Supporting Information for:
**Direct Solid-State NMR Spectroscopic Evidence for the NH_4AlF_4 Crystalline
Phase Derived from Zeolite HY Dealuminated with Ammonium
Hexafluorosilicate**

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Figure S1. ^{27}Al triple-quantum (3Q) MAS NMR spectrum of the washed HY/AHFS-1.6 sample. Asterisks denote spinning sidebands. The spectrum was obtained using a three-pulse sequence with z-filter.^{9b} The length of the first and second hard pulse ($B_1 = 110$ kHz) used to excite the triple-quantum coherences ($\pm 3Q$) and to transfer them back to zero quantum were $6.50\ \mu\text{s}$ and $1.75\ \mu\text{s}$, respectively. A soft pulse ($B_1 = 22$ kHz) with a length of $11.50\ \mu\text{s}$ was applied after the z-filter period ($200\ \mu\text{s}$). The 3QMAS data were processed with a shearing transformation prior to the complex Fourier transformation to obtain a 2D spectrum with an isotropic frequency component along the F1 spectral axis and an anisotropic frequency along the F2 axis.

Figure S2. (A) ^{27}Al and (B) ^{19}F MAS NMR spectra of HY/AHFS-*x*, prepared in the presence of NH_4OAc , where *x* = (a) 0.3, (b) 0.5, (c) 0.6, and (d) 1.0. These samples were prepared in a 50 mL of 3M aqueous solution of NH_4OAc by following the method of Skeels and Breck.⁵ The ^{27}Al signal of NH_4AlF_6 was enlarged as shown in the inset.