

The molecule shows significant deviations from ideal threefold symmetry. The same appears to apply to DPT and PAT. In Table 4 the angles between the benzene rings and the deviations of the bridgehead atoms from the benzene rings are listed for TBPT, DPT and PAT.

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## 9,10-Dihydro-10-phenyl-9,10-*o*-benzeno-9-arsanthracene

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**Abstract.** C<sub>25</sub>H<sub>17</sub>As, monoclinic,  $P2_1/n$ ,  $a = 8.8392$  (4),  $b = 20.477$  (1),  $c = 10.082$  (1) Å,  $\beta = 102.04$  (1)°,  $Z = 4$ . Final  $R$  for 2799 independent reflexions 0.025. The As–C distances are 1.938 (3), 1.952 (3) and 1.955 (2) Å. The average C–As–C angle is 91.5°. The benzene rings are slightly folded as a result of intramolecular steric interaction.

**Introduction.** The investigation of the title compound, a derivative of arsatriptycene, was undertaken to obtain information about the geometry around the As atom. The crystal structure determination of arsatriptycene itself (Freijee & Stam, 1980) did not yield reliable results because of orientational disorder. The disorder is such that a fraction of the molecular sites in the ideal ordered structure are occupied by molecules rotated by 180° about a quasi-twofold axis perpendicular to the As–C axis of the molecules. In the title compound the quasi-twofold axes are suppressed by the substituent so that the same kind of disorder becomes impossible.

The title compound was prepared by Klebach & Bickelhaupt (1980). 2799 independent reflexions with  $I > 2\sigma(I)$  were collected on a Nonius CAD-4 diffractometer with graphite-monochromatized Cu  $K\alpha$  radiation. No absorption correction was applied (crystal dimensions 0.2 × 0.2 × 0.4 mm;  $\mu = 2.59$  mm<sup>-1</sup>). The structure was solved directly from an ( $E^2 - 1$ ) Patterson synthesis. Block-diagonal least-squares refinement, anisotropic for As and C, isotropic for H, resulted in  $R = 0.025$ . The H atoms were located in a  $\Delta F$  synthesis.

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### References

- DALY, J. J. (1964). *J. Chem. Soc.* pp. 3799–3810.  
 FREIJEE, F. J. M. & STAM, C. H. (1980). *Acta Cryst.* **B36**, 1247–1249.  
 OVERBEEK, A. R. & SCHENK, H. (1978). *Crystallographic Computing*, pp. 108–112. Delft Univ. Press.  
 SCHOMBURG, D. & SHELDRIK, W. S. (1975). *Acta Cryst.* **B31**, 2427–2431.  
 SCHOMBURG, D. & SHELDRIK, W. S. (1976). *Acta Cryst.* **B32**, 1021–1024.

Anomalous dispersion of As was taken into account and a weighting scheme  $w = 1/(4.0 + F_o + 0.015F_o^2)$  was used. An extinction correction

$$F_c^{\text{corr}} = F_c \left( 1 + g \frac{1 + \cos^4 2\theta}{1 + \cos^2 2\theta} \cdot \frac{1}{\sin 2\theta} \cdot F_c^2 \right)^{-1/4}$$

(Larson, 1969) was included resulting in  $g = 6.7 \times 10^{-6}$ . The final coordinates are listed in Table 1.†

**Discussion.** The atomic numbering and the shape of the molecule are indicated in Fig. 1. The bond distances and angles are given in Table 2. The three As–C bonds are not equal, As–C(1) [1.938 (3) Å] differing significantly from the other two [1.952 (3) and 1.955 (2) Å]. We have no explanation for this discrepancy, but it is interesting to note that the differences conform to the overall symmetry of the molecule (Fig. 1). The rather large differences between the three C–As–C angles reflect the flexibility of the rings with respect to the bridgehead atoms As and C(19) (see below). There is good agreement between corresponding bond lengths and angles in the three rings of the arsatriptycene moiety. The bond lengths show a significant trend: the inner bonds [average 1.430 (2) Å] are longer and the outer bonds [average 1.377 (2) Å] are shorter than the overall average of 1.390 Å.

† Lists of structure factors, anisotropic thermal parameters for the non-hydrogen atoms and isotropic for the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35043 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ( $\times 10^5$  for As,  $\times 10^4$  for C,  $\times 10^3$  for H)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
As	7707 (3)	4662 (1)	32706 (3)	2.9
C(1)	-10 (3)	1354 (2)	3068 (2)	2.5
C(2)	1089 (2)	1848 (1)	3062 (2)	2.2
C(3)	562 (3)	2493 (2)	2992 (2)	2.7
C(4)	-998 (3)	2636 (1)	2897 (3)	3.2
C(5)	-2061 (3)	2143 (2)	2867 (3)	3.4
C(6)	-1565 (3)	1499 (2)	2960 (3)	3.2
C(7)	1942 (3)	605 (1)	1854 (3)	2.5
C(8)	2884 (3)	1164 (1)	1948 (2)	2.1
C(9)	3912 (3)	1204 (1)	1071 (3)	2.7
C(10)	3888 (3)	744 (1)	59 (3)	3.5
C(11)	2879 (4)	222 (1)	-85 (3)	3.8
C(12)	1942 (3)	143 (1)	839 (3)	3.3
C(13)	2582 (3)	709 (1)	4653 (2)	2.6
C(14)	3467 (3)	1244 (1)	4386 (2)	2.2
C(15)	4954 (3)	1311 (2)	5183 (3)	2.6
C(16)	5485 (3)	888 (1)	6257 (3)	3.2
C(17)	4565 (3)	390 (2)	6561 (3)	3.3
C(18)	3119 (3)	296 (1)	5741 (3)	3.2
C(19)	2815 (2)	1663 (1)	3106 (2)	2.0
C(20)	3669 (3)	2309 (1)	3003 (2)	2.1
C(21)	3647 (3)	2603 (1)	1751 (2)	2.5
C(22)	4349 (3)	3207 (1)	1662 (3)	3.4
C(23)	5017 (3)	3540 (1)	2819 (3)	3.8
C(24)	4942 (3)	3285 (1)	4060 (3)	3.6
C(25)	4268 (3)	2676 (2)	4159 (2)	2.8
H(3)	126 (3)	285 (1)	300 (3)	
H(4)	-134 (3)	308 (2)	284 (3)	
H(5)	-311 (3)	225 (1)	277 (3)	
H(6)	-233 (3)	114 (2)	297 (3)	
H(9)	461 (3)	155 (1)	112 (3)	
H(10)	456 (3)	80 (2)	-54 (3)	
H(11)	280 (3)	-9 (1)	-84 (3)	
H(12)	133 (3)	-23 (2)	81 (3)	
H(15)	562 (3)	165 (1)	496 (3)	
H(16)	653 (3)	94 (1)	681 (3)	
H(17)	487 (3)	10 (2)	731 (3)	
H(18)	247 (3)	-5 (1)	588 (3)	
H(21)	313 (3)	240 (2)	90 (3)	
H(22)	436 (3)	338 (2)	70 (3)	
H(23)	553 (3)	394 (2)	276 (3)	
H(24)	535 (3)	351 (1)	488 (3)	
H(25)	418 (3)	251 (1)	505 (3)	

Table 2. Bond distances (Å) and angles (°)

As-C(1)	1.938 (3)	C(11)-C(12)	1.379 (4)
As-C(7)	1.952 (3)	C(13)-C(14)	1.403 (3)
As-C(13)	1.955 (2)	C(13)-C(18)	1.387 (4)
C(1)-C(2)	1.402 (3)	C(14)-C(15)	1.397 (3)
C(1)-C(6)	1.388 (3)	C(14)-C(19)	1.556 (3)
C(2)-C(3)	1.396 (3)	C(15)-C(16)	1.389 (3)
C(2)-C(19)	1.564 (3)	C(16)-C(17)	1.377 (4)
C(3)-C(4)	1.393 (4)	C(17)-C(18)	1.383 (4)
C(4)-C(5)	1.374 (4)	C(19)-C(20)	1.536 (3)
C(5)-C(6)	1.385 (4)	C(20)-C(21)	1.395 (4)
C(7)-C(8)	1.406 (3)	C(20)-C(25)	1.394 (3)
C(7)-C(12)	1.393 (4)	C(21)-C(22)	1.393 (4)
C(8)-C(9)	1.396 (3)	C(22)-C(23)	1.373 (4)
C(8)-C(19)	1.561 (3)	C(23)-C(24)	1.370 (4)
C(9)-C(10)	1.385 (4)	C(24)-C(25)	1.393 (4)
C(10)-C(11)	1.379 (4)		
C(1)AsC(7)	91.0 (1)	AsC(13)C(18)	120.2 (2)
C(1)AsC(13)	93.3 (1)	C(14)C(13)C(18)	120.9 (2)
C(7)AsC(13)	90.2 (1)	C(13)C(14)C(15)	117.5 (2)
AsC(1)C(2)	116.3 (2)	C(13)C(14)C(19)	117.6 (2)
AsC(1)C(6)	122.2 (2)	C(15)C(14)C(19)	124.4 (2)
C(2)C(1)C(6)	121.4 (2)	C(14)C(15)C(16)	120.8 (2)
C(1)C(2)C(3)	117.2 (2)	C(15)C(16)C(17)	121.0 (2)
C(1)C(2)C(19)	119.9 (2)	C(16)C(17)C(18)	119.0 (2)
C(3)C(2)C(19)	122.9 (2)	C(13)C(18)C(17)	120.6 (2)
C(2)C(3)C(4)	121.1 (2)	C(2)C(19)C(8)	119.1 (2)
C(3)C(4)C(5)	120.7 (3)	C(2)C(19)C(14)	110.3 (2)
C(4)C(5)C(6)	119.4 (2)	C(2)C(19)C(20)	106.3 (2)
C(1)C(6)C(5)	120.2 (2)	C(8)C(19)C(14)	101.2 (2)
AsC(7)C(8)	117.8 (2)	C(8)C(19)C(20)	114.5 (2)
AsC(7)C(12)	121.4 (2)	C(14)C(19)C(20)	115.3 (2)
C(8)C(7)C(12)	120.7 (2)	C(19)C(20)C(21)	121.2 (2)
C(7)C(8)C(9)	117.4 (2)	C(19)C(20)C(25)	120.8 (2)
C(7)C(8)C(19)	117.8 (2)	C(21)C(20)C(25)	117.1 (2)
C(9)C(8)C(19)	124.6 (2)	C(20)C(21)C(22)	121.2 (2)
C(8)C(9)C(10)	121.0 (2)	C(21)C(22)C(23)	120.2 (3)
C(9)C(10)C(11)	120.9 (3)	C(22)C(23)C(24)	119.5 (3)
C(10)C(11)C(12)	119.1 (3)	C(23)C(24)C(25)	120.6 (3)
C(7)C(12)C(11)	120.6 (2)	C(20)C(25)C(24)	121.0 (2)
AsC(13)C(14)	117.9 (2)		

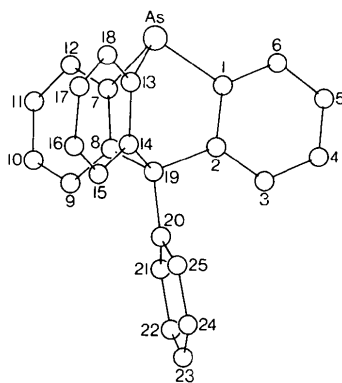


Fig. 1. Atomic numbering and shape of the molecule.

The four rings are not exactly planar, the maximum deviations being 0.012, 0.040, 0.030 and 0.039 Å for rings (I), (II), (III) and (IV) respectively [(I) = C(1)-C(6), (II) = C(7)-C(12), (III) = C(13)-C(18), (IV) = C(20)-C(25)]. All four rings appear to be folded: (I) only slightly along C(2)-C(5), (II) and (III) along corresponding lines C(8)-C(11) and C(14)-C(17) and (IV) along C(20)-C(23) (Table 3). The folding angles are 1.5, 5.1, 3.7 and 4.7° respectively. The deformations of (II), (III) and (IV) can be understood in terms of the steric interaction of (IV) with (II) and (III). (IV) is nearly perpendicular to (I) (angle between plane normals 87°). There is direct contact between H(21) of (IV) and H(9) of (II), and between H(25) of (IV) and H(15) of (III) (H...H distances 2.15 and 2.19 Å respectively). The interaction results in (IV) being pushed towards (I) [angle between the normal of (IV) and the As-C(19) axis 80°]. In the process (IV) is folded about C(10)-C(23). The lower parts of (II) and (III) are pushed the other

Table 3. Distances (Å) from various planes involving the ring atoms

Atoms defining the planes have been indicated by an asterisk. E.s.d.'s are  $3 \times 10^{-4}$  for As and  $4 \times 10^{-3}$  Å for C.

C(1)	0.010*	0.005*	0.037
C(2)	-0.012*	-0.002*	0.000*
C(3)	0.004*	0.029	-0.001*
C(4)	0.007*	0.033	0.001*
C(5)	-0.009*	0.002*	0.000*
C(6)	0.000*	-0.005*	0.025
As	0.092	0.064	0.141
C(19)	-0.079	-0.070	-0.064
C(7)	-0.020*	-0.003*	-0.107
C(8)	0.040*	0.001*	0.004*
C(9)	-0.026*	-0.114	-0.008*
C(10)	-0.010*	-0.094	0.008*
C(11)	0.013*	-0.001*	-0.004*
C(12)	-0.016*	0.003*	-0.103
As	-0.248	-0.160	-0.409
C(19)	0.044	0.004	0.002
C(13)	-0.022*	-0.009*	-0.089
C(14)	0.030*	0.004*	0.001*
C(15)	-0.013*	-0.077	-0.001*
C(16)	-0.013*	-0.078	0.001*
C(17)	0.022*	-0.005*	-0.001*
C(18)	-0.004*	0.009*	-0.067
As	-0.460	-0.391	-0.581
C(19)	-0.065	-0.086	-0.100
C(20)	-0.039*	-0.006*	-0.001*
C(21)	0.030*	0.013*	0.113
C(22)	0.003*	-0.013*	0.082
C(23)	-0.026*	0.007*	0.001*
C(24)	0.016*	0.097	-0.003*
C(25)	0.017*	0.098	0.003*
C(19)	0.071	0.104	0.114

way accounting for the folding about C(8)–C(11) and C(14)–C(17).

The As and C(19) atoms show considerable deviations from the rings to which they are attached

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## 9,10-Dihydro-9-methyl-9,10-*o*-benzeno-9-arsoniaanthracene Chloride Monohydrate

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**Abstract.**  $C_{20}H_{16}As^+ \cdot Cl^- \cdot H_2O$ , orthorhombic, *Pnca*,  $a = 15.388$  (7),  $b = 20.481$  (4),  $c = 11.388$  (2) Å,  $Z = 8$ . Final  $R$  for 1661 observed reflexions 0.038. Cl and  $H_2O$  are disordered. The average As–C<sub>ar</sub> distance is 1.905 (3) Å, the As–CH<sub>3</sub> length 1.883 (8) Å, the

(Table 3). The overall planes of rings (I), (II) and (III) make angles of 124.0 [(I)–(II)], 131.6 [(I)–(III)] and 104.0° [(II)–(III)]. The geometry of the arsatriptycene moiety thus deviates considerably from the ideal threefold symmetry to be expected for the free arsatriptycene molecule. From the crystal structures of arsa- and phosphatriptycene and derivatives (Freijee & Stam, 1980; Smit & Stam, 1980; van der Putten & Stam, 1980), it is obvious that these molecules are not rigid but behave as consisting of rigid rings flexibly connected to the bridgehead atoms. In triptycene itself (Neuman, 1970), significant deviations, of up to 0.043 Å, of the bridgehead atoms from the benzene planes occur, whilst the inter-ring angles are 117.1, 121.5 and 121.4°. Unsymmetrical packing is sufficient to cause deviations of 5° or more from the ideal inter-ring angles of 120° and deviations of 0.1 Å or more of the bridgehead atoms from the ring planes. In the title compound the overcrowding at C(19) is responsible for the additional deformation of the arsatriptycene moiety.

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### References

- FREIJEE, F. J. M. & STAM, C. H. (1980). *Acta Cryst.* B36, 1247–1249.  
 KLEBACH, TH. & BICKELHAUPT, F. (1980). To be published.  
 LARSON, A. C. (1969). *Crystallographic Computing*, p. 292. Copenhagen: Munksgaard.  
 NEUMAN, M. A. (1970). *Trans. Am. Crystallogr. Assoc.* 6, 111–128.  
 PUTTEN, N. VAN DER & STAM, C. H. (1980). *Acta Cryst.* B36, 1250–1252.  
 SMIT, F. & STAM, C. H. (1980). *Acta Cryst.* B36, 1254–1256.

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