JOC The Journal of Organic Chemistry

J. Org. Chem., 1998, 63(20), 6782-6783, DOI:10.1021/jo9816360

Supporting Information

General Experimental. Tetrahydrofuran was distilled from dark blue solutions of sodium/benzophenone. Acetone was distilled from anhydrous calcium sulfate. Pyridine was distilled from CaH_2 and stored over molecular sieves. Toluene was distilled from sodium. The concentration of *n*-butyllithium (Aldrich) was determined by titration with *sec*-butyl alcohol using 1,10-phenanthroline as the indicator. The following compounds were purchased from Aldrich and used without further purification: benzenesulfonylchloride, diisopropylamine, iodomethane, trimethylsilylchloride. Hexamethylphosphoramide was purchased from Aldrich and stored over molecular sieves.

Literature procedures were followed for the preparation of dimethyltitanocene (**8**),[1] 3-allyl-3-phenyloxetan-2-one (**7a**),[2] 3-methyl-4-spirocyclohexyloxetan-2-one (**7c**),[3] 3-methyl-3-phenyloxetan-2-one (**7d**),[2] *trans*-3-methyl-4-phenylethyloxetan-2-one (**11a**),[4] 3-methyl-3-phenyl-2-methyleneoxetane (**9d**).[2]

5-(*t*-Butyldiphenylsilyloxy)-2-(1-hydroxy-1-methylethyl)pentanoic acid. *n*-Butyllithium (5.60 mmol, 4.3 mL) was added dropwise to a stirred solution of diisopropylamine (0.79 g, 5.60 mmol) in dry THF (5.6 mL) at 0 °C under N_2. The solution was maintained at 0 °C for 10 min and then warmed to RT. 5-(*t*-Butyldiphenylsilyloxy)pentanoic acid[5] (1.00 g, 2.80 mmol) in dry THF (2.8 mL) was then added to the resulting solution, and the reaction was maintained at RT for 1.5 h. Dry acetone (0.24 mL, 3.40 mmol) in THF (1.36 mL) was added, and the mixture was stirred for 16 h. The mixture was cooled to 0 °C and acidified to pH 2 with 2N HCl. The resulting mixture was extracted with ether (3 x 30 mL), and the combined organic extracts were dried ($MgSO_4$) and concentrated *in vacuo*. Purification by chromatography on flash silica (petroleum ether/ethyl acetate/acetic acid 74.7: 25: 0.3) afforded 5-(*t*-butyldiphenylsilyloxy)-2-(1-hydroxy-1-methylethyl)pentanoic acid as a pale yellowish oil (0.70 g, 60 %). IR (film) 3500 (br), 2980, 1710, 1510, 1490, 1110, 1190 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.65 (m, 4H), 7.39 (m, 6H), 3.69 (m, 2H), 2.42 (dd, J = 10.6, 4.3 Hz, 1H), 1.78 (m, 2H), 1.61 (m, 2H), 1.31 (s, 3H), 1.26 (s, 3H), 1.05 (s, 9H);^{13}C NMR (100 MHz, $CDCl_3$) δ 179.8, 135.6, 133.8, 129.7, 127.7, 71.4, 63.4, 55.4, 30.8, 29.2, 26.8, 26.5, 23.9, 19.2; MS (EI) *m/z*

413 (M^+-H), 377, 327 (100), 273, 207, 195, 135, 55; Anal Calcd for $C_{24}H_{34}O_4Si$: C, 69.53; H, 8.27. Found C, 69.52; H, 8.28.

2-(3-*t*-Butyldiphenylsilyloxypropyl)-3,3-dimethyloxetan-2-one (7b). Benzenesulfonylchloride (0.51 g, 2.88 mmol) was added dropwise to a solution of 5-(*t*-butyldiphenylsilyloxy)-2-(1-hydroxy-1-methylethyl)pentanoic acid (0.40 g, 0.96 mmol) in dry pyridine (20 ml) at 0 °C under nitrogen. The solution was maintained at 0 °C for 24 h. Ice cold water (200 ml) and diethyl ether (100 ml) were then added to the pink mixture, and the layers were separated. The aqueous layer was further extracted with diethyl ether (4 x 25 mL). The combined organic layers were then washed with saturated aqueous sodium bicarbonate (25 ml), dried with magnesium sulfate, and concentrated under reduced pressure. Purification by chromatography on flash silica (petroleum ether/ethyl acetate/triethyl amine 97: 2.0: 1.0 to 89: 10.0: 1.0) afforded **7b** as a viscous pale yellow oil (0.33 g, 85 %). IR (film) 3150, 3100, 2990, 2900, 1780, 1500, 1450, 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 (m, 4H), 7.39 (m, 6H), 3.66 (m, 2H), 3.17 (dd, J = 8.0, 8.0 Hz, 1H), 1.77 (m, 3H), 1.59 (m, 1H), 1.54 (s, 3H), 1.45 (s, 3H), 1.03 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 135.6, 133.7, 129.7, 127.7, 80.0, 63.1, 57.9, 30.0, 28.0, 26.9, 21.8, 21.6, 19.2; HRMS (EI) Calcd for $C_{20}H_{23}O_3Si$ (M^+- *t*-butyl) 339.1417. Found: 339.1409.

General procedure for methylenation. 3-Allyl-3-phenyl-2-methyleneoxetane (9a): Dimethyltitanocene (0.5 M in toluene, 10.8 mL, 5.4 mmol) and 3-allyl-3-phenyloxetan-2-one (0.50 g, 2.7 mmol) were stirred at 80 °C under N_2 in the dark. The reaction was monitored by TLC, and after the disappearance of the starting material (2-15 h) the solution was cooled. An equal volume of petroleum ether was then added, at which point a yellow precipitate formed. The mixture was passed through celite with petroleum ether until the filtrate was colorless. After concentration, if large amounts of solid were still present, the mixture was diluted with petroleum ether and filtered through celite a second time. The residue was then purified by flash chromatography on flash silica (petroleum ether/ethyl acetate/triethylamine 98.5:0.5:1) which afforded methyleneoxetane **9a** (0.38g, 76%) as a

pale yellow oil: IR (film) 3150, 3000, 2900, 1650, 1500, 1490, 1190, 980 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 5H), 5.76 (m, 1 H), 5.15 (m, 1 H), 5.12 (m, 1H), 4.76 (d, *J* = 5.0 Hz, 1H), 4.72 (d, *J* = 5.0 Hz, 1H), 4.32 (d, *J* = 4.0 Hz, 1 H), 4.03 (d, *J* = 4.0 Hz, 1 H), 2.74 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 141.9, 133.2, 128.5, 126.9, 126.2, 118.8, 80.4, 79.1, 54.0, 44.0; MS (EI) *m/z* 186 (M^+), 158, 115, 103 (100), 77; HRMS (EI) Calcd for $C_{13}H_{14}O$ (M^+) 186.1045. Found: 186.1044.

3-(3-*t*-Butyldiphenylsilyloxypropyl)-4,4-dimethyl-2-methyleneoxetane (9b). Purification by chromatography on flash silica (petroleum ether/ethyl acetate/triethylamine 99:0.5:0.5) afforded methyleneoxetane **9b** (60%) as a pale yellow oil: ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (m, 4H), 7.42 (m, 6H), 4.04 (dd, *J* = 3.2, 2.3 Hz, 1H), 3.72 (dd, *J* = 3.2, 1.8 Hz, 1H), 3.68 (m, 2H), 3.01 (m, 1H), 1.65 (m, 4H), 1.47 (s, 3H), 1.37 (s, 3H), 1.06 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 135.6, 133.9, 129.6, 127.7, 85.8, 78.5, 63.6, 49.9, 30.5, 29.2, 26.9, 25.0, 22.2, 19.2; MS (EI) *m/z* 338 (M^+- *t*-butyl)), 337 (100), 259, 225, 199, 139, 121; Anal Calcd for $C_{25}H_{34}O_2Si$: C, 76.09; H, 8.68. Found C, 76.15; H, 8.59.

3-Methyl-4-spirocyclohexyl-2-methyleneoxetane (9c). Purification by chromatography on flash silica (petroleum ether/triethylamine 99:1) afforded methyleneoxetane **9c** (24%) as a pale yellow liquid: IR (film) 3000, 2850, 1700, 1450, 990 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.06 (dd, *J* = 3.2, 2.3 Hz, 1H), 3.71 (dd, *J* = 3.2, 1.9 Hz, 1H), 3.04 (qdd, *J* = 7.3, 2.3, 1.9 Hz, 1H), 1.70 (m, 2H), 1.56 (m, 4H), 1.48 (m, 3H), 1.24 (m, 1H), 1.19 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 87.5, 77.6, 44.6, 38.3, 31.5, 25.2, 22.6, 22.4, 12.4; MS (EI) *m/z* 152 (M^+), 137, 109, 99, 81 (100), 67, 55; HRMS (FAB) Calcd for $C_{10}H_{16}O$ (M^++1) 153.1279. Found: 153.1278.

***trans*-3-Methyl-4-phenylethyl-2-methyleneoxetane (11b).** Purification by chromatography on flash silica (petroleum ether/ethyl acetate/triethylamine 98.5:0.5:1) afforded

methyleneoxetane **11b** (74%) as a pale yellow oil: IR (film) 3150, 3100, 2900, 2850, 1650, 1590, 1490, 1150, 950 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.31 (m, 2H), 7.21 (m, 3H), 4.40 (ddd, *J* = 7.0, 5.5, 5.5 Hz, 1H), 4.10 (dd, *J* = 3.5, 2.3 Hz, 1H), 3.75 (dd, *J* = 3.5, 1.8 Hz, 1H), 3.05 (m, 1H), 2.73 (m, 1H), 2.64 (m, 1H), 2.16 (m, 1H), 2.04 (m, 1H), 1.25 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 141.0, 128.5, 128.4, 126.1, 86.5, 77.8, 41.9, 37.2, 30.7, 16.6; MS (EI) *m/z* 188 (M^+), 173, 117, 91(100), 77, 65; Anal Calcd for $C_{13}H_{16}O$: C, 82.93; H, 8.57. Found C, 82.74; H, 8.67.

General procedure for the preparation of terminal homopropargylic alcohols. 2-Methyl-2-phenyl-3-butyn-1-ol (10d): *n*-BuLi (1.18mL, 1.6 M, 1.8 mmol) was added dropwise to a solution of diisopropylamine (0.21 g, 2.1 mmol) in dry THF (4 mL) at 0 °C under N_2. The solution was warmed to room temperature over 15 min and then cooled to 0 °C. 3-Methyl-3-phenyl-2-methyleneoxetane (0.10 g, 0.62 mmol) in THF (2 mL) was then added dropwise to the resulting solution. When all of the methyleneoxetane was consumed (based on TLC), the reaction was quenched with H_2O (5 mL), diluted with diethyl ether (5 mL), and the layers separated. The aqueous layer was then further extracted with diethyl ether (3 x 5 mL), and the combined organic extracts were dried (Na_2SO_4) and concentrated. Purification by chromatography on flash silica gel (petroleum ether/ethyl acetate 80:20) afforded homopropargylic alcohol **10d** (0.088g, 88%) as a white solid: mp 57.5-59 °C; IR (KBr) 3500, 3250, 2900, 2130, 1500, 1490, 1090 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (m, 5H), 3.72 (m, 2H), 2.47 (s, 1H), 1.81 (dd, *J* = 7.1, 7.1 Hz, 1H), 1.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.6, 128.5, 127.2, 126.4, 87.3, 72.7, 71.8, 43.3, 25.0; MS (EI) *m/z* 160 (M^+) 145, 129 (100), 115, 77, 51; HRMS (EI) Calcd for $C_{11}H_{11}$ (M^+- OH) 143.0861. Found: 143.0863. Anal Calcd for $C_{11}H_{12}O$: C, 82.46; H, 7.55. Found C, 82.08; H, 7.15.

2-(1-ethynyl)-2-phenyl-4-penten-1-ol (10a). Purification by flash chromatography on silica gel (petroleum ether/ethyl acetate 85:15) afforded homopropargylic alcohol **10a** (85%) as a colorless oil: IR (film) 3500, 3270, 3100, 2900, 2130, 1650, 1550, 1490, 1090 cm^{-1}; ^1H NMR (400

MHz, $CDCl_3$) δ 7.53 (m, 2H), 7.37 (m, 2H), 7.26 (m, 1H), 5.74 (m, 2H), 5.04 (m, 2H), 3.80 (d, *J* = 7.0 Hz, 2H), 2.74 (dd, *J* = 13.9, 7.2 Hz, 1H), 2.66 (dd, *J* = 13.9, 7.2 Hz, 1H), 2.54 (s, 1H), 1.77 (dd, *J* = 7.2, 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 133.6, 128.5, 127.3, 127.0, 118.2, 85.5, 74.5, 70.5, 48.0, 42.0; MS (EI) *m/z* 186 (M^+), 153, 141, 115 (100), 91, 77, 51; HRMS (EI) Calcd for $C_{13}H_{14}O$ (M^+) 186.1045. Found: 186.1036.

3-Ethynyl-2-methyl-5-(*t*-butyldiphenylsiloxy)hexan-2-ol (10b). Purification by chromatography on flash silica gel (petroleum ether/ethyl acetate 85:15) afforded homopropargylic alcohol **10b** (48%) as a colorless oil: IR (film) 3500, 3360, 3100, 2850, 2130, 1650, 1500, 1490, 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.64 (m, 4H), 7.34 (m, 6H) 3.70 (m, 2H), 2.35 (m, 1H), 2.12 (d, *J* = 2.4 Hz, 1H), 1.85 (m, 1H), 1.81 (s, 1H), 1.75 (m, 1H), 1.65 (m, 1H), 1.42 (m, 1H), 1.28 (s, 3H), 1.24 (s, 3H), 1.03(s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 134.0, 129.6, 127.6, 84.8, 72.0, 71.9, 63.5, 44.6, 31.0, 27.0, 26.9, 26.4, 26.0, 19.2; MS (EI) *m/z* 337 (M^+- C_4H_9), 279, 199 (100), 139, 121, 59; HRMS (FAB) Calcd for $C_{25}H_{34}O_2Si$ (M^+) 395.2406. Found: 395.2409.

1-(1-Methyl-2-propynyl)cyclohexan-1-ol (10c). Purification by chromatography on flash silica gel (petroleum ether/ethyl acetate 95:5) afforded homopropargylic alcohol **10c** (86%) as a colorless liquid: IR (film) 3550, 3400, 2900, 2130, 1650, 1490, 1250, 990 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.51 (qd, *J* = 7.1, 2.5 Hz, 1H), 2.14 (d, *J* = 2.5 Hz, 1H), 1.54 (m, 11 H), 1.20 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 86.2, 72.0, 70.9, 37.5, 35.1, 33.6, 25.7, 21.9, 21.8, 14.6; MS (EI) *m/z* 137 (M^+-CH_3), 123, 109, 99,(100), 81, 55; HRMS (FAB) Calcd for $C_{10}H_{16}O$ (M^+) 152.1201. Found: 152.1196.

4-methyl-1-phenyl-5-hexyn-3-ol (12). Purification by chromatography on flash silica gel (petroleum ether/ethyl acetate 96: 4) afforded homopropargylic alcohol **12** (81%) as colorless oil: IR (film) 3500, 3400, 3050, 2980, 1610, 1510, 1490, 1400, 1410, 1050 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.29 (m, 2H), 7.20 (m, 3H), 3.47 (m, 1H), 2.85 (m, 1H), 2.71 (m, 1H), 2.57 (m, 1H),

2.15 (d, J = 2.4 Hz, 1H), 1.88 (m, 2H), 1.81 (d, J = 7.0 Hz, 1H), 1.26 (d J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.9, 128.4, 128.4, 125.8, 85.0, 73.4, 71.2, 36.8, 33.1, 32.0, 17.4; MS (EI) *m/z* 188 (M^+) 155, 145, 117, 91(100), 77, 65; Anal Calcd for $C_{13}H_{16}O$: C, 82.93; H, 8.57. Found: C, 82.65; H, 8.79.

1-[1-(Trimethylsilyloxymethyl)-1-methyl-3-trimethylsilyl-2-propynyl)benzene (10e). *n*-BuLi (1.18 mL, 1.6 M, 1.8 mmol) was added dropwise to a stirred solution of diisopropylamine (0.21 g, 2.1 mmol) in dry THF (4 mL) at 0 °C under N_2. The solution was warmed to room temperature over 15 min and then cooled to 0 °C. 3-Methyl-3-phenyl-2-methyleneoxetane (0.10 g, 0.62 mmol) in THF (2 mL) was added dropwise to the resulting solution. When all of the methyleneoxetane was consumed (10 min), TMSCl (1.12 g, 2.5 mmol) was added dropwise. The solution was warmed to room temperature slowly, and after 2 h the reaction was quenched with H_2O (5 mL), diluted with diethyl ether (5 mL), and the layers separated. The aqueous layer was then further extracted with diethyl ether (3 x 5 mL), and the combined organic extracts were dried (Na_2SO_4) and concentrated. Purification by chromatography on flash silica gel (petroleum ether/ethyl acetate/triethylamine 99:0.5:0.5) afforded homopropargylic ether **10e** (0.18 g, 87%) as a colorless oil: IR (film) 3100, 3000, 2890, 2130, 1300, 1150, 850 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 (m, 5H), 3.68 (d, J = 9.5 Hz, 1H), 3.61(d, J = 9.5 Hz, 1H), 1.60 (s, 3H), 0.20 (s, 9H), 0.01 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.0, 127.9, 126.9, 126.6, 110.9, 87.2, 71.8, 43.2, 24.5, 0.2, -0.5; MS (EI) *m/z* 259 (M^+- C_3H_9), 245, 201, 159, 142(100), 103, 73; HRMS (EI) Calcd for $C_{16}H_{25}OSi_2$ (M^+-CH_3) 289.1444. Found: 289.1438.

1-[1-(Methoxymethyl)-1-methyl-2-butynyl]benzene (10f). *n*-BuLi (0.59 mL, 1.6 M, 0.9 mmol) was added dropwise to a stirred solution of diisopropylamine (0.10 g, 1.0 mmol) in dry THF (2 mL) at 0 °C under N_2. The solution was warmed to room temperature over 15 min and then cooled to 0 °C. 3-Methyl-3-phenyl-2-methyleneoxetane (0.05 g, 0.31 mmol) in THF (1 mL) was added dropwise to the resulting solution. When all of the methyleneoxetane was consumed (10 min), the

solution was cooled to -78 °C. A solution of iodomethane (0.27 g, 1.8 mmol) in HMPA (1.5 mL) was then added, and the solution was maintained for 2 h, then allowed to warm slowly to RT. A white precipitate formed during this time. The reaction was quenched with H_2O (5 mL) and diluted with of diethyl ether (5 mL). The layers were separated, and the aqueous layer was then further extracted with diethyl ether (3 x 5 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated *in vacuo*. Purification by flash chromatography on silica gel (petroleum ether/ethyl acetate 98:2) afforded **10f** (0.043 g, 74%) as a colorless oil: IR (film) 3150, 3100, 3000, 2950, 2850, 1650, 1540, 1510, 1480, 1180, 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.57 (m, 2H), 7.31 (m, 2H), 7.25 (m, 1H), 3.55 (d, J = 9.0 Hz, 1H), 3.48 (d, J = 9.0, 1H), 3.35 (s, 3H), 1.92 (s, 3H), 1.59 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 128.1, 126.6, 126.5, 83.0, 81.8, 78.8, 59.6, 41.2, 26.0, 3.8; MS (EI) *m/z* 173 (M^+- CH_3) 158, 143 (100), 120, 115, 65; HRMS (EI) Calcd for $C_{12}H_{13}O$ (M^+-CH_3) 173.0966. Found: 173.0969.

References

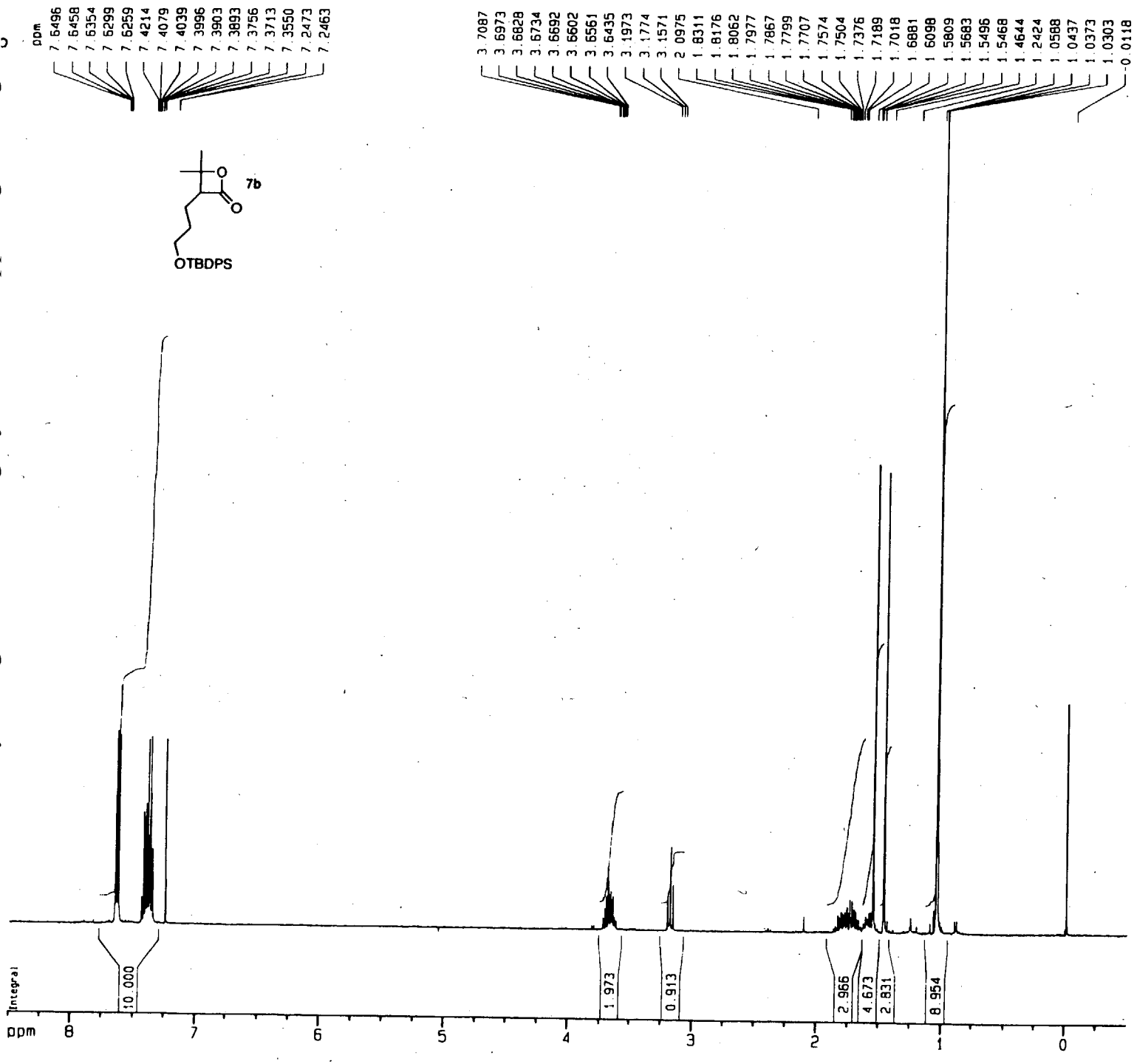
(1) Staszewski, J. P.; Akritopoulou-Zane, I.; Patane, M. A.; Hu, Y.-H. *Pure and Appl. Chem.* **1996**, *68*, 667-670.

(2) Dollinger, L. M.; Howell, A. R. *J. Org. Chem.* **1996**, *61*, 7248-7249.

(3) Adam, W.; Baeza, J.; Liu, J.-C. *J. Am. Chem. Soc.* **1972**, *94*, 2000-2006.

(4) Danheiser, R. L.; Nowick, J. S. *J. Org. Chem.* **1991**, *56*, 1176-1185.

(5) Barrett, A. G. M.; Bezuidenhoudt, B. C. B.; Howell, A. R.; Russell, M. A. *J. Org. Chem.* **1989**, *54*, 2275-2277.



Current Data Parameters

NAME	lmd-tbdps.lac
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

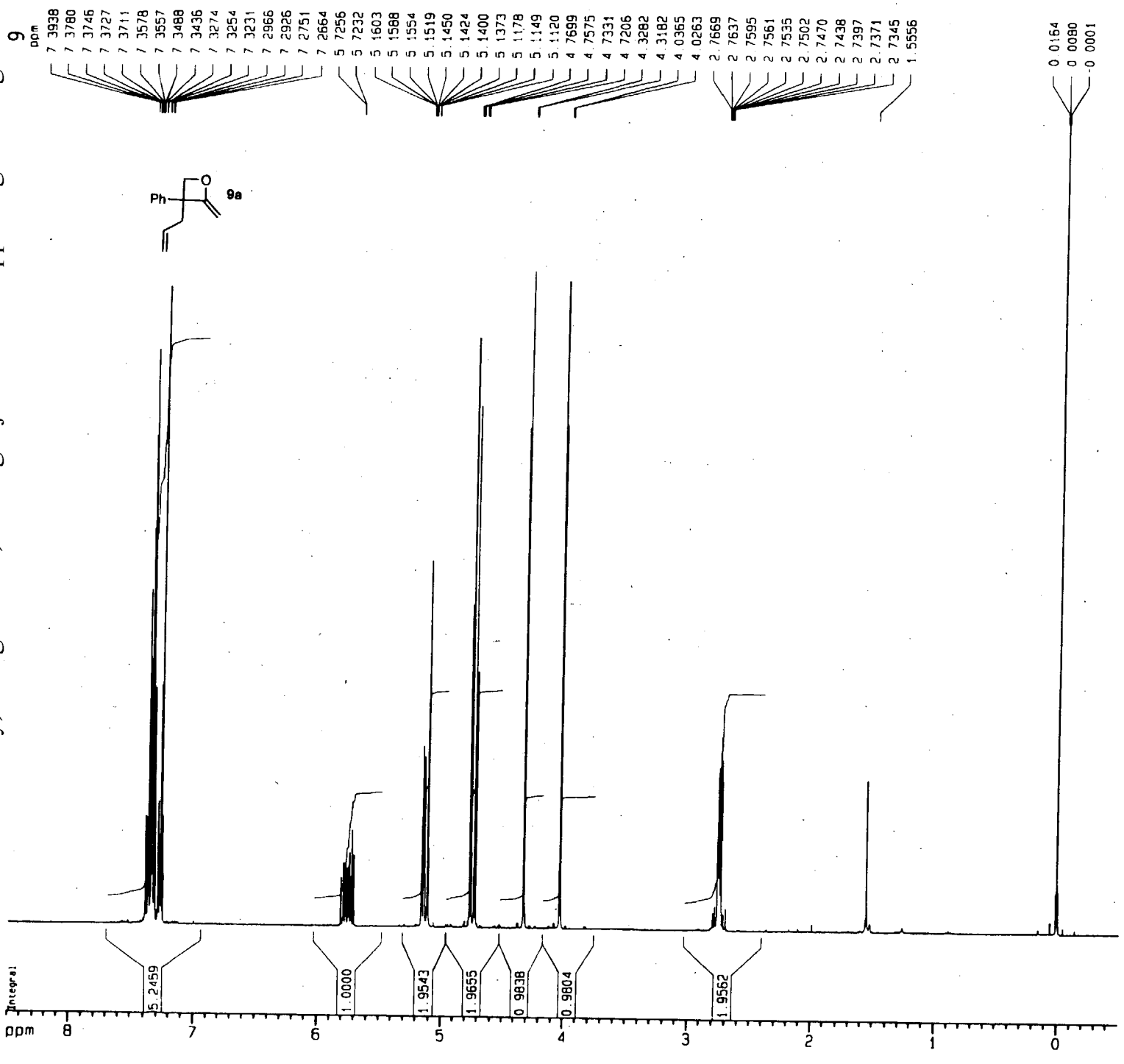
Date_	980307
Time	14.44
INSTRUM	drx400
PROBHD	5 mm GNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	574.7
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300147 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/



Current Data Parameters

NAME	ph-al.ox
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

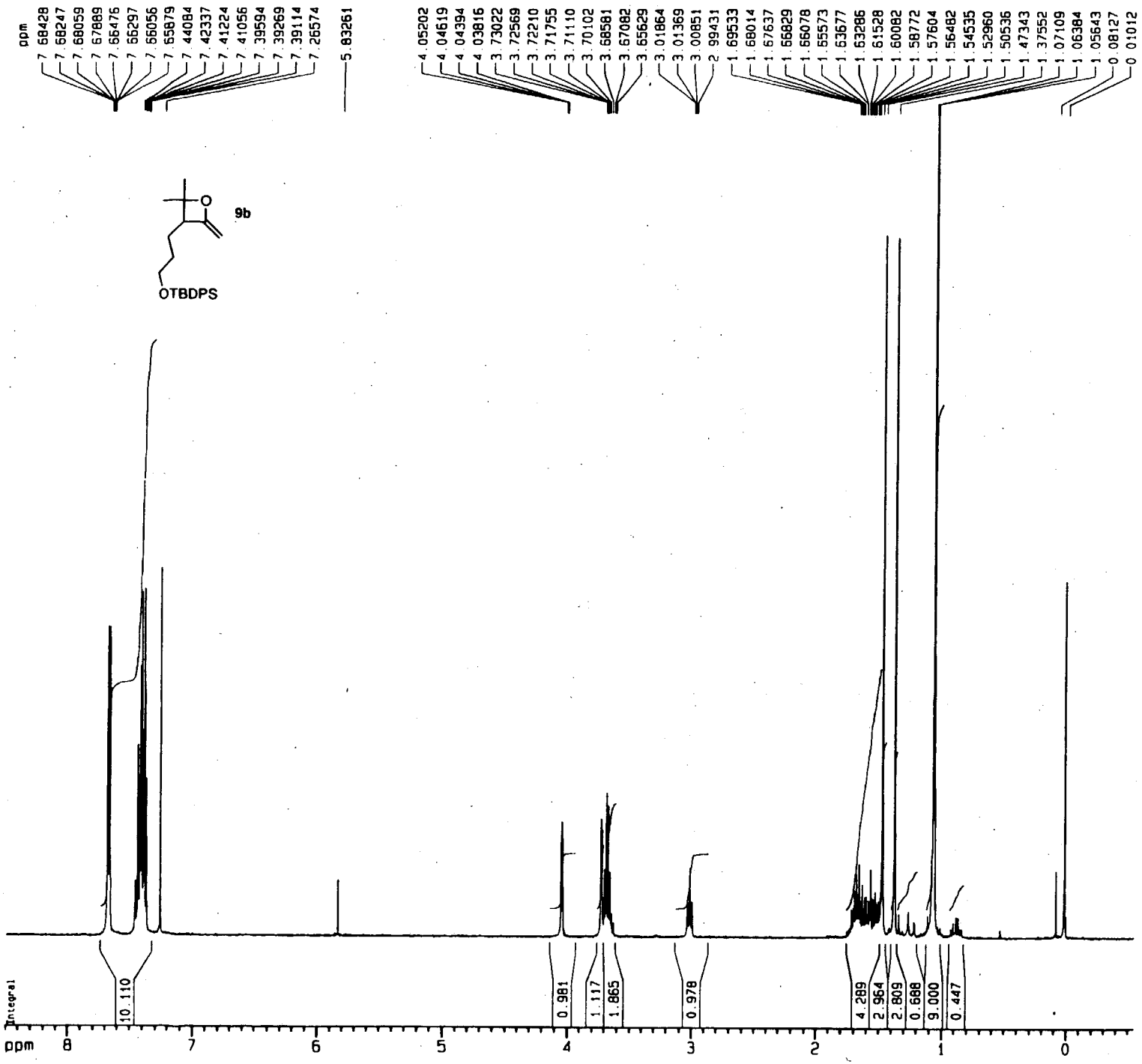
Date_	980221
Time	11.10
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	1149.4
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/



Current Data Parameters

NAME	djg1-115-3
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

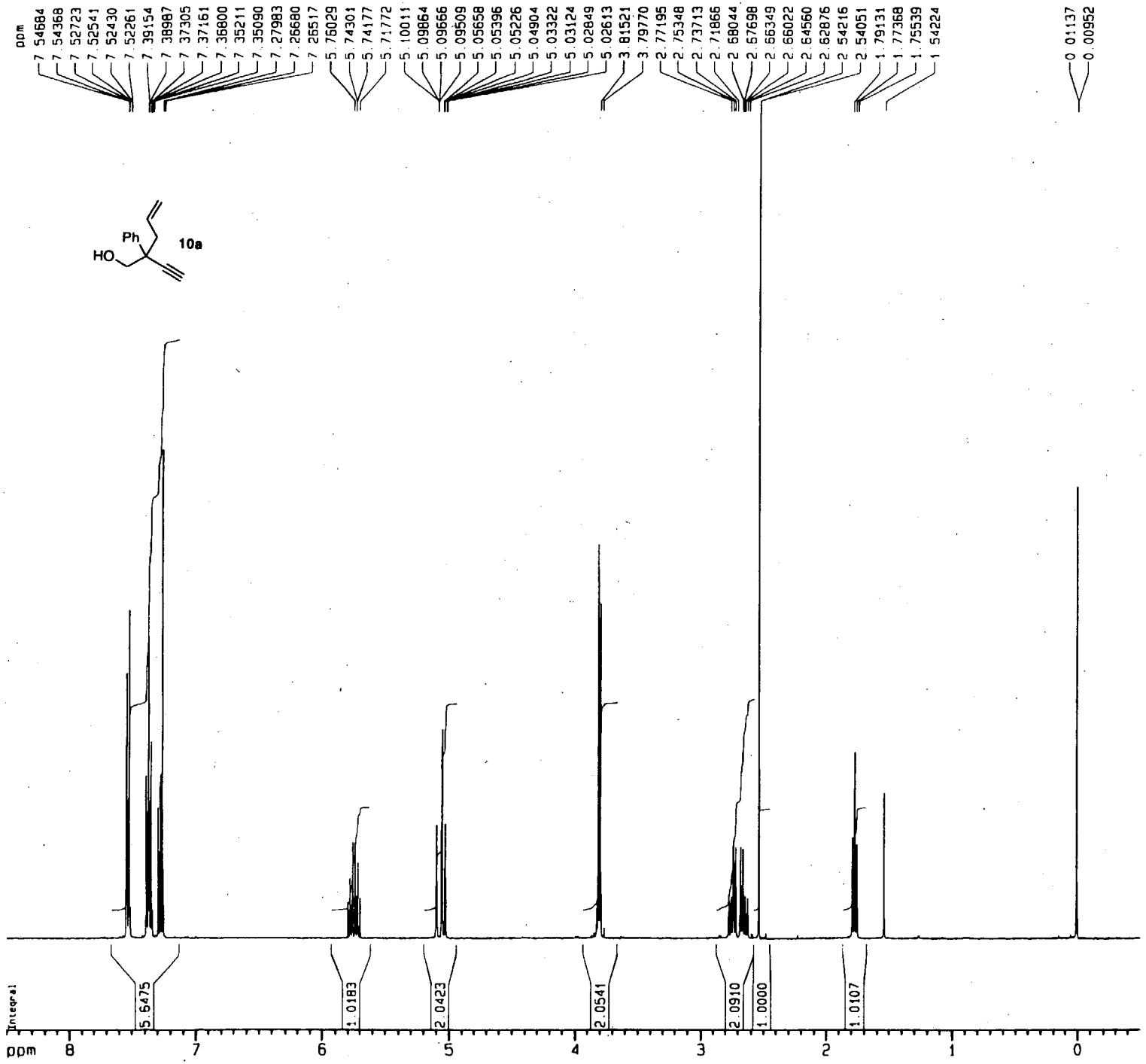
Date_	970725
Time	17.40
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	256
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.550 ppm
F2	-220.07 Hz
PPMCM	0.45250 ppm
HZCM	181.05884 Hz/



Current Data Parameters

NAME	lmd-phalhomo
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

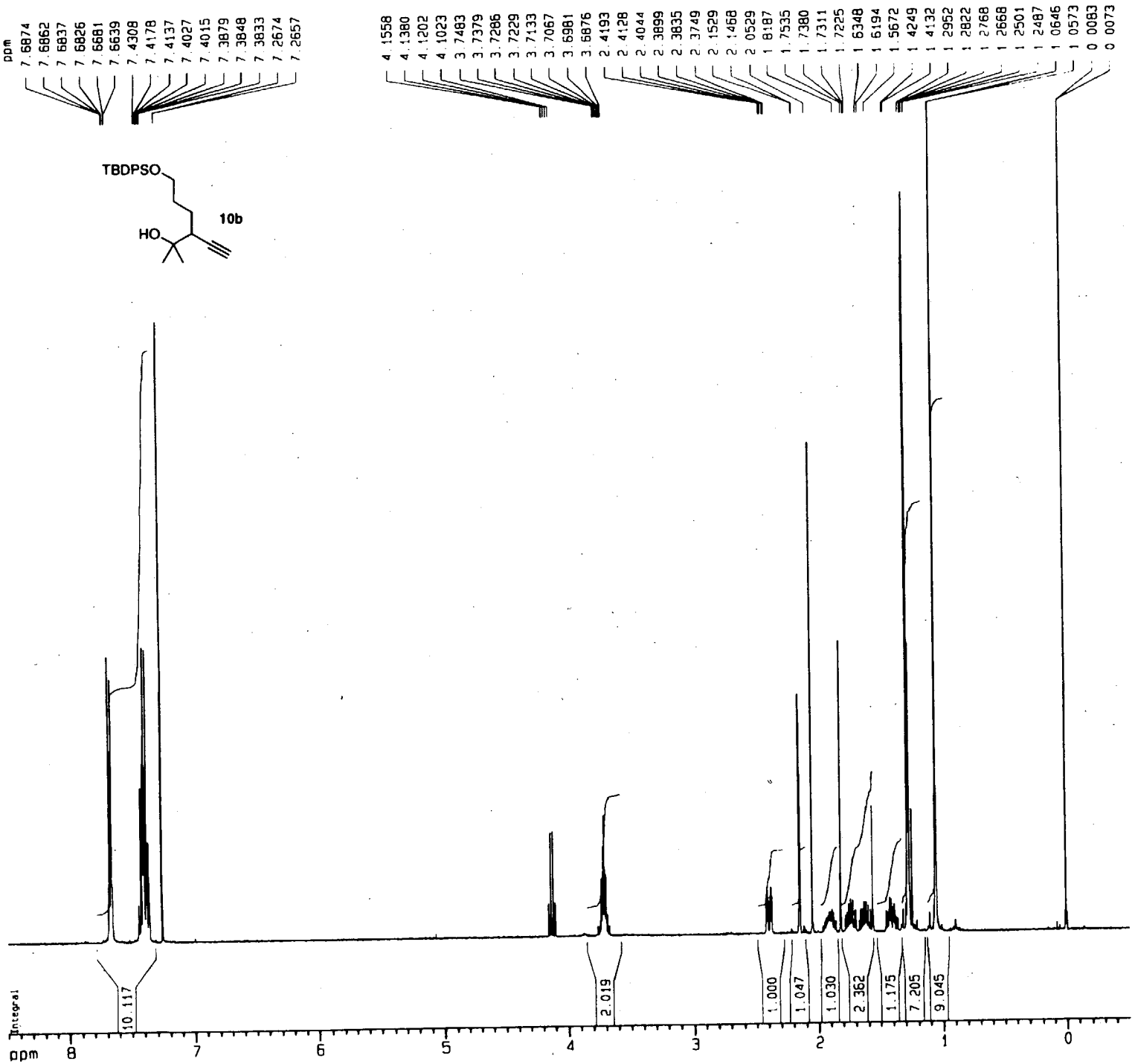
Date_	970724
Time	21.35
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	574.7
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/



Current Data Parameters

NAME	jmd-3-077-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

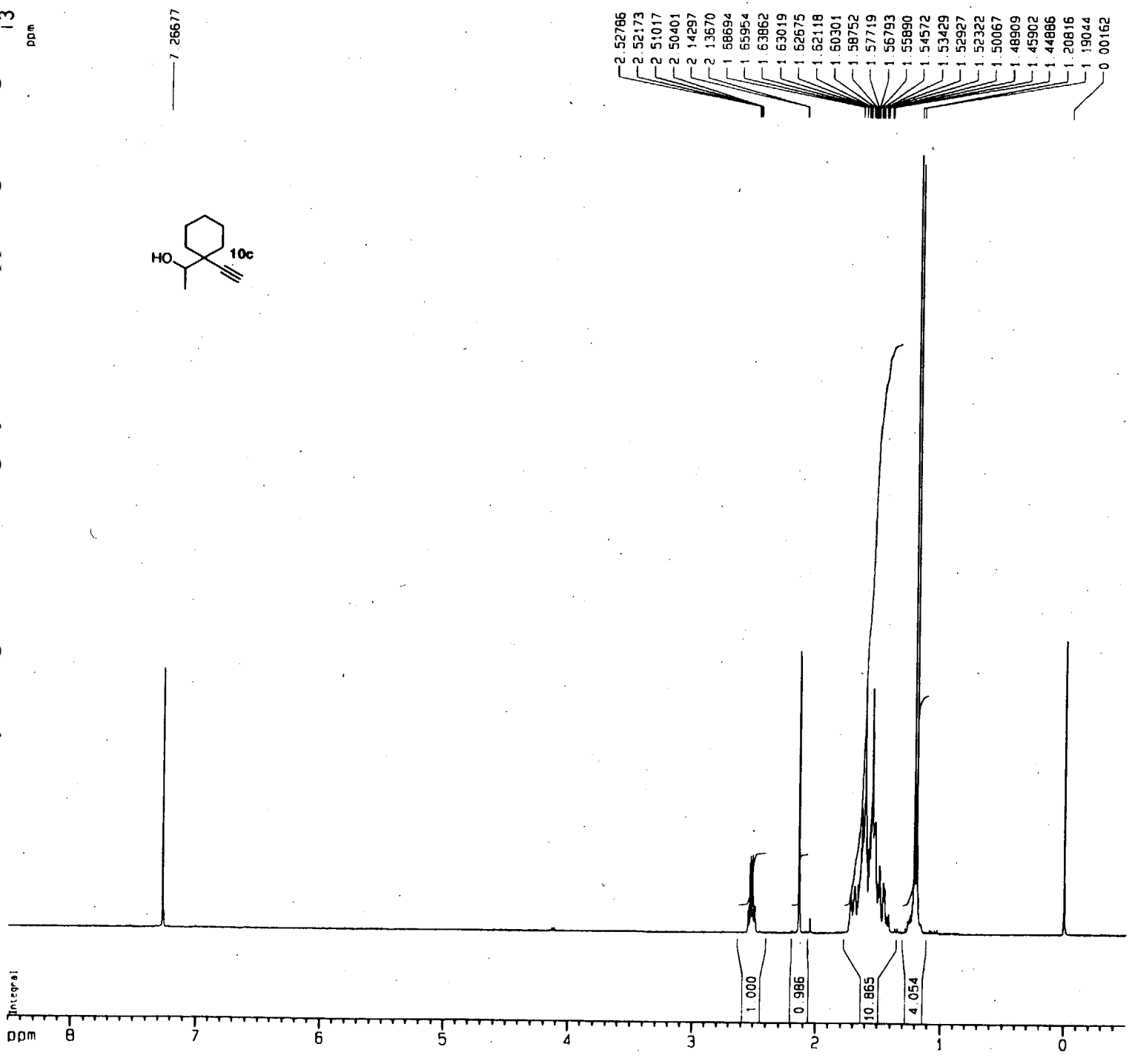
Date_	970801
Time	14.55
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	574.7
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	3.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	05850 Hz/



Current Data Parameters

NAME	lmd-3-054-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

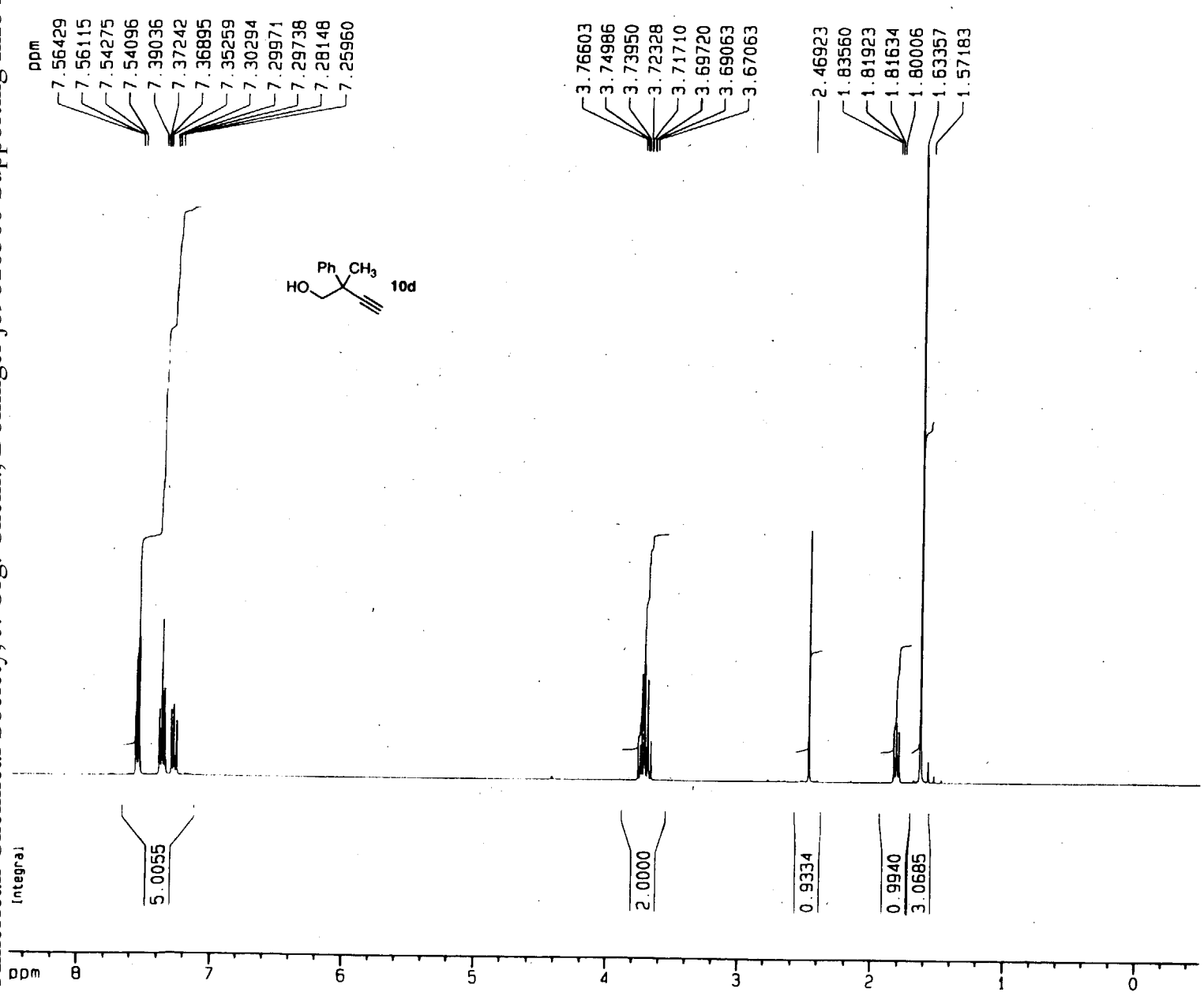
Date_	970617
Time	21.41
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	574.7
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.0000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/



Current Data Parameters

NAME	lmd-3-phmealco
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

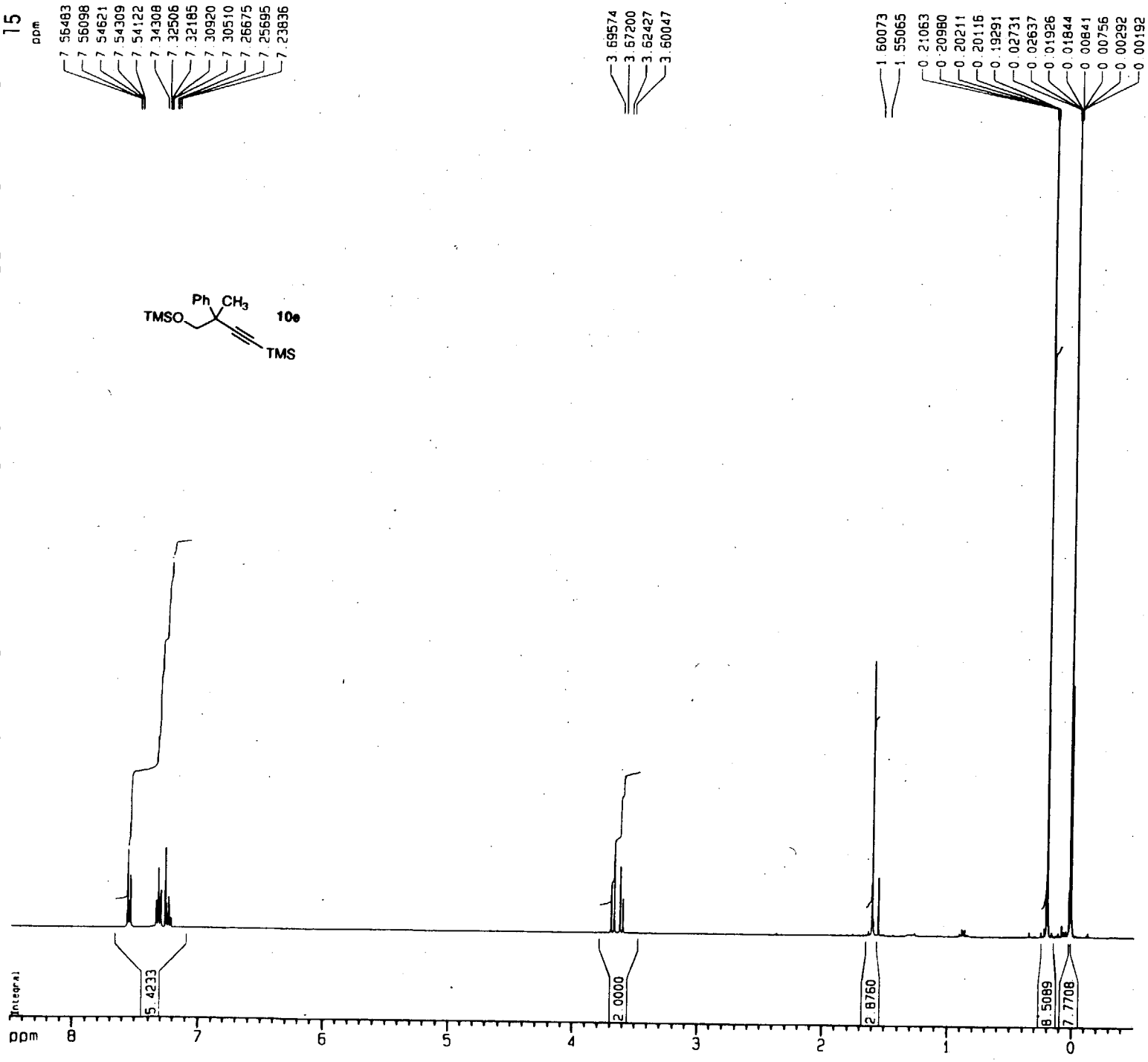
Date_	970609
Time	13.13
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	456.1
DW	60.800 usec
DE	4.50 usec
TE	300.0 K
D1	4.00000000 sec
P1	7.40 usec
DE	4.50 usec
SFO1	400.1324710 MHz
NUC1	1H
PL1	-6.00 dB

F2 - Processing parameters

SI	16384
SF	400.1300094 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm/cm
HZCM	180.05850 Hz/cm



Current Data Parameters

NAME	lmd-3-196-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

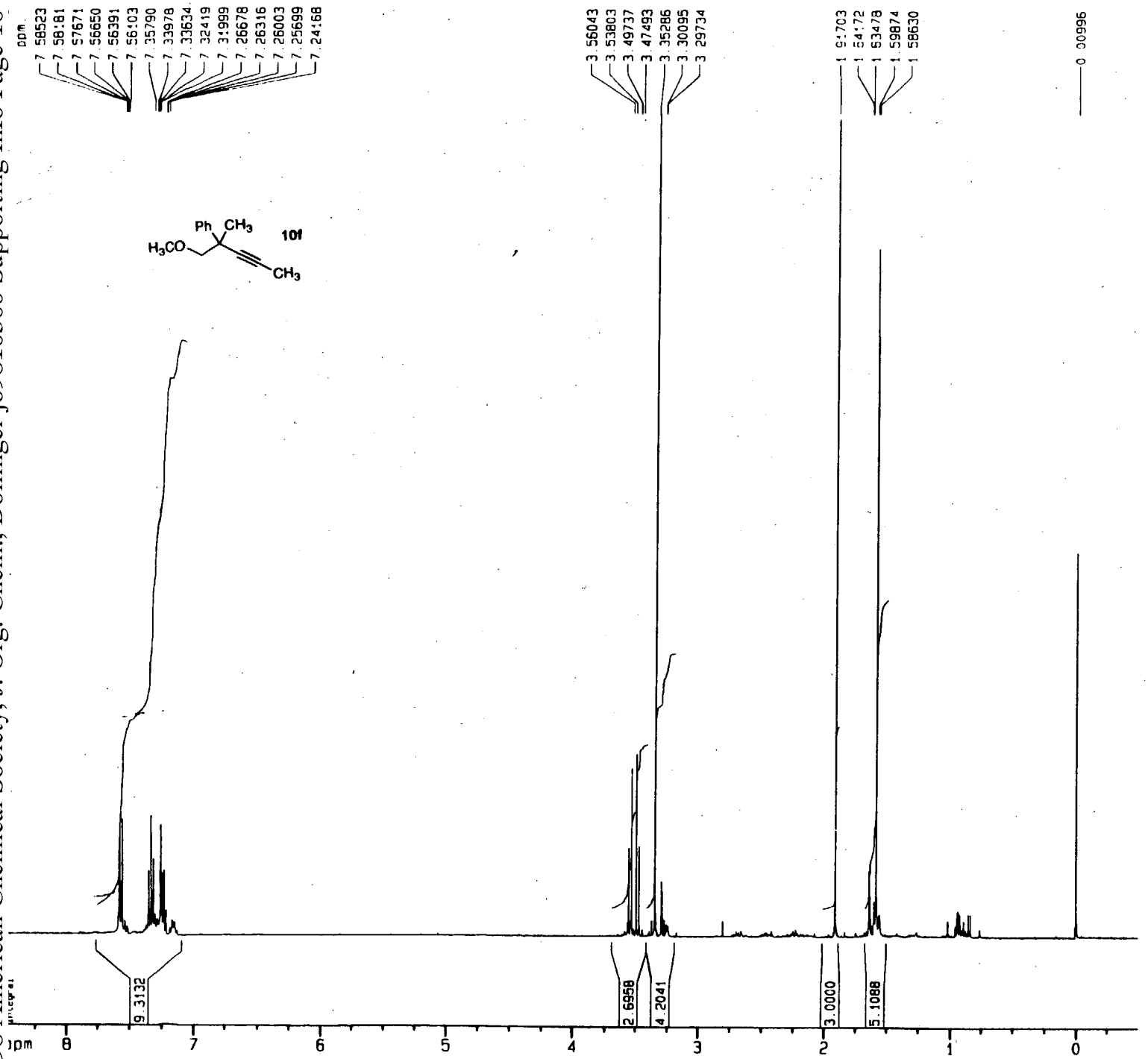
Date_	980306
Time	16.29
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	645.1
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/



Current Data Parameters

NAME	lmd-4-018-3a
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	980416
Time	14.02
INSTRUM	drx400
PROBHD	5 mm QNP 1H
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	574.7
DW	62.400 use
DE	7.14 use
TE	240.0 K
D1	5.00000000 sec
P1	7.40 use
DE	7.14 use
SFO1	400.1320340 MHz
NUC1	1H
PL1	0.00 dB

F2 - Processing parameters

SI	32768
SF	400.1300068 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.45000 ppm
HZCM	180.05850 Hz/