

Convergent Diastereoselective Preparation of Adjacent Quaternary Stereocenters in an Acyclic System

Tom Mejuch^a, Bishnu Dutta^a, Mark Botoshansky^b, Ilan Marek^{a*}

^aThe Mallat Family Laboratory of Organic Chemistry, Schulich Faculty of Chemistry and The Lise Meitner-Minerva Center for Computational Quantum Chemistry, Technion-Israel Institute of Technology, Haifa 32000, Israel. ^bLaboratory for X-Ray Analyses, Technion-Israel Institute of Technology, Haifa 32000, Israel.

Email Address: chilanm@tx.technion.ac.il

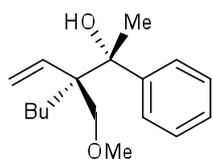
Supporting Information

General methods:

All the reactions involving organometallic compounds were carried out under positive pressure of argon. All glass apparatus were flame dried and cooled under argon. Ether and THF were distilled over Na-benzophenone ketyl under argon. Copper (I) iodide, diethylzinc (1M solution in hexanes), dibutylzinc (1M solution in heptane), dimethylzinc (1M solution in heptane) were purchased from Aldrich. Ketones were distilled over magnesium sulfate prior to use. ¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker Ultrashield AV300 spectrometer, operating at 300 MHz (¹H) or 75.46 MHz (¹³C) and AV500, operating at 500 MHz (¹H) or 125.76 MHz (¹³C) using CDCl₃ as a solvent (unless otherwise specified). Chemical shifts reported (in ppm) are relative to internal standard Me₄Si (δ=0.0).

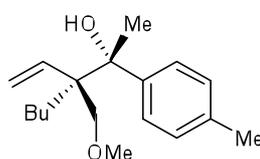
General Procedure A: Into a flame-dried, 100-mL three-neck flask, equipped with a magnetic stirrer, low-temperature thermometer, rubber septum and inert gas inlet, CuI (90 mg, 0.5 mmol) and dry THF (5 mL) were added. The reaction flask was cooled down at -35 °C and *n*-BuLi (1.6 M sol. in hexanes, 0.7 mL, 1 mmol) was added. The resulting mixture was stirred at -30 °C for 30 minutes and propargylic ether (0.17 mL, 2 mmol) was added to the solution and stirred at -25 °C for 1 h. Reaction mixture was cooled at -60 °C and diluted with dry THF (5 mL). Freshly distilled CH₂I₂ (0.4 mL, 5 mmol), ketone (0.8 mmol) and Et₂Zn (1 M sol. in hexane, 3 mL, 3 mmol) were added in turn. The resulting mixture was stirred at -50 °C for 3h. Progress of the reaction was monitored by TLC. The reaction was stopped with NH₄Cl:NH₄OH (2:1). The aqueous phase was separated and extracted with Et₂O (3 x 10 mL). The combined organic phases were washed with NH₄Cl:NH₄OH (2:1), dried over MgSO₄ and concentrated under reduced pressure. The product was purified by silica-gel flash chromatography using hexane and ethyl acetate as eluents.

General procedure B: Into a flame-dried, 100-mL three-neck flask, equipped with a magnetic stirrer, low-temperature thermometer, rubber septum and inert gas inlet, CuI (228 mg, 1.2 mmol, 1.5 equiv) and dry THF (10 mL) were added. The reaction flask was cooled down at -40 °C. Grignard reagent (1.5 mmol, 1.2 equiv) was added dropwise, the resulting mixture was allowed to warm-up to -30 °C and stirred for 1 hour at the same temperature. The reaction mixture was cooled down at -50 °C and alkynyl ether (2 mmol, 2.5 equiv) was added dropwise. The mixture was stirred at -35 °C for 2.5 hours. Then the reaction mixture was cooled down at -60 °C and diluted with THF (10 mL). Freshly distilled CH₂I₂ (0.35 mL, 4 mmol, 5 equiv), ketone (0.8 mmol, 1 equiv) and R₂Zn (2 mL, 1M, 2 mmol, 2.5 equiv) were added in turn. The resulting mixture was stirred at -50 °C for 3 hours and then allowed to warm-up slowly to an ambient temperature over 12 hours. The reaction was stopped with NH₄Cl:NH₄OH (2:1). The aqueous phase was separated and extracted with Et₂O (3 x 10 mL). The combined organic phases were washed with NH₄Cl:NH₄OH (2:1), dried over MgSO₄ and concentrated under reduced pressure. The product was purified by silica-gel flash chromatography using hexane and ethyl acetate as eluents.



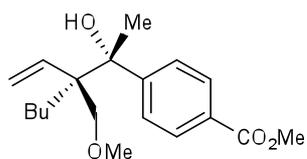
(2R*,3R*)-3-(Methoxymethyl)-2-phenyl-3-vinylheptan-2-ol

(10a): general procedure A, yield 60%. ¹H-NMR (300 MHz, CDCl₃): 0.80-0.95 (t, *J* = 7.3 Hz, 3H), 1.04-1.07 (m, 2H), 1.20-1.25 (m, 3H), 1.53 (s, 3H), 1.98-2.1 (m, 1H), 2.98-3.01 (d, *J* = 9.0 Hz, 1H), 3.22 (s, 3H), 3.20-3.21 (d, *J* = 9.0 Hz, 1H), 4.66 (d, *J* = 18 Hz, 1H), 5.06 (s, 1H), 5.08 (d, *J* = 11.3 Hz, 1H), 5.44 (dd, *J* = 11.3 Hz, *J* = 18 Hz, 1H), 7.13-7.23 (m, 3H), 7.29-7.31 (m, 2H). ¹³C-NMR (75 MHz, CDCl₃): 14.2, 23.5, 24.3, 26.4, 29.3, 50.0, 59.0, 74.7, 79.7, 115.6, 126.2, 126.8, 126.9, 139.3, 146.2. HRMS (EI) *m/z*: 285.1826 [M+Na]⁺, (calc. 285.1825).



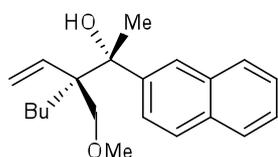
(2R*,3R*)-3-(Methoxymethyl)-2-p-tolyl-3-vinylheptan-2-ol

(10b): general procedure A, yield 50%. ¹H-NMR (300 MHz, CDCl₃): 0.79-0.83 (t, *J* = 7.5 Hz, 3H), 1.00-1.08 (m, 2H), 1.18-1.27 (m, 3H), 1.49 (s, 3H), 1.98-2.05 (m, 1H), 2.21 (s, 3H), 2.99-3.02 (d, *J* = 9.3 Hz, 1H), 3.16 (s, 3H), 3.16-3.30 (d, *J* = 9.3 Hz, 1H), 4.57 (brs, 1H), 4.57-4.64 (d, *J* = 17.7 Hz, 1H), 5.02-5.06 (d, *J* = 11.4 Hz, 1H), 5.37-5.47 (dd, *J* = 11.4 Hz, *J* = 17.7 Hz, 1H), 6.97-6.99 (d, *J* = 7.8 Hz, 2H), 7.14-7.17 (d, *J* = 7.8 Hz, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ : 14.1, 20.8, 23.4, 24.3, 26.3, 29.3, 49.9, 58.8, 74.7, 79.5, 115.3, 126.6, 127.5, 135.5, 139.4, 143.2. HRMS (EI) *m/z*: 299.1982 [M+Na]⁺, (calc. 299.1982).



Methyl-4-((2R*,3R*)-2-hydroxy-3-(methoxymethyl)-3-vinylheptan-2-yl)benzoate

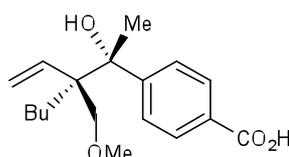
(10c): general procedure A, yield 51%. ¹H-NMR (300 MHz, CDCl₃): 0.86-0.91 (t, *J* = 7.2 Hz, 3H), 1.09-1.12 (m, 2H), 1.28-1.33 (m, 3H), 1.47-1.48 (m, 1H), 1.49 (s, 3H), 2.05-2.15 (m, 1H), 2.97-3.00 (d, *J* = 9.3 Hz, 1H), 3.25-3.28 (m, 4H), 3.88 (s, 3H), 4.61-4.68 (d, *J* = 18 Hz, 1H), 5.08-5.17 (d, *J* = 11.3 Hz, 1H), 5.17-5.52 (dd, *J* = 11.3 Hz, *J* = 18 Hz, 1H), 7.42-7.45 (d, *J* = 8.4 Hz, 2H), 7.91-7.92 (d, *J* = 8.4 Hz, 2H). ¹³C-NMR (75 MHz, CDCl₃): 14.1, 23.4, 24.2, 26.3, 29.3, 36.6, 51.9, 59.0, 77.6, 79.8, 116.0, 126.9, 128.2, 128.3, 139.0, 150.3, 167.2.



(2R*,3R*)-3-(Methoxymethyl)-2-(naphthalen-2-yl)-3-vinylheptan-2-ol

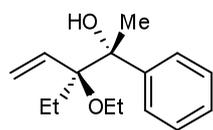
(10d): general procedure A, yield 52%. ¹H-

NMR (300 MHz, CDCl₃): 0.89-0.94 (t, $J = 7.5$ Hz, 3H), 1.14-1.16 (m, 2H), 1.32-1.36 (m, 2H), 1.56 (m, 1H), 1.61-1.63 (m, 3H), 2.15-2.12 (m, 1H), 3.09-3.12 (d, $J = 9.3$ Hz, 1H), 3.27-3.28 (m, 4H), 4.67-4.74 (d, $J = 18$ Hz, 1H), 5.16 (s, 1H), 5.17-5.21 (d, $J = 11.4$ Hz, 1H), 5.55-5.60 (dd, $J = 11.4$ Hz, $J = 18$ Hz, 1H), 7.44-7.55 (m, 3H), 7.73-7.87 (m, 4H). ¹³C-NMR (75 MHz, CDCl₃): 14.2, 23.5, 24.5, 26.4, 29.5, 50.2, 59.0, 74.8, 79.9, 115.8, 125.49, 125.52, 125.63, 125.68, 126.1, 127.2, 128.3, 132.1, 132.6, 139.4, 143.9. HRMS (EI) m/z : 311.2038 [M-H]⁻, (calc. 311.2016).



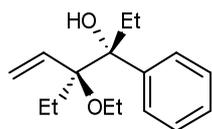
4-((2R*,3R*)-2-Hydroxy-3-(methoxymethyl)-3-vinylheptan-2-yl)-benzoic acid (11): Methyl ester **10c** (160 mg,

0.5 mmol) was dissolved in methanol (2 mL). KOH (1 M sol. in water, 2 mL, 2 mmol) was added dropwise and the resulting mixture was refluxed for 30 min. Methanol was removed under reduced pressure, the mixture was diluted with cold water (2 mL), acidified with HCl (32% aqueous solution, 2mL) and extracted with ethyl acetate (3x10 mL). The organic phase was dried over Na₂SO₄ and concentrated under reduced pressure to afford the product as white crystalline solid in 78% yield. ¹H-NMR (300 MHz, CDCl₃): 0.86-0.91 (t, $J = 7.2$ Hz, 3H), 1.08-1.13 (m, 2H), 1.24-1.43 (m, 2H), 1.46-1.49 (m, 4H), 2.05-2.16 (m, 1H), 2.99-3.02 (d, $J = 9.3$ Hz, 1H), 3.27-3.30 (m, 4H), 4.63-4.69 (d, $J = 18$ Hz, 1H), 5.15-5.19 (d, $J = 11.4$ Hz, 1H), 5.47-5.57 (dd, $J = 11.4$ Hz, $J = 18$ Hz, 1H), 7.47-7.50 (d, $J = 8.4$ Hz, 2H), 7.99-8.02 (d, $J = 8.4$ Hz, 2H). ¹³C-NMR (75 MHz, CDCl₃) δ : 14.1, 23.4, 24.1, 26.2, 29.2, 49.8, 59.0, 74.5, 80.0, 116.1, 126.9, 127.3, 128.8, 138.8, 152.4, 171.8. HRMS (EI) m/z : 305.1777 [M-H]⁻, (calc. 305.1758).



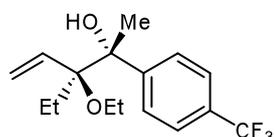
(2R*,3S*)-3-Ethoxy-3-ethyl-2-phenylpent-4-en-2-ol (14a):

general procedure B, yield 71%. ¹H-NMR (500 MHz, CDCl₃): 7.42-7.19 (m, 5H), 5.43 (dd, $J = 11.2$ Hz, 17.7 Hz, 1H), 5.22 (d, $J = 11.2$ Hz, 1H), 5.10 (d, $J = 17.7$ Hz, 1H), 3.40-3.30 (m, 3H), 1.85-1.75 (m, 2H), 1.56 (s, 3H), 1.2 (t, $J = 7$ Hz, 3H), 0.91 (t, $J = 7.5$ Hz, 3H). ¹³C-NMR (125.76 MHz, CDCl₃): 145.6, 139.5, 127.4, 126.9, 126.3, 116.8, 83.7, 78.3, 59.3, 24.4, 23.8, 15.9, 9.2. HRMS (EI) m/z : 257.1523 [M+Na]⁺ (calc. 257.1517).



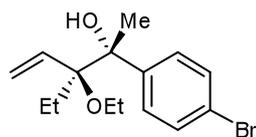
(3R*,4S*)-4-Ethoxy-4-ethyl-3-phenylhex-5-en-3-ol (14b):

general procedure B, yield 70%. ¹H-NMR (500 MHz, CDCl₃): 7.42 (m, 2H), 7.27 (m, 2H), 7.20 (m, 1H), 5.54 (dd, *J* = 11.2 Hz, 17.7 Hz, 1H), 5.24 (d, *J* = 11.2 Hz, 1H), 5.09 (d, *J* = 17.7 Hz, 1H), 3.36 (m, 2H), 2.97 (bs, 1H), 2.18 (m, 1H), 1.86 (m, 1H), 1.77 (q, *J* = 7.5 Hz, 2H), 1.15 (t, *J* = 7 Hz, 3H), 0.85 (t, *J* = 7.5 Hz, 3H), 0.66 (t, *J* = 7.4 Hz, 3H). ¹³C-NMR (125.76 MHz, CDCl₃): 142.3, 139.3, 128.1, 126.9, 126.1, 116.8, 84.3, 81.2, 59.4, 27.7, 24.1, 15.9, 9.3, 7.7. HRMS (EI) *m/z*: 249.1828 [M+H]⁺ (calc. 249.1855).



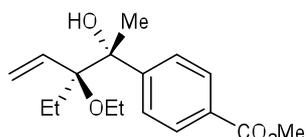
(2R*,3S*)-3-Ethoxy-3-ethyl-2-(4-(trifluoromethyl)-phenyl)-pent-4-en-2-ol (14c):

general procedure B, yield 58%. ¹H-NMR (500 MHz, CDCl₃): 7.56 (d, *J* = 8.5 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 5.41 (dd, *J* = 11.2 Hz, 17.7 Hz, 1H), 5.26 (d, *J* = 11.2 Hz, 1H), 5.12 (d, *J* = 17.7 Hz, 1H), 3.4-3.3 (m, 3H), 1.9-1.7 (m, 2H), 1.56 (s, 3H), 1.16 (t, *J* = 7 Hz, 3H), 0.92 (t, *J* = 7.5 Hz, 3H). ¹³C-NMR (125.76 MHz, CDCl₃): 149.7, 139.3, 128.5 (d, *J* = 32.3 Hz), 127.8, 124.4 (d, *J* = 271.8 Hz), 123.8 (q, *J* = 3.8 Hz), 117.6, 83.5, 78.2, 59.3, 24.4, 23.9, 15.8, 9.1. HRMS (EI) *m/z*: 301.1456 [M-H]⁻ (calc. 301.1415).



(2R*,3S*)-2-(4-Bromophenyl)-3-ethoxy-3-ethylpent-4-en-2-ol (14d):

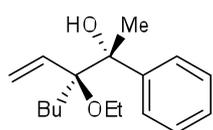
general procedure B, yield 54%. ¹H-NMR (300 MHz, CDCl₃): 7.39 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.7 Hz, 2H), 5.42 (dd, *J* = 11.2 Hz, 17.6 Hz, 1H), 5.24 (d, *J* = 11.2 Hz, 1H), 5.10 (d, *J* = 17.6 Hz, 1H), 3.35 (m, 3H), 1.79 (m, 2H), 1.53 (s, 3H), 1.15 (t, *J* = 7 Hz, 3H), 0.92 (t, *J* = 7.5 Hz, 3H). ¹³C-NMR (125.76 MHz, CDCl₃): 144.7, 139.4, 130.0, 129.3, 120.5, 117.3, 83.5, 78.1, 59.2, 24.3, 23.9, 15.8, 9.1. HRMS (EI) *m/z*: 311.0617 [M-H]⁻ (calc. 311.0647).



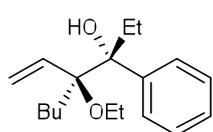
Methyl 4-((2R*,3S*)-3-ethoxy-3-ethyl-2-hydroxypent-4-en-2-yl)benzoate (14e):

general procedure B, yield 68%. ¹H-NMR (300 MHz, CDCl₃): 7.92 (d, *J* = 8.8 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 5.41 (dd, *J* = 11.1 Hz, 17.6 Hz, 1H), 5.22 (d, *J* = 11.1 Hz, 1H),

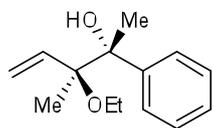
5.09 (d, $J = 17.6$ Hz, 1H), 3.89 (s, 3H), 3.41 (bs, 1H), 3.33 (m, 2H), 1.79 (m, 2H), 1.55 (s, 3H), 1.13 (t, $J = 7$ Hz, 3H), 0.90 (t, $J = 7.5$ Hz, 3H). ^{13}C -NMR (75.46 MHz, CDCl_3): 167.2, 151.0, 139.2, 128.15, 128.10, 127.5, 117.4, 83.6, 78.3, 59.2, 51.9, 24.3, 23.8, 15.8, 9.1. HRMS (EI) m/z : 315.1563 $[\text{M}+\text{Na}]^+$ (calc. 315.1572).



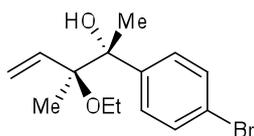
(2R*,3S*)-3-Ethoxy-2-phenyl-3-vinylheptan-2-ol (14f): general procedure B, yield 68%. ^1H -NMR (500 MHz, CDCl_3): 7.43-7.20 (m, 5H), 5.39 (dd, $J = 11.2$ Hz, 17.7 Hz, 1H), 5.20 (d, $J = 11.2$ Hz, 1H), 5.10 (d, $J = 17.7$ Hz, 1H), 3.49 (bs, 1H), 3.34 (m, 2H), 1.84-1.65 (m, 2H), 1.56 (s, 3H), 1.47-1.25 (m, 4H), 1.15, (t, $J = 7$ Hz, 3H), 0.89 (t, $J = 7.2$ Hz, 3H). ^{13}C -NMR (125.76 MHz, CDCl_3): 145.5, 139.8, 127.4, 126.9, 126.3, 116.6, 83.5, 78.3, 59.1, 31.2, 26.6, 24.2, 23.8, 15.8, 14.1. HRMS (EI) m/z : 263.2042 $[\text{M}+\text{H}]^+$ (calc. 263.2011).



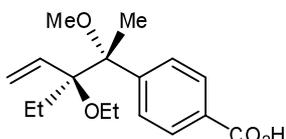
(3R*,4S*)-4-Ethoxy-3-phenyl-4-vinyloctan-3-ol (14g): general procedure B, yield 57%. ^1H -NMR (500 MHz, CDCl_3): 7.41-7.20 (m, 5H), 5.51 (dd, $J = 11.2$ Hz, 17.8 Hz, 1H), 5.22 (d, $J = 11.2$ Hz, 1H), 5.08 (d, $J = 17.7$ Hz, 1H), 3.34 (m, 2H), 3.05 (bs, 1H), 2.30-1.80 (m, 2H), 1.75-1.60 (m, 2H), 1.35-1.19 (m, 4H), 1.14, (t, $J = 7$ Hz, 3H), 0.86 (t, $J = 7.13$ Hz, 3H), 0.67 (t, $J = 7.4$ Hz, 3H). ^{13}C -NMR (125.76 MHz, CDCl_3): 142.2, 139.6, 128.1, 126.9, 126.1, 116.5, 84.2, 81.2, 59.3, 31.5, 27.6, 26.7, 23.8, 15.9, 14.1, 7.7. HRMS (EI) m/z : 299.1990 $[\text{M}+\text{Na}]^+$ (calc. 299.1987).



(2R*,3S*)-3-Ethoxy-3-methyl-2-phenylpent-4-en-2-ol (14h): general procedure B, yield 61%. ^1H -NMR (500 MHz, CDCl_3): 7.46 (m, 2H), 7.30-7.20 (m, 3H), 5.84 (dd, $J = 11$ Hz, 17.7 Hz, 1H), 5.31 (d, $J = 11$ Hz, 1H), 5.05 (d, $J = 17.7$ Hz, 1H), 3.5 (bs, 1H), 3.36 (q, $J = 7$ Hz, 2H), 1.55 (s, 3H), 1.19 (t, $J = 7$ Hz, 3H), 1.01 (s, 3H). ^{13}C -NMR (125.76 MHz, CDCl_3): 144.1, 139.3, 127.1, 126.8, 126.5, 117.7, 82.4, 78.0, 58.1, 24.6, 16.1, 15.9. HRMS (EI) m/z : 243.1356 $[\text{M}+\text{Na}]^+$ (calc. 243.1361).



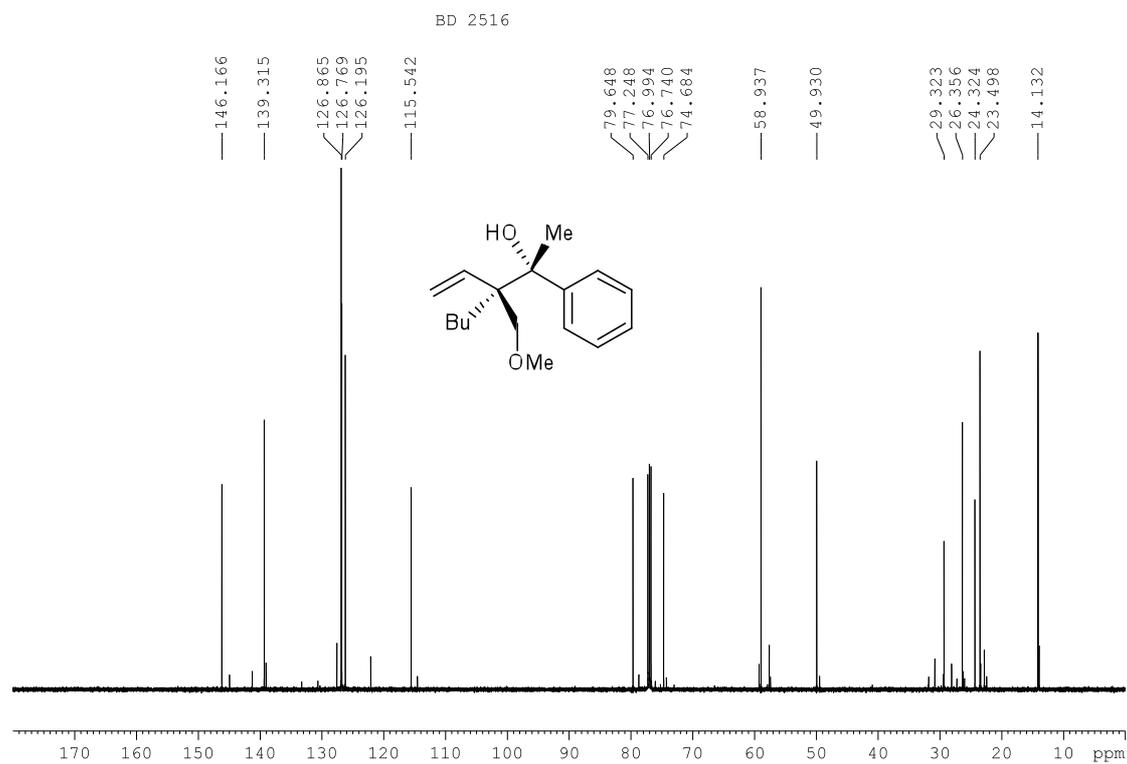
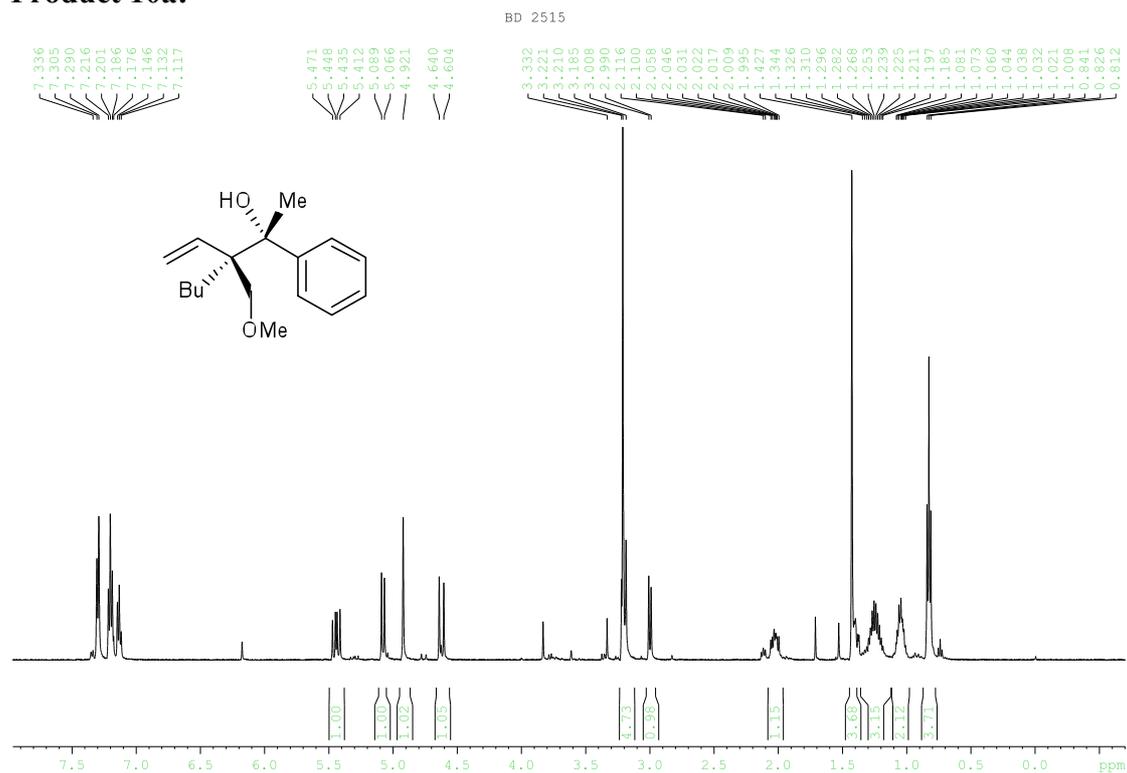
(2R*,3S*)-2-(4-bromophenyl)-3-ethoxy-3-methylpent-4-en-2-ol (14i): general procedure B, yield 57%. ¹H-NMR (500 MHz, CDCl₃): 7.39 (d, *J* = 8.9 Hz, 2H), 7.33 (d, *J* = 8.9 Hz, 2H), 5.70 (dd, *J* = 11 Hz, 17.7 Hz, 1H), 5.20 (d, *J* = 11 Hz, 1H), 5.03 (d, *J* = 17.8 Hz, 1H), 3.30 (m, 2H), 3.17 (bs, 1H), 1.55 (s, 3H), 1.19 (s, 3H), 1.14 (t, *J* = 7 Hz, 3H). ¹³C-NMR (125.76 MHz, CDCl₃): 143.3, 139.7, 130.0, 129.2, 120.6, 117.7, 81.9, 77.7, 58.2, 24.3, 16.2, 15.8.



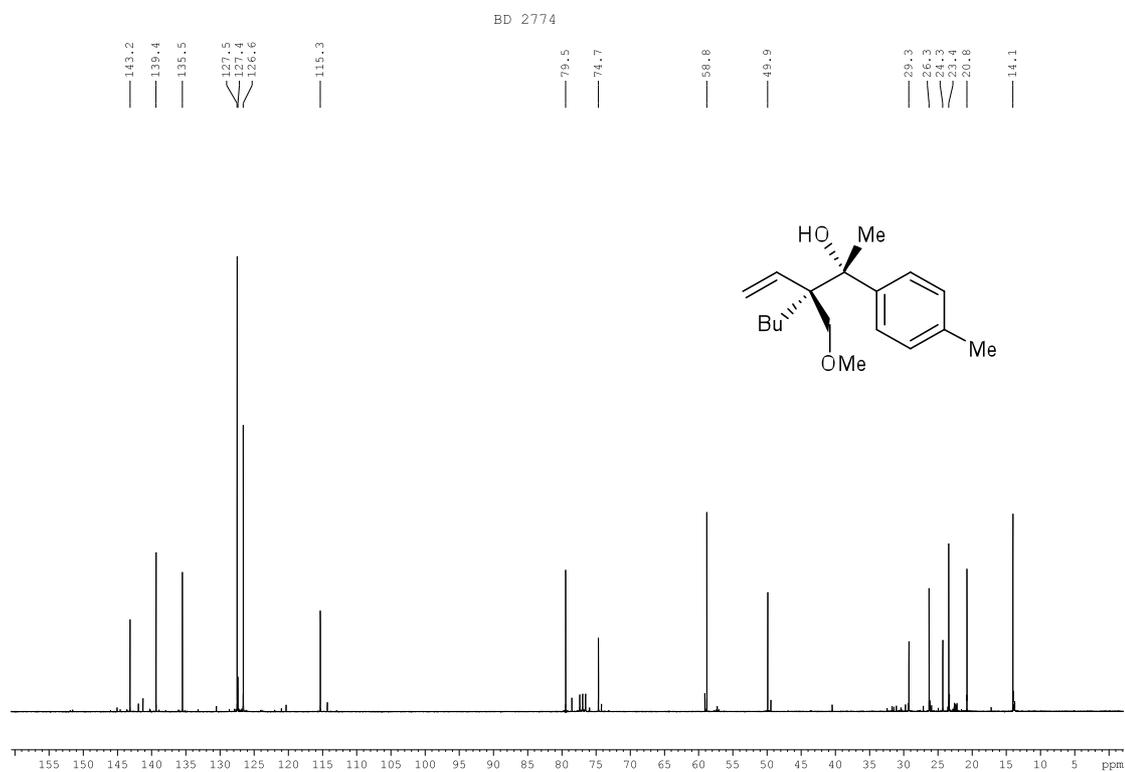
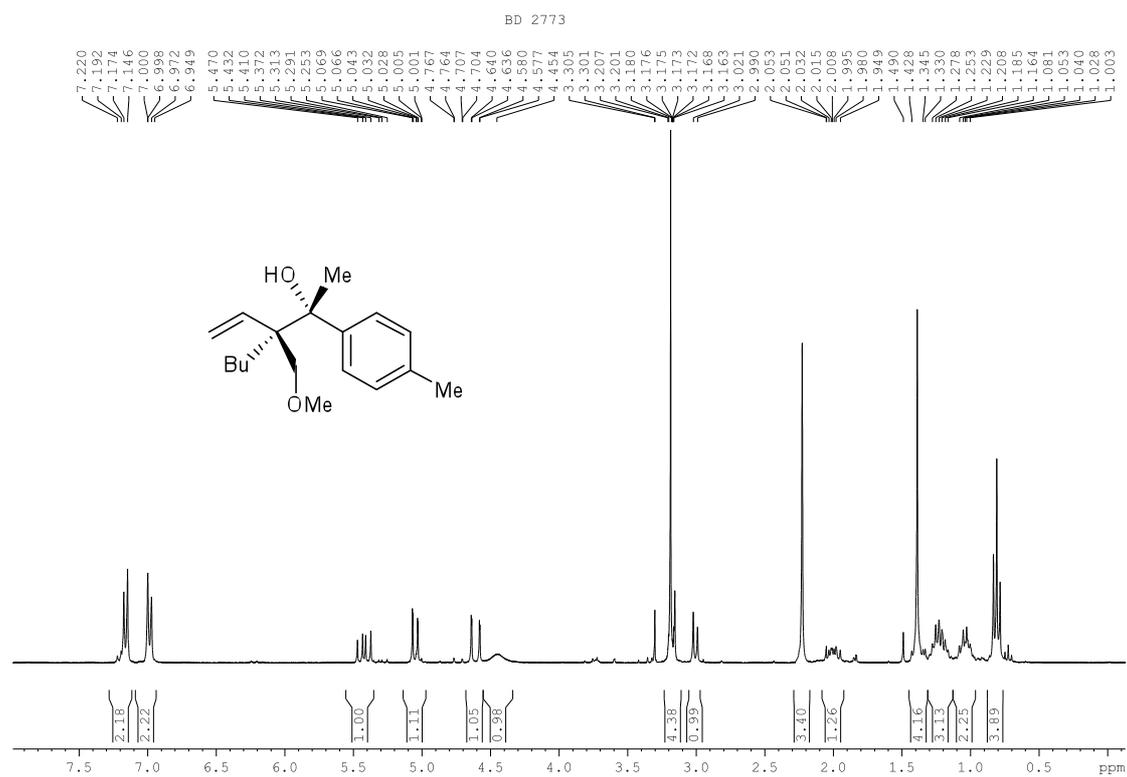
4-((2R*,3S*)-3-Ethoxy-3-ethyl-2-methoxy-pent-4-en-2-yl)-benzoic acid (15): In a 20-mL flask, KH (25% in mineral oil, 128 mg, 0.8 mmol) was washed with dry hexane (3 x 10 mL) and dissolved in dry THF (3 mL). Methyl ester **14e** (60 mg, 0.2 mmol) and dry THF (2 mL) were added at 0°C and the resulting mixture was stirred at the ambient temperature for 30 min. Then the flask was cooled down at 0°C and methyl iodide (0.065 mL, 142 mg, 1 mmol) was added dropwise. The resulting mixture was stirred at the ambient temperature for 48 h. The reaction was stopped with NH₄Cl (sat. aqueous solution, 5 mL) and the product was extracted with Et₂O (3 x 10 mL). The organic phase was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure to give the product that was subjected to the ester hydrolysis without purification. The product was added to the 20-mL flask, followed by the addition of KOH (15% solution in MeOH / H₂O, 3 mL). The resulting suspension was heated at 60°C for 12 h. Then the reaction mixture was cooled down and diluted with water (5 mL). Methanol was removed under reduced pressure and the resulting mixture was washed with Et₂O (2 x 5 mL), acidified with HCl (1M aqueous solution) until pH=1. The product was extracted with EtOAc (3 x 5 mL). The organic phase was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure to give a pure product as light yellow solid in 84% yield over two steps. ¹H-NMR (300 MHz, CDCl₃): 11.16 (bs, 1H), 8.05 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 5.71 (dd, *J* = 11.1 Hz, 17.5 Hz, 1H), 5.25 (d, *J* = 11.1 Hz, 1H), 5.16 (d, *J* = 17.5 Hz, 1H), 3.38-3.14 (m, 2H), 3.11 (s, 3H), 1.82 (m, 1H), 1.58 (s, 3H), 1.51 (m, 1H), 1.07 (t, *J* = 7 Hz, 3H), 0.80 (t, *J* = 7.5 Hz, 3H). ¹³C-NMR (75.46 MHz, CDCl₃): 172.4, 149.0, 138.2,

129.0, 128.8, 127.5, 115.8, 84.4, 83.9, 59.4, 50.6, 23.5, 19.4, 15.8, 9.4. HRMS (EI)
m/z: 291.1595 [M-H]⁻ (calc. 291.1596).

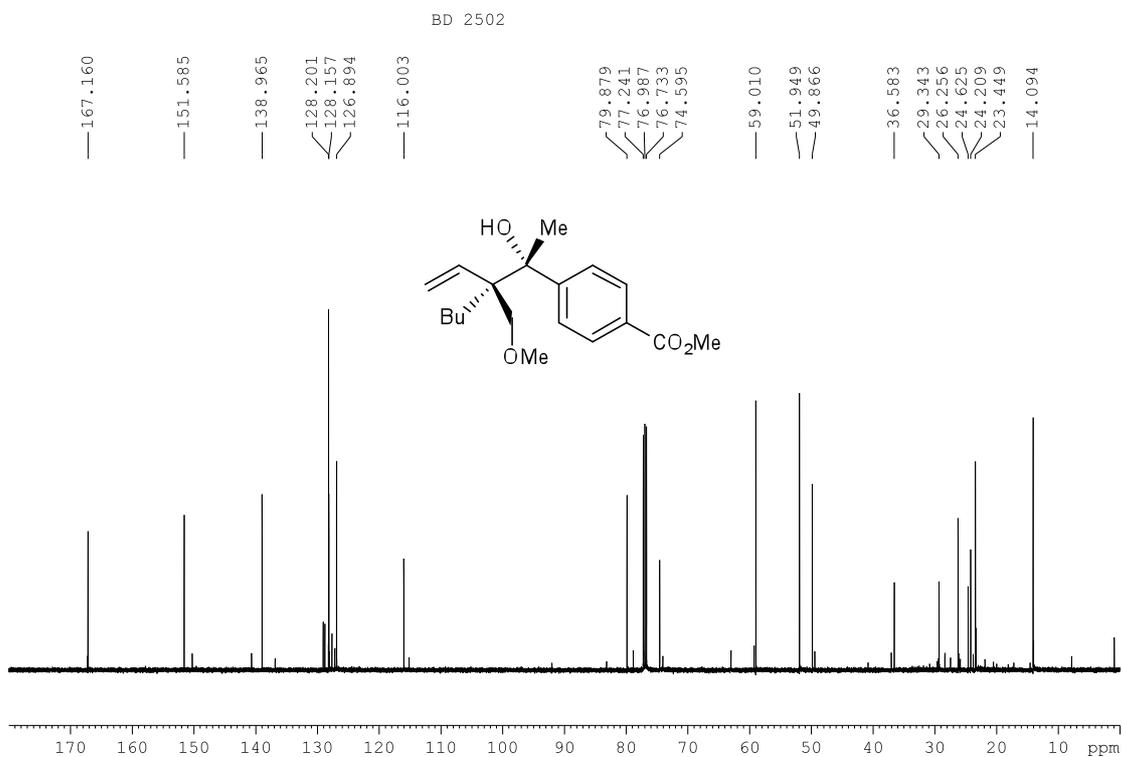
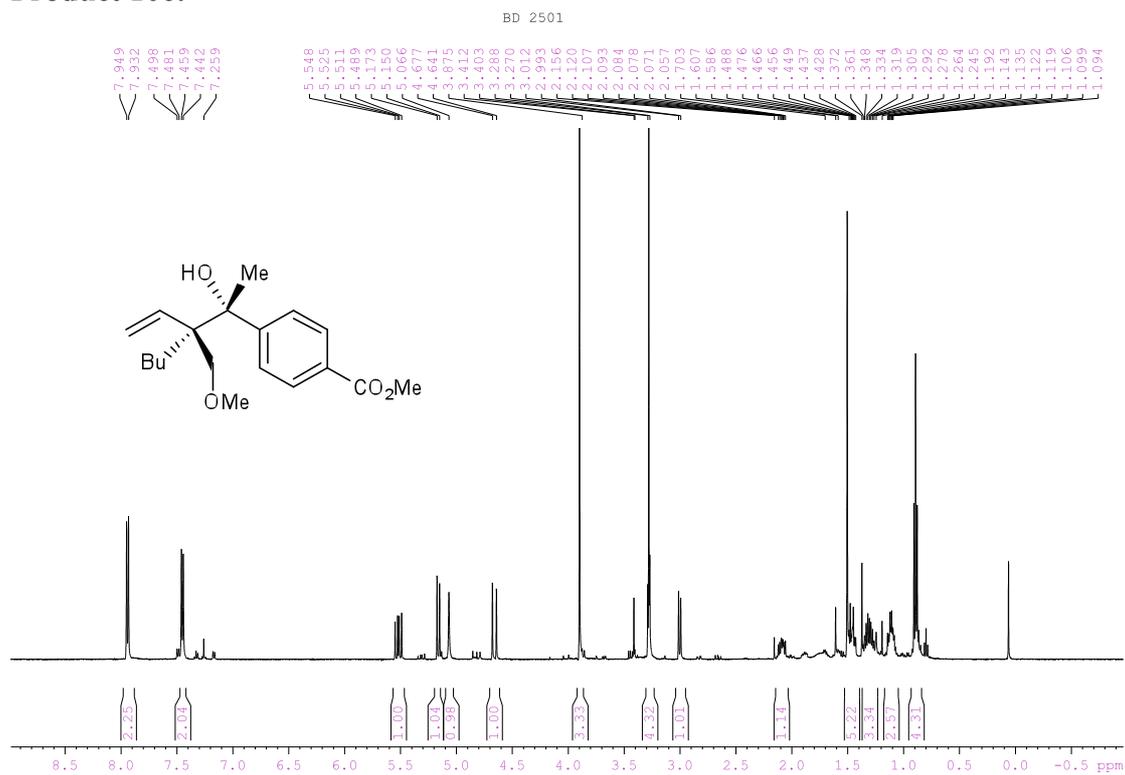
Product 10a:



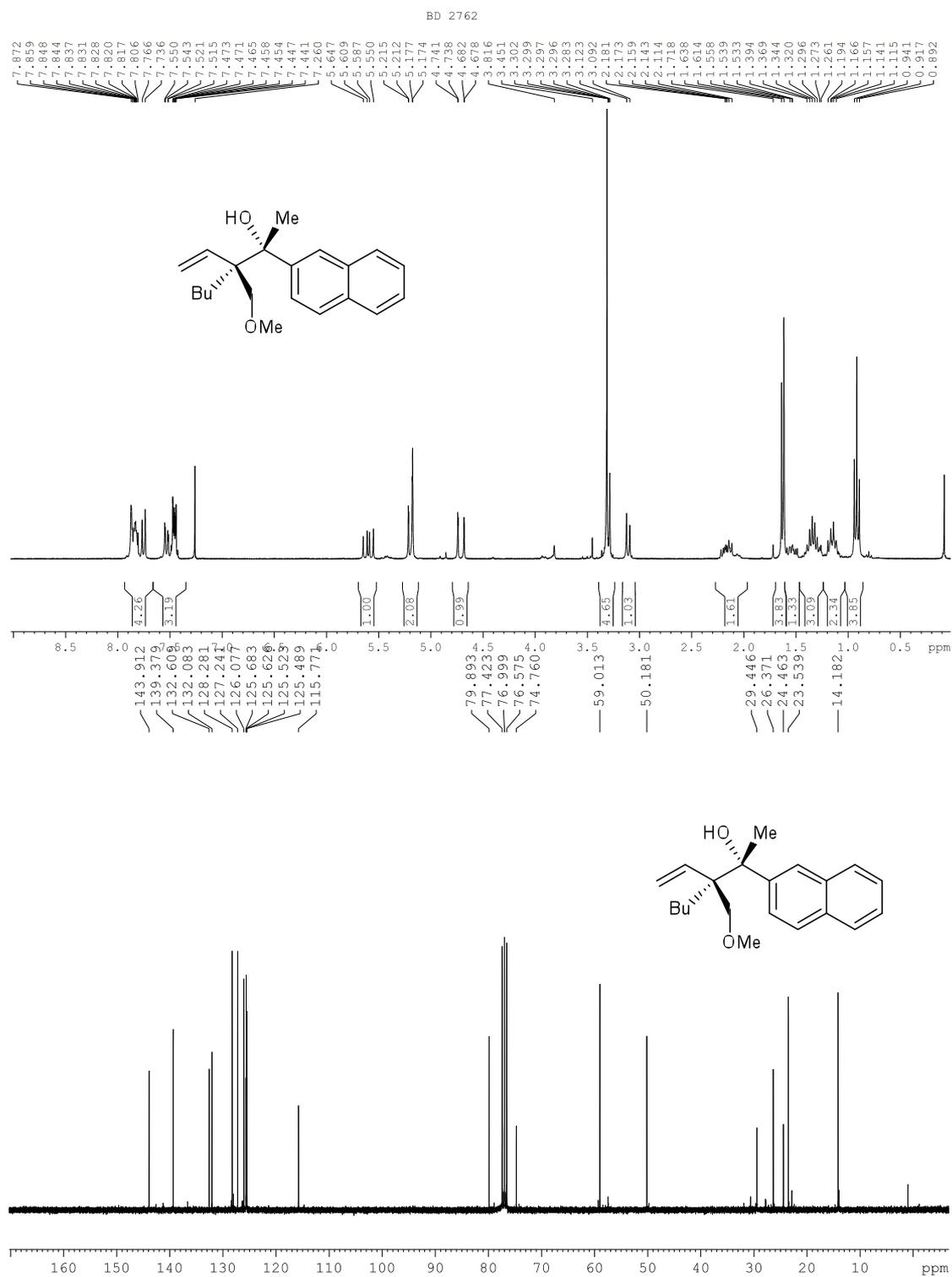
Product 10b:



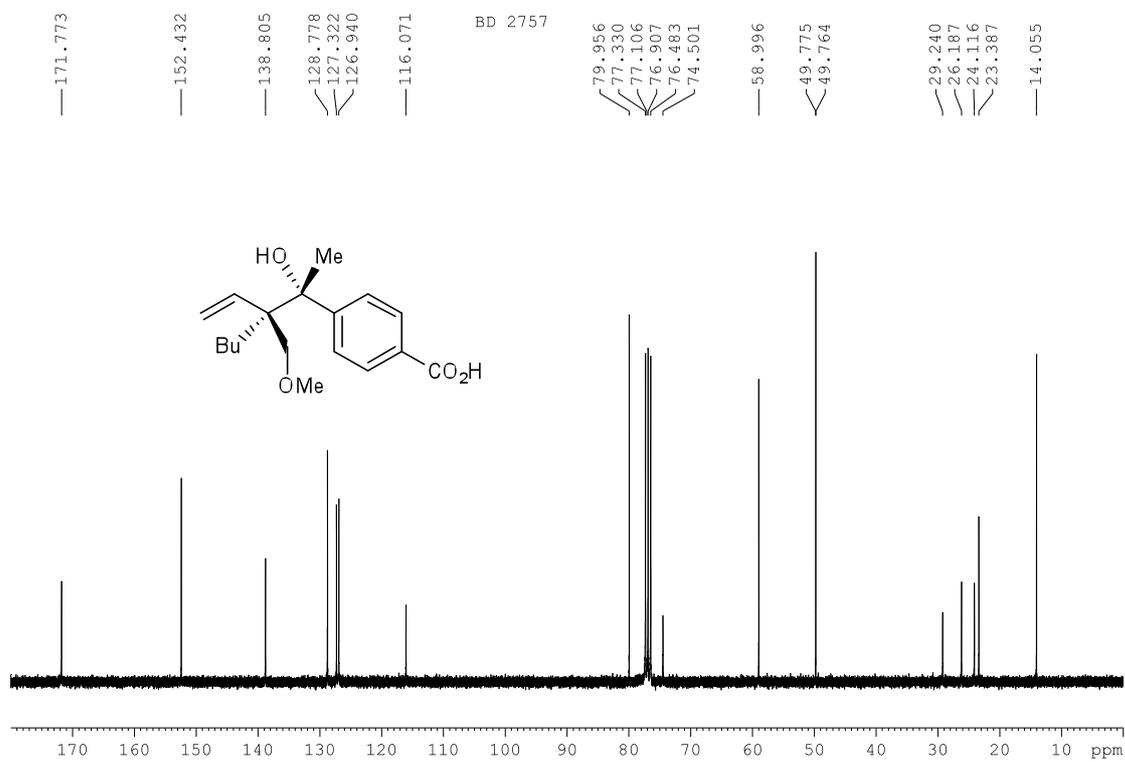
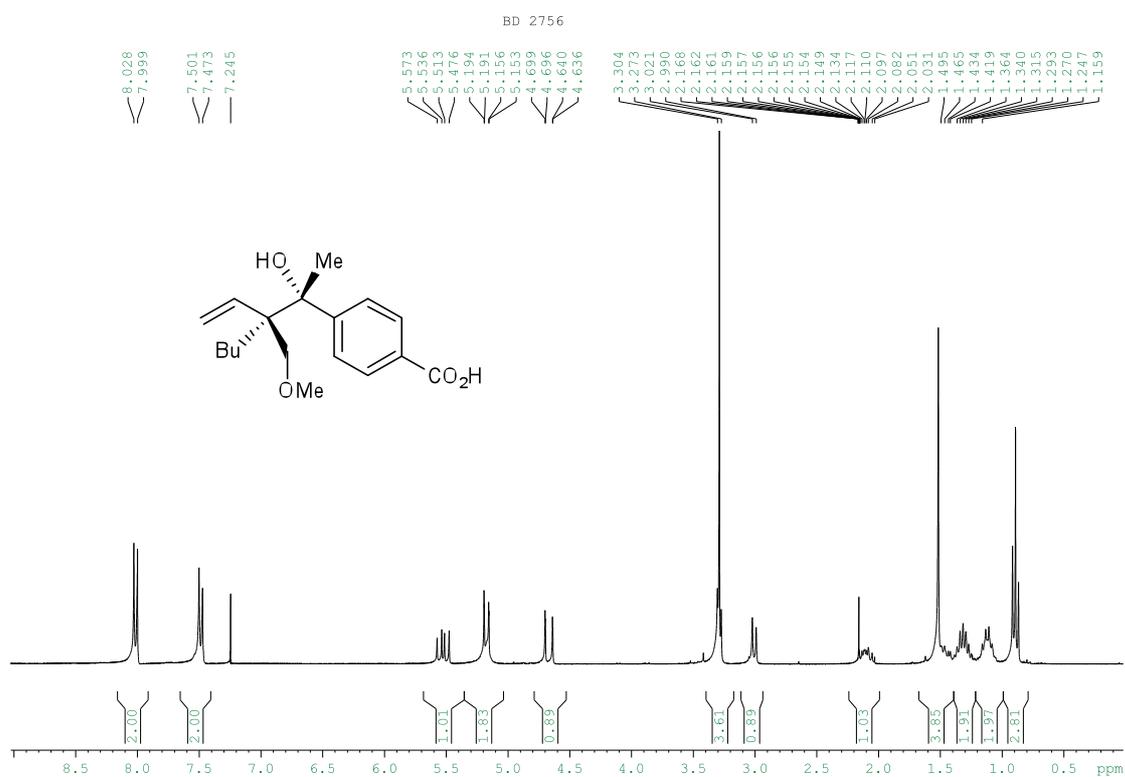
Product 10c:



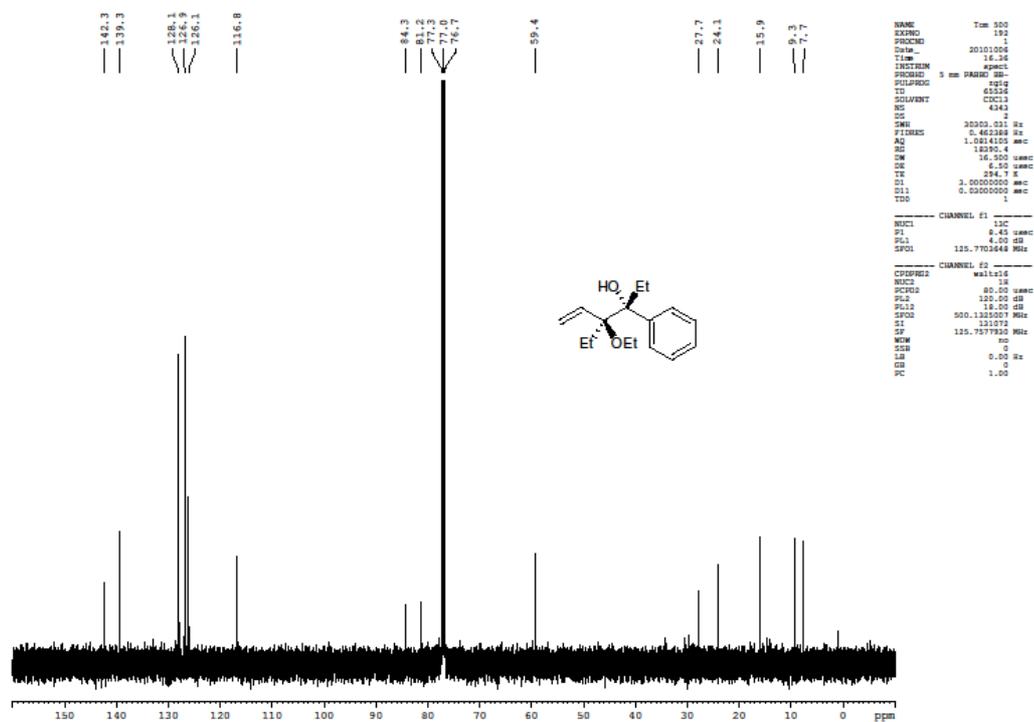
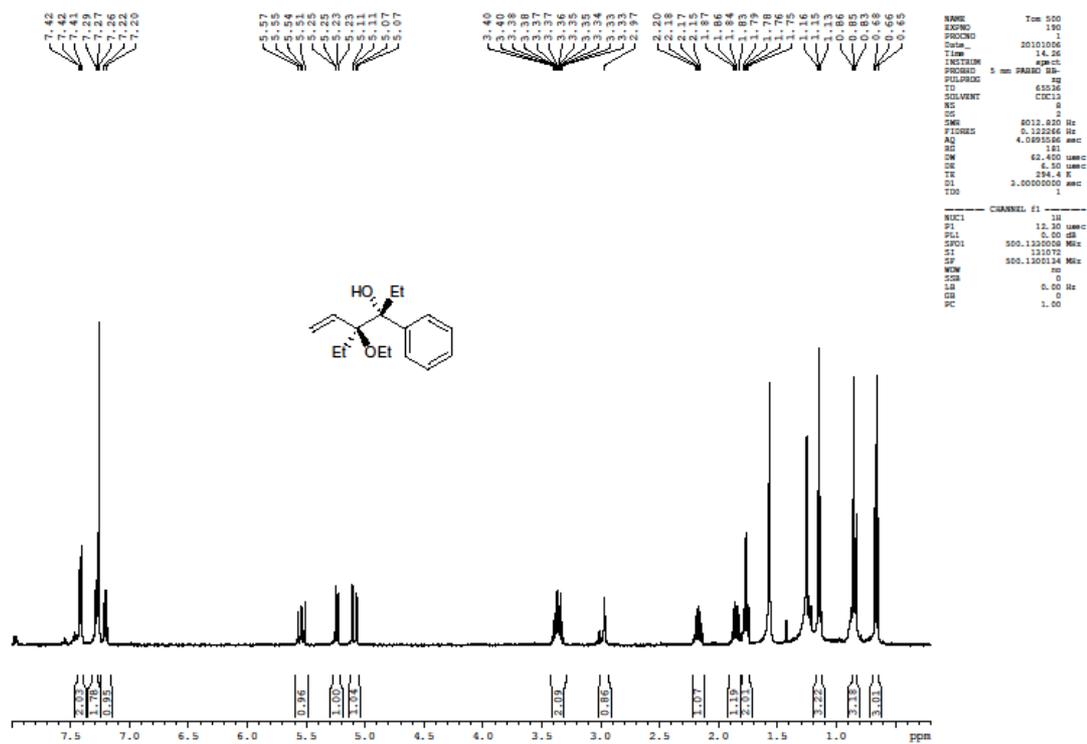
Product 10d:



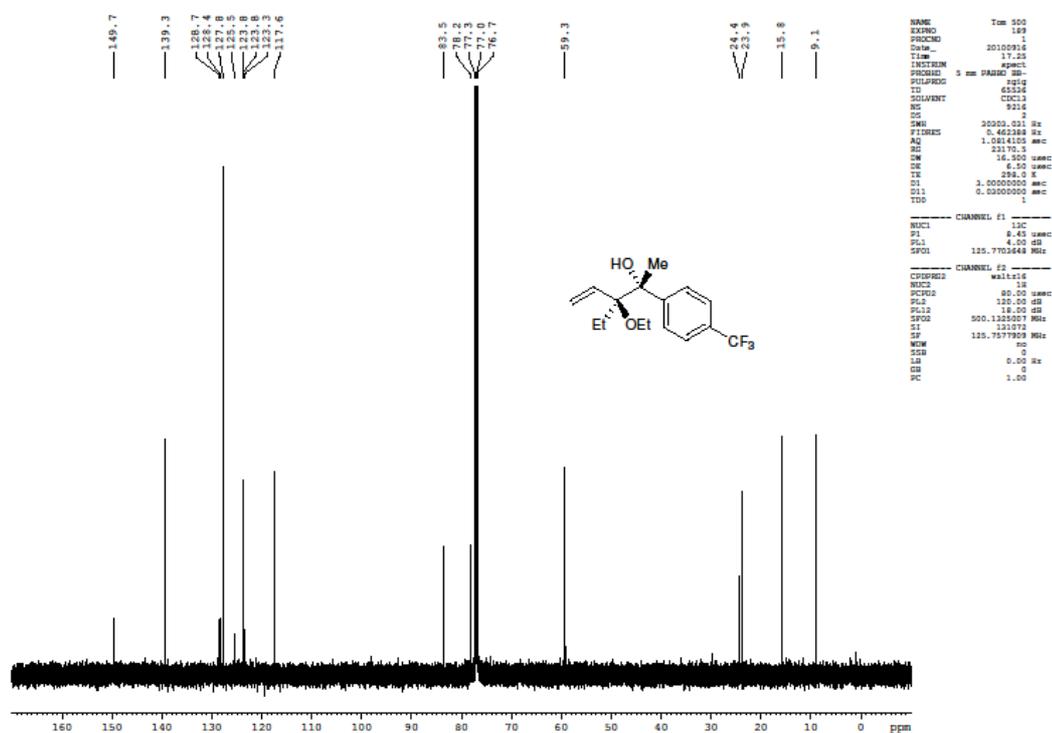
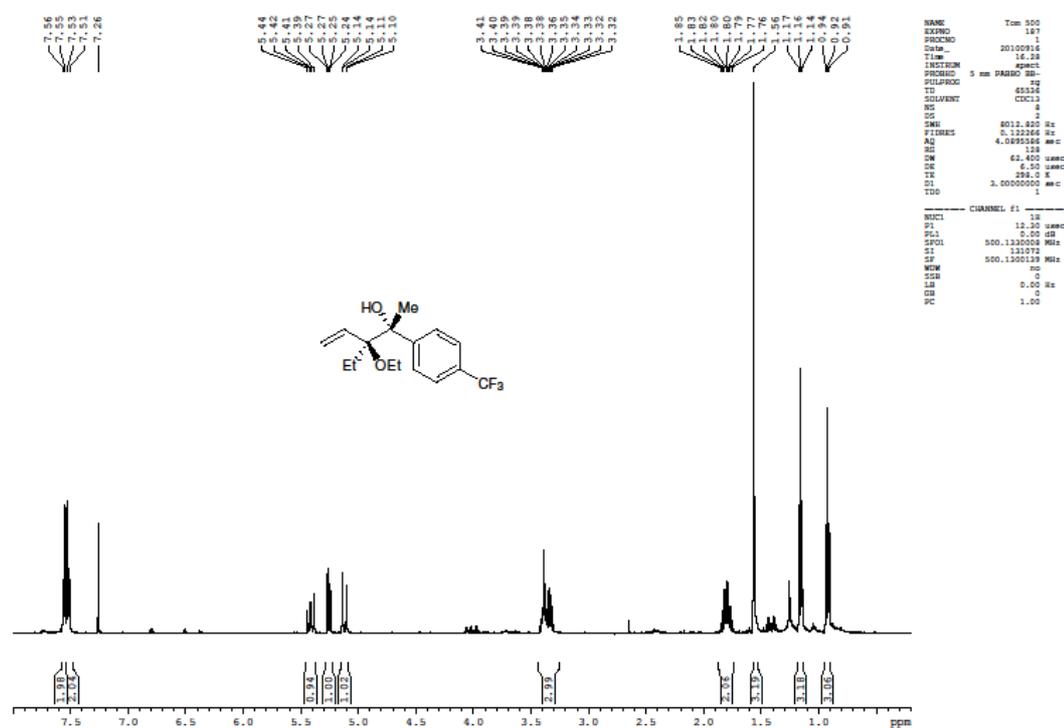
Product 11:



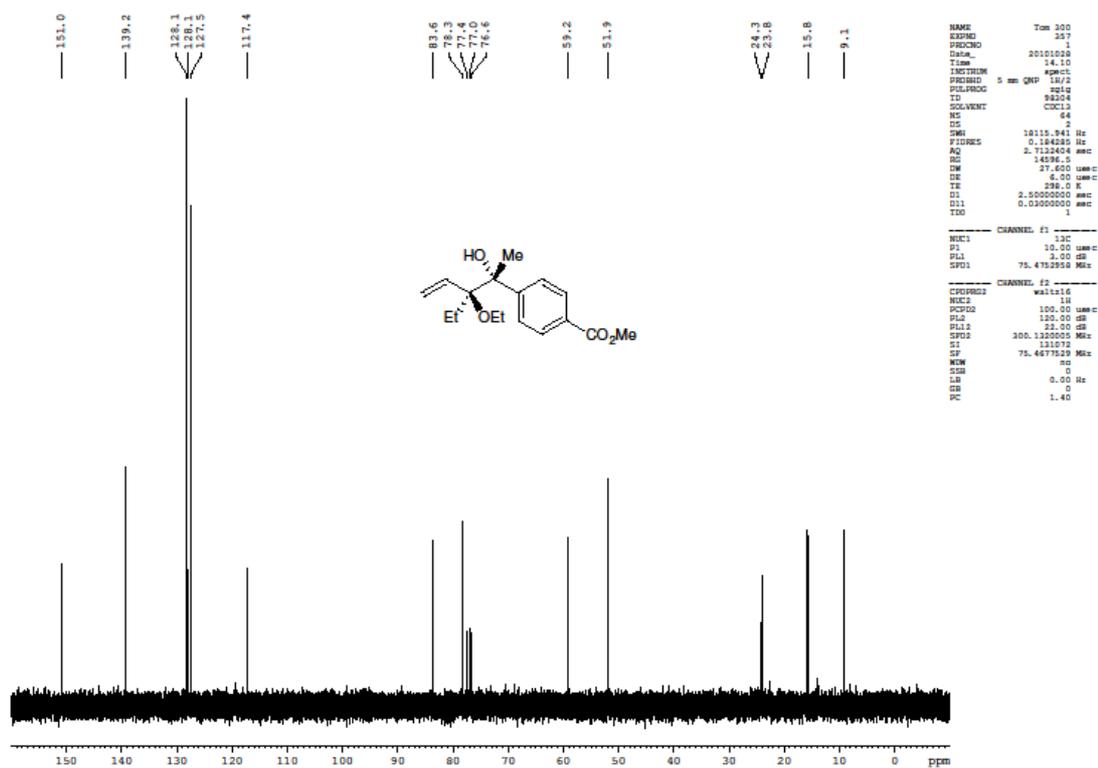
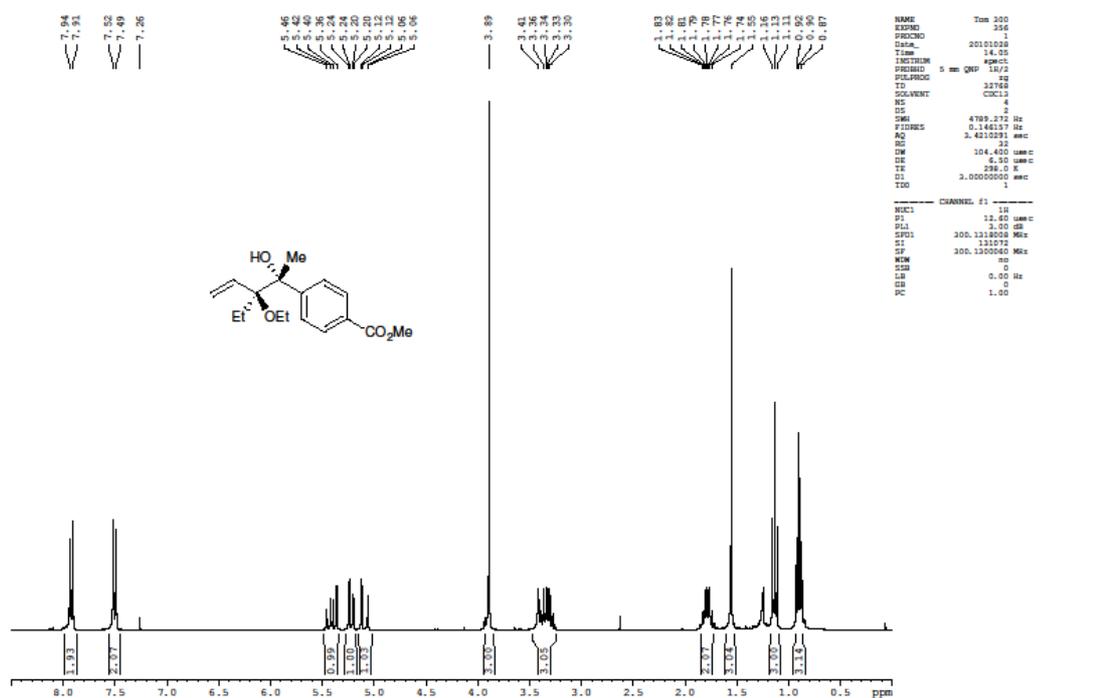
Product 14b:



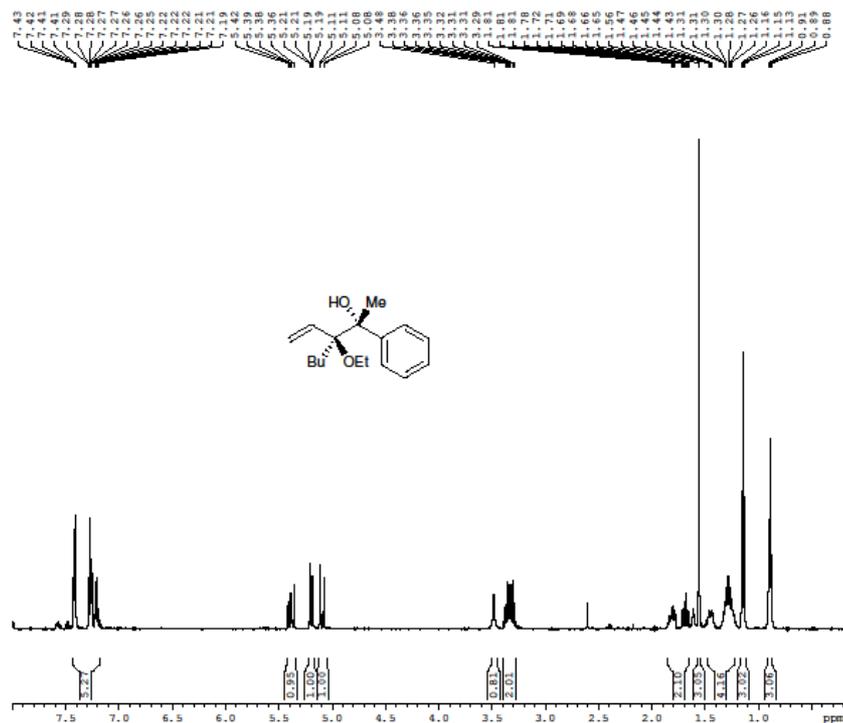
Product 14c:



Product 14e:

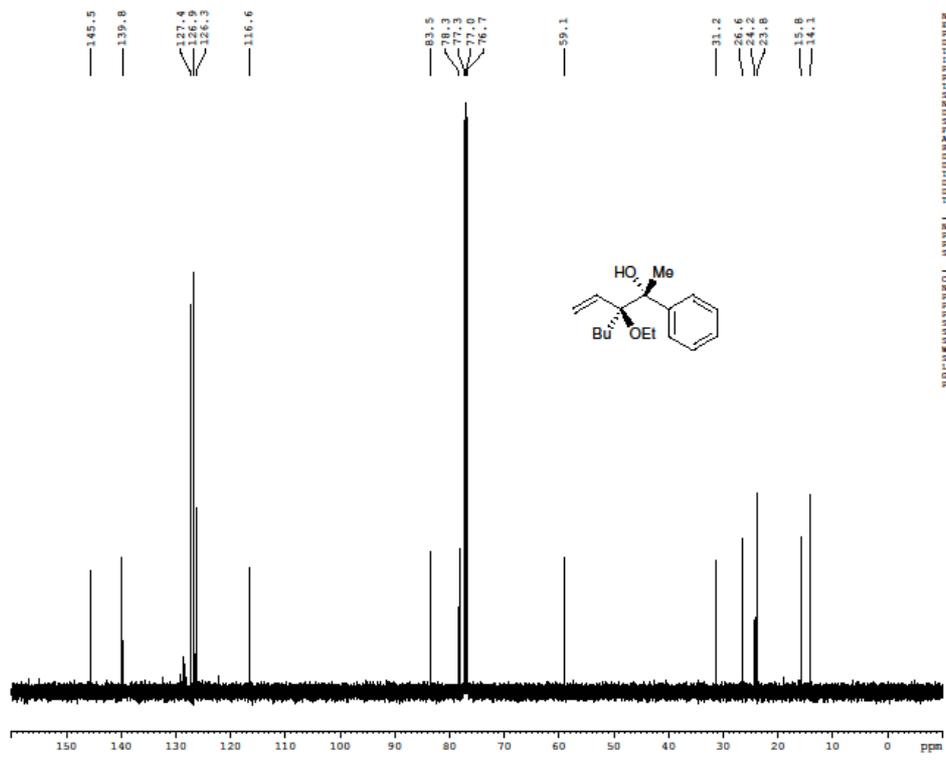


Product 14f:



```

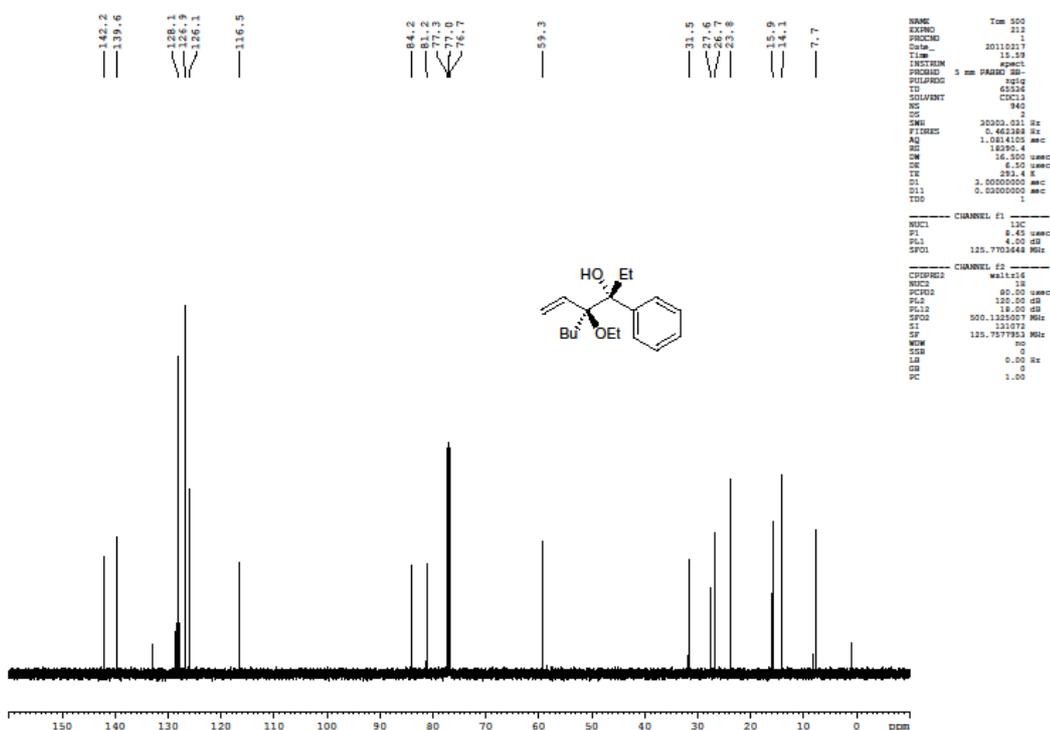
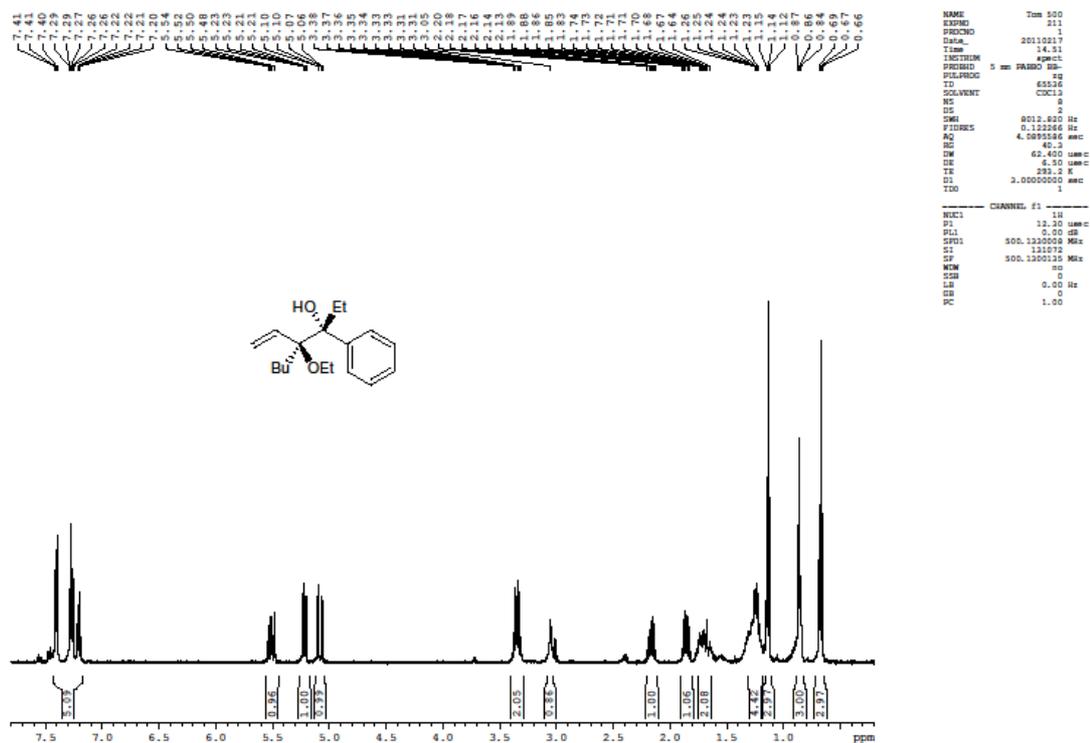
NAME          14f
EXPNO         200
PROCNO        1
Date_         20110217
Time          12.47
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            2
DS            2
SWH           8012.820 Hz
FIDRES       0.122566 Hz
AQ           4.5895586 sec
RG           71.0
WDW           EM
SSB           0
GB            0
PC            1.00
===== CHANNEL f1 =====
NUC1          13C
P1            12.50 usec
PL1           0.00 dB
SFO1          500.1300000 MHz
SI            131072
SF            500.1300136 MHz
WDW           EM
SSB           0
GB            0
PC            1.00
    
```



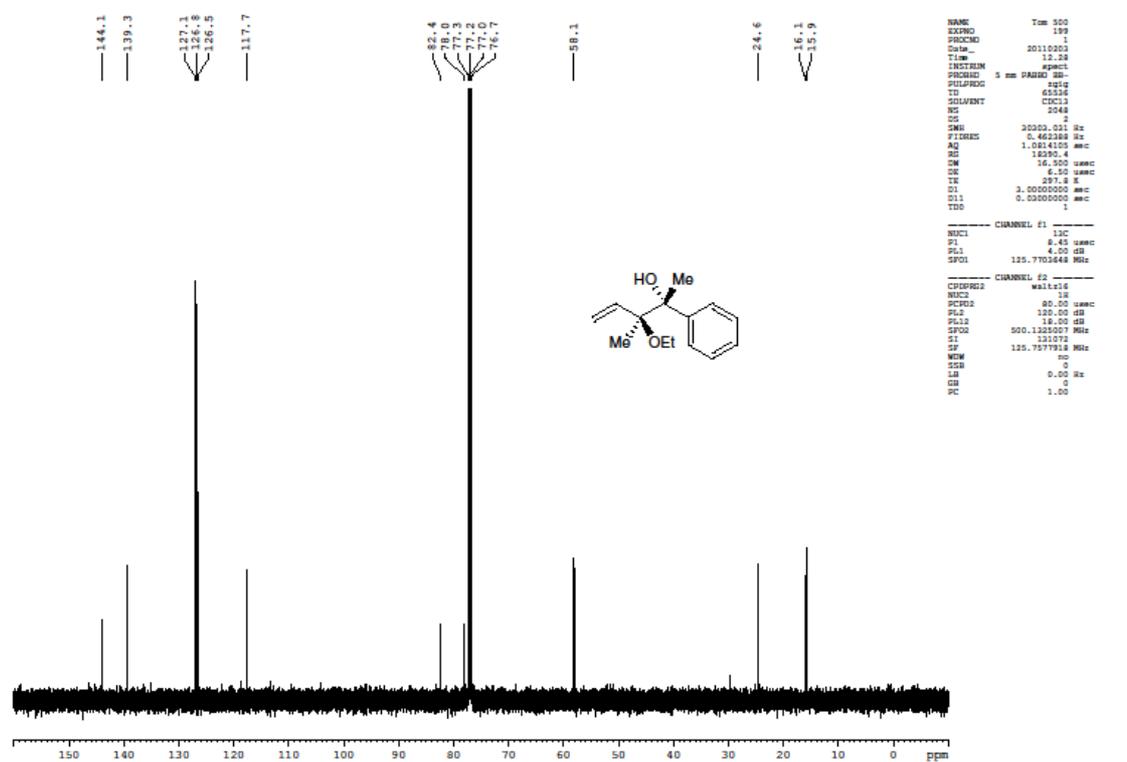
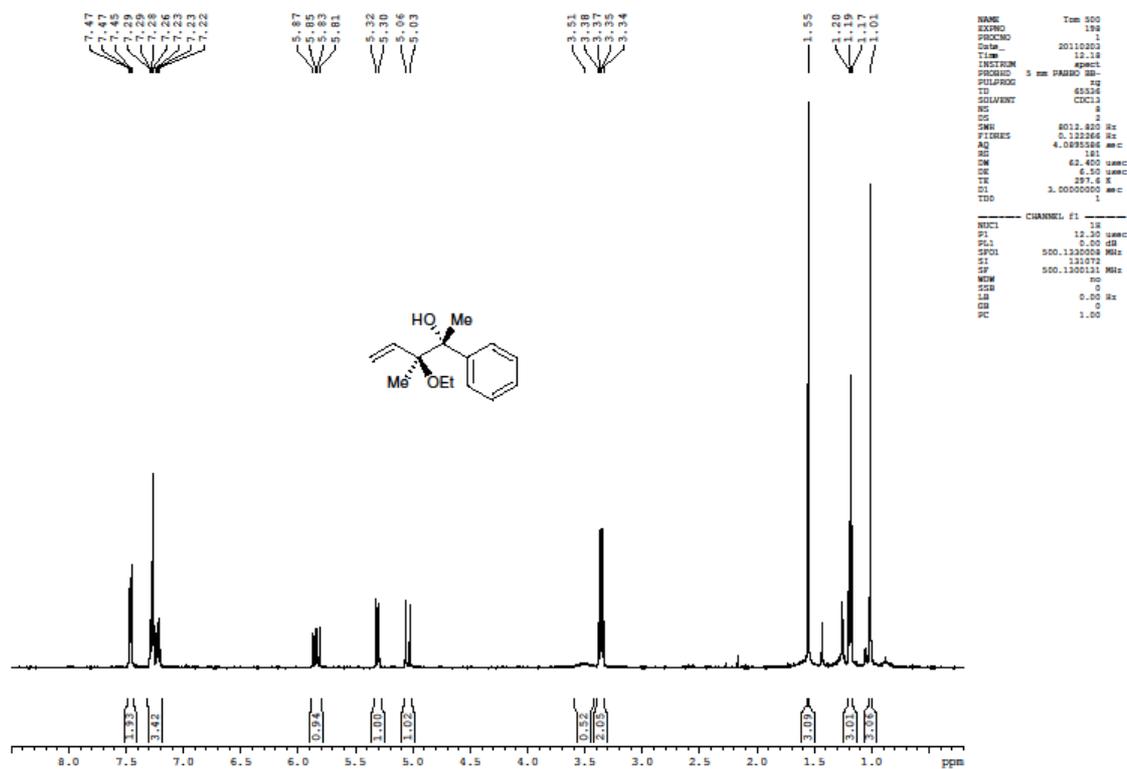
```

NAME          14f
EXPNO         200
PROCNO        1
Date_         20110217
Time          14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            2
DS            2
SWH           30003.021 Hz
FIDRES       0.462288 Hz
AQ           1.0814155 sec
RG           18290.4
WDW           EM
SSB           0
GB            0
PC            1.00
===== CHANNEL f1 =====
NUC1          13C
P1            8.45 usec
PL1           4.00 dB
SFO1          125.7702648 MHz
===== CHANNEL f2 =====
NAME          14f
EXPNO         200
PROCNO        1
Date_         20110217
Time          14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            2
DS            2
SWH           30003.021 Hz
FIDRES       0.462288 Hz
AQ           1.0814155 sec
RG           18290.4
WDW           EM
SSB           0
GB            0
PC            1.00
===== CHANNEL f2 =====
NUC1          13C
P1            8.45 usec
PL1           4.00 dB
SFO1          125.7702648 MHz
===== CHANNEL f2 =====
NAME          14f
EXPNO         200
PROCNO        1
Date_         20110217
Time          14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            2
DS            2
SWH           30003.021 Hz
FIDRES       0.462288 Hz
AQ           1.0814155 sec
RG           18290.4
WDW           EM
SSB           0
GB            0
PC            1.00
===== CHANNEL f2 =====
NUC1          13C
P1            8.45 usec
PL1           4.00 dB
SFO1          125.7702648 MHz
===== CHANNEL f2 =====
NAME          14f
EXPNO         200
PROCNO        1
Date_         20110217
Time          14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            2
DS            2
SWH           30003.021 Hz
FIDRES       0.462288 Hz
AQ           1.0814155 sec
RG           18290.4
WDW           EM
SSB           0
GB            0
PC            1.00
===== CHANNEL f2 =====
NUC1          13C
P1            8.45 usec
PL1           4.00 dB
SFO1          125.7702648 MHz
    
```

Product 14g:



Product 14h:



Product 14i:

