Supplemental information

for

Preparation, Structure and Analysis of the Bonding in the Molecular Entity (OSO)$_2$Li{[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).
Notes and interpretation. The Al-F spin-spin coupling constant is reported only for one aluminum fluoride complex [AlF₄].¹ ¹⁹F NMR resonance of [AlF₄] is located at -194.2 ppm, $J_{Al-F} = 37.8$ Hz; ²⁷Al NMR resonance at 49.2 ppm, $J_{Al-F} = 37.8$ Hz. The spin-spin coupling constants are equal in ¹⁹F and ²⁷Al NMR spectra. Symmetry of environment of both aluminums in ¹ is lower than tetrahedral, however separation of components of multiplet (-192.0 ppm) in ¹⁹F NMR spectrum and separation between two resonances of A in ²⁷Al NMR spectra are not comparable. Therefore ²⁷Al 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 ¹⁹F NMR resonance may be due to spin-spin coupling of fluorine nuclei with the ²⁷Al nuclei and ⁶Li and ⁷Li nucleas.

Figure S3. Sublimation vessel (Ace Glass Inc.) used for subliming Li[Al(OR\textsubscript{F})\textsubscript{4}].