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[14] In order to accumulate *Bn*-FCC-2, which occurs as traces in yellowing rape cotyledons, aqueous, particle-free extracts were applied to SepPak cartridges and eluted with a small amount of MeOH/H_2O (9/1, v/v). *Bn*-FCC-2 from rape cotyledons was compared to **3** by means of analytical HPLC (see ref. [9a]), UV/Vis-spectroscopy, and luminescence properties.

[15] Experiments by Eschenmoser and co-workers provided evidence of favorable thermodynamics for the formation of pyrrole units in tautomerization reactions of (metal-free) hydroporphyrins: A. Eschenmoser, *Angew. Chem.* **1988**, *100*, 5–40; *Angew. Chem. Int. Ed. Engl.* **1988**, *27*, 5–40. The feasibility of the tautomerization of a mixture of 4,5-seco-4,5-dioxooctahydrophytoporphyrinates related to **3** to linear tetrapyrroles structurally similar to **1** has recently been reported: N. Engel, C. Curty, A. Gossauer, *Plant Physiol. Biochem.* **1996**, *34*, 77–83.

[16] In *Hv*-NCC-1 (**1**) [4], as well as in several other NCCs of plants [6,7], an intact methoxycarbonyl function is found at the 13^2 position (as in *Bn*-FCC-2, **3**). Accordingly, in contrast to a recent proposal by Gossauer & Engel [17b], there is no indication of an "early" hydrolysis of this ester function occurring during the chlorphyll breakdown in plants.

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Synthesis and Structure of the First Si-Al-NH Cage Compound from a Stable Triaminosilane and Trimethylaluminum**

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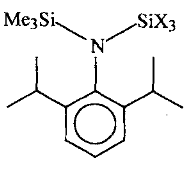
Andreas Klemp, Herbert W. Roesky,*
Christiane Brönneke, Jörg Kärcher, and
Regine Herbst-Irmer

Dedicated to Professor Oskar Glemser
on the occasion of his 85th birthday

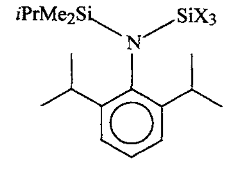
Aluminosilicates serve as important catalysts in many industrial processes,[1–3] and Al–Si oxonitrides have been known as ceramic materials since the 1970s.[4] In this context it seemed a reasonable goal to construct model compounds for a nitrogen-analogous aluminosilicate chemistry from stable triaminosilanes. Compounds of this kind are hitherto unknown and should offer a new range of properties.

Conversions of stable silanetrioles of the type $RSi(OH)_3$ to inorganic cage molecules have already been frequently reported. Thus, for instance, a titanium silasesquioxane could be obtained from $RSi(OH)_3$ ($R = i Pr_2C_6H_3NSiMe_3$) and $Ti(O i Pr)_4$. This can be considered as a model compound for titanium-doped zeolites.[5] Another example is the product of the reaction of $RSi(OH)_3$ with diisobutylaluminum hydride giving a soluble zeolite precursor.[6] The reaction of $RSi(OH)_3$ ($R = i Pr_2C_6H_3NSiMe_3$) with Me_3Al leads to a corresponding cubane.[7]

In 1993 Power et al. reported on the synthesis of the first monomeric triaminosilane, 2,4,6-$Ph_3C_6H_2Si(NH_2)_3$, by ammonolysis of the corresponding trichlorosilane.[8] In analogy, stable triaminosilanes can be synthesized from the precursors of the above-mentioned silanetrioles, the trichlorosilanes of the type $RSiCl_3$ ($R = i Pr_2C_6H_3NSiMe_3$ (**1a**), 2,4,6-$t Bu_3C_6H_2O$).[9] Our primary goal was the synthesis of Si-Al-NH cage compounds by reaction of triaminosilanes $RSi(NH_2)_3$ with simple organoaluminum compounds.



1a : X = Cl
1b : X = NH_2



2a : X = Cl
2b : X = NH_2

Me_3Al reacts with $RSi(NH_2)_3$ ($R = i Pr_2C_6H_3NSiMe_3$) (**1b**) to give a product that is almost insoluble in all common organic solvents and thus difficult to characterize. Replacement of a methyl group in the trimethylsilyl group by an isopropyl group should lead to an increase in the solubility of this product. Thus, the trichlorosilane **2a** was prepared and converted to the triamine **2b** by ammonolysis. Single crystals of **2b** were obtained from pentane/hexane. In the asymmetric unit there are two molecules of **2b**,[13] and one pentane molecule disordered about the twofold axis (Figure 1). The bond lengths and angles of the two independent molecules show negligible differences. These two molecules are linked to each other and to two additional symmetry-related molecules through hydrogen bonds (N3B–N3A 336.4, N3A–N2A 329.2 pm); thus, unlike in **1b**, which is dimeric, **2b** exists as a tetramer.

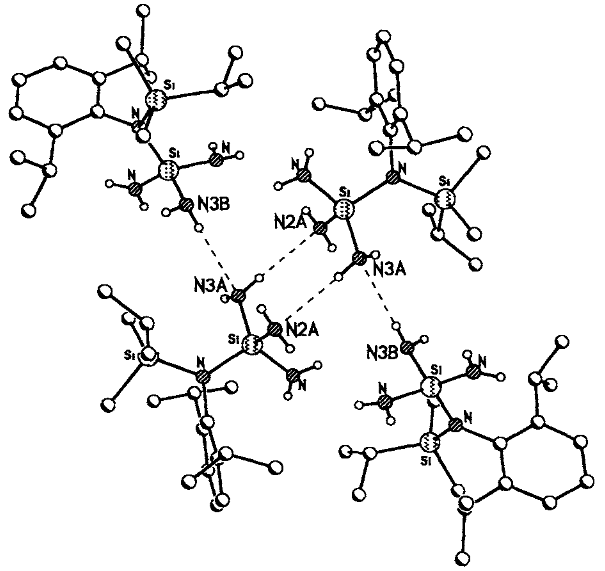


Figure 1. Crystal structure of **2b**.

The reaction of **2b** with Me_3Al leads to **3** [Eq. (a)]. Single crystals suitable for X-ray structure analysis were obtained from THF/hexane. The molecular structure of **3** in the crystal shows point symmetry (Figure 2).[14] The central cage framework consists of two six-membered Al_2N_3Si rings present in the boat

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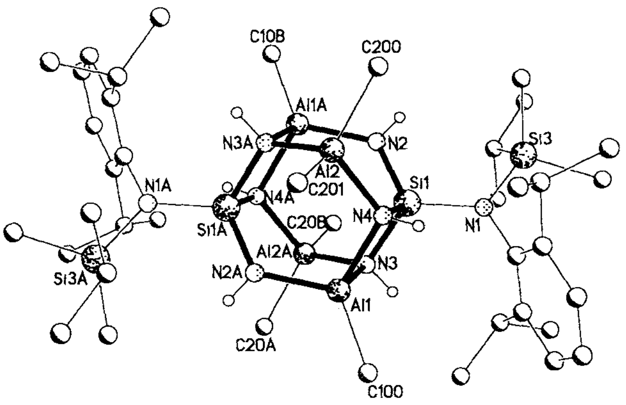
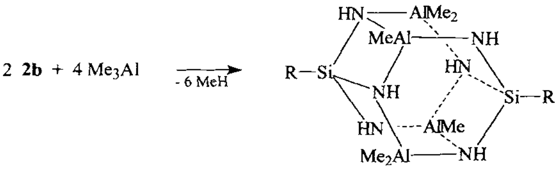


Figure 2. Crystal structure of 3. Selected bond lengths [pm] and angles [°]: N1–Si1 172.8(1), Si1–N3 178.4(1), N4–Al2 195.0(1), Si1–N2 169.4(1), Si1–N4 179.4(1), N3–Al1 197.2(1), N4–Al1 195.0(1); N2-Si1-N1 114.2(1), N1-Si1-N3 115.4(1), N1-Si1-N4 112.8(1), N2-Si1-Al1 122.0(1), Si1-N2-Al1A 123.3(1), Si1-N4-Al2 118.9(1), Si1-N3-Al1 92.2(1), N2A-Al1-Si1 114.7(1), N2-Si1-N3 108.8(1), N3-Si1-N4 92.5(1), N1-Si1-Al1 123.7(1), N4-Al1-N3 82.5(1).

conformation, which are linked to each other through two Al–N and two Si–N bonds. The aluminum atoms are four coordinate; in each case two interactions involve dipolar bonds to the nitrogen atoms. The ^{1}H NMR spectrum of **3** shows the



(a)

R = 2,6-iPr$_2$C$_6$H$_3$NSiMe$_2i$Pr

3

expected signals. Thus, the singlets ($\delta = -1.38, -0.85$) are assigned to the protons of the methyl groups at the aluminum atom. The corresponding signals for the ligands of **3** can be assigned correctly through integration.[11] The most intense signals in the mass spectrum (EI) appear at m/z 881 (100%) and 433 (38%), and were assigned to the molecular ion and half the molecular ion minus a methyl group in each case, respectively.

Although the NH group is isoelectronic to the O atom, a cubane compound analogous to that obtained in the reaction of the silanetriole with Me$_3$Al is not formed;[7] however, the structure of **3** is similar to that of the product from the reaction of Me$_3$Ga with iPr$_2$C$_6$H$_3$NSiMe$_3$Si(OH)$_3$.[10]

Compound **3** is unusually thermally stable and decomposes only at 280 °C. Interestingly, even when the stoichiometry of the reactants was altered, **3** was always obtained as the product. Thus, reactions of the starting materials in a molar ratio 1:1 also gave **3**. Even, the reaction of **2b** with the dimethylaminoalane Me$_2$Al(iPr$_2$C$_6$H$_3$NSiMe$_3$) in a molar ratio 1:1 afforded **3**, further emphasizing the preferred formation of this product.

Since **3** is a bifunctional compound, many subsequent reactions can be envisaged. We are currently interested in the metalation of the imino groups and in the substitution of the methyl groups at the aluminum atoms. Furthermore **3** could serve as a precursor for ternary ceramics and open up the possibility of many applications in the context of chemistry analogous to that established for the aluminosilicates. Thus, the planned construction of supramolecular structures with characteristic properties is conceivable with **3** and also with the above-mentioned siloxanes.

Experimental Section

2a was prepared from iPr$_2$C$_6$H$_3$NHSiMe$_2i$Pr and SiCl$_4$ analogous to the synthesis of **1a** [4]; yield 3.2 g (72%); m.p. 35 °C; ^{1}H NMR (200 MHz, C$_6$D$_6$): $\delta = 0.13$ (s, 6H, Si(CH_3)$_2i$Pr), 0.92 (d, ^3J(H,H) = 7.1 Hz, 6H, CH(CH_3)$_2$), 1.19 (d, ^3J(H,H) = 6.8 Hz, 6H, CH(CH_3)$_2$), 1.23 (d, ^3J(H,H) = 6.8 Hz, 6H, CH(CH_3)$_2$), 1.31 (sept., ^3J(H,H) = 7.1 Hz, 1H, CH(CH$_3$)$_2$), 3.60 (sept., ^3J(H.H) = 6.8 Hz, 2H, CH(CH$_3$)$_2$), 7.03 (m, 3H, ArH); ^{29}Si NMR (50 MHz, C$_6$D$_6$): $\delta = 16.3$ (Si(CH$_3$)$_2i$Pr), −27.0 (SiCl$_3$); IR (KBr, Nujol): $\tilde{\nu} = 1362, 829, 752$ cm^{-1}; MS (EI): m/z (%): 411 (8) [M^+], 368 (100) [$M^+ - i$Pr]; correct elemental analysis [12].

2b: **2a** (1.09 g, 2.7 mmol) was dissolved at −78 °C in liquid ammonia (ca. 15 g), which had previously been dried over sodium metal. The ammonia was allowed to evaporate from the reaction mixture under stirring over 14 h. During this period the mixture was allowed to warm slowly from −55 to −30 °C. The remaining white residue was taken up in CH$_2$Cl$_2$ (15 mL), the ammonium chloride that formed was filtered off, and the solvent was removed in vacuo. Crystals suitable for X-ray structure analysis were obtained by recrystallization from hexane/pentane (5 mL). After removal of all of the volatile components, the colorless product was purified by sublimation (55 °C, 10^{-2} bar). Colorless crystals of **2b** were obtained in 70% yield (0.67 g); m.p. 62 °C; ^{1}H NMR (200 MHz, C$_6$D$_6$): $\delta = 0.99$ (s, 6H, Si(CH_3)$_2i$Pr), 0.45 (br. s, 6H, Si(NH$_2$)$_3$), 1.03 (d, ^3J(H,H) = 7.1 Hz, 6H, CH(CH_3)$_2$), 1.23 (d, ^3J(H,H) = 6.8 Hz, 6H, CH(CH_3)$_2$), 1.27 (d, ^3J(H,H) = 6.8 Hz, 6H, CH(CH_3)$_2$), 1.31 (sept., ^3J(H,H) = 7.1 Hz, 1H, CH(CH$_3$)$_2$), 3.65 (sept., ^3J(H,H) = 6.8 Hz, 2H, CH(CH$_3$)$_2$), 7.06 (s, 3H, ArH); ^{29}Si NMR (50 MHz, C$_6$D$_6$): $\delta = 7.9$ (Si(CH$_3$)$_2i$Pr), −41.8 (Si(NH$_2$)$_3$); IR (KBr, Nujol): $\tilde{\nu} = 3489, 3407, 1362, 829, 752$ cm^{-1}; MS (EI): m/z (%): 352 (4) [M^+], 309 (100) [$M^+ - i$Pr]; correct elemental analysis [12].

3 (from **2b** and Me$_3$Al): Me$_3$Al (2.3 mL, 2 M solution in heptane, 4.6 mmol) was added dropwise to a solution of **2b** (0.82 g, 2.3 mmol) in toluene (20 mL) at −70 °C. As soon as the gas evolution had ceased, the mixture was allowed to warm to room temperature and stirred for 12 h. The solvent was removed in vacuo, and the remaining yellow oil was taken up in hexane. A white solid precipitated that was filtered off. Compound **3** was obtained in 75% yield (0.77 g). Starting from **2b** and Me$_3$Al in the molar ratio 1:1 the yield of **3** was 0.39 g (75%).

3 (from **2b** and Me$_2$Al(iPr$_2$C$_6$H$_3$NSiMe$_3$): A solution of Me$_2$Al(iPr$_2$C$_6$H$_3$NSiMe$_3$) (0.70 g, 2.2 mmol) in hexane (20 mL) was added dropwise to a solution of **2b** (0.80 g, 2.2 mmol) in hexane (30 mL) at 0 °C. After the gas evolution had ceased, the reaction mixture was stirred for 12 h at room temperature, followed by 1 h at 30 °C. The solvent was concentrated under vacuum to 15 mL, and the white solid residue was filtered off; yield 0.40 g (82%); decomp 280–285 °C; ^{1}H NMR (200 MHz, [D$_8$]THF): $\delta = -1.37$ (s, 6H, AlCH$_3$), −0.83 (s, 12H, Al(CH$_3$)$_2$), 0.08 (s, 12H, Si(CH_3)$_2i$Pr), 0.89 (br. s, 6H, Si(NH)$_3$), 1.15 (d, ^3J(H,H) = 6.8 Hz, 12H, CH(CH_3)$_2$), 1.20 (d, ^3J(H,H) = 6.8 Hz, 12H, CH(CH_3)$_2$), 1.32 (d, ^3J(H,H) = 6.8 Hz, 12H, CH(CH_3)$_2$), 1.38 (sept., ^3J(H,H) = 7.1 Hz, 2H, CH(CH$_3$)$_2$), 3.52 (sept., ^3J(H,H) = 6.8 Hz, 4H, CH(CH$_3$)$_2$), 7.23 (s, 6H, ArH); IR (KBr, Nujol): $\tilde{\nu} = 3407, 3345, 3328, 1383, 825, 753$ cm^{-1}; MS (EI): m/z (%): 433 (38) [$(M/2)^+ -$ Me], 881 (100) [$M^+ -$ Me]; correct elemental analysis [12].

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[11] The ^{1}H NMR spectrum also shows a second series of signals for the ligand protons and a broad signal ($\delta = -0.85$) for the protons of the methyl groups at the aluminum atom. From this we conclude that apart from **3**, RSi(NH)$_3$Al$_2$Me$_3$ is probably also present in THF. This is also supported by temperature-dependent ^{1}H NMR measurements.

[12] Small deviations in the elemental analyses are attributed to the formation of metal carbides and metal nitrides.

[13] a) Crystallographic data of **2b**: $C_{17}H_{36}N_4Si_2 \cdot 0.25\,C_5H_{12}$, $M_r = 370.71$, space group $P2/c$, $a = 1511.1(1)$, $b = 980.8(1)$, $c = 3137.1(3)$ pm, $\beta = 96.59(1)^\circ$, $V = 4.6187(7)$ nm^3, $Z = 8$, $\rho_{calcd} = 1.066$ Mg m^{-3}, $F(000) = 1636$, $\lambda = 71.073$ pm, $\mu(Mo_{K\alpha}) = 0.162$ mm^{-1}. Data for the structure were collected on a Stoe-Siemens AED diffractometer. Intensity measurements were performed at 193 K on a rapidly cooled crystal in an oil drop [15] according to the $2\theta/\omega$ method in the range $7^\circ \leq 2\theta \leq 48^\circ$. The monoclinic crystal was colorless and had the dimensions $0.7 \times 0.5 \times 0.3$ mm. Of 8892 measured reflections 7243 were independent ($R_{int} = 0.0362$). The structure was solved by direct methods (SHELXS-90) [17] and refined according to the least-squares method on F^2 [16]. Hydrogen atoms, apart from those of the amino groups, were calculated in their ideal geometrical positions and refined according a riding model in which the methyl groups were able to rotate about their local axes. The hydrogen atoms of the amino groups could be localized by difference-Fourier synthesis and refined with distance restraints. The pentane molecule is disordered about the twofold axis. 218 restraints were used for the final refinement of the 558 parameters. The final difference-Fourier synthesis gave a minimum of -248 and a maximum of $+392$ e nm^{-3}. $R1 = 0.0406$ (for reflections with $I > 2\sigma(I)$) and $wR2 = 0.1045$ (all data). $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR2 = \{[\Sigma w(F_c^2 - F_o^2)^2]/[\Sigma w(F_o^2)^2]\}^{1/2}$. b) Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100052. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: Int. code +(1223) 336-033; e-mail deposit@chemcrys.cam.ac.uk).

[14] Crystallographic data of **3**: $C_{40}H_{84}Al_4N_8Si_4$, $M_r = 897.43$, triclinic, space group $P\bar{1}$, $a = 1042.2(3)$, $b = 1164.5(3)$, $c = 1218.6(3)$ pm, $\alpha = 94.67(1)$, $\beta = 107.16(1)$, $\gamma = 104.14(1)^\circ$, $V = 1.3514(7)$ nm^3, $Z = 1$, $\rho_{calcd} = 1.103$ Mg m^{-3}, $F(000) = 488$, $\lambda = 71.073$ pm, $T = 193$ K, $\mu(Mo_{K\alpha}) = 0.209$ mm^{-1}. Data for the structure were collected on a Stoe-Siemens-Huber diffractometer with a Siemens CCD detector. Intensity measurements were performed at 193 K on a rapidly cooled crystal with the dimensions $0.9 \times 0.7 \times 0.4$ mm in an oil drop [15] in the range $6.36^\circ \leq 2\theta \leq 56.56^\circ$. Of 21086 measured reflections 6569 were independent ($R_{int} = 0.0203$). A semiempirical absorption correction was carried out on all data. The structure was solved by direct methods (SHELXS-90) [17] and refined according to the full-matrix least-squares method on F^2 [16]. The positions of the hydrogen atoms of N–H bonds were refined with distance restraints and those of C–H bonds were calculated in their ideal geometrical positions and refined according to the riding model. An extinction correction was carried out. In total 277 parameters were refined. $R1(I > 2\sigma(I)) = 0.033$, $wR2 = 0.094$ (all data). The largest deviations in the final difference-Fourier synthesis are $+714$ and -439 e nm^{-3} [13b].

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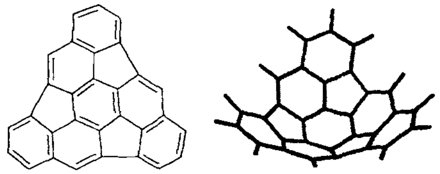
Novel Syntheses of Three $C_{30}H_{12}$ Bowl-Shaped Polycyclic Aromatic Hydrocarbons[**]

Stefan Hagen, Matthew S. Bratcher, Mark S. Erickson, Gerhard Zimmermann, and Lawrence T. Scott[*]

Bowl-shaped polycyclic aromatic hydrocarbons (PAH) are currently attracting considerable attention as the "missing links" between normal planar PAH and the tremendously strained, closed polyhedral fullerenes.[1] In our continuing efforts to define the scope and limitations of the flash vacuum pyrolysis (FVP) strategy for preparing such compounds,[2] we have developed short syntheses for the three isomeric $C_{30}H_{12}$ bowl-shaped hydrocarbons **1**, **2**, and **3**.[3]

1



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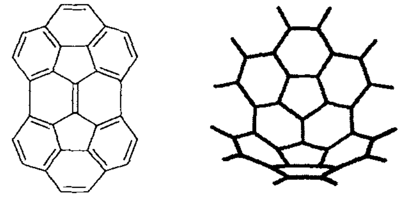

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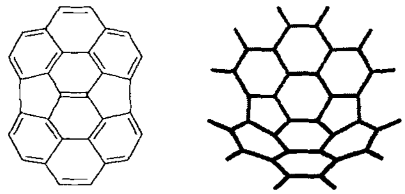

"Buckybowls" **1** and **2** both map directly onto the surface of C_{60} and have recently been prepared by distinctly different routes in quantities sufficient for NMR characterization by Rabideau et al.[4, 5] The bowl-shaped PAH **3**, on the other hand, was unknown until now and does not map onto the ring system of any known fullerenes smaller than C_{78}.[6] Herein we describe new syntheses for all three compounds.

When looking for potential precursors to **1** that already embody the C_3 symmetry of the final target compound, one is naturally drawn to the $C_{30}H_{18}$ PAH benzo[c]naphtho[2,1-p]chrysene (**4**, Scheme 1). This attractive compound is now conveniently available in multigram quantities by our new four-step synthesis.[7] Earlier, unsuccessful attempts to prepare **1** by FVP (1100 °C) of **4**[8] underscore the generalization that synthetic routes to strained bowl-shaped PAH based on uncatalyzed cyclodehydrogenation reactions of hydrocarbons rarely work well[9, 10] and sometimes fail completely (as in this case). On the other hand, we have previously found that aromatic hydrocarbons bearing a bromine atom at the site of each intended cyclization reaction significantly outperform the unsubstituted parent hydrocarbons as precursors for the synthesis of bowl-shaped PAH such as dibenzo[a,g]corannulene and diindeno[1,2,3,4-*defg*;1′,2′,3′,4′-mnop]chrysene.[2b] The weakly bound bromine atoms are easily lost at elevated temperatures, and this prearranged homolysis can be used to generate reactive aryl radicals specifically at the sites required for ring-closure reactions.[11] To implement this aryl radical cyclization strategy in the present case, we simply brominated hydrocarbon **4** and subjected the resulting tribromide to flash vacuum pyrolysis.

As anticipated, bromination of **4** with Br_2 (9 equiv, refluxing chlorobenzene, 1 h) results in the selective formation of 6,12,18-tribromobenzo[c]naphtho[2,1-p]chrysene (**5**, 90%).[12] We found it necessary to use a high-boiling solvent in order to keep

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