

## Extending Chain Growth Beyond C<sub>1</sub> → C<sub>4</sub> in CO Homologation: Aluminyl Promoted Formation of the [C<sub>5</sub>O<sub>5</sub>]<sup>5-</sup> Ligand

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## General Experimental Procedures

All manipulations were performed under dry argon using standard Schlenk-line techniques, or in a conventional nitrogen-filled glovebox. Solvents were dried over appropriate drying agents and degassed prior to use. NMR spectra were recorded using a Jeol JNM-ECZ500S 500 MHz spectrometer equipped with a ROYAL digital auto tune probe S, operating at 500.1 ( $^1\text{H}$ ) and 125.7 ( $^{13}\text{C}\{^1\text{H}\}$ ) MHz. Spectra were recorded at 298 K (unless stated otherwise) and proton and carbon chemical shifts were referenced internally to residual solvent resonances. Coupling constants are quoted in Hz.  $\text{K}[\text{Al}(\text{NON}^{\text{DiPP}})]^1$  and  $(\text{NON}^{\text{DiPP}})\text{Al}-\text{K}(\text{TMEDA})_2$ ,<sup>2</sup> were prepared according to the literature procedures. Oxygen free CO was purchased from BOC NZ and used without further purification. All other chemicals were purchased from Sigma-Aldrich and used without further purification.

### Synthesis of $\text{K}_2[\{\text{Al}(\text{NON}^{\text{DiPP}})\}_2(\text{C}_4\text{O}_4)]$ (1)

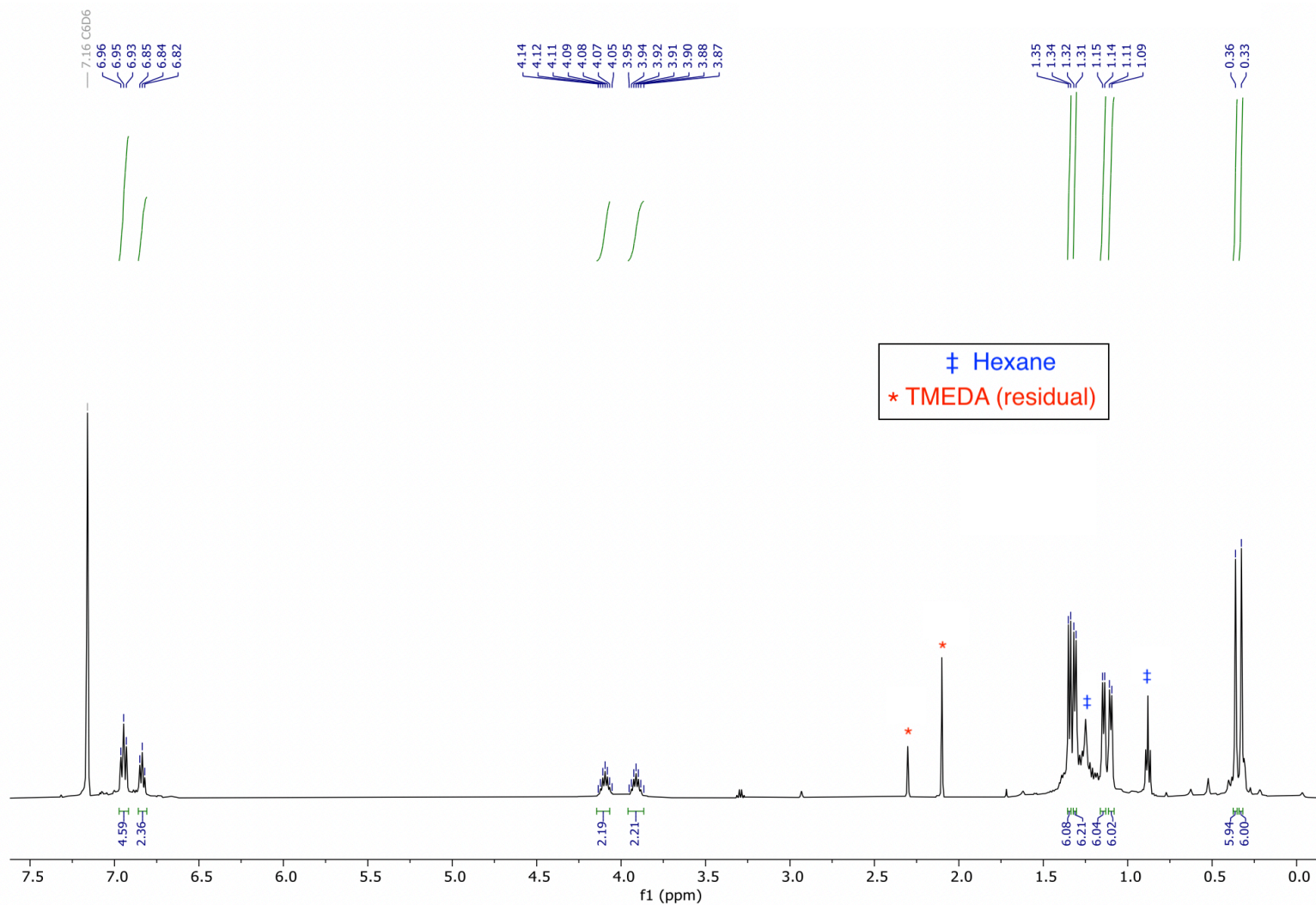
$(\text{NON}^{\text{DiPP}})\text{Al}-\text{K}(\text{TMEDA})_2$  (I, 115 mg, 0.15 mmol) was dissolved in toluene- $\text{D}_8$  and transferred to an NMR tube fitted with a J. Young tap. The solution was de-gassed and carbon monoxide (~1.5 bar) was introduced to the NMR tube. The reaction mixture was stored at room temperature for 4 days to give a dark violet solution. The solution was transferred to a vial and the solvent removed *in vacuo*. Crystallisation was achieved by slow evaporation a hexane/diethyl ether mixture (2:1, 5 mL) at room temperature. Yield 56 mg, 63 %. Signals corresponding to hexane (solvate) and residual TMEDA are present in the  $^1\text{H}$  NMR spectrum.

Anal. Calcd. for  $\text{C}_{60}\text{H}_{92}\text{Al}_2\text{K}_2\text{N}_4\text{O}_6\text{Si}_4$  (1209.92): C, 59.56; H, 7.66; N, 4.63 %. Found: C, 58.83; H, 7.91; N, 4.32 %. We acknowledge that these values do not correspond to an 'accurate' elemental analysis. However, these data are the best that we were able to obtain, and the poor match likely reflects the high sensitivity to air / moisture and the inclusion of varying amounts of solvent / TMEDA in the isolated crystals.

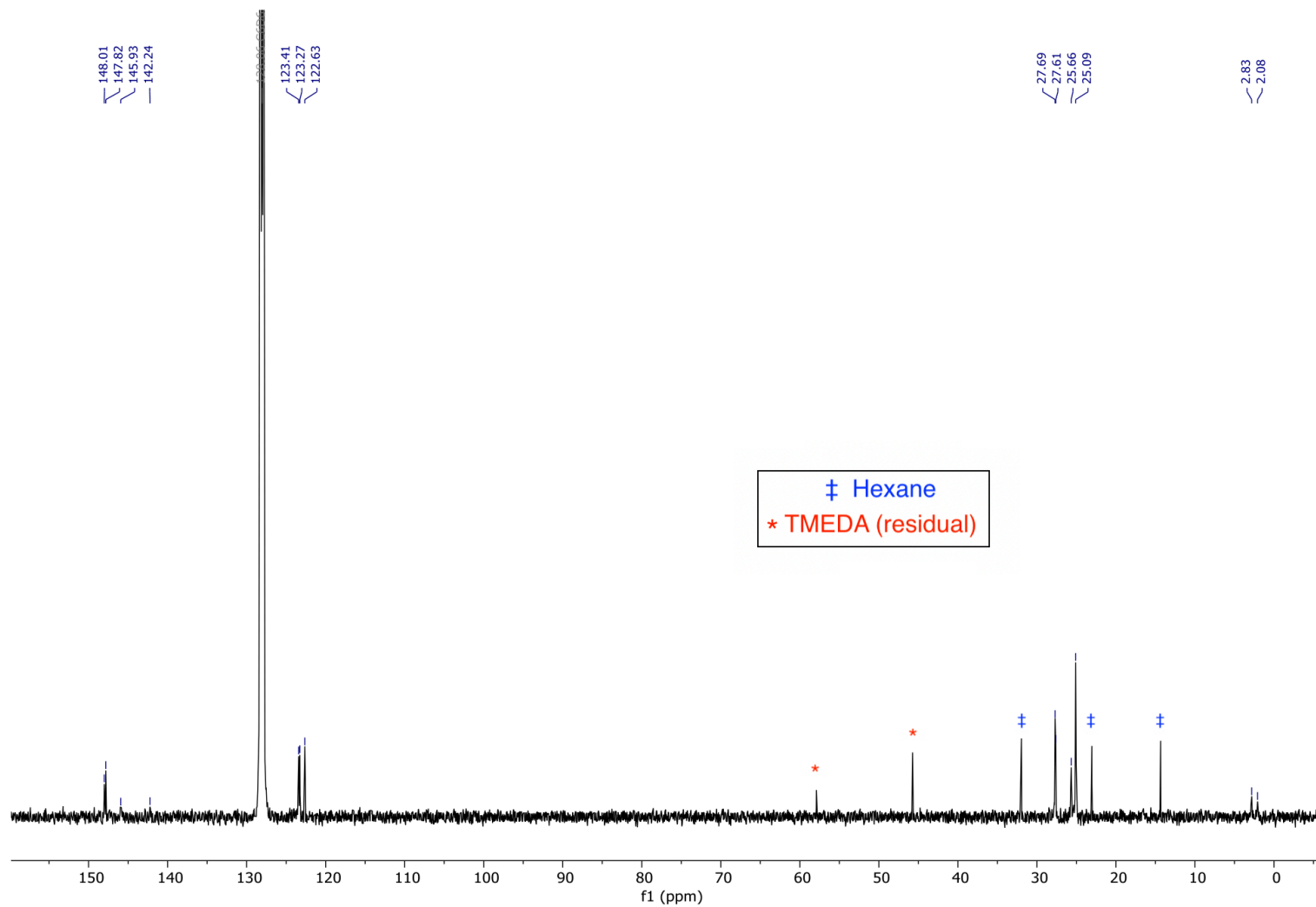
$^1\text{H}$  NMR (500 MHz,  $\text{C}_6\text{D}_6$ , 343 K):  $\delta$  6.95 (t,  $J = 8.1$ , 4H,  $\text{C}_6\text{H}_3$ ), \*, 6.84 (t,  $J = 7.6$ , 2H,  $\text{C}_6\text{H}_3$ ), 4.09 (sept,  $J = 6.8$ , 2H,  $\text{CHMe}_2$ ), 3.91 (sept,  $J = 6.8$ , 2H,  $\text{CHMe}_2$ ), 1.34 (d,  $J = 6.8$ , 6H,  $\text{CHMe}_2$ ), 1.31 (d,  $J = 6.8$ , 6H,  $\text{CHMe}_2$ ), 1.14 (d,  $J = 6.8$ , 6H,  $\text{CHMe}_2$ ), 1.10 (d,  $J = 6.8$ , 6H,  $\text{CHMe}_2$ ), 0.36 (s, 6H,  $\text{SiMe}_2$ ), 0.33 (s, 6H,  $\text{SiMe}_2$ ).

$^{13}\text{C}\{^1\text{H}\}$  NMR (126 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta$  148.0, 147.8, 145.9 ( $\text{C}_6\text{H}_3$ ), 142.2 (CO), 123.4, 123.3, 122.6 ( $\text{C}_6\text{H}_3$ ), 27.7, 27.6 ( $\text{CHMe}_2$ ), 25.7, 25.1 ( $\text{CHMe}_2$ ), 2.8, 2.1 ( $\text{SiMe}_2$ ).

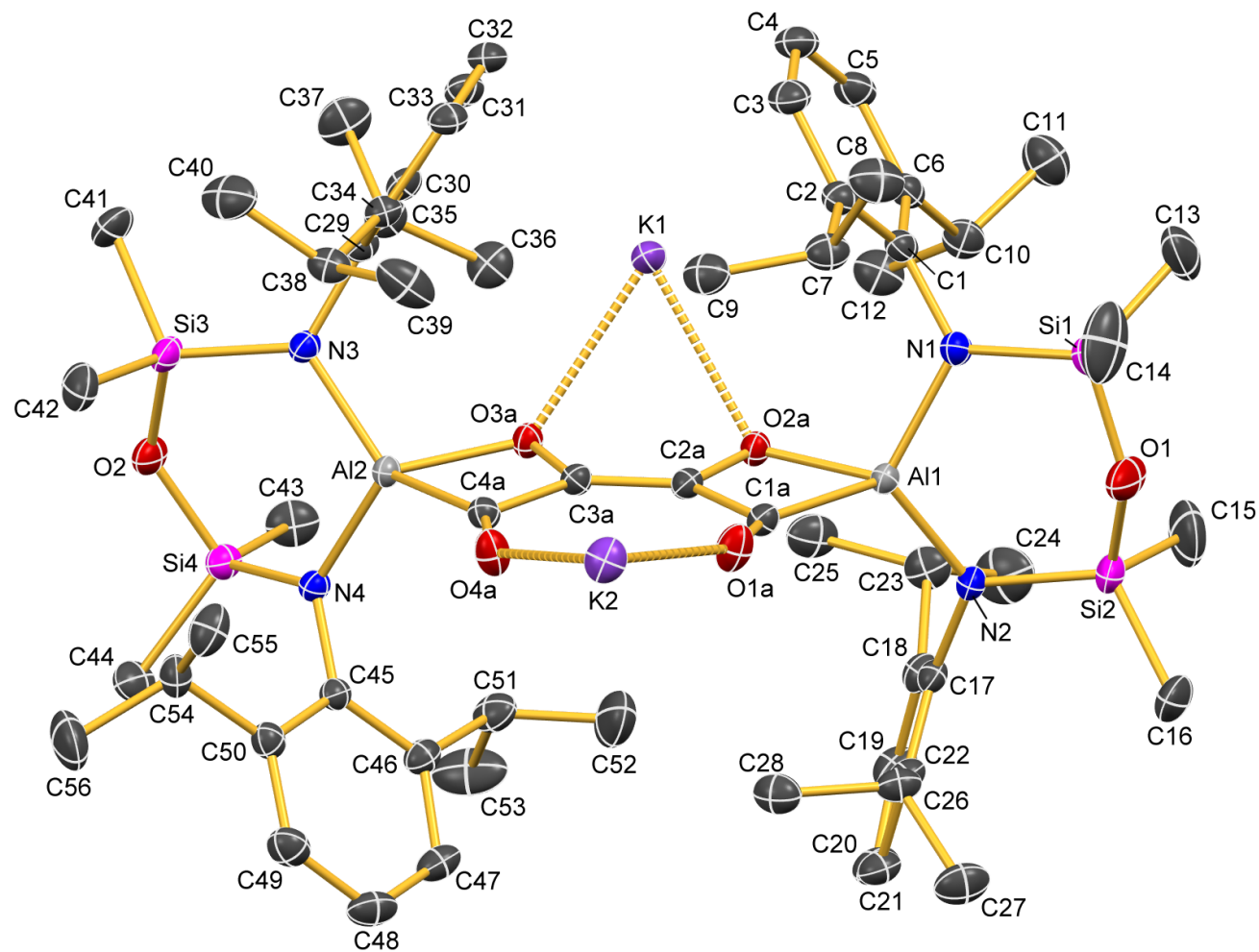
**Figure S1**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{C}_6\text{D}_6$ , 343 K) of  $\text{K}_2[\{\text{Al}(\text{NON}^{\text{Dipp}})\}_2(\text{C}_4\text{O}_4)]$  (**1**)



**Figure S2**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum (126 MHz,  $\text{C}_6\text{D}_6$ ) of  $\text{K}_2[\{\text{Al}(\text{NON}^{\text{Dipp}})\}_2(\text{C}_4\text{O}_4)]$  (**1**)



**Figure S3** Displacement ellipsoid plot (30 % probability, H-atoms and disordered solvent omitted) of the asymmetric unit of  $K_2[\{Al(NON^{Dipp})\}_2(C_4O_4)]$  (**1**)



## Synthesis of $K_5[K(TMEDA)][\{Al(NON^{Dipp})\}_4(C_5O_5)_2]$ (**2**)

*Method 1:* A solution of  $[K\{Al(NON^{Dipp})\}]_2$  (**II**), 320 mg, 0.29 mmol) in toluene was prepared in an ampoule. The solution was degassed and carbon monoxide (~1.5 bar) introduced to the reaction vessel. The mixture was stirred at room temperature for 20 days to give a dark red solution. The solution was transferred to a vial and the solvent removed *in vacuo*. The product was then extracted in pentane (3 x 3 mL) and the volatiles removed *in vacuo*. Crystallisation was achieved from a TMEDA/toluene mixture (1:10, ~3 mL) *via* slow evaporation at room temperature. Yield 23 mg, 6 %.

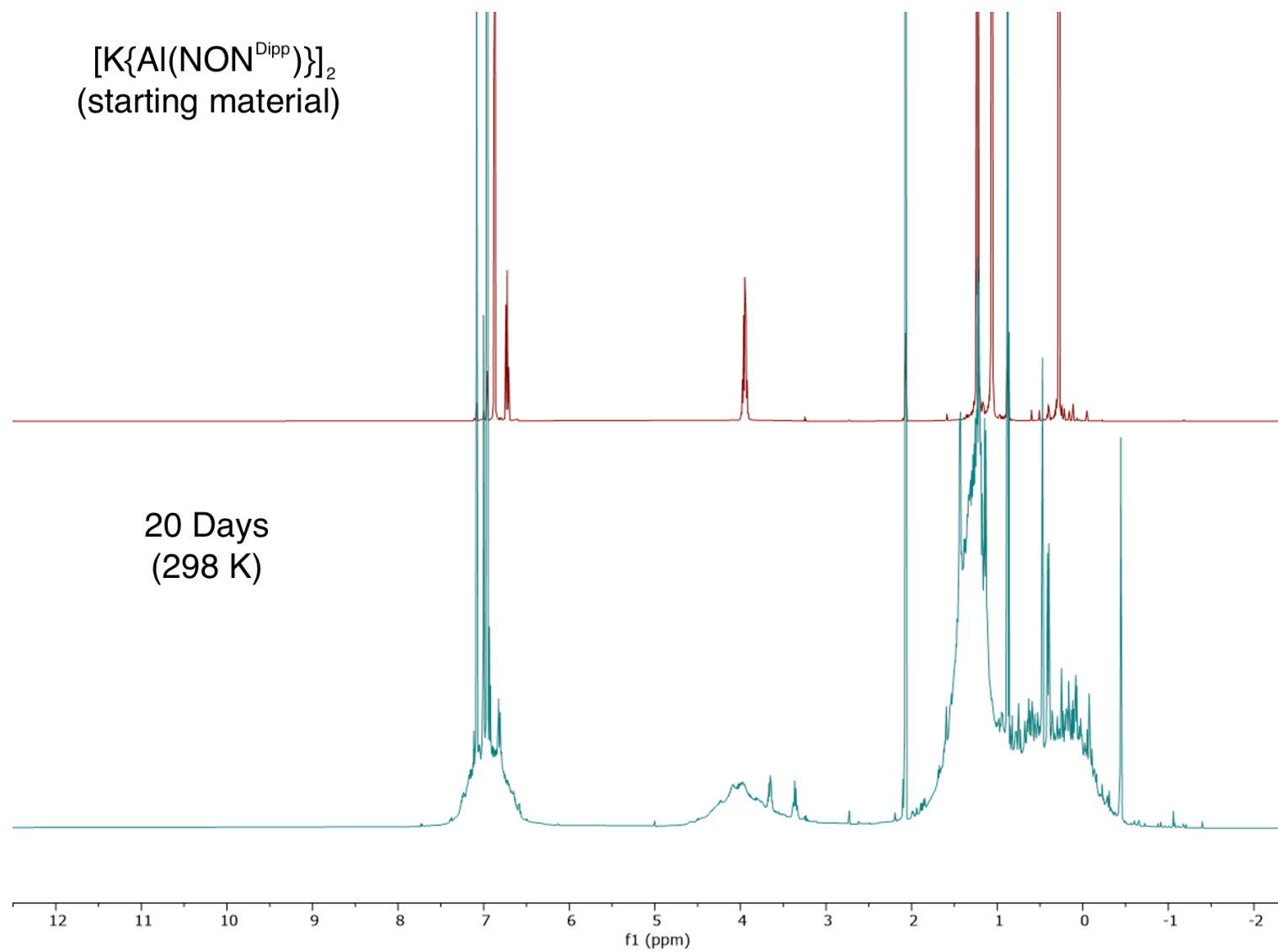
Repeating the reaction at 80 °C and monitoring by  $^1H$  NMR spectroscopy indicated the same product was formed.

*Method 2:*  $(NON^{Dipp})Al-K(TMEDA)_2$  (**I**), 105 mg, 0.14 mmol) was dissolved in a toluene/ $C_6D_6$  mixture (1:50, 0.60 mL) and transferred to an NMR tube fitted with a J. Young tap. The solution was degassed and carbon monoxide (~1.5 bar) introduced to the NMR tube. The reaction mixture was heated at 60 °C for 2 days to give an intense purple solution. The solution was transferred to a scintillation vial and the solvent removed *in vacuo*. Crystallisation was achieved from a (neat) toluene solution stored at room temperature *via* slow evaporation. Yield 31 mg, 33 %. A sample was analysed by X-ray crystallography, confirming, it as  $K_5[K(TMEDA)][\{Al(NON^{Dipp})\}_4(C_5O_5)_2]$  (**2**).

$^1H$  NMR (500 MHz,  $C_6D_6$ , 333 K):  $\delta$  6.95 – 6.79 (m, 4H,  $C_6H_3$ ), 6.75 (t,  $J = 7.4$ , 1H,  $C_6H_3$ ), 6.64 (t,  $J = 7.4$ , 1H,  $C_6H_3$ ), 4.06 – 3.94 (m, 4H,  $CHMe_2$ ), 2.32 (s, 2H, TMEDA), 2.12 (s, 6H, TMEDA), 1.34 – 1.14 (m, 24H,  $CHMe_2$ ), 0.63 (s, 6H,  $SiMe_2$ ), 0.53 (s, 6H,  $SiMe_2$ ).

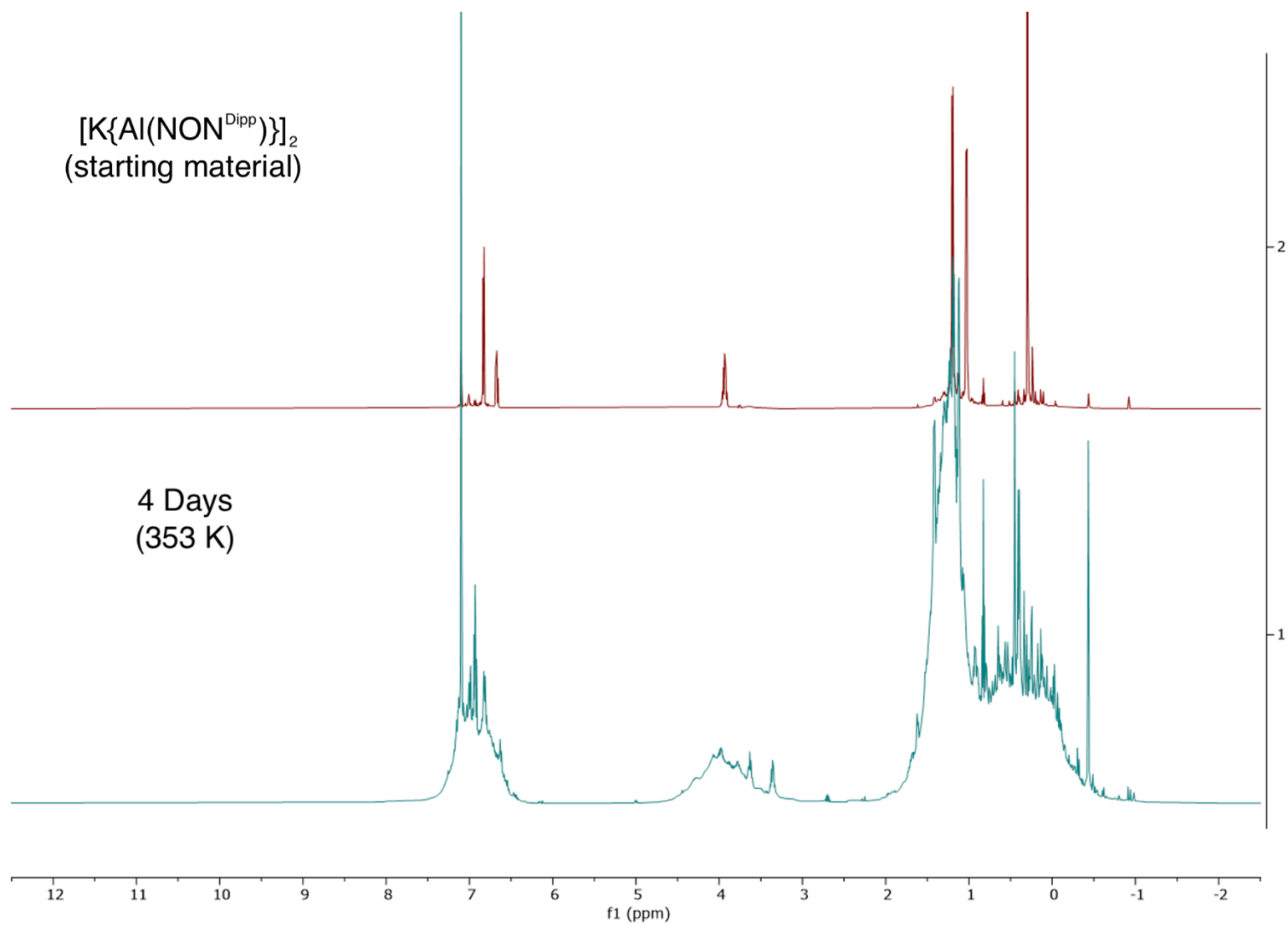
We are unable to confidently assign the  $^{13}C\{^1H\}$  NMR spectrum (Figure S7) due to the presence of unidentified  $NON^{Dipp}$ -containing impurities (see Figure S6), and decomposition of the sample during the acquisition of  $^{13}C\{^1H\}$  NMR data. The high sensitivity of the product to air and moisture prevented analysis for elemental composition.

**Figure S4**  $^1\text{H}$  NMR spectra (500 MHz,  $\text{C}_6\text{D}_6$ ) of reaction of  $[\text{K}\{\text{Al}(\text{NON}^{\text{Dipp}})\}]_2$  with CO (after 20 days at 298 K)

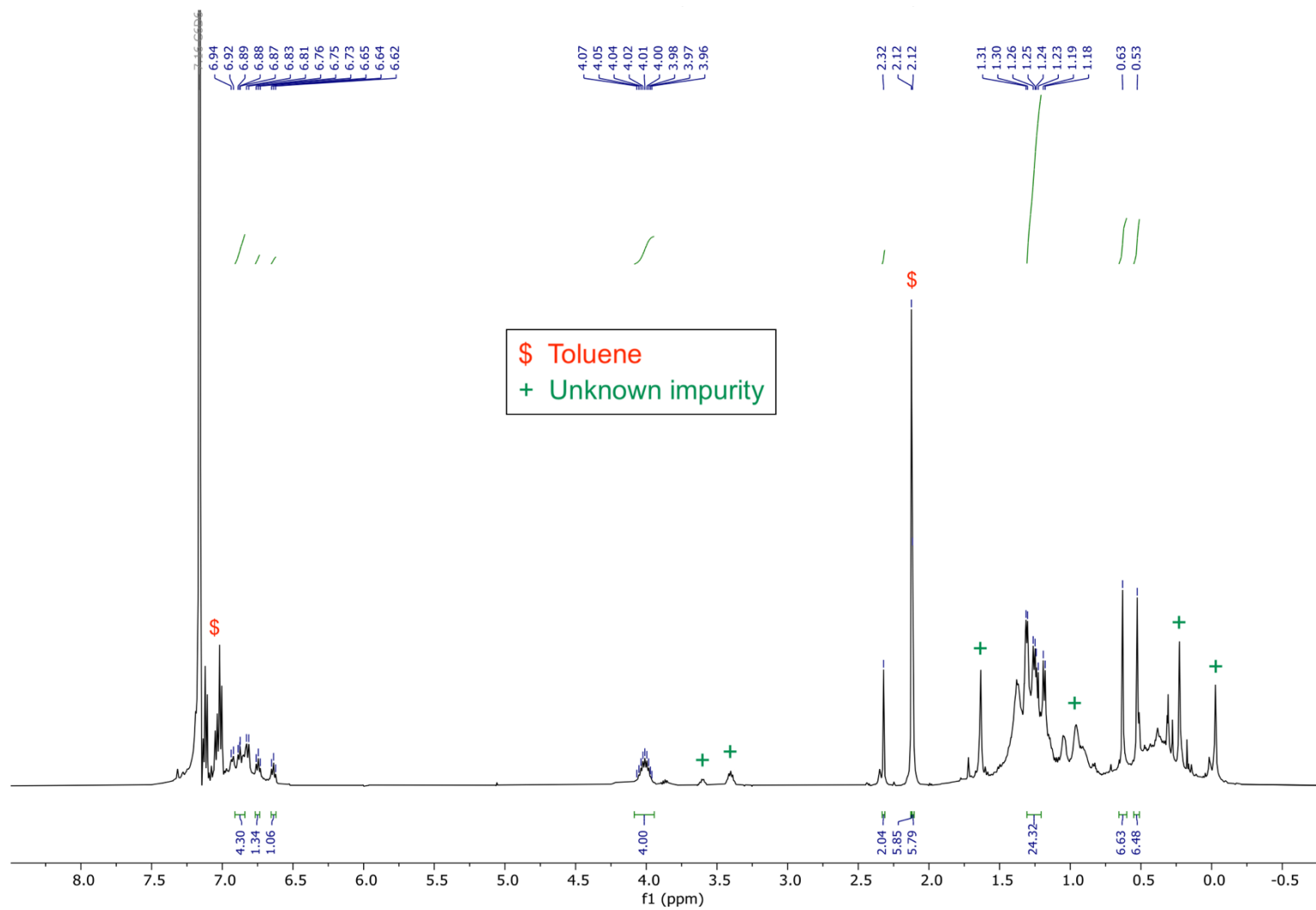




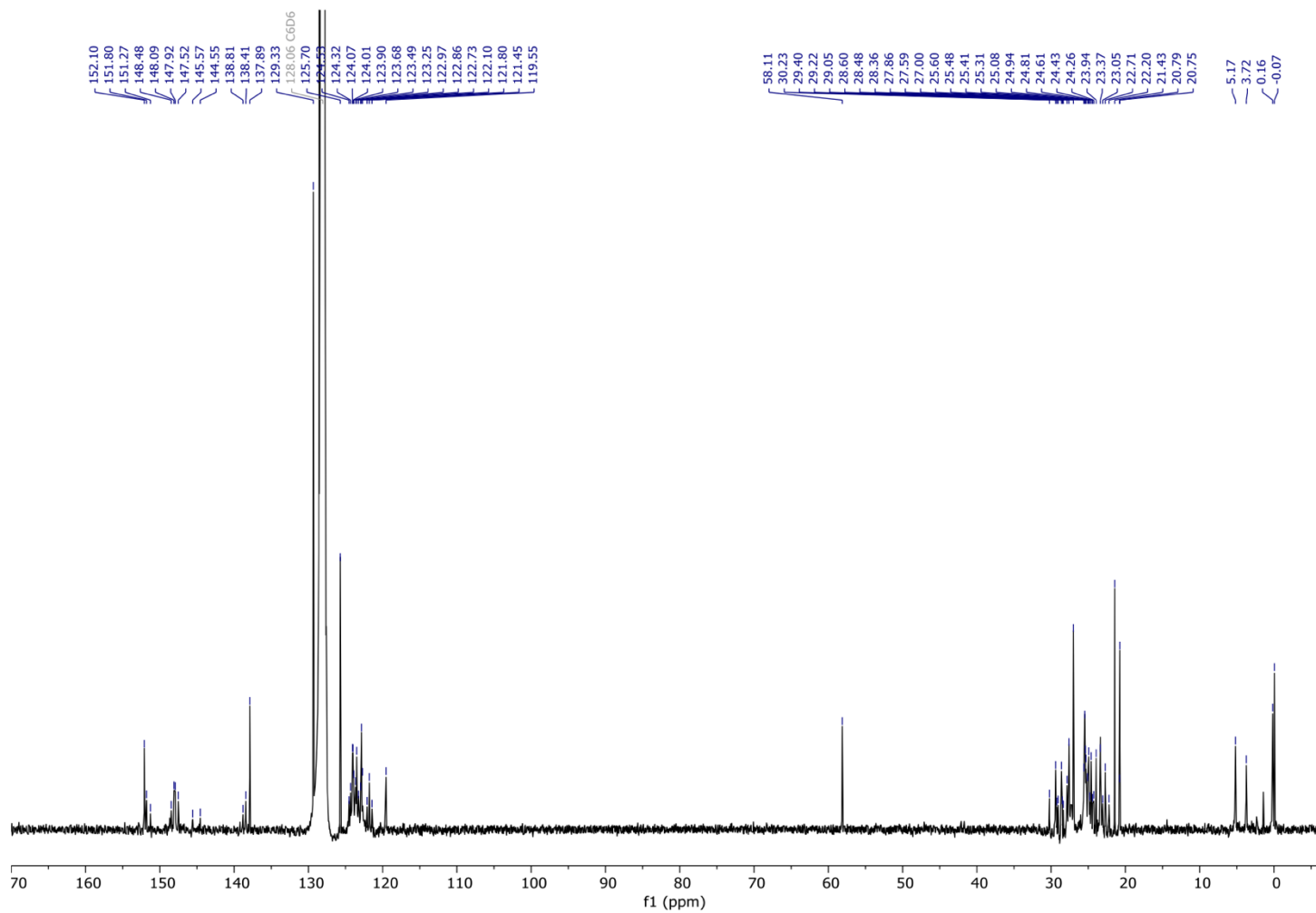
**Figure S5**  $^1\text{H}$  NMR spectra (500 MHz,  $\text{C}_6\text{D}_6$ ) of reaction of  $[\text{K}\{\text{Al}(\text{NON}^{\text{Dipp}})\}]_2$  with CO (after 4 days at 353 K)



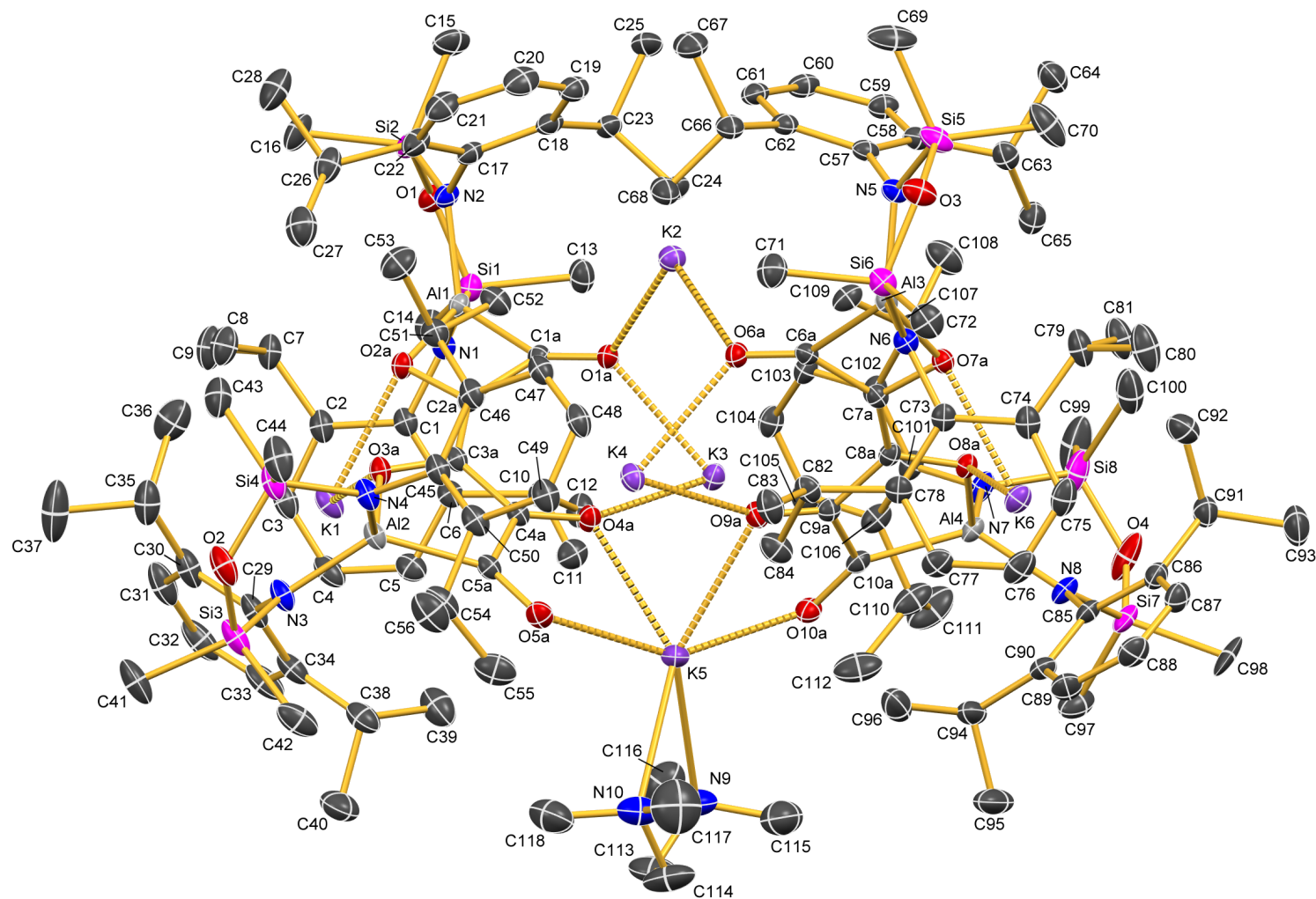
**Figure S6**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{C}_6\text{D}_6$ ) of isolated crystals of  $\text{K}_5[\text{K}(\text{TMEDA})][\{\text{Al}(\text{NON}^{\text{Dipp}})\}_4(\text{C}_5\text{O}_5)_2]$  (**2**)



**Figure S7**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum (126 MHz,  $\text{C}_6\text{D}_6$ ) of isolated crystals of  $\text{K}_5[\text{K}(\text{TMEDA})][\{\text{Al}(\text{NON}^{\text{Dipp}})\}_4(\text{C}_5\text{O}_5)_2]$  (**2**)



**Figure S8** Displacement ellipsoid plot (30 % probability, H-atoms omitted) of the asymmetric unit of  $K_5[K(TMEDA)][\{Al(NON^{Dipp})\}_4(C_5O_5)_2]$  (**2**)



### Synthesis of $[K(TMEDA)]_2[\{Al(NON^{DiPP})\}_2(C_4O_4)]$ (**3**)

$K_2[\{Al(NON^{DiPP})\}_2(C_4O_4)]$  (13 mg, 0.01 mmol) and ~0.2 mL TMEDA (excess, 12 equiv.) were dissolved in benzene- $D_6$  in an NMR tube fitted with a J Young tap. The solution was degassed and carbon monoxide (~1.5 bar) introduced to the NMR tube. The reaction mixture was heated at 60 °C for 2 days to give a dark red solution. The solution was transferred to a vial and the solvent removed *in vacuo*. Crystallisation was achieved from a hexane solution (2 mL) *via* slow evaporation at room temperature. Yield 21 mg (72 %, calculated for  $[K(TMEDA)]_2[\{Al(NON^{DiPP})\}_2(C_4O_4)] \cdot 12(C_6H_{14}) \cdot 4(TMEDA)$ , with the equivalents of hexane and TMEDA calculated from  $^1H$  NMR integration).

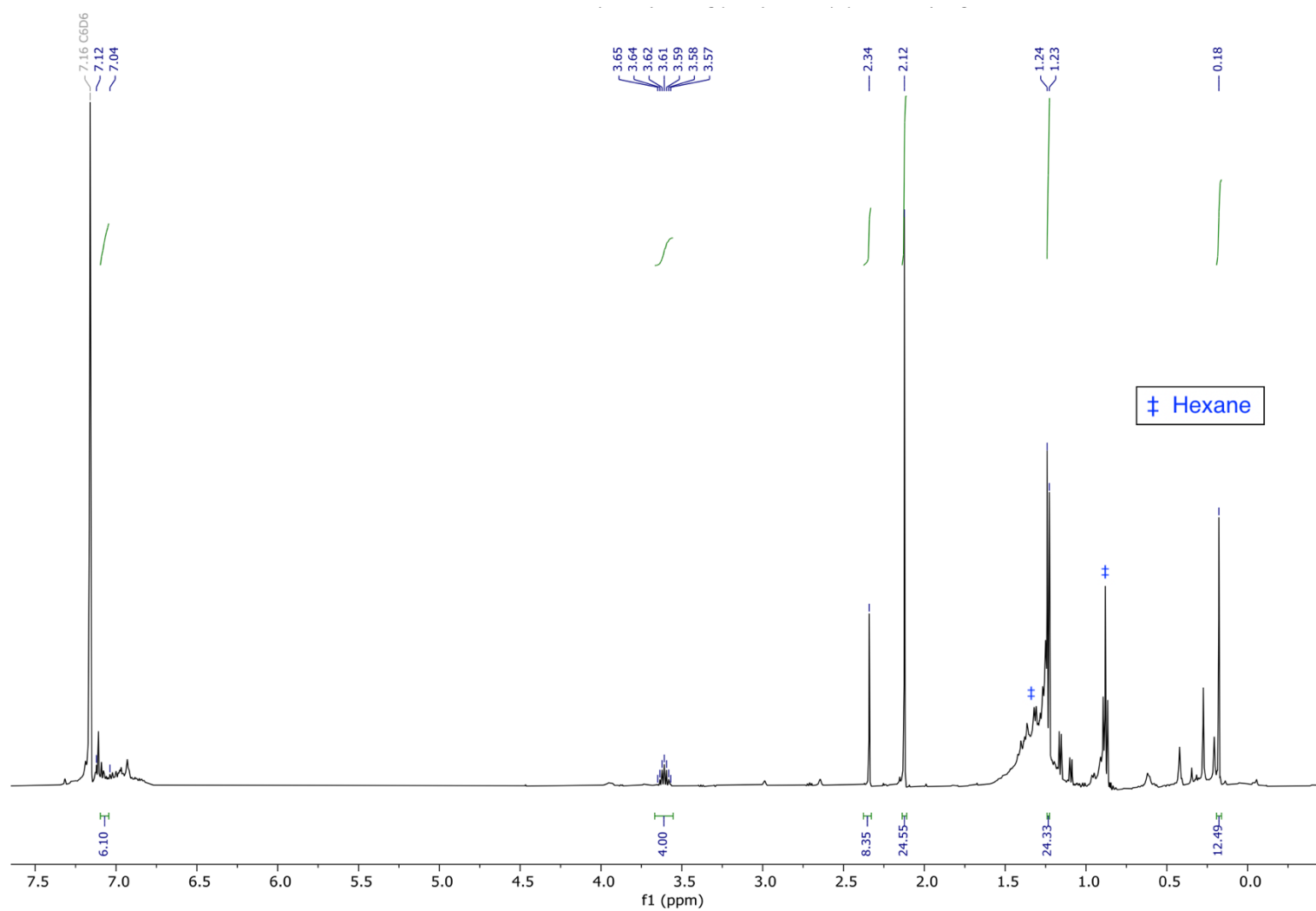
The isolated crystals indicate the presence of a major species which contains time-averaged  $C_{2h}$  symmetry in solution. Hexane and TMEDA are present in the  $^1H$  NMR spectrum.

Insufficient material was isolated to allow a sample to be analysed for elemental composition.

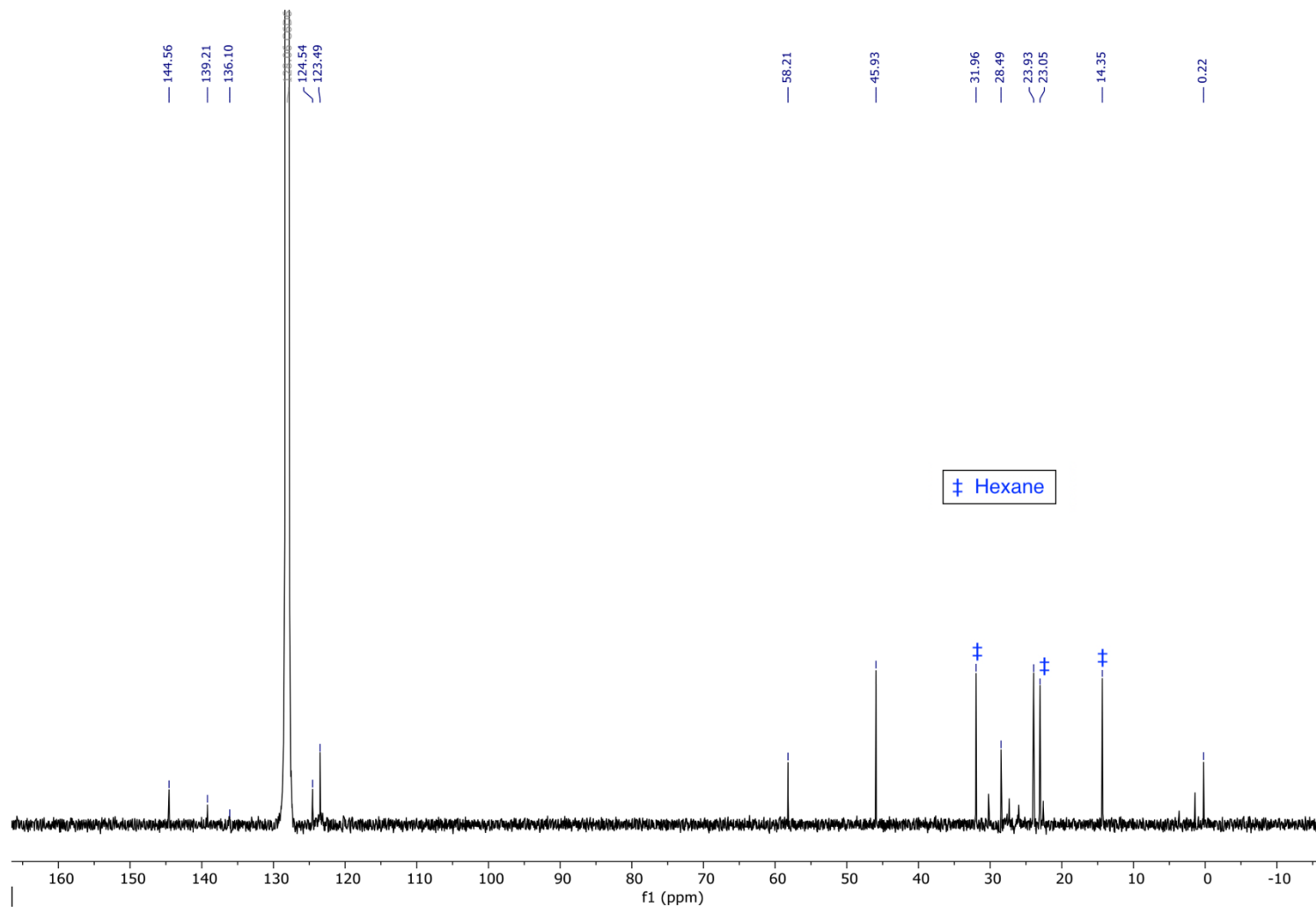
$^1H$  NMR (500 MHz,  $C_6D_6$ , 343 K):  $\delta$  7.12–7.04 (m, 6H,  $C_6H_3$ ), 3.61 (sept,  $J = 6.8$ , 4H,  $CHMe_2$ ), 2.34 (s, 8H, TMEDA- $CH_2$ ), 2.12 (s, 24H, TMEDA- $CH_3$ ), 1.24 (d,  $J = 6.8$ , 24H,  $CHMe_2$ ), 0.18 (s, 12H,  $SiMe_2$ ).

$^{13}C\{^1H\}$  NMR (126 MHz,  $C_6D_6$ ):  $\delta$  144.6, 139.2 ( $C_6H_3$ ), 136.1 (CO), 124.5, 123.5 ( $C_6H_3$ ), 28.5 ( $CHMe_2$ ), 23.9 ( $CHMe_2$ ), 0.2 ( $SiMe_2$ ).

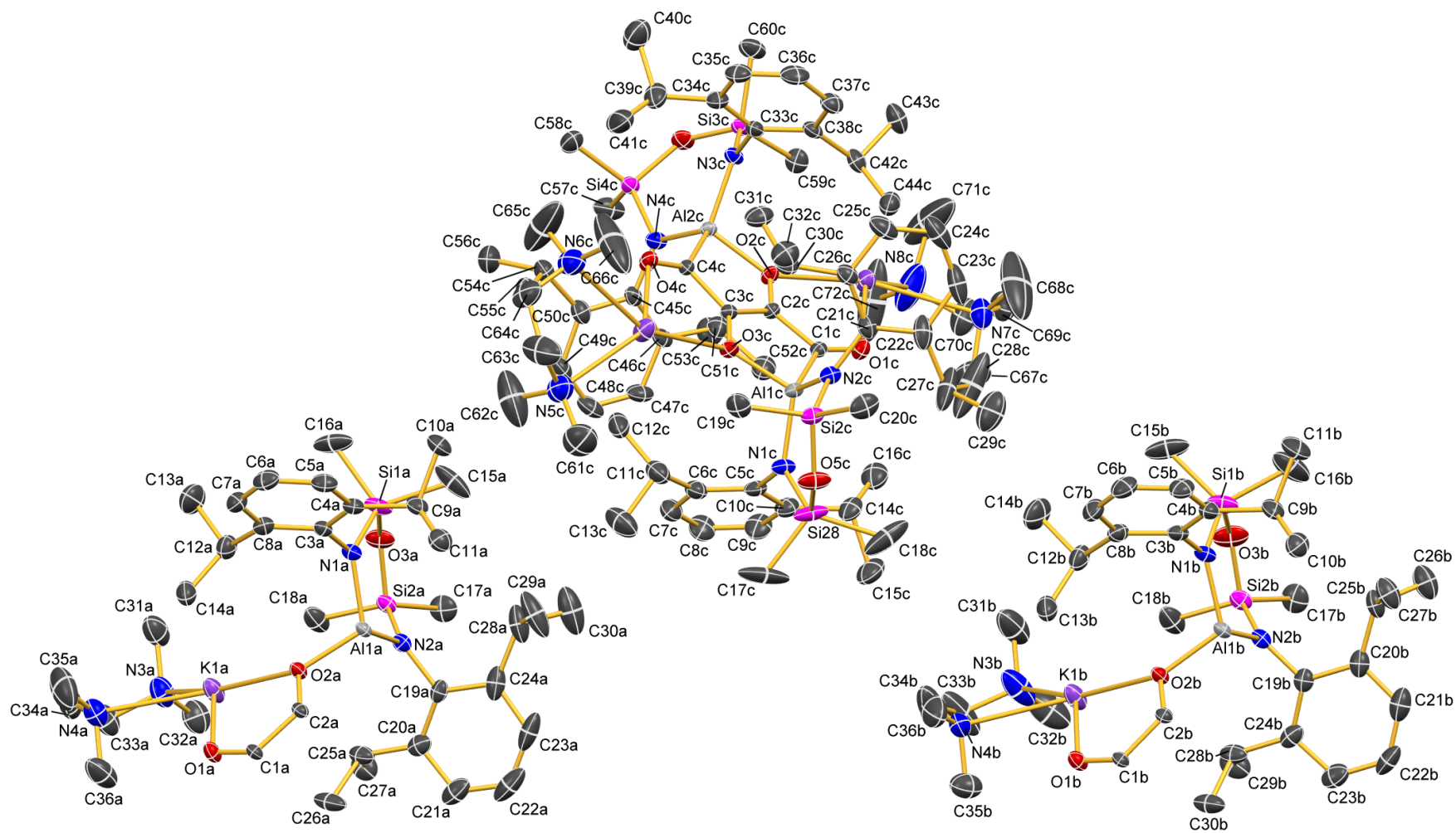
**Figure S9**  $^1\text{H}$  NMR spectrum (500 MHz,  $\text{C}_6\text{D}_6$ ) of  $[\text{K}(\text{TMEDA})]_2[\{\text{Al}(\text{NON}^{\text{Dipp}})\}_2(\text{C}_4\text{O}_4)]$  (**3**)



**Figure S10**  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum (126 MHz,  $\text{C}_6\text{D}_6$ ) of  $[\text{K}(\text{TMEDA})]_2[\{\text{Al}(\text{NON}^{\text{Dipp}})\}_2(\text{C}_4\text{O}_4)]$  (**3**)

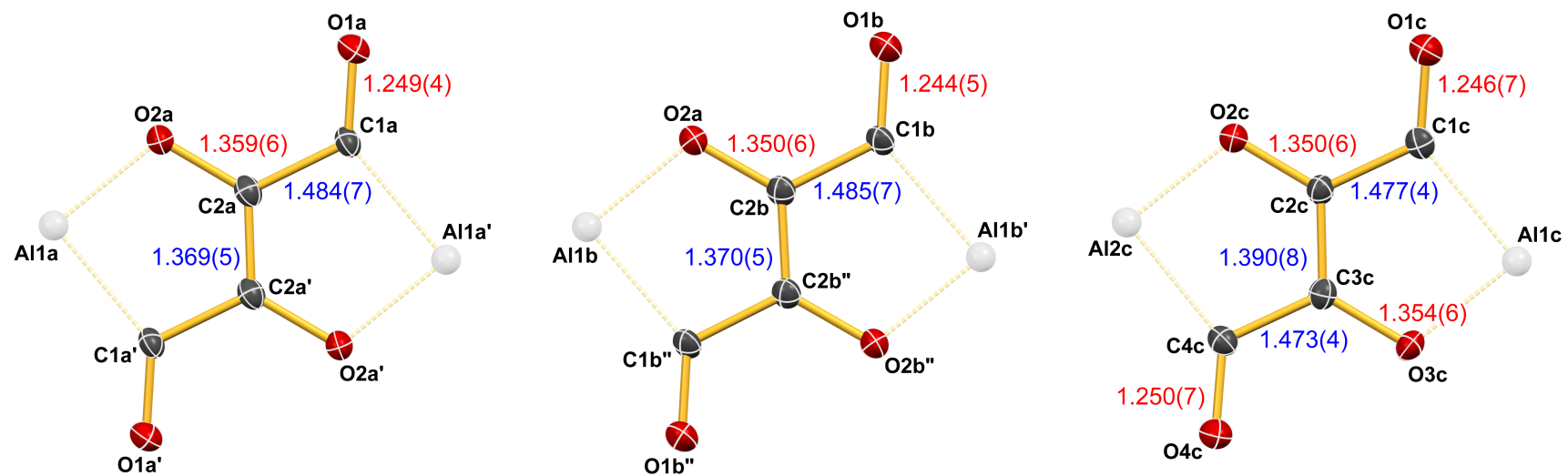


**Figure S11** Displacement ellipsoid plot (30 % probability, H-atoms, disordered atoms and hexane solvates omitted) of the asymmetric unit of  $[\text{K}(\text{TMEDA})]_2[\{\text{Al}(\text{NON}^{\text{Dipp}})\}_2(\text{C}_4\text{O}_4)]$ ,  $2 [\text{K}(\text{TMEDA})][\text{Al}(\text{NON}^{\text{Dipp}})(\text{C}_2\text{O}_2)]$  (**3**)

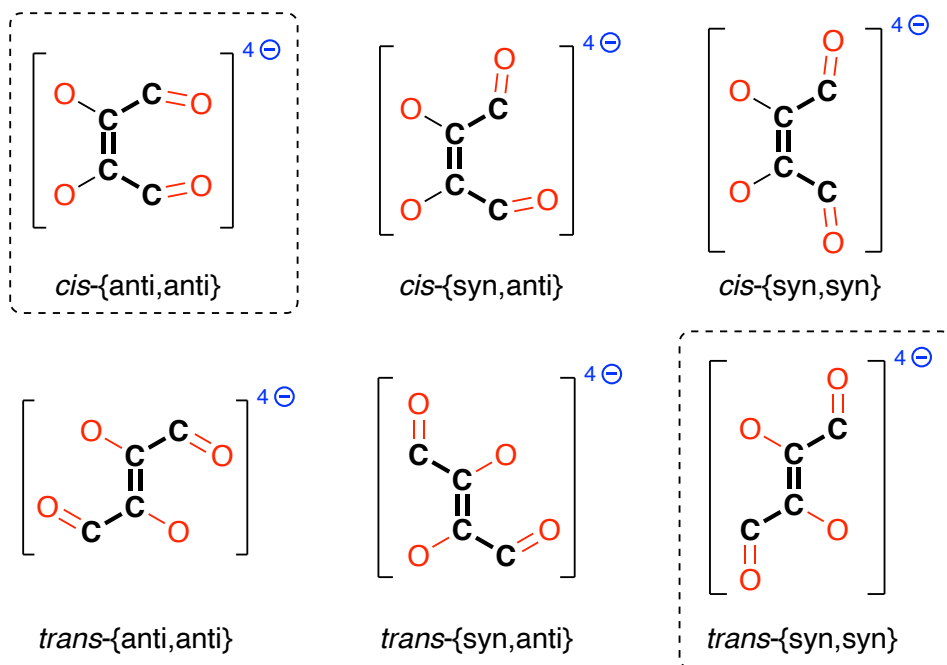




**Figure S12** C–C (blue) and C–O (red) bond lengths in the three independent *trans*-[C<sub>4</sub>O<sub>4</sub>]<sup>4-</sup> ligands (' = -x, 2-y, 2-z; " = -1-x, 1-y, -z).



**Figure S13** Classification of different isomeric forms of the  $[\text{C}_4\text{O}_4]^{4-}$  anion



## Crystallographic Details

Crystals were covered in inert oil and suitable single crystals were selected under a microscope and mounted on an Agilent SuperNova diffractometer fitted with an EOS S2 detector. Data were collected at 150 K (unless indicated otherwise) using focused microsource Cu K $\alpha$  radiation at 1.54184 Å. Intensities were corrected for Lorentz and polarisation effects and for absorption using multi-scan methods.<sup>3</sup> Space groups were determined from systematic absences and checked for higher symmetry. All structures were solved using direct methods with SHELXS,<sup>4</sup> refined on  $F^2$  using all data by full matrix least-squares procedures with SHELXL-97,<sup>5</sup> within the WinGX (**1** and **2**)<sup>6</sup> or OLEX2 (**3**)<sup>7</sup> programs. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were placed in calculated positions or manually assigned from residual electron density where appropriate, unless otherwise stated. The functions minimized were  $\sum w(F2o-F2c)$ , with  $w = [\sigma^2(F2o) + aP^2 + bP]^{-1}$ , where  $P = [\max(Fo)^2 + 2F2c]/3$ . The isotropic displacement parameters are 1.2 or 1.5 times the isotropic equivalent of their carrier atoms.

### Additional Information:

$K_2[\{Al(NON^{Dipp})\}_2(C_4O_4)]$  (**1**): The asymmetric unit contains a poorly resolved hexane solvate molecule. This was modelled with two of the methylene units disordered over two positions and all of the carbon atoms isotropic.

$K_5[K(TMEDA)][\{Al(NON^{Dipp})\}_4(C_5O_5)_2]$  (**2**): One of the SiMe<sub>2</sub> groups is disordered and was modelled over two positions. In addition, the asymmetric unit contains 2 molecules of toluene and 2.5 molecules of TMEDA that were poorly defined. These were treated as a diffuse contribution to the overall scattering without specific atom positions by SQUEEZE/PLATON. Details are included in the .cif.

$[K(TMEDA)]_2[\{Al(NON^{Dipp})\}_2(C_4O_4)]$ ,  $2[K(TMEDA)][Al(NON^{Dipp})(C_2O_2)]$  (**3**): Refined as a 2-component twin, which is outlined in the cif. Some of the solvent molecules were behaved, but others required a modest range of restraints, which were used in preference to a solvent mask approach. Some of the TMEDA ligands showed some type of apparent disorder, but suitable models couldn't be built for these (this is outlined in detail in the relevant B level VRF responses below). Additional disorder was noted in 3 of the 4 AlN<sub>2</sub>Si<sub>2</sub>O chelating units that relates to ring conformations, which could only be partly modelled due to the low occupancy of the minor components of 4, 9 and 15%, such that only the Si atoms of the minor components

were included. For the other atoms, the electron density (for C, O and N) were either too small and/or too near other atoms of the main component to allow stable refinement.

The asymmetric unit consists of one dimer and two half-molecules, each located on an inversion centre. The B level alert in the category relates mainly to the large, elongated ADPs of the Me groups of the some of the TMEDA ligands. Consideration was given to potential disorder of the ligands but no consistent model could be conceived due to the appearance of the ADPs of the C<sub>2</sub>H<sub>4</sub> linker and N atoms. Nevertheless, two site complementary occupancy disorder models for the Me groups alone were investigated but proved to be not effective.

**Table S1** Crystal structure and refinement data for **1**, **2** and **3**

	<b>1</b>	<b>2</b>	<b>3</b>
Empirical formula	C <sub>66</sub> H <sub>106</sub> Al <sub>2</sub> K <sub>2</sub> N <sub>4</sub> O <sub>6</sub> Si <sub>4</sub>	C <sub>157</sub> H <sub>256</sub> Al <sub>4</sub> K <sub>6</sub> N <sub>15</sub> O <sub>14</sub> Si <sub>8</sub>	C <sub>84</sub> H <sub>152</sub> Al <sub>2</sub> K <sub>2</sub> N <sub>8</sub> O <sub>6</sub> Si <sub>4</sub>
CCDC Number	2159813	2159814	2159815
<i>M<sub>r</sub></i>	1296.06	3144.99	1614.65
<i>T</i> [K]	150.0(1)	150(3)	150.0(1)
Crystal size [mm]	0.34 × 0.24 × 0.11	0.26 × 0.16 × 0.12	0.22 × 0.13 × 0.11
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	P2 <sub>1</sub> /n (alternative No. 14)	P2 <sub>1</sub> /c (No. 14)	P -1 (No. 2)
<i>a</i> [Å]	16.3863(2)	15.48560(7)	19.3051(7)
<i>b</i> [Å]	13.1487(2)	32.8594(2)	24.5006(10)
<i>c</i> [Å]	34.6063(4)	36.3809(2)	24.9778(13)
<i>α</i> [°]	90	90	115.548(6)
<i>β</i> [°]	96.7646(14)	97.0291(5)	108.416(5)
<i>γ</i> [°]	90	90	97.403(4)
<i>V</i> [Å <sup>3</sup> ]	7404.27(18)	18373.18(19)	9616.1(9)
<i>Z</i>	4	4	4
<i>D</i> <sub>calc.</sub> [mg m <sup>-3</sup> ]	1.163	1.137	1.115
Absorption coefficient [mm <sup>-1</sup> ]	2.359	2.398	1.913
<i>θ</i> range for data collection [°]	3.514 to 73.605	3.530 to 73.304	3.639 to 68.250
Reflections collected	98397	148511	114242
Independent reflections	14834 [ <i>R</i> <sub>int</sub> 0.053]	36547 [ <i>R</i> <sub>int</sub> 0.031]	35070 [ <i>R</i> <sub>int</sub> 0.064]
Reflections with <i>I</i> > 2σ( <i>I</i> )	13095	31728	23048
Data/restraints/parameters	14834 / 0 / 760	36547 / 0 / 1613	35070 / 85 / 1937
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.048, <i>wR</i> <sub>2</sub> = 0.130	<i>R</i> <sub>1</sub> = 0.043, <i>wR</i> <sub>2</sub> = 0.124	<i>R</i> <sub>1</sub> = 0.094, <i>wR</i> <sub>2</sub> = 0.244
Final <i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.054, <i>wR</i> <sub>2</sub> = 0.135	<i>R</i> <sub>1</sub> = 0.050, <i>wR</i> <sub>2</sub> = 0.129	<i>R</i> <sub>1</sub> = 0.136, <i>wR</i> <sub>2</sub> = 0.292
GOOF on <i>F</i> <sup>2</sup>	1.014	1.01	1.030
Largest diff. peak/hole [e.Å <sup>-3</sup> ]	0.69 and -0.61	0.34 and -0.57	1.04 and -0.50

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