Supporting Information

Synthesis of Symmetrical Secondary Oligoethylene Glycolated Amines from Diethanolamine

Hao Yang,[a†] Yu Li,[b†] Ruoyun Lin,[a] Zhen Ouyang,[a] Mingli Han,[a] Lijun Zhu,[a] Shizhen Chen,[b] Xin Zhou,[b] and Zhong-Xing Jiang*[b]

[a]*Hubei Province Engineering and Technology Research Center for Fluorinated Pharmaceuticals, School of Pharmaceutical Sciences, Wuhan University, Wuhan 430071, China*

[b]*State Key Laboratory of Magnetic Resonance and Atomic and Molecular Physics, National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovative Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences, Wuhan 430071, China*

[†]*The authors contributed equally to this work.*

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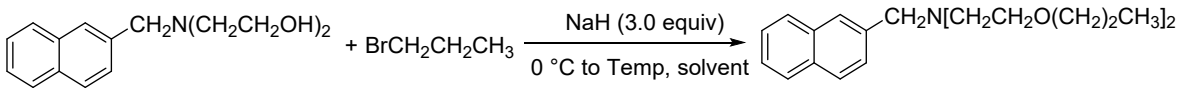
1. General Information

All commercially available reagents and solvents were used as received unless otherwise mentioned. All NMR data were recorded using a Bruker 400 MHz instrument. The chemical shifts are reported as δ ppm using tetramethylsilane (TMS) as the internal standard, and coupling constants *J* values were expressed in hertz. The following abbreviations were used to indicate multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; dd = doublet of doublets; dt = doublet of triplets; m = multiplet. ^{19}F NMR spectra were referenced to 2% perfluorobenzene (s, -164.90 ppm). MALDI-TOF mass spectra were recorded on a MALDI-TOF/TOF 5800 (AB SCIEX) spectrometer using the reflector mode for positive ions with α-cyano-4-hydroxylcinnamic acid as matrix. Crude mixtures were purified by flash column chromatography on silica gel (200–300 mesh).

2. Optimization of Reaction Conditions

Table S1. Optimization of reaction condition for functionalization of **2** with 1-bromopropane [a]

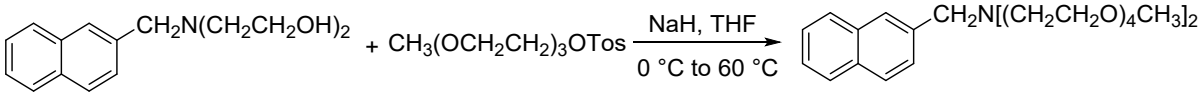


2 **3a** **4a**

Entry	Solvent	**3a** [equiv]	Temp [°C][b]	Yield [%][c]
1	THF	2.5	25	0
2	THF	2.5	60	0
3	DMF	2.5	60	62
4	DMF	2.0	60	37
5	DMF	3.0	60	52

[a]Unless otherwise stated, reactions were carried out with **2** (0.40 mmol), **3a**, and NaH (1.20 mmol) in solvent for 10 min to 12 h. [b]The reaction temperature after the addition of **3a**. [c]Isolated yields.

Table S2. Optimization of reaction condition for functionalization of **2** with tosylate triethylene glycol monomethyl ether[a]



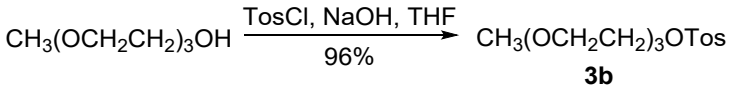
2 **3b** **4b**

Entry	**3b** [equiv]	NaH [equiv]	Yield [%][b]
1	2.5	3.0	74
2	2.0	2.3	50
3	2.3	2.3	68

[a]Unless otherwise stated, reactions were carried out with **2** (1.0 equiv), **3b**, and NaH in THF for 10 min to 12 h. [b]Isolated yields.

3. Synthetic procedures

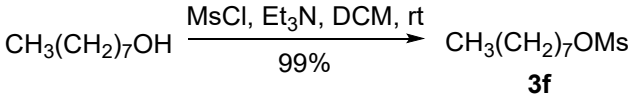
3.1 Synthesis of alkyl mesylate 3b[1]



3b

To a stirring solution of methoxy triethylene glycol (8.0 g, 48.72 mmol) and NaOH (7.8 g, 195.00 mmol, in 24 mL water) in THF (80 mL) was added a solution of *p*-toluenesulfonyl chloride (18.6 g, 97.57 mmol, in 50 mL THF) slowly. After the addition, the mixture was stirred at rt for 3 h. Then, the reaction mixture was diluted with brine (200 mL) and extracted with DCM (150 mL, three times). The combined organic layers were dried over anhydrous Na_2SO_4, concentrated under vacuum, and purified by flash chromatography on silica gel to give **3b** (14.8 g, 96% yield) as a yellowish oil (chromatographic eluent: DCM/MeOH = 20/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 4.15–4.00 (m, 2H), 3.65–3.38 (m, 10H), 3.27 (s, 3H), 2.35 (s, 3H).

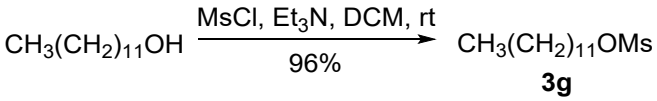
3.2 Synthesis of alkyl mesylate 3f[2]



3f

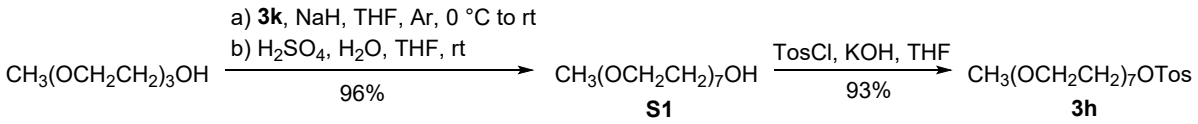
1-Octanol (0.50 g, 3.84 mmol) and Triethylamine (Et_3N, 1.07 mL, 0.78 g, 7.71 mmol) were dissolved in DCM (20 mL). Then methanesulfonyl chloride (MsCl, 0.60 mL, 0.88 g, 7.71 mmol) was added to the mixture, and the resulting reaction mixture was stirred at rt for 12 h. The reaction mixture was diluted with brine (100 mL) and extracted with DCM (50 mL, three times). The combined organic layers were dried over anhydrous Na_2SO_4, concentrated under vacuum, and purified by flash chromatography on silica gel to give **3f** (0.80 g, 99% yield) as a yellowish oil (chromatographic eluent: PE/EtOAc = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.22 (t, J = 6.6 Hz, 2H), 3.01 (s, 3H), 1.74 (dd, J = 14.6, 6.8 Hz, 2H), 1.44–1.22 (m, 10H), 0.88 (t, J = 6.7 Hz, 3H).

3.3 Synthesis of alkyl mesylate 3g[3]



0.68 g of alkyl mesylate **3g** was prepared from 1-dodecanol (0.50 g, 2.68 mmol) in 96% yield (chromatographic eluent: PE/EtOAc = 5/1) as a white wax by employing the same synthetic procedures as compound **3f**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.24 (t, J = 6.6 Hz, 2H), 3.02 (s, 3H), 1.81–1.71 (m, 2H), 1.45–1.23 (m, 19H), 0.90 (t, J = 6.8 Hz, 3H).

3.4 Synthesis of tosylate M-OEGs 3h[4]

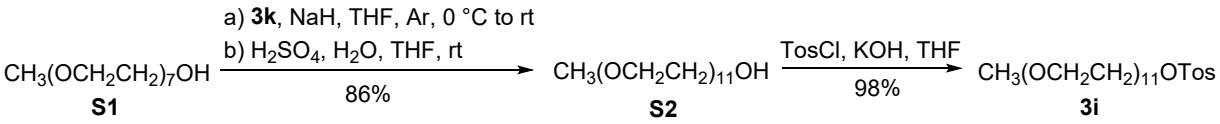


Step 1: A suspension of NaH (60% dispersed in mineral oil, 1.5 g, 37.50 mmol) in THF (10 mL) was cooled to 0 ℃ under an argon atmosphere, then a solution of methoxytriglycol (5.0 g, 30.45 mmol) in THF (40 mL) was added. After stirring for 15 min at this temperature, a solution of tetraethylene glycol MCS **3k** (9.4 g, 36.68 mmol) in THF (50 mL) was added. The resulting mixture was warmed to rt and stirred for 6 h. Then, water (0.85 mL, 47.22 mmol) was added to the mixture, and H_2SO_4 was added to adjust the pH to 3.0. The mixture was stirred for an additional 4 h at rt. Upon the completion of hydrolysis, the reaction was neutralized with saturated $NaHCO_3$ solution, concentrated under vacuum to give a residue, which was purified

by flash column chromatography on silica gel to give compound **S1**[5] (9.8 g, 96% yield) as a slightly yellow oil (chromatographic eluent: DCM/MeOH = 15/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72–3.69 (m, 2H), 3.63 (t, *J* = 5.1 Hz, 22H), 3.60–3.57 (m, 2H), 3.53 (dd, *J* = 5.8, 3.5 Hz, 2H), 3.36 (s, 3H).

Step 2: To a stirring solution of **S1** (1.0 g, 2.94 mmol) and KOH (0.66 g, 11.74 mmol, in 1.5 mL water) in THF (10 mL) was added a solution of *p*-toluenesulfonyl chloride (1.12 g, 5.87 mmol, in 25 mL THF) slowly. After the addition, the mixture was stirred at rt for 12 h. Then, the reaction mixture was diluted with brine (150 mL) and extracted with DCM (100 mL, three times). The combined organic layers were dried over anhydrous Na_2SO_4, concentrated under vacuum, and purified by flash chromatography on silica gel to give **3h** (1.3 g, 93% yield) as a slightly yellow oil (chromatographic eluent: DCM/MeOH = 20/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 4.17–4.13 (m, 2H), 3.70–3.53 (m, 26H), 3.38 (s, 3H), 2.45 (s, 3H).

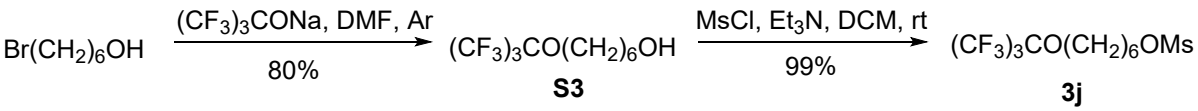
3.5 Synthesis of tosylate M-OEGs 3i[6]



Step 1: M-OEGs **S2**[7] was prepared from M-OEGs **S1** and MCS **3k** as a slightly yellow oil in 86% yield (chromatographic eluent: DCM/MeOH = 15/1) by employing the same synthetic procedures as M-OEGs **S1**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73–3.53 (m, 44H), 3.37 (s, 3H).

Step 2: 1.9 g of tosylate M-OEGs **3i** was prepared from M-OEG **S2** (1.5 g, 2.90 mmol) as a slightly yellow oil in 98% yield (chromatographic eluent: DCM/MeOH = 15/1) by employing the same synthetic procedures as **3h**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83–7.76 (m, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 4.16 (dd, *J* = 5.4, 4.3 Hz, 2H), 3.71–3.53 (m, 42H), 3.38 (s, 3H), 2.45 (s, 3H).

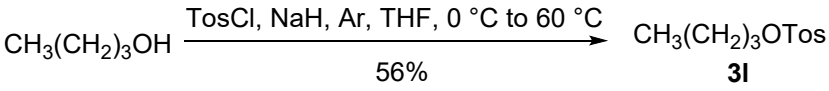
3.6 Synthesis of alkyl mesylate 3j



Step 1: Under an argon atmosphere, sodium perfluoro-*tert*-butoxide (0.86 g, 3.33 mmol) was added to a solution of 6-bromo-1-hexanol (0.50 g, 2.76 mmol) in DMF (20 mL), the resulting reaction mixture was stirred at rt for 24 h. Then DMF was removed under reduce pressure, and the residue was purified by flash chromatography on silica gel to give **S3** (0.74 g, 80% yield) as a slightly yellow oil (chromatographic eluent: PE/EtOAc = 3/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.00 (t, *J* = 6.3 Hz, 2H), 3.61 (t, *J* = 6.6 Hz, 2H), 1.73–1.64 (m, 2H), 1.61–1.53 (m, 2H), 1.45–1.34 (m, 4H).^{19}F NMR (376 MHz, $CDCl_3$) δ -73.33. ^{13}C NMR (101 MHz, $CDCl_3$) δ 120.4 (q, *J* = 290.9 Hz), 79.7 (dd, *J* = 59.0, 29.5 Hz), 69.6, 62.4, 32.4, 29.6, 25.2, 25.1. HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{10}H_{14}F_9O_2^+$,337.0845; found,337.0887.

Step 2: 0.36 g of alkyl mesylate **3j** was prepared from **S3** (0.20 mg, 0.59 mmol) in 99% yield (chromatographic eluent: PE/EtOAc = 5/1) as a yellowish oil by employing the same synthetic procedures as compound **3f**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 (t, *J* = 6.4 Hz, 2H), 4.03 (t, *J* = 6.1 Hz, 2H), 3.03 (s, 3H), 1.83–1.68 (m, 4H), 1.52-1.42 (m, 4H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -73.57. ^{13}C NMR (101 MHz, $CDCl_3$) δ 120.4 (q, *J* = 293.6 Hz), 79.4 (dd, *J* = 58.8, 29.3 Hz), 69.8, 69.5, 37.3, 29.5, 29.0, 25.0, 24.9. HRMS (ESI) m/z: $[M+K]^+$ calcd for $C_{11}H_{15}F_9KO_4S^+$: 453.0179, found 453.0179.

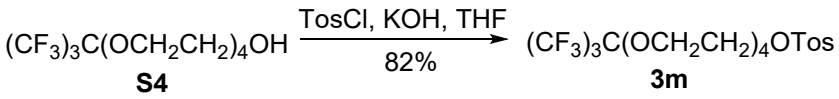
3.7 Synthesis of tosylate M-OEGs 3l[8]



Under an argon atmosphere, a suspension of NaH (60% dispersed in mineral oil, 0.20 g, 4.88 mmol) in THF (5 mL) was cooled to 0 ℃, then a solution of 1-butanol (0.3 g, 4.05 mmol) in THF (10 mL) was added. After stirring for 10 min at this temperature, a solution of *p*-toluenesulfonyl chloride (0.93 g, 4.88 mmol) in THF (10 mL) was added. The resulting mixture was heated to 60 ℃ and stirred for 12 h. After quenching with water, the reaction mixture was washed with brine (100 mL) and extracted with DCM (100 mL, three times). The combined organic layers were dried over anhydrous Na_2SO_4, filtrated, and concentrated under vacuum. The crude product was purified by flash column chromatography on silica gel to give **3l** (0.51

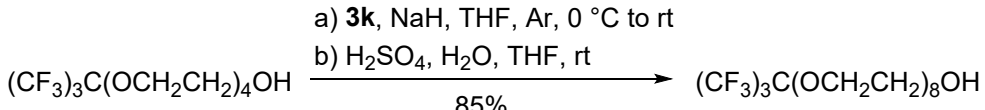
g, 56% yield) as a yellowish oil (chromatographic eluent: PE/EtOAc = 5/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82–7.76 (m, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 4.03 (t, *J* = 6.5 Hz, 2H), 2.45 (s, 3H), 1.66–1.58 (m, 2H), 1.34 (dd, *J* = 15.0, 7.5 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

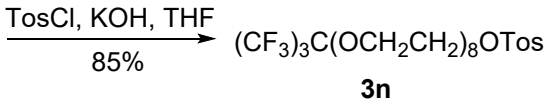
3.8 Synthesis of tosylate M-OEGs 3m



1.1 g of tosylate M-OEGs **3m** was prepared from **S4**[9] (1.0 g, 2.43 mmol) as a slightly yellow oil in 82% yield (chromatographic eluent: PE/EtOAc = 1/1) by employing the same synthetic procedures as **3h**, except that 6.0 equiv of KOH (0.82 g, 14.54 mmol) was used. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 4.18–4.13 (m, 4H), 3.75–3.57 (m, 12H), 2.45 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -73.59. ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8, 132.9, 129.8, 128.0, 120.3 (q, *J* = 298.0 Hz), 80.4-79.3 (m), 71.0, 70.7, 70.60, 70.55, 69.4, 69.2, 68.6, 21.6. HRMS (ESI) m/z: $[M + H]^+$ calcd for $C_{19}H_{24}F_9O_7S^+$,567.1094; found,.567.1088.

3.9 Synthesis of tosylate M-OEGs 3n



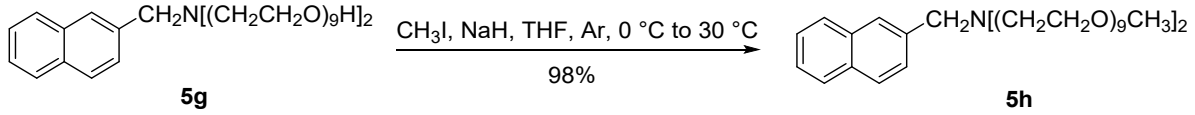


Step 1: 1.2 g of compound **S5**[9] was prepared from **S4** (1.0 g, 2.43 mmol) and MCS **3k** (0.75 g, 2.91 mmol) as a slightly yellow oil in 85% yield (chromatographic eluent: DCM/MeOH = 15/1) by employing the same synthetic procedures as M-OEGs **S1**.

Step 2: 1.2 g of tosylate M-OEGs **3n** was prepared from **S5** (1.1 g, 1.90 mmol) as a slightly yellow oil in 85% yield (chromatographic eluent: DCM/MeOH = 20/1) by employing the same synthetic procedures as **3h**, except that 8.0 equiv of KOH (0.85 g, 15.20 mmol) was used. ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 4.16 (dd, *J* = 5.4, 4.2 Hz, 4H), 3.75–3.57 (m, 29H), 2.45 (s, 3H). ^{19}F NMR (376 MHz, $CDCl_3$) δ -73.56. ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8, 132.9, 129.8, 128.0, 120.3 (q, *J* = 293.6 Hz), 79.7 (dd, *J* = 59.2, 29.6 Hz), 71.0, 70.7, 70.5, 69.4, 69.3, 68.6, 21.6. HRMS (ESI) m/z: $[M+K]^+$ calcd for $C_{27}H_{40}F_9O_{11}S^+$: 743.2142, found 743.2142.

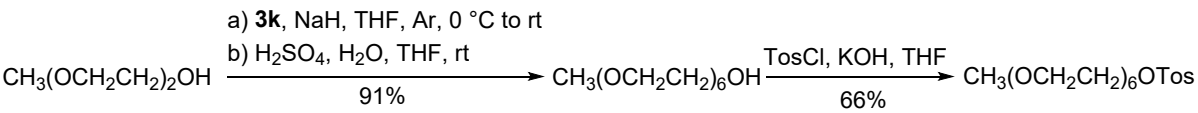
3.10 Synthesis of derivative 5h



5g **5h**

To a suspension of NaH (60% dispersed in mineral oil, 12 mg, 0.30 mmol) in THF (1.0 mL) was slowly added a solution of diol **5g** (90 mg, 0.10 mmol) in THF (5.0 mL) at 0 ℃ under an argon atmosphere. The reaction mixture was stirred for 10 min at 0 ℃ and iodomethane (35 mg, 15 μL, 0.25 mmol) was added. Then the resulting mixture was heated to 30 ℃ and stirred for 12 h. After quenching with water, the reaction mixture was washed with saturated NaCl solution and extracted with DCM. The combined organic layers were dried over anhydrous Na_2SO_4, filtrated, and concentrated under vacuum. The crude product was purified by flash column chromatography on silica gel to give **5h** (90 mg, 98% yield) as a yellowish oil (chromatographic eluent: DCM/MeOH = 10/1). ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (t, *J* = 20.4 Hz, 4H), 7.69–7.43 (m, 3H), 4.15 (s, 2H), 3.89–3.40 (m, 72H), 3.38–3.33 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.2, 133.0, 128.2, 127.9, 127.6, 126.2, 71.8, 71.7, 70.45, 70.38, 70.2, 59.5, 59.2, 53.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{49}H_{88}NO_{18}^+$: 978.5996, found 978.5996.

3.11 Synthesis of tosylate M-OEGs 9[10]



7 **8** **9**

Step 1: M-OEGs **8** was prepared from M-OEGs **7** and MCS **3k** as a slightly yellow oil in 91% yield (chromatographic eluent: DCM/MeOH = 15/1) by employing the same synthetic procedures as M-OEGs **S1**. ^1H NMR (400 MHz, $CDCl_3$) δ 3.66–3.47 (m, 24H), 3.31 (s, 3H).

Step 2: 8.7 g of tosylate M-OEGs **9** was prepared from M-OEG **8** (8.8 g, 29.70 mmol) as a slightly yellow oil in 66% yield by employing the same synthetic procedures as **3h** (chromatographic eluent: EtOAc/PE = 3/1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 4.16–4.12 (m, 2H), 3.68–3.52 (m, 22H), 3.36 (s, 3H), 2.43 (s, 3H).

References

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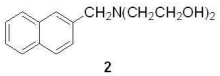
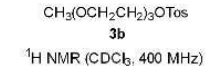
Copies of ^{1}H NMR, ^{13}C NMR, ^{19}F NMR spectra of compounds



2

^{1}H NMR ($CDCl_3$, 400 MHz)



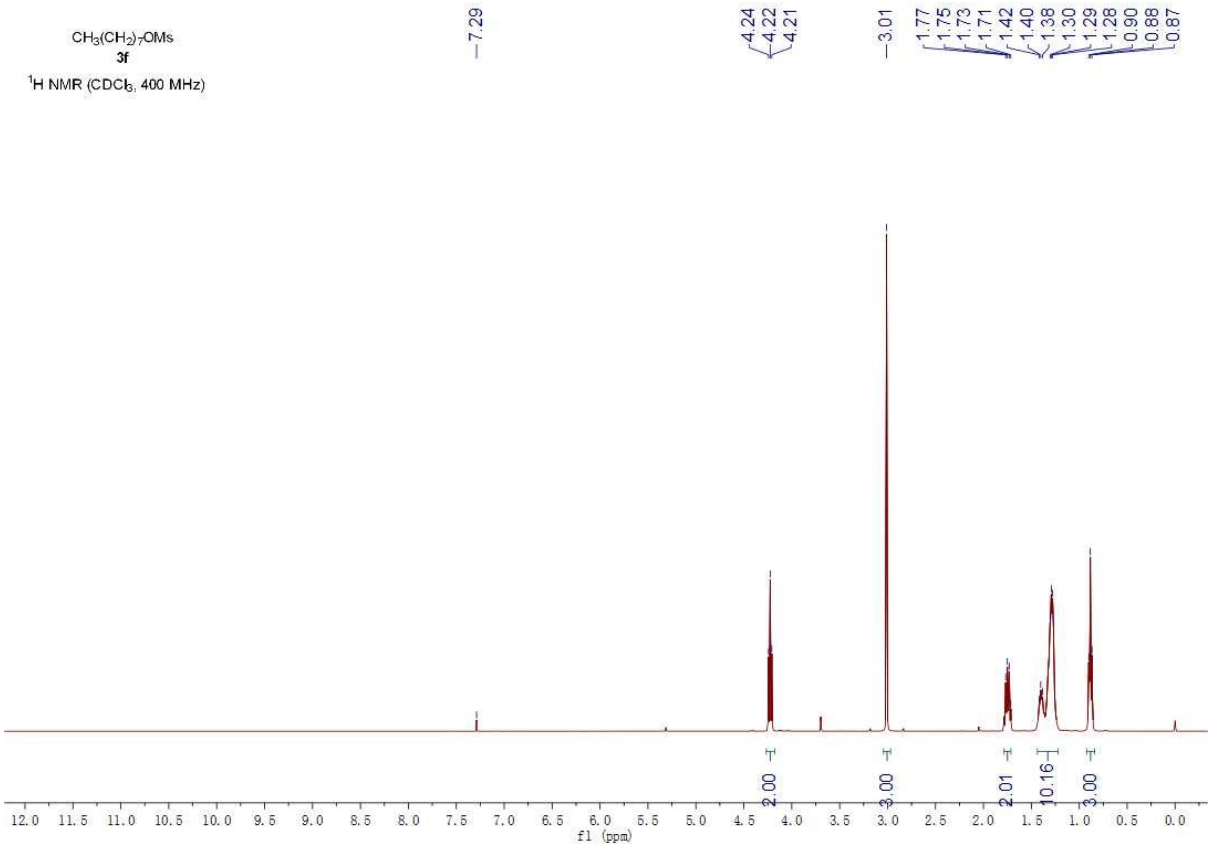
3b

^{1}H NMR ($CDCl_3$, 400 MHz)

$CH_3(CH_2)_7OMs$

3f

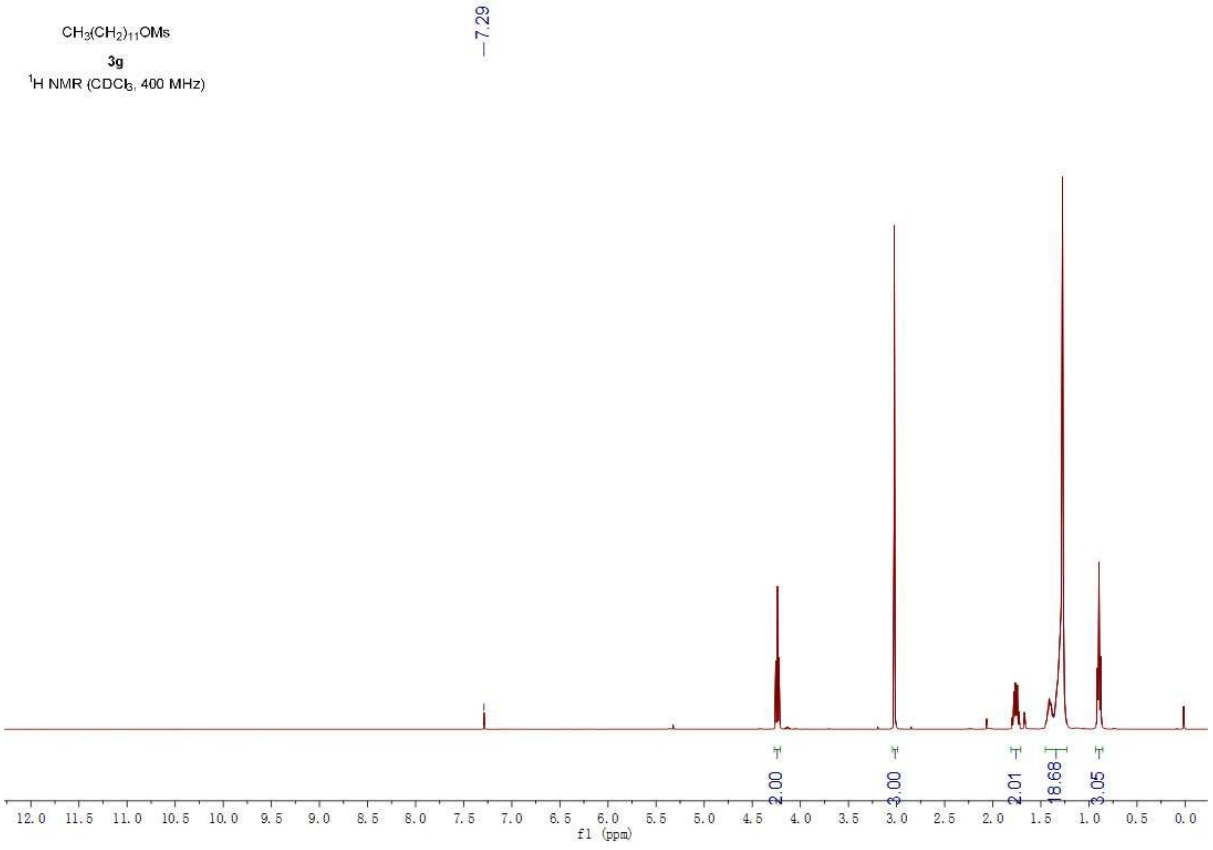
^1H NMR ($CDCl_3$, 400 MHz)



$CH_3(CH_2)_{11}OMs$

3g

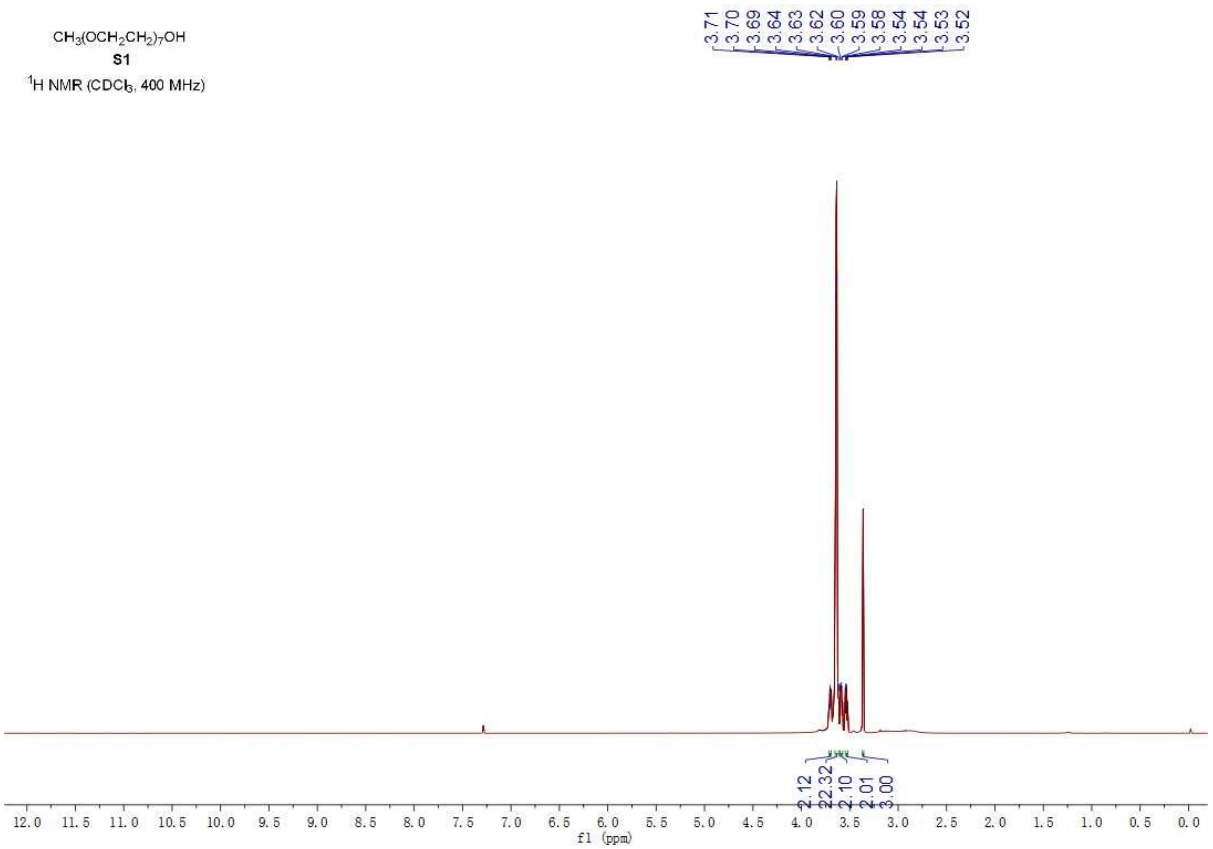
^1H NMR ($CDCl_3$, 400 MHz)



$CH_3(OCH_2CH_2)_7OH$

S1

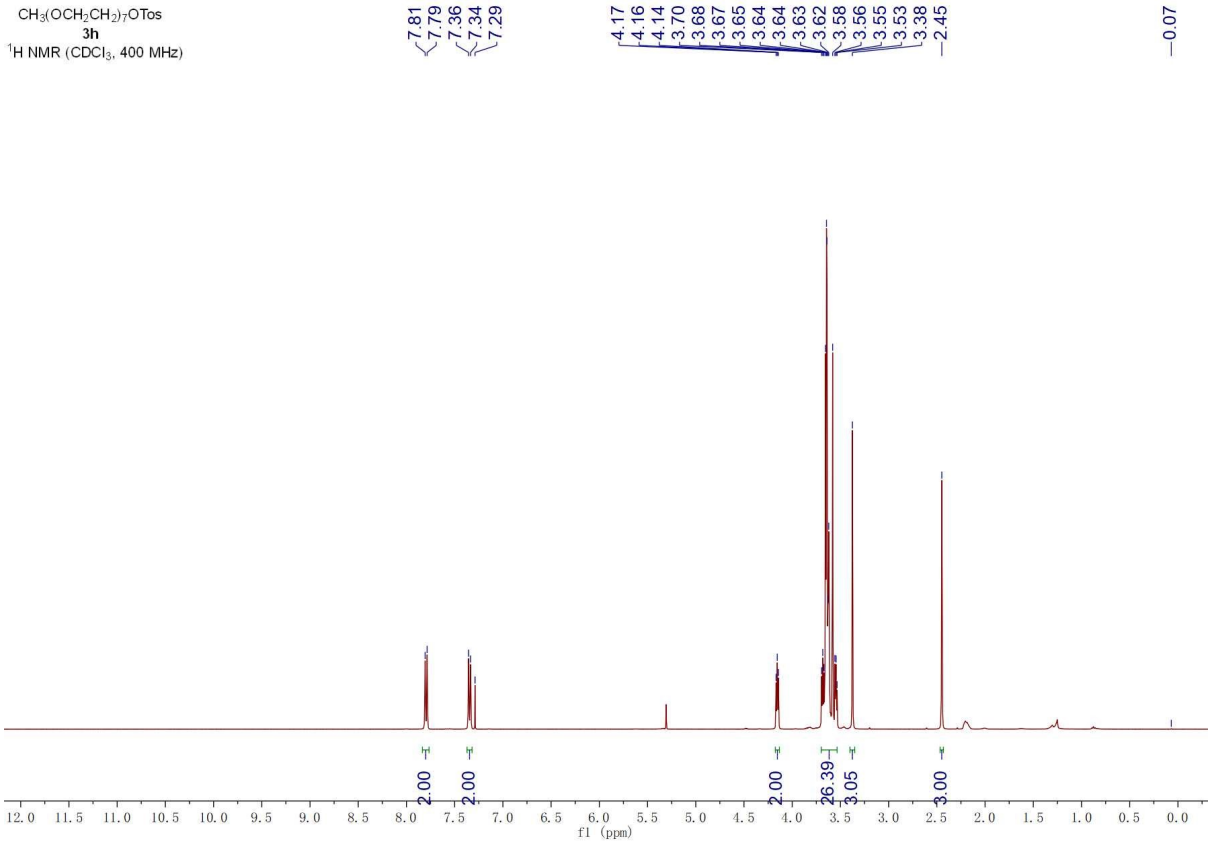
^1H NMR ($CDCl_3$, 400 MHz)



$CH_3(OCH_2CH_2)_7OTos$

3h

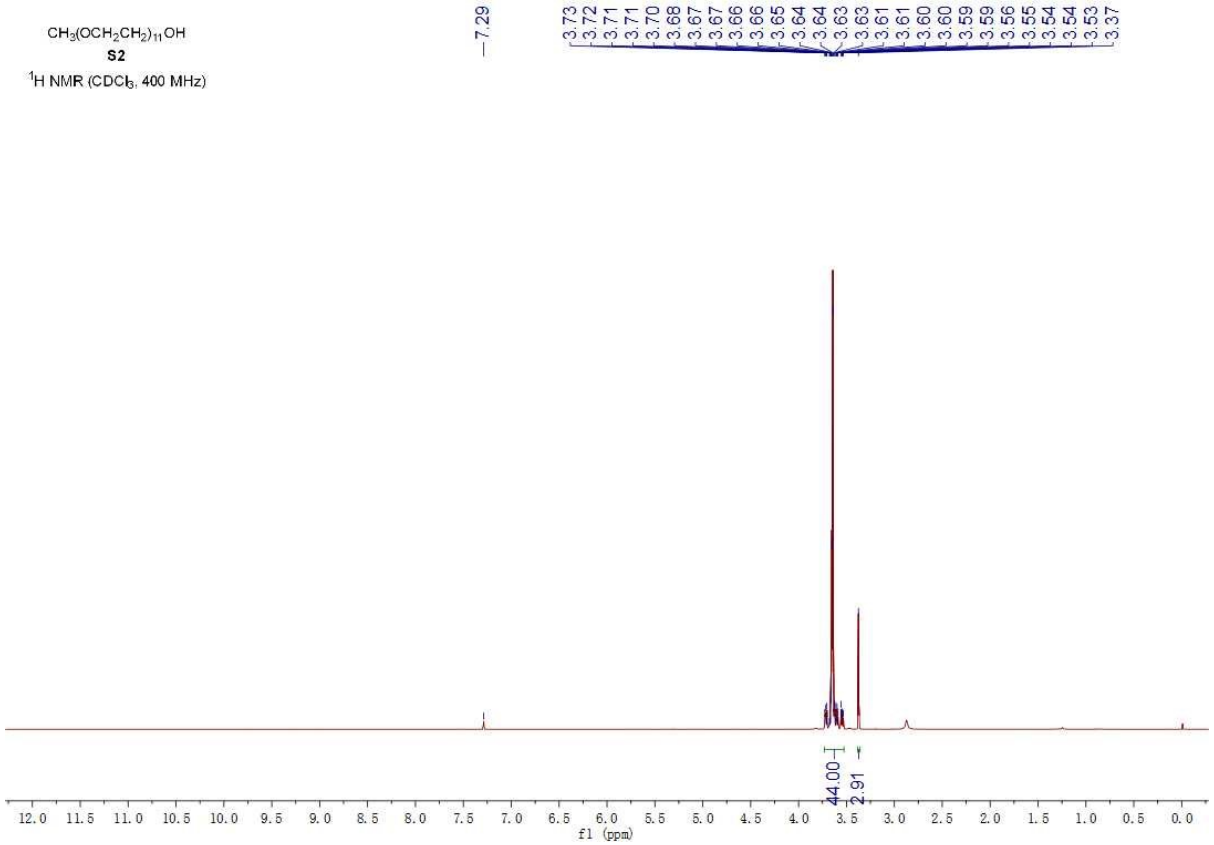
^1H NMR ($CDCl_3$, 400 MHz)



$CH_3(OCH_2CH_2)_{11}OH$

S2

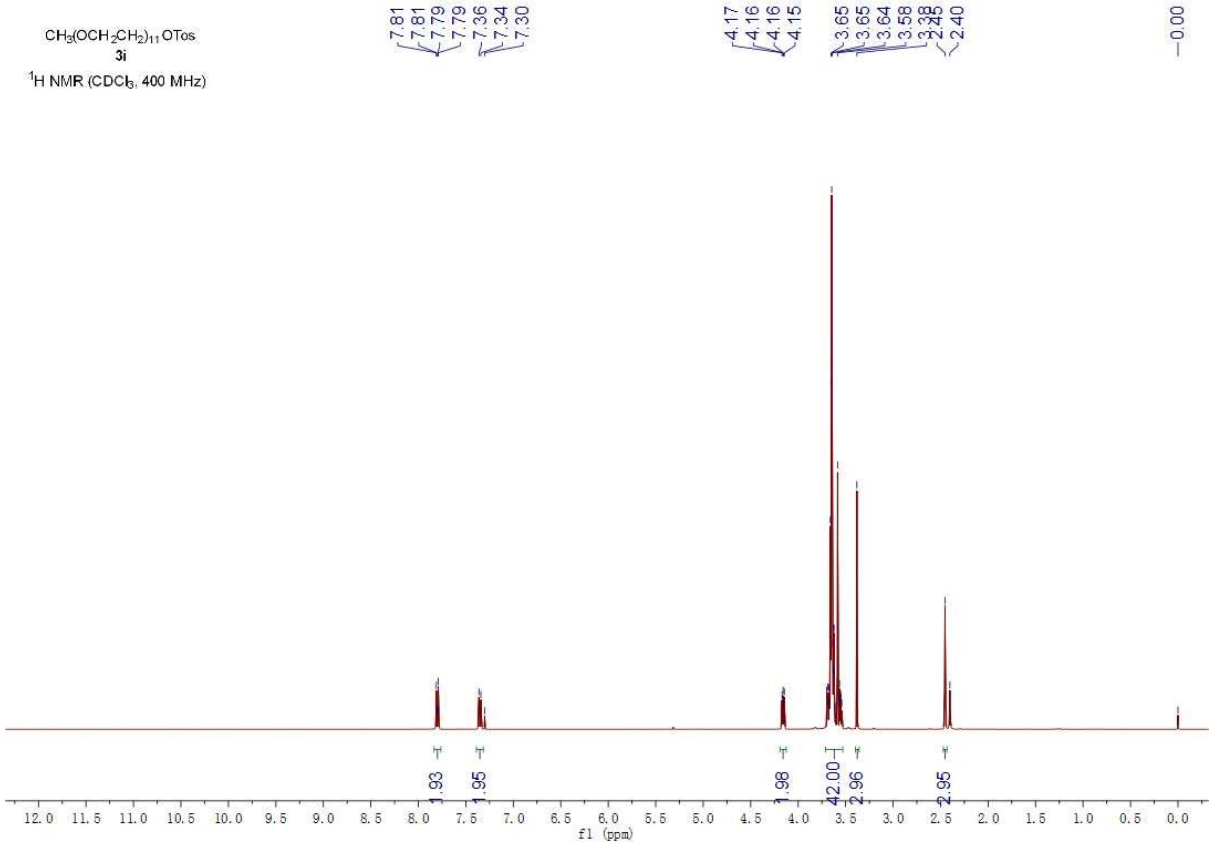
^1H NMR ($CDCl_3$, 400 MHz)



$CH_3(OCH_2CH_2)_{11}OTos$

3i

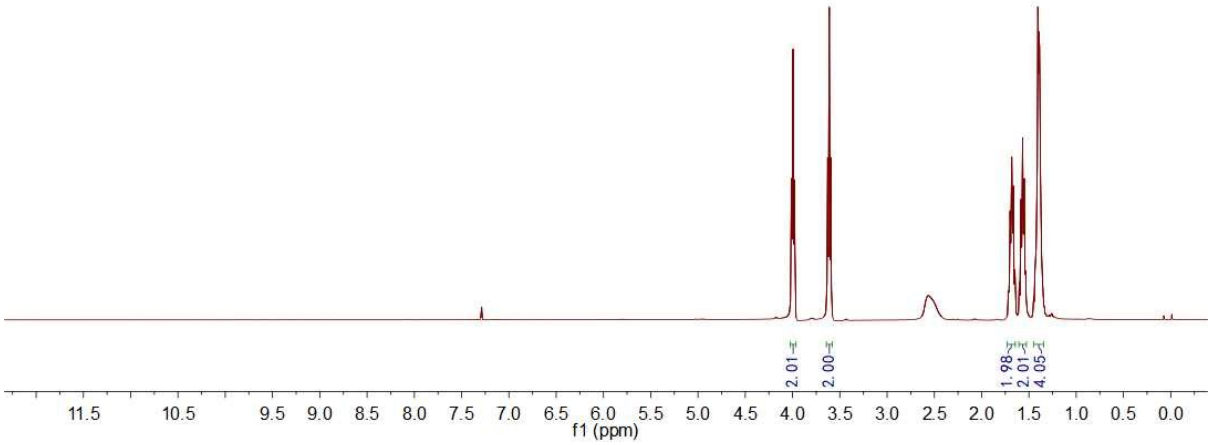
^1H NMR ($CDCl_3$, 400 MHz)



$(CF_3)_3CO(CH_2)_6OH$

S3

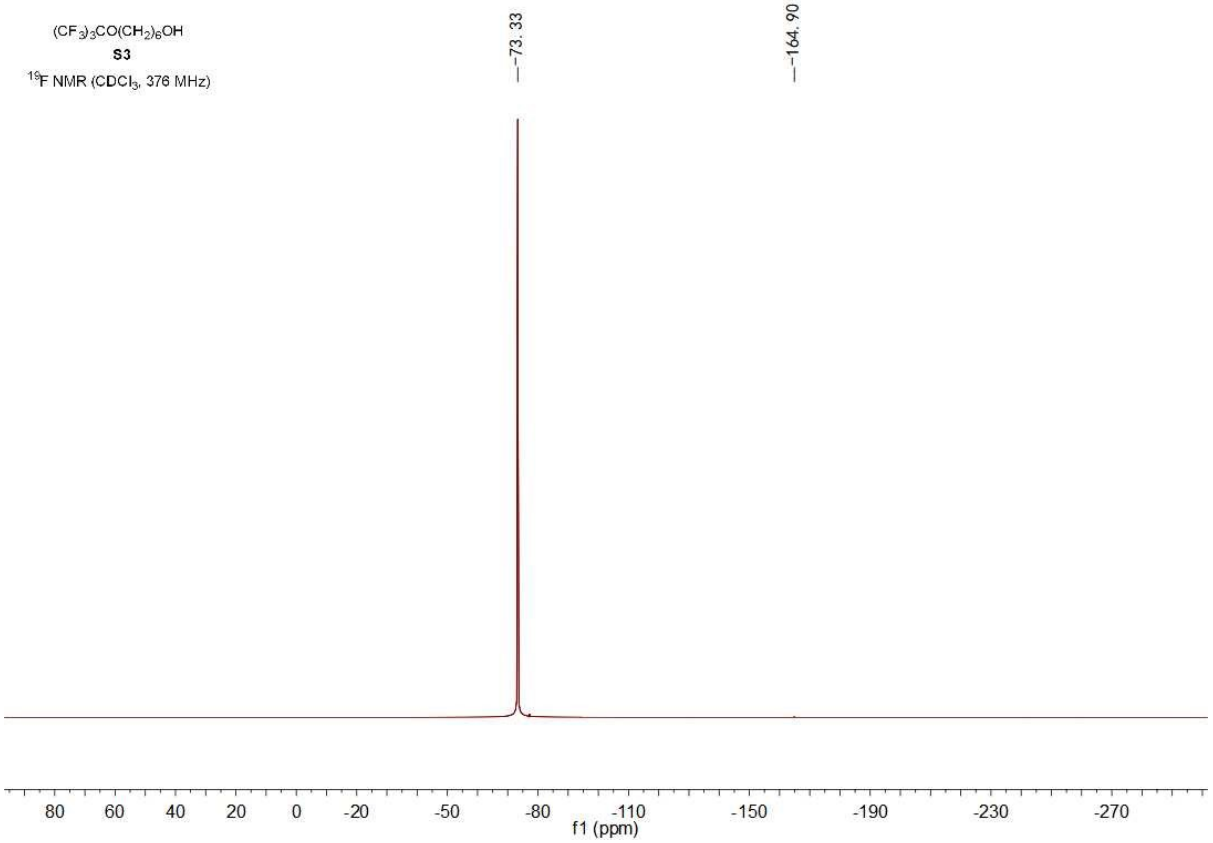
^1H NMR ($CDCl_3$, 400 MHz)



$(CF_3)_3CO(CH_2)_6OH$

S3

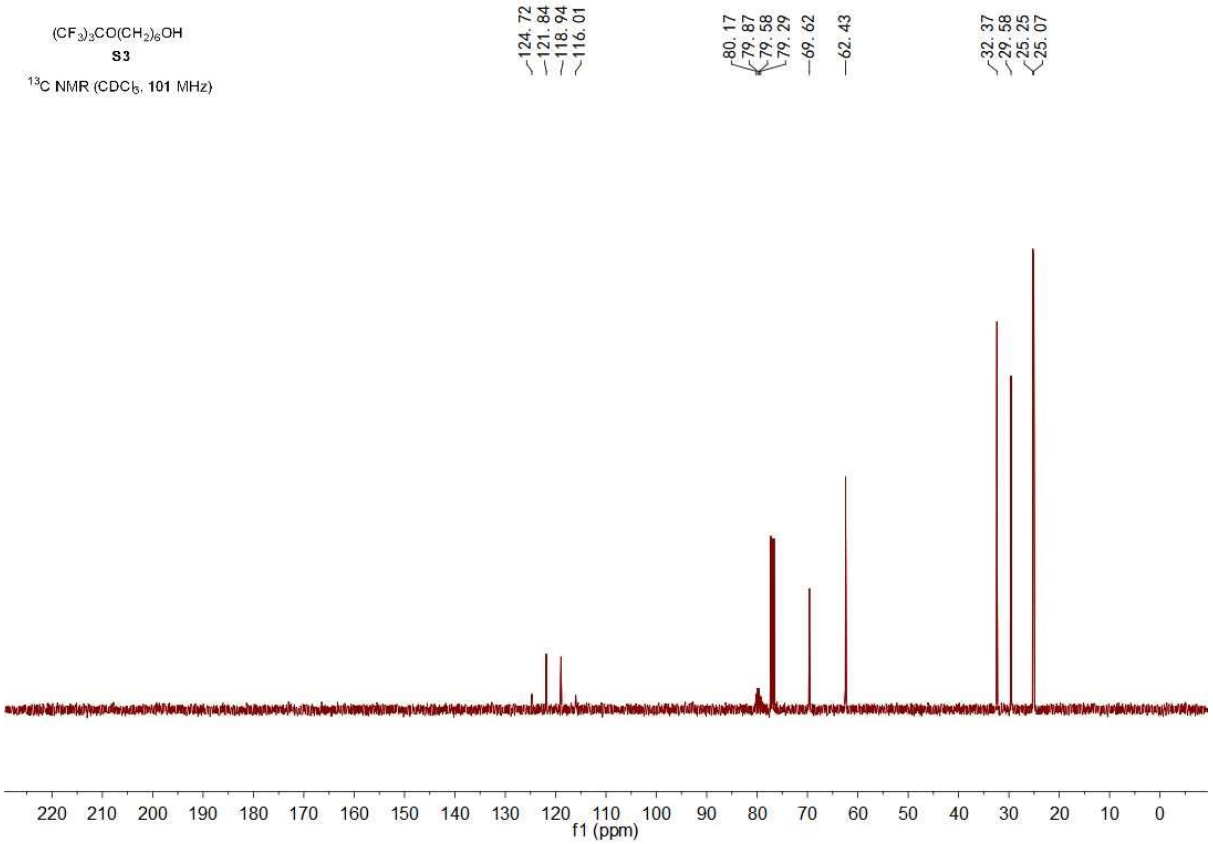
^{19}F NMR ($CDCl_3$, 376 MHz)



$(CF_3)_3CO(CH_2)_6OH$

S3

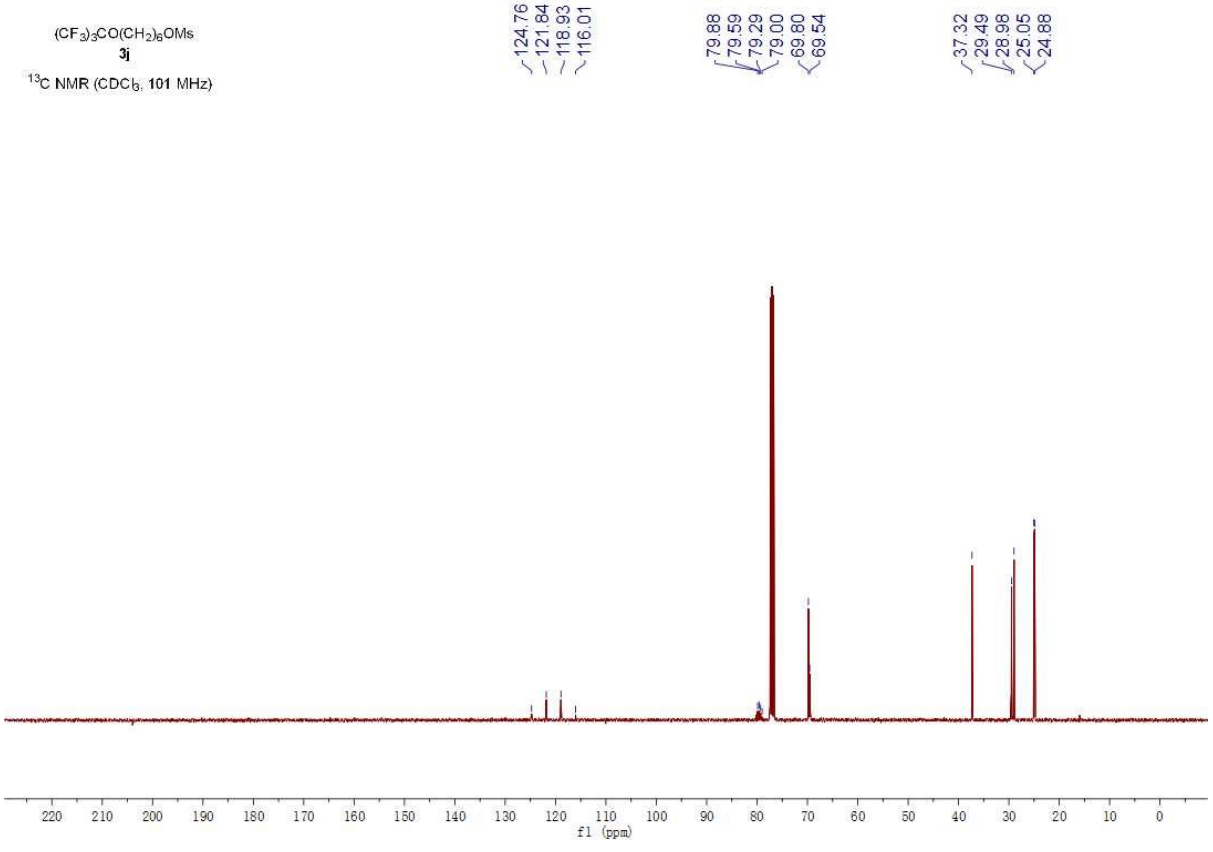
^{13}C NMR ($CDCl_3$, 101 MHz)



$(CF_3)_3CO(CH_2)_6OMs$

3j

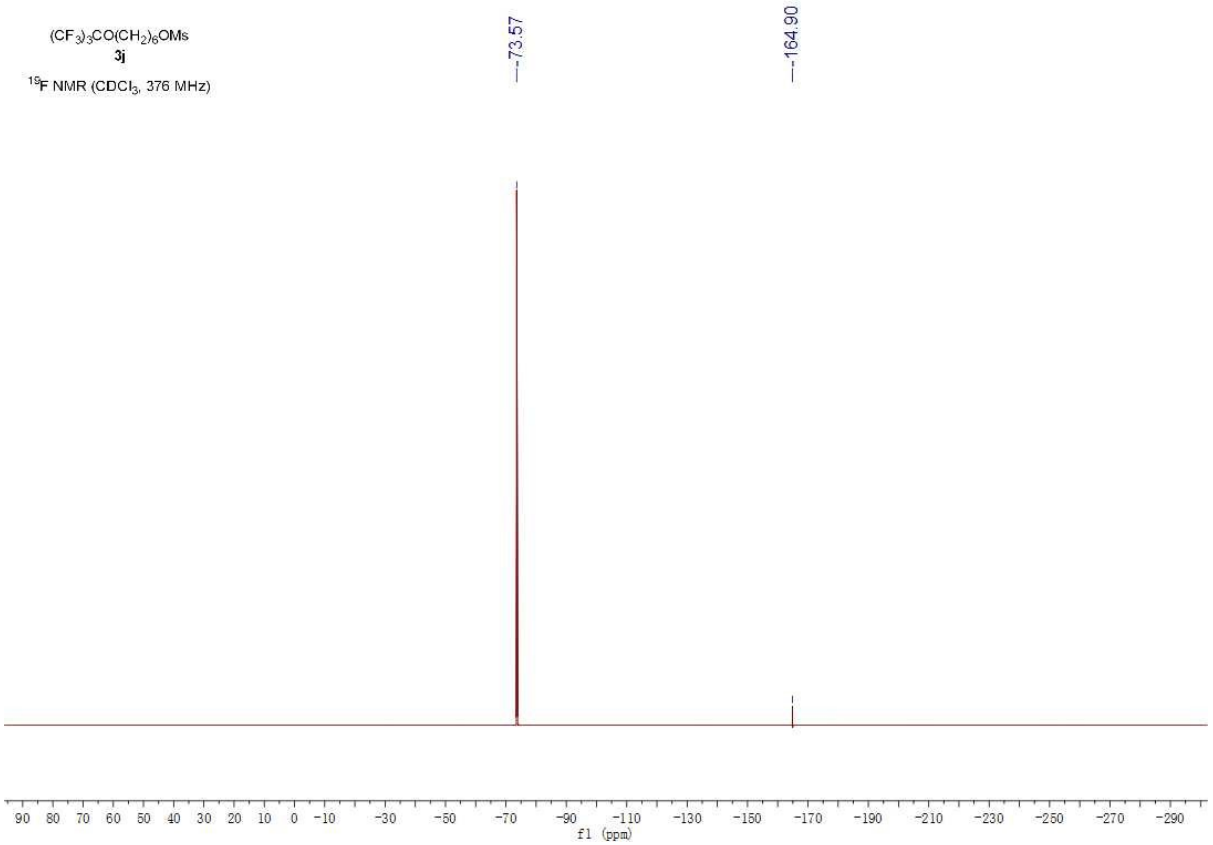
^{13}C NMR ($CDCl_3$, 101 MHz)



$(CF_3)_3CO(CH_2)_6OMs$

3j

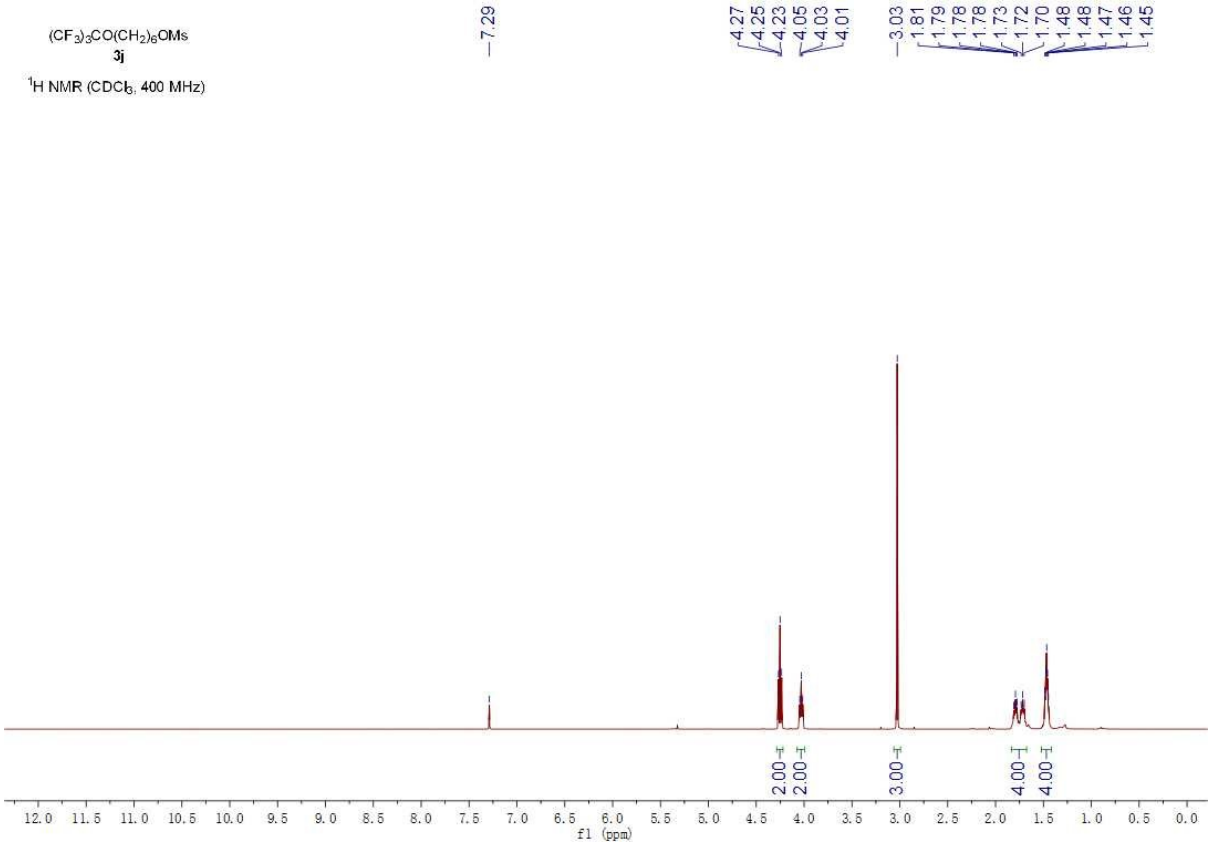
^{19}F NMR ($CDCl_3$, 376 MHz)



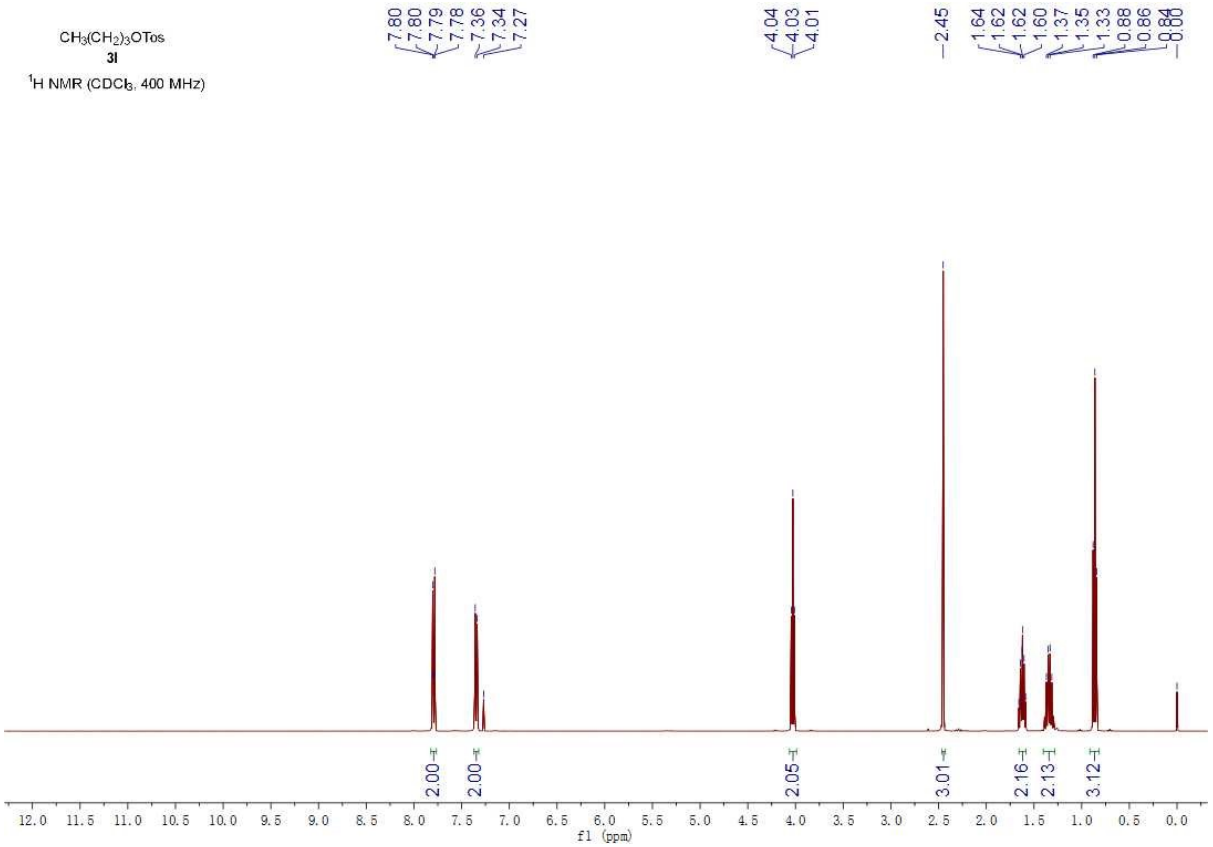
$(CF_3)_3CO(CH_2)_6OMs$

3j

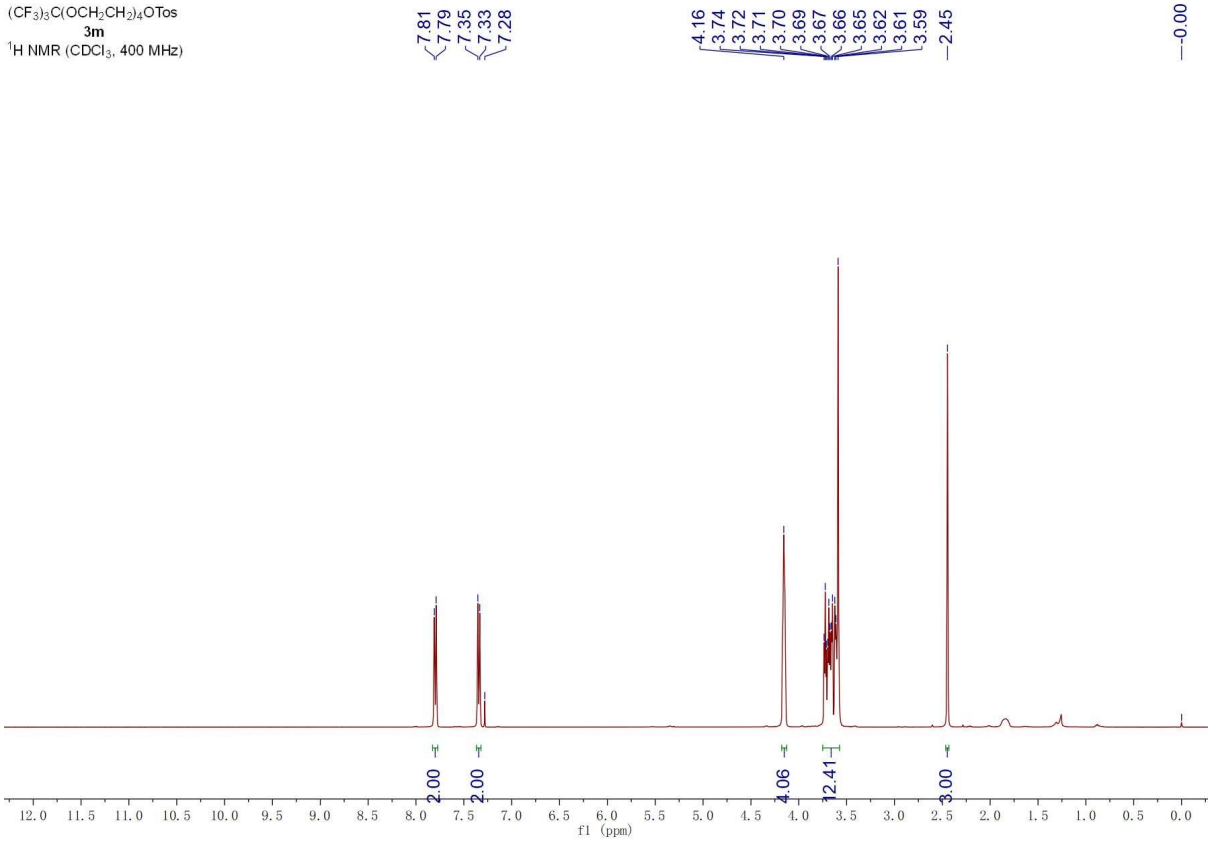
^{1}H NMR ($CDCl_3$, 400 MHz)



$CH_3(CH_2)_3OTos$
3l
^1H NMR ($CDCl_3$, 400 MHz)



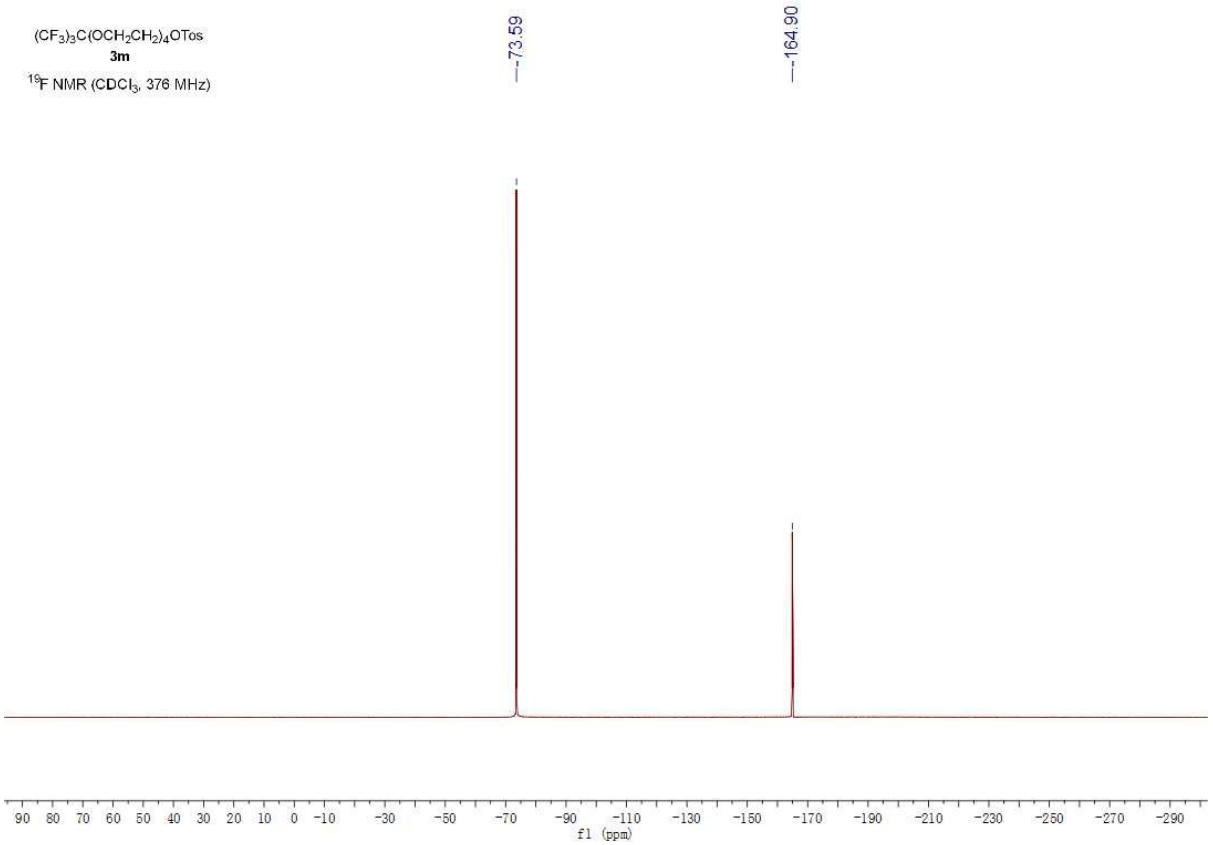
$(CF_3)_3C(OCH_2CH_2)_4OTos$
3m
^1H NMR ($CDCl_3$, 400 MHz)



$(CF_3)_3C(OCH_2CH_2)_4OTos$

3m

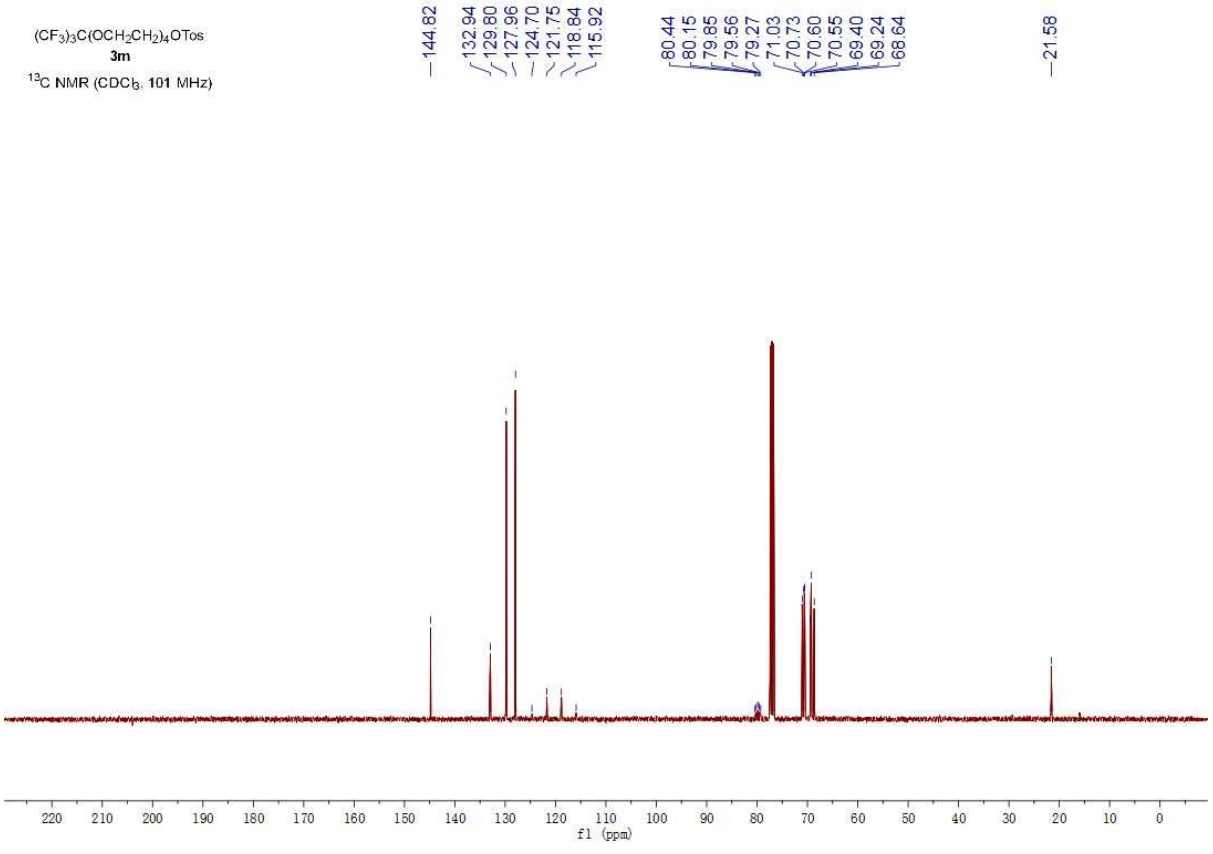
^{19}F NMR ($CDCl_3$, 376 MHz)



$(CF_3)_3C(OCH_2CH_2)_4OTos$

3m

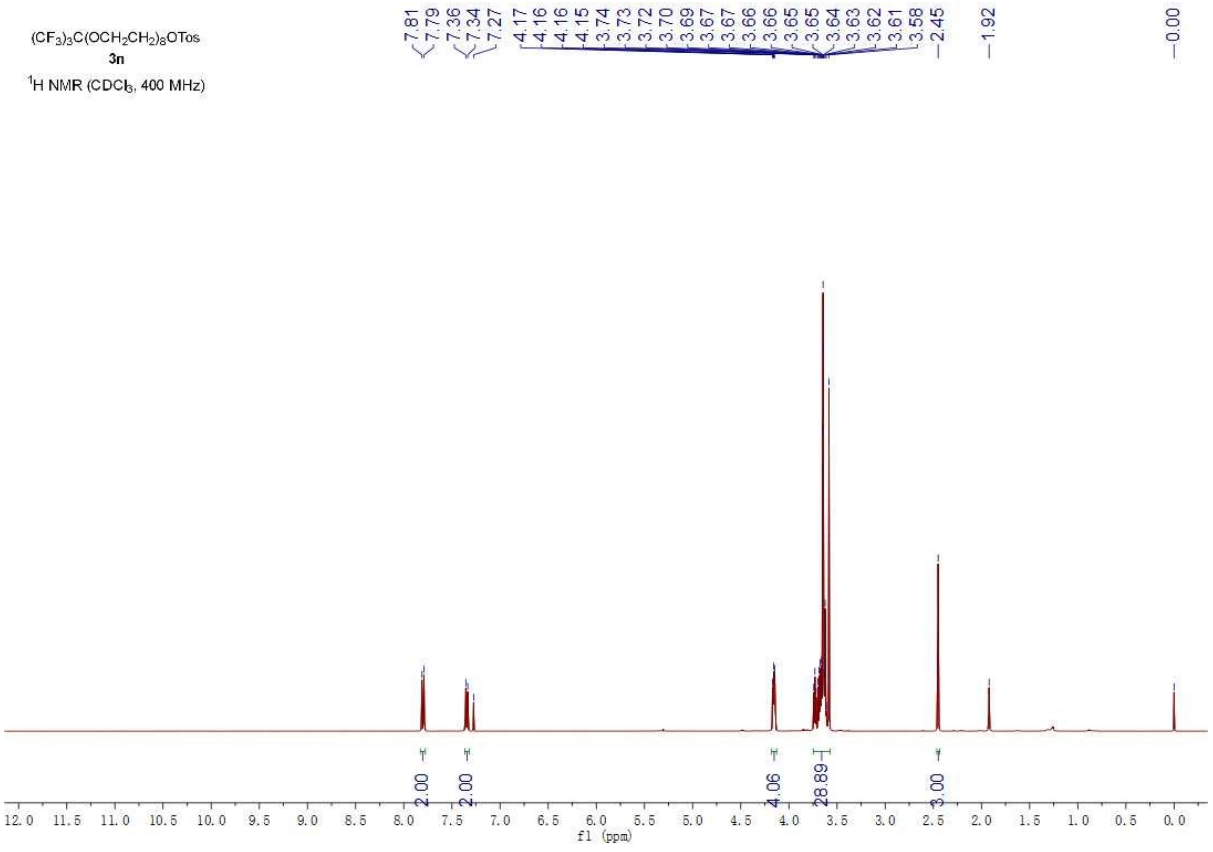
^{13}C NMR ($CDCl_3$, 101 MHz)



$(CF_3)_3C(OCH_2CH_2)_8OTos$

3n

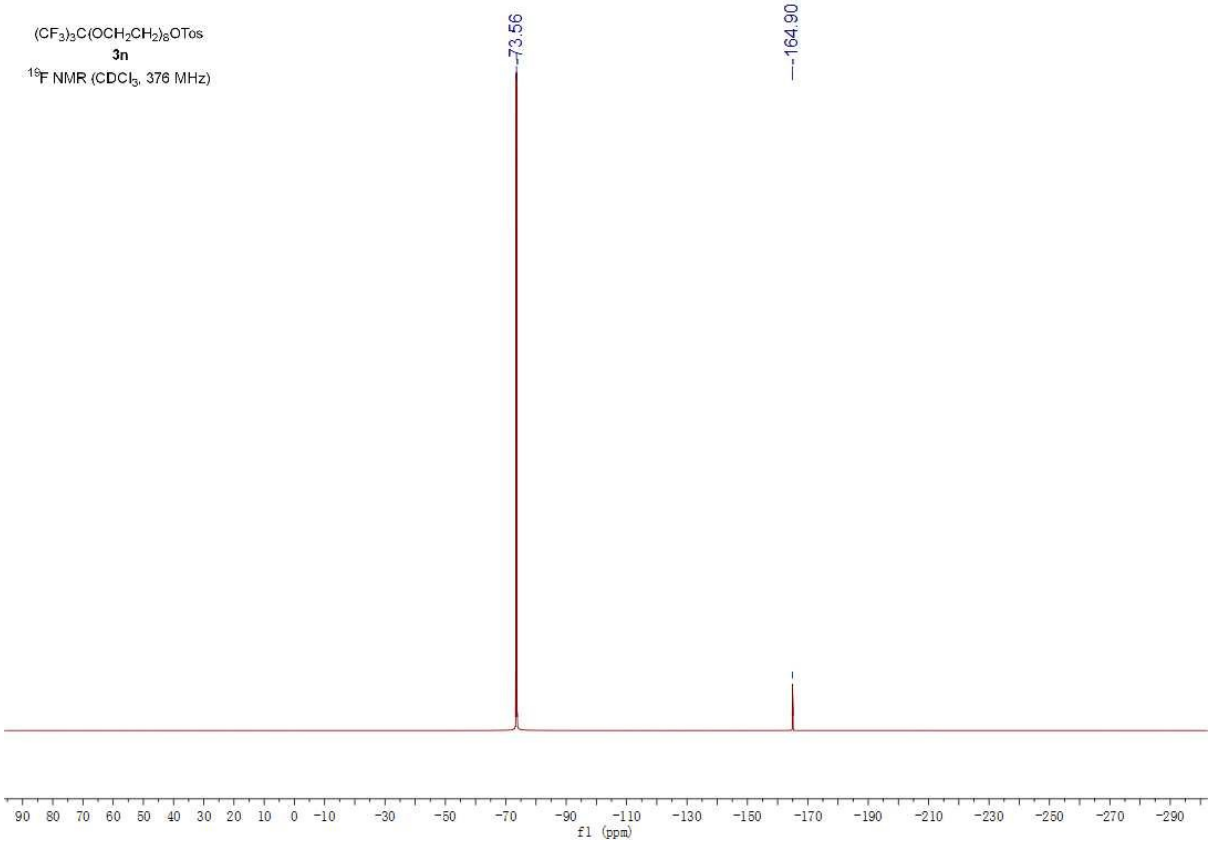
^1H NMR ($CDCl_3$, 400 MHz)



$(CF_3)_3C(OCH_2CH_2)_8OTos$

3n

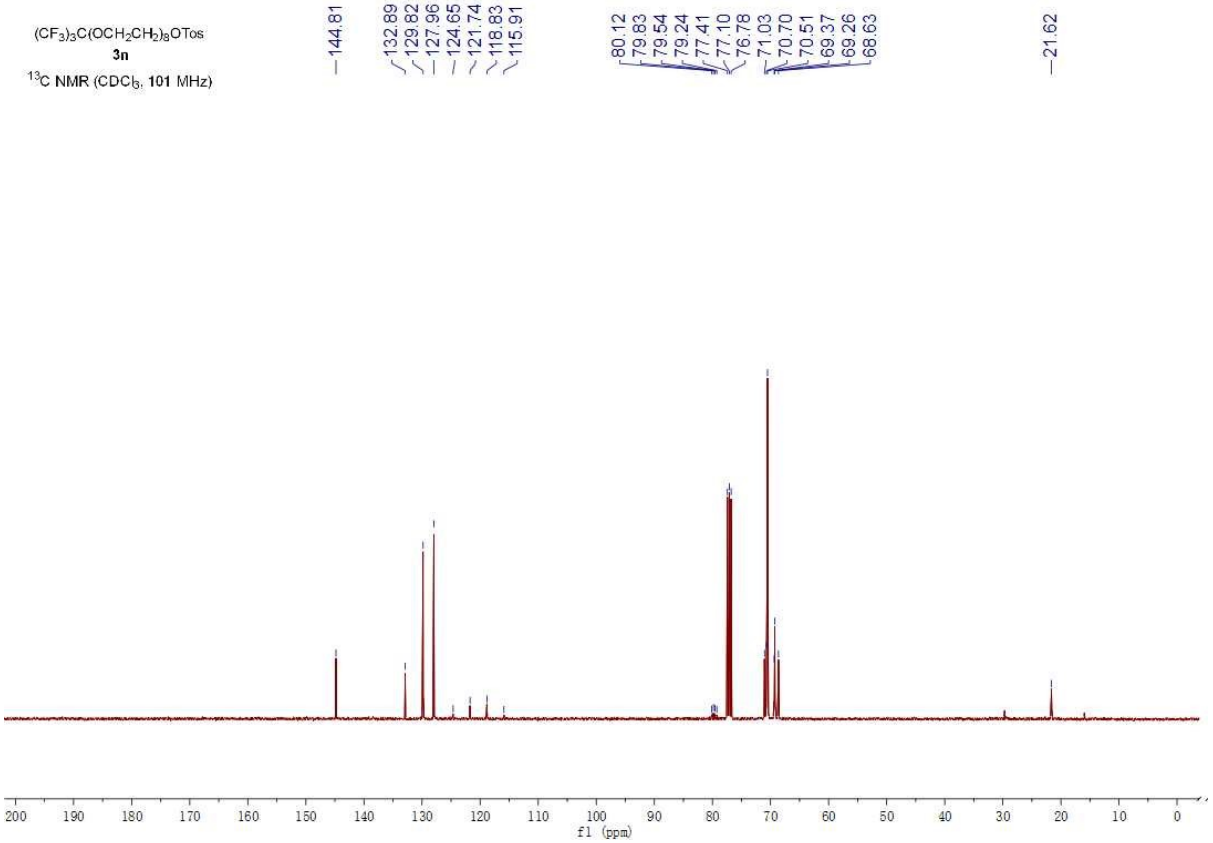
^{19}F NMR ($CDCl_3$, 376 MHz)



$(CF_3)_3C(OCH_2CH_2)_8OTos$

3n

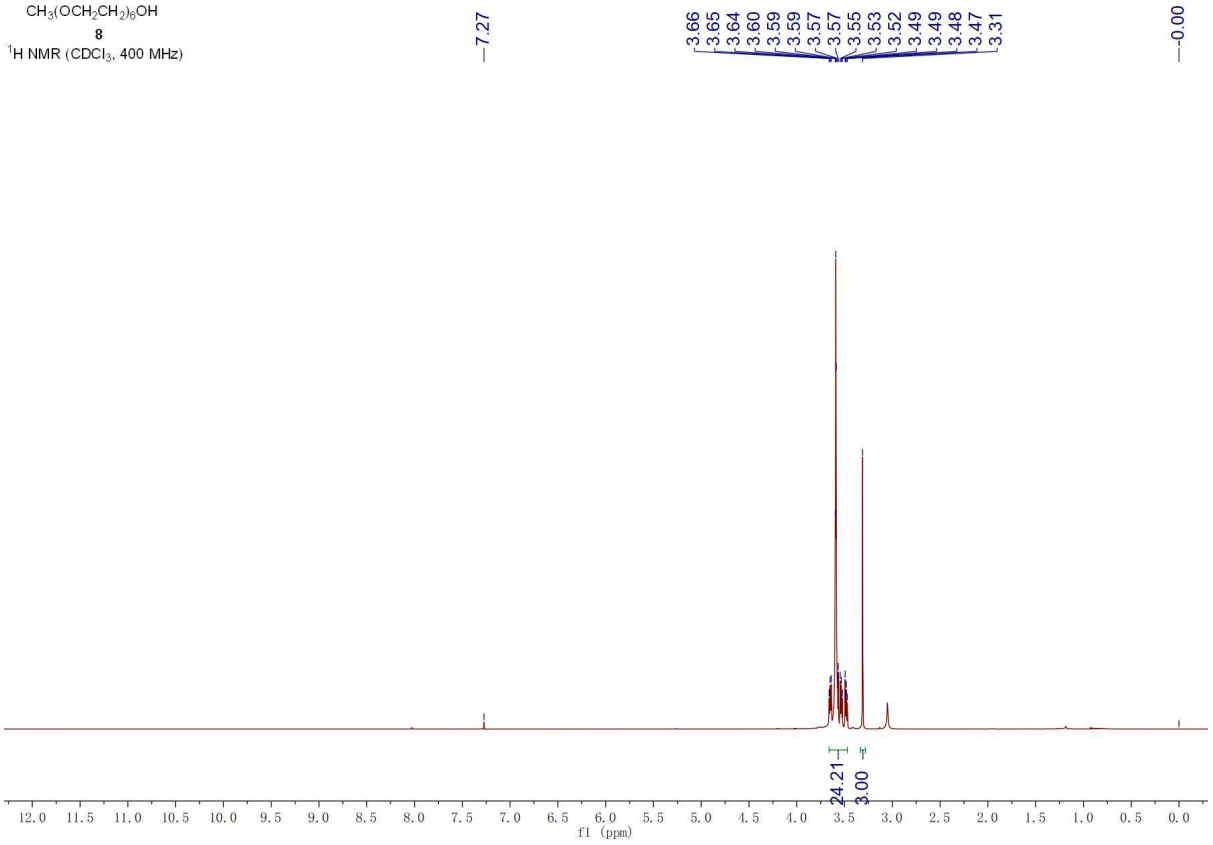
^{13}C NMR ($CDCl_3$, 101 MHz)



$CH_3(OCH_2CH_2)_6OH$

8

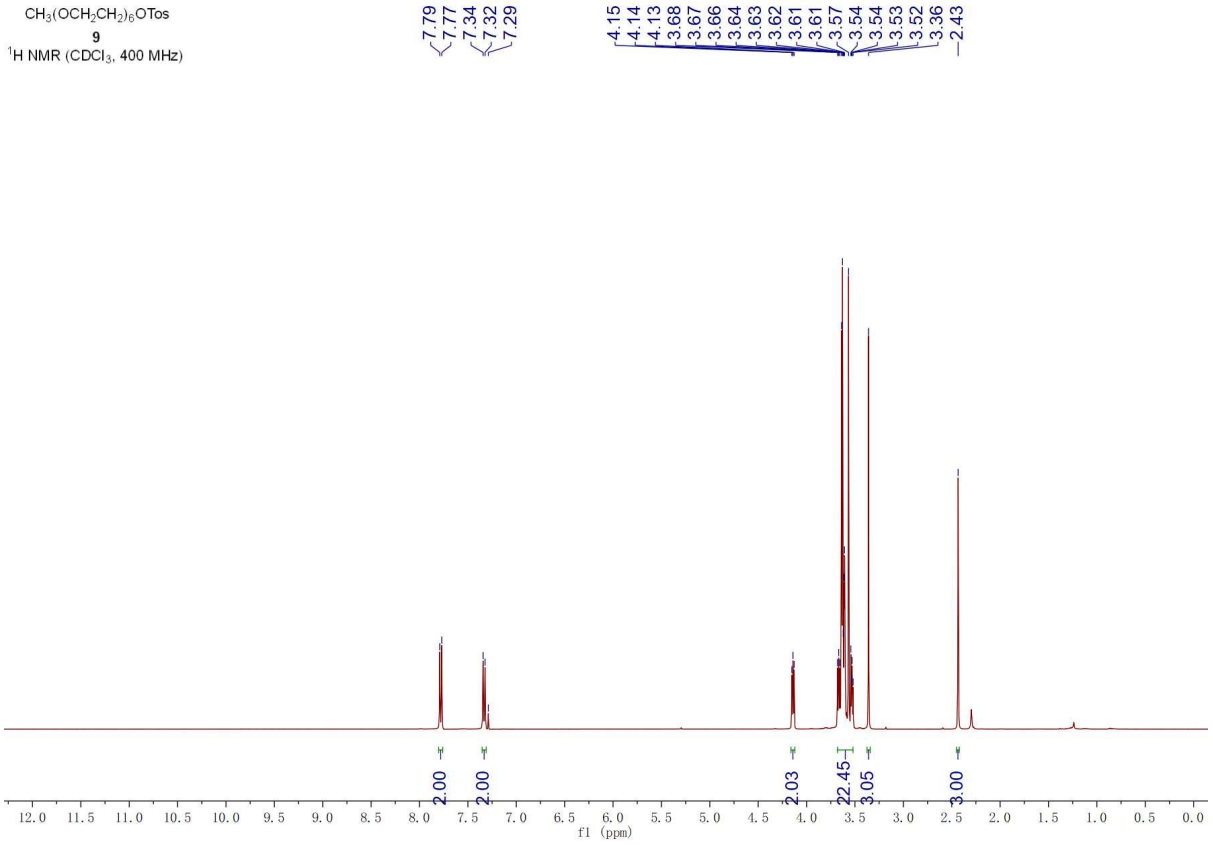
^{1}H NMR ($CDCl_3$, 400 MHz)



$CH_3(OCH_2CH_2)_6OTos$

9

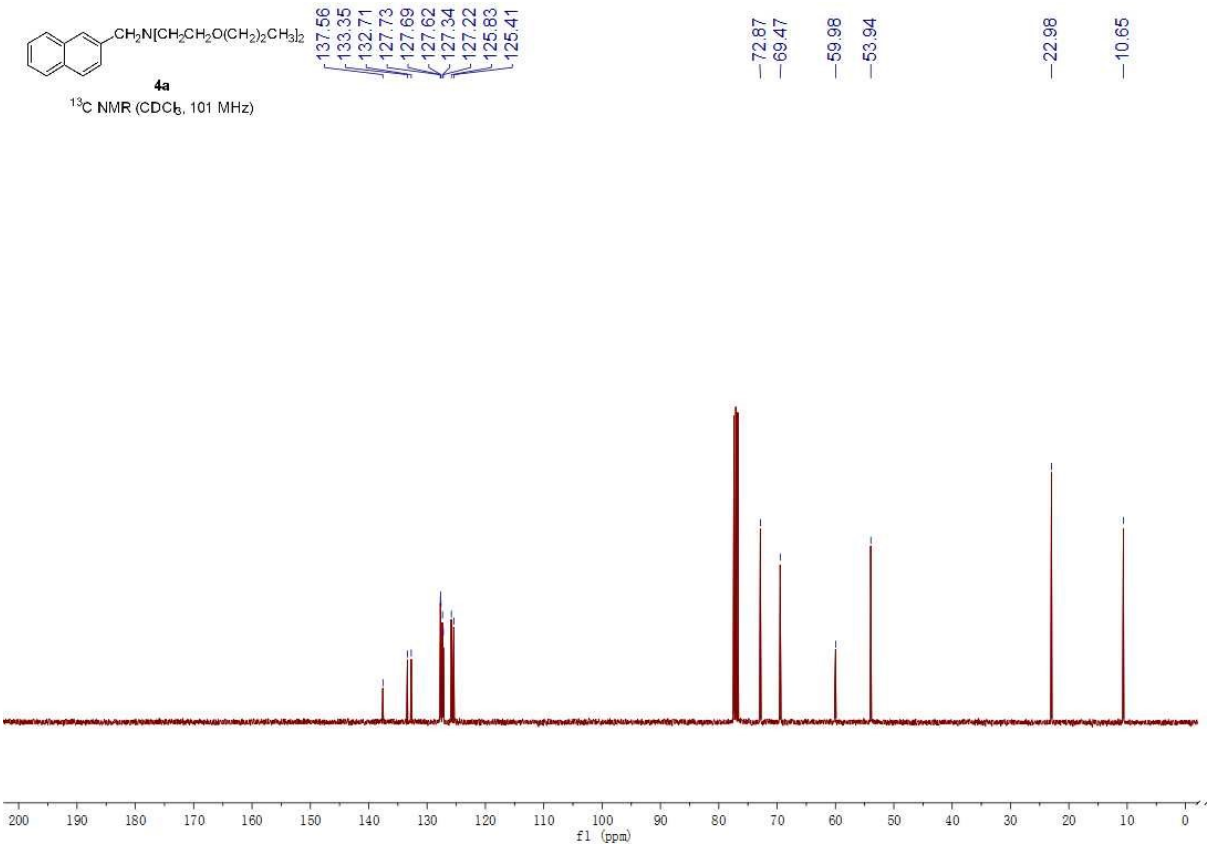
^1H NMR ($CDCl_3$, 400 MHz)

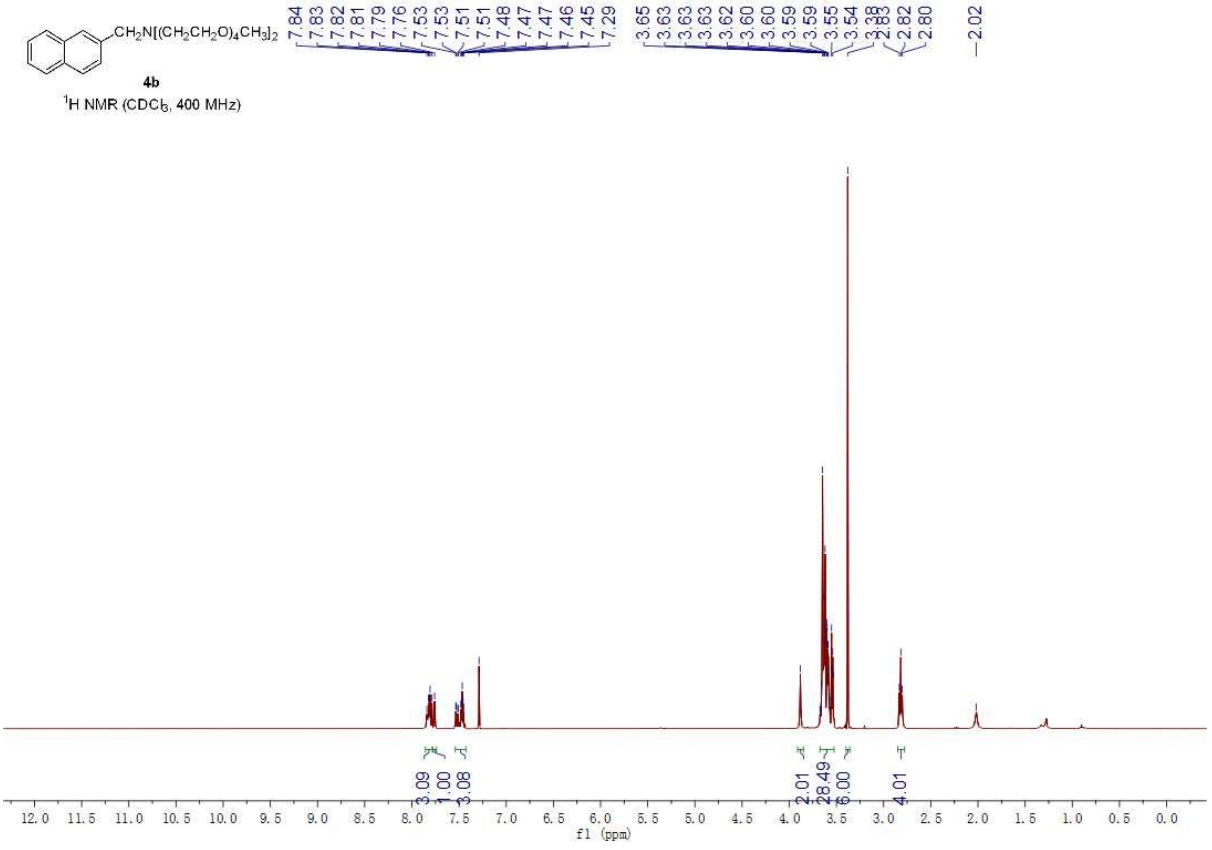


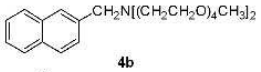
$CH_2N[CH_2CH_2O(CH_2)_2CH_3]_2$

4a

^1H NMR ($CDCl_3$, 400 MHz)


CH2N[CH2CH2O(CH2)2CH3]2
4a
13C NMR (CDCl3, 101 MHz)
137.56
133.35
132.71
127.73
127.69
127.62
127.34
127.22
125.83
125.41
72.87
69.47
59.98
53.94
22.98
10.65
f1 (ppm)


CH2N[(CH2CH2O)4CH3]2
4b
1H NMR (CDCl3, 400 MHz)
7.84
7.83
7.82
7.81
7.79
7.76
7.53
7.53
7.51
7.51
7.48
7.47
7.47
7.46
7.45
7.29
3.65
3.63
3.63
3.62
3.60
3.59
3.59
3.55
3.54
2.83
2.82
2.80
2.02
3.09
1.00
3.08
2.01
28.49
6.00
4.01
f1 (ppm)

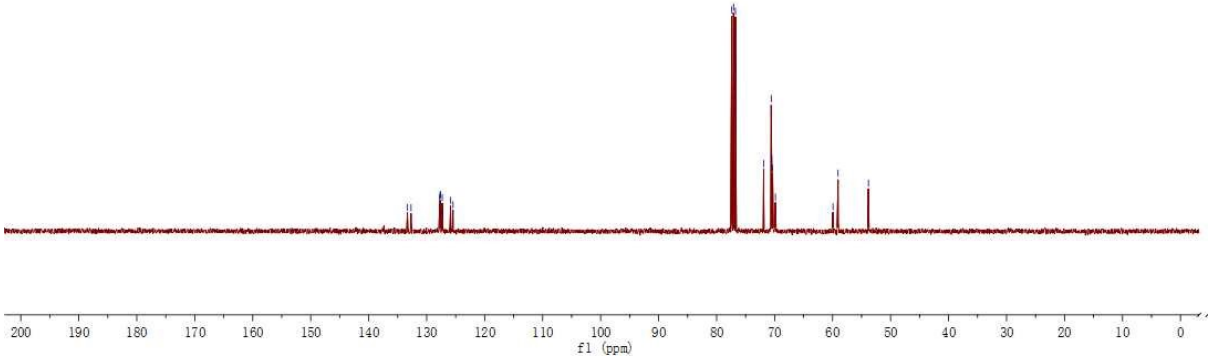


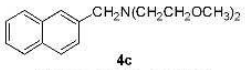
4b

^{13}C NMR (CDCl$_3$, 101 MHz)

133.30
132.69
127.76
127.68
127.61
127.31
127.24
125.86
125.45

77.39
77.07
76.75
71.89
70.58
70.49
70.34
69.85
59.93
59.05
53.82

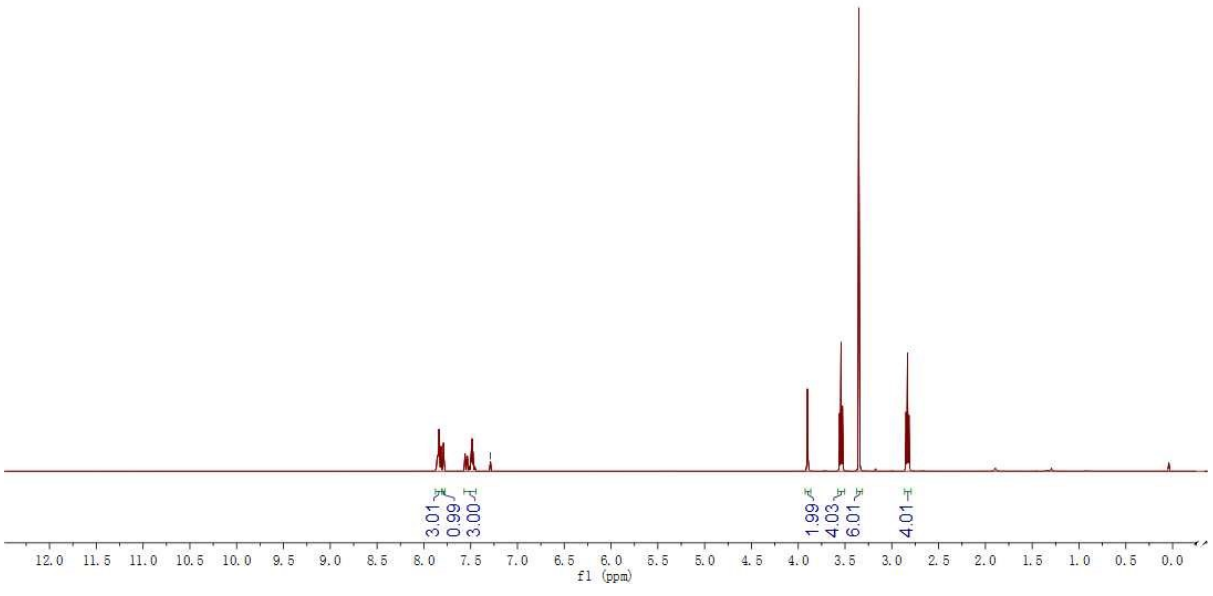


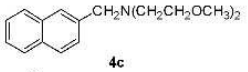


4c

^{1}H NMR (CDCl$_3$, 400 MHz)

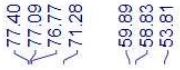
7.29

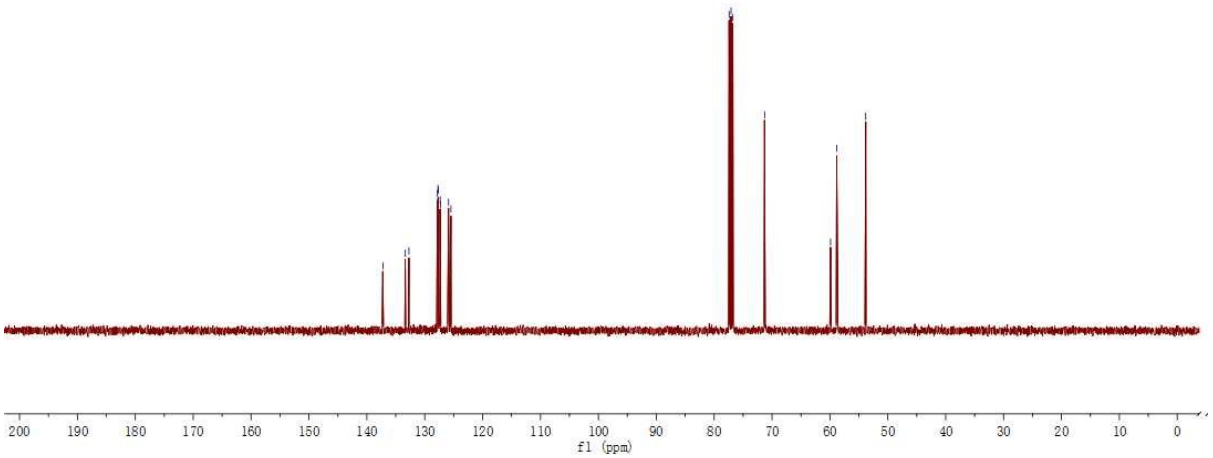


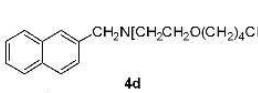


4c

^{13}C NMR ($CDCl_3$, 101 MHz)

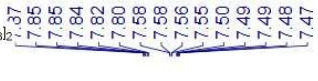


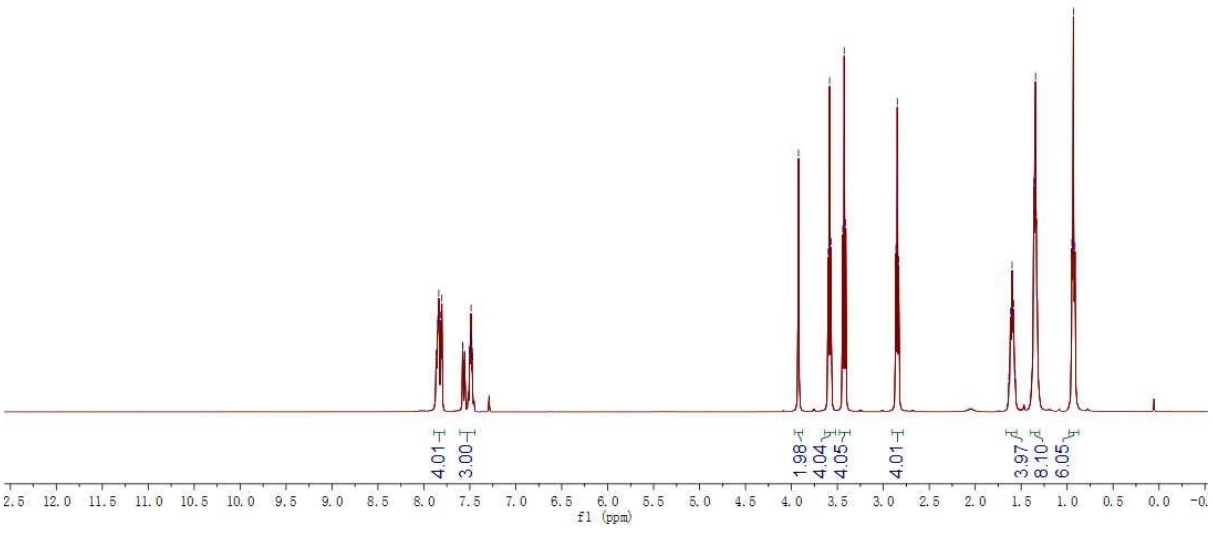


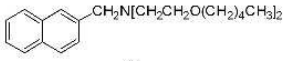


4d

^{1}H NMR ($CDCl_3$, 400 MHz)







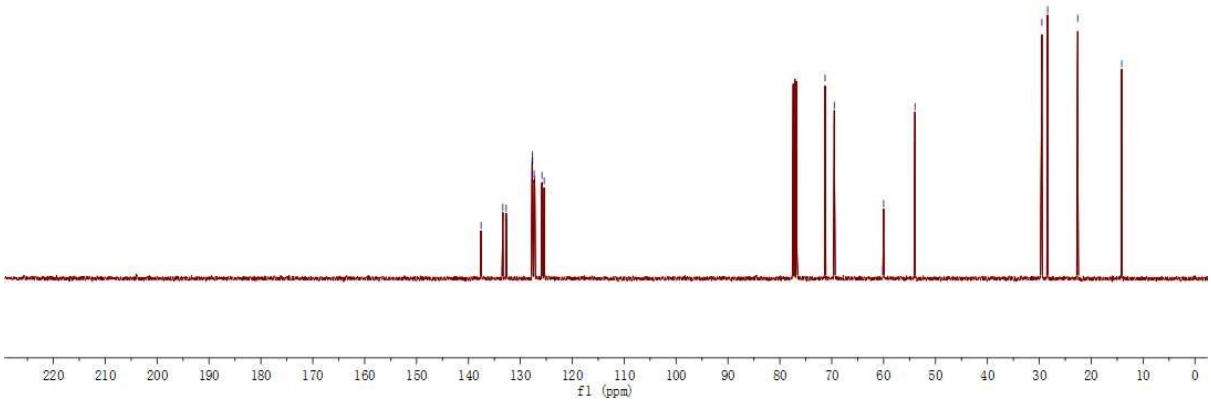
4d

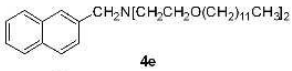
^{13}C NMR ($CDCl_3$, 101 MHz)

137.58
133.37
132.73
127.74
127.70
127.63
127.35
127.22
125.83
125.41

71.29
69.51
60.00
53.96

29.51
28.40
22.59
14.12



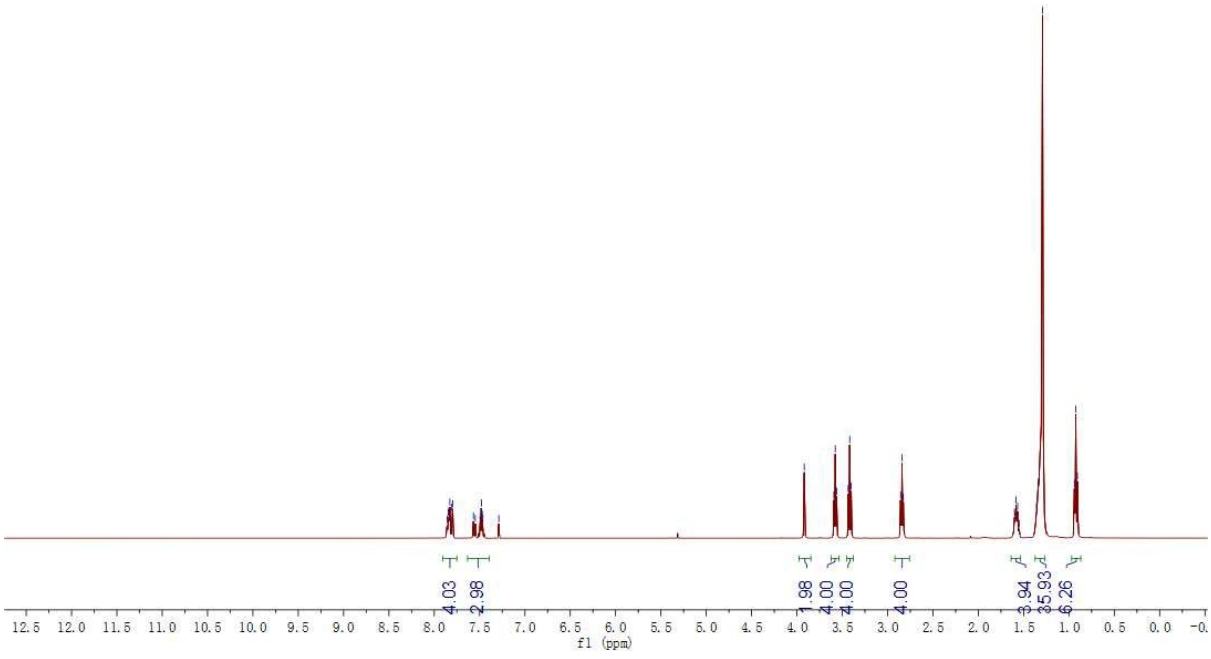


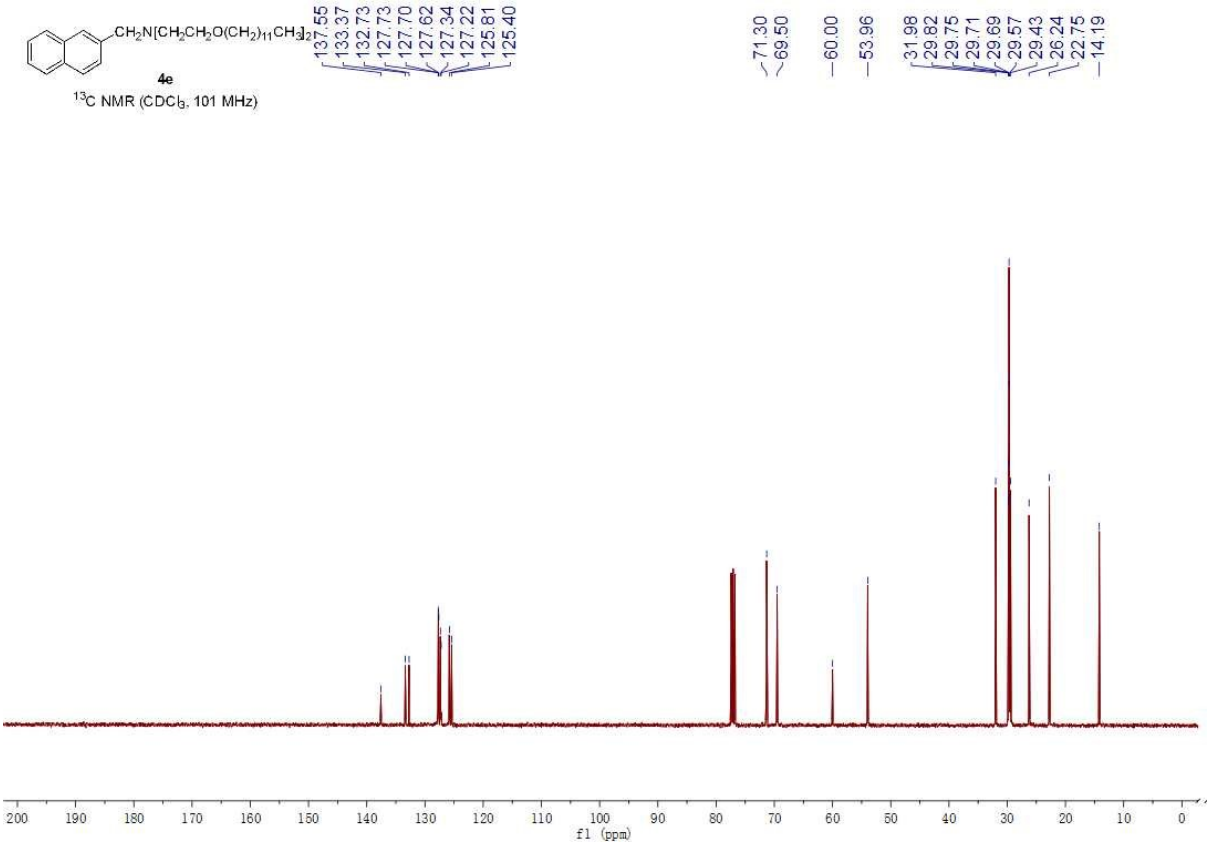
4e

^{1}H NMR ($CDCl_3$, 400 MHz)

7.86
7.84
7.84
7.83
7.81
7.80
7.57
7.55
7.49
7.49
7.48
7.47
7.46
7.29

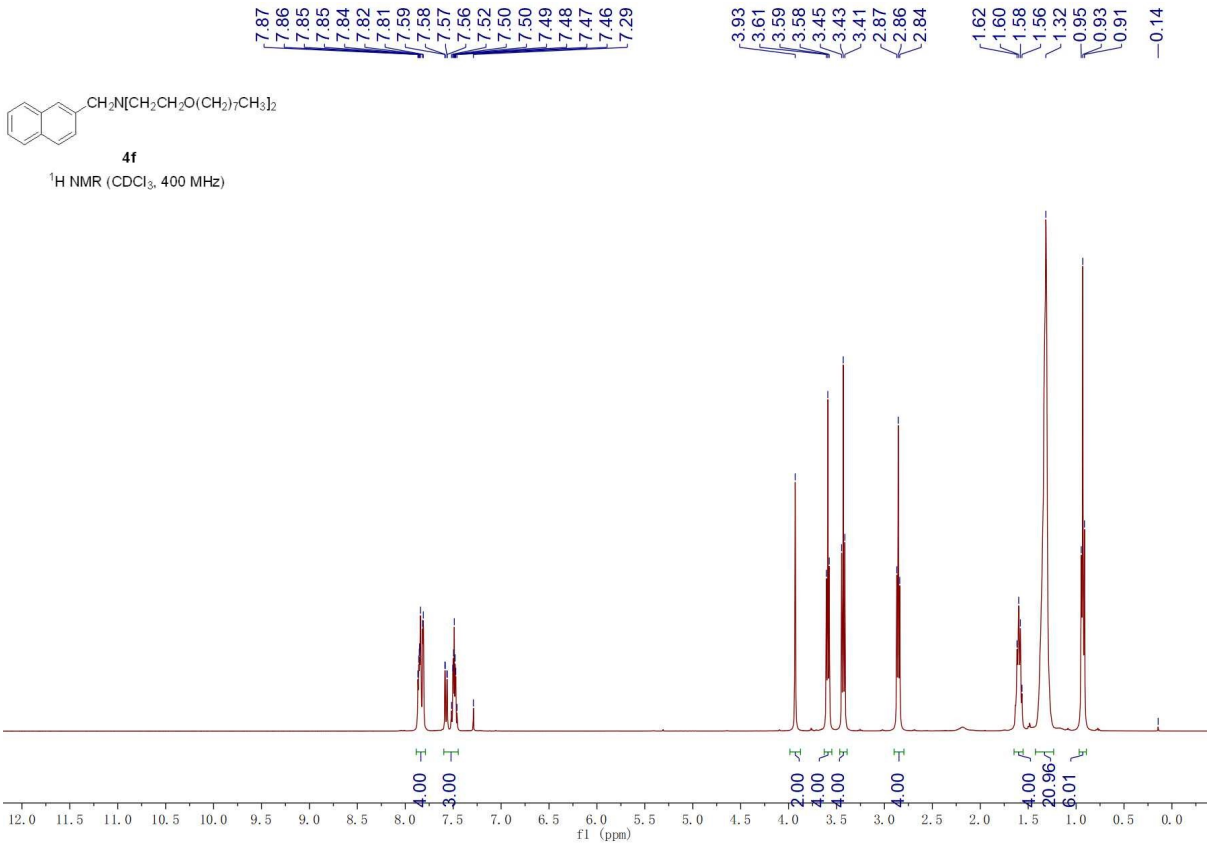
3.92
3.59
3.58
3.56
3.44
3.42
3.40
2.86
2.84
2.83
1.60
1.58
1.57
1.55
1.36
1.34
1.29
0.94
0.93
0.91





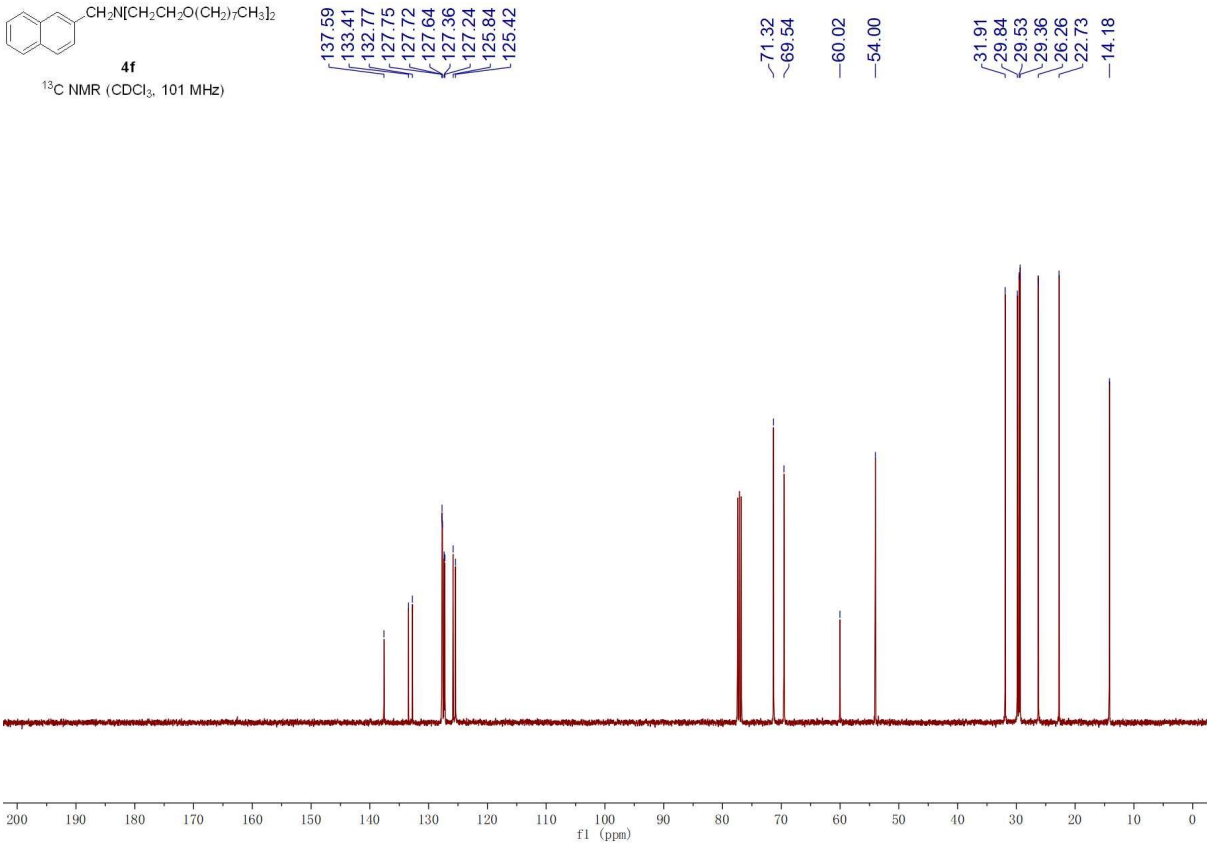
4e

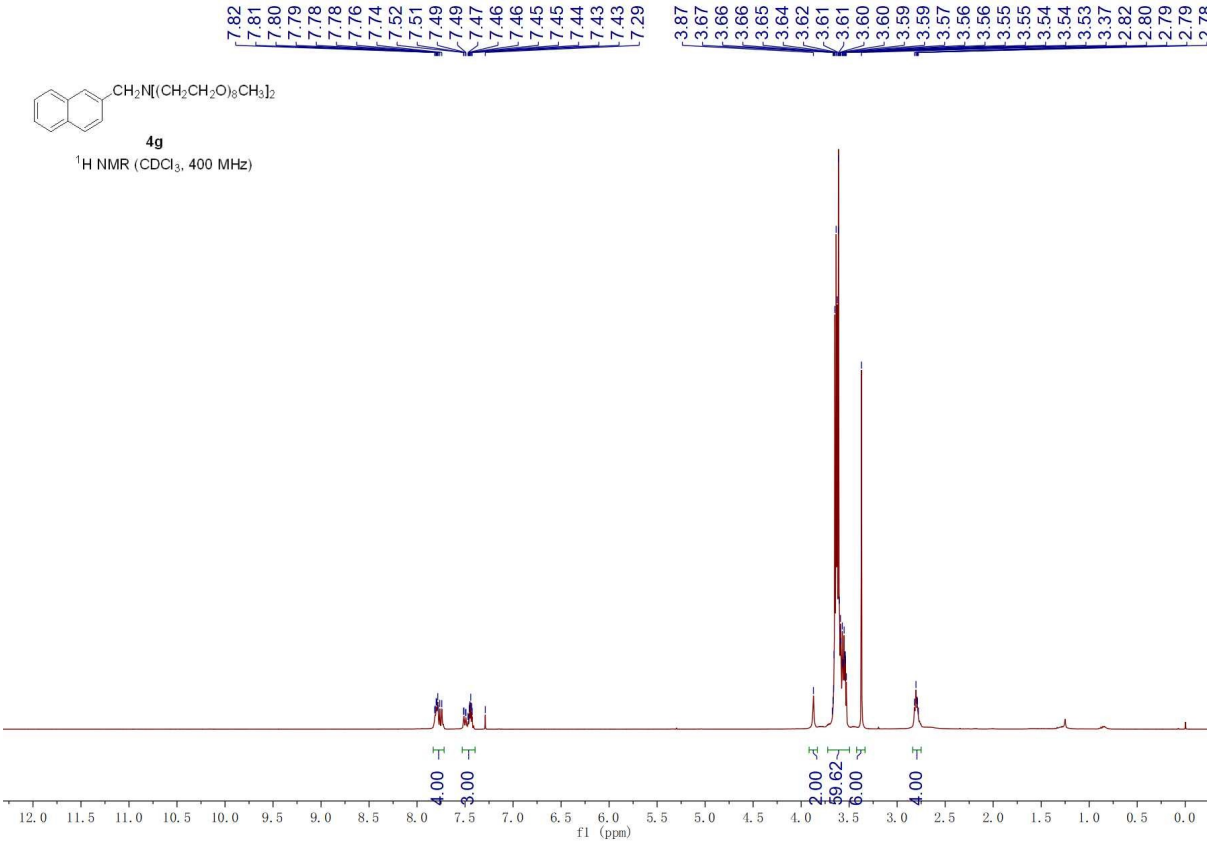
^{13}C NMR ($CDCl_3$, 101 MHz)

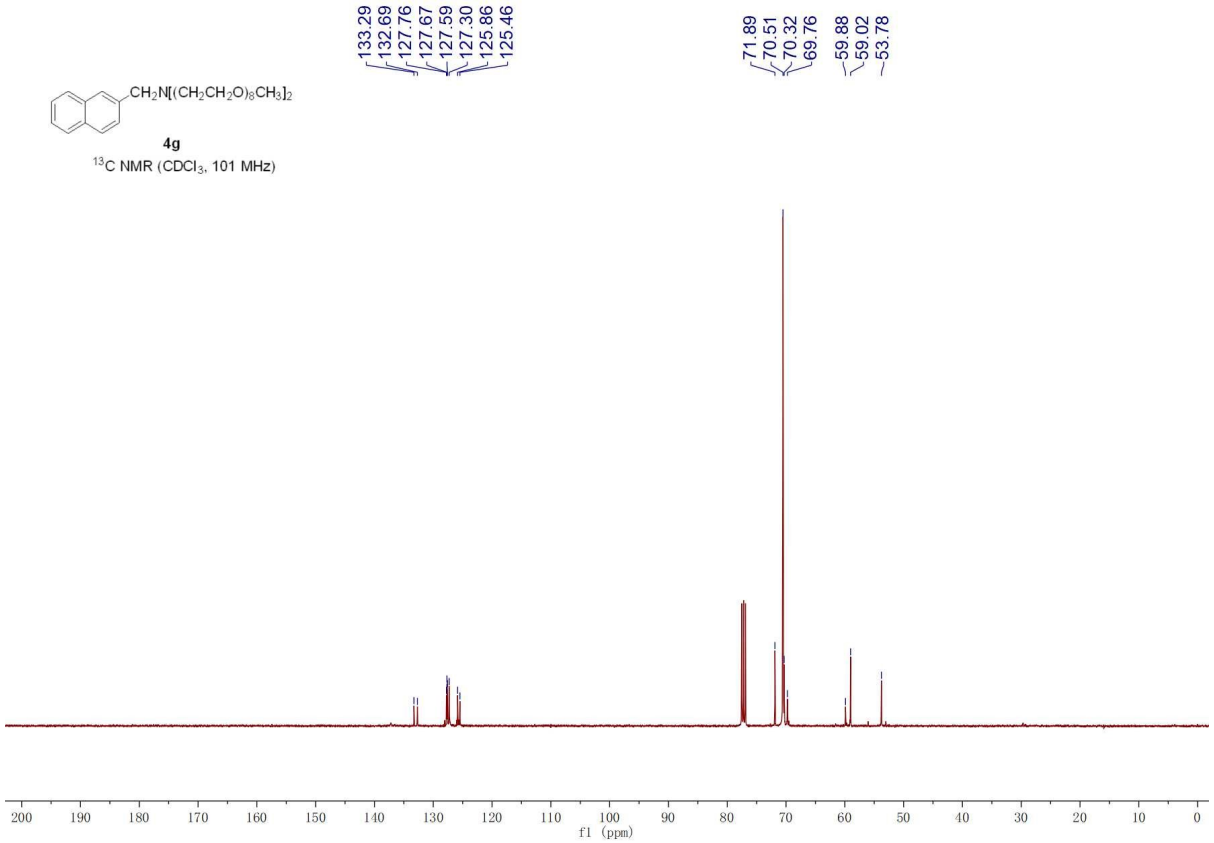


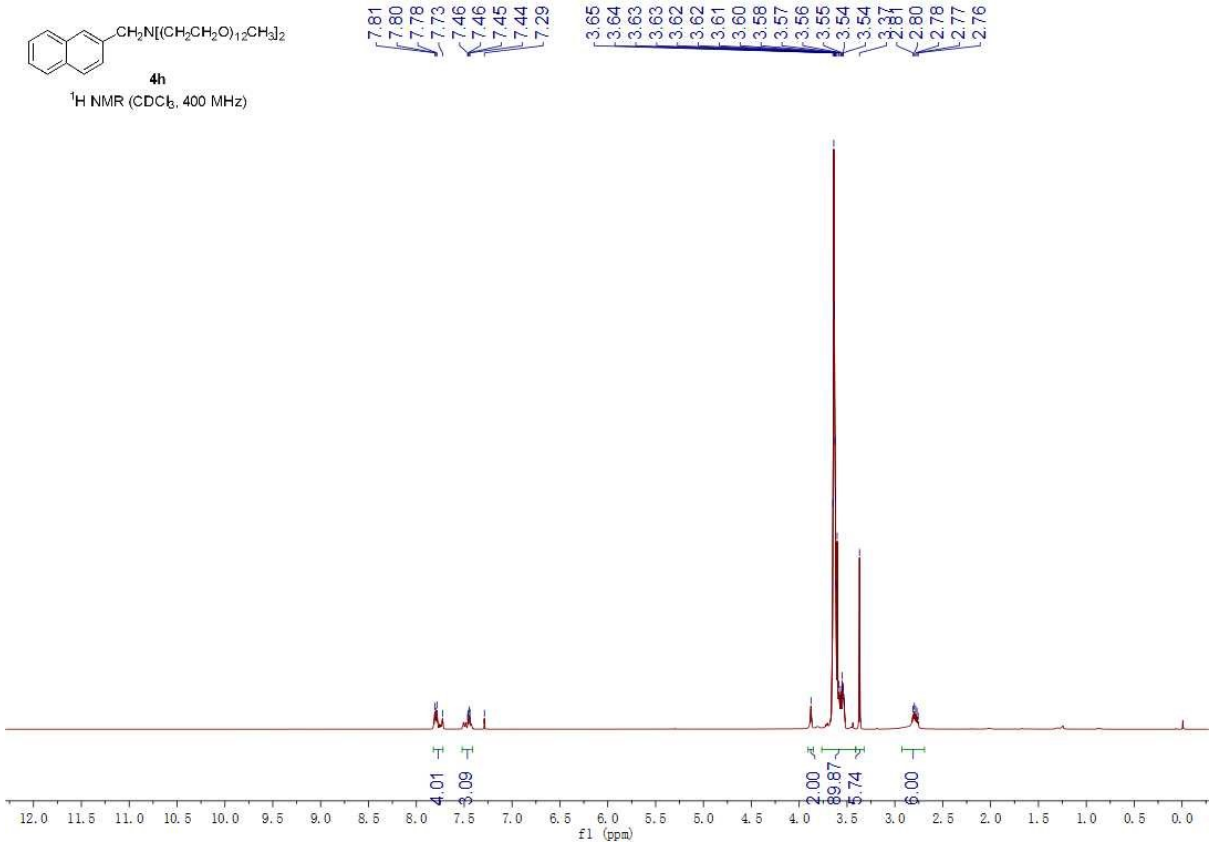
4f

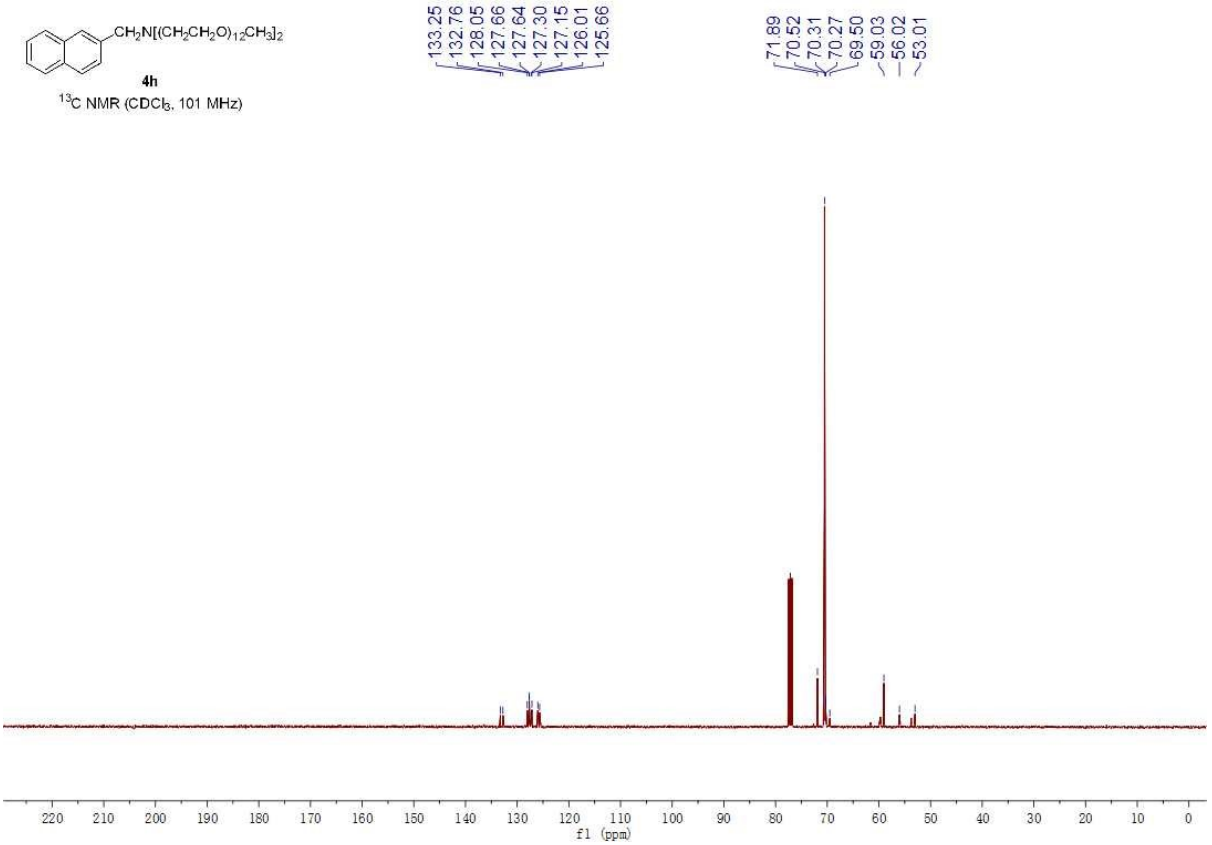
^{1}H NMR ($CDCl_3$, 400 MHz)

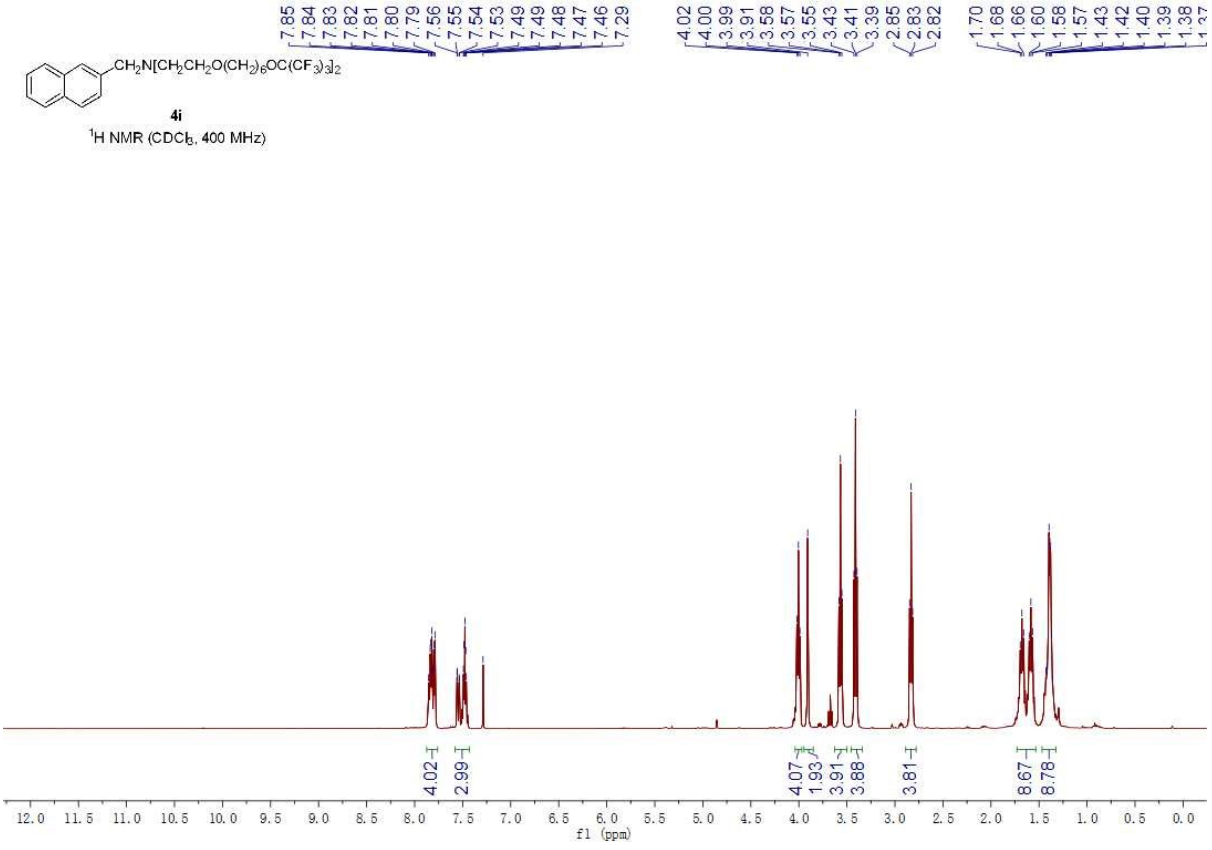

CH2N[CH2CH2O(CH2)7CH3]2
4f
13C NMR (CDCl3, 101 MHz)
137.59
133.41
132.77
127.75
127.72
127.64
127.36
127.24
125.84
125.42
71.32
69.54
60.02
54.00
31.91
29.84
29.53
29.36
26.26
22.73
14.18
f1 (ppm)

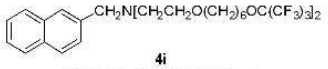

CH2N[(CH2CH2O)8CH3]2
4g
1H NMR (CDCl3, 400 MHz)
7.82
7.81
7.80
7.79
7.78
7.78
7.76
7.74
7.52
7.51
7.49
7.49
7.47
7.46
7.46
7.45
7.45
7.44
7.43
7.43
7.29
3.87
3.67
3.66
3.65
3.64
3.62
3.61
3.61
3.60
3.60
3.59
3.59
3.57
3.56
3.56
3.55
3.55
3.54
3.54
3.53
3.37
2.82
2.80
2.79
4.00
3.00
2.00
59.62
6.00
4.00
f1 (ppm)


133.29
132.69
127.76
127.67
127.59
127.30
125.86
125.46
71.89
70.51
70.32
69.76
59.88
59.02
53.78
CH2N[(CH2CH2O)8CH3]2
4g
13C NMR (CDCl3, 101 MHz)
f1 (ppm)


7.81
7.80
7.78
7.73
7.46
7.46
7.45
7.44
7.29
3.65
3.64
3.63
3.63
3.62
3.62
3.61
3.60
3.58
3.57
3.56
3.55
3.54
3.54
3.37
2.81
2.80
2.78
2.77
2.76
CH2N[(CH2CH2O)12CH3]2
4h
1H NMR (CDCl3, 400 MHz)
4.01
3.09
2.00
9.87
5.74
6.00
f1 (ppm)

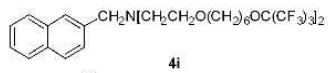

CH2N[(CH2CH2O)12CH3]2
4h
13C NMR (CDCl3, 101 MHz)
133.25
132.76
128.05
127.66
127.64
127.30
127.15
126.01
125.66
71.89
70.52
70.31
70.27
69.50
59.03
56.02
53.01
f1 (ppm)


CH2N[CH2CH2O(CH2)6OC(CF3)3]2
4i
1H NMR (CDCl3, 400 MHz)
7.85
7.84
7.83
7.82
7.81
7.80
7.79
7.56
7.55
7.54
7.53
7.49
7.49
7.48
7.47
7.46
7.29
4.02
4.00
3.99
3.91
3.58
3.57
3.55
3.43
3.41
3.39
2.85
2.83
2.82
1.70
1.68
1.66
1.60
1.58
1.57
1.43
1.42
1.40
1.39
1.38
4.02
2.99
4.07
1.93
3.91
3.88
3.81
8.67
8.78
f1 (ppm)



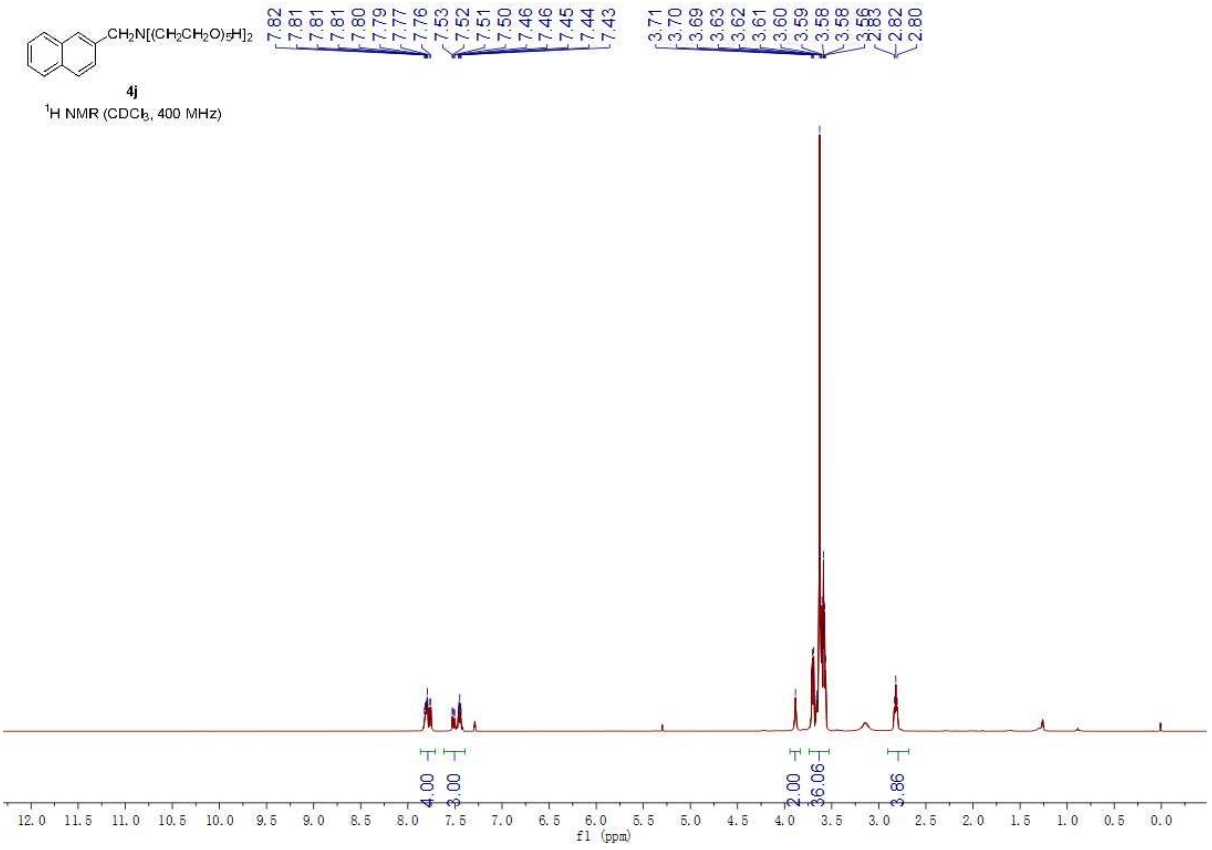
4i

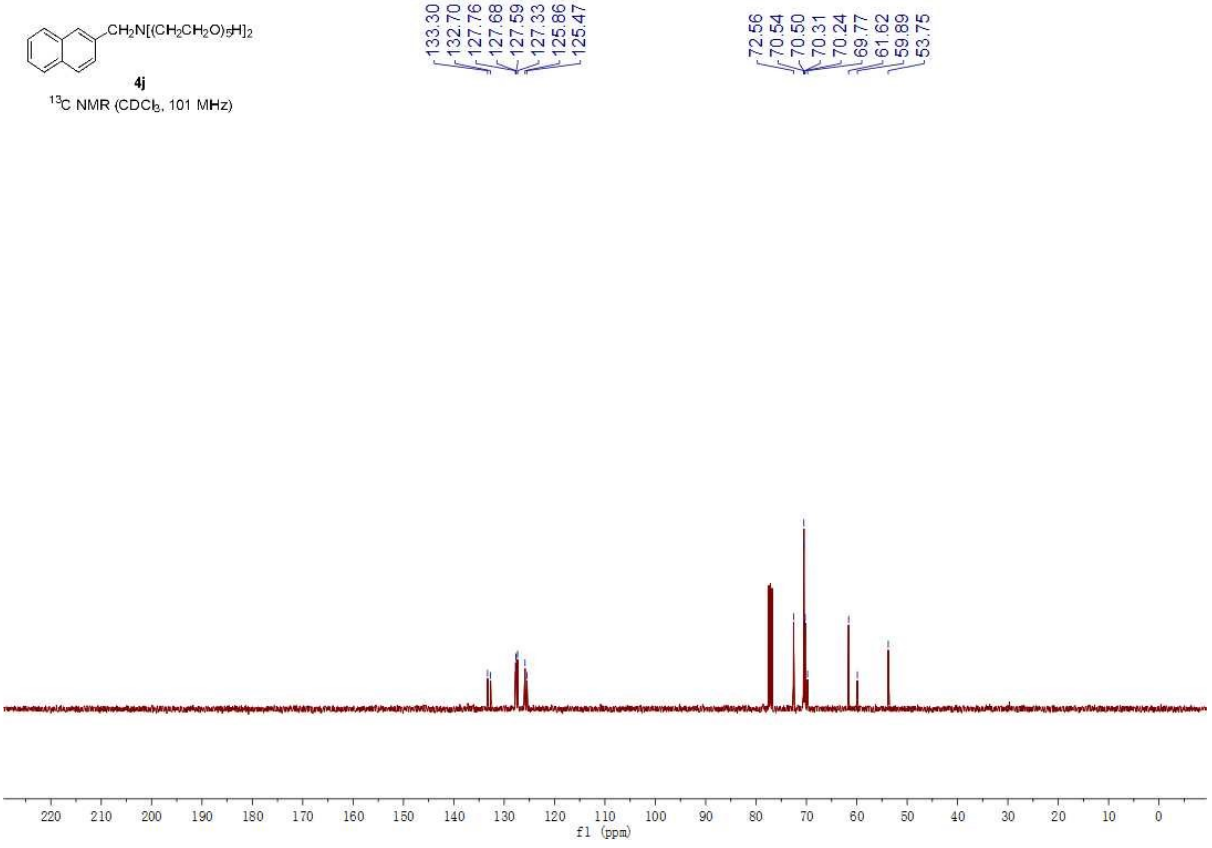
^{19}F NMR (CDCl3, 376 MHz)

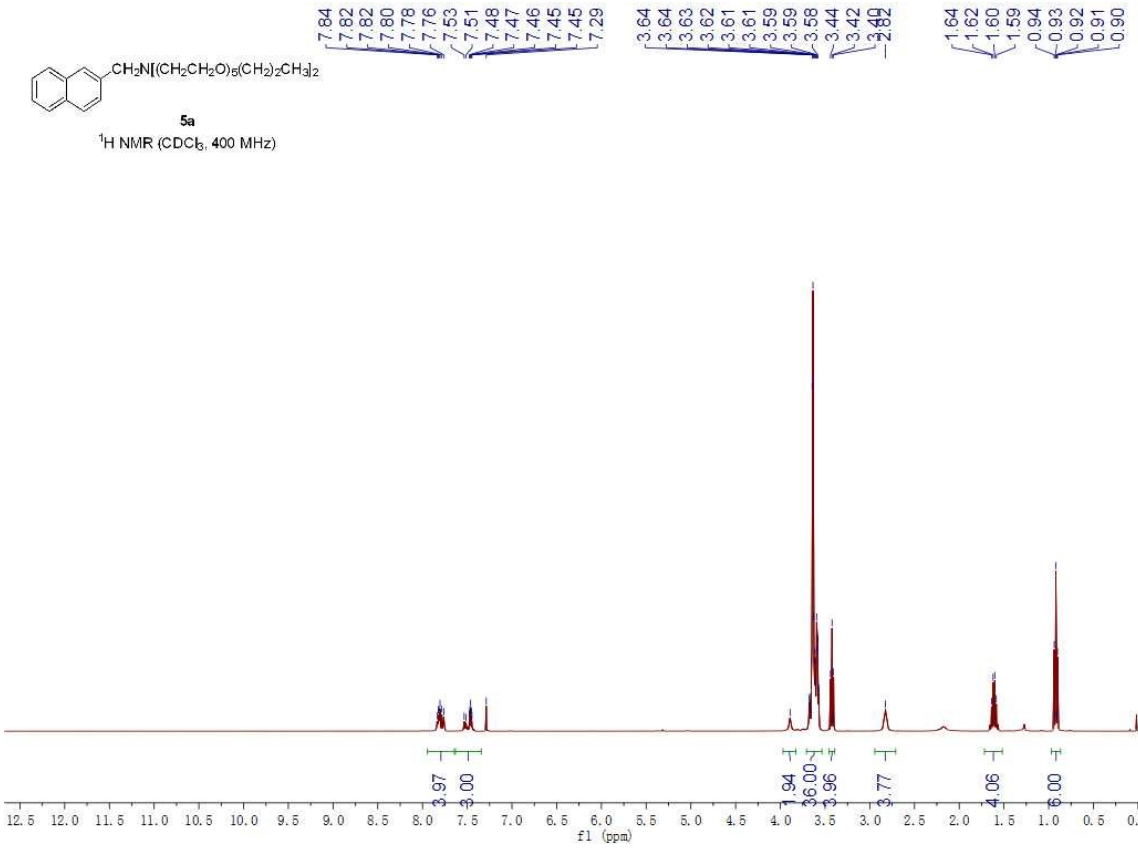


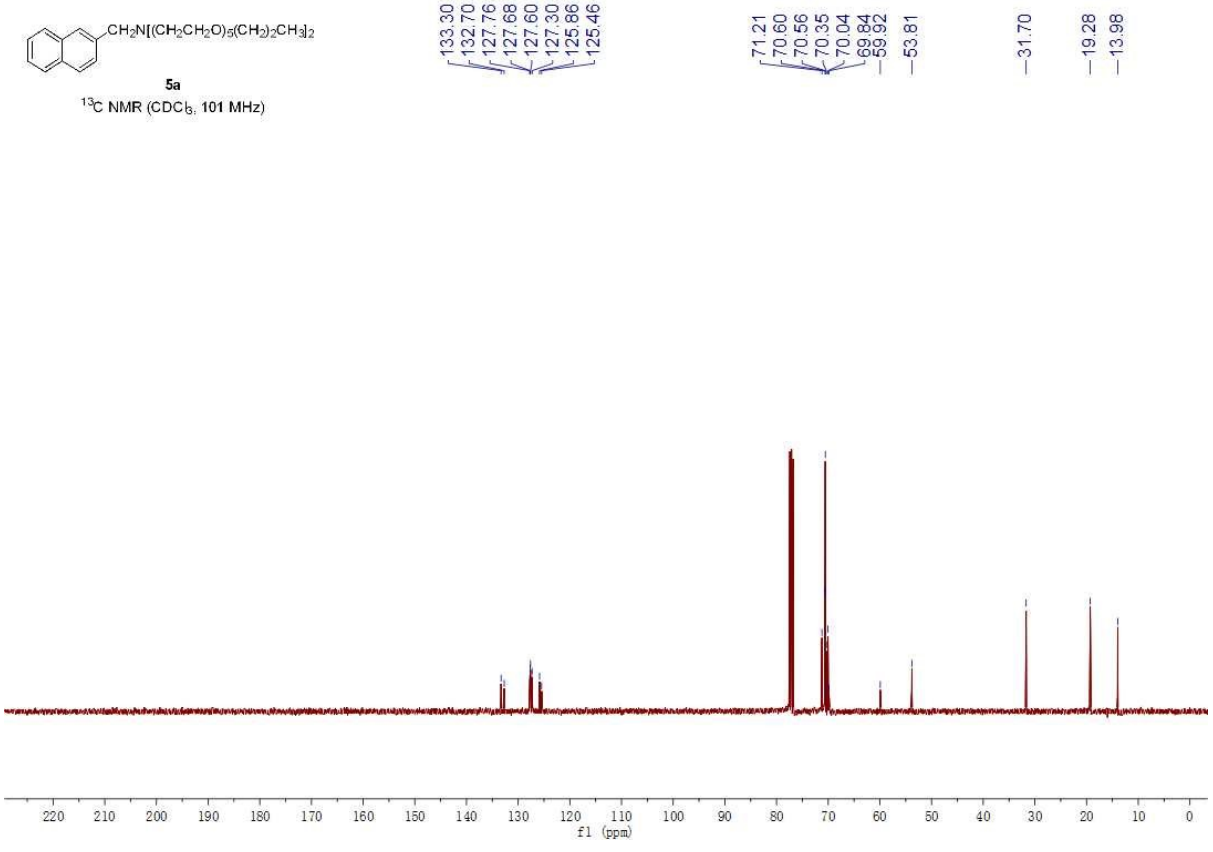
4i

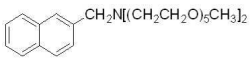
^{13}C NMR (CDCl$_3$, 101 MHz)


CH2N[(CH2CH2O)5H]2
4j
1H NMR (CDCl3, 400 MHz)
7.82
7.81
7.81
7.81
7.80
7.79
7.77
7.76
7.53
7.52
7.51
7.50
7.46
7.45
7.44
7.43
3.71
3.70
3.69
3.63
3.62
3.61
3.60
3.59
3.58
3.58
2.83
2.82
2.80
4.00
3.00
2.00
36.06
3.86
f1 (ppm)


CH2N[(CH2CH2O)5H]2
4j
13C NMR (CDCl3, 101 MHz)
133.30
132.70
127.76
127.68
127.59
127.33
125.86
125.47
72.56
70.54
70.50
70.31
70.24
69.77
61.62
59.89
53.75
f1 (ppm)

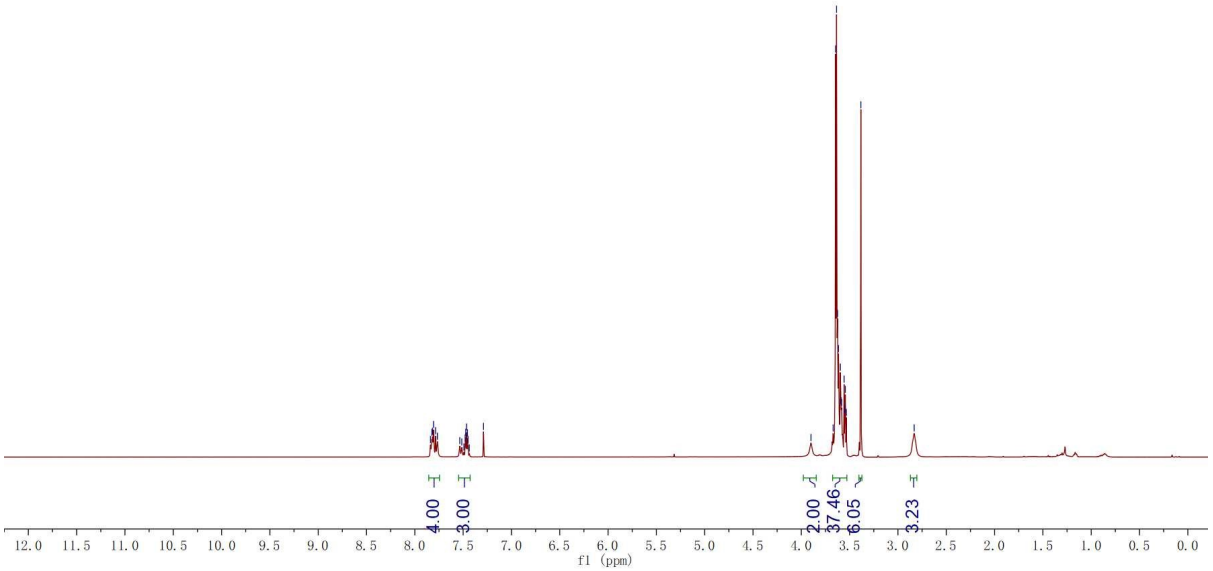

7.84
7.82
7.82
7.80
7.78
7.76
7.53
7.51
7.48
7.47
7.46
7.45
7.45
7.29
3.64
3.64
3.63
3.62
3.61
3.61
3.59
3.59
3.58
3.44
3.42
1.64
1.62
1.60
1.59
0.94
0.93
0.92
0.91
0.90
CH2N[(CH2CH2O)5(CH2)2CH3]2
5a
1H NMR (CDCl3, 400 MHz)
3.97
3.00
1.94
6.00
3.96
3.77
4.06
6.00
f1 (ppm)

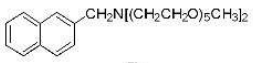

133.30
132.70
127.76
127.68
127.60
127.30
125.86
125.46
71.21
70.60
70.56
70.35
70.04
69.84
59.92
53.81
31.70
19.28
13.98
CH2N[(CH2CH2O)5(CH2)2CH3]2
5a
13C NMR (CDCl3, 101 MHz)
f1 (ppm)



5b

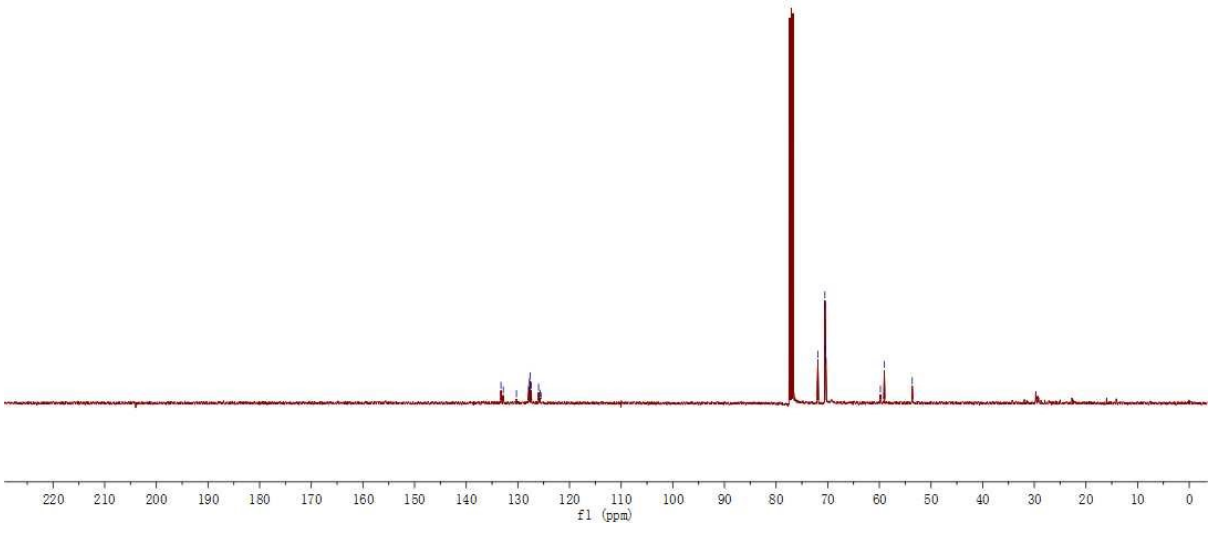
^{1}H NMR ($CDCl_3$, 400 MHz)

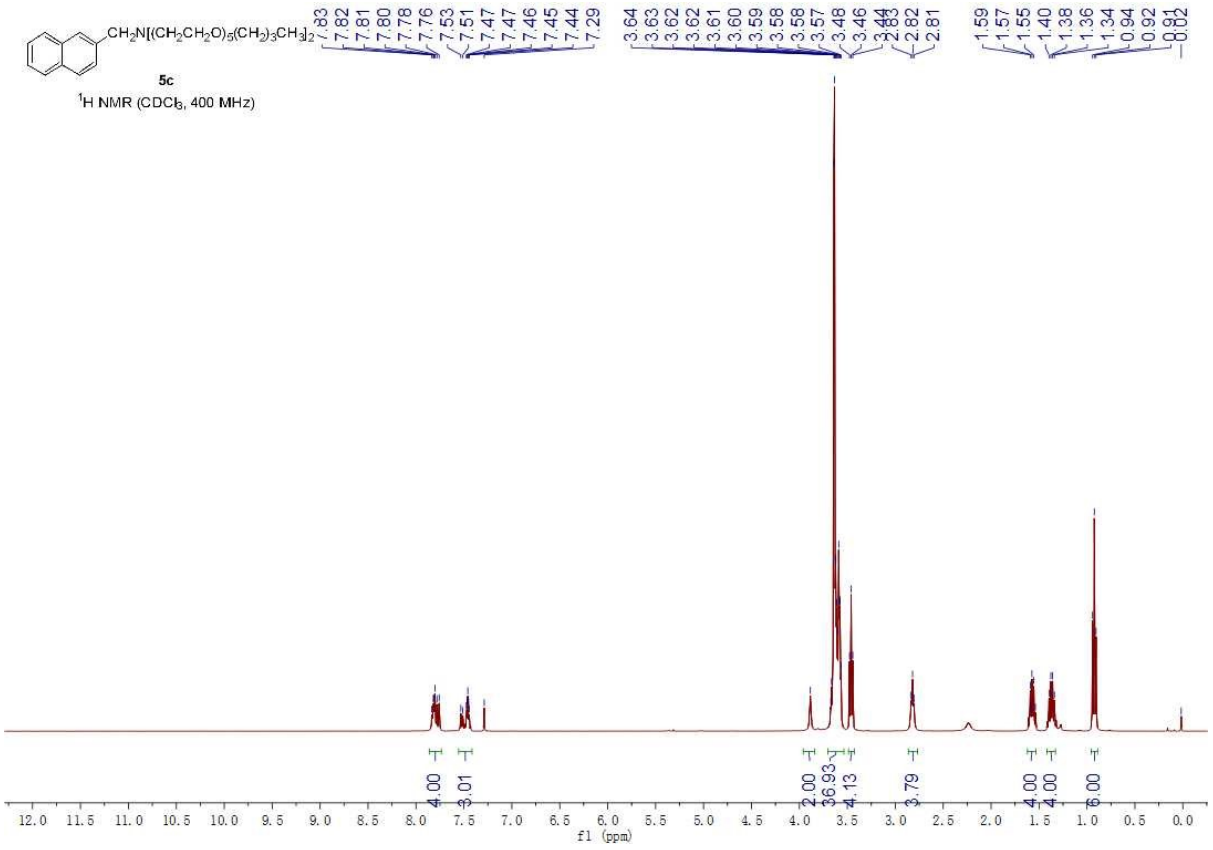


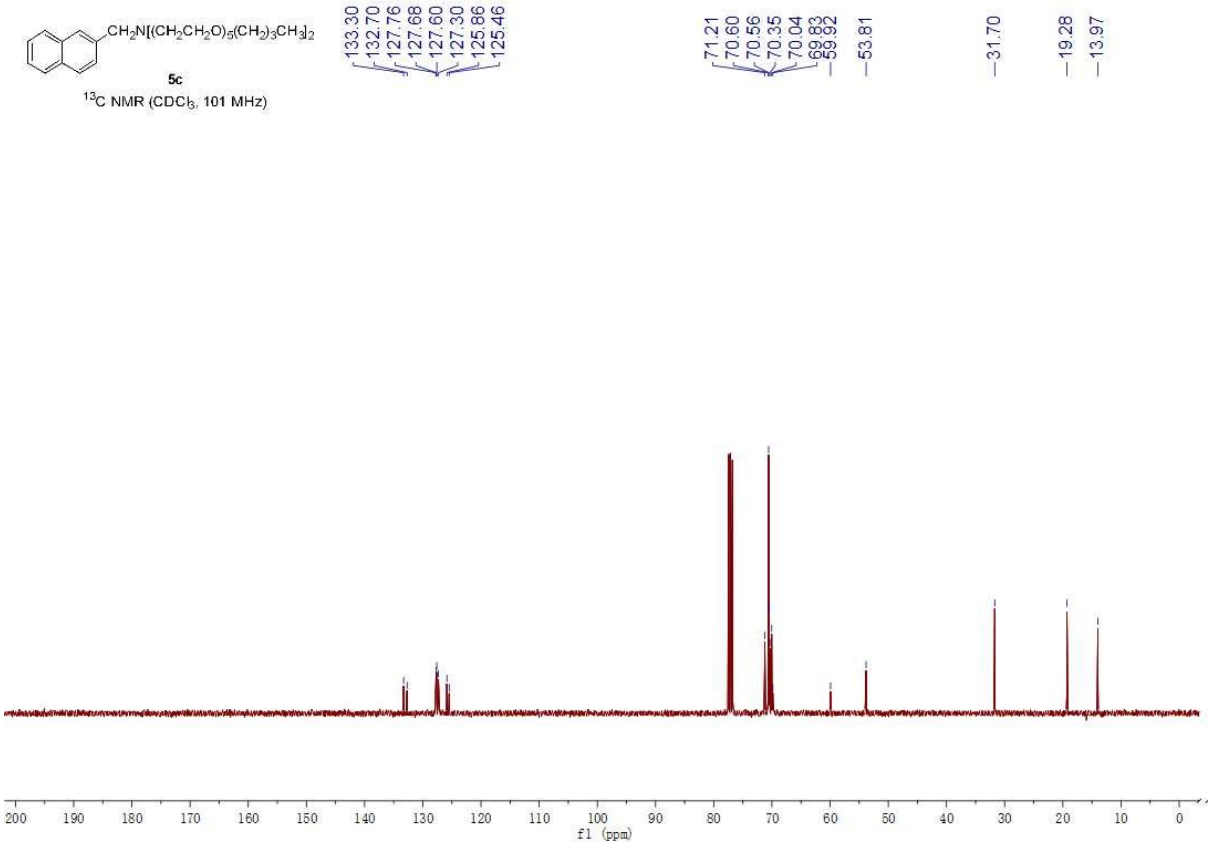


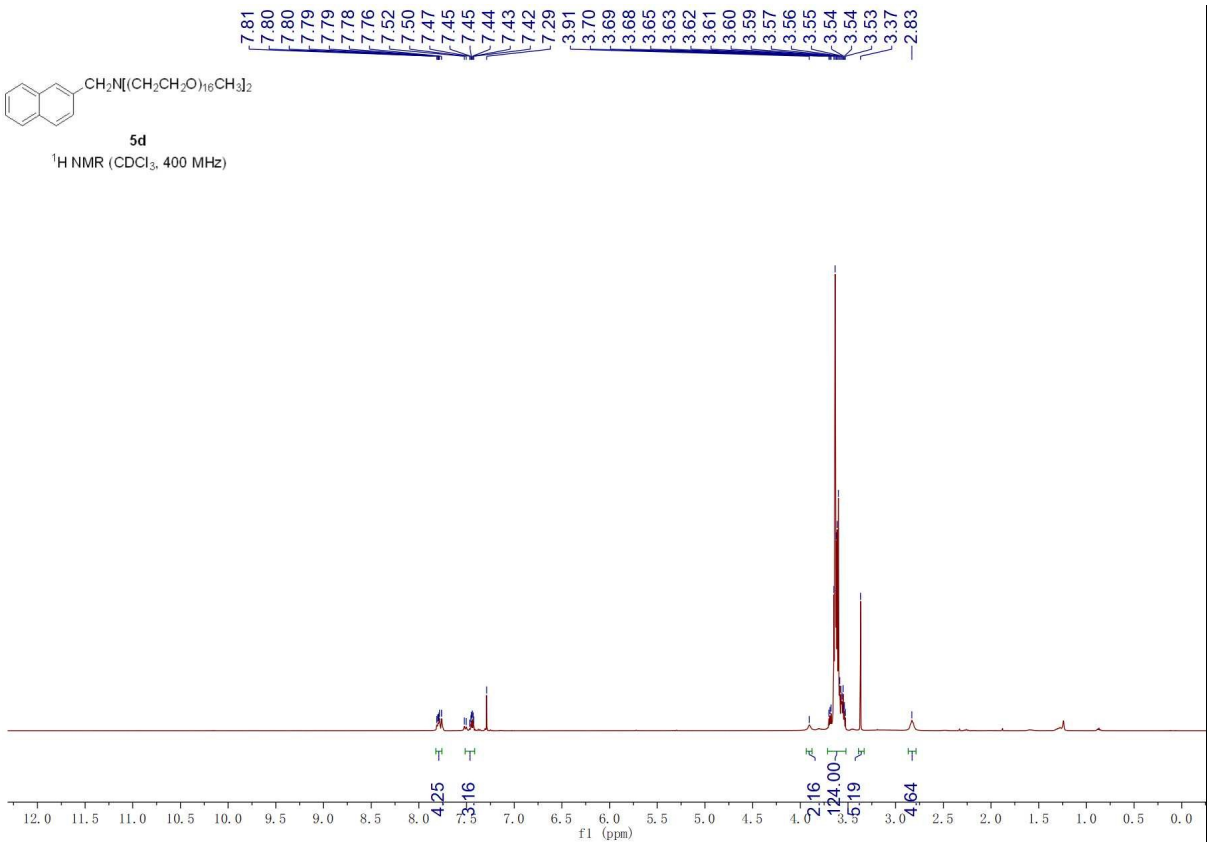
5b

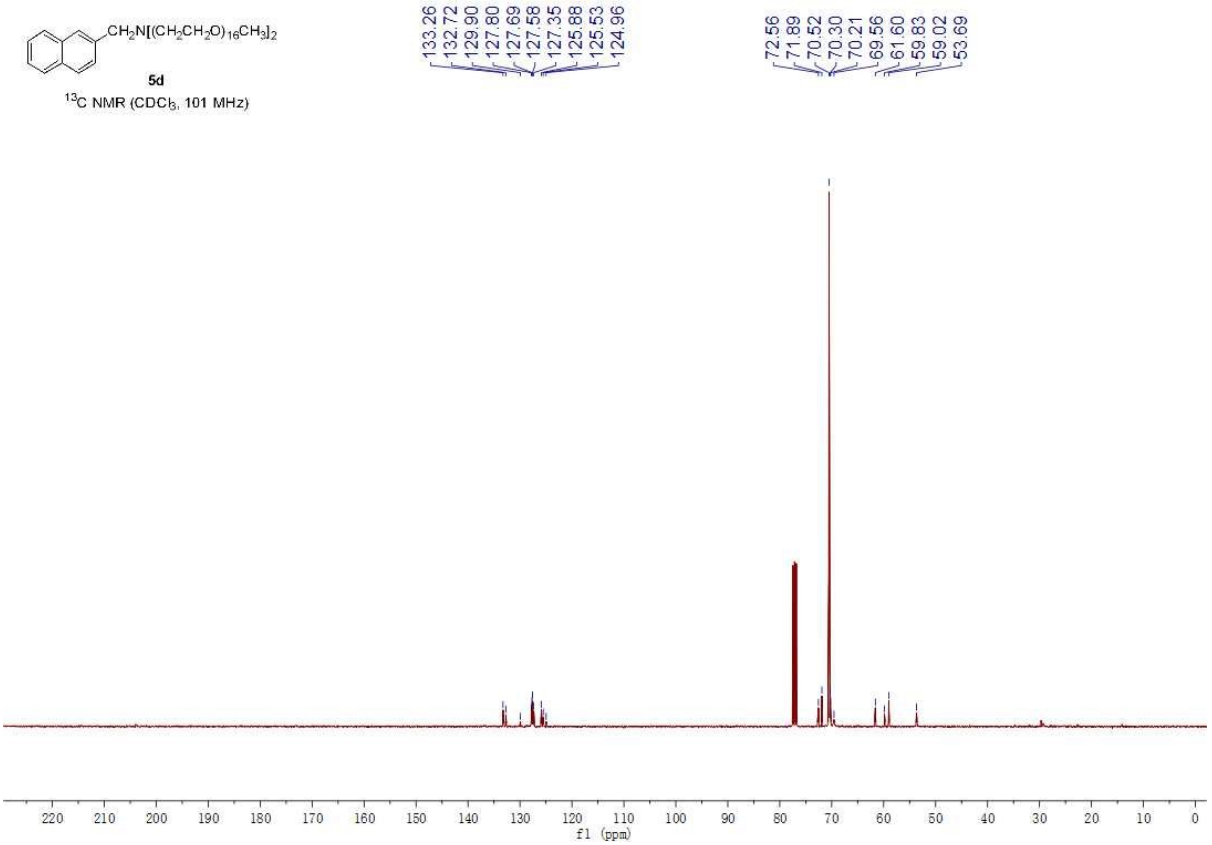
^{13}C NMR ($CDCl_3$, 101 MHz)

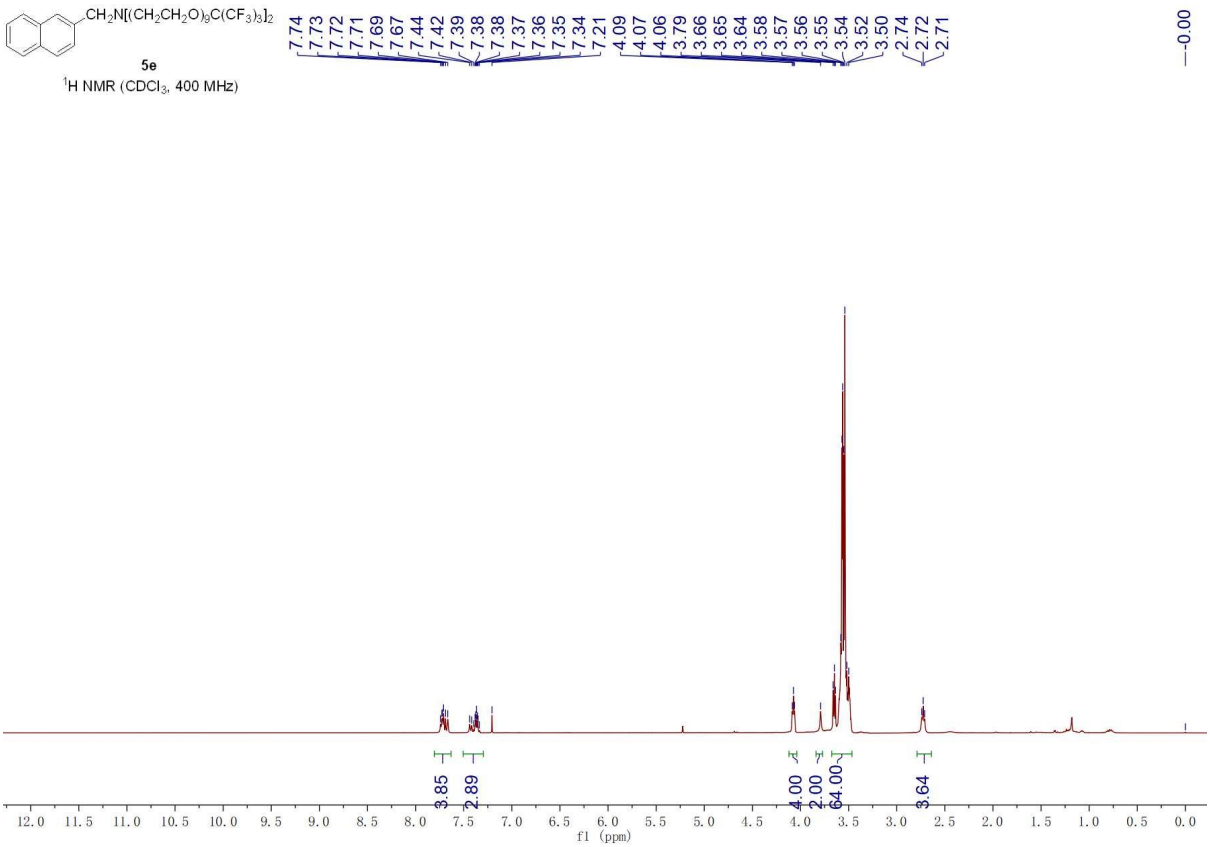


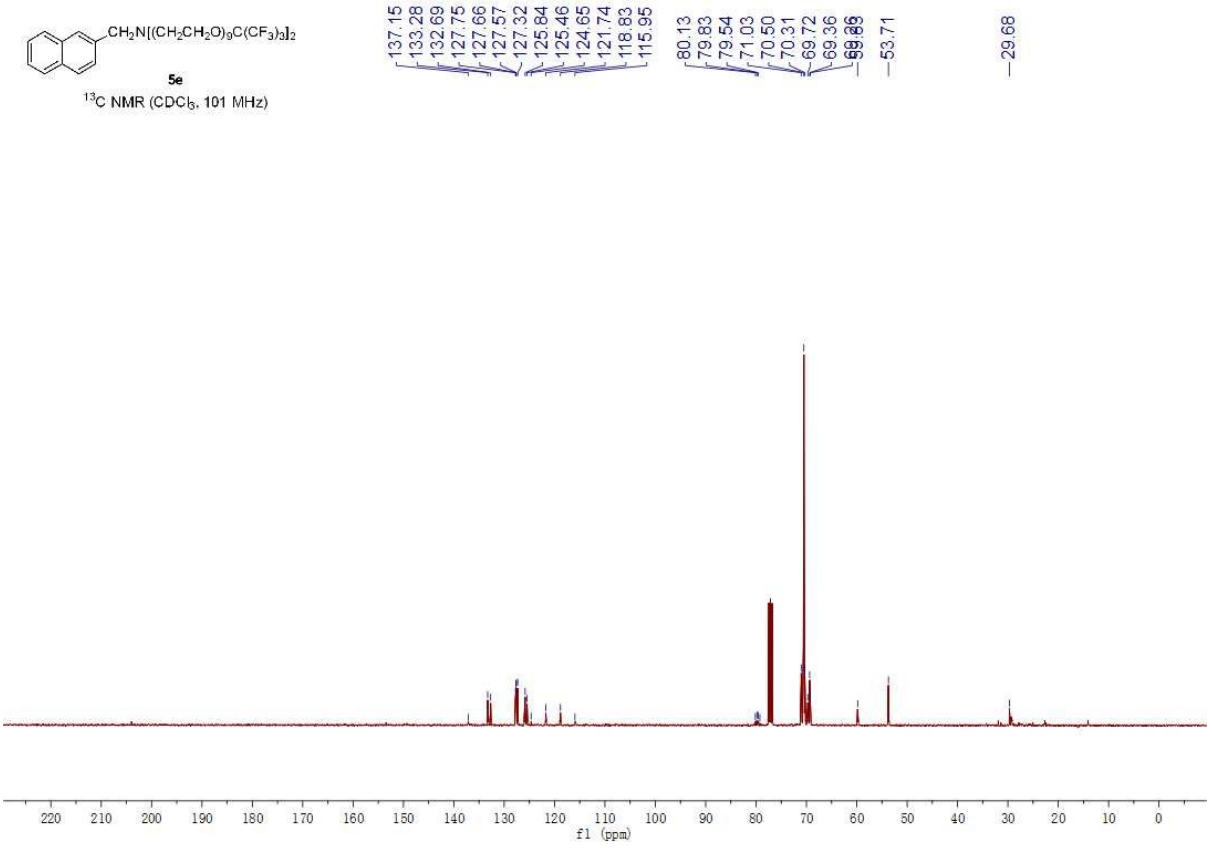

CH2N[(CH2CH2O)5(CH2)3CH3]2
5c
1H NMR (CDCl3, 400 MHz)
7.83
7.82
7.81
7.80
7.78
7.76
7.53
7.51
7.47
7.47
7.46
7.45
7.44
7.29
3.64
3.63
3.62
3.62
3.61
3.60
3.59
3.58
3.58
3.57
3.48
3.46
3.44
3.43
2.82
2.81
1.59
1.57
1.55
1.40
1.38
1.36
1.34
0.94
0.92
0.91
0.82
4.00
3.01
2.00
36.93
4.13
3.79
4.00
4.00
6.00
f1 (ppm)

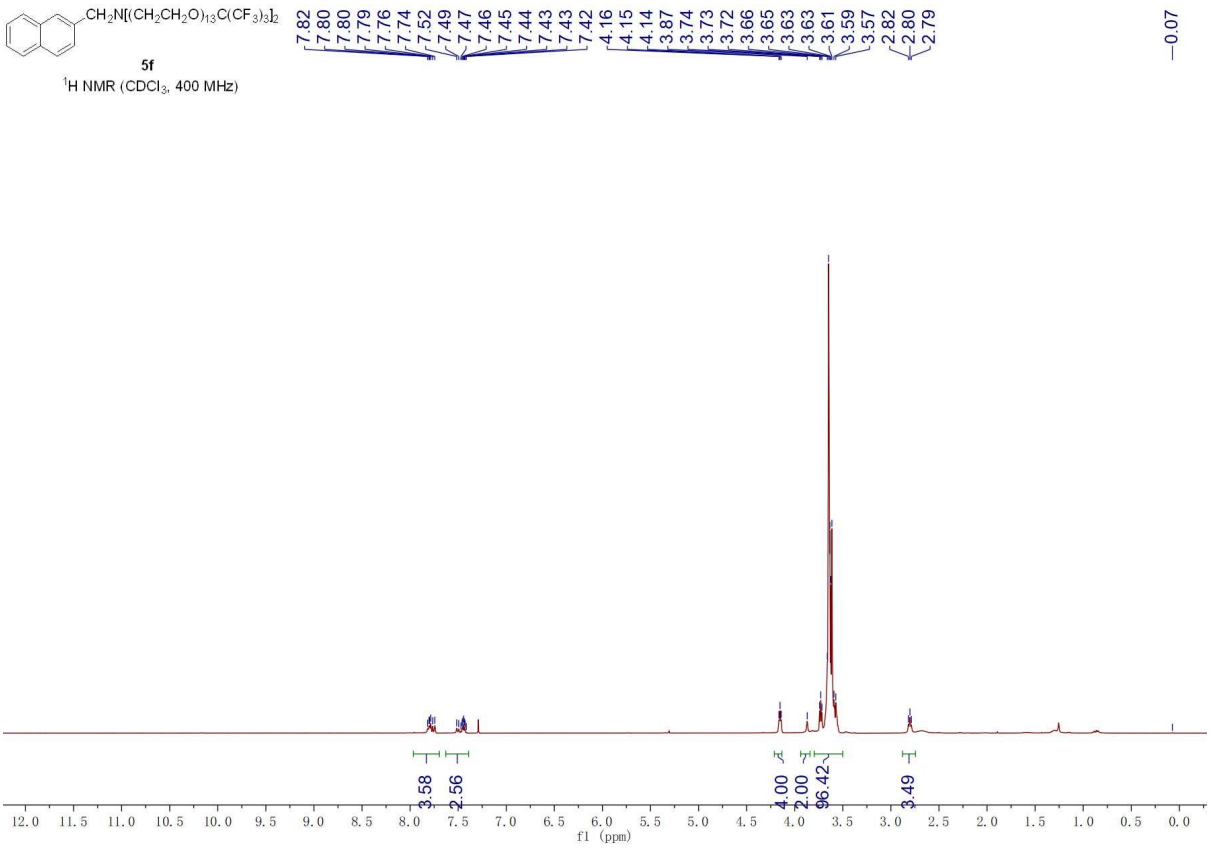

CH2N[(CH2CH2O)5(CH2)3CH3]2
5c
13C NMR (CDCl3, 101 MHz)
133.30
132.70
127.76
127.68
127.60
127.30
125.86
125.46
71.21
70.60
70.56
70.35
70.04
69.83
59.92
53.81
31.70
19.28
13.97
f1 (ppm)

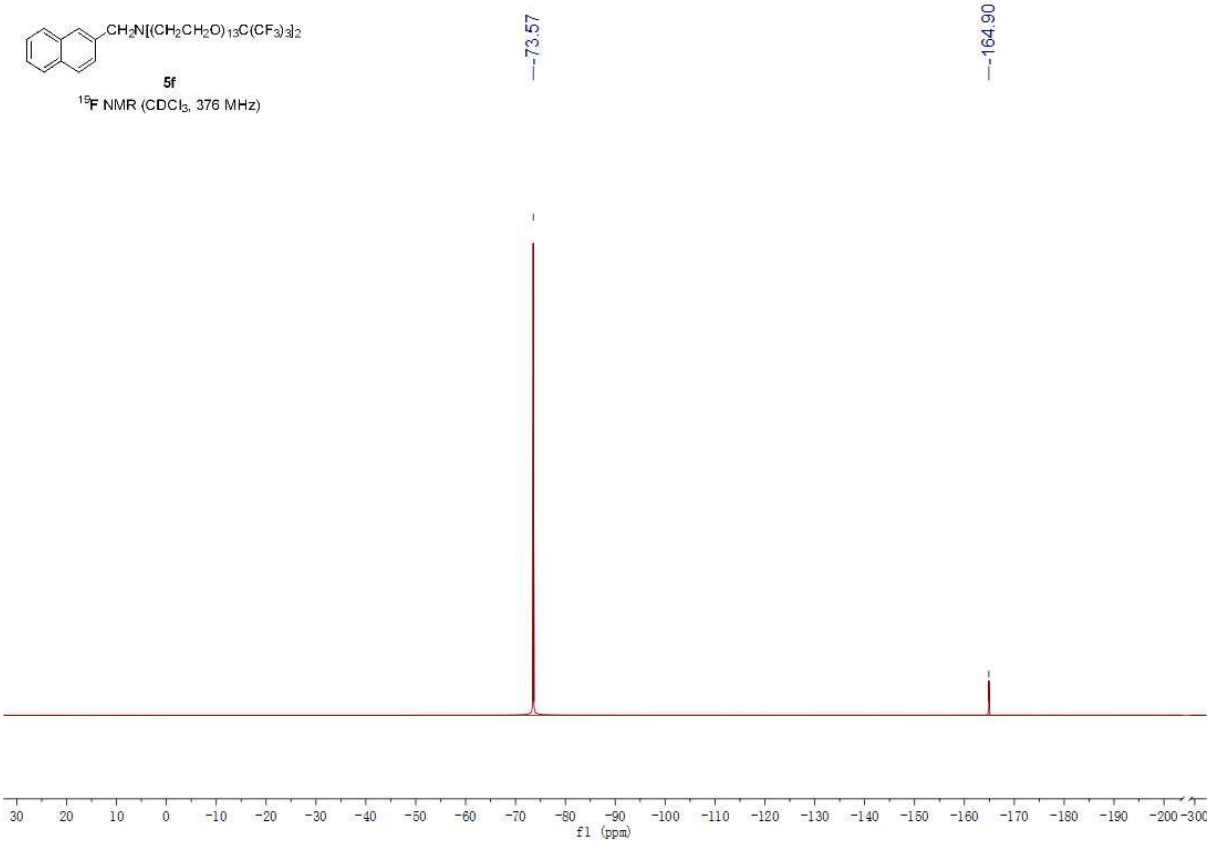

7.81
7.80
7.80
7.79
7.79
7.78
7.76
7.52
7.50
7.47
7.45
7.45
7.44
7.43
7.42
7.29
3.91
3.70
3.69
3.68
3.65
3.63
3.62
3.61
3.60
3.59
3.57
3.56
3.55
3.54
3.54
3.53
3.37
2.83
CH2N[(CH2CH2O)16CH3]2
5d
1H NMR (CDCl3, 400 MHz)
4.25
3.16
2.16
24.00
5.19
4.64
f1 (ppm)

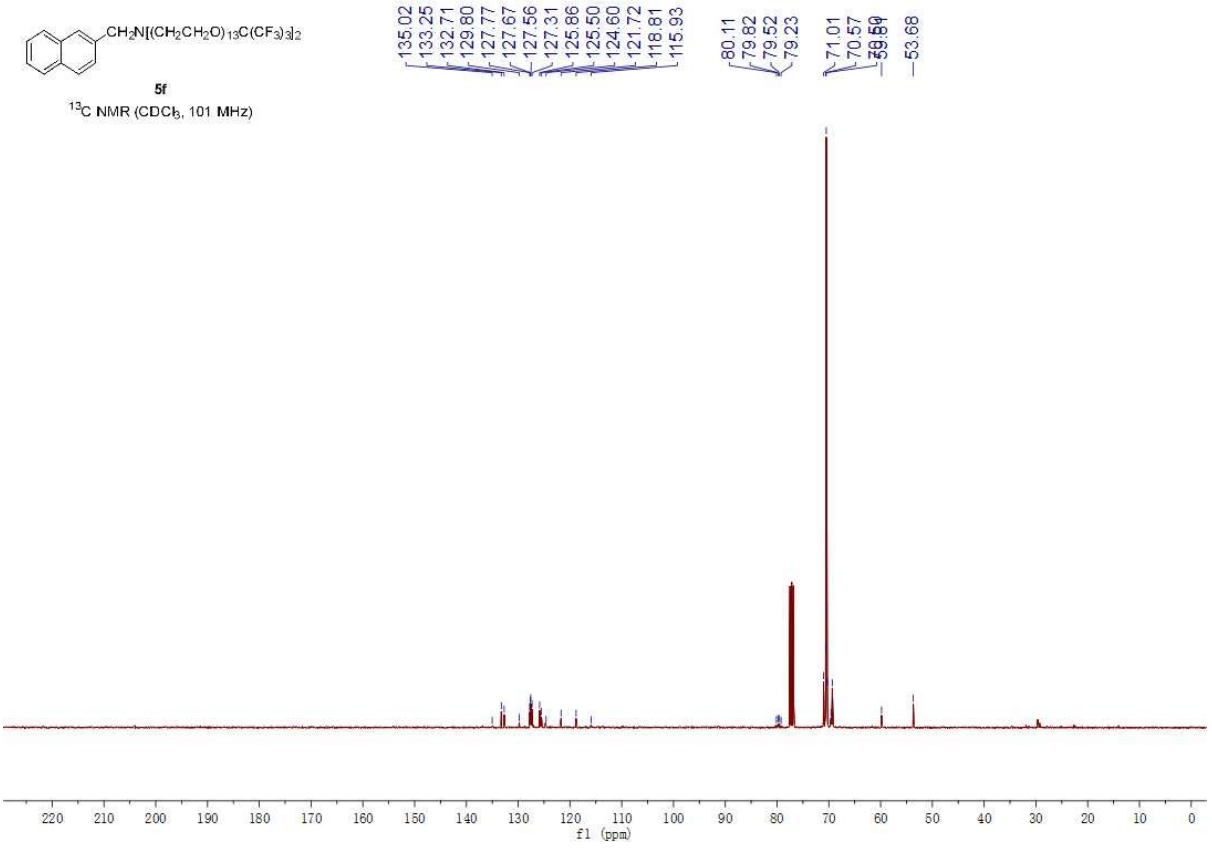

133.26
132.72
129.90
127.80
127.69
127.58
127.35
125.88
125.53
124.96
72.56
71.89
70.52
70.30
70.21
69.56
61.60
59.83
59.02
53.69
CH2N[(CH2CH2O)16CH3]2
5d
13C NMR (CDCl3, 101 MHz)
f1 (ppm)

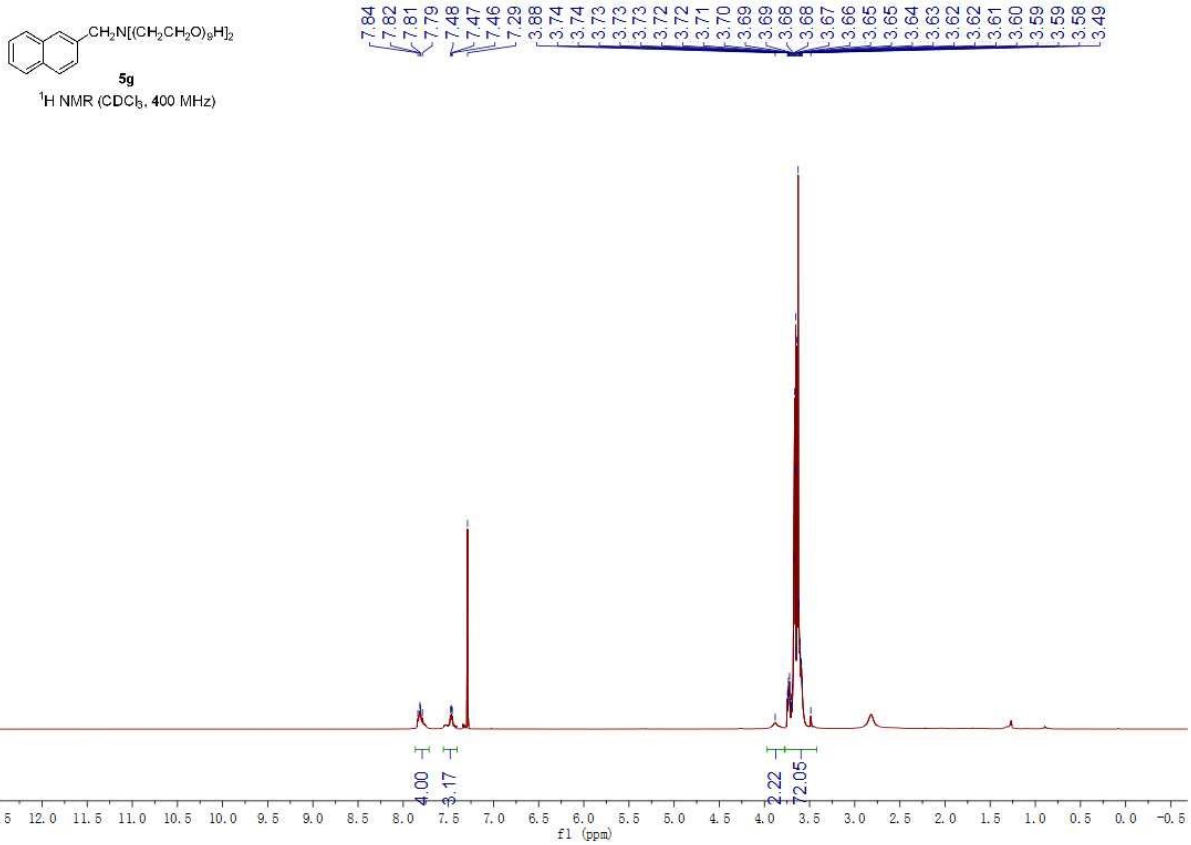

CH2N[(CH2CH2O)3C(CF3)3]2
5e
1H NMR (CDCl3, 400 MHz)
7.74
7.73
7.72
7.71
7.69
7.67
7.44
7.42
7.39
7.38
7.38
7.37
7.36
7.35
7.34
7.21
4.09
4.07
4.06
3.79
3.66
3.65
3.64
3.58
3.57
3.56
3.55
3.54
3.52
3.50
2.74
2.72
2.71
0.00
3.85
2.89
4.00
2.00
64.00
3.64
f1 (ppm)
CH2N[(CH2CH2O)3C(CF3)3]2
5e
19F NMR (CDCl3, 376 MHz)
-73.59
-164.90
f1 (ppm)

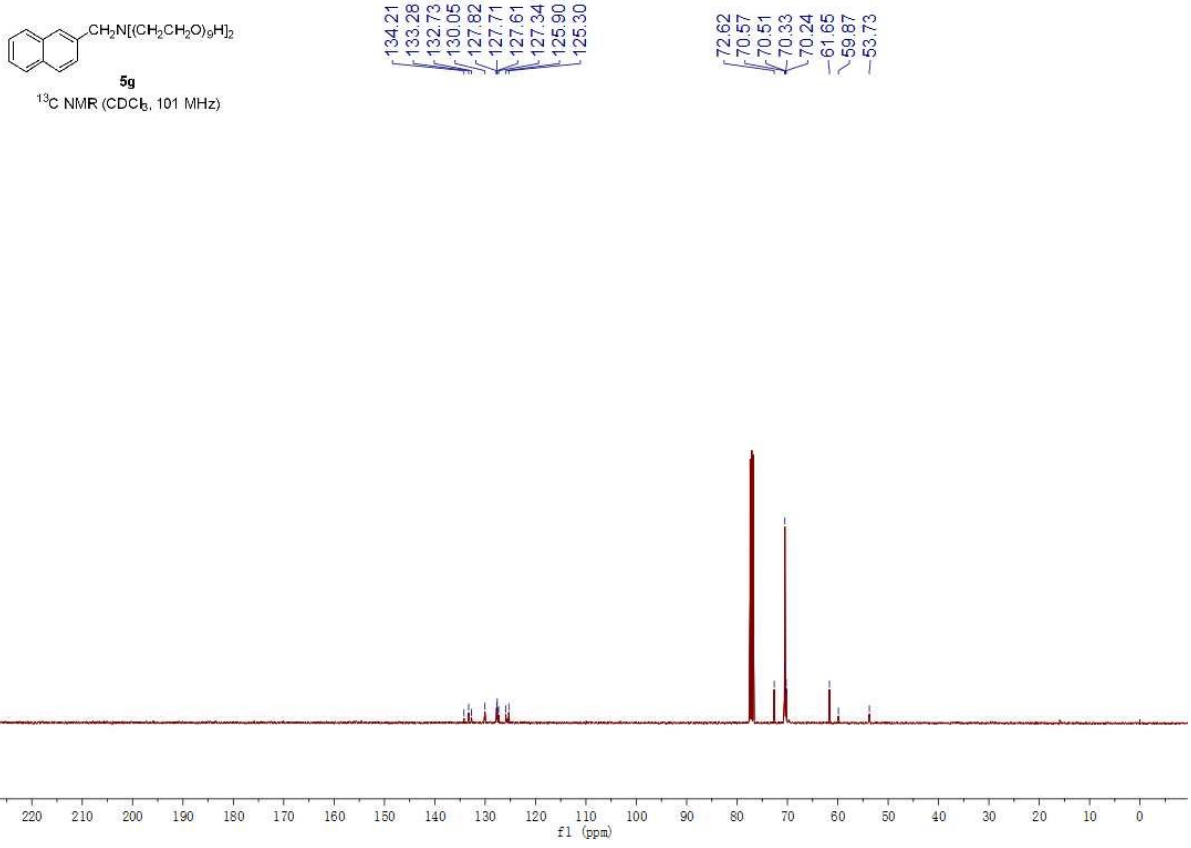

CH2N[(CH2CH2O)9C(CF3)3]2
5e
13C NMR (CDCl3, 101 MHz)
137.15
133.28
132.69
127.75
127.66
127.57
127.32
125.84
125.46
124.65
121.74
118.83
115.95
80.13
79.83
79.54
71.03
70.50
70.31
69.72
69.36
53.71
29.68
f1 (ppm)

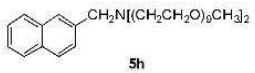

CH2N[(CH2CH2O)13C(CF3)3]2
5f
1H NMR (CDCl3, 400 MHz)
7.82
7.80
7.80
7.79
7.76
7.74
7.52
7.49
7.47
7.46
7.45
7.44
7.43
7.43
7.42
4.16
4.15
4.14
3.87
3.74
3.73
3.72
3.66
3.65
3.63
3.63
3.61
3.59
3.57
2.82
2.80
2.79
0.07
3.58
2.56
4.00
2.00
96.42
3.49
f1 (ppm)


CH2N[(CH2CH2O)13C(CF3)3]2
5f
19F NMR (CDCl3, 376 MHz)
-73.57
-164.90
f1 (ppm)


CH2N[(CH2CH2O)13C(CF3)3]2
5f
13C NMR (CDCl3, 101 MHz)
135.02
133.25
132.71
129.80
127.77
127.67
127.56
127.31
125.86
125.50
124.60
121.72
118.81
115.93
80.11
79.82
79.52
79.23
71.01
70.57
53.68
f1 (ppm)

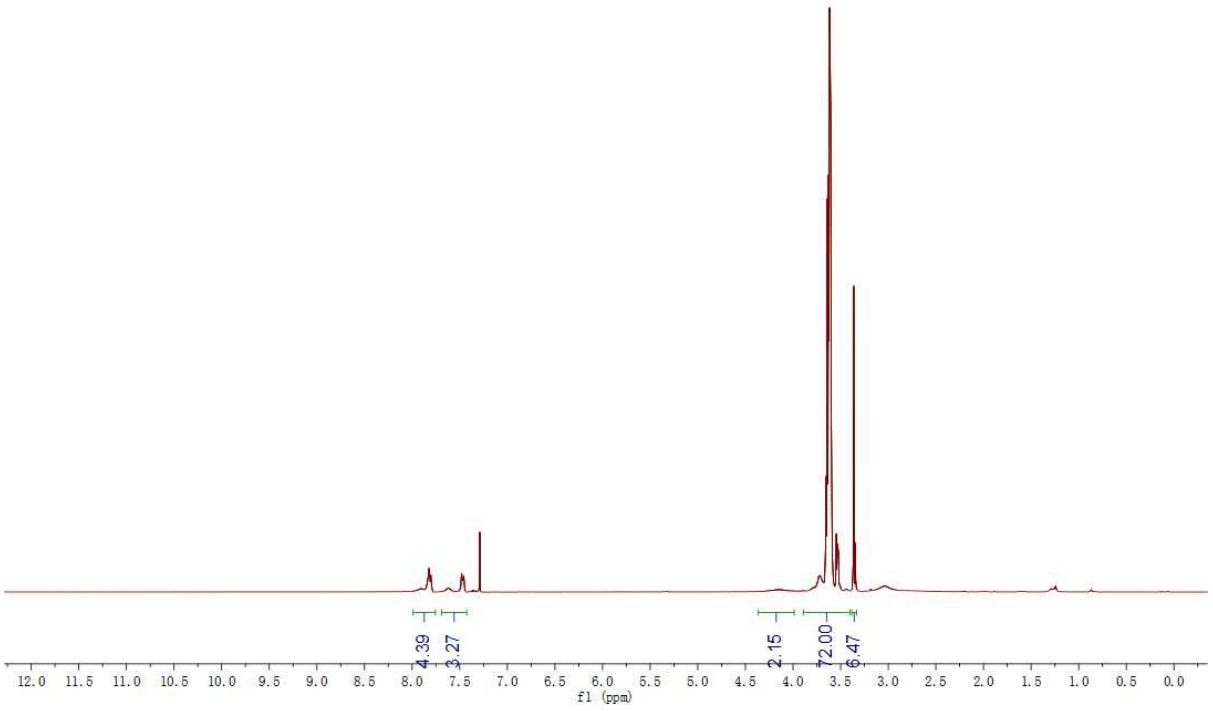

CH2N[(CH2CH2O)8H]2
5g
1H NMR (CDCl3, 400 MHz)
7.84
7.82
7.81
7.79
7.48
7.47
7.46
7.29
3.88
3.74
3.74
3.73
3.73
3.73
3.72
3.72
3.71
3.70
3.69
3.69
3.68
3.68
3.67
3.66
3.65
3.65
3.64
3.63
3.62
3.62
3.61
3.60
3.59
3.59
3.58
3.49
4.00
3.17
2.22
72.05
f1 (ppm)

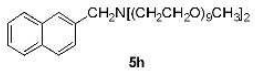

CH2N[(CH2CH2O)8H]2
5g
13C NMR (CDCl3, 101 MHz)
134.21
133.28
132.73
130.05
127.82
127.71
127.61
127.34
125.90
125.30
72.62
70.57
70.51
70.33
70.24
61.65
59.87
53.73
f1 (ppm)



5h

^{1}H NMR ($CDCl_3$, 400 MHz)

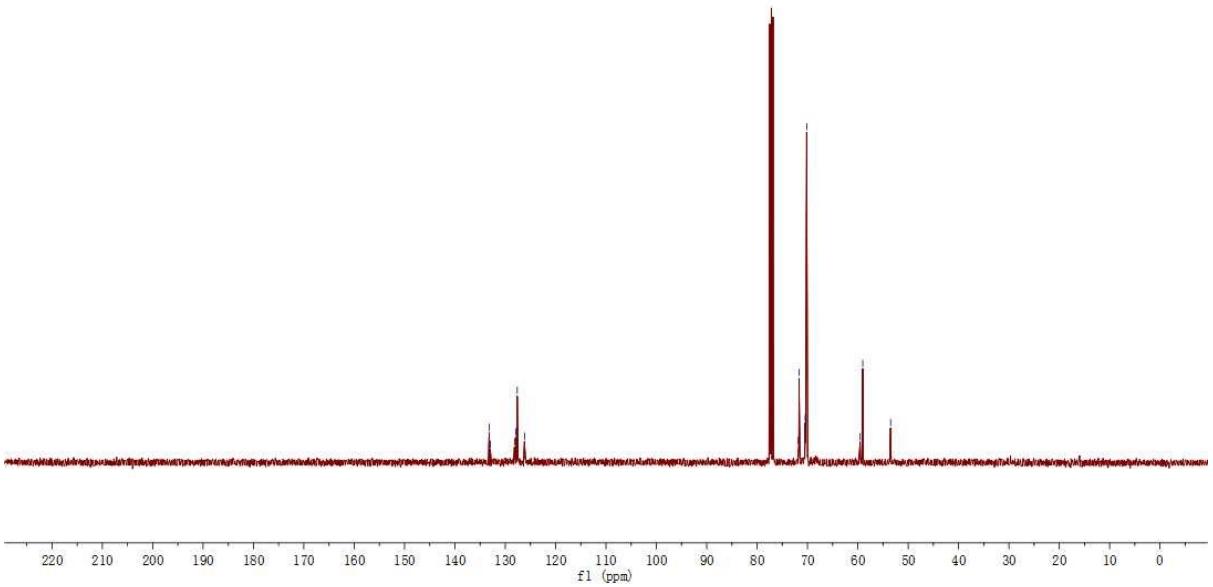


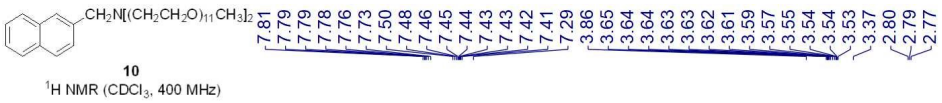


5h

^{13}C NMR ($CDCl_3$, 101 MHz)

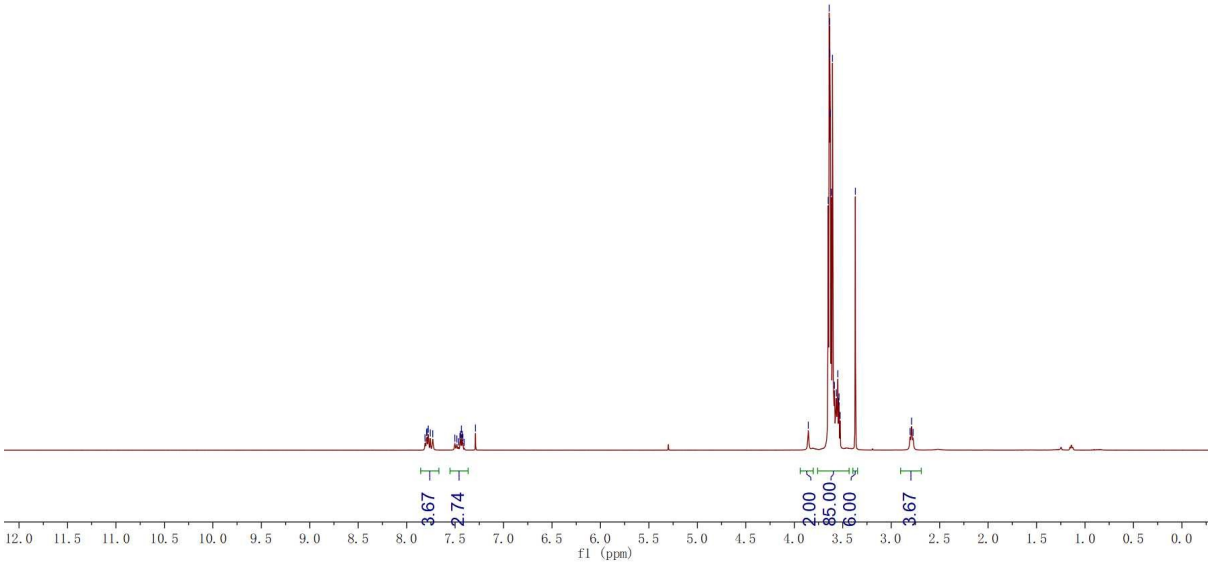
133.18
132.95
128.16
127.88
127.62
126.18
71.82
71.66
70.45
70.38
70.15
59.54
59.02
53.48

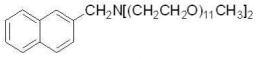


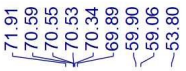


10

^{1}H NMR ($CDCl_3$, 400 MHz)

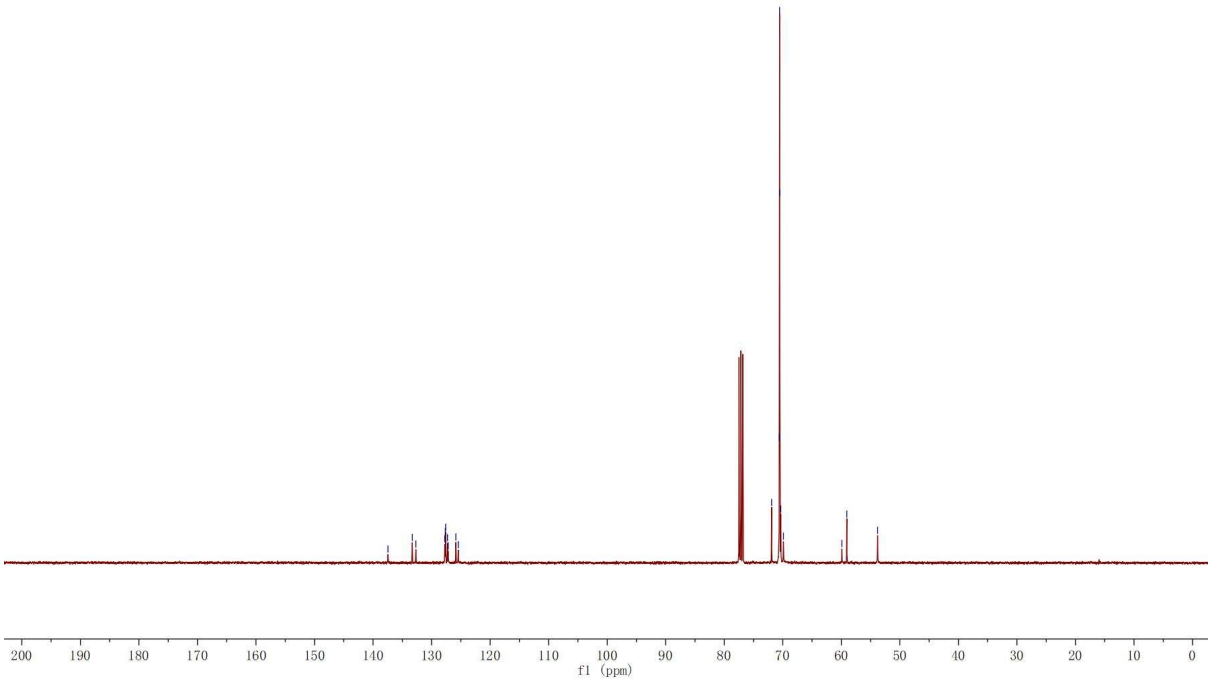




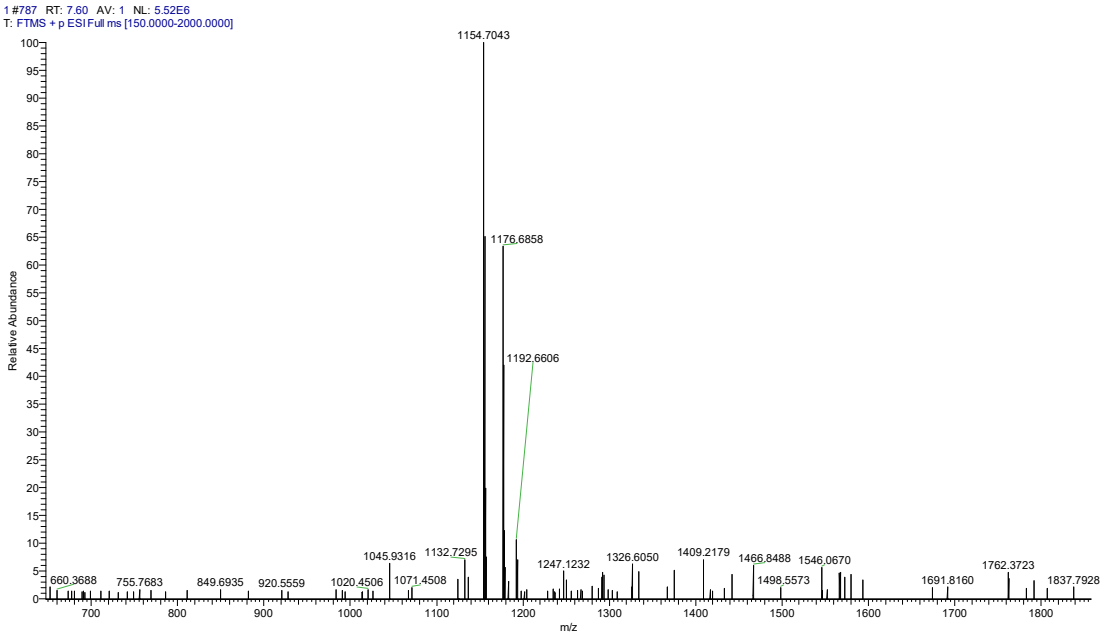


10

^{13}C NMR ($CDCl_3$, 101 MHz)



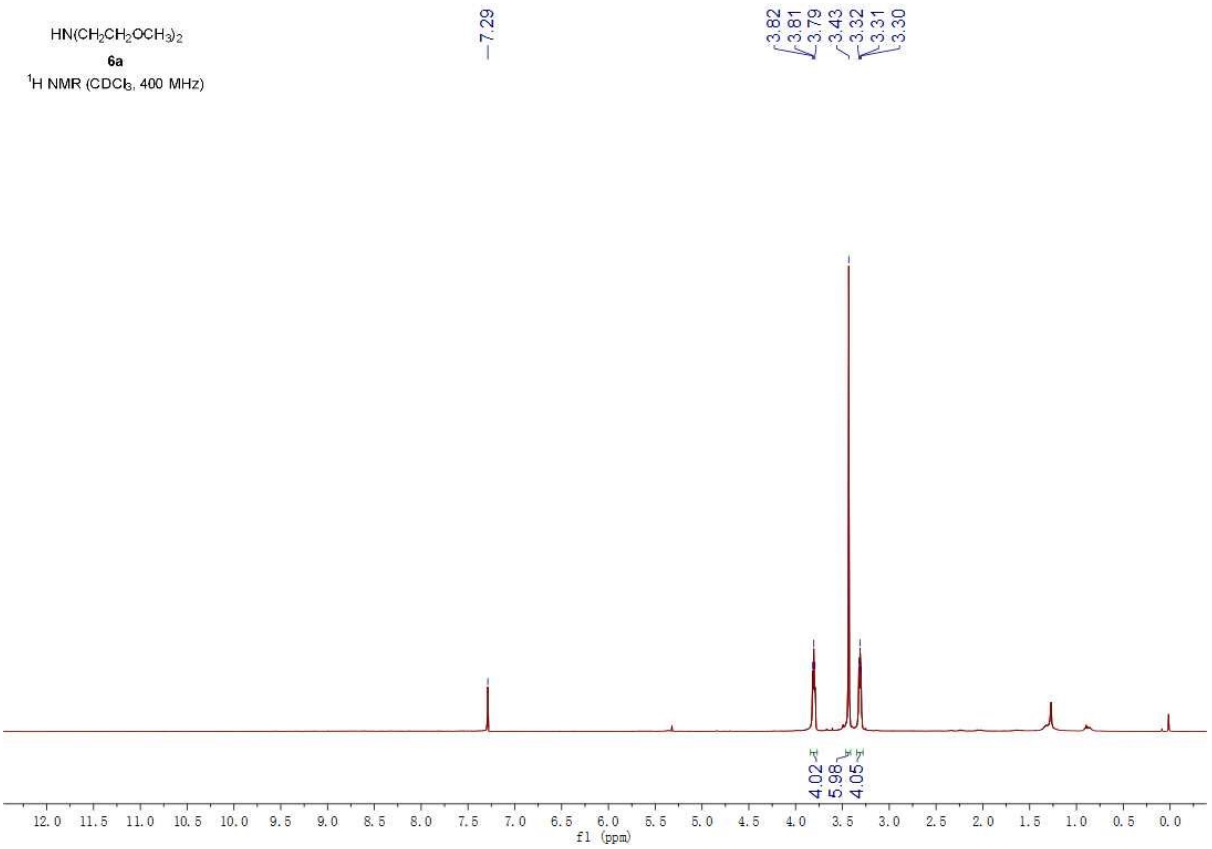
HRMS of compound **10**



$HN(CH_2CH_2OCH_3)_2$

6a

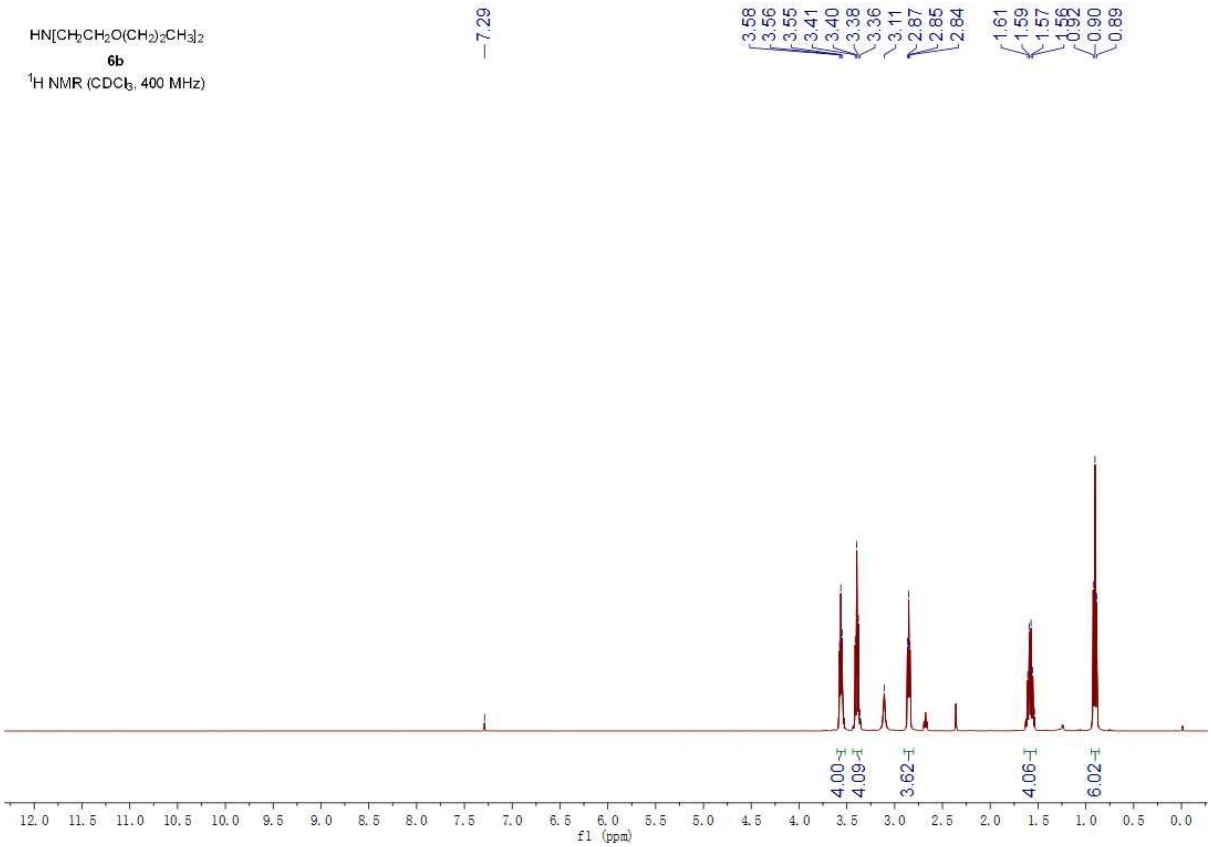
^{1}H NMR ($CDCl_3$, 400 MHz)



$HN[CH_2CH_2O(CH_2)_2CH_3]_2$

6b

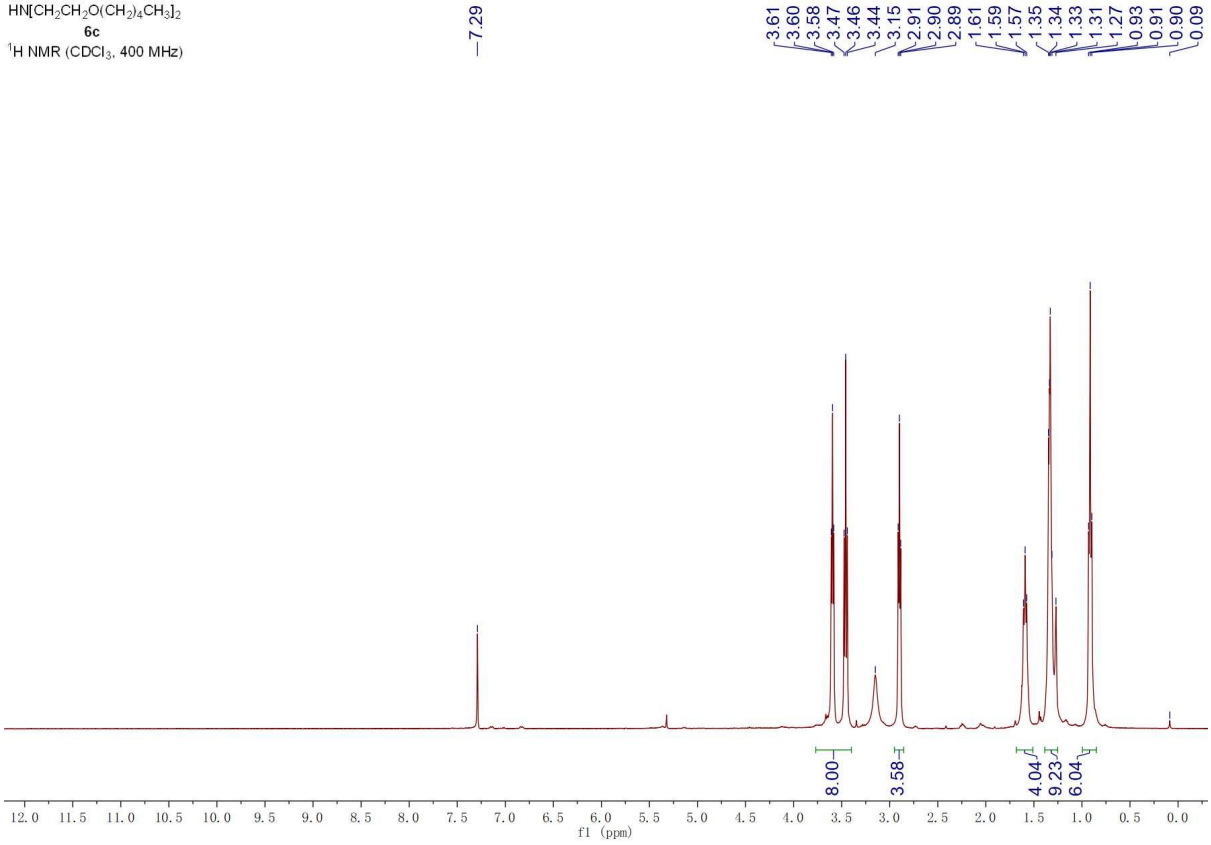
^1H NMR ($CDCl_3$, 400 MHz)

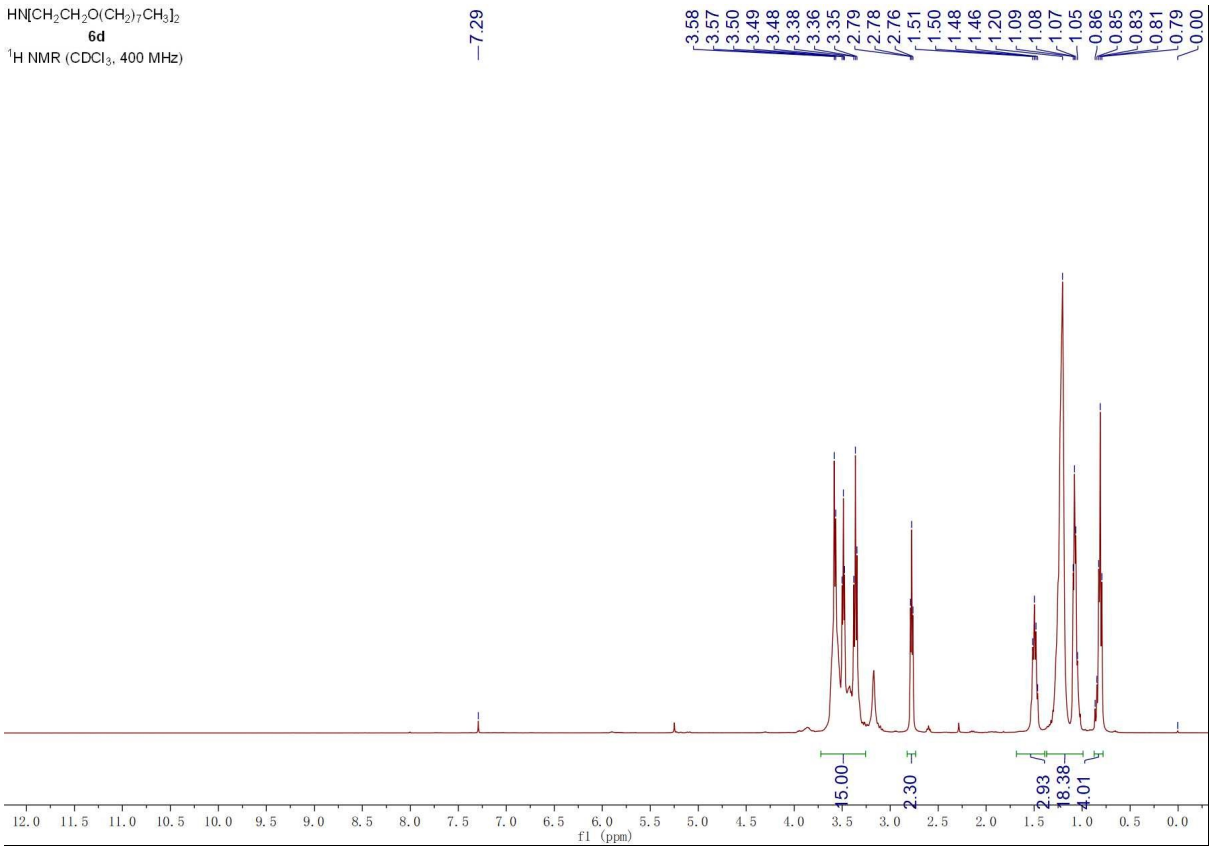


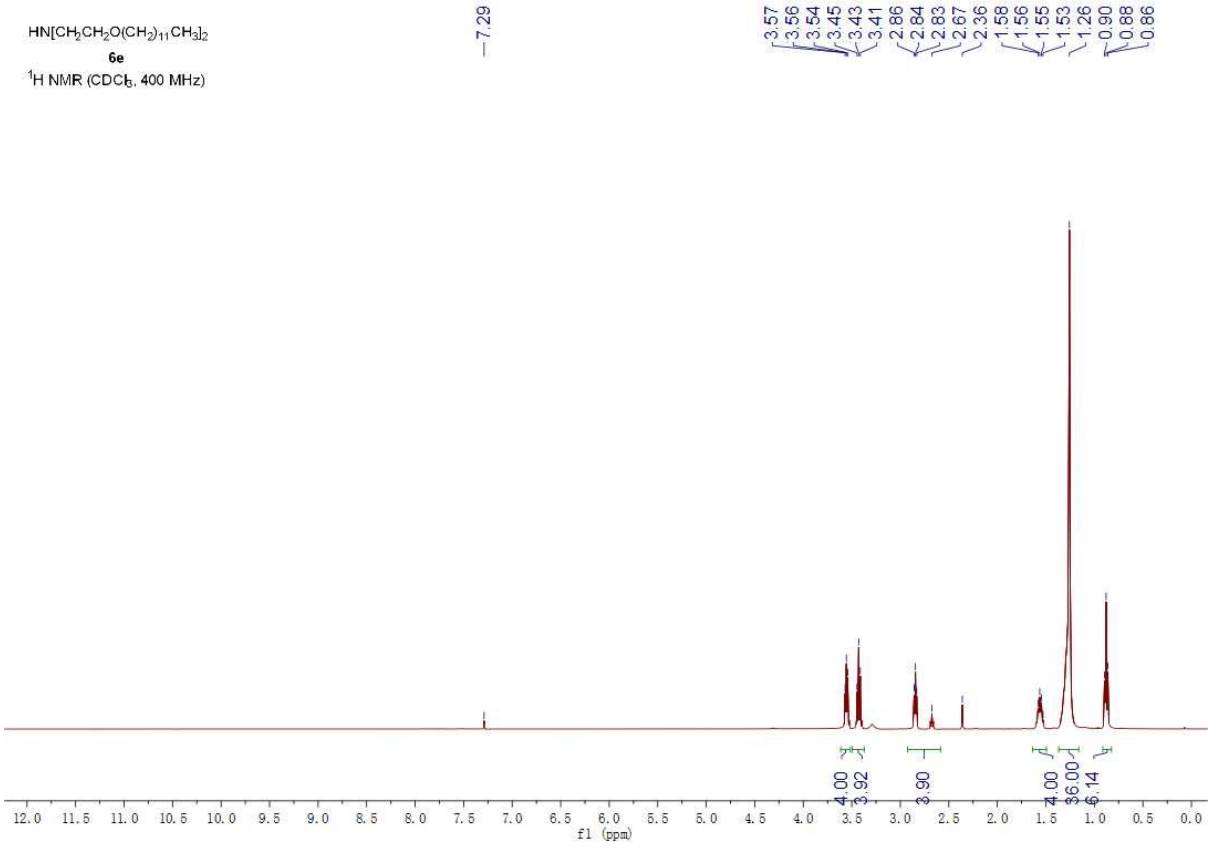
$HN[CH_2CH_2O(CH_2)_4CH_3]_2$

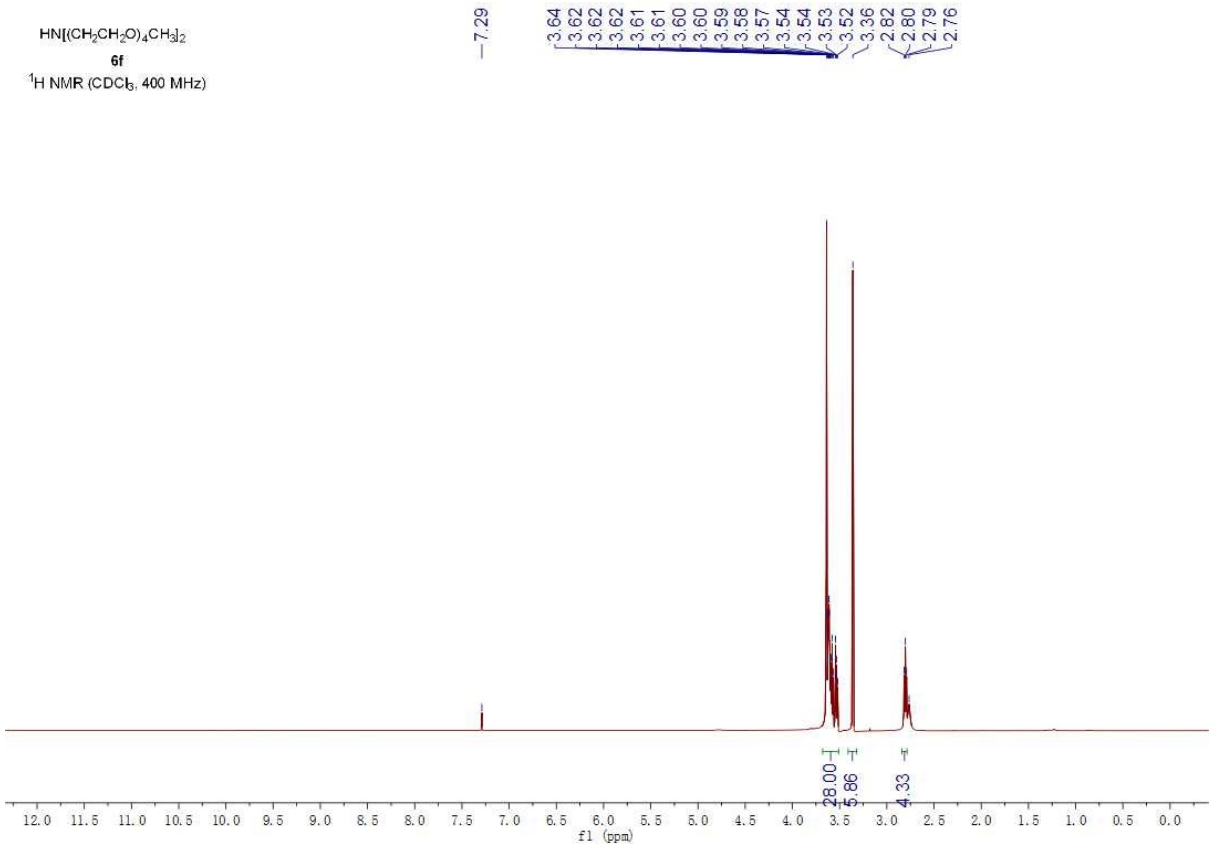
6c

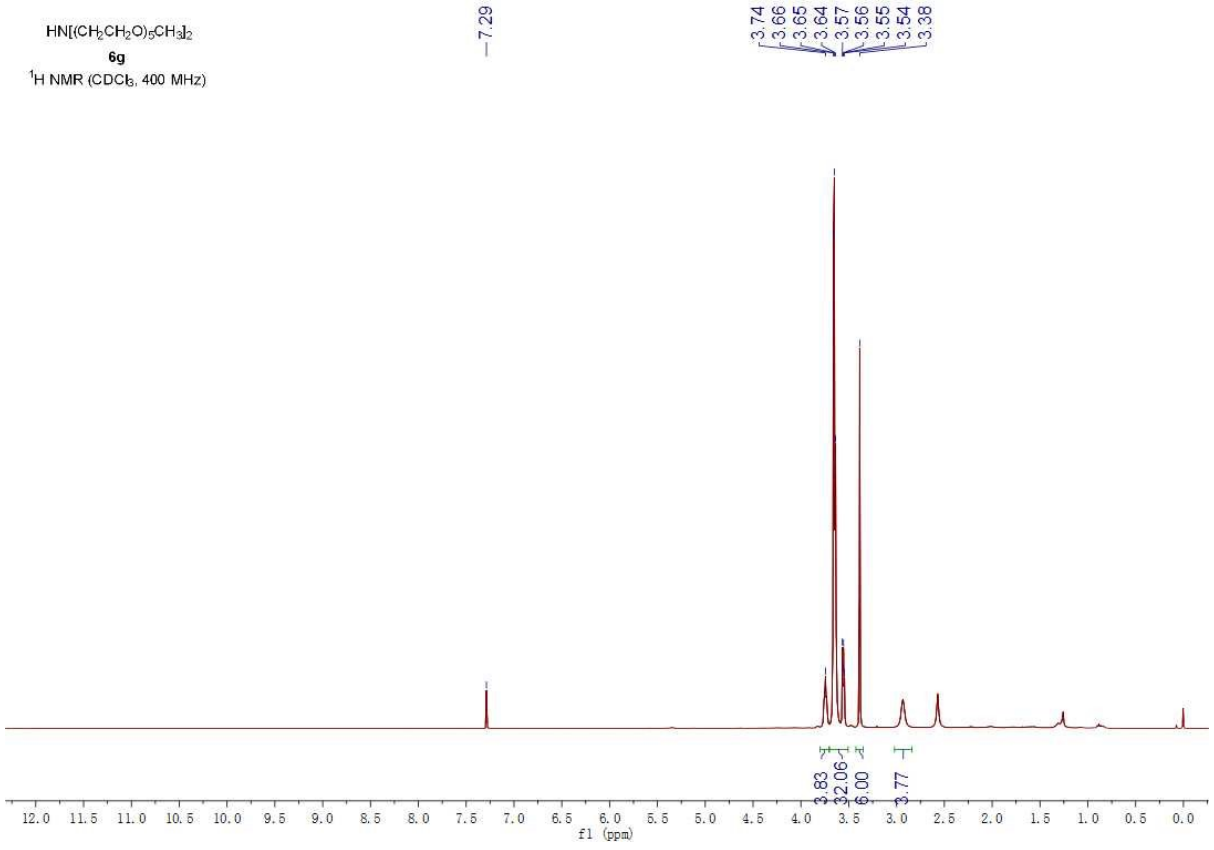
^1H NMR ($CDCl_3$, 400 MHz)




HN[CH2CH2O(CH2)7CH3]2
6d
1H NMR (CDCl3, 400 MHz)
7.29
3.58
3.57
3.50
3.49
3.48
3.38
3.36
3.35
2.79
2.78
2.76
1.51
1.50
1.48
1.46
1.20
1.09
1.08
1.07
1.05
0.86
0.85
0.83
0.81
0.79
0.00
15.00
2.30
2.93
18.38
4.01
f1 (ppm)


HN[CH2CH2O(CH2)11CH3]2
6e
1H NMR (CDCl3, 400 MHz)
7.29
3.57
3.56
3.54
3.45
3.43
3.41
2.86
2.84
2.83
2.67
2.36
1.58
1.56
1.55
1.53
1.26
0.90
0.88
0.86
4.00
3.92
3.90
4.00
36.00
6.14
f1 (ppm)

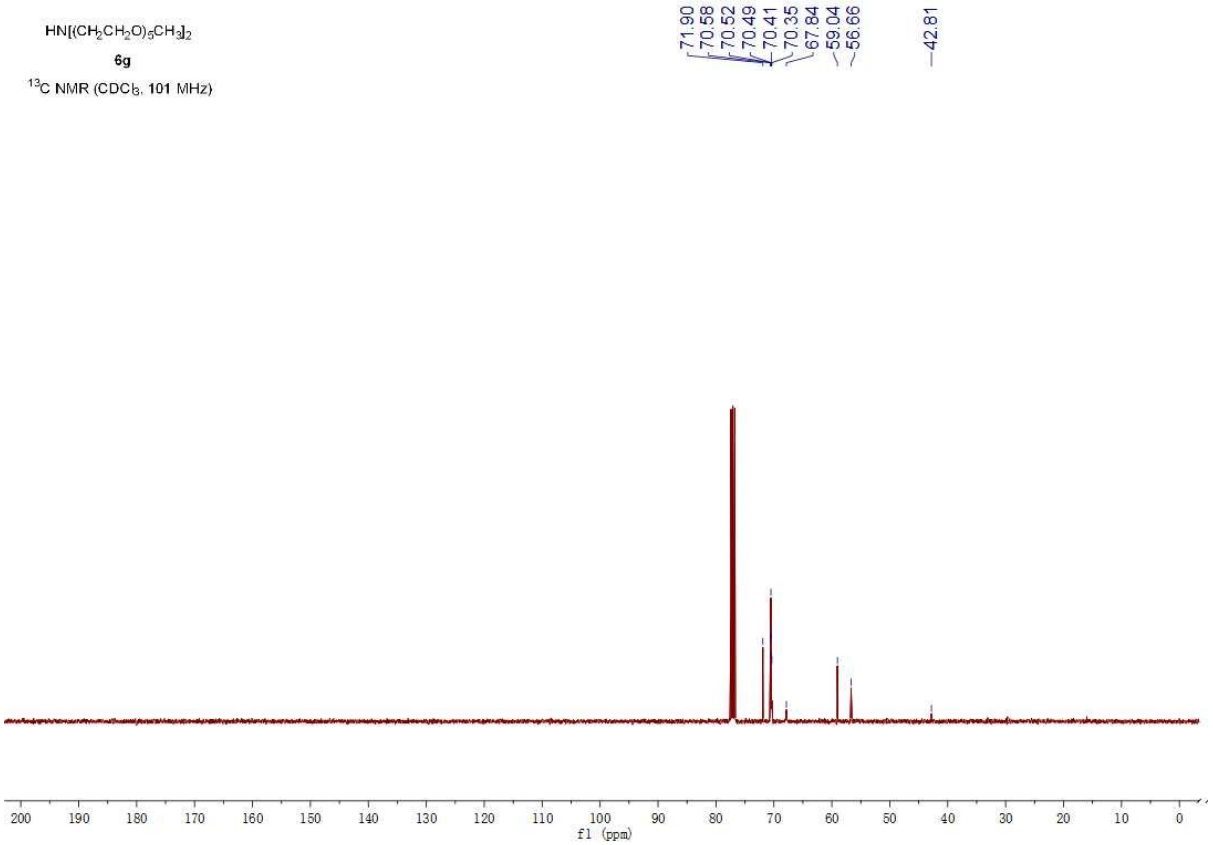

HN[(CH2CH2O)4CH3]2
6f
1H NMR (CDCl3, 400 MHz)
7.29
3.64
3.62
3.62
3.62
3.61
3.61
3.60
3.60
3.59
3.58
3.57
3.54
3.54
3.53
3.52
3.36
2.82
2.80
2.79
2.76
28.00
5.86
4.33
f1 (ppm)


HN[(CH2CH2O)5CH3]2
6g
1H NMR (CDCl3, 400 MHz)
7.29
3.74
3.66
3.65
3.64
3.57
3.56
3.55
3.54
3.38
3.83
32.06
6.00
3.77
f1 (ppm)

$HN[(CH_2CH_2O)_5CH_3]_2$

6g

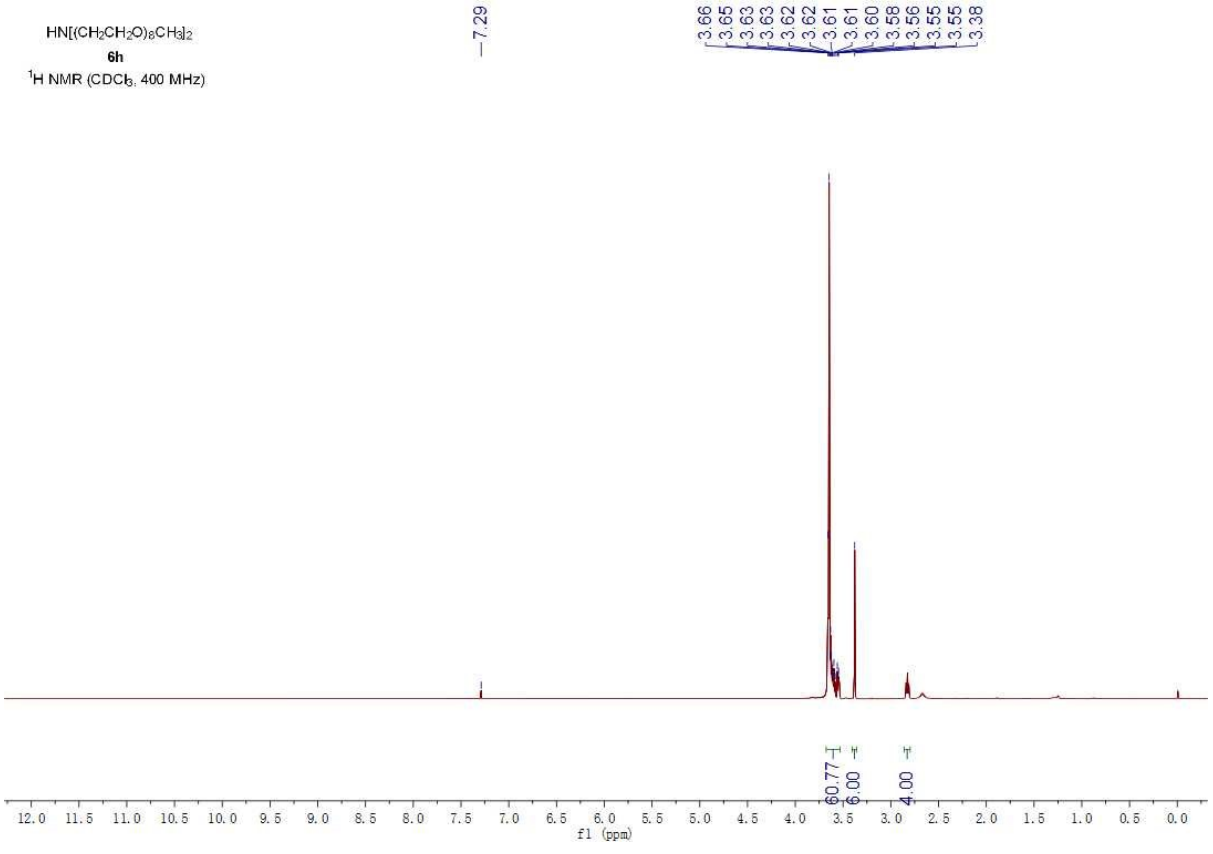
^{13}C NMR ($CDCl_3$, 101 MHz)

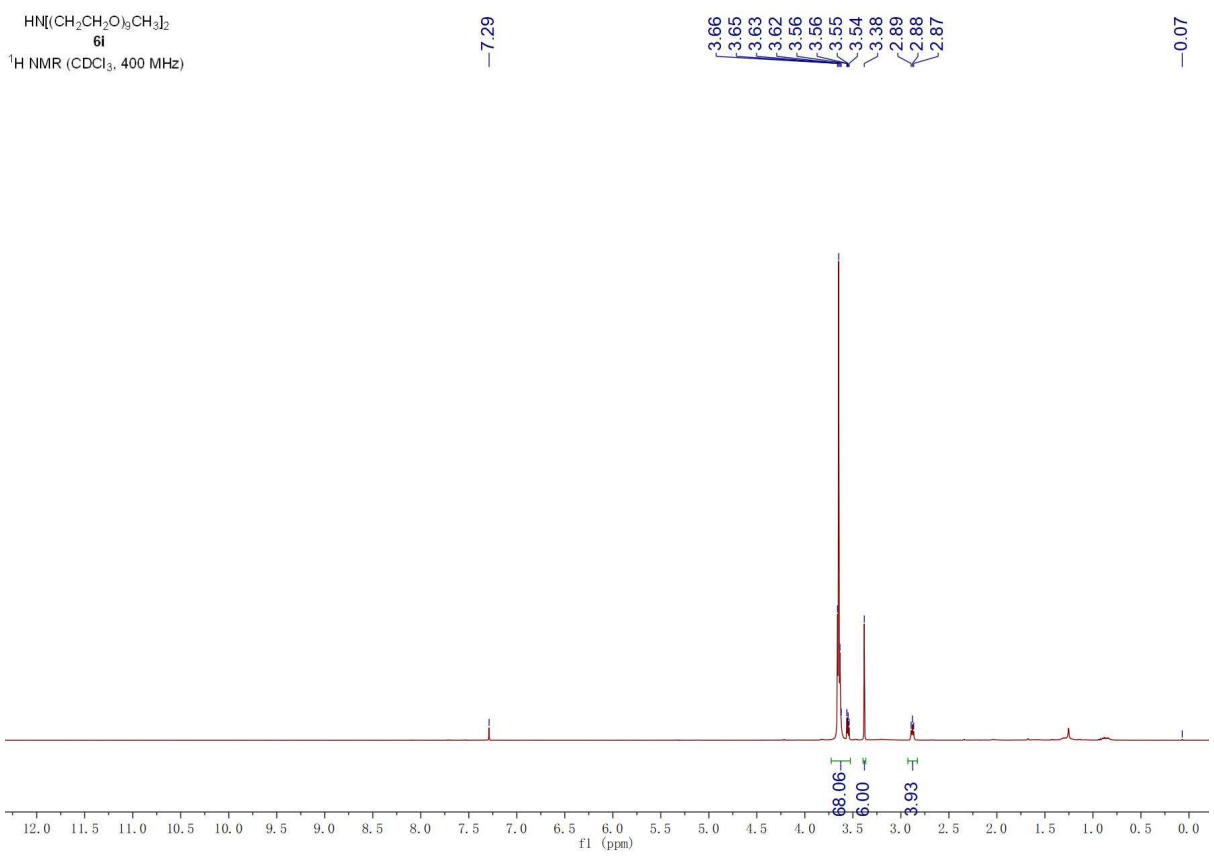


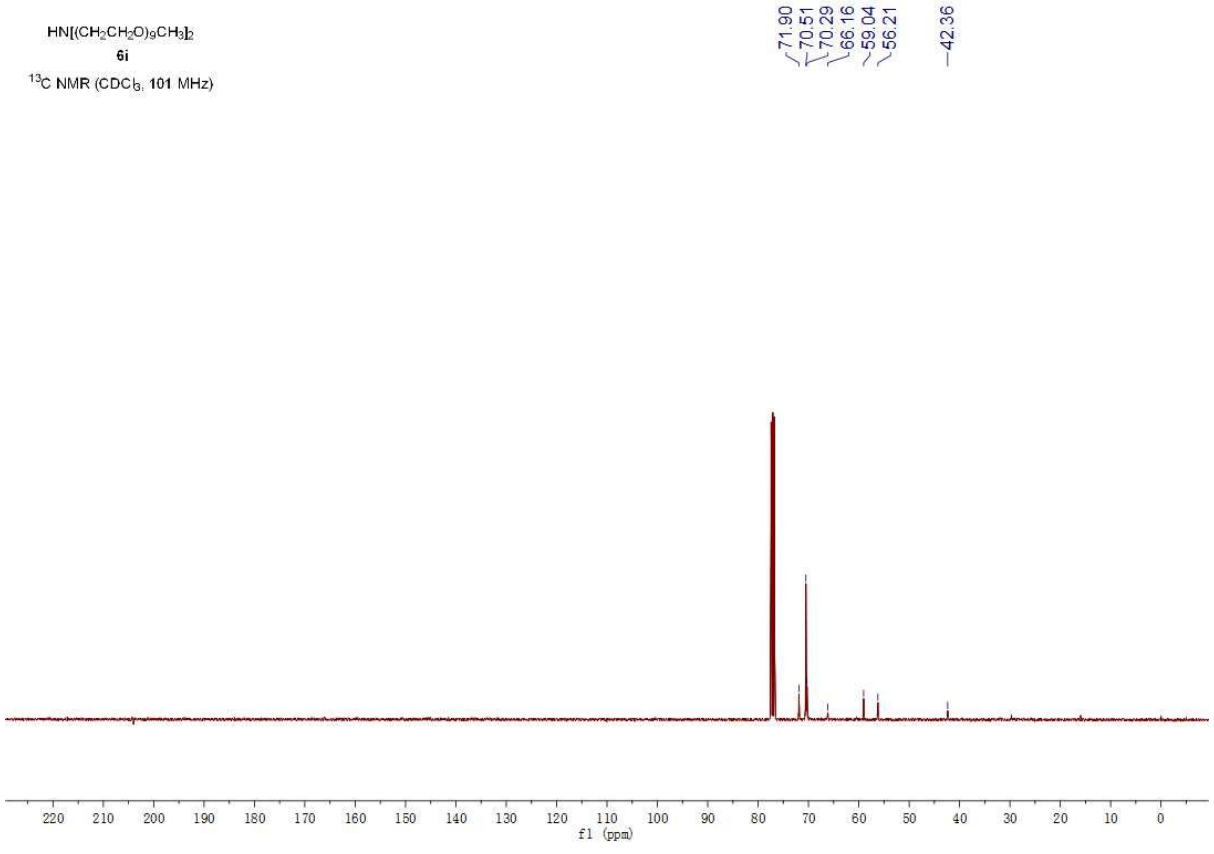
$HN[(CH_2CH_2O)_8CH_3]_2$

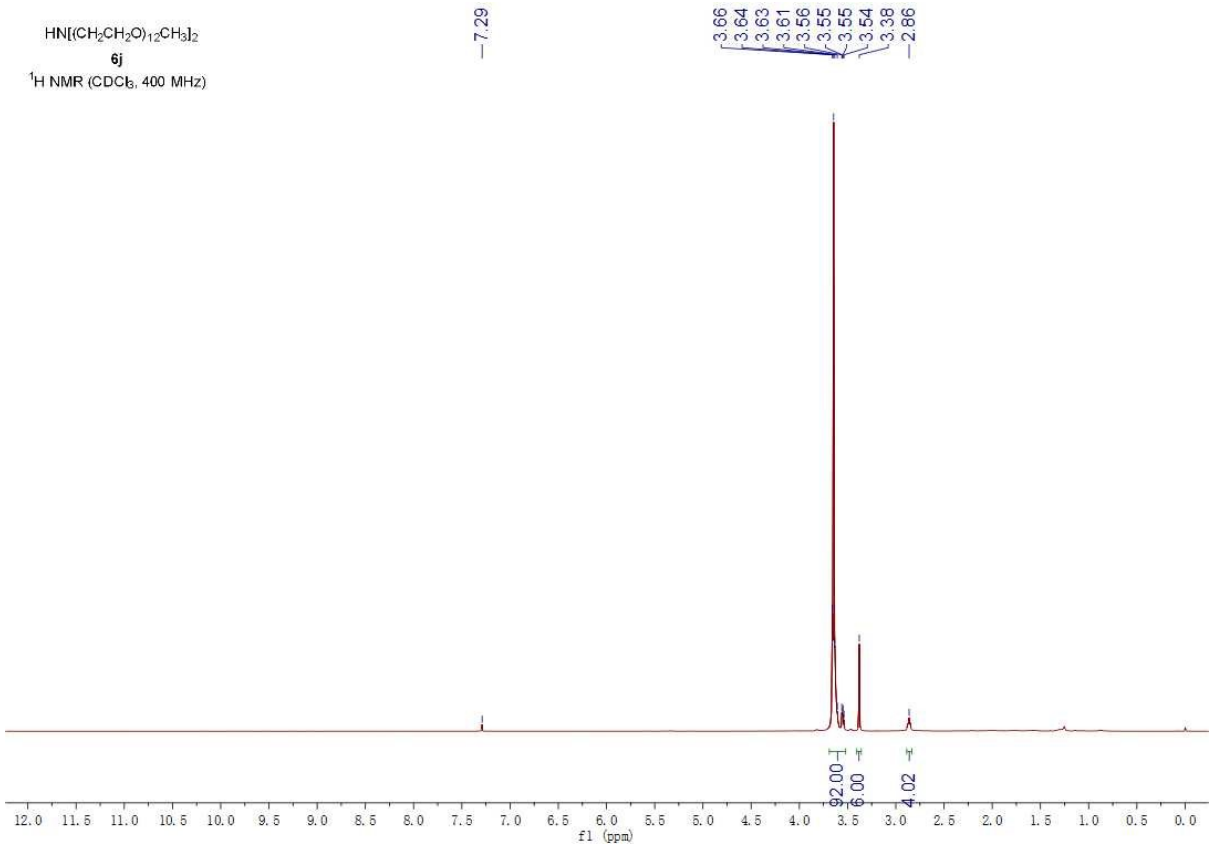
6h

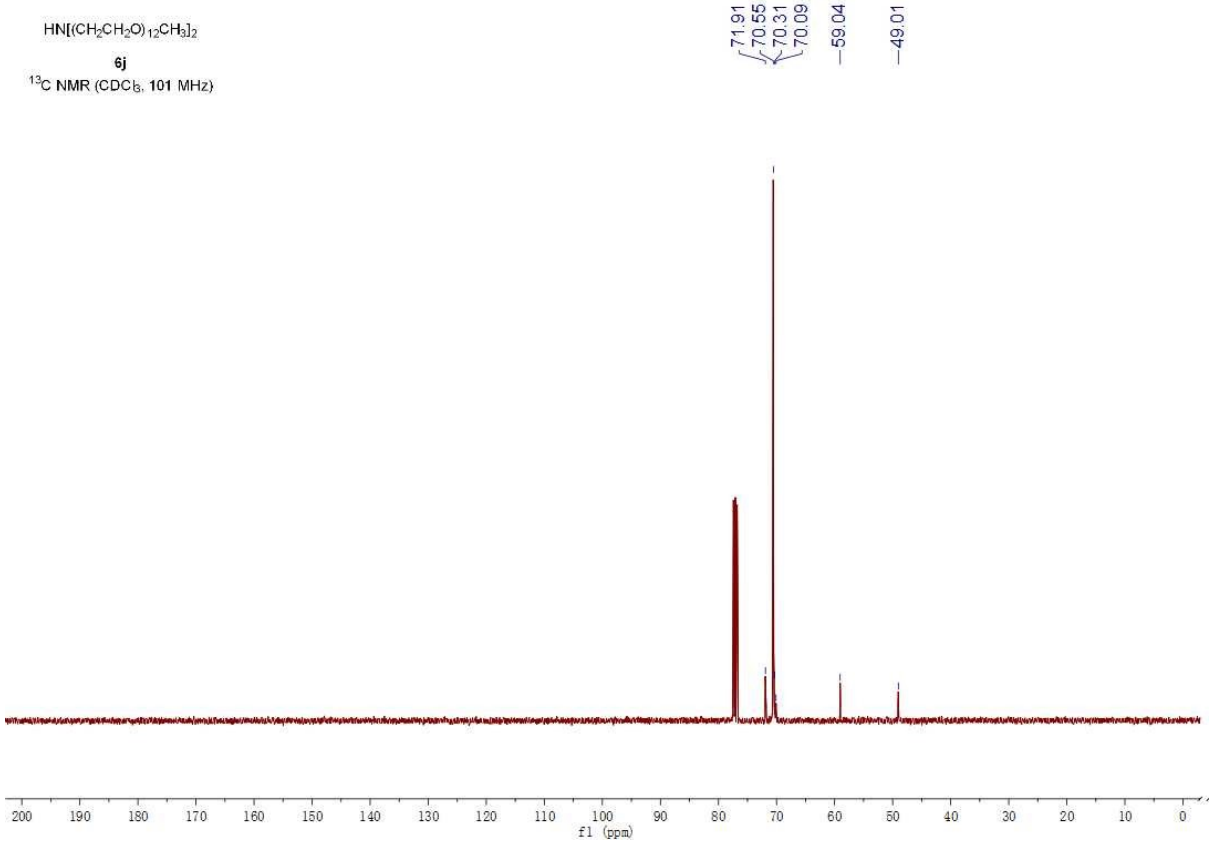
^{1}H NMR ($CDCl_3$, 400 MHz)




HN[(CH2CH2O)9CH3]2
6i
1H NMR (CDCl3, 400 MHz)
7.29
3.66
3.65
3.63
3.62
3.56
3.56
3.55
3.54
3.38
2.89
2.88
2.87
0.07
68.06
6.00
3.93
f1 (ppm)


HN[(CH2CH2O)9CH3]2
6i
13C NMR (CDCl3, 101 MHz)
71.90
70.51
70.29
66.16
59.04
56.21
42.36
f1 (ppm)

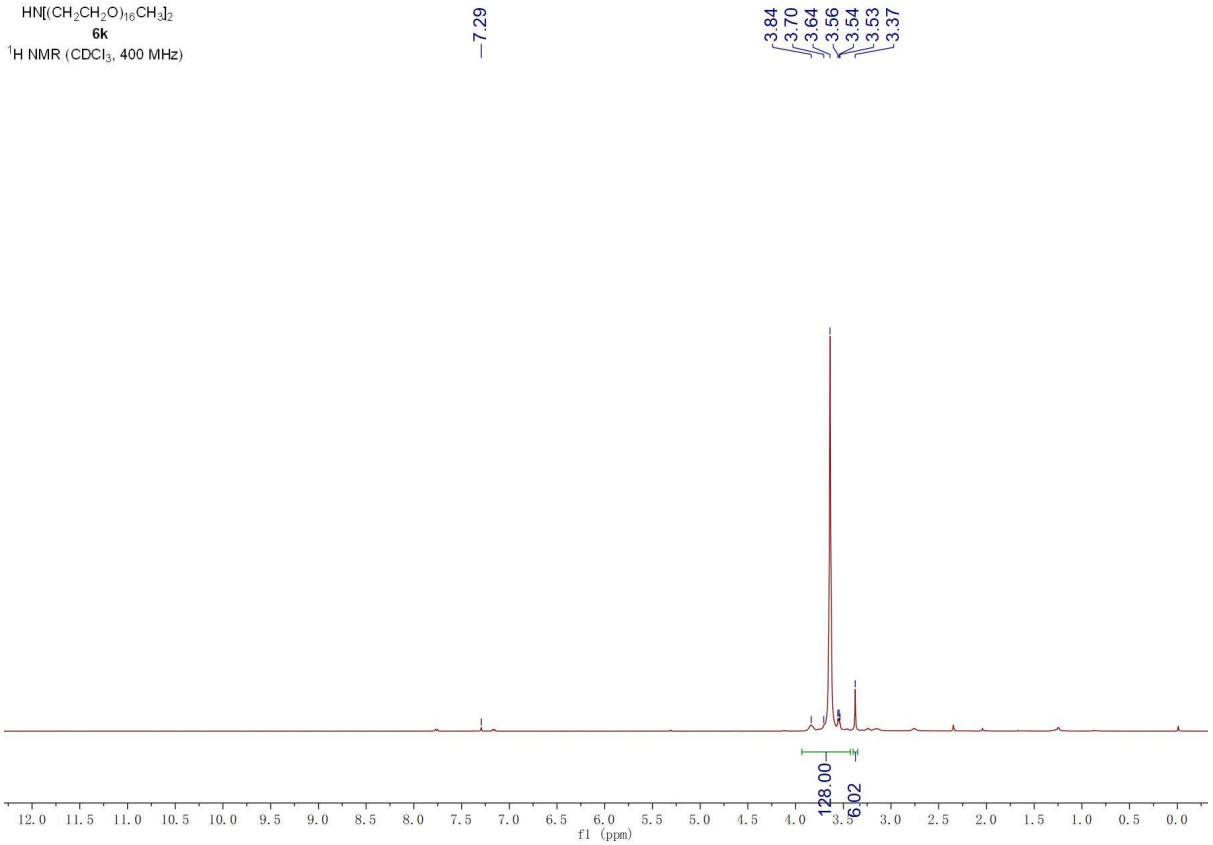

HN[(CH2CH2O)12CH3]2
6j
1H NMR (CDCl3, 400 MHz)
7.29
3.66
3.64
3.63
3.61
3.56
3.55
3.55
3.54
3.38
2.86
92.00
6.00
4.02
f1 (ppm)


HN[(CH2CH2O)12CH3]2
6j
13C NMR (CDCl3, 101 MHz)
71.91
70.55
70.31
70.09
59.04
49.01
f1 (ppm)

$HN[(CH_2CH_2O)_{16}CH_3]_2$

6k

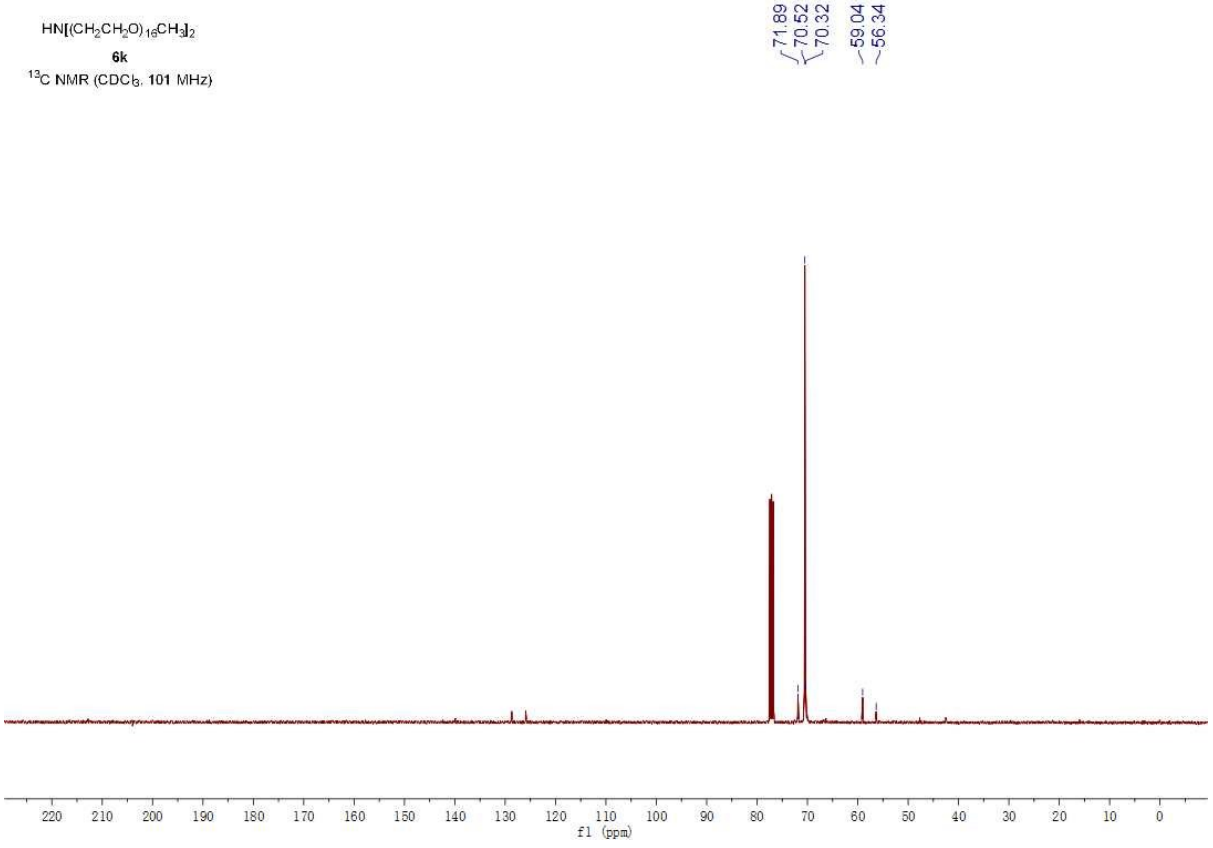
^1H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_{16}CH_3]_2$

6k

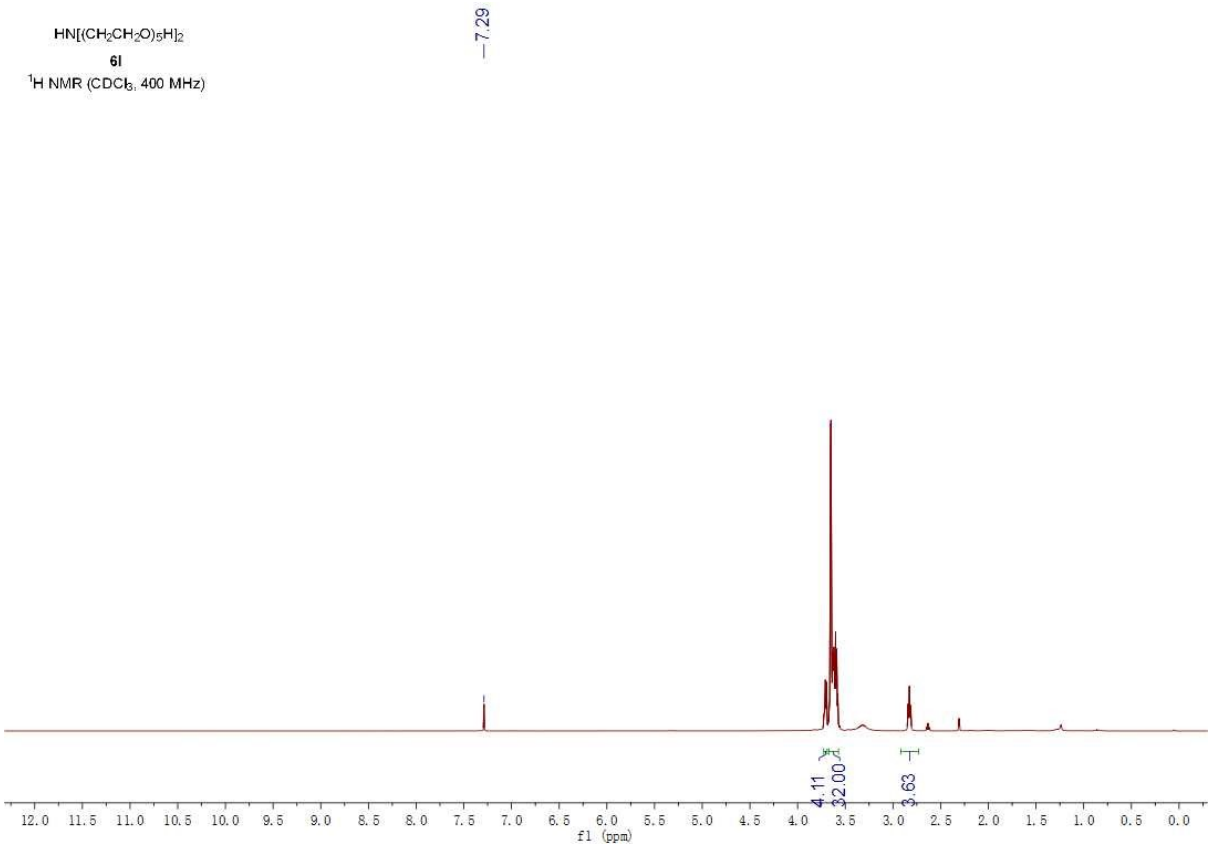
^{13}C NMR ($CDCl_3$, **101** MHz)



$HN[(CH_2CH_2O)_5H]_2$

6l

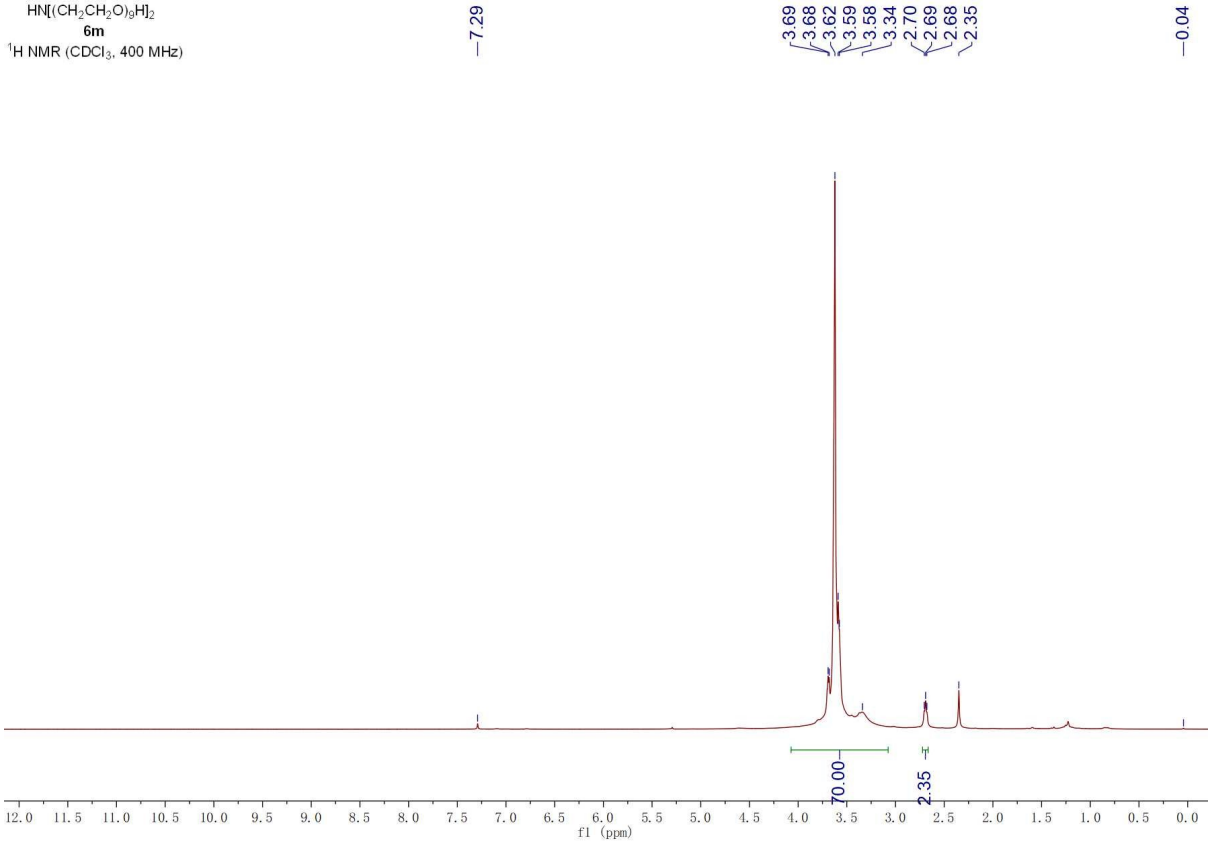
^1H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_9H]_2$

6m

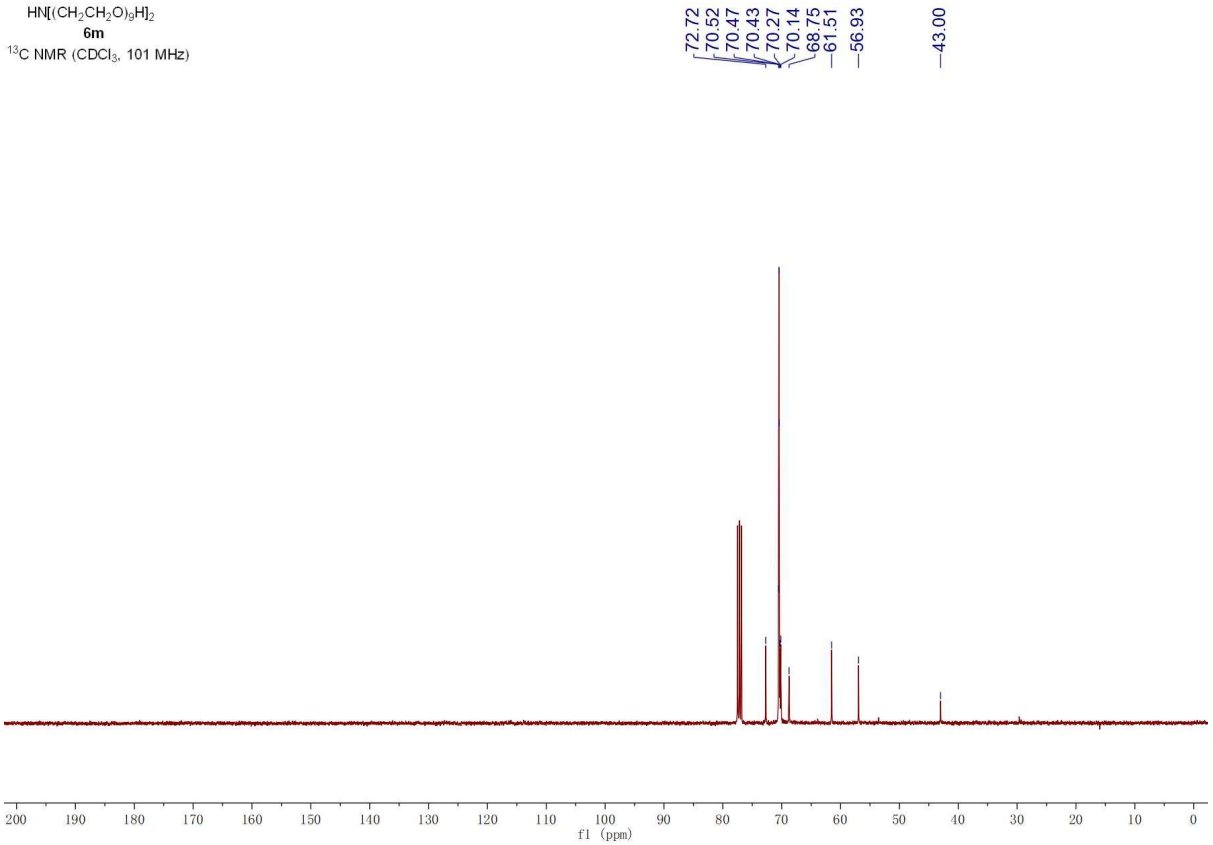
^1H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_9H]_2$

6m

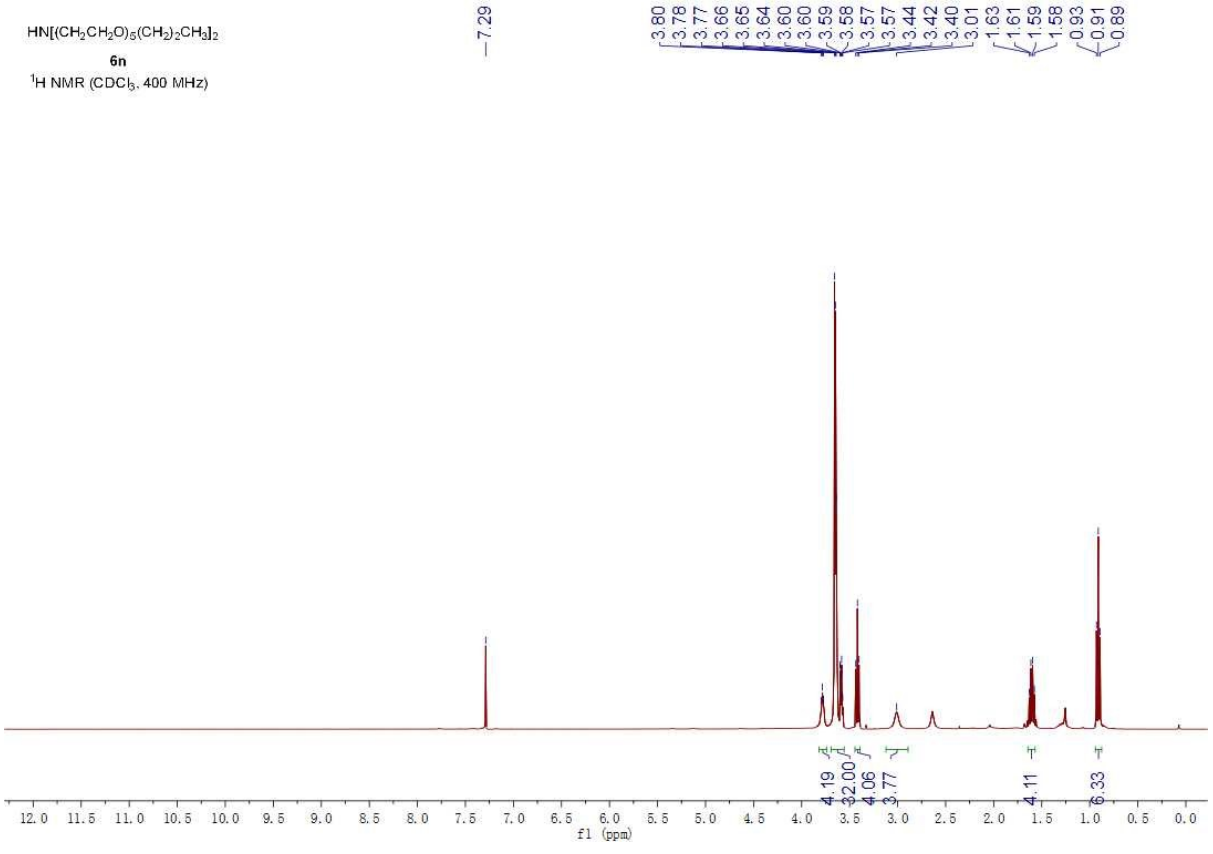
^{13}C NMR ($CDCl_3$, 101 MHz)



$HN[(CH_2CH_2O)_5(CH_2)_2CH_3]_2$

6n

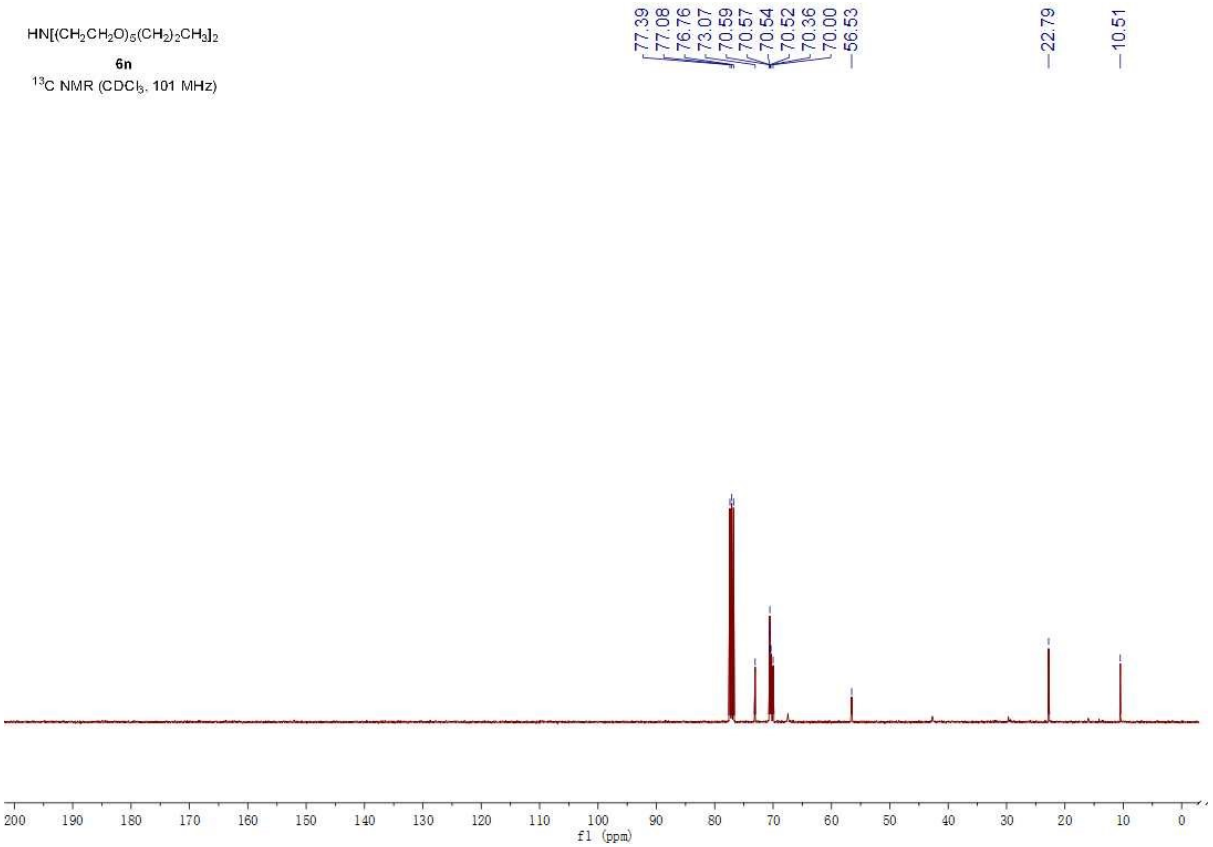
^{1}H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_5(CH_2)_2CH_3]_2$

6n

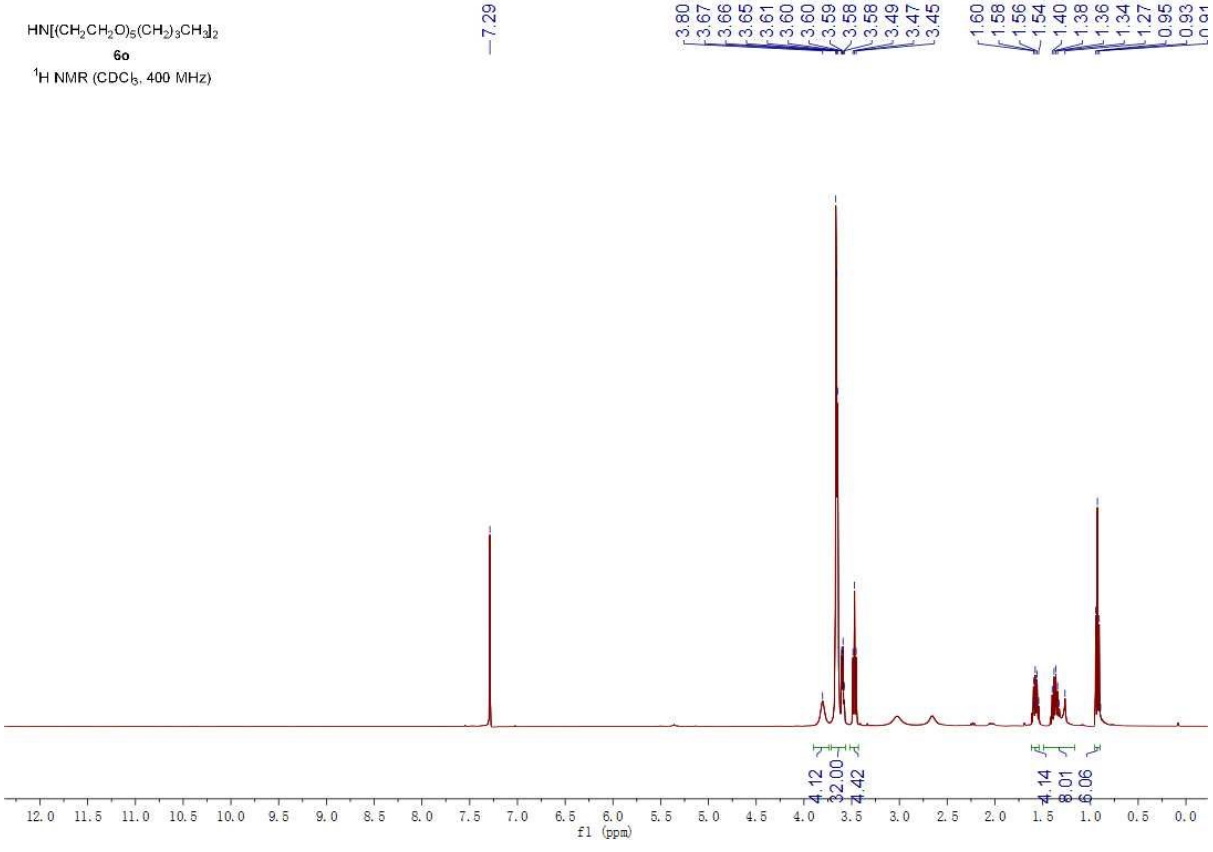
^{13}C NMR ($CDCl_3$, 101 MHz)



$HN[(CH_2CH_2O)_5(CH_2)_3CH_3]_2$

6o

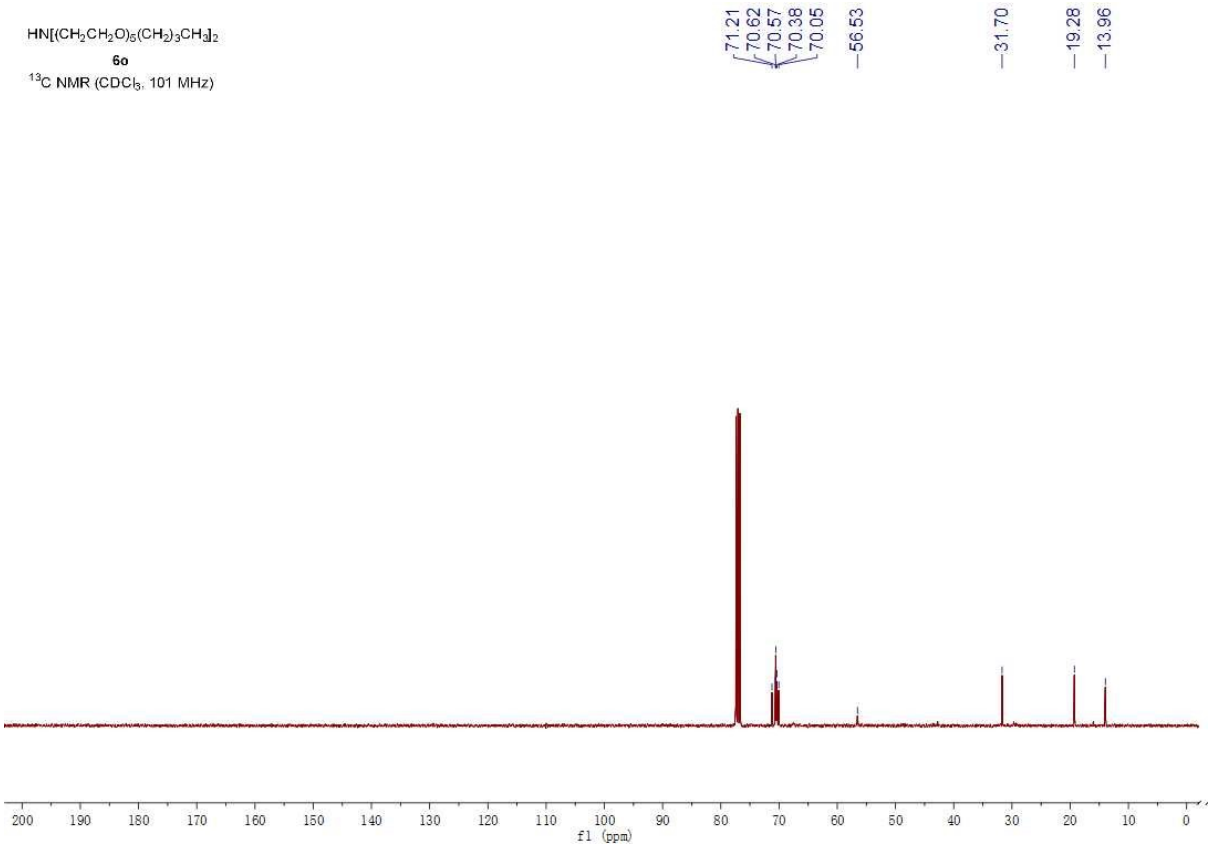
^{1}H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_5(CH_2)_3CH_3]_2$

6o

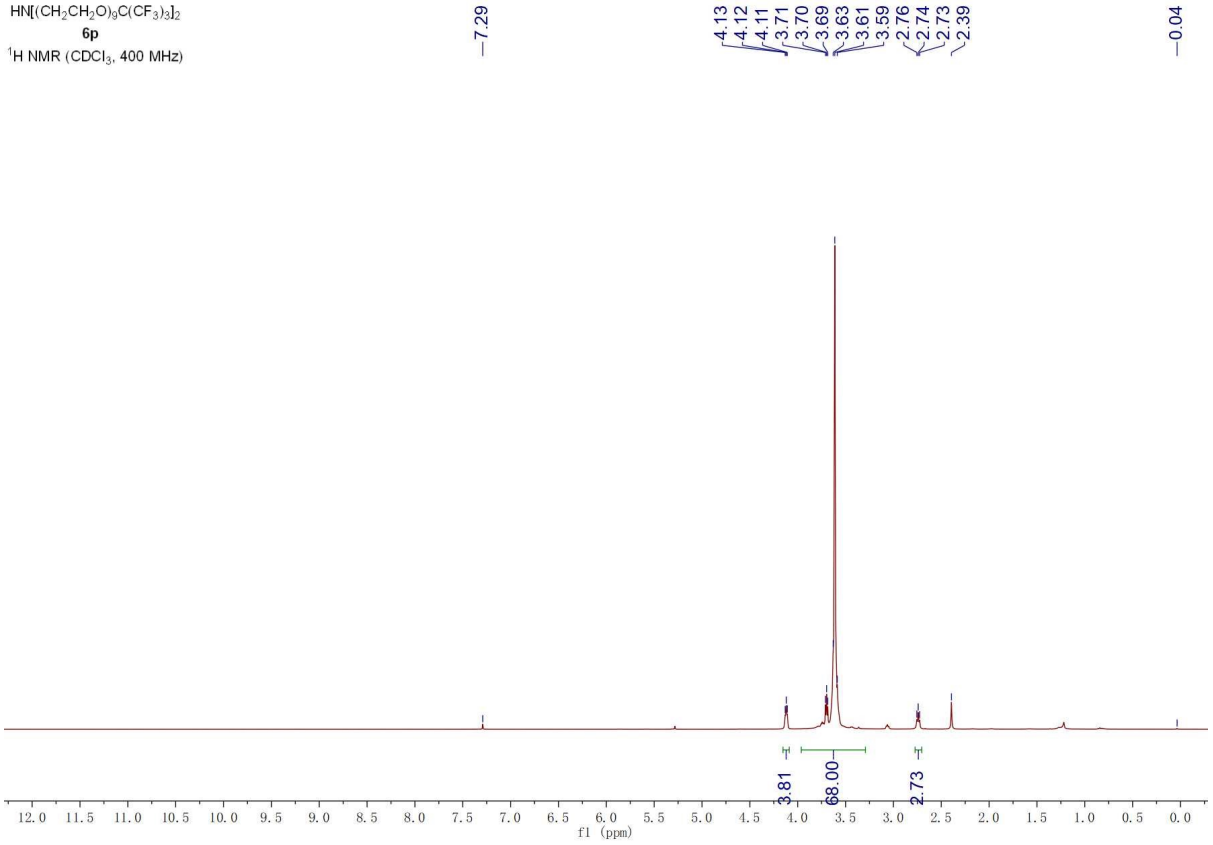
^{13}C NMR ($CDCl_3$, 101 MHz)



$HN[(CH_2CH_2O)_9C(CF_3)_3]_2$

6p

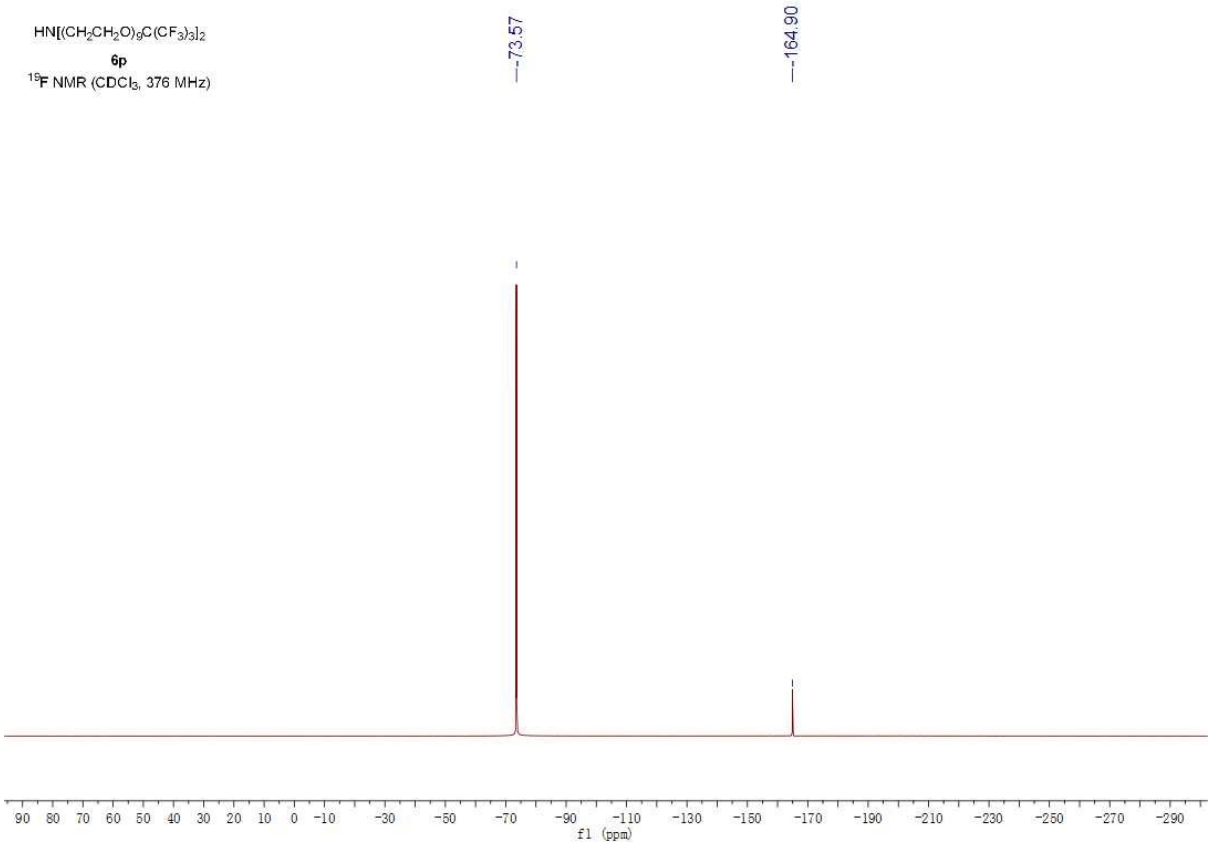
^{1}H NMR ($CDCl_3$, 400 MHz)



$HN[(CH_2CH_2O)_9C(CF_3)_3]_2$

6p

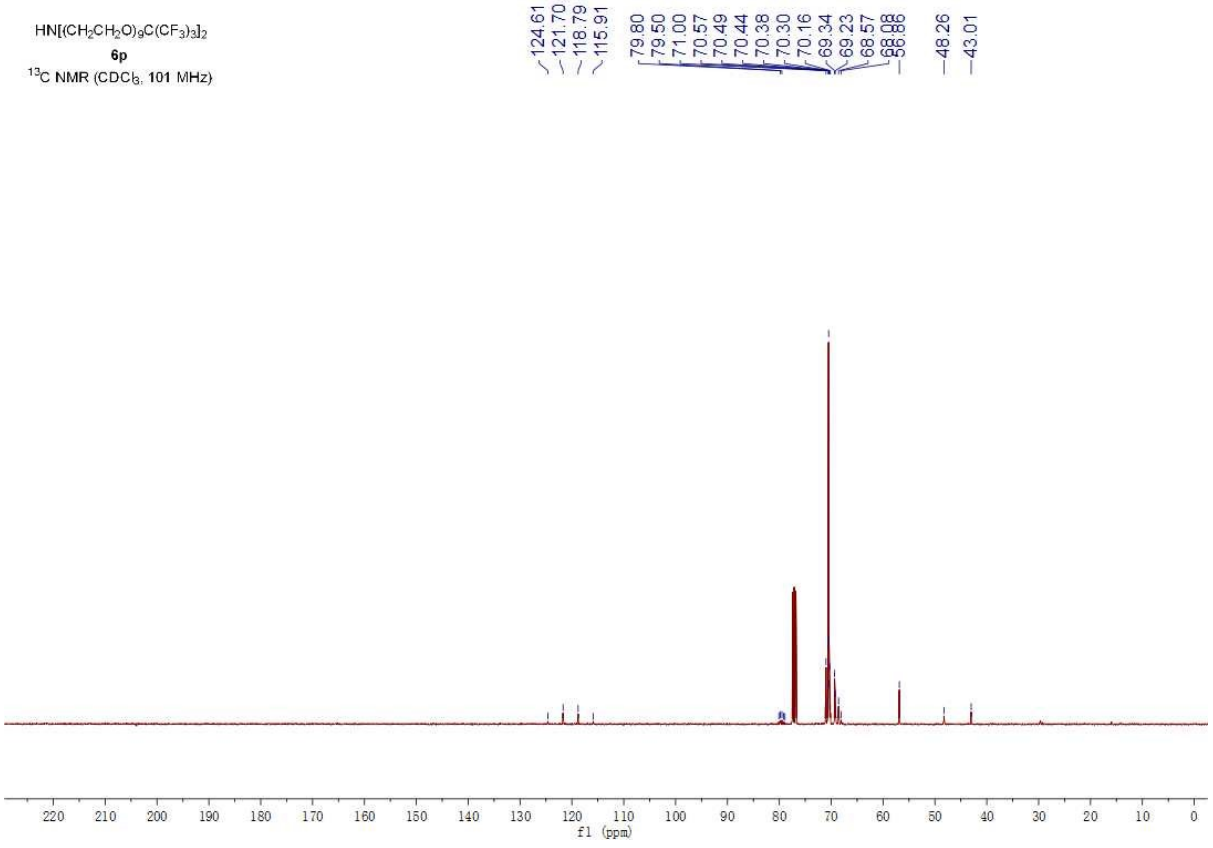
^{19}F NMR ($CDCl_3$, 376 MHz)

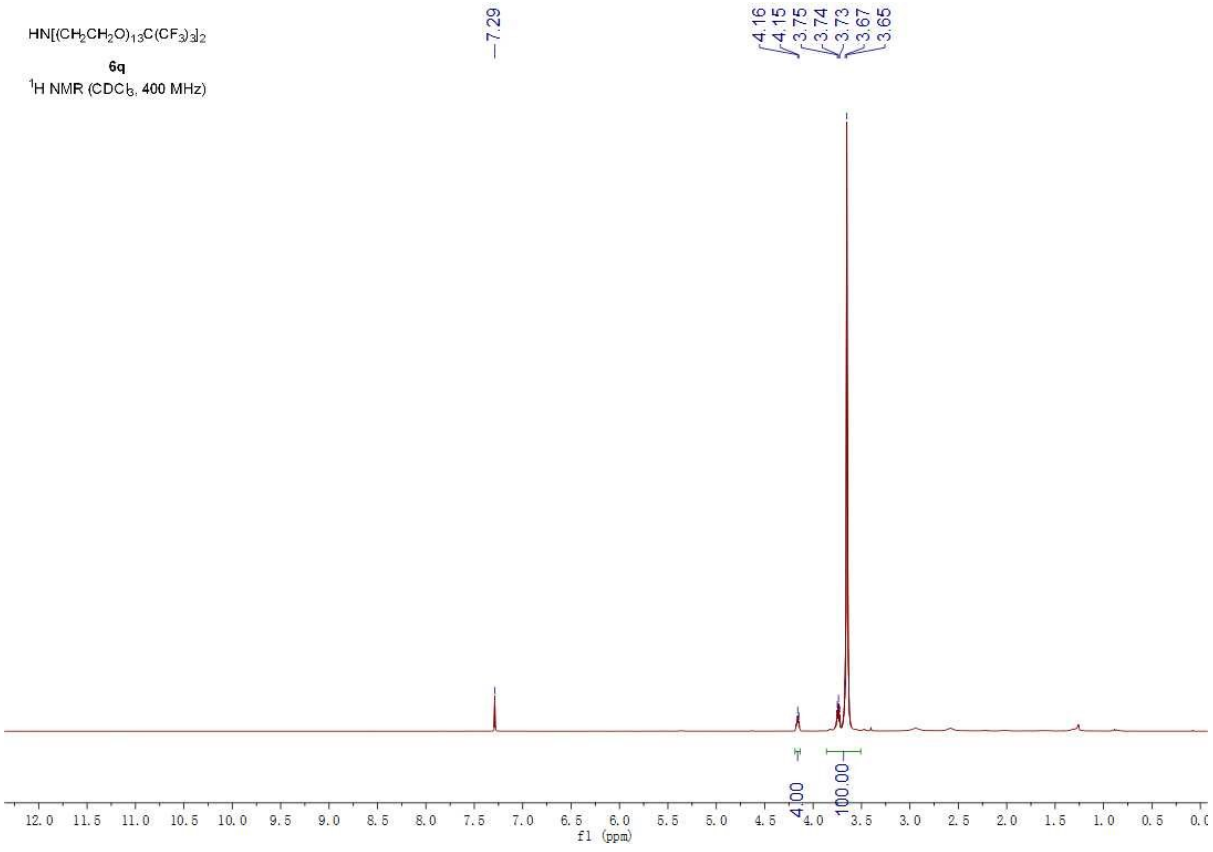


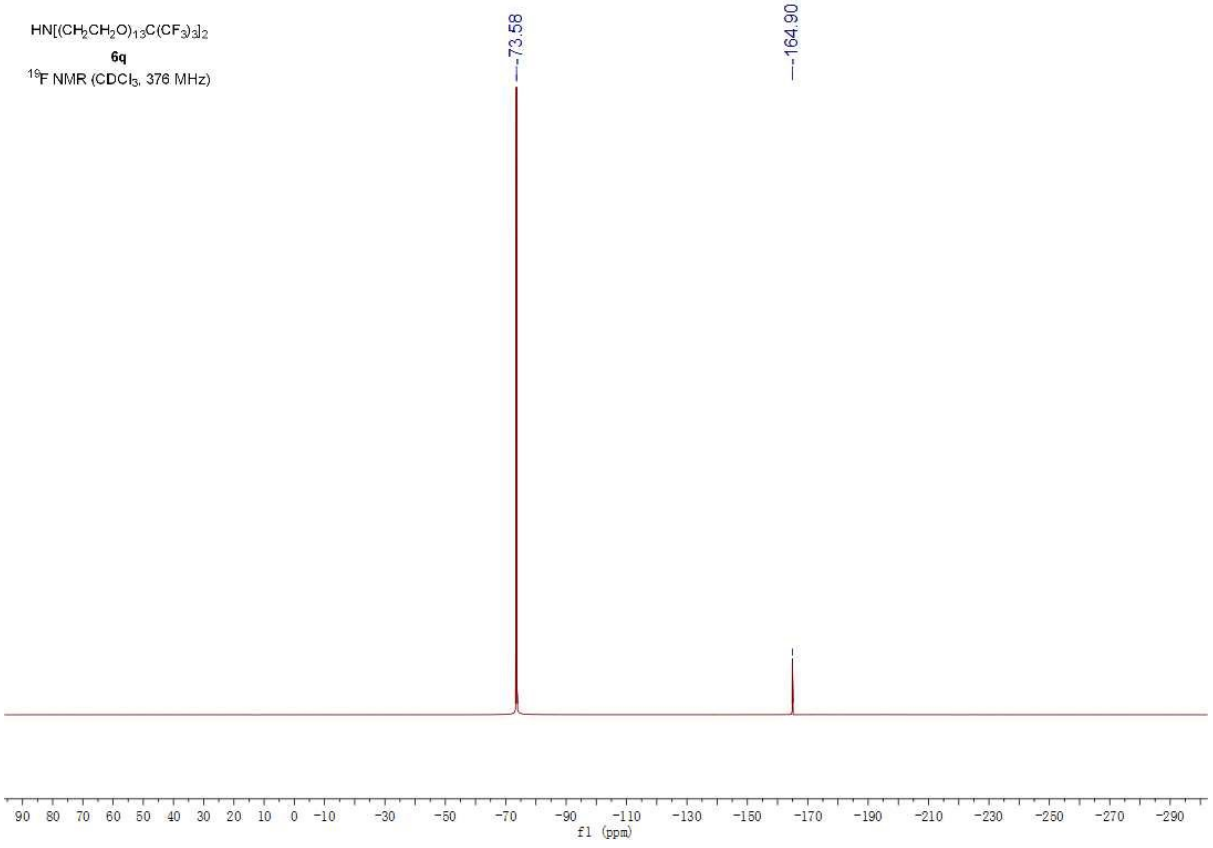
$HN[(CH_2CH_2O)_9C(CF_3)_3]_2$

6p

^{13}C NMR ($CDCl_3$, 101 MHz)



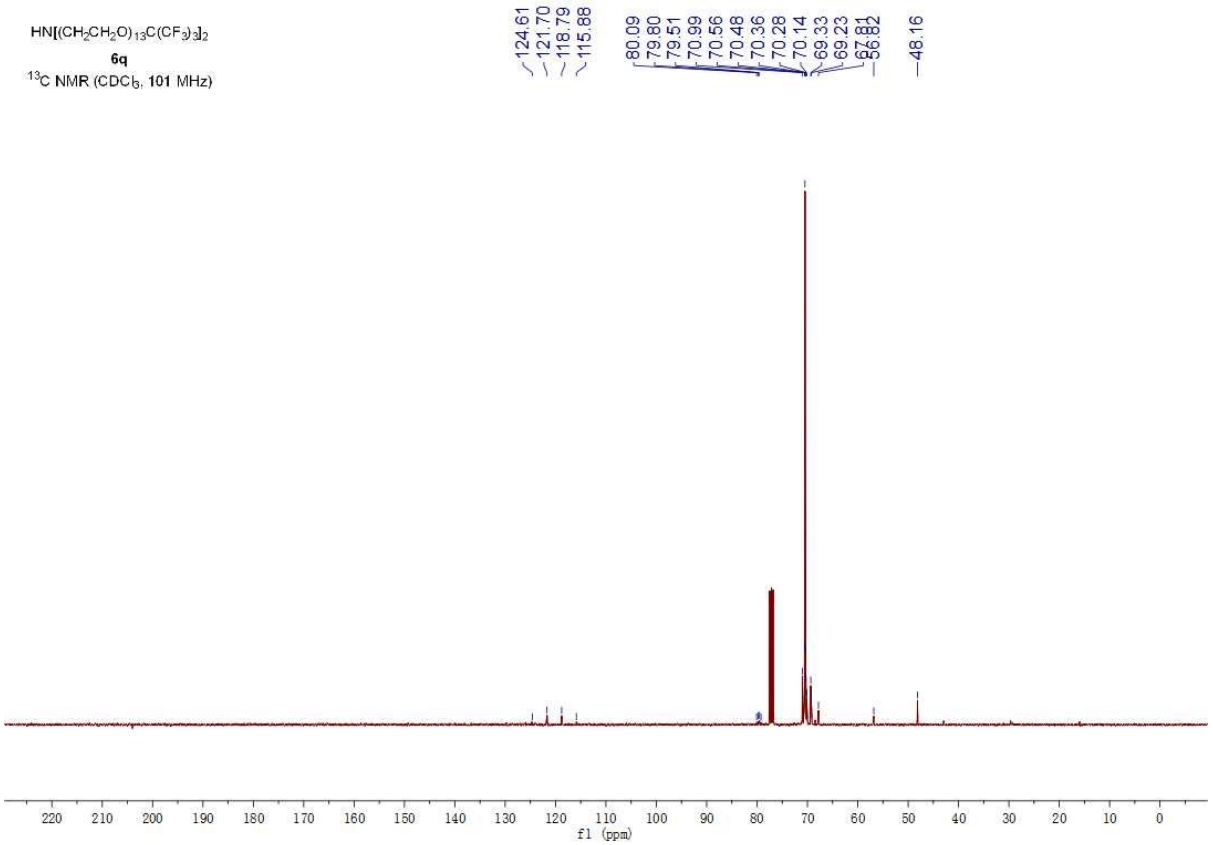

HN[(CH2CH2O)13C(CF3)3]2
6q
1H NMR (CDCl3, 400 MHz)
7.29
4.16
4.15
3.75
3.74
3.73
3.67
3.65
4.00
100.00
f1 (ppm)


HN[(CH2CH2O)13C(CF3)3]2
6q
19F NMR (CDCl3, 376 MHz)
-73.58
-164.90
f1 (ppm)

$HN[(CH_2CH_2O)_{13}C(CF_3)_3]_2$

6q

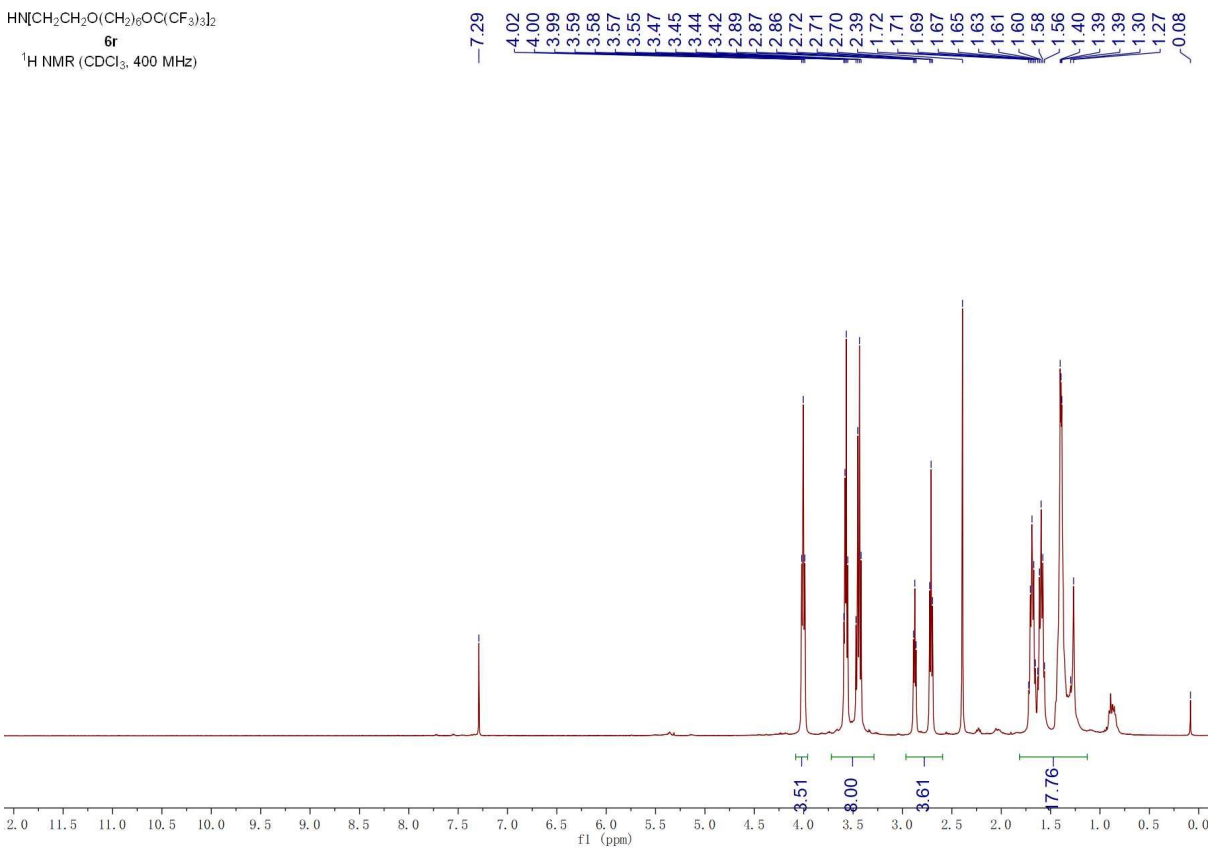
^{13}C NMR ($CDCl_3$, 101 MHz)



$HN[CH_2CH_2O(CH_2)_6OC(CF_3)_3]_2$

6r

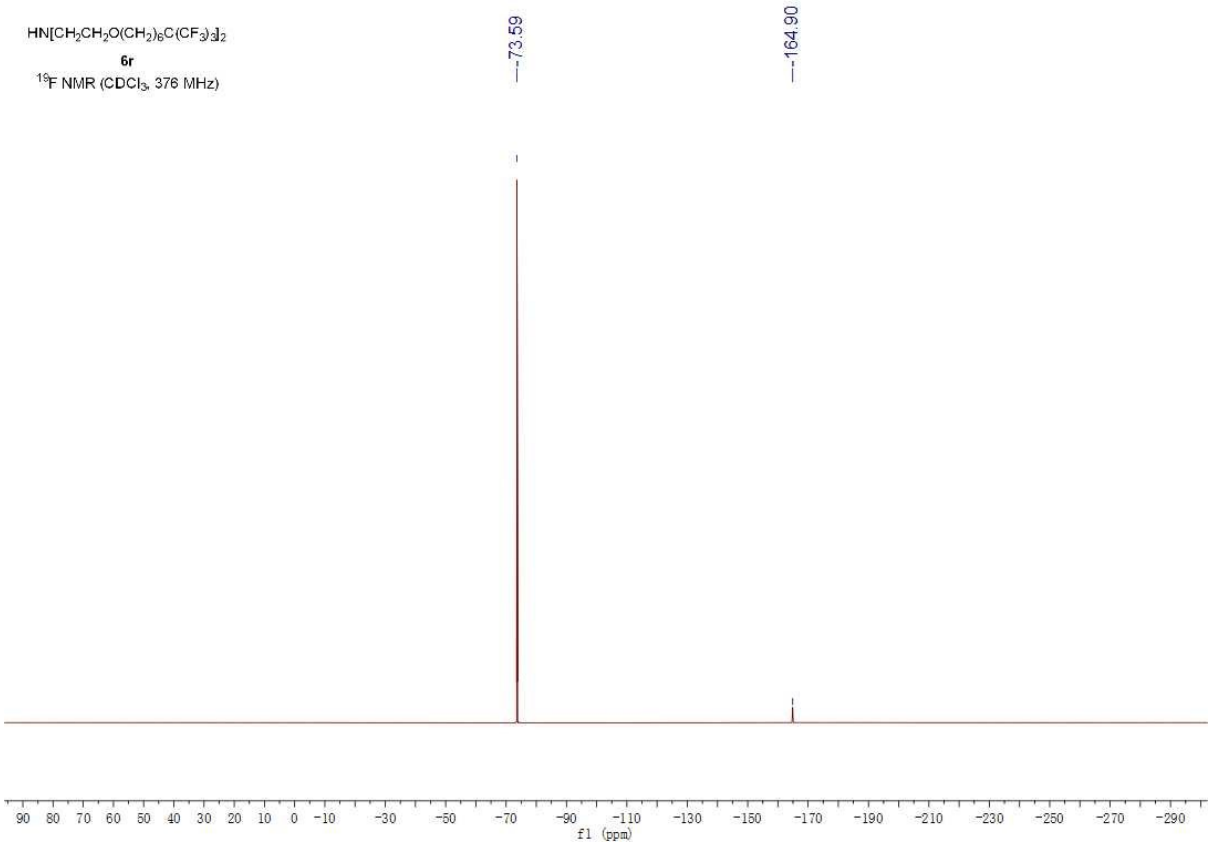
^1H NMR ($CDCl_3$, 400 MHz)



$HN[CH_2CH_2O(CH_2)_6C(CF_3)_3]_2$

6r

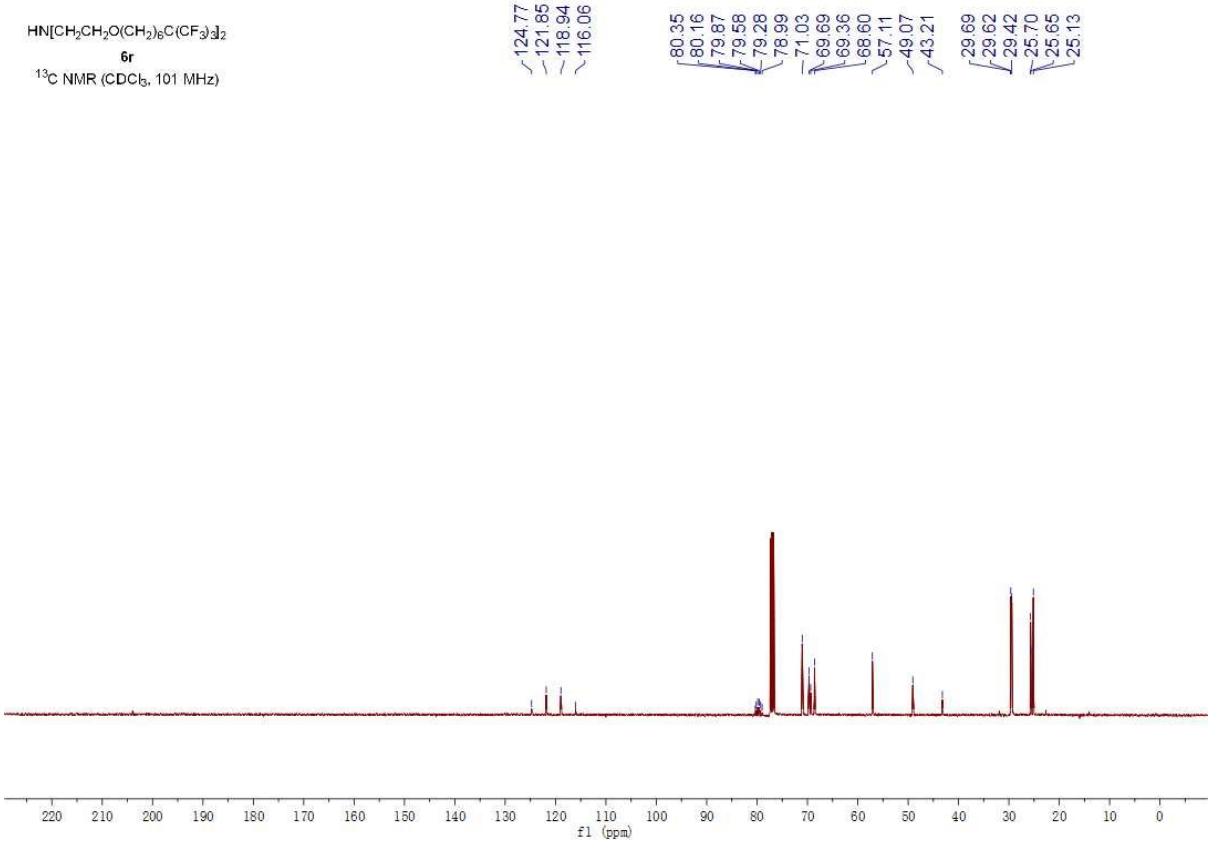
^{19}F NMR ($CDCl_3$, 376 MHz)

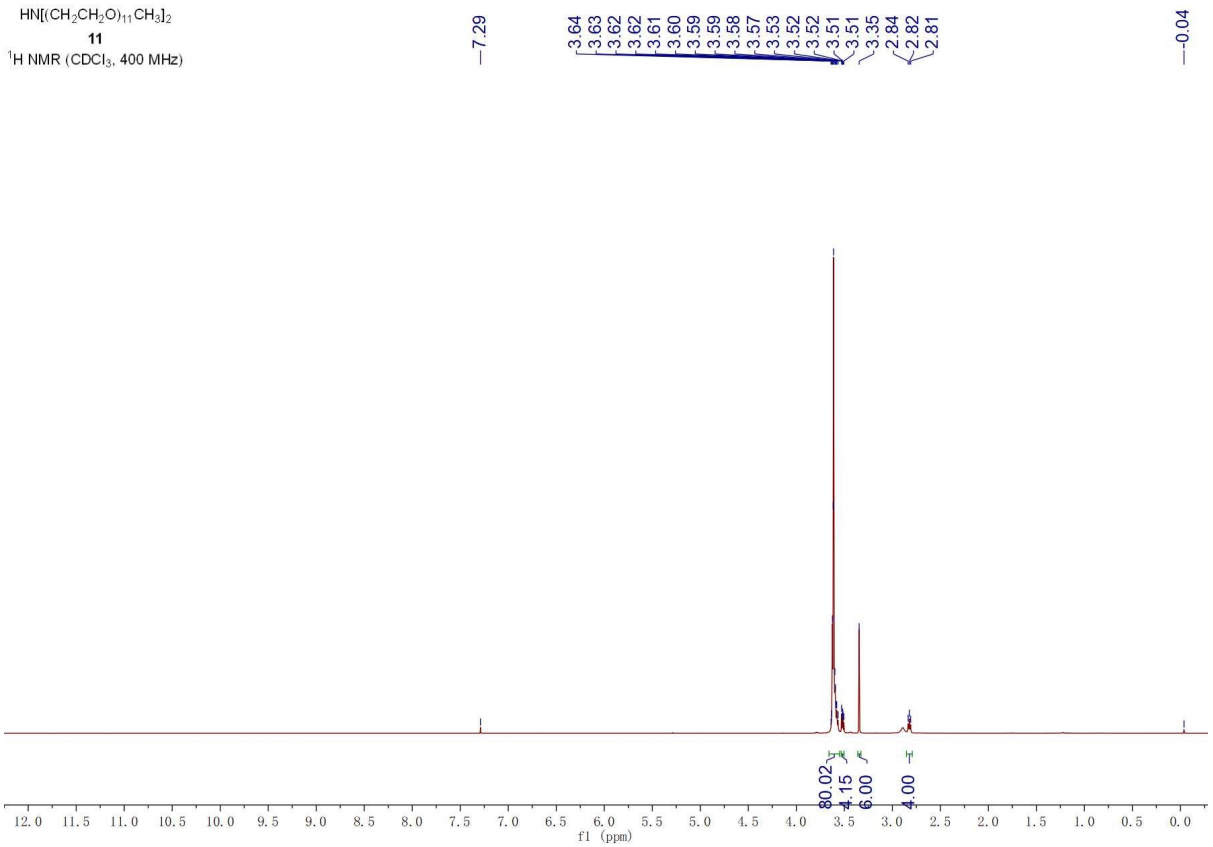


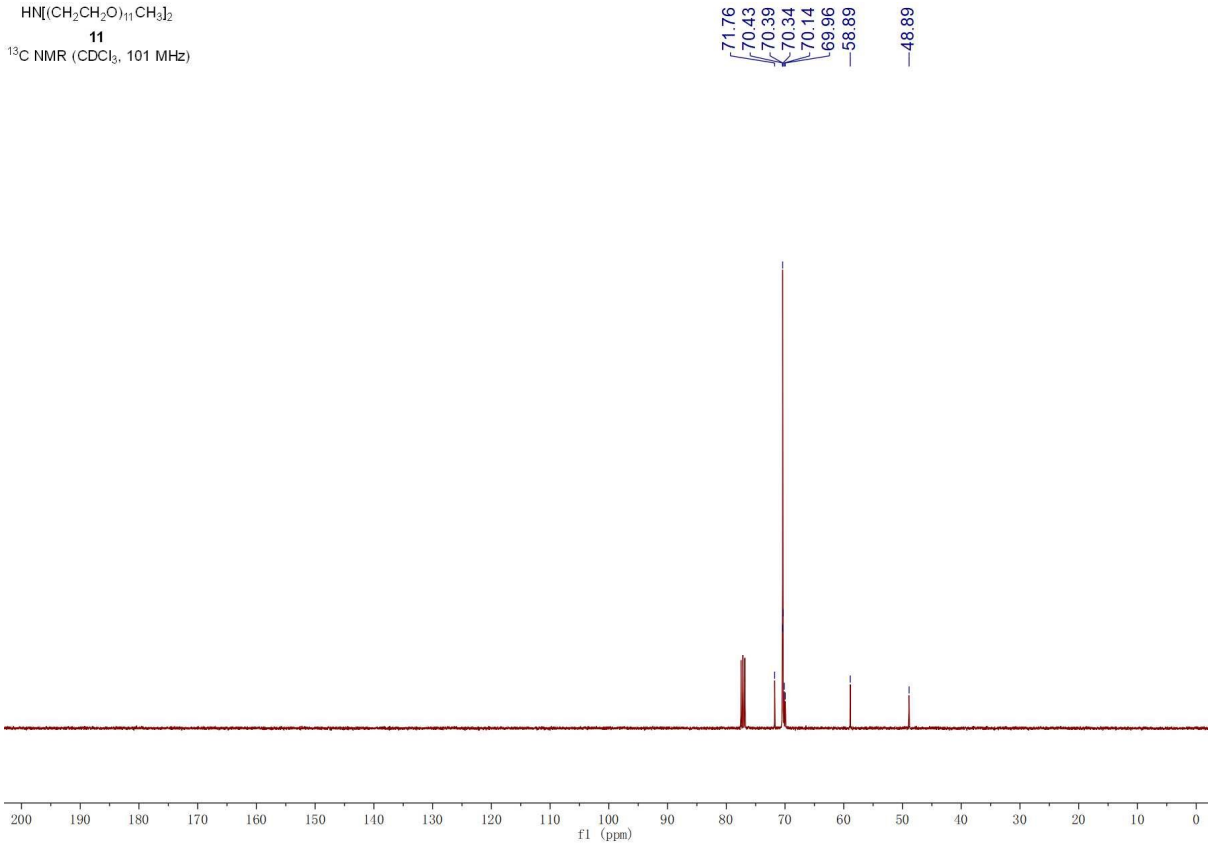
$HN[CH_2CH_2O(CH_2)_6C(CF_3)_3]_2$

6r

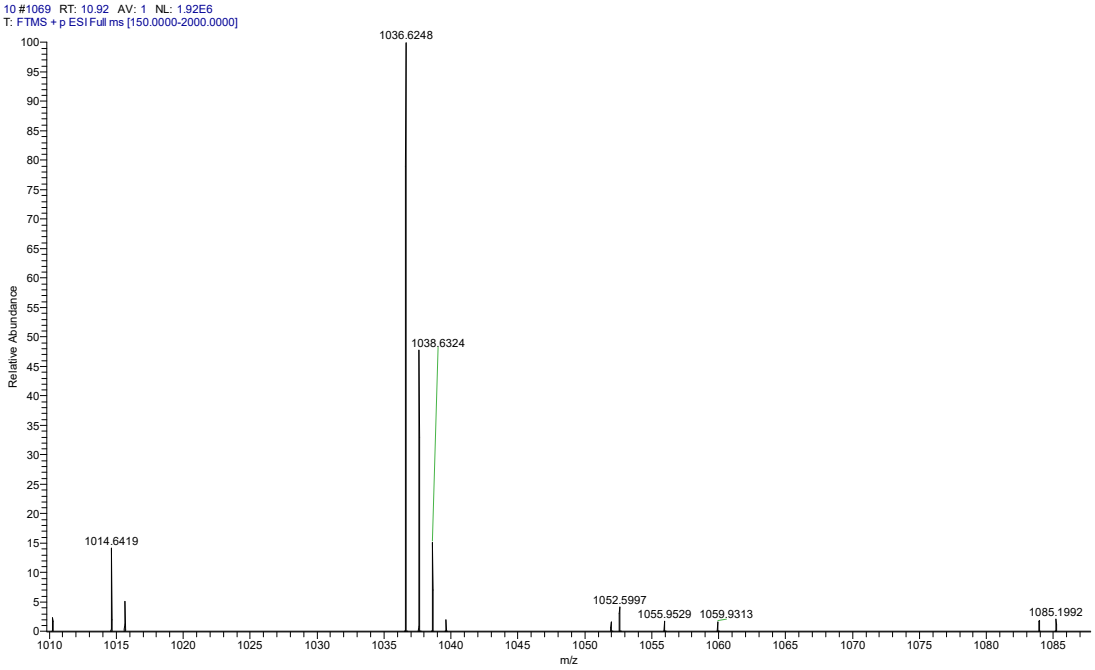
^{13}C NMR ($CDCl_3$, 101 MHz)







HRMS of compound **11**


10 #1069 RT: 10.92 AV: 1 NL: 1.92E6
T: FTMS + p ESI Full ms [150.0000-2000.0000]
1036.6248
1038.6324
1014.6419
1052.5997
1055.9529
1059.9313
1085.1992
Relative Abundance
m/z