

Experimental and Supplementary Information for

Diverse Reactivity of an Al(I)-centred Anion Towards Ketones

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1.1. General information

Except stated otherwise, all the experiments were conducted using standard Schlenk line and/or glovebox techniques under an inert atmosphere of argon. NMR spectra were recorded with an Agilent ProPulse spectrometer (^1H at 500 MHz, ^{13}C at 126 MHz). The spectra are referenced relative to residual protio solvent resonances. Elemental analyses were performed at Elemental Microanalysis Ltd., Okehampton, Devon, UK. Solvents were dried by passage through a commercially available solvent purification system and stored under argon in ampoules over 4 Å molecular sieves. C_6D_6 and d_8 -THF was purchased from Sigma-Aldrich, dried over a potassium mirror before distilling and storage over molecular sieves. $[\{\text{SiN}^{\text{Dipp}}\}\text{AlK}]_2$ (**8**) was prepared according to the reported procedure.¹ All other chemicals were purchased from Merck. Acetophenone, 2,4-dimethyl-3-pentanone and 2,2,4,4-tetramethyl-3-pentanone were degassed by three freeze-pump-thaw cycles and stored over 4 Å molecular sieves, while benzophenone was sublimed and stored in the glovebox under an argon atmosphere, before usage.

1.2. Synthetic Procedures

Synthesis of $K[\{\text{SiN}^{\text{Dipp}}\}\text{Al}-\kappa^2\text{-O}, \text{O}'\text{-}\{\text{OC}^{\text{Ph}}\}_2\text{C}^{\text{H}}(\text{CH}=\text{CHCH}=\text{CH})\text{C}=\text{C}^{\text{Ph}}\text{O}\}$ (11)

In a J Young's tube, [$\{\text{SiN}^{\text{Dipp}}\}\text{AlK}$]₂ (**8**, 28 mg, 0.025mmol), was dissolved in 0.4 mL of toluene before the addition of benzophenone (18mg, 0.10 mmol) to the bright yellow solution. The reaction mixture was then kept at 60 °C overnight to afford a colourless solution with bright yellow oil. THF was then added to the reaction mixture to afford a homogeneous bright yellow solution. Bright yellow single crystals suitable for X-ray crystallography were obtained by slow evaporation at room temperature. Yield 37 mg, 80%. Anal. Calcd. For C₇₂H₁₀₁AlKN₂Si₂O₆ (**11**(C₄H₈O)₂, 1211.86) C, 71.30; H, 8.39; N, 2.31 %. Found: C, 70.84; H, 8.06, N, 2.70 %. The recrystallised yellow crystalline solids were then collected and put under vacuum before re-dissolved in d₈-THF for NMR characterisation. ¹H NMR (500 MHz, 298K, THF-d₈) δ 7.78-7.69 (m, 2H, ArH), 7.50-7.45 (m, 2H, ArH), 7.04-6.99 (m, 2H, ArH), 6.99-6.91 (m, 4H, ArH), 6.91-6.84 (m, 1H, ArH), 6.84-6.80 (m, 1H, ArH), 6.78 – 6.73 (m, 1H, ArH), 6.72 – 6.67 (m, 2H, ArH), 6.66 – 6.58 (m, 2H, ArH), 6.53-6.44 (m, 3H, ArH), 6.43 – 6.17 (m, 1H, ArH), 6.04 (d, J = 9.4 Hz, 1H, AlOC^{Ph}=CCH=CH), 5.60 (dd, J = 9.8, 5.3 Hz, 1H, AlOC^{Ph2}-CHCH=CH), 5.21 (dd, J = 9.8, 5.3 Hz, 1H, AlOC^{Ph2}-CHCH=CH), 5.10 (dd, J = 9.4, 5.3 Hz, 1H, AlOC^{Ph}=CCH=CH), 4.71-4.60 (m, 1H, CHMe₂), 4.24 – 4.03 (m, 3H, CHMe₂), 3.69 (d, J = 5.3 Hz, 1H, AlOC_{Ph2}-CH), 1.48-1.39 (m, 1H, SiCH₂) 1.30 (d, J = 6.7 Hz, 3H, CHMe₂), 1.25-1.22 (m, 1H, SiCH₂), 1.19 (d, J = 6.7 Hz, 3H, CHMe₂), 1.15 (d, J = 6.7 Hz, 3H, CHMe₂), 1.13 (d, J = 6.7 Hz, 3H, CHMe₂), 1.02 (d, J = 6.7 Hz, 3H, CHMe₂), 0.82 – 0.75 (m, 1H, SiCH₂), 0.73 (s, 3H, SiMe₂), 0.66-0.58 (m, 1H, SiCH₂), 0.49 (d, J = 6.7 Hz, 3H, m, 1H, CHMe₂), 0.46 (s, 3H, SiMe₂), 0.41 (d, J = 6.7 Hz, 3H, CHMe₂), 0.04 (d, J = 6.7 Hz, 3H, CHMe₂), -0.45 (s, 3H, SiMe₂), -0.55 (s, 3H, SiMe₂). ¹³C {¹H} NMR (126 MHz, 298K, THF-d₈) δ 162.2 (Al-OC^{Ph}=C), 153.7 (ArC), 152.7 (ArC), 152.4 (ArC), 151.3 (ArC), 150.5 (ArC), 148.7 (ArC), 147.6 (ArC), 147.2 (ArC), 147.1 (ArC), 143.8 (ArC), 135.7 (ArC), 133.2 (ArC), 132.9 (ArC), 131.8 (ArC), 131.4 (AlOC^{Ph2}-CHCH=CH) , 131.0 (ArC), 130.7 (ArC), 129.2 (ArC), 129.0 (ArC), 126.9 (ArC), 126.9 (ArC), 126.7 (ArC), 126.2 (ArC), 124.8 (ArC), 124.7 (ArC), 123.9 (ArC), 123.8 (ArC), 123.6 (ArC), 123.5 (ArC), 122.7 (AlOC^{Ph2}-CHCH=CH), 121.7 (ArC), 121.6

(ArC), 112.0 (AlOC^{Ph}=CCH=CH), 109.9 (AlOC^{Ph}=C), 87.0 (AlOCPh₂), 54.0 (AlOC^{Ph}₂-CH), 27.9 (CHMe₂), 27.9 (CHMe₂), 27.8 (CHMe₂), 27.8 (CHMe₂), 27.7 (CHMe₂), 27.6 (CHMe₂), 27.4 (CHMe₂), 27.0 (CHMe₂), 26.6 (CHMe₂), 26.3 (CHMe₂), 25.4 (CHMe₂), 24.8(CHMe₂), 15.1 (SiCH₂), 13.1 (SiCH₂), 5.0 (SiMe₂), 4.4 (SiMe₂), 2.8 (SiMe₂), 1.9 (SiMe₂).

Figure S1. ^1H NMR (500MHz, 298K, d_8 -THF) spectrum of **11**. *toluene

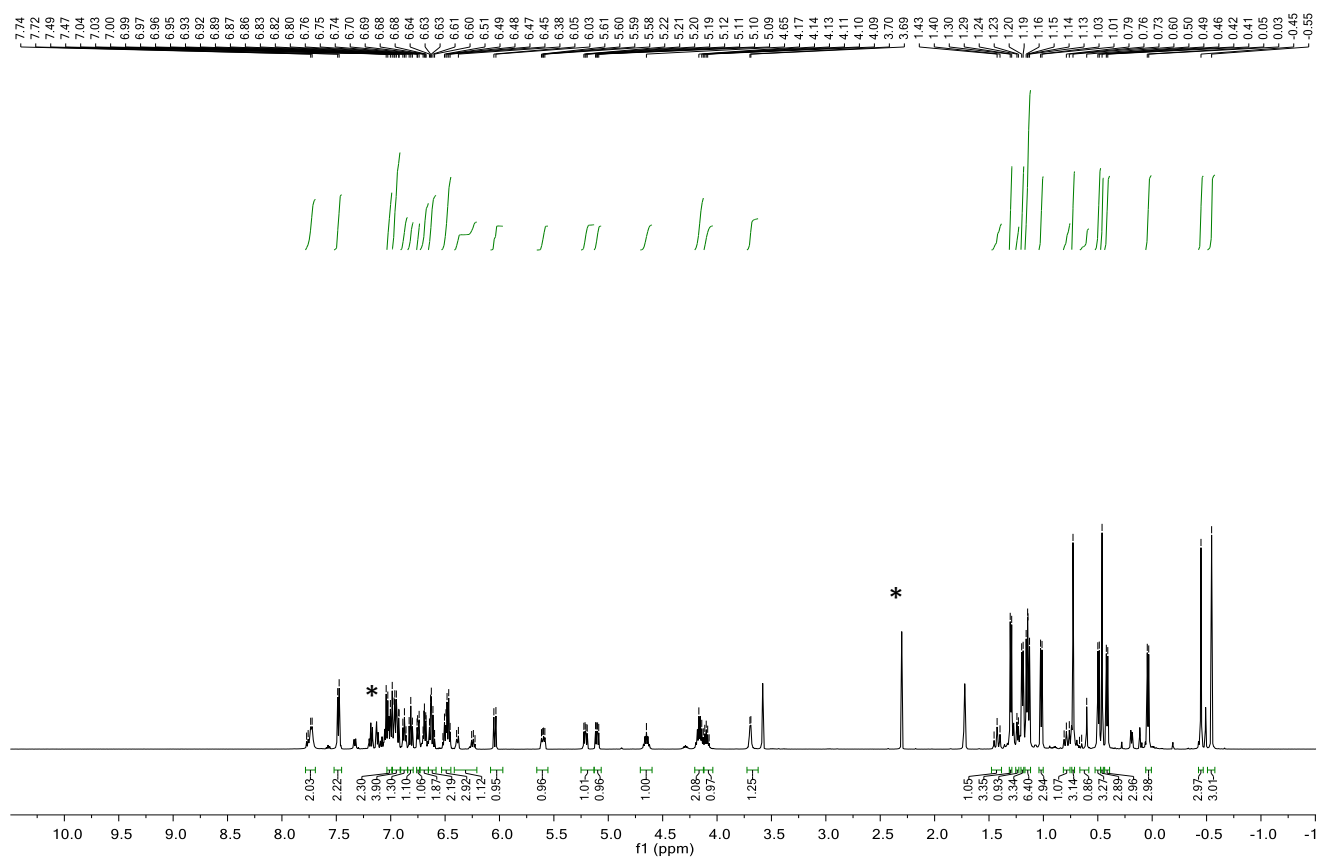


Figure S2. $^{13}\text{C}\{^1\text{H}\}$ NMR (126MHz, 298K, d_8 -THF) spectrum of **11**. *toluene

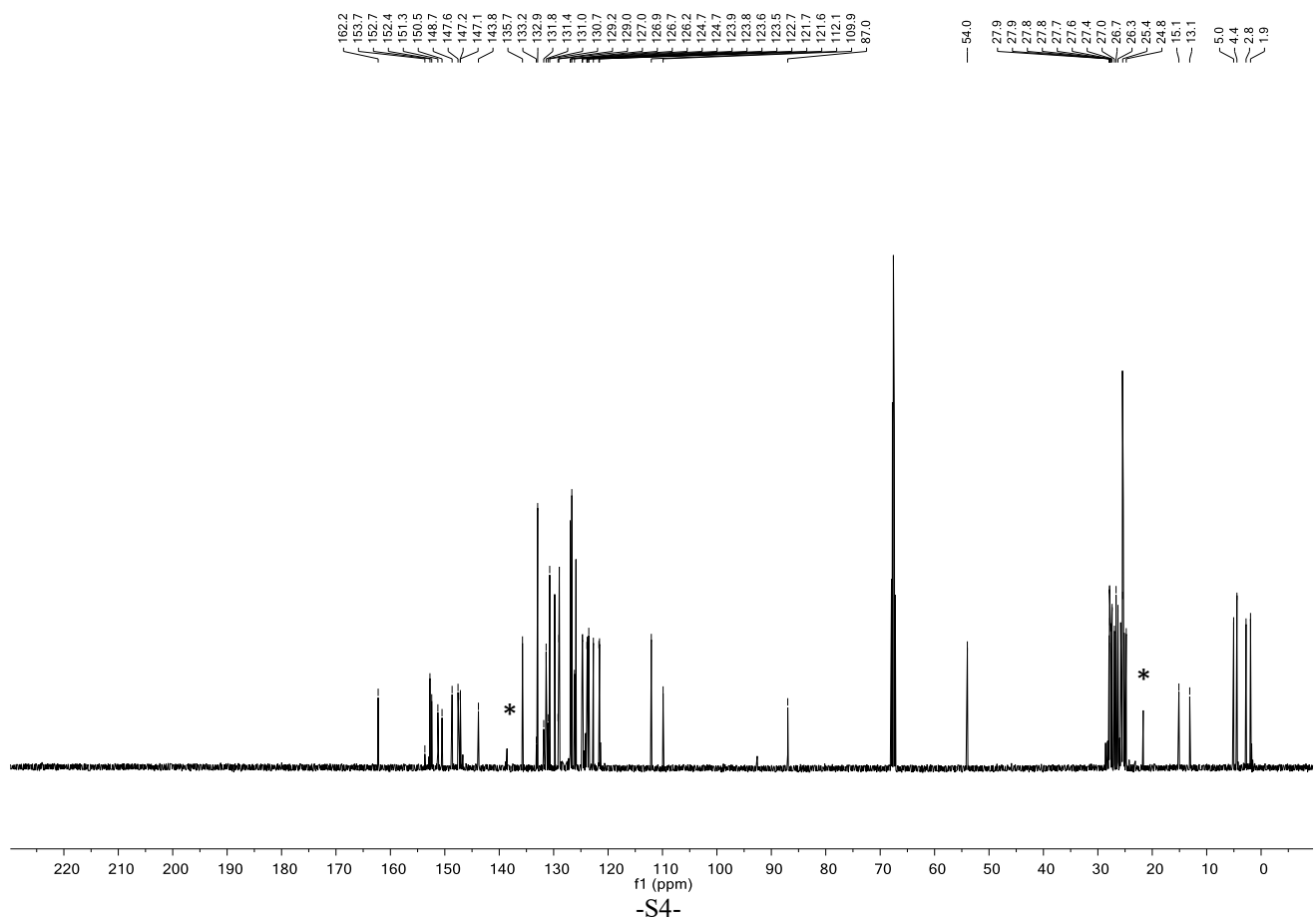


Figure S3. ^1H - ^1H COSY spectrum of **11**.

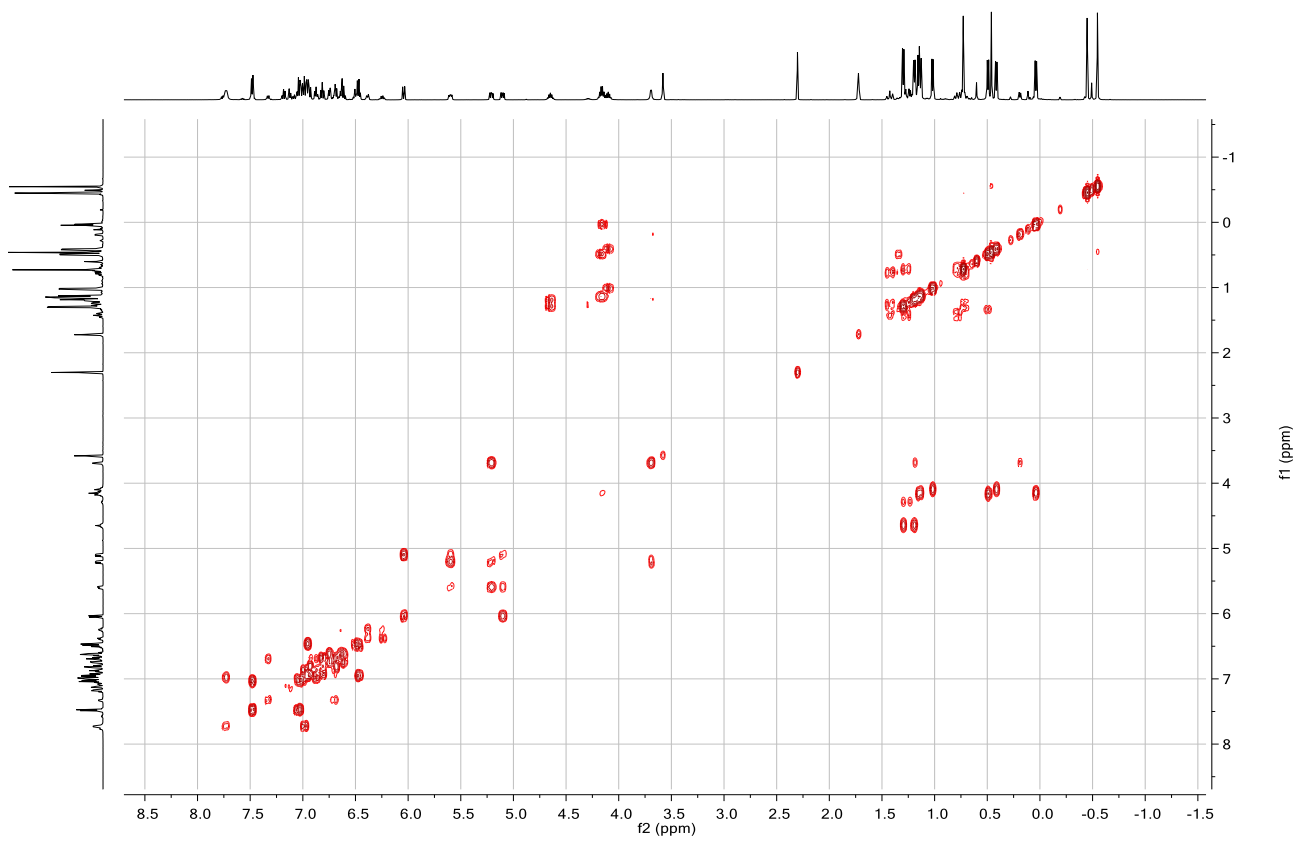


Figure S4. ^1H - ^{13}C HSQC spectrum of **11**.

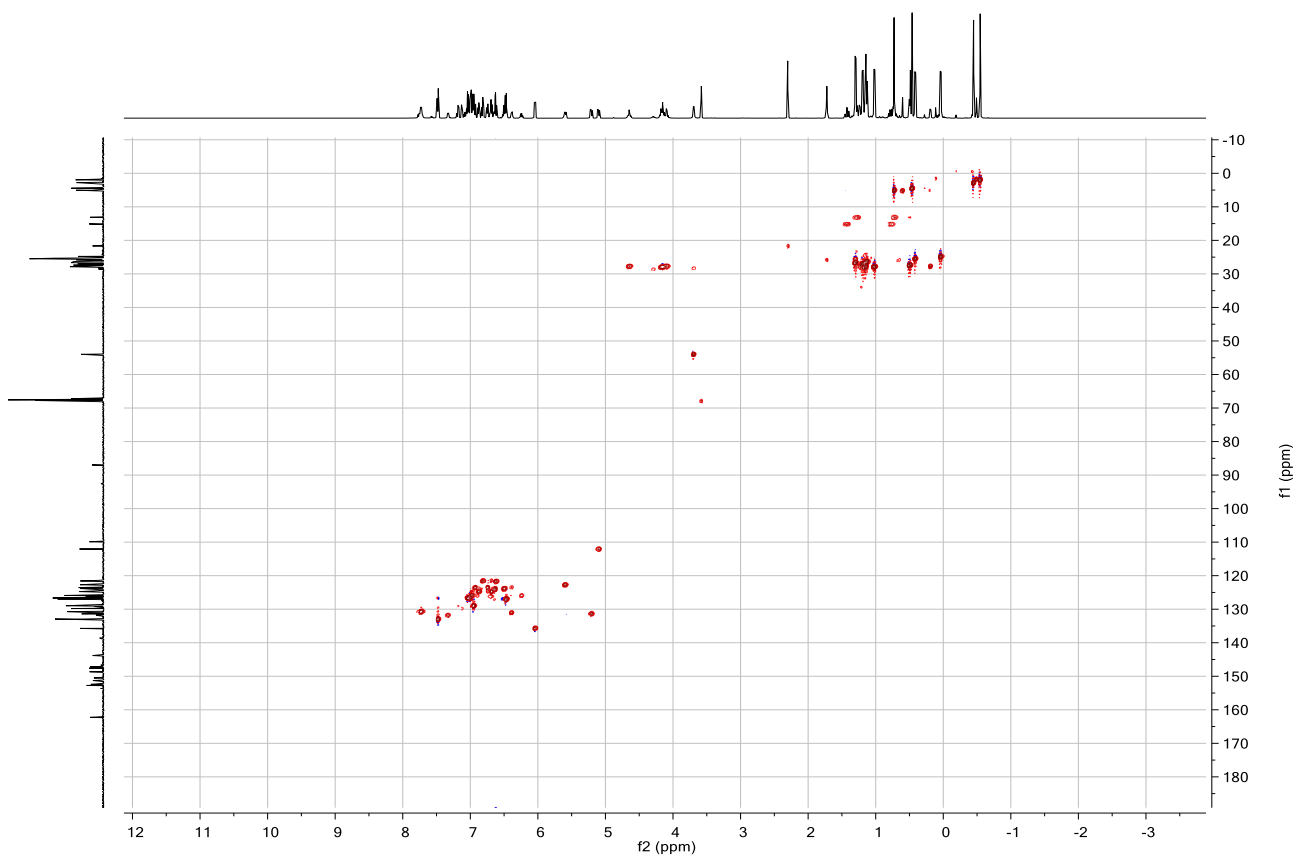
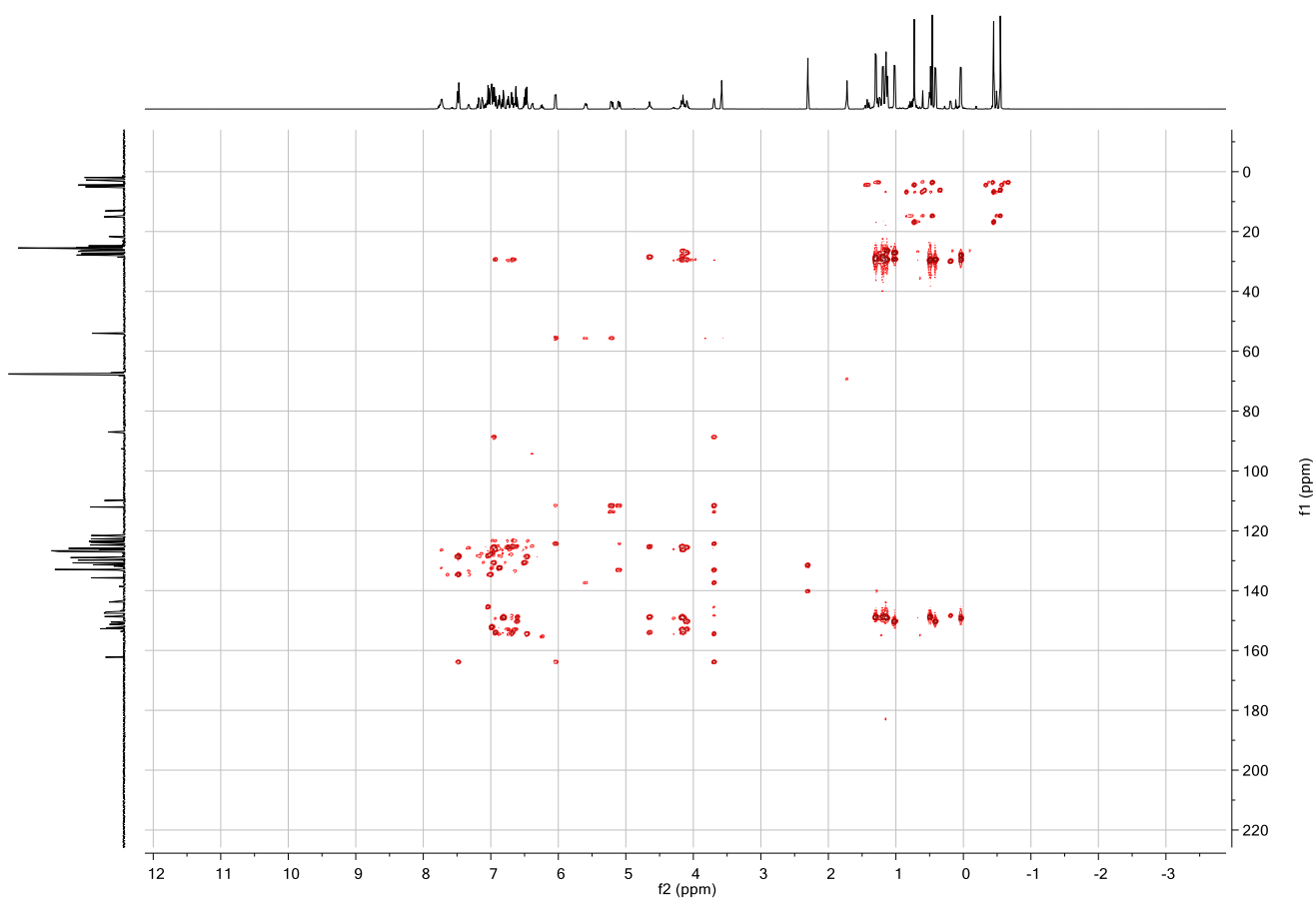


Figure S5. ^1H - ^{13}C HMBC spectrum of **11**.



*Synthesis of $K[\{\text{SiN}^{\text{Dipp}}\}\text{Al-}\kappa^2\text{-O,O'-(OCPHMe)}_2]$ (**12**)*

In a J Young's tube, $[\{\text{SiN}^{\text{Dipp}}\}\text{AlK}]_2$ (**8**, 28 mg, 0.025mmol) was dissolved in 0.4 mL of d^6 -benzene before the addition of acetophenone (11.5 μ L, 11.8mg, 0.10 mmol) *via* a micropipette. The resulting pale yellow reaction mixture was kept at 60 °C overnight to afford a colourless solution with colourless crystals. A single crystal suitable for X-ray crystallography was picked from the crystalline solid. The remaining colourless solids were then collected, washed with hexane (0.5mL x 2), and dried under vacuum to give **12** as a colourless powder. Yield 29 mg, 72%. Synthesis was also conducted in toluene with the same result. Yield 30 mg, 74%. Anal. Calcd. For $\text{C}_{53}\text{H}_{74}\text{AlKN}_2\text{Si}_2\text{O}_2$ (**12**. C_7H_8) C, 71.25; H, 8.35; N, 3.14 %. Found: C, 70.72; H, 8.25, N, 2.86 %. The powder was dissolved in THF- d_8 for NMR characterisation. ^1H NMR (500 MHz, 298K, THF- d_8) δ 7.85 – 7.15 (m, 2H, ArH of AlOCPH), 7.11 – 6.93 (d_{app} , 4H, *m*- C_6H_3), 6.92-6.77 (m, 8H, ArH of AlOCPH), 6.77-6.68 (t_{app} , 2H, *p*- C_6H_3), 4.19 (m, 4H, CHMe₂), 1.32 – 1.20 (m, 3H, AlOCMePh), 1.16 (d, J = 6.9 Hz, 6H, CHMe₂), 1.13 (d, J = 6.9 Hz, 6H, CHMe₂), 1.08 (d, J = 6.9 Hz, 6H, CHMe₂), 1.06-0.98 (m, 3H, AlOCMePh), 0.96 (s, 4H, SiCH₂), 0.86 (d, J = 6.9 Hz, 6H CHMe₂), 0.03 (s, 6H, SiMe₂), -0.04 (s, 6H, SiMe₂). $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, 298K, THF- d_8) δ 163.0 (ArC of AlOCMePh), 160.6 (ArC of AlOCMePh), 150.5 (ArC of AlOCMePh), 148.7 (*i*- C_6H_3), 147.9 (ArC of AlOCMePh), 143.6 (*o*- C_6H_3), 127.3 (ArC of AlOCMePh), 126.9 (ArC of AlOCMePh), 125.9 (*m*- C_6H_3), 123.4 (ArC of AlOCMePh), 123.3 (ArC of AlOCMePh), 121.5 (*p*- C_6H_3), 83.8 (AlOC), 28.0 (CHMe₂), 28.0 (CHMe₂), 26.7 (AlOCMePh), 26.1 (AlOCMePh), 25.8 (CHMe₂), 25.6 (CHMe₂), 24.2 (CHMe₂), 23.7 (CHMe₂), 15.9 (SiCH₂), 1.9 (SiMe₂), 1.6 (SiMe₂).

Figure S6. ^1H NMR (500MHz, 298 K, d_8 -THF) spectrum of **12**. *unidentified impurities, plausibly THF coordinated species

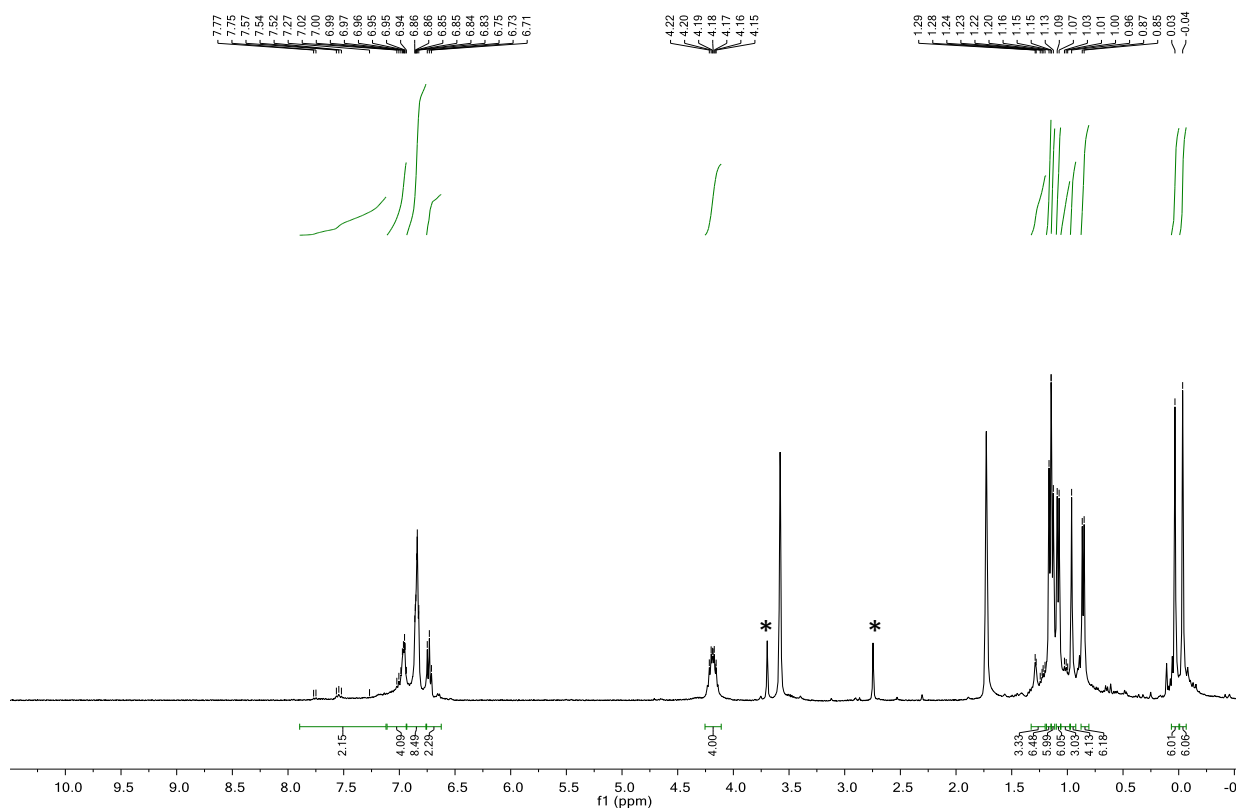


Figure S7. $^{13}\text{C}\{^1\text{H}\}$ NMR (126MHz, 298 K, d_8 -THF) spectrum of **12**.

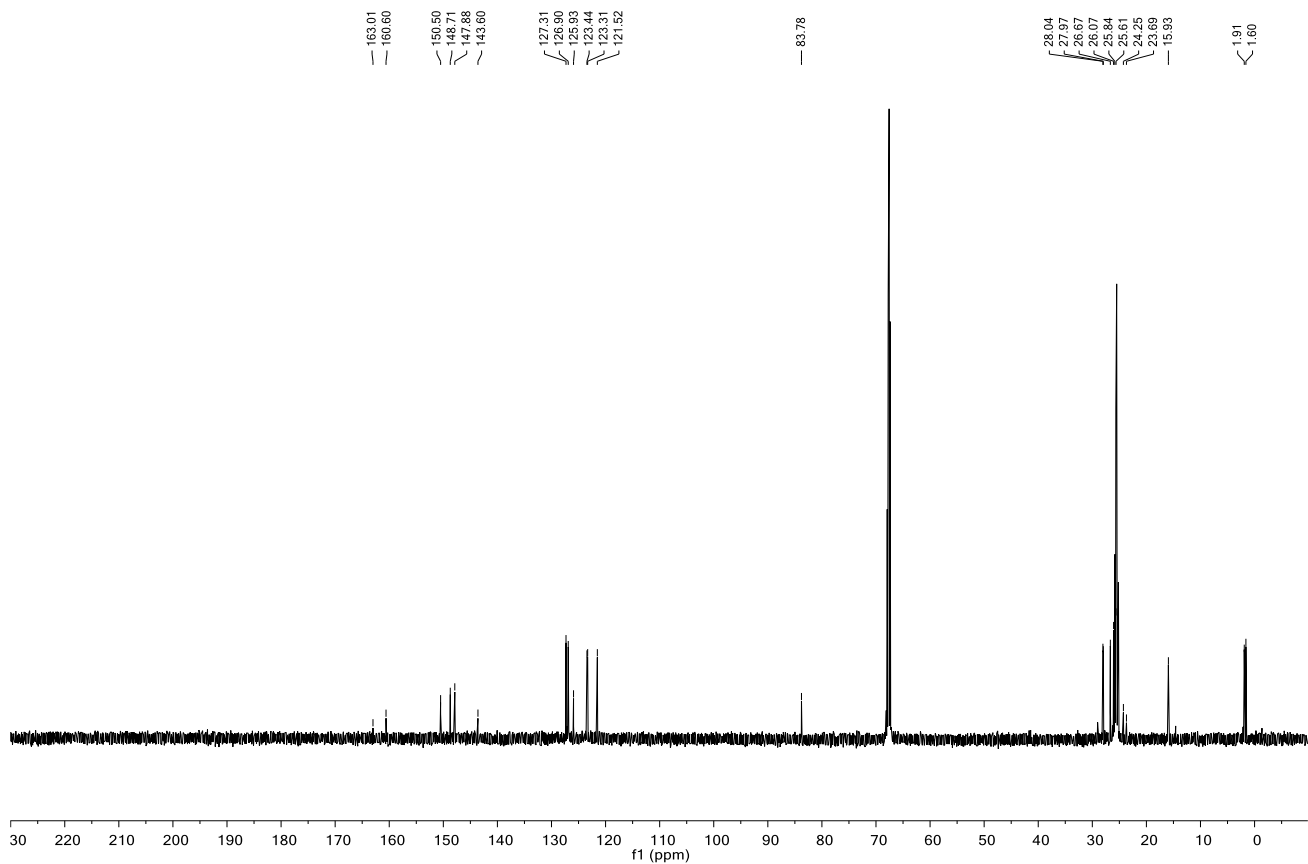


Figure S8. ^1H - ^{13}C HSQC spectrum of **12**.

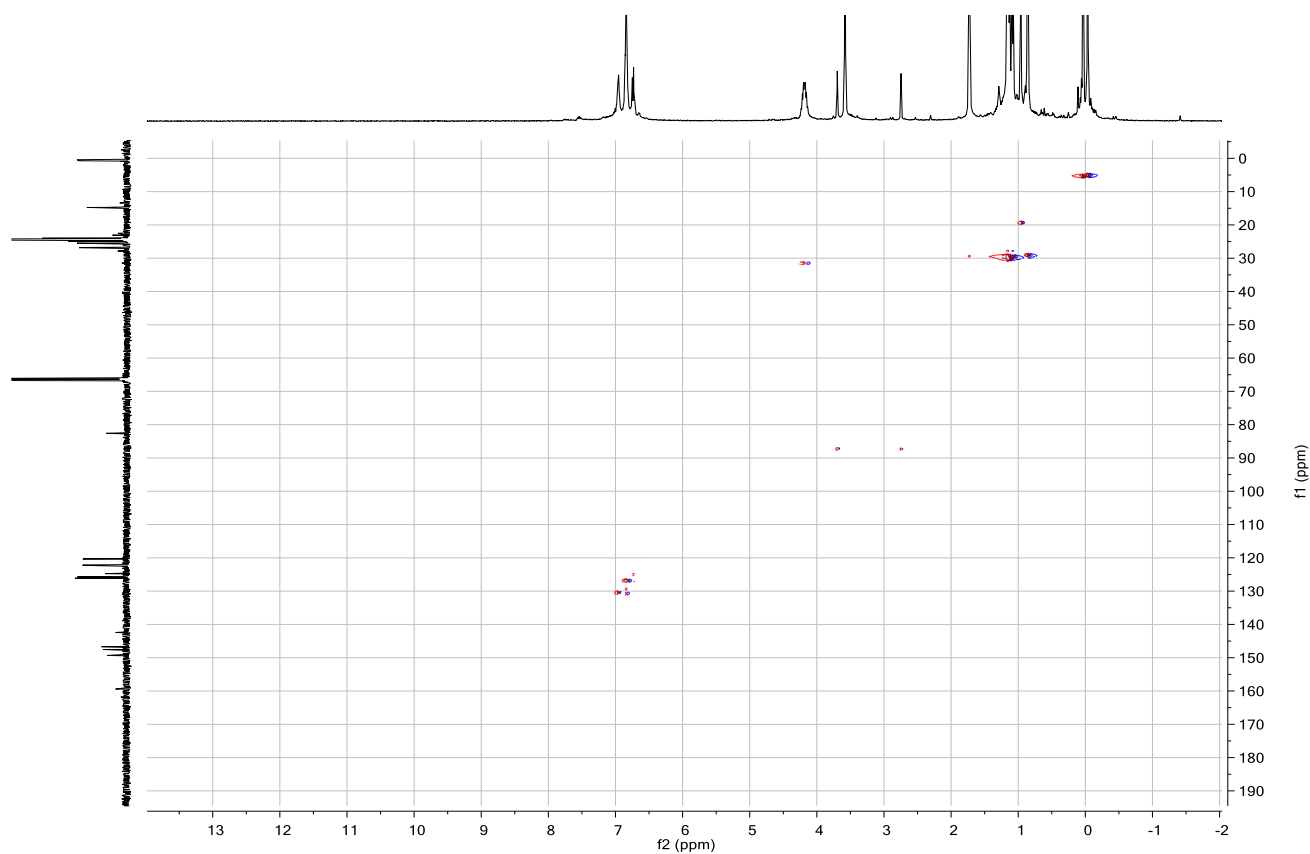
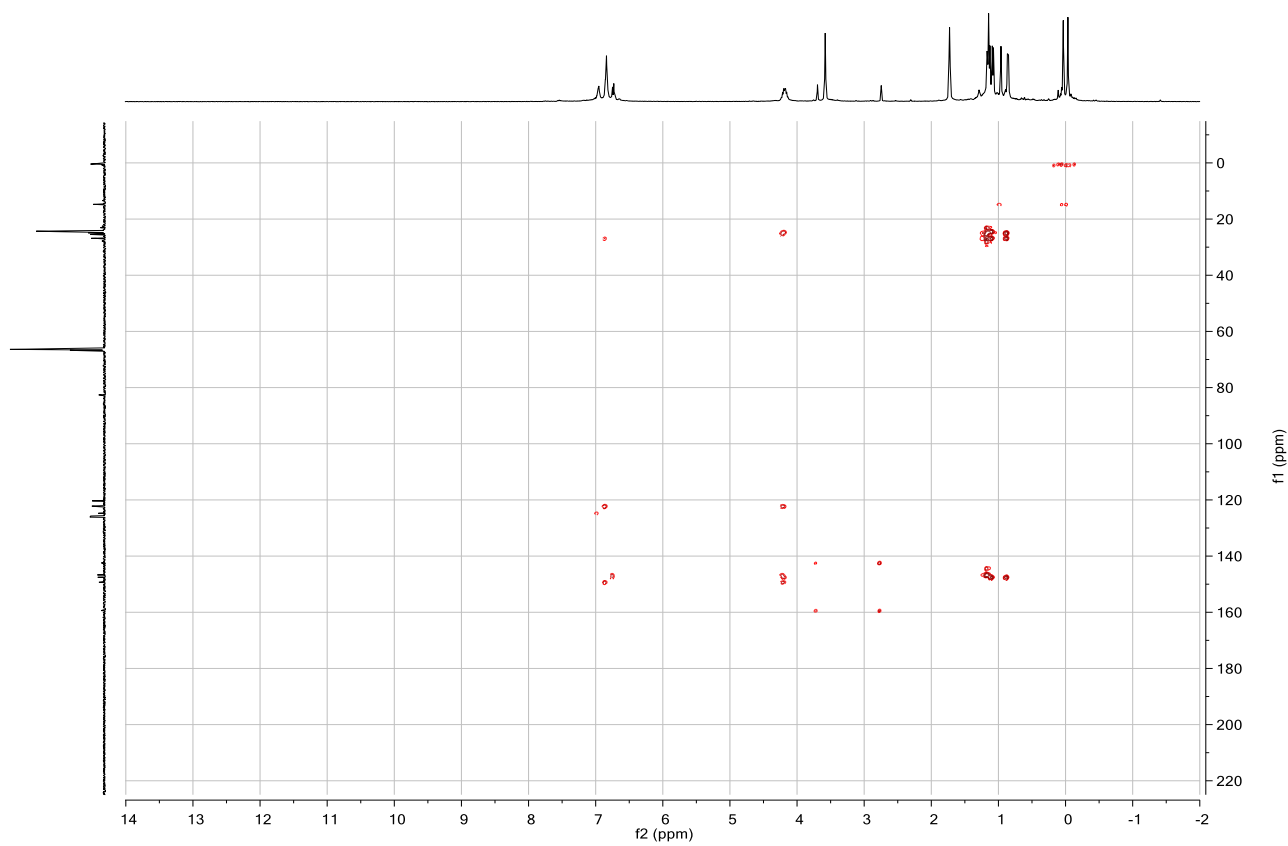


Figure S9. ^1H - ^{13}C HMBC spectrum of **12**.



*Synthesis of $K[\{\text{SiN}^{\text{Dipp}}\}\text{Al}(\text{H})\text{OC}(\text{iPr})=\text{CMe}_2]$ (**13**)*

In a J Young's tube, [$\{\text{SiN}^{\text{Dipp}}\}\text{AlK}$]₂ (**8**, 28 mg, 0.025mmol) was dissolved in 0.4 mL of toluene before the addition of 2,4-dimethyl-3-pentanone (7.1 μL , 5.7mg, 0.05 mmol) *via* a micropipette. The resulting bright yellow reaction mixture was kept at 60 °C overnight to afford a colourless solution with colourless crystals. A single crystal suitable for X-ray crystallography was picked from the crystalline solid. The remaining colourless solids were then collected, washed with hexane (0.5mL x 2), and dried under vacuum to give **13** as a colourless powder. Yield 30 mg, 89%. Anal. Calcd. For $\text{C}_{37}\text{H}_{64}\text{AlKN}_2\text{Si}_2\text{O}$ (**13**) C, 65.82; H, 9.55; N, 4.15 %. Found: C, 64.76; H, 8.93, N, 3.92 %. ¹H NMR (500 MHz, 298K, THF-d₈) δ 6.81 (d, J = 7.5 Hz, 4H, *m*-C₆H₃), 6.65 (t, J = 7.5 Hz, 2H, *p*-C₆H₃), 4.26 (sept, J = 6.5 Hz, 2H, CHMe₂ on N^{Dipp}), 4.20 (sept, J = 6.5 Hz, 2H, CHMe₂ on N^{Dipp}), 1.82 (sept, J = 7.2 Hz, 1H, OCCHMe₂), 1.33 (s^{app}, 6H, OCCMe₂), 1.17-1.14 (m, 12H, CHMe₂ on N^{Dipp}), 1.13 (d, J = 6.5 Hz, 6H, CHMe₂ on N^{Dipp}), 1.06 (d, J = 6.5 Hz, 6H, CHMe₂ on N^{Dipp}), 0.89 (s, br, 4H, SiCH₂), 0.40 (d, J = 7.2 Hz, 6H, OCCHMe₂), -0.05 (s, 6H, SiMe₂), -0.09 (s, 6H, SiMe₂). ¹H resonance correlated to AlH was not observed. ¹³C {¹H} NMR (126 MHz, 298K, THF-d₈) δ 155.7 (OC), 151.5 (*i*-C₆H₃), 148.8 (*o*-C₆H₃), 147.9 (*o*-C₆H₃), 123.3 (*m*-C₆H₃), 123.2 (*m*-C₆H₃), 121.2 (*p*-C₆H₃), 94.7 (OCCMe₂), 35.4 (OCCHMe₂), 29.0 (CHMe₂ on N^{Dipp}), 27.9 (CHMe₂ on N^{Dipp}), 27.8 (CHMe₂ on N^{Dipp}), 26.4 (CHMe₂ on N^{Dipp}), 26.0 (CHMe₂ on N^{Dipp}), 24.2 (CHMe₂ on N^{Dipp}), 21.2 (OCCHMe₂), 20.5 (OCCMe₂), 19.4 (OCCMe₂), 16.2 (SiCH₂), 2.4 (SiMe₂), -1.4 (SiMe₂).

Figure S10. ^1H NMR (500 MHz, 298 K, THF- d_8) spectrum of **13**. *silicone grease

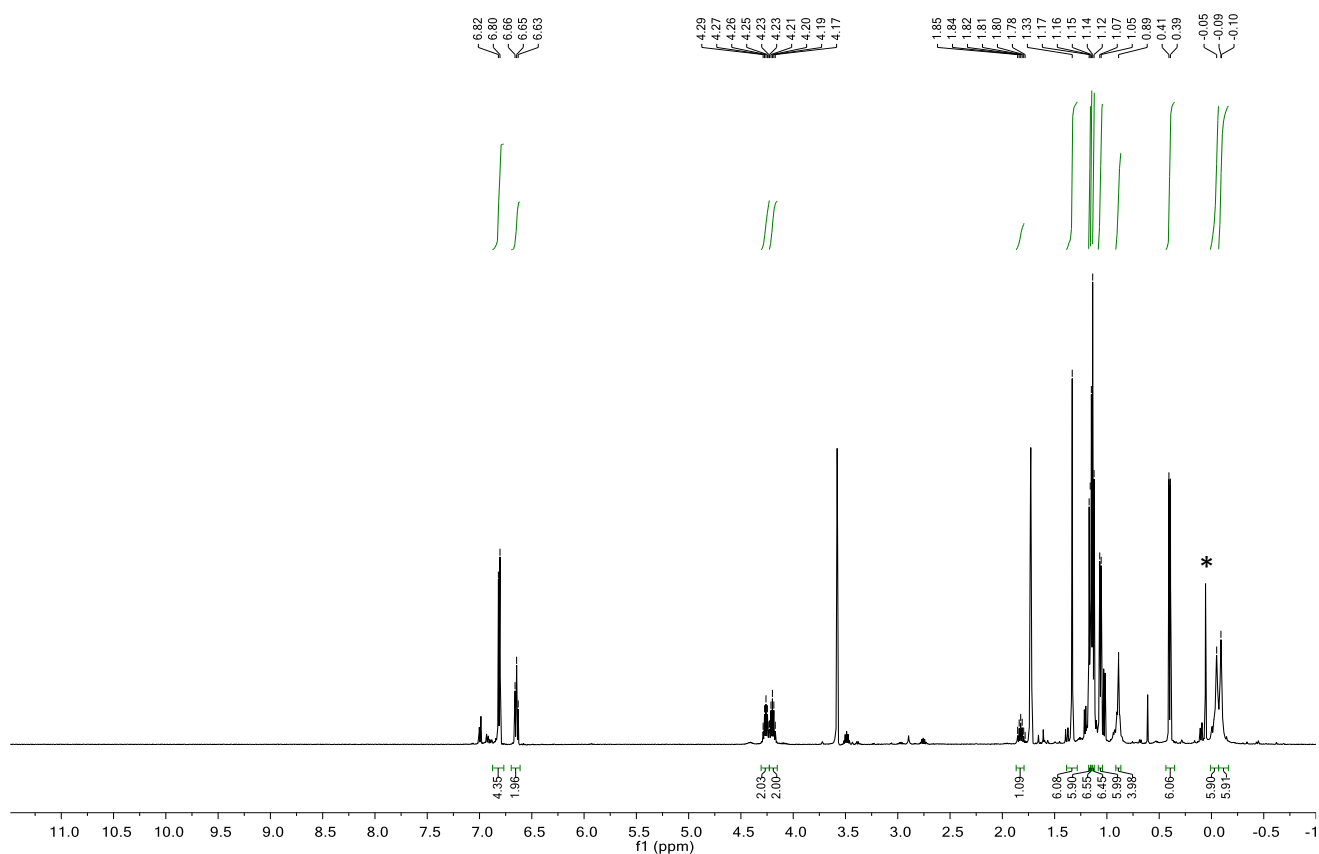


Figure S11. $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, 298 K, THF- d_8) spectrum of **13**. *silicone grease

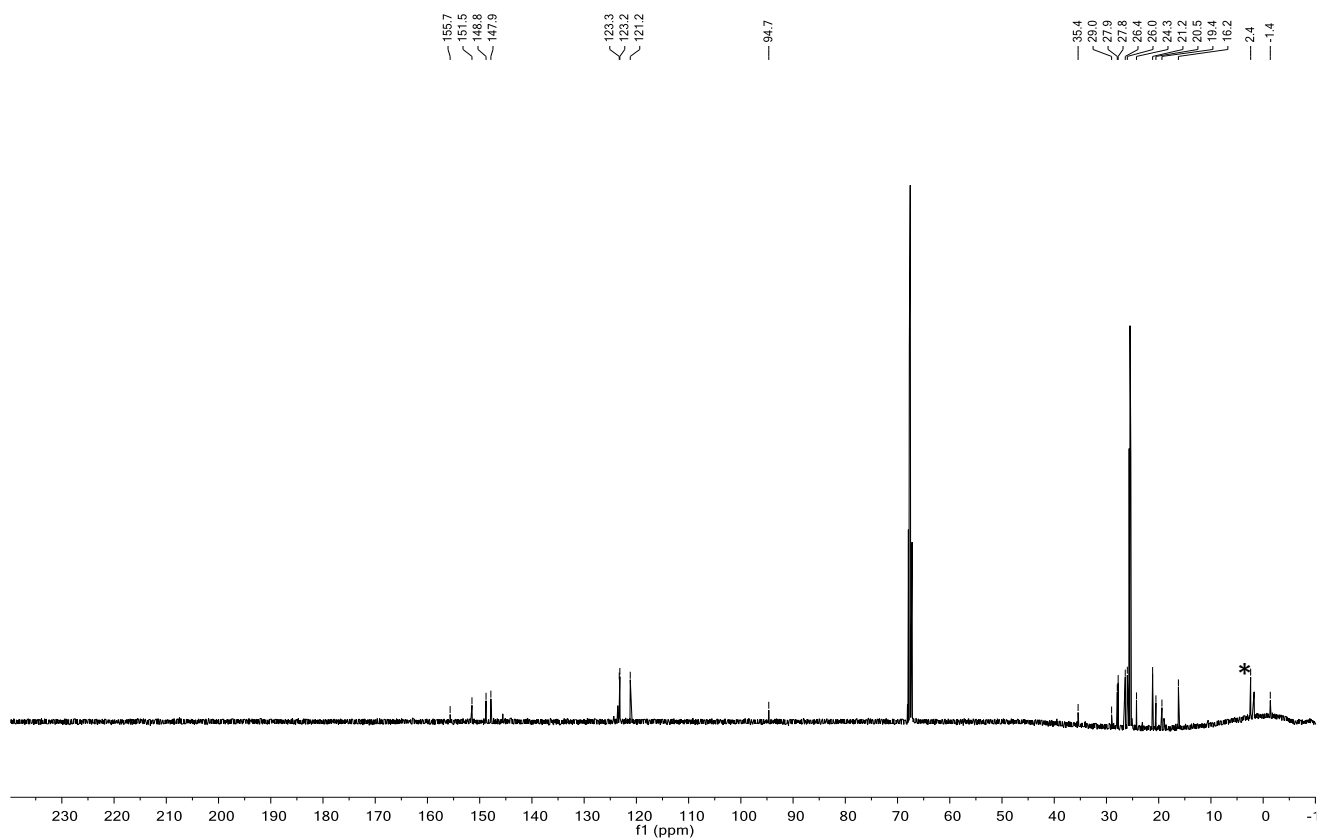


Figure S12. ^1H - ^1H COSY spectrum of **13**.

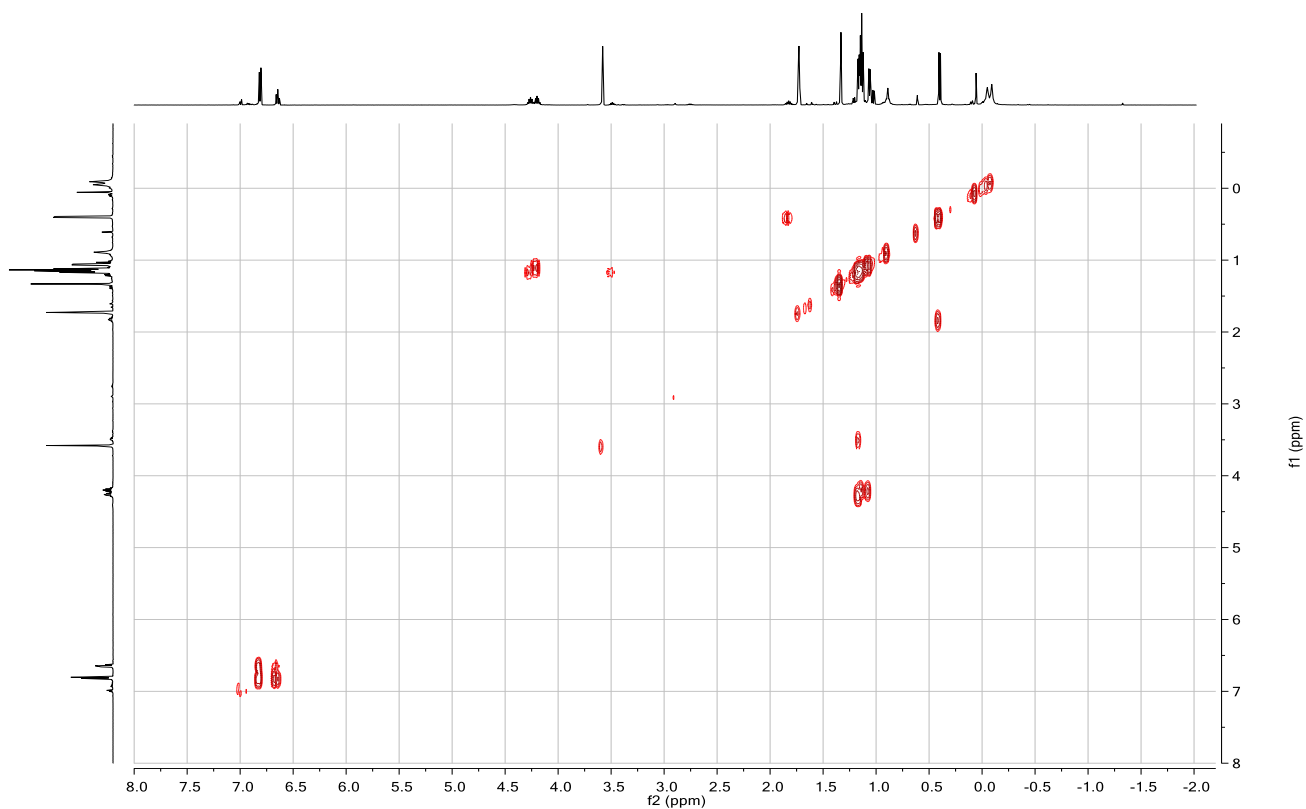


Figure S13. ^1H - ^{13}C HSQC spectrum of **13**.

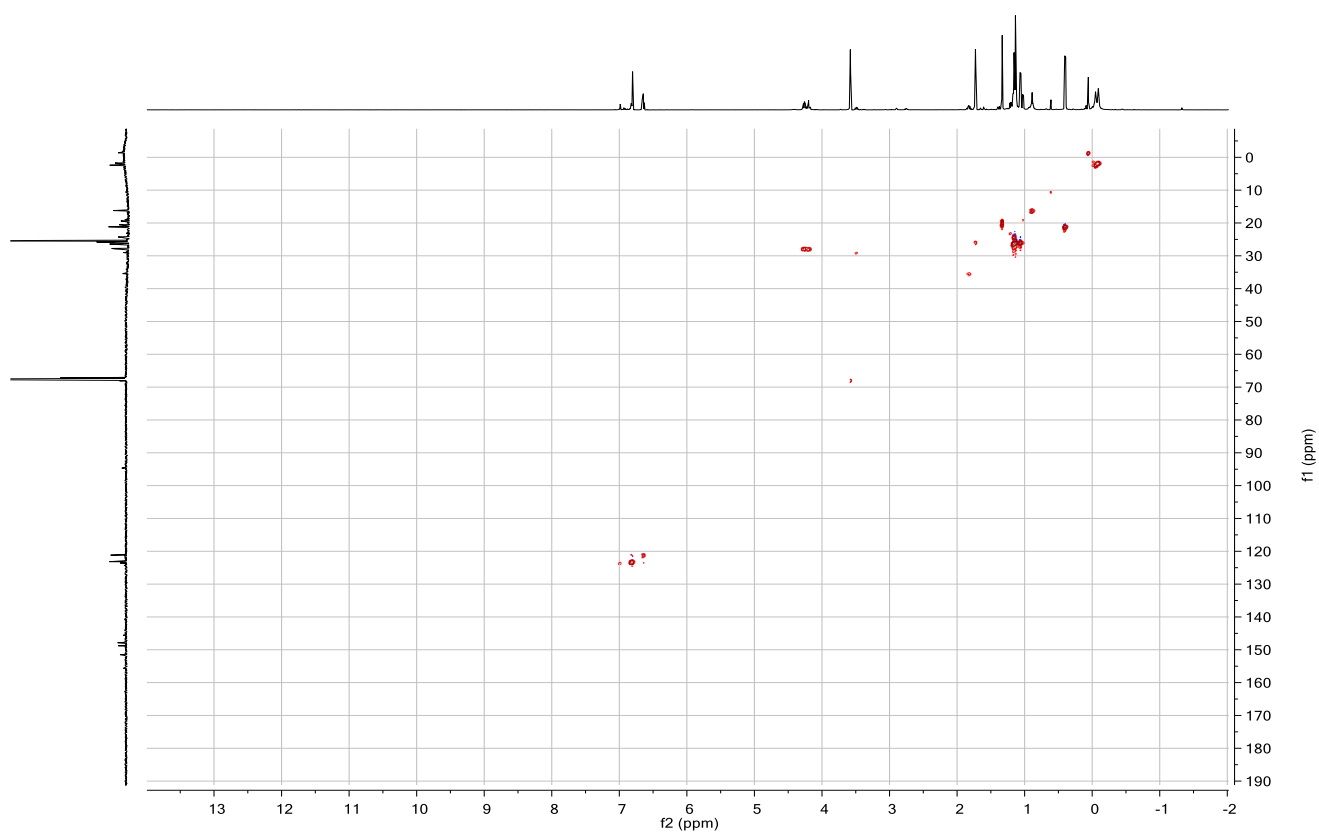
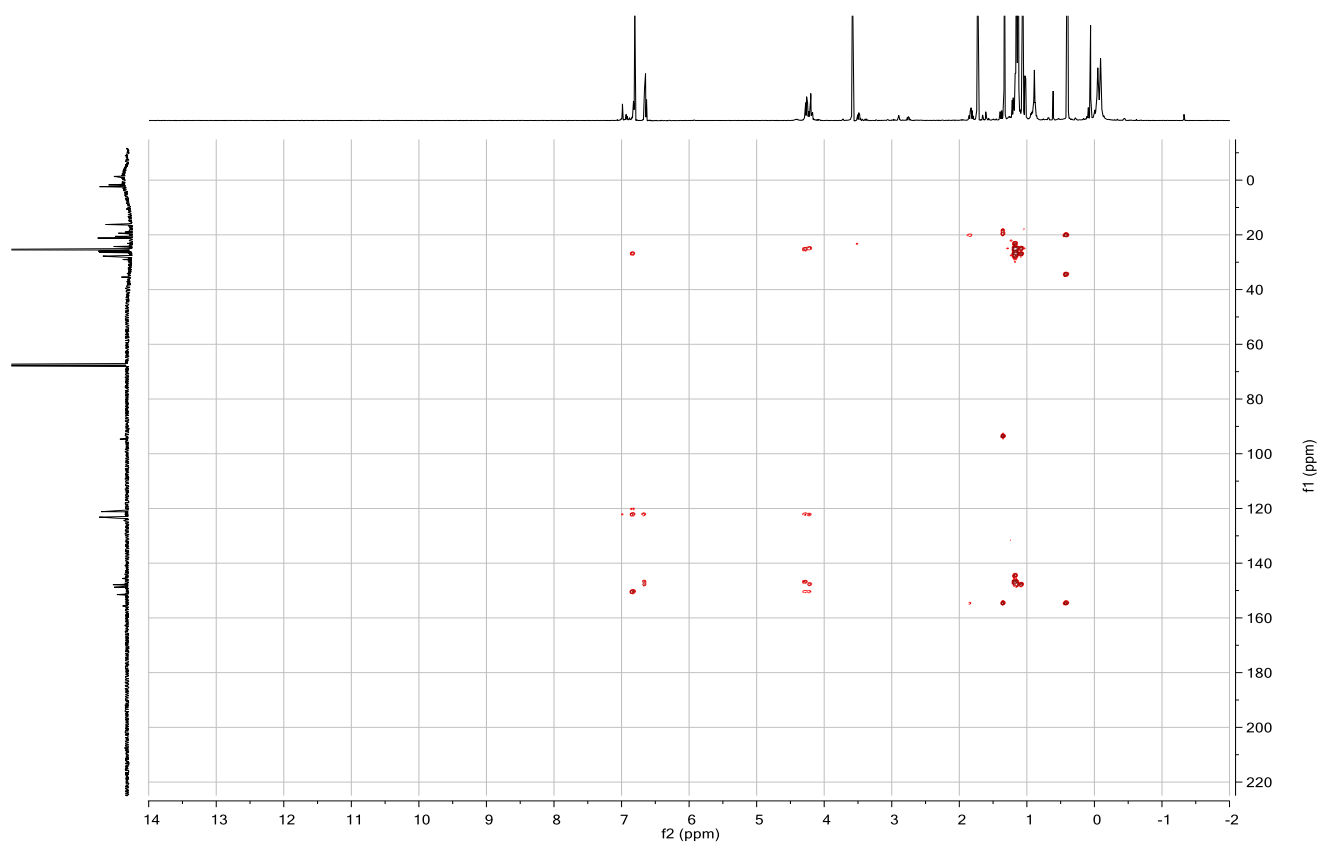


Figure S14. ^1H - ^{13}C HMBC spectrum of **13**.



*Synthesis of 14; Reaction of [$\{\text{SiN}^{\text{Dipp}}\}\text{AlK}\}_2$ (**8**) with 2,2,4,4-tetramethylpentanone*

In a J Young's tube, [$\{\text{SiN}^{\text{Dipp}}\}\text{AlK}\}_2$ (**8**, 28 mg, 0.025mmol) was dissolved in 0.4 mL of toluene before the addition of 2,4-dimethyl-3-pentanone (8.6 μL , 7.1mg, 0.05 mmol) *via* a micropipette. The resulting bright yellow reaction mixture was kept at 60 °C overnight to afford a colourless solution with colourless crystals. A single crystal suitable for X-ray crystallography was picked from the crystalline solid. The colourless solids were then collected, washed with hexane (0.5mL x 2), and dried under vacuum to give **14** as a colourless powder. Yield 28 mg, 83%. Anal. Calcd. For $\text{C}_{39}\text{H}_{68}\text{AlKN}_2\text{Si}_2\text{O}$ (**14**) C, 66.01; H, 9.73; N, 3.98 %. Found: C, 65.97; H, 9.76, N, 3.74 %. NMR characterisation was performed with a mixture of diastereomers of compound **14**. ^1H NMR (500 MHz, 298K, THF- d_8) δ 6.83 – 6.74 (m, 5H, ArH), 6.69 – 6.67 (m, 1H, ArH), 6.64 – 6.55 (m, 4H, , ArH), 6.48– 6.46 (m, 1H, ArH), 6.15 (t, $J = 7.3$ Hz, $p\text{-C}_6\text{H}_3$), 4.30 (sept, $J = 6.8$ Hz, 1H, CHMe₂), 4.18 (sept, $J = 6.8$ Hz, 1H, CHMe₂), 4.05 (sept, $J = 6.8$ Hz, 1H, CHMe₂), 3.97 (sept, $J = 6.8$ Hz, 2H, CHMe₂), 3.77 (sept, $J = 6.8$ Hz, 1H, CHMe₂), 3.63 (s, 1H, AlOCH), 3.19 (br, 1H, CHCH₂Al), 2.75 (s, 1H, AlOCH), 2.16 (t, $J = 14.3$ Hz, 1H, CHCH₂Al), 1.33 – 1.31 (m, 6H, CHMe₂), 1.28 (d, $J = 6.8$ Hz, 3H, CHMe₂), 1.25 (d, $J = 6.8$ Hz, 3H, CHMe₂), 1.21 (br, 2H, CHCH₂Al), 1.20 – 1.18 (m, 9H, CHMe₂), 1.17 (s br, 9H, CMe₃), 1.16 – 1.10 (m, 6H, CHMe₂), 1.10 (d, $J = 6.8$ Hz, 3H, CHMe₂), 1.07 (d, $J = 6.7$ Hz, 6H, CHMe₂), 1.04 (d, $J = 6.8$ Hz, 3H, CHMe₂), 1.02 – 1.00 (m, 2H, CHCH₂Al), 0.97 (s br, 9H, CMe₃), 0.91 – 0.59 (m, 8H, SiCH₂), 0.49 (s br, 9H, CMe₃), 0.45 (s, 3H, SiMe₂), 0.39 (s br, 9H, CMe₃), 0.36 (s, 3H, SiMe₂), 0.33 (s, 3H, SiMe₂), 0.29 (s, 3H, SiMe₂), 0.11 (s, 3H, SiMe₂), -0.45 (s, 3H, SiMe₂), -0.56 (s, 6H, SiMe₂). ^{13}C NMR (126 MHz, 298K, THF- d_8) δ 153.7 (4° ArC), 153.7 (4° ArC), 153.3 (4° ArC), 152.8 (4° ArC), 152.5 (4° ArC), 152.2 (4° ArC), 151.3 (4° ArC), 150.0 (4° ArC), 148.3 (4° ArC), 148.0 (4° ArC), 147.7 (4° ArC), 145.4 (4° ArC), 123.7 (ArCH), 123.6 (ArCH), 123.4 (ArCH), 123.1 (ArCH), 122.9 (ArCH), 122.7 (ArCH), 122.0 (ArCH), 121.4 (ArCH), 121.3 (ArCH), 121.1 (ArCH), 120.7 (ArCH), 119.7 (ArCH), 87.2 (AlOCH), 84.0 (AlOCH), 39.5 (CMe₃), 39.2 (CMe₃), 38.9 (CMe₃), 38.5 (CMe₃), 36.8 (CHCH₂Al), 34.6 (CHCH₂Al), 32.1 (AlCHCH₂), 31.4 (CMe₃), 31.4 (CMe₃), 31.3 (CMe₃), 30.8 (CMe₃), 29.9 (AlCHCH₂), 29.0 (CHMe₂), 29.0 (CHMe₂), 28.4 (CHMe₂), 28.2 (CHMe₂), 27.7 (CHMe₂), 27.6

(CHMe₂), 27.4 (CHMe₂), 27.3 (CHMe₂), 27.3 (CHMe₂), 27.1 (CHMe₂), 27.1 (CHMe₂), 27.0 (CHMe₂),
27.0 (CHMe₂), 26.9 (CHMe₂), 26.8 (CHMe₂), 26.7 (CHMe₂), 26.7 (CHMe₂), 26.3 (CHMe₂), 26.0
(CHMe₂), 15.5 (SiCH₂), 15.4 (SiCH₂), 15.3 (SiCH₂), 15.0 (SiCH₂), 6.1 (SiMe₂), 4.8 (SiMe₂), 4.7
(SiMe₂), 3.7 (SiMe₂), 2.1 (SiMe₂), 1.0 (SiMe₂), 0.6 (SiMe₂), -0.6 (SiMe₂).

Figure S15. ^1H NMR (500 MHz, 298 K, THF- d_8) spectrum of **14**.

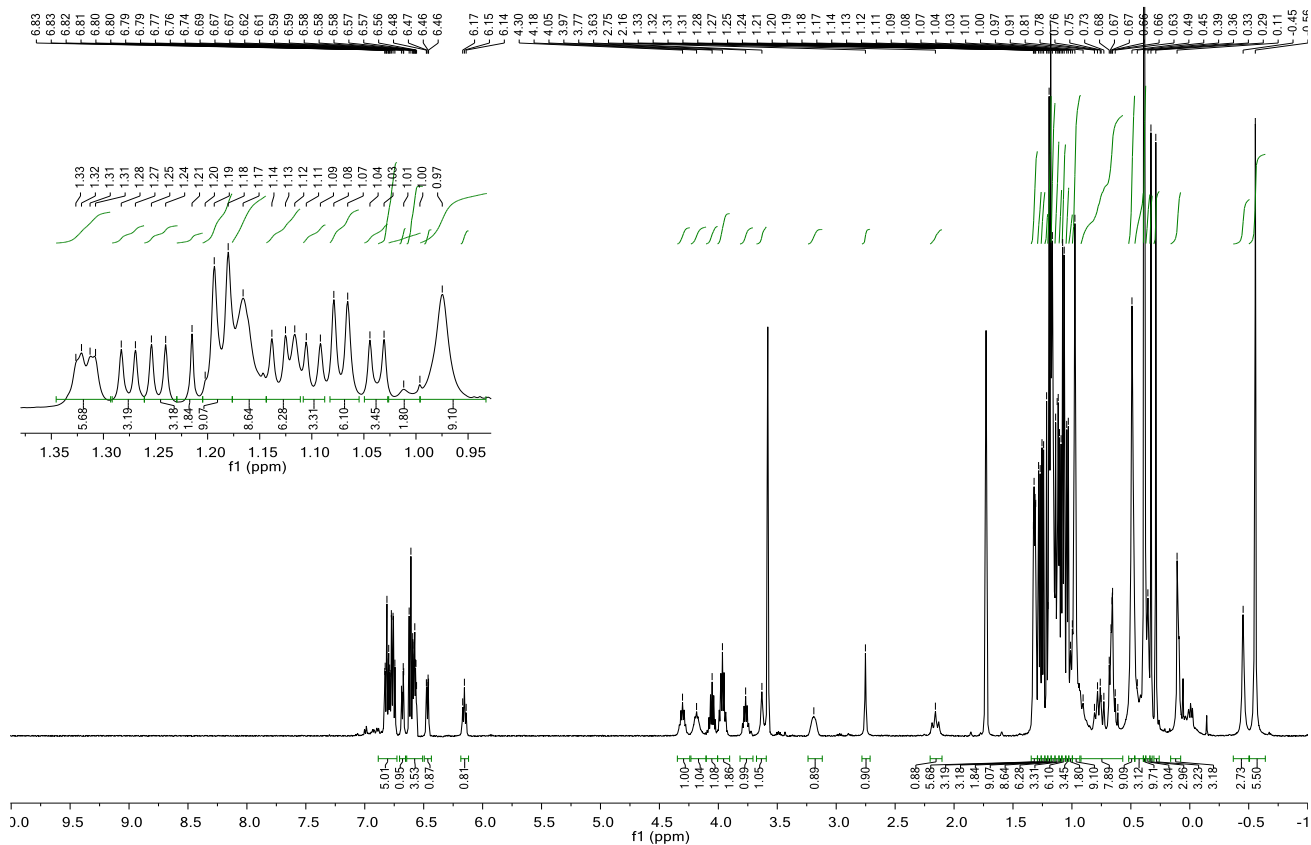


Figure S16. $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, 298 K, THF- d_8) spectrum of **14**.

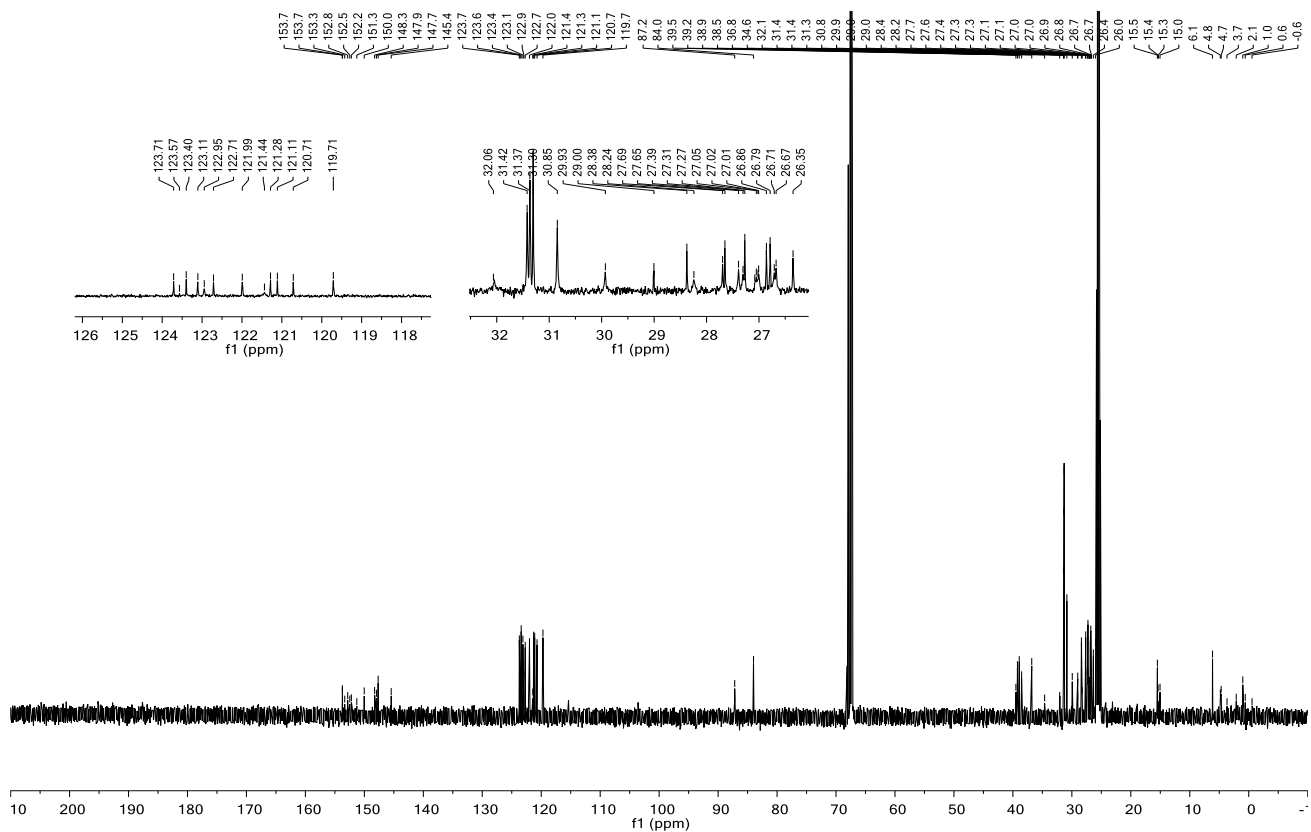


Figure S17. ^1H - ^1H COSY spectrum of **14**.

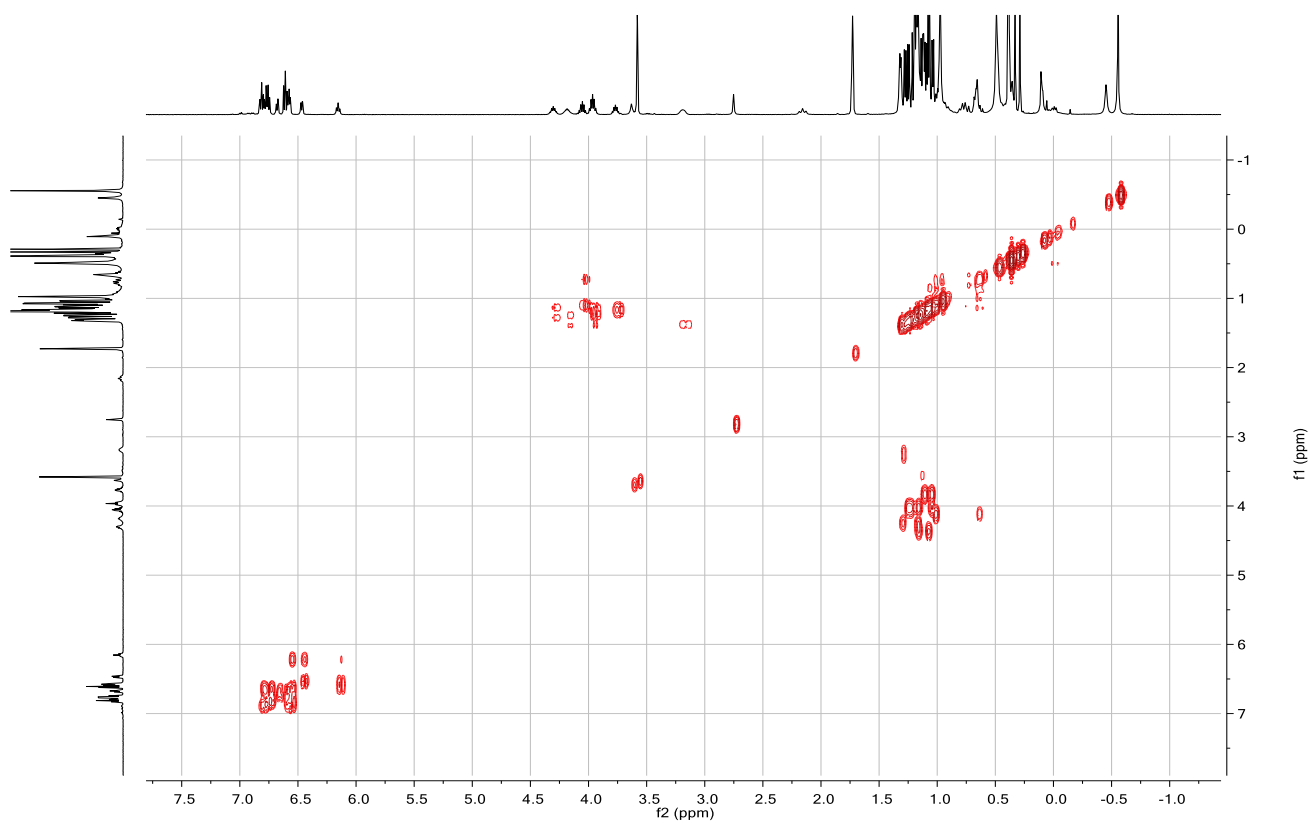


Figure S18. ^1H - ^{13}C HSQC spectrum of **14**.

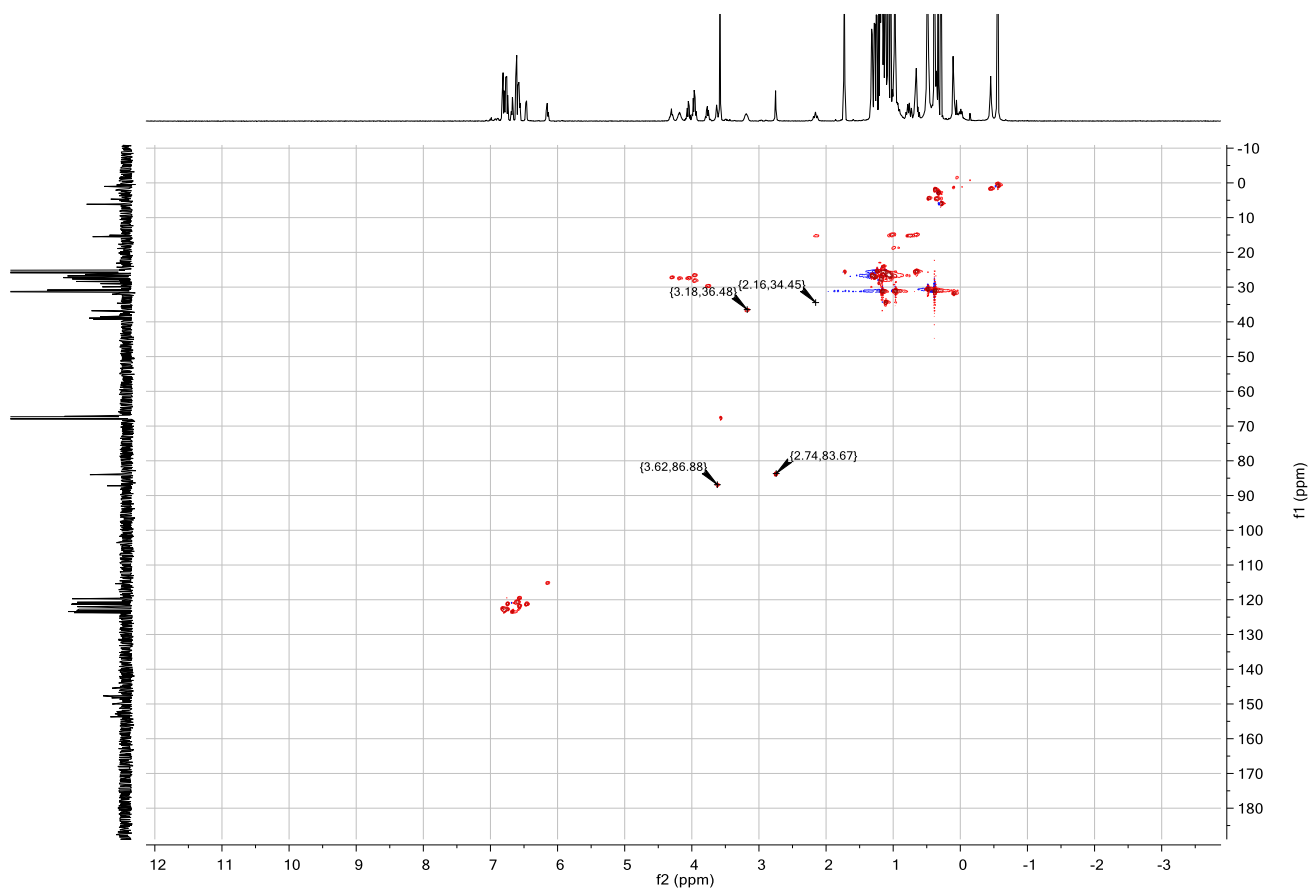
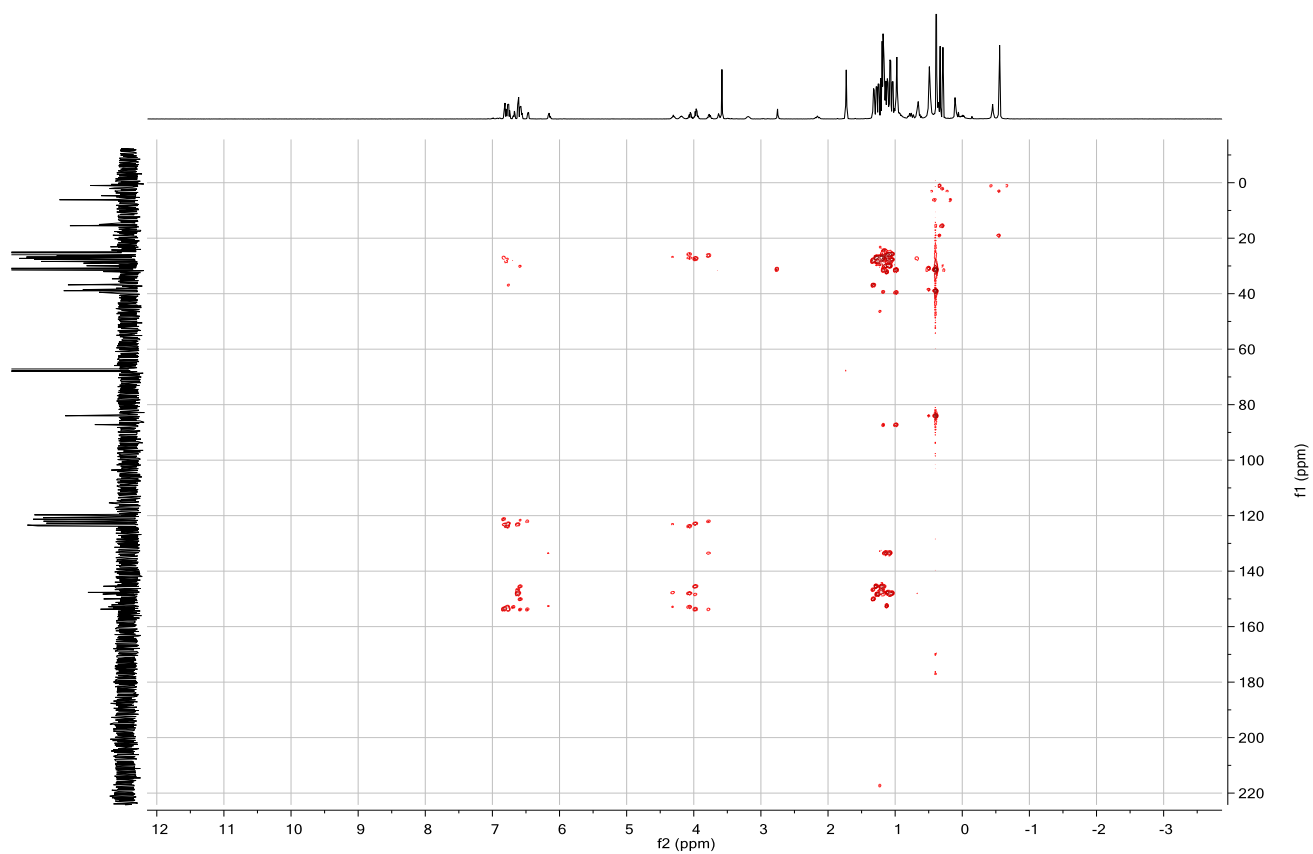


Figure S19. ^1H - ^{13}C HMBC spectrum of **14**.



1.3. Single Crystal X-ray Diffraction Analysis

Single Crystal X-ray diffraction data for compounds **11** – **14** were collected on a SuperNova, EosS2 diffractometer using CuK α ($\lambda = 1.54184 \text{ \AA}$) radiation throughout. The crystals were maintained at 150 K during data collection. Using Olex2,² the structures were solved with the olex2.solve³ structure solution program or ShelXT and refined with the ShelXL⁴ refinement package using Least-Squares minimization.

The asymmetric unit in the structure of **12** is a monomer, which contributes to the formation of 1-D polymers in the gross structure.

Similarly, in **13**, the asymmetric unit also comprises a monomer (which gives rise to 1-D polymers in the gross structure) plus a molecule of benzene with half site-occupancy. The latter straddles a crystallographic inversion centre, which necessarily means that it is disordered with itself. As such, this moiety was refined as a rigid hexagon and with the inclusion of ADP restraints. The hydride in the main feature was located and refined without restraints. A residual electron density maximum, proximate to K1, may indicate a modicum of disorder at this centre. However, efforts to model same did not improve convergence and indicated that, at best, that any alkali metal disorder was < 5%. Given the paucity of evidence for such disorder credibility, partitioning K1 between two sites was abandoned.

The asymmetric unit in the structure of **14** corresponds to one quarter of a tetramer. Disorder prevailed in two regions. In particular, the methyl groups which form part of the tert-butyl functionalities based on C32 and C36 were each treated for an 80:20 site-occupancy split while the Dipp moiety attached to N1 was modelled to take account of a 2-component disorder in a 55:45 ratio. All hydrogens were included at calculated positions, but those attached at C8, C18, C30, C48, C57 and C69 were refined with free U_{iso} values as a measure of credibility with which to assess any interactions with the potassium centres present. Distance and ADP restraints were employed, on merit, in disordered regions to assist convergence.

Table S1: Crystal data and structure refinement for compounds **11** – **14**.

Compound	11	12	13	13
Empirical formula	C ₆₄ H ₈₆ AlKN ₂ O ₄ Si ₂	C ₄₆ H ₆₆ AlKN ₂ O ₂ Si ₂	C ₄₀ H ₆₇ AlKN ₂ OSi ₂	C ₃₉ H ₆₈ AlKN ₂ OSi ₂
Formula weight	1069.60	801.26	714.21	703.21
Crystal system	monoclinic	orthorhombic	orthorhombic	monoclinic
Space group	<i>Cc</i>	<i>Pbca</i>	<i>Pbcn</i>	<i>C2/c</i>
<i>a</i> /Å	21.7511(4)	22.4043(4)	20.3555(1)	44.6873(10)
<i>b</i> /Å	13.3392(2)	17.8222(3)	23.0087(1)	12.4327(1)
<i>c</i> /Å	20.8348(3)	22.5974(5)	18.3408(1)	37.5655(8)
α /°	90	90	90	90
β /°	101.877(2)	90	90	127.202(3)
γ /°	90	90	90	90
Volume/Å ³	5915.64(17)	9023.0(3)	8589.98(7)	16623.8(7)
<i>Z</i>	4	8	8	16
ρ_{calc} g/cm ³	1.201	1.180	1.105	1.124
μ /mm ⁻¹	1.686	2.012	2.035	2.094
<i>F</i> (000)	2304.0	3456.0	3112.0	6144.0
Crystal size/mm ³	0.102 × 0.071 × 0.037	0.137 × 0.028 × 0.024	0.209 × 0.163 × 0.104	0.112 × 0.068 × 0.057
2 θ range /°	7.822 to 144.238	7.448 to 146.122	7.54 to 145.934	7.522 to 146.568
Index ranges	-26 ≤ <i>h</i> ≤ 26, -16 ≤ <i>k</i> ≤ 12, -21 ≤ <i>l</i> ≤ 25	-27 ≤ <i>h</i> ≤ 24, -18 ≤ <i>k</i> ≤ 21, -27 ≤ <i>l</i> ≤ 27	-24 ≤ <i>h</i> ≤ 25, -28 ≤ <i>k</i> ≤ 23, -22 ≤ <i>l</i> ≤ 22	-53 ≤ <i>h</i> ≤ 55, -15 ≤ <i>k</i> ≤ 15, -46 ≤ <i>l</i> ≤ 46
Reflections collected	15239	30723	112622	156548
Independent reflections	7932, 0.0209	8870, 0.0464	8574, 0.0487	16566, 0.0535
Data/restraints/parameters	7932/2/683	8870/0/512	8574/42/470	16566/499/1030
Goodness-of-fit on <i>F</i> ²	1.035	1.015	1.022	1.019
Final <i>R</i> indexes [<i>I</i> ≥ 2 σ (<i>I</i>)]	0.0281, 0.0720	0.0386, 0.0875	0.0443, 0.1260	0.0414, 0.1008
Final <i>R</i> indexes [all data]	0.0295, 0.0731	0.0571, 0.0959	0.0471, 0.1289	0.0512, 0.1066
Largest diff. peak/hole (e Å ⁻³)	0.24/-0.21	0.31/-0.25	0.97/-0.56	0.60/-0.46
Flack parameter	0.027(7)	–	–	–

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