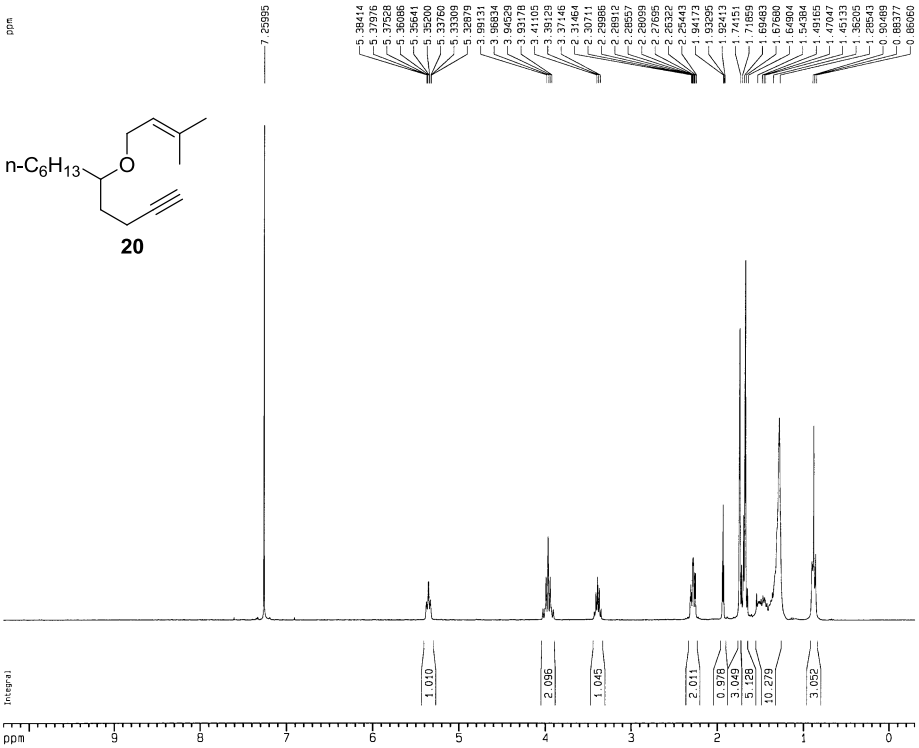
Current Data Parameters
NAME DH-1-209-1rcCa
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 23.49
INSTRUM spect
PROBHD 5 mm QNP Pr
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 807
DS 2
SWH 18382.354 Hz
FIDRES 0.280492 Hz
AQ 1.7826293 sec
RG 2048
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 25.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 1.00 dB
PL12 23.00 dB
P1 7.10 usec
DE 4.50 usec
SFO1 75.4759022 MHz
NUC1 13C
PL1 2.00 dB

F2 - Processing parameters
SI 32768
SF 75.4677334 MHz
WDW EM
SSB 0
LB 1.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 220.000 ppm
F1 16602.90 Hz
F2P -20.000 ppm
F2 -1509.36 Hz
PPMCM 10.90909 ppm/cm
HZCM 823.28436 Hz/cm

DH-1-129-1rc 1 1 v yhr 20091212



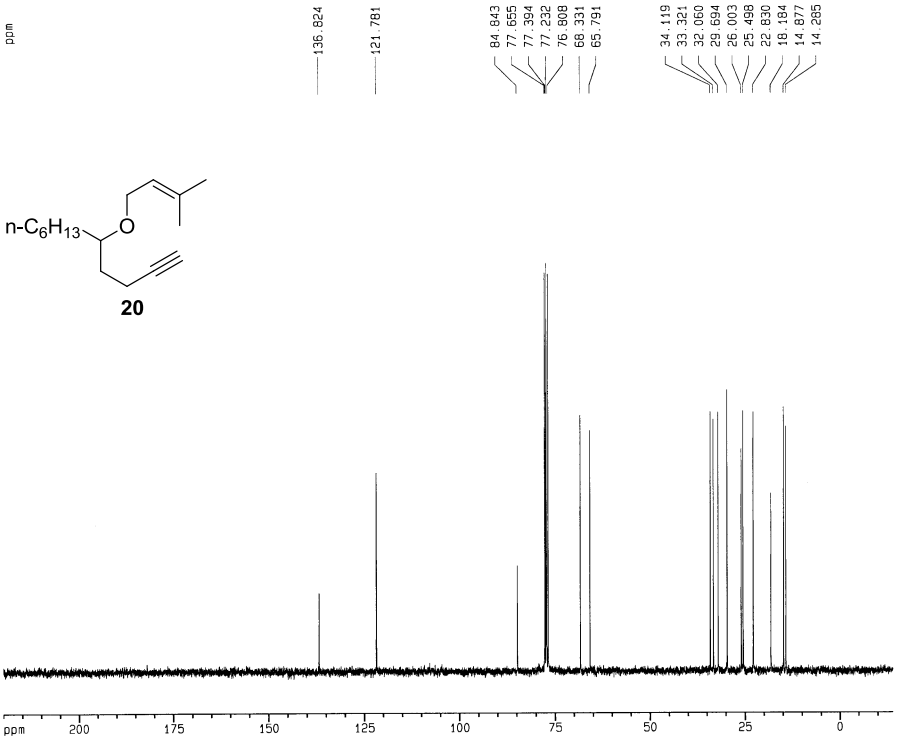

```
Current Data Parameters
NAME        DH-1-129-1rc
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_            500000
Time              21.23
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            4761.905 Hz
FIDRES         0.145322 Hz
AQ             3.4406900 sec
RG                 456.1
DW               105.000 usec
DE                 4.50 usec
TE                300.0 K
D1             2.00000000 sec
P1                11.40 usec
DE                 4.50 usec
SFO1         300.1318365 MHz
NUC1                  1H
PL1                -4.00 dB

F2 - Processing parameters
SI                 16384
SF           300.1300060 MHz
WDW                   EM
SSB                    0
LB                  0.20 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               10.300 ppm
F1               3091.34 Hz
F2P               -0.300 ppm
F2                -90.04 Hz
PPMCM            0.48182 ppm/cm
HZCM           144.60609 Hz/cm
```

DH-1-129-1rc 13 1 v yhr 20100421

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Current Data Parameters
NAME        DH-1-129-1rc
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_            500000
Time              22.49
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                51848
SOLVENT           CDCl3
NS                 1000
DS                    2
SWH           18382.354 Hz
FIDRES         0.354543 Hz
AQ             1.4103156 sec
RG                1625.5
DW               27.200 usec
DE                 4.50 usec
TE                300.0 K
D12            0.00002000 sec
PL13              17.00 dB
D1             1.00000000 sec
CPDPRG2          waltz16
PCPD2            100.00 usec
SFO2         300.1301000 MHz
NUC2                  1H
PL2                -4.00 dB
PL12               15.80 dB
P1                 7.50 usec
DE                 4.50 usec
SFO1          75.4758696 MHz
NUC1                 13C
PL1                -2.00 dB
D11            0.03000000 sec

F2 - Processing parameters
SI                 32768
SF            75.4677322 MHz
WDW                   EM
SSB                    0
LB                  1.20 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.00 cm
F1P               220.000 ppm
F1              16602.90 Hz
F2P               -20.000 ppm
F2              -1509.36 Hz
PPMCM            10.90909 ppm/cm
HZCM           823.28436 Hz/cm
```
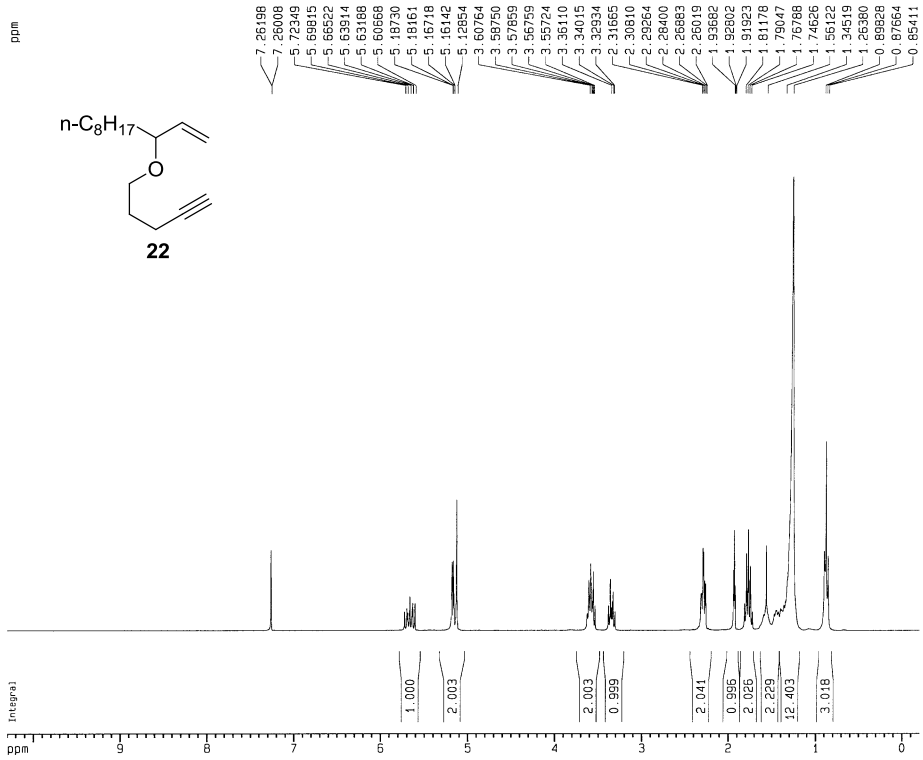

cjy-4-189-1 1 1 v yhr 20100531



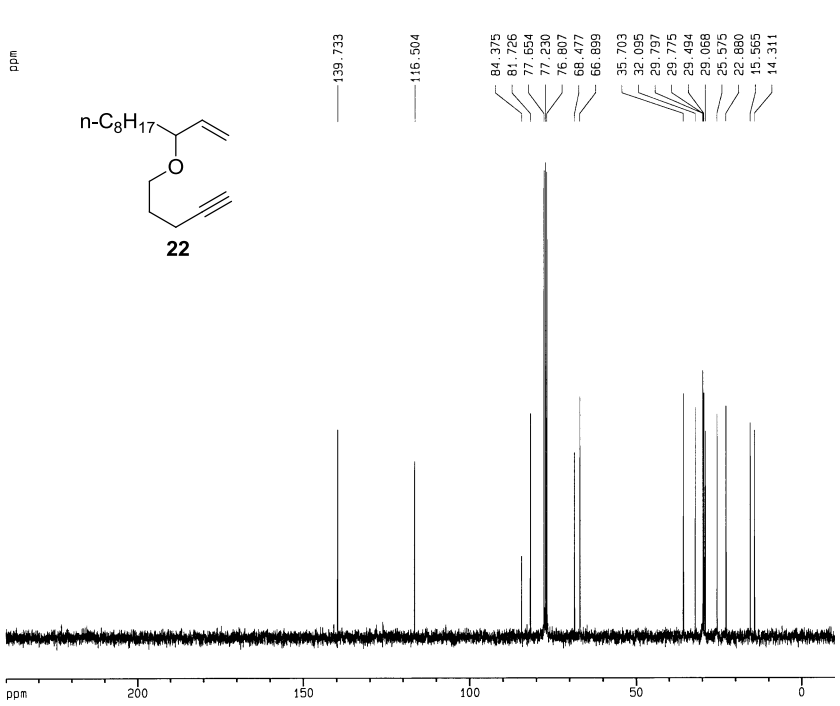
Current Data Parameters
NAME cjy-4-189-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 17.57
INSTRUM spect
PROBHD 5 mm QNP Pr
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 5411.255 Hz
FIDRES 0.082569 Hz
AQ 6.0555763 sec
RG 322.5
DW 92.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
P1 9.50 usec
DE 4.50 usec
SFO1 300.1317887 MHz
NUC1 1H
PL1 1.00 dB

F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm

WT-1-45-NMR 13 1 v yhr 20100418



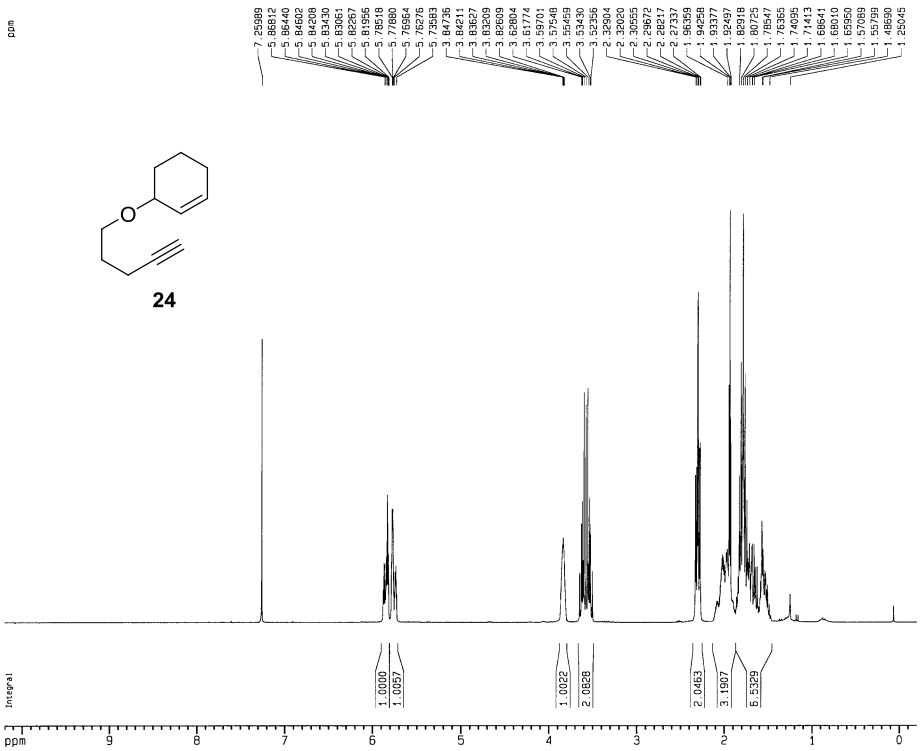
Current Data Parameters
NAME WT-1-45-NMR
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 20.44
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 501
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 4096
DW 21.000 usec
DE 6.00 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 6.00 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677320 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 240.000 ppm
F1 18112.26 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 12.50000 ppm/cm
HZCM 943.34668 Hz/cm

MR-01-159-P 3 1 v yhr 20100509



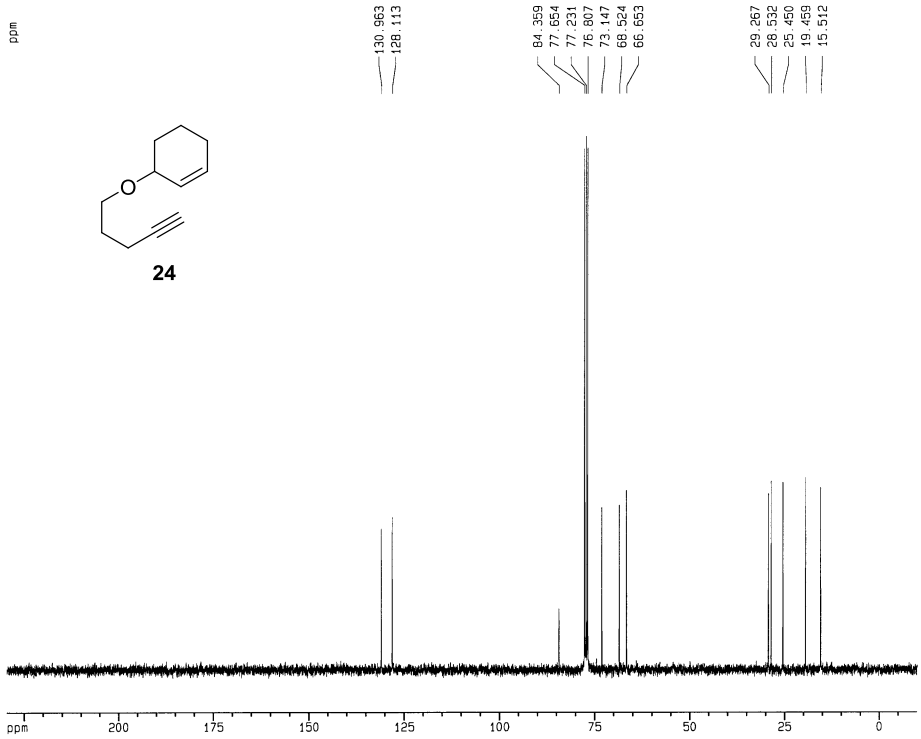
Current Data Parameters
NAME MR-01-159-P
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 20.53
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 362
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.200 ppm
F1 3061.33 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47273 ppm/cm
HZCM 141.87965 Hz/cm

MR-01-159-P 13 1 v yhr 20100509



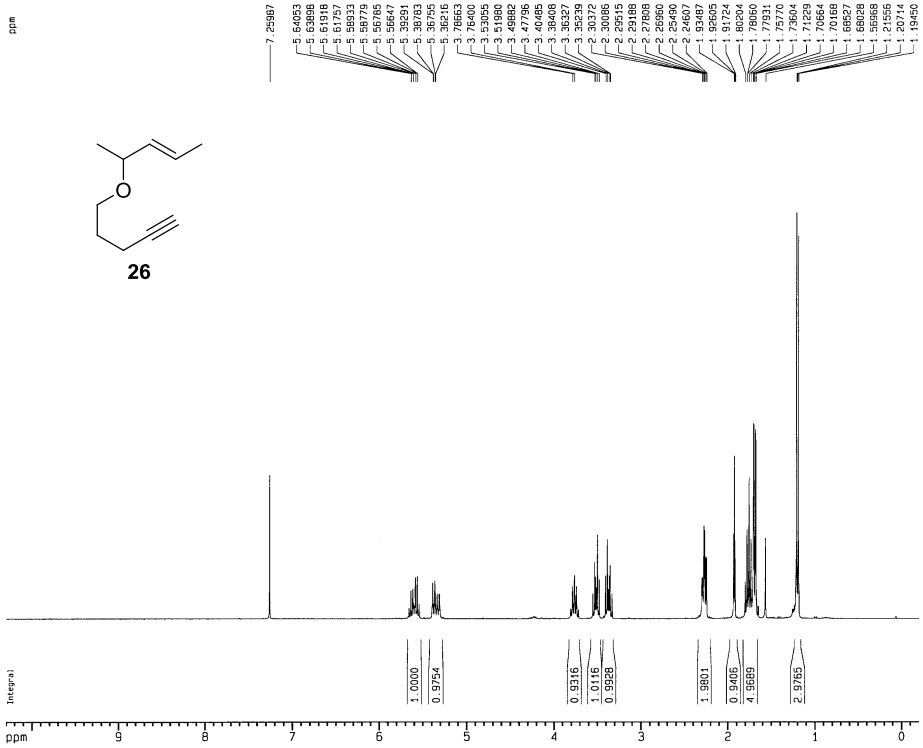
Current Data Parameters
NAME MR-01-159-P
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 21.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 1092
DS 2
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758696 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677322 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 230.000 ppm
F1 17357.58 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 10.90909 ppm/cm
HZCM 823.28436 Hz/cm

MR-01-149-P2 1 1 v yhr 20100524



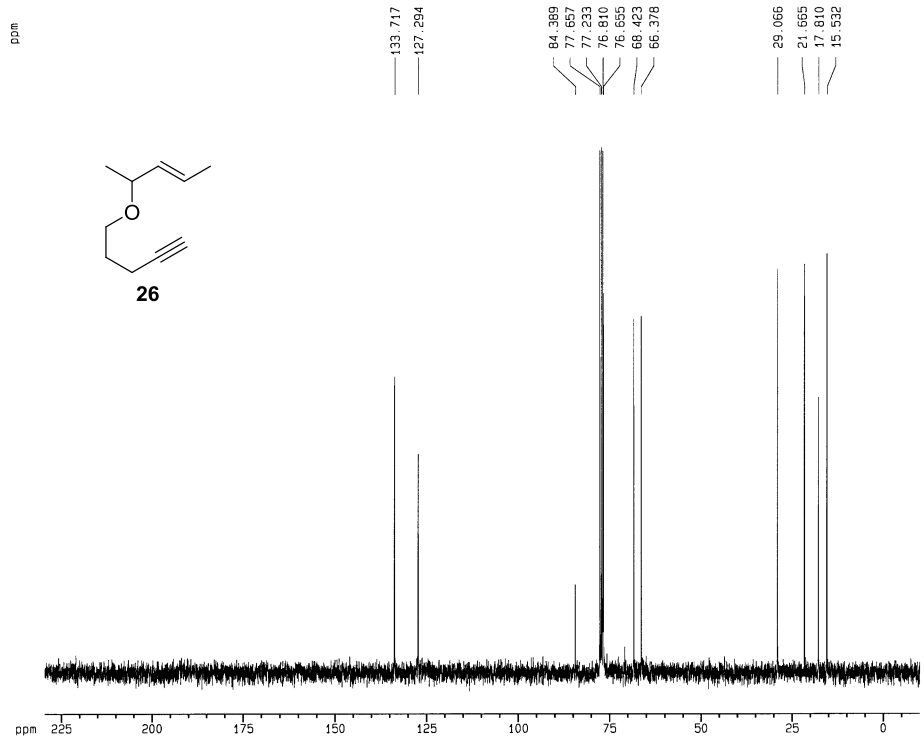
Current Data Parameters
NAME MR-01-149-P2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 0.28
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 362
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm

MR-01-149-P2 13 1 v yhr 20100524



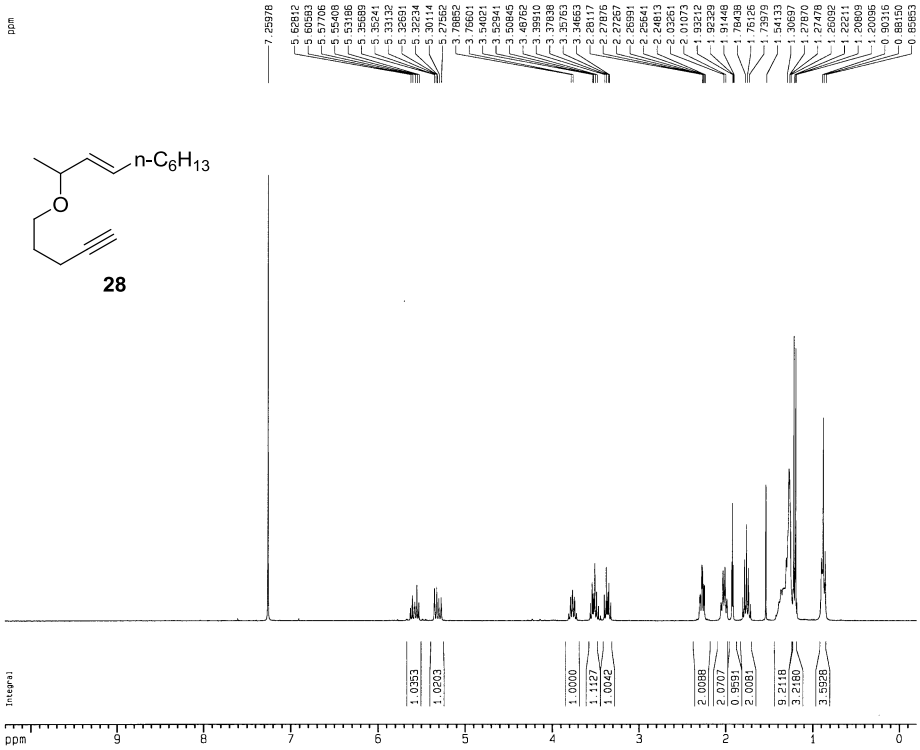
Current Data Parameters
NAME MR-01-149-P2
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 0.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 51848
SOLVENT CDCl3
NS 367
DS 2
SWH 18382.354 Hz
FIDRES 0.354543 Hz
AQ 1.4103156 sec
RG 1625.5
DW 27.200 usec
DE 4.50 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1301000 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 4.50 usec
SFO1 75.4758696 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 75.4677322 MHz
WDW EM
SSB 0
LB 1.20 Hz
GB 0
PC 1.00

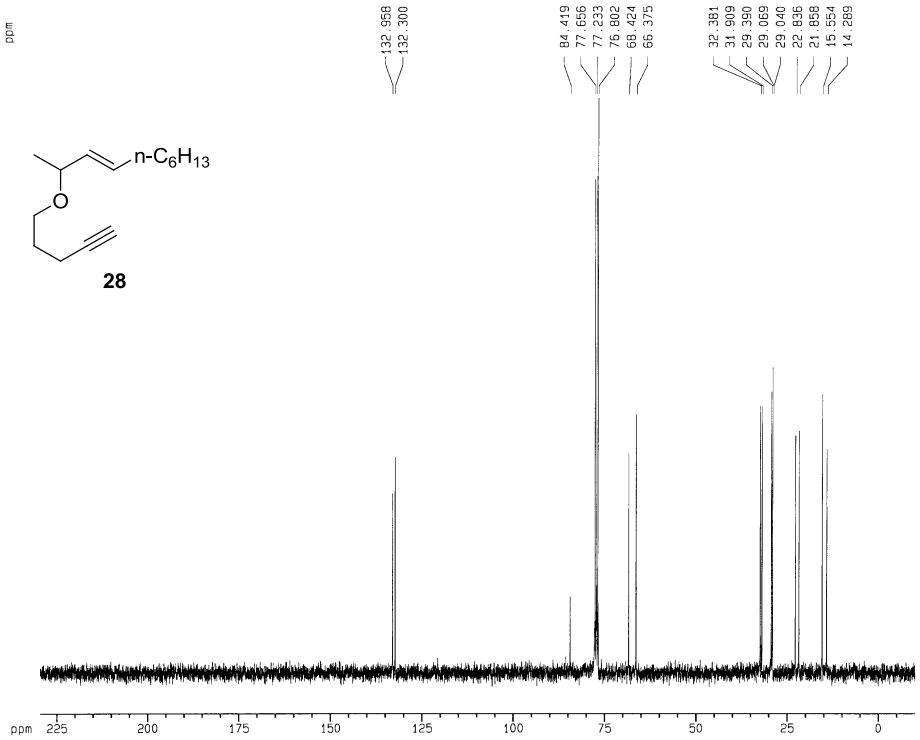
1D NMR plot parameters
CX 22.00 cm
F1P 240.000 ppm
F1 18112.26 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 11.36364 ppm/cm
HZCM 857.58789 Hz/cm

cjy-4-129-1 1 1 v yhr 20100406



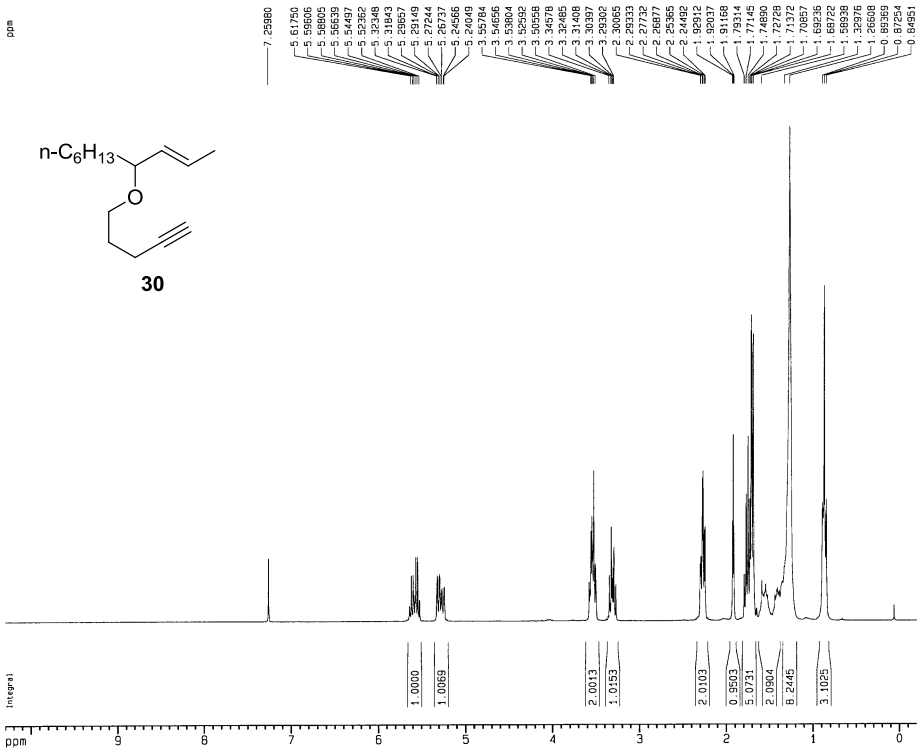

```
Current Data Parameters
NAME          cjy-4-129-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            500000
Time              22.26
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            4761.905 Hz
FIDRES         0.145322 Hz
AQ             3.4406900 sec
RG                 1024
DW              105.000 usec
DE                4.50 usec
TE                300.0 K
D1             2.50000000 sec
P1                11.40 usec
DE                4.50 usec
SFO1         300.1318365 MHz
NUC1                 1H
PL1               -4.00 dB

F2 - Processing parameters
SI                16384
SF           300.1300072 MHz
WDW                  EM
SSB                   0
LB                 0.20 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
F1P               10.300 ppm
F1               3091.34 Hz
F2P               -0.200 ppm
F2                -60.03 Hz
PPMCM           0.47727 ppm/cm
HZCM          143.24385 Hz/cm
```

cjy-4-119-1 13 1 v yhr 20100326




```
Current Data Parameters
NAME          cjy-4-119-1
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_            500000
Time              17.27
INSTRUM           spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                 51848
SOLVENT           CDCl3
NS                   530
DS                    0
SWH           18382.354 Hz
FIDRES         0.354543 Hz
AQ             1.4103156 sec
RG                 4096
DW              27.200 usec
DE                4.50 usec
TE                300.0 K
D12            0.00002000 sec
PL13              17.00 dB
D1             1.00000000 sec
CPDPRG2          waltz16
PCPD2           100.00 usec
SFO2         300.1301000 MHz
NUC2                 1H
PL2               -4.00 dB
PL12              15.80 dB
P1                 7.50 usec
DE                4.50 usec
SFO1          75.4758696 MHz
NUC1                 13C
PL1               -2.00 dB
D11            0.03000000 sec

F2 - Processing parameters
SI                32768
SF           75.4677322 MHz
WDW                  EM
SSB                   0
LB                 1.20 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.00 cm
F1P              240.000 ppm
F1             18112.26 Hz
F2P              -10.000 ppm
F2               -754.68 Hz
PPMCM           11.36364 ppm/cm
HZCM          857.58789 Hz/cm
```

WT-1-55-p 2 1 v yhr 20100502



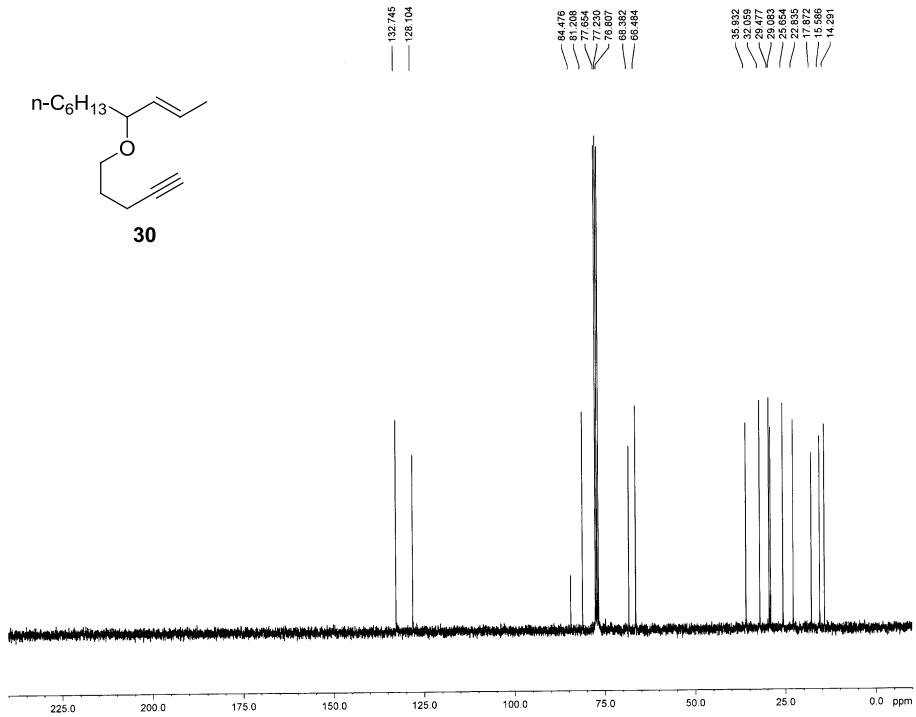
Current Data Parameters
NAME WT-1-55-p
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 500000
Time 23.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4761.905 Hz
FIDRES 0.145322 Hz
AQ 3.4406900 sec
RG 181
DW 105.000 usec
DE 4.50 usec
TE 300.0 K
D1 2.50000000 sec
P1 11.40 usec
DE 4.50 usec
SFO1 300.1318365 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 300.1300063 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

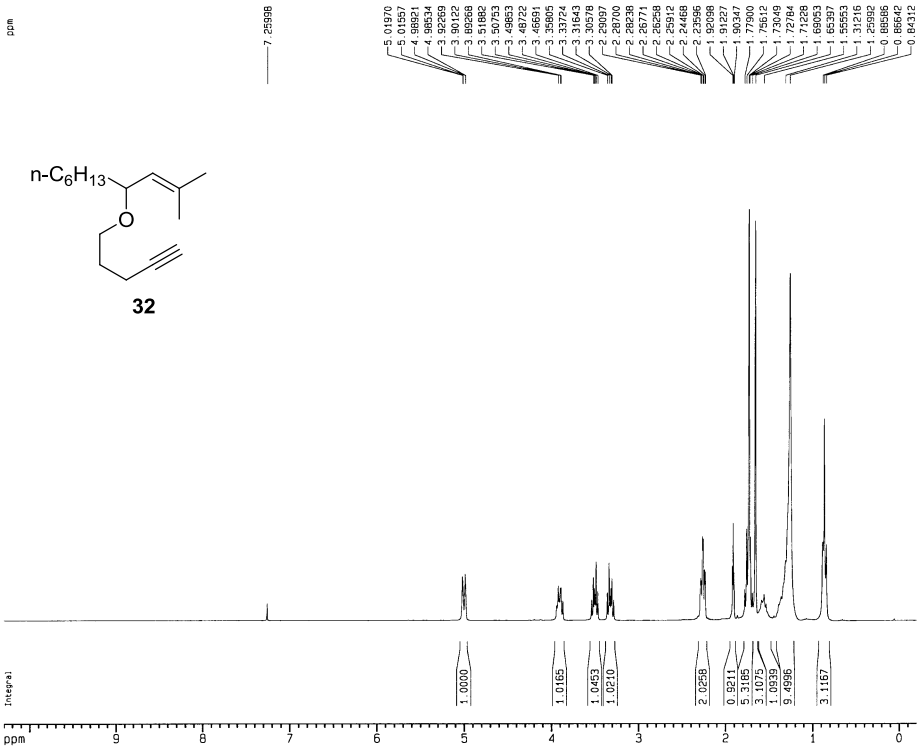
1D NMR plot parameters
CX 22.00 cm
F1P 10.300 ppm
F1 3091.34 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm

WT-1-55-P-1 13 1 v yhr 20100502



NAME WT-1-55-P-1
EXPNO 13
PROCNO 1
Date_ 500000
Time 16.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1025
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 13004
DW 21.000 usec
DE 6.00 usec
TE 300.0 K
D12 0.00002000 sec
PL13 17.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1312005 MHz
NUC2 1H
PL2 -4.00 dB
PL12 15.80 dB
P1 7.50 usec
DE 6.00 usec
SFO1 75.4772501 MHz
NUC1 13C
PL1 -2.00 dB
D11 0.03000000 sec
SI 32768
SF 75.4558278 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

MR-01-141-D2 1 1 v yhr 20100525

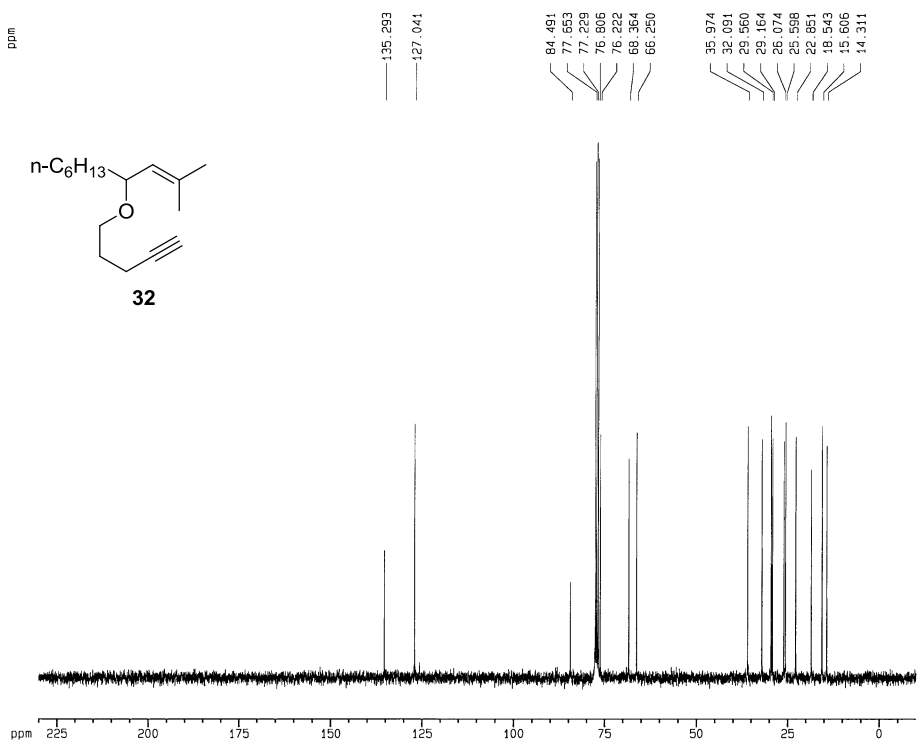


n-C_6H_{13}

32

```
Current Data Parameters
NAME        MR-01-141-D2
EXPNO              1
PROCNO             1

F2 - Acquisition Parameters
Date_          500000
Time            16.59
INSTRUM         spect
PROBHD     5 mm QNP  1H
PULPROG          zg30
TD               32768
SOLVENT          CDCl3
NS                32
DS                 0
SWH          4761.905 Hz
FIDRES       0.145322 Hz
AQ           3.4406900 sec
RG                64
DW            105.000 usec
DE              4.50 usec
TE             300.0 K
D1           2.50000000 sec
P1              11.40 usec
DE              4.50 usec
SFO1       300.1318365 MHz
NUC1               1H
PL1            -4.00 dB

F2 - Processing parameters
SI               16384
SF         300.1300063 MHz
WDW               EM
SSB                0
LB              0.20 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX              22.00 cm
F1P            10.300 ppm
F1            3091.34 Hz
F2P            -0.200 ppm
F2             -60.03 Hz
PPMCM         0.47727 ppm/cm
HZCM        143.24385 Hz/cm
```

MR-01-141-P2-2 13 1 v yhr 20100514(column 4~5)



n-C_6H_{13}

32

```
Current Data Parameters
NAME      MR-01-141-P2-2
EXPNO              13
PROCNO              1

F2 - Acquisition Parameters
Date_          500000
Time            22.28
INSTRUM         spect
PROBHD     5 mm  QNP Pr
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                585
DS                  2
SWH         18382.354 Hz
FIDRES       0.280492 Hz
AQ           1.7826293 sec
RG               2048
DW            27.200 usec
DE              4.50 usec
TE             300.0 K
d11          0.0300000 sec
d12          0.0000200 sec
PL13            25.00 dB
D1           1.00000000 sec
CPDPRG2        waltz16
PCPD2          100.00 usec
SFO2       300.1312005 MHz
NUC2               1H
PL2              1.00 dB
PL12            23.00 dB
P1               7.10 usec
DE              4.50 usec
SFO1        75.4759022 MHz
NUC1               13C
PL1              2.00 dB

F2 - Processing parameters
SI               32768
SF         75.4677328 MHz
WDW               EM
SSB                0
LB              1.30 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX              22.00 cm
F1P            240.000 ppm
F1           18112.26 Hz
F2P           -10.000 ppm
F2            -754.68 Hz
PPMCM        11.36364 ppm/cm
HZCM       857.58789 Hz/cm
```