Electronic Supplementary Information for Dalton Transactions

Supplemental information

for

Preparation, Structure and Analysis of the Bonding in the Molecular Entity $(OSO)_2Li\{[AlF(OR_F)_3]Li[Al(OR_F)_4]\}$ (R_F = $C(CF_3)_3$).

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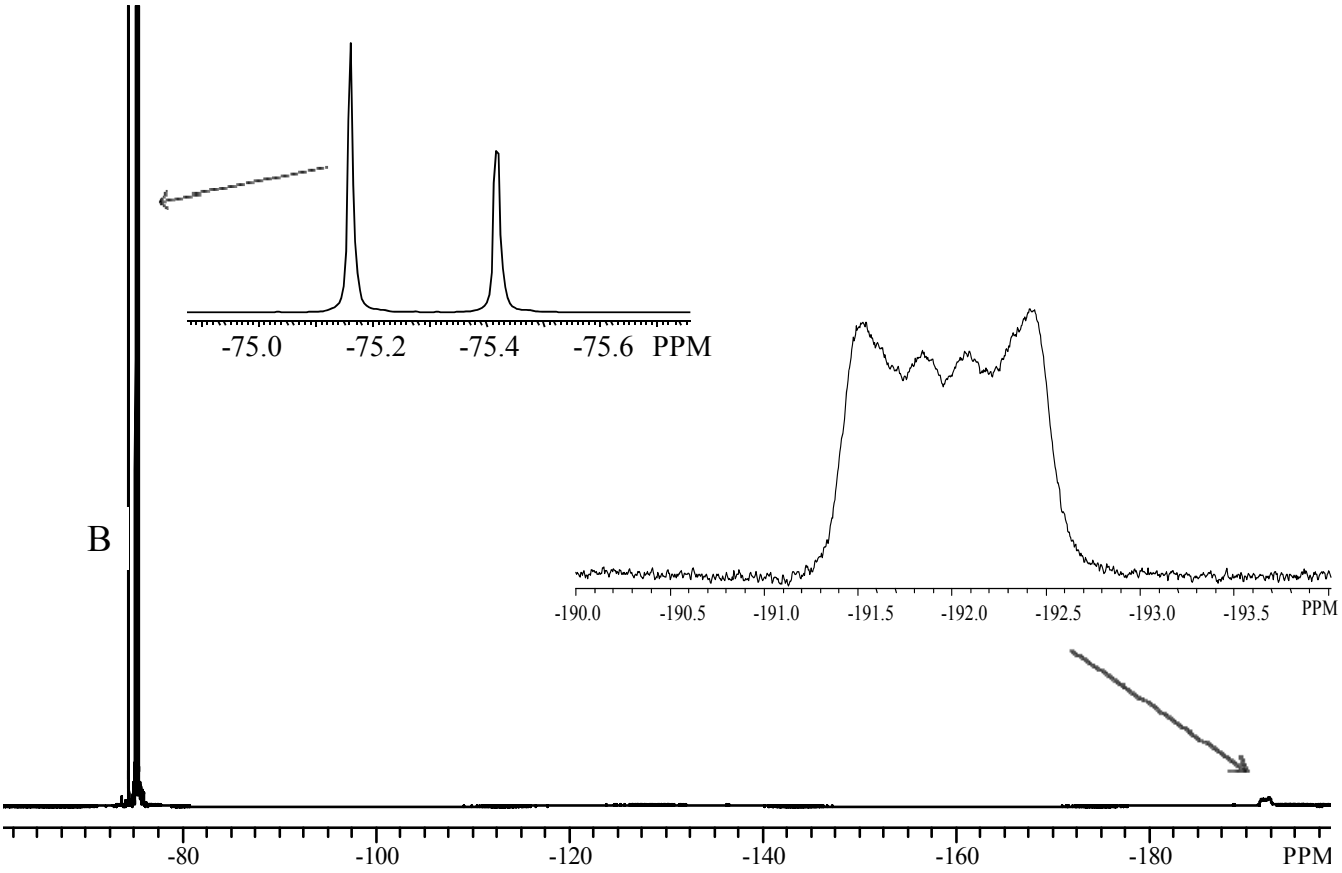

Figure S1. ^{19}F NMR spectrum of **1** in SO_2 at room temperature, **B** = minor impurity of $Li[Al(OC(CF_3)_3)_4]$. Relative intensities of three resonances of A = 36 : 28 : 1. Relative molar intensities of A : B = 100 : 16. Fine structure of resonance at -192 ppm has four components, separation between them are is 122 Hz, 89 Hz, 122 Hz (from left to right).

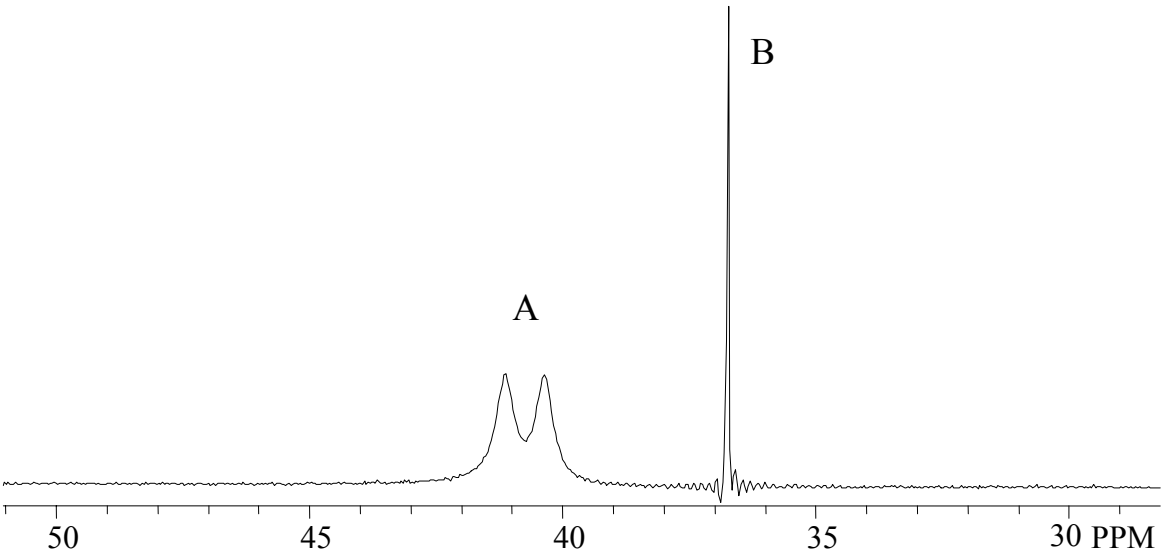

Figure S2. ^{27}Al NMR spectrum of 1 in SO_2 at room temperature, A = 1, B = impurity of $Li[Al(OC(CF_3)_3)_4]$. Relative intensities A : B = 100 : 19. Separation between two resonances of A is 80.0 Hz. The linewidth for two resonances of 1 is $\Delta\omega_{1/2}$ = 51 Hz and 50 Hz. Linewidth of B is $\Delta\omega_{1/2}$ = 10 Hz.

Notes and interpretation. The Al-F spin-spin coupling constant is repoted only for one aluminum fluoride complex $[AlF_4]^-$.[1] ^{19}F NMR resonance of $[AlF_4]^-$ is located at -194.2 ppm, $J_{Al\text{-}F}$ = 37.8 Hz; ^{27}Al NMR resonance at 49.2 ppm, $J_{Al\text{-}F}$= 37.8 Hz. The spin-spin coupling constants are equal in ^{19}F and ^{27}Al NMR spectra. Symmetry of environment of both aluminums in **1** is lower than tetrahedral, however separation of components of multiplet (-192.0 ppm) in ^{19}F NMR spectrum and separation between two resonances of **A** in ^{27}Al NMR spectra are not comparable. Therefore ^{27}Al NMR resonances at 40.4 and 41.3 ppm are due to two magnetically non equivalent aluminums in **1** and not due to coupling Al-F. Fine structure of ^{19}F NMR resonance may be due to spin-spin coupling of fluorine nuclei with the ^{27}Al nuclei and ^{6}Li and ^{7}Li nucleas.

[1] N. Herron, D. L. Thorn, R. L. Harlow, F. Davidson, *J. Am. Chem. Soc*. **1993**, *115*. 3028-3029.

Figure S3. Sublimation vessel (Ace Glass Inc.) used for subliming $Li[Al(OR_F)_4]$.