

Supporting Information

Synthesis and electrochemical characterization of bis(fluorosulfonyl)imide-based protic ionic liquids

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Synthesis of $\text{PYR}_{\text{H}_4}\text{FSI}$

For the synthesis of the PIL 1-butylpyrrolidine (Aldrich >98%) was distilled directly before use. Ultrapure HCl (37%), sodium bis(fluorosulfonyl)imide (NaFSI), $\text{PYR}_{14}\text{FSI}$ (Iolitec) and ethyl acetate (ACS grade, >99.5%) were used as received.. At the end of the synthesis $\text{PYR}_{\text{H}_4}\text{FSI}$ was dried under vacuum at 60 °C. The water content of the ILs was measured using coulometric Karl-Fisher titration, and was found to be less than 10 ppm. The conductivity and the viscosity of the prepared electrolytes were determined as reported elsewhere.

Electrode preparation

Lithium iron phosphate (LFP) composite electrodes were prepared according to the literature [1]. The composition of the electrodes was 85 wt.% of the active material LFP, 10 wt.% of the conductive agent Super C65[®] and 5 wt.% of carboxymethyl cellulose (CMC) as a binder. The electrode mass loading was 1.3 mg cm⁻² and the electrode area was 1.13 cm². All the electrochemical tests were carried out with 3-electrode Swagelok[®] type cells. The cells were assembled in an argon-filled glove box with oxygen and water contents lower than 1 ppm. LFP-based electrodes were used as working electrodes, a silver wire was used as the pseudo-reference electrode and an oversized activated carbon-based electrode was used as the counter electrode. For all experiments, a glass microfiber separator (Whatman) drenched with 100 μL of electrolyte was used.

The electrochemical measurements were carried out using a MACCOR Series 4000 battery tester. Constant current cycling (CC) was carried out at 30 °C using current densities ranging from 1 C to 10 C taking into account the theoretical capacity of LFP of 174 mAh g⁻¹.

Reference

[1] T. Vogl, S. Menne, R.-S. Kühnel and A. Balducci, *Journal of Materials Chemistry A*, 2014.

NMR spectra Pyr_{H4}TFSI

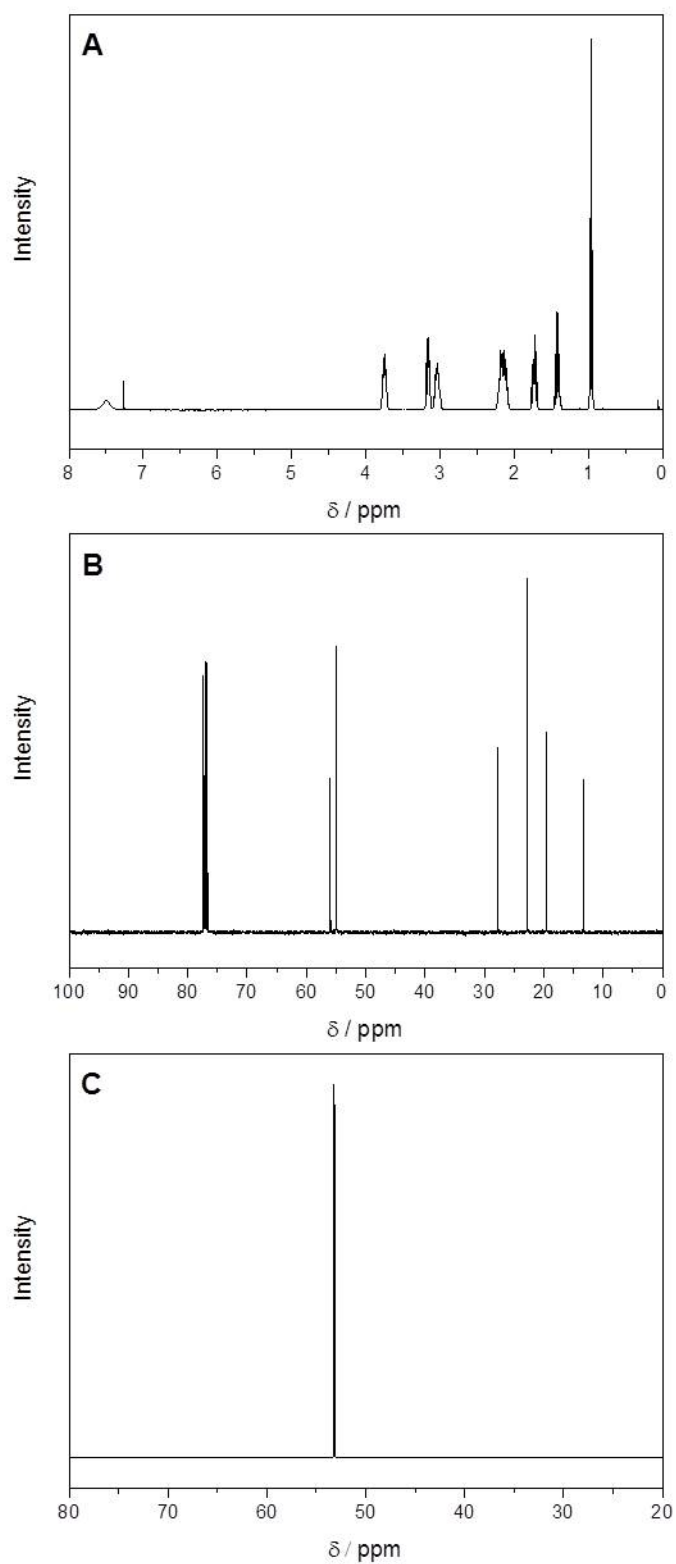


Fig. 1: ¹H-NMR (a), ¹³C-NMR (B) and ¹⁹F-NMR (C) spectra of Pyr_{H4}TFSI.