Remarkable effect of 2,2'-bipyridyl: mild and highly chemoselective deprotection of methoxymethyl (MOM) ethers in combination with TMSOTf (TESOTf)-2,2'-bipyridyl

Hiromichi Fujioka*, Ozora Kubo, Kento Senami, Yutaka Minamitsuji, Tomohiro Maegawa

Graduate School of Pharmaceutical Sciences, Osaka University
1-6 Yamada-oka, Suita, Osaka 565-0871, Japan
Fax: (+81)-6-6879-8229
E-mail: fujioka@phs.osaka-u.ac.jp

General techniques

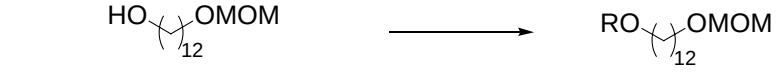
The ^{1}H and ^{13}C NMR spectra were measured by JEOL JNM-ECS 400 or JEOL JNM-AL 300 spectrometers with tetramethylsilane as an internal standard at 20-25 ^{o}C. IR spectra were recorded by Shimadzu FTIR 8400 using a diffuse reflectance measurement of samples dispersed in KBr powder. HRMS spectra were recorded by JEOL LMS-D 300 spectrometers. Merck silica gel 60 was used for column chromatography.

General Procedure for Preparation of MOM-ethers 1a-1d, 1j, and 1k

A solution of an alcohol (1 equiv.), MOMCl (1.5-1.8 equiv.), *i*Pr_2NEt (3-3.6 equiv.) and DMAP (0.1 equiv.) in dry CH_2Cl_2 (0.2-0.5 M) was stirred at $0^{o}C$ to rt under N_2. After disappearance of the alcohol on TLC, the mixture was evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give a MOM-ether. **1a**[1], **1b**[2], **1c**[3], **1d**[4], **1j**[5], and **1k**[6] are known compounds.

Preparation of MOM-ethers 1e-i

MOM-ethers **1e-i** were prepared from known 12-methoxymethoxydodecanol[7].



12-methoxymethoxydodecanol[7] **1e-i**

1e: (R = Ac); A solution of 12-methoxymethoxydodecanol (309 mg, 1.25 mmol), Ac_2O (0.21 ml, 2.25 mmol), *i*Pr_2NEt (0.78 ml, 4.51 mmol), and DMAP (14.6 mg, 0.120 mmol) in dry CH_2Cl_2 (6.3 ml) was stirred at rt under N_2 for 1 h. The reaction mixture was evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **1e** (352 mg, 97%). Eluent; hexanes-AcOEt (12/1). Colorless oil, IR (KBr) 2926, 2855, 1742, 1468, 1238 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 1.27 (16H, brs), 1.53-1.64 (4H, m), 2.05 (3H, s), 3.36 (3H, s), 3.52 (2H, t, *J* = 6.5 Hz), 4.05 (2H, t, *J* = 6.8 Hz), 4.62 (2H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 20.7, 25.7, 26.0, 28.4, 29.0, 29.2, 29.3 (3C), 29.4, 29.5, 54.8, 64.3, 67.6, 96.1, 170.8; HRMS (FAB) calcd for $C_{16}H_{33}O_4$ (M^++H) 289.2379, found 289.2377.

1f: (R = Bz); A solution of 12-methoxymethoxydodecanol (330 mg, 1.34 mmol), BzCl (0.28 ml, 2.41 mmol), *i*Pr_2NEt (0.82 ml, 4.82 mmol), and DMAP (16.3 mg, 0.133 mmol) in dry CH_2Cl_2 (6.7 ml) was stirred at rt under N_2 for 17.5 h. The mixture was evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **1f** (440 mg, 94%). Eluent; hexanes-banzene (1/10) to hexanes-AcOEt (5/1). Colorless oil, IR (KBr) 2930, 2855, 1713, 1468, 1279 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 1.28-1.43 (16H, m), 1.54-1.61 (2H, m), 1.71-1.82 (2H, m), 3.36 (3H, s), 3.52 (2H, t, *J* = 6.8 Hz), 4.31 (2H, t, *J* = 6.8 Hz), 4.62 (2H, s), 7.40-7.46 (2H, m), 7.52-7.59 (1H, m), 8.03-8.07 (2H, m); ^{13}C NMR (101 MHz, $CDCl_3$) δ 25.9, 26.1, 28.6, 29.2, 29.3, 29.4, 29.5 (2C), 29.7, 54.9, 65.0, 67.7, 96.3, 128.2, 129.4, 130.4, 132.7, 166.5; HRMS (FAB) calcd for $C_{21}H_{35}O_4$ (M^++H) 351.2535, found 351.2528.

1g: (R = Bn); To a solution of 12-methoxymethoxydodecanol (308 mg, 1.25 mmol) in dry THF (3.1 ml), BnBr (0.22 ml, 1.88 mmol) and NaH (76.8 mg, 1.92 mmol) were added successively at rt under N_2 and stirred for 20 h. H_2O was added to the reaction mixture and resulting solution was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, filtered, and evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **1g** (364 mg, 86%). Eluent; hexanes-AcOEt (15/1). Colorless oil, IR (KBr) 2926, 2853, 1454, 1360, 1209 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.26 (16H, brs), 1.56-1.53 (4H, m), 3.36 (3H, s), 3.44-3.54 (4H, m), 4.50 (2H, s), 4.62 (2H, s), 7.26-7.35 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.1 (2C), 29.4 (2C), 29.5, 29.7 (2C), 54.9, 67.7, 70.4, 72.7, 96.2, 127.3, 127.5, 128.2, 138.6; HRMS (FAB) calcd for $C_{21}H_{36}NaO_3$ (M^++Na) 359.2562, found 359.2563.

1h: (R = TBS); A solution of 12-methoxymethoxydodecanol (308 mg, 1.25 mmol), TBSCl (332 mg, 2.21 mmol), *i*Pr_2NEt (0.78 ml, 4.50 mmol), and DMAP (15.0 mg, 0.123 mmol) in dry CH_2Cl_2 (6.3 ml) was stirred at rt under N_2 for 4 h. The mixture was evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **1h** (428 mg, 95%). Eluent; hexanes-AcOEt (25/1). Colorless oil, IR (KBr) 2856, 2251, 1794, 1470, 1385 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.04 (6H, s), 0.89 (9H, s), 1.27 (16H, brs), 1.47-1.63 (4H, m), 3.36 (3H, s), 3.52 (2H, t, *J* = 6.8 Hz), 3.60 (2H, t, *J* = 6.8 Hz), 4.62 (2H, s); ^{13}C NMR (101 MHz, $CDCl_3$) δ -5.3, 18.4, 25.8, 26.0, 26.2, 29.4, 29.6 (3C), 29.7, 32.9, 55.1, 63.3, 67.8, 96.3; HRMS (FAB) calcd for $C_{20}H_{45}O_3Si$ (M^++H) 361.3138, found 361.3162.

1i: (R = Tr); A solution of 12-methoxymethoxydodecanol (232 mg, 0.942 mmol), TrCl (485 mg, 1.74 mmol), *i*Pr_2NEt (0.60 ml, 3.51 mmol) and TBAI (35.0 mg, 0.095 mmol) in dry CH_2Cl_2 (1.0 ml) was stirred at rt under N_2 for 24 h. The mixture was evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **1i** (448 mg, 97%). Eluent; hexanes-AcOEt (15/1). Colorless oil, IR (KBr) 2926, 2853, 1448, 1217, 1150 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.24 (16H, brs), 1.54-1.64 (4H, m), 3.03 (2H, t, *J* = 6.6 Hz), 3.36 (3H, s), 3.51 (2H, t, *J* = 6.6 Hz), 4.62 (2H, s), 7.19-7.32 (9H, m), 7.43-7.46 (6H, m); ^{13}C NMR (101 MHz, $CDCl_3$) δ 26.2 (2C), 29.4, 29.5, 29.6 (2C), 29.7, 30.0, 55.1, 63.7, 67.9, 86.2, 96.4, 126.7, 127.6, 128.7, 144.5; HRMS (EI) calcd for $C_{33}H_{44}O_3$ (M^+) 488.3290, found 488.3290.

General Procedure for Deprotection of MOM-ether (1a) by TMSOTf–pyridines Combination (Table 1)

TMSOTf (2.0 equiv.) was added dropwise to a solution of MOM-ether (**1a**) (1.0 equiv.) and pyridine derivatives (3.0 equiv.) in CH_2Cl_2 (0.2 M) at 0 ^oC under N_2. The reaction mixture was stirred for 30 min at 0 ^oC. H_2O and Et_2O was added to the reaction mixture and the resulting solution was stirred 24 h at rt. The mixture was extracted with CH_2Cl_2. The organic layer was washed twice with 3.5% HCl aq. (for the removal of pyridines) and with sat. $NaHCO_3$ aq. The combined organic layer was dried over Na_2SO_4, filtered, and evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **2a. 2a** is commercially available.

General Procedure for Deprotection of MOM-ethers in combination with TMSOTf (TESOTf)−2,2'-bipyridyl (Table 2)

TMSOTf (TESOTf) (2.0 equiv.) was added dropwise to a solution of MOM-ether (1.0 equiv.) and 2,2'-bipyridyl (3.0 equiv.) in CH_2Cl_2 (0.2 M) at 0 ^oC under N_2. The reaction mixture was stirred for 30 min at 0 ^oC. After disappearance of MOM-ether on TLC, H_2O and Et_2O was added to the reaction mixture and the resulting solution was stirred until disappearance of the pyridinium salt (high polar compound on TLC). The mixture was extracted with CH_2Cl_2. The organic layer was washed twice with 3.5% HCl aq. and with sat $NaHCO_3$ aq. The combined organic layer was dried over Na_2SO_4, filtered, and evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give an alcohol. **2e**[8a], **2f**[8b], **2g**[8c], **2h**[8d], and **2i**[8e] are known compounds. **2b**, **2c**, **2d**, and **2j** are commercially available.

Entry 1: According to the general procedure, the treatment of **1a** (31.0 mg, 0.153 mmol) with 2,2'-bipyridyl (71.8 mg, 0.460 mmol) and TMSOTf (55 μL, 0.306 mmol) gave **2a** (20.7 mg, 85%) for 6 h. Eluent; hexanes-AcOEt (4/1).

Entry 2: According to the general procedure, the treatment of **1a** (31.4 mg, 0.155 mmol) with 2,2'-bipyridyl (73.0 mg, 0.467 mmol) and TESOTf (70 μL, 0.310 mmol) gave **2a** (22.1 mg, 90%) for 6h. Eluent; hexanes-AcOEt (4/1).

Entry 3: According to the general procedure, the treatment of **1b** (40.5 mg, 0.200 mmol) with 2,2'-bipyridyl (93.7 mg, 0.600 mmol) and TMSOTf (72 μL, 0.400 mmol) gave **2b** (28.8 mg, 91%) for 2 h. Eluent; hexanes-AcOEt (4/1).

Entry 4: According to the general procedure, the treatment of **1b** (46.0 mg, 0.227 mmol) with 2,2'-bipyridyl (106.2 mg, 0.680 mmol) and TESOTf (103 μL, 0.454 mmol) gave **2b** (29.3 mg, 81%) for 2 h. Eluent; hexanes-AcOEt (30/1 to 4/1).

Entry 5: According to the general procedure, the treatment of **1c** (41.7 mg, 0.200 mmol) with 2,2'-bipyridyl (93.7 mg, 0.600 mmol) and TMSOTf (72 μL, 0.400 mmol) gave **2c** (32.4 mg, 99%). Eluent; hexanes- AcOEt (3/1).

Entry 6: According to the general procedure, the treatment of **1c** (44.6 mg, 0.214 mmol) with 2,2'-bipyridyl (100.4 mg, 0.643 mmol) and TESOTf (97 μL, 0.428 mmol) gave **2c** (32.2 mg,

92%) for 2 h. Eluent; hexanes-AcOEt (4/1).

Entry 7: According to the general procedure, the treatment of **1d** (41.1 mg, 0.231 mmol) with 2,2'-bipyridyl (108.1 mg, 0.692 mmol) and TMSOTf (83 μL, 0.462 mmol) gave **2d** (25.1 mg, 81%) for 10 h. Eluent; hexanes- AcOEt (3/1).

Entry 8: According to the general procedure, the treatment of **1d** (30.8 mg, 0.173 mmol) with 2,2'-bipyridyl (81.1 mg, 0.519 mmol) and TESOTf (78 μL, 0.346 mmol) gave **2d** (12.8 mg, 55%). Eluent; hexanes-AcOEt (2/1).

Entry 9: According to the general procedure, the treatment of **1e** (44.0 mg, 0.153 mmol) with 2,2'-bipyridyl (72.1 mg, 0.462 mmol) and TMSOTf (55 μL, 305 mmol) gave **2e** (33.3 mg, 89%) for 5 h. Eluent; hexanes-AcOEt (3/1 to 2/1).

Entry 10: According to the general procedure, the treatment of **1e** (48.2 mg, 0.167 mmol) with 2,2'-bipyridyl (78.3 mg, 0.501 mmol) and TESOTf (76 μL, 0.334 mmol) gave **2e** (37.3 mg, 91%) for 5 h. Eluent; hexanes-AcOEt (2/1).

Entry 11: According to the general procedure, the treatment of **1f** (36.9 mg, 0.105 mmol) with 2,2'-bipyridyl (49.3 mg, 0.316 mmol) and TMSOTf (38 μL, 0.210 mmol) gave **2f** (30.0 mg, 93%) for 5 h. Eluent; hexanes-AcOEt (4/1).

Entry 12: According to the general procedure, the treatment of **1f** (59.5 mg, 0.170 mmol) with 2,2'-bipyridyl (79.5 mg, 0.509 mmol) and TESOTf (77 μL, 0.340 mmol) gave **2f** (44.5 mg, 86%) for 5 h. Eluent; hexanes-AcOEt (10/1 to 2/1).

Entry 13: According to the general procedure, the treatment of **1g** (35.5 mg, 0.105 mmol) with 2,2'-bipyridyl (49.4 mg, 0.316 mmol) and TMSOTf (38 μL, 0.210 mmol) gave **2g** (26.5 mg, 86%) for 5 h. Eluent; hexanes-AcOEt (4/1).

Entry 14: According to the general procedure, the treatment of **1g** (61.3 mg, 0.182 mmol) with 2,2'-bipyridyl (85.5 mg, 0.547 mmol) and TESOTf (82 μL, 0.364 mmol) gave **2g** (46.5 mg, 87%) for 5 h. Eluent; hexanes-AcOEt (3/1).

Entry 15: According to the general procedure, the treatment of **1h** (30.5 mg, 0.085 mmol) with 2,2'-bipyridyl (39.6 mg, 0.254 mmol) and TMSOTf (31 μL, 0.170 mmol) gave **2h** (23.7 mg, 88%) for 5 h. Eluent; hexanes-AcOEt (8/1).

Entry 16: According to the general procedure, the treatment of **1h** (56.3 mg, 0.156 mmol) with 2,2'-bipyridyl (73.4 mg, 0. 470 mmol) and TESOTf (71 μL, 0.312 mmol) gave **2h** (44.8 mg, 91%) for 4 h. Eluent; hexanes-AcOEt (5/1).

Entry 17: According to the general procedure, the treatment of **1i** (51.6 mg, 0.106 mmol) with 2,2'-bipyridyl (49.5 mg, 0.317 mmol) and TMSOTf (38 μL, 0.210 mmol) gave **2i** (43.5

mg, 92%). Eluent; hexanes-AcOEt (4/1).

Entry 18: According to the general procedure, the treatment of **1i** (55.0 mg, 0.113 mmol) with 2,2'-bipyridyl (52.6 mg, 0.337 mmol), TESOTf (51 μL, 0.226 mmol) and CH_2Cl_2 (0.23ml) gave **2i** (40.8 mg, 82%) for 2.5 h. Eluent; hexanes-AcOEt (3/1).

Entry 19: According to the general procedure, the treatment of **1j** (25.6 mg, 0.152 mmol) with 2,2'-bipyridyl (72.2 mg, 0.462 mmol) and TMSOTf (55 μL, 0.304 mmol) gave **2j** (3.6 mg, 19%) for 24 h. Eluent; hexanes- AcOEt (4/1 to 2/1).

Entry 20: According to the general procedure, the treatment of **1j** (24.3 mg, 0.144 mmol) with 2,2'-bipyridyl (67.4 mg, 0.432 mmol) and TESOTf (65 μL, 0.288 mmol) did not react for 24 h.

Selective deprotection of aliphatic MOM group in the presence of phenolic MOM group (Scheme 2)

According to the general procedure for deprotection of MOM-ethers in Table 2, the treatment of **1k**[6] (39.4 mg, 0.164 mmol) with 2,2'-bipyridyl (76.7 mg, 0.491 mmol) and TESOTf (74 μL, 0.328 mmol) gave **2k**[9] (27.3 mg, 85%) for 14 h. Eluent; hexanes- AcOEt (1/1).

Characterization of collidinium intermediate from MOM-ether (1a) (Scheme 3)

White solid, M.P. 63.7-65.0 ^oC; IR (KBr) 3568, 2926, 1641, 1259, 1157 cm^{-1}; ^1H NMR (400 MHz, CD_2Cl_2) δ 0.88 (3H, t, J = 6.9 Hz), 1.26 (14H, brs), 1.57-1.64 (2H, m), 2.56 (3H, s), 2.88 (6H, s), 3.62 (2H, t, J = 6.6 Hz), 5.78 (2H, s), 7.47 (2H, s); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 14.3, 21.2, 22.1, 23.1, 26.3, 29.6, 29.7 (2C), 29.9 (2C), 32.3, 70.8, 80.9, 128.8, 155.7, 160.5

Direct conversion to other methylether group from MOM-ether (1i) (Scheme 4)

3a: TMSOTf (83 μL, 0.458 mmol) was added dropwise to a solution of MOM-ether **1i** (112 mg, 0.229 mmol) and 2,2'-bipyridyl (107 mg, 0.687 mmol) in CH_2Cl_2 (0.46 ml) at 0 ^oC under N_2. The reaction mixture was stirred for 30 min at 0^oC. After disappearance of MOM-ether on TLC, BnOH (0.12 ml, 1.15 mmol) was added to the reaction mixture and the resulting

solution was stirred for 20 h at rt. Sat. $NaHCO_3$ aq. was added and the reaction mixture was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, filtered, and evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **3a** (95.1 mg, 73%). Eluent; hexanes-benzene (1/3). colorless oil, IR (KBr) 3059, 2930, 2855, 1448, 1265 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 1.25-1.31 (16H, m), 1.56-1.64 (4H, m), 3.03 (2H, t, J = 6.6 Hz), 3.58 (2H, t, J = 6.6 Hz), 4.60 (2H, s), 4.76 (2H, s), 7.19-7.46 (20H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.2 (2C), 29.4, 29.5, 29.7, 30.0, 63.6, 68.0, 69.1, 86.2, 94.5, 126.7, 127.6 (2C), 127.8, 128.3, 128.6, 137.9, 144.5; HRMS (EI) calcd for $C_{39}H_{48}O_3$ (M^+) 564.3603, found 564.3601.

3b: TMSOTf (88 μL, 0.484 mmol) was added dropwise to a solution of MOM-ether **1i** (119 mg, 0.242 mmol) and 2,2'-bipyridyl (114 mg, 0.728 mmol) in CH_2Cl_2 (0.48 ml) at 0 ^oC under N_2. The reaction mixture was stirred for 30 min at 0^oC. After disappearance of MOM-ether on TLC, $TMSCH_2CH_2OH$ (0.17 ml, 1.21 mmol) was added to the reaction mixture and the resulting solution was stirred for 6 h at rt. Sat. $NaHCO_3$ aq. was added and the reaction mixture was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, filtered, and evaporated in vacuo. The residue was purified by flash SiO_2 column chromatography to give **3b** (108 mg, 78%). Eluent; hexanes-benzene (1/3). colorless oil, IR (KBr) 2926, 2855, 1489, 1448, 1250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.02 (9H, s), 0.95 (2H, t, J = 8.4 Hz), 1.24-1.31 (16H, m), 1.53-1.66 (4H, m), 3.03 (2H, t, J = 6.5 Hz), 3.52 (2H, t, J = 6.5 Hz), 3.61 (2H, t, J = 8.4 Hz), 4.67 (2H, s), 7.19-7.31 (9H, m), 7.43-7.46 (6H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ -1.4, 18.1, 26.2, 29.4, 29.5 (2C), 29.6 (2C), 29.7, 30.0, 63.6, 64.8, 67.8, 86.2, 94.7, 126.7, 127.6, 128.6, 144.5; HRMS (EI) calcd for $C_{37}H_{54}O_3Si$ (M^+) 574.3842, found 574.3841.

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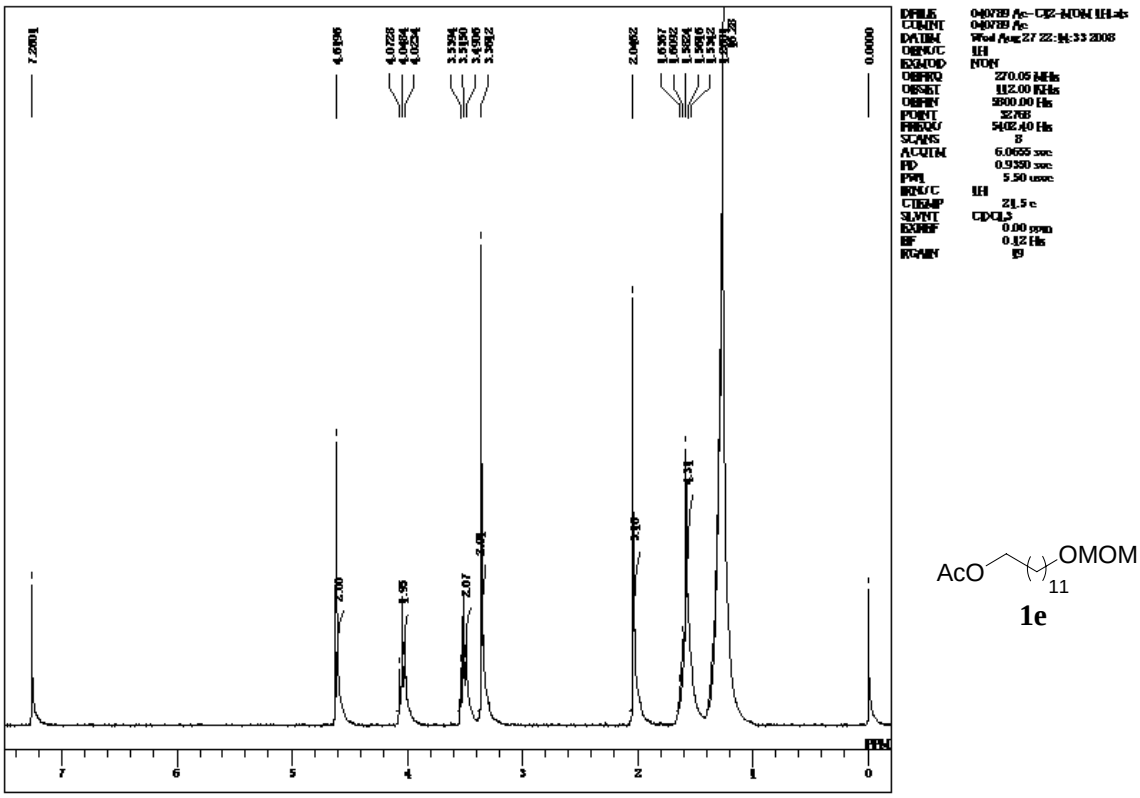
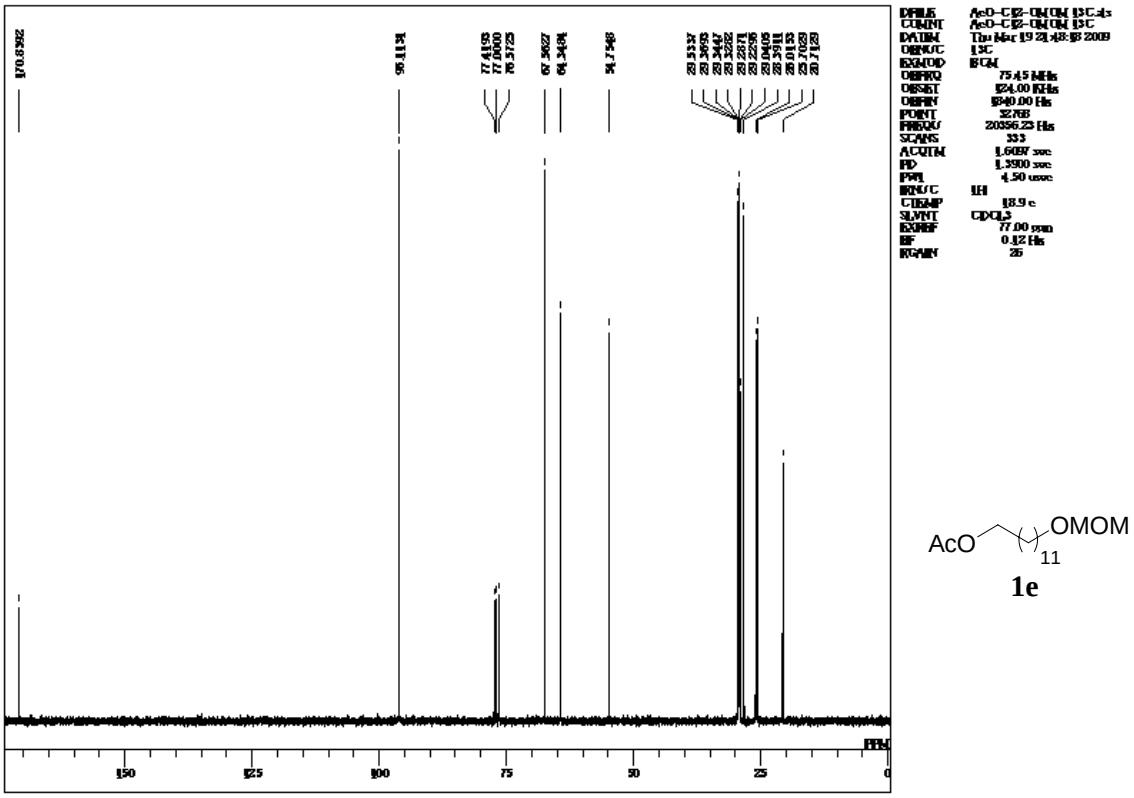
^{1}H and ^{13}C NMR of MOM ether **1e**





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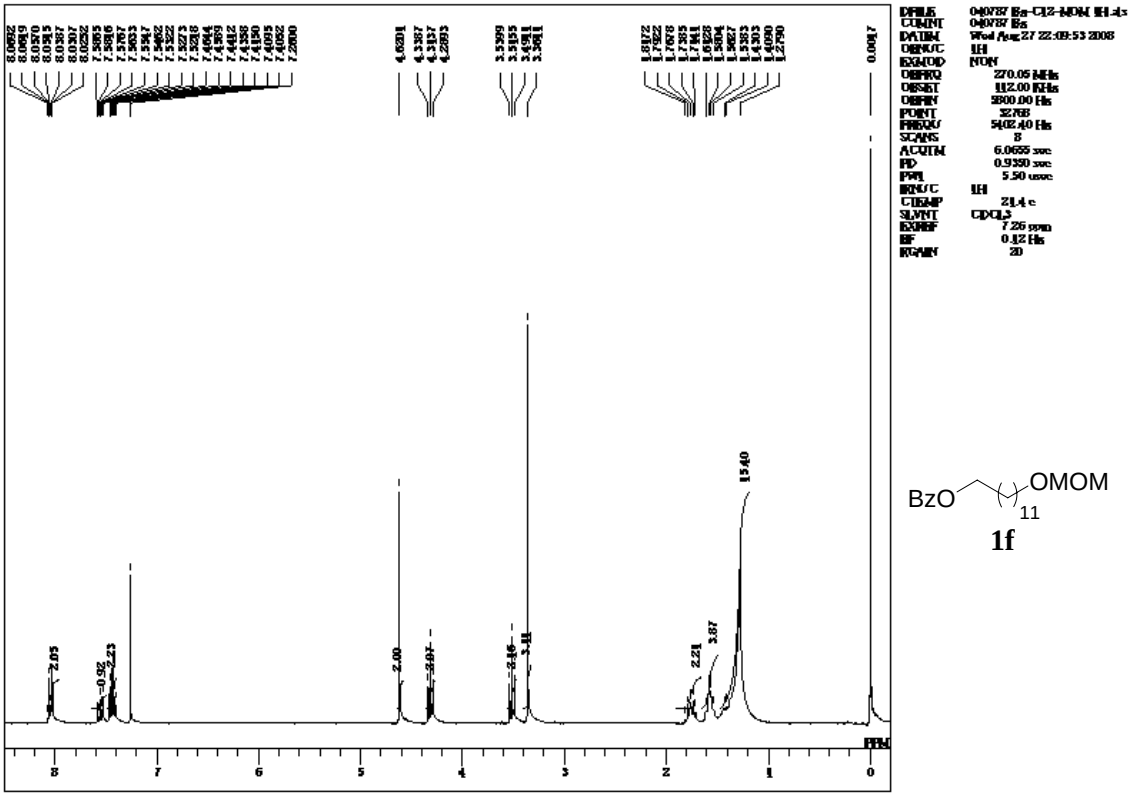
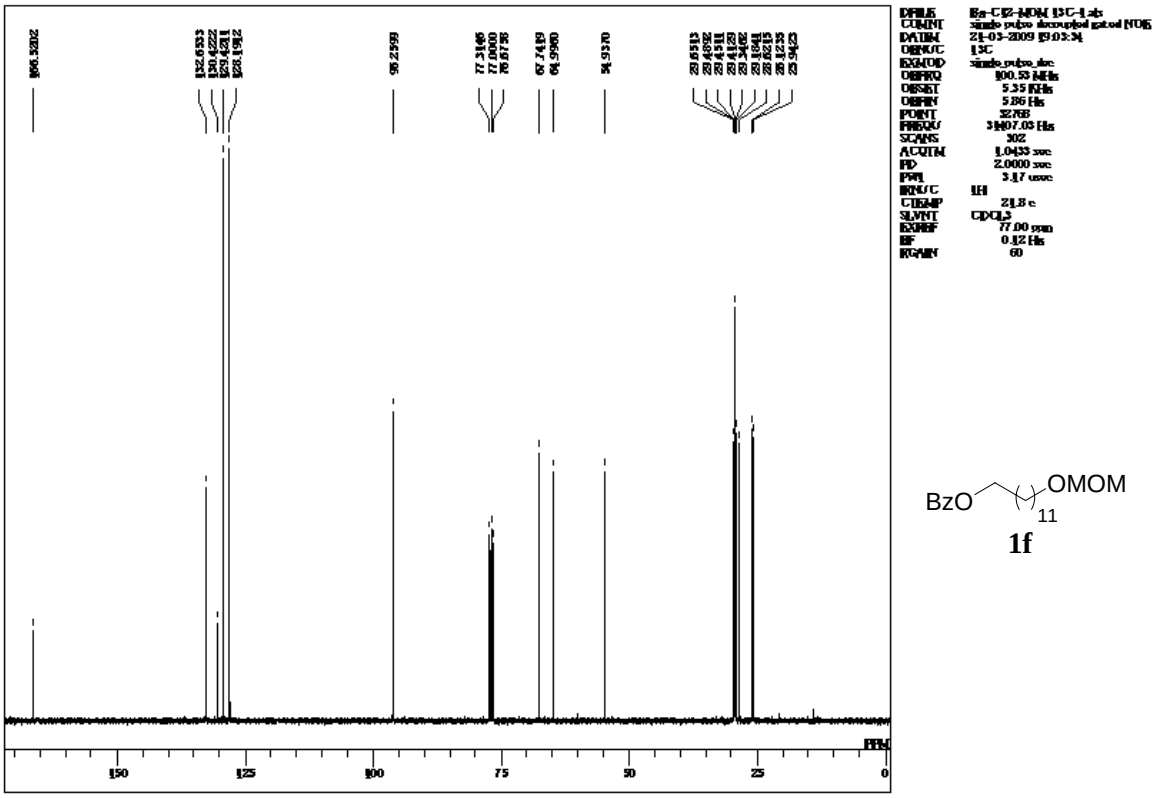
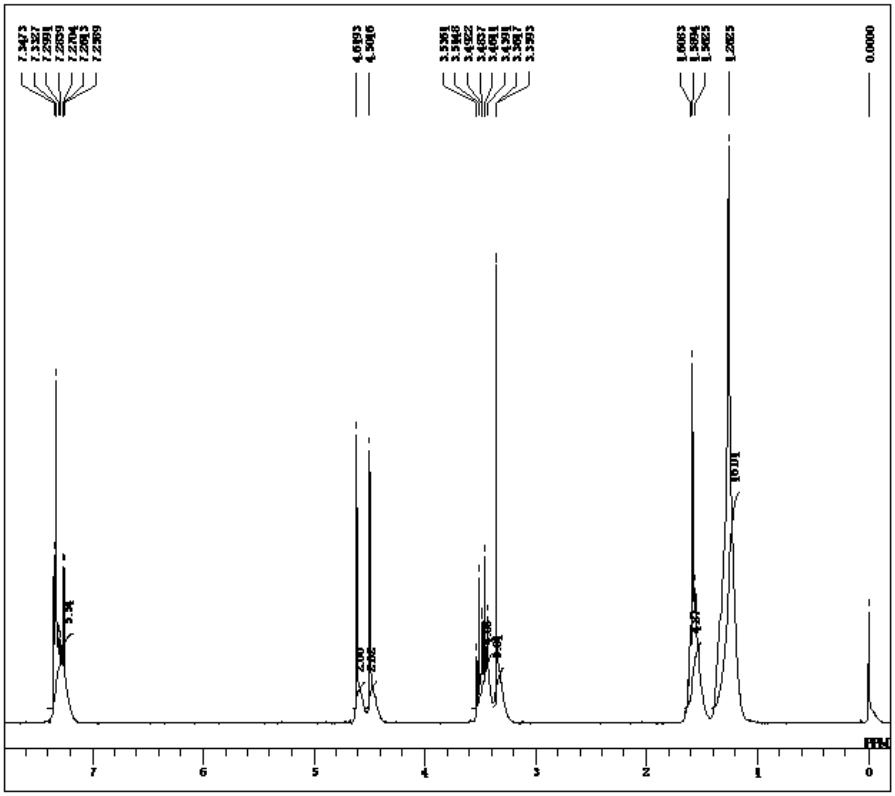
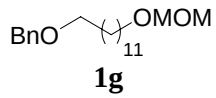


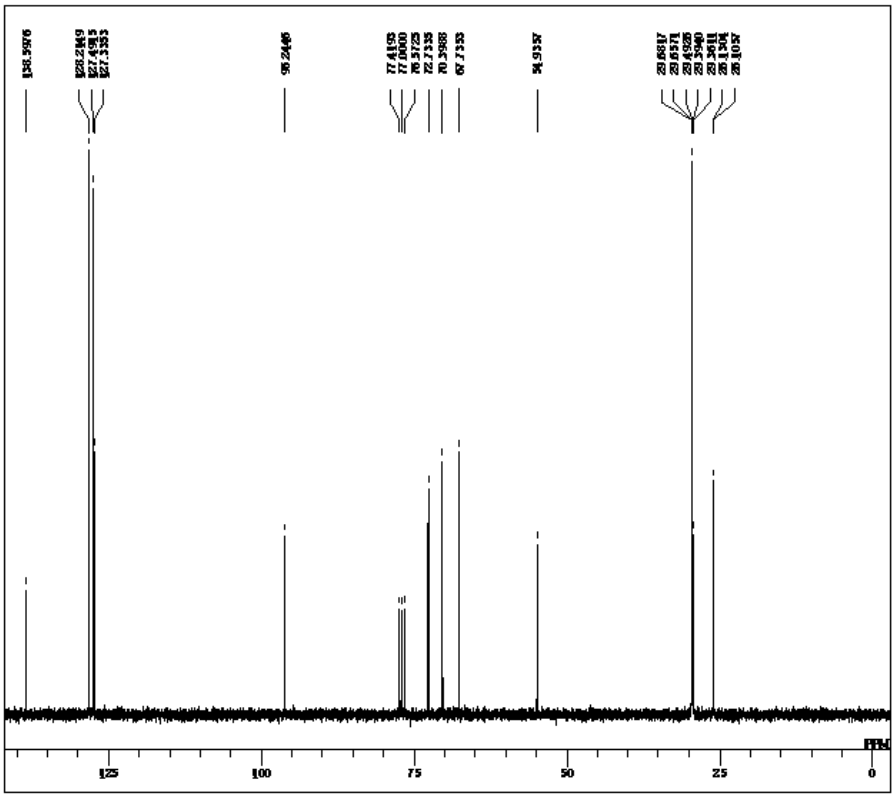


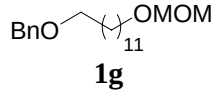
^{1}H and ^{13}C NMR of MOM ether **1g**





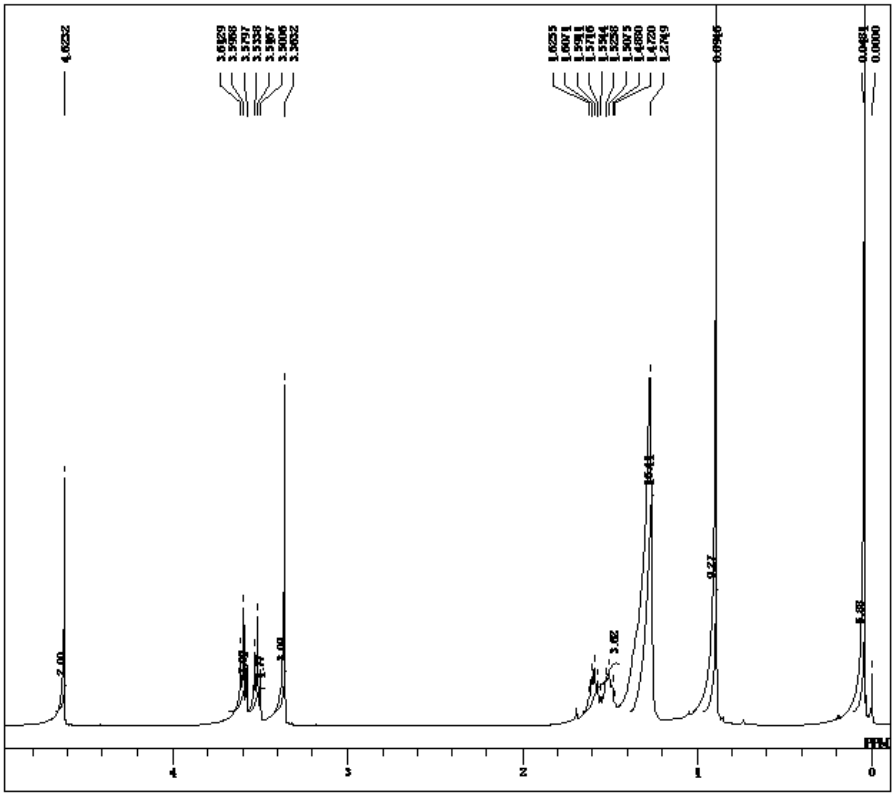
1g

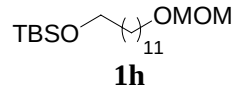




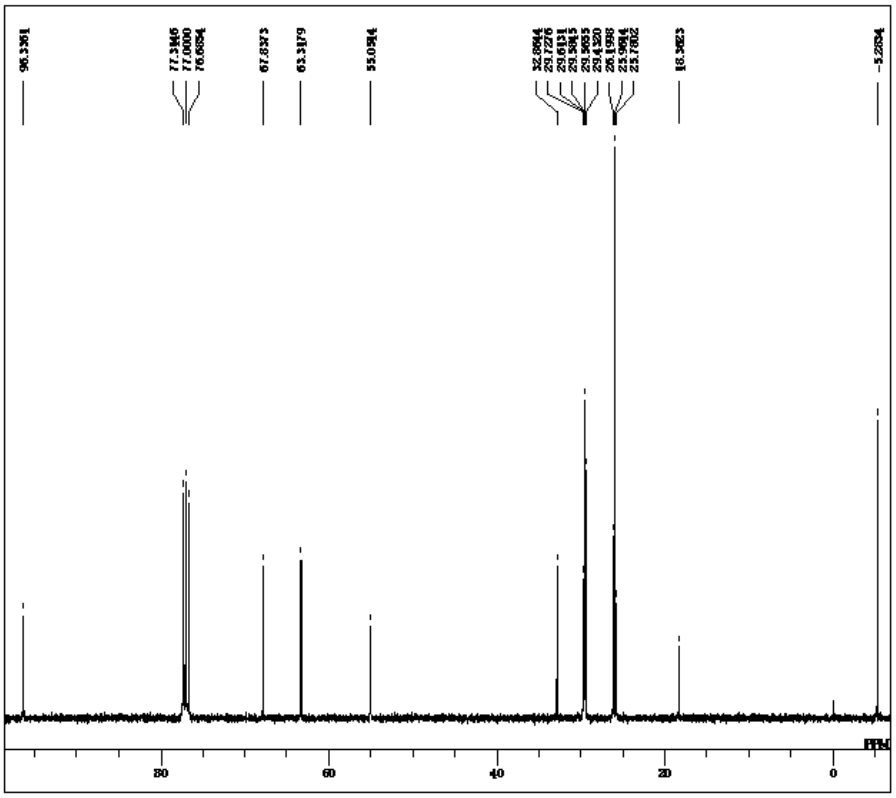
1g

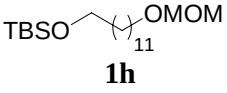
^{1}H and ^{13}C NMR of MOM ether **1h**





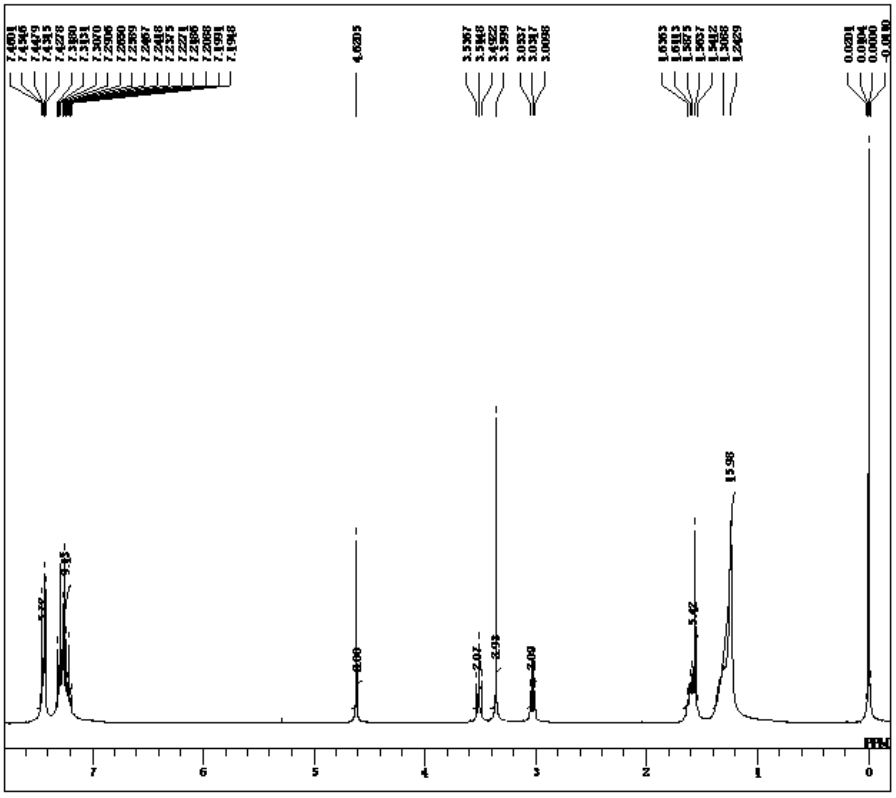
1h

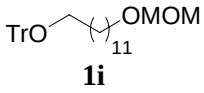




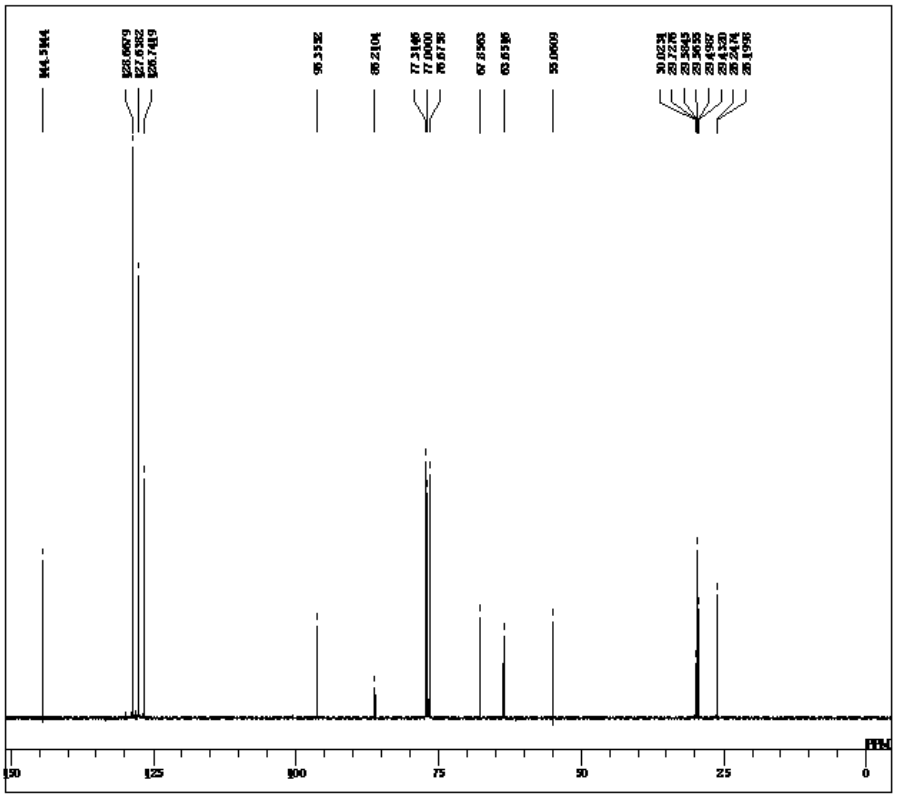
1h

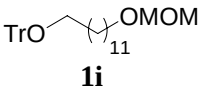
^{1}H and ^{13}C NMR of MOM ether **1i**





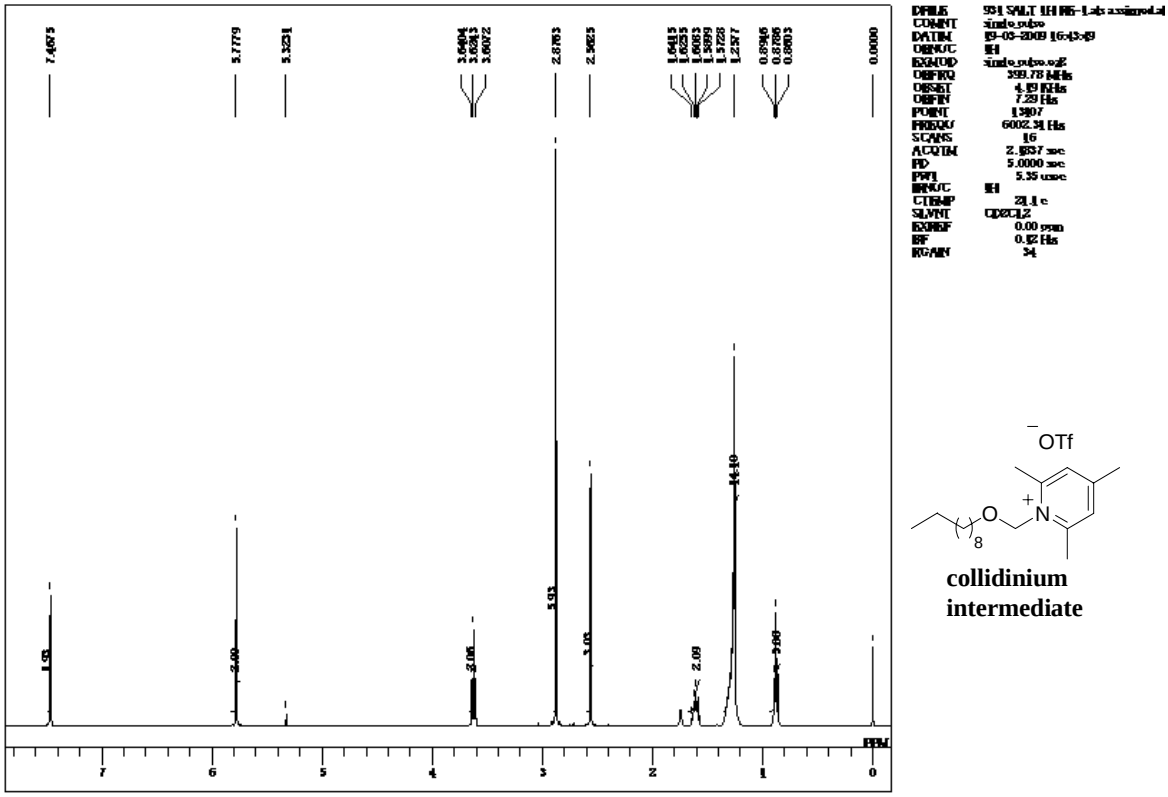
1i



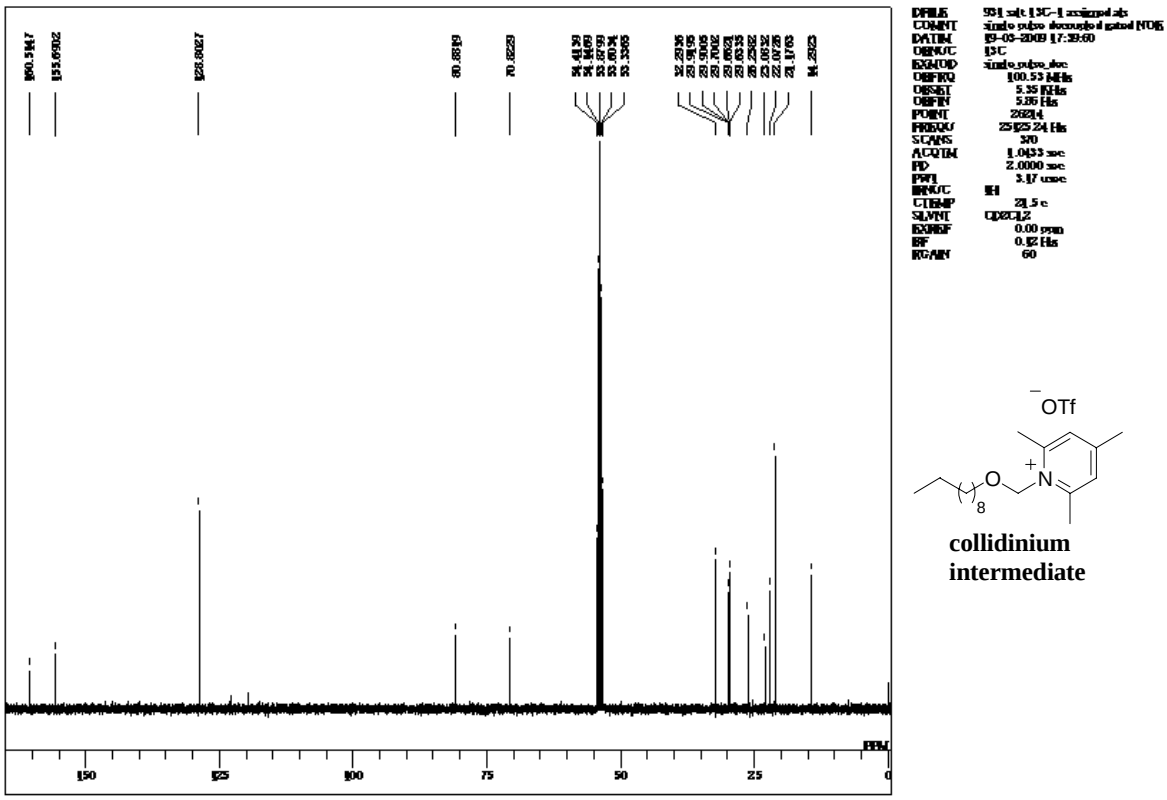


1i

^{1}H and ^{13}C NMR of collidinium intermediate

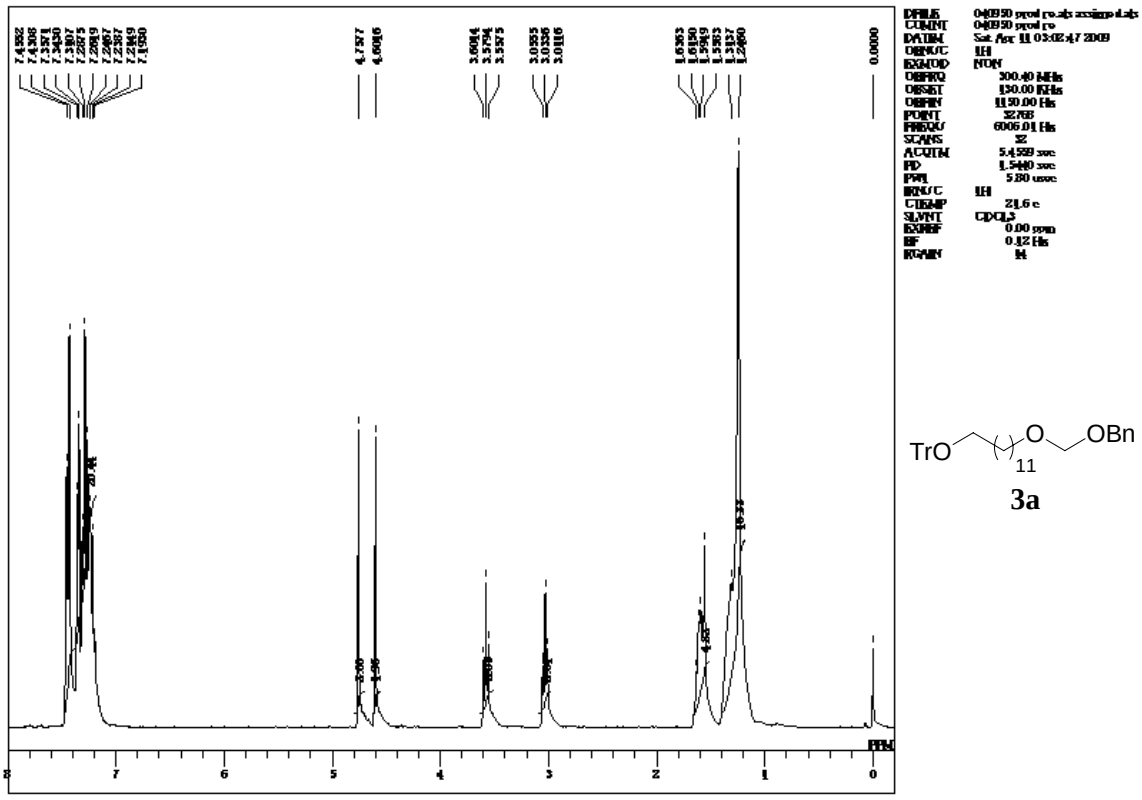


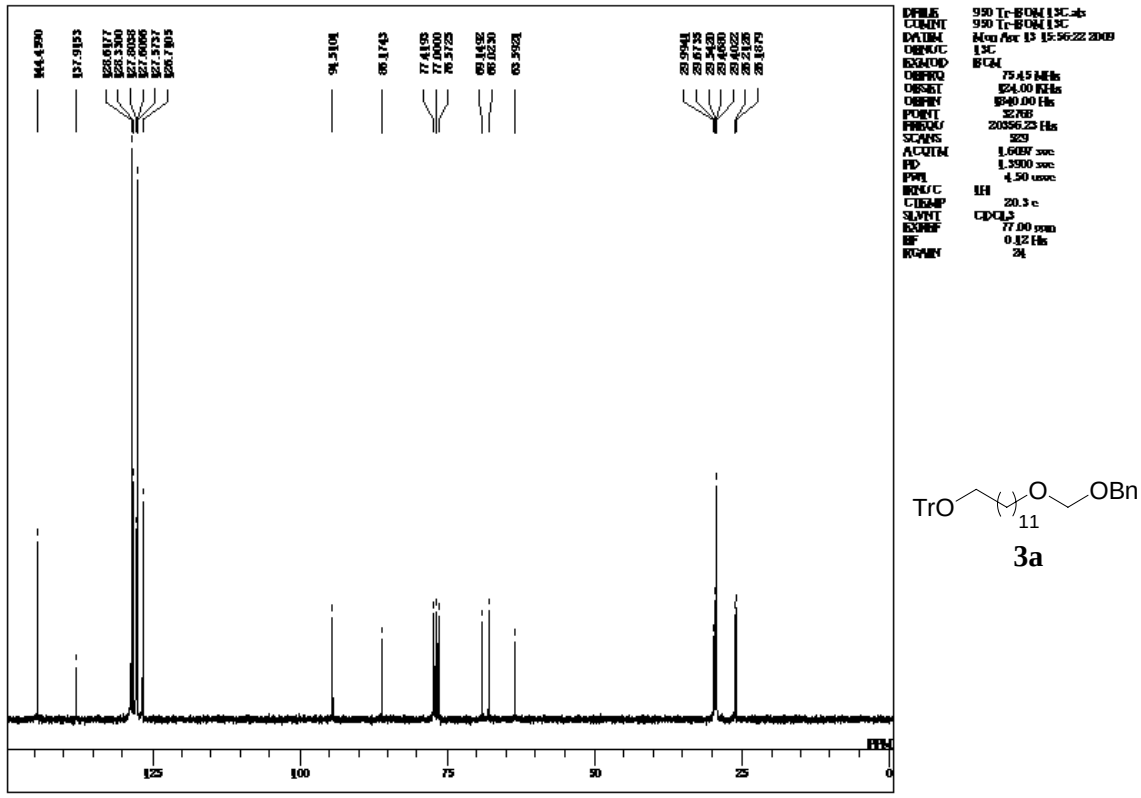
collidinium intermediate



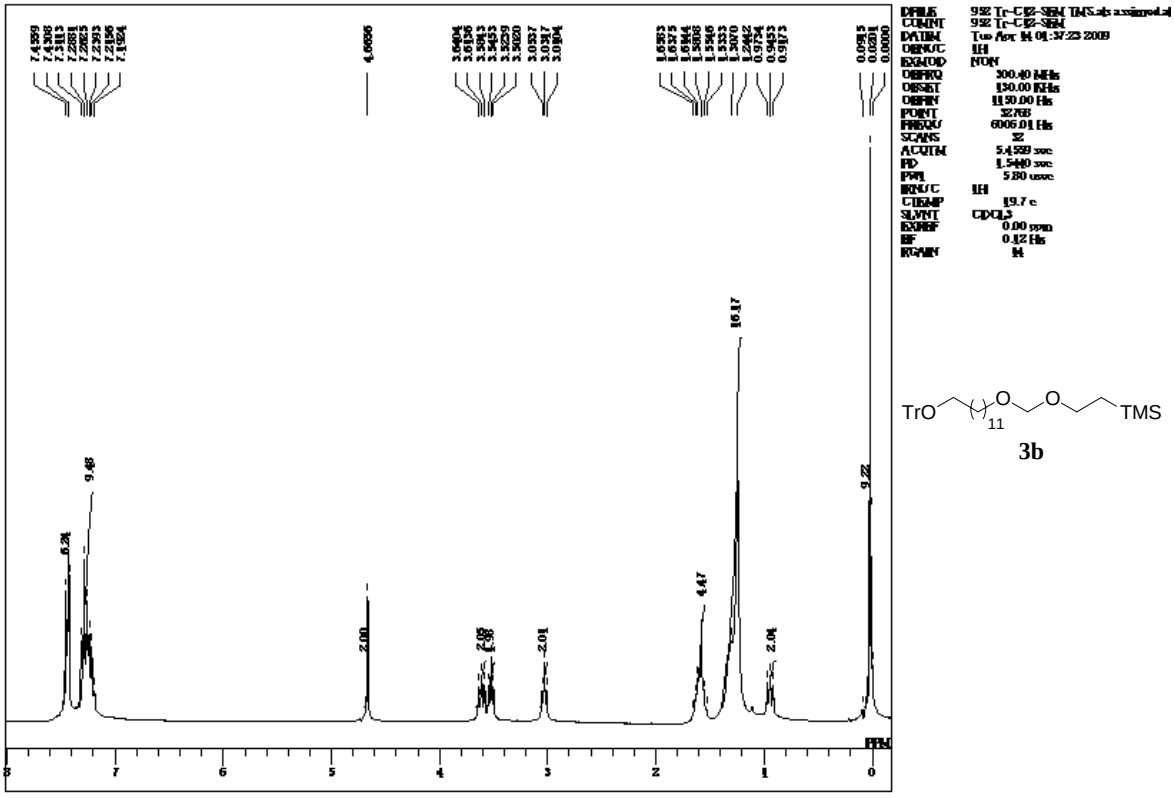
collidinium intermediate

^{1}H and ^{13}C NMR of **3a**

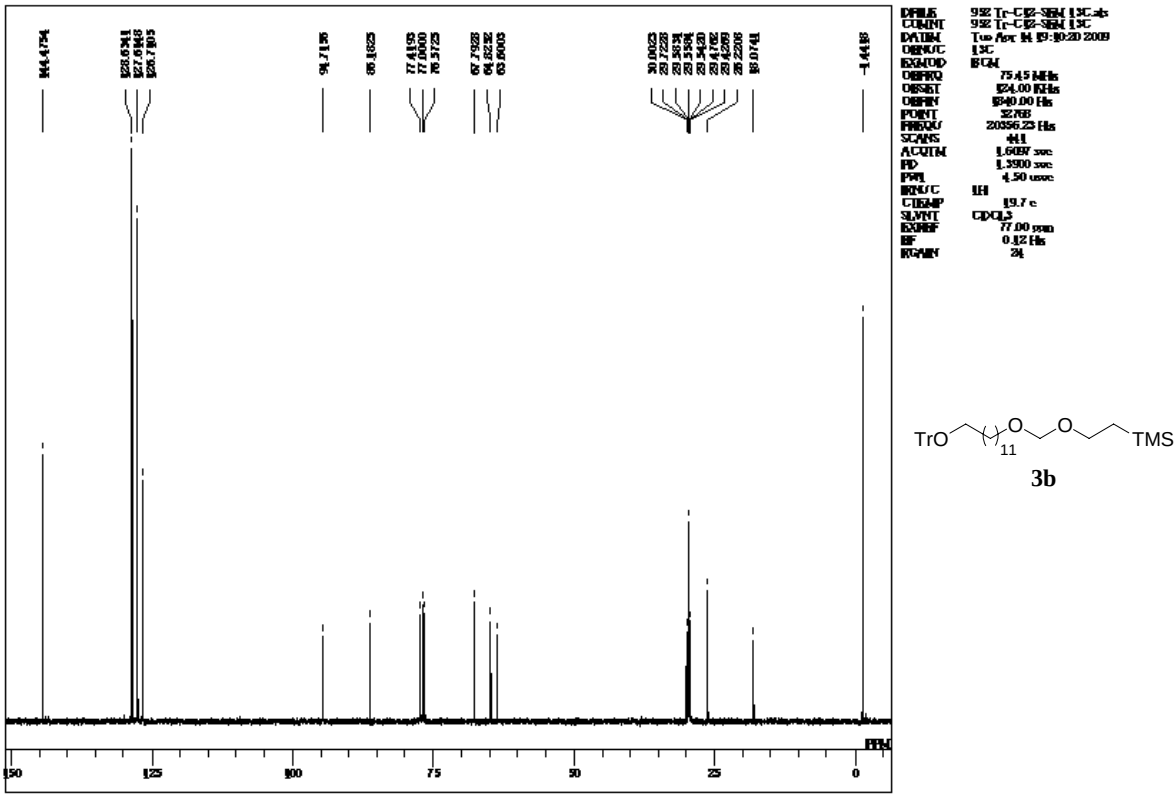




^{1}H and ^{13}C NMR of **3b**



3b



3b