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**From the Lithium-2-anilide-2-fluoro-1,3-diaza-2-sila-
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 Results**

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Keywords:	Gallium-silylamide, Ring-intermediates, Center two Electron Interaction, Proton-Migration, Computational Chemistry



From the Lithium-2-anilide-2-fluoro-1,3-diaza-2-sila-cyclopentene-GaCl₃-adduct to 1,4,6-Triaza-5-gallium-7-sila-cyclo-3-heptene- Experimental and Quantum-chemical Results

A. Fischer^a, D. Stern^a, A. Thorn^a, S. Abraham^b, D. Stalke^a and U. Klingebiel^{a*}

Professor Hubert Schmidbaur zum 75. Geburtstag gewidmet

Abstract. The lithium salt (HC-NCMe₃)₂SiFNLiR **1** R = C₆H₃(2,6-CHMe₂)₂ reacts with trichlorogallium under displacement of the lithium ion by GaCl₃ to give the adduct [(HC-NCMe₃)₂SiFN]⁺[(GaCl₃)R-Li(thf)₄]⁻ **1**. Thermally **1** loses LiCl forming the bicyclic ring intermediates **V** and **VI**. **VI** adds the aniline H₂NC₆H₃(2,6-CHMe₂)₂ and the unsaturated, seven-membered ring compound (-NCMe₃-CH₂-CH=NCMe₃GaCl₂-NR-SiFNHR-) **2** is obtained.

The addition is accompanied by an enamine-imine-tautomerism and proves the Lewis acid character of the silicon atom in an unknown 3-center-2-electron interaction of one nitrogen atom with the silicon and gallium atoms. Quantum chemical calculations of the thermal isomerisation process and crystal structures of **1** and **2** are reported.

Keywords: Gallium-silylamide/ Ring-intermediates/ three Center two Electron Interaction/ Proton-Migration/ Computational Chemistry

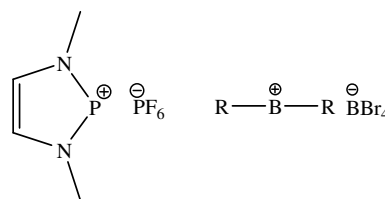
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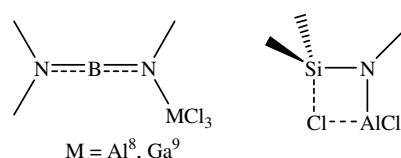
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Introduction

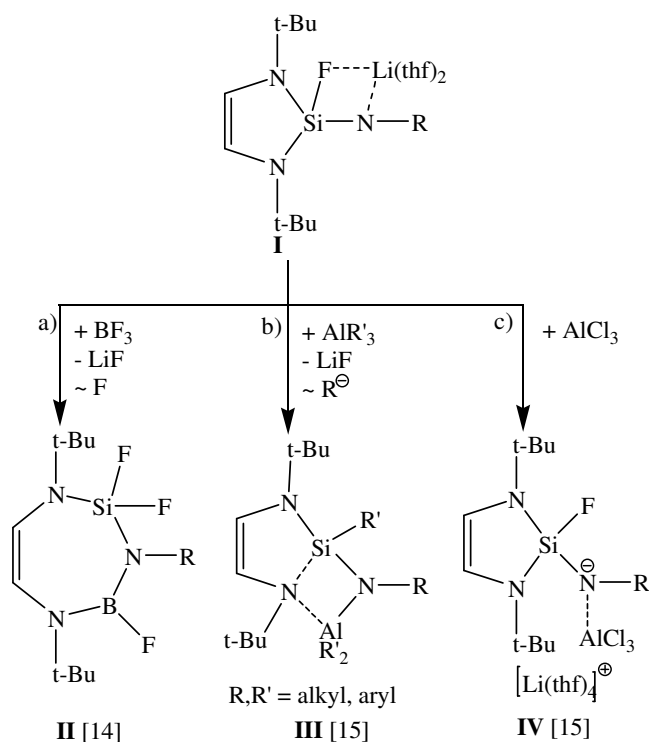
Unassociated compounds of the form R₂M-ER₂ have the potential for π-bonding between the electron deficient Group 13 element (M=B,Al,Ga) and the Group 15 element (E=N,P) through overlap of a lone pair at E with the empty p-orbital at M [1]. Such π-bonding is well established in molecules containing boron-nitrogen bonds [2]. For the heavier Group 13 elements, associated compounds (R₂M-NR'₂) are well-established [1-4]. For example triel adducts of the tert-butyliminopiperidinoborene and platinum silyliminoboryl-trichloroalane adducts are well-known. Threefold coordination is achieved by the use of bulky substituents at either M or N, or at both [1, 5]. All structurally characterized threefold coordinated Al-N and Ga-N compounds show essentially planar geometry at both the metal and the nitrogen center [1-4], which reflects the low inversion barrier at the nitrogen atom. The shortening of the M-N bond distances in comparison to the sum of the covalent radii for the aluminum or gallium and nitrogen centers arises from differences in electronegativity between M and N. It is known from chemistry textbooks that Lewis acids, e.g. halogen element compounds like GaCl₃ or PCl₅ often crystallize as complex homoleptic salts, e.g. 2 GaCl₃ → [GaCl₂]⁺[GaCl₄]⁻ or 2 PCl₅ → [PCl₄]⁺[PCl₆]⁻. The lattice energy is the driving force of these reactions. One ion reacts as Lewis acid and the other as Lewis base. Therefore it is understandable that a widely used method for preparing compounds with low-coordinated elements takes advantage of reactions between halogeno substituted elements and Lewis acids. This led to the syntheses of the first phosphonium [6] and borenium [7] cations,

as well as to the stabilisation of imino-borenes [8-10] and silenes, [11, 12] e.g.



Recently we were able to show that triel compounds like a) BF₃, b) AlMe₃, c) AlCl₃ undergo an unusual reaction behavior with lithium salts of 2-amino-2-fluoro-1,3-diaza-2-sila-cyclopentenes (HC-NCMe₃)₂SiFNLiR [13] (R=alkyl, aryl) forming (Scheme 1):

- a seven-membered (HC-NCMe₃)₂SiF₂-NRBF ring [14] via ring expansion and a fluorine atom migration from boron to the silicon atom,
- a bicyclic compound [(HC-NCMe₃)₂SiMe-NR-AlMe₂] [15] with one extremely long Si—N distance and a methanide ion migration from the aluminum to the silicon atom. One nitrogen atom binds in an unknown 3-center-2-electron interaction the silicon and aluminum atoms,
- a trichloroaluminum adduct of the amide (HC-NCMe₃)₂SiF-N⁻RAiCl₃ with [Li(thf)₄]⁺ as counter ion [15].

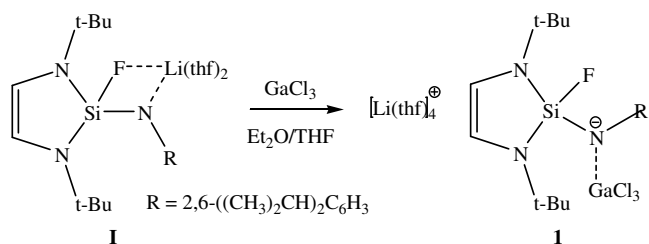


Scheme 1. Reaction behavior of a) BF₃, b) AlR₃ and c) AlCl₃ with the lithium salt I

Starting from 2-2,6-diisopropylanilino-2-fluoro-1,3-diaza-2-silacyclopentene [13] and its lithium salt (I) [15], we report in this paper its reaction with the Lewis acid GaCl₃.

Results and Discussion

The reaction between the lithium salt I and trichlorogallium is quite exothermic and affords in a solvent mixture of Et₂O/THF only one major product **1** (Scheme 2).



Scheme 2. Reaction of the lithium salt I with GaCl₃

The Lewis acid trichlorogallium displaces the hard Lewis acid Li⁺ from the amide ion. The lithium cation is stabilized by the coordination of four THF molecules.

Crystal structure of 1: Colorless crystals were grown from a THF solution of **1**. The crystal structure obtained is shown in Figure 1. **1** crystallizes in the orthorhombic space group *Pna*2₁ with four molecules in the unit cell. The charge of the anionic complex

is counterbalanced by one [Li(thf)₄]⁺ cation forming solvent separated ion pairs. One nitrogen atom of the five membered ring is pointing towards the gallium atom. The two possible isomers which could be formed by this coordination exist in the solid state and appear as a disorder of the ligand system whereas the GaCl₃ stays unchanged in position. All non-hydrogen atoms could be refined anisotropically and the disorder was described appropriately.

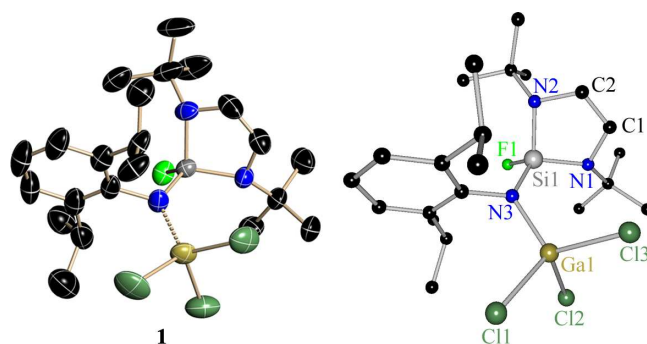


Figure 1. Crystal structure of **1** (left). Anisotropic displacement parameters are drawn at 50% probability level. Calculated molecular structure of **1** (right). Disorder, hydrogen atoms and the cation [Li(thf)₄]⁺ are omitted for clarity

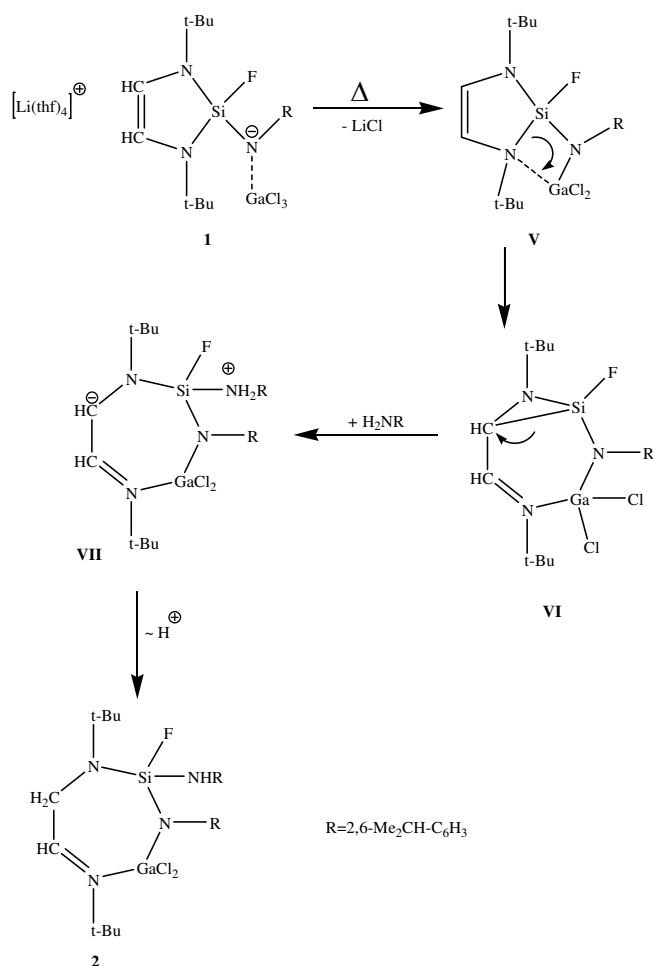
Table 1. Selected bond lengths [pm] and angles [°] of **1** (Calculated values obtained from B3LYP/6-31G(d)-calculations)

	found	Calculated
Ga(1)-N(3)	186.8(2)	192.1
Ga(1)-Cl(2)	219.54(8)	222.5
Ga(1)-Cl(3)	220.80(11)	224.1
Si(1)-F(1)	158.9(3)	163.4
Si(1)-N(1)	170.9(4)	174.5
Si(1)-N(2)	173.9(4)	177.0
Si(1)-N(3)	169.8(3)	170.7
N(3)-C(11)	150.7(4)	143.9
N(1)-C(1)	141.6(6)	141.9
N(1)-C(3)	149.7(6)	148.8
N(2)-C(7)	147.8(6)	142.4
C(1)-C(2)	135.5(6)	135.0
C(11)-N(3)-Si(1)	117.6(2)	119.4
C(11)-N(3)-Ga(1)	118.16(19)	116.4
Si(1)-N(3)-Ga(1)	123.80(15)	124.3
N(3)-Ga(1)-Cl(1)	109.20(8)	109.4
Cl(1)-Ga(1)-Cl(2)	105.36(4)	107.6
F(1)-Si(1)-N(3)	111.03(19)	109.4
F(1)-Si(1)-N(1)	114.0(2)	112.9
N(3)-Si(1)-N(1)	112.0(2)	113.5
N(1)-Si(1)-N(2)	98.0(2)	96.3
C(1)-N(1)-C(3)	118.7(3)	118.7
C(1)-N(1)-Si(1)	102.1(3)	103.0
C(3)-N(1)-Si(1)	135.1(3)	134.2
C(2)-N(2)-C(7)	117.6(3)	118.9
C(2)-N(2)-Si(1)	102.3(3)	102.5
C(7)-N(2)-Si(1)	135.1(3)	127.6

The Ga(1)-N(3) distance in compound **1** of 186.8(2) pm is shorter than the sum of the covalent radii of Ga and N (195 pm) [xx] and reflects a high ionic character of this bond. The Ga-N-bond found in threefold coordinated, monomeric gallium amides is 180.8 pm long [1]. The five-membered (HC-NCMe₃)₂Si-ring system is planar and N(3) has a planar environment. However, the sum of the angles around N(1) is 355.9° and around N(2) is 355.0°. This causes the *tert*-butyl groups into a slight *cis* position. The

Si(1)-N(1) (170.9(4) pm) and Si(1)-N(2) (173.9(4) pm) bond lengths of **1** are elongated, compared to the silicon-amide bond length (Si(1)-N(3) = 169.8(3) pm).

Encouraged by the reported seven-membered ring **II** [14] and the bicyclic compounds **III** [15] (Scheme 1), which were obtained in the reaction of the lithium salt **I** with trifluoroborane (**II**) or trimethylalane (**III**), we investigated the thermal reaction behavior of **1** and found that from 80 °C up to 150 °C LiCl elimination and ring expansion occur. LiCl elimination from lithiated aminochlorosilanes led in the past to the formation of iminosilenes; compounds with a Si=N double bond [5, 12, 16, 17]. Here, we obtained a brown oil. Its $^{29}\text{Si}\{^1\text{H}\}$ -NMR spectrum shows a signal in the range of an amine adduct of an iminosilene ($\delta^{29}\text{Si} = -12$ ppm) [15-17] and motivated us to carry out an addition reaction with an H-acidic molecule, because we didn't succeed in the identification of intermediates or the isolation of a pure compound. Choosing 2,6-diisopropyl-aniline we obtained in the reaction with the oil under the same conditions the seven-membered unsaturated ring compound **2**. Its formation is explained by the calculated reaction mechanism in Scheme 3.



Scheme 3. Proposed reaction mechanism of the formation of **2**

Computational details: For density functional theory (DFT) calculations, we approximated the exchange-correlation energy with an B3LYP functional. In the B3LYP hybrid method [18], the Becke 3-parameter exchange functional [19] is combined with the Lee, Yang and Parr [20] correlation functional. The 6-31G(d) basis

set (between 551 contracted Gaussian-type orbitals (cGTOSs) for compounds **V/VI** and 784 cGTOSs for compounds **VII/2**) was used throughout the optimization calculations which were performed using the Gaussian03 program package [21]. Additional single-point calculations at the stationary points of the B3LYP/6-31G(d) potential energy surface were performed employing the 6-311+G(2d,p) basis set (between 1082 and 1547 cGTOSs). The structures were optimized covering the full configuration space of the system. The minima were confirmed by analysis on the Hessian matrices, and zero-point vibrational energy (E_{zp}) effects were included within the harmonic oscillator-rigid rotator model.

Starting with compound **1**, LiCl elimination led to the formation of the intermediate bicyclic gallium ring compounds **V** and **VI**. In **V** the silicon and gallium atoms share the electrons of one formerly Si-N ring bond with one nitrogen atom in a three-centered two-electron interaction. Such an interaction has not been reported until now. **V** forms a new Si-C bond using the former C=C-double bond by a complete cleavage of the Si-N ring bond to the now threefold coordinated silicon. The gallium atom coordinates two chlorine atoms, the amide nitrogen (192.5 pm) and an imine nitrogen atom (208.9 pm) of the former Si-N bond. The shaped bicyclic intermediate **VI** consists of a three (CNSi)- and a six (CSiNGaNC)-membered ring system containing a imine-gallium-adduct bond (Table 2).

The Si-N bond lengths are calculated to be 166.4 pm and 166.6 pm. The cleaved Si-N distance is 333.5 pm. Without the Si-C bond the silicon atom has a nearly planar environment ($\Sigma\text{Si} = 355.1^\circ$). The C-C-bond length (145.4 pm) of the six-membered ring is calculated as a short single bond. The N-C bond length of the three-membered ring is also a single bond, whereas the C-N bond of the six-membered ring system (130.1 pm) is a lengthened double bond. The sum of the angles around the nitrogen atom is calculated with 359.5° proving the imine character of the nitrogen atom.

In the addition reaction of intermediate **VI** and 2,6-diisopropylaniline the intermediate unsaturated seven-membered ring compound **VII** is formed. The silicon atom of **VI** has added the nitrogen atom of the aniline, the Si-C-three-membered ring bond is cleaved, and from the positively polarised NH_2 -group a proton migrates to the negatively charged methine carbon in the neighborhood (Scheme 2).

Table 2. Calculated bond lengths [pm] and angles [$^\circ$] of **V**, **VI** and **VII** (obtained from B3LYP/6-31G(d)-calculations)

	V	VI	VII
Ga(1)-N(1)	208.9	204.3	193.4
Ga(1)-N(3)	192.5	192.9	195.5
Si(1)-N(1)	184.7	(333.5)	(367.2)
Si(1)-N(2)	174.0	166.4	170.1
Si(1)-N(3)	168.4	166.6	166.9
Si(1)-F(1)	161.4	160.4	162.1
N(1)-C(1)	146.3	130.1	136.1
N(1)-C(3)	154.1	152.0	150.1
N(2)-C(2)	140.2	150.2	145.5
C(1)-C(2)	134.6	145.4	136.3
Si(1)-C(2)	-	191.5	-
Si(1)-N(4)	-	-	198.8
N(1)-Ga(1)-N(3)	80.4	106.2	113.5
Ga(1)-N(3)-Si(1)	97.7	110.1	112.0
N(1)-Si(1)-N(3)	94.4	-	-
Ga(1)-N(1)-Si(1)	87.3	-	-
N(1)-Si(1)-N(2)	95.9	-	-
N(2)-Si(1)-N(3)	123.0	115.7	114.8

C(2)-N(2)- Si(1)	105.4	74.2	102.7
C(2)-Si(1)-N(2)	-	49.0	-
N(2)-C(2)-Si(1)	-	56.8	-
C(1) -C(2)- N(2)	118.6	118.5	123.3
C(2)-C(1)-N(1)	116.2	127.6	128.0
C(1)-N(1)-Si(1)	101.4	-	-
C(1)-N(1)-Ga(1)	107.3	121.5	117.6
C(1)-C(2)-Si(1)	-	119.7	-
C(2)-Si(1)-N(3)	-	116.8	-

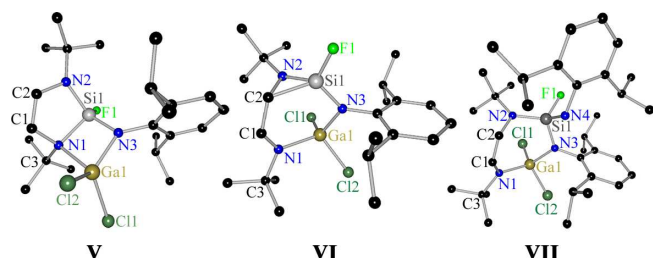


Figure 2. Calculated structures of the reaction intermediates V, VI and VII

Crystal structure of 2: Crystals suitable for X-ray structure determination were grown from a solution of **2** in *n*-hexane/THF. The investigated crystal in the monoclinic space group $P2_1/n$ was twinned. All non-hydrogen atoms could be refined anisotropically because the twinning was resolved appropriately. Figure 3 compares the result of the crystal structure analysis and the single point computational optimization from gas phase.

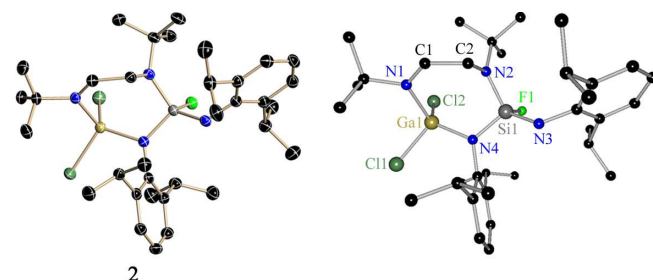


Figure 3. Crystal structure of **2** (left). Anisotropic displacement parameters are drawn at the 50% probability level. Ball-and-stick diagram depicts the calculated molecular structure of **2** (right). Hydrogen atoms are omitted for clarity

Table 3. Selected bond lengths [pm] and angles [°] of **2** (Calculated values obtained from B3LYP/6-31G(d)-calculations)

	Found	Calculated
Ga(1)-N(2)	188.3(2)	$\Sigma^\circ\text{N}(3)= 355.24(35)$ 190.9
Ga(1)-N(1)	204.1(2)	$\Sigma^\circ\text{N}(1)= 359.76(35)$ 209.3
Si(1)-F(1)	159.5(1)	$\Sigma^\circ\text{N}(2)= 359.32(19)$ 164.6
Si(1)-N(3)	172.76(15)	177.0
Si(1)-N(4)	170.45(15)	172.7
C(1)-N(1)	127.6(2)	127.9
C(1)-C(2)	149.7(2)	150.8
C(2)-N(2)	146.4(0)	145.7
Si(1)-N(2)	169.13(15)	170.6

The adduct bond of the gallium atom to the imine nitrogen N(1) is determined to 204.1(2) pm and hence is 16 pm elongated compared with the Ga(1)-N(2) bond (188.3(2) pm) which is also an effect of the different oxidation states (-II, -III) of the nitrogen atoms. The literature values of the Ga-N distance are in the range

of 196 pm. The N(3)-Si(1) bond (172.76(15) pm) in the seven-membered ring is elongated compared to the N(2)-Si(1) bond (169.13(15) pm) by 3.6 pm. The nitrogen atoms - with exception of N(3) - have a planar environment ($\Sigma\text{N}(3) = 355.2^\circ$, $\Sigma\text{N}(2) = 359.3^\circ$, $\Sigma\text{N}(1) = 359.8^\circ$). The slight pyramidal character of the N(3) atom forces the bonded tert-butyl groups out of the ring plane. Compared to the C(1)-C(2)-double bond length of **1** (135.6(6) pm) the C(1)-C(2) single bond length of **2** (149.7(2) pm) is about 12.2 pm elongated. The gallium contact to the imine nitrogen N(1) leads to a lengthening of the C(1)=N(1) double bond which is determined to be 127.6(2) pm long.

Conclusions

The GaCl₃ amide adduct (HC-NCMe₃)₂SiFN⁻GaCl₃R, with [Li(thf)₄]⁺ as counter ion, **1** was isolated, starting from the lithium salt of 2,6-diisopropylaniline-2-fluoro-1,3-diaza-2-silacyclopentene (HC-NCMe₃)₂SiFNLiR, (R = 2,6-(Me₂CH)₂C₆H₃), **1** and GaCl₃. Trichlorogallium displaces the Li⁺ ion from the amide nitrogen coordination. **1** loses LiCl thermally and a brown oil is obtained which reacts with the H-acidic 2,6-diisopropylaniline at the silicon atom - forming a seven-membered ring compound **2**. GaCl₂ is inserted into one Si-N bond of the five-membered ring. One hydrogen atom of the aniline substituent migrates to a close by methine group and the seven-membered ring system stabilizes itself in an imine-enamine-tautomerism. This is found in the crystal structure of **2**.

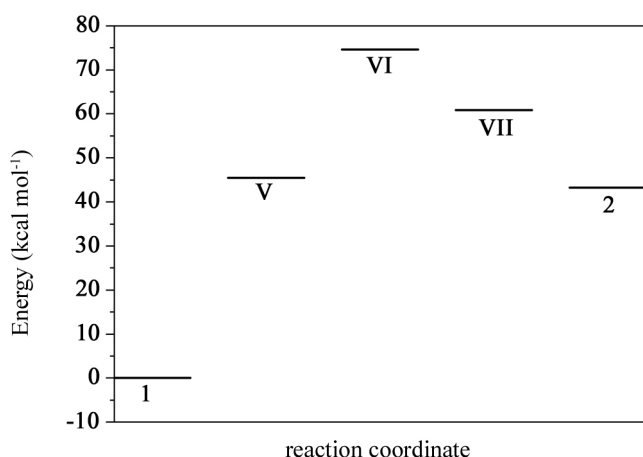


Figure 4. Energies for the compounds along the reaction path. B3LYP/6-311+G(2d,p) results. Energies of the amine and the Cl⁻ respectively are included within this comparison. [Li(thf)₄]⁺ was omitted for simplification.

Table 4. B3LYP energies (in kcal mol⁻¹) for the stationary points in the reaction for two different basis sets (values including zero-point energy effects [at B3LYP/6-31G(d) level] in parentheses). Energies of the amine and the Cl⁻ respectively are included within this comparison. [Li(thf)₄]⁺ was omitted for simplification.

	6-31G(d)	6-311+G(2d,p)
1	0 (0)	0 (0)
V	47.5 (47.9)	45.5 (45.8)
VI	75.8 (75.4)	74.6 (74.3)
VII	53.7 (56.0)	58.6 (60.9)
2	35.8 (37.7)	41.5 (43.3)

Experimental Section

The reactions were conducted under argon or nitrogen atmosphere. NMR spectra were recorded in C₆D₆ on either a Bruker AM 300 or an AVANCE 500 DRX spectrometer.

1,3-Di-tert-butyl-2-fluoro-2,2,6,6-diisopropylanilido-1,3-diaza-2-silacyclopentene-trichlorogallium-adduct-lithium-tetrahydrofuranate (1):

A solution of GaCl₃ (0.1 mol) in 100 mL *n*-hexane and 50 mL THF was added to 0.1 mol of the lithium salt **1** [14] in 50 mL *n*-hexane. The reaction mixture was heated at reflux for 3 h and compound **1** was isolated by recrystallization from *n*-hexane/THF.

C₃₈H₆₉Cl₃FGaLiO₄N₃Si (859.70): Analysis: Calculated. C 53.09, H 8.67, N 4.89. Found C 53.48, H 8.91, N 5.13.

Yield 83 %. ¹H-NMR (C₆D₆): δ = 1.18 [s, 18 H, NC(CH₃)₃], 1.25 [d, ³J_{HH}=6.8 Hz, 12 H, HC(CH₃)₂], 1.62 (s, 16 H, OCH₂CH₂), 3.66 (s, 16 H; OCH₂), 3.96 (sept.; ³J_{HH} = 6.8 Hz, 2H, HCCH₃), 5.66 (d, ⁴J_{HF} = 6.6 Hz, 2H, HC=CH), 6.67-6.94 (s, 3H, C₆H₃); ¹³C{¹H}-NMR: δ = 25.90 (O(C₂C₂)), 26.51 (HCC₂), 27.63 (HCC₂), 30.08 (d, ⁴J_{CF} = 2.26 Hz, NCC₃), 53.25 (d, ³J_{CF} = 1.16 Hz, NCC₃), 68.25 (OC₂C₂), 117.35 (d, ⁴J_{CF} = 2.64 Hz, HC=CH), 122.94 (C₆C(4)), 123.17 (C₆C(2,6)), 141.76 (C₆C(3,5)), 149.07 (d, ³J_{CF} = 0.72 Hz; C₆C(1)); ¹⁹F{¹H}-NMR: δ = 35.94; ²⁹Si{¹H}-NMR: δ = -42.32 (d, ¹J_{SiF} = 273.78 Hz); ⁷Li{¹H}-NMR: δ = -0.59; ⁷¹Ga{¹H}-NMR: δ = 248.53.

1,4-Di-tert-butyl-5,5-dichloro-7-fluoro-5,7-bis(2,6-diisopropylphenyl)-1,4,6-triaza-5-gallium-7-sila-cyclo-3-heptene (2):

To a solution of 0.01 mol **1** in 50 mL *n*-hexane 0.01 mol of 2,6-diisopropylphenylamine were added at room temperature. The solvent was removed in vacuum, the reaction mixture was heated up to 150 °C under reflux for 1h. **2** crystallized from the residue and was recrystallized from *n*-hexane. Because of the low solubility in unpolar solvents, we did not get useful NMR data of **2**.

C₂₈H₅₅Cl₂FGaN₄Si (635.49): Analysis: Calculated. C 52.92, H 8.72, N 8.82. Found. C 53.19, H 9.03, N 9.12. Yield: 13 %.

For X-ray diffraction analysis single crystals were selected from the Schlenk flasks under argon atmosphere and covered with perfluorated polyether oil on a microscope slide, which was cooled with a nitrogen gas flow using the X-TEMP2 [22]. Appropriate crystals were selected using a polarizing microscope, mounted on the tip of a glass fibre, fixed to a goniometer head and shock cooled by the crystal cooling device.

For **1** data were collected on a Bruker APEXII Quazar diffractometer with D8 goniometer at 100 K (Mo K α radiation, λ = 71.073 pm; INCOATEC Quazar mirror optics) [23] For **2** data was collected on a Bruker Smart 6000 CCD diffractometer (Cu K α radiation, λ = 154.178 pm, graphite-monochromator). Data reduction and processing, carried out with SAINT (1) or SAINT+ [24] (2) was followed by semi-empirical absorption correction with SADABS (1) or TWINABS [25] (2). Both structures were solved by direct methods (SHELXS) and refined on F² using the full-matrix least-squares methods of SHELXL [26]. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms bonded to sp² (sp³) carbon atoms were assigned ideal positions and refined using a riding model with U_{iso} constrained to 1.2 (1.5) times the U_{eq} value of the parent carbon atom. The amino hydrogen atom in **2** was refined free. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Centre, the CCDC numbers are listed in Table 4. Copies of the data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table 5. Crystallographic data and structure refinement for compounds **1** and **2**

	1	2
empirical formula	C ₃₈ H ₆₉ Cl ₃ FGaLiN ₃ Si	C ₃₄ H ₅₆ Cl ₂ FGaN ₄

	O ₄ Si	Si
CCDC No.	747520	744808
formula weight	862.06	708.54
T [K]	100(2)	100(2)
crystal system	orthorhombic	monoclinic
space group	Pna2 ₁	P2 ₁ /n
a [pm]	2088.69(17)	1468.0(3)
b [pm]	1900.83(15)	1495.8(3)
c [pm]	1162.47(9)	1684.4(4)
β [°]	90	98.48(3)
V [nm ³]	4.6153(6)	3.6582(14)
Z	4	4
ρ_{calcd} , Mg m ⁻³	1.241	1.286
μ [mm ⁻¹]	0.838	2.944
F(000)	1832	1504
θ range for data collection [°]	2.62 to 26.02	3.73 to 64.51
no. of reflections collected	91375	139230
no. of independent reflections	9086	6091
Data / restraints / parameters	9086 / 1442 / 837	6363 / 1 / 406
Absolute structure parameter ^[27]	0.045(8)	–
Goof	1.073	1.058
R1 ^a , wR2 ^b [I > 2 σ (I)]	0.0364, 0.1030	0.0255, 0.0687
R1 ^a , wR2 ^b (all data)	0.0387, 0.1051	0.0287, 0.0704
largest diff peak, hole [e Å ⁻³]	0.396, -0.500	0.378, -0.253

$$^a R1 = \sum |F_o| - |F_c| / \sum |F_o|; ^b wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)]^{0.5}$$

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