Crystal Structures of Octaphenylcyclotetraphosphazene and Octaphenylcyclotetra-arsazene

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The crystal structures of octaphenylcyclotetraphosphazene ($Ph_8P_4N_4$) (I) and of the arsenic analogue ($Ph_8As_4N_4$) (II) have been determined from diffractometer data by Patterson and Fourier methods and refined by least squares to R 0·046 [(I), 1000 observed reflexions] and 0·058 (II), 588 observed reflexions] respectively. The compounds are isostructural and have S_4 molecular symmetry with the ring conformation intermediate between the ideal boat (S_4) and the ideal saddle (D_{2d}) forms. The ring bonds for both compounds are equal and in each case the ring angle at nitrogen is small. The value for (II) (121·4°) is particularly low and may indicate a smaller degree of arsenic 4*d* orbital participation in the bonding than 3*d* orbital participation in (I). Crystals of both compounds are tetragonal, space group $P\bar{4}2_1c$, Z = 2 with unit cell dimensions: (I) a = 14·688(2), c = 9·755(2) Å; (II) a = 14·977(3), c = 9·708(3) Å.

SINGLE-CRYSTAL *X*-ray structures of a number of homogeneously substituted tetraphosphazenes, $P_4N_4X_8$, can be rationalized by relating the resulting ring conformation to the electronic requirements of the exocyclic groups, X. The influence of steric factors is not readily apparent in such compounds but their importance is shown by the results of structure determinations on non-geminally tetra-substituted compounds, $P_4N_4X_4Y_4$, where it appears that steric requirements can influence the resulting ring conformation to an extent comparable to that observed for electronic effects. The effect is shown in the structures adopted by two non-geminal isomers of composition $P_4N_4F_4(NMe_2)_4$ [1,2] and particularly in the distorted 'crown' conformation adopted by the ring in the *cis*-non geminal-$P_4N_4Ph_4Cl_4$ isomer.[3] The structure of the octaphenyl compound, $P_4N_4Ph_8$ (I) is of interest in this connection, and the structure has been determined. The analogous arsenic compound is known but since preliminary *X*-ray data[4] seemed to indicate different As–N bond lengths, which is unexpected, a re-examination of the structure was considered desirable.

EXPERIMENTAL

Preparation of $P_4N_4Ph_8$.—Iminobis(aminodiphenylphosphorus) chloride[5] $(Ph_2PNH_2)_2NCl$ (3·0 g) was heated at 250 °C for 2 h and extracted with hot benzene to separate the phosphazene from ammonium chloride. On cooling white needles of $P_4N_4Ph_8$ were obtained (1·9 g, 75%), m.p. 324 °C (lit.,[5] 326—328 °C) (Found: C, 71·7; H, 4·9; N, 6·8. Calc. for $P_4N_4C_{48}H_{40}$: C, 72·4; H, 5·1; N, 7·0%).

Preparation of $As_4N_4Ph_8$.—*Method* (i). Chlorodiphenylarsine, obtained by redistribution of a mixture of triphenylarsine and arsenic trichloride, was chlorinated at −78 °C in dichloromethane solution giving Ph_2AsCl_3 which was recrystallized from toluene, m.p. 173 °C (lit.,[6] 172 °C). Dried (sodium) ammonia gas (1·16 g, 68·1 mmol) was passed into a solution of Ph_2AsCl_3 (5·71 g, 17·0 mmol) in chloroform (100 ml) at 20 °C, and the solution filtered to remove precipitated ammonium chloride (2·73 g, 50·5 mmol). After evaporation of the chloroform the residue was recrystallized from toluene giving $As_4N_4Ph_8$, m.p. 319—320 °C (lit.,[7] 316—317 °C) (Found: C, 58·9; H, 4·8; N, 5·0. Calc. for $As_4N_4C_{48}H_{40}$: C, 59·2; H, 4·1; N, 5·8%).

Method (ii). Ph_2AsCl_3 (7·1 g, 21·0 mmol) in dichloromethane (100 ml) was added dropwise to a large excess of liquid ammonia at −78 °C and stirred for 3 h. When the solution was set aside the ammonia evaporated. Ammonium chloride was filtered off, the dichloromethane was removed, and the residue recrystallized from toluene (yield 3·2 g, 62%). A small quantity (0·3 g) of the trimer, $As_3N_3Ph_6$ (m.p. 155—156 °C, lit.,[6] 155—157 °C) was also obtained from the mother liquor.

I.r. spectra of both arsenic compounds show a strong band at 940 cm^{-1} attributed to As–N–As ring vibrations.[6,7]

Crystal Structure of (I) ($P_4N_4Ph_8$)

Crystal Data.—$C_{48}H_{40}N_4P_4$, M = 796·5, Tetragonal, a = 14·688(2), c = 9·755(2) Å, U = 2104·5 Å^3, Z = 2, D_c = 1·256 g ml^{-1}, $F(000)$ = 832, space group $P\bar{4}2_1c$ from systematic absences: $h00$ ($h = 2n + 1$) and hhl ($l = 2n + 1$). Mo-K_α radiation, λ = 0·7107 Å; μ(Mo-K_α) = 0·76 cm^{-1}. Crystal size *ca.* 0·6 × 0·3 × 0·3 mm.

Unit-cell parameters were determined initially from oscillation and Weissenberg photographs, and later refined on a Hilger and Watts four-circle diffractometer by use of monochromatic Mo-K_α radiation. Intensities were corrected for Lorentz and polarization effects but not for absorption or secondary extinction.

A total of 3371 reflexions in the range $0° \leqslant \theta \leqslant 30°$ was measured but, on account of the space group, all reflexions are duplicated ($hkl \equiv khl$). Intensities of such pairs of reflexions were averaged and in each case that with the larger h value was removed from the file. This now contained 1739 reflexions of which 1000 having $I > 3\sigma(I)$ were considered observed. Data reduction and subsequent calculations used the *X*-ray '70 programs;[8a] atomic scattering factors were taken from ref. 8(*b*).

The asymmetric unit consists of one quarter of a molecule and the position of the phosphorus atom was readily deduced from a three-dimensional Patterson map. After a structure-factor calculation a three-dimensional Fourier synthesis phased on this atom revealed the positions of the nitrogen and twelve carbon atoms. Because at this stage the phosphorus atom occupies a special position the

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[1] D. Millington, T. J. King, and D. B. Sowerby, *J.C.S. Dalton*, 1973, 396.

[2] M. J. Begley, D. Millington, T. J. King, and D. B. Sowerby, *J.C.S. Dalton*, 1974, 1162.

[3] G. J. Bullen and P. A. Tucker, *J.C.S. Dalton*, 1972, 1651.

[4] M. D. Glick, Ph.D. Thesis, University of Wisconsin, 1965.

[5] I. I. Bezman and J. H. Smalley, *Chem. and Ind.*, 1960, 839.

[6] H. H. Sisler and C. Stratton, *Inorg. Chem.*, 1966, **5**, 2003.

[7] W. T. Reichle, *Tetrahedron Letters*, 1962, 51.

[8] (*a*) '*X*-Ray '67,' eds. J. M. Stewart, F. A. Kindell, and J. C. Baldwin, University of Maryland Technical Report TR 67–58, 1967, revised 1970; (*b*) 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1965.

Fourier map also contains peaks associated with a centrosymmetrically related Ph_2PN unit. Four cycles of full-matrix least-squares refinement with isotropic temperature factors reduced R to 0·126 and with anisotropic temperature factors R converged at 0·074 after four more cycles of refinement. A weighting scheme: $w = \{1 + [(F_0 - 7{\cdot}0)/10{\cdot}1]^2\}^{-1}$ was introduced and four further cycles of least-squares refinement reduced R to 0·065. Modification of the data by removal of a small number of reflexions which had low net counts (as a result of low background counts) and two cycles of refinement gave R 0·059. A difference Fourier then revealed possible positions for the hydrogen atoms and, after four further cycles of refinement with the ten hydrogen atoms included with isotropic temperature factors, final convergence was achieved with R 0·046.

TABLE 1

Fractional co-ordinates (× 10^4), with estimated standard deviations in parentheses, for non-hydrogen atoms in (I)

	x/a	y/b	z/c
P(1)	−29(0)	1346(1)	299(1)
N(1)	−919(2)	769(2)	603(3)
C(1)	386(2)	1697(2)	1962(4)
C(2)	1165(3)	2228(3)	2050(6)
C(3)	1506(4)	2474(3)	3325(7)
C(4)	1097(3)	2202(3)	4499(6)
C(5)	320(3)	1675(3)	4425(5)
C(6)	−16(3)	1423(2)	3164(4)
C(11)	−337(2)	2393(2)	−564(4)
C(12)	−1052(3)	2905(3)	−55(6)
C(13)	−1299(4)	3704(4)	−716(9)
C(14)	−862(5)	3975(4)	−1869(8)
C(15)	−170(5)	3473(4)	−2377(6)
C(16)	97(3)	2670(3)	−1727(5)

TABLE 2

Anisotropic thermal parameters [a] (× 10^4), with estimated standard deviations in parentheses, for non-hydrogen atoms in (I)

	U_{11}	U_{22}	U_{23}	U_{12}	U_{13}	U_{23}
P(1)	546(4)	497(4)	831(5)	2(4)	29(5)	0(3)
N(1)	603(12)	629(13)	843(18)	−25(12)	57(14)	128(14)
C(1)	618(16)	538(15)	869(22)	−53(14)	−9(17)	−12(16)
C(2)	972(29)	1002(29)	1042(33)	−364(25)	−93(25)	87(25)
C(3)	1138(34)	1124(34)	1257(45)	−511(30)	−317(35)	95(34)
C(4)	1042(31)	880(26)	1134(38)	−83(25)	−301(30)	−7(27)
C(5)	952(27)	954(27)	964(31)	−16(23)	−73(23)	48(24)
C(6)	711(18)	792(19)	883(21)	−48(25)	−101(27)	−39(19)
C(11)	638(16)	580(16)	941(25)	15(14)	−17(18)	7(18)
C(12)	1045(29)	821(24)	1346(45)	326(23)	76(31)	3(28)
C(13)	1387(47)	935(33)	1754(54)	536(30)	−122(47)	14(39)
C(14)	1690(57)	865(31)	1564(52)	407(35)	−325(51)	277(37)
C(15)	1673(55)	1113(35)	1450(45)	328(39)	90(48)	530(35)
C(16)	985(27)	847(24)	1130(31)	162(22)	104(32)	253(24)

[a] In the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

TABLE 3

Fractional co-ordinates of hydrogen atoms (× 10^3) and isotropic thermal parameters (× 10^3), with estimated standard deviations in parentheses, for (I)

	x/a	y/b	z/c	U/Å^2
H(2)	148(4)	236(4)	128(7)	110(17)
H(3)	204(4)	290(5)	340(7)	148(22)
H(4)	133(4)	238(4)	544(7)	130(22)
H(5)	10(4)	149(4)	518(5)	106(19)
H(6)	−58(3)	105(3)	309(6)	99(16)
H(12)	−128(3)	276(3)	97(6)	92(17)
H(13)	−174(5)	406(5)	−37(7)	148(25)
H(14)	−102(5)	457(5)	−24(7)	170(33)
H(15)	21(5)	368(5)	−320(8)	170(26)
H(16)	61(3)	244(3)	−194(4)	71(12)

Crystal Structure of (II) ($As_4N_4Ph_8$)

Crystal Data.—$C_{48}H_{40}As_4N_4$, $M = 972{\cdot}2$, Tetragonal, $a = 14{\cdot}977(3)$, $c = 9{\cdot}708(3)$ Å, $U = 2177{\cdot}6$ Å^3, $Z = 2$, $D_c = 1{\cdot}482$ g ml^{-1}, $F(000) = 976$. Space group $P\bar{4}2_1c$ from systematic absences: $h00$ ($h = 2n + 1$) and hhl ($l = 2n + 1$). Mo-K_α radiation, μ(Mo-K_α) = 3·00 cm^{-1}. A crystal, dimensions *ca.* 0·6 × 0·25 × 0·25 mm, was sealed in a silica capillary.

Oscillation and Weissenberg photographs clearly indicated that (I) and (II) were isostructural. Intensities were measured initially for 2217 reflexions but after averaging the hkl and khl reflexions a total of 1101 remained, of which 588 having $I > 2{\cdot}5\sigma(I)$ were considered observed. The position of the arsenic atom was obtained from a Patterson map and subsequent solution of the structure was as described for (I). The R value converged at 0·103 after four cycles of full-matrix least-squares refinement with isotropic temperature factors and at 0·058 after six further cycles of refinement with anisotropic temperature factors. A final difference Fourier showed no peaks >0·4 eÅ^{-3} but a number of positions could probably be associated with hydrogen atoms. The data-to-parameter ratio, however, was considered too low to allow inclusion of the hydrogen atoms in the final refinement.

Observed and calculated structure factors for both compounds are listed in Supplementary Publication No. SUP 21116 (6 pp., 1 microfiche).* Final atomic co-

TABLE 4

Fractional co-ordinates (× 10^3), with estimated standard deviations in parentheses, for non-hydrogen atoms in (II)

	x/a	y/b	z/c
As(1)	−3(0)	139(0)	34(0)
N(1)	−97(1)	76(1)	77(1)
C(1)	44(1)	171(1)	210(3)
C(2)	119(2)	229(2)	216(3)
C(3)	153(2)	247(2)	341(4)
C(4)	116(2)	221(2)	460(4)
C(5)	36(2)	168(2)	460(3)
C(6)	7(2)	142(2)	331(2)
C(11)	−38(1)	247(2)	−56(3)
C(12)	−109(2)	293(2)	3(4)
C(13)	−142(3)	371(3)	−74(5)
C(14)	−90(3)	40(3)	−178(6)
C(15)	−18(3)	360(2)	−221(4)
C(16)	4(3)	276(2)	−169(3)

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue (items less than 10 pp. are supplied as full-size copies).

TABLE 5

Anisotropic thermal parameters[a] ($\times 10^3$), with estimated standard deviations in parentheses, for non-hydrogen atoms in (II)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{12}	U_{23}
As(1)	64(1)	60(1)	86(1)	4(1)	−2(3)	−2(1)
N(1)	85(13)	69(11)	83(14)	10(10)	11(11)	−11(11)
C(1)	61(14)	61(15)	134(22)	−25(11)	12(16)	7(15)
C(2)	124(25)	135(28)	110(28)	−66(22)	7(20)	−7(22)
C(3)	101(24)	136(28)	131(30)	−19(20)	−11(24)	−24(27)
C(4)	106(27)	123(24)	116(24)	48(20)	−78(22)	−30(25)
C(5)	105(24)	90(19)	128(22)	15(13)	−20(21)	−8(20)
C(6)	78(18)	120(20)	81(13)	−45(20)	54(21)	−12(15)
C(11)	77(14)	71(15)	117(21)	21(12)	−1(17)	−32(18)
C(12)	73(15)	79(16)	197(36)	24(13)	−64(21)	−19(21)
C(13)	143(37)	148(37)	188(44)	82(28)	−5(31)	−18(32)
C(14)	161(51)	120(35)	264(70)	17(28)	−73(46)	5(41)
C(15)	178(42)	145(30)	128(27)	40(31)	73(33)	55(23)
C(16)	129(26)	115(22)	131(22)	−49(29)	26(40)	24(19)

[a] See footnote to Table 2.

ordinates and thermal parameters for the heavier atoms in (I) are listed in Tables 1 and 2, and for the hydrogen atoms in Table 3. Atomic co-ordinates and anisotropic thermal parameters for the arsenic compound are listed in Tables 4 and 5. The numbering of the atoms is shown in Figure 1.

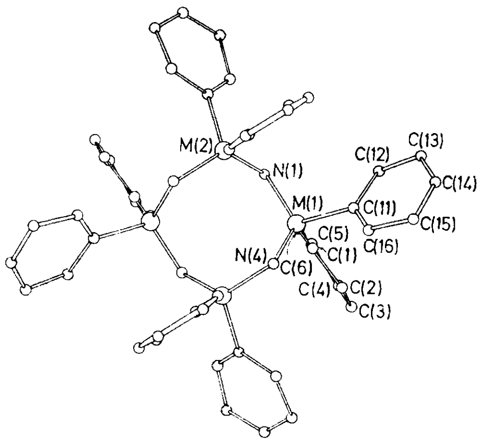

FIGURE 1 The numbering of atoms [(I) M = P, (II) M = As]

RESULTS AND DISCUSSION

Bond distances and angles are summarized in Tables 6 (I) and 7 (II), and Figure 2 shows the ring conformation and orientation of the phenyl groups.

Compound (I). Although the molecule has overall S_4 symmetry, the ring conformation is intermediate between the ideal boat (S_4) and the ideal saddle (D_{2d}) forms. The deviations toward the latter can be assessed by the magnitude of $\Delta P/\Delta N$, where ΔP is the displacement of the phosphorus atoms and ΔN of the nitrogen atoms from the mean ring plane.[9] The value here is 0·292/0·592, *i.e.* 0·49, and should be compared with values of 0·75 for the *K*-form of $P_4N_4Cl_8$ (closer to S_4 symmetry) and 0·35 for $P_4N_4(NMe_2)_8$ (closer to D_{2d} symmetry). Deviation from perfect D_{2d} symmetry can also be shown by the dihedral angles of the ring bonds P(1)–N(1) 25·9, and N(1)–P(2) 22·0°. The ring conformation is in fact very similar to that of $P_4N_4Me_8$.[10]

The ring angles are 119·8 at phosphorus and 127·8° at nitrogen, the latter being significantly smaller than for other tetrameric phosphonitriles.[11] The P–N bonds are equal (1·590 Å) and slightly shorter than the mean (1·597 Å) in the analogous trimeric compound.[12] Surprisingly, the octamethyl compound, to which (I) bears a strong similarity, has a P–N bond distance of 1·60 Å, while the angle at nitrogen is 132·0°; the inter-relationship between the P–N bond length, the ring angle at nitrogen, and the electronegativity of the substituent at phosphorus is thus more complex than expected.

The P–C bonds are equal and slightly longer than in the trimer. The equations for the mean planes (in orthogonal Ångstrom space) through the two phenyl groups are:

$$\text{C(1)—6):} \quad -0{\cdot}566x' + 0{\cdot}824y' + 0{\cdot}013z' = 1{\cdot}701$$

$$\text{C(11)—(16):} \quad 0{\cdot}648x' + 0{\cdot}519y' + 0{\cdot}557z' = 1{\cdot}192$$

and the maximum deviation of any atom from the respective plane is 0·01 Å. The C–C bonds vary between 1·34 and 1·39 Å (mean 1·37 Å) while the C–C–C angles range between 118·0 and 121·9° (mean 120·0°). The mean C–H bond distance is 0·97 Å.

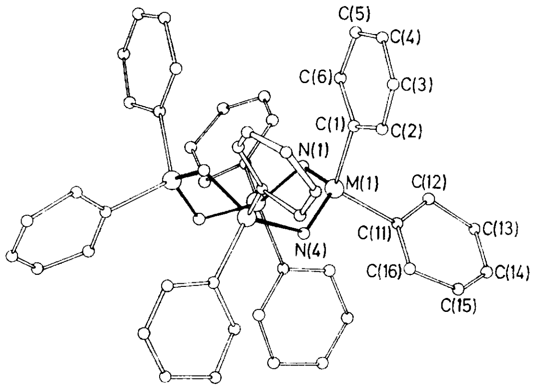

FIGURE 2 The ring conformation [(I) M = P, (II) M = As]

The P–C bonds are not orientated symmetrically with respect to the local P–N segment and the angle between the C(1),P(1),C(11) plane and the N(1),P(1),N(4) plane is 86·3°. Following from this there are also two sets of

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[9] N. L. Paddock, *Quart. Rev.*, 1964, **18**, 168.

[10] M. W. Dougill, *J. Chem. Soc.*, 1961, 5471.

[11] H. R. Allcock, *Chem. Rev.*, 1972, **72**, 315.

[12] F. R. Ahmed, P. Singh, and W. H. Barnes, *Acta Cryst.*, 1969, **B25**, 316.

N–P–C angles (*ca.* 111 and 105°). The dihedral angles between the two phenyl groups and the mean ring plane are 89·2 and 56·1° respectively for C(1)—(6) and C(11)—(16), but with respect to the plane of the local N–P–N segment the rings are symmetrically oriented with dihedral angles of 56·4 and 56·7° respectively. The planes of the two phenyl groups are almost normal to each other (dihedral angle 86·9°), thus ensuring minimum intramolecular interactions as, following the symmetry of the molecule, the relative orientations of the phenyl groups will be reversed at adjacent phosphorus atoms. Similar arrangements in the Ph_2P group are found in $P_3N_3Ph_6$ [12] and in the geminal di- [13] and tetra-chlorides,[14] but in the trimeric diphenyl tetrafluoride [15] the groups are eclipsed.

Compound (II).—Although there are considerably less reflexions for the arsenic compound, it is clearly isostructural with the phosphorus compound, thus confirming the overall structure suggested previously.[4] In the present investigation however, the data-to-parameter ratio is much more favourable and it has been possible to refine the structure further.

The deviations of the arsenic and nitrogen atoms from the mean ring plane are 0·334 and 0·750 Å, giving ΔAs/ΔN 0·445, and the overall ring conformation is almost identical to that already discussed for (I). This is confirmed by the dihedral angles of the As(1)–N(1) (28·4°) and N(1)–As(2) (27·9°) bonds. However, unlike results from the earlier determination, the two independent As–N distances are equal within experimental error (mean 1·73 Å) and shorter than the single-bond distance (1·87 Å).[16] Paralleling the situation in the phosphorus series, the ring bonds in the tetramer are shorter than those in the corresponding trimeric compound (1·76 Å).[16]

The orientation of the phenyl groups is similar to that in (I) and there are again two sets of N–As–C angles; the As–C distances are as expected. The phenyl groups are planar, no atom deviating by >0·07 Å from the respective plane.

Although the structures of (I) and (II) are closely similar, there are details which are significantly different. The most important of these is the smallness (121·4°) in (II) of the angle at the ring nitrogen atoms, indicating that hybridization is very close to sp^2. The nitrogen lone-pair is thus more completely localized than in (II) and any in-plane (π_s) component to the As–N bond will be very small. However, no other firm conclusions can be drawn about the nature of the As–N bond, until there are structure determinations of other cycloarsazenes, particularly those with more highly electronegative substituents. In general terms, some degree of π bonding is required by valence considerations and 4*d* orbital participation is to be expected. The general diffuseness of these orbitals is likely to make π interaction with nitrogen p_z orbitals less efficient than when phosphorus 3*d* orbitals are involved, and bond shortening over the single-bond distance for the arsenic compound will probably be comparatively small.

TABLE 6

Bond distances (Å) * and angles (°) for (I)

P(1)–N(1)	1·586(3)	N(1)–P(1)–C(1)	105·3(2)
P(1)–N(4)	1·594(3)	N(1)–P(1)–C(11)	109·6(2)
P(1)–C(1)	1·809(4)	N(4)–P(1)–C(1)	110·0(2)
P(1)–C(11)	1·810(3)	N(4)–P(1)–C(11)	105·1(2)
N(1)–P(1)–N(4)	119·9(1)	C(1)–P(1)–C(11)	105·1(2)
P(1)–N(1)–P(2)	127·9(2)		
C(1)–C(2)	1·387(6)	C(11)–C(12)	1·384(6)
C(2)–C(3)	1·388(8)	C(12)–C(13)	1·387(8)
C(3)–C(4)	1·353(8)	C(13)–C(14)	1·355(11)
C(4)–C(5)	1·381(7)	C(14)–C(15)	1·351(10)
C(5)–C(6)	1·376(6)	C(15)–C(16)	1·394(8)
C(6)–C(1)	1·373(5)	C(16)–C(1)	1·364(6)
			(Mean 1·374)
P(1)–C(1)–C(2)	119·6(3)	P(1)–C(11)–C(12)	119·0(3)
P(1)–C(1)–C(6)	122·5(3)	P(1)–C(11)–C(16)	121·6(3)
C(6)–C(1)–C(2)	117·8(4)	C(16)–C(11)–C(12)	119·4(4)
C(1)–C(2)–C(3)	120·0(5)	C(11)–C(12)–C(13)	119·5(5)
C(2)–C(3)–C(4)	121·4(5)	C(12)–C(13)–C(14)	120·7(6)
C(3)–C(4)–C(5)	119·1(5)	C(13)–C(14)–C(15)	120·0(6)
C(4)–C(5)–C(6)	119·6(5)	C(14)–C(15)–C(16)	120·4(6)
C(5)–C(6)–C(1)	122·0(4)	C(15)–C(16)–C(11)	120·0(5)
			(Mean 120·0)

* C–H 0·85—1·08 Å, mean 0·97 Å.

TABLE 7

Bond distances (Å) and angles (°) for (II)

As(1)–N(1)	1·739(17)	N(1)–As(1)–C(1)	103·0(9)
As(1)–N(4)	1·727(17)	N(1)–As(1)–C(11)	110·9(9)
As(1)–C(1)	1·907(25)	N(4)–As(1)–C(1)	113·4(9)
As(1)–C(11)	1·905(25)	N(4)–As(1)–C(11)	101·9(10)
N(1)–As(1)–N(4)	119·9(8)	C(1)–As(1)–C(11)	107·4(10)
As(1)–N(1)–As(2)	121·4(9)		
C(1)–C(2)	1·41(4)	C(11)–C(12)	1·39(4)
C(2)–C(3)	1·34(5)	C(12)–C(13)	1·47(5)
C(3)–C(4)	1·36(5)	C(13)–C(14)	1·34(7)
C(4)–C(5)	1·44(4)	C(14)–C(15)	1·30(7)
C(5)–C(6)	1·39(4)	C(15)–C(16)	1·40(5)
C(6)–C(1)	1·38(3)	C(16)–C(11)	1·33(4)
			(Mean 1·38)
As(1)–C(1)–C(2)	119(2)	As(1)–C(11)–C(12)	116(2)
As(1)–C(1)–C(6)	122(2)	As(1)–C(11)–C(16)	122(2)
C(6)–C(1)–C(2)	119(2)	C(16)–C(11)–C(12)	122(3)
C(1)–C(2)–C(3)	118(3)	C(11)–C(12)–C(13)	116(3)
C(2)–C(3)–C(4)	124(3)	C(12)–C(13)–C(14)	116(4)
C(3)–C(4)–C(5)	120(3)	C(13)–C(14)–C(15)	125(4)
C(4)–C(5)–C(6)	115(3)	C(14)–C(15)–C(16)	120(4)
C(5)–C(6)–C(1)	123(3)	C(15)–C(16)–C(11)	119(3)
			(Mean 120)

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