Supporting Information

Total Synthesis of (–)-Sigillin A: A Poly-Chlorinated and Poly-Oxygenated Natural Product

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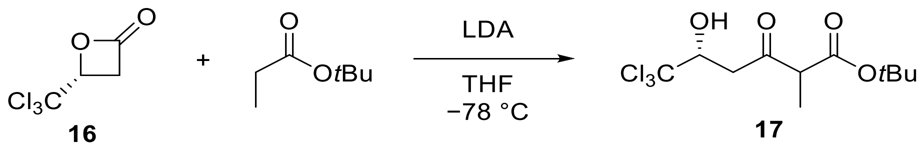
Table of Contents

1. General Methods

Dehydrated solvents for the reactions were purchased and used without further desiccation. For reactions that require heating, oil bath was used as a heat source. Reactions were monitored by thin-layer chromatography (TLC) carried out on Wako TLC silica gel 70 F_{254}. Column chromatography was performed using Fuji Silysia BW-200 silica gel with visualization by ultraviolet (UV) irradiation at 254 nm and/or indicated stains. Nuclear magnetic resonance (NMR) spectra were recorded on a JEOL JNM-LA (500 MHz for ^{1}H and 125 MHz for ^{13}C). Chemical shifts are presented in ppm relative to tetramethylsilane (^{1}H, 0.00) or solvents as follows: $CDCl_3$ (^{13}C, 77.0); acetone-d_6 (^{1}H, 2.04; ^{13}C, 29.8); DMSO-d_6 (^{1}H, 2.49; ^{13}C, 39.5). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were recorded on a JEOL MS700 spectrometer (FAB), a SHIMADZU LCMS-IT-TOF fitted with an ESI. IR experiments were recorded on a SHIMADZU IRAffinity-1 spectrometer. The wave numbers of maximum absorption peaks of IR spectroscopy are presented in cm^{-1}. Optical rotations were recorded on JASCO P-2000 polarimeter. All melting points were determined using a Yamato MP-21 melting point apparatus and are uncorrected. X-ray diffraction data were recorded on a RIGAKU R-AXIS RAPID system.

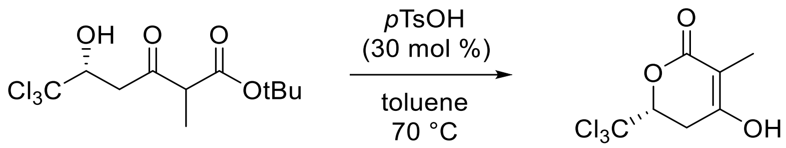
2. Experimental Procedures and Characterization Data for New Compounds



5-Hydroxy-3-oxoester 17: A solution of LDA was prepared by adding *n*-BuLi (104.0 mL, 240 mmol, 2.3 M in cyclohexane) to a solution of diisopropylamine (34.0 mL, 240 mmol) in THF (500 mL) at −78 °C. After being stirred for 30 min, *tert*-butyl propionate (36.0 mL, 240 mmol) in THF (50 mL) was added dropwise to the solution of LDA and stirring was continued for 30 min at –78 °C. Then a solution of (*R*)-4-trichloromethyl-2-oxetanone, which was prepared according literature[1], (15.2 g, 80 mmol) in THF (40 mL) was added in one portion and the mixture was stirred at −78 °C for 30 min. The reaction was quenched with saturated aqueous NH_4Cl (400 mL). The organic layer was separated, and the aqueous layer was extracted with diethyl ether (200 mL × 2). The combined organic layers were washed with brine (50 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography

[1] Ganta, A.; Shamshina, J. L. Cafiero, L. R. Snowden, T. S. *Tetrahedron,* **2012**, *68*, 5396–5405.

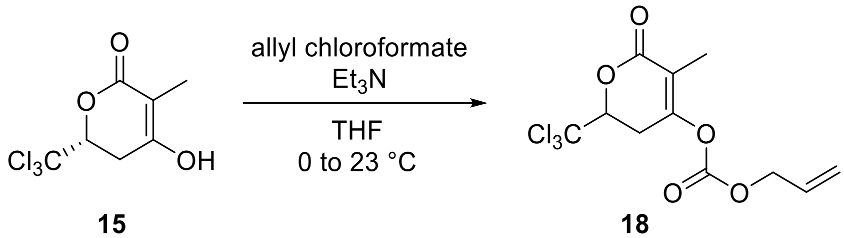
on silica gel (hexane/Et_2O) to afford product **17** as a mixture of diastereomers (20.9 g, 82%). The mixture was recrystallized with hexane/Et_2O at –78 °C to afford single diastereomer of **17** (18.9 g) as white solids; mp 75–77 °C (hexane/Et_2O); R_f = 0.23 (25% Et_2O/hexane, hanessian); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.71 (dd, J = 5.1, 5.1 Hz, 1H), 3.51 (br s, 1H), 3.50 (q, J = 6.6 Hz, 1H), 3.16 (d, J = 5.7 Hz, 2H), 1.48 (s, 9H), 1.35 (d, J = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 203.1, 169.0, 102.4, 82.6, 78.5, 54.4, 43.4, 27.9, 12.4. Spectroscopic properties were consistent with those reported in the literature.[2]



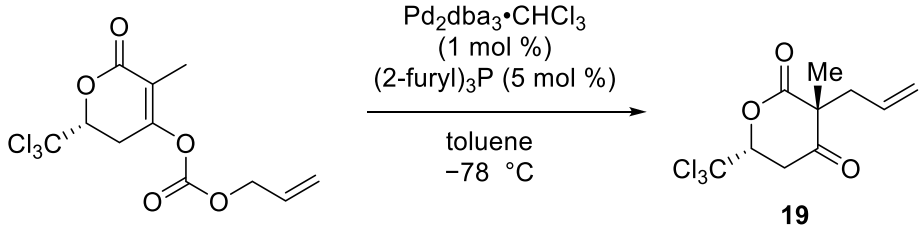
17 **15**

β-Keto-δ-valerolactone 15: To a stirred solution of **17** (13.8 g, 43.2 mmol) in toluene (800 mL) was added *p*TSA·H_2O (2.47 g, 13.0 mmol). After being stirred at 70 °C for 6 h, the resulting mixture was cooled to 23 °C. The organic layer was extracted with saturated aqueous $NaHCO_3$ (100 mL × 6). The aqueous layers were combined and neutralized by 10% aqueous HCl. The aqueous solution was extracted with AcOEt (100 mL × 4). The combined organic layers were washed with water (50 mL), followed by brine (50 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure to afford product **15** (8.24 g, 78%) as white solids. R_f = 0.25 (10% MeOH/$CHCl_3$, UV, cerium sulfate); mp 158–162 °C (AcOEt/hexane); $[\alpha]_D^{23}$ = +54.7 (c = 1.00, acetone); ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.2 (br s, 1H), 5.18 (dd, J = 11.6, 4.4 Hz, 1H), 2.94 (dd, J = 16.9, 4.3 Hz, 1H), 2.83 (m, 1H), 1.65 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 165.7, 163.9, 98.4, 96.7, 80.9, 29.2, 8.6; IR (neat) ν_{max}: 3190, 1743, 1654, 1396, 1373, 1338, 1246, 1095, 1049, 802 cm^{-1}; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_7H_7Cl_3O_3Na$ 266.9353; found 266.9351.

2 Schmidt, W.; Schulze, T. M.; Brasse, G.; Nagrodzka, E.; Maczka, M.; Zettel, J.; Jones, P. G.; Grunenberg, J.; Hilker, M.; Trauer-Kizilelma, U.; Braun, U.; Schulz, S. *Angew. Chem., Int. Ed.,* **2015**, *54*, 7698–7702.



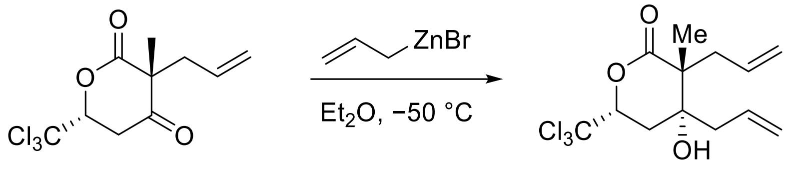
Allyl carbonate 18:[3] To a stirred solution of **15** (4.5 g, 18.3 mmol) and triethylamine (6.5 mL, 45.8 mmol) in THF (90 mL) was slowly added allyl chloroformate (3.9 mL, 36.7 mmol) at 0 °C. After being stirred at 23 °C for 3 h, the reaction was quenched by the addition of saturated aqueous NH_4Cl. The organic layer was separated, and the aqueous layer was extracted with AcOEt (100 mL). The combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane/AcOEt) to afford product **18** (6.0 g, 99%) as a colorless oil. R_f = 0.25 (10% AcOEt/hexane, UV); $[\alpha]_D^{23}$ = +39.0 (*c* = 1.03, acetone); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.98 (ddt, *J* = 17.1, 10.6, 6.0 Hz, 1H), 5.44 (ddd, *J* = 17.2, 2.6, 1.2 Hz, 1H), 5.38 (ddd, *J* = 10.5, 2.0, 1.0 Hz, 1H), 4.93 (dd, *J* = 11.9, 4.2 Hz, 1H), 4.75 (ddd, *J* = 6.0. 1.2, 1.2 Hz, 2H), 3.20 (ddq, *J* = 17.2, 12.1, 2.3 Hz, 1H), 3.08 (ddd, *J* = 17.3, 4.2, 1.2 Hz, 1H), 1.90 (dd, *J* = 2.6, 1.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 163.6, 155.4, 150.7, 130.2, 120.5, 116.0, 97.0, 82.8, 70.1, 28.7, 9.9; IR (neat) ν_{max}: 2990, 1763, 1736, 1223, 1138, 937, 802 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{11}H_{12}Cl_3O_5$ 328.9750; found 328.9744.



Allylated β-keto-δ-valerolactone 19: A solution of $Pd_2dba_3 \cdot CHCl_3$ (72.0 mg, 0.14 mmol, 1 mol % based on Pd) and tri(2-furyl)phosphine (162 mg, 0.70 mmol) in toluene (135 mL) was stirred for 10 min at 23 °C. Then, a solution of **18** (4.50 g, 13.7 mmol) in toluene (10 mL) was slowly added to the mixture at –78 °C. After the mixture was gradually warmed up to 23 °C, the solvent was evaporated. The residue was purified by column chromatography on silica gel (hexane/AcOEt) to afford product **19** (3.79 g, 96%, dr = 94:6 based on the crude ^{1}H NMR, concomitant with a small amount of dibenzylideneacetone) as a colorless oil. R_f = 0.28 (10%

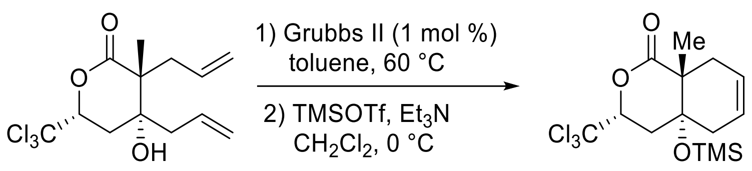
[3] Prantz, K.; Mulzer, J. *Chem. Eur. J.* **2010**, *16*, 485–506.

AcOEt/hexane, anisaldehyde); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.61–5.73 (m, 1H), 5.18–5.08 (m, 2H), 4.92 (dd, J = 11.9, 2.9 Hz, 1H), 3.29 (dd, J = 16.3, 2.9 Hz, 1H), 2.84–2.75 (m, 2H), 2.60 (dd, J = 13.5, 8.3 Hz, 1H), 1.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 202.8, 170.5, 131.3, 120.7, 97.3, 81.1, 56.1, 41.6, 41.2, 22.7; IR (neat) ν_{max}: 2982, 2924, 1767, 1721, 1454, 1225, 1134, 795 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{10}H_{12}Cl_3O_3$ 284.9852; found 284.9858.



19 20

Diallyl 20: To a stirred solution of **19** (4.02 g, 14.1 mmol) in Et_2O (144 mL) was added a 1.25 M THF solution of allylzinc bromide[4] (23.0 mL, 28.8 mmol) at –50 °C. After being stirred at –50 °C for 30 min, the solution was quenched with 3% aqueous HCl. The organic layer was separated, and the aqueous layer was extracted by AcOEt (50 mL × 2). The combined organic layers were washed with brine (20 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Directly recrystallization of (hexane/AcOEt) of the obtained crude mixture (dr = 94:6) afforded the desired product **20** as a single diastereomer (3.61 g, 78%) as white solids. R_f = 0.32 (20% AcOEt/hexane, potassium permanganate); mp 148–152 °C (hexane/AcOEt); $[\alpha]_D^{23}$ = –45.3 (c = 1.00, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.02–5.82 (m, 2H), 5.32 (ddd, J = 10.2, 1.2, 1.2 Hz, 1H), 5.26 (ddd, J = 17.0, 1.6, 1.3 Hz, 1H), 5.21–5.11 (m, 2H), 4.63 (dd, J = 11.3, 5.6 Hz, 1H), 2.65–2.56 (m, 3H), 2.46 (dd, J = 14.1, 5.5 Hz, 1H), 2.36 (ddd, J = 14.0, 11.2, 1.7 Hz, 1H), 2.13 (dd, J = 14.6, 8.3 Hz, 1H), 2.14 (br m, 1H), 1.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.4, 133.4, 131.6, 120.7, 119.3, 98.8, 82.9, 73.3, 51.0, 41.4, 39.2, 31.4, 15.7; IR (neat) ν_{max}: 3472, 3082, 2978, 1728, 1196, 1115, 1022, 999, 914, 814, 783 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{13}H_{18}Cl_3O_3$ 327.0322; found 327.0326.

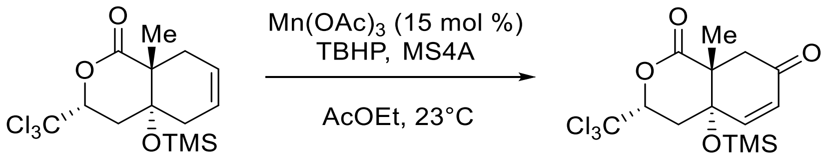


20 21

Hexahydroisocoumarin 21: To a stirred solution of **20** (4.8 g, 14.7 mmol) in toluene (150 mL) was added Grubbs 2nd catalyst (124 mg, 0.15 mmol) in one portion. After being stirred at 60 °C

[4] McNulty, J.; McLeod, D. *Eur. J. Org. Chem.* **2017**, 29–33.

for 1 h, the reaction mixture was cooled down to 23 °C, and the volatile was removed under reduced pressure. And then, the crude was dissolved in CH_2Cl_2 (150 mL) and triethylamine (10.0 mL, 73.3 mmol) and TMSOTf (8.0 mL, 44.0 mmol) successively added to the mixture at 0 °C. After being stirred at 23 °C for 2.5 h, the solution was quenched with water (50 mL). The organic phase was separated and washed with brine (20 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford product **21** (5.1 g, 94%) as white solids. R_f = 0.50 (20% AcOEt/hexane, potassium permanganate); mp 178–180 °C (hexane/$CHCl_3$); $[\alpha]_D^{23}$ = –71.4 (*c* = 0.135, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.78–5.67 (m, 1H), 5.57–5.47 (m, 1H), 5.00 (dd, *J* = 8.3, 8.3 Hz, 1H), 2.70 (br d, *J* = 18.0 Hz, 1H), 2.55–2.46 (m, 1H), 2.47 (dd, *J* = 14.9, 8.0 Hz, 1H), 2.30 (dd, *J* = 14.6, 8.3 Hz, 1H), 2.35–2.23 (m, 1H), 2.14 (dd, *J* = 17.9, 5.3 Hz, 1H), 1.21 (s, 3H), 0.08 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.6, 125.2, 121.7, 99.2, 81.9, 73.9, 45.1, 37.4, 36.2, 31.8, 17.0, 1.8; IR (neat) ν_{max}: 3036, 2943, 2920, 1740, 1389, 1292, 1250, 1227, 1146, 1111, 1076, 1076, 1011, 833, 787, 725 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{14}H_{22}Cl_3O_3Si$ 371.0404; found 371.0407.



Enone 22:[5] To a stirred solution of **21** (5.0 g, 13.5 mmol) in AcOEt (50 mL) were added MS 4A (10.0 g) and a solution of TBHP (5.0-6.0 M in decaline, 13.5 mL). After being stirred at 23 °C for 30 min, $Mn(OAc)_3{\cdot}2H_2O$ (540 mg, 2.0 mmol) was added to the mixture. The solution was degassed and filled with O_2 (ballon). After being stirred at 23 °C under O_2 for 120 h, the reaction mixture was filtered through a thin pad of Celite®, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford enone **22** (4.2 g, 81%) as white solids. R_f = 0.12 (20% AcOEt/hexane, UV, potassium permanganate); mp 149–153 °C (hexane/$CHCl_3$); $[\alpha]_D^{23}$ = +47.6 (*c* = 1.00, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 9.7 Hz, 1H), 6.13 (dd, *J* = 9.9, 1.0 Hz, 1H), 5.13 (dd, *J* = 8.7, 6.7 Hz, 1H), 3.14 (dd, *J* = 17.2, 1.2 Hz, 1H), 2.71 (d, *J* = 17.2 Hz, 1H), 2.59 (dd, *J* = 14.9, 6.9 Hz, 1H), 2.45 (dd, *J* = 14.8, 8.7 Hz, 1H), 1.31 (d, *J* = 1.2 Hz, 3H), 0.11 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.8, 170.6, 143.7, 130.7, 98.8, 81.9, 72.0, 49.2, 42.9, 35.6, 17.9, 1.9; IR (neat) ν_{max}: 2963, 2924, 2851,

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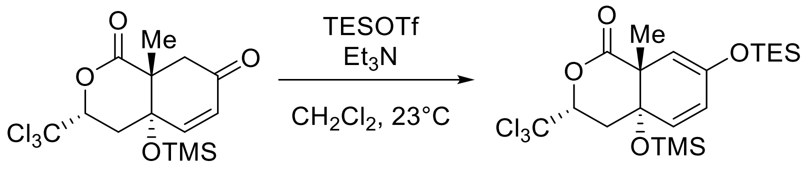
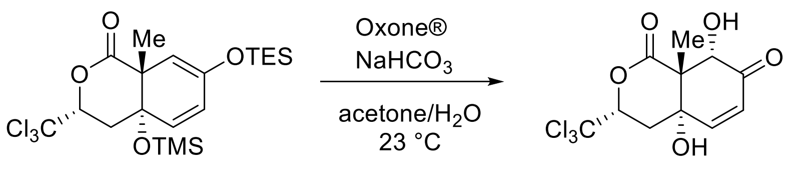
[5] (a) Shing, T. K. M.; Yeung, Y.-Y.; Su, P. L. *Org. Lett.* **2006**, *8*, 3149–3151. (b) Kumaran, R. S.; Mehta, G. *Tetrahedron* **2015**, *71*, 1547–1554.

1759, 1690, 1250, 1223, 1123, 1072, 999, 837, 795, 756, 718 cm^{-1}; HRMS (ESI) m/z: [M + Na]$^+$ Calcd for $C_{14}H_{19}Cl_3O_4SiNa$ 407.0010; found 407.0015.



22 **SI-1-TES**

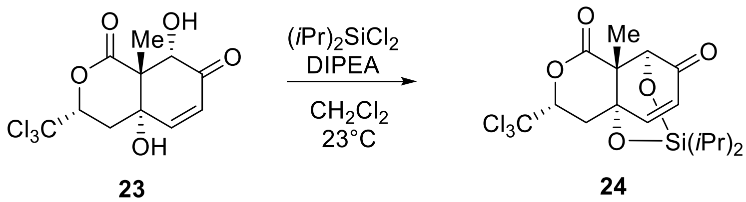
Triethylsiloxydiene SI-1-TES: To a stirred solution of **22** (4.2 g, 10.9 mmol) and triethylamine (4.6 mL, 33.0 mmol) in CH_2Cl_2 (110 mL) was added TESOTf (5.0 mL, 22.0 mmol) at 0 °C. After being stirred at 23 °C for 9 h, to the resulting mixture was quenched with saturated aqueous $NaHCO_3$ (30 mL). The organic layer was separated, and the aqueous layer was extracted with $CHCl_3$ (50 mL × 2). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford siloxydiene **SI-1-TES** (5.1 g, 94%) as white solids. R_f = 0.25 (10% AcOEt/hexane, UV, potassium permanganate); mp 147–149 °C (hexane); $[\alpha]_D^{23}$ = +219.3 (*c* = 1.05, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.92 (dd, *J* = 9.7, 2.0 Hz, 1H), 5.74 (d, *J* = 9.5 Hz, 1H), 5.52 (d, *J* = 1.7 Hz, 1H), 5.05 (dd, *J* = 8.5, 6.7 Hz, 1H), 2.47–2.29 (m, 2H), 1.27 (s, 3H), 1.00 (t, *J* = 7.9 Hz, 9H), 0.80-0.69 (m, 6H), 0.03 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 146.5, 129.9, 127.1, 108.2, 99.2, 83.0, 72.3, 50.2, 35.8, 18.4, 6.6, 4.8, 1.9; IR (neat) ν_{max}: 2959, 2878, 1744, 1651, 1589, 1458, 1400, 1258, 1173, 1126, 1045, 1003 cm^{-1}; HRMS (FAB) m/z: [M + Na]$^+$ Calcd for $C_{20}H_{33}Cl_3O_4Si_2Na$ 521.0881; found 521.0883.



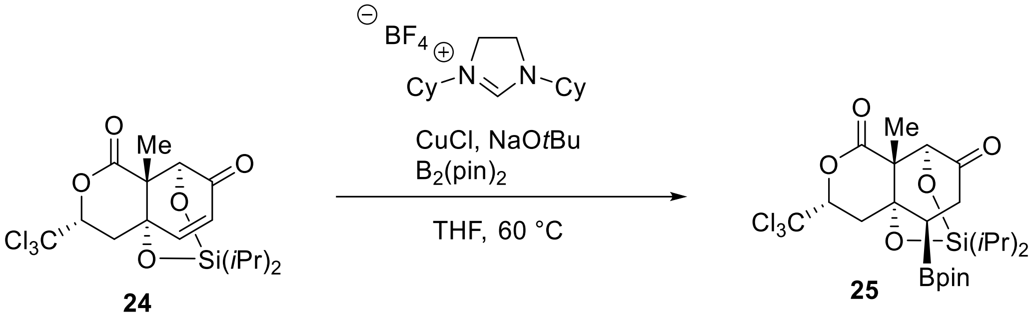
SI-1-TES **23**

Diol 23: To a stirred suspension of **SI-1-TES** (5.0 g, 10 mmol) and $NaHCO_3$ (8.4 g, 100 mmol) in acetone/H_2O = 4:1 (80 mL : 20 mL) at −15 °C was added Oxone® (15.0 g, 25 mmol). The reaction mixture was stirred at 23 °C for 3 h, and Oxone® (15.0 g, 25 mmol) was additionally added to the mixture at −15 °C. The reaction mixture was stirred at 23 °C for 6 h, before it was quenched with water (500 mL) at 0 °C. The aqueous layer was extracted with AcOEt (100 mL × 3). The combined organic layers were washed brine (50 mL), dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography

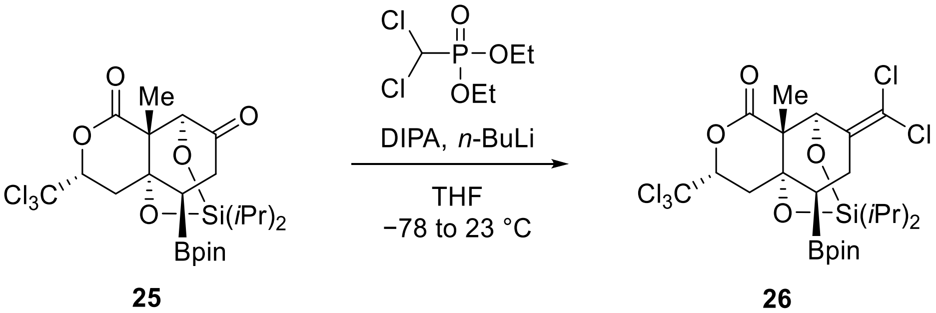
Hexanes-EtOAc) to afford product **23** (1.8 g, 55%) as white solids. R_f = 0.30 (40% AcOEt/hexane, potassium permanganate); mp 210 °C decomp (hexane/acetone); $[\alpha]_D^{23}$ = –45.5 (*c* = 1.12, acetone); ^{1}H NMR (500 MHz, acetone-*d6*) δ 6.96 (d, *J* = 10.3 Hz, 1H), 6.71 (br s, 1H), 6.02 (d, *J* = 10.2 Hz, 1H), 5.50 (dd, *J* = 8.6, 6.8 Hz, 1H), 5.31 (br s, 1H), 4.34 (s, 1H), 2.66 (d, *J* = 14.6, 8.9 Hz, 1H), 2.41 (dd, *J* = 14.6, 6.6 Hz, 1H), 1.30 (s, 3H); ^{13}C NMR (125 MHz, acetone-*d6*) δ 195.8, 169.8, 148.4, 126.5, 100.7, 83.1, 77.1, 71.6, 49.7, 35.3, 17.1; IR (neat) ν_{max}: 3507, 3456, 1747, 1697, 1411, 1219, 1168, 1123, 1022 cm^{-1}; HRMS (FAB) m/z: $[M + Na]^+$ Calcd for $C_{11}H_{11}Cl_3O_5Na$ 350.9570; found 350.9573.



Silyl protected enone 24: To a stirred solution of **23** (1.1 g, 3.34 mmol) in CH_2Cl_2 (40 mL) was added DIPEA (2.3 mL, 13.4 mmol) and dichlorodiisopropylsilane (0.9 mL, 5.0 mmol) at 0 °C. After being stirred at 23 °C for 2 h, the reaction mixture was quenched with water (10 mL). The organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (10 mL × 2). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel chromatography to afford **24** (1.3 g, 90 %) as white amorphous. R_f = 0.45 (30% AcOEt/hexane, potassium permanganate); $[\alpha]_D^{23}$ = +17.2 (*c* = 1.16, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73 (d, *J* = 10.0 Hz, 1H), 6.13 (dd, *J* = 10.0, 1.4 Hz, 1H), 5.07 (dd, *J* = 8.7, 7.3 Hz, 1H), 4.61 (d, *J* = 1.2 Hz, 1H), 2.66 (dd, *J* = 14.9, 7.2 Hz, 1H), 2.45 (dd, *J* = 14.9, 8.9 Hz, 1H), 1.24 (s, 3H), 1.04–0.85 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.4, 168.3, 144.7, 127.1, 98.4, 81.9, 76.3, 70.9, 49.0, 35.4, 17.2, 17.0, 16.58, 16.55, 16.50, 14.6, 14.5; IR (neat) ν_{max}: 2947, 1762, 1705, 1462, 1389, 1376, 1219, 1115 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{17}H_{24}Cl_3O_5Si$ 441.0459; found 441.0452.



Borylated compound 25:[6] To a stirred suspension of CuCl (158 mg, 1.6 mmol) in THF (16 mL, 0.15M) were sequentially added 1,3-dicyclohexylimidazolium tetrafluoroborate (256 mg, 0.8 mmol) and NaO*t*Bu (308 mg, 3.2 mmol) at 23 °C. After being stirred for 40 min, **24** (707 mg, 1.6 mmol) and B_2Pin_2 (813 mg, 3.2 mmol) was added to the reaction mixture. The reaction mixture was warmed up to 60 °C and stirred for 3 h, before the reaction was quenched with saturated aqueous NH_4Cl (10 mL) and diluted with Et_2O (50 mL). The organic layer was separated, and the aqueous layer was extracted with Et_2O (50 mL). The combined organic layers were washed with brine (10 mL) and dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel chromatography to afford product **25** (640 mg, 70%) as white amorphous. R_f = 0.35 (20% AcOEt/hexane, hanessian); $[\alpha]_D^{23}$ = –6.9 (*c* = 1.01, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.94 (dd, *J* = 9.2, 8.0 Hz, 1H), 4.43 (s, 1H), 3.40 (dd, *J* = 15.2, 7.7 Hz, 1H), 3.19 (dd, *J* = 15.8, 10.3 Hz, 1H), 2.68 (d, *J* = 15.5 Hz, 1H), 2.51 (dd, *J* = 15.3, 9.3 Hz, 1H), 2.17 (d, *J* = 10.0 Hz, 1H), 1.24 (s, 12H), 1.15 (s, 3H), 1.09–0.93 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 206.0, 170.0, 98.5, 84.5, 82.7, 79.2, 76.96, 50.4, 36.7, 33.9, 24.7, 24.6, 17.7, 17.5, 17.4, 16.9, 16.8, 16.5, 14.0; IR (neat) ν_{max}: 2947, 2866, 1763, 1728, 1462, 1354, 1327, 1215, 1130 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{23}H_{37}BCl_3O_7Si$ 569.1467; found 569.1460.



Dichloroalkene 26: Preparation of LDA solution: To a solution of diisopropylamine (63 μL, 0.45 mmol) in THF (0.75 mL) was added a 1.6 M solution of butyllithium in hexane (0.25 mL, 0.40 mmol) at −78 °C.

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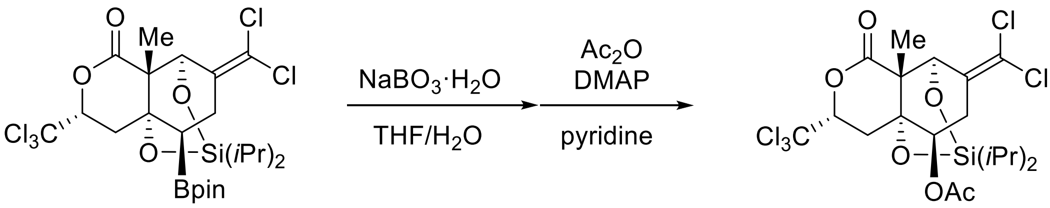
[6] Li, H.; Chen, Q.; Lu, Z.; Li, A. *J. Am. Chem. Soc.* **2016**, *138*, 15555–15558.

To a solution of diethyl (dichloromethyl)phosphonate[7] (132 mg, 0.60 mmol) and **25** (113 mg, 0.20 mmol) in THF (1.0 mL) was slowly added a solution of LDA via a canulla at −78 °C. The resulting mixture was stirred at −78 °C for 2 h. The mixture was warmed up to 23 °C and quenched with saturated aqueous NH_4Cl (2 mL) and diluted with AcOEt (10 mL). The organic layer was separated, and the aqueous layer was extracted with AcOEt (10 mL). The combined organic layers were washed with brine (2 mL), dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane/AcOEt) to afford product **26** (50.0 mg, 40%) as colorless amorphous. R_f = 0.52 (15% AcOEt/hexane, hanessian); $[\alpha]_D^{23}$ = +29.3 (*c* = 1.2, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.42 (s, 1H), 4.96 (dd, *J* = 8.5, 8.5 Hz, 1H), 3.29 (dd, *J* = 15.0, 8.2 Hz, 1H), 3.16 (d, *J* = 15.2 Hz, 1H), 2.67 (dd, *J* = 15.3, 7.9 Hz, 1H), 2.38 (dd, *J* = 15.2, 8.9 Hz, 1H), 1.83 (d, *J* = 7.5 Hz, 1H), 1.25 (s, 12H), 1.17 (s, 3H), 1.08–0.91 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 133.5, 118.9, 98.7, 84.2, 82.6, 76.7, 73.8, 49.9, 36.4, 24.74, 24.69, 24.6, 17.9, 17.8, 17.6, 17.0, 16.8, 16.6, 13.8; IR (neat) ν_{max}: 2943, 2886, 1763, 1620, 1462, 1358, 1327, 1242, 1215, 1135, 1053, 995 cm^{-1}; HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{36}BCl_5O_6SiNa$ 657.0709; found 657.0703.



26 **27**

Acetate 27: To a stirred solution of **26** (95.0 mg, 0.15 mmol) in (2.0 mL, THF/H_2O = 1:1) was added $NaBO_3 \cdot 4H_2O$ (230 mg, 1.5 mmol) at 23 °C. After being stirred at 23 °C for 1 h, the reaction mixture was diluted with water (5 mL) and extracted with EtOAc (5 mL × 3). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure to afford the crude alcohol. The crude was dissolved in pyridine (1.5 mL) and DMAP (1.9 mg, 15 μmol) and acetic anhydride (43.0 μL, 0.45 mmol) was added to the mixture at 23 °C. After being stirred at 23 °C for 2 h, the mixture was quenched with water (10 mL) and diluted with AcOEt (10 mL). The organic layer was separated, and the aqueous layer was extracted with AcOEt (5 mL× 3). The combined organic layers were washed with H_2O (5 mL× 3), brine (10 mL) and dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography to afford product **27** (66.1 mg, 78%) as white solids. R_f = 0.25 (15%

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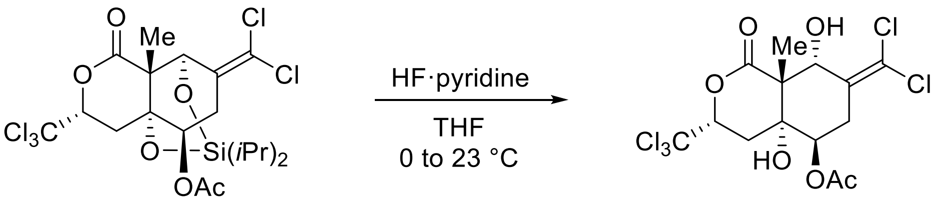
[7] Marinetti, A.; Savignac, P. *Org. Synth.,* **1997**, *74*, 108.

AcOEt/hexane, hanessian); mp 230−232 °C (hexane/AcOEt); $[\alpha]_D^{23}$ = −7.2 (*c* = 1.3, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.52 (s, 1H), 5.00 (t, *J* = 8.6 Hz, 1H), 4.99 (dd, *J* = 4.3, 1.7 Hz, 1H), 3.10 (d, *J* = 16.6 Hz, 1H), 2.95 (dd, *J* = 16.9, 4.3 Hz, 1H), 2.57 (dd, *J* = 14.9, 8.6 Hz, 1H), 2.29 (dd, *J* = 15.0, 8.2 Hz, 1H), 2.13 (s, 3H), 1.34 (s, 3H), 1.18-0.90 (m, 14H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.0, 169.2, 130.7, 120.6, 98.4, 82.3, 75.3, 73.6, 73.1, 49.3, 33.0, 28.4, 21.2, 17.59, 17.56, 17.1, 16.74, 16.71, 16.4, 14.2; IR (neat) ν_{max}: 2947, 2866, 1759, 1735, 1620, 1458, 1431, 1369, 1307, 1222, 1149 cm^{-1}; HRMS (ESI) m/z: [M + H]$^+$ Calcd for $C_{20}H_{28}Cl_5O_6Si$ 567.0092; found 567.0092.



27 sigillin A (**1**)

Sigillin A: To a stirred solution of **27** (68.0 mg, 0.12 mmol) in THF (2.0 mL) was added HF·pyridine (14.8 μL, 0.48 mmol) at 0 °C. After being stirred at 23 °C for 30 min, the reaction mixture was quenched with water (5 mL) and diluted with AcOEt (10 mL). The organic layer was separated, and the aqueous layer was extracted with AcOEt (5 mL). The combined organic layers were washed with brine (10 mL) and dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford (−)-sigillin A (**1**) (46.1 mg, 84%) as white solids. R_f = 0.21 (20% AcOEt/hexane, hanessian); mp 245−249 °C (hexane/Et_2O); $[\alpha]_D^{23}$ = −42.9 (*c* = 0.91, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.32 (d, *J* = 3.7 Hz, 1H), 5.21 (d, *J* = 2.3 Hz, 1H), 5.09 (dd, *J* = 8.6, 7.5 Hz, 1H), 5.00 (dd, *J* = 3.0, 3.0 Hz, 1H), 3.80 (d, *J* = 3.7 Hz, 1H), 3.15 (ddd, *J* = 15.8, 2.7, 1.0 Hz, 1H), 2.92 (dd, *J* = 16.0, 3.2 Hz, 1H), 2.50 (ddd, *J* = 14.8, 8.7, 2.6 Hz, 1H), 2.18 (dd, *J* = 14.5, 7.3 Hz, 1H), 2.12 (s, 3H), 1.34 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 169.2, 129.3, 121.8, 98.3, 82.7, 74.1, 73.4, 73.0, 50.0, 31.8, 26.9, 21.1, 15.3; IR (neat) ν_{max}: 3368, 3020, 2958, 1743, 1628, 1420, 1373, 1350, 1226, 1145, 1095, 1026 cm^{-1}; HRMS (FAB) m/z: [M + H]$^+$ Calcd for $C_{14}H_{16}Cl_5O_6$ 454.9390; found 454.9395.

3. Comparison of NMR Spectroscopic Data Natural and Synthetic Sigillin A

Table S1. Comparison of the isolated and our ^{1}H NMR data for sigillin A.

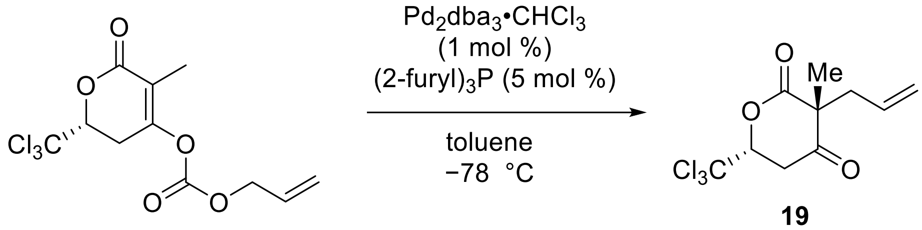
Natural sigillin A ^{1}H NMR Data: 600 MHz, $CDCl_3$	Our ^{1}H NMR Data: 500 MHz, $CDCl_3$
5.32 (dd, 1H, J = 3.8, 1.0 Hz)	5.32 (d, 1H, J = 3.7 Hz)
5.24 (dd, 1H, J = 2.4, 0.9 Hz)	5.21 (d, 1H, J = 2.3 Hz)
5.09 (dd, 1H, J = 8.8, 7.3 Hz)	5.09 (dd, 1H, J = 8.6, 7.5 Hz)
5.00[8] (dd, 1H, J = 3.2, 2.7 Hz)	5.00 (dd, 1H, J = 3.0, 3.0 Hz)
3.90 (d, 1H, J = 3.8 Hz)	3.80 (d, 1H, J = 3.7 Hz)
3.16 (ddd, 1H, J = 15.8, 2.7, 1.0 Hz)	3.15 (ddd, 1H, J = 15.8, 2.7, 1.0 Hz)
2.93 (dd, 1H, J = 15.8, 3.2 Hz)	2.92 (dd, 1H, J = 16.0, 3.2 Hz)
2.50 (ddd, 1H, J = 14.7, 8.8, 2.4 Hz)	2.50 (ddd, 1H, J = 14.8, 8.7, 2.6 Hz)
2.18 (ddd, 1H, J = 14.7, 7.3, 0.9 Hz)	2.18 (dd, 1H, J = 14.5, 7.3 Hz)
2.12 (s, 3H)	2.12 (s, 3H)
1.34 (s, 3H)	1.34 (s, 3H)

Table S2. Comparison of the isolated and our ^{13}C NMR data for sigillin A.

Isolated sigillin A ^{13}C NMR Data: 151 MHz, $CDCl_3$	Our ^{13}C NMR Data: 125 MHz, $CDCl_3$
171.8	171.7
169.2	169.2
129.3	129.3
121.9	121.8
98.3	98.3
82.7	82.7
74.1	74.1
73.4	73.4
73.0	73.0
50.0	50.0
31.8	31.8
27.0	26.9
21.1	21.1
15.3	15.3

[8] This chemical shift is assumed to be a typographic error. Their data for the isolated material indicated a chemical shift of 5.00 ppm.

4. Optimization Conditions of Allylation

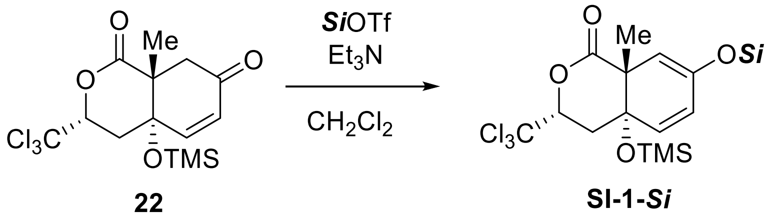


1.0 mmol scale optimization of reaction conditions: A solution of $Pd_2dba_3 \cdot CHCl_3$ and ligand in toluene was stirred for 10 min at 23 °C. Then, a solution of **18** in toluene was slowly added to the mixture at –78 °C. After the mixture was gradually warmed up to 23 °C, the solvent was evaporated. The residue was purified by column chromatography on silica gel (hexane/AcOEt) to afford product **19**.

entry	Pd (x mol %)	Ligand (x mol %)	solvent	temp	Yield (%)	dr
1	$Pd(PPh_3)_4$ (3)	none	THF	–78	87	73:27
2	$Pd(PPh_3)_4$ (3)	none	toluene	–78	93	83:17
3	Pd_2dba_3 (3)	PPh_3 (15)	toluene	–78	90	85:15
4	Pd_2dba_3 (3)	$(2\text{-furyl})_3P$ (15)	toluene	–78	97	94:6
5	Pd_2dba_3 (3)	$(2\text{-thienyl})_3P$ (15)	toluene	–78	99	87:13
6	Pd_2dba_3 (3)	$(c\text{-hex})_3P$ (15)	toluene	–78	10	81:19
7	Pd_2dba_3 (3)	dppe (7.5)	toluene	–78	90	81:19
8	Pd_2dba_3 (3)	dppf (7.5)	toluene	–78	95	89:11
9	Pd_2dba_3 (3)	none	toluene	–78	10	63:37
10	Pd_2dba_3 (2)	$(2\text{-furyl})_3P$ (10)	toluene	–78	96	93:7
11	Pd_2dba_3 (1)	$(2\text{-furyl})_3P$ (5)	toluene	–78	96	94:6

5. Optimization Conditions of Rubottom Oxidation

Preparation of Siloxydienes:



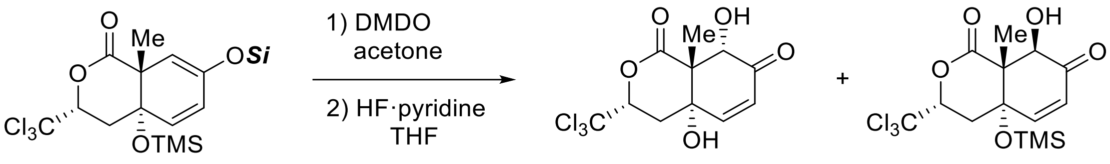
(**siloxydiene SI-1-TMS, TBS, TES**); To a solution of **22** (386 mg, 1.0 mmol) and triethylamine (1.1 mL, 8.0 mmol) in CH_2Cl_2 (10 mL) was added SiOTf (4.0 mmol) at 0 °C. After being stirred at 23 °C for 9 h, to the resulting mixture was quenched with saturated aqueous $NaHCO_3$ (10 mL). The organic layer was separated, and the aqueous layer was extracted with $CHCl_3$ (10 mL). The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford the corresponding siloxydiene **SI-1-*Si*** as white solids.

(**SI-1-TMS**) (264 mg, 58% yield) R_f = 0.28 (10% AcOEt/hexane, UV, potassium permanganate); mp 208–210 °C (hexane/AcOEt); $[\alpha]_D^{23}$ = +230.3 (*c* = 1.00, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.91 (dd, *J* = 9.6, 2.2 Hz, 1H), 5.74 (d, *J* = 9.5 Hz, 1H), 5.52 (d, *J* = 2.0 Hz, 1H), 5.05 (dd, *J* = 8.5, 7.0 Hz, 1H), 2.49–2.31 (m, 2H), 1.28 (s, 3H), 0.26 (s, 9H), 0.03 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 146.3, 129.9, 127.2, 108.7, 99.1, 83.1, 72.2, 50.3, 35.9, 18.5, 1.9, 0.2; IR (neat) ν_{max}: 2959, 2901, 1743, 1651, 1589, 1400, 1253, 1173, 1126, 1042 cm^{-1}; HRMS (FAB) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{27}Cl_3O_4Si_2Na$ 479.0411; found 479.0413.

(**SI-1-TBS**) (328 mg, 66% yield) R_f = 0.25 (10% AcOEt/hexane, UV, potassium permanganate); mp 214–217 °C (hexane/AcOEt); $[\alpha]_D^{23}$ = +179.3 (*c* = 0.93, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.90 (dd, *J* = 9.6, 1.9 Hz, 1H), 5.74 (d, *J* = 9.5 Hz, 1H), 5.49 (s, 1H), 5.05 (dd, *J* = 8.3, 7.2 Hz, 1H), 2.46–2.34 (m, 2H), 1.27 (s, 3H), 0.93 (s, 9H), 0.22 (s, 3H), 0.20 (s, 3H), 0.03 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.0, 146.5, 130.0, 127.1, 108.7, 99.2, 83.1, 72.3, 50.3, 35.9, 25.5, 18.4, 17.9, 1.9, –4.56, –4.73; IR (neat) ν_{max}: 2955, 2931, 2858, 1744, 1651, 1589, 1462, 1400, 1253, 1123, 1046 cm^{-1}; HRMS (FAB) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{33}Cl_3O_4Si_2Na$ 521.0881; found 521.0876.

(**SI-1-TES**) (436 mg, 87% yield) R_f = 0.25 (10% AcOEt/hexane, UV, potassium permanganate); mp 147–149 °C (hexane); $[\alpha]_D^{23}$ = +219.3 (*c* = 1.05, acetone); ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.92 (dd, *J* = 9.7, 2.0 Hz, 1H), 5.74 (d, *J* = 9.5 Hz, 1H), 5.52 (d, *J* = 1.7 Hz, 1H), 5.05 (dd, *J* = 8.5, 6.7 Hz, 1H), 2.47–2.29 (m, 2H), 1.27 (s, 3H), 1.00 (t, J = 7.9 Hz, 9H), 0.80-0.69 (m, 6H), 0.03 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.9, 146.5, 129.9, 127.1, 108.2, 99.2, 83.0, 72.3, 50.2, 35.8, 18.4, 6.6, 4.8, 1.9; IR (neat) ν_{max}: 2959, 2878, 1744, 1651, 1589, 1458, 1400, 1258, 1173, 1126, 1045, 1003 cm^{-1}; HRMS (FAB) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{33}Cl_3O_4Si_2Na$ 521.0881; found 521.0883.

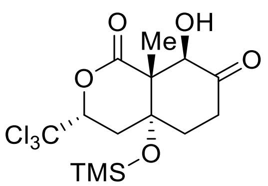
Optimization Conditions of Rubottom Oxidation of SI-1-*Si* with DMDO:



SI-1-*Si* **23** **SI-2**

General procedure: To a solution of the siloxydiene (0.3 mmol) in acetone (1 mL) was added a 0.06 M acetone solution DMDO (10 mL, 0.6 mmol) at –78 °C. After being stirred at 0 °C for 24 h, removal of volatile in vacuo to afford the crude. The crude was dissolved in THF (3 mL) and HF·pyridine (37 μL, 1.2 mmol) was added at 0 °C. After being stirred at 23 °C for 1 h, the reaction mixture was quenched with water (5 mL) and diluted with AcOEt (10 mL). The organic layer was separated, and the aqueous layer was extracted with AcOEt (5 mL). The combined organic layers were washed with brine (2 mL) and dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/AcOEt) to afford the desired diol **23** and **29**.

entry	reagents	Si	solvent	Temp (°C)	yield (%)	**23**:**SI-2**
1	*m*CPBA	TMS	CH_2Cl_2	23	14	67:23
2	DMDO	TMS	acetone	0	69	58:42
3	DMDO	TES	acetone	0	48	95:5
4	DMDO	TBS	acetone	0	15	95:5



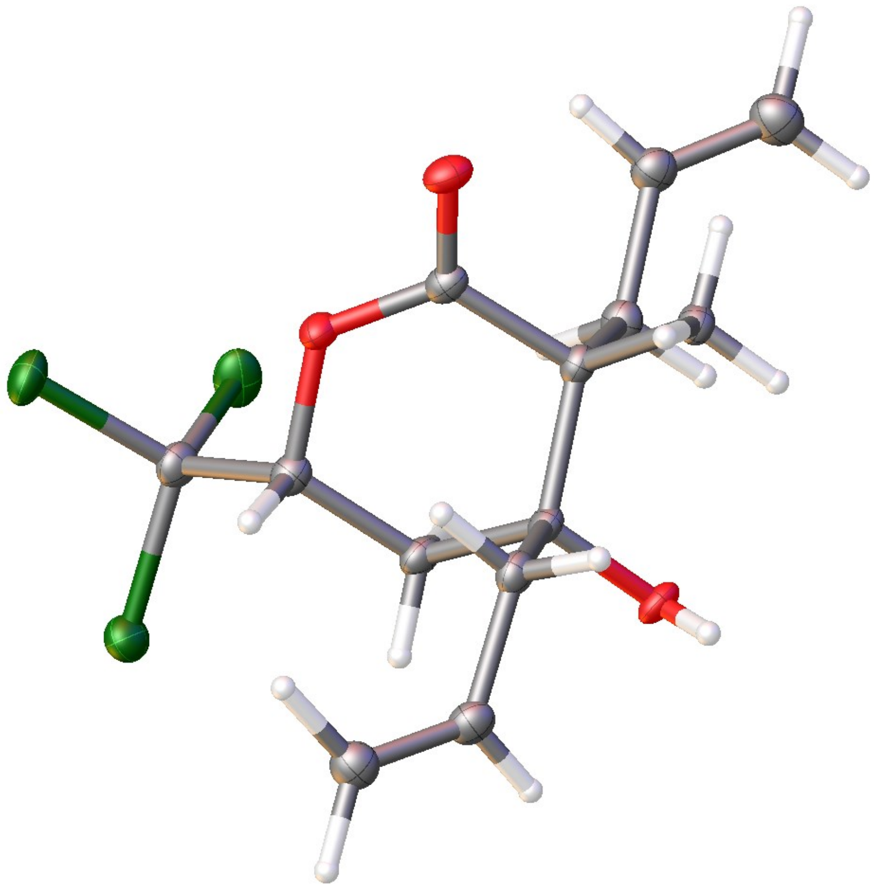
(**SI-2**): as amorphous. R_f = 0.28 (40% AcOEt/hexane, potassium permanganate); ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73 (d, J = 10.0 Hz, 1H), 6.23 (d, J = 10.0 Hz, 1H), 5.18 (dd, J = 8.6, 6.9 Hz, 1H), 5.08 (s, 1H), 3.78 (d, J = 2.6 Hz, 1H), 2.56 (dd, J = 14.6, 6.9 Hz, 1H), 2.49 (dd, J = 14.9, 8.6 Hz, 1H), 1.30 (s, 3H), 0.15 (s, 9H); ^{13}C NMR (125 MHz) δ 195.8, 170.6, 143.1, 129.4, 98.5, 82.2, 74.2, 73.8, 54.9, 35.9, 11.1, 1.9; IR (neat) ν_{max}: 3541, 2959, 1751, 1694, 1396, 1254, 1119 cm^{-1}; HRMS (FAB) m/z: $[M + H]^+$ Calcd for $C_{14}H_{20}Cl_3O_5Si$ 401.0146; found 401.0144.

6. X-ray Crystallographic Data

Data collection and Structure solution details: Single crystal X-ray data for compound **20**, **23** and **1** were collected on a Rigaku XtaLaB P200 diffractometer Cu-Kα radiation. Data collection, cell refinement, data reduction and analysis were carried out with the CrysAlisPro (Rigaku Oxford Diffraction). These structures were solved by intrinsic phasing methods with the SHELXT program and refines using SHELXL[9] with anisotropic displacement parameters for non-H atoms. CCDC 2015184, 2015183 and 2015179 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif

X-ray crystallographic data for compound **20** (CCDC 2015184).

Single crystals of **20** were obtained by slow evaporation of a solution containing **20** in the mixture of hexane and ethyl acetate at room temperature. A suitable crystal was selected and the crystal data and structure refinement results for compound **20** are listed in the Table S1.



[9] Sheldrick, G. M. A. Short History of SHELX. *Acta Crystallogr., Sect. A: Found. Crystallogr.* **2008**, *64*, 112– 122.

Figure S1. ORTEP view of the compound **20** with thermal ellipsoids drawn at the 80% probability level

Table S3. Crystal data and structure refinement for **20**.

Identification code	20171202tn
Empirical formula	$C_{13}H_{13}Cl_3O_3$
Formula weight	323.604
Temperature/K	293
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	5.9465(3)
b/Å	12.4317(5)
c/Å	20.1035(9)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1486.15(12)
Z	4
ρ_{calc}g/cm^3	1.446
μ/mm^{-1}	5.601
F(000)	670.2
Crystal size/mm^3	0.1 × 0.1 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.36 to 146.76
Index ranges	$-6 \le h \le 6, -14 \le k \le 15, -14 \le l \le 24$
Reflections collected	5395
Independent reflections	2685 [R_{int} = 0.0418, R_{sigma} = 0.0447]
Data/restraints/parameters	2685/0/190
Goodness-of-fit on F^2	1.010
Final R indexes [I>=2σ (I)]	R_1 = 0.0451, wR_2 = 0.1244
Final R indexes [all data]	R_1 = 0.0470, wR_2 = 0.1260
Largest diff. peak/hole / e Å^{-3}	0.27/-0.48
Flack parameter	-0.06(2)

X-ray crystallographic data for compound **23** (CCDC 2015183).

Single crystals of **23** were obtained by slow evaporation of a solution containing **23** in the mixture of hexane and ethyl acetate at room temperature. A suitable crystal was selected and the crystal data and structure refinement results for compound **23** are listed in the Table S2.

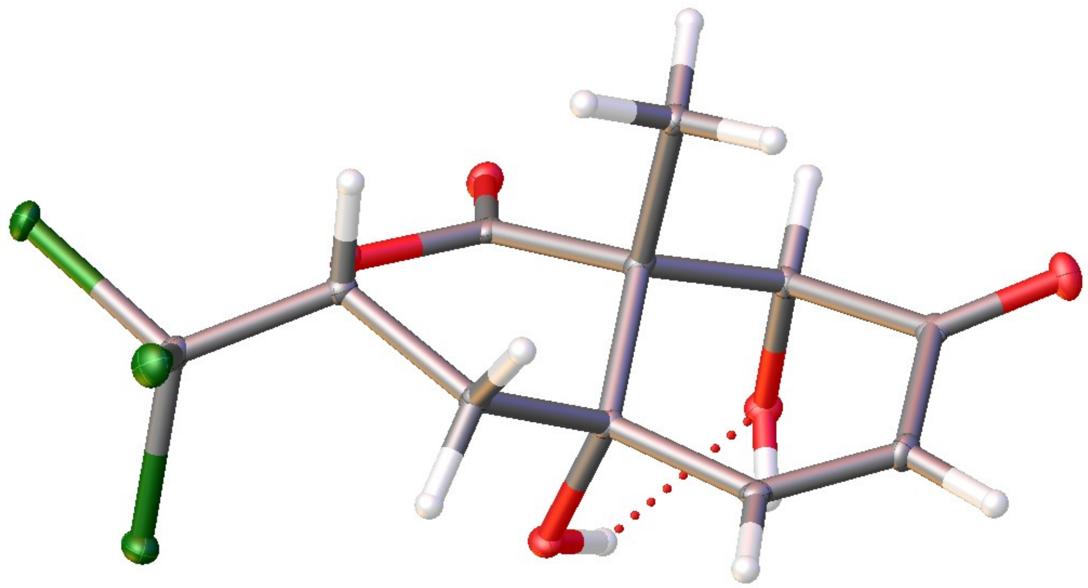


Figure S2. ORTEP view of the compound **23** with thermal ellipsoids drawn at the 80% probability level

Table S2. Crystal data and structure refinement for **23**.

Identification code	23
Empirical formula	$C_{11}H_{11}Cl_3O_5$
Formula weight	329.55
Temperature/K	93
Crystal system	monoclinic
Space group	$P2_1$
a/Å	10.9134(2)
b/Å	6.02950(10)
c/Å	10.9547(3)
α/°	90

β /°	117.801(3)
γ /°	90
Volume/Å^3	637.64(3)
Z	2
ρ_{calc}g/cm^3	1.716
μ /mm^{-1}	6.661
F(000)	336.0
Crystal size/mm^3	0.8 × 0.2 × 0.2
Radiation	CuK α (λ = 1.54184)
2Θ range for data collection/°	9.126 to 146.944
Index ranges	-13 ≤ h ≤ 7, -7 ≤ k ≤ 7, -9 ≤ l ≤ 13
Reflections collected	2907
Independent reflections	1819 [R_{int} = 0.0303, R_{sigma} = 0.0285]
Data/restraints/parameters	1819/1/175
Goodness-of-fit on F^2	1.052
Final R indexes [I>=2 σ (I)]	R_1 = 0.0388, wR_2 = 0.1023
Final R indexes [all data]	R_1 = 0.0390, wR_2 = 0.1026
Largest diff. peak/hole / e Å^{-3}	0.72/-0.44
Flack parameter	0.04(2)

X-ray crystallographic data for compound **1** (CCDC 2015179).

Single crystals of **1** were obtained by slow evaporation of a solution containing **1** in the mixture of hexane and ethyl acetate at room temperature. A suitable crystal was selected and the crystal data and structure refinement results for compound **1** are listed in the Table S3.

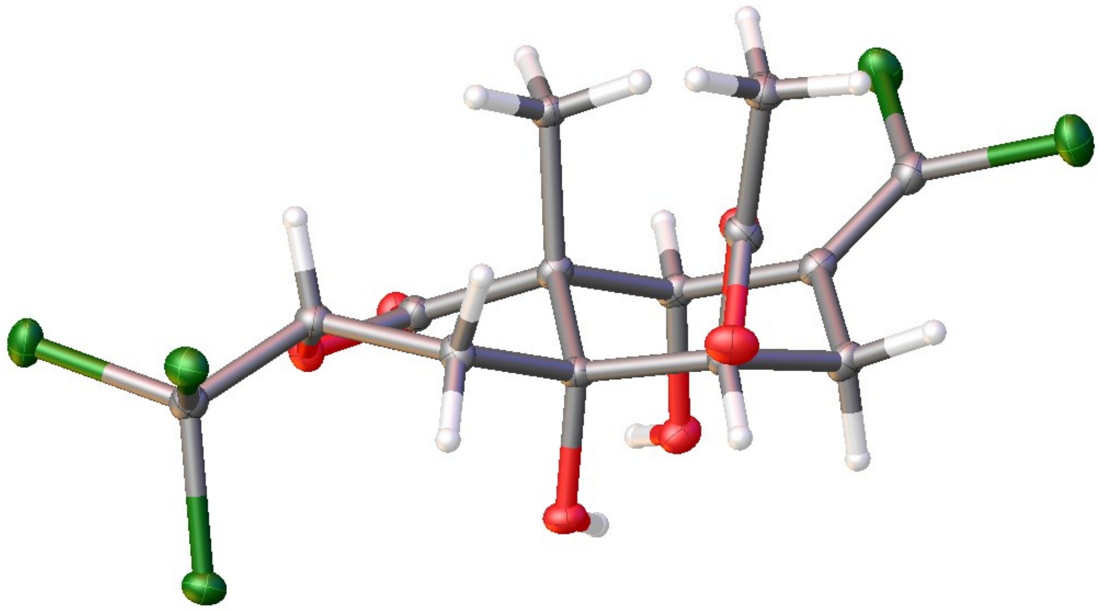


Figure S3. ORTEP view of **1** with thermal ellipsoids drawn at the 80% probability level.

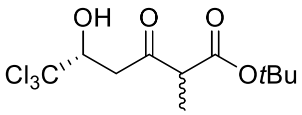
Table S3. Crystal data and structure refinement for **1**.

Empirical formula	$C_{14}H_{15}Cl_5O_6$
Formula weight	456.51
Temperature/K	293
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.27672(10)
b/Å	13.3587(2)
c/Å	18.5617(3)
α /°	90
β /°	90
γ /°	90
Volume/Å^3	1804.33(5)
Z	4
ρ_{calc}g/cm^3	1.681
μ /mm^{-1}	7.602
F(000)	928.0
Crystal size/mm^3	0.2 × 0.2 × 0.03

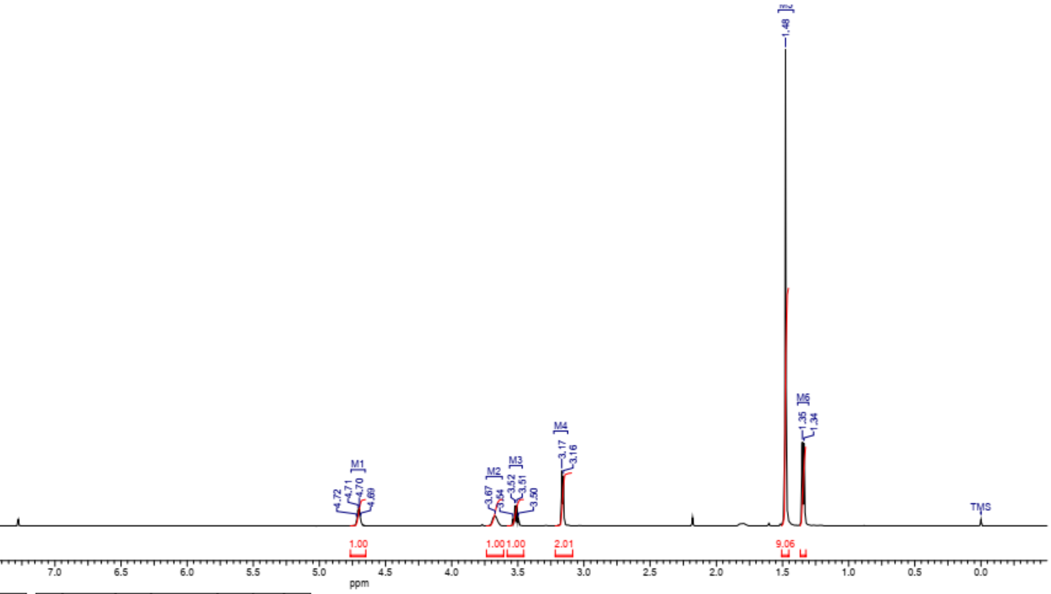
Radiation	Cu K α (λ = 1.54184)
2Θ range for data collection/°	8.154 to 146.776
Index ranges	$-8 \leq h \leq 9, -16 \leq k \leq 15, -23 \leq l \leq 23$
Reflections collected	17428
Independent reflections	3578 [R_{int} = 0.0420, R_{sigma} = 0.0206]
Data/restraints/parameters	3578/0/231
Goodness-of-fit on F^2	1.158
Final R indexes [I>=2 σ (I)]	R_1 = 0.0338, wR_2 = 0.0951
Final R indexes [all data]	R_1 = 0.0340, wR_2 = 0.0951
Largest diff. peak/hole / e Å^{-3}	0.39/-0.40
Flack parameter	0.001(10)

7. [1]H and [13]C NMR Spectra of New Compounds

[1]H NMR, $CDCl_3$, 500MHz



17

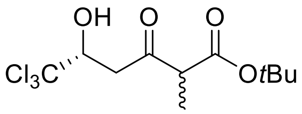


No.	(ppm)	(Hz)	Height
1	1.34	668.2	0.1736
2	1.35	675.3	0.1762
3	1.48	738.6	1.0000
4	3.16	1578.6	0.1046
5	3.17	1584.4	0.1159
6	3.50	1748.2	0.0153
7	3.51	1755.3	0.0436
8	3.52	1762.2	0.0427
9	3.54	1769.4	0.0140
10	3.67	1837.3	0.0205
11	4.69	2345.0	0.0156
12	4.70	2349.9	0.0350
13	4.71	2354.8	0.0330
14	4.72	2359.9	0.0139

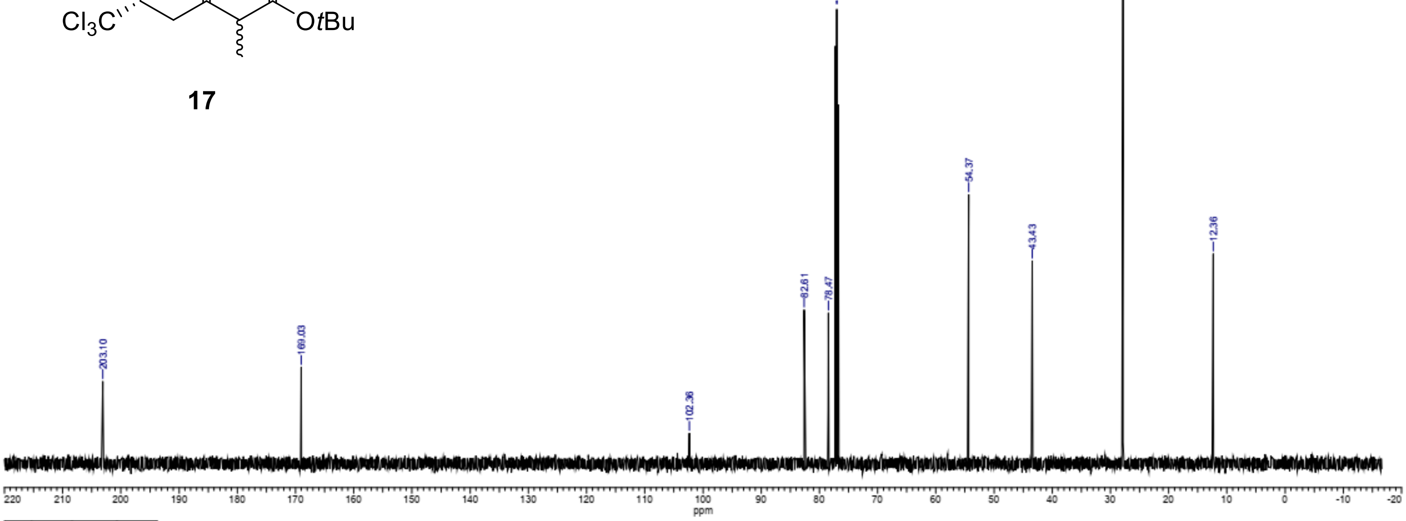
No.	(ppm)	Value	Absolute Value
1	[1.32 .. 1.37]	3.022	1.62693e+6
2	[1.45 .. 1.51]	9.062	4.87927e+6
3	[3.09 .. 3.22]	2.011	1.08305e+6
4	[3.46 .. 3.58]	1.004	5.40516e+5
5	[3.61 .. 3.74]	1.003	5.40242e+5
6	[4.65 .. 4.77]	0.997	5.37060e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M6	1.34	[1.30 .. 1.39]	7.16	d	3
2	M5	1.47	[1.42 .. 1.53]	-	s	9
3	M4	3.18	[3.12 .. 3.23]	5.73	d	2
4	M3	3.52	[3.47 .. 3.56]	7.16	q	1
5	M3	3.52	[3.47 .. 3.56]	7.16	q	1
6	M3	3.52	[3.47 .. 3.56]	7.16	q	1
7	M2	3.68	[3.62 .. 3.74]	-	s	1
8	M1	4.71	[4.65 .. 4.76]	4.96	q	1
9	M1	4.71	[4.65 .. 4.76]	4.96	q	1
10	M1	4.71	[4.65 .. 4.76]	4.96	q	1

[13]C NMR, $CDCl_3$, 125MHz

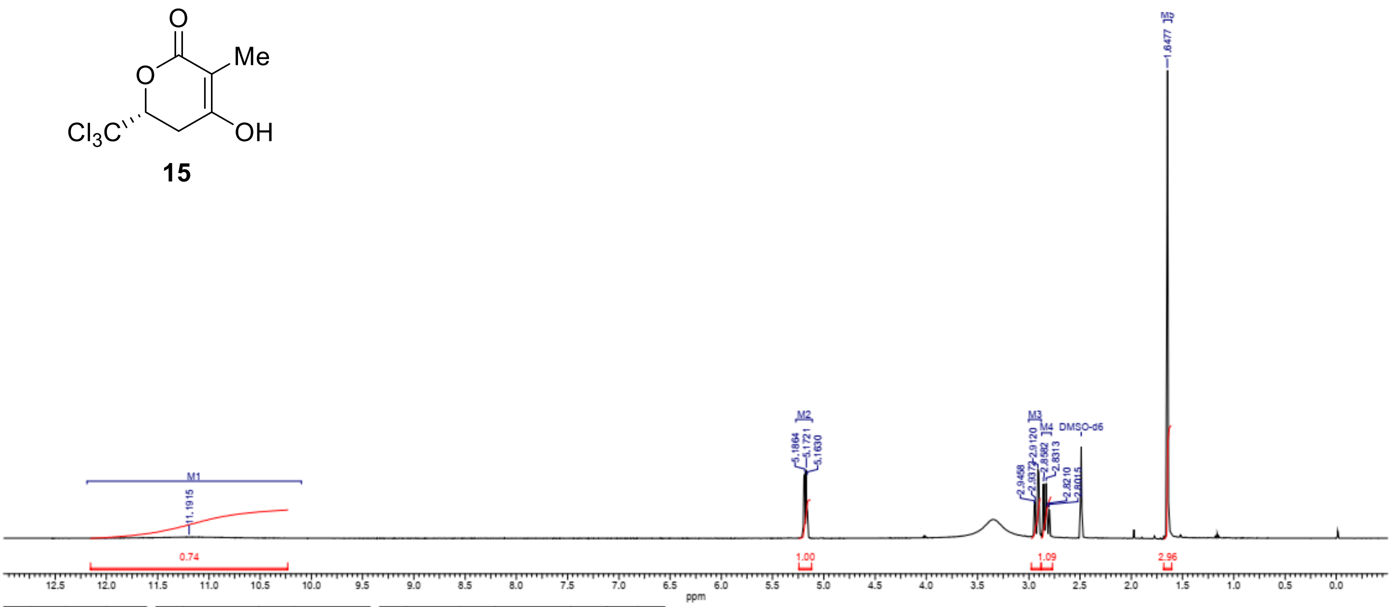


17



No.	(ppm)	(Hz)	Height
1	12.36	1554.4	0.4287
2	27.85	3502.6	1.0000
3	43.43	5462.8	0.4142
4	54.37	6837.6	0.5501
5	78.47	9869.0	0.3083
6	82.61	10389.7	0.3144
7	102.36	12874.1	0.0621
8	169.03	21258.4	0.1971
9	203.10	25543.5	0.1682

^{1}H NMR, DMSO-*d6*, 500MHz

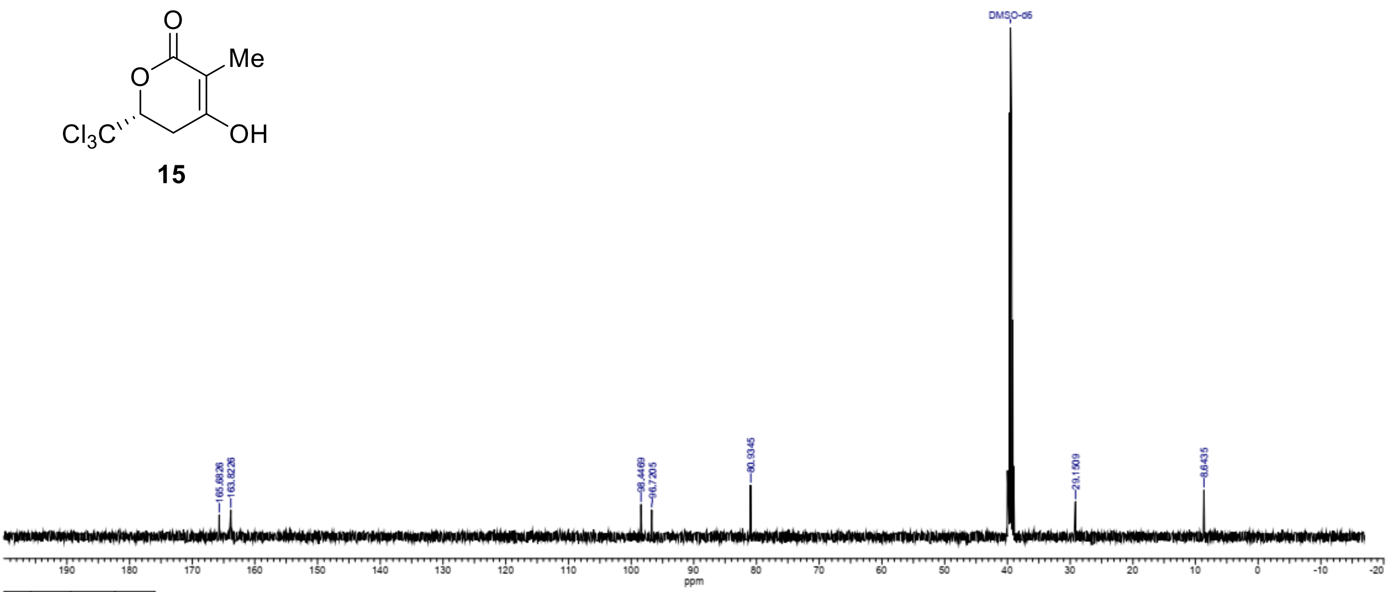


No.	(ppm)	(Hz)	Height
1	1.65	824.1	1.0000
2	2.80	1399.2	0.0604
3	2.80	1401.2	0.0616
4	2.82	1410.9	0.0632
5	2.82	1412.9	0.0739
6	2.83	1416.1	0.1177
7	2.84	1418.1	0.1100
8	2.85	1427.8	0.1144
9	2.86	1429.6	0.1166
10	2.90	1452.2	0.1419
11	2.91	1456.5	0.1466
12	2.94	1469.1	0.0766
13	2.95	1473.4	0.0688
14	5.16	2582.3	0.1306
15	5.17	2586.9	0.1461
16	5.19	2594.1	0.1370
17	5.20	2598.4	0.1302
18	11.19	5597.5	0.0026

No.	(ppm)	Value	Absolute Value
1	[1.61 .. 1.68]	2.956	2.92603e+6
2	[2.77 .. 2.88]	1.091	1.08012e+6
3	[2.88 .. 2.97]	1.063	1.05259e+6
4	[5.12 .. 5.24]	1.000	9.89813e+5
5	[10.23 .. 12.16]	0.736	7.28201e+5

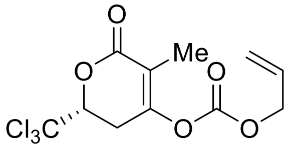
No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M5	1.64	[1.61 .. 1.68]	-	s	3
2	M4	2.83	[2.78 .. 2.87]	-	m	1
3	M3	2.94	[2.88 .. 3.00]	-	m	1
4	M2	5.19	[5.11 .. 5.27]	11.60	dd	1
5	M2	5.19	[5.11 .. 5.27]	4.44	dd	1
6	M1	11.14	[10.10 .. 12.19]	-	s	1

^{13}C NMR, DMSO-*d6*, 125MHz

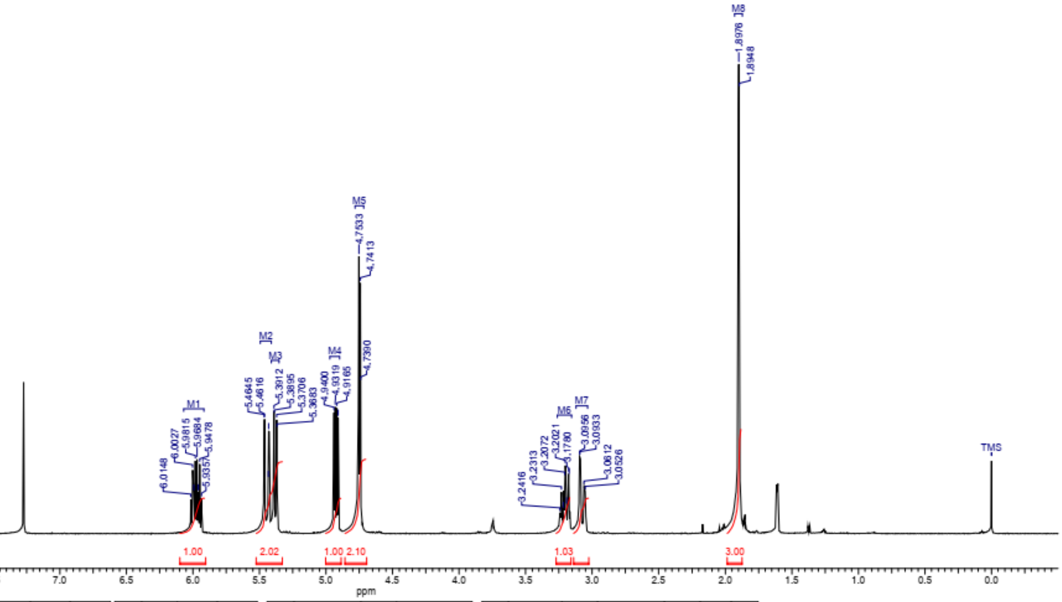


No.	(ppm)	(Hz)	Height
1	8.64	1087.1	0.0918
2	29.15	3666.3	0.0684
3	80.93	10179.1	0.1012
4	96.72	12164.5	0.0524
5	98.45	12381.7	0.0634
6	163.82	20604.0	0.0514
7	165.68	20837.9	0.0421

[1]H NMR, $CDCl_3$, 500MHz



18

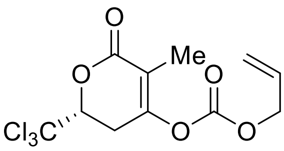


No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	1.89	946.6	0.8179	17	3.19	1596.4	0.0590	33	4.91	2454.7	0.2462	49	5.46	2730.2	0.1104
2	1.89	947.7	0.9480	18	3.20	1599.0	0.1271	34	4.92	2459.0	0.2672	50	5.46	2731.7	0.2434
3	1.90	949.1	1.0000	19	3.20	1601.5	0.1448	35	4.93	2466.8	0.2693	51	5.46	2733.1	0.2421
4	1.90	950.3	0.9072	20	3.21	1604.1	0.1195	36	4.94	2470.8	0.2578	52	5.47	2734.3	0.1290
5	3.05	1525.7	0.0832	21	3.21	1606.7	0.0906	37	5.37	2684.1	0.0934	53	5.94	2968.8	0.0743
6	3.05	1526.8	0.0887	22	3.22	1609.3	0.0494	38	5.37	2685.0	0.2221	54	5.95	2974.8	0.1476
7	3.06	1529.9	0.0991	23	3.23	1613.6	0.0461	39	5.37	2686.1	0.2426	55	5.96	2979.1	0.0837
8	3.06	1531.1	0.1000	24	3.23	1616.2	0.0885	40	5.37	2687.3	0.1217	56	5.96	2980.6	0.0867
9	3.09	1543.1	0.1534	25	3.24	1618.7	0.0876	41	5.39	2694.4	0.1133	57	5.97	2985.1	0.1580
10	3.09	1544.3	0.1579	26	3.24	1621.3	0.0432	42	5.39	2695.6	0.2517	58	5.98	2991.7	0.1546
11	3.09	1547.1	0.1630	27	4.74	2370.2	0.3223	43	5.39	2696.5	0.2613	59	5.99	2996.3	0.0835
12	3.10	1548.3	0.1640	28	4.74	2371.4	0.5335	44	5.39	2697.6	0.1428	60	5.99	2997.7	0.0864
13	3.17	1584.6	0.0470	29	4.74	2372.8	0.3644	45	5.42	2713.1	0.0986	61	6.00	3002.3	0.1349
14	3.17	1586.9	0.1220	30	4.75	2376.3	0.3752	46	5.43	2714.5	0.2169	62	6.01	3008.3	0.0724
15	3.18	1589.5	0.1269	31	4.75	2377.4	0.5911	47	5.43	2715.9	0.2173				
16	3.18	1592.1	0.0598	32	4.76	2378.5	0.4051	48	5.43	2717.1	0.1154				

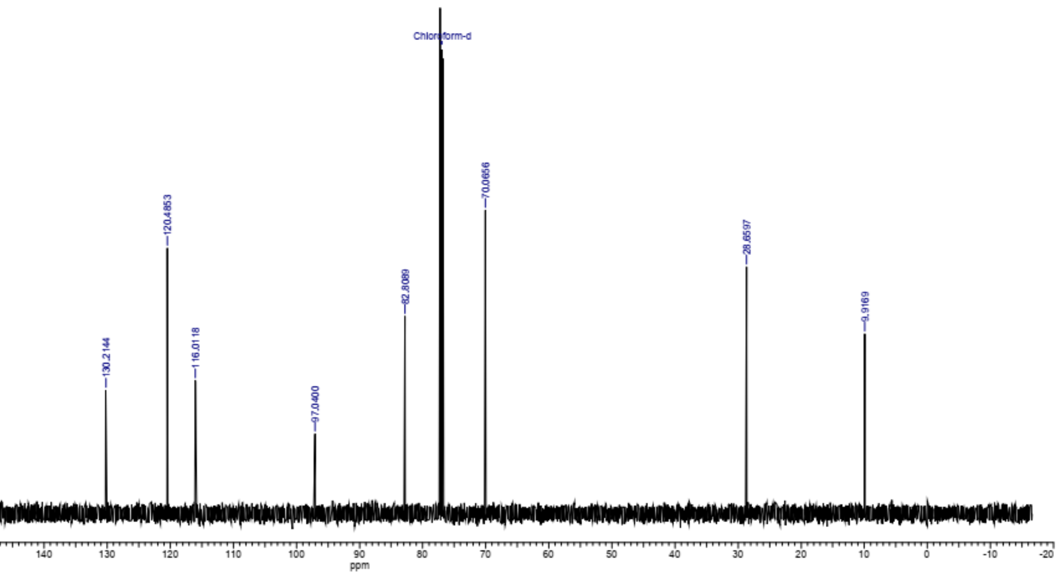
No.	(ppm)	Value	Absolute Value
1	[1.87 .. 1.99]	3.001	2.56645e+6
2	[3.03 .. 3.14]	0.997	8.52776e+5
3	[3.16 .. 3.27]	1.031	8.81359e+5
4	[4.69 .. 4.86]	2.101	1.79629e+6
5	[4.89 .. 5.00]	0.998	8.53158e+5
6	[5.33 .. 5.52]	2.023	1.72974e+6
7	[5.90 .. 6.10]	1.000	8.55125e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M8	1.90	[1.87 .. 1.93]	2.58	dd	3
2	M8	1.90	[1.87 .. 1.93]	1.15	dd	3
3	M7	3.08	[3.04 .. 3.12]	17.33	ddd	1
4	M7	3.08	[3.04 .. 3.12]	4.15	ddd	1
5	M7	3.08	[3.04 .. 3.12]	1.15	ddd	1
6	M6	3.21	[3.16 .. 3.26]	-	m	1
7	M5	4.75	[4.72 .. 4.78]	5.94	dt	2
8	M5	4.75	[4.72 .. 4.78]	1.18	dt	2
9	M5	4.75	[4.72 .. 4.78]	1.18	dt	2
10	M4	4.93	[4.90 .. 4.97]	11.89	dd	1
11	M4	4.93	[4.90 .. 4.97]	4.15	dd	1
12	M3	5.38	[5.35 .. 5.41]	10.38	ddd	1
13	M3	5.38	[5.35 .. 5.41]	2.08	ddd	1
14	M3	5.38	[5.35 .. 5.41]	1.00	ddd	1
15	M2	5.45	[5.41 .. 5.49]	17.18	ddd	1
16	M2	5.45	[5.41 .. 5.49]	2.72	ddd	1
17	M2	5.45	[5.41 .. 5.49]	1.29	ddd	1
18	M1	5.99	[5.92 .. 6.07]	-	m	1

[13]C NMR, $CDCl_3$, 125MHz

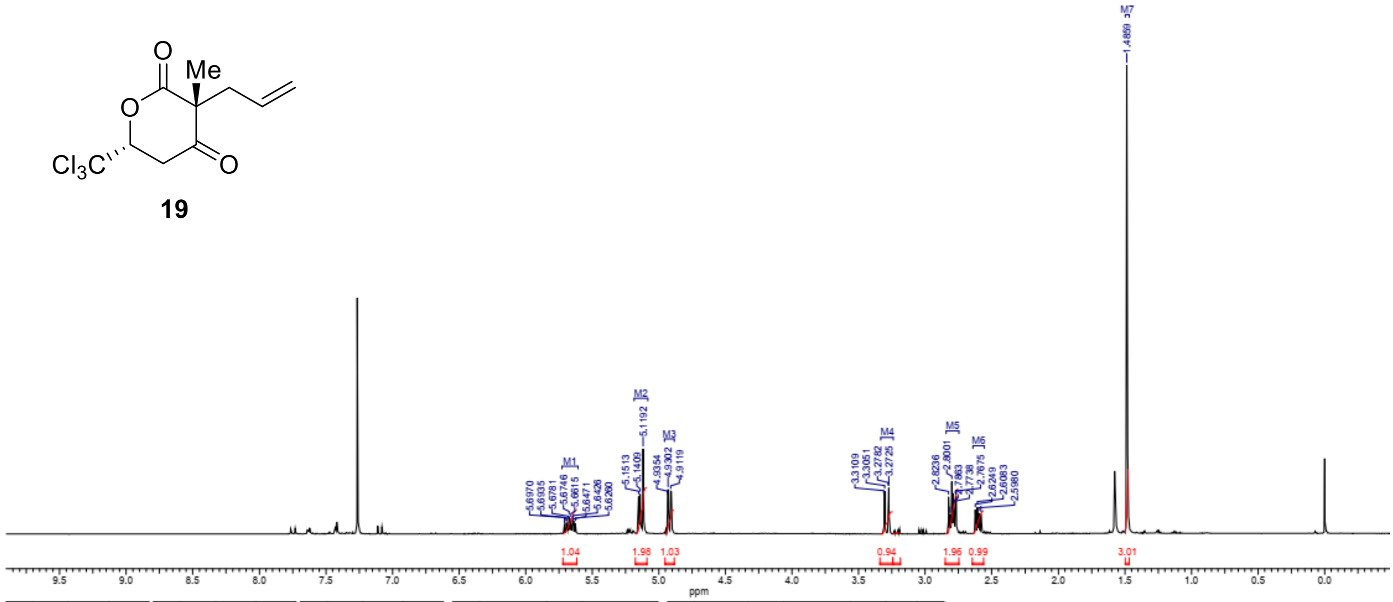


18



No.	(ppm)	(Hz)	Height
1	9.92	1247.3	0.3552
2	28.66	3604.5	0.4877
3	70.07	8812.2	0.5995
4	82.81	10414.9	0.3900
5	97.04	12204.7	0.1570
6	116.01	14590.8	0.2625
7	120.49	15153.4	0.5248
8	130.21	16377.1	0.2437
9	150.70	18953.9	0.2130
10	155.43	19548.9	0.2715
11	163.65	20581.8	0.1192

^{1}H NMR, $CDCl_3$, 500MHz

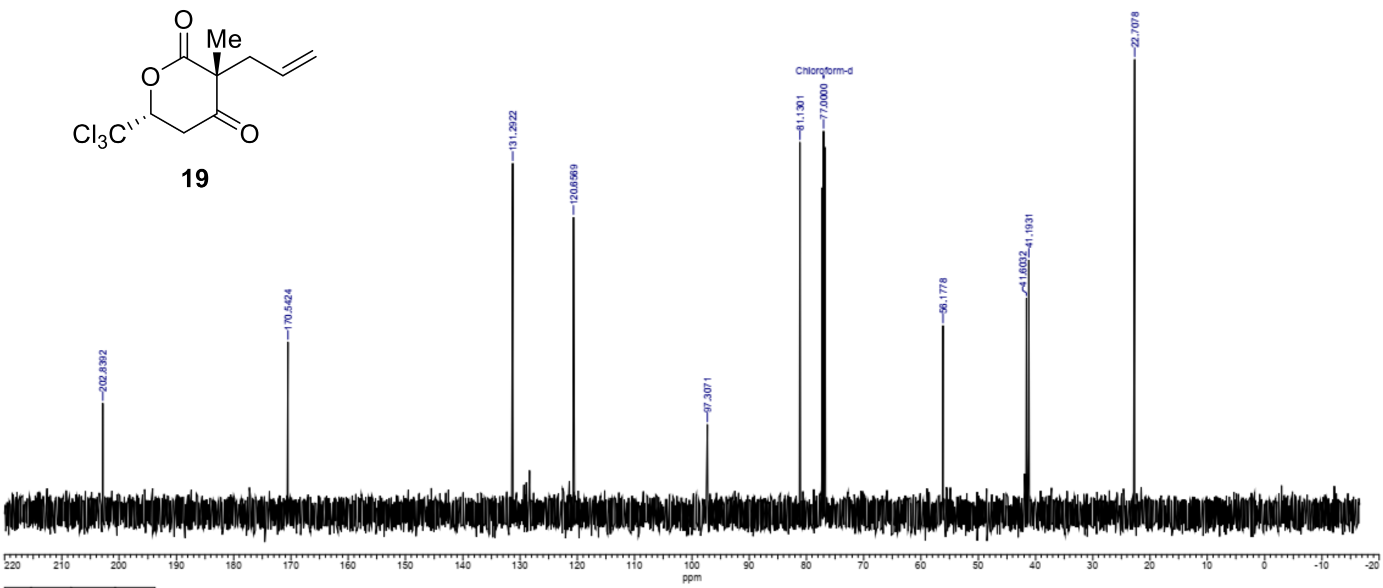


No.	(ppm)	(Hz)	Height
1	1.49	743.2	1.0000
2	2.58	1291.1	0.0425
3	2.60	1299.4	0.0445
4	2.61	1304.6	0.0574
5	2.62	1312.9	0.0521
6	2.77	1384.2	0.0806
7	2.77	1386.2	0.0420
8	2.77	1387.3	0.0588
9	2.78	1388.5	0.0372
10	2.78	1392.8	0.0476
11	2.79	1393.6	0.0597
12	2.79	1395.9	0.0870
13	2.80	1400.5	0.1118
14	2.81	1405.9	0.0321
15	2.81	1407.1	0.0405
16	2.82	1408.2	0.0297
17	2.82	1412.2	0.0793
18	3.27	1636.8	0.0980
19	3.28	1639.6	0.0980
20	3.31	1653.1	0.0916
21	3.31	1656.0	0.0808
22	4.91	2453.9	0.0903
23	4.91	2456.7	0.0913
24	4.93	2465.9	0.0947
25	4.94	2468.5	0.0798
26	5.12	2560.4	0.1815
27	5.14	2571.3	0.0850
28	5.15	2575.3	0.0529
29	5.15	2576.5	0.0796
30	5.15	2577.9	0.0609
31	5.63	2813.9	0.0229
32	5.64	2820.5	0.0278
33	5.64	2822.2	0.0281
34	5.65	2824.5	0.0201
35	5.66	2828.8	0.0289
36	5.66	2830.2	0.0279
37	5.66	2831.6	0.0317
38	5.66	2833.1	0.0185
39	5.67	2836.8	0.0197
40	5.67	2838.2	0.0365
41	5.68	2839.9	0.0266
42	5.68	2841.1	0.0235
43	5.69	2844.5	0.0132
44	5.69	2846.2	0.0175
45	5.69	2847.7	0.0232
46	5.70	2849.4	0.0231
47	5.71	2856.0	0.0181

No.	(ppm)	Value	Absolute Value
1	[1.47 .. 1.50]	3.008	1.78127e+6
2	[2.56 .. 2.65]	0.990	5.86421e+5
3	[2.75 .. 2.85]	1.957	1.15911e+6
4	[3.19 .. 3.24]	0.064	3.79489e+4
5	[3.24 .. 3.34]	0.936	5.54212e+5
6	[4.88 .. 4.95]	1.034	6.12542e+5
7	[5.09 .. 5.18]	1.982	1.17386e+6
8	[5.62 .. 5.72]	1.040	6.15648e+5

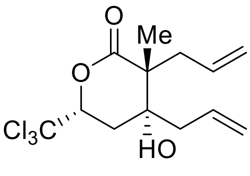
No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M7	1.48	[1.47 .. 1.50]	-	s	3
2	M6	2.60	[2.56 .. 2.64]	13.46	dd	1
3	M6	2.60	[2.56 .. 2.64]	8.31	dd	1
4	M5	2.79	[2.75 .. 2.84]	-	m	2
5	M4	3.29	[3.24 .. 3.33]	16.32	dd	1
6	M4	3.29	[3.24 .. 3.33]	2.86	dd	1
7	M3	4.92	[4.88 .. 4.96]	11.89	dd	1
8	M3	4.92	[4.88 .. 4.96]	2.72	dd	1
9	M2	5.13	[5.08 .. 5.19]	-	m	2
10	M1	5.67	[5.61 .. 5.73]	-	m	1

^{13}C NMR, $CDCl_3$, 125MHz

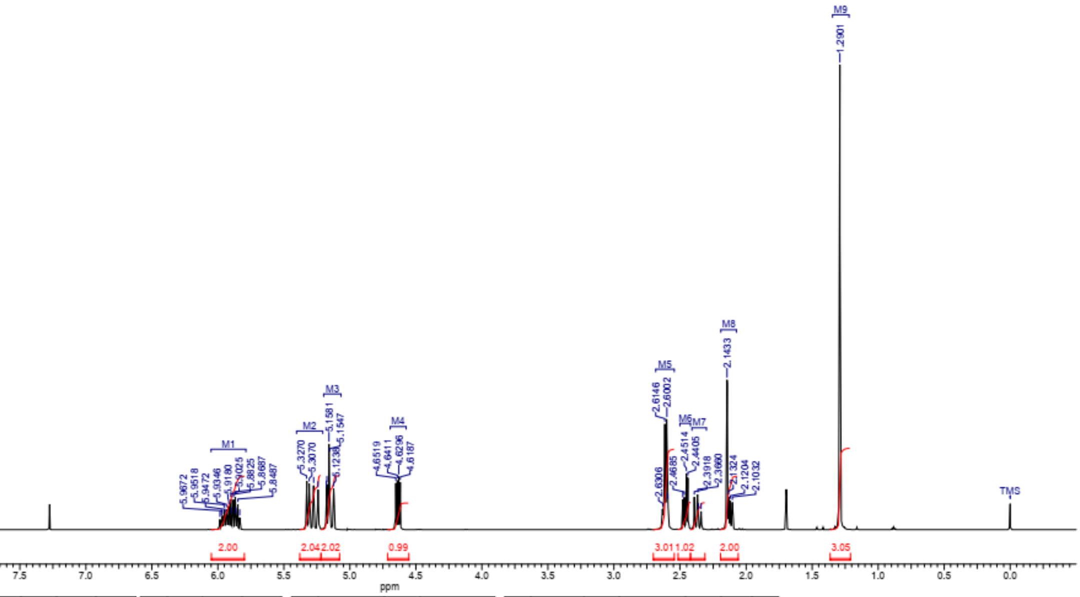


No.	(ppm)	(Hz)	Height
1	22.71	2856.0	1.0000
2	41.19	5180.9	0.5579
3	41.60	5232.4	0.4731
4	56.18	7065.5	0.4133
5	77.00	9684.3	0.8422
6	81.13	10203.7	0.8172
7	97.31	12238.3	0.1957
8	120.66	15175.0	0.6518
9	131.29	16512.6	0.7712
10	170.54	21449.1	0.3772
11	202.84	25511.1	0.2425

^{1}H NMR, $CDCl_3$, 500MHz



20

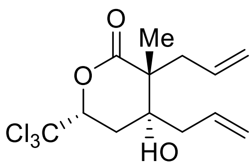


No.	(ppm)	(Hz)	Height
1	1.29	645.3	1.0000
2	2.10	1051.9	0.0586
3	2.12	1060.5	0.0613
4	2.13	1066.6	0.0710
5	2.14	1072.0	0.3222
6	2.34	1169.4	0.0353
7	2.34	1171.1	0.0391
8	2.36	1180.8	0.0388
9	2.37	1183.4	0.0752
10	2.37	1185.1	0.0704
11	2.39	1194.9	0.0624
12	2.39	1196.3	0.0700
13	2.44	1220.6	0.1115
14	2.45	1226.1	0.1212
15	2.47	1234.7	0.0695
16	2.48	1240.1	0.0644
17	2.59	1295.4	0.0501
18	2.59	1296.8	0.0608
19	2.60	1300.5	0.2377
20	2.61	1307.7	0.2274
21	2.63	1315.7	0.0448
22	2.63	1317.2	0.0441
23	4.62	2310.1	0.1018
24	4.63	2315.5	0.1143
25	4.64	2321.3	0.1057
26	4.65	2326.7	0.0997
27	5.12	2561.3	0.0760
28	5.12	2562.7	0.0881
29	5.15	2578.2	0.1626
30	5.16	2579.0	0.1608
31	5.16	2579.9	0.1846
32	5.17	2588.2	0.0977
33	5.18	2589.1	0.0974
34	5.18	2589.9	0.0873
35	5.24	2620.3	0.0779
36	5.24	2621.1	0.0861
37	5.27	2637.2	0.0862
38	5.27	2638.3	0.0953
39	5.31	2654.4	0.0985
40	5.33	2664.4	0.1046
41	5.83	2917.8	0.0259
42	5.85	2925.3	0.0541
43	5.85	2927.9	0.0294
44	5.86	2932.7	0.0295
45	5.87	2935.3	0.0700
46	5.88	2942.2	0.0645
47	5.89	2944.8	0.0298
48	5.90	2949.6	0.0296
49	5.90	2952.2	0.0649
50	5.91	2957.6	0.0314
51	5.92	2959.9	0.0508
52	5.93	2966.0	0.0307
53	5.93	2968.2	0.0428
54	5.94	2970.0	0.0343
55	5.95	2974.5	0.0320
56	5.95	2976.8	0.0424
57	5.96	2978.6	0.0297
58	5.96	2983.1	0.0279
59	5.97	2984.6	0.0291
60	5.97	2987.1	0.0257
61	5.98	2993.2	0.0218

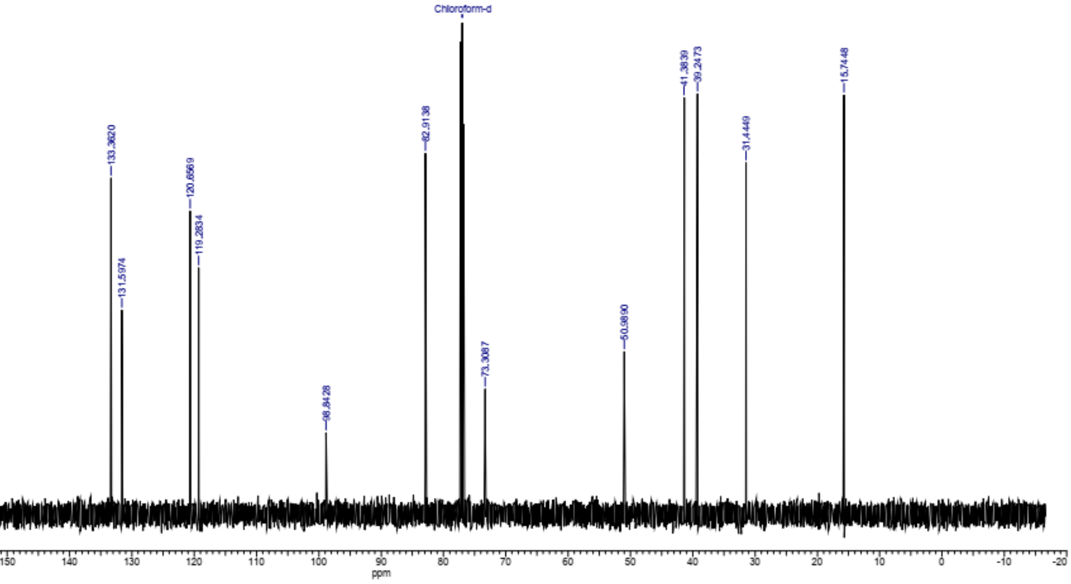
No.	(ppm)	Value	Absolute Value
1	[1.21 .. 1.36]	3.052	1.61069e+6
2	[2.06 .. 2.19]	2.000	1.05537e+6
3	[2.31 .. 2.42]	1.011	5.33355e+5
4	[2.42 .. 2.51]	1.023	5.40056e+5
5	[2.54 .. 2.70]	3.015	1.59096e+6
6	[4.55 .. 4.71]	0.988	5.21389e+5
7	[5.08 .. 5.22]	2.015	1.06351e+6
8	[5.22 .. 5.38]	2.036	1.07434e+6
9	[5.80 .. 6.05]	2.000	1.05549e+6

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	1.28	[1.22 .. 1.35]	-	s	3
2	M8	2.13	[2.07 .. 2.19]	-	m	2
3	M7	2.36	[2.30 .. 2.41]	-	m	1
4	M6	2.46	[2.42 .. 2.50]	-	m	1
5	M5	2.61	[2.54 .. 2.68]	-	m	3
6	M4	4.63	[4.57 .. 4.69]	11.17	dd	1
7	M4	4.63	[4.57 .. 4.69]	5.44	dd	1
8	M3	5.13	[5.07 .. 5.20]	-	m	2
9	M2	5.30	[5.21 .. 5.40]	-	m	2
10	M1	5.92	[5.79 .. 6.05]	-	m	2

^{13}C NMR, $CDCl_3$, 125MHz

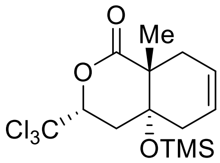


20

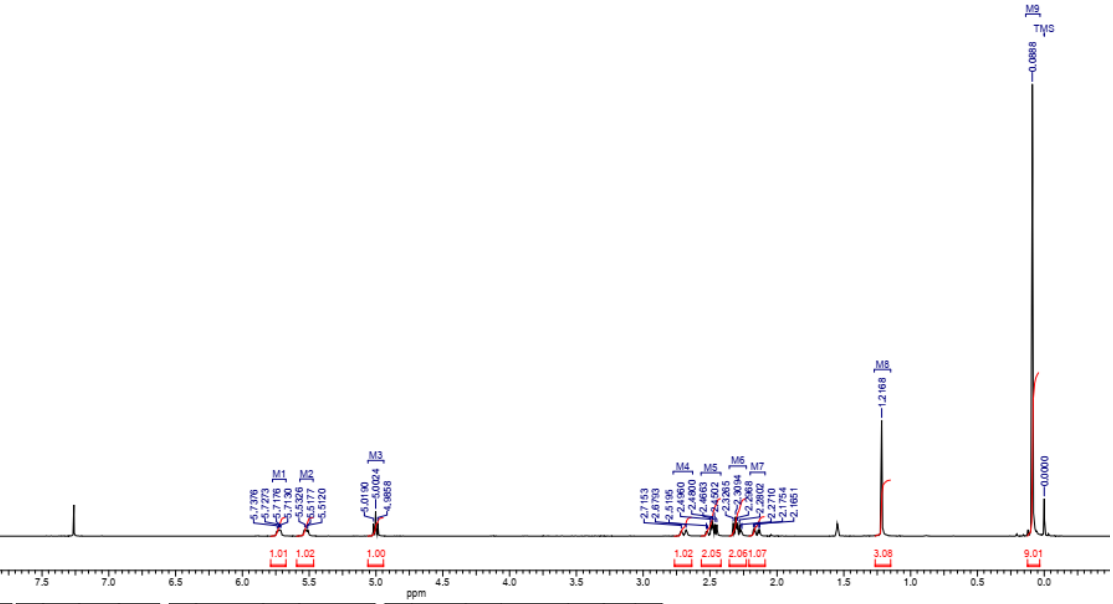


No.	(ppm)	(Hz)	Height
1	15.74	1980.2	0.8531
2	31.44	3954.8	0.7150
3	39.25	4936.1	0.8557
4	41.38	5204.8	0.8475
5	50.99	6412.9	0.3309
6	73.31	9220.0	0.2547
7	82.91	10428.1	0.7347
8	98.84	12431.5	0.1651
9	119.28	15002.3	0.5016
10	120.66	15175.0	0.6171
11	131.60	16551.0	0.4149
12	133.36	16772.9	0.6842
13	172.43	21686.6	0.1338

^{1}H NMR, $CDCl_3$, 500MHz



21

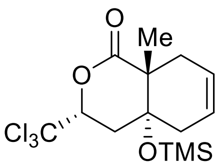


No.	(ppm)	(Hz)	Height
1	-0.00	-0.0	0.0830
2	0.09	44.4	1.0000
3	1.22	608.6	0.2567
4	2.13	1064.8	0.0134
5	2.14	1070.3	0.0131
6	2.17	1082.9	0.0153
7	2.18	1088.0	0.0145
8	2.26	1131.6	0.0104
9	2.27	1133.9	0.0134
10	2.27	1135.9	0.0137
11	2.28	1140.4	0.0245
12	2.30	1148.7	0.0288
13	2.30	1152.5	0.0208
14	2.31	1155.0	0.0426
15	2.33	1163.6	0.0283
16	2.45	1225.5	0.0254
17	2.47	1233.5	0.0265
18	2.48	1240.4	0.0309
19	2.50	1248.4	0.0254
20	2.52	1260.2	0.0109
21	2.52	1262.2	0.0104
22	2.53	1264.7	0.0101
23	2.68	1340.1	0.0139
24	2.72	1358.1	0.0119
25	4.99	2493.7	0.0287
26	5.00	2502.0	0.0550
27	5.02	2510.3	0.0269
28	5.51	2756.9	0.0126
29	5.52	2759.7	0.0119
30	5.52	2762.3	0.0127
31	5.53	2765.5	0.0140
32	5.53	2767.2	0.0144
33	5.71	2857.4	0.0130
34	5.72	2859.7	0.0137
35	5.72	2860.8	0.0133
36	5.72	2862.9	0.0137
37	5.73	2864.6	0.0135
38	5.73	2866.0	0.0130
39	5.74	2869.7	0.0109
40	5.74	2872.9	0.0097

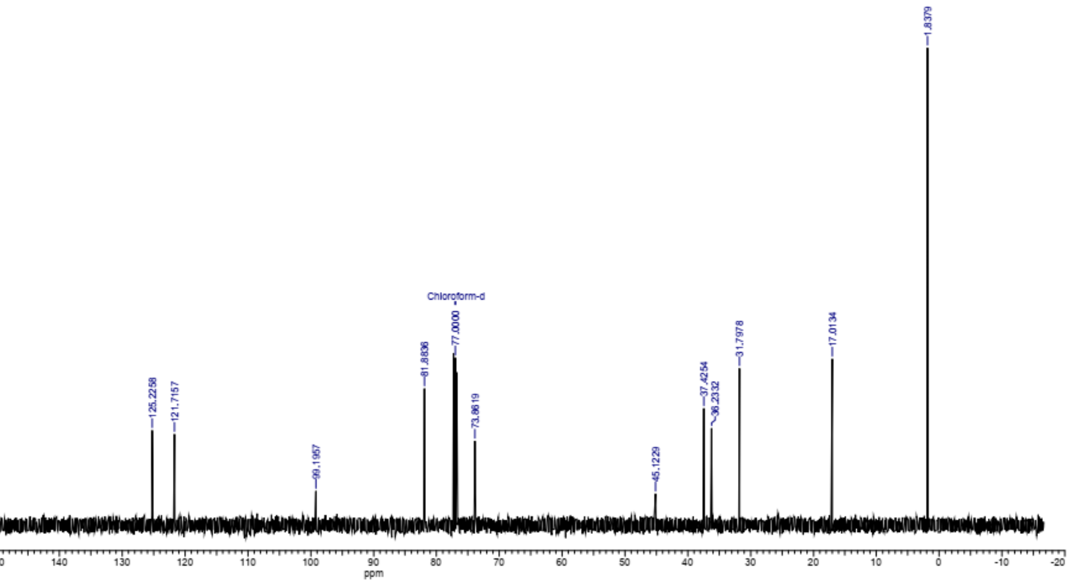
No.	(ppm)	Value	Absolute Value
1	[0.03 .. 0.12]	9.006	3.81283e+6
2	[1.15 .. 1.27]	3.077	1.30261e+6
3	[2.09 .. 2.21]	1.070	4.53053e+5
4	[2.23 .. 2.36]	2.057	8.70995e+5
5	[2.42 .. 2.57]	2.054	8.69693e+5
6	[2.64 .. 2.77]	1.024	4.33602e+5
7	[4.94 .. 5.06]	1.000	4.23363e+5
8	[5.47 .. 5.59]	1.018	4.30955e+5
9	[5.67 .. 5.79]	1.014	4.29369e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	0.08	[0.03 .. 0.14]	-	s	9
2	M8	1.21	[1.15 .. 1.27]	-	s	3
3	M7	2.14	[2.09 .. 2.20]	17.90	dd	1
4	M7	2.14	[2.09 .. 2.20]	5.30	dd	1
5	M6	2.29	[2.23 .. 2.36]	-	m	2
6	M5	2.49	[2.42 .. 2.57]	-	m	2
7	M4	2.70	[2.63 .. 2.78]	18.04	d	1
8	M3	5.00	[4.94 .. 5.06]	8.31	t	1
9	M3	5.00	[4.94 .. 5.06]	8.31	t	1
10	M2	5.52	[5.47 .. 5.57]	-	m	1
11	M1	5.73	[5.67 .. 5.78]	-	m	1

^{13}C NMR, $CDCl_3$, 125MHz

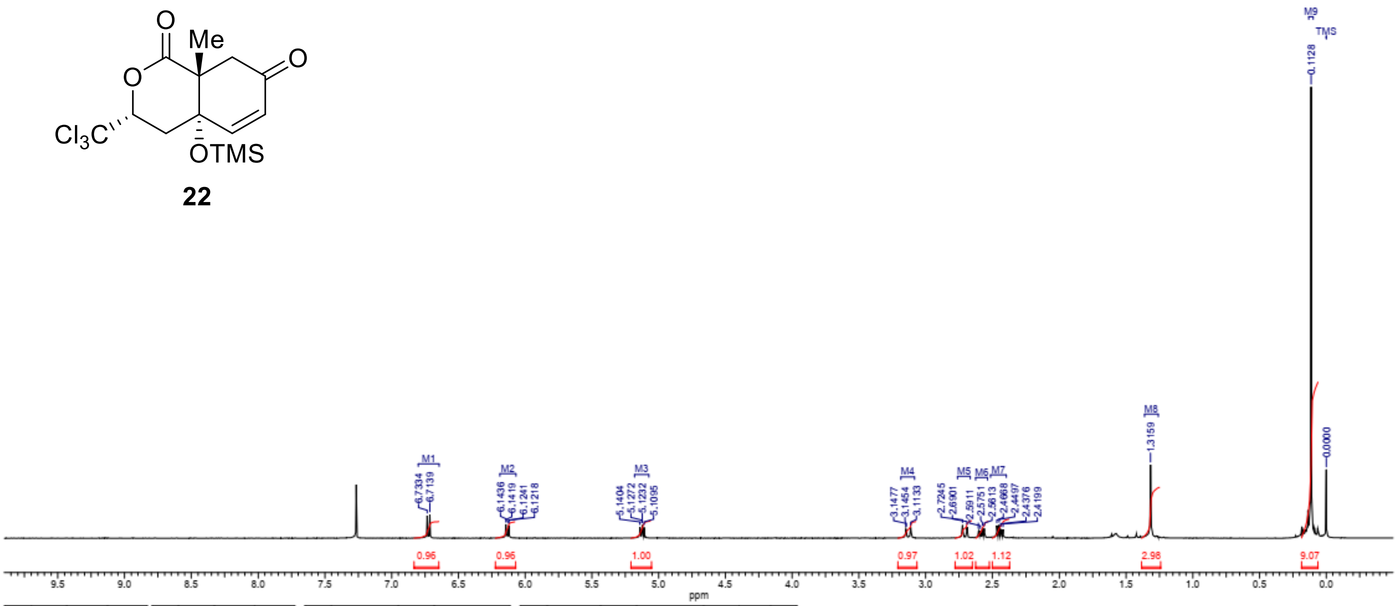


21



No.	(ppm)	(Hz)	Height
1	1.84	231.2	1.0000
2	17.01	2139.8	0.3484
3	31.80	3999.2	0.3290
4	36.23	4557.0	0.2034
5	37.43	4707.0	0.2449
6	45.12	5675.1	0.0656
7	73.86	9289.6	0.1767
8	77.00	9684.3	0.3509
9	81.88	10298.5	0.2865
10	99.20	12475.8	0.0713
11	121.72	15308.2	0.1907
12	125.23	15749.6	0.1989
13	173.58	21830.6	0.1325

^{1}H NMR, $CDCl_3$, 500MHz



22

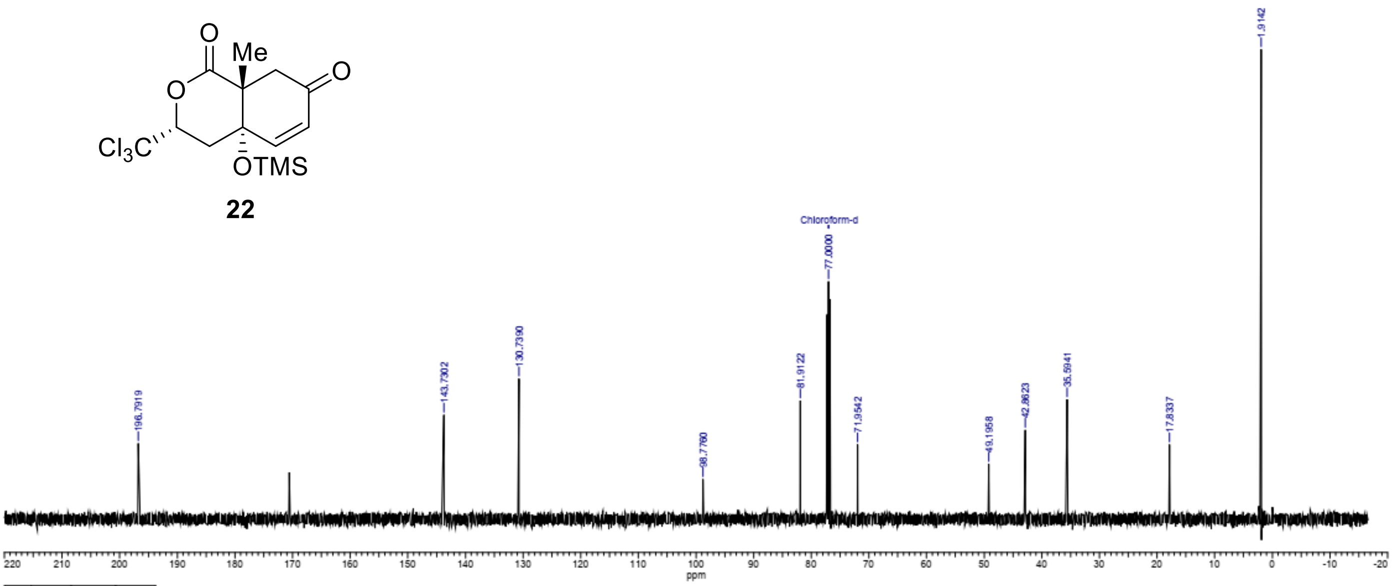
No.	(ppm)	(Hz)	Height
1	-0.00	-0.0	0.1521
2	0.11	56.4	1.0000
3	1.31	657.0	0.1611
4	1.32	658.1	0.1630
5	2.42	1210.3	0.0192
6	2.44	1219.2	0.0205
7	2.45	1225.2	0.0296
8	2.47	1233.8	0.0284
9	2.56	1281.1	0.0215
10	2.58	1287.9	0.0222
11	2.59	1296.0	0.0157
12	2.60	1302.8	0.0157
13	2.69	1345.5	0.0245
14	2.72	1362.7	0.0288

No.	(ppm)	(Hz)	Height
15	3.11	1556.0	0.0233
16	3.11	1557.2	0.0235
17	3.15	1573.2	0.0204
18	3.15	1574.3	0.0206
19	5.11	2555.5	0.0242
20	5.12	2562.4	0.0285
21	5.13	2564.4	0.0279
22	5.14	2571.0	0.0240
23	6.12	3061.9	0.0278
24	6.12	3063.0	0.0276
25	6.14	3071.9	0.0296
26	6.14	3072.8	0.0308
27	6.71	3358.0	0.0522
28	6.73	3367.8	0.0499

No.	(ppm)	Value	Absolute Value
1	[0.06 .. 0.19]	9.069	3.09024e+6
2	[1.24 .. 1.38]	2.981	1.01593e+6
3	[2.37 .. 2.50]	1.115	3.80021e+5
4	[2.52 .. 2.63]	1.005	3.42385e+5
5	[2.65 .. 2.78]	1.024	3.48991e+5
6	[3.07 .. 3.21]	0.970	3.30700e+5
7	[5.05 .. 5.21]	1.000	3.40752e+5
8	[6.07 .. 6.22]	0.957	3.26107e+5
9	[6.65 .. 6.83]	0.957	3.26055e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	0.11	[0.10 .. 0.13]	-	s	9
2	M8	1.31	[1.26 .. 1.36]	1.15	d	3
3	M7	2.45	[2.40 .. 2.51]	14.75	dd	1
4	M7	2.45	[2.40 .. 2.51]	8.74	dd	1
5	M6	2.58	[2.54 .. 2.63]	14.89	dd	1
6	M6	2.58	[2.54 .. 2.63]	6.87	dd	1
7	M5	2.71	[2.67 .. 2.76]	17.18	d	1
8	M4	3.14	[3.08 .. 3.19]	17.18	dd	1
9	M4	3.14	[3.08 .. 3.19]	1.15	dd	1
10	M3	5.13	[5.07 .. 5.18]	8.74	dd	1
11	M3	5.13	[5.07 .. 5.18]	6.73	dd	1
12	M2	6.13	[6.07 .. 6.19]	9.88	dd	1
13	M2	6.13	[6.07 .. 6.19]	1.00	dd	1
14	M1	6.72	[6.64 .. 6.80]	9.74	d	1

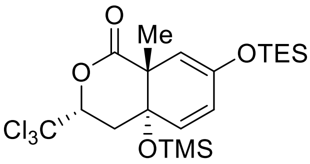
^{13}C NMR, $CDCl_3$, 125MHz



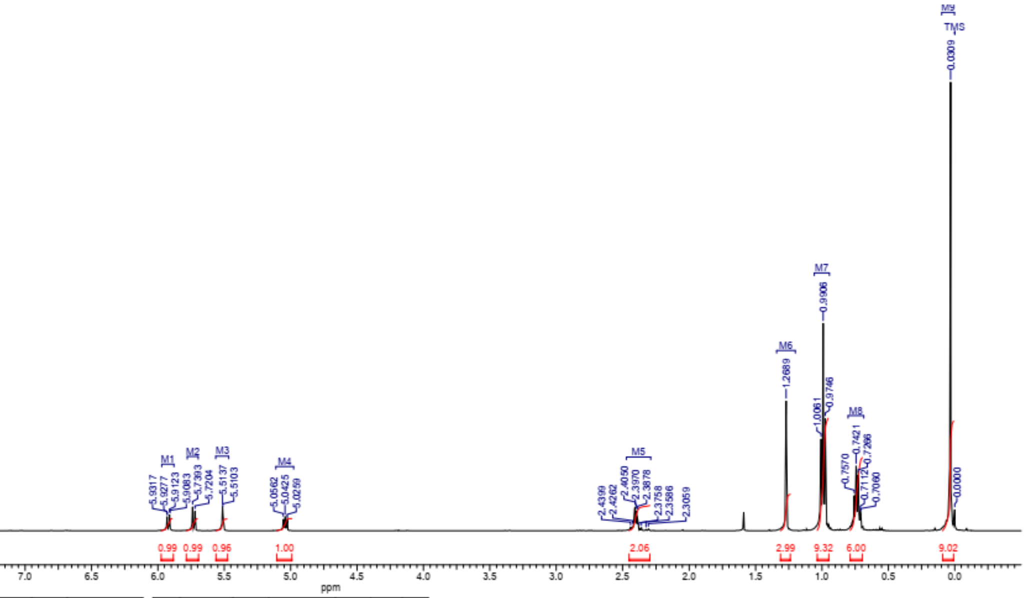
22

No.	(ppm)	(Hz)	Height
1	1.91	240.8	1.0000
2	17.83	2242.9	0.1590
3	35.59	4476.7	0.2551
4	42.86	5390.8	0.1895
5	49.20	6187.3	0.1173
6	71.95	9049.7	0.1589
7	77.00	9684.3	0.5064
8	81.91	10302.1	0.2525
9	98.78	12423.1	0.0850
10	130.74	16443.0	0.2992
11	143.73	18076.9	0.2218
12	196.79	24750.5	0.1613

^{1}H NMR, $CDCl_3$, 500MHz



SI-1-TES



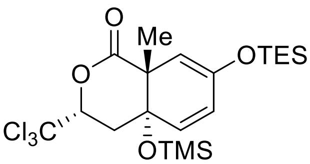
No.	(ppm)	(Hz)	Height
1	0.00	0.0	0.0477
2	0.03	15.5	1.0000
3	0.71	353.1	0.0372
4	0.71	355.7	0.0428
5	0.72	361.4	0.1074
6	0.73	363.4	0.1236
7	0.74	369.7	0.1275
8	0.74	371.2	0.1440
9	0.76	378.6	0.0784
10	0.97	487.5	0.2508
11	0.99	495.5	0.4622
12	1.01	503.2	0.2044
13	1.27	634.7	0.2892
14	2.31	1153.3	0.0040
15	2.33	1163.1	0.0027
16	2.36	1179.7	0.0062
17	2.38	1188.3	0.0061
18	2.39	1194.3	0.0428

No.	(ppm)	(Hz)	Height
19	2.40	1198.9	0.0440
20	2.40	1202.9	0.0521
21	2.41	1205.5	0.0507
22	2.43	1213.5	0.0108
23	2.44	1220.3	0.0070
24	5.03	2513.7	0.0241
25	5.04	2520.3	0.0307
26	5.04	2522.0	0.0310
27	5.06	2528.9	0.0259
28	5.51	2756.0	0.0478
29	5.51	2757.7	0.0537
30	5.72	2861.1	0.0433
31	5.74	2870.6	0.0535
32	5.91	2955.1	0.0334
33	5.91	2957.1	0.0363
34	5.93	2964.8	0.0290
35	5.93	2966.8	0.0318

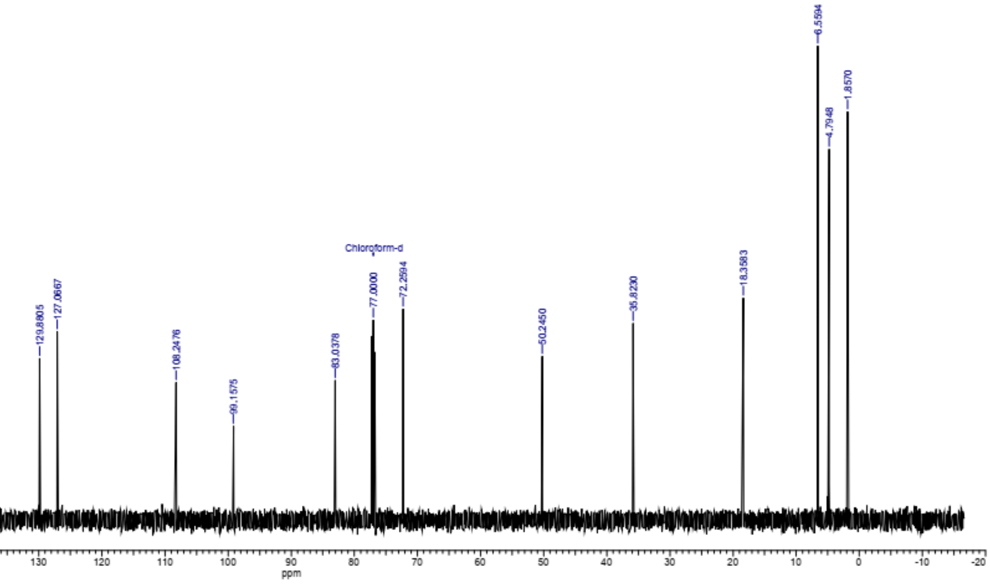
No.	(ppm)	Value	Absolute Value
1	[0.01 .. 0.09]	9.015	2.55582e+6
2	[0.70 .. 0.79]	5.998	1.70045e+6
3	[0.95 .. 1.03]	9.317	2.64130e+6
4	[1.24 .. 1.31]	2.990	8.47718e+5
5	[2.30 .. 2.45]	2.060	5.84153e+5
6	[4.99 .. 5.10]	1.000	2.83508e+5
7	[5.48 .. 5.56]	0.964	2.73407e+5
8	[5.69 .. 5.79]	0.994	2.81845e+5
9	[5.88 .. 5.98]	0.990	2.80712e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	0.05	[0.00 .. 0.10]	-	s	9
2	M8	0.74	[0.69 .. 0.80]	-	m	6
3	M7	1.00	[0.94 .. 1.06]	7.88	t	9
4	M7	1.00	[0.94 .. 1.06]	7.88	t	9
5	M6	1.27	[1.20 .. 1.34]	-	s	3
6	M5	2.38	[2.29 .. 2.47]	-	m	2
7	M4	5.05	[4.98 .. 5.11]	8.45	dd	1
8	M4	5.05	[4.98 .. 5.11]	6.73	dd	1
9	M3	5.52	[5.47 .. 5.57]	1.72	d	1
10	M2	5.74	[5.70 .. 5.78]	9.45	d	1
11	M1	5.92	[5.88 .. 5.97]	9.74	dd	1
12	M1	5.92	[5.88 .. 5.97]	2.00	dd	1

^{13}C NMR, $CDCl_3$, 125MHz

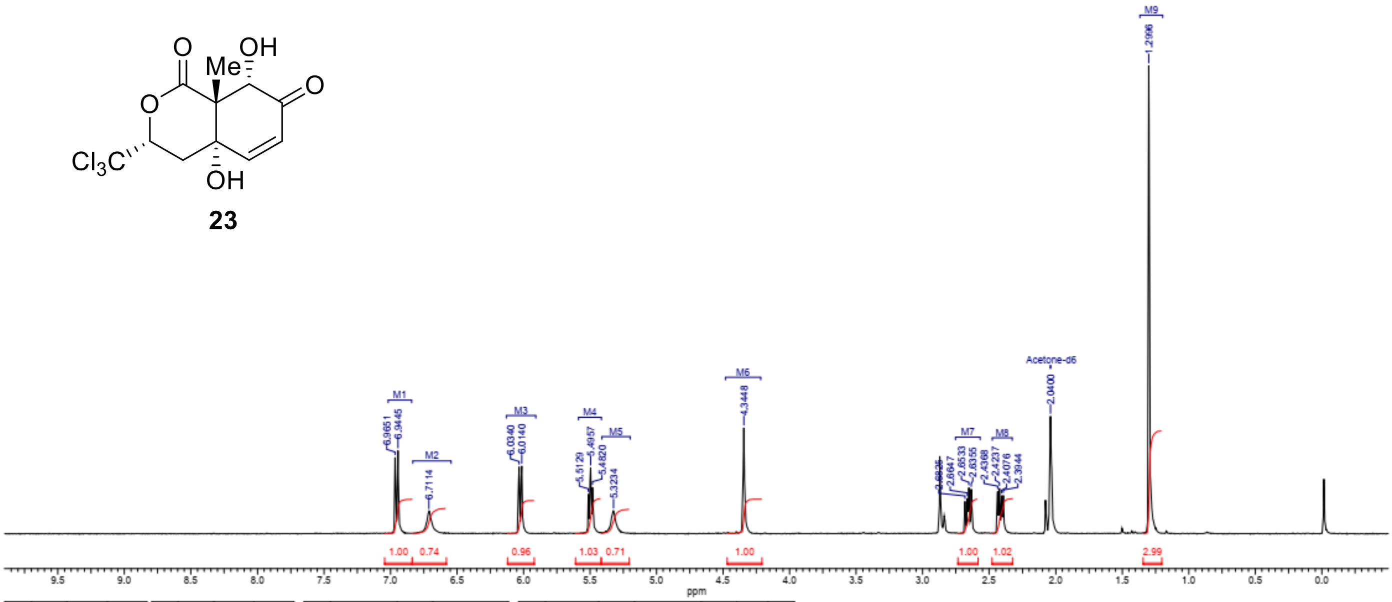


SI-1-TES



No.	(ppm)	(Hz)	Height
1	1.86	233.6	0.8616
2	4.79	603.0	0.7825
3	6.56	825.0	1.0000
4	18.36	2308.9	0.4684
5	35.82	4505.5	0.4149
6	50.24	6319.3	0.3453
7	72.26	9088.1	0.4451
8	77.00	9684.3	0.4216
9	83.04	10443.7	0.2943
10	99.16	12471.0	0.1977
11	108.25	13614.3	0.2897
12	127.07	15981.2	0.3973
13	129.88	16335.1	0.3407
14	146.47	18421.2	0.3387
15	170.92	21497.1	0.1217

^{1}H NMR, Acetone-*d6*, 500MHz



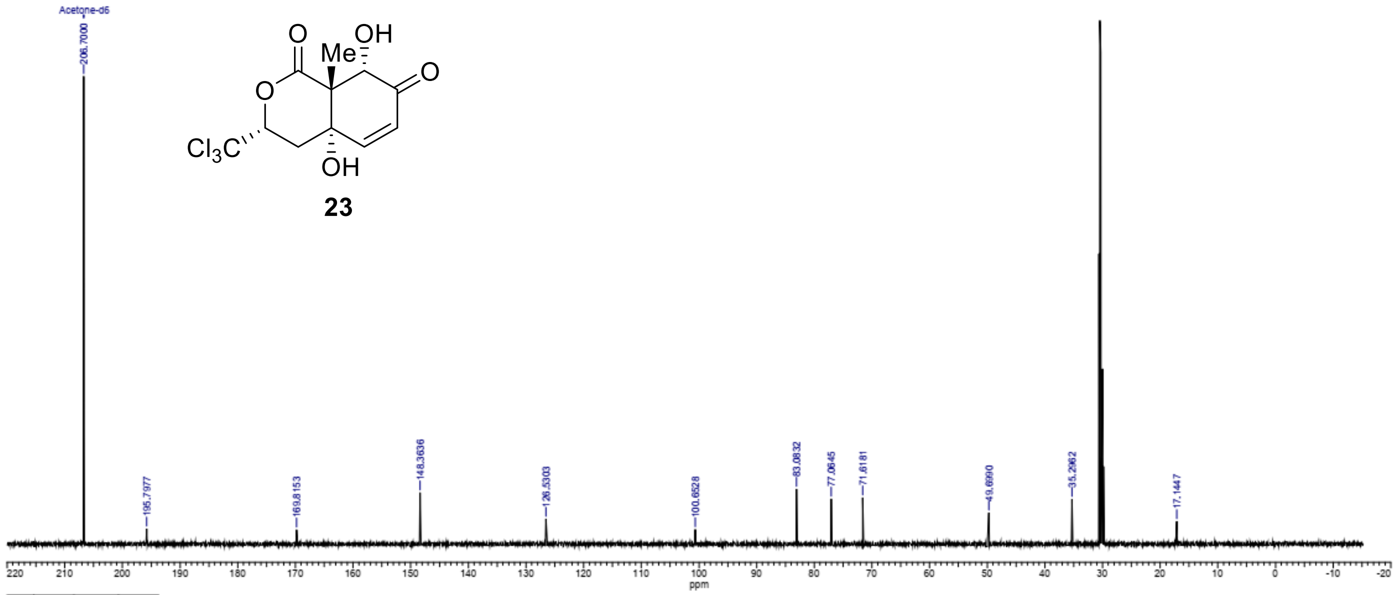
No.	(ppm)	(Hz)	Height
1	1.30	650.0	1.0000
2	2.04	1020.3	0.2508
3	2.39	1197.6	0.0817
4	2.41	1204.2	0.0809
5	2.42	1212.2	0.0970
6	2.44	1218.8	0.0901
7	2.64	1318.2	0.0953
8	2.65	1327.1	0.0984
9	2.66	1332.8	0.0754
10	2.68	1341.7	0.0693
11	4.34	2173.1	0.2264

No.	(ppm)	(Hz)	Height
12	5.32	2662.5	0.0490
13	5.48	2741.9	0.0993
14	5.50	2748.7	0.1405
15	5.50	2750.5	0.1144
16	5.51	2757.3	0.0844
17	6.01	3007.9	0.1445
18	6.03	3018.0	0.1429
19	6.71	3356.8	0.0478
20	6.94	3473.3	0.1778
21	6.97	3483.6	0.1630

No.	(ppm)	Value	Absolute Value
1	[1.20 .. 1.34]	2.990	2.18196e+6
2	[2.33 .. 2.48]	1.018	7.42788e+5
3	[2.58 .. 2.74]	1.004	7.32960e+5
4	[4.21 .. 4.47]	1.003	7.31842e+5
5	[5.21 .. 5.42]	0.707	5.15545e+5
6	[5.42 .. 5.61]	1.027	7.49485e+5
7	[5.92 .. 6.12]	0.963	7.02911e+5
8	[6.58 .. 6.84]	0.736	5.36896e+5
9	[6.84 .. 7.04]	0.999	7.28999e+5

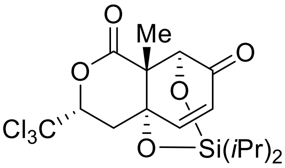
No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	1.28	[1.20 .. 1.37]	-	s	3
2	M8	2.40	[2.33 .. 2.48]	14.61	dd	1
3	M8	2.40	[2.33 .. 2.48]	6.59	dd	1
4	M7	2.66	[2.57 .. 2.75]	14.61	dd	1
5	M7	2.66	[2.57 .. 2.75]	8.88	dd	1
6	M6	4.35	[4.22 .. 4.48]	-	s	1
7	M5	5.31	[5.20 .. 5.41]	-	s	1
8	M4	5.50	[5.42 .. 5.59]	-	m	1
9	M3	6.02	[5.91 .. 6.13]	10.02	d	1
10	M2	6.69	[6.54 .. 6.83]	-	s	1
11	M1	6.93	[6.84 .. 7.02]	10.31	d	1

^{13}C NMR, Acetone-*d6*, 125MHz

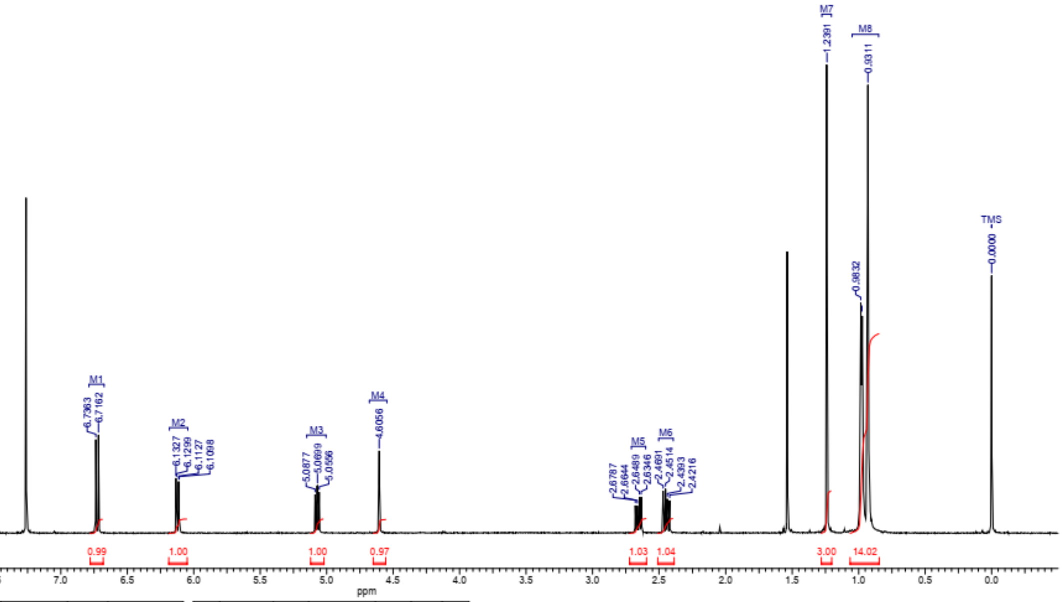


No.	(ppm)	(Hz)	Height
1	17.14	2156.3	0.0432
2	35.30	4439.2	0.0856
3	49.70	6250.6	0.0598
4	71.62	9007.4	0.0884
5	77.06	9692.4	0.0862
6	83.08	10449.4	0.1051
7	100.65	12659.1	0.0280
8	126.53	15913.7	0.0475
9	148.36	18659.7	0.0980
10	169.82	21357.7	0.0272
11	195.80	24625.5	0.0290
12	206.70	25996.7	0.8935

[1]H NMR, $CDCl_3$, 500MHz



24



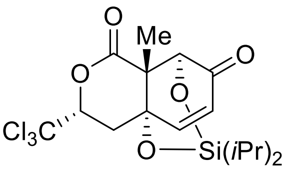
No.	(ppm)	(Hz)	Height
1	-0.00	-0.0	0.5502
2	0.93	465.7	0.9570
3	0.97	487.2	0.4683
4	0.98	491.7	0.4922
5	1.24	619.8	1.0000
6	2.42	1211.2	0.0703
7	2.44	1220.1	0.0732
8	2.45	1226.1	0.0950
9	2.47	1235.0	0.0902
10	2.63	1317.7	0.0778
11	2.65	1324.9	0.0777
12	2.66	1332.6	0.0588

No.	(ppm)	(Hz)	Height
13	2.68	1339.8	0.0592
14	4.61	2303.5	0.1762
15	5.06	2528.6	0.0876
16	5.07	2535.8	0.1016
17	5.07	2537.2	0.1007
18	5.09	2544.7	0.0827
19	6.11	3055.9	0.1095
20	6.11	3057.3	0.1105
21	6.13	3065.9	0.1159
22	6.13	3067.3	0.1179
23	6.72	3359.2	0.2106
24	6.74	3369.2	0.2003

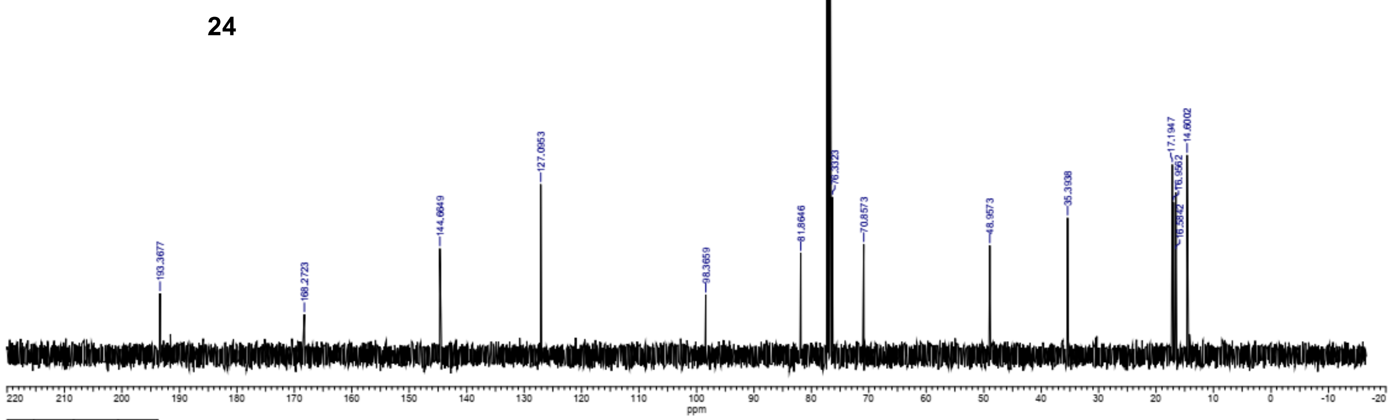
No.	(ppm)	Value	Absolute Value
1	[0.85 .. 1.06]	14.018	3.79373e+6
2	[1.20 .. 1.28]	3.004	8.13005e+5
3	[2.39 .. 2.51]	1.037	2.80705e+5
4	[2.59 .. 2.72]	1.034	2.79811e+5
5	[4.56 .. 4.65]	0.972	2.62946e+5
6	[5.02 .. 5.12]	1.000	2.70668e+5
7	[6.05 .. 6.19]	1.000	2.70638e+5
8	[6.68 .. 6.78]	0.989	2.67532e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M8	0.95	[0.85 .. 1.05]	-	m	14
2	M7	1.24	[1.20 .. 1.28]	-	s	3
3	M6	2.45	[2.39 .. 2.50]	14.89	dd	1
4	M6	2.45	[2.39 .. 2.50]	8.88	dd	1
5	M5	2.66	[2.60 .. 2.71]	14.89	dd	1
6	M5	2.66	[2.60 .. 2.71]	7.16	dd	1
7	M4	4.61	[4.55 .. 4.68]	-	s	1
8	M3	5.08	[5.00 .. 5.15]	8.74	dd	1
9	M3	5.08	[5.00 .. 5.15]	7.30	dd	1
10	M2	6.11	[6.04 .. 6.18]	10.02	dd	1
11	M2	6.11	[6.04 .. 6.18]	1.43	dd	1
12	M1	6.73	[6.67 .. 6.79]	10.02	d	1

[13]C NMR, $CDCl_3$, 125MHz

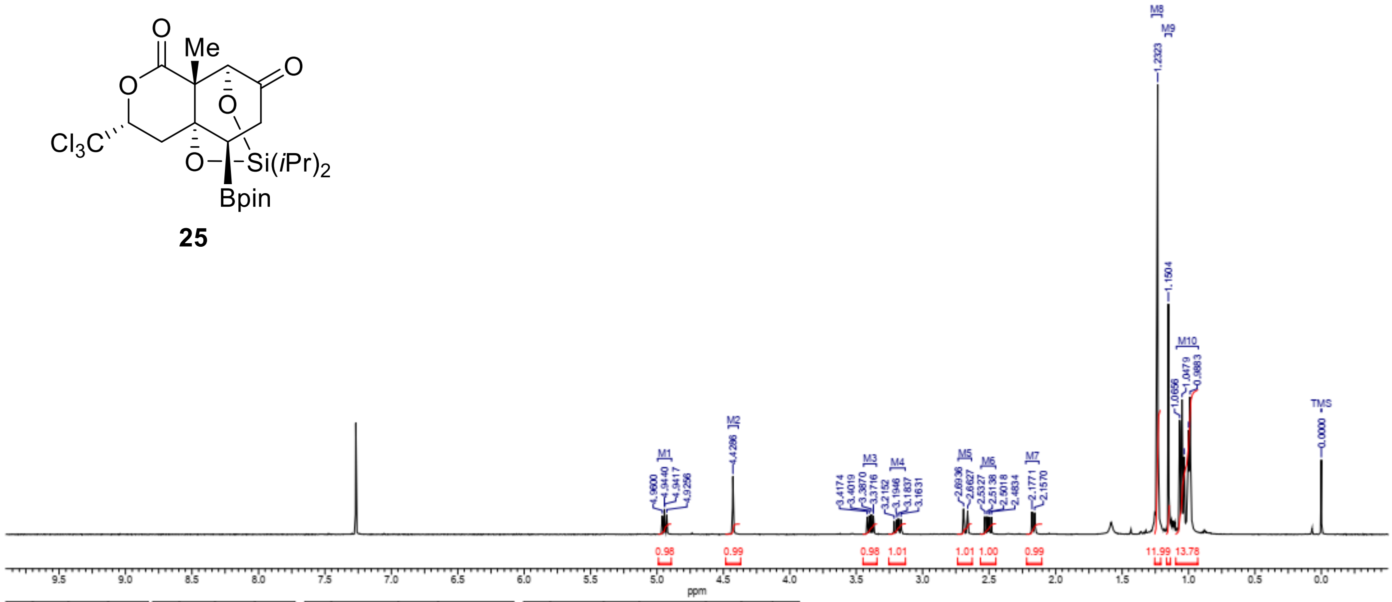


24



No.	(ppm)	(Hz)	Height
1	14.50	1824.3	0.3918
2	14.60	1836.3	0.4006
3	16.50	2075.0	0.2421
4	16.55	2081.0	0.3258
5	16.58	2085.8	0.2075
6	16.96	2132.6	0.3060
7	17.19	2162.6	0.3820
8	35.39	4451.5	0.2752
9	48.96	6157.4	0.2192
10	70.86	8911.7	0.2218
11	76.33	9600.3	0.3166
12	77.00	9684.3	0.8567
13	81.86	10296.1	0.2042
14	98.37	12371.5	0.1206
15	127.10	15984.8	0.3422
16	144.66	18194.5	0.2137
17	168.27	21163.6	0.0810
18	193.37	24319.9	0.1234

^{1}H NMR, $CDCl_3$, 500MHz



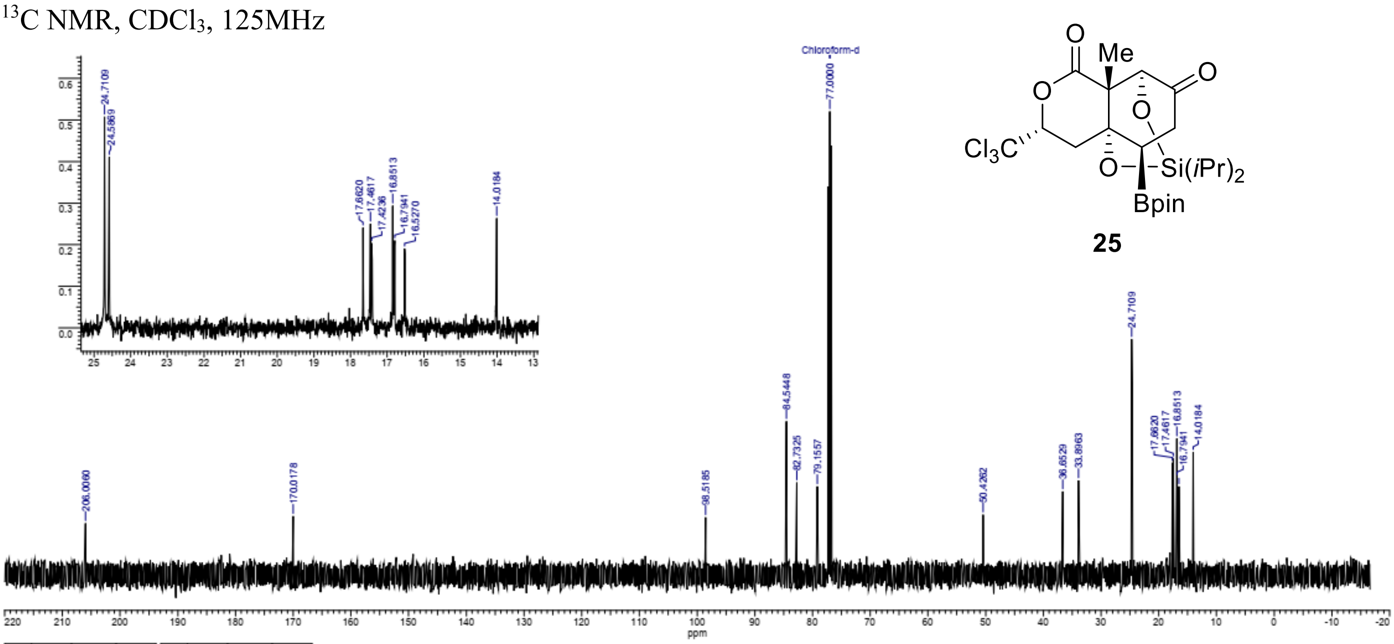
25

No.	(ppm)	(Hz)	Height
1	0.00	0.0	0.1660
2	0.99	494.3	0.3058
3	1.00	500.3	0.2317
4	1.03	517.0	0.1724
5	1.05	524.1	0.2997
6	1.07	533.0	0.2533
7	1.15	575.4	0.5124
8	1.23	616.3	1.0000
9	2.16	1078.9	0.0485
10	2.18	1088.9	0.0505
11	2.48	1242.1	0.0388
12	2.50	1251.3	0.0388
13	2.51	1257.3	0.0401
14	2.53	1266.7	0.0400
15	2.66	1331.8	0.0516
16	2.69	1347.2	0.0571
17	3.16	1582.1	0.0341
18	3.18	1592.4	0.0354
19	3.19	1597.8	0.0323
20	3.22	1608.1	0.0292
21	3.37	1686.3	0.0404
22	3.39	1694.1	0.0442
23	3.40	1701.5	0.0409
24	3.42	1709.2	0.0389
25	4.43	2215.0	0.1290
26	4.93	2463.6	0.0431
27	4.94	2471.6	0.0554
28	4.94	2472.8	0.0562
29	4.96	2480.8	0.0411

No.	(ppm)	Value	Absolute Value
1	[0.93 .. 1.09]	13.784	3.18991e+6
2	[1.14 .. 1.17]	3.074	7.11444e+5
3	[1.21 .. 1.25]	11.994	2.77567e+6
4	[2.10 .. 2.22]	0.987	2.28462e+5
5	[2.45 .. 2.57]	1.000	2.31434e+5
6	[2.63 .. 2.74]	1.005	2.32628e+5
7	[3.13 .. 3.25]	1.005	2.32591e+5
8	[3.35 .. 3.45]	0.976	2.25908e+5
9	[4.37 .. 4.48]	0.988	2.28563e+5
10	[4.89 .. 4.99]	0.979	2.26567e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M10	1.01	[0.93 .. 1.09]	-	m	14
2	M9	1.15	[1.13 .. 1.18]	-	s	3
3	M8	1.24	[1.20 .. 1.28]	-	s	12
4	M7	2.17	[2.13 .. 2.22]	10.02	d	1
5	M6	2.51	[2.45 .. 2.56]	15.32	dd	1
6	M6	2.51	[2.45 .. 2.56]	9.31	dd	1
7	M5	2.68	[2.63 .. 2.73]	15.47	d	1
8	M4	3.19	[3.13 .. 3.25]	15.75	dd	1
9	M4	3.19	[3.13 .. 3.25]	10.31	dd	1
10	M3	3.40	[3.35 .. 3.45]	15.18	dd	1
11	M3	3.40	[3.35 .. 3.45]	7.73	dd	1
12	M2	4.43	[4.39 .. 4.46]	-	s	1
13	M1	4.94	[4.89 .. 5.00]	9.16	dd	1
14	M1	4.94	[4.89 .. 5.00]	8.02	dd	1

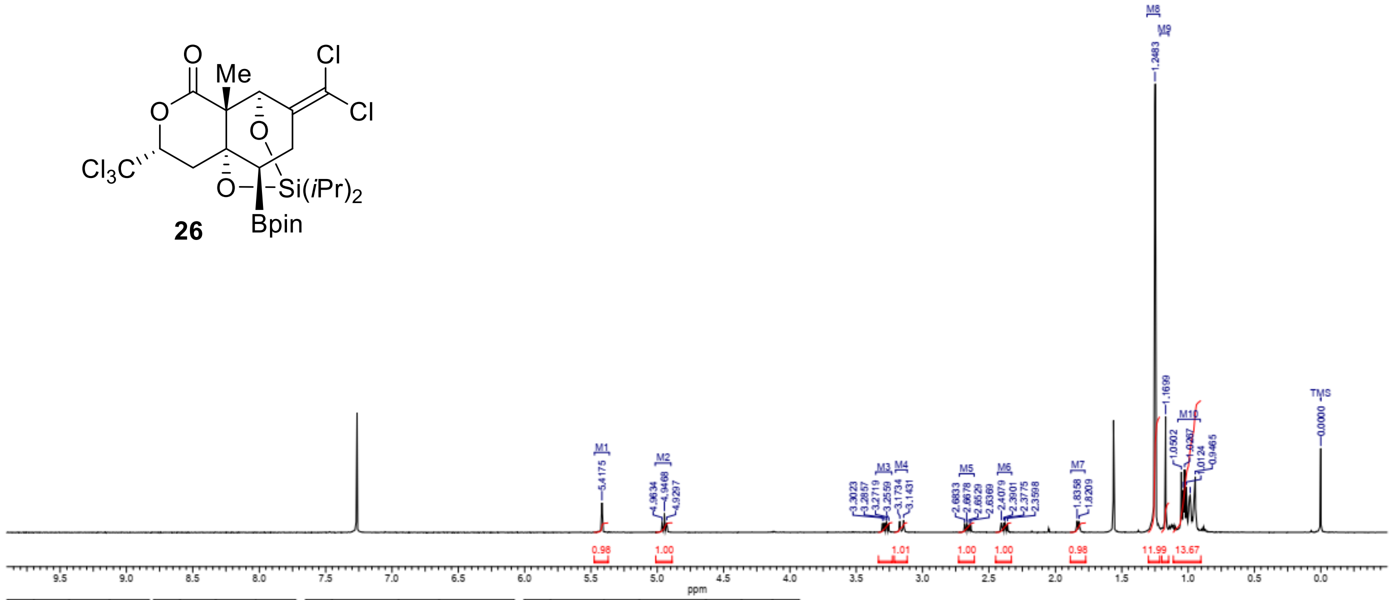
^{13}C NMR, $CDCl_3$, 125MHz



25

No.	(ppm)	(Hz)	Height
1	14.02	1763.1	0.2642
2	16.53	2078.6	0.1904
3	16.79	2112.2	0.2101
4	16.85	2119.4	0.2939
5	17.42	2191.4	0.2035
6	17.46	2196.2	0.2503
7	17.66	2221.4	0.2413
8	24.59	3092.3	0.4107
9	24.71	3107.9	0.5082
10	33.90	4263.1	0.2031
11	36.65	4609.8	0.1796
12	50.43	6342.1	0.1297
13	76.95	9678.3	0.2050
14	77.00	9684.3	1.0000
15	79.16	9955.4	0.1908
16	82.73	10405.3	0.1988
17	84.54	10633.2	0.3312
18	98.52	12390.7	0.1239
19	170.02	21383.1	0.1265
20	206.01	25909.4	0.1111

[1]H NMR, $CDCl_3$, 500MHz



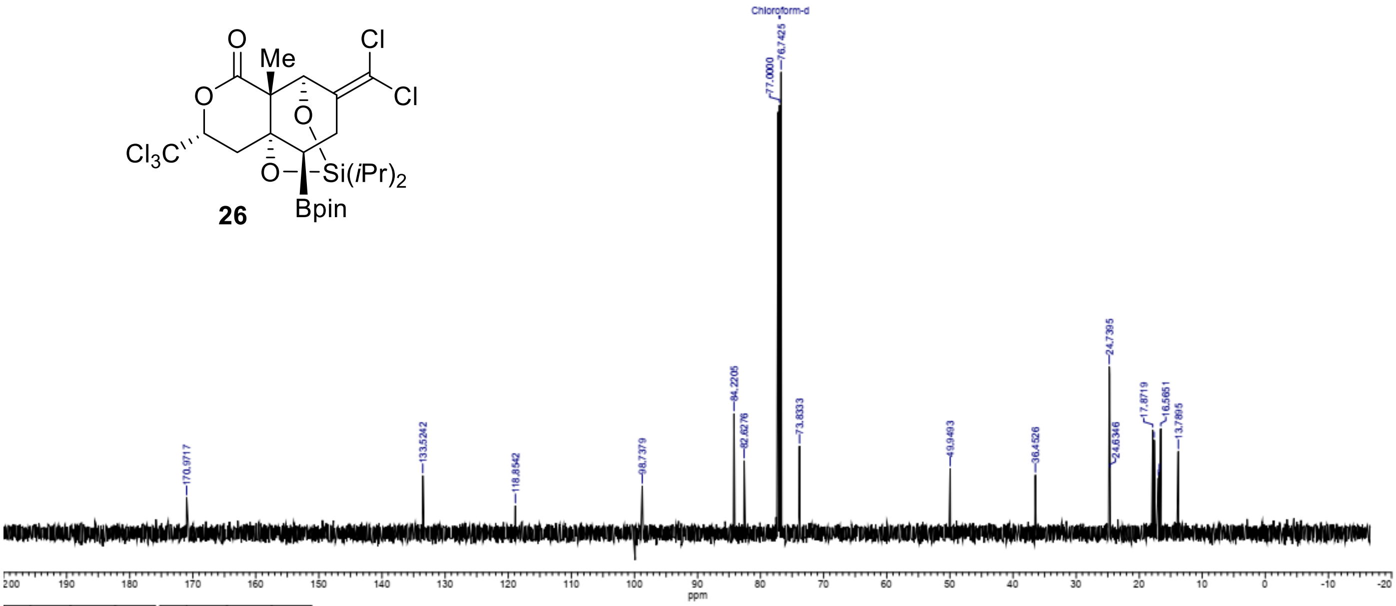
No.	(ppm)	(Hz)	Height
1	0.00	0.0	0.1878
2	0.95	473.4	0.1241
3	0.95	476.0	0.1161
4	0.98	491.7	0.0828
5	0.99	494.0	0.0798
6	0.99	495.8	0.0778
7	1.01	506.4	0.1002
8	1.03	513.5	0.1404
9	1.04	518.1	0.0948
10	1.05	525.3	0.1348
11	1.17	585.1	0.2581
12	1.25	624.4	1.0000
13	1.82	910.8	0.0252
14	1.84	918.2	0.0260
15	2.36	1180.3	0.0201
16	2.38	1189.1	0.0205

No.	(ppm)	(Hz)	Height
17	2.39	1195.4	0.0215
18	2.41	1204.3	0.0217
19	2.64	1318.9	0.0154
20	2.65	1326.9	0.0161
21	2.67	1334.3	0.0187
22	2.68	1342.1	0.0173
23	3.14	1572.0	0.0275
24	3.17	1587.2	0.0252
25	3.26	1628.5	0.0206
26	3.27	1636.5	0.0229
27	3.29	1643.4	0.0216
28	3.30	1651.7	0.0206
29	4.93	2465.6	0.0217
30	4.95	2474.2	0.0422
31	4.96	2482.5	0.0216
32	5.42	2709.6	0.0660

No.	(ppm)	Value	Absolute Value
1	[0.90 .. 1.11]	13.667	2.17123e+6
2	[1.15 .. 1.20]	3.043	4.83412e+5
3	[1.21 .. 1.30]	11.994	1.90538e+6
4	[1.77 .. 1.89]	0.981	1.55906e+5
5	[2.33 .. 2.45]	0.998	1.58597e+5
6	[2.61 .. 2.73]	0.998	1.58552e+5
7	[3.12 .. 3.21]	1.013	1.60977e+5
8	[3.23 .. 3.33]	1.013	1.60969e+5
9	[4.89 .. 5.01]	1.000	1.58868e+5
10	[5.37 .. 5.47]	0.983	1.56136e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M10	0.99	[0.91 .. 1.08]	-	m	14
2	M9	1.18	[1.15 .. 1.21]	-	s	3
3	M8	1.26	[1.21 .. 1.31]	-	s	12
4	M7	1.83	[1.78 .. 1.88]	7.45	d	1
5	M6	2.38	[2.33 .. 2.44]	15.18	dd	1
6	M6	2.38	[2.33 .. 2.44]	8.88	dd	1
7	M5	2.67	[2.61 .. 2.72]	15.32	dd	1
8	M5	2.67	[2.61 .. 2.72]	7.88	dd	1
9	M4	3.16	[3.12 .. 3.20]	15.18	d	1
10	M3	3.29	[3.23 .. 3.35]	15.04	dd	1
11	M3	3.29	[3.23 .. 3.35]	8.16	dd	1
12	M2	4.96	[4.90 .. 5.01]	8.45	t	1
13	M2	4.96	[4.90 .. 5.01]	8.45	t	1
14	M1	5.42	[5.36 .. 5.47]	-	s	1

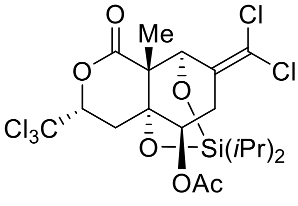
[13]C NMR, $CDCl_3$, 125MHz



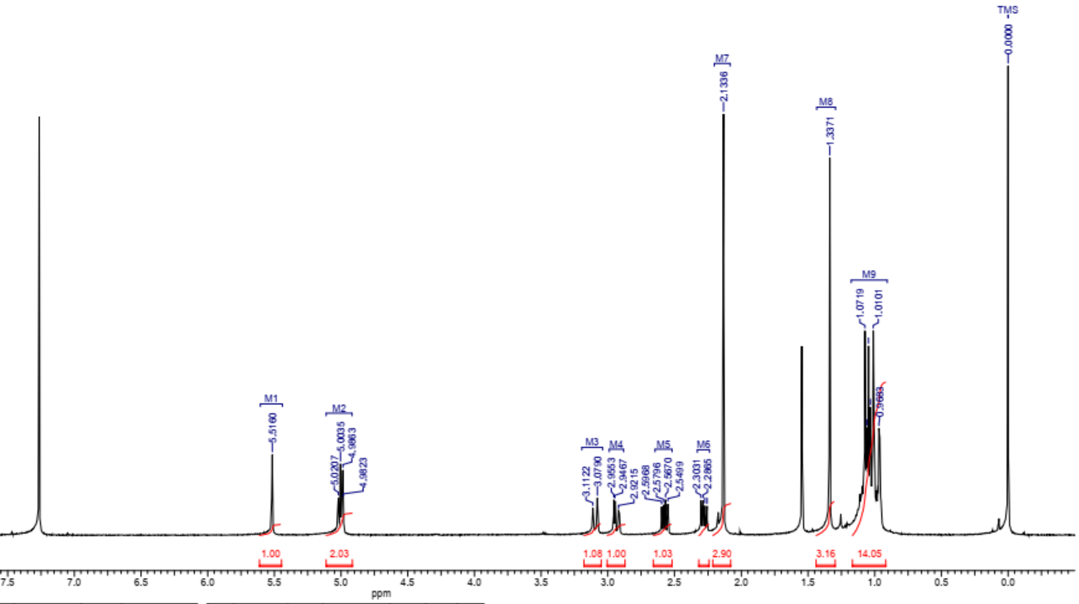
No.	(ppm)	(Hz)	Height
1	13.79	1734.3	0.1767
2	16.57	2083.4	0.2257
3	16.83	2117.0	0.1317
4	16.98	2135.0	0.1192
5	17.63	2217.8	0.2015
6	17.78	2235.8	0.1648
7	17.87	2247.7	0.2234
8	24.63	3098.3	0.1364
9	24.69	3105.5	0.3431
10	24.74	3111.5	0.3607
11	36.45	4584.6	0.1258

No.	(ppm)	(Hz)	Height
12	49.95	6282.1	0.1393
13	73.83	9286.0	0.1884
14	76.74	9651.9	1.0000
15	77.00	9684.3	0.9284
16	82.63	10392.1	0.1568
17	84.22	10592.4	0.2587
18	98.74	12418.3	0.1019
19	118.85	14948.3	0.0587
20	133.52	16793.3	0.1246
21	170.97	21503.1	0.0764

[1]H NMR, $CDCl_3$, 500MHz



27



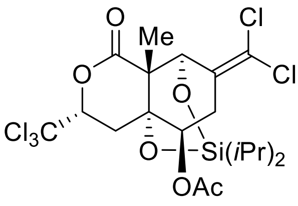
No.	(ppm)	(Hz)	Height
1	0.00	0.0	1.0000
2	0.96	481.4	0.2077
3	0.97	484.3	0.2284
4	1.01	503.2	0.3295
5	1.01	505.2	0.4359
6	1.03	517.0	0.2731
7	1.05	523.5	0.4038
8	1.06	529.8	0.2292
9	1.07	536.1	0.4359
10	1.34	668.7	0.8041
11	2.13	1067.1	0.8976
12	2.26	1128.7	0.0626
13	2.27	1136.7	0.0632
14	2.29	1143.6	0.0745
15	2.30	1151.9	0.0749
16	2.55	1275.3	0.0674

No.	(ppm)	(Hz)	Height
17	2.57	1283.9	0.0759
18	2.58	1290.2	0.0653
19	2.60	1298.8	0.0617
20	2.91	1456.9	0.0478
21	2.92	1461.2	0.0523
22	2.95	1473.8	0.0739
23	2.96	1478.1	0.0758
24	3.08	1540.0	0.0801
25	3.11	1556.6	0.0584
26	4.98	2492.0	0.0772
27	4.99	2494.0	0.1396
28	4.99	2496.3	0.1030
29	4.99	2498.0	0.0915
30	5.00	2502.6	0.1526
31	5.02	2511.2	0.0806
32	5.52	2758.9	0.1724

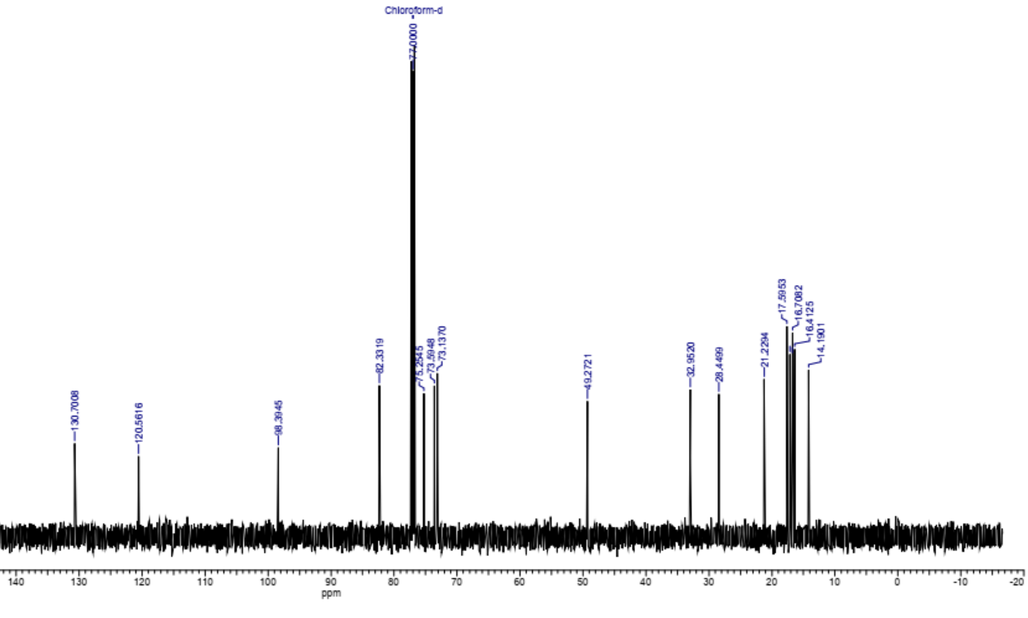
No.	(ppm)	Value	Absolute Value
1	[0.92 .. 1.17]	14.053	3.59367e+6
2	[1.30 .. 1.44]	3.160	8.08183e+5
3	[2.08 .. 2.21]	2.897	7.40907e+5
4	[2.24 .. 2.32]	1.011	2.58595e+5
5	[2.52 .. 2.66]	1.025	2.62158e+5
6	[2.87 .. 3.00]	0.999	2.55520e+5
7	[3.05 .. 3.18]	1.083	2.76967e+5
8	[4.92 .. 5.11]	2.029	5.18935e+5
9	[5.45 .. 5.61]	1.000	2.55713e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M9	1.04	[0.90 .. 1.18]	-	m	14
2	M8	1.36	[1.29 .. 1.43]	-	s	3
3	M7	2.14	[2.08 .. 2.20]	-	s	3
4	M6	2.28	[2.24 .. 2.33]	15.04	dd	1
5	M6	2.28	[2.24 .. 2.33]	8.16	dd	1
6	M5	2.58	[2.52 .. 2.65]	14.89	dd	1
7	M5	2.58	[2.52 .. 2.65]	8.59	dd	1
8	M4	2.94	[2.88 .. 2.99]	16.90	dd	1
9	M4	2.94	[2.88 .. 2.99]	4.30	dd	1
10	M3	3.12	[3.04 .. 3.20]	16.61	d	1
11	M2	5.01	[4.92 .. 5.11]	-	m	2
12	M1	5.52	[5.44 .. 5.61]	-	s	1

[13]C NMR, $CDCl_3$, 125MHz



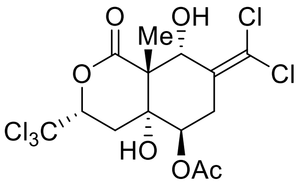
27



No.	(ppm)	(Hz)	Height
1	14.19	1784.7	0.3389
2	16.41	2064.2	0.3813
3	16.71	2101.4	0.4149
4	16.75	2106.2	0.3685
5	17.13	2154.2	0.3711
6	17.57	2209.4	0.4149
7	17.60	2213.0	0.4278
8	21.23	2670.0	0.3199
9	28.45	3578.1	0.2897
10	32.95	4144.4	0.2988
11	49.27	6196.9	0.2752

No.	(ppm)	(Hz)	Height
12	73.14	9198.4	0.3324
13	73.59	9256.0	0.3066
14	75.25	9464.8	0.2912
15	77.00	9684.3	0.9470
16	82.33	10354.9	0.3077
17	98.39	12375.1	0.1810
18	120.56	15163.0	0.1631
19	130.70	16438.2	0.1895
20	169.15	21274.0	0.1931
21	170.03	21384.3	0.1437

^{1}H NMR, $CDCl_3$, 500MHz



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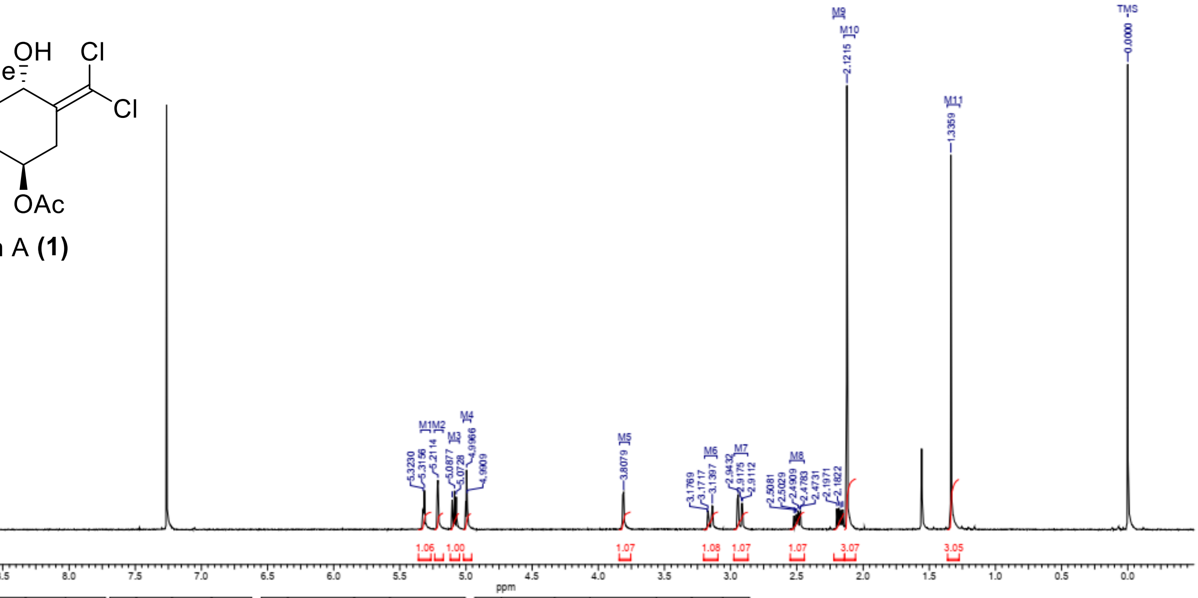
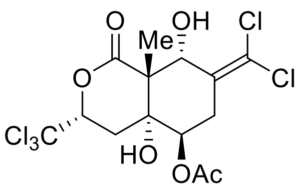


No.	(ppm)	(Hz)	Height
1	0.00	0.0	1.0000
2	1.34	668.2	0.8057
3	2.12	1061.1	0.9543
4	2.15	1077.1	0.0435
5	2.17	1084.3	0.0425
6	2.18	1091.5	0.0473
7	2.20	1098.9	0.0457
8	2.47	1237.0	0.0391
9	2.48	1239.5	0.0379
10	2.49	1245.8	0.0412
11	2.50	1248.4	0.0402
12	2.50	1251.9	0.0339
13	2.51	1254.4	0.0317
14	2.52	1260.4	0.0307
15	2.53	1263.0	0.0293
16	2.91	1456.1	0.0563
17	2.92	1459.2	0.0563
18	2.94	1472.1	0.0743
19	2.95	1475.2	0.0669
20	3.14	1569.5	0.0510
21	3.14	1570.3	0.0516
22	3.14	1572.0	0.0506
23	3.15	1573.2	0.0458
24	3.17	1585.2	0.0389
25	3.17	1586.4	0.0403
26	3.17	1587.8	0.0368
27	3.18	1588.9	0.0333
28	3.81	1904.6	0.0807
29	3.82	1908.3	0.0753
30	4.99	2496.3	0.0739
31	5.00	2499.1	0.1288
32	5.00	2502.3	0.0607
33	5.07	2537.2	0.0710
34	5.09	2544.7	0.0837
35	5.09	2545.8	0.0796
36	5.10	2553.3	0.0647
37	5.21	2606.5	0.1063
38	5.22	2608.8	0.0938
39	5.32	2658.6	0.0850
40	5.32	2662.4	0.0780

No.	(ppm)	Value	Absolute Value
1	[1.27 .. 1.36]	3.055	6.85776e+5
2	[2.06 .. 2.14]	3.075	6.90239e+5
3	[2.14 .. 2.22]	1.035	2.32448e+5
4	[2.44 .. 2.55]	1.066	2.39397e+5
5	[2.87 .. 2.98]	1.071	2.40453e+5
6	[3.10 .. 3.20]	1.083	2.43211e+5
7	[3.76 .. 3.84]	1.074	2.40995e+5
8	[4.96 .. 5.02]	0.996	2.23567e+5
9	[5.05 .. 5.12]	1.000	2.24494e+5
10	[5.17 .. 5.24]	1.020	2.29069e+5
11	[5.27 .. 5.36]	1.064	2.38874e+5

No.	Multiplet1	Shift1	(ppm)	J (Hz)	Type	H's
1	M11	1.32	[1.27 .. 1.36]	-	s	3
2	M10	2.10	[2.06 .. 2.14]	-	s	3
3	M9	2.18	[2.14 .. 2.22]	14.46	dd	1
4	M9	2.18	[2.14 .. 2.22]	7.30	dd	1
5	M8	2.50	[2.44 .. 2.55]	14.75	ddd	1
6	M8	2.50	[2.44 .. 2.55]	8.74	ddd	1
7	M8	2.50	[2.44 .. 2.55]	2.58	ddd	1
8	M7	2.92	[2.87 .. 2.97]	16.04	dd	1
9	M7	2.92	[2.87 .. 2.97]	3.15	dd	1
10	M6	3.15	[3.10 .. 3.20]	15.82	ddd	1
11	M6	3.15	[3.10 .. 3.20]	2.65	ddd	1
12	M6	3.15	[3.10 .. 3.20]	1.00	ddd	1
13	M5	3.80	[3.77 .. 3.84]	3.72	d	1
14	M4	4.99	[4.96 .. 5.02]	3.01	t	1
15	M4	4.99	[4.96 .. 5.02]	3.01	t	1
16	M3	5.09	[5.05 .. 5.13]	8.59	dd	1
17	M3	5.09	[5.05 .. 5.13]	7.45	dd	1
18	M2	5.21	[5.17 .. 5.24]	2.29	d	1
19	M1	5.31	[5.27 .. 5.34]	3.72	d	1

^{13}C NMR, $CDCl_3$, 125MHz



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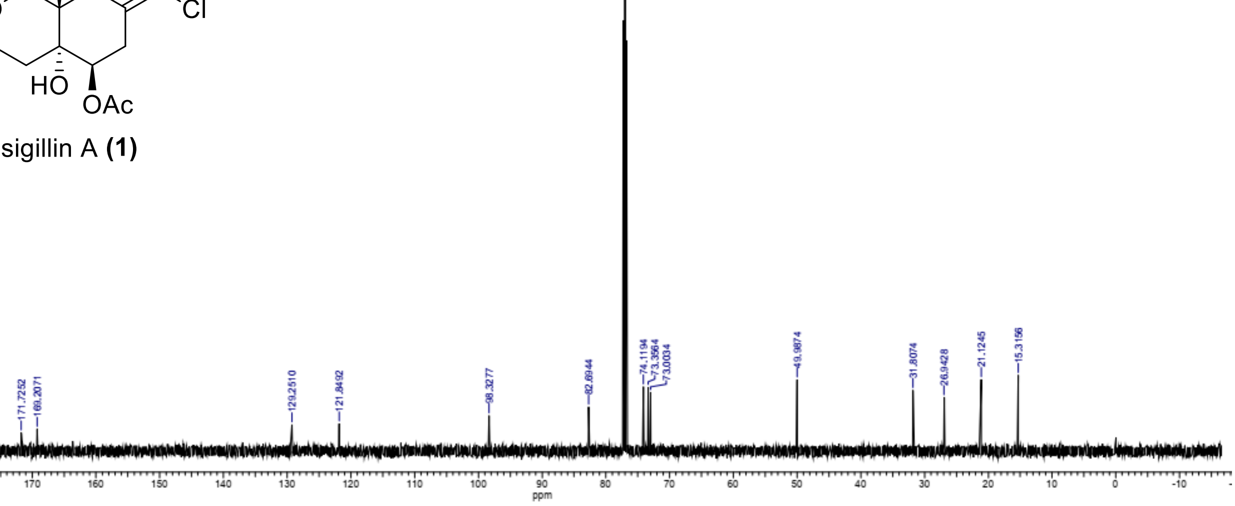

No.	(ppm)	(Hz)	Height
1	15.32	1926.2	0.1563
2	21.12	2656.8	0.1475
3	26.94	3388.6	0.1105
4	31.81	4000.4	0.1251
5	49.99	6286.9	0.1471
6	73.00	9181.6	0.1207
7	73.36	9226.0	0.1315
8	74.12	9322.0	0.1329
9	77.00	9684.3	1.0000
10	82.69	10400.5	0.0910
11	98.33	12366.7	0.0727
12	121.85	15325.0	0.0564
13	129.25	16255.9	0.0546
14	169.21	21281.2	0.0448
15	171.73	21597.9	0.0376