Electronic Supplementary Information

Highly efficient SO_2 absorption/activation and subsequent utilization by polyethylene glycol-functionalized Lewis basic ionic liquids

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1. General experimental methods:

Caution

Experiments using compressed gases SO_2 or CO_2 are potentially hazardous and must only be carried out by using the appropriate equipment and under rigorous safety precautions.

Materials

All the reagents used in this work are purchased from Alfa Aesar-A Johnson Matthey Company or Tianjin Guangfu Fine Chemical Research Institute, and directly used without further purification. SO_2 and CO_2 with a purity of 99.99% is commercially available. Lewis basic ionic liquids are synthesized according to the reported method.

Experimental methods

^{1}H NMR spectra was recorded at Bruker 400 spectrometer in $CDCl_3$ or d_6-DMSO and $CDCl_3$ (7.26 ppm) or d_6-DMSO (2.50 ppm) was used as internal reference, ^{13}C NMR was recorded at 100.6 MHz in $CDCl_3$ or d_6-DMSO and $CDCl_3$ (77.00 ppm) or d_6-DMSO (39.43 ppm) was used as internal reference. ESI-MS were recorded on a Thermo Finnigan LCQ Advantage spectrometer in ESI mode with a spray voltage of 4.8 kV. GC-MS were measured on a Finnigan HP G1800 A. GC analyses were performed on a Shimadzu GC-2014 equipped with a capillary column (RTX-WAX, 30 m * 0.25 μm) using a flame ionization detector. *In situ* FTIR was collected on a Mettler Toledo React IR ic10, Silica ATR probe, using ic IR analysis system. The probe is placed in the middle of the absorption mixture, which is constantly stirred by magnetic whisk, and the spectra are collected *in situ* during SO_2 absorption. Column chromatography was performed by using silica gel 200-300 mesh with CH_2Cl_2/ethyl acetate/petroleum as eluent. Melting points were measured on an X_4 apparatus and uncorrected.

General procedure for absorption and desorption of SO_2

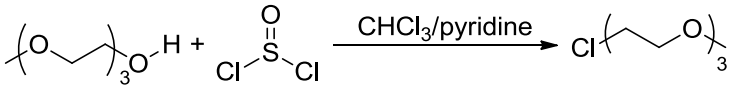
In a typical procedure, SO_2 capture was carried out in a 10 mL Schlenk flask. The absorbents were charged into the reactor at room temperature. Then, the air in the flask was replaced by SO_2 and a needle was used for SO_2 bubbling, which was inserted in the bottom of the flask. The absorption reaction was conducted at 25 ^{o}C with a SO_2 bubbling rate of 0.1 L/min. The amount of SO_2 absorbed was determined by an Analytical Balance within an accuracy of $\pm$0.0001 g every five minutes. During the absorption of SO_2 under reduced pressure, SO_2 is diluted with N2 in order to reduce the partial pressure of SO_2 passing through the system. The SO_2 partial pressure is controlled by changing the volume fraction of SO_2. In a typical desorption of SO_2, N_2 of atmospheric pressure is bubbled trough absorption system at 25 ^{o}C using the same equipment and procedure as SO_2 capture. Absorption/desorption is determined by several cycles of repeated experiments.

General procedure for the reaction of epoxides and SO_2

A typical procedure is as follows: PEG_{150}MeDABCOBr (3 mol%), biphenyl (0.05 g, internal standard of GC) and propylene oxide (10 mmol) were added successively into a glass tube. The suspension was cooled to -60 ^{o}C (liquid nitrogen/ethanol) and SO_2 (10 mmol) was introduced into the vessel. The glass tube was placed in a stainless steel autoclave (25 mL inner volume). The reaction mixture was heated at 120 ^{o}C with stirring for 3 h. After reaction, the autoclave was allowed to be cooled at 0 ^{o}C. The product yields for catalyst and reaction parameters screening were determined by GC with a flame ionization detector and were further identified using GC-MS by comparing retention times and fragmentation patterns with authentic samples. For substrate scope, the desired products are purified column chromatography on silica gel (200-300 mesh, eluting with petroleum ether/ethyl acetate or petroleum ether/dichloromethane), and further identified by GC-MS and NMR, which are consistent with those reported in the literature[1] and in good agreement with the assigned structures.

2. Synthesis and characterization of DABCO-derived Lewis basic ionic liquids

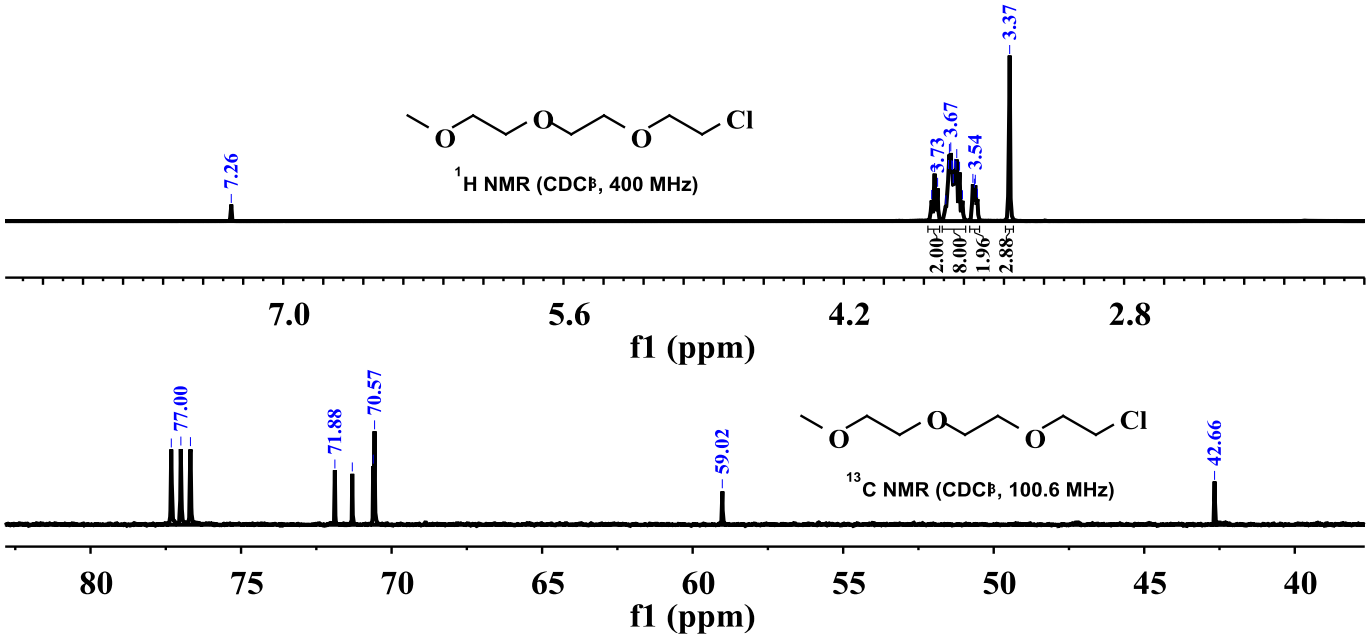
PEG_{150}MeCl[2]



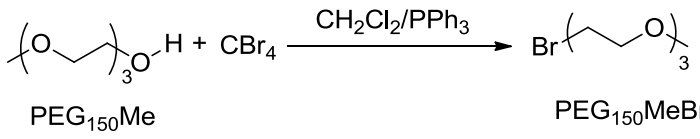
PEG_{150}Me PEG_{150}MeCl

A solution of thionyl chloride (0.45 mol) in $CHCl_3$ (90 mL) is added slowly over 60 min to a stirred solution of triethylene glycol monomethyl ether (0.3 mol) and pyridine (0.3 mol) in $CHCl_3$ (200 mL), followed by refluxing the above reaction mixture at 100 ^{o}C for 4 h, and then yellow is obtained, which is washed with water (4 * 125 mL), dried with $MgSO_4$, and concentrated under reduced pressure at 60 ^{o}C to remove $CHCl_3$. The crude product is purified under reduced pressure to give ClPEG_{150}Me as a light yellow liquid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.75 (t, ^3J = 6 Hz, 2H), 3.61-3.69 (m, 8H), 3.53-3.56 (m, 2H), 3.37 (s, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 71.8, 71.3, 70.6, 70.5, 59.0, 42.7; GC-MS: m/z (%):183.02 (100), 185.03 (33) [M^+], 151.08 (26), 153.07 (8) [M^+-CH_3O], 103.13 (61) [M^+-C_2H_4OCl].

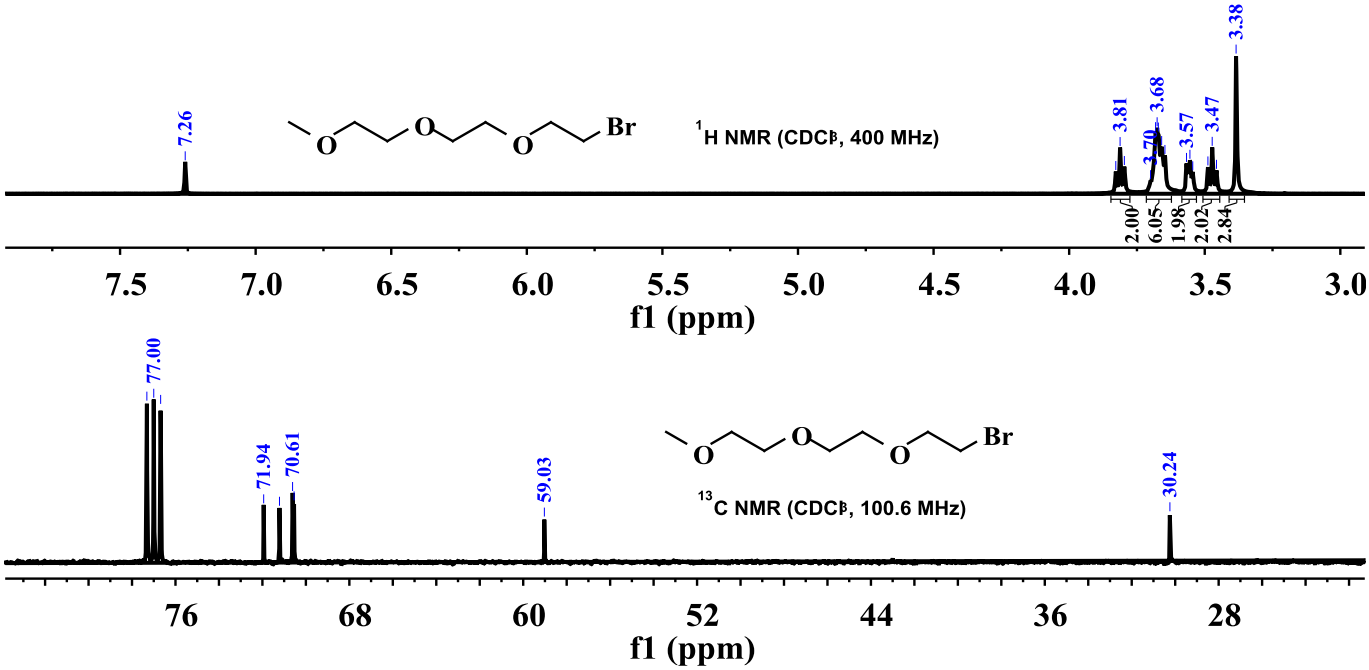


Synthesis of $PEG_{150}MeBr$[3]

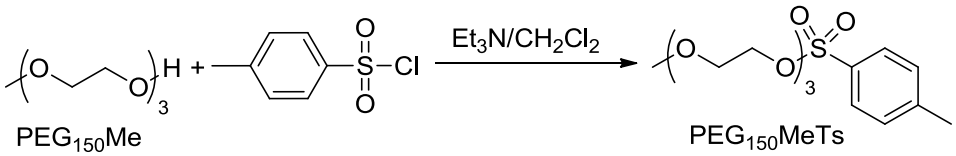


Carbon tetrabromide (24 mmol) was added to 100 mL CH_2Cl_2 solution of triethylene glycol monomethyl ether (20 mmol) under argon atmosphere. Reaction mixture was cooled to 0 ^oC and PPh_3 (30 mmol) in CH_2Cl_2 (20 mL) was added dropwise to it. After stirring for 12 h, the solvent from the reaction mixture was evaporated out. Diethyl ether (50 mL) was added, stirred for 5 min and filtered. The same process (addition of diethyl ether, filtration and evaporation of solvent) was repeated twice. The crude product was purified under reduced pressure to give $PEG_{150}MeBr$ as a colorless liquid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.83 (t, ^3J = 6.4 Hz, 2H), 3.65-3.68 (m, 6H), 3.55 (t, ^3J = 4.8 Hz, 2H), 3.47 (t, ^3J = 6.4 Hz, 2H), 3.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 71.9, 71.2, 70.6, 70.5, 59.0, 30.2; ESI-MS calcd for $C_7H_{15}BrO_3$ 226.02, found 227.0, 229.0 $[M+H]^+$, 249.1, 251.1 $[M+Na]^+$.

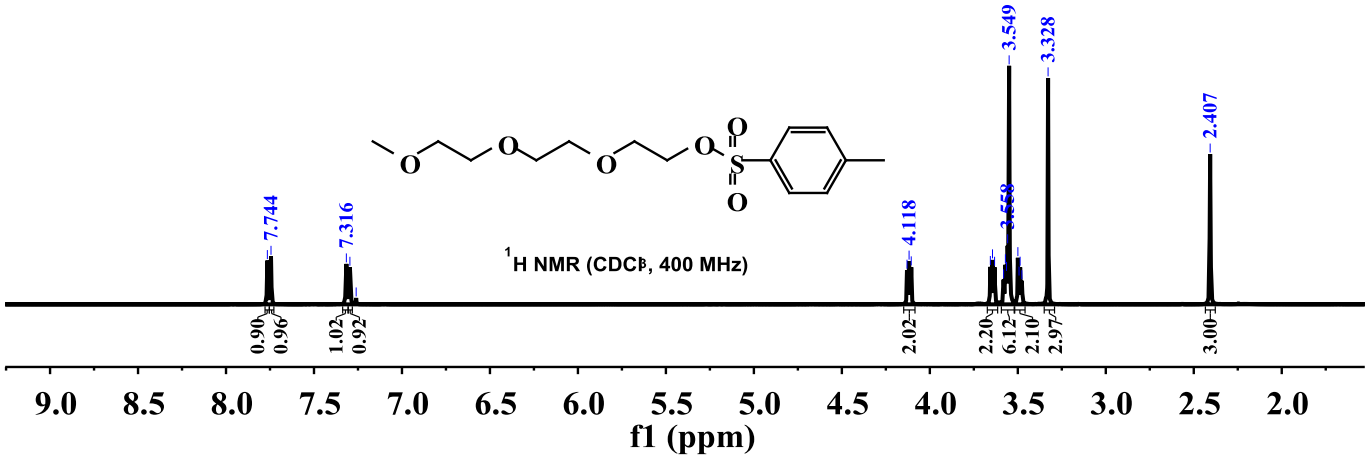


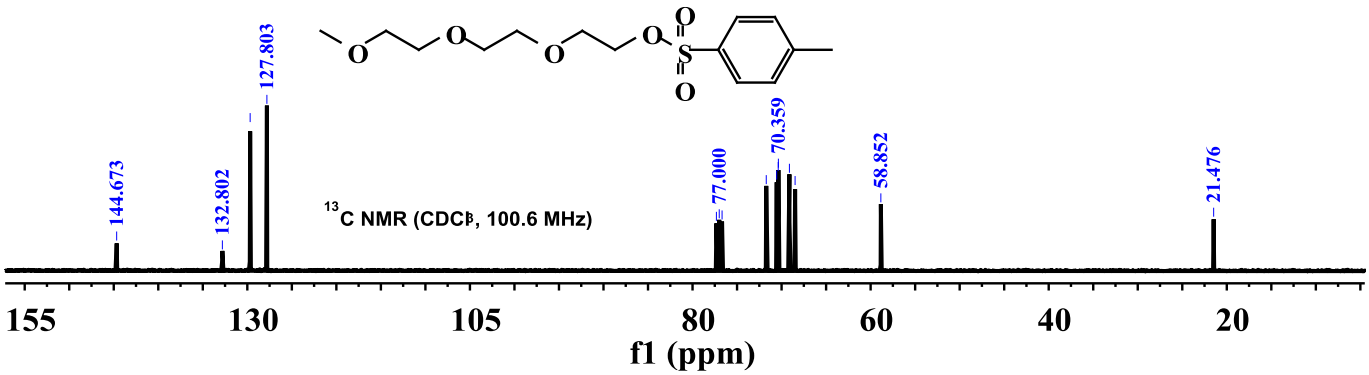
Synthesis of $PEG_{150}MeI$[4, 5]

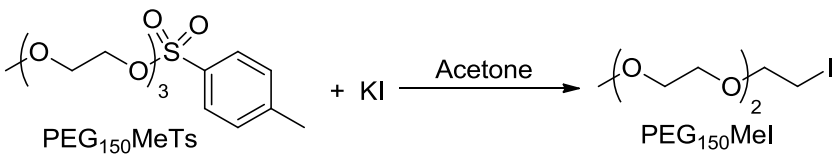


Triethylene glycol monomethyl ether (20 mmol) and triethylamine (30 mmol) were dissolved in dry dichloromethane (50 mL) and the solution was cooled to 0 °C. A solution of tosyl chloride (20 mmol) in dichloromethane (20 mL) was then added dropwise. The mixture was stirred for 2 h at 0 °C and 8 h at room temperature. After addition of diethyl ether (50 mL) the resulting precipitate was filtered and the filtrate concentrated to dryness. The crude product was purified by flash column chromatography (petroleum ether : ethyl acetate 2 : 1) to afford $PEG_{150}MeTs$ as a light yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz,) δ 7.76 (s, 1H), 7.74 (s, 1H), 7.32 (s, 1H), 7.30 (s, 1H), 4.12 (t, ^{3}J = 4.8 Hz, 2H), 3.64 (t, ^{3}J = 4.8 Hz, 2H), 3.55-3.58 (m, 6H), 3.48-3.50 (m, 2H), 3.33 (s, 3H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 144.7, 132.8, 129.7, 127.8, 71.7, 70.5, 70.37, 70.36, 69.1, 68.5, 58.9, 21.5.

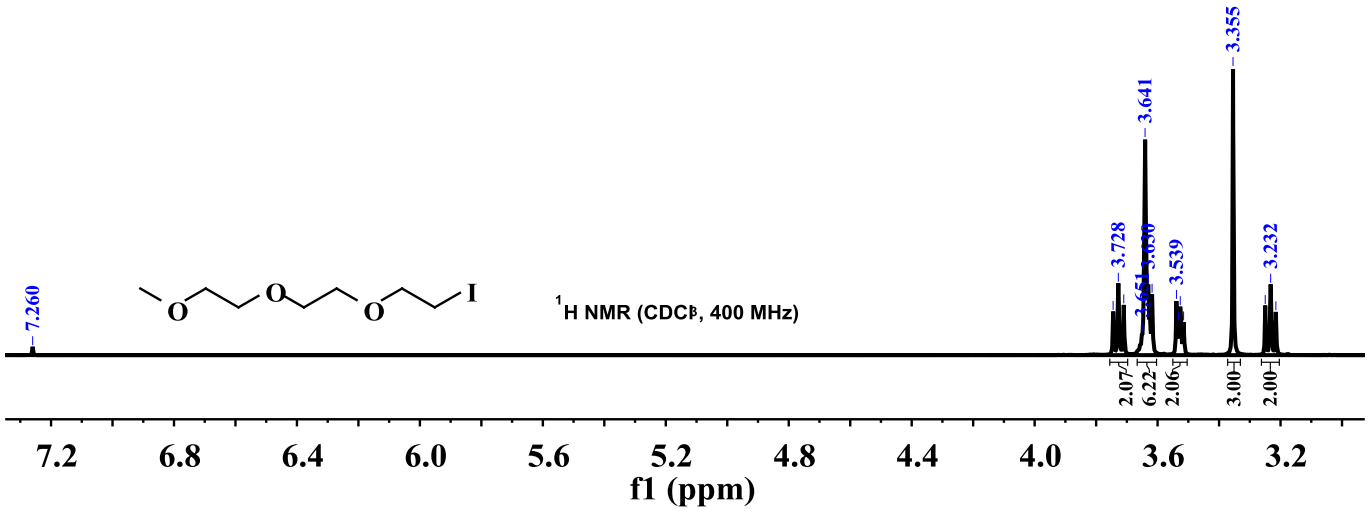


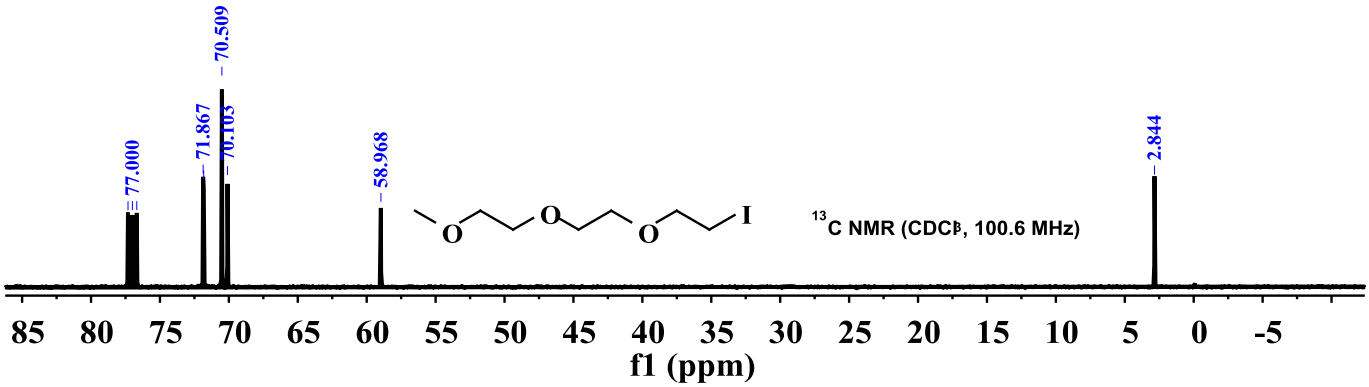




$PEG_{150}MeTs$ (13 mmol) was dissolved in acetone (50 mL), and KI (25 mmol) was added to the solution. The mixture was stirred at 40 °C for 4 days. The solvent was removed under reduced pressure, and water (10 mL) was added to the residue. The mixture was extracted with CH_2Cl_2 (4 × 50 mL), and then the combined extracts were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was purified under reduced pressure to give $PEG_{150}MeI$ as a colorless liquid.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.73 (t, ^{3}J = 6.8 Hz, 2H), 3.62-3.65 (m, 6H), 3.51-3.54 (m, 2H), 3.35 (s, 3H), 3.23 (t, ^{3}J = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 71.9, 71.8, 70.5, 70.1, 59.0, 2.8.



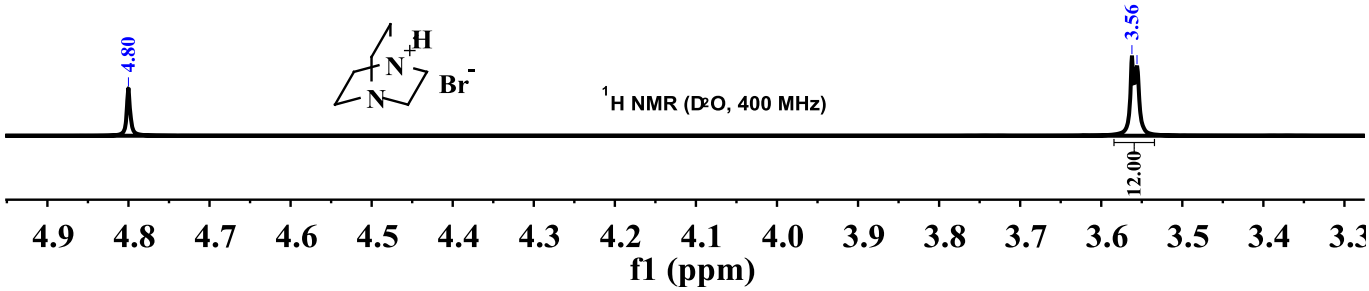


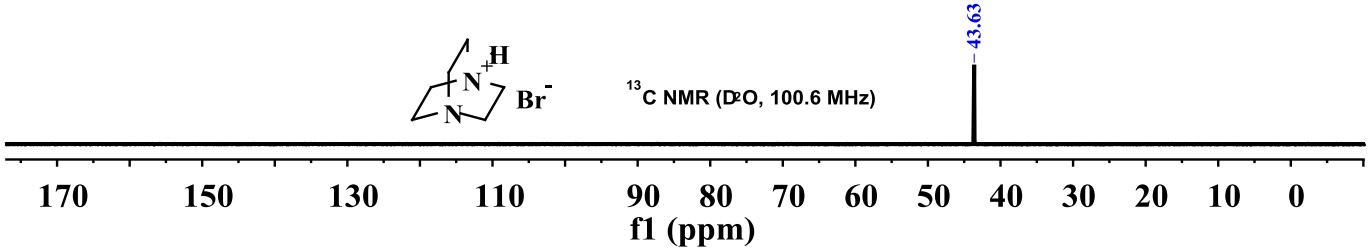
DABCO-based ionic liquids

HDABCOBr[6]

To a 50 mL three-neched flask was added DABCO (10 mmol). A solution of HBr(10 mmol) was then added dropwise at the temperature of < 5 ^{o}C cooled by ice bath. After addition, the ice bath was removed and the reaction mixture was stirred for further 10 h and then the solvent was evaporated. The remaining solid was dried under vacuum at 60 ^{o}C for 24 h after washing by ethyl ether for three times to afford HDABCOBr.

^{1}H NMR (D_2O, 400 MHz) δ 3.56 (m, 12 H); ^{13}C NMR (D_2O, 100.6 MHz) δ 43.6; ESI-MS calcd for $C_6H_{13}BrN_2$ 192.03, found 113.20 [M-Br]$^+$.





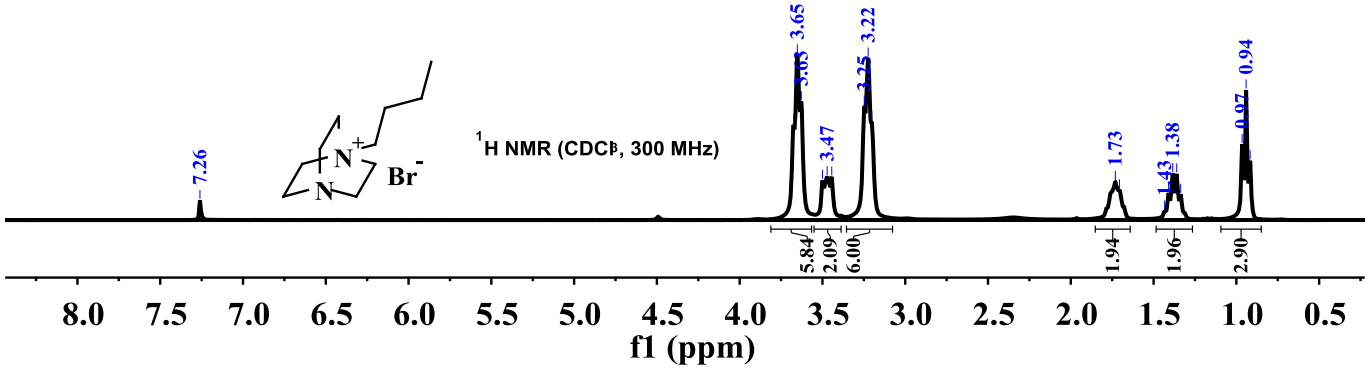
C_nDABCOBr (n = 4, 8, 12), PEG_{150}MeDABCOX (X = Cl, Br, I), $C_8(DABCO)_2Br_2$[7]

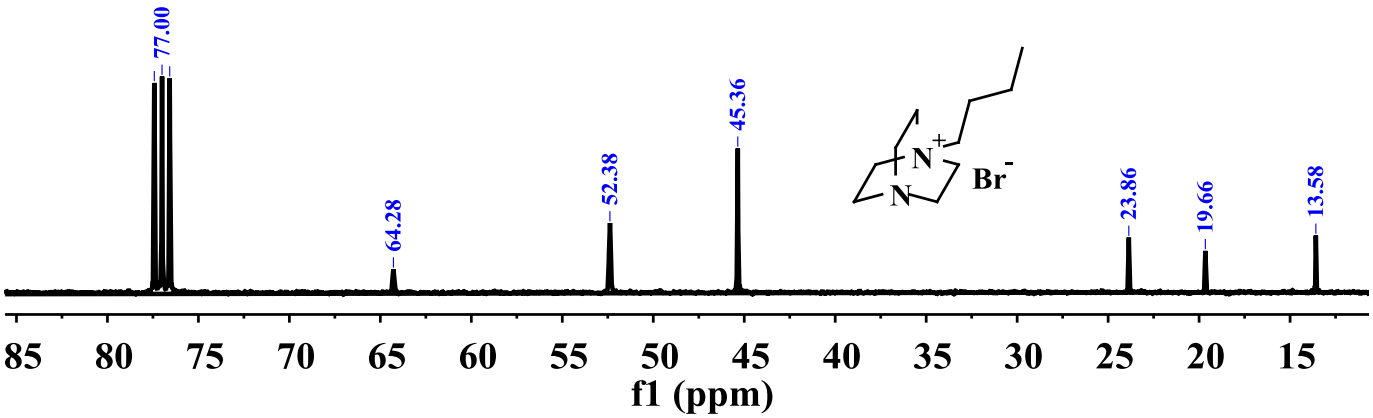
A solution of freshly sublimed DABCO (5 mmol) in MeCN (5 mL) was prepared at r.t. under Ar in a flame dried round-bottomed flask equipped with a reflux condenser. The resulting clear, colorless solution was treated with 1-bromobutane, 1-bromoctane, 1-bromododecane,

$PEG_{150}MeCl$, $PEG_{150}MeBr$, $PEG_{150}MeI$ (2.5 mmol), for $C_4DABCOBr$, $C_8DABCOBr$, $C_{12}DABCOBr$, $PEG_{150}MeDABCOCl$, $PEG_{150}MeDABCOBr$, $PEG_{150}MeDABCOI$ and 1,8-dibromoctane, $BrPEG_{150}Br$ (1.25 mmol) for $C_8(DABCO)_2Br_2$, respectively, added dropwise via syringe, and the reaction mixture was heated to 80 °C (oil bath temperature) and stirred for 22 h. After being cooled to r.t., the reaction mixture was transferred via cannula into 20 mL of Et_2O, yielding a white slurry which eventually separated into two layers. The whole was transferred to a separatory funnel, and the bottom layer was collected, washed repeatedly with Et_2O and dried under high vacuum at 60 °C for 16 h.

$C_4DABCOBr$

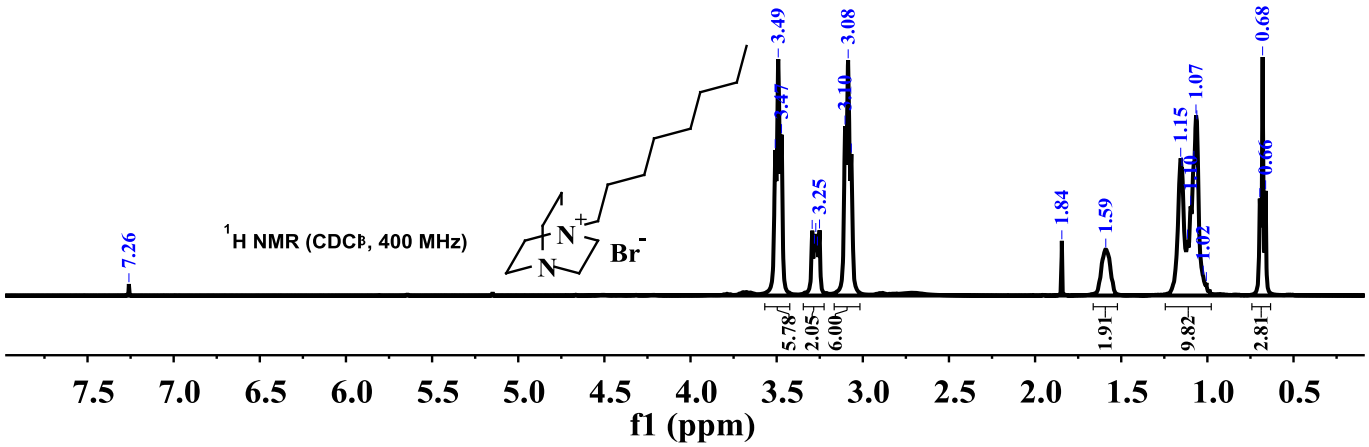
White glassy solid; Mp 36-37 °C; ^1H NMR ($CDCl_3$, 300 MHz) δ 3.65 (t, ^3J = 7.2 Hz, 6 H), 3.47 (t, ^3J = 8.4 Hz, 2 H), 3.22 (t, ^3J = 6.6 Hz, 6 H), 1.70-1.75 (m, 2 H), 1.36-1.40 (m, 2 H), 0.94 (t, ^3J = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 64.3, 52.4, 45.4, 23.9, 19.7, 13.6; ESI-MS calcd for $C_{10}H_{21}N_2Br$ 249.19, found 169.38 $[M-Br]^+$, 79.02 $[M-C_{10}H_{21}N_2]^-$

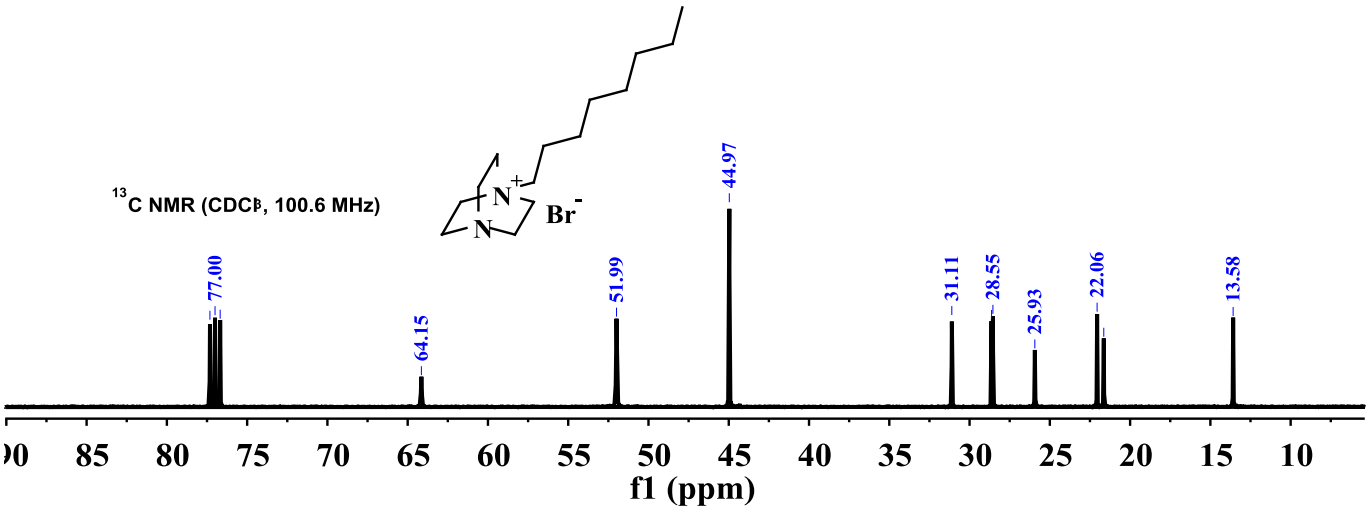




$C_8DABCOBr$

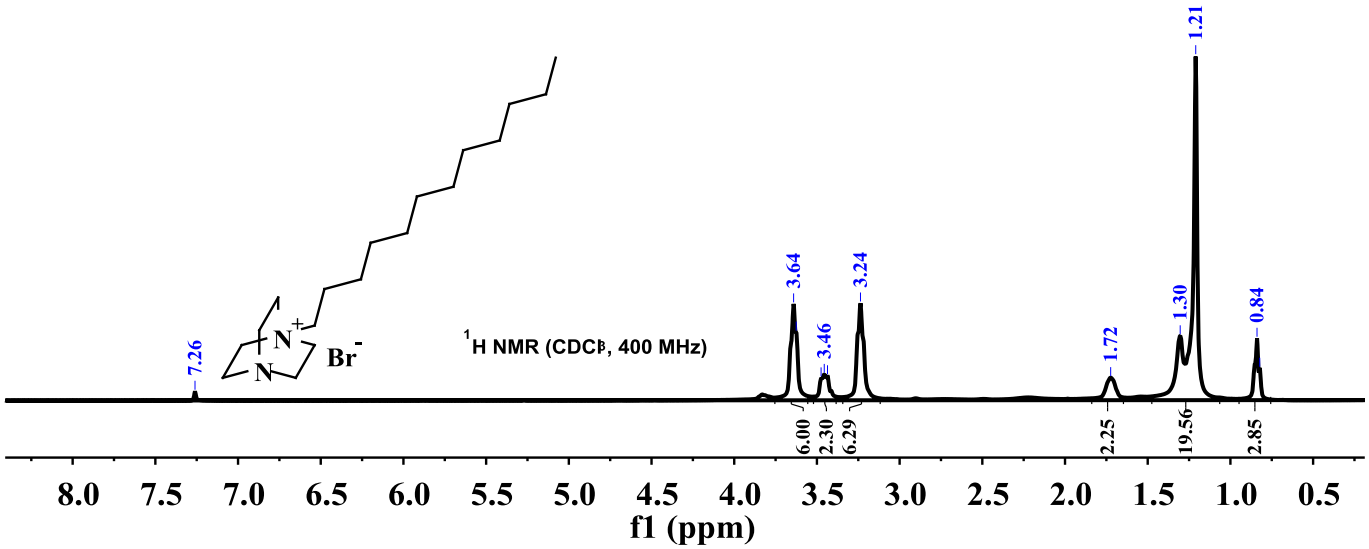
Yellow glassy solid; ^1H NMR ($CDCl_3$, 400 MHz) δ 3.49 (t, ^3J = 7.6 Hz, 6 H), 3.27 (t, ^3J = 8.4 Hz, 2 H), 3.08 (t, ^3J =7.2 Hz, 6 H) , 1.59 (s, 2 H), 1.06-1.15 (m, 10 H), 0.68 (t, ^3J = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 64.1, 52.0, 45.0, 31.1, 28.6, 28.5, 25.9,22.1, 21.6, 13.6; ESI-MS calcd for $C_{14}H_{29}N_2Br$ 305.3, found 225.46 $[M-Br]^+$, 79.03 $[M-C_{14}H_{29}N_2]^-$.

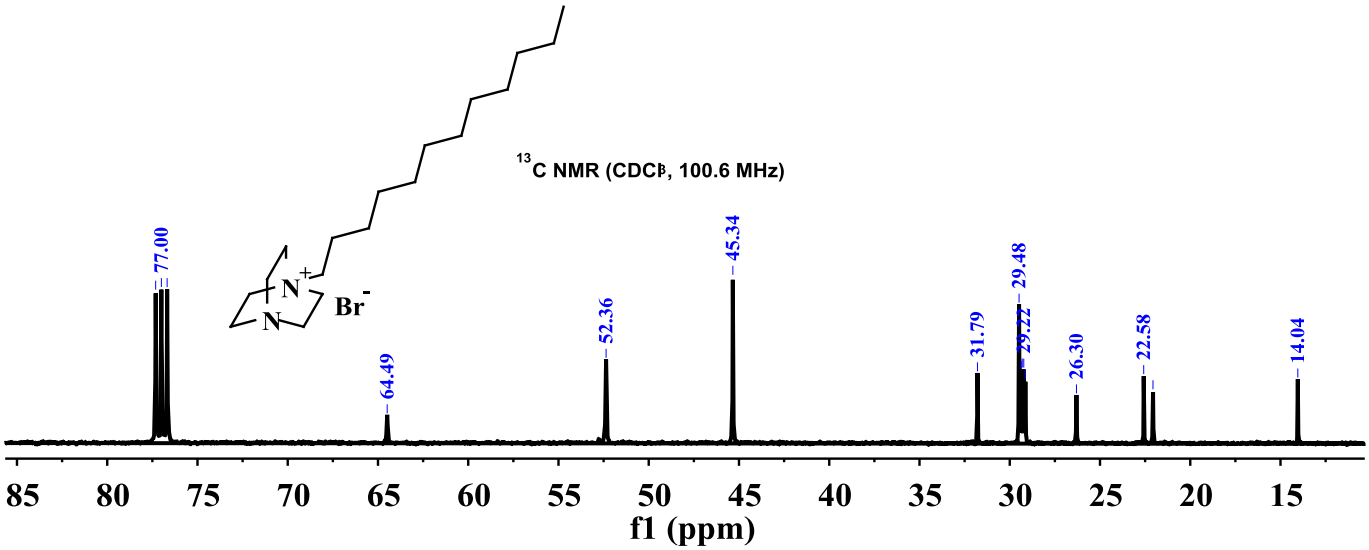




C_{12}DABCOBr

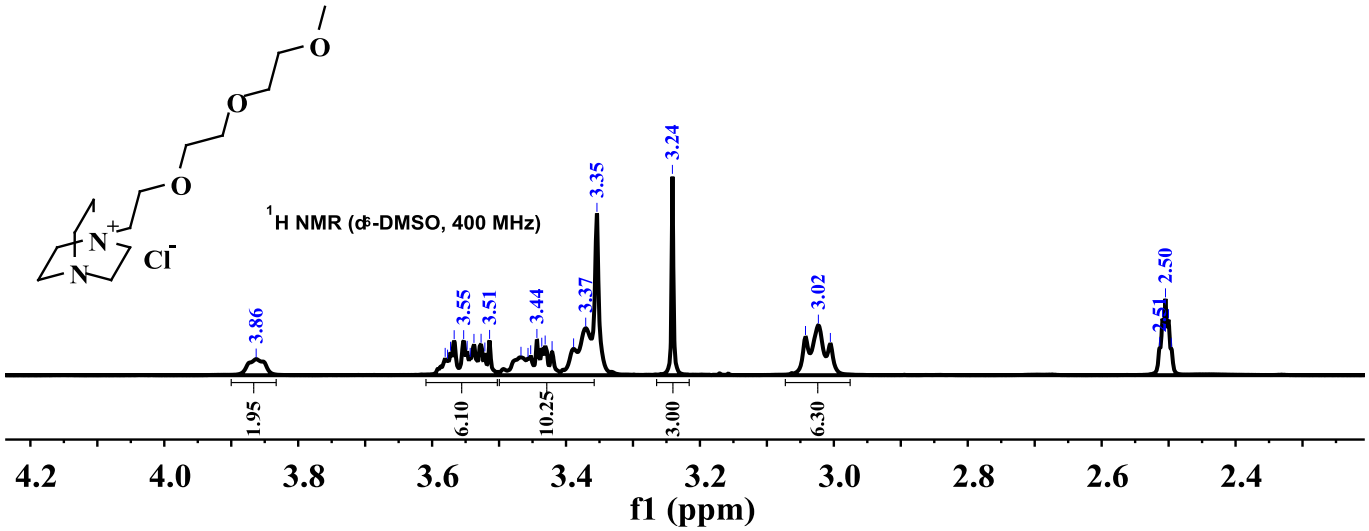
White solid; Mp 59-60 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.64 (s, 6 H), 3.46 (t, ^{3}J = 7.6 Hz, 2 H), 3.24 (s, 6 H), 1.21-1.30 (m, 20 H), 0.84 (t, ^{3}J = 5.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 64.5, 52.4, 45.3, 31.8, 29.5, 29.4, 29.3, 29.2, 29.1, 26.3, 22.6, 22.1, 14.0; ESI-MS calcd for $C_{18}H_7N_2Br$ 338.22, found 281.82 $[M\text{-}Br]^+$, 79.03 $[M\text{-}C_{18}H_{37}N_2]^-$.

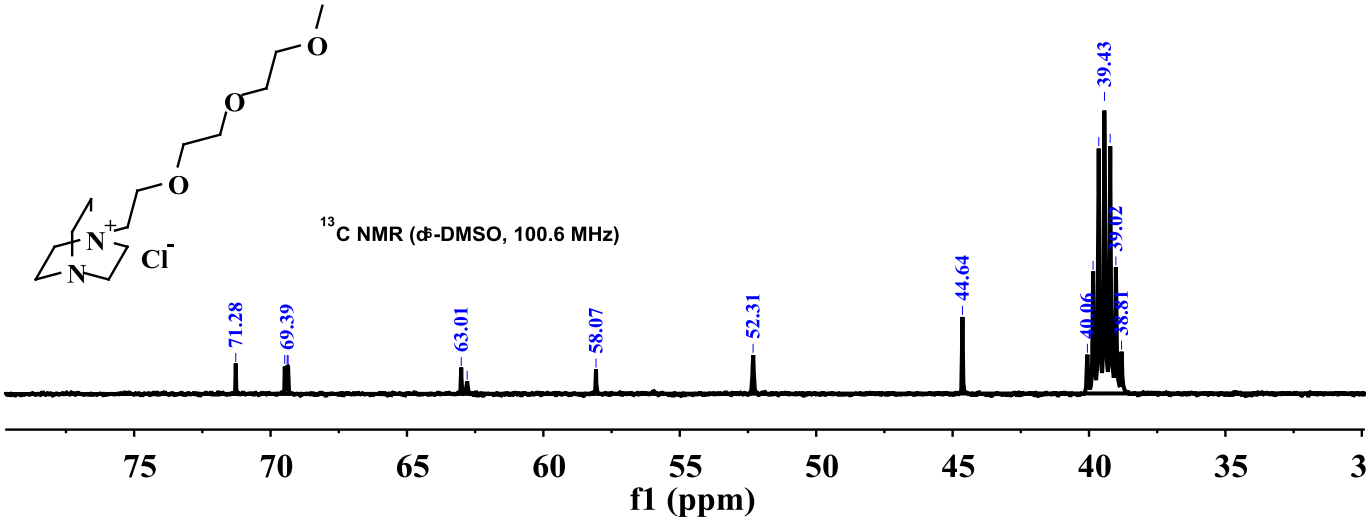




PEG_{150}MeDABCOCl

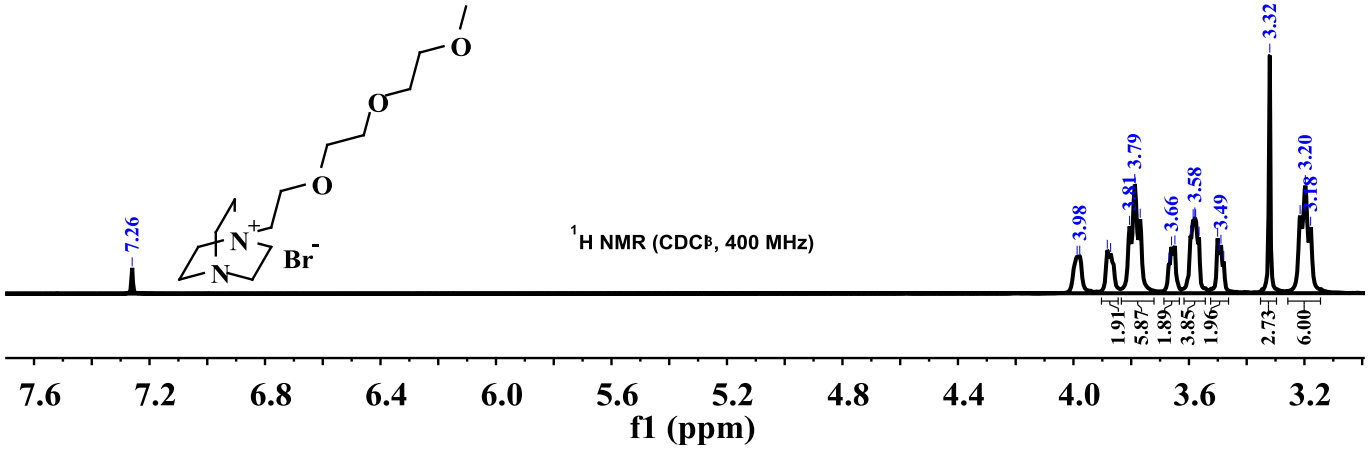
Yellow liquid; ^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.86 (s, 2H), 3.51-3.57 (m, 6H), 3.37-3.46 (m, 10H), 3.24 (s, 3H), 3.02 (t, ^{3}J = 8 Hz, 6H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 71.3, 69.5, 69.4, 69.3, 63.0, 62.8, 58.1, 52.3, 44.6; ESI-MS (4.8 kV) $C_{13}H_{27}ClN_2O_3$ 294.17, found 259.4, 260.4 $[M\text{-}Cl]^+$.

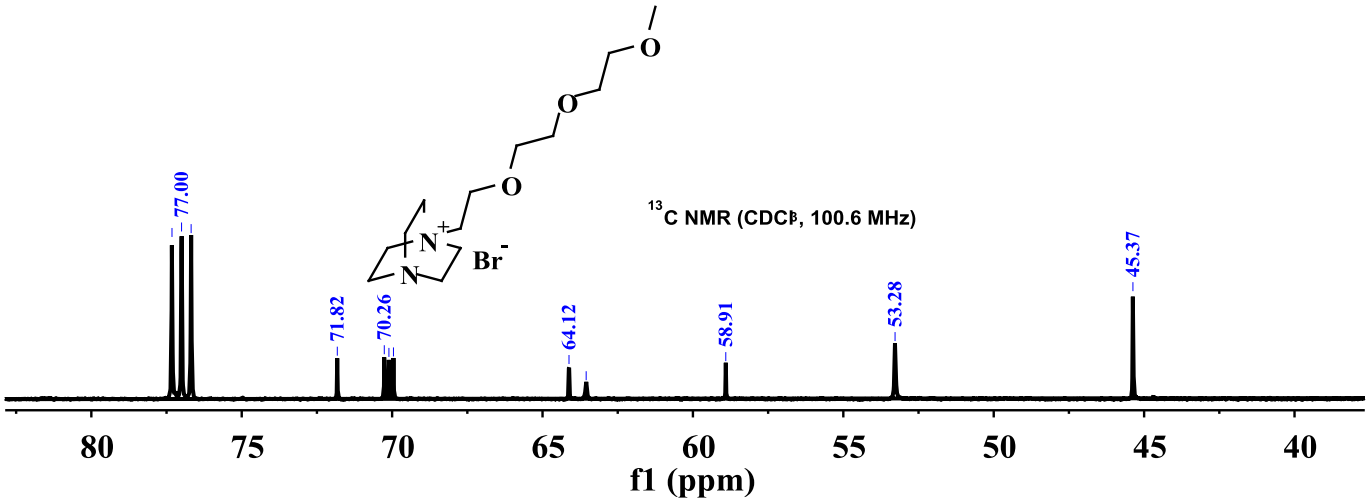




PEG_{150}MeDABCOBr

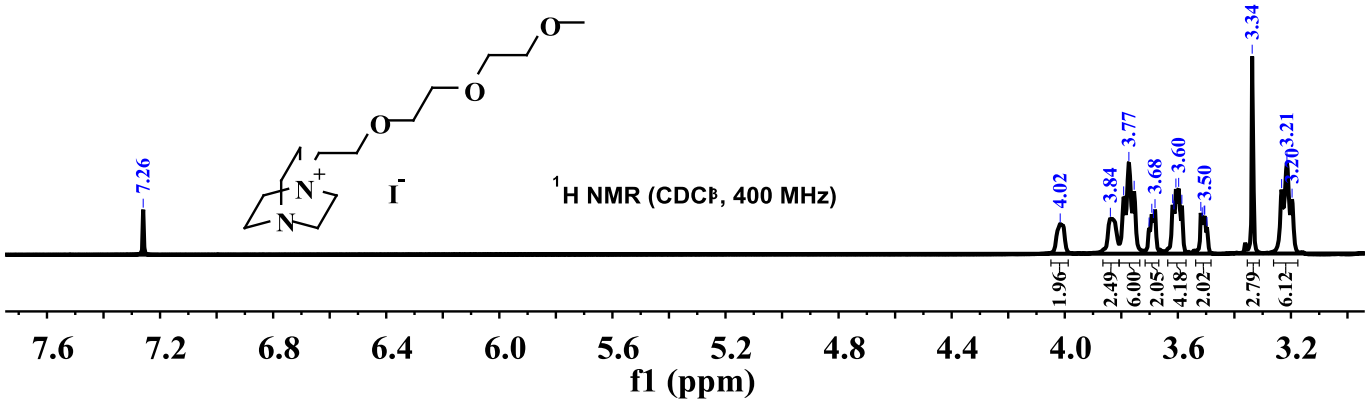
Yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.99 (s, 2H), 3.88 (s, 2H), 3.79 (t, ^{3}J = 7.2 Hz, 6H), 3.65-3.67 (m, 2H), 3.56-3.59 (m, 4H), 3.48-3.50 (m, 2H), 3.32 (s, 3H), 3.20 (t, ^{3}J = 7.2 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 71.8, 70.3, 70.1, 70.0, 64.1, 63.5, 58.9, 53.3, 45.4;

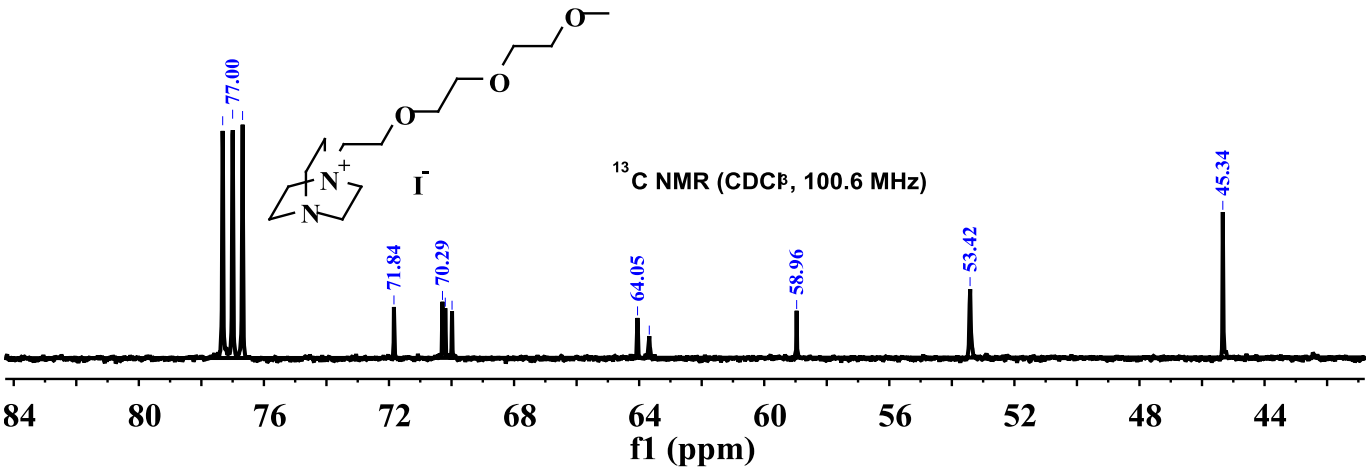




$PEG_{150}MeDABCOI$

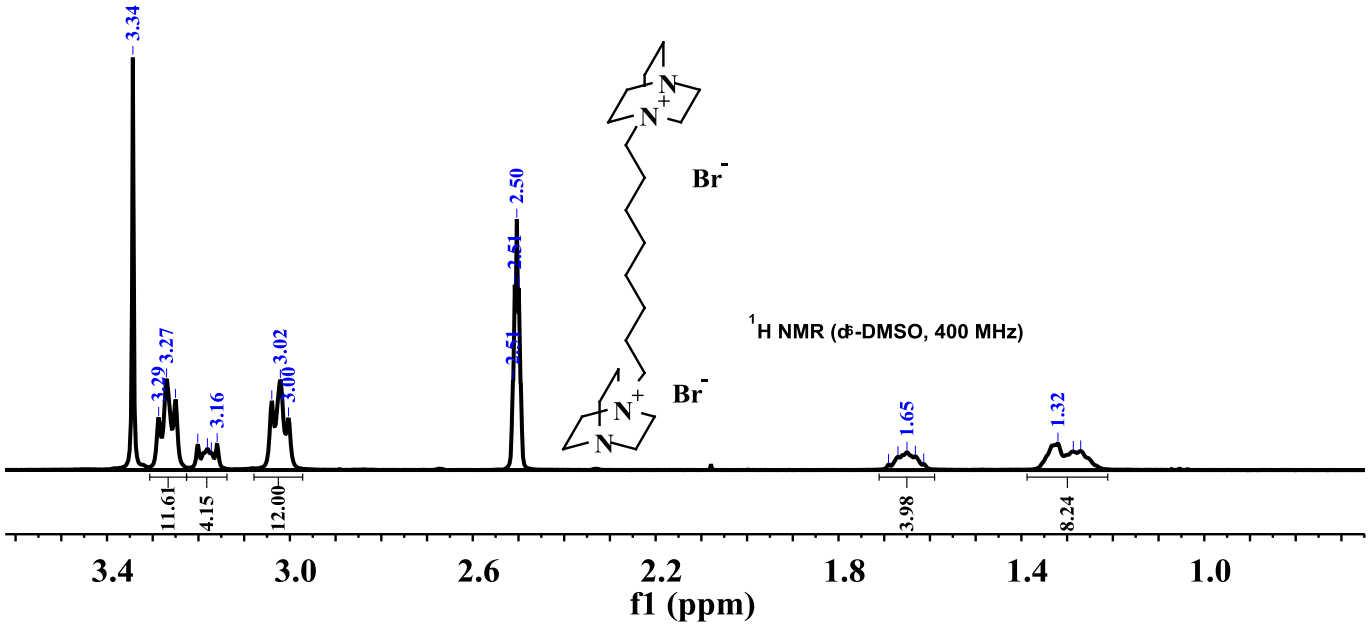
Light yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.02 (s, 2H), 3.84 (s, 2H), 3.77 (t, ^{3}J = 7.6 Hz, 6H), 3.68-3.70 (m, 2H), 3.59-3.62 (m, 4H), 3.50-3.52 (m, 2H), 3.34 (s, 3H), 3.21 (t, ^{3}J = 7.6 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 45.3, 53.4, 59.0, 63.7, 64.0, 70.0, 70.2, 70.3, 71.8; ESI-MS (4.8 kV) $C_{13}H_{27}IN_2O_3$ 386.11, found 259.27, 260.4 $[M\text{-}I]^+$, 126.93 $[M\text{-}C_{13}H_{27}N_2O_3]^-$, 512.80 $[2M\text{-}C_{13}H_{27}N_2O_3]^-$, 898.47 $[3M\text{-}C_{13}H_{27}N_2O_3]^-$.

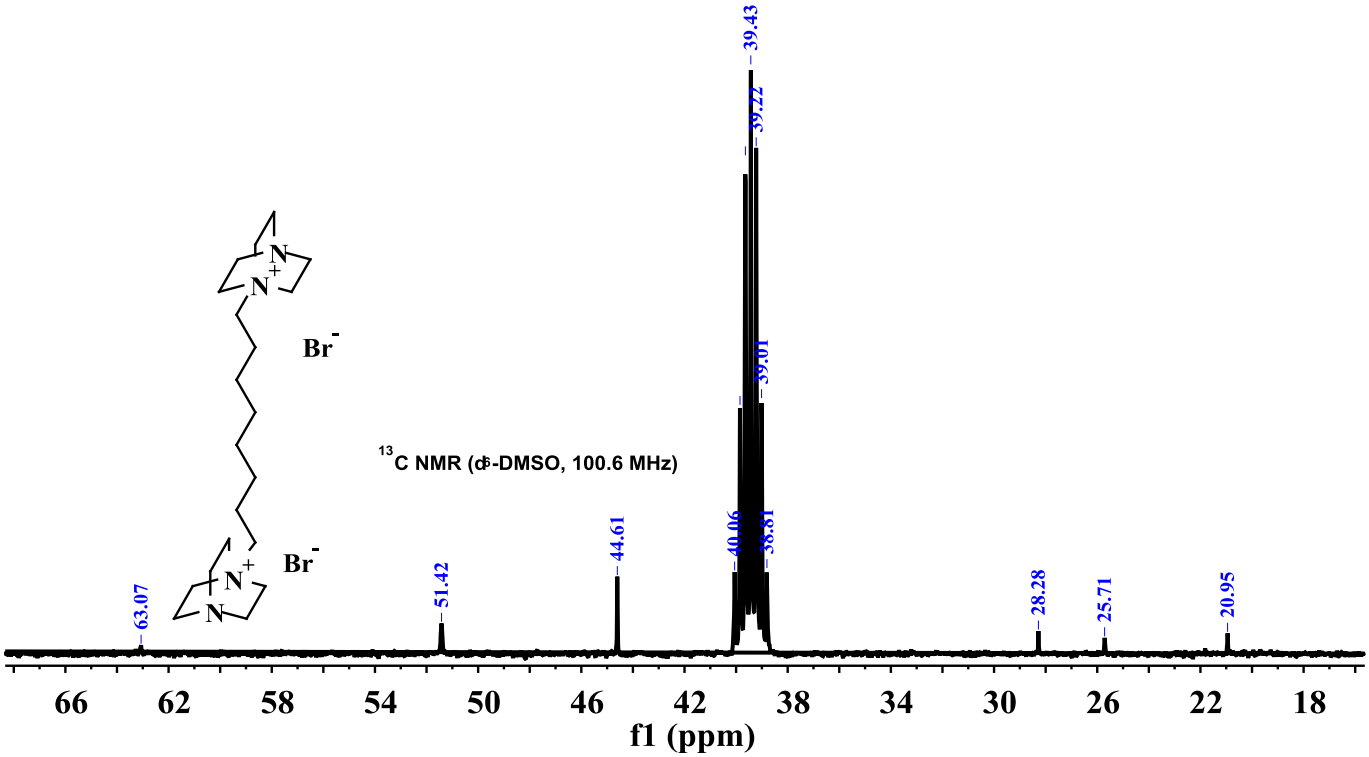




$C_8(DABCO)_2Br_2$

White solid; Mp. 173-174 °C; ^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.27 (t, ^{3}J = 6.8 Hz, 12H), 3.15-3.20 (m, 4H), 3.02 (t, ^{3}J = 7.6 Hz, 12H), 1.63-1.67 (m, 4H), 1.27-1.33 (m, 8H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 63.1, 51.4, 44.6, 28.3, 25.7, 20.9; ESI-MS (4.8 kV) $C_{20}H_{40}Br_2N_4$ 494.16, found 168.4 $[M\text{-}Br_2]^{2+}/2$, 415.4 $[M\text{-}Br]^+$, 79.0, 81.0 $[M\text{-}C_{20}H_{40}N_4]^-$.

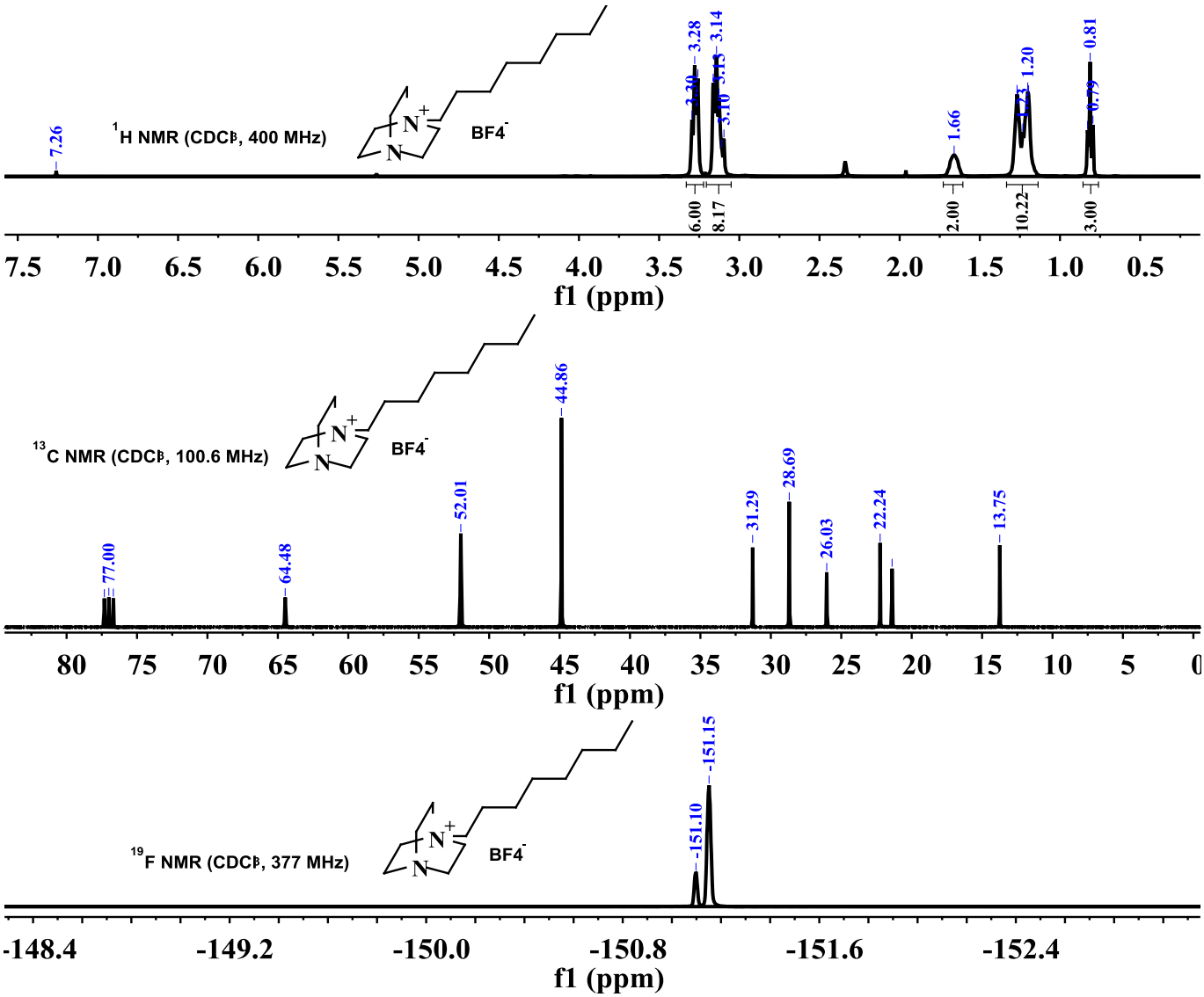




C_8DABCOBF$_4$

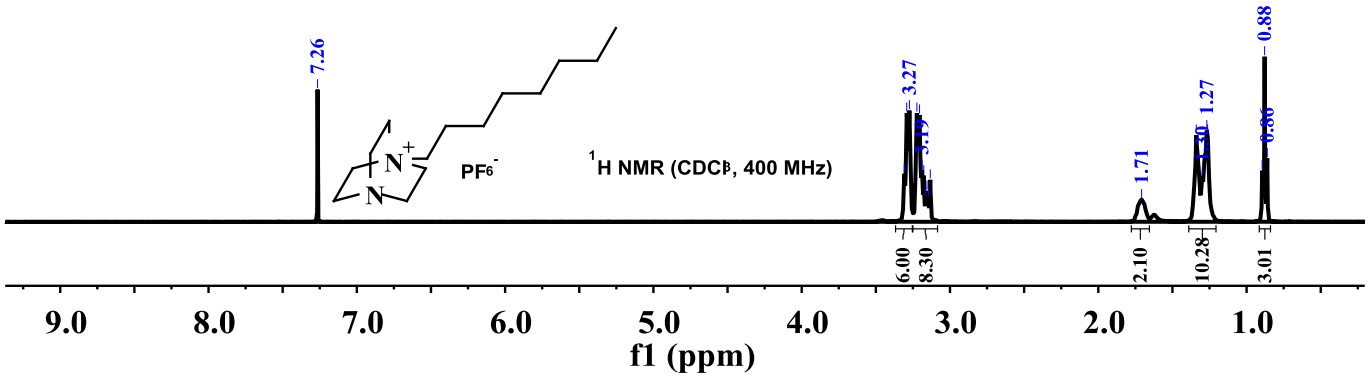
A solution of 1-octyl-4-aza-1-azaniabicyclo[2.2.2]octane bromide (C_8DABCOBr) (1.63 mmol) in MeCN (1.0 mL), prepared at r.t. under Ar in a flame-dried round-bottomed flask, was transferred via cannula to a slurry of $NaBF_4$ (1.65 mmol) in MeCN (1.5 mL). A 0.5 mL MeCN rinse was used to ensure complete transfer. A white precipitate formed immediately and the resulting slurry was stirred at r.t. for 24 h. Filtration followed by concentration of the filtrate in vacuo yielded a colorless, viscous liquid shown to be contaminated with bromide by a positive $AgNO_3$ test. The crude was partitioned between CH_2Cl_2 and H_2O, and the CH_2Cl_2 layer was washed repeatedly with H_2O until the aqueous layer gave a negative $AgNO_3$ test. The CH_2Cl_2 extract was dried (Na_2SO_4), subjected to filtration and concentrated in vacuo. Drying under high vacuum at 60 °C for 3 d yielded 1-octyl-4-aza-1-azaniabicyclo[2.2.2]octane tetrafluoroborate (C_8DABCOBF$_4$) as a white glassy solid.

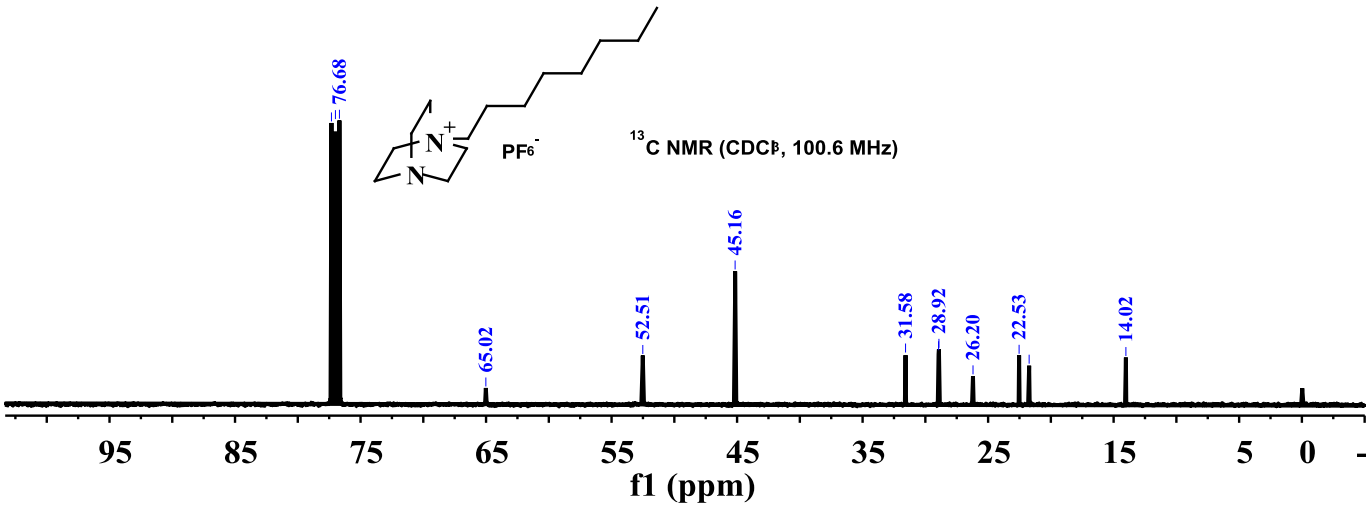
White glassy solid; Mp 37-38 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.28 (t, ^{3}J = 7.6 Hz, 6 H), 3.10-3.16 (m, 8 H), 1.66 (s, 2 H), 1.20-1.27 (m, 10 H), 0.81 (t, ^{3}J = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 64.5, 52.0, 44.9, 31.3, 28.7, 26.0, 22.2, 21.4, 13.7; ^{19}F NMR ($CDCl_3$, 377 MHz) δ -151.10, -151.15; ESI-MS calcd for $C_{14}H_{29}N_2BF_4$ 312.2, found 225.45 $[M\text{-}BF_4]^+$, 87.1 $[M\text{-}C_{14}H_{29}N_2]^-$.

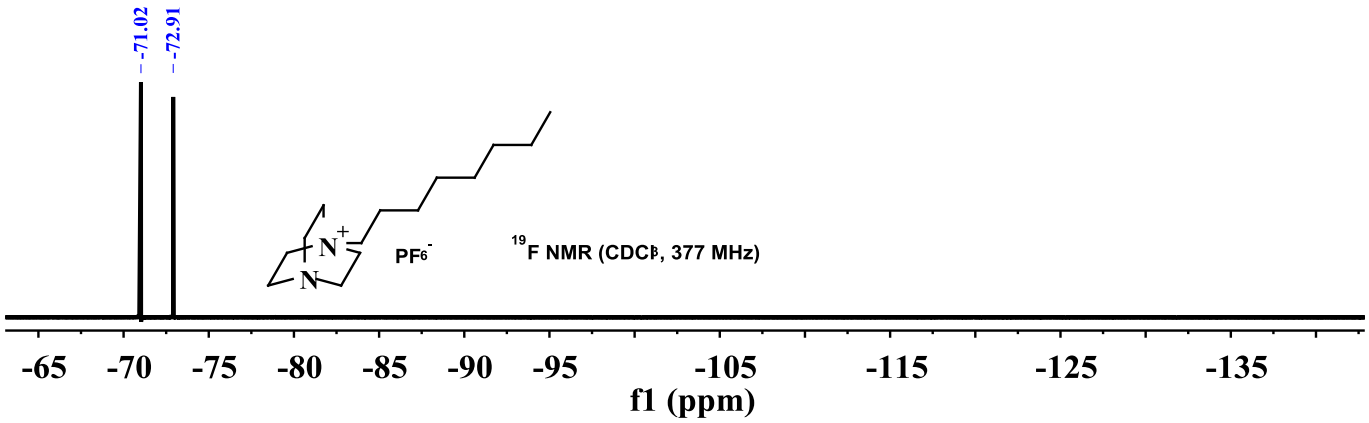


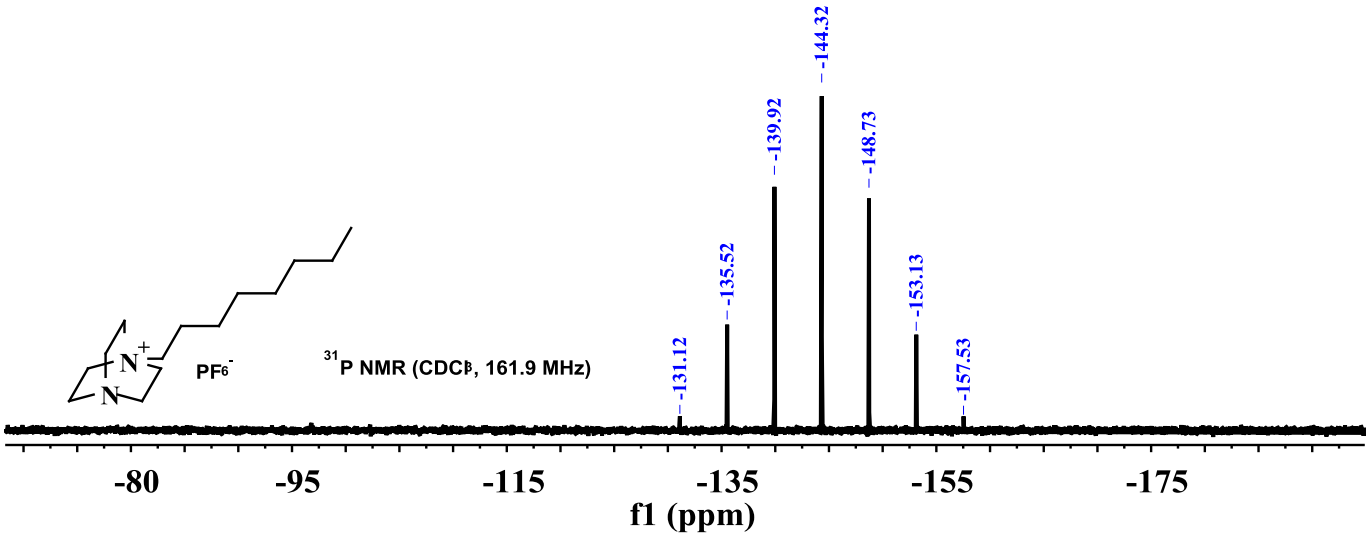
C_8DABCOPF$_6$

A solution of 1-octyl-4-aza-1-azaniabicyclo[2.2.2]octane bromide (C_8DABCOBr) (1.61 mmol) in MeCN (1.0 mL), prepared at r.t. under Ar in a flame-dried round-bottomed flask, was transferred via cannula to a slurry of KPF_6 (1.64 mmol) in MeCN (1.0 mL). A 0.5 mL MeCN rinse was used to ensure complete transfer. A white precipitate formed immediately and the resulting slurry was stirred at r.t. for 20 h. Filtration followed by concentration of the filtrate in vacuo yielded an oily, white solid shown to be contaminated with bromide by a positive $AgNO_3$ test. The crude was partitioned between CH_2Cl_2 and H_2O, and the CH_2Cl_2 layer was washed repeatedly with H_2O until the aqueous layer gave a negative $AgNO_3$ test. The CH_2Cl_2 extract was dried (Na_2SO_4), subjected to filtration and concentrated in vacuo. Drying under high vacuum at 60 °C for 5 d yielded 1-octyl-4-aza-1-azaniabicyclo[2.2.2]octane hexafluorophosphate (C_8DABCOPF$_6$) as a white solid.
White solid; Mp 161-163 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.27-3.30 (m, 6 H), 3.13-3.22 (m, 8 H), 1.70 (s, 2 H), 1.26-1.33 (m, 10 H), 0.87 (t, ^3J = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 65.0, 52.5, 45.2, 31.6, 28.94, 28.92, 26.2, 22.5, 21.7, 14.0; ^{19}F NMR ($CDCl_3$, 377 MHz) δ -71.02, -72.91; ^{31}P NMR ($CDCl_3$, 161.9 MHz) δ -144.33 (septet, $J_{P,F}$ = 712Hz); ESI-MS calcd for $C_{14}H_{29}N_2PF_6$ 370.36, found 225.43 [M-PF_6]$^+$, 145.20 [M-$C_{14}H_{29}N_2$]$^-$.







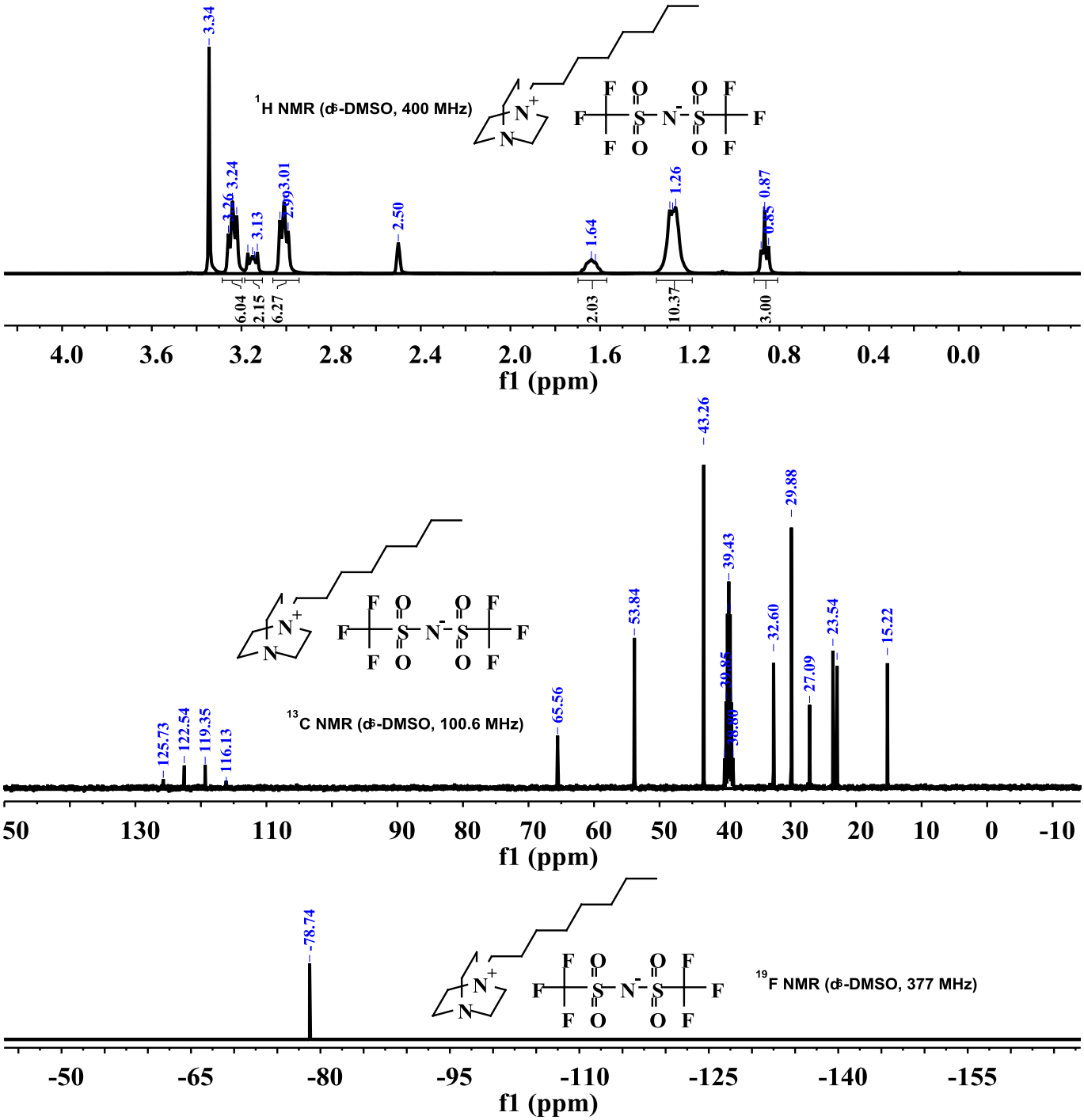


C_8DABCONTf$_2$, C_{12}DABCONTf$_2$, PEG$_{150}$MeDABCONTf$_2$, C_8(DABCO)$_2$(NTf$_2$)$_2$[7]

A solution of LiN(SO_2CF_3)$_2$ (1.62 mmol) in MeCN (1.0 mL), prepared at r.t. under Ar in a flame-dried round-bottomed flask, was transferred via cannula to a solution of C_8DABCOBr, C_{12}DABCOBr, PEG$_{150}$MeDABCOBr (1.60 mmol) and C_8(DABCO)$_2$Br$_2$ (0.80 mmol) in MeCN (1.5 mL). A 0.5 mL MeCN rinse was used to ensure complete transfer. A white precipitate formed within 5 min and the resulting slurry was stirred at r.t. for 47 h. Filtration followed by concentration of the filtrate in vacuo yielded a colorless liquid shown to be contaminated with chloride by a positive $AgNO_3$ test. The crude was partitioned between CH_2Cl_2 and H_2O, and the CH_2Cl_2 layer was washed repeatedly with H_2O until the aqueous layer gave a negative $AgNO_3$ test. The CH_2Cl_2 extract was dried (Na_2SO_4), subjected to filtration and concentrated in vacuo. Drying under high vacuum at 60 °C for 24 h.

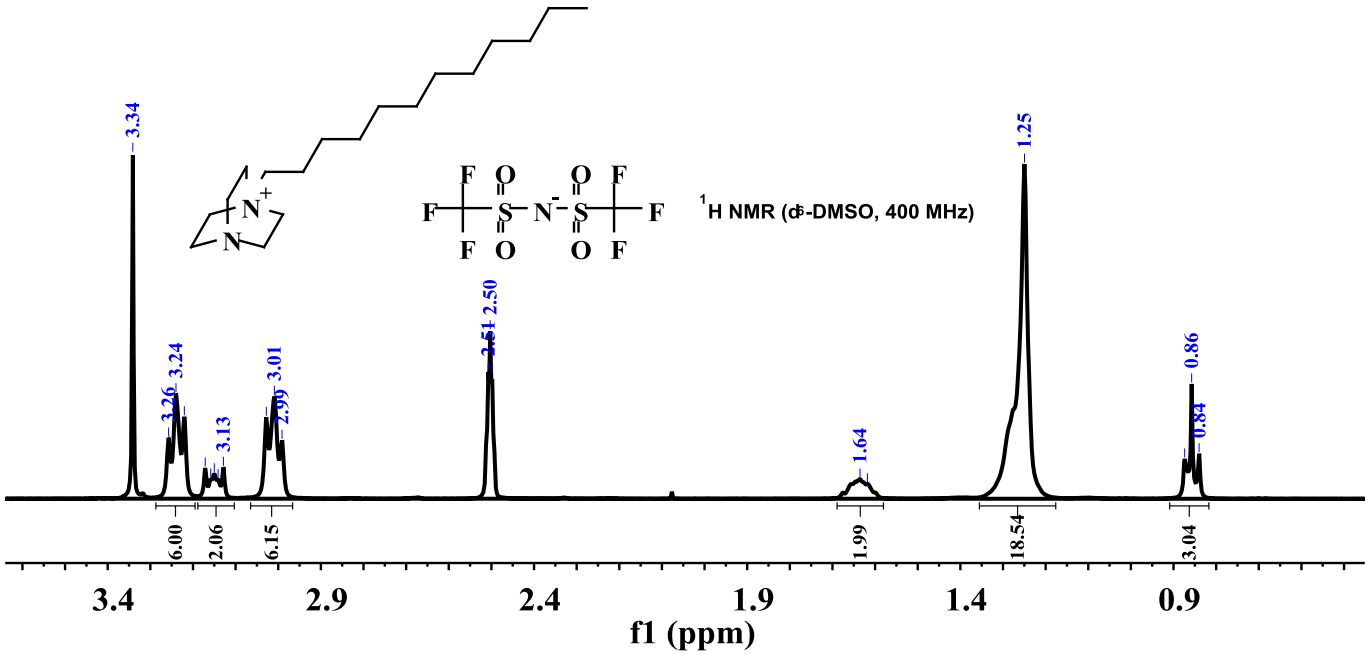
C_8DABCONTf$_2$

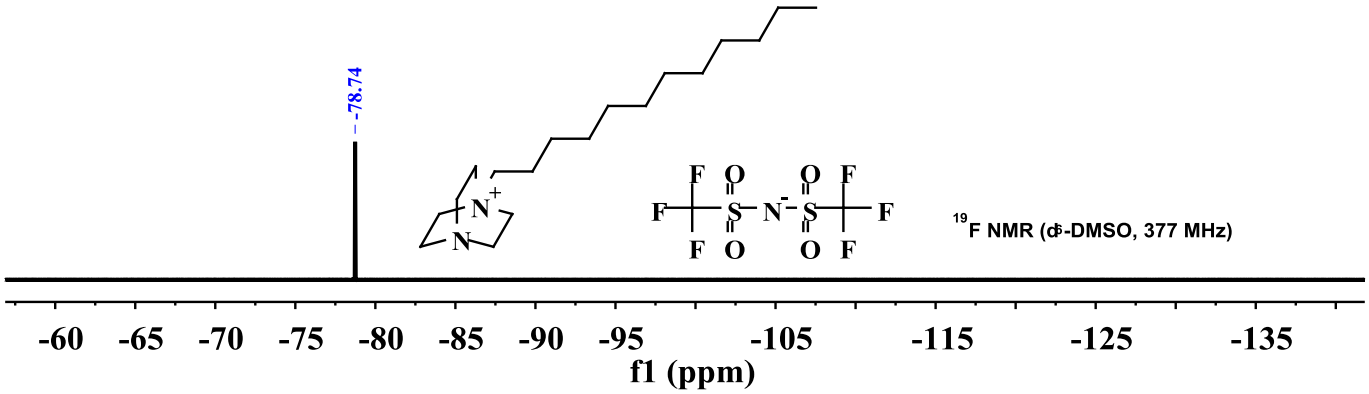
Yellow liquid; ^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.24 (t, ^3J = 7.2 Hz, 6H), 3.13-3.17 (m, 2H), 3.01 (t, ^3J = 7.6 Hz, 6H), 1.62-1.66 (m, 2H), 1.26-1.29 (m, 10H), 0.87 (t, ^3J = 6.4 Hz, 3H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -78.74; ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 124.2, 121.0, 117.8, 114.6, 63.2, 51.4, 44.6, 31.0, 28.4, 28.3, 25.8, 22.0, 20.9, 13.8. ESI-MS (4.8 kV) calcd for $C_{16}H_{29}N_3O_4S_2F_6$ 505.54, found 225.43 [M-$C_2F_6NO_4S_2$]$^+$, 280.10 [M-$C_{14}H_{29}N_2$]$^-$.

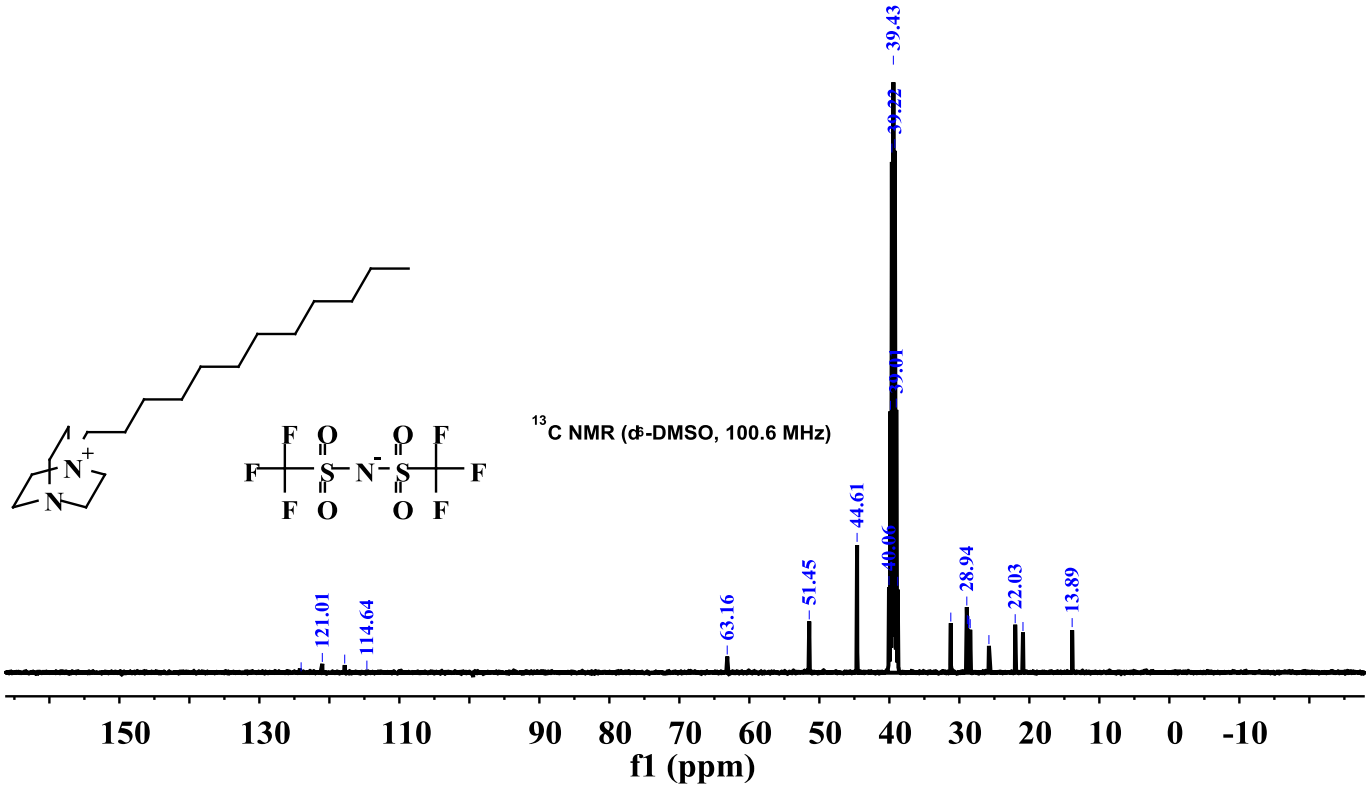


C_{12}DABCONTf$_2$

Yellow solid; Mp. 49-50 ^{o}C; ^{1}H NMR (400 MHz, d_6-DMSO) δ 3.24 (t, ^{3}J = 6.8 Hz, 6H), 3.15 (t, ^{3}J = 8.4 Hz, 2H), 3.01 (t, ^{3}J = 7.6 Hz, 6H), 1.61-1.67 (m, 2H), 1.25 (s, 18H), 0.86 (t, ^{3}J = 6.4 Hz, 3H); ^{19}F NMR (377 MHz , d_6-DMSO) δ -78.74; ^{13}C NMR (100.6 MHz, d_6-DMSO) δ 124.2, 121.0, 117.8, 114.6, 63.2, 51.4, 44.6, 31.2, 28.9, 28.8, 28.7, 28.6, 28.5, 25.8, 22.0, 20.9, 13.9; ESI-MS (4.8 kV) calcd for $C_{20}H_{37}F_6N_3O_4S_2$ 561.21, found 281.5 $[M\text{-}C_2F_6NO_4S_2]^+$, 280.1 $[M\text{-}C_{18}H_{37}N_2]^-$.

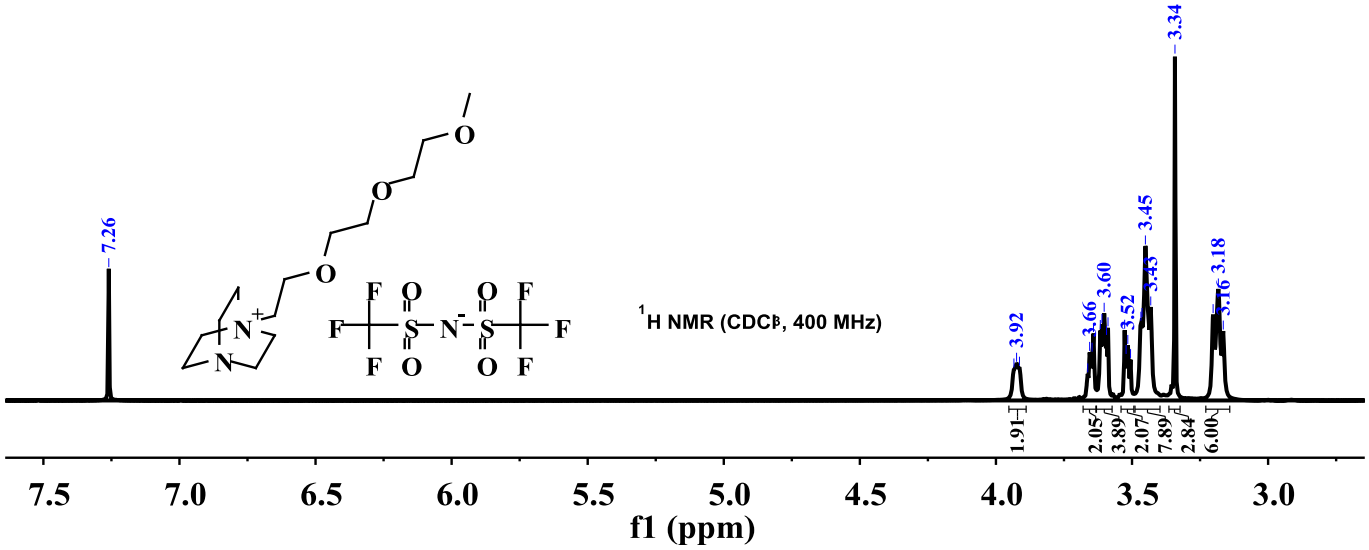


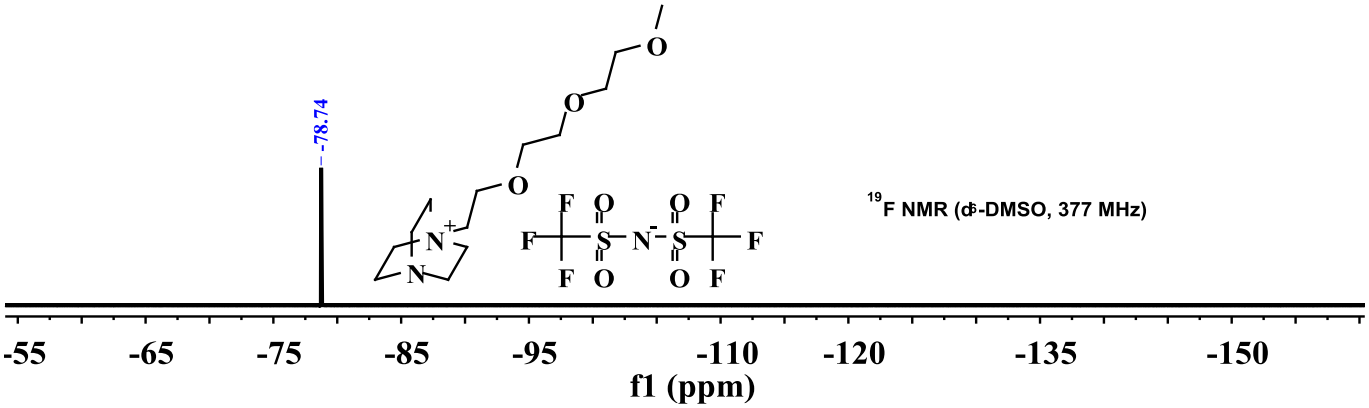


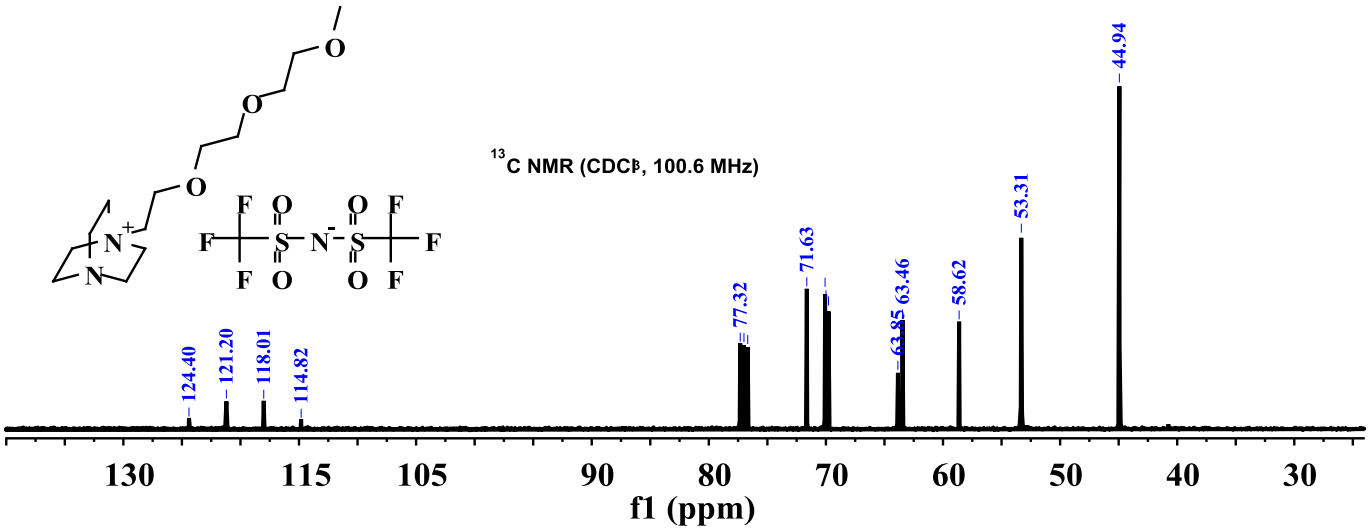


PEG$_{150}$MeDABCONTf$_2$

Yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.92 (s, 2H), 3.64-3.66 (m, 2H), 3.59-3.62 (m, 4H), 3.51-3.53 (m, 2H), 3.43-3.47 (m, 8H), 3.34 (s, 3H), 3.18 (t, ^{3}J = 7.6 Hz, 6H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -78.74; ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 124.4, 121.2, 118.0, 114.8, 71.6, 70.1, 70.0, 69.8, 63.8, 63.5, 58.6, 53.3, 44.9; ESI-MS (4.8 kV) $C_{15}H_{27}F_6N_3O_7S_2$ 539.12, found 259.4, 260.4 [M- $C_2F_6NO_4S_2$]$^{+}$, 280.1 [M- $C_{13}H_{27}N_2O_3$]$^{-}$.

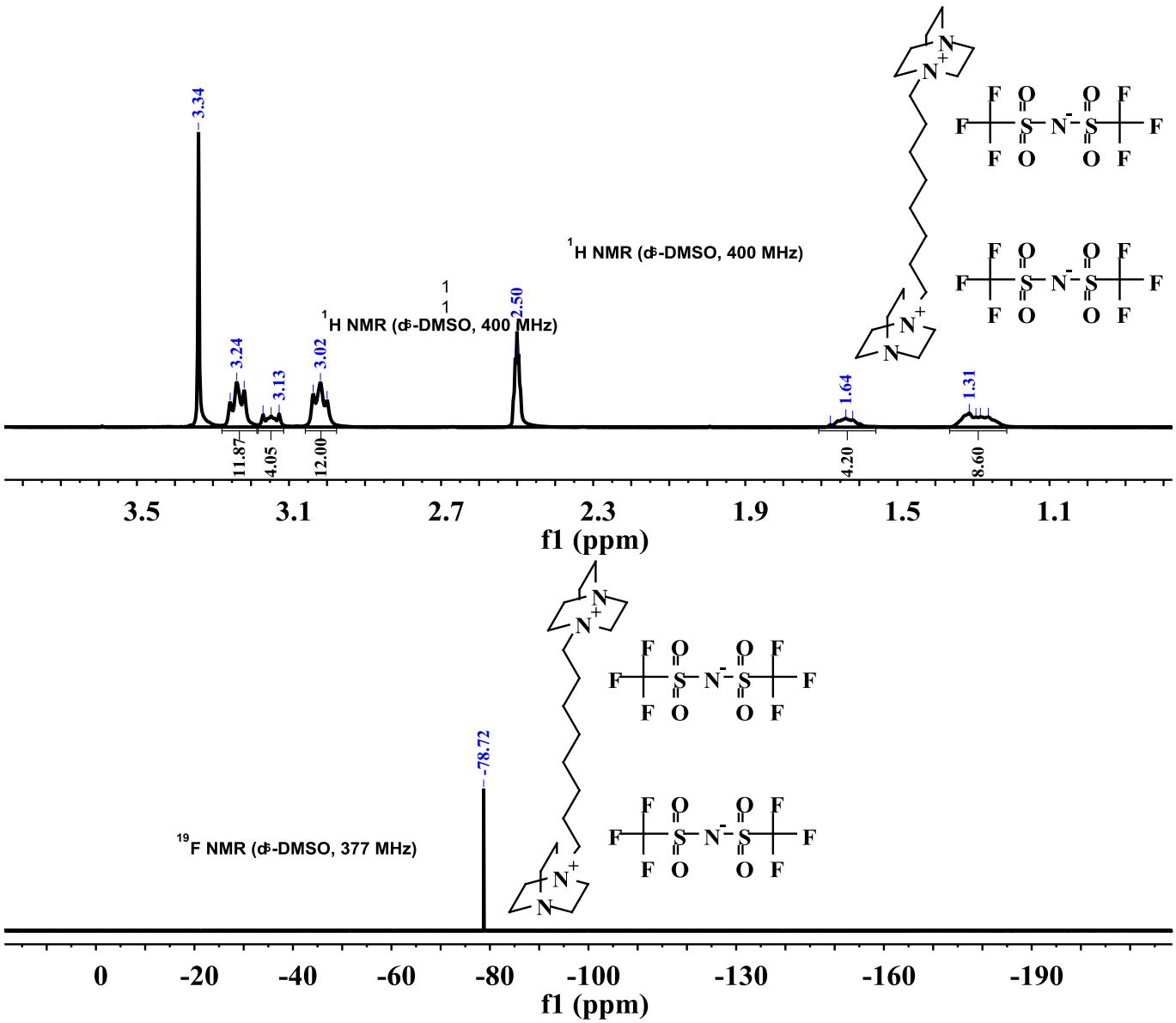




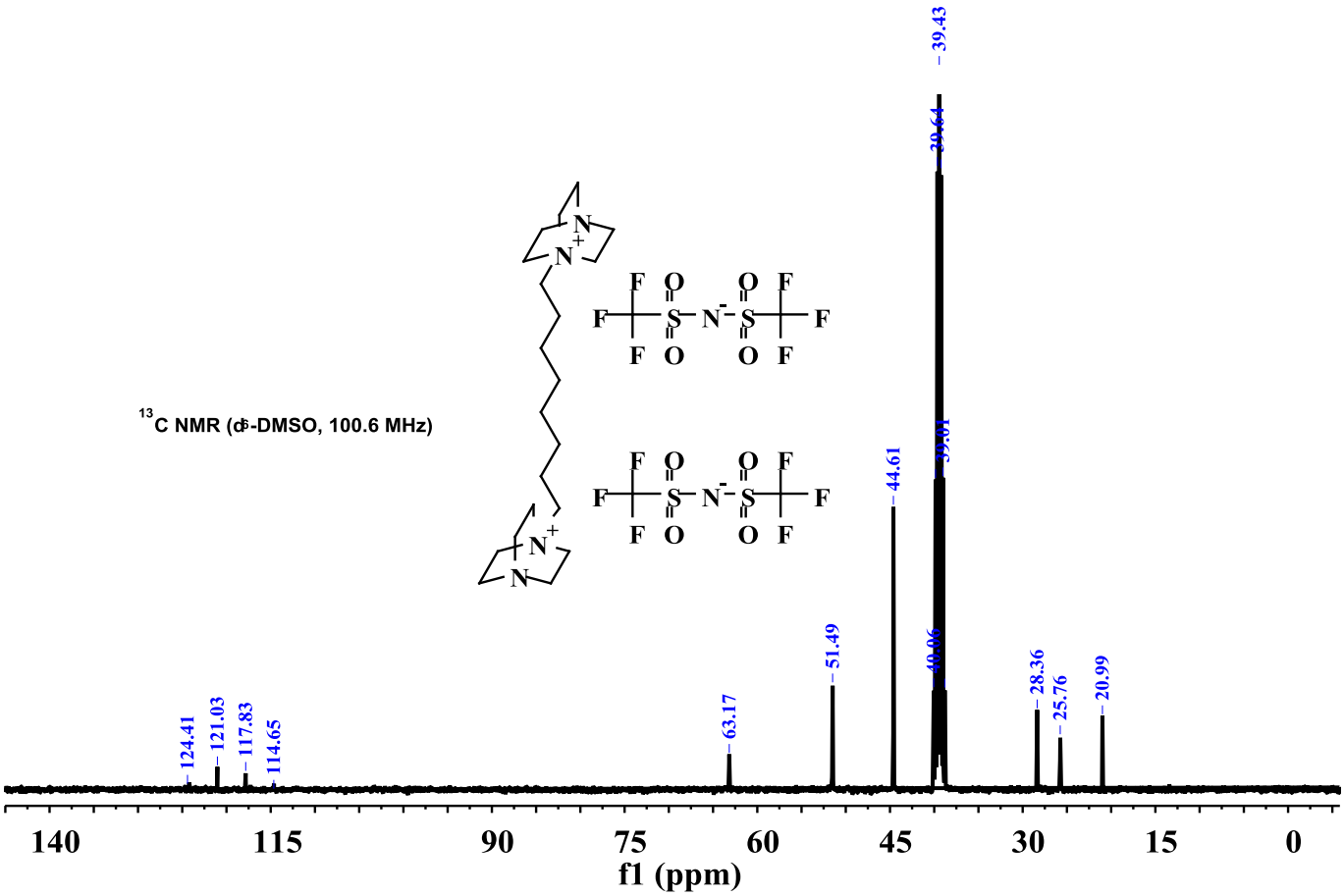


C_8(DABCO)$_2$(NTf_2)$_2$

White solid; Mp. 158-159 °C; ^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.25 (t, ^{3}J = 6.8 Hz, 12H), 3.12-3.16 (m, 4H), 3.01 (t, ^{3}J = 7.6 Hz, 12H), 1.59-1.67 (m, 4H), 1.26-1.31 (m, 8H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -78.72; ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 124.2, 121.0, 117.8, 114.6, 63.2, 51.5, 44.6, 28.4, 25.8, 21.0; ESI-MS (4.8 kV) $C_{24}H_{40}F_{12}N_6O_8S_4$ 896.16, found 168.4 [M-$C_4F_{12}N_2O_8S_4$]$^{2+}$/2, 616.5 [M- $C_2F_6NO_4S_2$]$^{+}$, 280.1 [M- $C_{20}H_{40}N_4$]$^{-}$.


3.34
3.24
3.13
3.02
2.50
1.64
1.31
11.87
4.05
12.00
4.20
8.60
^{1}H NMR (d^6-DMSO, 400 MHz)
3.5
3.1
2.7
2.3
1.9
1.5
1.1
f1 (ppm)

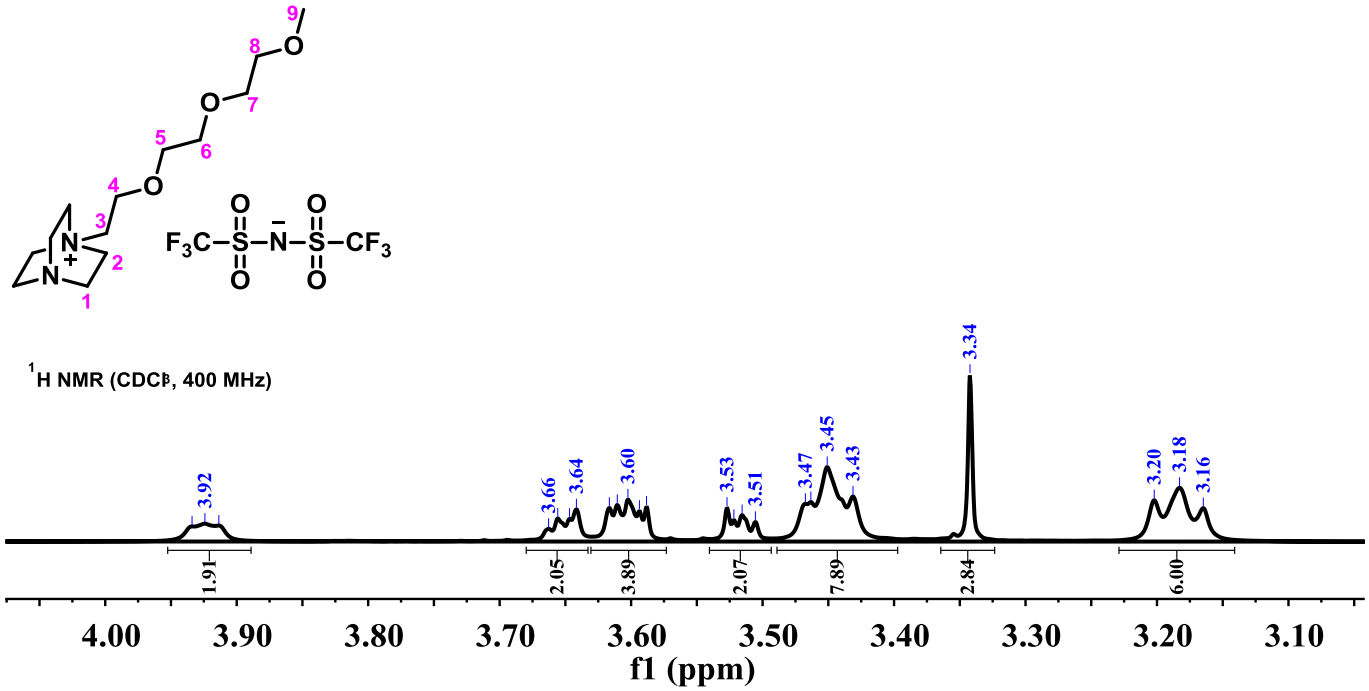
-78.72
^{19}F NMR (d^6-DMSO, 377 MHz)
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)



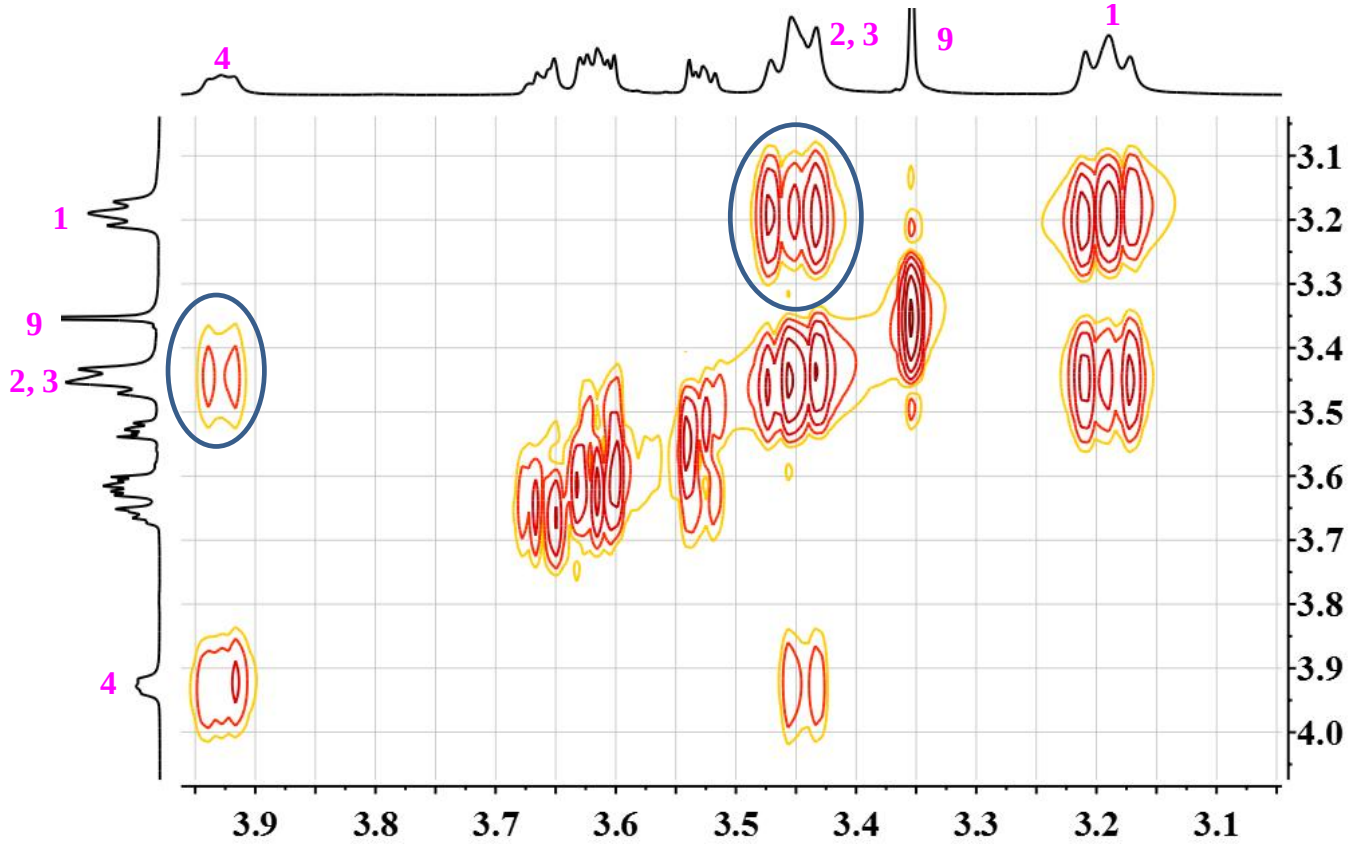
3. Characterization of $PEG_{150}MeDABCONTf_2$ (^1H NMR, ^{13}C NMR, ^1H-^1H COSY, ^1H-^1H NOESY and ^1H-^{13}C HSQC) before and after SO_2 capture

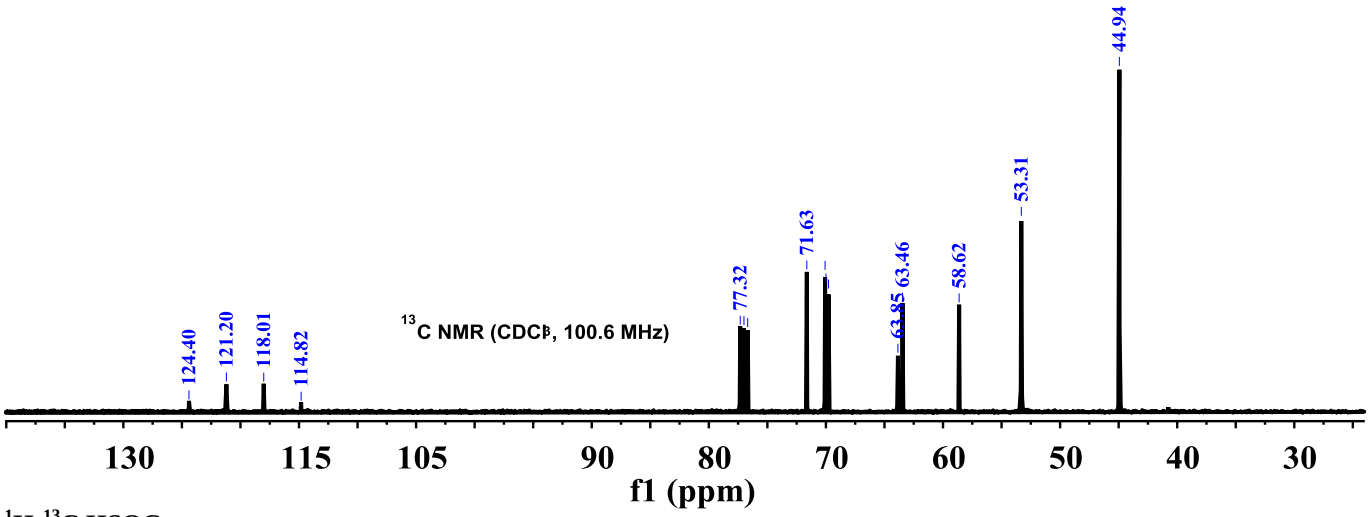
$PEG_{150}MeDABCONTf_2$

^1H NMR ($CDCl_3$, 400 MHz) δ 3.92 (s, 2H), 3.64-3.66 (m, 2H), 3.59-3.62 (m, 4H), 3.51-3.53 (m, 2H), 3.43-3.47 (m, 8H), 3.34 (s, 3H), 3.18 (t, ^3J = 7.6 Hz, 6H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -78.74; ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 124.4, 121.2, 118.0, 114.8, 71.6, 70.1, 70.0, 69.8, 63.8, 63.5, 58.6, 53.3, 44.9;

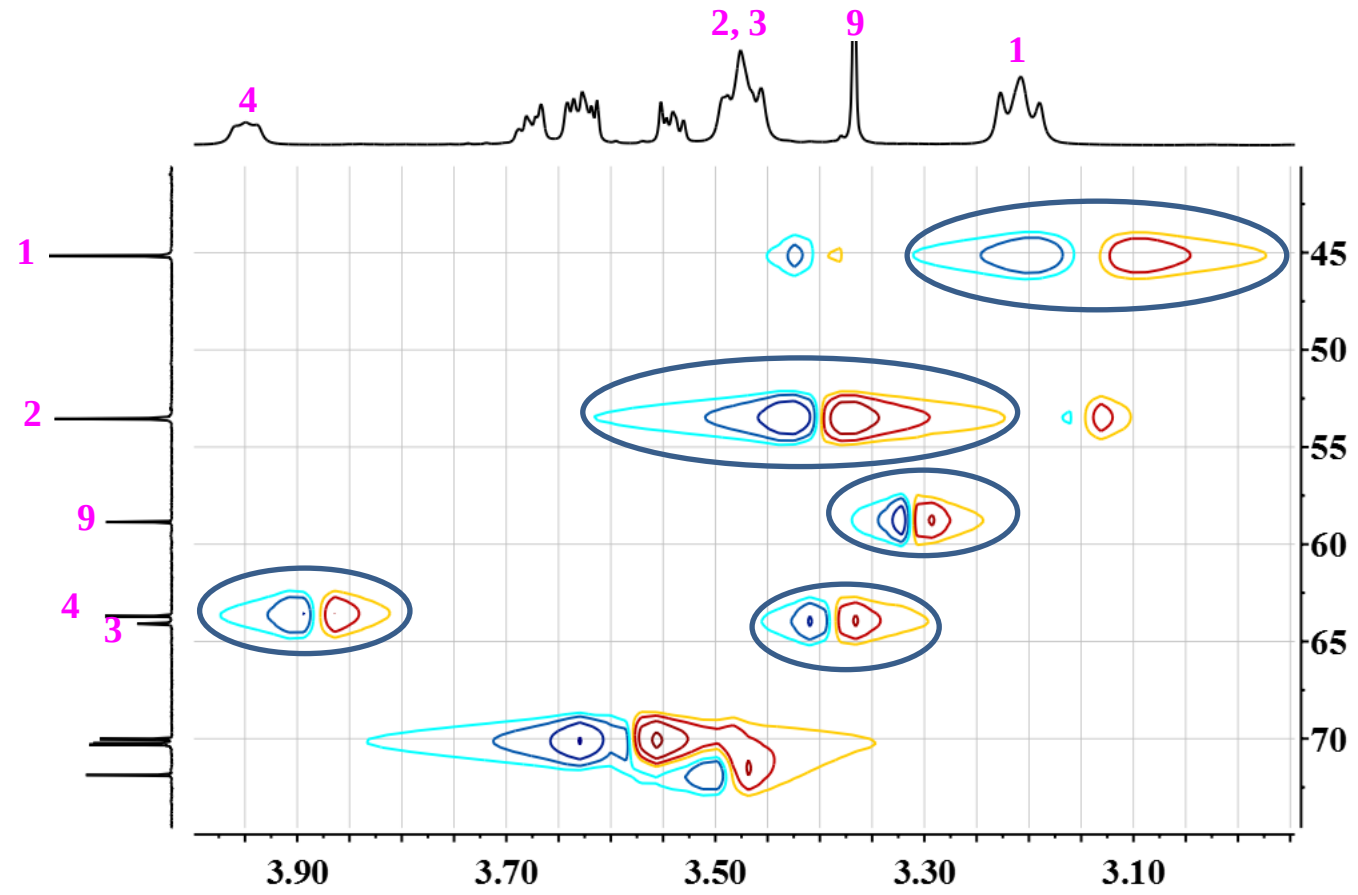


^{1}H-^{1}H COSY



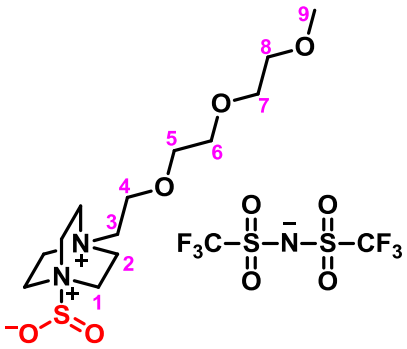


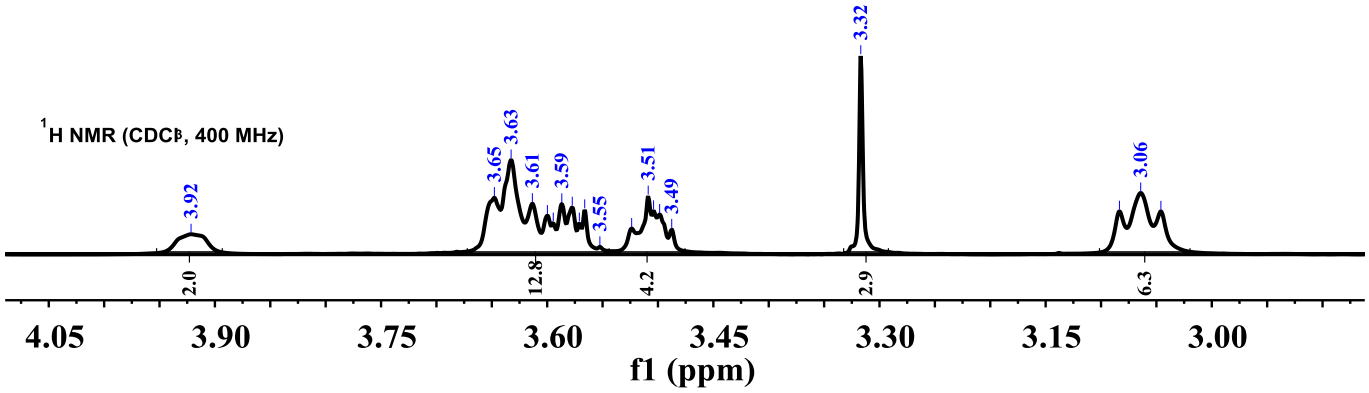
^{1}H-^{13}C HSQC



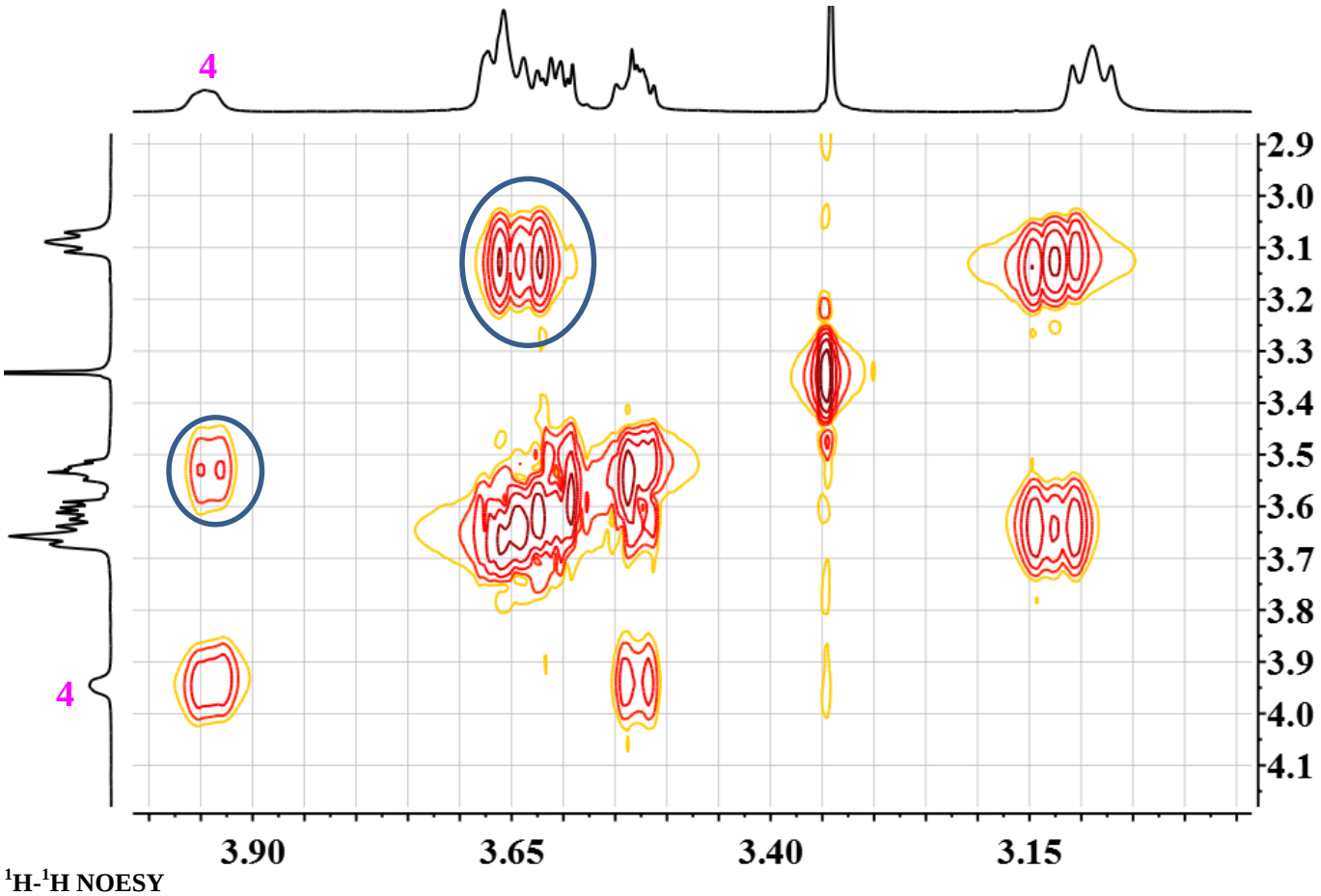
$PEG_{150}MeDABCONTf_2 + SO_2$

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.92 (s, 2H), 3.57-3.65 (m, 12H), 3.49-3.52 (m, 4H), 3.32 (s, 3H), 3.06 (t, ^3J = 8Hz, 6H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -79.04; ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 124.1, 121.0, 117.8, 114.6, 71.3, 69.7, 69.6, 69.3, 63.5, 63.2, 58.0, 53.3, 42.2.

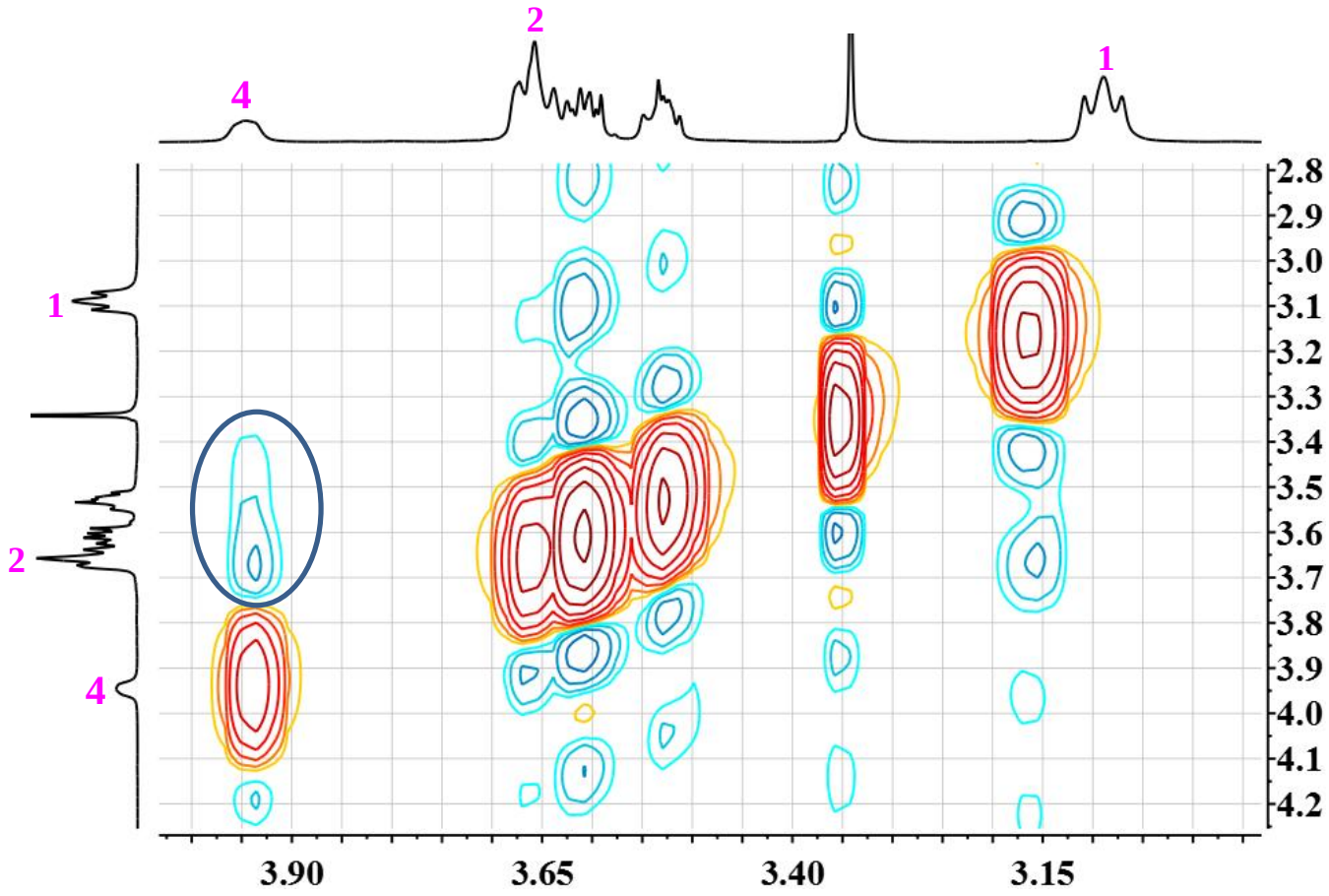




^{1}H-^{1}H COSY


4
4
2.9
3.0
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
4.1
3.90
3.65
3.40
3.15

^{1}H-^{1}H NOESY


2
1
4
1
2
4
2.8
2.9
3.0
3.1
3.2
3.3
3.4
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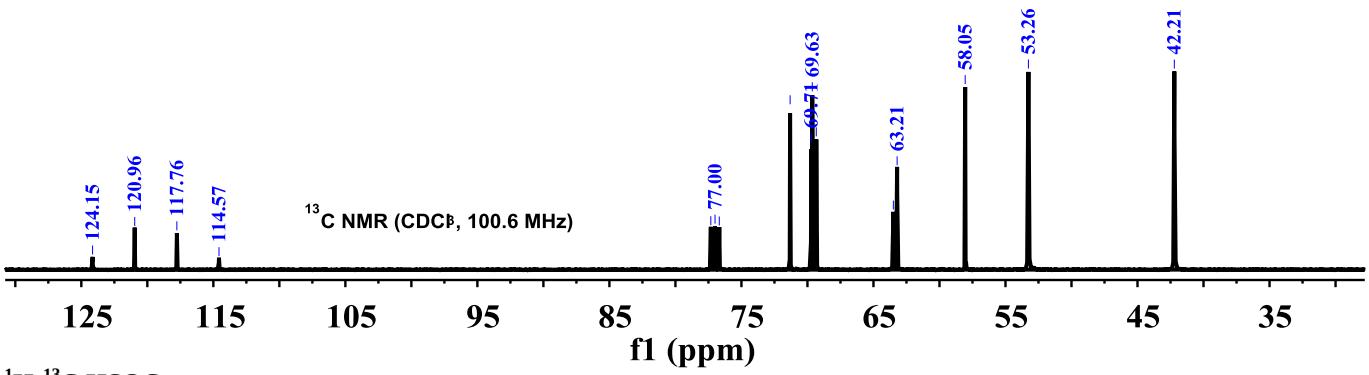
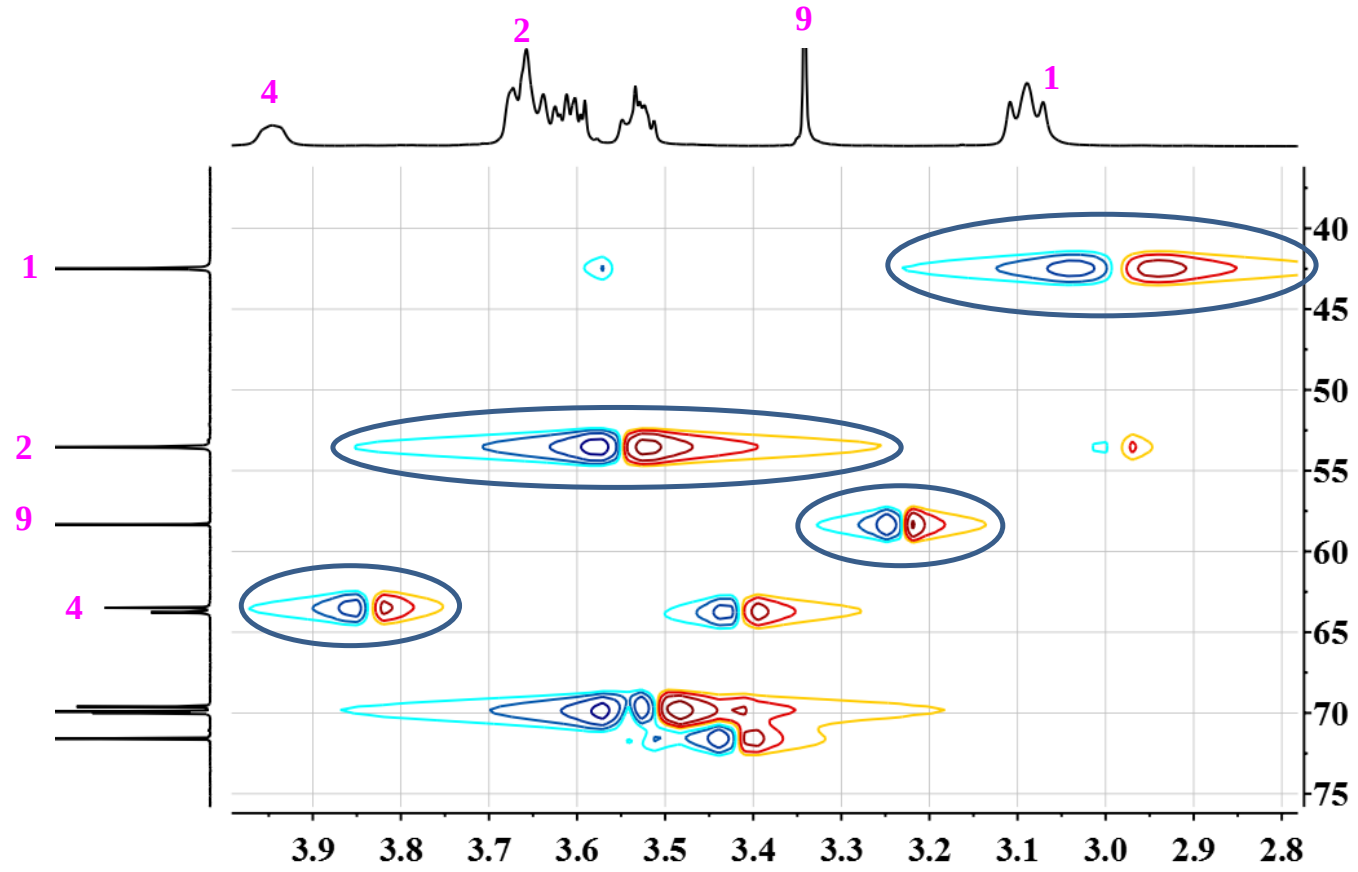
3.6
3.7
3.8
3.9
4.0
4.1
4.2
3.90
3.65
3.40
3.15


124.15
120.96
117.76
114.57
^{13}C NMR (CDCl$_3$, 100.6 MHz)
77.00
69.71
69.63
63.21
58.05
53.26
42.21
125
115
105
95
85
75
65
55
45
35
f1 (ppm)

^{1}H-^{13}C HSQC


2
9
4
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1
2
9
4
40
45
50
55
60
65
70
75
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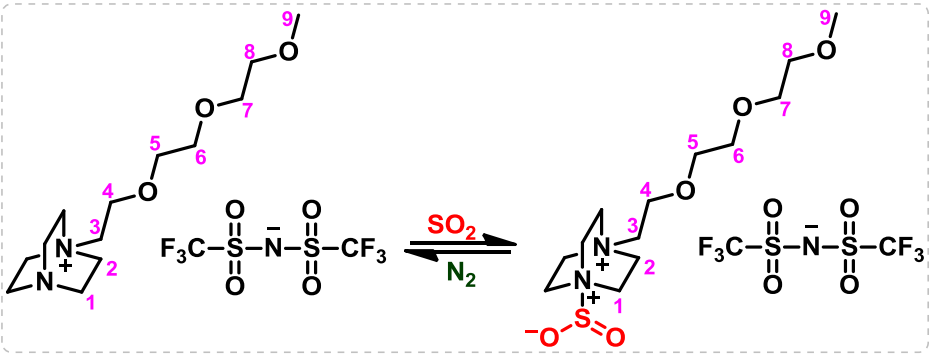
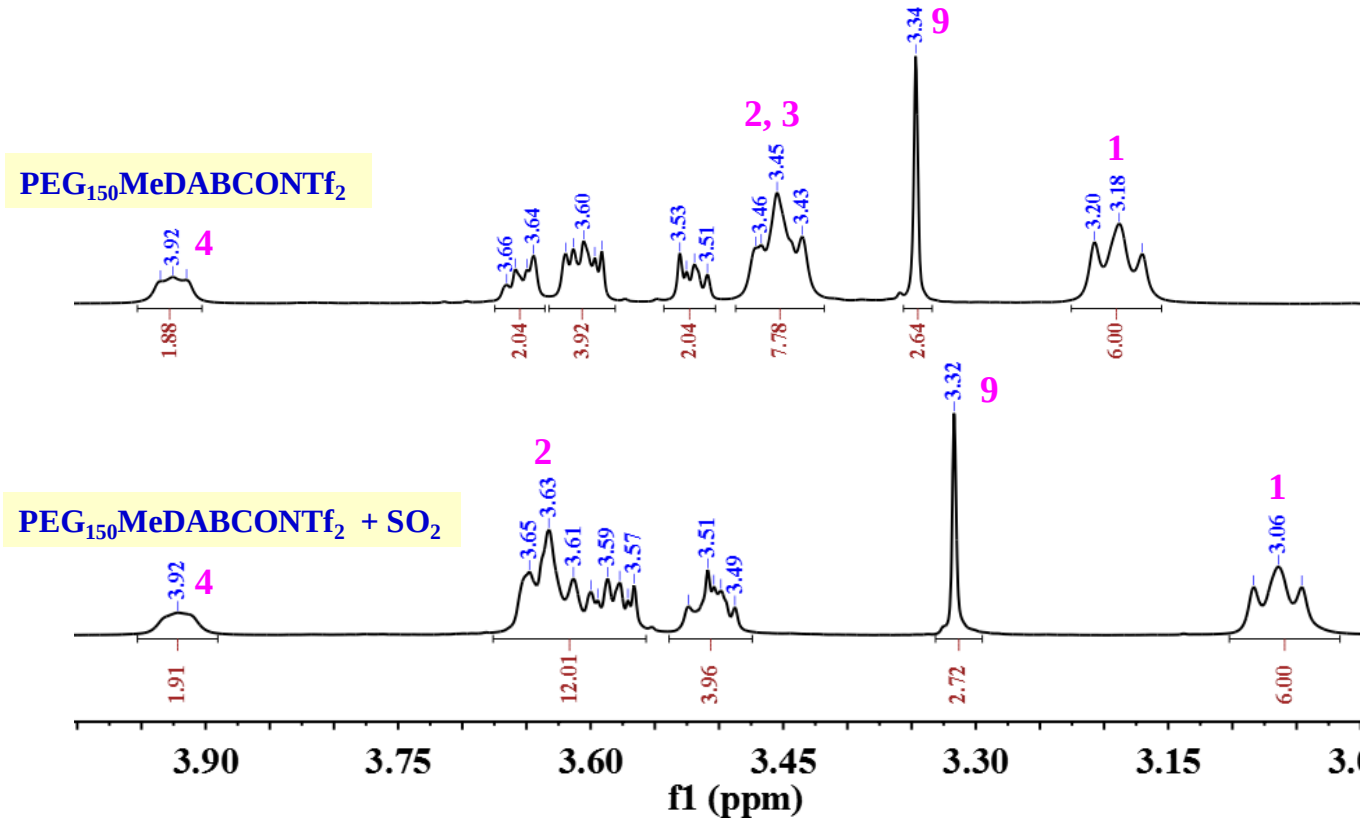
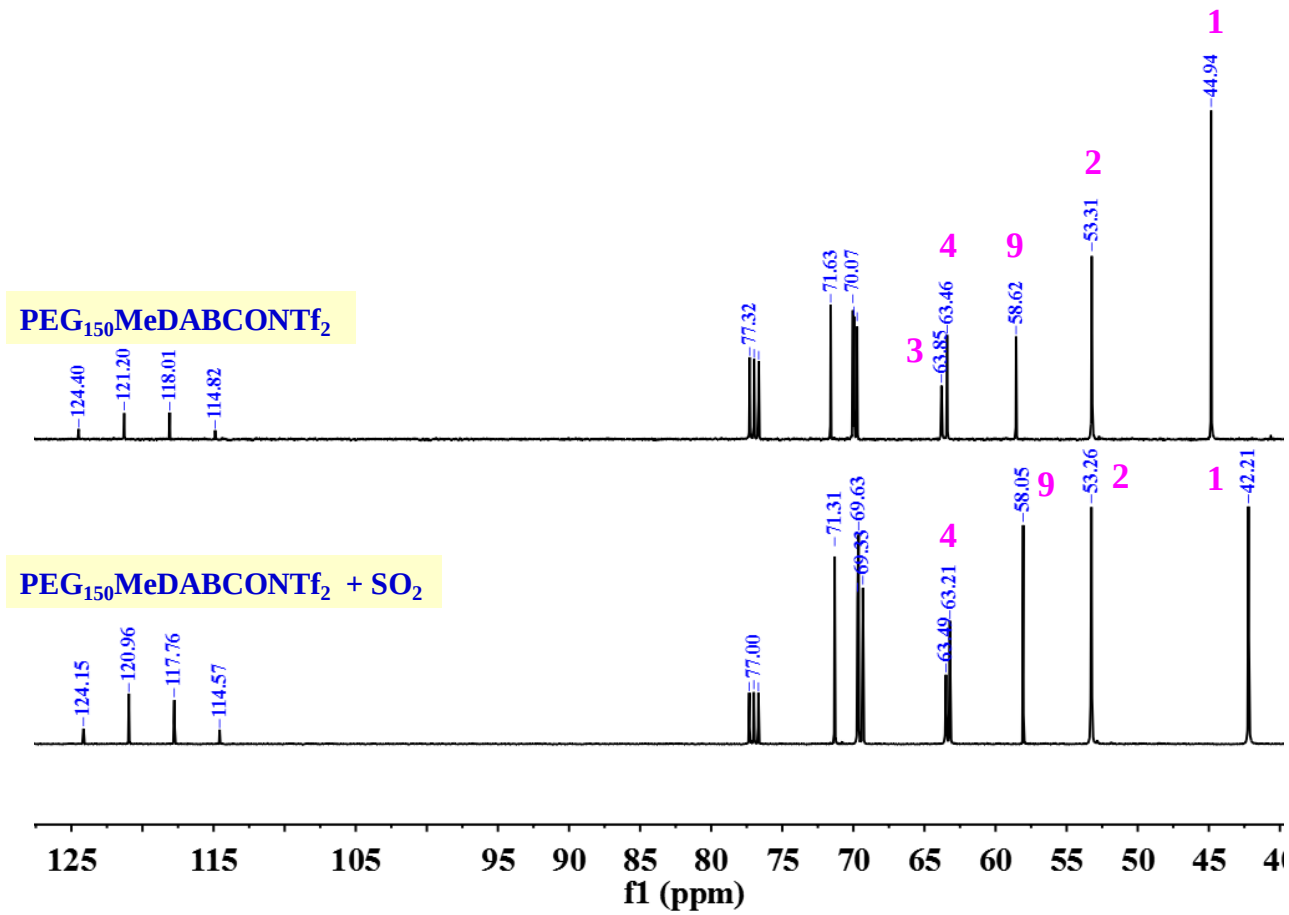
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
2.8


SO$_2$
N$_2$

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

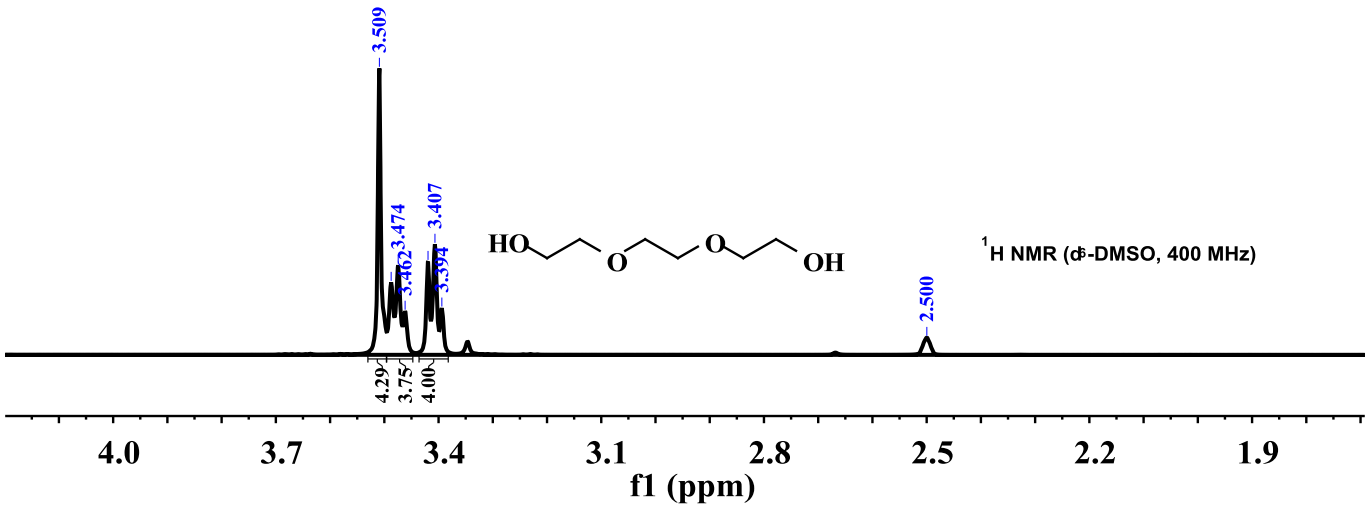


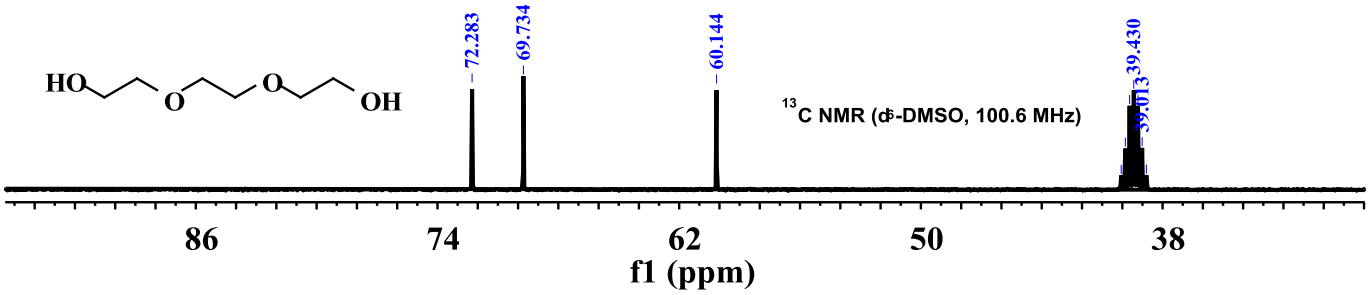


4. Characterization (^{1}H, ^{13}C, ^{19}F NMR) of other absorption systems before and after SO_2 capture

PEG_{150}

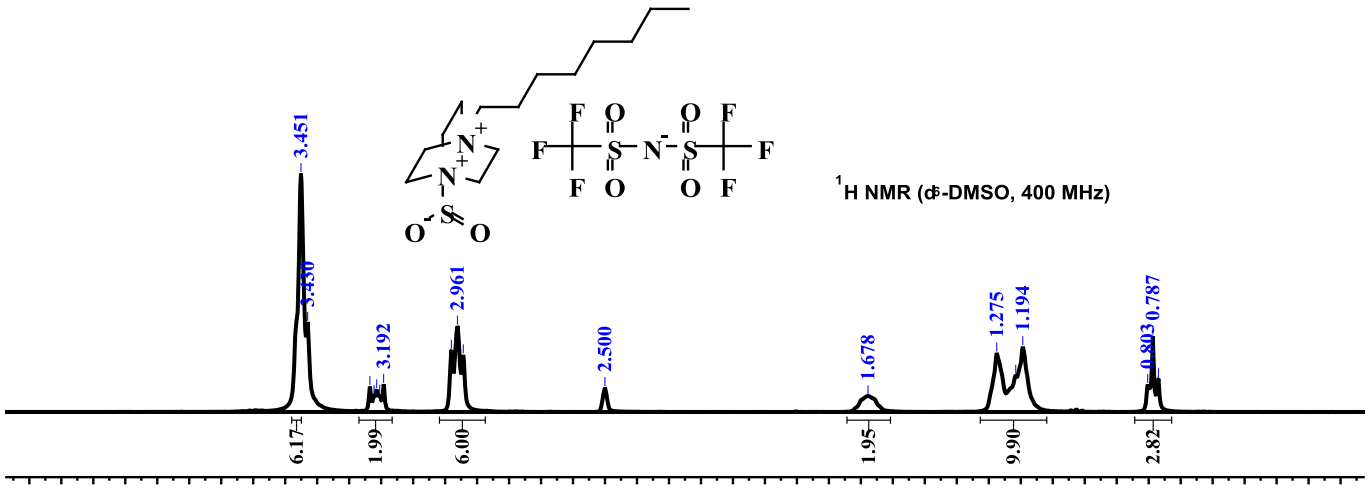
^{1}H NMR (400 MHz, d_6-DMSO) δ 4.59 (t, ^3J = 5.2 Hz, 2 H), 3.51 (s, 4 H), 3.47 (t, ^3J = 5.2 Hz, 4 H), 3.41 (t, ^3J = 5.2 Hz, 4 H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 72.3, 69.7, 60.1.

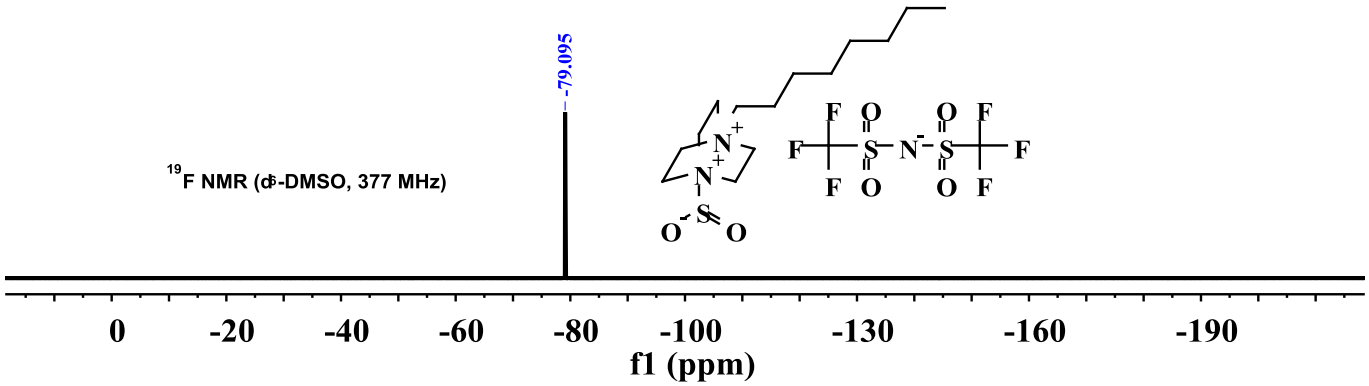


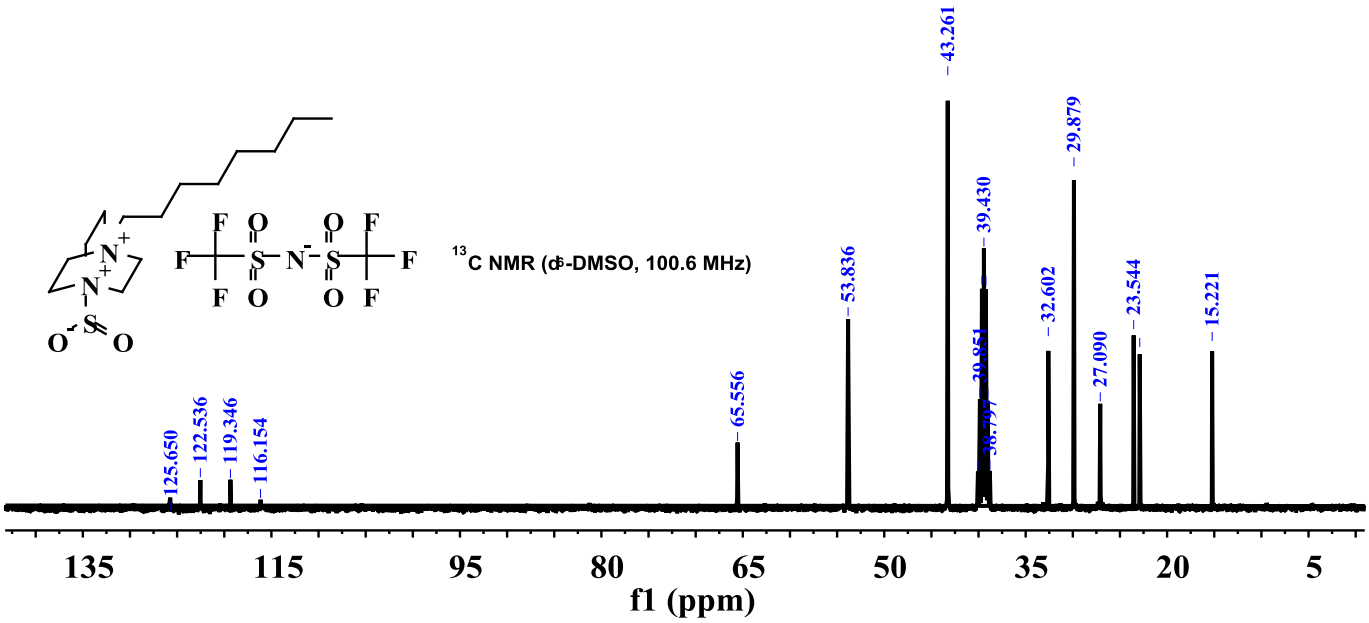


C_8DABCONTf$_2$ + SO_2

^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 3.45 (s, 6H), 3.19-3.23 (m, 2H), 2.96 (t, ^3J = 7.6 Hz, 6H), 1.68 (s, 2H), 1.19-1.27 (m, 10H), 0.79 (t, ^3J = 6.4 Hz, 3H); ^{19}F NMR (d$_6$-DMSO, 377 MHz) δ -78.74; ^{13}C NMR (d$_6$-DMSO, 100.6 MHz) δ 125.7, 122.5, 119.3, 116.1, 65.6, 53.8, 43.3, 32.6, 29.9, 27.1, 23.5, 22.9, 15.2.

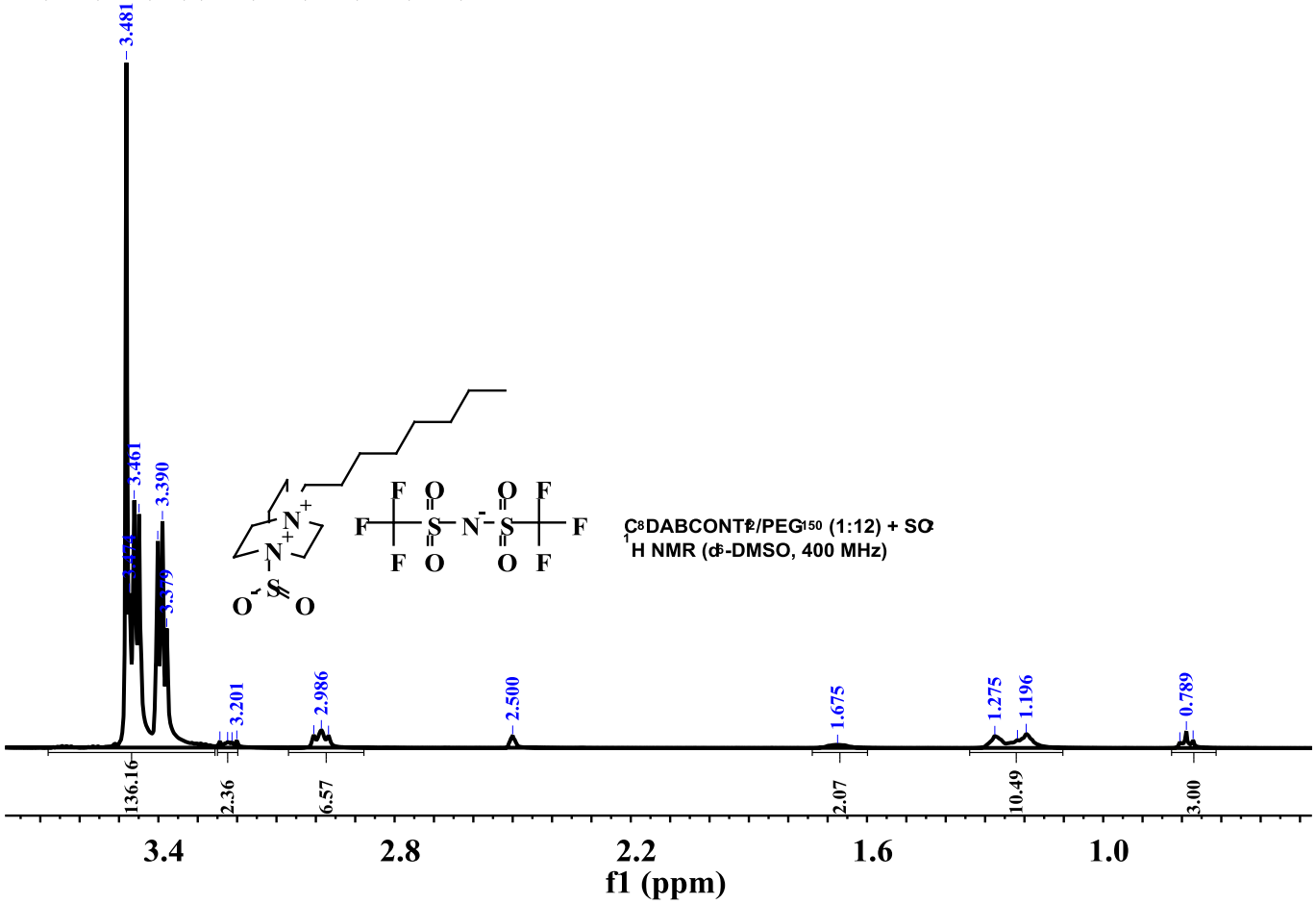


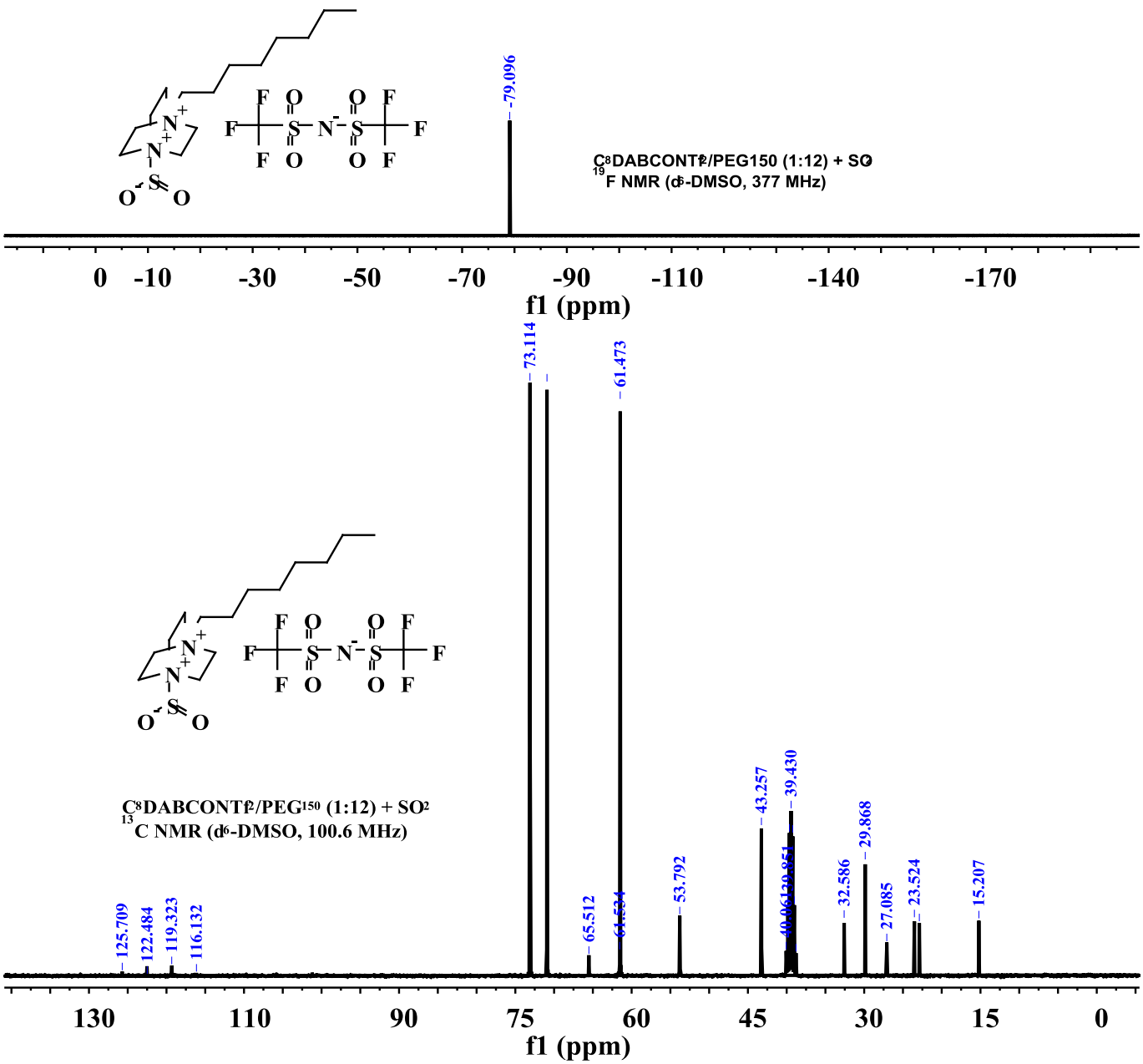




C_8DABCONTf$_2$/PEG150 (1:12) + SO_2

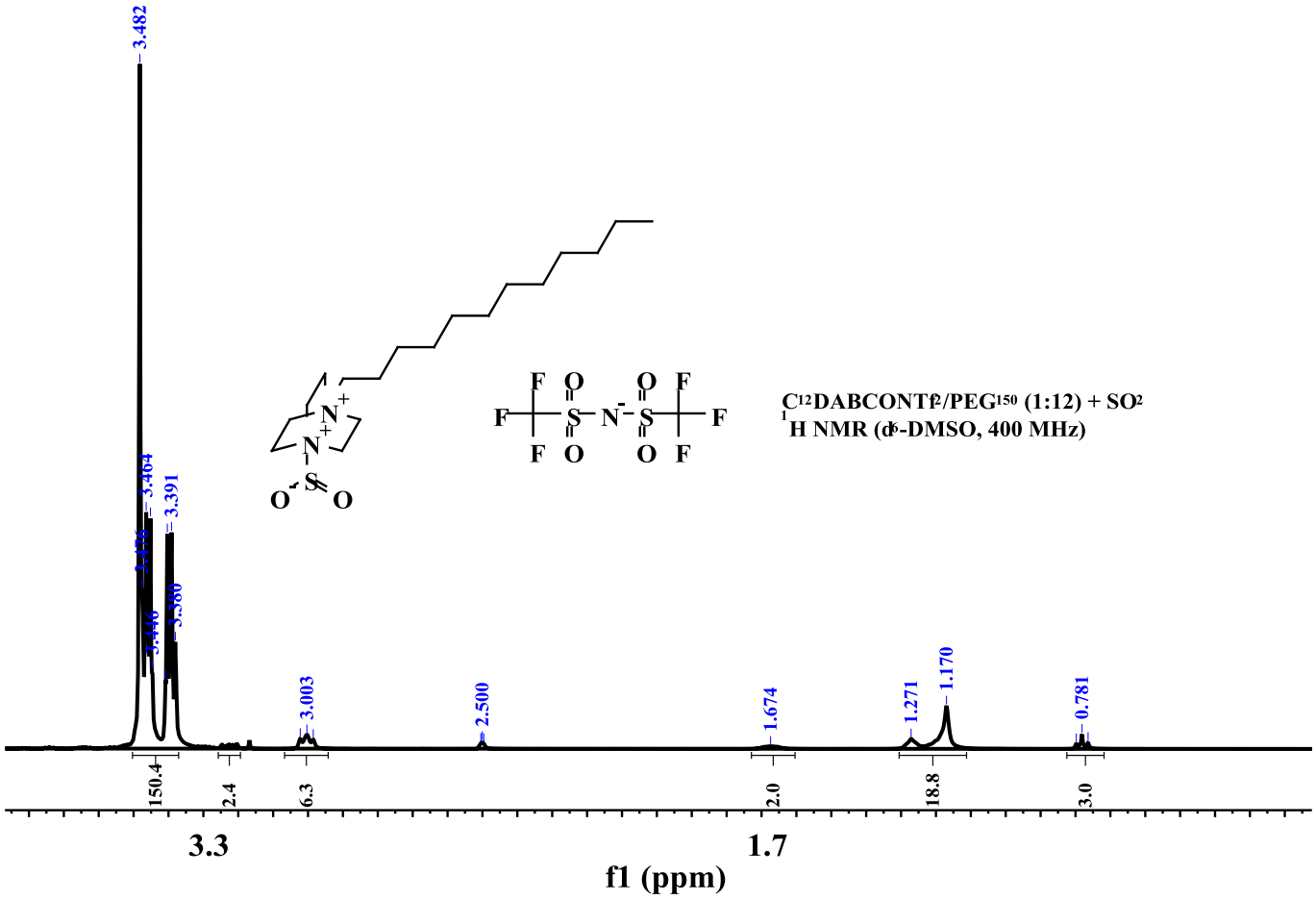
^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.38-3.48 (m, 136H), 3.20-3.24 (m, 2H), 2.99 (t, ^3J = 7.6 Hz, 6H), 1.67 (s, 2H), 1.20-1.28 (m, 10 H), 0.79 (t, ^3J = 6.4 Hz, 3H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -79.10; ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 125.7, 122.5, 119.3, 116.1, 73.1, 70.9, 65.5, 61.5, 53.8, 43,3, 32.6, 29.8, 27.1, 23.5, 22.9, 15.2.

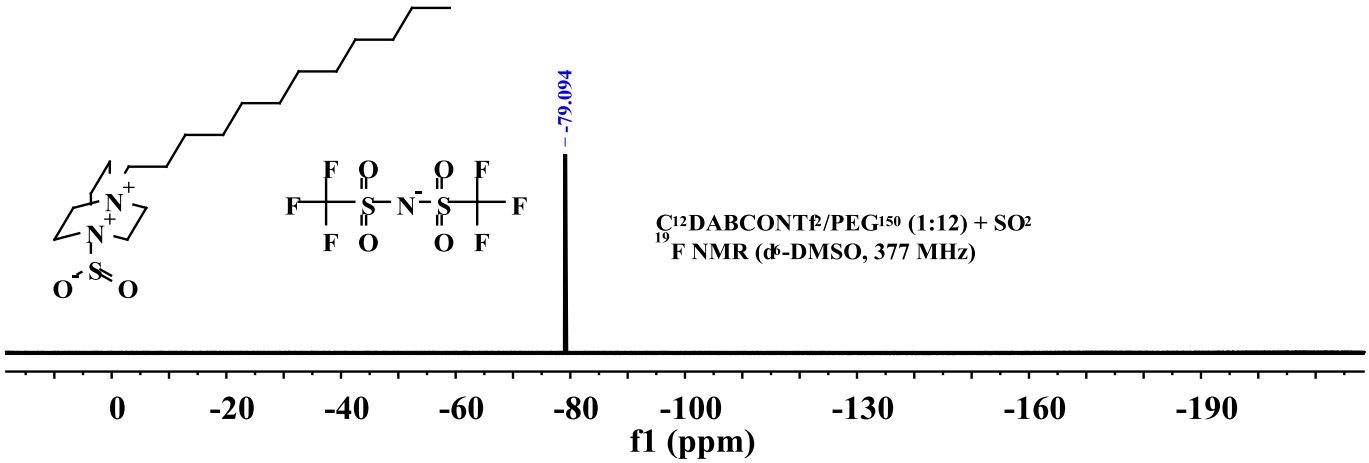


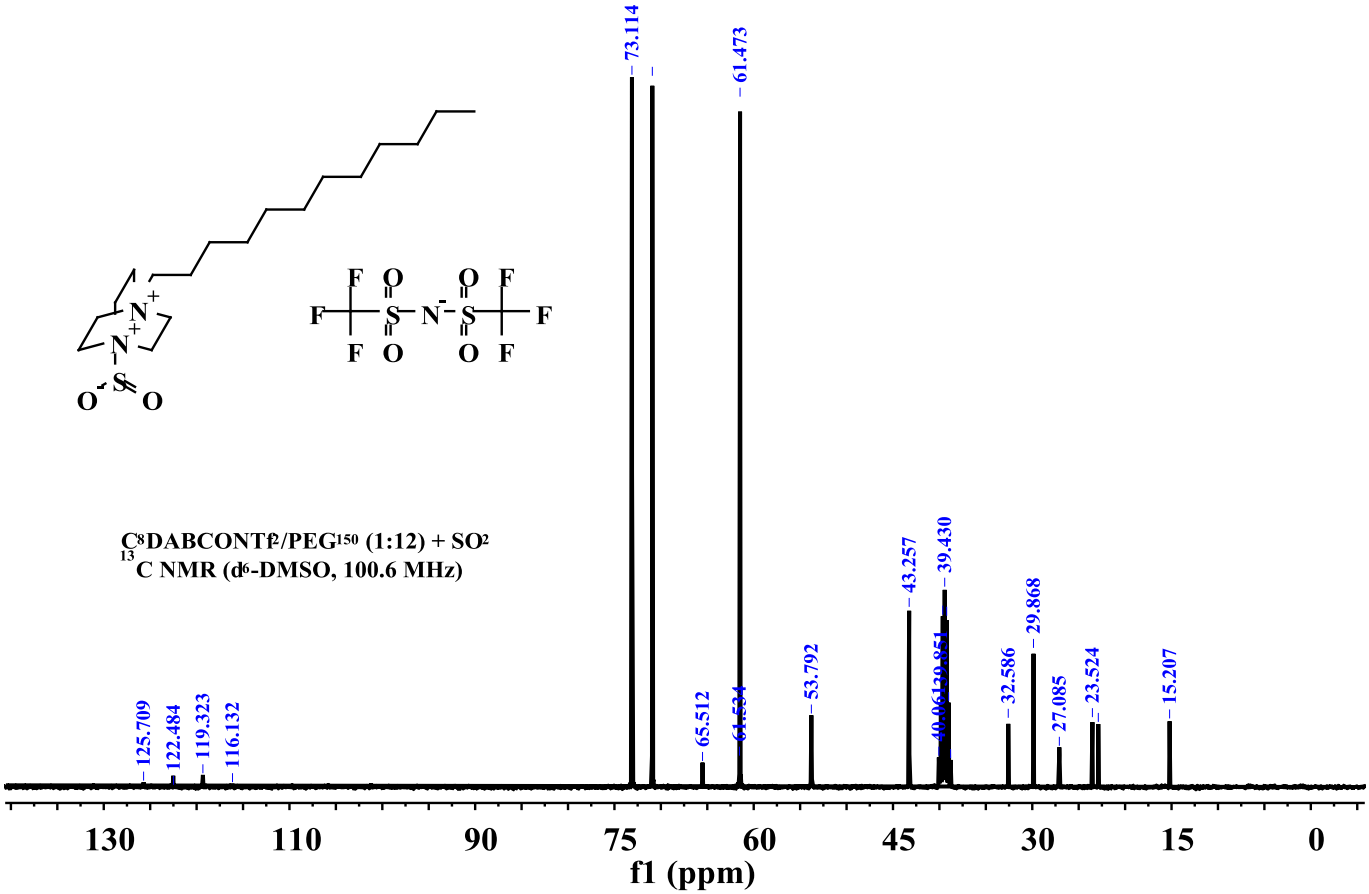


C_{12}DABCONTf_2/PEG150 (1:12) + SO_2

^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.45-3.49 (m, 150H), 3.38-3.41 (m, 2H), 3.01 (t, ^3J = 7.6 Hz, 6H), 1.68 (s, 2H), 1.17-1.28 (m, 18H), 0.79 (t, ^3J = 6.4 Hz, 3H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -79.09; ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 125.6, 122.4, 119.2, 116.0, 79.6, 79.3, 78.9, 73.1, 70.9, 65.4, 61.5, 53.5, 43.2, 32.7, 30.4, 30.3, 30.2, 29.9, 27.0, 23.5, 22.8, 15.1.

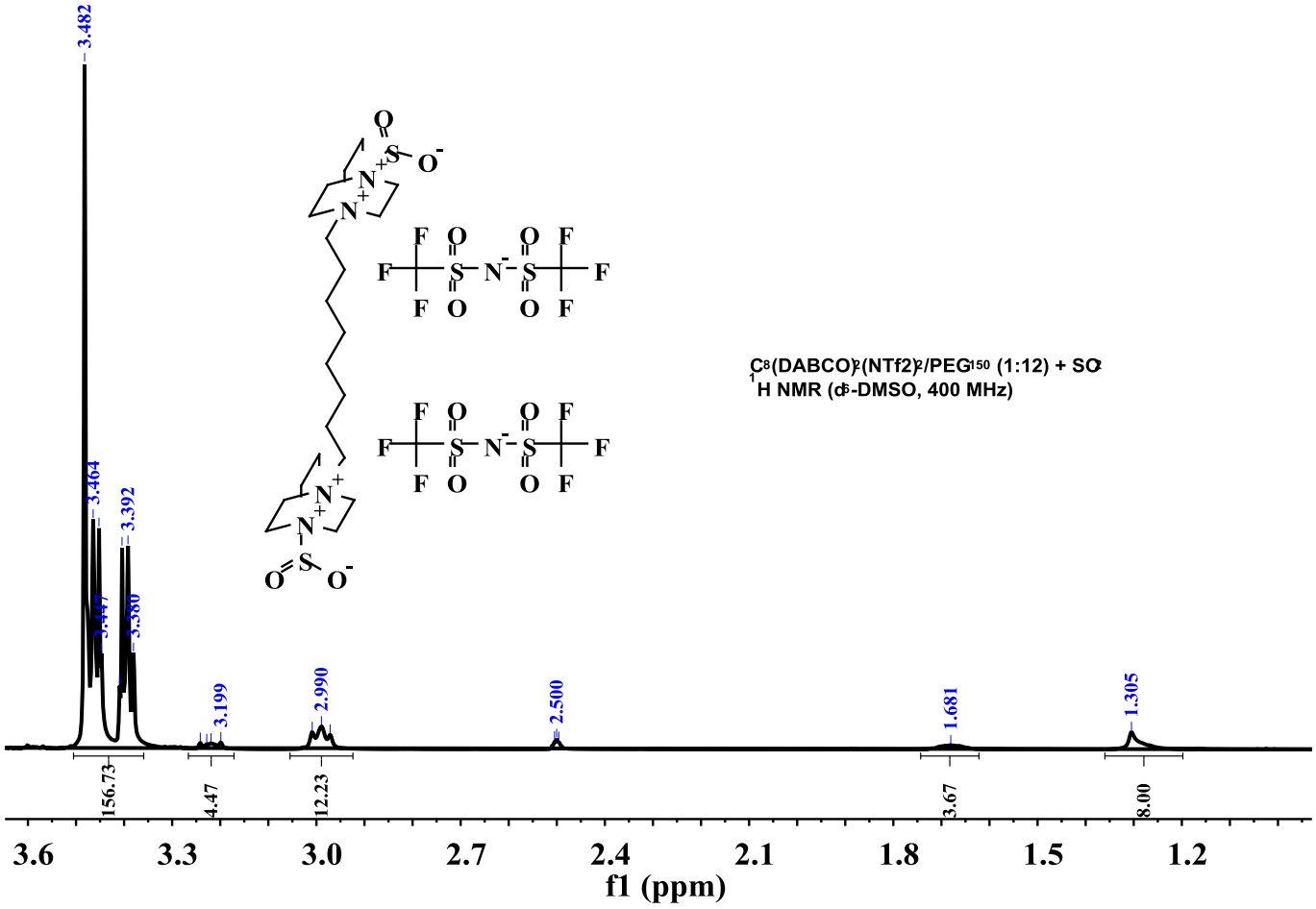

3.482
3.404
3.391
3.380
3.446
3.003
2.500
1.674
1.271
1.170
0.781
150.4
2.4
6.3
2.0
18.8
3.0
3.3
1.7
f1 (ppm)
C12DABCONTf2/PEG150 (1:12) + SO2
1H NMR (d6-DMSO, 400 MHz)

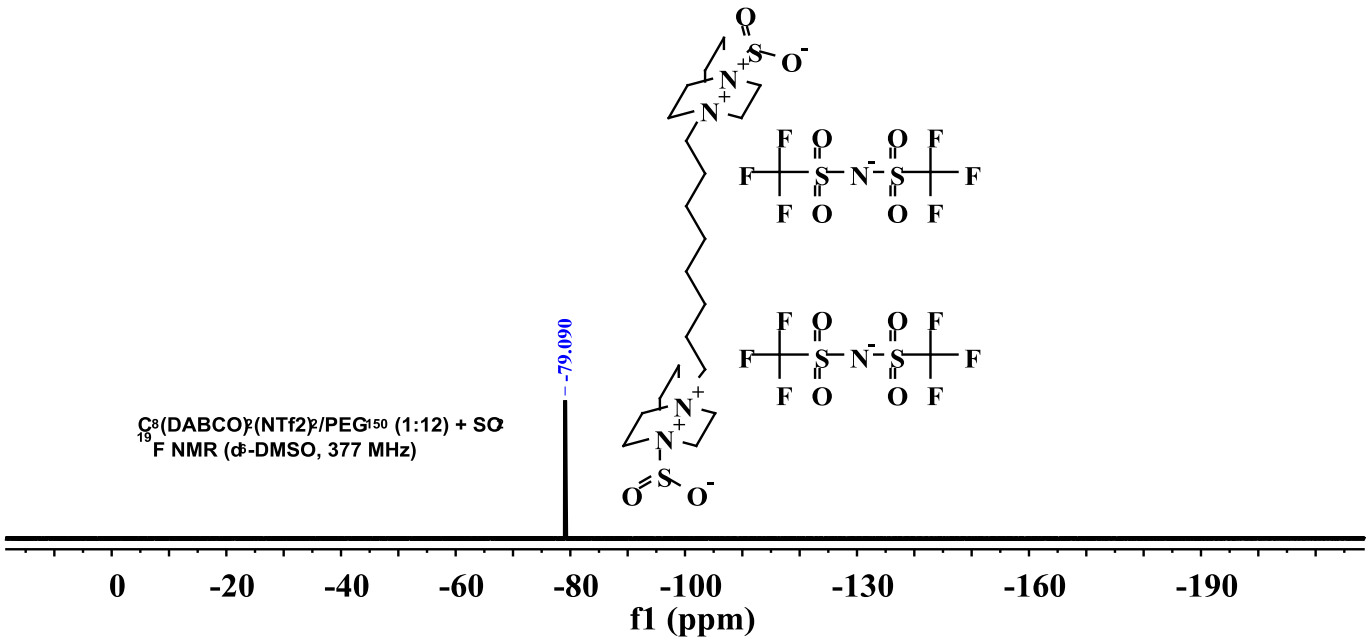

-79.094
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)
C12DABCONTf2/PEG150 (1:12) + SO2
19F NMR (d6-DMSO, 377 MHz)

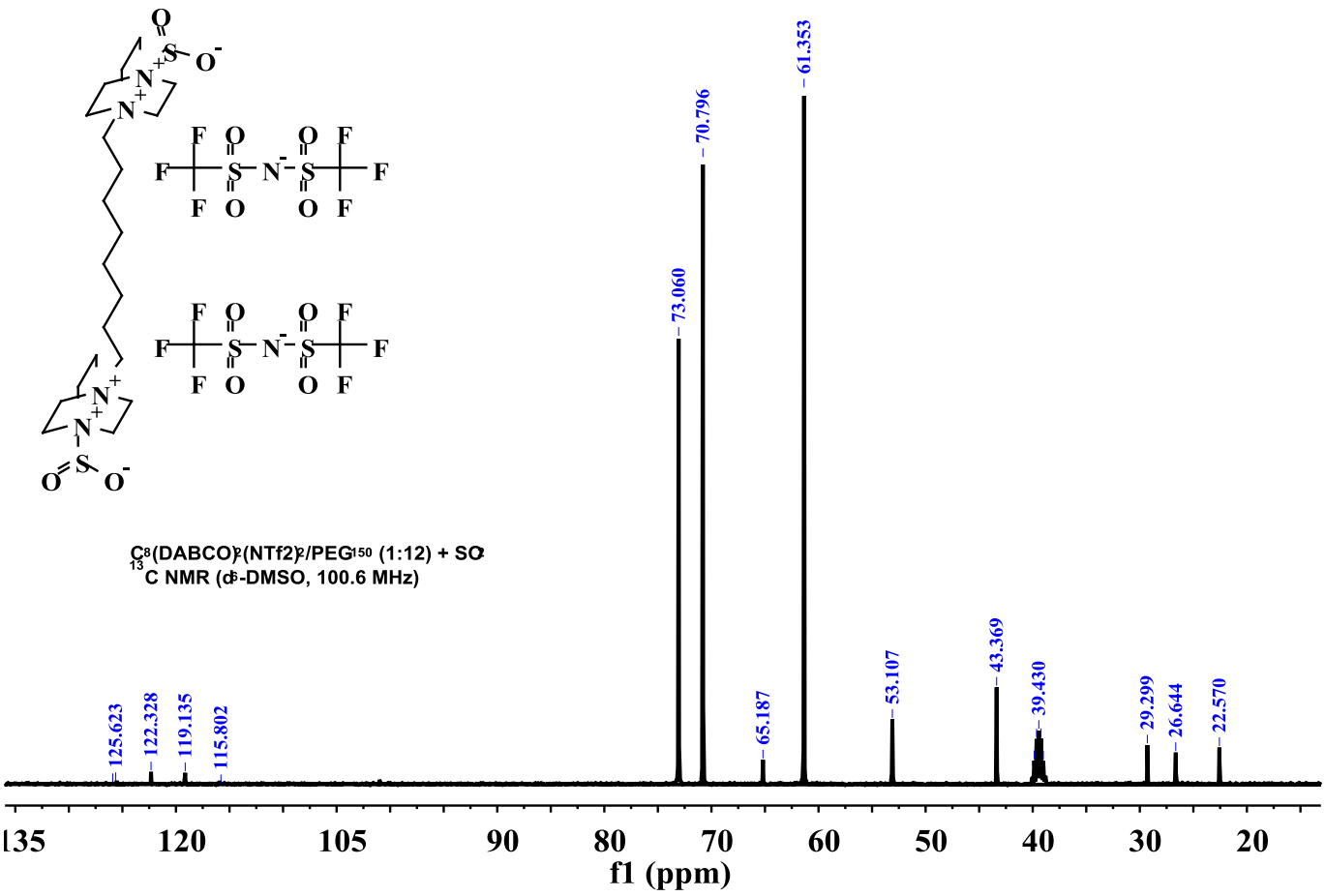


$C_8(DABCO)_2(NTf_2)_2/PEG_{150}$ (1:12) + SO_2

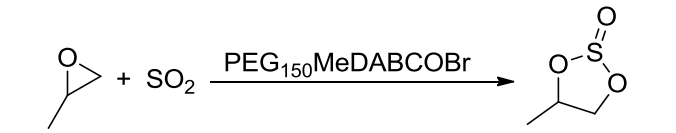
^{1}H NMR (d_6-DMSO, 400 MHz) δ 3.38-3.48 (m, 156H), 3.20-3.24 (m, 4H), 2.99 (t, ^{3}J = 7.6 Hz, 12H), 1.68 (s, 4H), 1.30 (s, 4H); ^{19}F NMR (d_6-DMSO, 377 MHz) δ -79.09; ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 125.5, 122.3, 119.1, 115.9, 73.1, 70.8, 65.2, 61.4, 53.1, 43.4, 29.3, 26.6, 22.6.


3.482
3.464
3.392
3.380
3.199
2.990
2.500
1.681
1.305
156.73
4.47
12.23
3.67
8.00
3.6
3.3
3.0
2.7
2.4
2.1
1.8
1.5
1.2
f1 (ppm)
C8(DABCO)2(NTf2)2/PEG150 (1:12) + SO
1H NMR (d6-DMSO, 400 MHz)


-79.090
0
-20
-40
-60
-80
-100
-130
-160
-190
f1 (ppm)
C8(DABCO)2(NTf2)2/PEG150 (1:12) + SO
19F NMR (d6-DMSO, 377 MHz)



5. Reaction conditions screening for the synthesis of propylene sulfite from propylene oxide and SO_2 catalyzed by PEG_{150}MeDABCOBr



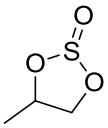
propylene oxide (PO)

Table S1 Reaction conditions screening for the synthesis of propylene sulfite (PS) from propylene oxide (PO) and SO_2 catalyzed by PEG_{150}MeDABCOBr[a]

Entry	Temperature/°C	Catalyst loading/mol%	Time/h	PO Conv./%[b]	PS Yield/%[b]
1	25	1	3	46	1
2	60	1	3	69	8
3	80	1	3	86	11
4	100	1	3	88	28
5	120	1	3	96	41
6	140	1	3	99	24
7	120	0.5	3	98	13
8	120	3	3	>99	57
9	120	5	3	>99	48
10	120	3	1	94	33
11	120	3	12	>99	58

[a] Reaction conditions: PO, 10 mmol; SO2, 10 mmol. [b] Determined by GC with biphenyl as an internal standard.

6. Characterization (NMR, GC-MS) of cyclic sulfites



4-methyl-1,3,2-dioxathiolane 2-oxide

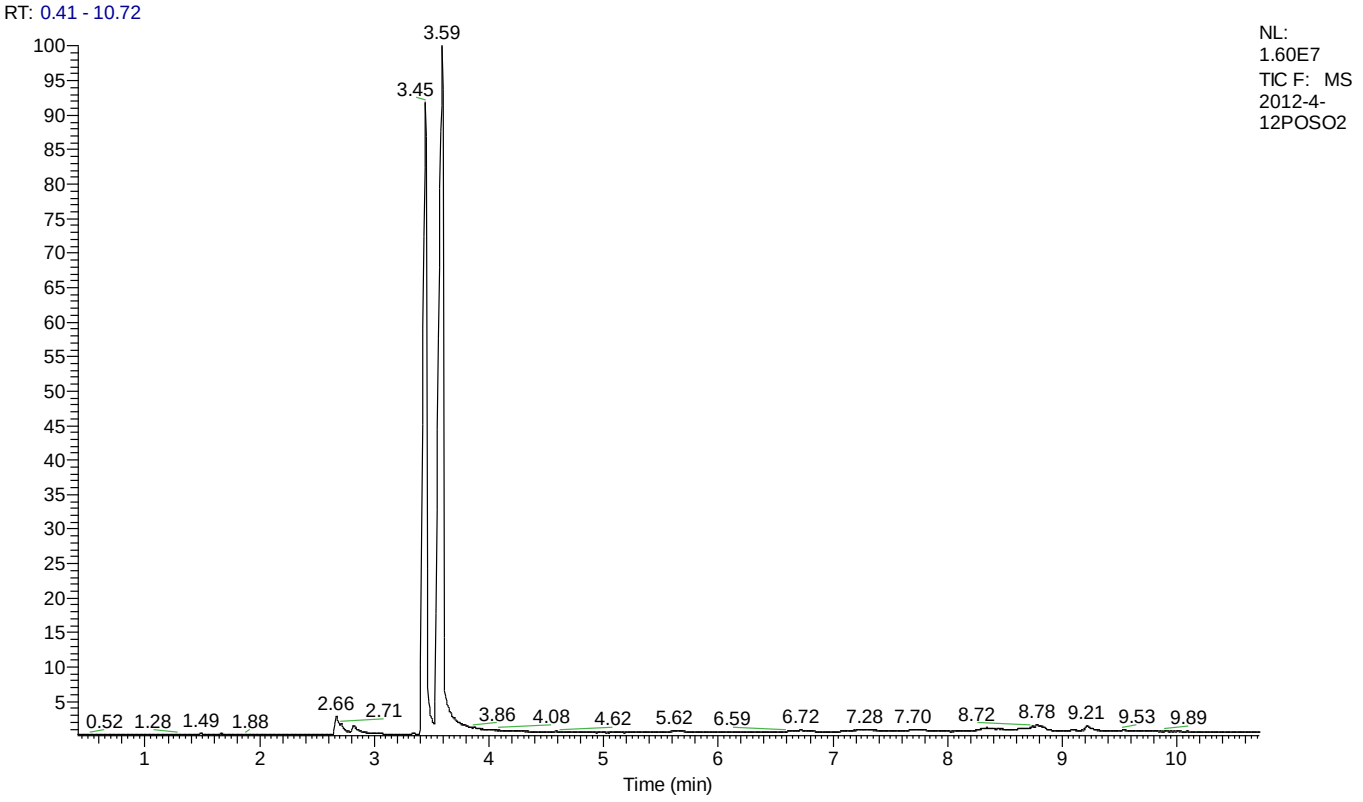
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 3:1; Light yellow liquid.

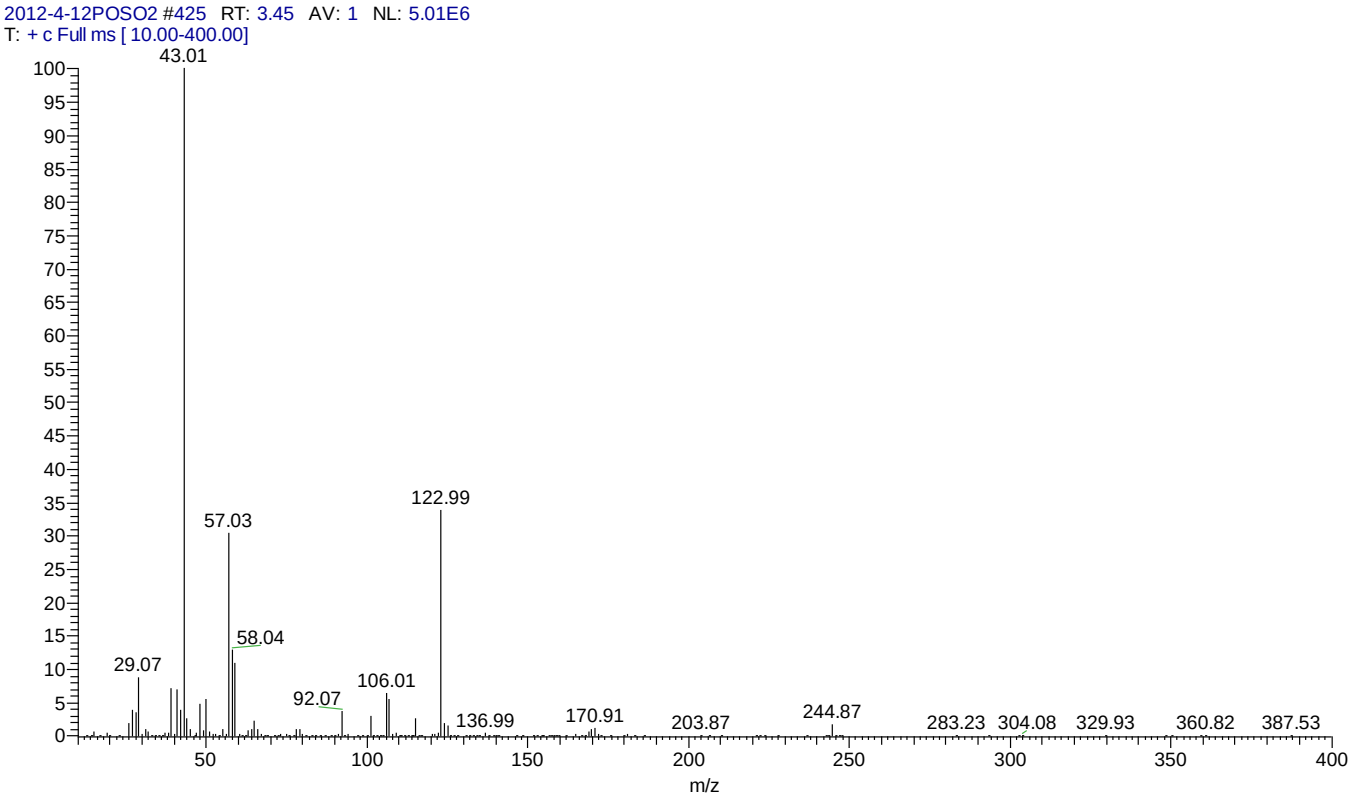
Isomer 1 (RT 3.45 min)

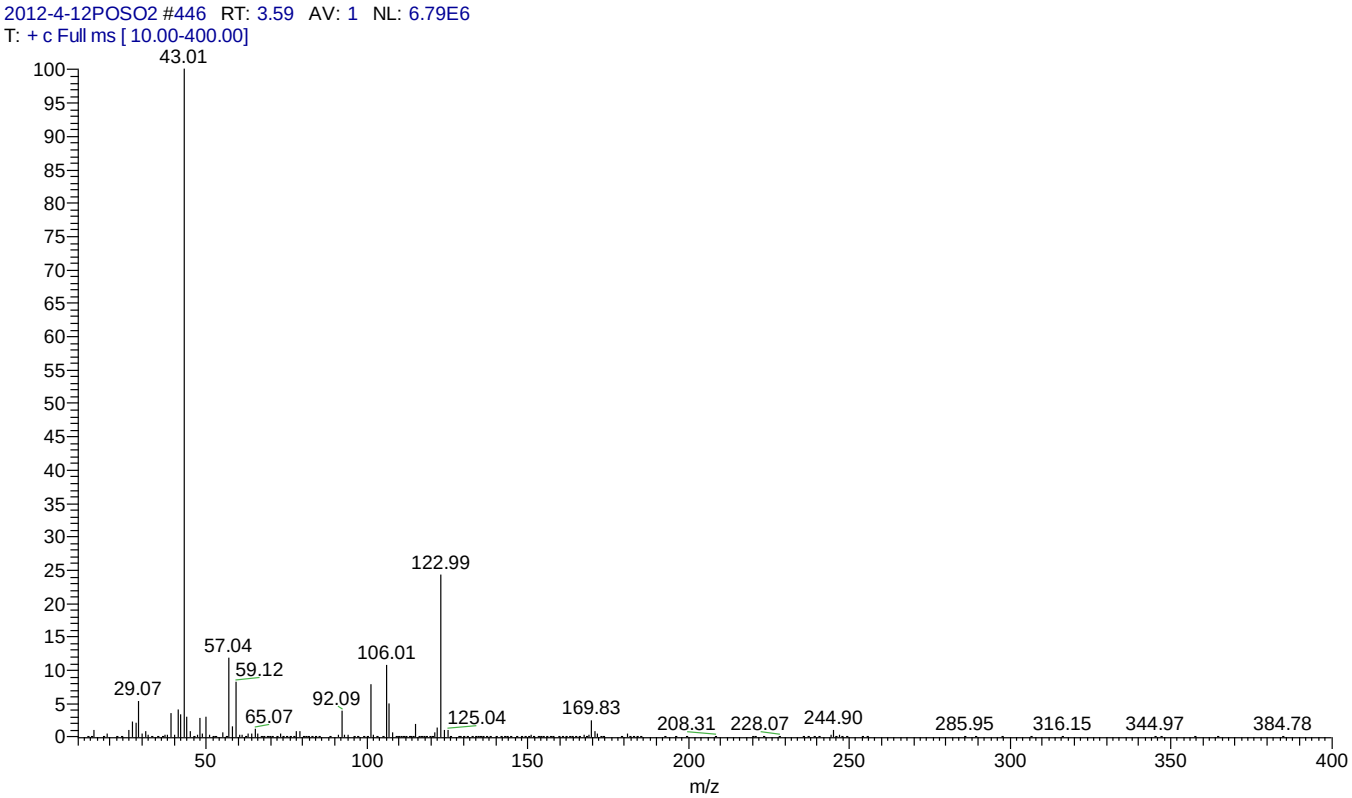
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.58-4.65 (m, 1H), 4.49-4.52 (m, 1H), 4.29 (t, ^{3}J = 8.8 Hz, 1H), 1.61 (d, ^{3}J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.2, 72.8, 18.7; GC-MS: m/z (%): 122.99 (34) [M^+], 57.03 (30) [M^+-O_2S], 43.01 (100) [M^+-O_3S].

Isomer 2 (RT 3.59 min)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.07-5.15 (m, 1H), 4.69-4.72 (m, 1H), 3.85-3.89 (m, 1H), 1.43 (d, ^{3}J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 17.6, 71.2, 76.4; GC-MS: m/z (%): 122.99 (24) [M^+], 57.04 (12) [M^+-O_2S], 43.01 (100) [M^+-O_3S].




2012-4-12POSO2 #425 RT: 3.45 AV: 1 NL: 5.01E6
T: + c Full ms [10.00-400.00]
43.01
122.99
57.03
58.04
29.07
106.01
92.07
136.99
170.91
203.87
244.87
283.23
304.08
329.93
360.82
387.53
m/z


2012-4-12POSO2 #446 RT: 3.59 AV: 1 NL: 6.79E6
T: + c Full ms [10.00-400.00]
43.01
122.99
57.04
59.12
106.01
29.07
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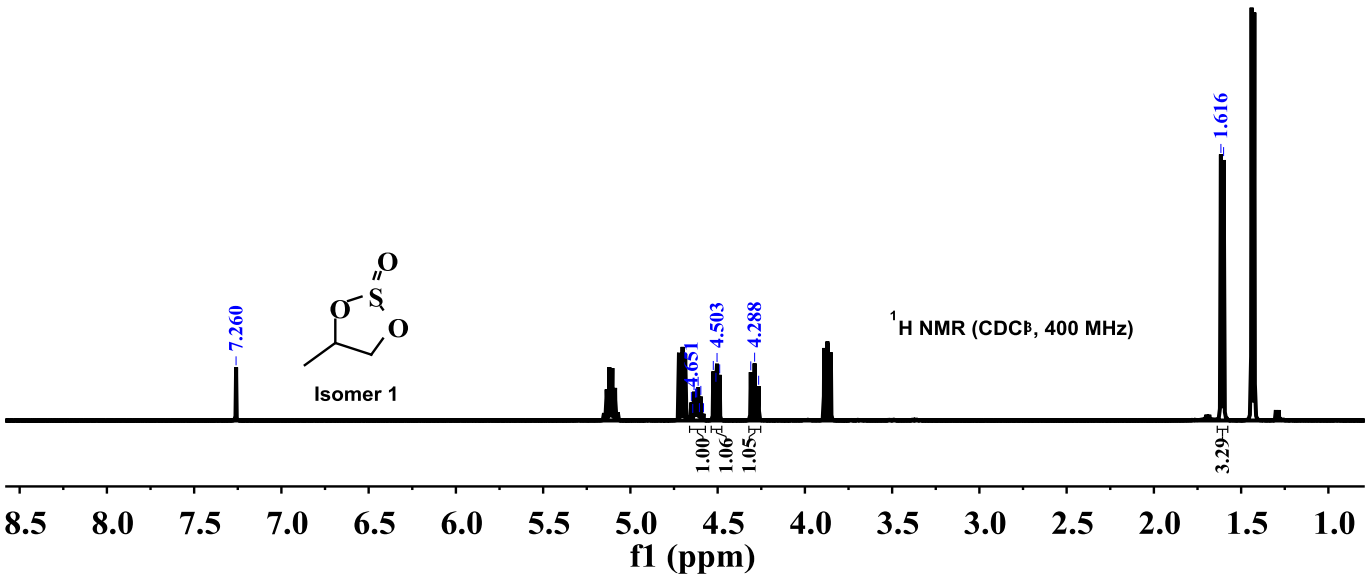
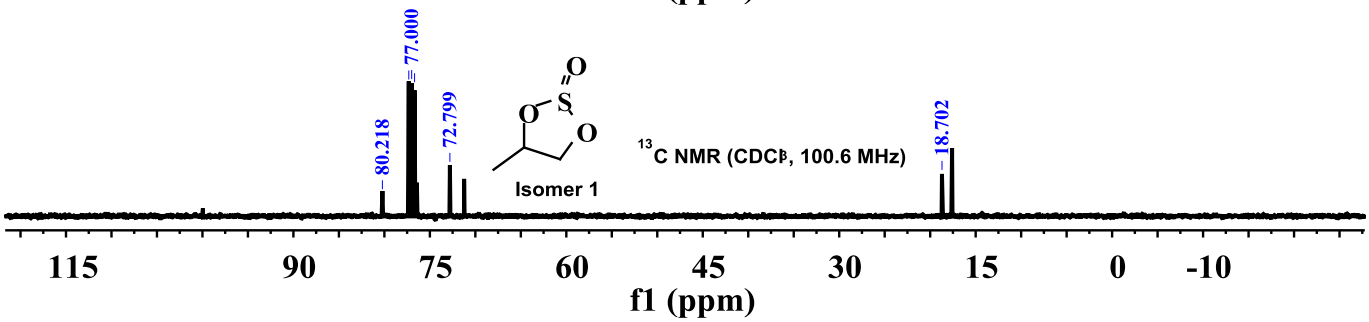
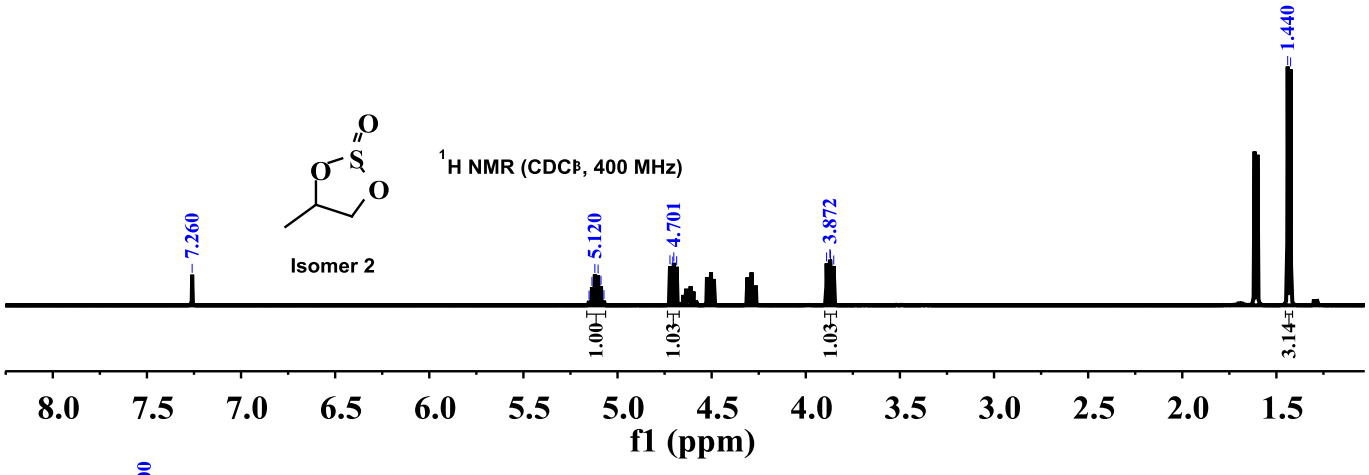
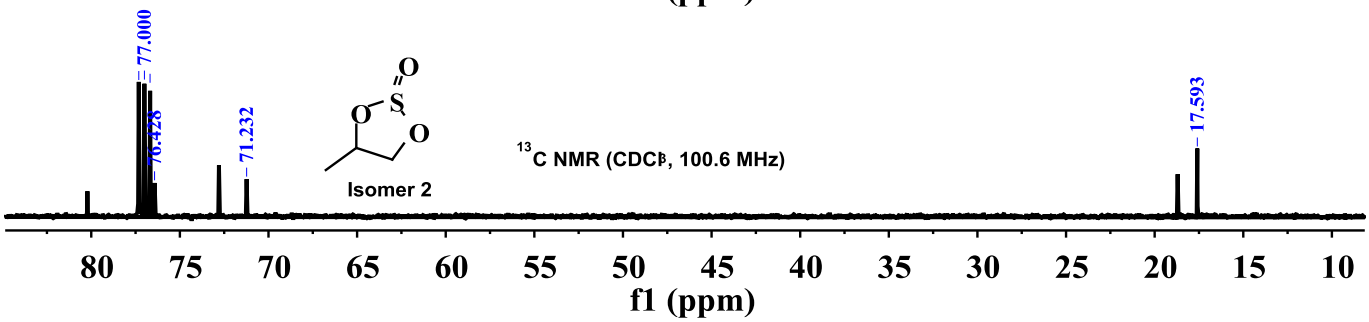
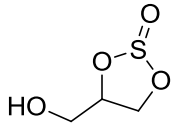
169.83
208.31
228.07
244.90
285.95
316.15
344.97
384.78
m/z











4-(hydroxymethyl)-1,3,2-dioxathiolane 2-oxide

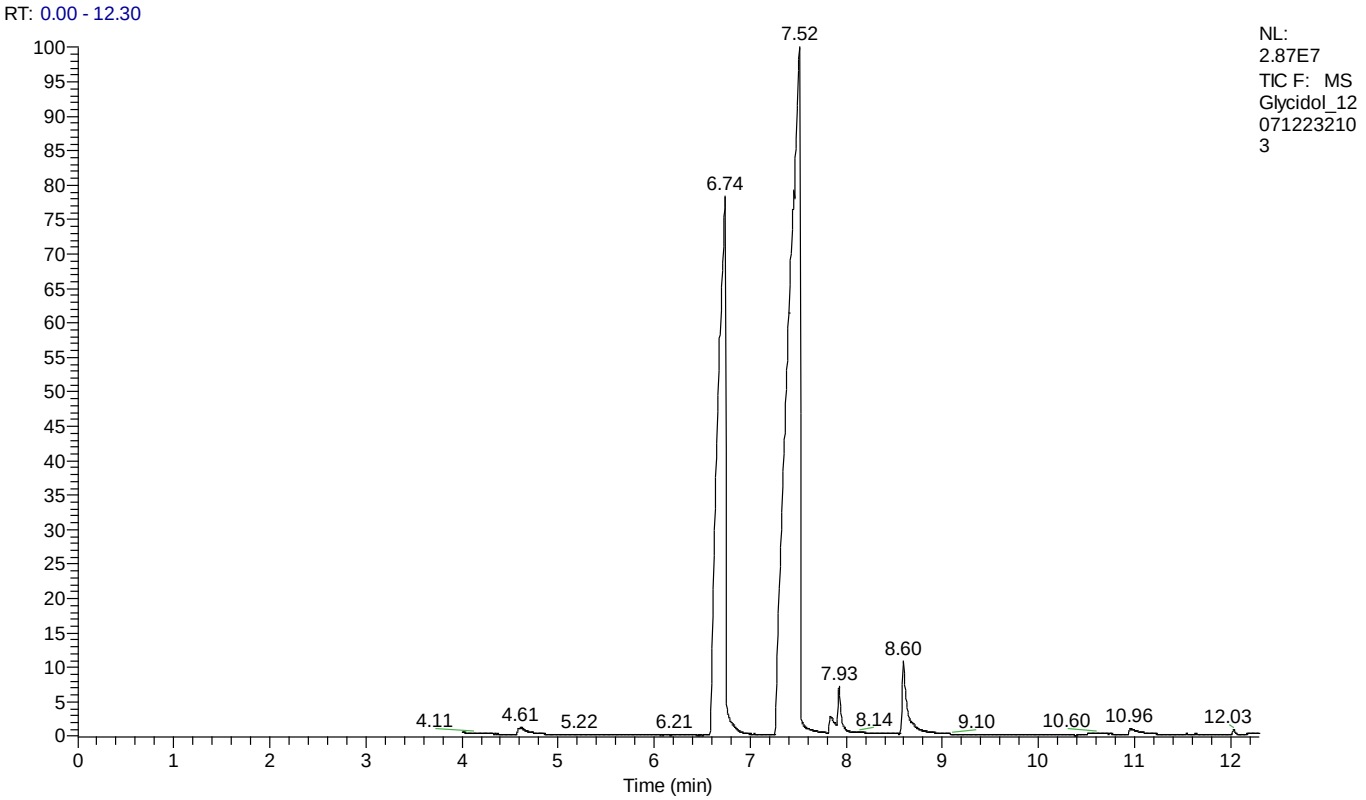
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 1:3; Light yellow liquid.

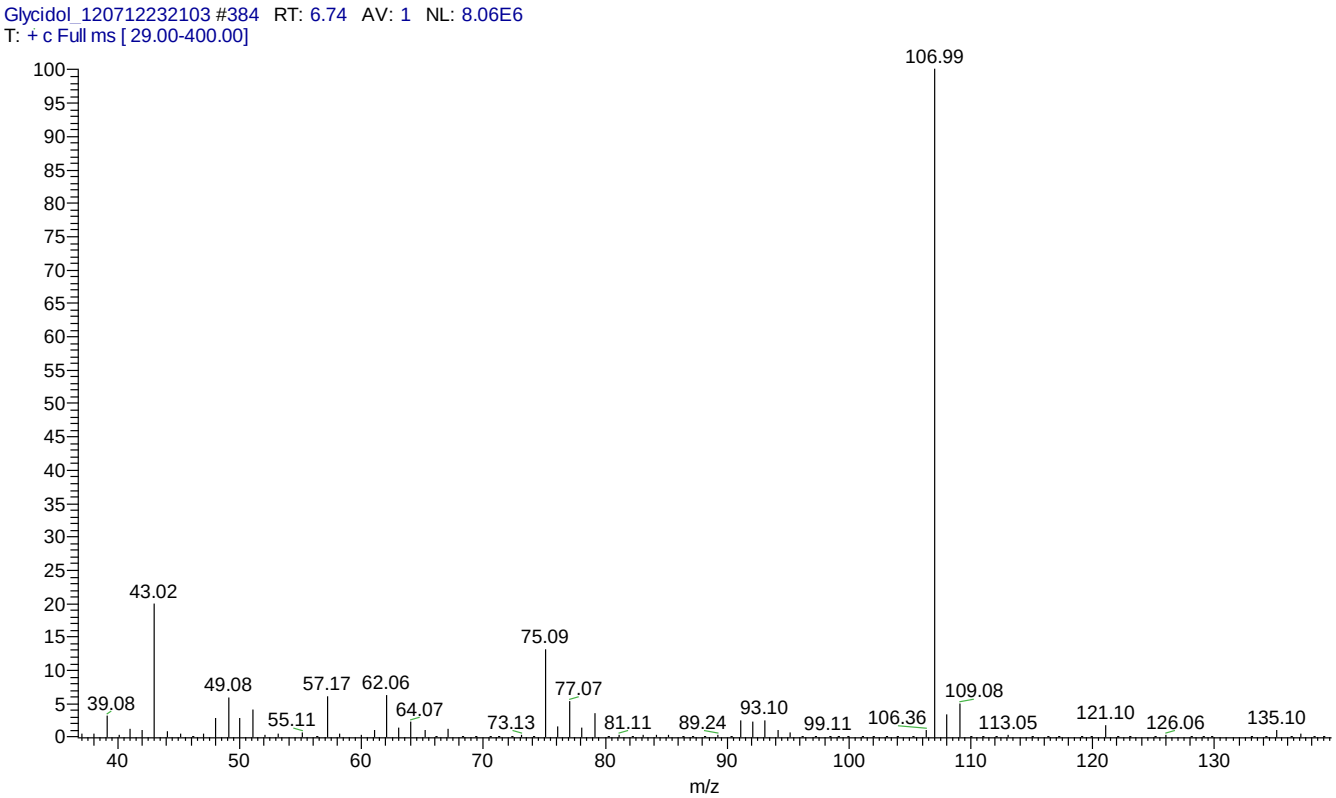
Isomer 1 (RT 6.74 min)

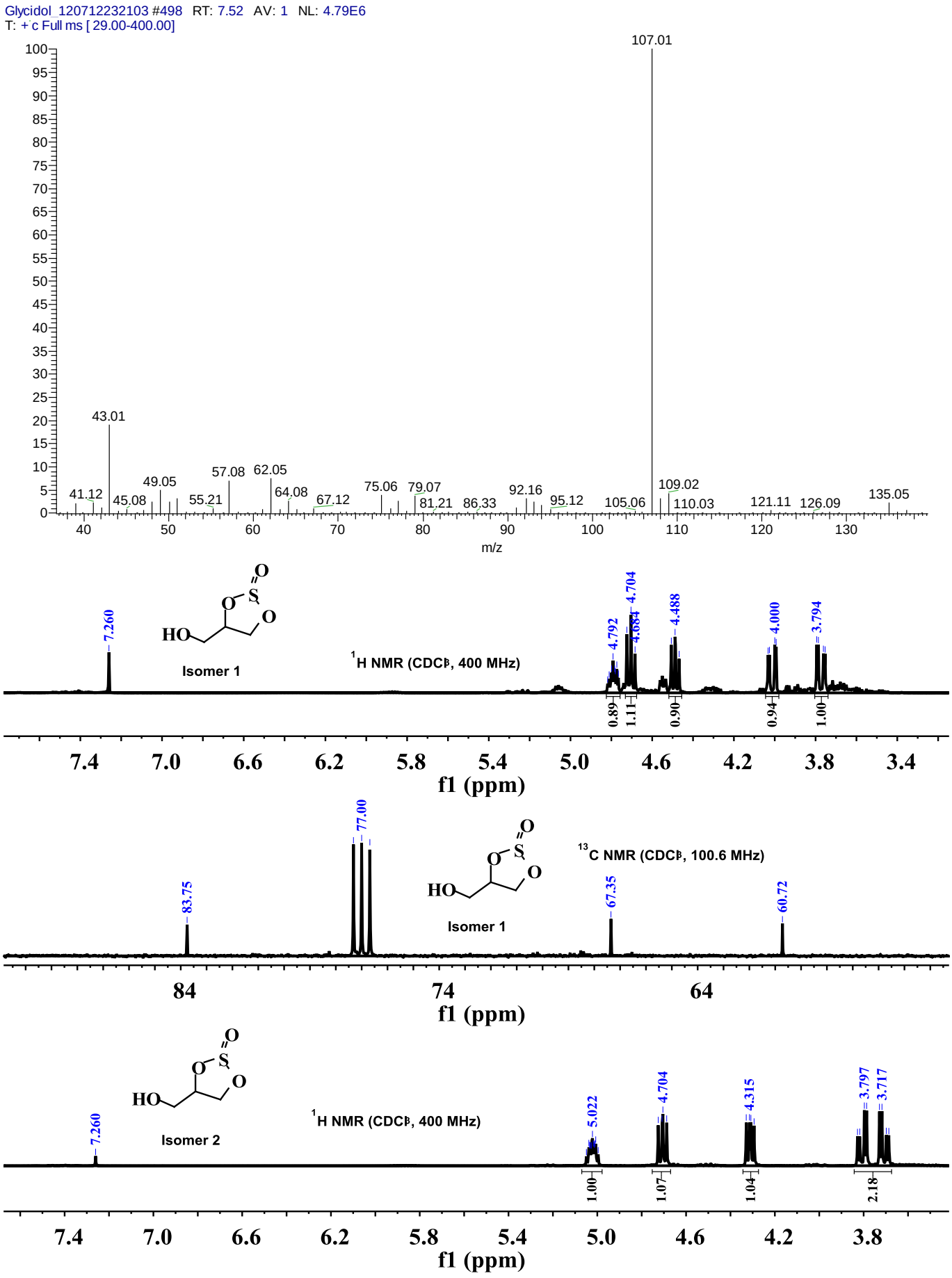
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.77-4.82 (m, 1H), 4.70 (t, ^{3}J = 8 Hz, 1H), 4.49 (t, ^{3}J = 7.2 Hz, 1H), 3.99-4.03 (m, 1H), 3.75-3.79 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 83.7, 67.3, 60.7; GC-MS: m/z (%):106.98 (100) [M^+-CH_3O].

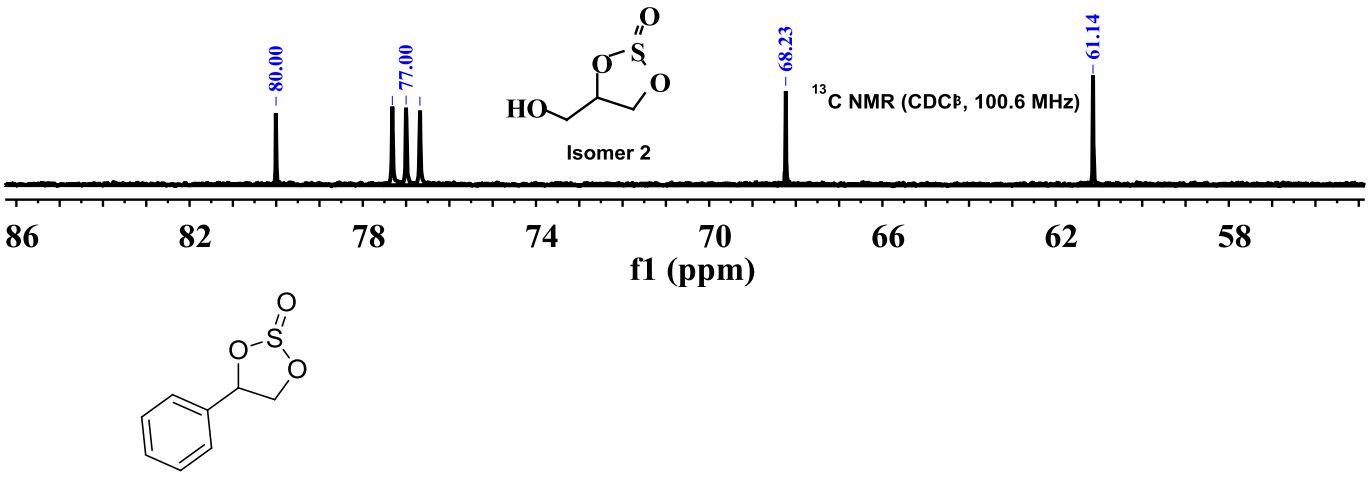
Isomer 2 (RT 7.52 min)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.00-5.05 (m, 1H), 4.69-4.72 (m, 1H), 4.29-4.33 (m, 1H), 3.69-3.83 (m, 2H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.0, 68.2, 61.1; GC-MS: m/z (%):107.01 (100) [M^+-CH_3O].






Glycidol_120712232103 #498 RT: 7.52 AV: 1 NL: 4.79E6
T: + c Full ms [29.00-400.00]
107.01
43.01
49.05
57.08
62.05
41.12
45.08
55.21
64.08
67.12
75.06
79.07
81.21
86.33
92.16
95.12
105.06
109.02
110.03
121.11
126.09
135.05
m/z
Isomer 1
^{1}H NMR (CDCβ, 400 MHz)
7.260
4.792
4.704
4.684
4.488
4.000
3.794
0.89
1.11
0.90
0.94
1.00
f1 (ppm)
^{13}C NMR (CDCβ, 100.6 MHz)
77.00
83.75
67.35
60.72
f1 (ppm)
Isomer 2
^{1}H NMR (CDCβ, 400 MHz)
7.260
5.022
4.704
4.315
3.797
3.717
1.00
1.07
1.04
2.18
f1 (ppm)



4-phenyl-1,3,2-dioxathiolane 2-oxide

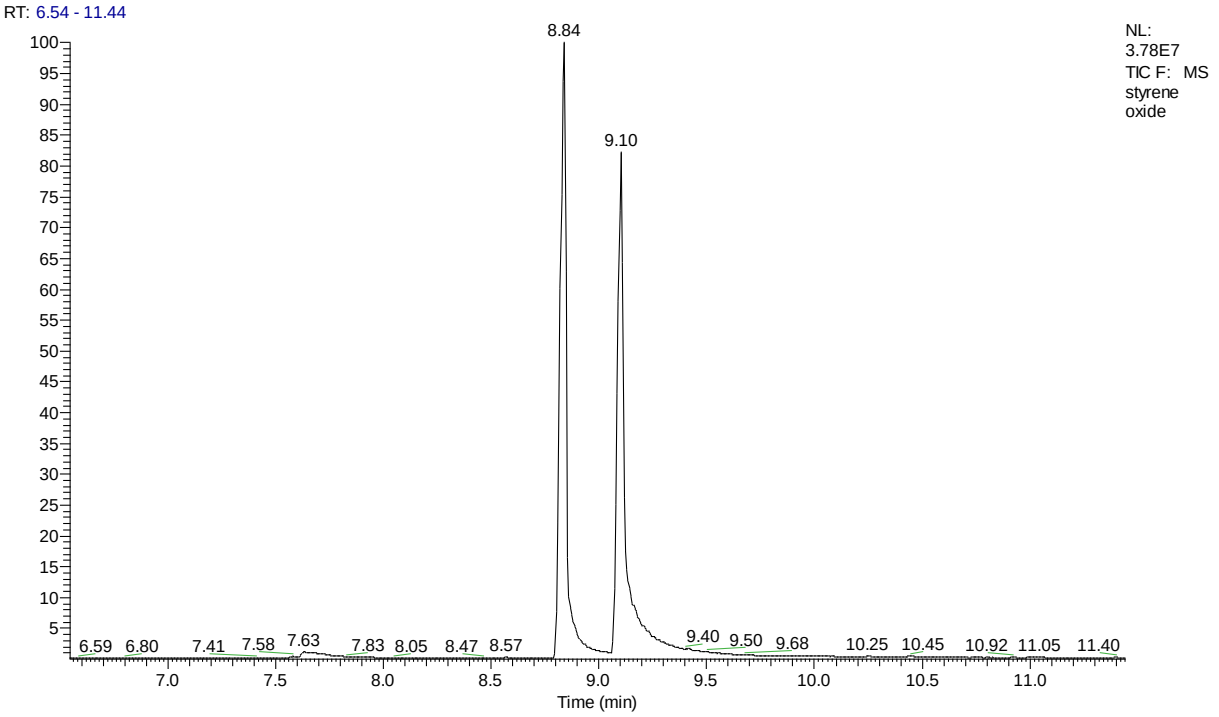
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 2:1; Light yellow liquid.

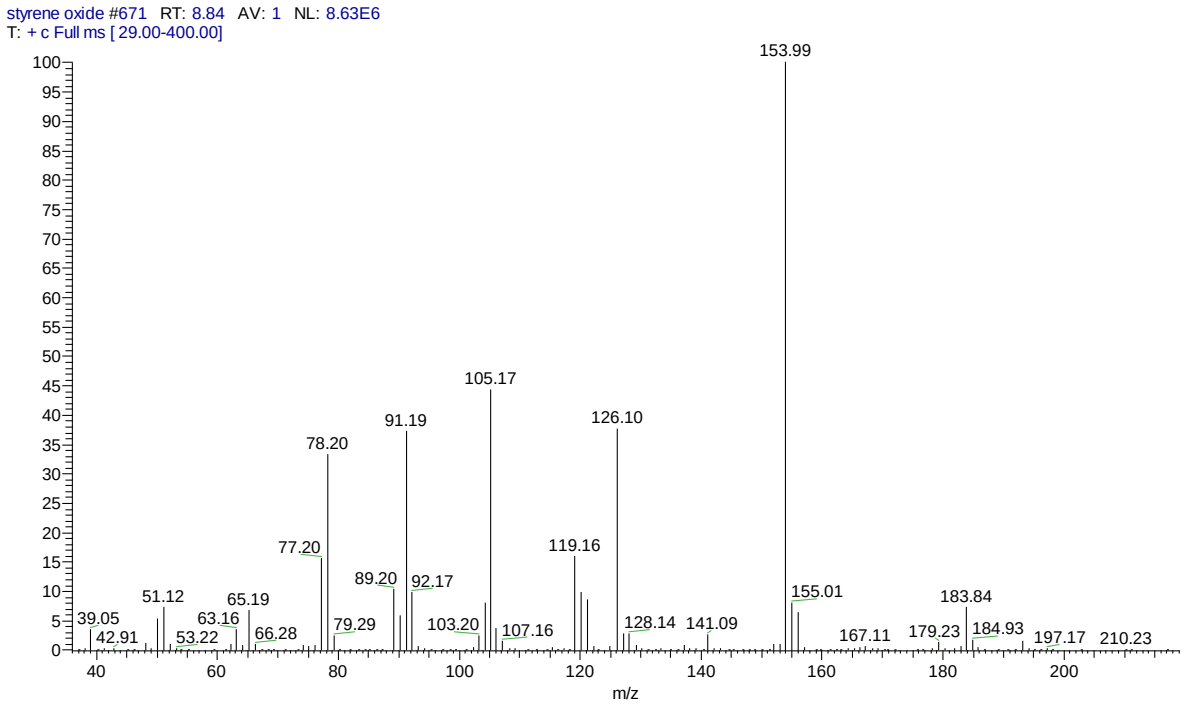
Isomer 1 (RT 8.84 min)

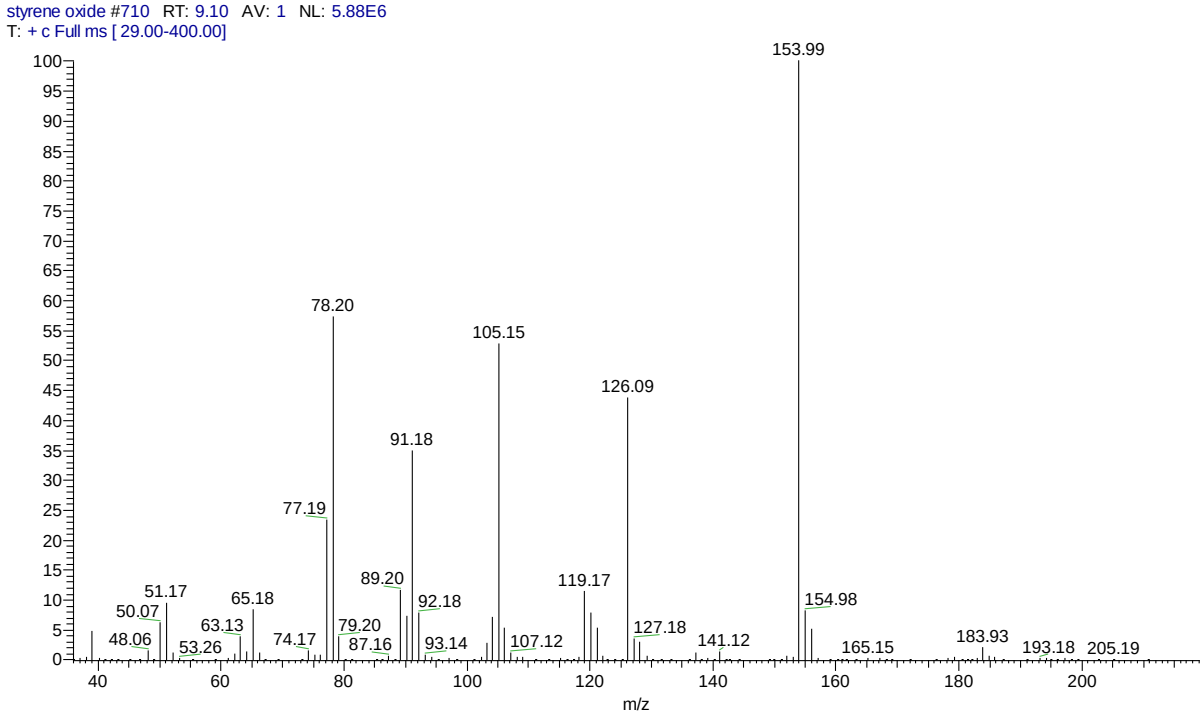
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40-7.50 (m, 5H), 5.40-5.44 (m, 1H), 4.73-4.77 (m, 1H), 4.46-4.51 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 129.4, 129.0, 127.5, 126.6, 85.5, 71.4; GC-MS: m/z (%): 183.84 (7) [M^+], 153.99 (100) [M^+-OCH_2], 126.10 (38) [M^+-C_3H_6O], 119.16 (16) [M^+-C_5H_5], 105.17 (44) [M^+-C_6H_7], 91.19 (37) [M^+-CHO_3S], 78.20 (33) [M^+-$C_2H_2O_3S$].

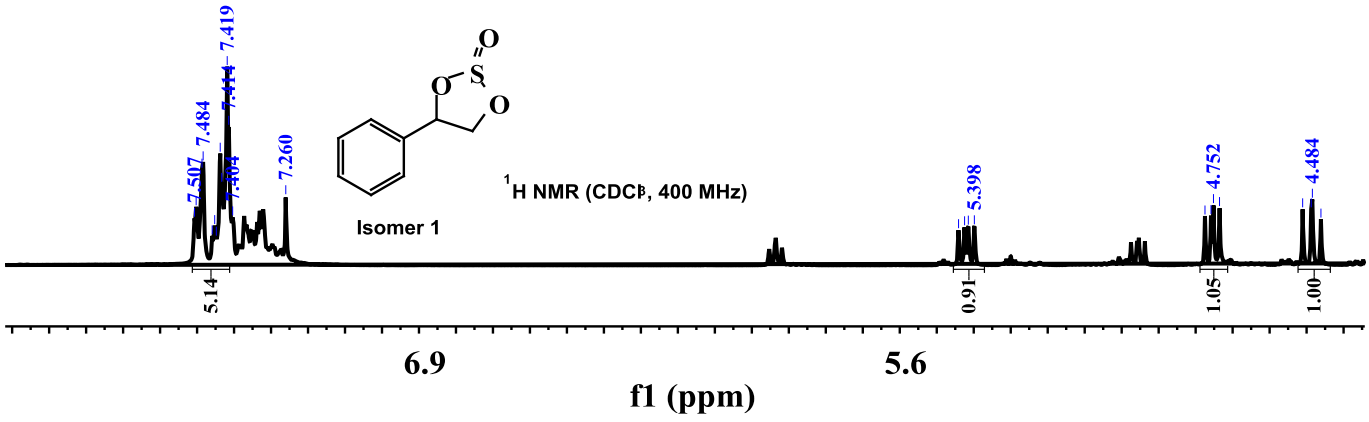
Isomer 2 (RT 9.10 min)

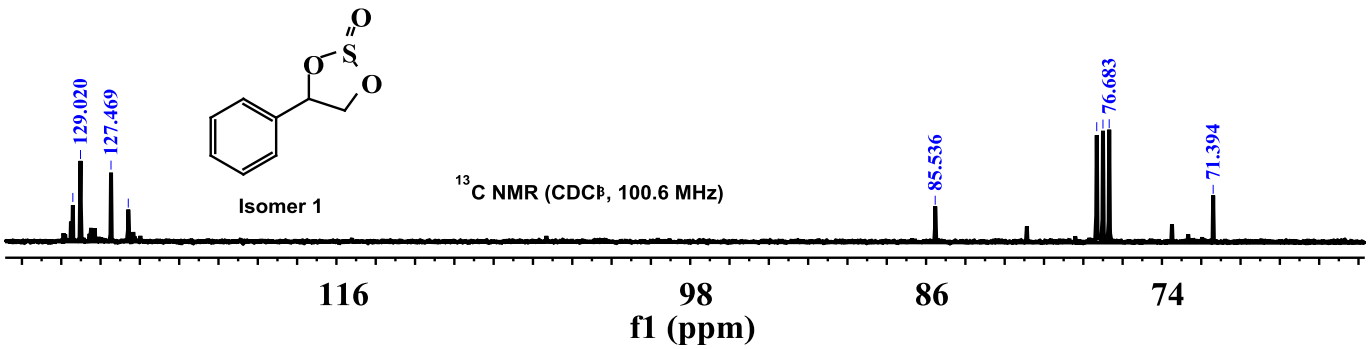
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.35-7.45 (m, 5H), 5.93 (t, ^{3}J = 6.8 Hz, 1H), 4.94-4.97 (m, 1H), 4.18-4.22 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 129.5, 129.0, 126.6, 80.9, 73.5; GC-MS: m/z (%): 183.93 (<5) [M^+], 153.99 (100) [M^+-OCH_2], 126.10 (44) [M^+-C_3H_6O], 119.17 (11) [M^+-C_5H_5], 105.15 (53) [M^+-C_6H_7], 91.18 (35) [M^+-CHO_3S], 78.20 (57) [M^+-$C_2H_2O_3S$].

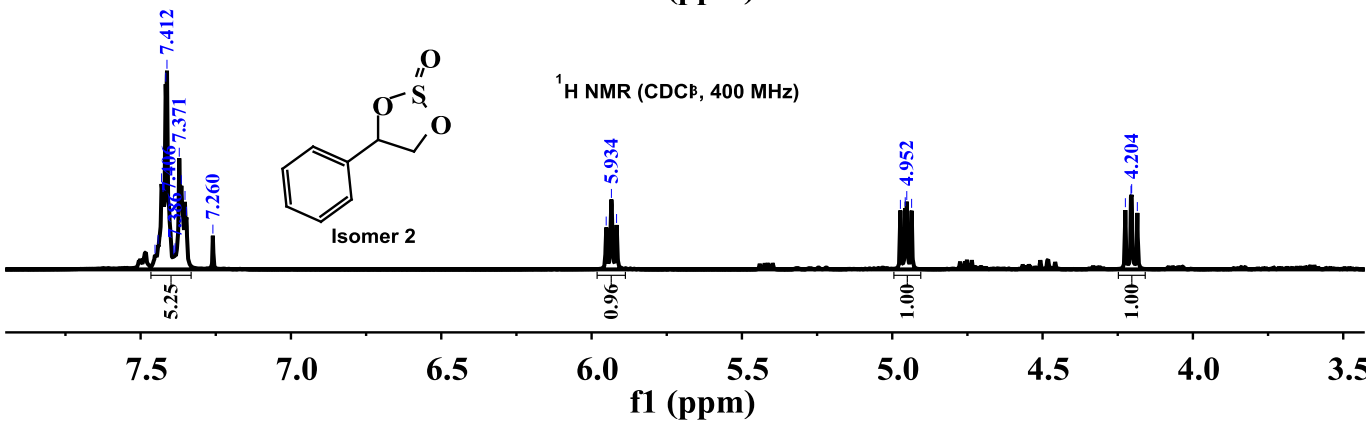


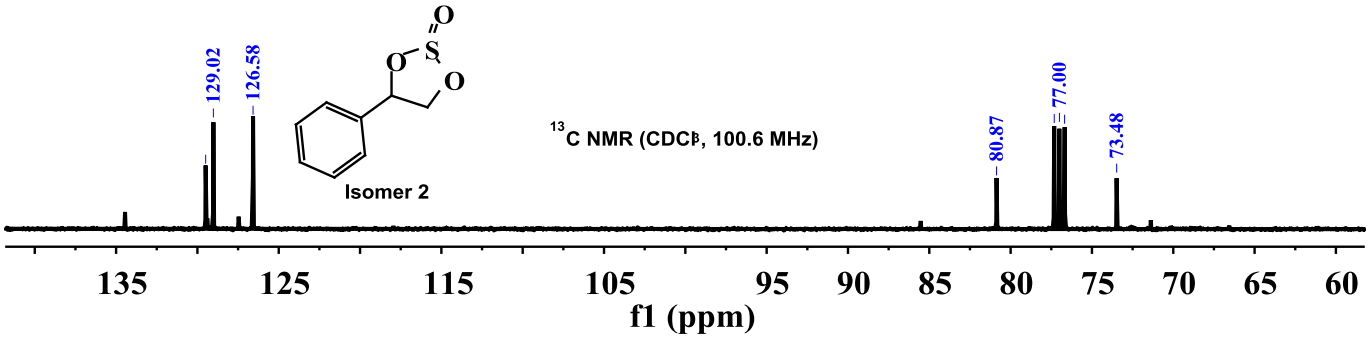

styrene oxide #671 RT: 8.84 AV: 1 NL: 8.63E6
T: + c Full ms [29.00-400.00]
153.99
105.17
91.19
126.10
78.20
77.20
119.16
89.20
92.17
51.12
65.19
155.01
183.84
39.05
63.16
79.29
103.20
107.16
128.14
141.09
42.91
53.22
66.28
167.11
179.23
184.93
197.17
210.23
m/z

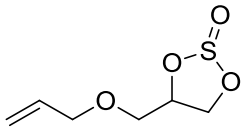

styrene oxide #710 RT: 9.10 AV: 1 NL: 5.88E6
T: + c Full ms [29.00-400.00]
153.99
78.20
105.15
126.09
91.18
77.19
89.20
119.17
51.17
65.18
92.18
154.98
50.07
63.13
79.20
127.18
48.06
74.17
87.16
93.14
107.12
141.12
53.26
165.15
183.93
193.18
205.19
m/z


7.507
7.484
7.419
7.414
7.304
7.260
5.398
4.752
4.484
5.14
0.91
1.05
1.00
Isomer 1
1H NMR (CDCl3, 400 MHz)
6.9
5.6
f1 (ppm)









4-((allyloxy)methyl)-1,3,2-dioxathiolane 2-oxide

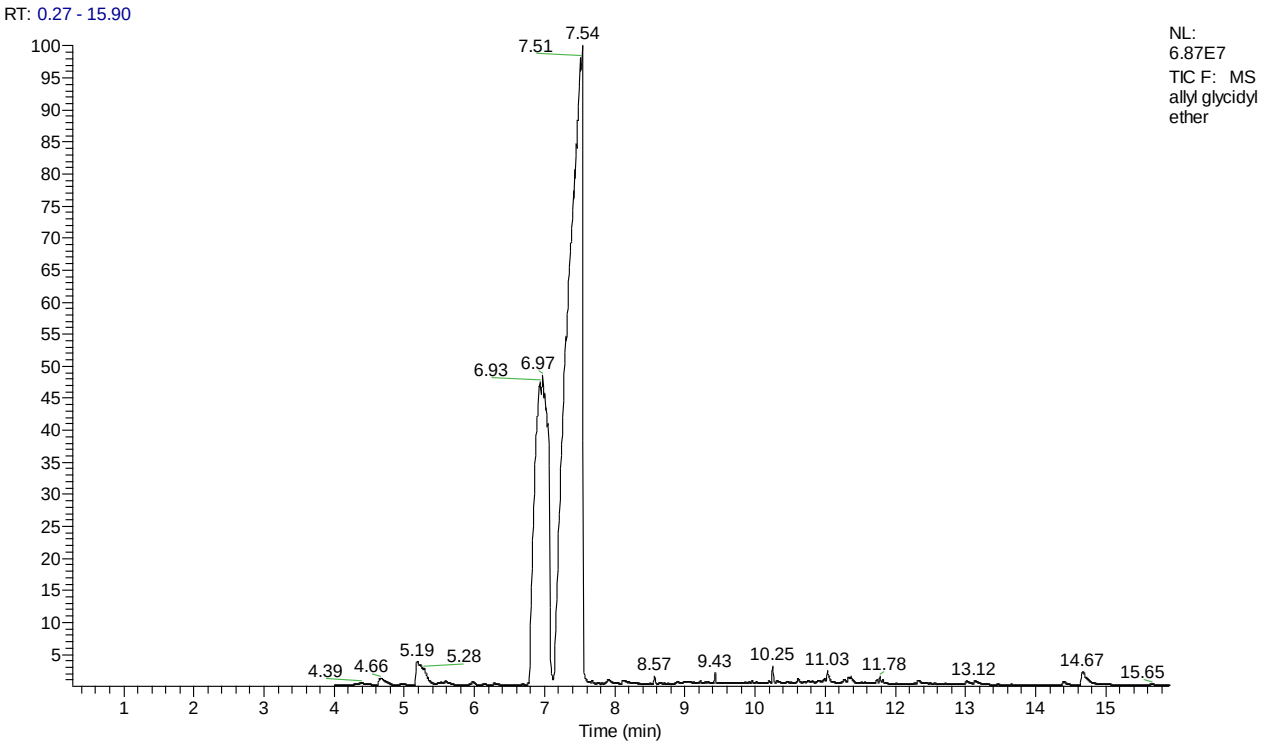
Column chromatography on silica gel: eluting with petroleum ether/ethyl acetae 25:1; Light yellow liquid.

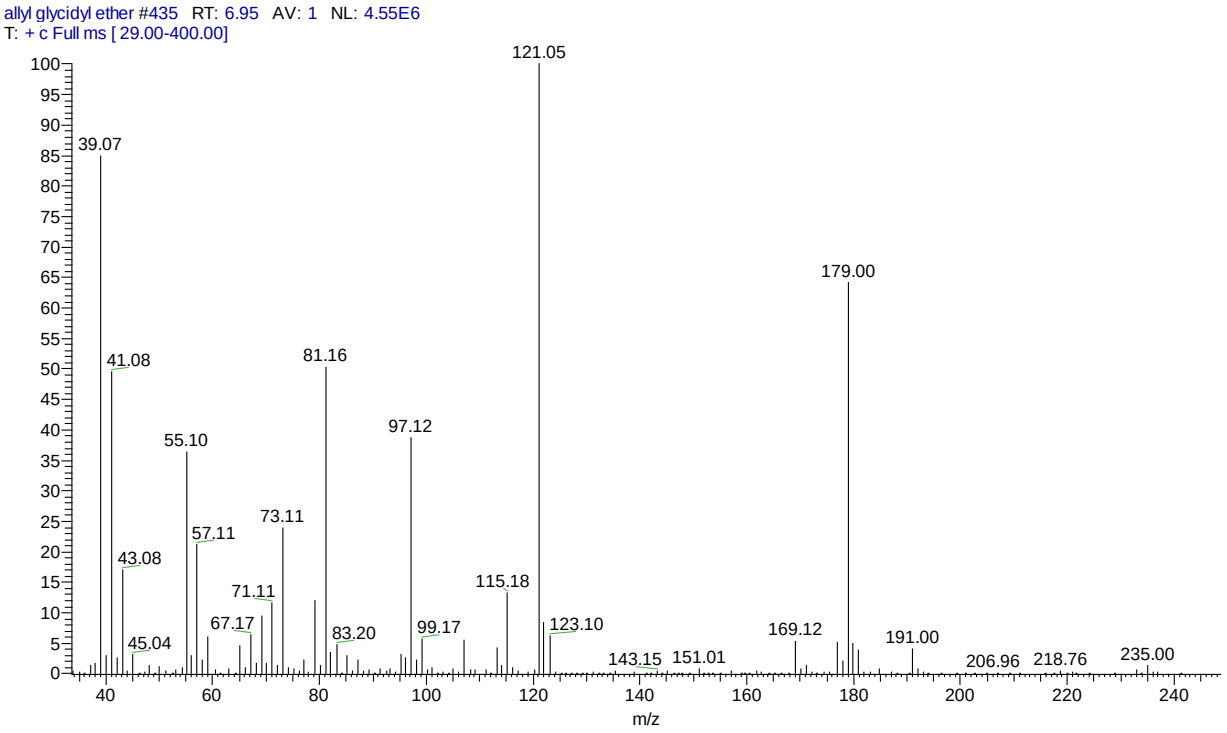
Isomer 1 (RT 6.97 min)

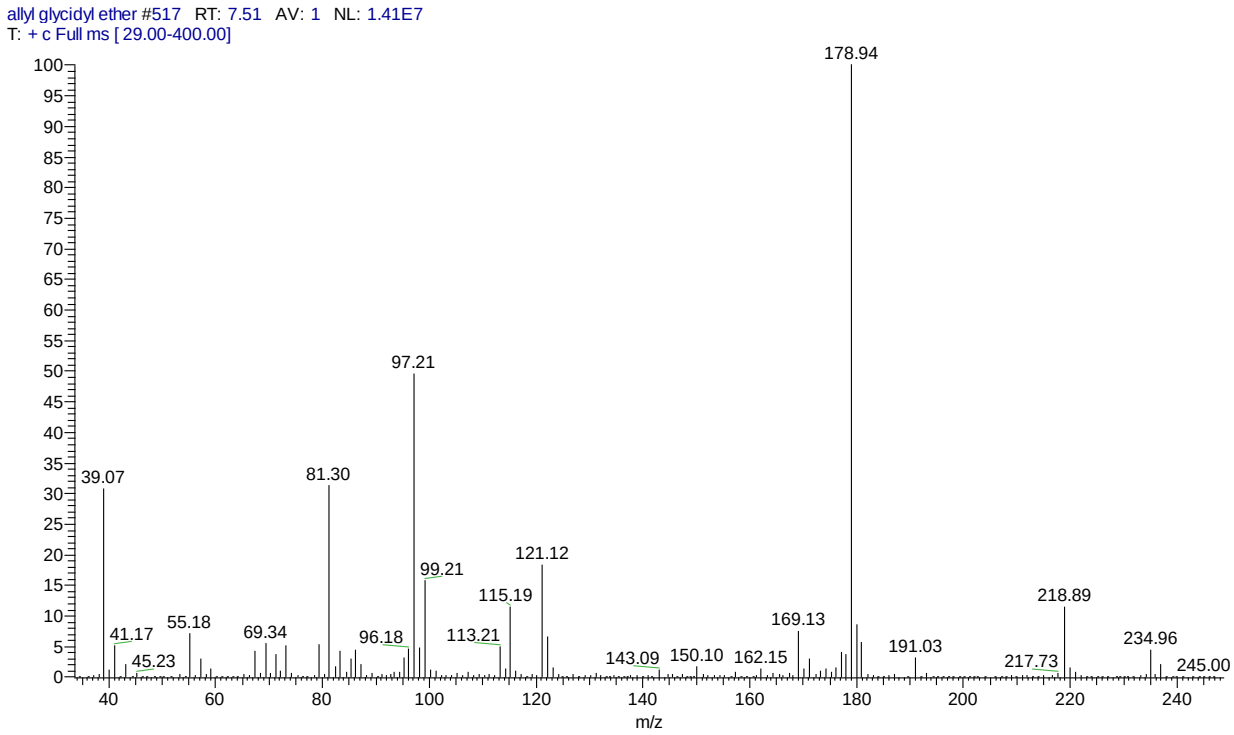
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.82-5.91 (m, 1H), 5.30 (s, 2H), 4.63-4.68 (m, 1H), 4.53-4.56 (m, 2H), 4.06 (s, ^{3}J = 5.6 Hz, 2H), 3.81-3.85 (m, 1H), 3.73-3.77 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 133.8, 81.0, 72.6, 70.1, 69.3; GC-MS: m/z (%): 178.94 (59) [M^+], 121.04 (100) [M^+-C_3H_5O], 97.18 (39) [M^+-H_2O_3S], 81.17 (48) [M^+-$C_6H_{10}O$], 39.07 (82) [M^+-$C_3H_7O_4S$].

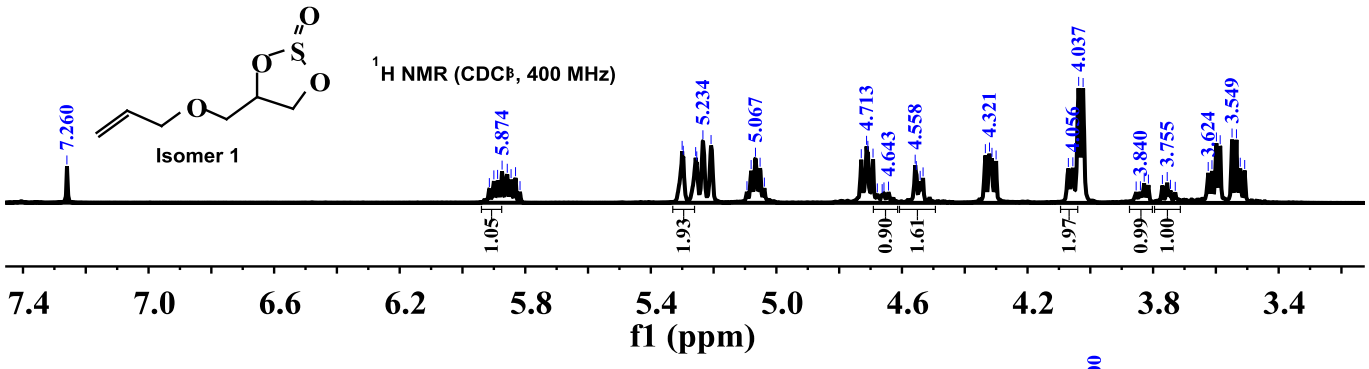
Isomer 2 (RT 7.54 min)

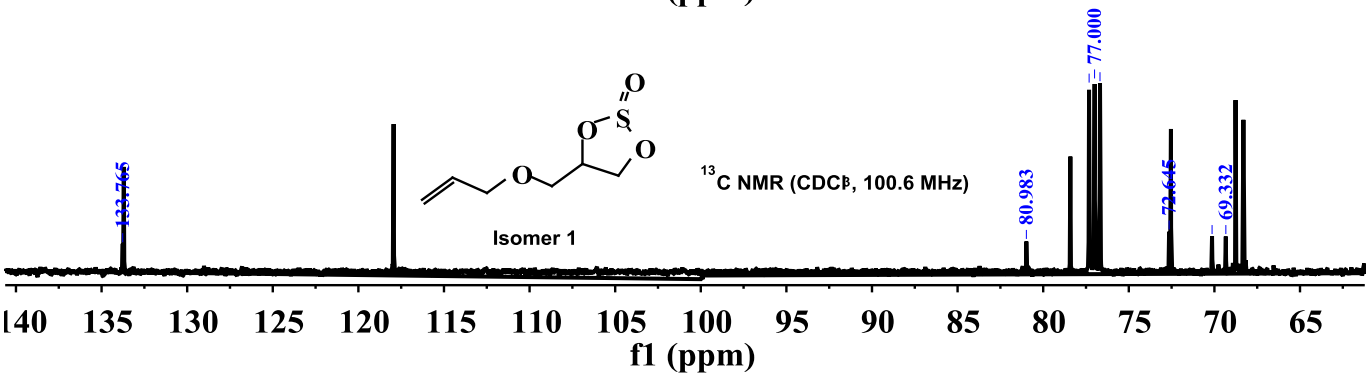
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.82-5.91 (m, 1H), 5.21-5.26 (m, 2H), 5.04-5.09 (m, 1H), 4.69-4.73 (m, 1H), 4.30-4.33 (m, 1H), 4.03 (d, ^{3}J = 5.6 Hz, 2H), 3.59-3.62 (m, 1H), 3.51-3.55 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 133.7, 117.9, 78.4, 72.5, 68.8, 68.3; GC-MS: m/z (%): 178.91 (100) [M^+], 121.13 (19) [M^+-C_3H_5O], 97.23 (48) [M^+-H_2O_3S], 81.30 (29) [M^+-$C_6H_{10}O$], 39.13 (30) [M^+-$C_3H_7O_4S$].

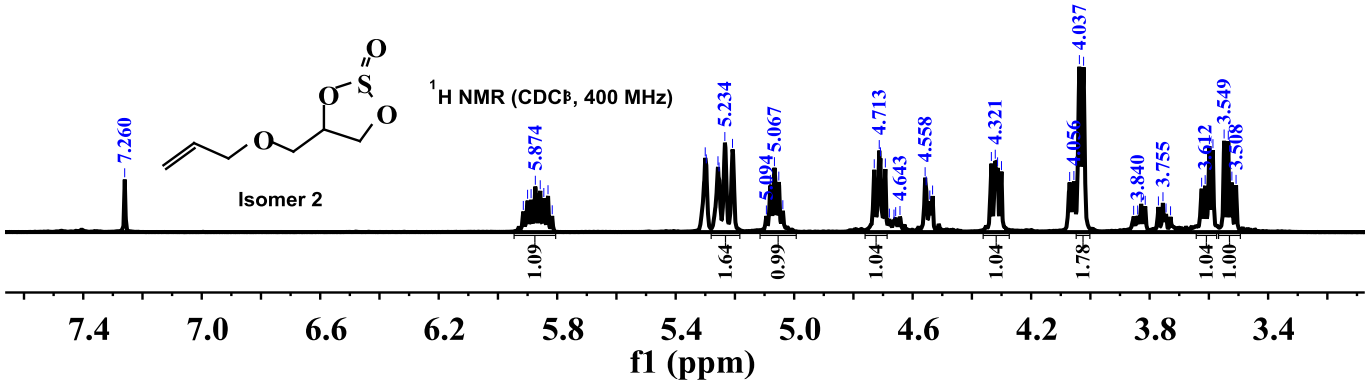

RT: 0.27 - 15.90
NL:
6.87E7
TIC F: MS
allyl glycidyl
ether
7.54
7.51
6.93
6.97
4.39
4.66
5.19
5.28
8.57
9.43
10.25
11.03
11.78
13.12
14.67
15.65
Time (min)

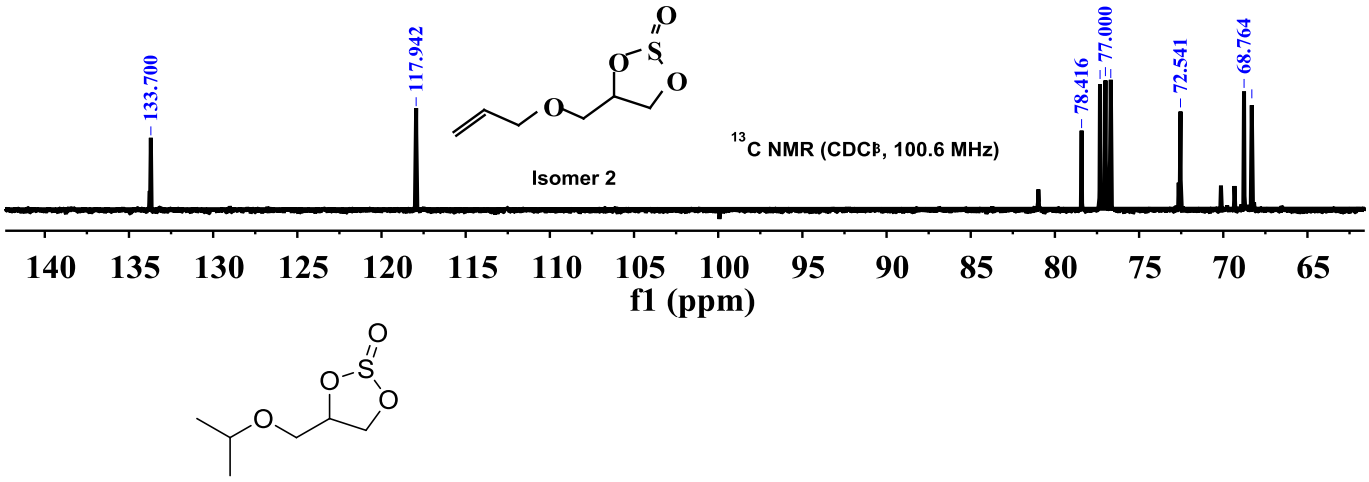

allyl glycidyl ether #435 RT: 6.95 AV: 1 NL: 4.55E6
T: + c Full ms [29.00-400.00]
121.05
39.07
179.00
41.08
81.16
97.12
55.10
73.11
57.11
43.08
115.18
71.11
67.17
83.20
99.17
123.10
169.12
191.00
45.04
143.15
151.01
206.96
218.76
235.00
m/z


allyl glycidyl ether #517 RT: 7.51 AV: 1 NL: 1.41E7
T: + c Full ms [29.00-400.00]
178.94
97.21
39.07
81.30
99.21
121.12
115.19
218.89
169.13
55.18
41.17
69.34
96.18
113.21
191.03
234.96
45.23
143.09
150.10
162.15
217.73
245.00
m/z


Isomer 1
1H NMR (CDCl3, 400 MHz)
7.260
5.874
5.234
5.067
4.713
4.643
4.558
4.321
4.056
4.037
3.840
3.755
3.624
3.549
1.05
1.93
0.90
1.61
1.97
0.99
1.00
f1 (ppm)


Isomer 1
13C NMR (CDCl3, 100.6 MHz)
133.765
80.983
77.000
72.645
69.332
f1 (ppm)


Isomer 2
1H NMR (CDCl3, 400 MHz)
7.260
5.874
5.234
5.094
5.067
4.713
4.643
4.558
4.321
4.056
4.037
3.840
3.755
3.612
3.549
3.508
1.09
1.64
0.99
1.04
1.04
1.78
1.04
1.00
f1 (ppm)



4-(isopropoxymethyl)-1,3,2-dioxathiolane 2-oxide

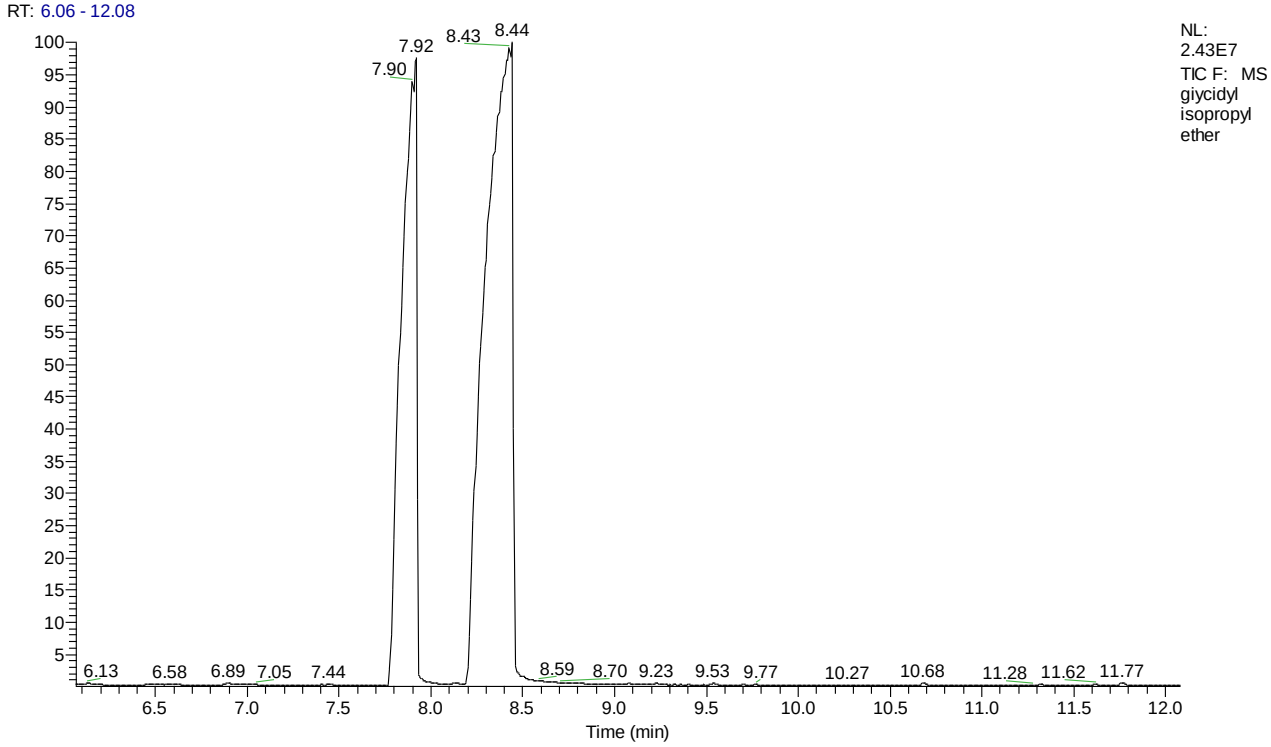
Column chromatography on silica gel: eluting with petroleum ether/ethyl acetae 25:1; Light yellow liquid.

Isomer 1 (RT 7.92 min)

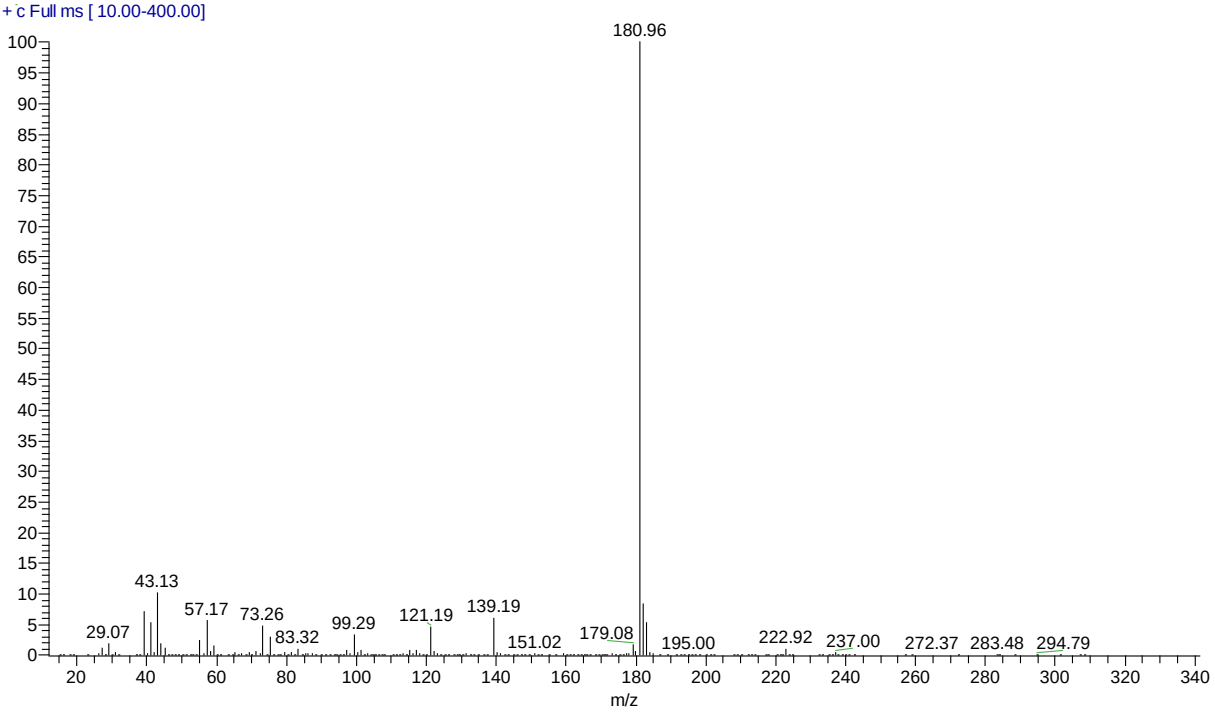
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.79-3.84 (m, 1H), 3.65-3.70 (m, 1H), 3.56-3.63 (m, 2H), 3.45-3.52 (m, 2H), 3.22 (s, 2H), 1.13-1.15 (m, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 21.9, 64.2, 66.5, 68.9, 70.6, 72.4, 78.7; GC-MS: m/z (%): 180.96 (100) [M^+].

Isomer 2 (RT 8.44 min)

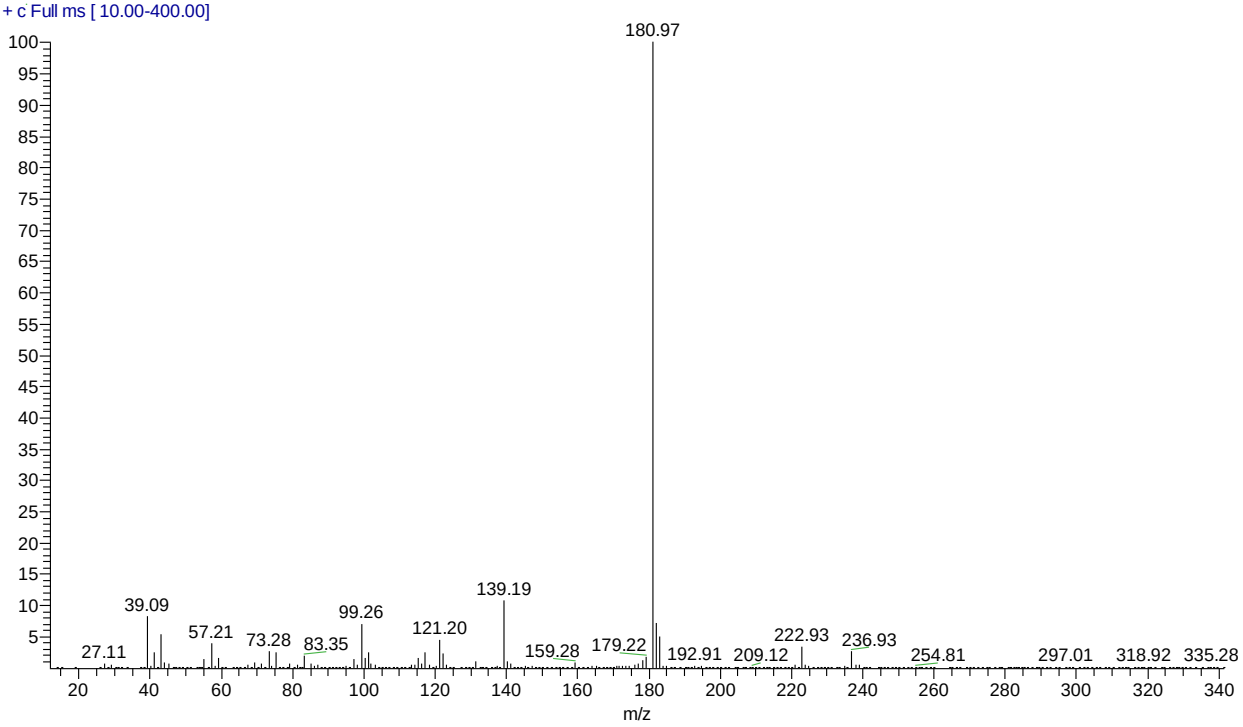
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.99-5.04 (m, 2H), 4.67-4.71 (m, 2H), 4.48-4.56 (m, 2H), 4.28-4.31 (m, 2H), 1.13-1.15 (m, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 81.2, 72.81, 72.78, 69.74, 69.71, 68.6, 21.8; GC-MS: m/z (%): 180.99 (100) [M^+].

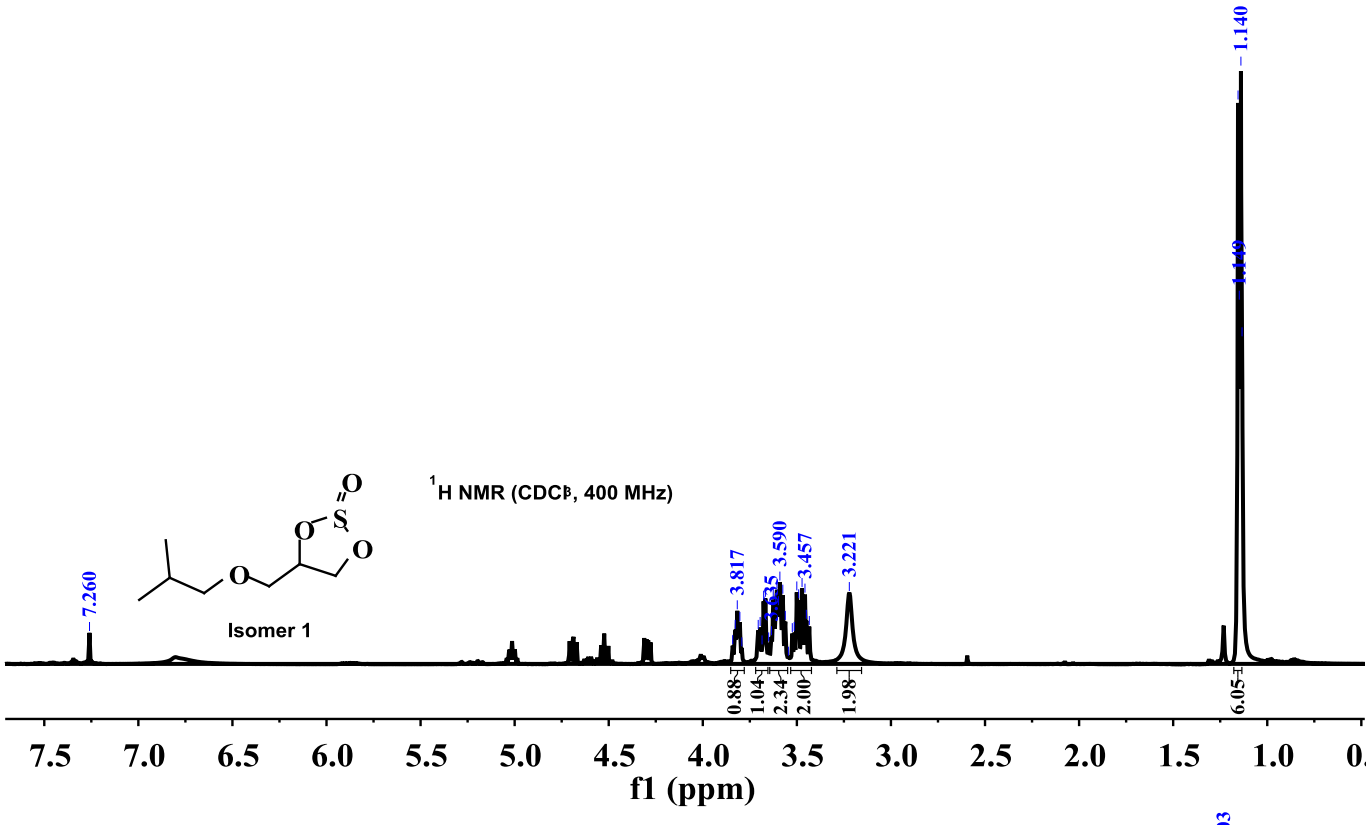


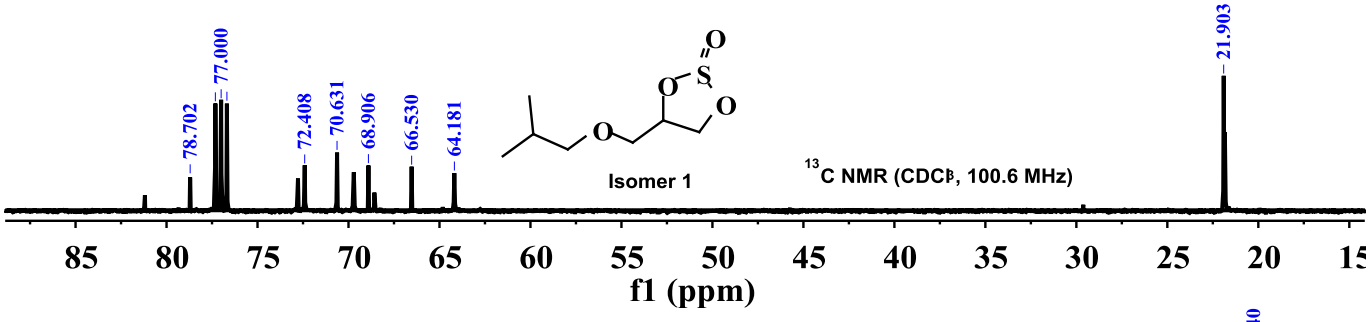
giycidyl isopropyl ether #516 RT: 7.90 AV: 1 NL: 1.14E7
T: + c Full ms [10.00-400.00]

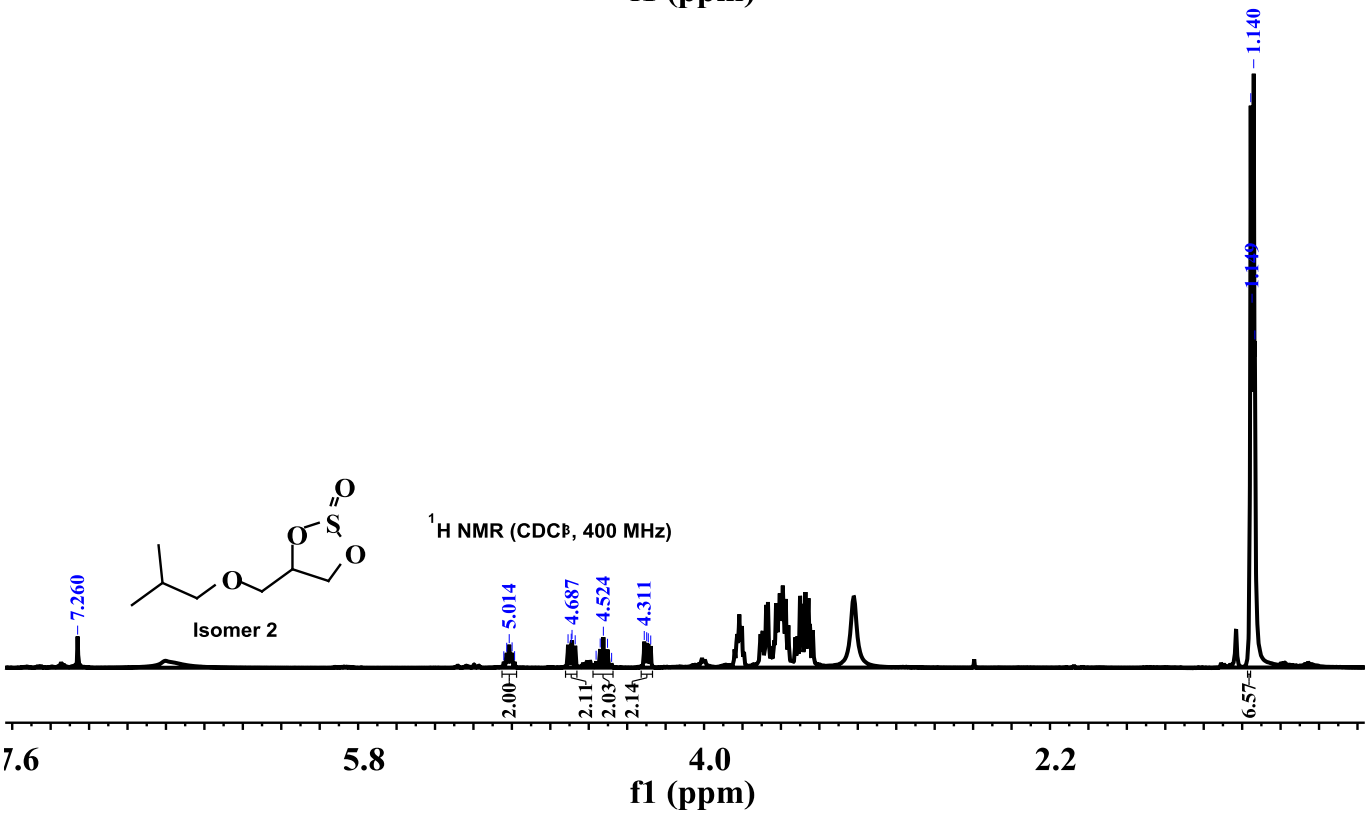


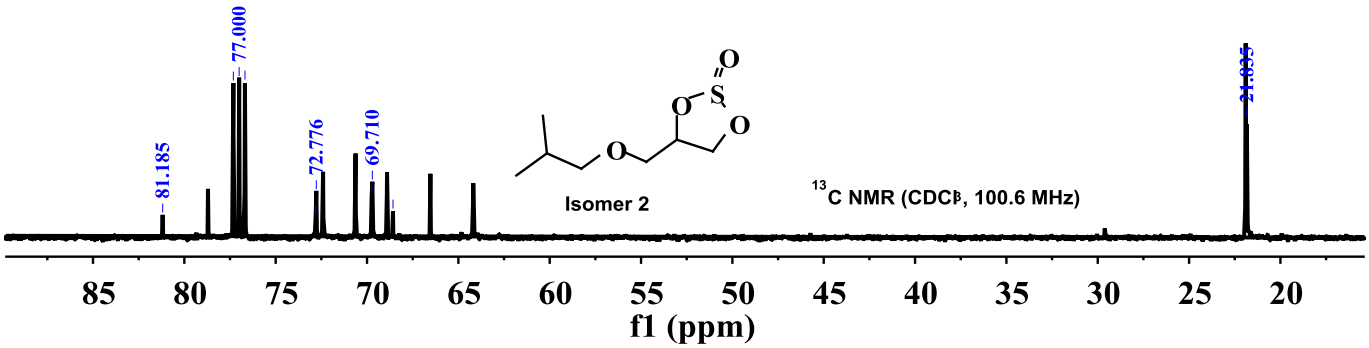
giycidyl isopropyl ether #588 RT: 8.43 AV: 1 NL: 1.04E7
T: + c Full ms [10.00-400.00]

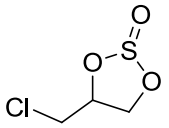



1.140
7.260
3.817
3.590
3.457
3.221
0.88
1.04
2.34
2.00
1.98
6.05
Isomer 1
^{1}H NMR (CDCl$_3$, 400 MHz)
f1 (ppm)


21.903
78.702
77.000
72.408
70.631
68.906
66.530
64.181
Isomer 1
^{13}C NMR (CDCl$_3$, 100.6 MHz)
f1 (ppm)


1.140
7.260
5.014
4.687
4.524
4.311
2.00
2.11
2.03
2.14
6.57
Isomer 2
^{1}H NMR (CDCl$_3$, 400 MHz)
f1 (ppm)





4-(chloromethyl)-1,3,2-dioxathiolane 2-oxide

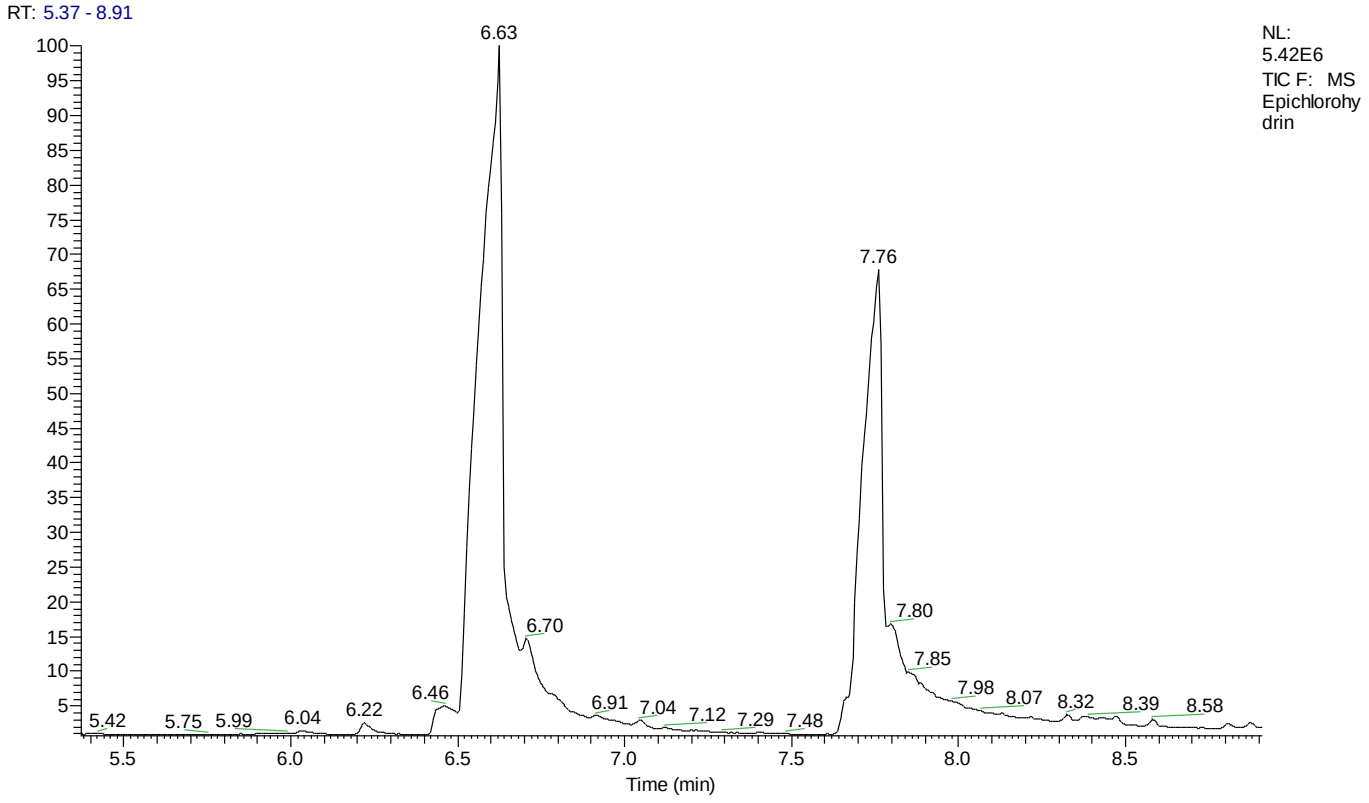
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 3:1; Light yellow liquid.

Isomer 1 (minor)

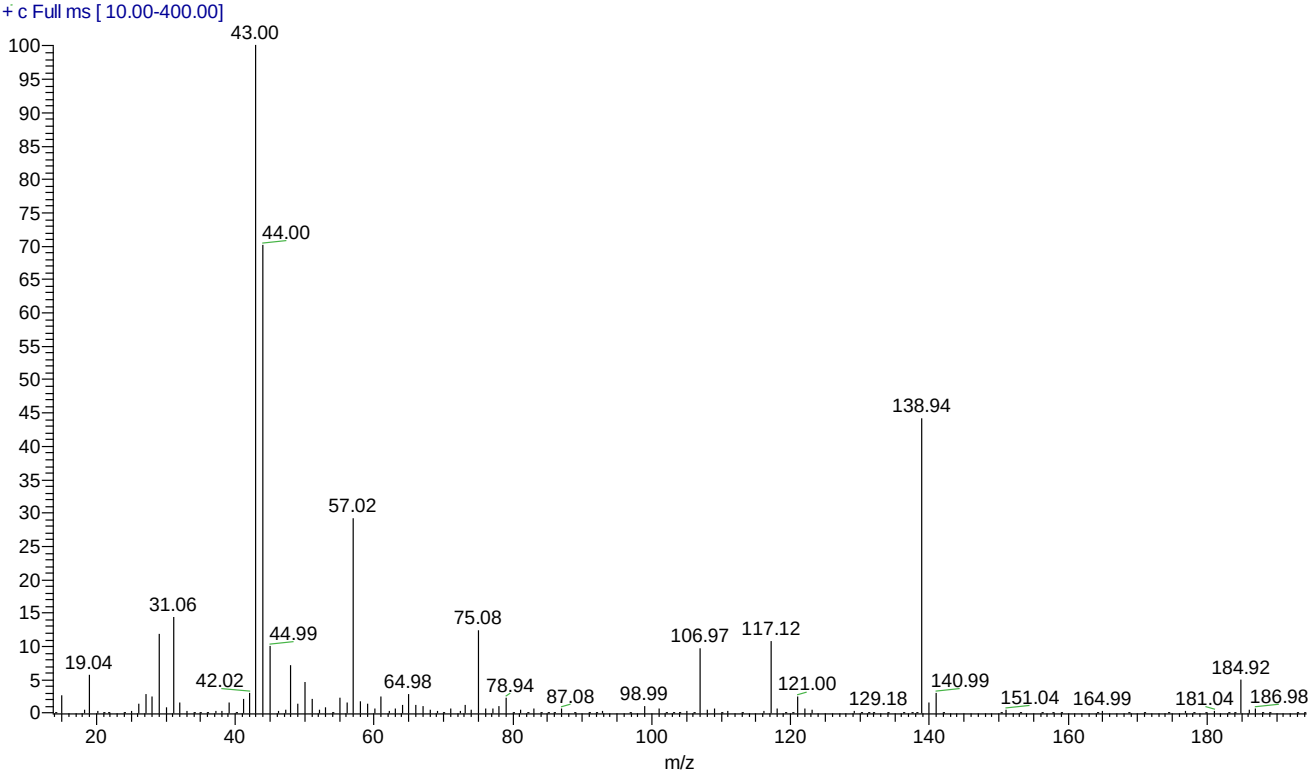
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.72-4.75 (m, 1H), 4.63-4.65 (m, 2H), 3.88-3.92 (m, 1H), 3.75-3.80 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.7, 70.5, 43; GC-MS: m/z (%):139.02 (40) [M^+ - OH], 106.98 (9) [M^+-CH_2Cl], 42.99 (100) [M^+-SO_3Cl].

Isomer 2 (major)

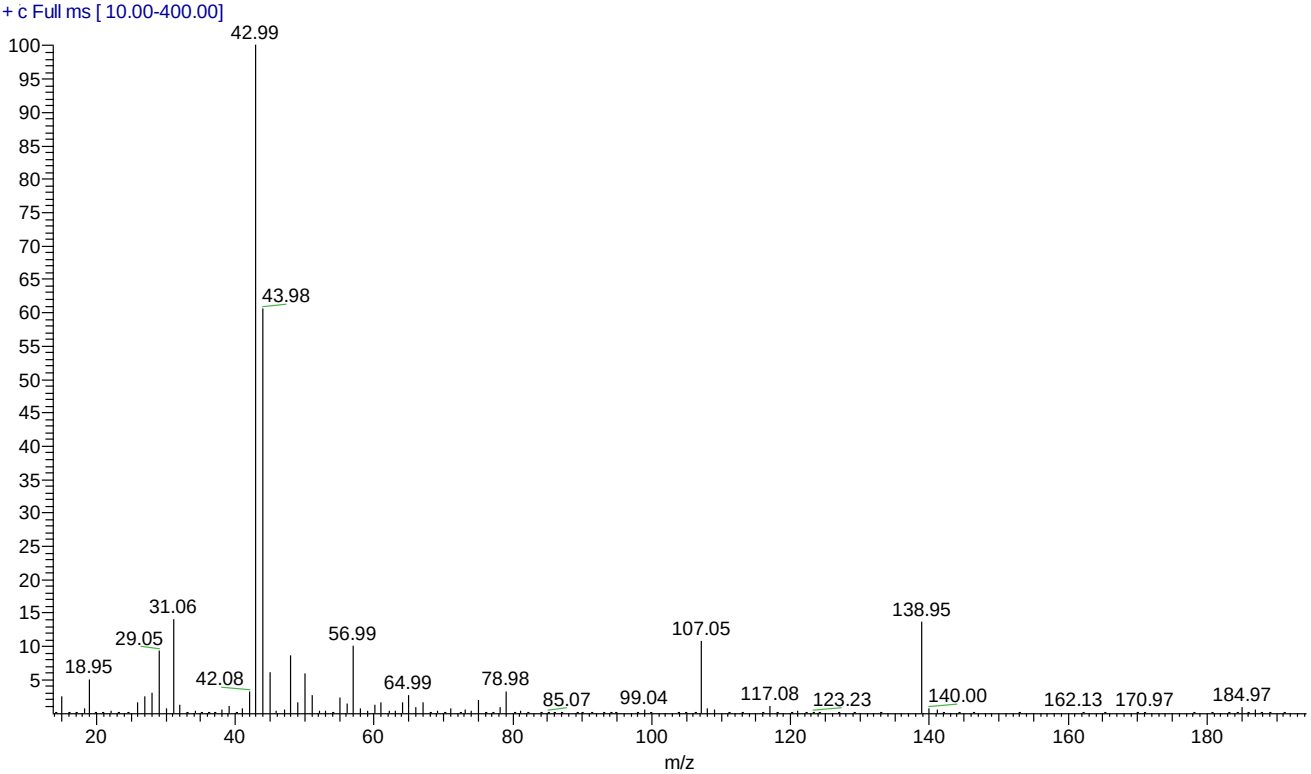
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.10-5.15 (m, 1H), 4.77-4.82 (m, 1H), 4.43-4.47 (m, 1H), 3.64-3.68 (m, 1H), 3.50-3.55 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 78.8, 69.0, 42.3; GC-MS: m/z (%):138.95 (14) [M^+ - OH], 107.05 (11) [M^+-CH_2Cl], 42.99 (100) [M^+-SO_3Cl].

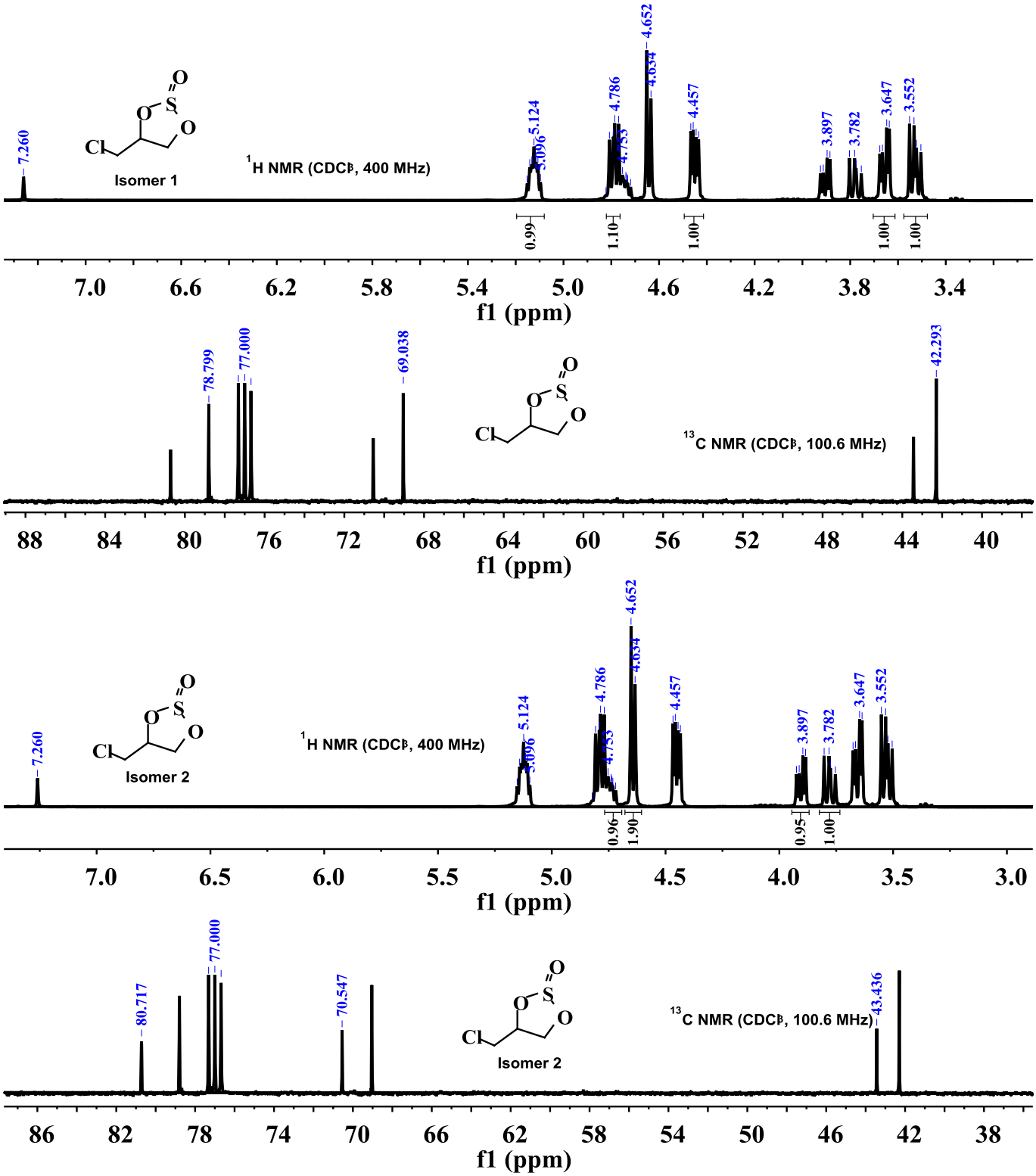


Epichlorohydrin #345 RT: 6.63 AV: 1 NL: 1.34E6
T: + c Full ms [10.00-400.00]



Epichlorohydrin #498 RT: 7.76 AV: 1 NL: 1.24E6
T: + c Full ms [10.00-400.00]





7. Reference

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