Supporting Information

A general and direct synthesis of imidazolium ionic liquids using orthoester

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Table of contents

1. General remark S2
   1.1 Chemical sources and general procedure S2
   1.2 Instrumentation S3

2. Experimental section S3
   2.1 Screen of other alkylating agents S3
   2.2 Synthesis of ionic liquids S3
   2.3 TGA data for [BMIM][BF₄] S11
   2.4 Ion Chromatography S12
   2.5 High resolution mass spectra S16

3. References S17

4. Spectral Data (¹H, ¹³C, ¹⁹F NMR) S18
1. General remark

1.1 Chemical sources and general procedure

Commercially available reagents were used without further purification. Chemical sources of chemicals are shown below.

Chemicals from C-TRI company (http://www.c-tri.co.kr/ctri_eng).

[BMIM][BF₄], Batch. No. ILI04C-131113
Halide content: 10 ppm, water content: 31.9 ppm according to the certificate of analysis

Chemicals from Aldrich
1-Butylimidazole, 98%, Cat. No. 348414
Trimethyl orthoformate, 99%, Cat. No. 108456
Ammonium tetrafluoroborate, 97%, Cat. No. 223727
Tetrafluoroboric acid solution 48 wt. % in H₂O, Cat. No. 207934
Imidazole ACS reagent, ≥99%, Cat. No. 436151
Ammonium hexafluorophosphate, ≥98.0 %, Cat. No. 0-9820
Ammonium iodide, >99%, Cat. No. 0-9874
Ammonium nitrate, 98+ %, Cat. No. 221244
Bis(trifluoromethane)sulfonimide ≥95.0%, Cat. No. 15220
Nitric acid ACS reagent, 70%, Cat. No. 438073
Hexafluorophosphoric acid ~55 wt. % in H₂O, Cat. No. 200956

Chemicals from TCI
1-Phenylimidazole >98.0%(GC), Cat. No. P2030
Trifluoromethanesulfonic acid >98.0%(T), Cat. No. T0751
Isoquinoline >95.0%(GC), Cat. No. I0182
1-Vinylimidazole >98.0%(GC)(T), Cat. No. V0045
Triethyl orthoformate >98%(GC), Cat. No. O0066
Triisopropyl orthoformate >97.0%(GC), Cat. No. O0215
Tributyl orthoformate >95.0%(GC), Cat. No. O0269

Chemical from Wako
Ammonium bromide >98%, Cat. No. 1294

Chemicals from Junsei
Ammonium chloride >98%, Cat. No. 9D1547
p-Toluenesulfonic acid (chemical pure), Cat. No. 811572

Chemicals from Daesung
Ethyl acetate 99%,
Dichloromethane 99.5%
Acetone 99.5%
Acetonitrile 99.5%
Methyl alcohol 99.5%
1-alkyl imidazole (1 eq) and ammonium salt (1.2 eq) were mixed with trialkyl orthoformate (5 eq) under N\textsubscript{2} atmosphere. In some reactions, protic acids (HX, 1 eq) were used to protonate imidazole. All reactions were monitored by \textsuperscript{1}H NMR using DMSO-d6. After the reaction, solvent was removed under reduced pressure. Reaction mixture was dissolved in suitable solvent. The mixture was filtered through basic alumina. And then solvent was removed under reduced pressure to have the desired product.

1.2 Instrumentation

\textsuperscript{1}H NMR and \textsuperscript{13}C NMR spectra were recorded in DMSO-d6 and CDCl\textsubscript{3} (Cambridge isotope) at a Varian Mercury Plus 300MHz spectrometers. \textsuperscript{19}F NMR spectra were recorded in DMSO-d6 at Unity-Inova 500 MHz spectrometers. TG analysis were performed on DSC Q200 (TA Instruments Korea) and STA6000/8000 (Perkin Elmer). Mass spectra (FAB) were obtained using a Jeol JMS700 high-resolution mass spectrometer at the Korea Basic Science Center, Daegu, Korea. Mass spectra (ESI) were obtained using Agilent, Q-TOF 6530 at PNU Center for Research Facilities, Pusan, Korea. Ion chromatographic analysis was performed using Dionex (ICS-5000), equipped with an Dionex IonPacTM As15 column (4 x 250mm). Karl-Fisher test was performed using 831 KFC coulometer.

2. Experimental section

2.1 Screen of other alkylating agents

<table>
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<tr>
<th>Entry</th>
<th>Alkylating agent</th>
<th>Temp. (°C)</th>
<th>Time (h)</th>
<th>Yield\textsuperscript{a} (%)</th>
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<tr>
<td>1</td>
<td>CH\textsubscript{3}(OCH\textsubscript{3})\textsubscript{3}</td>
<td>110</td>
<td>22</td>
<td>97</td>
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<tr>
<td>2</td>
<td>(CH\textsubscript{3})\textsubscript{2}(OCH\textsubscript{3})\textsubscript{2}</td>
<td>80</td>
<td>22</td>
<td>NR</td>
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<tr>
<td>3</td>
<td>(CH\textsubscript{3})\textsubscript{2}N(OCH\textsubscript{3})\textsubscript{3}</td>
<td>100</td>
<td>22</td>
<td>NR</td>
</tr>
</tbody>
</table>

\textsuperscript{a}isolated yield

2.2 Synthesis of ionic liquids

1-butyl-3-methylimidazolium bromide \textsuperscript{[1]}

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium bromide (2.18 mmol, 213 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.74 mmol, 383 mg) was collected in 96% yield.

\textsuperscript{1}H NMR δ 0.83 (t, J=7.5 Hz, 3H) 1.19 (sextet, J=7.5 Hz, 2H) 1.74 (quintet, J=7.5 Hz, 2H) 3.88 (s, 3H) 4.21 (t, J=7.5 Hz, 2H) 7.82 (s, 1H) 7.91 (s, 1H) 9.45 (s, 1H) \textsuperscript{13}C NMR δ 13.70 19.17 31.85 36.25 48.83 122.67 123.94 137.98
1-butyl-3-methylimidazolium iodide \(^{[1]}\)

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium iodide (2.18 mmol, 316 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.77 mmol, 472 mg) was collected in 97% yield.

\(^{1}\)H NMR \( \delta \): 0.89 (t, \( J=7.5 \) Hz, 3H) 1.24 (sextet, \( J=7.5 \) Hz, 2H) 1.76 (quintet, \( J=7.5 \) Hz, 2H) 3.85 (s, 3H) 4.17 (t, \( J=7.5 \) Hz, 2H) 7.72 (s, 1H) 7.80 (s, 1H) 9.16 (s, 1H) \(^{13}\)C NMR \( \delta \): 13.99 19.45 32.03 36.56 49.18 122.95 124.27 137.16

1-butyl-3-methylimidazolium nitrate \(^{[1]}\)

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium nitrate (2.18 mmol, 174 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 48 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.75 mmol, 353 mg) was collected in 96% yield.

\(^{1}\)H NMR \( \delta \): 0.86 (t, \( J=7.5 \) Hz, 3H) 1.21 (sextet, \( J=7.5 \) Hz, 2H) 1.74 (quintet, \( J=7.5 \) Hz, 2H) 3.85 (s, 3H) 4.17 (t, \( J=7.5 \) Hz, 2H) 7.72 (s, 1H) 7.80 (s, 1H) 9.24 (s, 1H) \(^{13}\)C NMR \( \delta \): 13.85 19.43 32.05 36.27 49.15 122.95 124.25 137.37

1-butyl-3-methylimidazolium tetrafluoroborate \(^{[2]}\)

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 17 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 400 mg) was collected in 97% yield.

\(^{1}\)H NMR \( \delta \): 0.89 (t, \( J=7.5 \) Hz, 3H) 1.24 (sextet, \( J=7.5 \) Hz, 2H) 1.76 (quintet, \( J=7.5 \) Hz, 2H) 3.85 (s, 3H) 4.16 (d, \( J=7.5 \) Hz, 2H) 7.65 (s, 1H) 7.72 (s, 1H) 9.02 (s, 1H) \(^{13}\)C NMR \( \delta \): 13.83 19.40 31.99 36.28 49.20 122.86 124.19 137.10

1-butyl-3-methylimidazolium hexafluorophosphate \(^{[2]}\)

1-butylimidazole (1.82 mmol, 0.24 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 17 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.61 mmol, 460 mg) was collected in 88% yield.

\(^{1}\)H NMR \( \delta \): 0.90 (t, \( J=7.5 \) Hz, 3H) 1.28 (sextet, \( J=7.5 \) Hz, 2H) 1.77 (quintet, \( J=7.5 \) Hz, 2H) 3.84 (s, 3H) 4.15 (d, \( J=7.5 \) Hz, 2H) 7.65 (s, 1H) 7.71 (s, 1H) 9.05 (s, 1H) \(^{13}\)C NMR \( \delta \): 13.80 19.40 31.97 36.28 49.20 122.83 124.19 137.12
1-butyl-3-methylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 °C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent removed under vacuum and then the resulting product (1.74 mmol, 394 mg) was collected in 96% yield.

1H NMR δ 0.89 (t, J=7.5 Hz, 3H) 1.24 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz, 2H) 3.85 (s, 3H) 4.16 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.72 (s, 1H) 9.02 (s, 1H) 13C NMR δ 13.83 19.40 31.99 36.29 49.18 122.86 124.19 137.10

1-butyl-3-methylimidazolium hexafluorophosphate

Aqueous hexafluorophosphoric acid (1.82 mmol, 0.247 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 °C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent removed under reduced pressure and then the resulting product (1.73 mmol, 494 mg) was collected in 95% yield.

1H NMR δ 0.90 (t, J=7.5 Hz, 3H) 1.28 (sextet, J=7.5 Hz, 2H) 1.77 (quintet, J=7.5 Hz, 2H) 3.84 (s, 3H) 4.15 (d, J=7.5 Hz, 2H) 7.65 (s, 1H) 7.71 (s, 1H) 9.05 (s, 1H) 13C NMR δ 13.80 19.40 31.97 36.28 49.20 122.83 124.19 137.12

1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide

1-butylimidazole (1.69 mmol, 0.22 mL) and bis(trifluoromethanesulfonyl)imide (1.69 mmol, 475 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.66 mmol, 645 mg) was collected in 99% yield.

1H NMR δ 0.90 (t, J=7.5 Hz, 3H) 1.26 (sextet, J=7.5 Hz, 2H) 1.78 (quintet, J=7.5 Hz, 2H) 3.85 (s, 3H) 4.16 (t, J=7.5 Hz, 2H) 7.66 (s, 1H) 7.72 (s, 1H) 9.09 (s, 1H) 13C NMR δ 13.37 19.12 31.75 35.98 48.96 113.51 117.77 122.03 122.58 123.92 126.28 136.90

1-butyl-3-methylimidazolium trifluoromethanesulfonate

1-butylimidazole (1.82 mmol, 0.24 mL) and trifluoromethanesulfonic acid (1.82 mmol, 0.16 mL) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.72 mmol, 496 mg) was collected in 95% yield.

1H NMR δ 0.89 (t, J=7.5 Hz, 3H) 1.27 (sextet, J=7.5 Hz, 2H) 1.76 (quintet, J=7.5 Hz, 2H) 3.85 (s, 3H) 4.16 (t, J=7.5 Hz, 2H) 7.68 (s, 1H) 7.75 (s, 1H) 9.08 (s, 1H) 13C NMR δ 13.56 19.15 31.75 36.07 48.93 114.66 118.92 122.62 123.19 123.97 127.45 136.90
1-butyl-3-methylimidazolium 4-methylbenzenesulfonate \(^{[4]}\)

1-butylimidazole (1.82 mmol, 0.24 mL) and \(p\)-toluenesulfonic acid (1.82 mmol, 346 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in dichloromethane. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.71 mmol, 532 mg) was collected in 94% yield.

\(^1\)H NMR 0.8 (t, \(J=7.5\) Hz, 3H) 1.22 (sextet, \(J=7.5\) Hz, 2H) 1.74 (quintet, \(J=7.5\) Hz, 2H) 3.84 (s, 3H) 4.15 (t, \(J=7.5\) Hz, 2H) 7.11 (d, \(J=7.8\) Hz, 2H) 7.48 (d, \(J=7.8\) Hz, 2H) 7.77 (s, 1H) 9.15 (s, 1H)

\(^{13}\)C NMR 13.73 19.21 21.23 31.82 36.15 48.90 122.74 124.05 125.92 128.52 137.01 138.09 146.18

1,3-dimethylimidazolium hexafluorophosphate \(^{[5]}\)

1-methylimidazole (1.82 mmol, 0.145 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.79 mmol, 433 mg) was collected in 98% yield.

\(^1\)H NMR 3.84 (s, 6H) 7.66 (s, 2H) 9.00 (s, 1H)

\(^{13}\)C NMR 36.28 124.08 137.6

1,3-dimethylimidazolium tetrafluoroborate \(^{[6]}\)

1-methylimidazole (1.82 mmol, 0.145 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.71 mmol, 314 mg) was collected in 94% yield.

\(^1\)H NMR 3.84 (s, 6H) 7.66 (s, 2H) 9.00 (s, 1H)

\(^{13}\)C NMR 36.09 123.89 137.46

1-allyl-3-methylimidazolium hexafluorophosphate

1-allylimidazole (1.82 mmol, 0.195 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.81 mmol, 487 mg) was collected in 97% yield. HRMS (FAB+) calcd for C\(_7\)H\(_{11}\)N\(_2\):123.0922; found, 123.0921

\(^1\)H NMR 3.86 (s, 3H) 4.83 (d, \(J=6\) Hz, 2H) 5.28 (d, \(J=17\) Hz, 1H) 5.39 (d, \(J=11.5\) Hz, 1H) 6.01 (m, 1H) 7.66 (s, 2H) 9.06 (s, 1H)

\(^{13}\)C NMR 51.25 12.68 122.70 124.16 131.96 137.04

1-allyl-3-methylimidazolium tetrafluoroborate \(^{[7]}\)

1-allylimidazole (1.82 mmol, 0.195 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl
acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.79 mmol, 376 mg) was collected in 96% yield.

\[ ^1H \text{NMR } \delta 3.85 \text{ (s, 3H)} \ 4.83 \text{ (d, J=6 Hz, 2H)} \ 5.28 \text{ (d, J=17 Hz, 1H)} \ 5.39 \text{ (d, J=11.5 Hz, 1H)} \ 6.02 \text{ (m, 1H)} \ 7.70 \text{ (s, 2H)} \ 9.08 \text{ (s, 1H)} \]

\[ ^13C \text{NMR } \delta 36.16 51.20 120.63 122.73 124.17 132.11 136.99 \]

1-benzyl-3-methylimidazolium hexafluorophosphate

1-benzylimidazole (1.82 mmol, 288 mg) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 19 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.80 mmol, 576 mg) was collected in 99% yield.

\[ ^1H \text{NMR } \delta 3.86 \text{ (s, 3H)} \ 5.42 \text{ (s, 2H)} \ 7.42 \text{ (s, 5H)} \ 7.71 \text{ (s, 1H)} \ 7.78 \text{ (s, 1H)} \ 7.78 \text{ (s, 1H)} \ 9.20 \text{ (s, 1H)} \]

\[ ^13C \text{NMR } \delta 36.30 52.36 122.80 124.45 128.70 129.19 129.45 135.25 137.13 \]

3-methyl-1-phenylimidazolium hexafluorophosphate

1-phenylimidazole (1.03 mmol, 0.13 mL) and ammonium hexafluorophosphoate (1.24 mmol, 201 mg) was mixed with trimethyl orthoformate (5.15 mmol, 0.56 mL). The reaction mixture was heated to 110 °C for 20 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and acetone. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.00 mmol, 318 mg) was collected in 97% yield. HRMS (FAB+) calcd for C\textsubscript{10}H\textsubscript{11}N\textsubscript{2}: 159.0922; found, 159.0923

\[ ^1H \text{NMR } \delta 3.94 \text{ (s, 1H)} \ 7.59 \text{ (d, J=7.2 Hz, 1H)} \ 7.69 \text{ (t, J=7.2 Hz, 2H)} \ 7.77 \text{ (d, J=7.2 Hz, 2H)} \ 7.93 \text{ (s, 1H)} \ 8.28 \text{ (s, 1H)} \ 9.73 \text{ (s, 1H)} \]

\[ ^13C \text{NMR } \delta 36.56 121.47 122.30 124.88 130.23 130.67 135.21 136.38 \]
3-methyl-1-vinylimidazolium hexafluorophosphate

1-vinylimidazole (1.82 mmol, 0.165 mL) and ammonium hexafluorophosphate (2.18 mmol, 356 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in acetonitrile. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 452 mg) was collected in 97% yield. HRMS (FAB+) calcd for C₆H₉N₂: 109.0766; found, 109.0765

¹H NMR δ3.88 (s, 3H) 5.39 (d, J=8.8 Hz, 1H) 5.90 (d, J=15.7 Hz, 1H) 7.26 (dd, J₁=15.7 Hz, J₂=8.8 Hz) 7.82 (s, 1H) 8.15 (s, 1H) 9.38 (s, 1H)

¹³C NMR δ36.42 108.86 119.27 124.83 129.28 136.50

3-methyl-1-vinylimidazolium tetrafluoroborate

1-vinylimidazole (1.82 mmol, 0.165 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 22 h. After the reaction, solvent was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol. The mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.76 mmol, 346 mg) was collected in 96% yield. HRMS (FAB+) calcd for C₆H₉N₂: 109.0766; found, 109.0765

¹H NMR δ3.85 (s, 3H) 5.39 (d, J=8.8 Hz, 1H) 5.90 (d, J=15.7 Hz, 1H) 7.26 (dd, J₁=15.7 Hz, J₂=8.8 Hz) 7.78 (s, 1H) 8.12 (s, 1H) 9.35 (s, 1H)

¹³C NMR δ36.42 108.86 119.27 124.83 129.28 136.50

1-butyl-3-ethylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then triethyl orthoformate (9.1 mmol, 1.5 mL) was added to residue. The reaction mixture was heated to 120 °C for 25 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.66 mmol, 399 mg) was collected in 91% yield. HRMS (FAB+) calcd for C₉H₁₇N₂: 153.1392; found, 153.1391

¹H NMR δ0.89 (t, J=7.5 Hz, 3H) 1.25 (sextet, J=7.5 Hz, 2H) 1.41 (t, J=7.5 Hz, 3H) 1.78 (quintet, J=7.5 Hz, 2H) 4.17 (m, 4H) 7.77 (s, 2H) 9,12 (s, 1H)

¹³C NMR δ13.62 15.34 19.22 31.72 44.66 49.02 122.51 122.79 136.02

1-butyl-3-propylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then tripropyl orthoformate (9.1 mmol, 1.9 mL) was added to residue. The reaction mixture was heated to 130 °C for 28 h. After the reaction, tripropyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under vacuum and then the resulting product (1.68 mmol, 429 mg) was collected in 93% yield. HRMS (FAB+) calcd for C₁₀H₁₉N₂: 167.1548; found, 167.1546

¹H NMR δ0.87 (m, 6H) 1.25 (sextet, J=7.5 Hz, 2H) 1.82 (m, 4H) 4.14 (m, 4H) 7.79 (s, 2H) 9.17 (s, 1H)

¹³C NMR δ10.90 13.80 19.42 23.43 31.93 49.27 51.02 123.05 136.53

S8
1-butyl-3-isopropylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then triisopropyl orthoformate (9.1 mmol, 1.9 mL) was added to the residue. The reaction mixture was heated to 130 °C for 24 h. After the reaction, triisopropyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.44 mmol, 366 mg) was collected in 79% yield. HRMS (FAB+) calcd for C_{10}H_{19}N_{2}: 167.1548; found, 167.1551

\[ ^1H \text{ NMR } \delta 0.90 (t, J=7.5 Hz, 3H) 1.26 (sextet, J=7.5 Hz, 2H) 1.48 (d, J=6.0 Hz, 6H) 1.80 (quintet, J=7.5 Hz, 2H) 4.16 (t, J=7.5 Hz, 2H) 4.62 (septet, J=6.0 Hz, 1H) 7.77 (s, 1H) 7.87 (s, 1H) 9.16 (s, 1H) \]

\[ ^{13}C \text{ NMR } \delta 13.62 19.26 31.71 49.06 52.71 120.97 122.88 135.06 \]

1,3-dibutylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to 1-butylimidazole (1.82 mmol, 0.24 mL) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then triisopropyl orthoformate (9.1 mmol, 2.4 mL) was added to the residue. The reaction mixture was heated to 140 °C for 24 h. After the reaction, triisopropyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.70 mmol, 457 mg) was collected in 94% yield.

\[ ^1H \text{ NMR } \delta 0.90 (t, J=7.5 Hz, 6H) 1.24 (sextet, J=7.5 Hz, 4H) 1.77 (t, J=7.5 Hz, 4H) 4.16 (t, J=7.5 Hz, 4H) 7.79 (s, 2H) 9.18 (s, 1H) \]

\[ ^{13}C \text{ NMR } \delta 13.62 19.19 31.71 49.02 122.85 136.30 \]

N-methylisoquinolinium tetrafluoroborate

Isoquinoline (1.82 mL, 0.21 mL) and ammonium tetrafluoroborate (2.18 mmol, 229 mg) was mixed with trimethyl orthoformate (9.1 mmol, 1 mL). The reaction mixture was heated to 110 °C for 24 h. After the reaction, solvent was removed under reduced pressure. Ethyl acetate was added to the residue for recrystallization. The resulting product (1.61 mmol, 372 mg) was obtained as orange solid in 89% yield. HRMS (FAB+) calcd for C_{10}H_{16}N: 144.0813; found, 144.0811

\[ ^1H \text{ NMR } \delta 4.47 (s, 3H) 8.06 (t, J=7.8 Hz, 1H) 8.24 (t, J=7.5 Hz, 1H) 8.33 (d, J=8.1 Hz, 1H) 8.46 (d, J=8.1 Hz, 1H) 8.54 (d, J=6.6 Hz, 1H) 8.69 (d, J=6.6 Hz, 1H) 9.97 (s, 1H) \]

\[ ^{13}C \text{ NMR } \delta 48.33 125.84 127.48 127.66 130.59 131.60 136.30 137.05 137.14 151.13 \]

1,3-dimethylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) in at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then trimethyl orthoformate (9.1 mmol, 1 mL) was added to the residue. The reaction mixture was heated to 110 °C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.54 mmol, 284 mg) was collected in 84% yield.

\[ ^1H \text{ NMR } \delta 3.84 (s, 6H) 7.64 (s, 2H) 8.99 (s, 1H) \]

\[ ^{13}C \text{ NMR } \delta 36.28 124.08 137.64 \]
1,3-diethylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then triethyl orthoformate (9.1 mmol, 1.5 mL) was added to the residue. The reaction mixture was heated to 130 °C for 20 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in ethyl acetate and methanol and the mixture was filtered through basic alumina. Solvent was removed under reduced pressure and then the resulting product (1.54 mmol, 284 mg) was collected in 68% yield. HRMS (FAB+) calcd for C_{7}H_{13}N_{2}: 125.1079; found, 125.1079

\(^1\)H NMR 1.42 (t, J= 7.5 Hz, 6H), 4.17 (q, J=7.5 Hz, 4H) 7.80 (s, 2H) 9.16 (s, 1H) \(^{13}\)C NMR δ15.45 44.62 122.54 135.80

1,3-diisopropylimidazolium tetrafluoroborate

Aqueous tetrafluoroboric acid (1.82 mmol, 0.237 mL) was added to imidazole (1.82 mmol, 124 mg) at 0 °C and water was removed under reduced pressure using phosphorus pentoxide. And then triisopropyl orthoformate (9.1 mmol, 1.5 mL) was added to the residue. The reaction mixture was heated to 130 °C for 48 h. After the reaction, triisopropyl orthoformate was removed under reduced pressure. Recrystallization was performed in the presence of ethyl acetate and the resulting product (0.57 mmol, 137 mg) was collected in 31% yield. HRMS (FAB+) calcd for C_{9}H_{17}N_{2}: 153.1392; found, 153.1393

\(^1\)H NMR 1.47 (d, J= 6.9 Hz, 12H), 4.60 (septet, J=6.9 Hz, 2H) 7.91 (s, 2H) 9.23 (s, 1H) \(^{13}\)C NMR δ22.73 52.69 121.08 133.94

1,3-diethylimidazolium 4-methylbenzenesulfonate

1-butyrimidazole (1.82 mmol, 124 mg) and p-toluenesulfonic acid (1.82 mmol, 346 mg) was mixed with triethyl orthoformate (9.1 mmol, 1.5 mL). The reaction mixture was heated to 130 °C for 24 h. After the reaction, triethyl orthoformate was removed under reduced pressure. Residue was dissolved in dichloromethane and the mixture was filtered through basic alumina. Solvent removed under reduced pressure and then the resulting product (1.61 mmol, 479 mg) was collected in 89% yield. HRMS (FAB+) calcd for C_{7}H_{13}N_{2}: 125.1079; found, 125.1076

\(^1\)H NMR 1.38 (t, J= 7.5 Hz, 6H), 2.28 (s, 3H) 4.16 (q, J=7.5 Hz, 4H) 7.13 (d, J=6.9 Hz, 2H) 7.53 (d, J=6.9 Hz, 2H) 7.82 (s, 2H) 9.29 (s, 1H) \(^{13}\)C NMR δ15.50 21.20 44.55 122.53 125.89 128.61 135.98 138.29 145.98
2.3 TGA data for [BMIM][BF₄]

Synthesized [BMIM][BF₄] from HBF₄

Commercial [BMIM][BF₄] (c-tri)
2.4 Ion chromatography

* Reagents and Standards: Deionized water, 18 MΩ-cm, Chloride Standard, 100 mg/L (seven anion standard II, Dionex)
* Instrument: Dionex ICS-5000 system
* Column: Analytical column AS15(Dionex, USA) (4x250mm), Guard column AS15 (Dionex, USA) (4x50mm)
* Flow : 1.5mL/min
* Eluent 32mM KOH
* Temperature: 35 °C
* Sampling: [BMIM]Br (from NH₄Br): 0.1408 g  [BMIM]I (from NH₄I): 0.1375 g
  [BMIM]BF₄ (from HBF₄): 0.9285 g  [BMIM]BF₄ (from NH₄BF₄): 0.9312 g
  [BMIM]NO₃ (from NH₄NO₃): 0.7092 g  [BMIM]N(Tf)₂ (from HN(Tf)₂): 0.2871 g
  [BMIM]OTs (from TsOH): 0.1204 g  [BMIM]OTf (from TfOH): 0.4962 g

(1) Calibration results for chloride and chromatogram of standard solution

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Calibration Batch Report

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| Instrument Method: organic acid_20130613 | Operator: user |
| Inj. Date / Time: 19-5-2012 / 22:14 | Run Time: 32.00067 |

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AVERAGE: 0.1136 0.1285 0.08%
(2) Chromatogram

[BMIM][Br] from NH₄Br

[BMIM][I] from NH₄I

[BMIM][BF₄] from HBF₄

[BMIM][BF₄] from NH₄BF₄
Commercial [BMIM][BF₄]

[BMIM][NO₃] from HNO₃

[BMIM][NO₃] from NH₄NO₃

[BMIM][N(Tf)₂] from HN(Tf)₂

[BMIM][OTf] from TfOH
2.5 High resolution mass spectra

ESI-negative

Our [BMIM][BF₄] from HBF₄

Commercial [BMIM][BF₄]

[Na][BF₄]₂

[BMIM][BF₄] from TsOH

(3) Cl content table

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3. References

4. Spectral data

Sample Name: 
Archive directory: 
Sample directory: 
FidFile: BNMHEr_final_H
Pulse Sequence: stdih (x2pul)
Solvent: DMSO
Data collected on: Oct 29 2013
Operator: laguta20
VNMRS-300 "Agilent-NMR"

Relax. Delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
20 repetitions
OBSERVE EI, 300.1143883 MHz
DATA PROCESSING
FT size 32768
Total time 6 min 24 sec

! Image of a spectrum showing peaks at various ppm values. The spectrum includes labeled peaks at 1.22, 1.34, 2.24, 3.23, 4.28, 4.56, and 7.93 ppm.
Sample Name: 
Archive directory: 
Sample directory: 
File: BM1Mr_final_C

Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

Operator: laguta20

VMRS-300 "Agilent-NMR"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
56 repetitions

OBSERVE Cl3, 75.4638242 MHz
DECOUPLE H1, 300.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.6 Hz
FT size 131072
Total time 48 min

Chemical shifts:
316.53 ppm
132.67 ppm
122.3 ppm
98.8 ppm
88.03 ppm
67.3 ppm
40.2 ppm
39.02 ppm
38.9 ppm
32.7 ppm
13.7 ppm
12.97 ppm
1-butyl-3-methylimidazolium iodide

**Sample Name:**

**Archive directory:**

**Sample directory:**

**FidFile:** FP_butyI_methyl_i

**Pulse Sequence:** std1h (z2pol)

**Solvent:** DMSO

**Data collected on:** Jan 7 2014

**Operator:** laputa20

**NMRS-300  “Agilent-NMR”**

**Relax. delay 1.000 sec**

**Pulse 45.0 degrees**

**Acq. time 1.999 sec**

**Width 4500.0 Hz**

**4 repetitions**

**OBSERVE R1, 300.1143 MHz**

**DATA PROCESSING**

**FT size 25768**

**Total time 8 min 24 sec**

**ppm**
FP_buty1_methyl_1_C
Pulse Sequence: t1pul
Solvent: DMSO
Ambient temperature
Operator: lapolit
Mercury-300 M - "PHROHEX"

Relax. delay 1.000 sec
Pulse 103.5 degrees
Acq. time 1.011 sec
Width 10761.7 Hz
1024 repetitions
Chem. shift 7.546020 MHz
DECOUPL. H1, 29.6153538 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 6 min, 46 sec
butyl_methyl_N03

Sample Name:
Archive directory:
Sample directory:
FidFile: HMMNNO3_final_E
Pulse Sequence: std1h (x2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

Operator: laputa29
VMRS-300 "Agilent-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
24 repetitions
OBSERVE 81, 300.1143872 MHz
DATA PROCESSING
FT size 32768
Total time 6 min 24 sec
butyl_methyl_103

Sample Name:
Archive directory:
Sample directory:
FidFile: NHNol_final_C
Pulse Sequence: stdl3c (spul)
Solvent: DMSO
Data collected on: Oct 29 2013

Operator: laputa20
VNMRS-300 "Agilent-300"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
24 repetitions

OBSSVE C13, 75.4638262 MHz
DECOUPLE H1, 300.1155330 MHz
Power 44 dB
on during acquisition
c1f off during delay

WAIS-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min

220 200 180 160 140 120 100 80 60 40 20 0 ppm
1-butyl-3-methylimidazolium tetrafluoroborate
Sample Name: NMIM_BF4

Archive directory:  
Sample directory:

FidFile: NMIM_BF4_final.C

Pulse Sequence: std3c (zpulu)  
Solvent: DMSO  
Data collected on: Oct 29 2013

Operator: laputa30  
VNMRS-300 "Agilent-DMS"  

Relax. delay 1.000 sec  
Pulse 103.8 degrees  
Acq. time 1.815 sec  
Width 18761.7 Hz  
44 repetitions

OBSERVED C13, 75.4638642 MHz  
DECOUPLE 81, 300.1155360 MHz  
Power 44 dB  
on during acquisition  
off during delay

WALTZ-16 modulated

DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 46 min

 ppm

220 200 180 160 140 120 100 80 60 40 20 0

135.705
122.798
122.287
48.963
40.311
10.529
19.845
19.875
19.479
18.356
36.036
31.754
19.169
13.594

BF4
1-butyl-3-methylimidazolium hexafluorophosphate
Sample Name:
Archive directory:
Sample directory:
File: DNN754_final_C
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 29 2013

Operator: laputa20
VMRS-300 "Agilent-300"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
60 repetitions

OBSERVE Cl3, 75.4638242 MHz
DECOUPLE H1, 300.1155338 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 46 min
butyl-methyl_NTF2

Sample Name: Butylmethyl
Data Collected on: Agilent-NMR-veesra300
Archive directory: /home/vnmri/veesra3/ds/data/fidlib
Sample directory: Butylmethyl
FidFile: PROTON

Pulse Sequence: PROTON (n2pol)
Soluvent: dmso
Data collected on: Feb 2 2014

Operator: laguta20

Delay, delay 1.000 sec
Pulse 45.0 degrees
Avg. time 1.704 sec
Width 697.7 Hz
Single scan
CONCEN: mL, 300.114665 MHz
DATA PROCESSING
PFT also 16398
Total time 9 min 3 sec

\[ \text{butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)amide} \]
butyl_methyl_NTF2

Sample Name: Ethylindanone
Data Collected on: Agilent=nmr-vmxs300
Archive directory: /home/venul/vensys/data/fidlib
Sample directory: Ethylindanone
FidFile: CARBON
Pulse Sequence: CARBON (e2pool)
Solvent: dmeo
Data collected on: Feb 2 2014

Temp. 17.1 C / 290.2 K
Operator: lupta20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 15933.6 Hz
448 repetitions

ODERIVE CL3, 75.4639242 MHz
DECODER H1, 300.1158938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min

220 200 180 160 140 120 100 80 60 40 20 0 ppm
butyl butyl-OTf

Sample Name: Ethylindazole
Data Collected on: Agilent-NMR-vnmrs300
Archive directory: /home/vvmr1/vnmrsy/d/data/fidlib
Sample directory: Ethylindazole
FidFile: PROTON

Pulse Sequence: PROTON (n2pul)
Solvent: d2o
Data collected on: Feb 2 2014

Operator: legota20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 6807.7 Hz
Single scan
DETECTOR W1 300.1143849 MHz
DATA PROCESSING
FT axis 16384
Total time 5 min 3 sec

1-butyl-3-methylimidazolium trifluoromethanesulfonate
butyl_methyl_PTSN

Sample Name: Ethyldiamine
Data Collected on: Agilent-NMR-zenmrx300
Archive directory: /home/vmsel/vmsrezs/data/zedlib
Sample directory: Ethyldiamine
FidFile: PROTON

Pulse Sequence: PROTON (e2psp)
Solvent: dmso
Data collected on: Feb 1 2014

Temp. 21.0 C / 294.1 K
Operator: laguta20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4807.7 Hz
Single scan

OBSERVE HL 350.1143878 MHz
DATA PROCESSING
FT size 16384
Total time 0 min 3 sec

ppm

9 8 7 6 5 4 3 2 1

1.00 1.04 2.01 2.03 2.13 2.78 2.19

0.981.99 2.90 2.13 3.15

1-butyl-3-methylimidazolium 4-methylbenzenesulfonate
butyl_methyl_PTSa

Sample Name: Ethylindanone
Data Collected on: Agilent-NNH-vnmrs300
Archive directory: /home/vnnr1/vnmrsy/data/tid1lib
Sample directory: Ethylindanone
FidFile: butyl_methyl_PTSa_C

Pulse Sequence: CARBON (s2pul)
Solvent: dmso
Data collected on: Feb 1 2014

Temp. 21.0 C / 294.1 K
Operator: laputa20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4639242 MHz
DECOUPLE H1, 300.1156938 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min

Agilent Technologies
methyl_methyl_PF6

Sample Name:
Archive directory:
Sample directory:
FidFile: 13-dimethylimidazoliumpf6

Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Oct 24 2013

Operator: laputa20
VNMRS-300 "Agilent-NMR"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
2774 repetitions
OBSERVED C13, 75.4638242 MHz
DECOUPLING B1, 300.115338 MHz
Power 44 dB
on during acquisition
do during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min

ppm
Sample Name: methyl_methyl_BF4

Data Collected on:
Agilent-NMR-vnmrs300

Sample directory:
File title: methyl_methyl_BF4

Pulse Sequence: PROTON (zspul)
Solvent: dmso
Data collected on: Jun 29 2014

Temp. 22.0 C / 295.1 K
Operator: laputa20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4807.7 Hz
32 repetitions

OBSEIVE: 81.300.1143866 MHz
DATA PROCESSING:
FT size 16384
Total time 1 min 27 sec

1.3-dimethylimidazolium tetrafluoroborate
Sample Name: FP_methyl_methyl_bf4_C

Archive directory:

Sample directory:

Pulse sequence: std3hc (4pul1)

Solvent: DMSO

Data collected on: Jan 6 2014

Operator: laputa20

VNMRS-300 "Agilent-SMS"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions
OBSERVE C3, 75.4638262 MHz
DECORREL H1, 300.1153538 MHz
Power 44 dB
C3 on during acquisition
C3 off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min

Agilent Technologies

BF₄⁻
Sample Name:

Archive directory:

Sample directory:

FidFile: 1-allyl-3-methylimidazoliumhexafluorophosphate

Pulse Sequence: stdlh

Solvent: DMSO

Data collected on: Oct 24 2013

Operator: Laputa20

VRMS-300 'Agilent-NMR'

Relax, delay 1.00 sec
Pulse 45.0 degrees
Aq. time 1.999 sec
Width 4500.5 Hz
24 repetitions

OBSERVE HL, 300.1143869 MHz
DATA PROCESSING
FT size 32768
Total time 4 min 24 sec
1-allyl-3-methylimidazolium tetrafluoroborate

\[
\text{BF}_4^- \quad \begin{array}{c}
\text{N} \\
\text{N} \\
\text{C} \\
\text{C} \\
\text{C} \\
\text{C} \\
\end{array} \quad \text{[CH}_2\text{CH}=	ext{CH}_2\text{]} 
\]
benzyl_methyl_Pf6

Sample Name:

Archive directory:

Sample directory:

P&dfile: FF_benzyl_methyl_pf6

Pulse Sequence: stdih (s2pul)

Solvent: DMSO

Data collected on: Oct 23 2013

Operator: laputa20

VNMRS-500 "Agilent-NMR"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.920 sec

Width 4500.5 Hz

128 repetitions

Observ. RL 300.1143955 MHz

DATA PROCESSING

FO size 32768

Total time 8 min 24 sec

![1-benzyl-3-methylimidazolium hexafluorophosphate](image)
Sample Name: benzyl_methyl_PF6
Archive directory:
Sample directory:
FidFile: FP_benzyl_methyl_PF6.c
Pulse Sequence: stdl3c (s2pul)
Solvent: DMSO
Data collected on: Oct 23 2013
Operator: laputa20
VNMRS-300 "Agilent-00m"
Relax. delay 1.000 sec
Pulse 103.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
3125 repetitions
OBSERVE C13, 75.4629262 MHz
DECOCUPLE H1, 300.1155338 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min

220 200 180 160 140 120 100 80 60 40 20 0 ppm
Ethyllindanone standard test sample
Recorded on 400-MR with OneNMR probe and PFT tuning

Sample Name: Ethyllindanone
Data Collected on: Agilent-600-0vnc-e300
Archive directory: /home/vnnc1/vnncsys/data/fidlib
Sample directory: Ethyllindanone
FidFile: Benzy1_methyl_bf4_R
Pulse Sequence: PROTON (x2pul)
Solvent: dmo
Data collected on: Jan 23 2014

Temp. 22.0 C / 295.1 K
Operator: lapesa20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4007.7 Hz
128 repetitions
OBSERVE H1, 300.1143878 MHz
DATA PROCESSING
FT size 16384
Total time 5 min 46 sec

![NMR Spectrum](image-url)
benzyl_methyl_BF4

Sample Name: Ethyllindanone
Data Collected on: Agilent-NMR-vnmrj300
Archive directory: /home/vnmr1/vnmrsys/data/fidlib
Sample directory: Ethyllindanone
Field: CARBON
Pulse Sequence: CARBON (x2pul)
Solvent: dmso
Data collected on: Feb 2 2014

Temp. 17.1 C / 290.2 K
Operator: jspate20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
932 repetitions

OBSERVE 13, 75.4638242 MHz
SINGLE 11, 300.1158928 MHz
Power 99 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min

-137.497
135.321
129.444
128.754
126.445
122.775
55.234
49.733
48.568
46.045
46.445
39.944
39.963
39.988
39.899

ppm
3-methyl-1-phenylimidazolium hexafluorophosphate
phenyl_methyl_PF6

Sample Name:
Archive directory:
Sample directory:
FidFile: FF phenyl methyl pf6
Pulse Sequence: std15c (a2pul)
Solvent: DMSO
Data collected on: Oct 16 2013

Operator: lexuta30
VNMRS-300 "Agilent-NMR"

Relax. delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.015 sec
Width 18761.7 Hz
4363 repetitions
OBSE4E C13, 75.6638642 MHz
DECultiple R1, 300.1153538 MHz
Power 44 dB
on during acquisition
c4 off during delay
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 7 hr, 49 min
phenyl_methyl_hbf4
Sample Name: Ethylindene
Data Collected on: Agilent-600-vnrs=300
Archive directory: /home/vuzi1/vnmrs/data/fidlib
Sample directory: Ethylindene
FidFile: phenyl_methyl_hbf4
Pulse Sequence: PROTON (tspul)
Solvent: dano
Data collected on: Jan 23, 2014

Temp. 24.0 C / 297.1 K
Operator: jangzi28
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4987.7 Hz
32 repetitions
OVERVIEW 1H. 300.1143873 MHz
DATA PROCESSING
FT size 16384
Total Time 1 min 27 sec
Sample Name:
Data Collected on:
Agilent-NMR-vnmr300
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: dmeo
Data collected on: Jan 28 2014

Temp. 22.0 C / 295.1 K
Operator: laguta20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4007.7 Hz
32 repetitions
OBSEVE 6L 300.1143861 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 27 sec

[Graph of NMR spectrum with peaks labeled]

3-methyl-1-vinylimidazolium hexafluorophosphate

Phosphorus tetrfluoride (PF₆⁻)
Sample Name:
Archive directory:
Sample directory:
FidFile: FP_vinyl_methyl_pf6_c
Pulse Sequence: std13c (s2pul)
Solvent: DMSO
Data collected on: Dec 25 2013

Operator: laputa20
VNMRS-300 "Agilent-MSK"

Relax, delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1824 repetitions
OBSERVE C13, 75.4438242 MHz
DECOUPLER H1, 306.1153538 MHz
Power 44 dB
on during acquisition
off during delay
WAUTE-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 Min

130 131 132 133 134 135 136 137
120 121 122 123 124 125
110 111 112 113 114 115
100 101 102 103 104 105
90 91 92 93 94 95
80 81 82 83 84 85
70 71 72 73 74 75
60 61 62 63 64 65
50 51 52 53 54 55
40 41 42 43 44 45
ppm

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Sample Name: Ethylindazone
Data Collected on: Agilent-NMR-vnmrs300
Archive directory: /home/voncl/vnmrs/data/fidlib
Sample directory: Ethylindazone
FidFile: vinyl_methyl_2F4_N

Pulse Sequence: PROTON (x2pul)
Solvent: dmeo
Data collected on: Jan 26 2014

Temp. 20.0 C / 293.1 K
Operator: laputa20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4807.7 Hz
16 repetitions

OBserve 81, 300.1145932 MHz
DATA PROCESSING
FT size 16384
Total time 0 min 43 sec

ppm

\[ 3.05 \]

\[ 2.94 \]

\[ 1.99 \]

\[ 1.00 \]

\[ 0.94 \]

\[ 0.95 \]

\[ 1.20 \]

\[ 1.21 \]

\[ 2.87 \]
1-butyl-3-ethylimidazolium tetrafluoroborate

Sample Name: 1-butyl-3-ethylimidazolium tetrafluoroborate
Archive directory: Sample directory:
File name: 1-butyl-3-ethylimidazolium tetrafluoroborate
Pulse Sequence: stdlh (2pug)
Solvent: DMSO
Data collected on: Dec 23 2013
Operator: laputa20
Vnmr 500 "Agilent-49E"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Avg. time 1.998 sec
Width 4500.5 Hz
128 repetitions
Observ. 61, 500.1142869 MHz
Data processing:
PT alk 3P/4
Total time 6 min 24 sec

\[\text{BF}_4^-\]
FP_butyl_ethyl_BF4_C

Sample Name:
Archive directory:
Sample directory:

FidFile: FP_butyl_ethyl_BF4_C

Pulse Sequence: stdls (x2pul)
Solvent: DMSO
Data collected on: Dec 25 2013

Operator: [Missing Information]

WBMS-300 "Agilent-NMR"

Relax: delay 1.000 sec
Pulse 103.8 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1024 repetitions

OBSERVE C13, 75.4638242 MHz
DECOUPLE H1, 300.115333 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated

DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 48 min
butyl-propyl-BF4

Sample Name: Ethylindaneone
Data Collected on: Agilent-MM5-nmrs300
Archive directory: /home/vusr1/mnrexps/data/fidlib
Sample directory: Ethylindaneone
Fidfile: PROTON

Pulses Sequence: PROTON (x2pul)
Solvent: dmac
Data collected on: Mar 31 2014

Operator: laputa20
Relax delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4807.7 Hz
Single scan
OBSERVE H1, 300.1143972 MHz
DATA PROCESSING
PT size 16384
Total time 0 min 3 sec

1-butyl-3-propylimidazolium tetrafluoroborate
1-butyl-3-isopropylimidazolium tetrafluoroborate

\[
\text{BF}_4
\]

N
N
\text{CH}_3
\text{CH}_2
Sample Name: butyl_isopropyl_BF4

Data Collected on: Agilent-NMR-vnmr300
Archive directory:
Sample directory:
File: CARBON
Pulse Sequence: CARBON {s2psl}
Solvent: dmo
Data collected on: Feb 4 2014

Temp. 22.0 C / 295.1 K
Operator: laputal20

Relax. delay 1.000 sec
Pulse 65.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
64 repetitions

OBSERVE C13, 75.4638242 MHz
GROUPS H1, 300.1158938 MHz
Power 39 dB continuously on

DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
1,3-dibutylimidazolium tetrafluoroborate

![Chemical Structure Image]
butyl-butyl_BF4

Sample Name: Butylbutyl
Data Collected on: Agilent-NMR-vnmrj300
Archive directory: /home/vnmrl/vnmrsys/data/fidlib
Sample directory: Butylbutyl
File type: CARBON

Pulse Sequence: CARBON {s2pol}
Solvent: dmos
Data collected on: Feb 1 2014

Temp. 21.0 C / 294.1 K
Operator: lapot20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec

Width 18939.4 Hz
999 repetitions

OBSERVE C13, 75.4638242 MHz
DECouple H1, 300.1159938 MHz

Power 39 dB
continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz
FT size 32768
Total time 31 min
1,3-diethylimidazolium tetrafluoroborate
Sample Name:  
Archive directory:  
Sample directory:  
FidFile: FP.IM_BBF4_ethylation_C  
Pulse Sequence: stdl3c (acpul)  
Solvent: DMSO  
Data collected on: Jan 6 2014  
Operator: leptha20  
VNMRS-300 “Agilent-NMR”  

Relax. delay 1.000 sec  
Pulse 103.8 degrees  
Acc. time 1.615 sec  
Width 18761.7 Hz  
1024 repetitions  
OBSERVE C13, 75.4638262 MHz  
DECOUPLE H1, 300.1155338 MHz  
Power 44 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 48 min
imidazolium_isopropyl_BF4

Sample Name: Ethylindanone
Data Collected on: Agilent-NMR-xwxs300
Archive directory: /home/venqy/venqsys/data/fidlib
Sample directory: Ethylindanone
FidFile: IM_dilispropyl_BBF4_H

Pulse Sequence: PROTON (x2pul)
Solvent: d2o
Data collected on: Feb 2 2014

Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.768 sec
Width 4007.2 Hz
Single scan
OBSERVE: H1, 300.1143870 MHz
DATA PROCESSING
FT size 16384
Total time 0 min 3 sec
Sample Name: Ethyldiimidazole
Data Collected on: Agilent-NMR-vnrna300
Archive directory: /home/vmr1/vmrna/data/fidlib
Sample directory: Ethyldiimidazole
FidFile: IM_dipropyl_NBF4_C

Pulse Sequence: CARBON (s2pul)
Solvent: deso
Data collected on: Feb 2 2014

Operator: lapsa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.465 sec
Width 18939.4 Hz
1024 repetitions

OBSERVE Cl3, 75.4659242 MHz
DECOPPE B1, 300.1158939 MHz
Power 39 dB continuously on

DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 31 min
Sample Name: Diethylimidazolium 4-methylbenzenesulfonate
Data Collected on:
Agilent-NMR-vnmr300
Archive directory: /home/vnmr1/vnmr3sys/data/fidlib
Sample directory: Ethylindeneone
FidFile: PROTON
Pulse Sequence: PROTON (s2pol)
Solvent: deo
Data collected on: Feb 1 2014

Temp. 21.0°C / 294.1 K
Operator: laputa20

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 4807.7 Hz
32 repetitions
OVEREVE 1H, 300.114K72 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 27 sec
imidasole_ethyl_PTSA

Sample Name: Ethylimidazole
Data Collected on: Agilent-NMR-vnmrs300
Archive directory: /home/vnmrs/vnmrs/data/fidlib
Sample directory: Ethylimidazole
FidFile: imidasole_ethyl_PTSA_c

Pulse Sequence: CAUSON (x2pul)
Solvent: dmso
Data collected on: Feb 1 2014

Temp. 21.0 C / 294.1 K
Operator: laputa20
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.865 sec
Width 18939.4 Hz
1024 repetitions
OBSERVE C13, 75.4430242 MHz
DECOUPLE H1, 300.1159338 MHz
Power 39 dB
continuously on
MUTE-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32766
Total time 31 min
$^{19}$F NMR spectrum
1-butyl-3-methylimidazolium tetrafluoroborate