Supporting Information

Highly efficient SO_2 absorption and its subsequent utilization by weak base/polyethylene glycol binary system

Zhen-Zhen Yang, Liang-Nian He, Ya-Nan Zhao and Bing Yu*

State Key Laboratory and Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, 300071, People's Republic of China.

* Corresponding author: Tel: +86-22-23503878; Fax: +86-22-23503878; E-mail: heln@nankai.edu.cn

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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 and directly used without further purification. SO_2 and CO_2 with a purity of 99.99% is commercially available. PEG_{150}MeIm and *n*-OctIm are synthesized according to the reported method.[1-3]

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.

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 ±0.0001 g every five minutes. During the absorption of SO_2 under reduced pressure, SO_2 is diluted with N_2 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 synthesis of cyclic sulfites using captured SO_2 and its recyclability

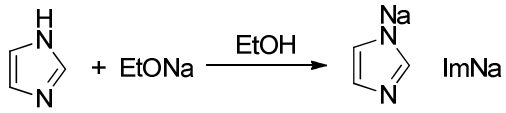
Firstly, PEG_{150}MeIm (2 mmol) and PEG_{150} (2 mmol) or choline chloride (2 mmol) were charged into a glass tube, in which SO_2 was bubbled through a needle. Then, epoxide and NH_4I (only for PEG_{150}MeIm/PEG_{150} system) was added after SO_2 absorption reached equilibrium. The tube was placed into a 25 mL stainless steel autoclave and then the mixture was stirred at predetermined temperature for 5 min to reach the equilibration. When the reaction finished, the reactor was cooled in ice-water and SO_2 was ejected slowly. 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 acetae or petroleum ether/dichloromethane), and further identified by GC-MS and NMR, which are consistent with those reported in the literature[4] and in good agreement with the assigned structures.

For (Table S1, Entry 20) and (Table S2, Entry 14), a typical procedure is as follows: PEG_{150} and NH_4I/Choline chloride, biphenyl (internal standard of GC) and propylene oxide were added successively into a glass tube. The suspension was cooled to -60 ^{o}C (liquid nitrogen/ethanol) and SO_2 (8.0/10.4 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 80 ^{o}C with stirring for 3/6 h.

For recycle of the absorbtion systems, the binary systems were recovered after separation of propylene sulfite from the reaction mixture by distillation under reduced pressure and then reused for the next run without further purification.

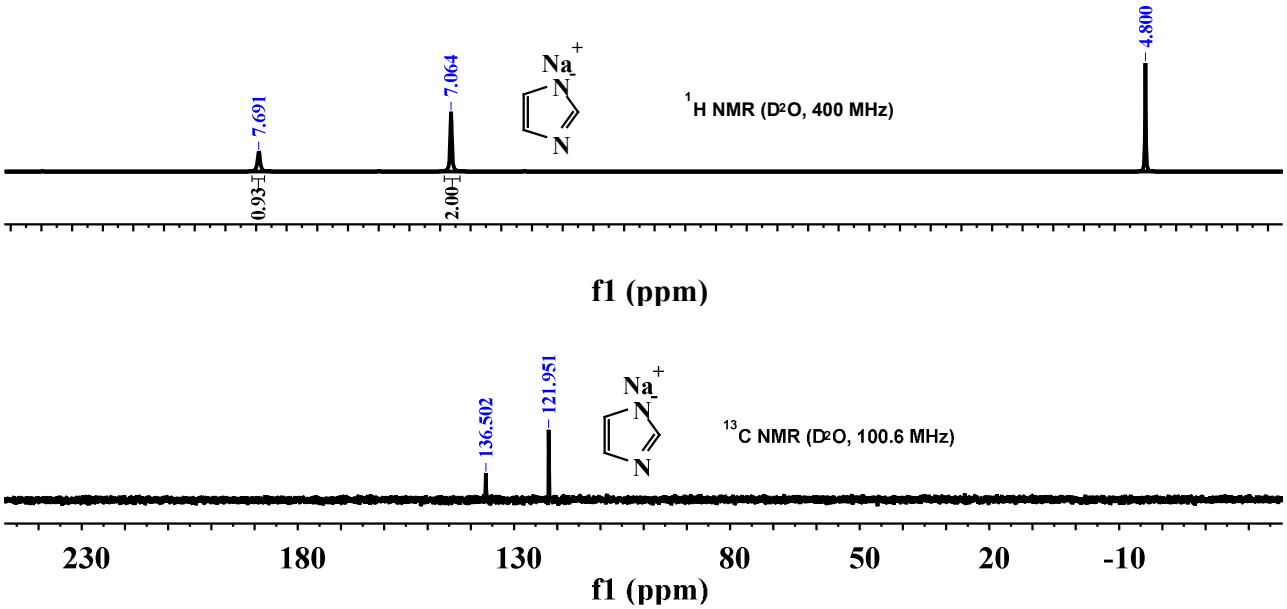
2. Synthesis and characterization of PEG_{150}MeIm and *n*-OctIm:

ImNa[1]

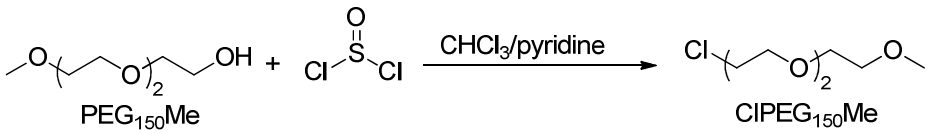


Imidazole (0.5 mol) and sodium ethoxide (0.5 mol) are dissolved in ethanol (50 mL) stirred at 70 ^{o}C for 8 h, and then ethanol is removed by rotator evaporation under reduced pressure. The residue is washed with diethyl ether three times and dried in vacuum to give intermediate ImNa as a yellow solid.

^{1}H NMR (D_2O, 400 MHz) δ 7.69 (s, 1H), 7.06 (s, 2H); ^{13}C NMR (D_2O, 100.6 MHz) δ 136.5, 122.0.

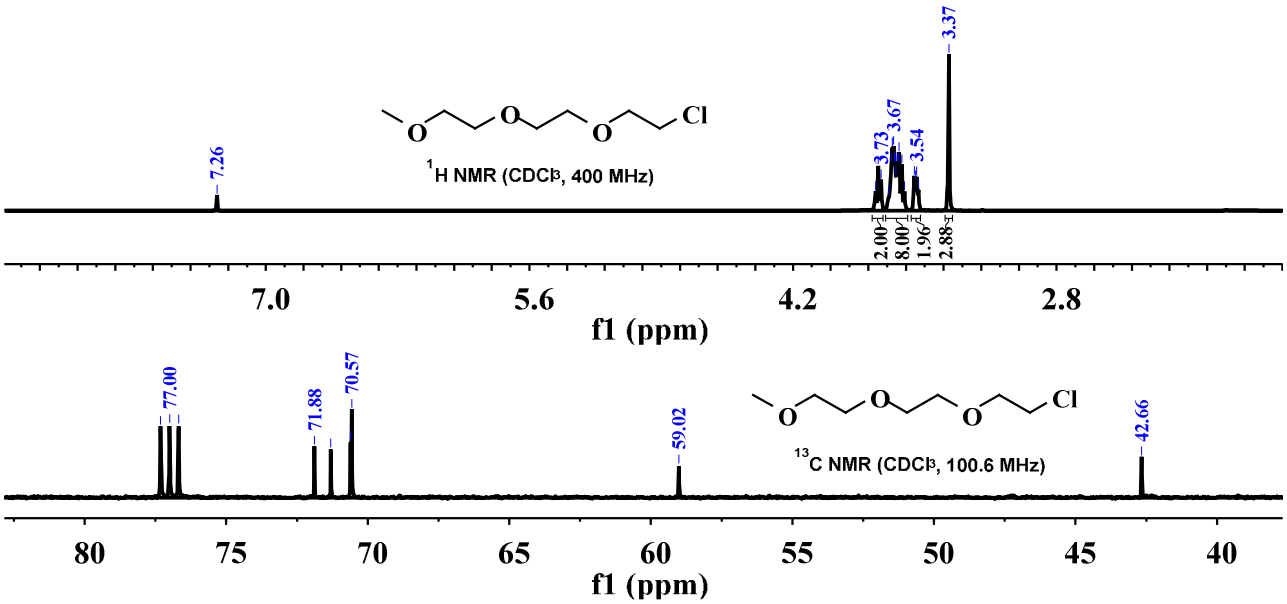


PEG$_{150}$MeCl[2]

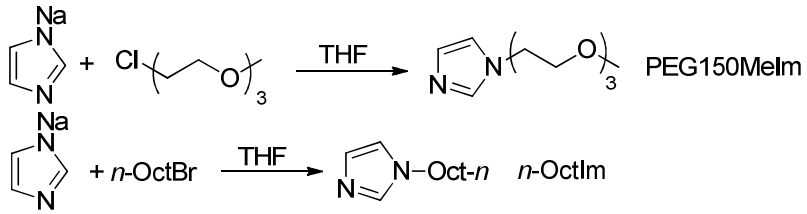


A solution of thionyl chloride (0.45 mol) in $CHCl_3$ (90 mL) is added slowly over 60 min to a stirred solution of triethylene glycol monomethyl ether (0.3 mol) and pyridine (0.3 mol) in $CHCl_3$ (200 mL), followed by refluxing the above reaction mixture at 100 °C for 4 h, and then yellow is obtained,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

Yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) 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 (100.6 MHz, $CDCl_3$) 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].



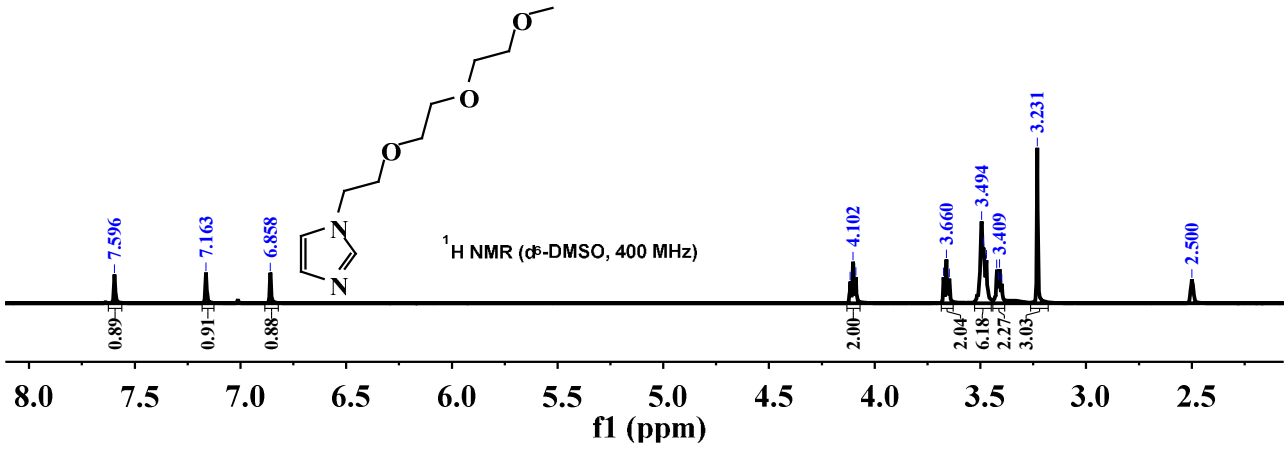
Synthesis of PEG$_{150}$MeIm and *n*-OctIm[3]

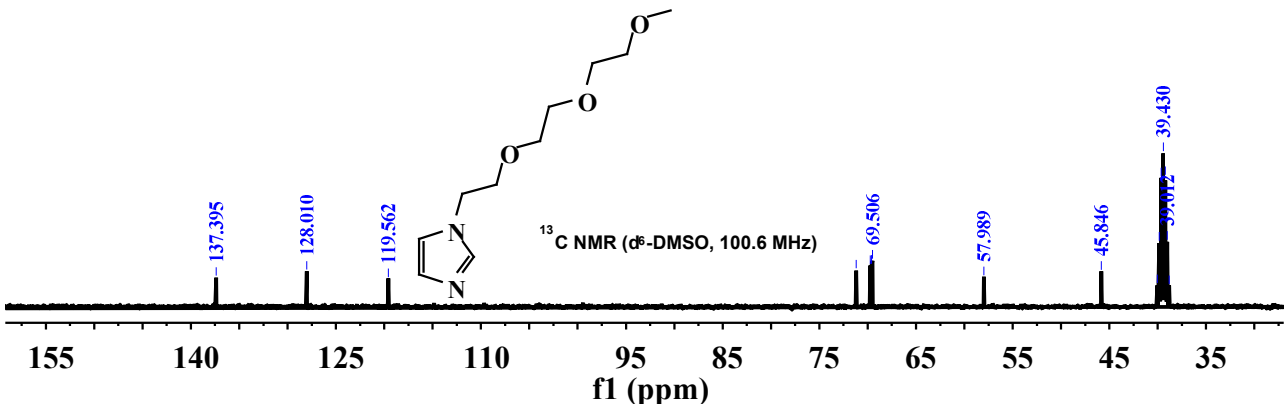


ImNa (0.05 mol) and ClPEG$_{150}$Me (0.045 mol) or 1-bromoctane (0.45 mol) is added in THF (30 mL) and the mixture is stirred at reflux for 10 h and filtered, the filtrate is concentrated under reduced pressure to removed THF. Then CH_2Cl_2 (30 mL) is added to the residue, which is filtered again to remove the precipitation. The filtrate is concen under reduced pressure to remove CH_2Cl_2. The crude product is purified under reduced pressure to give PEG$_{150}$MeIm or *n*-OctIm as colourless liquid.

PEG$_{150}$MeIm

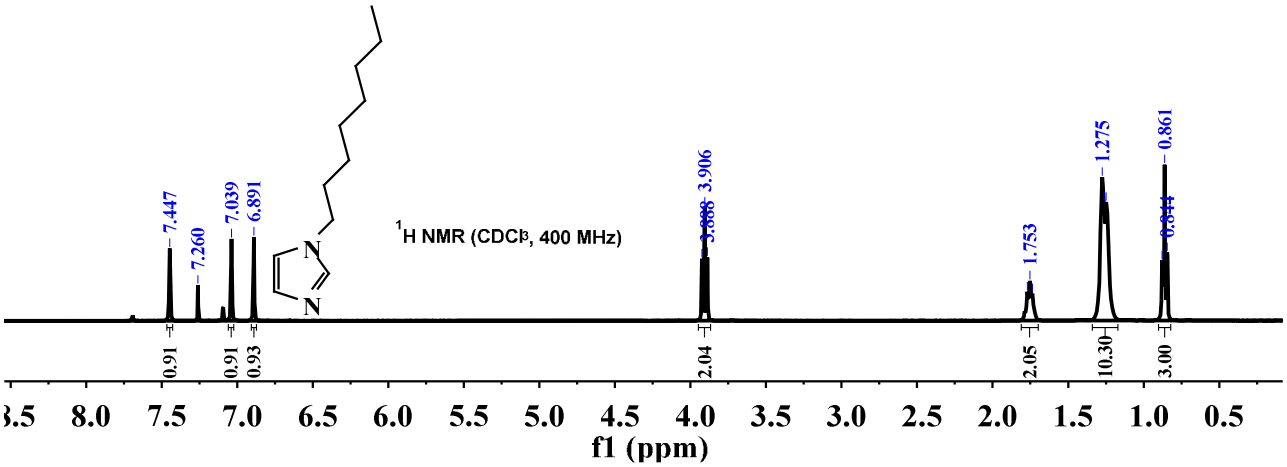
^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 7.60 (s, 1H), 7.16 (s, 1H), 6.86 (s, 1H), 4.10 (t, ^3J = 5.2 Hz, 2H), 3.66 (t, ^3J = 5.62, 2H), 3.47-3.49 (m, 6H), 3.40-3.42 (m, 2H), 3.23 (s, 3H); ^{13}C NMR (d$_6$-DMSO, 10.6 MHz) δ 137.4, 128.0, 119.6, 71.2, 69.7, 69.6, 69.5, 58.0, 45.8; ESI-MS calcd for $C_{10}H_{18}N_2O_3$ 214.13, found 215.3 $[M+H]^+$.

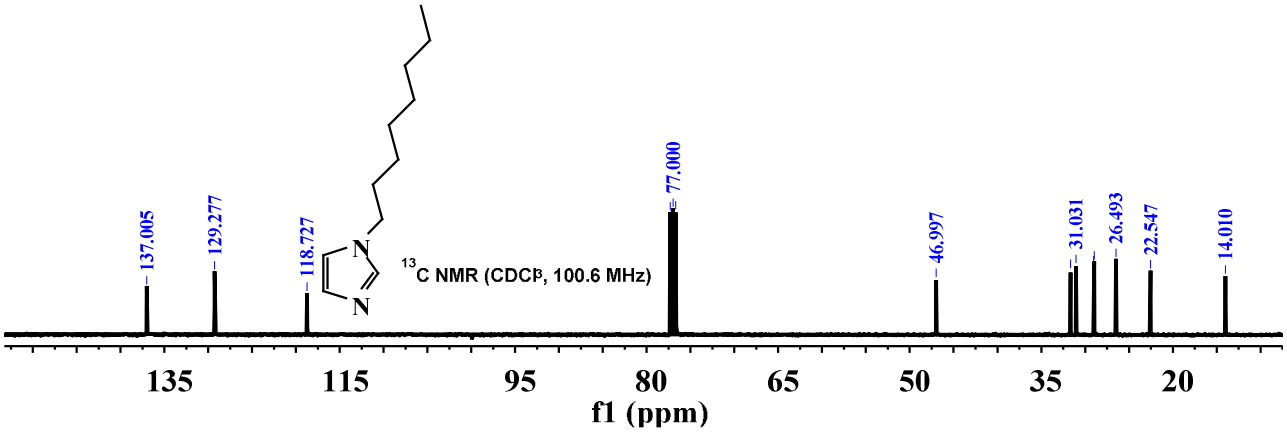




***n*-OctIm**

^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45 (s, 1H), 7.04 (s, 1H), 6.89 (s, 1H), 3.91 (t, ^3J = 7.2 Hz, 2H), 1.74-1.79 (m, 2H), 1.25-1.27 (m, 12H), 0.86 (t, ^3J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 137.0, 129.3, 118.7, 47.0, 31.7, 31.0, 29.0, 28.9, 26.5, 22.5, 14.0; ESI-MS calcd for $C_{11}H_{20}N_2$ 180.16, found 181.3 $[M+H]^+$.





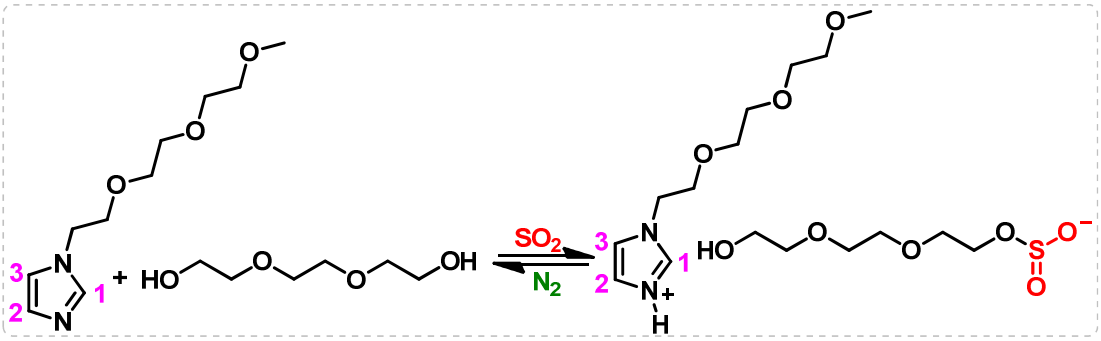
3. Characterization of $PEG_{150}MeIm/PEG_{150}$ (molar ratio 1:1) (^{1}H, ^{13}C NMR and ^{1}H-^{13}C HSQC) before and after SO_2 capture

$PEG_{150}MeIm$

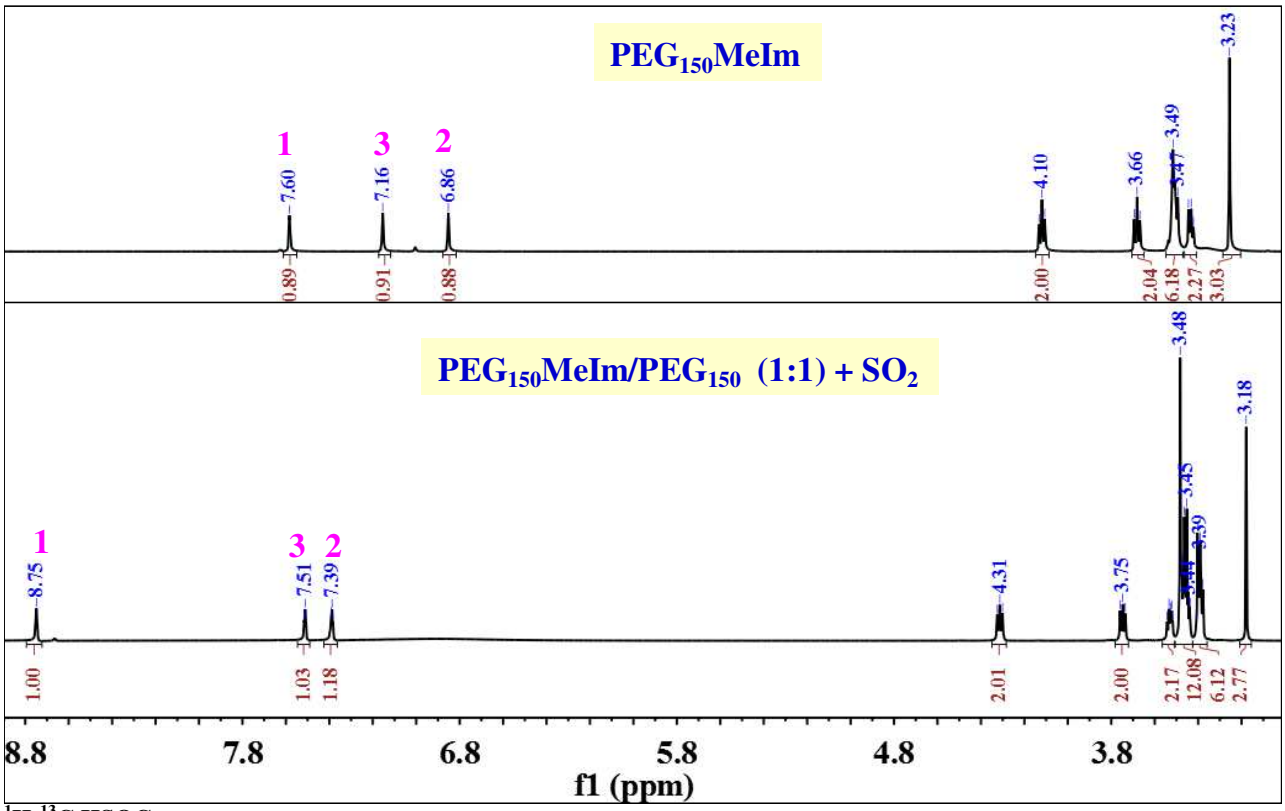
^{1}H NMR (d_6-DMSO, 400 MHz) δ 7.60 (s, 1H), 7.16 (s, 1H), 6.86 (s, 1H), 4.10 (t, ^3J = 5.2 Hz, 2H), 3.66 (t, ^3J = 5.62, 2H), 3.47-3.49 (m, 6H), 3.40-3.42 (m, 2H), 3.23 (s, 3H); ^{13}C NMR (d_6-DMSO, 10.6 MHz) δ 137.4, 128.0, 119.6, 71.2, 69.7, 69.6, 69.5, 58.0, 45.8;

$PEG_{150}MeIm/PEG_{150}$ (1:1) + SO_2

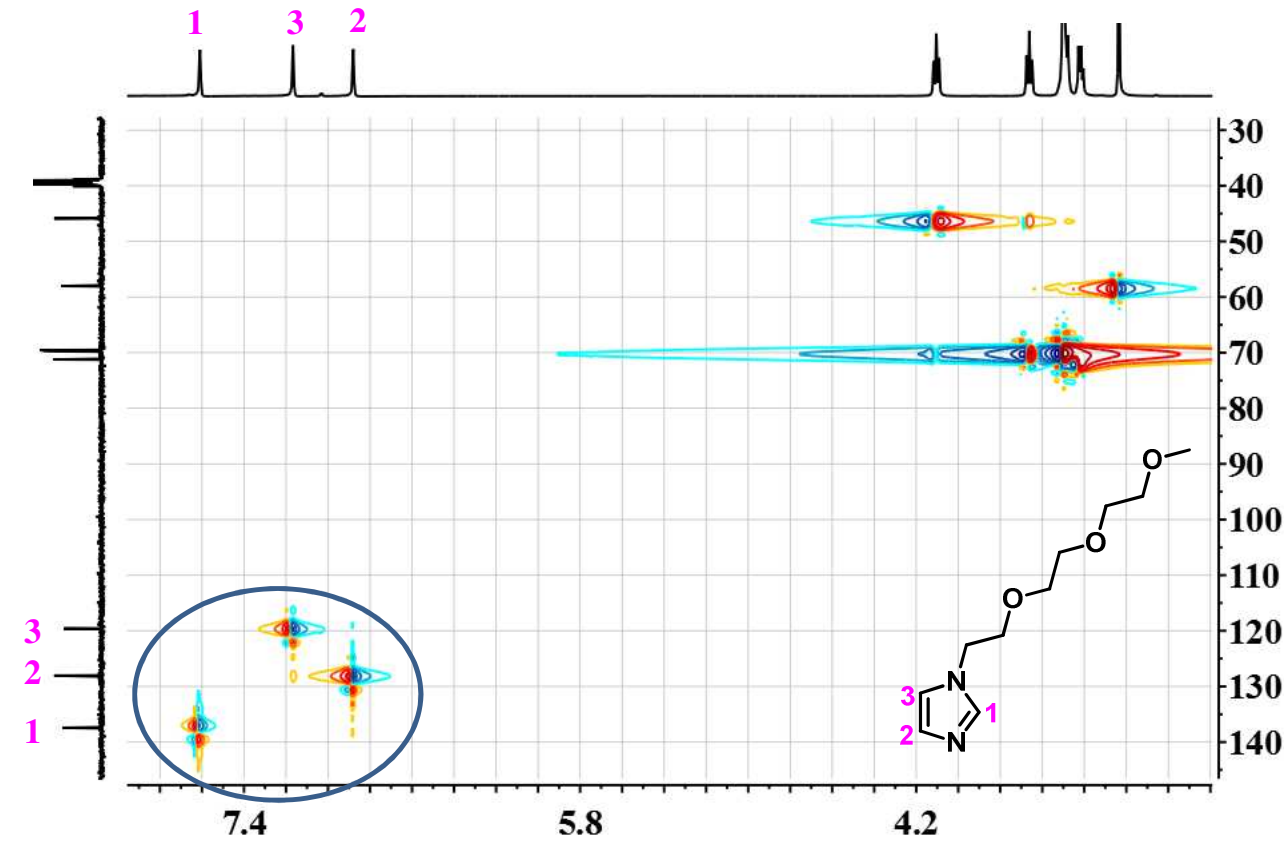
^{1}H NMR (d_6-DMSO, 400 MHz) δ 8.75 (s, 1H), 7.51 (s, 1H), 7.39 (s, 1H), 4.31 (t, ^3J = 4.4 Hz, 2H), 3.75 (t, ^3J = 4.8 Hz, 2H), 3.52-3.54 (m, 2H), 3.44-3.48 (m, 12H), 3.38-3.41 (m, 6H), 3.18 (s, 3H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 136.3, 123.5, 120.7, 73.0, 72.2, 70.7, 70.6, 70.5, 69.3, 61.1, 58.7, 49.9.

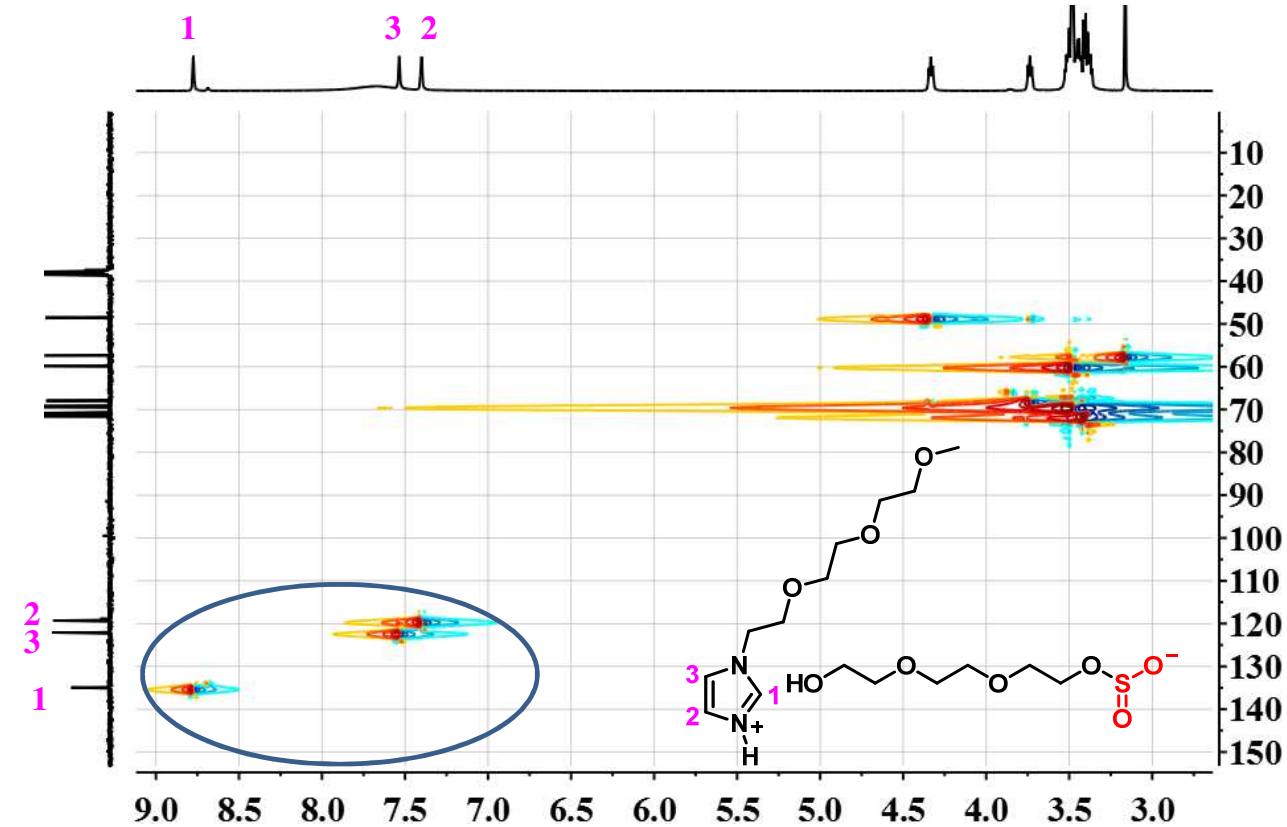


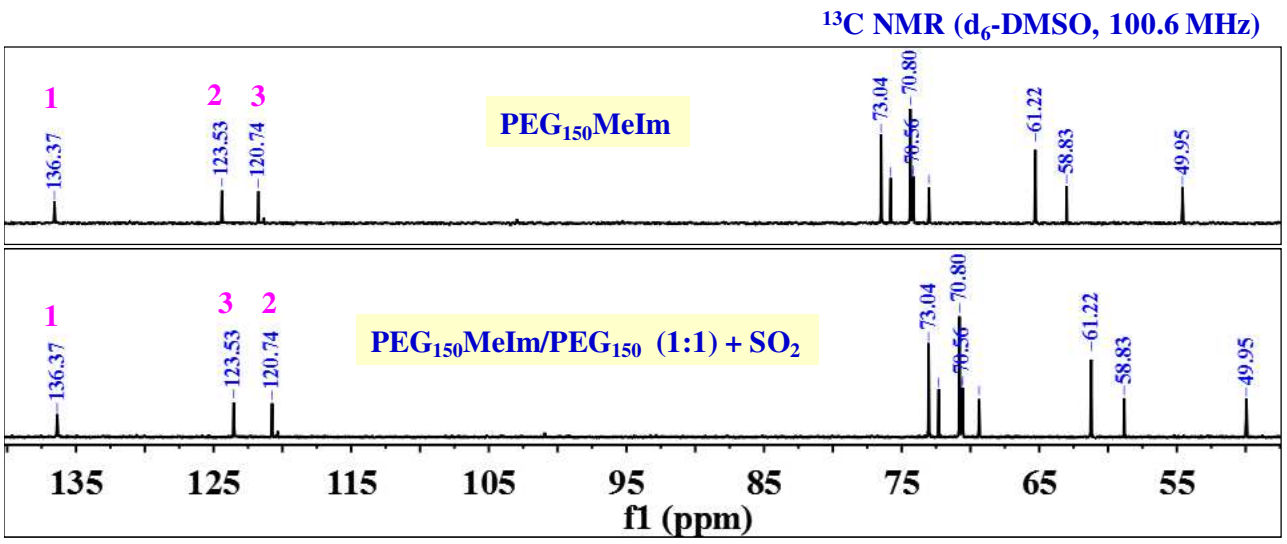
^{1}H NMR (d_6-DMSO, 400 MHz)



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



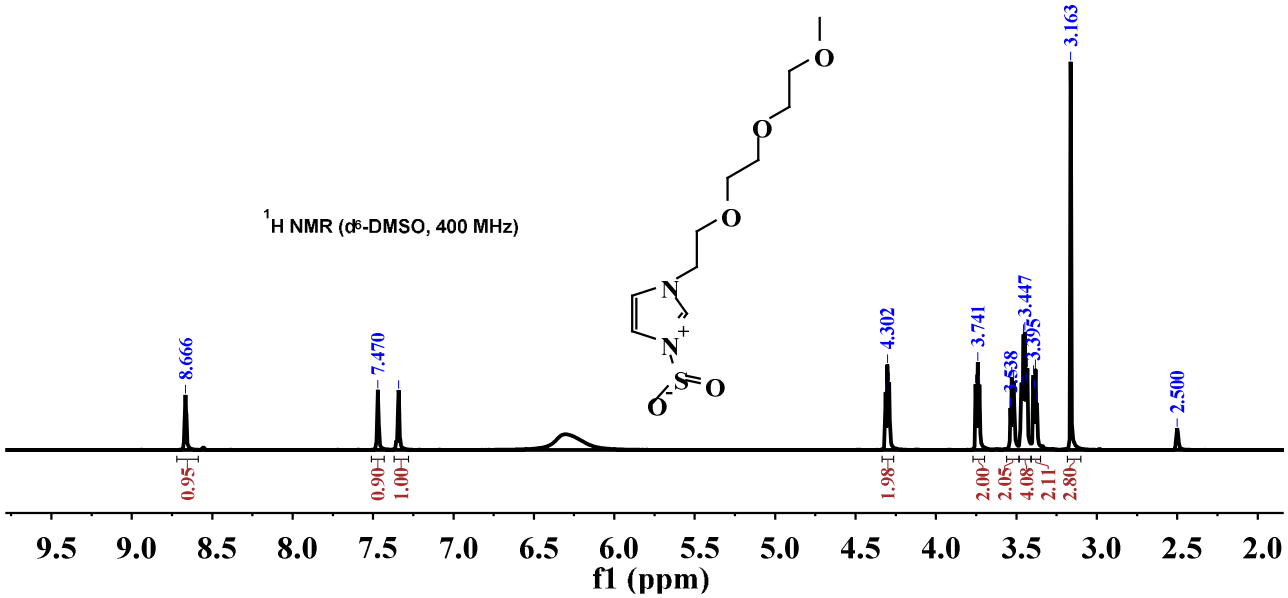


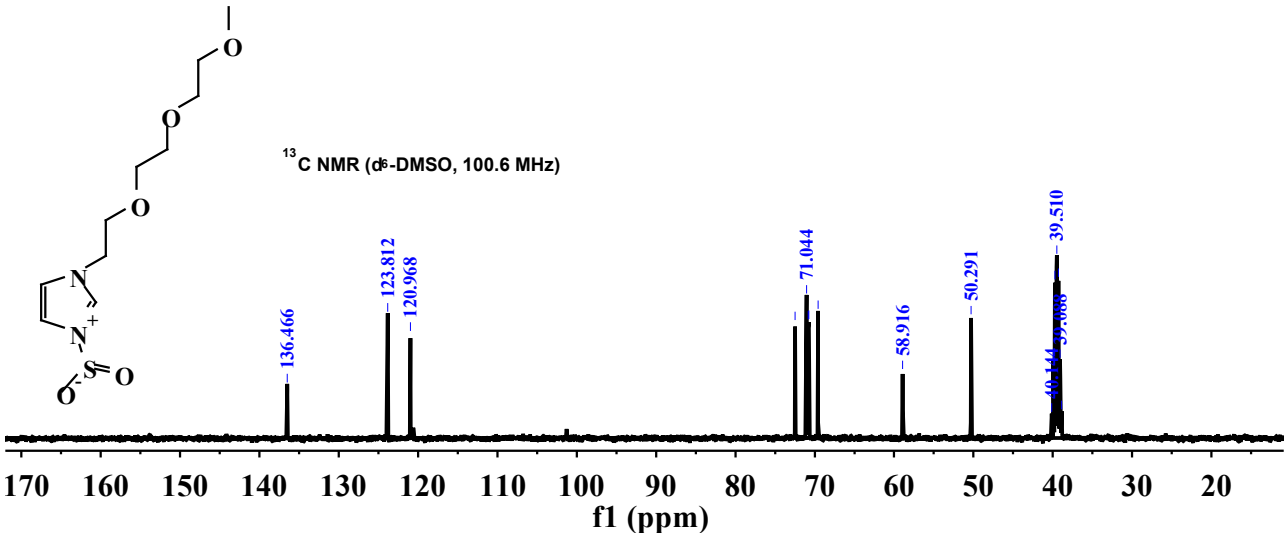


4. Characterization (^{1}H, ^{13}C NMR) of other absorption systems before and after SO_2 capture

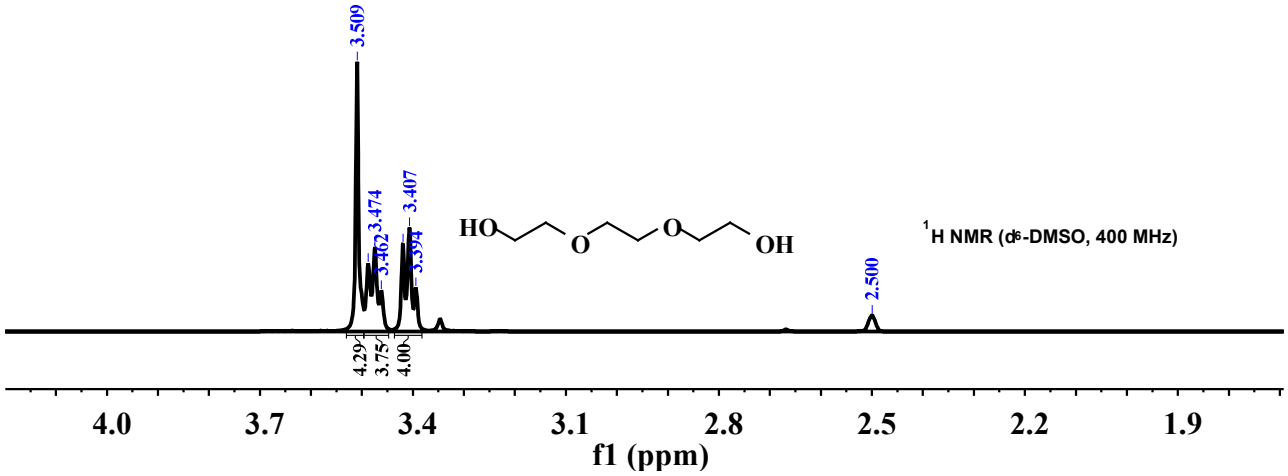
PEG_{150}MeIm + SO_2

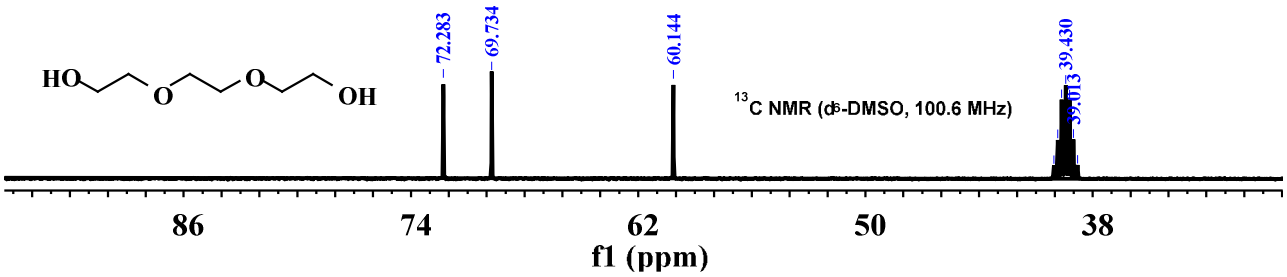
^{1}H NMR (d_6-DMSO, 400 MHz) δ 8.67 (s, 1H), 7.47 (s, 1H), 7.34 (s, 1H), 4.30 (t, 3J = 4.8 Hz, 2H), 3.74 (t, 3J = 4.8 Hz, 2H), 3.52-3.54 (m, 2H), 3.43-3.47 (m, 4H), 3.37-3.39 (m, 2H), 3.16 (s, 3H); ^{13}C NMR (d_6-DMSO, 10.6 MHz) δ 136.4, 123.7, 120.9, 72.4, 71.0, 70.8, 70.7, 69.5, 58.8, 50.2.





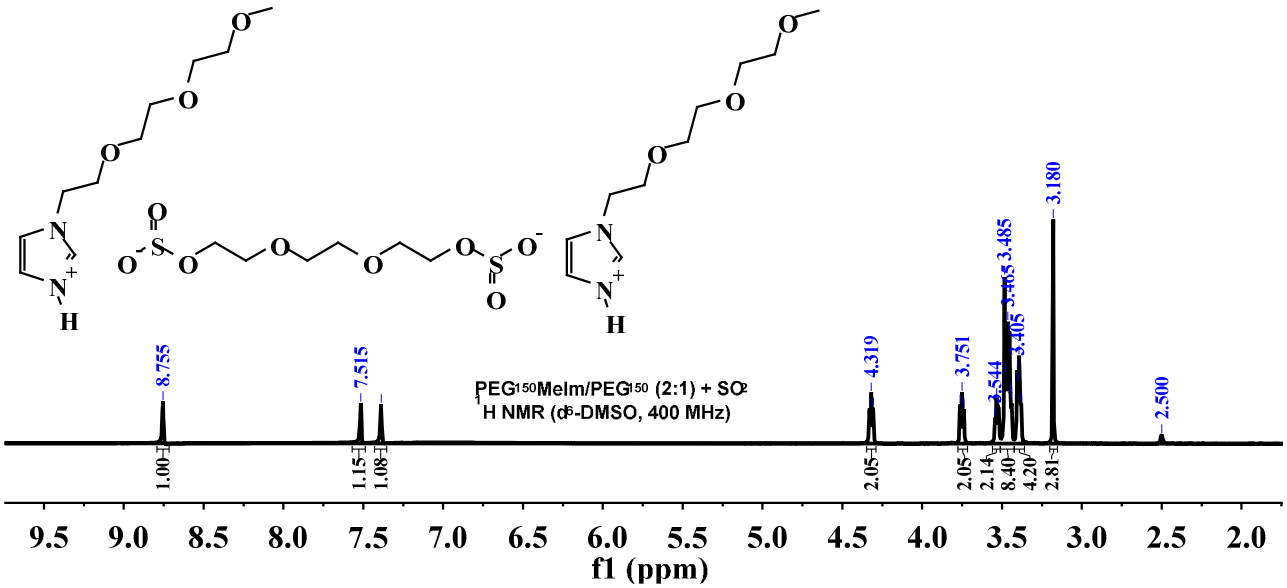
PEG$_{150}$
^{1}H NMR (400 MHz, d$_6$-DMSO) δ 4.59 (t, ^3J = 5.2 Hz, 2 H), 3.51 (s, 4 H), 3.47 (t, ^3J = 5.2 Hz, 4 H), 3.41 (t, ^3J = 5.2 Hz, 4 H); ^{13}C NMR (d$_6$-DMSO, 100.6 MHz) δ 72.3, 69.7, 60.1.

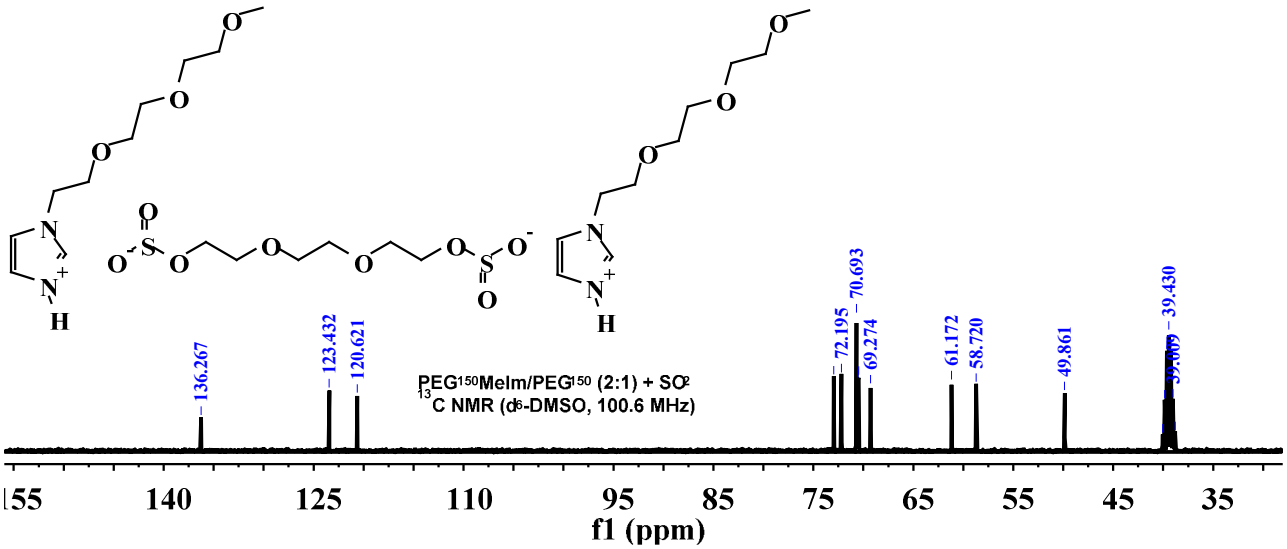




$PEG_{150}MeIm/PEG_{150}$ (2:1) + SO_2

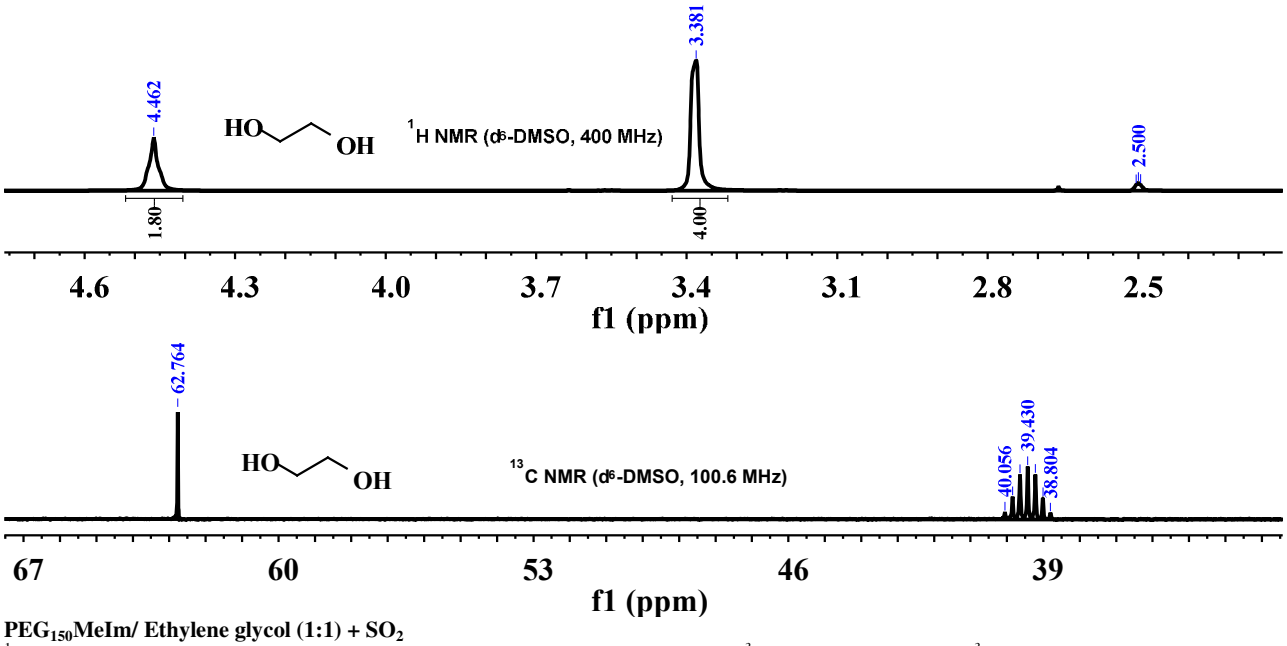
^{1}H NMR (d_6-DMSO, 400 MHz) δ 8.76 (s, 1H), 7.52 (s, 1H), 7.39 (s, 1H), 4.32 (t, ^3J = 4.4 Hz, 2H), 3.75 (t, ^3J = 4.8 Hz, 2H), 3.52-3.54 (m, 2H), 3.44-3.49 (m, 8H), 3.38-3.41 (m, 4H), 3.18 (s, 3H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 136.3, 123.4, 120.6, 73.0, 72.2, 70.7, 70.5, 70.4, 69.3, 61.2, 58.7, 49.9.





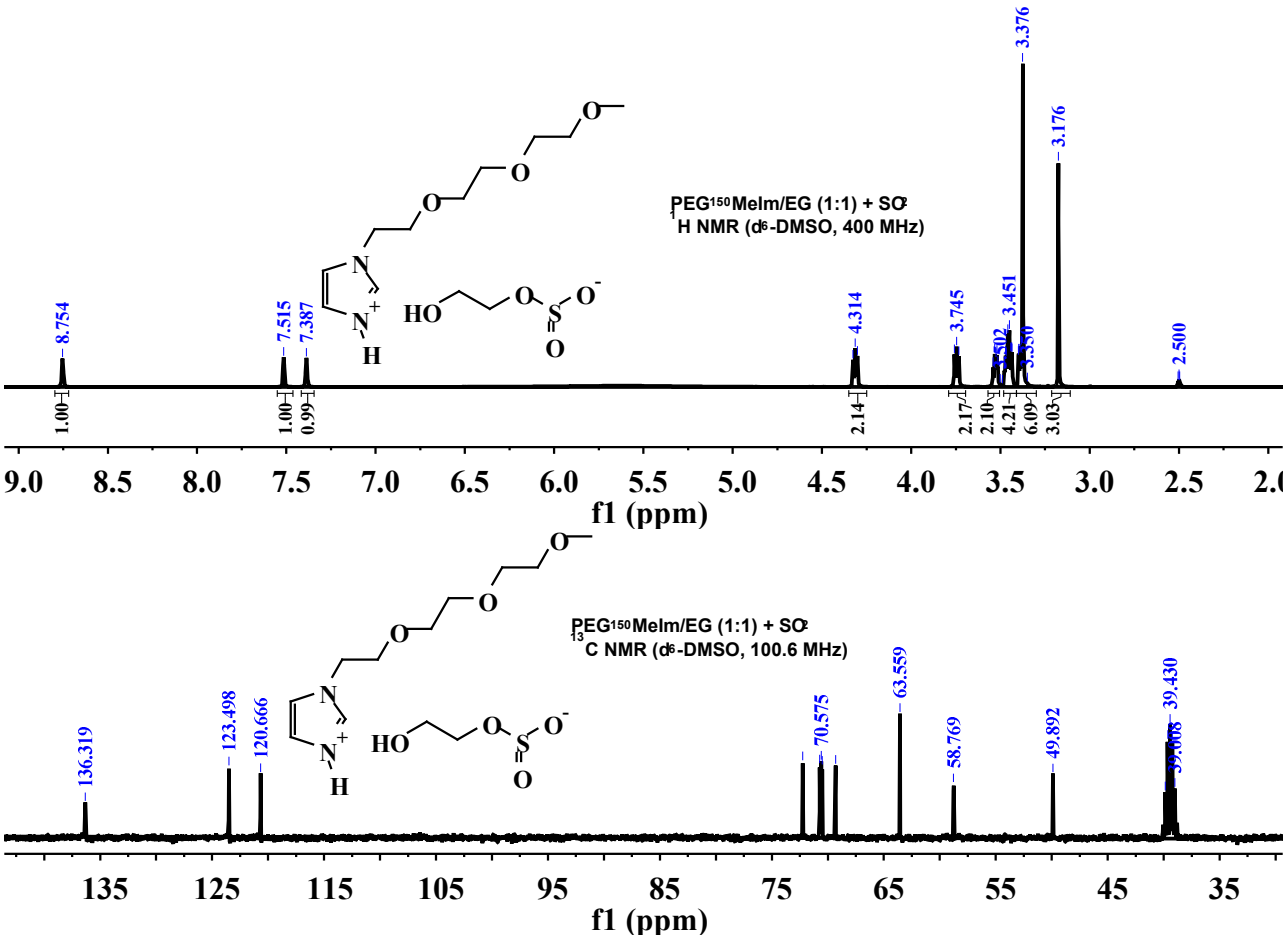
Ethylene glycol

^{1}H NMR (d_6-DMSO, 400 MHz) δ 4.46 (s, 2H), 3.38 (s, 4H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 62.8.



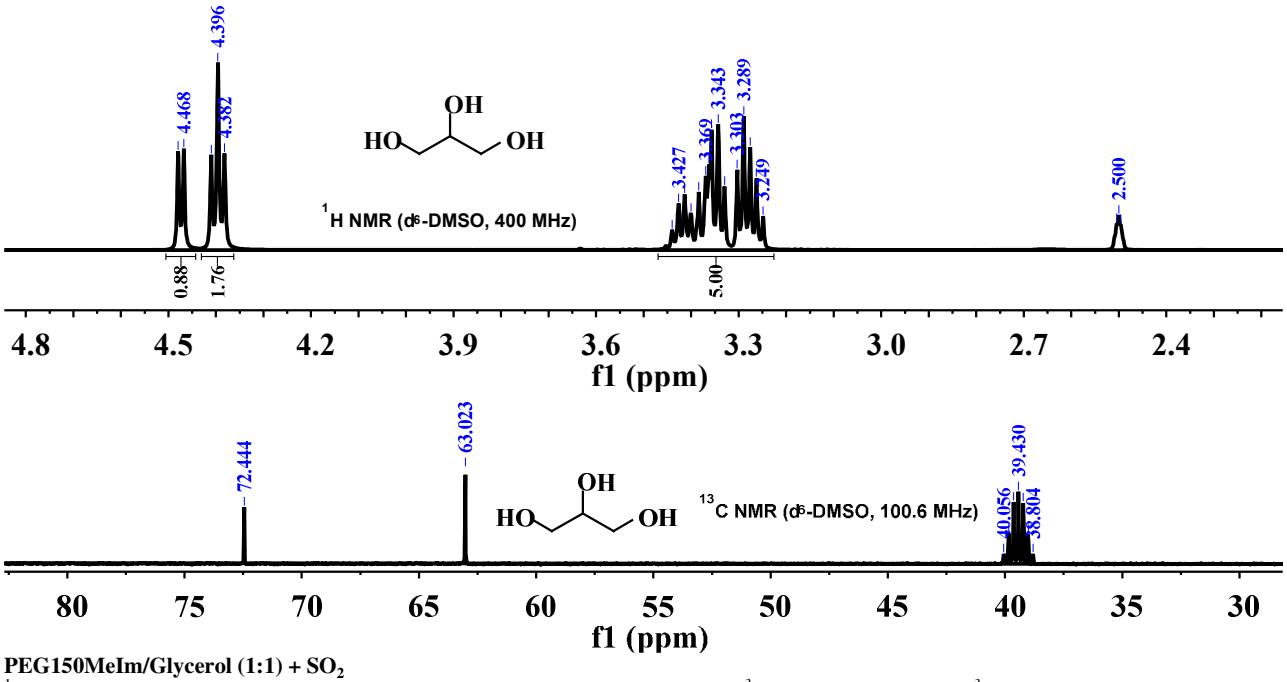
PEG$_{150}$MeIm/ Ethylene glycol (1:1) + SO$_2$

^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 8.75 (s, 1H), 7.51 (s, 1H), 7.39 (s, 1H), 4.31 (t, ^{3}J = 4.8 Hz, 2H), 3.74 (t, ^{3}J = 4.8 Hz, 2H), 3.51-3.54 (m, 2H), 3.44-3.48 (m, 4H), 3.38-3.40 (m, 6H), 3.18 (s, 3H); ^{13}C NMR (d$_6$-DMSO, 100.6 MHz) δ 136.3, 123.5, 120.7, 72.2, 70.7, 70.6, 70.5, 69.3, 63.6, 58.8, 49.9.



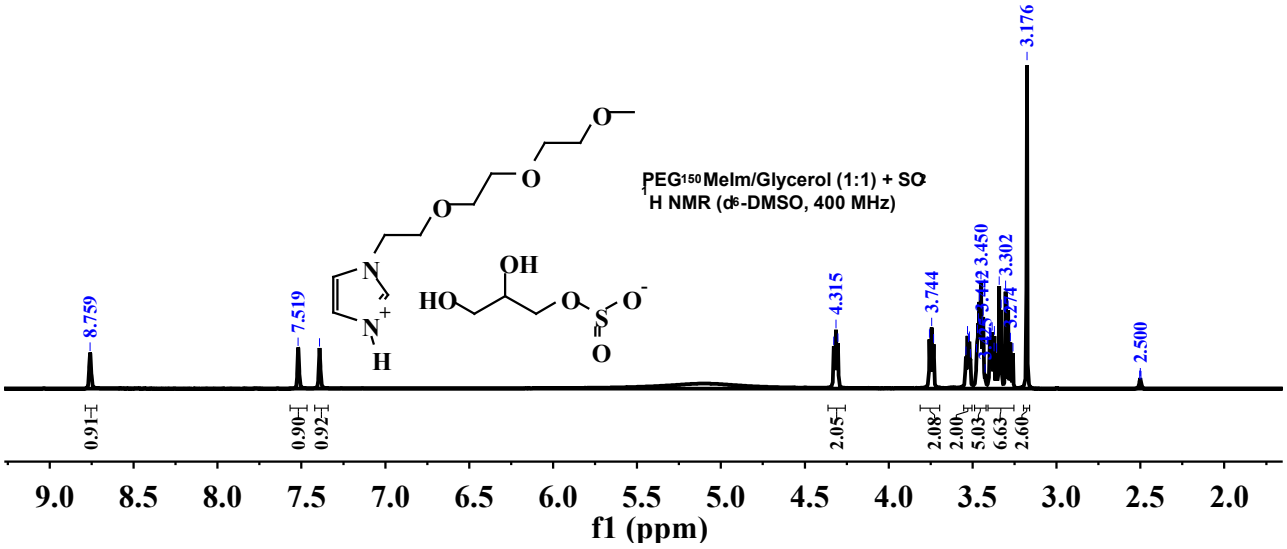
Glycerol

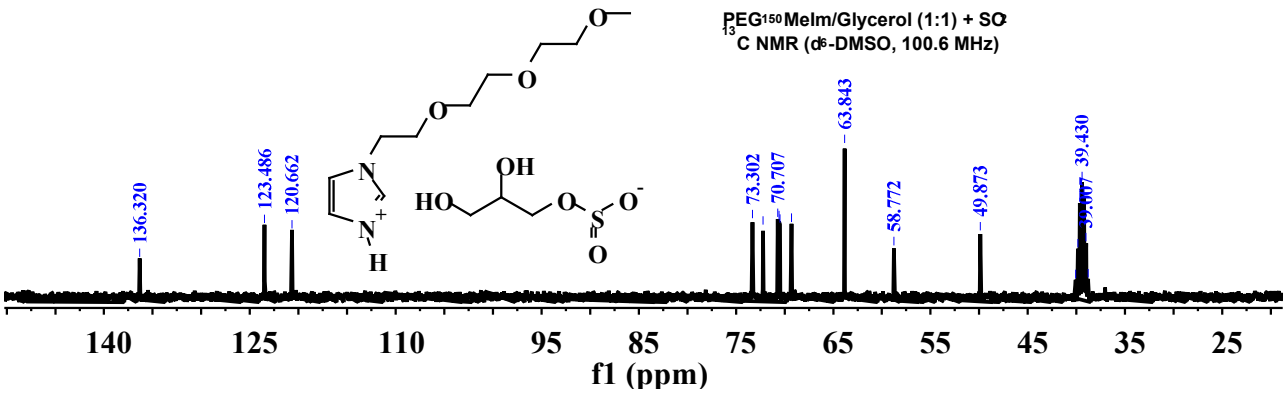
^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 4.48 (d, ^{3}J = 4.8 Hz, 1H), 4.40 (t, ^{3}J = 5.6 Hz, 2H), 3.25-3.44 (m, 5H); ^{13}C NMR (d$_6$-DMSO, 100.6 MHz) δ 72.4, 63.0.



PEG150MeIm/Glycerol (1:1) + SO_2

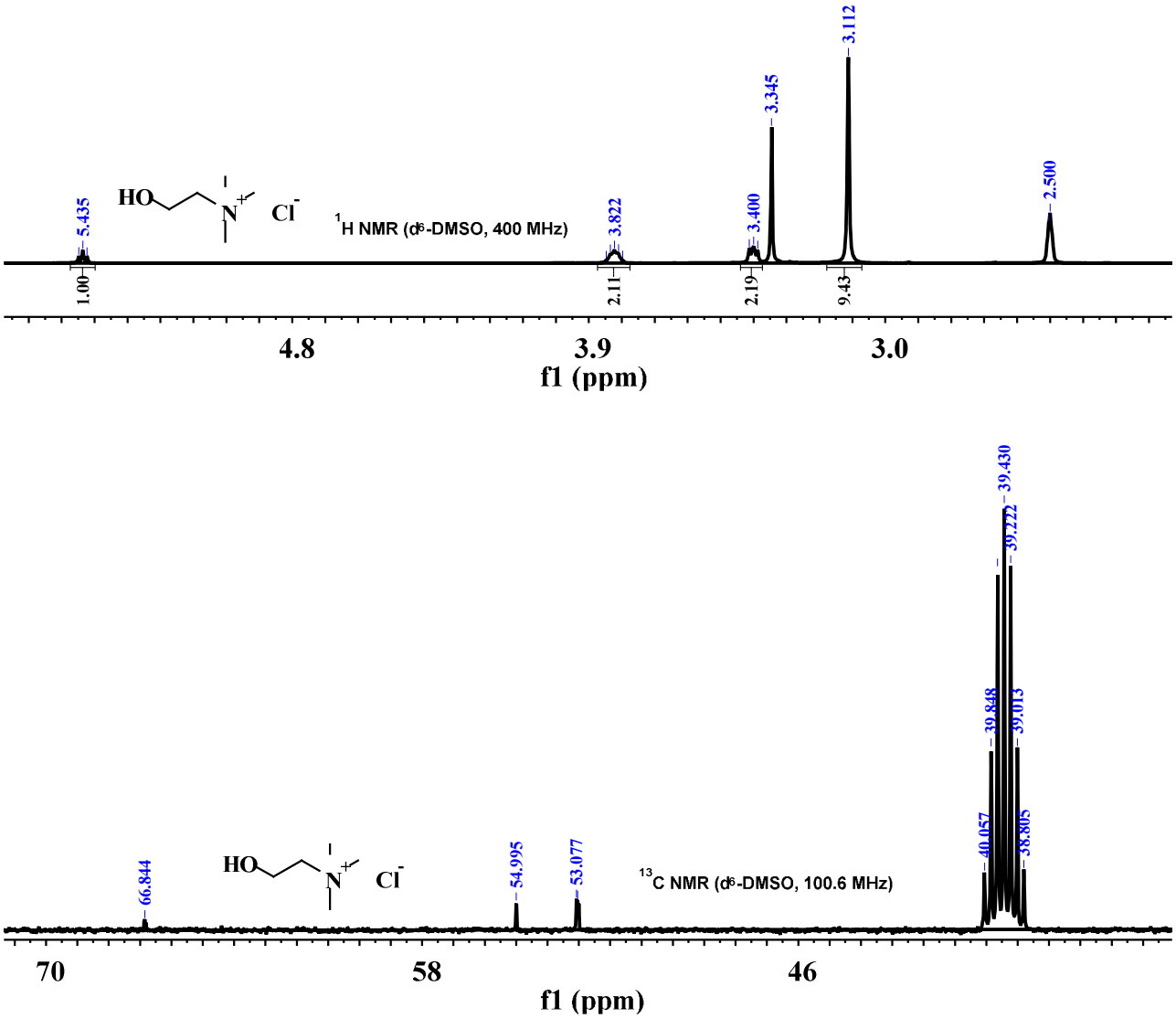
^{1}H NMR (d_6-DMSO, 400 MHz) δ 8.76 (s, 1H), 7.52 (s, 1H), 7.39 (s, 1H), 4.32 (t, ^{3}J = 4.4 Hz, 2H), 3.74 (t, ^{3}J = 4.8 Hz, 2H), 3.51-3.54 (m, 2H), 3.44-3.48 (m, 5H), 3.26-3.40 (m, 6H), 3.18 (s, 3H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 136.3, 123.5, 120.7, 73.3, 72.2, 70.7, 70.6, 70.5, 69.3, 63.8, 58.8, 49.9.





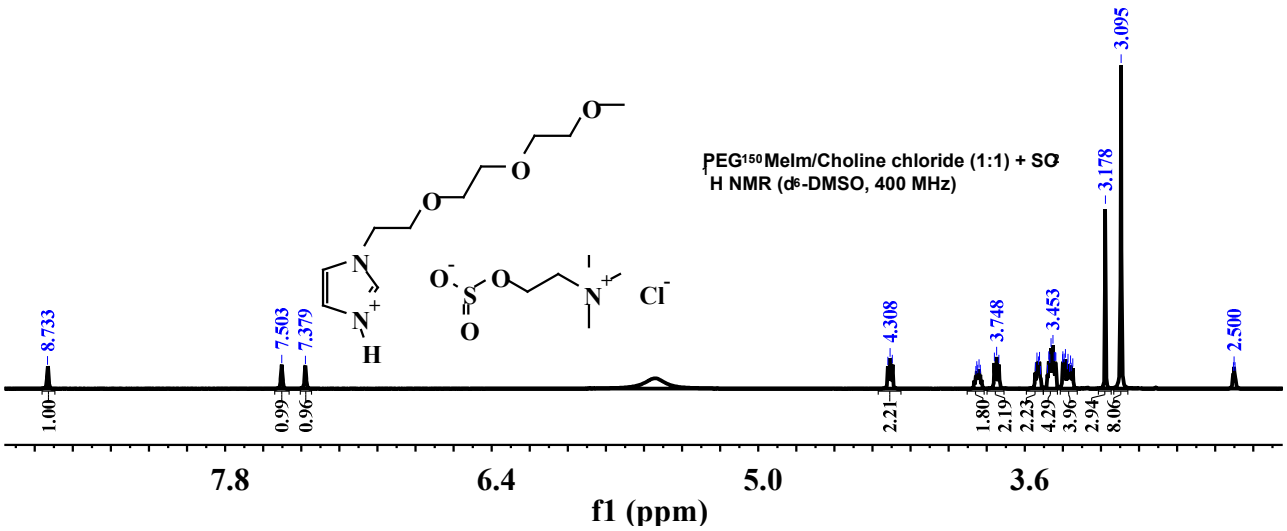
Choline chloride

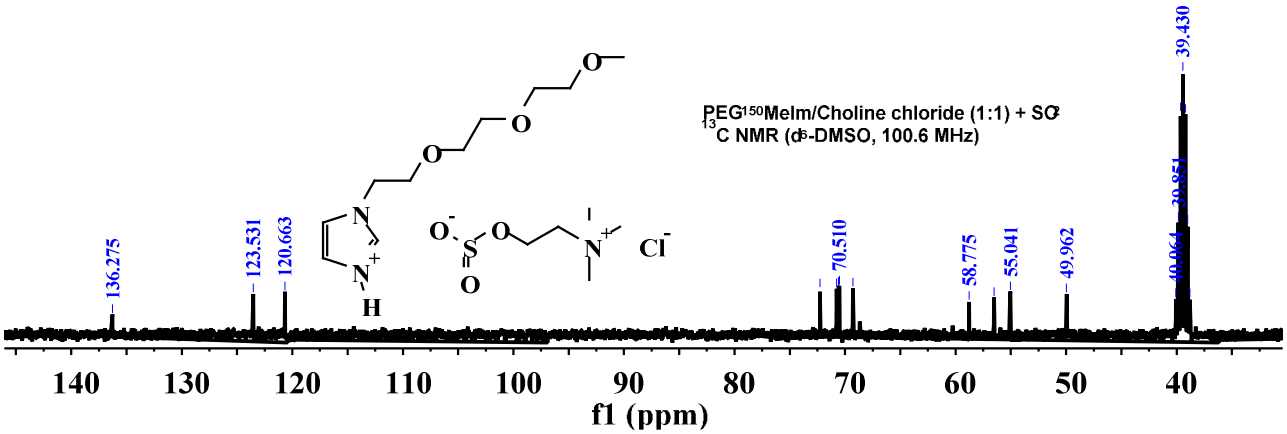
^{1}H NMR (d_6-DMSO, 400 MHz) δ 5.43 (t, ^{3}J = 4.8 Hz, 1H), 3.80-3.85 (m, 2H), 3.40 (t, ^{3}J = 5.2 Hz, 2H), 3.11 (s, 9H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 66.8, 55.0, 53.1, 53.0.



PEG150MeIm/Choline chloride (1:1) + SO_2

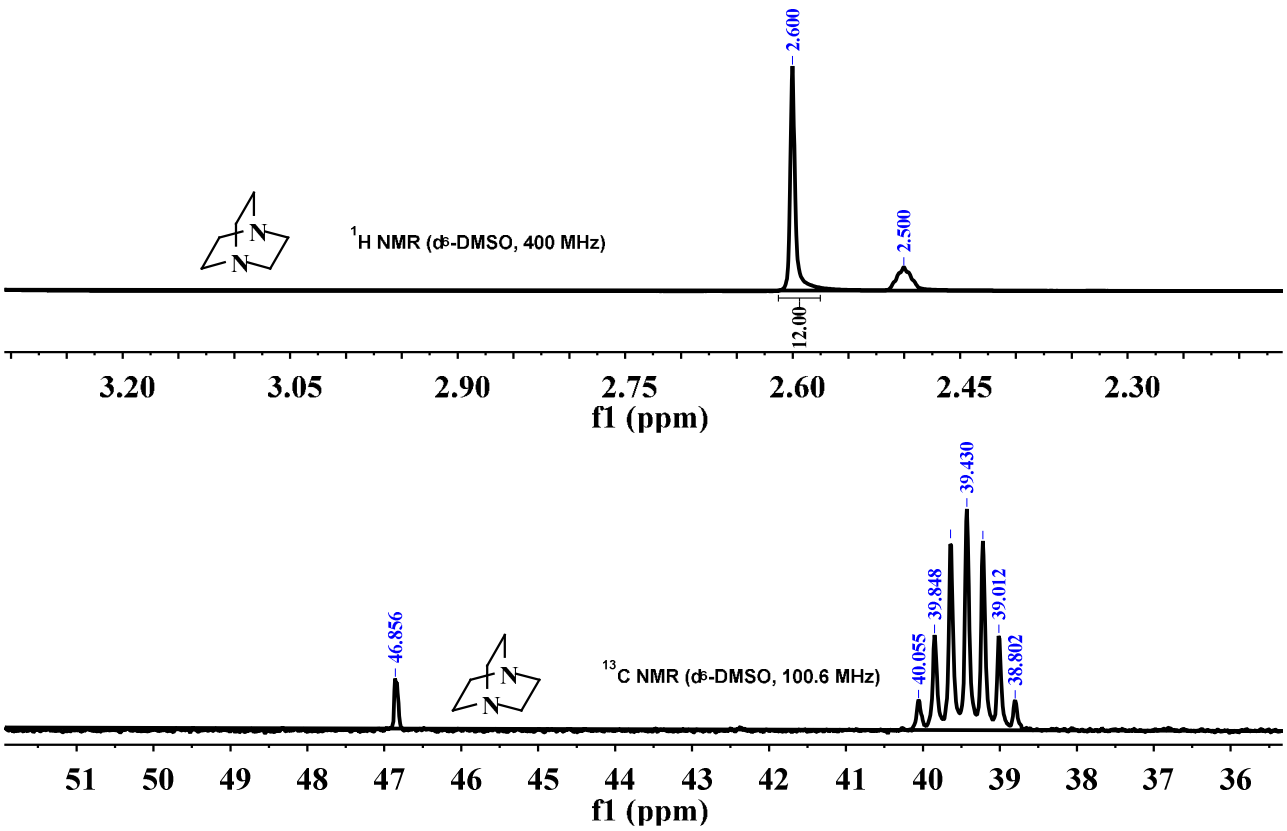
^{1}H NMR (d_6-DMSO, 400 MHz) δ 8.73 (s, 1H), 7.50 (s, 1H), 7.38 (s, 1H), 4.31 (t, ^{3}J = 4.8 Hz, 2H), 3.83-3.87 (m, 2H), 3.75 (t, ^{3}J = 5.2 Hz, 2H), 3.52-3.55 (m, 2H), 3.44-3.48 (m, 4H), 3.35-3.40 (m, 4H), 3.18 (s, 3H), 3.09 (s, 9H); ^{13}C NMR (d_6-DMSO, 100.6 MHz) δ 136.3, 123.5, 120.7, 72.3, 70.8, 70.6, 70.5, 69.3, 58.8, 56.5, 55.1, 55.0, 50.0.





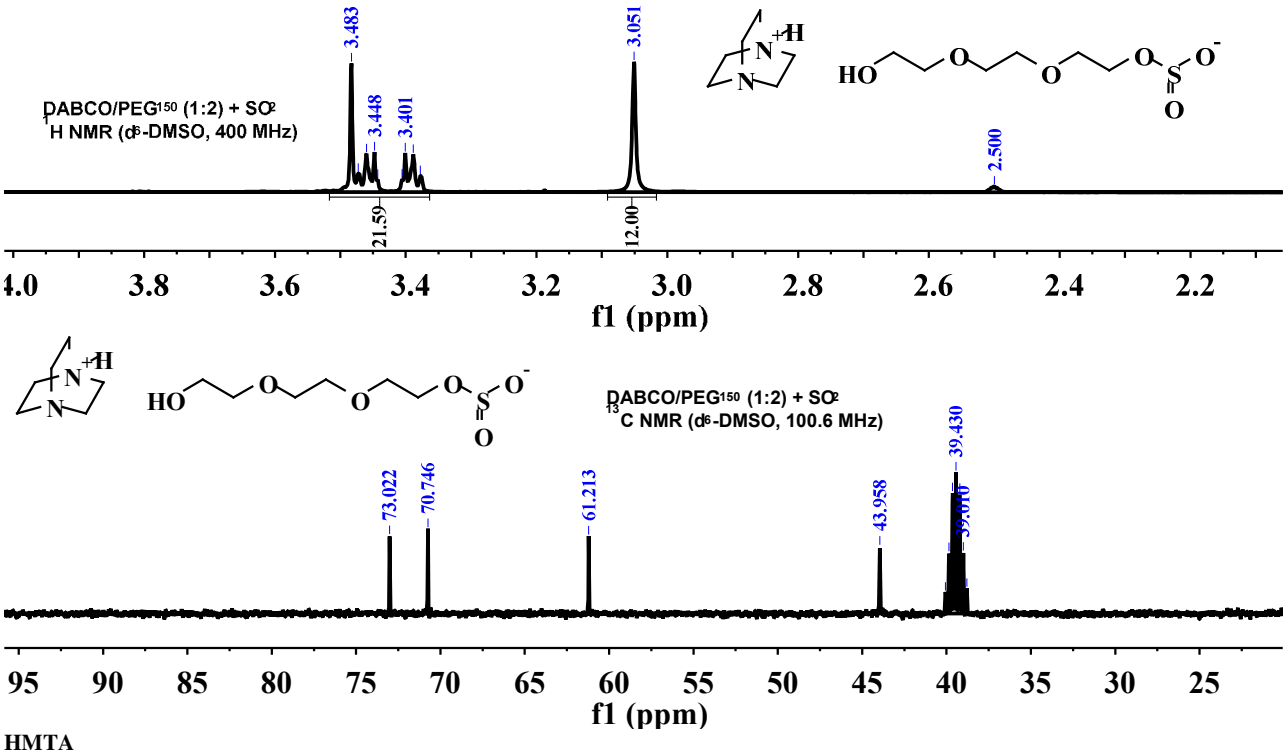
DABCO

^{1}H NMR (d_6-DMSO, 400 MHz) δ 2.60 (s, 12H); ^{13}C NMR (d_6-DMSO, 10.6 MHz) δ 46.9.



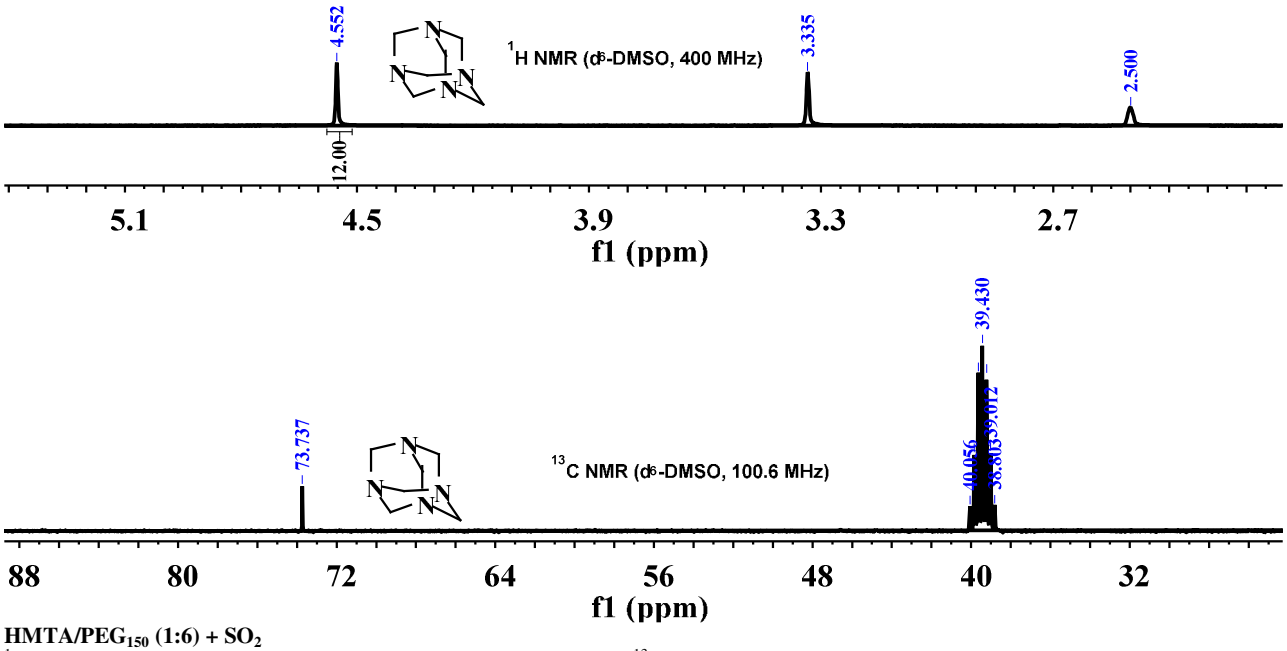
DABCO/PEG$_{150}$ (1:2) + SO$_2$

^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 3.37-3.48 (m, 22H), 3.05 (s, 12H); ^{13}C NMR (d$_6$-DMSO, 10.6 MHz) δ 73.0, 70.7, 61.2, 44.0.



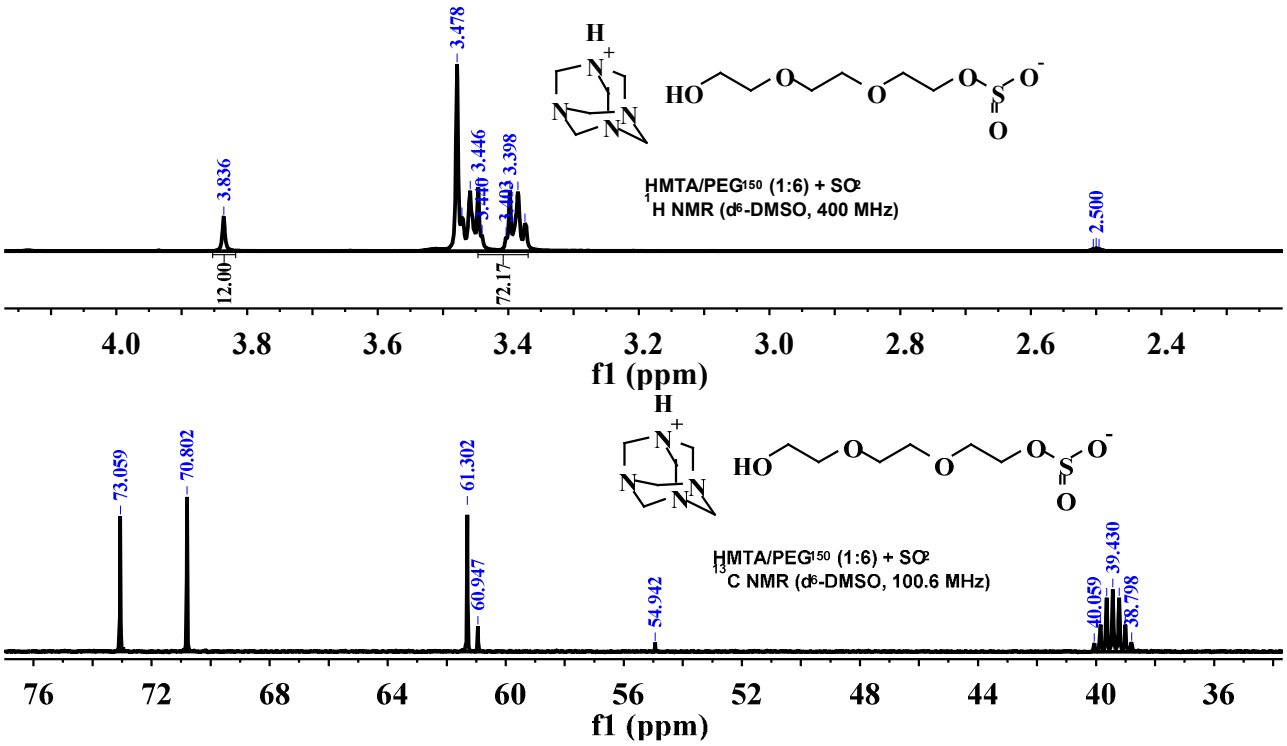
HMTA

^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 4.55 (s, 12H); ^{13}C NMR (d$_6$-DMSO, 10.6 MHz) δ 73.7.



HMTA/PEG$_{150}$ (1:6) + SO$_2$

^{1}H NMR (d$_6$-DMSO, 400 MHz) δ 3.84 (s, 12H), 3.37-3.48 (m, 72H); ^{13}C NMR (d$_6$-DMSO, 10.6 MHz) δ 73.1, 70.8, 61.3, 60.9, 54.9.



5. Reaction conditions screening for the synthesis of propylene sulfite from propylene oxide and SO$_2$ absorbed by PEG$_{150}$MeIm/PEG$_{150}$ or PEG$_{150}$MeIm/Choline chloride

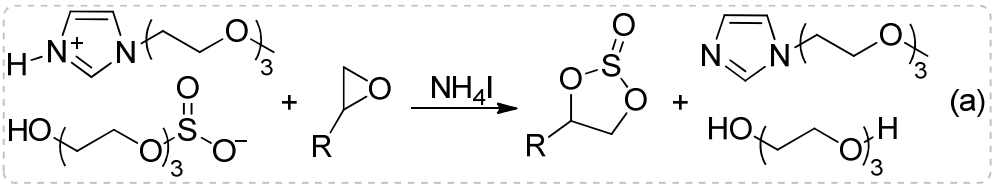


Table S1 Reaction conditions screening for the synthesis of propylene sulfite (PS) from propylene oxide (PO) and SO_2 absorbed by $PEG_{150}MeIm/PEG_{150}$[a]

Entry	Catalyst	Temperature/°C	Catalyst loading/mol%	Time/h	PO Conv./%[b]	PS Yield/%[b]
1	-	80	5	3	>99	50
2	Me_4NCl	80	5	3	>99	41
3	Me_4NBr	80	5	3	>99	46
4	*n*-Pr_4NBr	80	5	3	>99	64
5	*n*-Bu_4NBr	80	5	3	>99	65
6	NH_4I	80	5	3	>99	64
7	NH_4I	60	5	3	>99	32
8	NH_4I	100	5	3	>99	40
9	NH_4I	120	5	3	>99	16
10	NH_4I	140	5	3	>99	18
11	NH_4I	80	1	3	>99	28
12	NH_4I	80	3	3	>99	48
13	NH_4I	80	7	3	>99	55
14	NH_4I	80	10	3	>99	47
15	NH_4I	80	5	1	>99	35
16	NH_4I	80	5	2	>99	57
17	NH_4I	80	5	6	>99	51
18	NH_4I	80	5	9	>99	58
19	NH_4I	80	5	12	>99	57
20[c]	NH4I	80	5	3	>99	19

[a] $PEG_{150}MeIm/PEG_{150}$ (molar ratio 1:1), 2 mmol; SO_2 absorbed, 8.0 mmol under 1 bar SO_2 pressure; PO, 5 mmol. [b] Determined by GC with biphenyl as an internal standard. [c] PEG_{150}, 4 mmol; SO_2, 8.0 mmol; PO, 5 mmol.

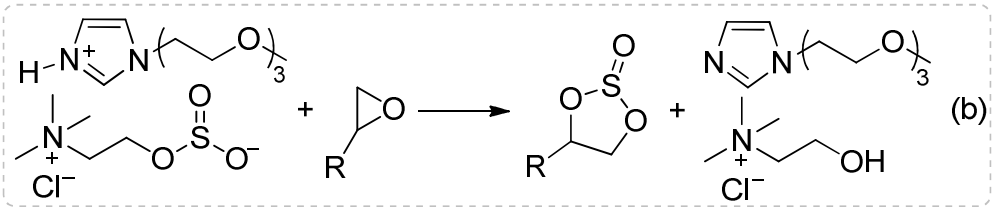
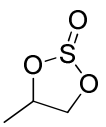


Table S2 Reaction conditions screening for the synthesis of propylene sulfite (PS) from propylene oxide (PO) and SO_2 absorbed by $PEG_{150}MeIm$/Choline chloride[a]

Entry	Temperature/°C	Absorbents/mmol	Time/h	PO Conv./%[b]	PS Yield/%[b]
1	25	2	3	89	38
2	60	2	3	97	44
3	80	2	3	>99	57
4	100	2	3	>99	56
5	120	2	3	>99	57
6	140	2	3	>99	54
7	80	3	3	98	52
8	80	4	3	>99	41
9[c]	80	2	3	98	56
10[d]	80	2	3	>99	47
11	80	2	2	99	53
12	80	2	6	98	62
13	80	2	9	98	52
14[e]	80	-	6	>99	54

[a] $PEG_{150}MeIm$/Choline chloride (molar ratio 1:1); SO_2 absorbed, 10.4 mmol under 1 bar SO_2 pressure; PO, 10 mmol. [b] Determined by GC with biphenyl as an internal standard. [c] $PEG_{150}MeIm$, 2 mmol; Choline chloride, 4 mmol. [d] PO, 5 mmol. [e] PEG_{150}, 2 mmol; Choline chloride, 2 mmol; SO_2, 10.4 mmol; PO 10 mmol.

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



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

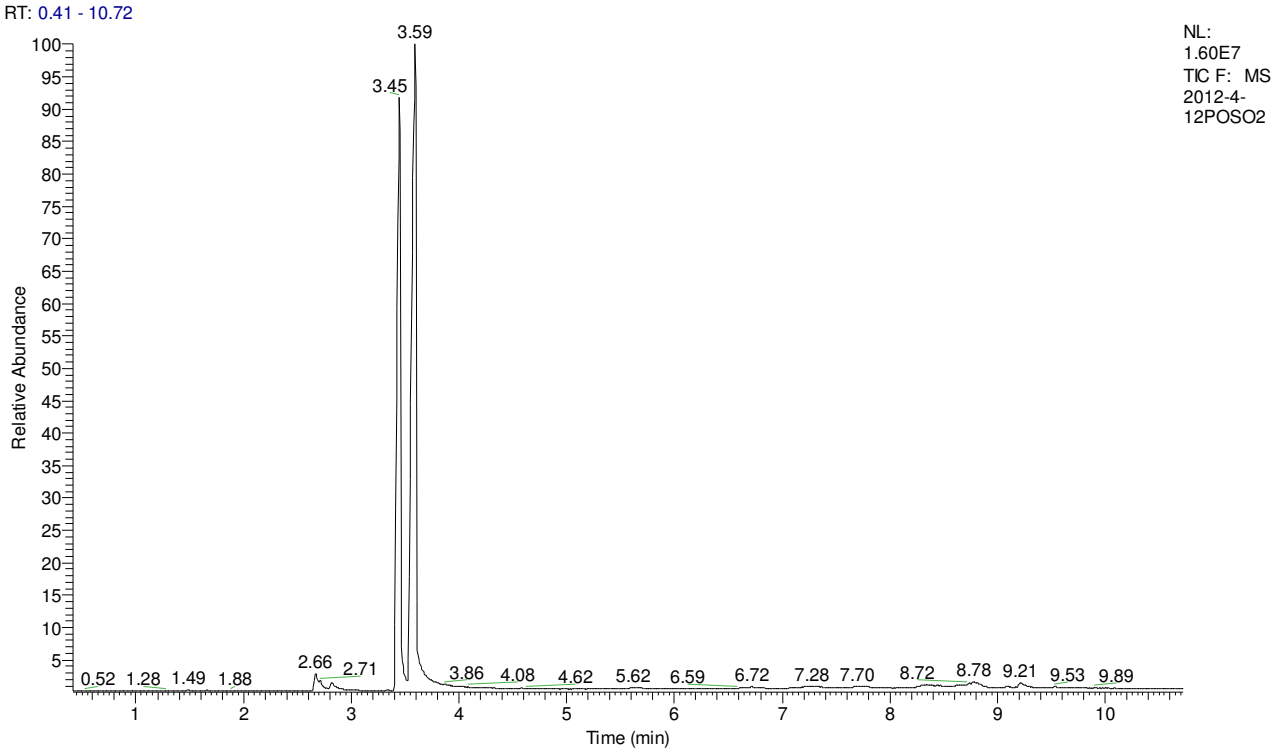
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 3:1; Light yellow liquid.

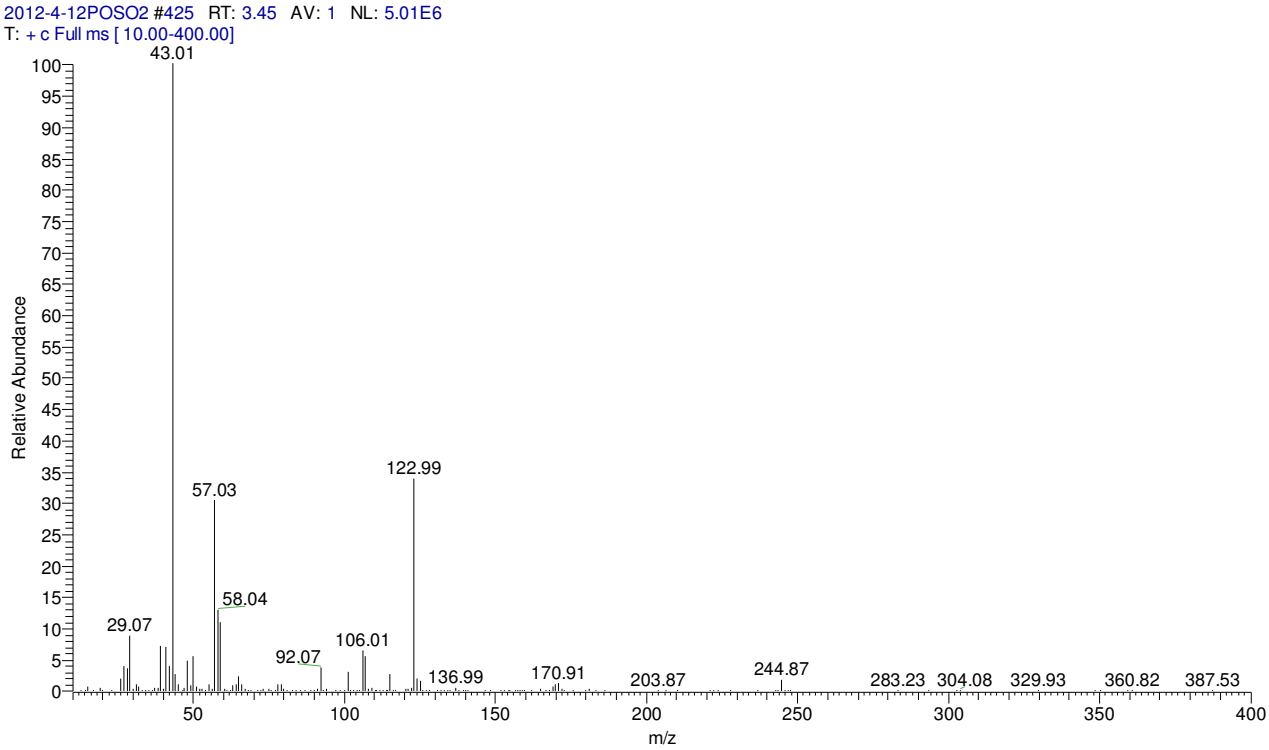
Isomer 1 (RT 3.45 min)

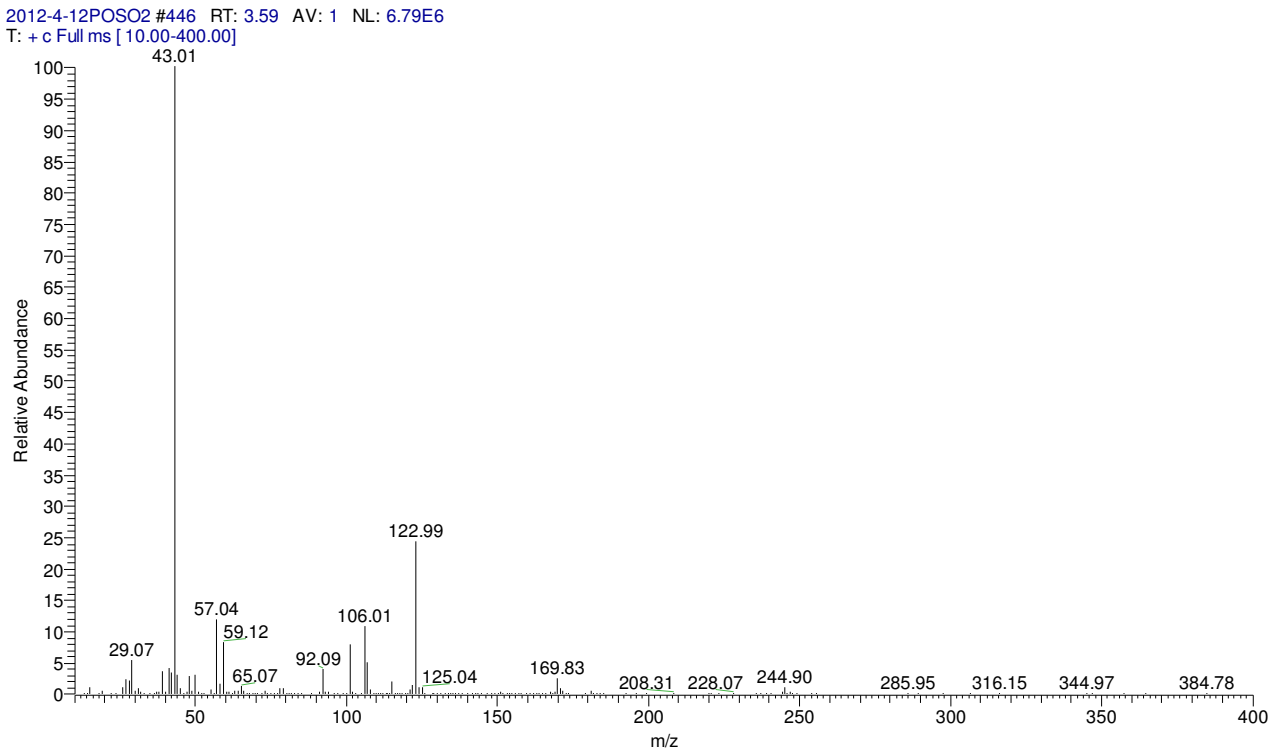
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.58-4.65 (m, 1H), 4.49-4.52 (m, 1H), 4.29 (t, ^{3}J = 8.8 Hz, 1H), 1.61 (d, ^{3}J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.2, 72.8, 18.7; GC-MS: m/z (%): 122.99 (34) [M^+], 57.03 (30) [M^+-O_2S], 43.01 (100) [M^+-O_3S].

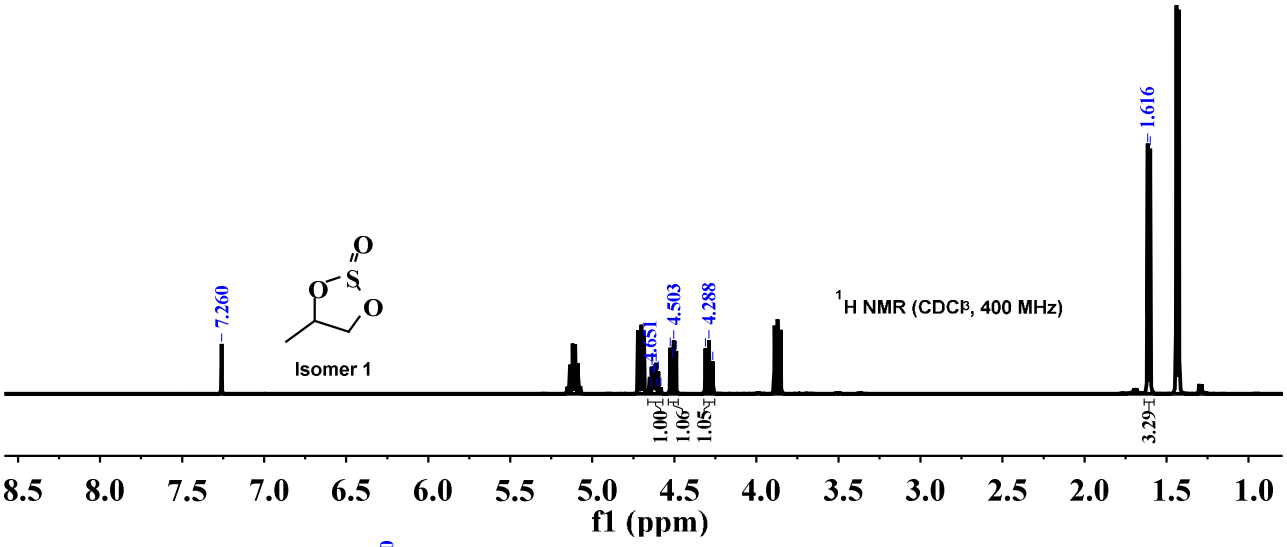
Isomer 2 (RT 3.59 min)

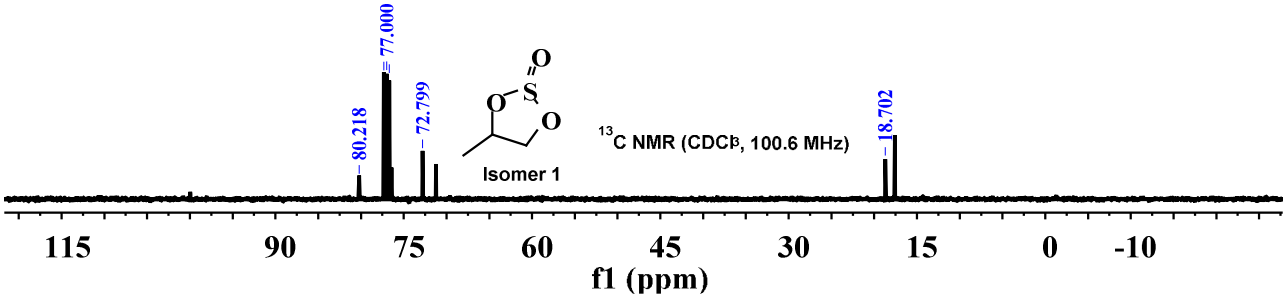
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.07-5.15 (m, 1H), 4.69-4.72 (m, 1H), 3.85-3.89 (m, 1H), 1.43 (d, ^{3}J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 17.6, 71.2, 76.4; GC-MS: m/z (%): 122.99 (24) [M^+], 57.04 (12) [M^+-O_2S], 43.01 (100) [M^+-O_3S].

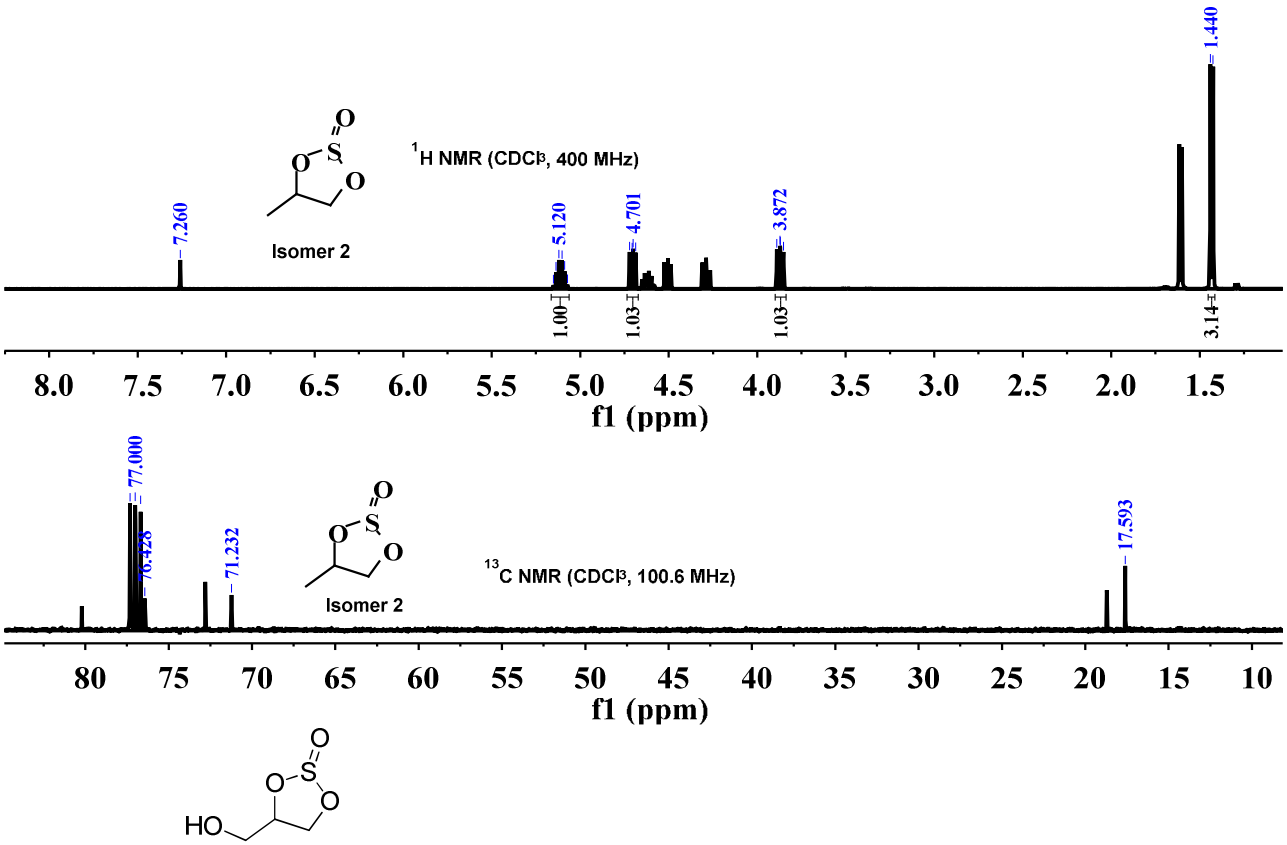





2012-4-12POSO2 #446 RT: 3.59 AV: 1 NL: 6.79E6
T: + c Full ms [10.00-400.00]
Relative Abundance
m/z
43.01
122.99
57.04
59.12
106.01
29.07
92.09
65.07
125.04
169.83
208.31
228.07
244.90
285.95
316.15
344.97
384.78


7.260
4.503
4.288
1.616
Isomer 1
1H NMR (CDCl3, 400 MHz)
1.00
1.06
1.05
3.29
f1 (ppm)


80.218
77.000
72.799
18.702
Isomer 1
13C NMR (CDCl3, 100.6 MHz)
f1 (ppm)



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

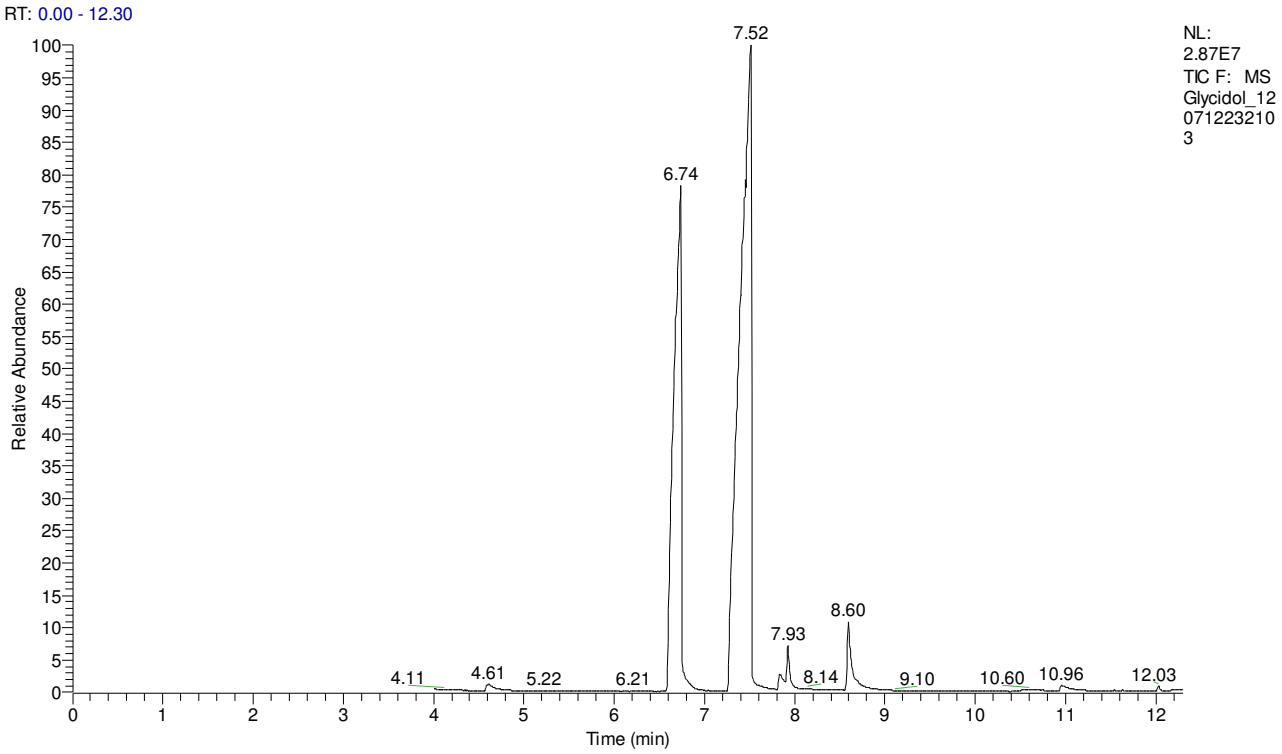
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 1:3; Light yellow liquid.

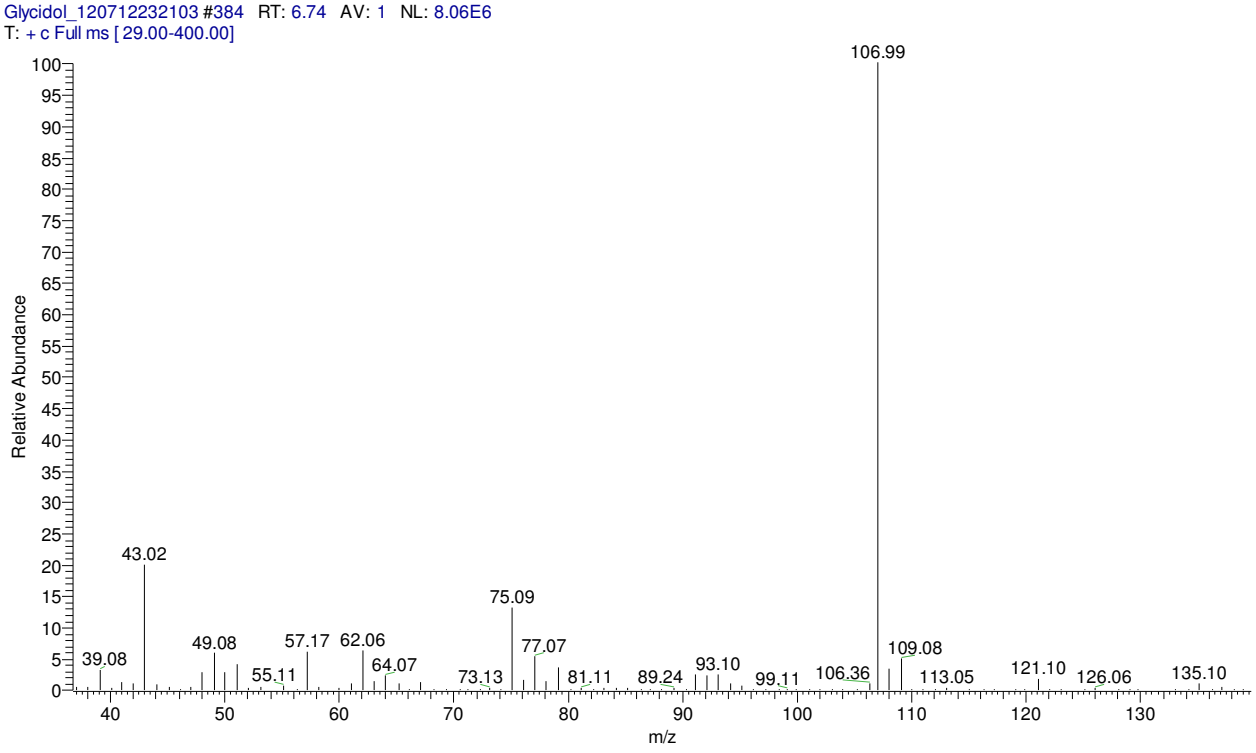
Isomer 1 (RT 6.74 min)

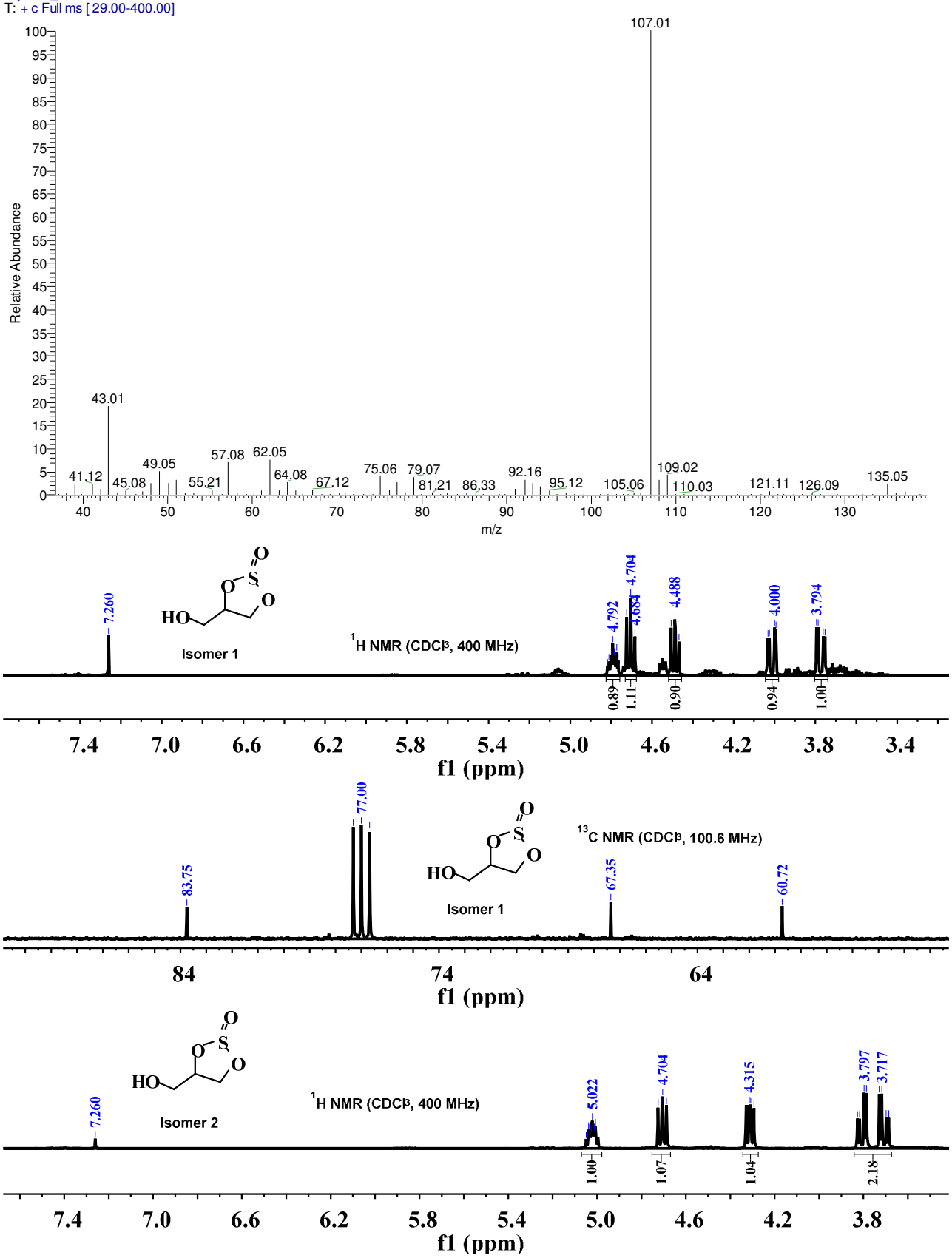
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.77-4.82 (m, 1H), 4.70 (t, ^{3}J = 8 Hz, 1H), 4.49 (t, ^{3}J = 7.2 Hz, 1H), 3.99-4.03 (m, 1H), 3.75-3.79 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 83.7, 67.3, 60.7; GC-MS: m/z (%):106.98 (100) [M^+-CH_3O].

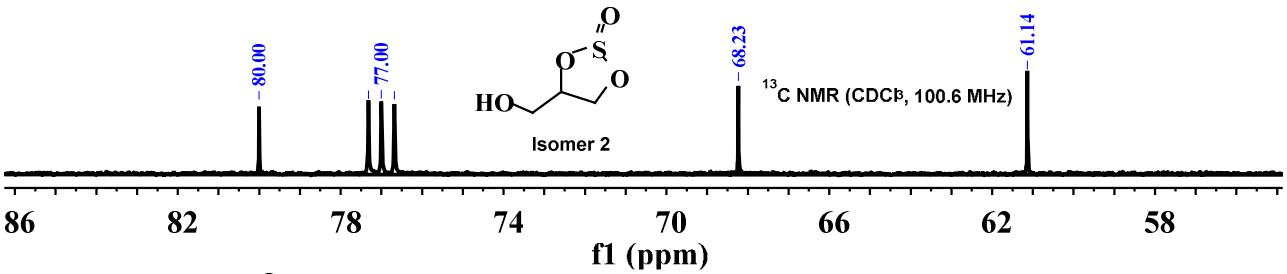
Isomer 2 (RT 7.52 min)

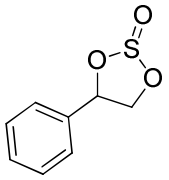
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.00-5.05 (m, 1H), 4.69-4.72 (m, 1H), 4.29-4.33 (m, 1H), 3.69-3.83 (m, 2H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.0, 68.2, 61.1; GC-MS: m/z (%):107.01 (100) [M^+-CH_3O].


RT: 0.00 - 12.30
NL:
2.87E7
TIC F: MS
Glycidol_12
071223210
3
Relative Abundance
Time (min)
7.52
6.74
8.60
7.93
4.11
4.61
5.22
6.21
8.14
9.10
10.60
10.96
12.03


Glycidol_120712232103 #384 RT: 6.74 AV: 1 NL: 8.06E6
T: + c Full ms [29.00-400.00]
Relative Abundance
m/z
106.99
43.02
75.09
49.08
57.17
62.06
77.07
39.08
64.07
55.11
73.13
81.11
89.24
93.10
99.11
106.36
109.08
113.05
121.10
126.06
135.10


Glycidol_120712232103 #498 RT: 7.52 AV: 1 NL: 4.79E6
T: + c Full ms [29.00-400.00]
Relative Abundance
m/z
107.01
43.01
41.12
45.08
49.05
55.21
57.08
62.05
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
Isomer 1
1H NMR (CDCl3, 400 MHz)
7.260
4.792
4.684
4.704
4.488
4.000
3.794
0.89
1.11
0.90
0.94
1.00
f1 (ppm)
Isomer 1
13C NMR (CDCl3, 100.6 MHz)
83.75
77.00
67.35
60.72
f1 (ppm)
Isomer 2
1H NMR (CDCl3, 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

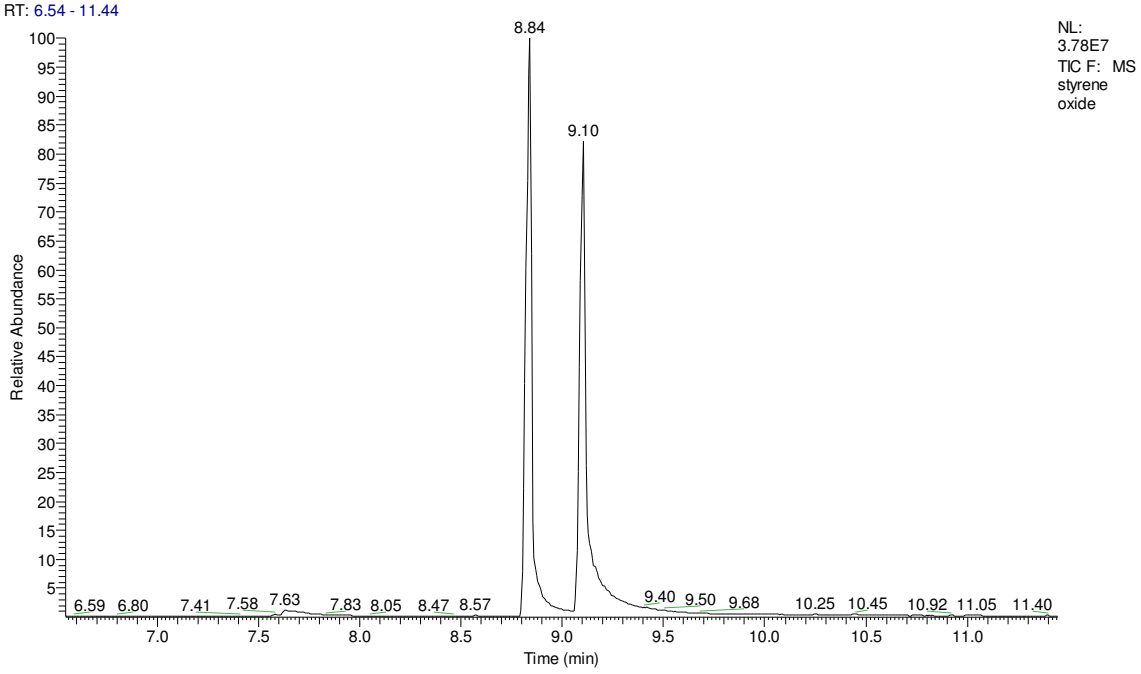
Column chromatography on silica gel: eluting with petroleum ether/dichloromethane 2:1; Light yellow liquid.

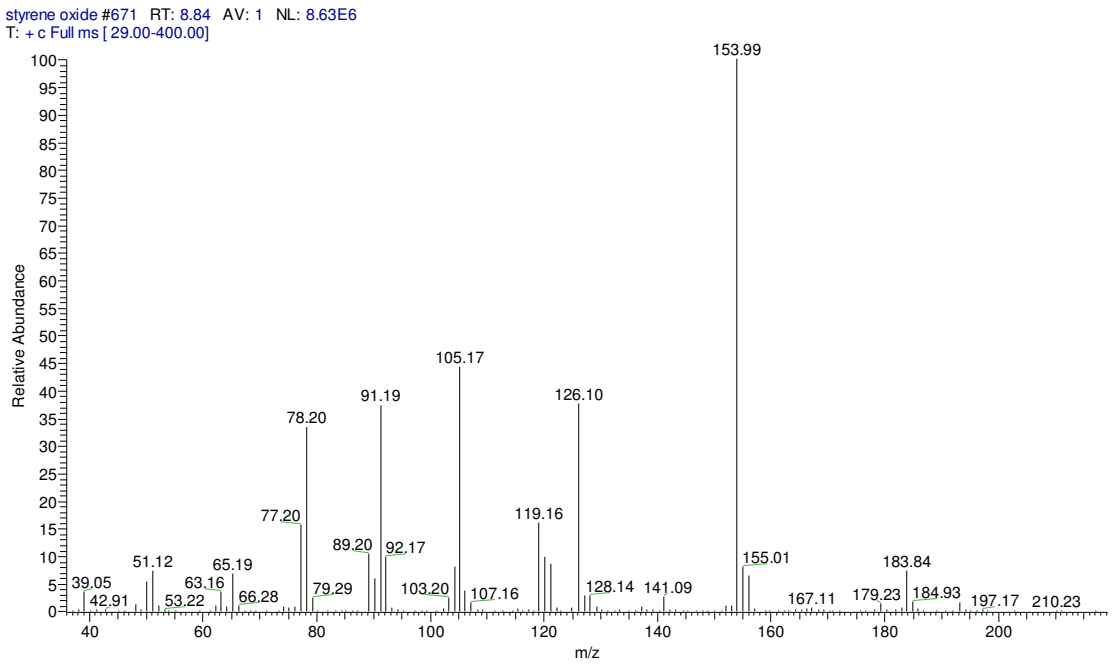
Isomer 1 (RT 8.84 min)

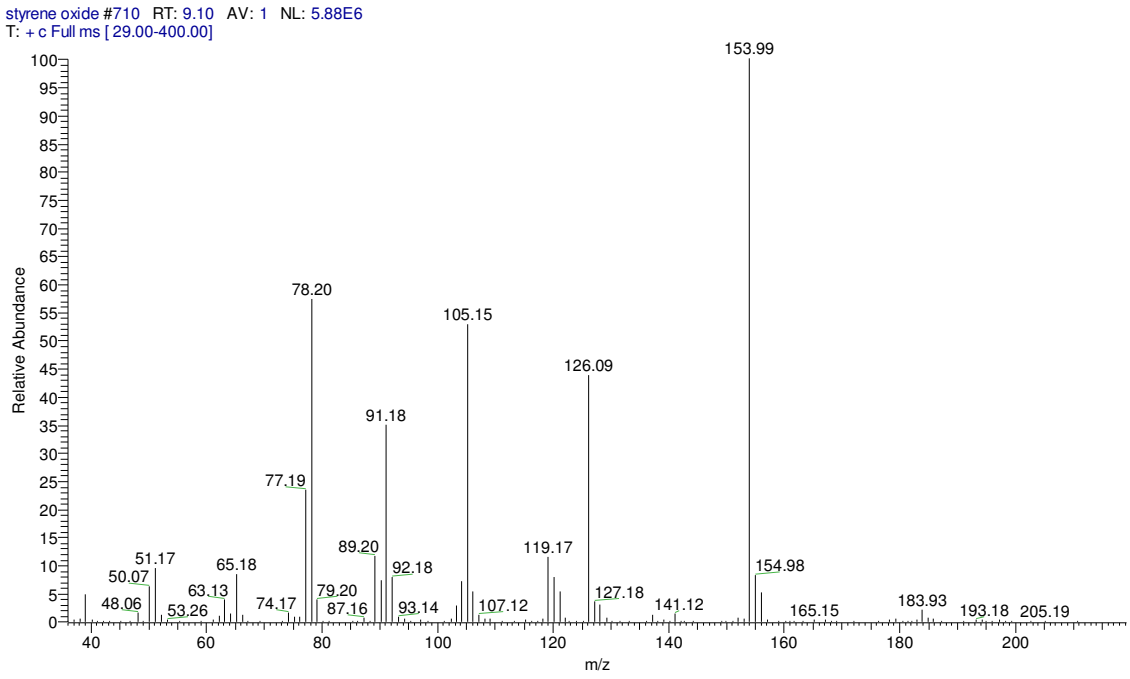
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40-7.50 (m, 5H), 5.40-5.44 (m, 1H), 4.73-4.77 (m, 1H), 4.46-4.51 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 129.4, 129.0, 127.5, 126.6, 85.5, 71.4; GC-MS: m/z (%): 183.84 (7) [M^+], 153.99 (100) [M^+-OCH_2], 126.10 (38) [M^+-C_3H_6O], 119.16 (16) [M^+-C_5H_5], 105.17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

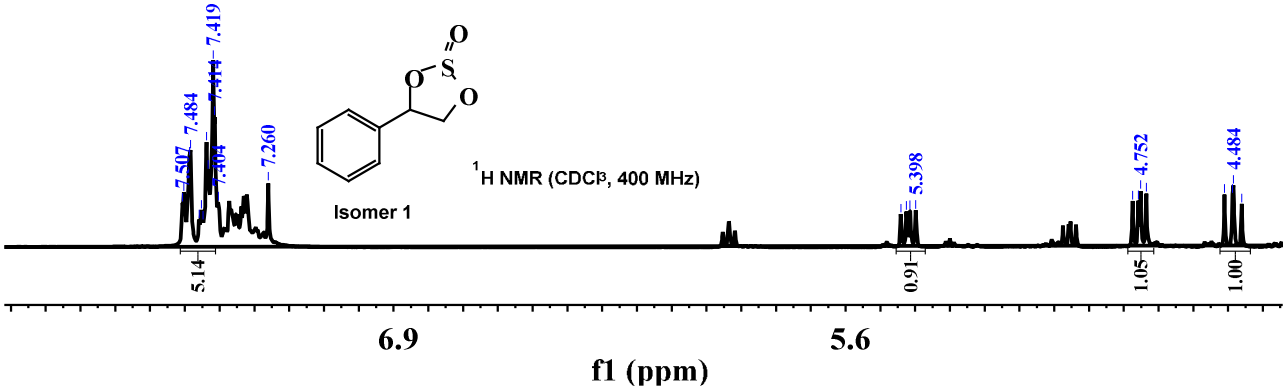
Isomer 2 (RT 9.10 min)

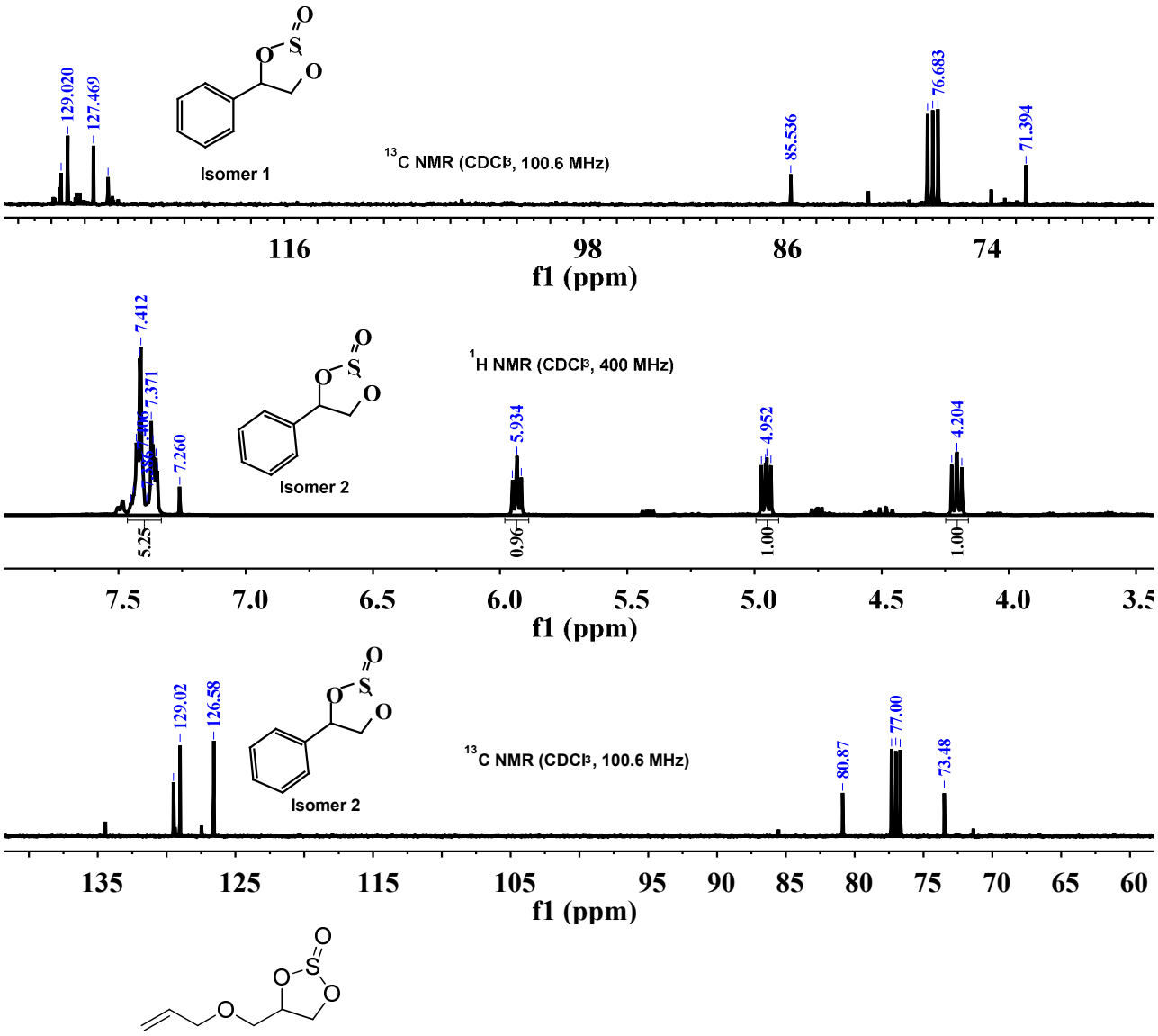
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.35-7.45 (m, 5H), 5.93 (t, ^3J = 6.8 Hz, 1H), 4.94-4.97 (m, 1H), 4.18-4.22 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 129.5, 129.0, 126.6, 80.9, 73.5; GC-MS: m/z (%): 183.93 (<5) [M^+], 153.99 (100) [M^+-OCH_2], 126.10 (44) [M^+-C_3H_6O], 119.17 (11) [M^+-C_5H_5], 105.15 (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]
Relative Abundance
m/z
153.99
105.17
91.19
126.10
78.20
77.20
119.16
89.20
92.17
155.01
183.84
51.12
65.19
39.05
63.16
42.91
53.22
66.28
79.29
103.20
107.16
128.14
141.09
167.11
179.23
184.93
197.17
210.23


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


7.419
7.414
7.484
7.507
7.404
7.260
5.398
4.752
4.484
Isomer 1
1H NMR (CDCl3, 400 MHz)
5.14
0.91
1.05
1.00
6.9
5.6
f1 (ppm)



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

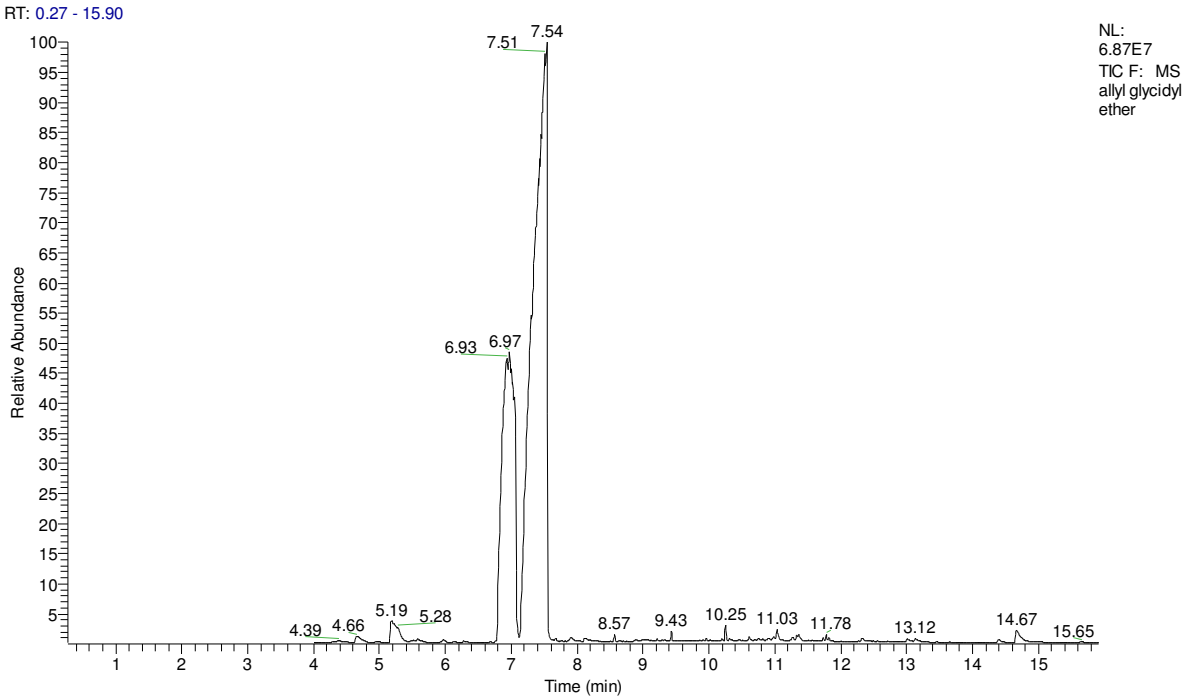
Column chromatography on silica gel: eluting with petroleum ether/ethyl acetae 25:1; Light yellow liquid.

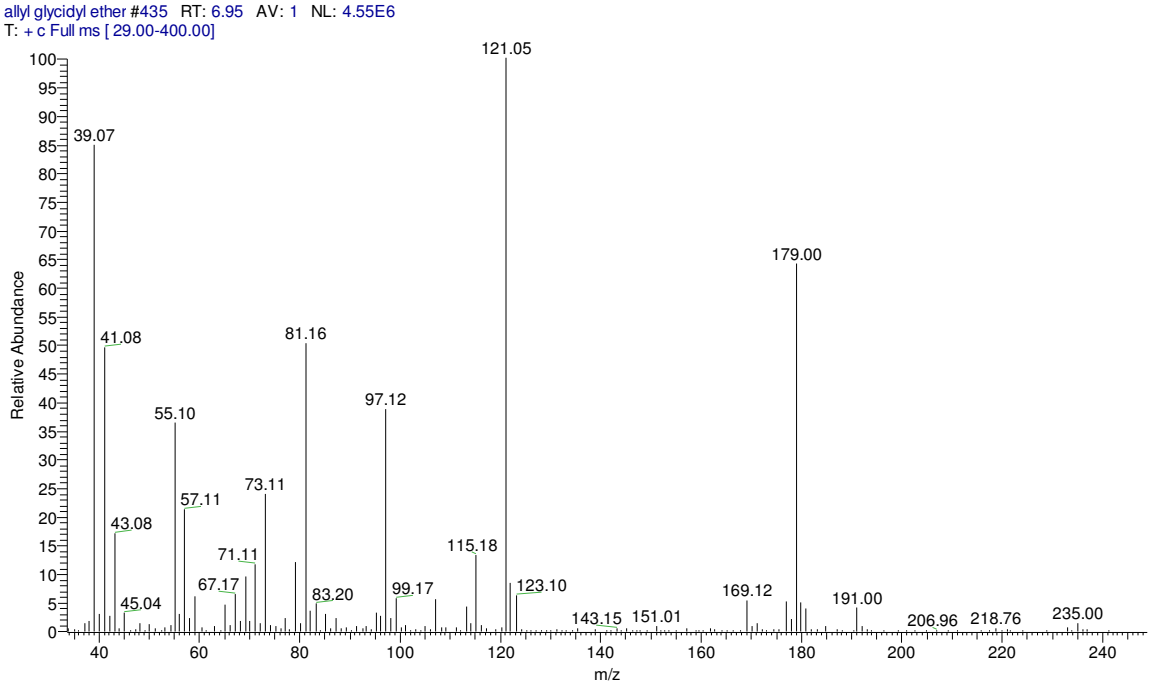
Isomer 1 (RT 6.97 min)

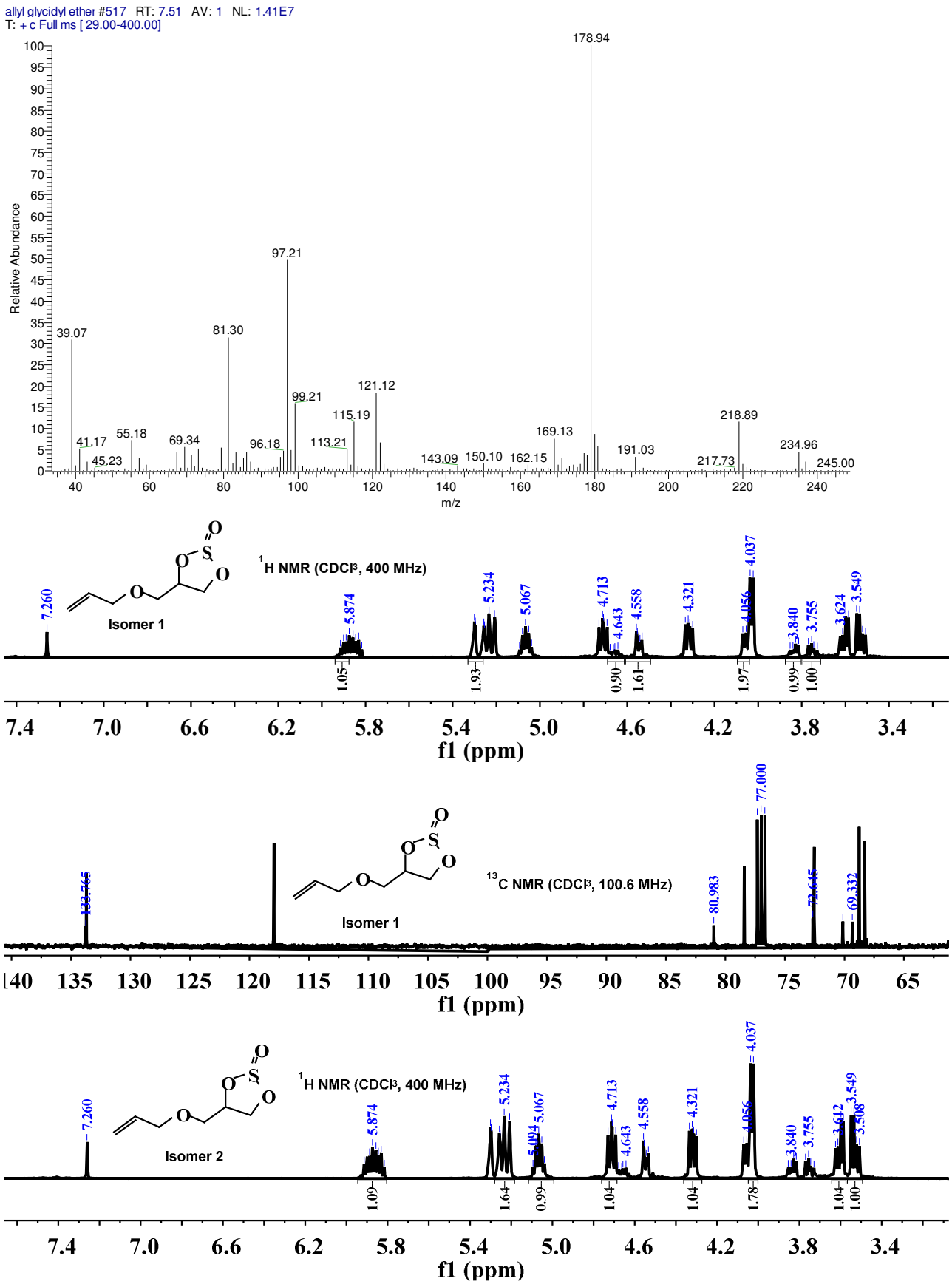
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.82-5.91 (m, 1H), 5.30 (s, 2H), 4.63-4.68 (m, 1H), 4.53-4.56 (m, 2H), 4.06 (s, ^{3}J = 5.6 Hz, 2H), 3.81-3.85 (m, 1H), 3.73-3.77 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 133.8, 81.0, 72.6, 70.1, 69.3; GC-MS: m/z (%): 178.94 (59) [M^+], 121.04 (100) [M^+-C_3H_5O], 97.18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

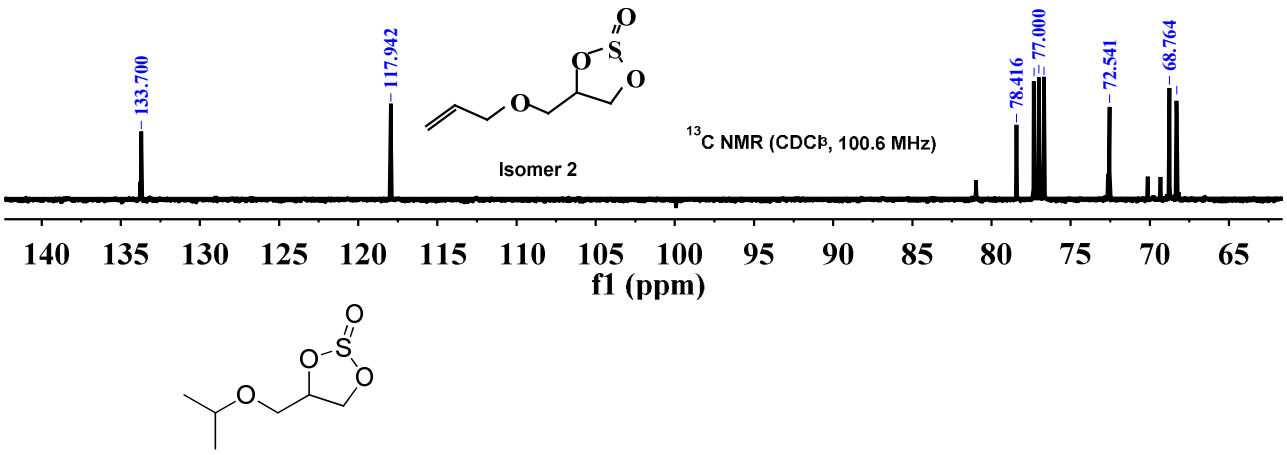
Isomer 2 (RT 7.54 min)

^{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
Relative Abundance
Time (min)
7.54
7.51
6.97
6.93
5.19
5.28
4.66
4.39
8.57
9.43
10.25
11.03
11.78
13.12
14.67
15.65


allyl glycidyl ether #435 RT: 6.95 AV: 1 NL: 4.55E6
T: + c Full ms [29.00-400.00]
Relative Abundance
m/z
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
123.10
99.17
83.20
45.04
169.12
191.00
143.15
151.01
206.96
218.76
235.00


allyl glycidyl ether #517 RT: 7.51 AV: 1 NL: 1.41E7
T: + c Full ms [29.00-400.00]
Relative Abundance
m/z
178.94
97.21
39.07
81.30
99.21
121.12
115.19
218.89
169.13
234.96
191.03
41.17
55.18
69.34
96.18
113.21
143.09
150.10
162.15
217.73
245.00
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)
77.000
80.983
72.646
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

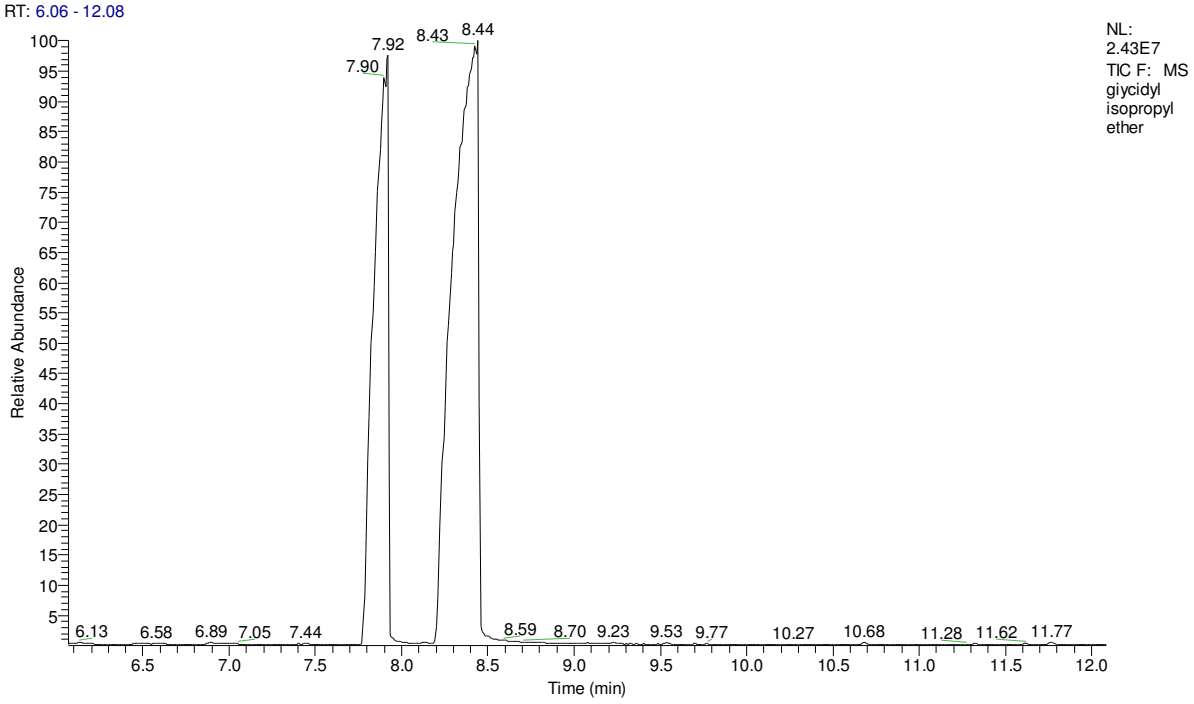
Column chromatography on silica gel: eluting with petroleum ether/ethyl acetae 25:1; Light yellow liquid.

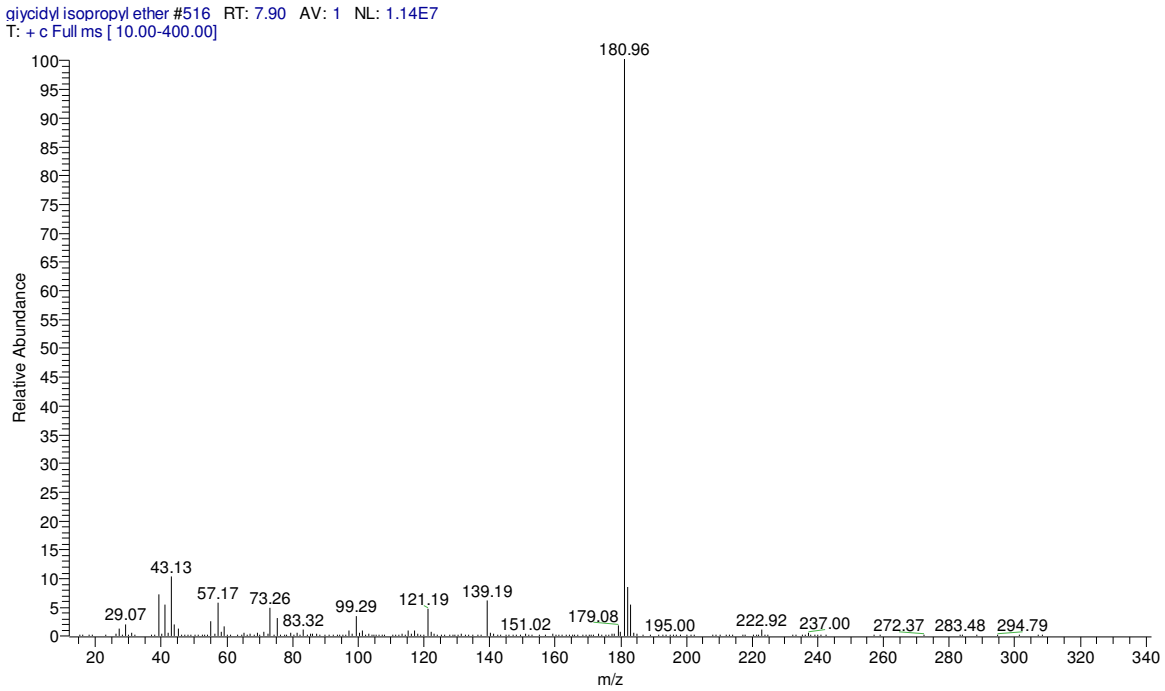
Isomer 1 (RT 7.92 min)

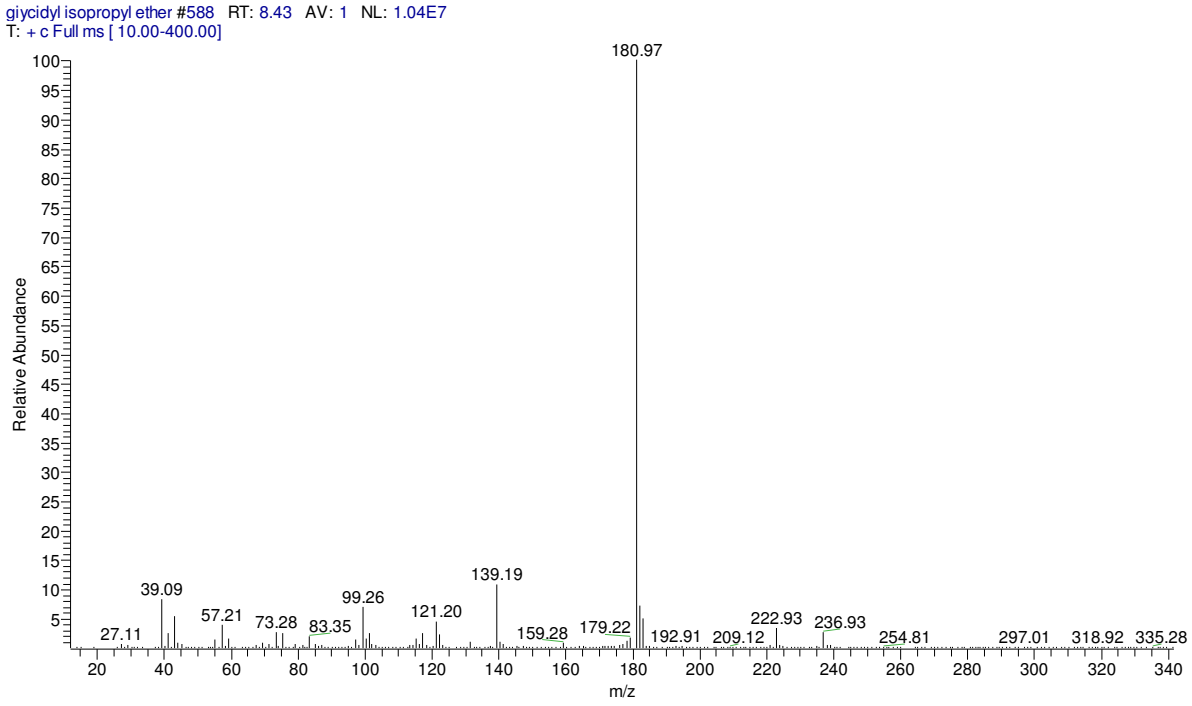
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.79-3.84 (m, 1H), 3.65-3.70 (m, 1H), 3.56-3.63 (m, 2H), 3.45-3.52 (m, 2H), 3.22 (s, 2H), 1.13-1.15 (m, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 21.9, 64.2, 66.5, 68.9, 70.6, 72.4, 78.7; GC-MS: m/z (%): 180.96 (100) [M^+].

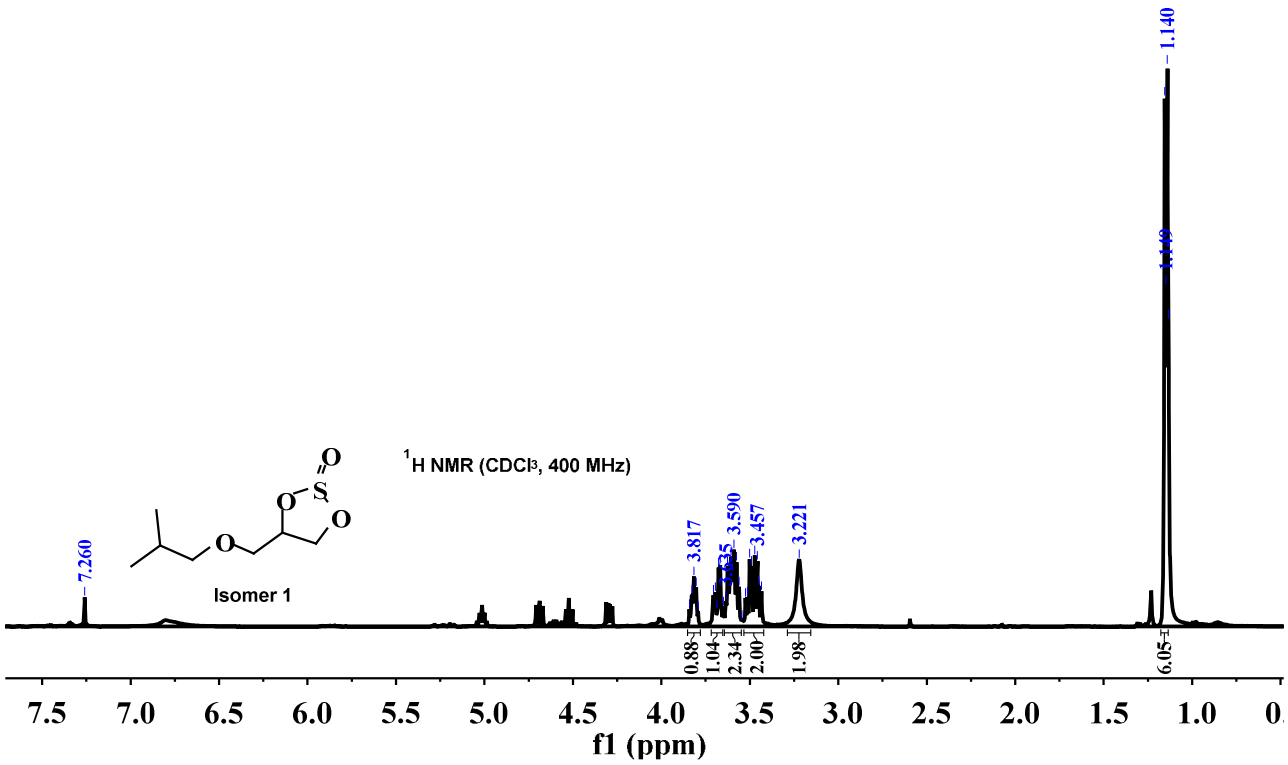
Isomer 2 (RT 8.44 min)

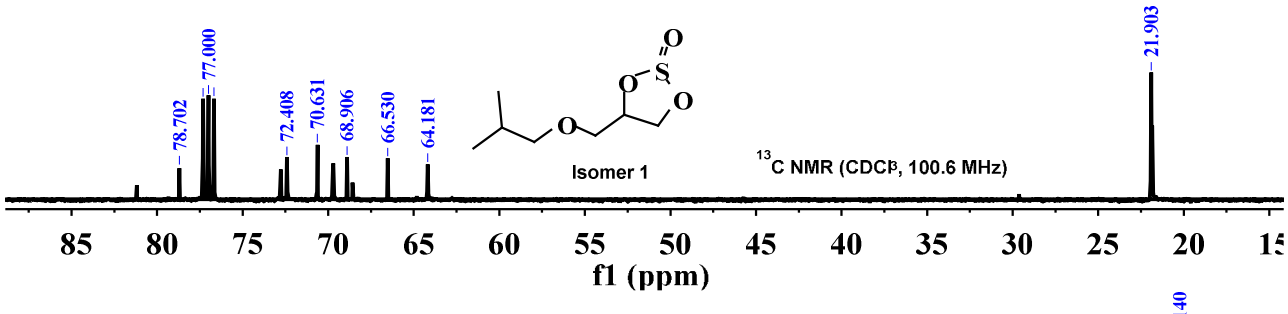
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.99-5.04 (m, 2H), 4.67-4.71 (m, 2H), 4.48-4.56 (m, 2H), 4.28-4.31 (m, 2H), 1.13-1.15 (m, 6H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 81.2, 72.81, 72.78, 69.74, 69.71, 68.6, 21.8; GC-MS: m/z (%): 180.99 (100) [M^+].

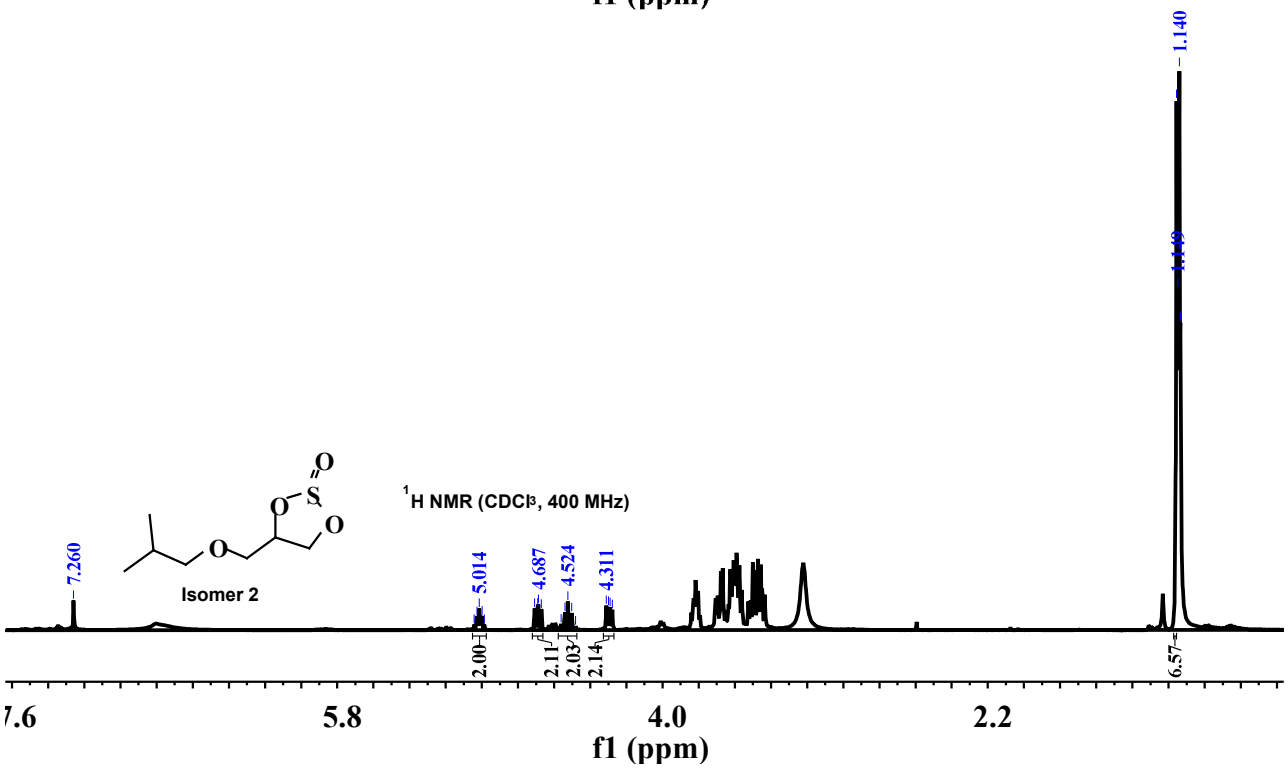


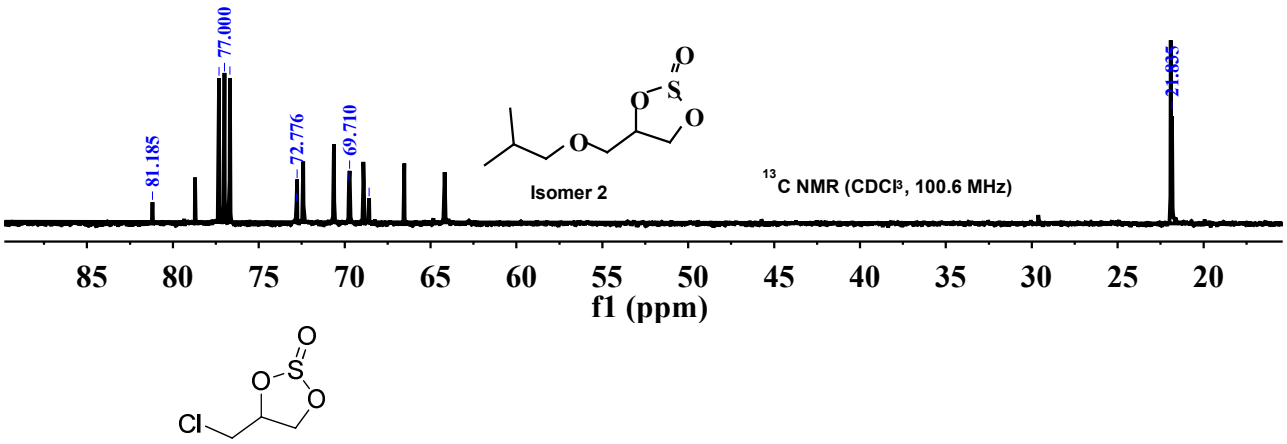

glycidyl isopropyl ether #516 RT: 7.90 AV: 1 NL: 1.14E7
T: + c Full ms [10.00-400.00]
Relative Abundance
m/z
180.96
43.13
57.17
73.26
29.07
83.32
99.29
121.19
139.19
151.02
179.08
195.00
222.92
237.00
272.37
283.48
294.79


glycidyl isopropyl ether #588 RT: 8.43 AV: 1 NL: 1.04E7
T: + c Full ms [10.00-400.00]
Relative Abundance
m/z
180.97
39.09
57.21
73.28
27.11
83.35
99.26
121.20
139.19
159.28
179.22
192.91
209.12
222.93
236.93
254.81
297.01
318.92
335.28


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)
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)


21.903
77.000
78.702
72.408
70.631
68.906
66.530
64.181
Isomer 1
^{13}C NMR (CDCl$_3$, 100.6 MHz)
85 80 75 70 65 60 55 50 45 40 35 30 25 20
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)
5.8 4.0 2.2
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.

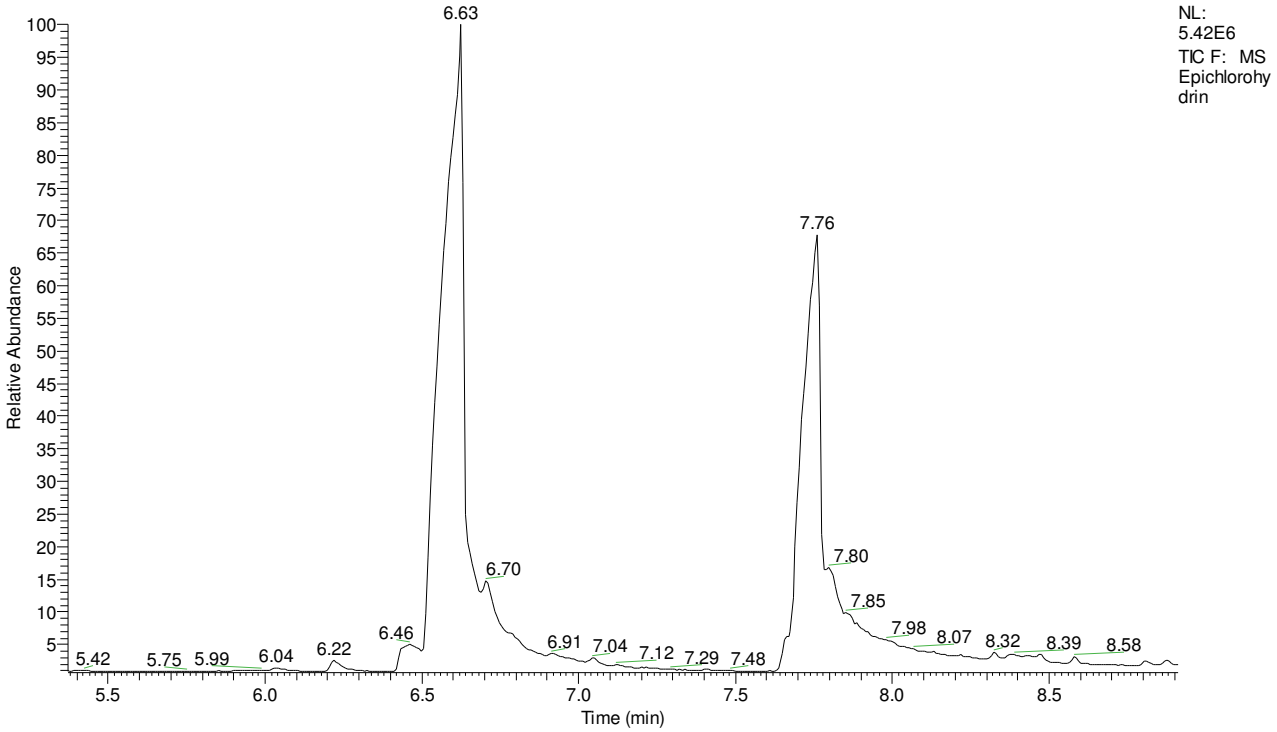
Isomer 1 (minor)

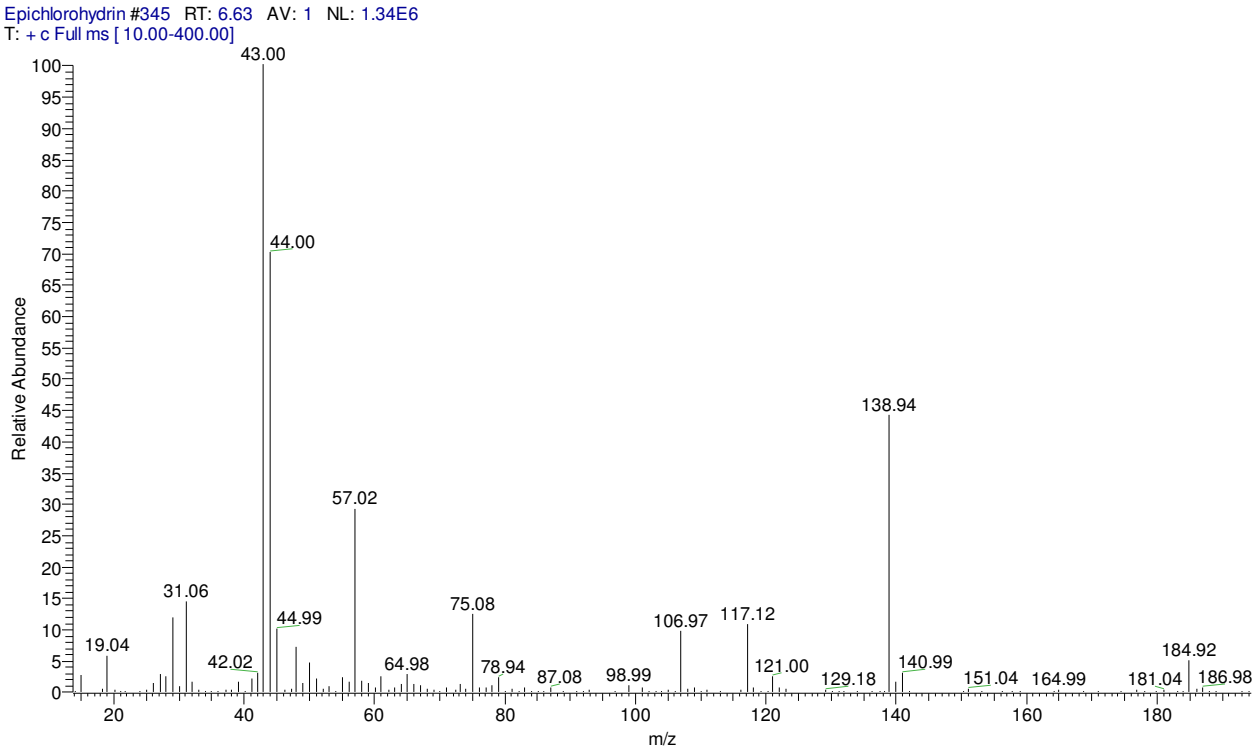
^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.72-4.75 (m, 1H), 4.63-4.65 (m, 2H), 3.88-3.92 (m, 1H), 3.75-3.80 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 80.7, 70.5, 43; GC-MS: m/z (%):139.02 (40) [M^+ - OH], 106.98 (9) [M^+-CH_2Cl], 42.99 (100) [M^+-SO_3Cl].

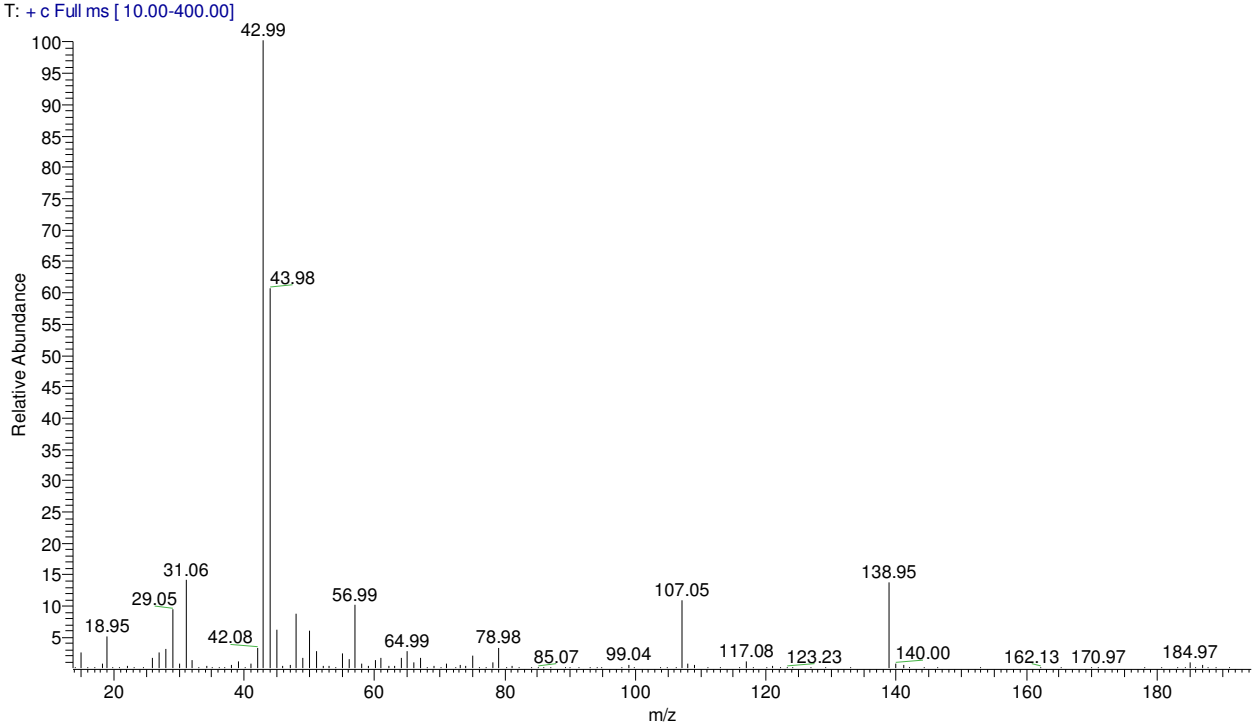
Isomer 2 (major)

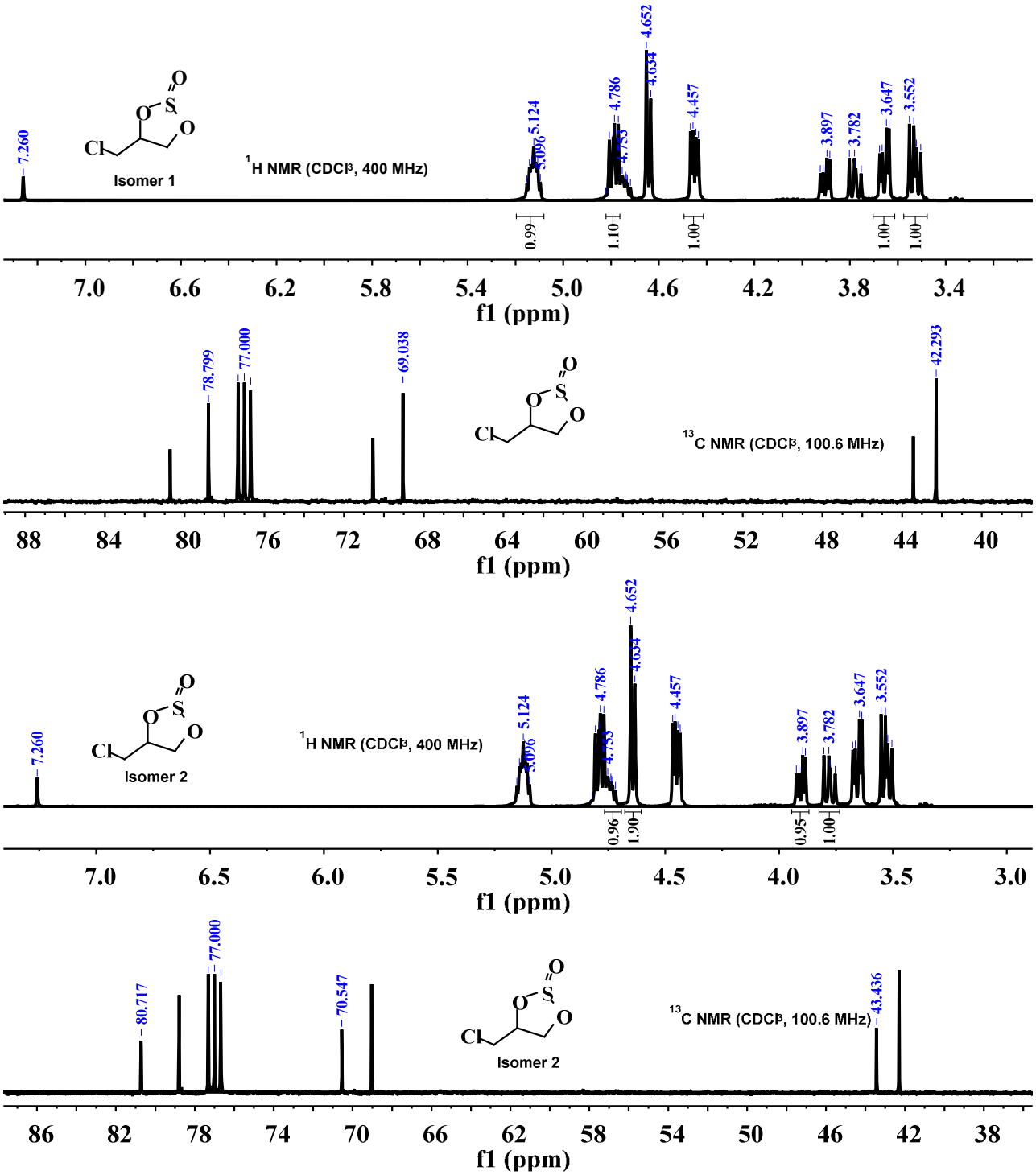
^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.10-5.15 (m, 1H), 4.77-4.82 (m, 1H), 4.43-4.47 (m, 1H), 3.64-3.68 (m, 1H), 3.50-3.55 (m, 1H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 78.8, 69.0, 42.3; GC-MS: m/z (%):138.95 (14) [M^+ - OH], 107.05 (11) [M^+-CH_2Cl], 42.99 (100) [M^+-SO_3Cl].

RT: 5.37 - 8.91




Epichlorohydrin #345 RT: 6.63 AV: 1 NL: 1.34E6
T: + c Full ms [10.00-400.00]
Relative Abundance
m/z
43.00
44.00
138.94
57.02
31.06
75.08
44.99
106.97
117.12
19.04
42.02
64.98
78.94
87.08
98.99
121.00
129.18
140.99
151.04
164.99
181.04
184.92
186.98


Epichlorohydrin #498 RT: 7.76 AV: 1 NL: 1.24E6
T: + c Full ms [10.00-400.00]
Relative Abundance
m/z
42.99
43.98
31.06
138.95
29.05
56.99
107.05
18.95
42.08
64.99
78.98
85.07
99.04
117.08
123.23
140.00
162.13
170.97
184.97



7. Reference

[1] H. Zhi, C. Lu, Q. Zhang, J. Luo, *Chem. Commun.* **2009**, 2878-2880.

[2] G. Cui, C. Wang, J. Zheng, Y. Guo, X. Luo, H. Li, *Chem. Commun.* **2012**, *48*, 2633-2635.

[3] J. E. Bara, *Ind. Eng. Chem. Res.* **2011**, *50*, 13614-13619.

[4] Y. Takenaka, T. Kiyosu, G. Mori, J.-C. Choi, N. Fukaya, T. Sakakura, H. Yasuda, *ChemSusChem* **2012**, *5*, 194-199.