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 3×10^{-4} for As and 4×10^{-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 (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.

The authors thank Professor F. Bickelhaupt and Dr T. C. Klebach for suggesting the investigation and providing the crystals and Mr D. Heijdenrijk for technical assistance.

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

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(*Received* 3 *December* 1979; *accepted* 22 *January* 1980)

Abstract. $C_{20}H_{16}As^+.Cl^-.H_2O$, orthorhombic, *Pnca*, $a = 15{\cdot}388$ (7), $b = 20{\cdot}481$ (4), $c = 11{\cdot}388$ (2) Å, $Z = 8$. Final R for 1661 observed reflexions 0·038. Cl and H_2O are disordered. The average $As—C_{ar}$ distance is 1·905 (3) Å, the $As—CH_3$ length 1·883 (8) Å, the average $C_{ar}—As—C_{ar}$ angle 97·7 (8)°, and the average $C_{ar}—As—CH_3$ angle 119·5 (21)°.

Introduction. This investigation was undertaken to obtain information about the geometry around the quarternary As atom. The title compound was prepared by Vermeer, Kevenaar & Bickelhaupt (1972).

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0567-7408/80/051254-03$01.00

1661 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 0·3 × 0·2 × 0·1 mm: $\mu = 4{\cdot}04$ mm^{-1}). The structure was derived by means of the heavy-atom technique. During the refinement a ΔF synthesis indicated that the assumed Cl position was electron deficient, whilst two unexplained, elongated maxima of heights 5 and 3·5 e Å^{-3} occurred. It appeared that the crystals contained one H_2O of crystallization which could account for one of the extra maxima. The Cl position being electron deficient, Cl was assumed to be distributed between the original position and the second extra maximum. Anisotropic least-squares refinement of the structure thus obtained resulted in $R = 0{\cdot}067$. The population parameters of the two partial Cl atoms were also refined and constrained to a total of 1. The resulting U_{ij} values for Cl and O had mostly become very large. A ΔF synthesis at this stage clearly indicated the H atoms, whilst around Cl and O positive areas of up to 0·6–1·0 e Å^{-3} occurred. In the next refinement cycles the H atoms were introduced with fixed parameters and each of the two partial Cl atoms and the O atom was split into two equal parts. This reduced R to 0·050 but did not improve significantly either the temperature parameters of Cl and O or the difference density.

Since we were interested in the arsatriptycene moiety we have not pursued further the interpretation of the situation around the assumed Cl and O positions, but have instead tried to minimize the interference of this part of the structure with the arsatriptycene moiety by *ad hoc* adjustment of the Cl and O parameters. Dropping the constraints on the population parameters removed most of the difference density near Cl and O. In the final cycles the H atoms were refined isotropically. The final R was 0·038. The positional parameters are given in Table 1.* Clearly little value can be attached to the parameters for Cl and O.

Table 1. *Fractional atomic coordinates* (As $\times 10^5$, Cl, O *and* C $\times 10^4$) *and their e.s.d.'s*

	x	y	z	Population parameter	B_{eq} (Å^2)
As	23759 (4)	31256 (3)	57041 (5)		2·9
C(1)	1870 (4)	3925 (3)	6212 (5)		2·4
C(2)	1582 (4)	4296 (3)	5245 (5)		2·4
C(3)	1172 (4)	4880 (3)	5460 (5)		3·0
C(4)	1062 (5)	5099 (3)	6612 (6)		3·6
C(5)	1351 (4)	4731 (4)	7532 (6)		3·9
C(6)	1764 (4)	4139 (3)	7354 (5)		3·1
C(7)	1470 (4)	2865 (3)	4661 (5)		2·7
C(8)	1262 (4)	3378 (3)	3883 (5)		2·5
C(9)	653 (4)	3269 (3)	3019 (5)		3·1
C(10)	248 (4)	2663 (4)	2950 (6)		3·9
C(11)	450 (4)	2170 (4)	3725 (6)		4·0
C(12)	1065 (4)	2260 (3)	4598 (6)		3·5
C(13)	3153 (4)	3490 (3)	4579 (5)		2·4
C(14)	2711 (4)	3907 (3)	3828 (5)		2·1
C(15)	3165 (4)	4201 (3)	2911 (5)		2·8
C(16)	4053 (4)	4093 (3)	2802 (5)		3·4
C(17)	4484 (4)	3692 (3)	3576 (6)		3·2
C(18)	4026 (4)	3380 (3)	4474 (5)		3·0
C(19)	1737 (3)	4021 (3)	4023 (5)		2·1
C(20)	2761 (5)	2540 (4)	6868 (6)		4·9
Cl(11)	3306 (4)	1627 (4)	4507 (5)	0·38	
Cl(12)	3619 (4)	1611 (3)	4309 (6)	0·38	
Cl(21)	5223 (8)	643 (5)	4289 (7)	0·31	
Cl(22)	5104 (8)	694 (5)	4760 (7)	0·29	
O(1)	2198 (10)	589 (8)	5306 (11)	0·65	
O(2)	1834 (10)	669 (8)	5596 (10)	0·65	

Discussion. The atomic numbering and the shape of the molecule are indicated in Fig. 1. Bond distances and angles are listed in Table 2. The three As—C_{ar} distances agree within the limits of accuracy [average = 1·905 (3) Å] and differ significantly from the As—CH_3 distance of 1·883 (8) Å. The mean C_{ar}—As—C_{ar} angle is 97·7 (8)° and the mean C_{ar}—C—CH_3 angle is 119·5 (21)°. Compared to the non-quarternized 7-phenylarsatriptycene (van Rooyen-Reiss & Stam,

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

As—C(1)	1·903 (5)	C(8)—C(19)	1·515 (7)
As—C(7)	1·908 (6)	C(9)—C(10)	1·391 (10)
As—C(13)	1·905 (5)	C(10)—C(11)	1·377 (11)
As—C(20)	1·883 (8)	C(11)—C(12)	1·385 (9)
C(1)—C(2)	1·409 (9)	C(12)—C(7)	1·389 (9)
C(2)—C(3)	1·374 (9)	C(13)—C(14)	1·387 (9)
C(2)—C(19)	1·520 (8)	C(14)—C(15)	1·393 (9)
C(3)—C(4)	1·397 (9)	C(14)—C(19)	1·533 (7)
C(4)—C(5)	1·365 (10)	C(15)—C(16)	1·390 (9)
C(5)—C(6)	1·384 (10)	C(16)—C(17)	1·375 (9)
C(6)—C(1)	1·382 (8)	C(17)—C(18)	1·397 (9)
C(7)—C(8)	1·411 (9)	C(18)—C(13)	1·367 (9)
C(8)—C(9)	1·377 (8)		
C(1)AsC(7)	97·5 (3)	C(7)C(8)C(19)	118·2 (5)
C(1)AsC(13)	97·1 (3)	C(9)C(8)C(19)	123·0 (5)
C(1)AsC(20)	117·6 (3)	C(8)C(9)C(10)	119·3 (6)
C(7)AsC(13)	98·6 (3)	C(9)C(10)C(11)	121·1 (6)
C(7)AsC(20)	119·3 (3)	C(10)C(11)C(12)	121·1 (7)
C(13)AsC(20)	121·7 (3)	C(7)C(12)C(11)	117·5 (7)
AsC(1)C(2)	110·8 (4)	AsC(13)C(14)	110·4 (5)
AsC(1)C(6)	127·4 (5)	AsC(13)C(18)	127·7 (5)
C(2)C(1)C(6)	121·8 (6)	C(14)C(13)C(18)	122·0 (6)
C(1)C(2)C(3)	118·3 (5)	C(13)C(14)C(15)	118·8 (6)
C(1)C(2)C(19)	117·8 (5)	C(13)C(14)C(19)	118·9 (6)
C(3)C(2)C(19)	123·9 (5)	C(15)C(14)C(19)	122·2 (5)
C(2)C(3)C(4)	120·2 (6)	C(14)C(15)C(16)	119·4 (6)
C(3)C(4)C(5)	120·3 (6)	C(15)C(16)C(17)	120·8 (6)
C(4)C(5)C(6)	121·4 (6)	C(16)C(17)C(18)	119·9 (6)
C(5)C(6)C(1)	118·0 (6)	C(12)C(18)C(13)	119·0 (6)
AsC(7)C(8)	110·4 (4)	C(2)C(19)C(8)	110·0 (5)
AsC(7)C(12)	127·6 (5)	C(2)C(19)C(14)	110·0 (5)
C(8)C(7)C(12)	122·0 (6)	C(8)C(19)C(14)	108·9 (5)
C(7)C(8)C(9)	118·8 (6)		

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* Lists of structure factors, anisotropic thermal parameters and H atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35044 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1980) [average As—C 1·948 (9) Å, average CAsC angle 91·5 (16)°] quaternization has decreased the As—C_{ar} distance and increased the C_{ar}—As—C_{ar} angle. In arsatriptycene itself (Freijee & Stam, 1980), As—C is 1·94 Å and C—As—C is 90°, but these results are less reliable because of disorder. There is good agreement between corresponding bonds and angles in the three benzene rings. As in 7-phenylarsatriptycene there is a tendency for the inner C—C bonds to be longer and for the outer C—C bonds to be shorter than average. The benzene rings [(I) = C(1)–C(6); (II) = C(7)–C(12); (III) = C(13)–C(18)] are planar within 0·005 (I), 0·008 (II) and 0·015 Å (III). The As atom deviates significantly from all three planes [0·06 (I), 0·09 (II) and 0·05 Å (III)]. The corresponding deviations for C(19) are 0·01, 0·01 and 0·06 Å. The angles between the planes are: (I)–(II) 120·4, (I)–(III) 120·4 and (II)–(III) 119·3°.

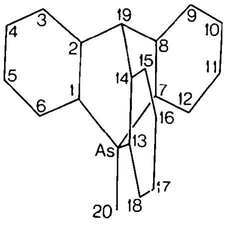

Fig. 1. Atomic numbering and shape of the molecule.

We thank Professor F. Bickelhaupt for suggesting the investigation and providing the crystals and Mr D. Heijdenrijk for technical assistance.

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Acta Cryst. (1980). B**36**, 1256–1258

2-Benzylidene-1-indanone

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(*Received* 21 *July* 1979; *accepted* 22 *January* 1980)

Abstract. $C_{16}H_{12}O$, M_r = 220·3, monoclinic, $P2_1/a$, Z = 4, a = 23·949 (2), b = 7·990 (1), c = 6·095 (1) Å, β = 91·18 (4)°, V = 1166·0 Å^3, D_m = 1·27, D_c = 1·25 Mg m^{-3}. The final R = 0·062 and R_w = 0·058 for 1035 non-zero reflections. The molecule has a *trans-s-cis* configuration.

Introduction. Spectroscopic investigations (IR, UV, NMR) of arylidene derivatives of 1-tetralone and 4-chromanone showed that an increase in the electron interactions of electron-donor substituents in a conjugated bond system leads to the flattening of the molecules (Orlov, Borovoi & Lavrushin, 1976; Orlov, Borovoi, Surov & Lavrushin, 1976).

In order to confirm this conclusion we are undertaking the X-ray study of a series of benzylidene derivatives of 1-tetralone, 1-indanone and 4-chromanone with electron-donor (Rabinovich, 1970) and electron-acceptor (Rabinovich, Schmidt & Shakked, 1970) substituents in the benzylidene rings.

We began our investigations with 2-benzylidene-1-tetralone (Kałuski, Skrzypczak-Jankun, Orlov & Borovoi, 1978) and now present the results of the determination of the molecular and crystal structure of 2-benzylidene-1-indanone. Crystals of this compound, synthesized as in Orlov, Borovoi & Lavrushin (1976) and Orlov, Borovoi, Surov & Lavrushin (1976), were obtained as light-yellow monoclinic plates elongated along the c axis by evaporating its solution in an ethanol–water mixture (3:1 v/v).

A crystal 0·1 × 0·3 × 0·5 mm was investigated on a Syntex $P2_1$ diffractometer to obtain precise lattice constants and to measure the diffraction pattern in the range 2θ = 0–114°. The θ–2θ technique was used with graphite-monochromated Cu $K\alpha$ radiation.

In order to determine the structure model, 1035 reflections with $I > 1{\cdot}96\sigma I$ were used; these were corrected for Lorentz and polarization factors. No absorption or extinction corrections were made. The structure was solved directly by the application of