SUPPORTING INFORMATION

Oxidative Cyclorelease from Soluble Polymer Supports

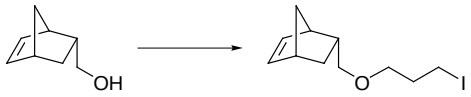
Hua Liu, Shuangyi Wan, and Paul E. Floreancig
Department of Chemistry, University of Pittsburgh, PA 15260

Table of Contents:

General Experimental:

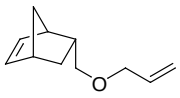
Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were recorded at 300 MHz and 75 MHz, respectively, or at 500 MHz and 125 MHz if specified. The chemical shifts are given in parts per million (ppm) on the delta (δ) scale. The solvent peak or the internal standard tetramethylsilane were used as reference values. For ^{1}H NMR: $CDCl_3$ = 7.27 ppm, TMS = 0.00 ppm. For ^{13}C NMR: $CDCl_3$ = 77.23, TMS = 0.00. For proton data: s = singlet; d = doublet; t = triplet; q = quartet; p = pentet; dd = doublet of doublets; dt = doublet of triplets; ddd = doublet of doublet of doublets; dddd = doublet of doublet of doublet of doublets; ddt = doublet of doublet of triplets; ddq = doublet of doublet of quartets; br = broad; m = multiplet; app t = apparent triplet; app q = apparent quartet; app p = apparent pentet.

Reagent grade ethyl acetate and hexanes (commercial mixture) were used without further purification for chromatography. Reagent grade methylene chloride (CH_2Cl_2) was distilled from CaH_2. Diethyl ether (Et_2O) and tetrahydrofuran (THF) were dried by passing through aluminum drying column. Anhydrous methanol (MeOH), acetonitrile (CH_3CN) were used without purification. All reactions were conducted under a nitrogen atmosphere unless otherwise specified.



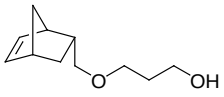
Reagents and Conditions
a) NaH, DMF, then allyl bromide. b) $(Sia)_2BH$, THF, then NaOOH.
c) MsCl, Et_3N, CH_2Cl_2. d) NaI, acetone, reflux.

Scheme 1.



5-Allyloxymethylbicyclo[2.2.1]hept-2-ene

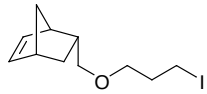
To 5-norbornene-2-methanol (1.10 g, 10 mmol) in DMF (100 mL) at 0 °C was added NaH (0.96g, 40 mmol). The reaction mixture was stirred for 5 min, then allyl bromide (4.84 g, 40 mmol) was added. The reaction mixture was slowly warmed to room temperature and stirred overnight. The reaction was quenched by adding water (50 mL) and extracted with prntane (3×100 mL). The organic layers were then combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10 % Et_2O in pentanes) to provide desired product (1.50 g, quantitative): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.10 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.82-5.97 (m, 2H), 5.28 (dd, *J* = 10.6, 4.4 Hz, 1H), 5.09-5.16 (m, 1H), 3.85-3.92 (m, 2H), 3.13 (dd, *J* = 9.1, 6.6 Hz, 1H), 3.02 (t, *J* = 9.0 Hz, 1H), 2.90 (br, 1H), 2.76 (br, 1H), 2.33 (m, 1H), 1.79 (ddd, *J* = 12.9, 9.2, 2.5 Hz, 1H), 1.19-1.42 (m, 2H), 0.46 (m, 1H).



3-(Bicyclo[2.2.1]hept-5-en-2ylmethoxy)propan-1-ol

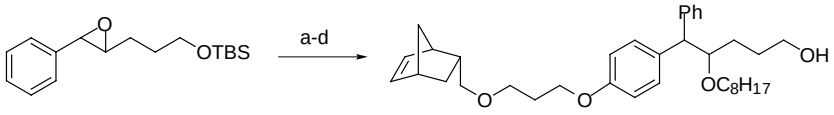
To 2-methylbutene (2.8 g, 4.24 mL, 40 mmol) in THF (100 mL) at –10 °C was added BH_3•THF (20 mL, 1.0 M, 20 mmol) dropwise. The reaction mixture was stirred at 0 °C for 45 minutes, then allyloxynorbornene (1.50 g, 10 mmol) was added. The reaction was stirred for 4 h and quenched by adding water, 10 % NaOH (40 mL), and 30% H_2O_2 (40 mL). The mixture was stirred overnight then extracted with EtOAc (2×100 mL). The organic layers were combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (30%-50% Et_2O in pentane) to provide desired product (1.45 g, 87%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.10 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.90 (dd, *J* = 5.6, 2.9 Hz, 1H), 3.76 (t, *J* = 5.5 Hz, 2H), 3.50-3.63 (m, 2H), 3.14 (dd, *J* = 9.2, 6.7 Hz, 1H), 3.03 (t, *J* = 9.0 Hz, 1H), 2.85 (br, 1H), 2.76 (br, 1H), 2.55 (bs, 1H), 2.31 (m, 1H), 1.74-1.81 (m, 3H), 1.40 (dd, *J* = 8.1, 2.0 Hz, 1H), 1.19-1.23 (m, 1H), 0.48 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 132.1, 74.8, 69.4, 61.2, 49.2, 43.7, 41.9, 38.4, 31.5, 28.9, IR (neat) 3333, 3058, 2959, 2867, 1445, 1373, 1345, 1096, 835, 720 cm^{-1}, HRMS (EI) Calcd for $C_{11}H_{18}O_2$ 182.1307, found 182.1312.

5-(3-Iodopropoxymethyl)bicyclo[2.2.1]hept-2-ene (4)



To the norbornenyl alcohol (1.45 g, 8.7 mmol) in CH_2Cl_2 (50 mL) at 0 °C was added Et_3N (3.51 g, 34.7 mmol). The reaction mixture was stirred for 5 mintues and MsCl (1.49 g, 13.0 mmol) was added dropwise. The reaction was stirred for 0.5 h then quenched by adding water (50 mL). The aqueous was extracted with CH_2Cl_2 (250 mL). The organic layers were then combined and washed with saturated aqueous $NaHCO_3$. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue was used for the next step without further purification.

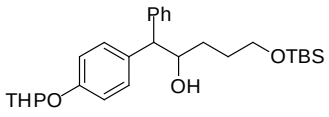
To the resulting mesylate in acetone (40 mL) was added NaI (3.90 g, 26.0 mmol). The mixture was stirred at reflux for 3 h. After cooling, the mixture was diluted with Et_2O (250 mL) and washed with 10% $Na_2S_2O_3$ and water. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (20% Et_2O in pentane) to provide the desired product (2.26 g, 89.1 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.06 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.88 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.31-3.44 (m, 2H), 3.23 (t, *J* = 6.7 Hz, 2H), 3.11 (dd, *J* = 9.2, 5.9 Hz, 1H), 2.96 (t, *J* = 9.2 Hz, 1H), 2.84 (br, 1H), 2.73 (br, 1H), 2.27 (m, 1H), 1.99 (tt, *J* = 6.2 Hz, 2H), 1.75 (ddd, *J* = 9.9, 8.6, 3.8 Hz, 1H), 1.37 (dd, *J* = 8.0, 1.8 Hz, 1H), 1.18 (d, *J* = 8.1 Hz, 1H), 0.40-0.45 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.2, 132.6, 74.8, 70.0, 49.5, 44.1, 42.3, 38.8, 33.5, 29.2, 3.9; IR (neat) 3056, 2963, 2864, 1462, 1344, 1181, 1111, 835, 720 cm^{-1}; HRMS (EI) Calcd for $C_{11}H_{17}OI$ 292.0324, found 292.0305.



Reagents and Conditions
a) THPOPhMgBr, CuI, THF. b) NaH, DMF, then $C_8H_{17}I$. c) PPTs, MeOH. d) **4**, K_2CO_3, DMF.

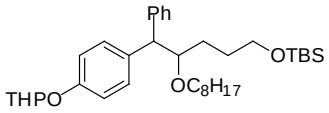
Scheme 2.

5-(*tert*-butyldimethylsilanyloxy)-1-phenyl-1-[4-(tetrahydropyran-2-yloxy)phenyl]-pentan-2-ol



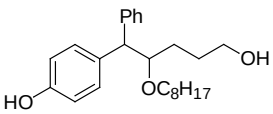
To a suspension of copper (I) iodide (1.95 g, 10.25 mmol) in THF (80 mL) at –40 °C was added 4-(tetrahydropyanyloxy)phenylmagnesiumbromide (140 mL, 1.46 M, 205 mmol). The mixture was stirred for 15 min then *tert*-butyldimethyl-[3-(3-phenyloxiranyl)propoxy]silane[1] (15 g, 51.25 mmol) was added. The reaction was warmed to room temperature and stirred overnight, then was quenched by adding saturated aqueous NH_4Cl (100 mL) and bubbling air through the mixture for one hour. The reaction

mixture was extracted with ethyl acetate (2×200 mL) and the organic layers were combined and washed with water and brine. The organic layer was then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was then purified by flash chromatography (10 % EtOAc in hexanes) to provide desired product (19.16 g, 79.7 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.3 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 2H), 7.32 (d, *J* = 8.6 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 5.47 (t, *J* = 3.0 Hz, 1H), 4.44 (d, *J* = 8.0 Hz, 1H), 3.99 (m, 2H), 3.72 (m, 3H), 2.37 (bs, 1H), 1.37-2.14 (m, 10H), 1.00 (s, 9H), 0.16 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.5, 142.0, 135.63, 129.0, 128.6, 128.4, 126.4, 116.3, 96.1, 73.4, 63.1, 61.8, 57.8, 31.8, 30.2, 29.0, 25.8, 25.1, 18.7, 18.1, -5.5; IR (neat) 3463, 3059, 3028, 2949, 2856, 1740, 1609, 1508, 1471, 1238, 1178, 1038, 835, 700 cm^{-1}; HRMS (EI) Calcd for $C_{28}H_{42}O_4Si$ 470.2852, found 470.2841.



***tert*-Butyldimethyl-(4-octyloxy-5-phenyl-[4-(tetrahydropyran-2-yloxy)phenyl]-pentyloxy)silane**

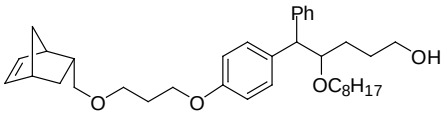
To the homobenzylic alcohol (9.38 g, 20 mmol) in DMF (150 mL) at 0 °C was added NaH (1.92g, 80 mmol) with stirring. The reaction mixture was allowed to stir for 5 min, then octyl iodide (19.2 g, 80 mmol) was added. The reaction mixture was slowly warmed to room temperature and stirred overnight. The reaction was quenched by adding water (100 mL) and extracted with hexanes (3×200 mL). The organic layers were then combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (10 % EtOAc in hexanes) to provide desired product (7.4 g, 64.2 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.0 Hz, 2H), 7.36 (t, *J* = 7.7 Hz, 3H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 8.8 Hz, 2H), 5.46 (t, *J* = 3.0 Hz, 1H), 3.87-4.09 (m, 3H), 3.66 (m 3H), 3.49 (dt, *J* = 8.6, 6.6 Hz, 1H), 3.16 (m, 1H), 1.19-2.15 (m, 25H), 0.98 (s, 9H), 0.13 (s, 3H), 0.12 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.5, 142.8, 135.9, 129.3, 128.8, 127.9, 125.9, 116.2, 96.3, 82.2, 82.1, 70.2, 70.16, 63.2, 61.9, 55.7, 31.8, 30.3, 30.0, 29.3, 29.2, 28.9, 28.8, 28.5, 26.0, 25.9, 25.2, 22.6, 18.8, 18.2, 14.1, -5.4; IR (neat) 2928, 2855, 1742, 1509, 1470, 1238, 1107, 1039, 835, 775, 699cm^{-1}; HRMS (EI) Calcd for $C_{36}H_{57}O_4Si$ (M^+-H) 581.4004, found 581.4051.



4-(5-hydroxy-2-octyloxy-1-phenylpentyl)phenol

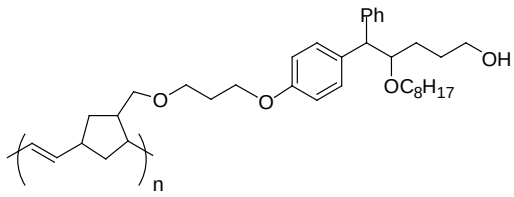
To a solution of *tert*-butyldimethyl-(4-octyloxy-5-phenyl-[4-(tetrahydropyran-2-yloxy)phenyl]-pentyloxy)silane (5.87 g, 10.10 mmol) in MeOH (80 mL) was added PPTs (127 mg, 0.51 mmol). The reaction mixture was stirred at room temperature overnight and then concentrated under reduced pressure. The residue was dissolved in 300 mL EtOAc and washed with water and brine. The organic layer was then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash column

chromatography (20 % EtOAc in hexanes to 50 % EtOAc in hexanes) to provide desired product (3.705 g, 95.7 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.2 Hz, 2H), 7.25-7.37 (m, 3H), 7.20 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.3 Hz, 2H), 4.10 (m, 2H), 3.67 (m, 2H), 3.51 (dt, *J* = 8.6, 6.3 Hz, 1H), 3.17 (dt, *J* = 8.5, 6.7 Hz, 1H), 1.20-1.76 (m, 18H), 1.01 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.8, 142.8, 133.6, 129.2, 128.4, 127.9, 125.9, 115.4, 82.1, 70.4, 62.7, 55.2, 31.6, 29.66, 29.1, 29.0, 28.9, 27.6, 25.7, 22.5, 14.0; IR (neat) 3325, 3062, 3026, 2928, 2856, 1614, 1514, 1244, 1174, 1101, 1048, 699 cm^{-1}; HRMS (EI) Calcd for $C_{25}H_{36}O_3$ (M^+-H_2O) 366.2559, found 366.2571.



5-{4-[3-(Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-4-octyloxy-5-phenylpentan-1-ol (6)

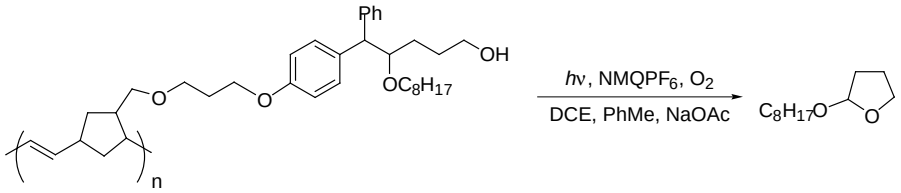
To a stirred mixture of the phenol (3.71 g, 9.66 mmol) and anhydrous potassium carbonate (1.60 g, 11.6 mmol) in DMF (10 mL) under N_2 was added 5-(3-iodopropoxymethyl)bicyclo[2.2.1]hept-2-ene (3.10 g, 10.6 mmol). The reaction mixture was stirred at room temperature for 36 hours and then partitioned between 50 mL water and 200 mL EtOAc. The aqueous was collected and extracted with EtOAc (200 mL) and the organic layers were combined and washed with water and brine. The organic layer was then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash column chromatography (20% EtOAc in hexanes) to provide desired product (4.73 g, 92%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.1 Hz, 2H), 7.29 (t, *J* = 7.1 Hz, 3H), 7.19-7.23 (m, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 6.13 (dd, *J* = 5.7, 3.0 Hz, 1H), 5.92 (dd, *J* = 5.7, 2.9 Hz, 1H), 3.98-4.07 (m, 4H), 3.50-3.67(m, 4H), 3.35-3.46 (m, 2H), 3.18 (dd, *J* = 9.2, 6.6 Hz, 1H), 3.02-3.11 (m, 1H), 2.91 (bs, 1H), 2.81 (bs, 1H), 2.37 (m, 1H), 2.03 (m, 3H), 1.83 (ddd, *J* = 11.6, 9.2, 3.8 Hz, 1H), 1.14-1.72 (m, 18H), 0.91 (t, *J* = 6.8 Hz, 3H), 0.50 (m, 1H); ^{13}C NMR (75 MHz, CDC_{l3}) δ 157.5, 142.9, 137.1, 134.6, 132.4, 129.3, 128.7, 128.1, 126.1, 114.5, 82.2, 74.7, 70.5, 67.3, 64.8, 63.1, 55.2, 49.4, 43.9, 42.1, 38.7, 31.8, 29.9, 29.7, 29.3, 29.2, 29.1, 28.3, 25.9, 22.6, 14.1; IR (neat) 3428, 3059, 3028, 2930, 2860, 1611, 1510, 1247, 1106, 699 cm^{-1}; HRMS (EI) Calcd for $C_{36}H_{50}O_3$ (M^+-H_2O) 530.3760, found 530.3755.



Polymer-supported 5-(4-propoxyphenyl)-4-octyloxy-5-phenylpentan-1-ol

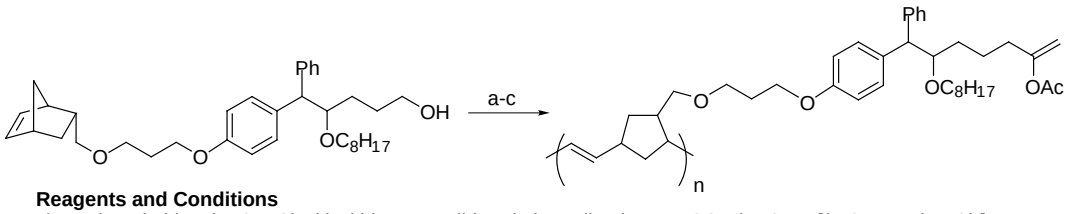
To **6** (112 mg, 0.21 mmol) in a nitrogen flushed flask were added degassed CH_2Cl_2 (0.5 mL) and Grubbs' second-generation catalyst (4 mg, 0.005 mmol). The reaction mixture was refluxed for 30 min, then ethylvinyl ether (1 mL) was added and the reaction was allowed to stir warm for 15 min. The reaction mixture was poured into 100 mL of MeOH. The precipitate was collected and washed with methanol, then dissolved in

toluene. The solvent was removed and the residue was dried under high vacuum overnight (106 mg, 78.8 %): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (d, *J* = 7.1 Hz, 2H), 7.24 (m, 3H), 7.13 (d, *J* = 7.4 Hz, 2H), 6.76 (m, 2H), 5.29 (s, 1H), 5.17 (s, 1H), 3.95 (m, 4H), 3.50 (m, 5H), 3.36 (m, 1H), 3.00 (m, 1H), 2.75 (m, 1H), 1.07-2.33 (complex m, 26H), 0.85 (t, *J* = 6.7 Hz, 3H).



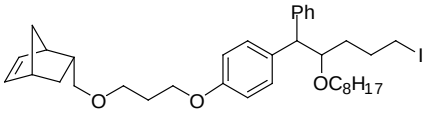
2-Octyloxytetrahydrofuran

To **7** (95 mg, 0.15 mmol) in dichloroethane (6 mL) in a borosilicate flask were added *N*-methylquinolinium hexafluorophosphate (4 mg, 0.015 mmol), sodium acetate (190 mg), anhydrous sodium thiosulfate (190 mg) and toluene (1 mL). The mixture was stirred at room temperature with gentle aeration while irradiating with a medium pressure mercury lamp for 5 h. The reaction mixture was filtered through a small plug of silica gel and the filtrate was concentrated to provide the desired product[1] (21 mg, 72% yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.11 (dd, J=4.0, 1.7 Hz, 1H), 3.91-3.82 (m, 2H), 3.65 (dt, J=9.5, 6.7 Hz, 1H), 3.36 (dt, J=9.5, 6.5, 1H), 2.02-1.80 (m, 4H), 1.58-1.51 (m, 2H), 1.29-1.27 (m, 10 H), 0.88 (t, *J* = 6.9 Hz, 3H).



Reagents and Conditions
a) I_2, Ph_3P, imidazole, CH_2Cl_2. b) Lithium acetylide•ethylene diamine, DMSO. c) HOAc, [(*p*-Cymene)$RuCl_2$]$_2$, Fur_3P, Na_2CO_3, PhMe, 60 °C. d) Grubbs' first generation catalyst, CH_2Cl_2, then ethyl vinyl ether

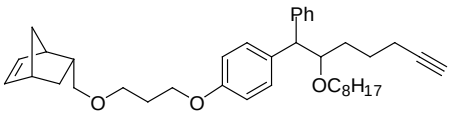
Scheme 3.



5-{4-[4-(5-Iodo-2-octyloxy-1-phenylpentyl)phenyl]butoxymethyl}bicyclo[2.2.1]-hept-2-ene

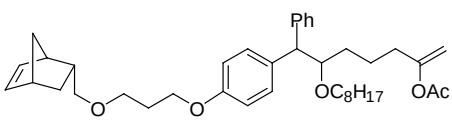
To **6** (3.0 g, 5.5 mmol) in CH_2Cl_2 (50 mL) were added triphenylphosphine (1.63 g, 6.2 mmol) and imidazole (442 mg, 6.2 mmol). The solution was cooled to 0 °C and stirred for 5 min. Iodine (1.6 g, 6.2 mmol) was added portionwise

over 2 min. The mixture was stirred for 2 h in the dark, then was concentrated under reduced pressure. The resulting residue was purified by flash chromatography (20% EtOAc in hexanes) to provide desired product (3.65 g, 99%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.2 Hz, 2H), 7.29 (t, *J* = 7.1 Hz, 3H), 7.20 (d, *J* = 8.3 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.13 (dd, *J* = 5.6, 3.1 Hz, 1H), 5.92 (dd, *J* = 5.6, 2.9, 1H), 3.95-4.07 (m, 4H), 3.50-3.61 (m, 2H), 3.34-3.41 (m, 1H), 3.03-3.21 (m, 5H), 2.91 (bs, 1H), 2.80 (bs, 1H), 2.35 (m, 1H), 1.24-1.86 (m, 21H), 0.92 (t, *J* = 6.6 Hz, 3H), 0.49 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.4, 142.6, 137.0, 134.4, 132.3, 129.2, 128.6, 128.0, 126.0, 114.4, 81.4, 74.6, 70.3, 67.2, 64.7, 55.5, 49.3, 43.8, 42.1, 38.6, 33.4, 31.7, 29.9, 29.6, 29.3, 29.3, 29.1, 29.0, 25.9, 22.6, 14.1, 6.9; IR (neat) 3059, 3028, 2928, 2857, 1610, 1510, 1247, 1101, 1031, 698 cm^{-1}; HRMS (EI) Calcd for $C_{36}H_{51}O_3INa$ (M^++Na) 681.2781, found 681.2813.



5-{4-[4-(2-octyloxy-1-phenylhept-6-ynyl)phenyl]butoxymethyl}bicyclo[2.2.1]-hept-2-ene

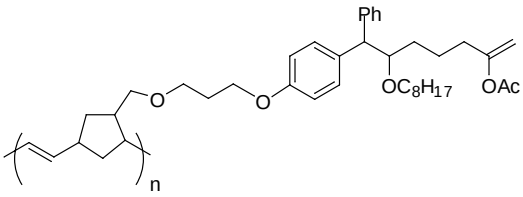
To the alkyl iodide (1.98 g, 3 mmol) in DMSO (16 mL) at 0 °C was added lithium acetylide, ethylendiamine complex, (0.61 g, 90%, 6 mmol). The reaction mixture was slowly warmed to room temperature and stirred for 3 h. Saturated aqueous NH_4Cl (50 mL) was added to quench the reaction. The reaction mixture was extracted with EtOAc (3×100 mL). The organic layers were combined and washed with water and brine. The organic layer was then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10% EtOAc in hexanes) to provide desired product (1.52 g, 91%): ^{1}H NMR (300 MHz $CDCl_3$) δ 7.42 (d, *J* = 7.1 Hz, 2H), 7.31 (t, *J* =7.2 Hz, 3H), 7.23 (d, *J* = 8.6, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.16 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.95 (dd, *J* = 5.6, 2.8 Hz, 1H), 4.08 (t, *J* = 6.3 Hz, 2H), 4.00 (m, 2H), 3.56-3.64 (m, 2H), 3.43 (m, 1H), 3.26 (dd, *J* = 9.2, 6.6 Hz, 1H), 3.12 (m, 2H), 2.95 (bs, 1H), 2.83 (bs, 1H), 2.40 (m, 1H), 2.10-2.19 (m, 2H), 2.08 (m, 2H), 1.97 (t, *J* = 2.6 Hz, 1H), 1.19-1.85 (m, 19H), 0.95 (t, *J* = 6.7 Hz, 3H), 0.53 (m, 1H); ^{13}C NMR (75 MHz, CDC_{l3}) δ 157.4, 142.8, 137.0, 134.8, 132.3, 129.3, 128.8, 128.0, 125.9, 114.3, 84.3, 81.9, 74.6, 70.3, 68.3, 67.2, 64.7, 55.5, 49.3, 43.9, 42.1, 38.7, 31.7, 30.0, 29.6, 29.3, 29.1, 29.0, 26.0, 24.2, 22.6, 18.4, 14.1; IR (neat) 3310, 3059, 3028, 2929, 2859, 1611, 1510, 1247, 1104, 1031, 699 cm^{-1}; HRMS (EI) Calcd for $C_{40}H_{51}O_3$ (M^+-H) 579.3838, found 579.3834.



Acetic acid 6-{4-[4-(bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-1-methylene-5-octyloxy-6-phenylhexylester (9)

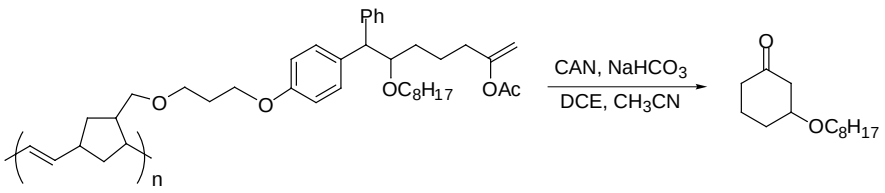
To the alkyne (1.44 g, 2.6 mmol) in toluene (6 mL) was added tri-2-furylphosphine (24 mg, 0.1 mmol), dichloro(*p*-cymene)ruthenium(II)

dimmer (32 mg, 0.05 mmol) and sodium carbonate (22 mg, 0.02 mmol). After stirring at room temperature for a few minutes, acetic acid (0.311 g, 0.3 mL, 5.2 mmol) was added and the reaction mixture was stirred at 60-65 °C for 48 h. The reaction was cooled down and concentrated under reduced pressured. The resulting residue was purified by flash column chromatography (5% EtOAc in hexanes) to provide desired product (0.75 g, 47%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.5 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 3H), 7.16 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 8.4, 2H), 6.09 (dd, *J* = 5.4, 2.9 Hz, 1H), 5.88 (m, 1H), 4.68 (bs, 1H), 4.65 (bs, 1H), 4.00 (t, *J* = 6.1 Hz, 2H), 3.92 (m, 2H), 3.45-3.56 (m, 3H), 3.39 (m, 1H), 3.14 (dd, *J* = 9.1, 6.6 Hz, 1H), 3.05 (dt, *J* = 8.9, 6.3 Hz, 1H), 2.88 (bs, 1H), 2.75 (bs, 1H), 2.33 (m, 1H), 2.14 (m, 2H), 2.07 (s, 3H), 1.98 (m, 2H), 1.78 (ddd, *J* = 11.8, 8.6, 3.8 Hz, 1H), 1.13-1.59 (m, 18H), 0.88 (t, *J* = 6.6 Hz, 3H), 0.46 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.7, 157.3, 156.0, 142.7, 136.8, 134.6, 132.2, 129.1, 128.6, 127.8, 125.8, 114.2, 100.9, 81.9, 74.5, 70.2, 67.1, 65.6, 64.6, 55.4, 49.2, 43.7, 42.0, 38.5, 33.1, 31.8, 31.6, 19.9, 29.5, 29.2, 29.0, 28.9, 25.9, 22.5, 21.8, 20.8, 15.1, 13.9; IR (neat) 3059, 3028, 2928, 2856, 1758, 1611, 1510, 1246, 1104, 1021, 699 cm^{-1}; HRMS (EI) Calcd for $C_{40}H_{56}O_5Na$ (M^++Na) 639.4025, found 639.4013.



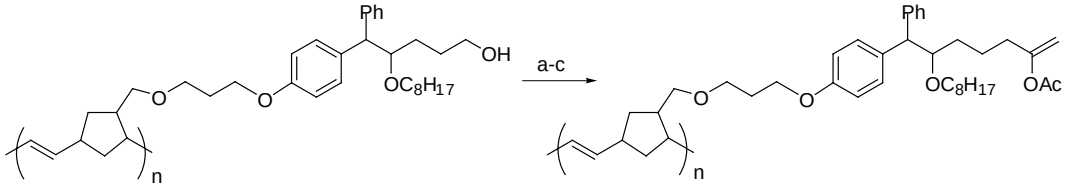
Polymer 10

To **9** (130 mg, 0.21 mmol) in degassed CH_2Cl_2 (1 mL) was added Grubbs' first-generation catalyst (3.5 mg, 0.0042 mmol). The reaction mixture was stirred at room temperature overnight then was warmed to 40 °C for two hours. Ethylvinyl ether (1 mL) was added and the reaction was allowed to stir for 15 min. The reaction mixture was then poured into 100 mL of MeOH. The precipitate was collected and washed with methanol, then dissolved in toluene. The solvent was removed and the residue was dried under high vacuum overnight (86.5 mg, 57%): ^{1}HNMR (300 MHz, $CDCl_3$) δ 7.30 (d, J=7.21 Hz, 2H), 7.21 (m, 3H), 7.14 (d, *J* = 7.4 Hz, 2H), 6.75 (m, 2H), 5.29 (s, 1H), 5.19 (s, 1H), 4.64 (d, *J* = 5.9 Hz, 2H), 4.00 (m, 4H), 3.29-3.46(m, 5H), 2.97 (m, 1H), 1.09-2.46 (m, 28H), 0.87 (t, *J* = 6.6 Hz, 3H); IR (in toluene) 3060, 3027, 2927, 2858, 1756, 1607, 1510, 1246, 1102, 696 cm^{-1}.



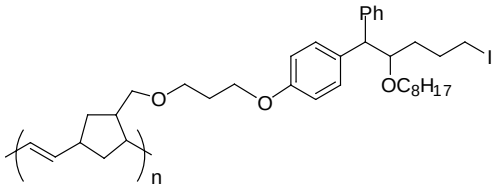
3-Octyloxycyclohexanone

To **10** (0.0865 g, 0.12 mmol) in dichloroethane (5 mL) were added 4 Å molecular sieved (173 mg), and $NaHCO_3$ (173 mg). The reaction mixture was then warmed to 40 °C and stirred for 15 min. Ceric ammomium nitrate (132 mg, 0.24 mmol) in CH_3CN (2 mL) was added dropwise and the reaction was stirred at 40 °C overnight. The mixture was poured into methanol and filtered through a plug of silica gel. The filtrate was concentrated and dissolved in 8:2 hexanes and EtOAc, filtered and concentrated to provide desired product[1] (21 mg, 85%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.65-3.75 (m, 1H), 3.38 (app t, *J* = 6.6 Hz, 2H), 2.57 (dd, *J* = 14.0, 3.8 Hz, 1H), 2.41 (dd, *J* = 14.0, 7.3 Hz, 1H), 2.28 (app t, J=6.4 Hz, 2H), 1.93-2.05 (m, 2H), 1.78-1.87 (m, 1H), 1.65-1.69 (m, 1H), 1.48-1.55 (m, 2H), 1.26 (br s, 10H), 0.85 (t, *J* = 6.3 Hz, 3H).



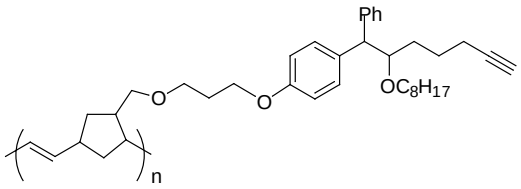
Reagents and Conditions
a) I_2, Ph_3P, imidazole, CH_2Cl_2. b) Lithium acetylide•ethylene diamine, DMSO. c) HOAc, $[(p\text{-Cymene})RuCl_2]_2$, Fur_3P, Na_2CO_3, PhMe, 60 °C.

Scheme 4.



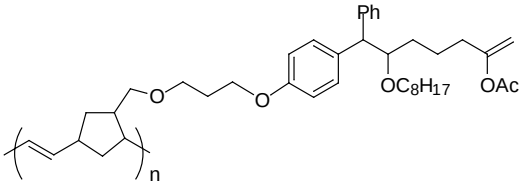
Polymer-supported 5-(4-propoxyphenyl)-4-octyloxy-5-phenylpentaniodide

To **7** (224 mg, 0.35 mmol) in CH_2Cl_2 (5 mL) were added PPh_3 (184 mg, 0.70 mmol) and imidazole (48 mg, 0.70 mmol). The solution was cooled to 0 °C then I_2 (179 mg, 0.70 mmol) was added. The reaction was slowly warmed to room temperature and stirred overnight (precipitate appeared after addition of I_2). The reaction mixture was filtered and concentrated down to 2 mL. The mixture was poured slowly into 100 mL MeOH and the red precipitate was dissolved in toluene. The solvent was concentrated and the residue was dried under vacuum overnight to provide the desired iodide (246 mg, 94%): ^{1}H NMR (300 MHz $CDCl_3$) δ 7.13-7.29 (m, 7H), 6.76 (d, J=7.49 Hz, 2H), 5.28 (m, 2H), 3.90 (m, 4H), 3.48 (m, 4H), 3.30 (dd, J=13.44, 6.89 Hz, 1H), 3.07 (t, *J* = 7.1 Hz, 2H), 3.05 (dd, *J* = 15.3, 7.3 Hz, 1H), 1.15-2.16 (m, 25H), 0.84 (t, *J* = 7.1 Hz, 3H).



Polymer-supported 5-(4-propoxyphenyl)-4-octyloxy-5-phenylpent-1-yne

To the polymer-supported iodide (291 mg, 0.39 mmol) in DMSO (0.5 mL) and THF (2 mL) at 0 °C was added lithium acetylide (71 mg, 0.77 mmol). The reaction was warmed to room temperature and stirred overnight. The mixture was poured slowly into methanol, then he precipitate was collected and dissolved in toluene. The solvent was concentrated and the residue was dried under vacuum to provide the desired alkyne (201 mg, 80%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.78-7.46 (m, 9H), 5.21-5.25 (m, 2H), 3.96 (m, 4H), 3.00-3.50 (m, 6H), 1.12-2.62 (complex m, 28H), 0.71 (t, J=7.10 Hz, 3H); IR (in toluene) 3309, 3061, 3027, 2925, 2859, 1640, 1509, 1246, 1103, 1030, 695 cm^{-1}.

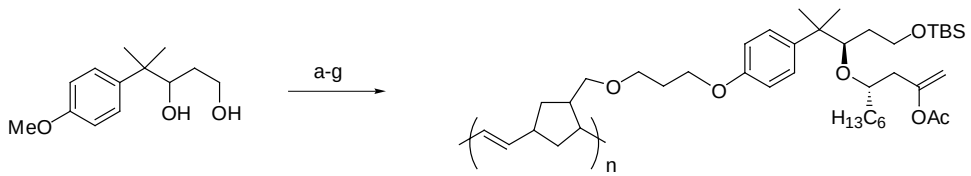


Enol acetate 10

To the polymer-supported alkyne (361 mg, 0.56 mmol) in toluene (6 mL) were added tri-2-furylphosphine (5 mg, 0.02 mmol), dichloro(*p*-cymene)ruthenium(II) dimer (7 mg, 0.01 mmol) and sodium carbonate (5 mg, 0.04 mmol). After stirring at room temperature for a few minutes, AcOH (334 mg, 5.6 mmol) was added and the reaction was stirred at 60-70 °C for 24 hours. Tri-2-furylphosphine (3 mg, 0.01 mmol), dichloro(*p*-cymene)ruthenium(II) dimer (3 mg, 0.006 mmol) and sodium carbonate (3 mg, 0.02 mmol) and acetic acid (0.167 g, 2.78 mmol) were added, and the reaction mixture was stirred at 60-70 °C for another 24 hours. The solution was concentrated and the residue was slowly poured into 100 mL methanol. The precipitate was collected and dissolved in toluene. The solvent was concentrated and the residue was dried under vacuum (385 mg, 96%).

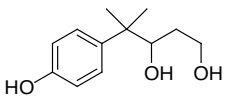
Cyclization of 10

Cyclization proceeded as before with **10** (125 mg, 0.17 mmol), CAN (190 mg, 0.34 mmol), $NaHCO_3$ (250 mg) and 4Å molecular sieves (250 mg) in DCE (7mL) and CH_3CN (3 mL) to provide **11** (30 mg, 70%).



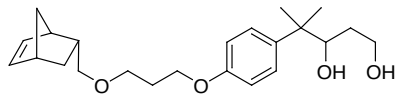
Reagent and Conditions
a) $AlCl_3$, EtSH, CH_2Cl_2. b) **4**, K_2CO_3, DMF. c) *n*-Heptanal, *p*-TsOH, C_6H_6, reflux. d) Grubbs' 1st generation catalyst, CH_2Cl_2, then ethyl vinyl ether. e) Allenyl tributyltin, $TiCl_4$/Ti(O*i*-Pr)$_4$ (6:5), CH_2Cl_2, -78 °C. f) HOAc, [(*p*-cymene)$RuCl_2$]$_2$, Na_2CO_3, PhMe, 60 °C. g) TBSCl, imidazole, CH_2Cl_2.

Scheme 5.



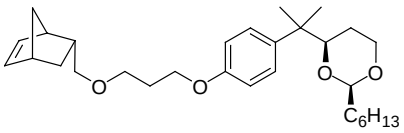
4-(4-hydroxyphenyl)-4-methylpentane-1, 3-diol

To $AlCl_3$ (6.8 g, 60 mmol) in EtSH (20 mL) and CH_2Cl_2 (100 ml) at 0 °C was added 4-(4-methoxyphenyl)-4-methylpentane-1, 3-diol[2] (2.24 g, 10 mmol). The reaction was stirred at 0 °C for 1 h, then was warmed to room temperature and stirred for 2 additional hours. The reaction was quenched by adding aqueous HCl. Air was gently bubbled into the stirred reaction mixture for 5 h, then the organics were extracted into EtOAc (6×50 mL). The organic layers were combined and washed with water and brine, then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was recrystallized from EtOAc/hexanes to provide desired product as white solid (1.26 g, 60%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 8.7, 2H), 4.98 (s, 1H), 3.78-3.83 (m, 3H), 2.5 (bs, 1H), 2.03 (bs, 1H), 1.60 (m, 2H), 1.30 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (75 MHz, acetone-d_6) δ 155.3, 138.6, 127.6, 114.7, 77.6, 60.9, 41.4, 34.2, 26.0, 22.1; IR (in acetone) 3345, 2967, 2882, 1613, 1515, 1365, 1233, 1181, 1050, 833 cm^{-1}; HRMS (EI) Calcd for $C_{12}H_{16}O_2$ (M^+-H_2O) 192.1250, found 192.1151.



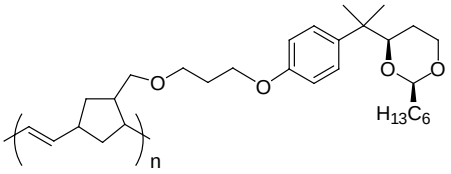
4-{4-[3-(bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-4-methylpentane-1,3-diol

To the phenol (1.05 g, 5 mmol) in DMF (30 mL) was added anhydrous potassium carbonate (0.83 g, 6 mmol) and the norbornenyl-tagged iodide (1.75 g, 6 mmol). The reaction was stirred at room temperature for 12 hours and then warmed to 60 °C for 20 h. After cooling the reaction was quenched by adding 50 mL water and extracted with EtOAc (3×80 mL). The organic layers were combined and washed with water and brine. The organic layer was then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (40% hexanes in EtOAc to 30 % hexanes in EtOAc) to provide desired product (1.18 g, 65.7%): ^{1}HNMR (300 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.9 Hz, 2H), 6.09 (dd, *J* = 5.6, 3.0 Hz, 1H), 5.86 (dd, *J* = 5.6, 2.9 Hz, 1H), 4.04 (t, *J* = 6.3 Hz, 2H), 3.77 (m, 3H), 3.54 (m, 2H), 3.14 (dd, *J* = 9.2, 6.6, 1H), 3.01 (t, *J* = 9.1 Hz, 1H), 2.86, (br, 1H), 2.76 (br, 1H), 2.31 (m, 1H), 2.00 (tt, *J* = 6.3, 4H), 1.78 (ddd, *J* = 11.5, 9.0, 3.6 Hz, 1H), 1.49-1.65 (m, 3H), 1.19-1.40 (m, 7H), 0.45 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.4, 138.4, 137.1, 132.4, 127.5, 114.3, 80.3, 74.7, 67.3, 64.9, 62.6, 49.4, 43.9, 42.1, 41.8, 38.7, 32.8, 29.7, 29.1, 24.4, 23.2; IR (neat) 3426, 3058, 2960, 2868 1671, 1513, 1387, 1250, 1185, 1102, 1064, 831, 721 cm^{-1}; HRMS (EI) Calcd for $C_{23}H_{32}O_3$ (M^+-H_2O) 356.2351, found 356.2339.



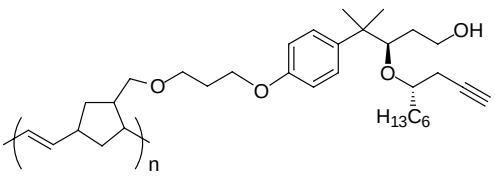
4-(1-{4-[3-(Bicyclo[2.2.1]hept-5-en-2-ylmthoxy)propoxy]phenyl}-1-ethylethyl)-2-hexyl-[1, 3]dioxane

To the norbornenyl-tagged diol (1.18 g, 3.28 mmol) in benzene (10 mL) was added heptanal (375 mg, 3.3 mmol) and a catalytic amount of *p*-TsOH. The reaction was warmed to reflux and stirred for 14 h. After cooling the reaction was concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10% EtOAc in hexanes) to provide the desired product (1.15 g, 77%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.2 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.08 (dd, *J* = 5.7, 3.0 Hz, 1H), 5.87 (dd, *J* = 5.6, 2.9, Hz, 1H), 4.46 (t, *J* = 5.1 Hz, 1H), 4.04 (m, 3H), 3.44-3.66 (m, 4H), 3.14 (dd, *J* = 9.2, 6.6 Hz, 1H), 3.01 (t, *J* = 9.1 Hz, 1H), 2.86 (br, 1H), 2.78 (br, 1H), 2.30 (m, 1H), 2.00 (tt, *J* = 6.2 Hz, 2H), 1.78 (ddd, *J* = 10.4, 7.9, 2.6 Hz, 1H), 1.19-1.59 (m, 20H), 0.86 (t, *J* = 6.3 Hz, 3H), 0.48 (m, 1H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.1, 138.5, 137.0, 132.4, 127.6, 113.7, 102.2, 84.1, 74.6, 67.3, 66.7, 64.7, 49.3, 43.9,42.1, 40.6, 38.7, 34.9, 31.7, 29.7, 29.0, 25.9, 23.9, 22.6, 22.5, 14.0; IR (neat) 3058, 2957, 2859, 1689, 1610, 1513, 1467, 1249, 1116, 1033, 827, 720 cm^{-1}; HRMS (EI) Calcd for $C_{30}H_{46}O_4$ 470.3396, found 470.3409.



Polymer-supported 4-(1-[4-propoxyphenyl]-1-methylethyl)-2-hexyl-[1, 3]dioxane (13)

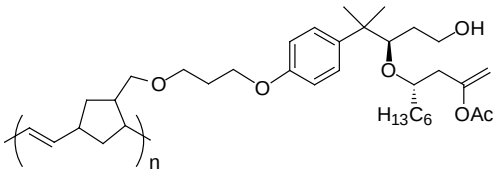
To the norbornenyl-tagged acetal (500 mg, 1.09 mmol) in degassed CH_2Cl_2 (5.0 mL) was added Grubbs' first-generation catalyst (45 mg, 0.06 mmol) and the reaction was stirred at reflux for 30 min. Ethyl vinyl ether (0.5 mL) was added and the reaction was allowed to stir for 15 min. The reaction mixture was poured into 100 mL of MeOH. The precipitate was collected and washed with methanol, then dissolved in toluene. The solvent was removed and the residue was dried under high vacuum overnight to provide the desired polymer (474 mg, 77%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (m, 2H), 6.79 (d, *J* = 8.1 Hz, 2H), 5.32 (s, 1H), 5.20 (m, 1H), 4.45 (t, *J* = 5.0 Hz, 1H), 3.97 (m, 3H), 3.19-3.63 (m, 6H), 1.29-2.60 (m, 27 H), 0.86 (t, *J* = 6.2 Hz, 3H).



Polymer-supported 4-[4-propoxyphenyl]-4-methyl-3-(1-prop-2-ynylheptyloxy)-pentan-1-ol

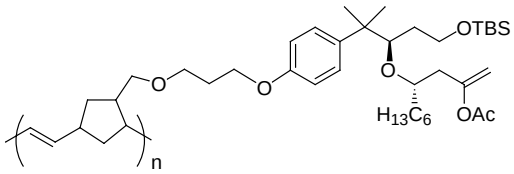
To the polymer-supported acetal (474 mg, 0.84 mmol) and allenyltributyltin (1.39 g, 4.2 mmol) in CH_2Cl_2 at –78 °C was added a mixture of $TiCl_4$ (990 mg, 5.2 mmol) and Ti(O*i*-Pr)$_4$ (1.19 g, 4.2 mmol) over two hours. The reaction was stirred at –60 °C overnight, then MeOH (2 mL) was added slowly. The reaction was warmed to room temperature and

concentrated to 2-5 mL, then was poured slowly into MeOH (100 mL). The precipitate was collected and dissolved in toluene. The solvent was concentrated and the residue was dried vacuum overnight to provide desired product (418 mg, 82.3%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.78-7.14 (m, 4H), 5.32 (m, 2H), 3.99 (m, 2H), 3.32-3.62 (m, 8H), 1.24-2.38 (m, 25H), 0.87 (t, *J* = 6.7 Hz, 3H); IR (in toluene) 3449, 3308, 2929, 2244, 1609, 1512, 1248, 1185, 830 cm^{-1}.



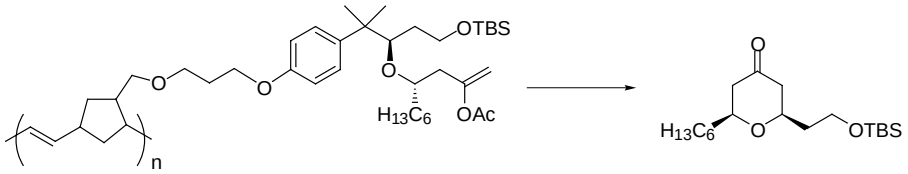
Polymer-supported acetic acid 3-[2-(4-propoxyphenyl)-1-(2-hydroxyethyl)-2-methylpropoxy]-1-methylenenonyl ester

To the polymer-supported alcohol (389 mg, 0.65 mmol) in toluene (9 mL) was added tri-2-furylphosphine (8 mg, 0.03 mmol), dichloro(*p*-cymene)ruthenium(II) dimmer (11 mg, 0.02 mmol) and Na_2CO_3 (8 mg, 0.08 mmol). After stirring at room temperature for a few minutes, AcOH (775 mg, 12.9 mmol) was added and the reaction was stirred at 60-70 °C for 24 h. Additional portions of tri-2-furylphosphine (4 mg, 0.02 mmol) and dichloro(*p*-cymene)ruthenium(II) dimer (5 mg, 0.01 mmol) was added. The reaction mixture continued to stir at 60-70 °C for another 24 h, then was concentrated. The resulting residue was poured into MeOH (100 mL). The precipitate was collected and dissolved in CH_2Cl_2. The solvent was concentrated and the residue was dried under vacuum to provide desired product (383 mg, 88%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.77-7.22 (m, 4H), 5.32 (m, 1H), 5.19 (m, 1H), 4.77 (br, 1H), 4.72 (br, 1H), 3.97 (br, 2H), 3.39-3.50 (m, 8H), 1.24-2.33 (m, 30H), 0.86 (t, *J* = 6.7 Hz, 3H); IR (in CH_2Cl_2) 3465, 2930, 2860, 1754, 1806, 1512, 1246, 1074, 829 cm^{-1}.



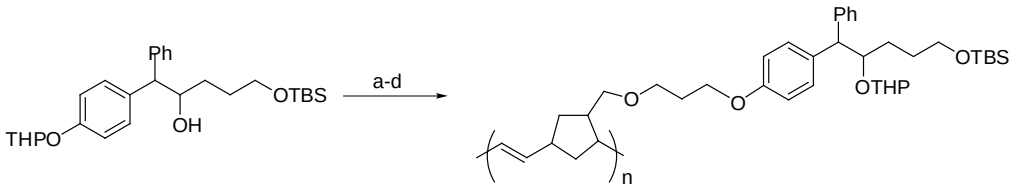
Polymer-supported Acetic Acid 3-[2-(4-propoxyphenyl)-1-[2-(tert-butyldimethylsilanyloxy)ethyl]-2-methylpropoxy]-1-methylenenonyl ester

To the polymer-supported enol acetate (383 mg, 0.57 mmol) in CH_2Cl_2 (25 mL) at 0 °C was added imidazole (194 mg, 2.84 mmol). The reaction was stirred for 10 minutes, then TBSCl (429 mg, 2.84 mmol) was added. The reaction was slowly warmed to room temperature and stirred overnight. The solution was concentrated to 2-3 mL and then poured slowly into MeOH (100 mL). The precipitate was collected and dissolved in toluene. The solution was concentrated and the residue was dried under vacuum to provide desired product (355 mg, 80.2%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.80-7.33 (m, 4H), 5.34 (m, 2H), 4.78 (br, 1H), 4.75 (br, 1H), 4.01 (br, 2H), 3.38-3.53 (m, 8H), 1.27-2.36 (m, 27 H), 0.87 (m, 9H), 0.07 (s, 3H), -0.04 (s, 3H).



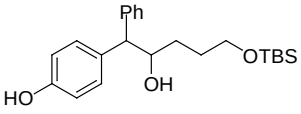
3-[2-(*tert*-Butyldimethylsilanyloxy)ethyl]-5-hexylcyclohexanone (15)

To the polymer-supported enol acetate (142 mg, 0.18 mmol) in dichloroethane (11 mL) were added 4 Å molecular sieves (285 mg), and $NaHCO_3$ (285 mg). The reaction mixture was stirred for 15 min, then ceric ammomium nitrate (602 mg, 1.1 mmol) in CH_3CN (5 mL) was added dropwise. The reaction was stirred at rt for 1 h, then was then filtered through a plug of silica gel. The filtrate was concentrated and the residue was purified by flash column chromatography (10% EtOAc in hexanes) to provide the desired product[2] (26 mg, 41.1 % yield): ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.79 (m, 3H), 3.70 (m, 1H), 2.34 (d, *J* = 12.5 Hz, 2H), 2.23 (m, 2H), 1.24-1.79 (m, 15H), 0.89 (s, 9H), 0.05 (s, 6H).



Reagents and conditions
a) $MgBr_2$, Et_2O. b) **4**, K_2CO_3, DMF. c) DHP, PPTs, CH_2Cl_2. d) Grubbs 2nd generation catalyst, CH_2Cl_2, then ethyl vinyl ether.

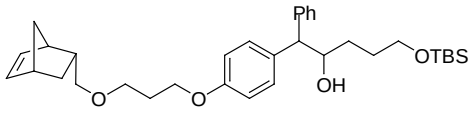
Scheme 6.



4-[5-(*tert*-Butyldimethylsilanyloxy)-2-hydroxy-1-phenylpentyl]phenol

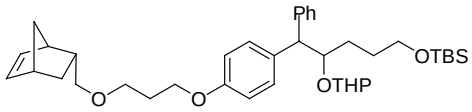
To $MgBr_2$ (prepared from Mg turnings (180 mg, 7.5 mmol) and 1,2-dibromoethane (1.41 g, 7.5 mmol)) in diethyl ether (25 mL) was added 5-(*tert*-butyldimethylsilanyloxy)-1-phenyl-1-[4-(tetrahydropyran-2-yloxy)phenyl]-pentan-2-ol (1.45 g, 3.1 mmol). The reaction was stirred at room temperature overnight, then was quenched by adding water (10 mL) and extracted with EtOAc (30 mL). The organic phase was washed with water and brine, then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (20 % EtOAc in hexanes) to provide desired product (556 mg, 46.7%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 7.3, 3H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.85 (s, 1H), 6.76 (d, *J* = 8.4 Hz, 2H), 4.46 (t, *J* = 7.8 Hz, 1H), 3.94 (d, *J* = 8.5 Hz, 1H), 3.74 (t, *J* = 5.7 Hz, 2H), 2.88 (br, 1H), 1.50-1.80 (m, 4H), 0.99 (s, 9H), 0.15 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.5, 142.0, 134.0, 129.1,

128.6, 128.5, 126.5, 115.5, 73.8, 63.4, 57.6, 31.8, 28.9, 25.9, 18.2, -5.4; IR (neat) 3355, 3061, 3025, 2028, 2856, 1614, 1513, 1451, 1255, 1175, 1029, 834, 698 cm^{-1}; HRMS (EI) Calcd for $C_{19}H_{25}O_3Si$ (M^+-C_4H_9) 329.1566, found 329.1579.



1-{4-[3-(Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-5-(*tert*-butyldimethylsilanyloxy)-1-phenylpentan-2-ol

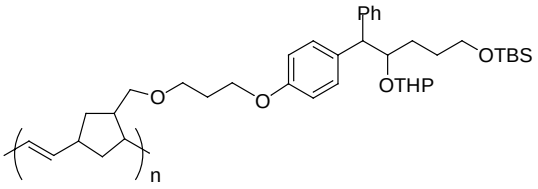
To the phenol (520 mg, 1.34 mmol) in DMF (5 mL) were added anhydrous K_2CO_3 (224 mg, 1.62 mmol) and **4** (430 mg, 1.48 mmol). The reaction mixture was stirred at room temperature for 36 h, then participated between water (20 mL) and EtOAc (100 mL). The aqueous layer was extracted with EtOAc (100 mL), then the organic layers were combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10 % EtOAc in hexanes) to provide desired product (493 mg, 66.4%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.3 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 3H), 7.30 (d, *J* = 8.6 Hz, 2H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.21 (dd, *J* = 5.5, 3.0 Hz, 1H), 5.98 (dd, *J* = 5.5, 2.7 Hz, 1H), 4.43 (t, *J* = 7.7 Hz, 1H), 4.12 (t, *J* = 6.2, 2H), 3.95 (d, *J* = 6.2 Hz, 1H), 3.60-3.74 (m, 4H), 3.26 (dd, *J* = 9.1, 6.7 Hz, 1H), 3.13 (m, 1H), 3.00 (bs, 1H), 2.88 (bs, 1H), 2.32 (m, 1H), 2.26 (br, 1H), 2.12 (m, 2H), 1.74-1.79 (m, 4H), 1.51 (m, 2H), 1.33 (m, 1H), 0.98 (s, 9H), 0.58 (m, 1H), 0.14 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.7, 142.3, 137.2, 134.9, 132.6, 129.3, 128.9, 128.8, 126.7, 114.7, 74.8, 73.7, 67.4, 65.0, 63.4, 58.0, 49.5, 44.1, 42.3, 38.9, 32.1, 29.8, 29.3, 29.25, 26.1, 18.4, -5.2; IR (neat) 3450, 3059, 3028, 2954, 2859, 1740, 1611, 1510, 1471, 1250, 1177, 835, 700 cm^{-1}; HRMS (EI) Calcd for $C_{34}H_{51}O_4Si$ (M^++H) 551.3537, found 551.3578.



[5-{4-[3-(Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-5-phenyl-4-(tetrahydropyan-2-yloxy)pentyloxy]*tert*-butyldimethylsilane

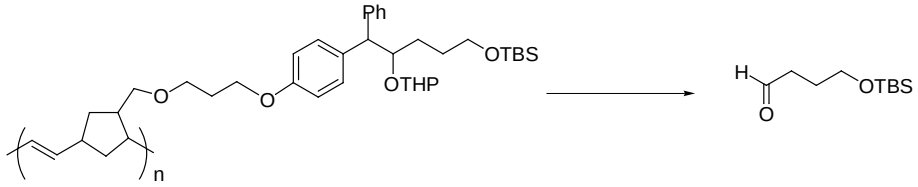
To the norbonenyl-tagged alcohol (241 mg, 0.44 mmol) in CH_2Cl_2 (2 mL) were added 3,4-dihydro-2H-pyran (40 mg, 0.5 mmol) and PPTS (5 mg, 0.02 mmol). The reaction mixture was stirred at room temperature for 7 h, then was dissolved in EtOAc (50 mL) and washed with water and brine. The organic layers was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10% EtOAc in hexanes) to provide desired product (270 mg 97%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.54 (m, 2H), 7.26-7.38 (m, 5H), 6.92 (d, *J* = 8.5 Hz, 2H), 6.20 (dd, *J* = 5.6, 3.0 Hz, 1H), 6.00 (dd, *J* = 5.5, 2.8 Hz, 1H), 4.94 (bs, 1H), 4.52 (m, 1H), 4.35 (m, 1H), 3.96-4.19 (m, 4H), 3.44-3.67 (m, 4H), 3.25 (dd, *J* = 9.1, 6.1 Hz, 2H), 3.13 (m, 1H), 2.99 (bs, 1H), 2.87 (bs, 1H), 2.11 (m, 1H), 2.10 (tt, *J* = 6.1 Hz, 1H), 1.89

(ddd, *J* = 11.9, 8.8, 3.8 Hz, 1H), 1.31-1.80 (m, 12 H), 0.96 (s, 9H), 0.58 (m, 1H), 0.10 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.4, 143.0, 142.5, 136.9, 134.5, 134.5, 132.3, 129.6, 129.4, 129.1, 127.9, 127.8, 126.0, 125.8, 114.3, 100.3, 94.0, 81.6, 76.4, 74.6, 67.2, 64.71, 63.2, 63.1, 62.7, 60.3, 55.5, 54.3, 49.3, 43.8, 42.0, 38.6, 30.7, 30.5, 30.4, 29.6, 29.0, 28.2, 26.9, 26.1, 25.9, 25.8, 25.4, 19.8, 18.2, 18.19, 18.15, -5.37, -5.41; IR (neat) 3060, 3028, 2951, 2858, 1611, 1510, 1471, 1250, 1112, 1022, 835, 699 cm^{-1}; HRMS (EI) Calcd for $C_{34}H_{48}O_3Si$ (M^+-$C_5H_{10}O_2$) 532.3373, found 532.3349.



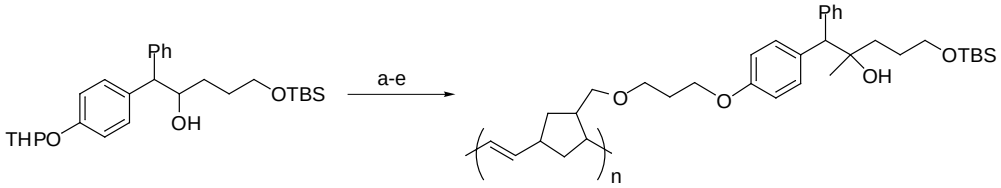
Polymer 16

To the norbornenyl-tagged ether (251 mg, 0.41 mmol) in degassed CH_2Cl_2 (1.0 mL) was added Grubbs' second-generation catalyst (6 mg, 0.007 mmol). The reaction mixture was stirred at reflux for 30 min, then ethyl vinyl ether (1 mL) was added and the reaction was allowed to stir for 15 min. The reaction mixture was then poured into MeOH (100 mL). The precipitate was collected and washed with methanol, then was dissolved in toluene. The solvent was removed and the residue was dried under high vacuum overnight (232 mg, 80.5%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.09-7.39 (m, 7H), 6.75 (d, *J* = 7.0Hz, 2H), 5.25 (m, 2H), 4.79 (m, 1H), 4.40 (m, 1H), 4.25 (m, 1H), 4.02 (d, *J* = 8.2 Hz, 1H), 3.34-3.96 (m, 9H), 1.23-2.65 (complex m, 19H), 0.81 (s, 9H), 0.04 (s, 6H).



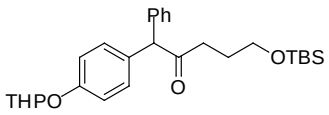
4-(*tert*-butyldimethylsilanyloxy)butyraldehyde

To **15** (937 mg, 0.13 mmol) in dichloroethane (6 mL) in a borosilicate flask were added *N*-methylquinolinium hexafluorophosphate (4 mg, 0.01 mmol), sodium acetate (187 mg), anhydrous sodium thiosulfate (187 mg) and toluene (1 mL). The mixture was stirred at room temperature with gentle aeration and irradiation with a medium pressure mercury lamp for 5 h. The reaction mixture was filtered through a small plug of silica gel and the filtrate was concentrated to provide the desired product[3] (21 mg, 78 %).



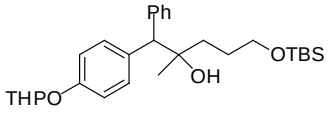
Reagents and Conditions
a) Dess-Martin periodinane, CH_2Cl_2. b) MeMgBr, THF. c) $MgBr_2$, Et_2O. d) **4**, K_2CO_3, DMF. e) Grubbs 2nd generation catalyst, CH_2Cl_2, then ethyl vinyl ether.

Scheme 7.



5-(*tert*-Butyldimethylsilanyloxy)-1-phenyl-1-[4-(tetrahydropyran-2-yloxy)-phenyl]pentane-2-one

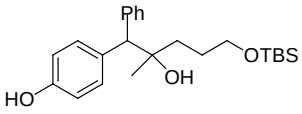
To the homobenzylic alcohol (581 mg, 1.23 mmol) in CH_2Cl_2 (10 mL) were added Na_2CO_3 (1.06 g, 10 mmol) and the Dess-Martin periodinane (789 mg, 1.86 mmol). The reaction mixture was stirred at room temperature overnight, then water (20 mL) was added. The reaction mixture was extracted with EtOAc (2×25 mL). The organic layers were combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10% EtOAc in hexanes) to provide desired product (477 mg 82%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24-7.32 (m, 5H), 7.26 (d, *J* = 8.3 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H), 5.94 (t, *J* = 3.1 Hz, 1H), 5.19 (s, 1H), 3.99 (td, *J* = 10.2, 3.1 Hz, 1H), 3.68 (t, *J* = 6.1, 3H), 2.74 (t, *J* = 7.2 Hz, 2H), 1.72-1.95 (m, 8H), 0.96 (s, 9H), 0.11 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.7, 156.2, 138.8, 131.4, 129.8, 128.8, 128.50, 127.0, 116.5, 116.5, 96.2, 96.2, 63.4, 61.9, 38.9, 30.3, 27.0, 25.8, 25.1, 18.7, 18.2, -5.1, -5.4; IR (neat) 3061, 3028, 2951, 2856, 1718, 1609, 1240, 1109, 1037, 835, 700 cm^{-1}; HRMS (EI) Calcd for $C_{28}H_{40}O_4Si$ 468.2696, found 468.2704.



5-(*tert*-Butyldimethylsilanyloxy)-2-methyl-1-phenyl-phenyl-1-[4-(tetrahydropyran-2-yloxy)phenyl]pentan-2-ol

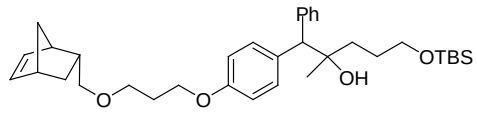
To the ketone (2.46 g, 5.245 mmol) in THF (17 mL) at –78 °C was added methylmagnesium bromide (5.7 mL, 3 M in Et_2O, 17 mmol). The reaction mixture was then slowly warmed to room temperature and stirred for 6 h. The reaction was quenched by adding saturated aqueous NH_4Cl (50 mL) and extracted with EtOAc (2×100 mL). The organic layers were combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (15% EtOAc in hexanes) to provide desired product (2.37 g, 93.5%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.5 Hz, 2H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.21-7.39 (m, 3H), 7.08 (dd, *J* = 8.6, 1.9 Hz, 2H), 5.47

(d, *J* = 2.5 Hz, 1H), 4.00 (t, *J* = 8.8 Hz, 1H), 3.91 (s, 1H), 3.63 (m, 3H), 1.66-2.14 (m, 11H), 1.28 (d, *J* = 2.9 Hz, 3H), 0.98 (d, *J* = 3.4 Hz, 9H), 0.12 (dd, *J* = 4.7, 1.7, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.6, 155.5, 142.4, 142.3, 142.0, 135.0, 134.7, 130.6, 130.6, 129.7, 128.1, 126.1, 116.0, 96.2, 74.6, 74.5, 63.4, 61.9, 60.8, 37.7, 30.3, 27.3, 26.2, 26.17, 26.1, 25.9, 25.1, 18.8, 18.2, -5.4; IR (neat) 3480, 3059, 3025, 2950, 2856, 1740, 1609, 1508, 1238, 1107, 1037, 835, 702 cm^{-1}; HRMS (EI) Calcd for $C_{29}H_{43}O_4Si$ (M^+-H) 483.2930, found 483.2929.



4-[5-(*tert*-Butyldimethylsilanyloxy)-2-hydroxy-2-methyl-1-phenylpentyl]phenol

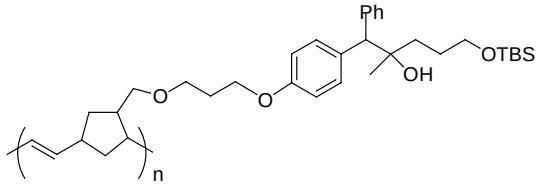
To $MgBr_2$ (prepared from Mg turnings (0.144 g, 6 mmol) and 1,2-dibromoethane (1.128 g, 6 mmol) in Et_2O (20 mL)) was added the tertiary alcohol (1.19 g, 2.46 mmol). The reaction mixture was stirred at room temperature for 24 h and quenched by adding water (10 mL). The reaction was extracted with EtOAc (2×100 mL), then the organic layers were then combined and washed with water and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (20% EtOAc in hexanes) to provide desired product (826 mg, 84%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.3 Hz, 2H), 7.38 (d, *J* = 7.2 Hz, 2H), 7.22-7.26 (m, 3H), 6.70-6.73 (m, 2H), 4.65 (br, 1H), 3.76 (s, 1H), 3.50 (t, *J* = 5.6 Hz, 2H), 2.03 (br, 1H), 1.57 (m, 4H), 1.14 (d, *J* = 3.0 Hz, 3H), 0.83 (d, *J* = 2.5 Hz, 9H), -0.029 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.4, 142.1, 141.9, 133.4, 133.0, 130.8, 130.8, 129.7, 129.6, 128.2, 126.2, 115.2, 75.1, 63.6, 63.6, 60.7, 60.6, 37.6, 37.6, 27.2, 26.1, 25.9, 18.3, -5.37, -5.40, -5.46, IR (neat) 3376, 3060, 3025, 2954, 2857, 1711, 1613. 1512, 1470, 1256, 1097, 1045, 835, 702 cm^{-1}, HRMS (EI) Calcd for $C_{24}H_{34}O_2Si$ (M^+-H_2O) 382.2328, found 382.2330.



1-{4-[3-(Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)propoxy]phenyl}-5-(*tert*-butyldimethylsilanyloxy)-2-methyl-1-phenylpentan-2-ol

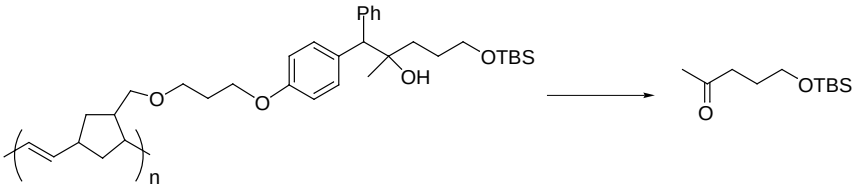
To the phenol (339 mg, 0.85 mmol) in DMF (5 mL) were added anhydrous potassium carbonate (140 mg, 1.0 mmol) and the norbornenyl-tagged iodide (272 mg, 0.93 mmol). The reaction was stirred at room temperature for 36 hours and then partitioned between water (15 mL) and EtOAc (50 mL). The aqueous layer was extracted with EtOAc (2×100 mL). The organic layers were combined and washed with water and brine. The organic layers were then dried (Na_2SO_4), filtered and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (10% EtOAc in hexanes) to provide desired product (359 mg, 74.9%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.5 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.25-7.39 (m, 3H), 6.93 (dd, *J* = 8.6, 2.4 Hz, 2H), 6.07 (dd, *J* = 5.6, 3.0 Hz, 1H), 6.00 (m, 1H), 4.12 (t, *J* = 6.1, 2H), 3.90 (s, 1H), 3.63 (m, 4H),

3.25 (dd, *J* = 9.2, 6.6 Hz, 1H), 3.12 (m, 1H), 2.99 (br, 1H), 2.87 (br, 1H), 2.45 (m, 1H), 2.12 (m, 3H), 1.90 (ddd, *J* = 11.9, 8.7, 3.8 Hz, 1H), 1.31-1.72 (m, 6H), 1.27 (s, 3H), 0.96 (s, 9H), 0.58 (m, 1H), 0.10 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.5, 142.4, 142.1, 137.0, 134.1, 133.7, 132.4, 130.7, 130.6, 129.7, 129.6, 128.1, 126.1, 114.1, 74.6, 67.3, 64.7, 63.5, 60.7, 49.3, 43.9, 42.1, 38.7, 37.8, 29.7, 29.0, 27.4, 26.2, 25.9, 18.3, -5.4; IR (neat) 3518, 3059, 3025, 2954, 2859, 1740, 1609, 1509, 1471, 1248, 1099, 835, 702 cm^{-1}; HRMS (EI) calcd for $C_{35}H_{50}O_3Si$ (M^+-H_2O) 546.3529, found 546.3539.



Polymer-supported 1-(4-propoxyphenyl)-5-(*tert*-butyl-dimethylsilanyloxy)-2-methyl-1-phenylpentan-2-ol

To the norbornenyl-tagged alcohol (204 mg, 0.36 mmol) in degassed CH_2Cl_2 (1.0 mL) was added Grubbs' second-generation catalyst (5 mg, 0.006 mmol). The reaction mixture was stirred at reflux for 30 min, then ethyl vinyl ether (0.5 mL) was added and the reaction was allowed to stir for 15 min. The reaction mixture was poured into 100 mL of MeOH. The precipitate was collected and washed with methanol, then dissolved in toluene. The solvent was removed and the residue was dried under high vacuum overnight (190 mg, 80%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.05-7.59 (m, 7H), 6.76 (m, 2H), 5.16-5.30 (m, 2H), 3.93 (m, 2H), 3.74 (s, 1H), 3.42-3.49 (m, 6H), 1.12-1.49 (m, 17 H), 0.82 (m, 9H), 0.04 (m, 6H).

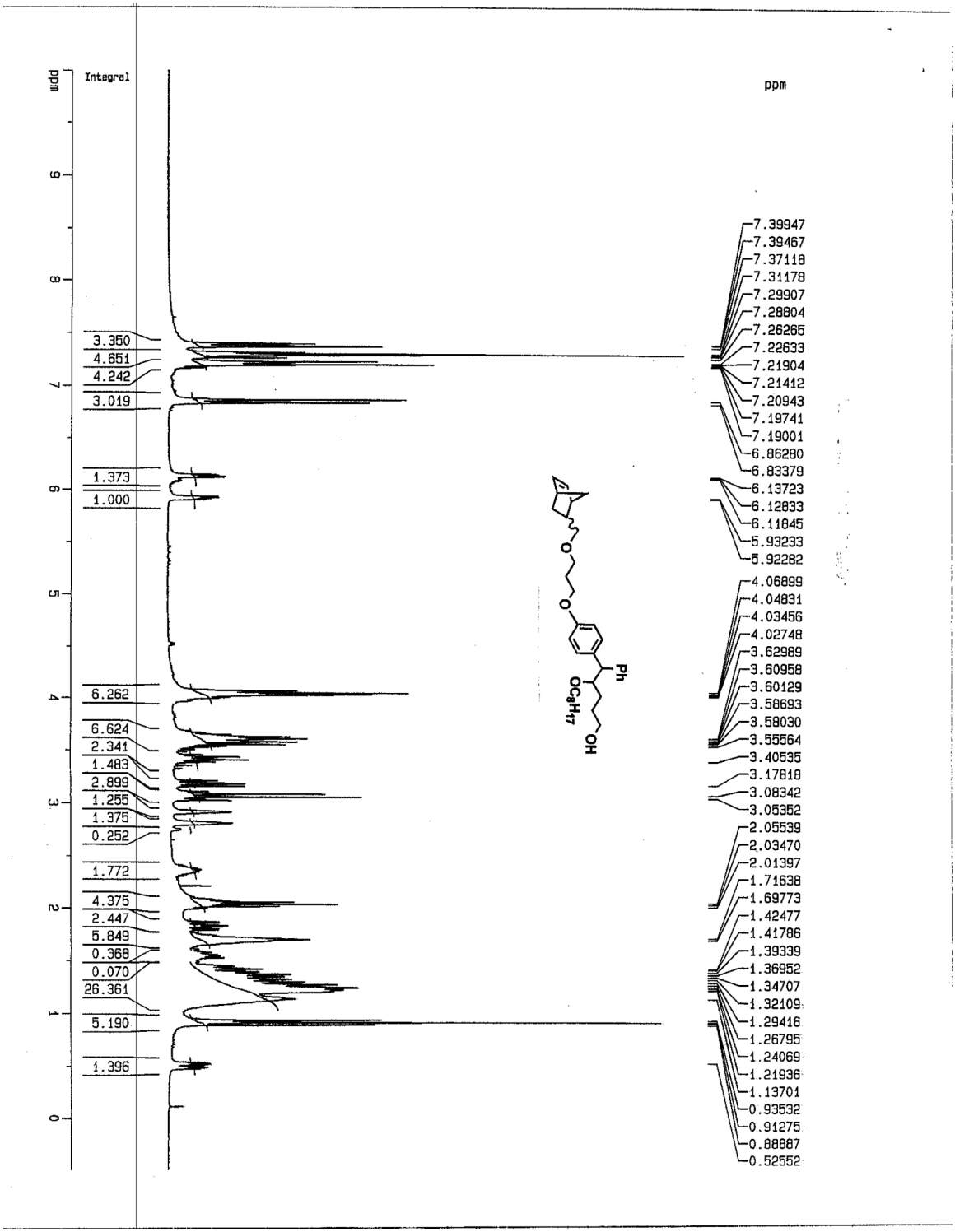


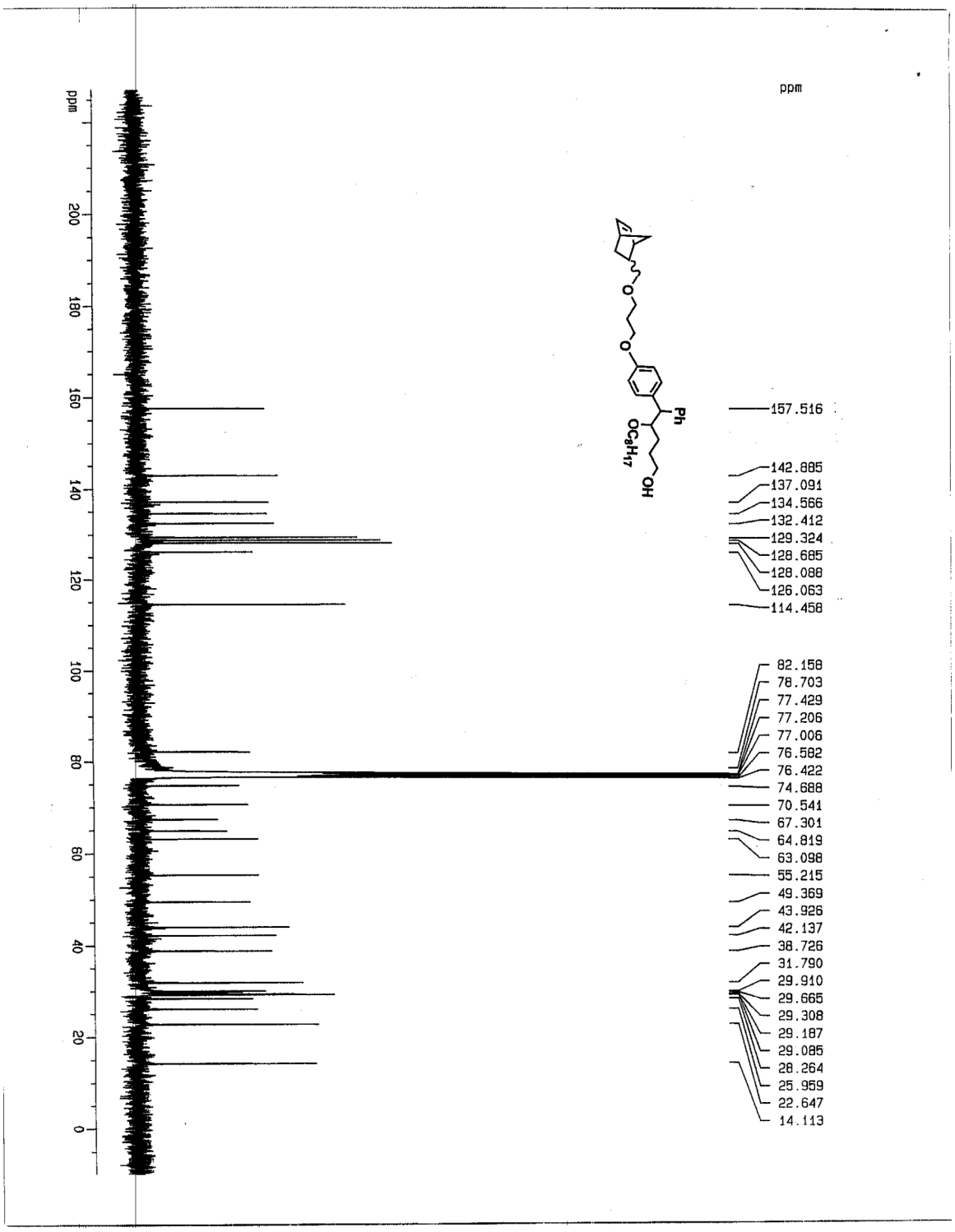
5-(*tert*-Butyldimethylsilanyloxy)pentan-2-one

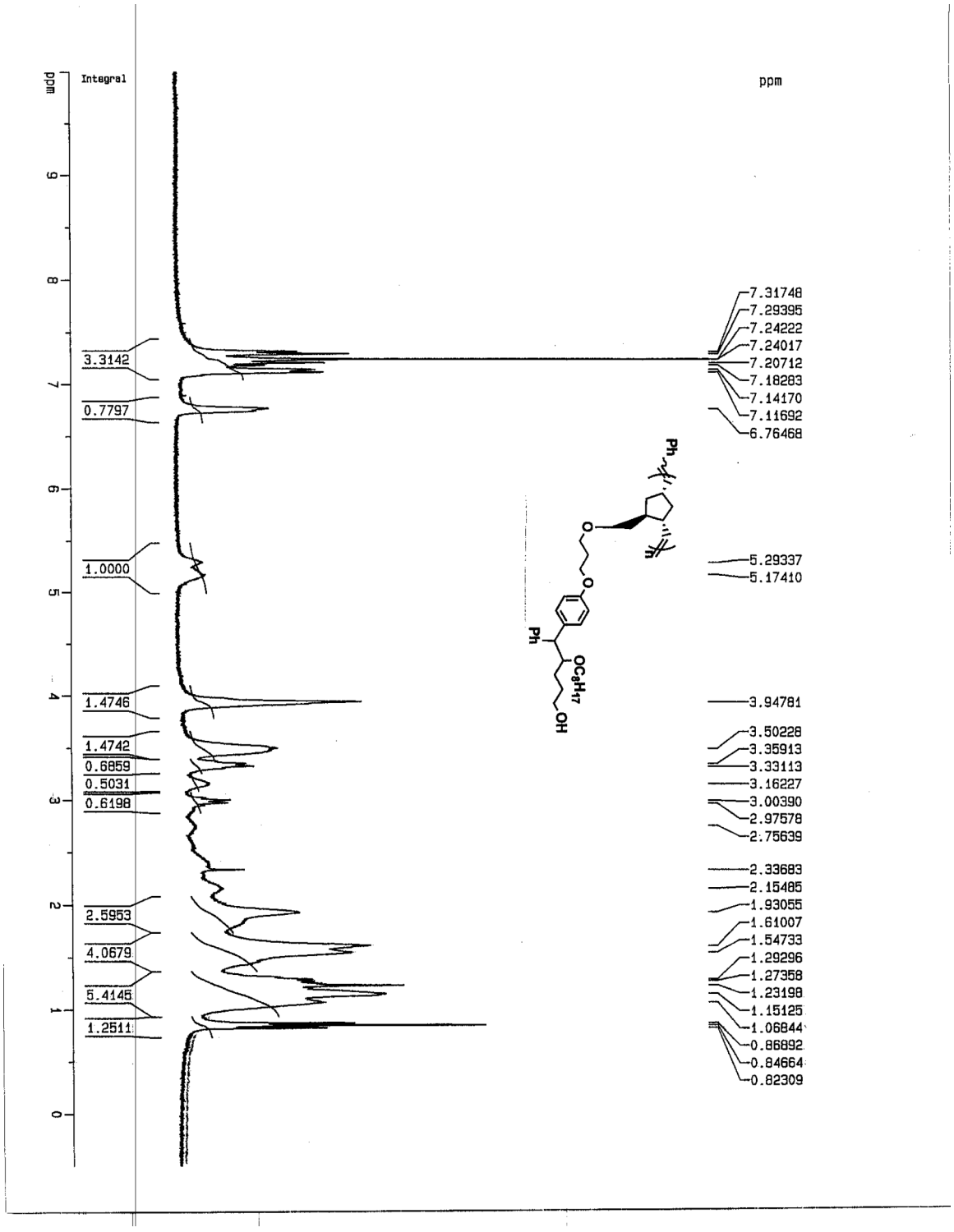
To the polymer-supported alcohol (140 mg, 0.21 mmol) in dichloroethane (6 mL) in a borosilicate flask were added *N*-methylquinolinium hexafluorophosphate (6 mg, 0.02 mmol), sodium acetate (281 mg), anhydrous sodium thiosulfate (281 mg) and toluene (1 mL). The mixture was stirred at room temperature with gentle aeration and irradiation with a medium pressure mercury lamp for 5 h. The reaction mixture was filtered through a small plug of silica gel and the filtrate was concentrated to provide the desired product.[4] (41 mg, 89%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.69 (t, *J* = 6.1 Hz, 2H), 2.59 (t, *J* = 7.3 Hz, 2H), 2.23 (s, 3H), 1.86 (tt, *J*=7.1 Hz, 2H), 0.96 (s, 9H), 0.11 (s, 6H).

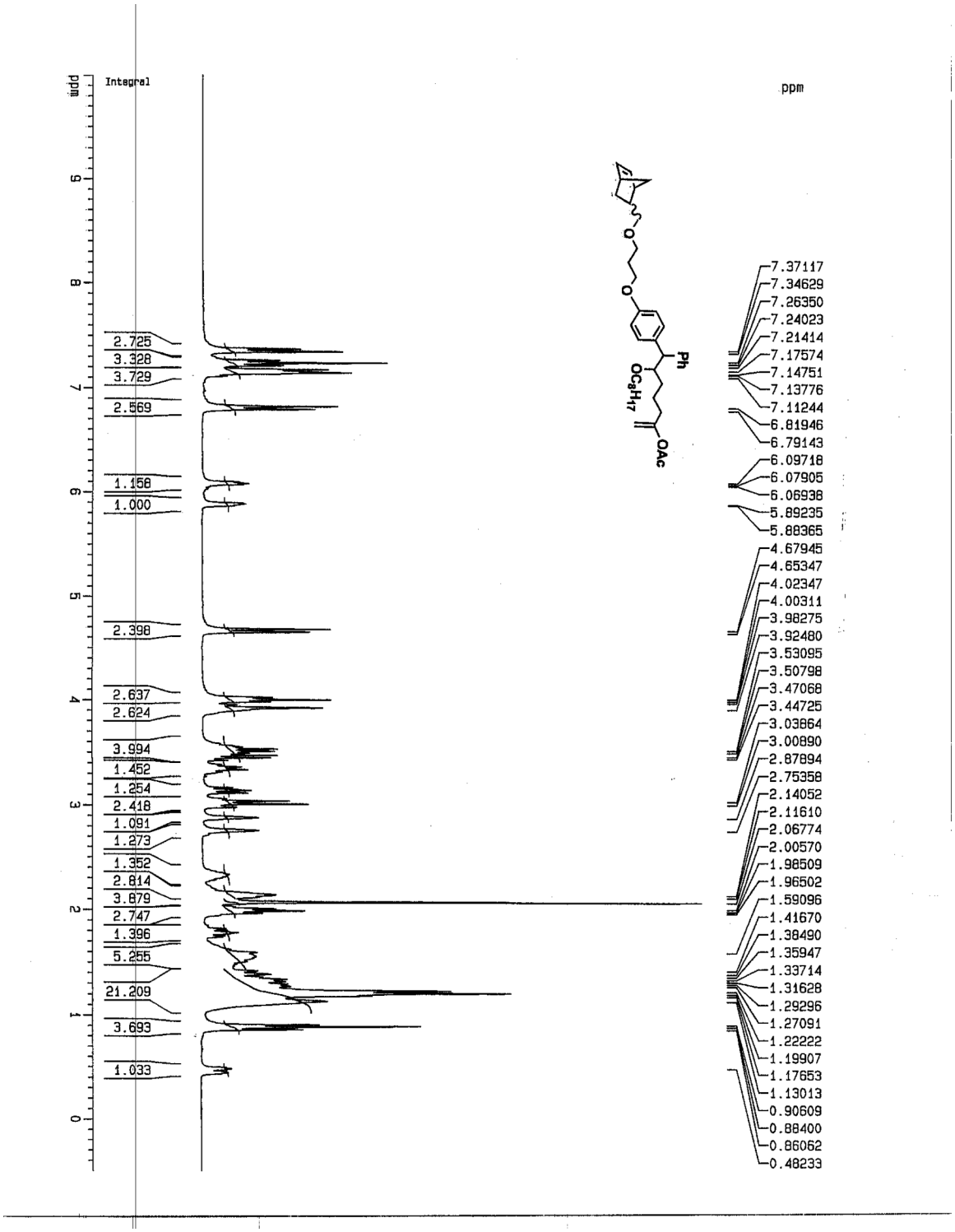
References

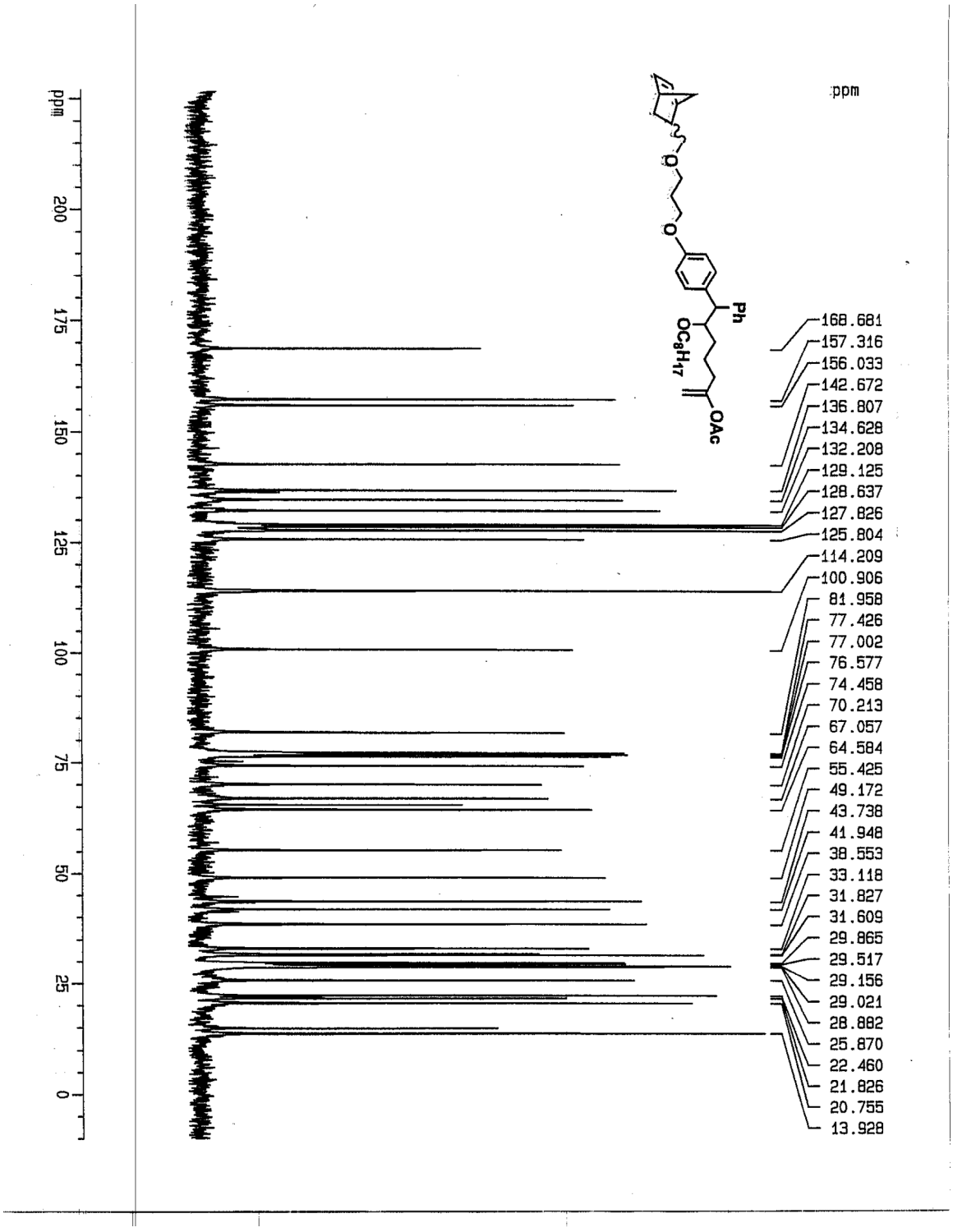
1) Seiders, J. R., II; Wang, L.; Floreancig, P. E. *J. Am. Chem. Soc.* **2003**, *125*, 2406.
2) Wang, L.; Seiders, J. R., II; Floreancig, P. E. *J. Am. Chem. Soc.* **2004**, *126*, 12596.
3) Ikeda, Y.; Ukai, J.; Ikeda, N.; Yamamoto, H. *Tetrahedron* **1987**, *43*, 731.
4) Chiarello, J.; Joullie, M. M. *Tetrahedron* **1988**, *44*, 41.

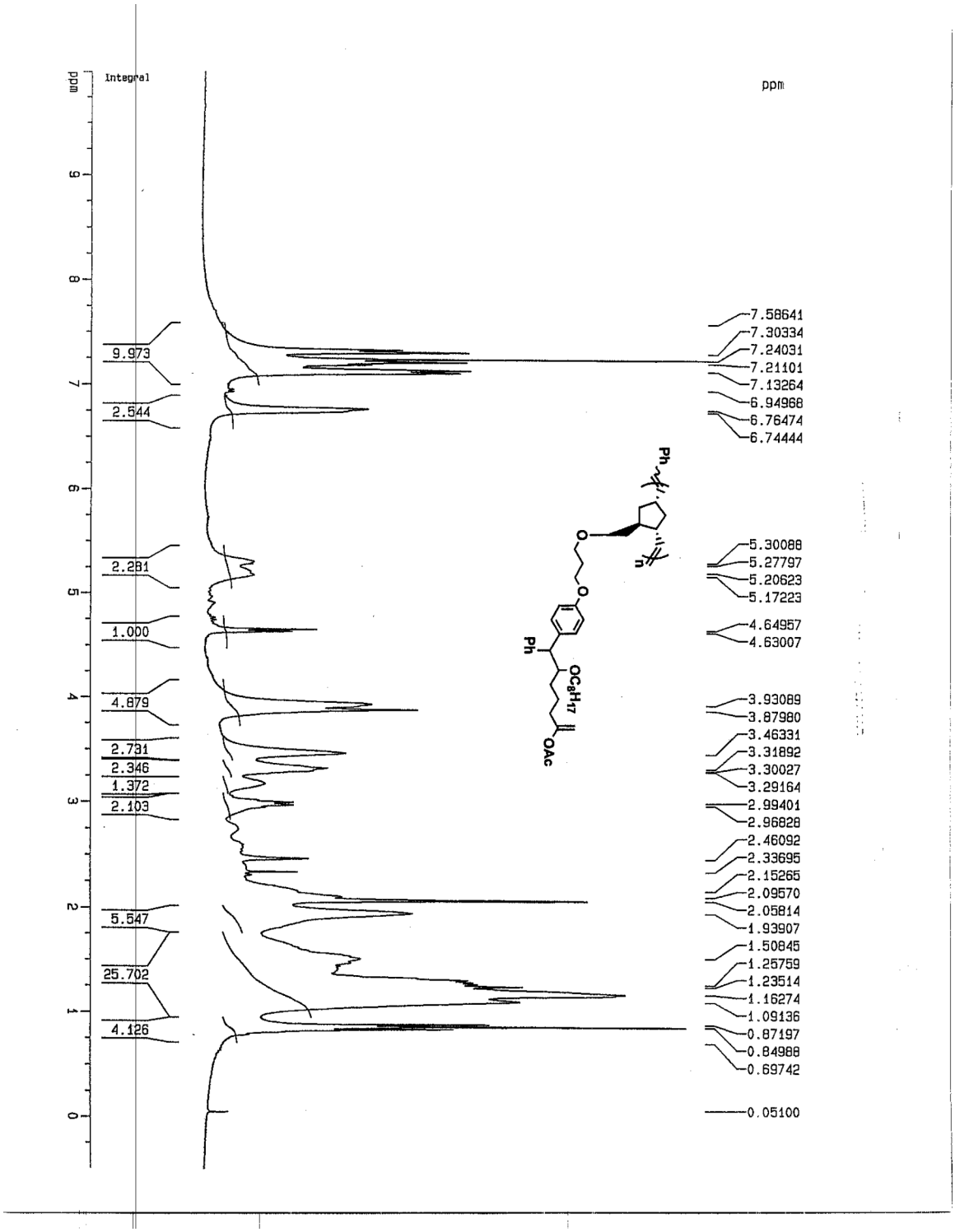

ppm
Integral
3.350
4.651
4.242
3.019
1.373
1.000
6.262
6.624
2.341
1.483
2.899
1.255
1.375
0.252
1.772
4.375
2.447
5.849
0.368
0.070
26.361
5.190
1.396
Ph
OC8H17
OH
ppm
7.39947
7.39467
7.37118
7.31178
7.29907
7.28804
7.26265
7.22633
7.21904
7.21412
7.20943
7.19741
7.19001
6.86280
6.83379
6.13723
6.12833
6.11845
5.93233
5.92282
4.06899
4.04831
4.03456
4.02748
3.62989
3.60958
3.60129
3.58693
3.58030
3.55564
3.40535
3.17818
3.08342
3.05352
2.05539
2.03470
2.01397
1.71638
1.69773
1.42477
1.41786
1.39339
1.36952
1.34707
1.32109
1.29416
1.26795
1.24069
1.21936
1.13701
0.93532
0.91275
0.88887
0.52552

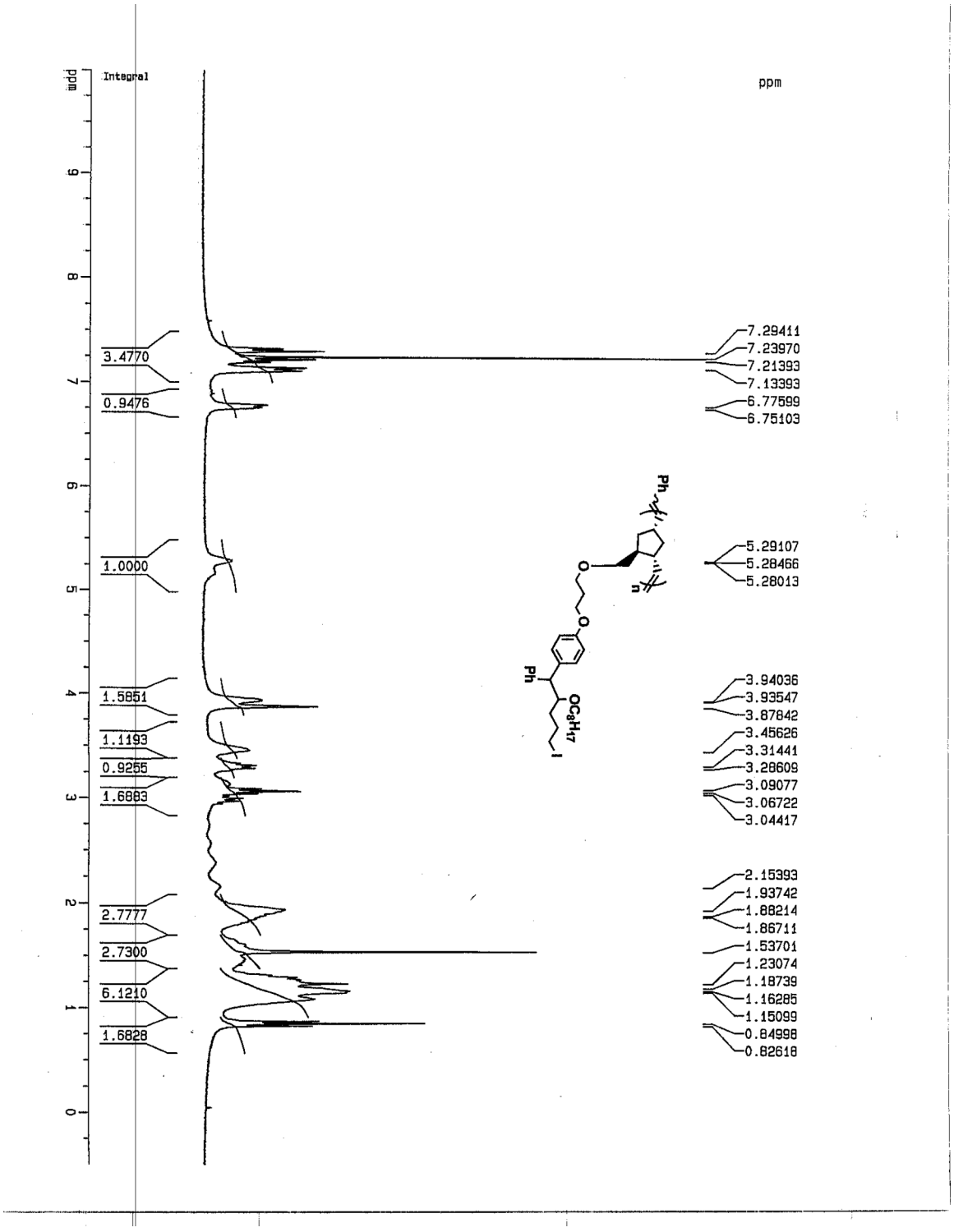

ppm
ppm
200
180
160
140
120
100
80
60
40
20
0
157.516
142.885
137.091
134.566
132.412
129.324
128.685
128.088
126.063
114.458
82.158
78.703
77.429
77.206
77.006
76.582
76.422
74.688
70.541
67.301
64.819
63.098
55.215
49.369
43.926
42.137
38.726
31.790
29.910
29.665
29.308
29.187
29.085
28.264
25.959
22.647
14.113
Ph
OC8H17
OH

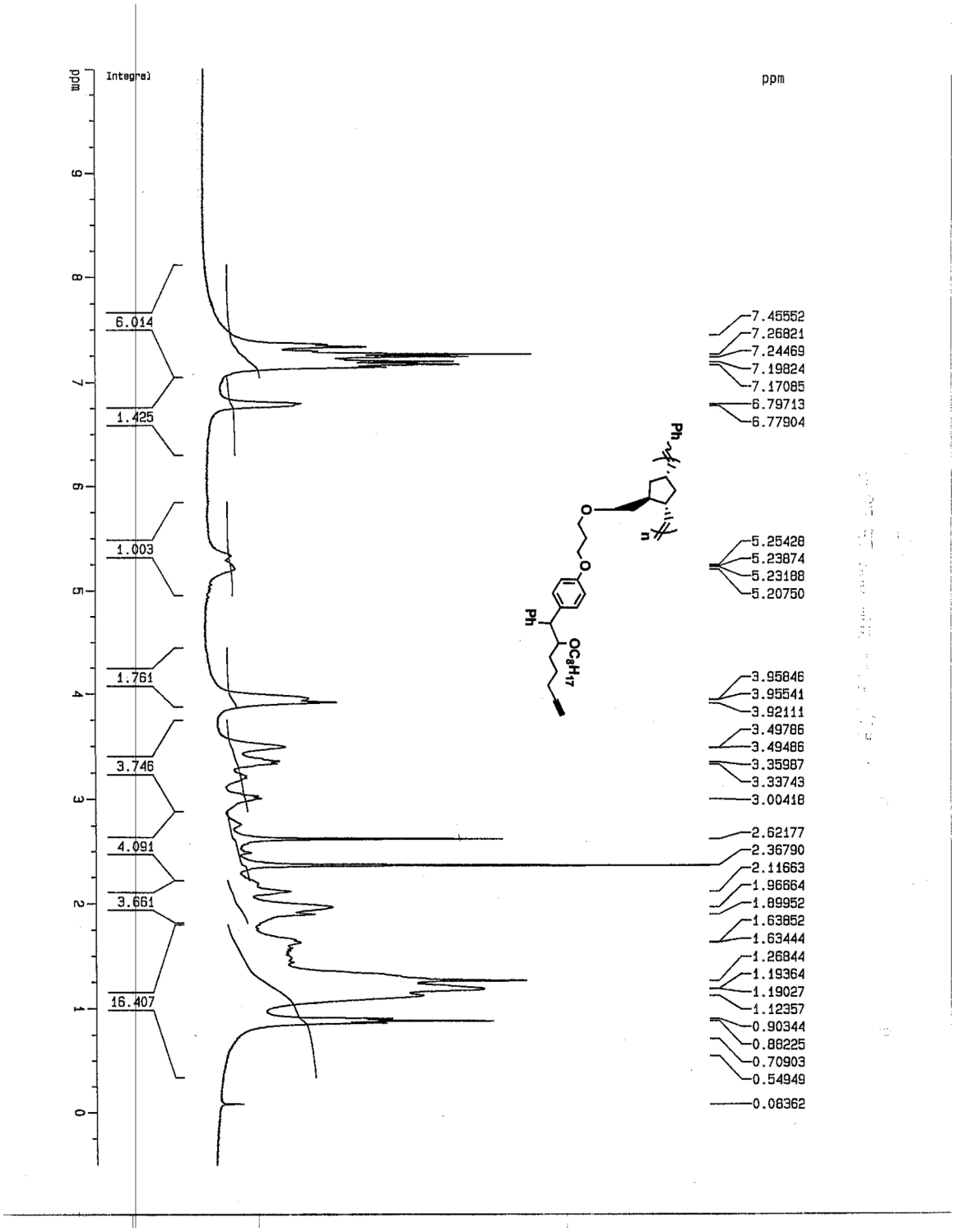

ppm
Integral
ppm
3.3142
0.7797
1.0000
1.4746
1.4742
0.6859
0.5031
0.6198
2.5953
4.0679
5.4145
1.2511
7.31748
7.29395
7.24222
7.24017
7.20712
7.18283
7.14170
7.11692
6.76468
5.29337
5.17410
3.94781
3.50228
3.35913
3.33113
3.16227
3.00390
2.97578
2.75639
2.33683
2.15485
1.93055
1.61007
1.54733
1.29296
1.27358
1.23198
1.15125
1.06844
0.86892
0.84664
0.82309
Ph
O
O
Ph
OC8H17
OH

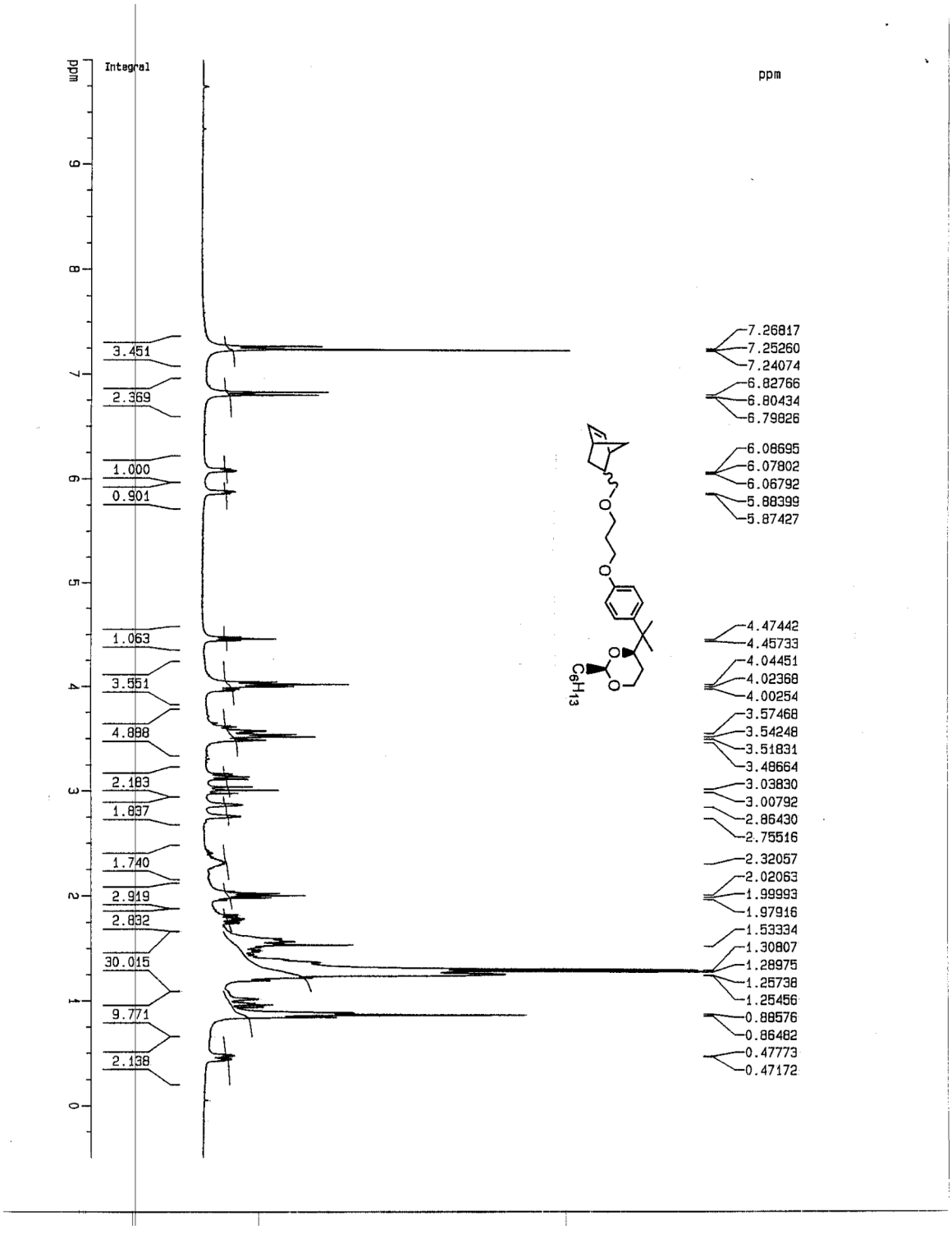

Integral
ppm
2.725
3.328
3.729
2.569
1.156
1.000
2.398
2.637
2.624
3.994
1.452
1.254
2.418
1.091
1.273
1.352
2.814
3.879
2.747
1.396
5.255
21.209
3.693
1.033
Ph
OC8H17
OAc
7.37117
7.34629
7.26350
7.24023
7.21414
7.17574
7.14751
7.13776
7.11244
6.81946
6.79143
6.09718
6.07905
6.06938
5.89235
5.88365
4.67945
4.65347
4.02347
4.00311
3.98275
3.92480
3.53095
3.50798
3.47068
3.44725
3.03864
3.00890
2.87894
2.75358
2.14052
2.11610
2.06774
2.00570
1.98509
1.96502
1.59096
1.41670
1.38490
1.35947
1.33714
1.31628
1.29296
1.27091
1.22222
1.19907
1.17653
1.13013
0.90609
0.88400
0.86062
0.48233

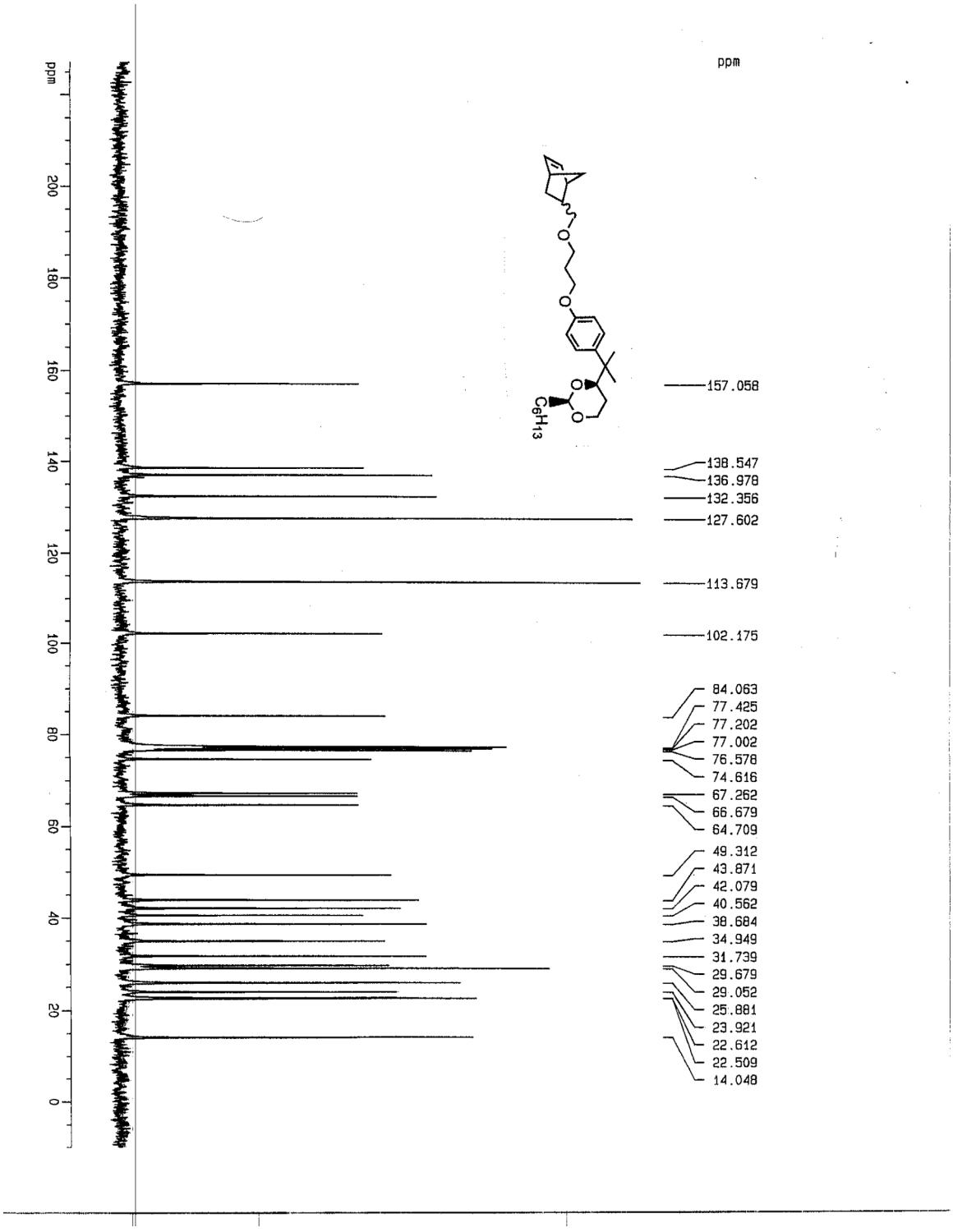

ppm
200
175
150
125
100
75
50
25
0
Ph
OC8H17
OAc
168.681
157.316
156.033
142.672
136.807
134.628
132.208
129.125
128.637
127.826
125.804
114.209
100.906
81.958
77.426
77.002
76.577
74.458
70.213
67.057
64.584
55.425
49.172
43.738
41.948
38.553
33.118
31.827
31.609
29.865
29.517
29.156
29.021
28.882
25.870
22.460
21.826
20.755
13.928

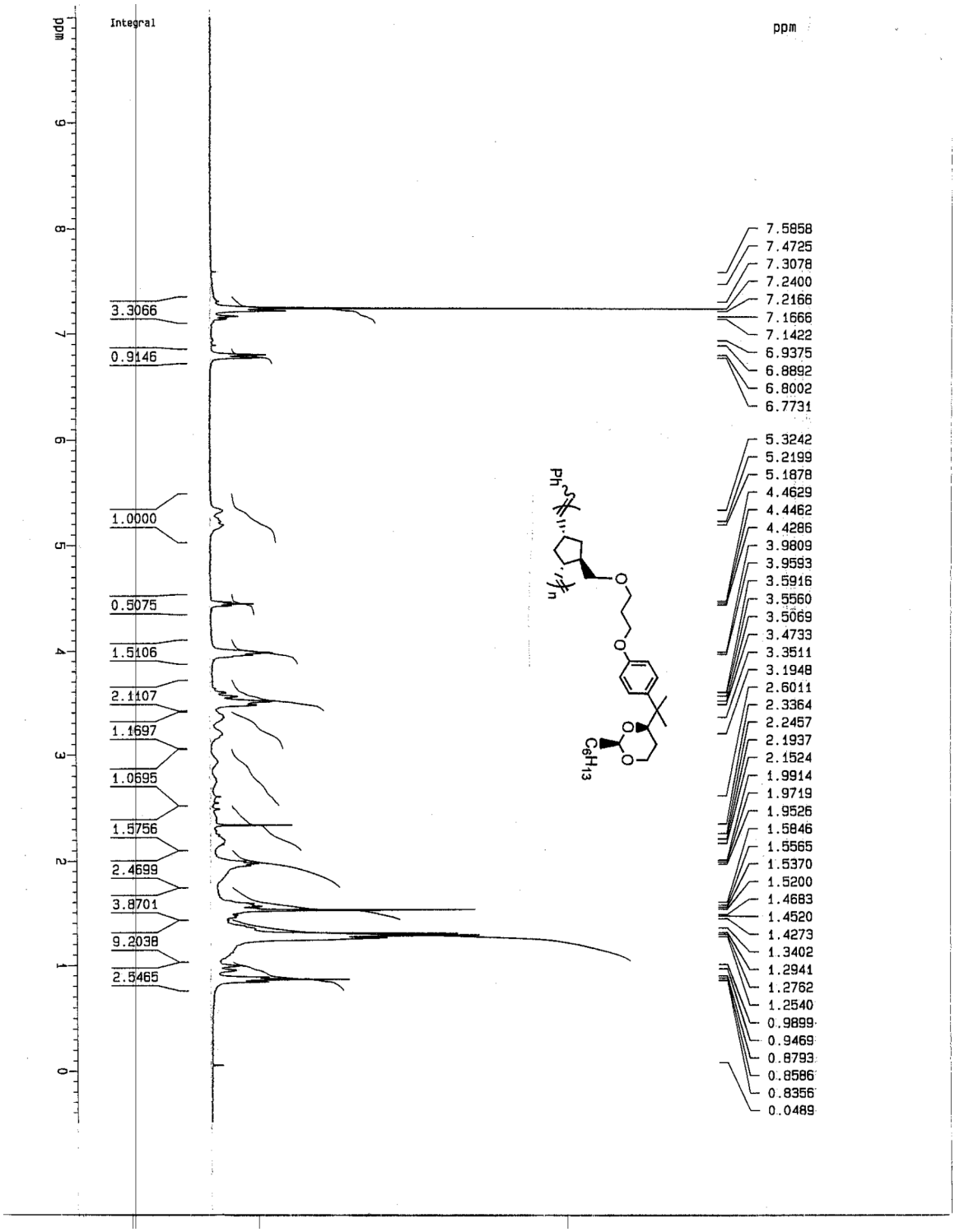

ppm
Integral
9.973
2.544
2.281
1.000
4.879
2.731
2.346
1.372
2.103
5.547
25.702
4.126
7.58641
7.30334
7.24031
7.21101
7.13264
6.94968
6.76474
6.74444
5.30088
5.27797
5.20623
5.17223
4.64957
4.63007
3.93089
3.87980
3.46331
3.31892
3.30027
3.29164
2.99401
2.96828
2.46092
2.33695
2.15265
2.09570
2.05814
1.93907
1.50845
1.25759
1.23514
1.16274
1.09136
0.87197
0.84988
0.69742
0.05100
Ph
OC8H17
OAc
n

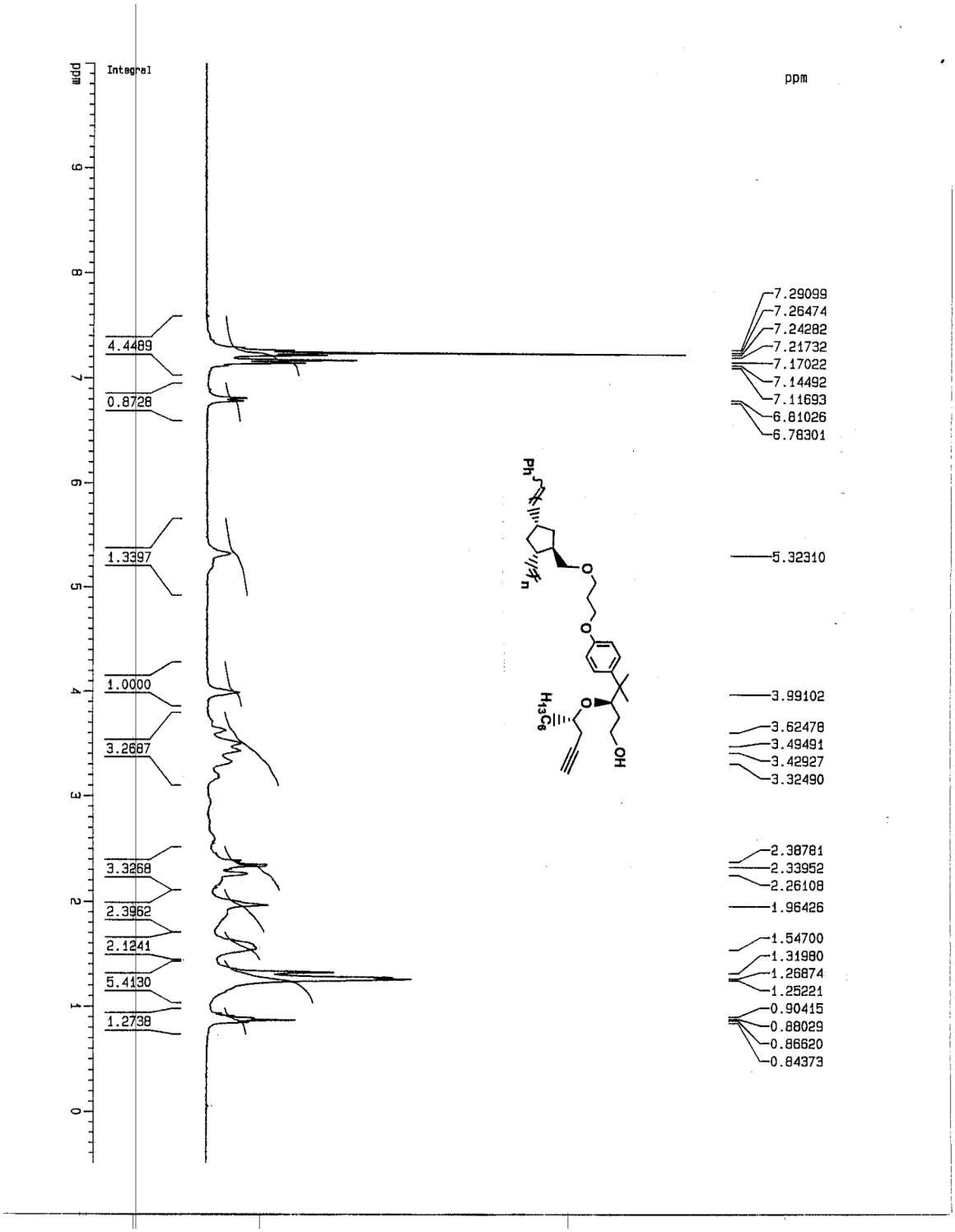

Integral
ppm
3.4770
0.9476
1.0000
1.5851
1.1193
0.9255
1.6883
2.7777
2.7300
6.1210
1.6828
7.29411
7.23970
7.21393
7.13393
6.77599
6.75103
5.29107
5.28466
5.28013
3.94036
3.93547
3.87842
3.45626
3.31441
3.28609
3.09077
3.06722
3.04417
2.15393
1.93742
1.88214
1.86711
1.53701
1.23074
1.18739
1.16285
1.15099
0.84998
0.82618
Ph
O
O
n
Ph
OC8H17
I

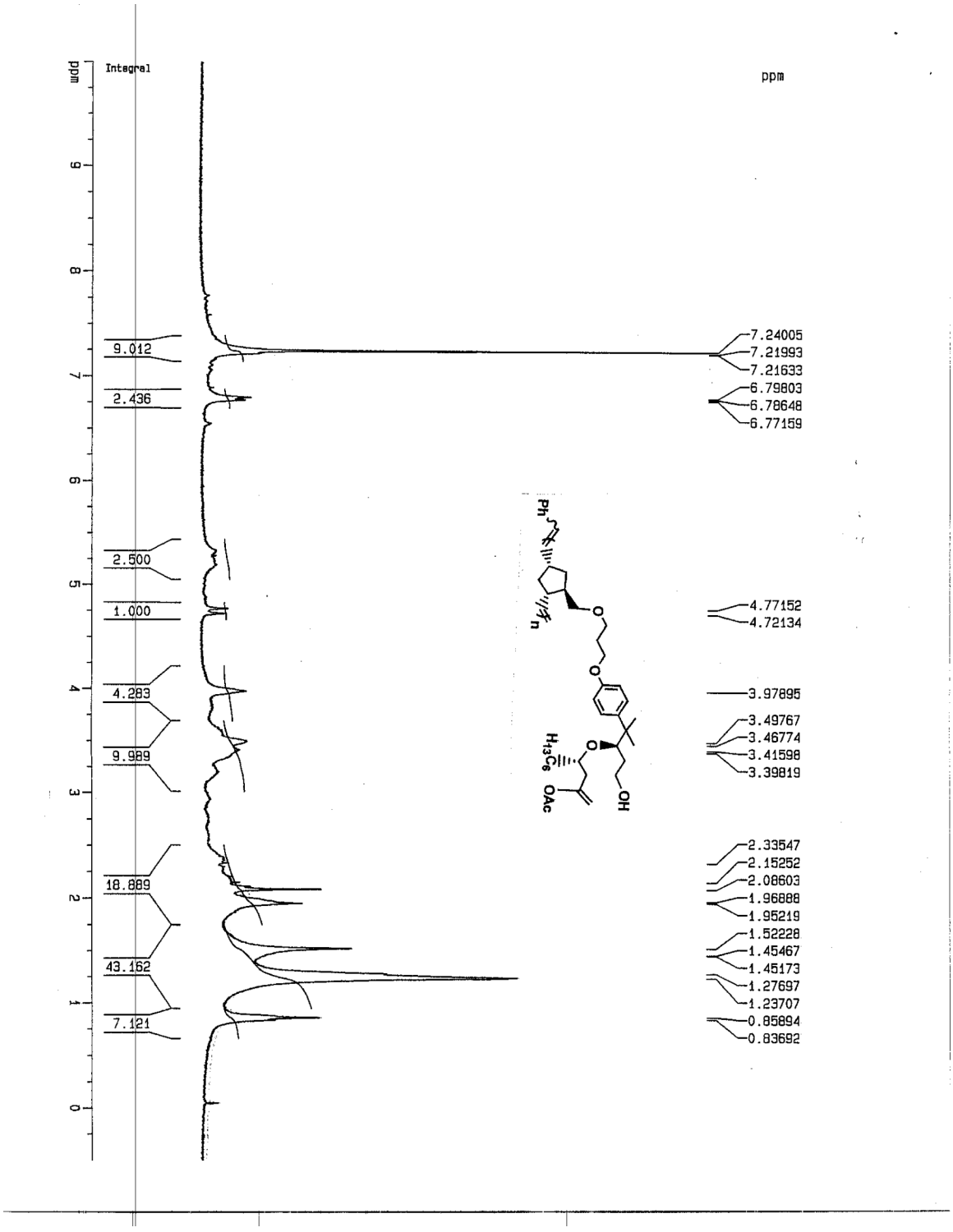

Integral
ppm
6.014
1.425
1.003
1.761
3.746
4.091
3.661
16.407
7.45552
7.26821
7.24469
7.19824
7.17085
6.79713
6.77904
5.25428
5.23874
5.23188
5.20750
3.95846
3.95541
3.92111
3.49786
3.49486
3.35987
3.33743
3.00418
2.62177
2.36790
2.11663
1.96664
1.89952
1.63852
1.63444
1.26844
1.19364
1.19027
1.12357
0.90344
0.88225
0.70903
0.54949
0.08362
Ph
O
O
Ph
OC8H17
n

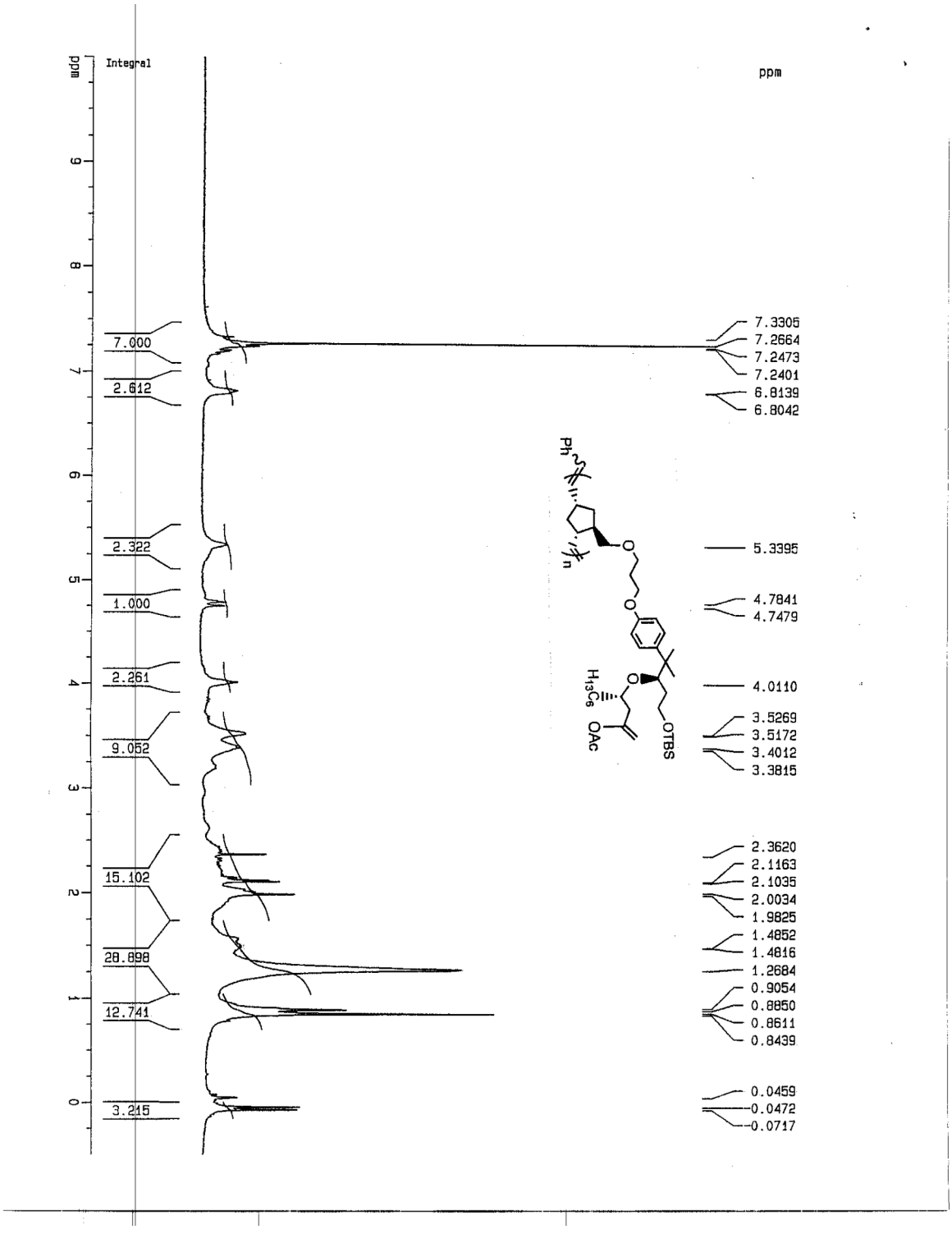

ppm
Integral
3.451
2.369
1.000
0.901
1.063
3.551
4.888
2.183
1.837
1.740
2.919
2.832
30.015
9.771
2.138
ppm
7.26817
7.25260
7.24074
6.82766
6.80434
6.79826
6.08695
6.07802
6.06792
5.88399
5.87427
4.47442
4.45733
4.04451
4.02368
4.00254
3.57468
3.54248
3.51831
3.48664
3.03830
3.00792
2.86430
2.75516
2.32057
2.02063
1.99993
1.97916
1.53334
1.30807
1.28975
1.25738
1.25456
0.88576
0.86482
0.47773
0.47172
C6H13

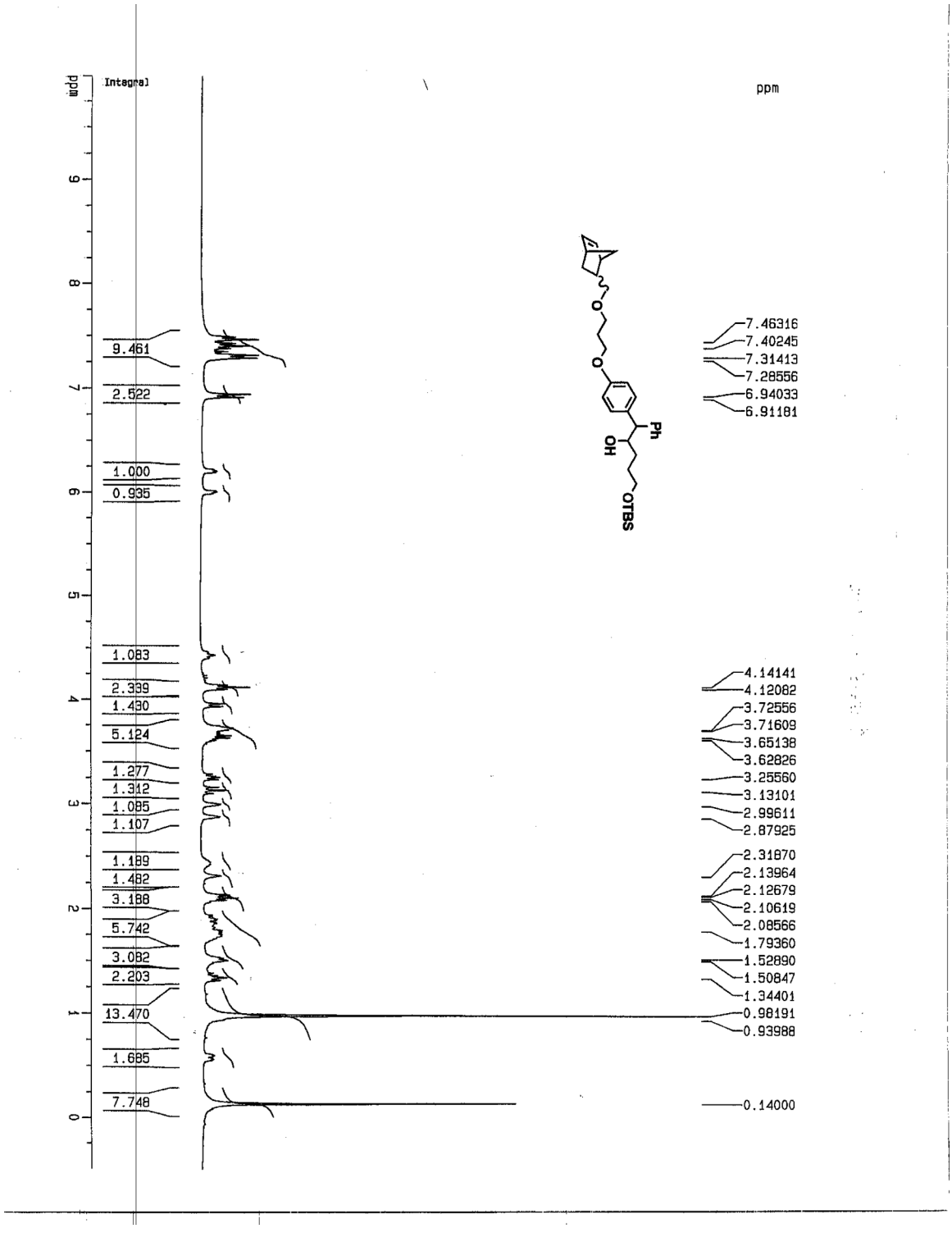

ppm
157.058
138.547
136.978
132.356
127.602
113.679
102.175
84.063
77.425
77.202
77.002
76.578
74.616
67.262
66.679
64.709
49.312
43.871
42.079
40.562
38.684
34.949
31.739
29.679
29.052
25.881
23.921
22.612
22.509
14.048
C6H13

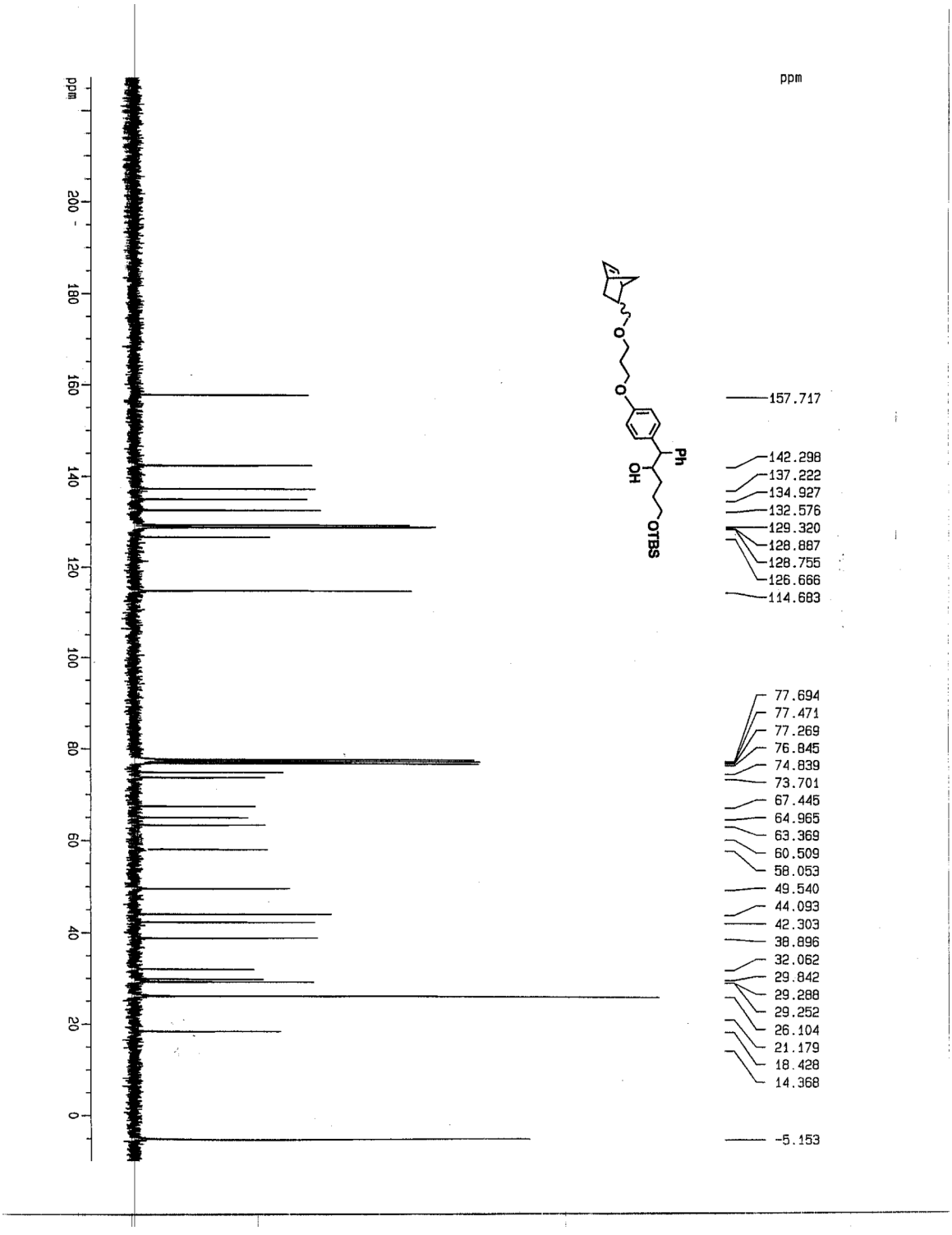

Integral
ppm
3.3066
0.9146
1.0000
0.5075
1.5106
2.1107
1.1697
1.0695
1.5756
2.4699
3.8701
9.2038
2.5465
7.5858
7.4725
7.3078
7.2400
7.2166
7.1666
7.1422
6.9375
6.8892
6.8002
6.7731
5.3242
5.2199
5.1878
4.4629
4.4462
4.4286
3.9809
3.9593
3.5916
3.5560
3.5069
3.4733
3.3511
3.1948
2.6011
2.3364
2.2457
2.1937
2.1524
1.9914
1.9719
1.9526
1.5846
1.5565
1.5370
1.5200
1.4683
1.4520
1.4273
1.3402
1.2941
1.2762
1.2540
0.9899
0.9469
0.8793
0.8586
0.8356
0.0489

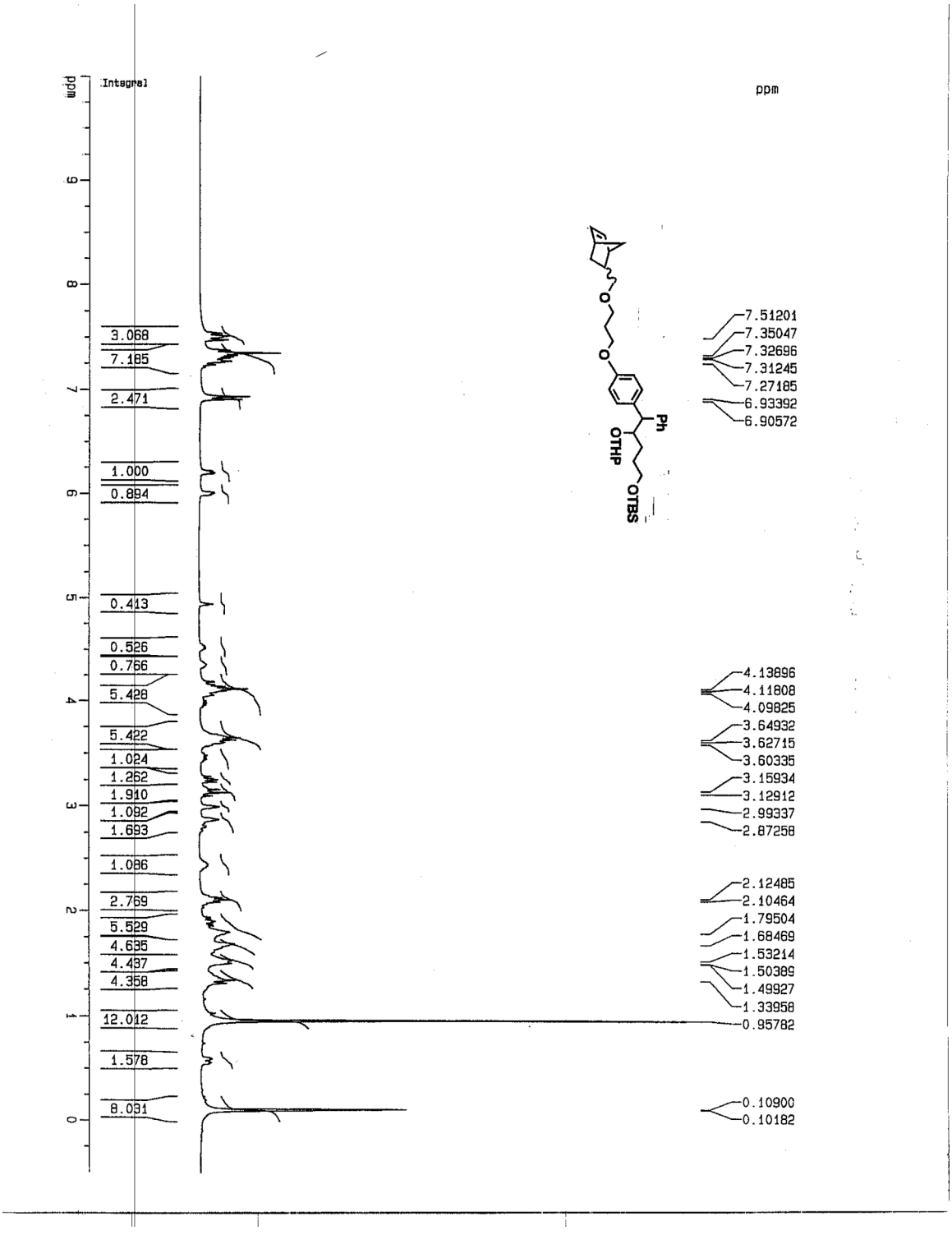

Integral
ppm
4.4489
0.8728
1.3397
1.0000
3.2687
3.3268
2.3962
2.1241
5.4130
1.2738
7.29099
7.26474
7.24282
7.21732
7.17022
7.14492
7.11693
6.81026
6.78301
5.32310
3.99102
3.62478
3.49491
3.42927
3.32490
2.38781
2.33952
2.26108
1.96426
1.54700
1.31980
1.26874
1.25221
0.90415
0.88029
0.86620
0.84373
Ph
n
H13C6
OH

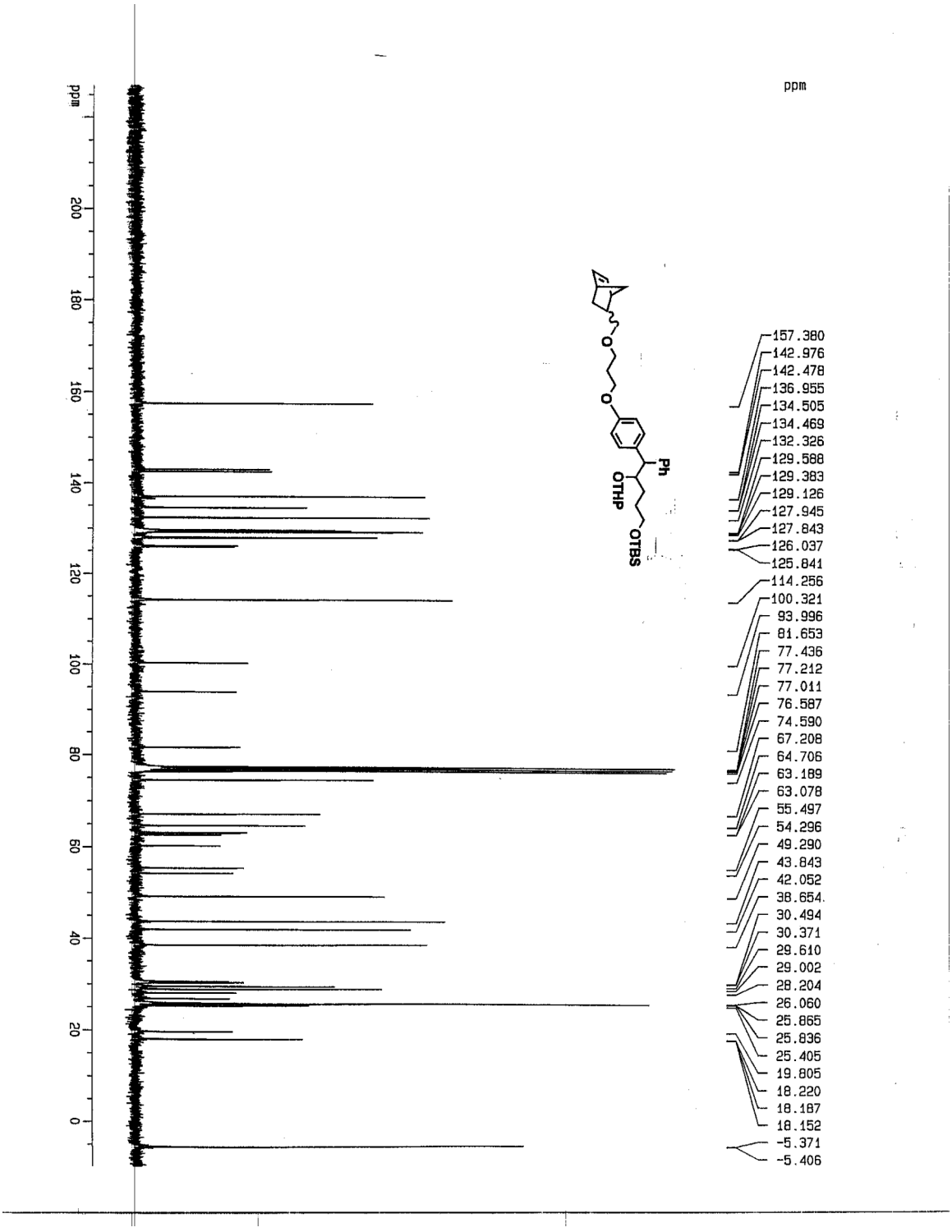

ppm
Integral
9.012
2.436
2.500
1.000
4.283
9.989
18.889
43.162
7.121
7.24005
7.21993
7.21633
6.79803
6.78648
6.77159
4.77152
4.72134
3.97895
3.49767
3.46774
3.41598
3.39819
2.33547
2.15252
2.08603
1.96888
1.95219
1.52228
1.45467
1.45173
1.27697
1.23707
0.85894
0.83692
Ph
n
O
O
O
OAc
OH
H13C6

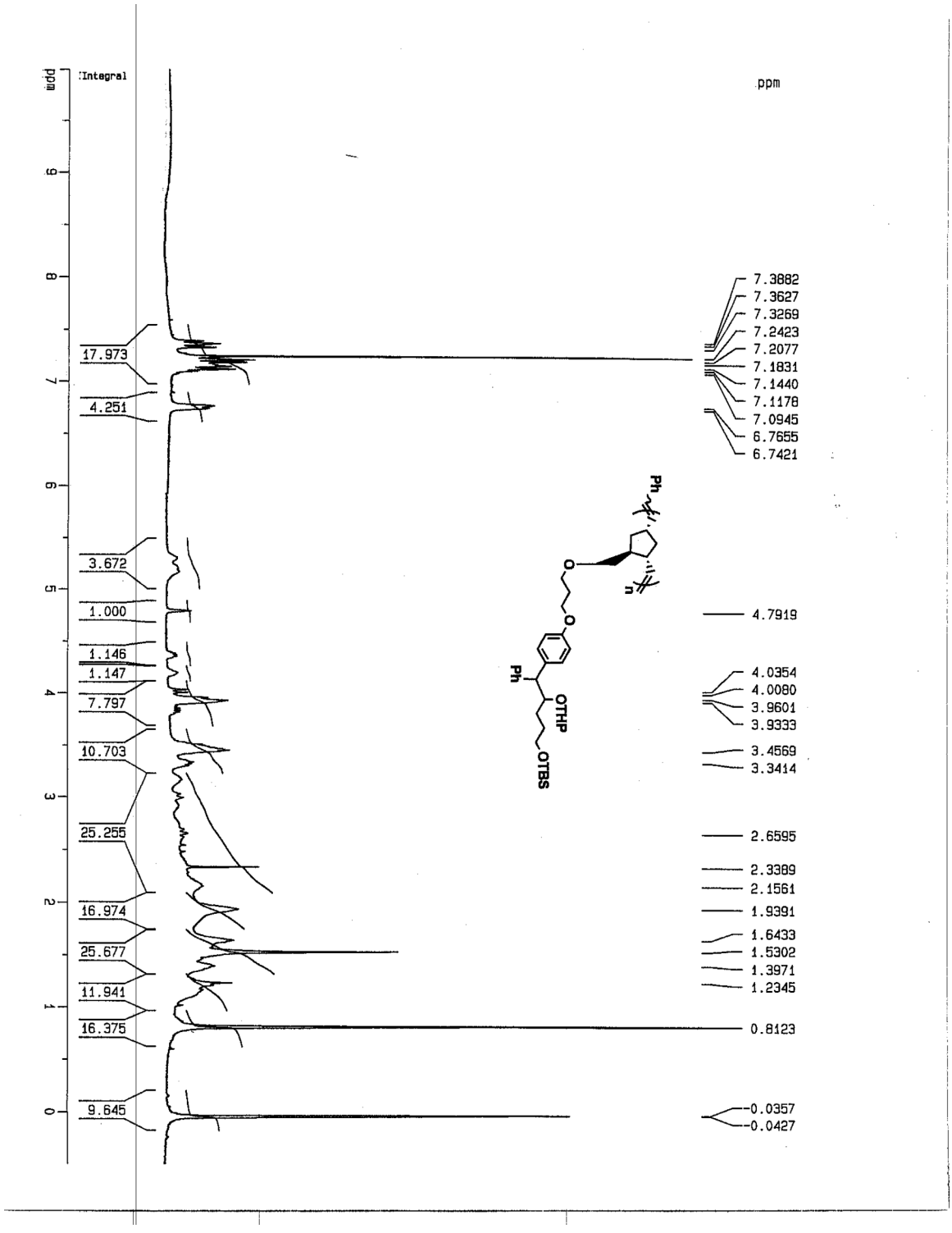

ppm
Integral
ppm
7.000
2.612
2.322
1.000
2.261
9.052
15.102
28.898
12.741
3.215
7.3305
7.2664
7.2473
7.2401
6.8139
6.8042
5.3395
4.7841
4.7479
4.0110
3.5269
3.5172
3.4012
3.3815
2.3620
2.1163
2.1035
2.0034
1.9825
1.4852
1.4816
1.2684
0.9054
0.8850
0.8611
0.8439
0.0459
0.0472
-0.0717
Ph
n
OAc
OTBS

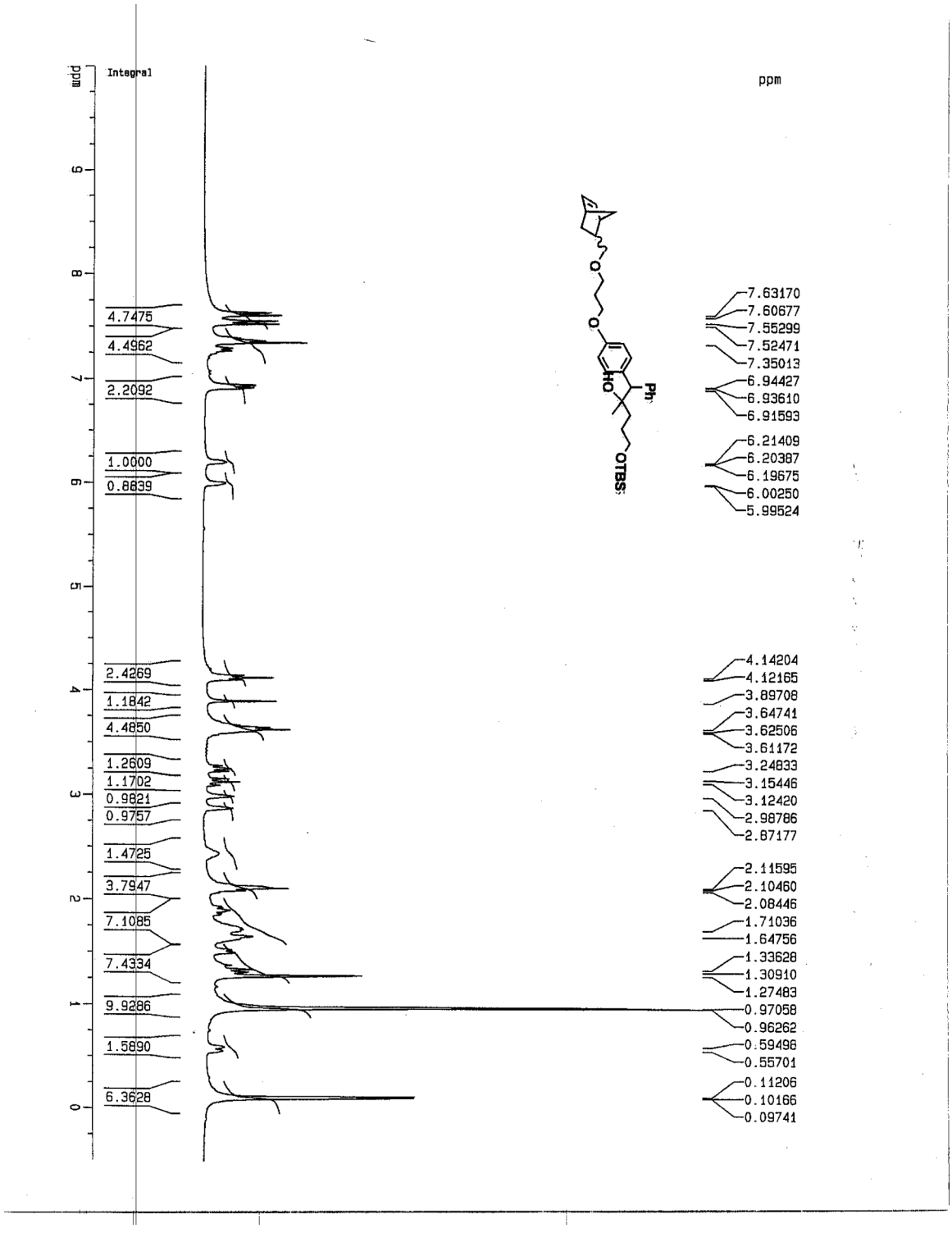

ppm
Integral
9.461
2.522
1.000
0.935
1.083
2.399
1.480
5.124
1.277
1.312
1.085
1.107
1.189
1.482
3.188
5.742
3.082
2.203
13.470
1.685
7.748
ppm
7.46316
7.40245
7.31413
7.28556
6.94033
6.91181
4.14141
4.12082
3.72556
3.71609
3.65138
3.62826
3.25560
3.13101
2.99611
2.87925
2.31870
2.13964
2.12679
2.10619
2.08566
1.79360
1.52890
1.50847
1.34401
0.98191
0.93988
0.14000
Ph
OH
OTBS

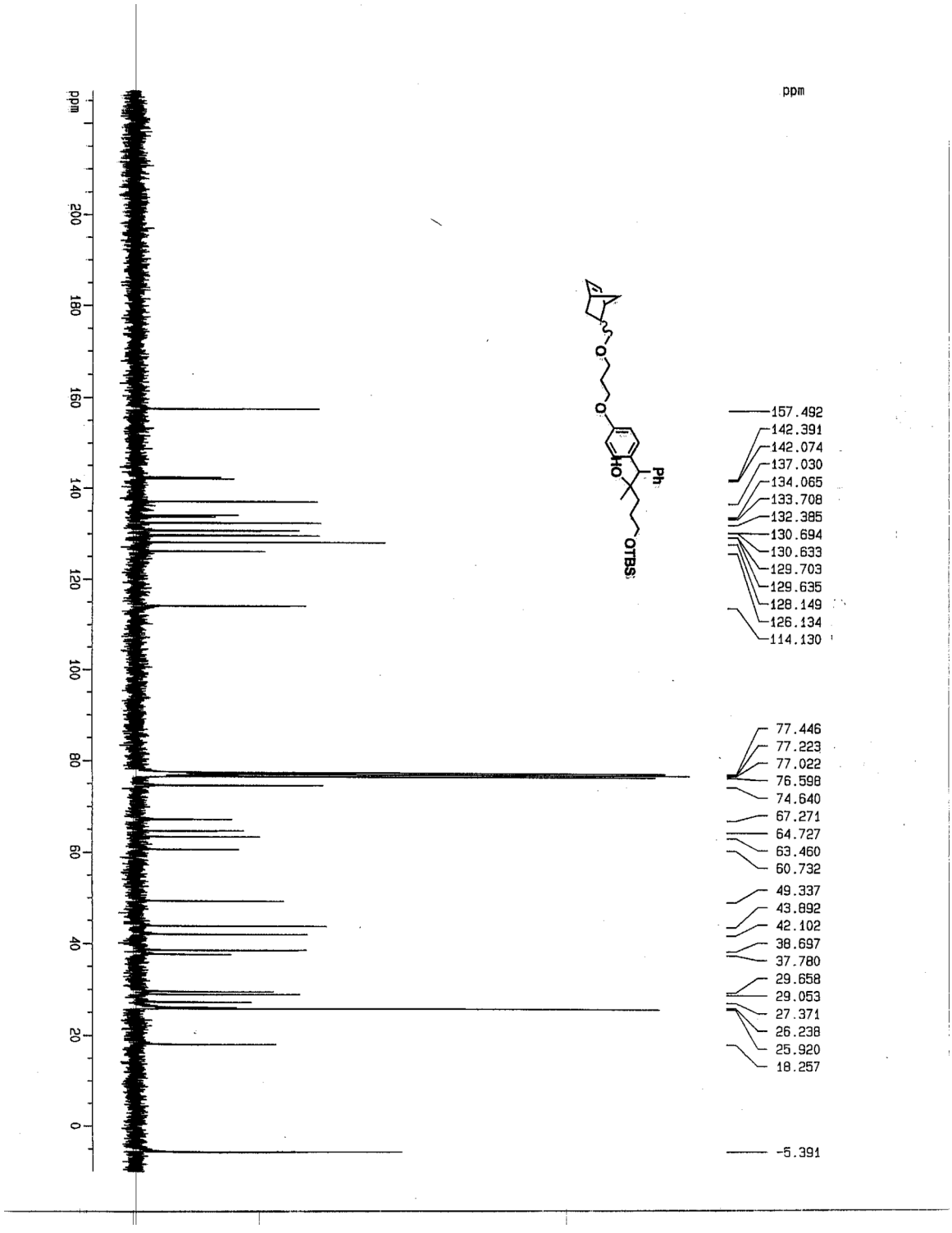

ppm
200
180
160
140
120
100
80
60
40
20
0
Ph
OH
OTBS
157.717
142.298
137.222
134.927
132.576
129.320
128.887
128.755
126.666
114.683
77.694
77.471
77.269
76.845
74.839
73.701
67.445
64.965
63.369
60.509
58.053
49.540
44.093
42.303
38.896
32.062
29.842
29.288
29.252
26.104
21.179
18.428
14.368
-5.153

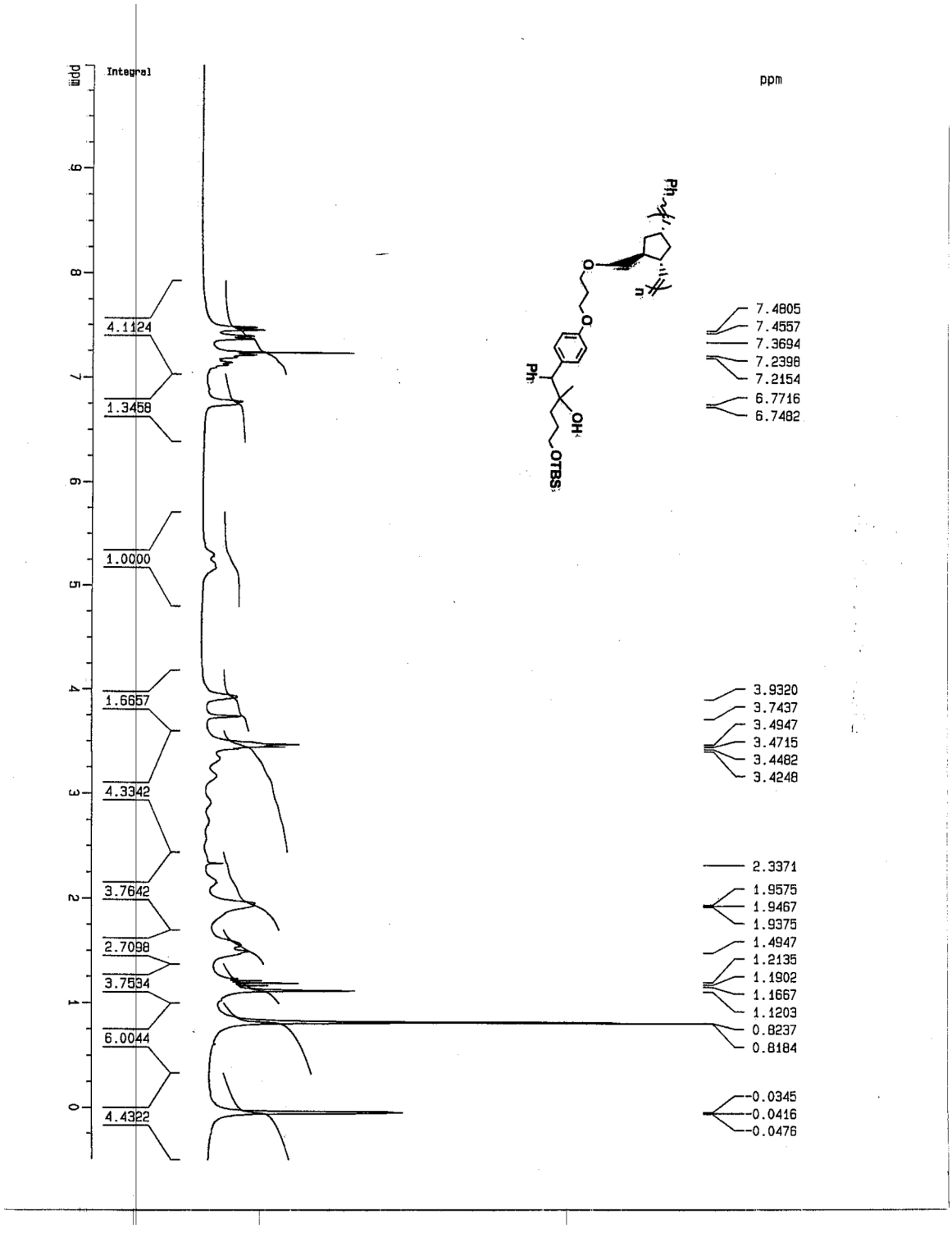

ppm
Integral
3.068
7.185
2.471
1.000
0.894
0.413
0.526
0.766
5.428
5.422
1.024
1.262
1.910
1.092
1.693
1.086
2.769
5.529
4.695
4.487
4.358
12.012
1.578
8.031
ppm
7.51201
7.35047
7.32696
7.31245
7.27185
6.93392
6.90572
4.13896
4.11808
4.09825
3.64932
3.62715
3.60335
3.15934
3.12912
2.99337
2.87258
2.12485
2.10464
1.79504
1.68469
1.53214
1.50389
1.49927
1.33958
0.95782
0.10900
0.10182
Ph
OTHP
OTBS


ppm
200
180
160
140
120
100
80
60
40
20
0
O
O
Ph
OTHP
OTBS
ppm
157.380
142.976
142.478
136.955
134.505
134.469
132.326
129.588
129.383
129.126
127.945
127.843
126.037
125.841
114.256
100.321
93.996
81.653
77.436
77.212
77.011
76.587
74.590
67.208
64.706
63.189
63.078
55.497
54.296
49.290
43.843
42.052
38.654
30.494
30.371
29.610
29.002
28.204
26.060
25.865
25.836
25.405
19.805
18.220
18.187
18.152
-5.371
-5.406


ppm
Integral
17.973
4.251
3.672
1.000
1.146
1.147
7.797
10.703
25.255
16.974
25.677
11.941
16.375
9.645
7.3882
7.3627
7.3269
7.2423
7.2077
7.1831
7.1440
7.1178
7.0945
6.7655
6.7421
4.7919
4.0354
4.0080
3.9601
3.9333
3.4569
3.3414
2.6595
2.3389
2.1561
1.9391
1.6433
1.5302
1.3971
1.2345
0.8123
-0.0357
-0.0427
Ph
O
O
Ph
OTHP
OTBS


ppm
Integral
4.7475
4.4962
2.2092
1.0000
0.8639
2.4269
1.1842
4.4850
1.2609
1.1702
0.9821
0.9757
1.4725
3.7947
7.1085
7.4334
9.9286
1.5890
6.3628
7.63170
7.60677
7.55299
7.52471
7.35013
6.94427
6.93610
6.91593
6.21409
6.20387
6.19675
6.00250
5.99524
4.14204
4.12165
3.89708
3.64741
3.62506
3.61172
3.24833
3.15446
3.12420
2.98786
2.87177
2.11595
2.10460
2.08446
1.71036
1.64756
1.33628
1.30910
1.27483
0.97058
0.96262
0.59496
0.55701
0.11206
0.10166
0.09741
HO
Ph
OTBS


ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
157.492
142.391
142.074
137.030
134.065
133.708
132.385
130.694
130.633
129.703
129.635
128.149
126.134
114.130
77.446
77.223
77.022
76.598
74.640
67.271
64.727
63.460
60.732
49.337
43.892
42.102
38.697
37.780
29.658
29.053
27.371
26.238
25.920
18.257
-5.391
HO
Ph
OTBS


ppm
Integral
4.1124
1.3458
1.0000
1.6657
4.3342
3.7642
2.7098
3.7534
6.0044
4.4322
ppm
7.4805
7.4557
7.3694
7.2398
7.2154
6.7716
6.7482
3.9320
3.7437
3.4947
3.4715
3.4482
3.4248
2.3371
1.9575
1.9467
1.9375
1.4947
1.2135
1.1902
1.1667
1.1203
0.8237
0.8184
-0.0345
-0.0416
-0.0476
Ph
O
O
Ph
OH
OTBS
n