Electronic Supplementary Material (ESI) for Materials Chemistry Frontiers.

Structural Fine-tuning of Zwitterionic Salts for the discovery of LCST-Type Thermoresponsive Materials

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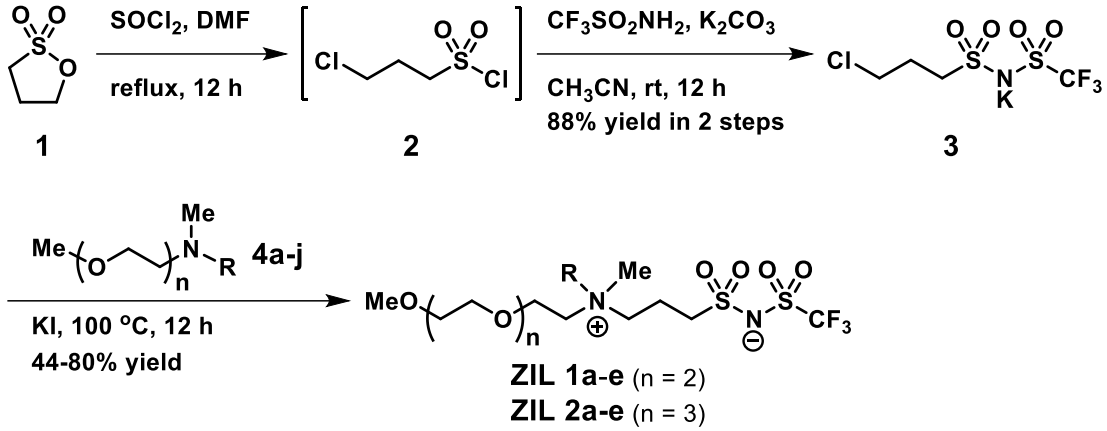
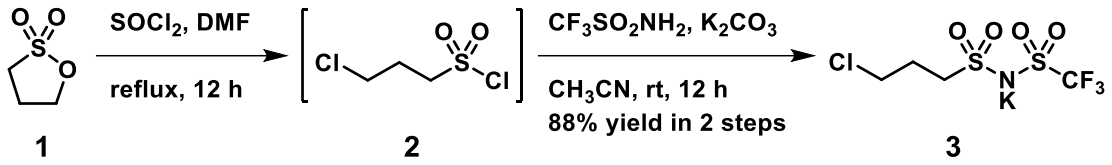
Synthesis of ZIL 1a-1e and ZIL 2a-2e



Synthesis of
potassium *N*-(3-chloropropylsulfonyl) trifluoromethylsulfonamide (3)

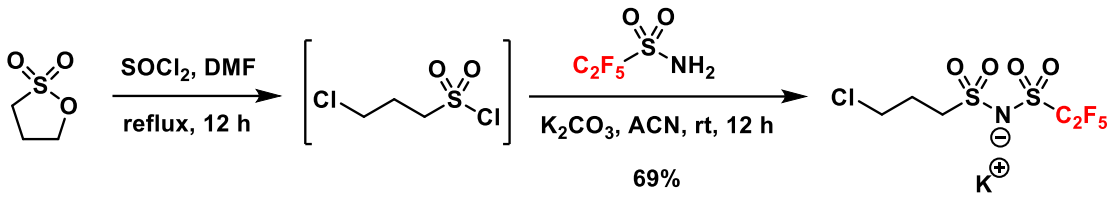


To a round-bottom flask containing 1,3-propanesultone **1** (1.0 g, 8.19 mmol) was added thionyl chloride (1.2 mL, 2 equiv) and DMF (0.063 mL, 0.1 equiv). The mixture was refluxed over 12 h. The excessive thionyl chloride was readily removed *in vacuo* to obtain sulfonyl chloride product as a pale yellow liquid **2**.

The sulfonyl chloride product **2** obtained was dissolved in acetonitrile (5 mL) and added dropwise to the stirring solution containing trifluoromethanesulfonamide (1.2 g, 1 equiv) and K_2CO_3 (1.69 g, 1.5 equiv) dissolved in acetonitrile (35 mL) at room temperature. After 12 h, the solid salt was filtered off. The filtrate was concentrated under reduced pressure to obtain yellow solid crude product. Then, this crude product was continuously washed using a solvent mixture of ethyl acetate and dichloromethane (1/2, v/v) to finally afford the desired product **3** as white solid (1.95 g, 88% yield).

white solid, mp 177 ºC; ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.05-2.16 (m, $ClCH_2CH_2$, 2H), 3.09 (t, J = 6.6 Hz , CH_2CH_2S, 2H), 3.74 (t, J = 7.6 Hz, $ClCH_2CH_2$, 2H); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.60 (CF_3, 3F).

Synthesis of
potassium *N*-(3-chloropropylsulfonyl) pentafluoroethylsulfonamide

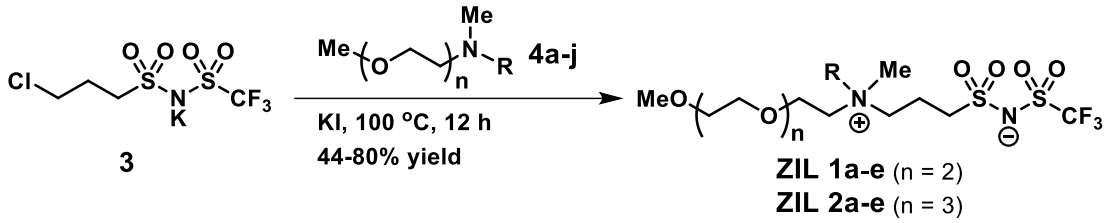


To a round-bottom containing 1,3-propanesultone **1** (1.0 g, 8.19 mmol) was added thionyl chloride (1.2 mL, 2 equiv) and DMF (0.063 mL, 0.1 equiv). The mixture was refluxed over 12 h. The excess thionyl chloride was removed *in vacuo* to obtain sulfonyl chloride product as a pale yellow liquid **2**.

The sulfonyl chloride product **2** obtained was dissolved in acetonitrile (5 mL) and added dropwise to the stirring solution containing pentafluoroethanesulfonamide (1.63 g, 1 equiv) and K_2CO_3 (1.69 g, 1.5 equiv) dissolved in acetonitrile (35 mL) at room temperature. After 12 h, the solid salt was filtered off. The filtrate was concentrated under reduced pressure to obtain yellow oil crude product. Then, the crude product was continuously washed using dichloromethane to eventually afford the desired product as white solid (2.12 g, 69% yield).

white solid, mp 175 ºC; ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.06-2.16 (m, $ClCH_2CH_2$, 2H), 3.09 (t, J = 7.2 Hz , CH_2CH_2S, 2H), 3.75 (t, J = 6.8 Hz, $ClCH_2CH_2$, 2H); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.60 (CF_2CF_3, 3F), -115.75 (CF_2CF_3, 2F).

Synthesis of ZIL 1a-1e and ZIL 2a-2e



To a reaction vial containing potassium *N*-(3-chloropropylsulfonyl) trifluoromethylsulfonamide **3** (100 mg, 0.305 mmol) and potassium iodide (5 mg, 0.1 equiv) was added tertiary amine (2 equiv), which could be readily prepared from reaction of commercial secondary amine with corresponding mesylate of triethylene or teraethylene glycol monomethyl ether.[1-3] The mixture was heated at 100 °C for 12 h. After completion of the reaction, acetonitrile solvent was added to the reaction vial to precipitate potassium chloride produced. The solid salt was filtered off, and its filtrate was concentrated under reduced pressure to obtain yellow liquid crude ZIL product. With the aid of sonication, excessive amine in crude product could be washed away by ether. This crude product was then dissolved in dichloromethane (5 mL), and mixed with ether (200 mL), leading to a white turbid solution. Upon centrifugation, pale yellow ZIL was precipitated. After the collection of the precipitate and ether washing, the residue was purified by silica gel chromatography (dichloromethane/methanol = 15/1, v/v) to afford the desired ZIL product as pale yellow liquid.

References:

1. *J. Med. Chem.* **2014**, *57*, 6183-6196.
2. *J. Am. Chem. Soc.* **2004**, *126*, 1110-1124.
3. *Chem. Eur. J.* **2014**, *20*, 12894-12900.

ZIL 1a pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.05-2.18 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.0-3.11 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.07 (s, 2 × $N^{+}CH_3$, 6H), 3.24 (s, OCH_3, 3H), 3.40-3.49 (m, $N^{+}CH_2CH_2CH_2S$ + $N^{+}CH_2CH_3O$, 4H), 3.49-3.61 (m, 4 × CH_2, 8H), 3.79-3.86 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 17.73, 51.00, 51.20, 62.33, 63.83, 69.42, 69.46, 71.25, 120.07 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.52 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{13}H_{28}F_3N_2O_7S_2$ 445.1285, found 445.1287 ($[M + H]^+$), 467.1106 ($[M + Na]^+$), 483.0846 ($[M + K]^+$).

ZIL 1b pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.23 (t, J = 6.0 Hz, $N^{+}CH_2CH_3$, 3H), 2.00-2.15 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.01 (s, $N^{+}CH_3$, 3H), 3.06 (t, J = 7.2 Hz , $N^{+}CH_2CH_2CH_2S$, 2H), 3.24 (s, OCH_3, 3H), 3.36-3.46 (m, $N^{+}CH_2CH_2CH_2S$ + $N^{+}CH_2CH_3$ + $N^{+}CH_2CH_3O$, 6H), 3.48-3.60 (m, 4 × CH_2, 8H), 3.78-3.84 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 7.53, 17.41, 47.84, 51.14, 57.38, 58.06, 59.01, 59.88, 63.66, 69.46, 69.49, 69.56, 71.27, 120.08 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.53 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{14}H_{30}F_3N_2O_7S_2$ 459.1441, found 459.1445 ($[M + H]^+$), 481.1265 ($[M + Na]^+$), 497.0999 ($[M + K]^+$).

ZIL 1c pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.93 (t, J = 7.2 Hz, $CH_2CH_2CH_3$, 3H), 1.24-1.37 (m, $N^{+}CH_2CH_2CH_2$, 2H), 1.58-1.71 (m, $N^{+}CH_2CH_2CH_2$, 2H), 2.02-2.16 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.03 (s, $N^{+}CH_3$, 3H), 3.06 (t, J = 7.2 Hz , $N^{+}CH_2CH_2CH_2$, 2H), 3.24 (s, OCH_3, 3H), 3.26-3.36 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.39-3.47 (m, $N^{+}CH_2CH_2CH_2S$ + $N^{+}CH_2CH_3O$, 4H), 3.48-3.62 (m, 4 × CH_2, 8H), 3.78-3.86 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 13.49, 17.52, 19.20, 23.43, 48.41, 51.17, 58.11, 57.29, 59.65, 60.45, 61.69, 63.74, 69.64, 71.31, 120.12 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.56 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{16}H_{34}F_3N_2O_7S_2$ 487.1754, found 487.1755 ($[M + H]^+$), 509.1576 ($[M + Na]^+$), 525.1316 ($[M + K]^+$).

ZIL 1d pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.87 (t, J = 6.8 Hz, $CH_2CH_2CH_3$, 3H), 1.20-1.38 (m, 3 × CH_2, 6H), 1.56-1.74 (m, $N^{+}CH_2CH_2CH_2$, 2H), 2.02-2.16 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.03 (s, $N^{+}CH_3$, 3H), 3.05 (t, J = 6.8 Hz , $N^{+}CH_2CH_2CH_2$, 2H), 3.24 (s, OCH_3, 3H), 3.26-3.32 (m, $N^{+}CH_2CH_2CH_2S$, 2H), 3.39-3.47 (m, $N^{+}CH_2CH_2CH_2S$ + $N^{+}CH_2CH_3O$, 4H), 3.49-3.62 (m, 4 × CH_2, 8H), 3.77-3.85 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 13.78, 17.47, 21.36, 21.88, 25.39, 30.65, 48.30, 51.11, 58.04, 59.62, 62.36, 61.83, 63.69, 69.49, 69.57, 71.26, 120.06 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.57 (CF_3, 3F); ESI-

HRMS m/z $[M + H]^+$ calculated for $C_{18}H_{38}F_3N_2O_7S_2$ 515.2067, found 515.2065 ($[M + H]^+$), 537.1882 ($[M + Na]^+$).

ZIL 1e pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.86 (t, J = 6.8 Hz, $CH_2CH_2CH_3$, 3H), 1.17-1.42 (m, 5 × CH_2, 10H), 1.56-1.75 (m, $N^+CH_2CH_2CH_2CH_2$, 2H), 2.00-2.17 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.02 (s, N^+CH_3, 3H), 3.05 (t, J = 7.2 Hz , $N^+CH_2CH_2CH_2$, 2H), 3.24 (s, OCH_3, 3H), 3.26-3.32 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.38-3.47 (m, $N^+CH_2CH_2CH_2S + N^+CH_2CH_3O$, 4H), 3.49-3.66 (m, 4 × CH_2, 8H), 3.76-3.87 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 13.92, 17.46, 21.41, 22.02, 25.75, 28.45, 28.47, 31.15, 48.29, 51.10, 58.04, 59.61, 60.34, 61.82, 63.68, 69.48, 69.56, 71.25, 120.05 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.57 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{20}H_{42}F_3N_2O_7S_2$ 543.2380, found 543.2387 ($[M + H]^+$), 565.2207 ($[M + Na]^+$).

ZIL 2a pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.05-2.17 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.00-3.14 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.08 (s, 2 × N^+CH_3, 6H), 3.24 (s, OCH_3, 3H), 3.40-3.49 (m, $N^+CH_2CH_2CH_2S + N^+CH_2CH_3O$, 4H), 3.49-3.62 (m, 6 × CH_2, 12H), 3.79-3.88 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 17.72, 51.00, 51.20, 58.04, 63.81, 69.43, 69.54, 69.70, 69.76, 71.27, 120.06 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.52 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{15}H_{32}F_3N_2O_8S_2$ 489.1547, found 489.1538 ($[M + H]^+$), 511.1357 ($[M + Na]^+$).

ZIL 2b pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.23 (t, J = 7.2 Hz, $N^+CH_2CH_3$, 3H), 2.00-2.15 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.01 (s, N^+CH_3, 3H), 3.06 (t, J = 7.2 Hz , $N^+CH_2CH_2CH_2S$, 2H), 3.24 (s, OCH_3, 3H), 3.36-3.46 (m, $N^+CH_2CH_2CH_2S + N^+CH_2CH_3 + N^+CH_2CH_3O$, 6H), 3.47-3.62 (m, 6 × CH_2, 12H), 3.78-3.86 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 7.52, 17.40, 47.86, 51.13, 57.34, 58.05, 58.99, 59.85, 63.63, 69.46, 69.56, 69.72, 69.79, 71.29, 120.07 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.53 (CF_3, 3F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{16}H_{34}F_3N_2O_8S_2$ 503.1703, found 503.1694 ($[M + H]^+$), 525.1513 ($[M + Na]^+$).

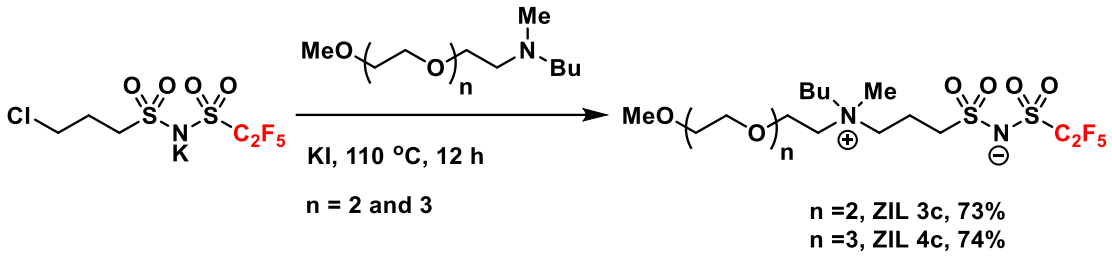
ZIL 2c pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.93 (t, J = 7.2 Hz, $CH_2CH_2CH_3$, 3H), 1.22-1.36 (m, $N^+CH_2CH_2CH_2$, 2H), 1.57-1.71 (m, $N^+CH_2CH_2CH_2$, 2H), 2.02-2.16 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.03 (s, N^+CH_3, 3H), 3.06 (t, J = 6.8 Hz , $N^+CH_2CH_2CH_2$, 2H), 3.24 (s, OCH_3, 3H), 3.26-3.36 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.38-3.46 (m, $N^+CH_2CH_2CH_2S + N^+CH_2CH_3O$, 4H), 3.48-3.62 (m, 6 × CH_2, 12H), 3.78-3.87

(m, C*H*$_2$OCH$_3$, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 13.43, 17.47, 19.15, 23.37, 48.37, 51.11, 58.04, 59.59, 60.38, 61.59, 63.67, 69.49, 69.57, 69.73, 69.78, 71.29, 120.06 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-*d*$_6$) δ -76.56 (CF$_3$, 3F); ESI-HRMS m/z [M + H]$^+$ calculated for $C_{18}H_{38}F_3N_2O_8S_2$ 531.2016, found 531.2011 ([M + H]$^+$), 553.1829 ([M + Na]$^+$).

ZIL 2d pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 0.87 (t, *J* = 6.8 Hz, CH$_2$CH$_2$C*H*$_3$, 3H), 1.20-1.36 (m, 3 × CH$_2$, 6H), 1.56-1.72 (m, N$^+$CH$_2$C*H*$_2$CH$_2$, 2H), 2.00-2.16 (m, N$^+$CH$_2$C*H*$_2$CH$_2$S, 2H), 3.03 (s, N$^+$CH$_3$, 3H), 3.05 (t, *J* = 7.2 Hz , N$^+$C*H*$_2$CH$_2$CH$_2$, 2H), 3.24 (s, OCH$_3$, 3H), 3.26-3.32 (m, N$^+$C*H*$_2$CH$_2$CH$_2$S, 2H), 3.38-3.46 (m, N$^+$C*H*$_2$CH$_2$CH$_2$S + N$^+$C*H*$_2$CH$_3$O, 4H), 3.48-3.62 (m, 6 × CH$_2$, 12H), 3.77-3.86 (m, C*H*$_2$OCH$_3$, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 13.77, 17.45, 21.34, 21.86, 25.38, 30.63, 30.89, 48.32, 51.10, 58.03, 59.59, 60.33, 61.79, 63.65, 69.47, 69.55, 69.71, 69.76, 71.27, 120.05 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-*d*$_6$) δ -76.56 (CF$_3$, 3F); ESI-HRMS m/z [M + H]$^+$ calculated for $C_{20}H_{42}F_3N_2O_8S_2$ 559.2329, found 559.2321 ([M + H]$^+$), 581.2142 ([M + Na]$^+$).

ZIL 2e pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 0.86 (t, *J* = 7.2 Hz, CH$_2$CH$_2$C*H*$_3$, 3H), 1.18-1.39 (m, 5 × CH$_2$, 10H), 1.58-1.72 (m, N$^+$CH$_2$C*H*$_2$CH$_2$, 2H), 2.00-2.16 (m, N$^+$CH$_2$C*H*$_2$CH$_2$S, 2H), 3.02 (s, N$^+$CH$_3$, 3H), 3.05 (t, *J* = 7.2 Hz , N$^+$C*H*$_2$CH$_2$CH$_2$, 2H), 3.24 (s, OCH$_3$, 3H), 3.26-3.32 (m, N$^+$C*H*$_2$CH$_2$CH$_2$S, 2H), 3.39-3.47 (m, N$^+$C*H*$_2$CH$_2$CH$_2$S + N$^+$C*H*$_2$CH$_3$O, 4H), 3.47-3.64 (m, 6 × CH$_2$, 12H), 3.78-3.86 (m, C*H*$_2$OCH$_3$, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 13.92, 17.46, 21.40, 22.02, 25.76, 28.46, 31.15, 48.32, 51.10, 58.04, 59.60, 60.33, 61.80, 63.66, 69.48, 69.60, 69.72, 69.77, 71.27, 120.05 (q, J_{CF} = 322 Hz); ^{19}F NMR (376 MHz, DMSO-*d*$_6$) δ -76.57 (CF$_3$, 3F); ESI-HRMS m/z [M + H]$^+$ calculated for $C_{22}H_{46}F_3N_2O_8S_2$ 587.2642, found 587.2636 ([M + H]$^+$), 609.2456 ([M + Na]$^+$).

Synthesis of ZIL 3c and 4c

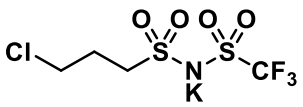


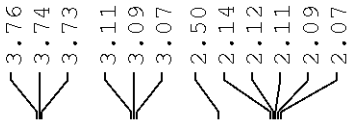
To a reaction vial containing potassium *N*-(3-chloropropylsulfonyl) pentafluoroethylsulfonamide (150 mg, 0.397 mmol) and potassium iodide (6.6 mg, 0.1 equiv) was added tertiary amine (2 equiv). The mixture was heated at 100 ºC for 12 h. After completion of reaction, acetonitrile was added to the vial for precipitation of potassium chloride. The solid salt was filtered off, and its filtrate was concentrated under reduced pressure to obtain yellow liquid crude ZIL product. With the aid of sonication, excessive amine in crude product could be washed away by ether. This crude product was then dissolved in dichloromethane (5 mL), and mixed with ether (200 mL), leading to a white turbid solution. Upon centrifugation, pale yellow ZIL was precipitated. After the collection of the precipitate and ether washing, the residue was purified by silica gel chromatography (dichloromethane/methanol = 15/1, v/v) to afford the desired ZIL product as pale yellow liquid.

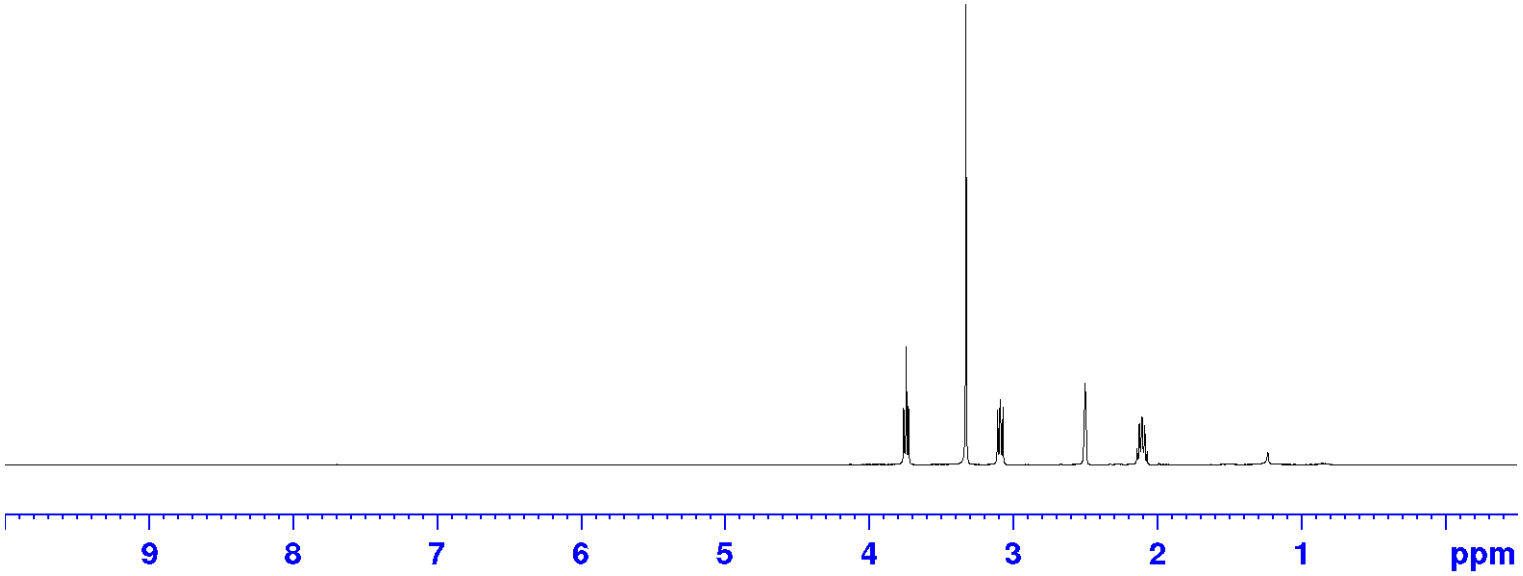
ZIL 3c pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.93 (t, J = 7.2 Hz, $CH_2CH_2CH_3$, 3H), 1.24-1.37 (m, $N^+CH_2CH_2CH_2$, 2H), 1.58-1.71 (m, $N^+CH_2CH_2CH_2$, 2H), 2.02-2.16 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.03 (s, N^+CH_3, 3H), 3.06 (t, J = 6.8 Hz , $N^+CH_2CH_2CH_2$, 2H), 3.28 (s, OCH_3, 3H), 3.26-3.36 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.38-3.47 (m, $N^+CH_2CH_2CH_2S$ + $N^+CH_2CH_3O$, 4H), 3.49-3.62 (m, 4 × CH_2, 8H), 3.77-3.86 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 13.39, 17.46, 19.13, 23.37, 48.33, 51.31, 58.02, 59.60, 60.39, 61.61, 63.68, 69.48, 69.57, 71.25, 111.53 (qt, ^{1}J = 291 Hz, ^{2}J = 37 Hz, CF_2CF_3), 118.14 (tq, ^{1}J = 286 Hz, ^{2}J = 34 Hz, CF_2CF_3); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.60 (CF_2CF_3, 3F), -115.75 (CF_2CF_3, 2F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{17}H_{34}F_5N_2O_7S_2$ 537.1722, found 537.1714 ($[M + H]^+$), 559.1533 ($[M + Na]^+$).

ZIL 4c pale yellow liquid; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.93 (t, J = 7.2 Hz, $CH_2CH_2CH_3$, 3H), 1.21-1.37 (m, $N^+CH_2CH_2CH_2$, 2H), 1.56-1.72 (m, $N^+CH_2CH_2CH_2$, 2H), 2.02-2.17 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.03 (s, N^+CH_3, 3H), 3.06 (t, J = 7.2 Hz , $N^+CH_2CH_2CH_2$, 2H), 3.24 (s, OCH_3, 3H), 3.26-3.36 (m, $N^+CH_2CH_2CH_2S$, 2H), 3.39-3.47 (m, $N^+CH_2CH_2CH_2S$ + $N^+CH_2CH_3O$, 4H), 3.47-3.63 (m, 6 × CH_2, 12H), 3.77-3.86 (m, CH_2OCH_3, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 13.38, 17.45, 19.13, 23.37, 48.35, 51.31, 58.01, 59.59, 60.38, 61.59, 63.66, 69.48, 69.56, 69.72, 69.77, 71.27, 111.52 (qt, ^{1}J = 292 Hz, ^{2}J = 37 Hz, CF_2CF_3), 118.14 (tq, ^{1}J = 286 Hz, ^{2}J = 34 Hz, CF_2CF_3); ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.60 (CF_2CF_3, 3F), -115.74 (CF_2CF_3, 2F); ESI-HRMS m/z $[M + H]^+$ calculated for $C_{19}H_{38}F5N_2O_8S_2$ 581.1984, found 581.1976 ($[M + H]^+$), 603.1795 ($[M + Na]^+$).

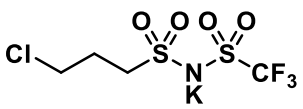
^{1}H NMR spectrum of potassium ((3-chloropropyl)sulfonyl)((trifluoromethyl)sulfonyl)amide

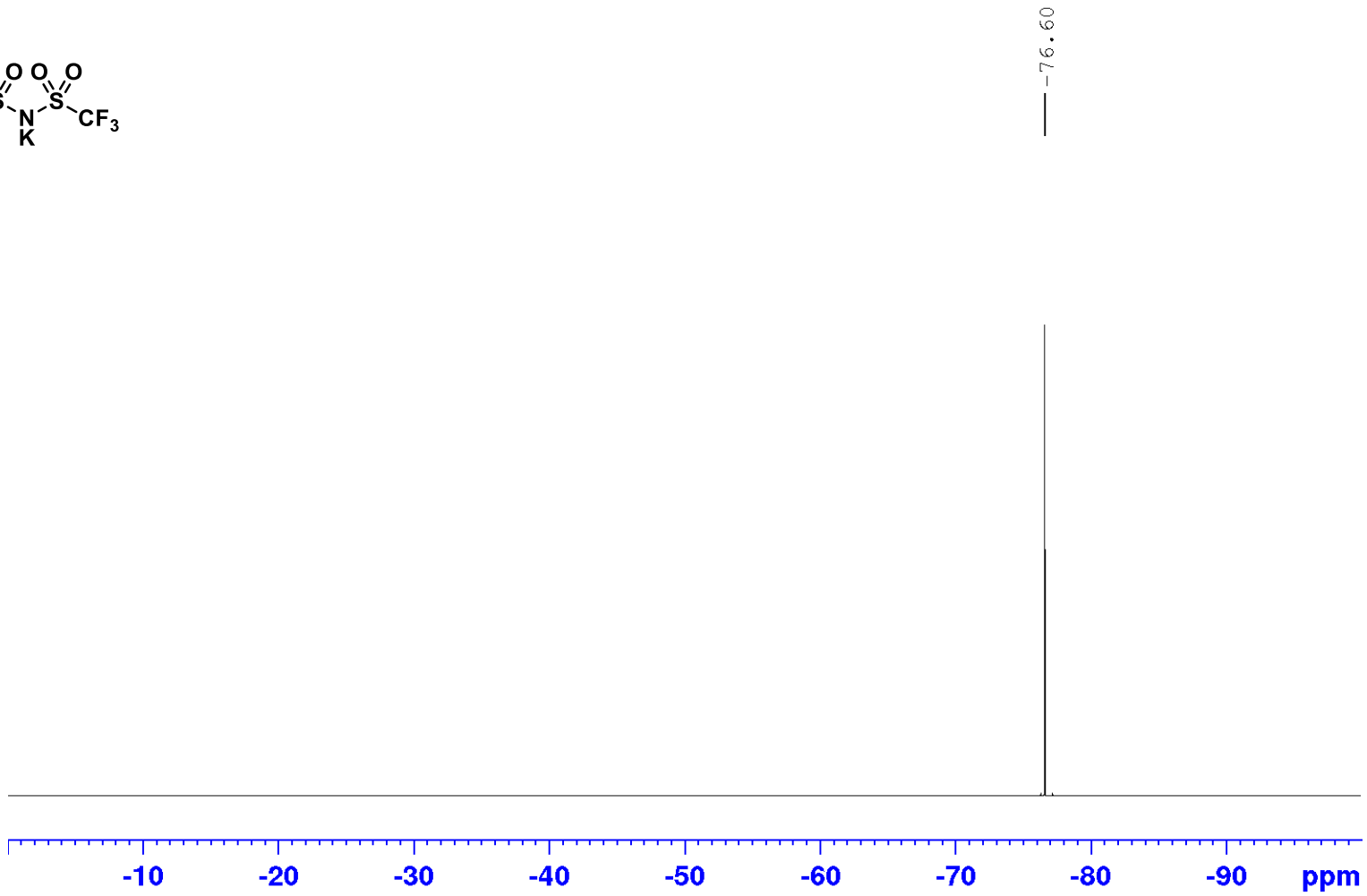




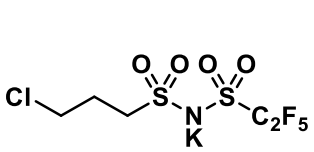


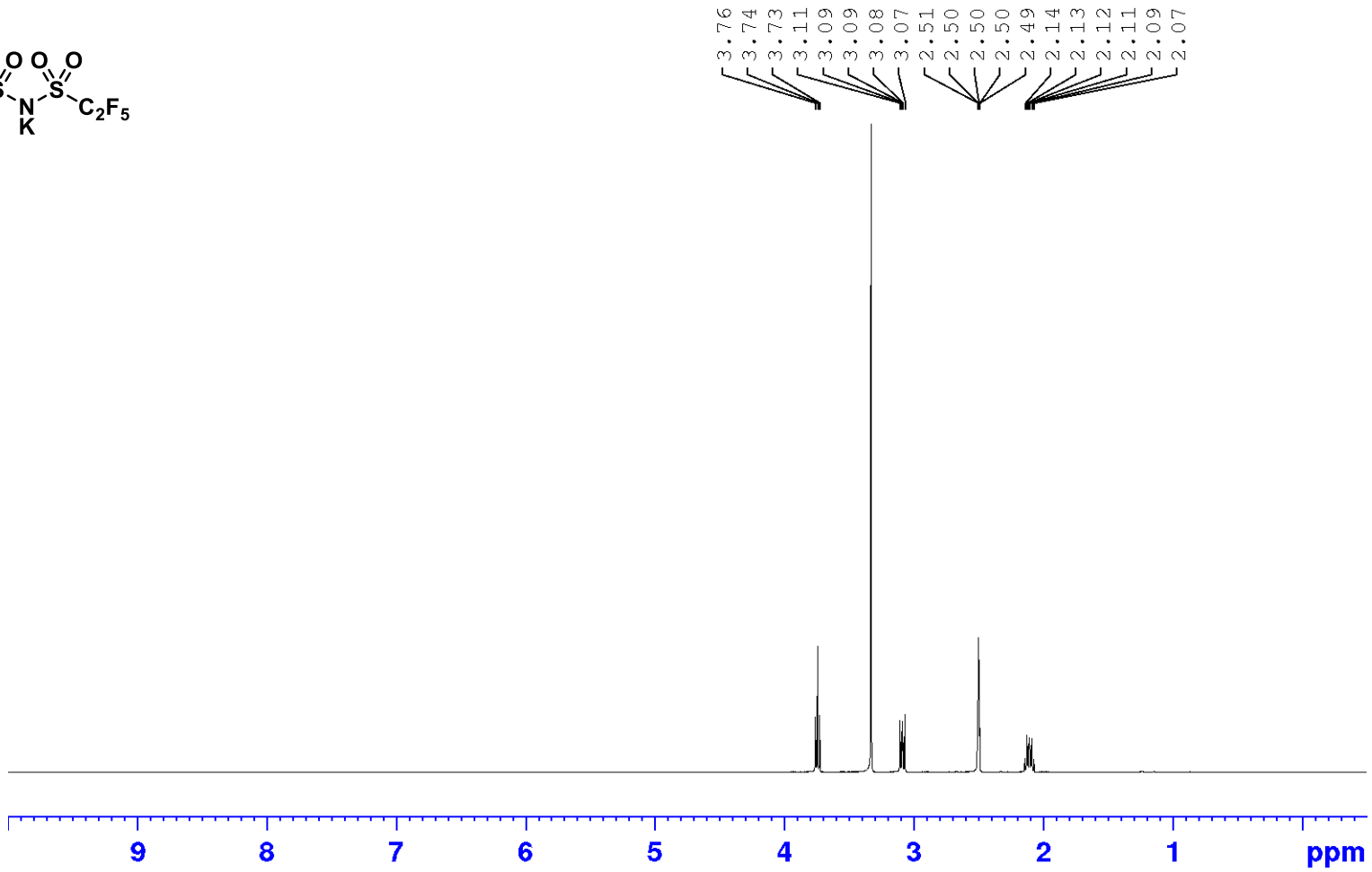
^{19}F NMR spectrum of potassium ((3-chloropropyl)sulfonyl)((trifluoromethyl)sulfonyl)amide



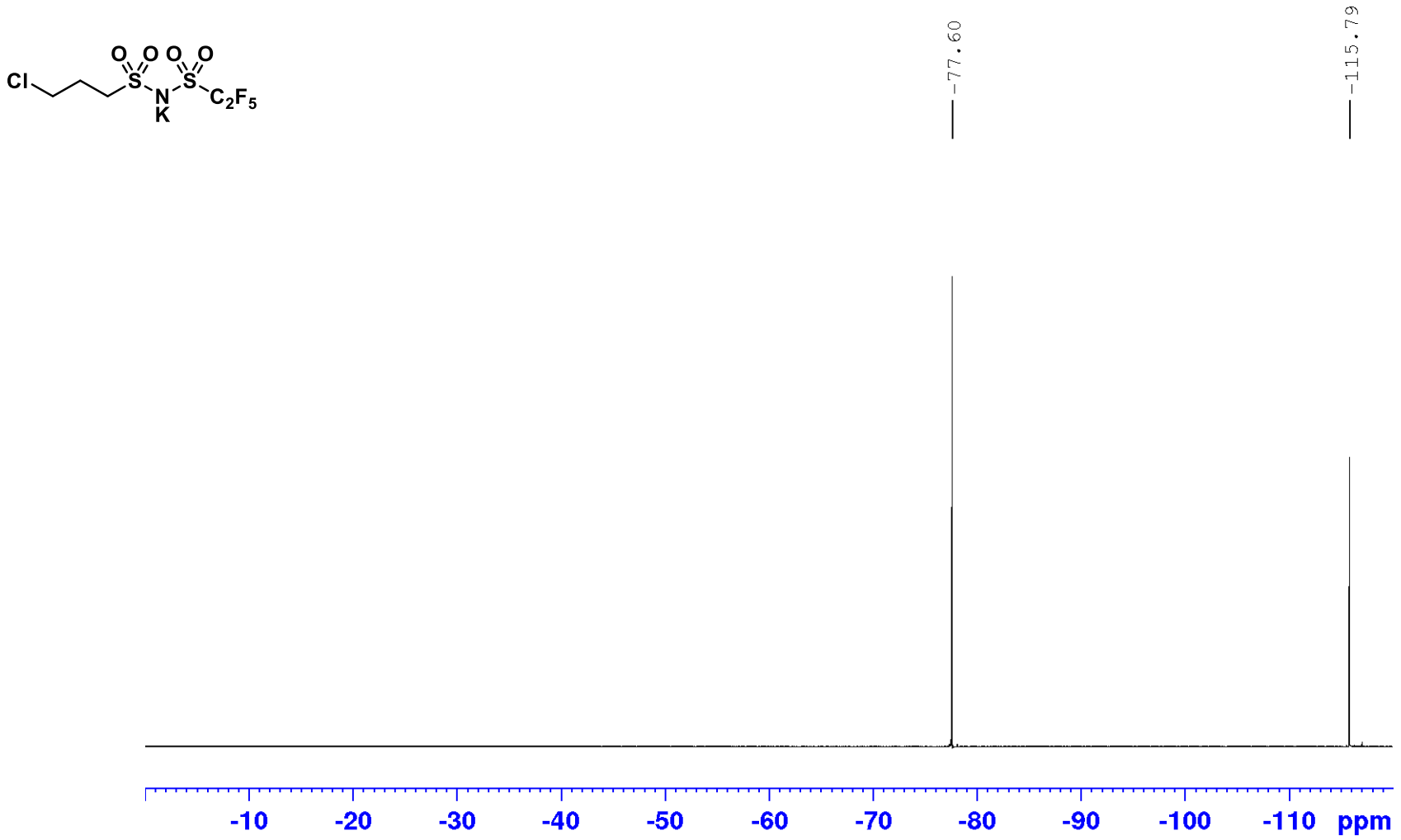


^{1}H NMR spectrum of potassium ((3-chloropropyl)sulfonyl)((perfluoroethyl)sulfonyl)amide

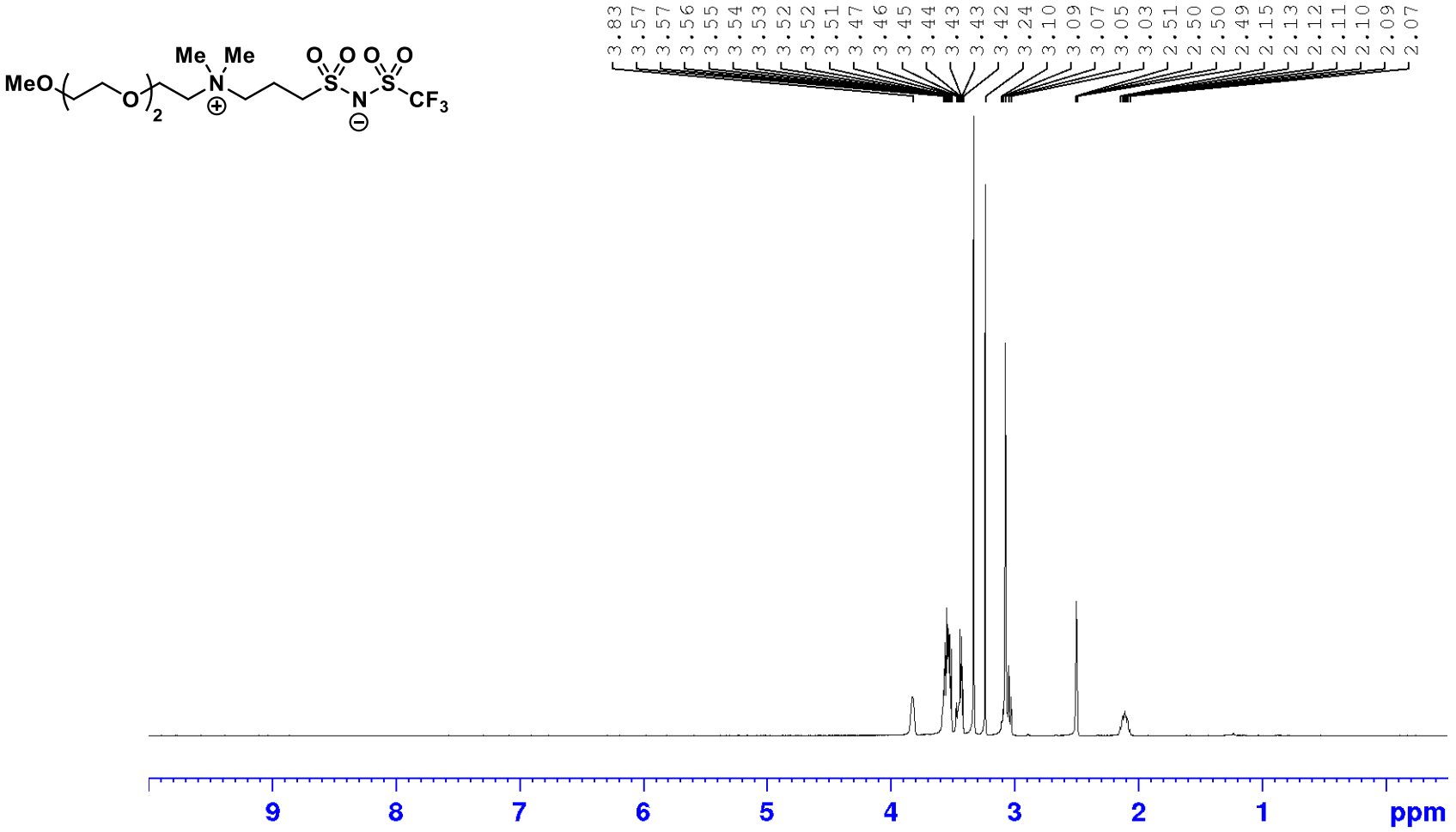




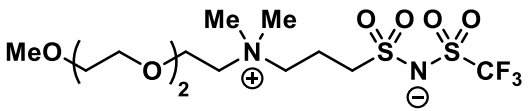
^{19}F NMR spectrum of potassium ((3-chloropropyl)sulfonyl)((trifluoromethyl)sulfonyl)amide



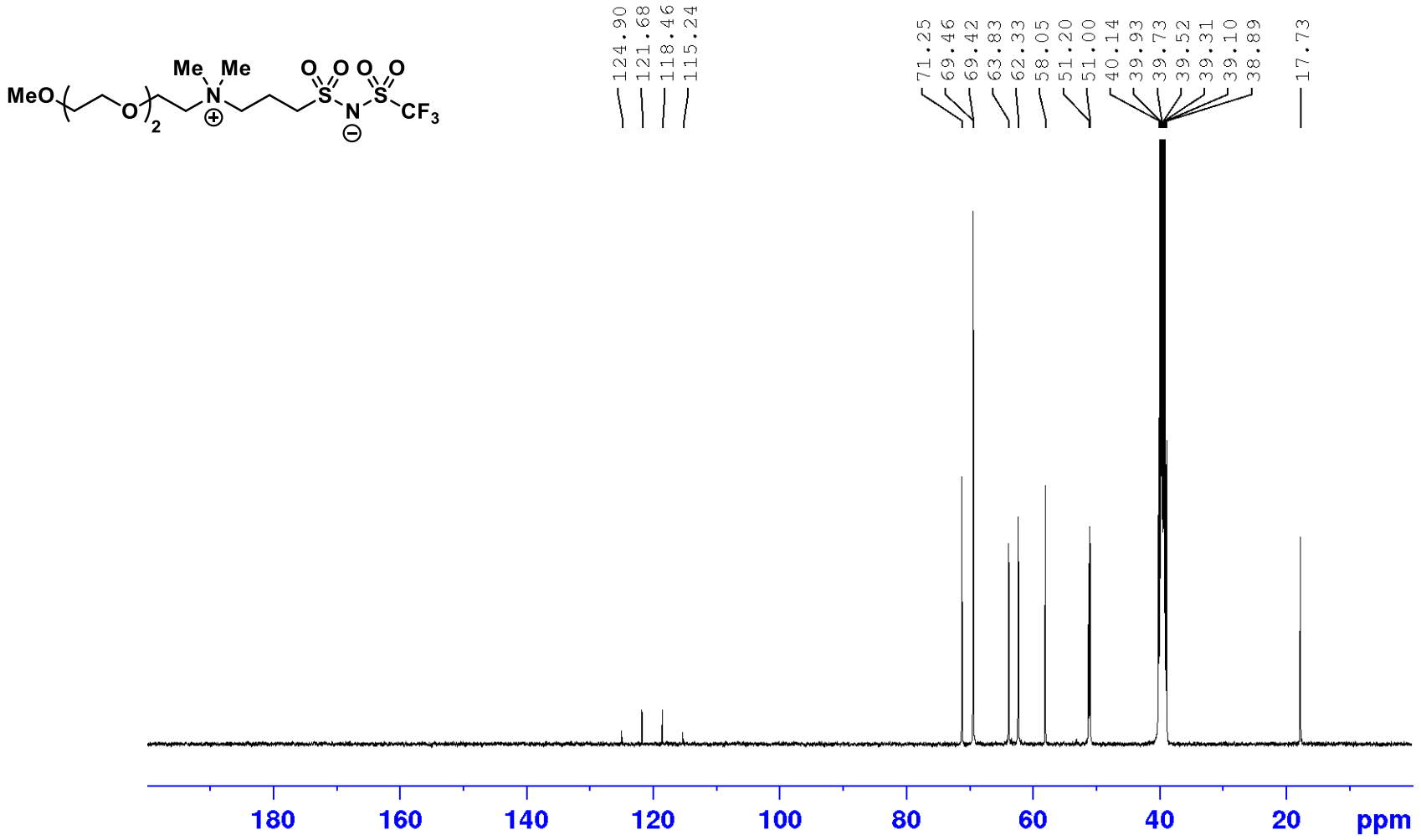
^{1}H NMR spectrum of ZIL 1a



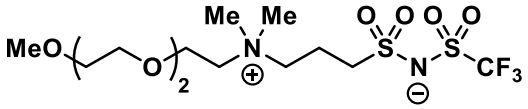
^{19}F NMR spectrum of ZIL 1a



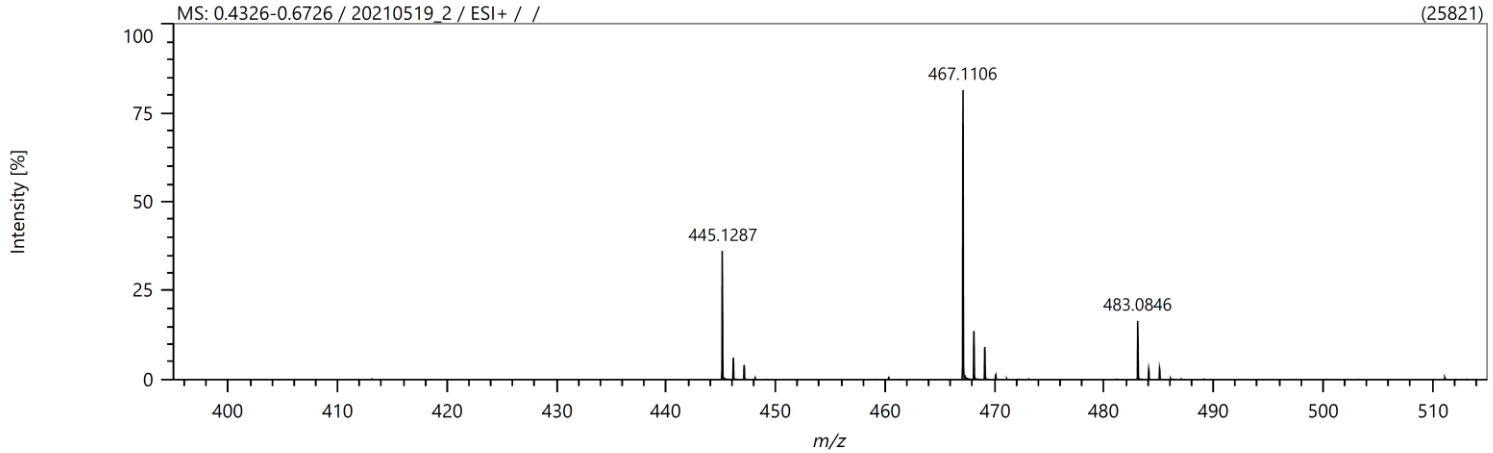
^{13}C NMR spectrum of ZIL 1a



Mass spectrum of ZIL 1a



Spectrum



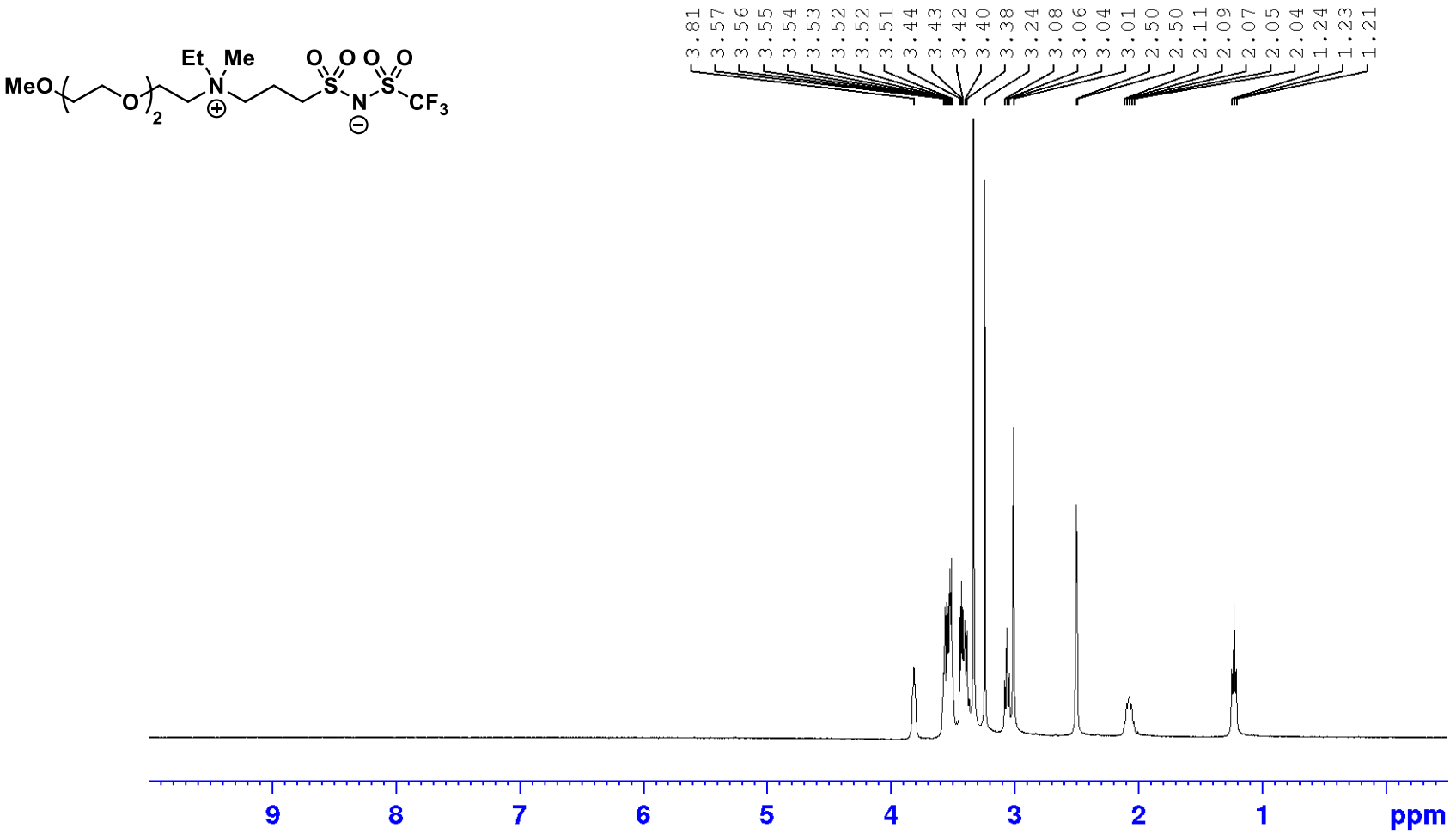
Elemental Composition

Parameters		Elements Set 1:								
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	3	2	7	2	0	0
Charge:	+1	Max	400	1000	3	2	7	2	1	1
DBE:	-99.0 - 999.0									

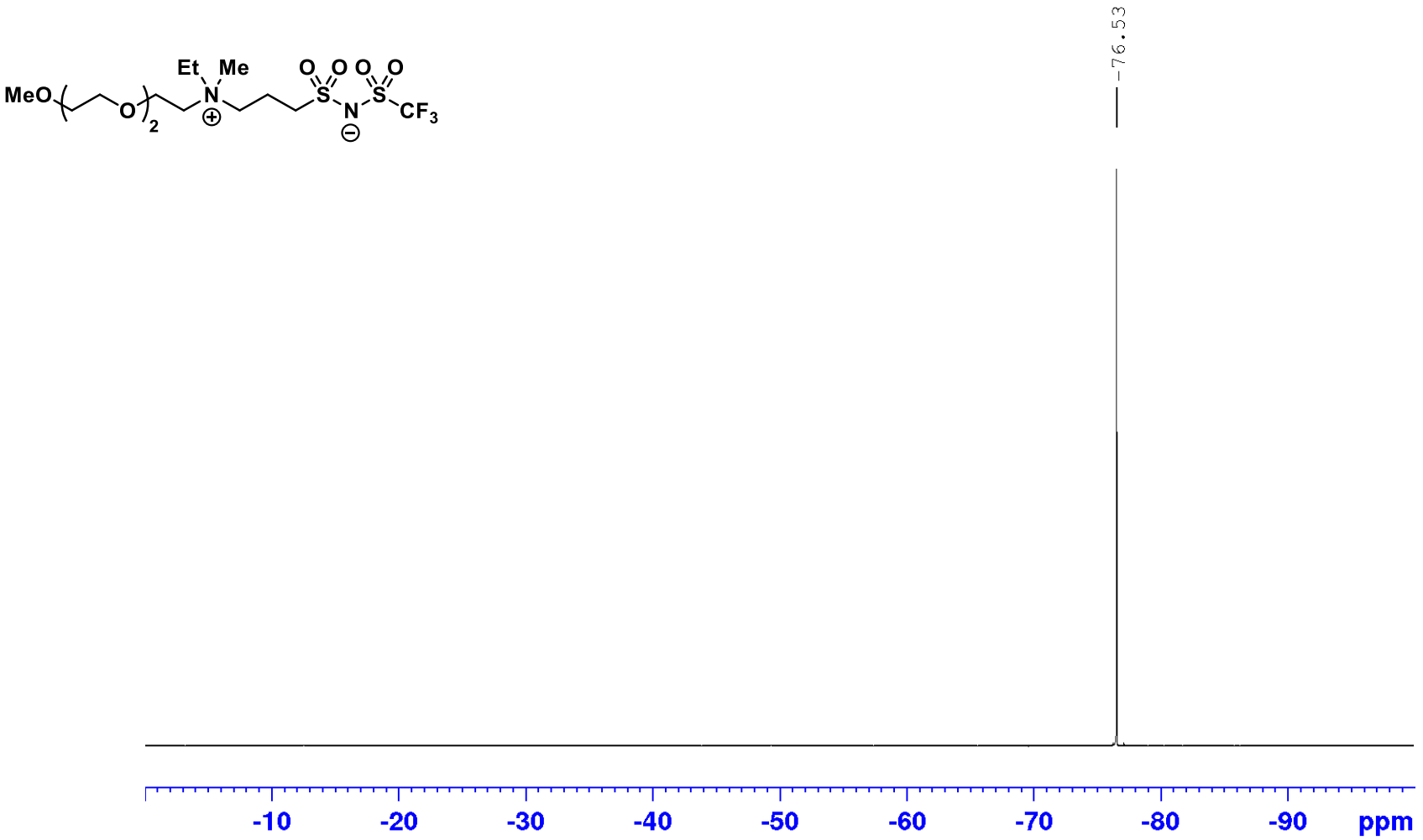
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
445.12868	C13 H28 N2 O7 F3 S2	445.12845	0.23	0.51	-0.5
467.11059	C13 H27 N2 O7 F3 Na S2	467.11040	0.19	0.41	-0.5
483.08460	C13 H27 N2 O7 F3 S2 K	483.08434	0.26	0.54	-0.5

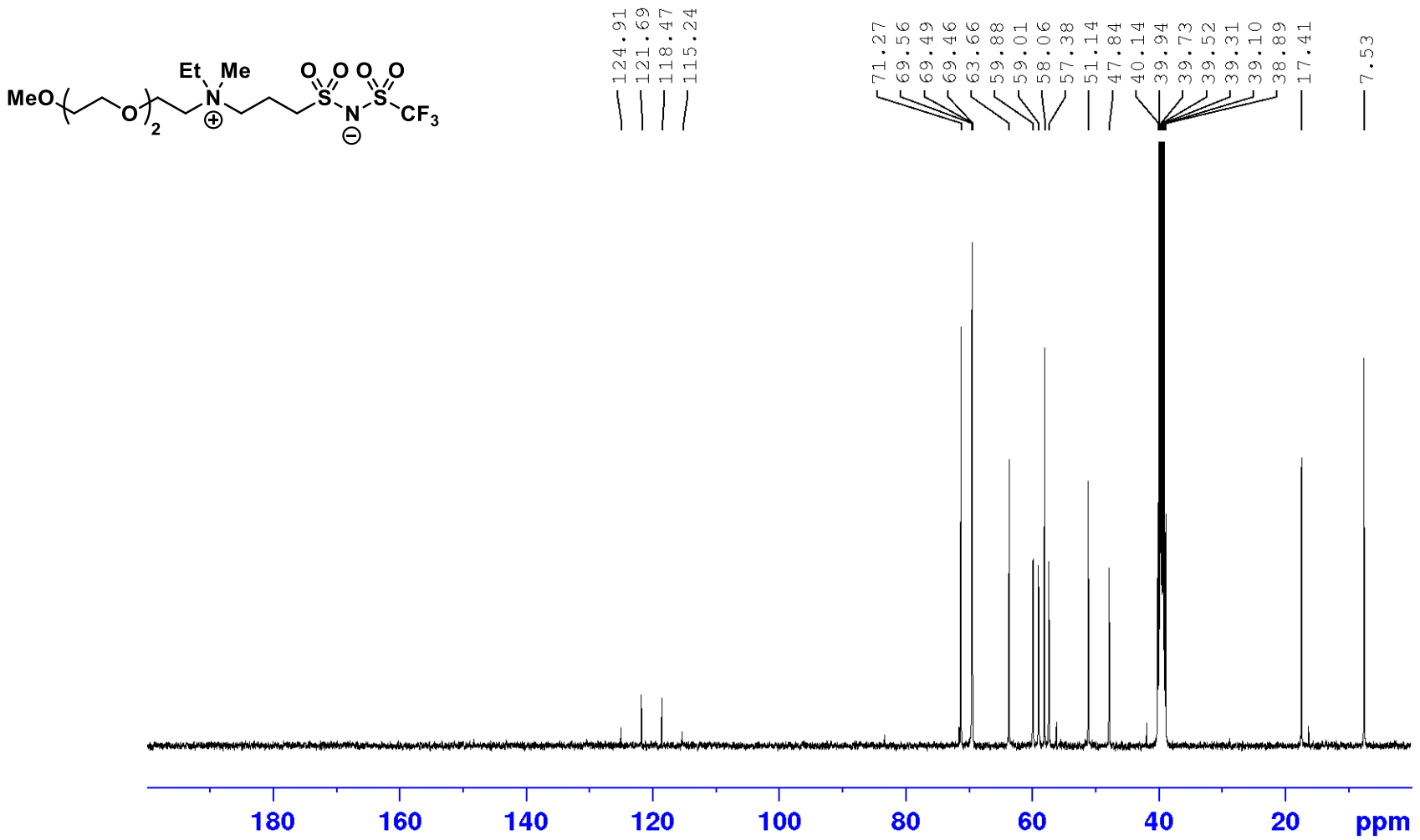
^{1}H NMR spectrum of ZIL 1b



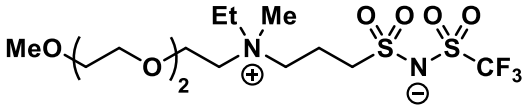
^{19}F NMR spectrum of ZIL 1b



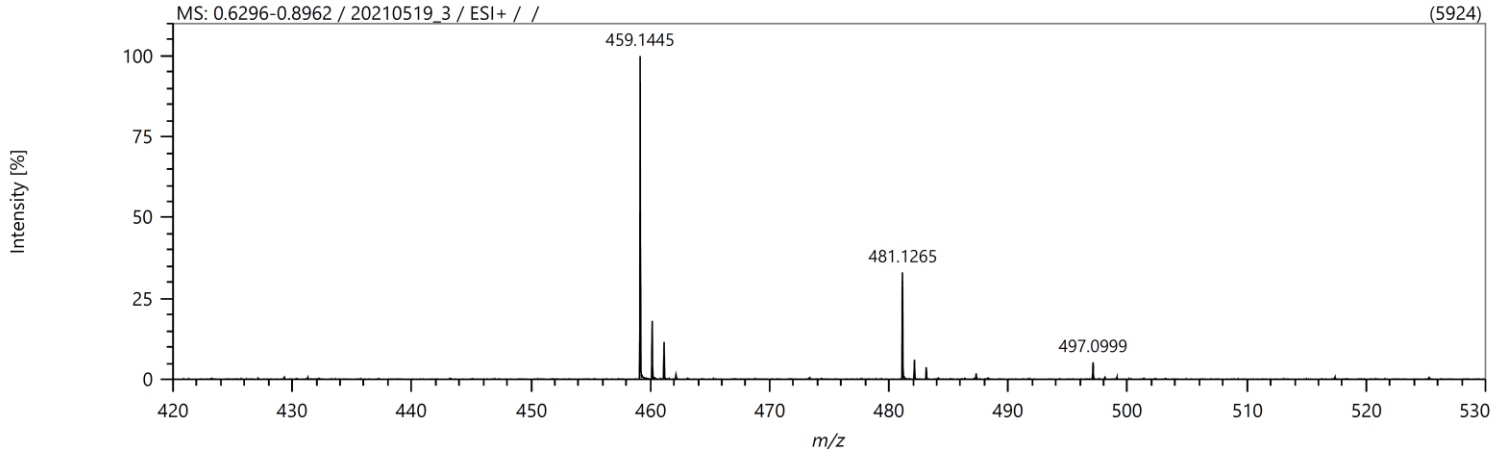
^{13}C NMR spectrum of ZIL 1b



Mass spectrum of ZIL 1b



Spectrum



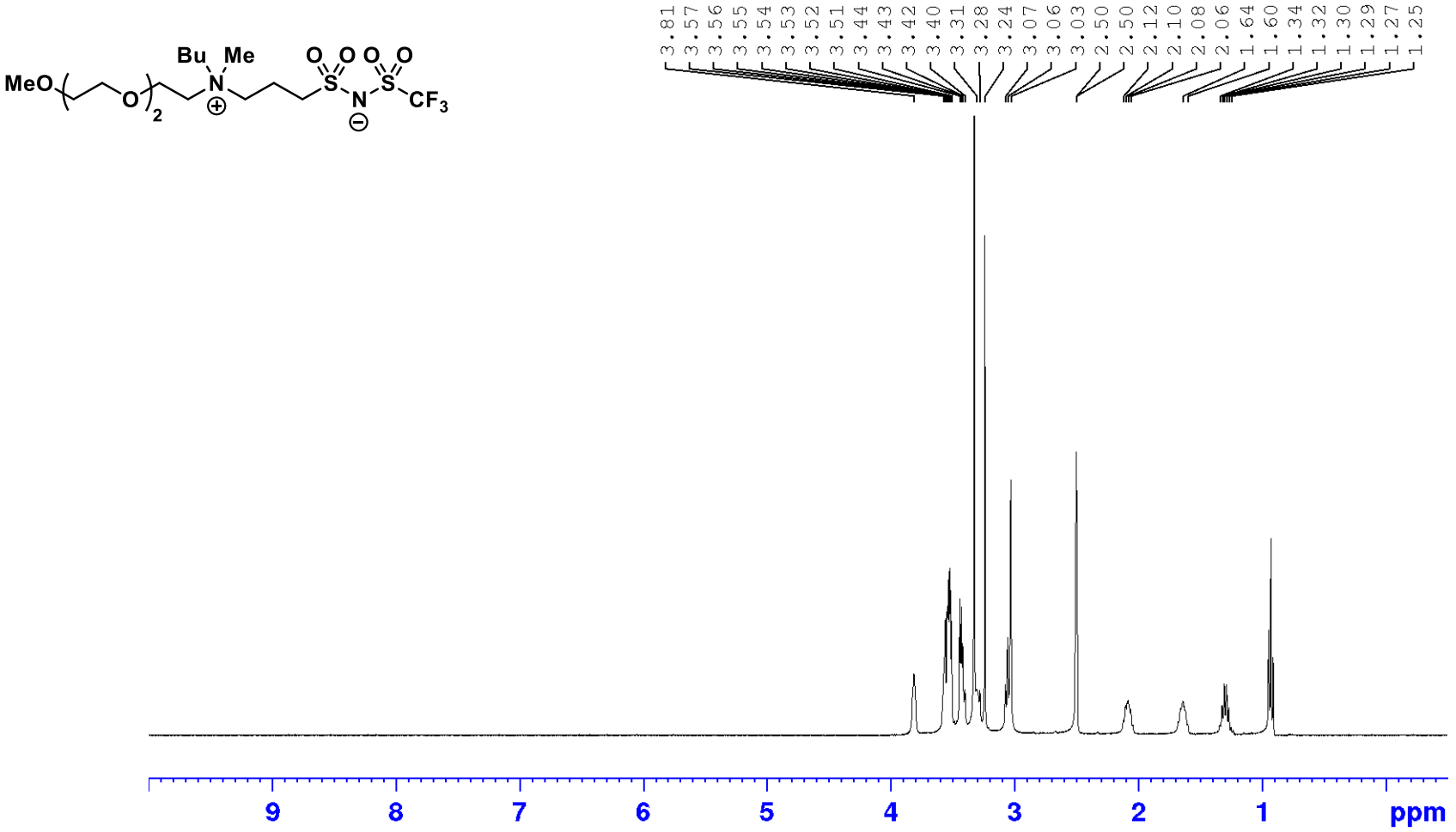
Elemental Composition

Parameters		Elements Set 1:								
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	3	2	7	2	0	0
Charge:	+1	Max	400	1000	3	2	7	2	1	1
DBE:	-99.0 - 999.0									

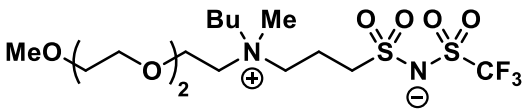
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
459.14446	C14 H30 N2 O7 F3 S2	459.14410	0.36	0.78	-0.5
481.12646	C14 H29 N2 O7 F3 Na S2	481.12605	0.41	0.85	-0.5
497.09987	C14 H29 N2 O7 F3 S2 K	497.09999	-0.11	-0.23	-0.5

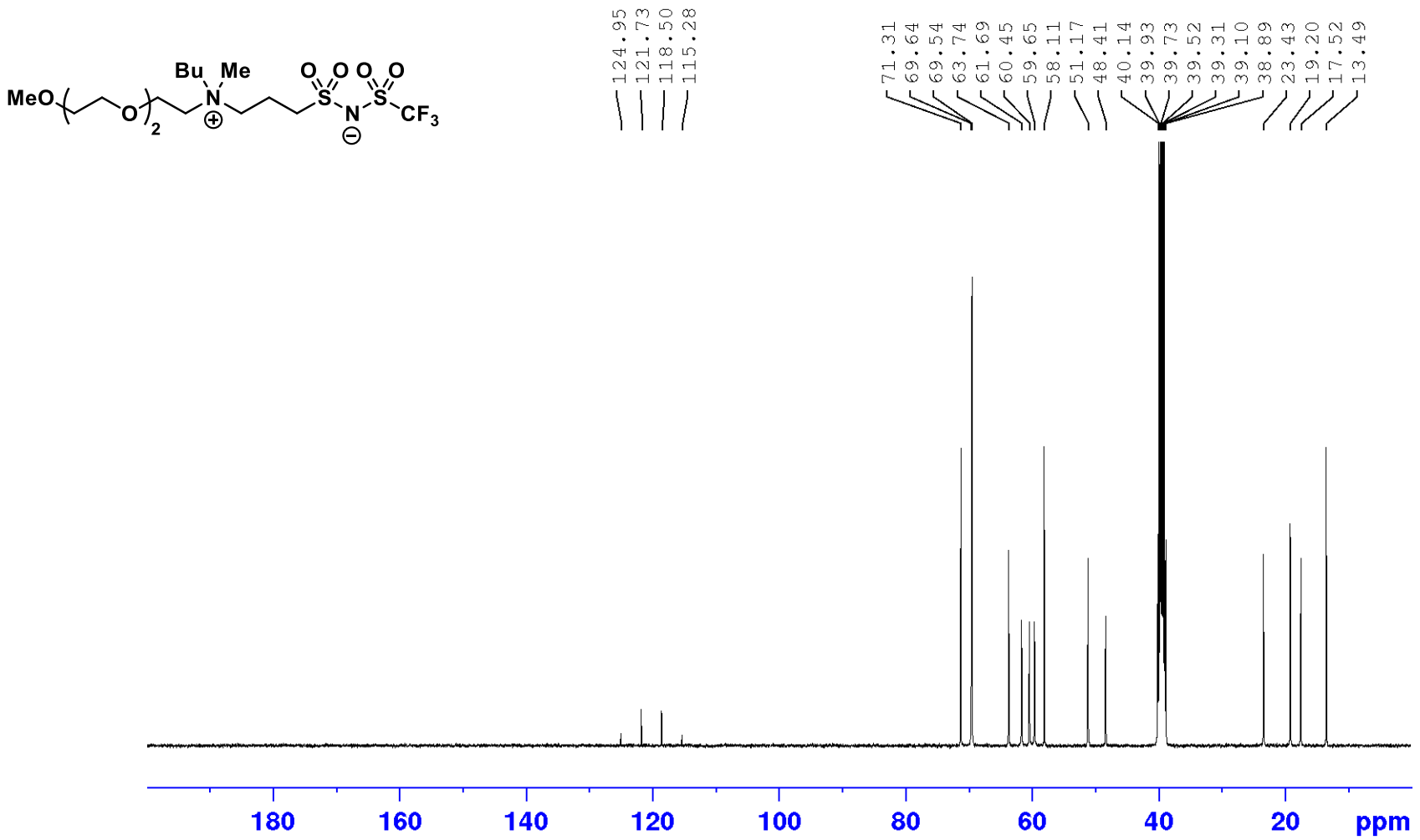
^{1}H NMR spectrum of ZIL 1c



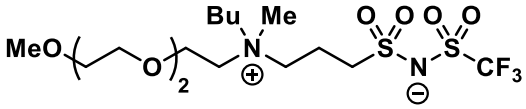
^{19}F NMR spectrum of ZIL 1c



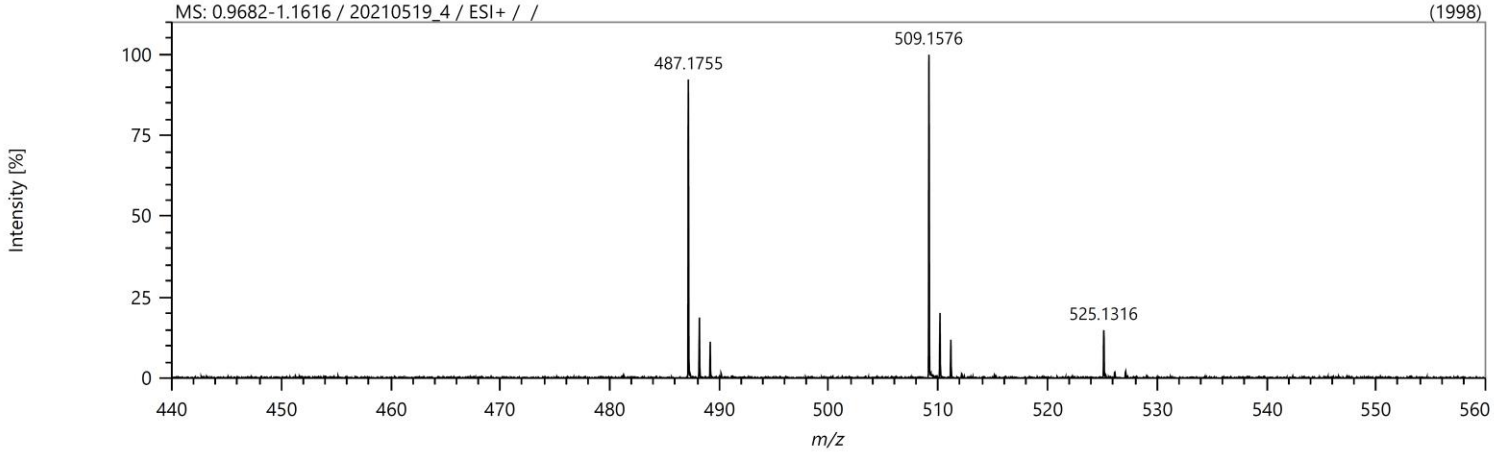
^{13}C NMR spectrum of ZIL 1c



Mass spectrum of ZIL 1c



Spectrum



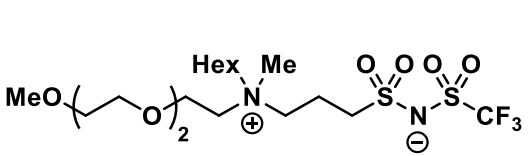
Elemental Composition

Parameters		Elements Set 1:								
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	3	2	7	2	0	0
Charge:	+1	Max	400	1000	3	2	7	2	1	1
DBE:	-99.0 - 999.0									

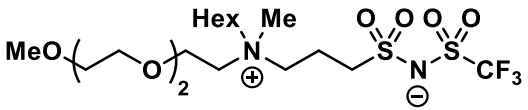
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
487.17552	C16 H34 N2 O7 F3 S2	487.17540	0.12	0.25	-0.5
509.15760	C16 H33 N2 O7 F3 Na S2	509.15735	0.25	0.50	-0.5
525.13164	C16 H33 N2 O7 F3 S2 K	525.13129	0.36	0.68	-0.5

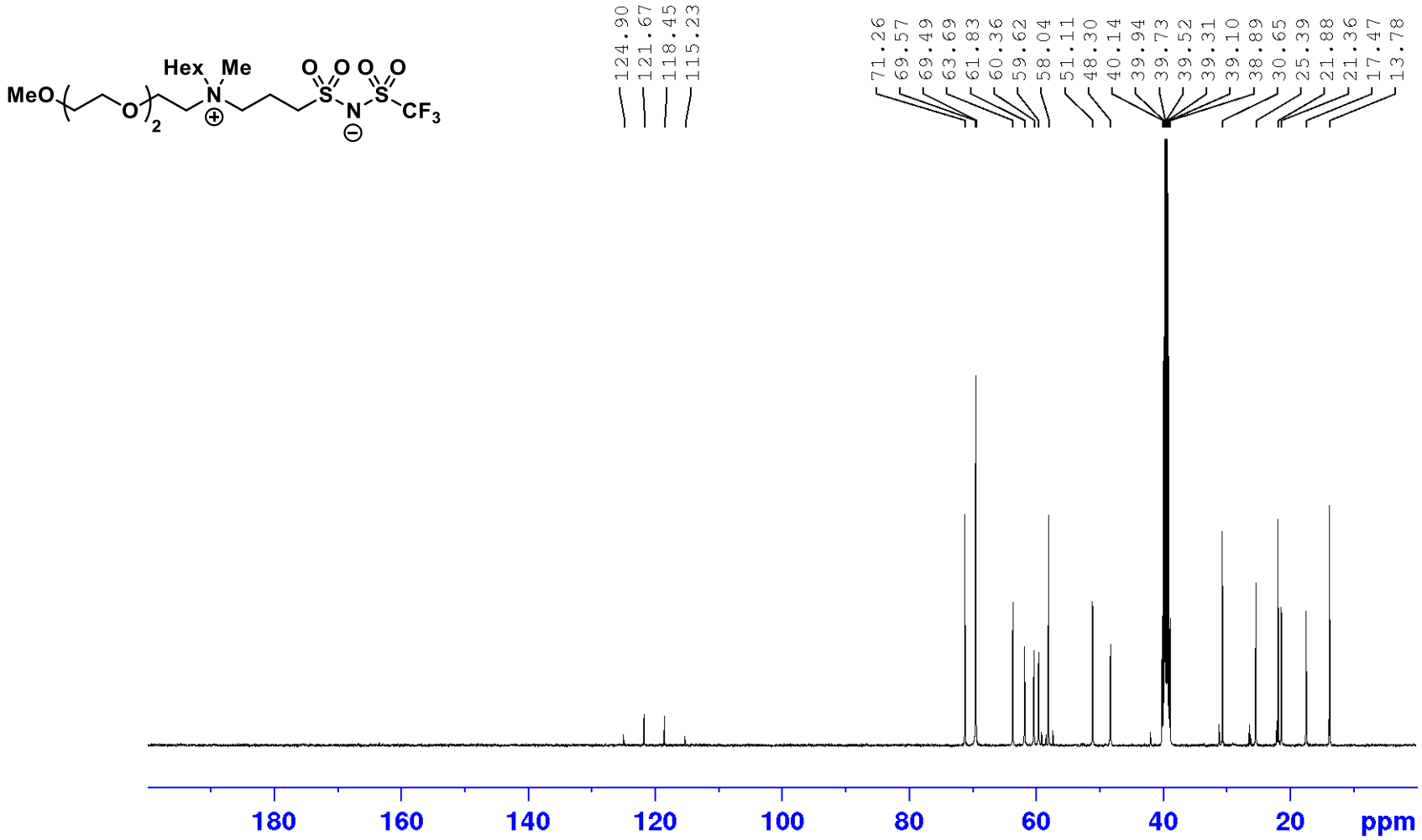
^{1}H NMR spectrum of ZIL 1d



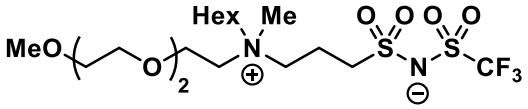
^{19}F NMR spectrum of ZIL 1d



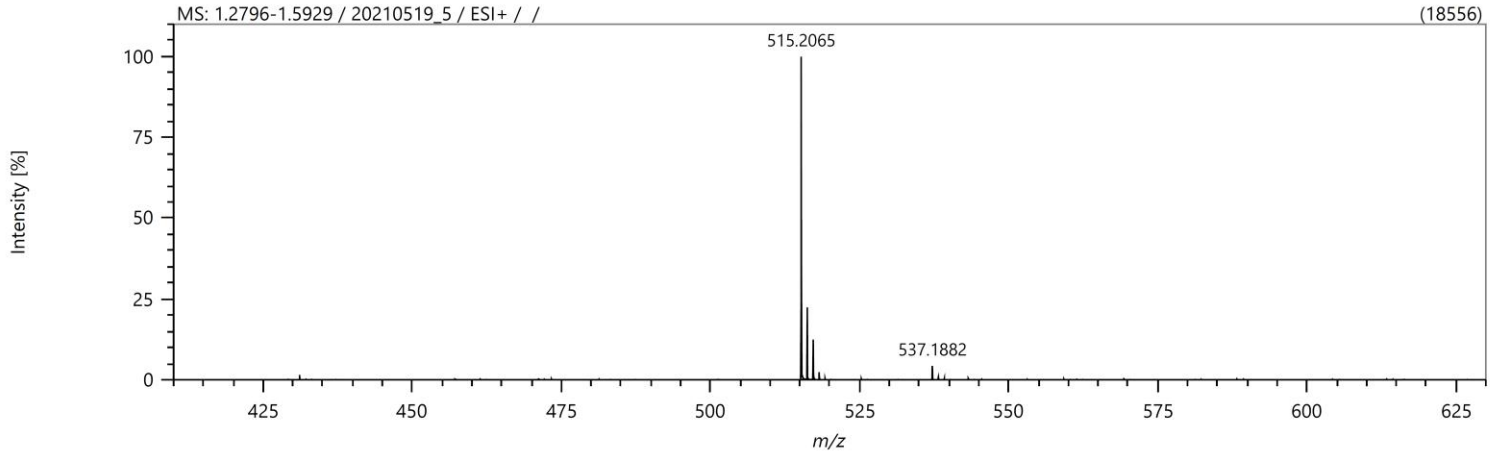
^{13}C NMR spectrum of ZIL 1d



Mass spectrum of ZIL 1d



Spectrum



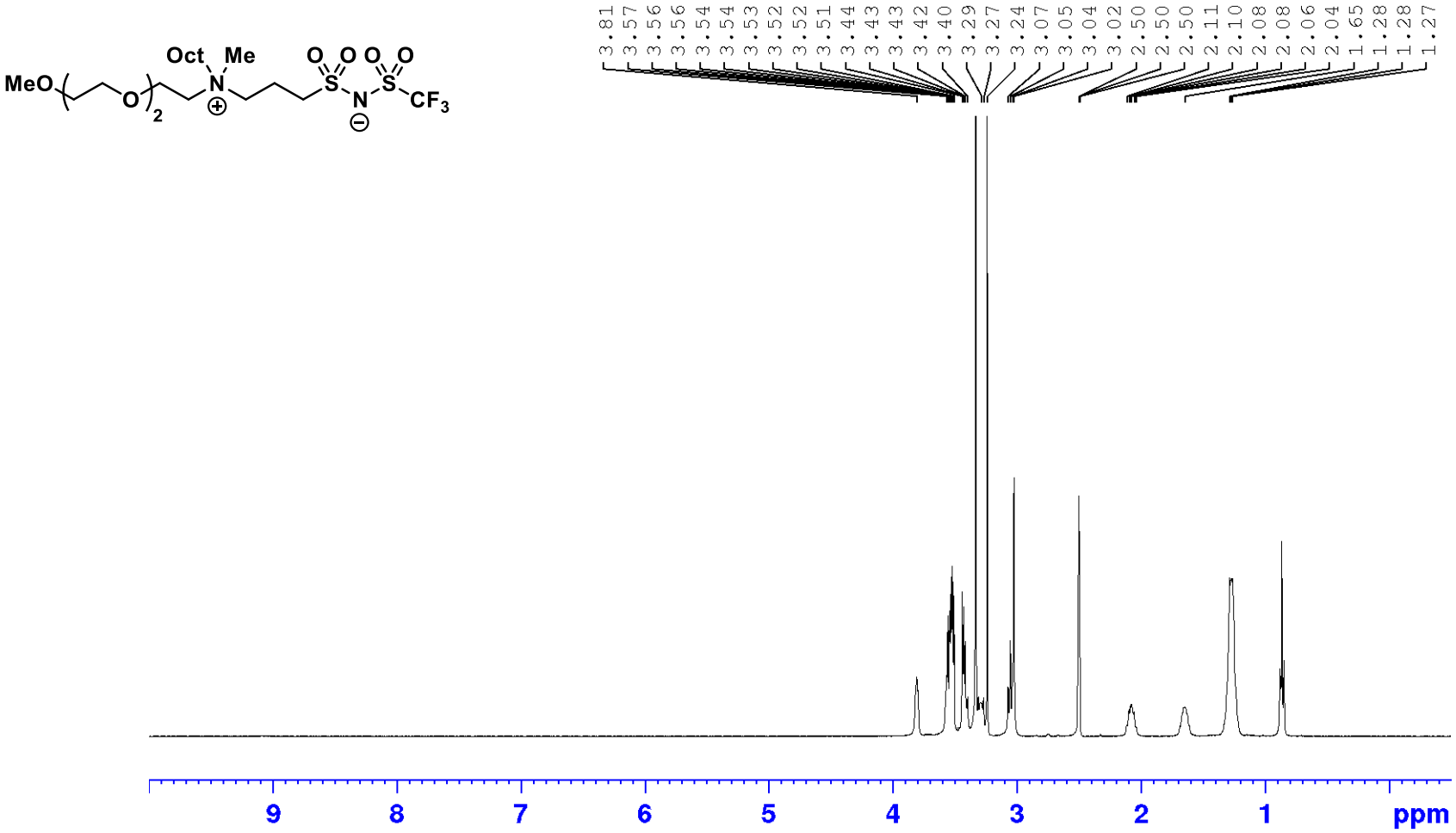
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	7	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

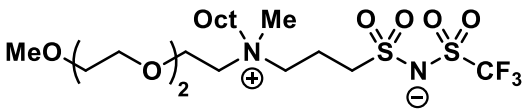
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
515.20645	C18 H38 N2 O7 F3 S2	515.20670	-0.25	-0.49	-0.5
537.18818	C18 H37 N2 O7 F3 Na S2	537.18865	-0.46	-0.86	-0.5

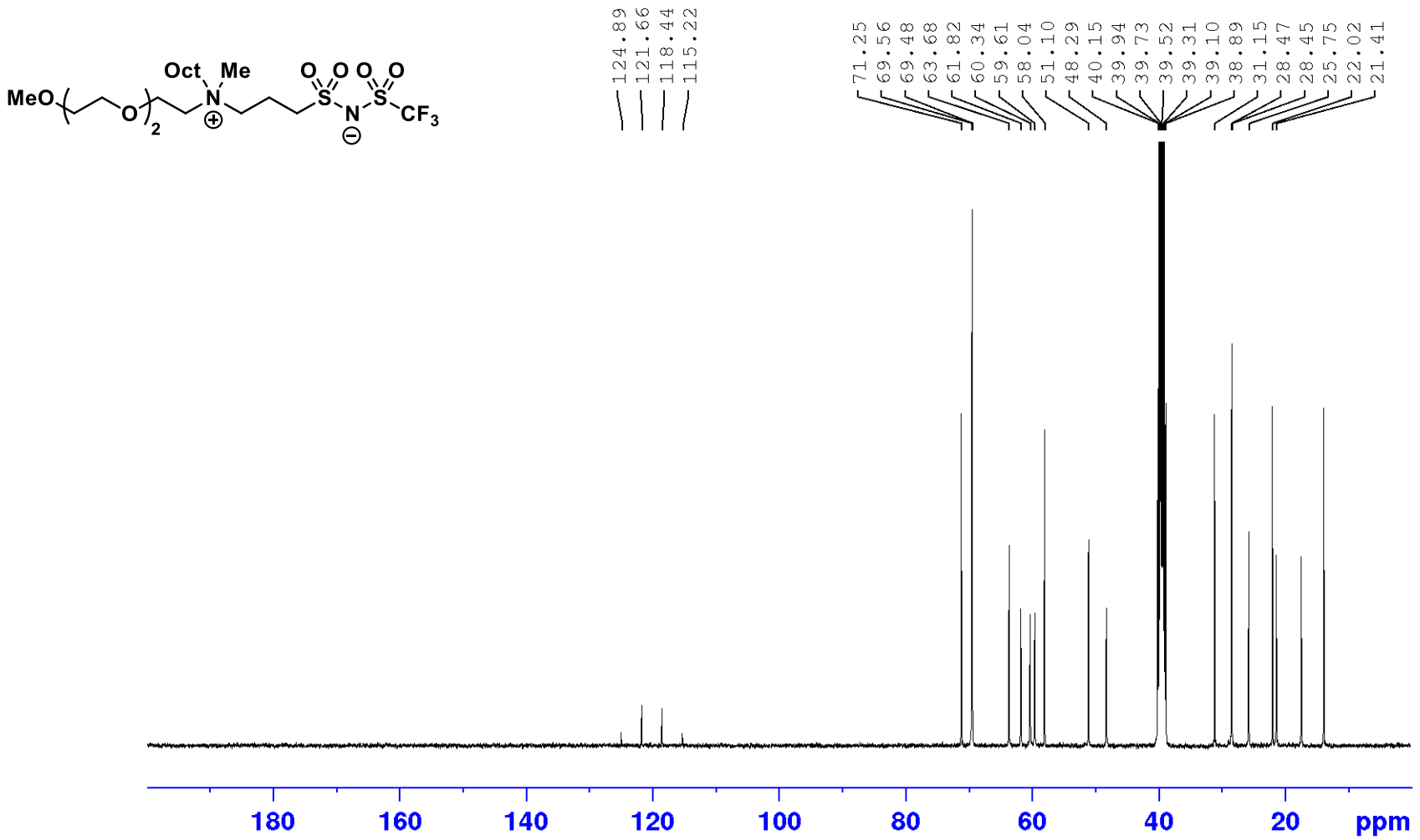
^{1}H NMR spectrum of ZIL 1e



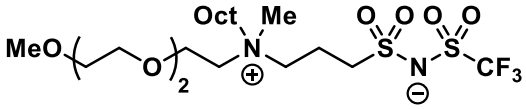
^{19}F NMR spectrum of ZIL 1e



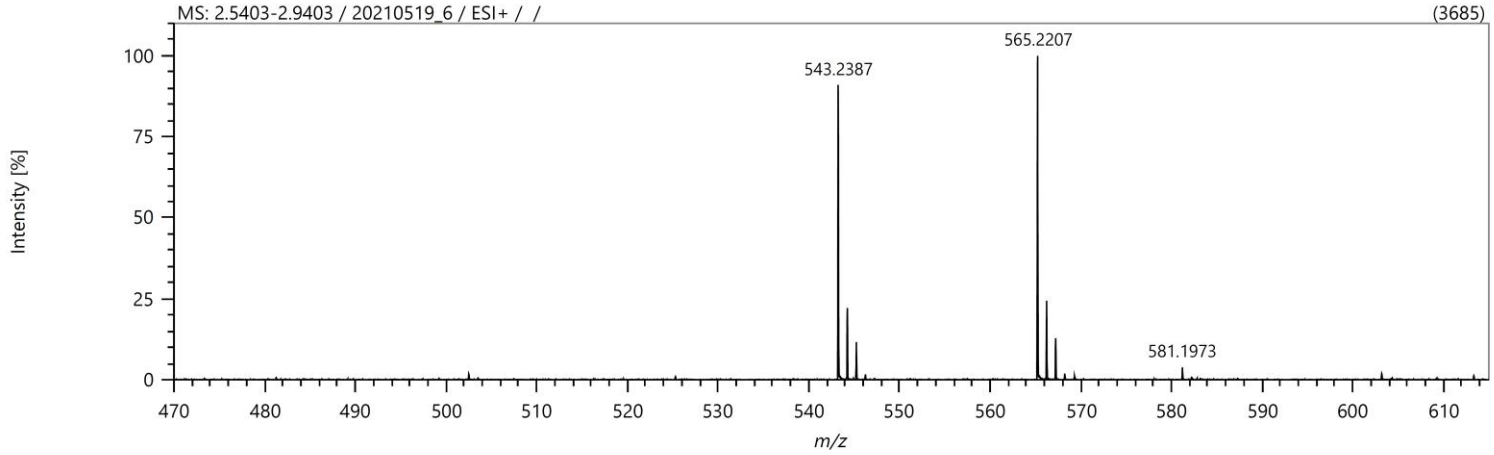
^{13}C NMR spectrum of ZIL 1e



Mass spectrum of ZIL 1e



Spectrum



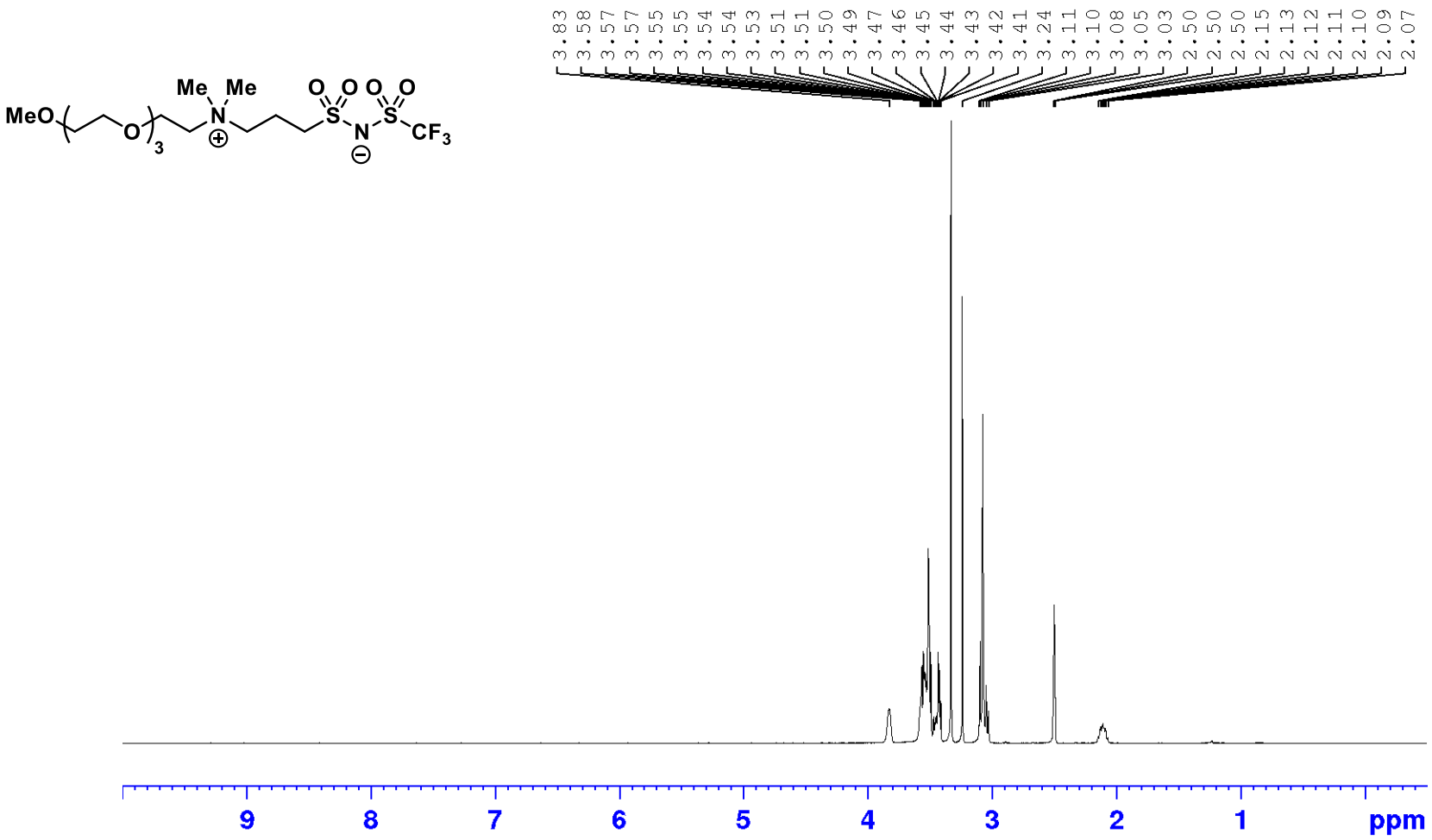
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	7	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

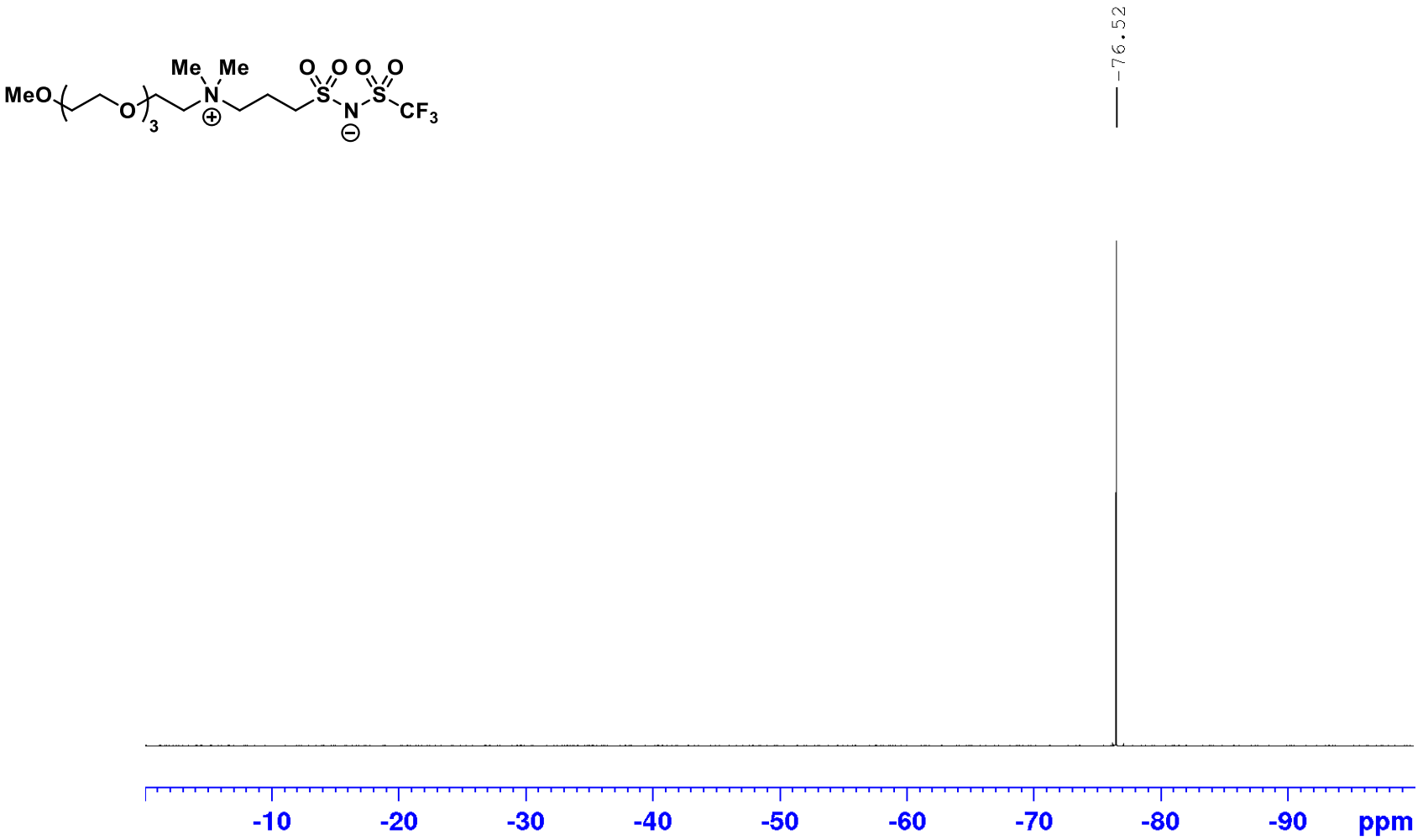
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
543.23867	C20 H42 N2 O7 F3 S2	543.23800	0.66	1.22	-0.5
565.22065	C20 H41 N2 O7 F3 Na S2	565.21995	0.70	1.24	-0.5

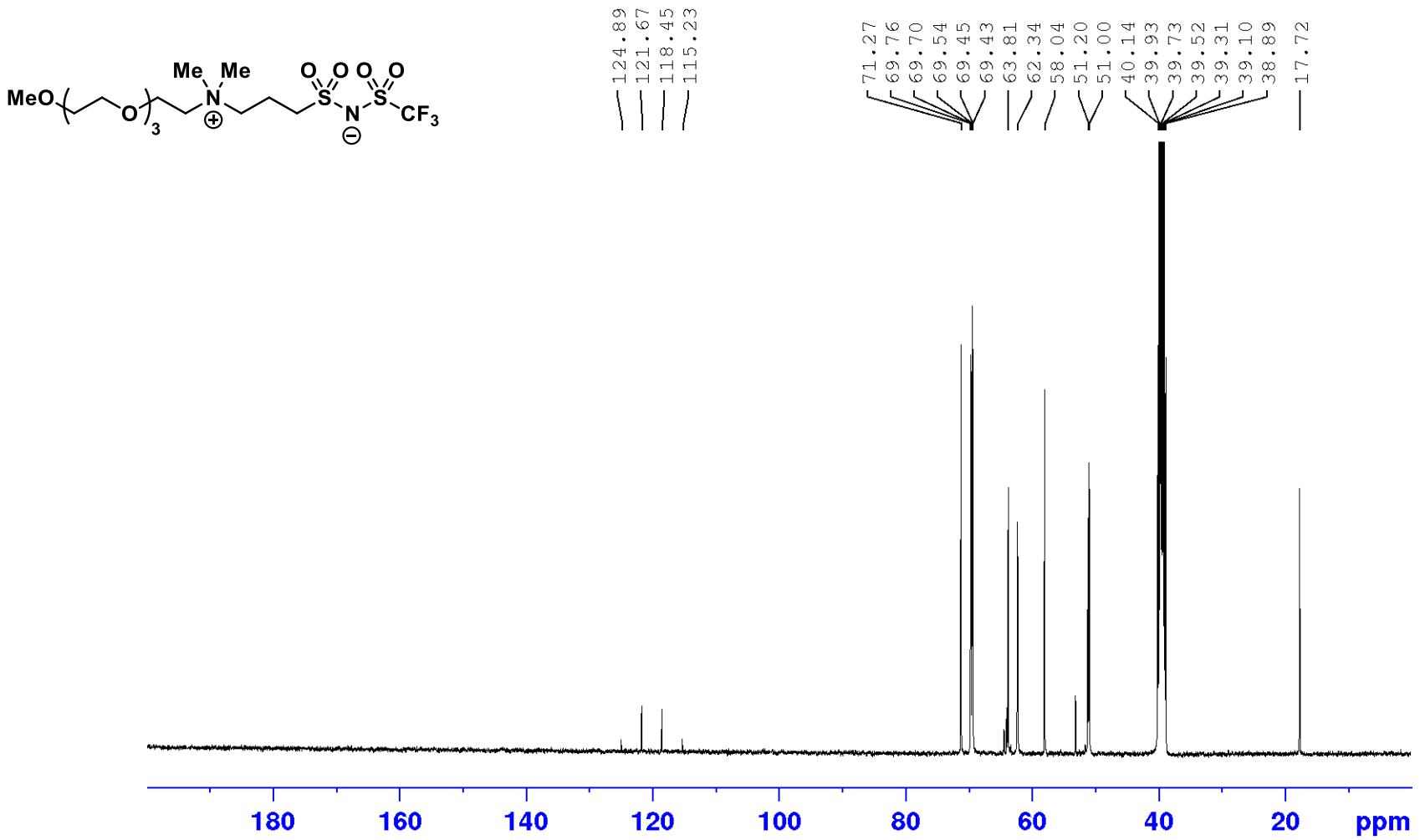
^{1}H NMR spectrum of ZIL 2a



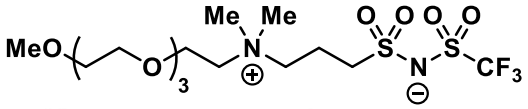
^{19}F NMR spectrum of ZIL 2a



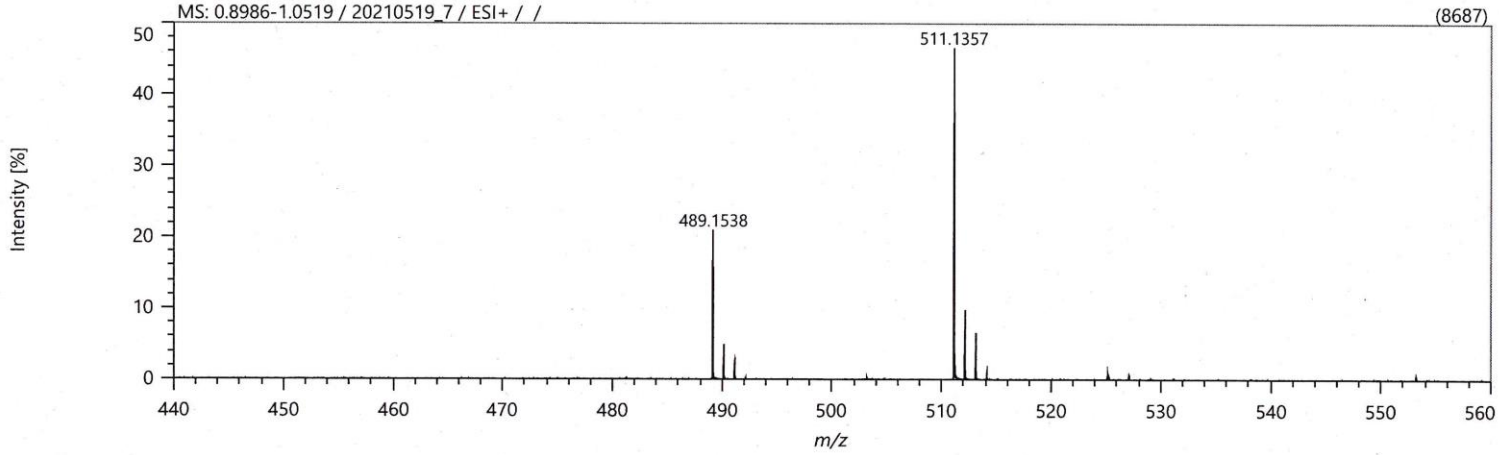
^{13}C NMR spectrum of ZIL 2a



Mass spectrum of ZIL 2a



Spectrum



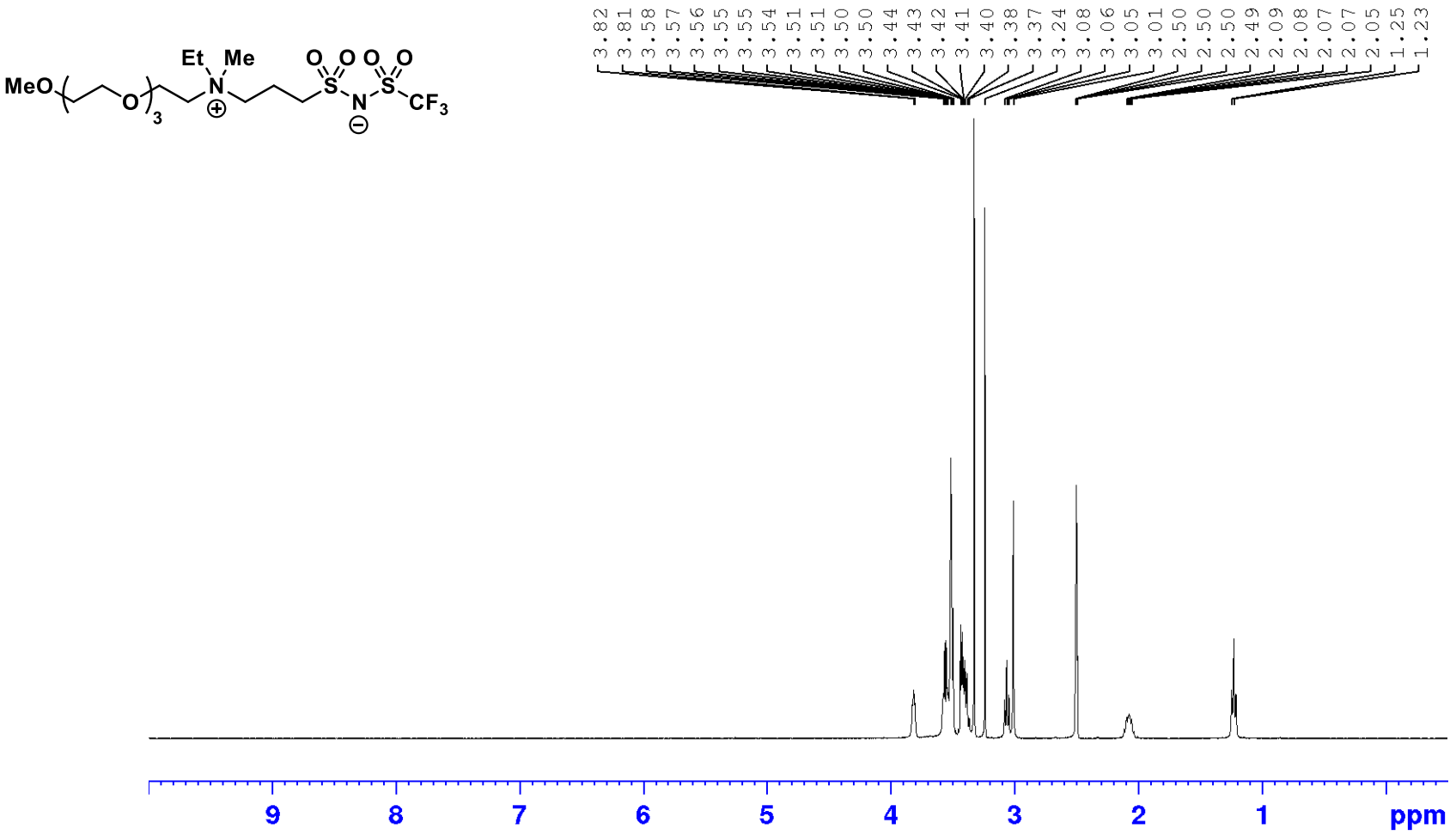
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	8	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

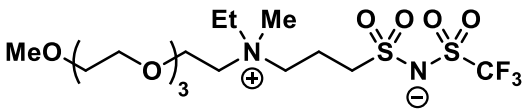
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
489.15385	C15 H32 N2 O8 F3 S2	489.15467	-0.82	-1.68	-0.5
511.13571	C15 H31 N2 O8 F3 Na S2	511.13661	-0.90	-1.76	-0.5

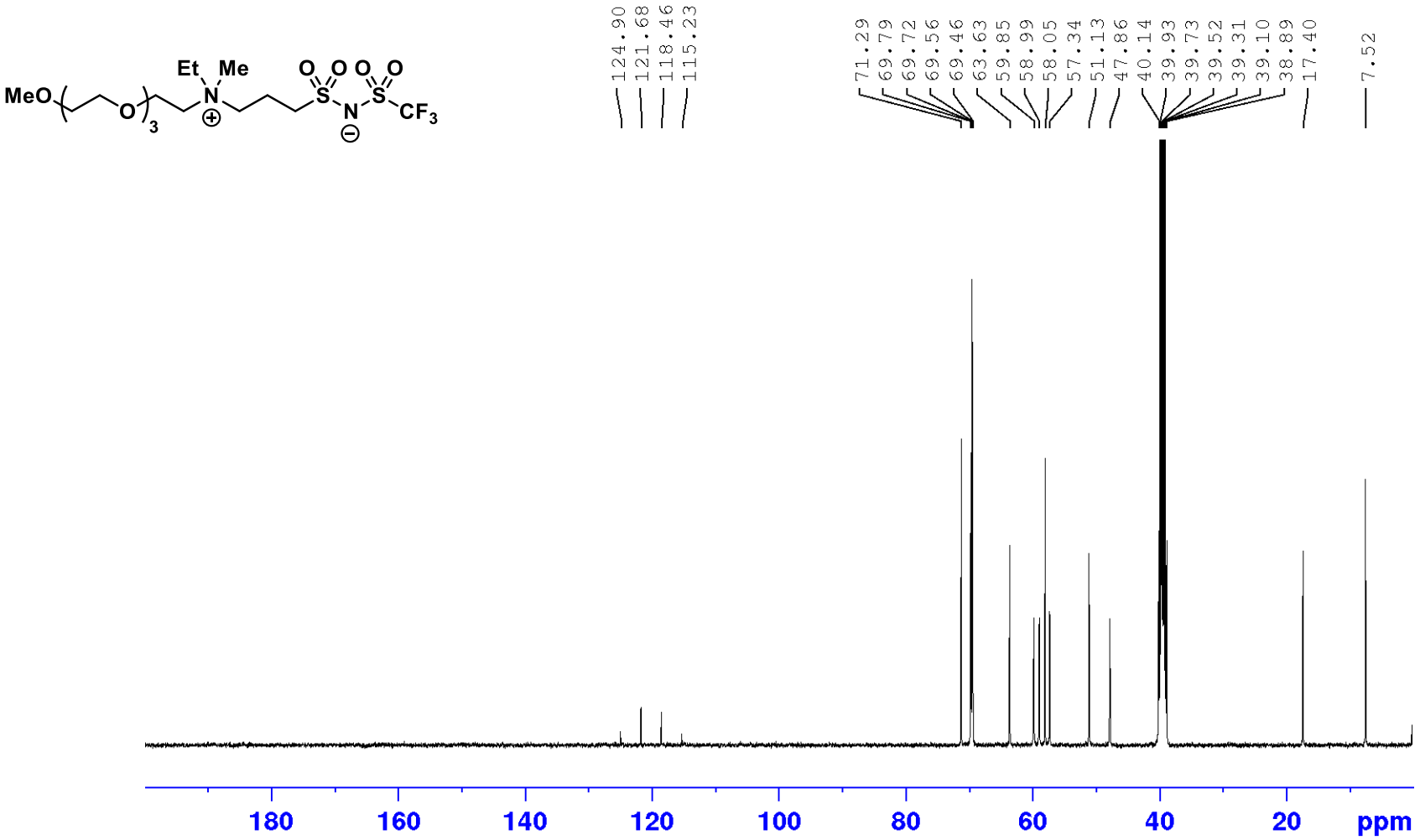
^{1}H NMR spectrum of ZIL 2b



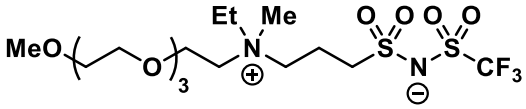
^{19}F NMR spectrum of ZIL 2b



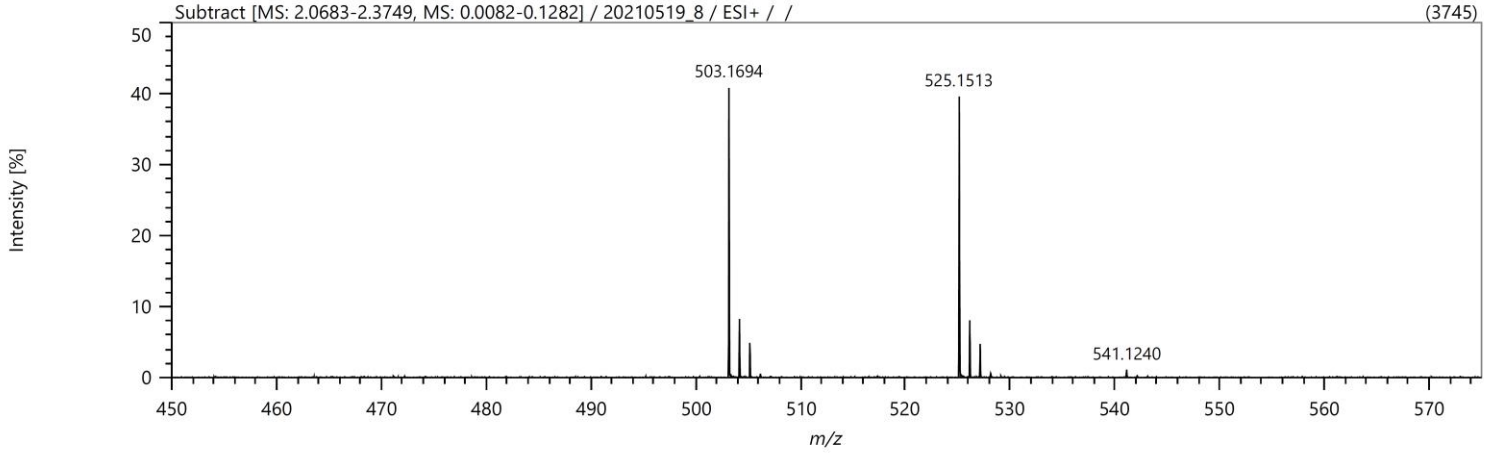
^{13}C NMR spectrum of ZIL 2b



Mass spectrum of ZIL 2b



Spectrum



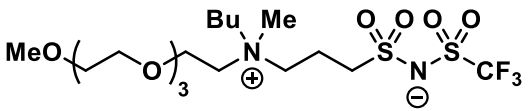
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	8	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

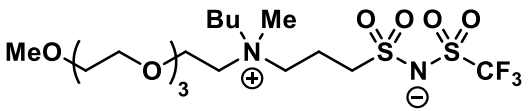
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
503.16939	C16 H34 N2 O8 F3 S2	503.17032	-0.92	-1.84	-0.5
525.15129	C16 H33 N2 O8 F3 Na S2	525.15226	-0.98	-1.86	-0.5

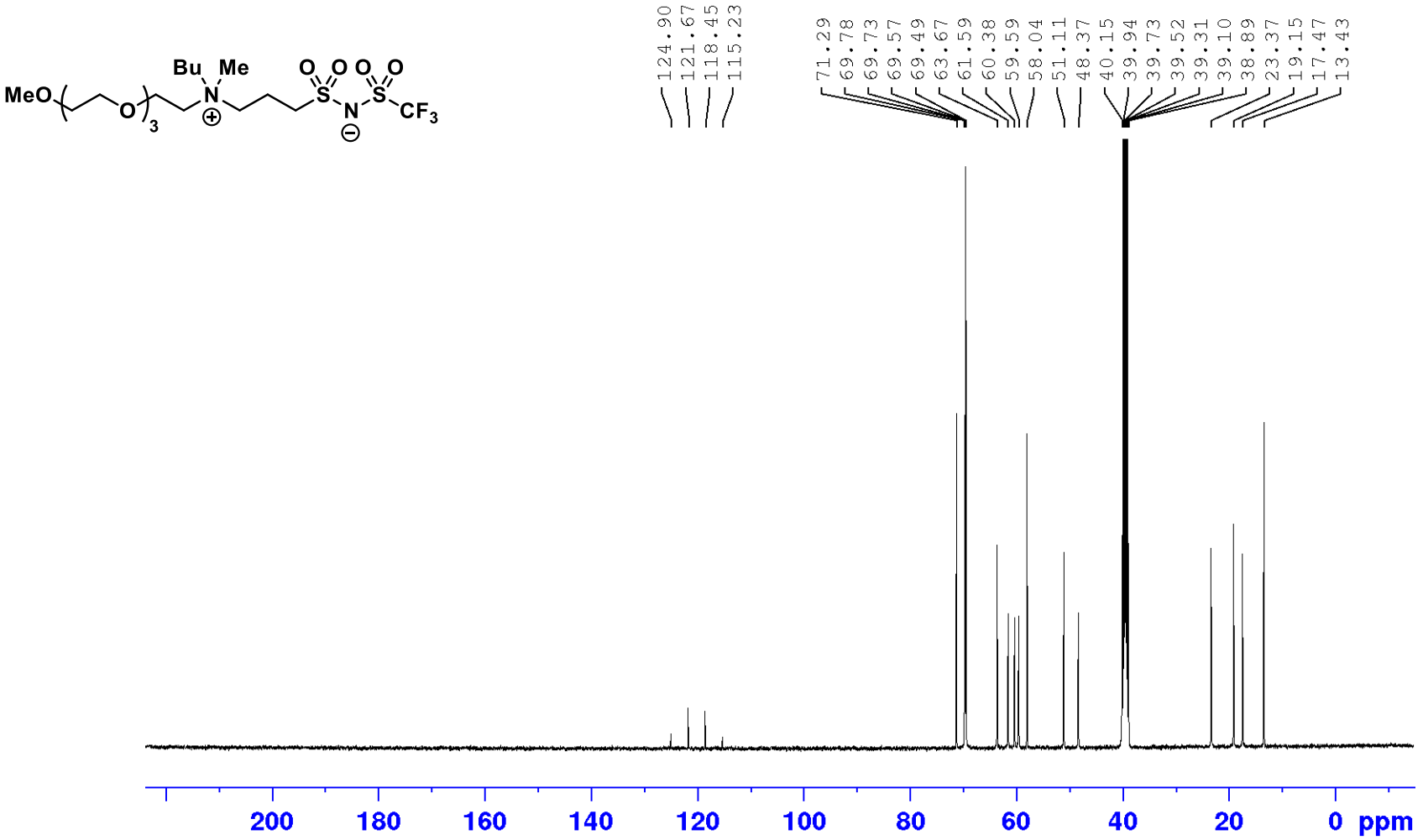
^{1}H NMR spectrum of ZIL 2c



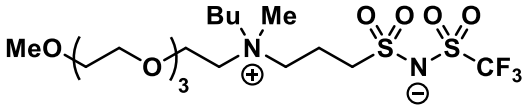
^{19}F NMR spectrum of ZIL 2c



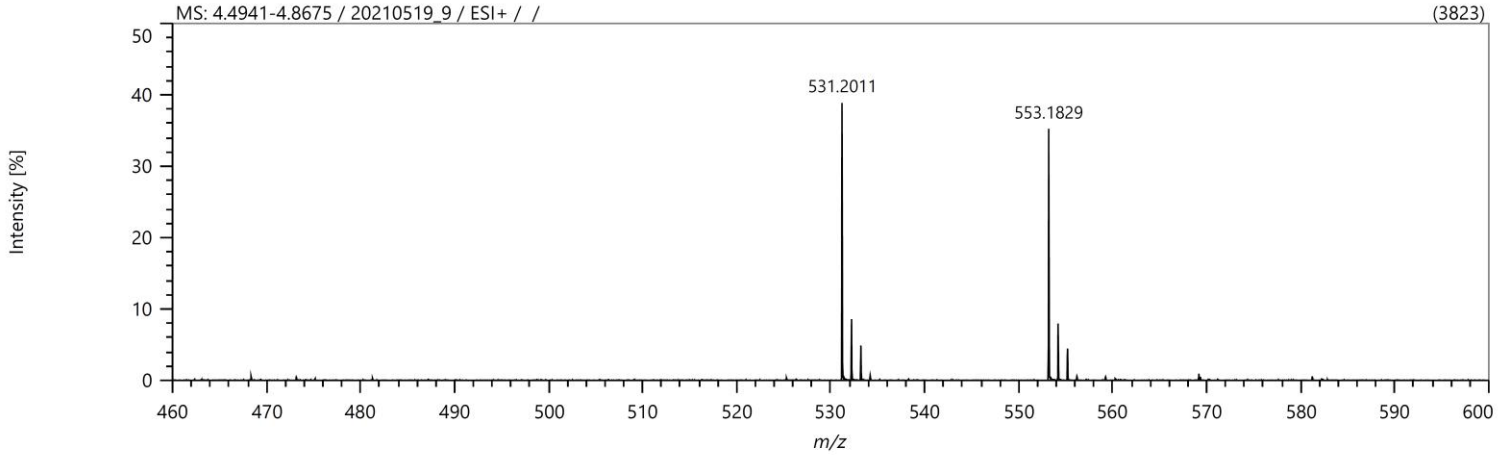
^{13}C NMR spectrum of ZIL 2c



Mass spectrum of ZIL 2c



Spectrum



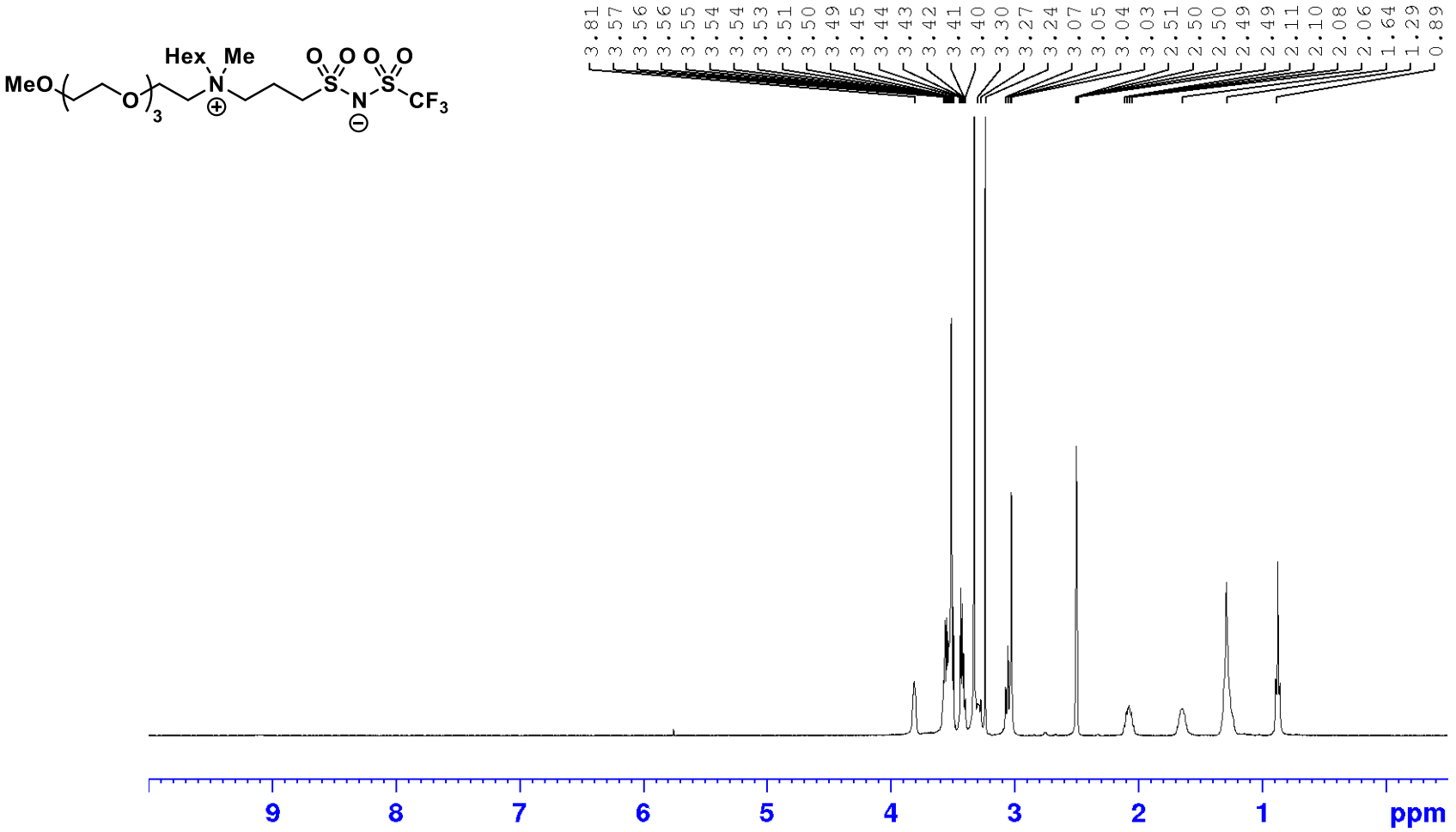
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	8	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

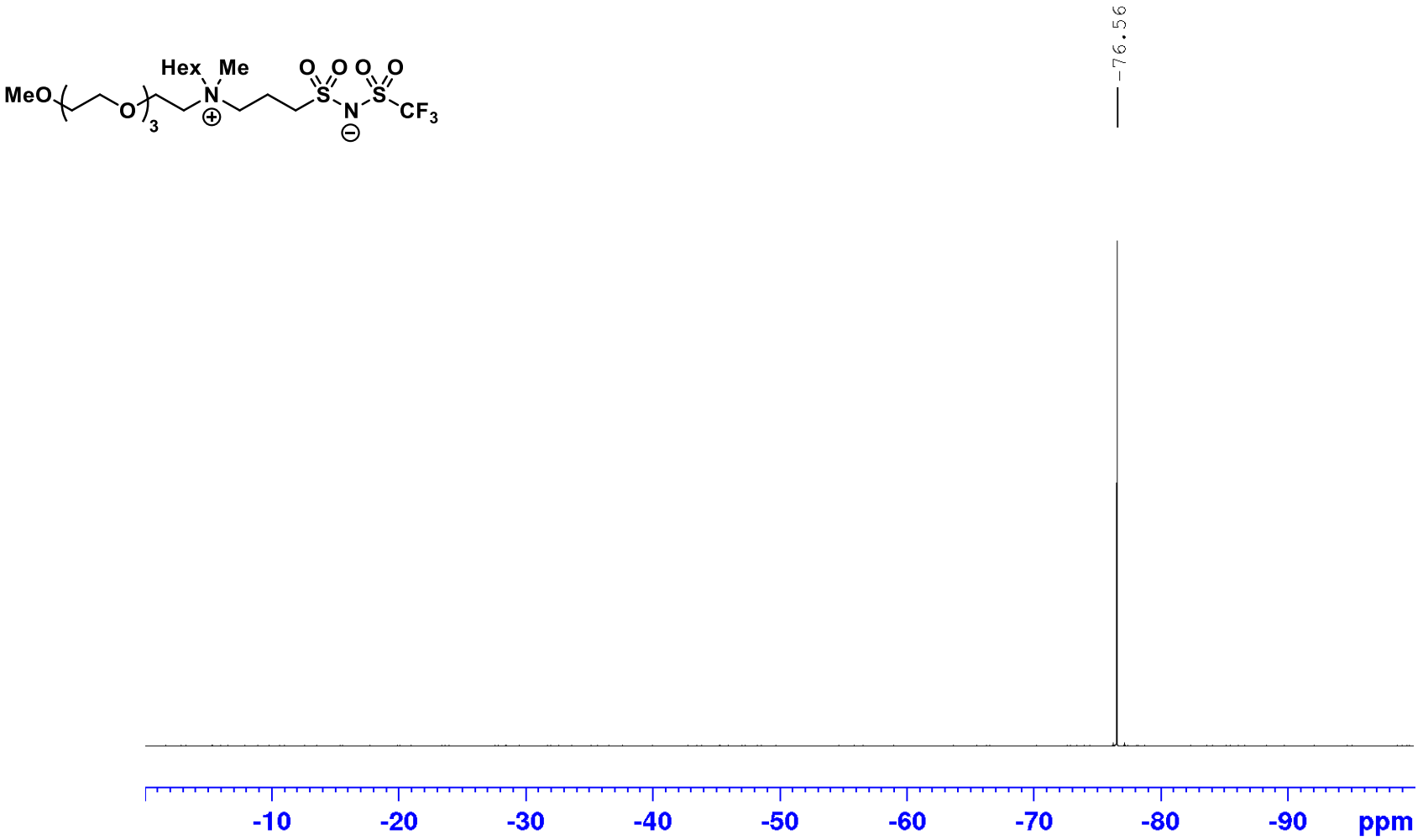
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
531.20105	C18 H38 N2 O8 F3 S2	531.20162	-0.57	-1.07	-0.5
553.18286	C18 H37 N2 O8 F3 Na S2	553.18356	-0.70	-1.27	-0.5

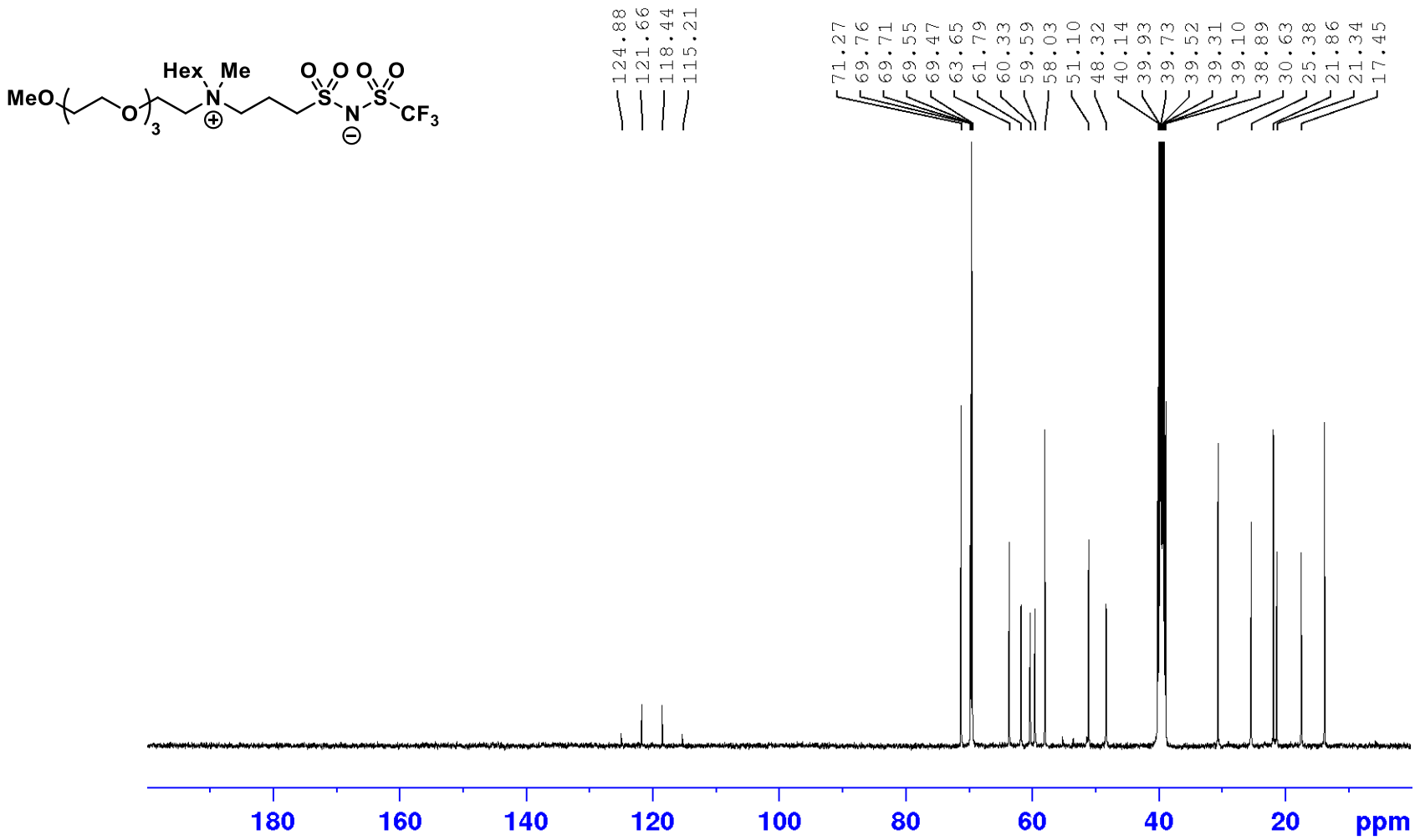
^{1}H NMR spectrum of ZIL 2d



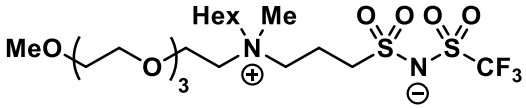
^{19}F NMR spectrum of ZIL 2d



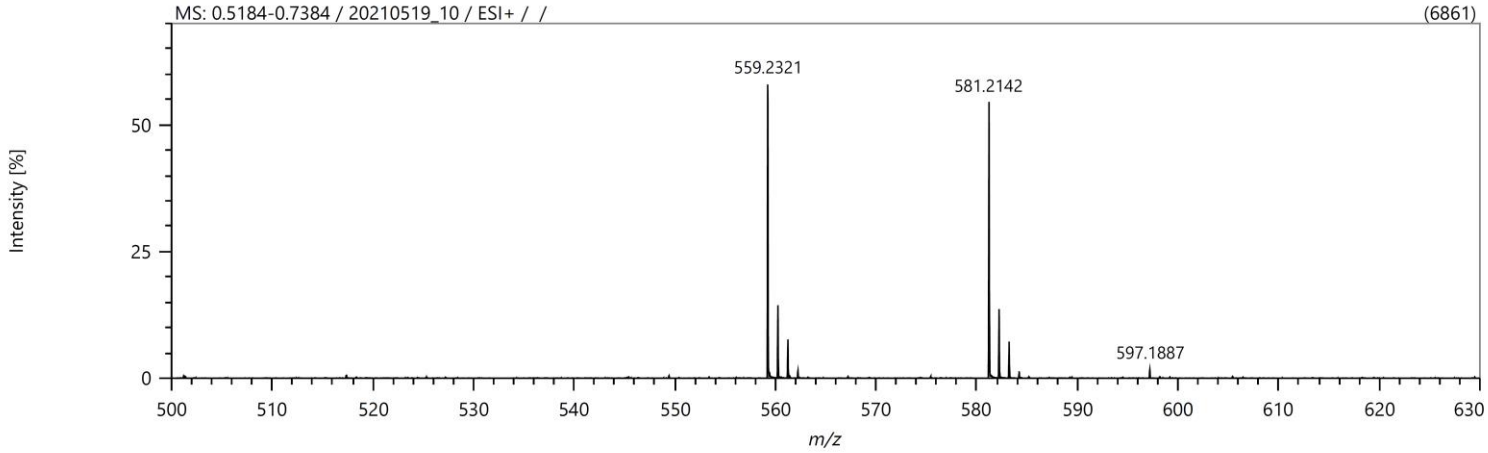
^{13}C NMR spectrum of ZIL 2d



Mass spectrum of ZIL 2d



Spectrum



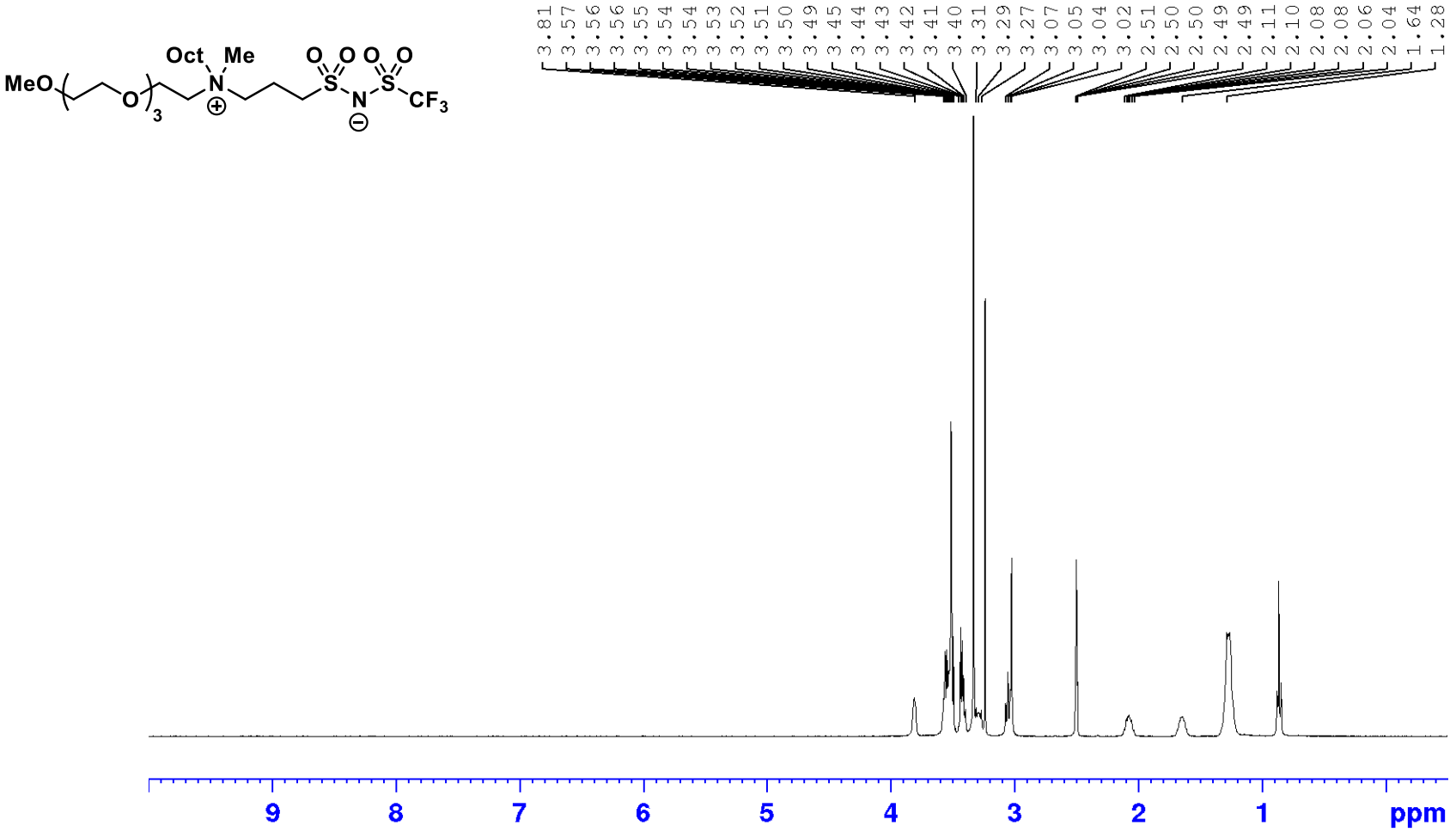
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	8	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

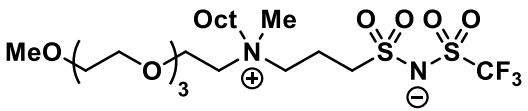
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
559.23205	C20 H42 N2 O8 F3 S2	559.23292	-0.87	-1.55	-0.5
581.21418	C20 H41 N2 O8 F3 Na S2	581.21486	-0.69	-1.18	-0.5

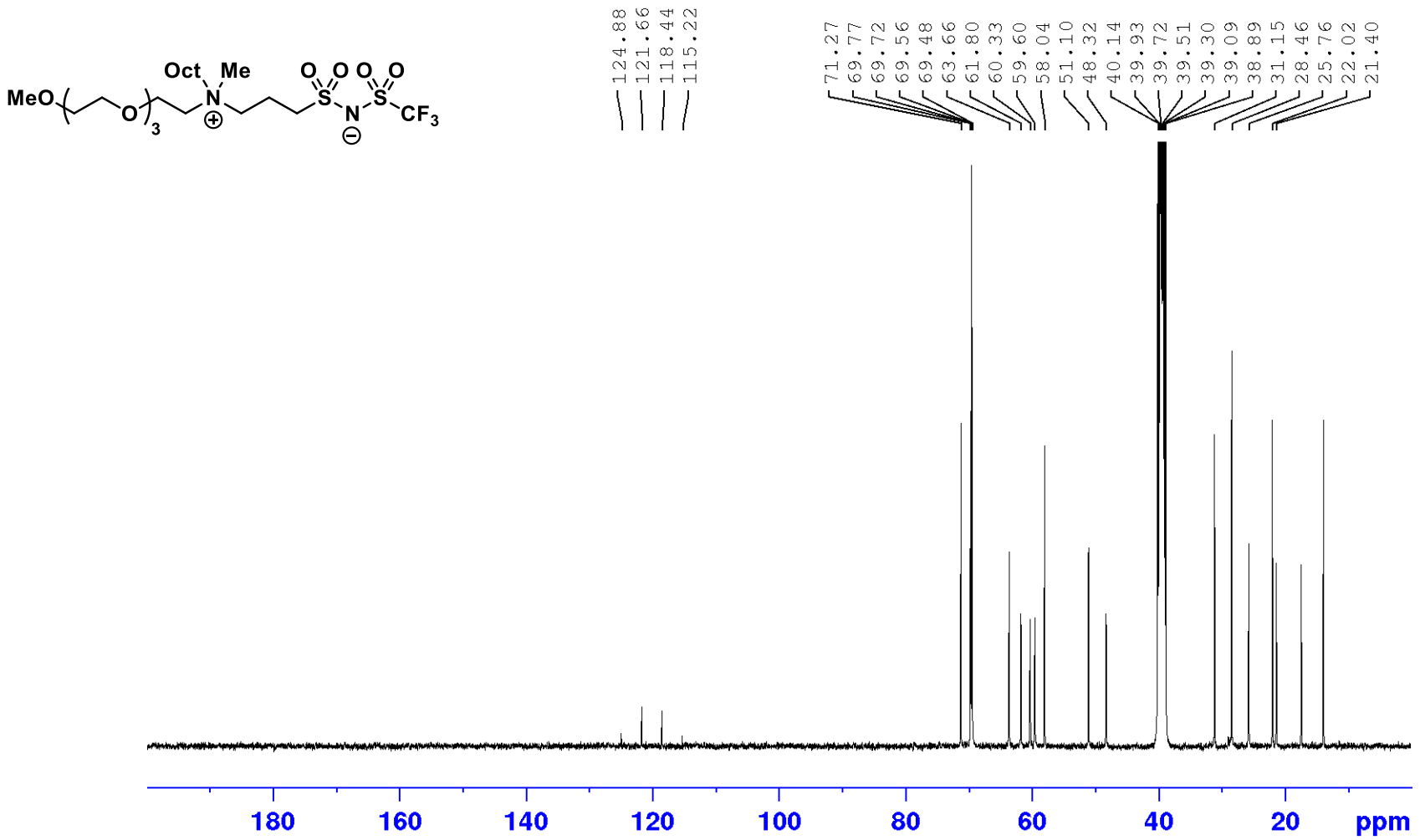
^{1}H NMR spectrum of ZIL 2e



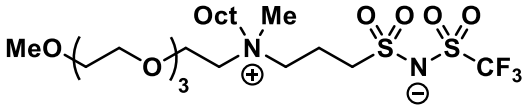
^{19}F NMR spectrum of ZIL 2e



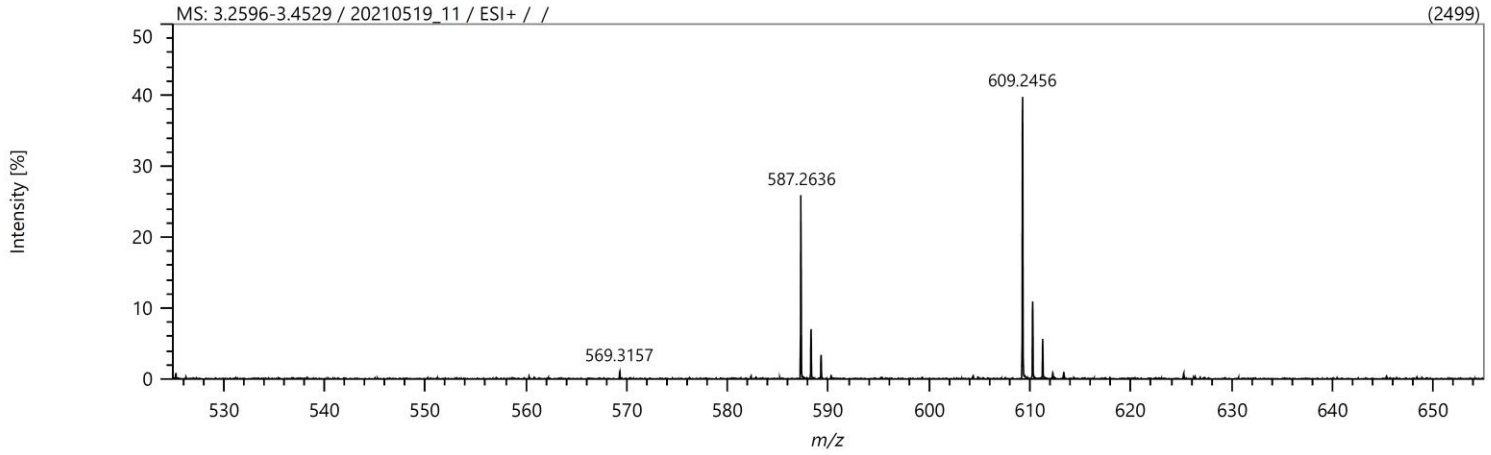
^{13}C NMR spectrum of ZIL 2e



Mass spectrum of ZIL 2e



Spectrum



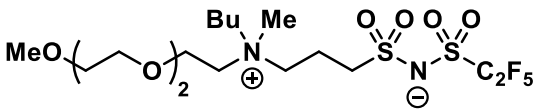
Elemental Composition

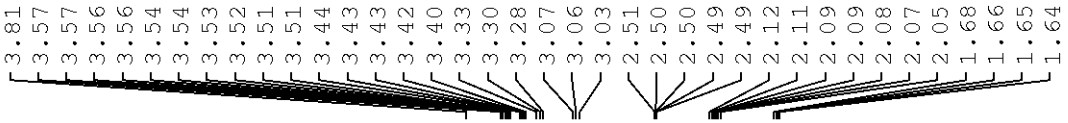
Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	3	2	8	2	0
Charge:	+1	Max	400	1000	3	2	8	2	1
DBE:	-99.0 - 999.0								

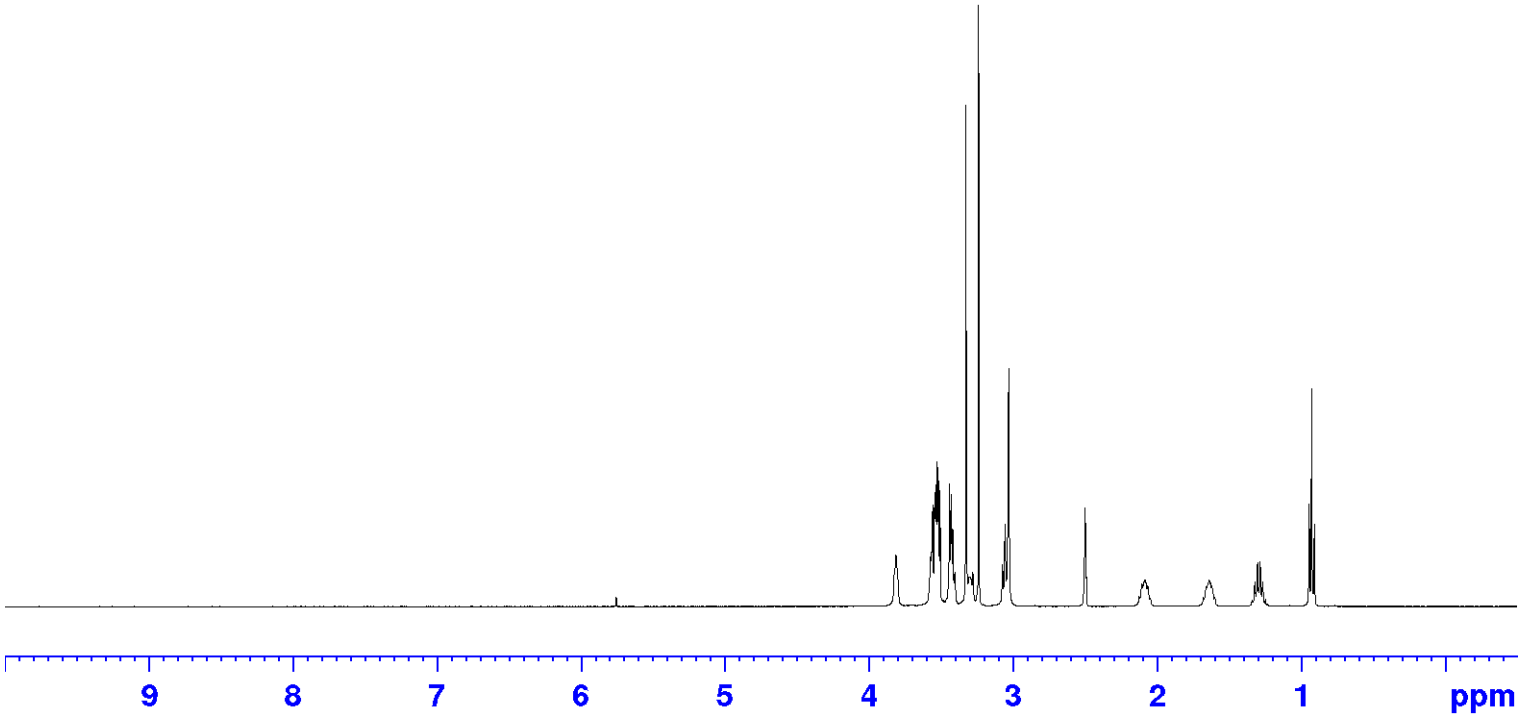
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
587.26365	C22 H46 N2 O8 F3 S2	587.26422	-0.57	-0.97	-0.5
609.24563	C22 H45 N2 O8 F3 Na S2	609.24616	-0.53	-0.87	-0.5

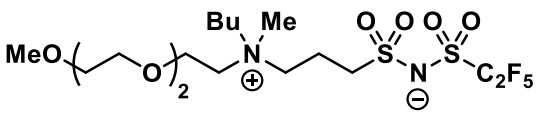
^{1}H NMR spectrum of ZIL 3c



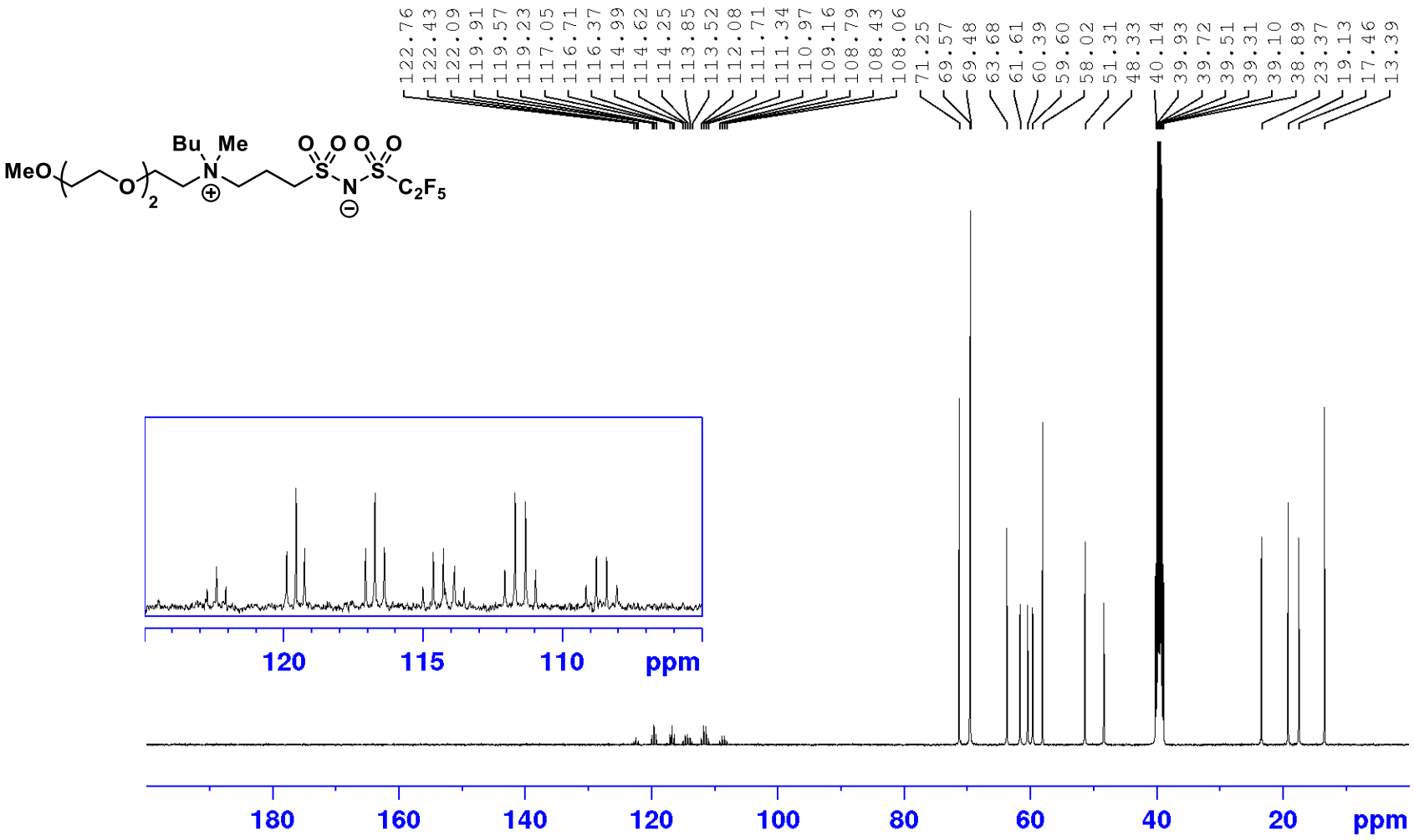




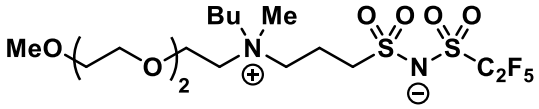
^{19}F NMR spectrum of ZIL 3c



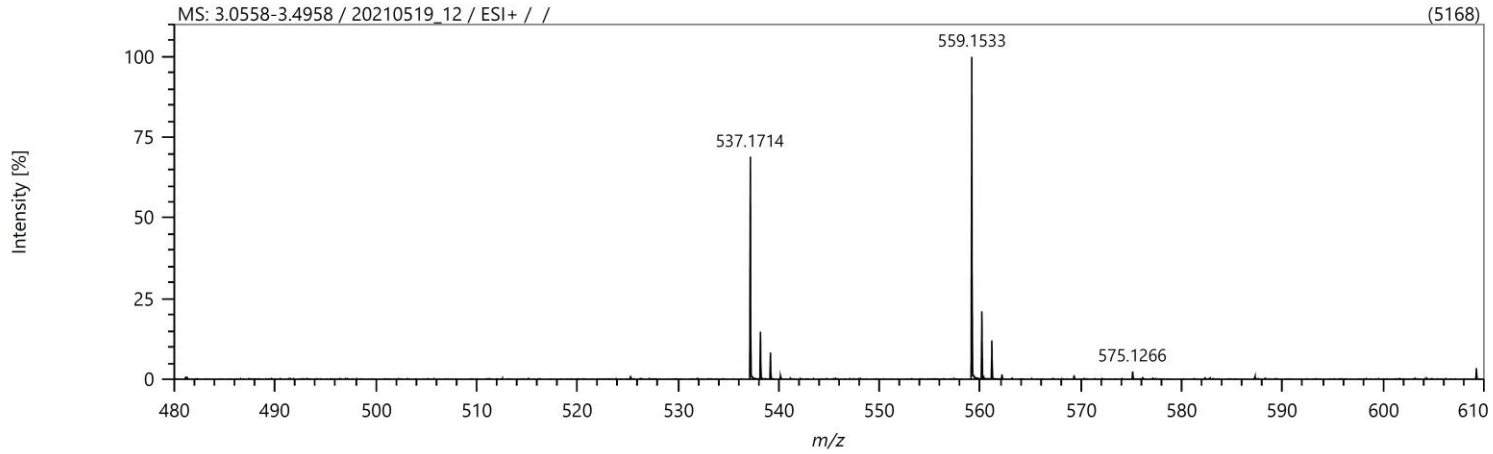
^{13}C NMR spectrum of ZIL 3c



Mass spectrum of ZIL 3c



Spectrum



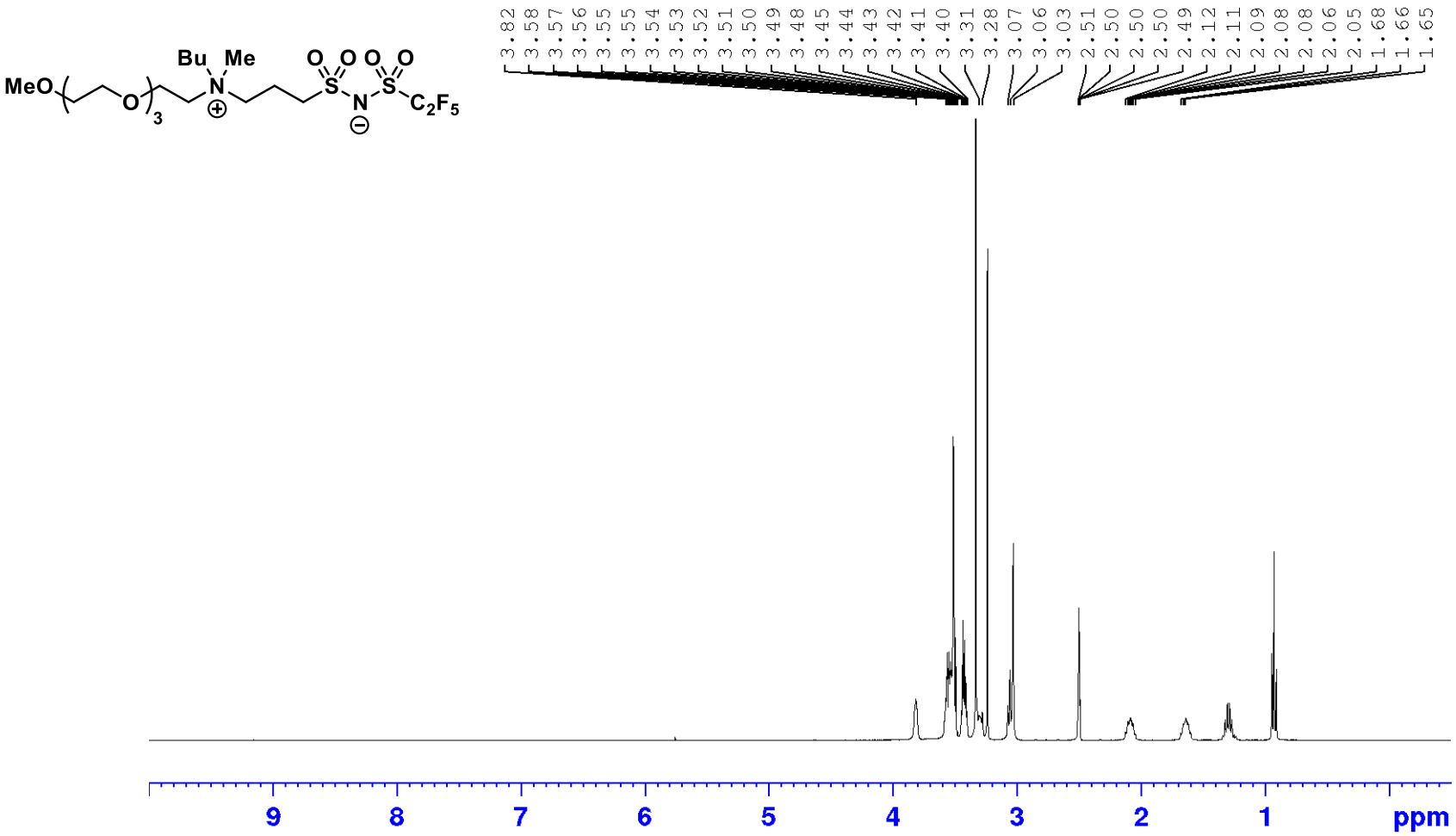
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	5	2	7	2	0
Charge:	+1	Max	400	1000	5	2	8	2	1
DBE:	-99.0 - 999.0								

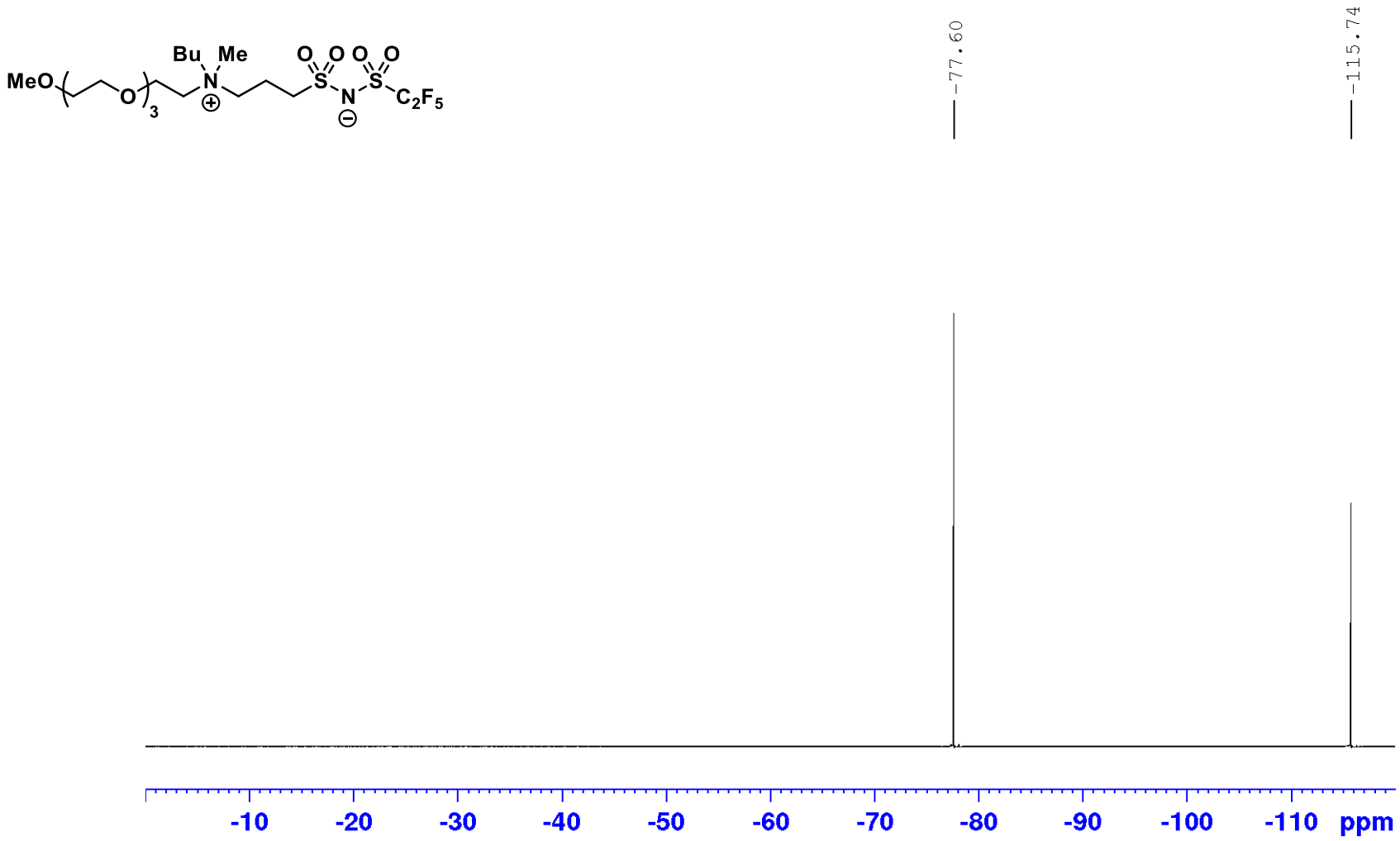
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
537.17141	C17 H34 N2 O7 F5 S2	537.17221	-0.80	-1.48	-0.5
559.15332	C17 H33 N2 O7 F5 Na S2	559.15415	-0.84	-1.50	-0.5

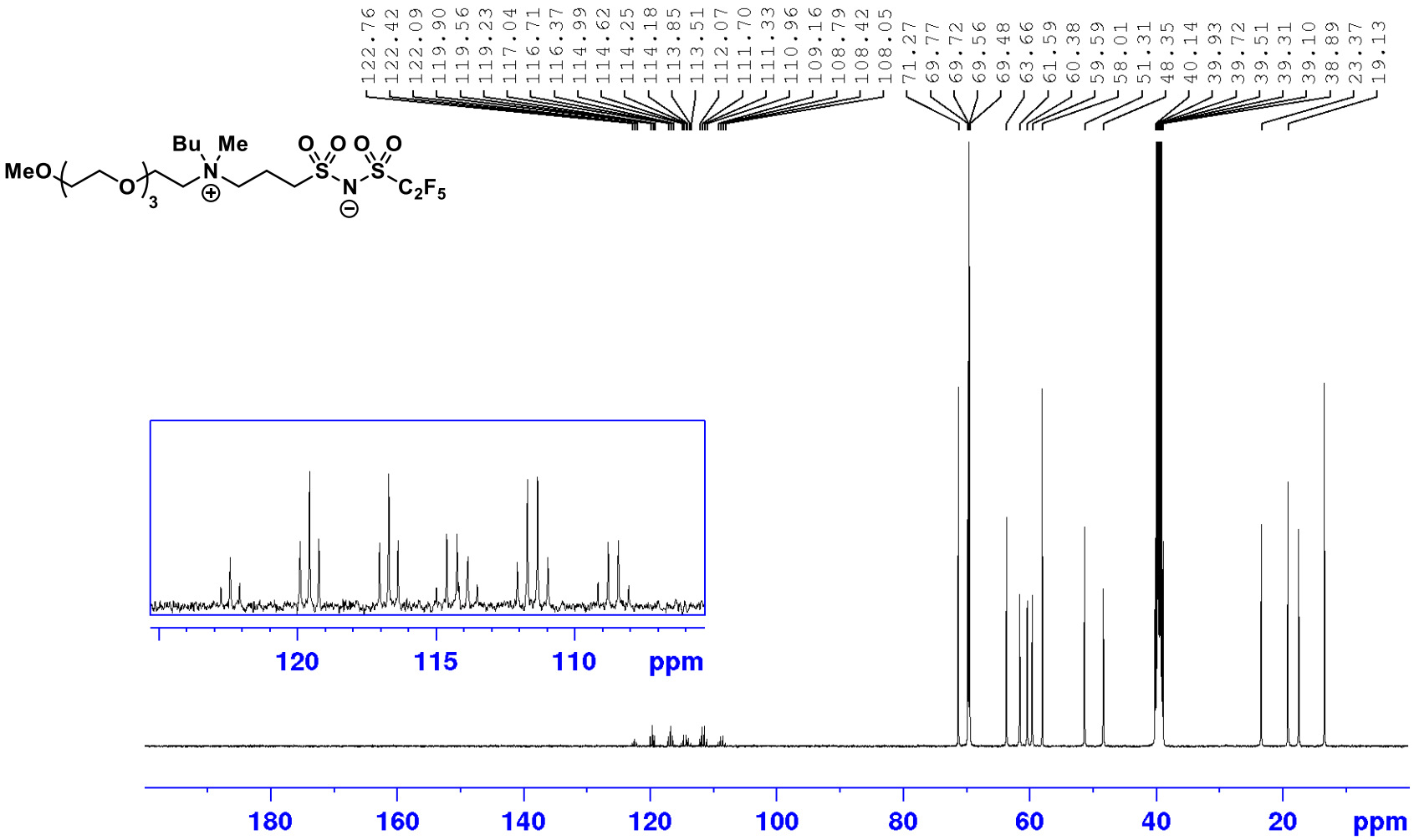
^{1}H NMR spectrum of ZIL 4c



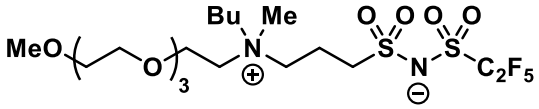
^{19}F NMR spectrum of ZIL 4c



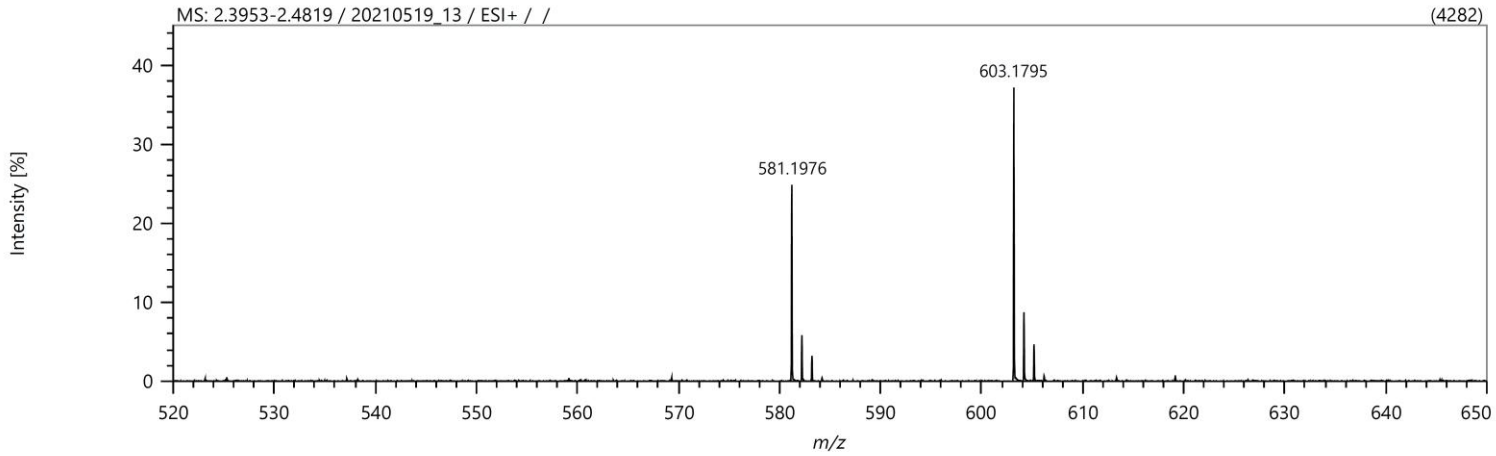
^{13}C NMR spectrum of ZIL 4c



Mass spectrum of ZIL 4c



Spectrum



Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	F	N	O	S	Na
Electron:	Odd/Even	Min	0	0	5	2	7	2	0
Charge:	+1	Max	400	1000	5	2	8	2	1
DBE:	-99.0 - 999.0								

Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
581.19756	C19 H38 N2 O8 F5 S2	581.19843	-0.87	-1.49	-0.5
603.17952	C19 H37 N2 O8 F5 Na S2	603.18037	-0.85	-1.41	-0.5