Synthesis and Characterization of Imidazolium Perrhenate Ionic Liquids

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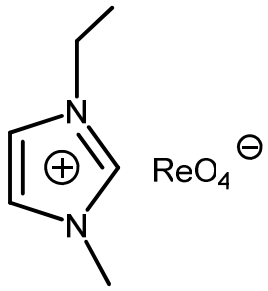
Supporting Information

Characteristic data:

IL **1**:



$C_6H_{11}N_2O_4Re$ (361.37), elemental analysis calcd.: C, 19.94, H, 3.07, N, 7.75; found: C, 19.85, H, 2.98, N, 7.71; IR (cm^{-1}): ν = 909 (Re=O), 848 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.08 (1H, s, mz-H^2), 7.75 (1H, d, mz-H^4), 7.66 (1H, d, mz-H^5), 4.21 - 4.16 (2H, q, -CH_2-), 3.85 (3H, s, N-CH_3), 1.42 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 136.84 (mz-C^2), 124.52 (mz-C^4), 122.52 (mz-C^5), 44.76 (-CH_2-), 36.34 (N-CH_3), 15.61 (-CH_3). ESI-MS (methanol, m/z, %): cation,111.0 ($C_6H_{11}N_2^+$, 100%).

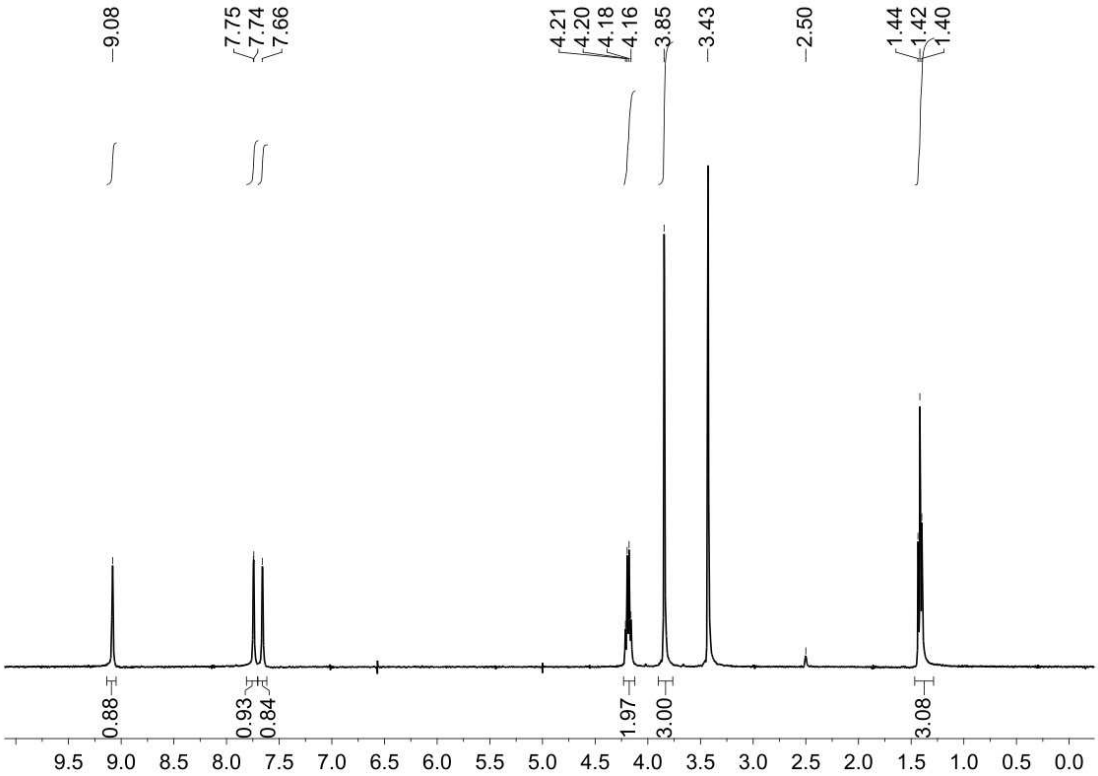


Figure S1-1 ^{1}H NMR spectrum of IL **1**

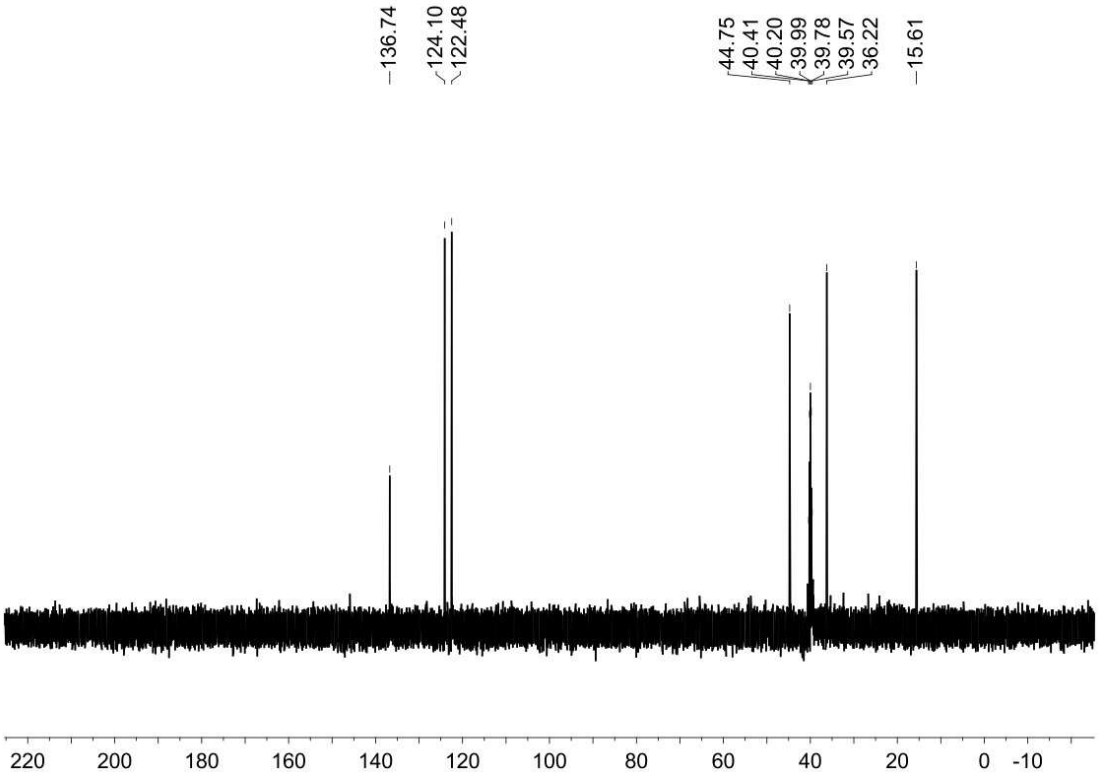


Figure S1-2 ^{13}C NMR spectrum of IL **1**

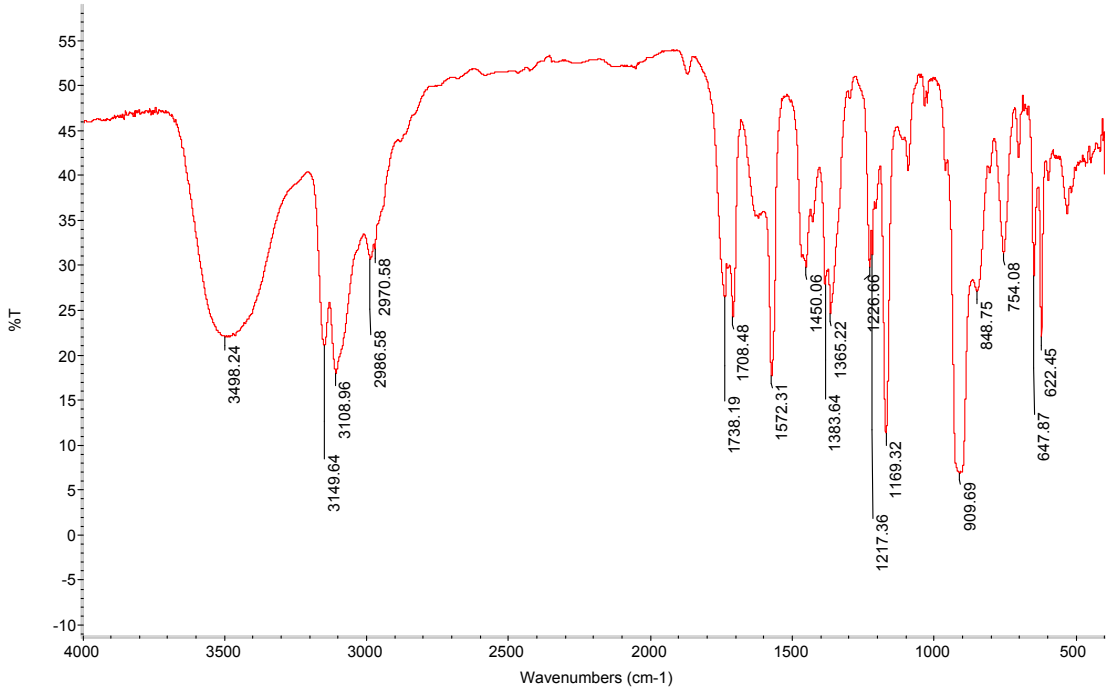


Figure S1-3 IR spectrum of IL **1**

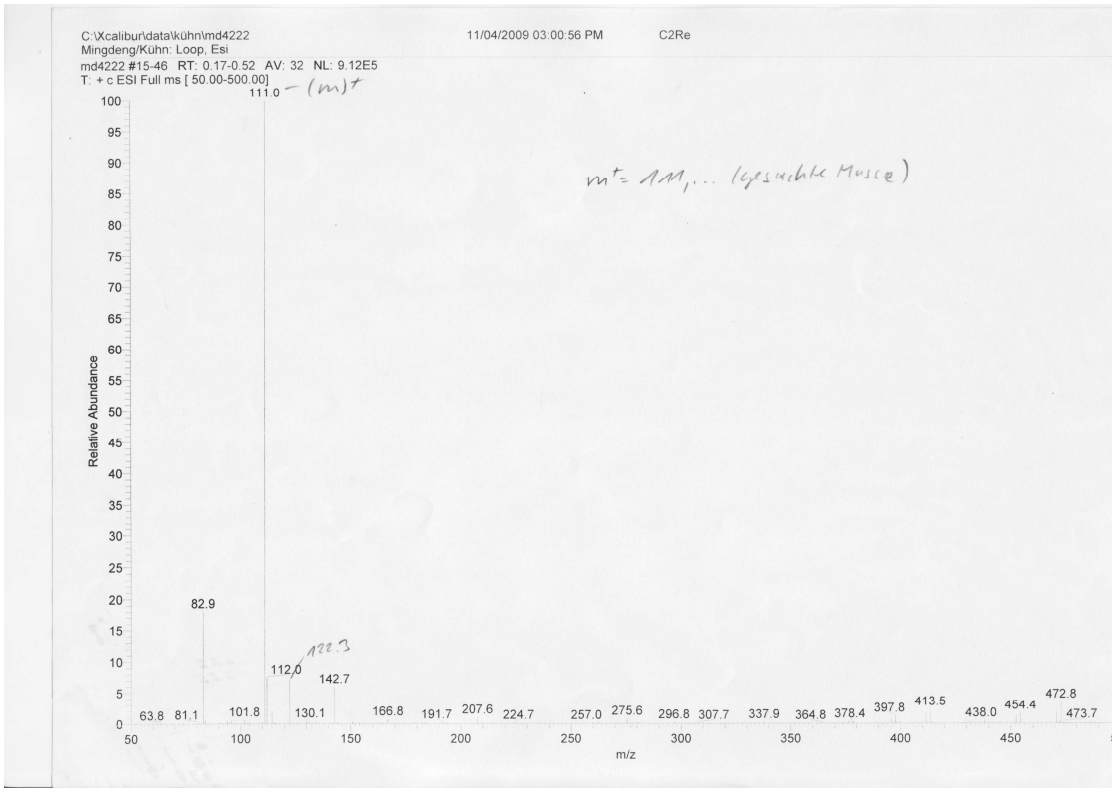


Figure S1-4 ESI-MS spectrum of IL **1**

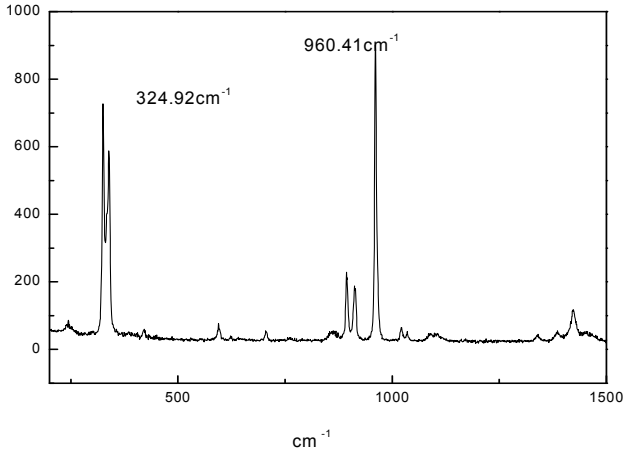


Figure S1-5 Raman spectrum of IL **1**

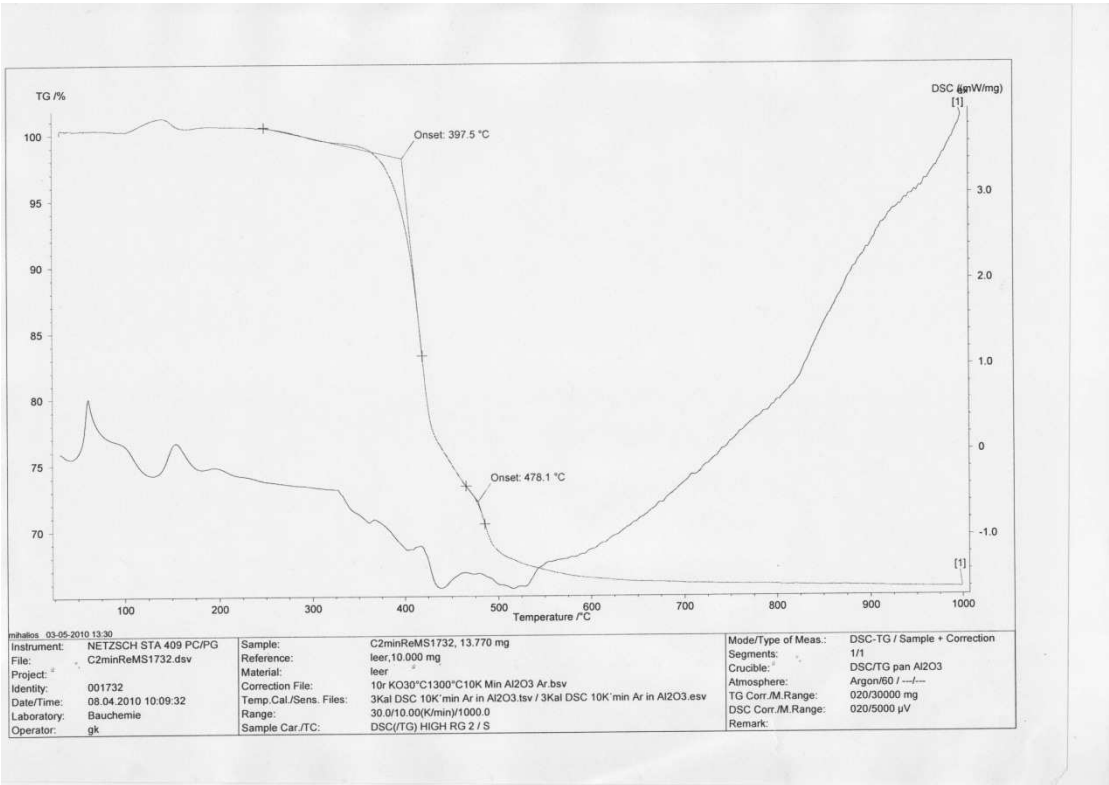
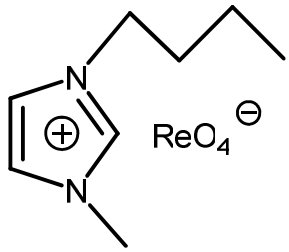


Figure S1-6 TG-MS spectrum of IL **1**

IL **2**:



$C_8H_{15}N_2O_4Re$ (389.42), elemental analysis calcd.: C, 24.67, H, 3.88, N, 7.19; found: C, 25.21, H, 4.12, N, 7.17; IR (cm^{-1}): ν = 905 (Re=O), 848 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.04 (1H, s, mz-H^2), 7.71 (1H, d, mz-H^4), 7.65 (1H, d, mz-H^5), 4.17 - 4.14 (2H, t, -CH_2-), 3.85 (3H, s, N-CH_3), 1.80-1.73 (2H, m, -CH_2-), 1.29-1.23 (2H, m, -CH_2-), 0.89 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 137.00 (mz-C^2), 124.11 (mz-C^4), 122.76 (mz-C^5), 49.17 (-CH_2-), 36.22 (N-CH_3), 31.93, 19.36 (-CH_2-), 13.77 (-CH_3). ESI-MS (methanol, m/z, %): cation,139.1 ($C_8H_{15}N_2^+$, 100%).

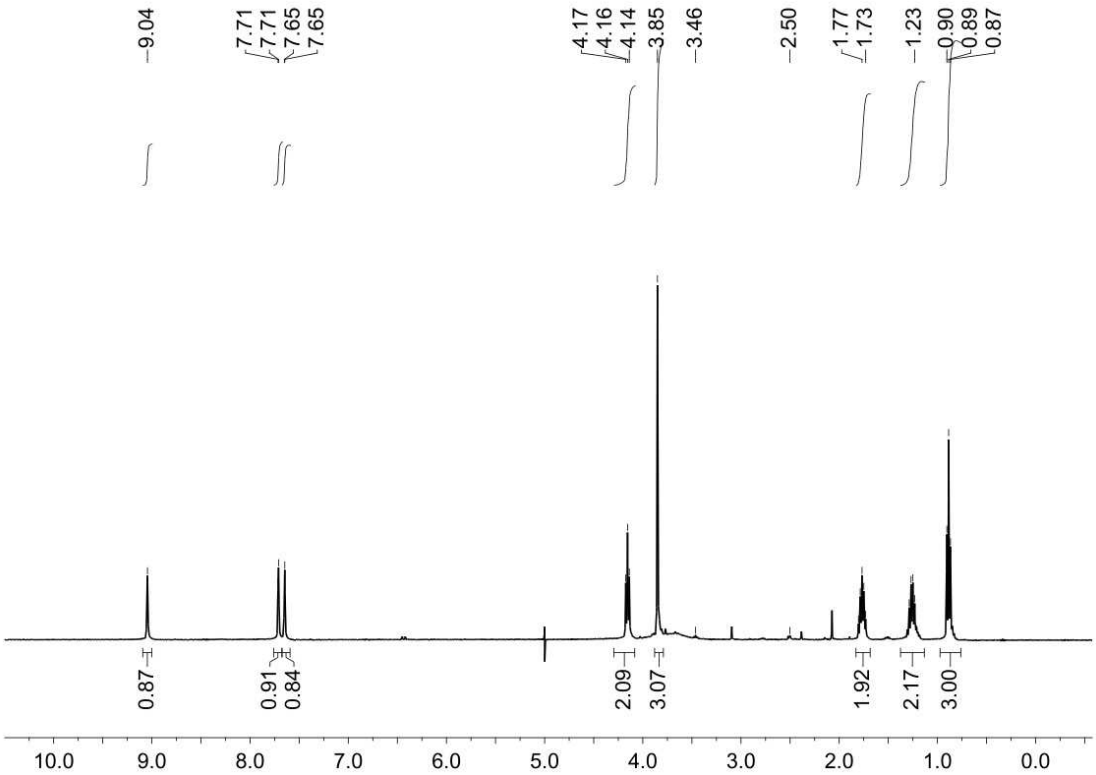


Figure S2-1 ^{1}H NMR spectrum of IL **2**

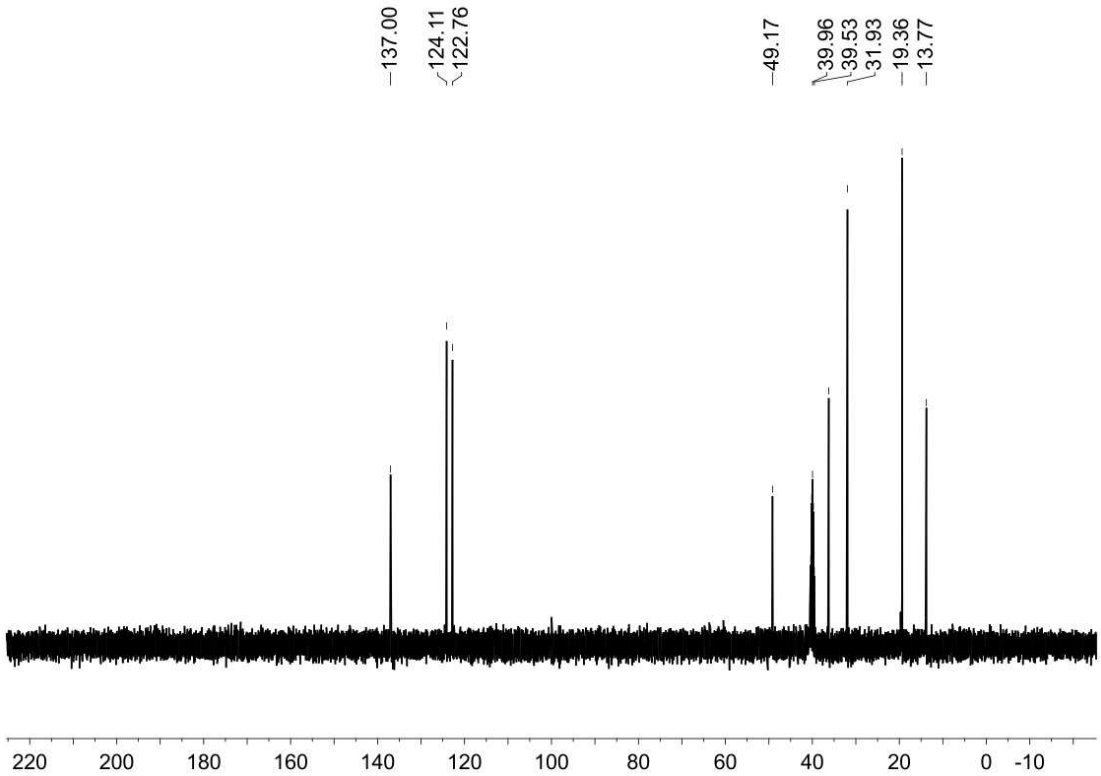


Figure S2-2 ^{13}C NMR spectrum of IL **2**

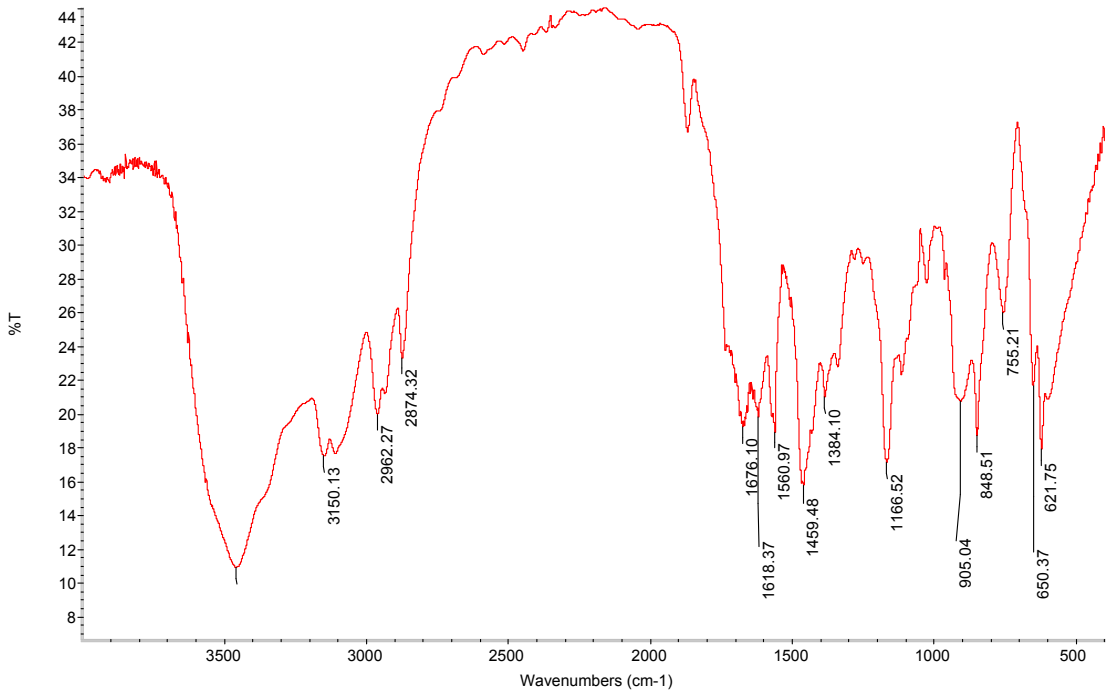


Figure S2-3 IR spectrum of IL **2**

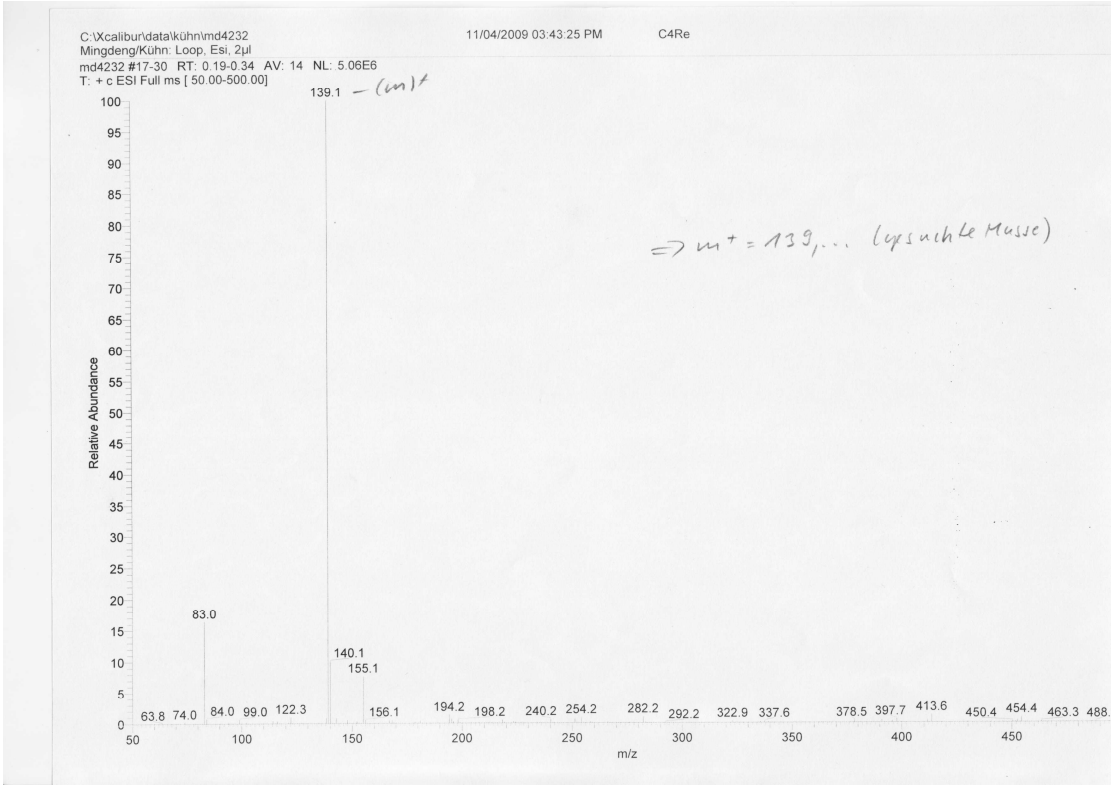


Figure S2-4 ESI-MS spectrum of IL **2**

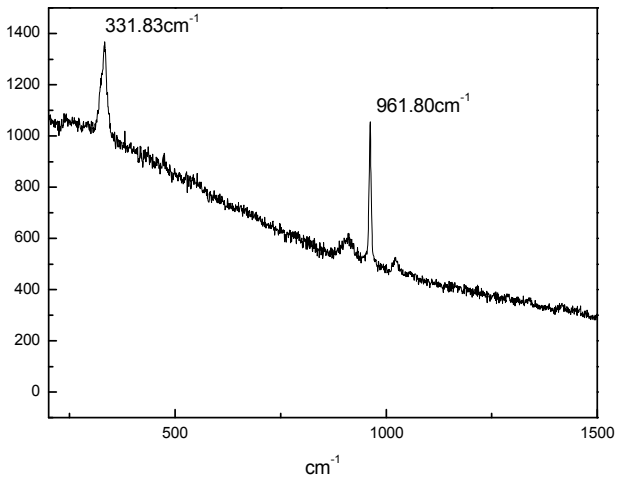


Figure S2-5 The Raman spectrum of IL **2**

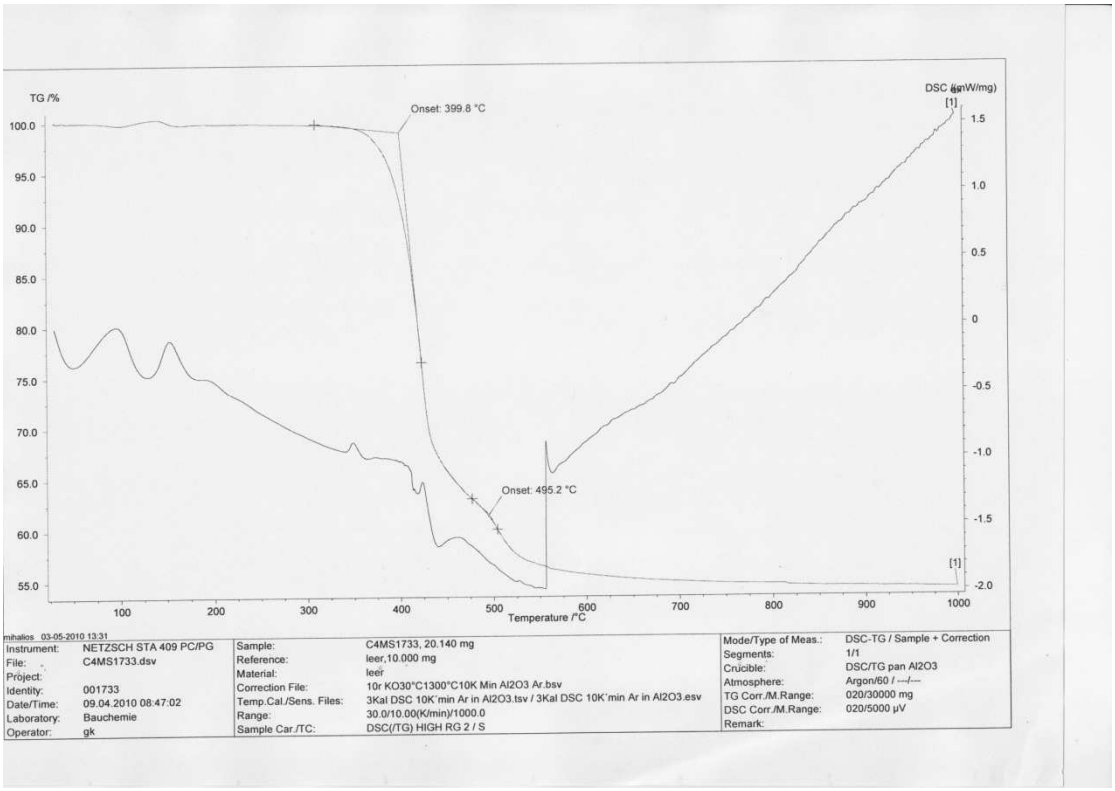
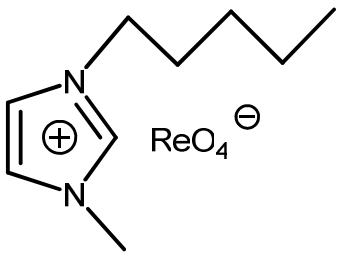


Figure S2-6 The TG-MS spectrum of IL **2**

IL **3**:



$C_9H_{17}N_2O_4Re$ (403.45), elemental analysis calcd.: C, 26.79, H, 4.25, N, 6.94; found: C, 26.48, H, 4.24, N, 6.90; IR (cm^{-1}): ν = 906 (Re=O), 853 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.03 (1H, s, mz-H^2), 7.71 (1H, d, mz-H^4), 7.64 (1H, d, mz-H^5), 4.17 - 4.13 (2H, t, -CH_2-), 3.86 (3H, s, N-CH_3), 1.81-1.77 (2H, m, -CH_2-), 1.33-1.18, (4H, m, 2×-CH_2-), 0.85 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 136.99 (mz-C^2), 124.10 (mz-C^4), 122.74 (mz-C^5), 49.47 (-CH_2-), 36.21 (N-CH_3), 29.64, 28.20, 22.07 (-CH_2-), 14.20 (-CH_3). ESI-MS (methanol, m/z, %): cation,153.1 ($C_9H_{17}N_2^+$, 100%).

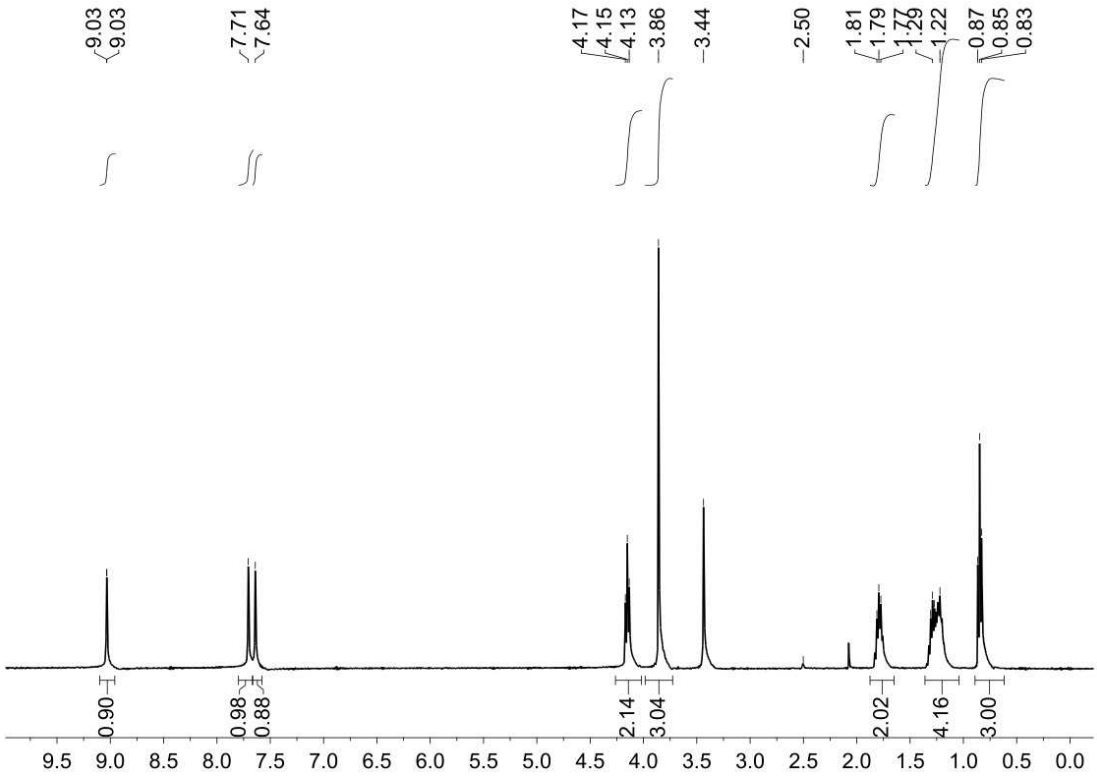


Figure S3-1 ^{1}H NMR spectrum of IL **3**

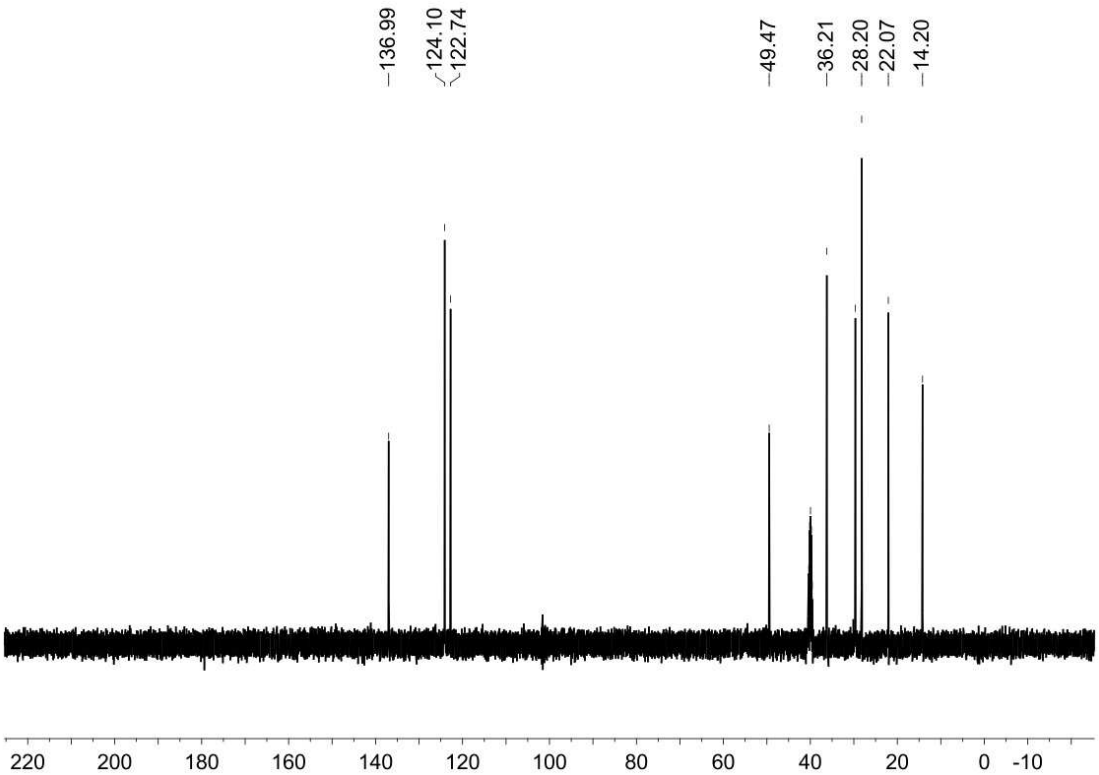


Figure S3-2 ^{13}C NMR spectrum of IL **3**

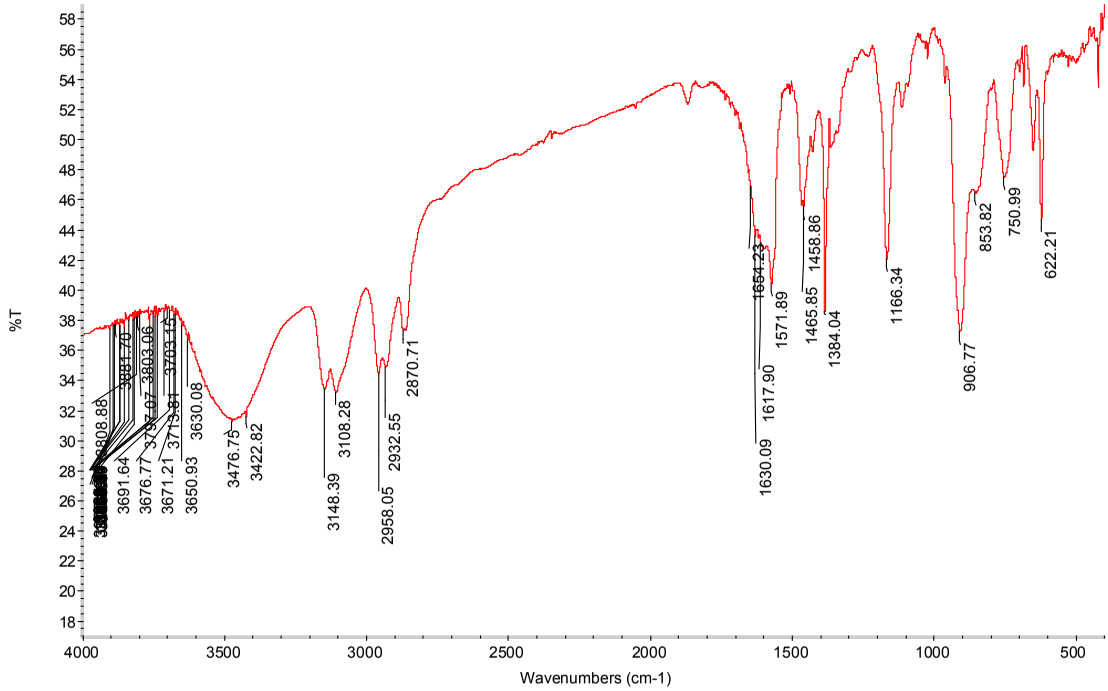


Figure S3-3 IR spectrum of IL **3**

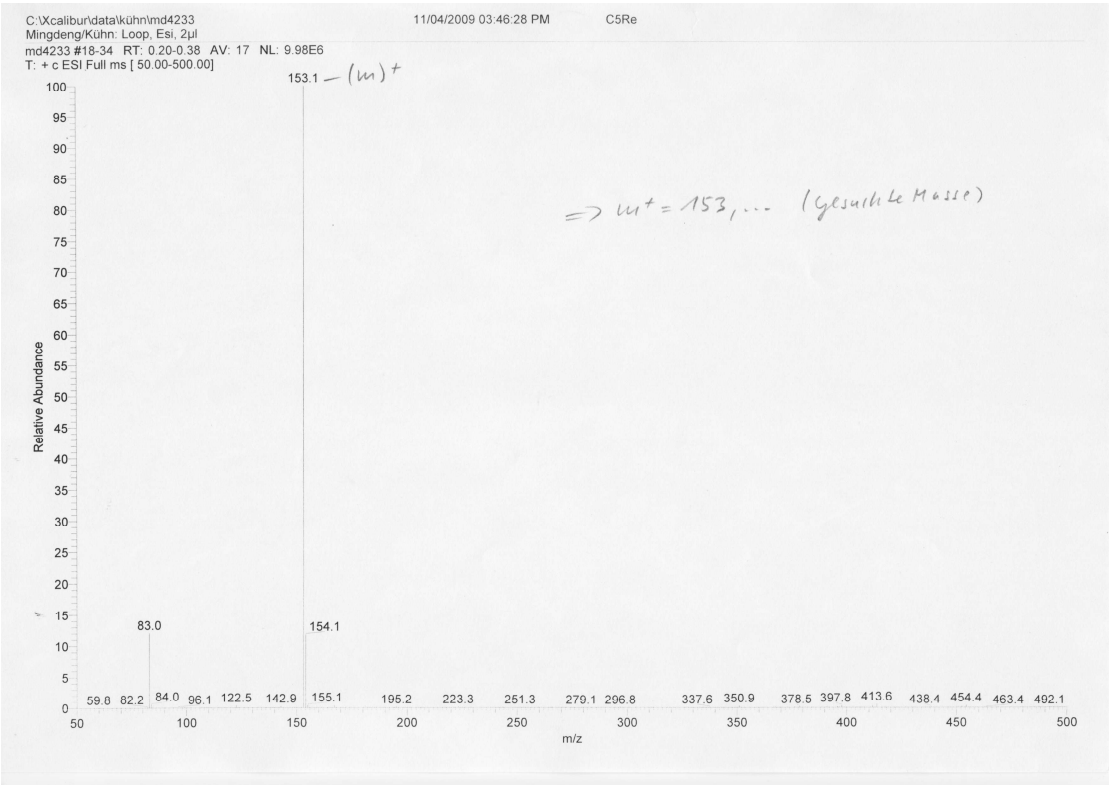


Figure S3-4 ESI-MS spectrum of IL **3**

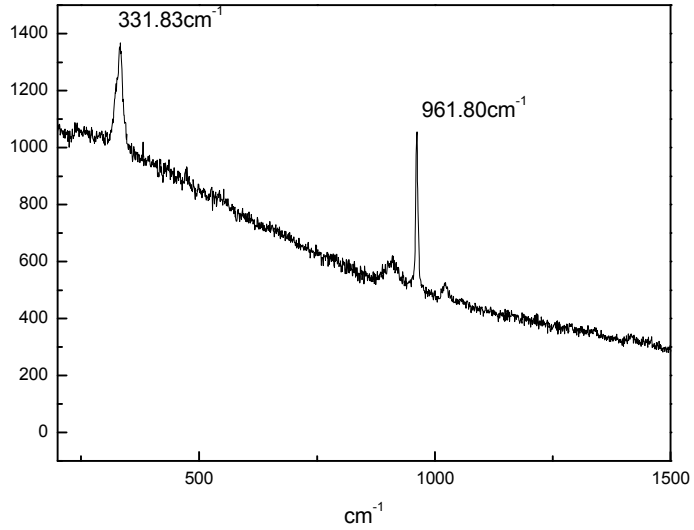


Figure S3-5 Raman spectrum of IL **3**

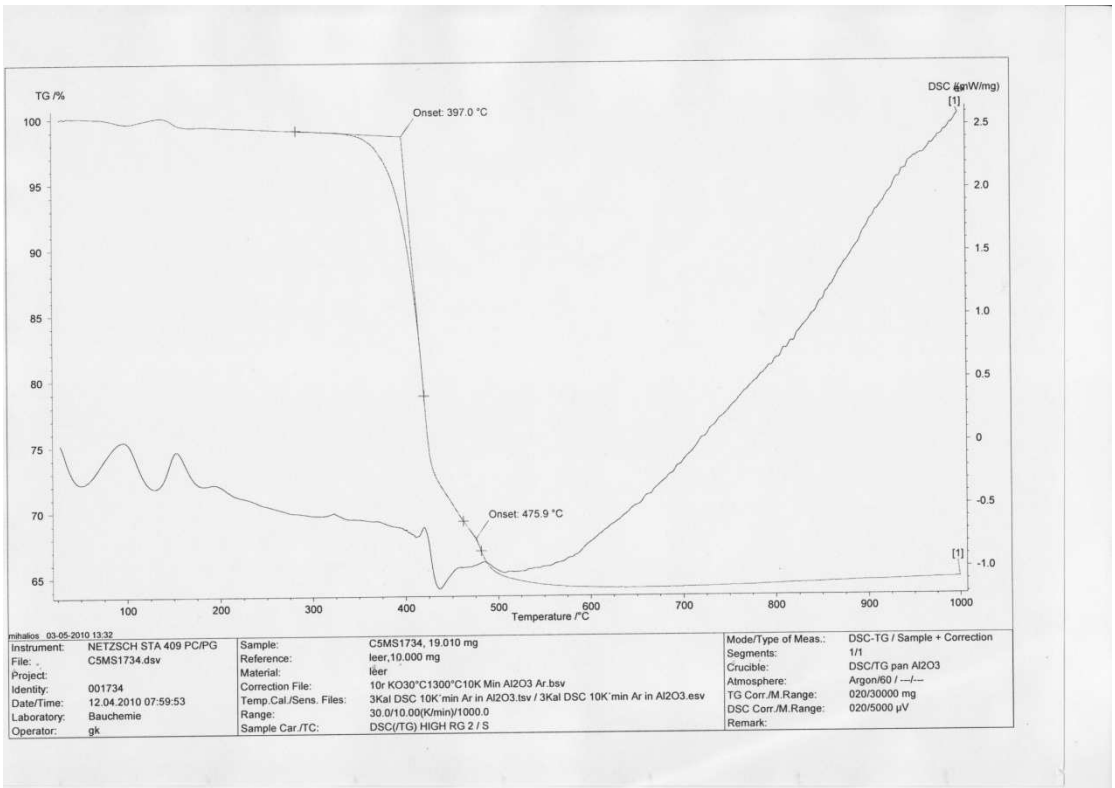
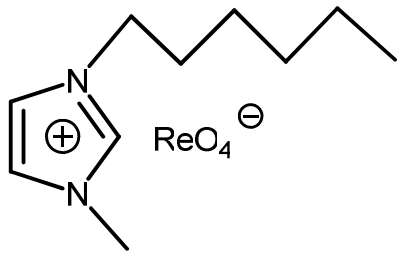


Figure S3-6 TG-MS spectrum of IL **3**

IL **4**:



$C_{10}H_{19}N_2O_4Re$ (417.48), elemental analysis calcd.: C, 28.77, H, 4.59, N, 6.71; found: C, 28.74, H, 4.69, N, 6.64; IR (cm^{-1}): ν = 908 (Re=O), 848 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.53 (1H, s, mz-H^2), 7.96 (1H, d, mz-H^4), 7.87 (1H, d, mz-H^5), 4.23 - 4.19 (2H, t, -CH_2-), 3.89 (3H, s, N-CH_3), 1.77-1.73 (2H, m, -CH_2-), 1.19 (6H, m, 3×-CH_2-), 0.77 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 137.00 (mz-C^2), 124.11 (mz-C^4), 122.76 (mz-C^5), 49.47 (-CH_2-), 36.22 (N-CH_3), 31.11, 29.93, 25.73, 22.42 (-CH_2-), 14.31 (-CH_3). ESI-MS (methanol, m/z, %): cation,167.1 ($C_{10}H_{19}N_2^+$, 100%).

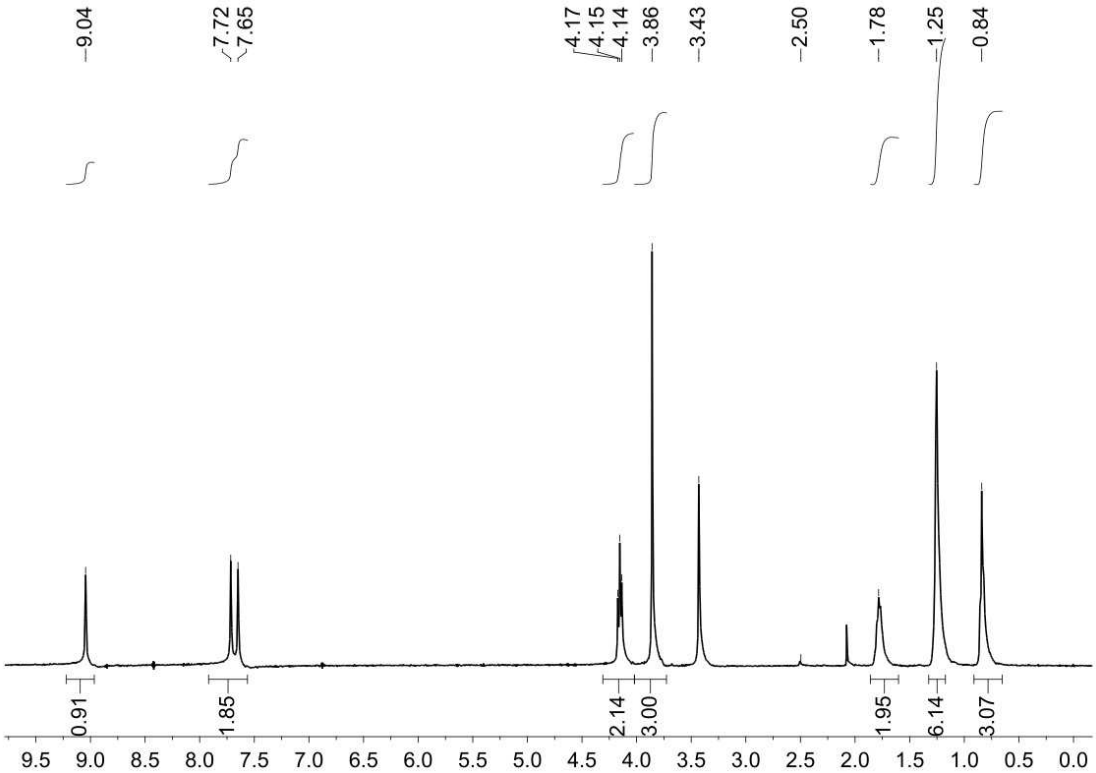


Figure S4-1 ^{1}H NMR spectrum of IL **4**

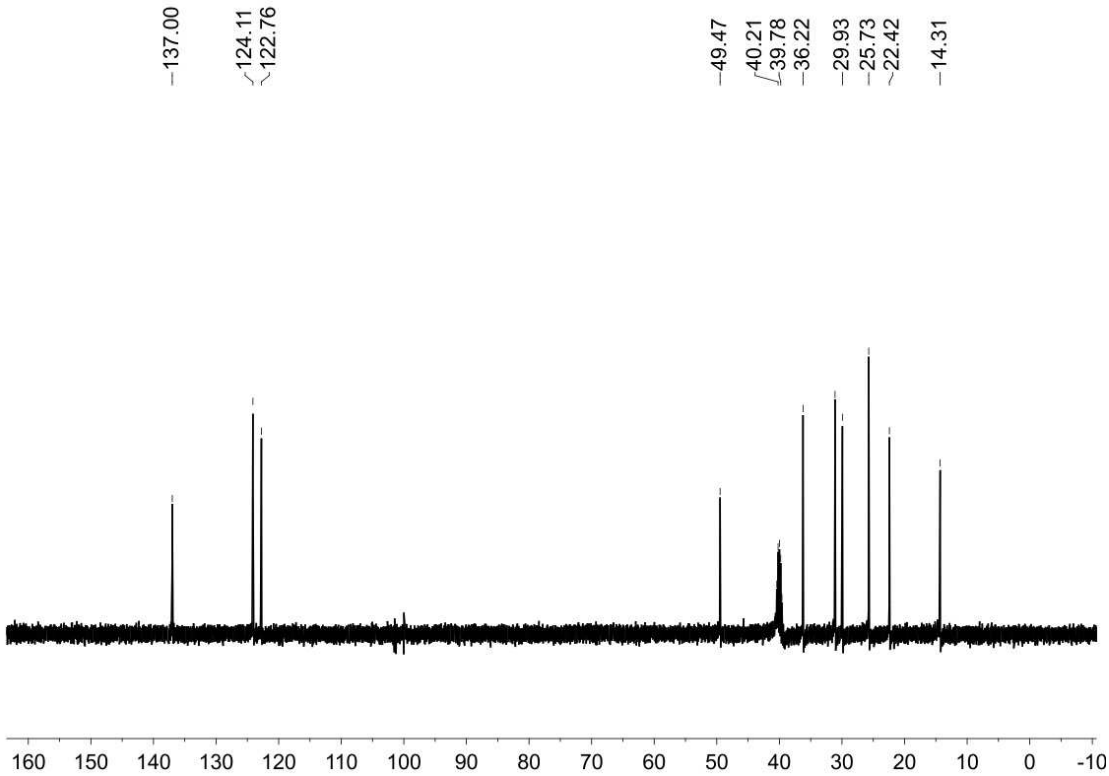


Figure S4-2 ^{13}C NMR spectrum of compound **4**

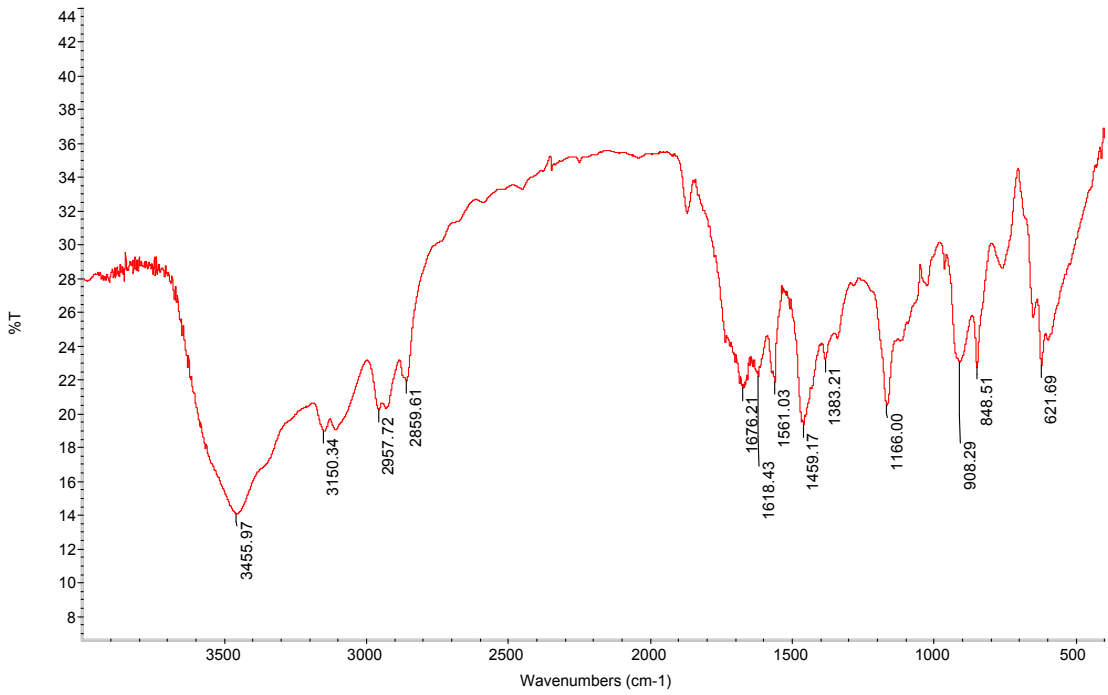


Figure S4-3 IR spectrum of IL **4**

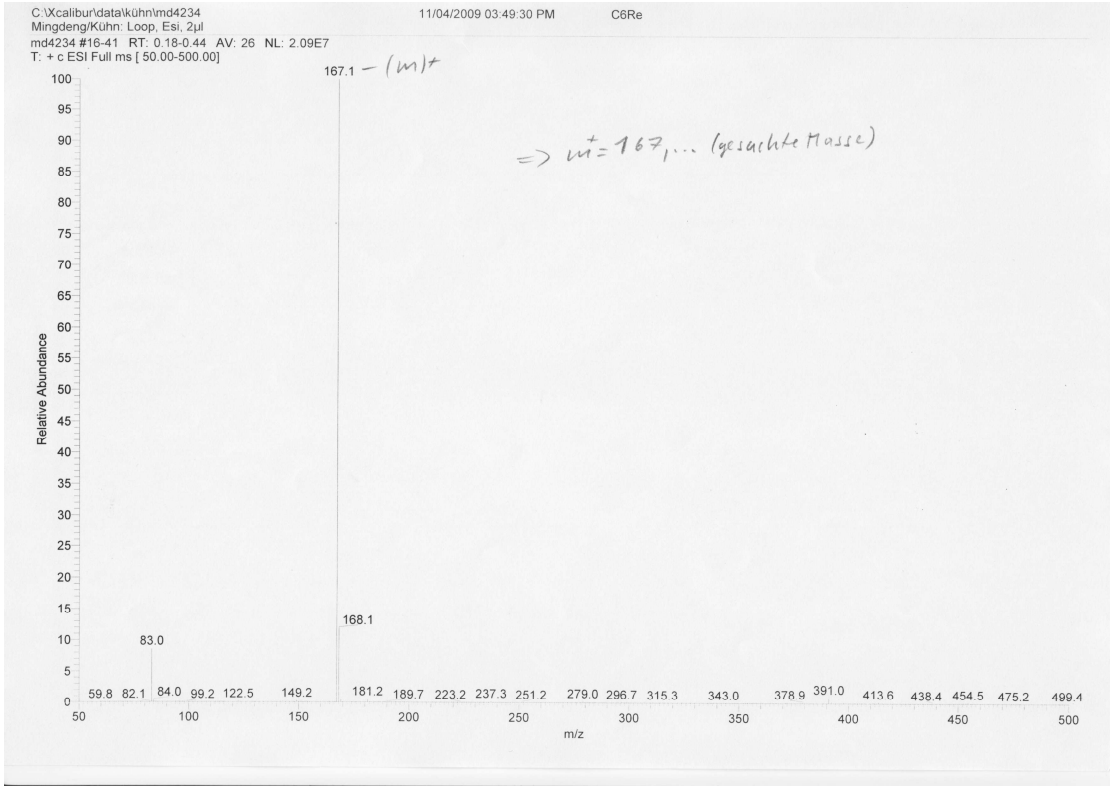


Figure S4-4 ESI-MS spectrum of IL **4**

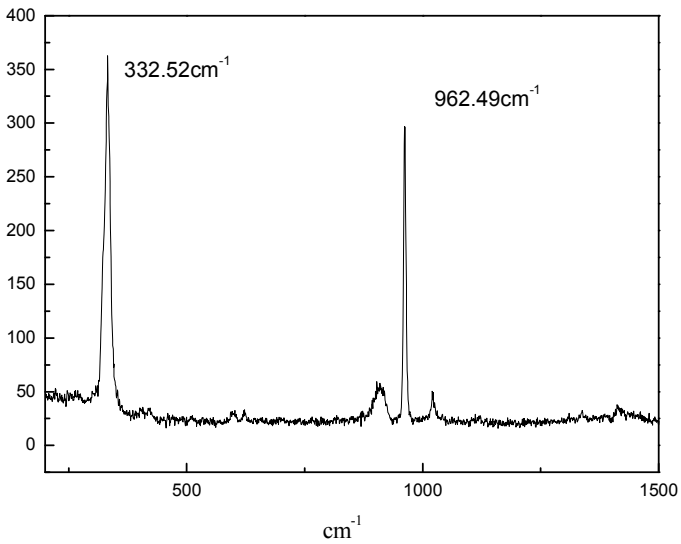


Figure S4-5 Raman spectrum of IL **4**

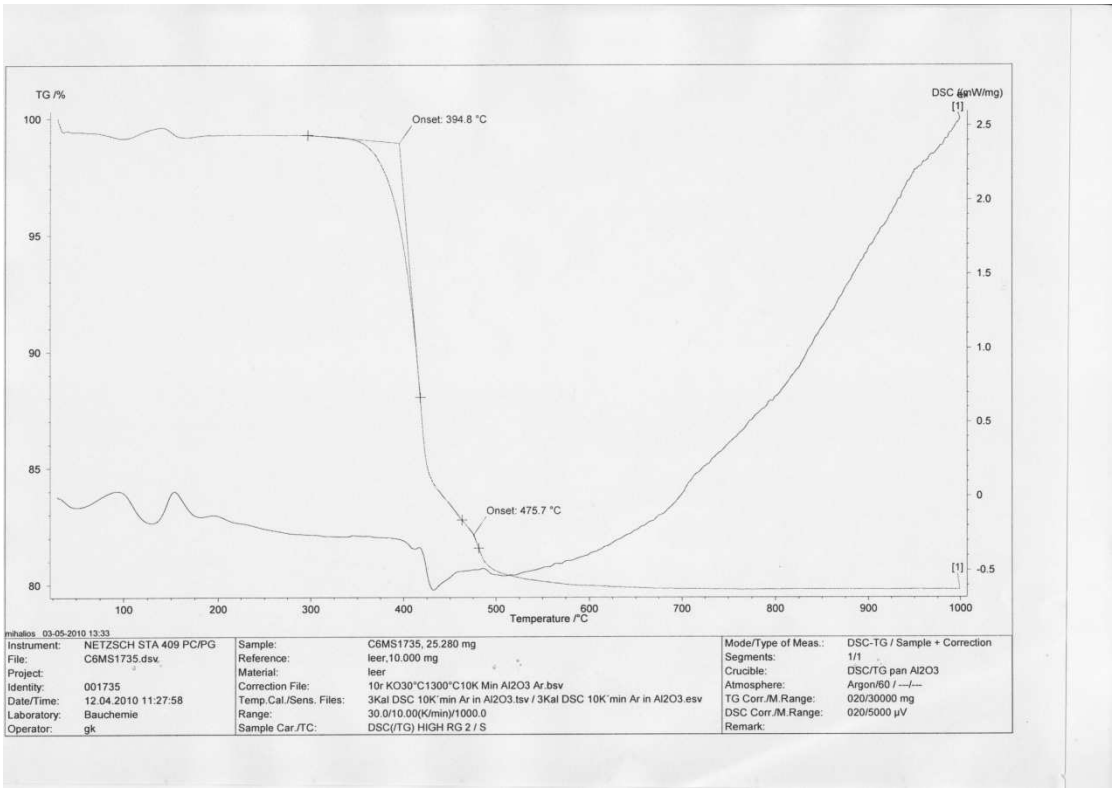
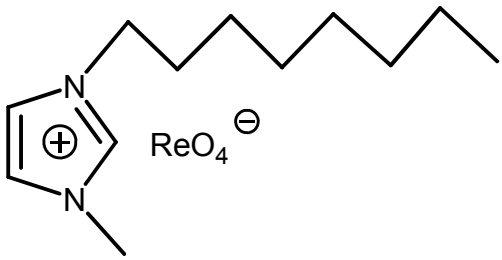


Figure S4-6 TG-MS spectrum of IL **4**

IL **5**:



$C_{12}H_{23}N_2O_4Re$ (445.53), elemental analysis calcd.: C, 32.35, H, 5.20, N, 6.29; found: C, 31.69, H, 4.92, N, 5.80; IR (cm^{-1}): ν = 913 (Re=O), 848 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.05 (1H, s, mz-H^2), 7.72 (1H, d, mz-H^4), 7.66 (1H, d, mz-H^5), 4.17 - 4.13 (2H, t, -CH_2-), 3.86 (3H, s, N-CH_3), 1.82-1.75 (2H, m, -CH_2-), 1.23 (10H, m, 5×-CH_2-), 0.84 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 137.00 (mz-C^2), 124.11 (mz-C^4), 122.77 (mz-C^5), 49.46 (-CH_2-), 36.23 (N-CH_3), 31.72, 29.97, 29.01, 28.90, 26.08, 22.60 (-CH_2-), 14.40 (-CH_3). ESI-MS (methanol, m/z, %): cation,195.2 ($C_{12}H_{23}N_2^+$, 100%).

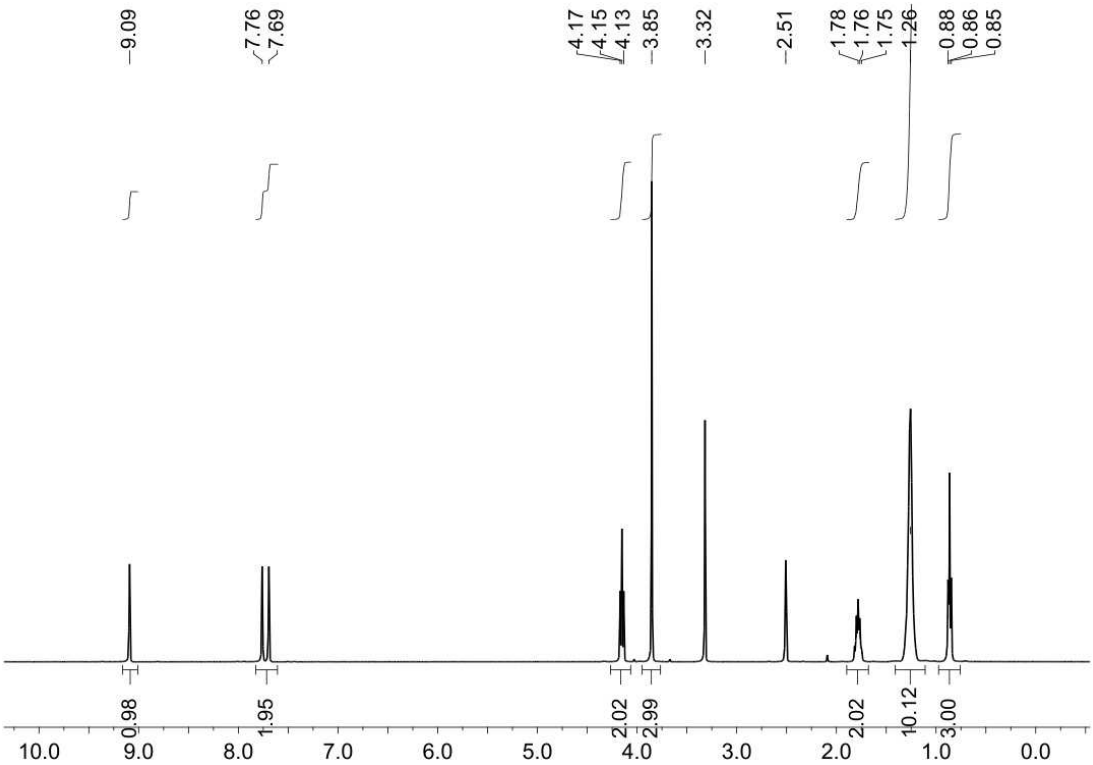


Figure S5-1 ^{1}H NMR spectrum of IL **5**

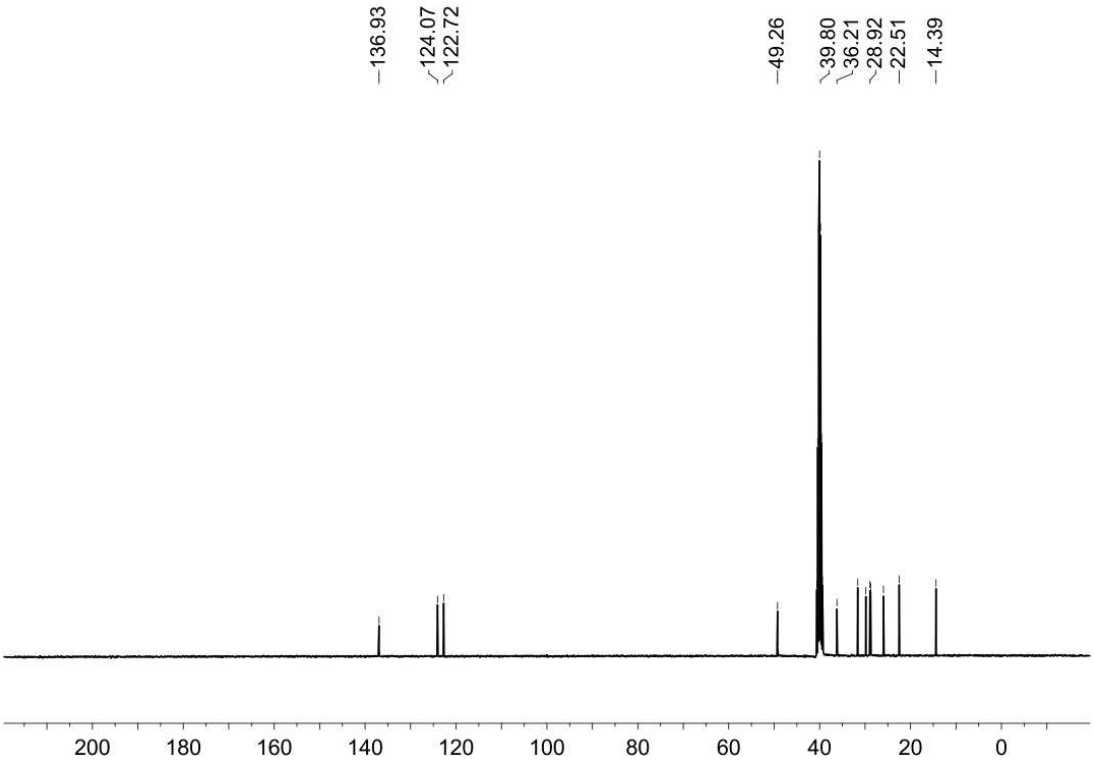


Figure S5-2 ^{13}C NMR spectrum of IL **5**

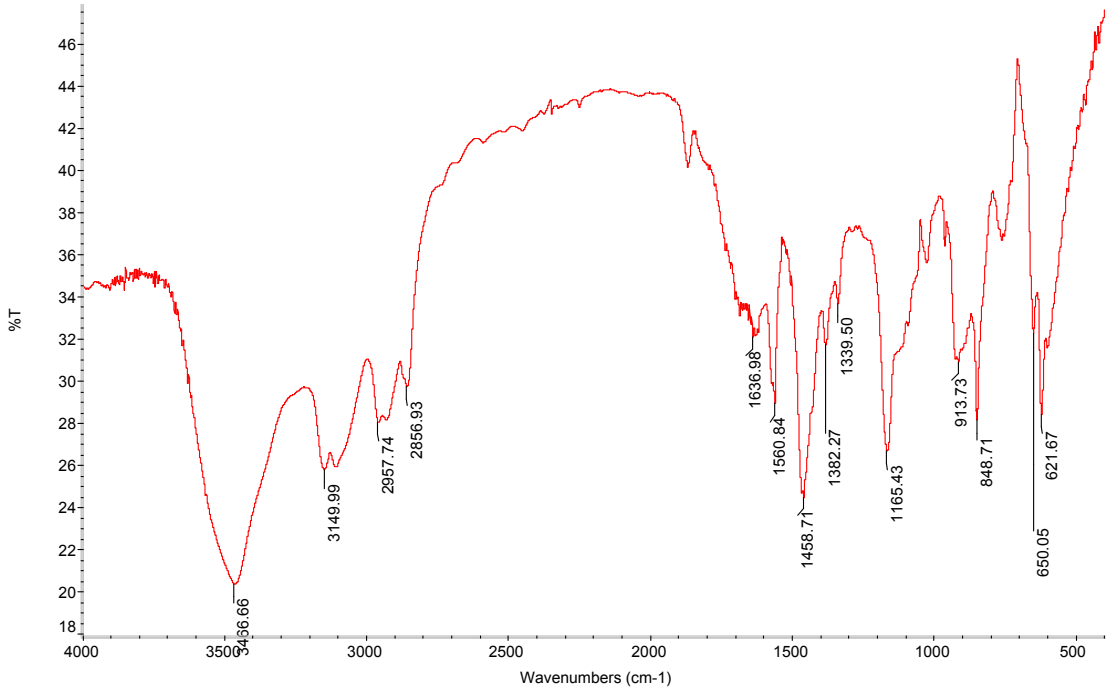


Figure S5-3 IR spectrum of IL **5**

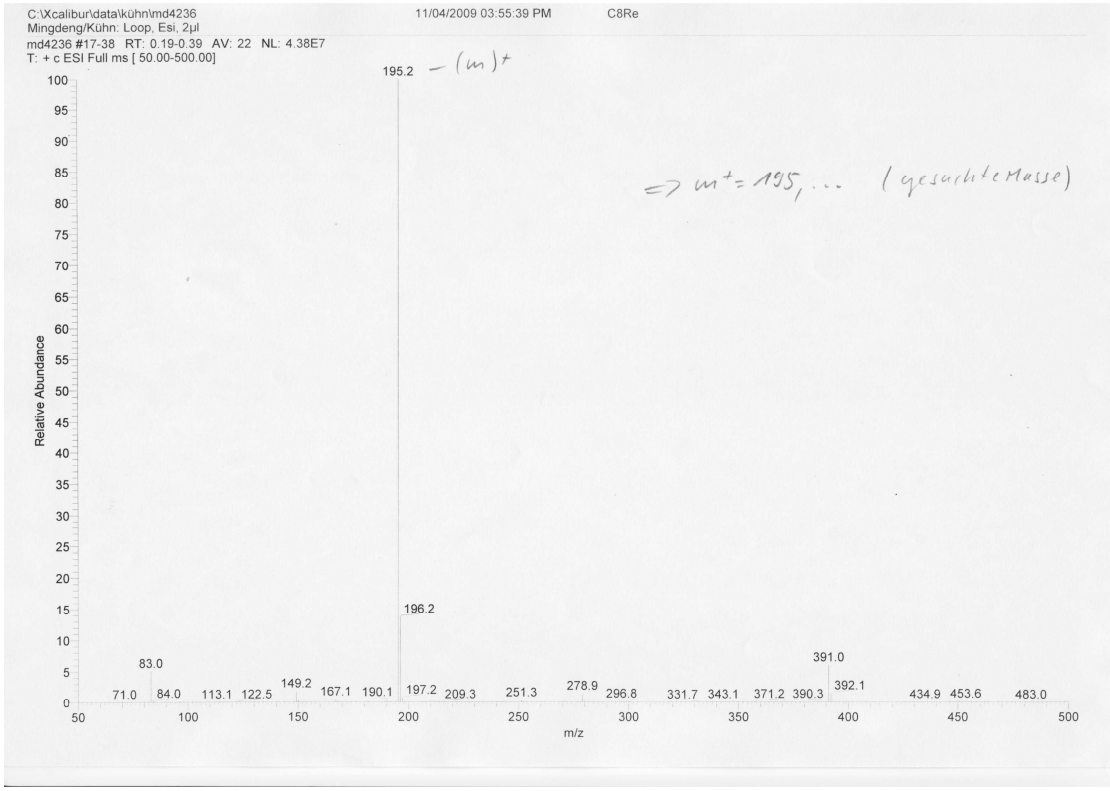


Figure S5-4 ESI-MS spectrum of IL **5**

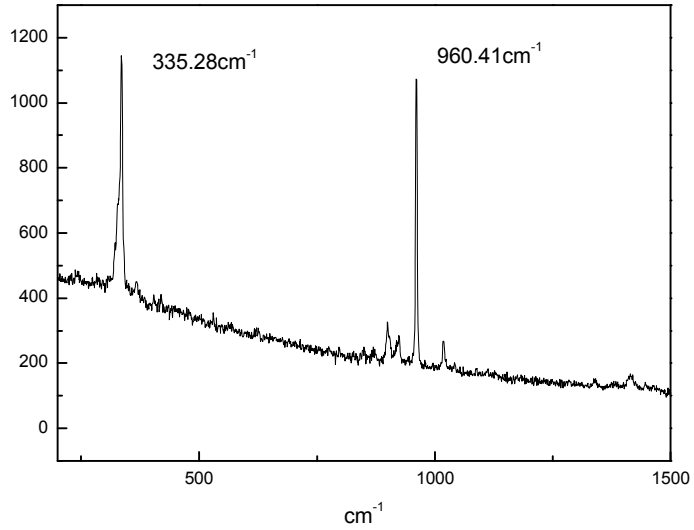


Figure S5-5 Raman spectrum of IL**5**

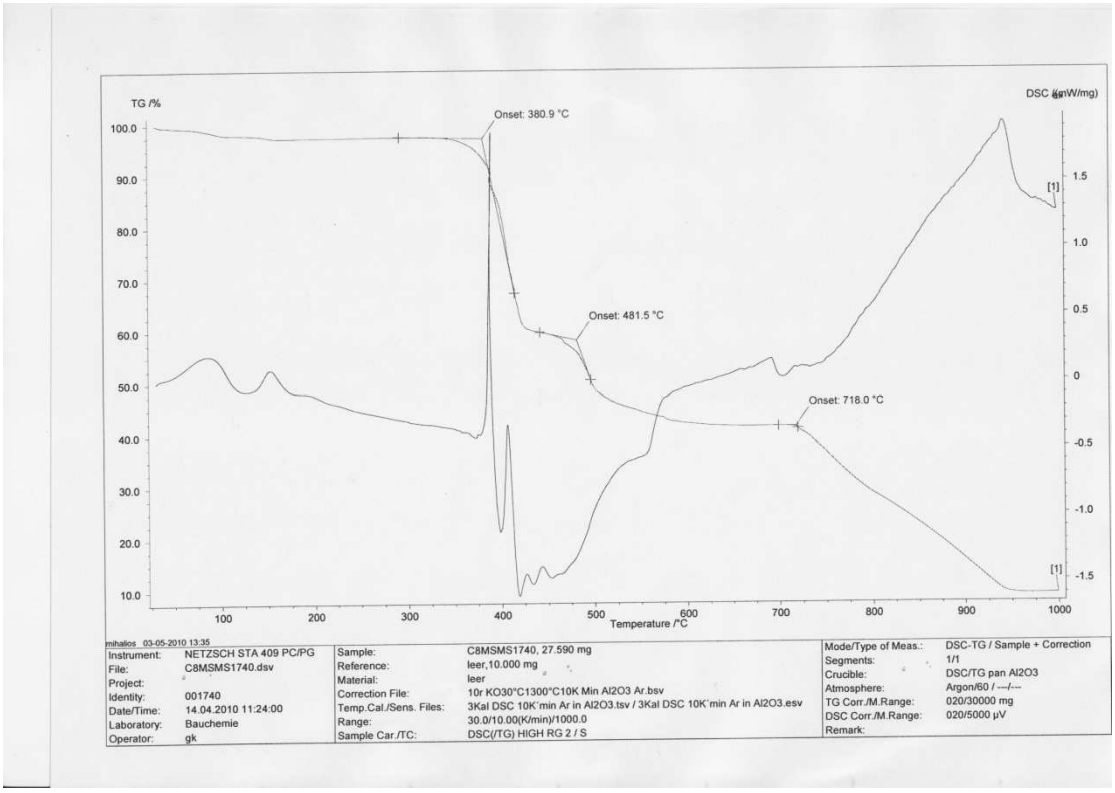
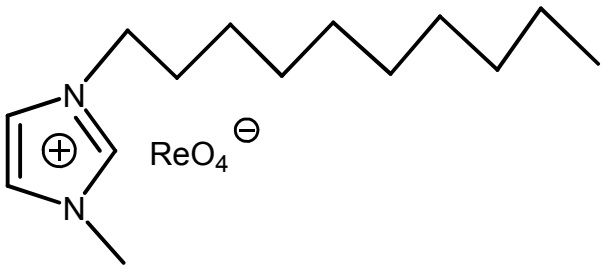


Figure S5-6 TG-MS spectrum of IL **5**

IL **6**:



$C_{14}H_{27}N_2O_4Re$ (473.58), elemental analysis calcd.: C, 35.51, H, 5.75, N, 5.92; found: C, 35.91, H, 5.88, N, 6.07; IR (cm^{-1}): ν = 902 (Re=O), 862 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.14 (1H, s, mz-H^2), 7.75 (1H, d, mz-H^4), 7.68 (1H, d, mz-H^5), 4.16 - 4.13 (2H, t, -CH_2-), 3.85 (3H, s, N-CH_3), 1.77 (2H, m,-CH_2-), 1.22 (14H, m, 7×-CH_2-), 0.84 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 137.02 (mz-C^2), 124.12 (mz-C^4), 122.77 (mz-C^5), 49.44 (-CH_2-), 36.23 (N-CH_3), 31.88, 30.01, 29.51, 29.42, 29.27, 28.99, 26.12, 22.68 (-CH_2-), 14.41 (-CH_3). ESI-MS (methanol, m/z, %): cation, 223.3 ($C_{14}H_{27}N_2^+$, 100%), anion, 251.2 (ReO_4^-, 100%).

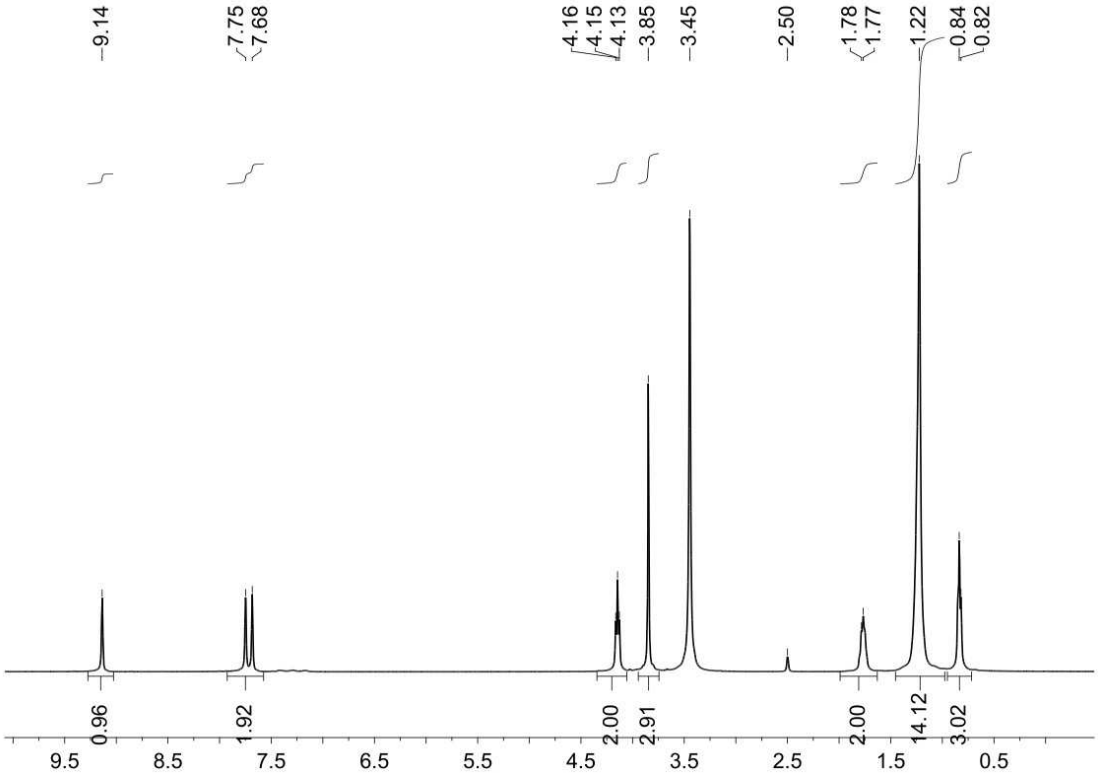


Figure S6-1 ^{1}H NMR spectrum of IL **6**

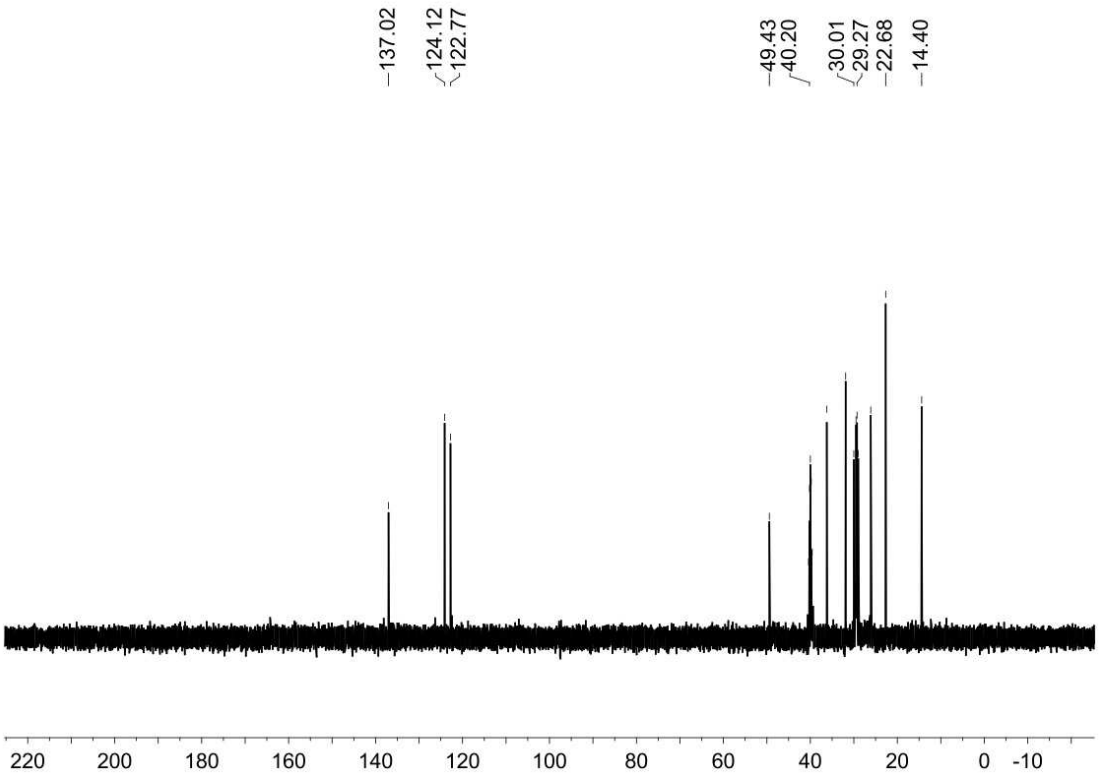
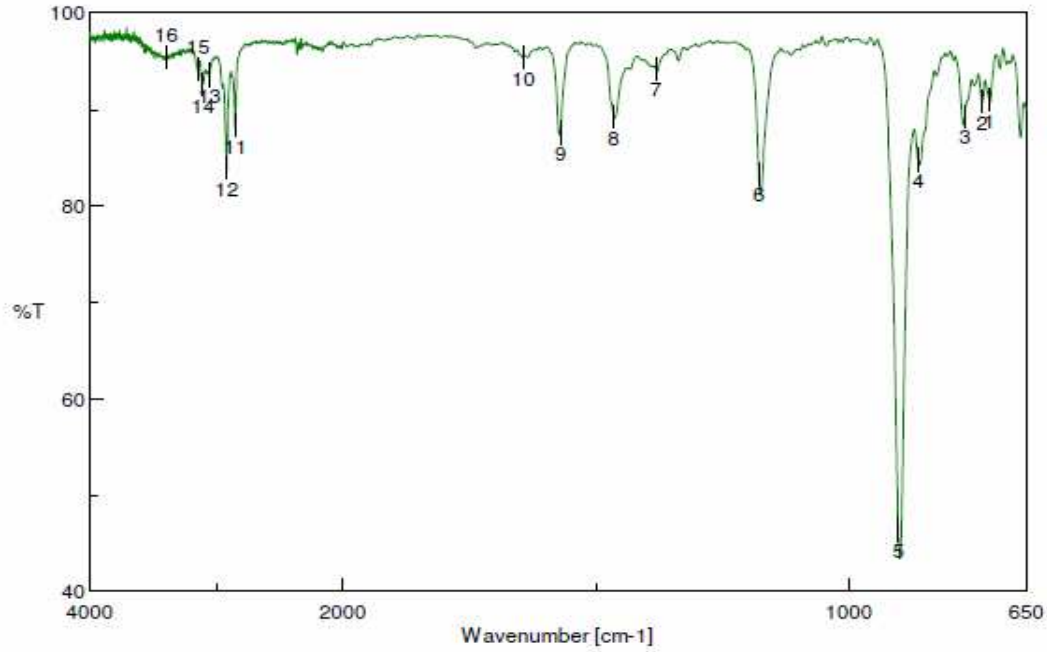


Figure S6-2 ^{13}C NMR spectrum of IL **6**



Result of Peak Picking

No.	Position	Intensity	No.	Position	Intensity	No.	Position	Intensity
1	723.175	90.9939	2	737.639	90.7925	3	771.387	89.3211
4	862.989	84.7485	5	902.523	46.3774	6	1178.29	83.3063
7	1381.75	94.1701	8	1464.67	89.2764	9	1570.74	87.5785
10	1644.02	95.3811	11	2851.24	88.3375	12	2918.73	83.9636
13	3054.69	93.5502	14	3107.72	92.5701	15	3146.29	94.1476
16	3394.1	95.4118						

Figure S6-3 IR spectrum of IL **6**

Figure S6-4 ESI-MS spectrum of IL **6**

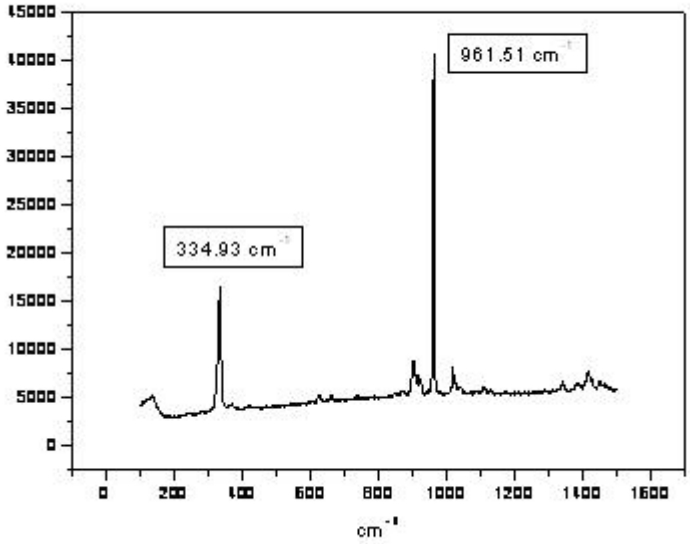


Figure S6-5 Raman spectrum of IL**6**

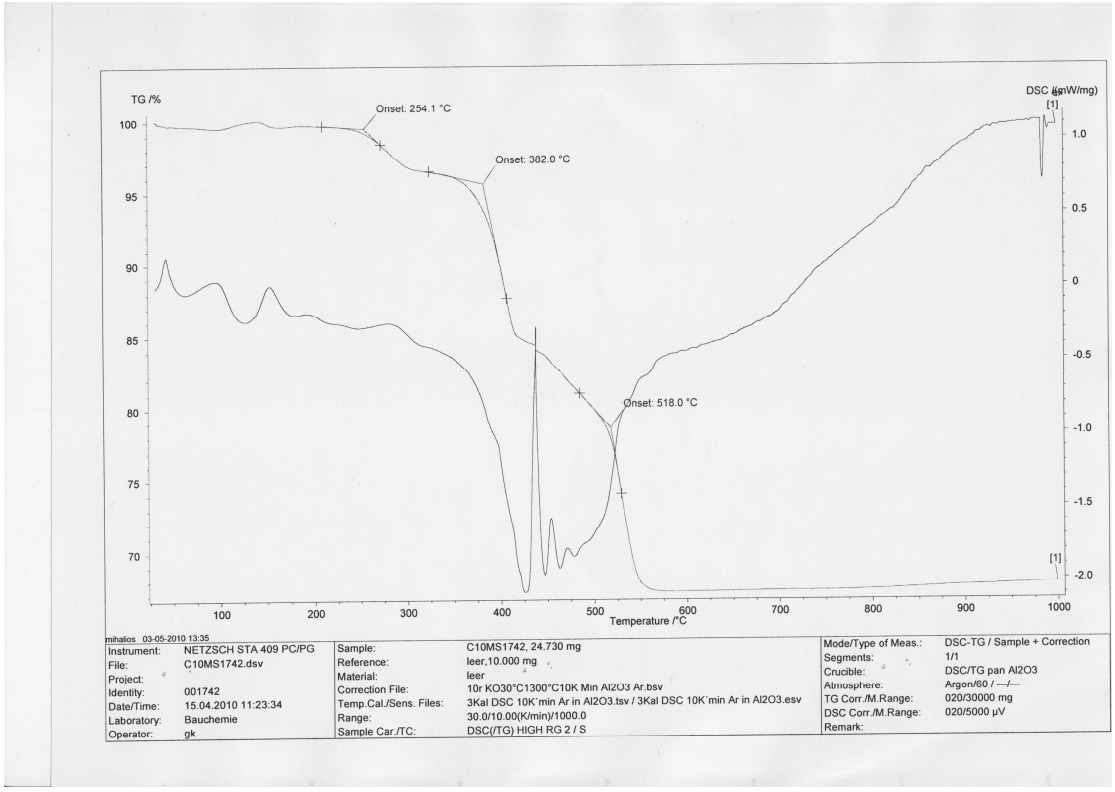
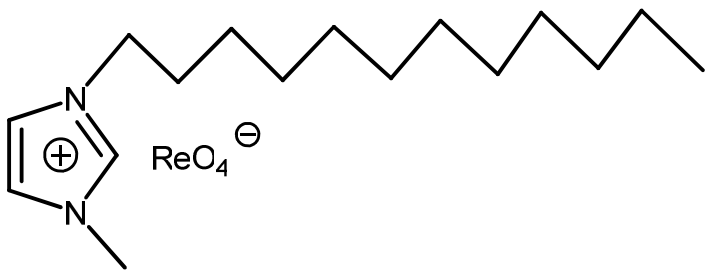


Figure S6-6 TG-MS spectrum of IL **6**

IL **7**:



$C_{16}H_{31}N_2O_4Re$ (501.64), elemental analysis calcd.: C, 38.31, H, 6.23, N, 5.58; found: C, 37.93, H, 6.28, N, 5.52; IR (cm^{-1}): ν = 910 (Re=O), 861 (Re=O); ^{1}H-NMR (DMSO, 400 MHz, r.t., ppm): δ = 9.06 (1H, s, mz-H^2), 7.73 (1H, d, mz-H^4), 7.67 (1H, d, mz-H^5), 4.15 - 4.13 (2H, t, -CH_2-), 3.85 (3H, s, N-CH_3), 1.77 (2H, m, -CH_2-), 1.23 (18H, m, 9×-CH_2-) 0.84 (3H, t, -CH_3); ^{13}C-NMR (DMSO, 100 MHz, r.t., ppm): δ = 137.01 (mz-C^2), 124.14 (mz-C^4), 122.80 (mz-C^5), 49.41 (-CH_2-), 36.27 (N-CH_3), 31.86, 29.59, 29.57, 29.39, 29.51, 29.39, 29.27, 28.95, 26.08, 22.65 (-CH_2-), 15.61 (-CH_3). ESI-MS (methanol, m/z, %): cation,251.3 ($C_{16}H_{31}N_2^+$, 100%).

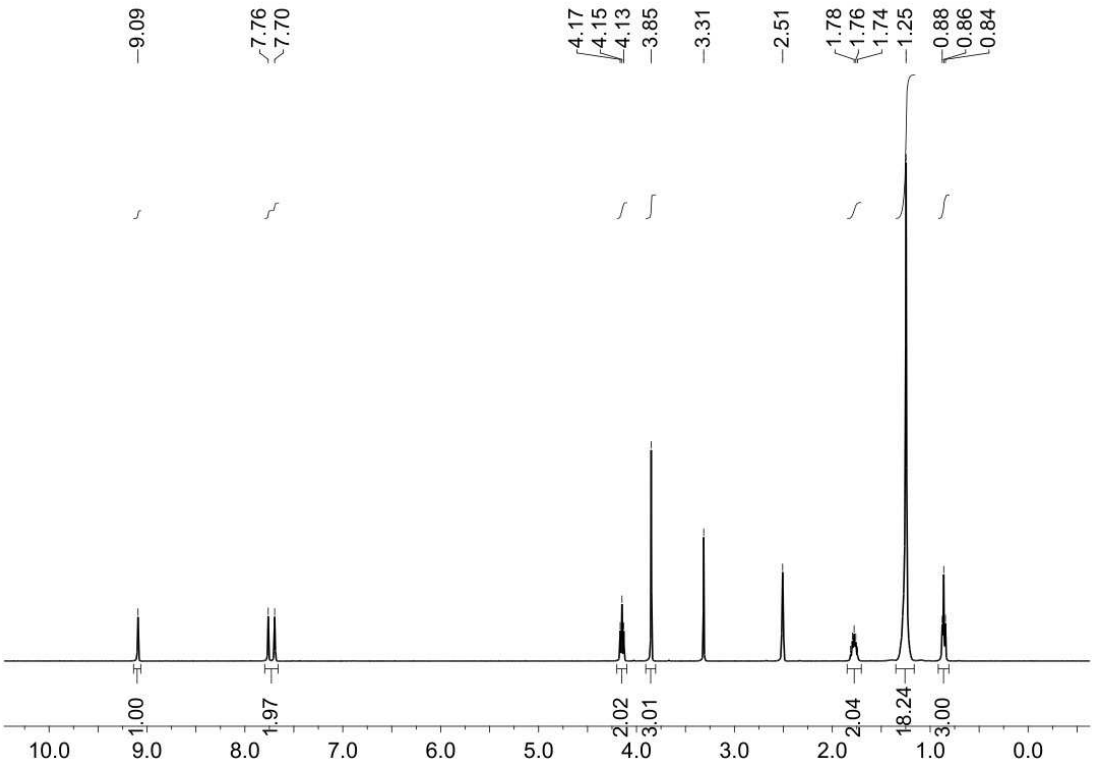


Figure S7-1 ^{1}H NMR spectrum of IL **7**

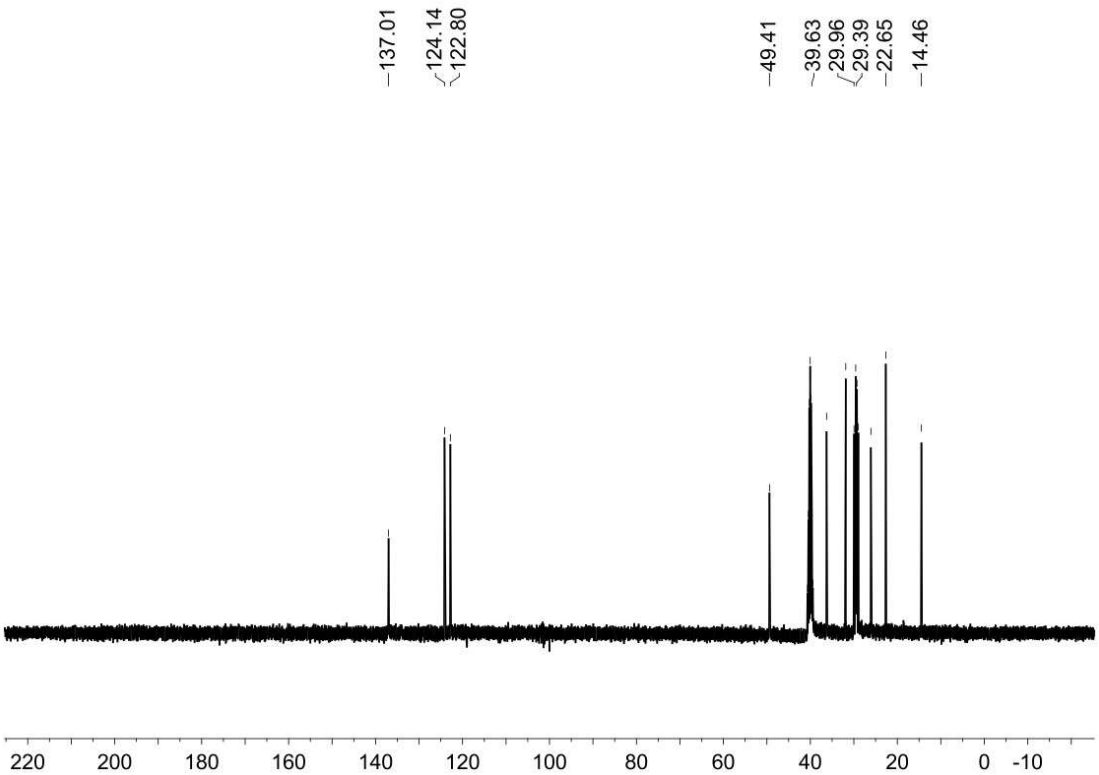


Figure S7-2 ^{13}C NMR spectrum of IL **7**

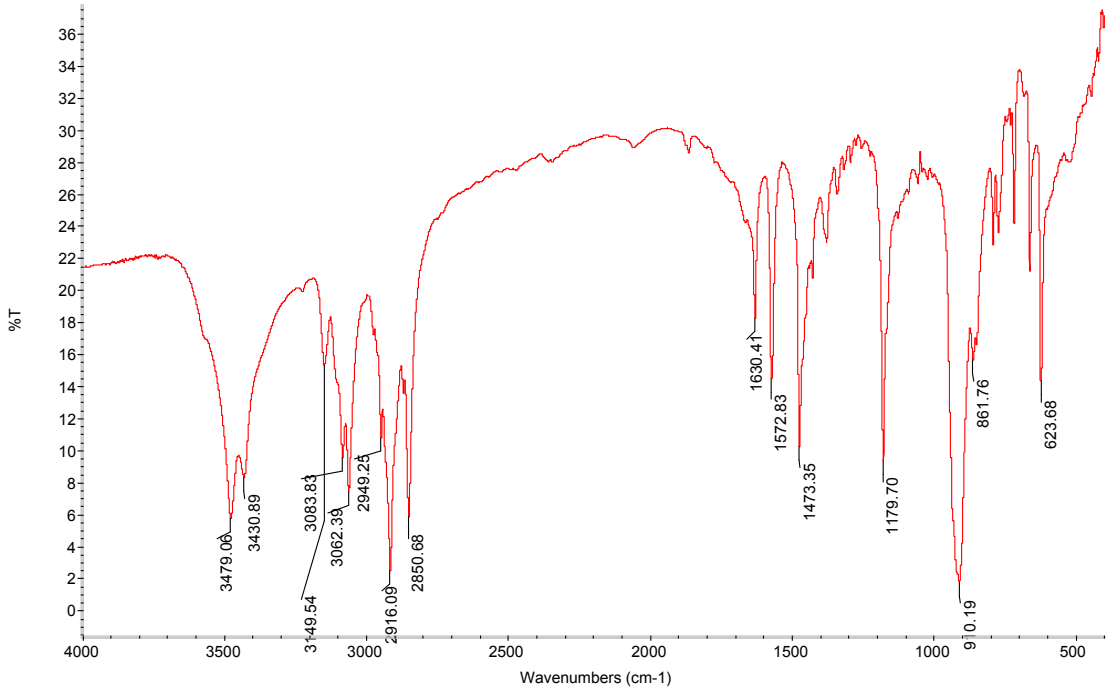


Figure S7-3 IR spectrum of IL **7**

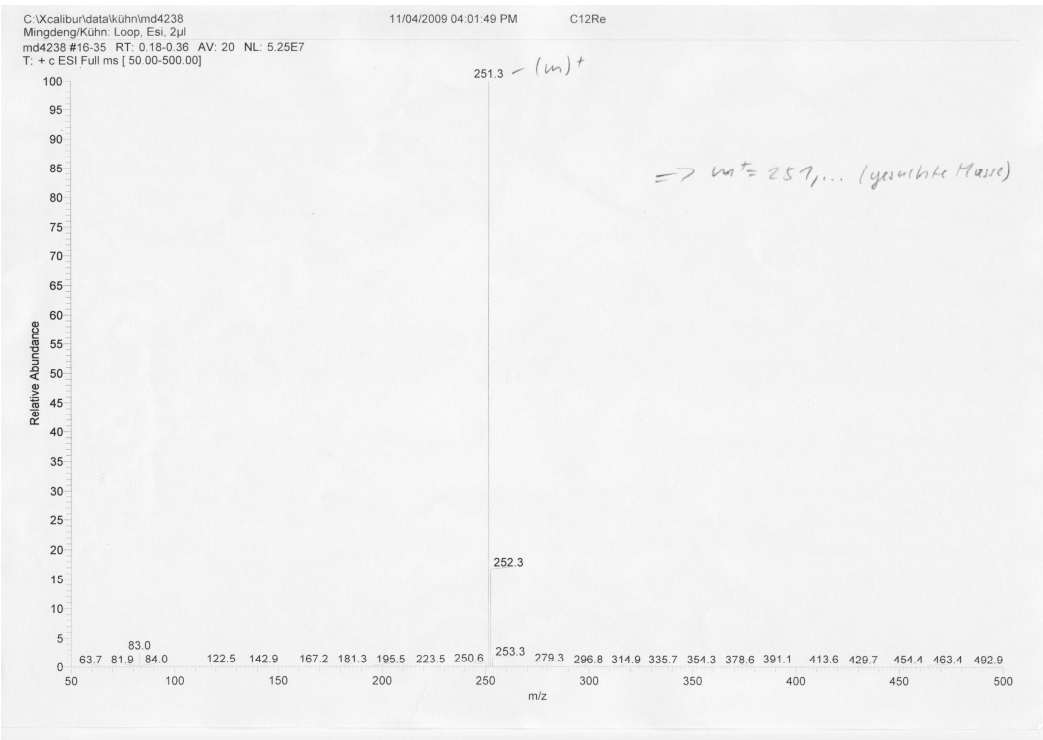


Figure S7-4 ESI-MS spectrum of IL **7**

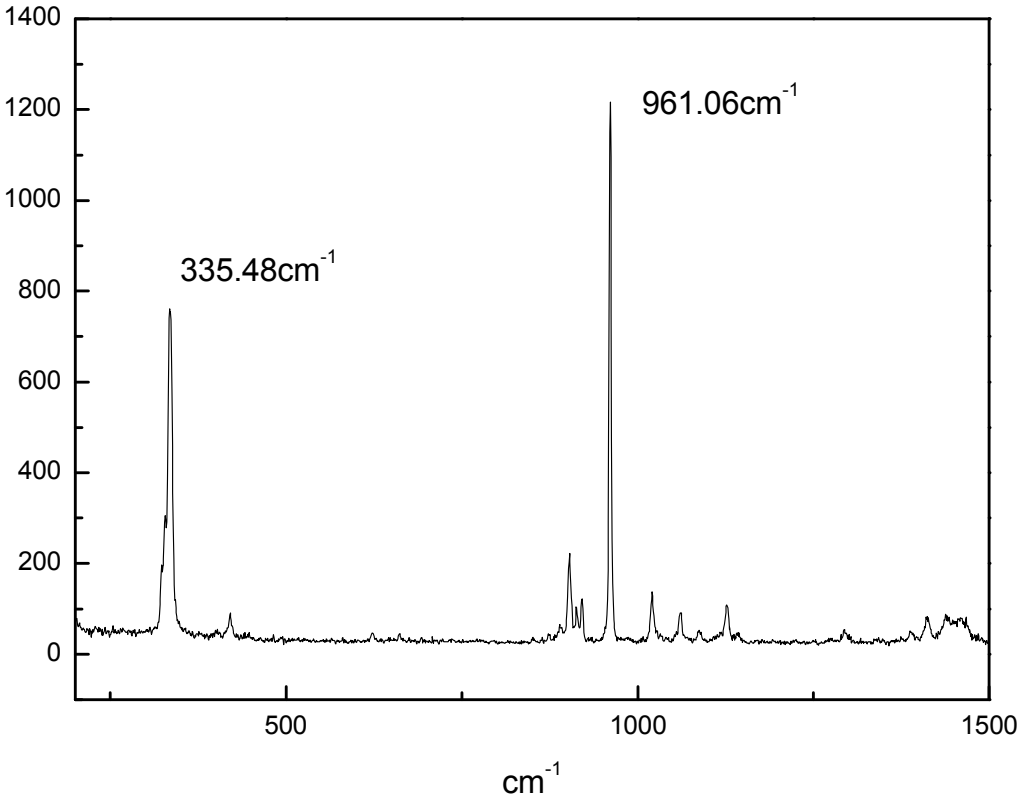


Figure S7-5 The Raman spectrum of IL**7**

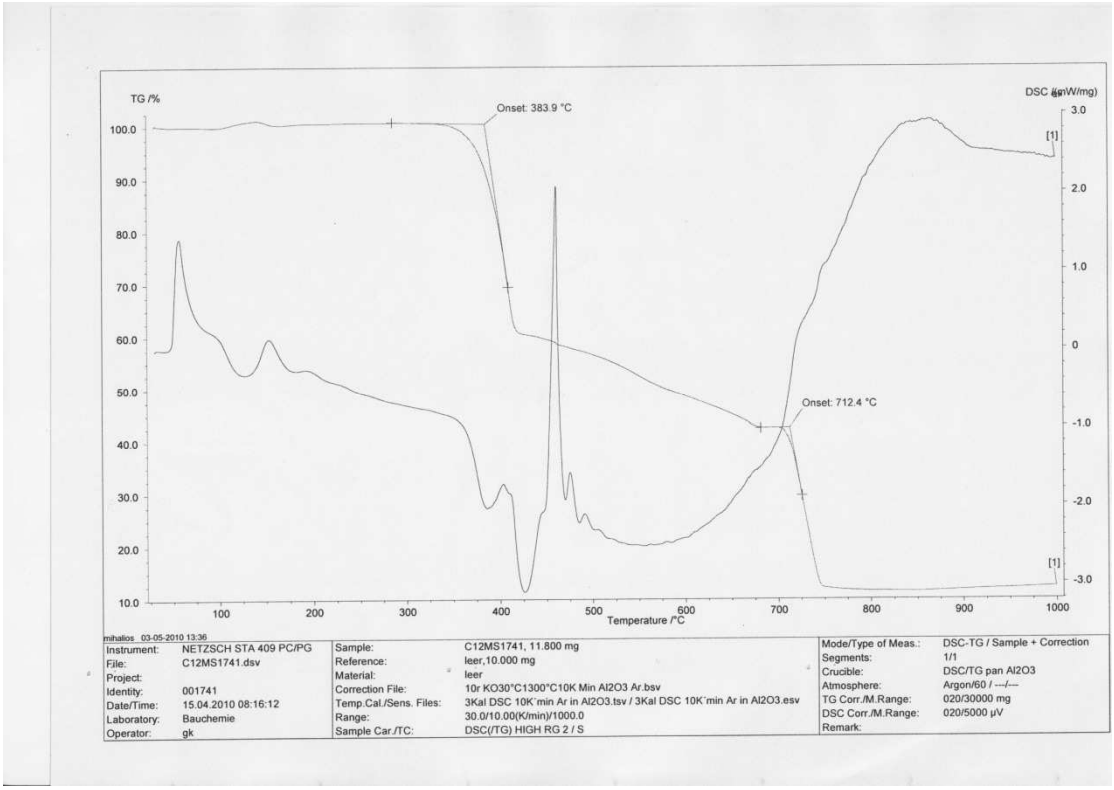


Figure S7-6 The TG-MS spectrum of IL **7**

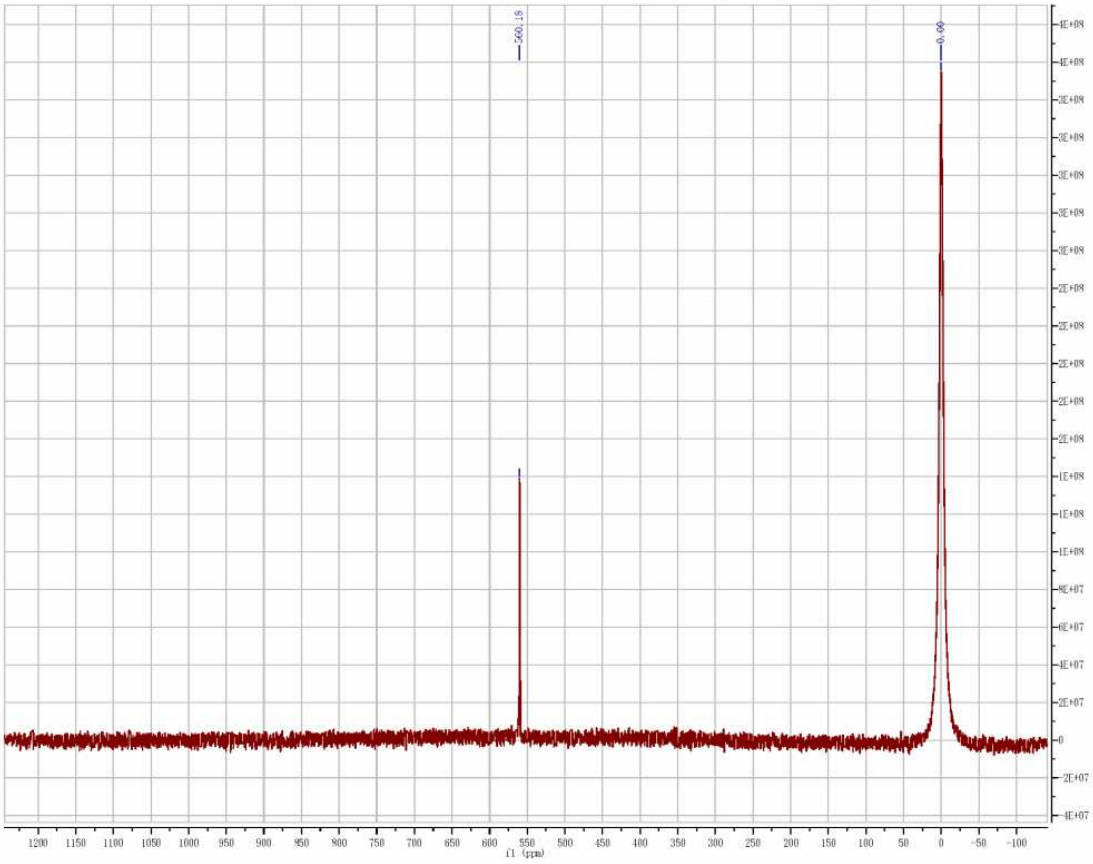


Figure S8-1 ^{17}O NMR (DMSO) spectrum of $[C_{12}mim][ReO_4]$before reaction

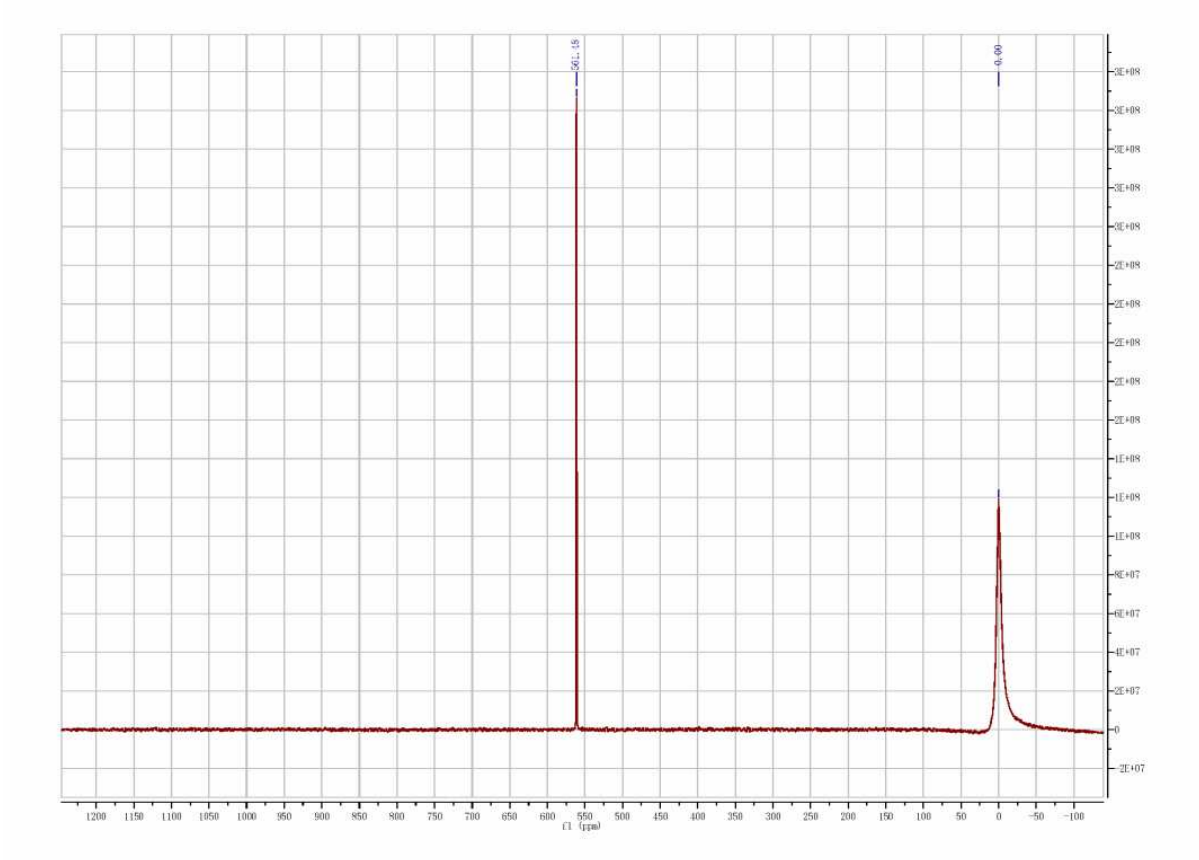


Figure S8-2 ^{17}O NMR (DMSO) spectrum of $[C_{12}mim][ReO_4]$ after reaction