## **Methimazole Based Ionic Liquids**

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#### Chemicals.

Iodomethane, chlorobutane, methimazole and trifluoromethane sulfonate were used as received from the manufacturer.

#### Instrumentation.

<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on 200, 300 and 400 MHz spectrometers and chemical shifts (ppm) were calibrated relative to an external TMS standard. <sup>19</sup>F NMR and <sup>15</sup>N NMR spectra were recorded on 300 MHz spectrometer and calibrated (ppm) relative to external CCl<sub>3</sub>F and NH<sub>4</sub>OH standard, respectively.

**Electrochemical Instrumentation**. Voltammetric studies were undertaken under 3-electrode potentiostated conditions using an electrochemical workstation. The working electrode was a 1mm diameter glassy carbon electrode disk carefully polished, ultrasonically rinsed in absolute ethanol and dried with lint free tissue paper before use. The counter electrode was a platinum wire and the quasi-reference electrode a silver wire. The oxidation of 5 mM of Ferrocene or reduction of 5 mM cobalticinium hexafluorophosphate was used to provide as an internal potential reference standard against which data are reported. The ionic liquid solution was purged with highly purified nitrogen for at least 30 min. prior to commencing electrochemical experiments and then maintained under a nitrogen atmosphere.

#### Analysis.

### Water content.

The water content of **3a**, **3b** and **3c** was determined coulometrically using the Karl Fisher method. Duplicate measurements agreed to within 5%.

### Melting point and glass transition temperatures.

Temperature dependent phase behavior over the range of -150 °C to 120 °C was obtained by differential scanning calorimetry using 10-20 mg masses of sample. Thermal scans below room temperature were calibrated using the cyclohexane solid-solid transition and melting points of -87.0 °C and 6.5 °C, respectively. Higher than room temperature data were calibrated against the melting point of indium (156.6 °C). Transition temperatures are reported as the peak maximum of the thermal transition.

#### **Decomposition temperatures**.

Thermogravimetric analysis was conducted in a flowing dry nitrogen atmosphere (50 mL/min.) at temperatures between 25 and 500 °C with a heating rate of 10 °C min<sup>-1</sup>. Calibration was achieved using the Curie points of four reference materials, alumel, perkin-alloy, iron and nickel. Platinum pans were used with sample masses between 5 and 10 mg.

## Viscosity.

Viscosity was determined from the time for a fixed volume to flow through a narrow orifice in a calibrated glass viscometer. Duplicate measurements were undertaken in a dry box.





<sup>1</sup>H NMR of **2a** [NH proton is at 11.09 ppm]

# <sup>1</sup>H NMR (neat) of **2a**





## <sup>1</sup>H NMR (CDCl<sub>3</sub>) of **2a** [No NH proton detected due to low concentration]

<sup>13</sup>C NMR of **2a** 



# <sup>15</sup>N NMR of **3a**



<sup>13</sup>C NMR of **3a** 



# <sup>1</sup>H NMR (neat) of **3a**



## <sup>1</sup>H NMR (CDCl<sub>3</sub>) of **3a**





### <sup>1</sup>H NMR (CDCl<sub>3</sub>) of **2b**



# <sup>13</sup>C NMR (CDCl<sub>3</sub>) of **3b**













<sup>13</sup>C NMR of **3c** 

