Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

to the paper

Facile C–H Bond Activation on a Transient Gallium Imide

by

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Synthetic methods

General: All manipulations were performed using standard inert atmosphere (N_2 gas) glovebox and Schlenk techniques. Toluene was dried using a Grubbs-type solvent purification system. d_6-Benzene was pre-dried and distilled from K/Na alloy and stored in a glass vessel in the glovebox. NMR spectra were obtained with a Bruker AVANCE III HD 400 and 600 MHz spectrometers (^{1}H, 400 and 600 MHz; ^{13}C, 101 and 151 MHz) at room temperature, unless stated otherwise, then processed and analyzed with MestReNova software (v10.0.2-15465). Elemental analyses were obtained by the analytical laboratories of University of Toronto. All reagents were purchased from Sigma Aldrich or Alfa Aesar and used as received. Compound NacNacGa was prepared according to a literature procedure.[1]

Preparation of NacNacGa(py)(NHSiMe$_3$) (2)

To a solution of NacNacGa (0.063 g, 0.13 mmol) in toluene was added pyridine (0.010 mL, 0.13 mmol) and the mixture was treated with N_3SiMe_3 (0.015 mL, 0.13 mmol). The reaction was allowed to stir for 10 min at room temperature. The volatiles were removed and NacNacGa(py)(NHSiMe$_3$) was obtained in quantitative yield. Colorless crystals of **2** were obtained from a toluene solution at -30^{0}C.

^{1}H NMR (400 MHz, C_6D_6) δ 8.65 (m, 1H C_5H_4N), 7.92 (d, 1H, C_5H_4N), 7.11 (m, 4H, C_6H_3), 7.08 (m, 1H, C_5H_4N) 7.02 (m, 2H, C_6H_3), 6.69 (m, 1H, C_5H_4N), 4.85 (s, 1H, CH), 3.59 (hept, 2H, $CH(CH_3)_2$ $^3J_{H\text{-}H}$ = 6.8 Hz), 3.05 (hept, 2H, $CH(CH_3)_2$ $^3J_{H\text{-}H}$ = 6.7 Hz), 1.62 (s, 6H, $NCCH_3$), 1.50 (d, 6H, $CH(CH_3)_2$, $^3J_{H\text{-}H}$ = 6.8 Hz),), 1.16 (d, 6H, $CH(CH_3)_2$, $^3J_{H\text{-}H}$ = 6.8 Hz), 1.05 (d, 6H, $CH(CH_3)_2$, $^3J_{H\text{-}H}$ = 6.7 Hz), 0.52 (d, 6H, $CH(CH_3)_2$, $^3J_{H\text{-}H}$ = 6.7 Hz), 0.04 (s, 9H, $Si(CH_3)_3$), -0.58 (s, 1H, NH).

^{13}C{1H} NMR (101 MHz, C_6D_6) δ 169.36 ($NCCH_3$), 145.0, 143.6, 141.6, 127.1, 124.72, 124.01 (C_6H_3), 179.5, 149.52, 132.9, 132.2, 121.81 (C_5H_4N), 96.6 (CH), 29.0, 28.1 ($CH(CH_3)_2$), 25.0, 24.7, 24.5, 24.4 ($CH(CH_3)_2$), 23.9 ($NCCH_3$), 3.6 ($Si(CH_3)_3$).

Elemental analysis: Calculated for $C_{37}H_{55}GaN_4Si$*Toluene: C, 70.86; H, 8.51; N, 7.51. Found: C, 70.57; H, 9.04; N, 7.07.

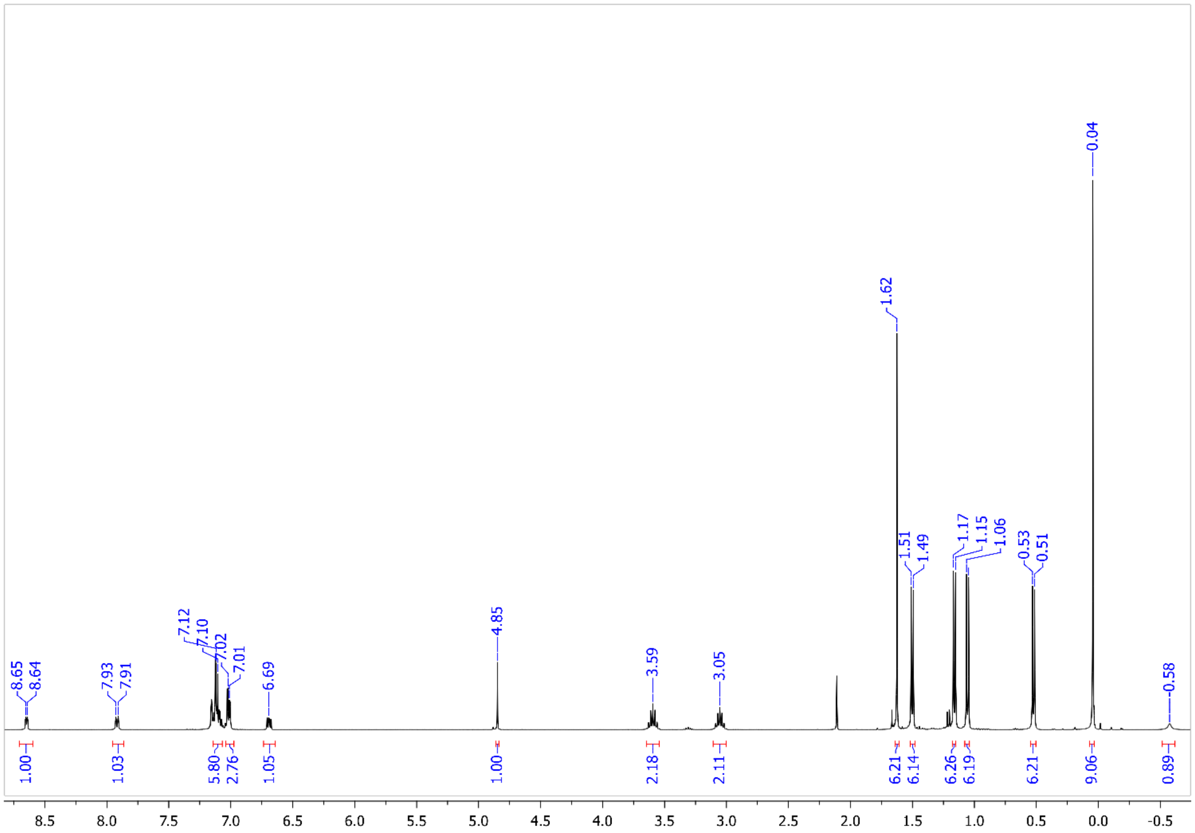

Figure S1. ^{1}H NMR (400 MHz, C_6D_6) spectrum of NacNacGa(py)($NHSiMe_3$).

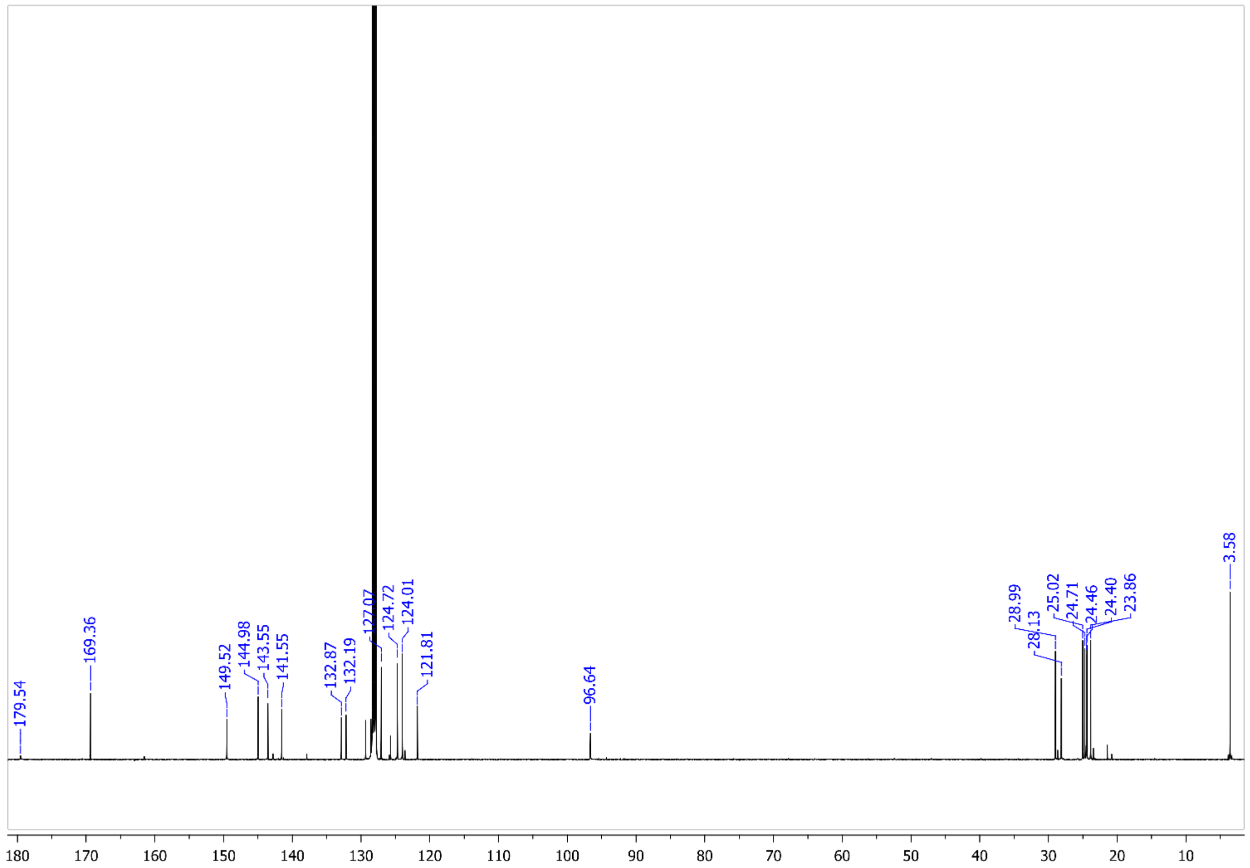

Figure S2. $^{13}C\{^{1}H\}$ NMR (101 MHz, C_6D_6) spectrum of NacNacGa(py)(NHSiME$_3$).

Preparation of NacNacGa(OC(=CH(CH$_2$)$_4$–)(NHSiMe$_3$) (3)

To a solution of NacNacGa (0.078 g, 0.16 mmol) in toluene was added cyclohexanone (0.017 mL, 0.16 mmol) and the mixture was treated with N_3SiMe_3 (0.021 mL, 0.16 mmol). The reaction was allowed to stir for 10 min at room temperature. The volatiles were removed to give NacNacGa(OC(=CH(CH$_2$)$_4$–)(NHSiMe$_3$) as a white powder in quantitative yield. X-ray quality single crystals of **3** were obtained from a toluene solution at -30^{0}C.

^{1}H NMR (400 MHz, C6D6) δ 7.17-7.01 (m, 6H, C_6H_3), 4.87 (s, 1H, CH), 4.53 (m, 1H, OC_6H_9), 3.56 (hept, 2H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H}$ = 6.8 Hz), 3.18 (hept, 2H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H}$ = 6.9 Hz), 2.32 (m, 4H, OC_6H_9), 1.81 (m, 2H, OC_6H_9), 1.71 (m, 2H, OC_6H_9), 1.49 (s, 6H, NCCH$_3$), 1.44 (d, 6H, CH(CH_3)$_2$, $^3J_{H\text{-}H}$ = 6.8 Hz), 1.37 (d, 6H, CH(CH_3)$_2$, $^3J_{H\text{-}H}$ = 6.9 Hz), 1.25 (d, 6H, CH(CH_3)$_2$, $^3J_{H\text{-}H}$ = 6.8 Hz), 1.08 (d, 6H, CH(CH_3)$_2$, $^3J_{H\text{-}H}$ = 6.9 Hz), -0.10 (s, 9H, Si(CH$_3$)$_3$), -0.89 (s, 1H, NH).

^{13}C{^{1}H} NMR (101 MHz, C6D6) δ 169.7 (N*C*CH$_3$), 155.5 (O*C*(=CH)(CH$_2$)$_4$), 145.6, 143.3, 140.8, 127.6, 124.9, 123.9, 96.7 (CH), 96.3 (OC(=*C*H)(CH$_2$)$_4$), 28.95, 28.35 (*C*H(CH$_3$)$_2$), 31.6, 25.63, 24.8, 24.1 (CH$_2$), 25.6, 24.9, 24.8, 24.6 (CH(*C*H$_3$)$_2$), 23.7 (NC*C*H$_3$), 2.9 (Si(CH$_3$)$_3$).

Elemental analysis: Calculated for $C_{38}H_{60}GaN_3OSi$*0.5Tol: C 69.35; H, 8.97; N, 5.85. Found: C 69.16, H 9.09, N 5.62.

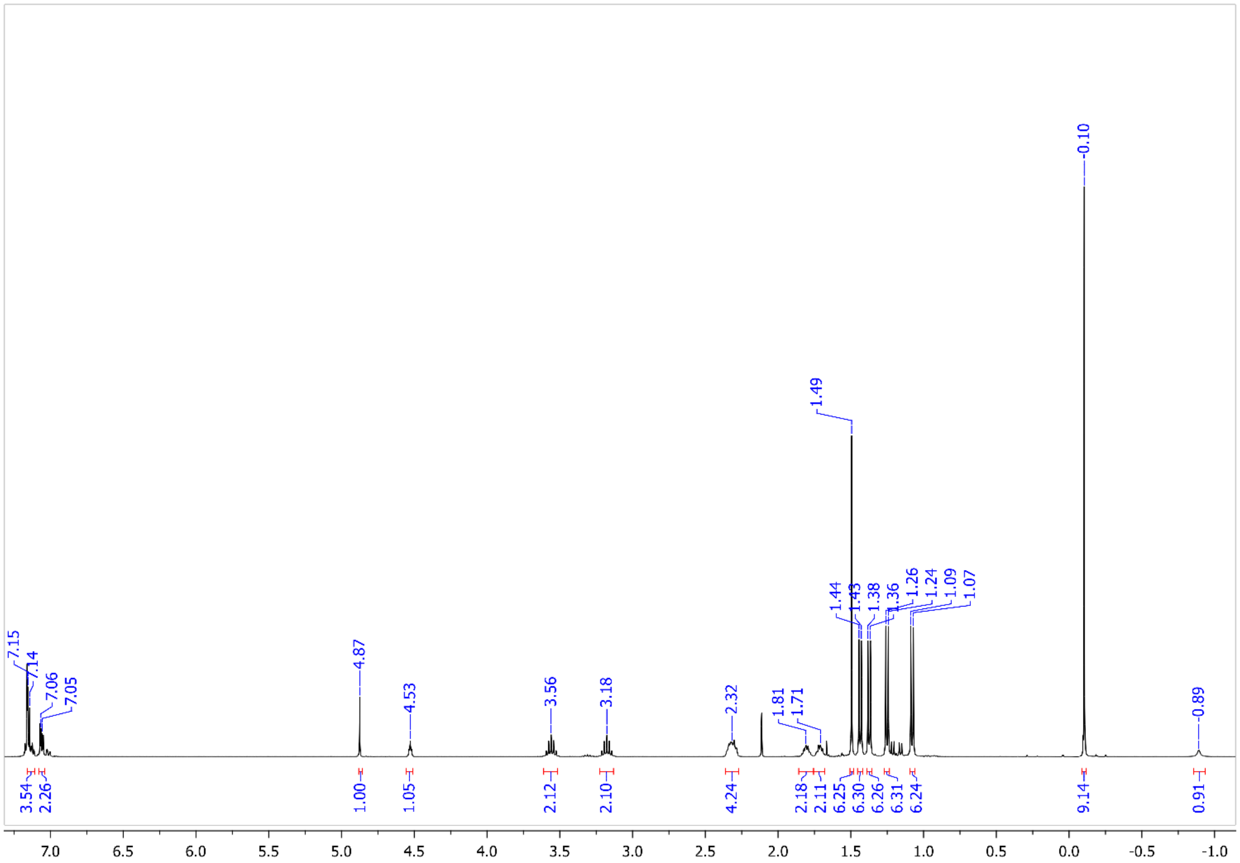

Figure S3. ^{1}H NMR (400 MHz, C_6D_6) spectrum of NacNacGa(OC(=CH(CH$_2$)$_4$–)(NHSiMe$_3$).

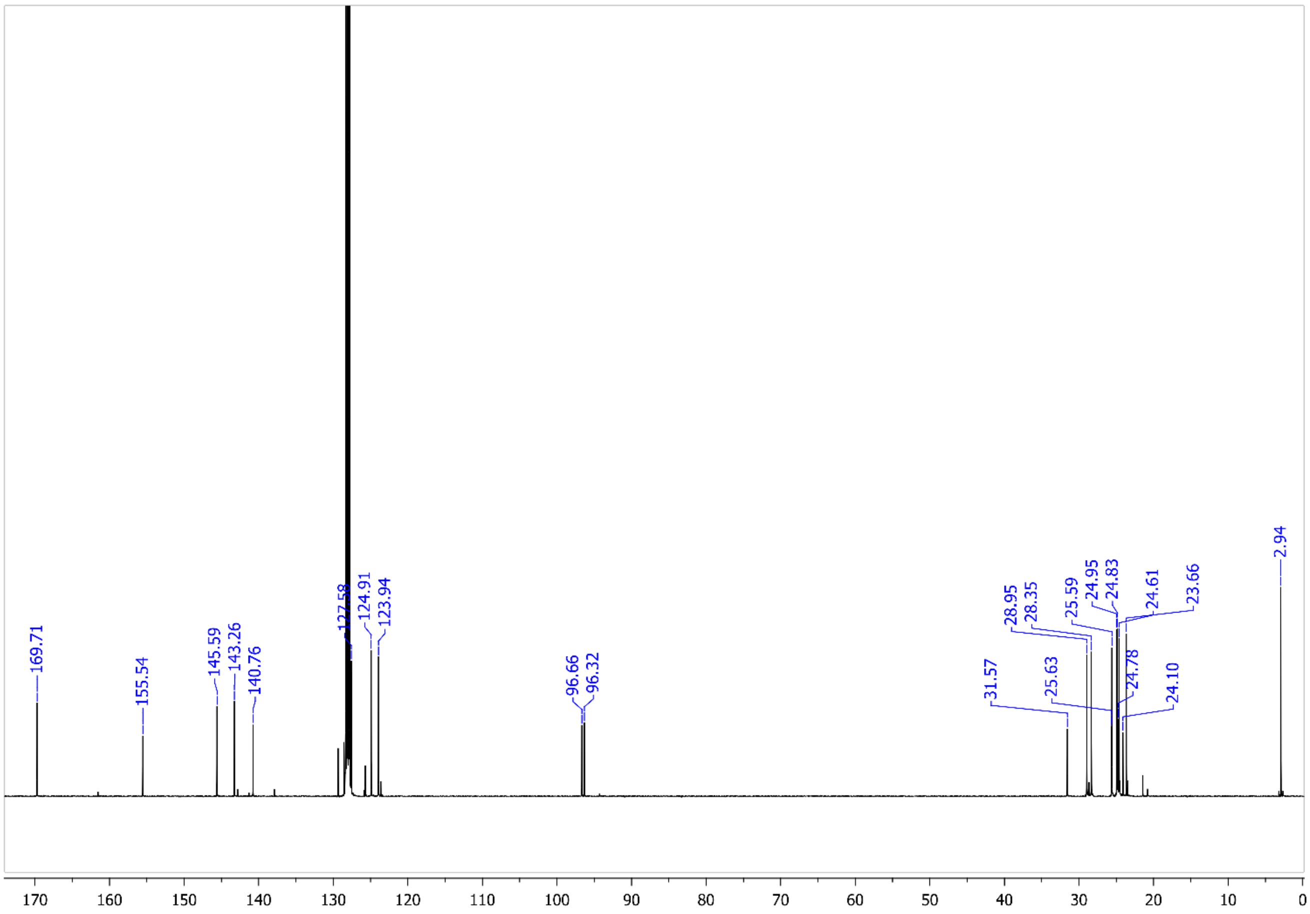

Figure S4. $^{13}C\{^{1}H\}$ NMR (101 MHz, C_6D_6) spectrum of NacNacGa(OC(=CH(CH_2)$_4$–)(NHSiMe$_3$).

Preparation of NacNacGa($CH_2CO_2CH_2CH_3$)(NHSiMe$_3$) (4)

To a solution of NacNacGa (0.078 g, 0.16 mmol) in toluene was added ethyl acetate (0.016 mL, 0.16 mmol) and the mixture was treated with N_3SiMe_3 (0.021 mL, 0.16 mmol). The reaction was allowed to stir for 10 min at room temperature. The volatiles were removed to give NacNacGa($CH_2CO_2CH_2CH_3$)(NHSiMe$_3$) as a white powder in quantitative yield. Analytically pure material was upon obtained by crystallization from ether. X-ray quality single crystals of **4** were obtained from toluene at -30°C.

^{1}H NMR (400 MHz, C_6D_6) δ 7.19-7.08 (m, 6H, C_6H_3), 4.72 (s, 1H, CH), 4.01 (q, 2H, OCH_2CH_3, $^{3}J_{H\text{-}H}$ = 7.14 Hz), 3.96 (hept, 2H, CH(CH_3)$_2$ $^{3}J_{H\text{-}H}$ = 6.7 Hz), 3.45 (hept, 2H, CH(CH_3)$_2$ $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.90 (s, 2H, CH_2), 1.56 (s, 6H, NCCH_3), 1.47 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.7 Hz), 1.37 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.33 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.7 Hz), 1.12 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.07 (t, 3H, OCH_2CH_3, $^{3}J_{H\text{-}H}$ = 7.14 Hz), -0.20 (s, 9H, Si(CH_3)$_3$), -0.59 (s, 1H, NH).

$^{13}C\{^1H\}$ NMR (101 MHz, C_6D_6) δ 175.2 (CO), 170.2 (N*C*CH_3), 145.7, 144.0, 141.1, 127.3, 125.2, 124.3 (C_6H_3), 97.6 (CH), 59.4 (O*C*H_2CH_3), 28.7, 26.9 (*C*H(CH_3)$_2$), 26.1, 25.12, 25.06, 24.9 (CH(*C*H_3)$_2$), 23.9 (NC*C*H_3), 20.9 (CH_2), 14.8 (OCH_2*C*H_3), 3.2 (Si(CH_3)$_3$).

Elemental analysis: Calculated for $C_{36}H_{58}GaN_3O_2Si$: C, 65.25; H, 8.82; N, 6.34. Found: C, 65.45; H, 9.65, N, 6.37.

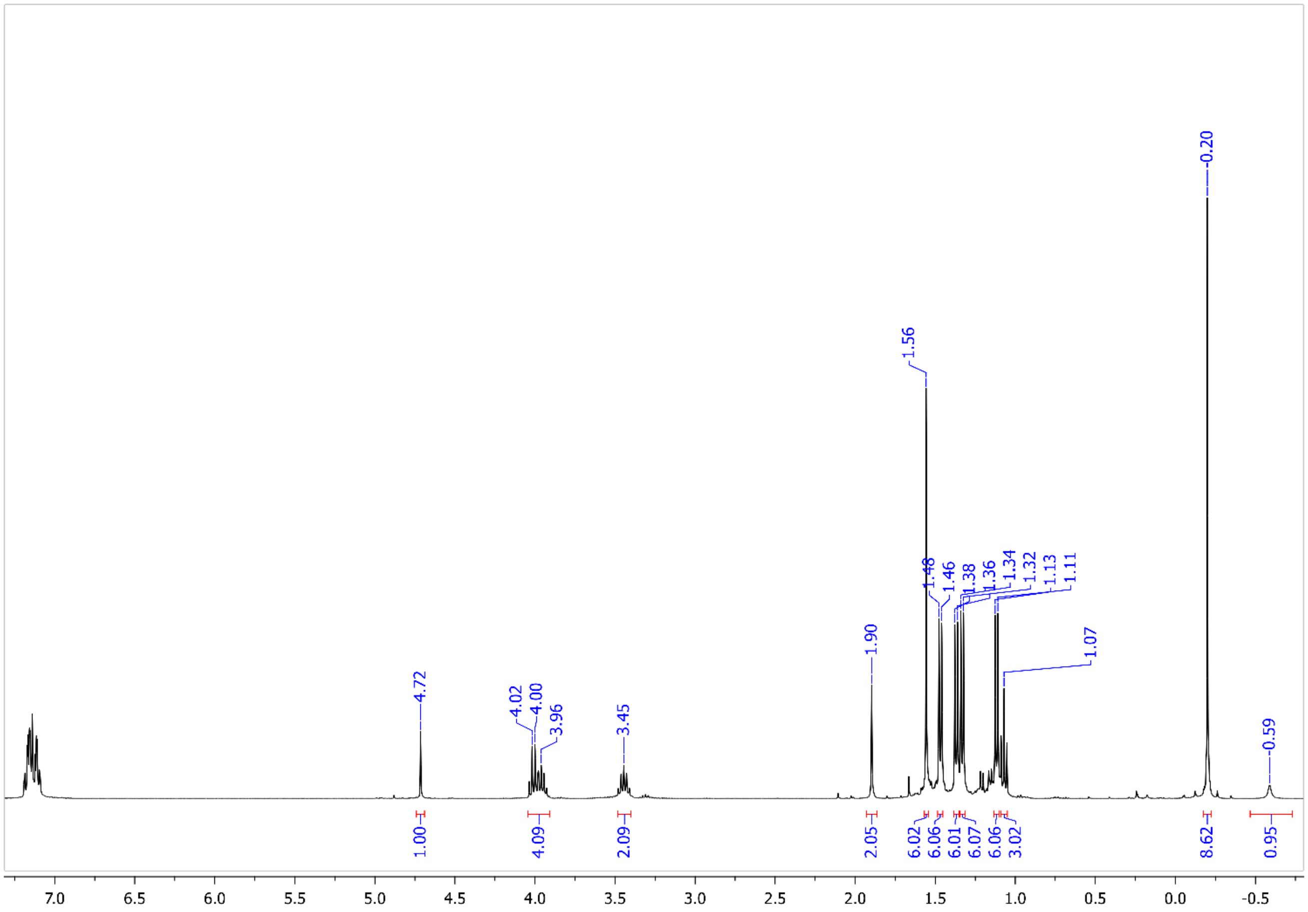

Figure S5. ^1H NMR (400 MHz, C_6D_6) spectrum of NacNacGa($CH_2CO_2CH_2CH_3$)($NHSiMe_3$).

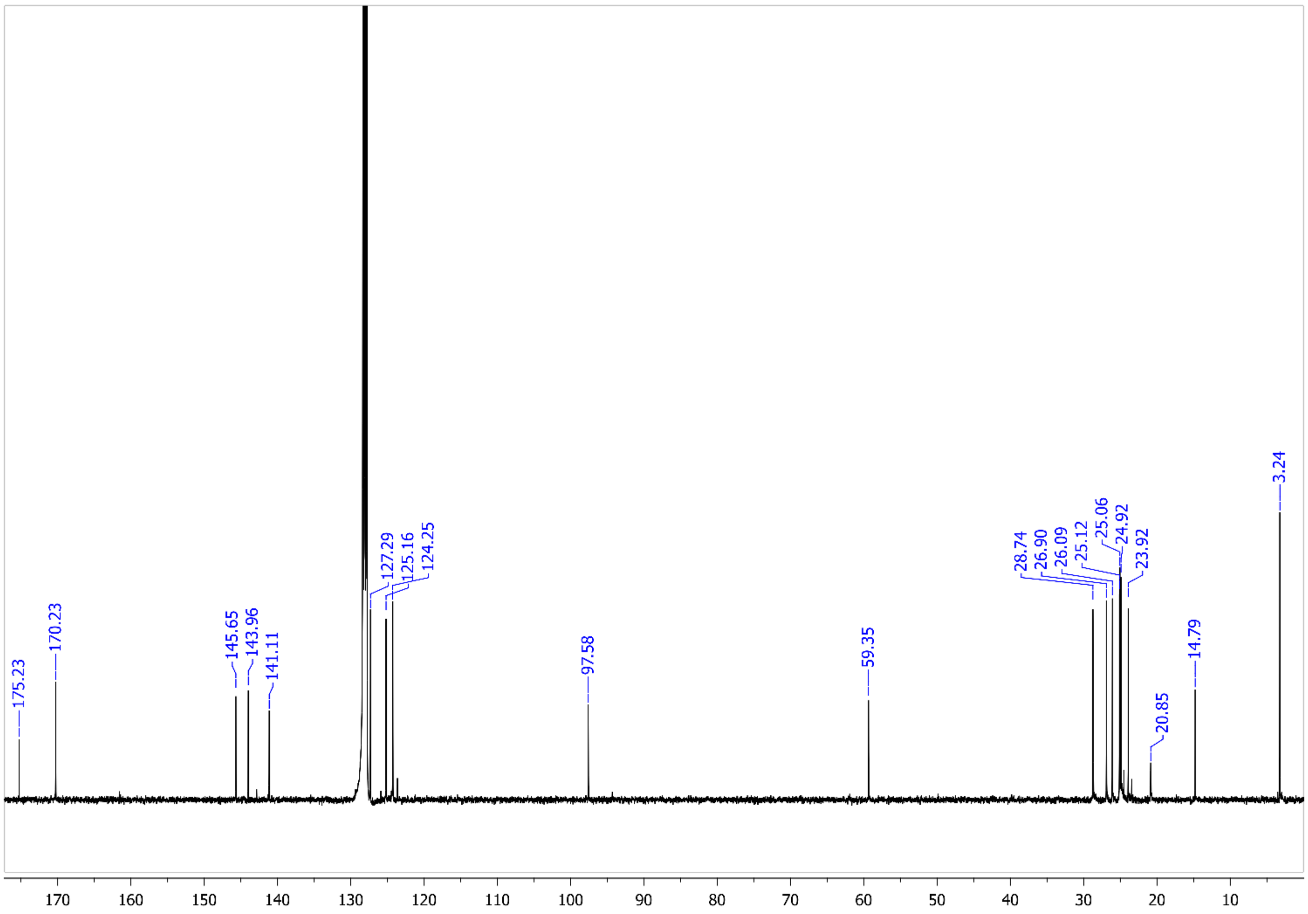

Figure S6. $^{13}C\{^{1}H\}$ NMR (101 MHz, C_6D_6) spectrum of NacNacGa($CH_2CO_2CH_2CH_3$)($NHSiMe_3$).

Preparation of NacNacGa(OS(CH_2)(CH_3))($NHSiMe_3$) (5)

To a solution of NacNacGa (0.071 g, 0.15 mmol) in toluene was added DMSO (0.010 mL, 0.15 mmol) and the mixture was treated with N_3SiMe_3 (0.019 mL, 0.15 mmol). The reaction was allowed to stir for 10 min at room temperature. The` volatiles were removed and NacNacGa(OS(CH_2)(CH_3))($NHSiMe_3$) was obtained as a white powder in quantitative yield.

^{1}H NMR (400 MHz, C6D6) δ 7.19-7.05 (m, 6H, C_6H_3), 4.91 (s, 1H, CH), 4.10 (hept, 1H, CH(CH_3)$_2$ $^{3}J_{H\text{-}H}$ = 6.8 Hz), 3.55 (m, 2H, CH(CH_3)$_2$), 3.39 (hept, 1H, CH(CH_3)$_2$ $^{3}J_{H\text{-}H}$ = 6.8 Hz), 2.28 (d, 1H, SCH_2, $^{2}J_{H\text{-}H}$ = 12.8 Hz), 2.12 (s, 3H, SCH_3), 1.81 (d, 1H, SCH_2, $^{2}J_{H\text{-}H}$ = 12.8 Hz), 1.62 (s, 3H, $NCCH_3$), 1.54 (s, 3H, $NCCH_3$), 1.44 (d, 3H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.9 Hz), 1.42 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.36 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.18 (d, 6H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.8 Hz), 1.13(d, 3H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.9 Hz), 1.12 (d, 3H, CH(CH_3)$_2$, $^{3}J_{H\text{-}H}$ = 6.9 Hz), -0.13 (s, 9H, Si(CH_3)$_3$), -0.27 (s, 1H, NH).

$^{13}C\{^{1}H\}$ NMR (101 MHz, C_6D_6) δ 170.8, 168.6 (N*C*CH$_3$), 145.3, 144.2, 144.0, 143.8, 140.9, 140.8, 126.94, 126.87, 124.7, 124.3, 123.9 (C_6H_3), 98.2 (CH), 45.3 (SCH$_3$), 41.1 (SCH$_2$), 28.4, 28.3, 27.1, 26.5 (*C*H(CH$_3$)$_2$), 26.0, 25.3, 24.8, 24.7, 24.6, 24.4, 23.9, 23.4 (NC*C*H$_3$), 3.1 (Si(CH$_3$)$_3$).

Elemental analysis: Calculated for $C_{34}H_{56}GaN_3OSSi$: C, 62.57; H, 8.65; N, 6.44. Found: C, 62.51; H, 8.75; N, 6.10.

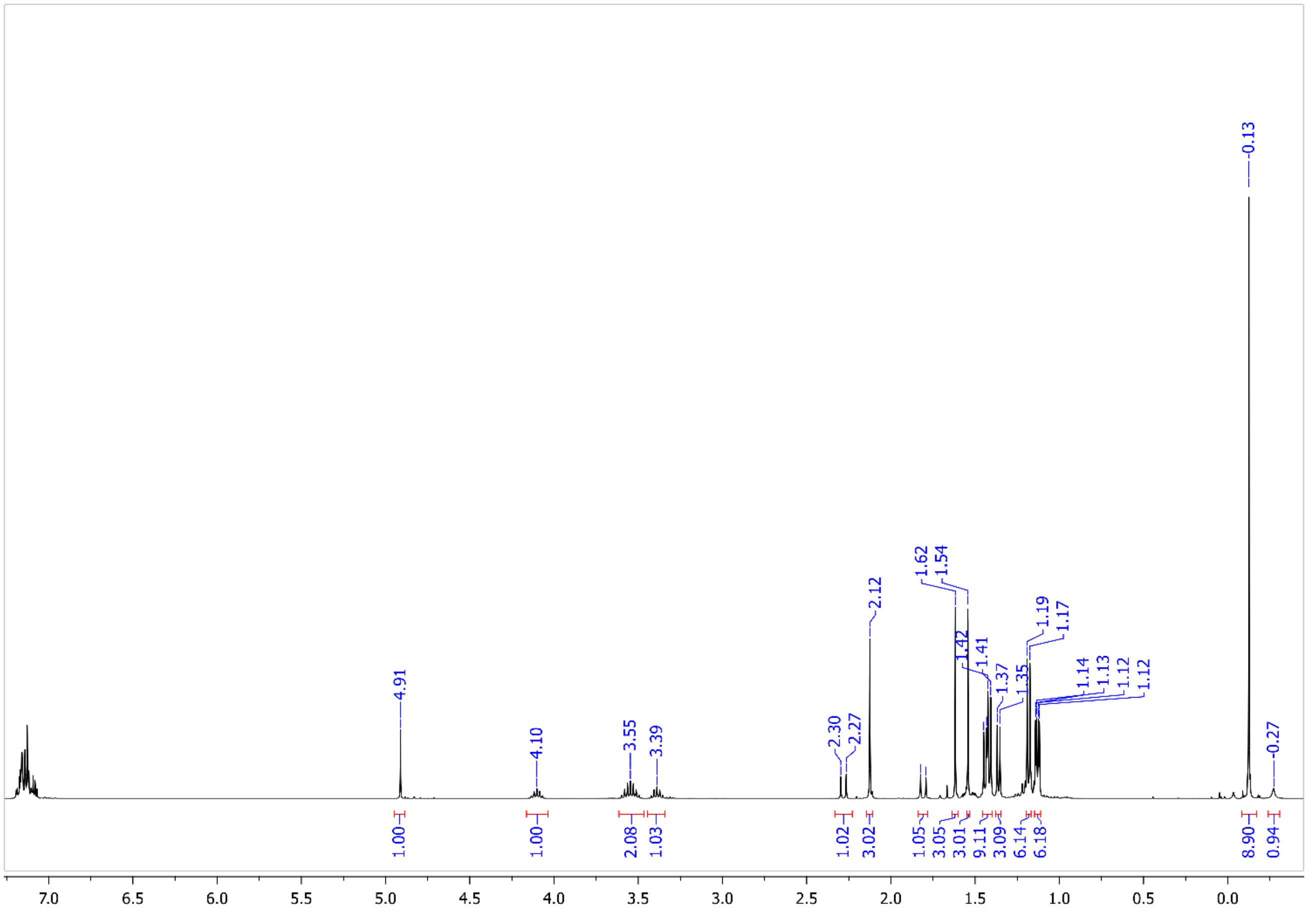

Figure S7. ^{1}H NMR (400 MHz, C_6D_6) spectrum of NacNacGa(OS(CH$_2$)(CH$_3$))(NHSiMe$_3$).

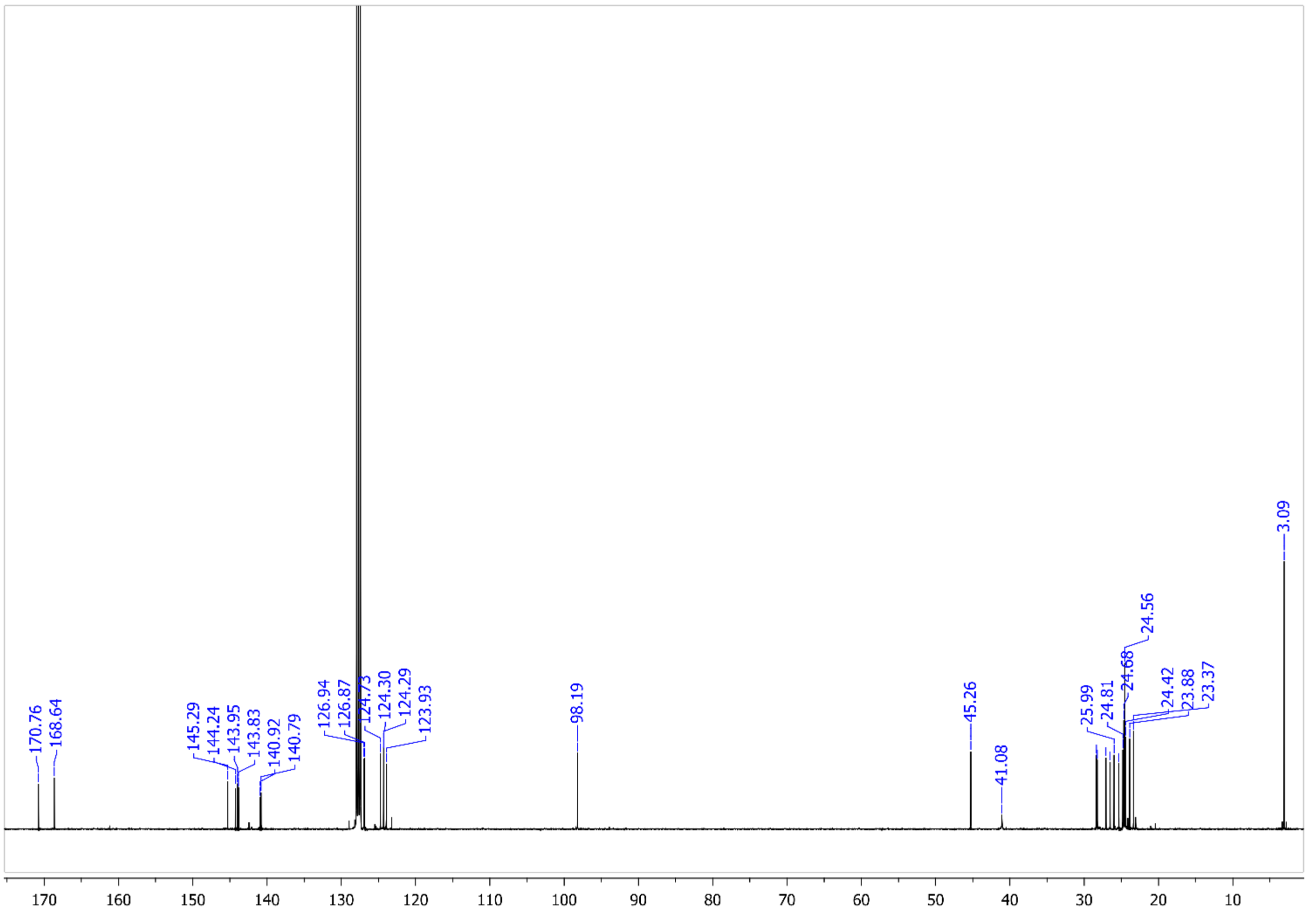

Figure S8. $^{13}C\{^1H\}$ NMR (101 MHz, C_6D_6) spectrum of NacNacGa(OS(CH_2)(CH_3))(NHSiMe$_3$)

Preparation of NacNacGa(OP(CHCH$_3$)(CH$_2$CH$_3$)$_2$)(NHSiMe$_3$) (6)

To a solution of NacNacGa (0.064 g, 0.13 mmol) in toluene was added $Et_3P=O$ (0.018 g, 0.13 mmol) and the mixture was treated with N_3SiMe_3 (0.017 mL, 0.13 mmol). The reaction was allowed to stir for 10 min at room temperature. The reaction mixture was concentrated and crystallized at -30^{0}C. Colourless crystals of NacNacGa(OP(CHCH$_3$)(CH$_2$CH$_3$)$_2$)(NHSiMe$_3$) was obtained (0.066 g, 0.09 mmol, 72%).

^{1}H {^{31}P} NMR (400 MHz, C6D6) δ 7.21-7.03 (m, 6H, C_6H_3), 4.94 (s, 1H, CH), 4.03 (hept, 1H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H} = 6.7$ Hz), 3.96 (hept, 1H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H} = 6.7$ Hz), 3.29 (hept, 1H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H} = 6.7$ Hz), 3.24 (hept, 1H, CH(CH$_3$)$_2$ $^3J_{H\text{-}H} = 6.7$ Hz), 2.20 (s, 1H, NH), 1.65 (s, 3H, NCCH$_3$), 1.64 (d, 3H, CH(CH_3)$_2$, $^3J_{H\text{-}H} = 6.7$ Hz),1.62 (s, 3H, NCCH$_3$), 1.61 (d, 3H, CH(CH_3)$_2$, $^3J_{H\text{-}H} = 6.7$ Hz), 1.28 (m, 12H, CH(CH_3)$_2$), 1.12 (d, 3H, CH(CH_3)$_2$, $^3J_{H\text{-}H} = 6.7$ Hz), 1.07 (d, 3H, CH(CH_3)$_2$, $^3J_{H\text{-}H} = 6.7$ Hz), 1.03 (m, 1H, PCH_2CH$_3$), 1.01 (q, 1H, PCHCH$_3$, $^3J_{H\text{-}H} = 7.6$ Hz), 0.86 (m, 1H, PCH_2CH$_3$), 0.78 (t, 3H,

$\mathrm{PCH_2C}\mathit{H}_3$, ${}^3J_{\mathrm{H\text{-}H}}$ = 7.2 Hz), 0.75 (t, 3H, $\mathrm{PCH_2C}\mathit{H}_3$, ${}^3J_{\mathrm{H\text{-}H}}$ = 7.3 Hz), 0.69 (m, 1H, $\mathrm{PC}\mathit{H}_2\mathrm{CH_3}$), 0.56 (m, 1H, $\mathrm{PC}\mathit{H}_2\mathrm{CH_3}$), 0.51 (d, 3H, $\mathrm{PCHC}\mathit{H}_3$, ${}^3J_{\mathrm{H\text{-}H}}$ = 7.6 Hz), 0.40 (s, 9H, $\mathrm{Si(CH_3)_3}$).

^{13}C{^{1}H} NMR (101 MHz, C6D6) δ 169.6, 169.4 (N*C*CH$_3$), 146.4, 146.2, 143.3, 143.2, 143.0, 142.1, 127.04, 127.0125.9, 125.7, 124.2, 124.0 ($\mathrm{C_6H_3}$), 98.7 (CH), 28.9, 28.7, 27.8, 27.7 ($\mathit{C}\mathrm{H(CH_3)_2}$), 26.4, 25.9, 25.8, 25.31, 25.29, 25.0, 24.9, 24.4 ($\mathrm{CH(}\mathit{C}\mathrm{H_3)_2}$), 24.33, 24.29 ($\mathrm{NC}\mathit{C}\mathrm{H_3}$), 18.4 ($\mathrm{P}\mathit{C}\mathrm{H_2CH_3}$), 16.7 ($\mathrm{P}\mathit{C}\mathrm{HCH_3}$), 19.7, 10.7 ($\mathrm{PCH}\mathit{C}\mathrm{H_3}$), 19.1, 19.0, 5.8 ($\mathrm{PCH_2}\mathit{C}\mathrm{H_3}$), 4.3 ($\mathrm{Si(CH_3)_3}$).

^{31}P{1H} NMR (162 MHz, $\mathrm{C_6D_6}$) δ 52.86 ppm.

Elemental analysis: Calculated for $\mathrm{C_{38}H_{65}GaN_3OPSi}$: C, 64.40; H, 9.24, N, 5.93. Found: C, 64.87; H, 9.76; N, 5.97.

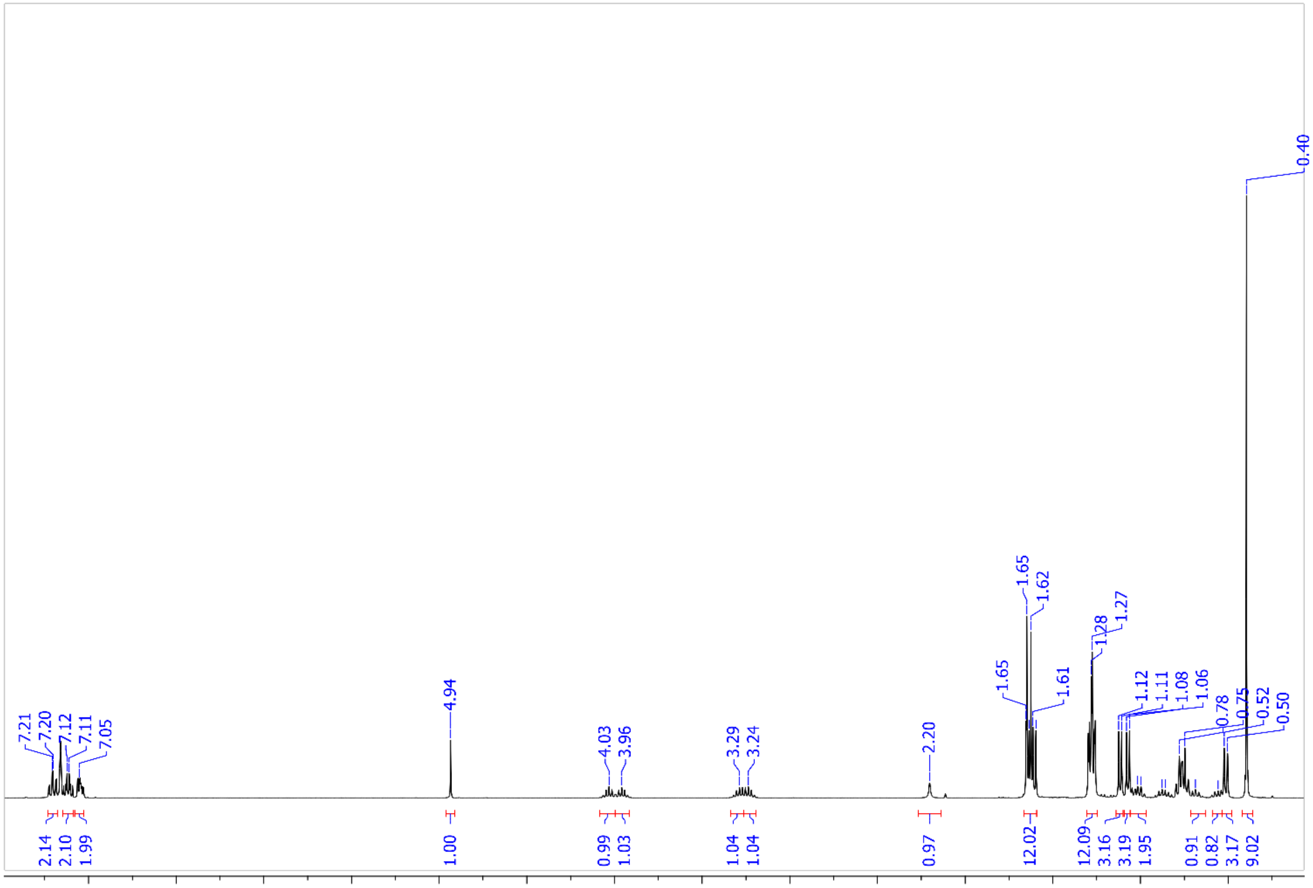

Figure S9. ^{1}H{^{31}P} NMR (400 MHz, $\mathrm{C_6D_6}$) spectrum of NacNacGa($\mathrm{OP(CHCH_3)(CH_2CH_3)_2}$)($\mathrm{NHSiMe_3}$).

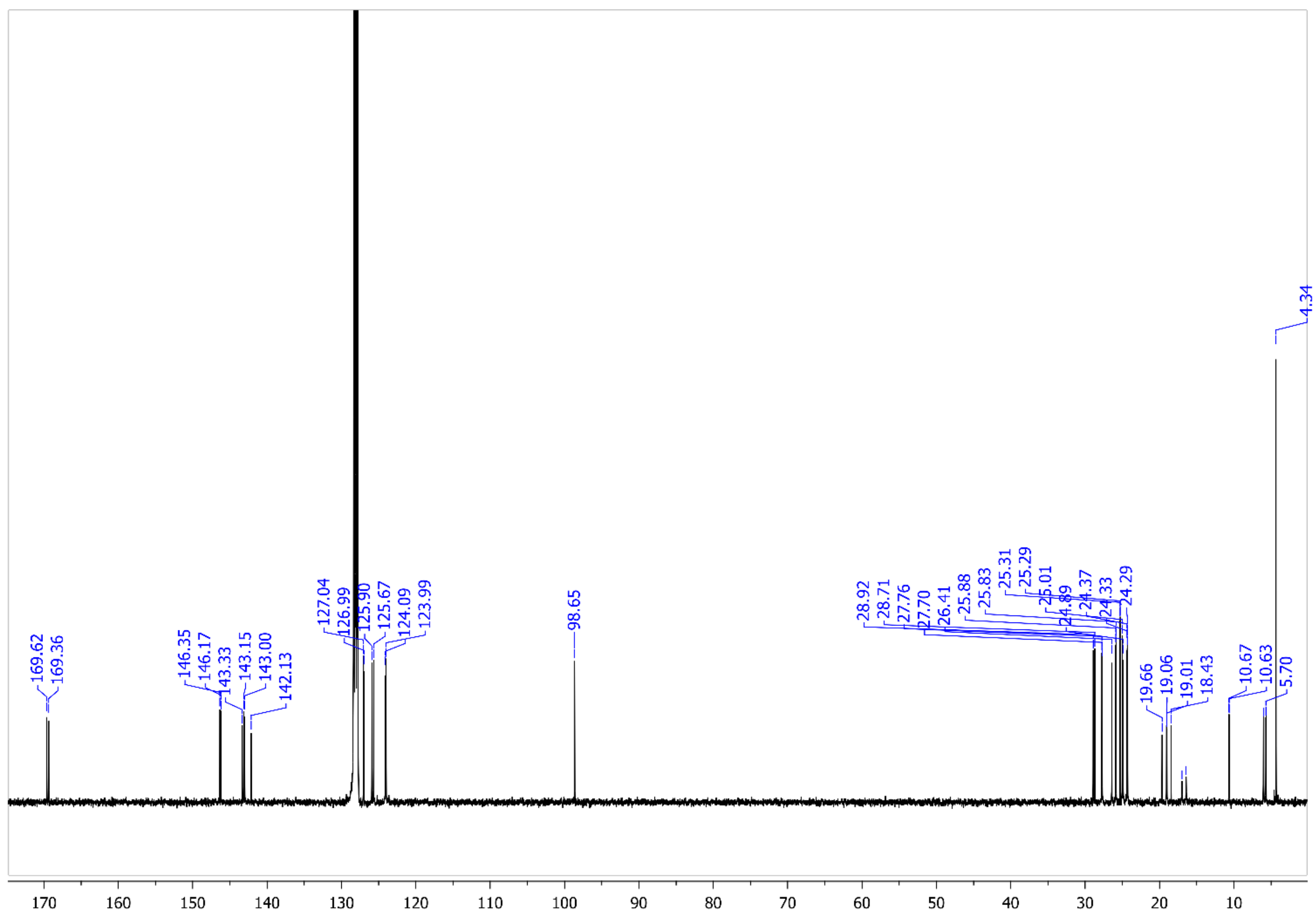

Figure S10. $^{13}C\{^{1}H\}$ NMR (101 MHz, C_6D_6) spectrum of NacNacGa(OP(CHCH$_3$)(CH$_2$CH$_3$)$_2$)(NHSiMe$_3$).

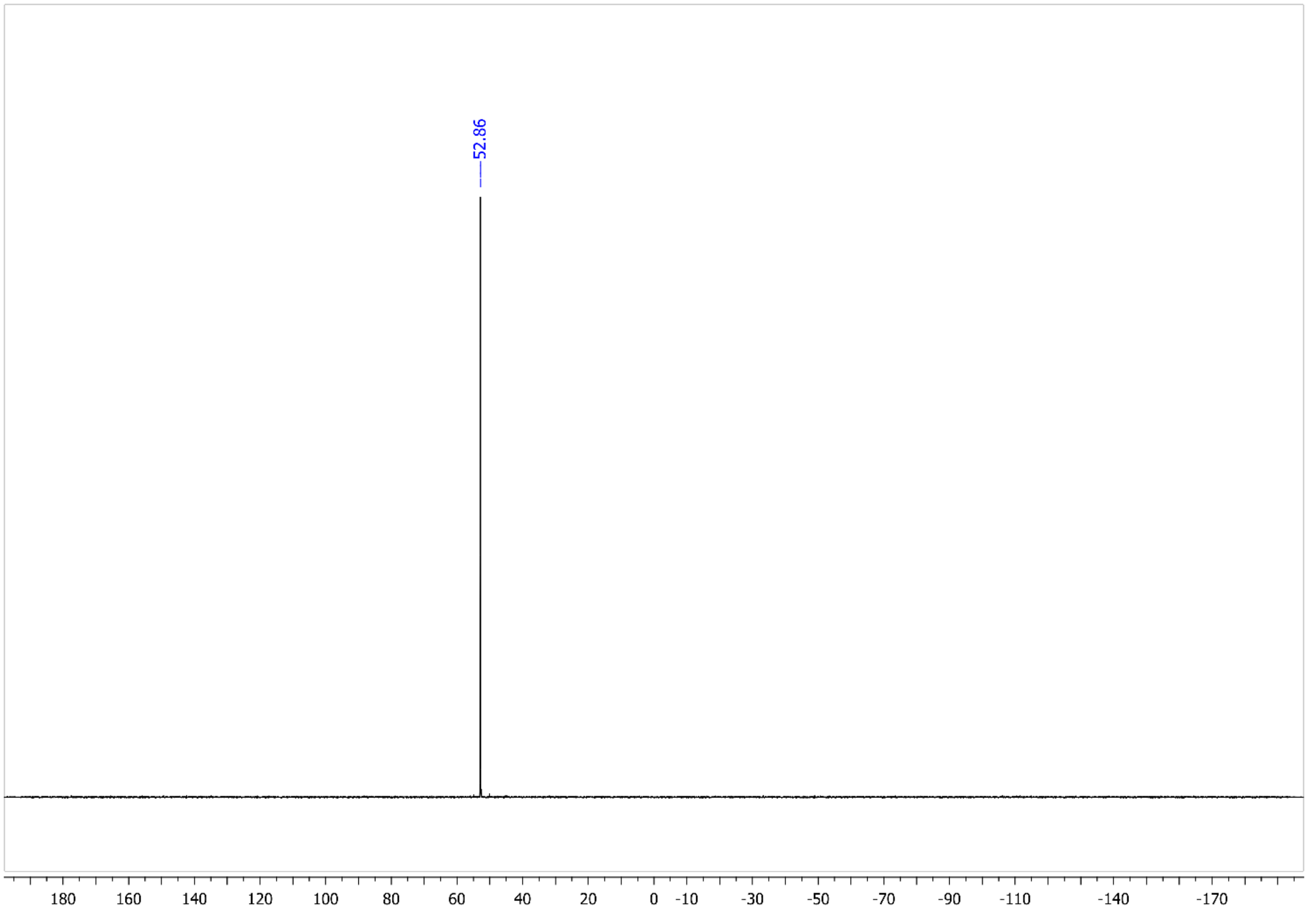

Figure S11. $^{31}P\{^{1}H\}$ NMR (162 MHz, C_6D_6) spectrum of NacNacGa(OP($CHCH_3$)(CH_2CH_3)$_2$)($NHSiMe_3$).

X-ray crystallography

Single crystals of **2**, **3**, **4**, and **6** were grown from diethyl ether at -30ºC.

A suitable single crystal of compound **2** was mounted on a glass cryoloop covered with Paratone N® oil and examined on a Bruker APEX-II CCD diffractometer equipped with a CCD area detector and an Oxford Cryoflex low temperature device. Data were collected at 150(2) K with Mo Kα radiation (λ = 0.71073 Å) using the APEX-II software.[2] Generic φ and ω scans were used for the data measurement. Unit cell determination, refinement and data-reduction were carried out with SAINT.[3] An absorption correction was performed with the multi-scan method implemented in SADABS.[4] The structures of the compounds were solved with SHELXT (Intrinsic phasing)[5] and refined using SHELXL-2014[6] in the Bruker SHELXTL suite.[7] Hydrogen atoms were added at calculated positions and refined using a riding model.

Single crystal X-ray diffraction data for compounds **3**,**4** and **6** were collected using the Canadian Macromolecular Crystallography Facility CMCF-BM beamline at the Canadian Light Source (CLS).[8] The CMCF-BM is a bending magnet beamline equipped with a Si (111) double crystal monochromator, Rayonix MX300HE CCD, and MD2 microdiffractometer equipped with Mini Kappa Goniometer Head. Data for compounds **3, 4,** and **6** were collected at 19.0000 keV (0.6526 Å). Data for compounds **3**, **4**, and **6** were acquired at 100 K using Oxford Instruments Cryojet5.

Cell refinement and data reduction were performed using XDS.[9] A semi-empirical absorption correction, based on the multiple measurements of equivalent reflections, and merging of data was performed using SADABS.[10] Data conversion from XDS file format to SADABS file format was performed using XDS2SAD.[11] The space group was confirmed by XPREP routine. [12]

The structures of compounds **3**, **4**, and **6** were solved by direct-methods and refined by full-matrix least-squares and difference Fourier techniques with SHELXL-2018.[13] The real-space refinement was performed using COOT[14] The disorder in the alkyl groups were modeled as two-component disorders. All atoms of the disordered fragments were restrained using SADI restraints (bonds and angles) whenever possible. SIMU, DELU, and ISOR restraints were applied to the U_{ij} components whenever necessary.

All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were assigned in calculated positions and refined as riding atoms with a common thermal parameter, with the exception of the N-H hydrogens which were extracted from the difference Fourier maps and freely refined. All publication materials were prepared using LinXTL[15] and the Mercury[16] programs. The checkCIF routine and structure factor analyses were performed using PLATON.[17] Select experimental crystallographic data for all four compounds is presented in Table SI1.

Table SI 1. Crystal data collection and refinement parameters for compounds 2- 4 and 6

	2	**3**	**4**	**6**
chemical formula	$C_{37}H_{55}GaN_4Si$	$C_{83}H_{127}Ga_2N_6O_2Si_2$	$C_{36}H_{58}GaN_3O_2Si$	$C_{38}H_{65}GaN_3OPSi$
crystal colour	yellow	colourless	colourless	colourless
Fw; F(000)	930.41; 1943	1436.52; 1546	662.66; 712	708.71; 1528
T (K)	100	100	100	100
wavelength (Å)	0.68879	0.65254 (sync)	0.65254 (sync)	0.65254 (sync)
space group	$P2_1/c$	$P2_1/n$	P-1	$P2_1/c$
a (Å)	8.2480(16)	11.940(2)	9.468(2)	12.943(3)
b (Å)	37.082(7)	17.146(3)	10.277(2)	12.950(3)
c (Å)	19.004(4)	20.083(4)	20.409(4)	23.406(5)
α (deg)	90	90	89.67(3)	90
β (deg)	94.33(3)	97.15(3)	87.02(3)	90.16(3)
γ (deg)	90	90	64.19(3)	90
Z	4	2	2	4
V ($Å^3$)	5796(2)	4079.5(14)	1785.0(7)	3923.1(14)
ρ_{calcd} ($g{\cdot}cm^{-3}$)	1.066	1.169	1.233	1.200
μ (mm^{-1})	0.303	0.577	0.656	0.639
θ range (deg); completeness	1.064 − 24.202; 0.953	1.438 − 24.499; 1.000	0.917 − 24.499; 0.987	1.444 − 22.855; 1.000
collected reflections; R_σ	19319; 0.0691	62238; 0.0369	55320; 0.0354	49408; 0.0486
unique reflections; R_{int}	9747; 0.0450	8774; 0.0632	7554; 0.0628	6944; 0.0753
$R1^a$; $wR2^b$ [I > 2σ(I)]	0.1196; 0.3150	0.0341; 0.0903	0.0331; 0.0882	0.0489; 0.1363
R1; wR2 [all data]	0.1938; 0.3962	0.0379; 0.0929	0.0338; 0.0887	0.0524; 0.1401
GOF	1.141	1.076	1.070	1.103
largest diff peak and hole	0.744 and -0.472	0.334 and -0.860	0.404 and -0.748	0.725 and -1.584

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