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Supporting Information

First Total Synthesis of (-)-AL-2

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Melting points are uncorrected. IR spectra were measured in $CHCl_3$. ^1H NMR spectra were taken in $CDCl_3$. $CHCl_3$ (7.26 ppm) was used as an internal standard for silyl compounds. TMS was employed as an internal standard for other compounds. ^{13}C NMR spectra were recorded in $CDCl_3$ with $CHCl_3$ (77.00 ppm) as an internal standard. Commercially available dry CH_2Cl_2 and THF were employed for reactions. Et_3N was distilled from CaH_2 prior to use. All reactions were carried out under nitrogen atmosphere otherwise stated. Silica gel (silica gel 60, 230-400 mesh, Merck) was used for chromatography. Organic extracts were dried over anhydrous Na_2SO_4.

Compound (-)-11: A solution of PivCl (1.19 mL, 9.70 mmol) in CH_2Cl_2 (12 mL) was added to a solution of **10** (3.00 g, 9.20 mmol), Et_3N (3.67 mL, 27.6 mmol) and DMAP (0.11 g, 0.92 mmol) in CH_2Cl_2 (80 mL) at –78 °C over a period of 3 h. After being stirred for 16 h at the same temperature, the reaction mixture was quenched by addition of water and extracted with CH_2Cl_2. The extract was washed with water and brine, dried, and concentrated to dryness. The residue was passed through a short pad of silica gel with hexane-AcOEt (1:1) to afford the crude secondary alcohol. To a solution of the crude alcohol and imidazole (1.88 g, 27.6 mmol) in DMF (4.5 mL) was added TBDPSCl (3.56 mL, 13.8 mmol) at room temperature. The reaction mixture was stirred at 50 °C for 42 h, quenched by addition of water, and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. The residue was passed through a short pad of silica gel with hexane-AcOEt (1:1) to afford the crudeTBDPS-protected product. To a solution of the crude product in Et_2O (90 mL) was added EtMgBr in Et_2O (1.00 M, 92.0 mL, 92.0 mmol) at room temperature, and the reaction mixture was stirred for 22 h, quenched by addition of water, and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (10:1) afforded (-)-**11** (3.94 g, 76%) as a colorless oil: $[\alpha]^{27}_{D}$ –18.0 (*c* 1.00, $CHCl_3$); IR 3612 cm^{-1}; ^1H NMR δ 7.68-7.64 (m, 4H), 7.43-7.32 (m, 6H), 7.28-7.16 (m, 5H), 4.51, 4.36 (AB-q, 2H, *J* =11.9 Hz), 3.95-3.83 (m, 3H), 3.62-3.57 (m, 2H), 3.49 (m, 1H), 1.06 (s, 9H), 0.89 (s, 9H), 0.05 (s, 6H); ^{13}C NMR δ 138.45, 135.87, 135.74, 133.71, 133.32, 129.83, 129.19, 127.69, 127.64, 127.44, 81.83, 72.65, 72.56, 63.36, 62.30, 26.99, 19.30, 18.21, -5.44, -5.48; MS *m/z* 564 (M^+, 1.1). HRMS calcd for $C_{33}H_{48}O_4Si_2$ 564.3091, found 564.3095.

Compound (-)-12: To a solution of (-)-**11** (400 mg, 0.71mmol) in CH_2Cl_2 (7.0 mL) was added Dess-Martin periodinane (470 mg, 1.01 mmol) at 0°C. The reaction mixture was stirred at room temperature for 1 h, poured into saturated aqueous solution of $Na_2S_2O_3$ and $NaHCO_3$ (1:1), and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. The crude aldehyde was used directly for the next reaction. To a solution of PPh_3 (0.81 g, 3.12 mmol) in CH_2Cl_2 (4.0 mL) was added CBr_4 (0.51 g, 1.56 mmol) at 0°C, and the reaction mixture was stirred for 30 min. A solution of crude aldehyde in CH_2Cl_2 (3.0 mL) was then added to a solution of the ylide in CH_2Cl_2 solution, thus prepared, at 0℃ and stirring was continued for 5 min at the same temperature. The reaction mixture was quenched by addition of saturated aqueous $NaHCO_3$ and the CH_2Cl_2 solution was washed with water and brine, dried, and concentrated to dryness. The residue was passed through a short pad of silica gel with hexane-AcOEt (5:1) to give the crude dibromoolefin derivative. To a solution of the dibromoolefin derivative in THF (10 mL) was added *n*-BuLi in hexane (1.36 M, 4.40 mL, 5.98 mmol) at –78 °C, and the reaction mixture was stirred for 10 min at the same temperature. The reaction mixture was quenched by addition of water and extracted with Et_2O, which was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (10:1)) afforded (-)-**12** (297 mg, 72%) as a colorless oil: $[\alpha]^{25}_{D}$ –0.5 (*c* 1.00, $CHCl_3$); IR 3306, 2120 cm^{-1}; ^1H NMR 7.74-7.71 (m, 2H), 7.66-7.64 (m, 2H), 7.44-7.30 (m, 6H), 7.27-7.20 (m, 5H), 4.54 (s, 2H), 4.51 (dd, 1H, *J* = 4.9, 2.0 Hz), 4.06 (dd, 1H, *J* = 10.7, 2.6 Hz), 3.88 (dd, 1H, *J* = 10.7, 7.3 Hz), 3.48 (ddd, 1H, *J* = 7.3, 4.9, 2.6 Hz), 2.27 (d, 1H, *J* = 2.0 Hz), 1.07 (s, 9H), 0.95 (s, 9H), 0.04 (s, 6H); ^{13}C NMR δ 138.62, 136.06, 135.83, 133.08, 129.83, 129.72, 128.10, 127.66, 127.62, 127.42, 127.30, 82.34, 82.18, 74.05, 72.89, 63.52, 63.36, 31.57, 26.87, 25.99, 25.90, 22.64, 19.27, 18.24, 14.13, 9.40, -5.32, -5.41; FABMS *m/z* 581 (M^{+}+1, 1.1). Anal. Calcd for $C_{34}H_{46}O_{3}Si_{2}$: C, 73.07; H, 8.30. Found: C, 72.71, H, 8.57.

Compound (-)-13: PPTS (570 mg, 2.27 mmol) was added to a solution of (-)-**12** (1.15 g, 2.06 mmol) in MeOH (10 mL) at room temperature, and the reaction mixture was stirred for 24 h. MeOH was evaporated off and the residue was taken up in AcOEt, which was washed with saturated aqueous $NaHCO_3$, water, and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (5:1) afforded (-)-**13** (683 mg, 75%) as a colorless oil: $[\alpha]^{27}_{D}$ −5.7 (*c* 1.00, $CHCl_3$); IR 3587, 3306, 2120 cm^{-1}; ^{1}H NMR 7.75-7.71 (m, 2H), 7.69-7.65 (m, 2H), 7.47-7.33 (m, 6H), 7.27-7.14 (m, 5H), 4.56 (dd, 1H, *J* = 5.3, 2.0 Hz), 4.47, 4.30 (AB-q, 2H, *J* =11.9 Hz), 4.01-3.82 (m, 2H), 3.52-3.47 (m, 1H), 2.37 (d, 1H, *J* = 1.7 Hz), 1.09 (s, 9H); ^{13}C NMR δ 137.77, 136.05, 135.81, 132.83, 132.61, 130.03, 129.92, 128.41, 127.99, 127.82, 127.76127.55, 81.53, 80.97, 74.97, 72.72, 63.72, 61.85, 26.85, 19.21; FABMS *m/z* 445 (M^{+}+1, 1.1). Anal. Calcd for $C_{28}H_{32}O_{3}Si$: C, 75.63; H, 7.25. Found: C, 75.55, H, 7.26.

Compound (+)-14: To a solution of (-)-**13** (100 mg, 0.22 mmol) in CH_2Cl_2 (2.2 mL) was added Dess-Martin periodinane (146 mg, 0.33 mmol) at 0°C. The reaction mixture was stirred at room temperature for 1 h, poured into saturated aqueous solution of $Na_2S_2O_3$ and $NaHCO_3$(1:1), and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. The crude aldehyde was used directly for the next reaction. A solution of Grignard reagent in Et_2O was prepared as follows; To a suspension of magnesium powder (440 mg, 18.3 mmol) in Et_2O (4.0 mL) was added 4-*tert*-Butyldimethylsioxybutyl iodide (2.00 g, 6.37 mmol) in Et_2O (8.00 mL). The mixture was stirred vigorously at room temperature for 30 min. Grignard reagent (0.50 M, 1.32 mL, 0.66 mmol), thus prepared, was added to a solution of crude aldehyde in CH_2Cl_2 (3.0 mL) at -78°C. After being stirred for 20 min, the reaction mixture was quenched by addition of saturated aqueous NH_4Cl and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. To a solution of the residue in CH_2Cl_2 (4.0 mL) was added Dess-Martin periodinone (146 mg, 0.33 mmol) at 0°C. The reaction mixture was stirred at room temperature for 1 h, poured into saturated aqueous solution of $Na_2S_2O_3$

and $NaHCO_3$(1:1), and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (30:1) afforded (+)-**14** (102 mg, 74%) as a colorless oil: $[\alpha]^{28}_{D}$ +33.6 (*c* 1.00, $CHCl_3$); IR 3306, 2122, 1716 cm^{-1}; ^1H NMR δ 7.75-7.66 (m, 4H), 7.43-7.24 (m, 11H), 4.71 (dd, 1H, *J*=4.9, 2.4), 4.58, 4.56 (AB-q, 2H, *J*=11.7), 3.89 (d, 1H, *J*=4.9), 3.57 (t, 2H, *J*=6.8), 2.66 (m, 1H), 2.57 (m, 1H), 2.29 (d, 1H, *J*=2.4) 1.61-1.45 (m, 4H), 1.04 (s, 9H), 0.88 (s, 9H), 0.03 (s, 6H); ^{13}C NMR δ 208.84, 137.25, 136.10, 135.87, 132.87, 132.67, 129.88, 129.70, 128.34, 127.92, 127.85, 127.60, 127.33, 86.72, 81.12, 75.78, 73.41, 64.64, 62.88, 39.95, 32.28, 26.78, 25.93, 19.25, 18.30, -5.32; MS *m/z* 628 (M^++1, 1.4). Anal. Calcd for $C_{38}H_{52}O_4Si_2$: C, 72.56; H, 8.33. Found: C, 72.20, H, 8.53.

Compound (-)-15: *p*-TsOH was added to a solution of (+)-**14** (61.0 mg, 0.10 mmol) in THF:H_2O (20:1, 3.0 mL) at room temperature. The reaction mixture was stirred at the same temperature for 4h, quenched by addition of saturated aqueous $NaHCO_3$, and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. The residue was passed through a short pad of silica gel with hexane-AcOEt (1:1) to afford the crude alcohol. A solution of $Pd_2(dba)_3{\cdot}CHCl_3$ (2.5 mg, 0.002 mmol) in MeOH (3.5 mL) was stirred under a CO atmosphere at room temperature for 30 min, to which a solution of the crude alcohol and benzoquinone (217 mg, 1.94 mmol) in MeOH (1.5 mL) was added. After stirring for 48 h, MeOH was evaporated off, and the residue was taken up in Et_2O, which was successively washed with saturated aqueous NaS_2O_3, saturated aqueous $NaHCO_3$, water, brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (30:1) afforded (-)-**15** (23.0 mg, 41%) as a colorless oil: $[\alpha]^{26}_{D}$ −75.0 (*c* 1.00, $CHCl_3$); IR 1707, 1659 cm^{-1}; ^1H NMR 7.95-7.93 (m, 2H), 7.69-7.66 (m, 2H), 7.49-7.32 (m, 6H), 7.24-7.19 (m, 3H), 6.97-6.95 (m, 2H), 5.42(s, 1H), 5.39 (s, 1H), 3.98 (m, 1H), 3.77 (m, 1H), 3.70 (s, 2H), 3.61 (s, 1H), 3.31 (s, 3H), 1.95-1.56 (m, 6H), 1.03 (s, 9H); ^{13}C NMR δ 173.64, 166.95, 137.63, 136.62, 135.99, 133.92, 133.29, 129.89, 129.39, 128.10, 127.68, 127.40, 127.15, 127.02, 110.77, 93.71, 86.05, 73.56, 70.95, 62.39, 50.60, 28.73, 26.72, 24.77, 19.37, 19.10; FABMS *m/z* 573 (M^++1, 1.1). FABHRMS calcd for $C_{34}H_{41}O_6Si$ 573.2672, found 573.2640.

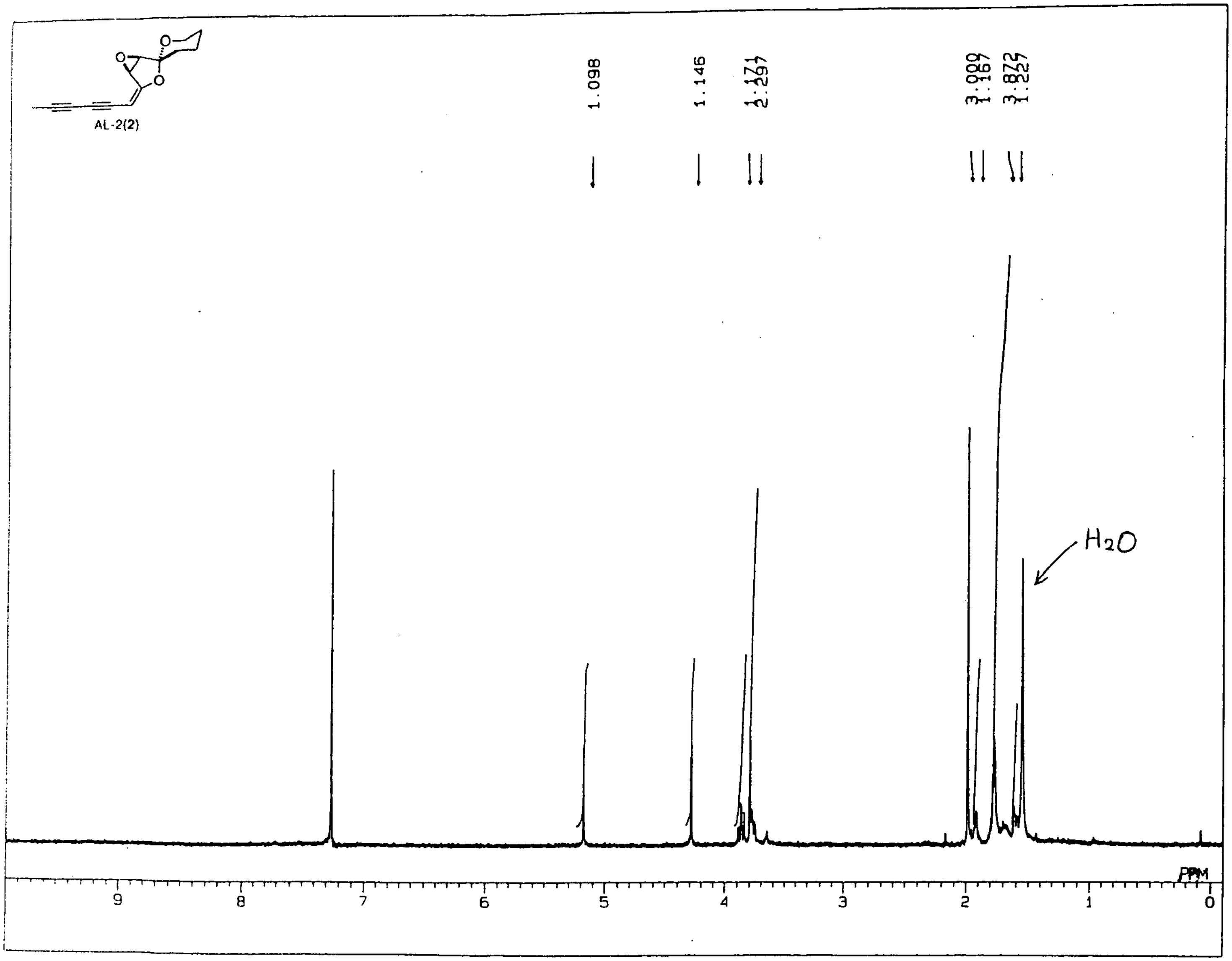
Compound (-)-19: A solution of DIBAL-H in hexane (1.00 M, 0.50 mL, 0.50 mmol) was added to a solution of (-)-**15** (79.0 mg, 0.14 mmol) in CH_2Cl_2 (3.0 mL) at –78°C. The mixture was stirred for 30 min, quenched by addition of saturated aqueous Na_2SO_4, and filtered off. The filtrate was concentrated to leave the residual oil, which was passed through a short pad of silica gel with hexane-AcOEt (2:1) to afford the crude alcohol. A solution of lithium *tert*-butylbiphenylide in THF was prepared as follows: Lithium (20.0 mg, 2.85 mmol) was added to a solution of *p,p'*-di-*tert*-butylbiphenyl (620 mg, 2.38 mmol) in THF (14 mL) at room temperature. The mixture was stirred vigorously at room temperature until dark green radical anion was developed, at which time the reaction mixture was cooled in ice bath. Stirring was continued at 0 °C for 4h. Thus prepared solution of LiDBB in THF was added to a solution of the crude alcohol in THF (3.0 mL) at –78 °C until the reaction mixture was turned to deep green. After being stirred for 10 min at –78 °C, the reaction mixture was quenched by addition of water and extracted with AcOEt. The extract was washed with brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (1:1) afforded (-)-**19** (45.0 mg, 72%) as colorless needles: mp. 178.5-179.5 °C (hexane- AcOEt); $[\alpha]^{24}_{D}$ –71.6 (*c* 0.74, THF); IR 3311, 3177, 1699 cm^{-1}; ^{1}H NMR 7.80-7.74 (4H, m) 7.48-7.41 (m, 6H), 5.25 (dt, 1H, *J* = 1.3, 7.9 Hz), 4.58 (br-s, 1H), 4.14-3.68(m, 5H), 1.85-1.60(m, 6H), 1.06 (s, 9H).; ^{13}C NMR d 158.51, 136.03, 135.97, 133.27, 132.86, 130.22, 130.04, 127.97, 127.80, 107.30, 101.96, 80.87, 62.21, 58.56, 28.19, 26.78, 24.89, 19.36, 19.00; MS *m/z* 454 (M$^+$, 0.1). HRMS calcd for $C_{26}H_{34}O_5Si$ 454.2175, found 454.2174.

Compound (-)-22: A suspension of (-)-**19** (28.0 mg, 0.62 x 10^{-1} mmol) and chemical manganese dioxide[21] (87.0mg, 1.23 mmol) in CH_2Cl_2 (5.0 mL) was stirred at room temperature for 1 h. The mixture was filtered off and the filtrate was concentrated to leave the crude aldehyde. To a solution of TMSCLi(N_2) in THF (1.0 mL), prepared from reaction of $TMSCHN_2$ in hexane (1.50 M, 1.00 mL, 1.50 mmol) and *n*-BuLi in hexane (1.43 M, 0.75 mL, 1.10 mmol) at –78 °C for 30 min, was added a solution of the crude aldehyde in THF (1.5 mL). The reaction mixture was stirred at –78°C for 30 min and then at 0°C for 30 min. The reaction mixture was quenched by addition of water and extracted with Et_2O, which was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (5:1) afforded (-)-**22** (23.3 mg, 84%) as a colorless oil: $[\alpha]^{25}_{D}$ –80.7 (*c* 0.44, $CHCl_3$); IR 3568, 3308, 2110, 1655 cm^{-1}; ^{1}H NMR d 7.82-7.78 (m, 4H), 7.46-7.37 (m, 6H), 5.18 (d, 1H, *J* = 3.0 Hz), 4.71 (s, 1H), 3.93 (dt, 1H, J = 3.4, 11.7 Hz), 3.75 (s, 1H), 3.73 (m, 1H), 2.77 (d, 1H J = 3.0 Hz), 1.82-1.48 (m, 6H), 1.10 (s, 9H);.^{13}C NMR d 168.82, 136.30, 136.14, 134.02, 133.03, 129.90, 129.81, 127.62, 127.58, 109.47, 82.80, 80.47, 80.31, 80.23, 78.76, 62.45, 28.09, 26.90, 24.73, 19.64, 18.87; MS *m/z* 448 (M^+, 3.4). HRMS calcd for $C_{27}H_{32}O_4Si$ 448.2070, found 448.2073.

Compound (+)-23: BzCl (0.05 mL, 0.42 mmol) was added to a solution of (-)-**22** (6.20 mg, 0.14 x 10^{-1} mmol) in pyridine (1.5 mL) at room temperature. After being stirred for 20 min, the reaction mixture was quenched by addition of water and extracted with Et_2O, which was washed with water and brine, dried, and concentrated to dryness. The residue was passed through a short pad of silica gel with hexane-AcOEt (30:1) to afford the crude benzoate. To a solution of crude benzoate in THF (6.0 mL) was added TBAF·$x$$H_2O$ (10.0 mg) and the reaction mixture was stirred for 4 h at room temperature, quenched by addition of water, and extracted with Et_2O. The extract was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (2: 1) afforded (+)-**23** (4.10 mg, 95%) as a colorless oil: $[\alpha]^{26}_D$ +38.4 (c 0.37, $CHCl_3$); IR 3308, 2108, 1728, 1657 cm^{-1}; ^{1}H NMR 8.03-7.99 (m, 2H), 7.62-7.57 (m, 1H), 7.48-7.42 (m, 2H), 5.30 (s, 1H), 5.22 (m, 1H), 4.80 (d, 1H, J = 9.9 Hz), 3.98-3.78 (m, 2H), 3.04-3.01 (m, 2H) 1.85-1.60(m, 6H); ^{13}C NMR δ 169.29, 164.89, 133.64, 129.87, 128.93, 128.55, 109.60, 83.85, 80.34, 79.64, 78.89, 74.54, 62.70, 27.60, 24.44, 18.53; MS *m/z* 314 (M^+, 2.8). HRMS calcd for $C_{18}H_{18}O_5$ 314.1154, found 314.1157.

Compound (+)-25: CuI (1.0 mg, 0.57 x 10^{-2} mmol) was added to a solution of (+)-**23** (5.9 mg, 0.19 x 10^{-1} mmol) and 1-propynyl iodide (0.20 x 10^{-1} mL, 0.19 mmol) in pyrrolidine (1.0 mL) at room temperature. After being stirred for 1.5 h, the reaction mixture was quenched by addition of water and extracted with Et_2O, which was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (3:1) afforded (+)-**25** (4.00 mg, 60%) as a colorless oil: $[\alpha]^{26}_D$ +183.0 (c 0.092, $CHCl_3$); IR 3547, 2142, 1728, 1649 cm^{-1}; ^{1}H NMR d 8.03-7.98 (m, 2H), 7.60 (m, 1H), 7.48-7.42 (m, 2H), 5.29 (s, 1H), 5.24 (d, 1H, J = 1.0 Hz), 4.76 (d, 1H, J = 9.9 Hz), 3.92 (dt, 1H, J = 3.3, 10.9 Hz), 3.80 (m, 1H), 2.98 (d, 1H, J = 9.9 Hz), 1.95 (d, 3H, J = 1.0 Hz), 1.92-1.58 (m, 6H); ^{13}C NMR 170.70, 164.89, 133.64, 129.86, 128.93, 128.55, 109.86, 84.14, 80.06, 79.68, 77.42, 74.60, 69.66, 67.95, 64.65, 62.77, 27.62, 24.43, 18.54, 4.58; MS *m/z* 352 (M^+, 14.5). HRMS calcd for $C_{21}H_{20}O_5$ 352.1311, found 352.1310.

(-)-AL-2 (2): MsCl (0.87 x 10^{-2} mL, 0.11 mmol) was added to a solution of (+)-**25** (4.00 mg, 0.11 x 10^{-1} mmol), Et_3N (0.28 x 10^{-1} mL, 0.20 mmol), and DMAP (0.80 mg, 0.67 x 10^{-2} mmol) in CH_2Cl_2 (0.5 mL) at 0℃. After being stirred for 5 min, the reaction mixture was quenched by addition of water and extracted with CH_2Cl_2, which was washed with water and brine, dried, and concentrated to dryness. The crude methanesulfonate was used directly for the next reaction. To a solution of the residue in MeOH (1.5 mL) was added K_2CO_3 (15.1 mg 0.11 mmol) and the reaction mixture was stirred for 1 h. MeOH was evaporated off and the residue was taken up in AcOEt, which was washed with water and brine, dried, and concentrated to dryness. Chromatography of the residue with hexane-AcOEt (10 : 1) afforded (-)-**2** (2.1 mg, 79%) as a colorless oil: $[\alpha]^{27}_{D}$ – 26.6 (*c* 0.14 $CHCl_3$) (lit.[2(b)] $[\alpha]^{20}_{D}$ – 14 (*c* 0.5, $CHCl_3$)); IR 2143, 1655 cm^{-1}; ^1H NMR 5.18 (s, 1H), 4.29 (d, 1H, *J* = 2.5 Hz), 3.87 (dt, 1H, *J* = 3.0, 11.2 Hz), 3.80 (d, 1H, *J* = 2.5 Hz), 3.77 (m, 1H), 1.99 (s, 1H), 1.93-1.90 (m, 1H), 1.79-1.58 (m, 5H); ^{13}C NMR 164.91, 105.91, 85.86, 79.61, 69.83, 63.22, 59.97, 51.92, 28.81, 24.76, 18.65, 4.62; MS *m/z* 230 (M^+, 13.8). HRMS calcd for $C_{14}H_{14}O_3$ 230.0943, found 230.0945.



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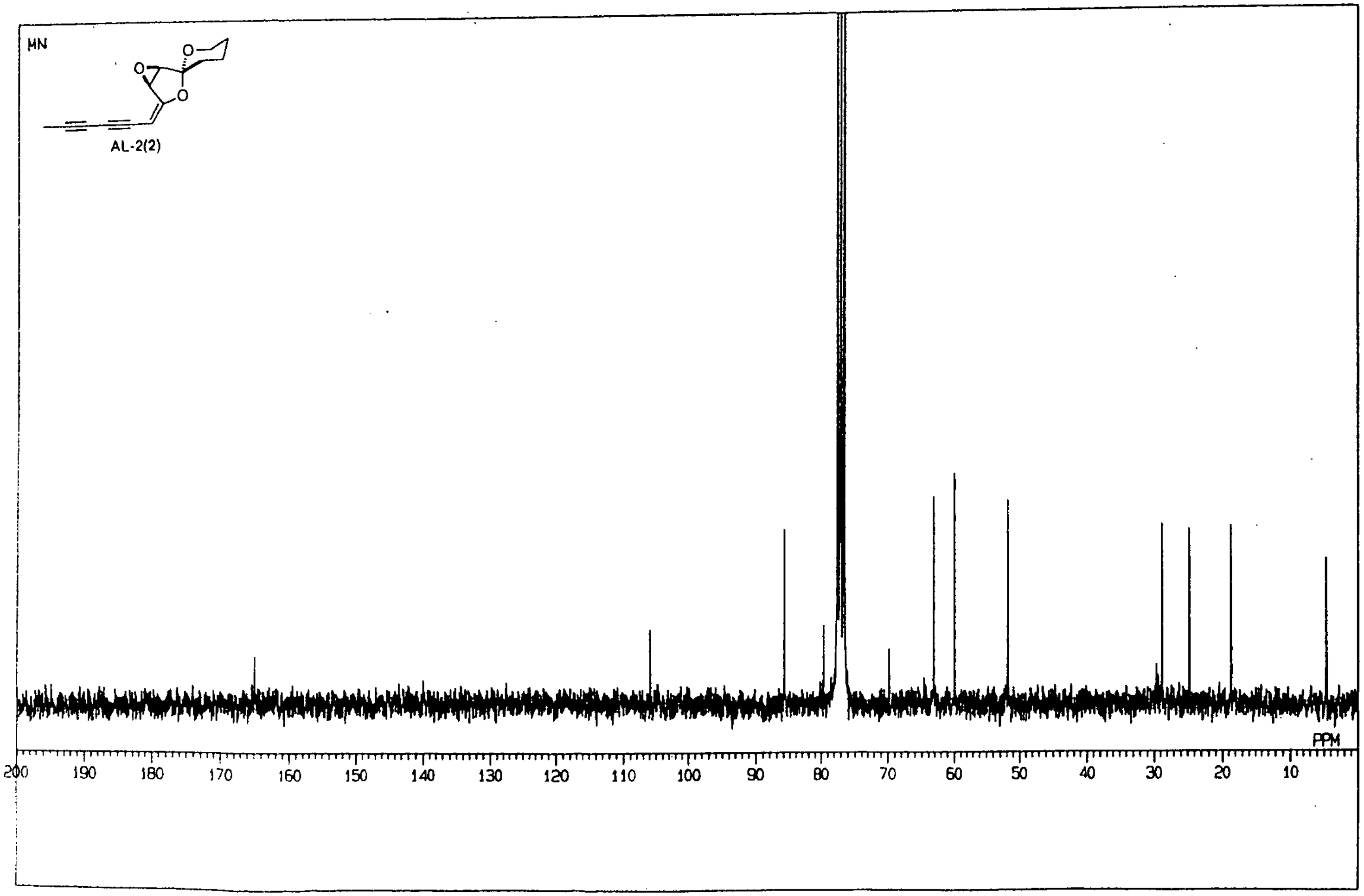

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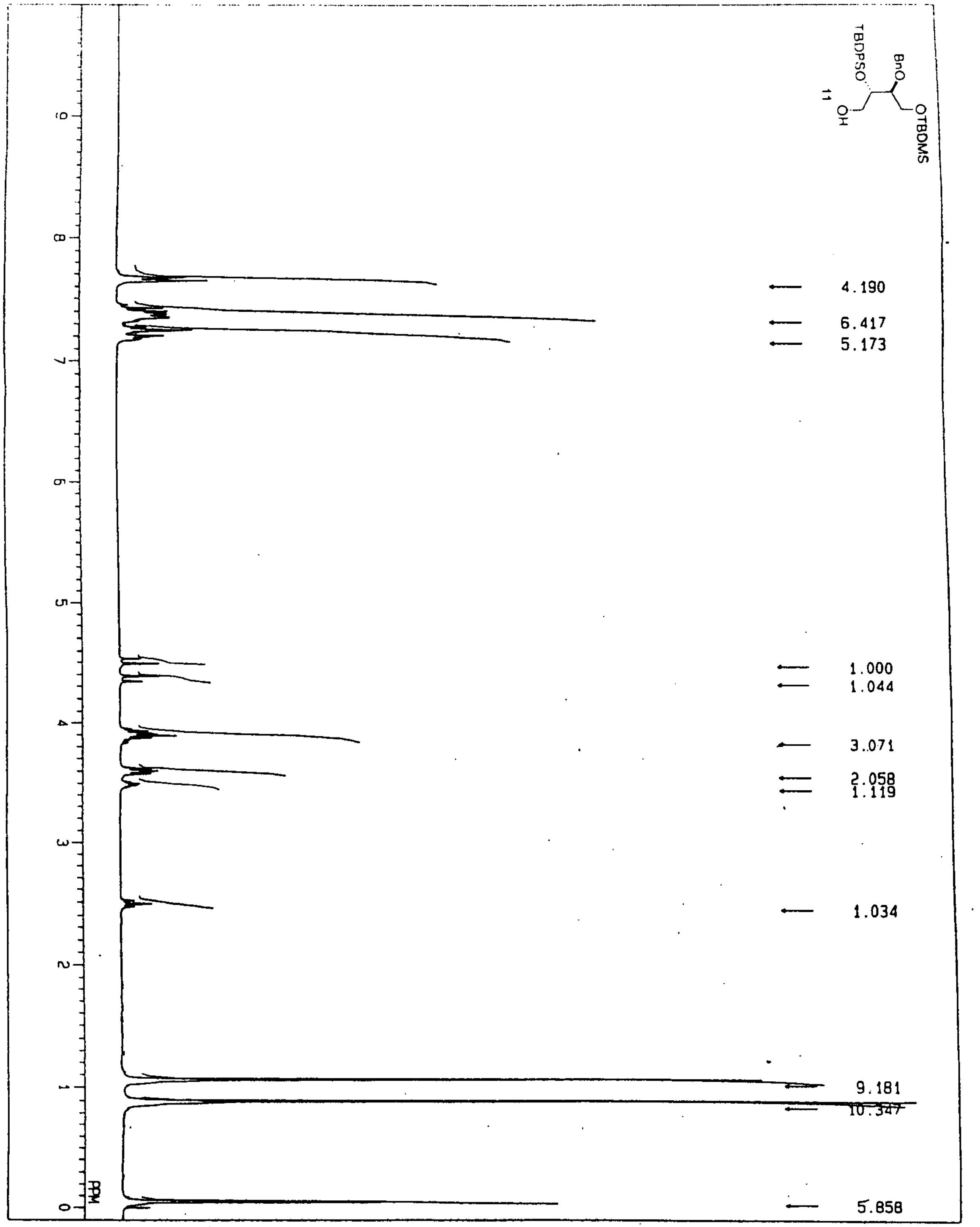

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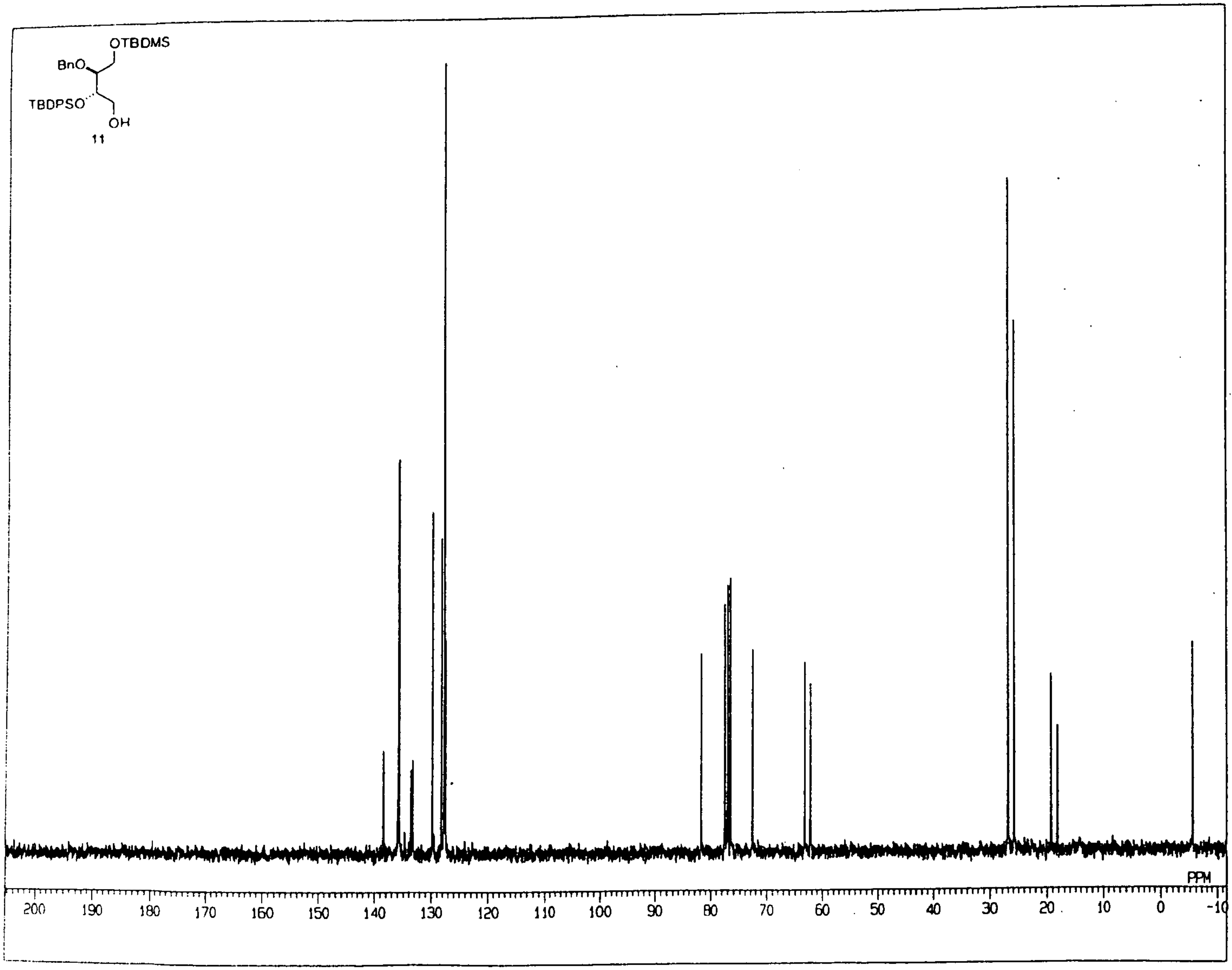

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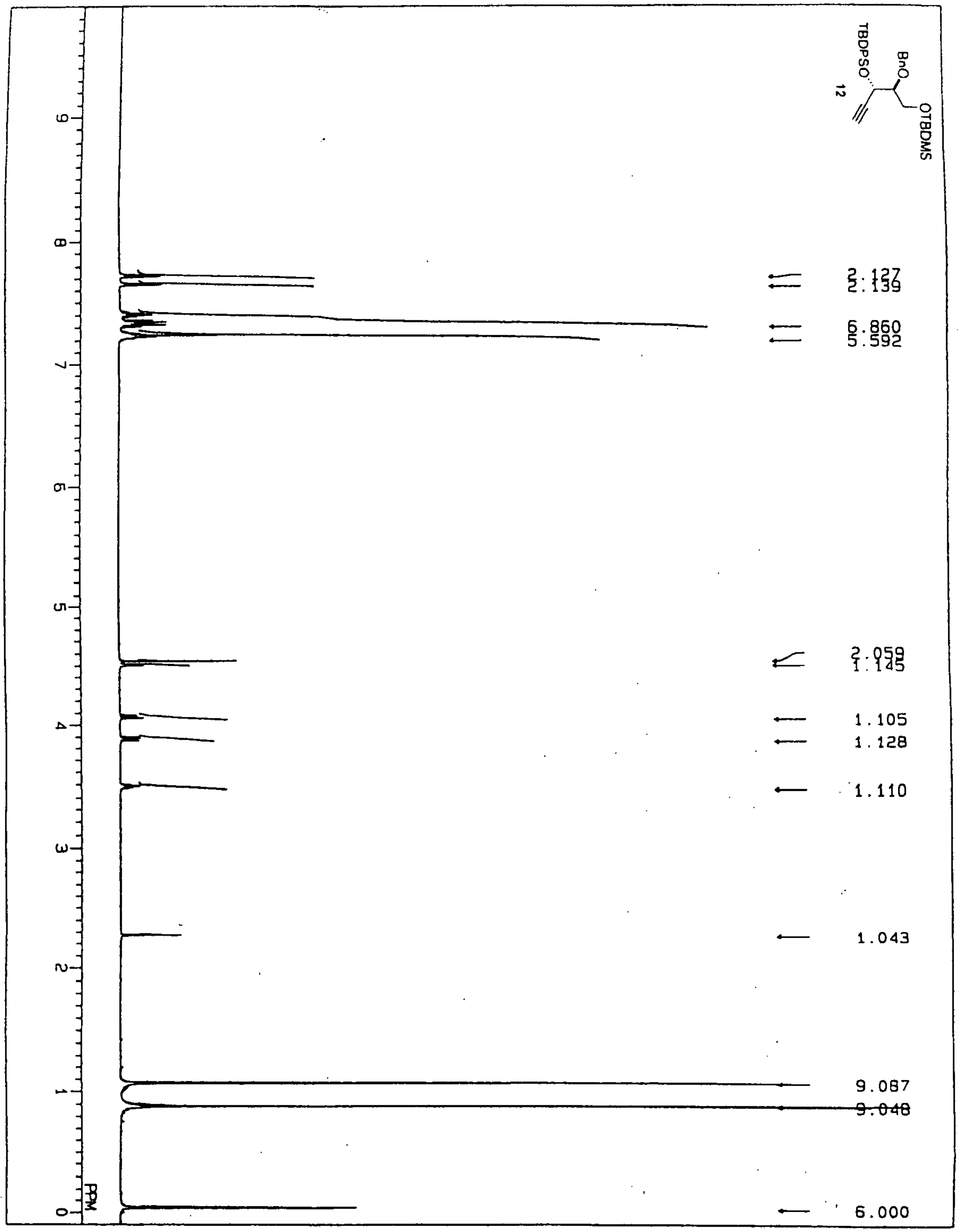

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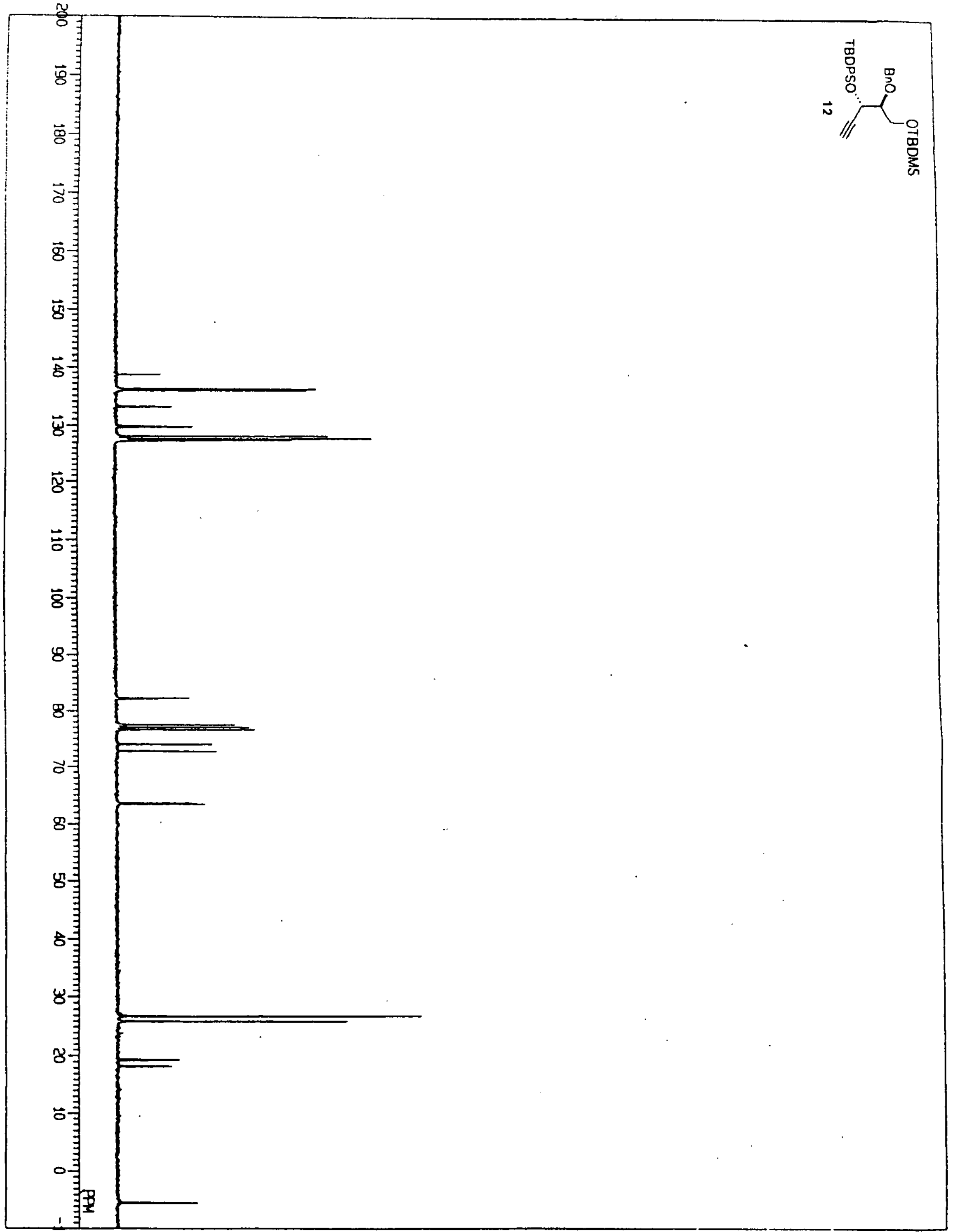
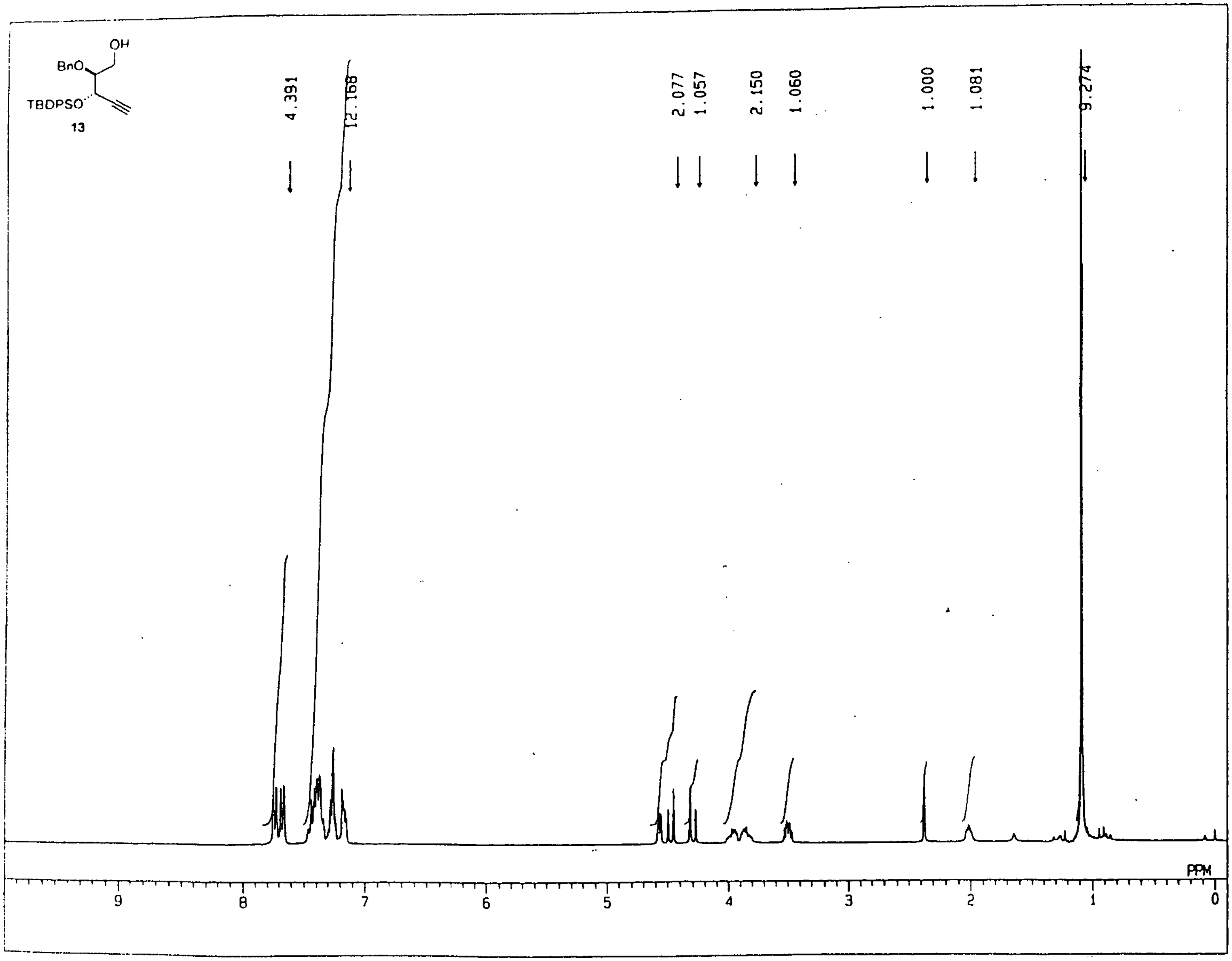
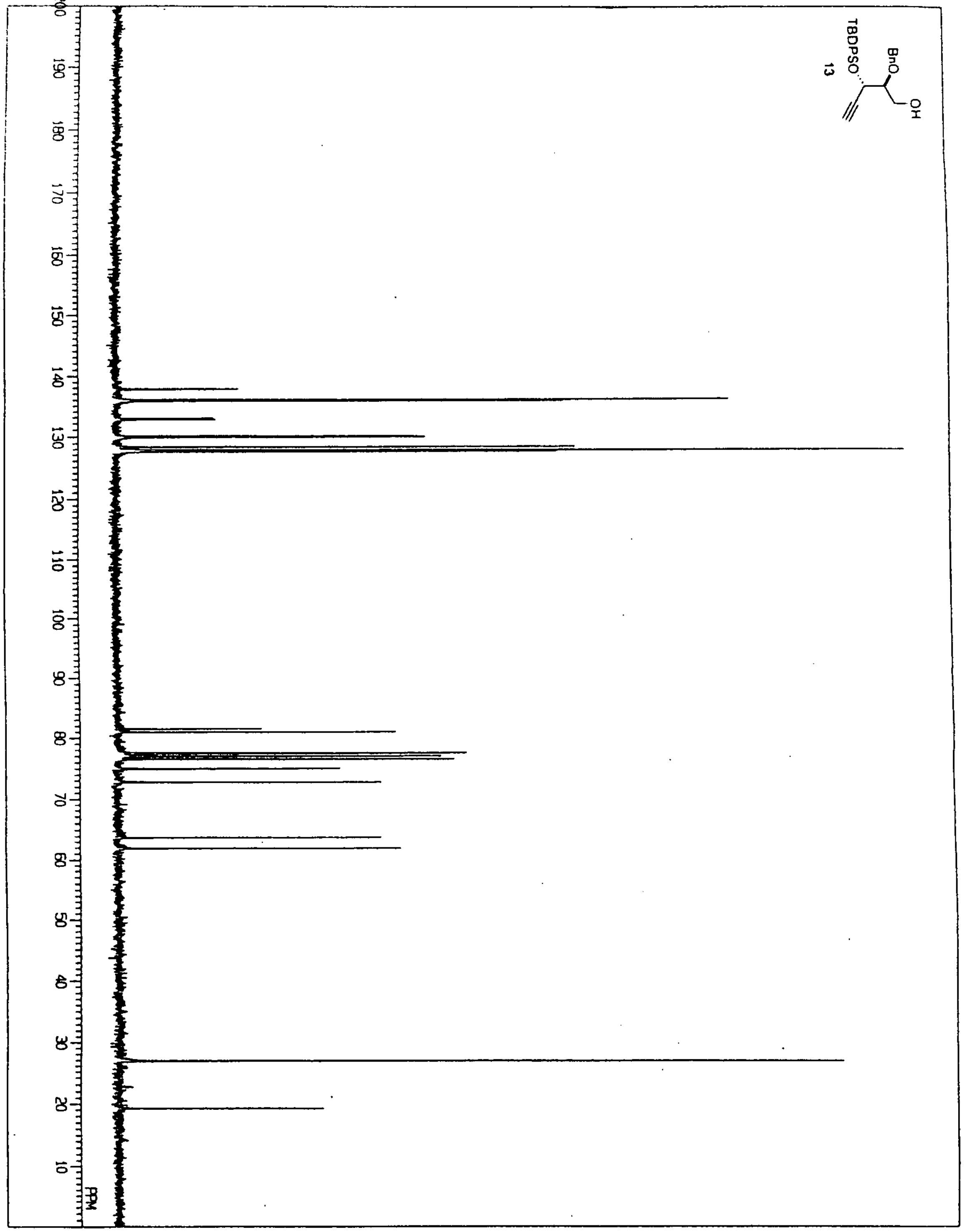
SLVNT CDCL3
EXREF       0.00 ppm
BF          0.10 Hz
RGAIN     13
XE     5001.5990 Hz
XS      604.8804 Hz
```



```
31-MAY-02 14:46:03
DFILE QCH
OBNUC 13C
EXMOD BCM
OFR        67.80 MHz
OBSET     135.00 KHz
OBFIN     5200.0 Hz
POINT      32768
FREQU    20000.0 Hz
SCANS        800
ACQTM      0.819 sec
PD         2.181 sec
PW1          3.6 us
IRNUC 1H
CTEMP       21.7 c
SLVNT CDCL3
EXREF      77.00 ppm
BF          1.22 Hz
RGAIN 27
OPERATOR : ____________
```

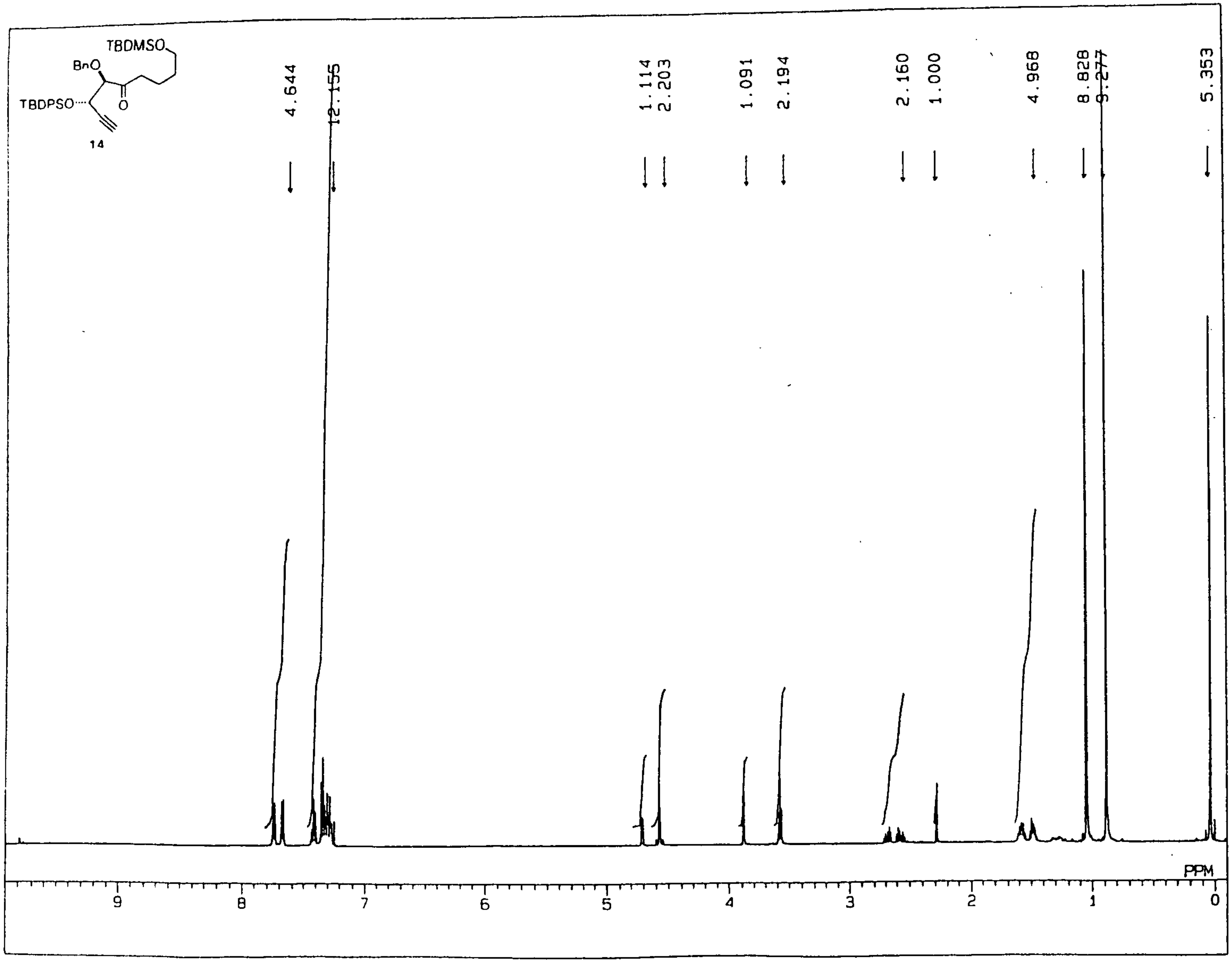


```
14-MAY-02 19:50:42
DFILE QDE1
OBNUC 1H
EXMOD NON
OFR          270.05 MHz
OBSET        112.00 kHz
OBFIN        5800.0 Hz
POINT         32768
FREQU        5405.4 Hz
SCANS            16
ACQTM         3.031 sec
PD            3.969 sec
PW1             4.9 us
IRNUC 1H
CTEMP          23.0 c
SLVNT CDCL3
EXREF          0.00 ppm
BF             0.16 Hz
RGAIN    13
OPERATOR : ____________
```

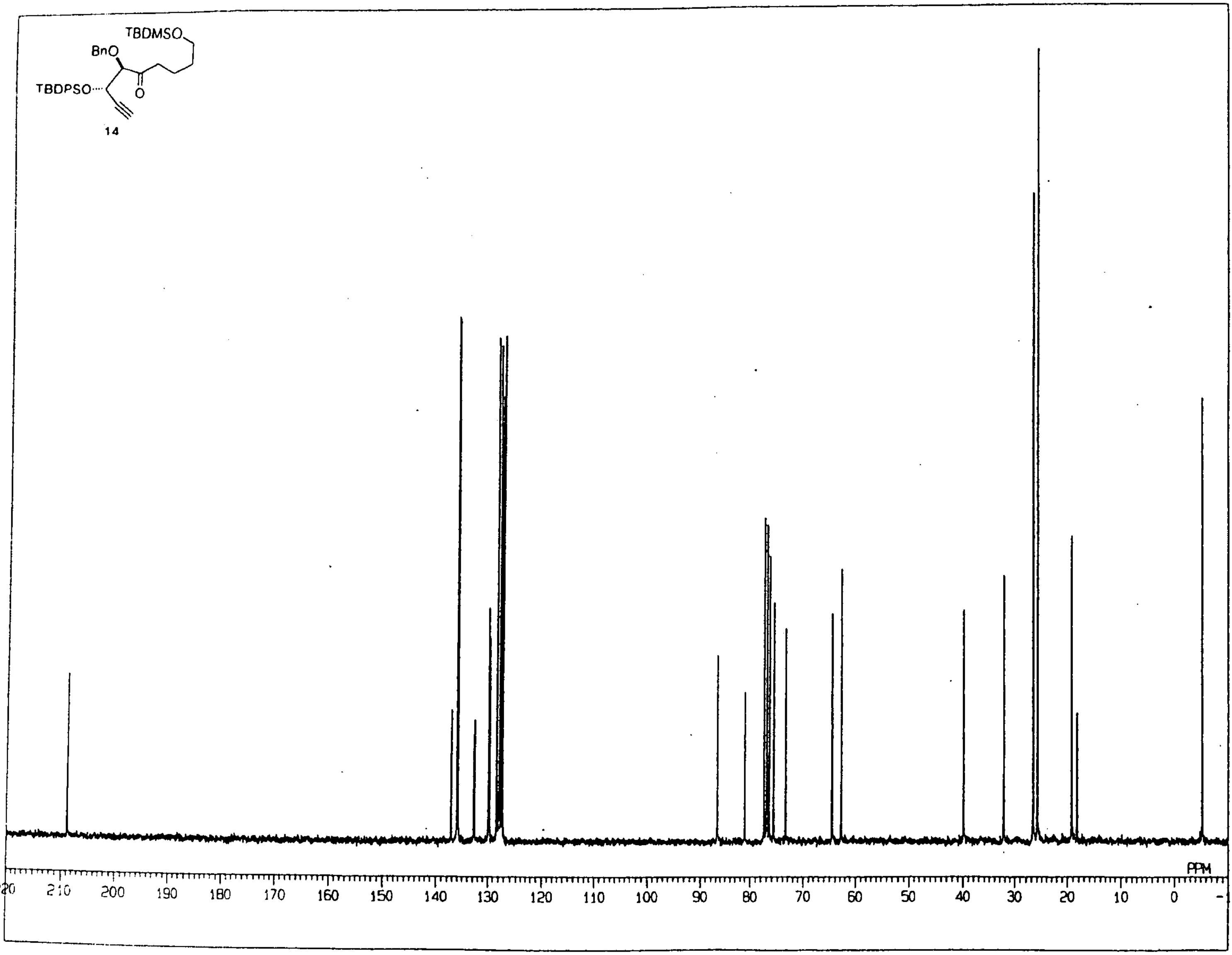


13

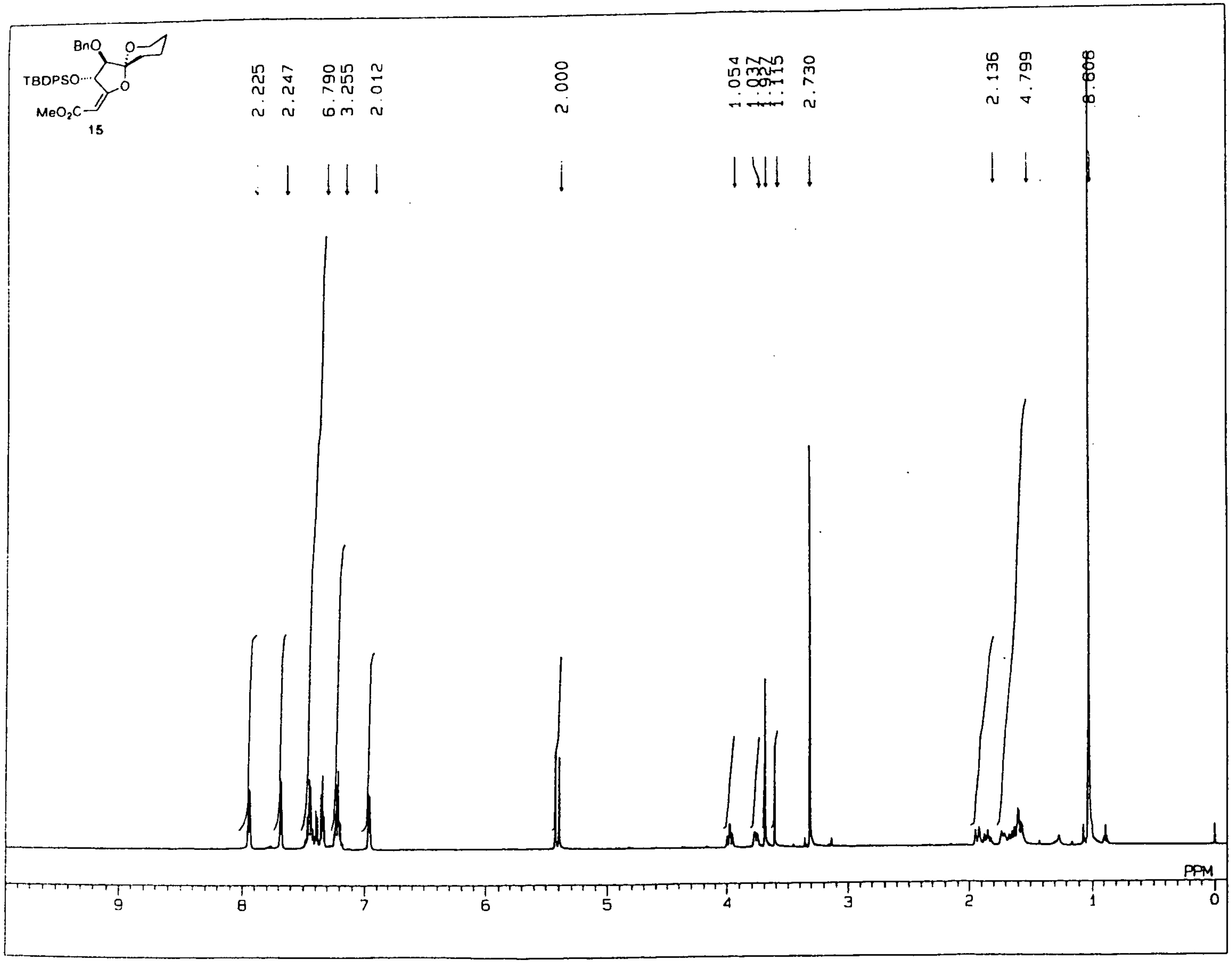
```
14-MAY-02 20:47:43
DFILE QDE1
OBNUC 13C
EXMOD BCM
OFR        67.80 MHz
OBSET     135.00 kHz
OBFIN     5200.0 Hz
POINT      32768
FREQU    20000.0 Hz
SCANS        600
ACQTM      0.819 sec
PD         2.181 sec
PW1          3.6 us
IRNUC 1H
CTEMP       23.4 c
SLVNT CDCL3
EXREF      77.00 ppm
BF          1.22 Hz
RGAIN    27
OPERATOR :
```



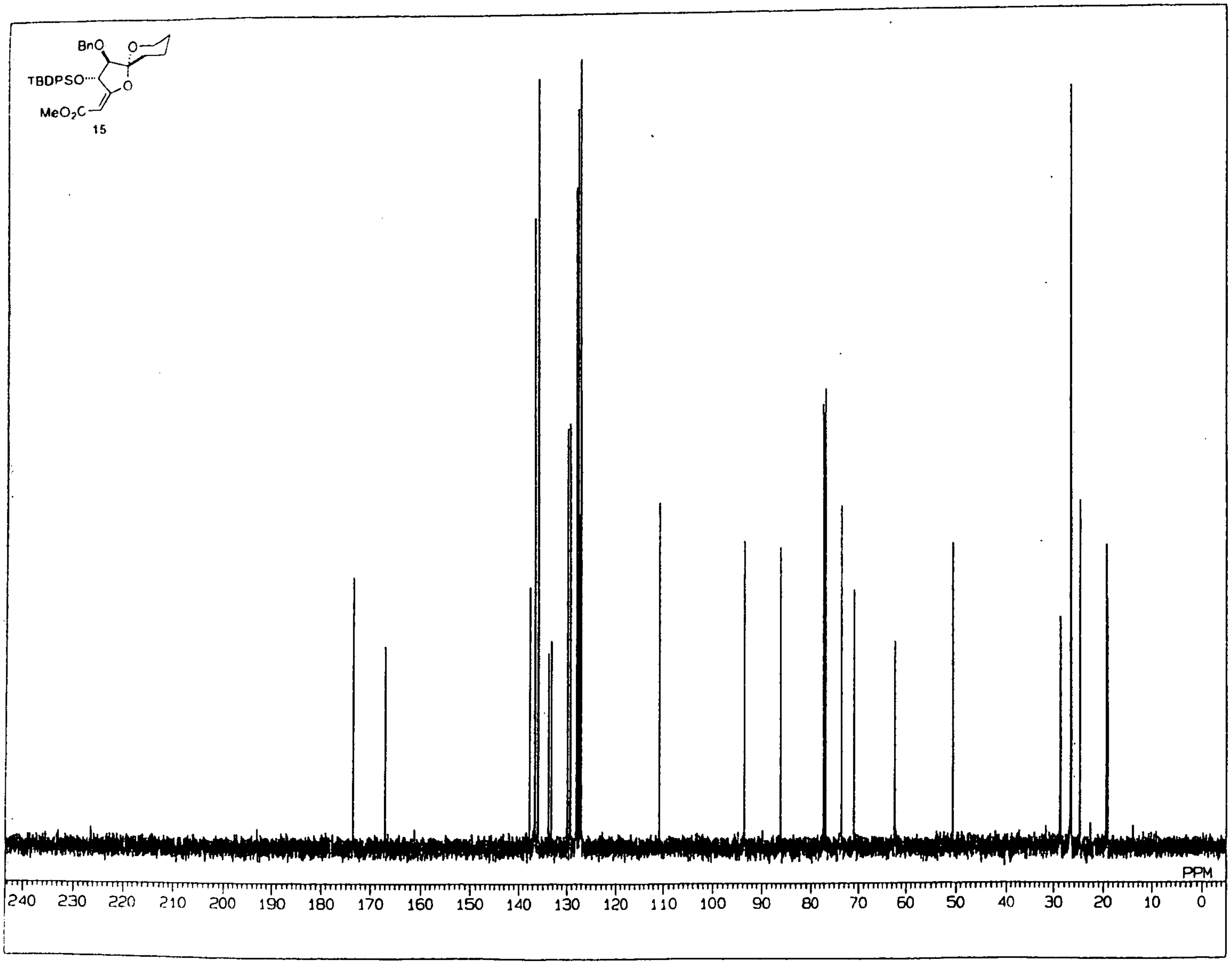
```
17-MAY-02 00: 11: 01
DFILE DU4:
COMNT 500MHZ
EXMOD SGNON
OBNUC 1H
OBFIN      10989.5 Hz
POINT        32768
FREQU       8000.0 Hz
SCANS           16
ACQTM        2.048 sec
PD           3.000 sec
PW1            3.9 us
IRFIN      10157.0 Hz.
IRATN            0
IRRPW           50 us
TEMP.   27.0 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.10 Hz
RGAIN     14
XE       5001.5990 Hz.
XS        612.2046 Hz
```



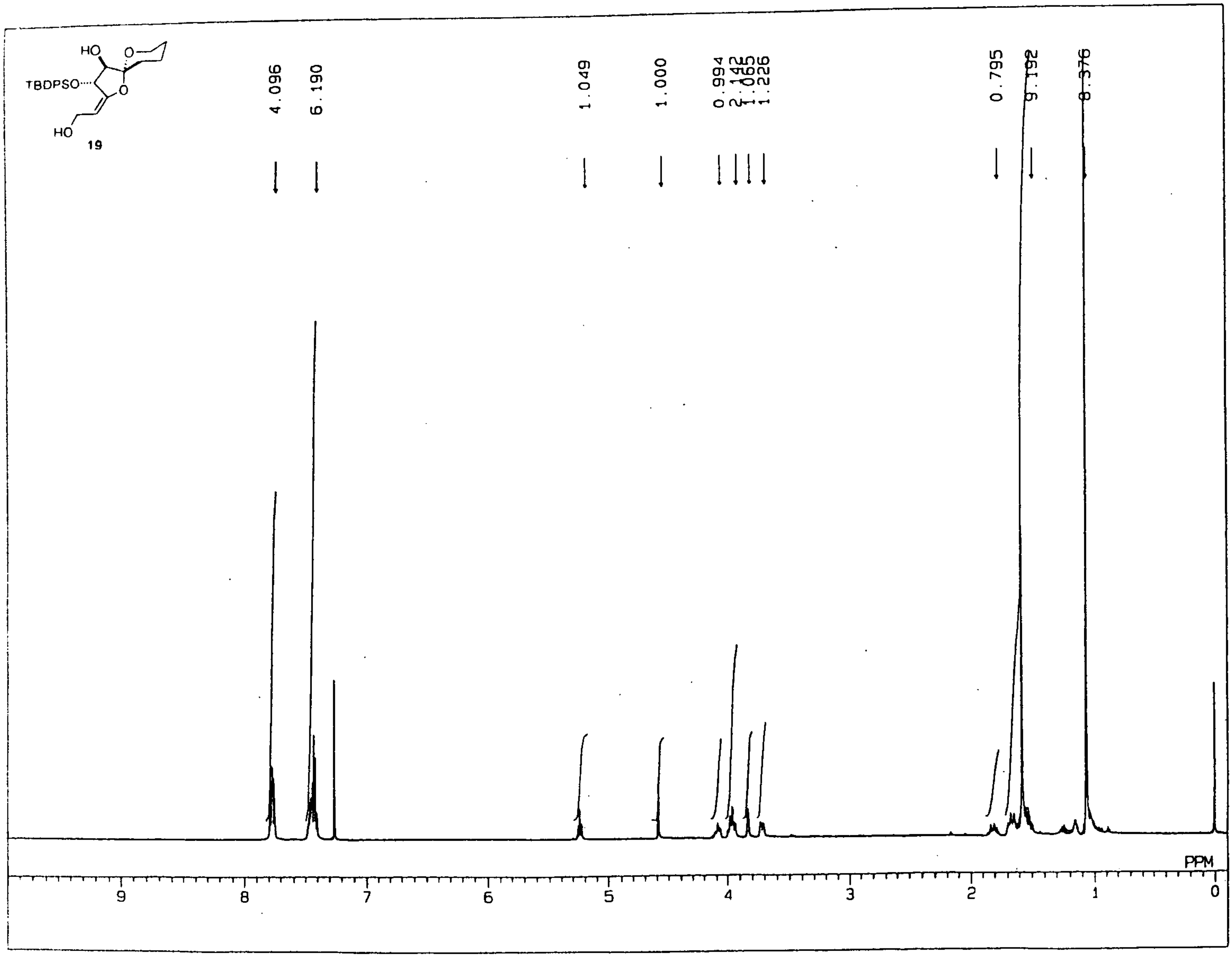
```
06-FEB-03 20:46:27
MENUF BCM
OBNUC 13C
OFR          67.80 MHz
OBSET       135.00 kHz
OBFIN       5200.0 Hz
PW1            3.6 us
PREDL       0.2000 ms
IWT            1.0 sec
POINT        32768
SPO          32768
TIMES          800
DUMMY            1
FREQU      20000.0 Hz
FLT          10000 Hz
DELAY         20.0 us
ACQTM        0.819 sec
PD           2.181 sec
ADBIT    12
RGAIN    26
BF            1.22 Hz
T1        0.0 %
T2        0.0 %
T3       90.0 %
T4      100.0 %
EXMOD BCM
EXPCM Bilevel_complete_decou
IRNUC 1H
IRFR        270.05 MHz
IRSET       112.00 kHz
IRFIN       5800.0 Hz
IRRPW           50 us
IRATN          511
DFILE Q13C
SF    TH5
LKSET         64.6 kHz
LKFIN         59.0 Hz
LKLEV          220
 GAIN           20
LKPHS          165 deg
LKSIG         1302
CSPED           15 Hz
FILDC          -70
FILDF           36
OPERATOR :
```



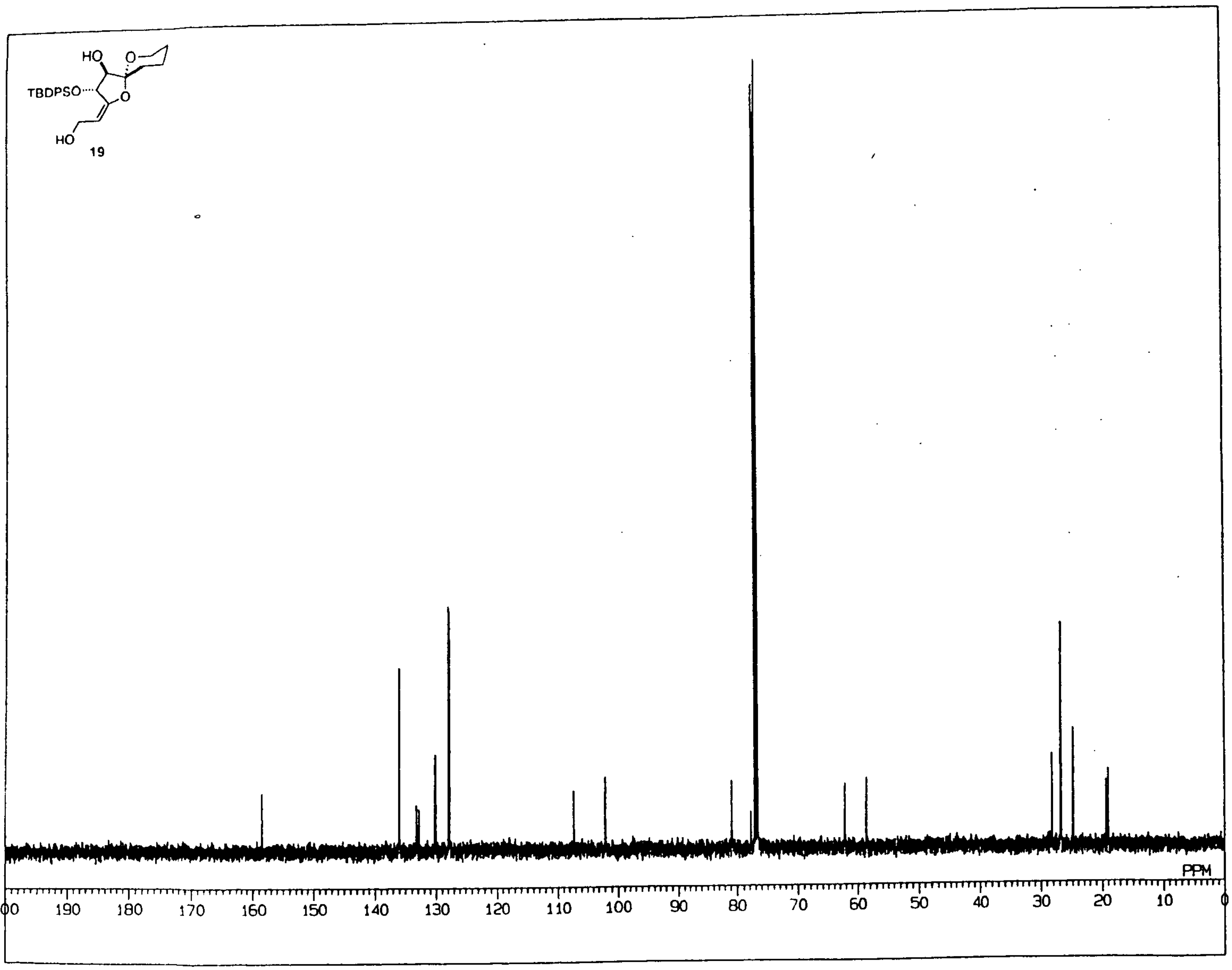
```
04-FEB-03 18:54:03
DFILE DU4:
COMNT 500MHZ
EXMOD SGNON
OBNUC 1H
OBFIN      10989.5 Hz
POINT        32768
FREQU       8000.0 Hz
SCANS            1
ACQTM        2.048 sec
PD           3.000 sec
PW1            3.9 us
IRFIN      10157.0 Hz
IRATN            0
IRRPW           50 us
TEMP.   27.0 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.10 Hz
RGAIN     21
XE       5001.5990 Hz
XS        606.8335 Hz
```



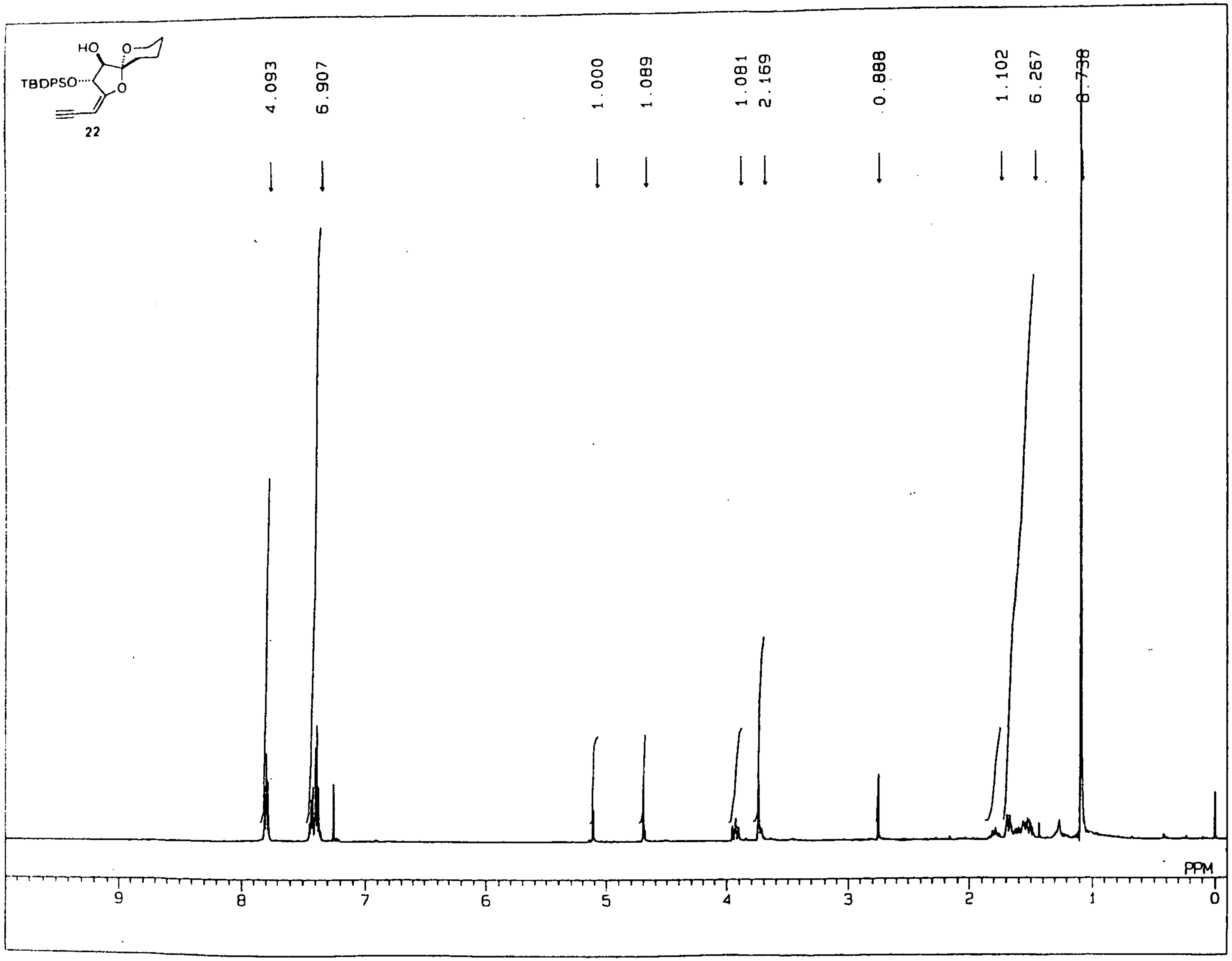
```
04-FEB-03 23:24:02
DFILE TH5CSN
COMNT TH5: 13C-S/N-
EXMOD SGBCM
OBNUC 13C
OBFIN      15308.7 Hz
POINT        65536
FREQU      31250.0 Hz
SCANS          200
ACQTM        1.049 sec
PD           0.700 sec
PW1            5.6 us
IRFIN      10157.0 Hz
IRATN           25
IRRPW           50 us
TEMP.   27.0 c
SLVNT CDCL3
EXREF        77.00 ppm
BF            0.77 Hz
RGAIN     31
XE      31250.0000 Hz
XS          0.0000 Hz
```


19

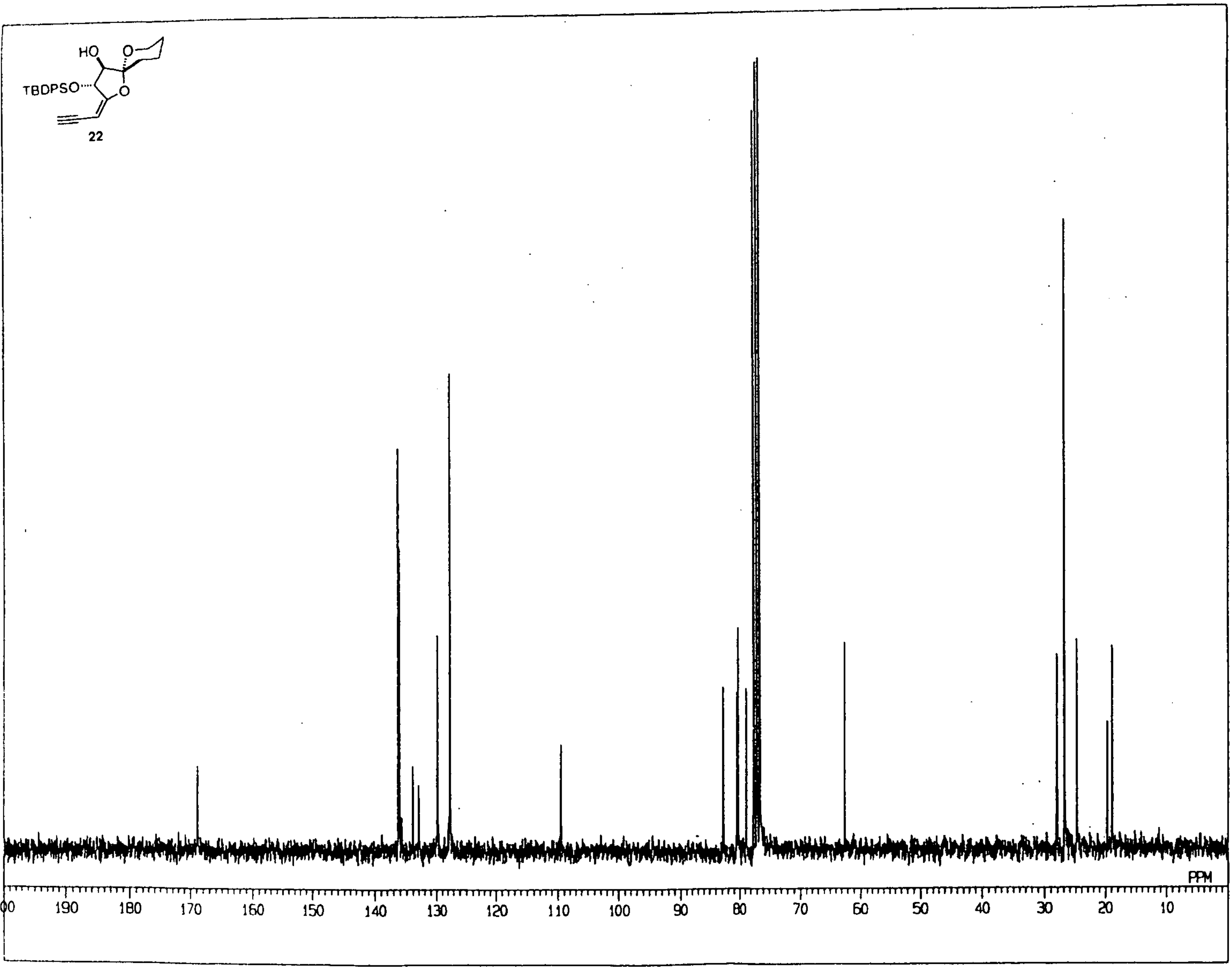
```
11-FEB-03 12: 18: 35
DFILE DU4:
COMNT 500MHZ
EXMOD SGNON
OBNUC 1H
OBFIN      10989.5 Hz
POINT        32768
FREQU       8000.0 Hz
SCANS           16
ACQTM        2.048 sec
PD           3.000 sec
PW1            3.9 us
IRFIN      10157.0 Hz
IRATN            0
IRRPW           50 us
TEMP.   27.0 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.10 Hz
RGAIN     22
XE       5001.5990 Hz
XS        620.5054 Hz
```



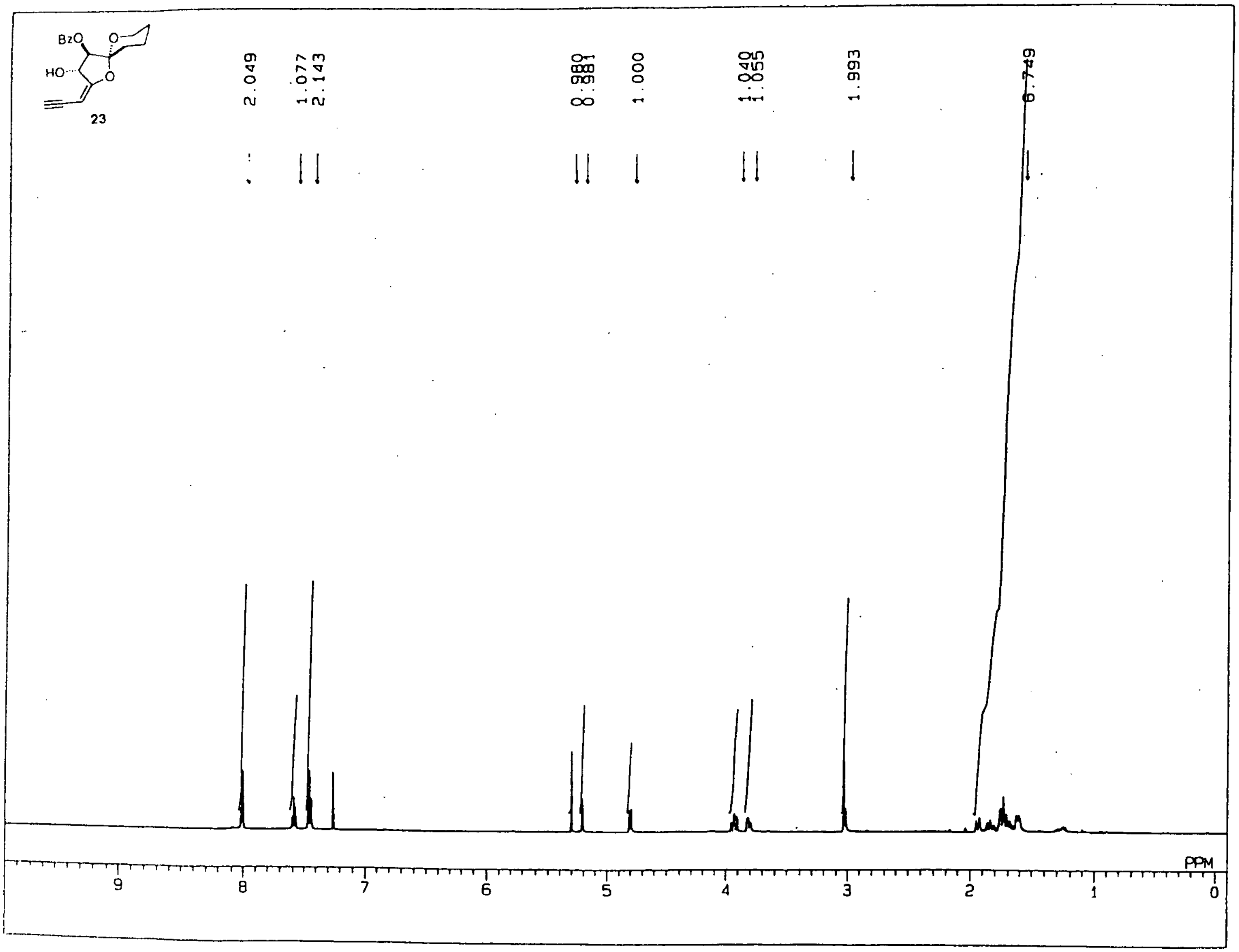
```
11-FEB-03 12:48:57
DFILE TH5CSN
COMNT TH5:13C-S/N-
EXMOD SGBCM
OBNUC 13C
OBFIN      15308.7 Hz
POINT        65536
FREQU      31250.0 Hz
SCANS         1000
ACQTM        1.049 sec
PD           0.700 sec
PW1            5.6 us
IRFIN      10157.0 Hz
IRATN           25
IRRPW           50 us
TEMP.   27.0 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            0.77 Hz
RGAIN     32
XE      25153.0600 Hz
XS       2421.2700 Hz
```



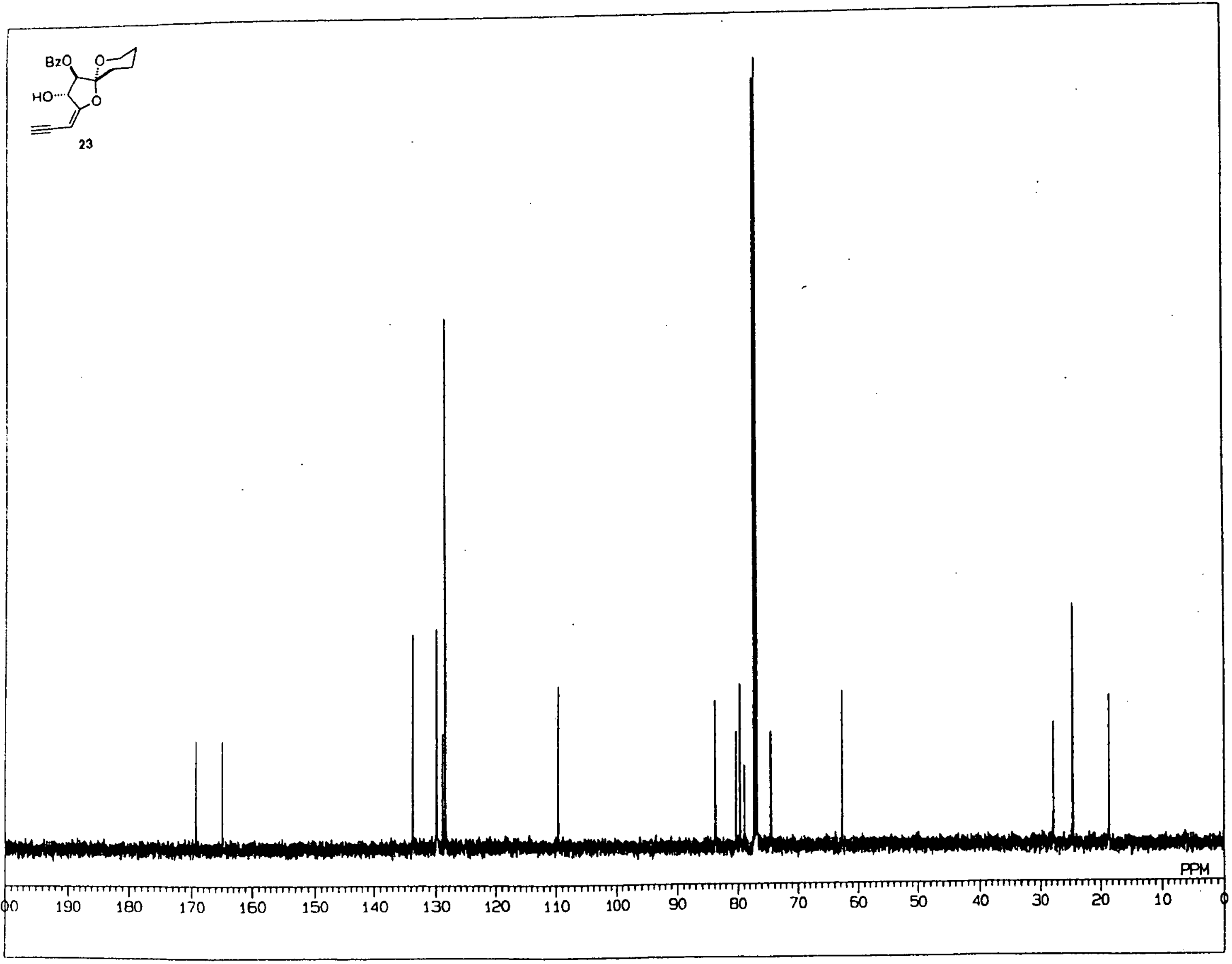
```
17-FEB-03 18: 47: 19
DFILE DU4:
COMNT 500MHZ
EXMOD SGNON
OBNUC 1H
OBFIN      10989.5 Hz
POINT        32768
FREQU       8000.0 Hz
SCANS           16
ACQTM        2.048 sec
PD           3.000 sec
PW1            3.9 us
IRFIN      10157.0 Hz
IRATN            0
IRRPW           50 us
TEMP.  27.0 c
SLVNT CDCL3
EXREF         0.00 ppm
BF            0.10 Hz
RGAIN    18
XE       5001.5990 Hz
XS        617.0874 Hz
```



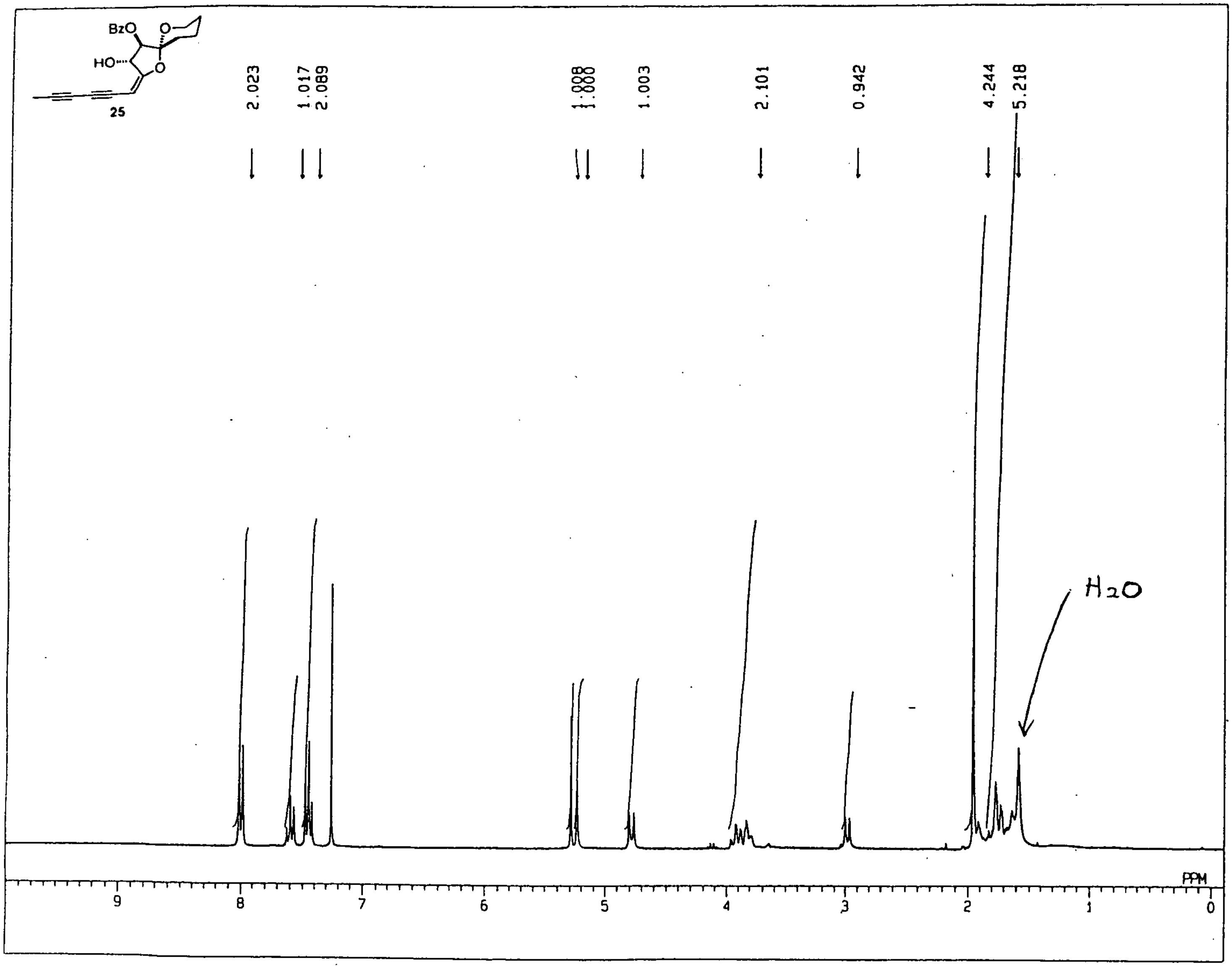
```
17-FEB-03 22:47:29
MENUF BCM
OBNUC 13C
OFR          67.80 MHz
OBSET       135.00 kHz
OBFIN       5200.0 Hz
PW1            3.6 us
PREDL       0.2000 ms
IWT            1.0 sec
POINT        32768
SPO          32768
TIMES          700
DUMMY            1
FREQU      20000.0 Hz
FLT          10000 Hz
DELAY         20.0 us
ACQTM        0.819 sec
PD           2.181 sec
ADBIT     12
RGAIN     27
BF             1.22 Hz
T1       0.0 %
T2       0.0 %
T3      90.0 %
T4     100.0 %
EXMOD BCM
EXPCM Bilevel.complete.decou
IRNUC 1H
IFR         270.05 MHz
IRSET       112.00 kHz
IRFIN       5800.0 Hz
IRRPW           50 us
TRATN          511
DFILE 01H
SF    TH5
LKSET         64.6 kHz
'KFIN         59.0 Hz
LKLEV          200
LGAIN           20
LKPHS         -165 deg
KSIG          2303
CSPED           16 Hz
FILDC          -70
FILDF           20
OPERATOR :
```



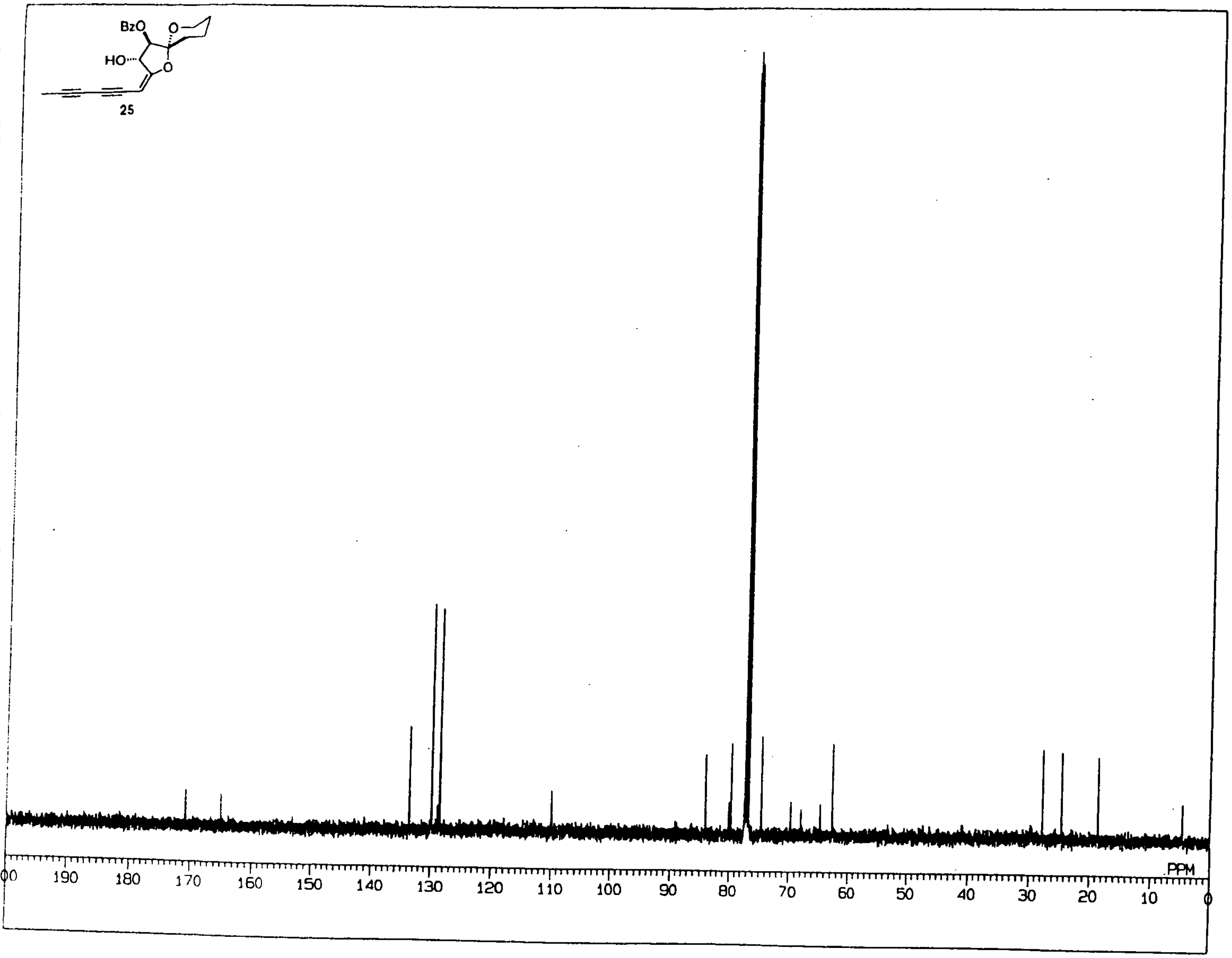
```
21-FEB-03 02: 20: 10
DFILE DU4:
COMNT 500MHZ
EXMOD SGNON
OBNUC 1H
OBFIN      10989.5 Hz
POINT        32768
FREQU       8000.0 Hz
SCANS            1
ACQTM        2.048 sec
PD           3.000 sec
PW1            3.9 us
IRFIN      10157.0 Hz
IRATN            0
IRRPW           50 us
TEMP.  27.0 c
SLVNT CDCL3
EXREF         7.26 ppm
BF            0.10 Hz
RGAIN    21
XE       5001.5990 Hz
XS        620.3191 Hz
```



```
21-FEB-03 02:30:53
DFILE TH5CSN
COMNT TH5:13C-S/N-
EXMOD SGBCM
OBNUC 13C
OBFIN      15308.7 Hz
POINT        65536
FREQU      31250.0 Hz
SCANS         1200
ACQTM        1.049 sec
PD           0.700 sec
PW1            5.6 us
IRFIN      10157.0 Hz
IRATN           25
IRAPW           50 us
TEMP.   27.0 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            0.77 Hz
RGAIN    32
XE      25153.0600 Hz
XS       2420.3170 Hz
```



```
18-MAR-03 21:09:52
MENUF  NON
OBNUC  1H
OFR         270.05 MHz
OBSET       112.00 kHz
OBFIN       5800.0 Hz
PW1            4.9 us
PREDL       0.2000 ms
IWT            1.0 sec
POINT        32768
SPO          32768
TIMES           16
DUMMY            1
FREQU       5405.4 Hz
FLT           2700 Hz
DELAY         74.1 us
ACQTM        3.031 sec
PD           3.969 sec
ADBIT     12
RGAIN     22
BF             0.16 Hz
T1       0.0 %
T2       0.0 %
T3      90.0 %
T4     100.0 %
EXMOD  NON
EXPCM  NON: Single.coupled: PW1
IRNUC  1H
IFR         270.05 MHz
IRSET       112.00 kHz
IRFIN       5800.0 Hz
IRRPW           50 us
IRATN          511
DFILE  Q13C
SF     TH5
LKSET         64.6 kHz
LKFIN         59.0 Hz
LKLEV          200
LGAIN           20
LKPHS          165 deg
LKSIG         1185
CSPED           17 Hz
FILDC          -70
FILDF           36
OPERATOR :
```



24-MAR-03 10:43:35
DFILE TH5CSN
COMNT TH5: 13C-S/N-
EXMOD SGBCM
OBNUC 13C
OBFIN 15308.7 Hz
POINT 65536
FREQU 31250.0 Hz
SCANS 1200
ACQTM 1.049 sec
PD 0.700 sec
PW1 5.6 us
IRFIN 10157.0 Hz
IRATN 33
IRRPW 50 us
TEMP. 27.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.77 Hz
RGAIN 31
XE 25153.0600 Hz
XS 2421.2700 Hz