Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

On the preparation and NMR spectroscopic characterization of potassium aluminium tetrahydride $KAlH_4$

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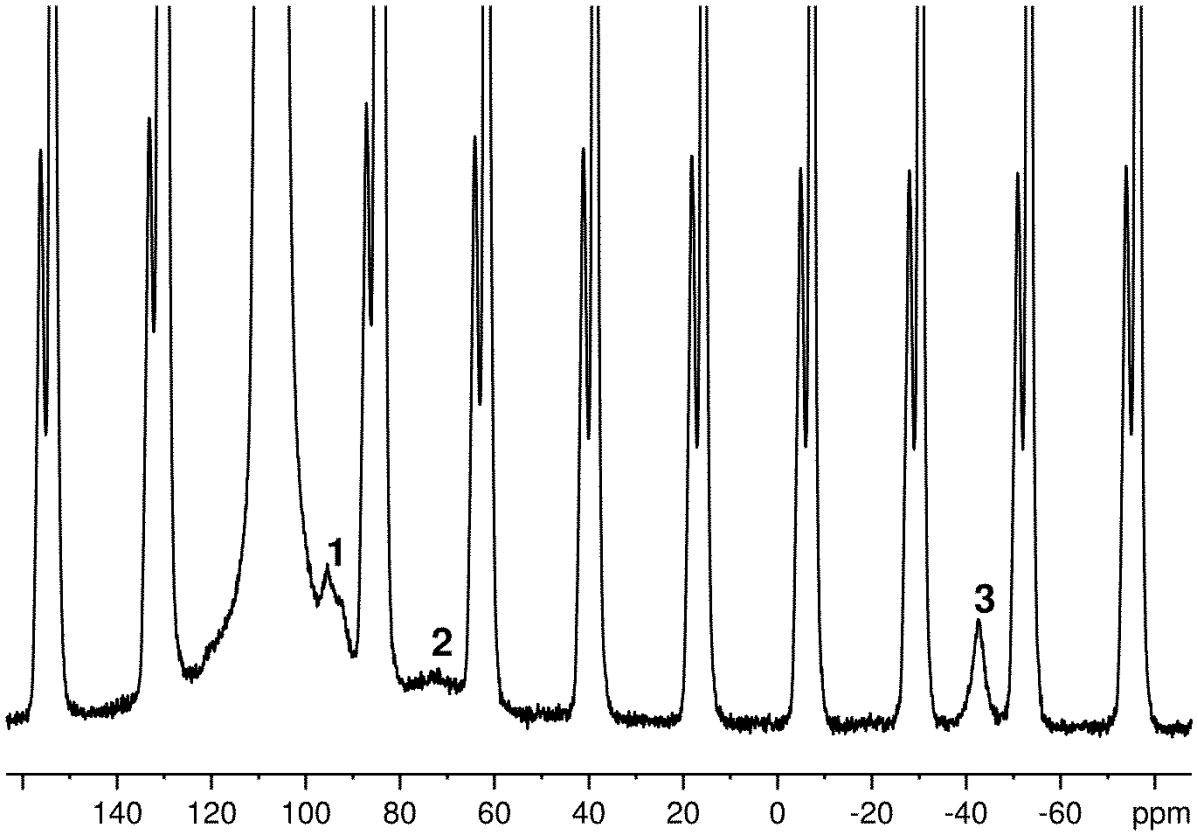

Fig. S1 ^{27}Al MAS NMR spectrum of $KAlH_4$ measured at ν_{MAS} = 3 kHz as in Fig. 2, but here enlarged to show the resonance lines due to impurities present: (1) $NaAlH_4$, (2) aluminium oxide, and (3) Na_3AlH_6. To estimate the amount of by-products one can use the intensity of the visible spinning sidebands of the $KAlH_4$ outer satellite transition that amounts to about 6‰ of the intensity of the centreband of the central transition. Hence, all aluminium containing by-products are traces in the per mille range.

Table S1 Parameters used to simulate the ^{27}Al MAS NMR sideband patterns shown in Fig. 4, Fig. S2 and Fig. S3 by means of the solids lineshape analysis module implemented in the TopSpin™ 3.2 NMR software package

Parameter	Value
TRIANG	64
Model	QUAD all
MASR	1400 / 3000
Angle	54.7
Side bands	278 /131
δ(iso) [ppm]	107.6
CQ(Quad) [kHz]	1290
η(Quad)	0.64
LB	60
GB	120

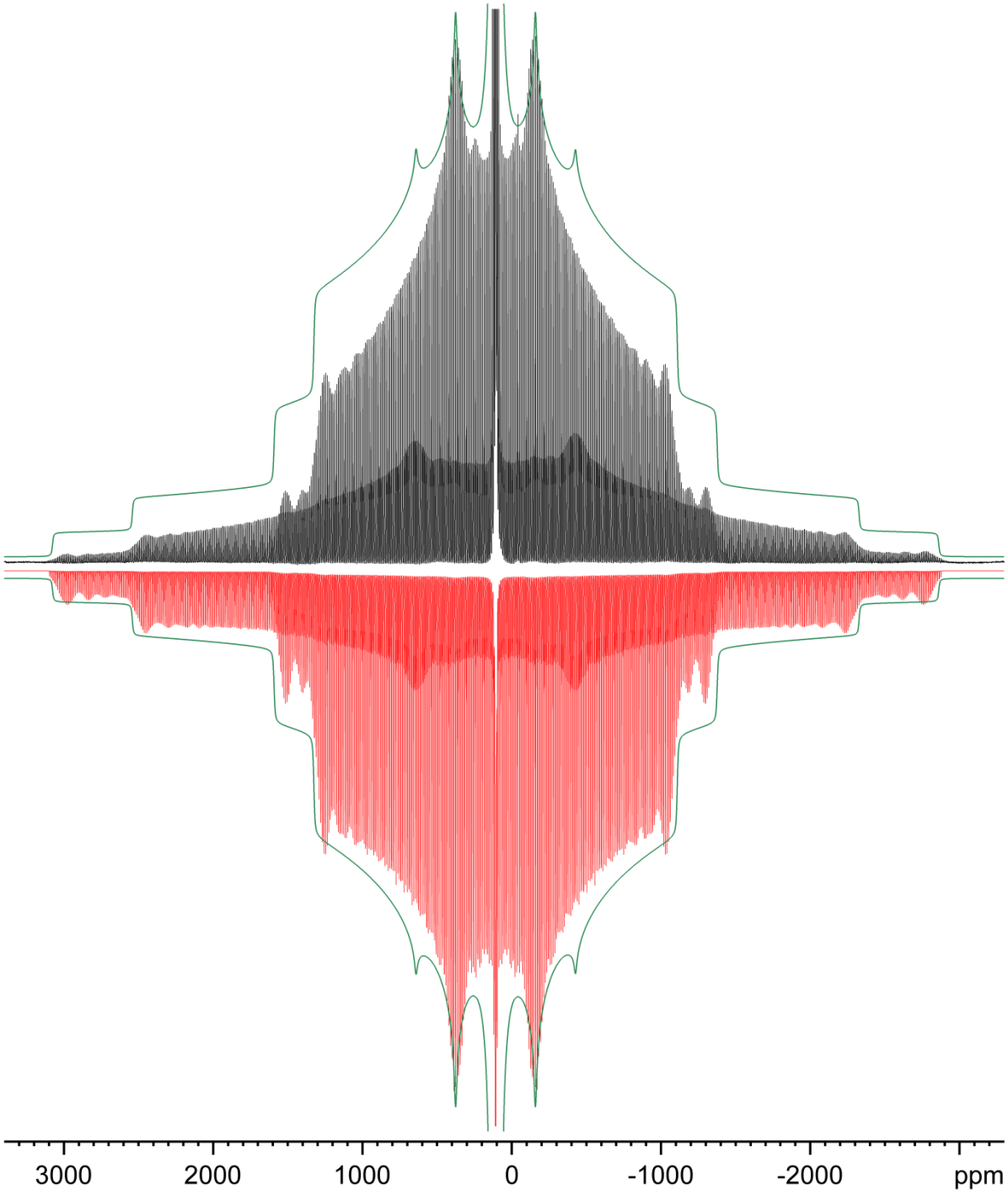

Fig. S2 Experimental ^{27}Al MAS NMR spectrum of $KAlH_4$ measured at ν_{MAS} = 1.4 kHz (black) and its simulation (red) with the parameters δ_{iso} = 107.6 ppm, C_Q = 1.29 MHz and η = 0.64. To ease the comparison, the simulated MAS NMR spectrum has been inverted. The green lines depict the lineshape one would obtain for a non-spinning sample.

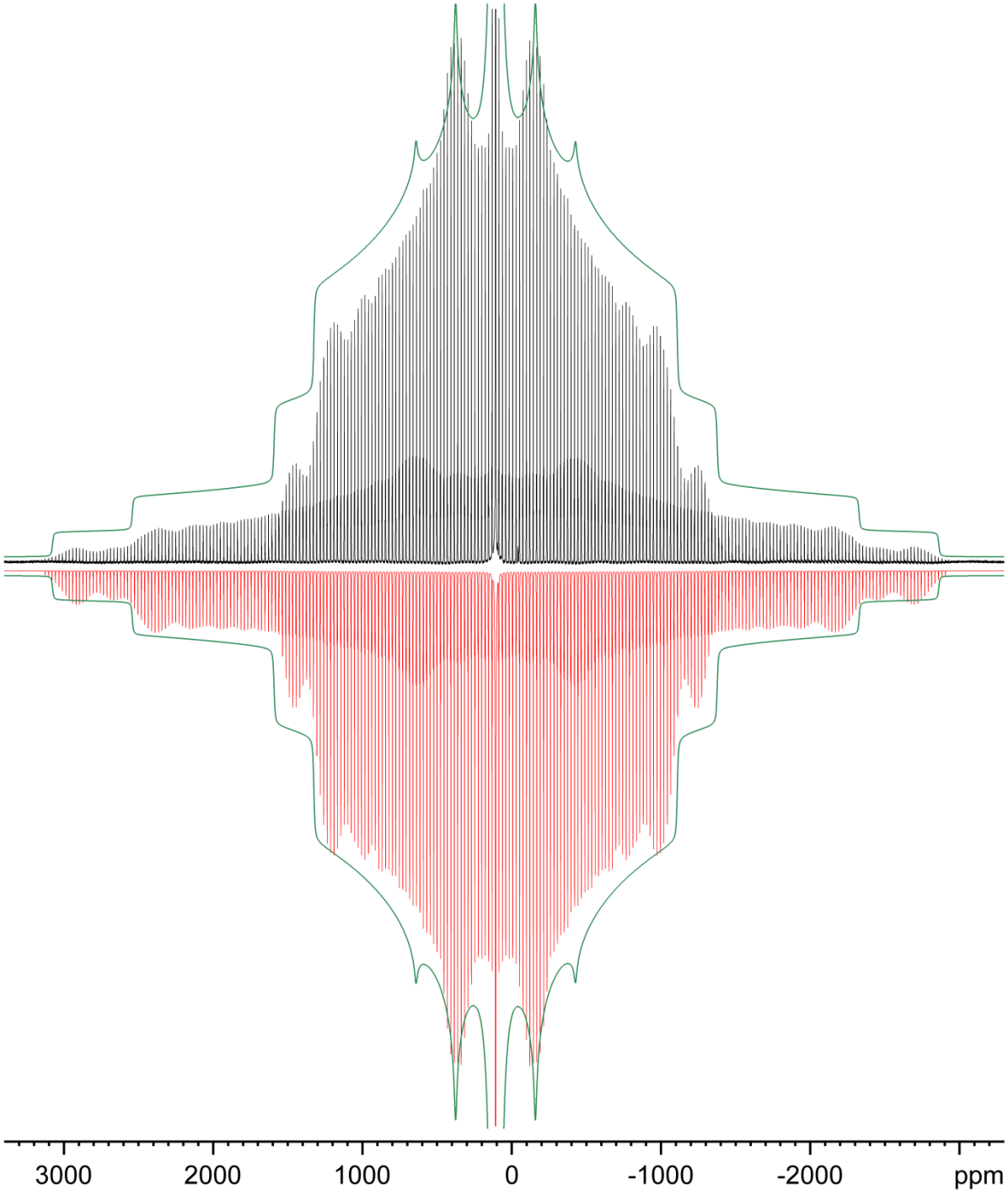

Fig. S3 Experimental ^{27}Al MAS NMR spectrum of $KAlH_4$ measured at ν_{MAS} = 3.0 kHz (black) and its simulation (red) with the parameters δ_{iso} = 107.6 ppm, C_Q = 1.29 MHz and η = 0.64. To ease the comparison, the simulated MAS NMR spectrum has been inverted. Again, the green lines depict the lineshape one would obtain for a non-spinning sample.

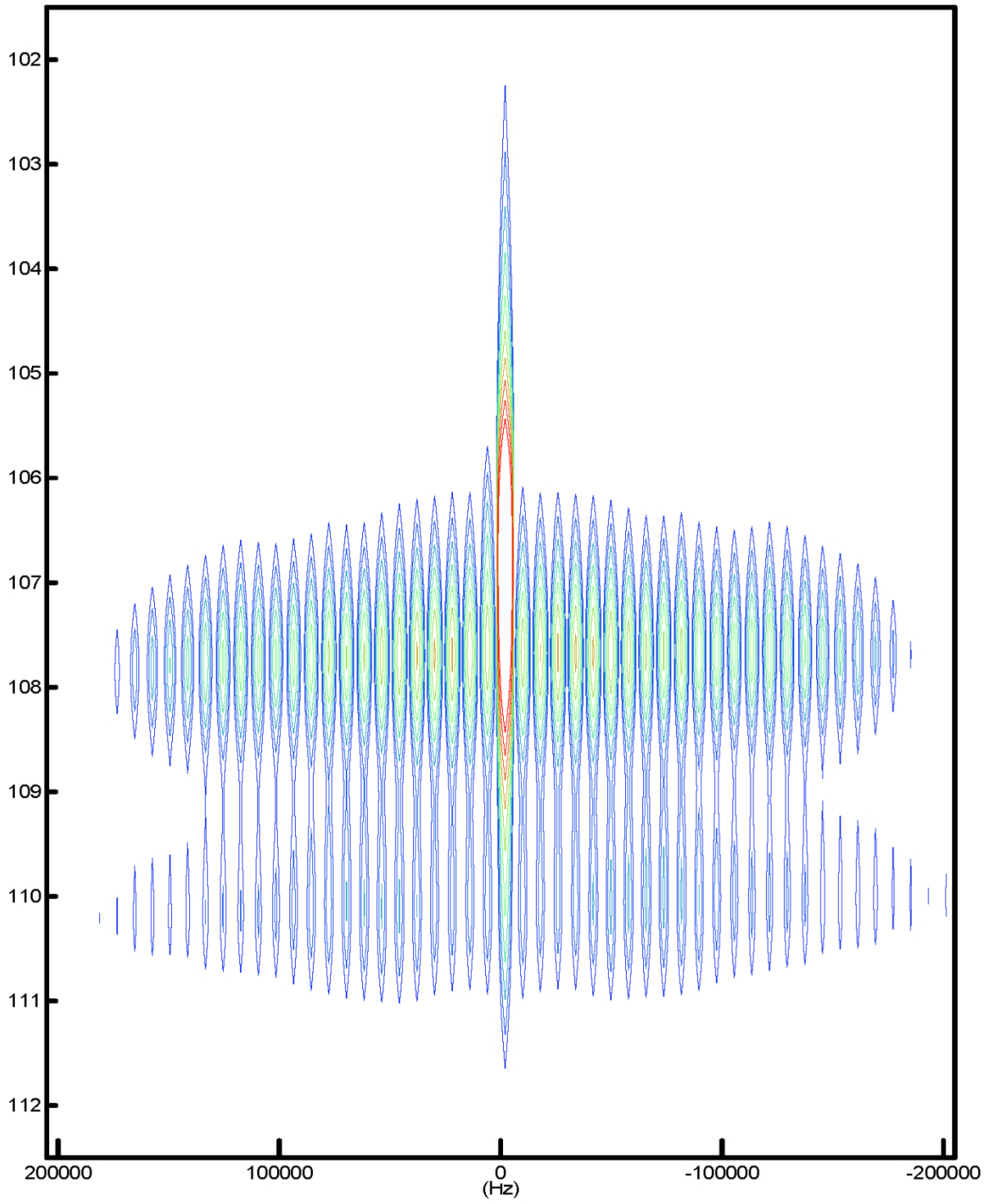

Fig. S4 ^{27}Al TOP NMR spectrum of $KAlH_4$. This spectrum is just a different presentation of the MAS NMR spectrum (ν_{MAS} = 8 kHz) given in Fig. 3c. Following the suggestion of one of the referees, we have used the software package dmfit (D. Massiot et al., Magn. Reson. Chem. 40 (2002) 70-76; dmfit/x64/release#20190125; available from: nmr.cemhti.cnrs-orleans.fr) to process the data recorded. The positions of the resonance lines of the different transitions can directly be read out from this plot. These values are slightly lower than those given in the text as derived from the spectrum measured at ν_{MAS} = 3 kHz. The small shift is due to frictional heating at higher spinning frequencies as mentioned in the paper.

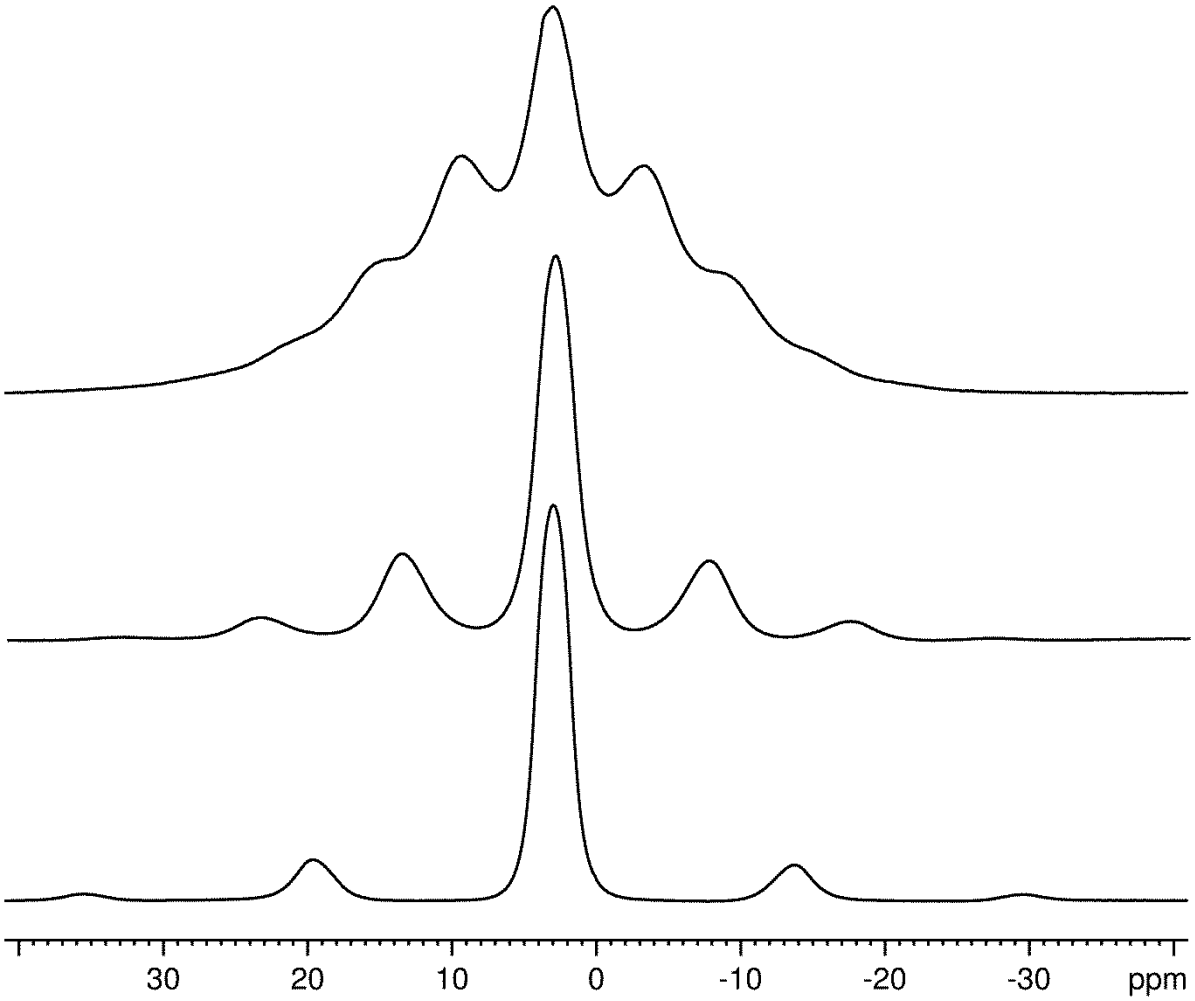

Fig. S5 ^{1}H MAS NMR spectra of $KAlH_4$ recorded at different spinning frequencies ν_{MAS}: 3 kHz (top), 5 kHz (middle), and 8 kHz (bottom). The experimental conditions are as follows: 500 MHz resonance frequency, 10 s repetition delay, and 3.0 µs π/4 pulses. At ν_{MAS} = 8 kHz, the full width at half height of the line at 2.95 ppm amounts to about 1.3 kHz. The spectra give no indication of the presence of by-products.