**5-amino-2-methylpyridinium hydrogen fumarate: an XRD and NMR crystallography analysis**

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Data recorded by Emily K. Corlett

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In the following, the names of all raw data files from the solid-state NMR measurements and different calculations are presented:

1. **Raw files for the PXRD experiments**

**Figure S1:** PXRD data of 5-amino-2-methylpyridinium hydrogen fumarate recorded on a Panalytical X’Pert Pro MPD equipped with a curved Ge Johansson monochromator, λ = 1.541 Å: **EC070\_5-50-13hr\_1.xrdml, EC070\_5-50-13hr\_1.xy** (recorded on 07/02/2019)

1. **Raw files from NMR experiments**

**Figure 2:** 1D MAS NMR spectra of 5-amino-2-methylpyridinium hydrogen fumarate:

**(top)** 1H (600 MHz) one-pulse MAS (60 kHz): **Figure 2 [1]** (recorded 21/03/2019)

**(bottom)** 1H (700 MHz)-13C CP MAS (12.5 kHz): **Figure 2 [2]** (recorded 24/05/2019)

**Figure 3:** 2D MAS (60 kHz) NMR spectra of 5-amino-2-methylpyridinium hydrogen fumarate:

1. 1H (600 MHz) DQ spectrum recorded with one rotor period of BaBa recoupling: **Figure 3 [1]** (recorded 21/03/2019)
2. 1H (600 MHz) SQ NOESY spectrum with tmix = 300 ms: **Figure 3 [2]** (recorded 22/03/2019)
3. 14N-1H (600 MHz) HMQC spectrum with 8 rotor periods of R3 recoupling: **Figure 3 [3]** (recorded 23/03/2019)

**Figure 4:** 1H (700 MHz)-13C CP (500 μs) HETCOR MAS (12.5 kHz) NMR spectra of 5-amino-2-methylpyridinium hydrogen fumarate: **Figure 4 [1]** (recorded 24/05/2019)

1. **For the calculations, the initial CIF file, the CIF file after geometry optimisation and the magres-files for the full crystal structure and isolated molecules**

5a2mpf150.cif (as determined by single-crystal X-ray diffraction)

5a2mpf150-out.cif

5a2mpf150-nmr.magres

5a2mp150-nmr.magres

hf150-nmr.magres

5a2mpf300.cif (as determined by single-crystal X-ray diffraction)

5a2mpf300-out.cif

5a2mpf300-nmr.magres

1. **TGA and DSC data**

**Figure S2:** DSC and TGA of 5-amino-2-methylpyridinium hydrogen fumarate: **TGA\_DSC.xlsx** (recorded 10/07/2019)