Octagallane ($(tBu_3Si)_6Ga_8$) and Its Reduction to $(tBu_3Si)_6Ga_8^{2-}$ − On the Existence of Isomeric Gallium Clusters[‡]

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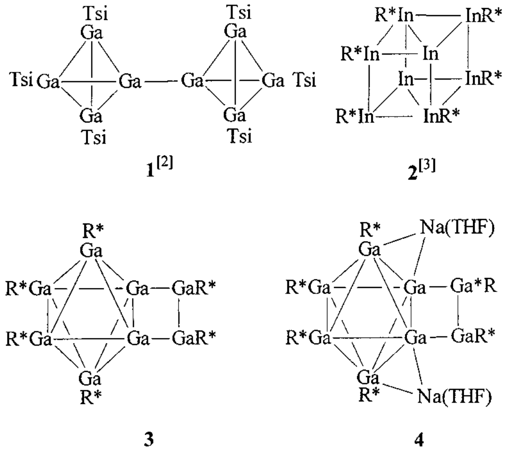
Thermolysis of the trigallanyl radical $R^*_4Ga_3^{\cdot}$ in heptane at 60 °C leads to the dark blue octagallane $R^*_6Ga_8$ (R^* = supersilyl SitBu$_3$), as well as the digallanyl radical $R^*_3Ga_2^{\cdot}$ and the *tetrahedro*-tetragallane $R^*_4Ga_4$. In addition, supersilyl radicals $R^{*\cdot}$ are formed which stabilize themselves either by dimerization or by addition of hydrogen atoms. $R^*_6Ga_8$ can be reduced in THF with $NaC_{10}H_8$ to the dark-red octagallanediide $Na_2Ga_8R^*_6 \cdot 2THF$. According to an X-ray structure analysis of $R^*_6Ga_8$ and $R^*_6Ga_8^{2-}$ one finds that the Ga atoms of four R*Ga moieties, together with two "naked" Ga atoms, occupy the corners of a distorted octahedron; the "naked" Ga atoms themselves are located, along with Ga atoms of two further R*Ga moieties, at the corners of a distorted square. The reduction of $R^*_6Ga_8$ leads only to a negligible shortening of the Ga–Ga distances from 2.64 to 2.61 Å (mean values) in $R^*_6Ga_8^{2-}$. Both the octagallanes possess Ga_8 frameworks previously unknown for group 13 clusters. They are isomeric to the recently described Ga_8 framework of the octagallane Tsi_6Ga_8 [Tsi = trisyl $C(SiMe_3)_3$]. Hence, not only boron but also the heavier group 13 atoms form isomeric clusters.

Introduction

Cluster compounds R_mE_n of the heavier group 13 metals Al, Ga, In, Tl — unlike those of B — became accessible only recently when it was realized that the tendency of R_mE_n to disproportionate into R_3E and E (or R_2 and E) can be stopped by employing bulky substituents such as trisyl (Tsi) [$C(SiMe_3)_3$], hypersilyl (Hsi) [$Si(SiMe_3)_3$] or supersilyl (R*) [$Si(CMe_3)_3$]. In fact, numerous clusters of Ga — the subject of this publication — have been isolated within the last few years (for more than 30 gallium cluster compounds see ref.[1]).

Schnöckel and co-workers[2] recently reported the preparation of hexatrisyloctagallane Tsi_6Ga_8 (**1**) with a framework of two *tetrahedro*-tetragallanyl moieties connected by a Ga−Ga bond (see Scheme 1). We now report the isolation of hexasupersilyloctagallane $R^*_6Ga_8$ (**3**) as well as its reduction product disodium hexasupersilyloctagallanediide-tetrahydrofuran(1/2) $Na_2Ga_8R^*_6 \cdot 2THF$ (**4**), the Ga_8 frameworks of which are isomeric with the Ga_8 cluster of Tsi_6Ga_8 (Scheme 1). It is worth mentioning here that the In_8 framework of hexasupersilyloctaindane $R^*_6In_8$ (**2**; Scheme 1), reported some years ago by us,[3] has a structure different to the structures of Tsi_6Ga_8 and $R^*_6Ga_8$.



1[2] **2**[3] **3** **4**

Scheme 1. Compounds with isomeric group 13 E_8 cluster frameworks [Tsi = $C(SiMe_3)_3$, R* = SitBu$_3$]

Results and Discussion

Syntheses of $R^*_6Ga_8$ (3) and $Na_2Ga_8R^*_6 \cdot 2THF$ (4)

The dark green radical $R^*_4Ga_3^{\cdot}$ (**5**), which may be synthesized, via isolable intermediates, from gallium trichloride $GaCl_3$, supersilyl sodium NaR*, and supersilyl bromide R* Br,[4] slowly decomposes in heptane at 40 °C with formation of the dark blue radical $R^*_3Ga_2^{\cdot}$ (**6**) and the dark violet *tetrahedro*-tetragallane $R^*_4Ga_4$ (**7**) [see Scheme 2, Equation

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[‡] Compounds of Silicon and Homologues, 152; Compounds of Boron and Homologues, 19. − Parts 151 and 18: Ref.[7]

[‡‡] X-ray structure analyses

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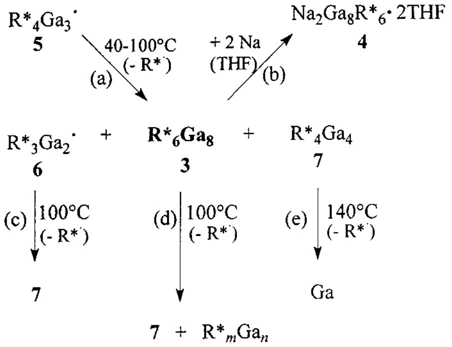
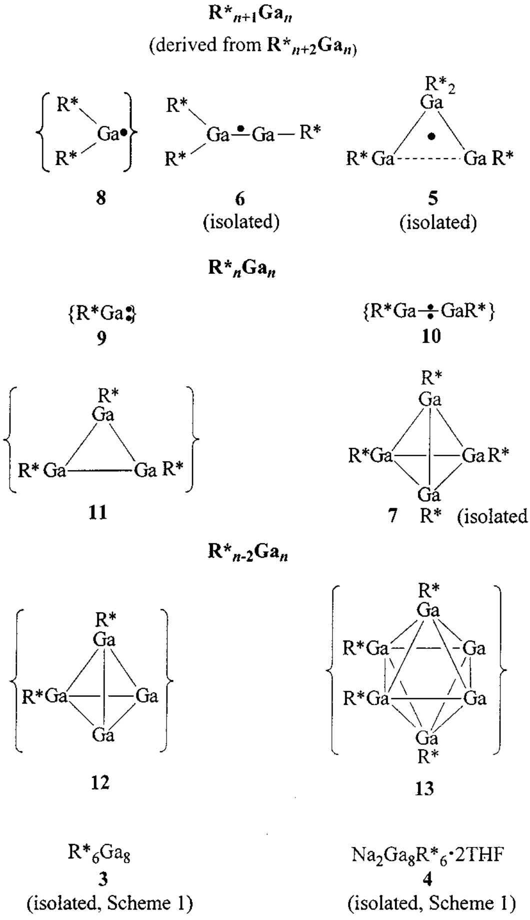
(a); for the structures see Scheme 3].[4] The radical **6** transforms quantitatively at 100 °C in heptane into the tetrahedrane **7** [Scheme 2, Equation (c)] which decomposes as a solid at 322 °C and in solution at 140 °C to metallic gallium [Scheme 2, Equation (e)].[5] The supersilyl radicals $R^{*\cdot}$ obtained in reactions (c) and (e) dimerize to give superdisilane molecules R^*_2, which at higher temperatures transform via $R^{*\cdot}$ into supersilane R^*H.[6]



Scheme 2. Decomposition of **5** (the produced supersilyl radical R* stabilizes with formation of R^*_2 and/or R*H; R* = Si*t*Bu$_3$)

The thermolysis of **5** in addition leads to the octagallane $R^*_6Ga_8$ (**3**) [Scheme 2, Equation (a); maximum yields at about 60 °C], which itself decomposes in benzene at 100 °C to give the tetrahedrane **7** (main product) as well as unidentified cluster compounds R_mGa_n [Scheme 2, Equation (d)]. The octagallane **3** can be reduced in THF with $NaC_{10}H_8$ to give the octagallanediide $Na_2Ga_8R^*_6\cdot 2THF$ (**4**) [Scheme 2, Equation (b)]. Reduction of the tetragallane **7** in THF with sodium occurs analogously to produce the tetragallanediide $Na_2Ga_4R^*_4\cdot 2THF$.[7]

To date our insight into the pathways of the formation and thermolysis of **3** is still incomplete. It is possible that compounds **8**−**13**, which are shown in Scheme 3 along with the isolated products **3**−**7**, may play the role of intermediates (cf. ref.[4] for formation of **3**, and the following for thermolysis of **3**). Radical **5** can react with cleavage of Ga−Ga bonds to form **6** and **9**, as well as **8** and **10**, and with cleavage of a Ga−Si bond to form **11** and $R^{*\cdot}$; radical **8**, after dimerization and R* elimination, transforms into isolable **6** (cf.[4]), and radical **6**, after elimination of R*, into non-isolable **10**. The formed gallylene **9** and the digallene **10** tetramerize and dimerize, respectively, to give isolable **7**. It is also possible that the cyclotrigallane **11** may eliminate R* and then dimerize with formation of the hitherto non-isolated hexagallane **13** which, on adding **10**, might form the isolable octagallane **3**. This compound decomposes on gentle heating to give the isolable tetrahedrane **7** and the non-isolable tetragallane **12** which possibly stabilizes with formation of unidentified oligogallanes (see above). Support for this suggestion is based on the formation of decagallanide $R^*_6Ga_{10}^-$ [8] as an isolable adduct of **12** and **13** [see also Hsi_6Ga_{10}, Hsi = $Si(SiMe_3)_3$ [8]] as well as the formation of dodecaindane $R^*_8In_{12}$ [9] from two molecules of the In analog of **13** (Hexaindane $R^*_4In_6$ — an obvious intermediate in the formation of $R^*_8In_{12}$ — has been observed by NMR spectroscopy[9].) In addition, a cluster formed from two equivalents of **12** with In/Dmp instead of Ga/R* has been synthesized[10] (Dmp = 2,6-$Mes^*_2C_6H_3$ with Mes* = 2,4,6-$tBu_3C_6H_2$). The preparation and isolation of hexagallane **13** is the next challenge for group 13 cluster chemists.



Scheme 3. Isolated and postulated products of the thermolysis of **5** as well as the reduction product **4** of **3** (R* = Si*t*Bu$_3$)

Characterization and Structures of $R^*_6Ga_8$ (3) and $Na_2Ga_8R^*_6\cdot 2THF$ (4)

Hexasupersilyloctagallane (**3**) crystallizes from benzene as water- and air-sensitive dark blue prisms (monoclinic, space group $P2_1/c$) which are thermolabile at elevated temperatures. Disodium hexasupersilyloctagallanediide-tetrahydrofuran(1/2) (**4**) crystallizes from benzene as extremely water- and air-sensitive dark red prisms (triclinic, space group $P\bar{1}$).

The molecular structures of the molecules **3** and **4** in the crystal are shown in Figure 1 and 2, respectively. In both compounds the Ga atoms of four R*Ga moieties occupy the corners of a distorted octahedron together with two "naked" Ga atoms. The "naked" Ga atoms are located next to each other, along with the Ga atoms of two further R* Ga moieties, at the corners of a distorted square. The Ga atoms that form the Ga_6 octahedron and the anellated Ga_4 square lie almost in a plane together with the Si atoms of the four connected R* groups. All quadrangle surfaces Ga1Ga2Ga7Ga8/Ga5Ga6Ga7Ga8/Ga3Ga5Ga4Ga7/Ga3Ga6Ga4Ga8 in **3** and **4** are planar (angles sum ca. 360°). The Si atoms connected at the apices of the Ga_6 octahedron are tilted towards the square. The two Na(THF) moieties of **4** bridge opposite edges between the Ga atoms of the apex and the central plane of the Ga_6 octahedron. Surprisingly, Na(THF) is not located above and below the Ga_4 square, probably due to steric reasons. $(Hsi)_6Ga_{10}$, which is derived from **4** after exchange of supersilyl for the sterically less crowded hypersilyl $Si(SiMe_3)_3$ and $Na(THF)^+$ by "isoelectronic" Ga^+, shows a Ga_{10} framework built from two anellated Ga_6 octahedrons. Scheme 4 shows the structural relationships between the octagallanediide $R_6Ga_8^{2-}$ (isolated as **4**) and the decagallane R_6Ga_{10} [isolated as $(Hsi)_6Ga_{10}$][8] via the nonagallanide $R_6Ga_9^-$ {isolated as $[Na(THF)_6]^+(Hsi)_6Ga_9^-$},[8] which are formally derived from $R_6Ga_8^{2-}$ by successive "neutralization" of the dianion with Ga^+ cations (it remains to be seen whether $R^*{}_6Ga_8^{2-}$ is transformed in the presence of a Ga^I compound into $R^*{}_6Ga_{10}$ via $R^*{}_6Ga_9^-$).

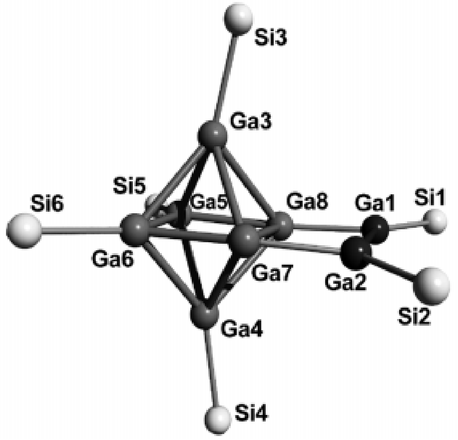


Figure 1. View of a molecule of **3** in the crystal (*t*Bu groups excluded for clarity); for bond lengths and angles see Table 1

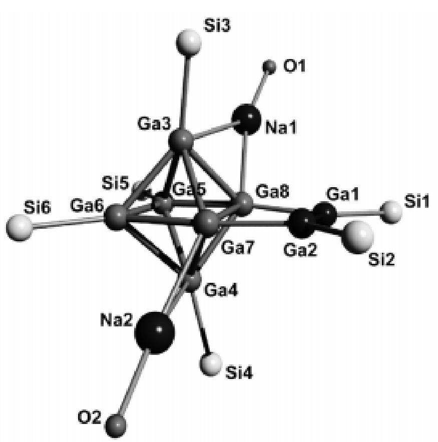
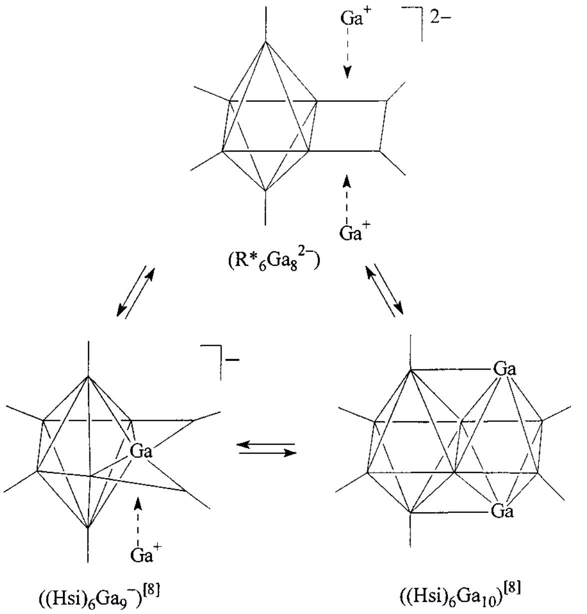


Figure 2. View of a molecule of **4** in the crystal (*t*Bu and CH_2 groups excluded for clarity); for bond lengths and angles see Table 1



Scheme 4. Topological relation between $R^*{}_6Ga_2^{2-}$, $(Hsi)_6Ga_9$, and $(Hsi)_6Ga_{10}$ [R* = Si*t*Bu$_3$, Hsi = $Si(SiMe_3)_3$]

Table 1 presents selected bond lengths [Å] and angles [°] of **3** and **4**. The Ga−Ga distances (2.41−2.93 Å for **3** and 2.45−2.76 for **4**) lie in the typical range observed in other gallium cluster compounds such as $R^*{}_4Ga_4$ [5] and $R^*{}_8Ga_{18}/R^*{}_8Ga_{22}$.[1] On reduction of **3** to **4** some of the Ga−Ga bonds lengthen and some shorten, but on average they remain practically constant (mean bond length of 2.64 Å in **3** and 2.61 Å in **4**). Although the reductions of **3** and **7** with sodium in THF formally proceed in the same manner ($R^*{}_6Ga_8/R^*{}_4Ga_4$ + 2 Na + 2 THF → $Na_2Ga_8R^*{}_6\cdot 2THF/Na_2Ga_4R^*{}_4\cdot 2THF$), in the first case very little structural change is observed, whereas in the second case a drastic structure change and significant Ga−Ga bond shortening occurs.[7] The Ga_8 framework of **3** is less symmetric than that of **4** (cf. Ga−Ga distances). The Na−GaR*, Na−Ga, and Na−O distances are 3.52 Å (mean value), 2.99 Å, and 2.31 Å (angles sum at Na 357°, mean value of the Ga−Na−Ga angles 46°), respectively.

Compounds **3** and **4** possess a framework of eight Ga atoms that was previously unknown for clusters of the group 13 elements B − Tl. The isolation of $R^*{}_6Ga_8$ (**3**) and Tsi_6Ga_8 (**1**)[2] shows for the first time that the heavier group 13 elements can also form isomeric clusters that hitherto were thought to be restricted to boron.

Compound **3** can be described as a *conjuncto*-octagallane, based on hexagallane $R^*{}_4Ga_6$ (**13**) and digallene $R^*{}_2Ga_2$ (**10**). Considering the Wade−Mingos rules[11] compound **13** may therefore be classified as a doubly capped *tetrahedro*-tetragallane, that is a *hypopreclso*-hexagallane [**13** possesses 10 cluster electrons ($2n - 2$ with n = number of cluster atoms), giving 1 electron for Ga and two electrons for GaR*], although this description does not agree at all with the structure obtained for **3** by X-ray crystallography (see Table 1).

Table 1. Selected bond lengths and angles of the molecules **3** and **4** in the crystal

Bond lengths [Å][a]	**3**	**4**
Ga7−Ga8	2.782(3)	2.678(1)
Ga1−Ga2/Ga5−Ga6	2.519(2)/2.561(3)	2.535(1)/2.685(1)
Ga1−Ga8/Ga2−Ga7	2.406(3)/2.446(3)	2.466(1)/2.453(1)
Ga5−Ga8/Ga6−Ga7	2.638(3)/2.502(3)	2.630(1)/2.561(1)
Ga3−Ga5/Ga3−Ga6	2.652(2)/2.713(2)	2.757(1)/2.641(1)
Ga3−Ga7/Ga3−Ga8	2.647(2)/2.849(2)	2.551(1)/2.592(1)
Ga4−Ga5/Ga4−Ga6	2.614(2)/2.731(2)	2.612(1)/2.761(1)
Ga4−Ga7/Ga4−Ga8	2.638(2)/2.925(2)	2.628(1)/2.536(1)
Ga−Si / Si−C (av.)	2.50/1.95	2.51/1.96
Ga4−Na1/Ga7−Na1	−	3.491(3)/2.983(2)
Ga3−Na2/Ga8−Na2	−	3.544(3)/2.989(2)
Na1−O1/Na2−O1	−	2.308(4)/2.308(5)
Bond angles [°] in quadrangles		
Ga1/Ga2/Ga7/Ga8	76.60(4)/109.36(4)/71.16(3)/102.44(4)	92.00(3)/90.93(3)/88.94(3)/87.38(3)
Ga5/Ga6/Ga7/Ga8	98.55(4)/85.97(4)/96.29(5)/79.11(4)	89.04(3)/90.76(3)/90.67(3)/89.44(2)
Ga3/Ga5/Ga4/Ga7	81.34(7)/98.46(7)/82.22(7)/97.98(7)	89.35(2)/87.56(2)/90.90(2)/91.70(3)
Ga3/Ga6/Ga4/Ga8	90.18(7)/94.19(7)/88.25(7)/87.36(7)	90.81(2)/86.92(2)/89.32(2)/92.88(3)
Bond angles [°] in triangles[b] and at other positions		
Ga(base)−Ga(apex)−Ga(base) (av.)	57.45	60.08
Ga(apex)−Ga(base)−Ga(base) (av.)	61.28	59.96
Ga1−Ga8−Ga5/Ga2−Ga7−Ga6	175.70(4)/167.11(4)	175.59(3)/174.32(3)
C−Si−C (av.)	111.1	109.9
O1−Na1−Ga4/Ga7	−	144.0(2)/163.4(2)
O2−Na2−Ga3/Ga8	−	148.0(2)/165.6(2)
Ga−Na−Ga (av.)	−	46.44
Torsion angles		
Ga5−Ga8−Ga7−Ga2	0.96	3.49
Ga6−Ga7−Ga8−Ga1	2.01	0.88
Si1−Ga1−Ga2−Si2	3.34	0.42
Si5−Ga5−Ga6−Si6	11.39	5.30

[a] Compound numbering from Figures 1 and 2. [b] Apex = Ga3, Ga4; basis = Ga5, Ga6, Ga7, Ga8.

However, **3** may also be derived from the hexagallane $R^{*}_{6}Ga_{6}$ − that should not be preparable for steric reasons − by substituting two neighbouring R* groups for digallanediyl -R*Ga−GaR*. According to its 12 (2*n*) cluster electrons, $R^{*}_{6}Ga_{6}$ should be classified as a monocapped trigonal-bipyramidal *closo*-Ga_5 cluster, that is a *precloso*-hexagallane. This description agrees with the solid-state structure found for **3** (Table 1). The long Ga8−Ga3/Ga4/Ga7 distances of the $R^{*}_{4}Ga_{6}$ moiety of **3** suggest that $R^{*}_{4}Ga_{6}$ contains a trigonal-bipyramidal Ga3Ga4Ga5-Ga6Ga7 cluster with Ga8 as the edge-bridging atom. By the same reasoning, the octagallanediide **4** may be described as a derivative of the *closo*-octagallanediide $R^{*}_{6}Ga_{6}^{2-}$ [14 (2*n* + 2) cluster electrons] by substituting two neighbouring R* groups for -R*Ga−GaR*-, and in fact the $R^{*}_{4}Ga_{6}^{2-}$ moiety of **4** shows a more regular (octahedral) Ga_6 framework than the $R^{*}_{4}Ga_{6}$ moiety of **3** (Table 1).

Information about the structures of **3** and **4** in solution is still limited. Only one (broad) NMR signal is found for the six R* groups of **3** in benzene, whereas three NMR signals (ratio of areas 1:1:1) are found for **4** in benzene. This either means that **3** gives equal NMR shifts for the nonequivalent R* groups, or that it is fluxional. Low temperature NMR studies of **3** in solution are planned. In addition, NMR studies of **4** in donor solvents D may confirm the expected dissociation of the cluster into $Na(D)_n^+$ and $R^{*}_{6}Ga_{8}^{2-}$ ions.

Experimental Section

All experiments were carried out in flame-dried glass apparatus with standard Schlenk techniques under dry argon or nitrogen. Air and moisture were strictly excluded. The solvents (heptane, benzene, THF) were dried with sodium plumbide or sodium in the presence of benzophenone. Available for use: C_6D_6. The following compounds were synthesized according to literature procedures: R*$_4$Ga$_3^{\cdot}$ (**3**),[4] NaC$_{10}$H$_8$ in THF.[12]

NMR Spectra: Jeol GX-270 (^{1}H/^{13}C/^{29}Si: 270.17/67.94/53.67 MHz), Jeol EX-400 (^{1}H/^{13}C/^{29}Si: 399.78/100.54/79.43 MHz). The NMR spectra were recorded with the INEPT and DEPT pulse sequences using empirically optimized parameters for the mentioned groups.

Synthesis of R*$_6$Ga$_8$ (3): A solution of **5** (0.151 g, 0.130 mmol) in 5 mL of heptane was kept 25 h at 60 °C, after which time it had quantitatively decomposed into R*H,[13] R*$_2$,[13] **6**,[4] **7**,[5] and **3** (ratio of ^{1}H NMR signal areas of the R* groups of molecules R*H, R*$_2$, **7**, **3** about 1:1:5:3, corresponding to a mol ratio of the compounds of about 4:2:5:2) according to ESR, and ^{1}H, ^{13}C and ^{29}Si NMR spectroscopy. After the removal of all volatile compounds under vacuum and dissolving the residue in 2 mL of benzene, compound **3** (0.115 g, 0.066 mmol, 15%) was obtained after eight days at 8 °C as water- and air-sensitive dark-blue crystals. ^{1}H NMR (C_6D_6): $\delta = 1.28$ (broad, 162 H, 6 SitBu$_3$). ^{13}C{^{1}H} NMR (C_6D_6): $\delta = 25.6/33.6$ (broad/broad, 18 C/54 C, 18 *C*Me$_3$/18 C*Me$_3$*). ^{29}Si NMR (C_6D_6): $\delta = 58.7$ (broad, 6 Si, 6SitBu$_3$). X-ray structure analysis: see Figure 1 (blue prisms from benzene). *Note*: The relative yields of **6** (seen only by ESR spectroscopy), **7** and **3** (seen only by NMR spectroscopy) depend, amongst others, on the reaction temperature. According to the NMR spectra, **7** and **3** are formed from **5** in heptane after 25 h at 40 or 60 or 100 °C in the mol ratio of about 99:1 or 71:29 or 100:0 (**3** is thermolabile at 100 °C).

Thermolysis of R*$_6$Ga$_8$ (3): A solution of **3** (0.073 g, 0.042 mmol) in 0.6 mL of C_6D_6 was kept for 14 h at 100 °C. After this time, according to the ^{1}H/^{13}C/^{29}Si NMR spectra, **3** had quantitatively decomposed into R*H,[13] R*$_2$,[13] **7**,[5] and an unidentified compound R$_m$Ga$_n$ (ratio of ^{1}H NMR signal areas of the R* groups of molecules R*H, R*$_2$, **7**, R$_m$Ga$_n$ about 2:1:10:(4+2), corresponding to a mol ratio of R*H, R*$_2$ and **7** of about 4:1:5). In addition, an insoluble product was formed (oligogallane ?). Characterization of the dissolved R$_m$Ga$_n$: ^{1}H NMR (C_6D_6): $\delta = 1.39$ (broad). ^{13}C{^{1}H} NMR (C_6D_6): $\delta = 25.59/26.01$ (s/s, 2x C/x C, 2x *C*Me$_3$/x *C*Me$_3$), 30.27/33.45 (s/s, 6x C/3x C, 2x C*Me$_3$*/x C*Me$_3$*). ^{29}Si NMR (C_6D_6): $\delta = 43.28/56.70$ (s/s, 2x Si/x Si, 2x SitBu$_3$/x SitBu$_3$). *Note*: Octagallane R*$_6$Ga$_8$ with the structure of R*$_6$In$_8$ (**2**) should give a ratio of SitBu$_3$ NMR signal areas of 2:1).

Synthesis of Na$_2$Ga$_8$R*$_6$·2THF (4): A solution of NaC$_{10}$H$_8$ (0.11 mmol) in 1.5 mL of THF was added dropwise to a solution of **3** (0.05 mmol) in 10 mL of THF at −78 °C. After 5 h stirring at −78 °C all volatile components were removed from the reaction mixture under vacuum at −45 °C and the residue was dissolved in 3 mL of C_6D_6. This solution was concentrated to 0.8 mL and water- and air-sensitive red crystals of **4** were obtained after two months at 5 °C. ^{1}H NMR (C_6D_6): $\delta = 1.289/1.367/1.380$ (s/s/s, 54 H/54 H/54 H, 2 SitBu$_3$/2 SitBu$_3$/2 SitBu$_3$), 1.124/3.325 (m/m, 8 H/8 H, 4 CH$_2$/4 CH$_2$O). ^{29}Si{^{1}H} NMR (C_6D_6): $\delta = 43.8/47.1/51.2$ (s/s/s, 2Si/2Si/2Si, 2 SitBu$_3$/2 SitBu$_3$/2 SitBu$_3$). X-ray structure analysis: see Figure 2 (red prisms from benzene).

X-ray Structure Determinations: Data collection was performed with a Siemens P4 diffractometer equipped with an area-detector (Mo-K_α radiation $\lambda = 0.71073$ Å). Crystals were mounted in perfluoropolyether oil and data collected at $T = 188(2)$ (**3**) and 193(2) K (**4**). The structures were solved by direct methods (SHELXS-97) and refined with full-matrix procedures against F^2 for all observed reflections. All non-hydrogen atoms were refined anisotropically and H atoms were included in the final refinement at calculated positions using a riding model and fixed isotropic U$_i$ values. A semiempirical absorption correction was applied by using the program SADABS. The structures of **3** and **4** are shown in Figure 1 and 2, crystallographic details are summarized in Table 2. CCDC-165237 (**3**) and CCDC-165238 (**4**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) + 44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

Table 2. Selected parameters of the X-ray structure analyses of the compounds **3** and **4**

	3	**4**
Empirical formula	$C_{72}H_{162}Ga_8Si_6$	$C_{92}H_{190}Ga_8Na_2O_2Si_6$
M_r	1754.32	2100.72
System	monoclinic	triclinic
Space group	$P2_1/c$	$P\bar{1}$
a [Å]	25.02(2)	16.641(2)
b [Å]	16.18(2)	16.717(2)
c [Å]	25.53(3)	21.589(2)
α [°]	90	91.996(2)
β [°]	118.195(1)	90.194(2)
γ [°]	90.000	113.033(2)
V [Å^3]	9110(2)	5525(1)
Z	4	2
D [Mg m^{-3}]	1.279	1.263
μ [mm^{-1}]	2.441	2.032
F(000)	3704	2224
2θ [°]	3.10−46.50	1.88−58.26
Ranges [°]	$-21 \le h \le 22$	$-20 \le h \le 20$
	$-17 \le k \le 17$	$-20 \le k \le 22$
	$-28 \le l \le 28$	$-27 \le l \le 28$
All reflections	37958	32188
Independent	10435	16939
Observed[a]	7428	10368
R_{int}	0.0496	0.0495
x/y[a]	0.8372/0.5403	0.6946/0.6205
R_1 [$F > 4\sigma(F)$]	0.0706	0.0439
wR_2[a] [$F > 4\sigma(F)$]	0.0965	0.0878
GOOF(F^2)	0.980	0.951
Max./min. electron density[e·Å^{-3}]	0.8372/−0.5403	0.6946/−0.6205

[a] $w^{-1} = \sigma^2F_0^2 + (xP)^2 + yP$ with $P = (F_0^2 + 2F_c^2)/3$.

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