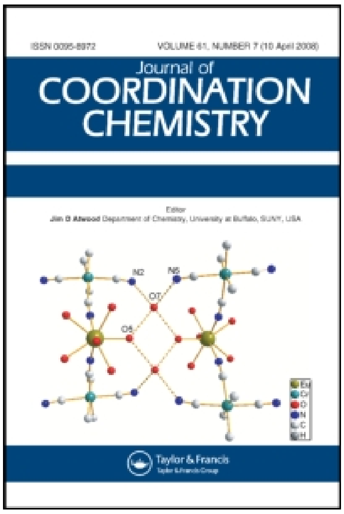

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ISOLATION AND X-RAY CRYSTAL STRUCTURES OF $\{Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ AND $[(Me_3Si)_2AsLi(THF)_2]_2$

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The lithium arsenides $\{Li_2[\mu_2\text{-}As(SiMe_3)_2]$ $[\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ (I) (THF = tetrahydrofuran) and $[(Me_3Si)_2AsLi(THF)_2]_2$ (II) were isolated and their solid-state structures determined by X-ray methods. Compound I crystallizes in the monoclinic system, space group $P2_1/c$, with a = 10.147(1)Å, b = 19.665(2)Å, c = 16.768(2)Å, β = 107.78(1)° and Z = 2. The core structure consists of an $[LiAs]_4$ ladder-like framework with four antiparallel adjacent As-Li rungs. The two lithium atoms of the central ring each bridge three arsenic centers while the two lithium atoms belonging solely to the outer rings each span two arsenic atoms and are coordinated to one THF molecule. Monoclinic crystals of compound II belong to space group $C2/c$, with a = 17.877(4) Å, b = 14.077(3) Å, c = 19.006(4) Å, β = 109.16(2)° and Z = 4. The structure consists of a centrosymmetric dimer containing a four-membered As-Li-As-Li ring wherein each lithium atom is coordinated to two molecules of THF.

KEYWORDS: arsenide, lithium, crystal structure

INTRODUCTION

Lithium compounds are among the most widely used organometallic reagents, yet their structures and bonding are far from trivial. The high degree of ionic character of lithium dominates its bonding and though the octet rule is not adequate for interpreting this bonding, the coordination geometry about lithium is often tetrahedral.[1] The degree of oligomerization or aggregation generally correlates with the reactivity of these reagents and the degree of association in solution is often reflected in the solid-state structure.[2] Predicting the structure of lithium compounds is difficult and appears to depend on the nature and size of ligands. The presence of electron-rich species can alter the aggregation of these complexes dramatically.

The isolation of $\{Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ (I) (THF = tetrahydrofuran) prompted attempts to crystallize two similar lithium arsenides, $[(Me_3Si)_2AsLi(THF)_2]_2$ (II) and solvent free $LiAs(SiMe_3)_2$. Although the crystal structures of the phosphorus analogues of these compounds have been determined,[3,4] the arsenides could not be assumed to be isostructural. The structures of $[(Et_2O)_2LiAsPh_2]_2$,[5] $[(Et_2O)LiPPh_2]_\infty$[6] and $[(THF)_2LiPPh_2]_\infty$[6] have

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been reported and are quite different. In addition, the solid-state structures of several lithium arsenides including $\{(THF)Li[As({}^tBu)As({}^tBu)_2]\}_2$[7]and $[(1,4\text{-dioxane})_3LiAsPH_2]$[5] have been established by X-ray analyses but the related phosphorus analogues have yet to be prepared. Herein, the isolation and solid-state structures of I and II and the attempted isolation of crystalline $LiAs(SiMe_3)_2$ are presented.

EXPERIMENTAL

General Considerations

All manipulations were performed by Schlenk techniques or in a Vacuum/ Atmospheres HE-493 Dri-Lab under argon. THF, benzene and benzene-d_6 were distilled from sodium/benzophenone under dry nitrogen. Pentane was distilled from $LiAlH_4$ under dry nitrogen. Ether-free $LiAs(SiMe_3)_2$ was prepared by heating the THF solvated compound[8] at 70°C for 48 h at 10^{-3} torr. ^1H and $^{13}C\{^1H\}$ NMR spectra were obtained on a Varian XL-300 (300 and 75.4 MHz, respectively) spectrometer using 5 mm tubes. ^1H and ^{13}C spectra were referenced to TMS using the residual protons or carbons of deuterated benzene at δ 7.15 or 128 ppm. NMR tubes were flame sealed under vacuum. All melting points (uncorrected) were obtained with a Thomas-Hoover Uni-melt apparatus and capillaries were flame sealed under argon. Crystals used in the X-ray analyses were flame sealed in 0.7 mm glass capillaries. Elemental analyses were performed by E + R Microanalytical Laboratory, Inc., Corona, New York.

$\{Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ *(I)*

Three mole equivalents of ether-free $LiAs(SiMe_3)_2$(0.300 g, 1.31 mmol) were added to a stirring benzene suspension of $InCl_3$ (0.969 g, 0.438 mmol). Several drops of THF were added to the vial in which the arsenide was weighed and the resulting solution was added to the $InCl_3$ suspension in order to assure a quantitative transfer. After addition of the THF portion, the solution immediately became dark as a brown/black precipitate formed. The solution was cannula filtered after it had been stirred for 24 h at room temperature. The volatiles were removed from the orange filtrate in *vacuo*, leaving an orange viscous oil. A black powder that remained in the original flask was washed with pentane which was then added to the filtrate. The orange oil was dissolved in pentane and the solution was cooled to − 15°C. After one week, colorless crystals of I suitable for an X-ray diffraction analysis had grown from this solution (0.007 g). *Anal.* Found for the black powder: As, 25.86; In, 40.13. ^1H NMR of crystals: δ 0.63 (s, $SiMe_3$), 1.18 (m, CH_2 of THF), 3.65 (bm, OCH_2 of THF). No signal was observed in the $^{13}C\{^1H\}$ NMR spectrum due to the small sample size.

$[(Me_3Si)_2AsLi(THF)_2]_2$ *(II)*

To a pentane suspension of ether-free $LiAs(SiMe_3)_2$ was added enough THF to dissolve all of the solid. Large colorless crystals of II were grown by slow evaporatation of the solution over a several day period. Upon decanting the supernatant, the crystals immediately began to turn white on the surface and they ultimately flaked apart to a powder. *This powder immediately ignites upon introduction to air and may be explosive in less than 0.1 g quantities.* Crystal fragments of an appropriate size for X-ray analysis were sealed inside glass capillaries the interior walls of which had been previously moistened with a pentane/THF solution. mp: The crystals became opaque white from 54–60°C and showed no further change up to 290°C. *Anal.* Calcd. for $C_{24}H_{68}As_2Li_2O_4Si_4$: C, 45.15; H, 9.2; As, 20.12; Li, 1.86. Found: C, 45.02; H, 9.21; As, 20.41; Li, 1.67. ^1H NMR: δ 0.62 (s, $SiMe_3$), 1.42 (m, CH_2 of THF), 3.72 (m, OCH_2 of THF). ^{13}C NMR: δ 8.13 (s, $SiMe_3$), 25.42 (s, CH_2 of THF), 68.61 (s, OCH_2 of THF).

Crystallographic Data Collection and Structure Determination

An Enraf-Nonius CAD-4 diffractometer (Cu-Kα radiation; graphite monochromator) was used for all measurements on compounds I and II. Crystallographic data and summaries of the analyses are provided in Table 1. Refined unit-cell parameters

Table 1 Summary of crystallographic data and measurements for $\{Li_2[\mu\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ (I) and $[(Me_3Si)_2AsLi(THF)_2]_2$ (II).

	I	II
Molecular formula	$C_{32}H_{88}As_4Li_4O_2Si_8$	$C_{28}H_{68}As_2Li_2O_4Si_4$
Formula weight	1057.20	744.92
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$C2/c$
a(Å)	10.147(1)	17.877(4)
b(Å)	19.665(2)	14.077(3)
c(Å)	16.768(2)	19.006(4)
β(°)	107.78(1)	109.16(2)
V(Å^3)	3186(1)	4518(4)
Z	2	4
D_{calcd} (g cm^{-3})	1.102	1.095
Radiation (wavelength)	Cu-Kα(1.5418 Å)	Cu-Kα(1.5418Å)
μ(cm^{-1})	41.0	30.6
Temperature (°C)	25	25
Crystal dimensions (mm)	0.18 × 0.30 × 0.40	0.20 × 0.30 × 0.80
T_{max}:$T_{min.}$ (relative)	1.00:0.60	1.00:0.72
Scan type	ω-2θ	ω-2θ
Scan width (°)	0.80 + 0.14tanθ	0.90 + 0.14tanθ
θ max. (°)	55	75
Intensity control refls.	342, $12\bar{2}$, $22\bar{3}$, 142	171, $22\bar{4}$, 331, $42\bar{2}$
Variation; repeat time (h)	<2%; 2	<2%; 2
No. of refls. ($+h$, $+k$, $\pm l$) recorded	4071	4809
No. of non-equiv. refls. recorded	3906	4658
R_{merge} (on I)	0.035	0.039
No. of refls. retained [$I > 3.0\sigma(I0)$])	1832	2463
No. of parameters refined	226	182
Extinction correction		$8(2) \times 10^{-7}$
R (R_w)[a]	0.053 (0.072)	0.060 (0.084)
Goodness-of-fit[b]	1.47	1.68
Max. shift: esd in final least-squares cycle	0.03	0.02
Final Δρ (e/Å^3) max.; min.	0.36; −0.28	0.53; −0.66

[a] $R = \Sigma\|F_0|-|F_c\|/\Sigma|F_0|$; $R_w = [\Sigma w(|F_0|-|F_c|)^2\Sigma w|F_0|^2]^{1/2}$; $\Sigma w\Delta^2[w = 1/\sigma^2(|F_0|)$, $\Delta = (|F_0|-|Fc|)]$ was minimized. [b] Goodness-of-fit = $[\Sigma w\Delta^2/(N_{observations}\text{-}N_{parameters})]^{1/2}$.

were derived in each case from the diffractometer setting angles for 25 reflections (30°<θ<35°for I, 36°<θ<40°for II) widely separated in reciprocal space. Intensity data were corrected for the usual Lorentz and polarization effects. Empirical absorption corrections, derived from the ϕ-dependency of the intensities of several reflections with ψ *ca.* 90°, were also applied. The space group $P2_1/c$ for I was established uniquely by the Laue symmetry and systematic absences (0k0 when $k \neq 2n$, h0l when $l \neq 2n$). For II, the systematic absences (hk0 when $h + k \neq 2n$, h0l when $l \neq 2n$) are compatible with space groups *Cc* and *C2/c*; the latter was assumed at the outset and shown to be correct by the structure solution and refinement.

The crystal structure of I was solved by direct methods (MULTAN11/82). Approximate coordinates for the As and Si atoms were derived from an E-map. The other non-hydrogen atoms were located in a series of weighted F_0 and difference Fourier syntheses phased successively by an increasing number of atoms. For II, coordinates for the isomorphous phosphorus analogue[3] were used as input for the initial structure-factor calculation. Non-hydrogen atom positional and thermal parameters for I and II (at first isotropic and then anisotropic) were adjusted by means of several rounds of full-matrix least-squares calculations. Hydrogen atoms were incorporated at their calculated positions during the later least-squares iterations; an extinction correction was also included as a variable for II. Parameter refinements converged at $R = 0.053$ ($R_w = 0.072$) for I and $R = 0.060$ ($R_w = 0.084$) for II over 1832 and 2463 reflections, respectively, with $I>3.0\sigma(I)$. Final difference Fourier syntheses contained no unusual features.

Crystallographic calculations were performed on PDP11/44 and Micro VAX computers by use of the Enraf-Nonius Structure Determination Package (SDP). For structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from ref. 9.

RESULTS AND DISCUSSION

On reaction of ether-free $LiAs(SiMe_3)_2$ with $InCl_3$ in a 3:1 mole ratio in a benzene/THF solution, the anticipated products were a trisarsinoindane and lithium chloride. The formation of a black precipitate indicated that this reaction did not proceed as expected. A partial elemental analysis revealed that the composition of this material was 25.86% As and 40.13% In; *i.e.*, a 1:1.01 mole ratio. Since all of the material was used for elemental analysis, its identity as InAs, a mixture containing elemental indium and arsenic, or an organometallic compound could not be established. In addition to the black precipitate, a small number of colorless crystals were isolated. ^1H NMR spectroscopy and a single-crystal X-ray analysis identified these crystals as I. An ORTEP diagram showing the solid-state structure and atom numbering scheme is presented in Figure 1. Several methyl carbon atoms are extremely anisotropic; the magnitude and orientations of their thermal ellipsoids suggest crystal packing disorder. Table 2 lists the non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters. Selected interatomic distances and angles are given in Table 3.

Crystals of compound I are not isomorphous with those of the phosphorus analogue $\{Li_2[\mu_2\text{-}P(SiMe_3)_2][\mu_3\text{-}P(SiMe_3)_2](THF)\}_2$ (III). These compounds are nevertheless isostructural and similar to $\{Li_2[\mu_2\text{-}P(^tBu)_2][\mu_3\text{-}P(^tBu)_2](THF)\}_2$(IV).[10] All three compounds lie on crystallographic inversion centers and contain a core

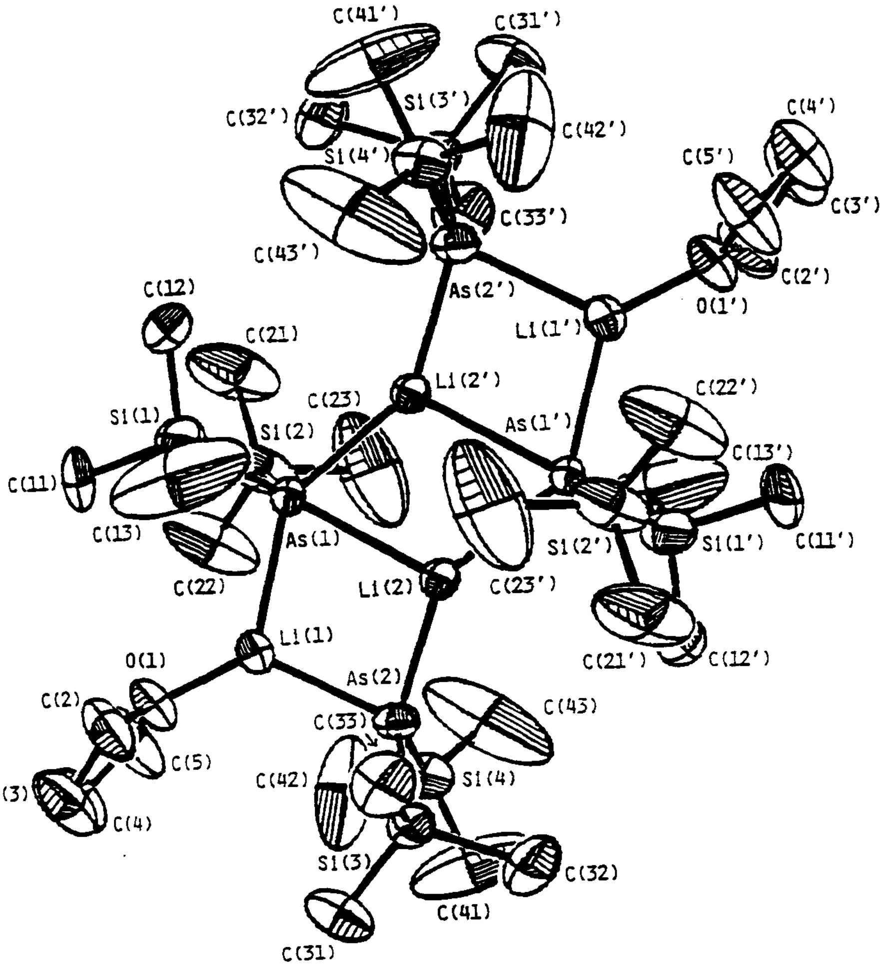

Figure 1 ORTEP diagram showing the solid-state conformation of $\{Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ (I), with thermal ellipsoids at the 30% probability level. Hydrogen atoms have been omitted. Primed atoms are related to the unprimed atoms by a crystallographic center of symmetry.

ladder structure consisting of four antiparallel E-Li (E = As, P) steps. Compound I is not only the first structurally characterized lithium arsenide exhibiting this type of skeletal structure but also it is the first containing five-coordinate arsenic. As-Li bond distances in I vary significantly with the mean of 2.63 Å for those associated with the five-coordinate As [As(1)-Li(1) = 2.63(2) Å, As(1)-Li(2) = 2.69(2) Å, As(1)-Li(2') = 2.57(2) Å] being longer than that of 2.53 Å for those involving the four-coordinate As [As(2)-Li(1) = 2.51(2) Å, As(2)-Li(2) = 2.54(2) Å]. The difference of 0.10 Å between these means is similar to those of 0.12 Å and 0.13 Å for the corresponding means in III and IV, respectively. The central ring is required crystallographically to be exactly planar and, with endocyclic torsion angles of only ±8° in the Li(1)-As(1)-Li(2)-As(2) rings, the Li_4As_4 framework is overall fairly flat

Table 2 Non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters[a] for $\{Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$(I), with estimated standard deviations in parentheses.

Atom	*x*	*y*	*z*	B_{eq}(Å^2)
As(1)	0.1219(1)	0.09049(5)	0.03215(6)	6.91(3)
As(2)	0.1771(1)	-0.02175(7)	0.24577(6)	8.33(3)
Si(1)	0.2632(5)	0.1359(3)	-0.0374(3)	18.7(2)
Si(2)	-0.0089(5)	0.1766(2)	0.0595(3)	17.7(2)
Si(3)	0.3494(4)	-0.0993(2)	0.2973(2)	9.3(1)
Si(4)	0.0753(4)	-0.0061(2)	0.3494(2)	10.7(1)
C(11)	0.3494(24)	0.2157(12)	0.0278(17)	48(1)
C(12)	0.1815(18)	0.1730(8)	-0.1392(8)	18(1)
C(13)	0.3890(21)	0.0817(22)	-0.0375(15)	56(2)
C(21)	-0.0842(25)	0.2302(11)	-0.0265(14)	31(1)
C(22)	0.0597(21)	0.2141(11)	0.1578(12)	28(1)
C(23)	-0.1793(24)	0.1344(19)	0.0694(24)	42(2)
C(31)	0.4829(16)	-0.0739(11)	0.3958(11)	18.4(7)
C(32)	0.2794(15)	-0.1856(7)	0.3151(9)	14.9(5)
C(33)	0.4365(15)	-0.1134(9)	0.2170(9)	15.6(5)
C(41)	0.1154(23)	-0.0605(16)	0.4330(10)	38(1)
C(42)	0.1197(24)	0.0683(12)	0.4035(11)	39(1)
C(43)	-0.0944(23)	-0.0058(24)	0.3131(15)	46(2)
O(1)	0.4232(8)	0.1308(5)	0.2514(5)	12.1(3)
C(2)	0.5521(15)	0.1236(11)	0.2517(10)	19(1)
C(3)	0.6421(16)	0.1450(11)	0.3362(13)	22(1)
C(4)	0.5541(17)	0.1777(11)	0.3726(11)	19(1)
C(5)	0.4221(17)	0.1655(10)	0.3185(11)	22(1)
Li(1)	0.2711(20)	0.0796(11)	0.1901(12)	9.3(6)
Li(2)	0.0520(18)	-0.0274(8)	0.0895(10)	7.4(4)

[a] $B_{eq} = (4/3)(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$

(max. deviation = 0.107 Å, mean deviation = 0.090 Å from the least-squares plane). Endocyclic bond angles at the Li atoms [71.1(5)°, 72.7(6)°, 77.2(6)°] are consistently much larger than those at the As centers [108.9(6)°, 103.2(5)°, 105.8(7)°], and consequently the four-membered rings are skewed significantly from a square geometry. In accord with the situation in III and IV, the bonding arrangement at the three-coordinate Li atom in I is planar. The THF oxygen atom lies close to the least-squares plane through the $[LiAs]_4$ core (Δ = 0.3 Å), and the Li-O distance of 1.87(2) Å in I is similar to those of 1.89(3) Å in III and 1.92(1) Å in IV.

The negligible solubility of $LiAs(SiMe_3)_2$ in non-etherial solvents and its decomposition upon sublimation prevented the successful crystallization of this material. However, crystals of II could be grown from a pentane/THF solution of ether-free $LiAs(SiMe_3)_2$. These crystals readily lose THF to become a white powder. Sealing crystals inside thin-walled glass capillaries previously moistened internally with a pentane/THF solution stabilized the crystals for X-ray data collection. An ORTEP diagram showing the solid-state structure of II is presented in Figure 2. Table 4 lists the non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters while selected bond distances and angles are presented in Table 5.

Dimer II is isomorphous with its phosphorus analogue $[Me_3Si)_2PLi(THF)_2]_2$. These compounds lie on crystallographic centers of symmetry and consequently their $(E\text{-}Li)_2$ rings (E = As, P) are strictly planar. The coordination geometry at Li and pnicogen is distorted tetrahedral. The only other dimeric lithium arsenides for

Table 3 Selected bond distances (Å) and angles (°) for {$Li_2[\mu_2\text{-}As(SiMe_3)_2][\mu_3\text{-}As(SiMe_3)_2](THF)\}_2$ (I), with estimated standard deviations in parentheses.

Bond distances			
As(1)-Si(1)	2.288(6)	As(2)-Si(3)	2.282(4)
As(1)-Si(2)	2.283(5)	As(2)-Si(4)	2.297(4)
As(1)-Li(1)	2.63(2)	As(2)-Li(1)	2.51(2)
As(1)-Li(2)	2.69(2)	As(2)-Li(2)	2.54(2)
As(1)-Li(2′)	2.57(2)	Li(1)-O(1)	1.87(2)
Bond angles			
Si(1)-As(1)-Si(2)	107.8(2)	Si(2)-As(1)-Li(2)	109.6(4)
Si(1)-As(1)-Li(1)	106.7(5)	Si(2)-As(1)-Li(2′)	102.5(4)
Si(1)-As(1)-Li(2)	142.6(4)	Li(1)-As(1)-Li(2)	72.7(6)
Si(1)-As(1)-Li(2′)	99.7(5)	Li(1)-As(1)-Li(2′)	143.6(6)
Si(2)-As(1)-Li(1)	92.8(5)	Li(2)-As(1)-Li(2′)	71.1(5)
Si(3)-As(2)-Si(4)	105.8(1)	Si(4)-As(2)-Li(1)	119.0(5)
Si(3)-As(2)-Li(1)	109.7(5)	Si(4)-As(2)-Li(2)	126.0(5)
Si(3)-As(2)-Li(2)	116.4(4)	Li(1)-As(2)-Li(2)	77.2(6)
As(1)-Li(1)-As(2)	105.8(7)	As(1)-Li(2)-As(2)	103.2(5)
As(1)-Li(1)-O(1)	130(1)	As(1)-Li(2)-As(1′)	108.9(6)
As(2)-Li(1)-O(1)	125(1)	As(2)-Li(2)-As(1′)	147.6(8)

which the solid-state structures have been determined are $[(Et_2O)_2LiAsPh_2]_2$ (V)[5] and $(DME)LiAs(SiMe_3)_2]_2$(DME = 1,2-dimethoxyethane) (VI),[11] both of which contain four-coordinate Li, and $\{(THF)Li[As(^tBu)As(^tBu)_2]\}_2$[7] (VII) wherein the Li is three-coordinate. Variations in intramolecular steric and electronic effects of the ligands in II, V, and VI are reflected in the ring geometries. The mean of the

Table 4 Non-hydrogen atom fractional coordinates and equivalent isotropic thermal parameters[a] for $[(Me_3Si)_2AsLi(THF)_2]_2$ (II), with estimated standard deviations in parentheses.

Atom	*x*	*y*	*z*	*B*eq($Å_2$)
As	0.27092(3)	0.10831(4)	0.01214(3)	6.94(1)
Si(1)	0.3390(1)	0.0273(2)	0.1181(1)	10.20(6)
Si(2)	0.2426(1)	-0.0072(2)	-0.0772(1)	9.90(5)
O(1)	0.1346(3)	0.2331(3)	0.1076(2)	11.3(1)
O(2)	0.0546(3)	0.2029(4)	-0.0562(3)	11.4(2)
C(1)	0.4337(6)	-0.0251(8)	0.1164(6)	16.0(4)
C(2)	0.3689(8)	0.1089(11)	0.1984(6)	19.4(5)
C(3)	0.2788(8)	-0.0650(8)	0.1431(6)	18.5(4)
C(4)	0.1755(7)	-0.1058(7)	-0.0651(6)	17.0(4)
C(5)	0.1901(7)	0.0536(9)	-0.1666(4)	15.8(4)
C(6)	0.3290(7)	-0.0678(7)	-0.0907(6)	18.8(3)
C(7)	0.1423(8)	0.1628(9)	0.1590(6)	23.6(4)
C(8)	0.0924(7)	0.1787(8)	0.2002(5)	21.9(3)
C(9)	0.0612(6)	0.2605(8)	0.1806(5)	19.3(4)
C(10)	0.0939(6)	0.2999(7)	0.1314(5)	21.9(3)
C(11)	-0.0084(7)	0.2648(7)	-0.0915(10)	18.6(6)
C(12)	-0.0724(7)	0.1963(9)	-0.1352(8)	20.9(5)
C(13)	-0.0487(9)	0.1027(9)	-0.1268(10)	27.8(8)
C(14)	0.0259(8)	0.1071(8)	-0.0660(11)	23.4(7)
Li	0.1583(6)	0.2350(7)	0.0138(6)	8.0(2)

[a] $B_{eq} = (4/3)(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$

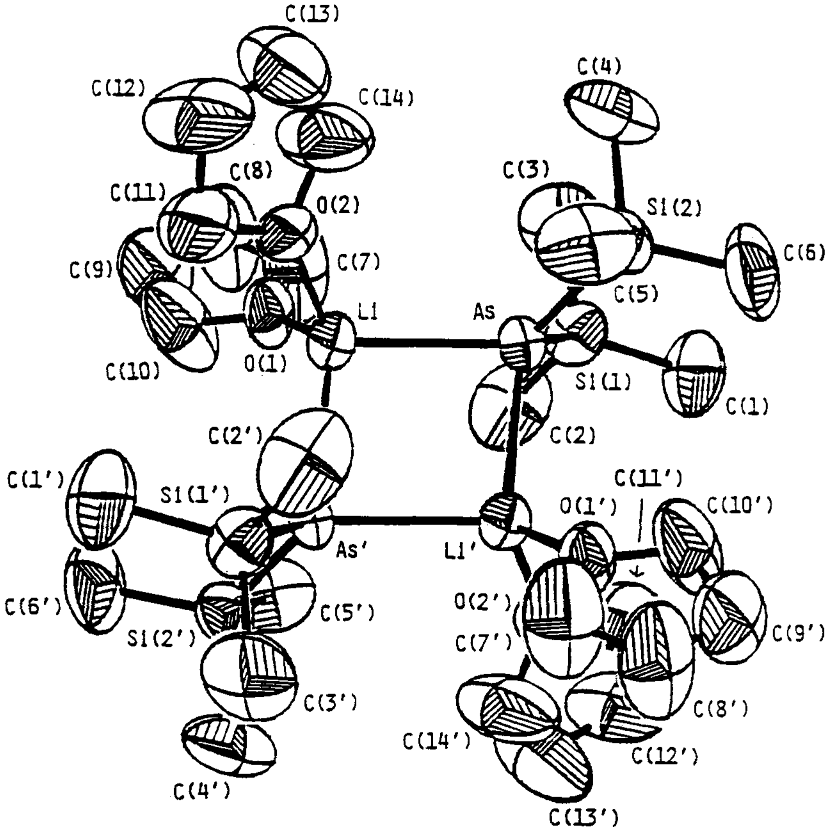

Figure 2 ORTEP diagram showing the solid-state conformation of $[(Me_3Si)_2AsLi(THF)_2]_2$ (II), with thermal ellipsoids at the 30% probability level. Hydrogen atoms have been omitted. Primed atoms are related to the unprimed atoms by a crystallographic center of symmetry.

essentially equal As-Li bond distances [2.70(1), 2.67(1) Å] at 2.69 Å in II is close to that of 2.73 Å in V but is considerably longer than the As-Li distance of 2.59(2) Å in VI. On the other hand, the As-Li-As angle at 98.2(4)° in II is essentially identical with that of 99(1)° in VI, and both are much larger than the Li-As-Li angles of 82.8(3)° and 81(1)°, respectively; thus, as in I, the rings are distorted from a regular square geometry. The presence of phenyl substituents at arsenic in V results in a much larger Li-As-Li angle [90.3(4)°] compared to those in II and VI and, with the mean Li-As-Li angle at 88.3(4)°, the ring is approximately square.

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Table 5 Selected bond distances (Å) and angles (°) for $[(Me_3Si)_2AsLi(THF)_2]_2$ (II), with estimated standard deviations in parentheses.

Bond distances			
As-Si(1)	2.287(2)	As-Li′	2.67(1)
As-Si(2)	2.285(2)	Li-O(1)	1.96(1)
As-Li	2.70(1)	Li-O(2)	1.95(1)
Bond angles			
Si(1)-As-Si(2)	102.9(1)	As-Li-As′	98.2(4)
Si(1)-As-Li	120.2(3)	As-Li-O(1)	112.7(4)
Si(1)-As-Li′	115.3(2)	As-Li-O(2)	112.7(5)
Si(2)-As-Li	119.7(2)	As′-Li-O(1)	115.8(5)
Si(2)-As-Li′	117.0(3)	As′-Li-O(2)	117.9(5)
Li-As-Li′	81.8(3)	O(1)-Li-O(2)	100.2(6)

Supplementary Data

Complete tables of anisotropic temperature factor parameters, interatomic distances and angles, hydrogen atom coordinates and isotropic thermal parameters, and a listing of observed and calculated structure amplitudes for I and II (43 pages) are available from R.L. Wells.

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