

Supporting Information

Gold(I)-Catalyzed Enantioselective Synthesis of Benzopyrans *via* Rearrangement of Allylic Oxonium Intermediates

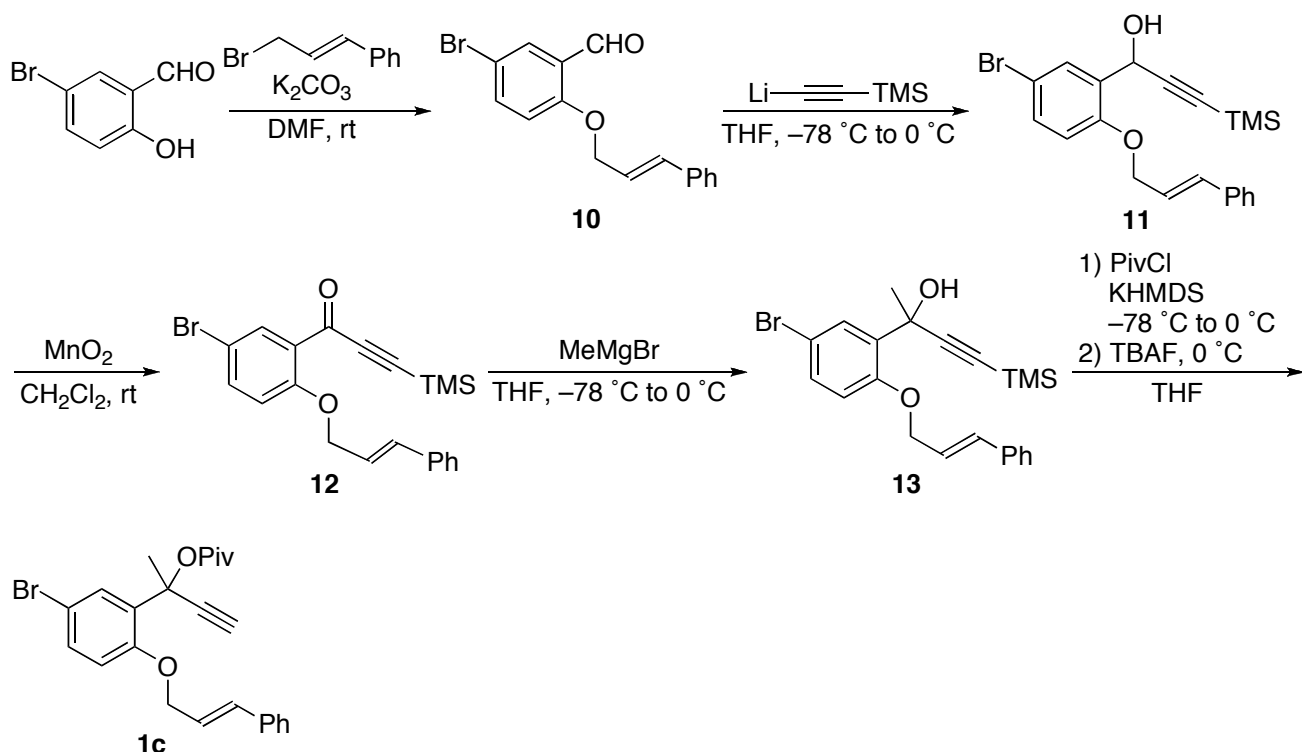
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General Information

Unless otherwise noted, reagents were obtained commercially and used without further purification. MeCN was distilled from CaH₂ under a nitrogen atmosphere. THF was purified by passage through a column of activated alumina under argon. TLC analysis of reaction mixtures was performed on Merck silica gel 60 F254 TLC plates. Flash chromatography was carried out on ICN SiliTech 32-63 D 60 Å silica gel. ¹H and ¹³C NMR spectra were recorded with Bruker AV-300, AVB-400, and AVQ-400 spectrometers and referenced to CDCl₃ unless otherwise noted. Mass spectral and analytical data were obtained via the Micro-Mass/Analytical Facility operated by the College of Chemistry, University of California, Berkeley. X-Ray crystallographic analysis was carried out by Dr. Fred Hollander at the College of Chemistry X-Ray Crystallographic Facility (CHEXRAY, University of California, Berkeley).

General preparation of propargyl pivaloate esters 1



A solution of cinnamyl bromide (5.52 g, 28 mmol) in DMF (7 mL) was added to a mixture of 5-bromosalicylaldehyde (4.02 g, 20 mmol) and potassium carbonate (3.86 g, 28 mmol) in DMF (67

mL) at room temperature. The mixture was stirred for 12 h at room temperature. After quenching the reaction with water, the mixture was extracted with hexanes/ethyl acetate (5:1). The combined organic layers were washed with brine, dried over anhydrous MgSO_4 , and concentrated in vacuo. Purification by recrystallization (hexanes– CH_2Cl_2) afforded **10** (6.19 g, 19.5 mmol) in 98% yield.

A solution of *n*-BuLi in hexanes (2.5 M, 2.64 mL, 6.6 mmol) was added to a solution of trimethylsilylacetylene (1.02 mL, 7.2 mmol) in THF (20 mL) at 0 °C. The solution was stirred for 30 min at 0 °C, then was cooled to –78 °C. To this was added a solution of **10** (1.90 g, 6.0 mmol) in THF (3 mL) at –78 °C. The solution was warmed to 0 °C, then stirred for 30 min. After quenching the reaction with water, the mixture was extracted with hexanes/ethyl acetate (5:1). The combined organic layers were washed with brine, dried over anhydrous MgSO_4 , and concentrated in vacuo to afford a propargylic alcohol **11**, which was used for the next step without further purification.

Manganese(IV) oxide (20.9 g, 240 mmol) was added to a solution of **11** in dichloromethane (20 mL) at room temperature. After the mixture was stirred for 5 min at room temperature, the reaction was filtered through Celite[®]. The filtrate was concentrated in vacuo, and purified by recrystallization (hexanes– CH_2Cl_2) to afford **12** (2.10 g, 5.08 mmol) in 85% yield (2 steps).

A solution of methylmagnesium bromide in ethyl ether (3 M, 0.73 mL, 2.2 mmol) was added to a solution of **12** (827 mg, 2.0 mmol) in THF (6.7 mL) at –78 °C. After the suspension was stirred at 0 °C for 30 min, the suspension was quenched with water. The mixture was extracted with hexanes/ethyl acetate (5:1). The combined organic layers were washed with brine, dried over anhydrous MgSO_4 , and concentrated in vacuo. The oil was filtered through a short pad of silica gel (hexanes/ethyl ether = 10:1) to afford **13**, which was used for the next step without further purification.

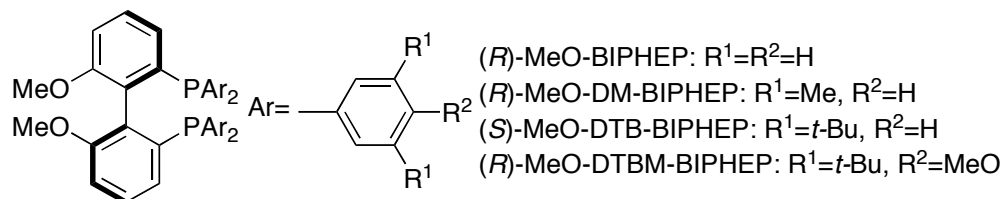
A solution of potassium bis(trimethylsilyl)amide (409 mg, 2.05 mmol) in THF (2.0 mL) was added dropwise to a solution of **13** in THF (20 mL) at –78 °C. The solution was stirred for 20 min at –78 °C. Pivaloyl chloride (0.26 mL, 2.15 mmol) was then added to the solution at –78 °C and the reaction mixture was stirred for 10 min at 0 °C. Tetrabutylammonium fluoride in THF (1 M, 2.93 mL, 2.93 mmol) was added to a solution at 0 °C, and the resulting mixture was stirred for 10 min at 0 °C. After the reaction was terminated by an addition of water, the organic layer was extracted with hexane/ethyl acetate (5:1). The combined organic parts were washed with brine, dried over MgSO_4 , and concentrated in vacuo to give a crude oil. The oil was purified by column chromatography (pretreated with 1% Et_3N in hexanes and eluted with hexanes/ethyl ether = 20:1) to afford propargyl ester **1c** (783 mg, 1.77 mmol) in 89% yield (2 steps).

General procedure of gold-catalyzed asymmetric carboalkoxylation with **1**

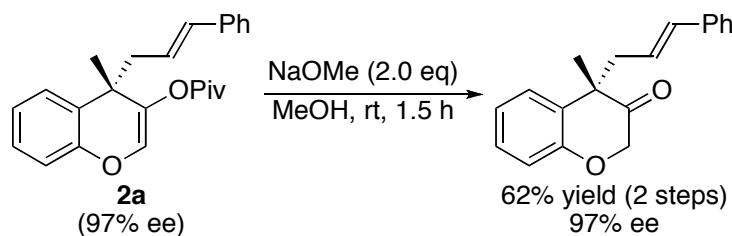
(*R*)-MeO-DTBM-BIPHEP(AuCl)₂ (7.8 mg, 0.0050 mmol) was added to a suspension of AgSbF_6 (3.4 mg, 0.010 mmol) in MeCN (0.5 mL) at room temperature. After the suspension was sonicated for 30 sec, the suspension was left for 10 min at room temperature. The suspension was filtered with glass microfibre filter (Whatman, grade GF/D), and the filtrate was added to a solution of **1a** (36.2 mg, 0.10 mmol) in MeCN (0.5 mL) at room temperature. The mixture was stirred for 1 h at room temperature. After quenching the reaction with Et_3N , the mixture was filtered with glass microfibre filter (Whatman, grade GF/D). The filtrate was concentrated in vacuo, and purified by

chromatography on silica gel (pretreated with 1% Et₃N in hexanes and eluted with hexanes/ethyl ether = 50:1) afforded **2a** (26.9 mg, 0.074 mmol) in 74% yield and 97% ee.

The structures of the ligands are shown below.

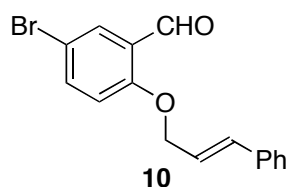


General procedure of cleavage of pivaloate group

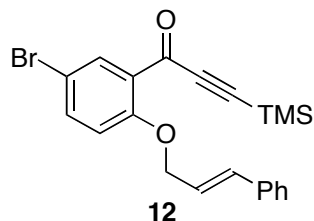


A solution of NaOMe in MeOH (30 wt%, 0.036 mL, 0.20 mmol) was added to a solution of crude mixture of **2a** (0.1 mmol scale from **1a**) in MeOH (0.33 mL) at 0 °C. The mixture was stirred for 1.5 h at room temperature. After the reaction was terminated by an addition of water, the organic layer was extracted with ethyl ether. The combined organic parts were washed with brine, dried over MgSO₄, and concentrated in vacuo to give a crude oil. The oil was purified by column chromatography (pretreated with 1% Et₃N in hexanes and eluted with hexanes/CH₂Cl₂ = 5:1 to 3:1) to afford the corresponding ketone (17.2 mg, 0.062 mmol) in 62% yield (2 steps) and 97% ee.

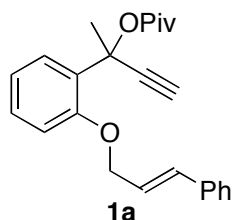
Characterization data for compounds



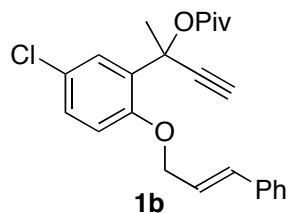
¹H NMR (CDCl₃) δ 4.82 (d, *J* = 5.6 Hz, 2H), 6.40 (dt, *J* = 5.6, 16.0 Hz, 1H), 6.76 (d, *J* = 16.0 Hz, 1H), 6.92–6.98 (m, 1H), 7.25–7.45 (m, 5H), 7.59–7.65 (m, 1H), 7.93–7.97 (m, 1H), 10.47 (s, 1H); ¹³C NMR (CDCl₃) δ 69.50, 113.68, 114.97, 122.78, 126.34, 126.61, 128.31, 128.70, 131.04, 134.01, 135.83, 138.21, 159.83, 188.33. HRMS (EI) Calcd for C₁₆H₁₃BrO₂ [M]: 316.0099. Found: 316.0106.



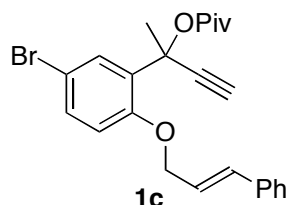
^1H NMR (CDCl_3) δ 0.24 (s, 9H), 4.82 (dd, $J = 1.6, 5.6$ Hz, 2H), 6.39 (dt, $J = 5.6, 16.0$ Hz, 1H), 6.80 (d, $J = 16.0$ Hz, 1H), 6.89–6.95 (m, 1H), 7.23–7.42 (m, 5H), 7.54–7.60 (m, 1H), 8.04–8.07 (m, 1H); ^{13}C NMR (CDCl_3) δ -0.76, 69.72, 100.03, 102.53, 112.76, 115.55, 123.18, 126.61, 128.07, 128.40, 128.60, 133.37, 134.84, 136.07, 137.23, 157.70, 175.10. HRMS (EI) Calcd for $\text{C}_{21}\text{H}_{21}\text{BrO}_2\text{Si}$ [M]: 412.0494. Found: 412.0487.



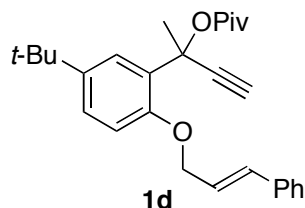
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.08 (s, 3H), 2.74 (s, 1H), 4.73 (d, $J = 5.4$ Hz, 2H), 6.41 (dt, $J = 5.4, 15.9$ Hz, 1H), 6.79 (d, $J = 15.9$ Hz, 1H), 6.90–7.01 (m, 2H), 7.22–7.44 (m, 6H), 7.79–7.85 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.07, 28.21, 39.05, 68.52, 74.32, 74.99, 83.86, 112.59, 120.43, 124.38, 126.46, 127.83, 128.26, 128.62, 128.91, 129.31, 132.32, 136.49, 155.37, 176.11. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{26}\text{LiO}_3$ [M + Li]: 369.2042. Found: 369.2035.



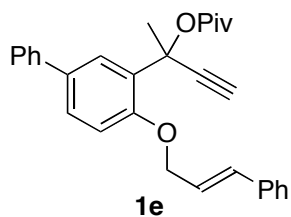
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.05 (s, 3H), 2.78 (s, 1H), 4.70 (dd, $J = 1.2, 5.6$ Hz, 2H), 6.38 (dt, $J = 5.6, 16.0$ Hz, 1H), 6.77 (d, $J = 16.0$ Hz, 1H), 6.83–6.87 (m, 1H), 7.19–7.43 (m, 6H), 7.78–7.82 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.03, 28.07, 39.01, 68.89, 74.42, 74.87, 83.14, 113.81, 123.80, 125.49, 126.47, 127.98, 128.43, 128.65, 128.90, 130.64, 132.73, 136.26, 153.91, 176.09. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{ClLiO}_3$ [M + Li]: 403.1652. Found: 403.1649.



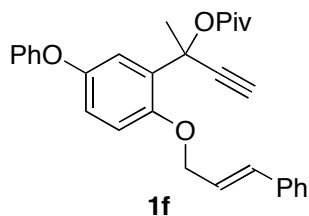
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.04 (s, 3H), 2.78 (s, 1H), 4.70 (d, $J = 5.2$ Hz, 2H), 6.37 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.76 (d, $J = 16.0$ Hz, 1H), 6.77–6.83 (m, 1H), 7.25–7.42 (m, 6H), 7.90–7.95 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.02, 28.06, 39.00, 68.82, 74.34, 74.95, 83.10, 112.85, 114.28, 123.72, 126.46, 127.98, 128.65, 128.65, 131.00, 131.21, 131.91, 132.76, 136.23, 154.40, 176.09. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{BrLiO}_3$ [$\text{M} + \text{Li}$]: 447.1147. Found: 447.1145.



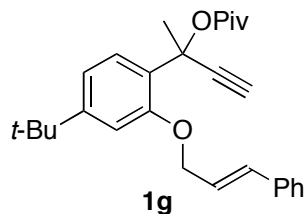
^1H NMR (CDCl_3) δ 1.22 (s, 9H), 1.32 (s, 9H), 2.07 (s, 3H), 2.74 (s, 1H), 4.71 (dd, $J = 1.2, 5.6$ Hz, 2H), 6.41 (dt, $J = 5.6, 16.0$ Hz, 1H), 6.79 (d, $J = 16.0$ Hz, 1H), 6.83–6.89 (m, 1H), 7.22–7.44 (m, 6H), 7.79–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.12, 28.32, 31.49, 34.24, 39.11, 68.65, 74.05, 75.15, 83.96, 112.16, 124.72, 125.00, 125.74, 126.45, 127.77, 128.38, 128.60, 132.09, 136.57, 142.84, 153.06, 176.02. HRMS (FAB) Calcd for $\text{C}_{28}\text{H}_{34}\text{O}_3$ [M]: 418.2508. Found: 418.2512.



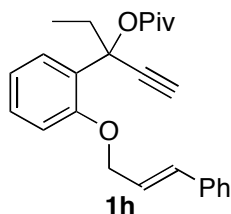
^1H NMR (CDCl_3) δ 1.30 (s, 9H), 2.20 (s, 3H), 2.85 (s, 1H), 4.81 (d, $J = 4.8$ Hz, 2H), 6.48 (dt, $J = 4.8, 16.0$ Hz, 1H), 6.88 (d, $J = 16.0$ Hz, 1H), 7.02–7.08 (m, 1H), 7.30–7.69 (m, 11H), 8.14–8.19 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.04, 28.24, 39.00, 68.62, 74.58, 74.98, 83.66, 112.90, 124.18, 126.40, 126.64, 126.74, 127.07, 127.64, 127.81, 128.57, 128.60, 129.12, 132.36, 133.29, 136.36, 140.63, 154.83, 176.03. HRMS (FAB) Calcd for $\text{C}_{30}\text{H}_{30}\text{O}_3$ [M]: 438.2195. Found: 438.2191.



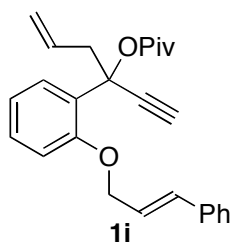
^1H NMR (CDCl_3) δ 1.18 (s, 9H), 2.06 (s, 3H), 2.68 (s, 1H), 4.73 (d, $J = 4.8$ Hz, 2H), 6.41 (dt, $J = 4.8, 16.0$ Hz, 1H), 6.80 (d, $J = 16.0$ Hz, 1H), 6.87–7.10 (m, 5H), 7.24–7.50 (m, 8H); ^{13}C NMR (CDCl_3) δ 27.02, 28.24, 39.03, 69.20, 74.15, 74.40, 83.47, 113.85, 118.16, 119.29, 122.68, 124.42, 126.47, 127.86, 128.63, 129.64, 130.76, 132.36, 136.46, 150.02, 151.35, 157.86, 175.94. HRMS (FAB) Calcd for $\text{C}_{30}\text{H}_{30}\text{O}_4$ [M]: 454.2144. Found: 454.2135.



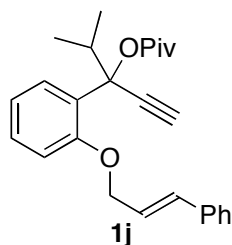
^1H NMR (CDCl_3) δ 1.21 (s, 9H), 1.31 (s, 9H), 2.08 (s, 3H), 2.72 (s, 1H), 4.75 (d, $J = 5.2$ Hz, 2H), 6.41 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.82 (d, $J = 16.0$ Hz, 1H), 6.92–7.01 (m, 2H), 7.23–7.45 (m, 5H), 7.65–7.72 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.08, 28.24, 31.28, 34.70, 39.09, 68.56, 73.99, 74.77, 84.05, 110.25, 117.35, 124.70, 126.12, 126.43, 127.53, 127.77, 128.60, 132.31, 136.59, 152.47, 155.11, 176.08. HRMS (FAB/LiCl) Calcd for $\text{C}_{28}\text{H}_{34}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 425.2668. Found: 425.2666.



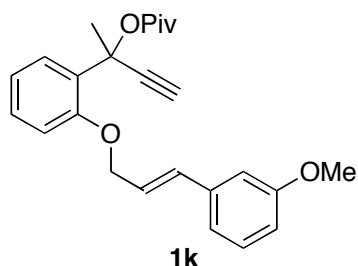
^1H NMR (CDCl_3) δ 1.00 (t, $J = 7.6$ Hz, 3H), 1.21 (s, 9H), 2.35 (dq, $J = 7.6, 14.4$ Hz, 1H), 2.50 (dq, $J = 7.6, 14.4$ Hz, 1H), 2.77 (s, 1H), 4.72 (d, $J = 5.2$ Hz, 2H), 6.40 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.79 (d, $J = 16.0$ Hz, 1H), 6.88–7.01 (m, 2H), 7.22–7.44 (m, 6H), 7.77–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 8.85, 27.08, 32.85, 39.11, 68.37, 75.31, 79.17, 82.48, 112.45, 120.34, 124.41, 126.42, 127.58, 127.80, 128.62, 129.18, 129.34, 132.11, 136.48, 155.17, 175.92. HRMS (FAB/LiCl) Calcd for $\text{C}_{25}\text{H}_{28}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 383.2199. Found: 383.2200.



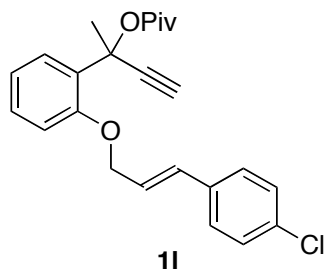
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.79 (s, 1H), 3.12 (dd, $J = 7.2, 13.6$ Hz, 1H), 3.29 (dd, $J = 7.2, 13.6$ Hz, 1H), 4.74 (dd, $J = 1.2, 5.2$ Hz, 2H), 5.07 (d, $J = 9.6$ Hz, 1H), 5.08 (d, $J = 17.2$ Hz, 1H), 5.79 (ddt, $J = 7.2, 9.6, 17.2$ Hz, 1H), 6.41 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.80 (d, $J = 16.0$ Hz, 1H), 6.90–7.00 (m, 2H), 7.24–7.43 (m, 6H), 7.76–7.81 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.07, 39.14, 43.91, 68.46, 75.75, 77.53, 82.29, 112.51, 118.51, 120.40, 124.29, 126.43, 127.27, 127.83, 128.62, 129.22, 129.38, 132.27, 132.67, 136.45, 155.22, 175.81. HRMS (FAB/LiCl) Calcd for $\text{C}_{26}\text{H}_{28}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 395.2199. Found: 395.2203.



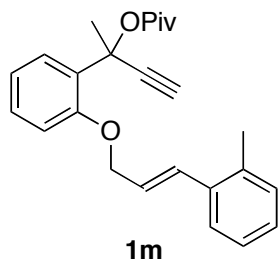
^1H NMR (CDCl_3) δ 0.75 (d, $J = 6.8$ Hz, 3H), 1.18 (s, 9H), 1.23 (d, $J = 6.8$ Hz, 3H), 2.76 (s, 1H), 3.07 (qq, $J = 6.8, 6.8$ Hz, 1H), 4.64–4.75 (m, 2H), 6.38 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.77 (d, $J = 16.0$ Hz, 1H), 6.86–6.99 (m, 2H), 7.21–7.43 (m, 6H), 7.80–7.87 (m, 1H); ^{13}C NMR (CDCl_3) δ 17.79, 27.08, 34.86, 39.19, 68.18, 80.12, 83.06, 112.33, 120.19, 124.45, 126.41, 127.29, 127.79, 128.64, 129.09, 130.50, 131.95, 136.53, 155.00, 175.81. HRMS (ESI) Calcd for $\text{C}_{26}\text{H}_{30}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 413.2087. Found: 413.2085.



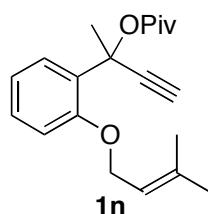
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.08 (s, 3H), 2.74 (s, 1H), 3.83 (s, 3H), 4.70–4.80 (m, 2H), 6.40 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.76 (d, $J = 16.0$ Hz, 1H), 6.80–6.86 (m, 1H), 6.91–7.02 (m, 4H), 7.23–7.30 (m, 2H), 7.80–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.06, 28.22, 39.03, 55.20, 68.44, 74.32, 74.99, 83.85, 111.89, 112.59, 113.32, 119.09, 120.44, 124.73, 128.25, 128.91, 129.30, 129.59, 132.15, 137.94, 155.34, 159.81, 176.06. HRMS (FAB/LiCl) Calcd for $\text{C}_{25}\text{H}_{28}\text{LiO}_4$ [$\text{M} + \text{Li}$]: 399.2148. Found: 399.2152.



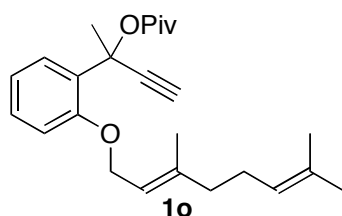
^1H NMR (CDCl_3) δ 1.19 (s, 9H), 2.08 (s, 3H), 2.74 (s, 1H), 4.72 (d, $J = 5.2$ Hz, 2H), 6.38 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.76 (d, $J = 16.0$ Hz, 1H), 6.72–6.79 (m, 1H), 6.89–6.94 (m, 1H), 6.95–7.01 (m, 1H), 7.23–7.35 (m, 4H), 7.78–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.03, 28.18, 39.01, 68.25, 74.30, 74.90, 83.79, 112.53, 120.51, 125.00, 127.62, 128.20, 128.76, 128.89, 129.30, 130.91, 133.42, 134.93, 155.20, 176.03. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{ClLiO}_3$ [$\text{M} + \text{Li}$]: 403.1652. Found: 403.1652.



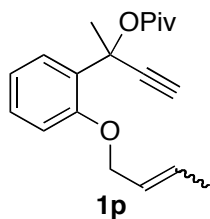
^1H NMR (CDCl_3) δ 1.20 (s, 9H), 2.10 (s, 3H), 2.36 (s, 3H), 2.74 (s, 1H), 4.73 (d, $J = 5.4$ Hz, 2H), 6.29 (dt, $J = 5.2, 16.0$ Hz, 1H), 6.92–6.99 (m, 2H), 7.02 (d, $J = 16.0$ Hz, 1H), 7.13–7.32 (m, 4H), 7.42–7.49 (m, 1H), 7.79–7.85 (m, 1H); ^{13}C NMR (CDCl_3) δ 19.81, 27.08, 28.15, 39.05, 68.64, 74.31, 74.98, 83.87, 112.60, 120.42, 125.65, 125.70, 126.14, 127.72, 128.26, 128.91, 129.29, 130.27, 130.32, 135.59, 135.72, 155.35, 176.07. HRMS (FAB/LiCl) Calcd for $\text{C}_{25}\text{H}_{28}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 383.2199. Found: 383.2195.



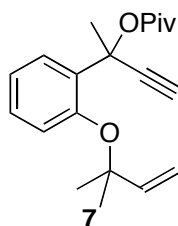
^1H NMR (CDCl_3) δ 1.19 (s, 9H), 1.71 (s, 3H), 1.78 (s, 3H), 2.03 (s, 3H), 2.73 (s, 1H), 4.47–4.60 (m, 2H), 5.44–5.52 (m, 1H), 6.84–6.97 (m, 2H), 7.21–7.29 (m, 1H), 7.78–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 18.19, 25.66, 27.00, 28.13, 38.98, 64.84, 74.28, 75.08, 83.90, 112.33, 119.96, 120.12, 128.27, 128.75, 129.19, 136.60, 155.65, 176.06. HRMS (FAB/LiCl) Calcd for $\text{C}_{20}\text{H}_{26}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 321.2042. Found: 321.2038.



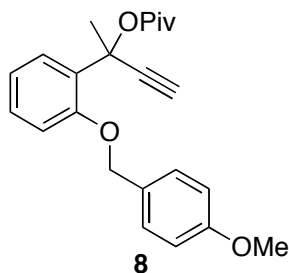
^1H NMR (CDCl_3) δ 1.19 (s, 9H), 1.61 (s, 3H), 1.69 (s, 3H), 1.70 (s, 3H), 2.02–2.16 (m, 4H), 2.03 (s, 3H), 2.73 (s, 1H), 4.50–4.61 (m, 2H), 5.06–5.14 (m, 1H), 5.44–5.52 (m, 1H), 6.84–6.97 (m, 2H), 7.21–7.29 (m, 1H), 7.78–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 16.57, 17.66, 25.67, 26.25, 27.04, 28.14, 38.97, 39.41, 64.92, 74.31, 75.09, 83.90, 112.35, 119.97, 123.76, 128.31, 128.72, 129.19, 131.74, 139.81, 155.68, 176.02. HRMS (FAB/LiCl) Calcd for $\text{C}_{25}\text{H}_{34}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 389.2668. Found: 389.2660.



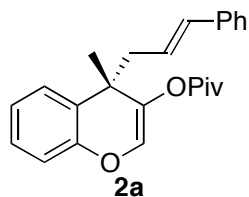
(*trans*:*cis* = 81:19) ^1H NMR (CDCl_3) δ 1.19 (s, 9H), 1.70–1.73 (m, $3 \times 0.19\text{H}$), 1.75 (dd, $J = 1.2, 5.2$ Hz, $3 \times 0.81\text{H}$), 2.03 (s, $3 \times 0.19\text{H}$), 2.04 (s, $3 \times 0.81\text{H}$), 2.73 (s, 1H), 4.48 (dd, $J = 1.2, 5.6$ Hz, $2 \times 0.81\text{H}$), 4.59–4.63 (m, $2 \times 0.19\text{H}$), 5.66–5.90 (m, 2H), 6.84–6.98 (m, 2H), 7.20–7.29 (m, 1H), 7.77–7.84 (m, 1H); ^{13}C NMR (*trans*, CDCl_3) δ 17.77, 27.03, 28.11, 39.01, 68.55, 74.29, 75.05, 83.88, 112.46, 120.09, 126.06, 128.25, 128.74, 129.20, 129.43, 155.51, 176.06. HRMS (FAB) Calcd for $\text{C}_{19}\text{H}_{24}\text{O}_3$ [M]: 300.1725. Found: 300.1720.



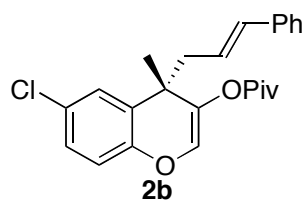
^1H NMR (CDCl_3) δ 1.32 (s, 9H), 1.65 (s, 6H), 2.17 (s, 3H), 2.74 (s, 1H), 5.23 (d, $J = 10.9$ Hz, 1H), 5.28 (d, $J = 17.7$ Hz, 1H), 6.23 (dd, $J = 10.9, 17.7$ Hz, 1H), 6.96 (m, 1H), 7.30–7.15 (m, 2H), 7.81 (d, $J = 7.6$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 26.5, 27.3, 27.8, 28.5, 39.3, 73.9, 75.0, 79.8, 84.5, 113.2, 117.3, 119.8, 127.9, 128.3, 130.1, 145.2, 153.6, 176.0.



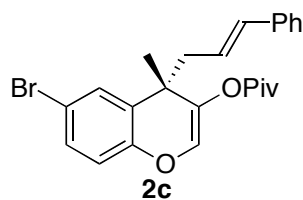
^1H NMR (CDCl_3) δ 1.12 (s, 9H), 2.07 (s, 3H), 2.72 (s, 1H), 3.83 (s, 3H), 5.01–5.07 (m, 2H), 6.88–7.01 (m, 4H), 7.23–7.30 (m, 1H), 7.34–7.41 (m, 2H), 7.78–7.84 (m, 1H); ^{13}C NMR (CDCl_3) δ 26.95, 28.10, 38.98, 55.26, 69.73, 74.25, 74.92, 83.89, 112.62, 113.80, 120.37, 128.13, 128.78, 128.94, 129.29, 155.53, 159.22, 176.11. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{26}\text{NaO}_4$ [M + Na]: 389.1723. Found: 389.1733.



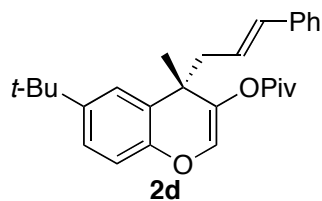
^1H NMR (CDCl_3) δ 1.33 (s, 9H), 1.50 (s, 3H), 2.52 (dd, $J = 7.2, 14.1$ Hz, 1H), 2.64 (dd, $J = 7.2, 14.1$ Hz, 1H), 6.00 (dt, $J = 7.2, 15.6$ Hz, 1H), 6.23 (d, $J = 15.6$ Hz, 1H), 6.79 (s, 1H), 6.85–6.91 (m, 1H), 7.01–7.32 (m, 8H); ^{13}C NMR (CDCl_3) δ 27.29, 27.35, 39.39, 40.05, 45.01, 116.34, 123.22, 126.10, 126.28, 126.47, 126.66, 126.94, 127.54, 128.33, 132.86, 133.60, 133.65, 137.57, 149.85, 176.72. HRMS (ESI) Calcd for $\text{C}_{24}\text{H}_{26}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 385.1774. Found: 385.1773. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_R 5.5 min (major), 6.5 min (minor): 97% ee.



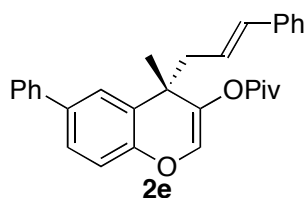
^1H NMR (CDCl_3) δ 1.33 (s, 9H), 1.49 (s, 3H), 2.47 (dd, $J = 7.2, 13.6$ Hz, 1H), 2.64 (dd, $J = 7.2, 13.6$ Hz, 1H), 5.98 (dt, $J = 7.2, 15.6$ Hz, 1H), 6.25 (d, $J = 15.6$ Hz, 1H), 6.79 (s, 1H), 6.79–6.85 (m, 1H), 7.06–7.28 (m, 7H); ^{13}C NMR (CDCl_3) δ 27.27, 27.31, 39.41, 40.31, 44.96, 117.84, 125.67, 126.14, 126.50, 127.09, 127.73, 128.09, 128.27, 128.37, 133.19, 133.30, 133.60, 137.39, 148.53, 176.62. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{ClLiO}_3$ [$\text{M} + \text{Li}$]: 403.1652. Found: 403.1641. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_R 5.5 min (major), 6.6 min (minor): 97% ee.



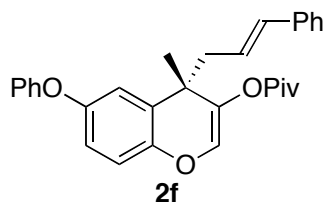
^1H NMR (CDCl_3) δ 1.32 (s, 9H), 1.49 (s, 3H), 2.47 (dd, $J = 7.2, 13.6$ Hz, 1H), 2.64 (dd, $J = 7.2, 13.6$ Hz, 1H), 5.98 (dt, $J = 7.2, 16.0$ Hz, 1H), 6.26 (d, $J = 16.0$ Hz, 1H), 6.75–6.79 (m, 1H), 6.79 (s, 1H), 7.14–7.30 (m, 6H), 7.36–7.40 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.27, 27.33, 39.41, 40.26, 45.01, 115.50, 118.25, 125.64, 126.14, 127.09, 128.37, 128.77, 129.50, 130.61, 133.27, 133.33, 133.56, 137.38, 149.02, 176.61. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{BrLiO}_3$ [$\text{M} + \text{Li}$]: 447.1147. Found: 447.1150. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_R 5.4 min (major), 6.6 min (minor): 94% ee.



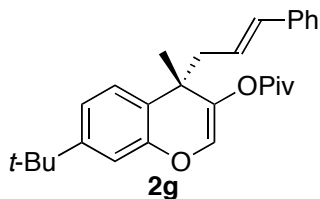
^1H NMR (CDCl_3) δ 1.30 (s, 9H), 1.33 (s, 9H), 1.50 (s, 3H), 2.53 (dd, $J = 6.9, 14.1$ Hz, 1H), 2.63 (dd, $J = 6.9, 14.1$ Hz, 1H), 6.01 (dt, $J = 6.9, 15.6$ Hz, 1H), 6.20 (d, $J = 15.6$ Hz, 1H), 6.76 (s, 1H), 6.76–6.84 (m, 1H), 7.11–7.29 (m, 7H); ^{13}C NMR (CDCl_3) δ 27.21, 27.31, 31.47, 34.36, 39.38, 40.20, 45.04, 115.67, 123.26, 124.57, 125.54, 126.06, 126.40, 126.91, 128.32, 132.88, 133.62, 133.68, 137.59, 145.80, 147.62, 176.78. HRMS (FAB/LiCl) Calcd for $\text{C}_{28}\text{H}_{34}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 425.2668. Found: 425.2658. HPLC Chiralpak AD-H column (Hex, 0.5 mL/min) t_{R} 8.5 min (major), 9.8 min (minor): 99% ee.



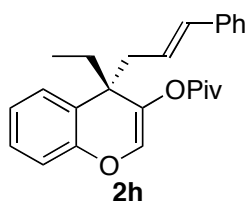
^1H NMR (CDCl_3) δ 1.34 (s, 9H), 1.56 (s, 3H), 2.59 (dd, $J = 8.0, 13.6$ Hz, 1H), 2.68 (dd, $J = 8.0, 13.6$ Hz, 1H), 6.06 (dt, $J = 7.2, 15.6$ Hz, 1H), 6.26 (d, $J = 15.6$ Hz, 1H), 6.82 (s, 1H), 6.92–6.98 (m, 1H), 7.12–7.57 (m, 12H); ^{13}C NMR (CDCl_3) δ 27.30, 27.33, 39.41, 40.24, 45.11, 116.73, 125.49, 126.13, 126.19, 126.49, 126.73, 126.87, 127.00, 128.35, 128.75, 133.07, 133.63, 133.69, 136.33, 137.52, 140.73, 149.40, 176.72. HRMS (FAB/LiCl) Calcd for $\text{C}_{30}\text{H}_{30}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 445.2355. Found: 445.2340. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_{R} 6.2 min (major), 7.6 min (minor): 97% ee.



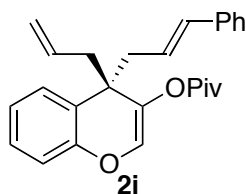
^1H NMR (CDCl_3) δ 1.33 (s, 9H), 1.48 (s, 3H), 2.43 (dd, $J = 7.2, 14.0$ Hz, 1H), 2.64 (dd, $J = 7.2, 14.0$ Hz, 1H), 6.06 (dt, $J = 7.2, 16.0$ Hz, 1H), 6.24 (d, $J = 16.0$ Hz, 1H), 6.78–7.09 (m, 6H), 6.81 (s, 1H), 7.14–7.33 (m, 7H); ^{13}C NMR (CDCl_3) δ 27.29, 27.43, 39.40, 40.36, 44.76, 117.42, 117.62, 117.92, 119.45, 122.54, 126.12, 127.04, 127.91, 128.38, 129.67, 132.91, 133.03, 133.81, 137.47, 146.22, 151.84, 158.28, 176.78. HRMS (ESI) Calcd for $\text{C}_{30}\text{H}_{30}\text{NaO}_4$ [$\text{M} + \text{Na}$]: 477.2036. Found: 477.2048. HPLC Chiralpak OD column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 14.1 min (minor), 18.8 min (major): 97% ee.



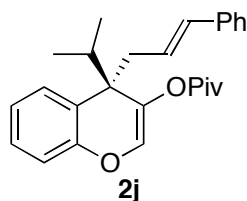
^1H NMR (CDCl_3) δ 1.29 (s, 9H), 1.32 (s, 9H), 1.48 (s, 3H), 2.53 (dd, $J = 7.2, 13.6$ Hz, 1H), 2.63 (dd, $J = 7.2, 13.6$ Hz, 1H), 6.02 (dt, $J = 7.2, 16.0$ Hz, 1H), 6.24 (d, $J = 16.0$ Hz, 1H), 6.78 (s, 1H), 6.87–6.91 (m, 1H), 7.06–7.27 (m, 7H); ^{13}C NMR (CDCl_3) δ 27.27, 27.30, 31.16, 34.39, 39.38, 39.75, 45.05, 113.16, 120.49, 123.42, 126.10, 126.18, 126.51, 126.88, 128.32, 132.73, 133.72, 133.82, 137.69, 149.51, 150.99, 176.73. HRMS (FAB/LiCl) Calcd for $\text{C}_{28}\text{H}_{34}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 425.2668. Found: 425.2662. HPLC Chiralpak OD column (Hex/*i*PrOH 99.9/0.1, 0.5 mL/min) t_{R} 10.8 min (major), 11.9 min (minor): 98% ee.



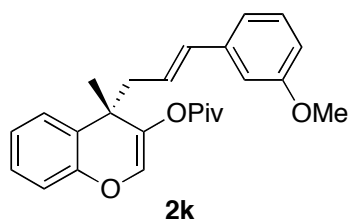
^1H NMR (CDCl_3) δ 0.77 (t, $J = 7.6$ Hz, 3H), 1.33 (s, 9H), 1.67–1.88 (m, 2H), 2.54 (dd, $J = 7.2, 14.0$ Hz, 1H), 2.67 (dd, $J = 7.2, 14.0$ Hz, 1H), 6.04 (dt, $J = 7.2, 16.0$ Hz, 1H), 6.23 (d, $J = 16.0$ Hz, 1H), 6.84–6.94 (m, 1H), 7.01–7.32 (m, 8H), 7.03 (s, 1H); ^{13}C NMR (CDCl_3) δ 9.31, 27.26, 32.00, 39.49, 44.26, 45.09, 116.22, 123.24, 124.09, 126.08, 126.27, 126.30, 126.87, 127.46, 128.30, 131.39, 132.64, 135.35, 137.62, 151.03, 176.22. HRMS (FAB) Calcd for $\text{C}_{25}\text{H}_{29}\text{O}_3$ [$\text{M} + \text{H}$]: 377.2117. Found: 377.2112. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_{R} 5.2 min (major), 6.0 min (minor): 99% ee.



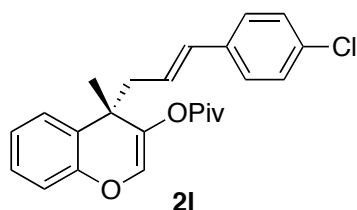
^1H NMR (CDCl_3) δ 1.34 (s, 9H), 2.48 (dd, $J = 7.2, 14.0$ Hz, 1H), 2.58 (dd, $J = 7.2, 14.4$ Hz, 1H), 2.60 (dd, $J = 7.2, 14.4$ Hz, 1H), 2.70 (dd, $J = 7.2, 14.4$ Hz, 1H), 4.91 (d, $J = 15.2$ Hz, 1H), 4.93 (d, $J = 10.0$ Hz, 1H), 5.66 (ddt, $J = 7.2, 10.0, 15.2$ Hz, 1H), 6.03 (dt, $J = 7.2, 15.6$ Hz, 1H), 6.24 (d, $J = 15.6$ Hz, 1H), 6.84–6.89 (m, 1H), 7.02 (s, 1H), 7.03–7.28 (m, 8H); ^{13}C NMR (CDCl_3) δ 27.29, 39.50, 43.66, 43.94, 44.45, 116.32, 117.80, 123.17, 123.86, 125.89, 126.10, 126.66, 126.94, 127.59, 128.32, 131.42, 132.92, 134.02, 135.02, 137.52, 150.65, 176.06. HRMS (ESI) Calcd for $\text{C}_{26}\text{H}_{28}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 411.1931. Found: 411.1937. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_{R} 5.5 min (major), 7.1 min (minor): 99% ee.



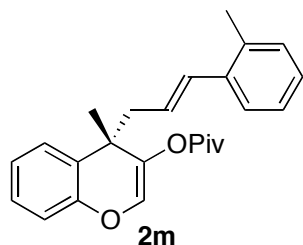
^1H NMR (CDCl_3) δ 0.90 (d, $J = 6.8$ Hz, 3H), 0.95 (d, $J = 6.8$ Hz, 3H), 1.32 (s, 9H), 1.89 (qq, $J = 6.8, 6.8$ Hz, 1H), 2.80 (dd, $J = 7.6, 14.8$ Hz, 1H), 2.86 (dd, $J = 7.6, 14.8$ Hz, 1H), 6.02 (dt, $J = 7.6, 15.6$ Hz, 1H), 6.34 (d, $J = 15.6$ Hz, 1H), 6.87–6.92 (m, 1H), 6.99–7.06 (m, 1H), 7.02 (s, 1H), 7.10–7.26 (m, 7H); ^{13}C NMR (CDCl_3) δ 17.90, 18.77, 27.28, 38.11, 38.85, 39.44, 47.27, 116.19, 122.74, 123.84, 126.03, 126.84, 127.16, 127.32, 127.42, 128.32, 131.97, 132.28, 135.62, 137.64, 151.40, 176.19. HRMS (ESI) Calcd for $\text{C}_{26}\text{H}_{30}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 413.2087. Found: 413.2098. HPLC Chiralpak WH column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_R 10.3 min (major), 11.6 min (minor): 98% ee.



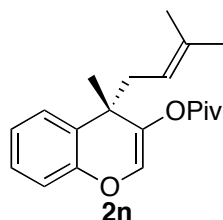
^1H NMR (CDCl_3) δ 1.33 (s, 9H), 1.50 (s, 3H), 2.52 (dd, $J = 7.2, 14.0$ Hz, 1H), 2.64 (dd, $J = 7.2, 14.0$ Hz, 1H), 3.77 (s, 3H), 5.99 (dt, $J = 7.2, 16.0$ Hz, 1H), 6.21 (d, $J = 16.0$ Hz, 1H), 6.79 (s, 1H), 6.69–6.91 (m, 4H), 7.03–7.09 (m, 1H), 7.11–7.18 (m, 2H), 7.25–7.30 (m, 1H); ^{13}C NMR (CDCl_3) δ 27.29, 39.38, 40.04, 45.00, 55.11, 111.75, 112.28, 116.34, 118.76, 123.21, 126.45, 126.64, 126.68, 127.55, 129.28, 132.74, 133.59, 133.65, 139.07, 149.86, 159.60, 176.68. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{28}\text{LiO}_4$ [$\text{M} + \text{Li}$]: 399.2148. Found: 399.2141. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_R 7.3 min (major), 10.3 min (minor): 99% ee.



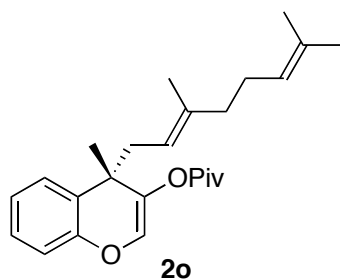
^1H NMR (CDCl_3) δ 1.33 (s, 9H), 1.51 (s, 3H), 2.50 (dd, $J = 7.6, 14.0$ Hz, 1H), 2.62 (dd, $J = 7.6, 14.0$ Hz, 1H), 5.98 (dt, $J = 7.6, 16.0$ Hz, 1H), 6.16 (d, $J = 16.0$ Hz, 1H), 6.79 (s, 1H), 6.85–6.91 (m, 1H), 7.02–7.30 (m, 7H); ^{13}C NMR (CDCl_3) δ 27.29, 39.39, 40.07, 44.95, 116.35, 123.27, 126.38, 126.59, 127.07, 127.28, 127.60, 128.46, 131.66, 132.51, 133.48, 133.72, 136.03, 149.85, 176.71. HRMS (FAB/LiCl) Calcd for $\text{C}_{24}\text{H}_{25}\text{ClLiO}_3$ [$\text{M} + \text{Li}$]: 403.1652. Found: 403.1664. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 1.0 mL/min) t_R 6.5 min (major), 8.0 min (minor): 98% ee.



$^1\text{H NMR}$ (CDCl_3) δ 1.34 (s, 9H), 1.50 (s, 3H), 2.14 (s, 3H), 2.52 (dd, $J = 7.6, 13.6$ Hz, 1H), 2.67 (dd, $J = 7.6, 13.6$ Hz, 1H), 5.84 (dt, $J = 7.6, 15.6$ Hz, 1H), 6.38 (d, $J = 15.6$ Hz, 1H), 6.80 (s, 1H), 6.84–6.90 (m, 1H), 7.03–7.34 (m, 7H); $^{13}\text{C NMR}$ (CDCl_3) δ 19.61, 27.29, 27.60, 39.39, 40.25, 45.01, 116.35, 123.19, 125.88, 125.91, 126.41, 126.69, 126.89, 127.51, 127.74, 129.85, 131.31, 133.48, 133.62, 134.99, 136.97, 149.86, 176.72. HRMS (ESI) Calcd for $\text{C}_{25}\text{H}_{28}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 399.1931. Found: 399.1919. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 6.6 min (major), 7.2 min (minor): 98% ee.

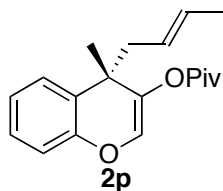


$^1\text{H NMR}$ (CDCl_3) δ 1.31 (s, 9H), 1.44 (s, 3H), 1.46 (s, 3H), 1.58 (s, 3H), 2.30 (dd, $J = 6.8, 13.6$ Hz, 1H), 2.44 (dd, $J = 6.8, 13.6$ Hz, 1H), 4.95 (t, $J = 6.8$ Hz, 1H), 6.77 (s, 1H), 6.86–6.90 (m, 1H), 6.97–7.04 (m, 1H), 7.10–7.16 (m, 1H), 7.20–7.26 (m, 1H); $^{13}\text{C NMR}$ (CDCl_3) δ 17.89, 25.86, 27.09, 27.27, 39.35, 39.73, 40.04, 116.08, 120.08, 123.00, 126.76, 126.98, 127.28, 133.43, 133.78, 134.09, 149.93, 176.74. HRMS (FAB/LiCl) Calcd for $\text{C}_{20}\text{H}_{26}\text{LiO}_3$ [$\text{M} + \text{Li}$]: 321.2042. Found: 321.2035. HPLC Chiralpak OJ column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 9.0 min (minor), 11.9 min (major): 97% ee.

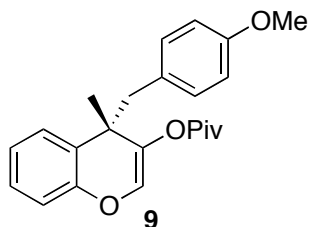


$^1\text{H NMR}$ (CDCl_3) δ 1.31 (s, 9H), 1.44 (s, 3H), 1.45 (s, 3H), 1.54 (s, 3H), 1.64 (s, 3H), 1.82–1.95 (m, 4H), 2.30 (dd, $J = 7.6, 14.4$ Hz, 1H), 2.44 (dd, $J = 7.6, 14.4$ Hz, 1H), 4.98 (t, $J = 6.4$ Hz, 2H), 6.77 (s, 1H), 6.84–6.90 (m, 1H), 6.97–7.04 (m, 1H), 7.08–7.16 (m, 1H), 7.20–7.26 (m, 1H); $^{13}\text{C NMR}$ (CDCl_3) 16.13, 17.61, 25.61, 26.62, 26.97, 27.27, 39.35, 39.86, 116.07, 120.18, 122.96, 124.20, 126.79, 126.90, 127.28, 131.15, 133.44, 134.02, 137.56, 149.97, 176.76. HRMS (ESI) Calcd for $\text{C}_{25}\text{H}_{34}\text{NaO}_3$ [$\text{M} + \text{Na}$]: 405.2400. Found: 405.2410. HPLC Chiralpak OJ column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 8.8 min

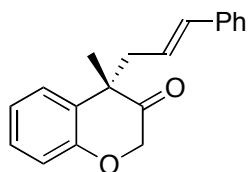
(minor), 10.6 min (major): 97% ee.



^1H NMR (CDCl_3) δ 1.31 (s, 9H), 1.42 (s, 3H), 1.52 (dd, $J = 0.8, 6.0$ Hz, 3H), 2.29 (dd, $J = 6.8, 14.4$ Hz, 1H), 2.41 (dd, $J = 6.8, 14.4$ Hz, 1H), 5.16–5.34 (m, 2H), 6.80 (s, 1H), 6.86–6.90 (m, 1H), 7.00–7.06 (m, 1H), 7.10–7.16 (m, 1H), 7.19–7.23 (m, 1H); ^{13}C NMR (CDCl_3) δ 17.99, 27.27, 27.42, 39.38, 39.80, 44.54, 116.15, 123.03, 126.63, 126.73, 126.81, 127.29, 128.31, 133.38, 133.86, 149.87, 176.66. HRMS (FAB) Calcd for $\text{C}_{19}\text{H}_{25}\text{O}_3$ [$\text{M} + \text{H}$]: 301.1804. Found: 301.1797. HPLC Chiralpak OJ column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 9.5 min (minor), 15.3 min (major): 91% ee.

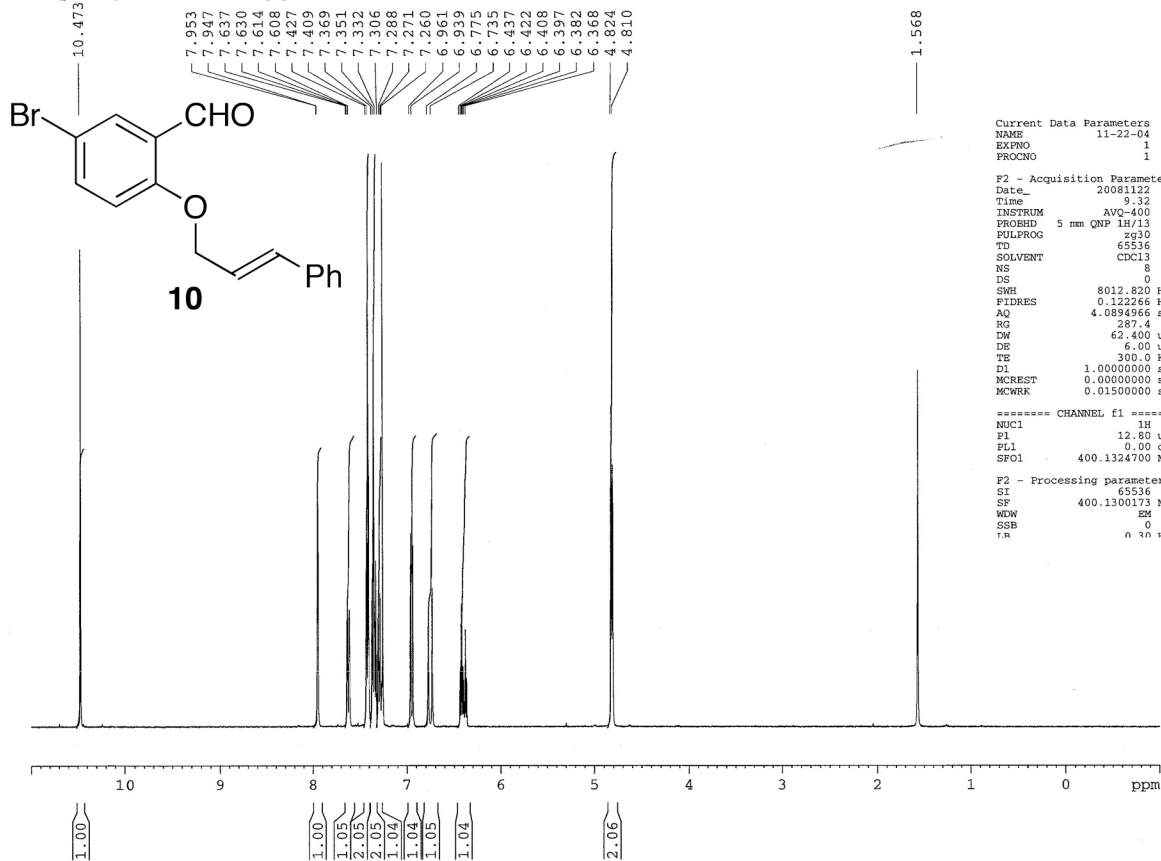


^1H NMR (CDCl_3) δ 1.36 (s, 9H), 1.55 (s, 3H), 2.81 (d, $J = 13.2$ Hz, 1H), 2.96 (d, $J = 13.2$ Hz, 1H), 3.72 (s, 3H), 6.56–6.75 (m, 6H), 7.02–7.14 (m, 2H), 7.28–7.35 (m, 1H); ^{13}C NMR (CDCl_3) δ 26.79, 27.36, 39.41, 41.39, 47.09, 55.02, 112.90, 116.22, 122.93, 126.09, 126.99, 127.49, 129.78, 130.99, 132.91, 133.78, 150.00, 157.94, 176.69. HRMS (ESI) Calcd for $\text{C}_{23}\text{H}_{26}\text{NaO}_4$ [$\text{M} + \text{Na}$]: 389.1723. Found: 389.1733. HPLC Chiralpak OD column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 15.6 min (minor), 19.8 min (major): 94% ee.



^1H NMR (CDCl_3) δ 1.51 (s, 3H), 2.62 (dd, $J = 7.5, 13.8$ Hz, 1H), 2.81 (dd, $J = 7.5, 13.8$ Hz, 1H), 4.42 (d, $J = 17.4$ Hz, 1H), 4.58 (d, $J = 17.4$ Hz, 1H), 5.86 (dt, $J = 7.5, 15.9$ Hz, 1H), 6.31 (d, $J = 15.9$ Hz, 1H), 7.02–6.91 (m, 1H), 7.01–7.30 (m, 8H); ^{13}C NMR (CDCl_3) δ 20.47, 42.07, 50.35, 72.17, 117.93, 123.19, 124.12, 126.18, 126.75, 127.35, 128.40, 128.45, 128.96, 133.91, 137.04, 154.18, 210.65. HRMS (EI) Calcd for $\text{C}_{19}\text{H}_{18}\text{O}_2$ [M]: 278.1307. Found: 278.1310. HPLC Chiralpak AD-H column (Hex/EtOH 99.5/0.5, 0.5 mL/min) t_{R} 11.0 min (major), 14.5 min (minor): 97% ee.

AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



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Current Data Parameters
NAME      11-22-04
EXFNO    1
PROCNO   1

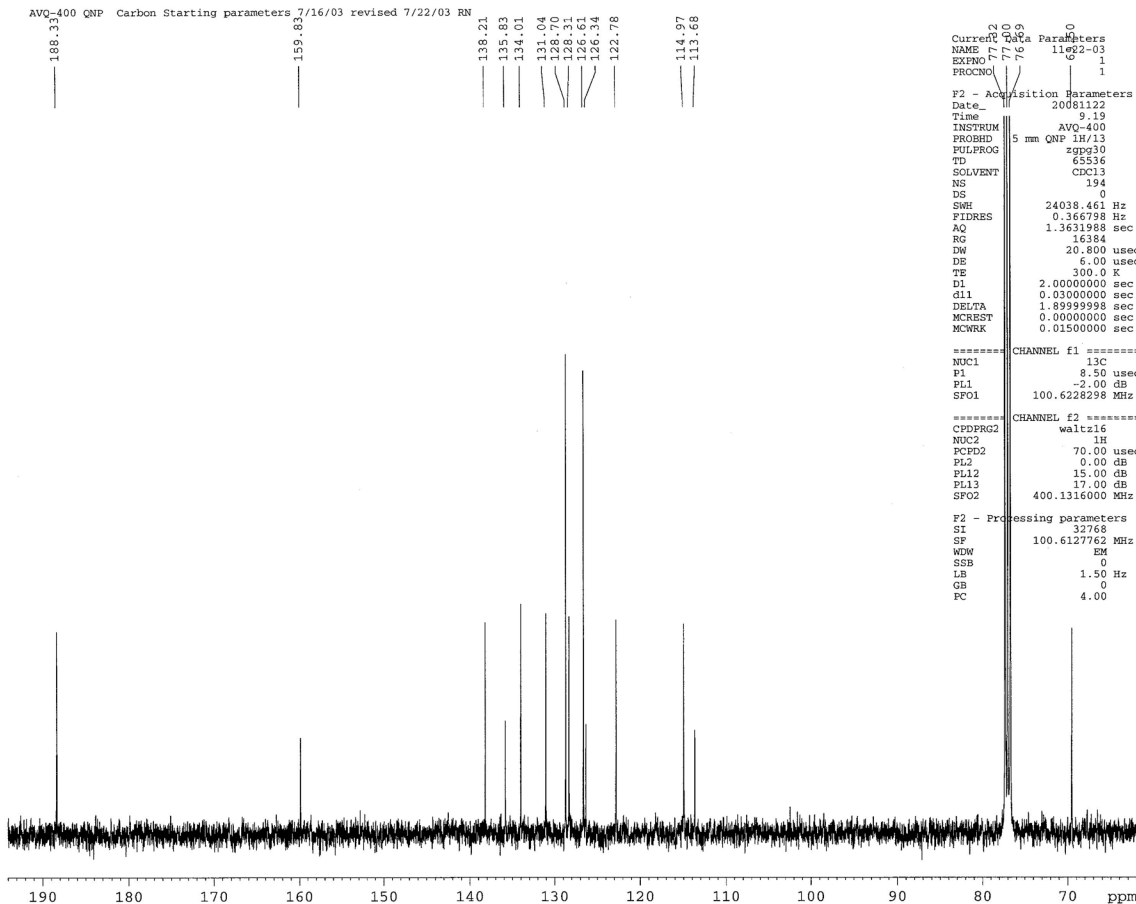
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Time     9.32
INSTRUM  AVQ-400
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PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       0
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       287.4
DW       62.400 usec
DE       6.00 usec
TE       300.0 K
D1       1.00000000 sec
MCREST   0.00000000 sec
MCWRK    0.01500000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       12.80 usec
PL1      0.00 dB
SFO1     400.1324700 MHz

F2 - Processing parameters
SI       65536
SF       400.1300173 MHz
WDW      EM
SSB      0
LB       0.30 Hz

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AVQ-400 QNP Carbon Starting parameters. 7/16/03 revised 7/22/03 RN



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Current Data Parameters
NAME      11-22-03
EXFNO    1
PROCNO   1

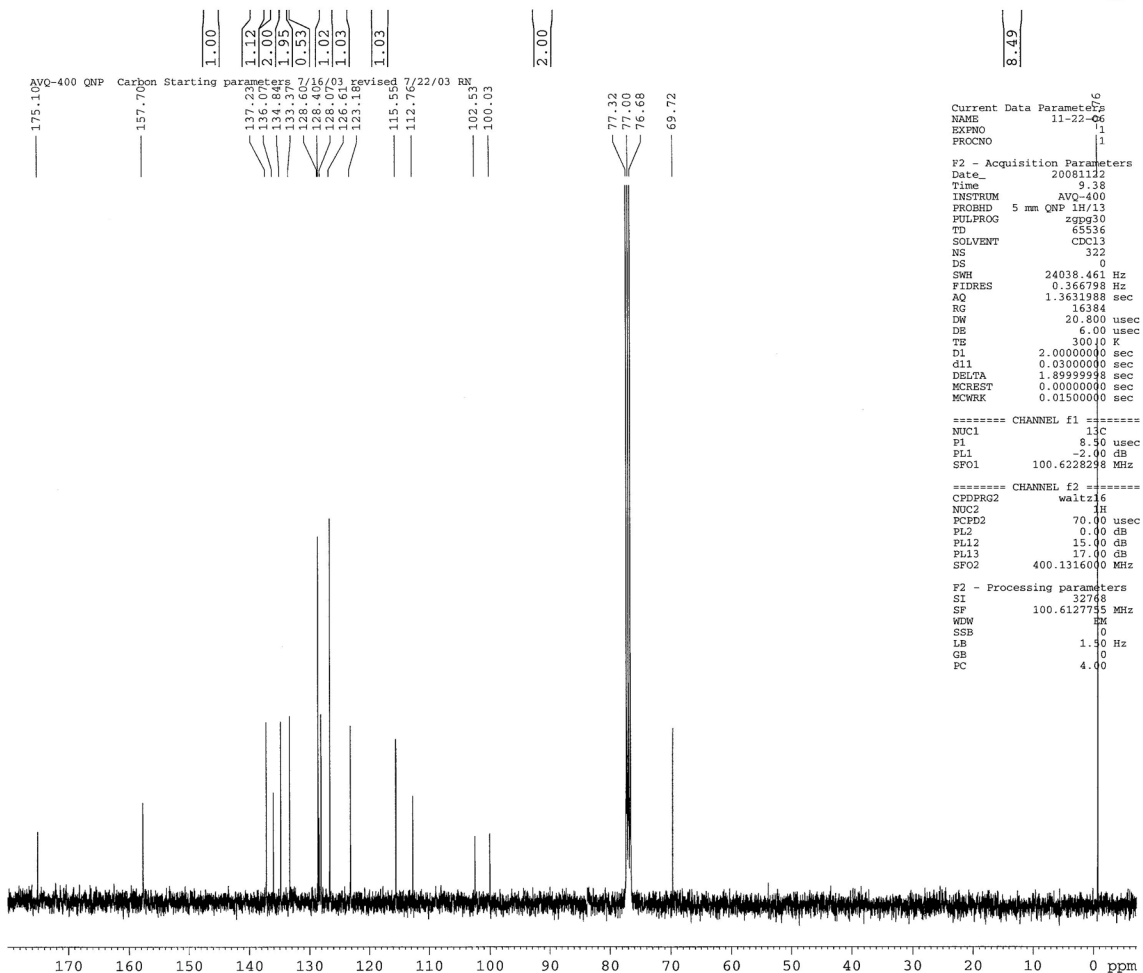
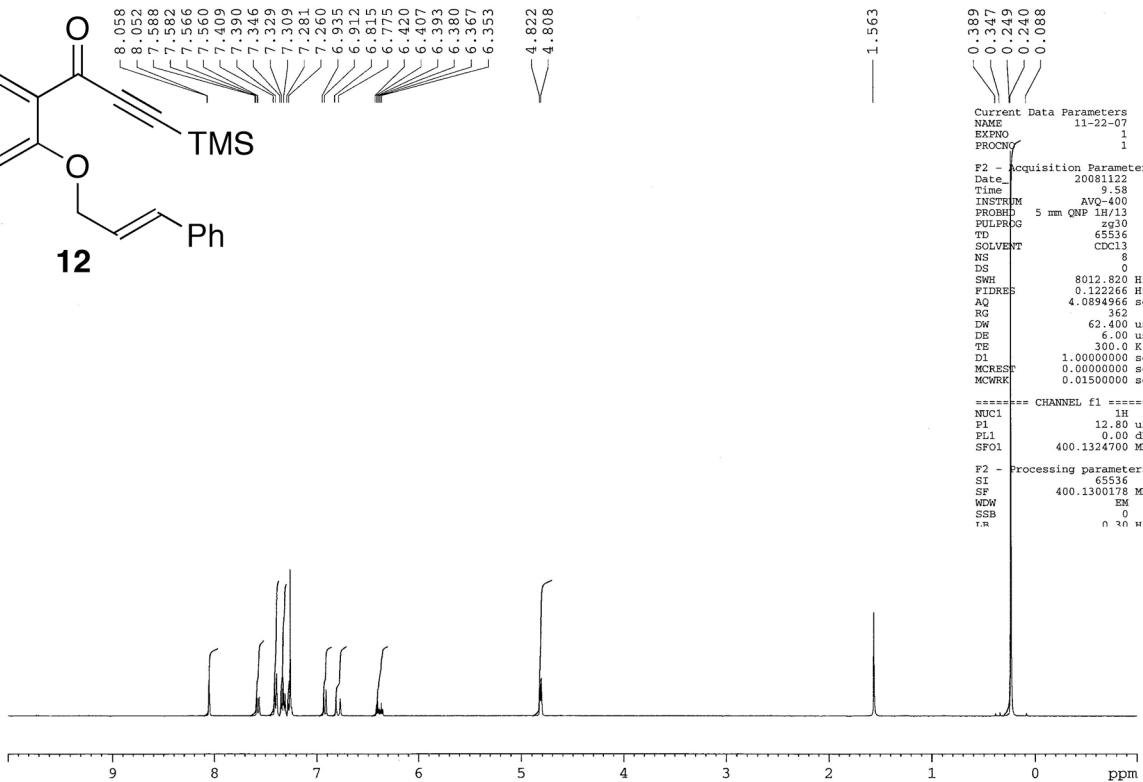
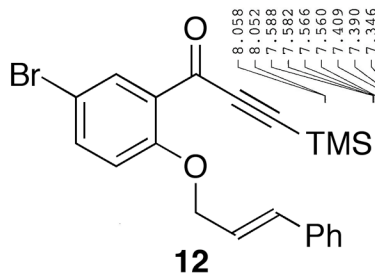
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PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       194
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       16384
DW       20.800 usec
DE       6.00 usec
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d11      0.03000000 sec
DELTA    1.89999998 sec
MCREST   0.00000000 sec
MCWRK    0.01500000 sec

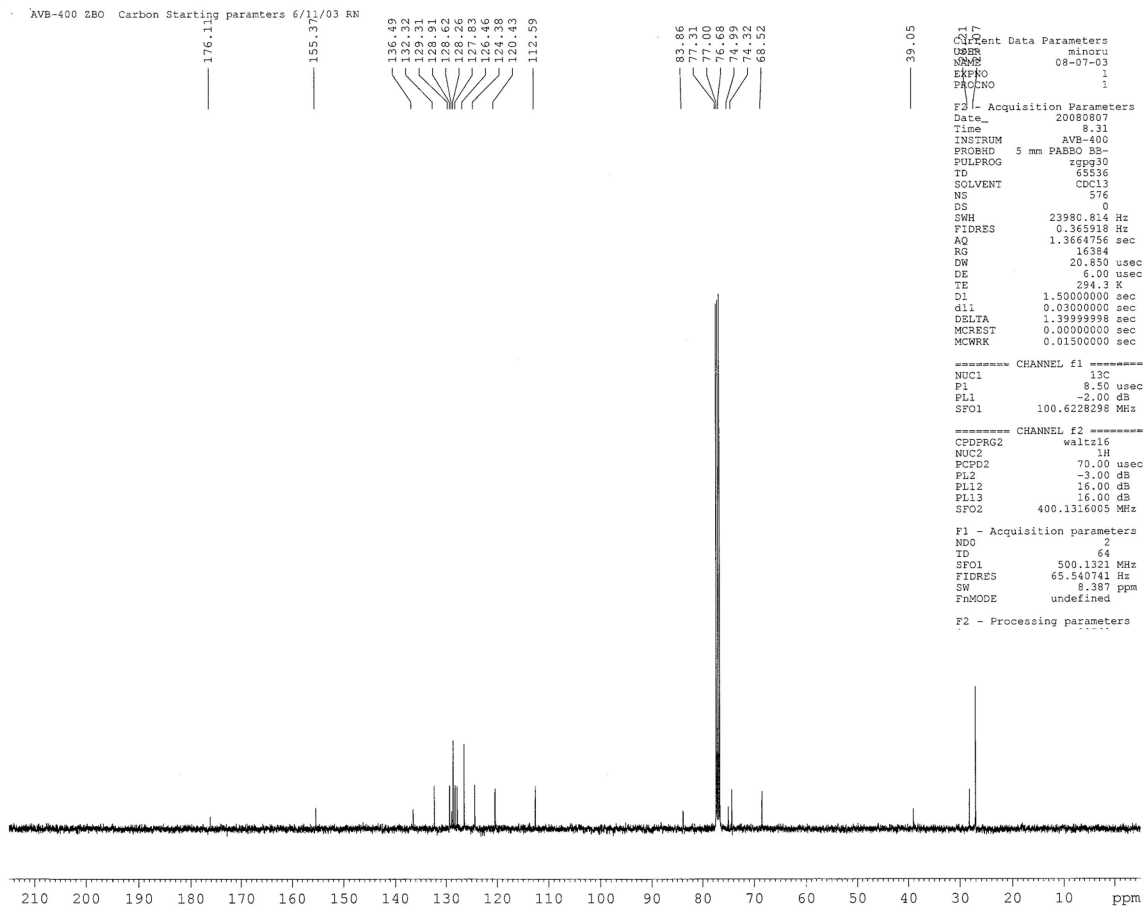
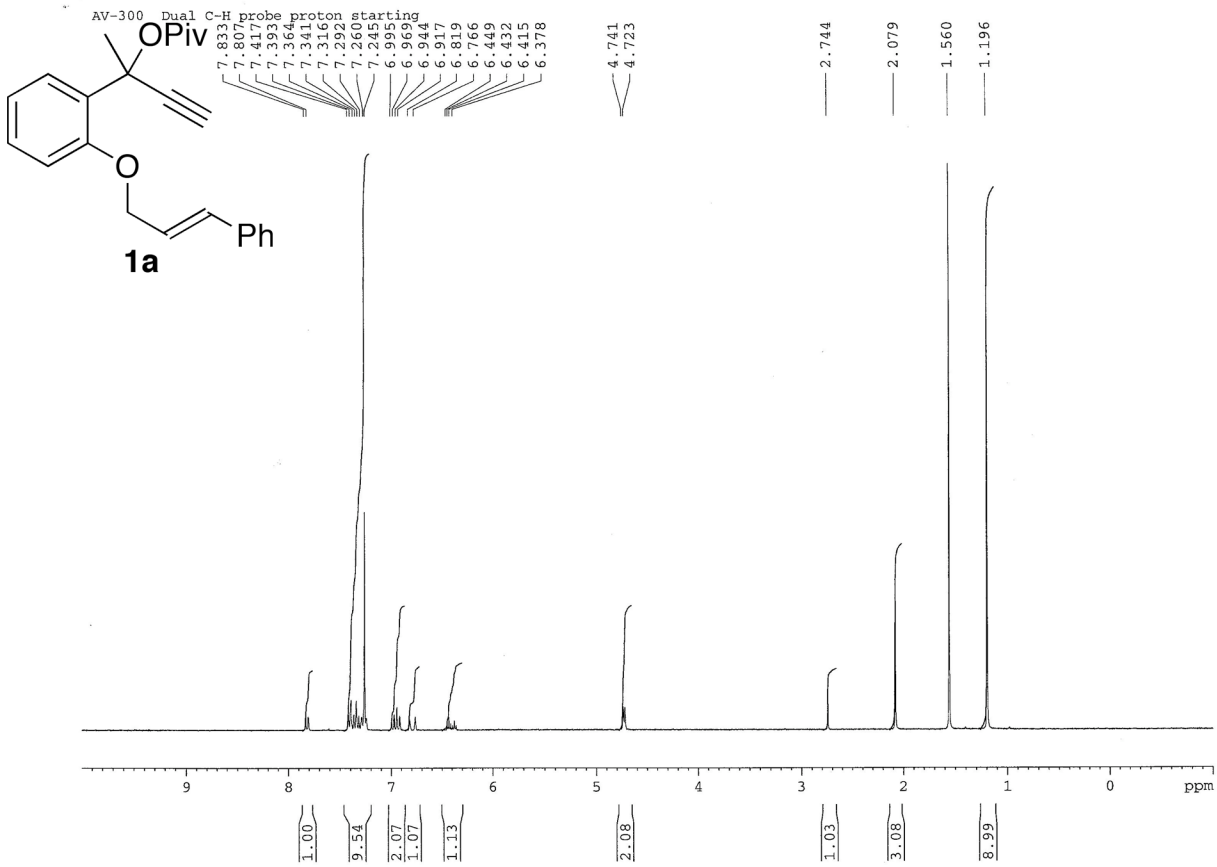
===== CHANNEL f1 =====
NUC1     13C
P1       8.50 usec
PL1      -2.00 dB
SFO1     100.6228298 MHz

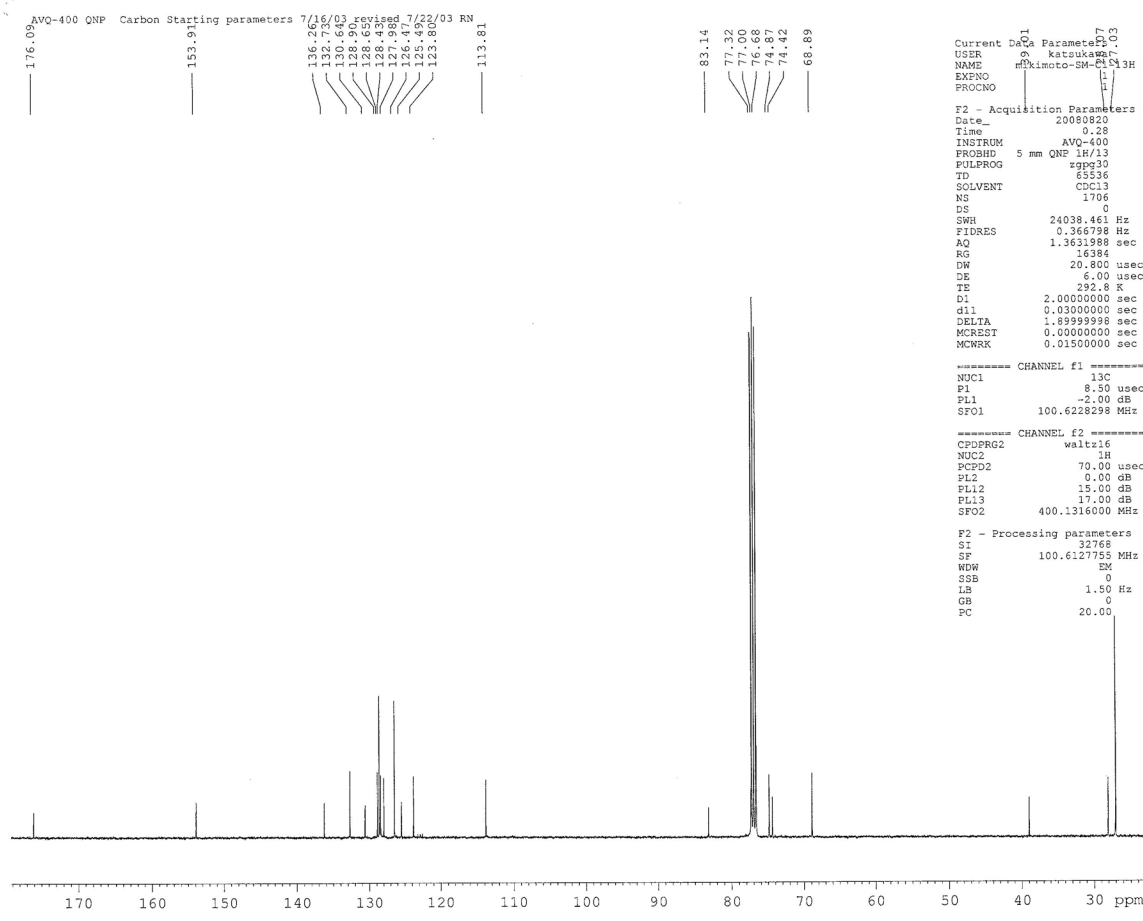
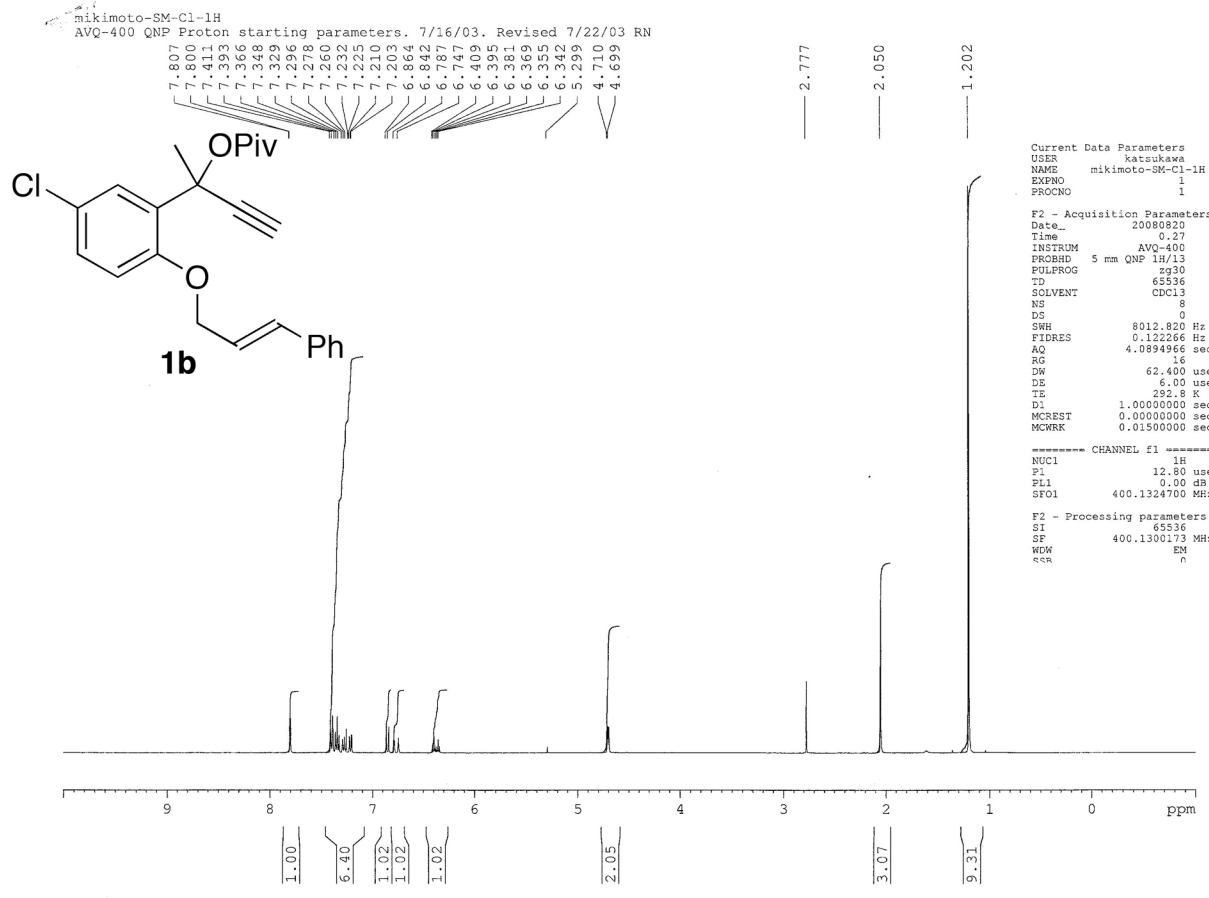
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL2      0.00 dB
PL12     15.00 dB
PL13     17.00 dB
SFO2     400.1316000 MHz

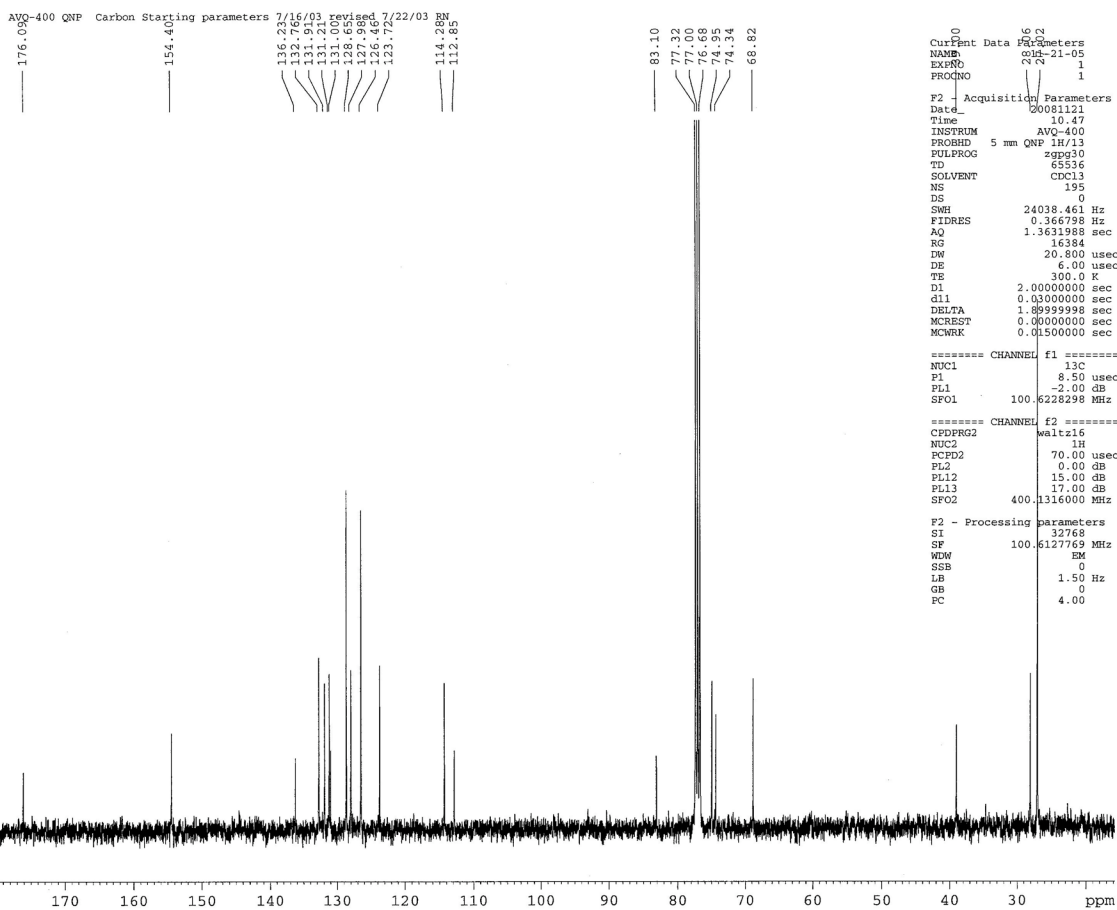
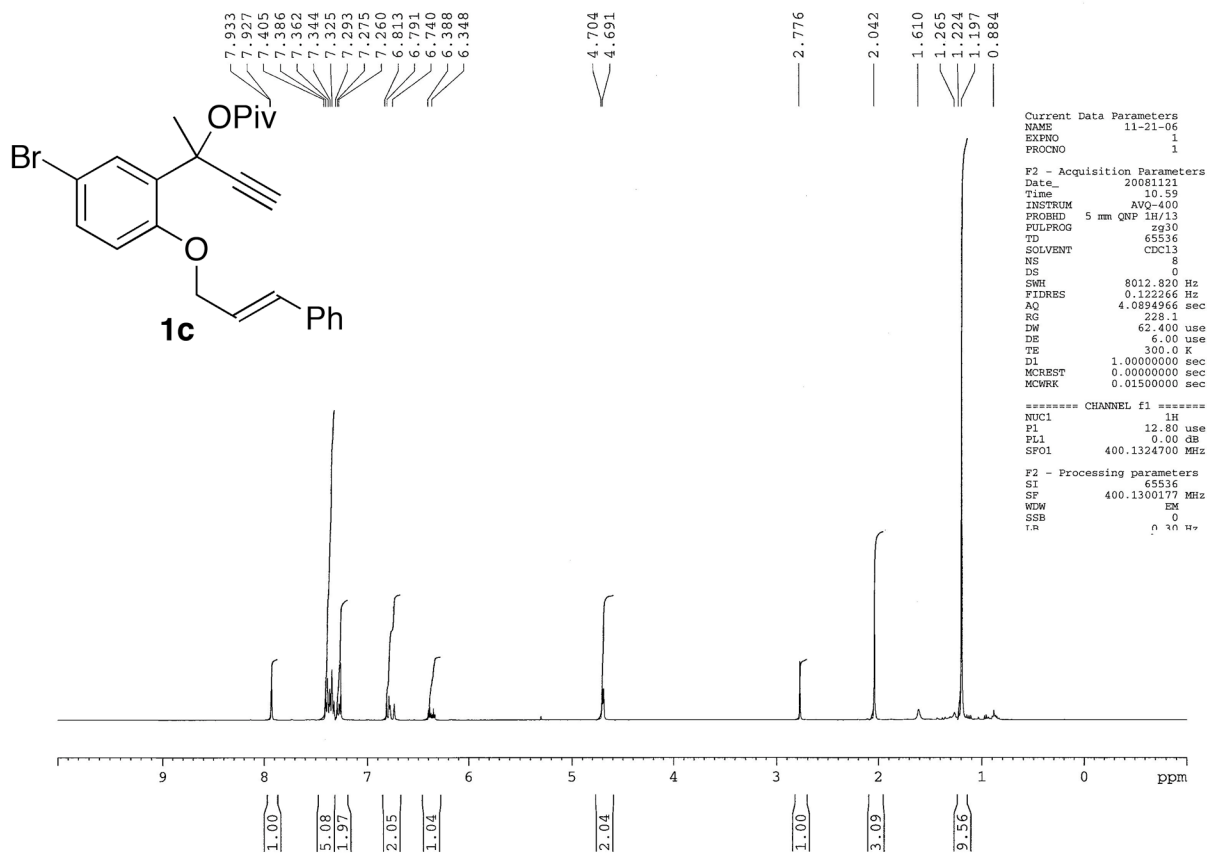
F2 - Processing parameters
SI       32768
SF       100.6127762 MHz
WDW      EM
SSB      0
LB       1.50 Hz
GB       0
FC       4.00

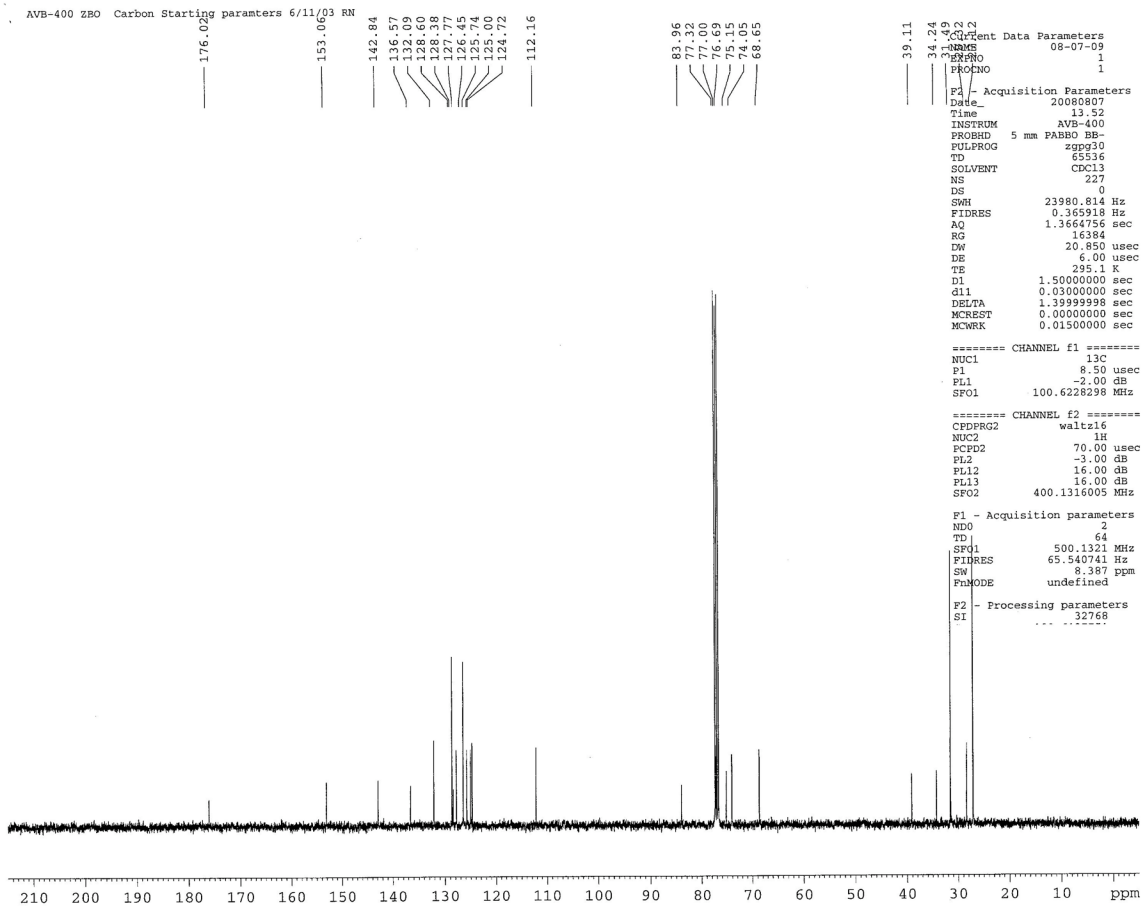
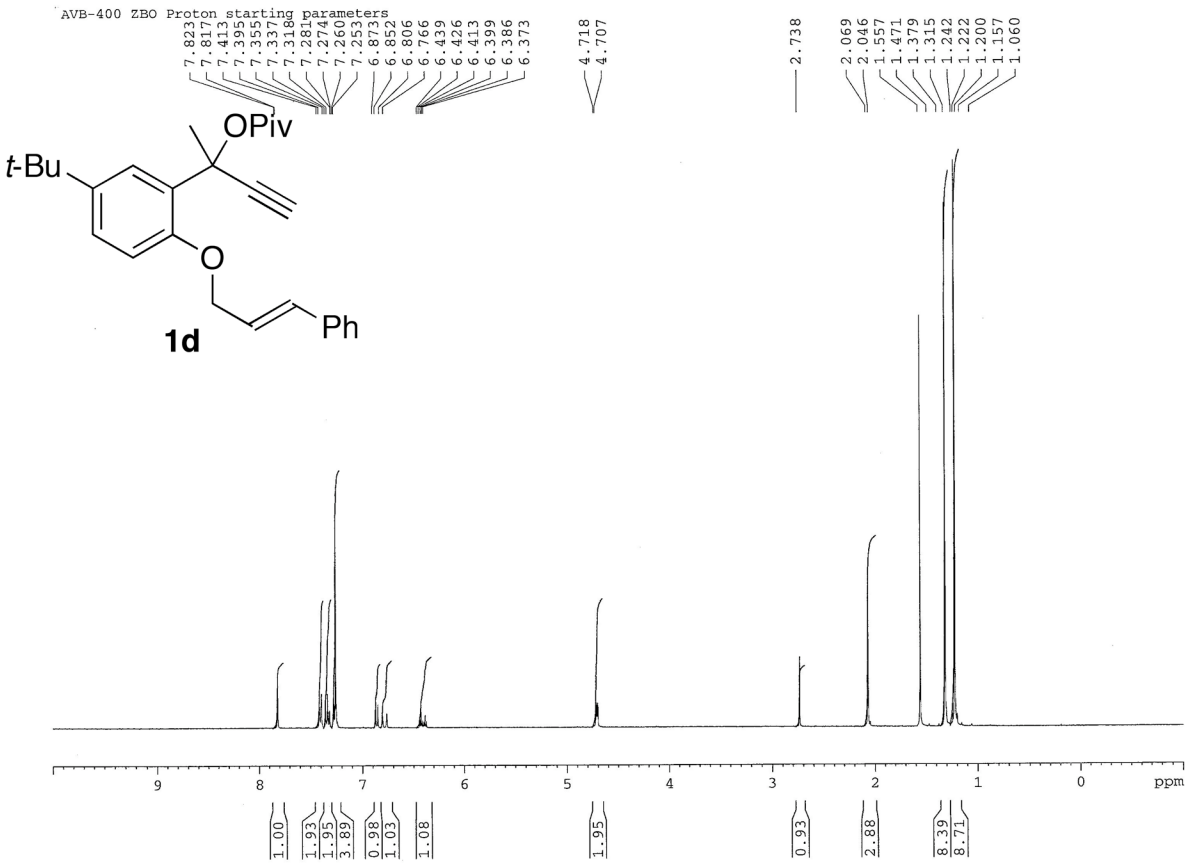
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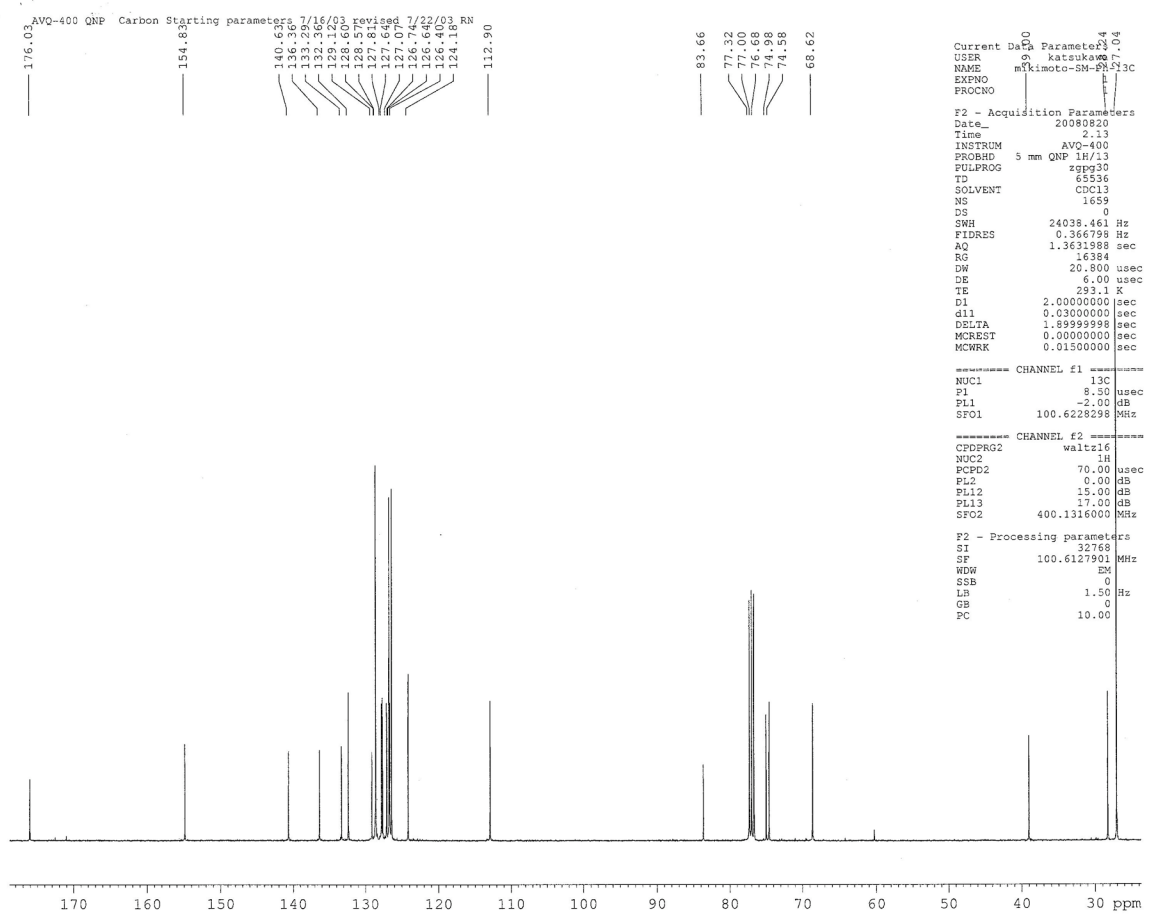
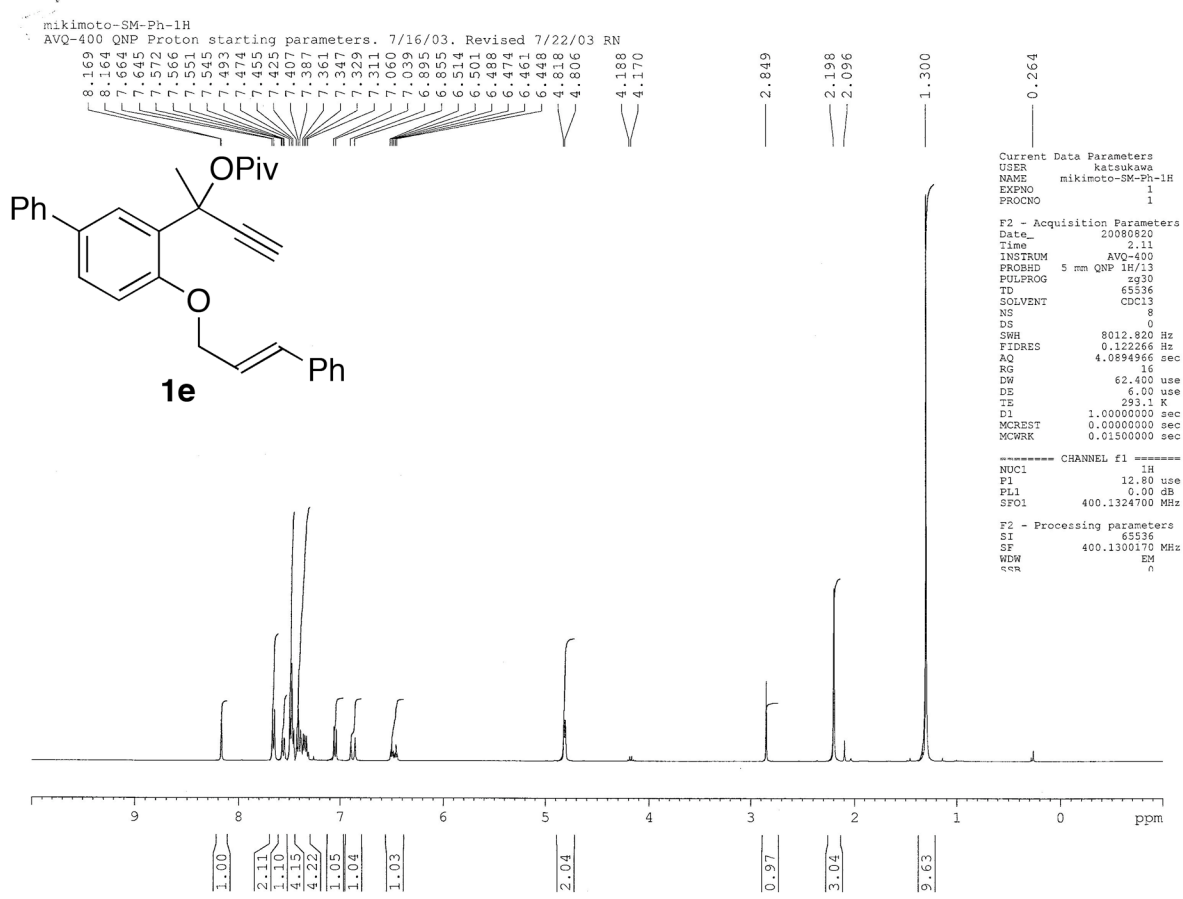



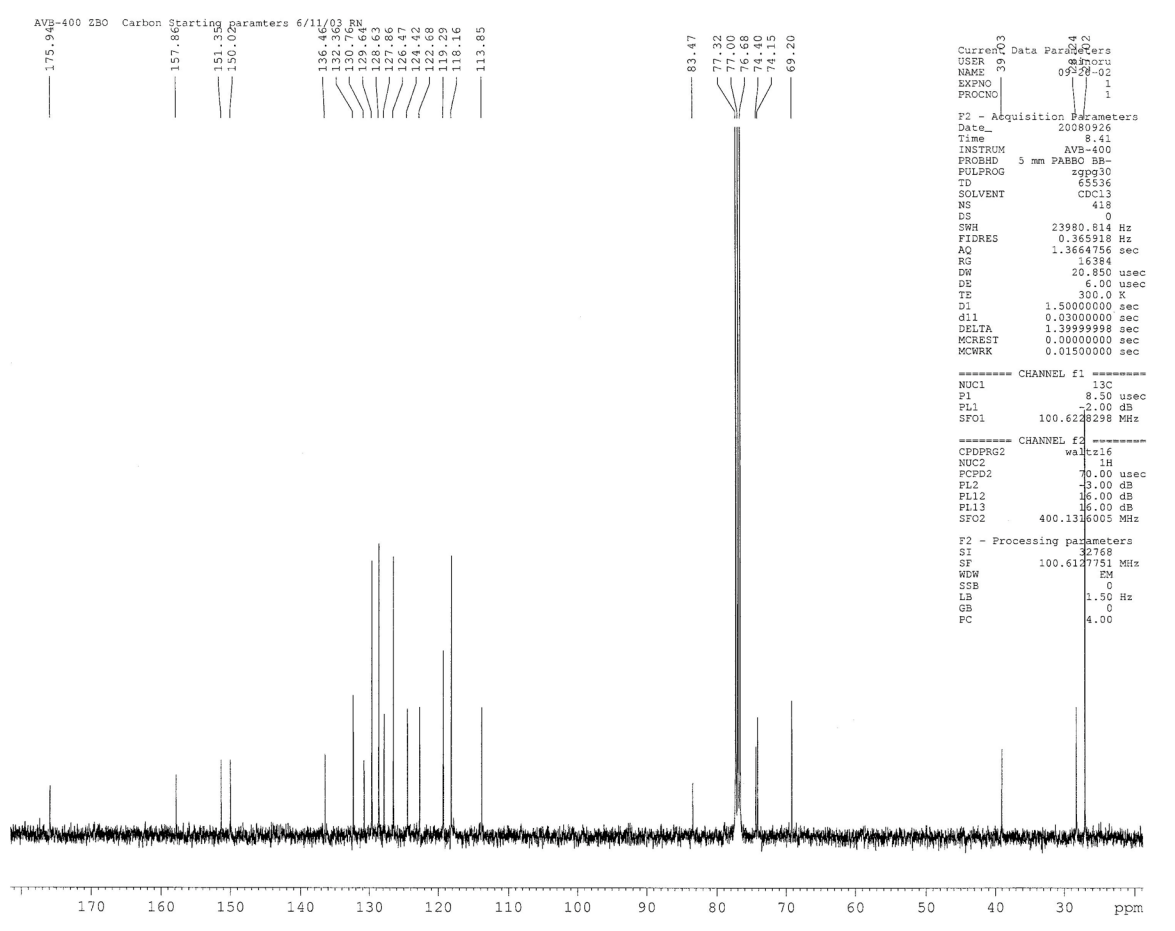
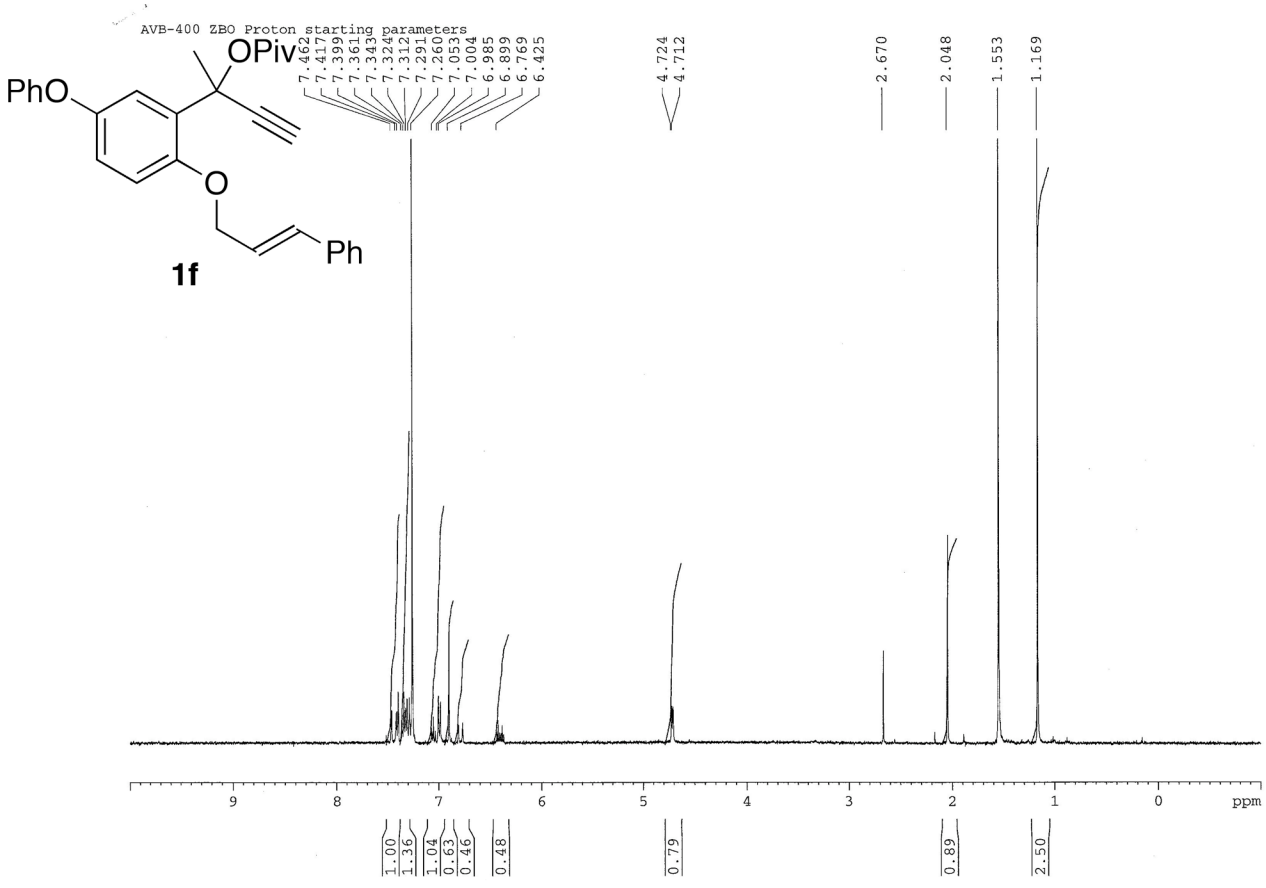




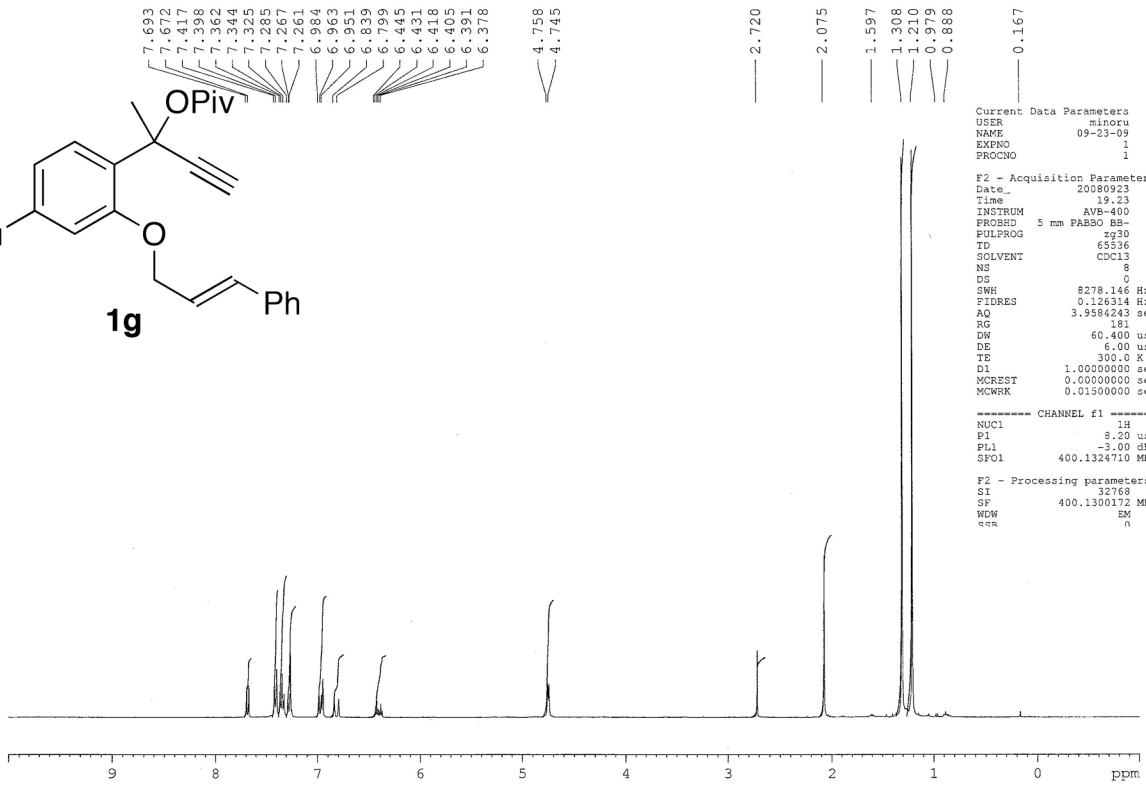
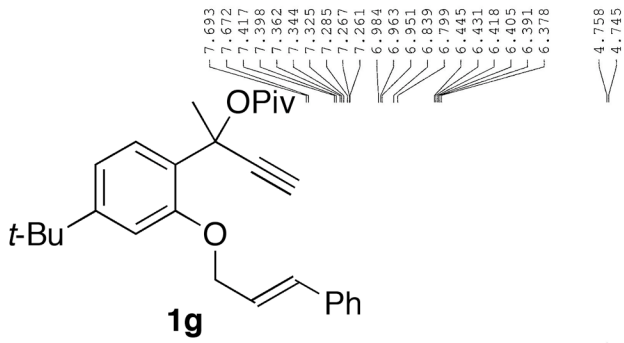




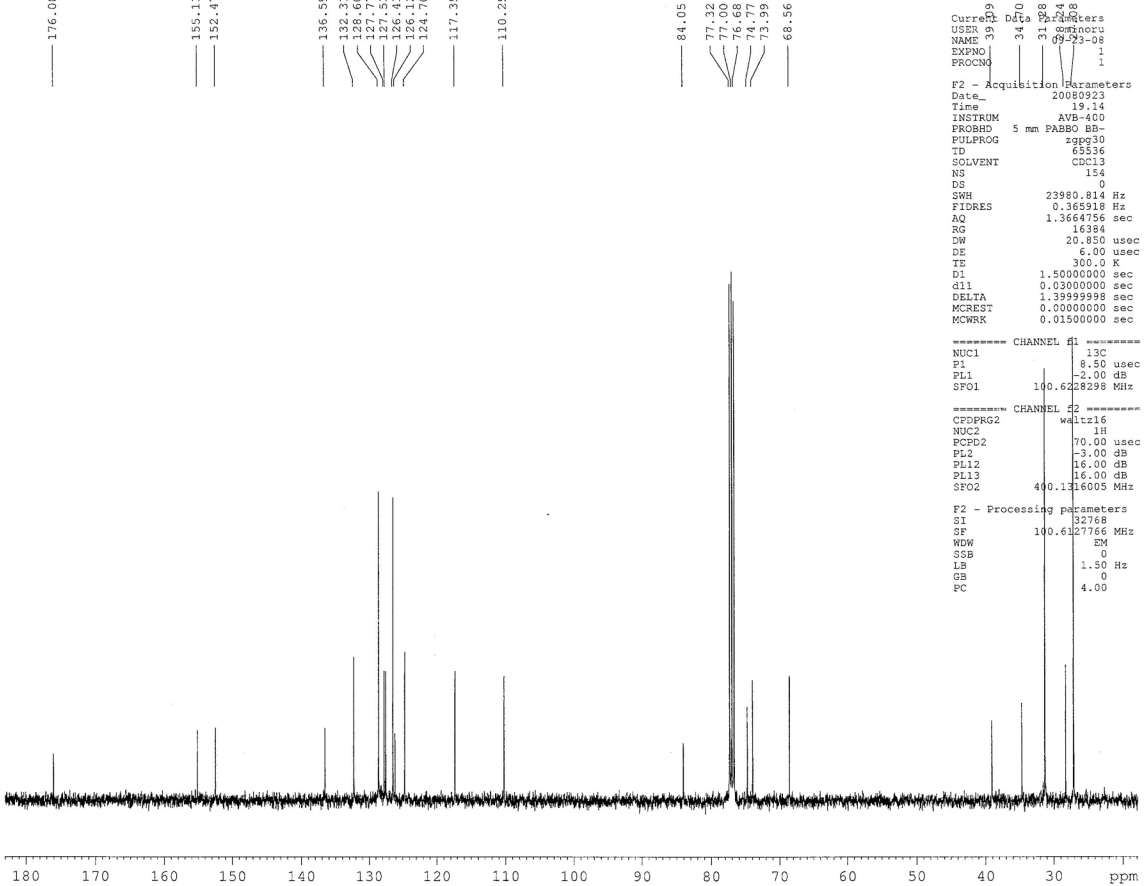


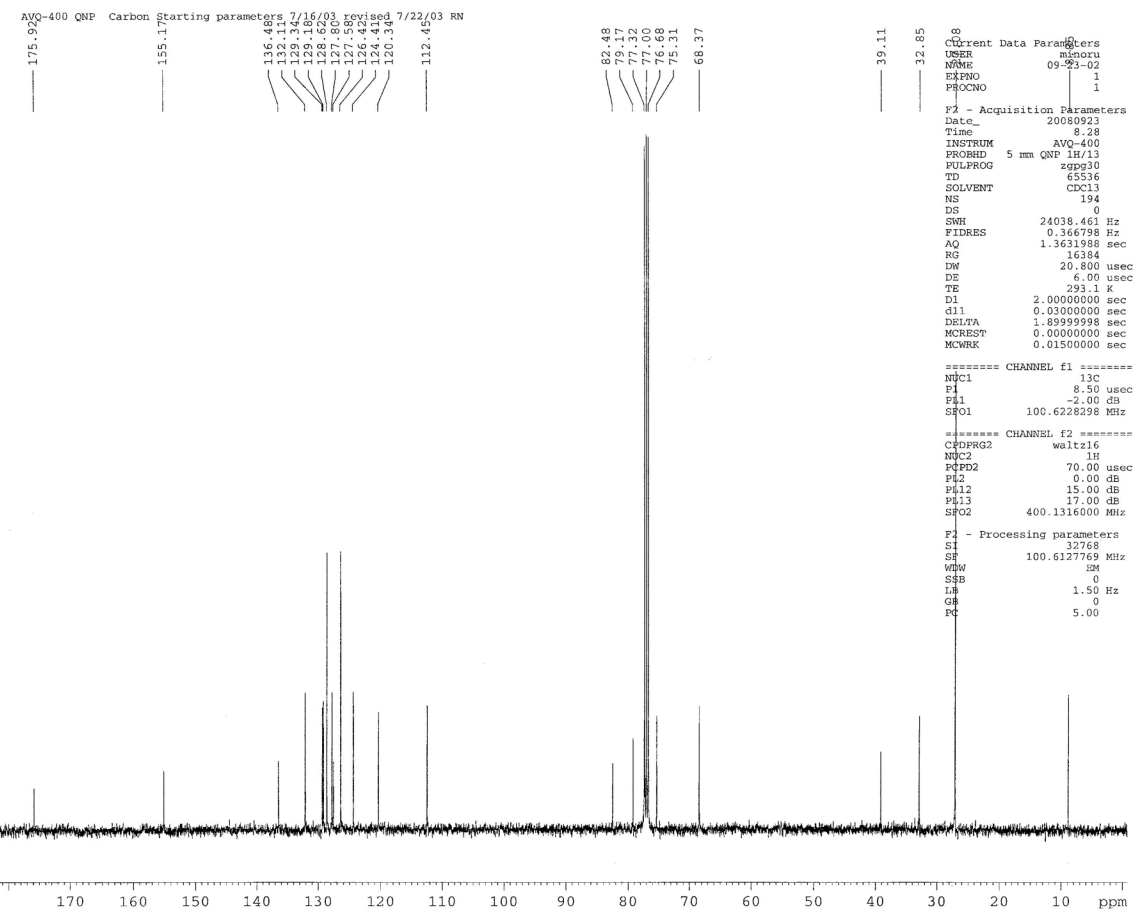
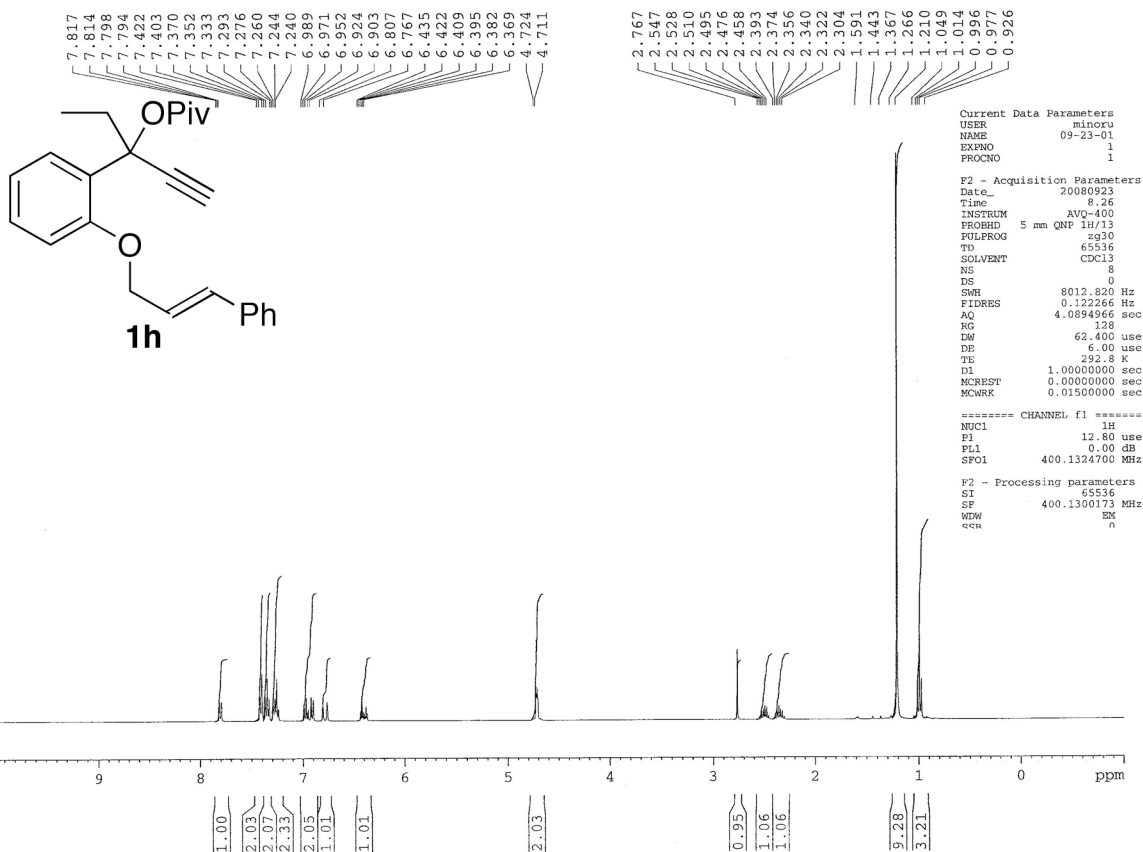


AVB-400 ZBO Proton starting parameters. 6/11/03 RN

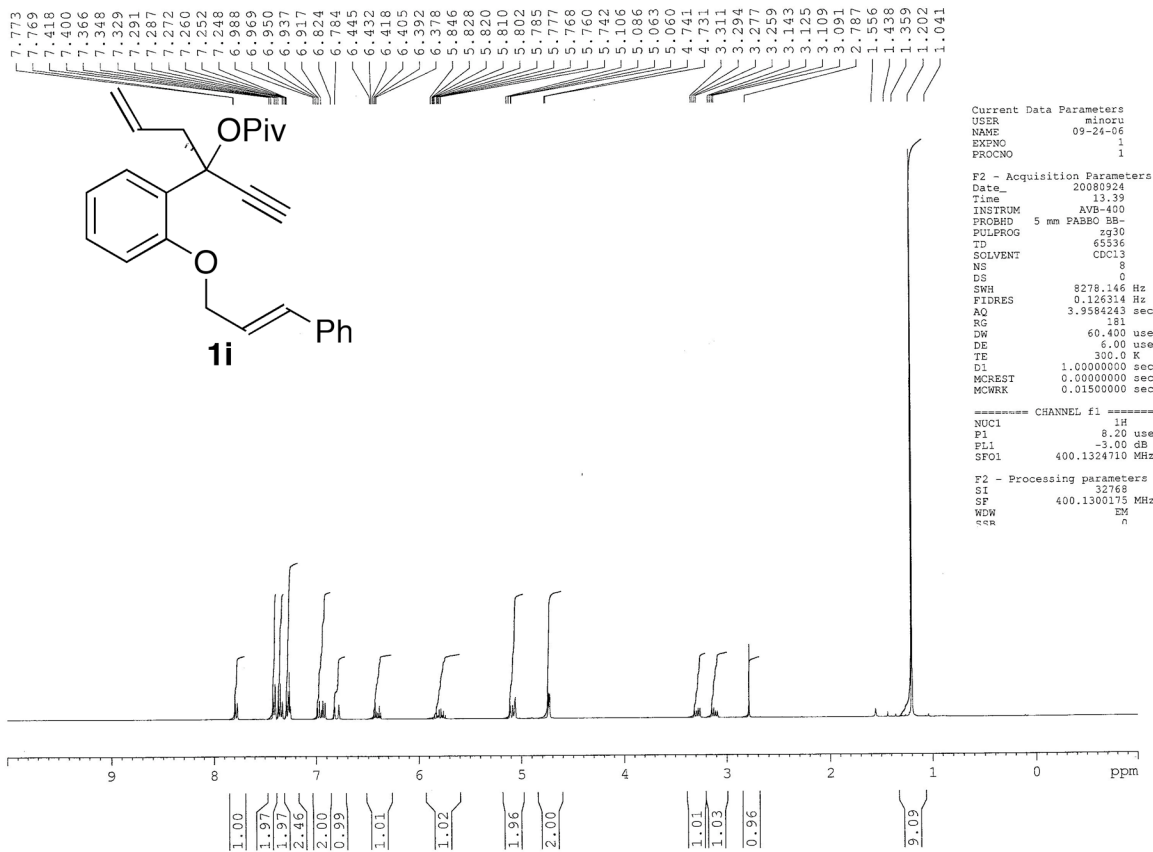


AVB-400 ZBO Carbon Starting parameters 6/11/03 RN





AVB-400 ZBO Proton starting parameters. 6/11/03 RN



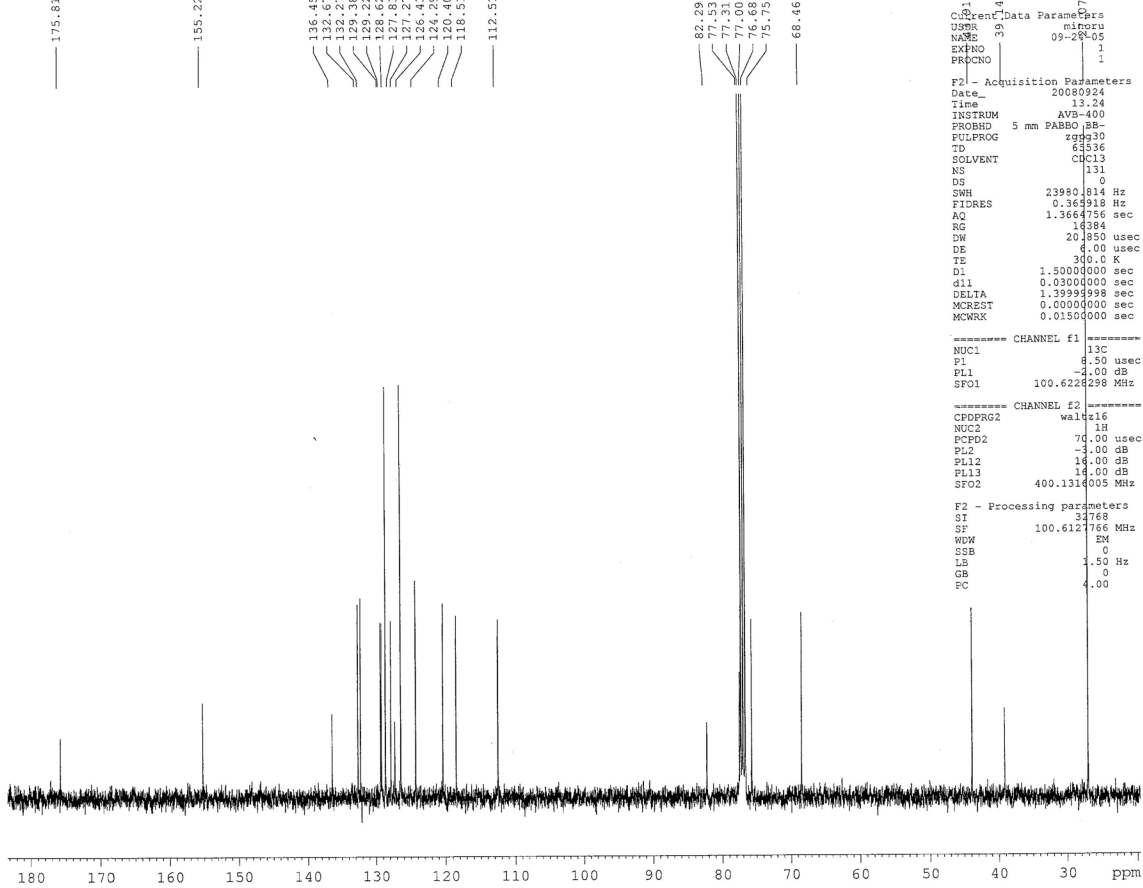
Current Data Parameters
 USER minoru
 NAME 09-24-06
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 13.39
 INSTRUM AVB-400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65336
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.124314 Hz
 AQ 3.9584243 sec
 RG 181
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 8.20 usec
 PL1 -3.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300175 MHz
 WDW EM
 SCA 0

AVB-400 ZBO Carbon Starting parameters 6/11/03 RN



Current Data Parameters
 USER minoru
 NAME 09-24-06
 EXPNO 1
 PROCNO 1

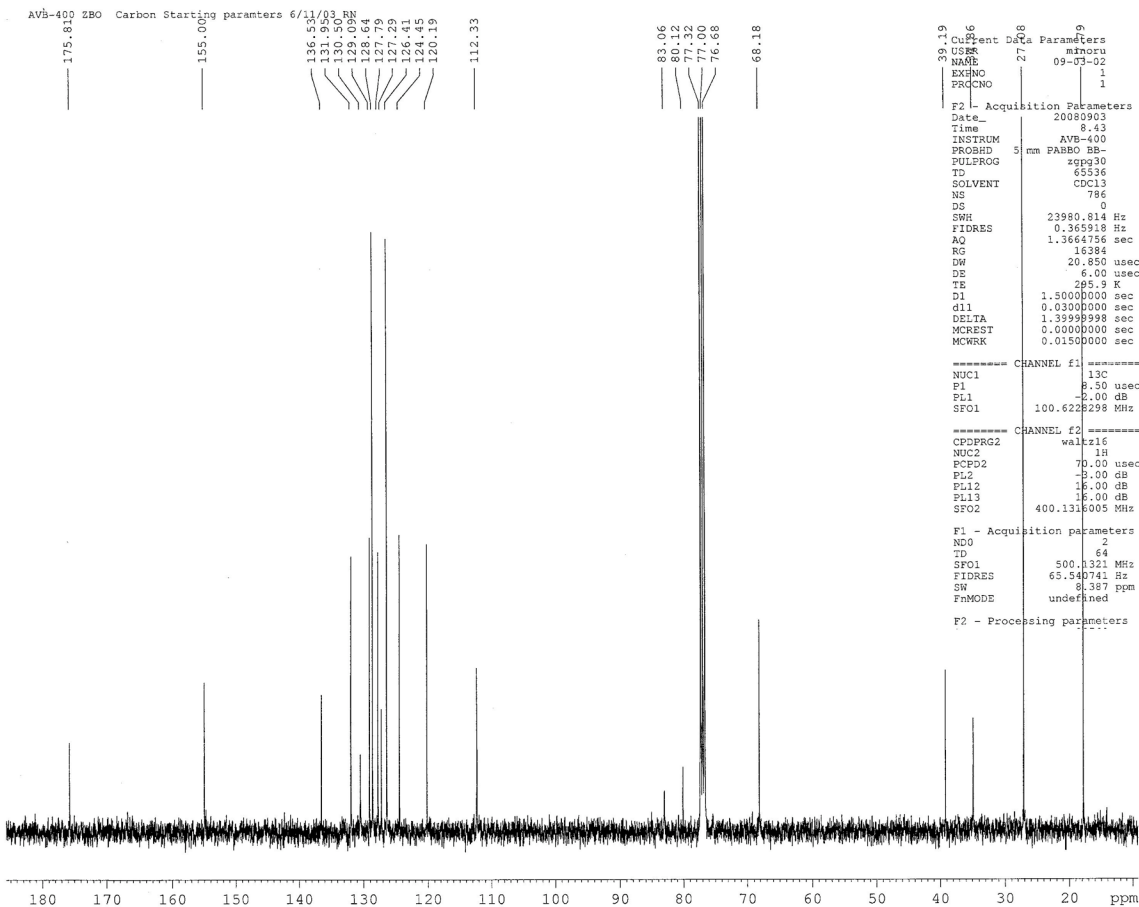
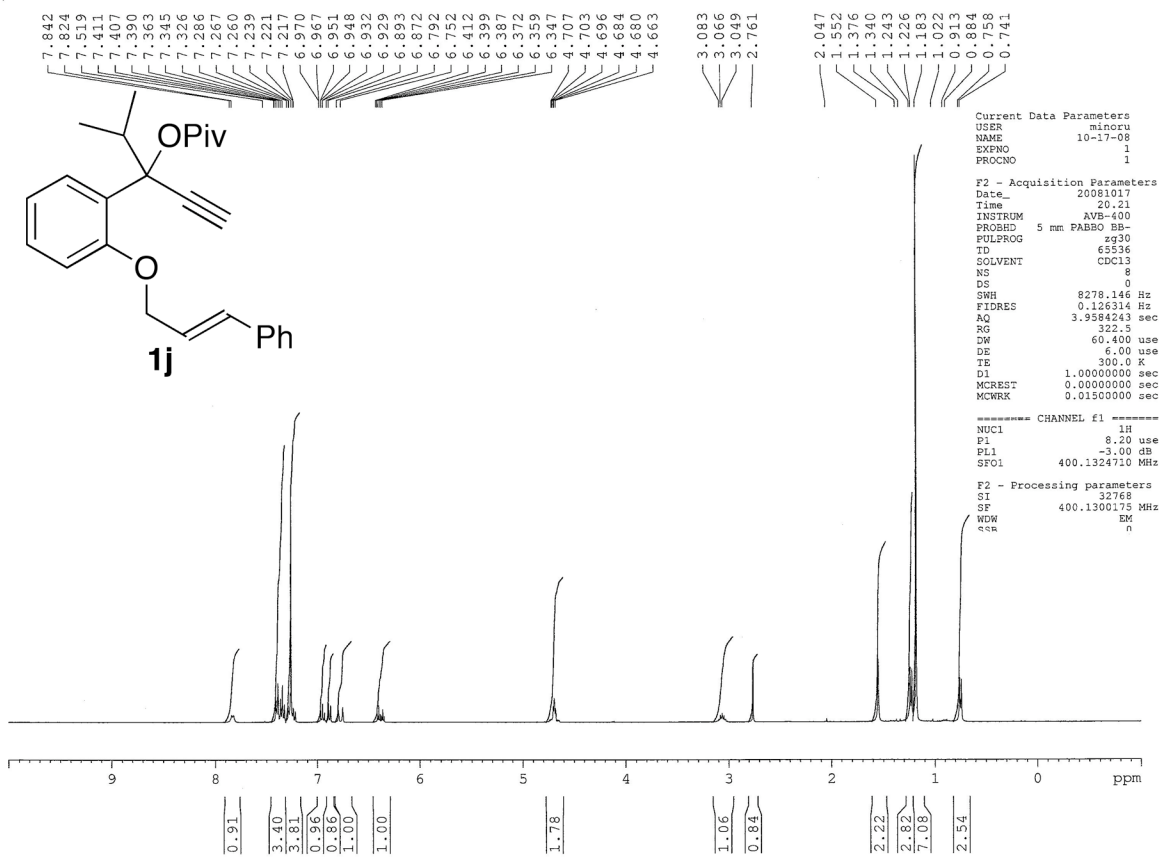
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 Date_ 20080924
 Time 13.24
 INSTRUM AVB-400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65336
 SOLVENT CDCl3
 NS 131
 DS 0
 SWH 23980.814 Hz
 FIDRES 0.364918 Hz
 AQ 1.3664756 sec
 RG 4884
 DW 20.850 usec
 DE 4.00 usec
 TE 300.0 K
 D1 1.5000000 sec
 d11 0.0300000 sec
 DELTA 1.3399998 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.50 usec
 PL1 -3.00 dB
 SF01 100.6228298 MHz

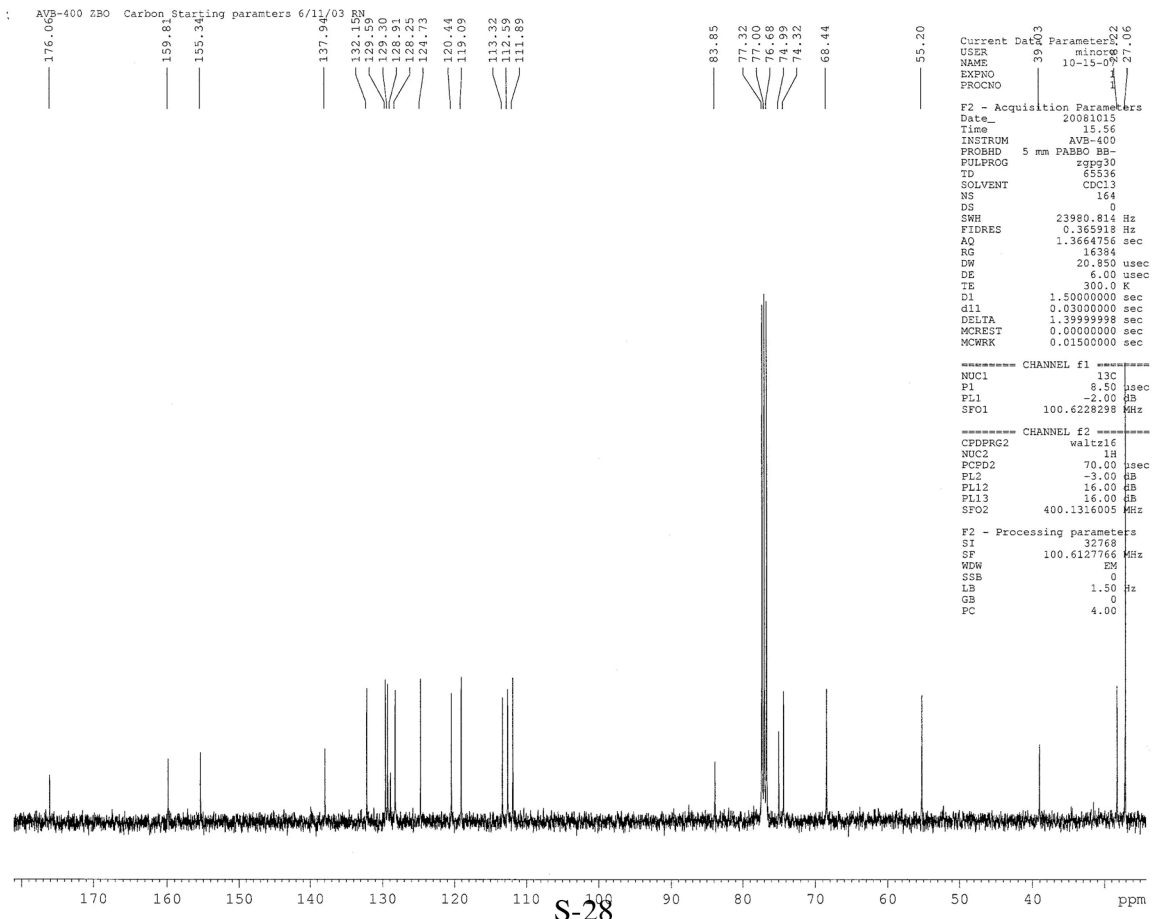
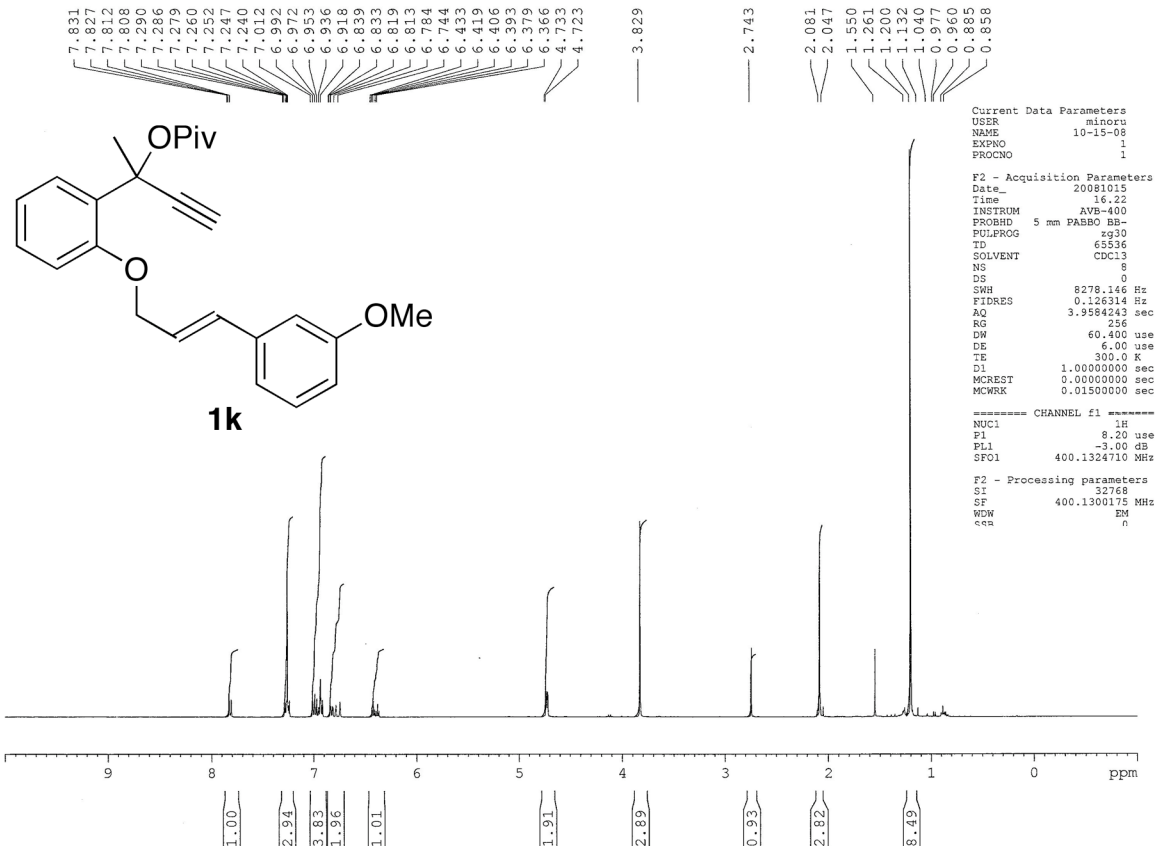
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 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 70.00 usec
 P2 3.00 dB
 PL12 16.00 dB
 PL13 16.00 dB
 SF02 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127766 MHz
 WDW EM
 SSB 0
 LB 1.50 Hz
 GB 0
 FC 4.00

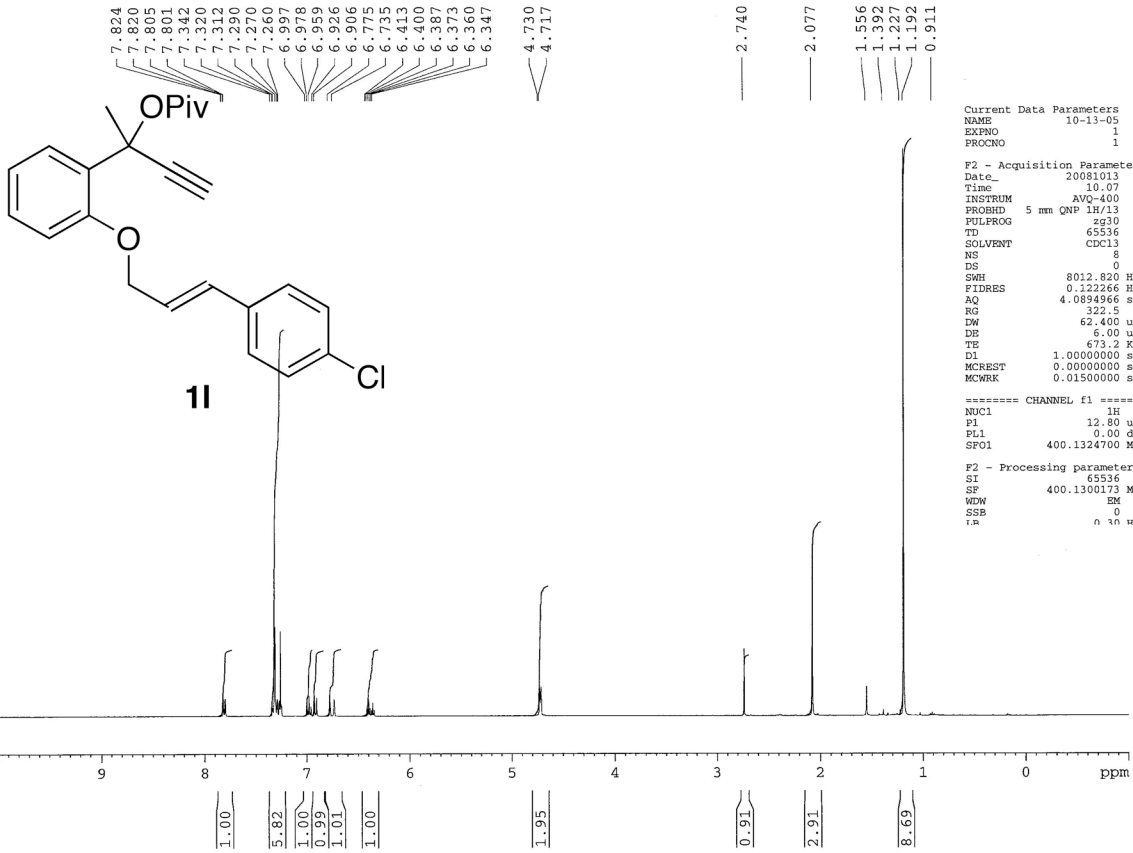
AVD-400 ZBO Proton starting parameters. 6/11/03 RN



AVB-400 ZBO Proton starting parameters. 6/11/03 RN



AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



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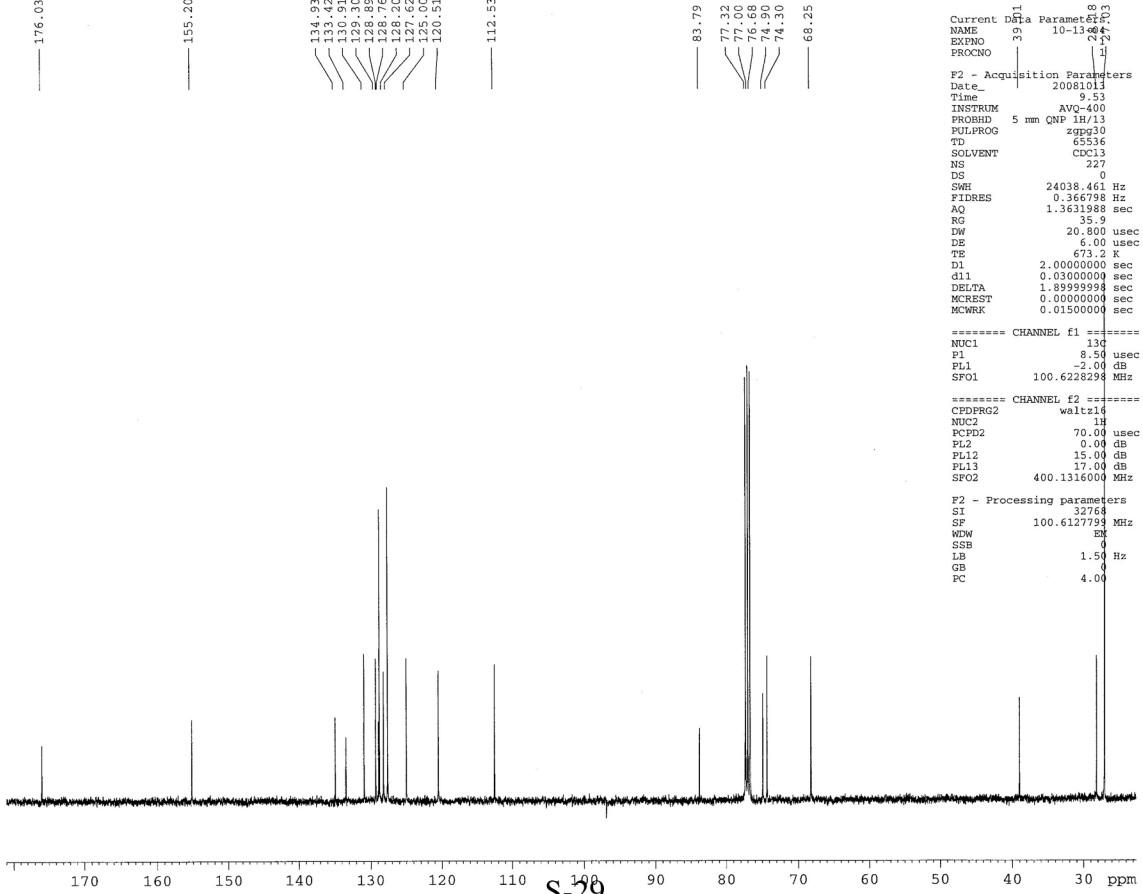
Current Data Parameters
NAME      10-13-05
EXPNO     1
PROCNO    1

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Time      10.07
INSTRUM   AVQ-400
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         322.5
DM         62.400 usec
DE         6.00 usec
TE         673.2 K
D1         1.00000000 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      1H
P1         12.80 usec
PL1        0.00 dB
SFO1      400.1324700 MHz

F2 - Processing parameters
SI         65536
SF         400.1300173 MHz
WDW        EM
SSB        0
LB         0.30 Hz
  
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AVQ-400 QNP Carbon Starting parameters 7/16/03 Revised 7/22/03 RN



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Current Data Parameters
NAME      10-13-05
EXPNO     1
PROCNO    1

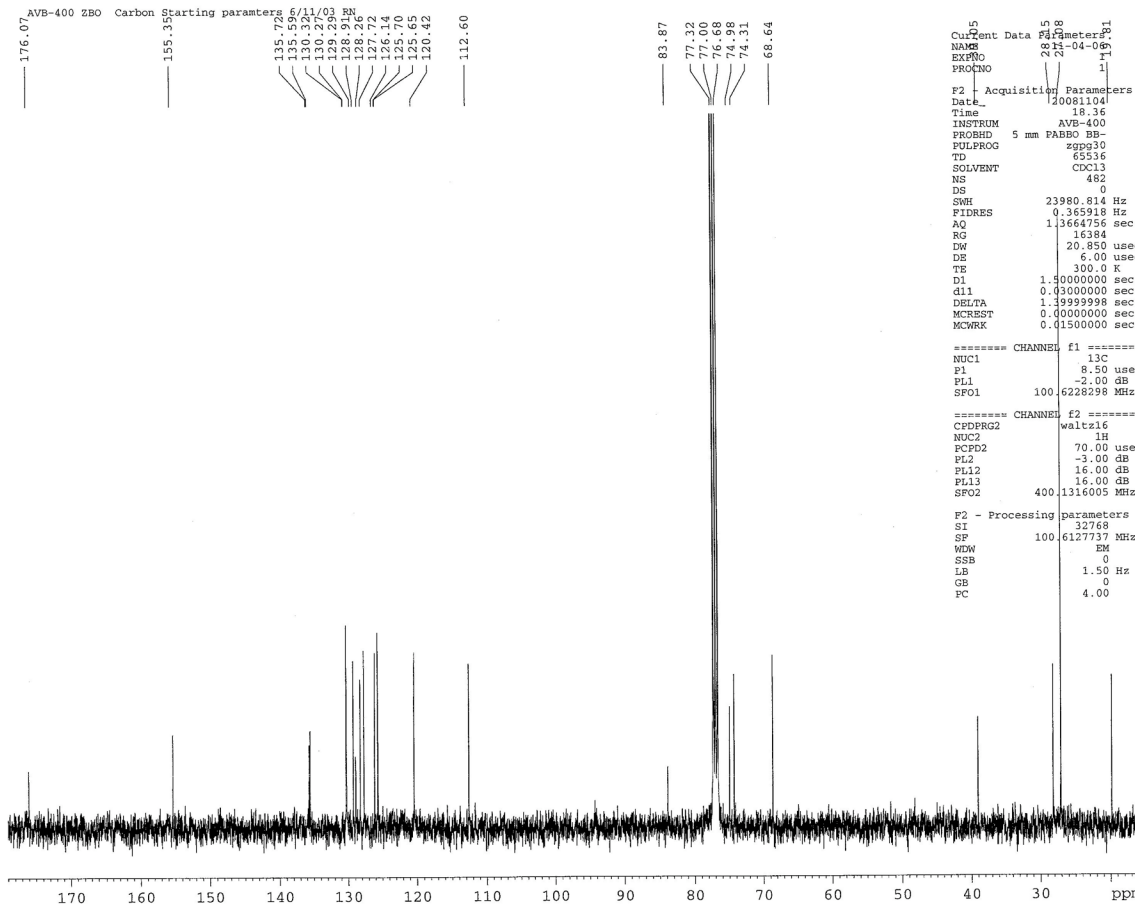
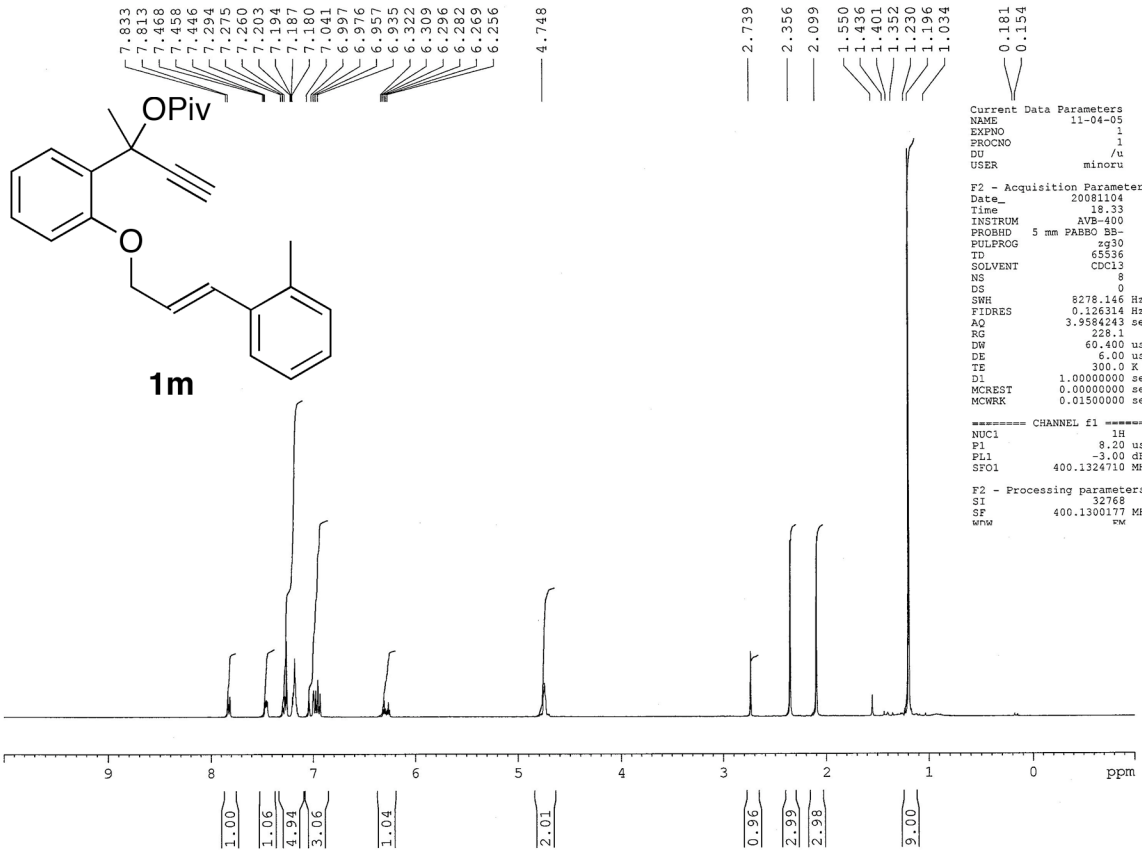
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Time      9.53
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PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         227
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         35.9
DM         20.800 usec
DE         6.00 usec
TE         673.2 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      13C
P1         8.50 usec
PL1        -2.00 dB
SFO1      100.6228298 MHz

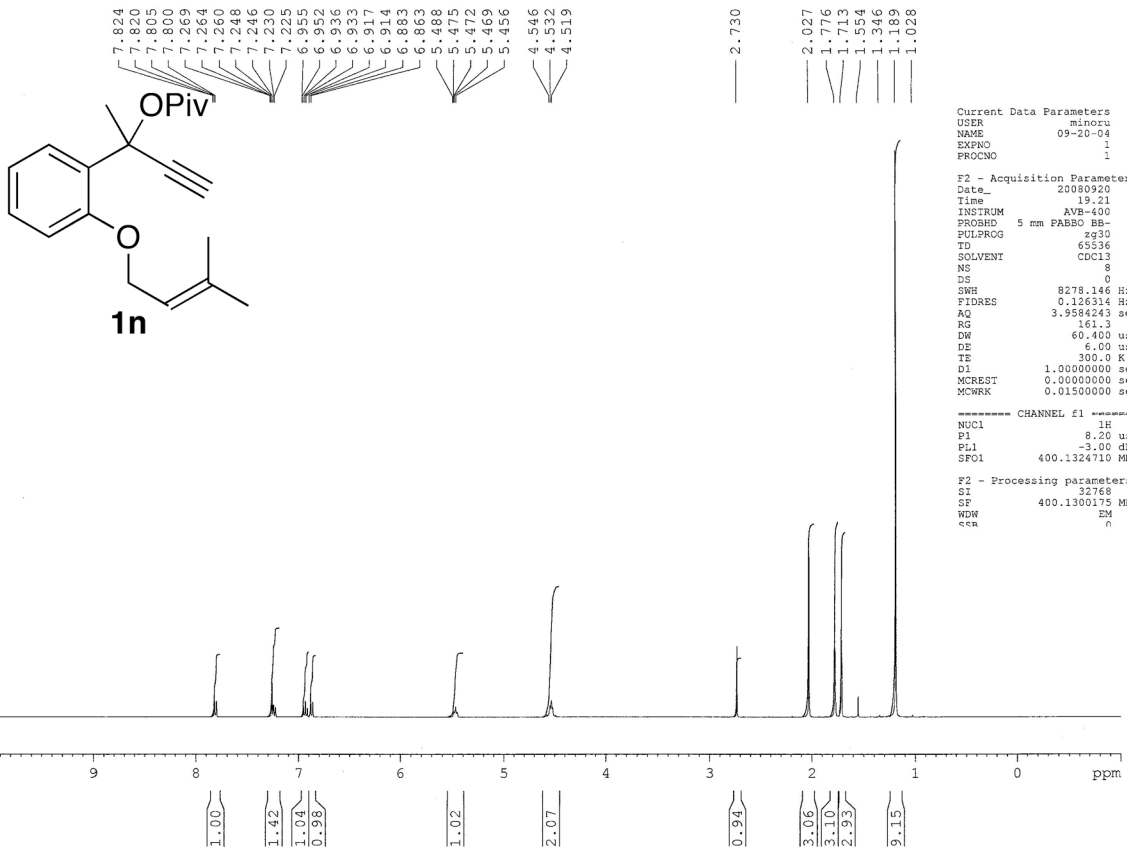
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CPDPRG2   waltz16
NUC2      1H
PCPD2     70.00 usec
PL2        0.00 dB
PL12       15.00 dB
PL13       17.00 dB
SFO2      400.1316000 MHz

F2 - Processing parameters
SI         32768
SF         100.6127793 MHz
WDW        EM
SSB        0
LB         1.50 Hz
GB         0
PC         4.00
  
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AVB-400 ZBO Proton starting parameters. 6/11/03 RN



AVB-400 ZBO Proton starting parameters. 6/11/03 RN



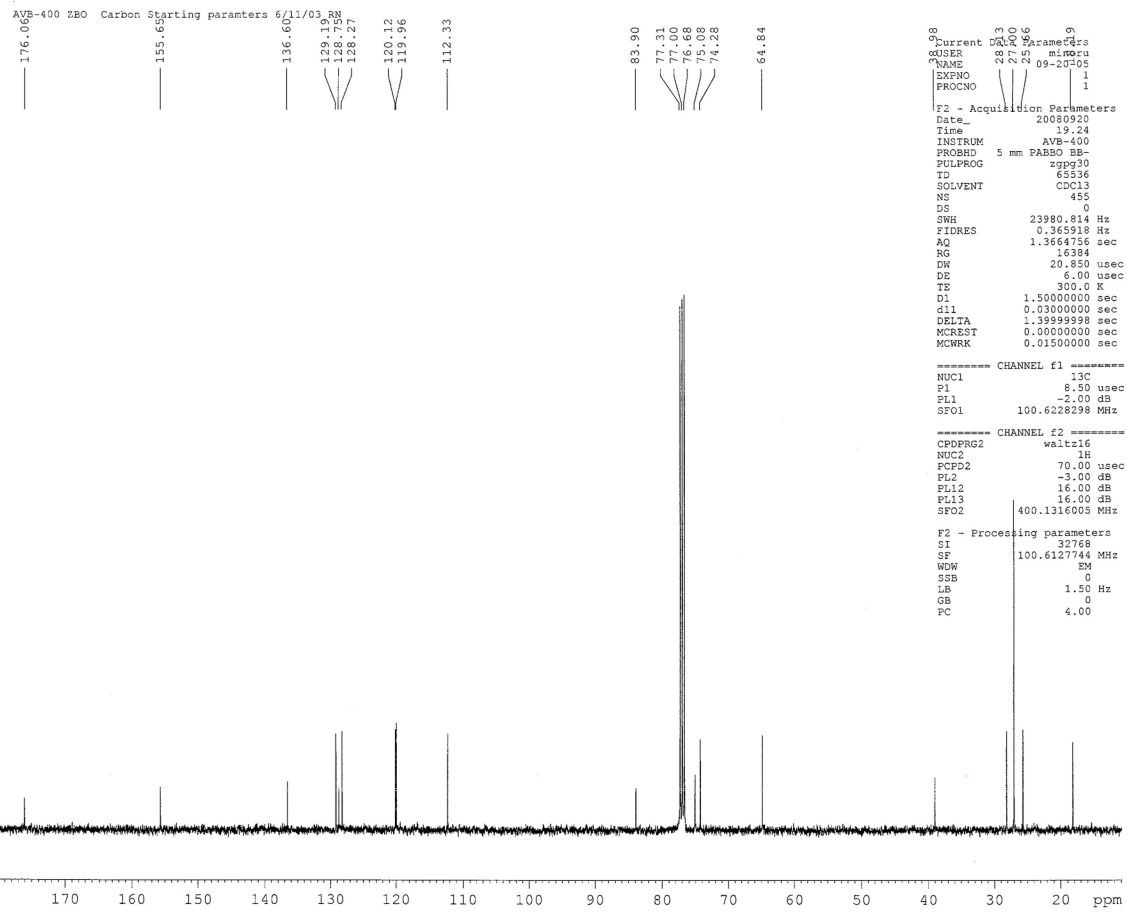
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Current Data Parameters
USER          minoru
NAME          09-20-04
EXPNO        1
PROCNO       1

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Time         19.21
INSTRUM      AVB-400
PROBHD       5 mm PABBO BB-
PULPRG       zgpg30
TD           65536
SOLVENT      CDCl3
NS           0
DS           0
SWH          8278.146 Hz
FIDRES       0.126514 Hz
AQ           3.9584243 sec
RG           161.3
DW           60.400 usec
DE           6.00 usec
TE           300.0 K
D1           1.0000000 sec
MCREST       0.0000000 sec
MCWRK        0.0150000 sec

----- CHANNEL f1 -----
NUC1          1H
P1            8.20 usec
PL1          -3.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI            32768
SF           400.1300175 MHz
WDW           EM
GB            0
PC            4.00
  
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Current Data Parameters
USER          minoru
NAME          09-20-05
EXPNO        1
PROCNO       1

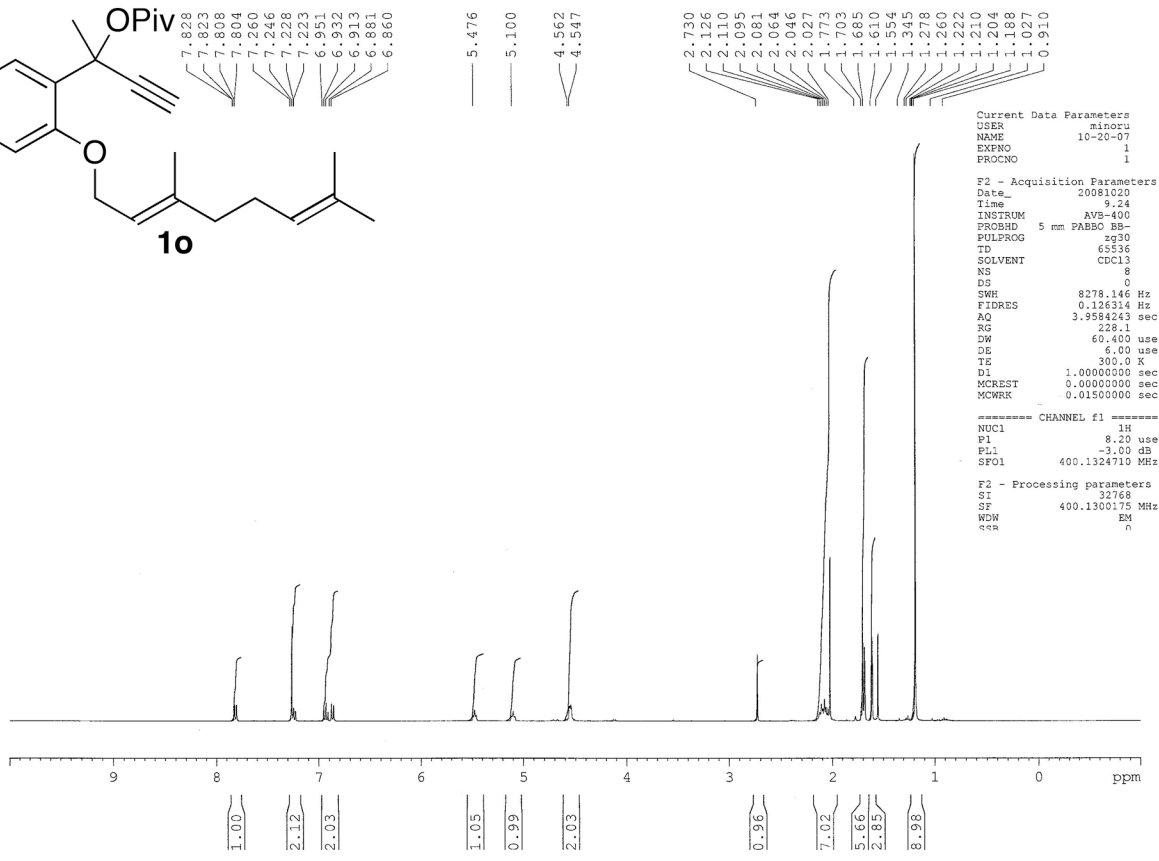
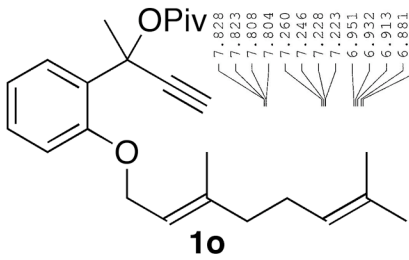
F2 - Acquisition Parameters
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Time         19.24
INSTRUM      AVB-400
PROBHD       5 mm PABBO BB-
PULPRG       zgpg30
TD           65536
SOLVENT      CDCl3
NS           0
DS           0
SWH          23980.814 Hz
FIDRES       0.365918 Hz
AQ           1.3664756 sec
RG           46384
DW           20.850 usec
DE           6.00 usec
TE           300.0 K
D1           1.5000000 sec
d11          0.0300000 sec
DELTA        1.3399998 sec
MCREST       0.0000000 sec
MCWRK        0.0150000 sec

----- CHANNEL f1 -----
NUC1          13C
P1            8.50 usec
PL1          -2.00 dB
SFO1         100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2      waltz16
NUC2          1H
PCPD2        70.00 usec
PL2          -3.00 dB
PL12         16.00 dB
PL13         16.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI            32768
SF           100.6127744 MHz
WDW           EM
GB            0
LB           1.50 Hz
GB            0
PC            4.00
  
```

AVB-400 ZBO Proton starting parameters. 6/11/03 RN



```

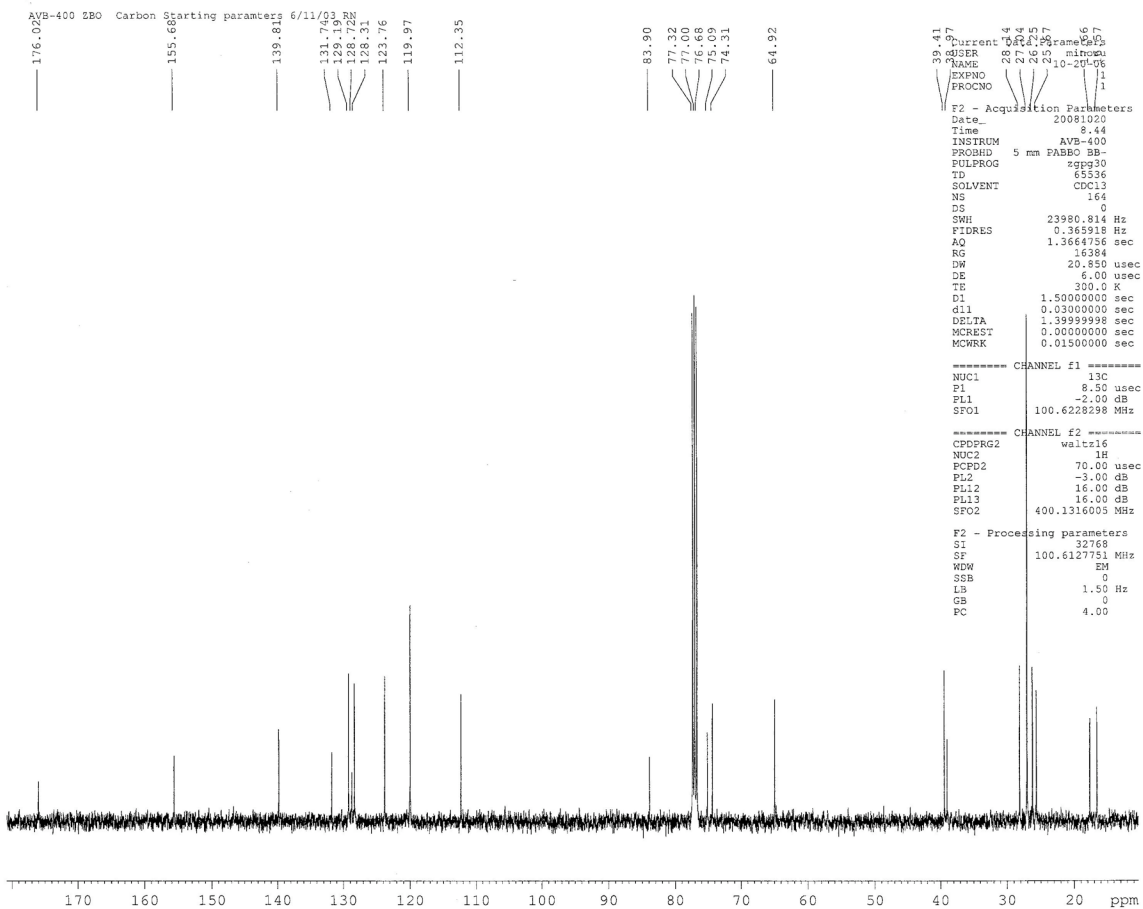
Current Data Parameters
USER      minoru
NAME      10-20-07
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20081020
Time      9.24
INSTRUM   AVB-400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         228.1
DW         60.400 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

----- CHANNEL f1 -----
NUC1      1H
P1        8.20 usec
PL1       -3.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI        32768
SF        400.1300175 MHz
WDW       EM
GB        0
PC        4.00
    
```

AVB-400 ZBO Carbon Starting parameters. 6/11/03 RN



```

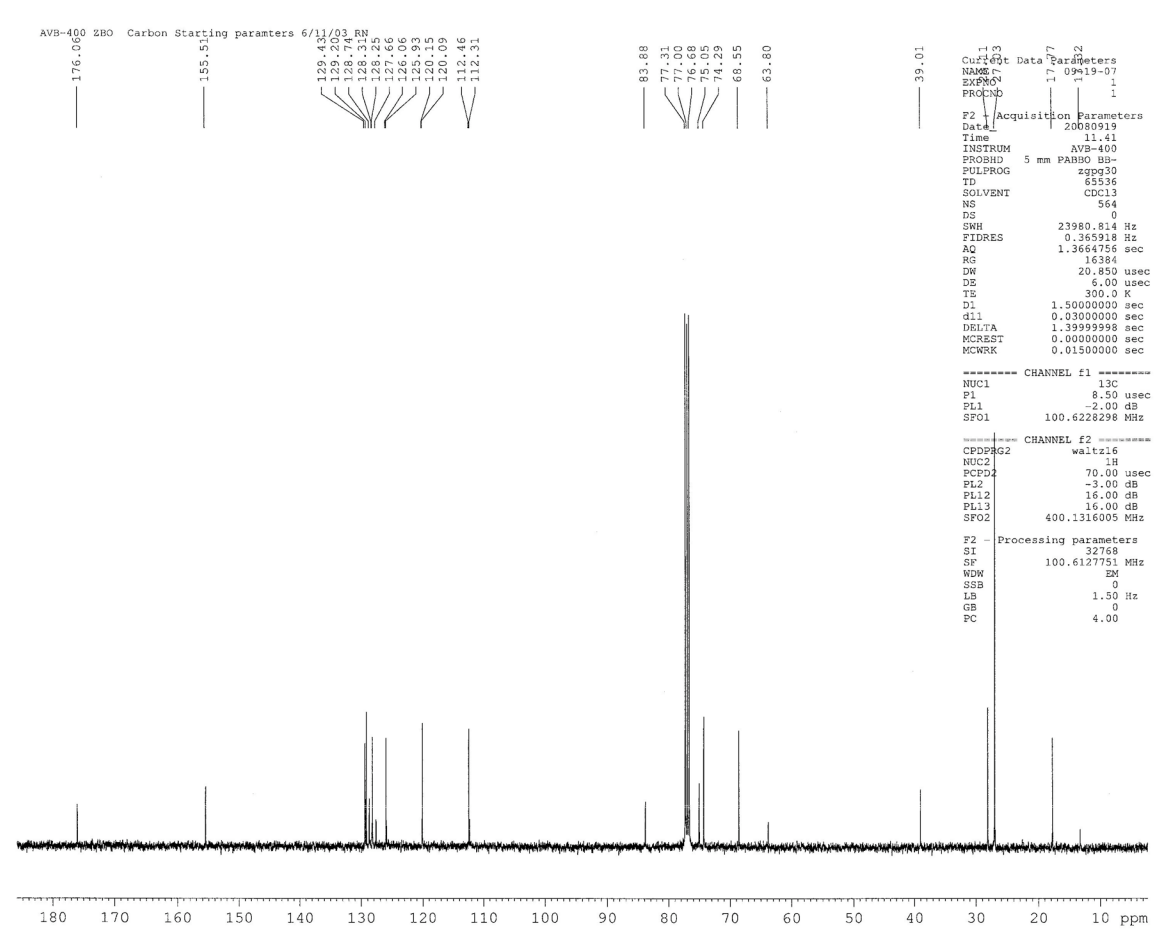
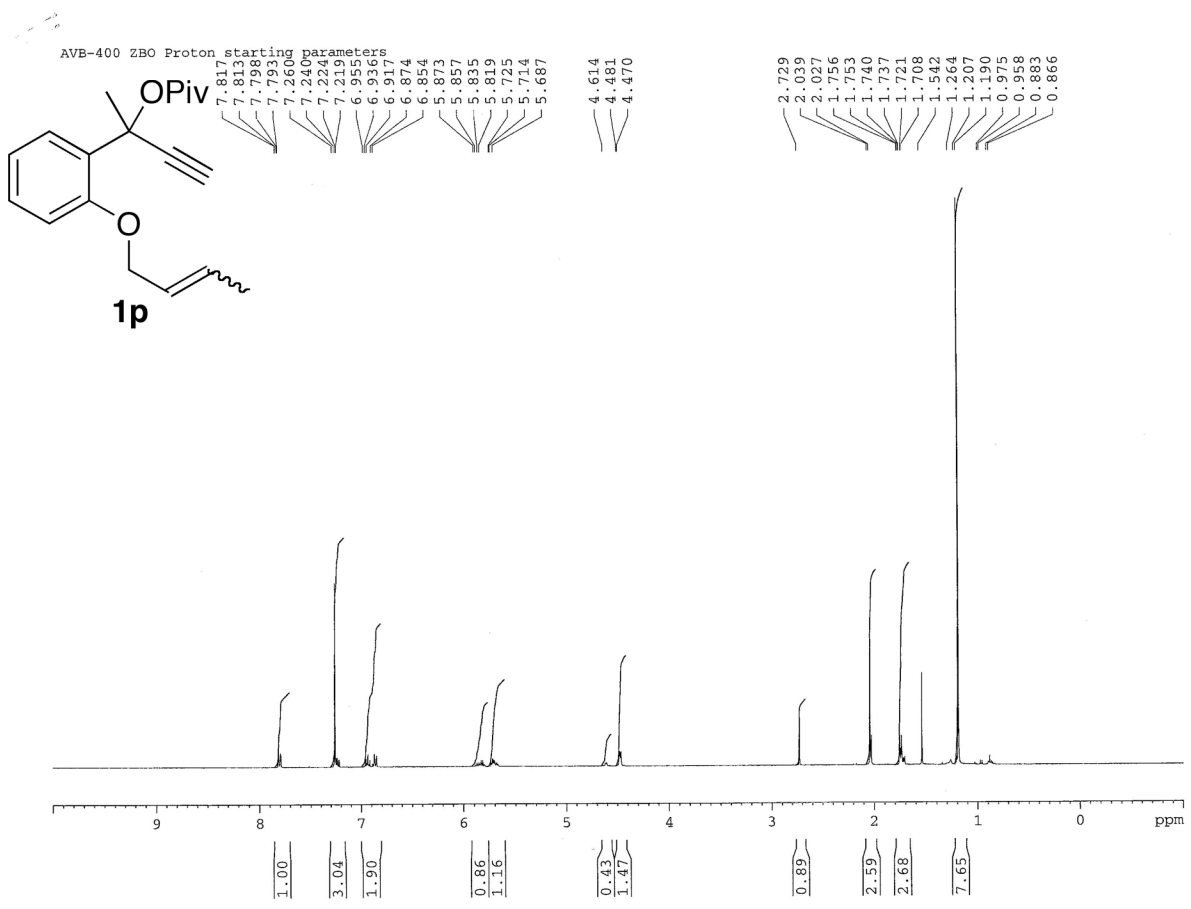
Current Data Parameters
USER      minoru
NAME      10-20-07
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20081020
Time      9.24
INSTRUM   AVB-400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         164
DS         0
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         16384
DW         20.850 usec
DE         6.00 usec
TE         300.0 K
D1         1.50000000 sec
d11        0.03000000 sec
DELTA      1.38999998 sec
MCREST    0.00000000 sec
MCWRK     0.01500000 sec

----- CHANNEL f1 -----
NUC1      13C
P1        8.50 usec
PL1       -2.00 dB
SFO1      100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     70.00 usec
PL2       -3.00 dB
PL12      16.00 dB
PL13      16.00 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127751 MHz
WDW       EM
GB        0
LB        1.50 Hz
PC        4.00
    
```

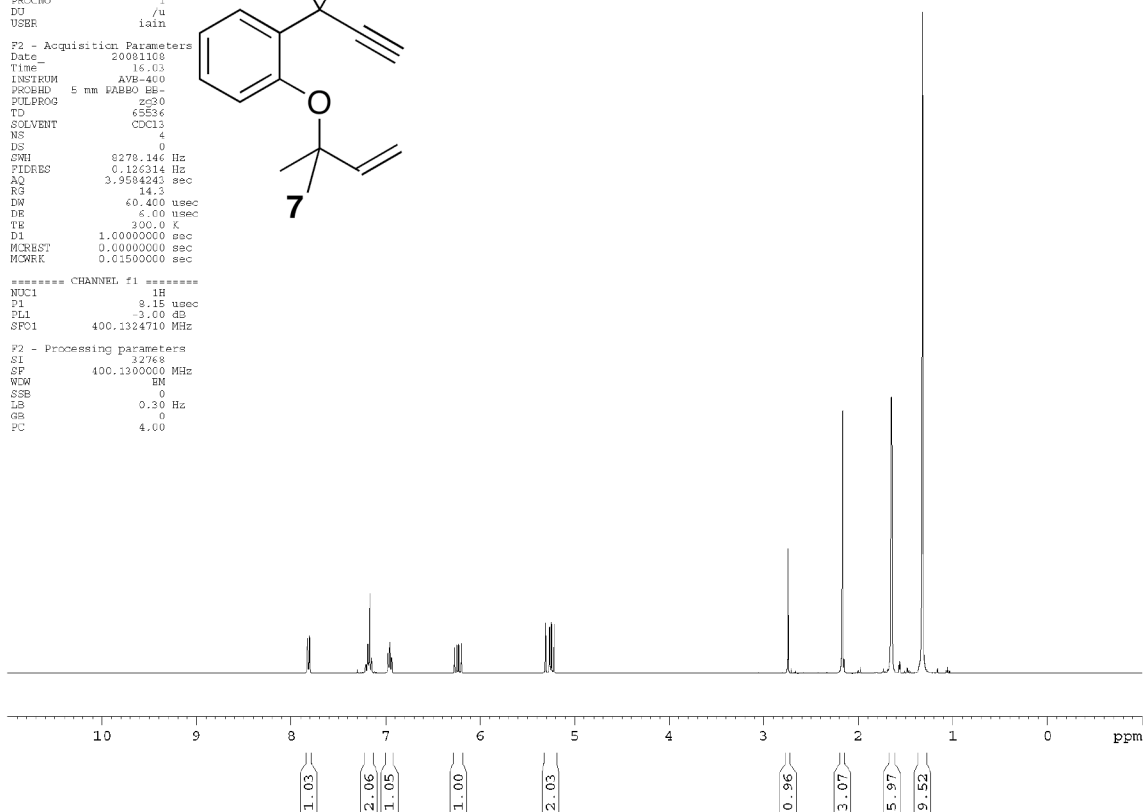
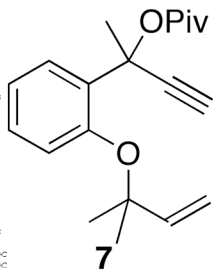
AVB-400 Z80 Proton starting parameters. 6/11/03 RN

Current Data Parameters
NAME IW16132bF03-82_H2
EXFNO 1
PROCNO 1
D3 /u
USBR iain

F2 - Acquisition Parameters
Date 20081108
Time 16.03
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 14.3
DW 40.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWREK 0.0150000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 8.15 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00



AVB-400 Z80 Carbon Starting parameters 6/11/03 RN

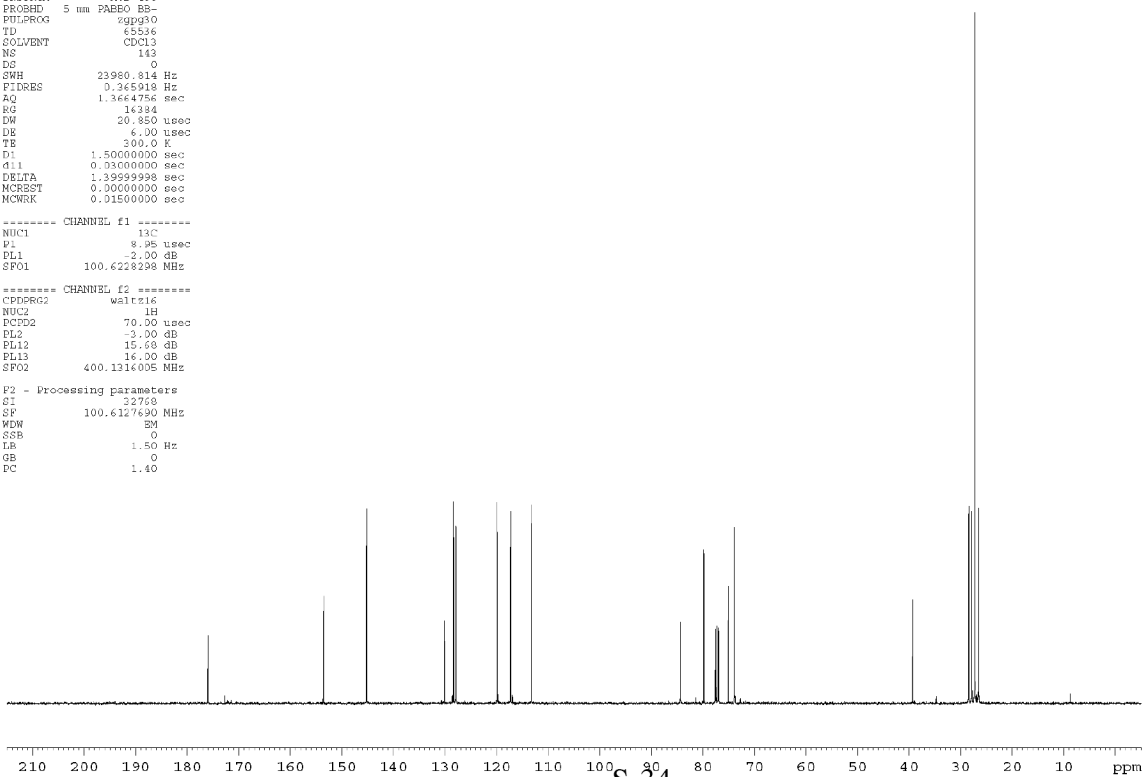
Current Data Parameters
NAME IW16132bF03-82_C
EXFNO 1
PROCNO 1

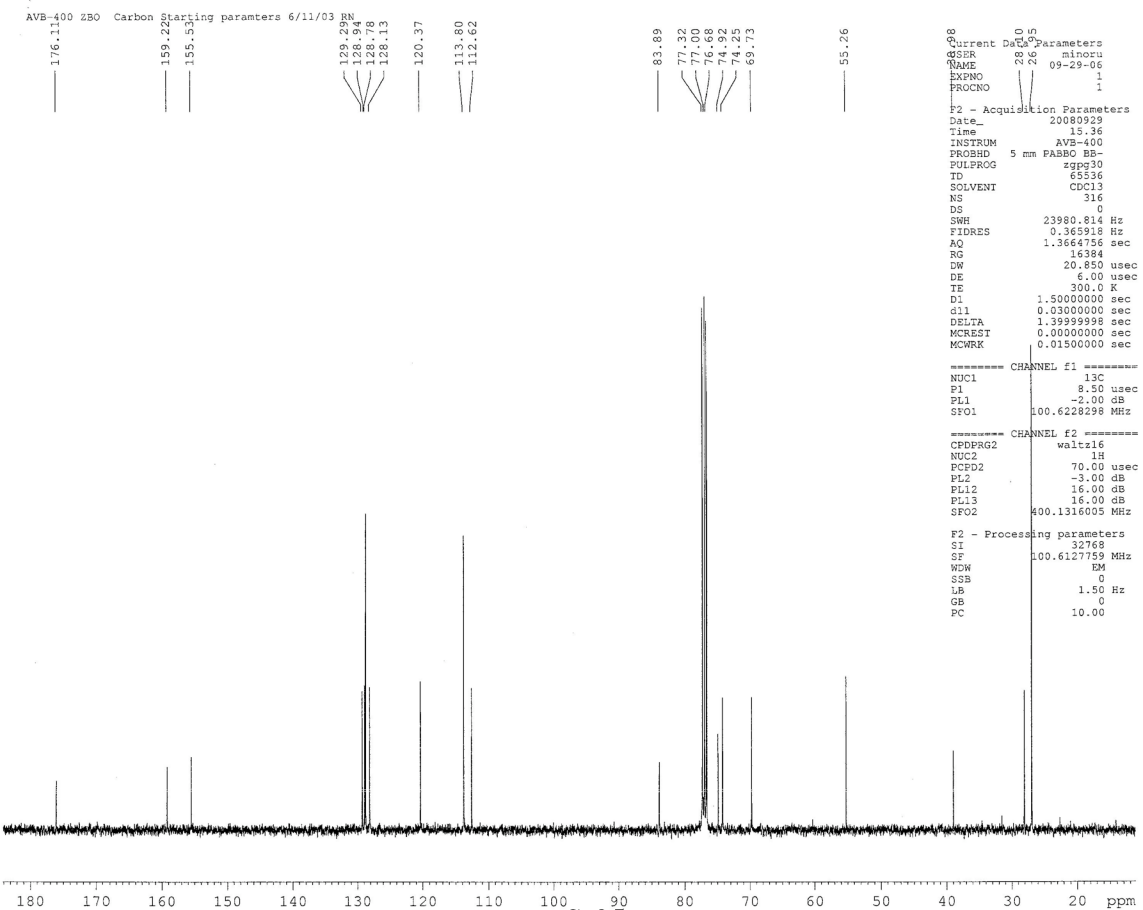
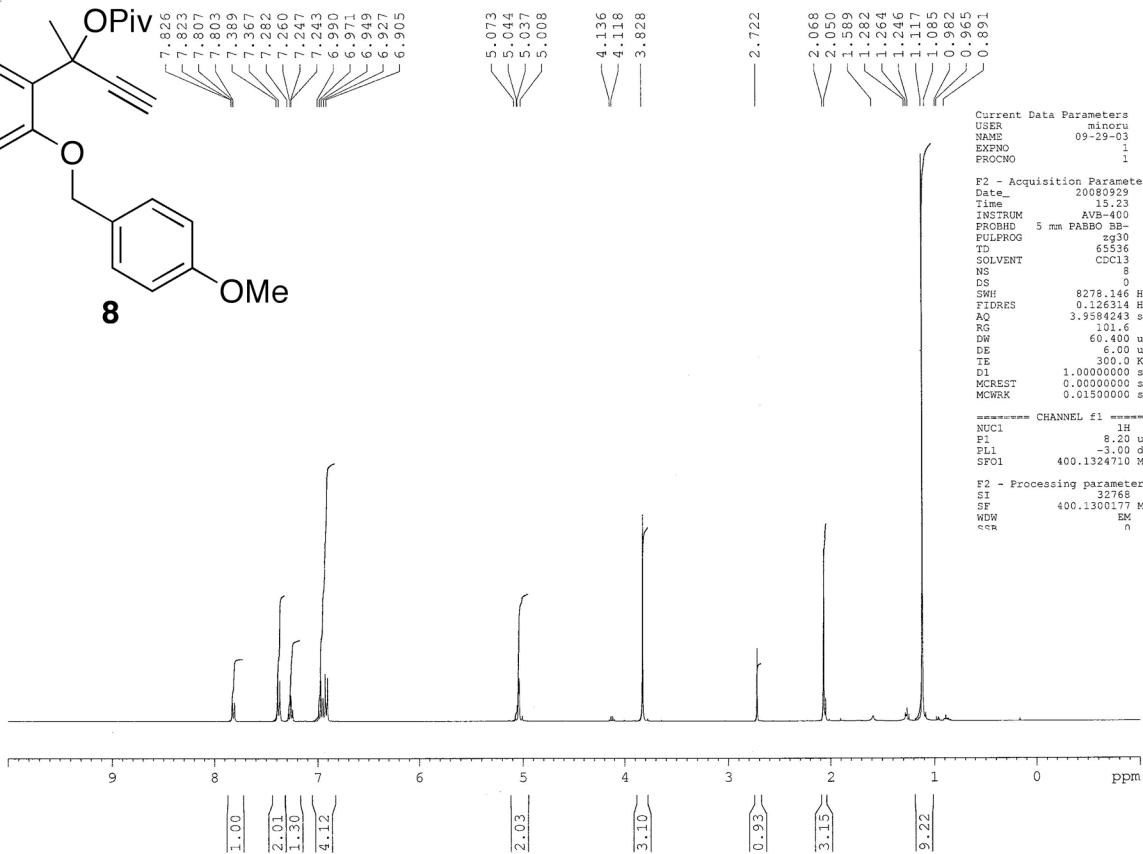
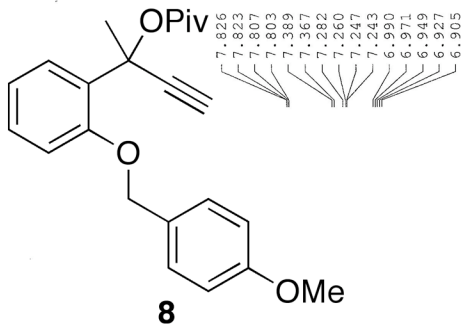
F2 - Acquisition Parameters
Date_ 20081108
Time_ 16.05
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 143
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3644758 sec
RG 14384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 1.5000000 sec
d11 0.0300000 sec
DELTA 1.3999998 sec
MCREST 0.0000000 sec
MCWREK 0.0150000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 8.95 usec
PL1 -2.00 dB
SFO1 100.6228298 MHz

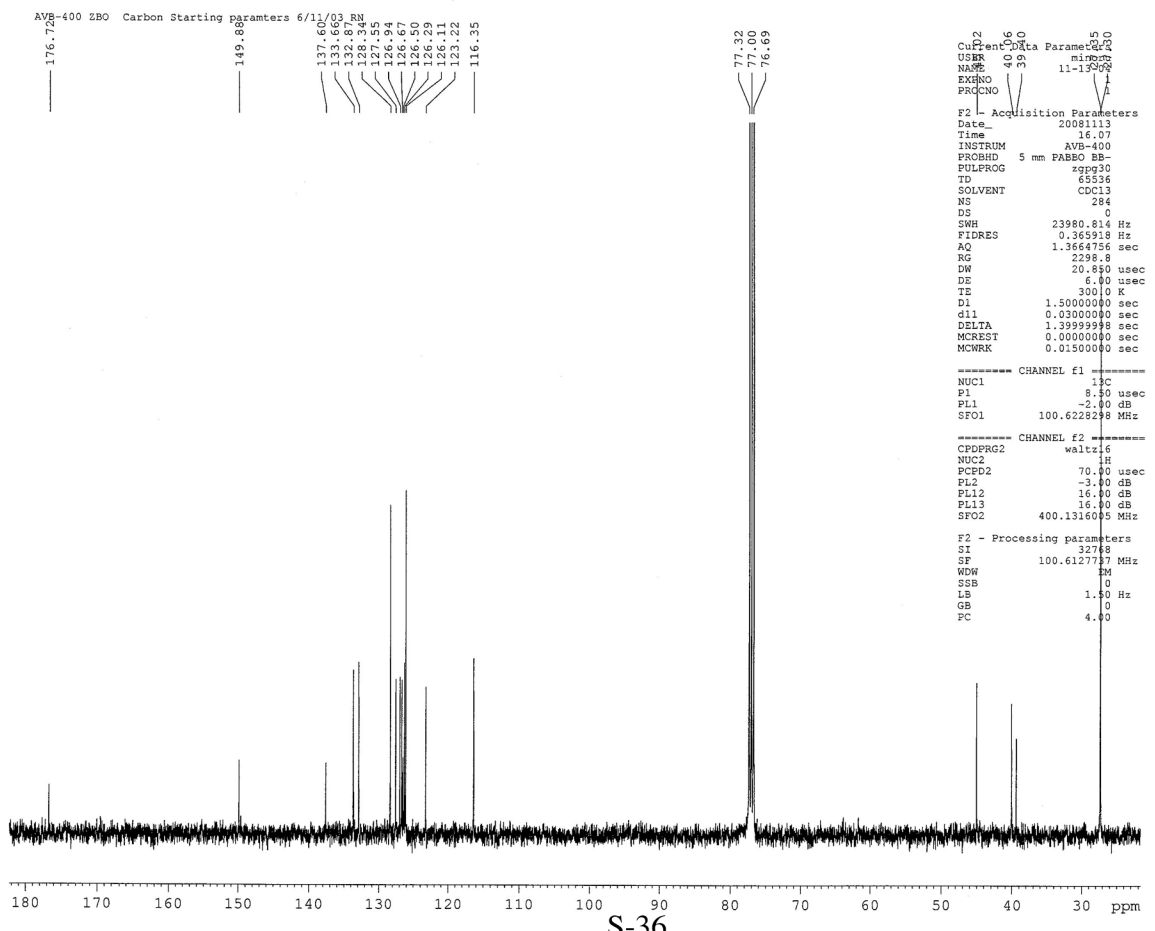
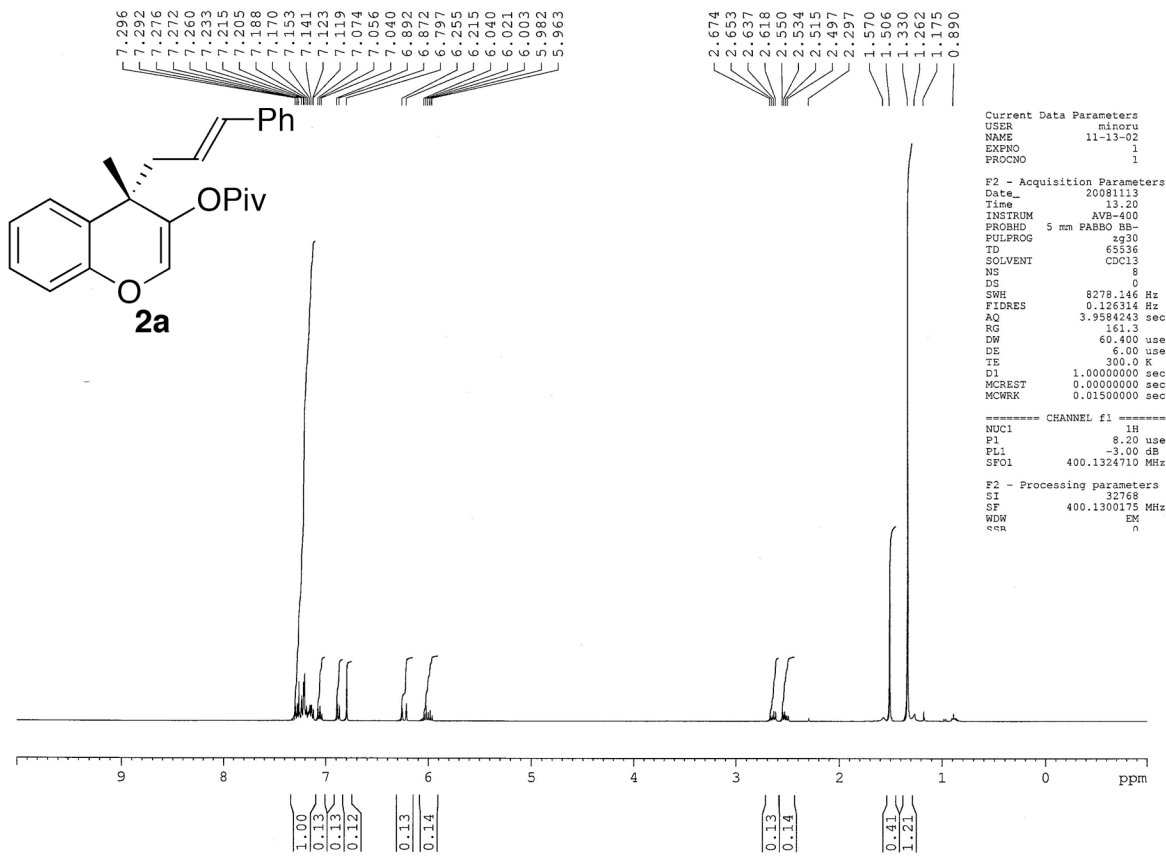
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
INSTR2 70.00 usec
PL2 -3.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 400.1316005 MHz

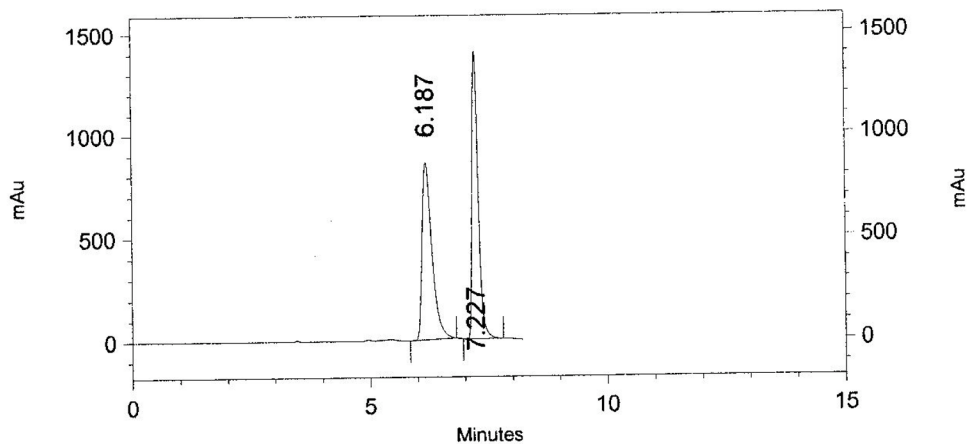
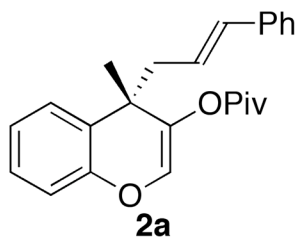
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40





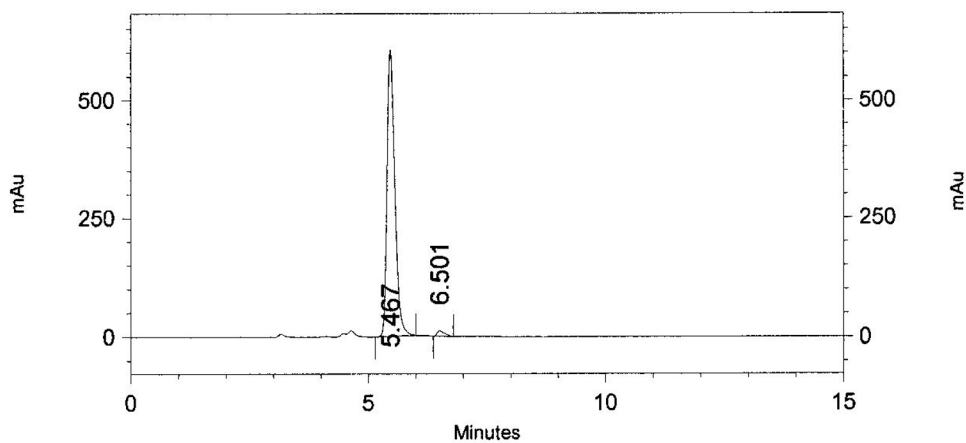
AVB-400 ZBO Proton starting parameters. 6/11/03 RN





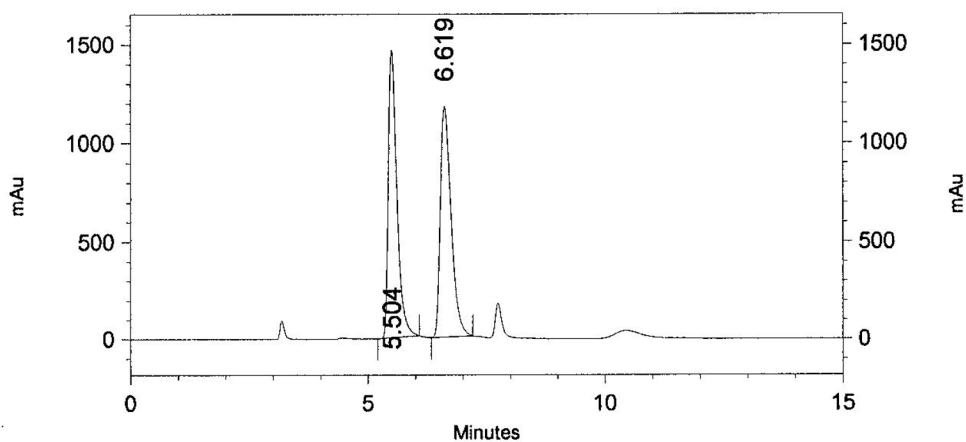
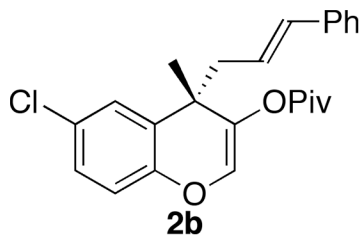
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
6.187	12376609	49.420
7.227	12667200	50.580



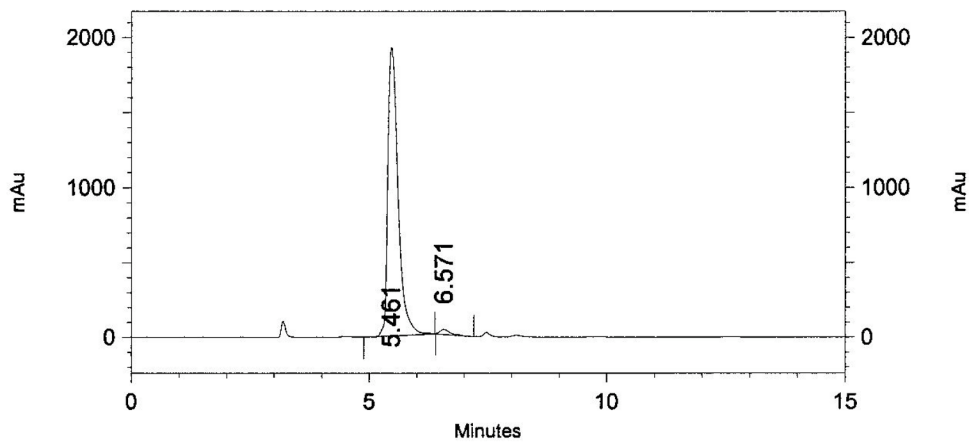
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
5.467	6909726	98.255
6.501	122699	1.745



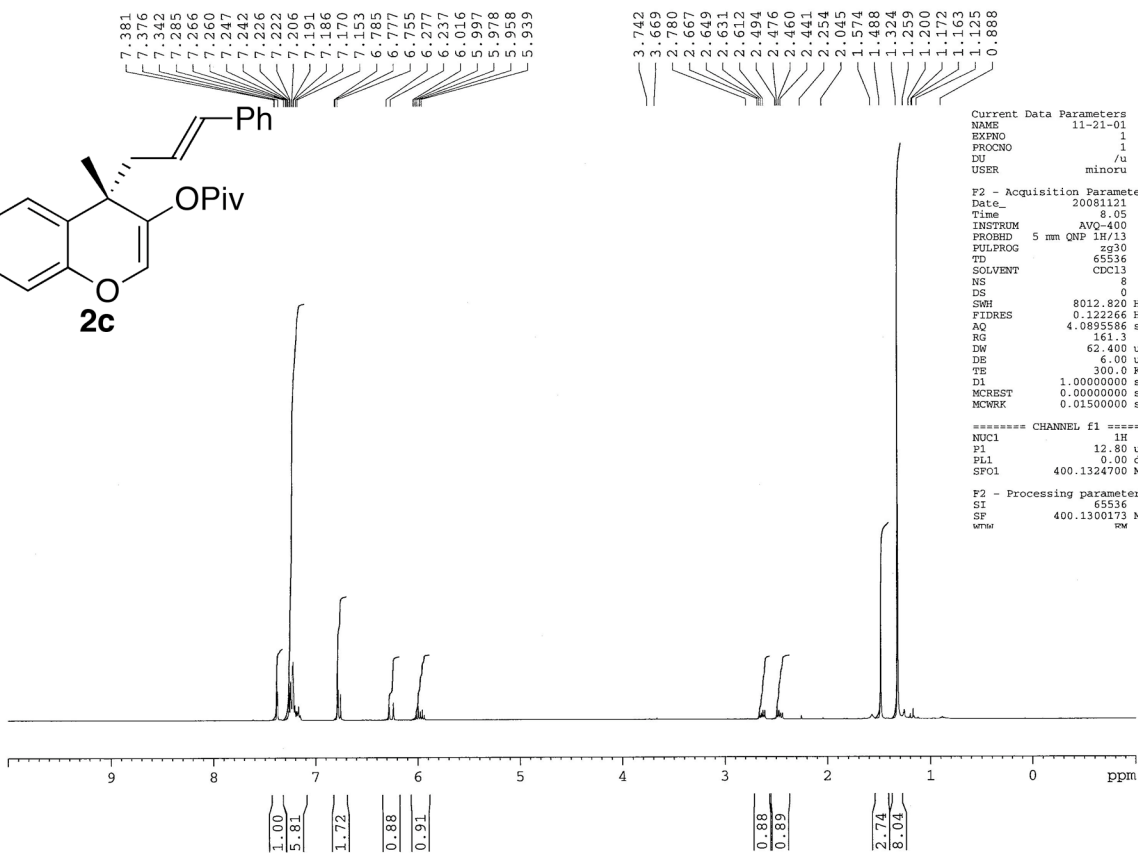
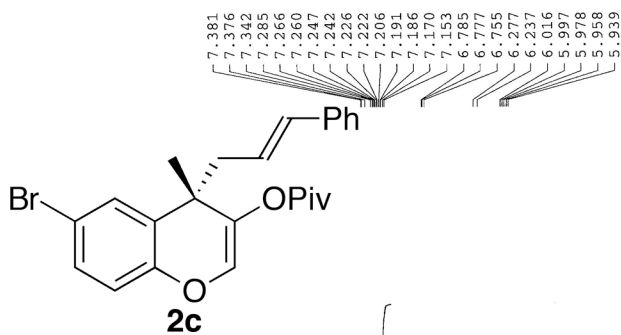
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
5.504	18518320	49.722
6.619	18725063	50.278



2: 260 nm, 4 nm Results

Retention Time	Area	Area Percent
5.461	28924585	98.306
6.571	498339	1.694



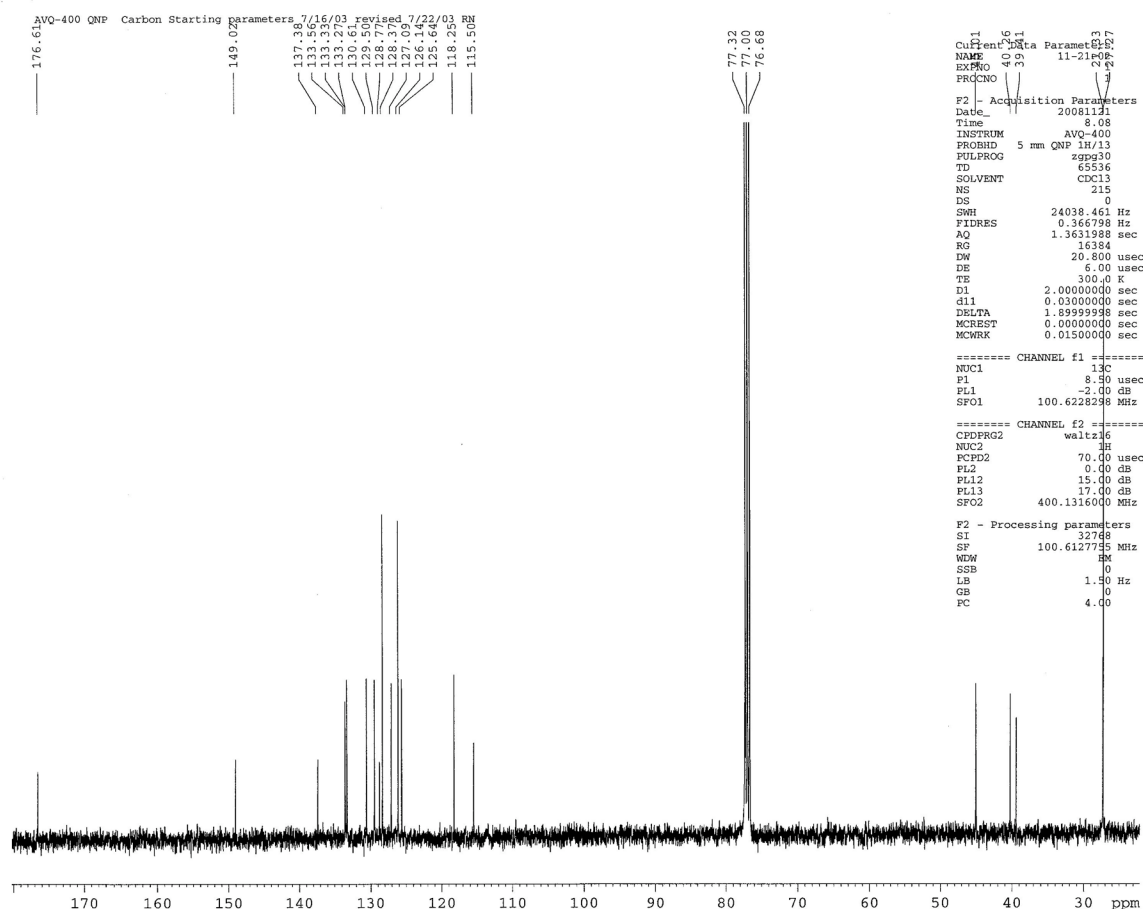
```

Current Data Parameters
NAME      11-21-01
EXPNO    1
PROCNO   1
DU       /u
USER     minoru

F2 - Acquisition Parameters
Date_    20081121
Time     8.05
INSTRUM AVQ-400
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0895586 sec
RG       161.3
DW       62.400 usec
DE       6.00 usec
TE       300.6 K
D1       1.0000000 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       12.80 usec
PL1      0.00 dB
SFO1     400.1324700 MHz

F2 - Processing parameters
SI       65536
SF       400.1300173 MHz
WDW      sm
  
```



```

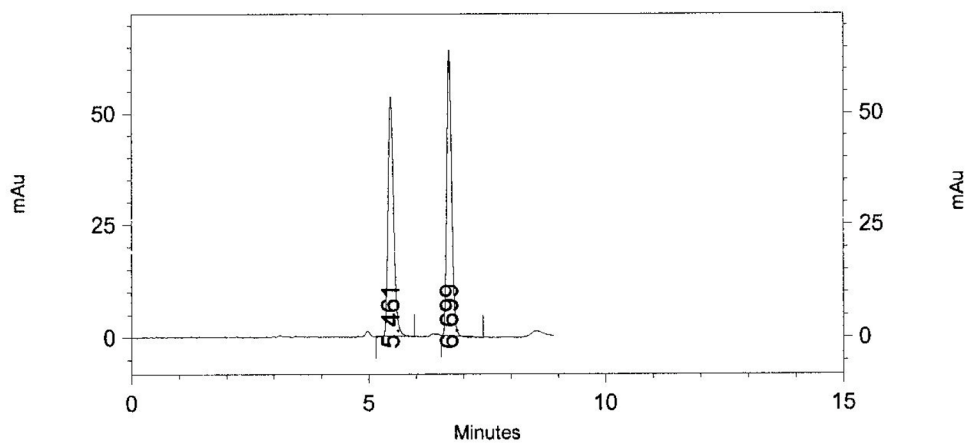
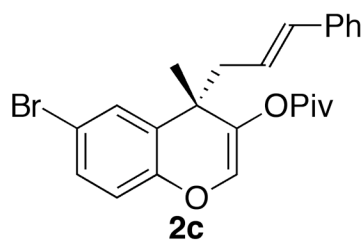
Current Data Parameters
NAME      11-21-01
EXPNO    1
PROCNO   1
DU       /u
USER     minoru

F2 - Acquisition Parameters
Date_    20081121
Time     8.08
INSTRUM AVQ-400
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       16384
DW       20.800 usec
DE       6.00 usec
TE       300.0 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999998 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1     13C
P1       8.80 usec
PL1      -2.00 dB
SFO1     100.6282800 MHz

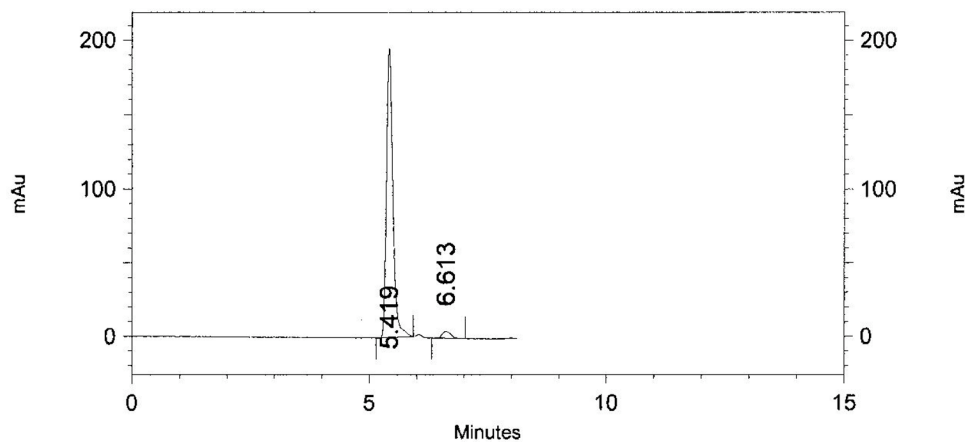
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    70.00 usec
PL2      0.00 dB
PL12     15.00 dB
PL13     17.00 dB
SFO2     400.1316000 MHz

F2 - Processing parameters
SI       32768
SF       100.6127745 MHz
WDW      EM
SSB      0
LB       1.50 Hz
GB       0
PC       4.00
  
```

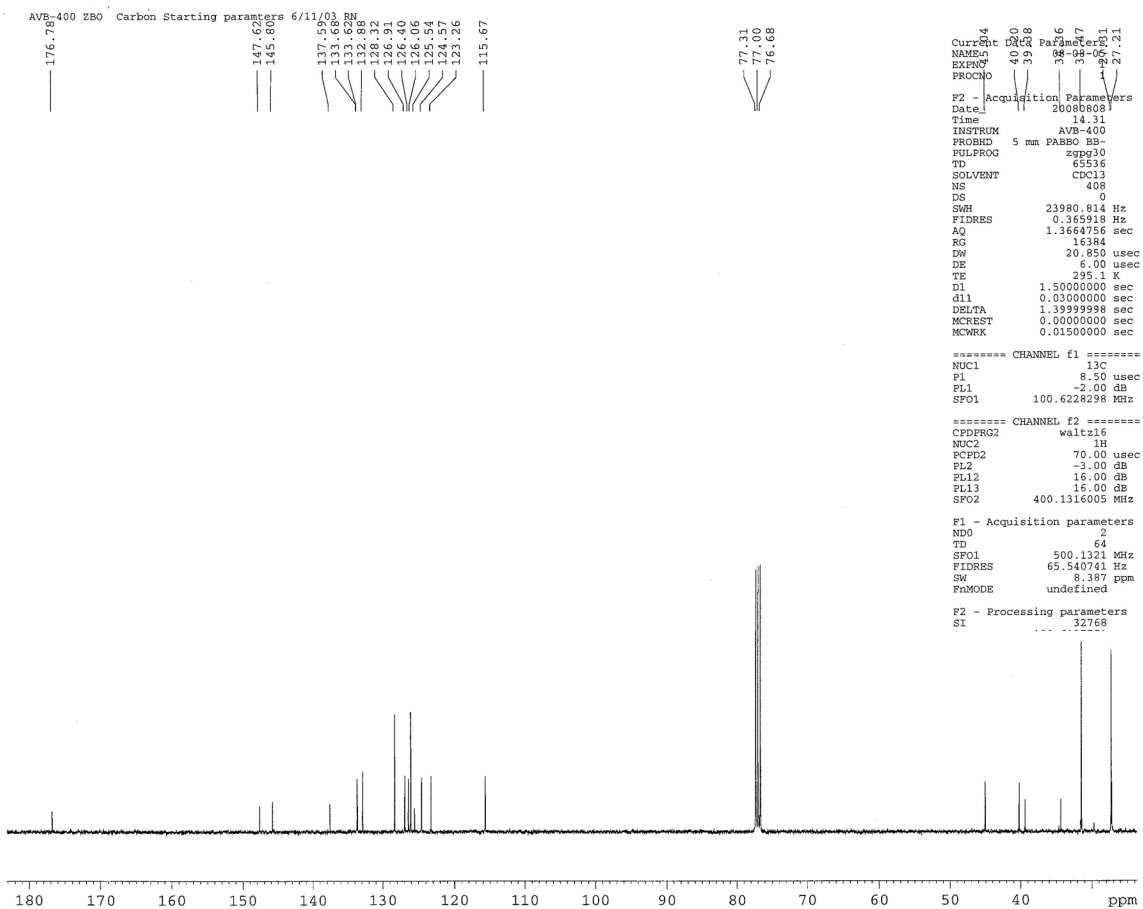
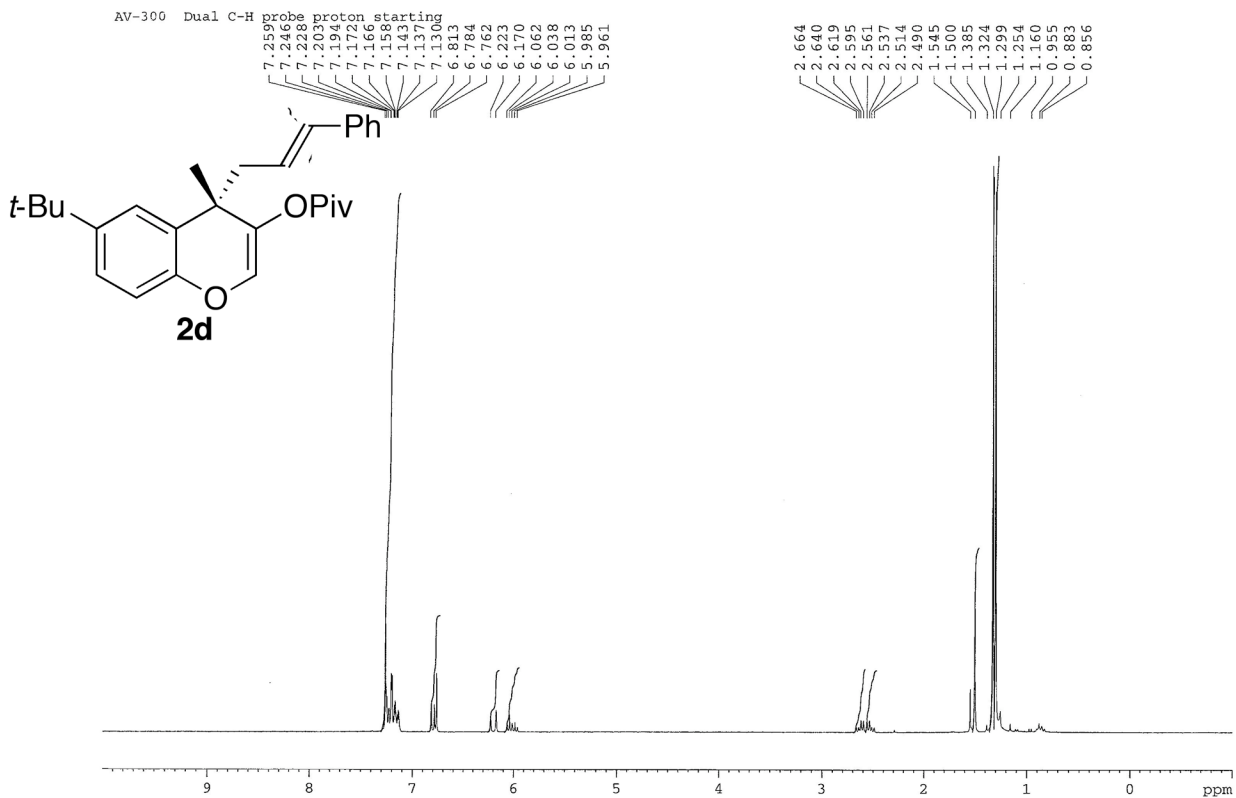
2: 254 nm, 4 nm Results

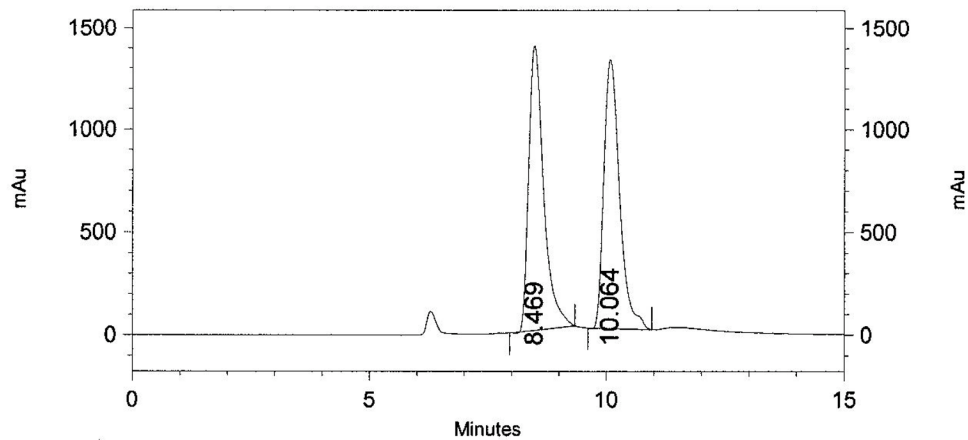
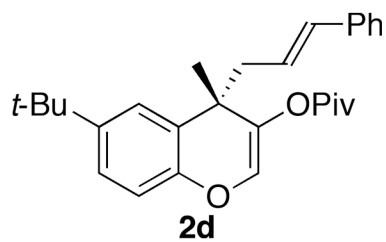
Retention Time	Area	Area Percent
5.461	433284	49.020
6.699	450610	50.980



2: 254 nm, 4 nm Results

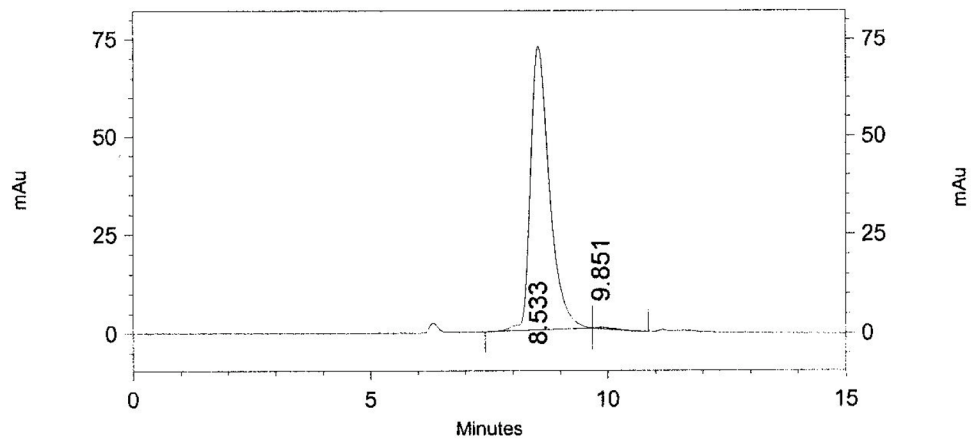
Retention Time	Area	Area Percent
5.419	1827773	96.914
6.613	58204	3.086





2: 254 nm, 4 nm Results

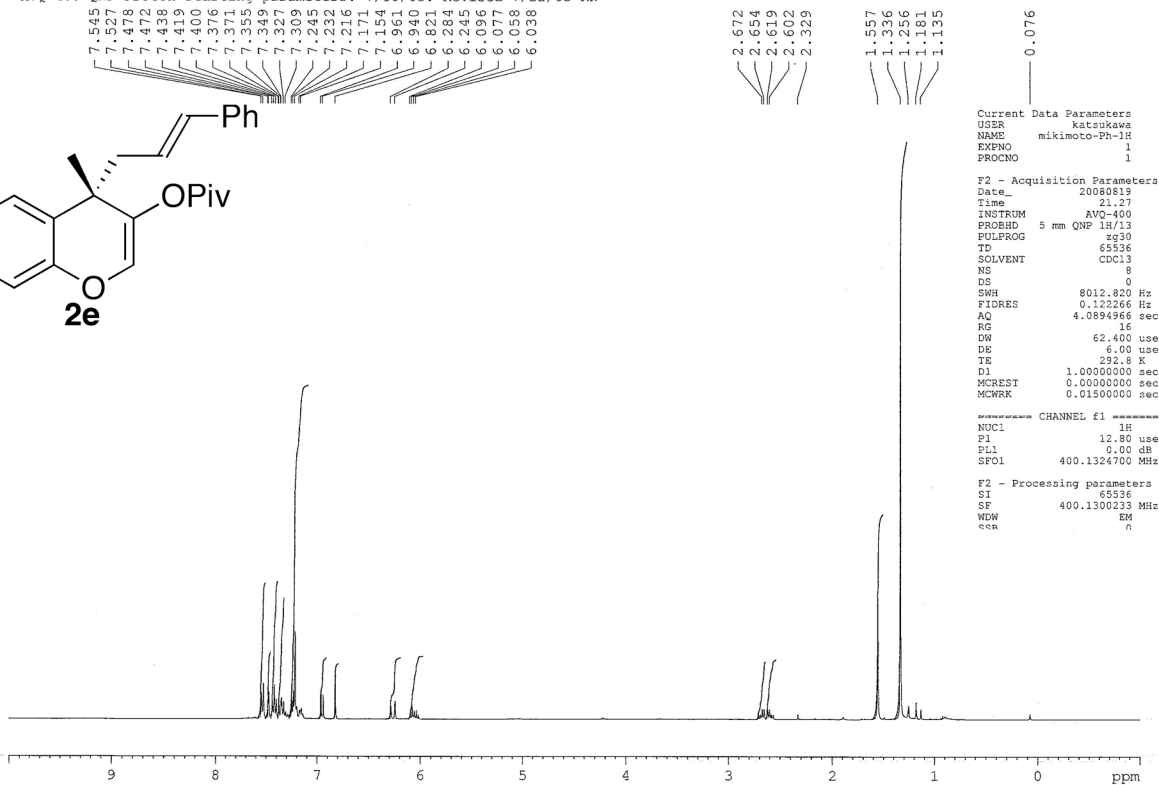
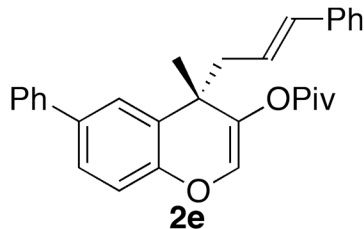
Retention Time	Area	Area Percent
8.469	30422185	50.155
10.064	30234486	49.845



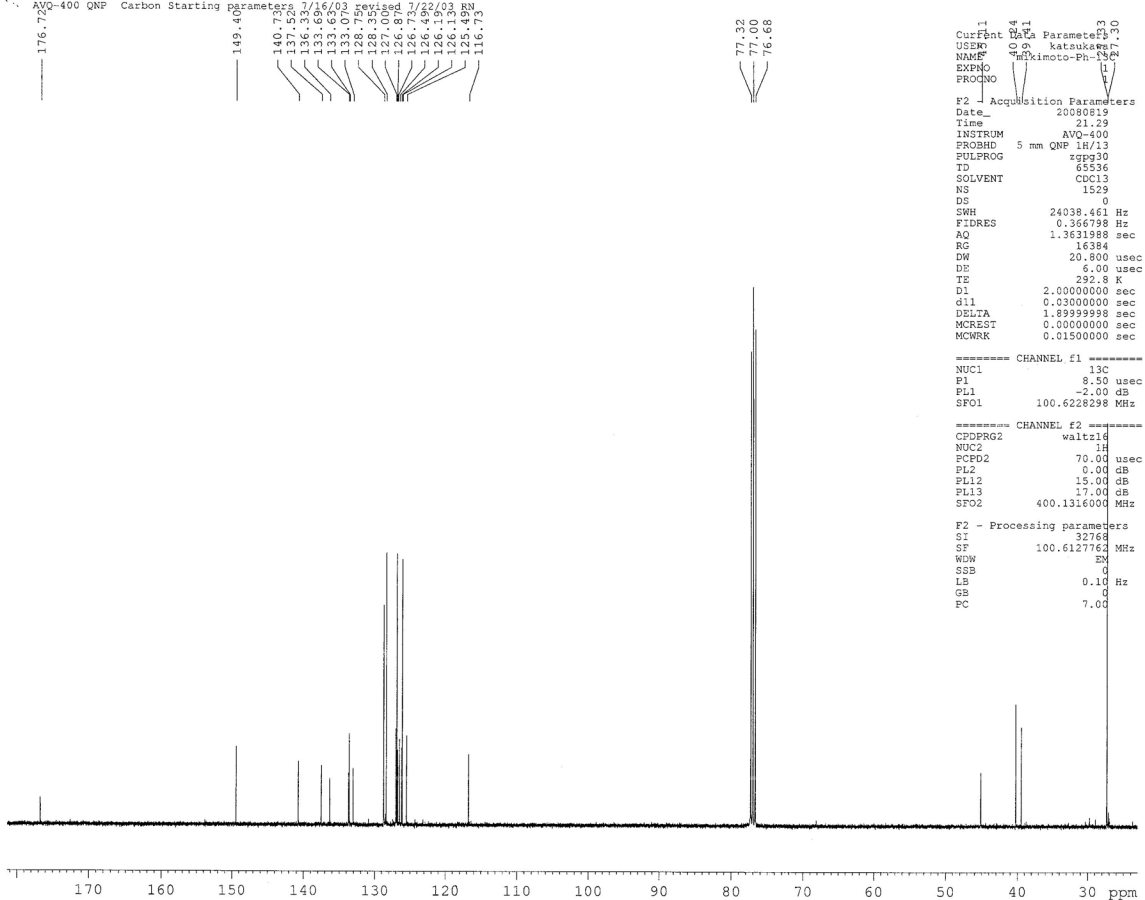
3: 279 nm, 4 nm Results

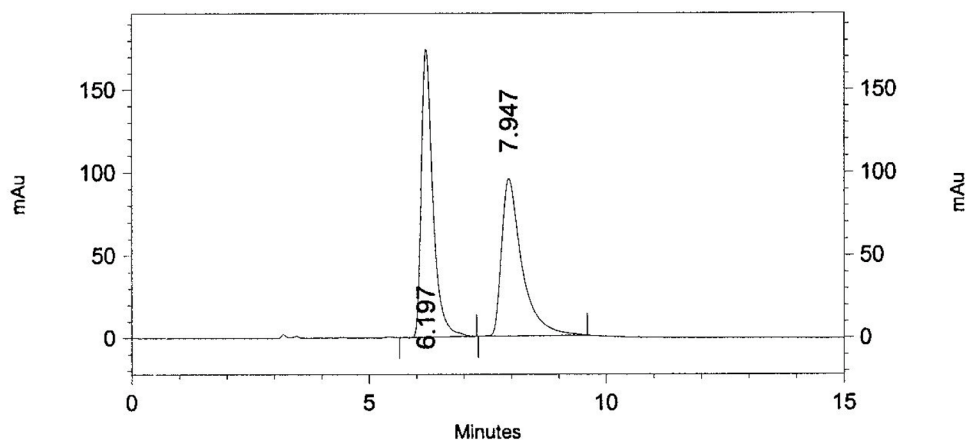
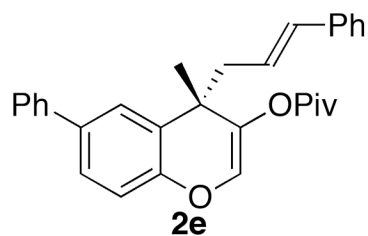
Retention Time	Area	Area Percent
8.533	1998255	99.743
9.851	5148	0.257

mikimoto-Ph-1H
AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN



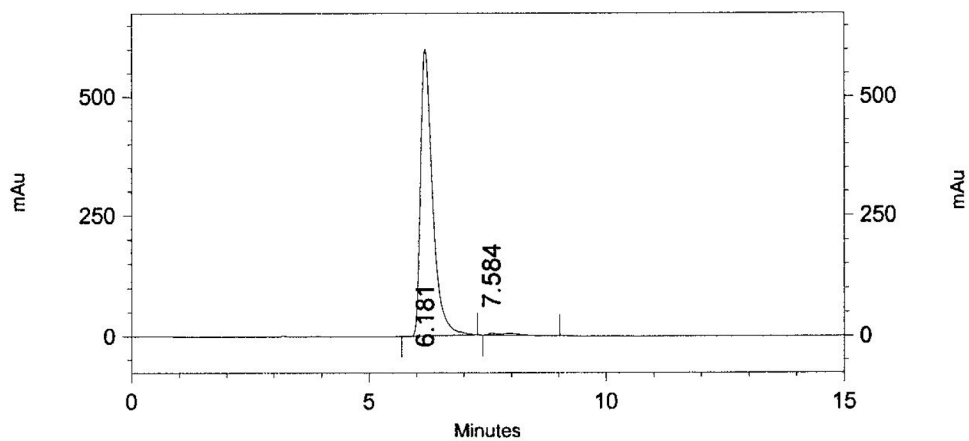
AVQ-400 QNP Carbon Starting parameters. 7/22/03 RN





2: 254 nm, 4 nm Results

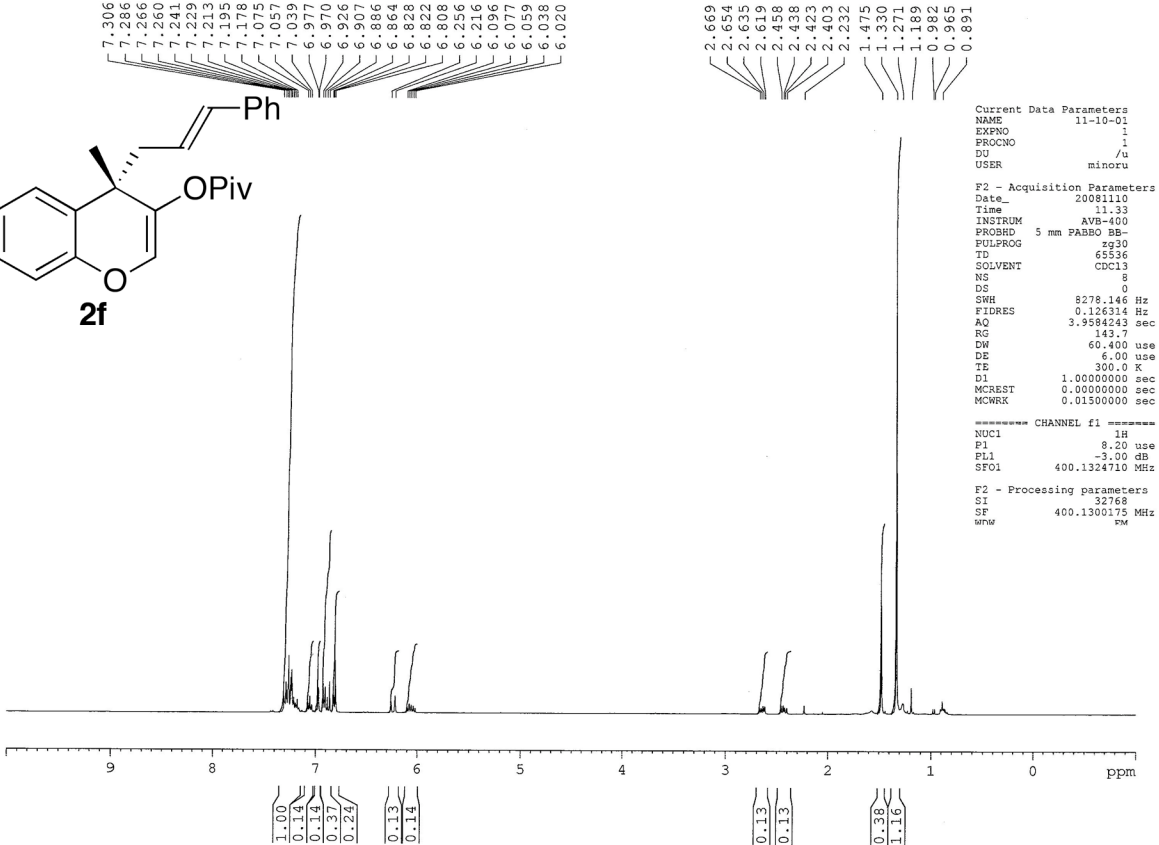
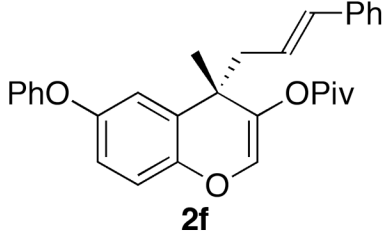
Retention Time	Area	Area Percent
6.197	2973069	51.105
7.947	2844548	48.895



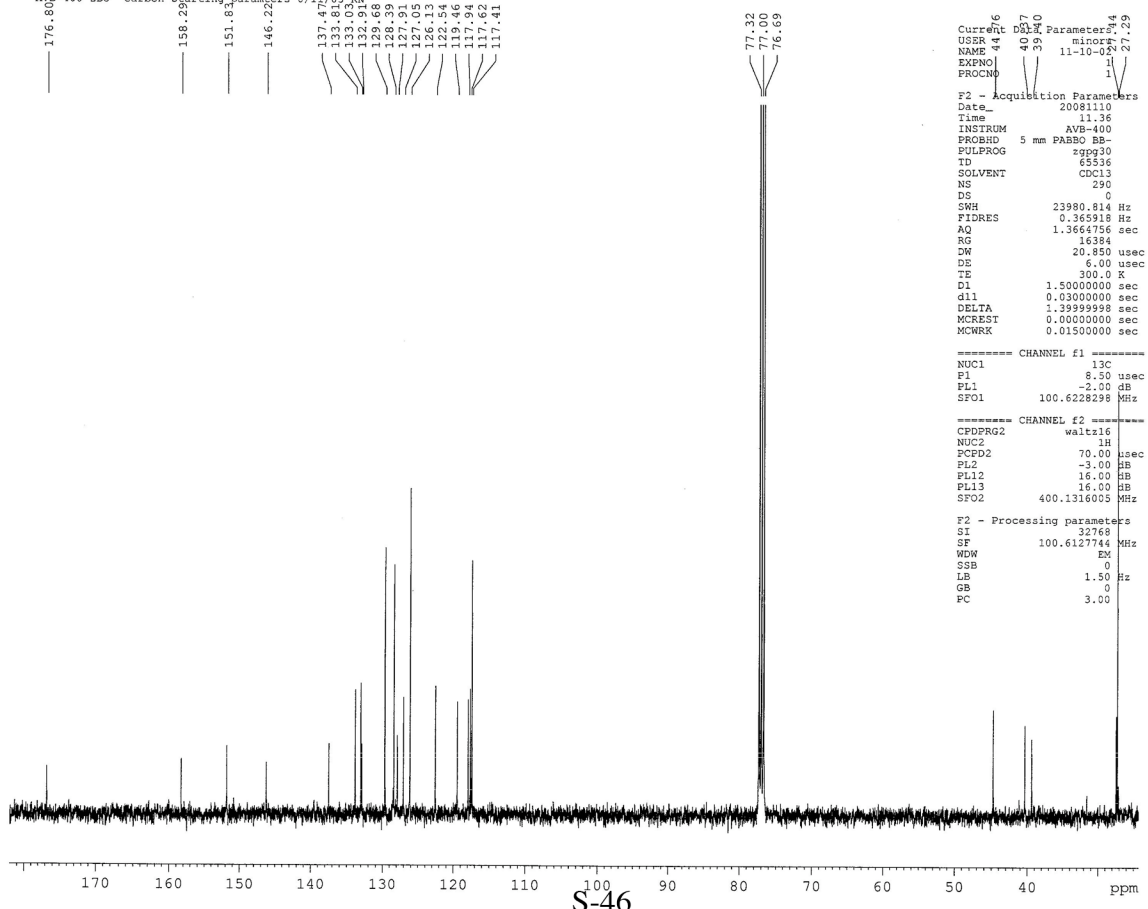
3: 280 nm, 4 nm Results

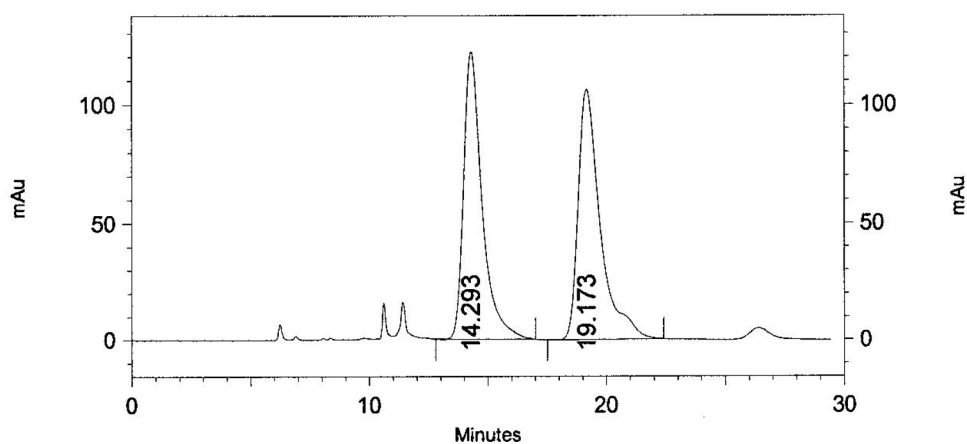
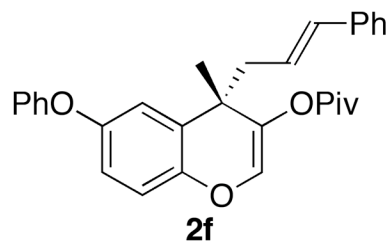
Retention Time	Area	Area Percent
6.181	10522737	98.695
7.584	139086	1.305

AVB-400 ZBO Proton starting parameters. 6/11/03 RN



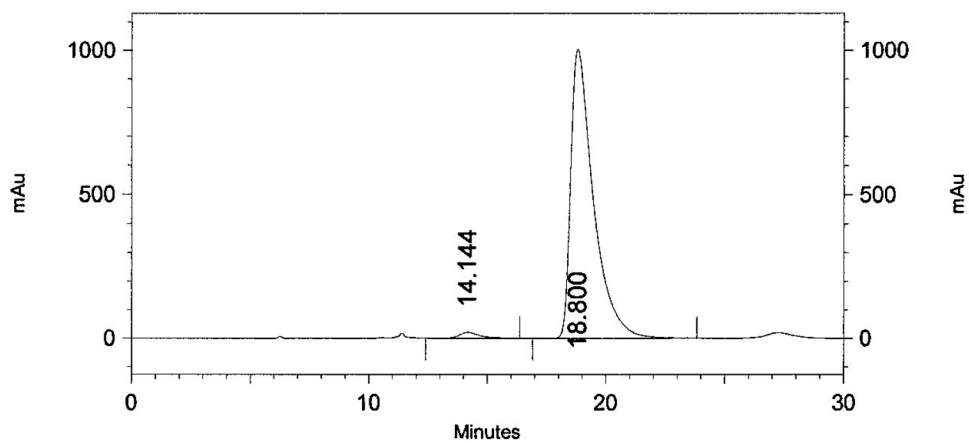
AVB-400 ZBO Carbon Starting parameters 6/11/03 RN





2: 254 nm, 4 nm Results

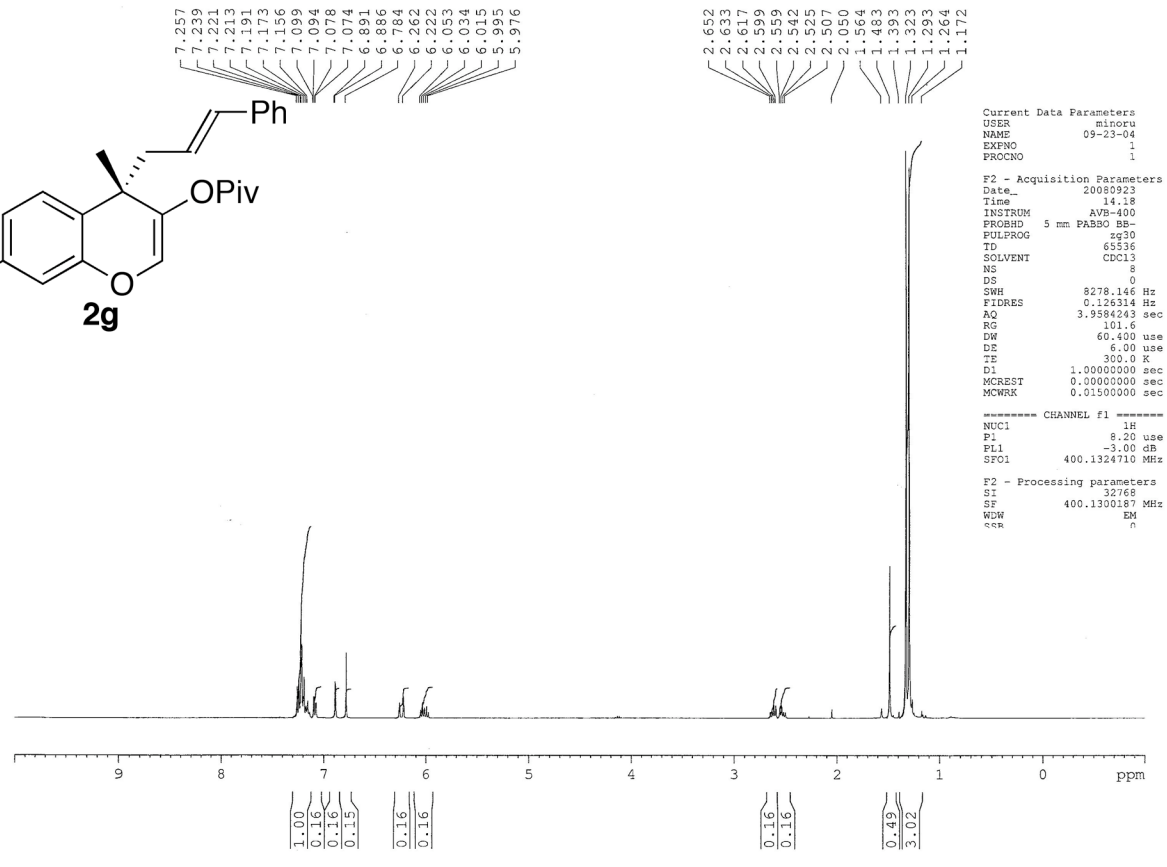
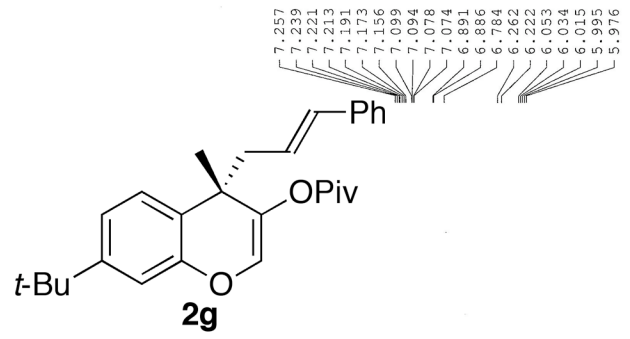
Retention Time	Area	Area Percent
14.293	6657322	49.001
19.173	6928810	50.999



2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
14.144	1176709	1.729
18.800	66877686	98.271

AVB-400 ZBO Proton starting parameters. 6/11/03 RN

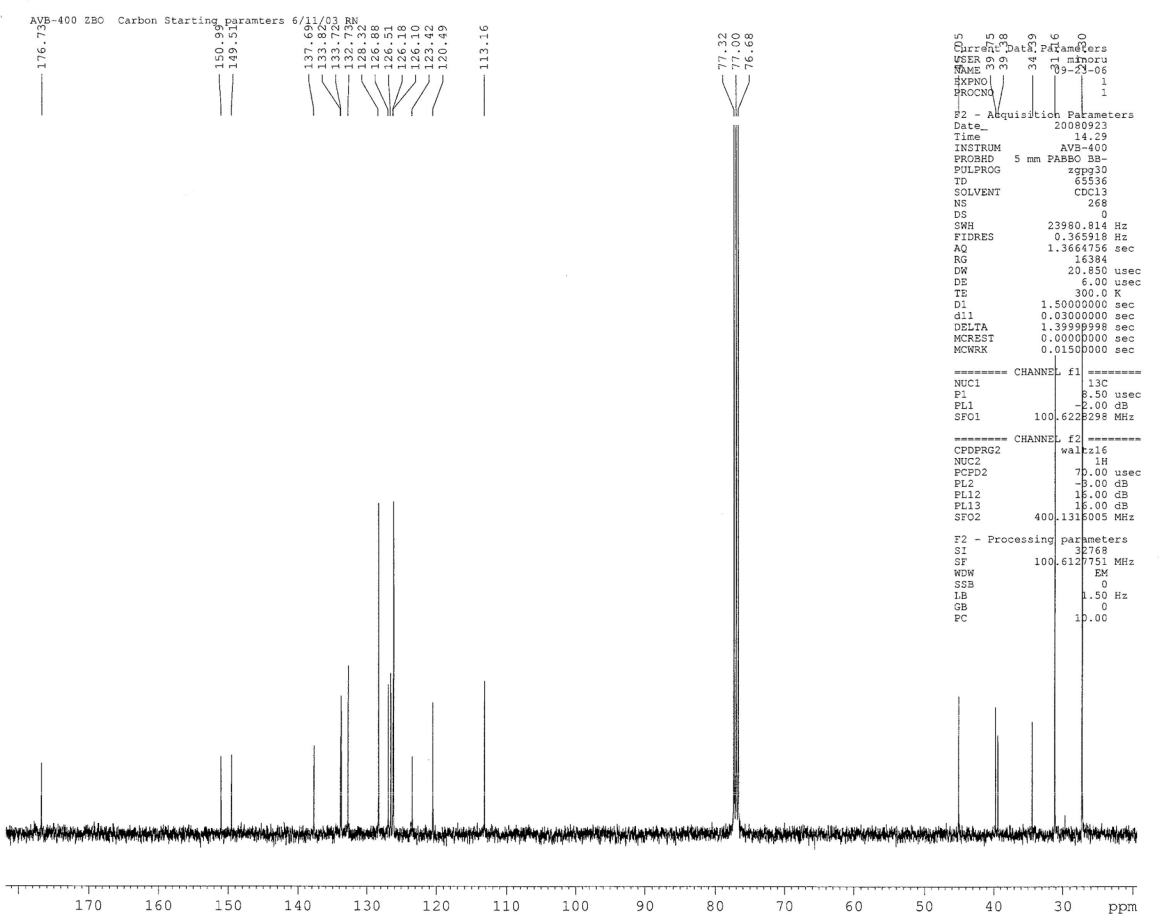


Current Data Parameters
 USER minoru
 NAME 09-23-04
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080923
 Time 14.18
 INSTRUM AVB-400
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 101.6
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 1H
 P1 8.20 usec
 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 32768
 SF 400.1300187 MHz
 WDW EM
 SSB 0



AVB-400 ZBO Carbon Starting parameters 6/11/03 RN

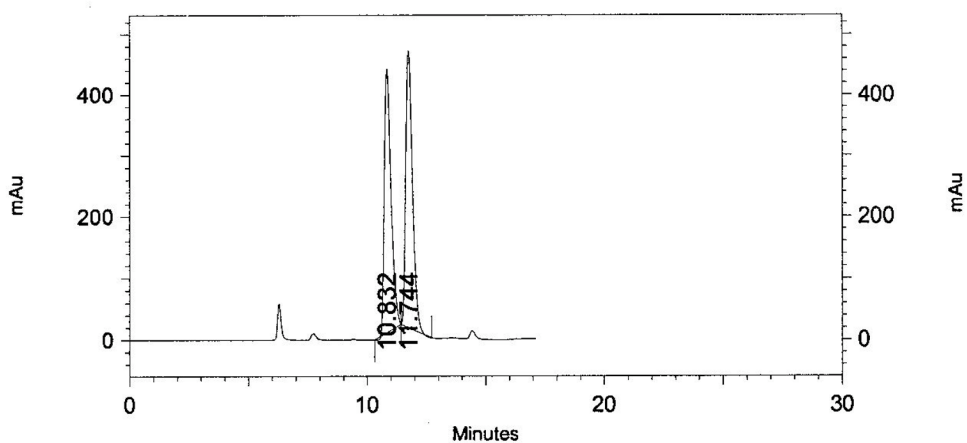
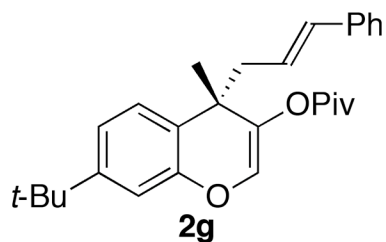
Current Data Parameters
 USER minoru
 NAME 09-23-06
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080923
 Time 14.29
 INSTRUM AVB-400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 268
 DS 0
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 16384
 DW 20.850 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.50000000 sec
 d11 0.03000000 sec
 DELTA 1.33999998 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 SFO1 100.6228298 MHz

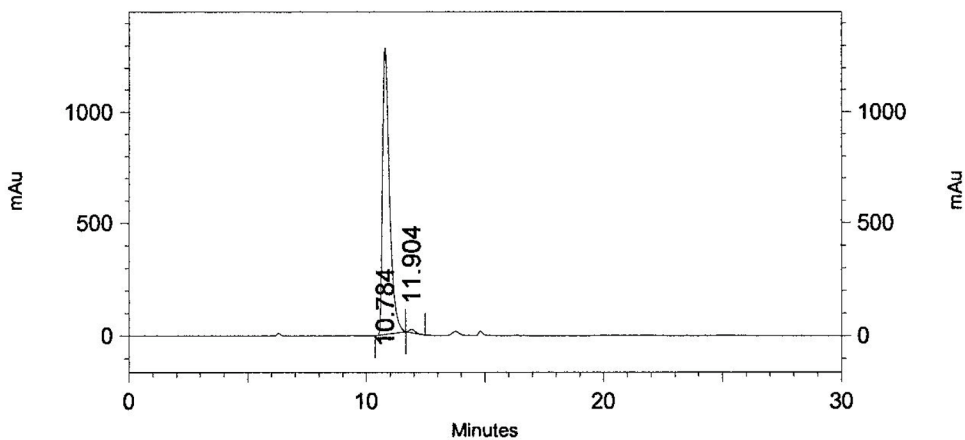
----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 70.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 400.1315005 MHz

F2 - Processing Parameters
 SI 32768
 SF 100.6127751 MHz
 WDW EM
 SSB 0
 IS 1.50 Hz
 GB 0
 FC 10.00



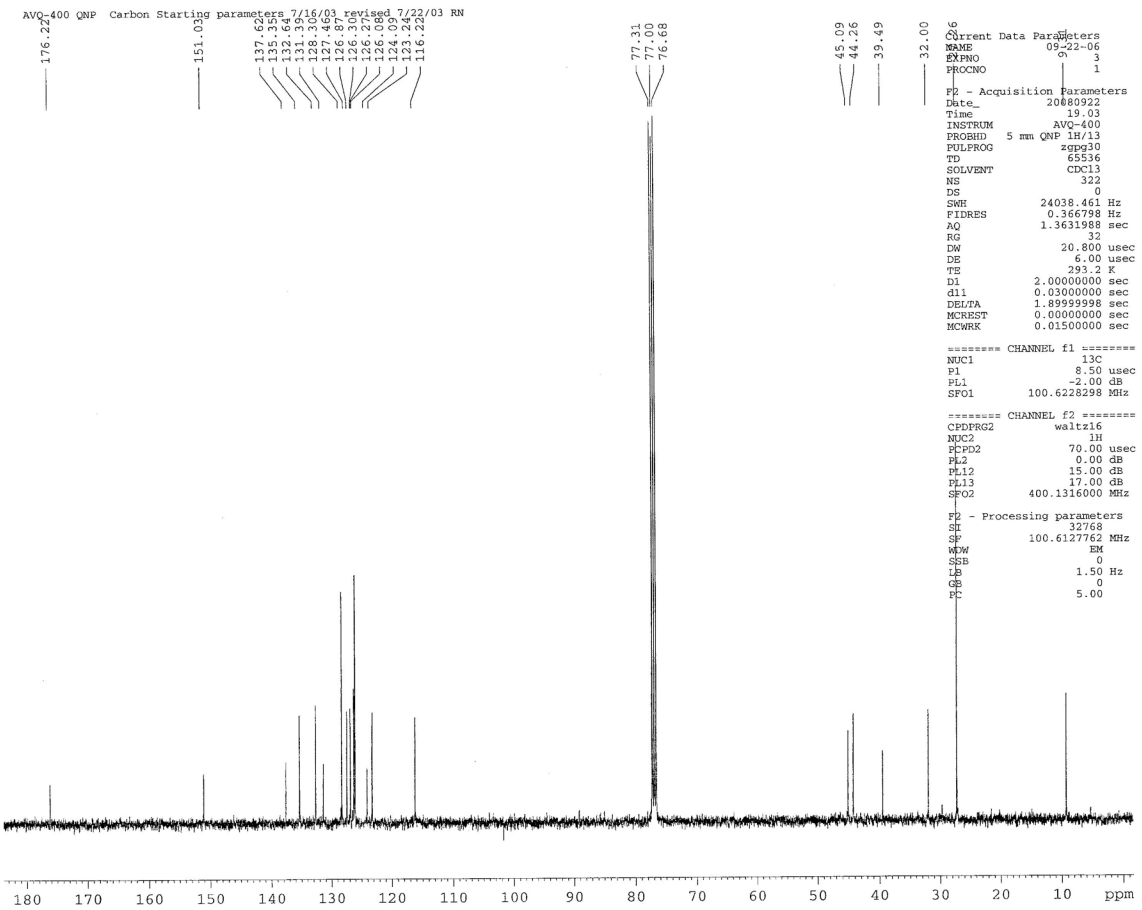
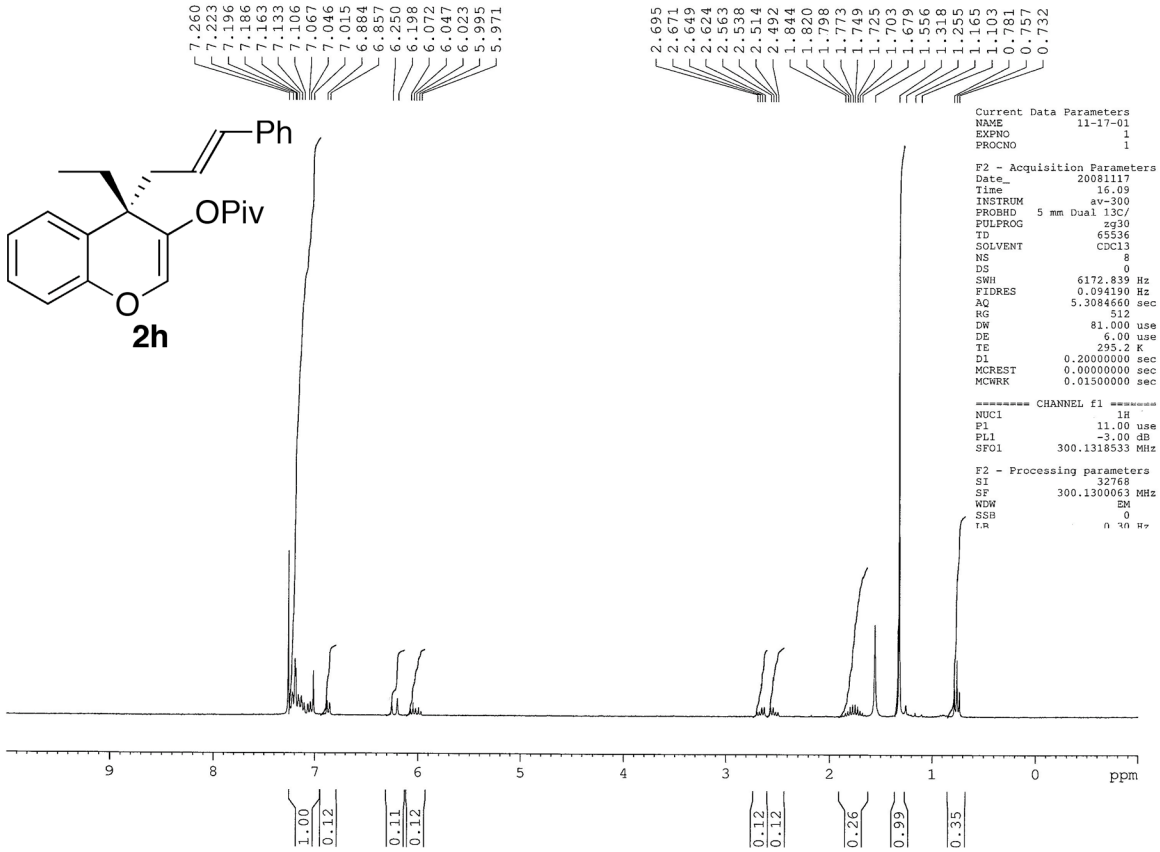
2: 254 nm, 4 nm Results

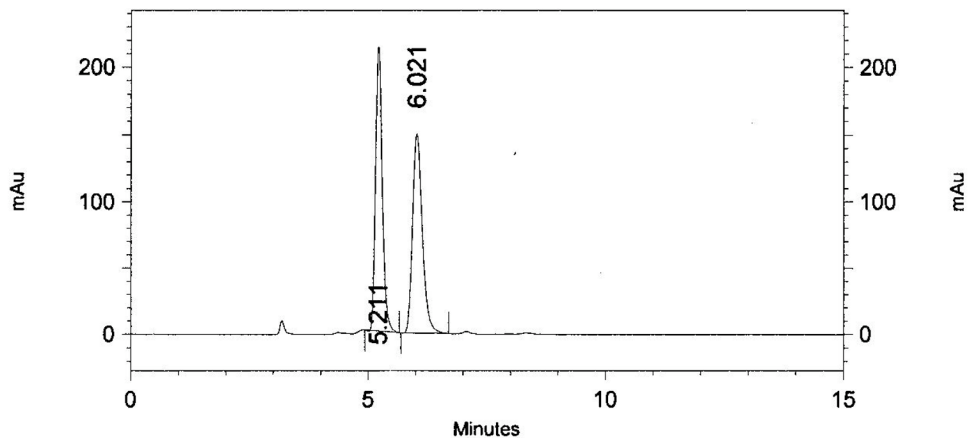
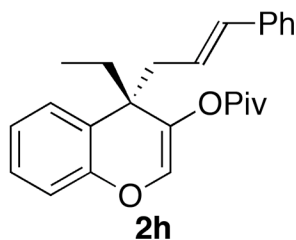
Retention Time	Area	Area Percent
10.832	8493881	48.700
11.744	8947180	51.300



2: 254 nm, 4 nm Results

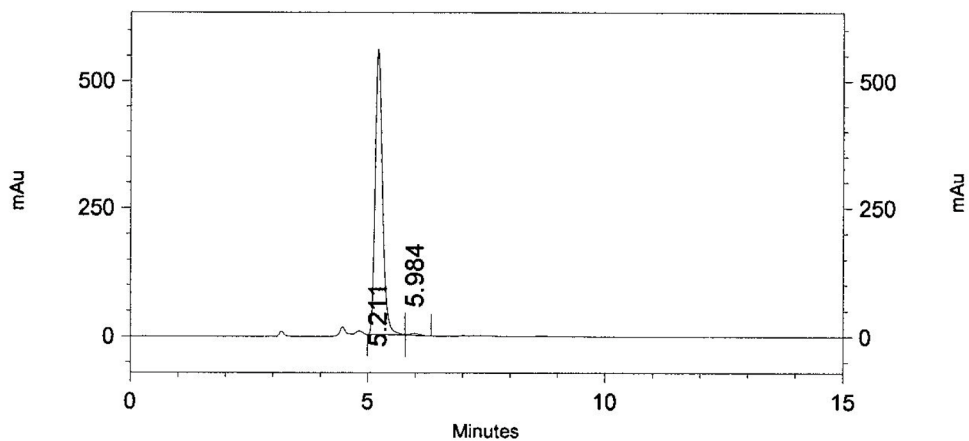
Retention Time	Area	Area Percent
10.784	26014496	99.109
11.904	233920	0.891





2: 254 nm, 4 nm Results

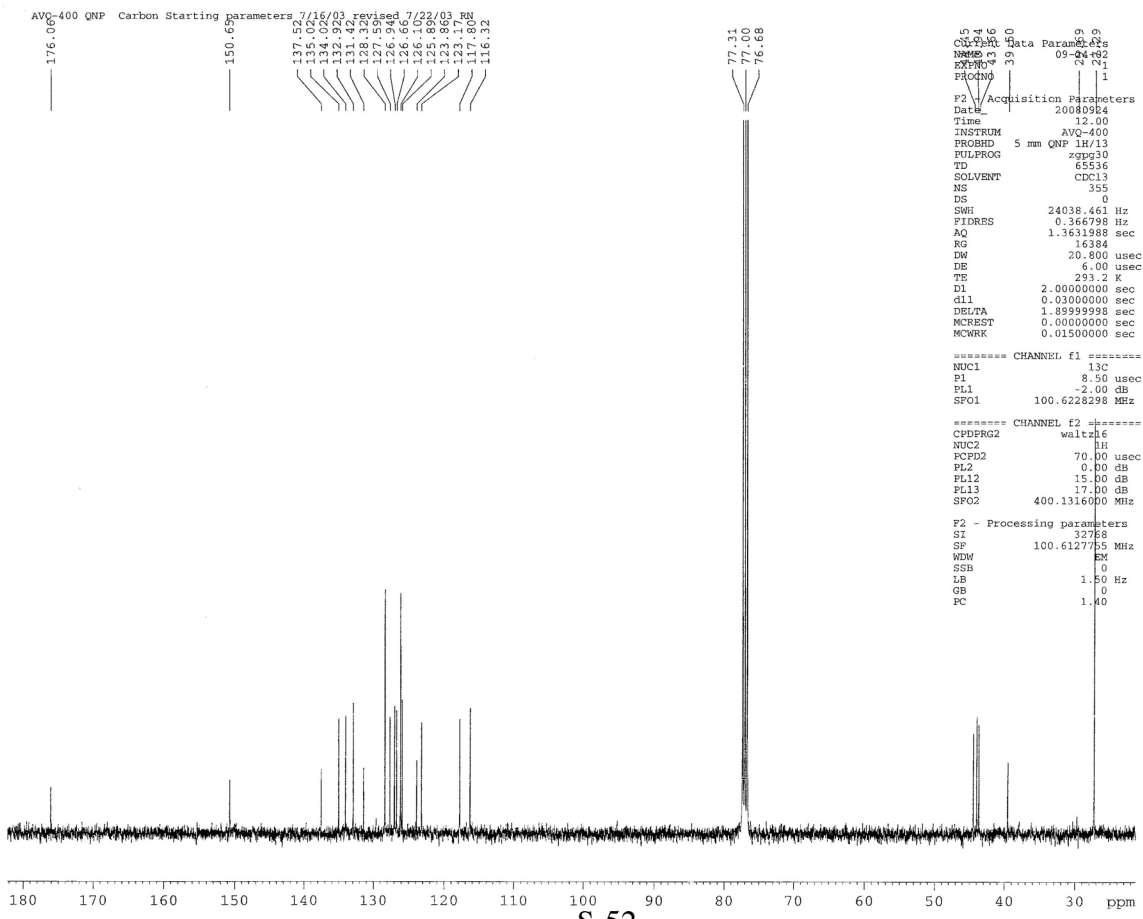
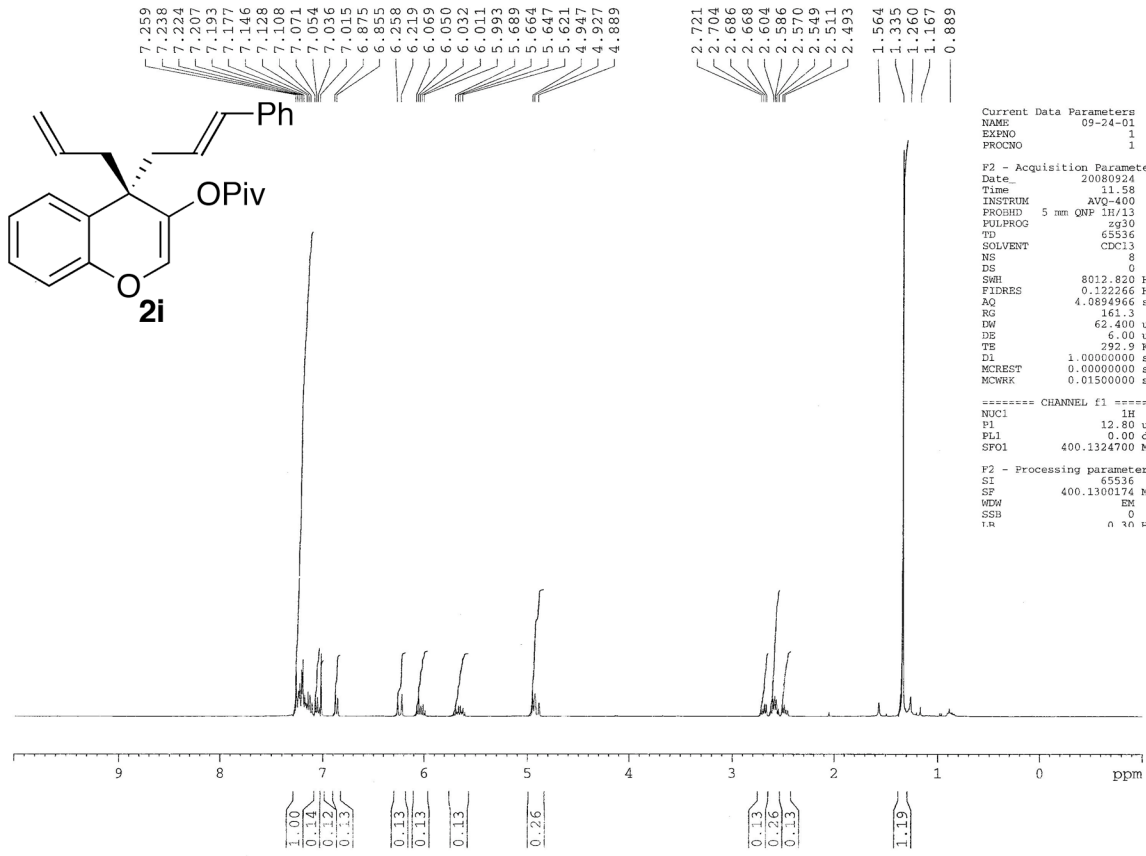
Retention Time	Area	Area Percent
5.211	2114122	49.593
6.021	2148860	50.407

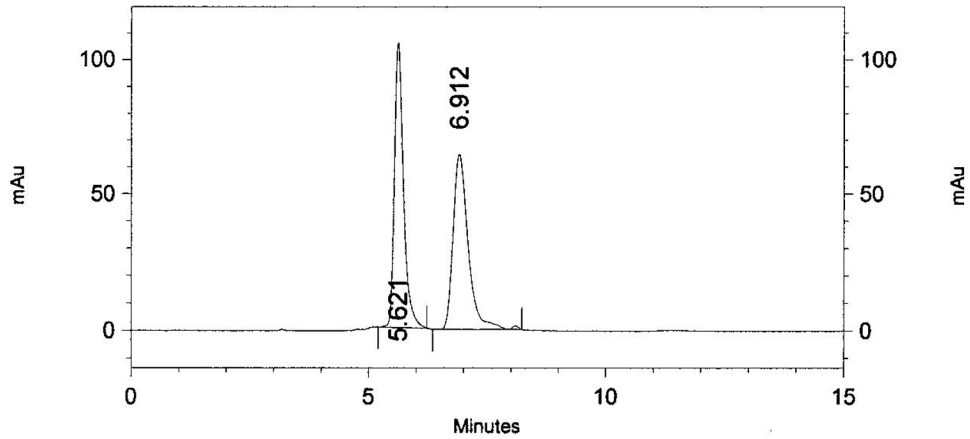
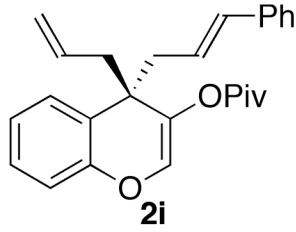


2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
5.211	5974377	99.265
5.984	44221	0.735

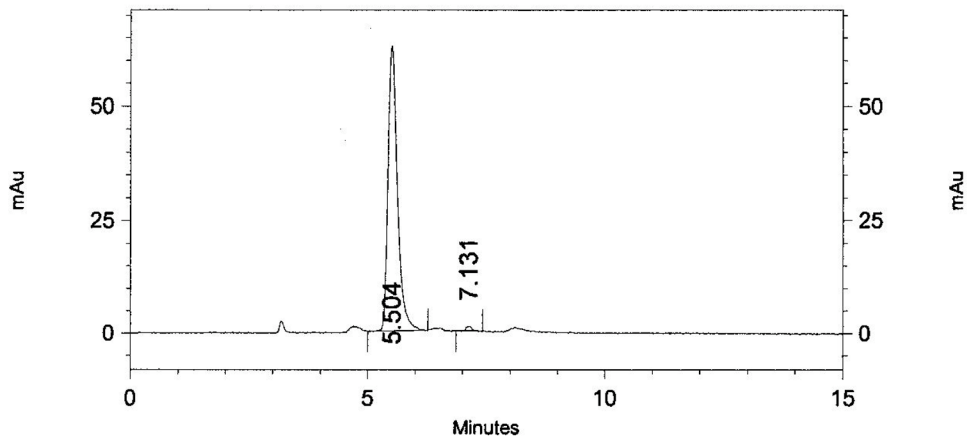
AVQ-400 QNP Proton starting parameters. 7/16/03. Revised 7/22/03 RN





2: 254 nm, 4 nm Results

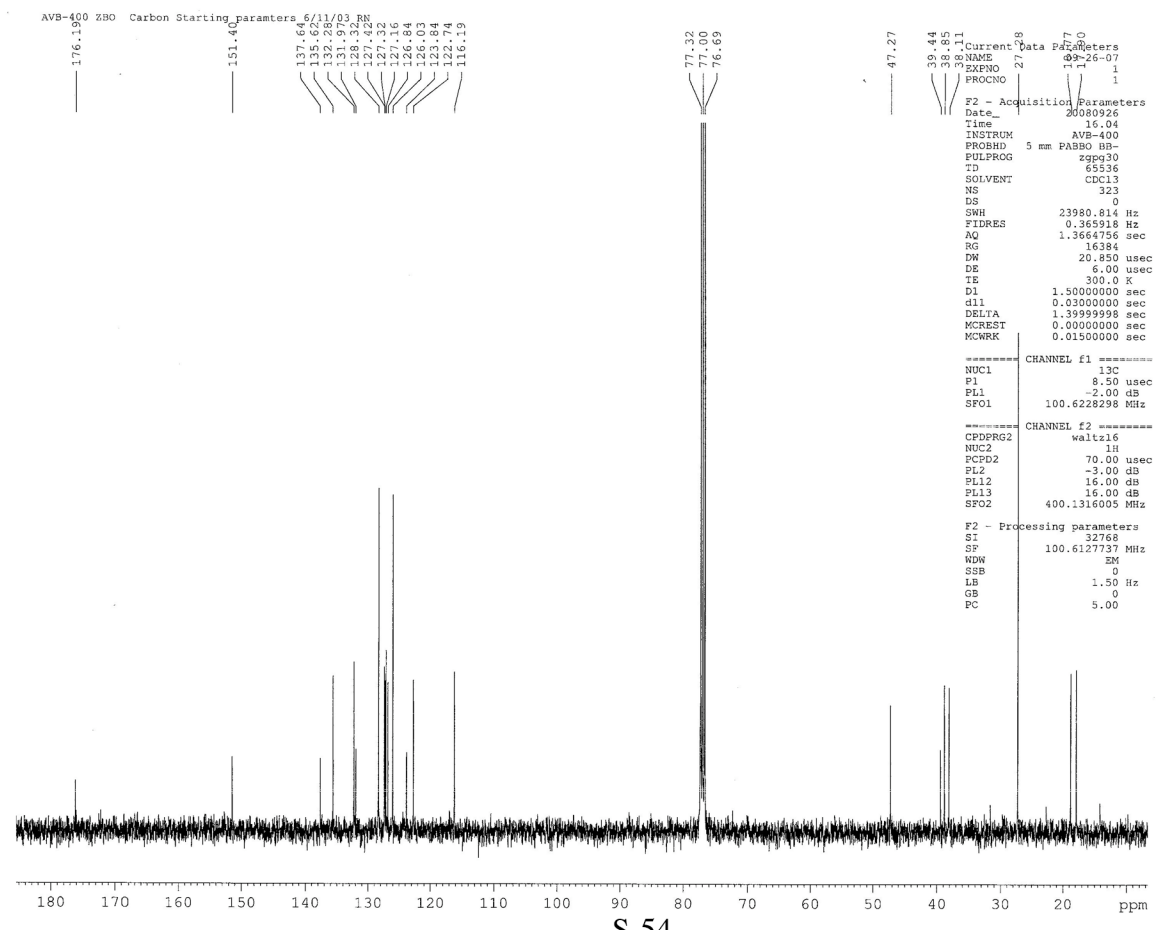
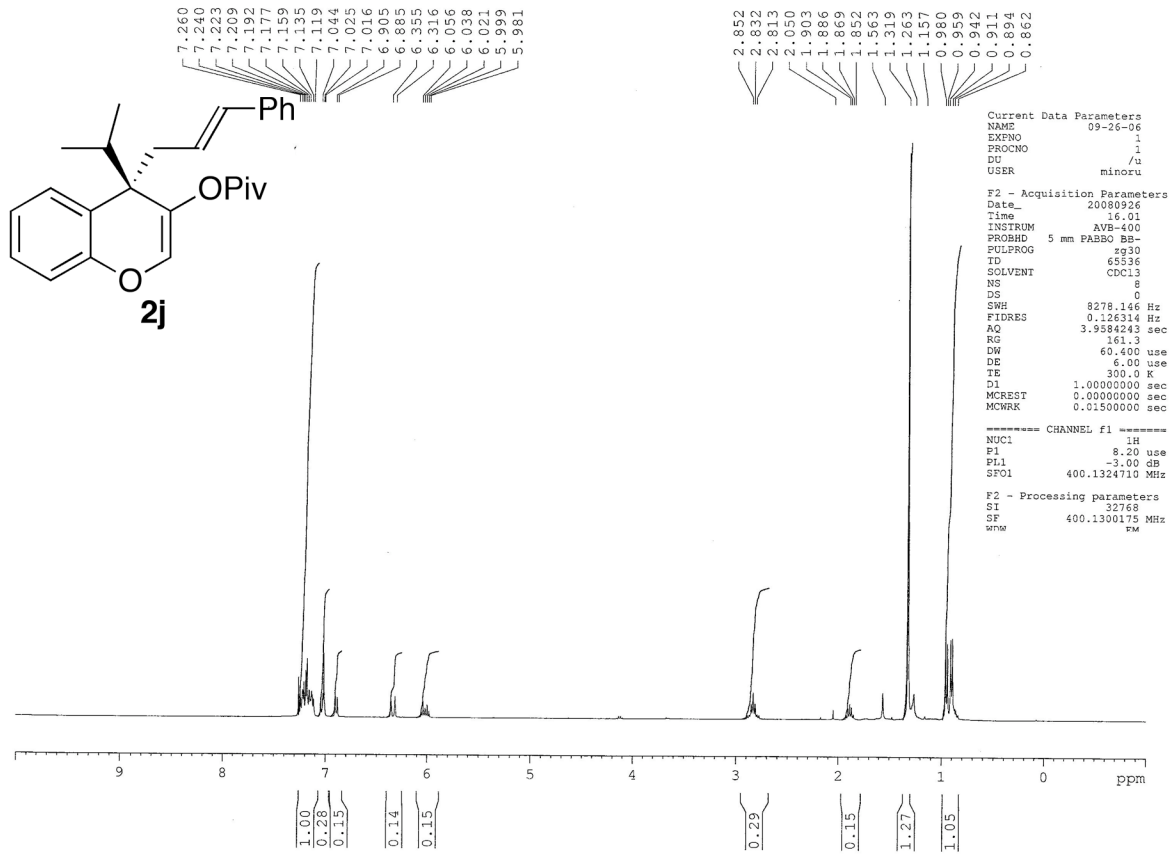
Retention Time	Area	Area Percent
5.621	1393908	49.718
6.912	1409746	50.282

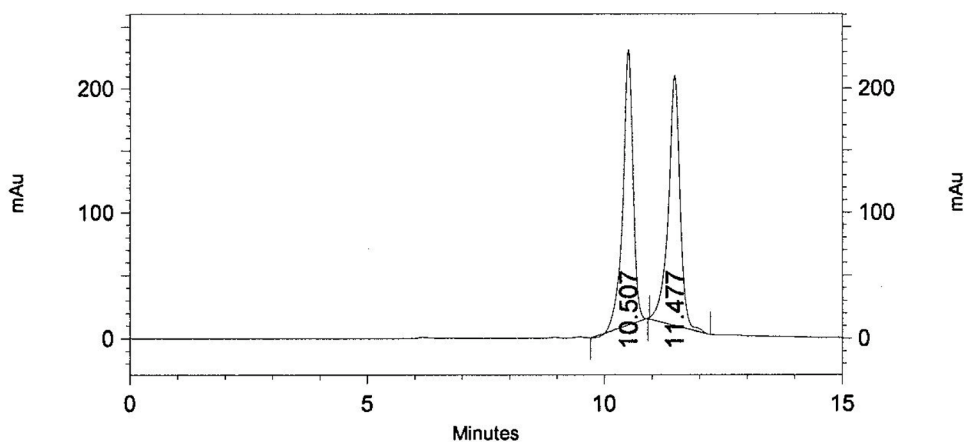
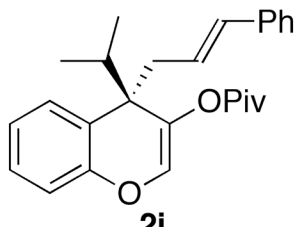


3: 280 nm, 4 nm Results

Retention Time	Area	Area Percent
5.504	871137	99.318
7.131	5986	0.682

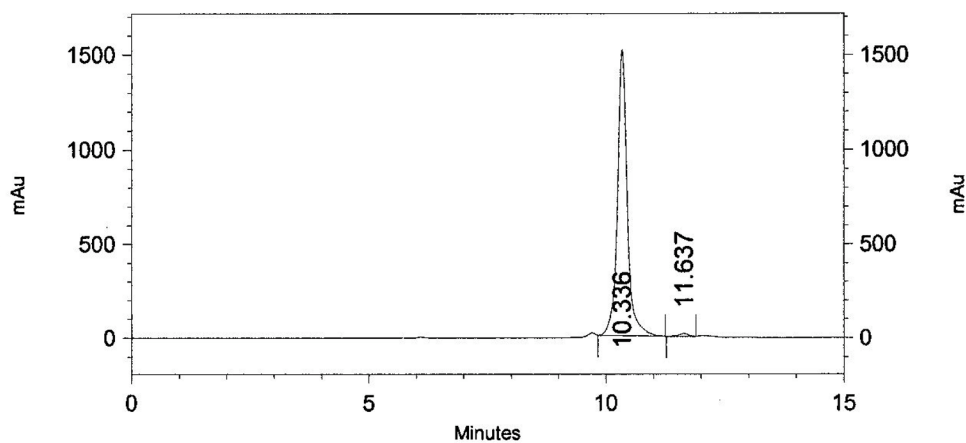
AVB-400 ZBO Proton starting parameters. 6/11/03 RN





2: 254 nm, 4 nm Results

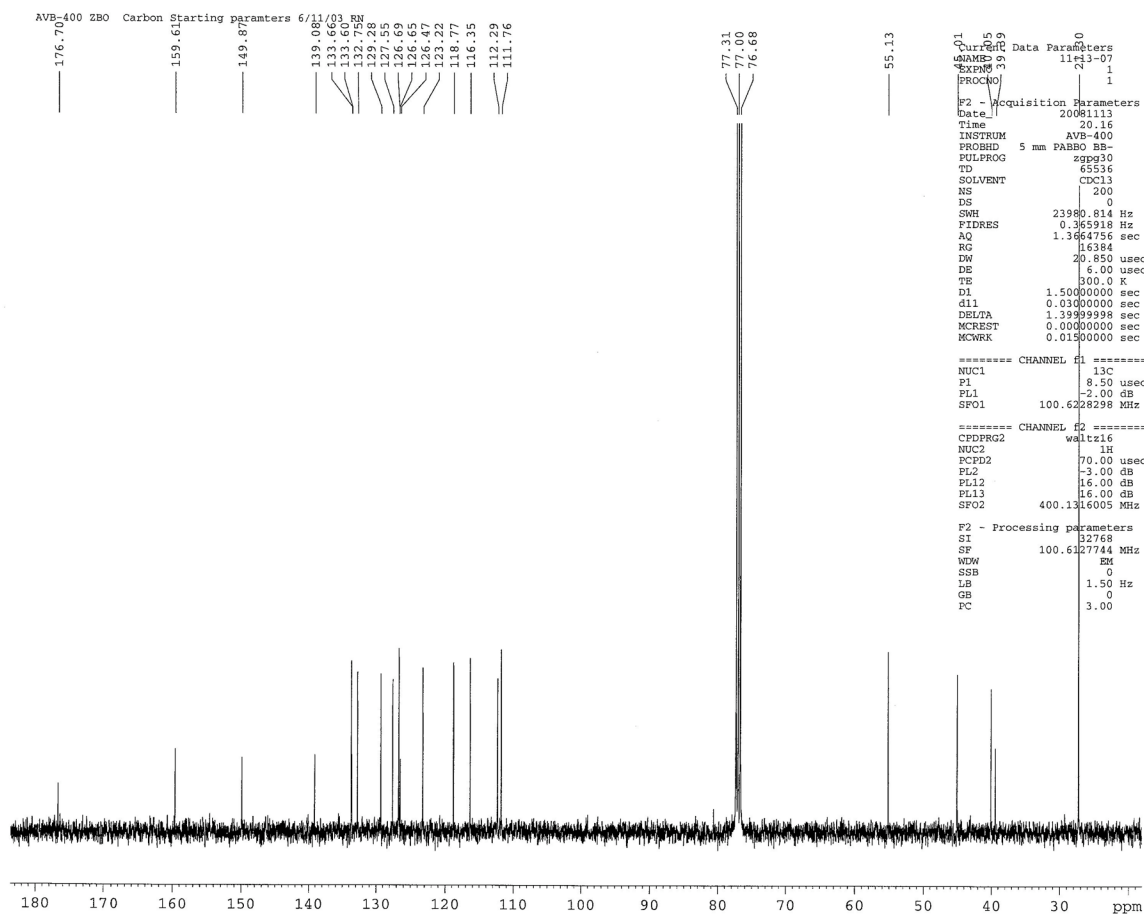
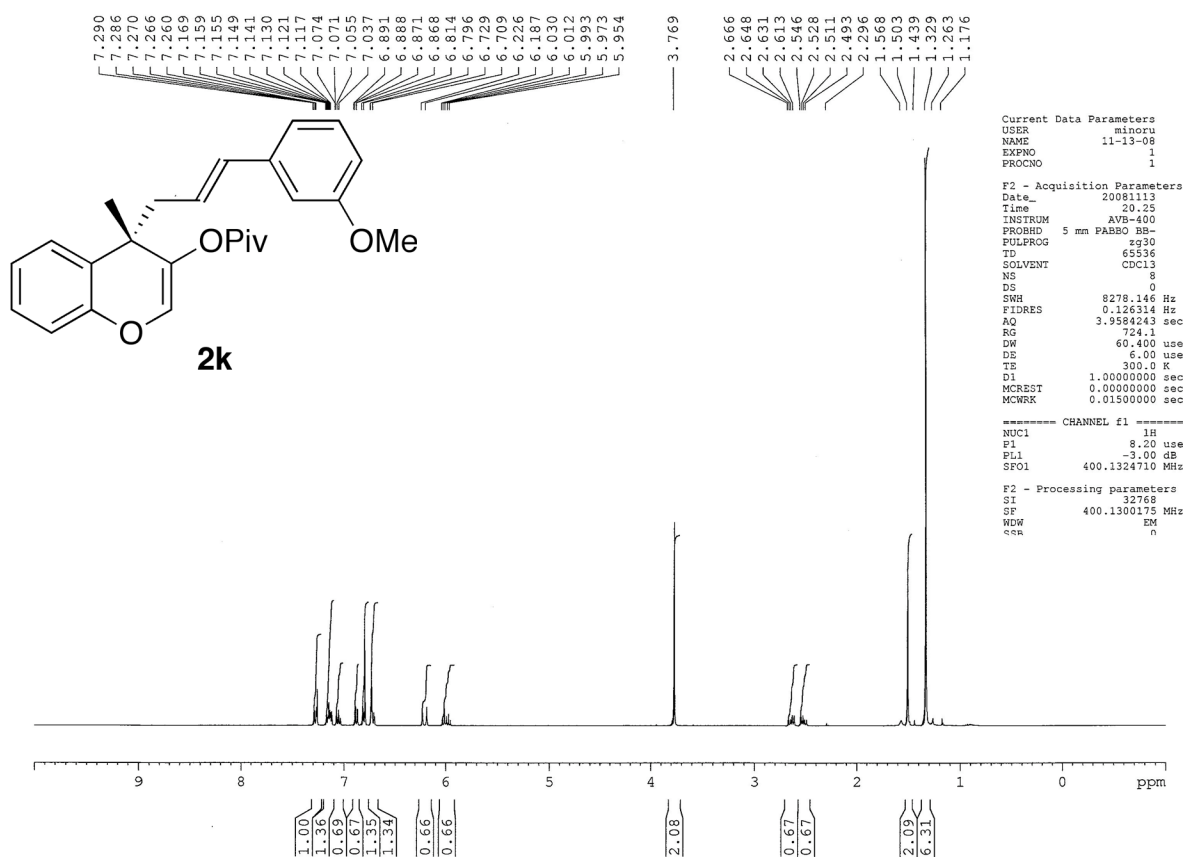
Retention Time	Area	Area Percent
10.507	3257190	48.857
11.477	3409543	51.143

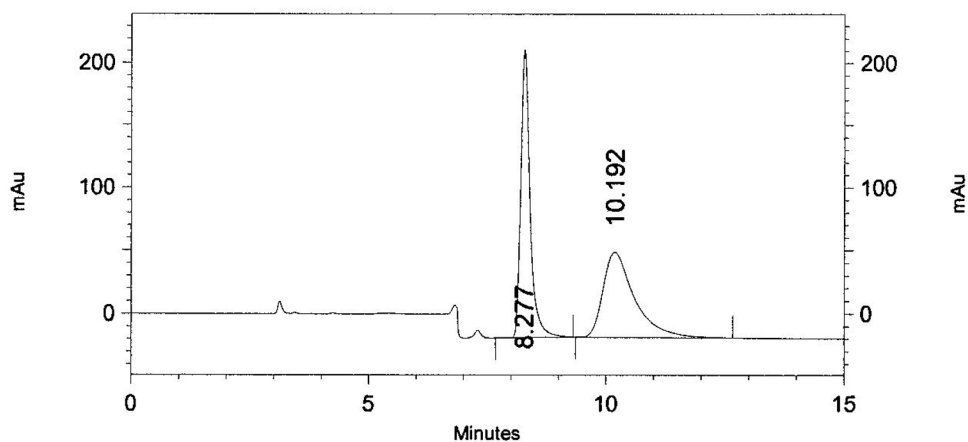
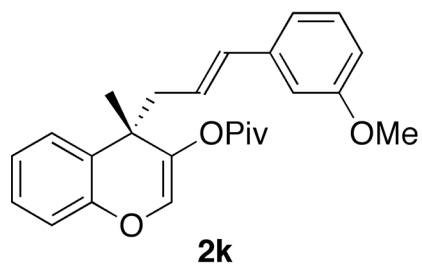


2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
10.336	21756522	99.020
11.637	215357	<u>0.980</u>

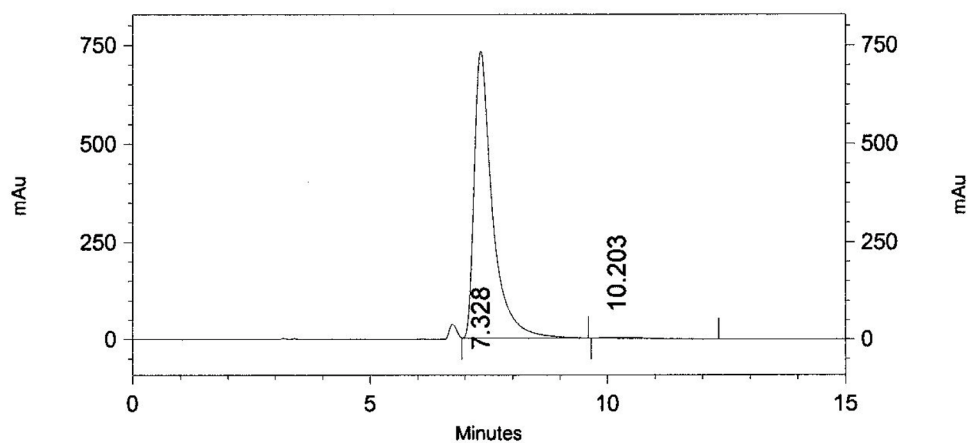
AVB-400 ZBO Proton starting parameters. 6/11/03 RN





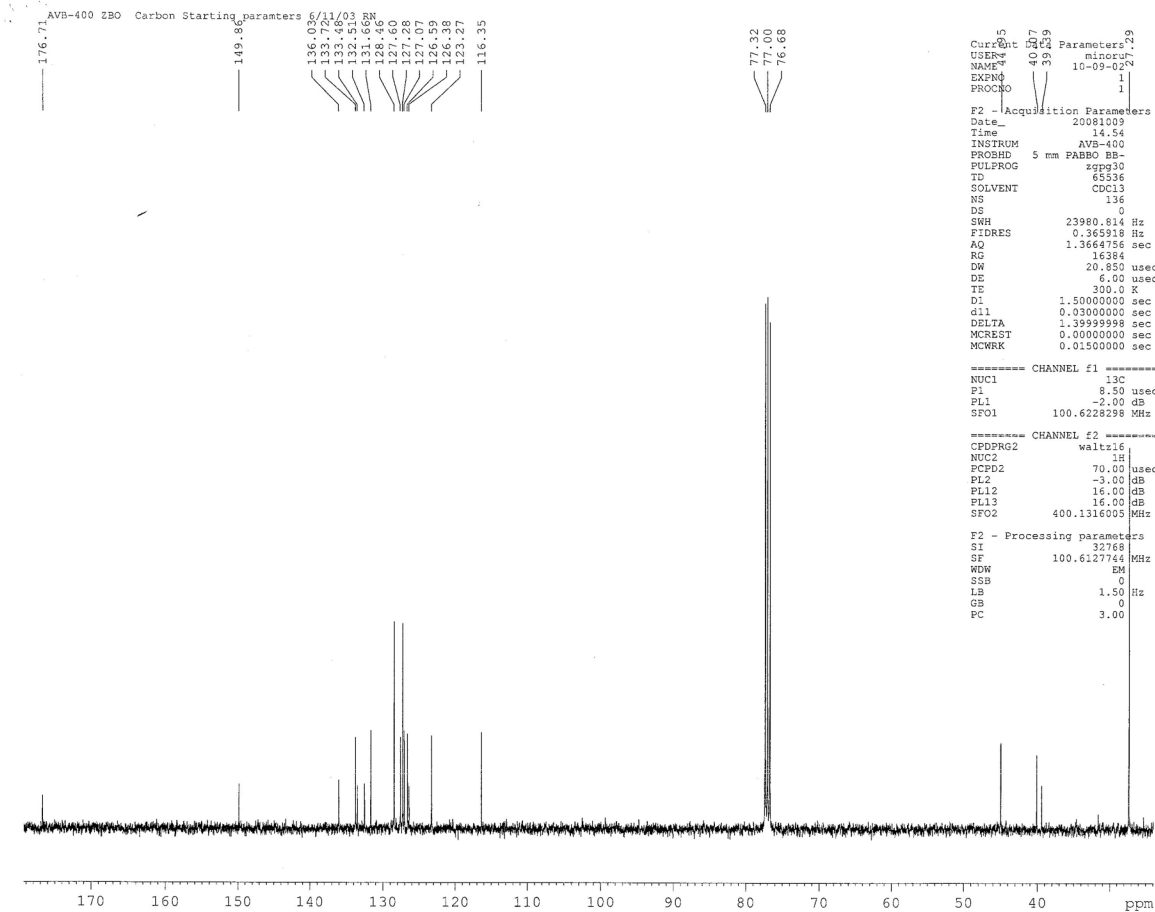
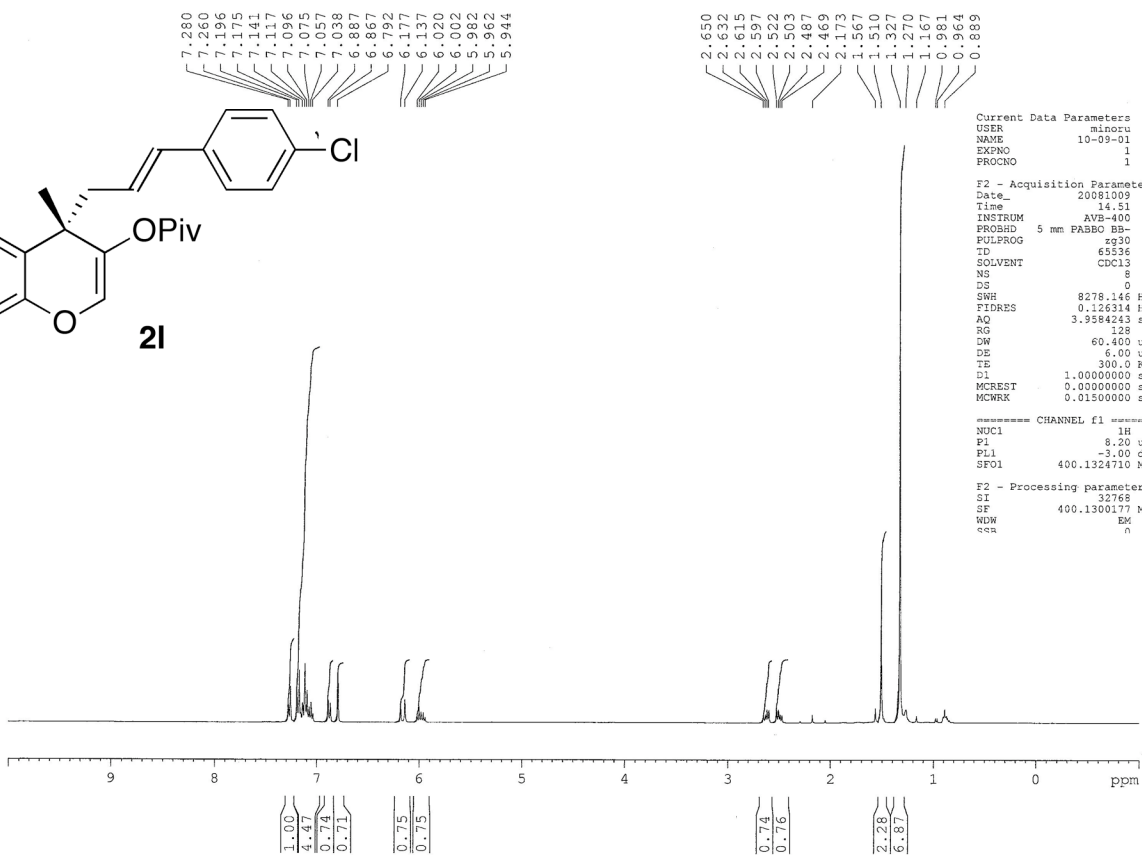
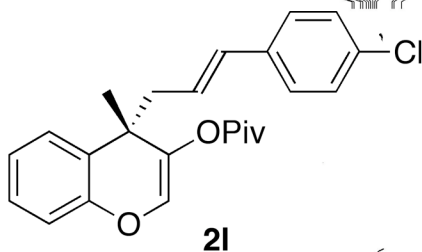
2: 255 nm, 4 nm Results

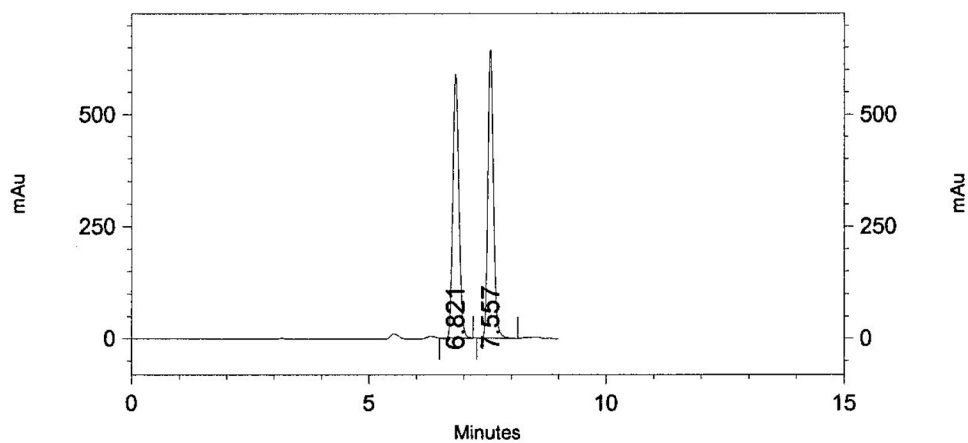
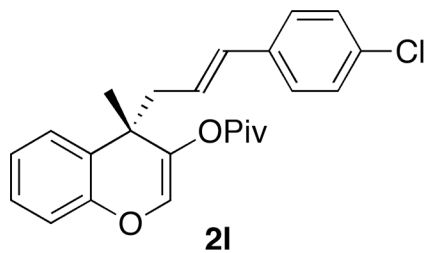
Retention Time	Area	Area Percent
8.277	3109804	49.924
10.192	3119221	50.076



2: 254 nm, 4 nm Results

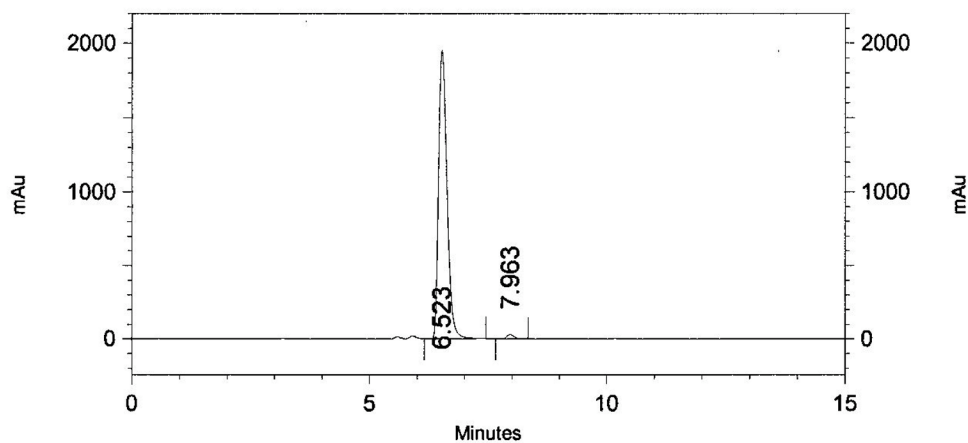
Retention Time	Area	Area Percent
7.328	19559311	99.399
10.203	118348	0.601





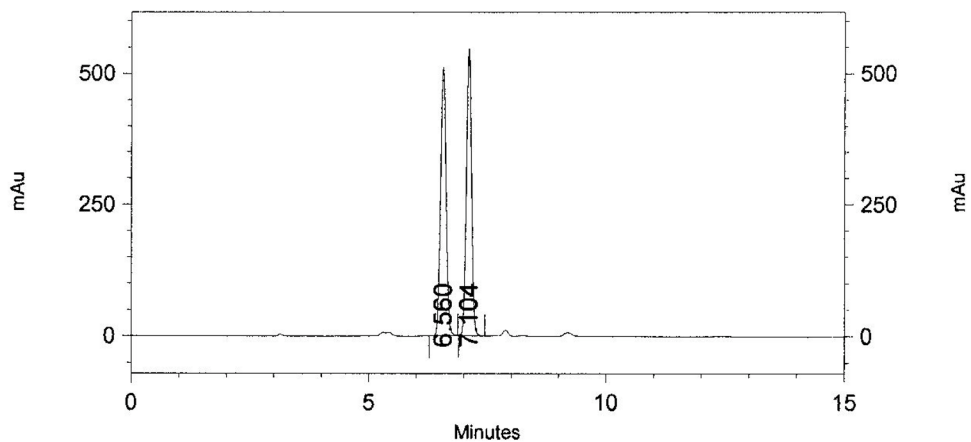
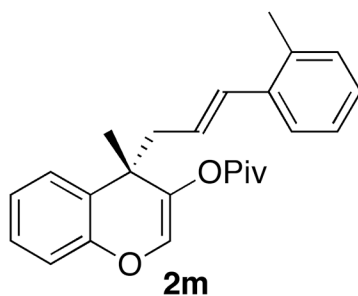
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
6.821	5240433	49.948
7.557	5251357	50.052



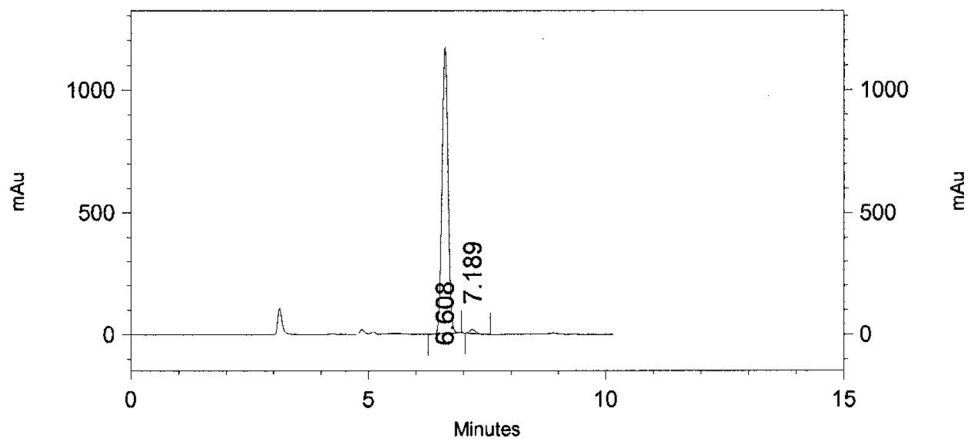
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
6.523	24219402	98.784
7.963	298152	1.216



2: 254 nm, 4 nm Results

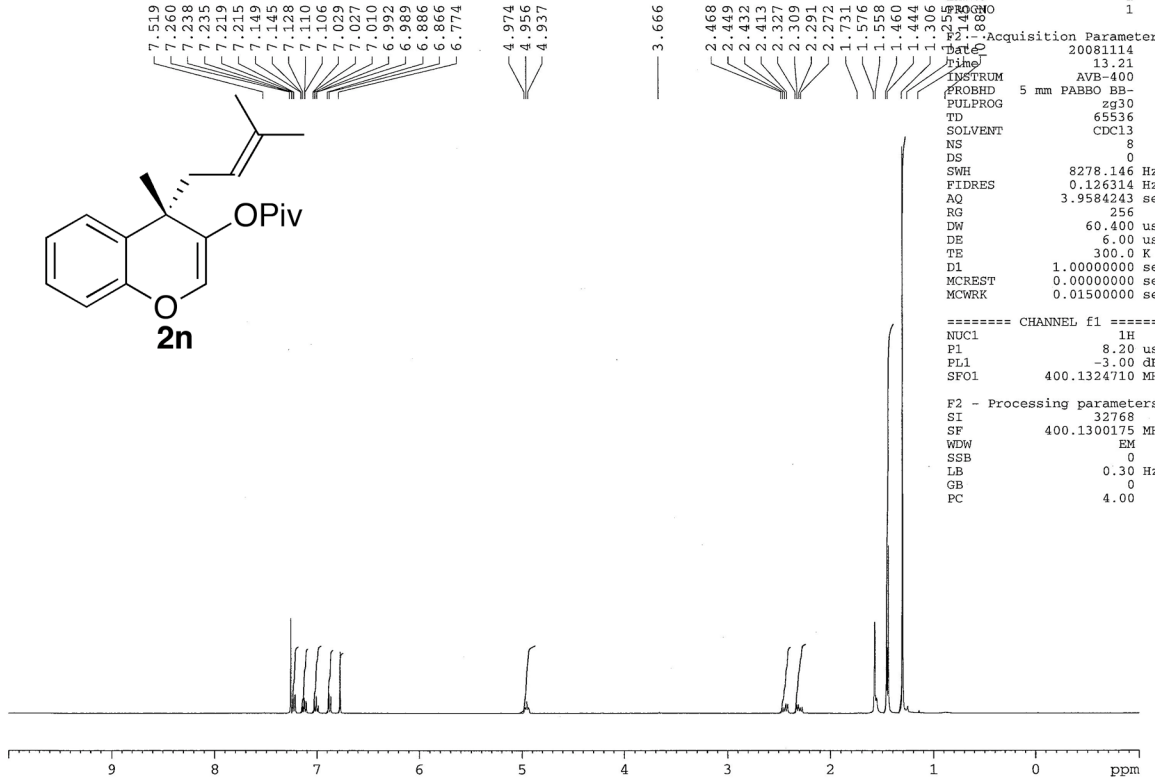
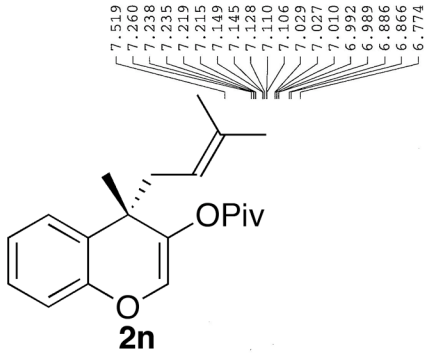
Retention Time	Area	Area Percent
6.560	4122356	49.921
7.104	4135365	50.079



1: 230 nm, 4 nm Results

Retention Time	Area	Area Percent
6.608	10353667	98.756
7.189	130393	1.244

AVB-400 ZBO Proton starting paramet



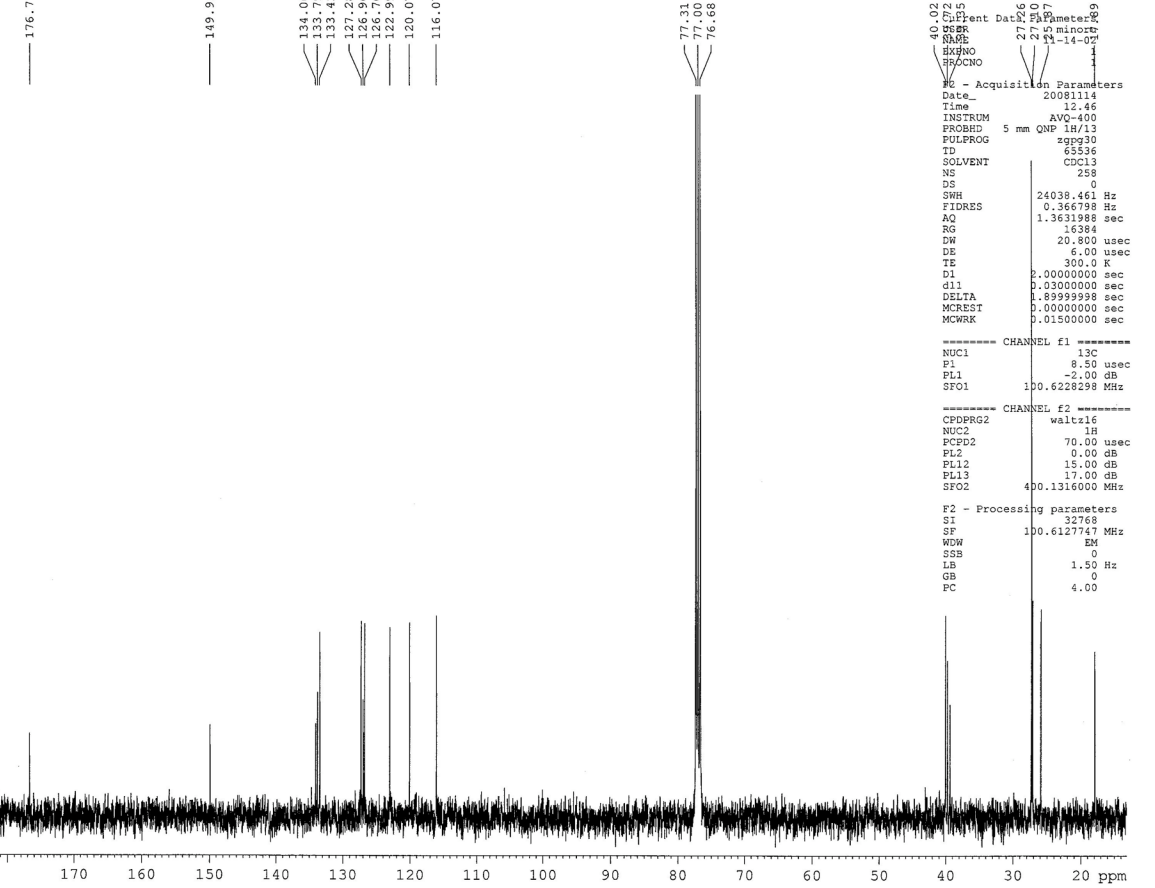
```

Current Data Parameters
NAME      11-14-03
EXPNO     1
PROCNO    1
F2 - Acquisition Parameter
Date_     20081114
Time      13.21
INSTRUM   AVB-400
PROBHD    5 mm PABBO BA-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 se
RG         256
DW         60.400 us
DE         6.00 us
TE         300.0 K
D1         1.0000000 se
MCREST    0.0000000 se
MCWRK     0.0150000 se

===== CHANNEL f1 =====
NUC1      1H
P1        8.20 us
PL1       -3.00 dB
SFO1     400.1324710 MHz

F2 - Processing parameters
SI        32768
SF        400.1300175 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        4.00
    
```

AVC-400 QNP Carbon Starting parameters



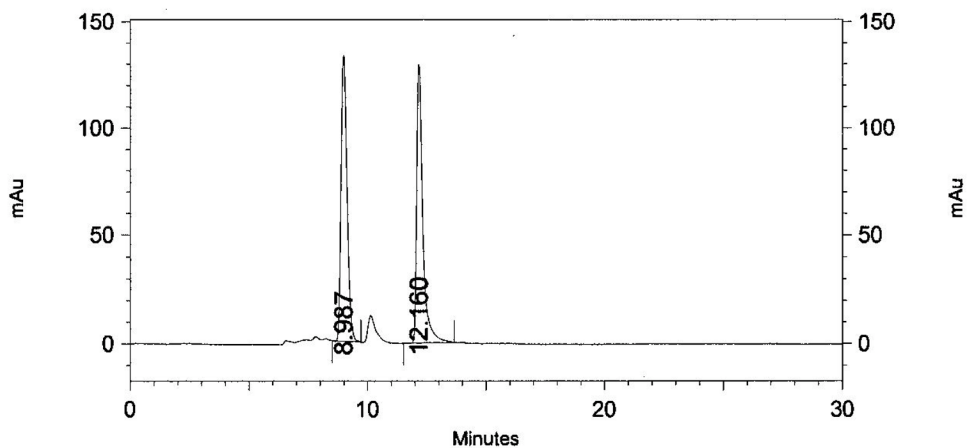
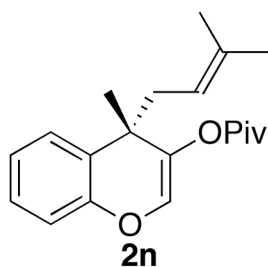
```

AVC-400 QNP Carbon Starting parameters
176.742
149.8
134.06
133.796
133.423
127.285
126.964
122.99
120.072
116.073
77.31
77.00
76.68

===== CHANNEL f1 =====
NUC1      13C
P1        8.50 usec
PL1       -2.00 dB
SFO1     100.6228238 MHz

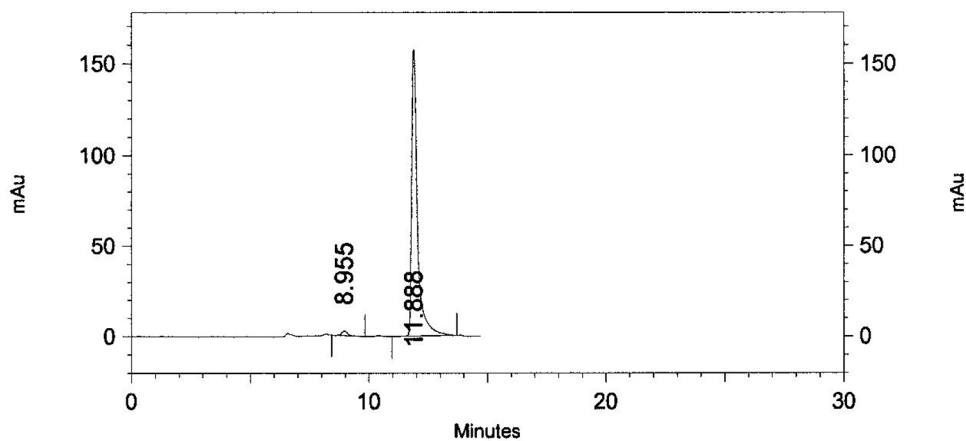
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     70.00 usec
PL2       0.00 dB
PL12      15.00 dB
PL13      17.00 dB
SFO2     400.1316000 MHz

F2 - Processing parameters
SI        32768
SF        100.6127747 MHz
WDW       EM
SSB       0
LB        1.50 Hz
GB        0
PC        4.00
    
```



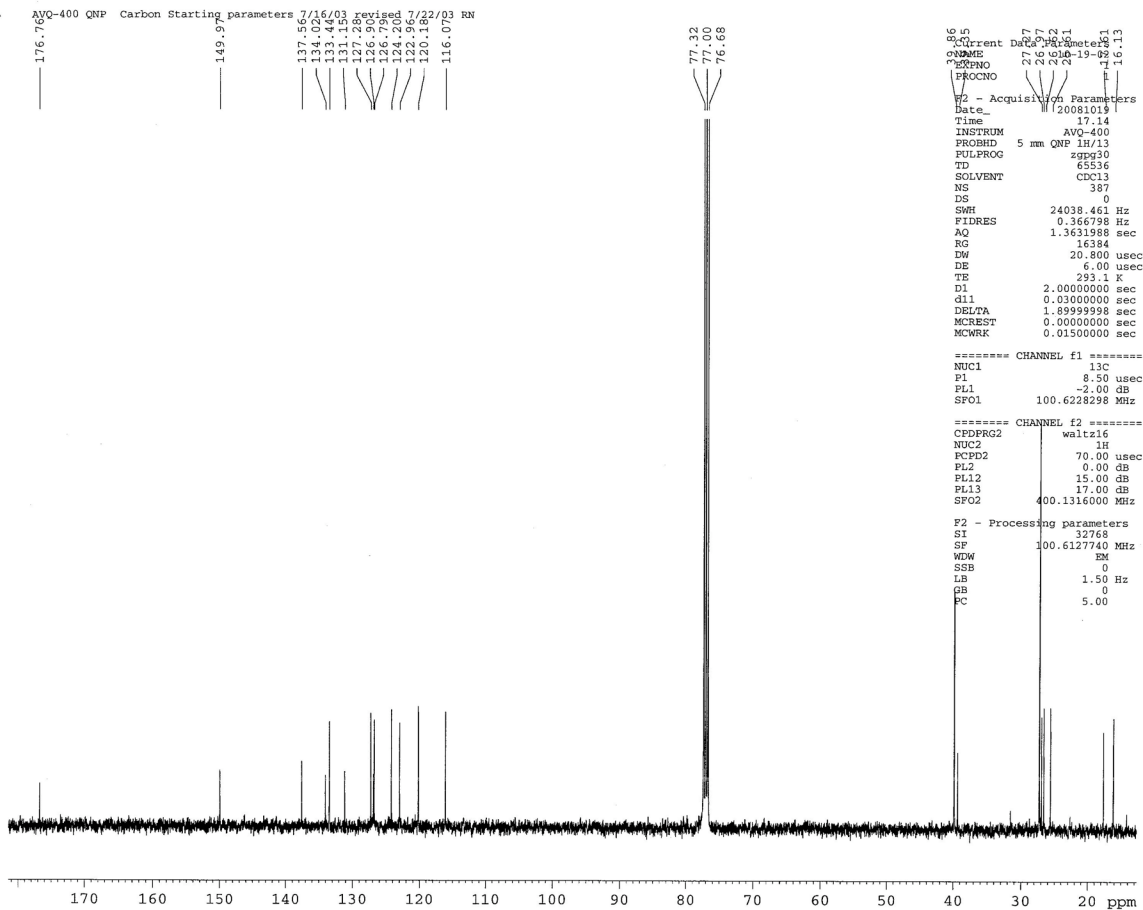
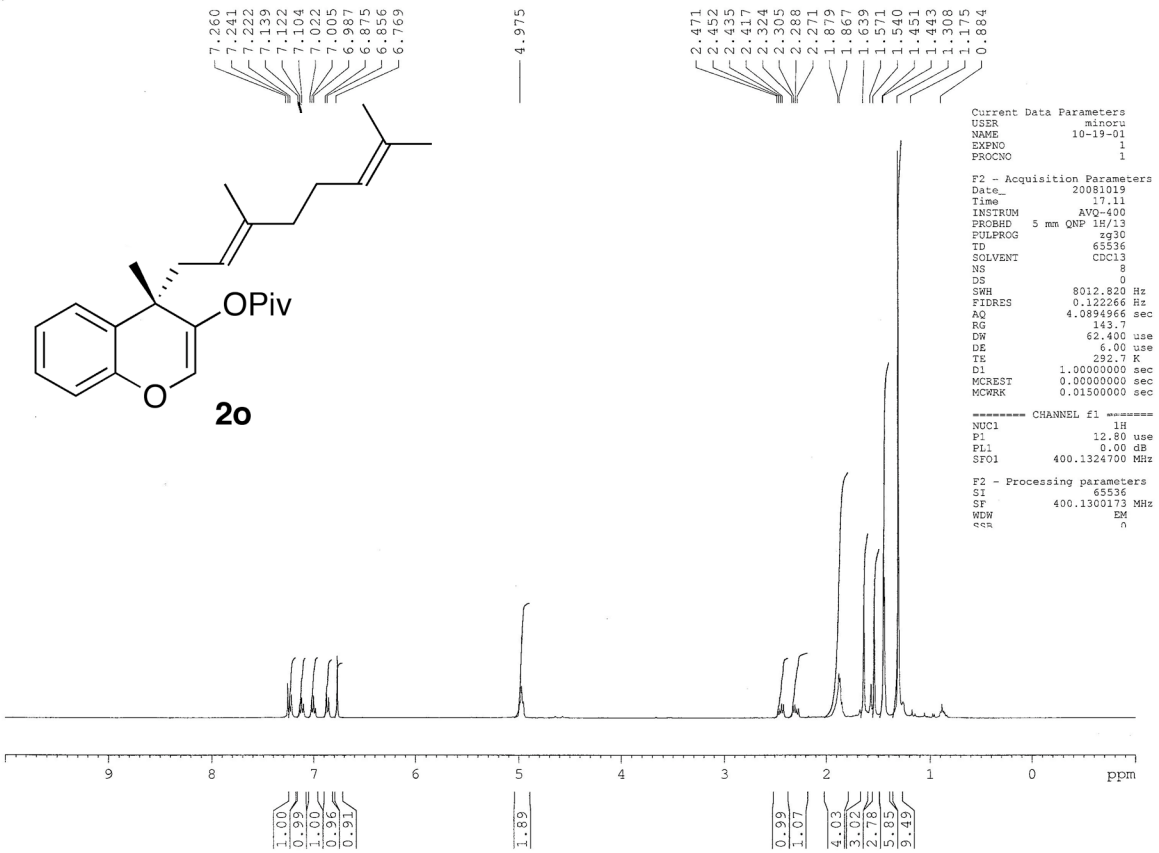
2: 254 nm, 4 nm Results

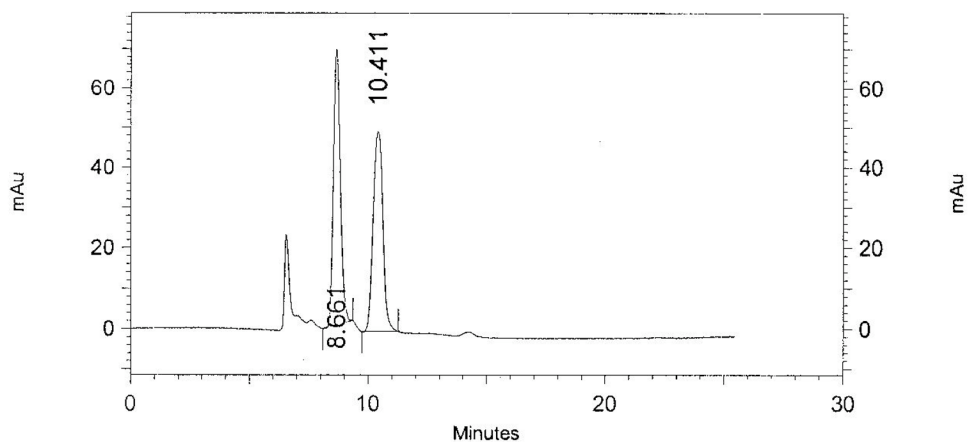
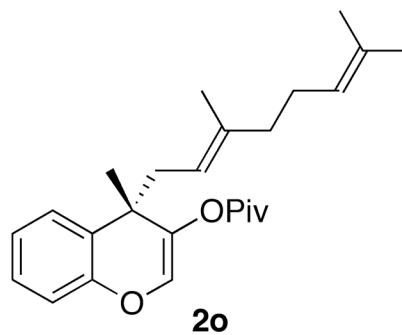
Retention Time	Area	Area Percent
8.987	2321202	50.284
12.160	2295000	49.716



2: 254 nm, 4 nm Results

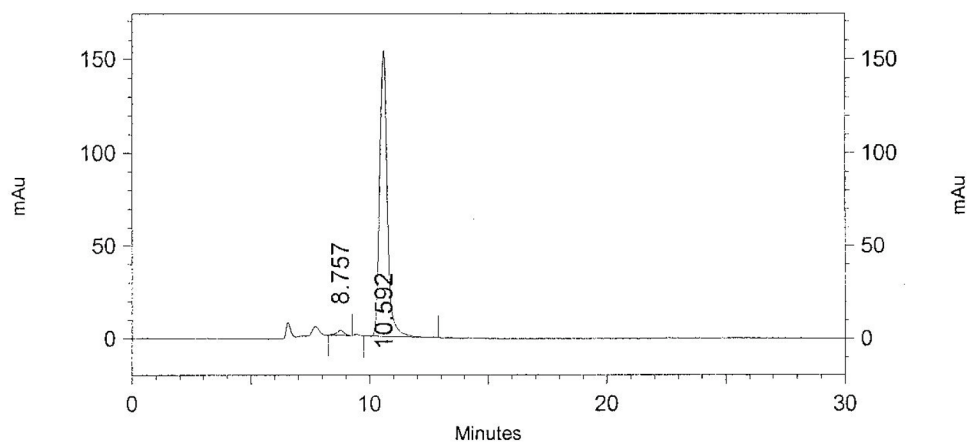
Retention Time	Area	Area Percent
8.955	39868	1.469
11.888	2674711	98.531





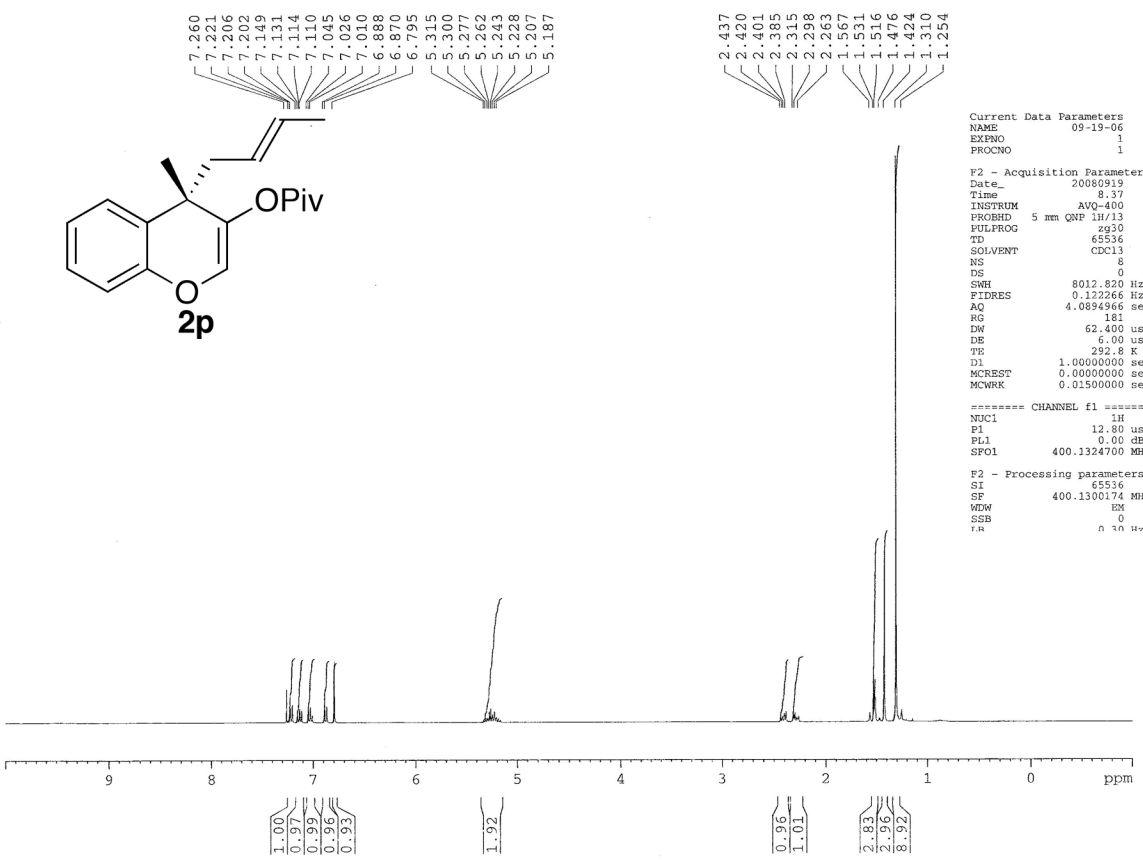
3: 280 nm, 4 nm Results

Retention Time	Area	Area Percent
8.661	1386849	49.648
10.411	1406531	50.352



3: 280 nm, 4 nm Results

Retention Time	Area	Area Percent
8.757	61363	1.753
10.592	3439047	98.247



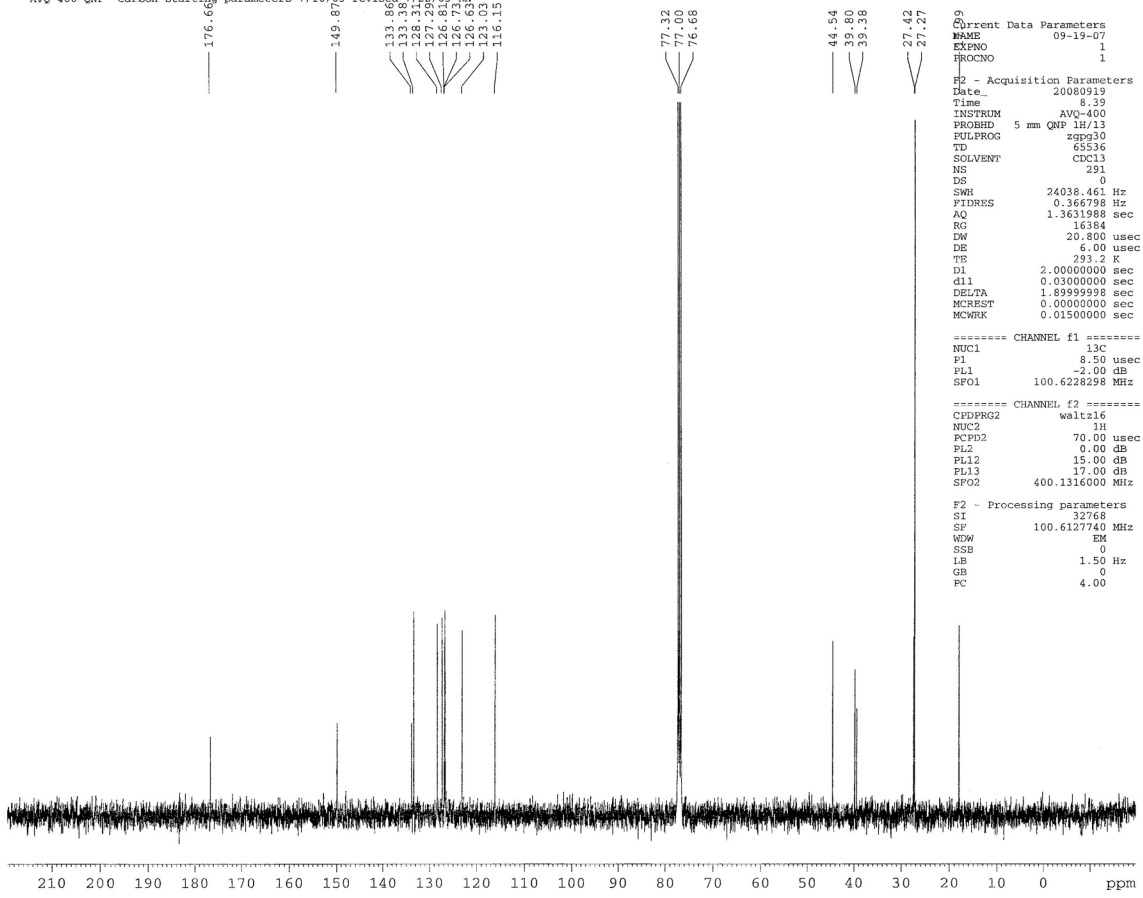
```

Current Data Parameters
NAME      09-19-06
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20080919
Time     8.37
INSTRUM  AVQ-400
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0834966 sec
RG       181
DW       62.400 usec
DE       6.00 usec
TE       293.2 K
D1       1.0000000 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1     1H
P1       12.80 usec
PL1      0.00 dB
SFO1     400.1324700 MHz

F2 - Processing parameters
SI       65536
SF       400.1300174 MHz
WDW      EM
SSB      0
GB       0
PC       0.00 usec
    
```



```

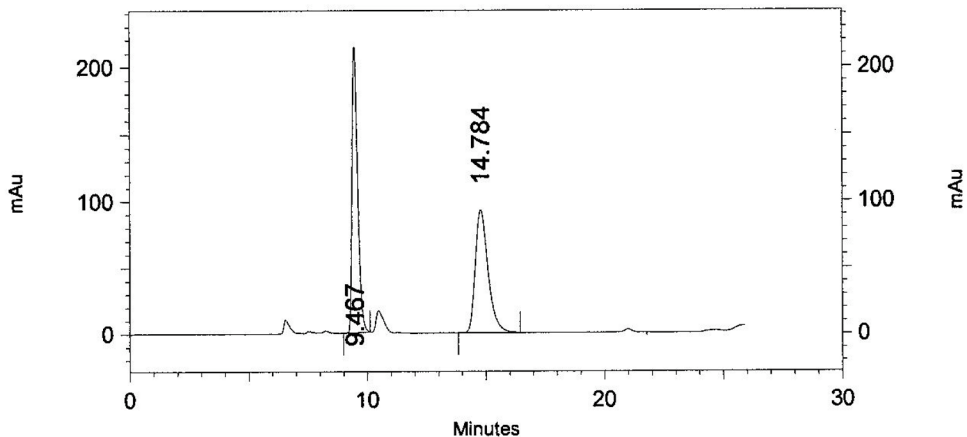
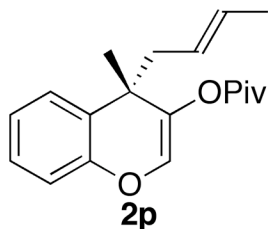
Current Data Parameters
NAME      09-19-07
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20080919
Time     8.39
INSTRUM  AVQ-400
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       291
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       16384
DW       20.800 usec
DE       6.00 usec
TE       293.2 K
D1       2.0000000 sec
d11      0.0300000 sec
DELTA    1.8999998 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1     13C
P1       8.50 usec
PL1      -2.00 dB
SFO1     100.6228298 MHz

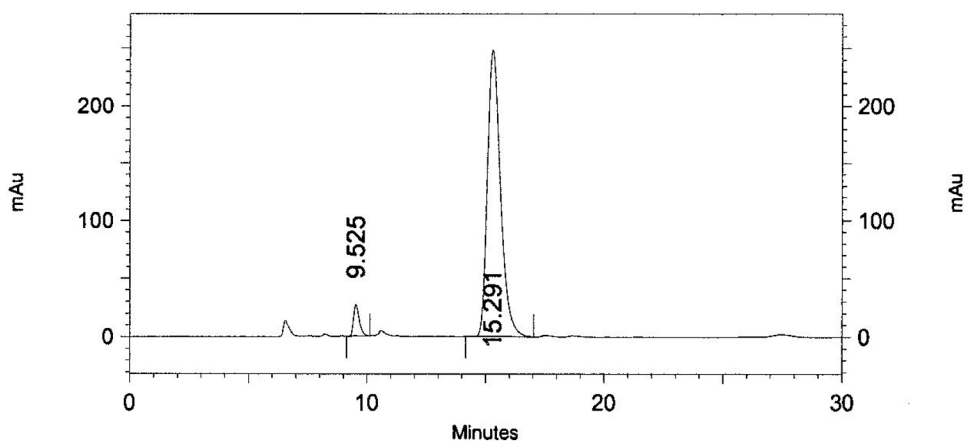
===== CHANNEL f2 =====
CHOPROG2 waltz16
NUC2     1H
PCPD2    70.00 usec
PL2      0.00 dB
PL12     15.00 dB
PL13     17.00 dB
SFO2     400.1316000 MHz

F2 - Processing parameters
SI       32768
SF       100.6127740 MHz
WDW      EM
SSB      0
LB       1.50 Hz
GB       0
PC       4.00
    
```



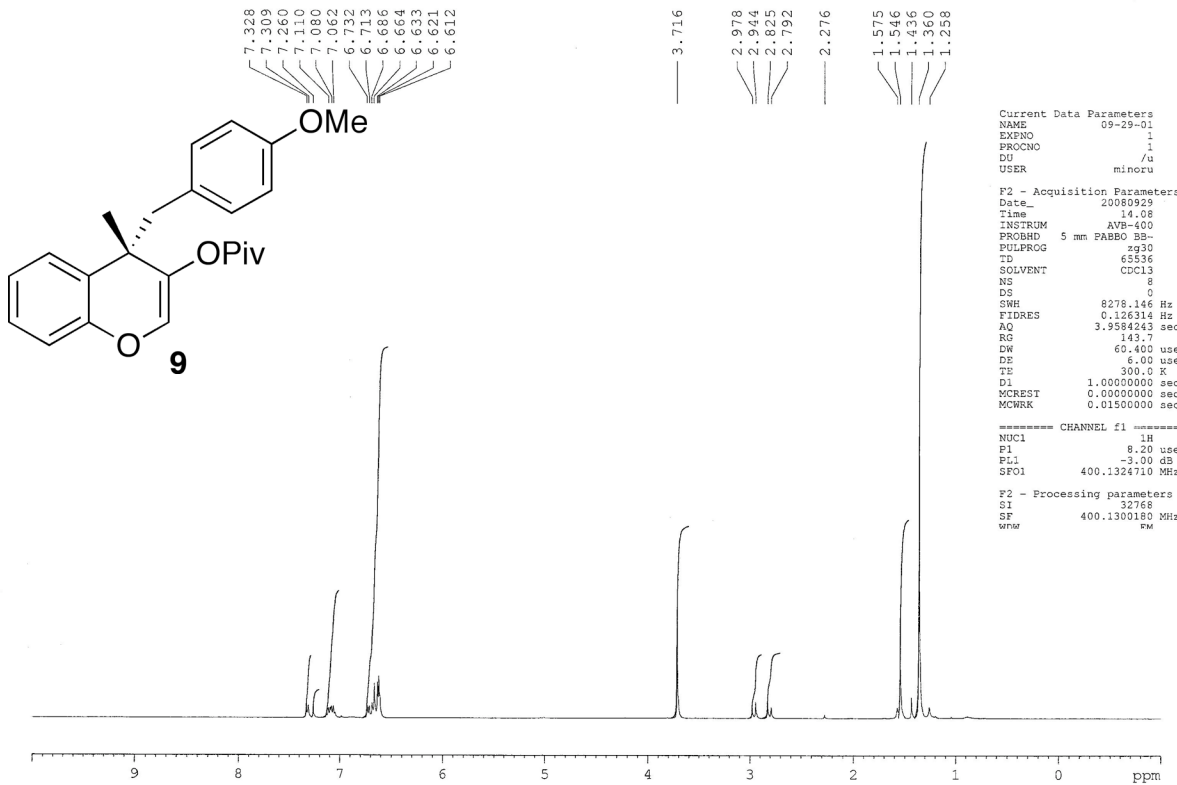
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
9.467	3550479	50.527
14.784	3476408	49.473



2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
9.525	449897	4.512
15.291	9520879	95.488



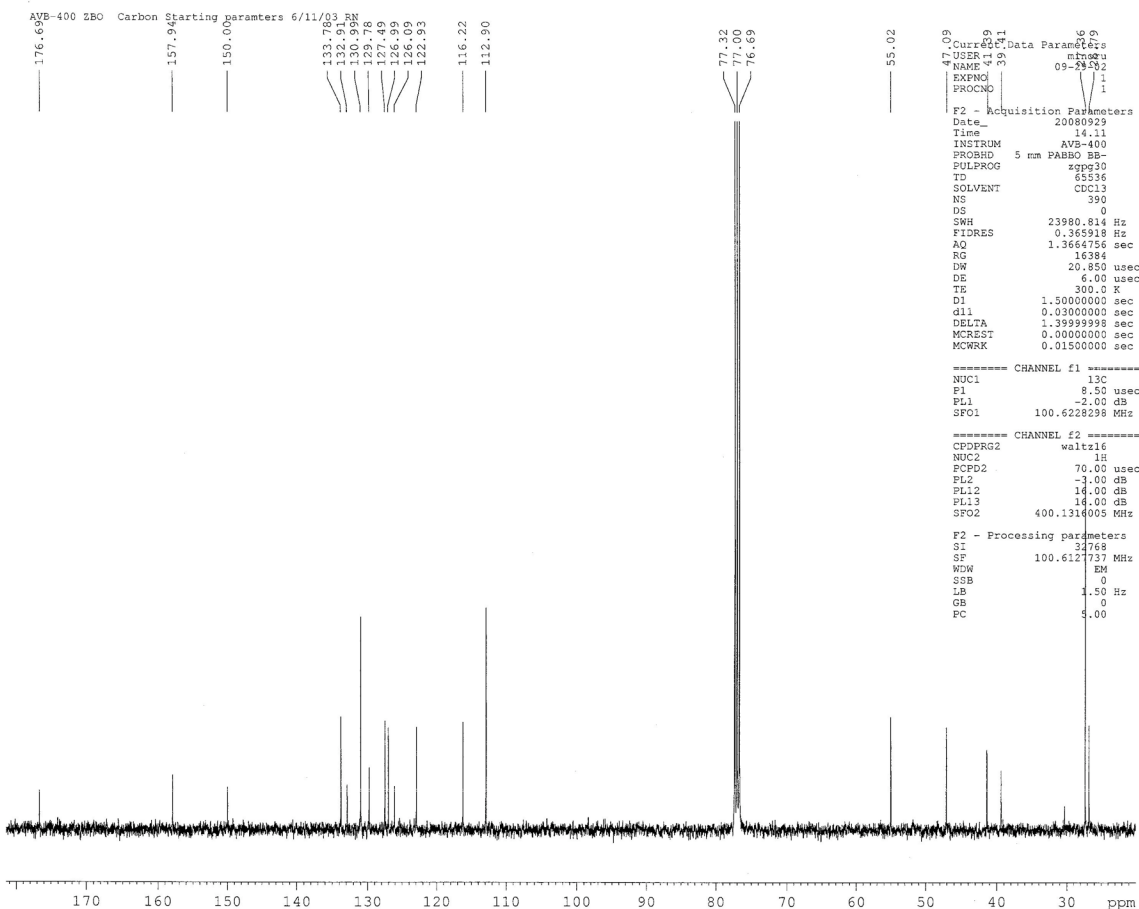
```

Current Data Parameters
NAME      09-29-01
EXPNO    1
PROCNO   1
DS       4
USER    minoru

F2 - Acquisition Parameters
Date_    20080929
Time     14.08
INSTRUM  AVB-400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       143.7
DW       60.400 usec
DE       6.00 usec
TE       300.0 K
D1       1.0000000 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        8.20 usec
PL1       -3.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI        32768
SF        400.1300180 MHz
WDW       EM
  
```



```

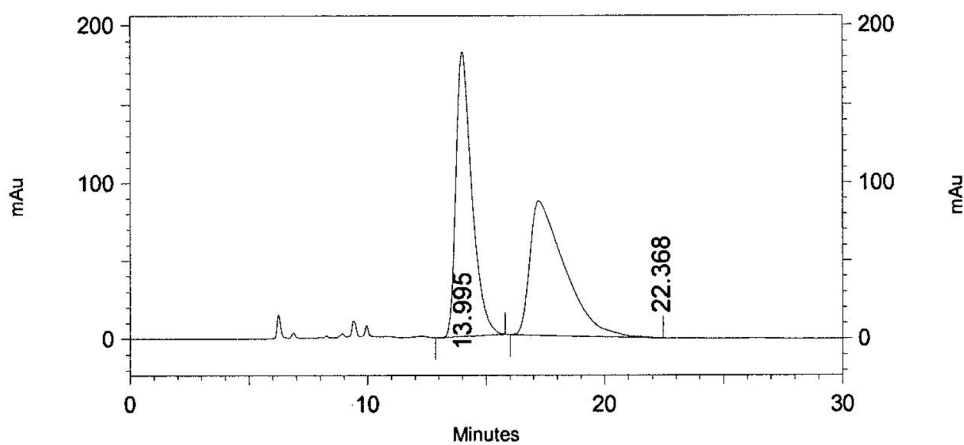
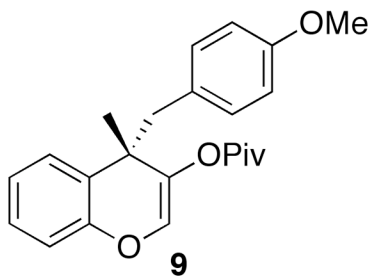
Current Data Parameters
NAME      09-29-01
EXPNO    1
PROCNO   1
DS       1
USER    minoru

F2 - Acquisition Parameters
Date_    20080929
Time     14.11
INSTRUM  AVB-400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       390
DS       0
SWH      23980.814 Hz
FIDRES   0.365918 Hz
AQ       1.3664756 sec
RG       16384
DW       20.850 usec
DE       6.00 usec
TE       300.0 K
D1       1.5000000 sec
d11      0.0300000 sec
DELTA    1.3999998 sec
MCREST   0.0000000 sec
MCWRK    0.0150000 sec

===== CHANNEL f1 =====
NUC1      13C
P1        8.50 usec
PL1       -2.00 dB
SFO1      100.6228298 MHz

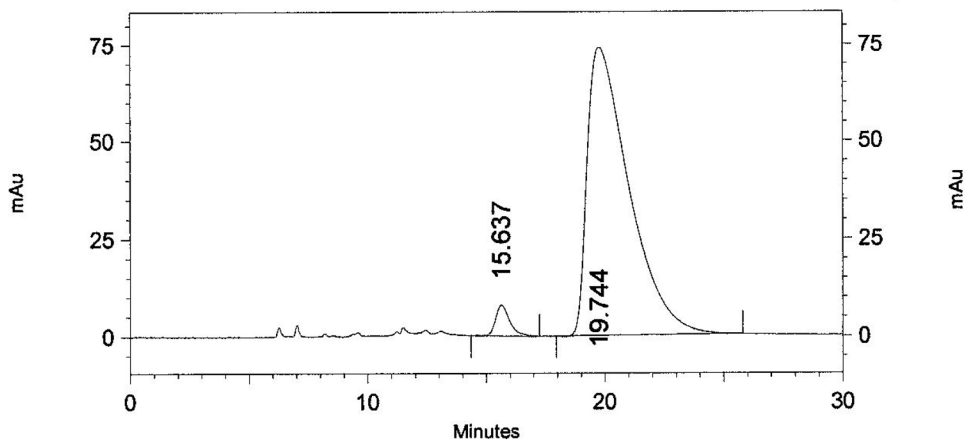
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    70.00 usec
PL2       3.00 dB
PL12     14.00 dB
PL13     14.00 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127737 MHz
WDW       EM
SSB       0
LB        1.50 Hz
GB        0
FC        5.00
  
```



2: 254 nm, 4 nm Results

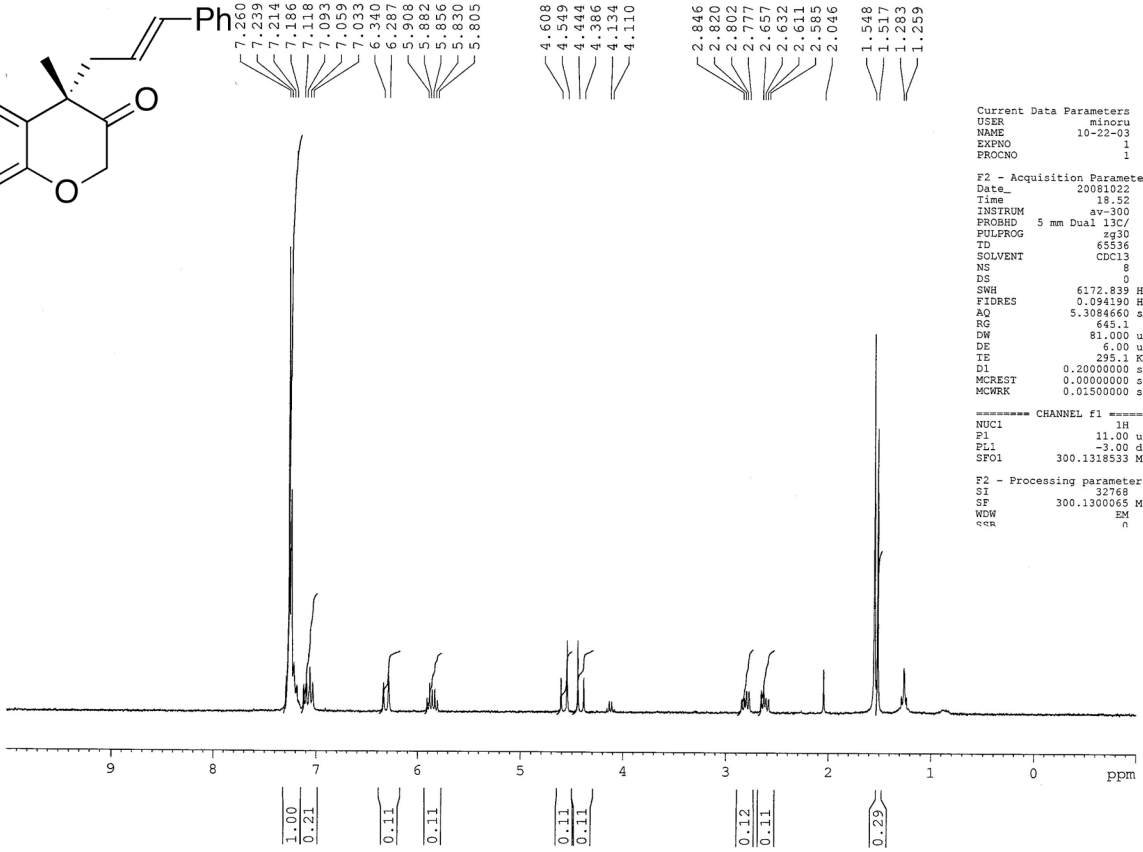
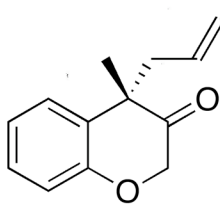
Retention Time	Area	Area Percent
13.995	8510996	49.276
22.368	8761126	50.724



2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
15.637	327511	3.495
19.744	9044023	96.505

A7-300 Dual C-H probe proton starting parameters 7/23/03 RN.



```

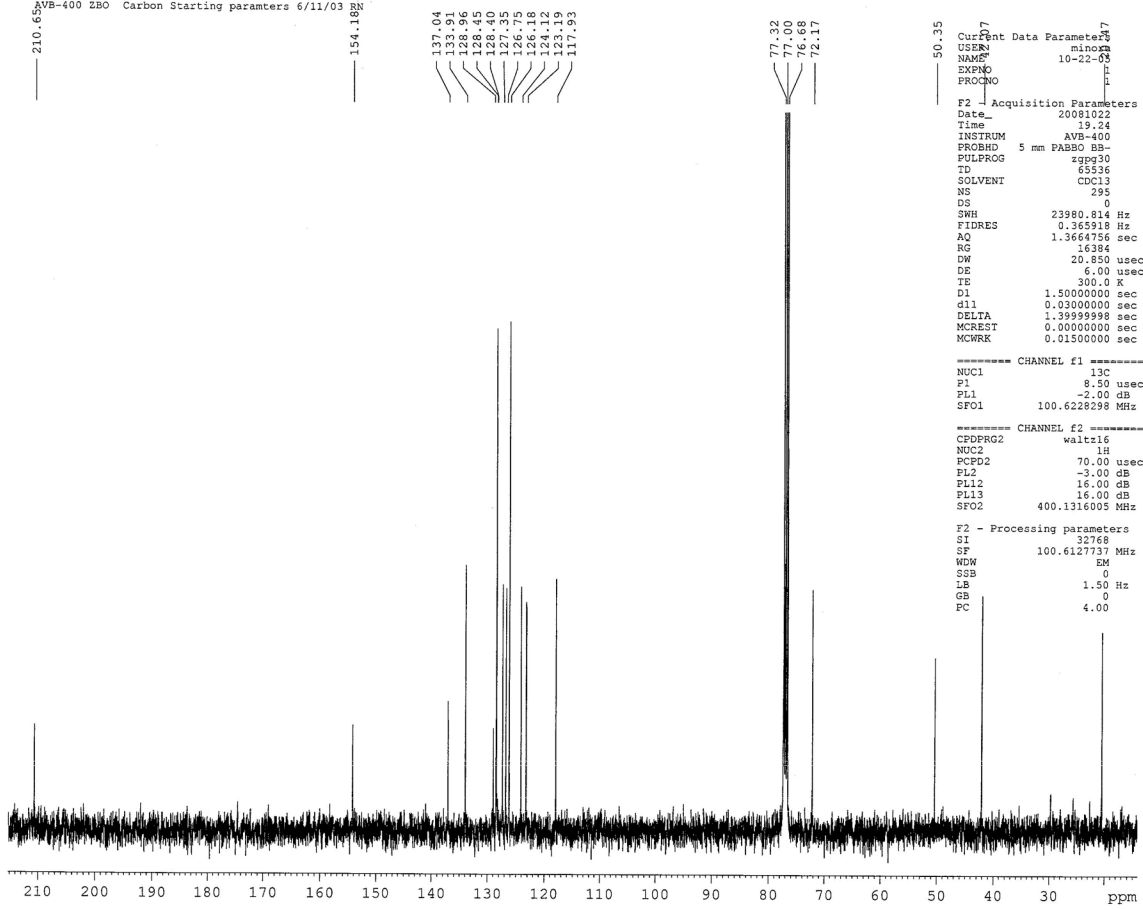
Current Data Parameters
USER      minoru
NAME      10-22-03
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20081022
Time      18.52
INSTRUM   av-300
PROBHD    5 mm Dual 13C/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        6172.839 Hz
FIDRES     0.094190 Hz
AQ         5.3084660 sec
RG         645.1
DW         81.000 usec
DE         6.00 usec
TE         295.1 K
D1         0.20000000 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        11.00 usec
PL1       -3.00 dB
SFO1      300.1318533 MHz

F2 - Processing parameters
SI         32768
SF         300.1300665 MHz
WDW        EM
GB         0
PC         4.00
    
```

AVB-400 Z80 Carbon Starting parameters 6/11/03 RN



```

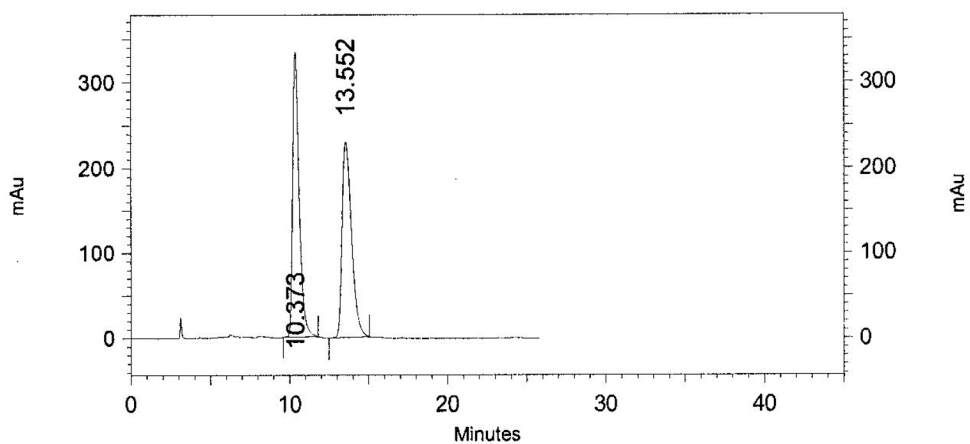
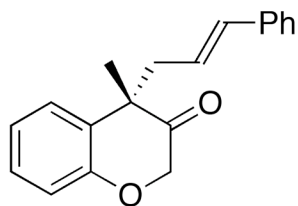
Current Data Parameters
USER      minoru
NAME      10-22-03
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20081022
Time      19.24
INSTRUM   AVB-400
PROBHD    5 mm PABBO B3-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         295
DS         0
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664736 sec
RG         16384
DW         20.850 usec
DE         6.00 usec
TE         300.0 K
D1         1.50000000 sec
d11        0.03000000 sec
DELTA      1.39999998 sec
MCREST     0.00000000 sec
MCWRK     0.01500000 sec

===== CHANNEL f1 =====
NUC1      13C
P1         8.50 usec
PL1        -2.00 dB
SFO1      100.6228298 MHz

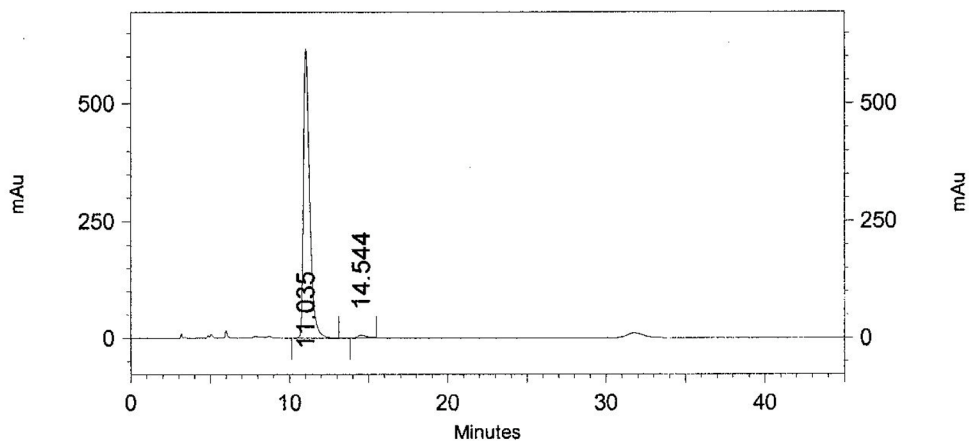
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     70.00 usec
PL2        -3.00 dB
PL12       16.00 dB
PL13       16.00 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127737 MHz
WDW        EM
SSB        0
LB         1.50 Hz
GB         0
PC         4.00
    
```



2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
10.373	9547551	50.012
13.552	9543092	49.988



2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
11.035	16768066	98.675
14.544	225203	1.325