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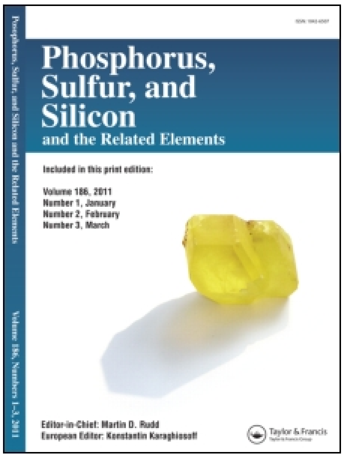

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# POLYCYCLES AND CAGES IN MOLECULAR ALUMO-POLYSILOXANES—$(Ph_2SiO)_8[Al(O)OH]_4$ Used as a Supramolecular Target and “Auto-Condensation” Within the Molecule

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# POLYCYCLES AND CAGES IN MOLECULAR ALUMO-POLYSILOXANES—$(Ph_2SiO)_8[Al(O)OH]_4$ USED AS A SUPRAMOLECULAR TARGET AND "AUTO-CONDENSATION" WITHIN THE MOLECULE

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*In this preliminary review the reaction of $[Ph_2SiO]_8[AlO(OH)]_4$ toward 1,3-diaminopropane and hexamethyldisilazane is discussed in view of supramolecular chemistry and access to structural transformations of the original polycycle. Two distinct adducts may be isolated in the first case: $[Ph_2SiO]_8[AlO(OH)]_4 \cdot 3H_2N\text{-}CH_2CH_2CH_2\text{-}NH_2$ and $[Ph_2SiO]_8[AlO(OH)]_4 \cdot 2H_2N\text{-}CH_2CH_2CH_2\text{-}NH_2$. Whereas in the 1:3 adduct the four protic hydrogen atoms of the inner $Al_4(OH)_4$ ring are involved in $O \cdots H \cdots N$ hydrogen bridges to two terminal diaminopropanes and a bridging diaminopropane thus forming an $O \cdots H \cdots N(H)_2\text{-}CH_2CH_2CH_2N(H)_2 \cdots H \cdots O$ loop, in the 1:2 adduct two such $O \cdots H \cdots N(H)_2\text{-}CH_2CH_2CH_2N(H)_2 \cdots H \cdots O$ loops are present. When $[Ph_2SiO]_8[AlO(OH)]_4$ is allowed to react with hexamethyldisilazane, again two different products may be obtained depending on the solvent: $[Ph_2SiO]_8[AlO_2]_2[AlOO\text{-}SiMe_3]_2[NH_4 \cdot THF]_2$ or $[Ph_2SiO]_8[AlOO_{0.5}]_4 \cdot 2\,py$. This last reaction may be viewed as an inner condensation within $[Ph_2SiO]_8[AlO(OH)]_4$ loosing two equivalents of water. Both products of the reaction with hexamethyldisilazane have an inner $Al_2O_2$ four-membered cycle in common, to which $Al_2O_4Si_2$ eight-membered cycles are partly fused.*

*Keywords:* Molecular alumosiloxanes; oxygen connected polycycles with silicon and aluminum; supramolecular chemistry; structural chemistry

Oligo- and poly-siloxanes of the general formula $(R_2SiO)_n$ (R = organic group) as well as sesquioxanes $RSiO_{1.5}$ are well established compounds

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with a very rich synthetic and structural chemistry. It is astonishing to find that systematic replacements of $R_2SiO$ or $RSiO_{1.5}$ groups within these chain-, ring-, and cage-like compounds by XAlO[1–7] or XGaO[8,9] entities (X = monovalent ligand) not attracted too much attention until now; although in solid state materials like silicates such replacements of silicon by aluminum they are well known and have important implications (see zeolites for example.)[10–12] In 1997 we reported a convenient route to synthesize the oligomeric compound $[Ph_2SiO]_8[AlO(OH)]_4$[13] and have shown in subsequent articles how this compound can be systematically modified and used in different reactions.[14–16] Recently we gathered some of our findings in a research report which will be available shortly.[17] In the following review we have collected our most recent results with respect to two fundamental questions: (1) What are the products when $[Ph_2SiO]_8[AlO(OH)]_4$ looses water? (2) Can $[Ph_2SiO]_8[AlO(OH)]_4$ be used as a template for supramolecular chemistry? Although we can not answer these questions completely, we want to discuss our first preliminary results.

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## RESULTS

### Formation of Polycyclic $[Ph_2SiO]_8[AlO(OH)]_4$ and Reactivities

Tert-butoxyalane, $(tBu\text{-}O\text{-}AlH_2)_2$, a dimeric volatile and molecular compound, has two different ligands on aluminum that easily can be displaced using protons.[18] When $(tBu\text{-}O\text{-}AlH_2)_2$ is reacted with a proton donor like diphenylsilanediol, $Ph_2Si(OH)_2$, in organic solvents dihydrogen and tert-butanol are immediately formed. As the alane also functions as a Lewis-acid, a simultaneous condensation of diphenylsilanediol $Ph_2Si(OH)_2$ to $(HO)SiPh_2\text{-}O\text{-}SiPh_2(OH)$ occurs in the reaction mixture (Scheme 1). The polycyclic compound of the general formula $(Ph_2SiO)_8(AlOOH)_4$ is obtained in up to 75% yield and can be purified easily by recrystallization in the form of its diethylether adduct.[13] The molecule has five eight-membered cycles which are mutually connected at the aluminum atoms: The center of the molecule thus disposes of a pure $Al_4[O(H)]_4$ ring to which four $O\text{-}SiPh_2\text{-}O\text{-}SiPh_2\text{-}O$ chains are fused.

As the aluminum atoms are in an almost tetrahedral $AlO_4$ environment, the $Si_2O_4Al_2$ rings are either directed upside or downside with respect to the interior $Al_4[O(H)]_4$ ring. This aluminum oxygen ring has (as the whole molecule) in a good approximation $S_4$-symmetry, which means that the $Al_4O_4$ ring has not a crown conformation but a conformation similar to $S_4N_4$. Always two of the four hydroxy groups are pointing

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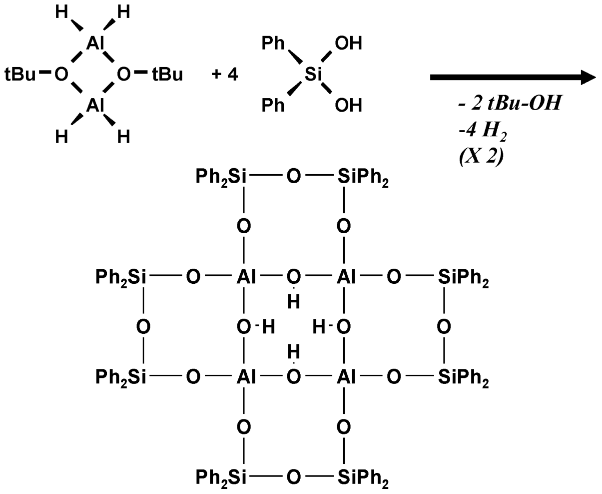

SCHEME 1

in pairs in opposite directions. On the outer sphere of the molecule phenyl groups are located wrapping up the interior inorganic skeleton. From a simple stick and ball model of $(Ph_2SiO)_8[Al(O)OH]_4$ (Figure 1) it becomes clear that the chemical access to the inner skeleton of the molecule is hindered by steric crowding of the phenyl groups. Until now

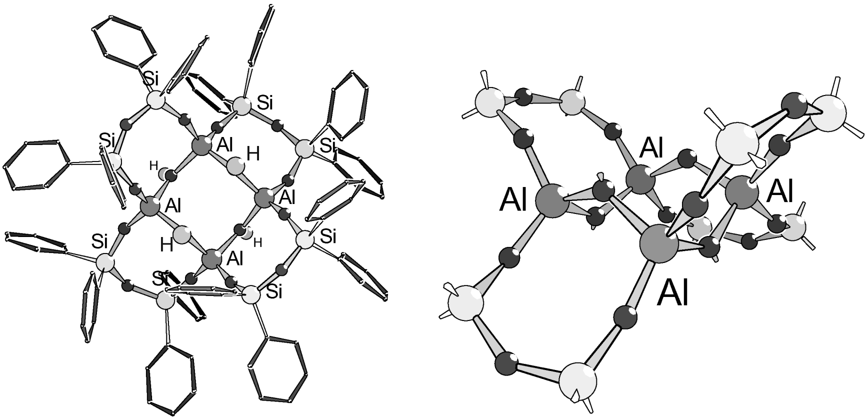

**FIGURE 1** Ball and stick representation of $(Ph_2SiO)_8(AlOOH)_4$ as result of x-ray single crystal analysis. On the left side a top-view of the molecule and all phenyl groups is shown, while on the right side the inorganic skeleton of the same molecule from a different view (almost perpendicular to the left image) is depicted.

we have established in principle three different discrete reactivities depending on the nature of the reactant:

1. The attack of organic nitrogen or oxygen Lewis bases on the hydroxyl-groups of the inner cycle is followed by the instant formation of Lewis-acid/base adducts with hydrogen bridges $O\cdots H\cdots O$ or $O\cdots H\cdots N$[13] (for further discussion see following chapters).
2. The attack of ammonia or water on the polycyclic molecule $(Ph_2SiO)_8[Al(O)OH]_4$ leads to rearrangements of the whole molecule with mostly degradations and reformations of new coordination polyhedra around aluminum.[15] In the case of the reaction with water a polycyclic molecule is obtained of the composition $[(Ph_2Si)_2O_2]_6[AlO(OH)_{0.5}]_6[Al(OH)_6] \cdot 3\ OEt_2$, which contains 6 $AlO_4$ tetrahedra and 1 $AlO_6$ octahedron. This different reactivity of these small bases compared to the larger organic bases may be explained easily by the less steric crowding of ammonia and water and therefore their possibility to attack the aluminum acidic centers of the polycycle $(Ph_2SiO)_8[Al(O)OH]_4$. Until now, this has never been observed with simple organic bases like $OR_2$ or $NR_3$ which are exclusively attracted by the protic hydrogens of the inner $Al_4(OH)_4$ cycle.
3. The displacement of the hydrogen atoms of the inner $Al_4(OH)_4$ ring in $(Ph_2SiO)_8[Al(O)OH]_4$ by monovalent and divalent metallic elements may be accomplished using organometallic compounds like butyl-lithium, phenyl-lithium, or alcoholates like potassium tert-butoxide, tin(II) or lead(II) alcoholates and amides like lithiumdimethylamide or $Sn[N(SiMe_3)_2]_2$.[14,17] In Table I a selection of compounds has been assembled, which all can be derived from $(Ph_2SiO)_8[Al(O)OH]_4$ by simple substitution of the protic hydrogen atoms. All these molecules have in common that the original skeleton of the starting compound is maintained although the metallic elements introduced in the polycycle lead to further coordination modes of the oxygen atoms.[17] In these metal derivatives of $(Ph_2SiO)_8[Al(O)OH]_4$, which can be resumed in the general formula $(Ph_2SiO)_8(AlO_2)_4(M)_n (n = 2 \text{ or } 4)$, the resemblance to solid state silicates $(SiO_2)_m(AlO_2)_nM_n$ is striking.

**TABLE I** Selection of "Molecular Alumosilicates" Obtained by Hydrogen-Substitution from $(Ph_2SiO)_8[Al(O)OH]_4$ with Persistence of the Inner Inorganic Skeleton

$(Ph_2SiO)_8(AlO_2)_4(Li{*}Et_2O)_4$
$(Ph_2SiO)_8(AlO_2)_4(Li{*}Et_2O)_2(Li{*}NH_3)(Li{*}OEt_2,NH_3)$
$(Ph_2SiO)_8(AlO_2)_2[AlO(OH)]_2Sn$
$(Ph_2SiO)_8(AlO_2)_4Sn_2$
$(Ph_2SiO)_8(AlO_2)_4Pb_2$

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Besides these reactions (with preservation of the original backbone, Table I) an important number of reactions have been observed with rearrangements of the structures implying even coordination numbers 5 of oxygen atoms around aluminum.[17]

In this preliminary review we would like to present two principally new reactivities of $(Ph_2SiO)_8[Al(O)OH]_4$. First, we would like to report on the reaction of $(Ph_2SiO)_8[Al(O)OH]_4$ towards bis(amino)alkanes, especially 1,3-diaminopropane. Secondly, we report the results obtained with hexamethyldisilazane, $(Me_3Si)_2NH$, leading for the first time to a condensation within $(Ph_2SiO)_8[Al(O)OH]_4$.

## The Reaction of $(Ph_2SiO)_8[Al(O)OH]_4$ with 1,3-Diaminopropane

As we have found earlier, $(Ph_2SiO)_8[Al(O)OH]_4$ reacts with triethylamine, diethylether, or pyridine to three different adducts even under high molar ratios of the bases under hydrogen bridging $O\cdots H\cdots O$ or $O\cdots H\cdots N$.[13] In each case the protic hydrogen atoms of the inner $Al_4(OH)_4$ ring serve as bridges between two oxygen or an oxygen and a nitrogen atom. Whereas with triethylamine a 1:2 adduct is formed, $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 2\ NEt_3$, diethylether forms a 1:3 adduct, $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 3\ OEt_2$, and pyridine (py) a 1:4 adduct, $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 4$ py.

Apparently the steric bulk of the bases combined with the flexibility of $(Ph_2SiO)_8[Al(O)OH]_4$ lead to different admittances for the bases, the less hindered base pyridine having the easiest access as the flat molecule can sandwich within the phenyl ligands of the polycycle.[13] Only three of bulkier diethylether molecules can be accommodated within $(Ph_2SiO)_8[Al(O)OH]_4$ as the twisting of the rings makes the forth O—H group not available. This is even more obvious with triethylamine, which at the basic nitrogen atom has the highest ligand number and which is the most sterically hindered molecule of the three bases. As a consequence of adaptation within $(Ph_2SiO)_8[Al(O)OH]_4$ only two molecules of triethylamine can be accommodated shown in Figure 2.

We were interested to replace these simple mono-bases by molecules which have two basic centers at the same time and which could simultaneously bind to two of the protic hydrogen atoms of the central $Al_4(OH)_4$ ring within $(Ph_2SiO)_8[Al(O)OH]_4$. As we stated earlier, two of these four central OH-groups are always pointing in the same direction and should be ideally suited for bis(amino)alkanes of the general formula $H_2N(CH_2)_nNH_2$ as host matrix. Indeed, when $(Ph_2SiO)_8[Al(O)OH]_4$ is reacted with 1,3-diaminopropane, depending on the stoichiometry

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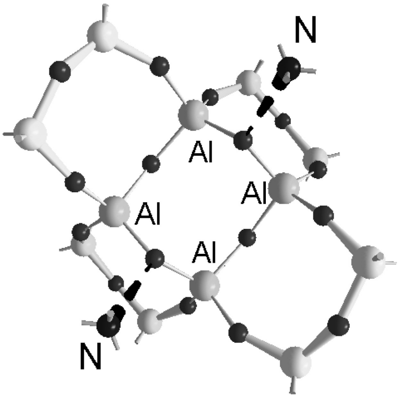

**FIGURE 2** SiOAl-backbone of the adduct $(Ph_2SiO)_8[Al(O)OH]_4{\cdot}2\ NEt_3$ omitting organic ligands for clarity. Whereas two of the four central OH-groups are involved in hydrogen bridging (fragmented lines), the two others are free due to steric hindrances. As may be seen in the picture the two eight membered $Si_2O_4Al_2$ cycles pointing in the same direction as the $NEt_3$ ligands are "opened-up," while the two other ones are "compressed," shielding the noncoordinated OH-groups.

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of the reactands, two different compounds can be isolated from organic solvents like toluene by simple crystallization in high yields (Scheme 2).

$$(Ph_2SiO)_8[Al(O)OH]_4 + n\ H_2N(CH_2)_3NH_2 \rightarrow (Ph_2SiO)_8[Al(O)OH]_4 \cdot n\ H_2N(CH_2)_3NH_2$$

$$\text{with } n = 2, 3$$

## SCHEME 2

Besides by the normal spectroscopic methods like IR and NMR, the two molecules $(Ph_2SiO)_8[Al(O)OH]_4 \cdot n\ H_2N(CH_2)_3NH_2 (n = 2, 3)$ have been characterized by single crystal x-ray diffraction techniques. In Figures 3 and 4 the skeleton of the molecular entities are shown. Whereas in the 1:2 adduct two $O{\cdots}H{\cdots}N(H)_2$-$CH_2CH_2CH_2N(H)_2{\cdots}H{\cdots}O$ loops are present connecting the OH-groups situated on different faces of the molecule, in the 1:3 adduct only one of these loops is present and the two other diaminopropane molecules coordinate to the two remaining O—H groups by one $NH_2$-side leaving the other free (the free nitrogen atoms in the picture are designated as N′). It is interesting to note that the mono coordinated diaminopropane molecules are tighter bonded to the O—H groups of the central ring which can be deduced from the neighboring Al—O bonds

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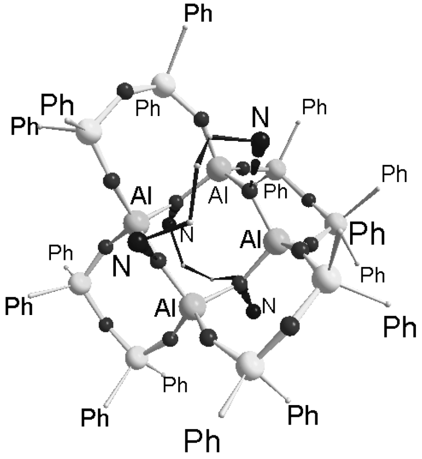

**FIGURE 3** Ball and stick representation of $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 2\ H_2N(CH_2)_3NH_2$ with two diaminopropane molecules coordinating simultaneously two OH-groups of the mother molecule trough hydrogen bridging. The phenyl groups are omitted for clarity. The non designed atoms are oxygen or silicon. The bridging loops $O \cdots H \cdots N(H)_2\text{-}CH_2CH_2CH_2N(H)_2 \cdots H \cdots O$ combine 9 atoms and are situated on the two opposite faces of the molecule.

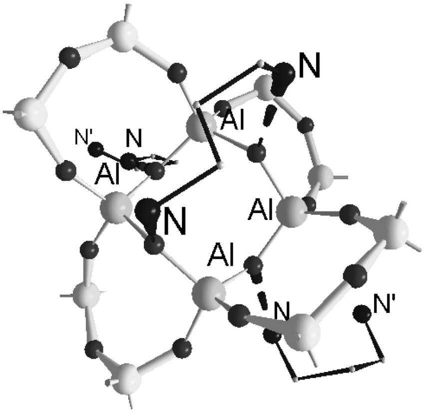

**FIGURE 4** Ball and stick representation of $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 3\ H_2N(CH_2)_3NH_2$ with one diaminopropane molecule coordinating simultaneously two OH-groups of the mother molecule trough hydrogen bridging and two other diaminopropane molecules coordinating only by one end. The non coordinating nitrogen atoms are designed by N′. Phenyl groups have been omitted; compare also caption to Figure 3.

within the ring (in $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 3\ H_2N(CH_2)_3NH_2$ the hydrogen bridges between N···O are almost similar for the loop [2.65(1) Å] and the mono coordinated species [2.65(1) Å] but the neighboring Al—O distances are distinctly different for the O—H group involved in the loop [1.81(1) Å] compared to the mono coordinated O—H groups [Al—O = 1.73(1) Å]).

By the way the engagement of the hydrogen atom in the hydrogen bridge and the neighboring Al—O distances should depend of the $pK_a$ value of the base (as we have shown earlier).[15] The value found for Al—O close to the O···H···N bridge engaged by the mono-hapto diaminopropane in $(Ph_2SiO)_8[Al(O)OH]_4 \cdot 3\ H_2N(CH_2)_3NH_2$ is with 1.732(9) Å very similar to the corresponding value in the triethylamine adduct (Al—O = 1.728(8) Å) which is in perfect agreement of the $pK_a$ values of the two bases ($pK_a$[triethylamine] = 11.0, $pK_a$[diaminopropane] = 10.5) being almost equal.

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## Reaction of $(Ph_2SiO)_8[Al(O)OH]_4$ with Hexamethyldisilazane

When the alumosiloxane $(Ph_2SiO)_8[Al(O)OH]_4$ is allowed to react with hexamethydisilazane in tetrahydrofurane a new polycyclic compound of the general formula $[Ph_2SiO]_8[AlO_2]_2[AlOO\text{-}SiMe_3]_2[NH_4{\cdot}THF]_2$ can be isolated in 45% yield. The structure of the new compound has been established by x-ray diffraction methods (Figure 5). As can be deduced

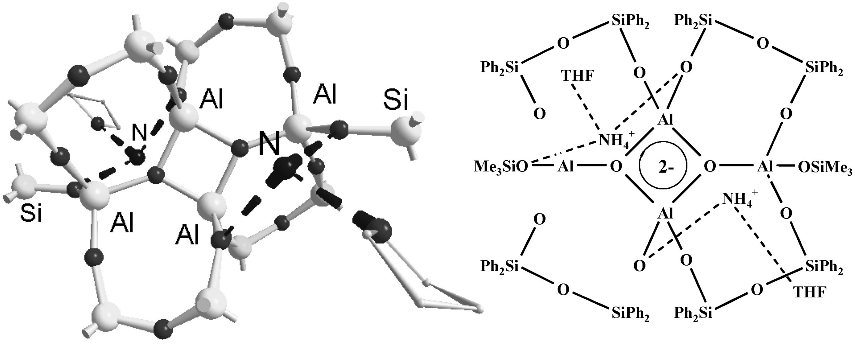

**FIGURE 5** Ball and stick representation (left side) and two-dimensional formula (right side) of $[Ph_2SiO]_8[AlO_2]_2[AlOO\text{-}SiMe_3]_2[NH_4{\cdot}THF]_2$. The phenyl and methyl groups have been omitted for clarity. The ammonium cations (N) show hydrogen bridges to THF molecules and two oxygen atoms of the polycyle or the terminal siloxy group. The two negative charges compensating the ammonium ions are randomly distributed over the polycycle.

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from Figure 5 the original polycycle has been altered completely: The central part of the molecule is made up of four almost equiplanar aluminum atoms, which are linked by two three coordinated oxygen atoms forming an $Al_2O_2$ square with two Al—O sticks. The point-symmetry of the whole molecule is $C_i$.

All aluminum atoms are bound to two different —O—$Ph_2Si$—O—$SiPh_2$—O— loops. The two external aluminum atoms have a further trimethylsiloxy-ligand; the former OH-groups of the starting compound $(Ph_2SiO)_8[Al(O)OH]_4$have all vanished, two of them presumably through proton/trimethylsilyl exchange. Into the polycyle, which has a 2-charge (4 $OPh_2Si$—O—$SiPh_2O^{2-}$ + 2 $O^{2-}$ + 2 $Me_3SiO^-$ + 4 $Al^{3+}$) two ammonium ions are incorporated and fixed through N—H···O hydrogen bridges (dashed lines in the Figure 5). Two tetrahydrofurane molecules participate from the outer sphere of the molecule to these hydrogen bonds and also can be easily recognized in the figure. We have tested by independent methods that the cationic species are ammonium and not oxonium, which are difficult to distinguish in the x-ray experiments. In essence the reaction of $(Ph_2SiO)_8[Al(O)OH]_4$ with hexamethyldisilazane leads to a complete decomposition of the silylamine and a dehydration of the alumosiloxane.

This last assumption has indeed been successfully verified. Apart from $[Ph_2SiO]_8[AlO_2]_2$-$[AlOO\text{-}SiMe_3]_2[NH_4]_2$ we could also isolate in low yield the compound $[Ph_2SiO]_8[AlOO_{0.5}]_4\cdot 2$ py, which is formally the inner-condensation product of the starting molecule (see caption of Figure 6), by using toluene as solvent and by adding high ratios of pyridine to the reaction of $(Ph_2SiO)_8[Al(O)OH]_4$ with hexamethyldisilazane.

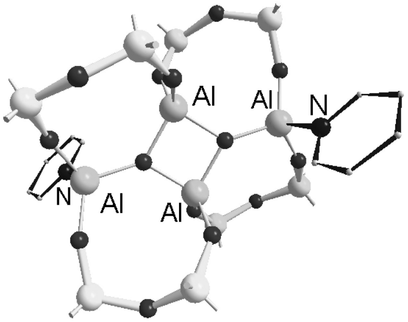

**FIGURE 6** Ball and stick model of $[Ph_2SiO]_8[AlOO_{0.5}]_4\cdot 2$ py. Refer also to the caption of Figure 5. The molecule is neutral and can formally be obtained from the starting compound by water elimination: $(Ph_2SiO)_8[Al(O)OH]_4 - 2\ H_2O + 2$ py.

As can be deduced from Figure 6 the structure of $[Ph_2SiO]_8[AlOO_{0.5}]_4 \cdot 2$ py is very similar to $[Ph_2SiO]_8[AlO_2]_2[AlOO\text{-}SiMe_3]_2[NH_4 \cdot THF]_2$ and results formally by subtraction of 2 $Me_3SiO^-NH_4^+ \cdot$ THF from the starting compound and addition of pyridine to the three coordinated aluminum atoms.

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