Optical and XPS Evidence for the Electrochemical Generation of an *N*-Heterocyclic Carbene and its CS_2 Adduct from the Ionic Liquid [bmim][PF_6]

Supplementary Information

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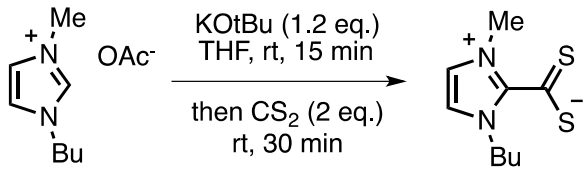
Synthesis of the Dithiocarboxylate Adduct 3 from [bmim][OAc] (4):



4 **3**
67% yield

A 100-mL, oven-dried, round-bottomed flask equipped with a magnetic stir bar was charged with 1-butyl-3-methylimidazolium acetate **4** (300 mg, 1.51 mmol). The flask was evacuated and refilled with nitrogen three times. The ionic liquid was dissolved in 20 mL of anhydrous THF, and KOtBu (204 mg, 1.82 mmol) was added as a solid. The resulting clear, light yellow solution was stirred for 15 min at room temperature under nitrogen. Afterwards, CS_2 (183 μL, 3.03 mmol) was added via syringe, and the color turned dark red immediately. The reaction mixture was stirred for 30 min, and directly concentrated under reduced pressure using a Rotary evaporator. Purification by silica gel flash column chromatography (DCM:EtOAc 1:1) gave pure imidazolium dithiocarboxylate **3** (217 mg, 67%) as a red-colored solid.

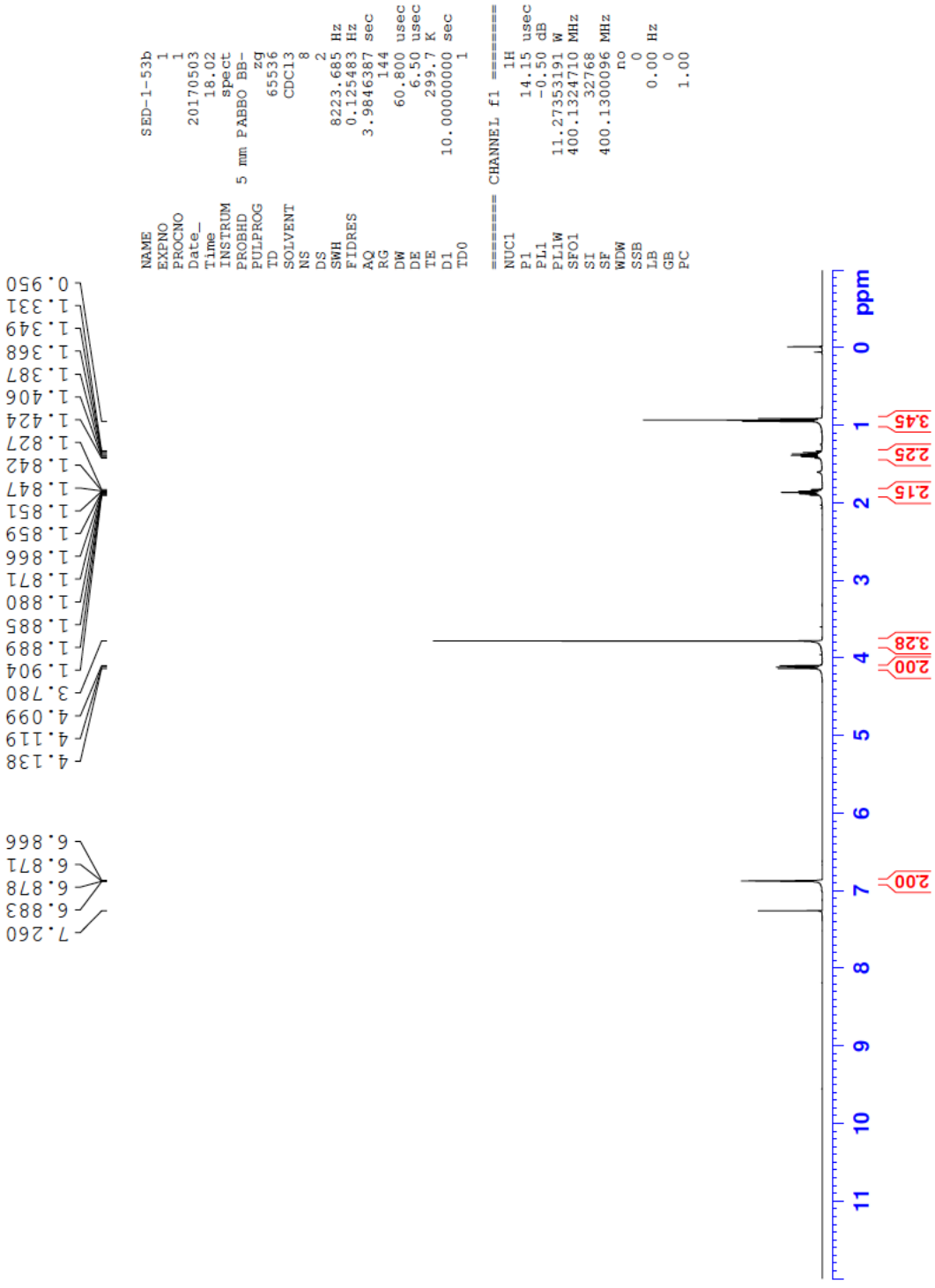


Figure S1. ^{1}H-NMR spectrum of **3** (400 MHz, $CDCl_3$) prepared from [bmim][PF_6]

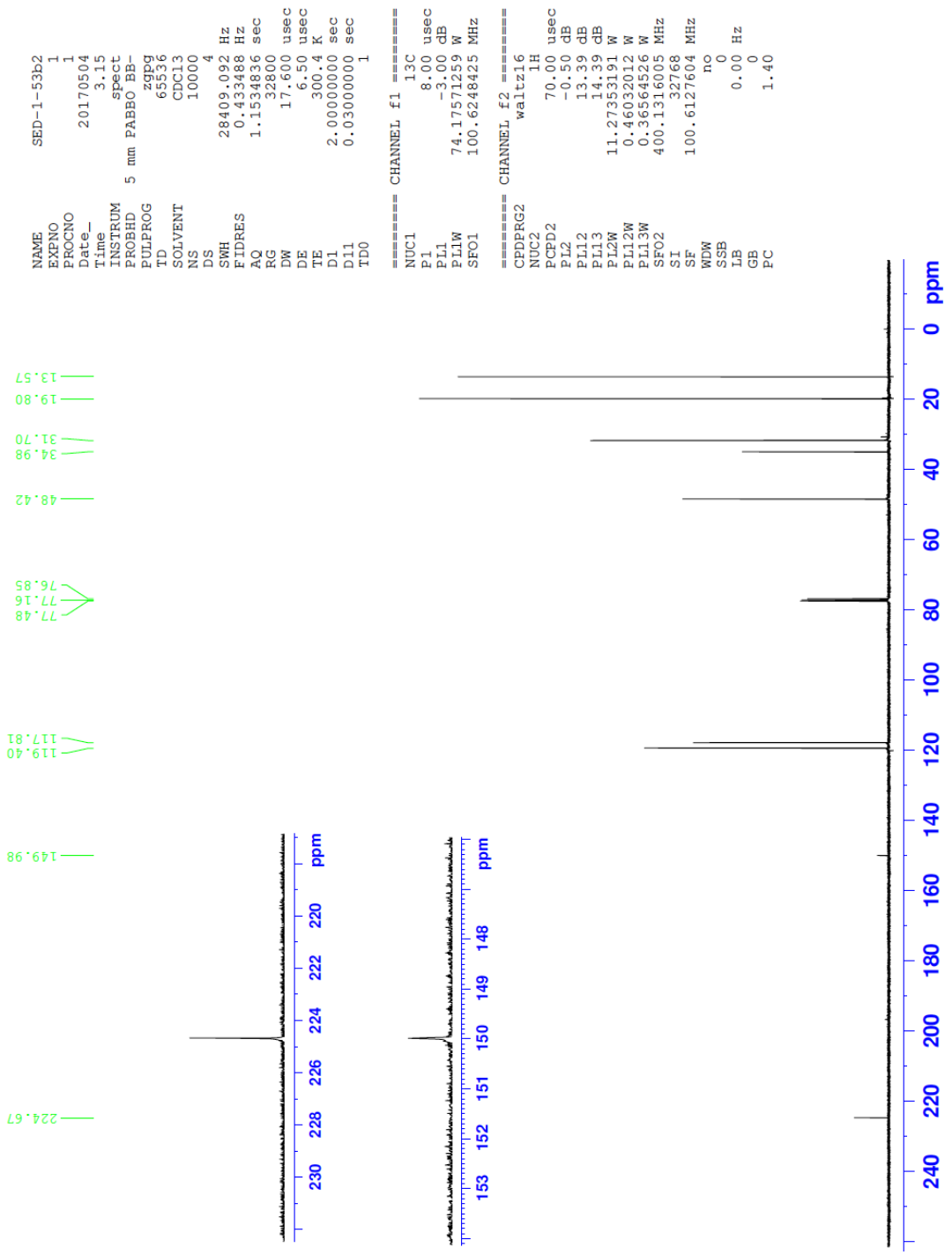


Figure S2. ^{13}C-NMR spectrum of **3** (100 MHz, $CDCl_3$) prepared from [bmim][PF_6]

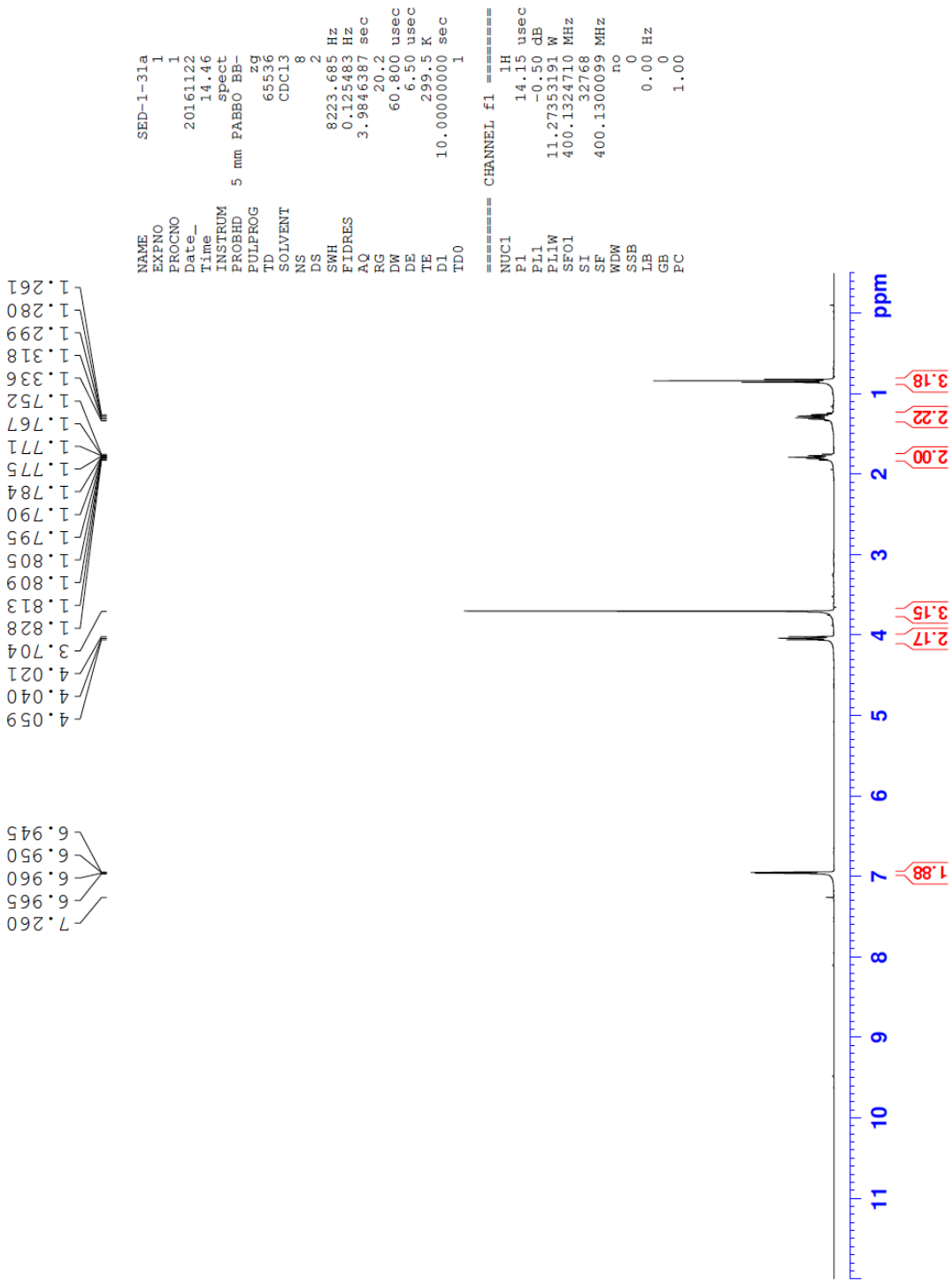


Figure S3. ^{1}H-NMR spectrum of **3** (400 MHz, $CDCl_3$) prepared from [bmim][OAc]

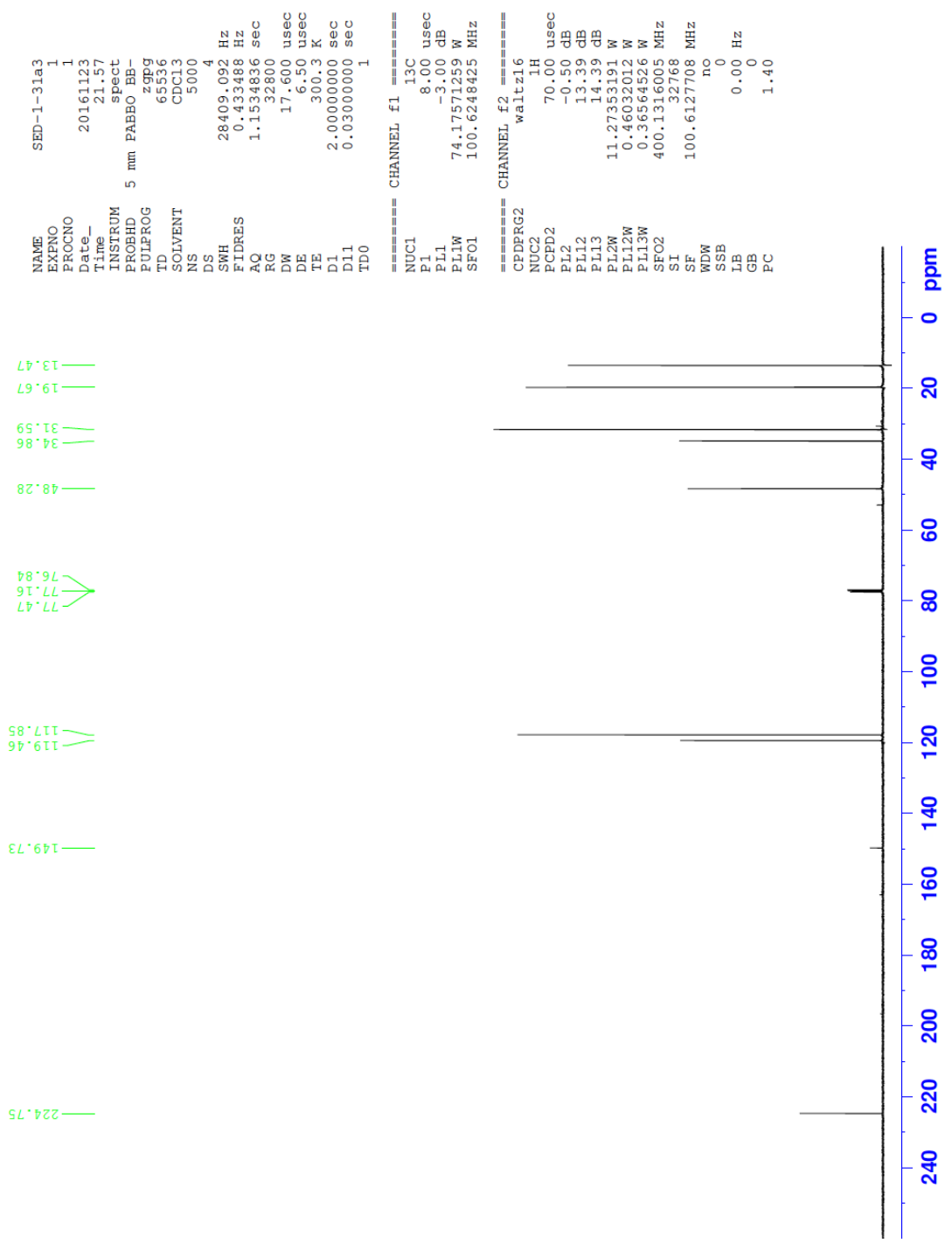


Figure S4. ^{13}C-NMR spectrum of **3** (100 MHz, $CDCl_3$) prepared from [bmim][OAc]