A solventless route to 1-ethyl-3-methylimidazolium fluoride hydrofluoride, [C_2mim][F]·xHF.

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Supporting Information

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General Experimental Methods

Single Crystal X-Ray Diffraction Analysis (SCXRD): A single crystal of 1-ethyl-3-methyl-imidazolium hydrogen carbonate 5 was mounted on a glass fiber attached on a goniometer head of a CCD diffractometer. X-rays were generated by a Mo/Kα radiation source (λ = 0.71073 Å) equipped with a graphite monochromator. Data collection was conducted at -100 ^{o}C which was achieved by streaming cold nitrogen over the crystal. Final unit cell parameters were determined by least squares refinement of the hemispherical data set obtained from 20 second exposures. Data were corrected for Lorentz and polarization effects and absorption using SADABS.[1] The initial structure solution was carried out using the direct methods option in SHELXTL version 5.[2] The positions of all non-hydrogen atoms were refined anisotropically. The hydrogen atoms were added and allowed to refine unconstrained in order to obtain proper close contact interactions.

Thermogravimetric Analysis (TGA): The range of thermal decomposition temperatures was determined by TGA using a thermogravimetric analyzer. Samples were analyzed on a platinum pan with dried air as the purge gas in the temperature range of 30-600 °C. The amount of IL used was between 2–10 mg. The experimental protocol included three sections with initial heating of the sample to 75 °C with a constant heating rate of 5 °C/min, an isotherm at 75 °C for 30 minutes to ensure all moisture in the sample was removed, and heating the sample to 600 °C with a constant heating rate of 5 °C/min.

Nuclear Magnetic Resonance (NMR) Analysis: ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded using DMSO-D_6 or CD_3OD as solvent. The solvent was used as the internal standard. Chemical shifts (δ) are given in ppm; coupling constants (J) in Hz. splitting patterns are designated as s (singlet), br (broad), d (doublet), dd (doublet of doublets), t (triplet), q (quartet), m (multiplet).

Fourier Transform Infra Red (FT-IR) Analysis: All spectra were recorded with a FT-IR diamond ATR Crystal spectrometer.

CHNF Elemental Analysis: The $[C_2mim][F]\cdot xHF$ compound was analyzed by an external company.

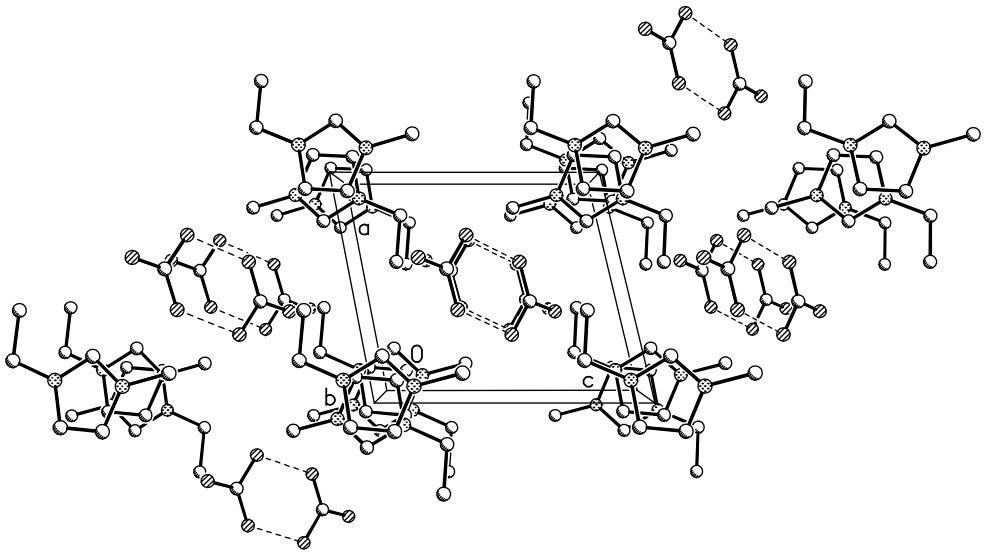

Figure 1: Packing diagram viewed down b of 1-ethyl-3-methylimidazolium hydrogen carbonate **5**, formed by reaction of 1 ethyl-3-methylimidazolium-2-carboxylate with water. Note the presence of the usual carboxylate hydrogen bonded dimer motif.

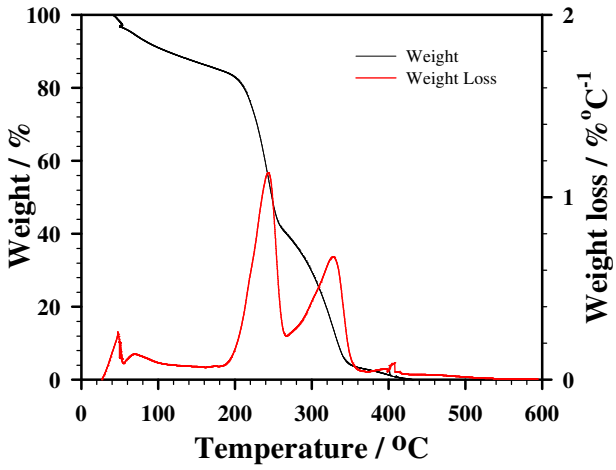

Figure 2: TGA of [C_2mim][F]·xHF **6**.

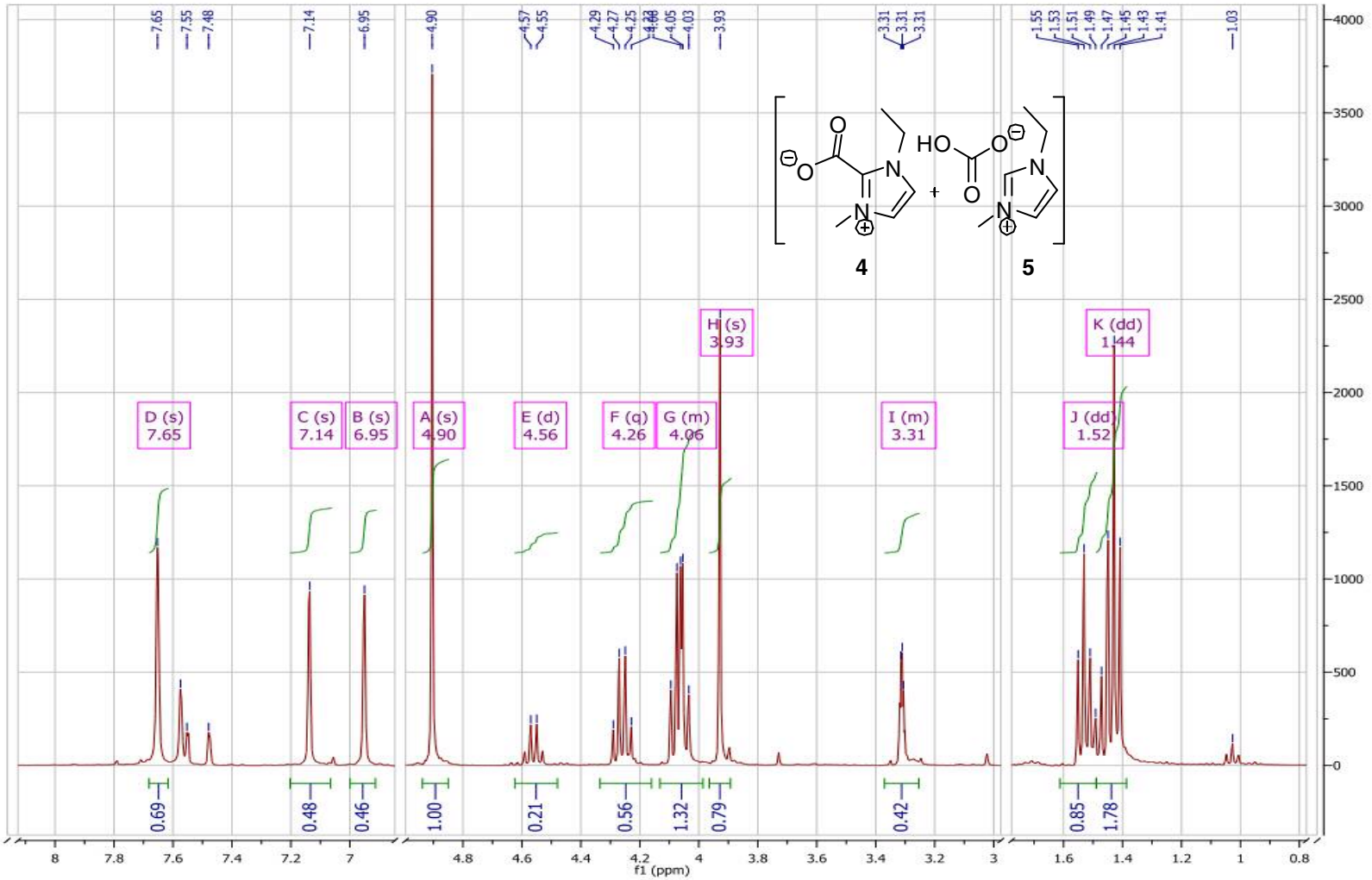

Figure 3: [1]H-NMR of the mixture of 1-ethyl-3-methylimidazolium-2-carboxylate **4** and 1-ethyl-3-methylimidazolium hydrogen carbonate **5** in CD_3OD.

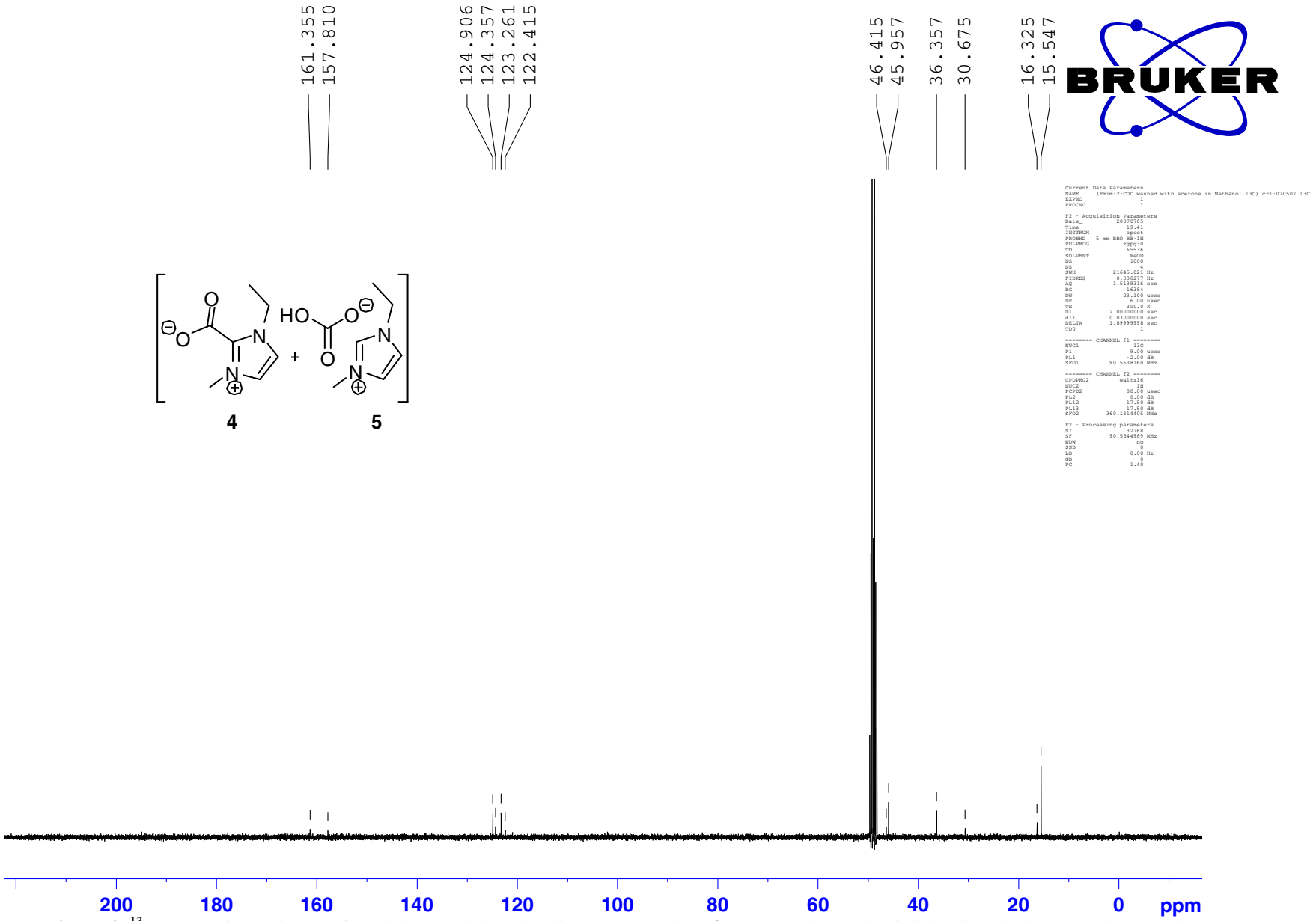

Figure 4: ^{13}C-NMR of the mixture of 1-ethyl-3-methylimidazolium-2-carboxylate **4** and 1-ethyl-3-methylimidazolium hydrogen carbonate **5** in CD_3OD.

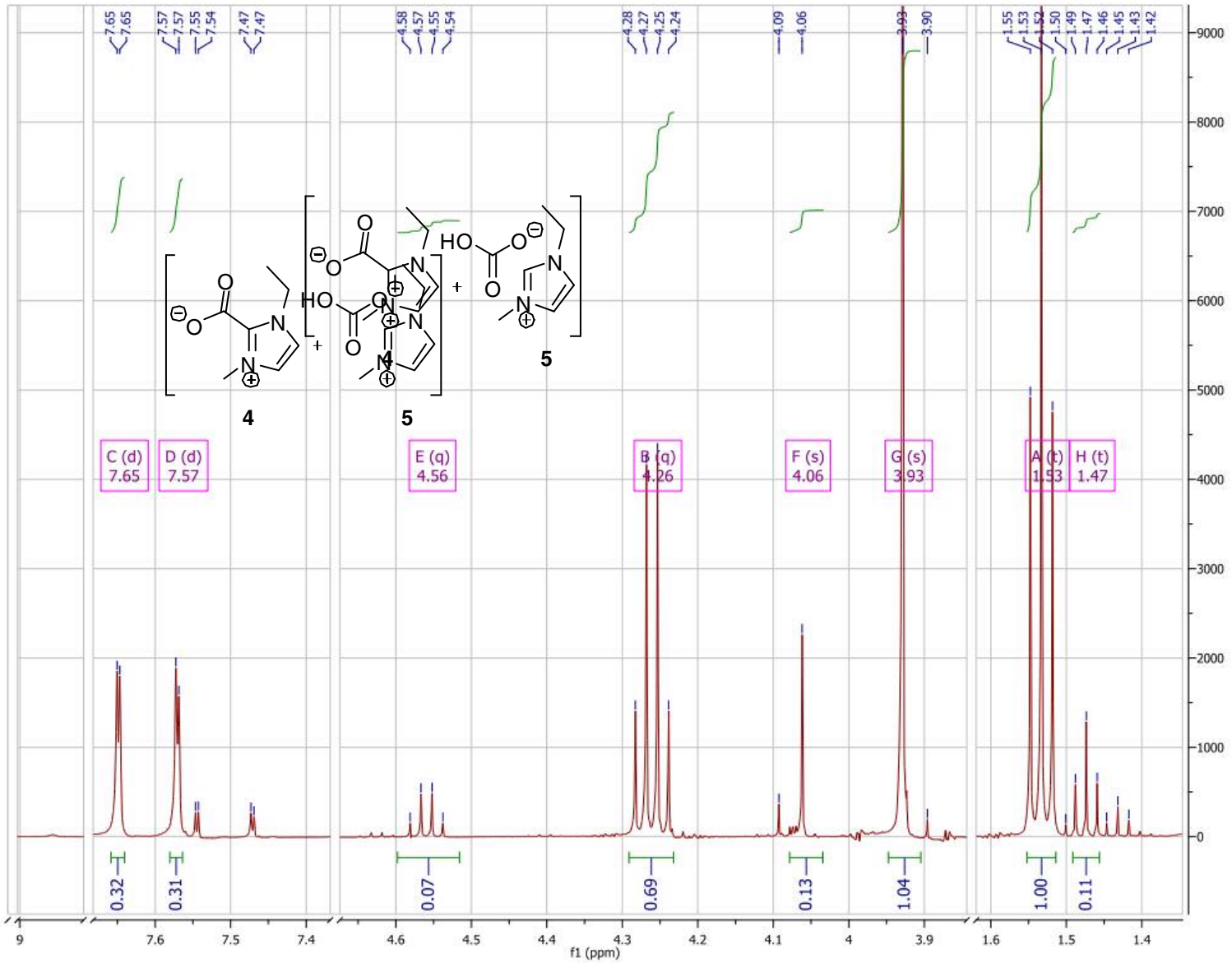

Figure 5: [1]H-NMR of the mixture of 1-ethyl-3-methylimidazolium-2-carboxylate **4** and 1-ethyl-3-methylimidazolium hydrogen carbonate **5** in DMSO.

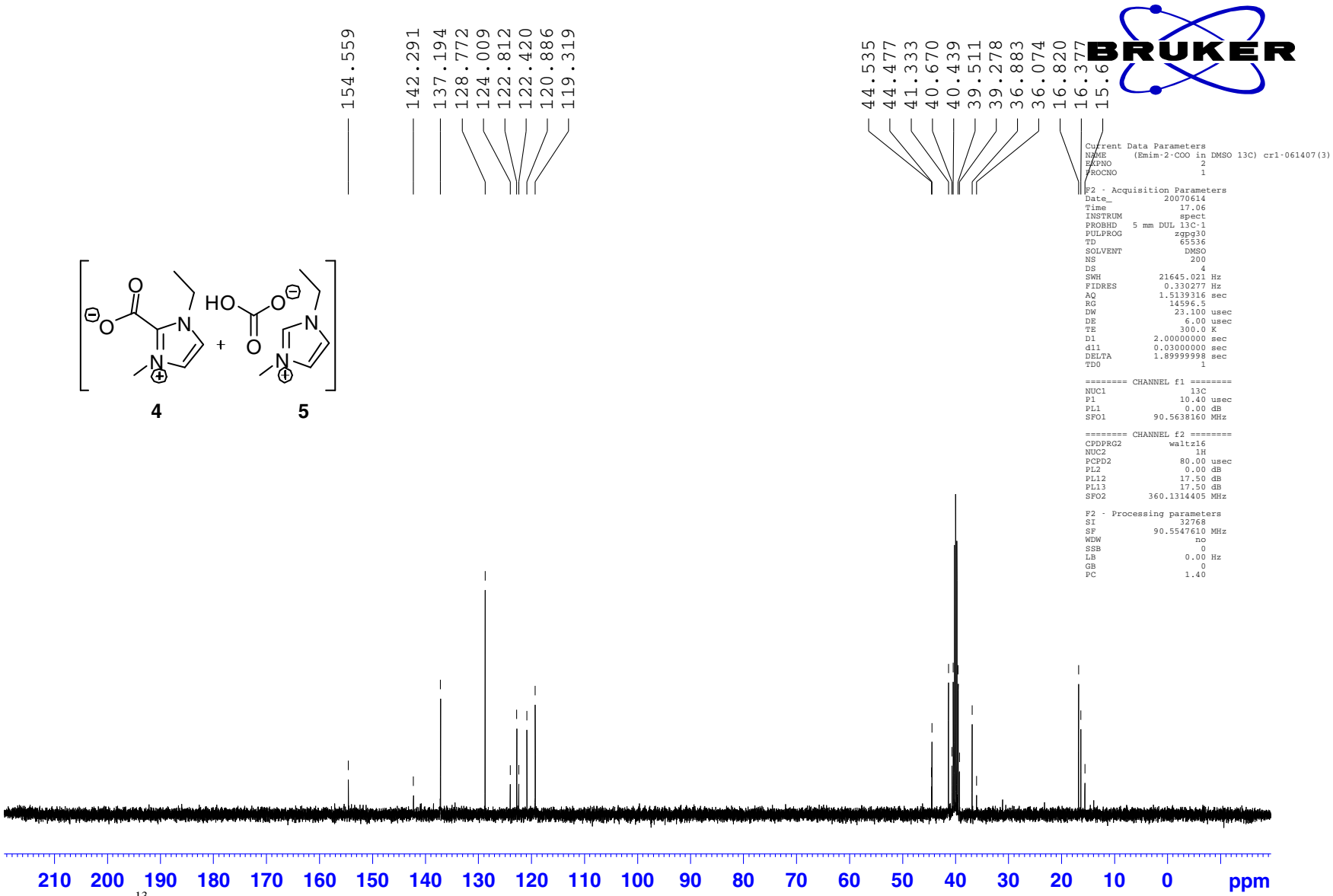

Figure 6: ^{13}C-NMR of the mixture of 1-ethyl-3-methylimidazolium-2-carboxylate **4** and 1-ethyl-3-methylimidazolium hydrogen carbonate **5** in DMSO.

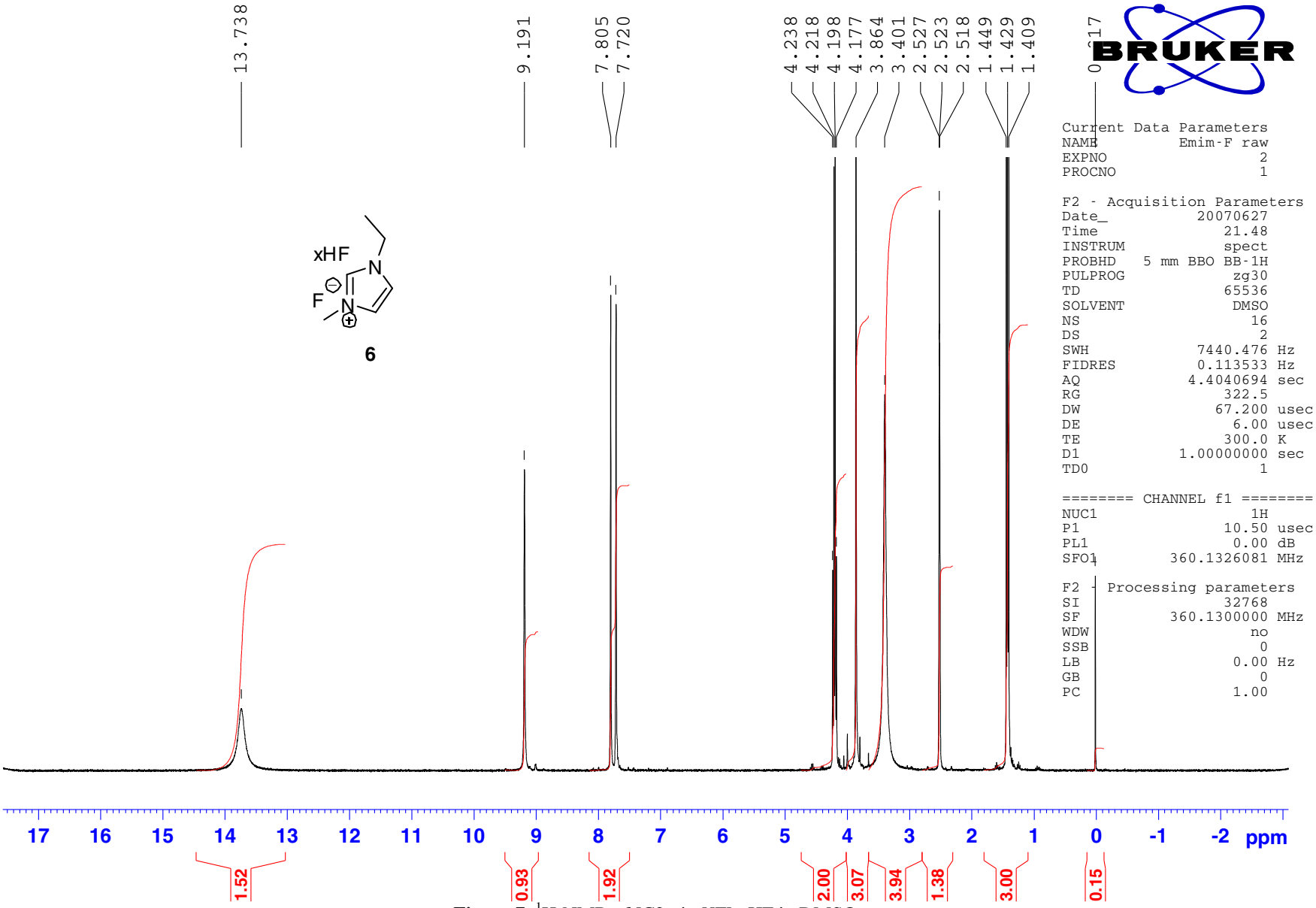

Figure 7: ^{1}H-NMR of [C2mim][F]·xHF in DMSO.

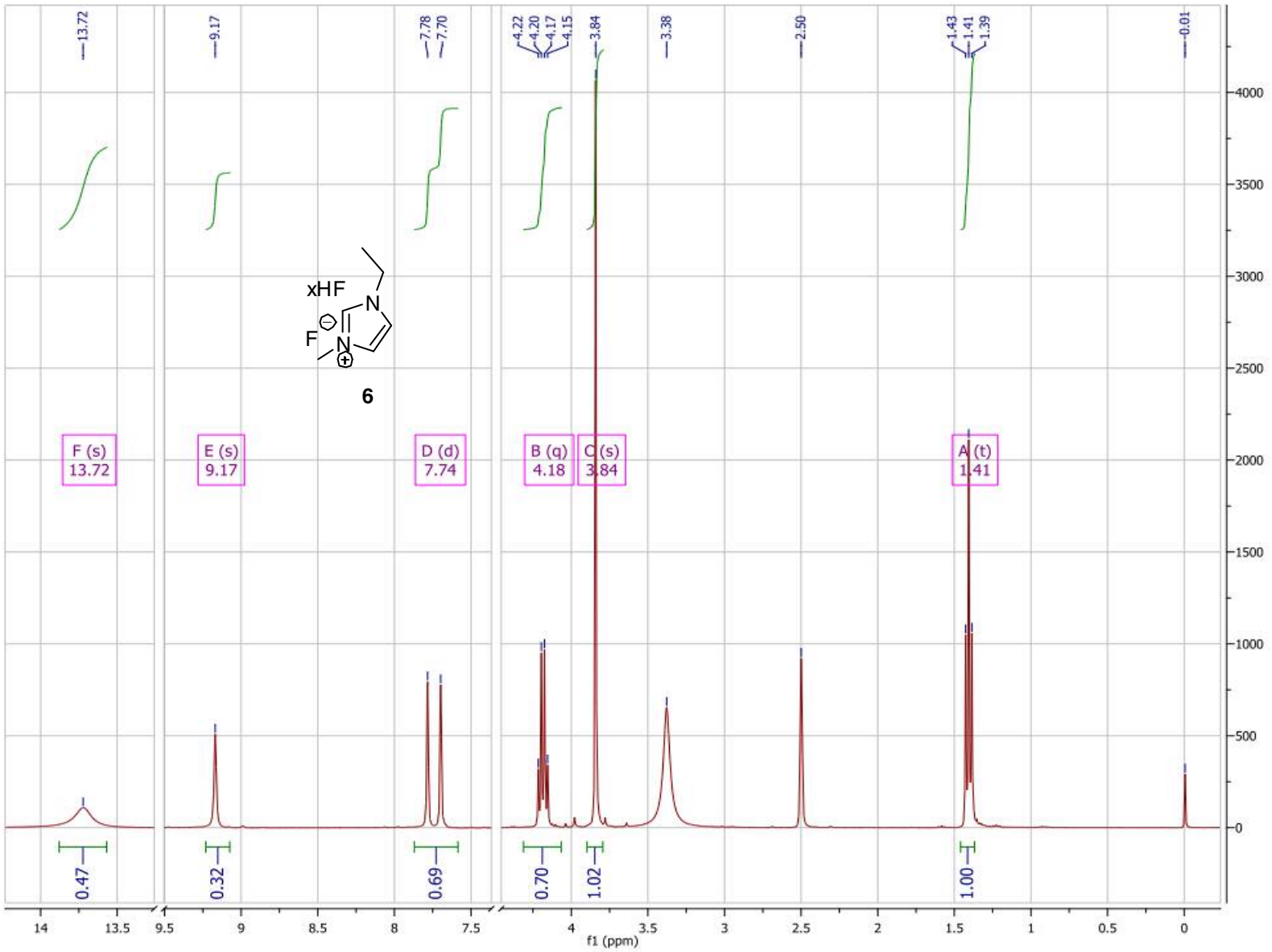

Figure 8: ^{1}H-NMR of [C2mim][F]·xHF in DMSO.

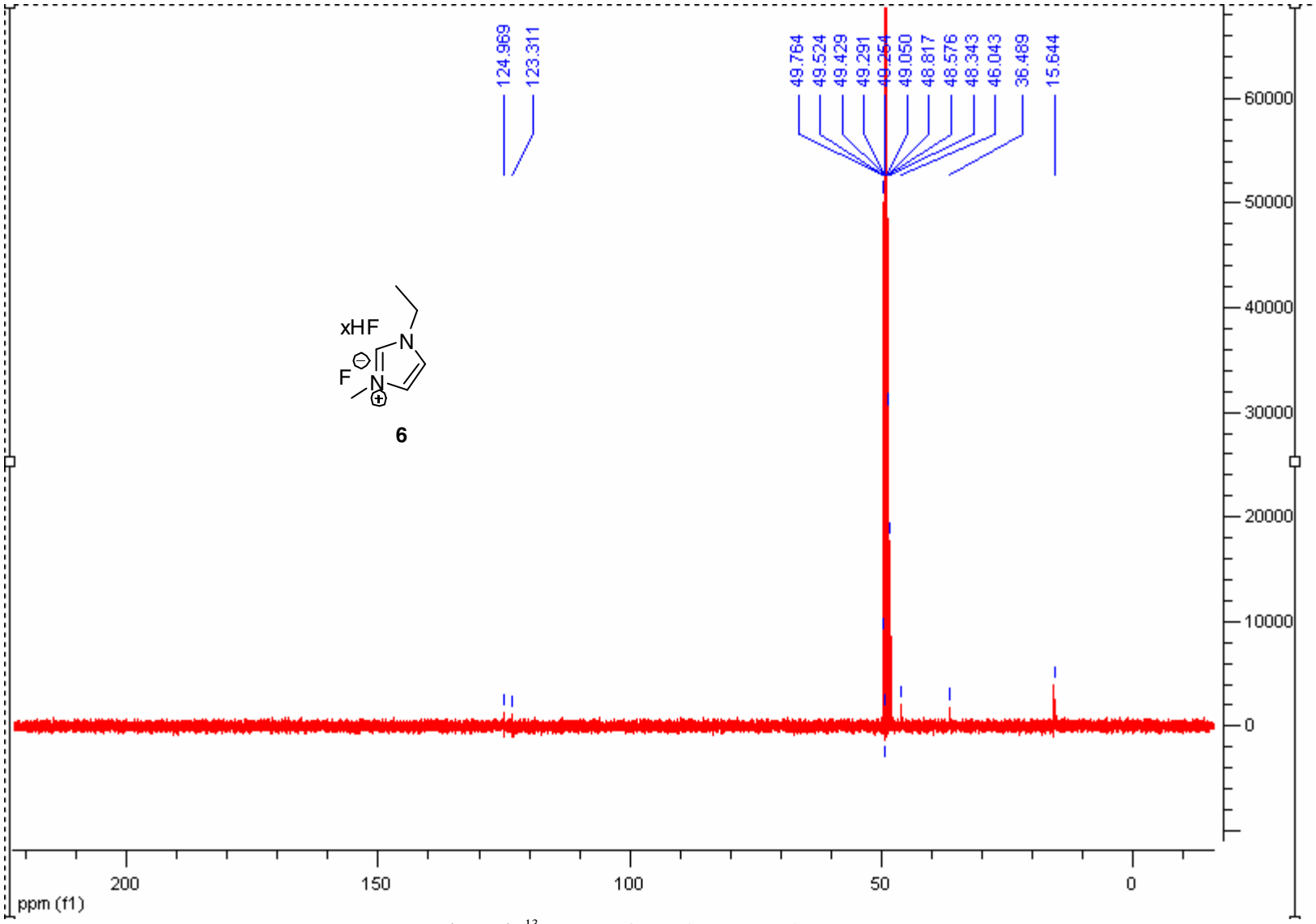

Figure 9: ^{13}C-NMR of [C2mim][F]·xHF in DMSO.

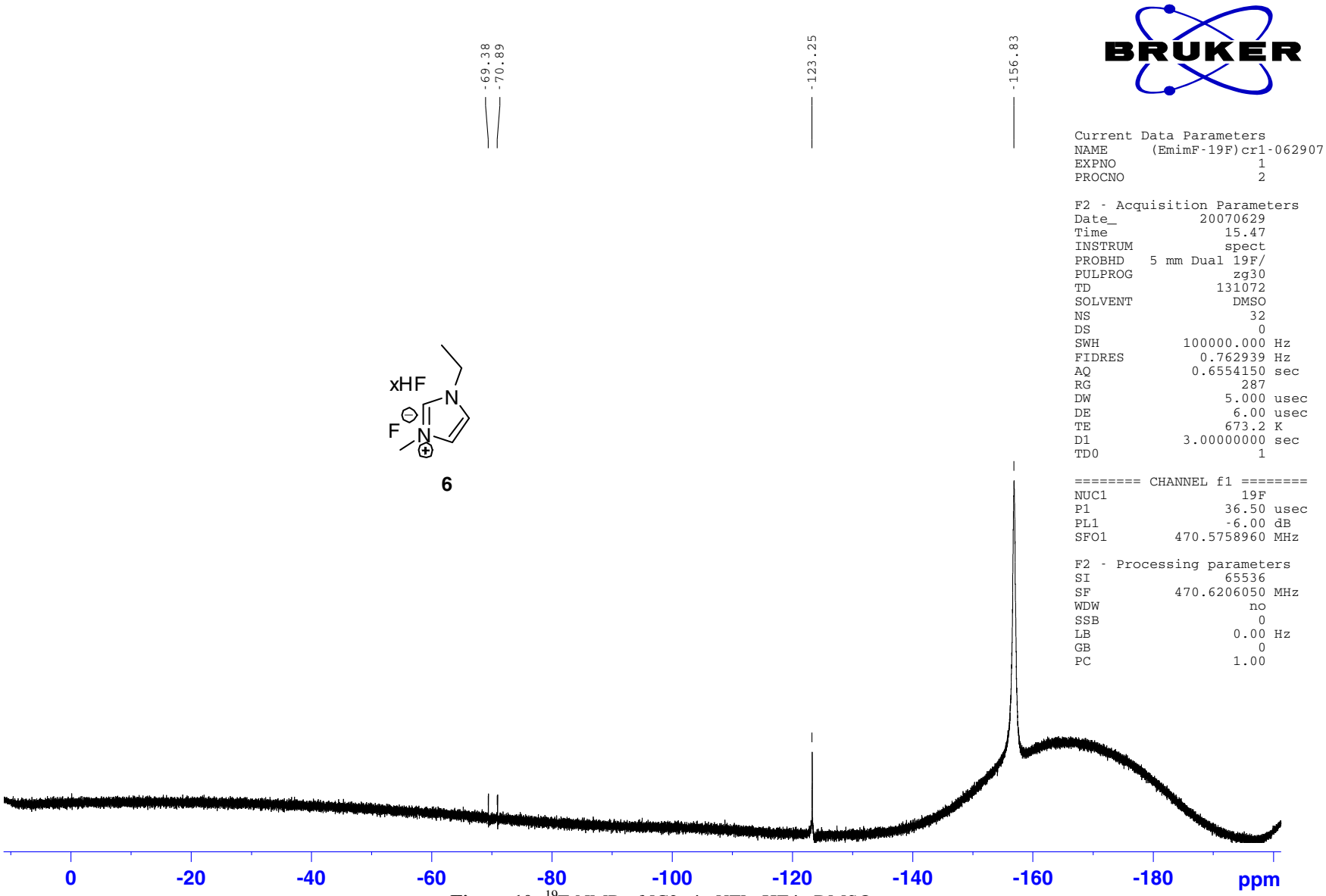

Figure 10: ^{19}F-NMR of [C2mim][F]·xHF in DMSO.

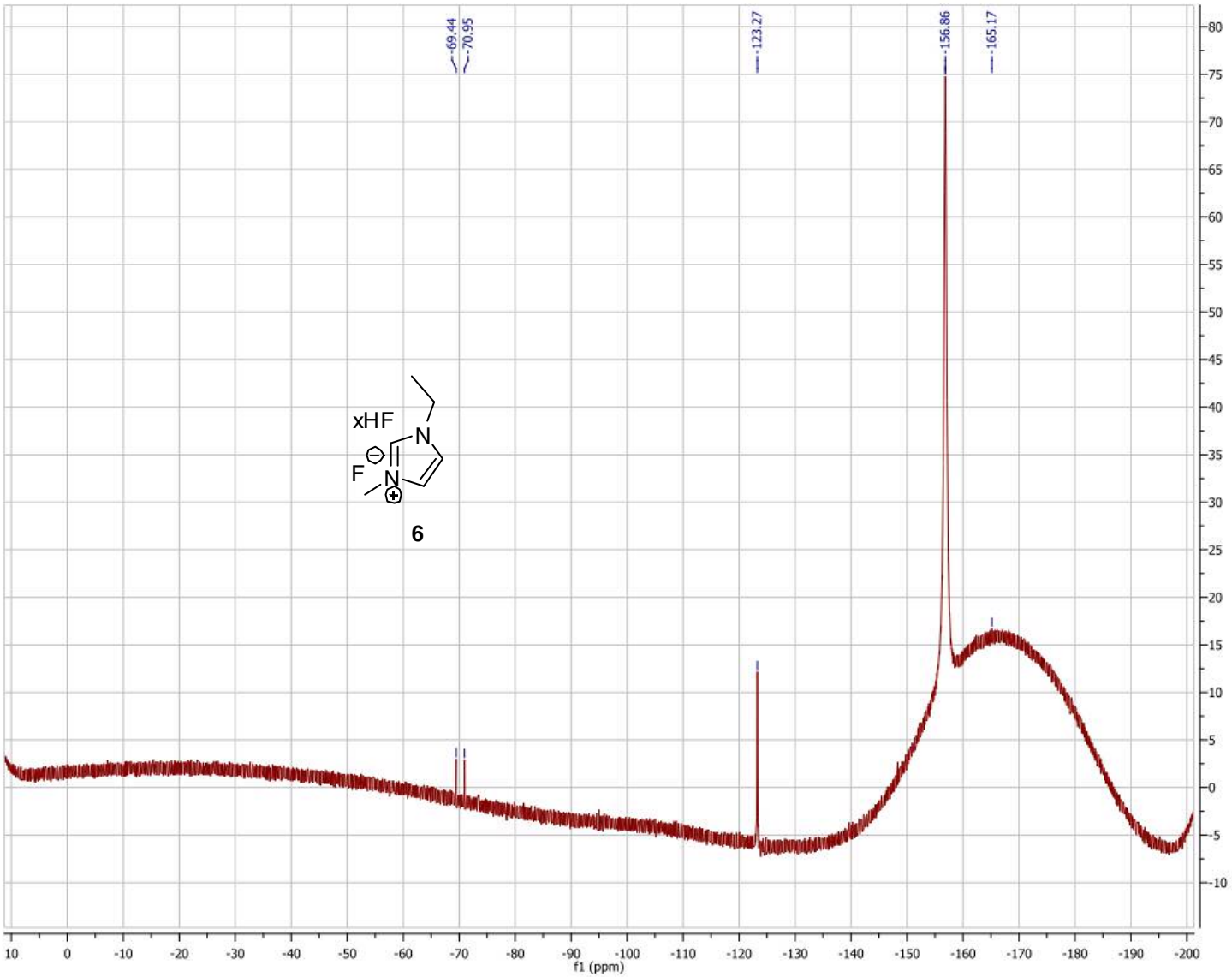

Figure 11: ^{19}F-NMR of [C2mim][F]·xHF in DMSO.

Literature

1 G. M. Sheldrick, Program for Semiempirical Absorption Correction of Area Detector Data, University of Göttingen, Germany, 1996.
2 G. M. Sheldrick, SHELXTL, version 5.05, Siemens Analytical X-ray Instruments Inc., 1996.