Supporting Information

Remote Stereoinduction in the Acylation of Fully-Substituted Enolates: Tandem Reformatsky/Quaternary Claisen Condensations of Silyl Glyoxylates and β-Lactones

Stephen N. Greszler, Justin T. Malinowski, and Jeffrey S. Johnson*

Department of Chemistry, University of North Carolina at Chapel Hill, Chapel Hill, North Carolina 27599-3290

jsj@unc.edu

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Methods: General. Infrared (IR) spectra were obtained using a Jasco 460 Plus Fourier transform infrared spectrometer. Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on a Bruker model Avance 400 (^{1}H NMR at 400 MHz and ^{13}C at 100 MHz) or a Bruker model Avance 500 (^{1}H NMR at 500 MHz and ^{13}C NMR at 125 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, t = triplet, br t = broad triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Mass spectra were obtained using a Bruker BioTOF II spectrometer with electrospray ionization calibrated with CsOAc. All samples were prepared in methanol. Analytical thin layer chromatography (TLC) was performed on Sorbent Technologies 0.20 mm Silica G TLC plates. Visualization was accomplished with UV light and/or aqueous ceric ammonium nitrate solution followed by heating. Purification of the reaction products was carried out by flash chromatography using Siliaflash-P60 silica gel (40-63μm) purchased from Silicycle. Purification via HPLC was performed on a Varian Prepstar SD-1 Solvent Delivery System equipped with a Cyano 60 Å 6u column from Berger Instruments. Specific parameters used in the separation of compounds are detailed under applicable entries. Unless otherwise noted, all reactions were carried out under an atmosphere of dry nitrogen in oven-dried glassware with magnetic stirring. Yield refers to isolated yield of analytically pure material unless otherwise noted. Yields are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments

Materials: General. Tetrahydrofuran, diethyl ether, dichloromethane, and toluene were dried by passage through a column of neutral alumina under nitrogen prior to use. Zinc metal was washed with 1 M HCl, water, acetone, and diethyl ether and then dried under vacuum at 60 °C for 16 h prior to storage in a nitrogen-filled glove box. Lithium chloride was dried and stored in a 100 °C oven. Diisopropylethylamine and triethylamine were freshly distilled from calcium hydride prior to use. Propionyl chloride, propionyl bromide, acetyl bromide, and hydrocinnimaldehyde were distilled under nitrogen immediately before use. Lactones **S1-S5**,[1] **S6**,[2] **S7**,[3] **S8**,[4] and lactams[5] **S9**[6] and **S10**[7] were obtained using known procedures. Silyl glyoxylates **S11-S12**[8] and **S13**[9] were prepared according to the published procedures. All other reagents were purchased from commercial sources and were used as received unless otherwise noted.

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[1] Nelson, S. G., Peelen, T. J., Wan, Z. *J. Am. Chem. Soc.* **1999**, *121*, 9742-9743.

[2] Yang, H. W., Romo, D. *J. Org. Chem.* **1997**, *62*, 4-5.

[3] Wang, Y., Zhao, C., Romo, D. *Org. Lett.* **1999**, *1*, 1197-1199.

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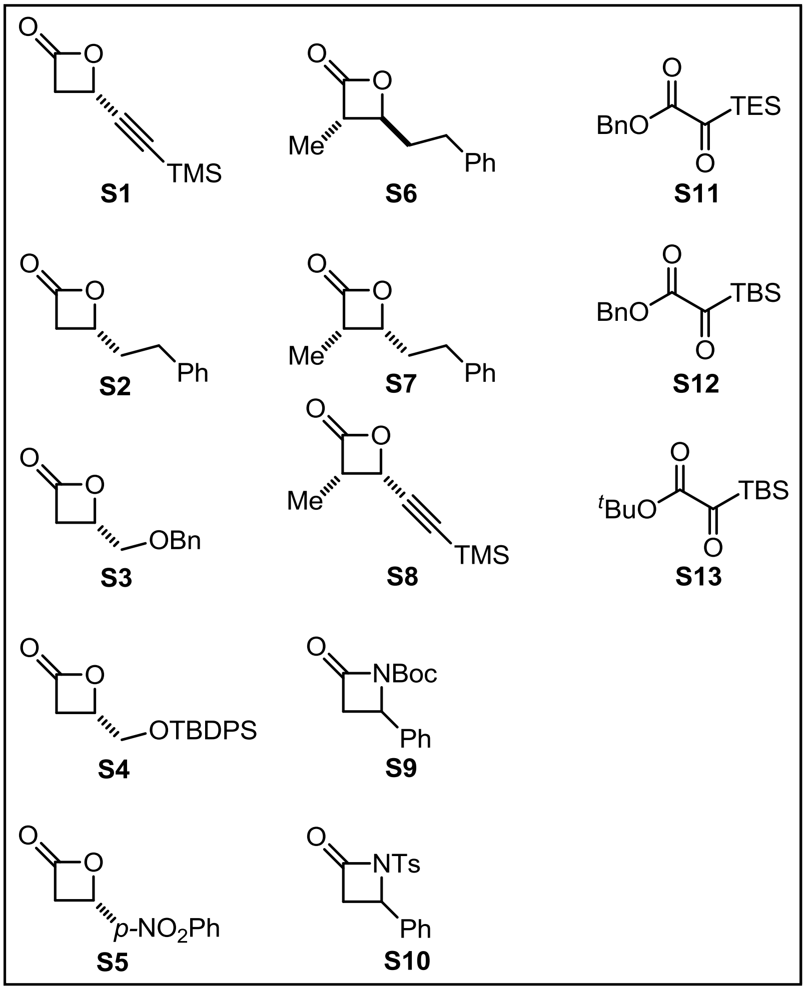
[5] Kim, S., Lee, P. H., Lee, T. A. *Synth. Commun.* **1988**, *18*, 247-252.

[6] Deshmukh, A. R., Chincholkar, P. M., Kale, A. S., Gumaste, V. K. *Tetrahedron.* **2009**, *65*, 2605-2609.

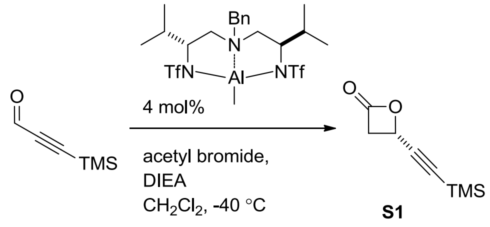
[7] Otto, H-H., Bergmann, H-J. *Arch. Pharm.* **1986**, *319*, 635-641.

[8] Greszler, S. N.; Johnson, J. S. *Angew. Chem. Int. Ed*. **2009**, *48*, 3689.

[9] Nicewicz, D. A.; Brétéché, G.; Johnson, J. S. *Org. Synth.* **2008**, *85*, 278.



Preparation of S1:



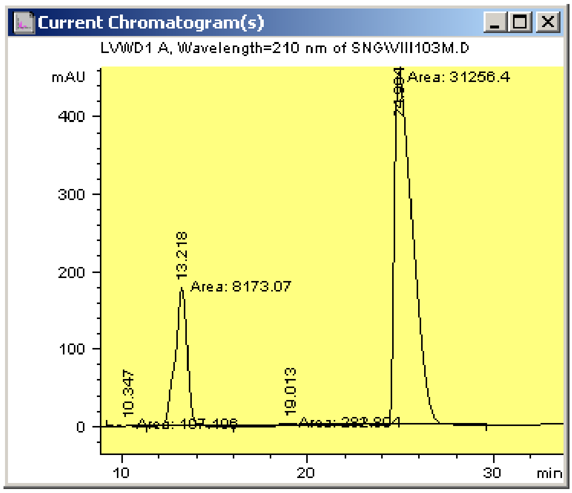
(*S*)-4-((trimethylsilyl)ethynyl)oxetan-2-one (S1):

The title compound was prepared according to the procedure described by Nelson[1] with the following modifications:

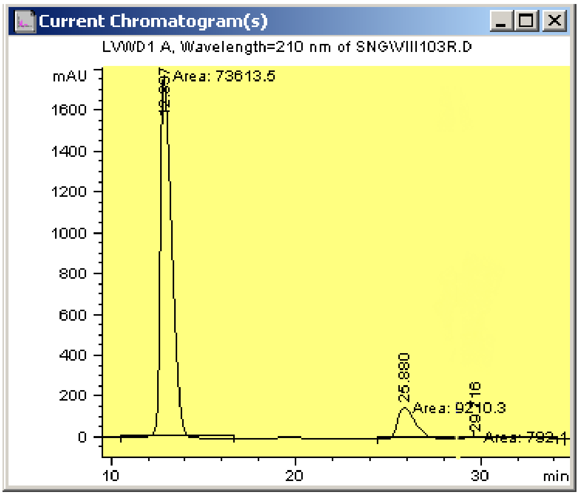
1. Instead of purification via Kugelrohr distillation, the crude β-lactone was purified via flash chromatography (92.5:7.5 to 85:15 hexanes:ethyl acetate), affording the title compound (67% yield) as a light yellow oil whose spectral properties matched those reported in the literature.[1]

2. The enantiomeric excess of the prepared lactone was assayed via supercritical fluid chromatographic (SFC) analysis of the corresponding β-hydroxyketone **3b** (*vide infra*). Enantiomeric excesses ranged from 78-83% using this method. CSP-SFC analysis of a sample of **3b** showed that the product was enriched to 78% ee (Chiralpak OD column, 3.0% MeOH, 1.0 mL/min, 150 psi, 24 °C, 210 nm, t_r-major enantiomer: 12.9 min, t_r-minor enantiomer: 25.9 min; CSP-SFC traces for a mixture of enantiomers and of the enantioenriched product are attached below:

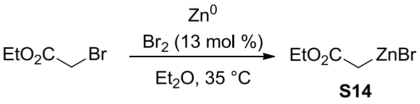
Enantiomeric Mixture:



Enantioenriched Sample:

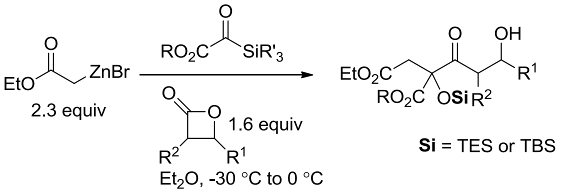


Preparation of Reformatsky Reagent (S14):

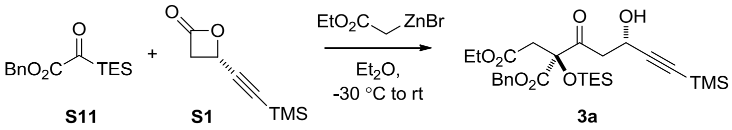


An oven-dried 100-mL round-bottomed flask equipped with a magnetic stir bar was charged with zinc dust (1.41 g, 21.6 mmol, 2 equiv) and diethyl ether (25 mL). The flask was fitted with a condenser and purged with nitrogen. Br_2 (0.07 mL, 1.4 mmol, 0.13 equiv) was added dropwise over 5 min with stirring (exotherm observed). The suspension was heated to reflux, and ethylbromoacetate (1.2 mL, 10.8 mmol, 1.0 equiv) was added dropwise over 15 min. The solution was stirred at this temperature for 4 h then cooled to RT. An aliquot was titrated with I_2, typically reflecting concentrations of active reagent of 0.35-0.43 M (81-100% yield). The solution was stored under nitrogen at 0 °C for up to one week and titrated immediately prior to each subsequent use.

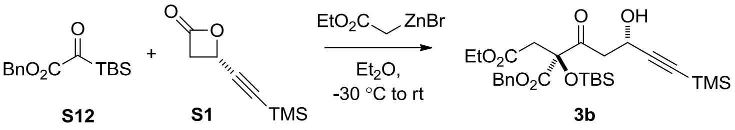
General Procedure A for the Reformatsky Initiated Cascade Coupling of Silyl Glyoxylate and β-Lactones Affording β-hydroxyketones 3a-c, 3h-k



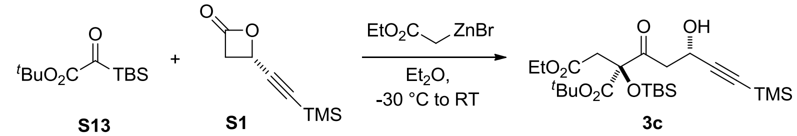
An oven-dried 20-mL scintillation vial equipped with magnetic stir bar was purged with nitrogen and a solution of Reformatsky reagent (0.38 M, 1.09 mL, 2.3 equiv) was added. The resulting suspension was diluted with diethyl ether (1.0 mL) and cooled to -30 °C in an acetone/dry ice bath (bath temperature, monitored with a thermocouple probe). A second oven-dried vial was charged with silyl glyoxylate **S11-S13** (0.18 mmol, 1.0 equiv) and β-lactone **S1-S8** (0.29 mmol, 1.6 equiv). The vial was purged with nitrogen, and diethyl ether (1.5 mL) was added. This solution was cooled and added to the solution of Reformatsky reagent. Additional diethyl ether (0.5 mL) was used to rinse the vial. The reaction was allowed to warm slowly in the acetone bath (generally over 30 min from -30 °C to 0 °C). Consumption of the silyl glyoxylate was generally observed by TLC analysis and disappearance of yellow color between -20 °C and -15 °C. The reaction was then held at 0 °C until judged complete by TLC analysis, generally 30 min. Saturated aqueous ammonium chloride (0.5 mL) was then added and the reaction was stirred until clear layers were observed. The organic layer was removed, and the aqueous layer was extracted with diethyl ether (3 x 1.0 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The crude product was purified via flash chromatography to give the desired product.



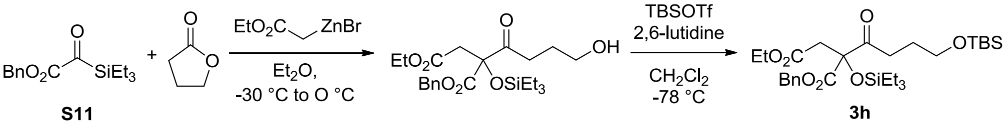
(*S*)-1-benzyl-4-ethyl-2-((*S*)-3-hydroxy-5-(trimethylsilyl)pent-4-ynoyl)-2-((triethylsilyl)oxy)succinate (3b): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv) and (*S*)-4-((trimethylsilyl)ethynyl)oxetan-2-one (**S1**, 49 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (93.5:7.5 to 70:30 petroleum ether: diethyl ether) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (59 mg, 61%). Analytical data**:** $[\alpha]_D^{25.3}$ -5.30 (*c* 1.25, $CHCl_3$); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.36-7.31 (m, 5H), 5.17 (d, *J* = 12.0 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 4.87 (dd, *J* = 6.5, 4.5 Hz, 1H), 4.09 (q, *J* = 7.5 Hz, 2H), 3.47 (d, *J* = 17 Hz, 1H), 3.36 (dd, *J* = 18.5, 2.5 Hz, 1H), 3.12 (dd, *J* = 18.5, 9.0 Hz, 1H), 2.91 (d, *J* = 17.0 Hz, 2H), 1.22 (t, *J* = 7.0 Hz, 3H), 0.89 (t, *J* = 8.0 Hz, 9H), 0.56 (q, *J* = 8.0 Hz, 6H), 0.56 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$)**:** δ 208.8, 169.3, 168.5, 134.4, 128.7, 128.6, 128.5, 104.8, 89.3, 83.7, 68.1, 61.1, 58.8, 46.0, 42.5, 14.0, 6.7, 5.7, -0.2; **LRMS** (ESI$^+$) Calcd. for $C_{27}H_{42}O_7Si_2$+H, 535.3; Found, 535.3; **IR** (thin film, cm^{-1}) 3515, 2958, 2911, 2878, 2176, 1738, 1456, 1373, 1343, 1250, 1181, 844, 699; **TLC** (80:20 Hex:EtOAc): R_f = 0.42.



(*R*)-1-benzyl 4-ethyl 2-((*tert*-butyldimethylsilyl)oxy)-2-((*R*)-3-hydroxy-5- (trimethylsilyl) pent-4-ynoyl)succinate (3b): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S12**, 50 mg, 0.18 mmol, 1.0 equiv) and (*S*)-4-((trimethylsilyl)ethynyl)oxetan-2-one (**S1**, 49 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (93.5:7.5 to 70:30 petroleum ether: diethyl ether) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (67 mg, 70%). Analytical data: $[\alpha]_D^{25.2}$ + 7.8 (*c* 0.43, CHCl$_3$); **^{1}H NMR** (400 MHz, CDCl$_3$): δ 7.38-7.27 (m, 5H), 5.17 (d, *J* = 12.0 Hz, 1H), 5.09 (d, *J* = 12.0 Hz, 1H), 4.86 (dd, *J* = 9.2, 4.8 Hz, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.46 (d, J = 16.8 Hz, 1H), 3.37 (dd, *J* = 18.8, 2.4 Hz, 1H), 3.13 (dd, *J* = 18.4, 8.8 Hz, 1H), 2.95 (d, *J* = 17.2, 1H), 2.91 (d, *J* = 4.4, 1H), 1.22 (t, *J* = 7.2 Hz, 3H), 0.86 (s, 9H), 0.15 (s, 9H), 0.04 (s, 3H), 0.02 (s, 3H); **^{13}C NMR** (100 MHz, CDCl$_3$)**:** δ 208.8, 169.3, 168.3, 134.3, 128.7, 128.6, 128.5, 104.9, 89.3, 83.6, 68.1, 61.1, 58.7, 46.3, 42.2, 25.5, 18.2, 14.0, -0.3, -3.6, -4.2; **LRMS (ESI$^+$)** Calcd. for $C_{27}H_{42}O_7Si_2$+Na, 557.3; Found, 557.2; **IR** (thin film, cm^{-1}) 3433, 2844, 2386, 2100, 1646, 1558, 1541, 1456, 1250, 1013, 494; **TLC** (80:20 Hexanes:EtOAc): R$_f$ = 0.42.

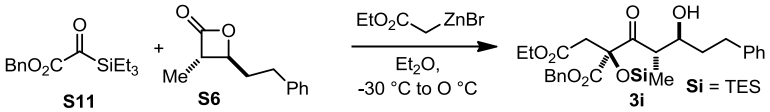


1-tert-butyl 4-ethyl 2-((tert-butyldimethylsilyl)oxy)-2-(3-hydroxy-5-(trimethylsilyl)pent-4-ynoyl)succinate (3c): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S13**, 44 mg, 0.18 mmol, 1.0 equiv) and (*S*)-4-((trimethylsilyl)ethynyl)oxetan-2-one (**S1**, 49 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (93.5:7.5 hexanes:ethyl acetate) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (31 mg, 34%). Analytical data: **^{1}H NMR** (500 MHz, CDCl$_3$): δ 4.88-4.86 (m, 1H), 4.08 (q, *J* = 7.0 Hz, 2H), 3.43 (d, *J* = 17.5 Hz, 1H), 3.39 (dd, *J* = 18.5, 2.5 Hz, 1H), 3.10 (dd, *J* = 18.5, 9.0 Hz, 1H), 3.02 (d, *J* = 5.0 Hz, 1H), 2.88 (d, *J* = 17.5 Hz, 1H), 1.44 (s, 9H), 1.23 (t, *J* = 7.5 Hz, 3H), 0.91 (s, 9H), 0.16 (s, 3H), 0.15 (s, 9H), 0.05 (s, 3H); **^{13}C NMR** (125 MHz, CDCl$_3$)**:** δ 209.6, 169.6, 167.4, 105.0, 89.2, 84.2, 83.7, 61.0, 58.7, 46.1, 42.0, 27.7, 25.6, 18.3, 14.1, -0.2, -3.4, -3.7; **LRMS (ESI$^+$)** Calcd. for $C_{24}H_{24}O_7Si_2$+Na, 523.3; Found, 523.2; **IR** (thin film, cm^{-1}) 3432, 2959, 2858, 2359, 1737, 1641, 1371, 1251, 1157, 911, 841, 733; **TLC**(75:25 Hex:EtOAc): R$_f$ = 0.48.

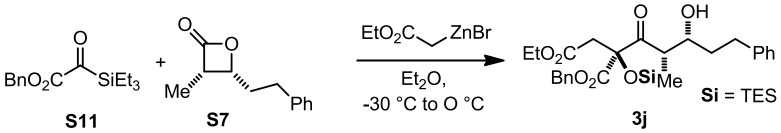


1-benzyl 4-ethyl 2-(4-((tert-butyldimethylsilyl)oxy)butanoyl)-2-((triethylsilyl)oxy)succinate (3h): The title compound was prepared according to General Procedure A using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and γ-butyrolactone (25 mg, 0.29 mmol, 1.6 equiv). The crude product was added to an oven-dried vial, which was purged with nitrogen. CH$_2$Cl$_2$ (1.5 mL) was added, and the resulting solution was cooled to -78

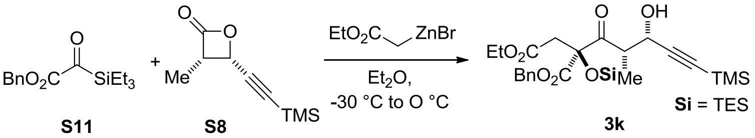
°C. 2,6-lutidine (0.02 mL, 0.17 mmol, 2 equiv) was then added, followed by tert-butyldimethylsilyl trifluoromethanesulfonate (0.02 mL, 0.09 mmol, 1.1 equiv). The reaction was allowed to stir for 20 min and was then quenched by the addition of an aqueous solution of HCl (1M, 0.5 mL). The layers were separated, and the organic layer was washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. Purification via flash chromatography (100:0 to 95:5 hexanes: ethyl acetate) provided the desired product as a light yellow oil (31 mg, 30%, 2 steps). Analytical data: **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.35-7.29 (m, 5H), 5.18 (d, *J* = 12.5 Hz, 1H), 5.09 (d, *J* = 12.5 Hz, 1H), 4.07 (q, *J* = 7.5 Hz, 2H), 3.60 (t, *J* = 6.5 Hz, 2H), 3.41 (d, *J* = 16.5 Hz, 1H), 2.99-2.95 (m, 1H), 2.91 (d, *J* = 16.5 Hz, 1H), 2.91-2.75 (m, 1H), 1.77-1.74 (m, 2H), 1.21 (t, *J* = 7.0 Hz, 3H), 0.90 (t, *J* = 7.0 Hz, 9H), 0.88 (s, 9H), 0.57 (q, *J* = 7.5 Hz, 6H), 0.03 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 209.3, 169.3, 169.1, 134.7, 128.6, 128.5, 128.4, 83.8, 67.8, 62.2, 60.8, 42.5, 34.5, 26.4, 25.9, 18.2, 14.0, 6.8, 5.8, -5.4; **LRMS (ESI$^+$)** Calcd. for $C_{29}H_{50}O_7Si_2$+Na, 589.3; Found, 589.3; **IR** (thin film, cm^{-1}) 3433, 3054, 2121, 1641, 1422, 126, 895, 738, 704; **TLC**(75:25 Hex:EtOAc): R_f = 0.58.



1-benzyl 4-ethyl 2-3-hydroxy-2-methyl-5-phenylpentanoyl)-2-((triethylsilyl)oxy)succinate (3i): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv) and *trans*-3-methyl-4-phenethyloxetan-2-one (**S6**, 55 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (50:50:0 to 0:100:0 to 0:95:5 hexanes: CH_2Cl_2: MeOH) provided the desired product as a light yellow oil with 5:1 diastereomeric ratio (56 mg, 56%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.43-7.09 (m, 10H), 5.17 (d, *J* = 12.0 Hz, 1H), 5.12 (d, *J* = 12.0 Hz, 1H), 4.10 (q, *J* = 7.2 Hz, 2H), 3.84-3.82 (m, 1H), 3.59-3.51 (m, 2H), 3.12 (d, *J* = 4.8 Hz, 1H), 2.93-2.84 (m, 2H), 2.75-2.67 (m, 1H), 1.97-1.78 (m, 1H), 1.78-1.59 (m, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 0.97 (d, *J* = 7.2 Hz, 3H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.70-0.54 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 212.2, 169.8, 169.2, 142.6, 134.4, 128.7, 128.6, 128.5, 125.6, 84.1, 72.2, 68.2, 61.3, 46.4, 42.9, 36.0, 31.6, 14.4, 14.0, 6.8, 5.9; **LRMS (ESI$^+$)** Calcd. for $C_{31}H_{44}O_7Si$+Na, 579.3; Found, 579.2; **IR** (thin film, cm^{-1}) 3528, 3028, 2855, 2912, 2877, 2733, 2359, 2249, 1950, 1740, 1455, 1373, 1343, 1211, 1020, 830, 737, 699; **TLC**(75:25 Hex:EtOAc): R_f = 0.42.

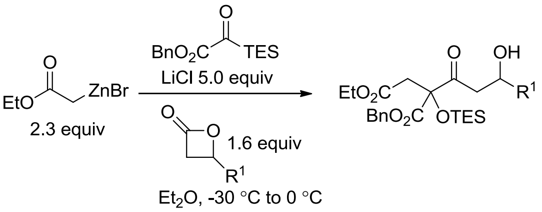


1-benzyl 4-ethyl 2-3-hydroxy-2-methyl-5-phenylpentanoyl)-2-((triethylsilyl)oxy)succinate (3j): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv) and *cis*-3-methyl-4-phenethyloxetan-2-one (**S7**, 55 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (50:50:0 to 0:100:0 to 0:95:5 hexanes: CH_2Cl_2: MeOH) provided the desired product as a light yellow oil with 5:1 diastereomeric ratio (79 mg, 79%). Analytical data: **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.35-7.14 (m, 10H), 5.16 (d, *J* = 12.0 Hz, 1H), 5.05 (d, *J* = 12.0 Hz, 1H), 4.23 (dd, *J* = 10.0, 2.0 Hz, 1H), 4.06 (q, *J* = 7.0 Hz, 2H), 3.61 (d, *J* = 17.0 Hz, 1H), 3.44-3.43 (m, 1H), 2.88-2.84 (m, 2H), 2.62-2.60 (m, 1H), 1.91-1.88 (m, 1H), 1.59-1.57 (m, 1H), 1.21 (t, *J* = 7.0 Hz, 3H), 1.00 (d, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 7.5 Hz, 9H), 0.57-0.51 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 213.2, 169.7, 168.9, 142.4, 134.3, 128.7, 128.6, 128.5, 128.3, 125.7, 84.2, 70.1, 68.1, 61.1, 45.9, 42.5, 36.0, 32.8, 14.1, 8.9, 6.7, 5.6; **LRMS (ESI$^+$)** Calcd. for $C_{31}H_{44}O_7Si$+Cs, 689.2; Found, 689.1; **IR** (thin film, cm^{-1}) 3440, 2955, 2877, 2247, 1735, 1642, 1455, 1374, 1343, 1210, 1022, 834, 733, 699; **TLC**(75:25 Hex:EtOAc): R_f = 0.34.

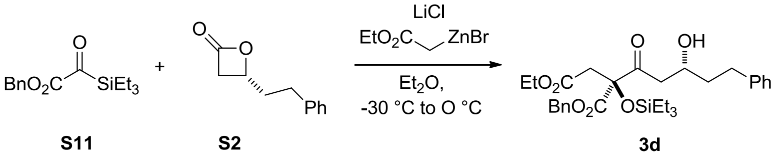


1-benzyl 4-ethyl 2-3-hydroxy-2-methyl-5-(trimethylsilyl)pent-4-ynoyl)-2-((triethylsilyl)oxy)succinate (3k): The title compound was prepared according to General Procedure A using silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv) and (3S,4S)-3-methyl-4-((trimethylsilyl)ethynyl)oxetan-2-one (**S8**, 56 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (92.5:7.5 hexanes: ethyl acetate) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (64 mg, 64%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.29 (m, 5H), 5.19 (d, *J* = 12.0 Hz, 1H), 5.13-5.12 (m, 1H), 5.07 (d, *J* = 12.0 Hz, 1H), 4.09 (q, *J* = 7.2 Hz, 2H), 3.62-3.59 (m, 1H), 3.61 (d, *J* = 21.6 Hz, 1H), 3.19 (d, *J* = 4.4 Hz, 1H), 2.89 (d, *J* = 17.2 Hz, 1H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.20 (d, *J* = 7.2 Hz, 3H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.63-0.56 (m, 6H), 0.16 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 211.7, 170.0, 168.6, 134.3, 128.8, 128.6, 104.2, 89.3, 84.2, 68.2, 63.3, 61.4, 47.2, 42.6, 14.0, 9.7, 6.7, 5.6, -0.2; **LRMS (ESI$^+$)** Calcd. for $C_{28}H_{44}O_7Si_2$+Na, 571.3; Found, 571.2; **IR** (thin film, cm^{-1}) 3543, 2959, 2878, 2361, 2178, 1731, 1374, 1212, 1020, 844; **TLC**(75:25 Hex:EtOAc): R_f = 0.45.

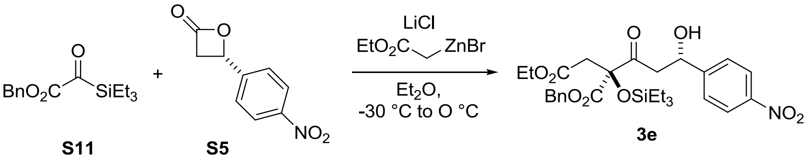
General Procedure B for the Reformatsky Initiated Cascade Coupling of Silyl Glyoxylate and β-Lactones Affording β-hydroxyketones 3d-g



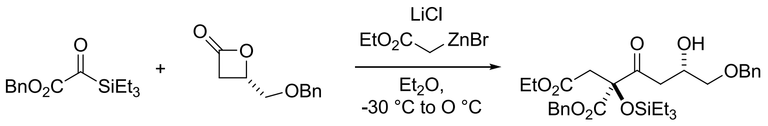
An oven-dried 20-mL scintillation vial equipped with magnetic stir bar was charged with LiCl (0.0-0.9 mmol, 0-5 equiv). The vial was purged with nitrogen, and a solution of Reformatsky reagent (0.38 M, 1.09 mL, 2.3 equiv) was added. The resulting suspension was diluted with diethyl ether (1.0 mL) and cooled to -30 °C in an acetone/dry ice bath (bath temperature, monitored with a thermocouple probe). A second oven-dried vial was charged with silyl glyoxylate **S11** (0.18 mmol, 1.0 equiv) and β-lactone **S2-S5** (0.29 mmol, 1.6 equiv). The vial was purged with nitrogen, and diethyl ether (1.5 mL) was added. The resulting solution was cooled and added to the solution of Reformatsky reagent. Additional diethyl ether (0.5 mL) was used to rinse the vial. The reaction was allowed to warm slowly in the acetone bath (generally over 30 min from -30 °C to 0 °C). Consumption of the silyl glyoxylate was generally observed by TLC analysis and disappearance of yellow color between -30 °C and -25 °C. The reaction was then held at 0 °C until judged complete by TLC analysis, generally 30 min. Saturated aqueous ammonium chloride (0.5 mL) was then added to quench the reaction, and it was stirred until clear layers were observed. The organic layer was removed, and the aqueous layer was extracted with diethyl ether (3x1.0 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The crude product was purified via flash chromatography to give the desired product.



1-benzyl 4-ethyl 2-(3-hydroxy-5-phenylpentanoyl)-2-((triethylsilyl)oxy)succinate (3d): The title compound was prepared according to General Procedure B using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and (*S*)-4-phenethyloxetan-2-one (**S2**, 50 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (CH_2Cl_2) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (63 mg, 65%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36-7.16 (m, 10H), 5.19 (d, *J* = 12.0 Hz, 1H), 5.10 (d, *J* = 12.4 Hz, 1H), 4.13 (s, 1H), 4.08 (q, *J* = 7.2 Hz, 2H), 3.48 (d, *J* = 16.8 Hz, 1H), 3.17 (dd, *J* = 18.0, 1.6 Hz, 1H), 3.05 (d, *J* = 1.2 Hz, 1H), 2.90 (d, *J* = 16.8 Hz, 1H), 2.87-2.57 (m, 3H), 1.90-1.51 (m, 2H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 8.0 Hz, 9H), 0.55 (q, *J* = 7.6 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 210.7, 169.3, 168.7, 142.1, 134.3, 128.7, 128.6, 128.5, 128.4, 128.3, 125.7, 83.8, 68.1, 66.9, 61.0, 45.3, 42.4, 38.3, 31.9, 14.0, 6.7, 5.7; **LRMS (ESI$^+$)** Calcd. for $C_{30}H_{42}O_7Si$+Na, 565.3; Found, 565.2; **IR** (thin film, cm^{-1}) 3459, 3028, 2955, 2877, 2360, 1737, 1455, 1373, 1213; **TLC**(75:25 Hex:EtOAc): R_f = 0.44.

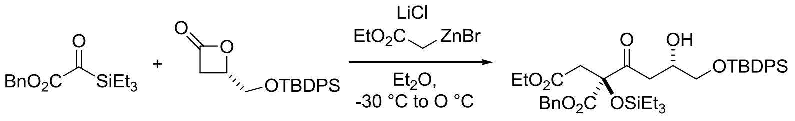


1-benzyl 4-ethyl 2-(3-hydroxy-3-(4-nitrophenyl)propanoyl)-2-((triethylsilyl)oxy)succinate (3e): The title compound was prepared according to General Procedure B using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and (*S*)-4-(4-nitrophenyl)oxetan-2-one (**S5**, 56 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (93.5:7.5 to 85:15 petroleum ether: diethyl ether) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (35 mg, 35%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 8.8 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.39-7.30 (m, 5H), 5.30 (d, *J* = 9.6 Hz, 1H), 5.20 (d, *J* = 12.4 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 3.57 (s, 1H), 3.54 (d, *J* = 13.2 Hz, 1H), 3.44 (dd, *J* = 18.0, 2.4 Hz, 1H), 2.95 (d, *J* = 17.2 Hz, 1H), 2.92 (d, *J* = 18.0 Hz, 1H), 1.25 (t, *J* = 7.2 Hz, 3H), 0.86 (t, *J* = 8.0 Hz, 9H), 0.58-0.52 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 210.1, 169.5, 168.5, 150.4, 147.1, 134.2, 128.8, 128.7, 128.5, 126.4, 123.6, 83.7, 69.0, 68.2, 61.2, 47.0, 42.5, 14.1, 6.7, 5.6; **LRMS (ESI$^+$)** Calcd. for $C_{28}H_{37}NO_9Si$+Na, 582.2; Found, 582.2; **IR** (thin film, cm^{-1}) 3502, 2057, 2877, 2360, 2341, 1732, 1606, 1521, 1347, 1214, 1009, 854; **TLC**(75:25 Hex:EtOAc): R_f = 0.36.



S11 **S3** **3f**

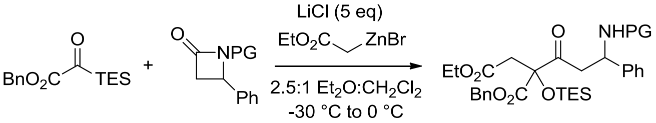
1-benzyl 4-ethyl 2-(4-(benzyloxy)-3-hydroxybutanoyl)-2-((triethylsilyl)oxy)succinate (3f): The title compound was prepared according to General Procedure B using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and (*S*)-4-((benzyloxy)methyl)oxetan-2-one (**S3**, 56 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (85:15 to 75:25 petroleum ether: diethyl ether) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (49 mg, 49%). Analytical data: ^{1}**H NMR** (400 MHz, CDCl$_3$): δ 7.34-7.27 (m, 10H), 5.18 (d, *J* = 12.0 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 4.57 (s, 2H), 4.37-4.31 (m, 1H), 4.07 (q, *J* = 7.2 Hz, 2H), 3.55-3.43 (m, 2H), 3.47 (d, *J* = 22.0 Hz, 1H), 3.19 (dd, *J* = 20.0, 4.0 Hz, 1H), 2.93 (d, *J* = 18.4 Hz, 2H), 2.91 (d, *J* = 16.8 Hz, 1H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.89 (t, *J* = 8.0 Hz, 9H), 0.57 (q, *J* = 7.6 Hz, 6H); ^{13}**C NMR** (125 MHz, CDCl$_3$)**:** δ 209.4, 169.2, 168.7, 138.1, 134.4, 128.6, 128.5, 128.4, 128.3, 127.6, 127.5, 83.7, 73.3, 73.2, 68.0, 66.6, 60.9, 42.4, 41.8, 14.0, 6.7, 5.7; **LRMS (ESI$^+$)** Calcd. for $C_{30}H_{42}O_8Si$+Na, 581.3; Found, 581.2; **IR** (thin film, cm^{-1}) 3459, 3065, 3032, 2955, 2876, 2360, 1737, 1455, 1213, 1117, 1021, 736; **TLC**(75:25 Hex:EtOAc): R_f = 0.29.



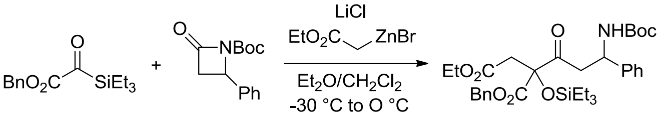
S11 **S4** **3g**

1-benzyl 4-ethyl 2-(4-((*tert*-butyldiphenylsilyl)oxy)-3-hydroxybutanoyl)-2-((triethylsilyl)oxy)succinate (3g): The title compound was prepared according to General Procedure B using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and (*S*)-4-(((tert-butyldiphenylsilyl)oxy)methyl)oxetan-2-one (**S4**, 98 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (93.5:7.5 to 85:15 petroleum ether: diethyl ether) provided the desired product as a light yellow oil with > 20:1 diastereomeric ratio (77 mg, 60%). Analytical data: ^{1}**H NMR** (500 MHz, CDCl$_3$): δ 7.68-7.66 (m, 4H), 7.49-7.29 (m, 11H), 5.18 (d, *J* = 12.0 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 4.27-4.25 (m, 1H), 4.08-4.03 (m, 2H), 3.66-3.65 (m, 2H), 3.47 (d, *J* = 17.0 Hz, 1H), 3.25 (dd, *J* = 18.0, 3.0 Hz, 1H), 2.97-2.88 (m, 3H), 1.19 (t, *J* = 7.0 Hz, 3H), 1.06 (s, 9H), 0.89 (t, *J* = 8.0 Hz, 9H), 0.59-0.55 (m, 6H); ^{13}**C NMR** (125 MHz, CDCl$_3$)**:** δ 209.4, 169.2, 168.8, 135.6, 135.5, 134.5, 133.3, 133.2, 129.7, 128.6, 128.5, 128.4, 127.7, 127.6, 83.8, 68.0, 67.9, 67.0, 60.9, 42.4, 41.8, 26.8, 19.2, 14.1, 6.8, 5.7; **LRMS (ESI$^+$)** Calcd. for $C_{39}H_{54}O_8Si_2$+Na, 729.3; Found, 729.3; **IR** (thin film, cm^{-1}) 3458, 2957, 2877, 2360, 2341, 1738, 1456, 1428, 1213, 1113, 740, 700; **TLC**(75:25 Hex:EtOAc): R_f = 0.42.

General Procedure C for the Reformatsky Initiated Cascade Coupling of Silyl Glyoxylate and β-Lactams Affording β-aminoketones 3l-m

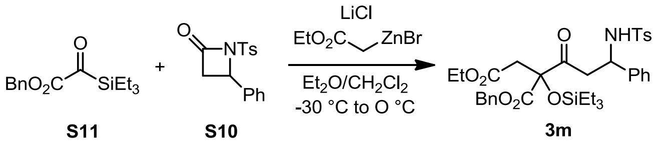


To an oven-dried 20-mL scintillation vial was added LiCl (38 mg, 0.9 mmol, 5 equiv). The vial was purged with nitrogen, and a solution of Reformatsky reagent (0.38 M, 1.09 mL, 2.3 equiv) was added. The resulting suspension was diluted with diethyl ether (1.0 mL) and cooled to -30 °C in an acetone/dry ice bath (bath temperature, monitored with a thermocouple probe). A second oven-dried vial was charged with silyl glyoxylate **S11** (50 mg, 0.18 mmol, 1.0 equiv) and purged with nitrogen, and diethyl ether (0.5 mL) was added. The resulting solution was added to the solution of Reformatsky reagent. The reaction was allowed to warm slowly in the acetone bath. Once consumption of the silyl glyoxylate was observed by TLC analysis and disappearance of yellow color, generally between -30 °C and -25 °C, a cooled solution of the β-Lactam (**S9-S10**, 0.29 mmol, 1.6 equiv) in CH_2Cl_2 (1.0 mL) was added. The reaction was then allowed to warm to 0 °C, where it was held until judged complete by TLC analysis, generally 30 min. Saturated ammonium chloride (0.5 mL) was then added to quench the reaction, and it was stirred until clear layers were observed. The organic layer was removed, and the aqueous layer was extracted with diethyl ether (3x1.0 mL). The combined organic extracts were washed with brine, dried with magnesium sulfate, and concentrated *in vacuo*. The crude product was purified via flash chromatography to give the desired product.



S11 **S9** **3l**

1-benzyl 4-ethyl 2-(3-((*tert*-butoxycarbonyl)amino)-3-phenylpropanoyl)-2-((triethylsilyl)oxy)succinate (3l): The title compound was prepared according to General Procedure C using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and tert-butyl 2-oxo-4-phenylazetidine-1-carboxylate (**S9**, 71 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (92.5:7.5 hexanes: ethyl acetate) provided the desired product as a light yellow oil (62 mg, 56%), which was a 1:1 mixture of separable diastereomers. Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): (Diastereomer 1) δ 7.35-7.19 (m, 10H), 5.52 (br s, 1H), 5.11 (br s, 1H), 5.05-4.90 (m, 2H), 4.05 (q, *J* = 7.2 Hz, 2H), 3.47-3.28 (m, 3H), 2.86 (d, *J* = 16.8 Hz, 1H), 1.41 (s, 9H), 1.20 (t, *J* = 6.8 Hz, 3H), 0.88 (t, *J* = 8.0 Hz, 9H), 0.55-0.51 (m, 6H); (Diastereomer 2) δ 7.35-7.20 (m, 10H), 5.44 (br s, 1H), 5.10-5.02 (m, 2H), 4.10-4.08 (m, 2H), 3.54-3.41 (m, 2H), 3.05-2.93 (m, 1H), 2.87 (d, *J* = 20.0 Hz, 1H), 1.38 (br s, 9H), 1.22 (t, *J* = 7.2 Hz, 3H), 0.86 (t, *J* = 8.0 Hz, 9H), 0.56-0.54 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$), major diastereomer**:** δ 207.0, 168.9, 168.6, 155.2, 134.4, 128.6, 128.5, 128.3, 126.8, 126.1, 83.8, 79.3, 68.0, 60.9, 50.1, 43.2, 42.0, 28.3, 14.0, 6.8, 5.7; **LRMS (ESI$^+$)** Calcd. for $C_{33}H_{47}NO_8Si$+Na, 636.3; Found, 636.3; **IR** (thin film, cm^{-1}) 3417, 2959, 2877, 2086, 1641, 1495, 1455, 1367, 1343, 1167, 1018, 734, 698; **TLC**(75:25 Hex:EtOAc): R_f = 0.34.

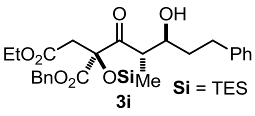


1-benzyl 4-ethyl 2-(3-(4-methylphenylsulfonamido)-3-phenylpropanoyl)-2-((triethylsilyl)oxy)succinate (3m): The title compound was prepared according to General Procedure C using LiCl (38 mg, 0.9 mmol, 5 equiv), silyl glyoxylate (**S11**, 50 mg, 0.18 mmol, 1.0 equiv), and 4-phenyl-1-tosylazetidin-2-one (**S10**, 87 mg, 0.29 mmol, 1.6 equiv). Purification via flash chromatography (CH_2Cl_2) provided the desired product as a light yellow oil (64 mg, 53%), which was a 1:1 mixture of separable diastereomers. Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): (Diastereomer 1) δ 7.56 (d, *J* = 8.0 Hz, 2H), 7.33-7.31 (m, 3H), 7.22-7.12 (m, 9H), 5.56 (d, *J* = 6.4 Hz, 1H), 4.99 (d, *J* = 12.4 Hz, 1H), 4.94 (d, *J* = 12.0 Hz, 1H), 4.68 (q, *J* = 6.0 Hz, 1H), 4.03 (q, *J* = 6.8 Hz, 2H), 3.37 (dd, *J* = 18.8, 6.4 Hz, 1H), 3.31 (d, *J* = 16.8 Hz, 1H), 3.18 (dd, *J* = 18.8, 4.8 Hz, 1H), 2.81 (d, *J* = 16.8 Hz, 1H), 2.34 (s, 3H), 1.18 (t, *J* = 7.2 Hz, 3H), 0.82 (t, *J* = 7.6 Hz, 9H), 0.48 (q, *J* = 8.0 Hz, 6H); (Diastereomer 2) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.32-7.30 (m, 3H), 7.22-7.12 (m, 9H), 5.58 (d, *J* = 3.6 Hz, 1H), 5.05-4.95 (m, 2H), 4.71-4.66 (m, 1H), 4.10 (q, *J* = 6.8 Hz, 2H), 3.60 (dd, *J* = 18.8, 8.8 Hz, 1H), 3.38 (d, *J* = 17.2 Hz, 1H), 2.88-2.79 (m, 2H), 2.34 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H), 0.81 (t, *J* = 7.6 Hz, 9H), 0.47 (q, *J* = 7.6 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$), mixture of diastereomers**:** δ 206.7, 206.5, 169.7, 169.0, 168.5, 142.9, 142.8, 140.7, 140.1, 134.4, 129.3, 129.1, 128.7, 128.6, 128.5, 128.3, 128.2, 127.4, 127.3, 126.9, 126.7, 83.8, 83.5, 68.1, 68.0, 61.4, 61.0, 53.7, 53.4, 44.8, 44.1, 42.8, 42.1, 21.4, 14.0, 6.7, 6.6, 5.8, 5.7; **LRMS (ESI$^+$)** Calcd. for $C_{35}H_{45}NO_8SSi$+Na, 690.3; Found, 690.2; **IR** (thin film, cm^{-1}) 3436, 3033, 2957, 2877, 1737, 1455, 1372, 1333, 1213, 1160, 1020, 739, 699, 666; **TLC**(75:25 Hex:EtOAc): R_f = 0.22.

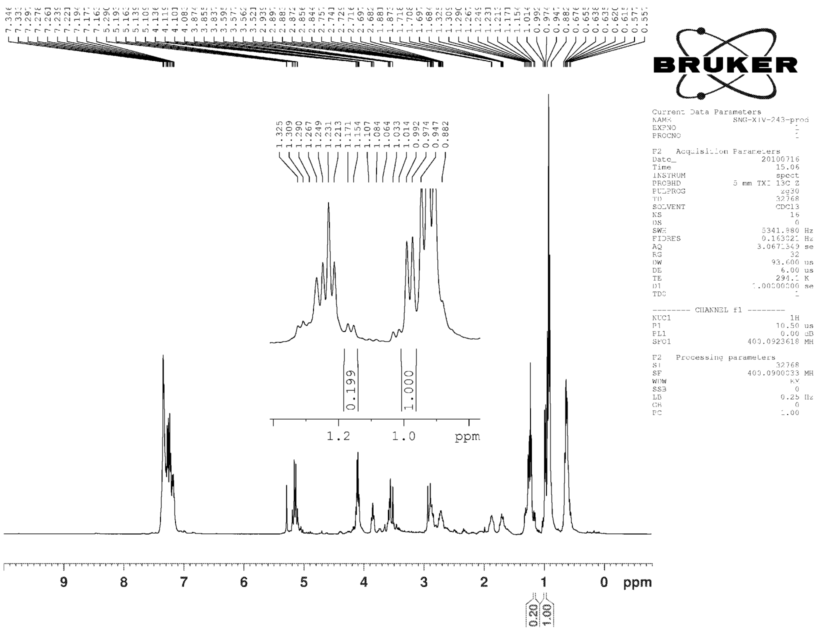
Stereochemical Analysis of β-hydroxyketone Products:

Calculation of Diastereomeric Ratios:

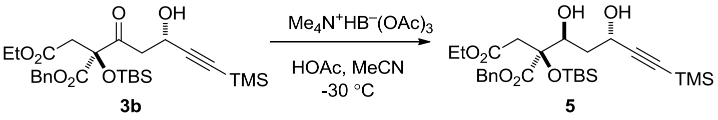
A sample calculation of diastereomeric ratio is shown at right for **3i**. The relative integration of the methyl groups of the two diastereomers in the crude reaction mixture is shown to be 5:1.



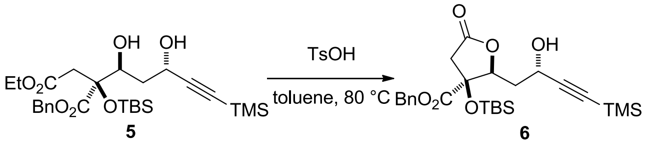
3i



Determination of Relative Stereochemistry:

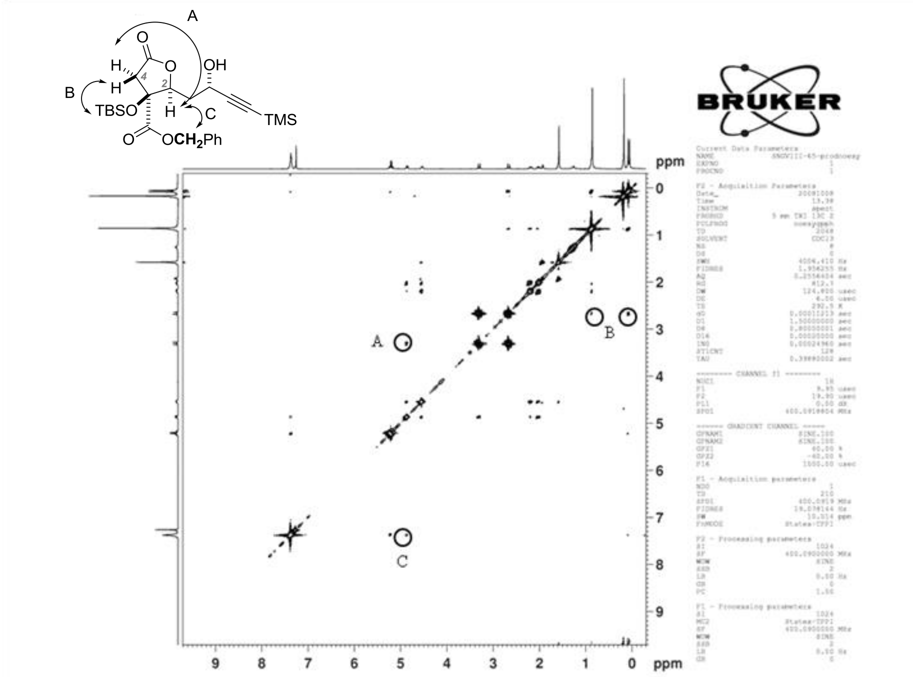


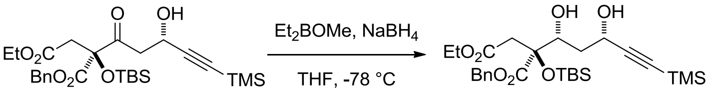
(*S*)-1-benzyl 4-ethyl 2-((*tert*-butyldimethylsilyl)oxy)-2-((1*S*,3*S*)-1,3-dihydroxy-5-(trimethylsilyl) pent-4-yn-1-yl)succinate (5): An oven-dried 20-mL scintillation vial equipped with a magnetic stir bar was charged with MeCN (1 mL), $Me_4NHB(OAc)_3$ (147 mg, 0.56 mmol, 5.0 equiv), and dry HOAc (0.45 mL). The resulting solution was cooled to -35 °C in a Cryocool apparatus. A solution of ketone **3b** (46 mg, 0.112 mmol, 1.0 equiv) in MeCN (1 mL) was added to the reaction dropwise, and additional MeCN (0.5 mL) was used to rinse the vial. The reaction was allowed to warm to -25 °C and was maintained at the same temperature for 60 h. The reaction was quenched by the addition of a 25% saturated aqueous solution of sodium potassium tartrate (0.3 mL) and was allowed to warm slowly to room temperature. A saturated aqueous solution of $NaHCO_3$ was added until the pH of the reaction was neutral. The resulting suspension was extracted with CH_2Cl_2. The combined organic extracts were washed with saturated aqueous $NaHCO_3$, dried with $MgSO_4$, and concentrated *in vacuo* to afford a colorless oil. The material was purified via column chromatography, eluting with 80:20 hexanes:EtOAc, to give the title compound as a viscous light yellow oil with >25:1 diastereomeric ratio. Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.43-7.26 (m, 5H), 5.22 (d, *J* = 12.0 Hz, 1H), 5.18 (d, *J* = 12.4 Hz, 1H), 4.61 (br. s, 1H), 4.32 (dd, *J* = 9.2, 8.4 Hz, 1H), 4.06 (q, *J* = 7.2 Hz, 2H), 3.13 (d, *J* = 7.2 Hz, 1H), 3.01 (d *J* = 7.2 Hz, 1H), 2.93 (d, *J* = 15.2 Hz, 1H), 2.86 (d, *J* = 15.2 Hz, 1H), 1.88 (ddd, *J* = 17.6, 14.4, 3.2 Hz, 1H), 1.67 (dd, *J* = 14.4, 6.4 Hz, 1H), 1.20 (t, *J* = 7.2 Hz, 3H) 0.85 (s, 9H), 0.16 (s, 9H), 0.15 (s, 3H), 0.09 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$)**:** δ 172.0, 170.3, 135.3, 128.7, 128.6, 128.5, 106.2, 89.9, 80.7, 73.6, 67.6, 61.0, 60.9, 41.7, 37.8, 25.9, 18.7, 14.0, -0.1, -2.9, -3.1; **LRMS (ESI$^+$)** Calcd. for $C_{27}H_{44}O_7Si_2$+Na, 559.2; Found, 559.2; **IR** (thin film, cm^{-1}): 3853, 2089, 1647, 1541, 1457, 1250, 1175, 1031, 521, 509, 496; **TLC**(80:20 Hexanes:EtOAc): R_f = 0.38.



(2*S*,3*S*)-benzyl 3-((*tert*-butyldimethylsilyl)oxy)-2-((*S*)-2-hydroxy-4-(trimethylsilyl)but-3-yn-1-yl)-5-oxotetrahydrofuran-3-carboxylate (6): An oven-dried and cooled vial equipped with a magnetic stir bar was charged with diol **5** (12 mg, 0.022 mmol) and toluene (0.75 mL). TsOH (cat.) was added, and the vial was sealed with a Teflon-lined cap. The solution was heated to 80 °C in a sand bath for 1 h. After cooling to rt, the solvent was removed *in vacuo*, and the crude residue was purified via column chromatography, eluting with 80:20 hexanes:EtOAc. The title compound was obtained as a colorless oil (5 mg, 45%). Analytical data: $[\alpha]_D^{25.4}$ -17.3 (*c* 0.23, $CHCl_3$); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.37 (br. s., 5H), 5.23 (d, *J* = 12.0 Hz, 1H), 5.18 (d, *J* = 12.0 Hz, 1H), 4.86 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.53 (br. s., 1H), 3.30 (d, J = 17.5 Hz, 1H), 2.66 (d, *J* = 17.0 Hz, 1H), 2.187 (ddd, *J* = 14.5, 9.5, 3.5 Hz, 1H), 2.00 (ddd, *J* = 12.5, 9.0, 3.0 Hz, 1H), 1.92 (d, *J* = 5.5, 1H) 0.85 (s, 9H), 0.17 (s, 9H), 0.08 (s, 3H), 0.04 (s, 3H); **^{13}C NMR** (100 MHz, CDCl$_3$)**:** δ 172.7, 170.1, 128.8, 90.4, 83.3, 81.0, 68.3, 59.4, 42.6, 36.8, 25.6, -0.2, -3.5, -3.7; **LRMS (ESI$^+$)** Calcd. for $C_{25}H_{38}O_6Si_2$+Na, 513.2; Found, 513.2; **IR** (thin film, cm^{-1}) 3433, 3021, 2961, 2330, 2089, 1646, 1361, 1215, 775, 668; **TLC**(80:20 Hexanes:EtOAc): R$_f$ = 0.40.

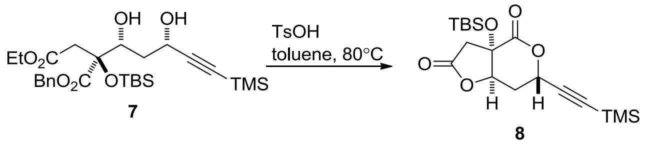
Spectral analysis (NOESY) supported the structural assignment shown for **6**: A strong nOe was observed between the C2 methine C–**H** and the α-C–**H** at C4 as well as between the C2 methine C–**H** and the CO$_2$**CH$_2$**Ph benzyl protons (interactions A and C, respectively). Additionally, an nOe was observed between the β-C–**H** at C4 and the methyl and *tert*-butyl substituents of the TBS ether, which suggested their relative *syn* orientation (interaction B).





3b **7**

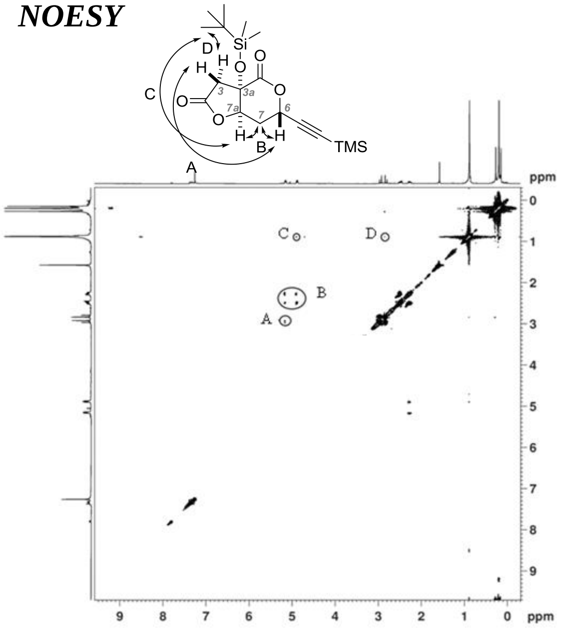
(*S*)-1-benzyl 4-ethyl 2-((*tert*-butyldimethylsilyl)oxy)-2-((1*R*,3*S*)-1,3-dihydroxy-5-(trimethylsilyl)pent-4-yn-1-yl)succinate (7): The title compound was prepared analogously to **7′** (*vide infra*). Yield = 95%, > 25:1 dr. Analytical data: **^{1}H NMR** (400 MHz, CDCl$_3$): δ 7.34 (br. s, 5H), 5.19 (s, 2H), 4.58 (br. s, 1H), 4.09-3.95 (m, 1H), 4.07 (t, *J* = 7.6 Hz, 2H), 2.96 (br. s, 1H), 2.72 (d *J* = 15.2 Hz, 1H), 1.95 (dd, *J* = 13.6, 6.4 Hz, 1H), 1.77 (dd, *J* = 17.6, 10 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H), 0.85 (s, 9H), 0.17 (s, 3H), 0.14 (s, 9H), 0.10 (s, 3H); **^{13}C NMR** (100 MHz, CDCl$_3$)**:** δ 172.0, 169.8, 135.0, 128.5, 128.5, 128.4, 105.7, 89.7, 80.8, 75.2, 67.5, 61.7, 60.8, 41.3, 38.7, 25.9, 18.7, 13.9, -0.3, -2.9, -3.2; **LRMS (ESI$^+$)** Calcd. for $C_{27}H_{44}O_7Si_2$+Na, 559.3; Found, 559.2; **IR** (thin film, cm^{-1}): 3436, 2957, 2856, 2360, 1739, 1637, 1457, 1372, 1251, 1188, 1112, 840; **TLC**(80:20 Hexanes:EtOAc): R$_f$ = 0.38.



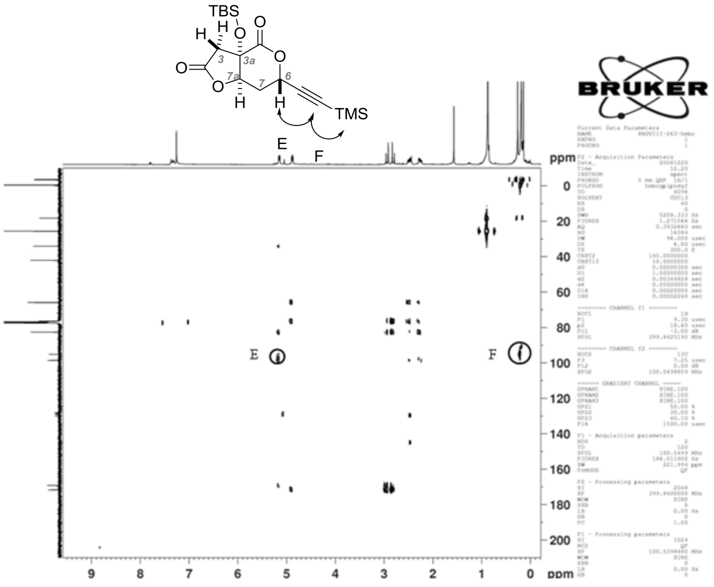
(3a*S*,6*S*,7a*R*)-3a-((*tert*-butyldimethylsilyl)oxy)-6-((trimethylsilyl)ethynyl)tetrahydro-2H-furo[3,2-c]pyran-2,4(6H)-dione (8): An oven-dried and cooled vial equipped with a magnetic stir bar was charged with diol **7** (15 mg, 0.0275 mmol) and toluene (1 mL). TsOH (cat.) was added, and the vial was sealed with a Teflon cap. The solution was heated to 80 °C in a sand bath for 1 h. After cooling to rt, the solvent was removed *in vacuo*, and the crude residue was purified via column chromatography, eluting with 10:90 to 20:80 EtOAc: hexanes. The title compound was obtained as a colorless oil (6.4 mg, 60%). Analytical data: $[\alpha]_D^{25.1}$ -13.1 (*c* 0.15, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.15 (dd, *J* = 7.2, 3.6 Hz, 1H), 4.89 (dd, *J* = 6.8, 4.8 Hz, 1H), 2.94 (d, *J* = 18.4 Hz, 1H), 2.81 (d, *J* = 18.0 Hz, 1H), 2.48 (ddd, *J* = 12.4, 7.6, 4.8 Hz, 1H), 2.26 (ddd, *J* = 10.4, 7.2, 3.6 Hz, 1H), 0.88 (s, 9H), 0.26 (s, 3H), 0.19 (s, 9H), 0.14 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$)**:** δ 171.6, 169.1, 98.5, 95.1, 82.6, 76.3, 65.8, 42.0, 34.0, 25.5, 18.1, -0.5, -3.5, -3.7; **LRMS (ESI$^+$)** Calcd. for $C_{18}H_{30}O_5Si_2$+Na, 405.2; Found, 405.2; **IR** (thin film, cm^{-1}) 3433, 3019, 1645, 1215, 771, 669; **TLC**(80:20 Hexanes:EtOAc): R_f = 0.50.

Spectral analysis (HMBC, NOESY) supported the stereochemical assignment shown for compound **8**: The NOESY spectrum shows an nOe between the methine C–H at C6 and the β-C–H at C3 (interaction A), which suggests their orientation on the concave face of the bicycle. Additionally, both the α-C3 C–**H** proton on the convex face of the bicycle and the C7a methine C–**H** show an nOe with the *tert*-butyl group of the TBS ether, which suggests their mutual orientation on the convex face of the molecule (interactions D and C). The assignment of the C7a and C6 methine C–**H** protons was a result of the observation of a mutual correlation between the latter and the TMS methyl groups with the distal alkyne carbon (indicated by interactions E and F) in the HMBC spectrum.

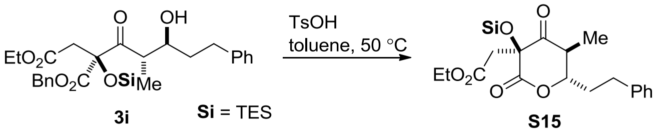
NOESY



HMBC



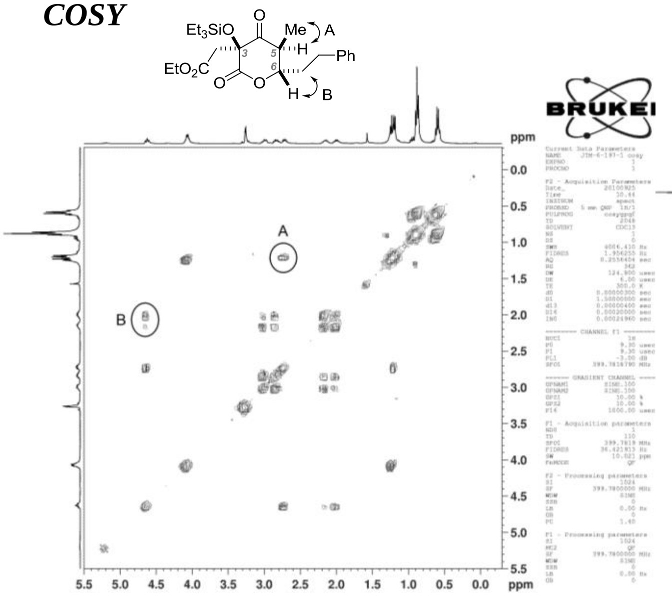
*Determination of Relative Stereochemistry for Coupling Product **3i**:*



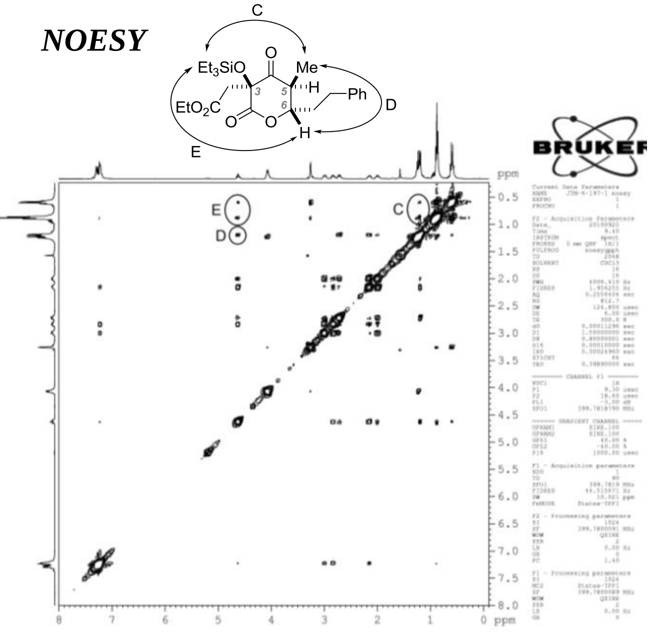
Ethyl 2-((3*S*,5*S*,6*S*)-5-methyl-2,4-dioxo-6-phenethyl-3-((triethylsilyl)oxy)tetrahydro-2H-pyran-3-yl)acetate (S15): An oven-dried and cooled vial equipped with a magnetic stir bar was charged with **3i** (26 mg, 0.0467 mmol) and toluene (1 mL). TsOH (cat.) was added, and the vial was sealed with a Teflon-lined cap. The solution was heated to 50 °C in an oil bath for 3 h. After cooling to rt, the solvent was removed *in vacuo*, and the crude residue was purified via column chromatography, eluting with 7.5:92.5 EtOAc: hexanes. The title compound was obtained as a pale yellow oil (14 mg, 67%). Analytical data: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.33-7.19 (m, 5H), 4.66-4.60 (m, 1H), 4.11-4.03 (m, 2H), 3.29 (d, J = 16.4 Hz, 1H), 3.24 (d, J = 16.0 Hz, 1H), 3.05-2.95 (m, 1H), 2.88-2.78 (m, 1H), 2.77-2.67 (m, 1H), 2.21-2.11 (m, 1H), 2.05-1.94 (m, 1H), 1.23 (t, J = 7.2 Hz, 3H), 1.19 (d, J = 6.8 Hz, 3H), 0.88 (t, J = 8.0 Hz, 9H), 0.59 (q, J = 8.0 Hz, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 202.2, 170.7, 169.4, 140.7, 128.6, 128.5, 126.2, 75.4, 61.2, 45.6, 38.0, 34.4, 30.4, 14.0, 10.8, 6.6, 5.9; **LRMS (ESI$^+$)** Calcd. for $C_{18}H_{21}O_6Si$+Cs, 581.1; Found, 581.1; **IR** (thin film, cm^{-1}) 3433, 2959, 2878, 2092, 1760, 1726, 1637, 1456, 1373, 1339, 1309, 1212, 1103, 1027, 842, 700; **TLC**(75:25 Hexanes:EtOAc): R_f = 0.60.

Spectral analysis (COSY, NOESY) supported the stereochemical assignment shown for compound **S15**. The identities of the two methine protons (C5 and C6) were determined through correlation spectroscopy. A strong correlation was observed between the methyl group and methine proton at C5 (interaction A), while the methine proton at C6 showed a strong correlation with the methylene group of the hydrocinnamyl substituent (interaction B). The relative stereochemistry was assigned through NOESY analysis, which showed nOe's between the triethylsilyloxy group at C3 and both the methyl group at C5 and the methine proton at C6 (interactions C and E). A third nOe was observed between the C5 methyl group and the methine proton at C6, suggesting that the trans relationship of the original β-lactone substituents (interaction D) was preserved under the reaction conditions.

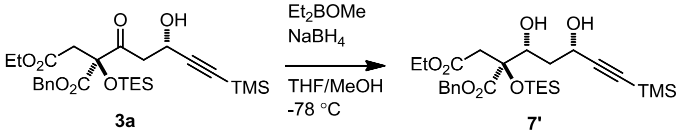
COSY



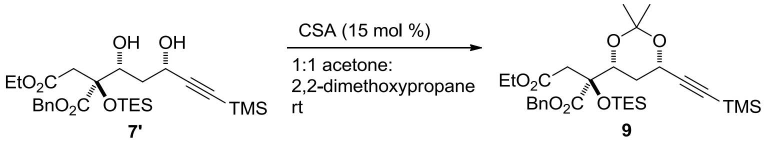
NOESY



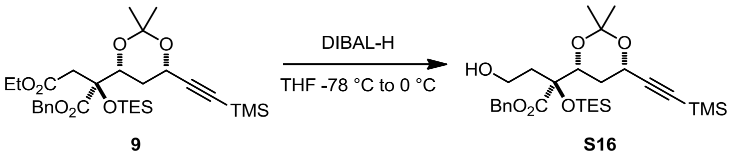
Diester Functionalization of β-hydroxyketone Products:



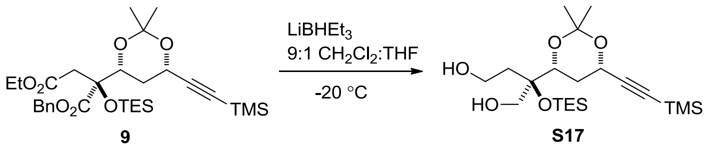
(*S*)-1-benzyl 4-ethyl 2-((1*R*,3*S*)-1,3-dihydroxy-5-(trimethylsilyl)pent-4-yn-1-yl)-2-((triethylsilyl)oxy)succinate (7′): A flame-dried and N_2-purged 500-mL round-bottomed flask was charged with ketone **3a** (6.0 g, 11.2 mmol, 1.0 equiv). Tetrahydrofuran (200 mL) and methanol (50 mL) were added. The solution was cooled to -78 °C (acetone/dry ice), and diethylmethoxyborane (1 M in tetrahydrofuran, 14.6 mL, 14.6 mmol) was added dropwise. After stirring for 45 minutes at -78 °C, sodium borohydride (1.27 g, 33.7 mmol, 3.0 equiv) was added in one portion and the reaction was maintained at the same temperature. Once TLC analysis indicated complete consumption of the starting material (3.5 h), the reaction was quenched with acetic acid (9.0 mL). After warming to room temperature, the reaction was stirred for 1.5 h and was then concentrated *in vacuo*. Methanol (30 mL) was added, and the solution was again concentrated *in vacuo*; this procedure was repeated with four additional portions of methanol (30 mL). The residue was partitioned between ethyl acetate and saturated sodium bicarbonate, and the organic layer was washed with saturated sodium bicarbonate, water, and brine. The organic extracts were dried with magnesium sulfate and concentrated *in vacuo* to give a light yellow viscous oil (5.7 g, 95%) that was used without additional purification. Analytical data**:** $[\alpha]_D^{25.2}$ -1.74 (*c* 1.25, $CHCl_3$); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.36-7.33 (m, 5H), 5.20 (s, 2H), 4.60 (br. s., 1H), 4.10-3.99 (m, 3H), 3.05 (d, *J* = 6.0 Hz, 1H), 2.87 (d, *J* = 4.0 Hz, 1H), 2.83 (d, *J* = 9.0 Hz, 2H), 1.98 (dd, *J* = 13.5, 5.5 Hz, 1H), (dd, *J* = 10.5, 2.5 Hz, 1H), 1.21 (t, *J* = 7.0 Hz, 3H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.67-0.62 (m, 6H), 0.15 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$)**:** δ 172.1, 169.9, 135.0, 128.6, 128.5, 128.4, 105.6, 89.7, 80.6, 75.3, 67.6, 62.1, 60.9, 41.2, 38.7, 14.0, 7.1, 6.4, -0.2; **LRMS (ESI$^+$)** Calcd. for $C_{27}H_{44}O_7Si_2$+Na, 559.3; Found, 559.3; **IR** (thin film, cm^{-1}) 3470, 2957, 2876, 2172, 1740, 1185, 1022, 844, 734; **TLC**(80:20 Hex:EtOAc): R_f = 0.21.



(*S*)-1-benzyl-4-ethyl-2-((4*R*,6*S*)-2,2-dimethyl-6-((trimethylsilyl)ethynyl)-1,3-dioxan-4-yl)-2-((triethylsilyl)oxy) succinate (9) A 500-mL round-bottomed flask was charged with diol **7′** (11.0 g, 20.6 mmol, 1.0 equiv), acetone (250 mL) and 2,2-dimethoxypropane (250 mL). CSA (0.716 g, 3.09 mmol, 0.15 equiv) was added, and the reaction was allowed to stir at room temperature for 16 h. The reaction was quenched by the addition of 0.5 mL of triethylamine and was concentrated *in vacuo*. The residue was purified via column chromatography (90:10 hexanes: ethyl acetate) to give the product as a white solid (8.6 g, 73%). Analytical data: $[\alpha]_D^{25.4}$ -13.96 (*c* 1.5, $CHCl_3$); melting point: 75-79 °C**; ^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.35-7.33 (m, 5H), 5.30 (d, *J* = 12.5 Hz, 1H), 5.05 (d, *J* = 12.0 Hz, 1H), 4.62 (dd, *J* = 12.0, 2.5 Hz, 1H), 4.12-4.06 (m, 3H), 2.74 (d, *J* = 14.5 Hz, 1H), 2.65 (d, *J* = 14.0 Hz, 1H), 1.88-1.60 (m, 2H), 1.33 (s, 3H), 1.23 (t, *J* = 7.5 Hz, 3H), 1.19 (s, 3H), 0.95 (t, *J* = 16.0 Hz, 9H), 0.74-0.65 (m, 6H), 0.17 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.6, 169.4, 135.6, 128.7, 128.4, 128.3, 103.7, 99.6, 89.4, 80.2, 72.9, 67.0, 60.7, 60.5, 41.9, 30.8, 29.5, 18.8, 14.0, 7.3, 6.7, -0.21; **LRMS (ESI$^+$)** Calcd. for $C_{30}H_{48}O_7Si_2$+Na, 599.3; Found, 599.3; **IR** (thin film, cm^{-1}) 2956, 2875, 2181, 1739, 1457, 1379, 1251, 1163, 844, 734; **TLC**(80:20 Hex:EtOAc): R_f = 0.30.

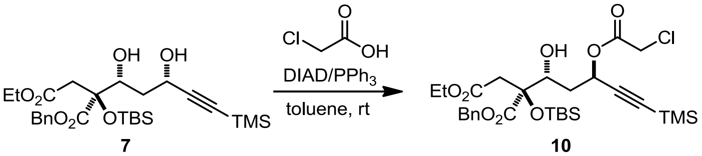


Benzyl 2-(2,2-dimethyl-6-((trimethylsilyl)ethynyl)-1,3-dioxan-4-yl)-4-hydroxy-2-((triethylsilyl)oxy)butanoate (S16): An oven-dried and cooled 20-mL scintillation vial was charged with diester **9** (95 mg, 0.164 mmol, 1.0 equiv) and THF (4 mL). The resulting solution was cooled to -78 °C, and a solution of DIBAL-H (1.46 mL, 0.562 M in THF, 0.820 mmol, 5.0 equiv) was added dropwise. When the addition of DIBAL-H was complete, the reaction was maintained at -78 °C for 20 min then was allowed to warm to 0 °C for 30 min. The reaction was quenched by the addition of saturated aqueous sodium potassium tartrate (3 mL) and was diluted with Et_2O (5 mL). The resulting biphasic mixture was stirred vigorously until clear layers were observed. Additional diethyl ether was added, and the layers were separated. The aqueous layer was extracted with Et_2O (3x 5 mL), and combined organic extracts were washed with water and brine, dried ($MgSO_4$), and concentrated *in vacuo* to give a light yellow oil, which was purified via column chromatography, eluting with 70:30 hexanes: EtOAc , to afford the title compound as a colorless oil (52 mg, 60%). Analytical data: **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.38-7.33 (m, 5H), 5.32 (d, *J* = 12.0 Hz, 1H), 4.99 (d, *J* = 12.0 Hz, 1H), 4.63 (dd, *J* = 12.0, 3.0 Hz, 1H), 4.11 (dd, *J* = 12.0, 2.5 Hz, 1H), 3.71-3.68 (m, 2H), 1.92-1.87 (m, 4H), 1.67 (dt, *J* = 13.0, 2.5 Hz, 1H), 1.32 (s, 3H), 1.19 (s, 3H), 0.97 (t, *J* = 8.0 Hz, 9H), 0.81-0.75 (m, 6H), 0.16 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$)**:** δ 173.1, 135.5, 128.8, 128.5, 128.4, 103.7, 99.5, 89.4, 81.7, 73.0, 67.0, 60.5, 58.7, 38.5, 30.6, 29.4, 18.8, 7.3, 6.8, -0.2; **LRMS (ESI$^+$)** Calcd. for $C_{28}H_{48}O_6Si_2$+Cs, 667.2; Found, 667.2; **IR** (thin film, cm^{-1}) 3432, 2958, 2911, 2875, 2248, 2181, 1752, 1641, 1457, 1381, 1251, 1194, 1160, 1112, 909, 845, 732; **TLC**(75:25 Hex:EtOAc): R_f = 0.23.

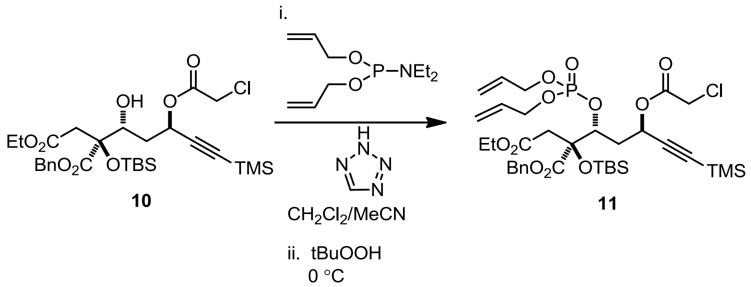


(*R*)-2-((4*R*,6*S*)-2,2-dimethyl-6-((trimethylsilyl)ethynyl)-1,3-dioxan-4-yl)-2-((triethylsilyl) oxy)butane-1,4-diol (S17): A flame-dried and cooled 1L round-bottomed flask was charged with acetonide **9** (5.0 g, 8.7 mmol, 1.0 equiv). The flask was purged with N_2, and CH_2Cl_2 (500 mL) was added. The solution was cooled to -30 °C, and lithium triethylborohydride (1M in THF, 57 mmol, 57 mL) was added dropwise over 15 min via syringe pump. The reaction temperature was maintained for 2 h, at which point the temperature was increased to -20 °C for 1 h. The reaction was quenched by the dropwise addition of HOAc (8 mL) and MeOH (30 mL). The resulting suspension was warmed to room temperature and concentrated *in vacuo*, keeping the bath temperature at or below 30 °C to avoid migration of the triethylsilyl group. The residue was redissolved in MeOH (30 mL) and concentrated *in vacuo* an additional four times. The residue was partitioned between ethyl acetate and saturated aqueous sodium bicarbonate, and the organic extracts were washed successively with saturated sodium bicarbonate (x2), water, and brine. The combined organic extracts were dried with sodium sulfate and concentrated *in vacuo*. The resulting crude oil was purified via column chromatography, eluting with a gradient of 80:20 to 70:30 hexanes: ethyl acetate to give the desired diol as a white solid (2.1 g, 56%). Analytical data: $[\alpha]_D^{25.2}$ -2.82 (*c* 1.8, $CHCl_3$); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 4.67 (dd, *J* = 11.5, 2.5 Hz, 1H), 3.93 (dd, *J* = 11.5, 2.5 Hz, 1H), 3.86-3.73 (m, 3H), 3.50 (dd, *J* = 11.0, 3.0 Hz, 1H), 2.83 (s, 1H), 2.65 (s, 1H), 1.98-1.70 (m, 4H), 1.46 (s, 6H), 0.95 (t, *J* = 3.5 Hz, 9H), 0.74-0.65 (dq, *J* = 16.0, 3.0 Hz 6H), 0.17 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 103.7, 99.6, 89.6, 78.0, 73.2, 65.6, 60.9, 58.3, 37.7, 31.3, 29.9, 19.2, 7.1, 6.8, -0.2; **LRMS (ESI$^+$)** Calcd. for $C_{21}H_{42}O_5Si_2$+Na, 453.3; Found, 453.4; **IR** (thin film, cm^{-1}) 3389, 2956, 2876, 2183, 1739, 1460, 1380, 1250, 1161, 1055, 844, 733; **TLC**(75:25 Hex:EtOAc): R_f = 0.09.

Synthesis of Leustroducsin B Core:

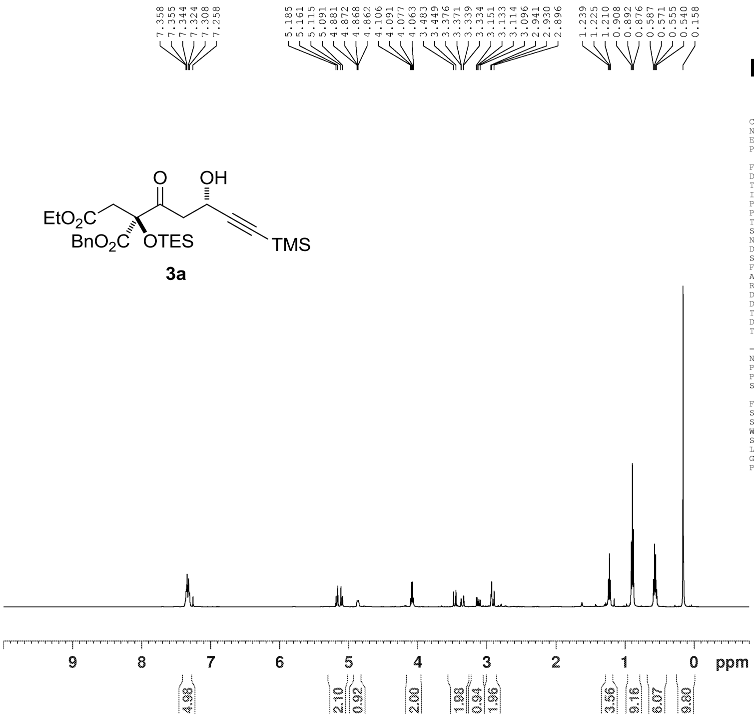


(*S*)-1-benzyl 4-ethyl 2-((*tert*-butyldimethylsilyl)oxy)-2-((1*R*,3*R*)-3-(2-chloroacetoxy)-1-hydroxy-5-(trimethylsilyl)pent-4-yn-1-yl)succinate (10): An oven-dried and cooled 20-mL scintillation vial equipped with magnetic stir bar was charged with diol **7** (37 mg, 0.068 mmol, 1.0 equiv), PPh_3 (55 mg, 0.211 mmol, 3.1 equiv), and chloroacetic acid (12 mg, 0.127 mmol, 1.9 equiv). The vial was purged with N_2, and toluene (2 mL) was added. DIAD (0.031 mL, 36 mg, 0.204 mmol, 3.0 equiv) was added dropwise, and the resulting solution was stirred at rt for 30 min, at which point TLC analysis indicated complete consumption of the starting material (R_f = 0.24, 80:20 hexanes:EtOAc). The reaction was loaded directly onto an SiO_2 plug and eluted with 100:0 to 90:10 hexanes:EtOAc to afford the desired chloroacetate (**10**) as a colorless oil (27 mg, 65%). Analytical data**:** **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.35 (br. s., 5H), 5.62 (d, *J* = 7.6 Hz, 1H), 5.21 (br. s., 2H), 4.05 (br. s., 4H), 3.84 (dd, *J* = 16.8, 8.4 Hz, 1H), 2.92 (d, *J* = 15.6 Hz, 1H), 2.73 (d, *J* = 15.6 Hz, 1H), 2.44 (d, *J* = 7.2 Hz, 1H), 2.15 (dd, 23.6, 13.6 Hz, 1H), 1.82 (dd, *J* = 23.6, 11.6 Hz, 1H), 1.20 (t, *J* = 6.8 Hz, 3H), 0.85 (s, 9H), 0.16 (s, 9H), 0.16 (s, 3H; 2 coincident resonances), 0.10 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.9, 169.7, 116.2, 135.1, 128.7, 128.6, 128.5, 101.4, 91.7, 80.9, 72.3, 67.6, 63.4, 60.9, 41.6, 40.7, 37.1, 26.0, 18.8, 14.0, -0.3, -2.9, -3.1; **LRMS (ESI$^+$)** Calcd. for $C_{21}H_{45}ClO_8Si_2$+Na, 635.2; Found, 635.2; **IR** (thin film, cm^{-1}) 3434, 2359, 2341, 2089, 1644, 1539, 1470, 1250, 508; **TLC** (80:20 hexanes:EtOAc): R_f = 0.4.



(*S*)-1-benzyl-4-ethyl-2-((1*R*,3*R*)-1-((bis(allyloxy)phosphoryl)oxy)-3-(2-chloroacetoxy)-5-(trimethylsilyl)pent-4-yn-1-yl)-2-((*tert*-butyldimethylsilyl)oxy)succinate (11): A flame-dried and cooled 20-mL scintillation vial equipped with magnetic stir bar was charged with chloroacetate **10** (20 mg, 0.0326 mmol, 1.0 equiv), the phosphoramidite (32 mg, 0.147 mmol, 4.5 equiv), and CH_2Cl_2 (2.0 mL). The resulting solution was cooled to 0 °C, and a solution of tetrazole in MeCN (0.5 M, 0.5 mL, 0.244 mmol, 7.5 equiv) was added. The reaction was allowed to warm to rt, and it was stirred for 2.5 h. The solution was again cooled to 0 °C, and a solution of tBuOOH in decane (5.2 M, 0.1 mL) was added dropwise. After stirring for 1h at the same temperature, an additional 0.1 mL of the tBuOOH solution was added, and the reaction was stirred for an additional 30 min. The reaction was quenched by the addition of saturated $NaSO_3$ (aq), diluted with Et_2O, and washed with saturated aqueous $NaHCO_3$ and brine. The solvent was removed *in vacuo*, and the crude organic material was purified via flash chromatography, eluting with 90:10 to 85:15 Hexanes:EtOAc, to afford the title compound as a colorless oil (18 mg, 71%). Analytical data**:** **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.42-7.26 (m, 5H), 6.01-5.79 (m, 2H), 5.47 (d, *J* = 10.8 Hz, 1H),5.36-5.19 (m, 5H), 5.10 (d, *J* = 12.0 Hz, 1H), 4.74 (t, *J* = 9.2 Hz, 1H), 4.52-4.34 (m, 4H), 4.18 (d, *J* = 14.8 Hz, 1H), 4.08 (d, *J* = 14.8 Hz, 1H), 4.06-4.02 (m, 2H), 3.0 (d, *J* = 15.2 Hz, 1H), 2.69 (d, *J* = 15.2 Hz, 1H), 2.39-2.33 (m, 1H), 2.19-2.13 (m, 1H), 1.18 (t, *J* = 7.2 Hz, 3H), 0.87 (s, 9H), 0.21 (s, 3H), 0.15 (s, 9H), 0.13 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$, ^{13}C-^{31}P coupling constants indicated): δ 170.6, 170.0, 166.1, 135.2, 132.2 (d, *J* = 6.3 Hz), 132.1 (d, *J* = 7.5 Hz), 128.5, 128.3, 118.9, 118.3, 101.1, 91.5, 80.5 *(*d, *J* = 5.0 Hz), 78.5 (d, *J* = 6.3 Hz), 68.7 (d, *J* = 6.3 Hz), 68.4 (d, *J* = 6.3 Hz), 67.8, 62.3, 60.9, 53.4, 42.7, 41.0, 36.5, 26.0, 19.0, 14.0, -0.4, -2.8, -3.0; **^{31}P NMR** (160 MHz, $CDCl_3$): δ -1.79 Hz; **LRMS (ESI$^+$)** Calcd. for $C_{35}H_{54}ClO_{11}PSi_2$+Na, 795.3; Found, 795.2; **IR** (thin film, cm^{-1}) 3436, 2360, 2090, 1644, 1463, 1252, 1081; **TLC**(70:30 Hex:EtOAc): R_f = 0.52.

^{1}H and ^{13}C Spectra



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PULPROG                    zg30
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SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    7507.507 Hz
FIDRES                 0.114555 Hz
AQ                    4.3647475 sec
RG                           57
DW                       66.600 usec
DE                         6.00 usec
TE                        292.8 K
D1                   1.00000000 sec
TD0                           1

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P1                        17.50 usec
PL1                       -3.00 dB
SFO1                500.1330008 MHz

F2 - Processing parameters
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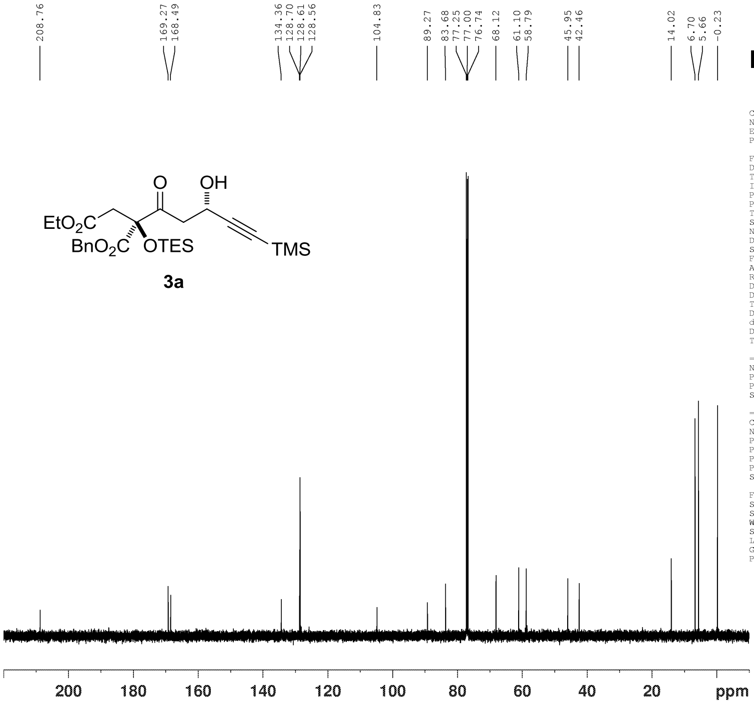

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PROCNO                        1

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SOLVENT                   CDCl3
NS                           96
DS                            2
SWH                   30581.039 Hz
FIDRES                 0.466630 Hz
AQ                    1.0715636 sec
RG                        32768
DW                       16.350 usec
DE                         6.00 usec
TE                        293.4 K
D1                   1.00000000 sec
d11                  0.03000000 sec
DELTA                0.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         6.80 usec
PL1                        0.00 dB
SFO1                125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    100.00 usec
PL2                       -3.00 dB
PL12                      14.00 dB
PL13                      14.00 dB
SFO2                500.1320005 MHz

F2 - Processing parameters
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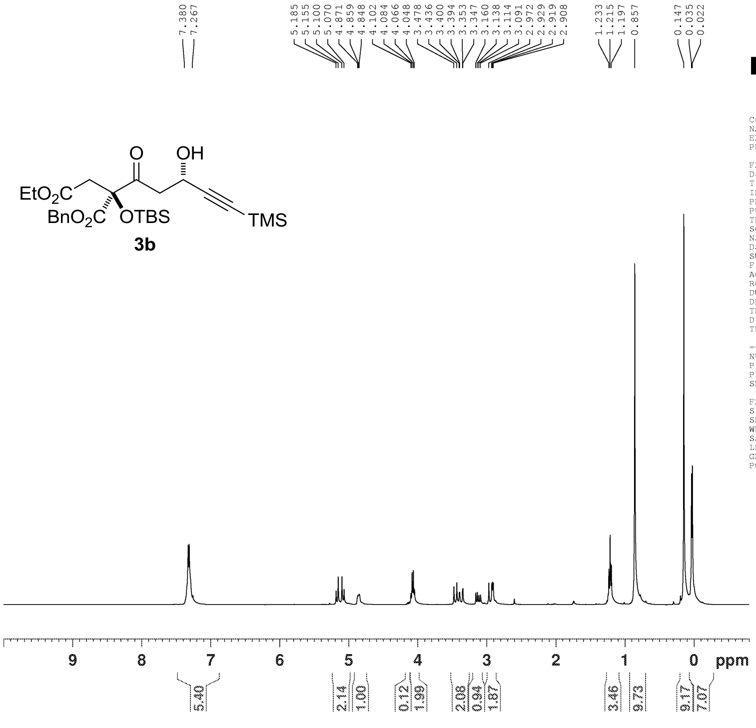

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PROCNO                           1

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INSTRUM                      spect
PROBHD           5 mm QNP  1H/1
PULPROG                      zg30
TD                           32768
SOLVENT                      CDCl3
NS                              32
DS                               0
SWH                       6218.905 Hz
FIDRES                    0.189786 Hz
AQ                       2.6345973 sec
RG                            40.3
DW                          80.400 usec
DE                            6.00 usec
TE                           300.0 K
D1                      1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                            1H
P1                            9.30 usec
PL1                          -3.00 dB
SFO1                   399.8424924 MHz

F2 - Processing parameters
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WDW                             EM
SSB                              0
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PC                            1.00
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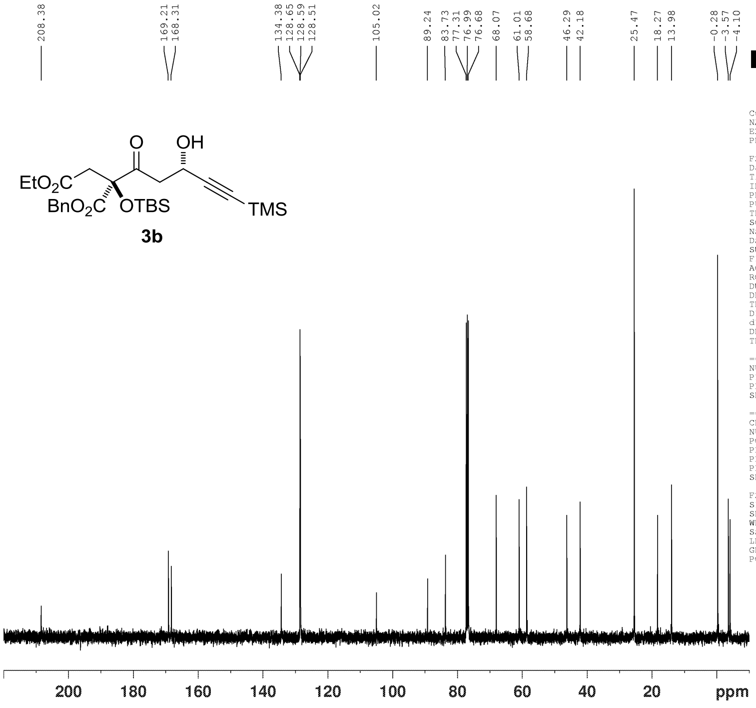

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PROCNO                           1

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FIDRES                    0.383387 Hz
AQ                       1.3042164 sec
RG                           32768
DW                          19.900 usec
DE                            6.00 usec
TE                           300.0 K
D1                      2.00000000 sec
d11                     0.03000000 sec
DELTA                   1.89999998 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           13C
P1                            7.25 usec
PL1                           0.00 dB
SFO1                   100.5499020 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                        90.00 usec
PL2                          -3.00 dB
PL12                         16.72 dB
PL13                         20.00 dB
SFO2                   399.8415994 MHz

F2 - Processing parameters
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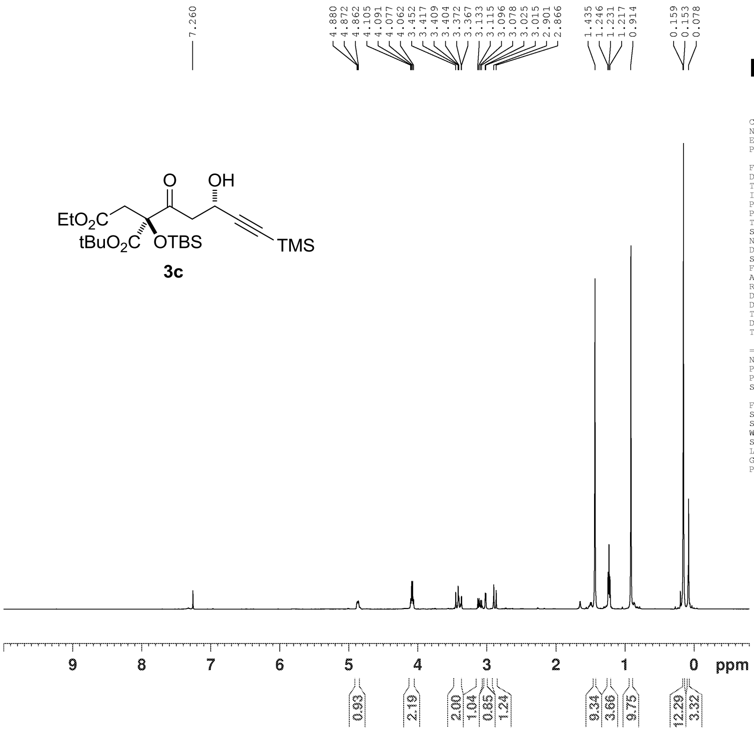

3c

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Current Data Parameters
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EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
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INSTRUM                  spect
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PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                   7507.507 Hz
FIDRES                0.114555 Hz
AQ                   4.3647475 sec
RG                          57
DW                      66.600 usec
DE                        6.00 usec
TE                       292.1 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.50 usec
PL1                       0.00 dB
SFO1               500.1330008 MHz

F2 - Processing parameters
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SF                 500.1300135 MHz
WDW                         EM
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LB                        0.25 Hz
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PC                        1.00
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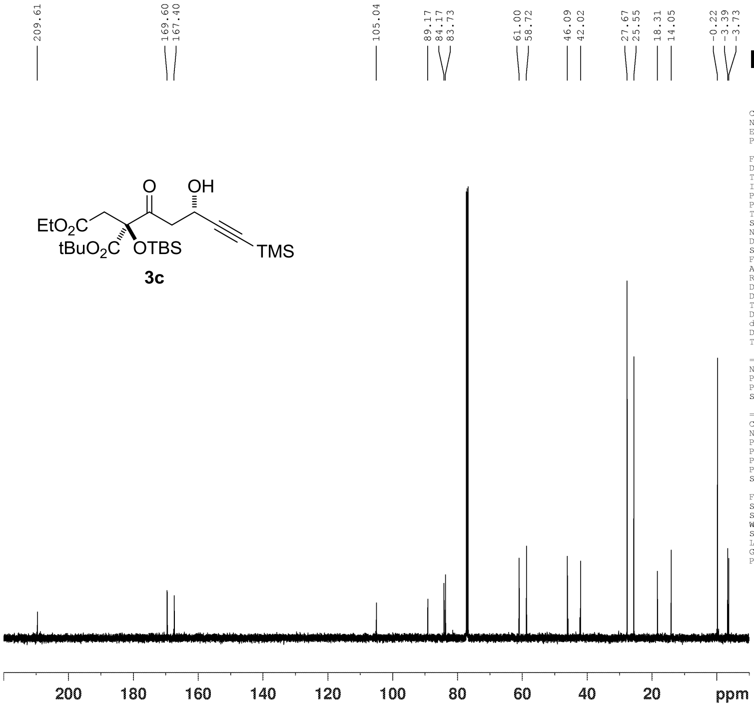

3c

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Current Data Parameters
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EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100709
Time                     10.24
INSTRUM                  spect
PROBHD          5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         295
DS                           0
SWH                  30581.039 Hz
FIDRES                0.466630 Hz
AQ                   1.0715636 sec
RG                       32768
DW                      16.350 usec
DE                        6.00 usec
TE                       293.3 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        7.00 usec
PL1                       0.00 dB
SFO1               125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    70.00 usec
PL2                       0.00 dB
PL12                     14.30 dB
PL13                     18.00 dB
SFO2               500.1320005 MHz

F2 - Processing parameters
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SF                 125.7577945 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
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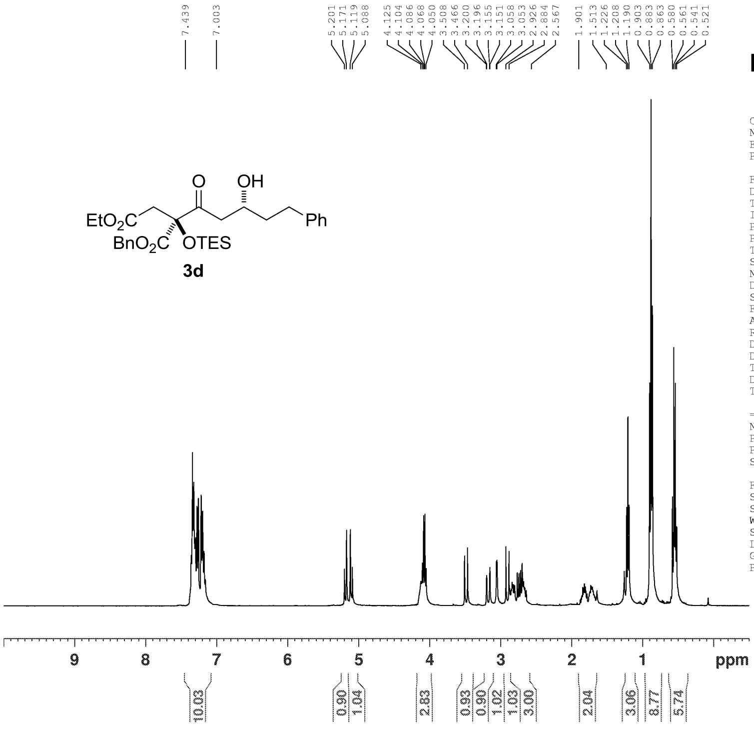

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EXPNO                  1
PROCNO                 1

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TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6218.905 Hz
FIDRES          0.189786 Hz
AQ             2.6345973 sec
RG                  90.5
DW                80.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.30 usec
PL1                -3.00 dB
SFO1         399.7824920 MHz

F2 - Processing parameters
SI                 32768
SF           399.7800115 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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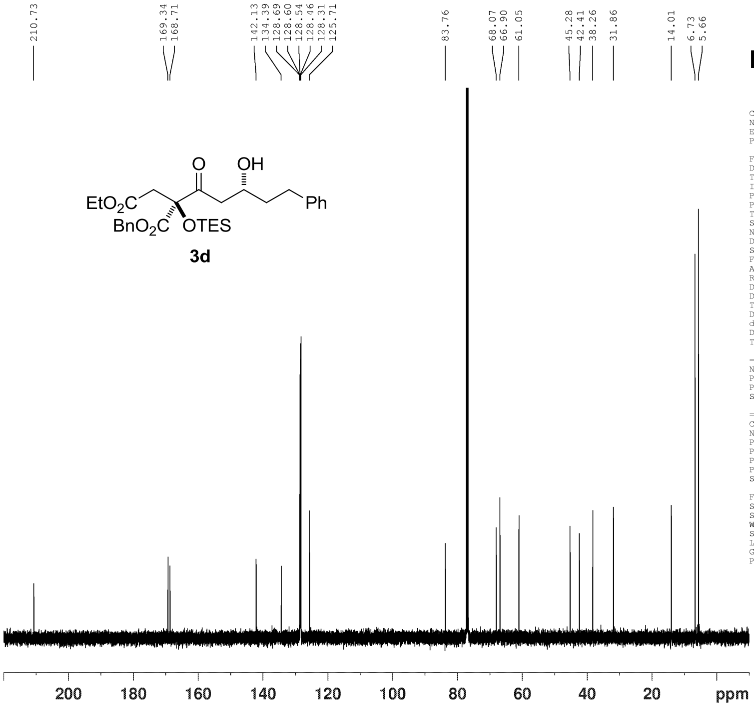

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Current Data Parameters
NAME     SNG-Cinn3comC13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
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Time               14.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   222
DS                     0
SWH            30581.039 Hz
FIDRES          0.466630 Hz
AQ             1.0715636 sec
RG                 32768
DW                16.350 usec
DE                  6.00 usec
TE                 293.2 K
D1            1.00000000 sec
d11           0.03000000 sec
DELTA         0.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  6.80 usec
PL1                 0.00 dB
SFO1         125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -3.00 dB
PL12               14.00 dB
PL13               14.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
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SF           125.7577973 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
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PC                  1.40
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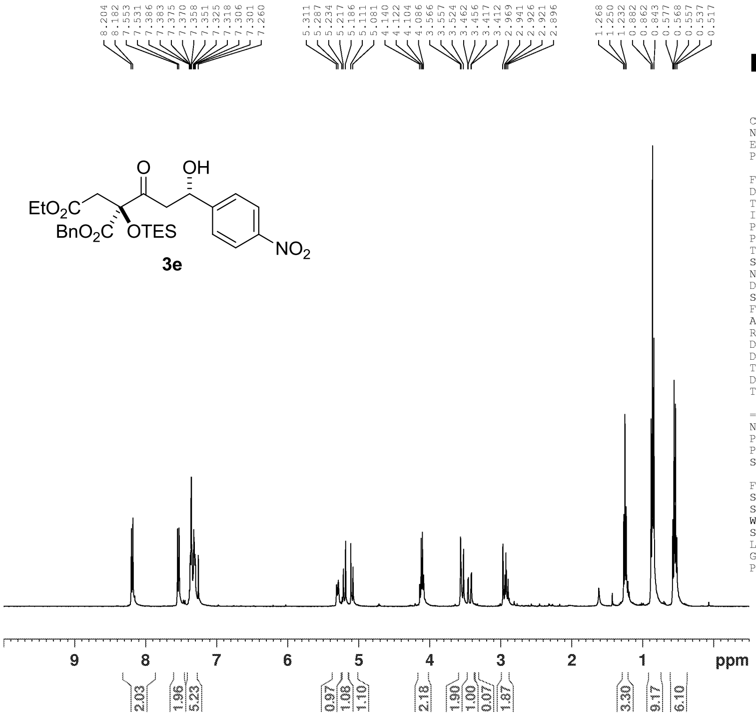

3e

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Current Data Parameters
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EXPNO                   1
PROCNO                  1

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INSTRUM             spect
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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              6218.905 Hz
FIDRES           0.189786 Hz
AQ              2.6345973 sec
RG                  161.3
DW                 80.400 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.30 usec
PL1                 -3.00 dB
SFO1          399.7824920 MHz

F2 - Processing parameters
SI                  32768
SF            399.7800103 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
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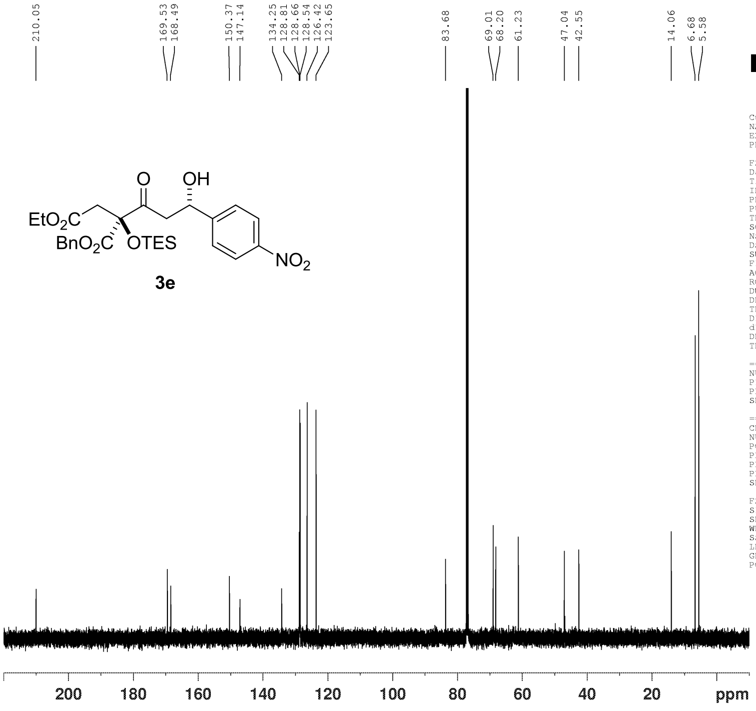

3e

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Current Data Parameters
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PROCNO                      1

F2 - Acquisition Parameters
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Time                    14.11
INSTRUM                 spect
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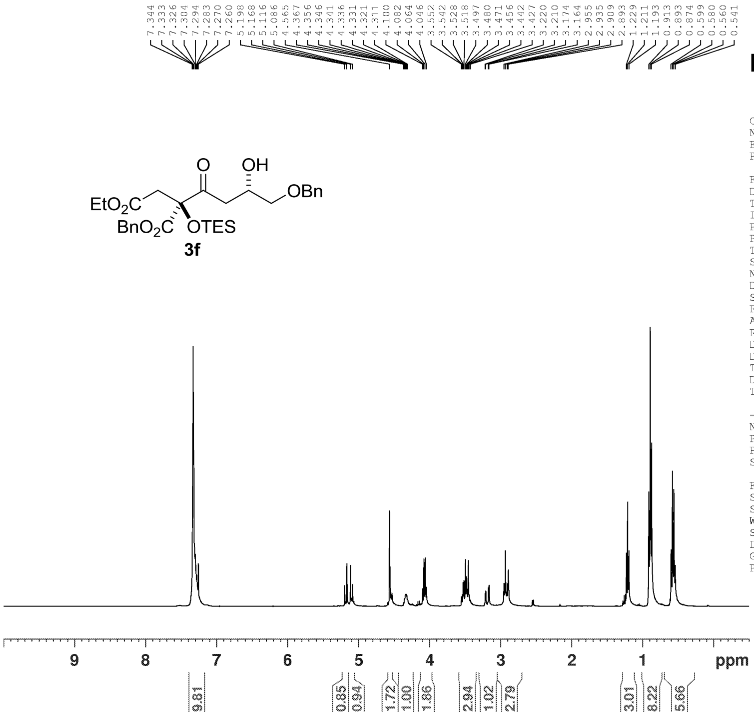
PULPROG               zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        262
DS                          0
SWH                 30581.039 Hz
FIDRES               0.466630 Hz
AQ                  1.0715636 sec
RG                      32768
DW                     16.350 usec
DE                       6.00 usec
TE                      292.7 K
D1                 1.00000000 sec
d11                0.03000000 sec
DELTA              0.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                       6.80 usec
PL1                      0.00 dB
SFO1              125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                       1H
PCPD2                  100.00 usec
PL2                     -3.00 dB
PL12                    14.00 dB
PL13                    14.00 dB
SFO2              500.1320005 MHz

F2 - Processing parameters
SI                      65536
SF                125.7577960 MHz
WDW                        EM
SSB                         0
LB                       0.50 Hz
GB                          0
PC                       1.40
```

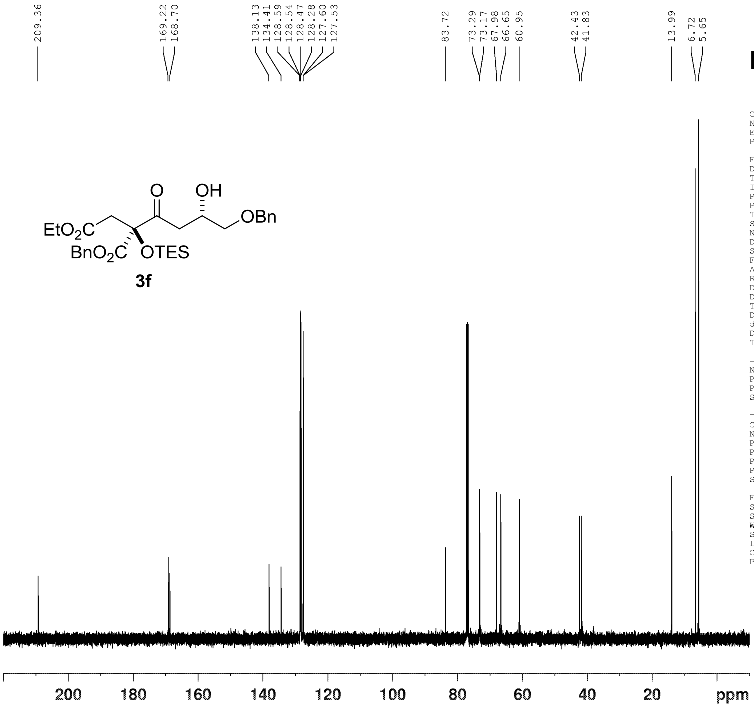


```
Current Data Parameters
NAME        SNG-Bncom-H1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100706
Time               16.05
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             6218.905 Hz
FIDRES          0.189786 Hz
AQ             2.6345973 sec
RG                    64
DW                80.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.30 usec
PL1                -3.00 dB
SFO1         399.7824920 MHz

F2 - Processing parameters
SI                 32768
SF           399.7800103 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



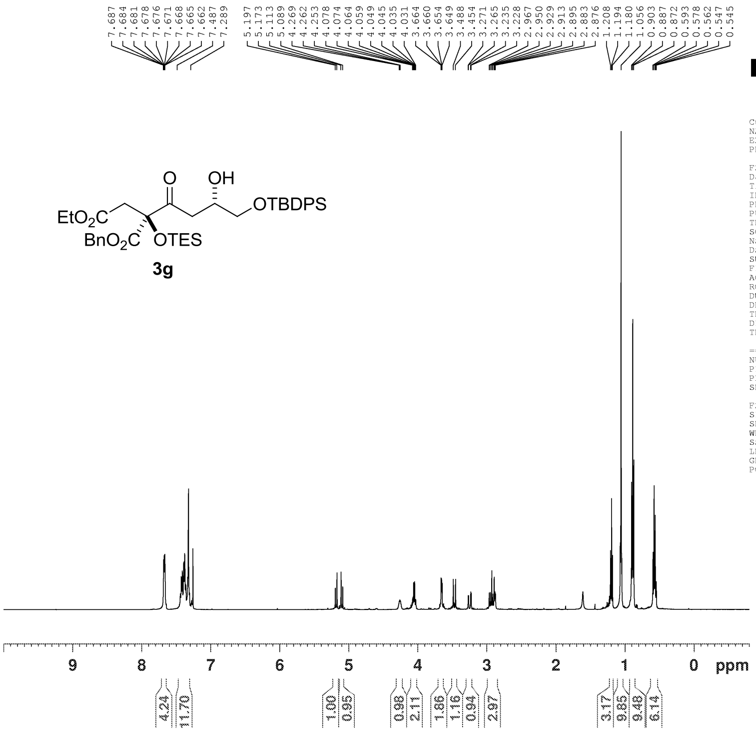
```
Current Data Parameters
NAME               SNG-Bn3comC13
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20100706
Time                       13.52
INSTRUM                    spect
PROBHD           5 mm PABBO BB/
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           198
DS                             0
SWH                    30581.039 Hz
FIDRES                  0.466630 Hz
AQ                     1.0715636 sec
RG                         32768
DW                        16.350 usec
DE                          6.00 usec
TE                         292.7 K
D1                    1.00000000 sec
d11                   0.03000000 sec
DELTA                 0.89999998 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                          7.00 usec
PL1                         0.00 dB
SFO1                 125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                      70.00 usec
PL2                         0.00 dB
PL12                       14.30 dB
PL13                       18.00 dB
SFO2                 500.1320005 MHz

F2 - Processing parameters
SI                         65536
SF                   125.7578021 MHz
WDW                           EM
SSB                            0
LB                          0.50 Hz
GB                             0
PC                          1.40
```



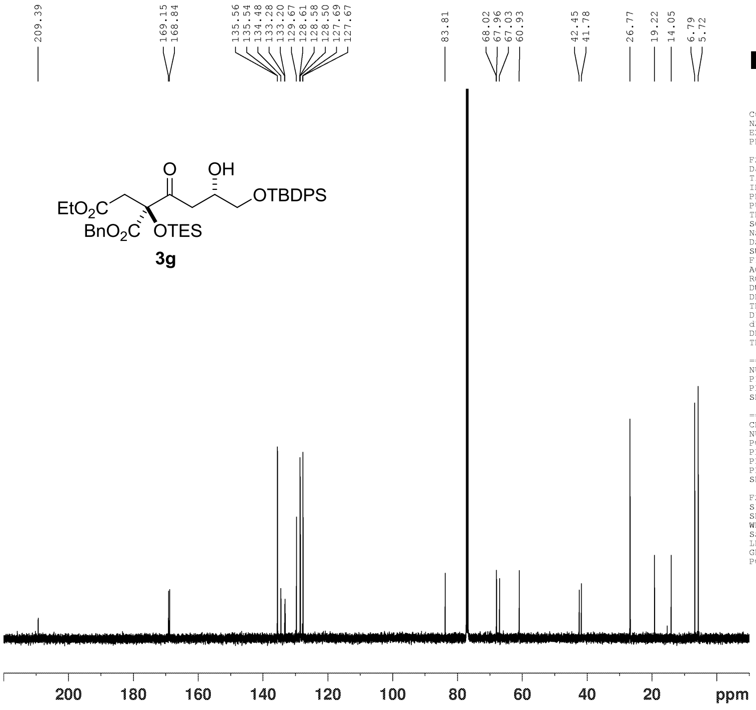
3g

```
Current Data Parameters
NAME             SNG-OTBDPS3comH1
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100706
Time                        15.02
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                              8
DS                              0
SWH                      7507.507 Hz
FIDRES                   0.114555 Hz
AQ                      4.3647475 sec
RG                          143.7
DW                         66.600 usec
DE                           6.00 usec
TE                          292.6 K
D1                     1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          13.50 usec
PL1                          0.00 dB
SFO1                  500.1330008 MHz

F2 - Processing parameters
SI                          32768
SF                    500.1300132 MHz
WDW                            EM
SSB                             0
LB                           0.25 Hz
GB                              0
PC                           1.00
```



3g

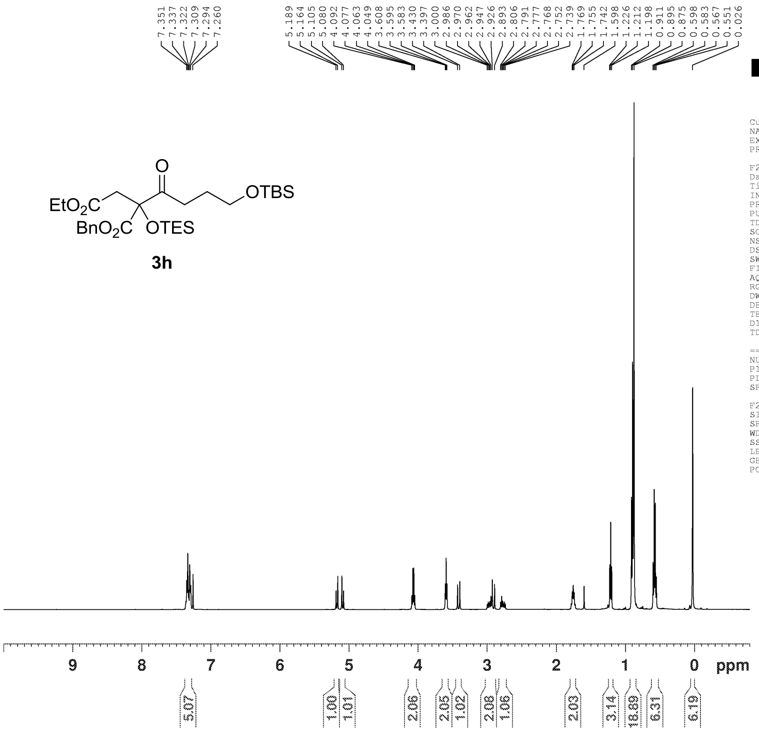
```
Current Data Parameters
NAME            SNG-OTBDPS3comC13
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100706
Time                        15.07
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            599
DS                              0
SWH                     30581.039 Hz
FIDRES                   0.466630 Hz
AQ                      1.0715636 sec
RG                          32768
DW                         16.350 usec
DE                           6.00 usec
TE                          293.6 K
D1                     1.00000000 sec
d11                    0.03000000 sec
DELTA                  0.89999998 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           6.80 usec
PL1                          0.00 dB
SFO1                  125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                      100.00 usec
PL2                         -3.00 dB
PL12                        14.00 dB
PL13                        14.00 dB
SFO2                  500.1320005 MHz

F2 - Processing parameters
SI                          65536
SF                    125.7577952 MHz
WDW                            EM
SSB                             0
LB                           0.50 Hz
GB                              0
PC                           1.40
```

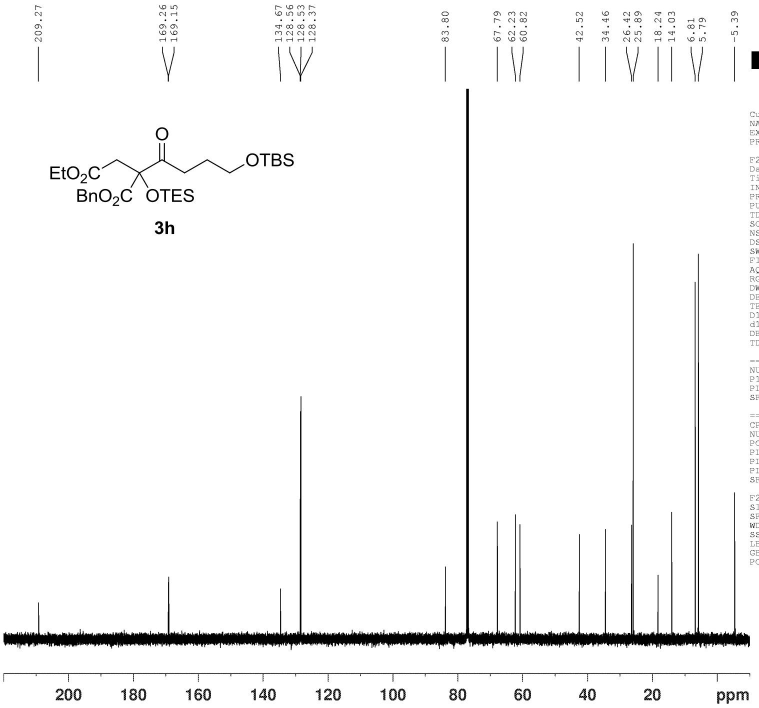


```
Current Data Parameters
NAME             SNG-Butyro3comH1
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100709
Time                         9.58
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     cdcl3
NS                              8
DS                              0
SWH                      7507.507 Hz
FIDRES                   0.114555 Hz
AQ                      4.3647475 sec
RG                          101.6
DW                         66.600 usec
DE                           6.00 usec
TE                          292.0 K
D1                     1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          13.50 usec
PL1                          0.00 dB
SFO1                  500.1330008 MHz

F2 - Processing parameters
SI                          32768
SF                    500.1300133 MHz
WDW                            EM
SSB                             0
LB                           0.25 Hz
GB                              0
PC                           1.00
```



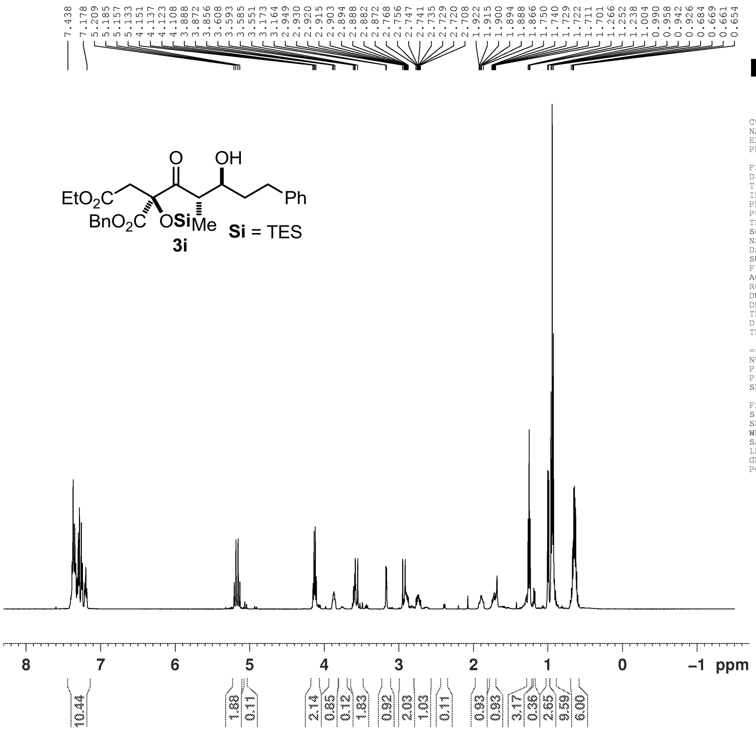
```
Current Data Parameters
NAME             SNG-Butyro3comC13
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100709
Time                        10.02
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                   zgpg30
TD                          65536
SOLVENT                     cdcl3
NS                            336
DS                              0
SWH                     30581.039 Hz
FIDRES                   0.466630 Hz
AQ                      1.0715636 sec
RG                          32768
DW                         16.350 usec
DE                           6.00 usec
TE                          293.0 K
D1                     1.00000000 sec
d11                    0.03000000 sec
DELTA                  0.89999998 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           7.00 usec
PL1                          0.00 dB
SFO1                  125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                       70.00 usec
PL2                          0.00 dB
PL12                        14.30 dB
PL13                        18.00 dB
SFO2                  500.1320005 MHz

F2 - Processing parameters
SI                          65536
SF                    125.7577941 MHz
WDW                            EM
SSB                             0
LB                           0.50 Hz
GB                              0
PC                           1.40
```

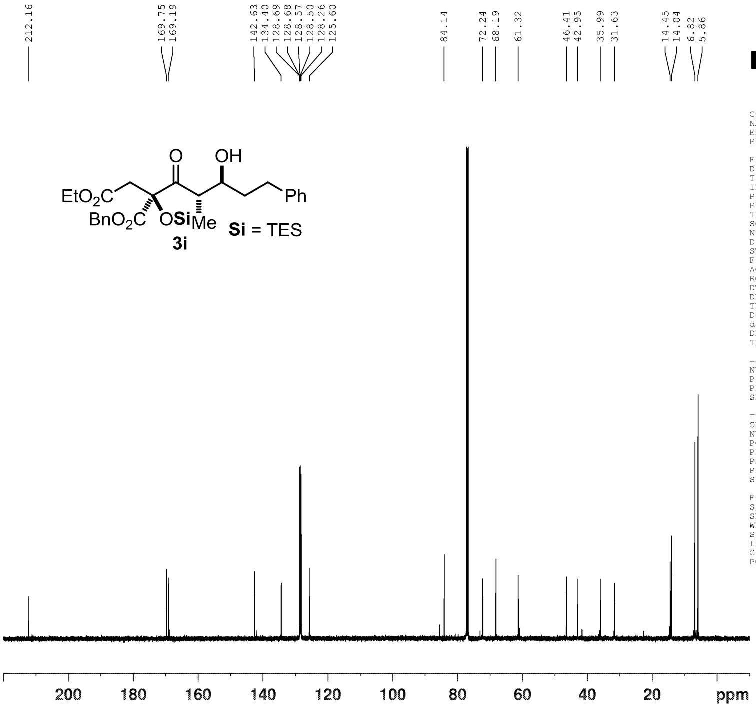


```
Current Data Parameters
NAME             SNG-transcinn-H1
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100719
Time                         9.27
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              0
SWH                      7507.507 Hz
FIDRES                   0.114555 Hz
AQ                      4.3647475 sec
RG                            181
DW                         66.600 usec
DE                           6.00 usec
TE                          291.2 K
D1                     1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          17.50 usec
PL1                         -3.00 dB
SFO1                  500.1330008 MHz

F2 - Processing parameters
SI                          32768
SF                    500.1300007 MHz
WDW                            EM
SSB                             0
LB                           0.25 Hz
GB                              0
PC                           1.00
```



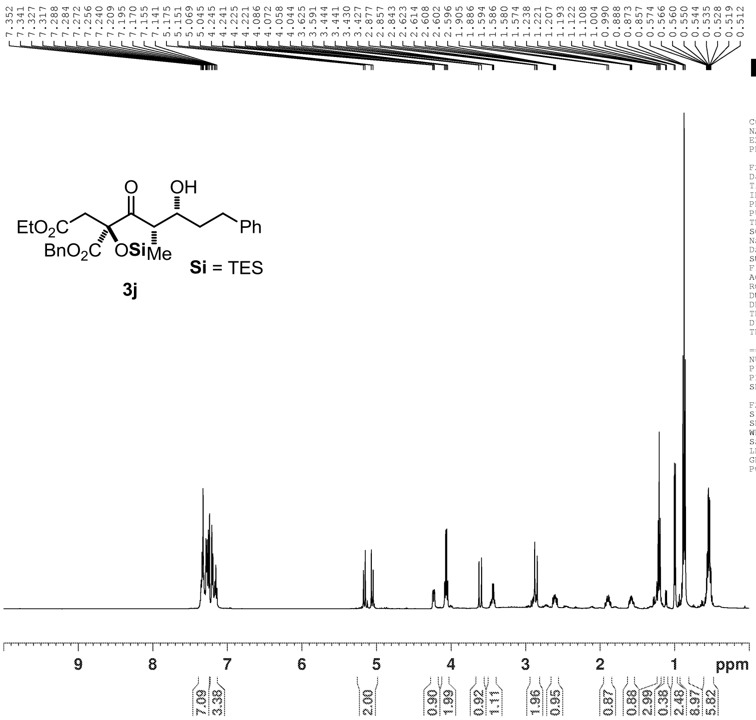
```
Current Data Parameters
NAME            SNG-transcinn-C13
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20100719
Time                         9.34
INSTRUM                     spect
PROBHD           5 mm PABBO BB/
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                           1312
DS                              2
SWH                     30581.039 Hz
FIDRES                   0.466630 Hz
AQ                      1.0715636 sec
RG                          32768
DW                         16.350 usec
DE                           6.00 usec
TE                          291.4 K
D1                     1.00000000 sec
d11                    0.03000000 sec
DELTA                  0.89999998 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          13C
P1                           7.00 usec
PL1                          0.00 dB
SFO1                  125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                           1H
PCPD2                       70.00 usec
PL2                          0.00 dB
PL12                        14.30 dB
PL13                        18.00 dB
SFO2                  500.1320005 MHz

F2 - Processing parameters
SI                          65536
SF                    125.7577978 MHz
WDW                            EM
SSB                             0
LB                           0.50 Hz
GB                              0
PC                           1.40
```



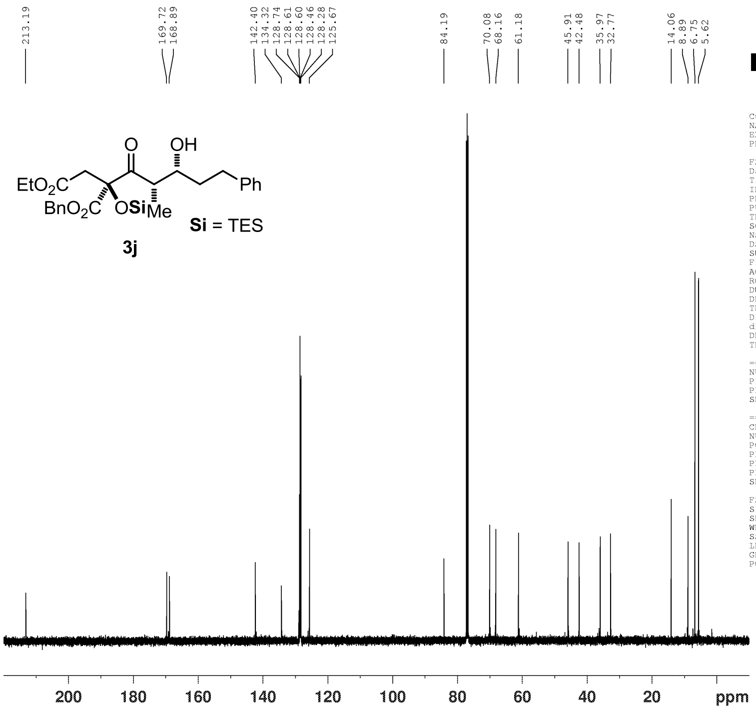
3j

```
Current Data Parameters
NAME             JTM-6-29-a1+2
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100812
Time                      9.08
INSTRUM                  spect
PROBHD          5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          17
DS                           0
SWH                   7507.507 Hz
FIDRES                0.114555 Hz
AQ                   4.3647475 sec
RG                        71.8
DW                      66.600 usec
DE                        6.00 usec
TE                       295.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.50 usec
PL1                       0.00 dB
SFO1               500.1330008 MHz

F2 - Processing parameters
SI                       32768
SF                 500.1300234 MHz
WDW                         EM
SSB                          0
LB                        0.25 Hz
GB                           0
PC                        1.00
```



3j

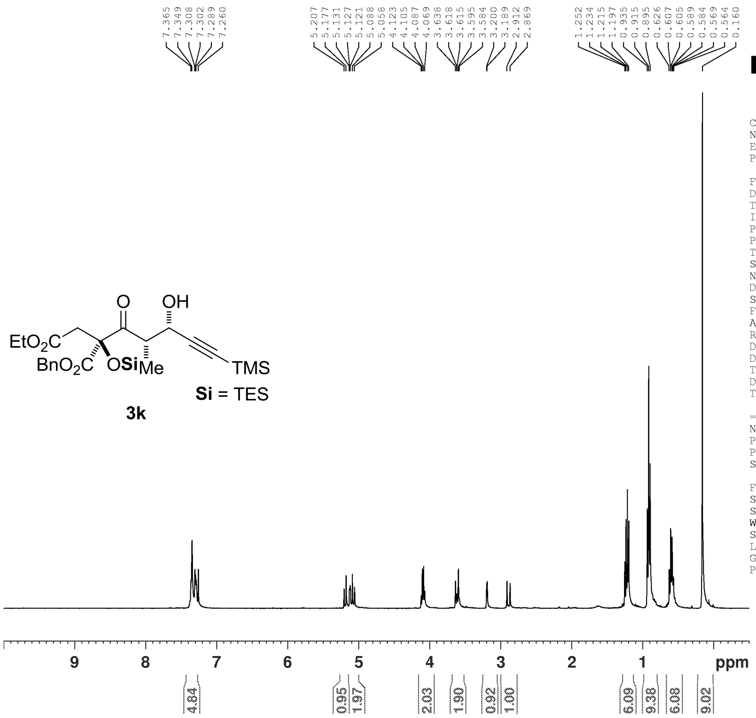
```
Current Data Parameters
NAME         JTM-6-29-a1+2 13C
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100812
Time                      9.17
INSTRUM                  spect
PROBHD          5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         327
DS                           0
SWH                  30581.039 Hz
FIDRES                0.466630 Hz
AQ                   1.0715636 sec
RG                      4597.6
DW                      16.350 usec
DE                        6.00 usec
TE                       296.4 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        7.00 usec
PL1                       0.00 dB
SFO1               125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    70.00 usec
PL2                       0.00 dB
PL12                     14.30 dB
PL13                     18.00 dB
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                       65536
SF                 125.7577969 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
GB                           0
PC                        1.40
```

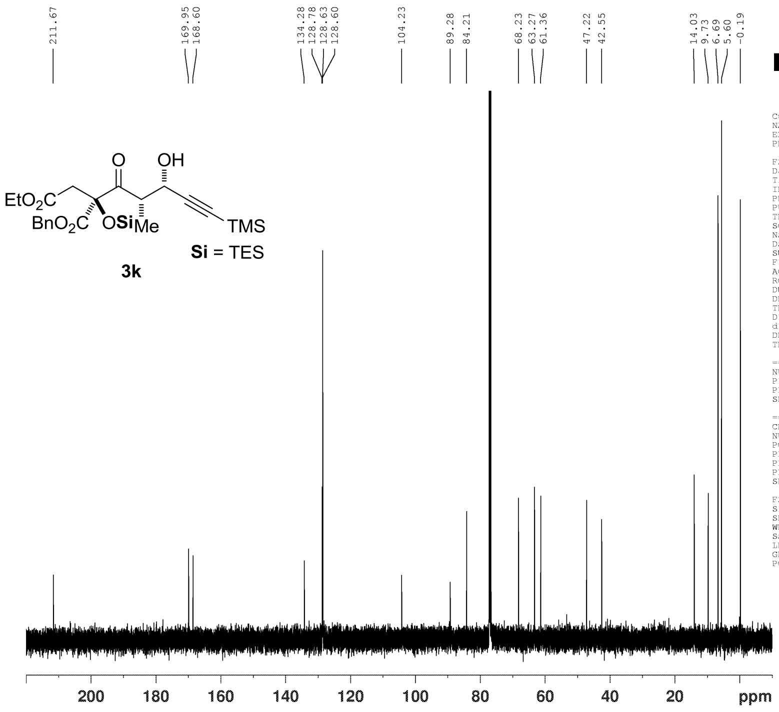


```
Current Data Parameters
NAME      SNG-Me3com-H1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20100706
Time              16.03
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            6218.905 Hz
FIDRES         0.189786 Hz
AQ            2.6345973 sec
RG                161.3
DW               80.400 usec
DE                 6.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.30 usec
PL1               -3.00 dB
SFO1        399.7824920 MHz

F2 - Processing parameters
SI                32768
SF          399.7800105 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



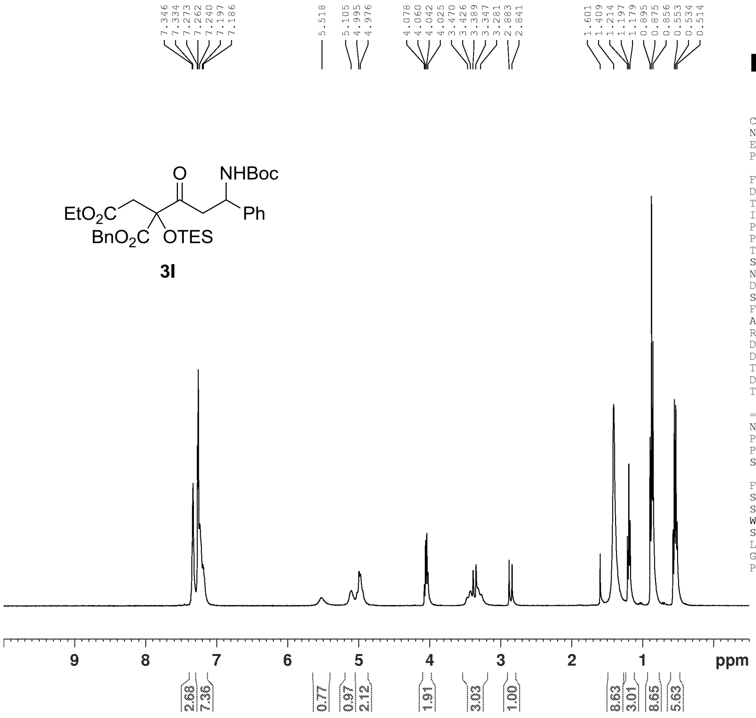
```
Current Data Parameters
NAME      SNG-Me3comC13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20100706
Time              14.43
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  453
DS                    0
SWH           30581.039 Hz
FIDRES         0.466630 Hz
AQ            1.0715636 sec
RG                32768
DW               16.350 usec
DE                 6.00 usec
TE                293.6 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 6.80 usec
PL1                0.00 dB
SFO1        125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -3.00 dB
PL12              14.00 dB
PL13              14.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                65536
SF          125.7577945 MHz
WDW                  EM
SSB                   0
LB                 0.50 Hz
GB                    0
PC                 1.40
```

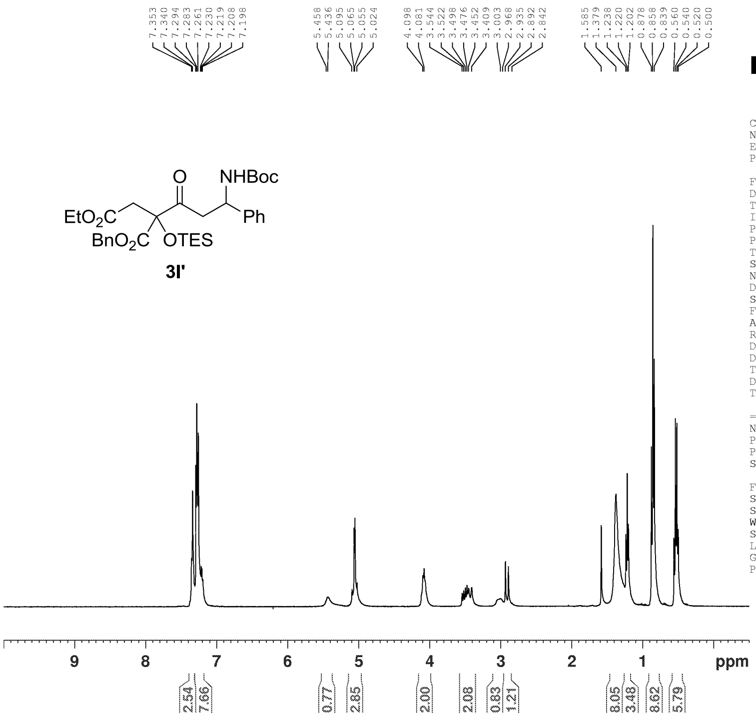


```
Current Data Parameters
NAME          JTM-5-253-5
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100717
Time                12.48
INSTRUM             spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     15
DS                      0
SWH              6218.905 Hz
FIDRES           0.189786 Hz
AQ              2.6345973 sec
RG                  161.3
DW                 80.400 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.30 usec
PL1                 -3.00 dB
SFO1          399.7824920 MHz

F2 - Processing parameters
SI                  32768
SF            399.7800105 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

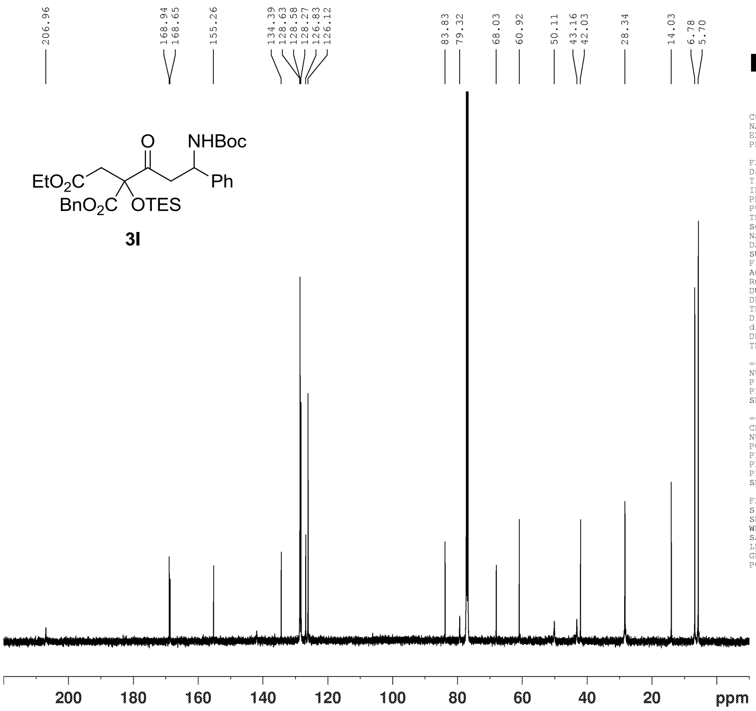


```
Current Data Parameters
NAME          JTM-5-253-6
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100717
Time                12.52
INSTRUM             spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     30
DS                      0
SWH              6218.905 Hz
FIDRES           0.189786 Hz
AQ              2.6345973 sec
RG                  161.3
DW                 80.400 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.30 usec
PL1                 -3.00 dB
SFO1          399.7824920 MHz

F2 - Processing parameters
SI                  32768
SF            399.7800105 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



3l

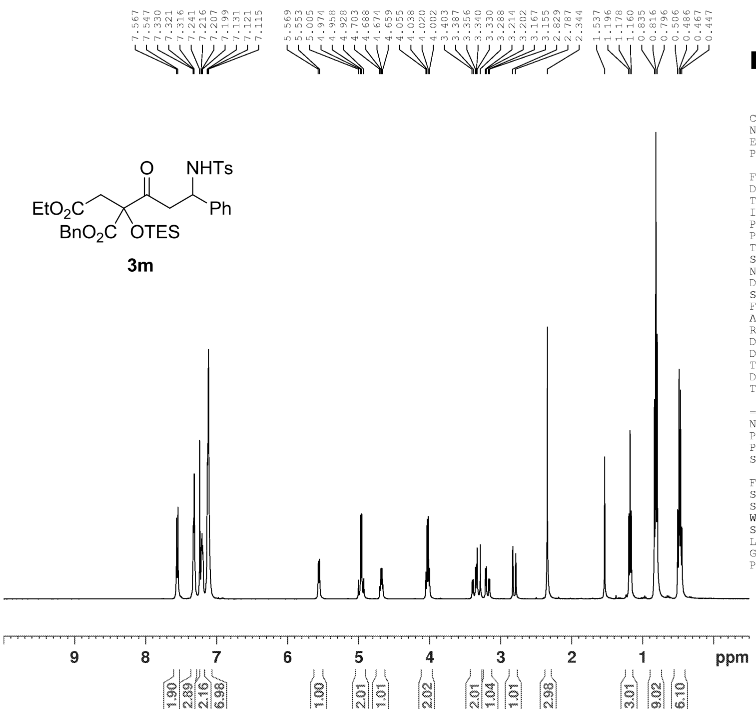
```
Current Data Parameters
NAME             SNG-lactam-D1-C13
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20100719
Time                         10.24
INSTRUM                      spect
PROBHD            5 mm PABBO BB/
PULPROG                    zgpg30
TD                           65536
SOLVENT                      CDCl3
NS                            3524
DS                               0
SWH                      30581.039 Hz
FIDRES                    0.466630 Hz
AQ                       1.0715636 sec
RG                           32768
DW                          16.350 usec
DE                            6.00 usec
TE                           291.9 K
D1                      1.00000000 sec
d11                     0.03000000 sec
DELTA                   0.89999998 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           13C
P1                            7.00 usec
PL1                           0.00 dB
SFO1                   125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                        70.00 usec
PL2                           0.00 dB
PL12                         14.30 dB
PL13                         18.00 dB
SFO2                   500.1320005 MHz

F2 - Processing parameters
SI                           65536
SF                     125.7577965 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```



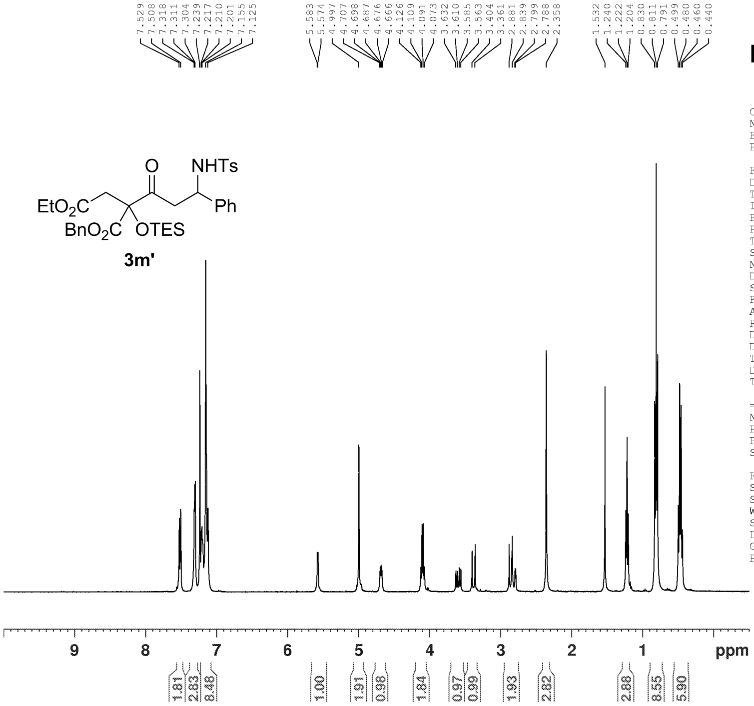
3m

```
Current Data Parameters
NAME                JTM-6-45-3
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100814
Time                     13.52
INSTRUM                  spect
PROBHD   5 mm TXI 13C Z
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          24
DS                           0
SWH                   5341.880 Hz
FIDRES                0.163021 Hz
AQ                   3.0672283 sec
RG                         256
DW                      93.600 usec
DE                        6.00 usec
TE                       295.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       10.50 usec
PL1                       0.00 dB
SFO1               400.0923618 MHz

F2 - Processing parameters
SI                       32768
SF                 400.0900118 MHz
WDW                         EM
SSB                          0
LB                        0.25 Hz
GB                           0
PC                        1.00
```



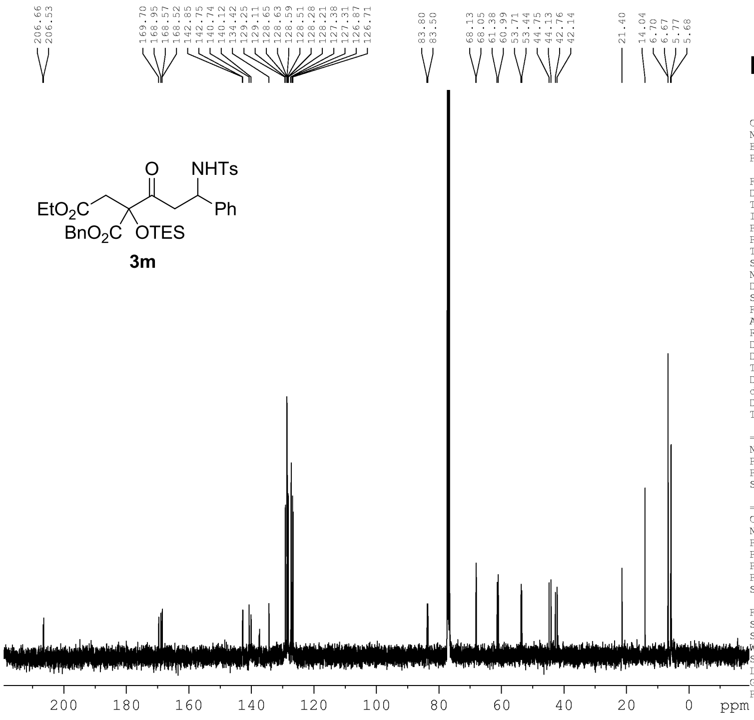
3m'

```
Current Data Parameters
NAME          JTM-6-45-4
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100814
Time               13.56
INSTRUM            spect
PROBHD   5 mm TXI 13C Z
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    14
DS                     0
SWH             5341.880 Hz
FIDRES          0.163021 Hz
AQ             3.0672283 sec
RG                   256
DW                93.600 usec
DE                  6.00 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.50 usec
PL1                 0.00 dB
SFO1         400.0923618 MHz

F2 - Processing parameters
SI                 32768
SF           400.0900125 MHz
WDW                   EM
SSB                    0
LB                  0.25 Hz
GB                     0
PC                  1.00
```



3m

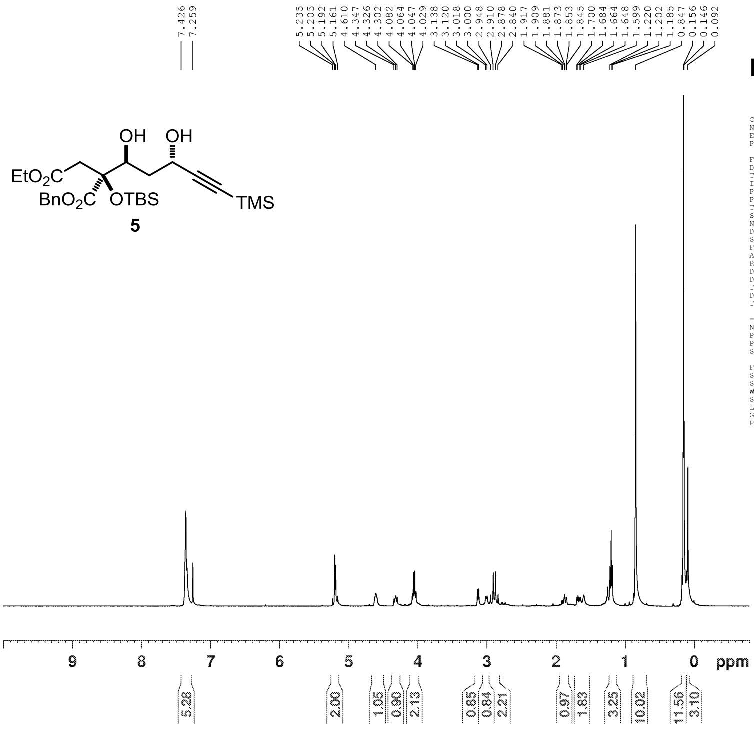
```
Current Data Parameters
NAME      JTM-6-45-2 13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100814
Time                5.51
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg30
TD                 32768
SOLVENT            cdcl3
NS                  6839
DS                     0
SWH            23980.814 Hz
FIDRES          0.731836 Hz
AQ             0.6832628 sec
RG                 16384
DW                20.850 usec
DE                  6.00 usec
TE                 300.0 K
D1            1.00000000 sec
d11           0.03000000 sec
DELTA         0.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  7.25 usec
PL1                 0.00 dB
SFO1         100.5348134 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -3.00 dB
PL12               16.72 dB
PL13               20.00 dB
SFO2         399.7815991 MHz

F2 - Processing parameters
SI                 32768
SF           100.5247622 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



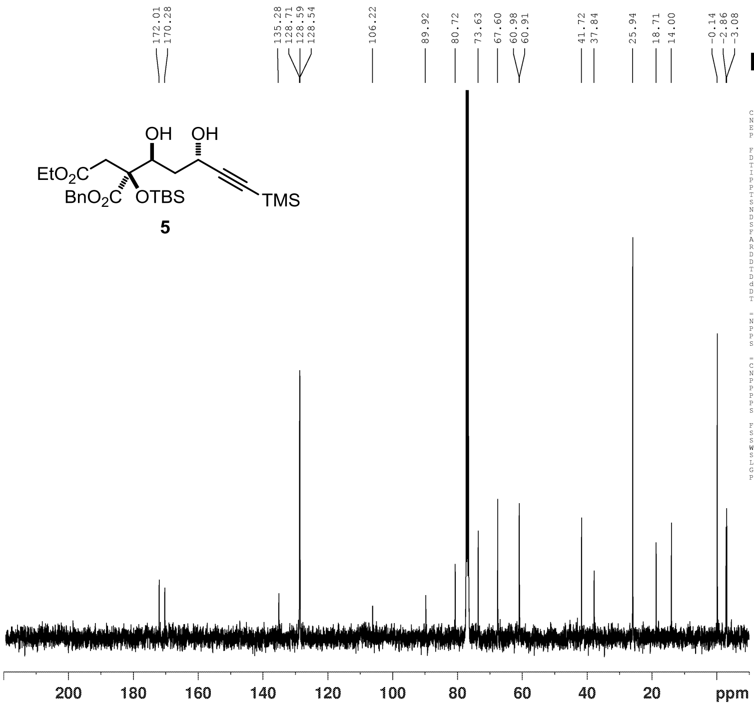
5

```
Current Data Parameters
NAME            SNGXIV-JTM-73-2-antidiol
EXPNO                             1
PROCNO                            1

F2 - Acquisition Parameters
Date_                      20100530
Time                          10.18
INSTRUM                       spect
PROBHD            5 mm QNP  1H/1
PULPROG                        zg30
TD                            32768
SOLVENT                     Acetone
NS                                8
DS                                0
SWH                        6218.905 Hz
FIDRES                     0.189786 Hz
AQ                        2.6345973 sec
RG                              181
DW                           80.400 usec
DE                             6.00 usec
TE                            300.0 K
D1                       1.00000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                             1H
P1                             9.30 usec
PL1                           -3.00 dB
SFO1                    399.7824920 MHz

F2 - Processing parameters
SI                            32768
SF                      399.7800101 MHz
WDW                              EM
SSB                               0
LB                             0.30 Hz
GB                                0
PC                             1.00
```



5

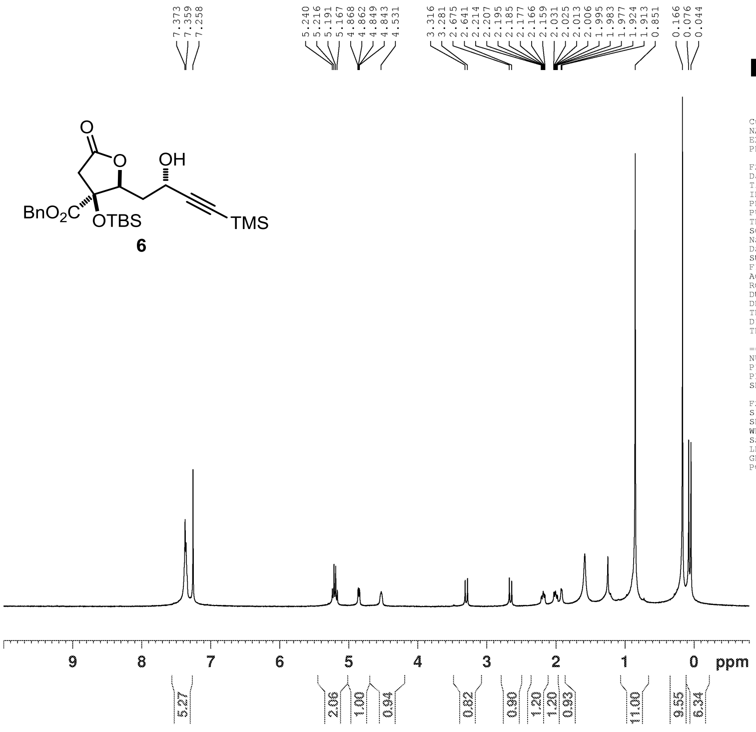
```
Current Data Parameters
NAME          SNGXIV-JTM-73-2-antidiolC13
EXPNO                             1
PROCNO                            1

F2 - Acquisition Parameters
Date_                      20100530
Time                          10.23
INSTRUM                       spect
PROBHD            5 mm QNP  1H/1
PULPROG                      zgpg30
TD                            32768
SOLVENT                       CDCl3
NS                             2807
DS                                0
SWH                       23980.814 Hz
FIDRES                     0.731836 Hz
AQ                        0.6832628 sec
RG                            16384
DW                           20.850 usec
DE                             6.00 usec
TE                            300.0 K
D1                       1.00000000 sec
d11                      0.03000000 sec
DELTA                    0.89999998 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                            13C
P1                             7.25 usec
PL1                            0.00 dB
SFO1                    100.5348134 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                         90.00 usec
PL2                           -3.00 dB
PL12                          16.72 dB
PL13                          20.00 dB
SFO2                    399.7815991 MHz

F2 - Processing parameters
SI                            32768
SF                      100.5247591 MHz
WDW                              EM
SSB                               0
LB                             2.00 Hz
GB                                0
PC                             1.40
```



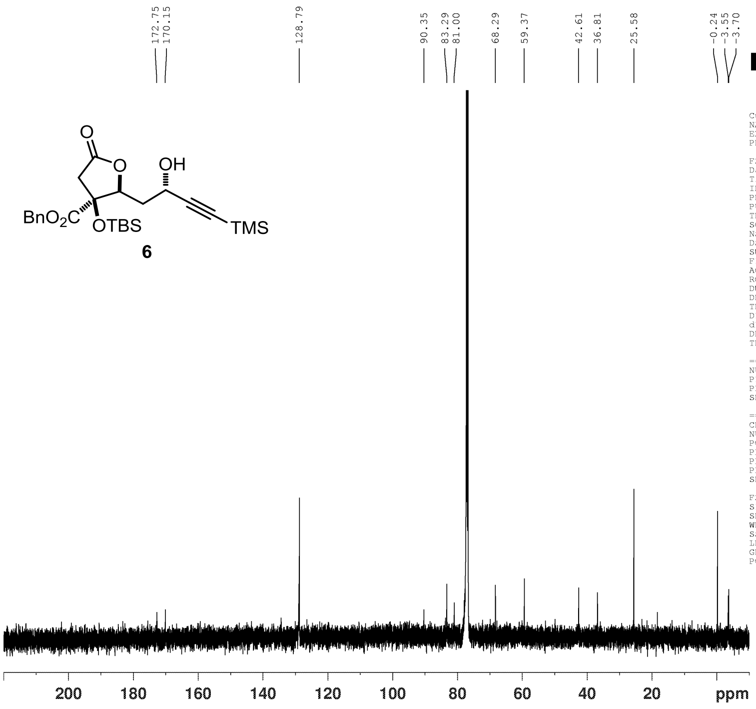
6

```
Current Data Parameters
NAME             SNG-monolactoneH1
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20100501
Time                         12.06
INSTRUM                      spect
PROBHD            5 mm PABBO BB/
PULPROG                       zg30
TD                           65536
SOLVENT                      CDCl3
NS                               8
DS                               0
SWH                       7507.507 Hz
FIDRES                    0.114555 Hz
AQ                       4.3647475 sec
RG                           203.2
DW                          66.600 usec
DE                            6.00 usec
TE                           292.4 K
D1                      1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                            1H
P1                           17.50 usec
PL1                          -3.00 dB
SFO1                   500.1330008 MHz

F2 - Processing parameters
SI                           32768
SF                     500.1300138 MHz
WDW                             EM
SSB                              0
LB                            0.25 Hz
GB                               0
PC                            1.00
```



6

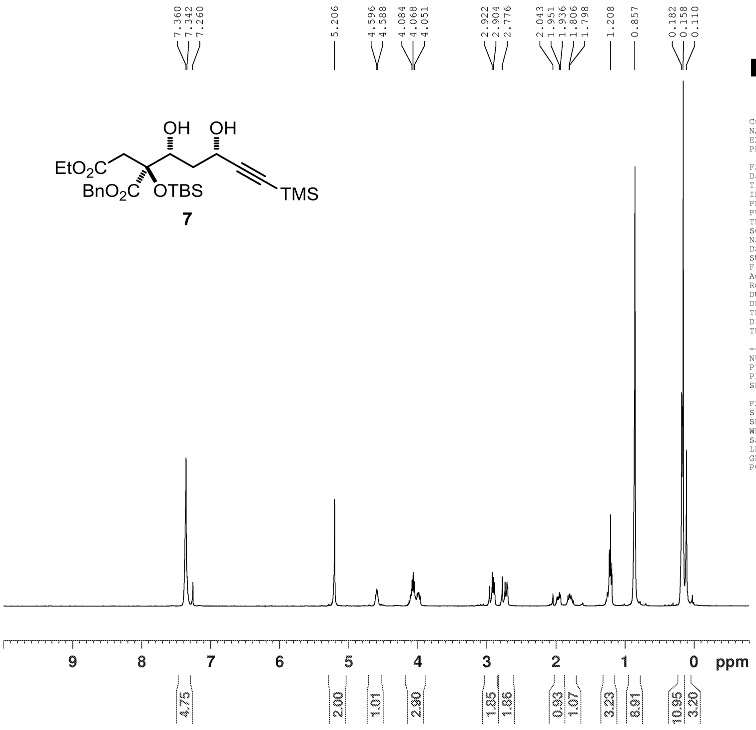
```
Current Data Parameters
NAME            SNG-monolactoneC13
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20100501
Time                         12.12
INSTRUM                      spect
PROBHD            5 mm PABBO BB/
PULPROG                     zgpg30
TD                           65536
SOLVENT                      CDCl3
NS                            1500
DS                               0
SWH                      30581.039 Hz
FIDRES                    0.466630 Hz
AQ                       1.0715636 sec
RG                           32768
DW                          16.350 usec
DE                            6.00 usec
TE                           293.4 K
D1                      1.00000000 sec
d11                     0.03000000 sec
DELTA                   0.89999998 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           13C
P1                            7.00 usec
PL1                           0.00 dB
SFO1                   125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                        70.00 usec
PL2                           0.00 dB
PL12                         14.30 dB
PL13                         18.00 dB
SFO2                   500.1320005 MHz

F2 - Processing parameters
SI                           65536
SF                     125.7577935 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```



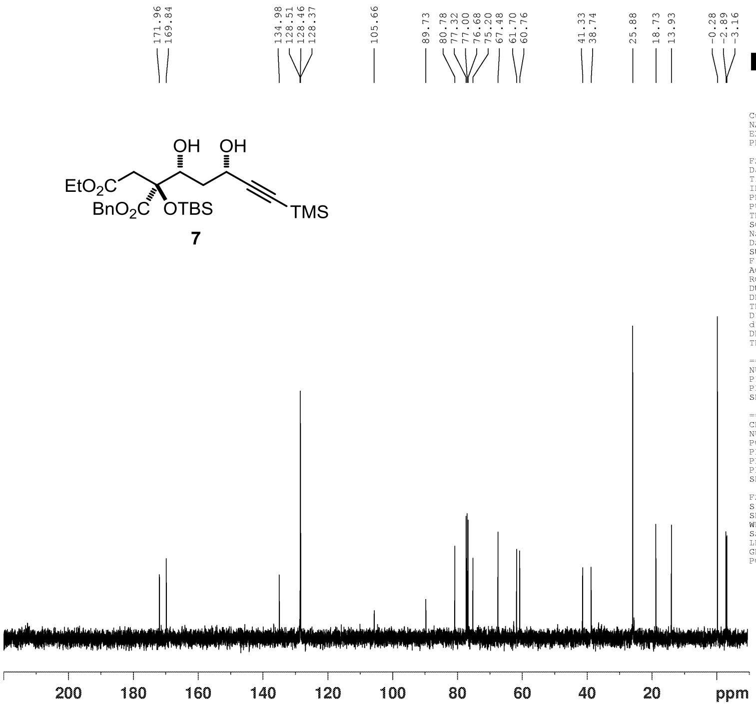
7

```
Current Data Parameters
NAME             SNGIX-33-prod
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090115
Time                      9.51
INSTRUM                  spect
PROBHD          5 mm QNP  1H/1
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   6218.905 Hz
FIDRES                0.189786 Hz
AQ                   2.6345973 sec
RG                        90.5
DW                      80.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.30 usec
PL1                      -3.00 dB
SFO1               399.8424924 MHz

F2 - Processing parameters
SI                       32768
SF                 399.8400072 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



7

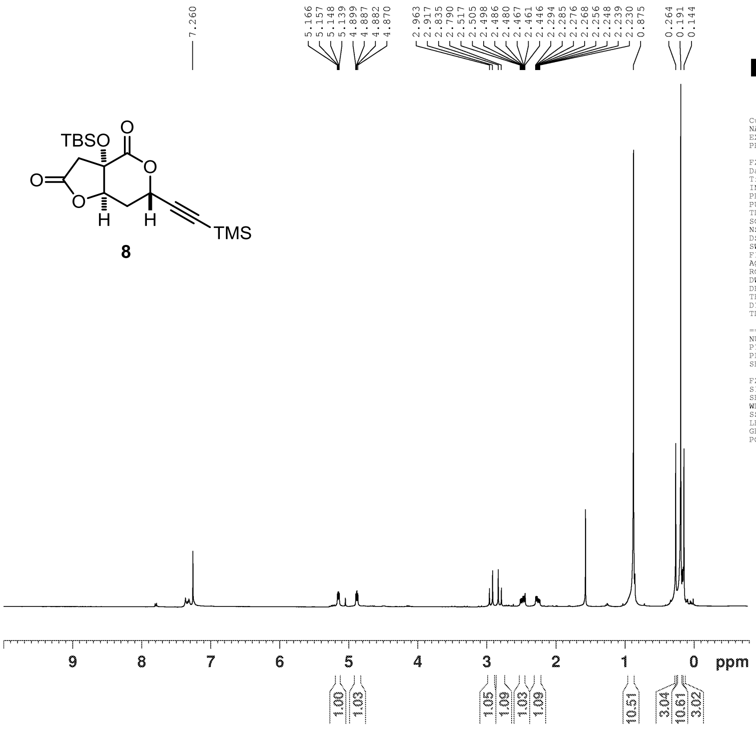
```
Current Data Parameters
NAME           SNGIX-33-prodc13
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090114
Time                     17.03
INSTRUM                  spect
PROBHD          5 mm TXI 13C Z
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         264
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                   1.3631988 sec
RG                       32768
DW                      20.800 usec
DE                        6.00 usec
TE                       293.7 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       20.00 usec
PL1                       0.00 dB
SFO1               100.6137773 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       0.00 dB
PL12                     17.64 dB
PL13                     23.00 dB
SFO2               400.0916004 MHz

F2 - Processing parameters
SI                       65536
SF                 100.6027215 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

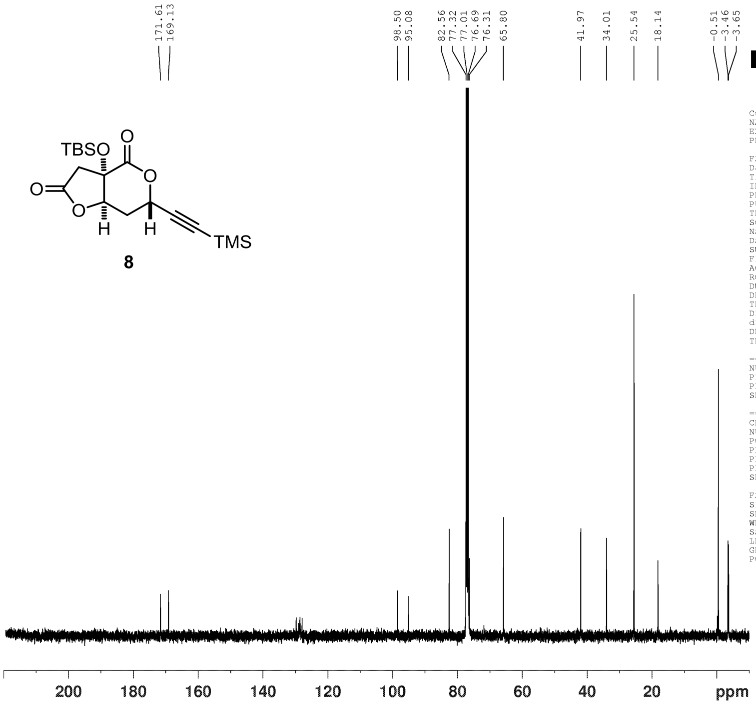


```
Current Data Parameters
NAME             SNGVIII-263-bislactone
EXPNO                             1
PROCNO                            1

F2 - Acquisition Parameters
Date_                      20081217
Time                          14.21
INSTRUM                       spect
PROBHD                 5 mm TXI 13C Z
PULPROG                        zg30
TD                            32768
SOLVENT                        DMSO
NS                               16
DS                                0
SWH                        5341.880 Hz
FIDRES                     0.163021 Hz
AQ                        3.0671349 sec
RG                              128
DW                           93.600 usec
DE                             6.00 usec
TE                            293.5 K
D1                       1.00000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                             1H
P1                            10.50 usec
PL1                            0.00 dB
SFO1                    400.0923618 MHz

F2 - Processing parameters
SI                            32768
SF                      400.0900033 MHz
WDW                              EM
SSB                               0
LB                             0.25 Hz
GB                                0
PC                             1.00
```



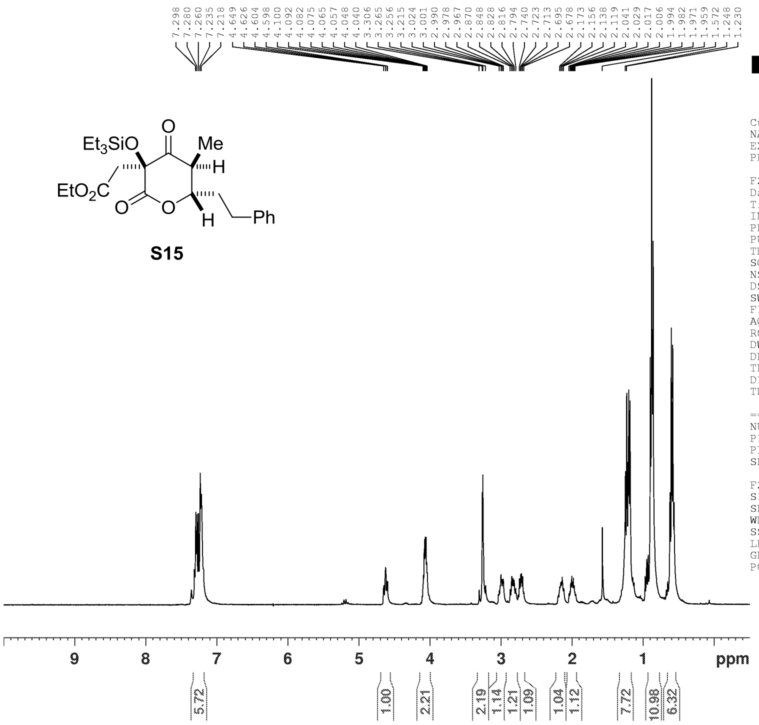
```
Current Data Parameters
NAME                SNGVIII-263-c13
EXPNO                             1
PROCNO                            1

F2 - Acquisition Parameters
Date_                      20081217
Time                          21.37
INSTRUM                       spect
PROBHD                5 mm QNP  1H/1
PULPROG                      zgpg30
TD                            65536
SOLVENT                       CDCl3
NS                            11200
DS                                2
SWH                       23980.814 Hz
FIDRES                     0.365918 Hz
AQ                        1.3664756 sec
RG                            32768
DW                           20.850 usec
DE                             6.00 usec
TE                            300.0 K
D1                       2.00000000 sec
d11                      0.03000000 sec
DELTA                    1.89999998 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                            13C
P1                             7.25 usec
PL1                            0.00 dB
SFO1                    100.5499020 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                         90.00 usec
PL2                           -3.00 dB
PL12                          16.72 dB
PL13                          20.00 dB
SFO2                    399.8415994 MHz

F2 - Processing parameters
SI                            32768
SF                      100.5398459 MHz
WDW                              EM
SSB                               0
LB                             1.00 Hz
GB                                0
PC                             1.40
```

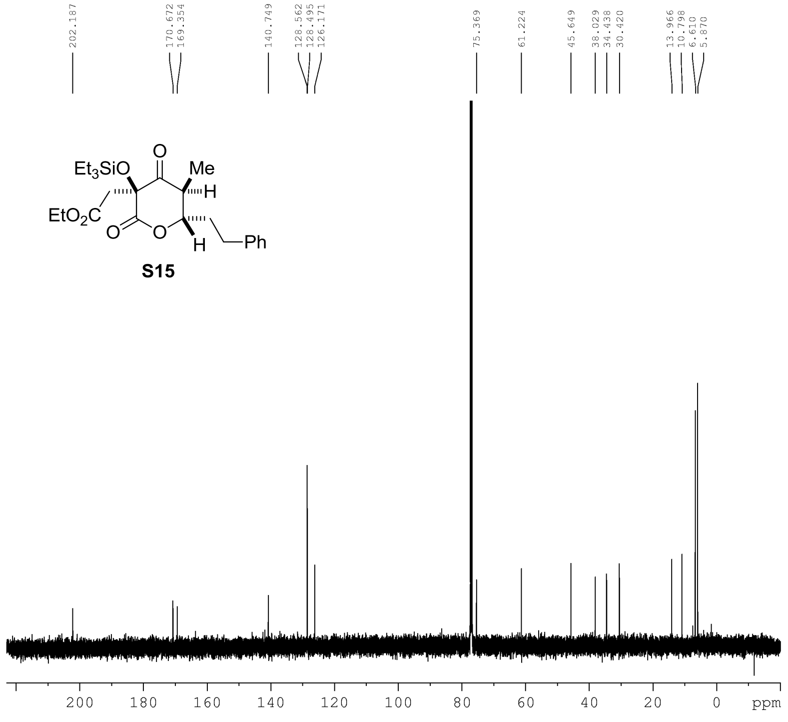


```
Current Data Parameters
NAME          JTM-6-197-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100925
Time                 9.37
INSTRUM             spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              6218.905 Hz
FIDRES           0.189786 Hz
AQ              2.6345973 sec
RG                  161.3
DW                 80.400 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.30 usec
PL1                 -3.00 dB
SFO1          399.7824920 MHz

F2 - Processing parameters
SI                  32768
SF            399.7800108 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



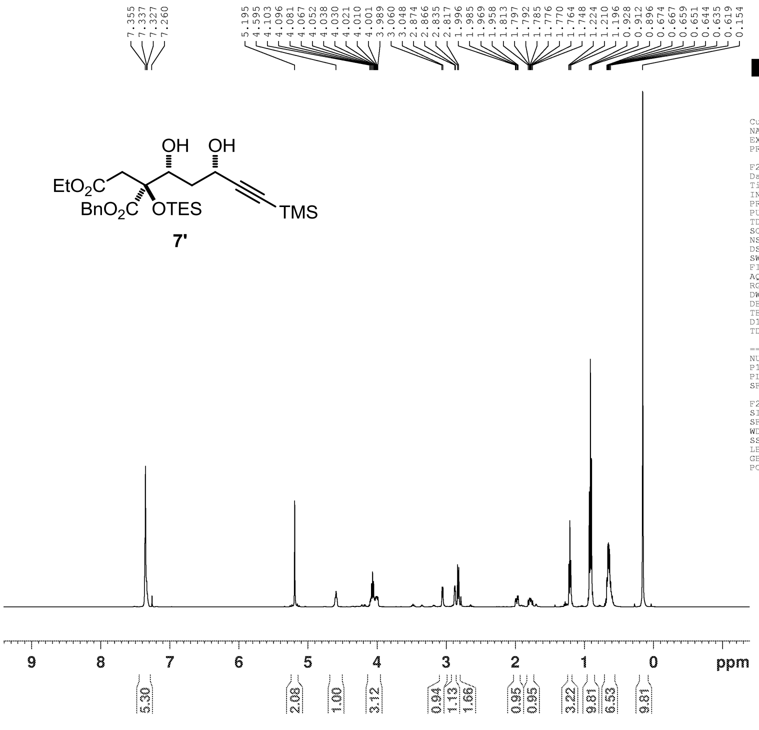
```
Current Data Parameters
NAME     JTM-6-195-2 13C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Paramete
Date_           20100923
Time               16.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            cdcl3
NS                  1469
DS                     0
SWH            30581.039 H
FIDRES          0.466630 H
AQ             1.0715799 s
RG               10321.3
DW                16.350 u
DE                  6.00 u
TE                 295.1 K
D1            1.00000000 s
d11           0.03000000 s
DELTA         0.89999998 s
TD0                    1

======== CHANNEL f1 =====
NUC1                 13C
P1                  6.80 u
PL1                 0.00 d
SFO1         125.7705462 M

======== CHANNEL f2 =====
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 u
PL2                -3.00 d
PL12               14.00 d
PL13               14.00 d
SFO2         500.1320005 M

F2 - Processing parameter
SI                 65536
SF           125.7577936 M
WDW                   EM
SSB                    0
LB                  0.50 H
GB                     0
PC                  1.40
```

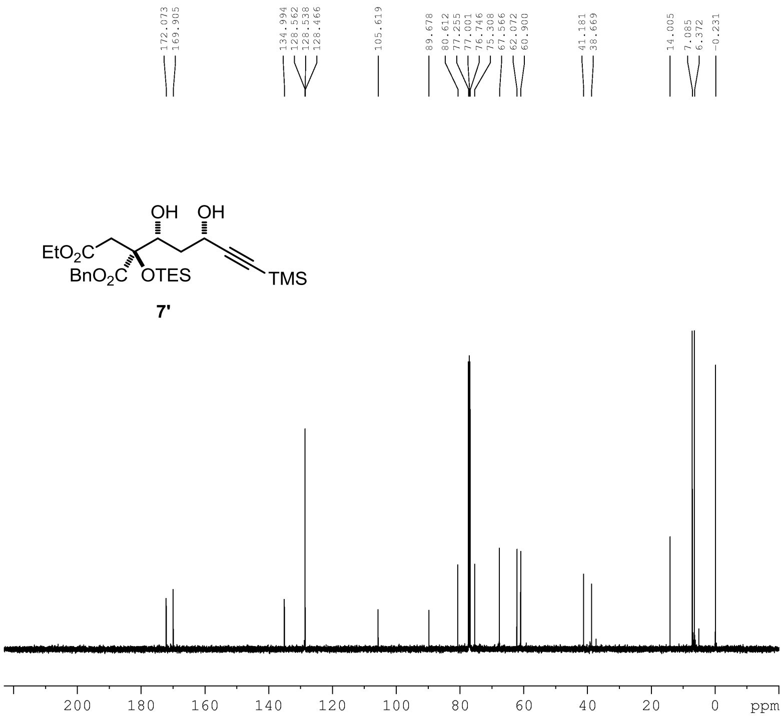


```
Current Data Parameters
NAME             SNGXI-139-prod
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20090722
Time                      11.04
INSTRUM                   spect
PROBHD         5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            0
SWH                    7507.507 Hz
FIDRES                 0.114555 Hz
AQ                    4.3647475 sec
RG                           32
DW                       66.600 usec
DE                         6.00 usec
TE                        292.5 K
D1                   1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                        13.50 usec
PL1                        0.00 dB
SFO1                500.1330008 MHz

F2 - Processing parameters
SI                        32768
SF                  500.1300136 MHz
WDW                          EM
SSB                           0
LB                         0.25 Hz
GB                            0
PC                         1.00
```



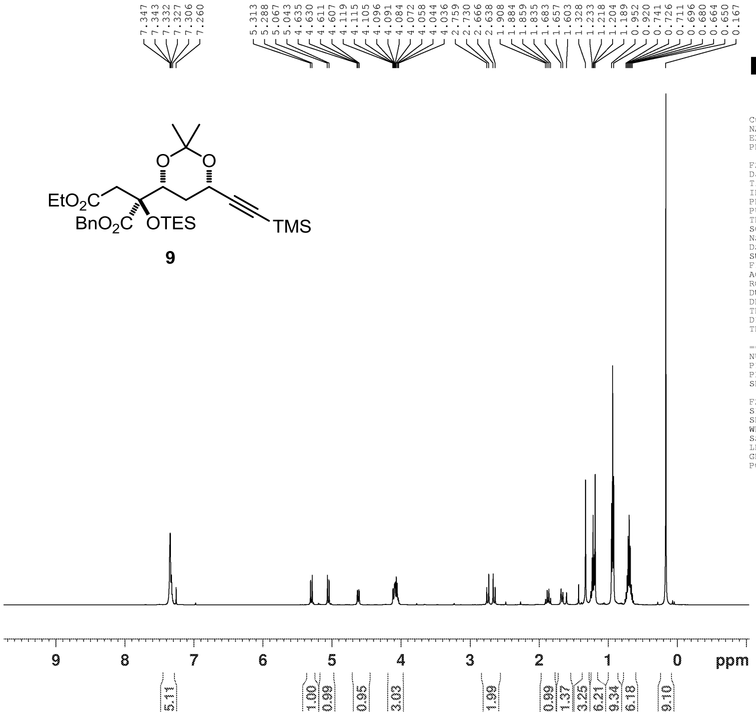
```
Current Data Parameters
NAME      SNGXI-139-prodc1.
EXPNO                   1
PROCNO                  1

F2 - Acquisition Paramete
Date_            20090722
Time                11.09
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   176
DS                     0
SWH            30581.039 H
FIDRES          0.466630 H
AQ             1.0715799 s
RG                 32768
DW                16.350 u.
DE                  6.00 u.
TE                 292.9 K
D1            1.00000000 s
d11           0.03000000 s
DELTA         0.89999998 s
TD0                    1

======== CHANNEL f1 =====
NUC1                 13C
P1                  7.00 u.
PL1                 0.00 d
SFO1         125.7705462 M

======== CHANNEL f2 =====
CPDPRG2          waltz16
NUC2                  1H
PCPD2              70.00 u.
PL2                 0.00 d
PL12               14.30 d
PL13               18.00 d
SFO2         500.1320005 M

F2 - Processing parameter.
SI                 65536
SF           125.7577900 M
WDW                   EM
SSB                    0
LB                  0.50 H.
GB                     0
PC                  1.40
```

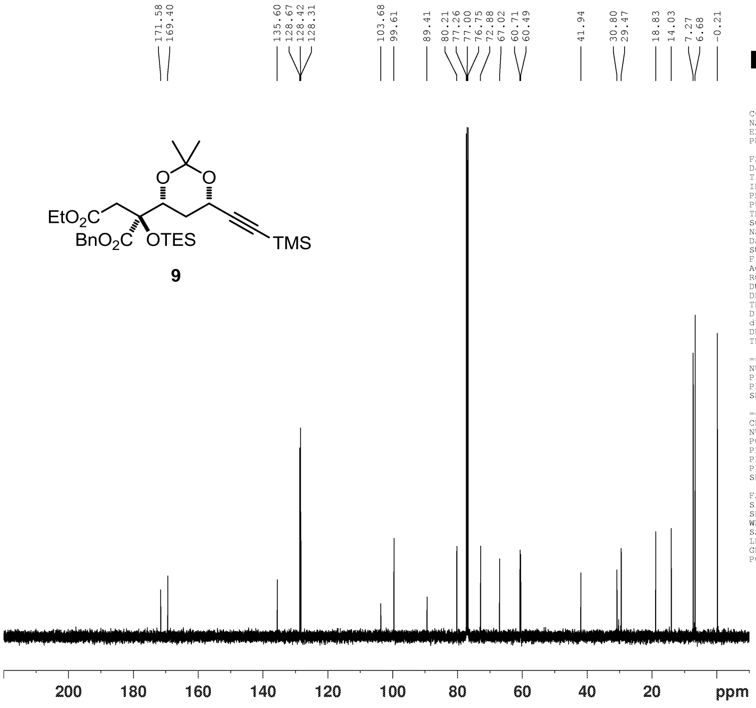


```
Current Data Parameters
NAME             SNGXI-acetonideH1
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20090727
Time                         16.13
INSTRUM                      spect
PROBHD             5 mm PABBO BB/
PULPROG                       zg30
TD                           65536
SOLVENT                      CDCl3
NS                              16
DS                               2
SWH                       7507.507 Hz
FIDRES                    0.114555 Hz
AQ                       4.3647475 sec
RG                              57
DW                          66.600 usec
DE                            6.00 usec
TE                           292.3 K
D1                      1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                            1H
P1                           13.50 usec
PL1                           0.00 dB
SFO1                   500.1330008 MHz

F2 - Processing parameters
SI                           32768
SF                     500.1300136 MHz
WDW                             EM
SSB                              0
LB                            0.25 Hz
GB                               0
PC                            1.00
```



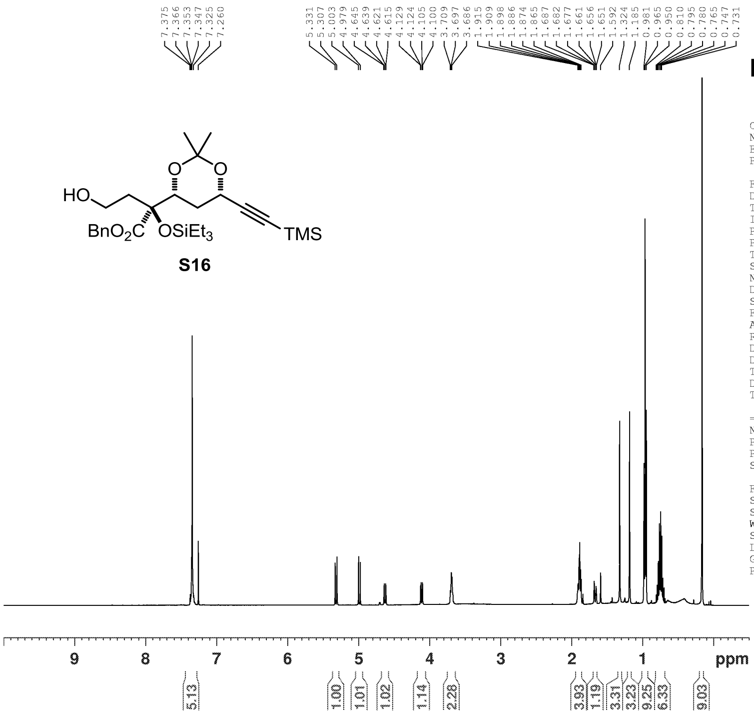
```
Current Data Parameters
NAME            SNGXI-acetonideC13
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20090727
Time                         16.20
INSTRUM                      spect
PROBHD             5 mm PABBO BB/
PULPROG                     zgpg30
TD                           65536
SOLVENT                      CDCl3
NS                             128
DS                               0
SWH                      30581.039 Hz
FIDRES                    0.466630 Hz
AQ                       1.0715636 sec
RG                           32768
DW                          16.350 usec
DE                            6.00 usec
TE                           293.7 K
D1                      1.00000000 sec
d11                     0.03000000 sec
DELTA                   0.89999998 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           13C
P1                            6.80 usec
PL1                           0.00 dB
SFO1                   125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                            1H
PCPD2                       100.00 usec
PL2                          -3.00 dB
PL12                         14.00 dB
PL13                         14.00 dB
SFO2                   500.1320005 MHz

F2 - Processing parameters
SI                           65536
SF                     125.7577951 MHz
WDW                             EM
SSB                              0
LB                            0.50 Hz
GB                               0
PC                            1.40
```

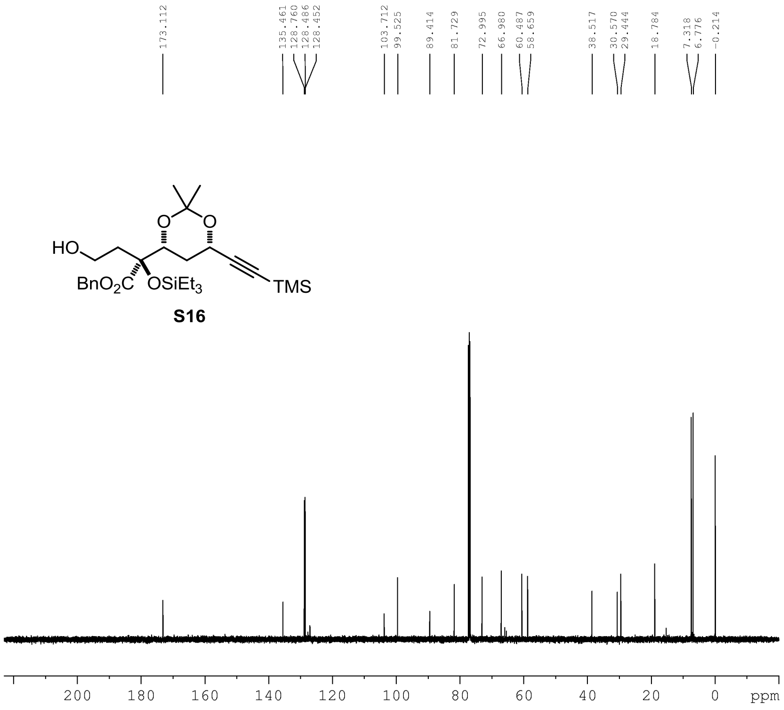


```
Current Data Parameters
NAME     JTM-6-53-2 xH1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20100819
Time              15.38
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            7507.507 Hz
FIDRES         0.114555 Hz
AQ            4.3648143 sec
RG                101.6
DW               66.600 usec
DE                 6.00 usec
TE                295.8 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.50 usec
PL1                0.00 dB
SFO1        500.1330008 MHz

F2 - Processing parameters
SI                32768
SF          500.1300134 MHz
WDW                  EM
SSB                   0
LB                 0.25 Hz
GB                    0
PC                 1.00
```



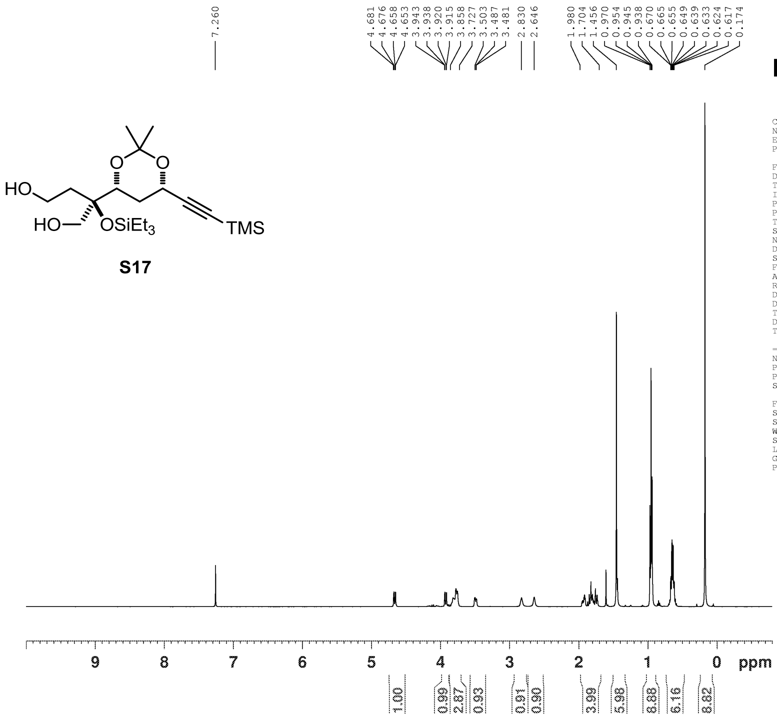
```
Current Data Parameters
NAME     JTM-6-53-2 C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Paramete
Date_          20100819
Time              13.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  189
DS                    0
SWH           30581.039 H
FIDRES         0.466630 H
AQ            1.0715799 s
RG              14596.5
DW               16.350 u
DE                 6.00 u
TE                296.0 K
D1           1.00000000 s
d11          0.03000000 s
DELTA        0.89999998 s
TD0                   1

======== CHANNEL f1 =====
NUC1                13C
P1                 7.00 u
PL1                0.00 d
SFO1        125.7705462 M

======== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2                 1H
PCPD2             70.00 u
PL2                0.00 d
PL12              14.30 d
PL13              18.00 d
SFO2        500.1320005 M

F2 - Processing parameter
SI                65536
SF          125.7577950 M
WDW                  EM
SSB                   0
LB                 0.50 H
GB                    0
PC                 1.40
```

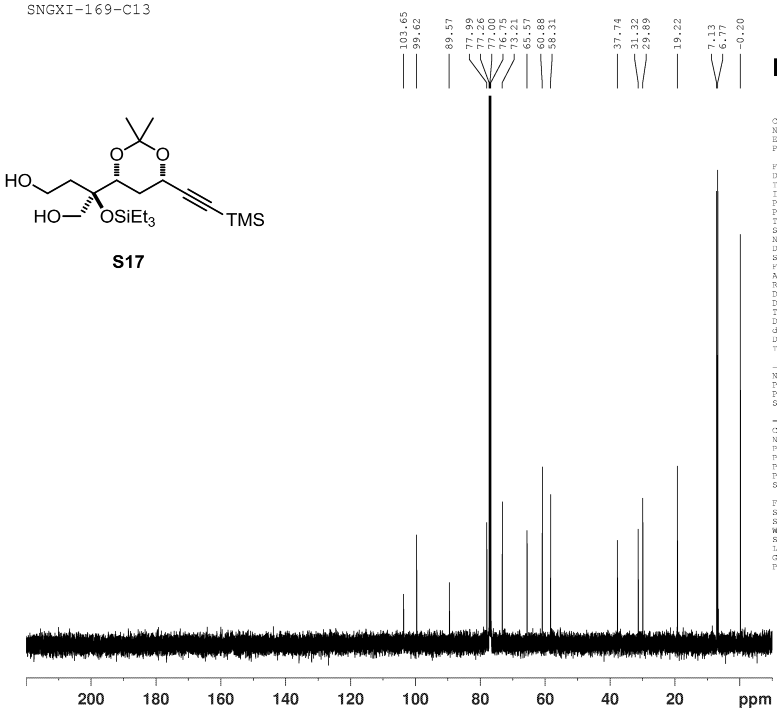


```
Current Data Parameters
NAME              SNGXI-169-h1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090731
Time                      8.17
INSTRUM                  spect
PROBHD        5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   7507.507 Hz
FIDRES                0.114555 Hz
AQ                   4.3647475 sec
RG                       161.3
DW                      66.600 usec
DE                        6.00 usec
TE                       292.4 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       13.50 usec
PL1                       0.00 dB
SFO1               500.1330008 MHz

F2 - Processing parameters
SI                       32768
SF                 500.1300135 MHz
WDW                         EM
SSB                          0
LB                        0.25 Hz
GB                           0
PC                        1.00
```



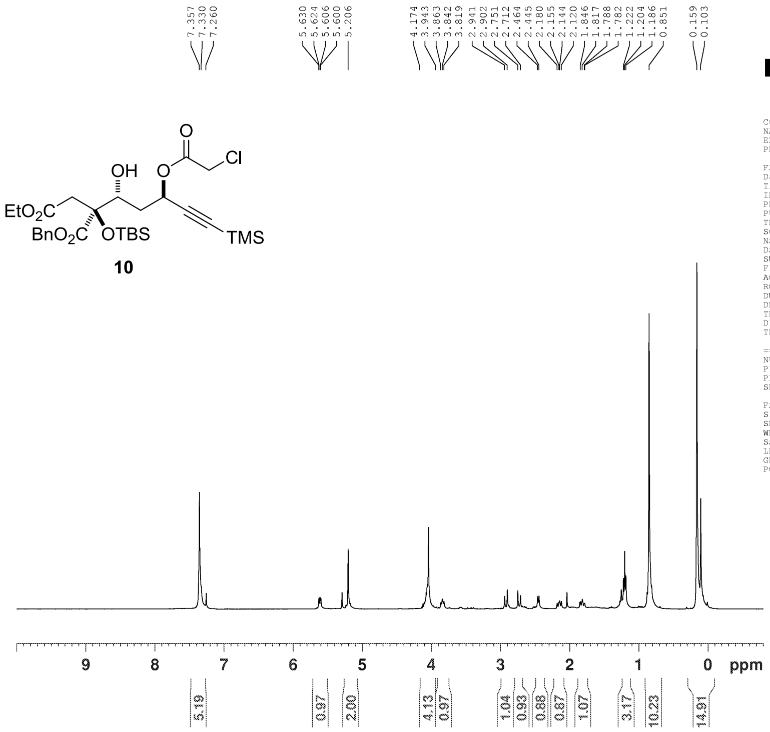
```
Current Data Parameters
NAME             SNGXI-169-c13
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090731
Time                      8.21
INSTRUM                  spect
PROBHD        5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         384
DS                           0
SWH                  30581.039 Hz
FIDRES                0.466630 Hz
AQ                   1.0715636 sec
RG                       32768
DW                      16.350 usec
DE                        6.00 usec
TE                       293.0 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        7.00 usec
PL1                       0.00 dB
SFO1               125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    70.00 usec
PL2                       0.00 dB
PL12                     14.30 dB
PL13                     18.00 dB
SFO2               500.1320005 MHz

F2 - Processing parameters
SI                       65536
SF                 125.7577936 MHz
WDW                         EM
SSB                          0
LB                        0.50 Hz
GB                           0
PC                        1.40
```

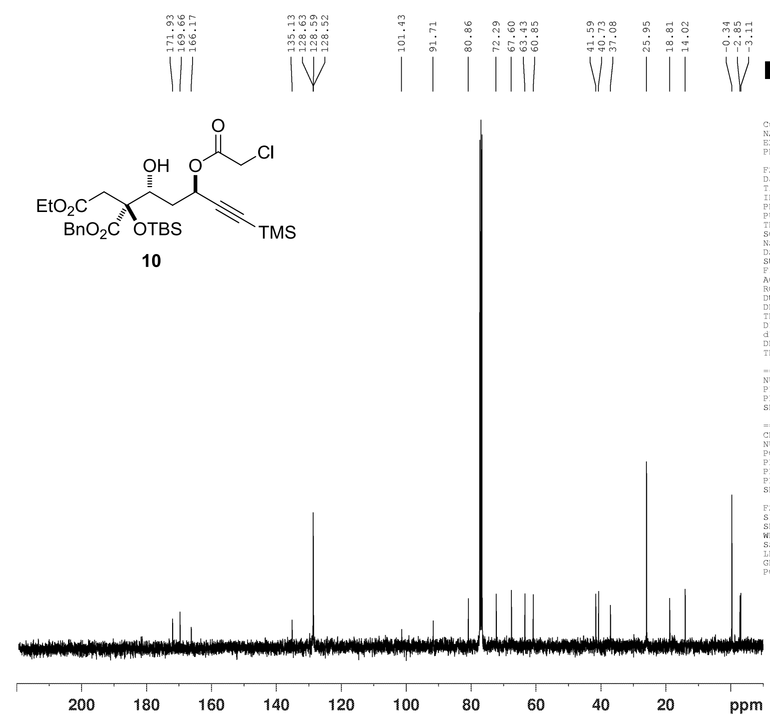


```
Current Data Parameters
NAME              SNG-221-prod
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100711
Time                     11.25
INSTRUM                  spect
PROBHD          5 mm QNP  1H/1
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   6218.905 Hz
FIDRES                0.189786 Hz
AQ                   2.6345973 sec
RG                         114
DW                      80.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.30 usec
PL1                      -3.00 dB
SFO1               399.7824920 MHz

F2 - Processing parameters
SI                       32768
SF                 399.7800109 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



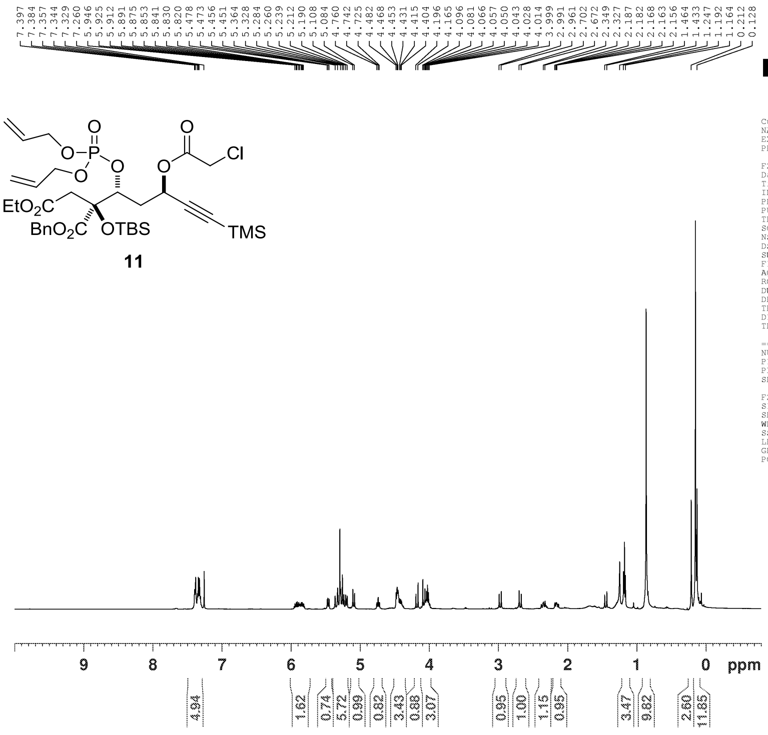
```
Current Data Parameters
NAME           SNG-221-prodC13
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100711
Time                     11.30
INSTRUM                  spect
PROBHD          5 mm QNP  1H/1
PULPROG                 zgpg30
TD                       32768
SOLVENT                  CDCl3
NS                        2150
DS                           0
SWH                  23980.814 Hz
FIDRES                0.731836 Hz
AQ                   0.6832628 sec
RG                       16384
DW                      20.850 usec
DE                        6.00 usec
TE                       300.0 K
D1                  1.00000000 sec
d11                 0.03000000 sec
DELTA               0.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        7.25 usec
PL1                       0.00 dB
SFO1               100.5348134 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     16.72 dB
PL13                     20.00 dB
SFO2               399.7815991 MHz

F2 - Processing parameters
SI                       32768
SF                 100.5247600 MHz
WDW                         EM
SSB                          0
LB                        1.50 Hz
GB                           0
PC                        1.40
```



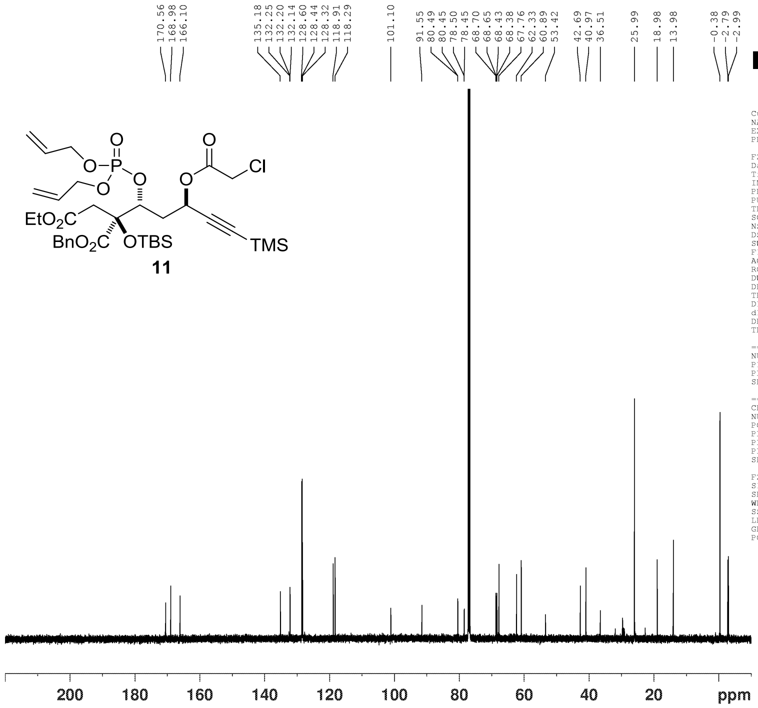
11

```
Current Data Parameters
NAME                SNG-227-phosphate-H1
EXPNO                              1
PROCNO                             1

F2 - Acquisition Parameters
Date_                       20100713
Time                           17.43
INSTRUM                        spect
PROBHD               5 mm PABBO BB/
PULPROG                         zg30
TD                             65536
SOLVENT                        CDCl3
NS                                16
DS                                 0
SWH                         7507.507 Hz
FIDRES                      0.114555 Hz
AQ                         4.3647475 sec
RG                               128
DW                            66.600 usec
DE                              6.00 usec
TE                             291.5 K
D1                        1.00000000 sec
TD0                                1

======== CHANNEL f1 ========
NUC1                              1H
P1                             13.50 usec
PL1                             0.00 dB
SFO1                     500.1330008 MHz

F2 - Processing parameters
SI                             32768
SF                       500.1300133 MHz
WDW                               EM
SSB                                0
LB                              0.25 Hz
GB                                 0
PC                              1.00
```



11

```
Current Data Parameters
NAME               SNG-227-phosphate-C13
EXPNO                              1
PROCNO                             1

F2 - Acquisition Parameters
Date_                       20100713
Time                           17.45
INSTRUM                        spect
PROBHD               5 mm PABBO BB/
PULPROG                      zgpg30
TD                             65536
SOLVENT                        CDCl3
NS                              1690
DS                                 0
SWH                        30581.039 Hz
FIDRES                      0.466630 Hz
AQ                         1.0715636 sec
RG                             32768
DW                            16.350 usec
DE                              6.00 usec
TE                             291.8 K
D1                        1.00000000 sec
d11                       0.03000000 sec
DELTA                     0.89999998 sec
TD0                                1

======== CHANNEL f1 ========
NUC1                             13C
P1                              7.00 usec
PL1                             0.00 dB
SFO1                     125.7705462 MHz

======== CHANNEL f2 ========
CPDPRG2                      waltz16
NUC2                              1H
PCPD2                          70.00 usec
PL2                             0.00 dB
PL12                           14.30 dB
PL13                           18.00 dB
SFO2                     500.1320005 MHz

F2 - Processing parameters
SI                             65536
SF                       125.7577945 MHz
WDW                               EM
SSB                                0
LB                              0.50 Hz
GB                                 0
PC                              1.40
```