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Polyhedron 22 (2003) 211–216
POLYHEDRON
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Organo-functionalised arsine and stibine organometallics; syntheses and structural characterisations of 1,3-$[(PhC{\equiv}C)_2Sb]_2(CH_2)_3$, $As(C{\equiv}CPh)_3$, $R_2AsCH_2AsR_2$ [R = $Me_3SiC{\equiv}C$-, $(Me_3Si)_2N$- and 2-SPy] with π-stacking in the latter

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Received 29 August 2002; accepted 28 October 2002

Abstract

A series of dinuclear organo-functionalised stibine and arsine ligands incorporating a range of hard, soft and π-donor moieties have been synthesised from the reactions of $Cl_2Sb(CH_2)_3SbCl_2$ and $Cl_2AsCH_2AsCl_2$ with a range of organolithium reagents. The X-ray structures of 1,3-$[(PhC{\equiv}C)_2Sb]_2(CH_2)_3$ (**1**), $As(C{\equiv}CPh)_3$ (**2**), $R_2AsCH_2AsR_2$ [R = $Me_3SiC{\equiv}C$, **3**; $(Me_3Si)_2N$, **4**; and 2-SPy **5**] have been determined. Compound **5** associates into pseudo dimers as a result of intermolecular π–π stacking between the pyridyl groups on one end of two separate diarsine molecules. Whereas the bridging unit remains intact during the syntheses of **1** and **3**–**5**, the formation of the mononuclear trisacetylide species **2**, involves the unexplained cleavage of the bridging methylene unit.

Keywords: Stibine; Arsine; Synthesis; X-ray structures; π-Stacking; Group 15

1. Introduction

The organometallic and ligand chemistry of Group 15 is now well established [1]. Previous work into the reactivities of Group 15 dimethylamido derivatives, $E(NMe_2)_3$ (E = As, Sb, Bi) [2], has generated a range of heteropolymetallic cage species, the structural diversity of which is the subject of a great deal of interest [3,4]. To further this work, we have investigated the synthesis and characterisation of mixed-metal compounds based around polynuclear Group 15 backbones [5,6]. In the course of this work we have thus investigated the possible functionalisation of polynuclear Group 15 bimetallic precursors.

Recently there has been great interest in the use of binuclear Group 15 complexes as Lewis base donors towards transition metal species [7,8]. The complexes $R_2E(CH_2)_nER_2$ (where E = P, As, Sb; $n = 1, 2, 3$; R = Me, Ph), have been used to generate a host of heterometallic coordination complexes [9]. However, to date there have been only limited examples of organo-functionalised binuclear species [10]. It was thus our intention to investigate the syntheses and reactivities of a series of novel organo-substituted polynuclear Group 15 ligands containing side-arm donors possessing a more diverse set of donor functionalities incorporating hard (N) and soft (As, S) Lewis base sites as well as π-systems (C≡C and Py). By having multiple sites on the ligand available for reaction it may be possible to tune reactivity and create larger heterometallic species incorporating a number of different metal complexes. Utilising dinuclear arsine and stibine frameworks we have been able to synthesise a number of new organometallic species. Here we report the syntheses of the organofunctionalised stibine and arsine compounds 1,3-$[(PhC{\equiv}C)_2Sb]_2(CH_2)_3$ (**1**), $As(C{\equiv}CPh)_3$ (**2**), $R_2AsCH_2AsR_2$ [R = $Me_3SiC{\equiv}C$, **3**; $(Me_3Si)_2N$, **4**; and 2-SPy **5**] (Fig. 1).

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0277-5387/02/$ - see front matter
PII: S0277-5387(02)01339-6

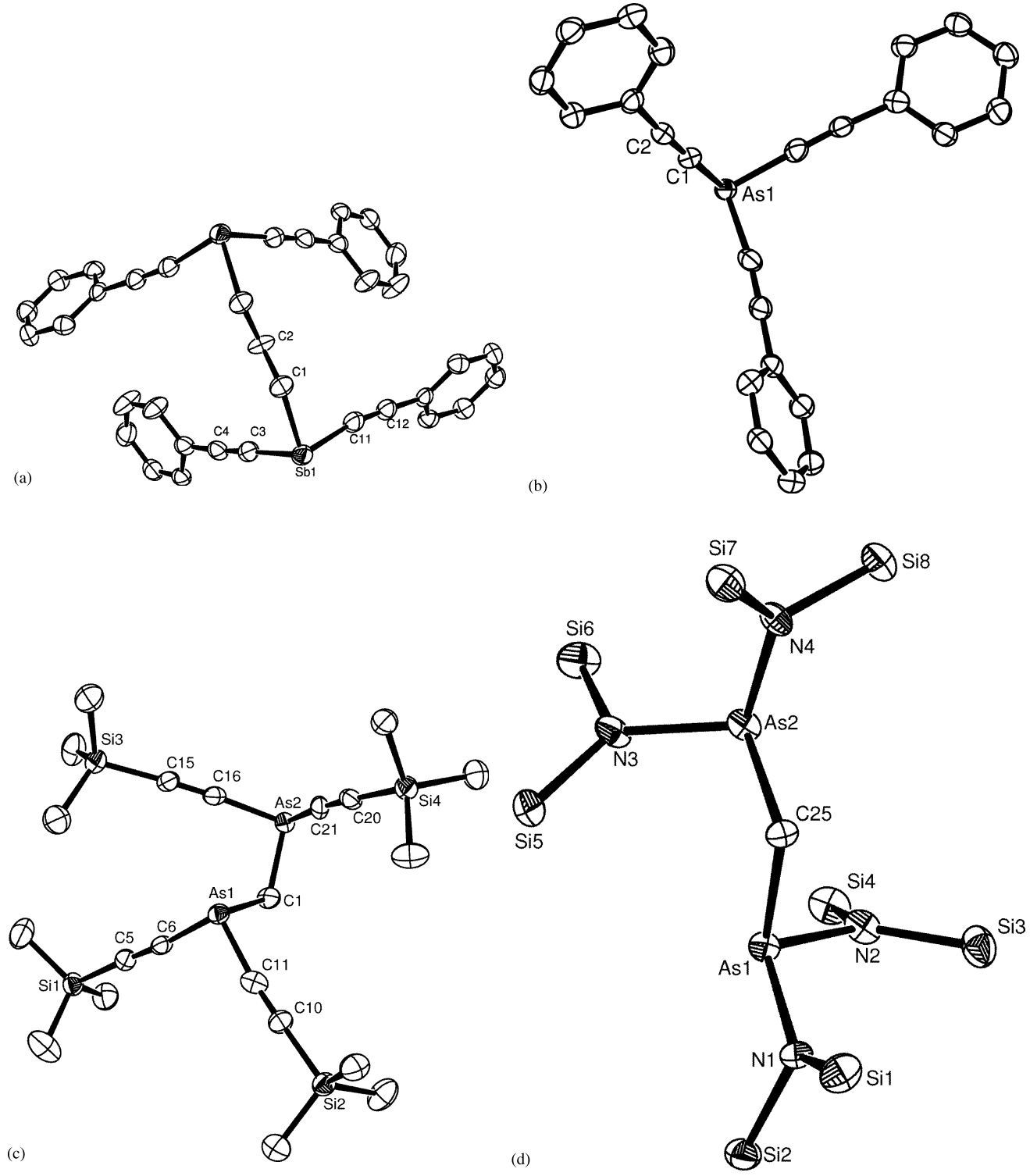


Fig. 1. Crystal structures of compounds **1**–**5** showing important bond lengths (Å) and angles (°). H-atoms and selected (chemically non-important) C atoms are omitted for clarity: (a) molecular structure of **1**. Sb(1)–C(1) 2.155(6), Sb(1)–C(11) 2.096(7), C(11)–C(12) 1.198(9), C(3)–C(4) 1.200(10), C(1)–Sb(1)–C(3) 96.5(2), C(11)–Sb(1)–C(3) 94.8(3), Sb(1)–C(3) 2.096(7), C(11)–Sb(1)–C(1) 95.4(2); (b) molecular structure of **2**. As(1)–C(1) 1.907(6), C(1)–C(2) 1.199(9), C(1)#1–As(1)–C(1) 96.4(3); (c) molecular structure of **3**. As(1)–C(1) 1.976(6), As(1)–C(11) 1.912(7), As(1)–C(6) 1.913(6), As(2)–C(1) 1.957(6), As(2)–C(21) 1.908(6), As(2)–C(16) 1.902(7), As(1)–C(1)–As(2) 115.3(3), C(11)–As(1)–C(6) 97.5(2), C(11)–As(1)–C(1) 96.2(3), C(6)–As(1)–C(1) 96.9(3), C(16)–As(2)–C(21) 97.5(3), C(21)–As(2)–C(1) 96.0(3), C(16)–As(2)–C(1) 96.4(3); (d) molecular structure of **4**. As(1)–C(25) 1.972(7), As(1)–N(1) 1.893(6), As(1)–N(2) 1.898(6), As(2)–C(25) 1.981(7), As(2)–N(3) 1.893(6), As(2)–N(4) 1.901(6), As(1)–C(25)–As(2) 107.5(3), N(1)–As(1)–N(2) 107.0(3), N(1)–As(1)–C(25) 105.2(3), N(2)–As(1)–C(25) 98.7(3), N(3)–As(2)–N(4) 107.1(3), N(3)–As(2)–C(25) 104.5(3), N(4)–As(2)–C(25) 100.0(3); (e) structure of **5** showing orientation of weak dimer pairs. As(1)–C(21) 1.972(4), As(1)–S(1) 2.2802(14), As(1)–S(2) 2.267(2), As(2)–C(21) 1.975(4), As(2)–S(3) 2.279(2), As(2)–S(4) 2.267(2), As(1)–C(21)–As(2) 115.9(2), S(1)–As(1)–S(2) 85.19(6), S(1)–As(1)–C(21) 99.78(13), S(2)–As(1)–C(21) 99.04(14), S(3)–As(2)–S(4) 91.04(6), S(3)–As(2)–C(21) 97.31(13), S(4)–As(2)–C(21) 92.88(14).

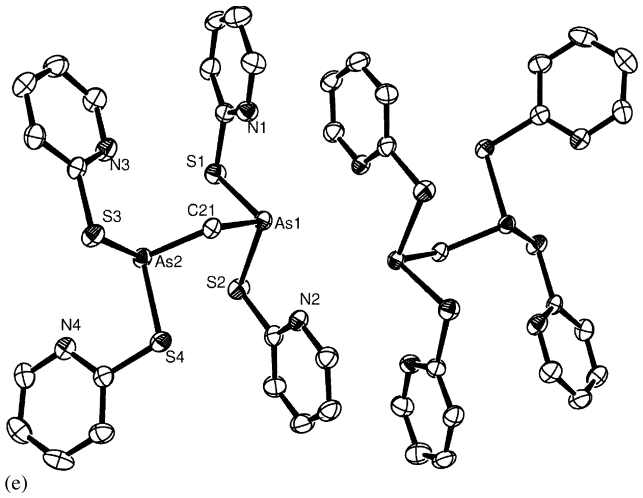


Fig. 1 (*Continued*)

2. Experimental

2.1. General procedure

The products **1**, **2**, **3**, **4**, **5**, the starting materials $Cl_2Sb(CH_2)_3SbCl_2$, $Cl_2AsCH_2AsCl_2$ and ^nBuLi are all air and/or moisture sensitive. They were handled on a vacuum line using standard inert atmosphere techniques under dry and oxygen-free N_2. The $C_6H_5CH_3$, C_6H_{14}, Et_2O and THF solvents were dried using sodium/benzophenone and were degassed prior to their use in reactions. The hexamethyldisilazane and trimethylsilylacetylene were dried using molecular sieve (13 ×). The celite used for filtration was previously dried at 120 °C for 2 days. All complexes were isolated and characterised with the aid of an N_2 filled glove box (Belle Technology) fitted with an O_2 and H_2O recirculation system. Elemental analyses were performed by firstly sealing samples under Ar in airtight aluminium boats (1–2 mg). ^1H NMR spectra were recorded on Bruker AM 300MHz spectrometer in dry d^6 C_6H_6 (using the solvent resonances as the internal reference standard and are reported in δ ppm). Samples of $Cl_2Sb(CH_2)_3SbCl_2$ and $Cl_2AsCH_2AsCl_2$ were prepared in accordance with the previously reported methods [11,12].

2.2. Synthesis of *1*

A solution of 0.409 g (4 mmol) of PhC≡CH in 20 ml of Et_2O was reacted with 2.5 ml of ^nBuLi (4 mmol, 1.6 M sol.) at −78 °C. The reaction was then stirred at room temperature (r.t.) for approximately 30 min. The colourless solution obtained was then cooled to 0 °C and a solution of 0.427 g (1 mmol) of $Cl_2Sb(CH_2)_3SbCl_2$ in $C_6H_5CH_3$ was added. The mixture was allowed to react at r.t. for 2 h after which time the Et_2O removed in vacuo and the product was extracted using $C_6H_5CH_3$. The mixture was filtered through celite to remove LiCl and the product was then crystallised from approximately 20 ml of Et_2O (65%). ^1H NMR (C_6D_6): 1.45 (t, 4H, Sb–CH_2–), 2.85 (qn, 2H, –CH_2–), 6.8–7.2 (m, 20H, phenyl). C, H, N: *Anal.* Found: C, 59.8; H, 3.7. Calc. for $C_{35}H_{26}Sb_2$: C, 60.9; H, 3.8%.

2.3. Synthesis of *2*

A solution of 1.021 g (10 mmol) of PhC≡CH in 40 ml Et_2O was stirred at −78 °C and 6.25 ml (10 mmol, 1.6 M sol.) of ^nBuLi was added. This mixture was stirred for approximately 30 min and allowed to warm back to r.t. The solution obtained was then cooled to 0 °C and to it was added a solution of 0.764 g (2.5 mmol) of $Cl_2AsCH_2AsCl_2$ in Et_2O. The reaction was allowed to stir for 2 h and the Et_2O then removed in vacuo. The product was extracted using $C_6H_5CH_3$ (40 ml) and filtered through celite (to remove LiCl), then recrystallised in 15 ml of $C_6H_5CH_3$. Storage overnight at 5 °C yielded colourless needles (40%). ^1H NMR (C_6D_6): 6.8–7.4 (m, phenyl). C, H, N: *Anal.* Found: C, 74.2; H, 4.0. Calc. for $C_{24}H_{15}As_1$: C, 76.2; H, 4.0%.

2.4. Synthesis of *3*

A solution of 0.982 g (10 mmol) of $Me_3SiC{\equiv}CH$ in 20 ml of $C_6H_5CH_3$ was reacted at −78 °C with 6.25 ml (10 mmol, 1.6 M sol.) of ^nBuLi with stirring and the reaction mixture was allowed to warm to r.t. over a further 30 min. The resultant colourless solution was cooled to 0 °C and 0.764 g (2.5 mmol) of $Cl_2AsCH_2AsCl_2$ was added and the mixture stirred at r.t. for approximately 30 min. The white precipitate of LiCl was removed by filtration through celite to yield a colourless solution. This was reduced to dryness in

vacuo, and 20 ml of C_6H_{14} added to dissolve the residue. Reduction in vacuo to approximately 5 ml yielded a white precipitate which redissolved upon gentle warming. Storage at 5 °C overnight produced a crop of colourless crystalline blocks of **3** in 75% yield. ^{1}H NMR (C_6D_6): 0.07 (s, 36H, Me), 2.07 (s, 2H, As–CH_2–As). C, H, N: *Anal.* Found: C, 45.2; H, 6.8. Calc. for $C_{21}H_{38}As_2Si_4$: C, 45.7; H, 6.9%.

2.5. Synthesis of 4

An identical procedure to the synthesis of **3** was employed utilising 1.614 g (10 mmol) of $(Me_3Si)_2NH$ and 6.25 ml (10 mmol, 1.6 M sol.) of ^{n}BuLi. The product was again isolated as colourless crystalline blocks from C_6H_{14} solution in 70% yield. ^{1}H NMR (C_6D_6): 0.08 (s, 72H, Me), 2.51 (s, 2H, As–CH_2–As). C, H, N: *Anal*. Found: C, 36.8; H, 9.2; N, 6.8. Calc. for $C_{25}H_{74}As_2N_4Si_8$: C, 37.2; H, 9.2; N, 7.0%.

2.6. Synthesis of 5

A solution of 1.112 g (10 mmol) of 2-PySH in 10 ml of THF was reacted at 0 °C with 6.25 ml (10 mmol) of ^{n}BuLi and allowed to warm to r.t. over approximately 30 min to yield a pale yellow solution. After chilling to −78 °C, 0.764 g (2.5 mmol) of $Cl_2AsCH_2AsCl_2$ was added and the reaction mixture stirred for 30 min at r.t. to yield a yellow solution. This was reduced to dryness in vacuo and the product extracted into 20 ml of hot $C_6H_5CH_3$. Filtration through celite (to remove LiCl) yielded a lemon yellow solution, which upon reduction in vacuo to approximately 12 ml yielded a yellow precipitate. This readily redissolved upon heating and storage at 5 °C overnight produced a crop of lemon yellow crystals of **5** in 75% yield. ^{1}H NMR (C_6D_6): 2.28 (s, 2H, As–CH_2–As), 7.15–7.62 (m, 16H, pyridyl groups). C, H, N: *Anal.* Found: C, 41.1; H, 3.1; N, 9.0. Calc. for $C_{21}H_{18}As_2N_4S_4$: C, 41.7; H, 3.0; N, 9.3%.

2.7. X-ray crystallography

Data for **1** and **2**, were collected in an Enraf–Nonius KappaCCD area detector diffractometer whilst **3**, **4** and **5** were collected on a AFC5 Rigaku diffractometer, both fitted with Mo Kα radiation and a graphite monochromator. All compounds are air sensitive and were kept under a flow of nitrogen at 150 K for **1**–**4** and 180 K for **5**; data collection and cell refinement [13] gave the cell dimensions shown in Table 1. For **1** and **2** a multi-reflection absorption correction [14,15] was applied and for **3**, **4** and **5** a semiempirical absorption correction based on psi-scans was applied. The data solved via direct methods [16] and the structures refined using the WINGX [17] version of SHELXS-97 [18]. All non-hydrogen atoms were treated anisotropically. In **1** the hydrogen atoms were located in the difference map and refine isotropically. For compounds **2**, **3**, **4** and **5** the hydrogen atoms were included in idealised positions with C–H set at distances between 0.950 and 0.990 Å and with isotropic thermal parameters set at 1.2 times that of the carbon atom to which they were attached. Details of the structural solutions and crystal information for **1**, **2**, **3**, **4** and **5** are presented in Table 1. Diagrams of the five structures are given as Fig. 1(a–e).

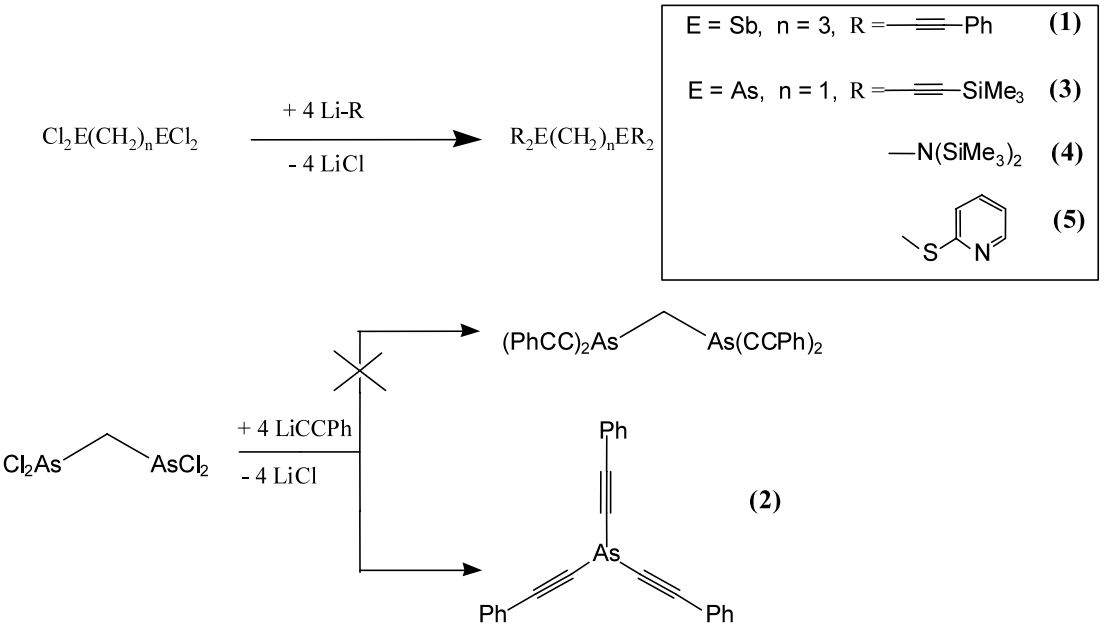
3. Results and discussion

The five compounds reported represent significant additions to the polynuclear arsine and stibine ligands available to coordination chemists, and we have demonstrated that it is possible to readily add functionalised organic groups onto the metal centres. Compound **1** is an example of a binuclear organo-functionalised stibine and comparison with the previously reported structure of $Ph_2Sb(CH_2)_3SbPh_2$ reveals a similar structural motif (Fig. 1a) [19]. The pyramidal angles about the antimony centres range from 94.8(3)°–96.5°(2) compared to 94.4(1)°–98.7°(1) for $Ph_2Sb(CH_2)_3SbPh_2$. During the synthesis of compound **2** (Fig. 1b) the methylene bridge between the arsenic centres has broken (Scheme 1). This behaviour in relation to the other four examples is a noticeable exception. However, previous work on the $AsCH_2As$ backbone had led us to investigate this cleavage before. Thus, it has been noted that during the formation of benzo 1,3,2 diazaarsolyl lithium·2THF from the reaction of 1,2-LiHN–2-$NH_2C_6H_4$ with $(Me_2N)_2AsCH_2As(NMe_2)_2$ (4:1 equiv.) the methylene bridge in the precursor is broken during the course of the reaction [5]. Once again the carbon backbone here has broken during synthesis, creating a mononuclear trisacetylide species. Further investigation of the reaction mother liquor did not yield any readily identifiable arsenic or methyl-arsine species. However, it is reasonable to assume that the mother solution is likely to contain a second, as yet unidentified, arsenic species. Efforts are continuing to identify these products and also, therefore, to provide a better understanding of the reaction mechanism. It should also be noted that following the synthesis of this acetylide from the dinuclear precursor the same reaction was attempted using $AsCl_3$ instead of the diarsine, leading to an enhanced yield of **2** as the only product. In contrast to this, compounds **3**, **4** and **5** are all synthesised from the dinuclear precursor and maintain their framework during the synthesis. There is no clear rationale behind this, but as noted with previous work, the cleavage of such bridges is possible in the syntheses of related complexes. It is sufficient at this time to comment that whether the bridge is likely to break or remain intact depends solely on the reaction constituents and the

Table 1
Crystal data and structure solution of 1,3-[(PhC≡C)$_2$Sb]$_2$(CH$_2$)$_3$ (**1**), As(C≡CPh)$_3$ (**2**) [(Me$_3$SiC≡C)$_2$As]$_2$CH$_2$ (**3**), {[(Me$_3$Si)$_2$N]$_2$As$_2$}CH$_2$ (**4**), [(2-SPy)$_2$As]$_2$CH$_2$ (**5**)

	1	**2**	**3**	**4**	**5**
Empirical formula	$C_{35}H_{26}Sb_2$	$C_{24}H_{15}As_1$	$C_{21}H_{38}As_2Si_4$	$C_{25}H_{74}As_2N_4Si_8$	$C_{21}H_{18}As_2N_4S_4$
M	690.06	378.28	552.71	805.44	604.47
T (K)	150(2)	150(2)	150(2)	150(2)	180(2)
Wavelength (Å)	0.71073	0.71073	0.71069	0.71069	0.71069
Crystal system	monoclinic	trigonal	monoclinic	monoclinic	triclinic
Space group	$C2$	$R3$	$P2_1/c$	$P2_1/n$	$P\bar{1}$
a (Å)	19.054(4)	18.6408(15)	11.581(5)	12.901(5)	9.725(3)
b (Å)	5.5344(11)	18.6408(15)	16.681(5)	19.307(3)	14.576(4)
c (Å)	13.790(3)	4.4235(7)	16.406(5)	18.405(3)	8.545(4)
α (°)		90			93.58(3)
β (°)	91.94(3)	90	110.029(5)	101.39(2)	90.85(4)
γ (°)		120			103.34(2)
U (Å^3)	1453.4(5)	1331.1(3)	2978(2)	4494(2)	1175.8(7)
Z	2	3	4	4	2
D_c (Mg m^{-3})	1.577	1.416	1.233	1.190	1.707
θ Range	$2.14 \le \theta \le 27.45$	$3.79 \le \theta \le 25.03$	$2.64 \le \theta \le 25.01$	$2.65 \le \theta \le 22.51$	$2.70 \le \theta \le 27.54$
Crystal size (mm)	$0.25 \times 0.02 \times 0.02$	$0.1 \times 0.02 \times 0.02$	$0.2 \times 0.1 \times 0.1$	$0.3 \times 0.25 \times 0.22$	$0.3 \times 0.2 \times 0.15$
Reflections collected	6022	2363	5506	6152	5669
Independent reflections	3010	955	5233	5849	5354
Goodness-of-fit on F^2	0.733	1.072	1.028	1.009	1.027
R indices [$F > 4\sigma(F)$]: R_1, wR_2	0.0399, 0.0984	0.0536, 0.1260	0.0563, 0.1140	0.0536, 0.1057	0.0458, 0.0917
R indices (all data): R_1, wR_2	0.0507, 0.1088	0.0579, 0.1290	0.0991, 0.1327	0.1518, 0.1800	0.0750, 0.1035
Final difference peak and hole (e Å^{-3})	1.408, −0.891	0.659 −0.847	0.718, −0.800	0.521, −0.575	0.497, −0.653
CCDC number	192078	192077	192080	192081	192079

stability of the potential products formed. Compounds **3**, **4** and **5** all appear to be similar in nature, however, there are significant structural differences. For **3** (Fig. 1c) the addition of four acetylide groups is as expected. Comparison of the angles around the arsenic centres with those obtained from **2** and other literature [16] diarsine compounds reveals that the angles are very similar. This tends to indicate that, as expected, the sterically non-bulky acetylide groups do not have any noticeable influences on the ideally pyramidal arsenic centres. The As–CH$_2$–As angle (115.3°) is also within the expected angle given the literature values available (for Ph$_2$AsCH$_2$AsPh$_2$ the angle about bridging methylene 113.33°) [20–22]. The more sterically bulky



Scheme 1.

$-N(SiMe_3)_2$ groups in **4** (Fig. 1d) contribute significantly to a distorted pyramidal geometry around the arsenic atoms (angles about arsenic centres, As(1) 98.7(3)°–107.0(3), As(2) 100.0(3)°–107.1(3)°) and an expected trigonal planar arrangement around the sp^2 nitrogen atoms. Most significantly though is the angle of 107.5(3)° about the bridging methylene group. Unlike the other methylene bridged examples both here and in the literature, the angle is smaller than the uncoordinated or monodentate complexes published. Several transition metal complexes incorporating bidentate chelation of the $As-CH_2-As$ moiety possess a strained geometry (90.4°–97.1°) [23–25], however, this is to be expected given the geometrical constraints of the four membered rings formed. Since **4** is a free ligand this restricted geometry is almost certainly a result of steric effects due to the arrangement of the ligands around the arsenic centres. For compound **5** another type of structural arrangement is observed, involving partial π-stacking of the aromatic pyridyl groups. The molecule can be split into two with regards to the geometry. The angles about As(2) (S(3)–As(2)–S(4) 91.04(6), S(3)–As(2)–C(21) 97.31(13), S(4)–As(2)–C(21) 92.88(14)°) are consistent with those seen in the literature and in **2** and **3**. The angle of the methylene bridge (As(1)–C(21)–As(2) 115.9°) is also consistent with a non-sterically hindered methylene arsine compound [20]. However, the geometry is significantly different around As(1). Here the angles are no longer consistent with a traditional pyramidal geometry, in particular the S(2)–As(1)–S(1) angle of 85.19° is particularly small compared to the other angles. This distortion is a result of intermolecular π–π stacking between the pyridyl groups on one end of two separate diarsine molecules. This in effect forms a pseudo-dimeric structure (Fig. 1e) since only one half of each diarsine is involved in π–π stacking. The effect of π-stacking is common place in organic structures and it is widely recognised that the centroid–centroid distance between the rings must be within the range 3.3–3.8 Å [26]. Here the centroid–centroid distance is 3.855 Å, which although at the upper limit, does represent a realistic π–π interaction especially when related to the observed distortions. It is intended to use the potential selective reactivities of these substituted arsine and stibine ligands to generate a host of mixed-metal compounds incorporating s-/d- and p-block metals.

4. Supplementary material

Atomic coordinates, bond lengths and angles, and thermal parameters have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 192078, 192077, 192080, 192081 and 192079 for compounds **1**, **2**, **3**, **4** and **5**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

We wish to acknowledge the EPSRC for project studentship (J.S.J) and the provision of crystallographic equipment.

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