Electronic Supplementary Material (ESI) for RSC Advances.

Atom-economical synthesis of 3,3,3-trifluoropropanal dialkyl acetals through Pd/C catalyzed acetalization of 3,3,3-trifluoropropene

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Contents

1. General Information

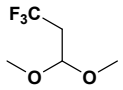
Unless otherwise mentioned, solvents and reagents were purchased from commercial sources and used as received. ^{1}H NMR spectra at 500 MHz, ^{13}C NMR spectra at 125 MHz and ^{19}F NMR spectra at 470 MHz were obtained on a Bruker-AV500 spectrometer. ^{1}H NMR and ^{13}C NMR were recorded using tetramethylsilane (TMS) in the solvent of $CDCl_3$ as the internal standard (^{1}H NMR: TMS at 0.00 ppm, $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). Data for ^{1}H, ^{13}C and ^{19}F NMR were recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, br = broad). Infrared spectra were recorded on a Bruker TENSOR27 spectrometer using KBr pellets. The high-resolution mass spectrometry(MS) spectra were acquired in electrospray ionization (ESI) positive mode using a Bruker Maxis UHR-TOF mass spectrometer. Mass spectra using electron impact (EI) techniques were recorded on a Thermo ITQ700 spectrometer. Gas chromatograph were recorded on Shimadzu GC-2014C.

2. General method for the synthesis of 3,3,3-trifluoropropanal dialkyl acetals (3a-o)

A 150-mL autoclave was charged with 1 %mol of 5% Pd/C and 2 %mol of $CuCl_2$ in 20 mL of alcohol, and TFP gas (30 mmol) was introduced at -30℃. Then, the autoclave was filled with 1 MPa oxygen. The mixture was stirred magnetically at 120℃ for 8h. After the reaction, the autoclave was cooled to -30℃ and the pressure was carefully released from the autoclave, the reaction mixture was submitted to GC analysis. By further distillation, we obtained the desired product (3d-h). The resulting solution was concentrated under reduced pressure and purified by silica gel chromatography (eluent: acetate and petroleum) to give the desired product.(3a-c, 3i-o).

3. Characterization data of compounds (3a-o)

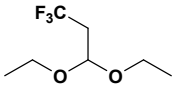
1,1-dimethoxy-3,3,3-trifluoropropane (3a)



^{1}HNMR (500 MHz, $CDCl_3$) δ 4.70 (t, 1 H, *J* = 5.5 Hz), 3.36 (s, 6 H), 2.44 (dq, 2 H, JCF = 10.7 Hz, *J* = 5.5 Hz).^{13}CNMR (125 MHz, $CDCl_3$) δ125.2 (q, *J* = 274.4 Hz), 98.9, 52.9, 37.6 (q, *J* = 27.5 Hz).^{19}F NMR (470.0 MHz) δ-63.6 (t, *J* = 9.4 Hz, CF_3).

IR (KBr): 2945, 2841, 1447, 1343, 1121, 1064 cm^{-1}. EI-MS $[M+H]^+$ *m/z* 158.

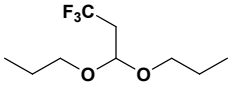
1,1-diethoxy-3,3,3-trifluoropropane (3b)



^{1}HNMR (500 MHz, $CDCl_3$) δ 4.82 (t, *J* = 5.5 Hz, 1H), 3.67 (dq, *J* = 9.3, 7.1 Hz, 2H), 3.54 (dq, *J* = 9.3, 7.1 Hz, 2H), 2.46 (qd, *J* = 10.7, 5.5 Hz, 2H), 1.22 (t, *J* = 7.1 Hz, 6H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.6 Hz), 97.3(q, *J* = 4.0 Hz), 61.8, 37.6 (q, *J* = 27.1 Hz), 15.0. ^{19}F NMR (470.0 MHz) δ-63.6 (t, *J* = 9.4 Hz, CF_3).

IR (KBr): 2983, 2888, 1432, 1383, 1149, 1060 cm^{-1}. EI-MS $[M+H]^+$ *m/z* 186.

1,1-dipropoxy-3,3,3-trifluoropropane (3c)

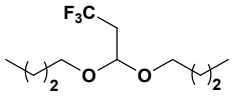


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.81 (t, *J* = 5.4 Hz, 1H), 3.57 (dt, *J* = 9.1, 6.6 Hz, 2H), 3.43 (dt, *J* = 9.1, 6.7 Hz, 2H), 2.46 (qd, *J* = 10.7, 5.5 Hz, 2H), 1.68 – 1.54 (m, 4H), 0.94 (t, *J* = 7.4 Hz, 7H).^{13}CNMR (125 MHz, $CDCl_3$)δ125.3 (q, *J* = 274.7 Hz), 97.3(q, *J* = 4.0 Hz), 68.0, 38.6 (q, *J* = 27.2 Hz), 22.8, 10.5. ^{19}F NMR (470.0 MHz) δ-63.5 (t, *J* = 10.4 Hz, CF_3).

IR (KBr): 2967, 2881, 1465, 1375, 1149, 1072 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 237.1079; found: 237.1074.

1,1-dibutoxy-3,3,3-trifluoropropane (3d)

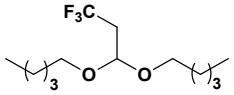


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.80 (t, *J* = 5.4 Hz, 1H), 3.60 (dt, *J* = 9.2, 6.5 Hz, 2H), 3.46 (dt, *J* = 9.2, 6.6 Hz, 2H), 2.45 (qd, *J* = 10.7, 5.4 Hz, 2H), 1.61~1.51 (m, 4H), 1.44 ~1.33 (m, 4H), 0.92 (t, *J* = 7.4 Hz, 6H).^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.7 Hz), 97.5(q, *J* = 4.0 Hz), 66.0,38.5 (q, *J* = 27.2 Hz), 31.7, 19.2, 13.7. ^{19}FNMR (470.0 MHz) δ-63.6 (t, *J* = 10.9 Hz, CF_3).

IR (KBr): 2936, 2876, 1465, 1377, 1151, 1074 cm^{-1}.

HRMS (ESI): calc. for ($M + Na^+$) 265.1392; found: 265.1389.

1,1-dipentyloxy-3,3,3-trifluoropropane (3e)

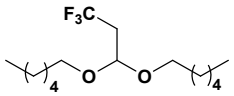


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.80 (t, *J* = 5.4 Hz, 1H), 3.59 (dt, *J* = 9.1, 6.6 Hz, 2H), 3.45 (dt, *J* = 9.2, 6.7 Hz, 2H), 2.45 (qd, *J* = 10.7, 5.5 Hz, 2H), 1.67~1.49 (m, 4H), 1.33 (dd, *J* = 7.4, 3.6 Hz, 8H), 0.90 (t, *J* = 7.1 Hz, 6H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.7 Hz), 97.5(q, *J* = 3.9 Hz), 66.4, 38.6 (q, *J* = 27.2 Hz), 29.3, 28.3, 22.4, 13.9. ^{19}F NMR (470.0 MHz) δ-63.5 (t, *J* = 10.8 Hz, CF_3).

IR (KBr): 2935, 2874, 1468, 1378, 1151, 1120, 1059 cm^{-1}.

HRMS (ESI): calc. for ($M + Na^+$) 293.1705; found: 293.1699.

1,1-dihexyloxy-3,3,3-trifluoropropane (3f)

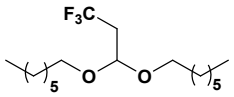


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.80 (t, *J* = 5.4 Hz, 1H), 3.59 (dt, *J* = 9.1, 6.6 Hz, 2H), 3.45 (dt, *J* = 9.2, 6.7 Hz, 2H), 2.45 (qd, *J* = 10.7, 5.4 Hz, 2H), 1.63 ~ 1.52 (m, 4H), 1.30 (s, 12H), 0.89 (t, *J* = 6.9 Hz, 6H).^{13}CNMR (125 MHz, $CDCl_3$)δ125.3 (q, *J* = 274.7 Hz), 97.5(q, *J* = 3.9 Hz), 66.4, 38.6 (q, *J* = 27.2 Hz), 31.6, 29.6, 25.8, 22.6, 13.9. ^{19}FNMR (470.0 MHz) δ-63.5 (t, J = 10.5 Hz, CF_3).

IR (KBr): 2933, 2862, 1467, 1378, 1150, 1120, 1069 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 321.2018; found: 321.2013.

1,1-diheptyloxy-3,3,3-trifluoropropane (3g)



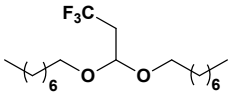
^{1}HNMR (500 MHz, $CDCl_3$) δ 4.80 (t, *J* = 5.4 Hz, 1H), 3.59 (dt, *J* = 9.1, 6.6 Hz, 2H), 3.45 (dt, *J* = 9.1, 6.7 Hz, 2H), 2.45 (qd, *J* = 10.7, 5.5 Hz, 2H), 1.69 ~ 1.52 (m,4H), 1.28 (s, 16H), 0.88 (t, *J* = 6.9 Hz, 6H).^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.7 Hz), 97.5(q, *J* = 3.9 Hz), 66.4, 38.6, 31.8, 29.7, 29.0, 26.0, 22.6, 14.0

^{19}FNMR (470.0 MHz) δ-63.5 (t, J = 10.6 Hz, CF_3).

IR (KBr): 2931, 2859, 1467, 1377, 1150, 1120, 1075 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 349.2331; found: 349.2320.

1,1-dioctyloxy-3,3,3-trifluoropropane (3h)

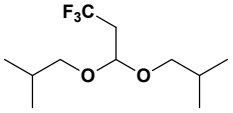


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.79 (t, *J* = 5.4 Hz, 1H), 3.59 (dt, *J* = 9.1, 6.6 Hz, 2H), 3.45 (dt, *J* = 9.1, 6.7 Hz, 2H), 2.45 (qd, *J* = 10.7, 5.4 Hz, 2H), 1.61 – 1.54 (m, 4H), 1.29 (s, 20H), 0.88 (t, *J* = 6.9 Hz, 6H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.8 Hz), 97.5(q, *J*=4.0 Hz), 66.4, 38.6 (q, *J* = 27.2 Hz), 31.8, 29.7, 29.4, 29.2, 26.0, 22.6, 14.0. ^{19}F NMR (470.0 MHz) δ-63.5 (t, *J* = 10.5 Hz, CF_3).

IR (KBr): 2928, 2858, 1467, 1377, 1150, 1120, 1079 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 377.2644; found: 377.2634.

1,1-diisobutyloxy-3,3,3-trifluoropropane (3i)



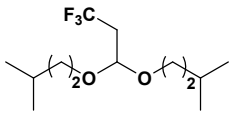
^{1}HNMR (500 MHz, $CDCl_3$) δ 4.79 (t, *J* = 5.4 Hz, 1H), 3.38 (dd, *J* = 8.9, 6.4 Hz, 2H), 3.22 (dd, *J* = 8.9, 6.6 Hz, 2H), 2.46 (qd, *J* = 10.7, 5.4 Hz, 2H), 1.85 (dp, *J* = 13.3, 6.7 Hz, 2H), 0.92 (d, *J* = 6.7 Hz, 12H).^{13}CNMR (125 MHz, $CDCl_3$) δ125.4 (q, *J* = 274.8

Hz), 97.7(q, *J* = 4.0 Hz), 73.0, 38.5 (q, *J* = 27.3 Hz), 28.5, 19.3. ^{19}F NMR (470.0 MHz) δ-63.5 (t, *J* = 10.3 Hz, CF_3).

IR (KBr): 2961, 2876, 1473, 1386, 1253, 1148, 1064 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 265.1392; found: 265.1386.

1,1-diisopentyloxy-3,3,3-trifluoropropane (3j)

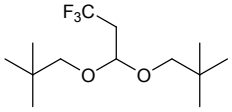


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.79 (t, *J* = 5.4 Hz, 1H), 3.62 (dt, *J* = 9.2, 6.7 Hz, 2H), 3.49 (dt, *J* = 9.2, 6.9 Hz, 2H), 2.46 (qd, *J* = 10.7, 5.5 Hz, 2H), 1.71 (dp, *J* = 13.4, 6.7 Hz, 2H), 1.47 (ddd, *J* = 13.8, 6.9, 2.3 Hz, 4H), 0.91 (dd, *J* = 6.6, 1.7 Hz, 12H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.8 Hz), 97.5(q, *J* = 4.0 Hz), 64.7, 38.6 (q, J = 27.2 Hz), 24.9, 22.6, 22.5. ^{19}F NMR (470.0 MHz) δ-63.6 (t, *J* = 10.5 Hz, CF_3).

IR (KBr): 2959, 2874, 1468, 1371, 1255, 1120, 1071 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 293.1705; found: 293.1702.

1,1-diterbutylmeoxy-3,3,3-trifluoropropane (3k)

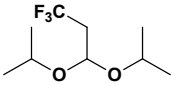


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.79 (t, J = 5.4 Hz, 1H), 3.18 (dd, J = 95.9, 8.6 Hz, 4H), 2.46 (qd, J = 10.8, 5.5 Hz, 2H), 0.92 (s, 18H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.4 (q, *J* = 274.6 Hz), 98.0(q, *J* = 4.0 Hz), 76.5, 38.3, (q, *J* = 27.5 Hz), 31.8, 26.6. ^{19}F NMR (470.0 MHz) δ-63.4 (t, *J* = 9.4 Hz, CF_3).

IR (KBr): 2958, 2871, 1481, 1365, 1150, 1071 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 293.1700; found: 293.1699.

1,1-diisopropyloxy-3,3,3-trifluoropropane (3l)



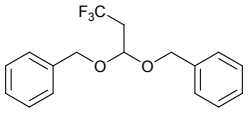
^{1}HNMR (500 MHz, $CDCl_3$) δ 4.87 (t, *J* = 5.3 Hz, 1H), 3.87 (dt, *J* = 12.3, 6.1 Hz, 2H), 2.43 (qd, *J* = 10.7, 5.3 Hz, 2H), 1.19 (dd, *J* = 24.2, 6.2 Hz, 12H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, *J* = 274.8 Hz), 94.8(q, *J* = 4.0 Hz), 68.9, 40.6 (q, *J* = 27.3 Hz), 23.2,

22.2. ^{19}F NMR (470.0 MHz) δ-63.3 (t, J = 10.3 Hz, CF_3).

IR (KBr): 2977, 2935, 1468, 1384, 1155, 1037 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 237.1079; found: 237.1073.

1,1-dibenzyloxy-3,3,3-trifluoropropane (3m)

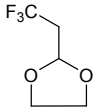


^{1}HNMR (500 MHz, $CDCl_3$) δ 7.33 (m, 10H), 5.08 (t, J = 5.5 Hz, 1H), 4.63 (dd, J = 38.8, 11.6 Hz, 4H), 2.60 (qd, J = 10.6, 5.5 Hz, 2H). ^{13}CNMR (125 MHz, $CDCl_3$) δ 137.2, 128.6, 128.0, 127.8, 125.4, 96.6 (q, J = 4.0 Hz), 68.1, 38.6, (q, J = 27.7 Hz),. ^{19}F NMR (470.0 MHz) δ-63.3 (t, J = 9.4 Hz, CF_3).

IR (KBr): 3033, 2878, 1455, 1343, 1256, 1052 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 333.1900; found: 333.1079.

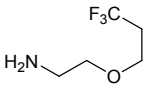
1-ethylenedioxy-3,3,3-trifluoropropane (3n)



^{1}HNMR (500 MHz, $CDCl_3$) δ 5.15 (t, J = 4.8 Hz, 1H), 3.94 (m, 4H), 2.49 (qd, J = 10.8, 4.8 Hz, 2H). ^{13}CNMR (125 MHz, $CDCl_3$) δ125.3 (q, J = 274.6 Hz), 98.7(q, J = 3.9 Hz), 65.0, 39.0 (q, J = 27.7 Hz). ^{19}F NMR (470.0 MHz) δ-63.5 (t, J = 9.4 Hz, CF_3).

IR (KBr): 2966, 2898, 1431, 1343, 1128, 1044 cm^{-1}. EI-MS $[M+H]^+$ *m/z* 157.2.

1-aminoethoxy-3,3,3-trifluoropropane (3o)



^{1}HNMR (500 MHz, $CDCl_3$) δ 3.66 (m, 2H), 2.91 (t, J = 7.1 Hz, 2H), 2.79 (m, 2H), 2.35 (s, 4H).^{13}CNMR (125 MHz, $CDCl_3$) δ126.6 (q, J = 274.0 Hz), 60.7, 50.9, 42.3, (q, J = 3.3 Hz), 34.3. ^{19}F NMR (470.0 MHz) δ -63.2 (t, J = 9.4 Hz, CF_3).

IR (KBr): 3300, 2927, 1662, 1256, 1152, 1069 cm^{-1}.

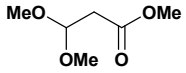
HRMS (ESI): calc. for (M + H^+) 158.0700; found: 158.0789.

4. General method for the synthesis of esters 3,3-dimethoxypropanoate (4b-e)

A 150-mL autoclave was charged with 1 %mol of 5% Pd/C and 2 %mol of $CuCl_2$ in 20 mL of methanol, and functionalized olefin (30 mmol) was added. Then, the autoclave was filled with 1 MPa dioxygen. The mixture was stirred magnetically at 50℃ for 8h. After the reaction, the autoclave was cooled to -30℃ and the pressure was carefully released from the autoclave, the reaction mixture was submitted to GC analysis. The resulting solution was concentrated under reduced pressure and purified by silica gel chromatography (eluent: acetate and petroleum) to give the desired product.

5. Characterization data of compounds (4b-e)

methyl 3,3-dimethoxypropanoate (4b)

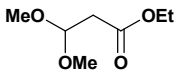


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.84 (t, *J* = 5.9 Hz, 1H), 3.70 (s, 3H), 3.37 (s, 6H), 2.66 (d, *J* = 5.9 Hz, 2H).^{13}C NMR (125 MHz, $CDCl_3$) δ 170.3 (s), 101.3 (s), 53.5 (s), 51.8 (s), 38.7 (s).

IR (KBr): 2959, 2874, 1460, 1401, 1445, 1189, 1125, 1069 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 171.0634; found: 171.0632.

ethyl 3,3-dimethoxypropanoate (4c)

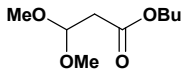


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.84 (t, *J* = 5.9 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.37 (s, 6H), 2.65 (d, *J* = 5.9 Hz, 2H), 1.34 ~ 1.17 (m, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9 (s), 101.2 (s), 60.6 (s), 53.5 (s), 38.9 (s), 14.1 (s).

IR (KBr): 2985, 2840, 1740, 1378, 1445, 1179, 1124, 1069 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 185.079; found: 185.0787.

butyl 3,3-dimethoxypropanoate (4d)

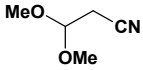


^{1}HNMR (500 MHz, $CDCl_3$) δ 4.76 (t, *J* = 5.9 Hz, 1H), 4.04 (t, *J* = 6.7 Hz, 2H), 3.29 (s, 6H), 2.57 (d, *J* = 5.9 Hz, 2H), 1.65 ~ 1.44 (m, 2H), 1.40 ~ 1.21 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9 (s), 101.3 (s), 64.5 (s), 53.4 (s), 38.9 (s), 30.6 (s), 19.0 (s), 13.6 (s).

IR (KBr): 2985, 2840, 1740, 1378, 1445, 1179, 1124, 1069 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 213.1103; found: 213.1102.

3,3-dimethoxypropanenitrile (4e)



^{1}HNMR (500 MHz, $CDCl_3$) δ 4.68 (t, *J* = 5.4 Hz, 1H), 3.42 (s, 6H), 2.67 (d, *J* = 5.4 Hz, 2H).^{13}C NMR (125 MHz, $CDCl_3$) δ 116.1 (s), 99.7 (s), 54.2 (s), 22.9 (s).

IR (KBr): 2935, 2846, 2254, 1455, 1418, 1124, 1069 cm^{-1}.

HRMS (ESI): calc. for (M + Na^+) 138.0531; found: 138.0526.

6. References for known products

entry	product	reference
1	3a	[S2]
2	3b	NO
3	3c	NO
4	3d	NO
5	3e	NO

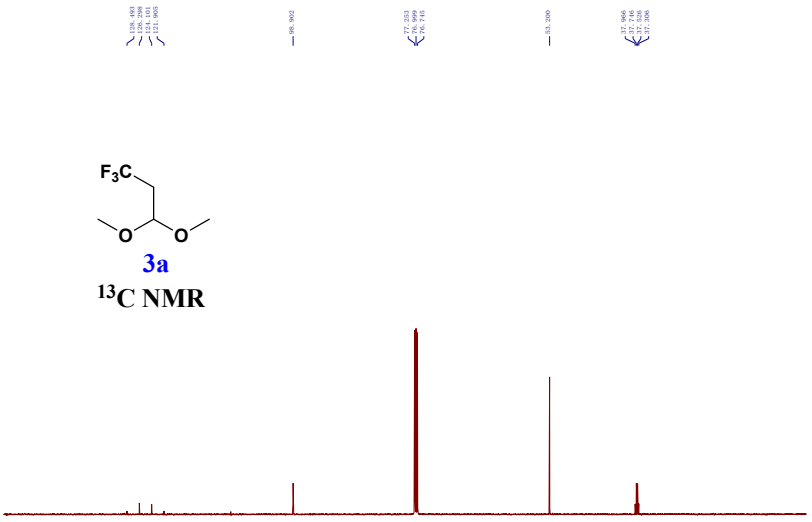
6	3f	NO
7	3g	NO
8	3h	NO
9	3i	NO
10	3j	NO
11	3k	NO
12	3l	NO
13	3m	NO
14	3n	NO
15	3o	NO
16	4b	[S1]
17	4c	[S1]
18	4d	[S1]
19	4e	[S1]

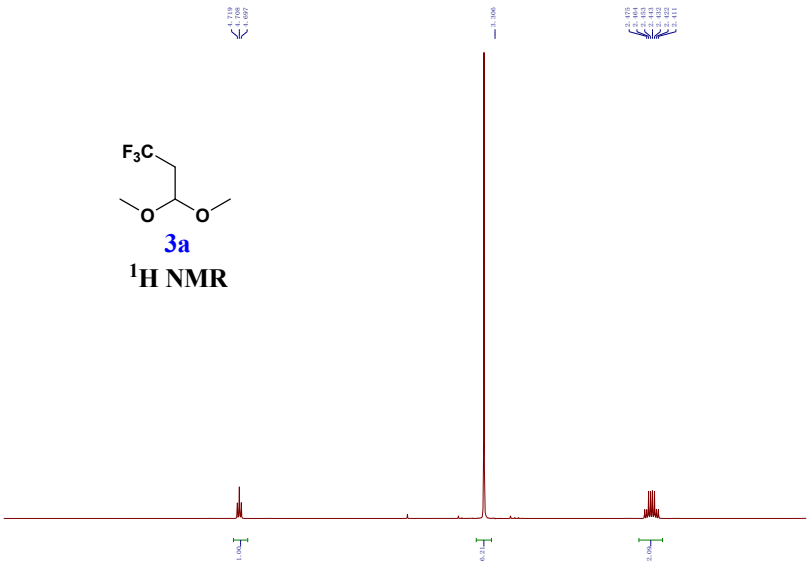
NO: No reference was found via SCIfinder.

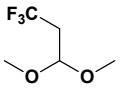
[S1] H. F. Jiang, Y. X. Shen, Z.Y. Wang. *Tetrahedron*, 2008, 64,508-514.

[S2] T. Komata, S. Akiba, K. Hosoi, K. Ogura. *J. Fluo. Chem.,* 2008, **129**, 35-39.

7. Copies of the ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of products

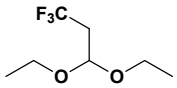






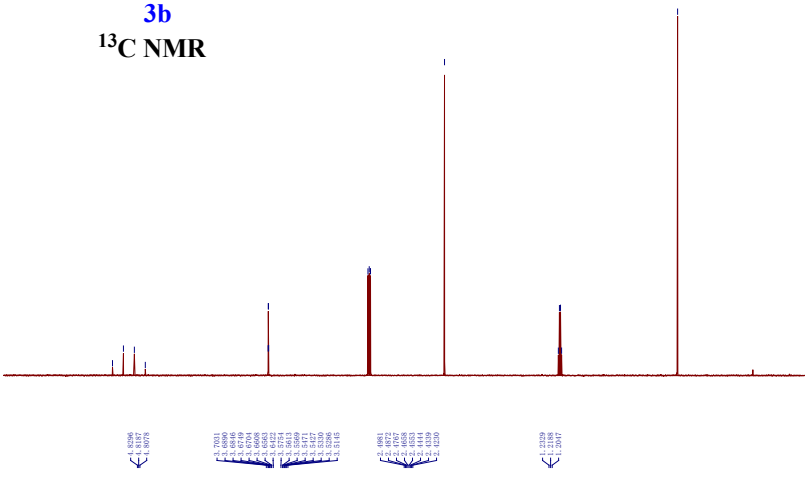
3a

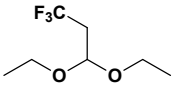
^{19}F NMR



3b

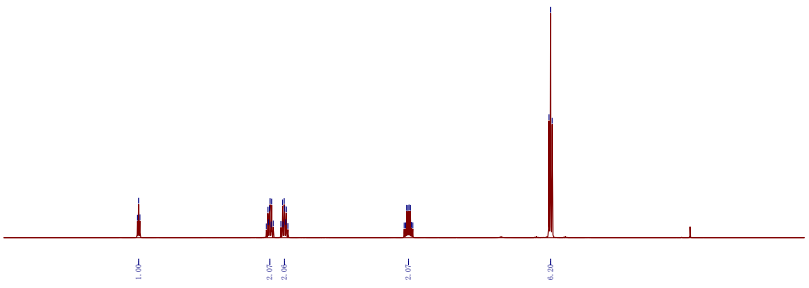
^{13}C NMR

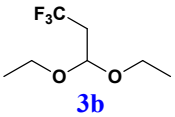




3b

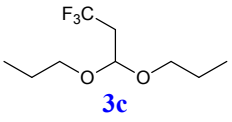
^{1}H NMR





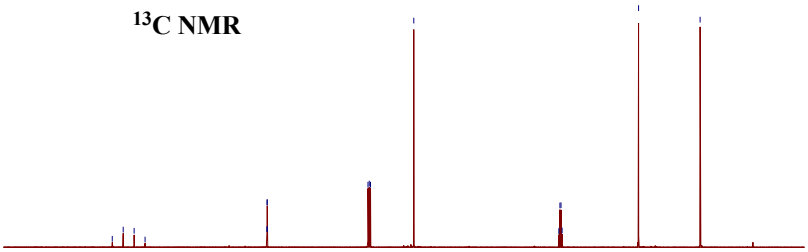
3b

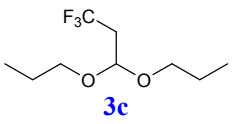
^{19}F NMR



3c

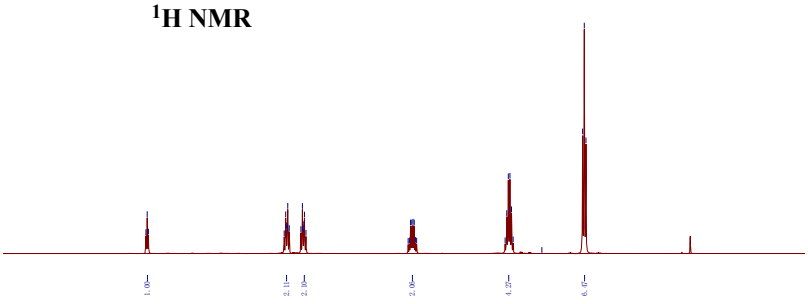
^{13}C NMR

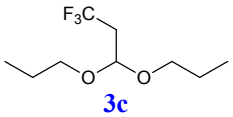




3c

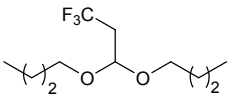
^{1}H NMR





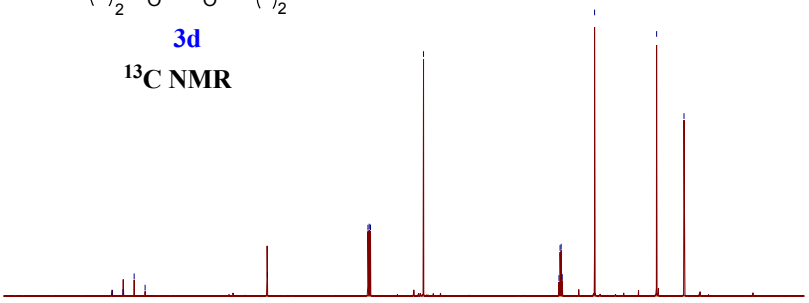
3c

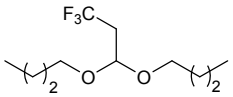
^{19}F NMR



3d

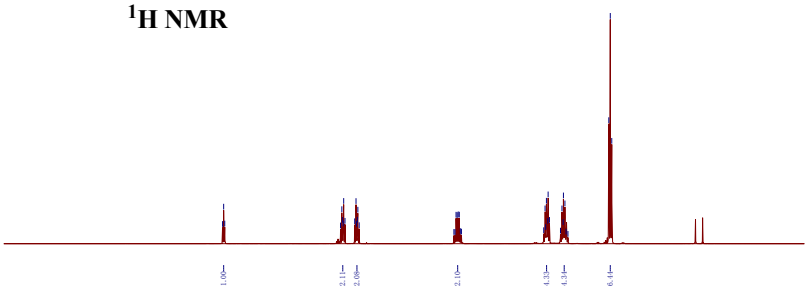
^{13}C NMR

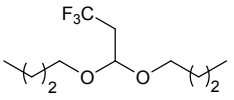




3d

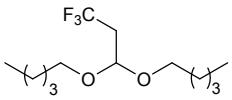
^{1}H NMR





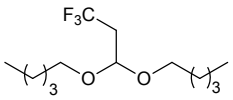
3d

^{19}F NMR



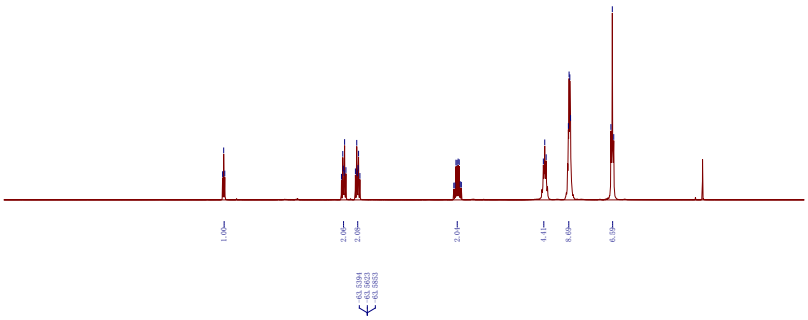
3e

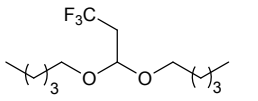
^{13}C NMR



3e

^{1}H NMR





3e

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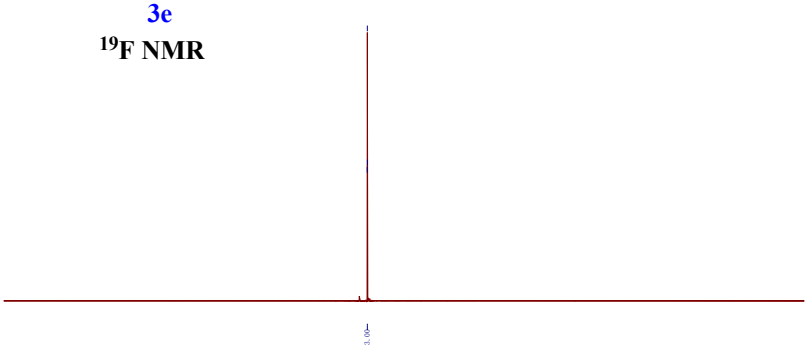
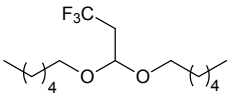
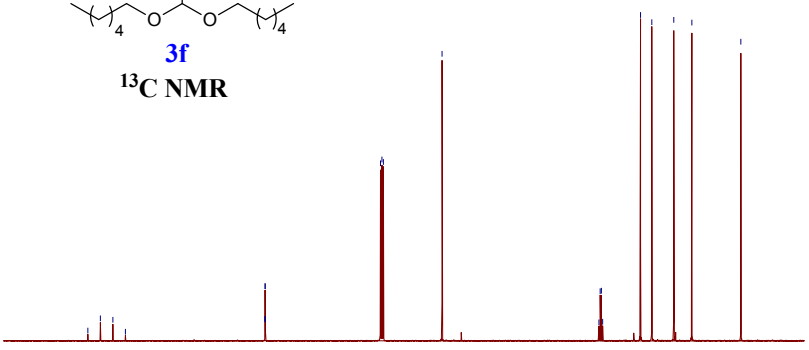
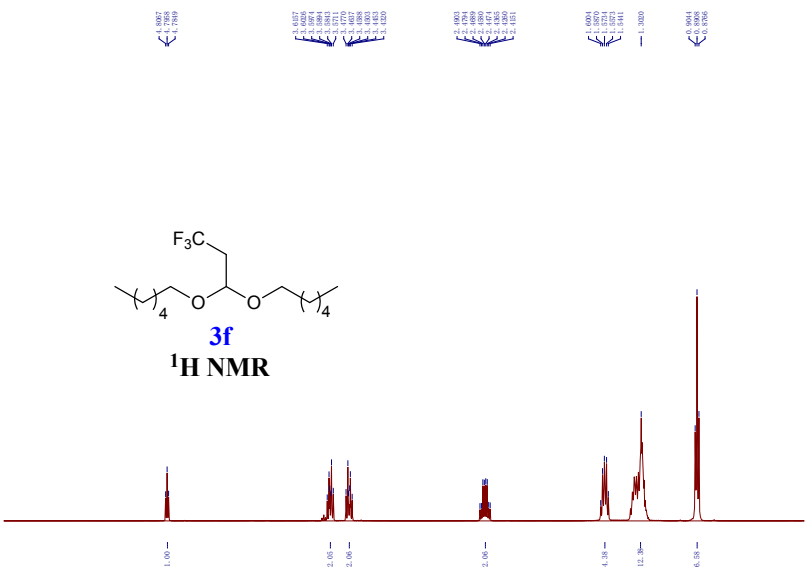




3f

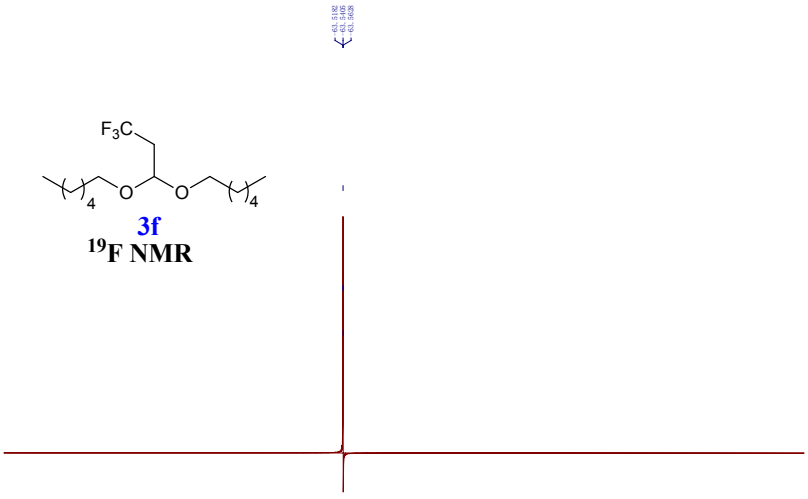
^{13}C NMR





3f

^{1}H NMR



3f

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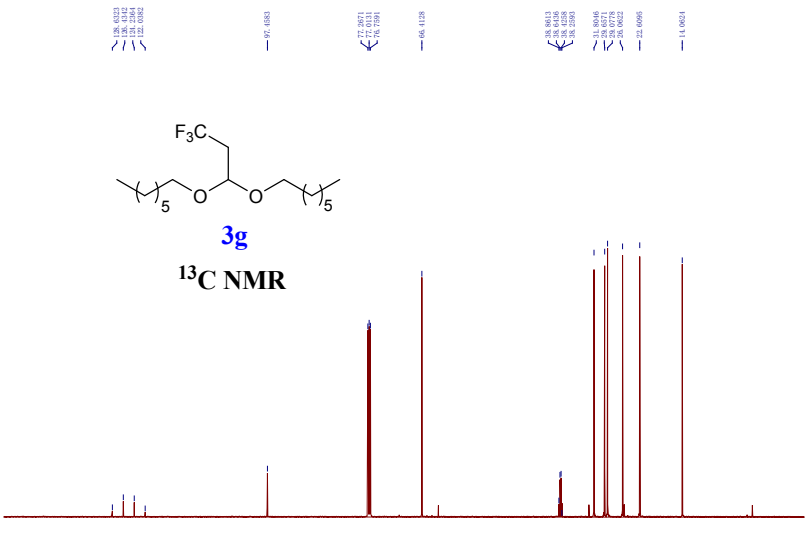
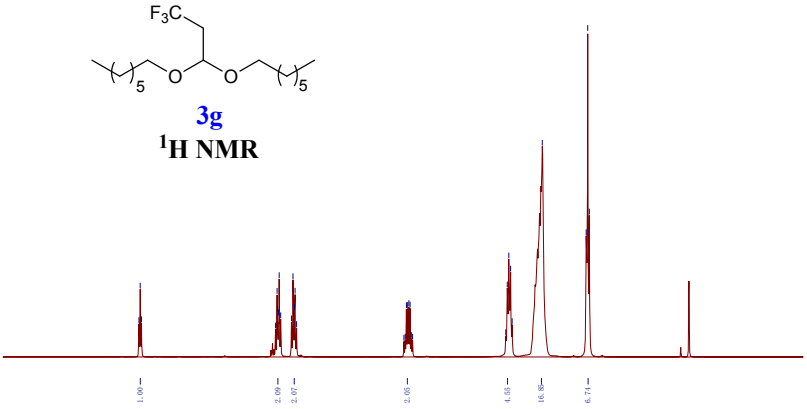


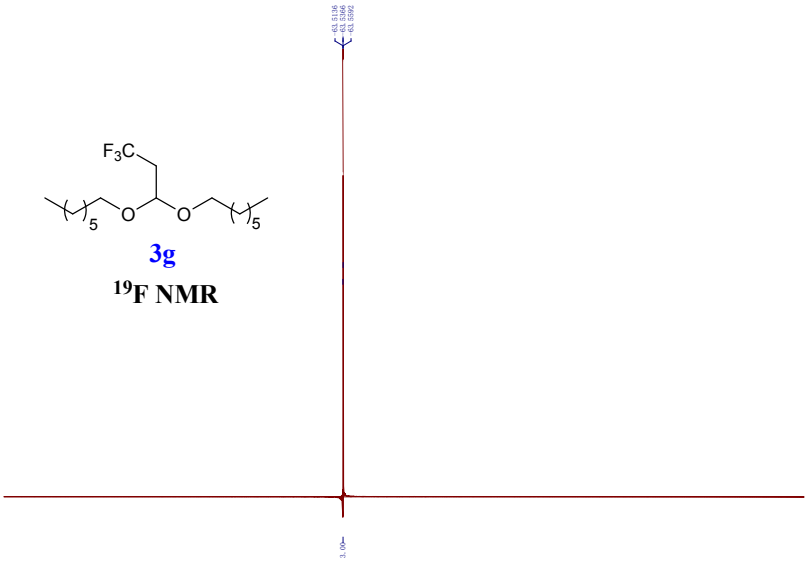
3g

^{13}C NMR



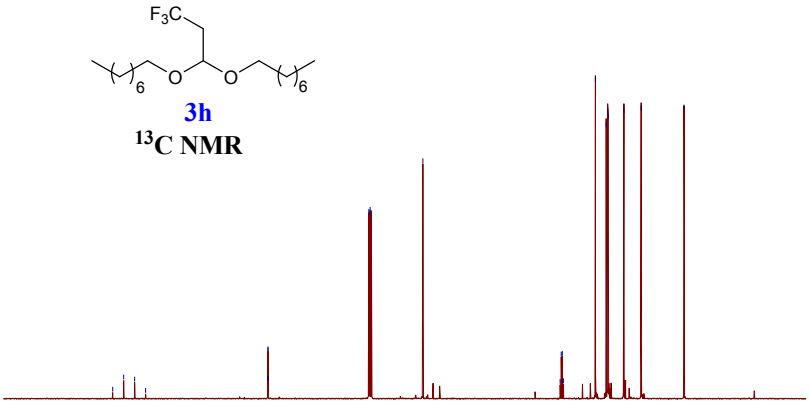
3g

^{1}H NMR



3g

^{19}F NMR



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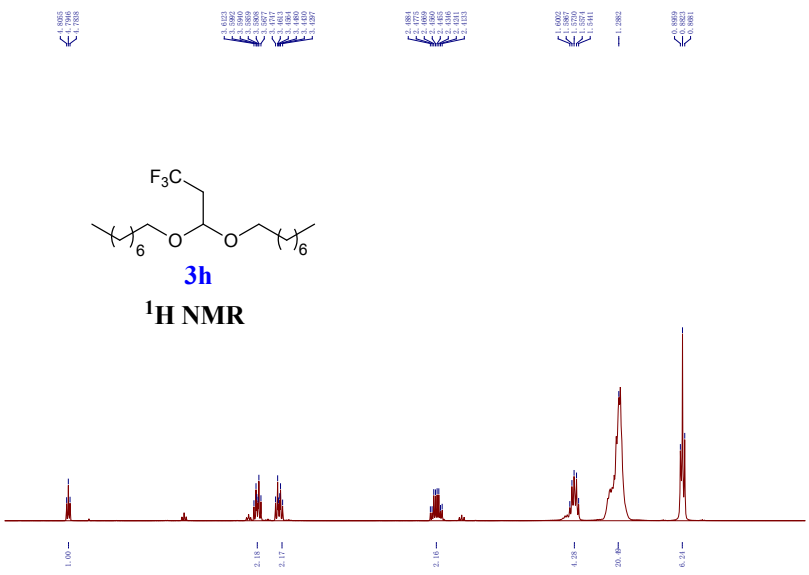
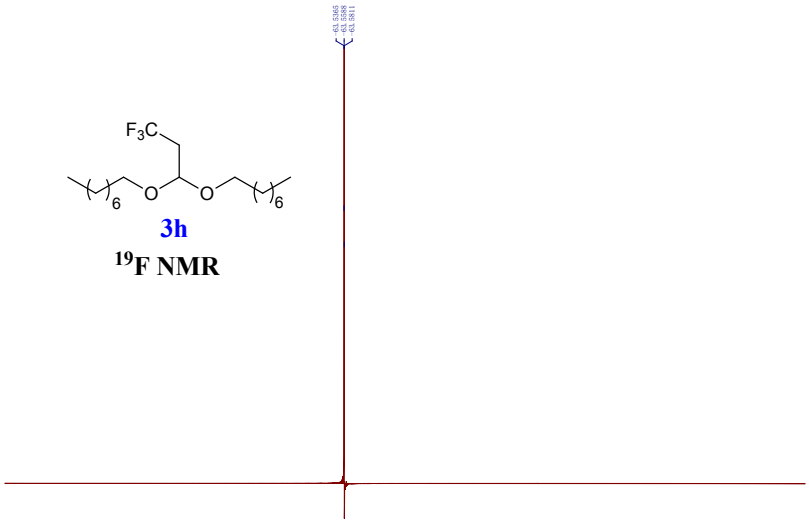


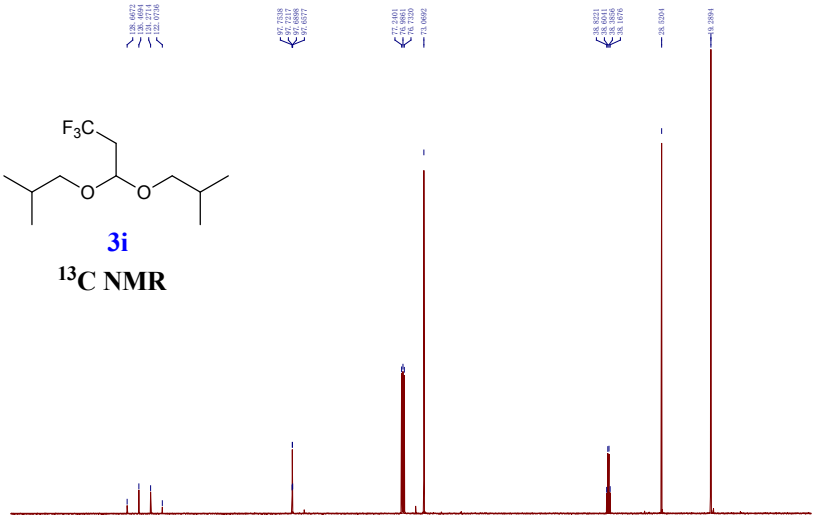
3h

^{1}H NMR



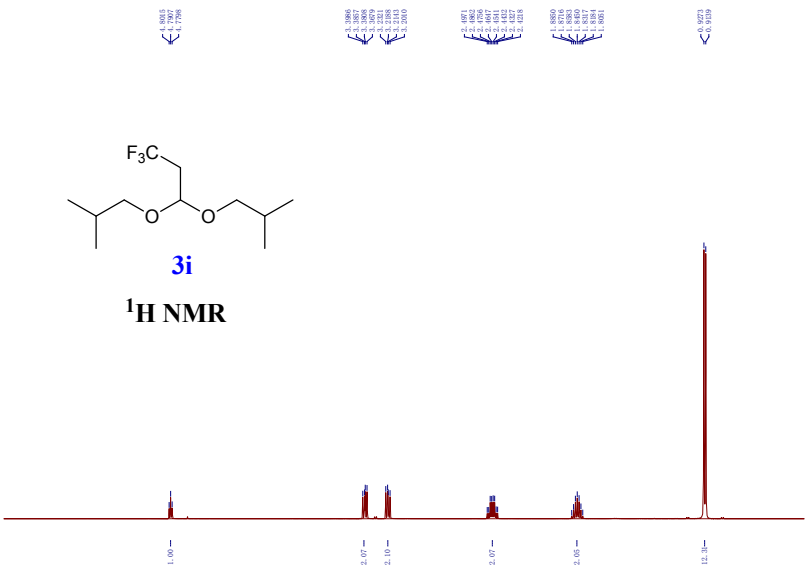
3h

^{19}F NMR



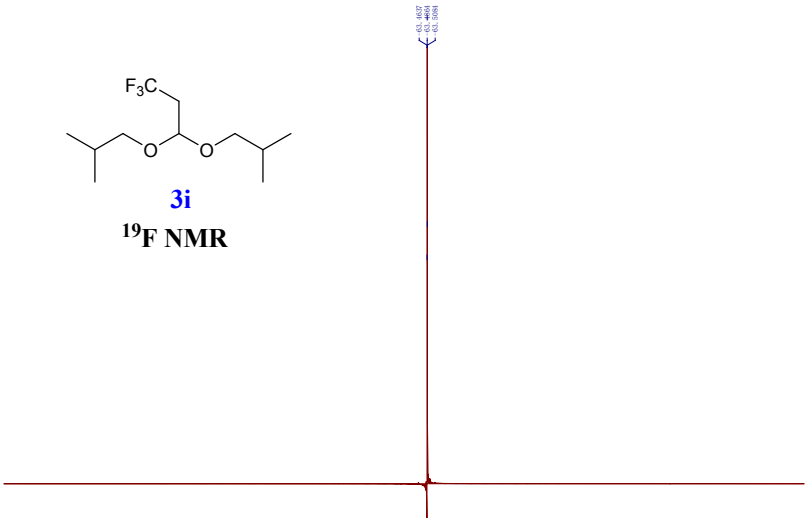
3i

^{13}C NMR



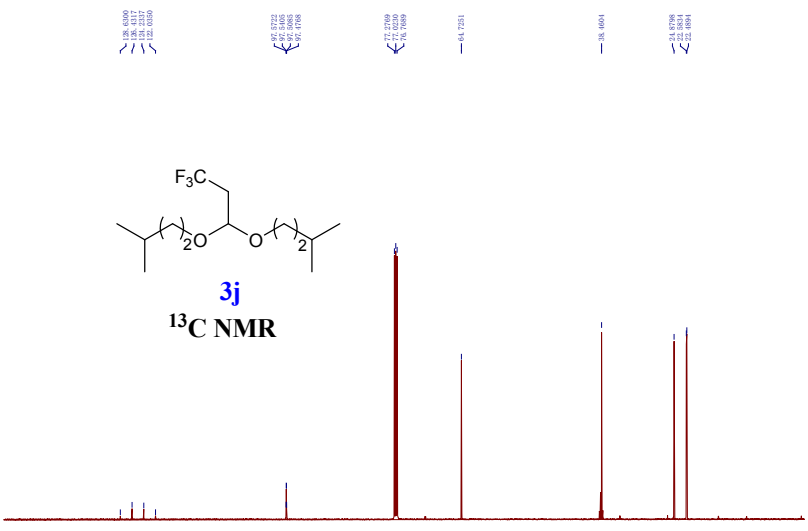
3i

^{1}H NMR



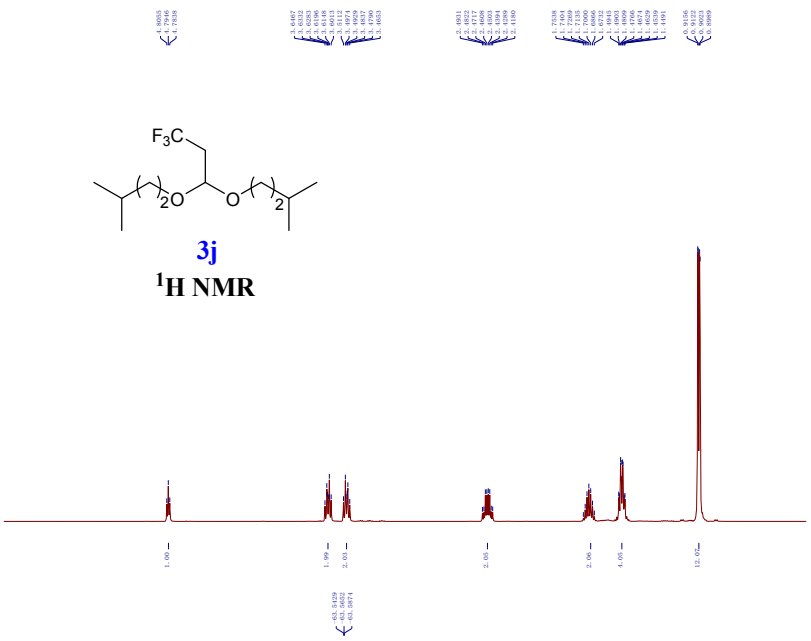
3i

^{19}F NMR



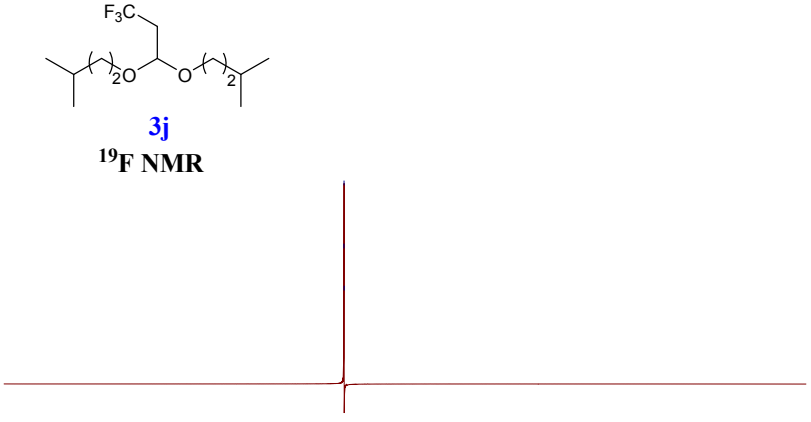
3j

^{13}C NMR



3j

^{1}H NMR

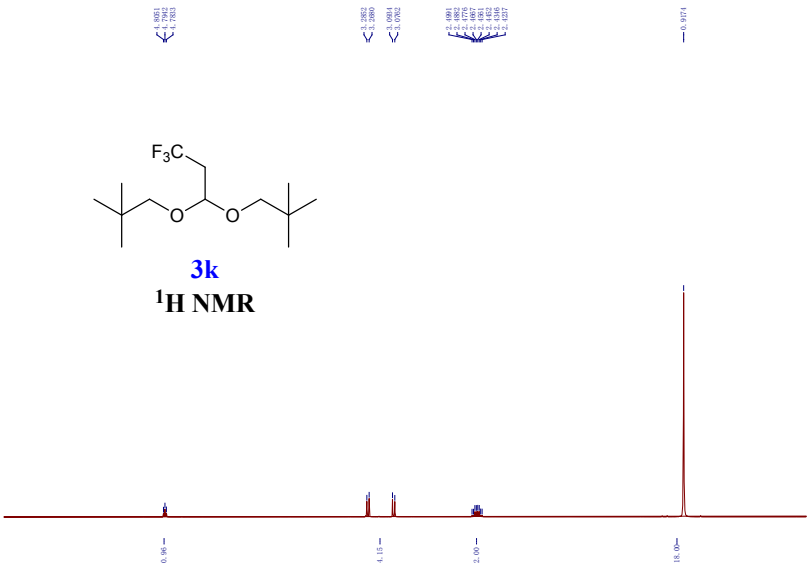


3j

^{19}F NMR

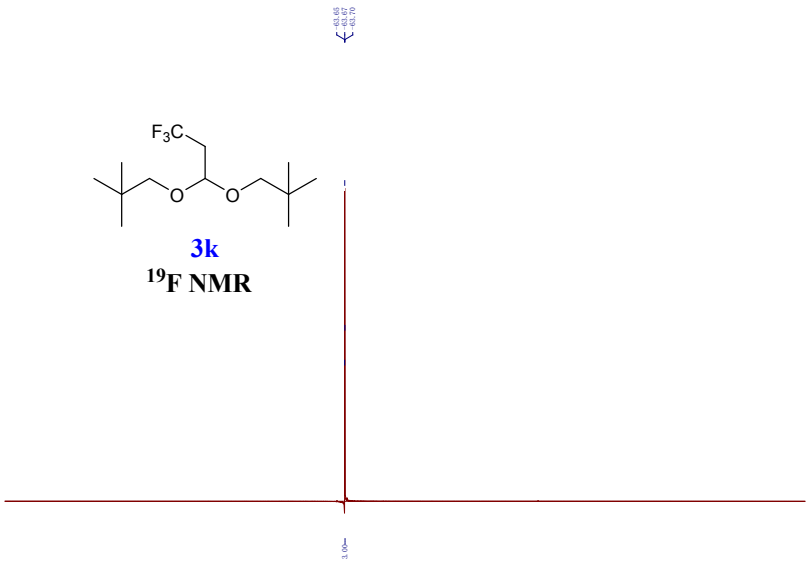
3k

^{13}C NMR



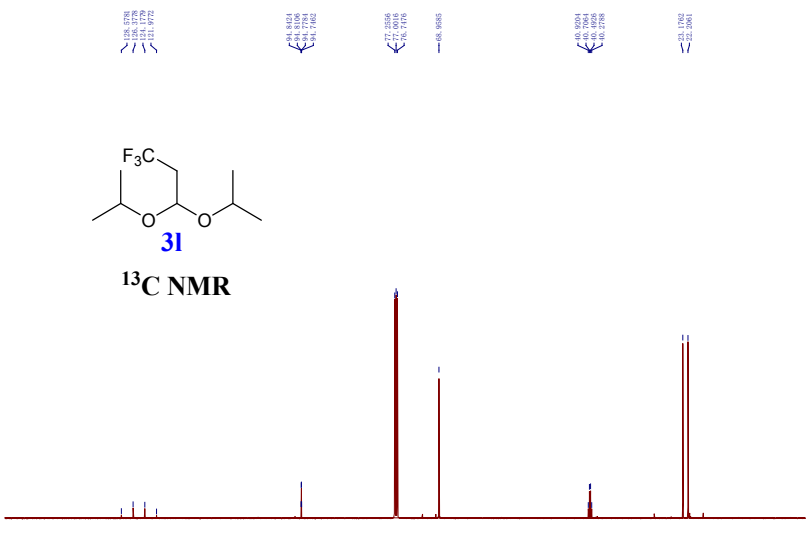
3k

^{1}H NMR



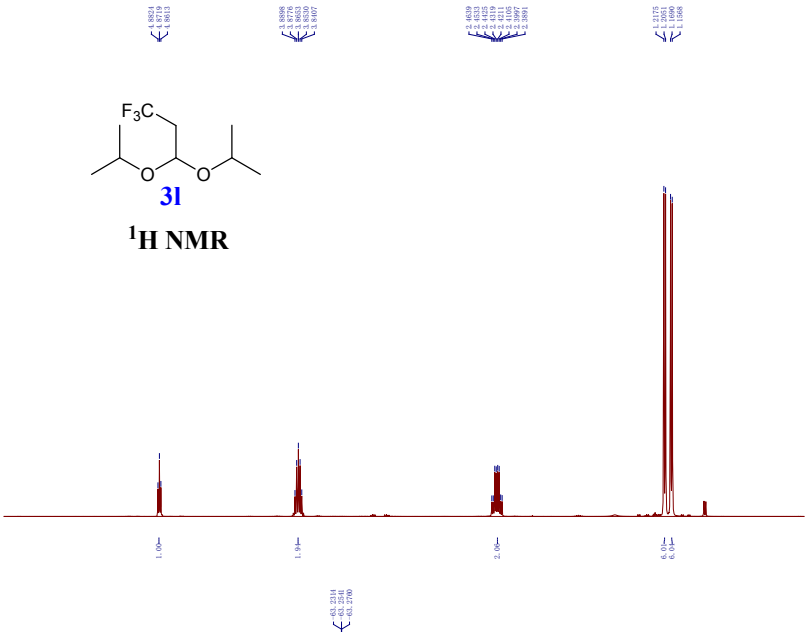
3k

^{19}F NMR



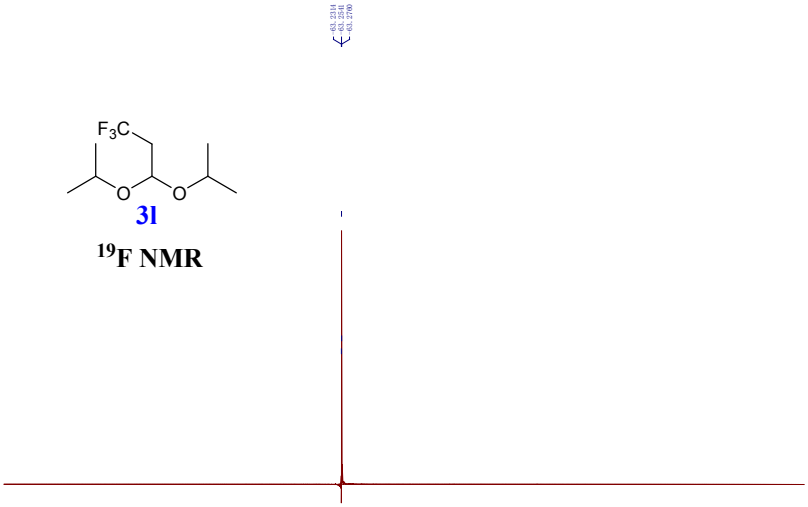
3l

^{13}C NMR



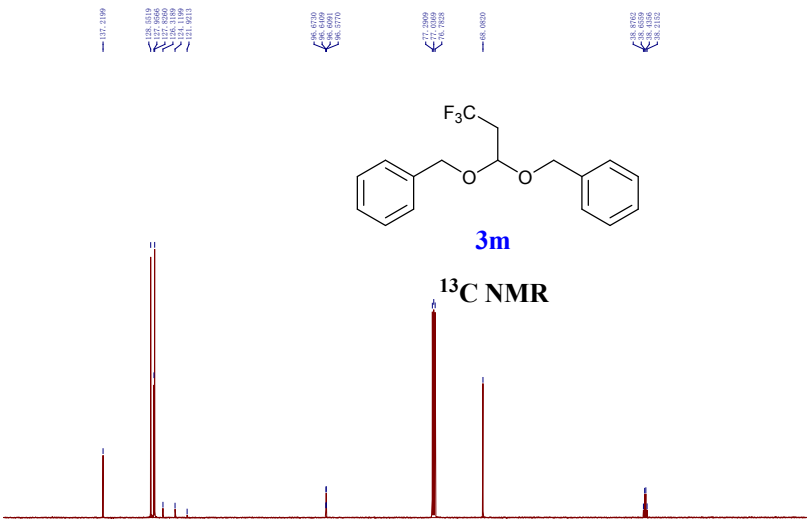
3l

^{1}H NMR



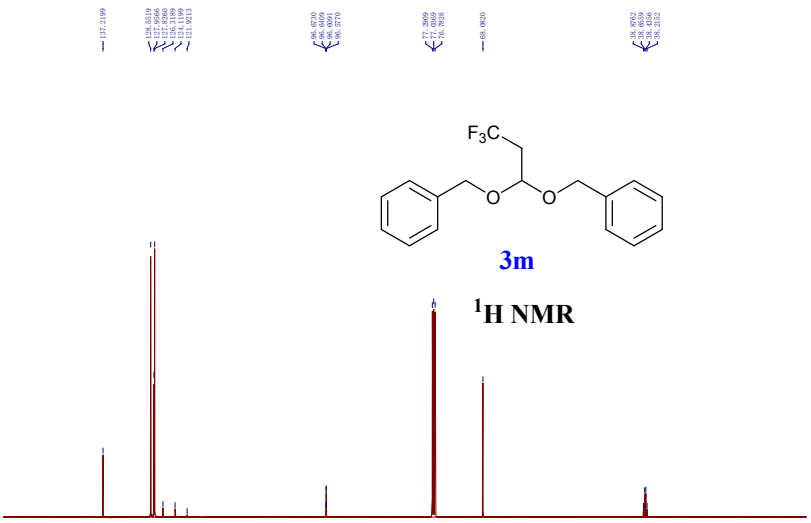
3l

^{19}F NMR



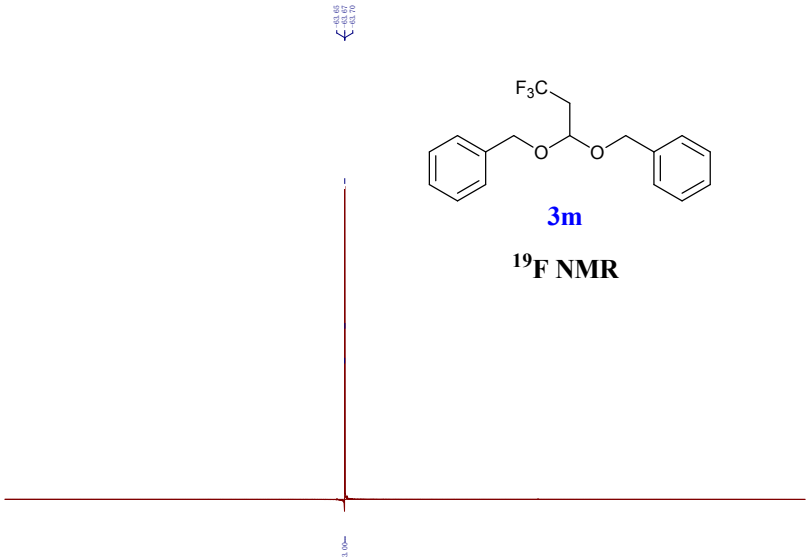
3m

^{13}C NMR



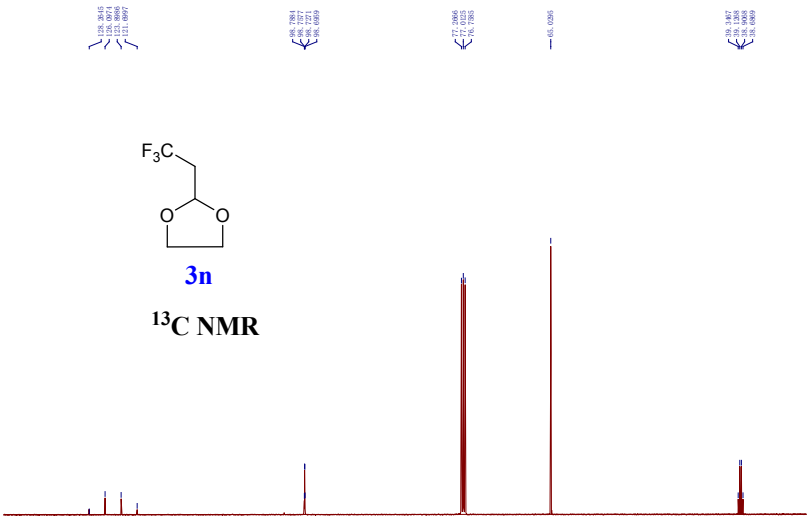
3m

^{1}H NMR



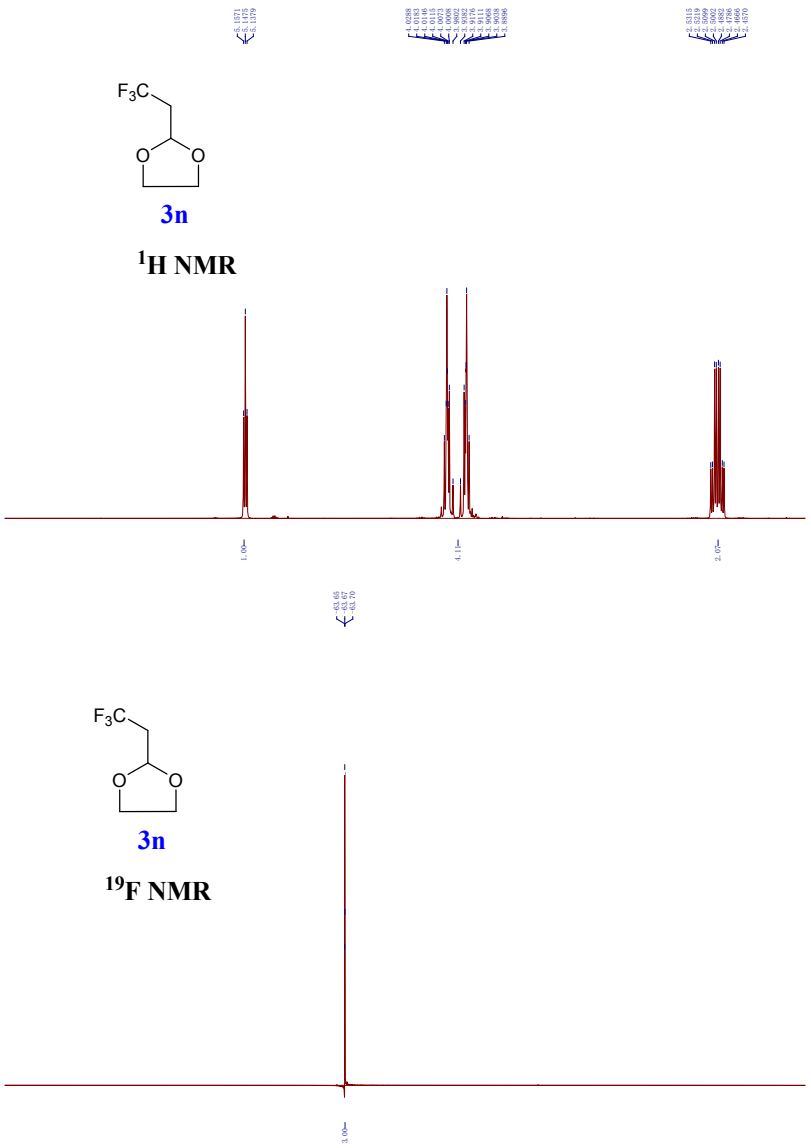
3m

^{19}F NMR



3n

^{13}C NMR

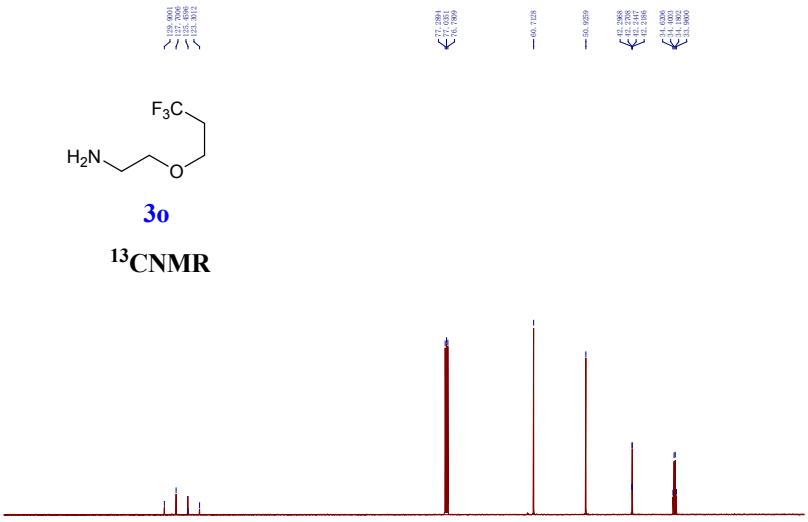


3n

^{1}H NMR

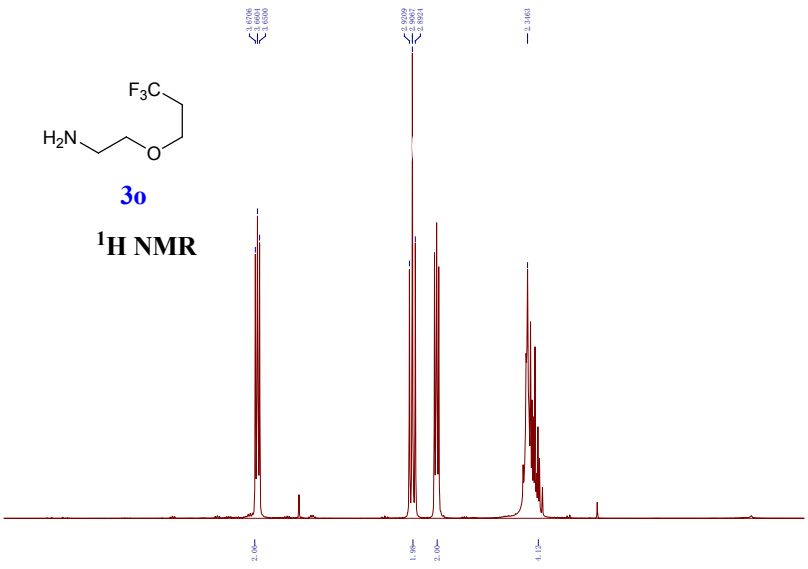
3n

^{19}F NMR



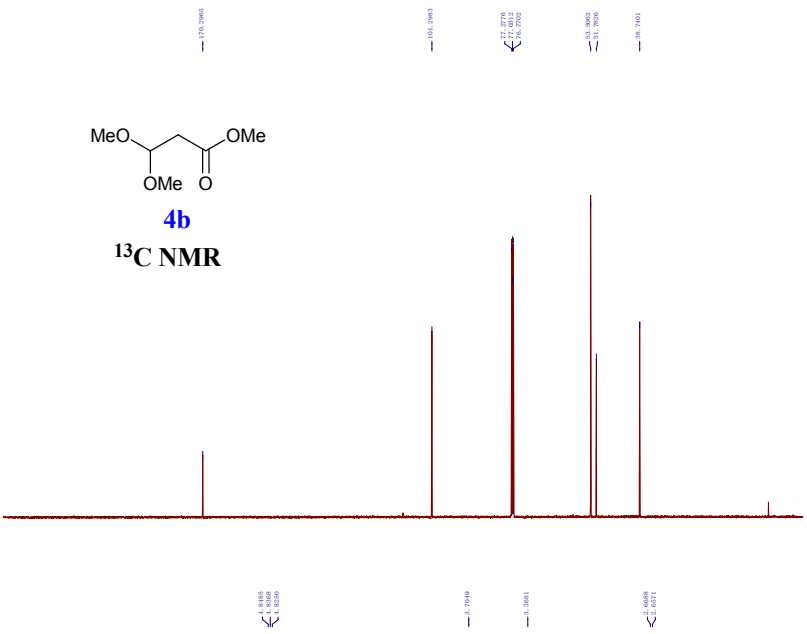
3o

^{13}CNMR



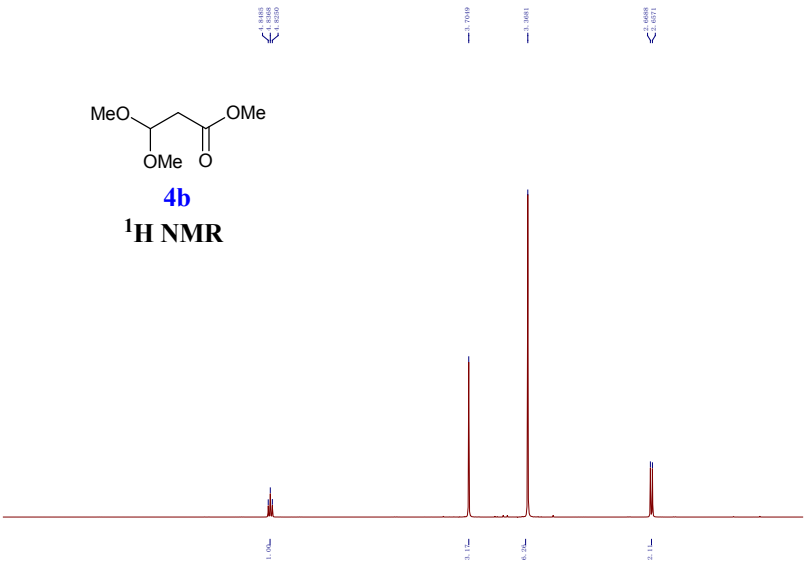
3o

^{1}H NMR



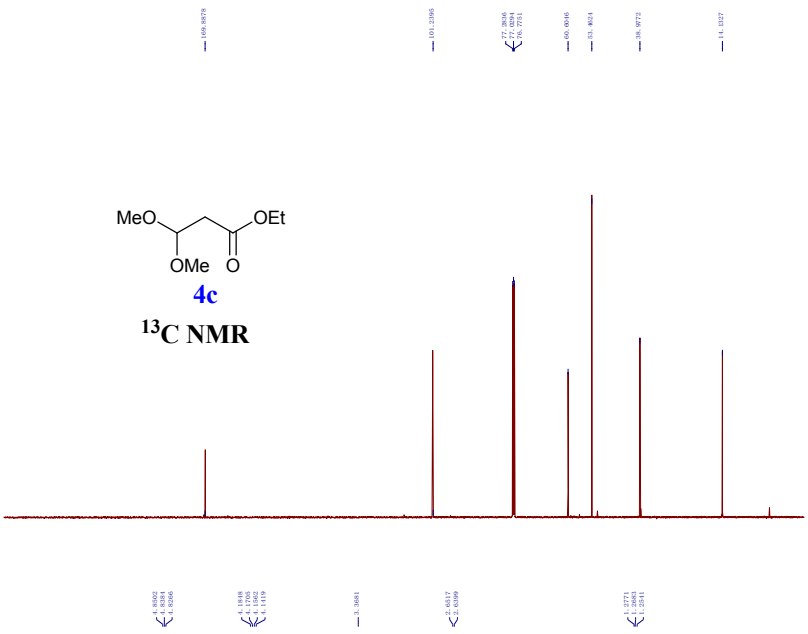
4b

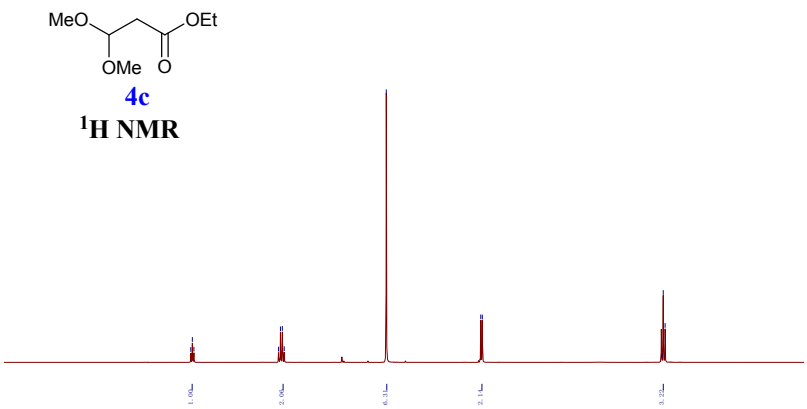
^{13}C NMR

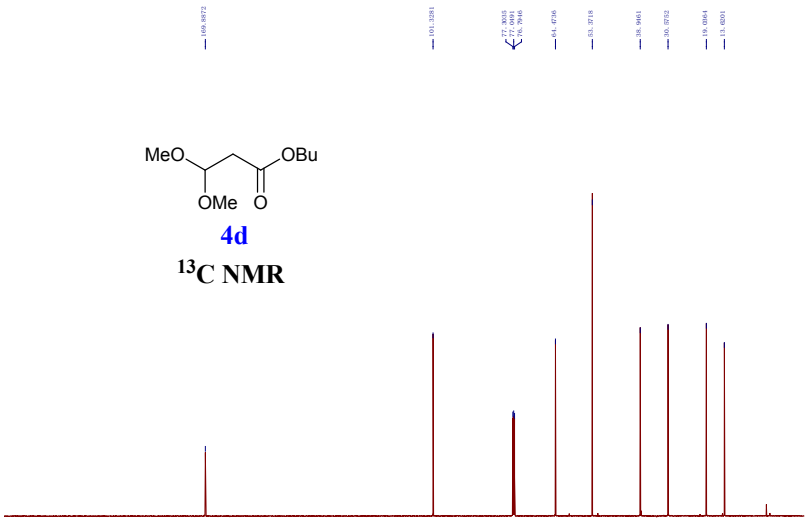


4b

^{1}H NMR

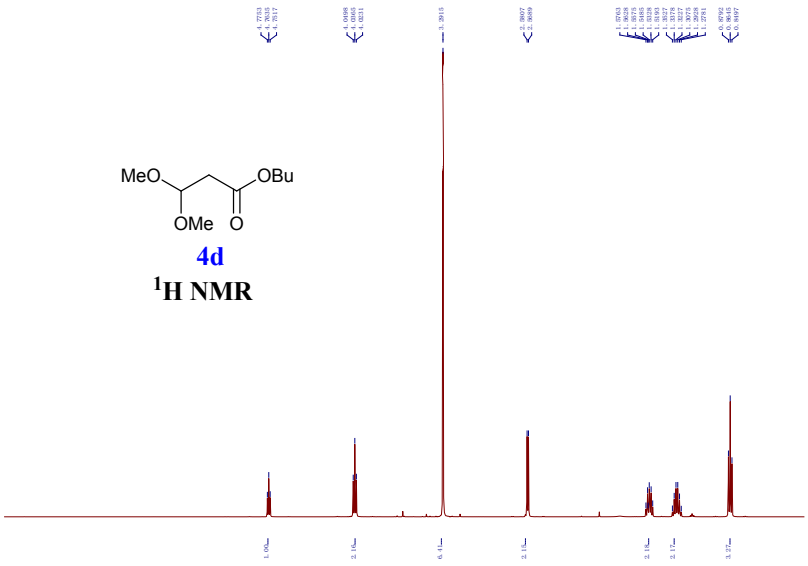

MeO
OEt
OMe
O
4c
13C NMR


MeO
OEt
OMe
O
4c
1H NMR



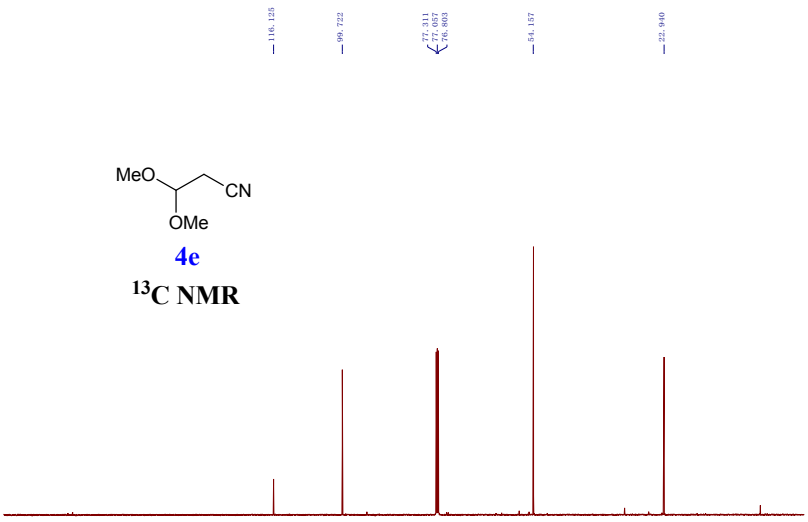
4d

^{13}C NMR



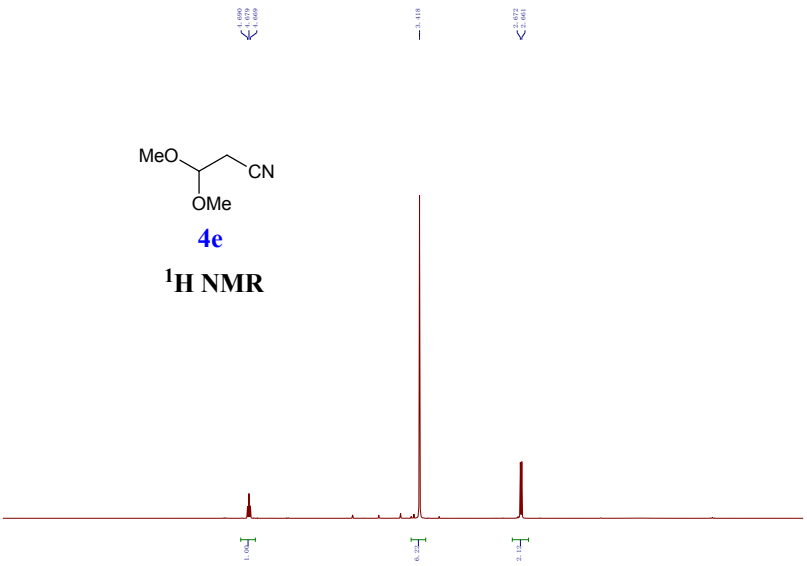
4d

^{1}H NMR



4e

^{13}C NMR



4e

^{1}H NMR