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# Synthesis and Structure of the First Soluble Ternary Metal Amide−Imide Compounds with an $M_2Al_2Si_2N_6$ Core ($M = Li, Na$)

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*Summary: The reaction of the silyl triamide 2,6-i-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)_3$ (**1**) with $MAlMe_4$ yielded ionic aluminosilazanes, [2,6-i-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)(NH)_2AlMe_2]_2[M\cdot THF]_2$ ($M = Li$ (**2**), Na (**3**)), the first examples of ionic aluminosilazanes containing both amido and imido groups. The molecular structures of **2** and **3**, determined by single-crystal X-ray diffraction, show that these drum-shaped aluminosilazanes have a central $M_2Al_2Si_2N_6$ core.*

## Introduction

Since the first preparation of metallasiloxanes toward the end of the 19th century,[1−3] several research groups around the world have been contributing continuously to our basic understanding of the structural and catalytic aspects of silicate-based materials.[4−8] In the past few years, we have taken up a new and unique approach in understanding the structures of the naturally occurring metallasilicates and zeolites by synthesizing soluble model compounds in the laboratory, starting from simple metal precursors and silanols.[9−11] Also in recent years the importance of nitridosilicates in materials science has been growing. New nitridosilicates with a highly polymeric, three-dimensional framework and incorporated cations have been synthesized using silicon diimide, $[Si(NH)_2]_n$, and various metals at high temperatures.[12−18]

Our success in the preparation of neutral and ionic metallasiloxanes[9−11] and the valence isoelectronic neutral metallasilazanes[19−25] coupled with the recent interest in nitridosilicate chemistry prompted us to explore the possibility of extending this chemistry to the synthesis of ionic metallasilazanes. We report herein the synthesis of the first soluble ionic aluminosilazanes, [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)(NH)_2AlMe_2]_2[M\cdot THF]_2$ ($M = Li$(**2**), Na(**3**)), and their unambiguous structural characterization.

## Results and Discussion

The reaction between 2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)_3$ (**1**)[19] and an equivalent quantity of $MAlMe_4$ ($M = Li$, Na) in a 5:1 mixture of boiling toluene and THF for 3 h gave compounds **2** (yield: 72%) and **3** (yield: 77%) (Scheme 1).

Compounds **2** and **3** have been fully characterized by elemental analysis and mass, IR and NMR spectroscopy.

The EI-MS spectrum of **2** shows a fragment ion at *m/z* 450 for $M^+ - $ 2,6-*i*-$Pr_2C_6H_3NSiMe_3 - 4\ Me - 2\ Li - 2\ THF$ (5%), and that of **3** gives a peak at *m/z* 418 for $M^+ - $ 2,6-*i*-$Pr_2C_6H_3NSiMe_3 - 2\ NH_2 - 4\ Me - 2\ Na - 2\ THF$ (3%). For both compounds the base peak observed at *m/z* 162 corresponds to the aryl cation 2,6-*i*-$Pr_2C_6H_4^+$. The infrared spectra of **2** and **3** show three characteristic absorptions (3402, 3380, and 3353 $cm^{-1}$ (**2**); 3498, 3373, and 3334 $cm^{-1}$ (**3**)), which are assigned to the N−H stretching frequencies. The $^1H$ and $^{29}Si$ NMR data in solution are consistent with the structures of **2** and **3**

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[†] Dedicated to Professor Leopold Horner on the occasion of his 90th birthday.

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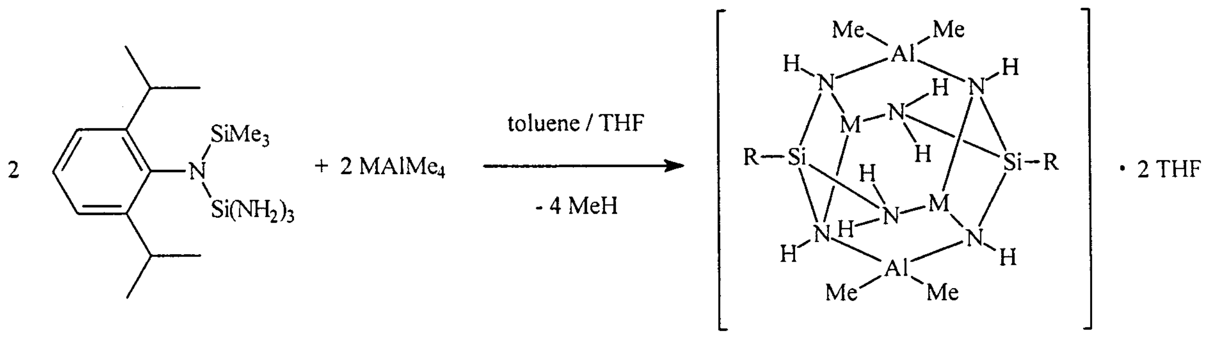
**Scheme 1. Reaction of 2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)_3$ (1) and $MAlMe_4$ (M = Li, Na)**



1

M = Li (2), Na (3)

R = 2,6-*i*-$Pr_2C_6H_3N(SiMe_3)$

**Table 1. Crystal Data and Structure Refinement Details for [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)SiNH_2(NH)_2AlMe_2]_2[Li\cdot THF]_2$ (2) and [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)SiNH_2(NH)_2AlMe_2]_2[Na\cdot THF]_2$ (3)**

| | 2 | 3 |
|---|---|---|
| empirical formula | $C_{42}H_{88}Al_2Li_2N_8O_2Si_4$ | $C_{42}H_{88}Al_2N_8Na_2O_2Si_4$ |
| fw | 917.40 | 949.50 |
| temp, K | 203(2) | 200(2) |
| cryst syst | triclinic | monoclinic |
| space group | $P\bar{1}$ | $P2_1/c$ |
| unit cell dimens | | |
| *a*, Å | 10.486(3) | 14.740(4) |
| *b*, Å | 11.714(4) | 14.453(2) |
| *c*, Å | 12.264(4) | 14.436(7) |
| α, deg | 101.65(4) | 90 |
| *β*, deg | 90.53(2) | 111.65(4) |
| *γ*, deg | 110.466(12) | 90 |
| *V*, Å$^3$; *Z* | 1377.0(8); 1 | 2859(2); 2 |
| calcd density, Mg/m$^3$ | 1.106 | 1.103 |
| abs coeff, mm$^{-1}$ | 0.179 | 0.188 |
| *F*(000) | 500 | 1032 |
| cryst size, mm | 0.70 × 0.30 × 0.10 | 0.60 × 0.50 × 0.10 |
| *θ* range for data collecn, deg | 3.52−22.55 | 2.05−22.49 |
| limiting indices | $-11 \le h \le 6$<br>$-12 \le k \le 12$<br>$0 \le l \le 13$ | $-14 \le h \le 15$<br>$-12 \le k \le 15$<br>$-7 \le l \le 15$ |
| no. of rflns collected | 4053 | 3919 |
| no. of indep rflns | 3191 ($R_{int}$ = 0.0182) | 3462 ($R_{int}$ = 0.0313) |
| no. of data/restraints/params | 3188/0/288 | 3459/0/288 |
| goodness of fit on $F^2$ | 1.068 | 1.070 |
| final *R* indices ($I > 2\sigma(I)$) | | |
| R1 | 0.0371 | 0.0580 |
| wR2 | 0.0891 | 0.1481 |
| *R* indices (all data) | | |
| R1 | 0.0435 | 0.0707 |
| wR2 | 0.0978 | 0.1636 |
| largest diff peak and hole, e Å$^{-3}$ | 0.245, −0.230 | 0.674, −0.370 |

in the solid state. The $^1$H NMR spectrum shows, in addition to the signals for the organic substituent R and the coordinated THF, three signals due to the $AlMe_2$ (−0.51 (**2**), −0.43 (**3**) ppm), the $NH_2$ (1.12 (**2**), 0.91 (**3**) ppm) and the NH moieties (1.75 (**2**), 1.38 (**3**) ppm).

Crystal data for compounds **2** and **3** are summarized in Table 1. Selected bond distances and angles are given in Table 2. Single crystals suitable for X-ray diffraction studies were obtained by crystallization from THF (**2**) and from toluene (**3**) at ambient temperature. The molecular structures are shown in Figures 1 and 2.

**Table 2. Selected Bond Distances (Å) and Bond Angles (deg) for [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)SiNH_2(NH)_2AlMe_2]_2[Li\cdot THF]_2$ (2), [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)SiNH_2(NH)_2AlMe_2]_2[Na\cdot THF]_2$ (3), and [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)NSi(NH)_3(AlMe)AlMe_2]_2$ (4)[a]**

| | **2** (M = Li) | **3** (M = Na) | **4** |
|---|---|---|---|
| Si(1)−N(2) | 1.759(2) | 1.759(3) | 1.684(2) |
| Si(1)−N(3) | 1.698(2) | 1.694(3) | 1.775(2) |
| Si(1)−N(4) | 1.698(2) | 1.693(4) | 1.780(2) |
| Al(1)−N(3A) | 1.888(2) | 1.880(3) | 1.940(2) |
| Al(1)−N(4) | 1.887(2) | 1.881(3) | 1.951(2) |
| M(1)-N(2) | 2.063(5) | 2.385(4) | |
| M(1)-N(3A) | 2.158(5) | 2.590(4) | |
| M(1)-N(4A) | 2.166(5) | 2.508(4) | |
| N(1)−Si(1)−N(2) | 115.05(10) | 113.6(2) | 114.2(1) |
| N(1)−Si(1)−N(3) | 110.66(11) | 108.9(2) | 115.4(1) |
| N(1)−Si(1)−N(4) | 111.38(11) | 108.8(2) | 112.8(1) |
| N(2)−Si(1)−N(3) | 106.26(11) | 104.3(2) | 108.8(1) |
| N(2)−Si(1)−N(4) | 103.13(11) | 107.5(2) | 113.3(1) |
| N(3)−Si(1)−N(4) | 109.99(11) | 113.9(2) | 92.50(9) |
| N(4)−Al(1)−N(3A) | 106.05(11) | 107.5(2) | 100.47(8) |
| N(2)−M(1)−N(3A) | 111.4(2) | 98.21(13) | |
| N(2)−M(1)−N(4A) | 112.4(2) | 107.04(13) | |
| N(3A)−M(1)−N(4A) | 80.1(2) | 67.65(12) | |

[a] Symmetry transformations used to generate equivalent atoms (#1): −*x*, −*y*, -*z* + 1 (**2**); −*x* + 1, −*y*, −*z* + 2 (**3**); −*x*, −*y*, −*z* (**4**).

Compounds **2** and **3** lie on a crystallographic center of symmetry. Both molecules **2** and **3** are isostructural, and they have a drum-shaped central core which is made up of two types of six-membered rings and two four-membered rings. The two Si−N−Al−N−M−N six-membered rings are in a twist-boat conformation. These two rings are joined to each other by two Si−N−M−N rings which deviate considerably from planarity. The mean deviation shown by the atoms in this four-membered ring is 6.50 (**2**) and 1.17 Å (**3**). The geometry around each atom in the drum is tetrahedral. The amino moieties of the silyltriamide are partially reacted and two of the amino groups are in the imido (NH) form and one is in the amido ($NH_2$) form. The average Si−N(imido) bond is slightly shorter (1.70 Å (**2**), 1.69 Å (**3**)) than the Si−N(amido) bond (1.76 Å (**2**, **3**)). The average M−N(imido) bond is slightly longer (2.16 Å (**2**), 2.55 Å (**3**)) than the M−N(amido) bond (2.06 Å (**2**), 2.39 Å (**3**)). One molecule of THF is coordinated to each alkali-metal ion, which are tetracoordinated with a distorted tetrahedral geometry. Each aluminum center is tetrahedral and is bonded to two methyl groups and to the

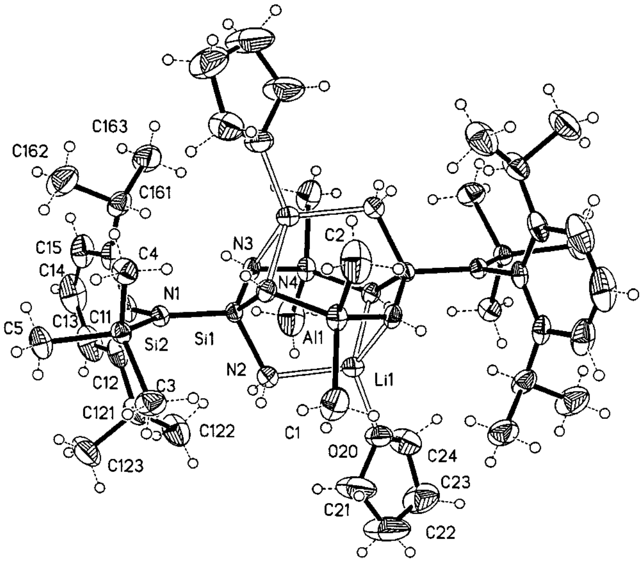


**Figure 1.** Crystal structure of [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)$-$Si(NH_2)(NH)_2AlMe_2]_2[Li{\cdot}THF]_2$ (**2**) showing 50% probability of the thermal ellipsoids and the atom-numbering scheme.

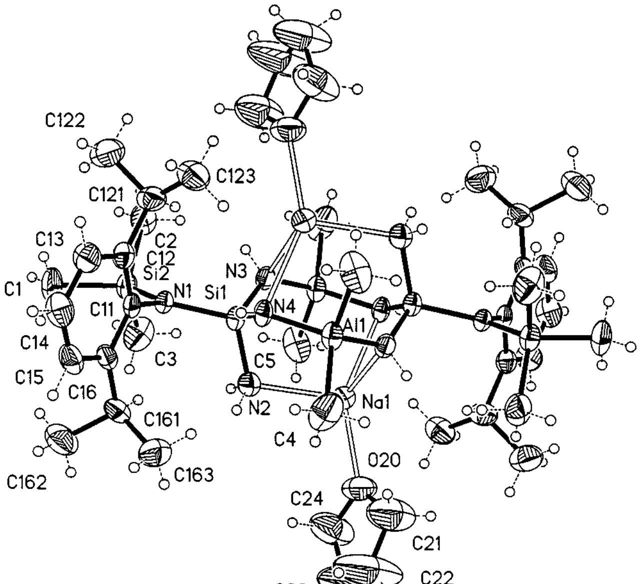


**Figure 2.** Crystal structure of [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)$-$Si(NH_2)(NH)_2AlMe_2]_2[Na{\cdot}THF]_2$ (**3**) showing 50% probability of the thermal ellipsoids and the atom-numbering scheme.

two imido nitrogens. The average Al−N bond lengths are 1.89 Å (**2**) and 1.88 Å (**3**), respectively. It is remarkable that the comparable Si−N and Al−N bond lengths in **2** and **3** are nearly the same.

The only other structurally characterized compound with a comparable structure is the nonionic metallasilazane [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)NSi(NH)_3(AlMe)AlMe_2]_2$ (**4**)[22,25] (selected bond distances and angles are given in Table 2). The difference in the structure of **4** is that the alkali-metal ions are replaced by Al−Me groups and no THF is coordinated within the molecule (Figure 3).

The Si−N(imido) and the Al−N(imido) bond lengths in **2** and **3** are slightly shorter than those found in **4** (average Si−N 1.75 Å; average Al−N 1.96 Å). Compound **4** has also a tetrahedral geometry around each atom in the drum. The N−Al−N angles in **2** (106.1°) and **3** (107.5°) are slightly greater than those reported for **4** (100.5°).

In summary, we have shown that the discrete silane triamide **1** is an excellent synthon for generating a new class of soluble metal amide−imide systems containing Al, N, Si, and alkali metal in the framework. The synthetic methodology is likely to be quite general and can possibly be extended to the preparation of other metal-containing oligomeric silazanes. Furthermore, the presence of easily hydrolyzable Al−C bonds, apart from several reactive N−H groups, offer possible access to supramolecular networks based on silazane frameworks. We are actively pursuing these aspects.

## Experimental Section

**General Methods.** All experimental manipulations were carried out using standard Schlenk procedures. Solvents were dried according to standard methods and freshly distilled prior to use. NMR spectra were recorded on a Bruker AM 200, a Bruker MSL 400, or a Bruker Avance 500 instrument. The chemical shifts are reported in ppm relative to external $SiMe_4$. IR spectra were recorded on a Bio-Rad Digilab FTS 7 spectrometer. Mass spectra were obtained on a Finnigan MAT 8230 system and a Varian MAT CH5. Melting points were obtained on a Büchi B-500 instrument and are reported uncorrected. Elemental analyses were performed at the analytical laboratory of the Institute of Inorganic Chemistry, Universität Göttingen.

**Starting Materials.** 2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)_3$, $LiAlMe_4$, and $NaAlMe_4$ were prepared using literature procedures.[19,26]

**Synthesis of [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)(NH)_2AlMe_2]_2$-$[Li{\cdot}THF]_2$ (2).** $LiAlMe_4$ (0.43 g, 5.00 mmol) dissolved in toluene/THF (10 mL/10 mL) was added to a solution of **1** (1.62 g, 5.00 mmol) in toluene (40 mL). The solution was heated under reflux for 3 h, during which time the evolution of methane ceased and a colorless solid precipitated. After the mixture was cooled to room temperature, the precipitate was filtered. Analytically pure **2** was obtained by washing the crude product with *n*-hexane (10 mL). Yield: 1.64 g (1.79 mmol, 72%). Mp: 226 °C dec. Anal. Calcd for $C_{42}H_{88}Al_2Li_2N_8O_2Si_4$ ($M_r$ = 917.40): C, 55.0; H, 9.7; N, 12.2. Found: C, 54.9; H, 9.9; N, 12.0. MS (EI, 70 eV): *m/z* 450 (5, $M^+$ − 2,6-*i*-$Pr_2C_6H_3$-$NSiMe_3$ − 4 Me − 2 Li − 2 THF), 162 (100, 2,6-*i*-$Pr_2C_6H_4^+$). IR (KBr, Nujol): 3402, 3380, 3353, 1568, 1312, 1248, 1178, 1106, 1063, 1045, 964, 951, 926, 901, 871, 838, 799, 776, 738, 677, 637, 575, 525, 507, 473 $cm^{-1}$. $^1H$ NMR (200 MHz, $C_5D_5N$, ppm): $\delta$ −0.51 (s, 12 H, $AlCH_3$), 0.28 (s, 18 H, $SiCH_3$), 1.12 (s, 4 H, $NH_2$), 1.27, 1.28 (d, $^3J_{HH}$ = 6.9 Hz, 12 H, $CH(CH_3)_2$), 1.59 (m, 8 H, THF $CH_2$), 1.75 (s, 4 H, N*H*), 3.63 (m, 8 H, THF $OCH_2$), 3.79 (sept, $^3J_{HH}$ = 6.9 Hz, 4 H, $CHMe_2$), 7.10−7.19 (m, 6 H, arom *H*). $^{29}Si$ NMR (50 MHz, DMSO-$d_6$, ppm): $\delta$ −50.1 ($SiN_3$), − 13.8 ($SiMe_3$).

**Synthesis of [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)(NH)_2AlMe_2]_2$-$[Na{\cdot}THF]_2$ (3).** To a solution of **1** (1.62 g, 5.00 mmol) in toluene (50 mL) was added a solution of $NaAlMe_4$ (0.55 g, 5.00 mmol) in THF (10 mL). The reaction mixture was heated under reflux for 3 h, during which time the evolution of methane ceased. After the mixture was cooled, the solution was concentrated in vacuo (10 mL). The resulting solution was cooled at 0 °C overnight to yield 1.82 g (1.92 mmol, 77%) of analytically pure **3**. Mp: 246 °C dec. Anal. Calcd for $C_{42}H_{88}Al_2N_8Na_2O_2Si_4$ ($M_r$ = 949.50): C, 53.1; H, 9.3; N, 11.8. Found: C, 52.9; H, 9.4; N, 11.7. MS (EI, 70 eV): *m/z* 418 (3, $M^+$ − 2,6-*i*-$Pr_2C_6H_3NSiMe_3$ − 2 $NH_2$ − 4 Me − 2 Na − 2 THF), 162 (100, 2,6-*i*-$Pr_2C_6H_4^+$).

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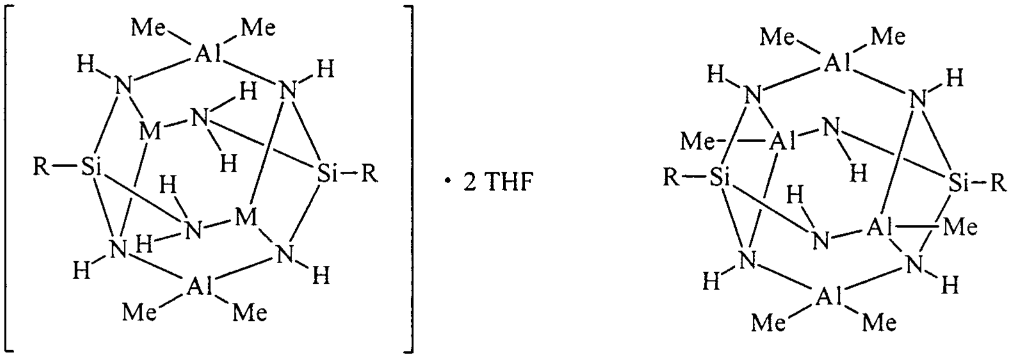

M = Li (**2**), Na (**3**)

**4**

R = 2,6-*i*-$Pr_2C_6H_3N(SiMe_3)$

**Figure 3.** [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH_2)(NH)_2AlMe_2]_2[M{\cdot}THF]_2$ (M = Li (**2**), Na (**3**)) and [2,6-*i*-$Pr_2C_6H_3N(SiMe_3)Si(NH)_3(AlMe)AlMe_2]_2$ (**4**).

IR (KBr, Nujol): 3498, 3373, 3334, 1562, 1312, 1248, 1182, 1106, 1078, 1053, 957, 929, 903, 871, 839, 801, 780, 673, 632, 543, 485, 460, 442 $cm^{-1}$. $^1H$ NMR (200 MHz, $C_5D_5N$, ppm): $\delta$ −0.43 (s, 12 H, $Al(CH_3)_2$), 0.38 (s, 18 H, $Si(CH_3)_3$), 0.91 (s, 4 H, $NH_2$), 1.22 (d, $^3J_{HH} = 6.8$ Hz, 12 H, $CH(CH_3)_2$), 1.38 (s, 4 H, NH), 1.43 (d, $^3J_{HH} = 6.8$ Hz, 12 H, $CH(CH_3)_2$), 1.56 (m, 8 H, THF $CH_2$), 3.60 (m, 8 H, THF $OCH_2$), 4.13 (sept, $^3J_{HH} = 6.8$ Hz, 4 H, $CHMe_2$), 6.95−7.16 (m, 6 H, arom H). $^{29}Si$ NMR (99 MHz, $C_5D_5N$, ppm): $\delta$ −32.2 ($SiN_3$), − 0.2 ($SiMe_3$).

**Crystal Structure Determination for 2 and 3.** Data for the structures of **2** and **3** were collected on a Stoe AED2 diffractometer; Mo Kα ($\lambda = 0.710\ 73$ Å) radiation was used. The structure solution (direct methods) and refinement (by full-matrix least squares on $F^2$) were carried out using the SHELXS-86[27] and the SHELXL-93 programs.[28] All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were included using the riding model with its $U_{iso}$ value tied to the $U_{iso}$ value of the parent atom and were refined isotropically.

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**Supporting Information Available:** Tables giving positional and thermal parameters and estimated standard deviations for all atoms, bond distances, bond angles, and anisotropic thermal parameters for **2** and **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

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