**Supporting Information for**

Lewis Acid fragmentation of a Lithium Aryloxide Cage: Generation of New Heterometallic Aluminium-Lithium species

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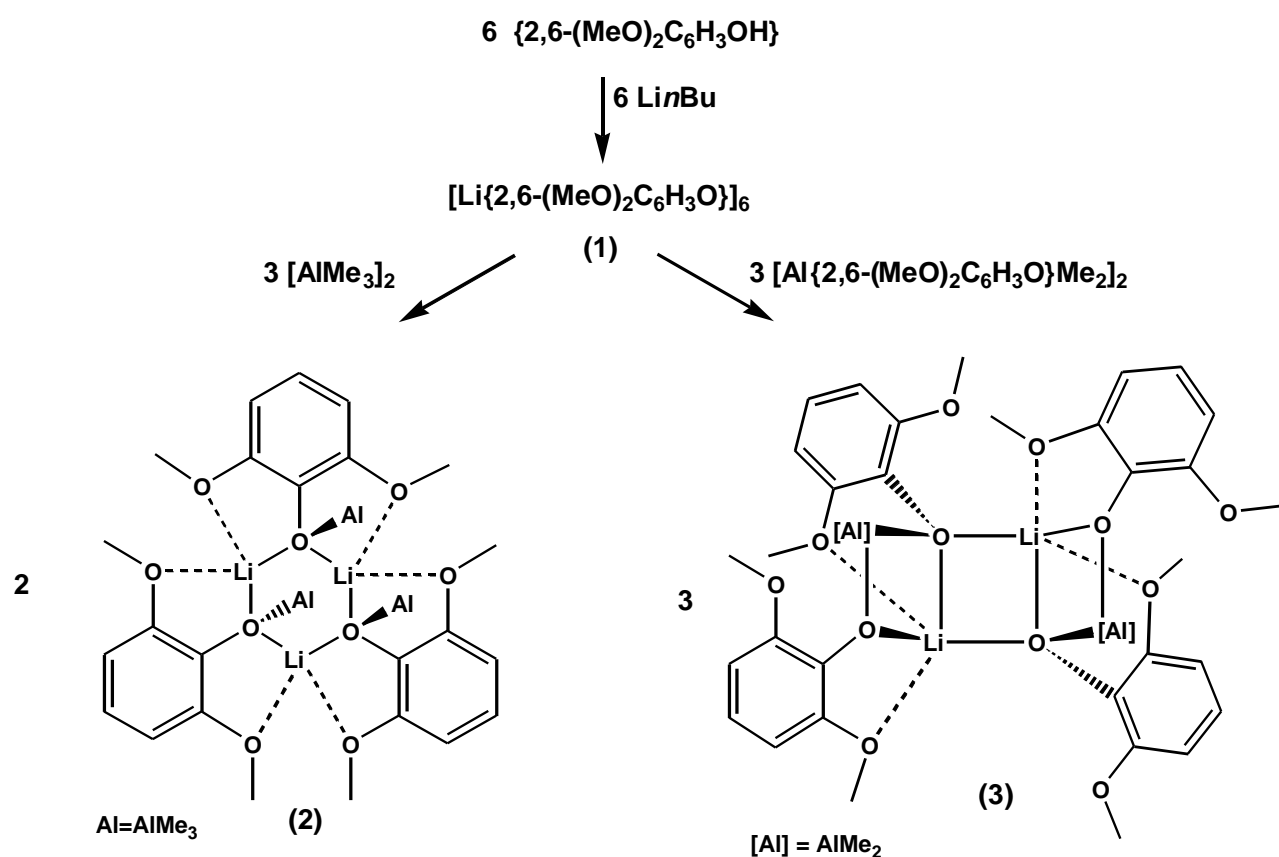
Scheme 1. Synthesis of compounds **1**, **2** and **3**

Experimental details and physical data

General Considerations. All manipulations were conducted using Schlenk techniques in conjunction to an inert atmosphere glove box. All solvents were rigorously dried prior to use. NMR spectra were recorded at 400.13 (^{1}H), 376.44 (^{19}F), 155.50 (^{7}Li) and 100.62 (^{13}C) MHz on a Bruker AV400. Chemical shifts (δ) are given in ppm using tol-d$_8$ or C$_6$D$_6$ as solvent. ^{1}H and ^{13}C resonances were measured relative to solvent peaks considering TMS δ = 0 ppm, while ^{19}F was measured relative to external CFCl$_3$ and Li to external LiCl in D$_2$O. Elemental analyses were obtained on a Perkin-Elmer Series II 2400 CHNS/O analyzer. All reagents were commercially obtained and used without further purification.

Synthesis of [Li{2,6-(MeO)$_2$C$_6$H$_3$O}]$_6$. (1). To a solution of 0.195 g (1.26 mmol) of 2,6-dimethoxyphenol in 20mL of toluene, 0.78 mL (1.26 mmol) of Li^{n}Bu 1.6 M was added, in a cold bath of dry ice/isopropanol, and the mixture was stirred for 10 minutes. Then the solution was allowed to reach room temperature. After stirring for an hour at room temperature, a white solid was formed, that was dissolved by adding 0.195 g (1.26 mmol) of 2,6-dimethoxyphenol in 20mL of toluene and heated to a brief reflux. After one day of storage at room temperature, white crystals of compounds **1** were observed. Yield: 85% (0.17 g, 1.07 mmol). ^{1}H NMR (C$_6$D$_6$): δ 3.16 (s, 6H, OCH_3), 6.48 (d, 2H, ^{3}J$_{HH}$ = 7 Hz, *m*-OAr-*H*), 6.56 (t, 1H, ^{3}J$_{HH}$ = 7 Hz, *p*-OAr-*H*). ^{13}C (C$_6$D$_6$) δ 54.91 (s, OCH_3), 104.85 (s, *p*-OAr-*H*), 105.16 (s, *m*-OAr-*H*). ^{7}Li-RMN (C$_6$D$_6$): δ 1.79 (s). Anal. Calcd. (%) for Li$_6$C$_{48}$H$_{54}$O$_{18}$ (159.9 g / mol) C, 60.04; H, 5.63. Found: C, 60.47; H, 5.75.
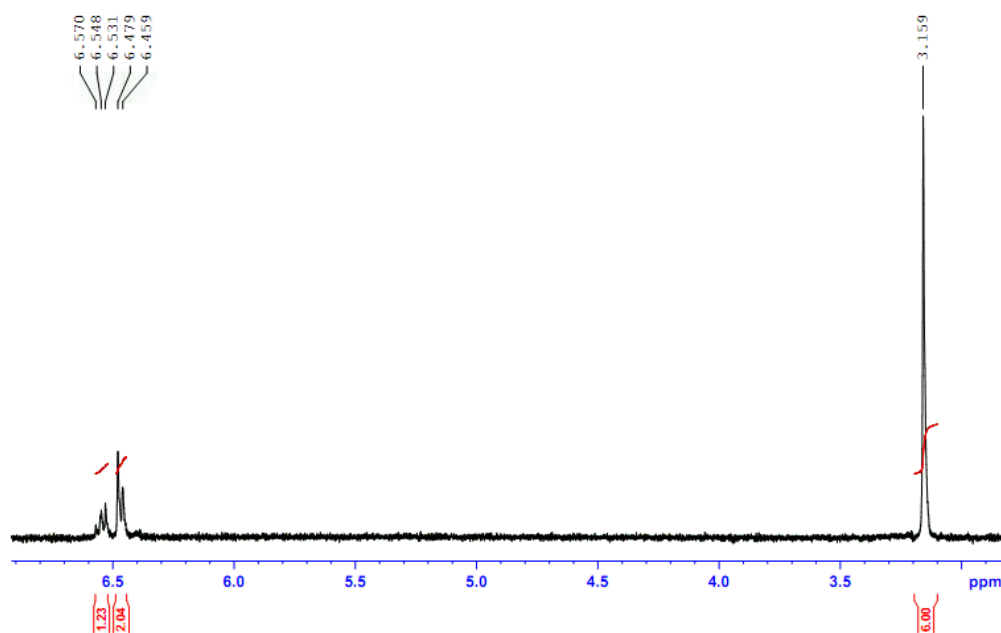
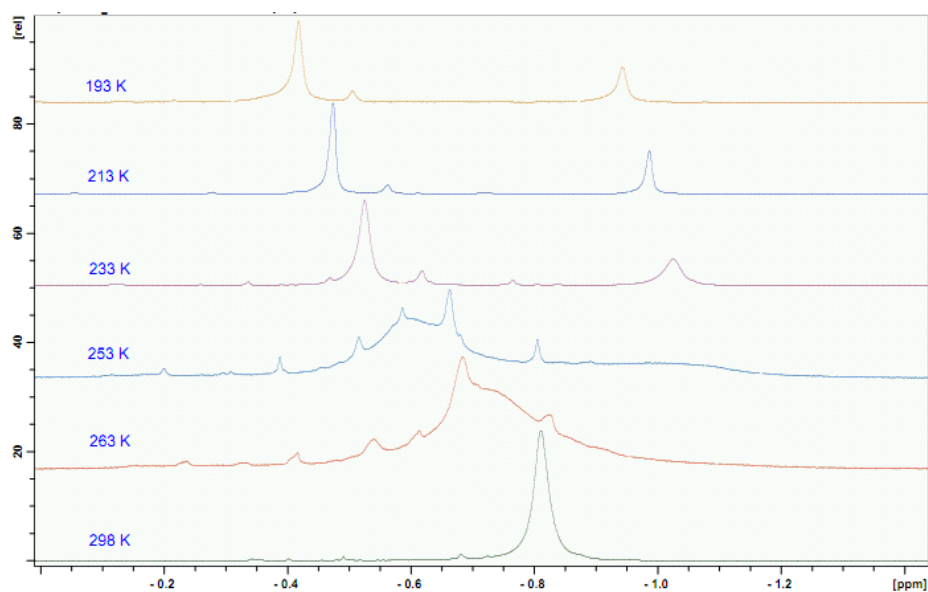**Figure 1.** ^{1}H NMR spectra of **1** in C$_6$D$_6$.

Synthesis of [LiAlMe$_3$\{2,6-(MeO)$_2$C$_6$H$_3$\}]$_3$. (2). To a solution of 0.5 g (3.24 mmol) of 2,6-dimethoxyphenol in 20mL of toluene, 2mL (3.24 mmol) of Li^{n}Bu was added, in a cold bath of dry ice/isopropanol, leaving it to react for 10 minutes. When the mixture of reaction reached room temperature, the solution was stirred for further 3 hours and the solvent was filtered off. The white solid obtained was washed with cold toluene three times. To this solid 20 mL of toluene were added and was placed again in a cold bath. To this suspension, 1.6 mL (3.24 mmol) of AlMe$_3$ 2M were added and stirred for 10 minutes. When the reaction mixture reaches room temperature the white solid formed was dissolved by heating. The solution was stored at -20 °C for two days, affording white crystals of compound **2**. Yield: 57 % (0.43 g, 0.62 mmol). ^{1}H NMR (Tol-d$_8$): δ -0.81 (s, 9H, AlCH_3), 3.42 (s, 6H, OCH_3), 6.26 (d, 2H, ^{3}J$_{HH}$ = 8 Hz, *m*-OAr-*H*), 6.64 (t, 1H, ^{3}J$_{HH}$ = 8 Hz, *p*-OAr-*H*). ^{1}H NMR (C$_6$D$_6$) δ -0.67 (s, 9H, AlCH_3), 3.39 (s, 6H, OCH_3), 6.28 (d, 2H, ^{3}J$_{HH}$ = 8 Hz, *m*-OAr-*H*), 6.69 (t, 1H, ^{3}J$_{HH}$ = 8 Hz, *p*-OAr-*H*). ^{13}C (C$_6$D$_6$) δ -8.06 (s, AlCH_3), 56.22 (s, OCH_3), 105.94 (s, *m*-OAr-*H*), 120.67 (s, *p*-OAr-*H*). ^{7}Li-RMN (C$_6$D$_6$): δ 0.45 (s). Anal. Calcd. (%) for Li$_3$Al$_3$C$_{27}$H$_{36}$O$_9$ (696.12 g/mol)) C, 60.49; H, 6.49. Found: C, 60.81; H, 6.99
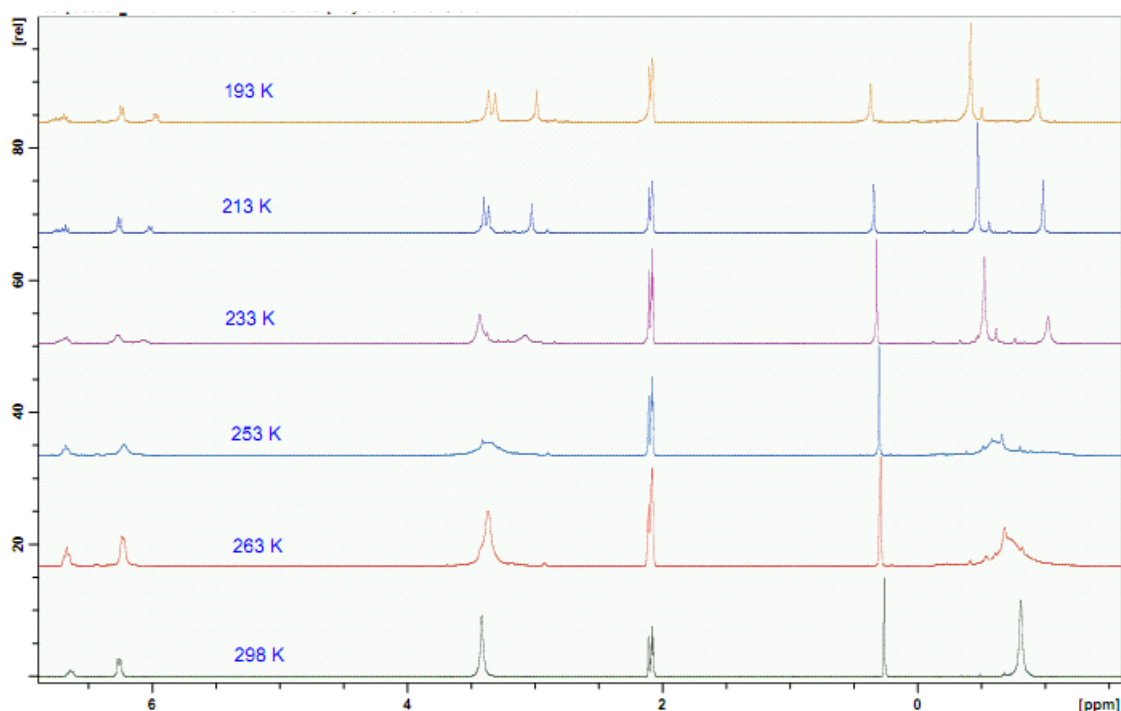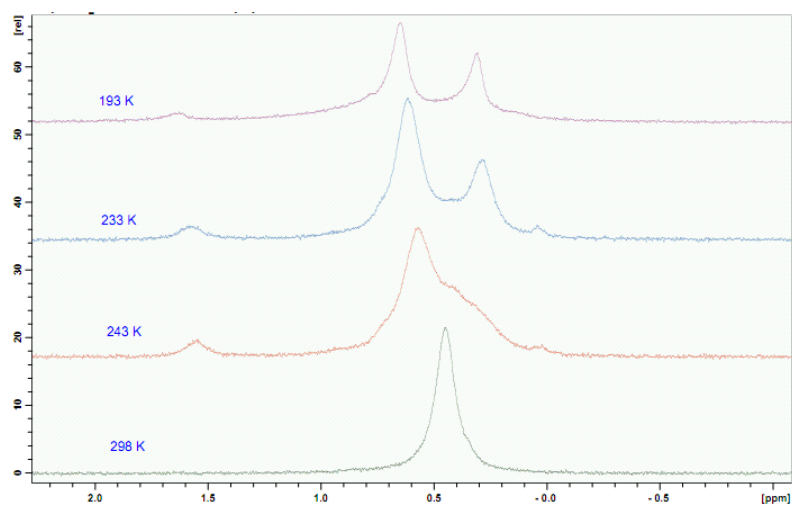

Figure 2. Variable temperature ^{1}H NMR spectra of **2** in tol-d$_8$.

Figure 3. Variable temperature ^{1}H NMR spectra of **2** in tol-d$_8$. Enlargement of the resonances corresponding to the methyl groups bonded to the aluminum atoms**.**

Table 1. ^{7}Li-NMR data for **2** in tol-d$_8$.

| T^a (K)	^{7}Li - δ (ppm)
298	0.45 (s)
243	0.57 (s wide)
233	0.62 (s), 0.29 (s)
193	0.65 (s), 0.31 (s)

Figure 4. Variable temperature ^{7}Li NMR spectra of **2** in tol-d$_8$.

Synthesis of [Al(2,6-(MeO)$_2$C$_6$H$_3$)Me$_2$]$_2$. A solution of 2,6-dimethoxyphenol (1 g, 6.42 mmol) in toluene (10 mL) was added drop wise to 3,26 mL of AlMe$_3$ 2 M (0.68 mmol) in 10 mL toluene at - 78ºC. The mixture was stirred for 15 min at low temperature and then allowed to reach room temperature. After stirring for an hour at room temperature the solid produced was dissolved by heating the mixture, the resulting colourless solution was stored at room temperature overnight, and some crystals of compound **3** were formed. Yield: 82.3 % (1.11 g, 2.64 mmol). ^{1}H NMR (C$_6$D$_6$): δ -

0.27 (s, 6H, AlCH_3), 3.38 (s, 6H, OCH_3), 6.24 (d, 2H, $^3J_{HH}$ = 8 Hz, OAr-H), 6.65 (t, 1H, $^3J_{HH}$ = 8 Hz, OAr-H). Anal. Calcd. (%) for $C_{20}H_{30}Al_2O_6$ (420.28 g/mol) : C, 57.7; H, 7.4. Found: C, 57.1; H, 7.1.
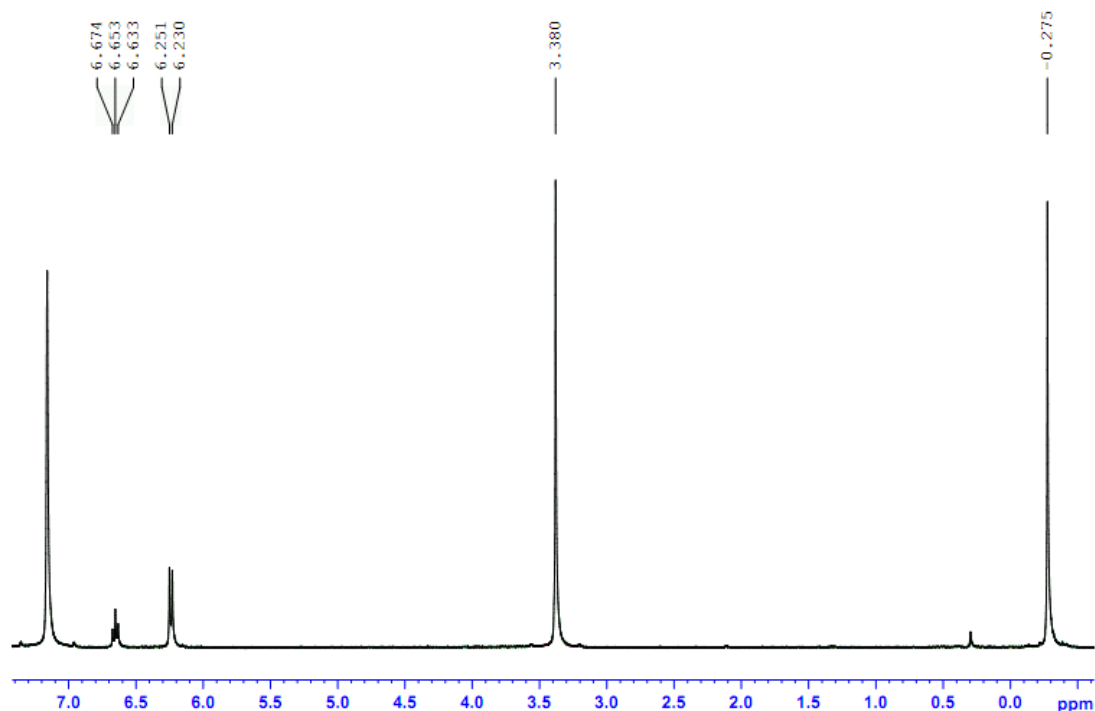
Figure 5. ^{1}H NMR spectra of [Al(2,6-(MeO)$_2$C$_6$H$_3$)Me$_2$]$_2$ in C$_6$D$_6$.

Synthesis of [LiAlMe$_2${2,6-(MeO)$_2$C$_6$H$_3$O}$_2$]$_2$ (3) To a solution of 0.11 g (0.72 mmol) of 1 in 20 mL of toluene, a solution of 3 (0.30 g, 0.72 mmol) in 20 mL of toluene was added at -78ºC. The mixture was stirred for 15 min at low temperature and then allowed to reach room temperature. After stirring for an hour at room temperature the precipitation of a white solid is observed. This solid was dissolved after a brief reflux. Storage of the solution at room temperature for a day allowed the formation of white crystals of compound **3**. Yield: 98 % (0.26 g, 0.35 mmol). ^{1}H NMR (Tol-d$_8$): δ -0.68 (s, 3H, AlCH_3), 3.35 (s, 6H, OCH_3), 6.23 (d, 2H, m-Ph, $^3J_{HH}$ = 2.4 Hz), 6.54 (t, 1H, p-Ph, $^3J_{HH}$ = 2.4 Hz). ^{1}H NMR (C$_6$D$_6$) δ -0.50 (s, 3H, AlCH_3), 3.36 (s, 6H, OCH_3), 6.31 (d, 2H, m-Ph, $^3J_{HH}$ = 8 Hz), 6.64 (t, 1H, p-Ph, $^3J_{HH}$ = 8 Hz). ^{13}C (C$_6$D$_6$) δ -8.06 (s, AlCH_3), 56.22 (s, OCH_3), 105.94 (s, m-OAr-H), 120.67 (s, p-OAr-H). ^{7}Li-RMN (Tol-d$_8$): δ 1.30 (s) Anal. Calcd. (%) for Li$_2$Al$_2$C$_{34}$H$_{42}$O$_{12}$ (740.4 g/mol) : C, 60.30; H, 6.23. Found: C, 61.03; H, 6.28.
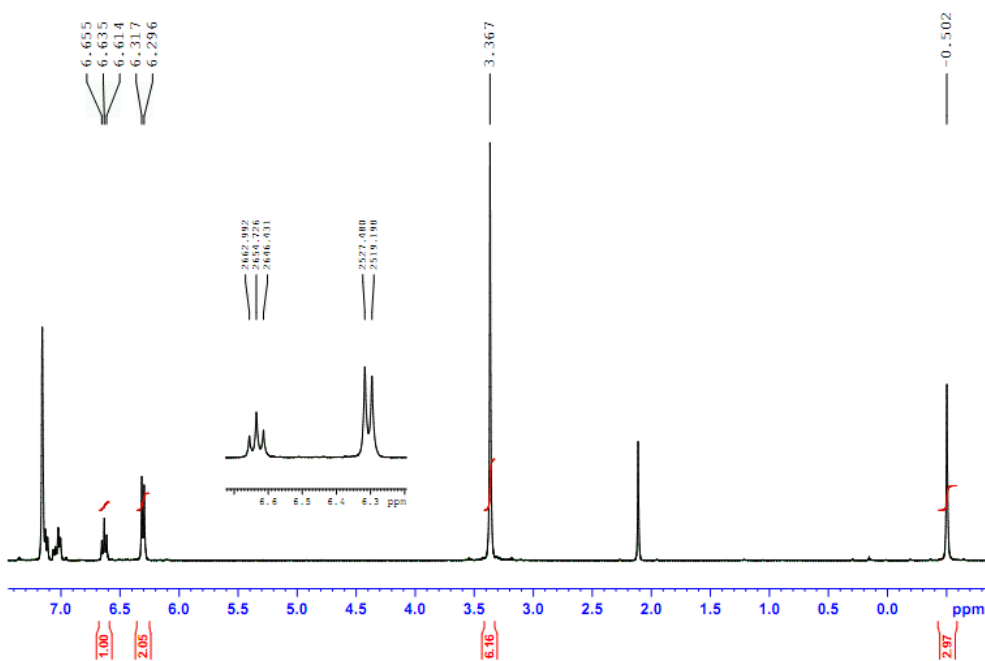
Figure 6. ^{1}H NMR spectra of **3** in C_6D_6.

Diffusion-Ordered NMR Spectroscopy (DOSY) experiments

In Diffusion-Ordered NMR Spectroscopy (DOSY) experiments, a series of pulsed field gradient (PFG) stimulated echo experiments is performed and the results allow to generate a two dimensional spectrum where signals are dispersed depending on their diffusion coefficients.[1] The signal intensity of a given resonance experiment decays following the Stejskal-Tanner equation[2] (1) as:

$$I_g = I_o \exp\left(-\gamma^2 g^2 \delta^2 (\Delta - \delta/3)\, D\right) \qquad (1)$$

where I_g and I_o are the signal intensities in the presence and absence of pulsed field gradients, respectively, γ is the gyromagnetic ratio (rad s g^{-1}), g is the strength of the diffusion gradients (gauss cm^{-1}), D is the diffusion coefficient of the observed spins (cm^2 s^{-1}), δ is the length of the diffusion gradients and Δ is the time separation between the leading edges of the two diffusion pulsed gradients. Diffusion coefficients are usually obtained by measuring the slope in the following linear relationship:

$$\ln (I_g/I_o) = -\gamma^2 g^2 \delta^2 (\Delta - \delta/3)\, D \qquad (2)$$

The diffusion coefficient (D) of a molecule is inversely proportional to its hydrodynamic radius as given in the Stokes-Einstein equation $D = kT/(6\pi\eta r_H)$, where k is the Boltzman constant, T is the temperature, η is the viscosity, and r_H is the hydrodynamic radius, however this is only strictly

valid for spherical molecules of a much bigger size than the solvent. From the Stokes-Einstein equation the relation between the diffusion coefficient of a molecule, D, and its volume, V, can be easily linearised by taking the logarithm of both sides $\log D = A \cdot \log V + B$.

The DOSY experiments are very sensitive to experimental conditions and temperature fluctuation, convention and viscosity change produce variations on the diffusion coefficient value from one experiment to another for the same particle.[3,4] This problem can be overcome by using internal standards of known size, by measuring their diffusion coefficients in the same experiment as the compound we are interested in, it will be possible to generate a calibration curve giving information on the size of the compound in solution.[4,5] Hence, if the volume of the compound estimated from the solid state structure fits the calibration curve, it is possible to consider that the nuclearity observed in the solid state is maintained in solution. Following this methodology several groups have attained remarkable achievements to reveal the nuclearity and aggregation in solution of various organometallic and coordination derivatives.[6]

[1]H DOSY of 1, 2 and 3 in the presence of internal references:

In our case, the internal standards chosen are the compounds: 1,2,3,4-tetraphenylnaphthlene-TPhN (432.55 g mol^{-1} 358.50 cm^3 mol^{-1}); 1-phenylnaphthalene-PhN (204.27 g mol^{-1}; 163.50 cm^3 mol^{-1}) and tetramethylsilane-TMS (82.22 g mol^{-1}; 101.50 cm^3 mol^{-1}). They satisfy the requirements needed for the method: (a) they are inert towards our compounds; (b) their chemical shifts do not overlap, (c) they have little coordinating ability to the complexes in solution; (d) they have good solubility in C_6D_6 and (e) they possess a wide molecular weight distribution in the range of the analyses of study.

All NMR experiments were carried out on a a Bruker AV400 spectrometer equipped with a probe PABBO BB-1H/D Z-GRD. 2D DOSY experiments were recorded under routine conditions at 298K with the bipolar-gradient LED (BPLED)[7] pulse sequence using a diffusion time of 100 ms and a LED delay of 5 ms. For each experiment, sine-shaped PFGs, with a duration of 1.5 ms followed by a recovery delay of 100 μs were incremented from 2% to 95% of the maximum strength in 16 equally spaced steps.

Figures 4-6 show the DOSY experiment of the mixture of compounds **1**-**3** and the reference compounds that allow the determination of the translational self-diffusion coefficients (D) for all species in solution. The plots of log D *vs* log V (V is estimated molecular volume, see below computational details) are shown in figures 7-10. We have included as well the plots against FW, considering the approximation that the FW is proportional to the volume and hence $\log D = A' \cdot \log FW + B'$, the adjustments are quite similar to the ones observed for the volume ones.

The relation between log D and log V for the internal standards used and compounds **1** and **3** proved to be approximately linear (Figures 7 and 10) and the trend-line showed a very good fit (R^2 = 0.9968 for 1 and R^2 = 0.9959 for **3**) indicating that these compounds maintain their nuclearity in solution. However for **2**, the adjustment is poorer (R^2 = 0.9466), getting a better fit if the molecule [AlLiMe$_3${2,6-(MeO)$_2$C$_6$H$_3$O}]$_2$] (**2'**) is considered, (R^2 = 0.9927) indicating that the nuclearity in solution of **2** is lower than the one observed in solid state.
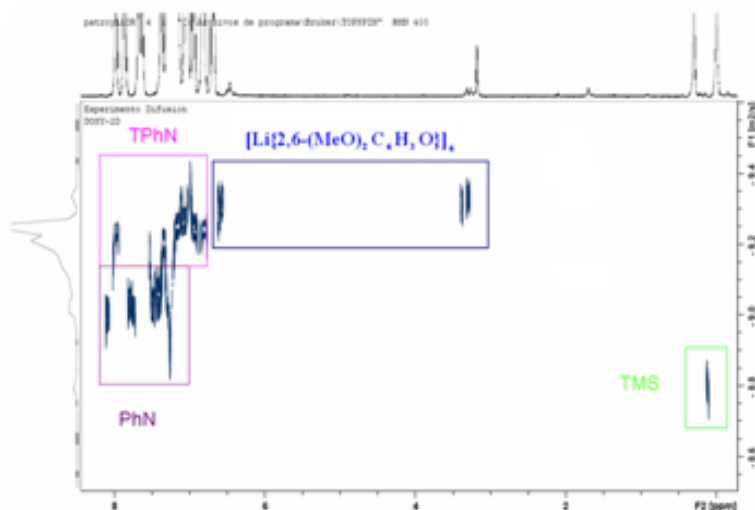
Figure 7. 2D DOSY NMR experiment of the mixture of TMS, PhN, TPhN and **1** in C$_6$D$_6$. The x- and y-axis represent the regular ^{1}H chemical shift and the relative diffusion rate, respectively

Figure 8. 2D DOSY NMR experiment of the mixture of TMS, PhN, TPhN and **2** in C$_6$D$_6$. The x- and y-axis represent the regular ^{1}H chemical shift and the relative diffusion rate, respectively.
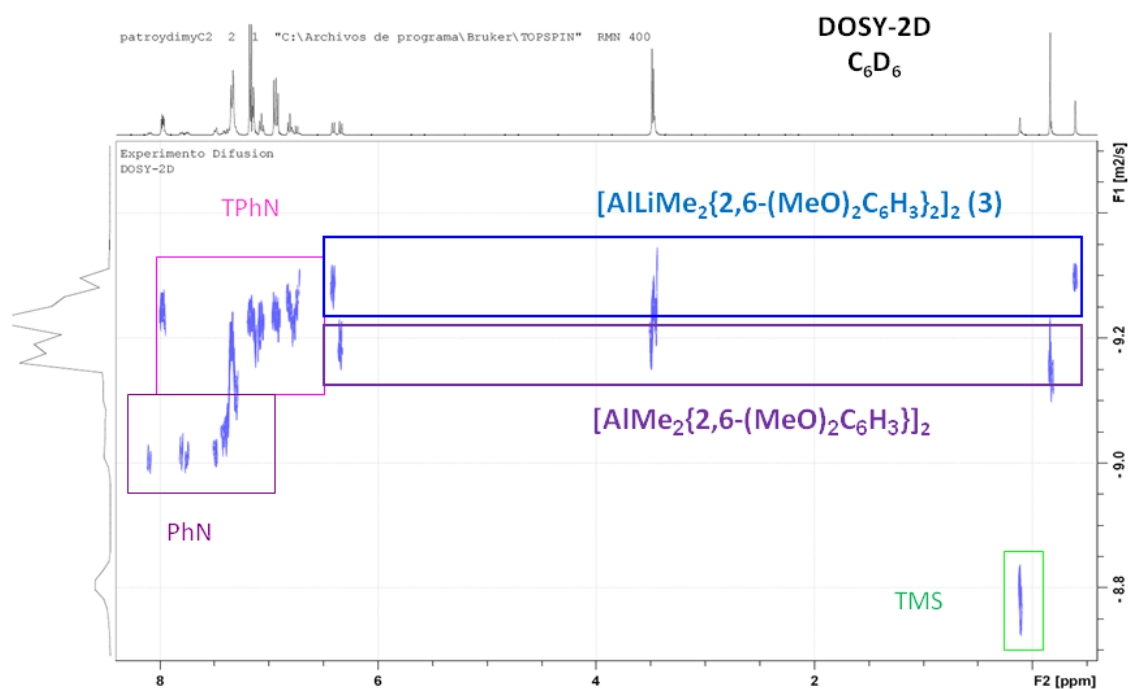
Figure 9. 2D DOSY NMR experiment of the mixture of TMS, PhN, TPhN, [AlMe$_2${2,6-(MeO)$_2$C$_6$H$_3$O}] and **3** in C$_6$D$_6$. The x- and y-axis represent the regular ^{1}H chemical shift and the relative diffusion rate, respectively.

a)   b)
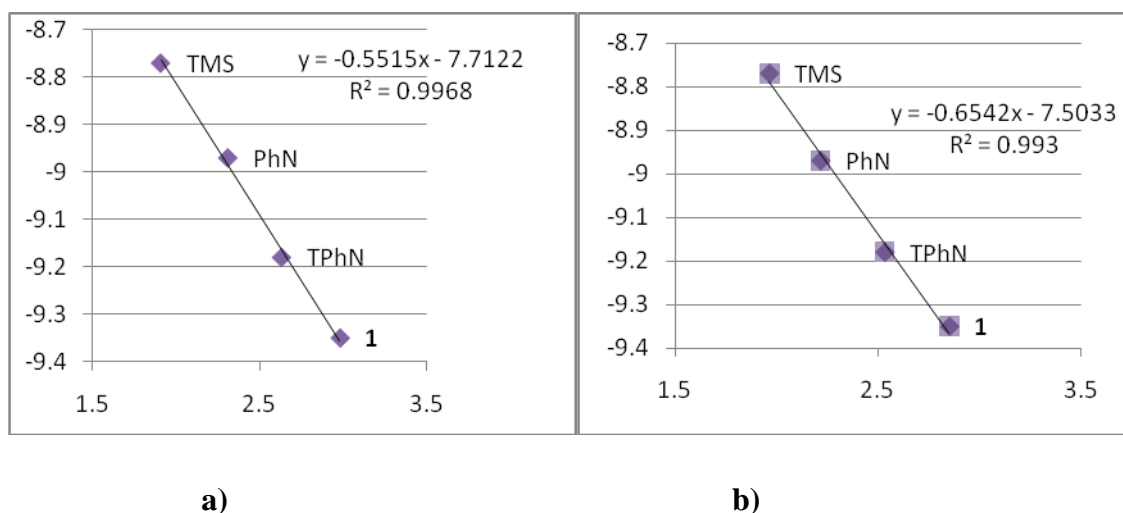
Figure 10. a) Correlation between lod D and log V from the ^{1}H DOSY data for the mixture of **1**, TPhN, PhN and TMS. b) Correlation between lod D and log FW from the ^{1}H DOSY data for the mixture of **1**, TPhN, PhN and TMS.
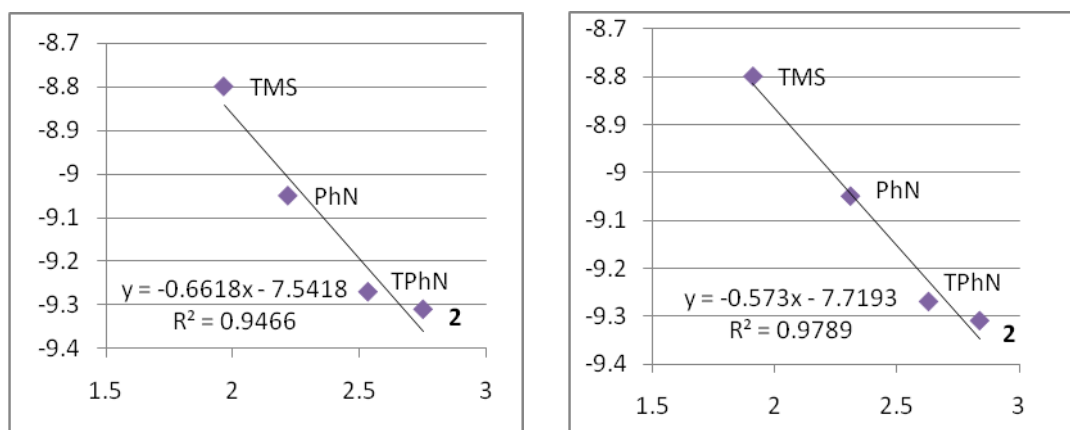
a)   b)

Figure 11.) Correlation between lod D and log V from the ^{1}H DOSY data for the mixture of **2**, TPhN, PhN and TMS. b) Correlation between lod D and log FW from the ^{1}H DOSY data for the mixture of **2**, TPhN, PhN and TMS.
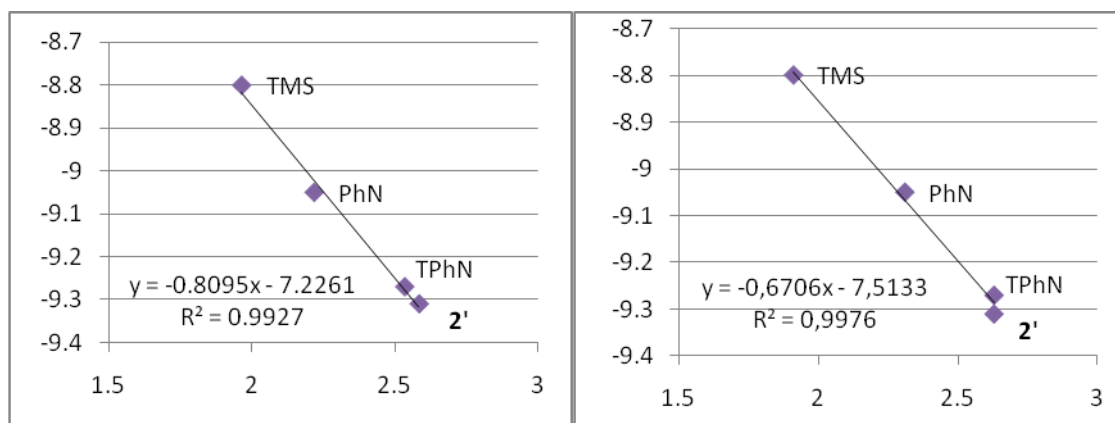
a)   b)

Figure 12.) Correlation between lod D and log V from the ^{1}H DOSY data for the mixture of $[AlLiMe_3\{2,6-(MeO)_2C_6H_3O\}]_2$ (**2'**), TPhN, PhN and TMS. b) Correlation between lod D and log FW from the ^{1}H DOSY data for the mixture of **2'**, TPhN, PhN and TMS.

a)   b)
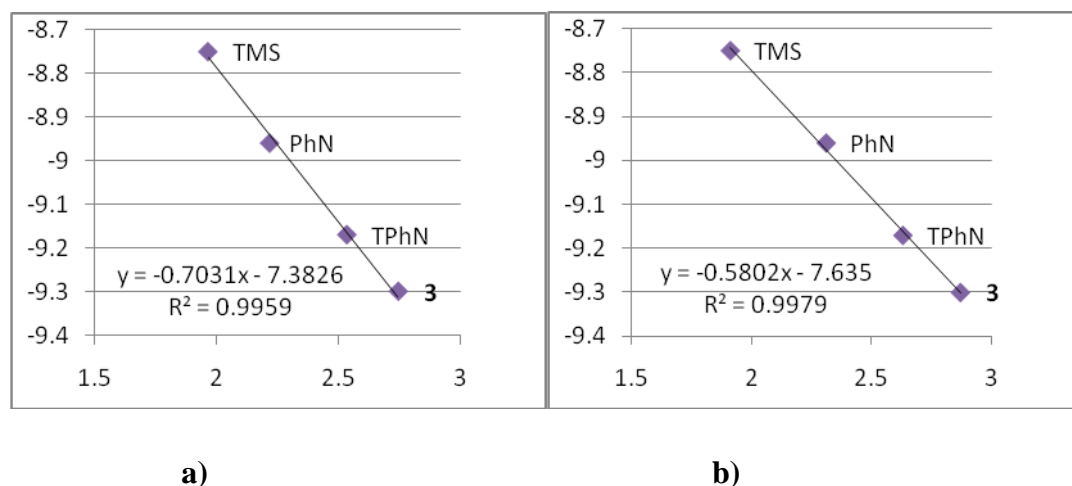
Figure 13. a) Correlation between lod D and log V from the ^{1}H DOSY data for the mixture of **3**, TPhN, PhN and TMS. b) Correlation between lod D and log FW from the ^{1}H DOSY data for the mixture of **3**, TPhN, PhN and TMS.

Computational Details.

Electronic structure calculations were carried out using the B3LYP[8] hybrid density functional with the 6-311G(d,p) basis set as implemented in the Gaussian 09 suite of programs.[9] Minima optimizations were performed by computing analytical energy gradients. The obtained stationary points were characterized by performing energy second derivatives, confirming them as minima by the absence of negative eigenvalues of the hessian matrix of the energy. Computed electronic energies were corrected for zero-point energy, and thermal energy to obtain the corresponding H^0 values. Estimated molecular volumes based on the electronic cloud of the molecule using numerical Monte Carlo integration (keyword volume in Gaussian[9]) were calculated according to reference 10. The volume calculation was repeated 10 times for each compound to reduce random errors.

Single-Crystal X-ray Structure Determination of Compounds 1, 2.C$_7$H$_8$, and 3.C$_7$H$_8$.

Details of the X-ray experiment, data reduction, and final structure refinement calculations are summarized in Table 2. Suitable single crystals of **1, 2.C$_7$H$_8$**, and **3.C$_7$H$_8$** for the X-ray diffraction study were selected. Data collection was performed at 200(2) K, with the crystals covered with perfluorinatedether oil. The crystals were mounted on a Bruker-Nonius Kappa CCD single crystal diffractometer equipped with a graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). Multiscan[11] absorption correction procedures were applied to the data. The structures were solved, using the WINGX package,[12] by direct methods (SHELXS-97) and refined using full-matrix least-squares against F^2 (SHELXL-97).[13] All non-hydrogen atoms were anisotropically refined. Hydrogen atoms were geometrically placed and left riding on their parent atoms except for the hydrogen atoms from the disordered toluene molecule that cocrystallize with **3**. As well, a disordered toluene molecule per molecule of **2** is present in the unit cell, this solvent molecule was found in the difference Fourier map but was very disordered and it was not possible to get a chemically sensible model for it, so the Squeeze procedure[14] was used to remove its contribution to the structure factors. Full-matrix least-squares refinements were carried out by minimizing $\Sigma w(F_o^2-F_c^2)^2$ with the SHELXL-97 weighting scheme and stopped at shift/err < 0.001. The final residual electron density maps showed no remarkable features.

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-835937 [**1**], CCDC-835938 [**2.C$_7$H$_8$**] and CCDC-835939 [**3.C$_7$H$_8$**]. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

Table 3. Crystallographic Data for 1-3.

	1	2.C_7H_8	3·C_7H_8
Formula	$C_{48}H_{54}Li_6O_{18}$	$C_{33}H_{54}Al_3Li_3O_9$·C_7H_8	$C_{36}H_{48}Al_2Li_2O_{12}$·$C_7H_8$
FW	960.55	788.66	832.72
Color/habit	White/prism	White/prism	White/prism
Cryst dimensions (mm^3)	0.42 x 0.33 x 0.19	0.43 x 0.41 x 0.37	0.41 x 0.38 x 0.34
Cryst syst	Monoclinic	Monoclinic	Orthorhombic
Space group	$P2_1/a$	$P2_1/n$	$Pnca$
a, Å	12.0342(9)	9.8598(13)	13.6968(12)
b, Å	16.4822(27)	22.364(3)	13.9464(18)
c, Å	12.6028(16)	21.162(5)	24.2111(19)
β, (deg)	111.6689(13)	99.163(14)	90
V, Å^3	2323.1(5)	4606.6 (14)	4624.8 (8)
Z	2	4	4
T, K	200	200	200
ρ_{calcd}, g cm^{-3}	1.373	1.137	1.196
μ, mm^{-1}	0.102	0.129	0.120
F(000)	1008.0	1688	1768
θ range, deg	3.07 – 27.53	3.05 – 25.91	3.27 – 27.50
no. of rflns collected	52973	81139	68740
no. of indep rflns / R_{int}	5337/0.0758	8911/0.0662	5314/0.0860
no. of data/restraints/params	5337 / 0 / 325	8911/ 0 / 433	5314 / 0 / 281
$R1/wR2$ $(I>2\sigma(I))^a$	0.0555 / 0.1258	0.0670 / 0.1644	0.0559 / 0.1262
$R1/wR2$ (all data)^a	0.1007 / 0.1457	0.1024 / 0.1832	0.0957 / 0.1462
Extinction coefficient		0.014(3)	0.0005(4)
GOF (on F^2)^a	1.026	1.050	1.018
Largest diff peak / hole (e Å^{-3})	0.302 and -0.238	+0.322 / -0.308	0.324 / -0.266

^{a} $R1 = \Sigma(||F_o|-|F_c||)/\Sigma|F_o|$; $wR2 = \{\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; $GOF = \{\Sigma[w(F_o^2-F_c^2)^2]/(n-p)\}^{1/2}$

Table 3. Selected bond lengths [Å] and angles [deg] for **1, 2.C₇H₈ and 3.C₇H₈.**

1	2.C₇H₈	3·C₇H₈
Bond lengths [Å]	**Bond lengths [Å]**	**Bond lengths [Å]**
Li(1)-O(2) 1.908(4)	Al(1)-O(1) 1.899(2)	Al(1)-O(1) 1.7987(16)
Li(1)-O(1) 1.929(4)	Al(1)-C(4) 1.981(4)	Al(1)-O(2) 1.8519(15)
Li(1)-O(3)#1 1.982(4)	Al(1)-C(5) 1.978(3)	Al(1)-C(1) 1.950(2)
Li(1)-O(4) 2.050(4)	Al(1)-C(6) 1.985(3)	Al(1)-C(2) 1.960(3)
Li(2)-O(2) 1.946(4)	Al(2)-O(2) 1.888(2)	Al(1)-Li(1) 2.974(4)
Li(2)-O(3) 1.951(4)	Al(2)-C(1) 1.971(3)	Li(1)-O(1) 1.917(4)
Li(2)-O(1)#1 2.058(4)	Al(2)-C(3) 1.977(3)	Li(1)-O(2)# 21.999(4)
Li(2)-O(7) 2.228(4)	Al(2)-C(2) 1.988(3)	Li(1)-O(6) 2.032(4)
Li(2)-O(8) 2.260(4)	Al(3)-O(3) 1.9208(19)	Li(1)-O(5) 2.177(4)
Li(3)-O(3) 1.959(4)	Al(3)-C(8) 1.973(3)	Li(1)-O(2) 2.238(4)
Li(3)-O(1) 1.961(4)	Al(3)-C(9) 1.976(3)	O(1)-C(11) 1.346(2)
Li(3)-O(2)#1 2.035(4)	Al(3)-C(7) 1.982(3)	O(2)-C(21) 1.359(2)
Li(3)-O(5) 2.179(4)	Li(1)-O(4) 2.053(6)	
Li(3)-O(9) 2.244(4)	Li(1)-O(1) 2.090(6)	
O(1)-C(1) 1.325(2)	Li(1)-O(2) 2.082(6)	**Angles[deg]**
O(1)-Li(2)#1 2.058(4)	Li(1)-O(6) 2.098(6)	
O(2)-C(21) 1.335(3)	Li(2)-O(2) 2.014(5)	Al(1)-O(1)-Li(1) 106.32(13)
O(2)-Li(3)#1 2.035(4)	Li(2)-O(3) 2.047(5)	Al(1)-O(2)-Li(1)#2 117.63(12)
O(3)-C(11) 1.326(2)	Li(2)-O(7) 2.078(5)	Al(1)-O(2)-Li(1) 92.82(11)
O(3)-Li(1)#1 1.982(4)	Li(2)-O(8) 2.146(5)	Li(1)#2-O(2)-Li(1) 86.51(15)
	Li(3)-O(3) 2.005(5)	O(1)-Al(1)-O(2) 86.93(7)
	Li(3)-O(5) 2.023(5)	O(1)-Al(1)-C(1) 115.35(10)
Angles[deg]	Li(3)-O(1) 2.070(5)	O(2)-Al(1)-C(1) 107.33(9)
	Li(3)-O(9) 2.073(6)	O(1)-Al(1)-C(2) 111.84(11)
O(2)-Li(1)-O(1) 119.7(2)	O(1)-C(34) 1.380(3)	O(2)-Al(1)-C(2) 111.11(10)
O(2)-Li(1)-O(3)# 197.96(18)	O(2)-C(48) 1.365(3)	C(1)-Al(1)-C(2) 119.35(12)
O(1)-Li(1)-O(3)# 195.84(18)	O(3)-C(27) 1.376(3)	O(1)-Li(1)-O(2)#2 128.5(2)
O(2)-Li(1)-O(4) 136.7(2)		O(1)-Li(1)-O(6) 122.8(2)
O(1)-Li(1)-O(4) 83.05(15)		O(2)#2-Li(1)-O(6) 100.64(17)
O(3)#1-Li(1)-O(4) 117.07(19)	**Angles[deg]**	O(1)-Li(1)-O(5) 77.95(14)
O(2)-Li(1)-C(1) 138.82(18)		O(2)#2-Li(1)-O(5) 117.3(2)
O(3)#1-Li(1)-C(1) 106.23(16)	O(4)-Li(1)-O(1)79.9(2)	O(6)-Li(1)-O(5) 106.59(17)
O(2)-Li(2)-O(3) 124.3(2)	O(4)-Li(1)-O(2)170.4(3)	
O(2)-Li(2)-O(1)# 191.47(16)	O(1)-Li(1)-O(2)104.4(2)	
O(3)-Li(2)-O(1)# 192.77(17)	O(4)-Li(1)-O(6)104.2(3)	
O(2)-Li(2)-O(7) 78.15(15)	O(1)-Li(1)-O(6)140.9(3)	
O(3)-Li(2)-O(7) 123.26(19)	O(2)-Li(1)-O(6)77.9(2)	
O(1)#1-Li(2)-O(7) 142.1(2)	O(2)-Li(2)-O(3)116.3(2)	
O(2)-Li(2)-O(8) 156.8(2)	O(3)-Li(3)-O(5)130.8(3)	
O(3)-Li(2)-O(8) 77.09(13)	O(3)-Li(3)-O(1)120.7(3)	
O(1)#1-Li(2)-O(8) 96.67(16)	O(5)-Li(3)-O(1)80.53(19)	
O(7)-Li(2)-O(8) 82.16(14)	O(3)-Li(3)-O(9)81.6(2)	
O(3)-Li(3)-O(1) 101.74(18)	O(5)-Li(3)-O(9)96.8(2)	
O(3)-Li(3)-O(2)#1 94.57(17)	Al(1)-O(1)-Li(3)98.66(17)	
O(1)-Li(3)-O(2)#1 91.73(16)	Al(1)-O(1)-Li(1)108.57(18)	
O(1)-Li(3)-O(5) 79.09(14)	Li(3)-O(1)-Li(1)123.3(2)	
O(2)#1-Li(3)-O(5) 103.63(17)	Al(2)-O(2)-Li(2)100.62(17)	
O(3)-Li(3)-O(9) 77.51(13)	Al(2)-O(2)-Li(1)95.94(18)	
O(1)-Li(3)-O(9) 143.5(2)	Li(2)-O(2)-Li(1)129.2(2)	
O(2)#1-Li(3)-O(9) 124.8(2)	Al(3)-O(3)-Li(3)105.32(17)	

O(5)-Li(3)-O(9)	90.97(15)	Al(3)-O(3)-Li(2)116.89(16)		
Li(1)-O(1)-Li(3)	132.41(17)	Li(3)-O(3)-Li(2)113.2(2)		
Li(1)-O(2)-Li(2)	109.97(17)			
Li(2)-O(3)-Li(3)	126.82(17)			
Symmetry transformations used to generate equivalent atoms: for **1**: #1 -x+1,-y,-z+1, for **3·C₇H₈** #1 -x+2,-y+1,-z #2 -x+3/2,-y,z				

Symmetry transformations used to generate equivalent atoms: for **1**: #1 -x+1,-y,-z+1, for **3·C$_7$H$_8$** #1 -x+2,-y+1,-z   #2 -x+3/2,-y,z

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