SciForschen
Open HUB for Scientific Research
ISSN 2470-1009
Journal of Drug Research and Development
Research Article
Volume: 3.4
Open Access

# Synthesis and Characterization of Antibacterial Ionic Liquids Moieties under Multiple Routes and their Catalytic Responses

**Pandurangan Ganapathi, and Kilivelu Ganesan***

*PG & Research Department of Chemistry, Presidency College (Autonomous), Chennai, India*

## Supplementary data

### Experimental section:

### General procedure for *N*-alkylation reaction:

2-Methyl-4(5)-nitro-1H-imidazole ($1.573 \times 10^{-2}$ mmol; 1.0 equi.) mixed with slight excess of benzyl bromide/4-nitro benzyl bromide ($1.652 \times 10^{-2}$ mmol; 1.05 equi.) in presence of 30 mL of the MeCN under refluxing condition for about 9-13 hours afforded the *N*-alkylated products of (**1a-b)** in quantitative yield after the purification.

**General procedure for solvent free Muffle furnace condition:** Required equivalent as mentioned in conventional method except solvent, we have used 5 g of (80-120 mesh) silica gel with fine grinding using mortar and pestle. The reaction mixture is kept it in Muffle furnace at 100°C for required period.

**2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumbromide 1a:** 4.60 g; 97%; Mp: 168-170 °C; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.35 (s, 3H), 5.28 (s, 2H), 7.08-7.17 (m, 5H), 8.42 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 14.1, 50.1, 122.9, 127.6, 128.3, 129.3, 130.9, 136.0, 145.7; MS: *m/z*: 298.13; Elemental analysis: Molecular formula ($C_{11}H_{12}BrN_3O_2$) Calculated: C: 44.30; H: 4.02; N: 14.09; Found C: 44.26; H: 3.96; N: 14.04.

**2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumbromide 1b:** 5.20 g; 93%; Mp: 98-100°C; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.30 (s, 3H), 5.31 (s, 2H), 7.27-7.29 (d, 2H), 7.39-7.41 (d, 2H), 8.46 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 15.4, 51.5, 121.3, 125.6, 128.3, 131.4, 139.4, 142.1,147.4; MS: *m/z*: 343.13; Elemental analysis: Molecular formula ($C_{11}H_{11}BrN_4O_4$) Calculated: C: 38.48; H: 3.26; N: 16.32; Found C: 38.44; H: 3.24; N: 16.28.

### General procedure for anion exchange reaction:

*N*-alkylated product of imidazolium bromide **1a,1b** (1.0 equi.) is treated with $NaBF_4$/KPF6/$LiCF_3SO_3$ (1.05 equi.) in the presence of 10 mL of deionized water at room temperature stirring for about 1 h afforded the anion exchanged product of imidazolium cation with different anion. After the anion exchanged reaction, we have used Soxhlet extraction to remove metal bromide from di/trimeric imidazolium salts using 100 mL of dry THF for about 1 h refluxtion to give respective imidazolium salts in quantitative yield.

**2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumtetrafluoroborate 2a:** 0.57 g; 83%; Mp: 127-130°C; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.38 (s, 3H), 5.26 (s, 2H), 7.12-7.17 (m, 5H), 8.45 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 14.2, 50.3, 122.8, 127.9, 128.4, 129.6, 130.8, 136.1, 145.5; MS: *m/z*: 305.09; Elemental analysis: Molecular formula ($C_{11}H_{12}BF_4N_3O_2$) Calculated: C: 43.26; H: 3.93; N: 13.77; Found C: 43.21; H: 3.88; N: 13.73.

**2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumhexafluorophosphate 2b:** 0.74 g; 91%; Semisolid; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.33 (s, 3H), 5.33 (s, 2H), 7.11-7.19 (m, 5H), 8.47 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 13.1, 49.8, 122.7, 127.5, 128.9, 129.7, 130.7, 136.5, 145.9; MS: *m/z*: 363.19; Elemental analysis: Molecular formula ($C_{11}H_{12}F_6N_3O_2P_2$) Calculated: C: 36.38; H: 3.33; N: 11.57; Found C: 36.32; H: 3.30; N: 11.54.

**2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumtrifluoromethanesulfonate 2c:** 0.70 g; 91%; Semisolid; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.42 (s, 3H), 5.35 (s, 2H), 7.07-7.16 (m, 5H), 8.43 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 14.5, 50.9, 121.7, 126.5, 128.0, 129.5, 130.6, 135.7, 144.3; MS: *m/z*: 367.30; Elemental analysis: Molecular formula ($C_{12}H_{12}F_3N_3O_5S$) Calculated: C: 39.24; H: 3.29; N: 11.44; Found C: 39.19; H: 3.25; N: 11.40.

**2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumtetrafluoroborate 2d:** 0.60 g; 89%; Mp: 110-112°C; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.27 (s, 3H), 5.40 (s, 2H), 7.20-7.23 (d, 2H), 7.34-7.35 (d, 2H), 8.31 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 16.3, 50.9, 122.1, 128.9, 129.8, 130.7, 132.5, 145.8, 146.4; MS: *m/z*: 350.03; Elemental analysis: Molecular formula ($C_{11}H_{11}BF_4N_4O_4$) Calculated: C: 37.74; H: 3.17; N: 16.01; Found C: 37.68; H: 3.13; N: 15.97.

**2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumhexafluorophosphate 2e:** 0.74 g; 94%; Mp: 120-1122°C; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.39 (s, 3H), 5.51 (s, 2H), 7.42-7.51 (d, 2H), 7.35-7.38 (d, 2H), 8.33 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 15.2, 50.9, 121.9, 128.0, 129.7, 130.3, 132.8, 145.9, 146.4; MS: *m/z*: 408.19; Elemental analysis: Molecular formula ($C_{11}H_{11}F_6N_4O_4P$) Calculated: C: 32.37; H: 2.72; N: 13.73; Found C: 32.33; H: 2.68; N: 13.68.

**2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazolium trifluoromethanesulfonate 2f:** 0.69 g; 93%; Semisolid; $^1$H NMR (400 MHz, DMSO-$d_6$): δ; 2.41 (s, 3H), 5.48 (s, 2H), 7.30-7.34 (d, 2H), 7.39-7.42 (d, 2H), 8.43 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$): δ; 16.4, 51.4, 121.0, 128.7, 129.0, 130.3, 132.7, 145.8, 146.2; MS: *m/z*: 412.29; Elemental analysis: Molecular formula ($C_{12}H_{11}F_3N_4O_7S$) Calculated: C: 34.96; H: 2.69; N: 13.59; Found C: 34.92; H: 2.63; N: 13.54.

### General procedure for imidazolium salt assisted Pechmann reaction

Phenol/4-nitrophenol/4-chlorophenol ($2.126 \times 10^{-3}$ mmol; 1.0 eq.), EAA ($2.126 \times 10^{-3}$ mmol; 1.0 eq.), solvent (5 mL) are mixed along with optimized concentration of imidazolium salt ($2.021 \times 10^{-4}$ mmol) at room temperature stirring, after disappearance of starting material monitored by TLC, the reaction mixture was poured into 5 mL of ice cold water and 100 mL of diethyl ether stirred with 5 min. two layer are formed; take the organic layer which was then dried over anhydrous $Na_2SO_4$. Filtrate organic layer under the reduced pressure and evaporated to dryness to obtained pure compound which is confirmed by spectral and analytical data's.

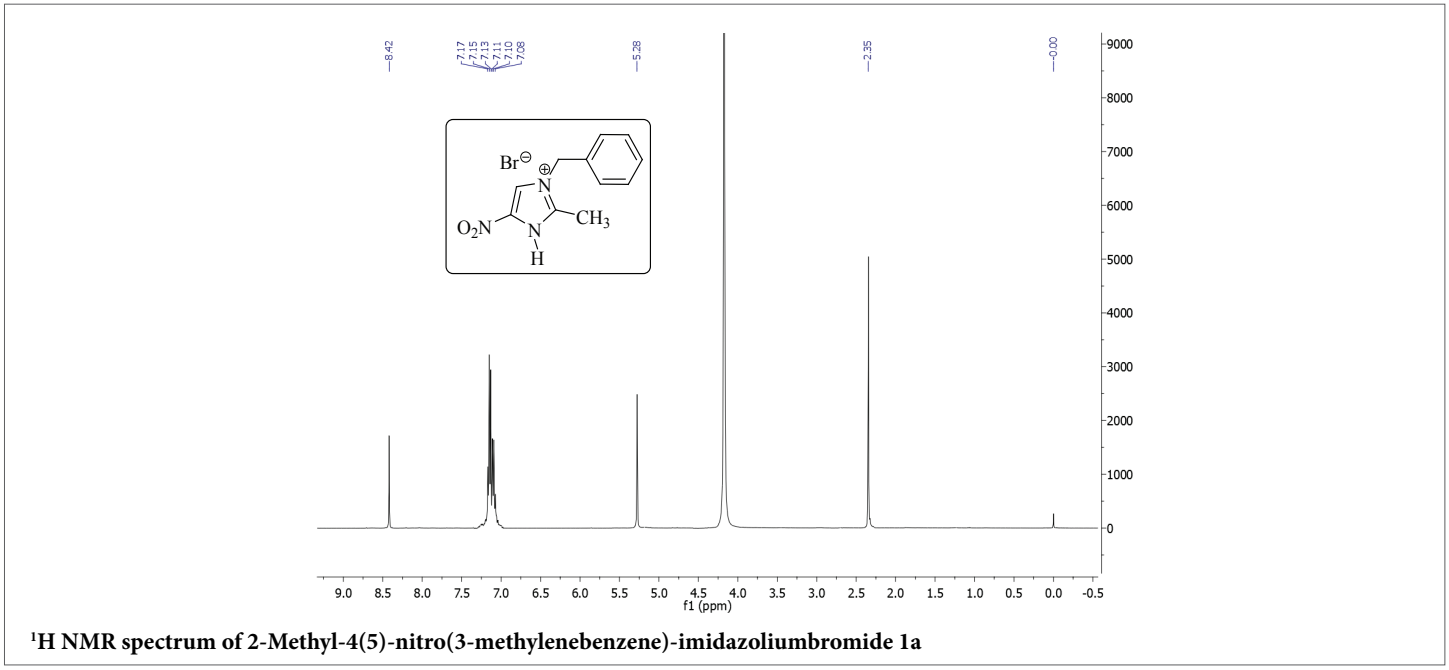
**4-Methyl-2H-chromen-2-one (3):** 0.29 g, 85%, Mp: 81-83°C, $^1$H NMR (400 MHz, $CDCl_3$): δ; 2.42 (s, 3H), 6.32 (s, 1H), 7.15-7.42 (m, 4H); $^{13}$C

NMR (100 MHz, $CDCl_3$): δ; 19.1, 116, 117.9, 122, 124.1, 124.6, 132.6, 154.5, 161.6; MS: *m/z*: 160.05 Elemental analysis: Molecular formula ($C_{10}H_8O_2$) Calculated: C: 74.99; H: 5.03; Found: C: 74.94, H: 4.99.

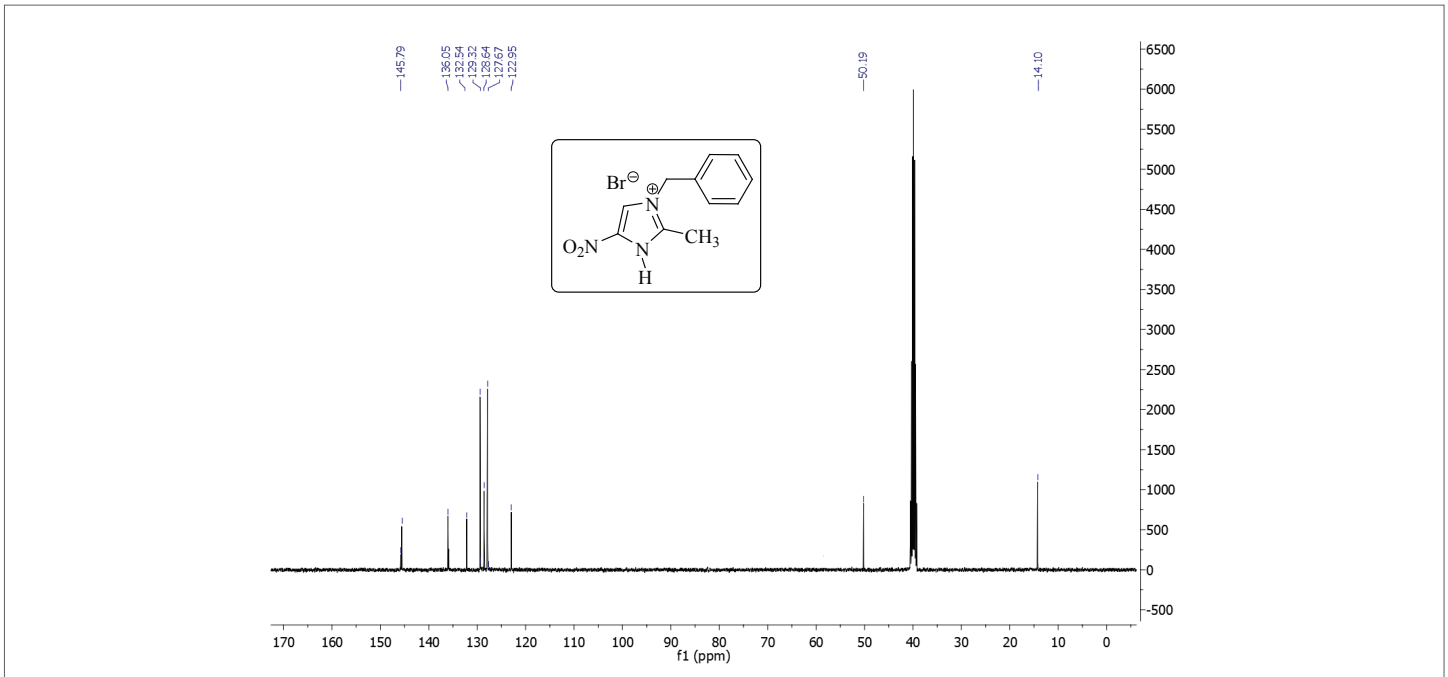
**4-Methyl-6-nitro-2H-chromen-2-one (4):** 0.34 g, 80%, Mp: 153-155°C, $^1$H NMR (400 MHz, $CDCl_3$): δ; 2.43 (s, 3H), 6.14 (s, 1H), 7.30 (d, 1H), 8.10-8.15 (d, 1H), 8.21-8.24 (d, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ; 20.3, 111.4, 120.9, 129.5, 122.0, 122.6, 144.3, 151.7, 155.9, 161.8; MS: *m/z*: 205.03. Elemental analysis: Molecular formula ($C_{10}H_7NO_4$) Calculated: C: 58.84, H: 3.44, N: 6.83, Found: C: 58.80, H: 3.40, N: 6.80.

**6-Chloro-4-methyl-2H-chromen-2-one (5):** 0.35 g, 86%, Mp: 179-182°C, $^1$H NMR (400 MHz, $CDCl_3$): δ; 2.05 (s, 3H), 6.18 (s, 1H), 7.12-7.16 (d, 1H), 7.19-7.22 (d, 1H), 7.29 (s, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$): δ; 21.4, 115.2, 122.4, 123.4, 127.3, 129.8, 132.3, 149.2, 154.5, 160.9; MS: *m/z*: 194.01 Elemental analysis: Molecular formula ($C_{10}H_7ClO_2$) Calculated: C: 61.72, H:3.63, Found: C: 61.67, H: 3.58.

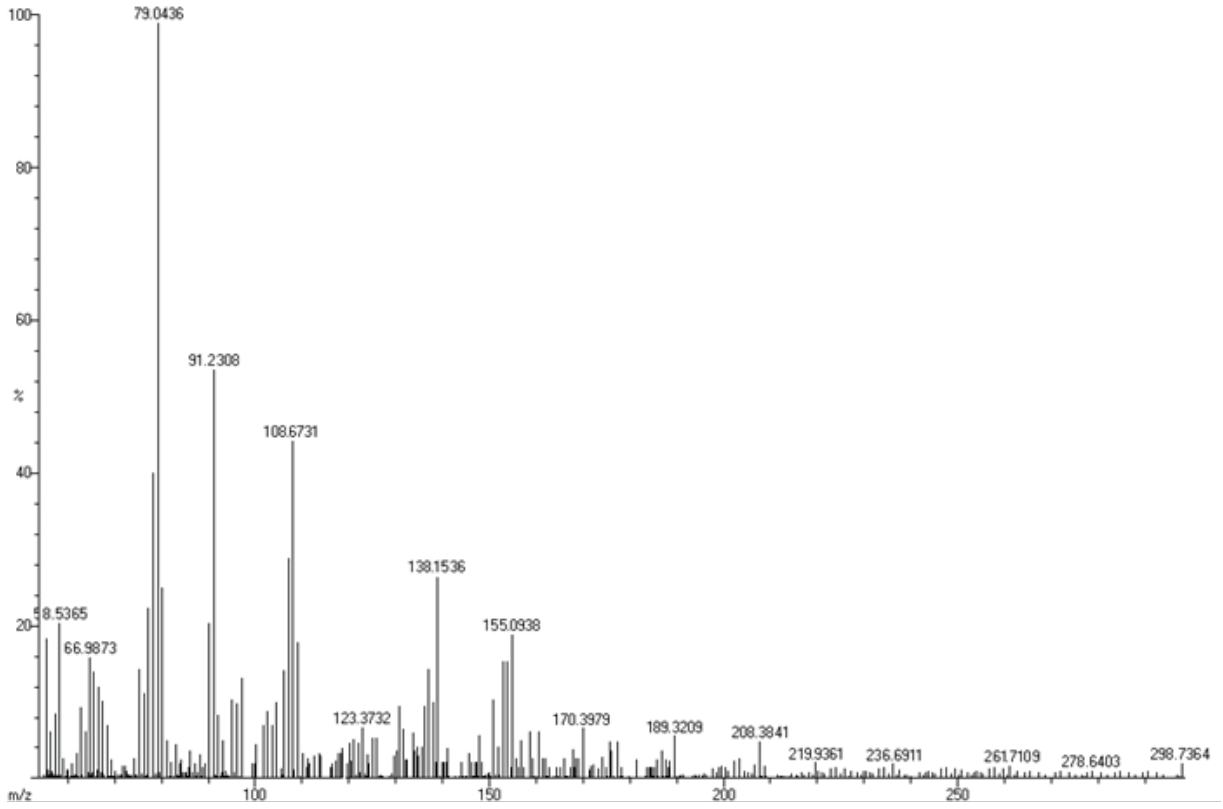
**1-Methyl-3H-benzo[f]chromen-3-one (6):** 0.39 g, 89%, Mp: 182-185°C, $^1$H NMR (400 MHz, $CDCl_3$): δ; 2.45 (s, 3H), 6.32 (s, 1H), 7.32-7.60 (m, 4H), 7.90-7.94 (d, 1H), 8.54-8.57 (d, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ; 24.40, 109.47, 115.70, 116.80, 117.76, 123.60, 126.53, 129.84, 134.58, 153.40, 154.90, 160.30. MS: *m/z*: 210.06 Elemental analysis: Molecular formula ($C_{14}H_{10}O_2$) Calculated: C: 79.98, H: 4.79, Found: C: 79.92, H: 4.74.



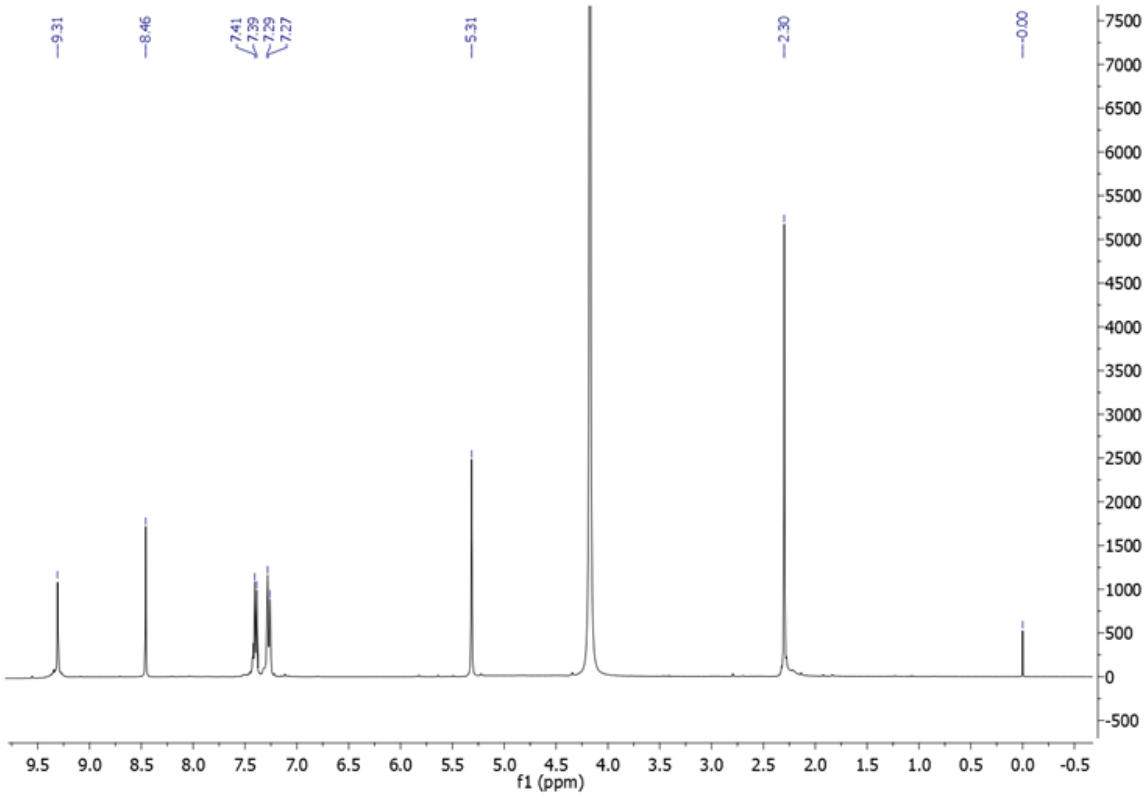

**$^1$H NMR spectrum of 2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumbromide 1a**



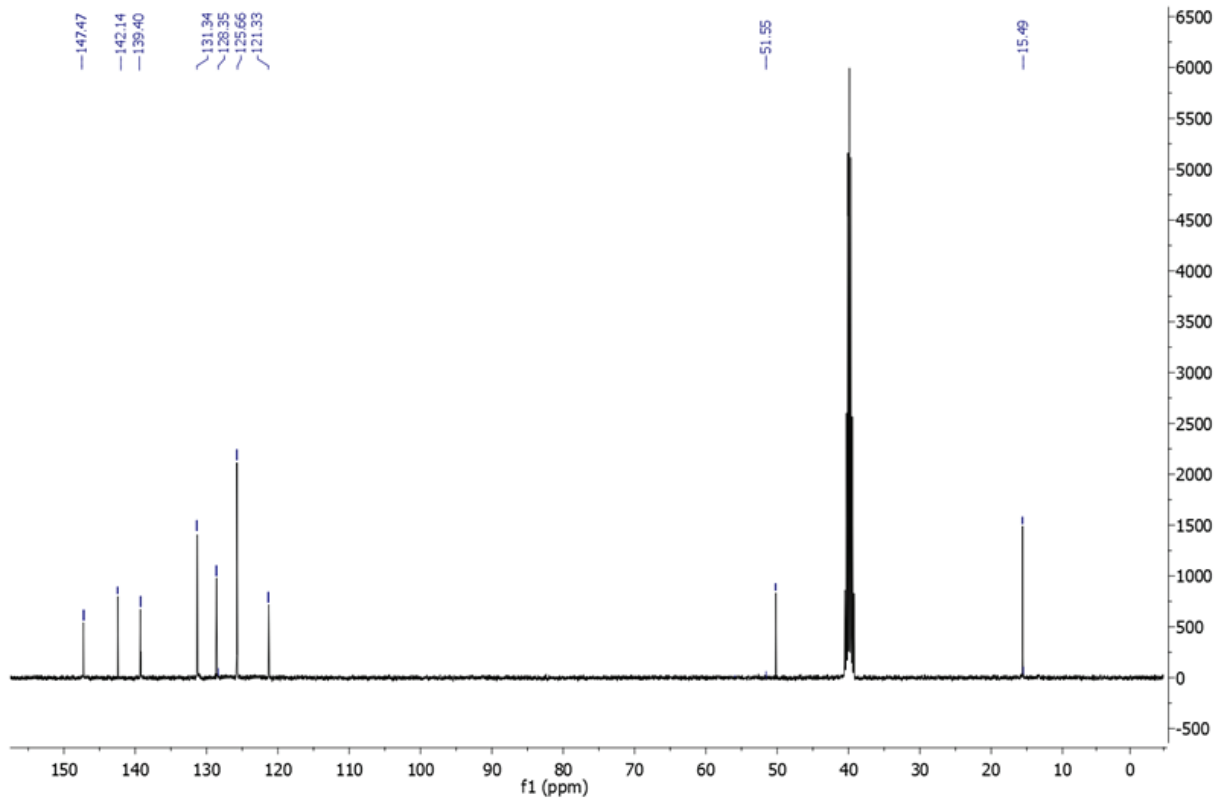

**$^{13}$C NMR spectrum of 2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumbromide 1a**



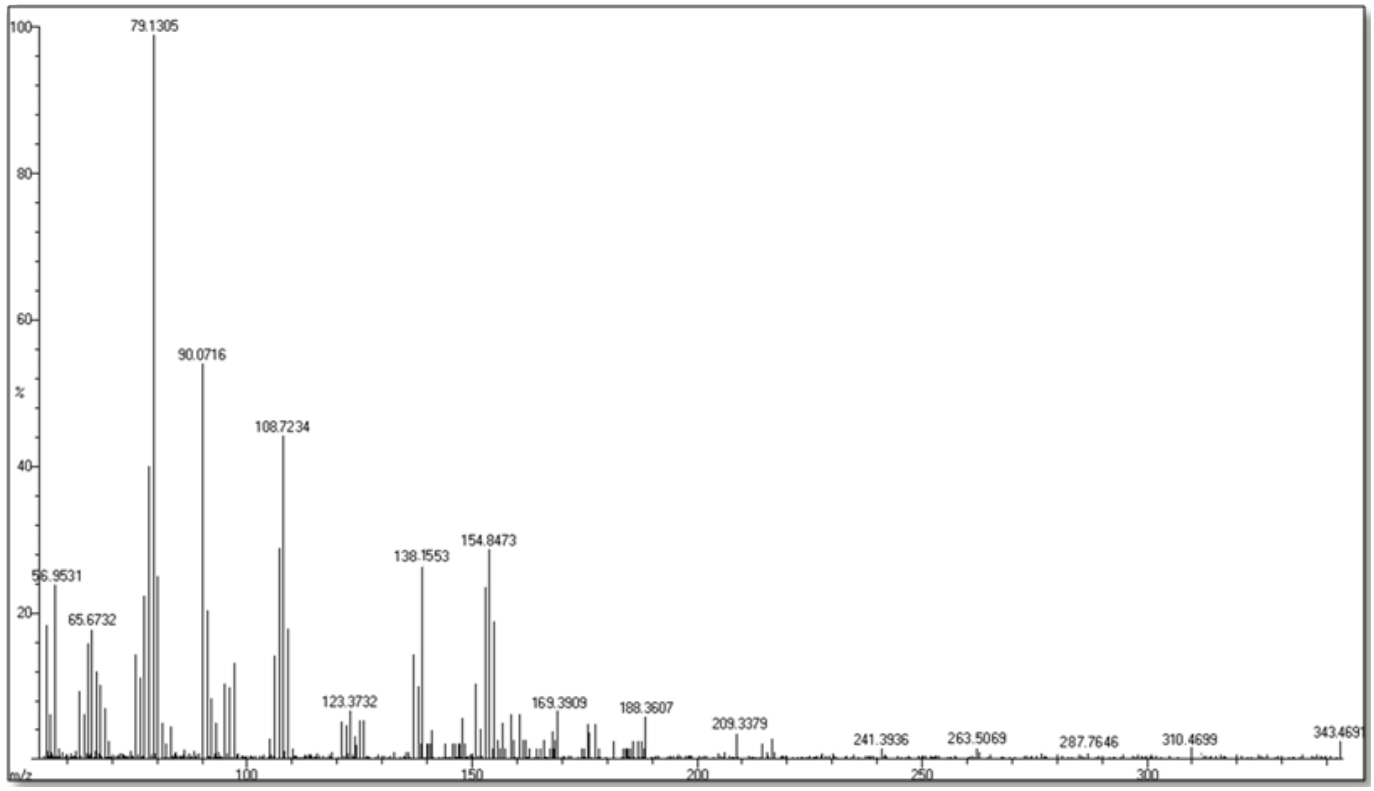

**Mass spectrum of 2-Methyl-5-nitro(3-methylenebenzene)-imidazoliumbromide 1a**




**$^{1}$H NMR spectrum of 2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumbromide 1b**




**$^{13}$C NMR spectrum of 2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumbromide 1b**




**Mass spectrum of 2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumbromide 1b**

**Citation:** Ganapathi P, Ganesan K (2017) Synthesis and Characterization of Antibacterial Ionic Liquids Moieties under Multiple Routes and their Catalytic Responses. J Drug Res Dev 3(4): doi http://dx.doi.org/10.16966/2470-1009.138