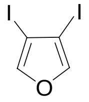
Supporting Information

Synthetic Studies Toward the Guanacastepenes

Chambers C. Hughes, Joshua J. Kennedy-Smith, and Dirk Trauner

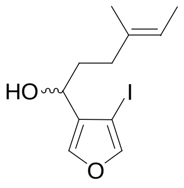
Center for New Directions in Organic Synthesis, Department of Chemistry, University of California—Berkeley, Berkeley, California 94720

General: Tetrahydrofuran (THF) was distilled from Na/benzoquinone, triethylamine (Et_3N) was distilled from CaH_2, and dichloromethane (CH_2Cl_2) and ether (Et_2O) were dried by passing through a column of activated alumina prior to use. *n*-Butyllithium (*n*-BuLi) was titrated periodically with diphenylacetic acid. All other starting materials and solvents are commercially available and were used without further purification. Chromatography was carried out with ICN SiliTech 32-63 D 60 Å silica gel. Reactions and chromatography fractions were analyzed with Merck silica gel 60 F_{254} plates. Extracts were dried over $MgSO_4$, and solvents were removed with a rotary evaporator at aspirator pressure. Unless otherwise noted, ^1H and ^{13}C NMR spectra were recorded in $CDCl_3$ (7.27 ppm) on a Bruker AVB 400 MHz spectrometer.



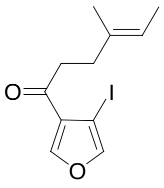
3

Large-scale preparation: To a 5 L three-neck flask fitted with a mechanical stirrer, a water cooled condenser, and a 100 mL dropping funnel was added 2,3-diiodo-2-butene-1,4-diol (100 g, 294 mmol), NMP (1.25 L), and hexanes (2 L). The mixture was stirred vigorously at 85 °C. To this solution was added a preheated (85 °C) solution of $K_2Cr_2O_7$ (86.5 g, 294 mmol) in H_2SO_4 (3 M, 365 mL) dropwise in portions (60 mL) over 2 h. The biphasic mixture was stirred at 85 °C for 5 h and then allowed to cool to rt. The hexane layer was decanted, and the remaining solvent was extracted once with hexanes (1 L). The hexane layers were combined, washed successively with water (2 x 500 mL), a saturated $Na_2S_2O_3$ solution (500 mL), and brine (500 mL), dried, passed over a short plug of silica gel, and concentrated to yield **3** (10-20 g, 10-20%) as a pale yellow liquid: ^1H NMR (300 MHz): δ 7.45 (s, 2H).



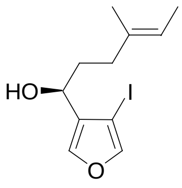
5

To a solution of iodide **3** (13.6 g, 42.5 mmol) in Et_2O (210 mL) at –78 °C was added a solution of *n*-BuLi in hexanes (17.0 mL, 2.5 M, 43 mmol) over 2 min. The yellow mixture was stirred for 15 min, after which a solution of (*E*)-4-methylhex-4-enal (4.14 g, 36.9 mmol) in Et_2O (100 mL) was added dropwise via cannula. After 20 min at –78 °C, the reaction was quenched with a saturated NH_4Cl solution (200 mL), diluted with ether, and washed with water (150 mL) and brine (150 mL). The organic layer was dried, filtered, and concentrated. The product was purified by column chromatography (6% EtOAc in hexanes) to give 7.03 g (62%) of racemic **5** as a colorless oil: IR (film): 3368 (br), 2917, 2859 cm^{-1}; ^{1}H NMR: δ 7.40 (d, *J* = 1.2 Hz, 1 H), 7.34 (s, 1 H), 5.27 (m, 1 H), 4.54 (m, 1 H), 2.34-2.01 (m, 3 H), 1.99-1.78 (m, 2 H), 1.64 (s, 3 H), 1.59 (d, *J* = 6.8 Hz, 3 H); ^{13}C NMR: δ 146.2, 140.2, 135.3, 130.5, 119.4, 67.8, 66.3, 35.9, 35.1, 15.9, 13.6; HRMS (EI+): *m/z* (M+) calcd for $C_{11}H_{15}IO_2$ 306.0117; found 306.0111.



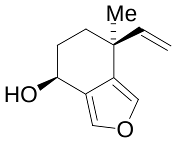
4

To a solution of alcohol **5** (6.96 g, 22.7 mmol) in CH_2Cl_2 (115 mL) at 0 °C was added Dess-Martin periodinane (12.0 g, 28.3 mmol) in one portion. The solution was allowed to warm to rt over 2 h and then quenched with isopropanol. The solvent was removed, and the resulting white residue was redissolved in ether and washed successively with a 1:1 solution of $NaHCO_3/Na_2S_2O_3$ (100 mL), a saturated $NaHCO_3$ solution (100 mL), water (100 mL), and brine (100 mL). The organic layer was dried, filtered, and concentrated. The product was purified by column chromatography (4% EtOAc in hexanes) to furnish 6.10 g (88%) of **4** as a colorless oil: IR (film): 3134, 2975, 2915, 2859, 1679 cm^{-1}; ^{1}H NMR: δ 8.00 (d, *J* = 1.6 Hz, 1 H), 7.48 (d, *J* = 1.2 Hz, 1 H), 5.30-5.17 (m, 1 H), 2.85 (m, 2 H), 2.38 (m, 2 H), 1.63 (s, 3 H), 1.57 (d, *J* = 6.4 Hz, 3 H); ^{13}C NMR: δ 193.9, 148.5, 148.3, 134.4, 126.2, 119.5, 63.0, 40.0, 33.8, 16.0, 13.6; HRMS (EI+): *m/z* (M+) calcd for $C_{11}H_{13}IO_2$ 303.9960; found 303.9954.

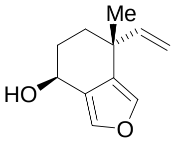


5

To a solution of (–)-*B*-chlorodiisopinocampheylborane (DIP-Cl, 665 mg, 2.07 mmol) in THF (2 mL) at –20 °C was added a solution of ketone **4** (570 mg, 1.87 mmol) in THF (1 mL) via cannula. The reaction mixture was stirred at –20 °C for 66 h. The solvent was removed, the resulting residue was dissolved in ether, and neat diethanolamine (0.40 mL, 4.2 mmol) was added. The mixture was stirred for another 12 h and then filtered through Celite. The product was purified by column chromatrography (4% EtOAc to 5% EtOAc in hexanes) to afford 430 mg (75%) of enantioenriched **5** as a colorless oil: $[\alpha]_D = -27.4$ (c = 1.48, $CHCl_3$). The enantiomeric excess was determined by chiral HPLC [Chiralpak OD^{TM} column, 5% *i*-PrOH in hexanes at 1.0 mLmin^{-1}; t_R (major enantiomer) = 7.8 min, t_R (minor enantiomer) = 8.7 min] to be 94%.

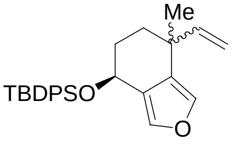


1



6

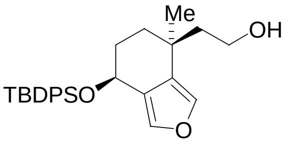
To a solution of alcohol **5** (2.90 g, 9.47 mmol) in MeCN (95 mL) was added H_2O (3.9 mL), Et_3N (3.9 mL, 28 mmol), and tetrabutylammonium bromide (TBAB, 9.16 g, 28.4 mmol). The solution was purged with nitrogen for 15 min, and then $Pd(OAc)_2$ (105 mg, 0.468 mmol) was quickly added. The reaction flask was placed in a preheated oil bath and stirred for 17 h at 75 °C. The mixture was cooled, filtered through Celite with EtOAc washings, and concentrated. The resulting oil was triturated with Et_2O and again filtered through Celite. The product was purified by column chromatography (15% EtOAc in hexanes) to yield 1.40 g (83%) of **1** and **6** as a 5.1:1 mixture of diastereomers: IR (film): 3345 (br), 2929, 2865 cm^{-1}; HRMS (EI+): *m/z* (M+) calcd for $C_{11}H_{14}O_2$ 178.0994; found 178.0989.



16

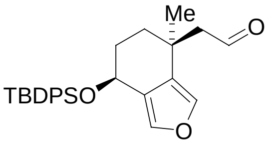
To a solution of alcohols **1** and **6** (3.10 g, 17.4 mmol) in CH_2Cl_2 (175 mL) was added imidazole (4.74 g, 69.6 mmol) and DMAP (20 mg, 0.16 mmol). This mixture was cooled to 0 °C, and TBDPSCl

(6.0 mL, 23 mmol) was added dropwise via syringe. The solution was allowed to warm to rt for 12 h, filtered through Celite with CH_2Cl_2 washings, and concentrated. The product was purified by column chromatography (2% EtOAc in hexanes) to give 7.10 g (98%) of **16** as a mixture of diastereomers: IR (film): 3071, 2956, 2931, 2857 cm^{-1}; HRMS (EI+): *m/z* (M+) calcd for $C_{27}H_{32}O_2Si$ 416.2172; found 416.2167.



7

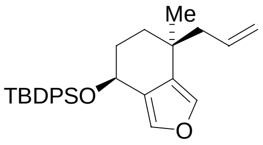
To a solution of 9-BBN dimer (435 mg, 1.78 mmol) in THF (7.4 mL) was added a solution of alkene **16** (742 mg, 1.78 mmol) in THF (7.4 mL) dropwise via cannula. The flask containing the alkene was rinsed with an additional 1 mL THF. The mixture was heated at reflux for 4 h and then cooled to 0 °C. EtOH (2.5 mL) was slowly added. A 3 M aqueous NaOH solution (1.8 mL) and a 30% aqueous H_2O_2 solution (1.8 mL) were then added. After 10 min at 0 °C, the solution was allowed to warm to rt over 3 h. The mixture was poured into water (50 mL) and extracted with ether (3 x 100 mL). The combined organic extracts were washed once with brine (30 mL), dried, filtered, and concentrated. The product was purified by column chromatography (20% EtOAc in hexanes) to yield 623 mg (81%) of **7** as a colorless oil: IR (film): 3350 (br), 3070, 2932, 2857 cm^{-1}; ^1H NMR: δ 7.67 (m, 4 H), 7.46-7.30 (m, 6 H), 7.12 (s, 1 H), 6.76 (s, 1 H), 4.80 (m, 1 H), 3.74 (m, 2 H), 2.03 (m, 1 H), 1.88-1.76 (m, 1 H), 1.74-1.60 (m, 2 H), 1.55-1.39 (m, 2 H), 1.30 (m, 1 H), 1.13 (s, 3 H), 1.05 (s, 9 H); ^{13}C NMR: δ 139.4, 137.5, 136.1, 136.1, 134.5, 134.2, 130.0, 129.9, 127.8, 127.8, 124.8, 63.8, 60.2, 45.7, 32.5, 31.8, 30.3, 30.1, 27.2, 19.4; HRMS (EI+): *m/z* (M+) calcd for $C_{27}H_{34}O_3Si$ 434.2277; found 434.2279.



17

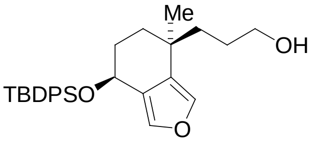
Dry CH_2Cl_2 (7.5 mL) was added to a solution of oxalyl chloride in CH_2Cl_2 (1.7 mL, 2.0 M, 3.4 mmol) at –78 °C. DMSO (0.48 mL, 6.8 mmol) was added to this solution dropwise via syringe. After 30 min at –78 °C, a solution of alcohol **7** (492 mg, 1.13 mmol) in CH_2Cl_2 (7.5 mL) was added via cannula. The flask containing the alcohol was rinsed with an additional 1 mL CH_2Cl_2. The reaction mixture was stirred at –78 °C for 1 h, and Et_3N (1.4 mL, 10 mmol) was added dropwise via syringe. The mixture was allowed to warm to rt over 2 h, then quenched with water, poured into water (50 mL) and a saturated

NH_4Cl solution (50 mL), and extracted with CH_2Cl_2 (3 x 50 mL). The combined organic extracts were dried, filtered, and concentrated. The product was purified by column chromatography (10% EtOAc in hexanes) to furnish 445 mg (91%) of **17** as a colorless oil: IR (film): 3018, 2932, 2858, 1719 cm^{-1}; ^{1}H NMR: δ 9.79 (t, *J* = 2.8 Hz, 1 H), 7.69 (m, 4 H), 7.50-7.35 (m, 6 H), 7.19 (d, *J* = 1.6 Hz, 1 H), 6.79 (m, 1 H), 4.86 (m, 1 H), 2.60 (t, *J* = 2.8 Hz, 2 H), 2.12 (m, 1 H), 1.91-1.72 (m, 2 H), 1.48 (m, 1 H), 1.28 (s, 3 H), 1.07 (s, 9 H); ^{13}C NMR: δ 203.3, 139.8, 137.8, 136.1, 136.1, 134.4, 134.1, 129.9, 129.0, 127.9, 127.8, 124.6, 63.5, 55.8, 33.0, 31.7, 30.0, 29.9, 27.1, 19.4; HRMS (EI+): *m/z* (M+) calcd for $C_{27}H_{32}O_3Si$ 432.2121; found 432.2118.



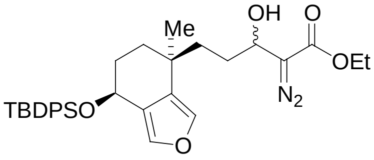
18

To a solution of methyltriphenylphosphonium bromide (1.32 g, 3.70 mmol) in THF (12.4 mL) was added a solution of *n*-BuLi in hexanes (1.3 mL, 2.4 M, 3.1 mmol) at 0 °C dropwise via syringe. After 1 h at 0 °C, a solution of aldehyde **17** (445 mg, 1.03 mmol) in THF (6.8 mL) was added via cannula. The flask containing the aldehyde was rinsed twice with 1 mL THF. After 5 min the mixture was allowed to warm to rt and then quenched with water after 1 h. The solution was poured into water (50 mL) and extracted with ether (3 x 50 mL). The combined organic extracts were washed once with brine (30 mL), dried, filtered, and concentrated. The product was purified by column chromatography (2% EtOAc in hexanes) to afford 422 mg (95%) of **18** as a colorless oil: IR (film): 3071, 2956, 2931, 2857, 2360 cm^{-1}; ^{1}H NMR: δ 7.79-7.69 (m, 4 H), 7.52-7.35 (m, 6 H), 7.16 (d, *J* = 1.6 Hz, 1 H), 6.85 (m, 1 H), 5.87 (m, 1 H), 5.15-5.06 (m, 2 H), 4.88 (m, 1 H), 2.34 (m, 2 H), 2.01 (m, 1 H), 1.93-1.82 (m, 1 H), 1.79-1.70 (m, 1 H), 1.32 (m, 1 H), 1.15 (s, 3 H), 1.12 (s, 9 H); ^{13}C NMR: δ 139.1, 137.5, 136.1, 136.1, 135.6, 134.7, 134.4, 130.3, 129.8, 127.8, 127.8, 125.1, 117.7, 64.4, 47.7, 32.8, 32.8, 30.1, 29.3, 27.2, 19.5; HRMS (EI+): *m/z* (M+) calcd for $C_{28}H_{34}O_2Si$ 430.2328; found 430.2327.



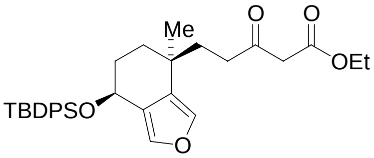
19

To a solution of 9-BBN dimer (259 mg, 1.06 mmol) in THF (4 mL) was added a solution of alkene **18** (416 mg, 0.966 mmol) in THF (4 mL) dropwise via cannula. The flask containing the alkene was rinsed twice with 1 mL THF. After 5 h at rt, the mixture was cooled to 0 °C, and EtOH (1.4 mL) was slowly added. A 3 M aqueous NaOH solution (1 mL) and a 30% aqueous H_2O_2 solution (1 mL) were then added. After 10 min at 0 °C, the solution was allowed to warm to rt over 1 h. The mixture was poured into water (50 mL) and extracted with ether (3 x 50 mL). The combined organic extracts were washed once with brine (30 mL), dried, filtered, and concentrated. The product was purified by column chromatography (20% EtOAc in hexanes) to yield 398 mg (92%) of **19** as a colorless oil: IR (film): 3350 (br), 3070, 2933, 2857 cm^{-1}; ^1H NMR: δ 7.78-7.67 (m, 4 H), 7.50-7.36 (m, 6 H), 7.15 (d, *J* = 1.6 Hz, 1 H), 6.84 (m, 1 H), 4.87 (m, 1 H), 3.69-3.63 (m, 2 H), 2.00 (m, 1 H), 1.93-1.83 (m, 1 H), 1.79-1.58 (m, 6 H), 1.34 (m, 1 H), 1.16 (s, 3 H), 1.11 (s, 9 H); ^{13}C NMR: δ 139.2, 137.3, 136.1, 136.1, 134.6, 134.3, 130.4, 129.8, 129.8, 127.8, 127.7, 125.1, 64.2, 63.7, 39.3, 32.5, 32.4, 30.1, 29.6, 28.2, 27.1, 19.4; HRMS (EI+): *m/z* (M+) calcd for $C_{28}H_{36}O_3Si$ 448.2434; found 448.2427.



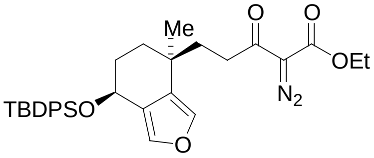
12

Dry CH_2Cl_2 (5.3 mL) was added to a solution of oxalyl chloride in CH_2Cl_2 (1.2 mL, 2.0 M, 2.4 mmol) at –78 °C. DMSO (0.34 mL, 4.8 mmol) was added to this solution dropwise via syringe. After 30 min at –78 °C, a solution of alcohol **19** (354 mg, 0.789 mmol) in CH_2Cl_2 (5.3 mL) was added via cannula. The flask containing the alcohol was rinsed twice with 1 mL CH_2Cl_2. The reaction mixture was stirred at –78 °C for 1 h, and Et_3N (1.0 mL, 7.2 mmol) was added dropwise via syringe. The mixture was allowed to warm to rt over 2 h, then quenched with water, poured into water (50 mL) and a saturated NH_4Cl solution (50 mL), and extracted with CH_2Cl_2 (3 x 50 mL). The combined organic extracts were dried, filtered, and concentrated. The product was filtered through a plug of silica (20% EtOAc in hexanes) to furnish **11** as a colorless oil, which was immediately taken on into the next step: IR (film): 3071, 2932, 2858, 1725 cm^{-1}; ^1H NMR: δ 9.81 (m, 1 H), 7.76-7.65 (m, 4 H), 7.50-7.36 (m, 6 H), 7.13 (d, *J* = 1.2 Hz, 1 H), 6.77 (m, 1 H), 4.85 (m, 1 H), 2.62-2.41 (m, 2 H), 1.99 (m, 1 H), 1.93-1.82 (m, 3 H), 1.78-1.67 (m, 1 H), 1.33 (m, 1 H), 1.15 (s, 3 H), 1.09 (s, 9 H). To a solution of aldehyde **11** in MeCN (7.8 mL) was added ethyl diazoacetate (0.22 mL, 1.6 mmol) followed by 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 22 μL, 0.15 mmol) dropwise via syringe. The mixture was stirred at rt for 15 h, then poured into water (50 mL) and extracted with ether (3 x 50 mL). The combined organic extracts were washed once with brine (30 mL), dried, filtered, and concentrated. The product was purified by column chromatography (20% EtOAc in hexanes) to afford 336 mg (76%) of **12** as a colorless oil: IR (film): 3441 (br), 3071, 2930, 2857, 2094, 1681 cm^{-1}; ^1H NMR: δ 7.71 (m, 4 H), 7.50-7.35 (m, 6 H), 7.13 (s, 1 H), 6.79 (s, 1 H), 4.84 (m, 1 H), 4.67 (m, 1 H), 4.27 (q, *J* = 7.2 Hz, 2 H), 2.84 (br s, 1 H), 2.08-1.94 (m, 1 H), 1.92-1.49 (m, 6 H), 1.38-1.26 (m, 4 H), 1.15 (s, 3 H), 1.08 (s, 9 H); ^{13}C NMR: δ 166.8, 139.3, 137.3, 136.1, 136.1, 134.6, 134.3, 130.1, 129.9, 129.8, 127.8, 127.7, 124.9, 67.5, 67.4, 63.9, 63.8, 61.2, 39.2, 39.0, 32.5, 32.1, 31.9, 30.0, 29.7, 29.5, 27.1, 19.4, 14.7; HRMS (FAB+): *m/z* (MLi+) calcd for $C_{32}H_{40}N_2O_5SiLi$ 567.2867; found 567.2876.



20

To a solution of alcohol **12** (336 mg, 0.559 mmol) in CH_2Cl_2 (12 mL) was added $Rh_2(OAc)_4$ (26 mg, 0.060 mol). The reaction mixture bubbled. After 30 min the solution was concentrated, and the crude material was filtered through a plug of silica (30% EtOAc in hexanes) to give 302 mg (95%) of **20** as a colorless oil: IR (film): 3070, 2932, 2857, 1744, 1719 cm^{-1}; ^{1}H NMR[1]: δ 7.76-7.64 (m, 4 H), 7.50-7.35 (m, 6 H), 7.12 (d, *J* = 1.2 Hz, 1 H), 6.76 (m, 1 H), 4.85 (m, 1 H), 4.21 (q, *J* = 7.2 Hz, 2 H), 3.44 (s, 2 H), 2.62 (m, 2 H), 2.09-1.82 (m, 4 H), 1.78-1.67 (m, 1 H), 1.36-1.23 (m, 4 H), 1.15 (s, 3 H), 1.10 (s, 9 H); ^{13}C NMR: δ 203.1, 167.3, 139.4, 137.4, 136.1, 136.0, 134.4, 134.2, 129.9, 129.2, 127.8, 127.7, 124.9, 63.4, 61.5, 49.5, 39.2, 36.2, 32.3, 31.5, 30.0, 30.0, 27.1, 19.4, 14.3; HRMS (FAB+): *m/z* (MLi+) calcd for $C_{32}H_{40}O_5SiLi$ 539.2805; found 539.2815.



13

To a solution of ketoester **20** (392 mg, 0.736 mmol) in MeCN (7.4 mL) at 0 °C was added Et_3N (0.31 mL, 2.2 mmol) followed by 4-acetamidobenzenesulfonyl azide (354 mg, 1.47 mmol). After 10 min at 0 °C, the solution was allowed to warm to rt over 4 h. The mixture was poured into water (50 mL) and extracted with ether (3 x 50 mL). The combined organic extracts were washed once with brine (30 mL), dried, filtered, and concentrated. The product was purified by column chromatography (10% EtOAc in hexanes) to afford 377 mg (92%) of **13** as a colorless oil: IR (film): 3071, 2932, 2857, 2134, 1713, 1658 cm^{-1}; ^{1}H NMR: δ 7.77-7.65 (m, 4 H), 7.49-7.35 (m, 6 H), 7.19 (d, *J* = 1.2 Hz, 1 H), 6.84 (m, 1 H), 4.83 (m, 1 H), 4.30 (q, *J* = 7.2 Hz, 2 H), 2.95 (m, 2 H), 2.03-1.83 (m, 4 H), 1.79-1.68 (m, 1 H), 1.40-1.29 (m, 4 H), 1.16 (s, 3 H), 1.08 (s, 9 H); ^{13}C NMR: δ 193.3, 161.5, 139.3, 137.5, 136.1, 136.1, 134.6, 134.3, 129.8,

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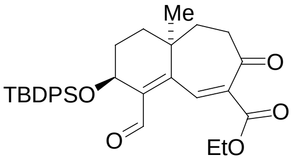
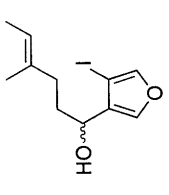
[1] The NMR spectra of this compound indicate that about 15% of the β-ketoester exists in the enol form: ^{1}H NMR: 12.20 (s, 0.17 H), 5.03 (s, 0.18 H).

129.8, 127.8, 127.7, 125.0, 64.2, 61.5, 37.0, 36.4, 32.6, 32.5, 30.1, 29.2, 27.2, 19.4, 19.1, 14.6; HRMS (FAB+): *m/z* (MLi+) calcd for $C_{32}H_{38}N_2O_5SiLi$ 565.2710; found 565.2717.

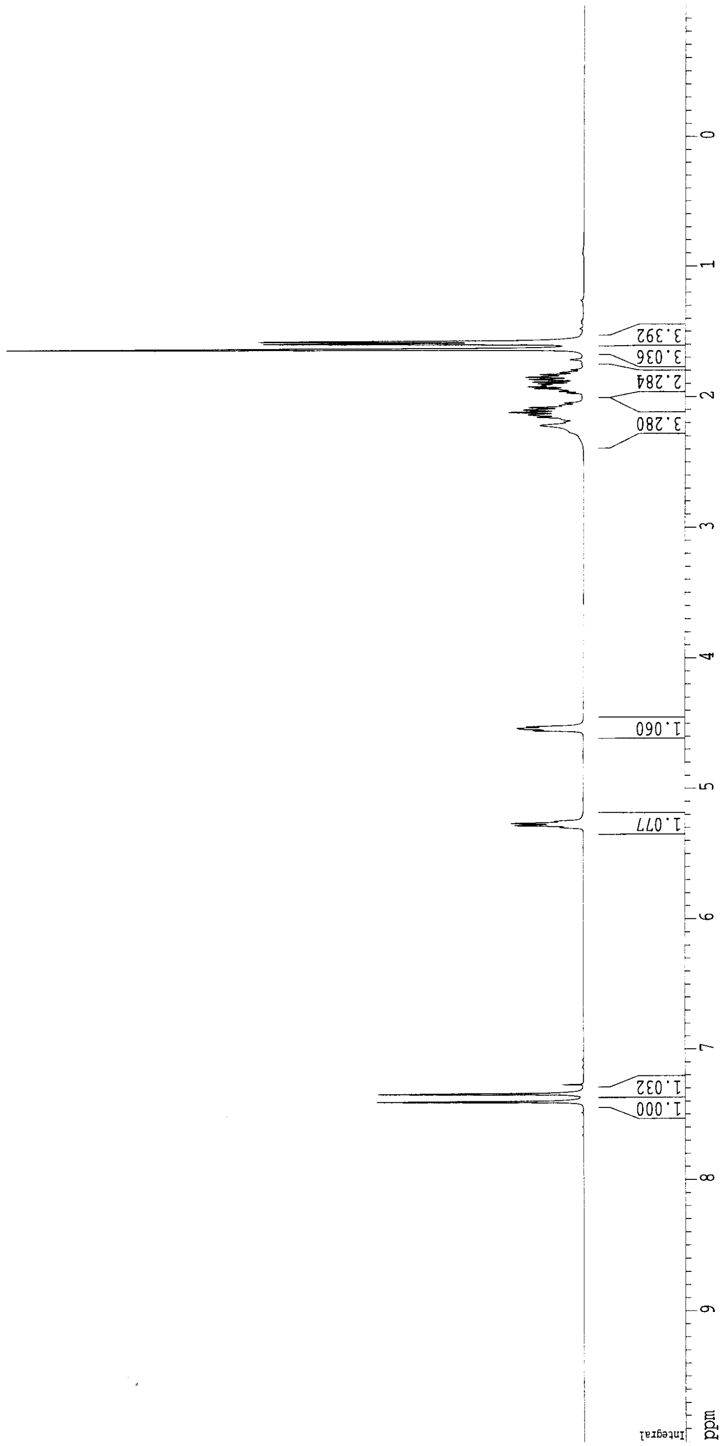


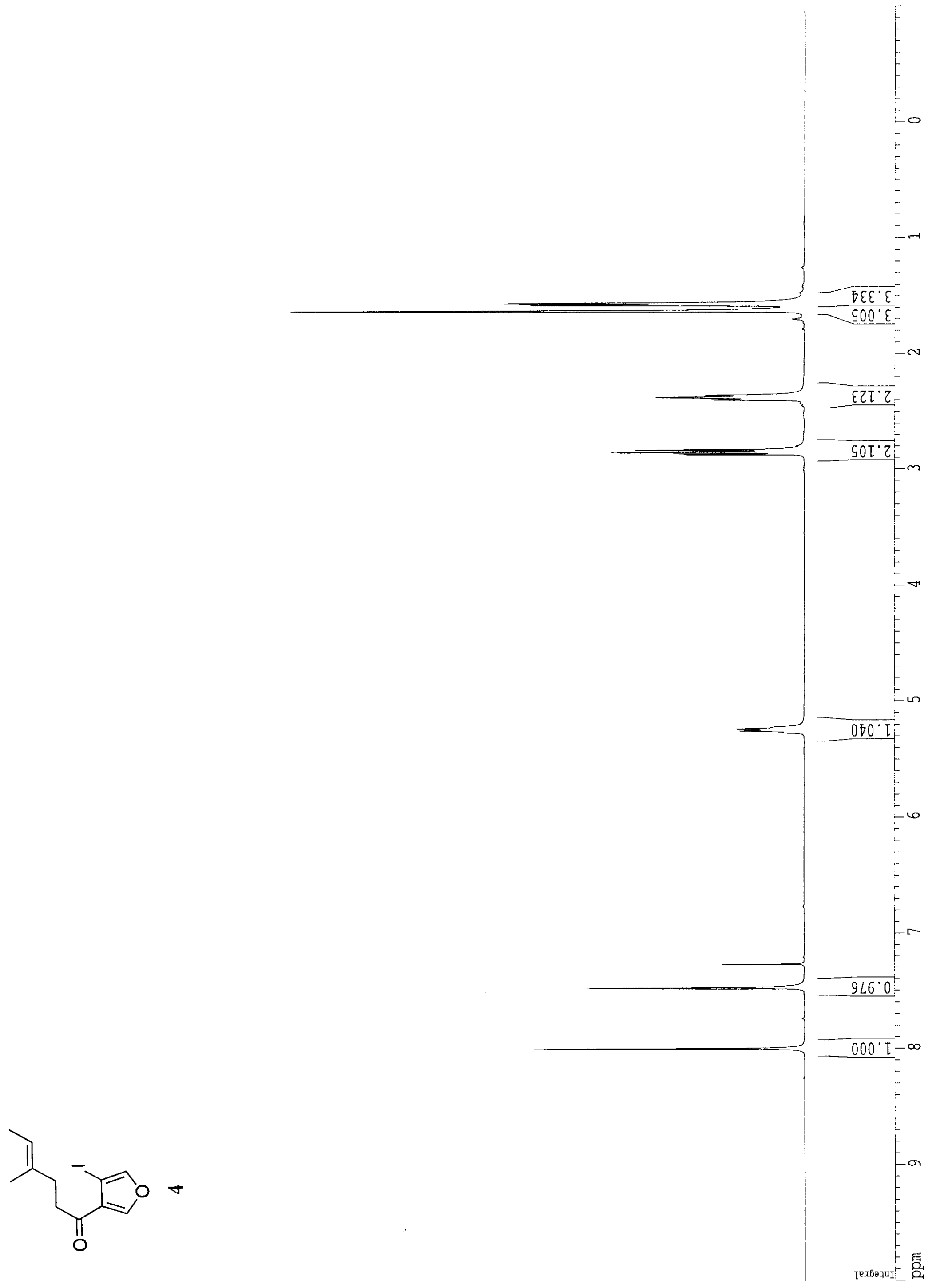
15

To a solution of $Rh_2(OAc)_4$ (10 mg, 0.023mol) in CH_2Cl_2 (40 mL) was added a solution of diazo compound **13** (44.0 mg, 0.0787 mmol) in CH_2Cl_2 (2 mL) dropwise via syringe. The flask containing the diazo compound was rinsed twice with 1 mL CH_2Cl_2. The solution was stirred for 4 h at rt and then concentrated. The product was purified by column chromatography (20% EtOAc in hexanes) to yield 20.4 mg (50%) of **15** as a colorless oil: IR (film): 2932, 2857, 2254, 1729, 1688 cm^{-1}; ^1H NMR: δ 9.57 (s, 1 H), 8.00 (s, 1 H), 7.75-7.68 (m, 2 H), 7.60-7.52 (m, 2 H), 7.49-7.30 (m, 6 H), 4.66 (m, 1 H), 4.33 (q, *J* = 7.2 Hz, 2 H), 2.75-2.65 (m, 1 H), 2.61-2.49 (m, 1 H), 2.27 (m, 1 H), 1.96 (m, 1 H), 1.83-1.72 (m, 2 H), 1.63-1.50 (m, 2 H), 1.36 (t, *J* = 7.2 Hz, 3 H), 1.06 (s, 3 H), 1.00 (s, 9 H); ^{13}C NMR: δ 200.2, 191.0, 164.3, 155.1, 143.9, 138.1, 136.2, 136.1, 134.0, 133.5, 130.0, 130.0, 127.8, 127.8, 62.2, 62.0, 39.6, 38.7, 34.1, 29.7, 27.3, 27.2, 19.5, 14.4; HRMS (EI+): *m/z* (M+) calcd for $C_{32}H_{38}O_5Si$ 530.2489; found 530.2494.

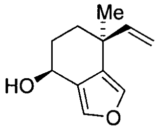

HO
I
O

5

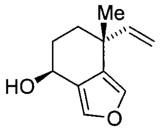

3.392
3.036
2.284
3.280
1.060
1.077
1.032
1.000
Integral
0
1
2
3
4
5
6
7
8
9
ppm



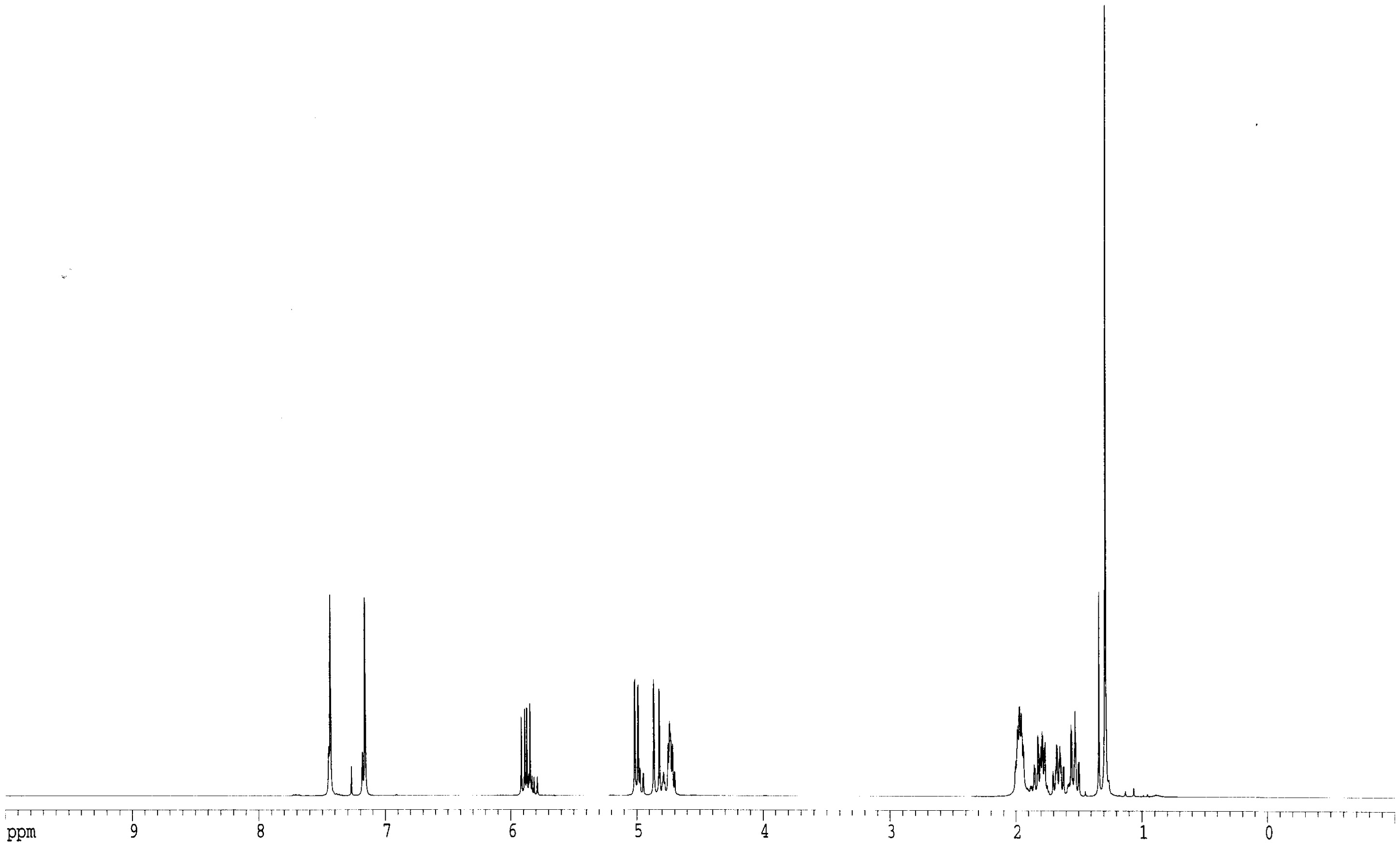
4

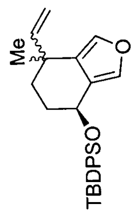


1

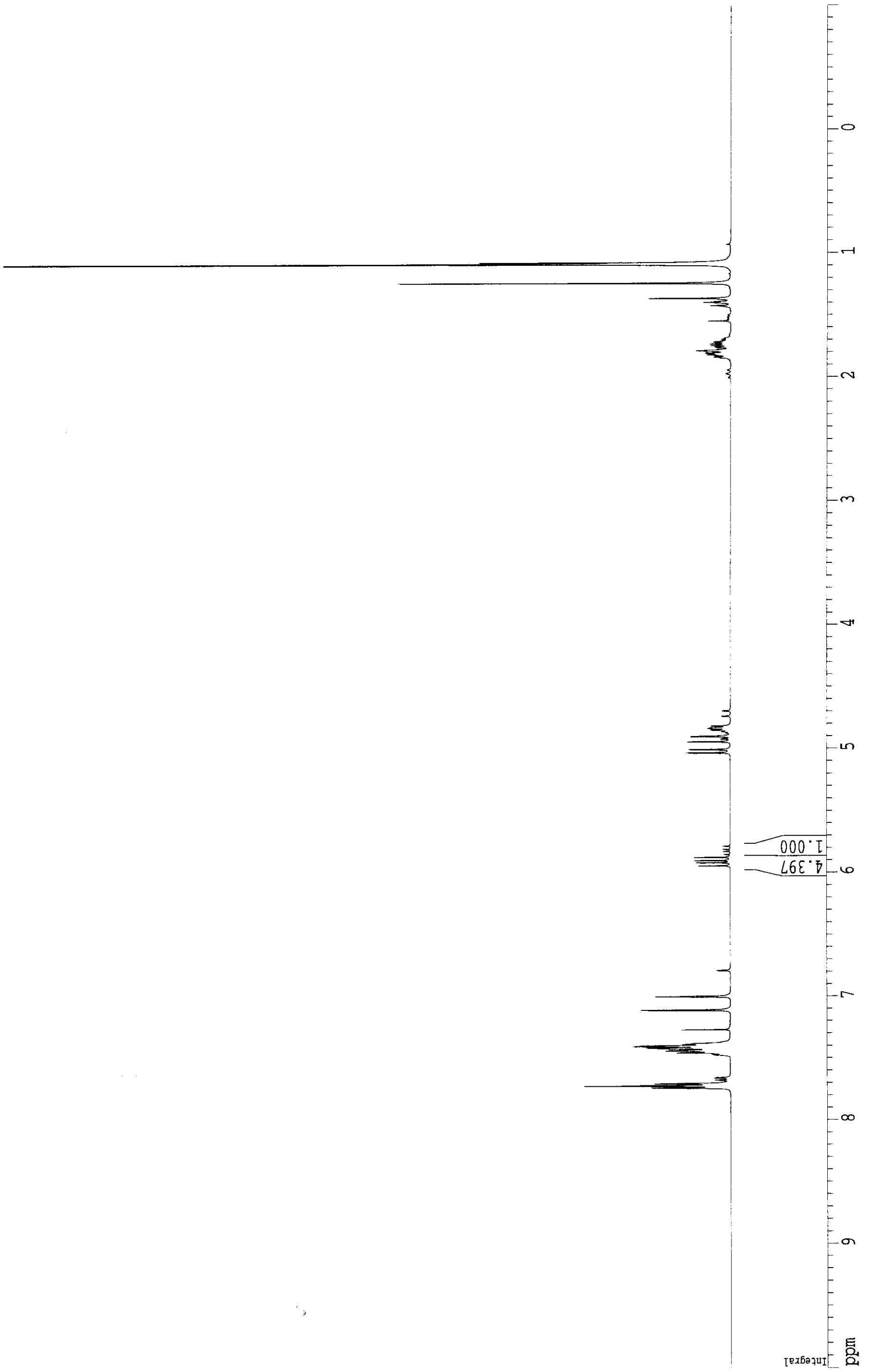


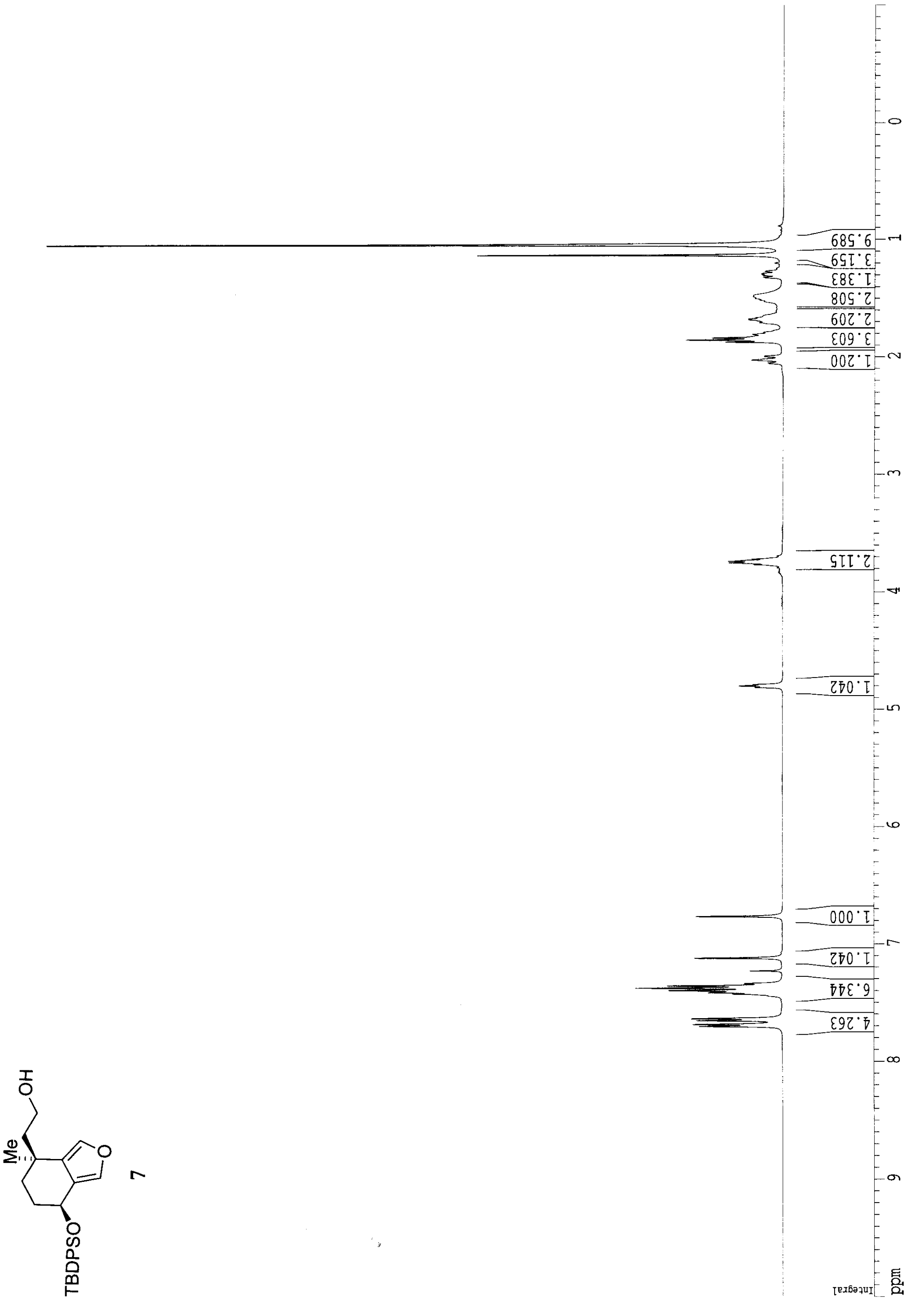
6





16

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Me
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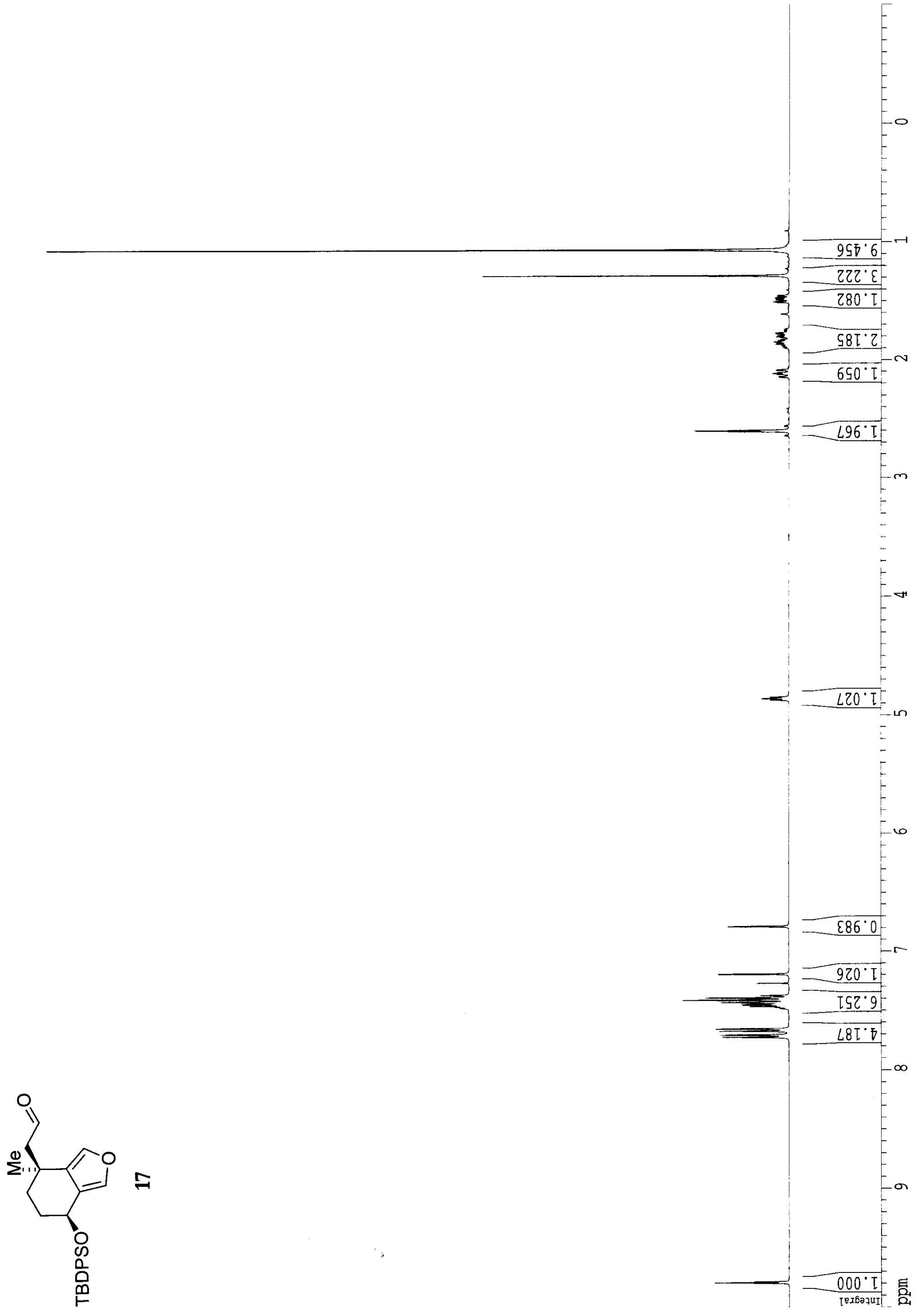
9.589
3.159
1.383
2.508
2.209
3.603
1.200
2.115
1.042
1.000
1.042
6.344
4.263
Integral
0
1
2
3
4
5
6
7
8
9
ppm

7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Me
O
O
Integral
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6.251
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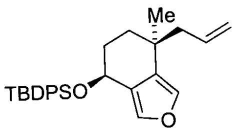
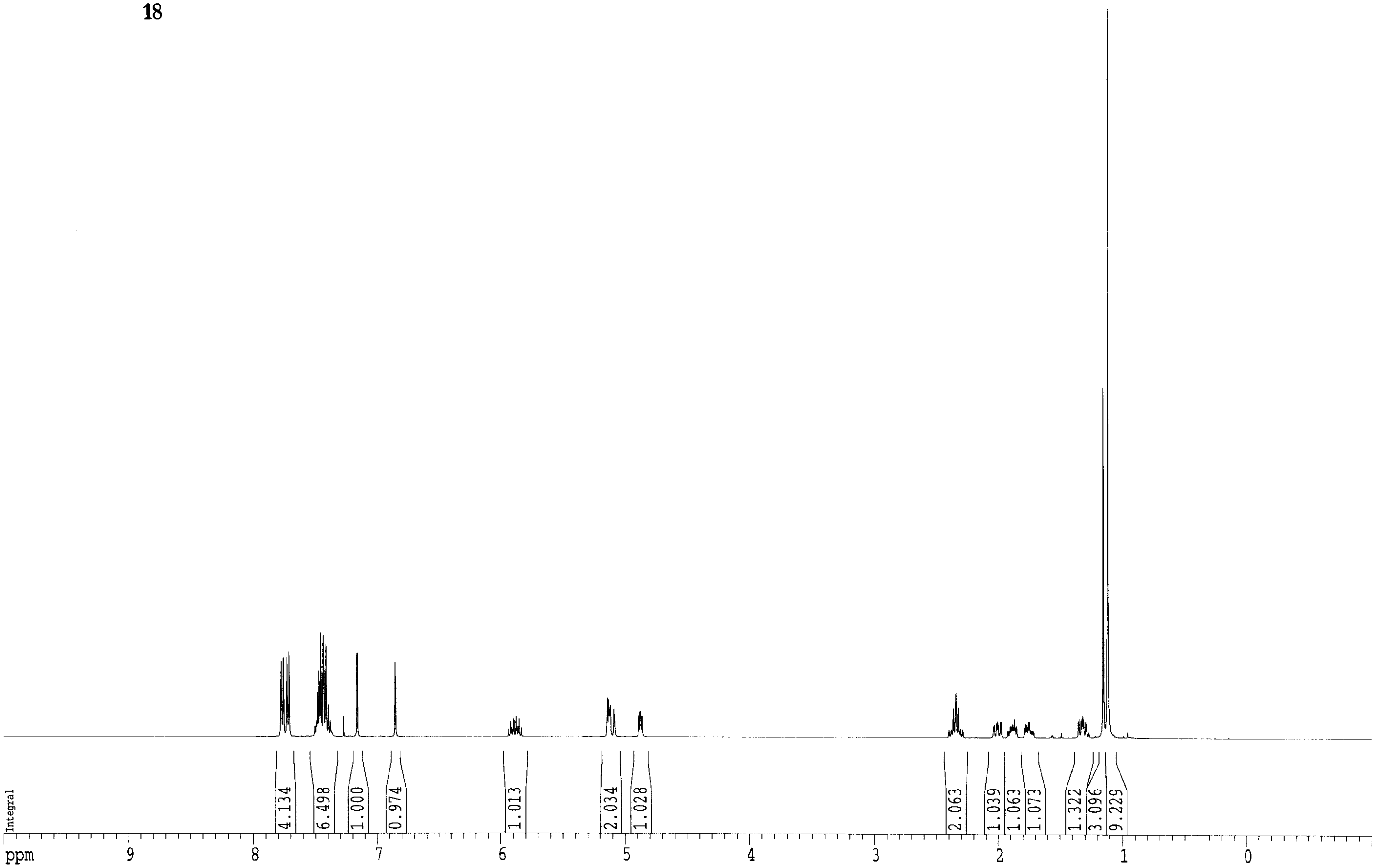
0.983
1.027
1.967
1.059
2.185
1.082
3.222
9.456
9
8
7
6
5
4
3
2
1
0
ppm

17


Me
TBDPSO
O

18


Integral
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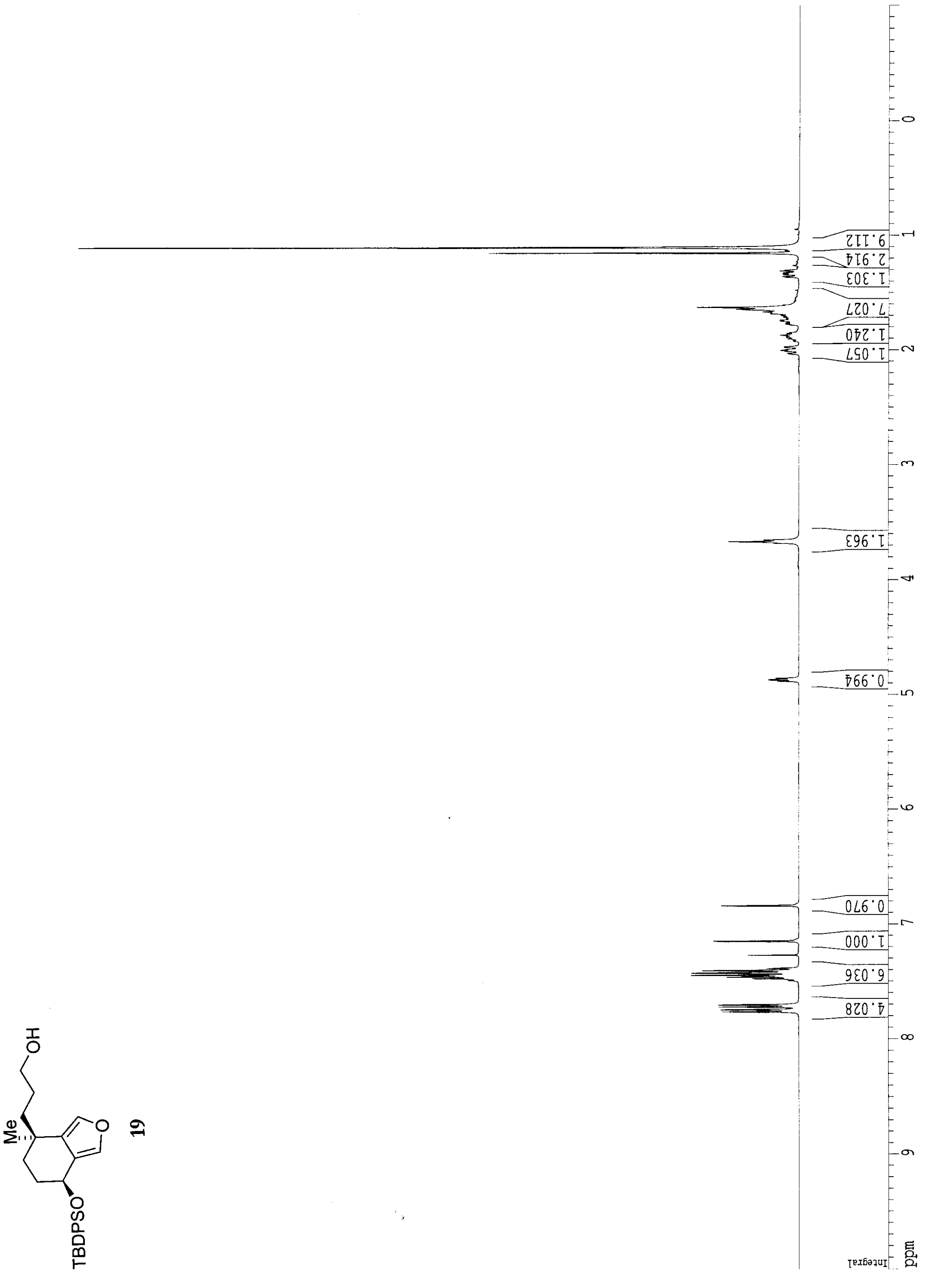
6.498
1.000
0.974
1.013
2.034
1.028
2.063
1.039
1.063
1.073
1.322
3.096
9.229
ppm
9
8
7
6
5
4
3
2
1
0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OH
O
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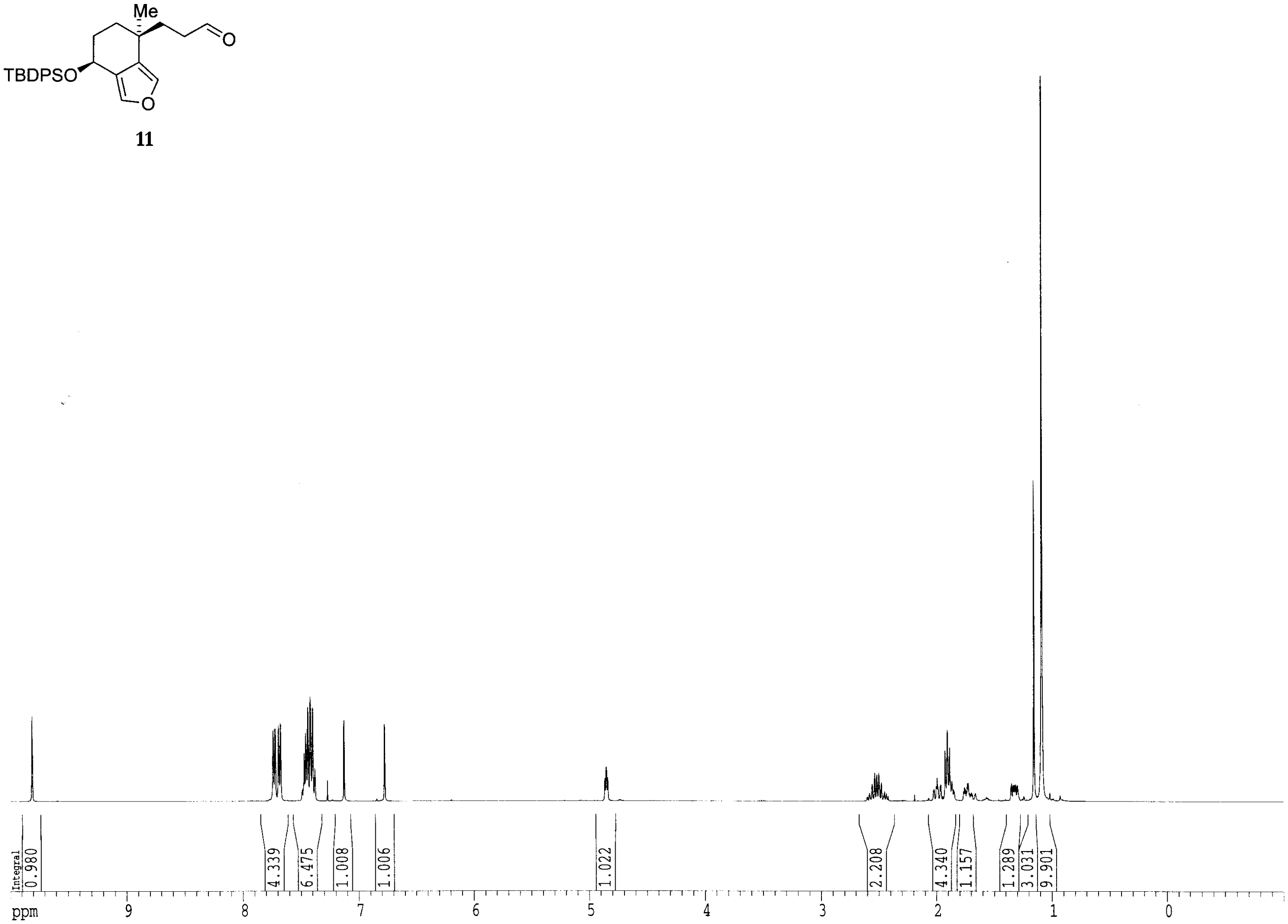
1.303
7.027
1.240
1.057
1.963
0.994
0.970
1.000
6.036
4.028
Integral
0
1
2
3
4
5
6
7
8
9
ppm

19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TBDPSO
O
O
Integral
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1.006
1.022
2.208
4.340
1.157
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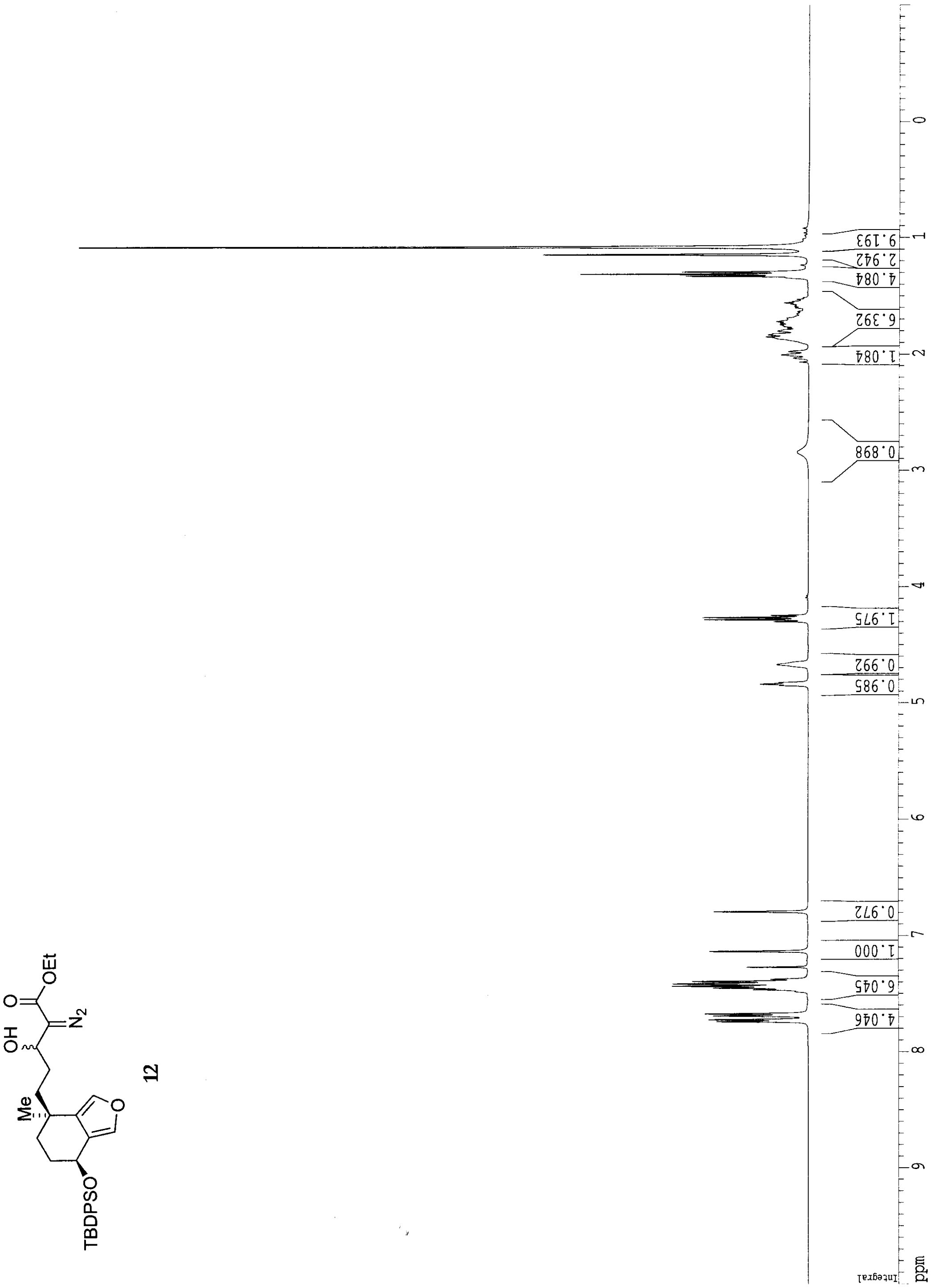
3.031
9.901
ppm
9
8
7
6
5
4
3
2
1
0

11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Me
OH
O
OEt
N$_2$
9.193
2.942
4.084
6.392
1.084
0.898
1.975
0.992
0.985
0.972
1.000
6.045
4.046
Integral
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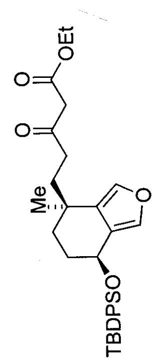
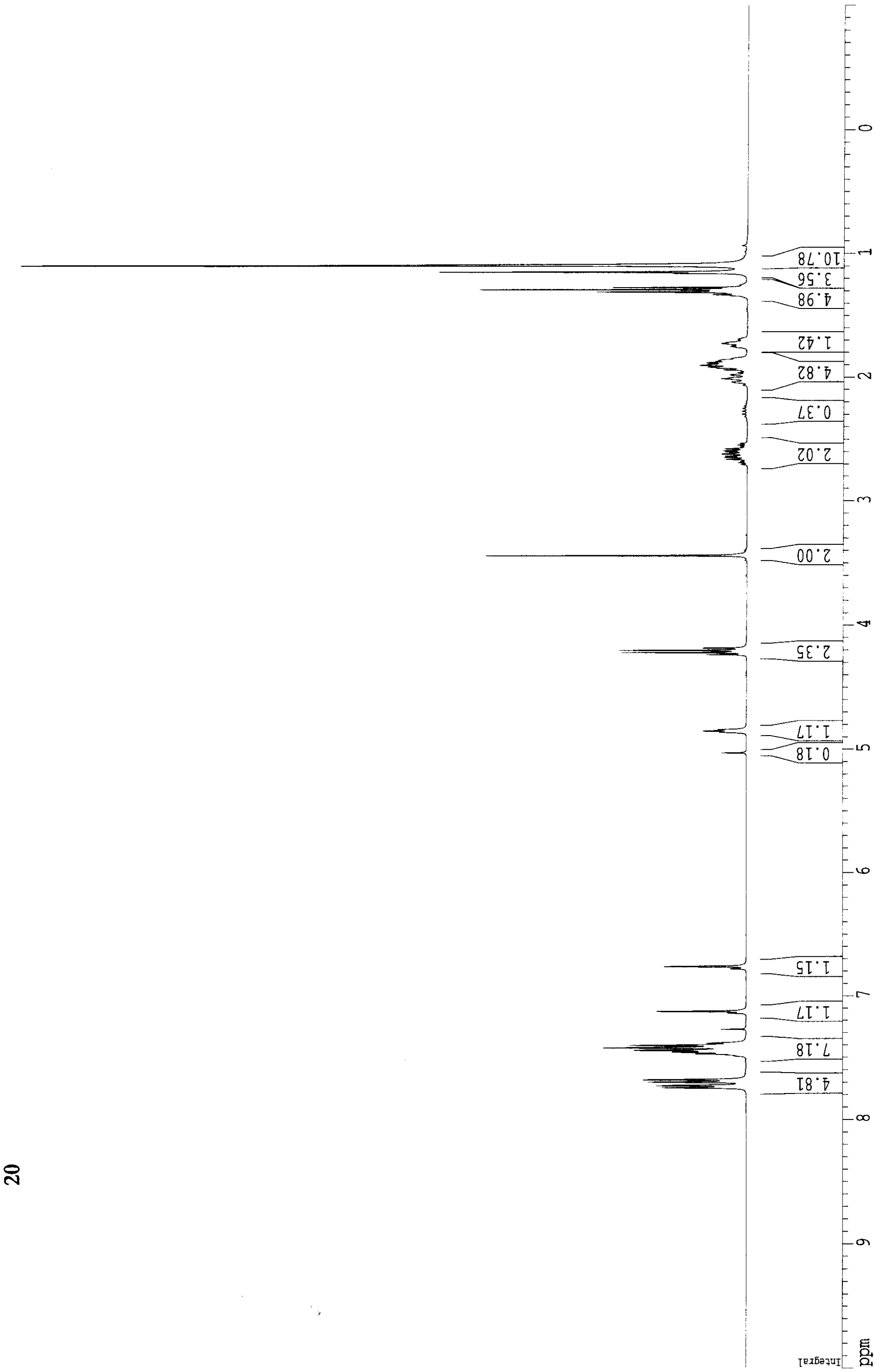
1
2
3
4
5
6
7
8
9
ppm

12

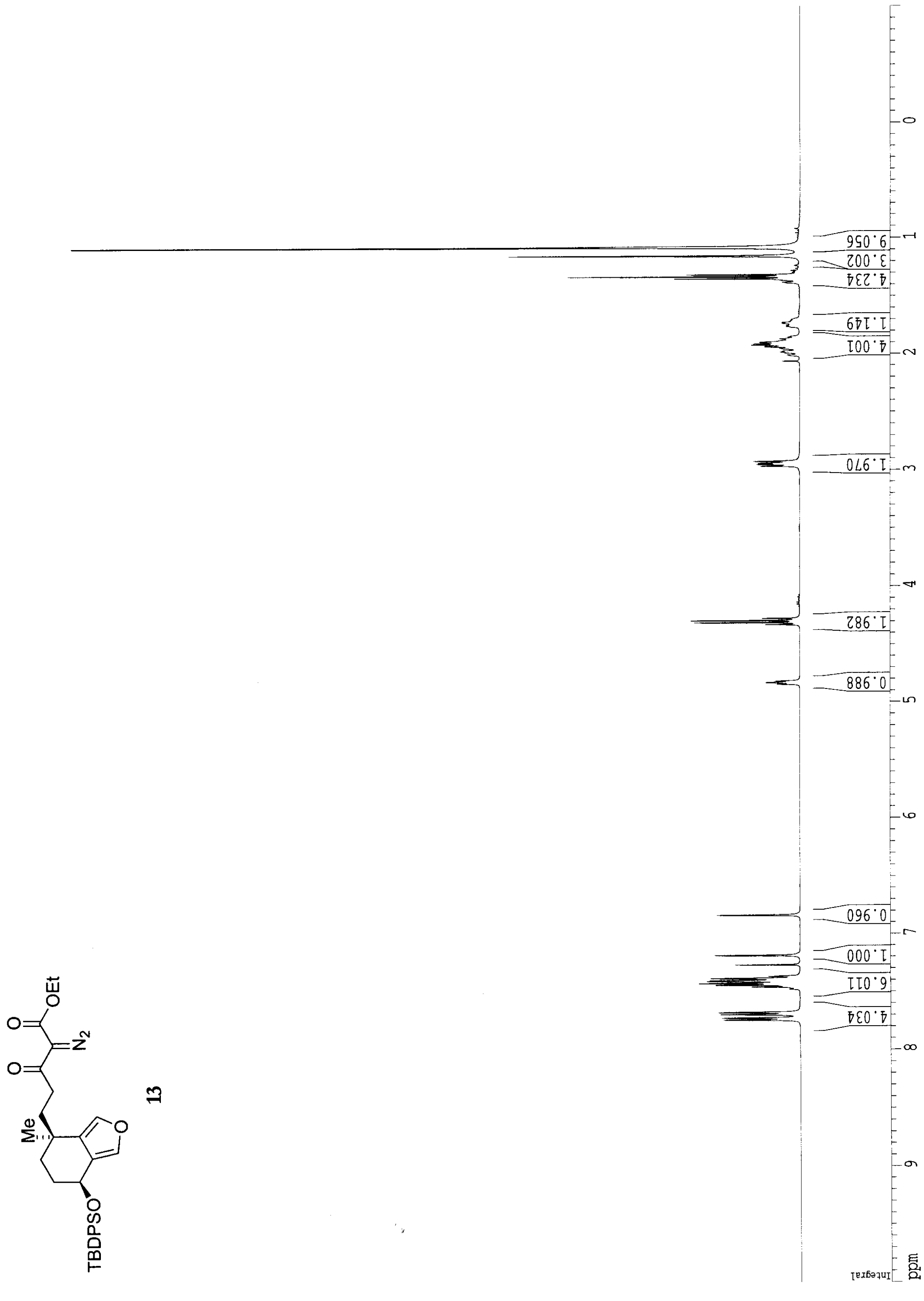


TBDPSO
Me
O
O
O
OEt

20



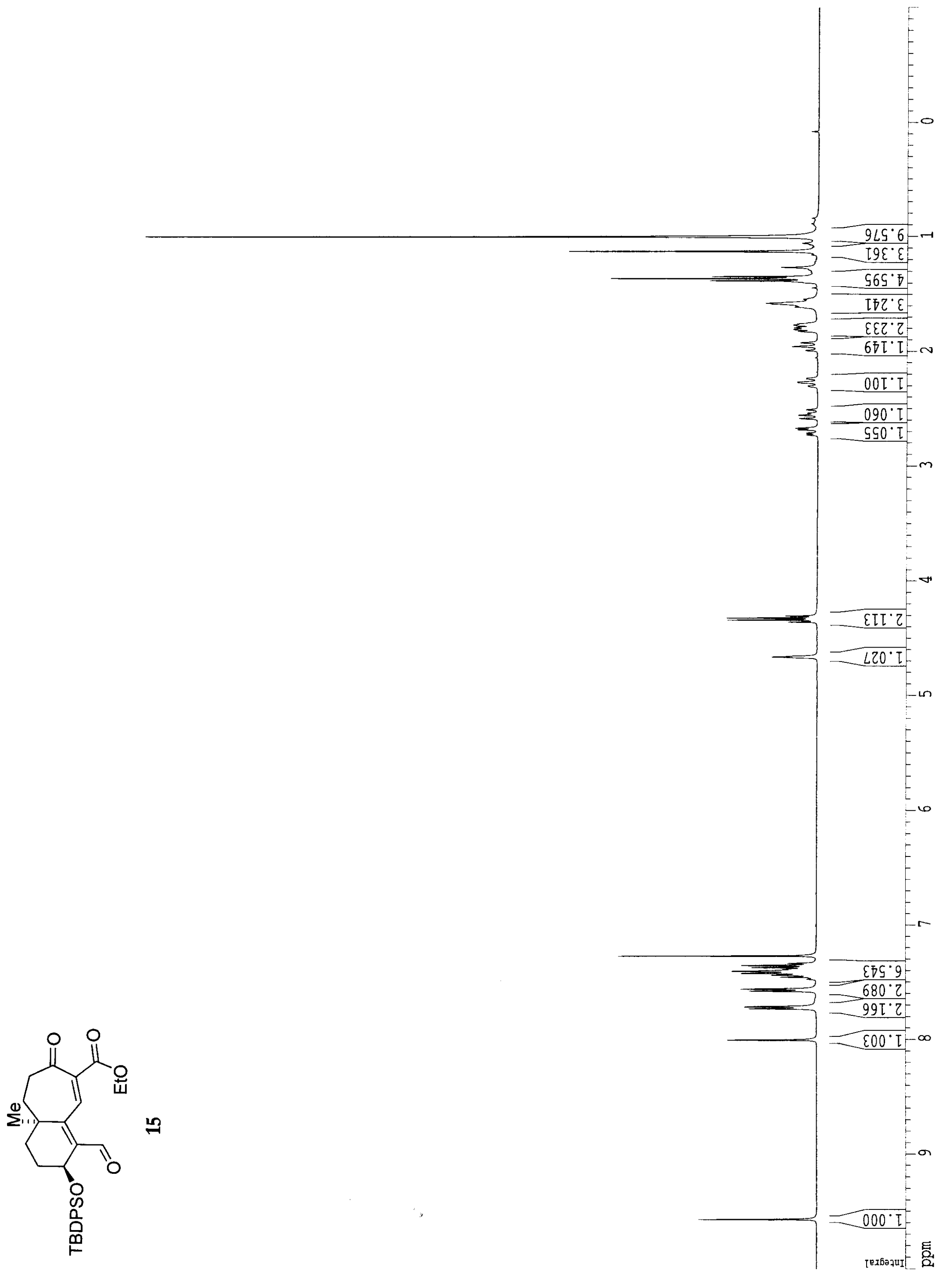
10.78
3.56
4.98
1.42
4.82
0.37
2.02
2.00
2.35
1.17
0.18
1.15
1.17
7.18
4.81
Integral
0
1
2
3
4
5
6
7
8
9
ppm



TBDPSO
Me
O
O
O
N2
OEt

13

9.056
3.002
4.234
1.149
4.001
1.970
1.982
0.988
0.960
1.000
6.011
4.034
Integral
0
1
2
3
4
5
6
7
8
9
ppm


TBDPSO
Me
O
O
O
EtO
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3.361
4.595
3.241
2.233
1.149
1.100
1.060
1.055
2.113
1.027
6.543
2.089
2.166
1.003
1.000
Integral
ppm
0
1
2
3
4
5
6
7
8
9

15

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{11}O_3H_{16}$
Formula Weight	196.25
Crystal Color, Habit	colorless, blocklike
Crystal Dimensions	0.10 X 0.18 X 0.19 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Lattice Parameters	a = 7.898(1)Å b = 14.181(2) Å c = 17.856(2) Å V = 1999.8(4) $Å^3$
Space Group	Pbca (#61)
Z value	8
D_{calc}	1.304 g/cm^3
F_{000}	848.00
μ(MoKα)	0.93 cm^{-1}

B. Intensity Measurements

Diffractometer	Bruker SMART CCD
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Detector Position	60.00 mm
Exposure Time	10.0 seconds per frame.
Scan Type	ω (0.3 degrees per frame)
$2\theta_{max}$	49.4°
No. of Reflections Measured	Total: 8066 Unique: 1958 (R_{int} = 0.065)
Corrections	Lorentz-polarization Absorption (Tmax = 1.00 Tmin = 0.62)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\lvert Fo\rvert - \lvert Fc\rvert)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0300
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	1105

No. Variables	133
Reflection/Parameter Ratio	8.31
Residuals: R; Rw; Rall	0.044 ; 0.050; 0.078
Goodness of Fit Indicator	1.74
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.22 $e^-/\text{Å}^3$
Minimum peak in Final Diff. Map	-0.23 $e^-/\text{Å}^3$