Design, preparation and characterization of ionic liquid 1,3-disulfonic acid benzimidazolium chloride as an efficient and recyclable catalyst for the synthesis of tetrahydropyridine under solvent-free conditions

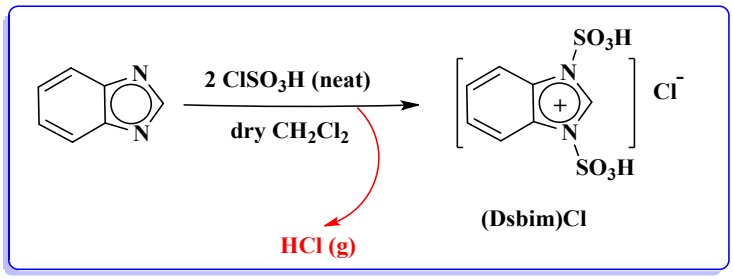
Mohsen Abbasi

Department of Chemistry, Lamerd Branch, Islamic Azad University, Lamerd, Fars, Iran, E-mail: mohsen.abbasi90@gmail.com.

Preparation of the ionic liquid

To a round-bottomed flask (50 mL) containing benzimidazole (0.708 g, 6.0 mmol) in dry CH_2Cl_2 (15 mL), was added chlorosulfonic acid (1.40 g, 12 mmol) dropwise over a period of 5 min at room temperature. After the addition was completed, the reaction mixture was stirred for 3 h under pressure of nitrogen (to remove the produced HCl), stand for 5 min, and the CH_2Cl_2 was decanted. The residue was washed with dry CH_2Cl_2 (3 × 50 mL) and dried under vacuum to give [Dsbim]Cl as a viscous pale yellow oil in (1.97 g, 98% yield).

Spectral data: IR (Nujol): υ 574, 679, 750, 886, 1063, 1189, 1331, 1530, 1631, 2536-3432 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 8.09 (t, J = 7.45 Hz, 2H), 8.43 (t, J = 7.81 Hz, 1H), 8.90 (d, J = 5.76 Hz, 2H), 13.64 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 112.80, 125.61, 136.79, 143.24; MS: m/z= 315 (M^++1), 314 (M^+), 279 (M^+-Cl), 232 (M^+-SO_3H), 198 (M^+-SO_3H and Cl), 162 ($2SO_3H$), 154 (M^+-$2SO_3H$), 118 (M^+-$2SO_3H$ and Cl) and 81 (SO_3H).



Scheme 1 General formulation for the preparation of 1,3-disulfonic acid benzimidazolium chloride [Dsbim]Cl).

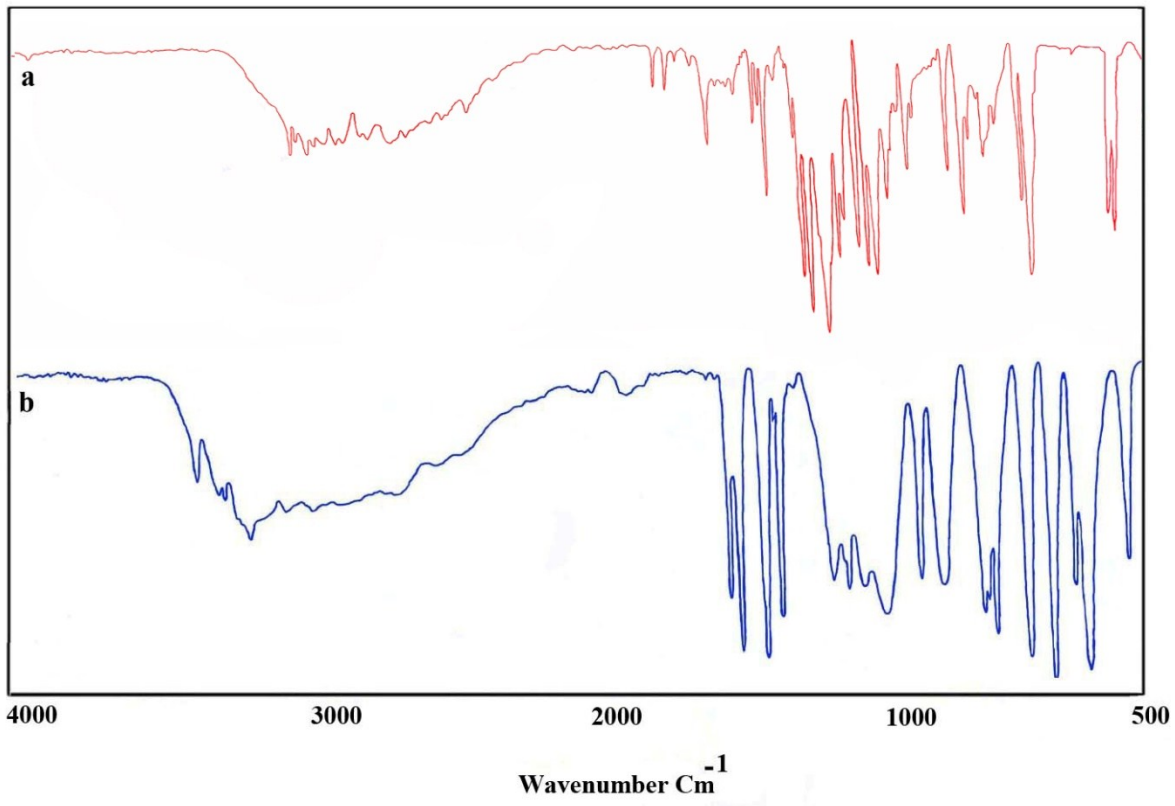


Fig. 1 FT-IR spectra of benzimidazole (a) and 1,3-disulfonic acid benzimidazolium chloride ([Dsbim]Cl) (b)

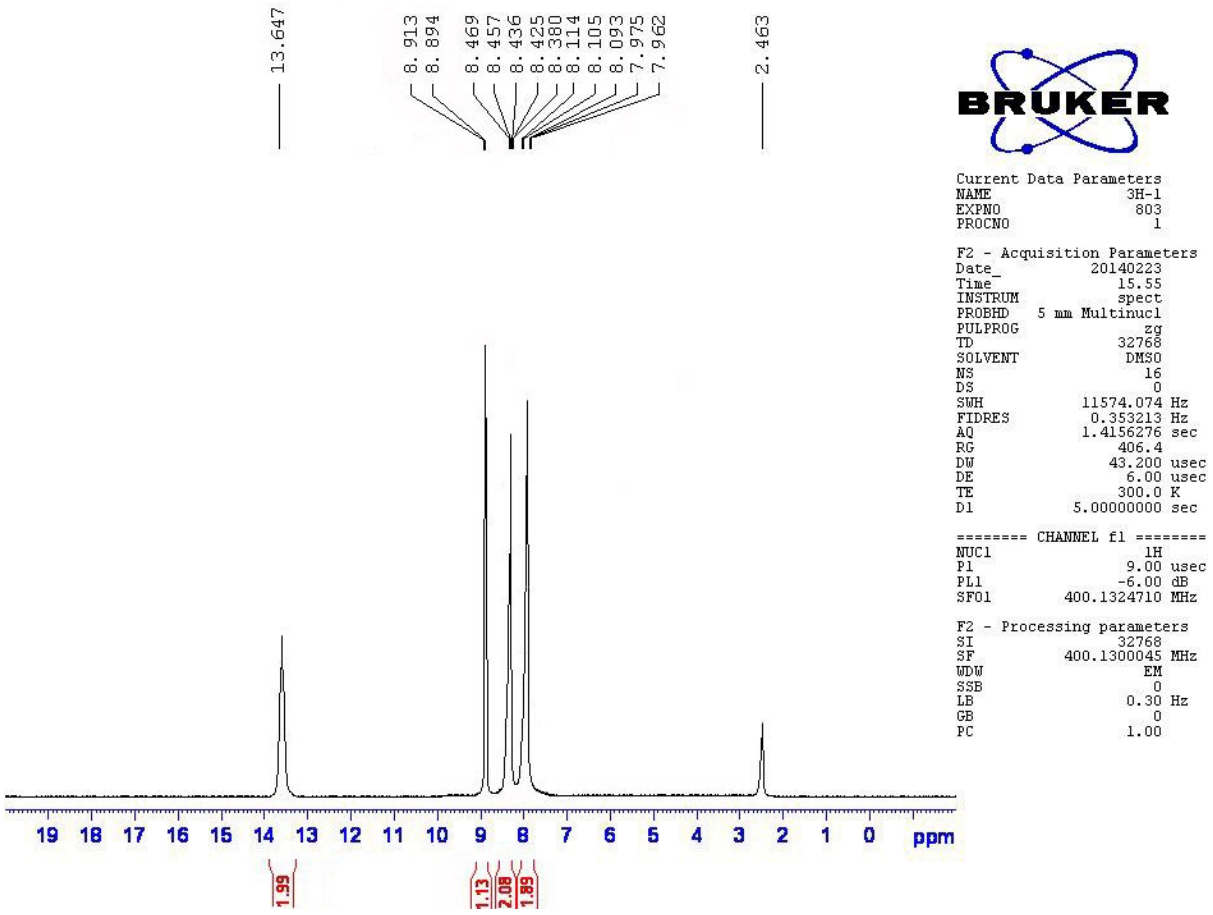


Fig. 2 ^{1}H NMR spectrum of 1,3-disulfonic acid benzimidazolium chloride ([Dsbim]Cl)

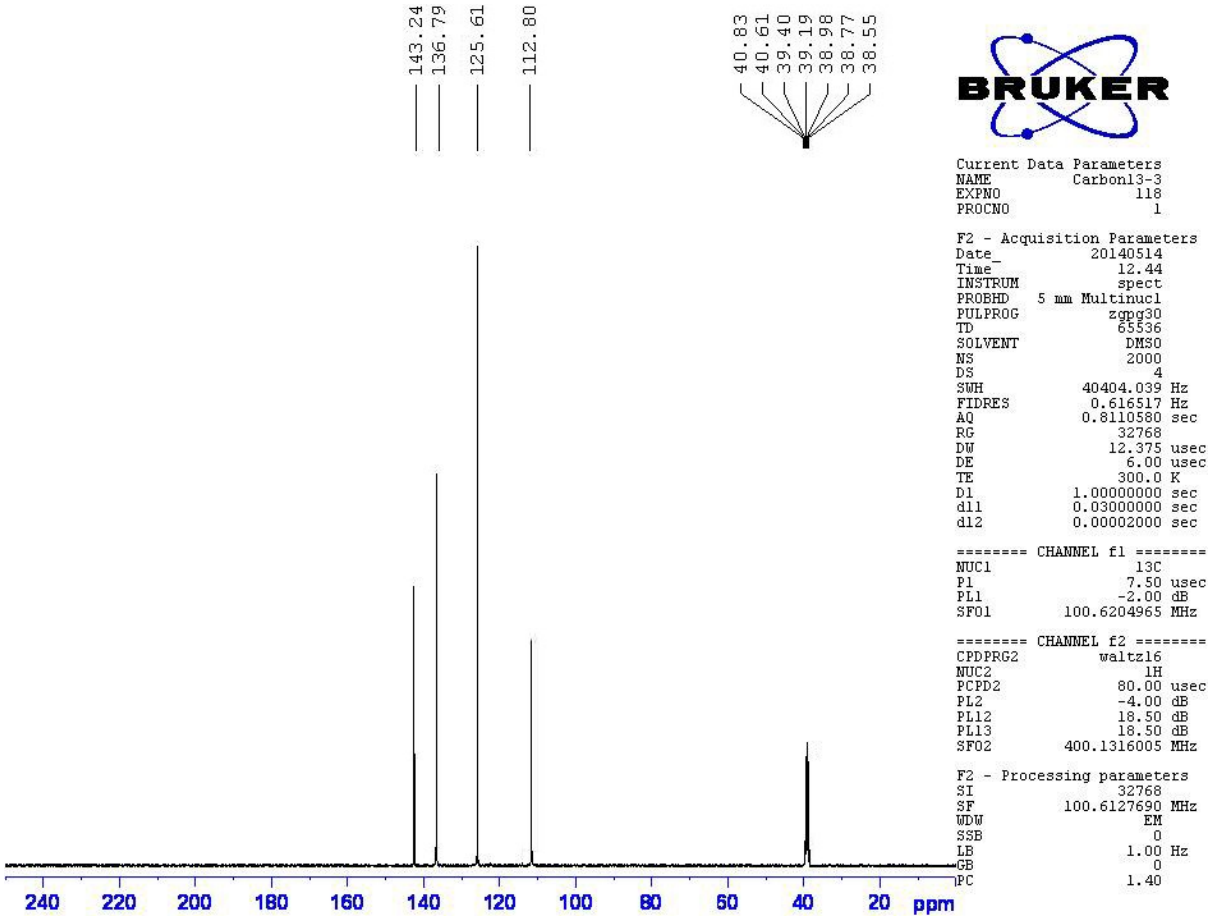


Fig. 3 The ^{13}C NMR spectrum of 1,3-disulfonic acid benzimidazolium chloride ([Dsbim]Cl)

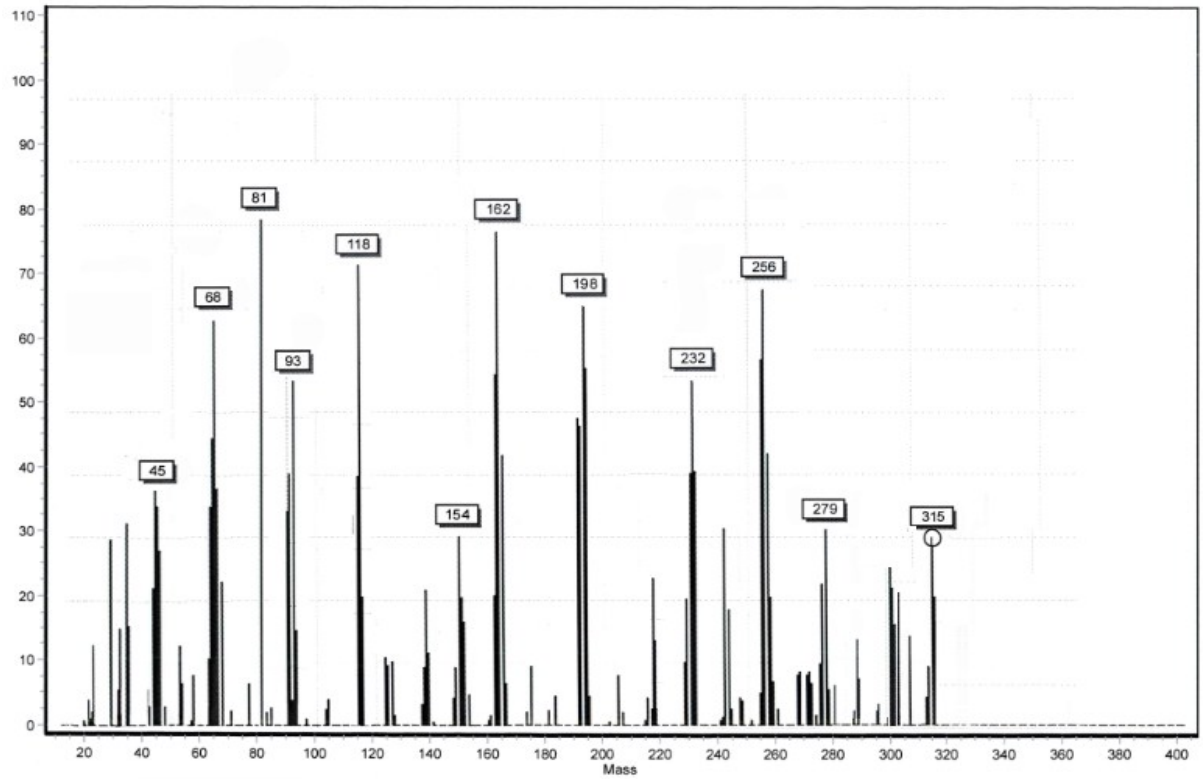


Fig. 4 Mass spectra of 1,3-disulfonic acid benzimidazolium chloride ([Dsbim]Cl)