An Unusual Alkyltrihydroaluminate. Crystal Structures of $[AlH_2\{C(SiMe_3)_2(SiMe_2OMe)\}(\mu\text{-}H)Li(THF)]$, $[AlCl_2\{C(SiMe_2OMe)(SiMe_3)_2(THF)]$, and $[Li\{C(SiMe_2OMe)(SiMe_3)_2\}(THF)_2]$ (THF = Tetrahydrofuran)

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Received May 19, 1998

Summary: Reaction of the organolithium reagent $[Li\{C(SiMe_2OMe)(SiMe_3)_2\}(THF)_2]$ *(THF = tetrahydrofuran),* ***1****, with* $LiAlH_4$ *gave the title organotrihydridoaluminate,* ***2****, which is unusual for an alkali metal organotrihydridoaluminate in being monomeric and in having the Al linked to the Li not only through hydride but also through another of its ligands. The corresponding reaction with* $AlCl_3$ *gave the title organoaluminum dichloride. The Si−O bonds in* ***1*** *and* ***2*** *are not significantly lengthened by the O−metal coordination. The bond from Li to the OMe in* ***1*** *oxygen is not significantly different in length from that to the THF oxygen.*

Introduction

We recently introduced use of the ligand $(MeOMe_2Si)(Me_3Si)_2C^-$, R*, in which there is comparable bulk around the carbanion center to that in the much used[1] ligand $(Me_3Si)_3C^-$ but which in addition has a neutral donor group available for further coordination, and we employed it to obtain novel derivatives of Li,[2] Mg,[2] Yb,[3] Zn,[4a] Cd,[4b] Hg,[4a] Sn,[5] and Pb.[5] We have now used the lithium reagent $[LiR^*(THF)_2]$, **1**,[2] to generate the alkyltrihydridoaluminate $[AlH_2R^*(\mu\text{-}H)Li(THF)]$, **2,** and the alkylaluminum dichloride $[AlCl_2R^*(THF)]$, **3**, and shown that the ligand has a different ligating mode in each of these compounds. Two of these modes, **A** and **B**, have been previously observed,[2−5] but the third, **C**, is new.

Results and Discussion

Reaction of the organolithium reagent **1** with $LiAlH_4$ in THF followed by recrystallization of the product from light petroleum gave the lithium alkylaluminum hydride **2**, with the structure shown in Figure 1 and the geometrical data given in Table 1.

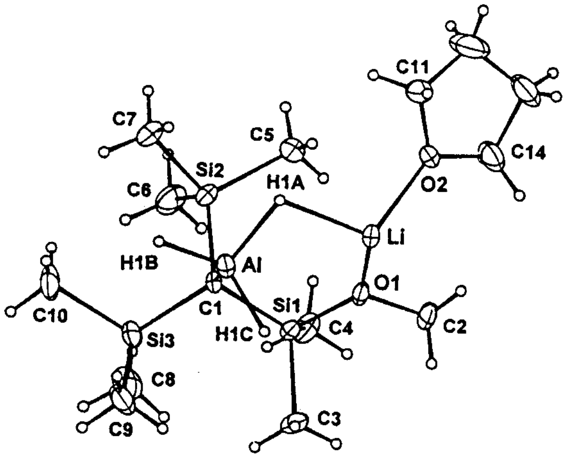
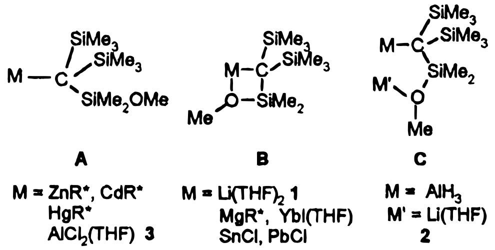


Figure 1. Molecular structure of $[AlH_2\{C(SiMe_3)_2(SiMe_2OMe)\}(\mu\text{-}H)Li(THF)]$, **2**.



The compound is unusual for an alkali metal organotrihydridoaluminate[6] in that (a) it is monomeric, whereas the others form dimers as a result of hydride bridging (the most relevant examples in the present context being the dimers **4a**−**e**[7,8] formed from LiR and $LiAlH_4$ or AlH_3-NMe_3), and (b) the Al is linked to the alkali metal not only through a hydride bridge but also through one of

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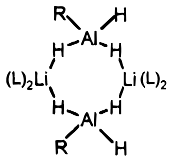
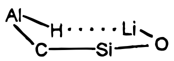
S0276-7333(98)00402-6 CCC: $15.00

Table 1. Selected Bond Lengths (Å) and Angles (deg) in 2

Al−H1A	1.66(7)	Si1−O1	1.699(4)
Al−H1B	1.66(7)	C1−Si1	1.842(5)
Al−H1C	1.57(7)	C1−Si2	1.868(6)
Al−C1	2.020(6)	C1−Si3	1.876(8)
Li−O1	1.932(11)	O1−C2	1.430(7)
Li−O2	1.926(10)	Me−Si (mean)	1.876(3)
Al−H1A−Li	106(4)	C1−Si1−O1	107.4(2)
Al−C1−Si1	107.3(3)	Si1−O1−C2	118.2(4)
Al−C1−Si2	107.1(3)	Si1−O1−Li	132.0(3)
Al−C1−Si3	105.4(3)	C2−O1−Li	108.5(4)
Si1−C1−Si2	111.9(3)	O1−Li−H1A	107(2)
Si1−C1−Si3	111.5(3)	O2−Li−H1A	106(2)
Si2−C1−Si3	113.2(3)	O1−Li−O2	116.1(6)
		Me−Si−Me (mean)	105.8(4)

its other ligands, the OMe group being coordinated to Li.





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4a	R = $(Me_3Si)_3C$	L = THF
4b	R = $(Me_2PhSi)_3C$	L = THF
4c	R = 2,4,6-$Bu^t_3C_6H_2$	L = THF
4d	R = 2,4,6-$Ph_3C_6H_2$	L = THF
4e	R = 2,6-(2,4,6-$Me_3C_6H_2)C_6H_3$	L = Et_2O

The hydrogen atoms attached to Al were located and refined. The bridging and terminal Al−H bonds were found not to be detectably different in length and to have a mean length of 1.61(7) Å, not significantly different from those in **4b**,[7] **4c,d**,[8a] and **4e**.[8b] The Li−H distance is markedly longer, at 1.94(7) Å (not significantly different from the corresponding distances in $LiAlH_4$, 1.88−2.00 Å,[9] or **4b−d)**. The compound is thus best described as an intimately bound $[AlRH_3]^-$ anion and $[Li(THF)]^+$ cation, the binding involving not only the Al−H⋯Li bridge but also the MeO−Li coordination. The form of the six-membered ring is shown as **5**; the Al−H−Li angle is 106(4)°, and the Al⋯Li distance is 2.891 Å.

There is near-planar geometry at the O atom, the sum of the angles being 358.7°. The Si−O bond, 1.699(4) Å, is distinctly longer than that of normal Si−O(alkyl) bonds, which have lengths in the range 1.64−1.66 Å,[10] but it is similar in length to the Si−O bonds in $[\{YbR^*I(OEt_2)\}_2]$, 1.684(10) Å,[3] or $[MgR^*_2]$, 1.705(6) Å,[2] in each of which there is MeO−metal coordination. It is noteworthy that the MeO−Li bond is not significantly different in length from that between Li and THF even though alkoxysilanes are normally thought to be much weaker bases, and thus poorer donors, than ethers.[11]

The geometry of the $(Me_3Si)_2(MeOMe_2Si)C$ group (mean Me_3Si−C bond length 1.872(6) Å, mean Si−Me bond length 1.876(7) Å, mean Si−C1−Si angle 112.4(3)°, mean Me−Si−Me angle 105.8(4)°) is much as expected from the electronegativity of Al.[12] The (endocyclic) C−$SiMe_2OMe$ bond, 1.842(5) Å, may be shorter than the (exocyclic) C−$SiMe_3$ bonds.

The NMR spectra of **2** in toluene-d_8 showed that the solid-state structure was essentially preserved in solution. The expected ^1H NMR signals were observed for the $SiMe_3$, $SiMe_2$, and OMe groups and the THF, but those from the AlH_3 group were very broad and difficult to locate even at −73 °C. However, when the ^{27}Al nuclei were decoupled, a broad AlH signal centered on δ 3.19, close to that of the OMe signal at δ 3.22, was clearly observed. The expected signals were observed in the ^{13}C NMR spectrum except, presumably as a result of quadrupolar interactions with the Al atom, that from the CSi_3 carbon. The chemical shift of the ^{27}Al signal, δ 117, is close to those for the dimeric hydrides **4a** and **4b**,[7] suggesting that the four-coordination is preserved in solution. The ^{29}Si spectrum showed signals for the $SiMe_3$ and $SiMe_2OMe$ groups at δ −4.1 and +22.8, respectively. The latter value, being fairly close to that, δ 15.8, for the corresponding group in **3**, might in itself suggest the absence of MeO−Li bonding, but the $^6Li\{^1H\}$ spectrum showed that for irradiation at various frequencies in the ^1H spectrum (cf. ref 13), the largest nuclear Overhauser effect (NOE) was that for irradiation of the OMe protons, indicating that these were close to the Li nucleus. A somewhat smaller NOE was observed for the α-protons of the THF, and there was a smaller but unambiguous NOE over the region in which the AlH signals appeared, indicating that Al−H⋯Li interaction persisted. Because the technique reveals only time-averaged effects, it is possible that the Li−H bonds are transiently broken in solution, perhaps with exchange of terminal and bridging Al−H bonds.

Reaction of **1** with $AlCl_3$ in THF followed by recrystallization of the product from light petroleum afforded the compound $[AlCl_2R^*(THF)]$, **3**. This was shown by an X-ray diffraction study to have the structure depicted in Figure 2 (with the geometrical data shown in Table 2), showing the OMe group not to be involved in coordination. (We think it likely that if the compound were formed without coordinated THF, it would have the OMe group coordinated to the Al.) The mean Si−C−Si angle, 110.2(4)°, may be a little smaller than that, 112.2(3)°, in **2**, which would be consistent with the smaller size of the substituents on the Al in the latter. The NMR spectra in solution in toluene-d_8 showed the expected signals, including one at δ 15.8 for the $SiMe_2$-OMe group in the ^{29}Si NMR spectrum (cf. δ 17.2 for the parent species $HC(SiMe_2OMe)(SiMe_3)_2$) and one at δ 115 in the ^{27}Al spectrum (cf. δ 108 in 2,4,6-$^tBu_3C_6H_2$-$AlCl_2\cdot THF$[8c] and δ 123 in $(Me_3Si)_3CAlCl_2\cdot THF$[14]).

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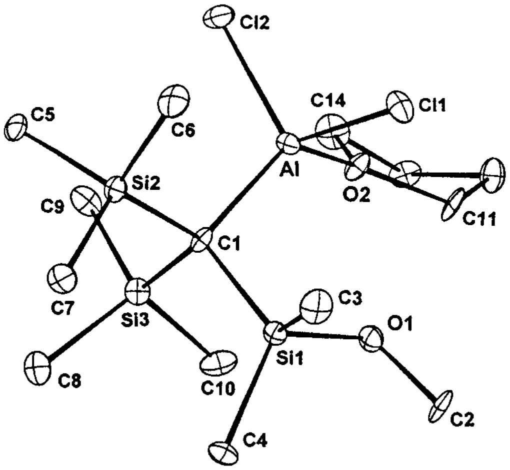

Figure 2. Molecular structure of $[AlCl_2\{C(SiMe_2OMe)(SiMe_3)_2(THF)]$, **3**.

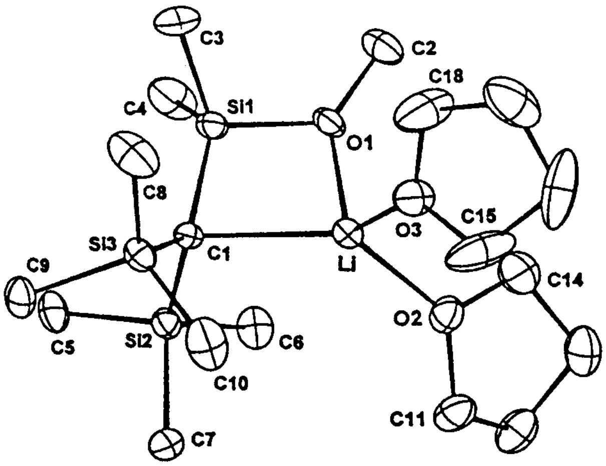

Figure 3. Molecular structure of $[Li\{C(SiMe_2OMe)(SiMe_3)_2\}(THF)_2]$, **1**.

Table 2. Selected Bond Lengths (Å) and Angles (deg) in 3

Al−C1	1.956(12)	C1−Si1	1.888(11)
Al−Cl1	2.149(5)	C1−Si2	1.903(11)
Al−Cl2	2.155(5)	C1−Si3	1.902(12)
Al−O2	1.878(9)	Si1−O1	1.664(9)
		Me−Si (mean)	1.883(6)
C1−Al−Cl1	118.0(4)	Al−C1−Si3	111.2(5)
C1−Al−Cl2	113.3(4)	Si1−C1−Si2	109.8(6)
O2−Al−Cl1	100.5(3)	Si1−C1−Si3	109.4(6)
Cl1−Al−Cl2	106.6(2)	Si2−C1−Si3	111.3(6)
Al−C1−Si1	106.2(6)	Si1−O1−C2	122.1(8)
Al−C1−Si2	108.8(6)	Me−Si−Me (mean)	105.8(4)

We take the opportunity to present the geometrical data for the lithium compound **1,** the structure of which is shown in Figure 3. We reported previously that **1** had this structure,[2] but the quality of the data was not good enough for a detailed account. Better crystals obtained during the present work yielded the results given in Table 3. Features worthy of brief comment are as follows: (a) The four-membered ring is nearly planar (sum of angles 358.7°) and there is a planar geometry at the ring-oxygen. The angles at Si1, C1, Li, and O1 are 103.4(2)°, 80.5(3)°, 79.7(4)°, and 95.1(3)°, respectively. (b) As in **2**, there is no significant difference between the lengths of the Li−O bonds to the OMe group and the THF ligands. (c) The Li−C bond is not significantly different in length from the electron-deficient bond in $[\{LiC(SiMe_3)_3\}_2]$,[15] suggesting that the coordination of the OMe group is accompanied by significant weakening of the Li−C bond compared with those in $Li\{C(SiMe_3)_{3-x}(SiMe_2Ph)_x\}(OEt_2)$ with $x = 1$[16a] or 3.[16b] (d) The Si−O bond, 1.684(4) Å, may be a little shorter than those in **2,** 1.699(4) Å, and MgR^*_2, 1.705(6) Å, and when account is taken of the experimental uncertainty cannot even be regarded as significantly longer than those in **3**, 1.664(9) Å, or ZnR^*_2, in neither of which is the oxygen coordinated to the metal. (e) As is usual for a $C(SiMe_3)_3$ or related ligand attached to an alkali metal, the C1−Si bonds, mean length 1.806(4) Å (that to the endocyclic Si may be a little shorter than the other two), are very short and the Si−C1−Si angles (116.1°, 116.2°, and 122.9°) large, implying a high degree of ionic character in the bond. (Compare the corresponding mean bond lengths and angles of 1.800(3) Å and 119.8(5)°, respectively, in the fully ionic species $[Li(THF)_4][(C(SiMe_2C_6H_4Me\text{-}o)_3$.[17]). It is noteworthy that two Me groups of each Me_3Si lie in toward the center of the molecule, as they do in as in the anions $[M\{C(SiMe_3)_3\}_2]^-$,[1b] whereas in neutral $M\{C(SiMe_3)_3\}_2$ compounds only one does so, and much further in than do those in **1**.[1a] This conformation in the case of **1** avoids the contacts between Me substituents in the Me_3Si groups and those on the ring that give rise to the small $Me_3Si−C−SiMe_3$ angle when the arrangement with a single inward-pointing Me from each Me_3Si group is adopted.[1a,18]

Table 3. Selected Bond Lengths (Å) and Angles (deg) in 1

Li−C1	2.304(11)	O1−C2	1.422(7)
Li−O1	1.933(11)	C1−Si1	1.792(6)
Li−O2	1.949(11)	C1−Si2	1.809(6)
Li−O3	1.970(11)	C1−Si3	1.818(6)
O1−Si1	1.684(4)	Me−Si (mean)	1.880(5)
Si1−C1−Li	80.5(3)	O1−Li−O3	105.8(5)
Si1−C1−Si2	116.1(3)	O2−Li−O3	98.3(5)
Si1−C1−Si3	122.9(3)	Li−O1−Si1	95.1(3)
Si2−C1−Si3	116.2(3)	Li−O1−C2	139.8(5)
C1−Li−O1	79.7(4)	C2−O1−Si1	124.5(4)
C1−Li−O2	129.6(5)	C3−Si1−C4	102.8(4)
		Me−Si−Me (mean)	103.9(5)

Experimental Section

Air and moisture were excluded as rigorously as possible by use of Schlenk techniques, flame-dried glassware, and argon as blanket gas. Solvents were dried by standard procedures and distilled immediately before use.

Spectra. Unless otherwise stated the NMR spectra were determined for solutions in toluene-d_8. The frequencies (MHz) used for the various nuclei were ^1H, 300.1; ^7Li, 97.2; ^{13}C, 125.8; ^{27}Al, 65.2; ^{29}Si, 99.4; and ^{119}Sn, 186.6. Chemical shifts are relative to $SiMe_4$ for H, C, and Si, external aqueous $LiNO_3$ for Li, and $[Al(OH_2)_6]^{3+}$ for Al. Enhancement by polarization transfer (INEPT) was used to reveal the ^{29}Si signals and those

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Table 4. Summary of Crystallographic Data for 1−3

	1	2	3
empirical formula	$C_{18}H_{43}LiO_3Si_3$	$C_{14}H_{38}AlLiO_2Si_3$	$C_{14}H_{35}AlCl_2O_2Si_3$
fw	398.7	356.6	417.6
temp (K)	173(2)	173(2)	173(2)
cryst size (mm)	0.4 × 0.3 × 0.1	0.5 × 0.5 × 0.5	0.20 × 0.05 × 0.05
cryst syst	orthorhombic	monoclinic	orthorhombic
space group	$P2_12_12_1$ (No. 19)	$P2_1/n$ (No. 14)	*Pbca* (No. 61)
a (Å)	16.518(5)	9.072(2)	12.272(4)
b (Å)	9.614(4)	22.655(11)	27.236(12)
c (Å)	15.776(6)	11.122(6)	13.176(7)
β (deg)	90	95.55(3)	90
Z	4	4	8
V (Å^3)	2505(2)	2275(2)	4404(3)
d_c (Mg m^{-3})	1.06	1.04	1.26
F(000)	880	784	1792
μ (mm^{-1})	0.20	0.25	0.50
θ range (deg)	2 to 25	2 to 25	2 to 25
index range	$0 \leq h \leq 19$	$0 \leq h \leq 10$	$0 \leq h \leq 14$
	$0 \leq k \leq 11$	$0 \leq k \leq 26$	$0 \leq k \leq 32$
	$0 \leq l \leq 18$	$-13 \leq l \leq 13$	$0 \leq l \leq 15$
no. of rflns collected	2523	4262	3874
no. of unique reflns	2523	4004, $R_{int} = 0.060$	3874
no. of rflns with $I > 2(\sigma)I$	1771	3130	1848
R1; wR2 ($I > 2\sigma(I)$)	0.060; 0.132	0.102; 0.263	0.111; 0.268
R1; wR2 (all data)	0.099; 0.154	0.127; 0.376	0.224; 0.384
no. of data/restraints/parameters	2523/0/226	3939/0/199	3864/0/199
GOF on F^2	0.999	1.119	0.981
max shift/error	0.001	0.001	0.001
largest diff peak/hole (e Å^{-3})	0.33 and −0.26	1.42 and −0.80	1.06 and −0.84
abs cor T_{max}; T_{min}	1.00; 0.92	not applied	not applied

from quaternary carbon in the ^{13}C spectra. Mass spectra were obtained by EI at 70 eV; m/z values refer to ^{28}Si and ^{35}Cl.

Compound 1. This was made as previously described[2] except that recrystallization was from light petroleum (bp 40−60 °C) at −30 °C. ^{1}H NMR: δ 0.43 (s, 18H, $SiMe_3$), 0.45 (s, 6H, $SiMe_2$), 1.28 (THF), 3.11 (s, 3H, OMe), 3.36 (THF). ^{13}C NMR: δ 3.3 ($SiMe_2$), 7.1 (CSi_3), 7.9 ($SiMe_3$), 25.3 (THF), 49.7 (OMe), 68.1 (THF). ^{29}Si NMR: δ −7.9 ($SiMe_3$), 19.4 ($SiMe_2$) (this latter value was previously[2] given in error as 0.7).

Compound 3. A solution of **1** (0.62 g, 1.60 mmol)[2] in THF (15 cm^3) was added dropwise to a solution of $AlCl_3$ (0.30 g, 2.3 mmol) in THF (15 cm^3) at room temperature, and the mixture was stirred overnight. The THF was removed under vacuum and the residue extracted with light petroleum (bp 40−60 °C, 30 cm^3). The extract was filtered and the filtrate concentrated to 10 cm^3, then kept at 5 °C to deposit crystals of **3**, yield 0.40 g, 62%, mp 82−84 °C. ^{1}H NMR: δ 0.19 (s, 6H, $SiMe_2$), 0.25 (s, 18H, $SiMe_3$), 1.10 (THF), 2.89 (s, 3H, OMe), 3.78 (THF). ^{13}C NMR: δ 2.3 ($SiMe_2$), 5.6 ($SiMe_3$), 24.9 (THF), 50.3 (OMe), 71.8 (THF); the signal from CSi_3 was not observed. ^{27}Al NMR: δ 115 ($\Delta\nu_{1/2}$ 2500 Hz). ^{29}Si NMR: δ −3.8 ($SiMe_3$), 15.8 ($SiMe_2$). MS: m/z 329 (65%, M − THF − Me), 233 (90, $CH(SiMe_3)_2(SiMe_2OMe)$ − Me), 217 (100, $Me_2Si{=}C(SiMe_2OMe)SiMe_3$ (or isomer) − Me), 187 (15), 129 (50, $Me_2Si{=}CHSiMe_3$ − Me). A satisfactory analysis was not obtained, but the identity of the product was confirmed by an X-ray diffraction study.

Compound 2. A solution of **1** (0.52 g, 1.30 mmol) in THF (20 cm^3) was added dropwise to a solution of $LiAlH_4$ (0.10 g, 2.6 mmol) in THF (15 cm^3) at room temperature, and the mixture was stirred overnight. Workup as described above gave crystals of **2**, yield 0.30 g, 60%, mp 64−65 °C. Anal. Calcd for $C_{14}H_{38}AlLiO_2Si_3$: C, 47.2; H, 10.7. Found: C, 47.7; H, 10.5. ^{1}H NMR: δ 0.32 (s, 6H, $SiMe_2$), 0.37 (s, 18H, $SiMe_3$), 1.38 (THF), 3.22 (s, 3H, OMe), 3.58 (THF). ^{1}H{^{27}Al} NMR δ 3.19 (AlH). ^{7}Li NMR: δ −0.11. ^{13}C NMR: δ 2.3 ($SiMe_2$), 4.2 ($SiMe_3$), 25.4 (THF), 50.3 (OMe), 68.6 (THF); the signal from CSi_3 was not observed. ^{27}Al NMR: δ 117 ($\Delta\nu_{1/2}$ 1000 Hz). ^{29}Si NMR: δ −4.1 ($SiMe_3$), 22.8 ($SiMe_2$). The identity of the product was confirmed by an X-ray diffraction study.

Crystal Structure Determinations. Data were collected at 173(2) K on an Enraf-Nonius CAD4 diffractometer using monochromated Mo Kα radiation (0.710 73 Å) and corrected for Lorentz and polarization effects; details are given in Table 4. The structures were determined by direct methods, with SHELXS 86 and SHELXL 93 programs used for structure solution and refinement on F^2 using all reflections. Non-hydrogen atoms were anisotropic. Except for those attached to Al in **2**, which were freely refined, H atoms were refined in riding mode with $U_{iso}(H) = 1.2U_{eq}(C)$ or, for Me groups, $1.5U_{eq}(C)$. In the ORTEP plots the thermal ellipsoids are shown at the 50% probability level.

Acknowledgment. We thank the EPSRC for financial support and the Turkish Government for the award of a postgraduate scholarship to S.E.S.

Supporting Information Available: Tables of atom coordinates, bond lengths and angles, and anisotropic displacement parameters for **1**−**3** (9 pages). Ordering information is given on any current masthead page.

OM9804024