Synthesis and characterization of aluminium fluoride hydrate with cationic vacancies: structure, thermal stability and acidic properties

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Supporting information

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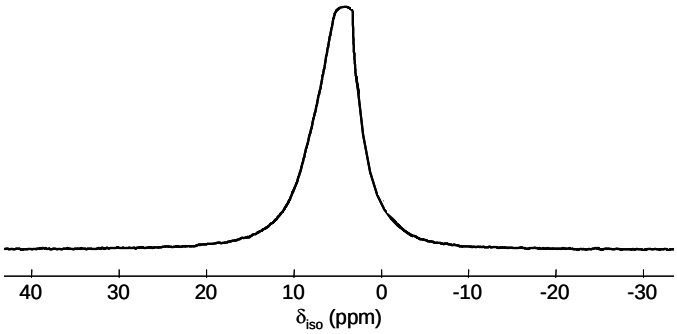

Figure 1. ^{1}H MAS (30 kHz) NMR spectrum of $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ after annealing at 773 K during 24 h under Ar.

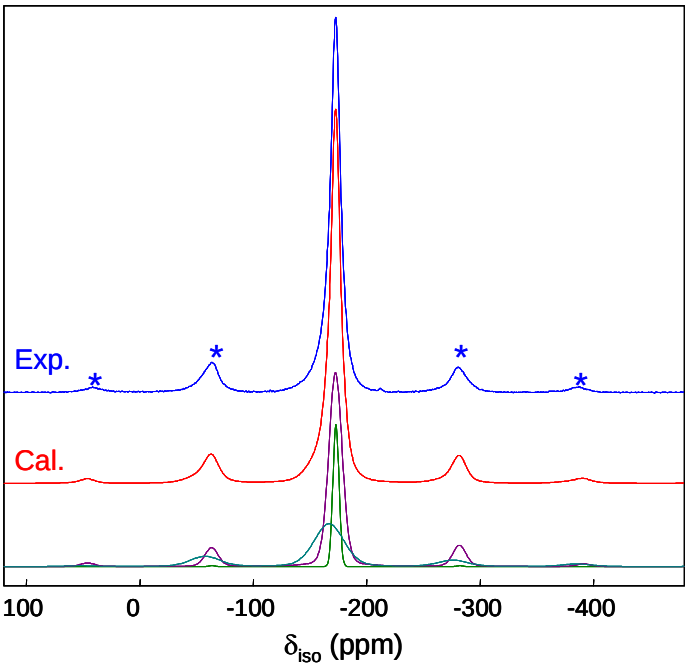

Figure 2. Experimental and calculated ^{19}F MAS (30 kHz) NMR spectra of $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ after annealing at 773 K during 24 h under Ar. The star symbols indicate the spinning sidebands. The three individual contributions to the reconstructed spectrum are shown.

Table 1. Isotropic chemical shift δ_{iso} (ppm), line width (ppm) and relative intensity (%) as deduced from the reconstruction of the ^{19}F NMR spectrum of $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ after annealing at 773 K during 24h under Ar.

Line	δ_{iso} (±0.5)	Width (±0.5)	Intensity (±0.5)
1	-172.9	6.3	13.7
2	-172.5	13.9	54.8
3	-166.8	30.9	31.5

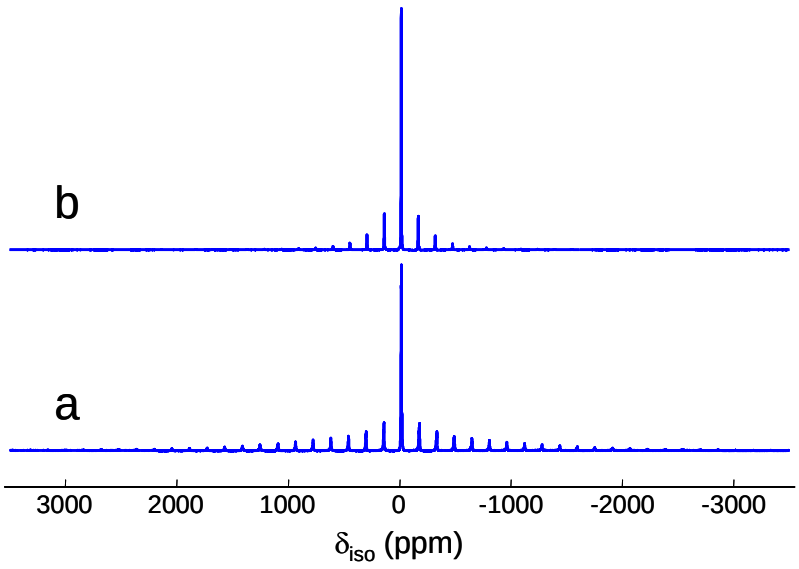

Figure 3. ^{27}Al MAS (30 kHz) one pulse NMR spectrum recorded at 17.6 T of (a) $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ and (b) $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ after annealing at 773 K during 24 h under Ar.

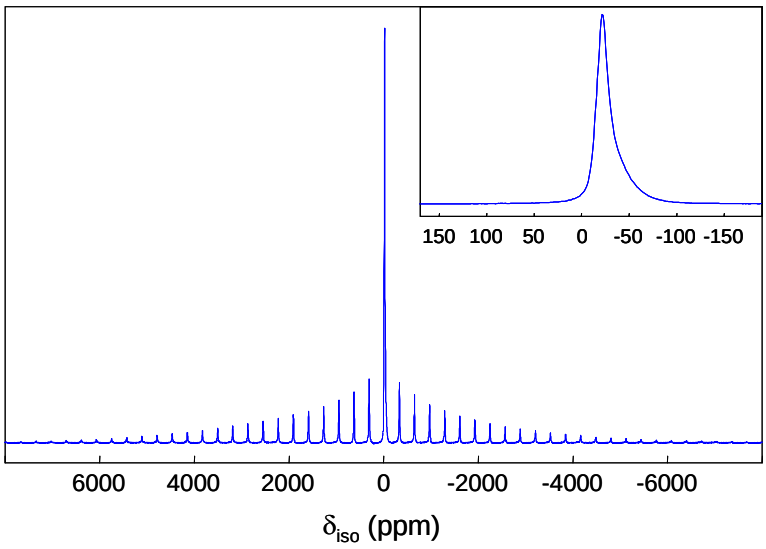

Figure 4. ^{27}Al MAS (25 kHz) one pulse NMR spectrum of $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ recorded at 7 T. Central transition is presented in the inset.

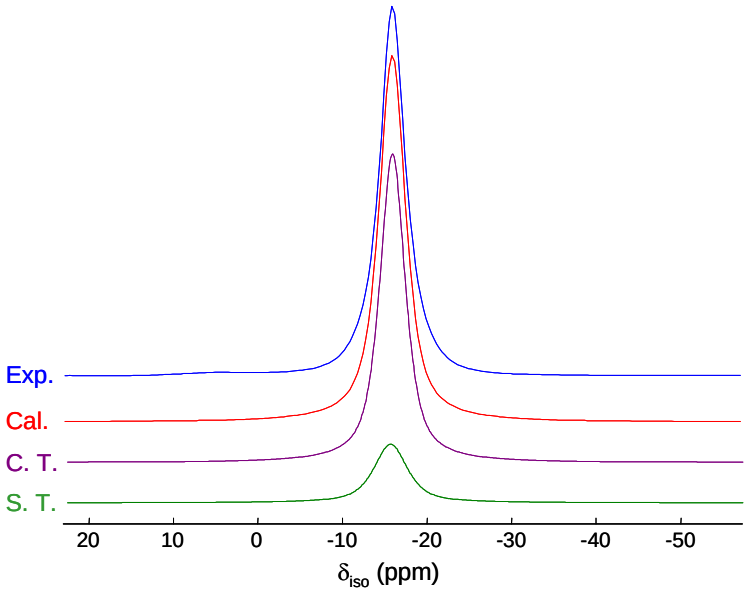

Figure 5. Experimental and calculated central transitions of the ^{27}Al MAS (30 kHz) NMR spectrum recorded at 17.6 T of $Al_{0.82}\square_{0.18}F_{2.46}(H_2O)_{0.54}$ after annealing at 773 K during 24 h under Ar. The fitting takes into account the N = 0 band of both the satellite transitions (S. T.) <3/2> and the central transition (C. T.) <1/2> and leads to the following parameters: δ_{iso}=-15.9 (±0.2) ppm and $\nu_{Q\eta}$=140 (±20) kHz.