Electronic Supplementary Material (ESI) for RSC Advances.

Long-Term thermal stabilities of ammonium ionic liquids designed as potential absorbents of ammonia†

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Supporting Information

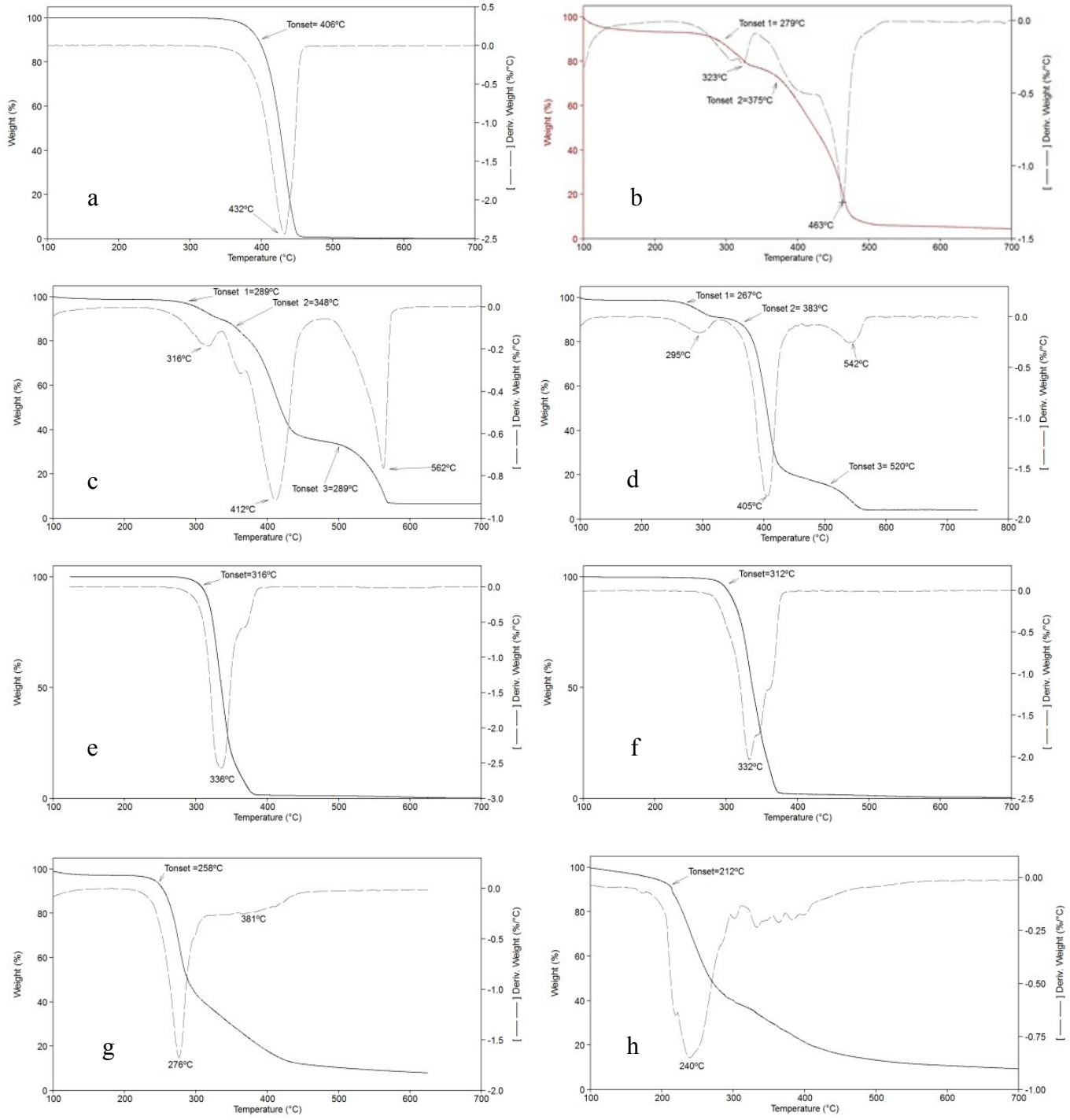


Figure S1 TG and DTG curves of the synthesized ILs. (a) $[EM_2NCH_2CH_2OH][NTf_2]$, (**b**) $[EMN(CH_2CH_2OH)_2][NTf_2]$, (c) $[EM_2NCH_2CH_2OH][OTf]$, (d) $[EMN(CH_2CH_2OH)_2][OTf]$, (e) $[EM_2NCH_2CH_2OH][DCA]$, (f) $[EMN(CH_2CH_2OH)_2][DCA]$, (g) $[EM_2NCH_2CH_2OH][FAP]$, (h) $[EMN(CH_2CH_2OH)_2][FAP]$.

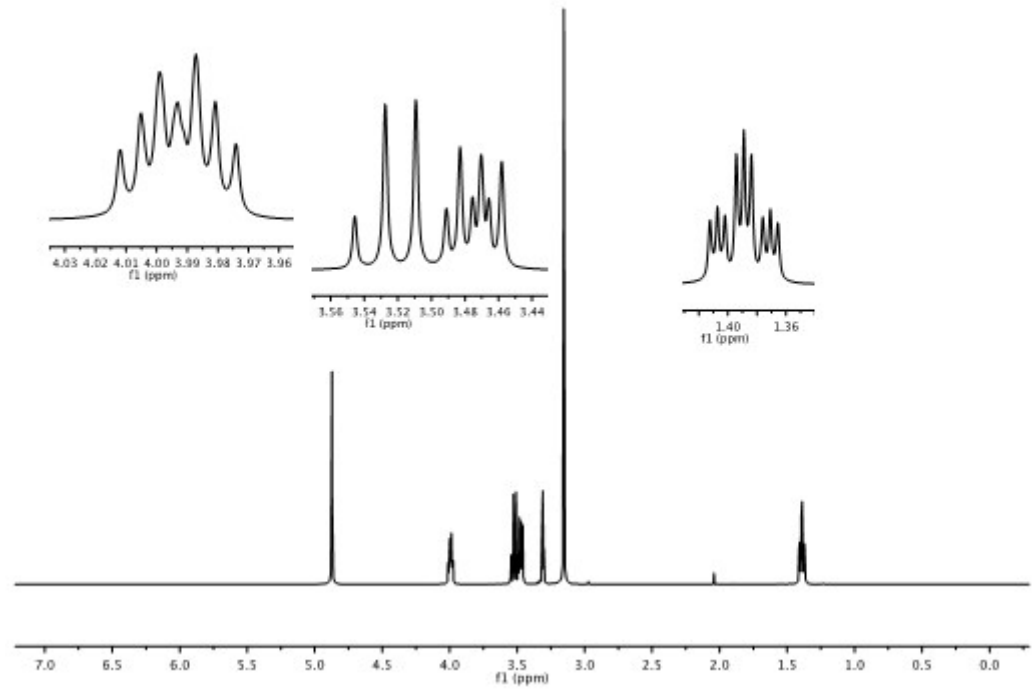
Synthesis

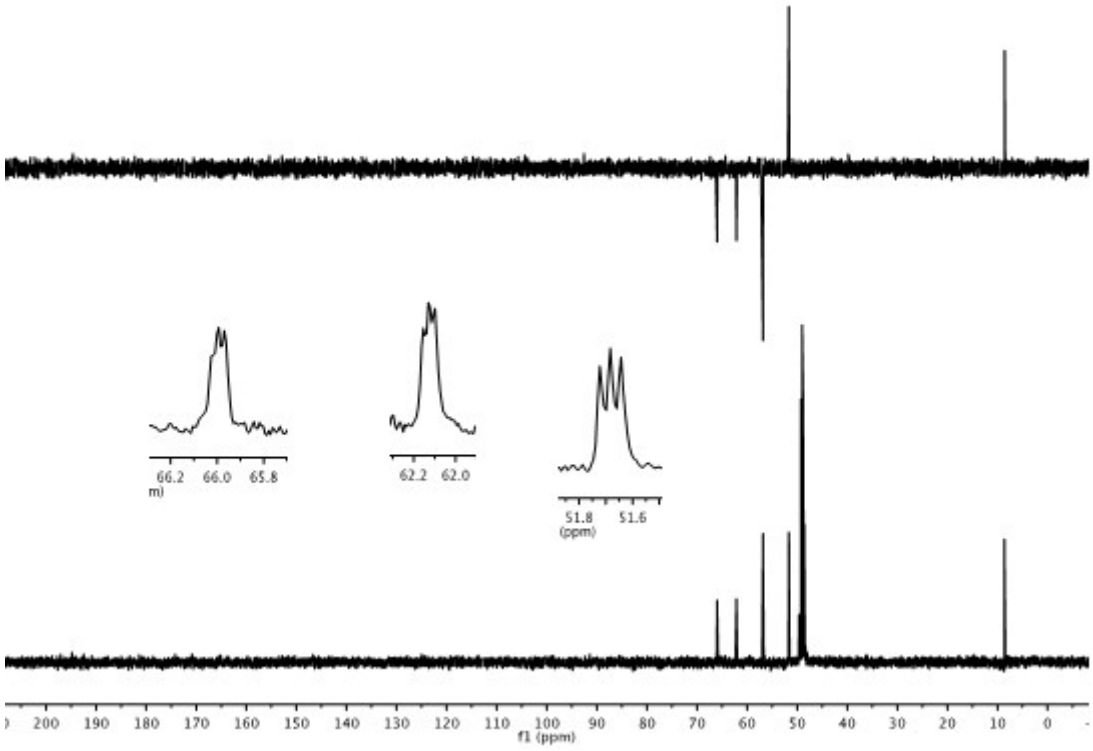
Bis(trifluoromethane)sulfonimide lithium salt (99.0%) was purchased from Sigma Aldrich. *N*-methyldiethanolamine (99.0%), *N,N*-dimethylethanolamine (99.0%), bromoethane (98.0%), silver nitrate (99.5%), sodium dicyanamide (97.0%) and trifluoromethanesulfonic acid (99.0%) were received from Acros Organics. Potassium hydroxide (99%) was purchased from Vorquimica S.L. (99%). Dichloromethane (≥99.9%), acetonitrile (≥99,9%), methanol (≥99,9%), diethyl ether (≥99.9%) and potassium tris(pentafluoroethyl) trifluorophosphate (99.0%) were obtained from Merck. Solvents were dried with suitable drying agents and distiled under Ar. The NMR (400 MHz) analysis of the obtained ILs confirmed impurity levels lower than 1 % (mole fraction) in all cases.

Preparative thin layer chromatography (TLC) was carried out on Aluminium sheets (TLC Silica gel 60 F_{254}) from Merck KGaA to follow the progress of the reactions. The chromatograms were developed in mixtures of methanol and dichloromethane or ethyl acetate and hexane in different proportions. Spots were visualized by UV light (254 nm) and developed using iodine (I_2).
The ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker ARX at 400.1621, 100.6314 and 376.5266 MHz respectively. CD_3OD (Sigma Aldrich, 99.8 %D) and $(CD_3)_2CO$ (Acros Organics, 99.8 %D) were used as solvents. Chemical shifts are given in parts per million and coupling constants (*J*) in hertz. MestReNova (8.0.2) was used for data interpretation. Electrospray MS spectra were recorded on a Bruker FTMS apex-Qe spectrometer.

General procedure for the synthesis of tetraalkylammonium bromides (1 and 6)
Bromoethane (1.1 equiv.) was added dropwise over a stirred solution of *N,N*-dimethylethanolamine or *N,N*-di-(2-hydroxyethyl)-*N*-methylamine in dry acetonitrile under Ar atmosphere. The reaction mixture was allowed to stir under reflux at 80 ºC until completion of the reaction as indicated by TLC (AcOEt-Hexane, 3:1). The reaction mixture was then cooled at 0 ºC. The residue was filtered in vacuum, washed with diethyl ether or ethyl acetate (3 x 20 mL) and remaining solvent was removed by heating under reduced pressure. The desired tetraalkylammonium bromides **1** and **6** were obtained as white solids of very low melting point.

***N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium bromide [$EM_2NCH_2CH_2OH$][Br] (1)**
Following the general procedure, *N,N*-dimethylethanolamine (15.00 mL, 147.60 mmol), and bromoethane (12.12 mL, 162.36 mmol) were reacted in dry acetonitrile (40 mL). Yield 96%. ^{1}H NMR (CD_3OD): δ 1.39 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.1 Hz, $NCH_2C\underline{H_3}$), 3.15 (s, 6H, $N(C\underline{H_3})_2$), 3.44-3.49 (m, 2H, $NC\underline{H_2}CH_2OH$), 3.52 (q, 2H, J_{HH} = 7.3 Hz, $NC\underline{H_2}CH_3$), 3.87-4.10 (m, 2H, $NCH_2C\underline{H_2}OH$). ^{13}C NMR (CD_3OD): 8.6 ($NCH_2\underline{C}H_3$), 51.7 (t, J_{CN} = 4.1 Hz, $N(\underline{C}H_3)_2$), 56.8 ($NCH_2\underline{C}H_2OH$), 61.2-62.8 (m, $N\underline{C}H_2CH_3$), 65.5-66.6 (m, $N\underline{C}H_2CH_2OH$). Electrospray MS *m/z* (%) 315.16397 $[(C_6H_{16}NO)_2(Br)]^+$ ($C_{12}H_{32}BrN_2O_2$ requires 315.16417, 100).

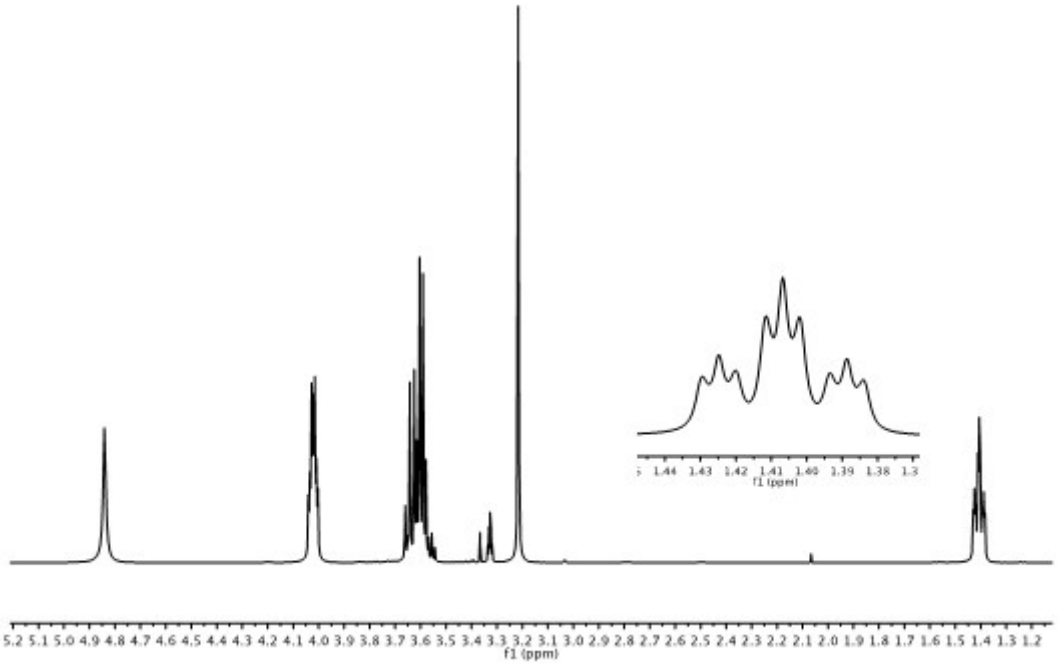


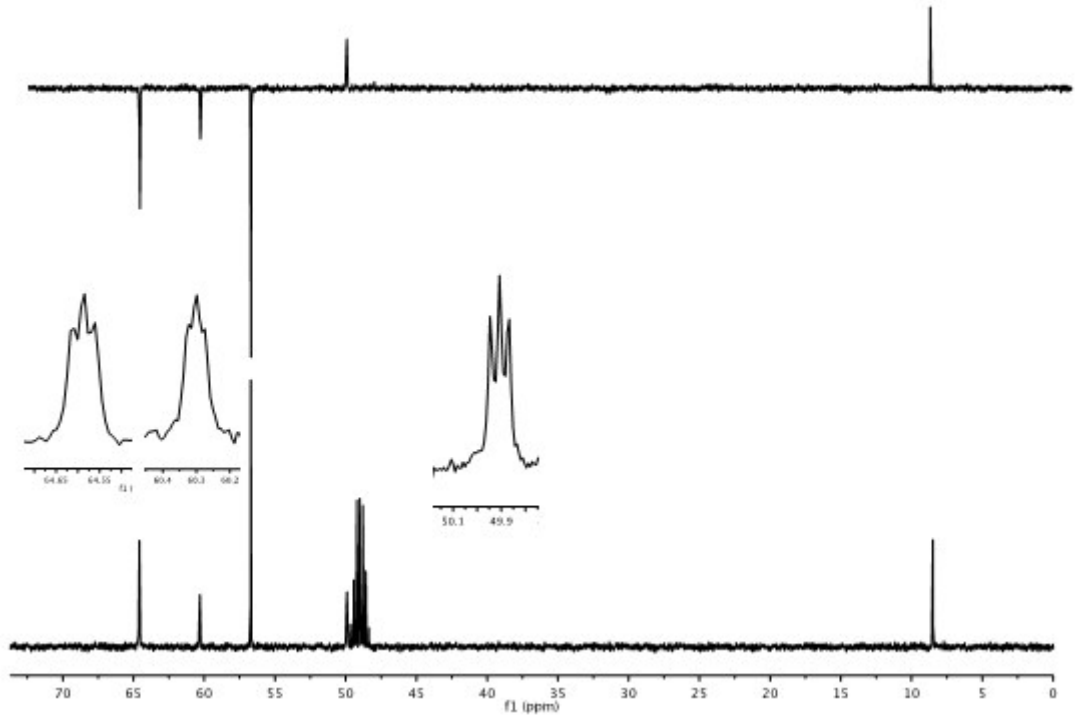


***N*-ethyl-*N*,*N*-di-(2-hydroxyethyl)-*N*-methylammonium bromide [EMN(CH_2CH_2OH)$_2$][Br] (6)**

Following the general procedure, *N,N*-di-(2-hydroxyethyl)-*N*-methylamine (4.00 mL, 34.84 mmol), and bromoethane (2.92 mL, 38.33 mmol) were treated in dry acetonitrile (15 mL). Yield 92%. ^{1}H NMR (CD_3OD): δ 1.41 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.0 Hz, $NCH_2C\underline{H_3}$), 3.22 (s, 3H, $N(C\underline{H_3})$), 3.54-3.67 (m, 6H, 2x($NC\underline{H_2}CH_2OH$), $NC\underline{H_2}CH_3$), 3.99-4.05 (m, 4H, 2x($NCH_2C\underline{H_2}OH$)). ^{13}C NMR (CD_3OD): 8.5 ($NCH_2\underline{C}H_3$), 49.9 (t, J_{CN} = 4.0 Hz, $N(\underline{C}H_3)$), 56.7 ($NCH_2\underline{C}H_2OH$), 60.3 (t, J_{CN} = 2.5 Hz, $N\underline{C}H_2CH_3$), 64.6 (t, J_{CN} = 2.7 Hz,

N$\underline{C}$H$_2$CH$_2$OH). Electrospray MS *m/z* (%) 375.18502 $[(C_7H_{18}NO_2)_2(Br)]^+$ ($C_{14}H_{36}BrN_2O_4$ requires 375.18530, 100).



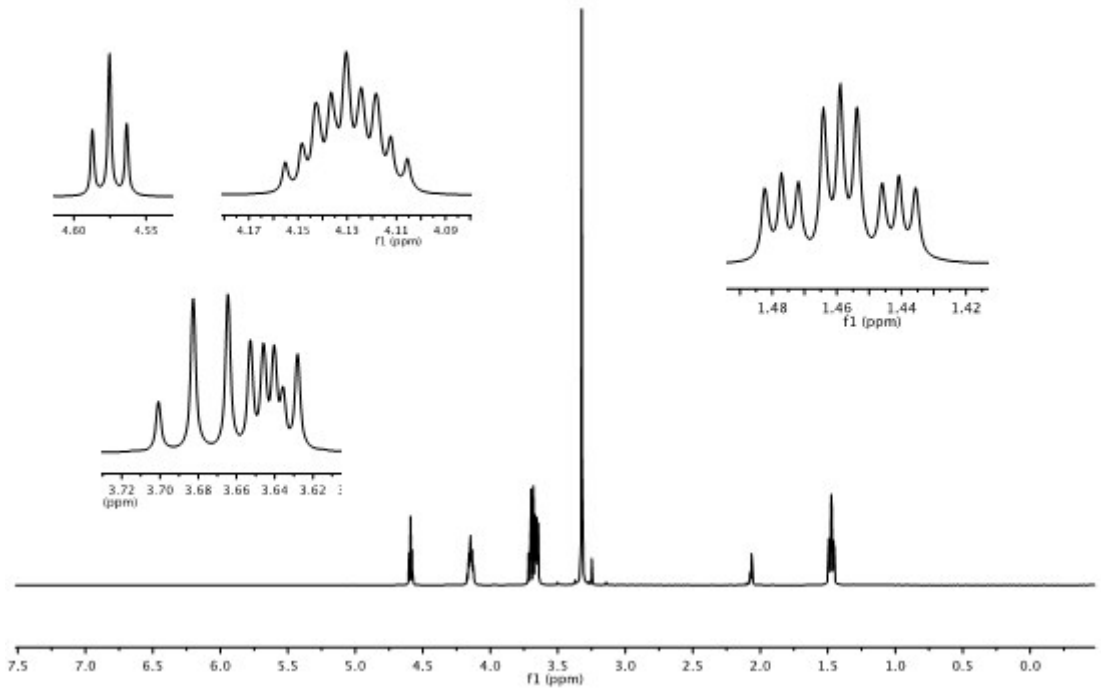


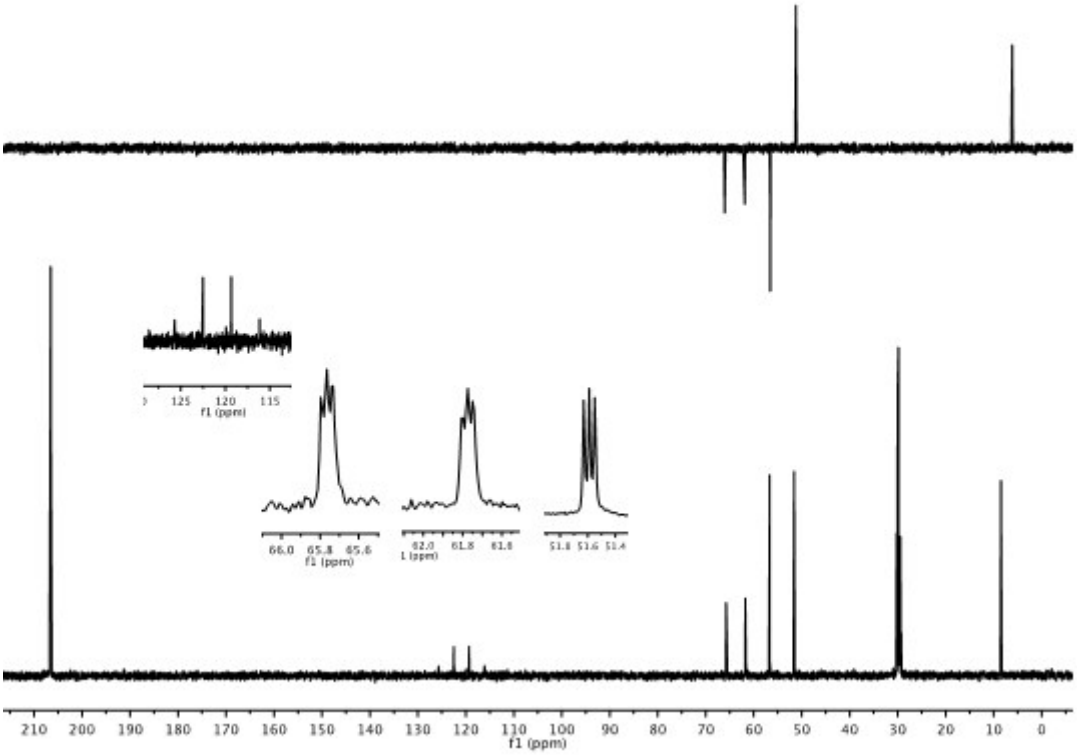
General procedure for the synthesis of tetraalkylammonium bis(trifluoromethanesulfonyl)imides (2 and 7)

The earlier prepared tetraalkylammonium bromides **1** or **6** were dissolved in water or MeOH respectively, and a solution of $LiNTf_2$ (1.1 equiv.) in distilled water or MeOH was added. The reaction mixtures were stirred at r. t. overnight. Isolation of IL **2**: after removal of the solvent, the obtained residue was washed several times with small portions of distilled water until no bromide was indicated by adding a few drops of $AgNO_3$ solution to the last residual washing water portion. The remaining solvent was removed by heating under reduced pressure. Isolation of IL **7**: the obtained residue was dissolved in acetone, a few drops of Et_2O were added and the solution was kept at -18 ºC for several hours; most of the LiBr was then eliminated by filtration and the low amount of still remaining salt was removed by applying Solid Phase Extraction.[c] The remaining solvent was removed by heating under reduced pressure. The desired ILs **2** and **7** were finally dried under vacuum (2 x 10^{-1} Pa).

***N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium bis(trifluoromethanesulfonyl)imide $[EM_2NCH_2CH_2OH][NTf_2]$ (2)**

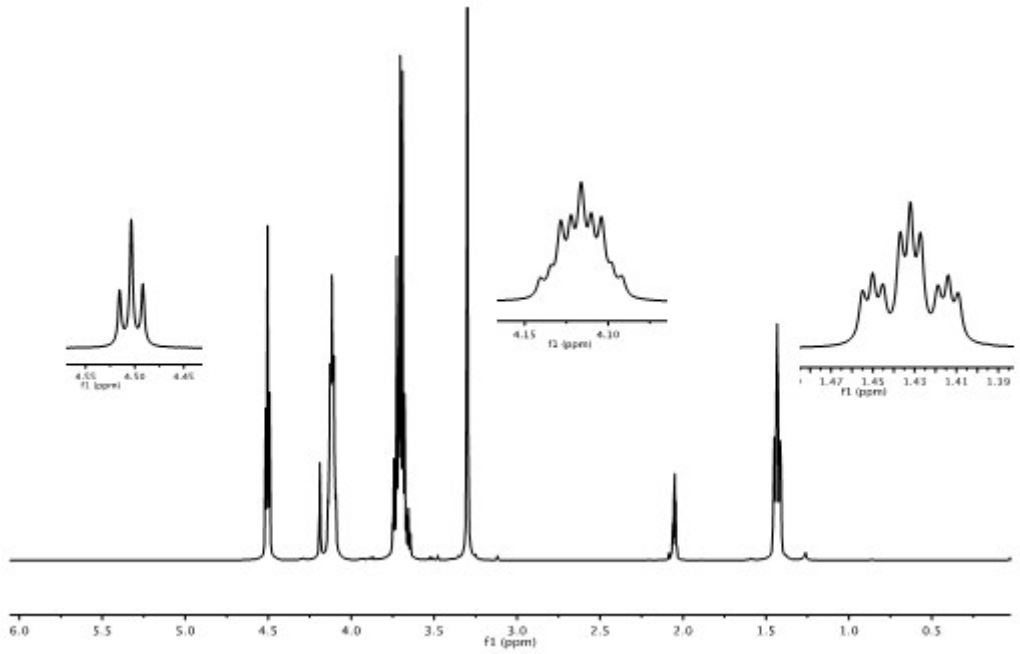
Following the general procedure, *N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium bromide (**1**, 11.00 g, 55.53 mmol), and $LiNTf_2$ salt (17.53 g, 61.08 mmol) were stirred in water (30 mL). Yield 90%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.46 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.1 Hz, $NCH_2C\underline{H_3}$), 3.31 (s, 6H, $N(C\underline{H_3})_2$), 3.61-3.72 (m, 4H, $NC\underline{H_2}CH_2OH$, $NC\underline{H_2}CH_3$), 4.08-4.18 (m, 2H, $NCH_2C\underline{H_2}OH$), 4.58 (t, 1H, J_{HH} = 4.8 Hz, $NCH_2CH_2\underline{OH}$). ^{13}C NMR ($(CD_3)_2CO$): 8.5 ($NCH_2\underline{C}H_3$), 51.6 (t, J_{CN} = 4.0 Hz, $N(\underline{C}H_3)_2$), 56.7 ($NCH_2\underline{C}H_2OH$), 59.9-62.7 (m, $N\underline{C}H_2CH_3$), 64.1-66.7 (m, $N\underline{C}H_2CH_2OH$), 120.9 (q, J_{CF} = 321.1 Hz, $N(SO_2\underline{C}F_3)_2$). ^{19}F NMR ($(CD_3)_2CO$): δ -79.90. Electrospray MS *m/z* (%) 516.16249 $[(C_6H_{16}NO)_2(N(SO_2CF_3)_2)]^+$ ($C_{14}H_{32}F_6N_3O_6S_2$ requires 516.16312, 100).

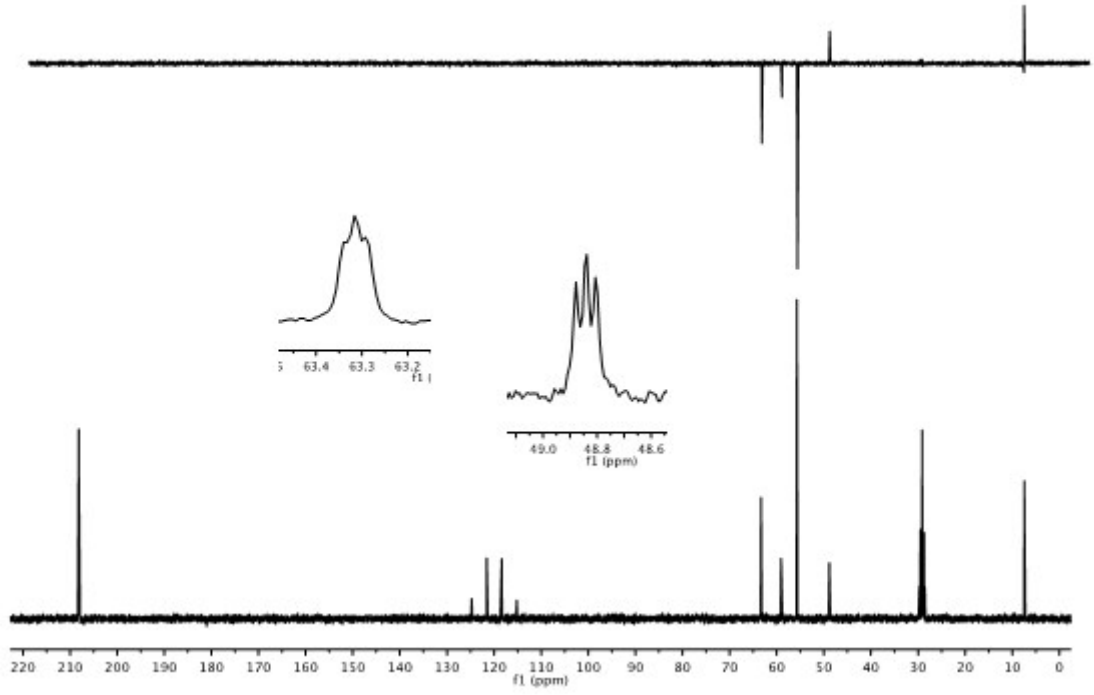




_N_-ethyl-_N,N_-di-(2-hydroxyethyl)-_N_-methylammonium bis(trifluoromethanesulfonyl)imide [EMN(CH_2CH_2OH)$_2$][NTf_2] (7)

Following the general procedure, N-ethyl-N,N-di-(2-hydroxyethyl)-N-methylammonium bromide (**6**, 1.00 g, 4.38 mmol), and $LiNTf_2$ salt (1.38 g, 4.82 mmol) were stirred in MeOH (10 mL) overnight. Yield 91%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.43 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 3.8 Hz, $NCH_2C\underline{H_3}$), 3.30 (s, 3H, $N(C\underline{H_3})$), 3.63-3.76 (m, 6H, 2x($NC\underline{H_2}CH_2OH$), $NC\underline{H_2}CH_3$), 4.07-4.16 (m, 4H, 2x($NCH_2C\underline{H_2}OH$)), 4.50 (t, 2H, J_{HH} = 4.7 Hz, 2x($NCH_2CH_2O\underline{H}$)). ^{13}C NMR ($(CD_3)_2CO$): δ 8.2 ($NCH_2\underline{C}H_3$), 49.6 (t, J_{CN} = 4.0 Hz, $N(\underline{C}H_3)$), 56.4 $NCH_2\underline{C}H_2OH$), 59.8 ($N\underline{C}H_2CH_3$), 64.0 (t, J_{CN} = 2.8 Hz, $N\underline{C}H_2CH_2OH$), 120.7 (q, J_{CF} = 321.0 Hz, $N(SO_2\underline{C}F_3)_2$). ^{19}F NMR ($(CD_3)_2CO$): δ -79.95. Electrospray MS *m/z* (%) 576.18350 $[(C_7H_{18}NO_2)_2(N(SO_2CF_3)_2)]^+$ ($C_{16}H_{36}F_6N_3O_8S_2$ requires 576.18425 , 100).



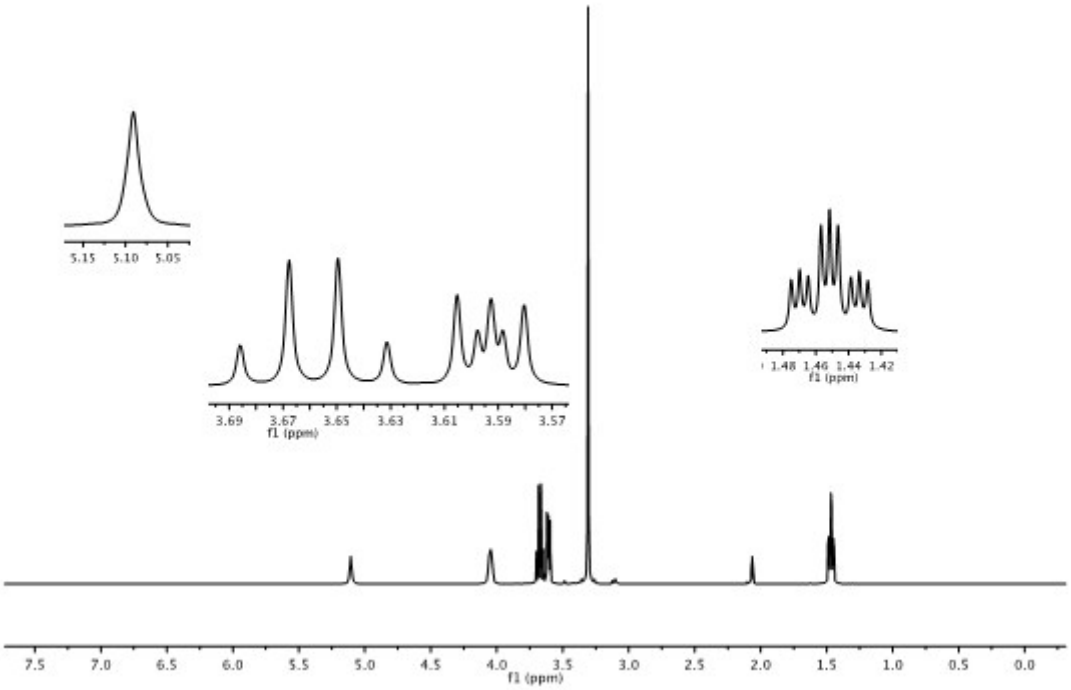


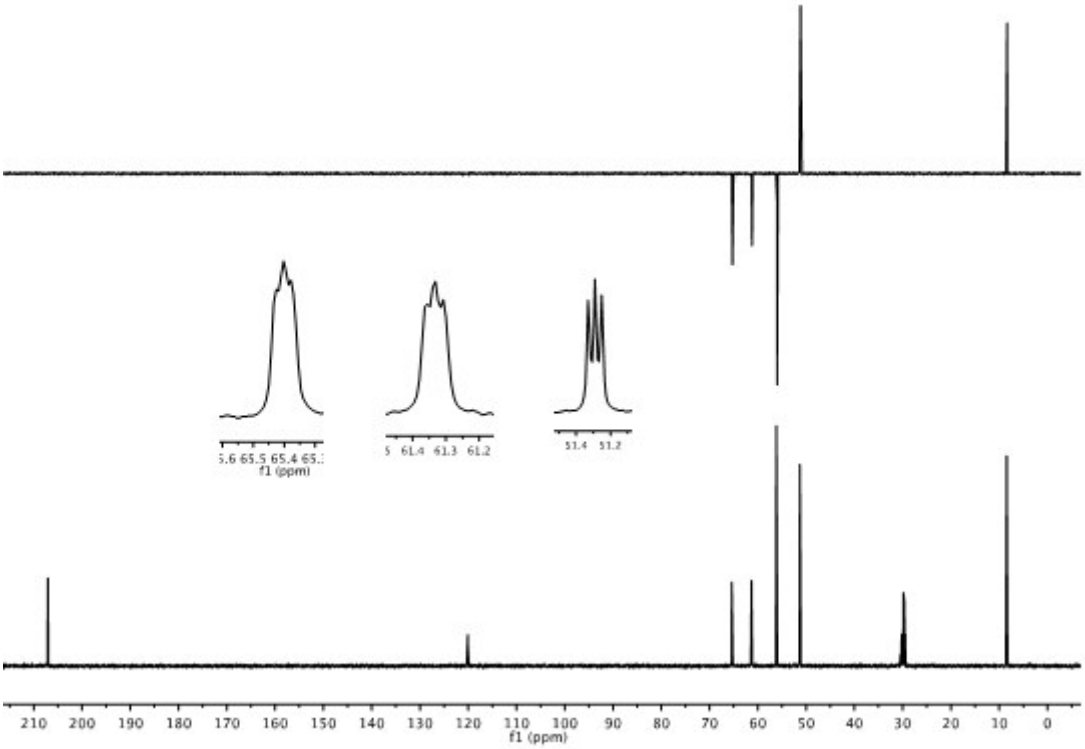
General procedure for the synthesis of tetraalkylammonium dicyanamides (3 and 8)

A solution of silver nitrate in water was added over a solution of sodium dicyanamide (1 equiv.) in water. The reaction mixture was stirred at r. t. in the dark for 1 h. The obtained white solid was filtrated, washed with water (3 x 10 mL) and dried under high vacuum for 2 h affording $AgN(CN)_2$. A stoichiometric amount of the earlier prepared tetraalkylammonium bromides **1** and **6** were dissolved in water and added over a suspension of $AgN(CN)_2$ (1 equiv.) in water. The resulting mixture was stirred at r. t. in the dark for 4 h. After removing the the AgBr precipitated, the filtrate was concentrated by rotary evaporation and dried by heating at 60 ºC under high vacuum for 12 h.

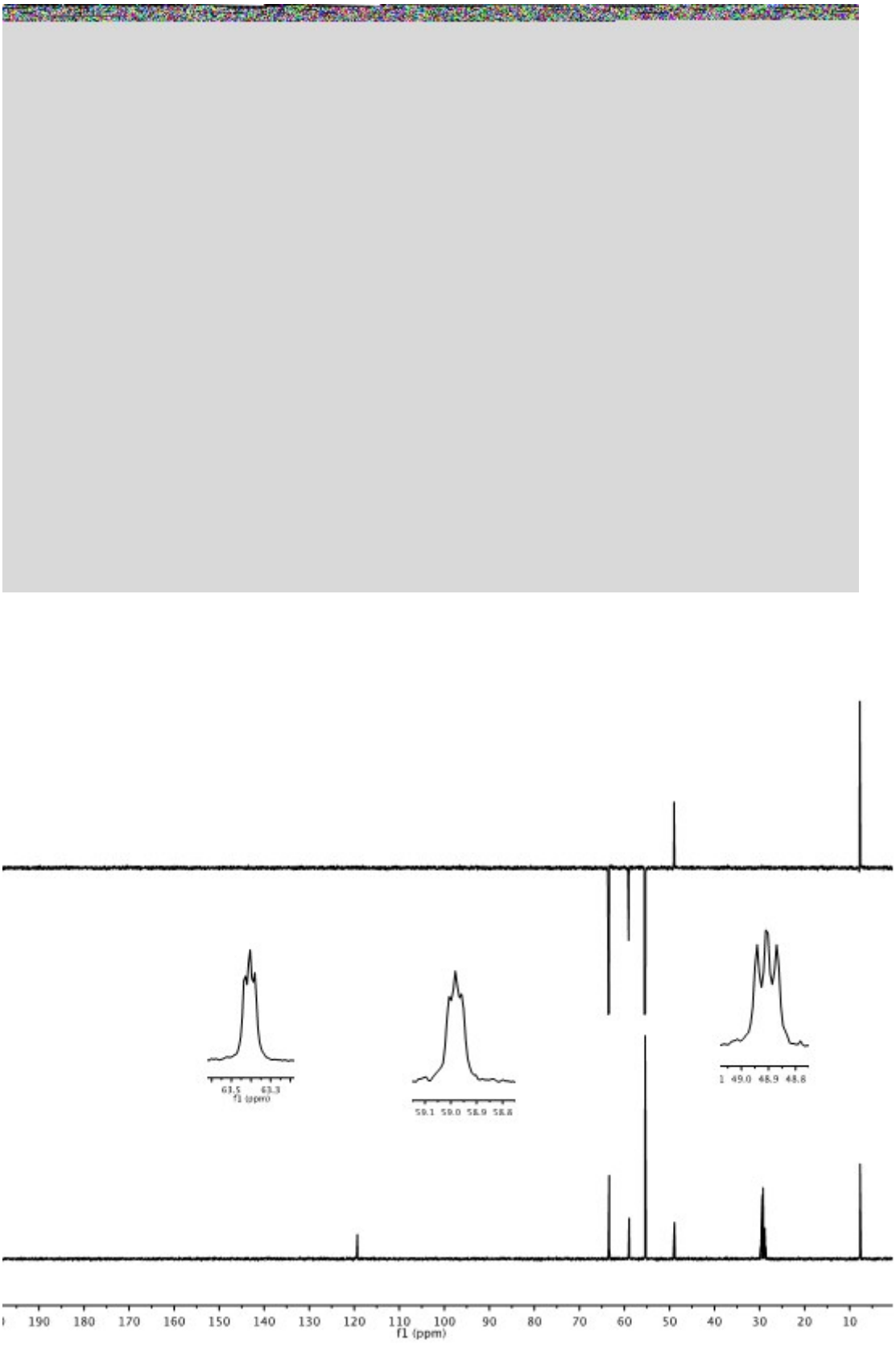
***N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium dicyanamide [$EM_2NCH_2CH_2OH$][DCA] (3)**

Following the general procedure, silver nitrate (21.39 g, 125.30 mmol) and sodium dicyanamide (11.50 g, 125.30 mmol) in water (40 mL) were reacted to afford silver dicyanamide. Yield 97%. Silver dicyanamide (20.80 g, 119.60 mmol) and *N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium bromide (**1**, 23.70 g, 119.60 mmol) were reacted in water (40 mL). Yield 91%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.45 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.1 Hz, $NCH_2C\underline{H_3}$), 3.29 (s, 6H, $N(C\underline{H_3})_2$), 3.52-3.62 (m, 2H, $NC\underline{H_2}CH_2OH$) 3.66 (q, J_{HH} = 7.3 Hz, $NC\underline{H_2}CH_3$), 3.96-4.10 (m, 2H, $NCH_2C\underline{H_2}OH$), 5.09 (broad s, 1H, $NCH_2CH_2\underline{OH}$). ^{13}C NMR ($(CD_3)_2CO$): δ 8.5 ($NCH_2\underline{C}H_3$), 51.3 (t, J_{CN} = 4.0 Hz, $N(\underline{C}H_3)_2$), 56.1 ($NCH_2\underline{C}H_2OH$), 61.06-61.44 (m, $N\underline{C}H_2CH_3$), 65.28-65.47 (m, $N\underline{C}H_2CH_2OH$), 120.1 ($N(\underline{C}N)_2$). Electrospray MS *m/z* (%) 302.25506 $[(C_6H_{16}NO)_2(N(CN)_2)]^+$ ($C_{14}H_{32}N_5O_2$ requires 302.25505, 100).





***N*-ethyl-*N*,*N*-di-(2-hydroxyethyl)-*N*-methylammonium dicyanamide [EMN(CH_2CH_2OH)$_2$][DCA] (8)**
Following the general procedure, silver nitrate (1.30 g, 7.63 mmol) and sodium dicyanamide (0.70 g, 7.63 mmol) in water (10 mL) were reacted to afford silver dicyanamide. Yield 98%. Silver dicyanamide (1.14 g, 6.57 mmol) and *N*-ethyl-*N*,*N*-di-(2-hydroxyethyl)-*N*-methylammonium bromide (**6**, 1.50 g, 6.57 mmol) were reacted in water (10 mL). Yield 98%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.46 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.0 Hz, $NCH_2C\underline{H_3}$), 3.32 (s, 3H, $N(C\underline{H_3})$), 3.60-3.80 (m, 6H, $2x(NC\underline{H_2}CH_2OH)$, $NC\underline{H_2}CH_3$), 3.98-4.15 (m, 4H, $2x(NCH_2C\underline{H_2}OH)$), 4.95 (t, 2H, J_{HH} = 4.9 Hz, $2x(NCH_2CH_2O\underline{H})$). ^{13}C NMR ($(CD_3)_2CO$): δ 7.7 ($NCH_2\underline{C}H_3$), 48.9 (t, J_{CN} = 3.4 Hz, $N(\underline{C}H_3)$), 55.4 ($NCH_2\underline{C}H_2OH$), 59.0 (t, J_{CN} = 2.6 Hz, $N\underline{C}H_2CH_3$), 63.4 (t, J_{CN} = 2.7 Hz, $N\underline{C}H_2CH_2OH$), 119.3 ($N(\underline{C}N)_2$). Electrospray MS *m/z* (%) 362.27611 $[(C_7H_{18}NO_2)_2((N(CN)_2)]^+$ ($C_{16}H_{36}N_5O_4$ requires 362.27618, 100)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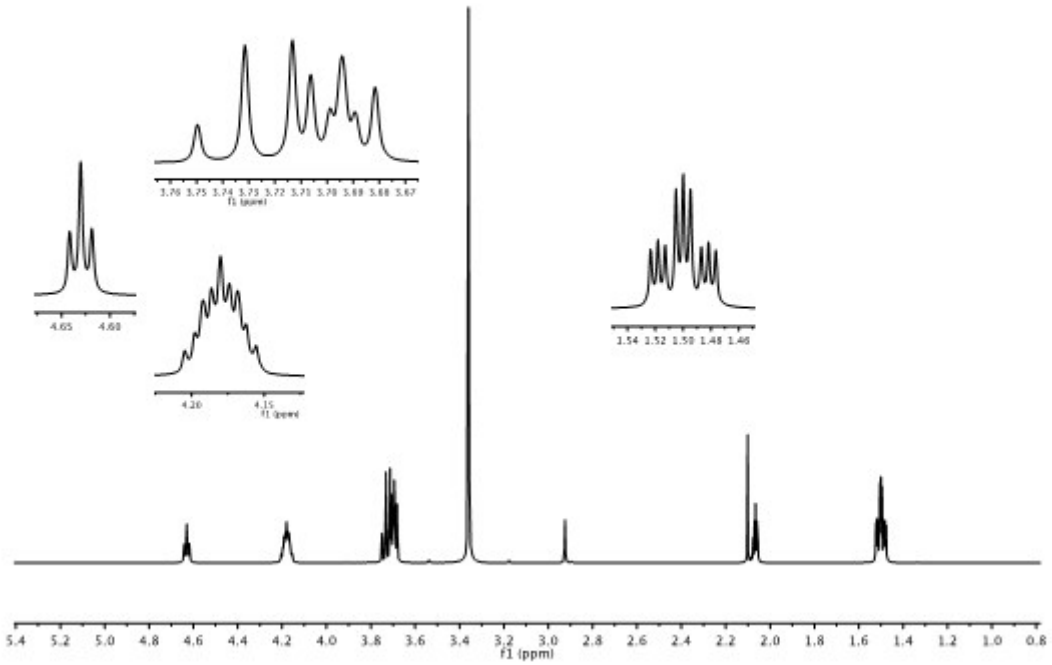
f1 (ppm)

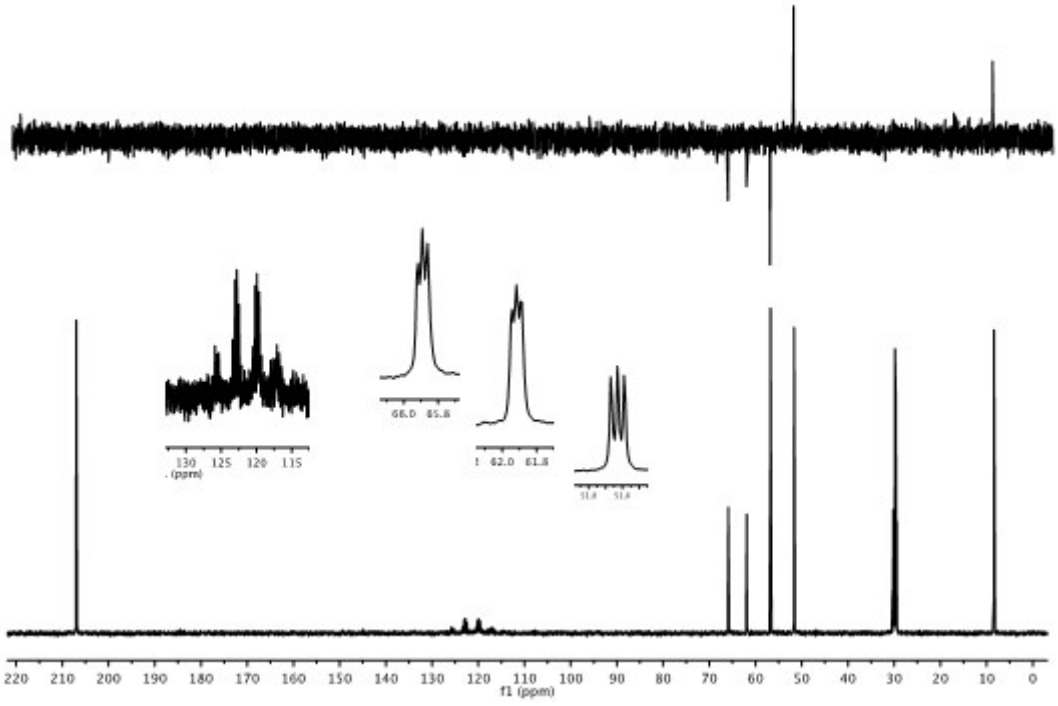
General procedure for the synthesis of tetraalkylammonium tris(pentafluoroethyl)methanesulfonates (4 and 9)

The earlier prepared tetraalkylammonium bromides **1** and **6** were dissolved in distilled water. Then, a water solution of potassium tris(pentafluoroethyl)methanesulfonate (1 equiv.) was added by one portion. The reaction mixtures continued to stir at r. t. for 1 h. The residue was dissolved in dichloromethane and washed several times with small portions of distilled water until no bromide was indicated by adding a few drops of $AgNO_3$ solution to the last residual washing water portion. The organics portions were dried over Na_2SO_4 and filtered. Finally, the solvent was removed on the rotary evaporator and the ILs were dried under vacuum (2 x 10^{-1} Pa).

***N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium tris(pentafluoroethyl)methanesulfonate [$EM_2NCH_2CH_2OH$] [FAP] (4)**

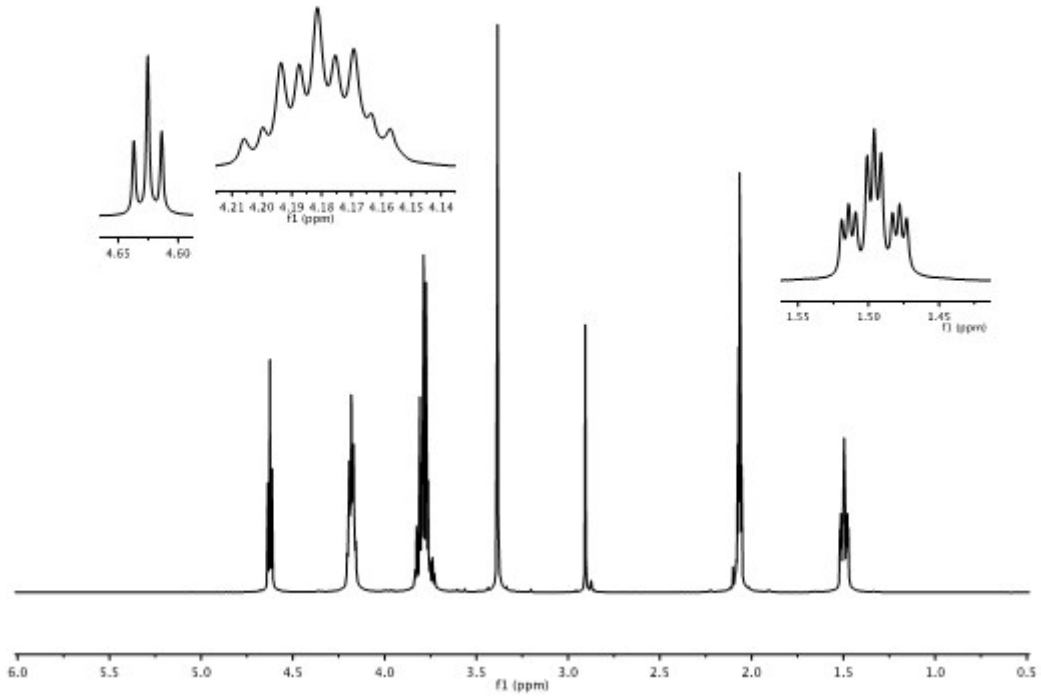
Following the general procedure, tris(pentafluoroethyl)methanesulfonate potassium (0.86 g, 1.80 mmol) and N-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethylammonium bromide (**1**, 0.35 g, 1.80 mmol) were reacted in water (10 mL). Yield 96%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.48 (tt, 3H, J_{HH} = 7.2 Hz, J_{NH} = 2.1 Hz, $NCH_2C\underline{H_3}$), 3.34 (s, 6H, $N(C\underline{H_3})_2$), 3.65-3.75 (m, 4H, $NC\underline{H_2}CH_2OH$, $NC\underline{H_2}CH_3$), 4.12-4.21 (m, 2H, $NCH_2C\underline{H_2}OH$), 4.62 (t, 1H, J_{HH} = 4.6 Hz, $NCH_2CH_2\underline{OH}$). ^{13}C NMR ($(CD_3)_2CO$): δ 8.4 ($NCH_2\underline{C}H_3$), 51.6 (t, J_{CN} = 3.9 Hz, $N(\underline{C}H_3)_2$), 56.8 ($NCH_2\underline{C}H_2OH$), 61.9 (t, J_{CN} = 2.8 Hz, $N\underline{C}H_2CH_3$), 65.9 (t, J_{CN} = 3.0 Hz, $N\underline{C}H_2CH_2OH$), 112.9-128.9 (m, $PF_3(\underline{C}F_2\underline{C}F_3)_3$). ^{19}F NMR ($(CD_3)_2CO$): δ -113.1-(-122.8) (m, $PF_3(\underline{CF_2}CF_3)_3$), -85.0-(-91.3) (m, $\underline{P}F_3(C\underline{F_2}CF_3)_3$), -79.1-(-84.0) (m, $PF_3(CF_2\underline{CF_3})_3$), -42.6-(-46.8) (m, $\underline{PF_3}(CF_2CF_3)_3$). Electrospray MS *m/z* (%) 681.18967 $[(C_6H_{16}NO)_2((CF_3CF_2)_3PF_3)]^+$ ($C_{18}H_{32}F_{18}N_2O_2P$ requires 681.19085, 100).

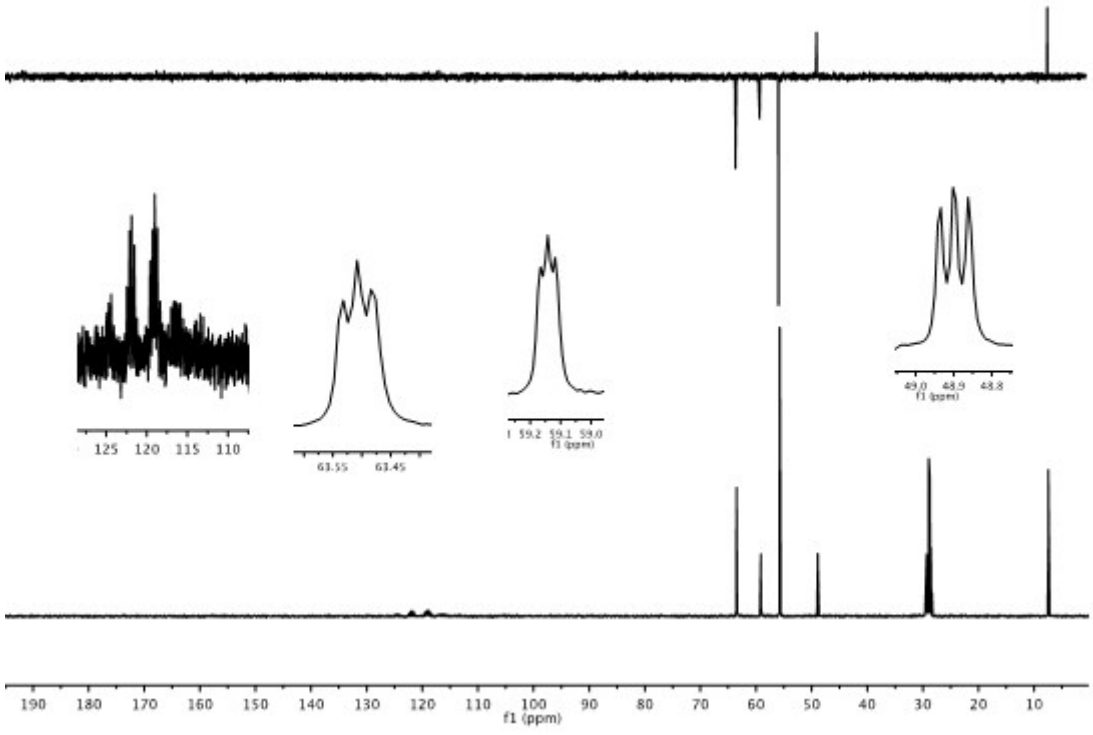




***N*-ethyl-*N*,*N*-di-(2-hydroxyethyl)-*N*-methylammonium tris(pentafluoroethyl)methanesulfonate [EMN(CH_2CH_2OH)$_2$][FAP] (9)**

Following the general procedure, tris(pentafluoroethyl)methanesulfonate potasium (1.50 g, 3.10 mmol) and *N*-ethyl-*N,N*-di-(2-hydroxyethyl)-*N*-methylammonium bromide (**6**, 0.71 g, 3.10 mmol) were reacted in water (10 mL). Yield 88%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.50 (tt, 3H, J_{HH} = 7.2 Hz, J_{NH} = 2.0 Hz, $NCH_2C\underline{H_3}$), 3.39 (s, 3H, $N(C\underline{H_3})$), 3.69-3.87 (m, 6H, $2x(NC\underline{H_2}CH_2OH)$, $NC\underline{H_2}CH_3$), 4.13-4.22 (m, 4H, $2x(NCH_2C\underline{H_2}OH)$), 4.63 (t, 2H, J_{HH} = 5.1 Hz, $2x(NCH_2CH_2O\underline{H})$). ^{13}C NMR ($(CD_3)_2CO$): δ 7.4 ($NCH_2\underline{C}H_3$), 48.9 (t, J_{CN} = 3.8 Hz, $N(\underline{C}H_3)$), 55.8 ($NCH_2\underline{C}H_2OH$), 59.1 (t, J_{CN} = 2.5 Hz, $N\underline{C}H_2CH_3$), 63.5 (t, J_{CN} = 2.7 Hz, $N\underline{C}H_2CH_2OH$), 114.6-126.1 (m, $PF_3(CF_2CF_3)_3$). ^{19}F NMR ($(CD_3)_2CO$): δ -111.8-(-120.8) (m, $PF_3(\underline{CF_2}CF_3)_3$), -85.3-(-90.9) (m, $PF_3(C\underline{F_2}CF_3)_3$), -79.3-(-83.6) (m, $PF_3(CF_2\underline{CF_3})_3$), -40.2-(-48.8) (m, $\underline{PF}_3(CF_2CF_3)_3$). Electrospray MS *m/z* (%) 741.21127 $[(C_7H_{18}NO_2)_2((CF_3CF_2)_3PF_3)]^+$ ($C_{20}H_{36}F_{18}N_2O_4P$ requires 741.21198, 100).





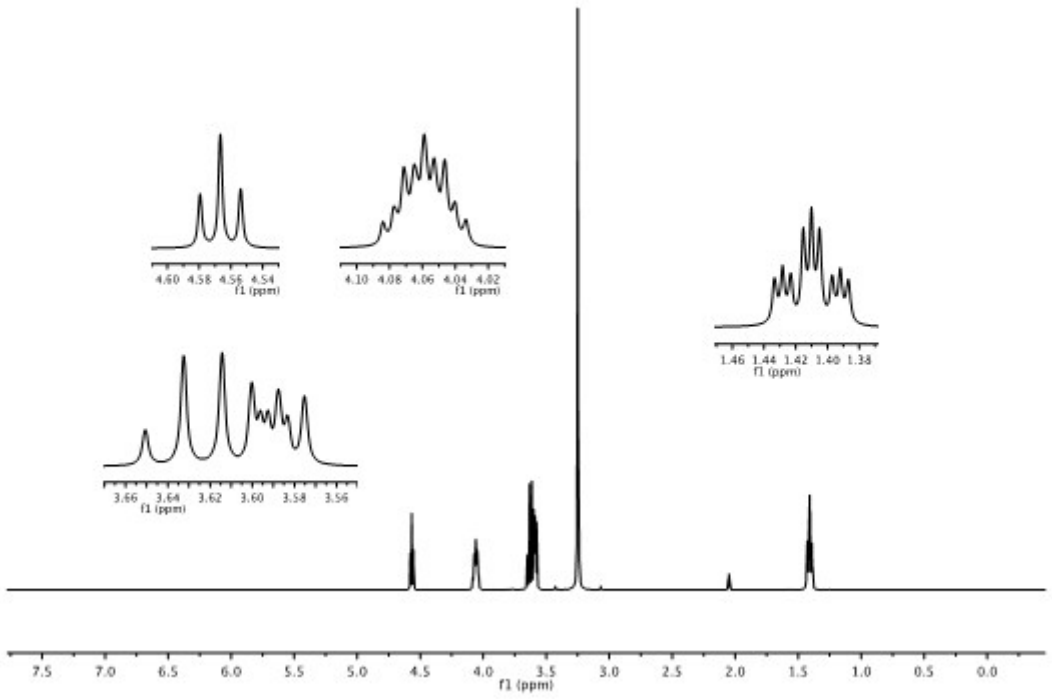
General procedure for the synthesis of tetraalkylammonium trifluoromethanesulfonates (5 and 10)

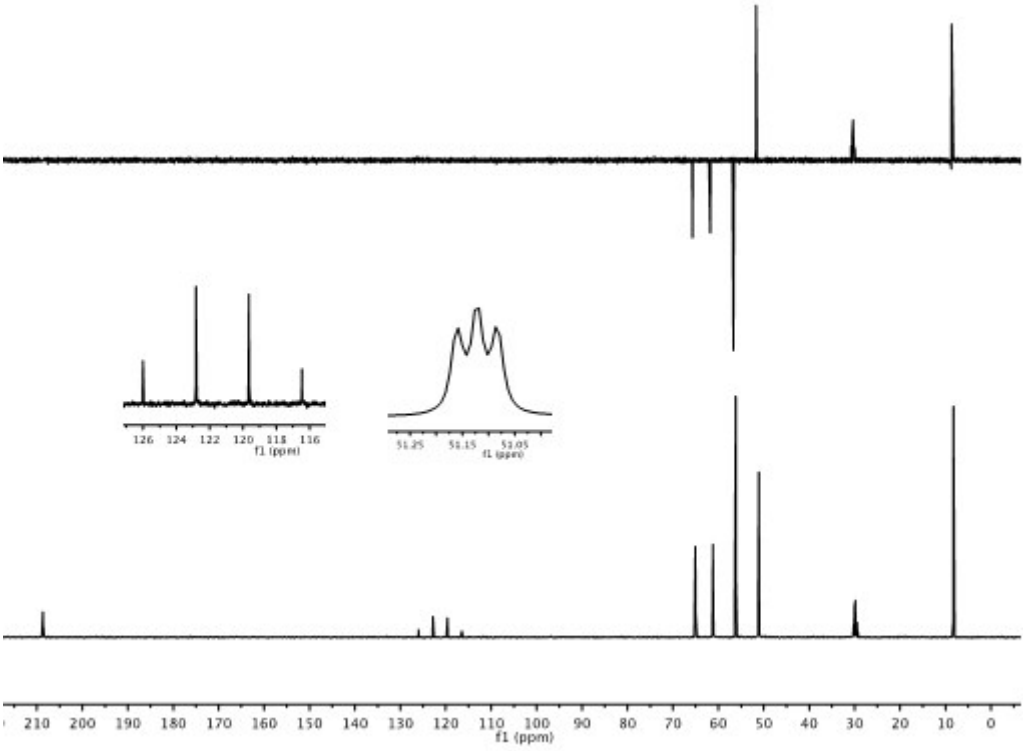
A potassium hydroxide (1 equiv.) solution in dry MeOH was cooled in an ice-water bath and a cold solution of trifluoromethanesulfonic acid (1 equiv.) in dry MeOH was added dropwise under Ar atmosphere. The

resulting mixture was stirred at r. t. for 1 h and a solution of the earlier bromides **1** or **6** (1 equiv.) in dry methanol was added dropwise. The reaction mixture was stirred at r.t. for 5 h. The solvent was then removed by heating under reduced pressure. IL **5** isolation: acetone was added and the resulting mixture was kept at -18 ºC for several hours. The precipitated KBr was eliminated by filtration, the remaining solvent was removed by heating under reduced pressure and the residue was washed with diethyl ether (3 x 15 mL). The IL was re-dissolved in acetone and decolorized by stirring with activated charcoal (charcoal: IL 1:4) at r. t. overnight. After that, the mixture was filtered through Celite. The solvent was removed and the obtained liquid was dried by heating at 50 ºC under high vacuum for 12 h. IL **10** isolation: after removal of the solvent, the obtained residue was dissolved in acetone, a few drops of Et_2O were added and the solution was kept at -18 ºC for several hours; most of the LiBr was then eliminated by filtration and the low amount of still remaining salt was removed by applying Solid Phase Extraction.

_N_-ethyl-_N_-(2-hydroxyethyl)-_N,N_-dimethylammonium trifluoromethanesulfonate [$EM_2NCH_2CH_2OH$][OTf] (5)

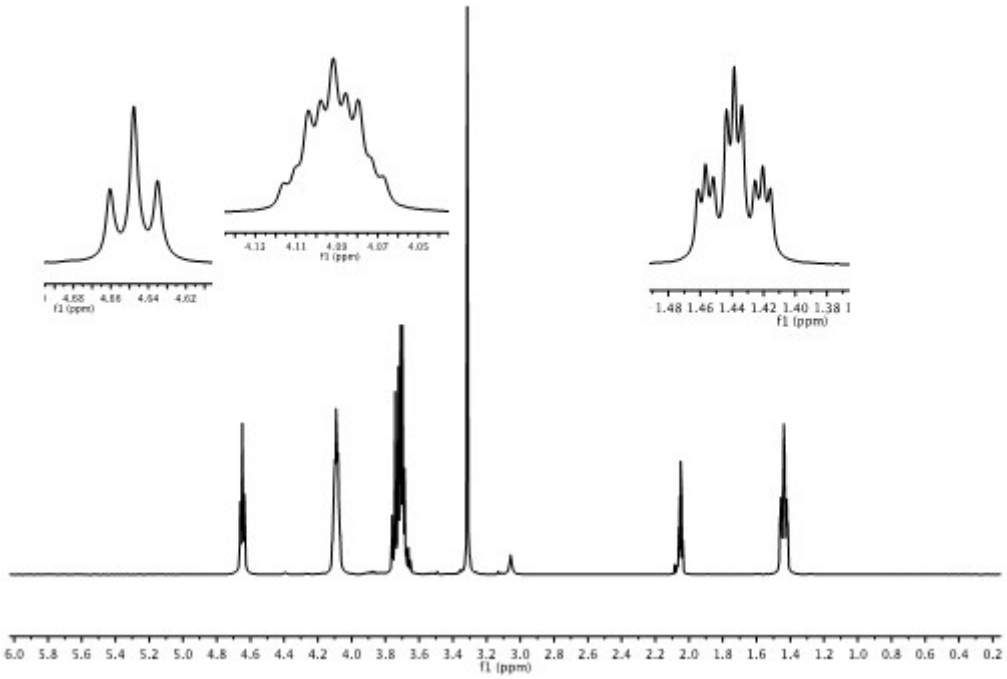
Following the general procedure, potassium hydroxide (4.00 g, 71.11 mmol), trifluoromethanesulfonic acid (7.00 mL, 78.81 mmol) and *N*-ethyl-*N*-(2-hydroxyethyl)-*N,N*-dimethyl ammonium bromide (**1**, 14.09 g, 71.11 mmol) were reacted in dry MeOH (50 mL). Yield 98%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.41 (tt, 3H, J_{HH} = 7.2 Hz, J_{NH} = 2.0 Hz, $NCH_2C\underline{H_3}$), 3.25 (s, 6H, $N(C\underline{H_3})_2$), 3.50-3.70 (m, 4H, $NC\underline{H_2}CH_2OH$, $NC\underline{H_2}CH_3$), 4.01-4.11 (m, 2H, $NCH_2C\underline{H_2}OH$), 4.57 (t, 1H, J_{HH} = 5.1 Hz, $NCH_2CH_2\underline{OH}$). ^{13}C NMR ($(CD_3)_2CO$): δ 8.2 ($NCH_2\underline{C}H_3$), 51.1 (t, J_{CN} = 3.6 Hz, $N(\underline{C}H_3)_2$), 56.2 ($NCH_2\underline{C}H_2OH$), 61.0–61.6 (m, $N\underline{C}H_2CH_3$), 64.9-65.1 (m, $N\underline{C}H_2CH_2OH$), 121.2 (q, J_{CF} = 319.8 Hz, $\underline{C}F_3SO_3$). ^{19}F NMR ($(CD_3)_2CO$): δ -79.06. Electrospray MS *m/z* (%) 385.19746 $[(C_6H_{16}NO)_2(CF_3SO_3)]^+$ ($C_{13}H_{32}F_3N_2O_5S_2$ requires 385.19785, 100).

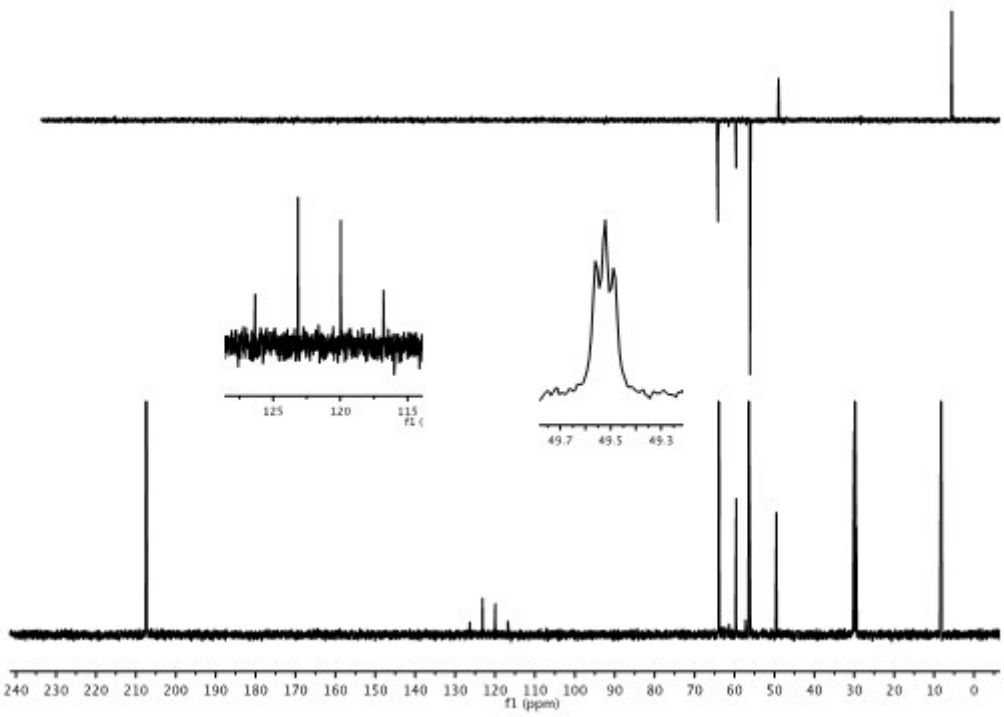




***N*-ethyl-*N,N*-di-(2-hydroxyethyl)-*N*-methylammonium trifluoromethanesulfonate [EMN(CH_2CH_2OH)$_2$][OTf] (10)**

Following the general procedure, potassium hydroxide (0.50 g, 8.87 mmol), trifluoromethanesulfonic acid (0.79 mL, 8.87 mmol) and *N*-ethyl-*N,N*-di-(2-hydroxyethyl)-*N*-methyl ammonium bromide (**6**, 2.02 g, 8.87 mmol) were reacted in dry MeOH (25 mL). Yield 98%. ^{1}H NMR ($(CD_3)_2CO$): δ 1.44 (tt, 3H, J_{HH} = 7.3 Hz, J_{NH} = 2.0 Hz, NCH_2CH_3), 3.31 (s, 3H, $N(CH_3)$), 3.62-3.80 (m, 6H, 2x(NCH_2CH_2OH), NCH_2CH_3), 4.00-4.18 (m, 4H, 2x(NCH_2CH_2OH)), 4.65 (t, 2H, J_{HH} = 5.1 Hz, 2x(NCH_2CH_2OH)). ^{13}C NMR ($(CD_3)_2CO$): δ 8.3 (NCH_2CH_3), 49.5 (t, J_{CN} = 3.6 Hz, $N(CH_3)$), 56.3 (NCH_2CH_2OH), 59.0-60.4 (m, NCH_2CH_3), 62.8-65.7 (m, NCH_2CH_2OH), 121.6 (q, J_{CF} = 320.3 Hz, CF_3SO_3). ^{19}F NMR ($(CD_3)_2CO$): δ -78.99. Electrospray MS *m/z* (%) 445.21897 $[(C_7H_{18}NO_2)_2(CF_3SO_3)]^+$ ($C_{15}H_{36}F_3N_2O_7S$ requires 445.21898, 100).


f1 (ppm)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