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Supporting Information

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1. Experimental section

General procedures: All experiments were perfomed under dry argon gas using standard Schlenk techniques. Toluene, di-*n*-butylether and *n*-heptane were dried over Na/K respectively LiAlH₄ and stored under argon-atmosphere. C₆D₆ was dried over Na/K and stored over activated molecular sieve (3 Å). (Dipp₂NacNac)Ga, *t*BuSbCl₂, MeBiCl₂, PhBiCl₂ and MesBiCl₂ were prepared according to literature procedures or based on them.^[1–4] LiAlH₄ was used as purchased.

¹H- and ¹³C-NMR-spectra were recorded on a Bruker AV II 300 MHz relative to the tetramethylsilane standard. IR-spectra were recorded on a BRUKER Alpha FT-IR with a diamond ATR (500-4000 cm⁻¹). Elemental analysis were performed on an ELEMENTAR vario Microcube and the content is reported in %. X-ray crystallographic data were collected on a STOE *IPDS 2* or a Bruker *D8 Quest* diffractometer using monochromatic Mo-K α radiation ($\lambda = 0.71073$ Å). The solution of the structure was performed with intrinsic phasing with the SHELXT-2015 solution program, while for the structure refinement with full-matrix least-squares against *F*² the SHELXL-2015 package was used, both within the OLEX² environment.^[5–7]

Synthesis of (Dipp2NacNac)GaH(SbHtBu) (1). The following synthesis was performed under exclusion of light. A solution of 1 g (4 mmol, 10 eq) tBuSbCl₂ in 10 mL nBu₂O was added dropwise to a solution of 0.9 g LiAlH₄ in 50 mL nBu₂O at -40 °C. Immediately, evolution of gas was observed which was passed through a cooling trap at -78 °C. The formed side product SbH₃ was trapped in a second cooling trap at -196 °C and the formed H₂ removed *in vacuo*. After 1 ½ h, the first cooling trap was separated and set under argon before a solution of 0.21 g (0.43 mmol, 1 eq) (Dipp₂NacNac)Ga in 10 mL toluene was added to the frozen tBuSbH₂ over a drip funnel. The cooling bath was removed and within a few minutes, the former yellow solution turned brown and was allowed to stir at room temperature overnight. The toluene was removed in vacuo and the remaining solid extracted with 20 mL n-heptane and centrifuged. A recorded in situ ¹H NMR spectrum of this solution in C₆D₆ showed the selective and almost quantitative formation of compound **1**. However, to remove traces of decomposition products formed by the decomposition of excess tBuSbH₂ crystallisation at low temperatures is necessary to obtain an analytically pure product. Yellow plates of 1 were obtained from the solution at -32 °C in a yield of 0.03 g (0.05 mmol, 12%) after washing the crystals for three times with a cold solution of *n*-heptane. After concentration of the combined mother liquor and wash solution, further fractions of highly soluble compound 1 can be crystallized at -32 °C to increase the yield.

Elemental analysis calcd. (%) for C₃₃H₅₂SbGaN₂: C, 59.31; H, 7.84; N, 4.19. Found: C, 59.39; H, 7.908; N, 4.63. ¹H NMR (300 MHz, C₆D₆) δ (ppm) = 0.74 (s, 1H, Sb*H*), 1.12-1.19 (m, 12H, CH(C*H*₃)₂), 1.26 (s, 9H, C(C*H*₃)₃), 1.40 (d, 3H, ²*J*_{H-H} = 6.7 Hz, CH(C*H*₃)₂), 1.45-1.49 (m, 6H CH(C*H*₃)₂), 1.56 (d, 3H, ²*J*_{H-H} = 6.9 Hz, CH(C*H*₃)₂), 1.61 (s, 3H, β-C*H*₃), 1.62 (s, 3H, β-C*H*₃), 3.27-3.45 (m, 3H, C*H*(CH₃)₂), 3.59 (hept, 1H, ²*J*_{H-H} = 6.8 Hz), 4.82 (s, 1H, γ-C*H*), 6.69 (s, 1H, Ga*H*), 7.06-7.14 (m, 6H, Aryl-*H*). ¹³C{¹H} NMR (75.5 MHz, C₆D₆) δ (ppm) = 21.8, 22.8, 23.3, 23.9, 24.2, 24.6, 25.3, 25.9, 24.7, 27.9, 28.9, 29.3 (C(CH₃), CH(CH₃)₂, C(CH₃)₃), 36.5 (C(CH₃)₃), 95.3 (γ-CH), 123.9, 124.1, 124.2, 124.6, 126.7, 141.7, 142.0, 142.4, 142.5, 144.6, 145.0 (Aryl-*C*), 168.4 (NCCH₃). **IR** (ATR) ν (cm⁻¹) = 3056, 3011, 2962, 2924, 2866, 2841, 1837 (*GaH*), 1813 (*SbH*), 1555, 1525, 1504, 1462, 1438, 1396, 1360, 1319, 1291, 1262, 1251, 1231, 1178, 1152, 1100, 1056, 1019, 934, 864, 795, 755, 723, 710, 632, 603, 589, 549, 519, 472, 438.

Synthesis of {(Dipp₂NacNac)GaH(BiMe)}₂(2). The following synthesis was performed under exclusion of light. A cold-trap with two stopcocks was filled with 0.4 g (0.82 mmol, 1 eq) (Dipp₂NacNac)Ga and 10 mL toluene. It was connected to a two-necked Schlenk flask with a bent transition piece of glassware and the flask was filled with 3 g LiAlH₄ and 150 mL nBu₂O and cooled to -65 °C. The apparature was set under a vacuum of ca. 1•10⁻² mbar before a suspension of 7 g MeBiCl₂ in nBu₂O was added to the LiAlH₄ solution in portions. After all MeBiCl₂ was added to the solution it was allowed to warm up to -45 °C and after 1 h the evolution of gas was observed and the Schlenk flask turned black. The gas was collected in the cooling trap at -196 °C and the formed H₂ removed *in vacuo* from the apparature. The cooling trap was separated from the apparature when the evolution of gas had stopped. It was warmed to -78 °C and flushed with argon. The resulting brownish-black suspension was allowed to warm up to room temperature before the toluene was removed in vacuo and the remaining solid extracted with 20 mL *n*-heptane and centrifuged. An *in situ* ¹H NMR spectrum confirmed that the resulting yellowish brown solution contained 2 and remaining amounts of (Dipp₂NacNac)Ga (which was used in excess because the exact amount of formed MeBiH₂ cannot be determined beforehand) such as formed (Dipp₂NacNac)GaH₂. The latter were removed by their selective crystallization from the solution at -32 °C. This process was repeated until red blocks of 2 in a yield of 108 mg (0.076 mmol, 9%, referred to the whole amount of used (Dipp₂NacNac)Ga) could be obtained after washing them three times with cold *n*-heptane. A satisfactory elemental analysis of compound **2** could not be obtained since compound 2 decomposes very rapidly at room temperature or removal of the mother liquor.

¹**H NMR** (300 MHz, C₆D₆) δ (ppm) = 1.06 (d, 6H, ³J_{H-H} = 6.8 Hz, CH(CH₃)₂), 1.14-1.16 (m, 12H, CH(CH₃)₂, Bi(CH₃)), 1.18-1.22 (m, 12H, CH(CH₃)₂), 1.30 (d, 6H, ³J_{H-H} = 6.8 Hz, CH(CH₃)₂), 1.37 (d, 6H, ³J_{H-H} = 6.8 Hz, CH(CH₃)₂), 1.52 (d, 6H, ³J_{H-H} = 6.9 Hz, CH(CH₃)₂), 1.56 (s, 6H, β -CH₃),

1.60 (s, 6H, β -C*H*₃), 3.21-3.59 (m, 8H, C*H*(CH₃)₂), 4.87 (s, 2H, γ -C*H*), 6.96-7.10 (m, 12H, Aryl-*H*), 11.37 (s, 2H, Ga*H*). **IR** (ATR) ν (cm⁻¹) = 3059, 2960, 2922, 2864, 1784 (*GaH*), 1550, 1521, 1459, 1436, 1384, 1317, 1260, 1179, 1096, 1057, 1018, 936, 858, 794, 719, 708, 638, 598, 575, 515, 432.

Synthesis of {(Dipp₂NacNac)GaCl}₂(BiMe) (3). 0.036 g (0.13 mmol, 1 eq) of MeBiCl₂ were added to a solution of 0.126 g (0.26 mmol, 2 eq) (Dipp₂NacNac)Ga in toluene at 0 °C. The solution turned orange-brown within an hour and the solvent was removed *in vacuo*, extracted with 20 mL *n*-heptane and centrifuged. After removing the solvent, **3** was obtained as an orange solid in a yield of 0.143 g (0.11 mmol, 87%). Crystals suitable for structure determination were obtained by crystallizing from heptane.

Elemental analysis calcd. (%) for C₅₉H₈₅BiCl₂Ga₂N₄: C, 55.81; H, 6.75; N, 4.41. Found: C 55.32; H, 6.836; N, 4.59. ¹H NMR (300 MHz, C₆D₆) δ (ppm) = 0.12 (s, 3H, Bi(C*H*₃)), 0.95-0.99 (m, 12H, CH(C*H*₃)₂), 1.17-1.23 (m, 12H, CH(C*H*₃)₂), 1.27 (d, 6H, ³*J*_{H-H} = 6.9 Hz, CH(C*H*₃)₂), 1.49-1.51 (m, 18H, CH(C*H*₃)₂, β -C*H*₃), 3.10-3.27 (m, 4H, C*H*(CH₃)₂), 3.61 (hept, 2H, ³*J*_{H-H} = 6.6 Hz, C*H*(CH₃)₂), 3.90 (hept, 2H, ³*J*_{H-H} = 6.7 Hz, C*H*(CH₃)₂), 4.92 (s, 2H, *γ*-C*H*), 6.91-7.14 (m, 12H, Aryl-*H*), 11.37. ¹³C{¹H} NMR (75.5 MHz, C₆D₆) δ (ppm) = 24.0, 24.2, 24.6, 24.8, 25.1, 27.7, 28.0, 28.3, 28.9, 29.5, 30.6 (C(CH₃), CH(CH₃)₂, Bi(CH₃)), 98.6 (*γ*-CH), 123.5, 123.7, 125.5, 125.6, 127.5, 141.7, 142.0, 142.9, 143.0, 146.4 (Aryl-*C*), 168.5, 168.7 (NCCH₃). IR (ATR) *ν* (cm⁻¹) = 3059, 2956, 2927, 2866, 1551, 1521, 1459, 1435, 1382, 1362, 1315, 1255, 1176, 1147, 1099, 1019, 936, 860, 796, 758, 707, 638, 529, 441, 427, 417.

Synthesis of {(Dipp₂NacNac)GaCl}₂(BiPh) (4). To a solution of 0.037 g (0.08 mmol, 2 eq) (Dipp₂NacNac)Ga in toluene 0.014 g PhBiCl₂ (0.04 mmol, 1 eq) was added of at 0 °C. The solution turned orange within an hour and the solvent was removed *in vacuo*, extraced with *n*-heptane and centrifuged. Yellow blocks of **4** in a mixture with formed (Dipp₂NacNac)GaCl₂ were obtained in a total yield of 0.021 g (0.016 mmol, 39%) at -32 °C.

¹**H NMR** (300 MHz, C₆D₆) δ (ppm) = 0.80 (d, 6H, ³*J*_{H-H} = 6.8 Hz, CH(C*H*₃)₂), 0.94 (d, 6H, ³*J*_{H-H} = 6.7 Hz, CH(C*H*₃)₂), 0.98 (d, 6H, ³*J*_{H-H} = 6.7 Hz, CH(C*H*₃)₂), 1.12-1.17 (m, 12H, CH(C*H*₃)₂), 1.25-1.28 (m, 12H, CH(C*H*₃)₂), 1.36 (d, 6H, ³*J*_{H-H} = 6.6 Hz, CH(C*H*₃)₂), 1.43 (s, 6H, β -C*H*₃), 1.51 (s, 6H, β -C*H*₃), 2.95-3.13 (m, 4H, C*H*(CH₃)₂), 3.58-3.73 (m, 4H, C*H*(CH₃)₂), 4.92 (s, 2H, γ -C*H*), 6.39 (t, 2H, ³*J*_{H-H} = 7.6 Hz, Aryl-*H*), 6.80 (t, 1H, ³*J*_{H-H} = 7.4 Hz, Aryl-*H*), 6.86 (dd, 2H, ³*J*_{H-H} = 7.4 Hz, ⁴*J*_{H-H} = 1.3 Hz, Aryl-*H*), 7.16-7.25 (m, 12H, Aryl-*H*).

Synthesis of {(Dipp₂NacNac)GaCl(BiMes)}₂ **(5).** To a solution of 0.057 g (0.12 mmol, 2 eq) (Dipp₂NacNac)Ga in toluene 0.024 g (0.06 mmol, 1 eq) of MesBiCl₂ were added at 0 °C. The solution turned immediately brown and the solvent was removed *in vacuo*, extraced with *n*-4

heptane and centrifuged. A small amount red blocks of **5** suitable for single crystal X-ray diffraction were obtained next to $(Dipp_2NacNac)GaCl_2$ as main product. Further NMR spectroscopic data of **5** could not be obtained due to the overlapping and dominating signals of $(Dipp_2NacNac)GaCl_2$ in the spectra.





Figure S3. AT-IR spectrum of (Dipp₂NacNac)GaH(SbH*t*Bu) (1).



Figure S4. ¹H NMR spectrum (300 MHz, C₆D₆, 25 °C) of {(Dipp₂NacNac)GaH(BiMe)}₂(2).



Figure S5. Detail of the ¹H NMR spectrum (300 MHz, C₆D₆, 25 °C) of {(Dipp₂NacNac)GaH(BiMe)}₂(2).



Figure S6. AT-IR spectrum of {(Dipp₂NacNac)GaH(BiMe)}₂(2).





Figure S9. AT-IR spectrum of {(Dipp₂NacNac)GaCl}₂(BiMe) (3).



Figure S10. ¹H NMR spectrum (300 MHz, C₆D₆, 25 °C) of {(Dipp₂NacNac)GaCl₂(BiPh) (**4**). Due to the similar solubility, (Dipp₂NacNac)GaCl₂ (#) cannot be separated completely from the solution.



Figure S11: *t*BuSbH₂ at a) -78 °C; b) +1 °C; c) after heating up to ca. 250 °C.



Figure S12. ¹H NMR spectrum (300 MHz, C₆D₆, 25 °C) after decomposition of *t*BuSbH₂. # marks signals of *n*Bu₂O.

Crystallographic details

| | 1 | 2 |
|---|---|---------------------------|
| Empirical formula | C ₃₃ H ₅₂ GaN ₂ Sb | $C_{60}H_{90}Bi_2Ga_2N_4$ |
| <i>M</i> [g•mol⁻¹] | 668.28 | 1424.75 |
| Crystal colour and habitus | Yellow plate | Red block |
| Crystal size [mm] | 0.253 x 0.209 x 0.171 | 0.176 x 0.107 x 0.086 |
| <i>T</i> [K] | 100 | 100 |
| Crystal system | Monoclinic | Monoclinic |
| Space group (No.) | P21/n | P21/c |
| a [Å] | 10.4394(4) | 12.2670(5) |
| b [Å] | 21.1659(7) | 14.2718(6) |
| c [Å] | 15.5452(5) | 19.6277(9) |
| β[°] | 100.403(2) | 99.647(2) |
| V [Å ³] | 3378.4(2) | 3387.7(3) |
| Ζ | 4 | 2 |
| D_{calc} [g•cm ⁻³] | 1.3138 | 1.397 |
| μ[mm ⁻¹] | 1.619 | 6.000 |
| F(000) | 1382.9 | 1412.0 |
| 2θ range for data | 3.84 to 52 | 4.21 to 57.782 |
| collection [°] | | |
| Reflections collected | 98202 | 130245 |
| Independent reflections | 6629 | 8895 |
| $R_{\rm int}, R_{\sigma}$ | 0.0348, 0.0313 | 0.0674, 0.0348 |
| Data/restraints/parameters | 6629/2/364 | 8895/1/322 |
| R_1 [$l>2\sigma(l)$, all data] | 0.0224, 0.0246 | 0.0566, 0.0744 |
| wR_2 [$I > 2\sigma(I)$, all data] | 0.0622, 0.0629 | 0.1549, 0.1640 |
| S (all data) | 1.071 | 1.044 |
| $\Delta \rho_{\text{final}}$ (max/min) [e•Å ⁻³] | 0.64/-0.44 | 7.31/-1.31 |
| CCDC | 2125090 | 2125093 |

 Table S1: Crystallographic data of compounds 1-3.

 Table S2: Crystallographic data of compounds 4 and 5.

| | 3 | 4 | 5 |
|---|--|--|----------------------|
| Empirical formula | C ₅₉ H ₈₅ BiCl ₂ Ga ₂ N ₄ | C ₆₄ H ₈₇ BiCl ₂ Ga ₂ N ₄ | C76H104Bi2Cl2Ga2N4 x |
| | | | 2 toluene |
| <i>M</i> [g•mol ⁻¹] | 1269.62 | 1331.76 | 1886.19 |
| Crystal colour and habitus | Orange blocks | Yellow blocks | Red block |
| Crystal size [mm] | 0.224 x 0.129 x 0.111 | | |
| <i>T</i> [K] | 100 | 100 | 100 |
| Space group | Triclinic | Triclinic | Monoclinic |
| Crystal system (No.) | PĪ | PĪ | P21/c |
| a [Å] | 11.9815(6) | 14.3525(11) | 16.3919(16) |
| b [Å] | 19.7185(8) | 15.4208(13) | 18.5682(13) |
| c [Å] | 25.2053(12) | 17.6984(14) | 13.807(11) |
| α [°] | 97.837(2) | 105.047(6) | |
| β[°] | 93.446(2) | 100.889(6) | 90.248(7) |
| γ [°] | 96.306(2) | 98.819(7) | |
| V [Å ³] | 5846.5(5) | 3629.2(5) | 4200.8(6) |
| Ζ | 4 | 2 | 2 |
| D _{calc} [g•cm ⁻³] | 1.442 | 1.2186 | 1.491 |
| μ[mm ⁻¹] | 4.048 | 3.264 | 4.920 |
| F(000) | 2584.0 | 1350.1 | 1900.0 |
| 2θ range for data | 3802 to 56.946 | 3.12 to 58.36 | 3.314 to 58.418 |
| collection [°] | | | |
| Reflections collected | 226883 | 58852 | 51477 |

| Independent reflections | 29466 | 19467 | 11288 |
|---|----------------|----------------|----------------|
| $R_{\text{int}}, R_{\sigma}$ | 0.0675, 0.0505 | 0.0361, 0.0428 | 0.0445, 0.0472 |
| Data/restraints/parameters | 29466/0/1314 | 19467/0/678 | 11288/0/465 |
| R_1 [<i>I</i> >2 σ (<i>I</i>), all data] | 0.0294, 0.0474 | 0.0276, 0.0364 | 0.0270, 0.0426 |
| wR_2 [$l>2\sigma(l)$, all data] | 0.0594, 0.0618 | 0.0681, 0.0702 | 0.0594, 0.0612 |
| S (all data) | 1.066 | 0.930 | 0.963 |
| $\Delta \rho_{\text{final}}$ (max/min) [e•Å ⁻³] | 0.66/-0.72 | 1.46/-0.61 | 1.98/-0.69 |
| CCDC | 2125094 | 2125091 | 2125092 |



Figure S11. Molecular structure of **1**. All hydrogen atoms bonding to carbon are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Antimony atoms were refined with split positions 0.75 : 0.25 (Sb1, Sb1'). Selected bond length [Å] and angles [°]: Ga1-Sb1 2.6107(14), Ga1-Sb1' 2.626(4), Ga1-N1 1.9690(13), Ga1-N2 1.9798(13), N1-Ga1-N2 94.72(5), Ga1-Sb1-C30 99.39(7), Ga1-Sb1'-C30 100.69(16).



Figure S12. Molecular structure of **2**. All hydrogen atoms bonding to carbon are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Selected bond length [Å] and angles [°]: Ga1-Bi1 2.7171(8), Bi1-Bi1' 3.0032(6), Ga1-N1 1.983(5), Ga1-N2 1.982(5), N1-Ga1-N2 93.5(2), N1-Ga1-Bi1 113.71(15), N2-Ga1-Bi1 115.41(15), Ga1-Bi1-Bi1' 92.77(2), Ga1-Bi1-C1 92.4(2), C1-Bi1-Bi1' 92.4(3).



Figure S13. Both independent molecules in the unit cell of **3**. All hydrogen atoms bonding to carbon are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Bismuth atoms were refined with split positions 0.95 : 0.05 (Bi1, Bi1' and Bi2, Bi2').



Figure S14. Molecular structure of one molecule of **3**. All hydrogen atoms bonding to carbon and disordered parts are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Selected bond length [Å] and angles [°]: Ga1-Bi1 2.6854(3), Ga2-Bi1 2.6782(3), Ga1-N1 1.973(2), Ga1-N2 1.962(2), Ga2-N3 1.9745(19), Ga2-N4 1.9617(19), Ga1-Cl1 2.2155(8), Ga2-Cl2 2.2094(7), Ga1-Bi1-Ga2 103.865(10), N1-Ga1-N2 94.96(9), N3-Ga2-N4 94.89(8), N1-Ga1-Bi1 102.51(6), N2-Ga1-Bi1 129.64(6), N3-Ga2-Bi1 115.71(6), N4-Ga2-Bi1 113.81(6), Cl1-Ga1-Bi1 118.87(2), Cl2-Ga2-Bi1 122.40(2), Ga1-Bi1-C59 95.25(8), Ga2-Bi1-C59 97.13(7).



Figure S15. Molecular structure of **4**. All hydrogen atoms bonding to carbon are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Selected bond length [Å] and angles [°]: Ga1-Bi1 2.7248(4), Ga2-Bi1 2.7230(3), N1-Ga1 1.975(2), N2-Ga1 1.997(2), N3-Ga2 1.9877(19), N4-Ga2 1.9588(19), Ga1-Cl1 2.2101(6), Ga2-Cl2 2.2246(7), N1-Ga1-N2 93.61(8), N3-Ga2-N4 94.28(8), Ga1-Bi1-Ga2 116.124(8), N1-Ga1-Bi1 129.48(6), N2-Ga1-Bi1 103.60(6), N3-Ga2-Bi1 107.22(6), N4-Ga2-Bi1 125.62(6), Cl1-Ga1-Bi1 118.670(19), Cl2-Ga2-Bi1 117.359(19), Ga1-Bi1-C59 97.14(6), Ga2-Bi1-C59 92.65(6).



Figure S16. Molecular structure of **5**. All hydrogen atoms bonding to carbon and solvent molecules are omitted for clarity. Carbon atoms are shown in the *wires and sticks* model. Thermal ellipsoids represent a 50% probability level. Selected bond length [Å] and angles [°]: Ga1-Bi1 2.7572(4), Bi1-Bi1' 3.0322(3), Ga1-N1 1.979(2), Ga1-N2 1.981(2), Ga1-Cl1 2.2477(7), N1-Ga1-N2 94.63(10), N1-Ga1-Cl1 101.21(7), N2-Ga1-Cl1 100.46(7), N1-Ga1-Bi1 137.04(7), N2-Ga1-Bi1 108.15(7), Cl1-Ga1-Bi1 109.45(2), Ga1-Bi1-Bi1' 97.010(11), Ga1-Bi1-C30 101.44(7), Bi1'-Bi1-C30 99.76(7).

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