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The hydrogallation of C=N double bonds — reactions of $GaH_3 \cdot NMe_2Et$ with tetramethyl-2,3-diazabutadiene

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Abstract

The reaction of 1,1,4,4-tetramethyl-2,3-diazabutadiene with $GaH_3 \cdot NMe_2Et$ yielded two different products depending on the reaction conditions. The hydrogallation of only one C=N double bond was observed at low temperature and for a 1:1 molar ratio of the starting compounds. The resulting dimeric gallium hydrazonide $[H_2Ga–N(i\mathrm{Pr})–N=CMe_2]_2$ (**4**) has a six-membered Ga_2N_4 heterocycle in its molecular centre. In contrast, N–N bonds were partially cleaved when the same reaction was carried out at room temperature with an excess of the gallane–amine adduct. The structure of the tricyclic tetragallium compound $\{(GaH)(GaH_2)(N–i\mathrm{Pr})[N(i\mathrm{Pr})–N=CMe_2]\}_2$ (**5**) isolated in low yield consists of two five-membered Ga_2N_3 heterocycles anellated to a central four-membered Ga_2N_2 ring. © 2001 Published by Elsevier Science B.V.

Keywords: Gallium; Hydrogallation; Heterocycles

1. Introduction

The hydroalumination of 2,3-diazabutadiene derivatives has been employed by our group only recently as a new method for the syntheses of hydrazine derivatives. However, special conditions are required for the complete addition of Al–H groups to both C=N double bonds. Only one equivalent of dimethylaluminium hydride reacted with 1,1,4,4-tetramethyl-2,3-diazabutadiene to afford an aluminium hydrazonide (**1**, Scheme 1) which via Al–N interactions gave a dimer possessing a six-membered Al_2N_4 heterocycle [1]. Similarly, di(*tert*-butyl)aluminium hydride gave the monoaddition product only, which probably owing to the steric shielding by the bulky *tert*-butyl groups did not form dimers. Instead, a second di(*tert*-butyl)aluminium hydride molecule is coordinated by a dative Al–N bond and a 3c-2e Al–H–Al bond to yield five-membered Al_2N_2H heterocycles (**2**, Scheme 1) [1]. Complete hydroalumination with the formation of aluminium hydrazides was finally observed when we employed the trihydrido compound $AlH_3 \cdot NMe_2Et$. Two products were isolated and characterised by crystal structure determinations. One was identified as a cage compound with a complicated structure formed by four aluminium atoms and three hydrazido groups [2]. The second product (**3**, Scheme 1) contained a macrocyclic Al_3N_6 ligand, all six nitrogen atoms of which were coordinated to a fourth aluminium atom in the molecular centre [2]. Such aluminium and gallium hydrazido derivatives found considerable interest in recent literature, because they might be suitable for the generation of aluminium or gallium nitride by thermolysis [3]. In contrast to hydroalumination [4], the addition of Ga–H bonds (hydrogallation) to heteronuclear E=C double bonds has been investigated to a far lesser extent. About 10 years ago, two reports dealt with reactions of $GaH_3 \cdot NMe_3$ with 1,4-diazabutadienes, which afforded ethylenediamine derivatives [5]. We were interested in the reactivity of GaH_3 toward 2,3-diazabutadienes in order to synthesise gallium hydrazine derivatives analogous to the aluminium compounds described above (e.g. **3**) containing more than one gallium atom per hydrazido group.

2. Reactions of $GaH_3 \cdot NMe_2Et$ with 1,1,4,4-tetramethyl-2,3-diazabutadiene

Equimolar quantities of $AlH_3 \cdot NMe_2Et$ and 1,1,4,4-tetramethyl-2,3-diazabutadiene gave mixtures of un-

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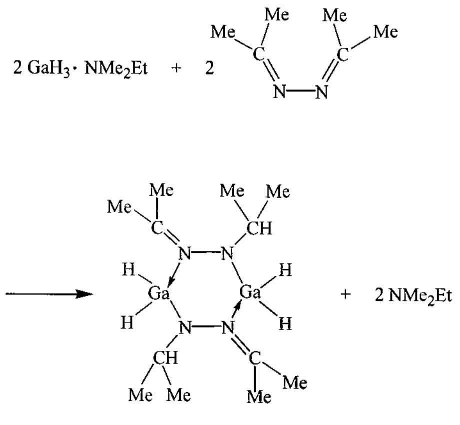
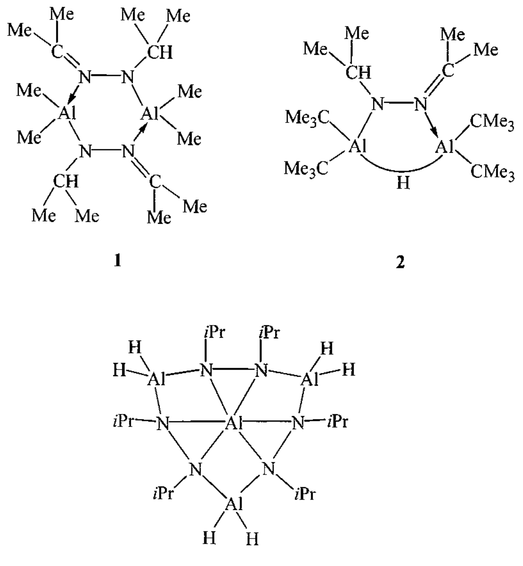
known products only, and the successful synthesis of compound **3** or the isomeric cage compound $(AlH_2)_2(AlH)_2(N_2i Pr_2)_3$ required the thorough optimisation of the molar ratio of the starting materials [2]. In contrast, equimolar quantities of the gallane adduct $GaH_3{\cdot}NMe_2Et$ and the diazabutadiene yielded one main product (**4**, Eq. (1)), which was isolated in 67% yield after crystallisation from a mixture of *n*-hexane and toluene. As a by-product we isolated compound **5** in trace amounts as the less soluble first fraction. Compound **5** was synthesised on a more specific route as described below. The ^{1}H-NMR spectrum of **4** shows a broad resonance of two hydrogen atoms attached to gallium ($\delta = 5.23$), a septet and a doublet characteristic of an isopropyl group and two singlets of two chemically different methyl groups. These observations are in accordance with a hydrazonido structure similar to that of **1**, in which only one Ga–H group was added to a C=N double bond. Compound **4** is readily soluble in non-polar organic solvents and is moderately stable as a crystalline solid at room temperature. It decomposes rapidly at about 42 °C to yield a mixture of many unknown products. Compound **5** was not detected as a constituent of that mixture by NMR spectroscopy.



4

(1)

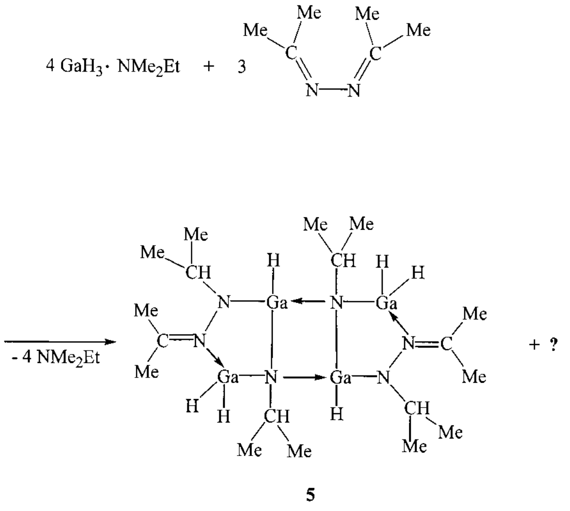


1 **2**

3

Scheme 1.

The by-product **5** showed a complicated ^{1}H-NMR spectrum with two chemically different isopropyl and methyl groups. Two very broad resonances were observed for hydrogen atoms attached to gallium ($\delta = 5.75$ and 5.60; integration ratio 1:2). The constitution of **5** was clarified by a crystal structure determination (see below). It is a dimer in the solid state possessing two hydrazonido ligands and two isopropylimido groups. It has four gallium atoms, two of which are attached to only one hydrogen atom, while the remaining ones are bonded to two hydrogen atoms. Owing to the molecular symmetry (C_i) the methyl groups of all isopropyl substituents become diastereotopic, and one pseudo-septet and two doublets are observed for each kind of chemically identical isopropyl groups in the ^{1}H-NMR spectrum. The ratio of three intact or cleaved N–N bonds to four gallium atoms in the molecular structure led us to treat the diazabutadiene with an excess of the hydride (molar ratio 3:4, Eq. (2)). We now succeeded in isolating **5** in a still low, but better yield of 9%. A grey, amorphous, and insoluble powder precipitated from the reaction mixture, and a small quantity of a gaseous product evolved. Compound **5** is only slightly more stable than compound **4** and decomposes rapidly above 60 °C. As was shown before, compound **5** is not the product of the thermal decomposition of **4**. It may be formed by the complete hydrogallation of part of the diazabutadiene. The intermediate hydrazido derivative may be unstable and may decompose by cleavage of its N–N bond to generate the imido groups present in **4**. Systematic variation of the reaction conditions did not give a better yield of **5**. Furthermore, we were not able to detect a product similar to those isolated from the corresponding reaction of the alane–amine adduct.



5

(2)

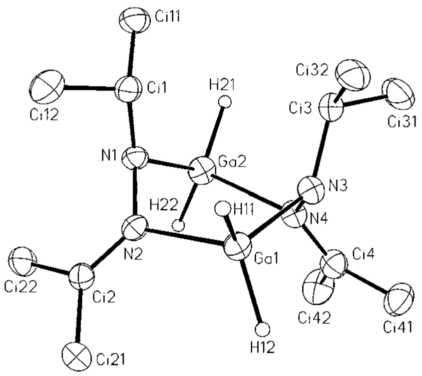


Fig. 1. Molecular structure and numbering scheme of **4**; the thermal ellipsoids are drawn at the 40% probability level; hydrogen atoms are omitted with the exception of those attached to gallium (arbitrary radius). Selected bond lengths (pm) and angles (°): Ga1–N2 206.8(2), Ga1–N3 191.6(2), Ga2–N1 192.0(2), Ga2–N4 207.0(2), N1–N2 144.0(3), N3–N4 144.2(3), N1–Ci1 147.7(3), N2–Ci2 127.4(3), N3–Ci3 146.4(3), N4–Ci4 128.6(4), N2–Ga1–N3 100.61(9), Ga1–N3–N4 107.7(2), N3–N4–Ga2 114.8(2), N4–Ga2–N1 100.6(1), Ga2–N1–N2 108.4(2), N1–N2–Ga1 115.4(2).

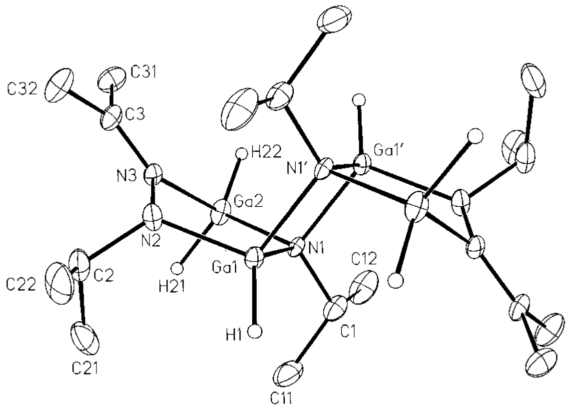


Fig. 2. Molecular structure and numbering scheme of **5**; the thermal ellipsoids are drawn at the 40% probability level; hydrogen atoms attached to gallium with arbitrary radius, remaining hydrogen atoms omitted. Selected bond lengths (pm) and angles (°): Ga1–N1 197.1(3), Ga1–N1′ 199.0(3), Ga1–N2 190.9(3), Ga2–N1 195.3(3), Ga2–N3 204.8(3), N2–N3 143.1(5), N1–C1 149.4(5), N2–C2 148.2(5), N3–C3 128.6(5), N1–Ga1–N1′ 89.8(1), N2–Ga1–N1 102.3(1), N2–Ga1–N1′ 107.0(1), Ga1–N1–Ga1′ 90.2(1), Ga1–N1–Ga2 106.7(2), Ga1′–N1–Ga2 110.6(1), N1–Ga2–N3 95.1(1), Ga2–N3–N2 114.3(2), N3–N2–Ga1 111.3(2); N1′ and Ga1′ generated by $-x+1$, $-y+1$, $-z$.

3. Crystal structures of 4 and 5

The molecular structure of **4** (Fig. 1) comprises a six-membered heterocycle, in which two endocyclic N–N single bonds are bridged by two GaH_2 groups. One nitrogen atom of each N_2 moiety (N1, N3) is attached to an isopropyl group, while the second one (N2, N4) is part of a C=N double bond. Compound **4** is thus formed by the addition of one Ga–H group to one C=N double bond of the diazabutadiene derivative. Dimers are formed by the interaction between the imine nitrogen atom and the coordinatively unsaturated gallium atom of a second molecule. The Ga–N distances differ strongly in accordance with their different bonding modes, which have been classified as normal covalent and dative [6]. Short distances of 191.8 pm on average are observed to the negatively charged isopropylamido nitrogen atoms, while longer ones (206.9 pm) are detected to the imine nitrogen atoms. The C=N bond lengths (128.0 pm) correspond to the standard value [7] and are similar to those observed in organic hydrazone derivatives [8]. In contrast, the N–N distances of **4** are significantly lengthened (144.1 pm compared to < 138 pm). Corresponding distances were found in some organo aluminium and gallium hydrazonides which were published only recently and which were obtained on quite another route [9]. The Ga–H bond lengths (150 pm) are in the characteristic range of terminal Ga–H bonds [10]. The six-membered heterocycle adopts a twist-boat conformation.

The structure of **5** (Fig. 2) comprises a four-membered, ideally planar Ga_2N_2 heterocycle in the molecular centre. Both gallium atoms are further attached to one hydrogen atom and one amido nitrogen atom of a hydrazonido ligand, and each nitrogen atom of the heterocycle is further bonded to an isopropyl and a GaH_2 group. These gallium atoms become coordinatively saturated by an interaction with the imine nitrogen atoms of the hydrazonido ligands. Two five-membered Ga_2N_3 heterocycles result, which adopt an envelope conformation with the atom N3 50 pm above the plane of the remaining atoms (Ga1, N1, Ga2, N2). Overall, a ladder-type structure is formed possessing three anellated rings. Their normals include angles of 109.4° (excluding N3). The Ga–N bond lengths are 197.1 (Ga1–N1) and 199.0 pm (Ga1–N1′) in the Ga_2N_2 heterocycle, 190.9 pm (Ga1–N2) to the negatively charged amido nitrogen atoms of the hydrazonido ligands and 195.3 pm to the GaH_2 groups (Ga2–N1). As expected, the distances between the imino nitrogen atoms and gallium (Ga2–N3) are lengthened to 204.8 pm (dative bond [6]). N–N (143.1 pm) and N=C bond lengths (128.6 pm) are similar to those discussed above.

4. Experimental

All procedures were carried out under purified Ar in dried solvents (toluene over Na/benzophenone; *n*-pentane and *n*-hexane over $LiAlH_4$). Commercially available 1,1,4,4-tetramethyl-2,3-diazabutadiene (Aldrich) and $GaH_3 \cdot NMe_2Et$ (MOCHEM, Marburg, Germany) were employed without further purification.

4.1. Synthesis of $[H_2Ga{-}N(iPr){-}N{=}CMe_2]_2$ **4**

A solution of 2.34 g (0.016 mol) of $GaH_3{\cdot}NMe_2Et$ in 5 ml of toluene was added dropwise to a cooled (−50 °C) solution of 1,1,4,4-tetramethyl-2,3-diazabutadiene (2.1 ml, 1.79 g, 0.016 mol) in 20 ml of *n*-pentane. The mixture was warmed to 10 °C over a period of 6 h. At that temperature, all volatile components were distilled off in vacuum. The solid residue was dissolved in a mixture of *n*-hexane with a small quantity of toluene. Trace amounts of compound **5** crystallised upon cooling to +5 °C. Concentration and cooling of the mother liquor to −15 °C afforded colourless crystals of **4**. Yield: 1.98 g (67%) — M.p. (dec.) (Ar, sealed capillary): 42 °C — Molar mass (in benzene, by cryoscopy): obs. 380, calc. 369.8 g mol^{-1} — EIMS; *m*/*z* (relative intensity): 370.4 (<1% [M^+] $^{69}Ga^{71}Ga$), 253.1, 255.2, 257.2 (6%, $[GaNC(H)Me_2]_2H$), 181.1, 183.1 (19%, $Ga(NCMe_2)_2$) — ^{1}H-NMR (C_6D_6, 300 MHz, 300 K): $\delta = 5.23$ (4H, s, br., GaH_2), 3.91 (2H, septet, $^3J_{HH} = 6$ Hz, CH of *i*Pr), 1.89, 1.78 (each 6H, s, $N{=}CMe_2$), 1.28 (12H, d, $^3J_{HH} = 6$ Hz, Me of *i*Pr) — ^{13}C-NMR (C_6D_6 75.5 MHz, 300 K): $\delta = 145.7$ (N=C), 50.8 (NC(H)), 23.4 (Me of *i*Pr), 24.5, 21.9 (Me of $N{=}CMe_2$) — IR (CsBr, paraffin): $\tilde{\nu} = 1906$ vs, 1872 vs, 1852 vs νGaH; 1661 w, 1613 vs νC=N; 1465 vs, 1378 vs paraffin; 1342 vs, 1316 vs, 1262 vs δCH_3; 1210 vw, 1169 vs, 1121 vs, 1114 vs, 1083 s, 1001 s, 978 s, 941 w, 920 w, 858 w, 830 w νCC, νCN, νNN; 720 br., vs, 694 vs δGaH_2; 616 s, 594 vs, 573 vs, 531 vs, 518 vs, 474 vs, 464 vs νGaC, νGaN; 417 w, 366 m, 306 s δCC.

Table 1
Crystal data and structure refinement parameters for **4** and **5** [a]

	4	**5**
Empirical formula	$C_{12}H_{30}Ga_2N_4$	$C_{18}H_{46}Ga_4N_6$
Temperature (K)	193(2)	193(2)
Crystal system	Orthorhombic	Monoclinic
Space group [11]	$P2_12_12_1$; No. 19 [b]	$P2_1/n$; No. 14
Unit cell dimensions		
a (pm)	805.8(1)	848.0(1)
b (pm)	1459.7(1)	1067.7(2)
c (pm)	1534.1(1)	1550.6(3)
β (°)	90	90.83(1)
V (10^{-30} m^3)	1804.4(3)	1403.8(4)
Z	4	2
D_{calc} (g cm^3)	1.361	1.480
μ (mm^{-1})	2.978	3.812
Crystal size (mm)	0.7 × 0.3 × 0.3	0.5 × 0.5 × 0.2
Diffractometer	CAD-4	CAD-4
Radiation	Mo–K_α	Mo–K_α
θ range (°)	2.66–25	2.75–25
Index ranges	$-9 \le h \le 9$, $-17 \le k \le 17$, $-18 \le l \le 18$	$-10 \le h \le 10$, $0 \le k \le 12$, $0 \le l \le 18$
Independent reflections	3174 [$R_{int} = 0.0233$]	2298 [$R_{int} = 0.0244$]
Reflections $I > 2\sigma(I)$	3127	1792
Parameters	187	145
$R = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\| (I > 2\sigma(I))$	0.0244	0.0361
$wR_2 = \{\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$ (all data)	0.0667	0.0947
Max/min residual electron density (10^{30} e m^{-3})	0.275/−0.545	0.423/−0.479

[a] Programmes SHELXL-97; SHELXTL-PLUS [12]; solutions by direct methods, full-matrix refinement with all independent structure factors.

[b] Flack parameter 0.01(12).

4.2. Synthesis of $\{(GaH)(GaH_2)(N{-}iPr)[N(iPr){-}N{=}CMe_2]\}_2$ **5**

A solution of 4.52 g (0.031 mmol) of $GaH_3{\cdot}NMe_2Et$ in 10 ml of toluene was added dropwise to a cooled (−30 °C) solution of 1,1,4,4-tetramethyl-2,3-diazabutadiene (3.1 ml, 2.58 g, 0.023 mol) in 10 ml of toluene. The mixture was warmed to room temperature over a period of 3 h and further stirred for 3 h. All volatile components were distilled off in vacuum. The residue was treated with a mixture of *n*-hexane and toluene. After filtration and cooling to −15 °C a colourless solid of **5** was obtained. Yield: 0.435 g (9%). M.p. (dec.) (Ar, sealed capillary): 60 °C — EIMS; *m*/*z* (relative intensity): 624.4, 626.5 (2% [M^+]), 507.3, 509.3, 511.3, 513.3, 515.3 (74%, $Ga_4H_3[NiPr]_4$), 253.1, 255.1, 257.1 (100%, $[HGaNiPr]_2H$) — ^{1}H-NMR (C_6D_6, 300 MHz, 300 K): $\delta = 5.75$ (2H, s, very broad, GaH), 5.60 (4H, s, br., GaH_2), 3.37, 2.92 (each 2H, pseudo-septet, $^3J_{HH} = 6$ Hz, NCH of *i*Pr), 1.83, 1.64 (each 6H, s, $N{=}CMe_2$), 1.60, 1.39, 1.33, 1.32 (each 6H, d, $^3J_{HH} = 6$ Hz, diastereotopic Me of *i*Pr) — ^{13}C-NMR (C_6D_6, 75.5 MHz, 300 K): $\delta = 176.3$ (N=C), 57.2, 50.4 (NC of *i*Pr), 30.0, 25.4, 25.1, 24.9, 23.8, 21.7 (methyl groups) — IR (CsBr, paraffin): $\tilde{\nu} = 1898$ m, 1883 m, 1857 s νGaH; 1596 m νC=N; 1460 vs, 1378 vs paraffin; 1361 s, 1341 m, 1316 w, 1264 w δCH_3; 1159 m, 1147 m, 1124 m, 1088 w, 1022 w, 965 m, 947 w, 912 w, 825 m, 767 m, 732 s, 709 s νCC, νCN, νNN; 633 s, br. δGaH; 593 s, 576 s, 516 w, 492 w, 451 w νGaC, νGaN; 411 w, 353 vw, 306 w δCC.

4.3. Crystal structure determinations

Single crystals of compounds **4** and **5** were obtained by recrystallisation from mixtures of *n*-hexane and toluene. Crystal data and structure refinement parameters are given in Table 1.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic

Data Centre, CCDC nos. 164424 and 164425 for compounds **4** and **5**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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