Electronic supplementary information

Synthesis, characterization and evaluation of surface properties of cyclohexyloxyoxoethylbipyridinium gemini Ionic Liquids: A comparative study with single chain ionic liquids

Rajni Aggarwal, Sukhprit Singh*

Department of chemistry, UGC sponsored centre for advanced studies-I, Guru Nanak Dev University, Amritsar 143005 (India)

Tel.: +91 985 555 7324; fax: +91 183 225 8820.

E-mail address: sukhprit@gmail.com .

TABLE OF CONTENT

Characterization of Gemini Amphiphiles:

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)

Yield- 2.73 g (71.5%), T_m = 222 ^{0}C, IR stretching for C-O (1224 cm^{-1}), C-N (aromatic ring) 1357 cm^{-1}, C=C (aromatic ring) 1564 cm^{-1}, C-H (bending) 1455 cm^{-1}, C=N (aromatic ring) 1643 cm^{-1}, C=O (1747 cm^{-1}), C-H (stretching) 2930 cm^{-1}. ^{1}H NMR, δ ppm (400MHz; $CDCl_3$; Me_4Si) 1.24−1.81 (m, 20 H, 10 X CH_2), 4.78−4.84 (m, 2H, 2 X –CH-O-), 5.88 (S, 2H, -CO-CH_2), 5.96 (S, 2H, -CO-CH_2), 8.78-8.84 (dd, J = 6.9 Hz, 4H, 2 X H-3, 5 & 3', 5' of $C_{10}H_{10}N_2^{2+}$), 9.37-9.39 (d, J = 6.8 Hz, 2H, 1 X H-2, 6 of $C_{10}H_{10}N_2^{2+}$), 9.55-9.57 (d, J = 6.8 Hz, 2H, 1 X H-2', 6' of $C_{10}H_{10}N_2^{2+}$),

^{13}C NMR δ ppm (normal/DEPT-135; $CDCl_3$) 22.84-30.76 (-ve, hexyl ring CH_2), 59.86-60.29 (-ve, 2 X py^+ CH_2), 74.64-74.79 (+ve, 2 X -O-CH), 125.02-126.48 (+ve, C -3, C-5 & C -3', C-5' of $C_{10}H_{10}N_2^{2+}$), 146.87-147.33 (+ve, C-2, C-6 & C-2', C-6' of $C_{10}H_{10}N_2^{2+}$), 150.81 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 165.52-165.68 (-C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.1 (parent peak), (for $C_{18}H_{21}N_2O_2^{\cdot+}$) 297.0, (for $C_{12}H_{10}N_2O_2^{\cdot+}$) 214.9. Calculated (C% 61.30, H% 6.73, N% 5.50); found (C% 61.28, H% 6.71, N% 5.51).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium bromide (6b)

Yield- 3.55 g (79.3%), T_m = 220^{0}C, IR stretching for C-O (1225 cm^{-1}), C-N (aromatic ring) 1370 cm^{-1}, C=C (aromatic ring) 1565 cm^{-1}, C-H (bending) 1455 cm^{-1}, C=N (aromatic ring) 1644 cm^{-1}, C=O (1749 cm^{-1}), C-H (stretching) 2930 cm^{-1}. ^{1}H NMR δ ppm (400MHz; $CDCl_3$; Me_4Si) 1.21−1.83 (m, 20H, 10 X CH_2), 4.77−4.83 (m, 2H, 2 X –CH-O-), 5.91 (S, 4H, 2 X -CO-CH_2), 9.02-9.04 (d, J = 6.9 Hz, 4H, 2 X H-3, 5 & 3', 5' of $C_{10}H_{10}N_2^{2+}$), 9.50-9.52 (d, J = 6.8 Hz, 4H, 2 X H-2, 6 & 2', 6' of $C_{10}H_{10}N_2^{2+}$),

^{13}C NMR δ ppm (normal/DEPT-135; $CDCl_3$) 22.93-30.82 (-ve, hexyl ring CH_2), 60.36 (-ve, 2 X py^+ CH_2), 74.99 (+ve, 2 X -O-CH), 126.46 (+ve, C -3, C-5 & C -3', C-5' of $C_{10}H_{10}N_2^{2+}$), 147.29 (+ve, C-2, C-6 & C-2', C-6' of $C_{10}H_{10}N_2^{2+}$), 149.16 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 165.11 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.1 (parent peak), (for $C_{18}H_{21}N_2O_2^{\cdot+}$) 297.0, (for $C_{12}H_{10}N_2O_2^{\cdot+}$) 215.0. Calculated (C% 52.19, H% 5.73, N% 4.68); found (C% 52.17, H% 5.73, N% 4.67).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium chloride (7a)

Yield- 2.64 g (69.3%), T_m = 174^{0}C, IR stretching for C-O (1223 cm^{-1}), C-N (aromatic ring) 1354 cm^{-1}, C=C (aromatic ring) 1565 cm^{-1}, C-H (bending) 1445 cm^{-}1, C=N (aromatic ring) 1622 cm^{-1}, C=O (1741 cm^{-1}), C-H (stretching) 2943 cm^{-1}. ^{1}H NMR δ ppm (400MHz; $CDCl_3$; Me_4Si) 1.51−1.72 (m, 20H, 10 X CH_2), 4.70−4.74 (m, 2H, 2 X –CH-O-), 6.01 (S, 4H, 2 X -CO-CH_2), 7.90−7.92 (t, J = 7.6 Hz, 2H, C-5 &

C-5' of $C_{10}H_{10}N_2^{2+}$), 7.98-8.01 (d, J = 7.4 Hz 2H, C-3 & C-3' of $C_{10}H_{10}N_2^{2+}$), 8.26−8.28 (t, J = 6.3 Hz, 2H, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 8.64−8.65 (d, J = 4.3 Hz, 2H, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$)

^{13}C NMR δ ppm (normal/DEPT-135; $CDCl_3$) 23.44-31.21 (-ve, hexyl ring CH_2), 60.49 (-ve, 2 X py^+ CH_2), 75.51-76.83 (+ve, 2 X -O-CH), 126.04-126.62 (+ve, C -3 & C-3' of $C_{10}H_{10}N_2^{2+}$) 127.49-127.82 (+ve, C-5 & C-5' of $C_{10}H_{10}N_2^{2+}$), 138.62 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 149.48-149.54 (+ve, C-2 & C-2' of $C_{10}H_{10}N_2^{2+}$), 150.41-152.04 (+ve, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$), 165.35 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.24 (parent peak), (for $C_{26}H_{34}N_2O_4^{2+}$+1) 438.25, (for $C_{26}H_{34}N_2O_4^{2+}$+2) 439.25, (for $C_{26}H_{34}N_2O_4^{2+}$+3) 440.25. Calculated (C% 61.30, H% 6.73, N% 5.50); found (C% 61.30, H% 6.71, N% 5.49).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium bromide (7b)

Yield- 3.38g (75.6%), T_m = 160^{0}C, IR stretching for C-O (1224 cm^{-1}), C-N (aromatic ring) 1354 cm^{-1}, C=C (aromatic ring) 1565 cm^{-1}, C-H (bending) 1445 cm^{-1}, C=N (aromatic ring) 1622 cm^{-1}, C=O (1741 cm^{-1}), C-H (stretching) 2942 cm^{-1}. ^{1}H NMR δ ppm (400MHz; $CDCl_3$; Me_4Si) δ_H ppm 1.03−1.98 (m, 20H, 10 X CH_2), 4.60−4.64 (m, 2H, 2 X –CH-O-), 5.78 (S, 4H, 2 X -CO-CH_2), 7.91−7.93 (t, J = 6.5 Hz 2H, C-5 & C-5' of $C_{10}H_{10}N_2^{2+}$), 8.03−8.06 (d, J = 6.4 Hz 2H, C-3 & C-3' of $C_{10}H_{10}N_2^{2+}$), 8.30−8.34 (t, J = 7.8 Hz, 2H, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 8.75-8.79 (d, J = 7.1 Hz, 2H, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$)

^{13}C NMR δ ppm (normal/DEPT-135; $CDCl_3$) 23.31-30.90 (-ve, hexyl ring CH_2), 59.97-60.36 (-ve, 2 X py^+ CH_2), 74.69-75.36 (+ve, 2 X -O-CH), 126.07-126.17 (+ve, C -3 & C-3' of $C_{10}H_{10}N_2^{2+}$), 127.32-127.41 (+ve, C-5 & C-5' of $C_{10}H_{10}N_2^{2+}$), 138.64-138.71 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 147.64-147.79 (+ve, C-2 & C-2' of $C_{10}H_{10}N_2^{2+}$), 149.25-149.47 (+ve, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$), 164.99 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.2 (parent peak), (for $C_{26}H_{34}N_2O_4^{2+}$+1) 438.2, (for $C_{18}H_{21}N_2O_2)^{\cdot+}$ 298.0. Calculated (C% 52.19, H% 5.73, N% 4.68); found (C% 52.15, H% 5.71, N% 4.67).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium hexafluorophosphate (11a)

Yield- 4.74g (86.97%), T_m = 180^{0}C, IR stretching for C-O (1229 cm^{-1}), C-N (aromatic ring) 1362 cm^{-1}, C=C (aromatic ring) 1565 cm^{-1}, C-H (bending) 1454 cm^{-1}, C=N (aromatic ring) 1646 cm^{-1}, C=O (1751 cm^{-1}), C-H (stretching) 2936 cm^{-1}. ^{1}H NMR δ ppm (300MHz; DMSO-d^6) 1.33−1.98 (m, 20H, 10 X CH_2), 4.81−4.86 (m, 2H, 2 X –CH-O-), 5.69 (S, 4H, 2 X -CO-CH_2), 8.79-8.81 (d, J = 7.0 Hz, 4H, 2 X H-3, 5 & 3', 5' of $C_{10}H_{10}N_2^{2+}$), 9.31-9.33 (d, J = 6.9 Hz, 4H, 2 X H-2, 6 & 2', 6' of $C_{10}H_{10}N_2^{2+}$),

^{13}C NMR δ ppm (normal/DEPT-135; DMSO-d^6) 23.13-31.42 (-ve, hexyl ring CH_2), 61.13 (-ve, 2 X py^+ CH_2), 75.09 (+ve, 2 X -O-CH), 127.42 (+ve, C -3, C-5 & C -3', C-5' of $C_{10}H_{10}N_2^{2+}$), 148.19 (+ve, C-2, C-6 & C-2', C-6' of $C_{10}H_{10}N_2^{2+}$), 150.66 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 165.09 (2 X -C=O). MS (ESI)

(relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.1 (parent peak), (for $C_{18}H_{21}N_2O_2^{\cdot+}$) 297.0, Calculated (C% 42.87, H% 4.70, N% 3.85); found (C% 42.84, H% 4.73, N% 3.77).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium tetrafluoroborate (11b)

Yield- 3.10g (67.68%), T_m = 174^{0}C, IR stretching for C-O (1230 cm^{-1}), C-N (aromatic ring) 1357 cm^{-1}, C=C (aromatic ring) 1564 cm^{-1}, C-H (bending) 1457 cm^{-1}, C=N (aromatic ring) 1618 cm^{-1}, C=O (1746 cm^{-1}), C-H (stretching) 2935 cm^{-1}. ^{1}H NMR δ ppm (300MHz; DMSO-d^6) δ_H ppm 1.24−1.90 (m, 20H, 10 X CH_2), 4.80−4.88 (m, 2H, 2 X –CH-O-), 5.79 (S, 4H, 2 X -CO-CH_2), 8.83-8.96 (d, J = 6.8 Hz, 4H, 2 X H-3, 5 & 3', 5' of $C_{10}H_{10}N_2^{2+}$), 9.33-9.44 (d, J = 7.0 Hz, 4H, 2 X H-2, 6 & 2', 6' of $C_{10}H_{10}N_2^{2+}$),

^{13}C NMR δ ppm (normal/DEPT-135; DMSO-d^6) 22.55-30.66 (-ve, hexyl ring CH_2), 57.61 (-ve, 2 X py^+ CH_2), 75.09 (+ve, 2 X -O-CH), 126.46 (+ve, C -3, C-5 & C -3', C-5' of $C_{10}H_{10}N_2^{2+}$), 147.27 (+ve, C-2, C-6 & C-2', C-6' of $C_{10}H_{10}N_2^{2+}$), 149.16 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 161.62 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.1 (parent peak), (for $C_{18}H_{21}N_2O_2^{\cdot+}$) 297.0. Calculated (C% 51.09, H% 5.60, N% 4.58); found (C% 51.05, H% 5.61, N% 4.56).

1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium hexafluorophosphate (12a)

Yield- 4.15g (76.14%), T_m = 119^{0}C, IR stretching for C-O (1230 cm^{-1}), C-N (aromatic ring) 1360 cm^{-1}, C=C (aromatic ring) 1578 cm^{-1}, C-H (bending) 1451 cm^{-1}, C=N (aromatic ring) 1631 cm^{-1}, C=O (1753 cm^{-1}), C-H (stretching) 2947 cm^{-1}. ^{1}H NMR δ ppm (300MHz; DMSO-d^6) δ_H ppm 1.26−1.77 (m, 20H, 10 X CH_2), 4.75−4.82 (m, 2H, 2 X –CH-O-), 5.56 (S, 4H, 2 X -CO-CH_2), 8.13−8.17 (d, J = 4.8 Hz, 2H, C-5 & C-5' of $C_{10}H_{10}N_2^{2+}$), 8.46−8.50 (t, J = 7.4 Hz, 2H, C-3 & C-3' of $C_{10}H_{10}N_2^{2+}$), 8.82−8.86 (d, J = 6.3 Hz, 2H, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 9.20-9.23 (t, J = 9.2 Hz, 2H, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$)

^{13}C NMR δ ppm (normal/DEPT-135; DMSO-d^6) 22.30-30.45 (-ve, hexyl ring CH_2), 59.37-60.01 (-ve, 2 X py^+ CH_2), 73.65-74.16 (+ve, 2 X -O-CH), 126.04-126.15 (+ve, C -3 & C-3' of $C_{10}H_{10}N_2^{2+}$), 127.31-127.51 (+ve, C-5 & C-5' of $C_{10}H_{10}N_2^{2+}$), 130.04-130.52 (+ve, C-4 & C-4' of $C_{10}H_{10}N_2^{2+}$), 138.34-138.39 (+ve, C-2 & C-2' of $C_{10}H_{10}N_2^{2+}$), 146.25-146.47 (+ve, C-6 & C-6' of $C_{10}H_{10}N_2^{2+}$), 165.01 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.2 (parent peak), (for $C_{26}H_{34}N_2O_4^{2+}$+1) 438.2, (for $C_{18}H_{21}N_2O_2)^{\cdot+}$ 298.0. Calculated (C% 42.87, H% 4.70, N% 3.85); found (C% 42.81, H% 4.67, N% 3.82).

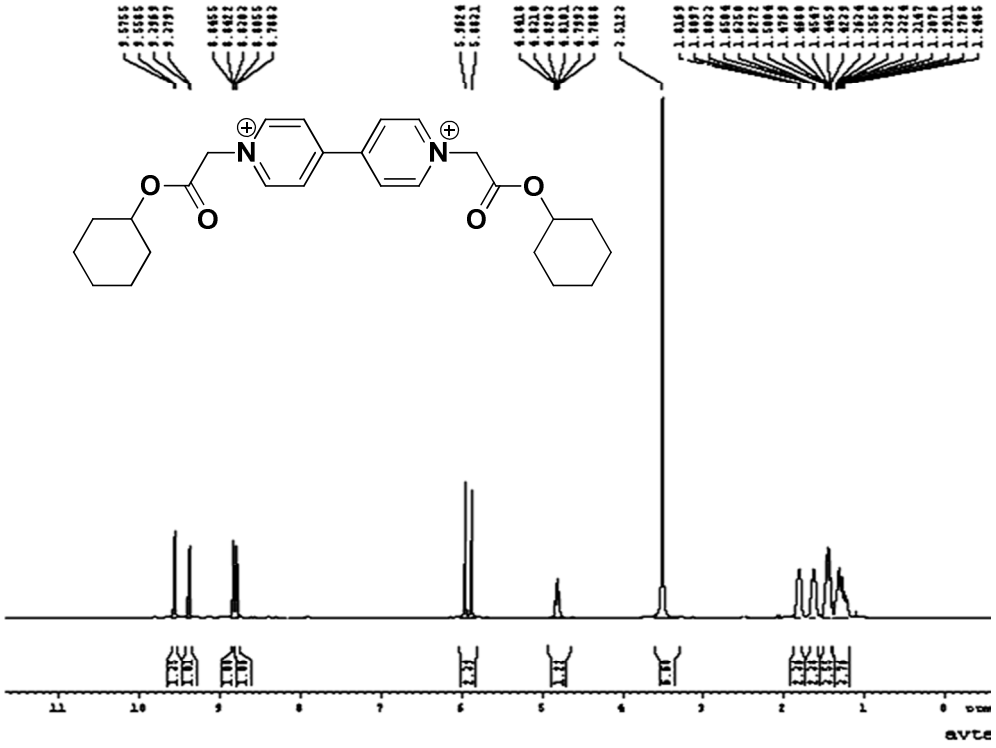
1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium tetrafluoroborate (12b)

Yield- 3.01g (65.63%), T_m = 115^{0}C, IR stretching for C-O (1225 cm^{-1}), C-N (aromatic ring) 1358 cm^{-1}, C=C (aromatic ring) 1567 cm^{-1}, C-H (bending) 1449 cm^{-1} C=N (aromatic ring) 1624 cm^{-1}, C=O (1752 cm^{-1}), C-H (stretching) 2942 cm^{-1}. ^{1}H NMR δ ppm (300MHz; DMSO-d^6) 1.22−1.78 (m, 20H, 10 X CH_2),

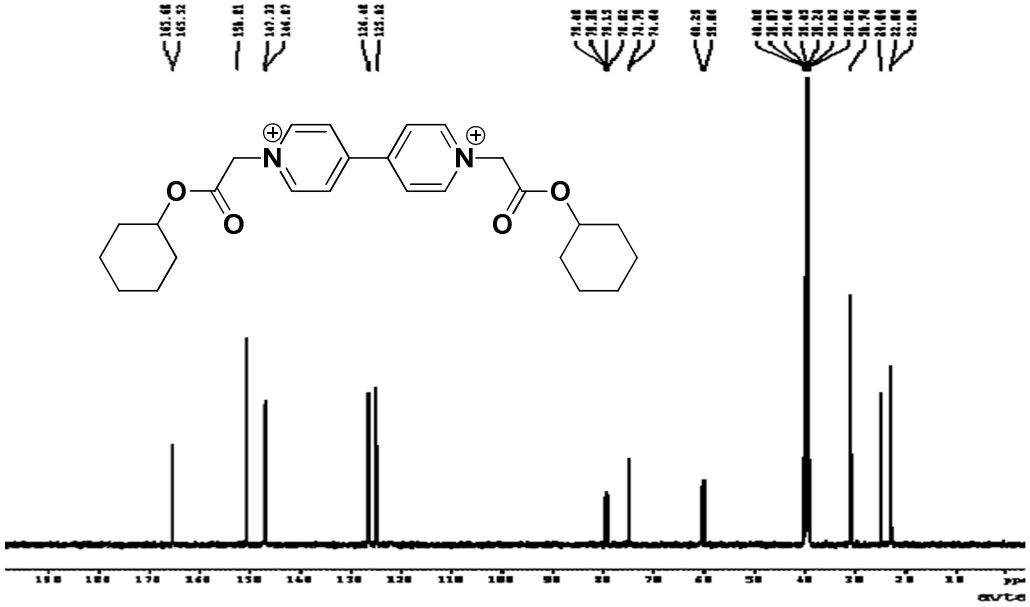
4.79−4.83 (m, 2H, 2 X –CH-O-), 5.55 (S, 4H, 2 X -CO-CH_2), 8.15−8.20 (t, J = 6.5 Hz 2H, C-5 & C-5’ of $C_{10}H_{10}N_2^{2+}$), 8.46−8.48 (d, J = 6.4 Hz, 2H, C-3 & C-3’ of $C_{10}H_{10}N_2^{2+}$), 8.82-8.87(t, J = 7.8 Hz, 2H, C-4 & C-4’ of $C_{10}H_{10}N_2^{2+}$), 9.17-9.19 (d, J = 7.1 Hz, 2H, C-6 & C-6’ of $C_{10}H_{10}N_2^{2+}$)

^{13}C NMR δ ppm (normal/DEPT-135; DMSO-d^6) 22.55-30.66 (-ve, hexyl ring CH_2), 59.86-60.06 (-ve, 2 X py^+ CH_2), 74.12-74.22 (+ve, 2 X -O-CH), 126.07-126.17 (+ve, C -3 & C-3’ of $C_{10}H_{10}N_2^{2+}$), 127.48-127.75 (+ve, C-5 & C-5’ of $C_{10}H_{10}N_2^{2+}$), 130.12-130.71 (+ve, C-4 & C-4’ of $C_{10}H_{10}N_2^{2+}$), 138.58-138.67 (+ve, C-2 & C-2’ of $C_{10}H_{10}N_2^{2+}$), 147.46-147.66 (+ve, C-6 & C-6’ of $C_{10}H_{10}N_2^{2+}$), 165.53 (2 X -C=O). MS (ESI) (relative intensity %): *m/z*, (for $C_{26}H_{34}N_2O_4^{2+}$) 437.2 (parent peak), (for $C_{26}H_{34}N_2O_4^{2+}$+1) 438.2, (for $C_{18}H_{21}N_2O_2)^{\cdot+}$ 298.0. Calculated (C% 51.09, H% 5.60, N% 4.58); found (C% 51.11, H% 5.53, N% 4.52).

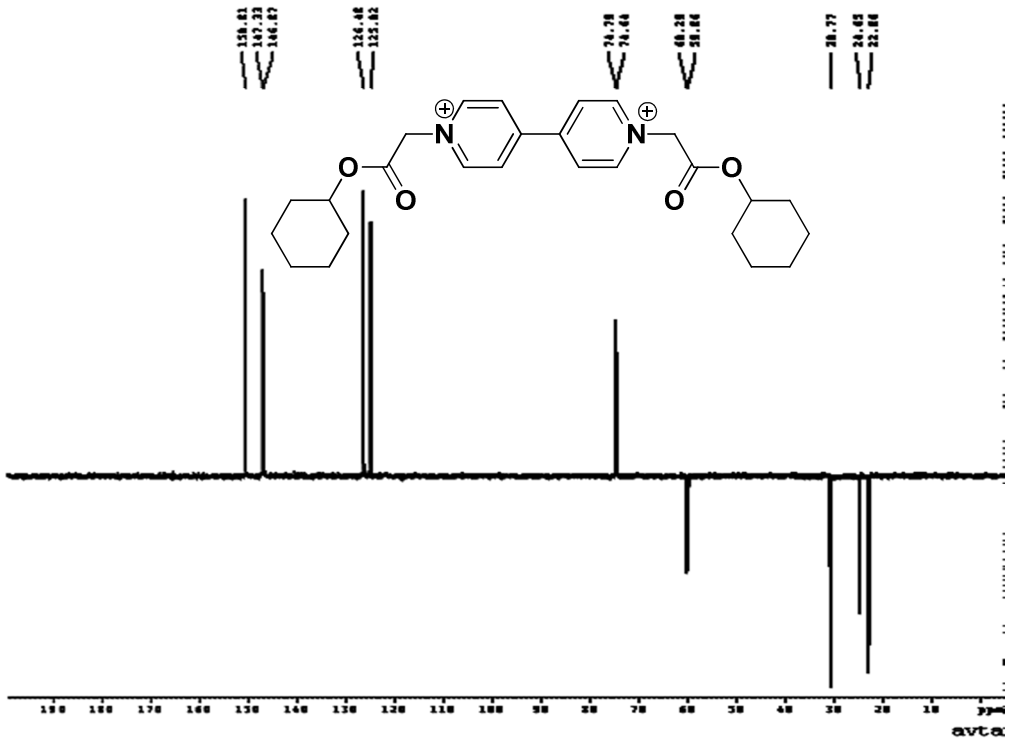
Graphs of NMR, IR and Mass spectra of ionic liquids



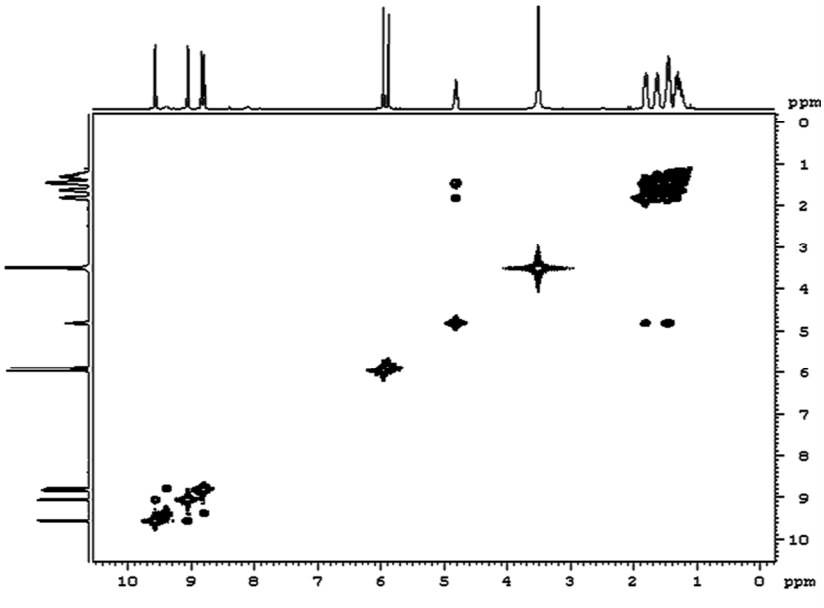
^{1}H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



^{13}C spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



^{13}C DEPT spectra of1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



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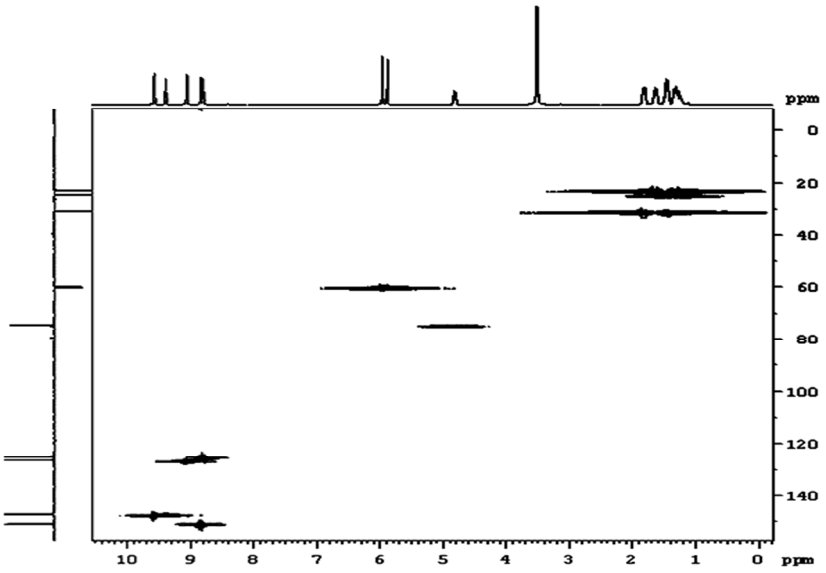
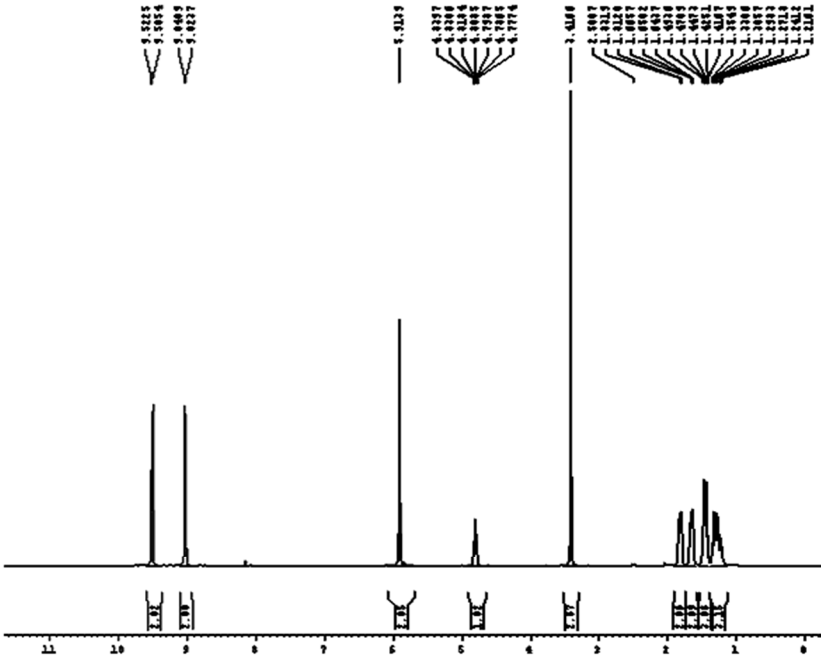
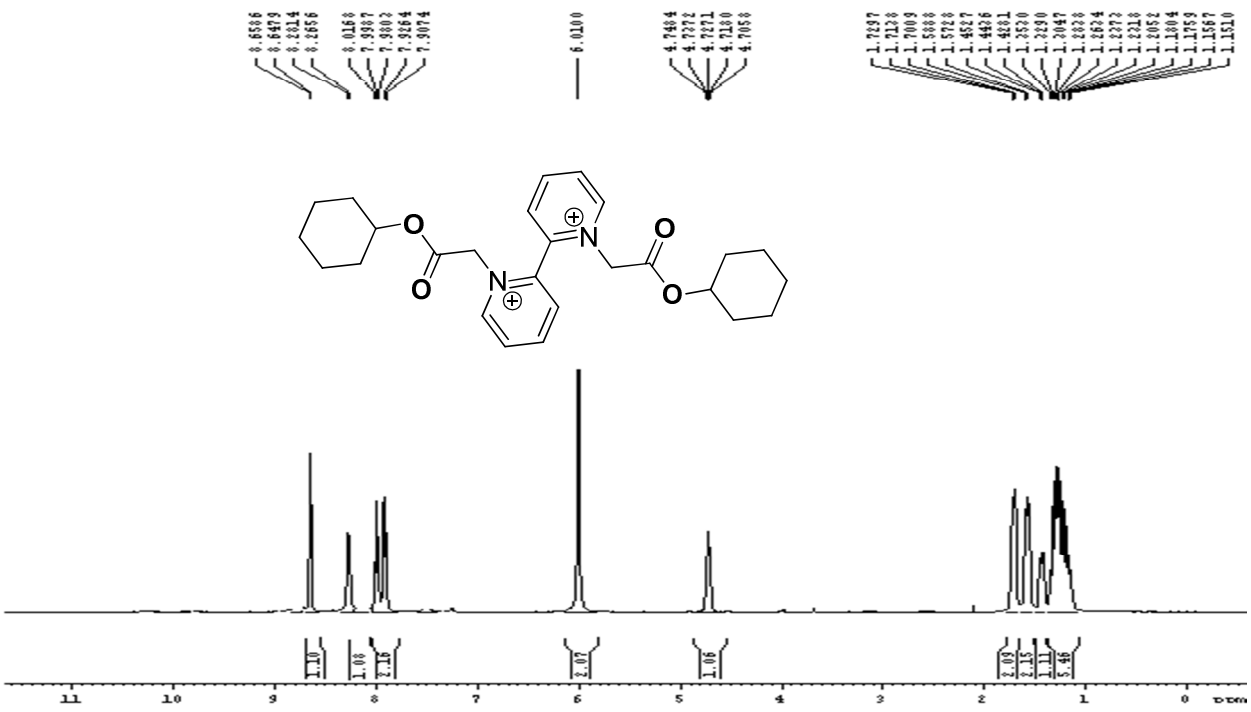


^{1}H- ^{13}C 2D spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



^{1}H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium bromide (6b)



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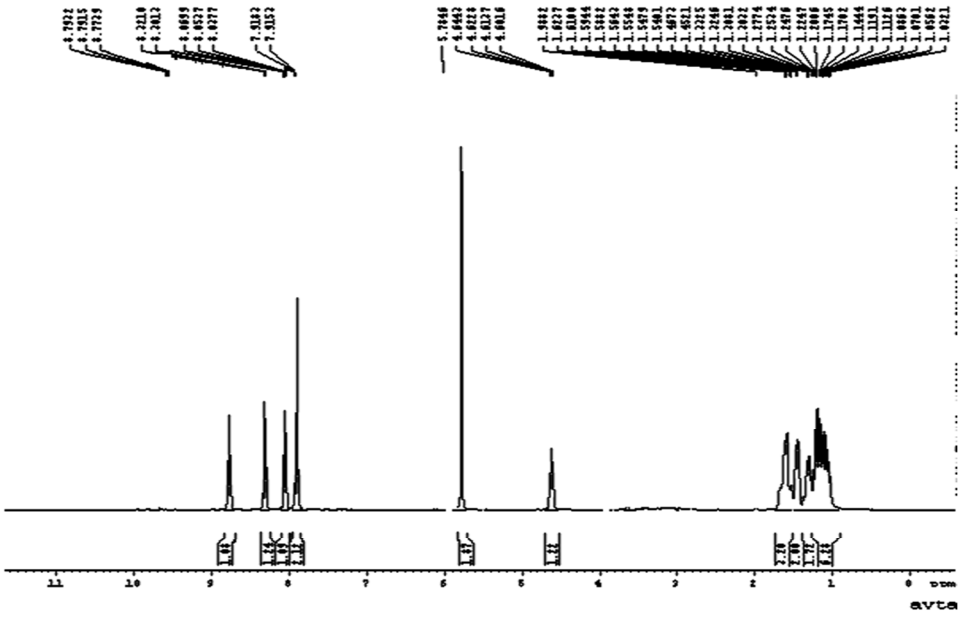
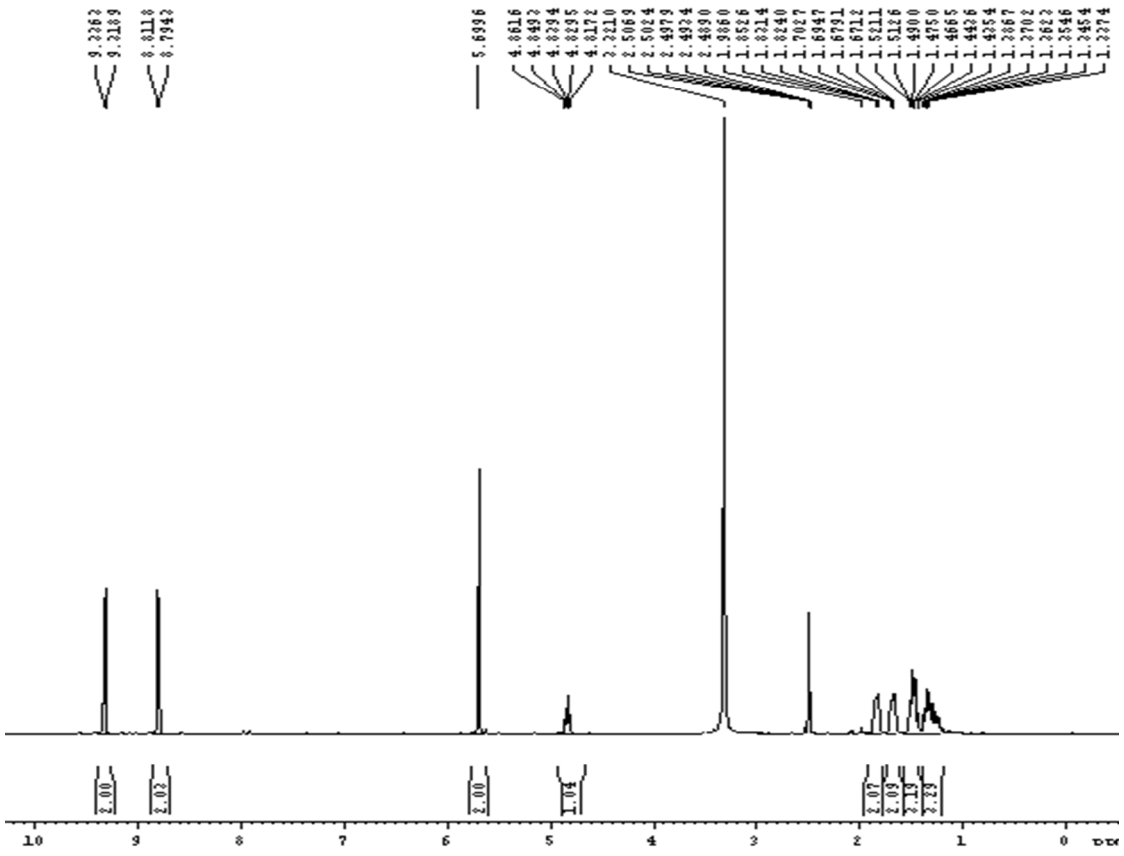
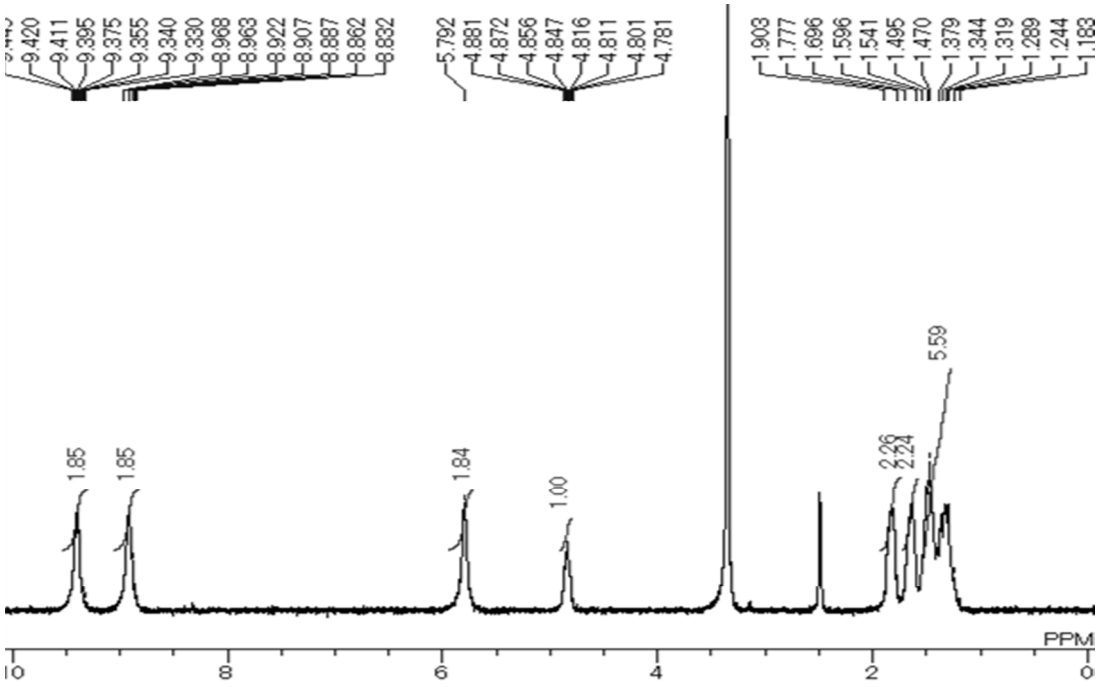


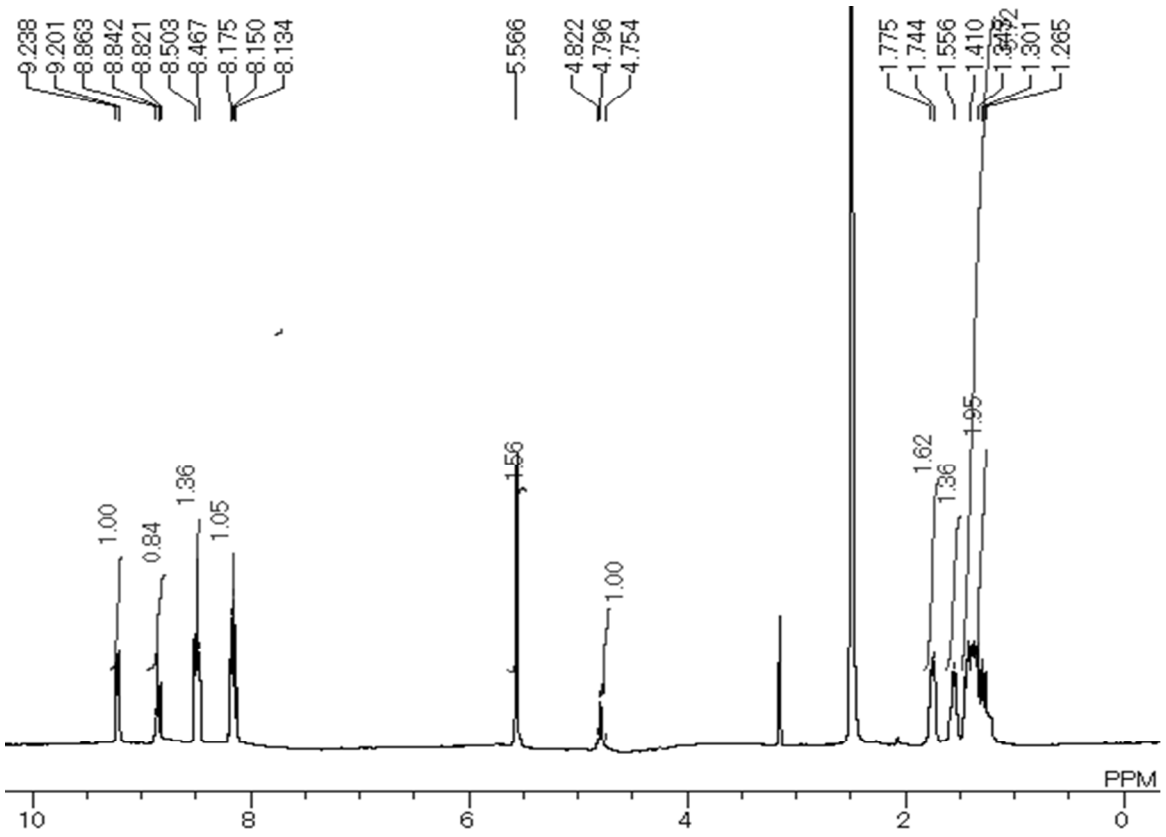
^{1}H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium bromide (7b)



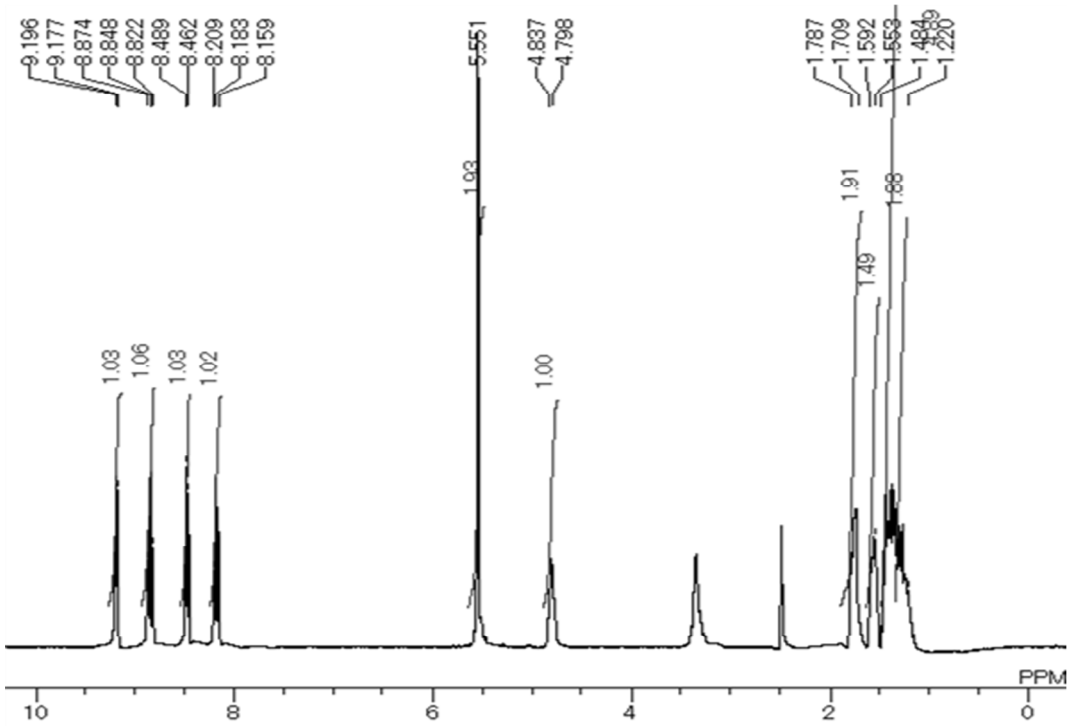
[1]H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium hexafluorophosphate (11a)



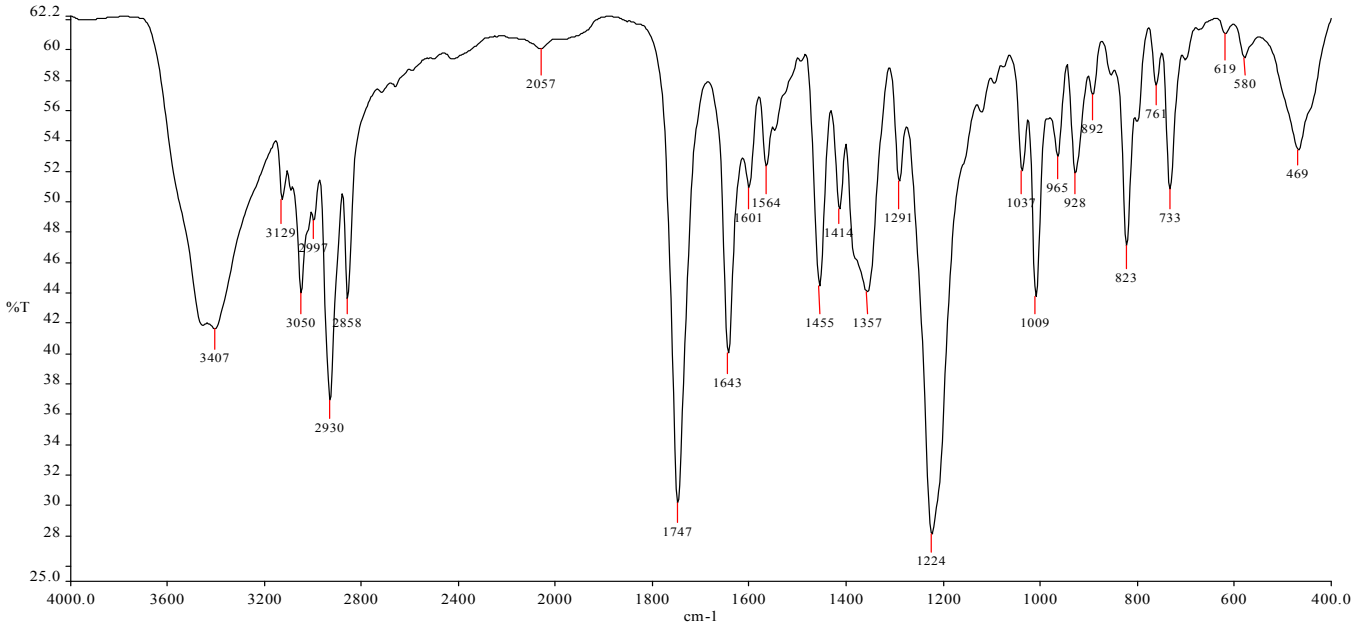
[1]H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium tetrafluoroborate (11b)



[1]H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium hexafluorophosphate (12a)

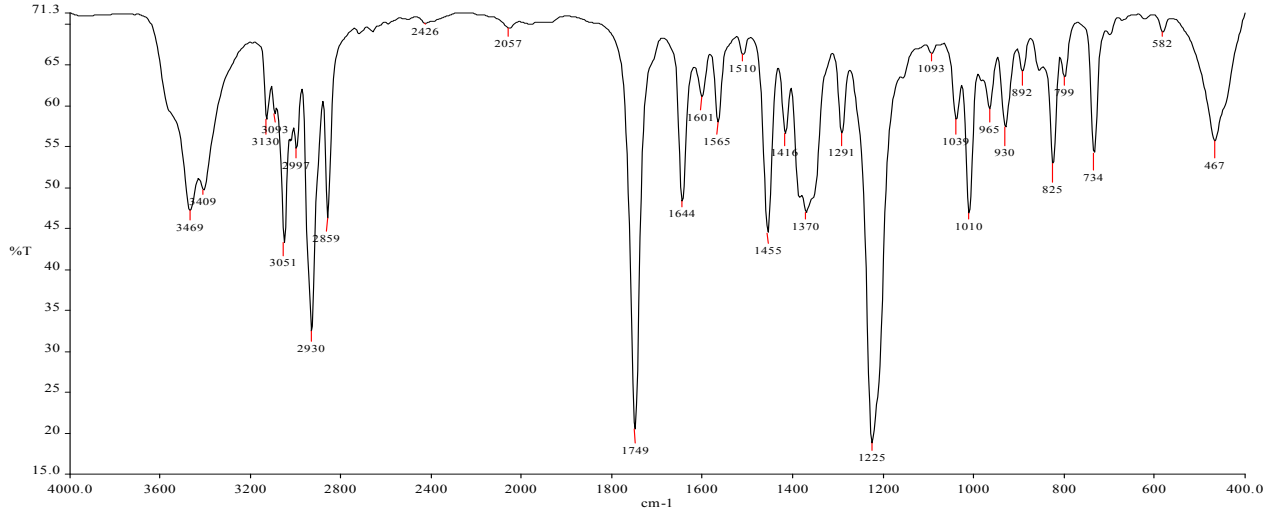


[1]H spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium tetrafluoroborate (12b)

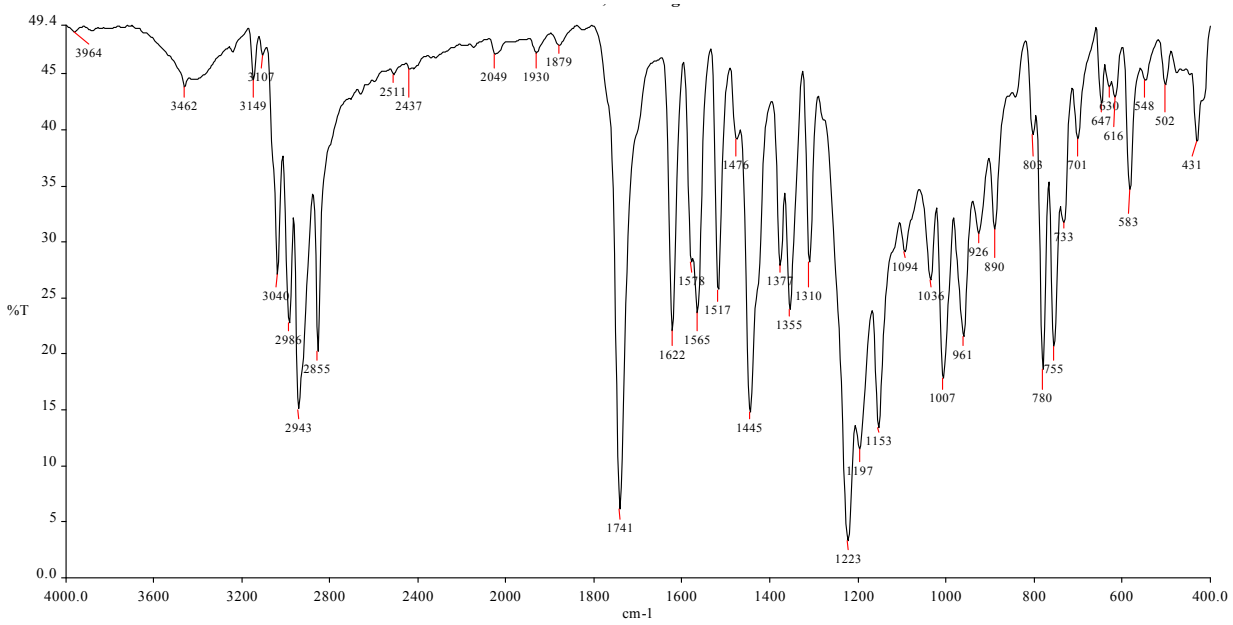


Spectrum Name: Rajani Aggarwal-4.sp Description: CAP-4

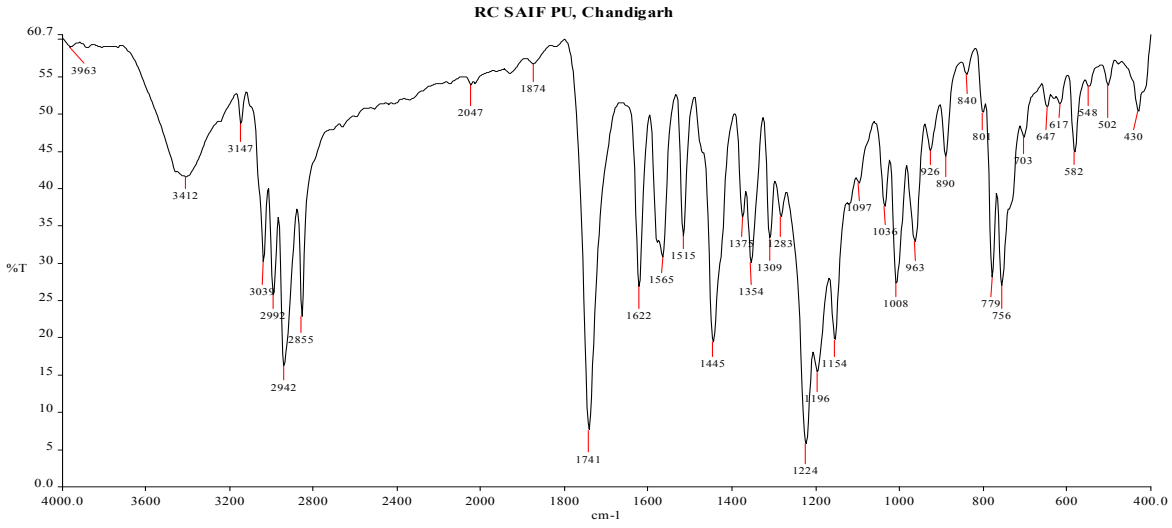
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



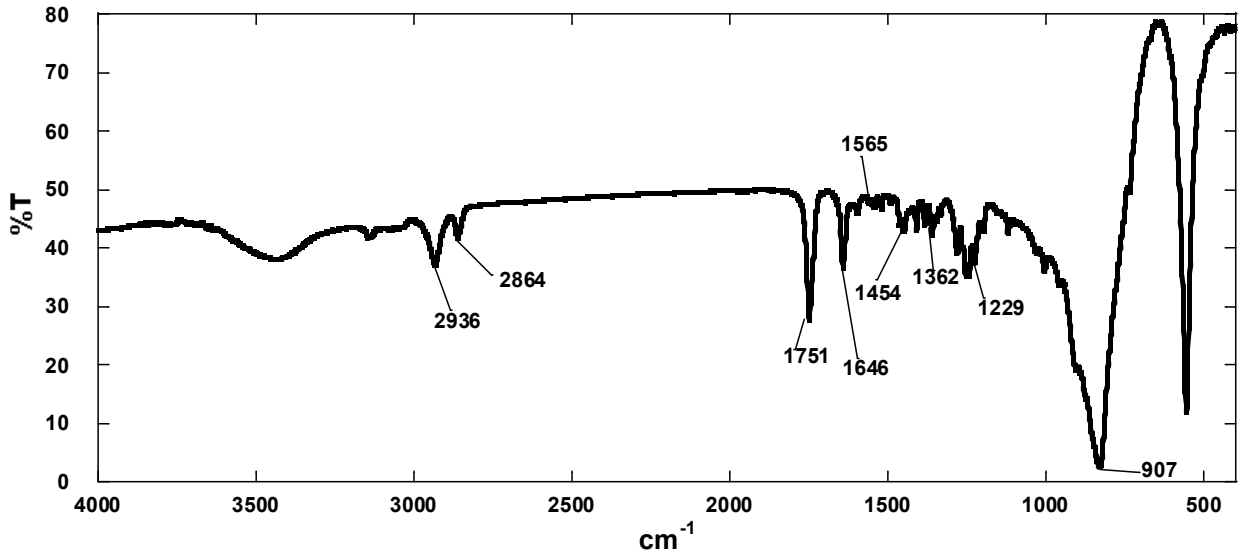
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium bromide (6b)



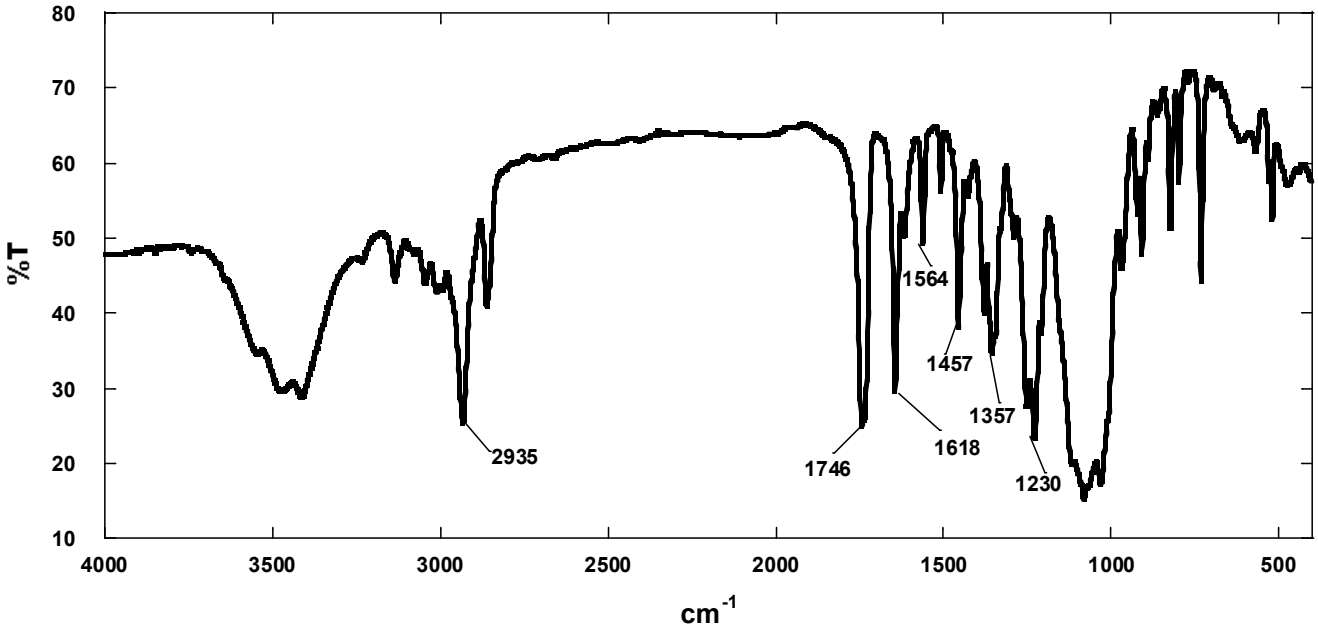
IR spectra of 1,1’-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2’-bipyridin]-1,1’-diium chloride (7a)



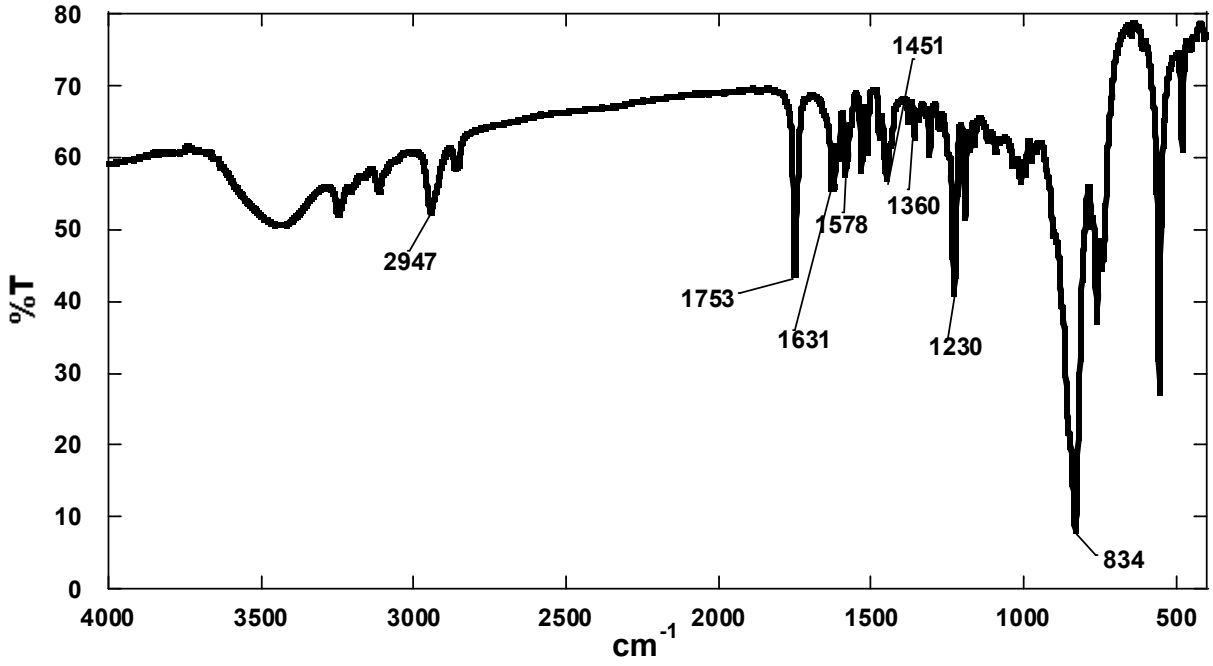
IR spectra of 1,1’-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2’-bipyridin]-1,1’-diium bromide (7b)



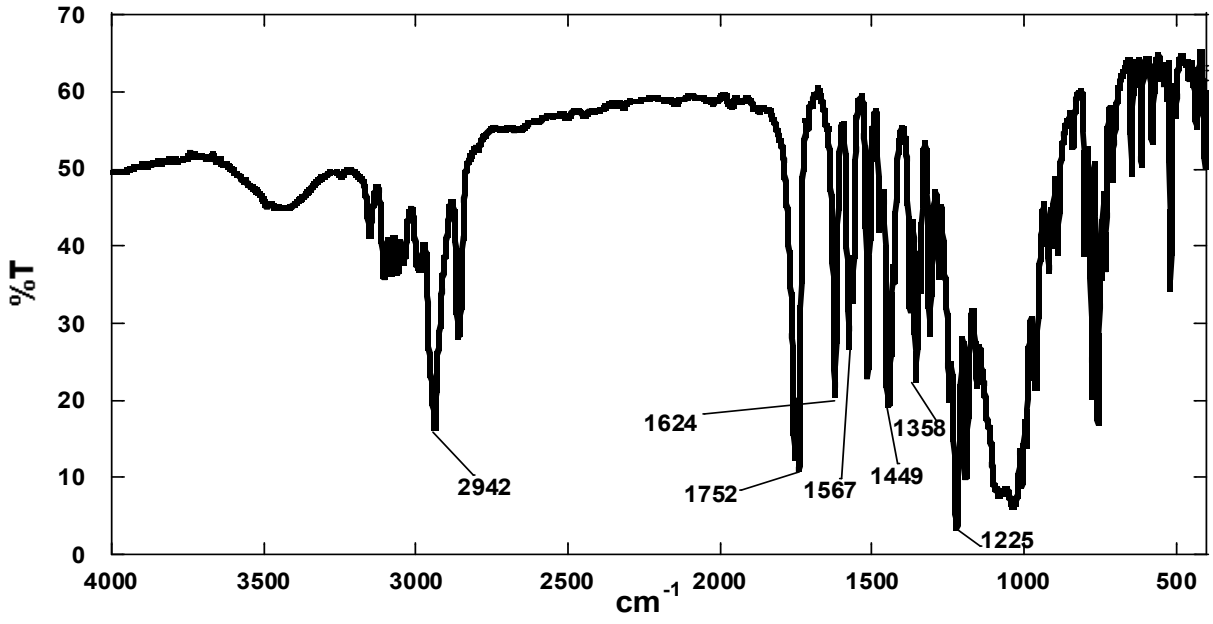
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium hexafluorophosphate (11a)



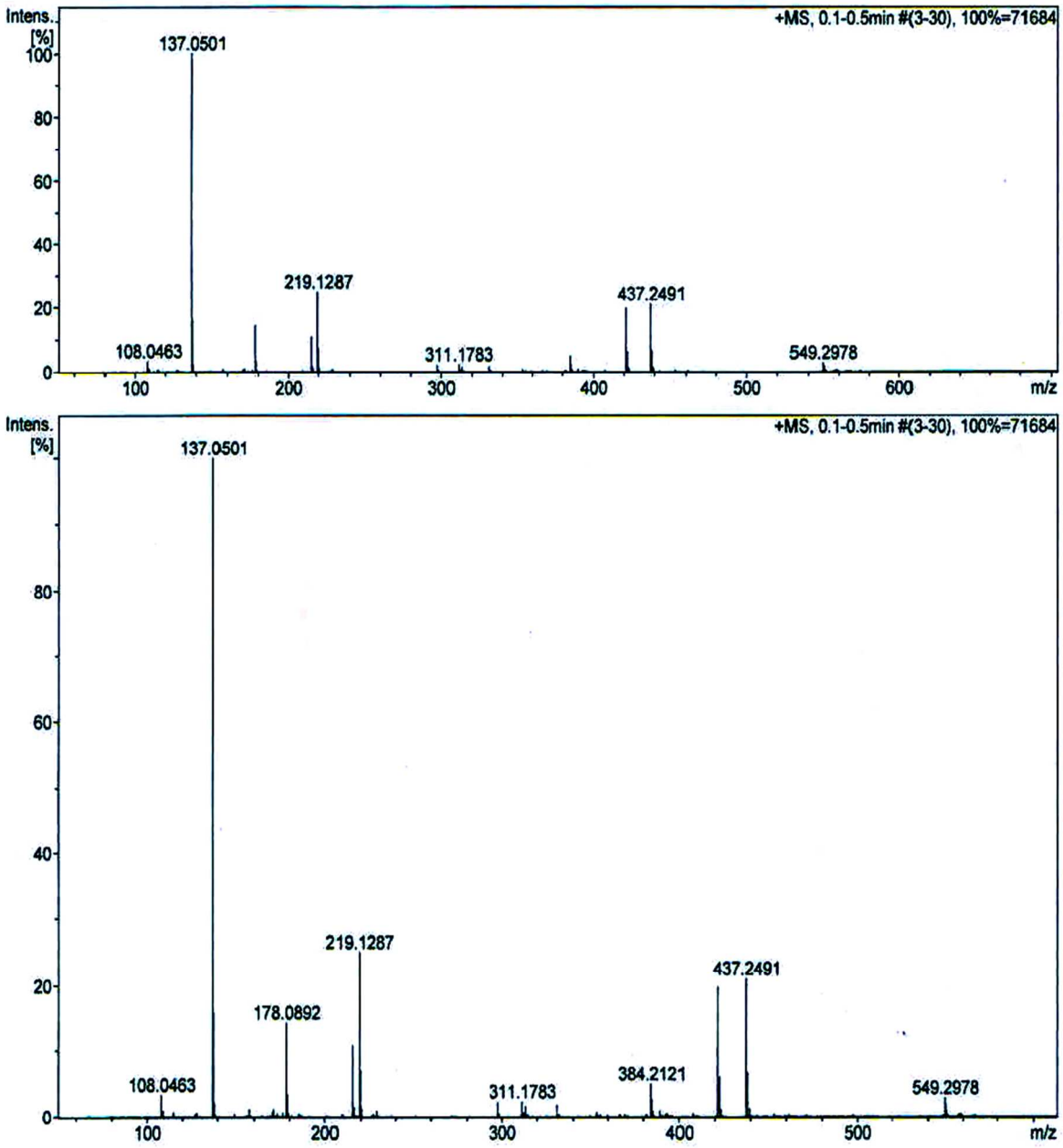
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium tetrafluoroborate (11b)



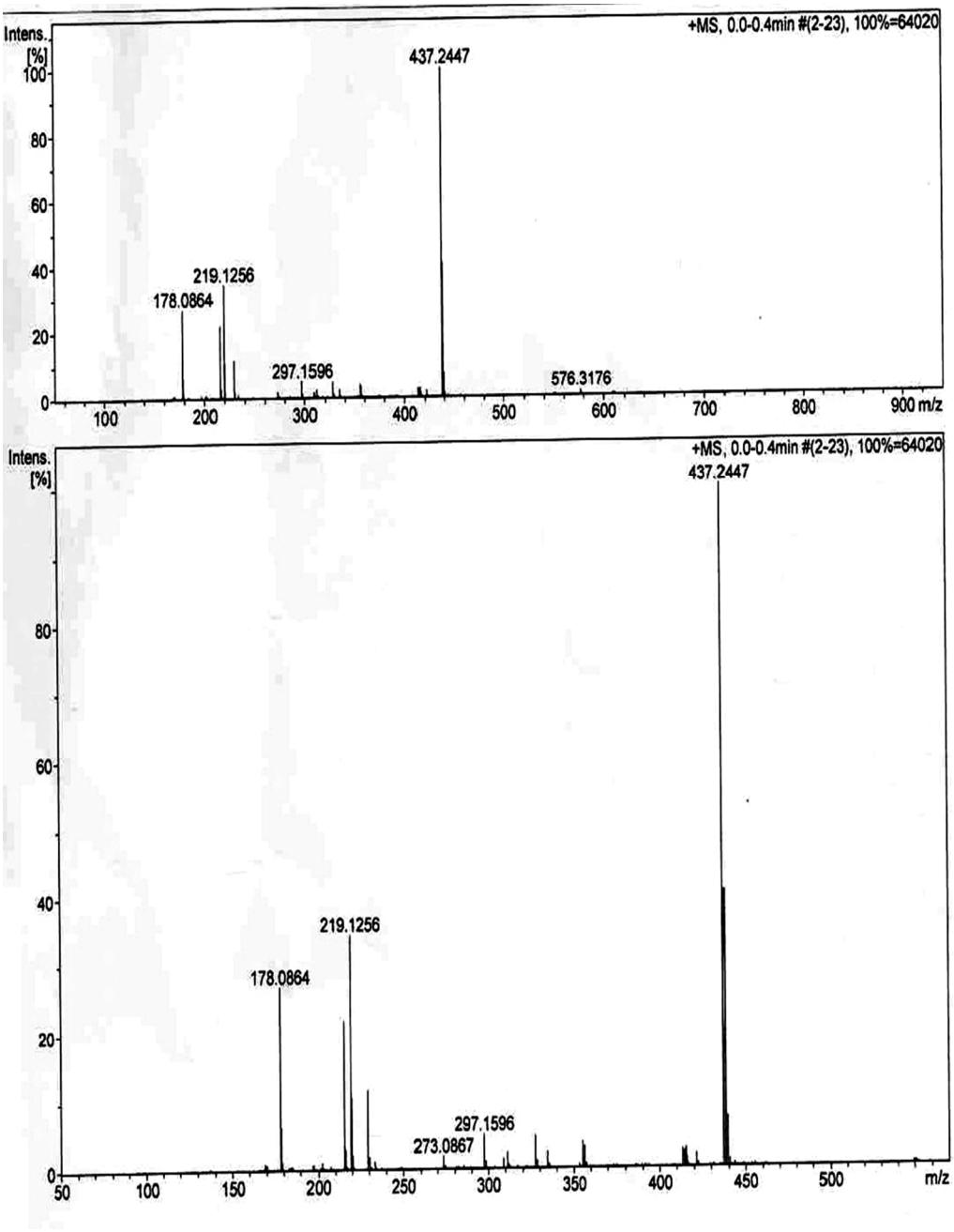
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium hexafluorophosphate (12a)



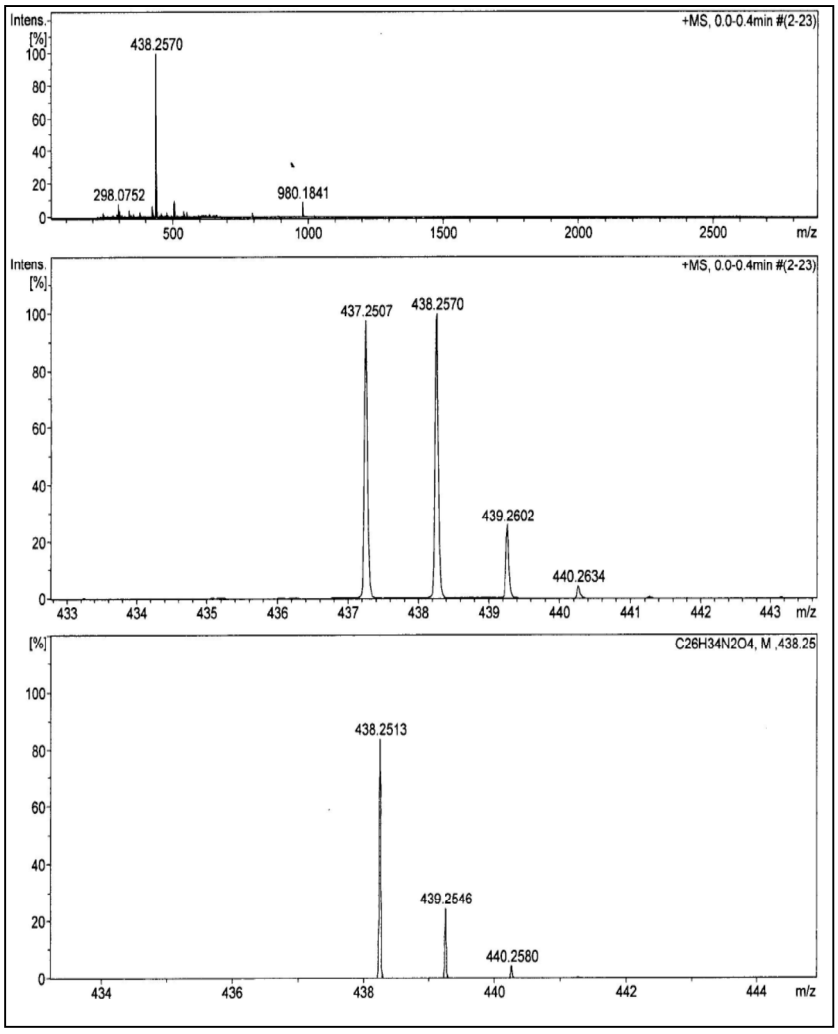
IR spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium tetrafluoroborate (12b)



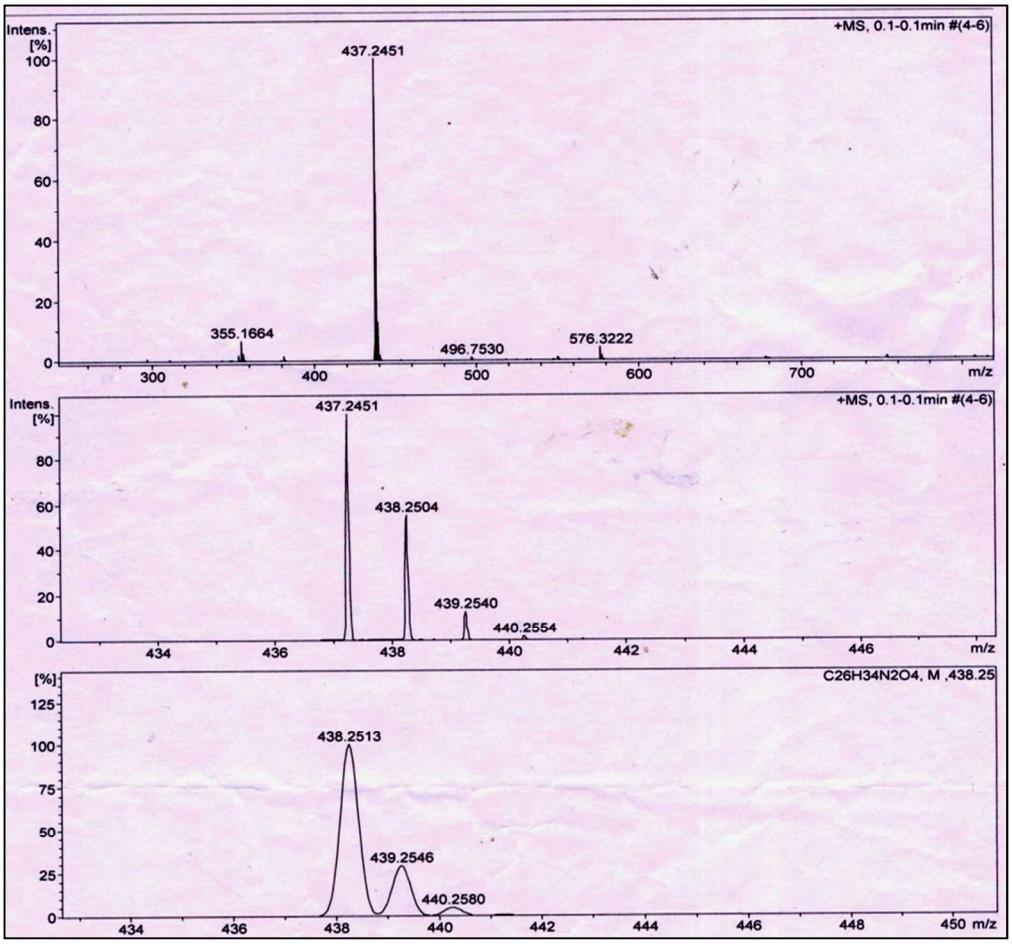
Mass spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4'-bipyridin]-1,1'-diium chloride (6a)



Mass spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[4,4''-bipyridin]-1,1'-diium bromide (6b)



Mass spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium chloride (7a)



Mass spectra of 1,1'-bis-(2-(cyclohexyloxy)-2-oxoethyl)-[2,2'-bipyridin]-1,1'-diium bromide (7b)