# Syntheses and structures of [Me$_2$Si{As(P$^t$Bu)$_3$}$_2$] and [(CyP)$_3$SiMe$_2$] (Cy = cyclohexyl, C$_6$H$_{11}$)

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## ARTICLE INFO

## ABSTRACT

The reaction of the anion [($^t$BuP)$_3$As]$^-$ (**1**) with Me$_2$SiCl$_2$ results in nucleophilic substitution of the Cl$^-$ anions, giving the di- and mono-substituted products [Me$_2$Si{As(P$^t$Bu)$_3$}$_2$] (**3a**) and [Me$_2$Si(Cl){As(P$^t$Bu)$_3$}] (**3b**). Analogous reactions of the pre-isolated [(CyP)$_4$As]$^-$ anion (**2**) (Cy = cyclohexyl) with Me$_2$SiCl$_2$ produced mixtures of products, from which no pure materials could be isolated. However, reaction of **2** [generated *in situ* from CyPHLi and As(NMe$_2$)$_3$] gives the heterocycle [(CyP)$_3$SiMe$_2$] (**4**). The X-ray structures of **3a** and **4** are reported.

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## 1. Introduction

Heterocyclic anions of the type [(RP)$_n$E]$^-$ (where E = P, As, Sb; $n$ = 3 or 4) are an interesting, and at present relatively little-studied, class of potential ligands for a range of metals and non-metals [1–3]. We have found that simple access to the As and Sb derivatives is offered by the direct reactions of alkali metal primary phosphides [RPHM] (M = an alkali metal) with the series of reagents [E(NMe$_2$)$_3$] (Scheme 1, step 1) [1–3]. These reactions are difficult to control in the case of the Sb heterocycles [(RP)$_n$Sb]$^-$ owing to the relative weakness of P–Sb bonds and the resulting thermal instability (Scheme 1, step 2). For example, the five-membered, heterocyclic anion [(CyP)$_4$Sb]$^-$ (Cy = cyclohexyl) decomposes at temperatures above *ca.* 0 °C into the Zintl ion [Sb$_7$]$^{3-}$ and the cyclic phosphane [CyP]$_4$ [4]. This type of main group dehydrocoupling reaction can be seen to be driven thermodynamically by the strength of the P–P single bond, which has the highest homoatomic bond energy between of any of the group 15 elements (*ca.* 201 kJ mol$^{-1}$) [5]. The phosphorus counterparts [($^t$BuP)$_4$P]$^-$ have been obtained by the *in situ* reactions of $^t$BuPCl$_2$ and PCl$_3$ with Na metal in THF (Scheme 2) [6].

A particular interest in the coordination chemistry of the [(RP)$_n$E]$^-$ anions is their potential to deliver group 15 atoms, by the reductive elimination of cyclic phosphanes. Our studies in this area have focused on the coordination behaviour and reactivity of the readily prepared [($^t$BuP)$_3$As]$^-$ anion (**1**) [7]. Disappointingly, reactions of **1** with a range of main group metals results in oxidative

coupling into the As–As bonded dimer [($^t$BuP)$_3$As]$_2$ (Scheme 3a) [8]. However, the *intact* [($^t$BuP)$_3$As] framework is transferred in reactions with transition metal half-sandwich compounds. Interestingly, while the reaction of **1** with [CpFe(CO)$_2$Cl] gives the expected addition to the Fe centre (Scheme 3b), addition of the [($^t$BuP)$_3$As] unit to the Cp ligand occurs for [CpM(CO)$_3$Cl] (M = Mo, W) (Scheme 3c) [7a,9]. The products of the latter reaction, [C$_5$H$_4${As(P$^t$Bu)$_3$}M(CO)$_3$Cl], probably result from a mechanism involving initial addition to the metal (M) followed by transfer to the Cp ligand (with formal elimination of H$^-$). In contrast, reactions of organic electrophiles (R–X; R = Me, X = I; R = PhCH$_2$, CH$_2$ = CHCH$_2$, X = Br) give the neutral triphosphanes [($^t$BuP)($^t$BuPR)$_2$], *via* an S$_N$2-type mechanism involving cleavage of the As–P bonds (Scheme 3d) [7b]. This route gives rise to a potentially broad range of new phosphane ligand frameworks.

Given the reactivity patterns described above, there is an obvious potential for the development of a general approach to neutral heterocycles of the type [(RP)$_n$ER′$_m$] using reactions of [(RP)$_n$As]$^-$ anions like **1** with group 13 or 14 organohalide electrophiles [group 13 R′EX$_2$ (E = P–Bi); group 14 R′$_2$EX$_2$; E = C–Sn; X = halogen] (Scheme 4). In the current work we have explored this potential for the first time in a study of the reactions of the anions **1** and the related five-membered anion [(CyP)$_4$As]$^-$ (**2**) with Me$_2$SiCl$_2$. We find, however, that the reaction of **1** with Me$_2$SiCl$_2$ in 3:2 stoichiometric ratio (suggested by analogy with reaction **d**, Scheme 3) does not produce the heterocyclic product [($^t$BuP)$_3$SiMe$_2$] (Scheme 4, top). Instead, *intact* transfer of the [($^t$BuP)$_3$As] unit occurs, producing a mixture of the di- and mono-substituted products [Me$_2$Si{As-(P$^t$Bu)$_3$}$_2$] (**3a**) and [Me$_2$Si(Cl){As(P$^t$Bu)$_3$}] (**3b**) (Scheme 4, bottom). Although no solid products could be obtained from the reaction of
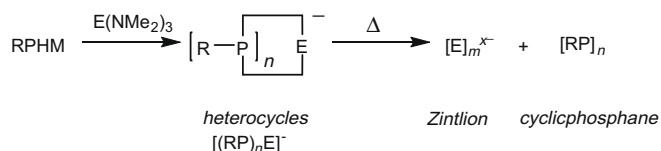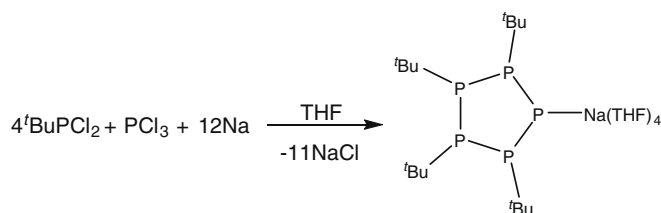
**Scheme 1.**



**Scheme 2.**

the pure anion **2** with Me$_2$SiCl$_2$, a low yield of the heterocycle [(Cy-P)$_3$SiMe$_2$] (**4**) could be formed from the addition of Me$_2$SiCl$_2$ to a 3:1 mixture of CyPHLi and As(NMe$_2$)$_3$ (Scheme 5).

## 2. Results and discussion

The previously reported complexes [{($^t$BuP)$_3$As]Li·2DABCO·THF] (**1**·2DABCO·THF) [DABCO = N{(CH$_2$)$_2$}$_3$] [2b] and [{(CyP)$_4$As}-Li·TMEDA·THF] (**2**·TMEDA·THF) [TMEDA = Me$_2$N(CH$_2$)$_2$] [3] were used as sources of the anions **1** and **2**, respectively. The 3:2 stoichiometric reaction of Me$_2$SiCl$_2$ (1.8 mmol) with the **1**·2DABCO·THF (1.2 mmol) in THF/toluene (Scheme 4, top) gives a mixture of two closely related products (**3a** and **3b**; ca. 3:2), as revealed by an in situ $^{31}$P{$^1$H} NMR study of the reaction mixture after filtration of the LiCl precipitate (see Table 1). Both products appear as a doublet and a triplet [3a,$\delta$-62.6{d,P(1,3)},$\delta$-15.7{t,P(2)} ($^1J_{P-P}$ = 159 Hz);3b,$\delta$-63.4{d,P(1,3)},$\delta$-13.5{t,P(2)}($^1J_{P-P}$ = 156 Hz)], consistent with presence of P$_3$ units in both products. The similarity of the chemical shifts and the coupling constants involved suggests that **3a** and **3b** are closely related chemically. Based on this, and the later full characterisation of the major product **3a** as the di-substituted species [Me$_2$Si{As(P$^t$Bu)$_3$}$_2$], the minor isomer (**3b**) is most likely to be the mono-substituted species [Me$_2$Si(Cl){As (P$^t$Bu)$_3$}]. This conclusion is also roughly consistent with fact that an excess of **1** was used in this initial reaction. Unfortunately, no further characterisation of **3b** (or **3a**) by mass spectroscopy was possible owing to air-sensitivity. However, even changing the reaction stoichiometry to 1:1 resulted in the formation of a mixture of **3a** and **3b**. Noticeably, although the chemical shifts of the central P atoms within the P$_3$As ring units of **3a** and **3b** [P(2)] are similar to those present in alkali metal complexes of the anion **1** ($\delta$ −15.7 and $\delta$ −13.5 in **3a** and **3b**, respectively), the terminal P atoms [P(1,3)] are significantly deshielded ($\delta$ −62.6 and −63.4 in **3a** and **3b**; cf $\delta$ −73.6 to −89.9 in alkali metal complexes of **1**) [2]. This increase in chemical shift is synonymous with a reduction in the partial charge of the [($^t$BuP)$_3$As] units in **3a** and **3b**, resulting from the formation of covalent As-Si bonds. Storage of the reaction solution at
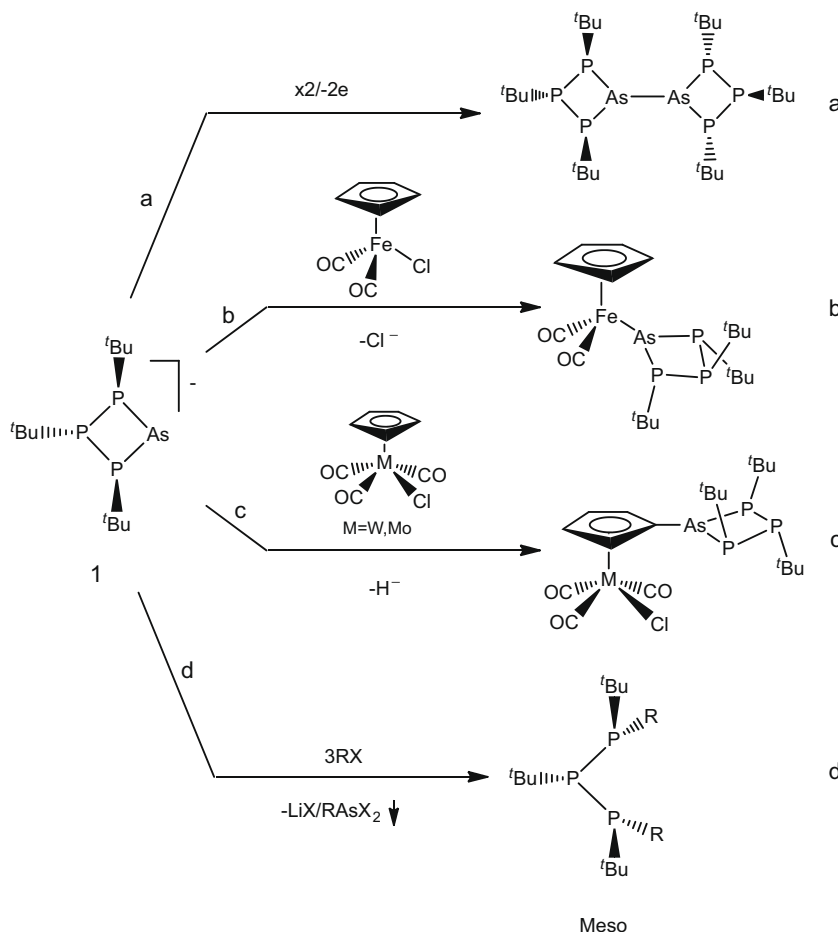


**Scheme 3.**
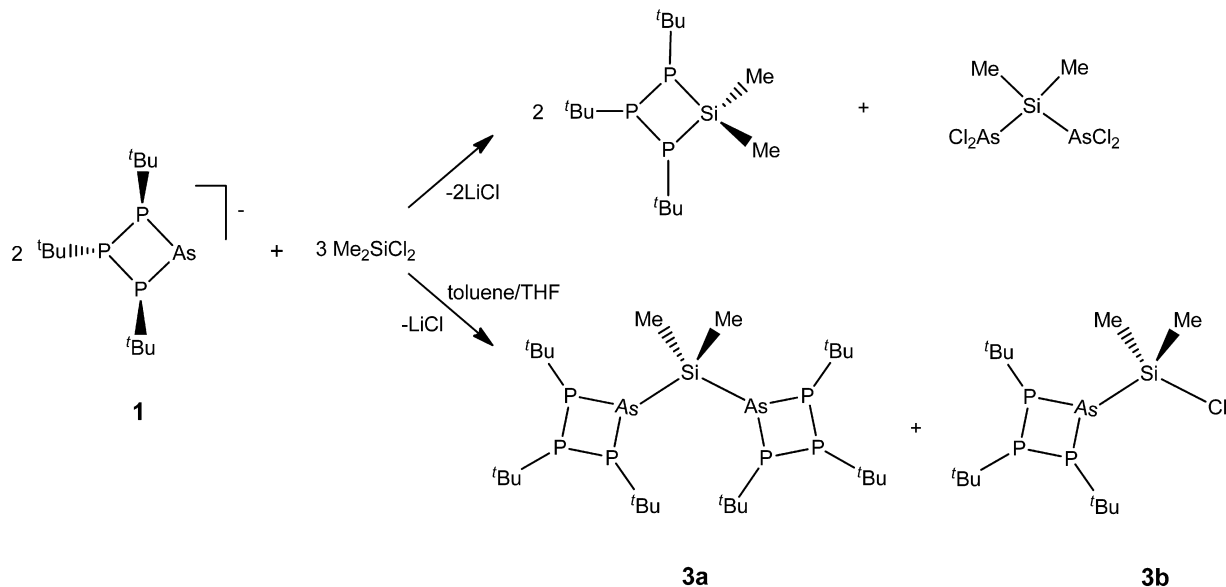
**Scheme 4.**
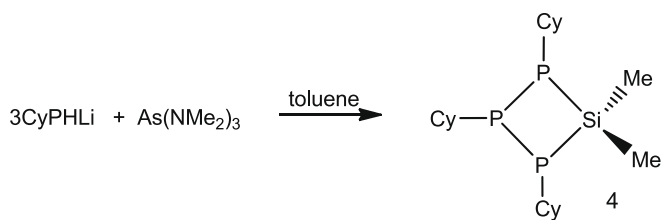


**Scheme 5.**

−20 °C gave a 23% yield of light-yellow crystals of **3a**, which was spectroscopically- and structurally-characterised. However, repeated attempts to isolate **3b** using various reaction stoichiometries and conditions failed. The isolation of **3a** as the product was confirmed by $^{31}$P{$^1$H} spectroscopy at room temperature (in d$_8$-THF). Interestingly, the $^1$H NMR spectrum of **3a** (in benzene) shows three doublets in the $^t$Bu region (ratio 1:1:1), consistent with the adoption of a rigid molecular conformation in solution in which the three $^t$Bu groups are magnetically inequivalent. This is not altogether unexpected bearing in mind the sterically congested nature of the solid-state molecular structure (Fig. 1), which is likely to result in restricted rotation of the Si–As bonds. Table 1 summarises the $^{31}$P NMR spectroscopic data on the new compounds **3a**, **3b** and **4**.

A number of reactions of **2**·TMEDA·THF with R$_2$SiCl$_2$ (R = Me, Ph) were undertaken. Several unidentified products were observed by in situ $^{31}$P{$^1$H} NMR spectroscopy. However, irrespective of the solvent or reaction conditions, no solid products could be isolated. A further problem which limited the extent of these studies was

the fact that the starting material **2**·TMEDA·THF, although the most easily prepared source of the anion **2**, is only isolated in relatively low yield from the 3:1 reaction of CyPHLi with As(NMe$_2$)$_3$ in the presence of TMEDA/THF and is difficult to purify (taking several weeks to crystallize). We therefore investigated the in situ generation of the anion **2** via the 3:1 reaction of CyPHLi with As(NMe$_2$)$_3$ in TMEDA/THF. An in situ $^{31}$P{$^1$H} spectroscopic study of this reaction mixture does indeed show the presence of significant amounts of the [(CyP)$_4$As]$^-$ anion, in addition to a number of other unidentified species. Reaction of this solution at −78 °C with an excess of Me$_2$SiCl$_2$ gave a precipitate of LiCl. The in situ NMR spectrum of the filtered solution clearly shows the presence of [(CyP)$_3$SiMe$_2$] (**4**) as a principal product of the reaction (see Table 2). Crystalline **4** was isolated in 10% yield after storage of the reaction solution at −20 °C. The room temperature $^{31}$P{$^1$H}NMR spectrum of **4** shows the expected triplet [δ −44.8, P(2)] and doublet [δ −97.6, P(1,3)] ($^1J_{P-P}$ = 128 Hz). These resonances are distinctly different from those found for either **3a** or **3b**, providing preliminary evidence of the presence of a very different (P$_3$Si as opposed to P$_3$As) ring unit in **4**. It is not certain how the four-membered P$_3$Si ring of **4** is formed in this reaction. However, it can be noted that no intermediate [(CyP)$_3$As]$^-$anion could be observed in the in situ reaction mixture of CyPHLi with As(NMe$_2$)$_3$, prior to the addition of Me$_2$SiCl$_2$. Thus, it appears that the four-membered ring of **4** is generated from the observed intermediate five-membered anion [(CyP)$_4$As]$^-$ (**2**). The observation that the reaction of Ph$_2$SiCl$_2$ with CyPHLi does not generate **4** (only the Si$_2$P$_2$ ring compound trans-[Ph$_2$SiPCy]$_2$) [8] further supports the view that As(NMe$_2$)$_3$ is vital to the formation of **4**, presumably because it is responsible for the formation of P–P bonds before electrophilic ring opening occurs with Me$_2$SiCl$_2$.

The low-temperature [180(2) K] X-ray structure of **3a** confirms the essential features ascertained from spectroscopic analysis. The molecular structure consists of two [($^t$BuP)$_3$As] ring units which are bridged by a Me$_2$Si group (Fig. 1). The structure provides final confirmation that nucleophilic attack of the [($^t$BuP)$_3$As]$^-$ anion at the silicon centre occurs in this case, as opposed to electrophilic ring opening. The bridging Si atom exhibits a distorted tetrahedral geometry [range of angle at Si(1) 101.69(7)–115.9(2)°], while the acute angles at the As centres [82.95(7)–98.53(7)°] a large degree of p-orbital character within the bonds involved. The mean planes

**Table 1**
Summary of $^{31}$P{$^1$H} NMR data for the new compounds **3a**, **3b** and **4**.

| Compound | Chemical shift (δ) (coupling constant, J) |
|----------|--------------------------------------------|
| 3a | −62.6 [d, P(1,3)], −15.7 [t, P(2)] ($^1J_{P-P}$ = 159 Hz) |
| 3b | −63.4 [d, P(1,3)], −13.5 [t, P(2)] ($^1J_{P-P}$ = 156 Hz) |
| 4 | −97.6 [d, P(1,3)], -44.8, [t, P(2)] ($^1J_{P-P}$ = 128 Hz) |
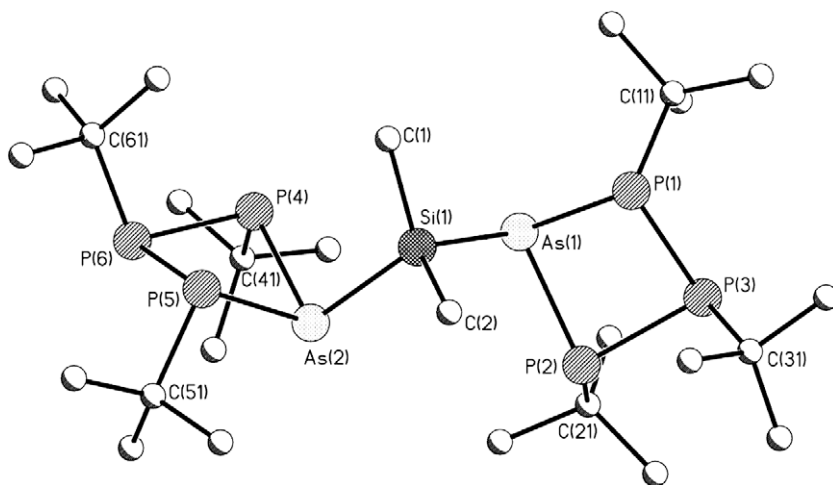
**Fig. 1.** Molecular structure of the di-substituted product **3a**. H-atoms have been omitted for clarity. Key bond lengths (Å) and angle (°); P(1)–P(2) 2.215(2), P(2)–P(3) 2.223(3), P(1)–As(1) 2.346(2), P(2)–As(1) 2.342(2), As(1)–Si(1) 2.358(2), P(4)–P(6) 2.221(3), P(5)–P(6) 2.216(2), P(4)–As(2) 2.350(2), P(5)–As(2) 2.340(2), As(2)–Si(1) 2.363(2), P(1)–P(3)–P(2) 88.90(9), P(3)–P(1)–As(1) 86.77(8), P(3)–P(2)–As(1) 86.70(9), P(1)–As(1)–P(2) 82.95(7), P(1)–As(1)–Si(1) 97.25(7), P(2)–As(1)–Si(1) 97.52(7), P(4)–P(6)–P(5) 89.30(9), P(6)–P(5)–As(2) 86.86(8), P(6)–P(4)–As(2) 86.50(8), P(4)–As(2)–P(5) 83.40(7), P(4)–As(2)–Si(1) 98.53(7), P(5)–As(1)–Si(1) 96.33(7), As(1)–Si(1)–As(2) 101.69(7) [range of other angles at Si 106.7(2)–115.9(2)], mean puckering of the $P_3As$ rings 140 (about the P···P vectors).

**Table 2**
Details of the data collections and structural solutions of **3a** and **4**.

| Compound | **3a** | **4** |
|---|---|---|
| Chemical formula | $C_{26}H_{60}As_2P_6Si$ | $C_{20}H_{39}P_3Si$ |
| Formula weight | 736.49 | 400.51 |
| Crystal system | triclinic | monoclinic |
| Space group | $P\bar{1}$ | $P2(1)/m$ |
| *Unit cell dimensions* | | |
| $a$ (Å) | 9.4278(2) | 5.934(2) |
| $b$ (Å) | 13.5822(3) | 15.006(4) |
| $c$ (Å) | 16.7416(4) | 13.576(3) |
| $\alpha$ (°) | 113.4493(9) | |
| $\beta$ (°) | 91.6253(9) | 100.74(3) |
| $\gamma$ (°) | 95.4497(10) | |
| $V$ (Å³) | 1952.69(8) | 1187.7(6) |
| $Z$ | 2 | 2 |
| $\rho_{calc}$ (Mg/m³) | 1.253 | 1.120 |
| $\mu$ (Mo K$\alpha$) (mm⁻¹) | 2.002 | 0.302 |
| Reflections collected | 23 800 | 2505 |
| Independent reflections ($R_{int}$) | 5592 | 1735 |
| | (0.080) | (0.045) |
| $R_1$, $wR_2$ [$I > 2\sigma(I)$] | 0.052, 0.133 | 0.052, 0.120 |
| $R_1$, $wR_2$ (all data) | 0.081, 0.149 | 0.091, 0.140 |

Data in common; $\lambda = 0.71073$ Å, $T = 180(2)$ K.

of the two $P_3As$ ring units are orientated roughly perpendicularly to each other largely in order to minimise steric congestion between the $^tBu$ and Me groups across the bridge. A further effect of this alignment of the $P_3As$ rings is the reduction of potential repulsion between the lone-pairs on the As centres (which are roughly perpendicular to each other). The As–Si (mean 2.36 Å), P–As (mean 2.34 Å) and P–P (mean 2.22 Å) bonds in **3a** are typical of single bonds of these types [10]. While the puckering of the [($^tBuP)_3As$] ring units in **3a** (dihedral angle of 140° about the P···P vectors of the $P_3As$ rings) is similar to that found in the structures a range of compounds containing neutral and anionic [($RP)_3As$] units; most notably in the As–As bonded dimer [($^tBuP)_3As]_2$ [11]. The conformation of the [($^tBuP)_3As$] ring units in **3a** and related compounds is (not surprisingly) largely influenced by steric rather than electronic factors.

The structure of **4** is that of a four-membered, $P_3Si$ heterocycle of crystallographic $Cs$ symmetry (Fig. 2), and is closely related to the previously reported tin(IV) heterocycle [($^tBuP)_3Sn^tBu_2$] [12].
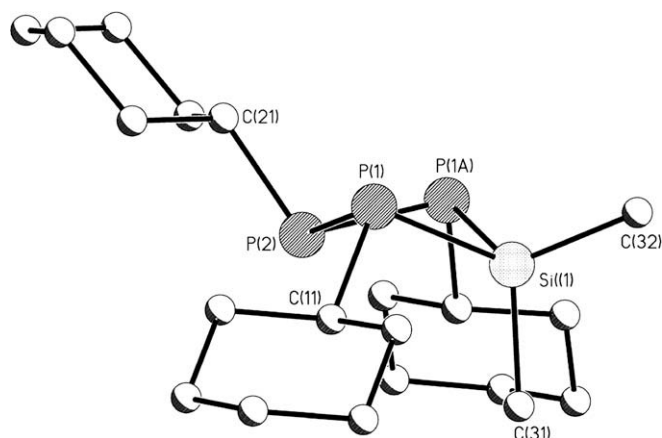


**Fig. 2.** Molecular structure of the $P_3Si$ heterocycle **4**. H-atoms have been omitted for clarity. Key bond lengths (Å) and angle (°); P(1)–P(2) 2.236(2), P(1)–Si(1) 2.274(2), P(1)–P(2)–P(1A) 91.21(8), P(2)–P(1)–Si(1) 81.28(6), P(1)–Si(1)–P(1A) 89.28(8), puckering of $P_3Si$ ring 134.7 (about the P(1)···P(1A) vector).

Nonetheless, **4** is the first example of a heterocycle containing a $P_3Si$ ring unit, although related five-membered $P_4Si$ and $P_4Ge$ systems have been reported previously [13]. As in the structure of **3a**, the P–Si [2.274(2) Å] and P–P [2.236(2) Å] bonds are as expected for single bonds [10]. Like [($^tBuP)_3Sn^tBu_2$] [12] and [($RP)_3As$]⁻ anions [2], the heterocyclic ring of **4** is puckered [dihedral angle of 134.7° at the P(1)···P(1A) vector].

## 3. Conclusions

The results presented in this paper show that the reactions of [($RP)_nAs$]⁻ anions with main group electrophiles (in this case $Me_2SiCl_2$) can provide access to a range of new heterocyclic species. The reaction characteristics observed in this study, nucleophilic substitution in the case of **2** and apparent electrophilic ring opening in the case of **4**, have been seen previously in studies of the interaction of the [($^tBuP)_3As$]⁻ anion with transition metal complexes and organic electrophiles (see Scheme 3). Further studies with main group metal complexes should be of interest, especially where lower oxidation state metal halides are involved.

## 3. Experimental

### 3.1. General experimental procedures

All compounds described in this paper are air- and moisture sensitive. Preparations were performed on a double-manifold vacuum line under argon atmosphere. Products were isolated and stored with the aid of a nitrogen-filled glove box (Saffron type β), equipped with Cu and molecular sieve columns in order to remove $O_2$ and moisture (respectively) from the atmosphere. $[\{(^tBuP)_3As]Li\cdot2DABCO\cdot THF]$ ($1\cdot2DABCO\cdot THF$) and $[\{(CyP)_4As\}Li\cdot TMEDA\cdot THF]$ ($2\cdot TMEDA\cdot THF$) were prepared as crystalline samples for further reactions according to the literature procedures. $Me_2SiCl_2$ (Aldrich) was used as supplied commercially. TMEDA (Aldrich) was dried over Na and stored under argon. In situ $^{31}P$ NMR studies were undertaken by placing ca. 0.8 ml of the reaction solutions within a Wilmad 528PP (thin-walled) NMR tube containing $d_6$-acetone capillary to obtain a lock. All $^1H$ and $^{31}P\{^1H\}$ and fully-coupled NMR spectra were recorded using a Bruker DPX 500 MHz NMR spectrometer. $^{31}P$ NMR spectra were referenced to an external standard of 85% $H_3PO_4/D_2O$, while $^1H$ NMR spectra were referenced internally to the solvent peaks. Elemental (C, H, N) analyses were obtained using an Exeter CE-440 Elemental Analyser. P analysis was obtained using spectrophotometric methods. Samples for analysis (1–2 mg) were placed in pre-weighed, air-tight aluminium boats in the glove box prior to analysis.

Synthesis of **3a**; $Me_2SiCl_2$ (0.22 ml, 1.8 mmol) was added to a solution of $2\cdot2DABCO\cdot THF$ (0.80 g, 1.2 mmol) in toluene (30 ml) and THF (30 ml) at room temperature. The resulting solution was allowed to stir for ca. 20 h, resulting in a yellow solution and a white precipitate. The solution was filtered through Celite and the solvent partially removed under vacuum to generate a saturated solution (ca. 30 ml remaining). Storage of the solution at $-20\,°C$ afforded colourless crystals of **3a**. Yield 0.42 g (23%). M.p. decomp. >185 °C. $^{31}P\{^1H\}$ (161.98 MHz, +25 °C, $d_8$-THF), $\delta = -62.6$ (d, 2P), $-15.7$ (t, P) ($^1J_{P–P} = 159$ Hz). $^1H$ NMR (400.16 MHz, +25 °C, $d_6$-benzene), $\delta = 1.26$ (d, 18H, $^tBu$, $^3J_{P–H} = 12.2$ Hz), 1.24 (d, 18H, $^tBu$, $^3J_{P–H} = 12.8$ Hz), 1.17 (d, 18H, $^tBu$, $^3J_{P–H} = 13.0$ Hz), 1.01 (s, 6H, $SiMe_3$). Calculated for **3a**, C, 42.4; H 8.2; cald. for **3a**, C 41.1, H, 7.1.

Synthesis of **4**; $^nBuLi$ (8.0 ml, 1.5 mol dm$^{-3}$ in hexanes, 12.0 mmol) was added dropwise to a stirred solution of $CyPH_2$ (1.6 ml, 12.0 mmol) in toluene (30 ml) was cooled to $-78\,°C$. Stirring at room temperature for 1 h gave a yellow precipitate of the lithiate. The mixture was cooled to $-78\,°C$ and a solution of $As(NMe_2)_3$ in toluene (2.0 ml, 2.0 mol dm$^{-3}$, 4.0 mmol). After stirring for 24 h at room temperature, $Me_2SiCl_2$ (1.47 ml, 12.0 mmol, excess) was added dropwise to the resulting orange solution at $-78\,°C$. After stirring for 48 h a yellow solution was produced. Reduction of the volume of this solution under vacuum gave a precipitate which was heated back into solution. Storage of the solu-

tion at $-25\,°C$ afforded yellow crystals of **4**. Yield 0.16 g (10%). $^{31}P\{^1H\}$ (161.98 MHz, +25 °C, $d_6$-benzene), $\delta = 2.0$–$0.8$ (m, 44H, Cy). $^1H$ NMR (400.16 MHz, +25 °C, $d_6$-benzene), $\delta = -44.8$ (t, 1P), $-97.6$ (d, 2P) ($^1J_{P–P} = 127.6$ Hz). $^{29}Si$ NMR (79.49 MHz, +25 °C, $d_6$-benzene, rel. $Me_4Si$), $\delta = -23.5$. Calculated for **4**, C, 60.0; H, 9.8; P, 23.2; cald. for **2**, C, 60.1, H, 10.1, P, 24.2.

### 3.2. X-ray crystallographic studies of synthesis of **2** and **4**

Data for both complex were collected on a Nonius Kappa CCD diffractometer and solved by direct methods and refined by full-matrix least squares on $F^2$ [14].

## 4. Supplementary material

CCDC 756885 and 756886 contain the supplementary crystallographic data for **3a** and **4**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

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