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Supporting Information

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Rapid Total Syntheses Utilizing "Supersilyl" Chemistry**

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General Techniques.

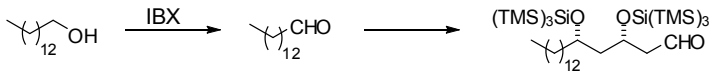
All non-aqueous reactions were carried out in flame-dried glassware under an atmosphere of dry nitrogen or argon and stirred via magnetic stir-plates. All reactions were carried out with anhydrous solvents unless otherwise noted. Anhydrous THF, CH_2Cl_2, Et_2O, and hexane were dried with an M BRAUN solvent purification system (A2 Alumina). Anhydrous DMF was purchased from Aldrich and used without further purification. Yields refer to chromatographically and spectroscopically (^{1}H NMR) homogeneous materials, unless otherwise stated. Silyl enol ethers **1**,[1] **8**,[2] and **14**[3] were prepared according to the literature procedures. Dimethylaluminum triflimide (Tf_2NAlMe_2) was prepared according to the literature method from Me_3Al and Tf_2NH.[4] The ruthenium metathesis catalyst, Zhan catalyst 1B, was purchased from Strem.

All reactions were monitored by thin–layer chromatography (TLC) carried out on 0.25 mm EMD silica gel plates (60F–254) using UV light (254 nm) with 2.4% phosphomolybdic acid/1.4% phosphoric acid/5% sulfuric acid in water, cerium sulfate in aqueous sulfuric acid, or anisaldehyde in ethanol and heat as developing agents. TSI silica gel (230–400 mesh) was used for flash chromatography.

Infrared spectra were recorded as thin films on sodium chloride plates using a Nicolet 20 SXB FTIR. ^{1}H NMR and ^{13}C spectra were recorded on a Bruker Avance 400 or a Bruker Avance 500. Chemical shift values (δ) are reported in ppm and calibrated to the residual solvent peak ($CDCl_3$ δ 7.26 ppm for ^{1}H, 77.0 ppm for ^{13}C; C_6D_6 δ 7.16 ppm for ^{1}H, 128.0 ppm for ^{13}C; CD_3OD δ 3.31 ppm for ^{1}H, 49.0 ppm for ^{13}C). The following abbreviations are used to indicate the multiplicities; s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; m, multiplet; br, broad; app, apparent.

Abbreviations: http://pubs.acs.org/paragonplus/submission/joceah/joceah_abbreviations.pdf

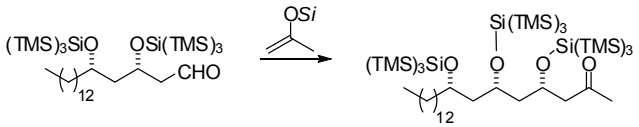
[1] Boxer, M. B.; Yamamoto, H. *Nature Protocols* **2006**, *1*, 2434.
[2] Boxer, M. B.; Akakura, M.; Yamamoto, H. *J. Am. Chem. Soc.* **2008**, *130*, 1580.
[3] Yamaoka, Y.; Yamamoto, H *J. Am. Chem. Soc.* **2010**, *132*, 5354.
[4] Marx, A.; Yamamoto, H. *Angew. Chem. Int. Ed.* **2000**, *39*, 178.



Preparation of 7 from 1-tetradecanol: The preparation of tetradecanal was accomplished by the IBX induced oxidation of commericially available 1-tetradecanol (our yield = 7.22 g, 85%), according to the literature.[5]

A stirred solution of **1** (4.92 g, 16.9 mmol) and tetradecanal (1.48 mg, 6.97 mmol) in CH_2Cl_2 (40 mL) was cooled to −40 °C, and then to the mixture was added Tf_2NAlMe_2 (0.10 M in CH_2Cl_2, 140 μL, 14 μmol) over a period of 2 min. The reaction mixture was stirred for 30 min at the same temperature then was quenched by the addition of pH 7 buffer (50 mL). The layers were separated, and the aqueous phase was extracted with CH_2Cl_2 then hexanes (10 mL each). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (250 mL) eluting with CH_2Cl_2/hexanes (1:9→1:4) to give **7** (4.68 g, 85%) as a colorless viscous oil.

Data for 7: R_f = 0.31 (1:4 CH_2Cl_2/hexanes); IR (neat): 2926, 2855, 1727 (C=O), 1467, 1395, 1245, 1084, 836 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, 293K) δ 9.77 (dd, 1H, *J* = 4.0, 1.1 Hz), 4.14–4.08 (m, 1H), 3.39 (app tt, 1H, *J* = 8.5, 4.3 Hz), 2.57 (ddd, 1H, *J* = 15.2, 4.7, 1.1 Hz), 2.36 (ddd, 1H, *J* = 15.2, 6.8, 4.0 Hz), 1.74 (ddd, 1H, *J* = 14.0, 9.0, 5.5 Hz), 1.64–1.56 (m, 1H), 1.47 (ddd, 1H, *J* = 13.2, 9.3, 4.0 Hz), 1.33–1.23 (m, 23H), 0.88 (t, 3H, *J* = 6.8 Hz), 0.19 (s, 27H), 0.18 (s, 27H); ^{13}C NMR (126 MHz, $CDCl_3$, 293K) δ 202.4, 73.4, 69.9, 49.5, 43.6, 37.9, 31.9, 29.9, 29.7 (3 carbons), 29.6 (2 carbons), 29.5, 29.4, 25.3, 22.7, 14.1, 0.7, 0.5; LRMS (APCI+) $C_{27}H_{61}O_2Si_4$ $[M - OSi(TMS)_3]^+$ 409.3 (26%).

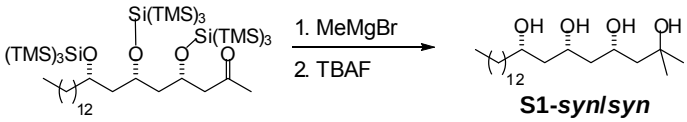


Preparation of 9 from 7: A stirred solution of aldehyde **7** (1.57 g, 1.98 mmol), silyl enol ether **8** (1.16 g, 3.81 mmol), and 1-iodo-2-phenylacetylene (43.1 mg, 0.189 mmol) in CH_2Cl_2 (10 mL) was cooled to −40 °C, and then to the mixture was added Tf_2NH (0.010 M in CH_2Cl_2, 600 μL, 6.0 μmol). The reaction mixture was stirred for 1 h at the same temperature then was quenched by the addition of pH 7 buffer (10 mL). The layers were separated, and the aqueous phase was extracted with CH_2Cl_2 (5 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography[6] on silica gel (150 mL)

[5] Wiseman, J. M.; McDonald, F. E.; Liotta, D. C. *Org. Lett.* **2005**, *7*, 3155.
[6] This diasteromer was separated from its minor diastereomers (major R_f = 0.39), with 1 or 2 chromatographies.

eluting with CH_2Cl_2/hexanes (1:9→1:4) to give **9** (1.71 g, 78% along with 19% of its diastereomers) as a colorless viscous oil.

Data for 9: R_f = 0.29 (1:4 CH_2Cl_2/hexanes); IR (neat): 2947, 1724 (C=O), 1438, 1379, 1244, 1070, 1005, 836, 689 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, 293K) δ 4.12 (br app tt, 1H, *J* = 8.7, 4.8 Hz), 3.67 (app tt, 1H, *J* = 9.3, 4.7 Hz), 3.42 (br app tt, 1H, *J* = 8.6, 4.3 Hz), 2.51 (dd, 1H, *J* = 14.8, 8.6 Hz), 2.41 (dd, 1H, *J* = 14.8, 4.4 Hz), 2.12 (s, 3H), 1.78 (ddd, 1H, *J* = 12.9, 9.2, 5.7 Hz), 1.68 (ddd, 1H, *J* = 12.9, 9.4, 5.9 Hz), 1.64−1.56 (m, 1H), 1.37 (ddd 1H, *J* = 13.0, 8.4, 4.7 Hz), 1.35−1.19 (m, 24H), 0.88 (t, 3H, *J* = 6.9 Hz), 0.193 (s, 27H), 0.181 (s, 27H), 0.177 (s, 27H); ^{13}C NMR (126 MHz, $CDCl_3$, 303K) δ 206.3, 73.7, 70.3, 69.7, 50.3, 45.5, 44.9, 38.3, 32.0, 31.5, 29.9, 29.6 (5 carbons), 29.5, 29.4, 26.0, 22.7, 14.1, 0.85, 0.79, 0.71; LRMS (APCI+) $C_{39}H_{93}O_3Si_8$ $[M - OSi(TMS)_3]^+$ 833.4 (9%), $C_{30}H_{65}O_2Si_4$ $[M - OSi(TMS)_3 - HOSi(TMS)_3]^+$ 569.4 (38%).



S1-*syn*/*syn*

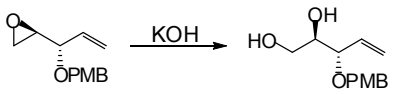
Preparation of tetraol S1-*syn/syn* from 9 for the determination of the relative stereochemistry: A stirred solution of **9** (220 mg, 0.200 mmol) in THF (1.0 mL) was cooled to −78 °C, then to this mixture was added MeMgBr (3.0 M in Et_2O, 350 μL, 0.405 mmol) dropwise. After 30 min the reaction was warmed to 0 °C, and after another 60 min at the same temperature, the reaction was warmed to 23 °C. After 75 additional min the reaction mixture was quenched by the addition of sat. aq. NH_4Cl (4 mL). The layers were separated, and the aqueous phase was extracted with hexanes (2 × 3 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (10 mL) eluting with CH_2Cl_2/hexanes (1:9→1:2) to give the alcohol (155 mg, 70%) as a colorless oil.

A stirred solution of this alcohol (151 mg, 0.135 mmol) in THF (0.80 mL) and MeOH (80 μL) was cooled to 0 °C, then to this mixture was added TBAF (1.0 M in THF, 200 μL, 0.20 mmol) dropwise over 3 min. After an additional 2 min at the same temperature, the reaction mixture was warmed to 23 °C. After an additional 40 min at the same temperature, AcOH (50 μL) was added and the resulting solution was concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (20 mL) eluting with *i*-PrOH/hexanes (1:19→1:7) to give **S1-*syn/syn*** (51 mg, quant.) as a colorless viscous oil.

Data for S1-*syn/syn*:[†] R_f = 0.38 (1:4 *i*-PrOH/hexanes); IR (neat): 3370 (br, O-H), 2919, 2850, 1467, 1325, 1115, 1089, 847 cm^{-1}; ^1H NMR (500 MHz, 5% $CDCl_3$ in CD_3OD, 293K) δ 4.15 (app tt, 1H, *J* = 7.5, 5.1 Hz), 3.97 (app tt, 1H, *J* = 8.3, 4.4 Hz), 3.77–3.71 (m, 1H), 1.64–1.50 (m, 5H), 1.48–1.38 (m, 3H), 1.34–1.24 (m, 25H), 1.23 (s, 3H), 0.88 (t, 3H, *J* = 6.9 Hz); ^{13}C NMR (126 MHz, 5% $CDCl_3$ in CD_3OD, 293K) δ 71.9, 71.5, 70.4, 69.0, 46.1, 44.8, 38.6, 32.9, 30.9, 30.6 (8 carbons), 30.3, 28.7, 26.3, 23.5, 14.4; LRMS (APCI–) $C_{22}H_{46}{}^{35}ClO_4$ [M + $^{35}Cl]^-$ 409.3 (100%), $C_{22}H_{46}{}^{37}ClO_4$ [M + $^{35}Cl]^-$ 411.2 (37%).

[†] This compound was confirmed to be *syn/syn* by the Kishi method for 1,3,5,7-tetraols (the 3- and 5-carbon chemical shifts are ~69 and ~70 ppm, then a *syn/syn* relationship exists).[7]

Preparation of 10. See *Angew. Chem. Int. Ed.* **2008**, 47, 7520 for the epoxidation reaction. Then see *J. Am. Chem. Soc.* **1990**, *112*, 5583 for the *para*-methoxybenzyl protection reaction (our yield = 90%).

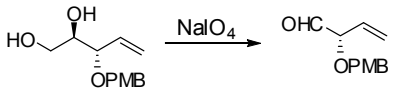


S2

Preparation of S2. A solution of epoxide **10** (1.11 g, 5.02 mmol) in DMSO (10 mL) and H_2O (10 mL) was stirred at 23 °C under an air atmosphere. To this solution was added KOH (1.12 g, 20.0 mmol), and after 5 min at the same temperature, the reaction mixture was warmed to 75 °C. After an additional 3 h at the same temperature, the reaction mixture was cooled to 23 °C, and it was subsequently poured onto aqueous HCl solution (0.1 N, 200 mL). The resulting mixture was extracted with EtOAc (6 × 25 mL). The combined extracts were dried over Na_2SO_4, filtered though cotton, and concentrated under reduced pressure. The resulting residue was purified by flash chromatography on silica gel (25 mL) eluting with EtOAc/hexanes (1:4→3:2) to afford **S2** as a colorless oil (1.11 g, 93%).

Data for S2: R_f = 0.13 (1:1 EtOAc/hexanes); $[\alpha]_D^{23}$ = +36.1 (*c* 1.44, MeOH); IR (neat): 3407 (br, O-H), 3075, 2934, 2871, 1613, 1514, 1248, 1035, 821 cm^{-1}; ^1H NMR (500 MHz, 293K, CD_3OD) δ 7.25 (br d, 1H, *J* = 8.8 Hz), 6.88 (br d, 1H, *J* = 8.8 Hz), 5.85 (ddd, 1H, *J* = 17.6, 10.4, 8.0 Hz), 5.36 (d, 1H, *J* = 10.4 Hz), 5.32 (d, 1H, *J* = 17.6 Hz), 4.52 (d, 1H, *J* = 11.3 Hz), 4.32 (d, 1H, *J* = 11.3 Hz), 3.82–3.76 (m, 4H), 3.66–3.61 (m, 2H), 3.51 (dd, 1H, 12.2, 7.8 Hz); ^{13}C NMR (126 MHz, 293K, CD_3OD) δ 160.7, 136.8, 131.7, 130.6, 119.9, 114.7, 82.5, 75.2, 71.2, 64.3, 55.7; LRMS (APCI+) $C_{13}H_{17}O_4$ $[M - H]^+$ 237.1 (100%), C_8H_9O $[PMB]^+$ 121.1 (68%).

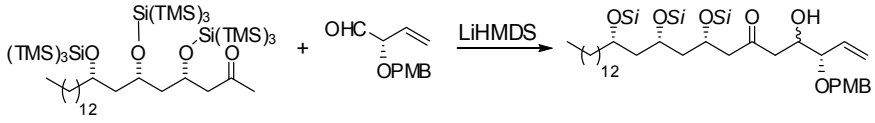
[7] Kobayashi, Y.; Tan, C.-H.; Kishi, Y. *Helv. Chim. Acta* **2000**, *83*, 2562.



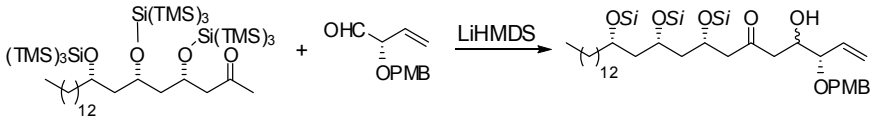
Preparation of 11. A solution of diol **S2** (360 mg, 1.50 mmol) in THF (7.5 mL) and H_2O (10 mL) was stirred at 0 °C under an air atmosphere. To this solution was added $NaIO_4$ (390 mg, 1.82 mmol), and after 5 min at the same temperature, the reaction mixture was warmed to 23 °C. After an additional 1 h at the same temperature, the reaction mixture was diluted with H_2O (25 mL). The resulting mixture was extracted with Et_2O/heanes (9:1 v/v, 4 × 10 mL). The combined extracts were dried over Na_2SO_4, filtered though cotton, and concentrated under reduced pressure. The resulting crude **11**, unstable to flash chromatography, was obtained colorless oil (294 mg, 95%) and used without further purification.

Data for 11: R_f = 0.34→0.50 (1:9 EtOAc/hexanes); $[\alpha]_D^{23}$ = +13.2 (*c* 1.26, CH_2Cl_2); IR (neat): 3001, 2936, 2837, 1736 (C=O), 1613, 1514, 1249, 1076, 1034, 934, 822 cm^{-1}; ^1H NMR (500 MHz, 293K, $CDCl_3$) δ *J* = 9.53 (d, 1H, *J* = 1.0 Hz), 7.29 (ddd, 1H, *J* = 9.5, 3.0, 2.0 Hz), 6.90 (ddd, 1H, *J* = 9.5, 3.0, 2.0 Hz), 5.76 (ddd, 1H, *J* = 17.3, 10.5, 6.5 Hz), 5.49 (ddd, 1H, *J* = 17.3, 1.5, 1.5 Hz), 5.45 (ddd, 1H, *J* = 10.5, 1.5, 1.3 Hz), 4.63 (d, 1H, *J* = 11.5 Hz), 4.52 (d, 1H, *J* = 11.5 Hz), 4.27 (app dq, 1H, *J* = 6.5, 1.5 Hz), 3.81 (s, 3H); ^{13}C NMR (126 MHz, 293K, $CDCl_3$) δ 200.0, 159.5, 130.8, 129.7, 129.0, 120.7, 113.9, 84.4, 71.1, 55.3; LRMS (APCI+) $C_{12}H_{13}O_3$ $[M - H]^+$ 205.1 (5%), C_8H_9O $[PMB]^+$ 121.1 (100%).

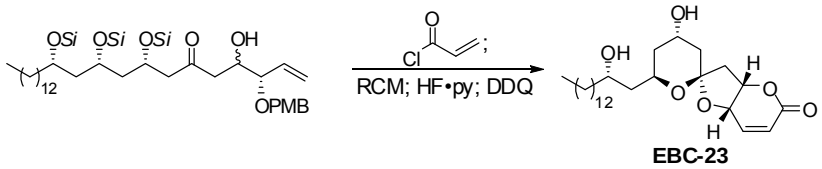
Table S1. Optimization of the aldol reaction of **9** and **11**.



Entry	Solvent(s)	Temp. (°C)	Yield (%) of 12	dr
1	DMF	−65	6	48:44:6:2
2	DMF/THF (5:1)	−65	10	48:44:6:2
3	THF	−78	56	47:38:12:3
4	Et_2O/DMF (19:1)	−78	43	47:40:10:3
5	*t*-BuOMe/DMF (19:1)	−78	36	47:40:10:3
6	CH_2Cl_2/DMF (19:1)	−78	29	48:43:7:2
7	toluene/DMF (19:1)	−78	63	48:43:7:2
8	CyMe/DMF (19:1)	−78	50	48:44:6:2



Preparation of 12 (Table S1, entry 7): A solution of ketone **9** (221 mg, 0.201 mmol) in toluene (1.6 mL) was stirred at −40 °C, and to this solution was added LiHMDS (1.0M in hexanes, 240 µL, 0.240 mmol). After 10 min at the same temperature, the reaction mixture was warmed to 0 °C. After an additional 2 h at the same temperature, the reaction was cooled to −78 °C, then to it was added DMF (100 µL, 1.29 mmol) in toluene (150 µL, then 50 µL rinse) down the flask side. After an additional 10 min, a solution of **11** in toluene (150 µL, then 50 µL rinse) was added down the flask side. After an additional 1 h at −78 °C, the mixture was quenched with sat. aq. NH_4Cl (5 mL). The resulting mixture was extracted with CH_2Cl_2 (3 mL). The combined organic layers were dried over Na_2SO_4, filtered though cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (150 mL) eluting with CH_2Cl_2/hexanes/EtOAc (10:90:0→20:78:2) to give **12** (166 mg, 63% as a 48:43:7:2 mixture of diastereomers) as a colorless viscous oil and returned **9** (70.0 mg, 32%).



EBC-23

Preparation of EBC-23 from ketoalcohol 12: A solution of ketoalcohol **12** (261 mg, 0.200 mmol) in THF (0.80 mL) was stirred at −78 °C, and to this solution was added LiHMDS (1.0M in hexanes, 220 µL, 0.220 mmol). After 30 min at the same temperature, acryloyl chloride (24 µL, 0.30 mmol) was added dropwise. After an additional 30 min at the same temperature, the reaction was warmed to ambient temperature and concentrated under high vacuum (4 mm Hg).

The residue was dissolved in toluene (5.0 mL), and to the solution was added Zhan-1B (2.9 mg, 4.0 µmol) in one portion. The mixture was then heated to 95 °C, and after 15 and 26 h more Zhan-1B was added (2.9 and 3.0 mg = 8.8 mg, 12 µmol total). After a total of 48 h at 95 °C, the mixture was cooled to ambient temperature and concentrated under high vacuum (4 mm Hg).

The residue was dissolved in THF (2.0 mL), and the stirred solution was cooled to 0 °C. To the mixture was added pyridine (50 µL, 0.62 mmol) followed by HF•py (70% v/v HF, 200 µL). After 2 h at the same temperature, the reaction was warmed to 23 °C. After an additional 2 h at the same temperature, the reaction was quenched by the addition of sat. aq. $NaHCO_3$ (10 mL). The resulting

mixture was extracted with Et_2O (4 × 5 mL). The combined extracts were dried over Na_2SO_4, filtered though cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (10 mL) eluting with hexanes/EtOAc (2:3→1:9) to give a mixture of hemiketal anomers **S3** (39.5 mg, 33%).

Hemiketals **S3** (27.9 mg, 0.0472 mmol) in CH_2Cl_2 (380 μL) and H_2O (95 μL) was rapidly stirred at 0 °C under an atmosphere of air, and to this mixture was added 2,3-dichloro-5,6-dicyanobenzoquinone (24.5 mg, 0.108 mmol). After 5 min at the same temperature, the reaction was warmed to 23 °C. After an addition 2.5 h at this temperature, the mixture was diluted with H_2O (5 mL) and sat. aq. $NaHCO_3$ (5 mL). The resulting mixture was extracted with Et_2O (4 × 5 mL). The combined extracts were dried over Na_2SO_4, filtered though cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (6 mL) eluting with hexanes/EtOAc (2:3→1:9) to give **EBC-23** (15.4 mg, 72%) as a white powder.

Data for EBC-23: R_f = 0.39 (3:2 CH_2Cl_2/EtOAc), 0.31 (1:4 hexanes/EtOAc); $[\alpha]_D^{24}$ +15.4 (*c* 0.20, $CHCl_3$); IR (neat): 3524, 3479, 2918, 2852, 1727 (C=O), 1470, 1280, 1243, 1103, 1067 cm^{-1}; LRMS (APCI−) $C_{26}H_{44}{}^{35}ClO_6$ [M + $^{35}Cl]^+$ 487.1 (100%), $C_{26}H_{44}{}^{37}ClO_6$ [M + $^{37}Cl]^+$ 489.1 (40%).

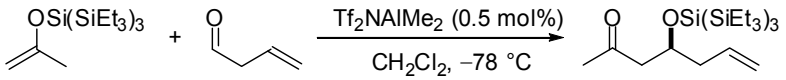
Table S2. Comparison of the isolated, Williams' and our ^{1}H NMR data for EBC-23.

Natural EBC-23 ^{1}H NMR Data: 750 MHz, $CDCl_3$	Williams' ^{1}H NMR Data: 500 MHz, $CDCl_3$	Our ^{1}H NMR Data: 500 MHz, $CDCl_3$
6.89 (dd, 1H, *J* = 10.0, 5.2 Hz)	6.87−6.90 (dd, 1H, *J* = 9.9, 5.1 Hz)	6.91 (dd, 1H, *J* = 9.9, 5.2 Hz)
6.21 (d, 1H, *J* = 10.0 Hz)	6.22−6.20 (dd, 1H, *J* = 9.9 Hz)	6.23 (d, 1H, *J* = 9.9 Hz)
5.04 (ddd, 1H, *J* = 6.9, 4.5, 2.5 Hz)	5.04−5.02 (ddd, 1H, *J* = 6.9, 4.5, 2.6 Hz)	5.06 (ddd, 1H, *J* = 6.9, 4.6, 2.5 Hz)
4.51 (dd, 1H, *J* = 5.2, 4.5 Hz)	4.51 −4.49 (t, 1H, *J* = 4.8 Hz)	4.52 (app t, 1H, *J* = 4.9 Hz)
4.40−4.35 (m, 1H)	4.39−4.34 (m, 1H)	4.43−4.37 (m, 1H)
4.13−4.09 (m, 1H)	4.11 (m, 1H)	4.16−4.10 (m, 1H)
3.82−3.76 (m, 1H)	3.81−3.76 (m, 1H)	3.84−3.78 (m, 1H)
3.05 (d, 1H, *J* = 9.2 Hz)	3.05 (br s, 1H)	3.08 (br s, 1H)
2.54 (dd, 1H, *J* = 14.9, 6.9 Hz)	2.56−2.51 (dd, 1H, *J* = 14.9, 6.8 Hz)	2.56 (dd, 1H, *J* = 14.9, 6.9 Hz)
2.30 (dd, 1H, *J* = 14.9, 2.5 Hz)	2.31−2.27 (dd, 1H, *J* = 15.0, 2.6 Hz)	2.32 (dd 1H, *J* = 14.9, 2.5 Hz)
2.05-1.98 (m, 2H)	2.05−1.97 (m, 2H)	2.08−1.99 (m, 2H)
1.80-1.75 (m, 1H)	1.79−1.75 (m, 1H)	1.82−1.76 (m, 1H)
1.65-1.56 (m, 2H)	1.65−1.56 (m, 3H)	1.68−1.58 (m, 2H)
1.52-1.47 (m, 1H)	1.52−1.47 (m, 2H)	1.58−1.35 (m, 3H)
1.47-1.42 (m, 1H)		
1.42-1.35 (m, 2H)		
1.33-1.20 (m, 21H)	1.23 (s, 23H)	1.32−1.20 (m, 23H)
0.86 (t, 3H, *J* = 7.1 Hz)	0.84 (t, 3H, *J* = 14.0[8] Hz)	0.88 (t, 3H, *J* = 7.0 Hz)

[8] This *J* value is assumed to be a typographic error. Their data for the isolated material indicated a *J* value of 7.1 Hz.

Table S3. Comparison of the isolated, Williams' and our ^{13}C NMR data for EBC-23.

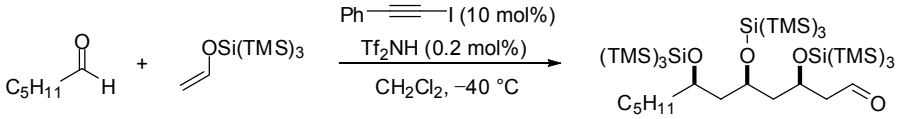
Natural EBC-23 ^{13}C NMR Data: 125 MHz, $CDCl_3$	Williams' ^{13}C NMR Data: 125 MHz, $CDCl_3$	Our ^{13}C NMR Data: 126 MHz, $CDCl_3$
161.0	161.0	161.0
138.6	138.6	138.6
124.6	124.6	124.6
106.6	106.6	106.6
78.8	78.8	78.8
71.8	71.8	71.9
68.9	68.9	68.9
67.6	67.8	67.8
64.2	64.2	64.2
47.7	47.7	47.7
42.2	42.2	42.2
38.8	38.7	38.7
37.7	37.7	37.7
31.9	31.9	31.9
29.67	29.67	29.68
29.65	29.66	29.67
29.63	29.64	29.65
29.61	29.62	29.62
29.58	29.59	29.60
29.3	29.3	29.3
25.4	25.4	25.4
22.7	22.7	22.7
14.1	14.1	14.1



Preparation of 15 from 3-butenal: The preparation of 3-butenal was prepared from glyoxal according to the literature procedure.[9]

A stirred solution of 3-butenal (40.0 g, 15.0 mmol, 1/37 w/w in CH_2Cl_2) and silyl enol ether **14** (5.27 g, 12.2 mmol) in CH_2Cl_2 (120 mL) was cooled to −78 °C, and then to the mixture was added Tf_2NAlMe_2 (0.10 M in CH_2Cl_2, 610 μL, 0.061 μmol). The reaction mixture was stirred for 12 h at 0 °C then was quenched by the addition of pH 7 buffer (10 mL). The layers were separated, and the aqueous phase was extracted with CH_2Cl_2 (20 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (100 mL) eluting with CH_2Cl_2/hexanes (1:7→1:5) to give **15** (4.57 g, 75%) as a colorless semisolid.

Data for 15: R_f = 0.42 (1:4 CH_2Cl_2/hexanes); IR (neat): 2952, 2909, 2875. 1717, 1416 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, 293K) δ 5.79−5.66 (m, 1H), 5.08−4.94 (m, 2H), 3.92−3.85 (m, 1H), 2.61 (dd, *J* = 16.4, 8.0 Hz), 2.47 (dd, *J* = 16.4, 4.4 Hz), 2.32−2.18 (m, 2H), 1.04 (t, 27H, *J* = 7.9 Hz), 0.77 (q, 18H, *J* = 7.9 Hz); ^{13}C NMR (126 MHz, $CDCl_3$, 293K) δ 207.2, 134.1, 117.7, 72.2, 49.5, 41.0, 31.3, 8.8, 5.4; LRMS (ES+) $C_{25}H_{56}O_2Si_4$ $[M + Na]^+$ 523.5 (100%).

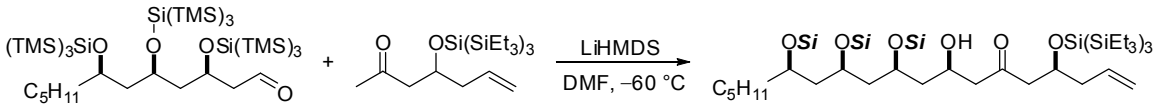


Preparation of 16: A stirred solution of silyl enol ether **1** (2.90 g, 10.0 mmol), *n*-hexanal (240 μL, 2.00 mmol) and 1-iodo-2-phenylacetylene (45.3 mg, 0.199 mmol) in CH_2Cl_2 (10.0 mL) was cooled to −40 °C, and then to the mixture was added Tf_2NH (0.010 M in CH_2Cl_2, 400 μL, 4.0 μmol). The reaction mixture was stirred for 30 min at the same temperature then was quenched by the addition of pH 7 buffer (5 mL). The layers were separated, and the aqueous phase was extracted with CH_2Cl_2 (5 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (100 mL) eluting with CH_2Cl_2/hexanes (1:20→1:7) to give **16** (1.66 g, 85%, dr = 80:12:5:3) as a white foam.

Data for 16: R_f = 0.52 (1:4 CH_2Cl_2/hexanes); IR (neat): 2949, 2894, 1733 (C=O), 1438, 1375 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, 293K) δ 9.94 (dd, 1H, *J* = 2.9, 1.3 Hz), 4.49 (m, 1H), 3.95 (m, 1H), 3.67 (m, 1H), 2.71 (ddd, 1H, *J* = 15.4, 3.9, 1.2 Hz), 2.63 (ddd, 1H, *J* = 15.3, 8.7, 3.1 Hz), 2.06 (ddd, 1H, *J* = 12.8,

[9] Crimmins, M. T.; Kirincich, S. J.; Wells A. J.; Choy, A. L. *Synth. Commun.* **1998**, *28*, 3675.

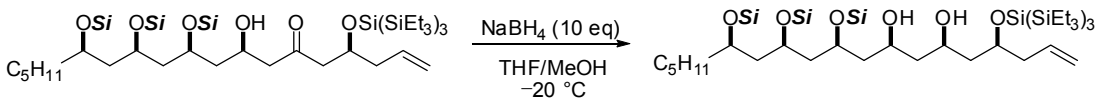
9.7, 5.4 Hz), 1.98 (ddd, 1H, *J* = 12.9, 9.9, 5.6 Hz), 1.93−1.84 (m, 1H), 1.74 (ddd, 1H, *J* = 13.2, 9.0, 4.5 Hz), 1.60 (ddd, 1H, *J* = 12.5, 8.9, 3.4 Hz), 1.50−1.36 (m, 7H), 0.94 (t, 3H, *J* = 6.9 Hz), 0.33 (s, 27H), 0.324 (s, 27H), 0.318 (s, 27H); ^{13}C NMR (126 MHz, $CDCl_3$, 293K) δ 199.8, 73.9, 70.17, 70.15, 50.5, 46.1, 45.0, 38.9, 32.4, 26.2, 23.0, 14.1, 1.0, 0.9, 0.8; LRMS (ES+) $C_{39}H_{102}NaO_4Si_{12}$ $[M + Na]^+$ 993.7 (7%), $C_{39}H_{103}NaO_4Si_{12}$ $[M + H]^+$ 971.7 (10%).



Preparation of 17. To a stirred solution of ketone **15** (446 mg, 0.890 mmol) and $LiBF_4$ (418 mg, 4.45 mmol) in DMF (9.0 mL) was added LiHMDS (1.0 M in hexane, 1.1 mL, 1.1 mmol) at –40 °C. After being stirred at same temperature for 30 min, the solution was cooled to –60 °C. After an additional 5 min at the same temperature, the toluene solution (1 mL) of the aldehyde **16** (1.73 g, 1.78 mmol, dr = 87:13) was slowly added. The reaction mixture was stirred for 1 h at the same temperature then was quenched by the addition of water (1 mL). The reaction mixture was diluted with hexanes (20 mL) and the organic phase was washed with brine (2 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (100 mL) eluting with CH_2Cl_2/hexanes (1:7→1:5) to give **17** (506 mg, 39%) as a white foam and other diastereomers (340 mg, 25%, R_f ~ 0.45, dr ~ 73:20:7).[10]

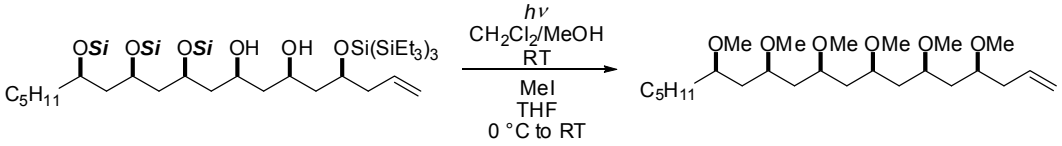
Data for 17: R_f = 0.27 (1:4 CH_2Cl_2/hexanes); IR (neat): 2950, 2875, 1716, 1461, 1244, 836 cm^{-1}; ^{1}H NMR (500 MHz, C_6D_6, 293K) δ 6.00−5.87 (m, 1H), 5.15−5.05 (m, 2H), 4.53−4.43 (m, 1H), 4.26-4.14 (m, 2H), 4.02−3.94 (m, 1H), 3.74−3.64 (m, 1H), 3.63 (br s, 1H), 2.90−2.77 (m, 1H), 2.75−2.62 (m, 2H), 2.55−2.62 (m, 3H), 2.00 (ddd, 1H, *J* = 12.6, 6.5 Hz), 1.92 (ddd, 1H, *J* = 12.7, 6.4 Hz), 1.87−1.77 (m, 3H), 1.73−1.65 (m, 1H), 1.64−1.57 (m, 1H), 1.49−1.36 (m, 7H), 1.16 (t, 27H, *J* = 7.8 Hz), 0.97−0.91 (m, 21H), 0.35 (s, 27H), 0.334 (s, 27H), 0.325 (s, 27H); ^{13}C NMR (126 MHz, C_6D_6, 338K) δ 207.1, 134.7, 117.8, 74.2 (2 carbons), 72.6, 70.4, 67.5, 52.1, 50.2, 46.1, 45.7, 43.9, 41.7, 38.8, 32.6, 26.1, 14.0, 9.1, 6.0, 1.2, 1.1 (2 carbons); LRMS (ES+) $C_{64}H_{158}NaO_6Si_{16}$ $[M + Na]^+$ 1496.3 (5%), $C_{25}H_{56}O_2Si_4$ [**15** + Na]$^+$ 523.5 (100%).

[10] Fortunately, the all-syn diastereomer **17** was easily separated from the mixture. Unfortunately, a more accurate diastereomeric ratio cannot be given due to the poor separation of the minor diastereomers. Since an excess of aldehyde was employed and 39% yield of **17** was obtained, a matched 1,3- and 1,5-selective reaction probably occurred.



Preparation of 18. To a stirred solution of $NaBH_4$ (36.0 mg, 0.952 mmol) in MeOH (1 mL) was added the THF (1 mL) solution of **17** (140 mg, 0.095 mmol) at −20 °C. After being stirred at same temperature for 12h, the reaction mixture was diluted with hexanes (5 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (10 mL) eluting with CH_2Cl_2/hexanes (1:7→1:4) to give **18** (128 mg, 89%) as a white foam.

Data for 18: R_f = 0.22 (1:4 CH_2Cl_2/hexanes); IR (neat): 2940, 2875, 1432, 1244, 835 cm^{-1}; ^1H NMR (500 MHz, C_6D_6, 293K) δ 6.21−6.09 (m, 1H), 5.22−5.12 (m, 2H), 4.61−4.50 (m, 1H), 4.41−4.33 (m, 1H), 4.32−4.16 (m, 3H), 4.15−4.06 (m, 1H), 4.01−3.91 (m, 1H), 3.67−3.58 (m, 1H), 2.72−2.60 (m, 1H), 2.59-2.48 (m, 1H), 2.07-1.98 (m, 2H), 1.97−1.87 (m, 4H), 1.84−1.75 (m, 2H), 1.73−1.64 (m, 2H), 1.60−1.53 (m, 1H), 1.47−1.34 (m, 1H), 1.19 (t, 27H, *J* = 7.8 Hz), 1.02−0.94 (m, 21H), 0.32 (s, 81H); ^{13}C NMR (126 MHz, C_6D_6, 338K) δ 135.5, 116.9, 75.7, 74.4, 74.1, 72.9, 70.4, 69.5, 46.21, 46.19, 45.5, 44.4, 44.3, 40.7, 39.2, 32.5, 26.2, 23.0, 13.9, 9.1, 6.1, 1.13 (2 carbons), 1.06; LRMS (ES+) $C_{64}H_{160}NaO_6Si_{16}$ $[M + Na]^+$ 1498.3 (10%), 345.5 (100%) .



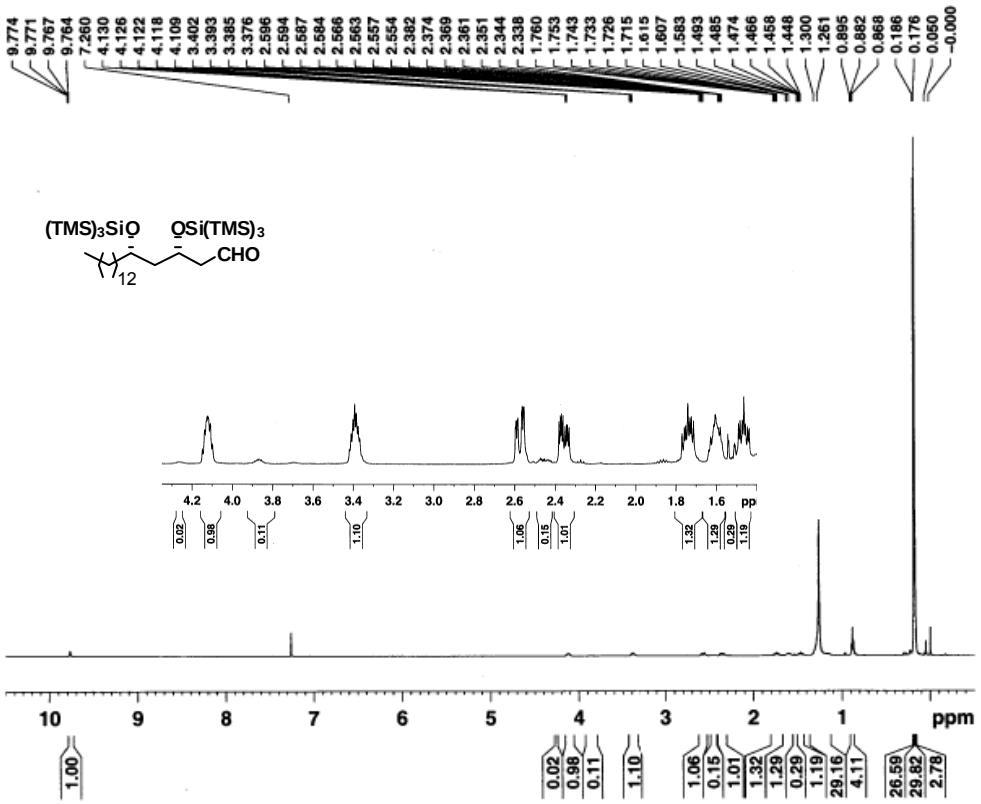
Preparation of 13. To a stirred solution of **18** (100 mg, 0.0678 mmol) in MeOH/CH_2Cl_2 (4:1) (5.0 mL) was irradiated in a quartz flask at room temperature. After being stirred for 12h, the solvent was removed under reduced pressure. The crude mixture was used without purification in the next step.

To a stirred solution of NaH (56.0 mg, 1.34 mmol) in THF (1 mL) was a solution of the crude mixture (THF, 1 mL) at 0 °C. After being stirred at same temperature for 10 min, and then to the mixture was added MeI (89.3 μL, 2.68 mmol). The reaction mixture was stirred for 12 h at room temperature then was quenched by the addition of water (1 mL). The layers were separated, and the aqueous phase was extracted with AcOEt (5 mL). The combined organic layers were dried over Na_2SO_4, filtered through cotton, and concentrated under reduced pressure. The resulting residue was purified by flask chromatography on silica gel (5 mL) eluting with AcOEt/hexanes (1:5→1:3) to give **13** (18.3 mg, 61%) as a colorless oil.

Data for 13[11,12]**:** R_f = 0.31 (1:3 AcOEt/hexanes); IR (neat): 2929, 2820, 1457, 1379, 1188, 1092 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, 293K) δ 5.88−5.76 (m, 1H), 5.18−5.04 (m, 2H), 3.34−3.26 (m, 6H), 3.34 (s, 3H), 3.32 (s, 3H), 3.312 (s, 6H), 3.308 (s, 6H), 2.41−2.23 (m, 2H), 1.88−1.76 (m, 4H), 1.64−1.45 (m, 8H), 1.39−1.22 (m, 6H), 0.90 (t, 3H, *J* = 6.9 Hz); ^{13}C NMR (126 MHz, $CDCl_3$, 293K) δ 134.4, 117.3, 77.9, 77.3, 75.4, 75.29, 75.28, 75.27, 56.4, 56.3, 56.24, 56.23, 56.21, 56.18, 38.16, 38.15, 38.1, 37.9, 37.64, 37.55, 33.4, 32.1, 24.6, 22.7, 14.1; LRMS (ES+) $C_{25}H_{50}O_6$ $[M + Na]^+$ 469.5 (100%).

[11] Liu, K.; Arico, J. W.; Taylor, R. E. *J. Org. Chem.* **2010**, *75*, 3953; ^1H, ^{13}C data reported in $CDCl_3$, which matches our data.
[12] Mori, Y.; Kohchi, Y.; Suzuki, M.; Carmeli, S.; Moore, R. E.; Paterson, G. M. *J. Org. Chem.* **1991**, *56*, 631; ^1H, ^{13}C data reported in C_6D_6, which also matches our data (not shown).

^{1}H NMR for 7: $CDCl_3$, 500 MHz, 293K

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F2 - Processing parameters
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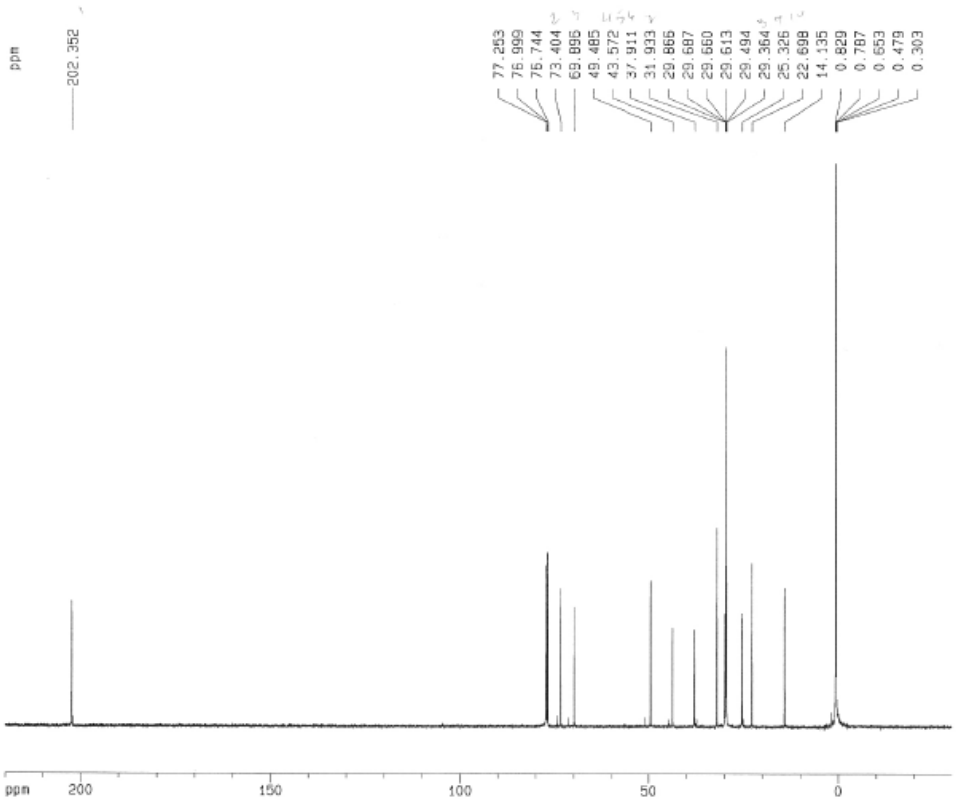
^{13}C NMR for 7: $CDCl_3$, 126 MHz, 293K

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P1                     8.00 usec
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PL2                   120.00 dB
PL12                   20.00 dB
SFO2            500.1330000 MHz

F2 - Processing parameters
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1D NMR plot parameters
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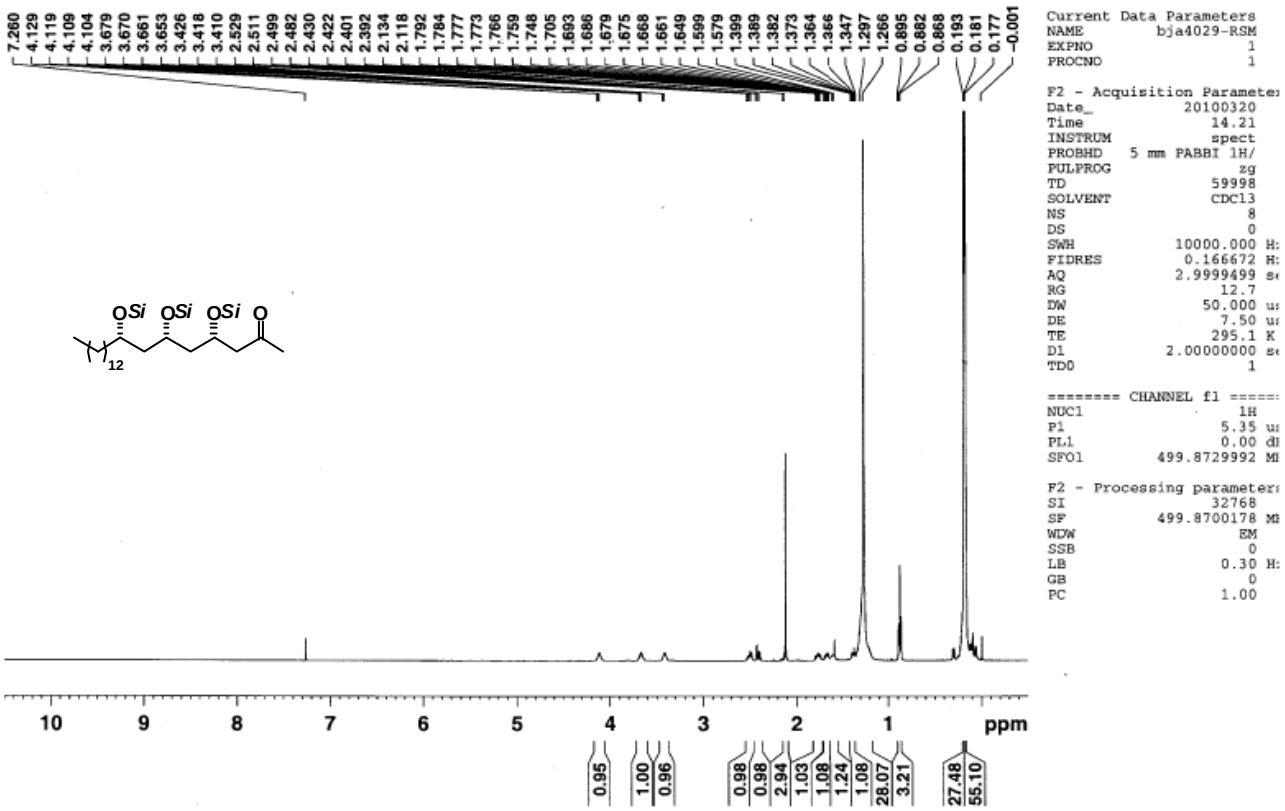
^{1}H NMR for 9: $CDCl_3$, 500 MHz, 293K



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TD 59998
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RG 12.7
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F2 - Processing parameter
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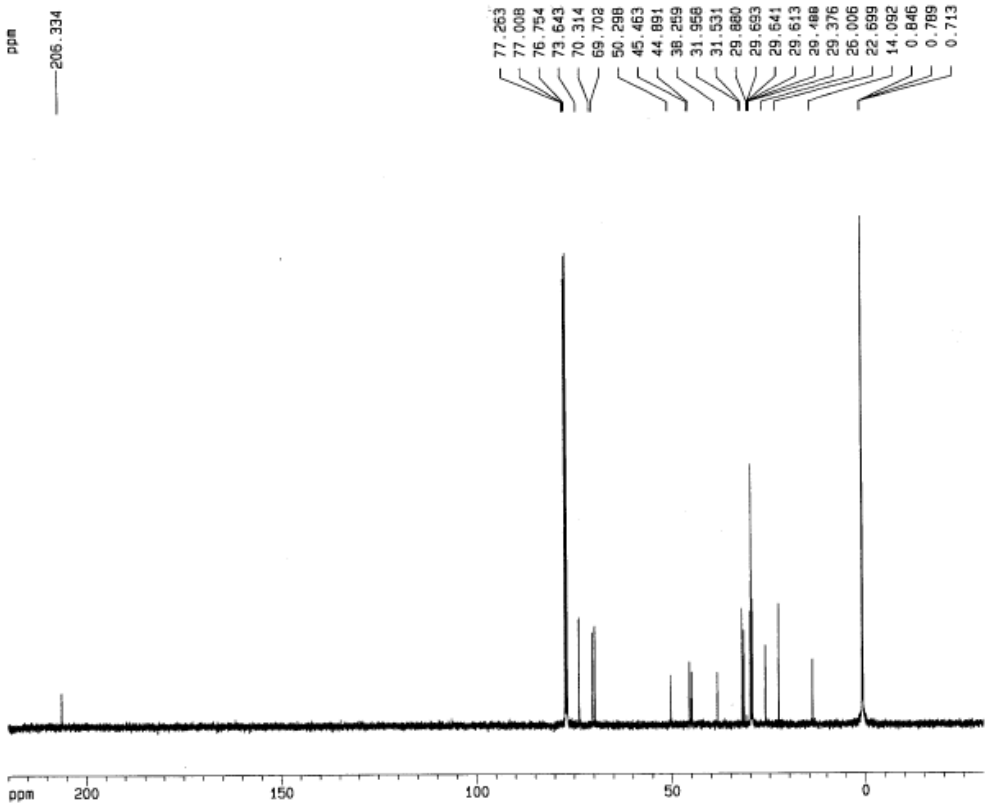
^{13}C NMR for 9: $CDCl_3$, 126 MHz, 293K



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TE 300.0 K
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d11 0.03000000 sec

========== CHANNEL f1 ==========
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PL1 3.00 dB
SFO1 125.7671708 MHz

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F2 - Processing parameters
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1D NMR plot parameters
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F2P -30.000 ppm
F2 -3772.73 Hz
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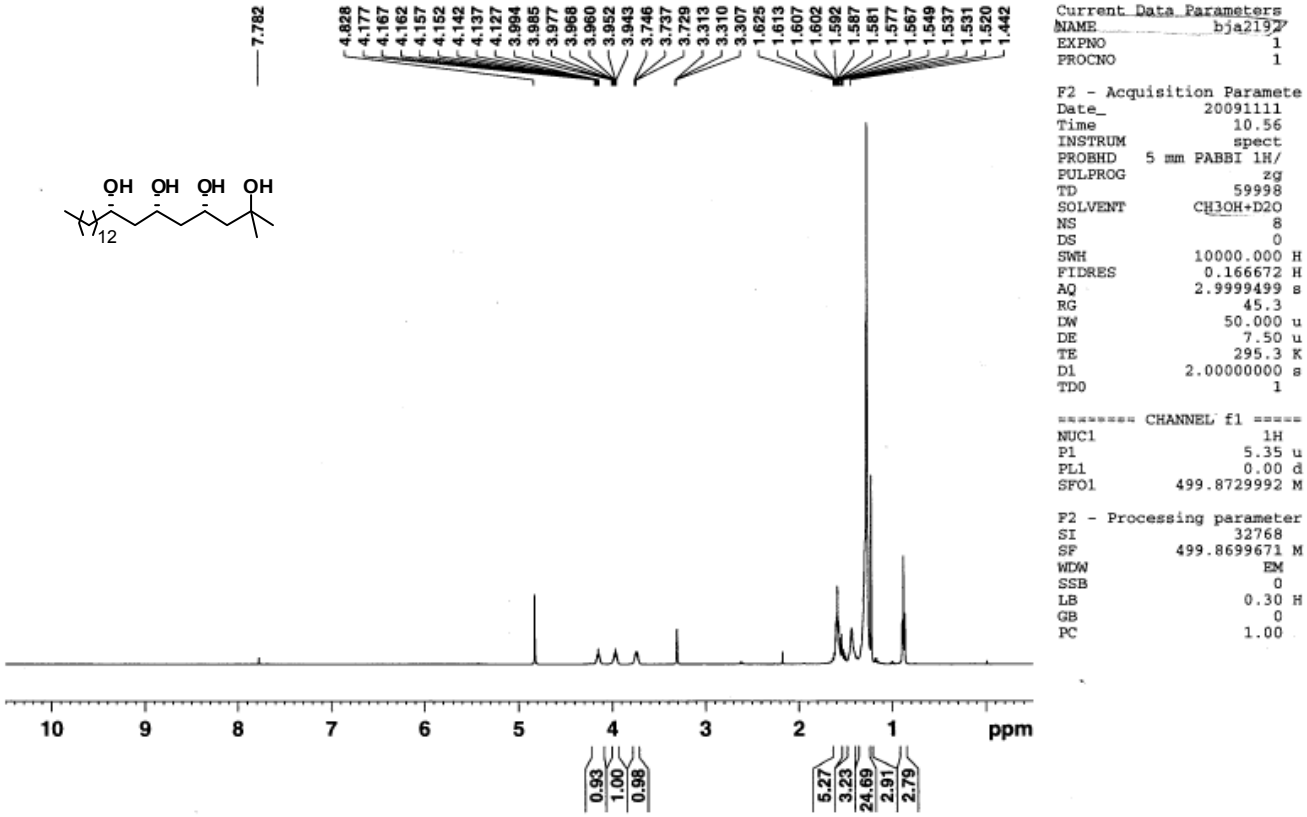
^{1}H NMR for S1-*syn/syn*: 5% $CDCl_3$ in CD_3OD, 500 MHz, 293K

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TE                       295.3 K
D1                  2.00000000 s
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======== CHANNEL f1 =====
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PL1                      0.00 d
SFO1                499.8729992 M

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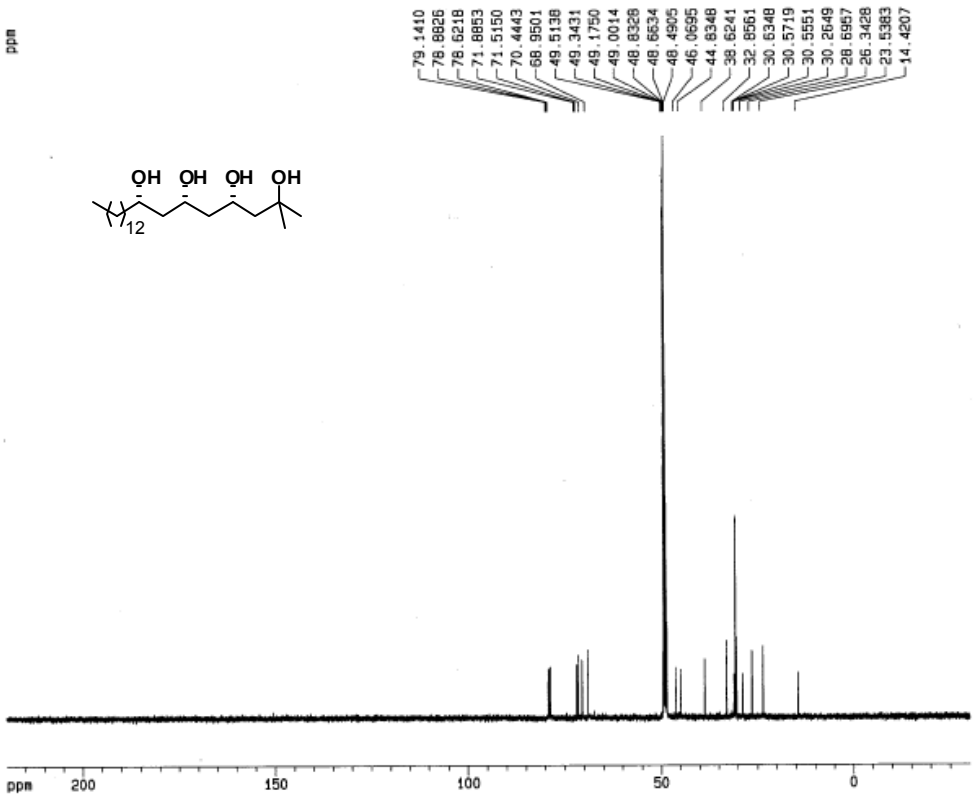
^{13}C NMR for S1-*syn/syn*: 5% $CDCl_3$ in CD_3OD, 126 MHz, 293K

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PROCNO                      2

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FIDRES                0.321262 Hz
AQ                    1.5564140 sec
RG                       8192
DW                     13.800 usec
DE                      7.50 usec
TE                      300.0 K
D1                  5.00000000 sec
d11                 0.03000000 sec

========== CHANNEL f1 ==========
NUC1                       13C
P1                        8.00 usec
PL1                       3.00 dB
SFO1               125.7671708 MHz

========== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL2                      120.00 dB
PL12                      20.00 dB
SFO2               500.1338000 MHz

F2 - Processing parameters
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1D NMR plot parameters
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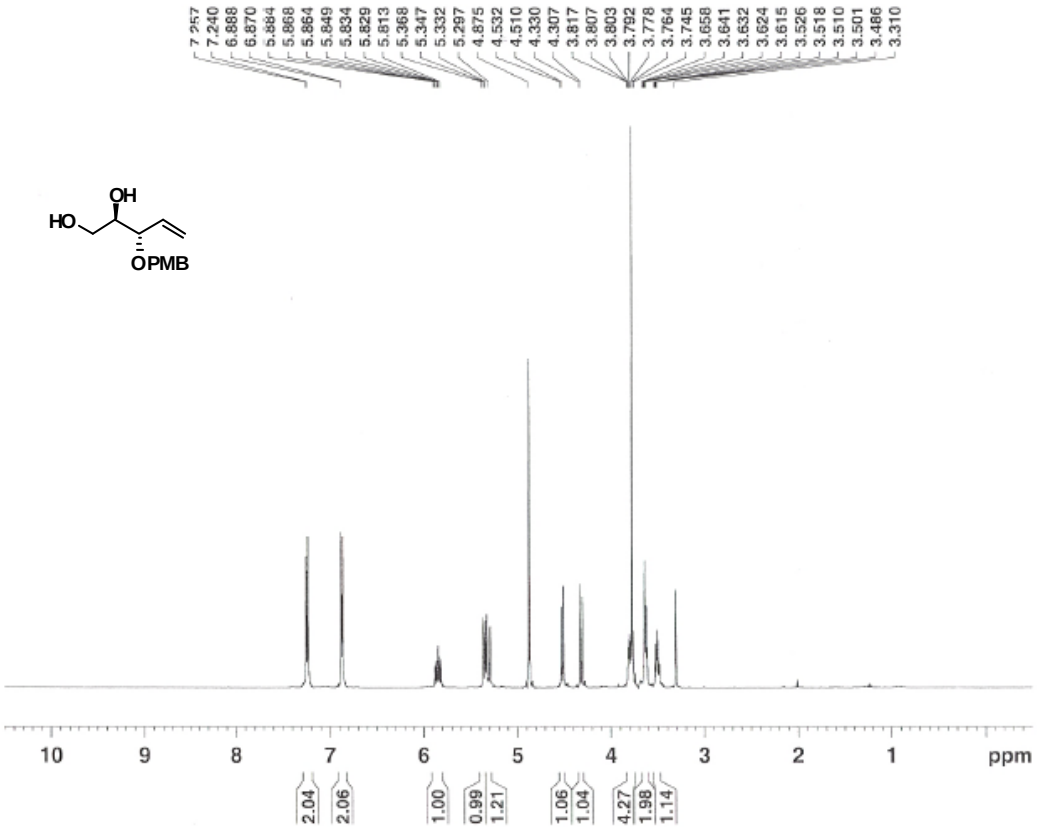
^{1}H NMR for S2: $CDCl_3$, 500 MHz, 293K

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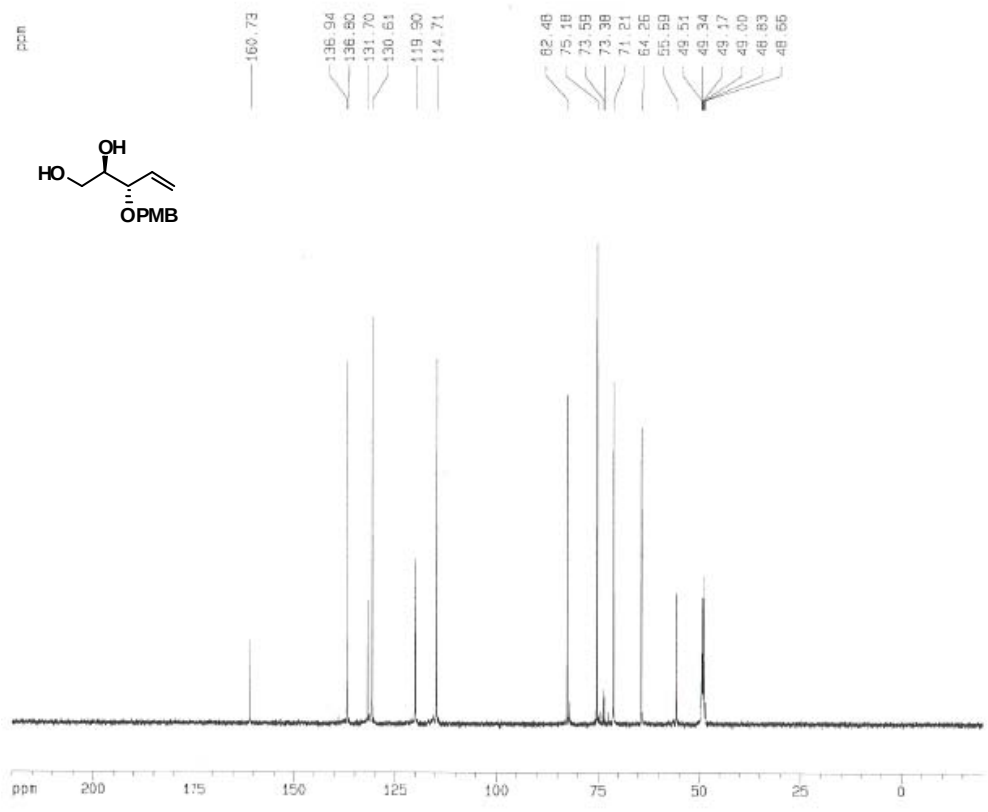
Time                  8.56
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG                 zg
TD                   59998
SOLVENT               MeOD
NS                       8
DS                       0
SWH           10000.000 Hz
FIDRES            0.166672 Hz
AQ                2.9999499 se
RG                     71.8
DW                  50.000 us
DE                   7.50 us
TE                  295.3 K
D1             2.00000000 se
TD0                      1

======== CHANNEL f1 ======
NUC1                    1H
P1                    5.35 us
PL1                   0.00 dB
SFO1          499.8729992 MH

F2 - Processing parameters
SI                   32768
SF            499.8692447 MH
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

^{13}C NMR for S2: $CDCl_3$, 126 MHz, 293K




```
Current Data Parameters
NAME          bja3241-CD3OD
EXPNO                     2
PROCNO                    2

F2 - Acquisition Parameters
Date_              20100113
Time                 17.45
INSTRUM              spect
PROBHD   5 mm QNP  1H
PULPROG              zgdc
TD                   75184
SOLVENT              CD3OD
NS                     800
DS                       0
SWH           37593.984 Hz
FIDRES            0.500026 Hz
AQ                0.9999872 sec
RG                    8192
DW                  13.300 usec
DE                   7.50 usec
TE                  300.0 K
D1             0.10000000 sec
D11            0.03000000 sec

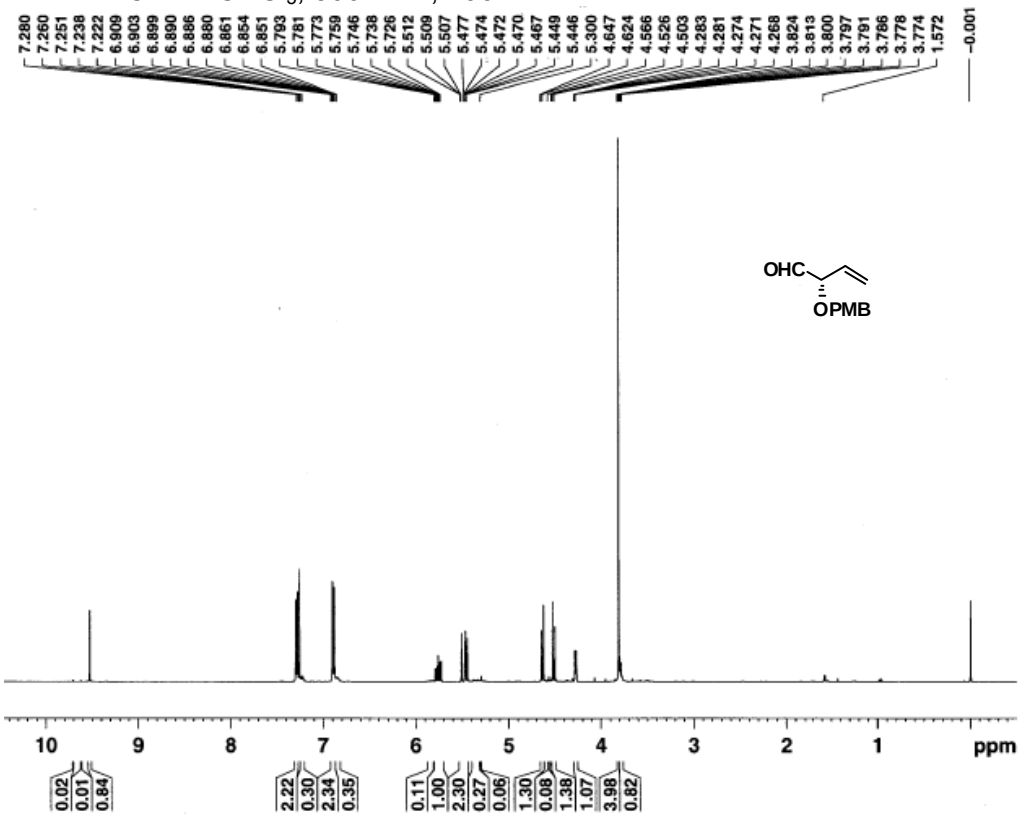
========== CHANNEL f1 =========
NUC1                    13C
P1                   4.60 usec
PL1                  9.00 dB
SFO1          125.7690572 MHz

========== CHANNEL f2 =========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                50.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2          500.1328005 MHz

F2 - Processing parameters
SI                   32768
SF            125.7576353 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 220.000 ppm
F1                27666.68 Hz
F2P                -20.000 ppm
F2                -2515.15 Hz
PPMCM             12.00000 ppm/cm
HZCM              1509.09131 Hz/cm
```

^{1}H NMR for 11: $CDCl_3$, 500 MHz, 293K



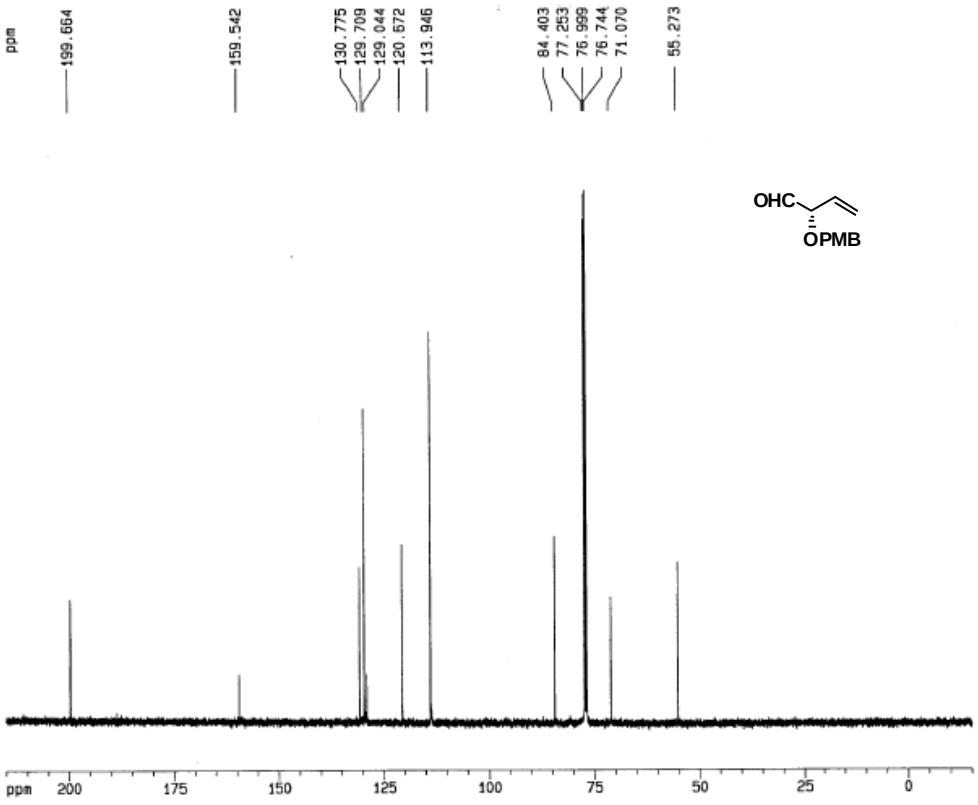

```
Current Data Parameters
NAME          bja4149-crude
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameter
Date_              20100624
Time                  11.25
INSTRUM               spect
PROBHD   5 mm PABBI 1H/
PULPROG                 zg
TD                   59998
SOLVENT              CDCl3
NS                       8
DS                       0
SWH           10000.000 H
FIDRES            0.166672 H
AQ                2.9999499 s
RG                     80.6
DW                50.000 u
DE                 7.50 u
TE                  292.9 K
D1             2.00000000 s
TD0                      1

======== CHANNEL f1 =====
NUC1                    1H
P1                   5.35 u
PL1                  0.00 d
SFO1          499.8729992 M

F2 - Processing parameter
SI                   32768
SF            499.8700179 M
WDW                     EM
SSB                      0
LB                0.30 H
GB                       0
PC                    1.00
```

^{13}C NMR for 11: $CDCl_3$, 126 MHz, 293K




```
Current Data Parameters
NAME       baj4025-crude
EXPNO                  2
PROCNO                 2

F2 - Acquisition Parameters
Date_           20100317
Time               13.16
INSTRUM            spect
PROBHD   5 mm QNP  1H
PULPROG            zgdc
TD               112780
SOLVENT            CDCl3
NS                   214
DS                     0
SWH          36231.883 Hz
FIDRES        0.321252 Hz
AQ           1.5564140 sec
RG                  8192
DW               13.800 usec
DE                7.50 usec
TE                300.0 K
D1           6.00000000 sec
d11          0.03000000 sec

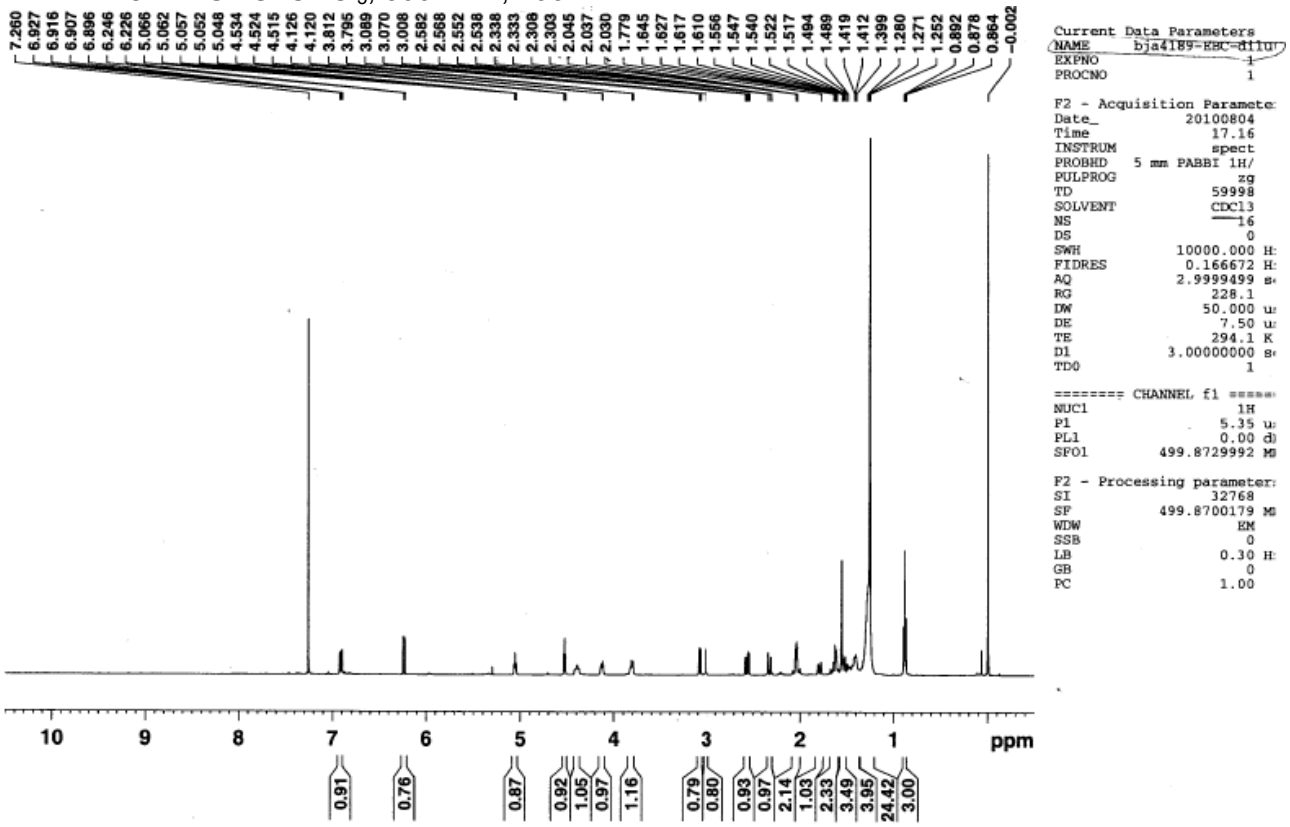
============ CHANNEL f1 ========
NUC1                  13C
P1                 8.00 usec
PL1                3.00 dB
SFO1        125.7671708 MHz

============ CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             90.00 usec
PL2               120.00 dB
PL12               20.00 dB
SFO2        500.1338000 MHz

F2 - Processing parameters
SI                   32768
SF          125.7577938 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                  20.00 cm
F1P                215.000 ppm
F1               27037.93 Hz
F2P                -15.000 ppm
F2               -1886.37 Hz
PPMCM             11.50000 ppm/cm
HZCM          1448.21472 Hz/cm
```

^{1}H NMR for EBC-23: $CDCl_3$, 500 MHz, 293K



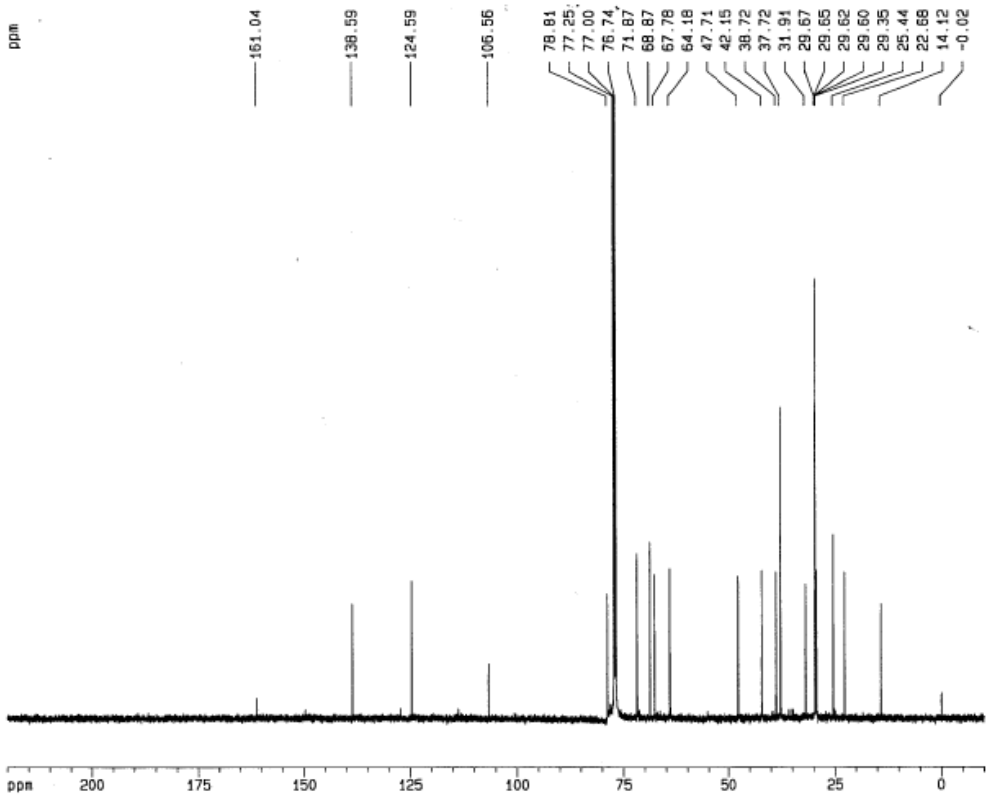

```
Current Data Parameters
NAME      bja4189-EBC-dilut
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameter
Date_              20100804
Time                  17.16
INSTRUM               spect
PROBHD   5 mm PABBI 1H/
PULPROG                 zg
TD                   59998
SOLVENT              CDCl3
NS                      16
DS                       0
SWH           10000.000 H
FIDRES            0.166672 H
AQ                2.9999499 s
RG                    228.1
DW                 50.000 u
DE                  7.50 u
TE                 294.1 K
D1            3.00000000 s
TD0                      1

======== CHANNEL f1 =====
NUC1                    1H
P1                  5.35 u
PL1                 0.00 d
SFO1          499.8729992 M

F2 - Processing parameter
SI                   32768
SF            499.8700179 M
WDW                     EM
SSB                      0
LB                  0.30 H
GB                       0
PC                    1.00
```

^{13}C NMR for EBC-23: $CDCl_3$, 126 MHz, 293K




```
Current Data Parameters
NAME      bja4189-dilute
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100804
Time                  21.21
INSTRUM               spect
PROBHD   5 mm QNP  1H
PULPROG                zgdc
TD                   75184
SOLVENT               CDCl3
NS                    32768
DS                       0
SWH           36231.883 Hz
FIDRES            0.481910 Hz
AQ                1.0375892 sec
RG                    8192
DW                13.800 usec
DE                 7.50 usec
TE                 300.0 K
D1            0.10000000 sec
d11           0.03000000 sec

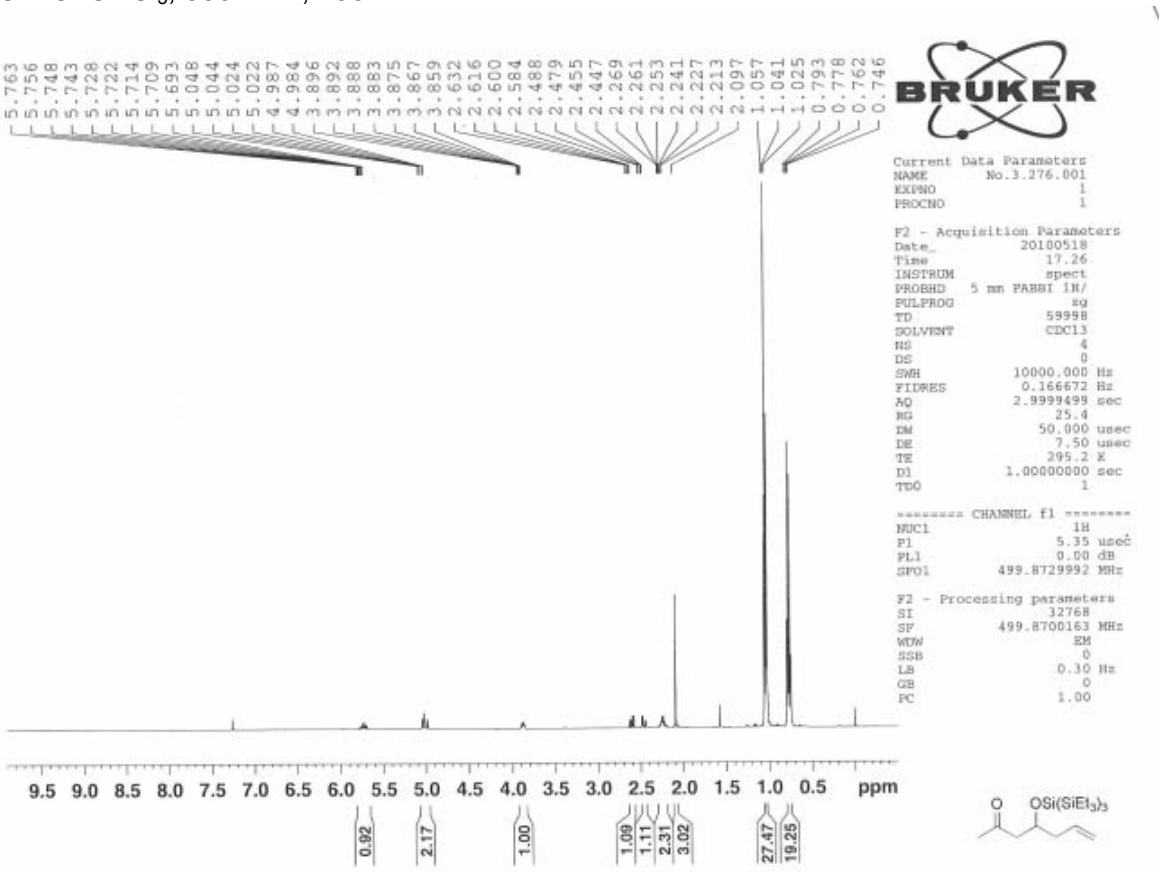
========== CHANNEL f1 ==========
NUC1                   13C
P1                 4.60 usec
PL1                0.00 dB
SFO1          125.7690572 MHz

========== CHANNEL f2 ==========
CPDPRG2            waltz16
NUC2                    1H
PCPD2              90.00 usec
PL2                120.00 dB
PL12                19.00 dB
SFO2          500.1320005 MHz

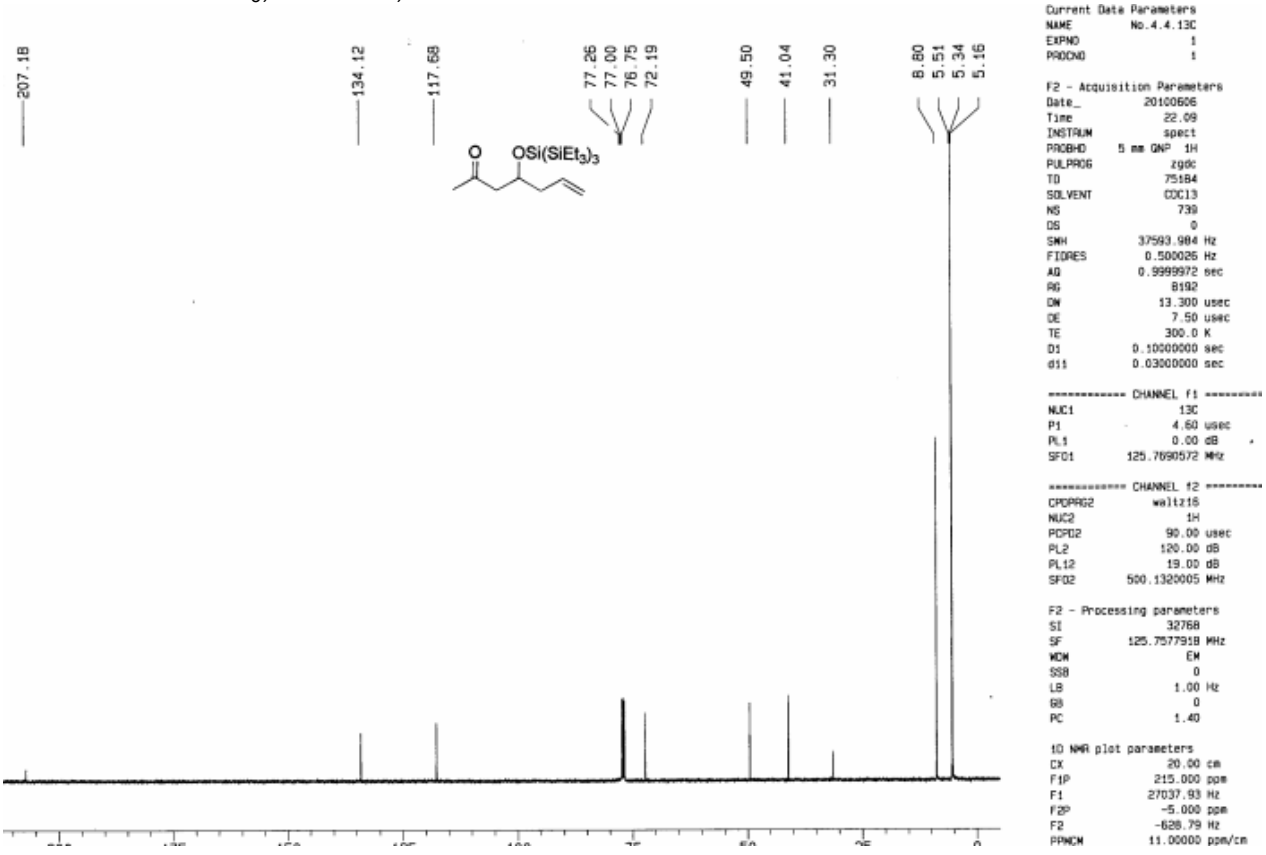
F2 - Processing parameters
SI                   32768
SF            125.7577927 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                  20.00 cm
F1P               220.000 ppm
F1                27666.71 Hz
F2P               -10.000 ppm
F2                -1257.58 Hz
PPMCM             11.50000 ppm/cm
HZCM          1446.21460 Hz/cm
```

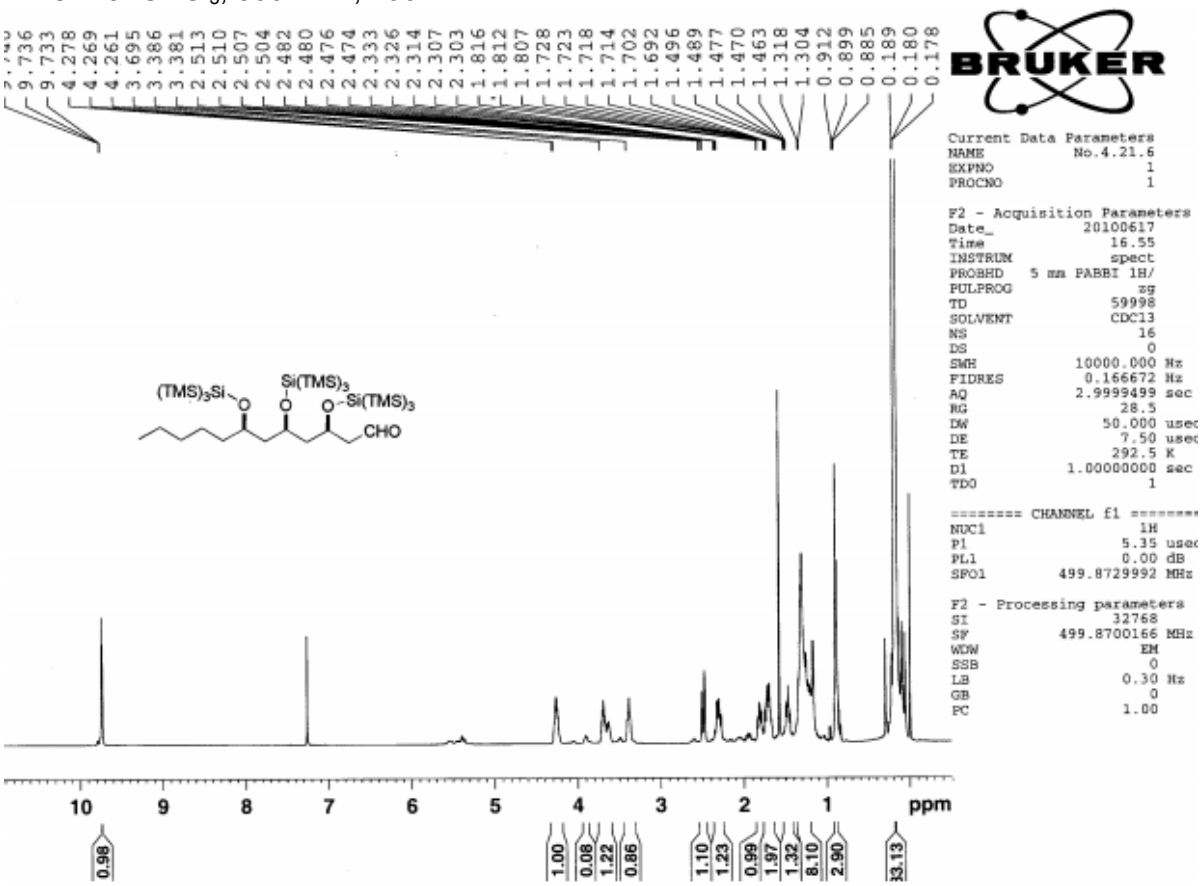
^{1}H NMR for 15: $CDCl_3$, 500 MHz, 293K



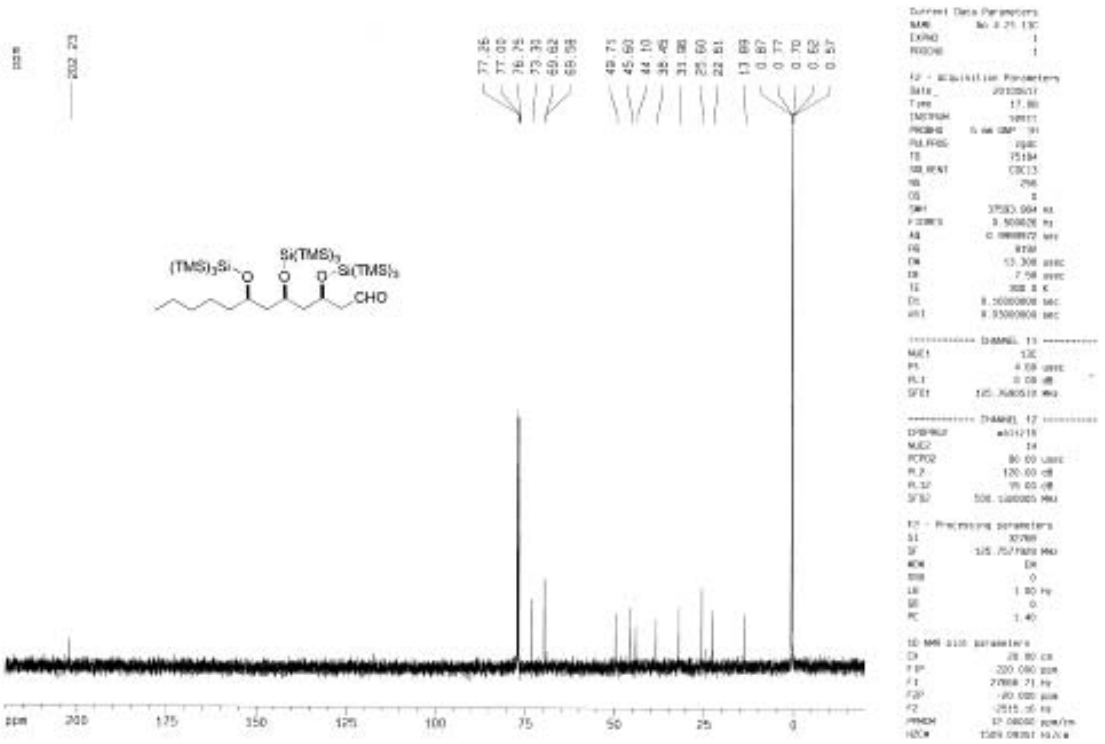
^{13}C NMR for 15: $CDCl_3$, 126 MHz, 293K



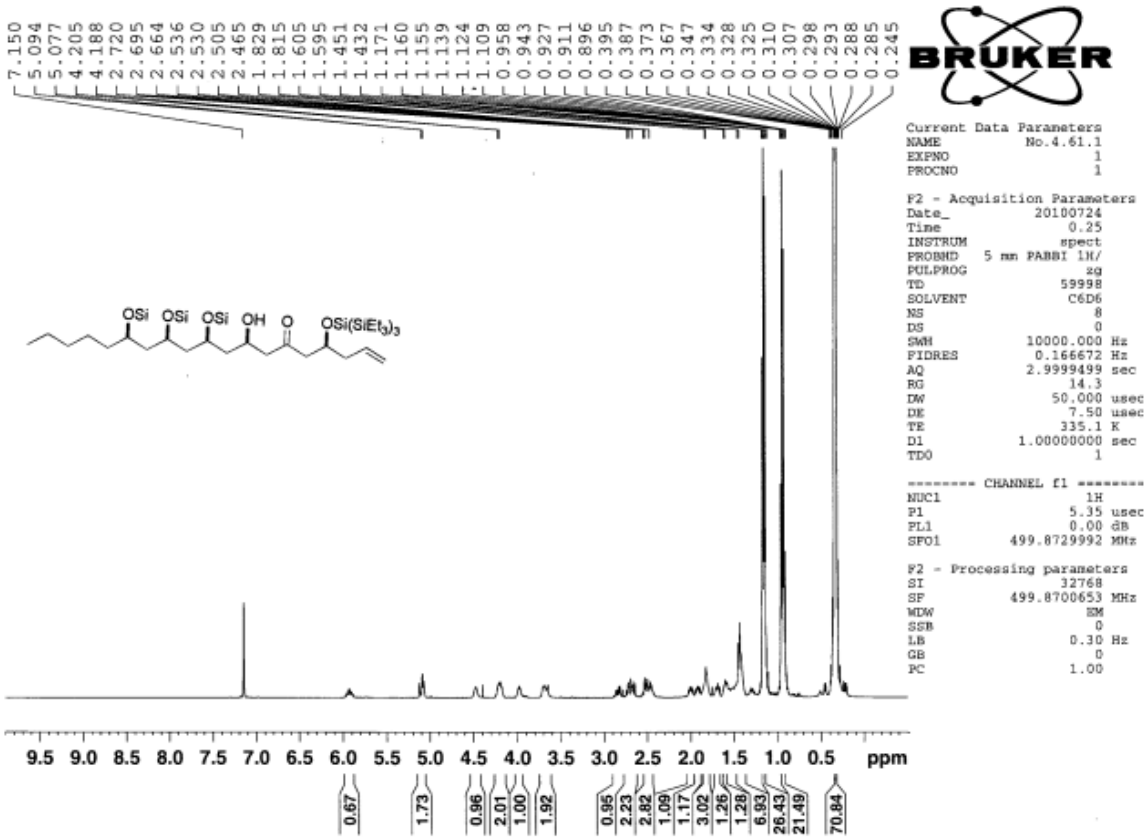
^{1}H NMR for 16: $CDCl_3$, 500 MHz, 293K



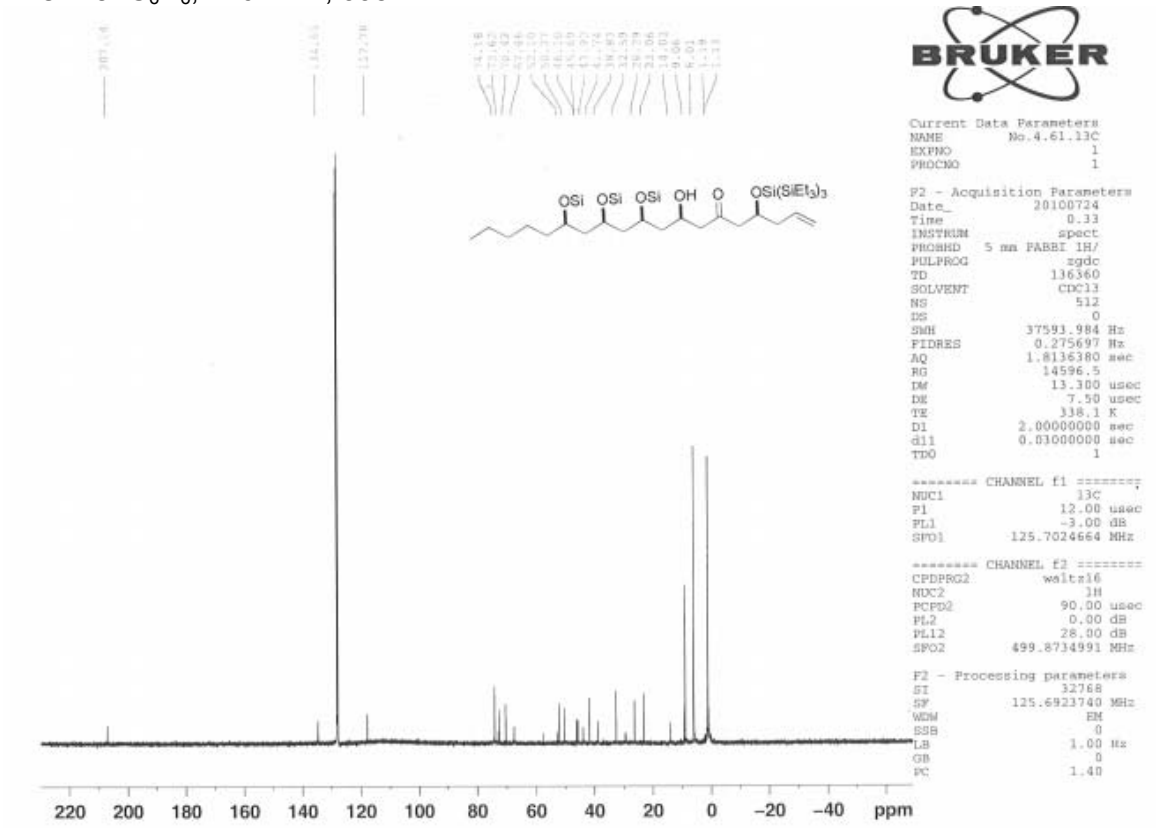
^{13}C NMR for 16: $CDCl_3$, 126 MHz, 293K



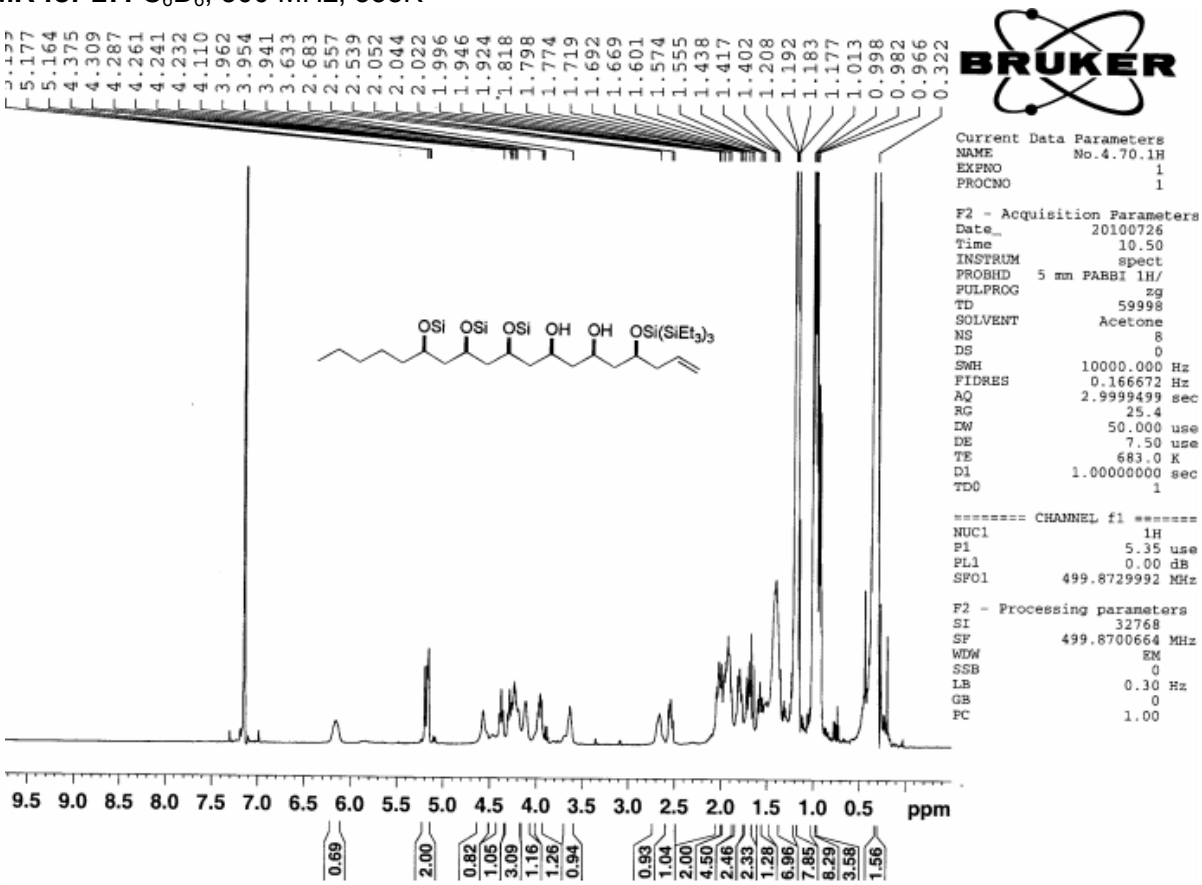
^{1}H NMR for 16: C_6D_6, 500 MHz, 338K



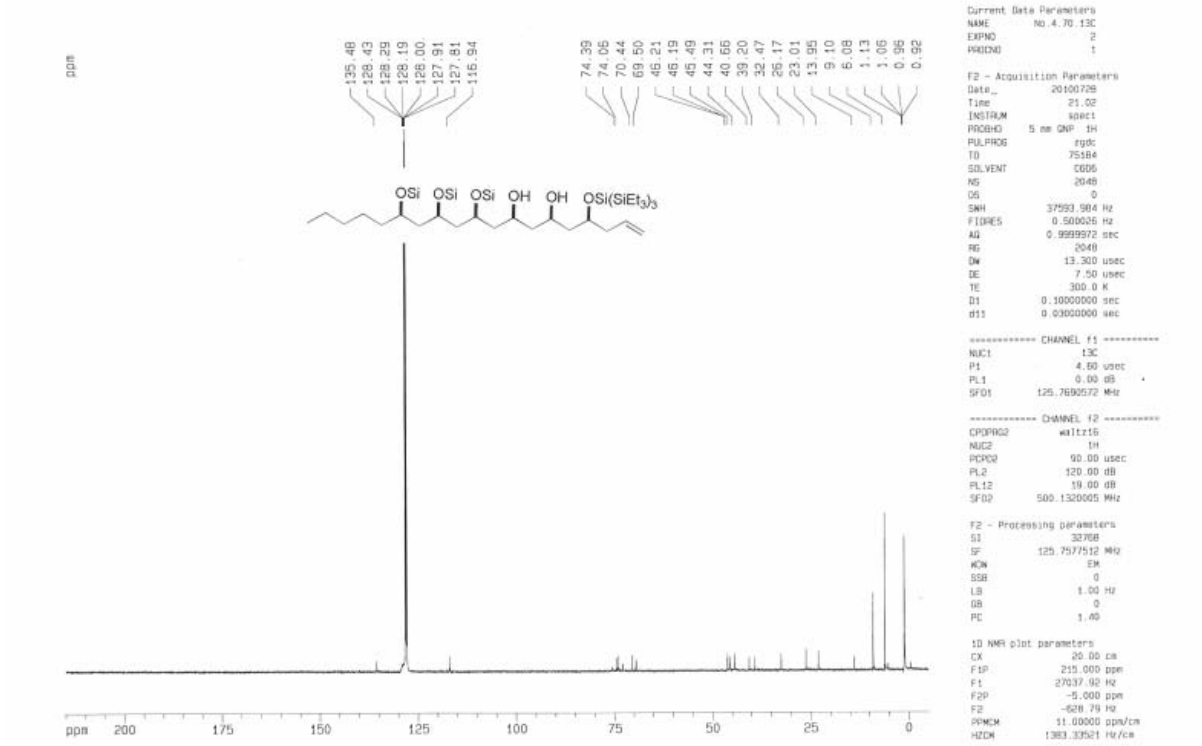
^{13}C NMR for 16: C_6D_6, 126 MHz, 338K



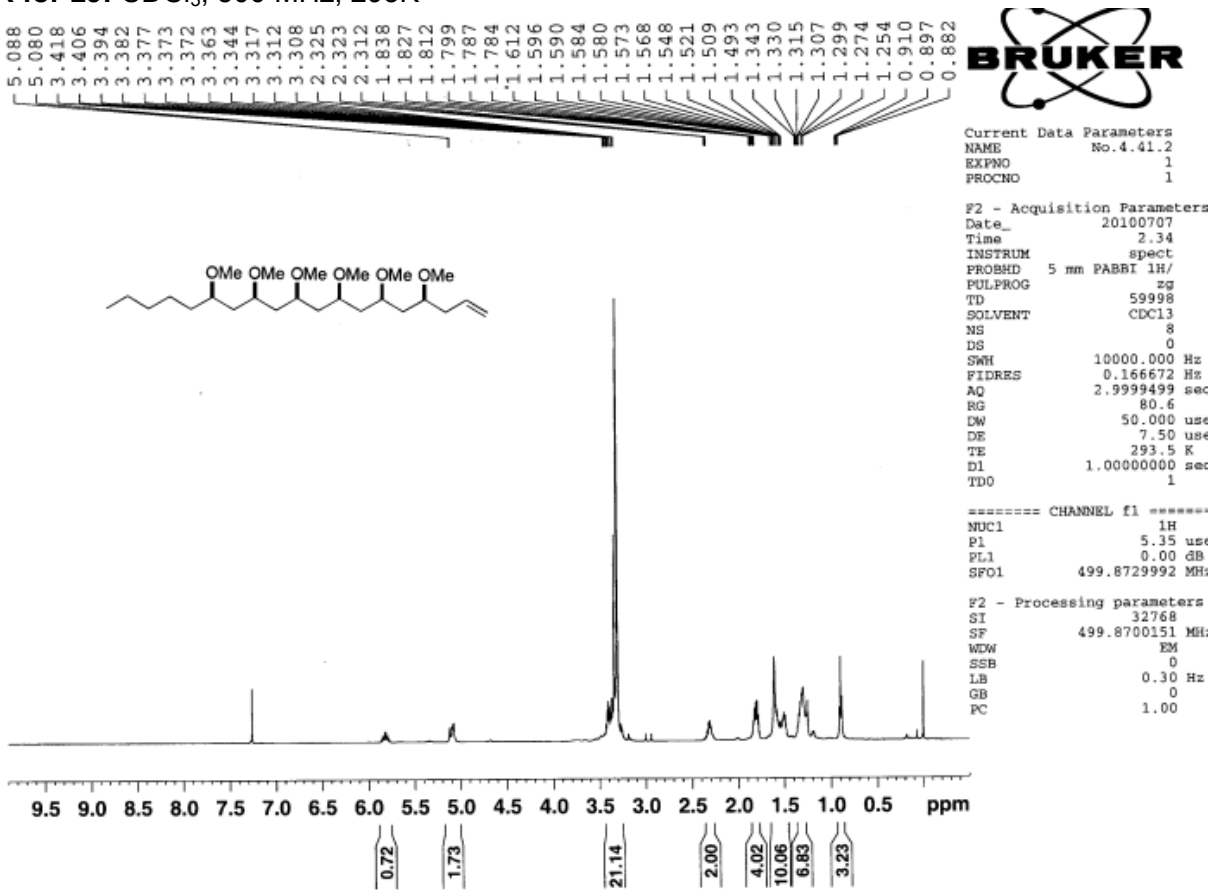
^{1}H NMR for 17: C_6D_6, 500 MHz, 338K



^{13}C NMR for 17: C_6D_6, 126 MHz, 338K



^{1}H NMR for 19: $CDCl_3$, 500 MHz, 293K



BRUKER

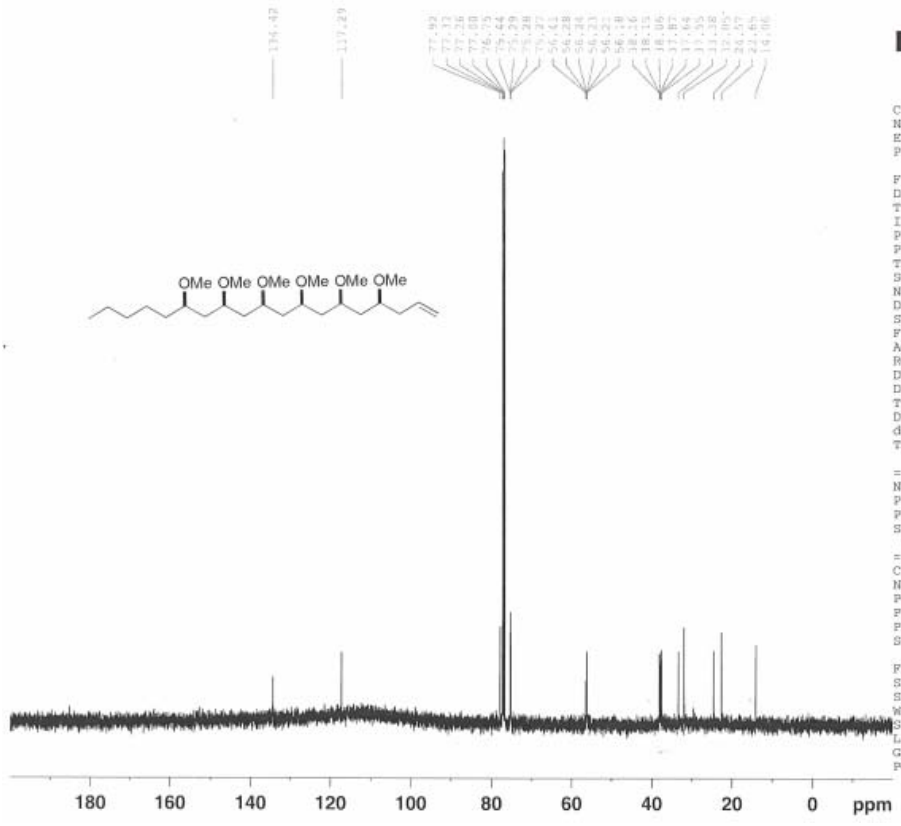
```
Current Data Parameters
NAME                  No.4.41.2
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20100707
Time                       2.34
INSTRUM                   spect
PROBHD    5 mm PABBI 1H/
PULPROG                     zg
TD                         59998
SOLVENT                    CDCl3
NS                             8
DS                             0
SWH                10000.000 Hz
FIDRES                 0.166672 Hz
AQ                     2.9999499 sec
RG                          80.6
DW                        50.000 use
DE                         7.50 use
TE                        293.5 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 =======
NUC1                          1H
P1                         5.35 use
PL1                        0.00 dB
SFO1                499.8729992 MHz

F2 - Processing parameters
SI                         32768
SF                  499.8700151 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

^{13}C NMR for 19: $CDCl_3$, 126 MHz, 293K



BRUKER

```
Current Data Parameters
NAME                  No.4.41.2C
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20100707
Time                       2.49
INSTRUM                   spect
PROBHD    5 mm PABBI 1H/
PULPROG                  zgdc
TD                       136360
SOLVENT                    CDCl3
NS                          1024
DS                             0
SWH                37593.984 Hz
FIDRES                 0.275697 Hz
AQ                     1.8136380 sec
RG                      18390.4
DW                        13.300 usec
DE                         7.50 usec
TE                        293.9 K
D1                     2.00000000 sec
d11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         13C
P1                        12.00 usec
PL1                       -3.00 dB
SFO1               125.7024664 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                     90.00 usec
PL2                        0.00 dB
PL12                      28.00 dB
SFO2               499.8734991 MHz

F2 - Processing parameters
SI                         32768
SF                  125.6924169 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```