Table (*cont.*)

hkl	$\|F\|_o$	F_c	*hkl*	$\|F\|_o$	F_c	*hkl*	$\|F\|_o$	F_c	*hkl*	$\|F\|_o$	F_c
28,4,2	22	19	26,6,2	12	10	0,10,2	89	92	6,12,2	51	50
30,4,2	7	9	28,6,2	52	49	2,10,2	23	17	8,12,2	80	78
						4,10,2	28	30	10,12,2	12	15
062	84	87	282	41	49	6,10,2	46	44	12,12,2	43	42
262	37	43	482	83	89	8,10,2	37	36	14,12,2	49	48
462	27	29	682	124	135	10,10,2	32	33	16,12,2	41	38
662	26	22	882	30	35	12,10,2	38	36	18,12,2	36	35
862	89	88	10,8,2	27	30	14,10,2	9	5			
10,6,2	52	44	12,8,2	17	21	16,10,2	10	13	0,14,2	31	39
12,6,2	58	52	14,8,2	72	71	18,10,2	35	34	2,14,2	31	33
14,6,2	18	22	16,8,2	60	61	20,10,2	59	50	4,14,2	25	28
16,6,2	41	42	18,8,2	32	28	22,10,2	43	41	6,14,2	22	24
18,6,2	31	30	20,8,2	12	13				8,14,2	15	15
20,6,2	73	65	22,8,2	58	53	2,12,2	30	26	10,14,2	28	32
22,6,2	15	11	24,8,2	43	38	4,12,2	53	57	12,14,2	27	33
24,6,2	53	44	26,8,2	33	30						

$$0{\cdot}4058X+0{\cdot}4593Y+0{\cdot}7901Z = 2{\cdot}344\ .$$

Atom O_1 deviates $+0{\cdot}085$ Å from this plane (on the side away from the origin), and atom O_2 deviates $-0{\cdot}065$ Å from the plane (on the side towards the origin). The angle O_1–N_1–N_2 is 129°, more than 10° larger than any other bond angle. This also suggests that steric effects occur here.

Table 2 lists distances of the potassium ions to the surrounding atoms. These are generally in agreement with the distances found in other similar potassium salts such as potassium dinitrososulfite (Cox, Jeffrey & Stadler, 1949) and dipotassium nitroacetate (Sutor, Llewellyn & Maslen, 1954).

I wish to thank Miss Joan Reinhart for supplying the compounds used in this investigation and for many discussions on the chemistry of these interesting substances.

References

Cox, E. G., Jeffrey, G. A. & Stadler, H. P. (1949). *J. Chem. Soc.* p. 1783.

Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.

Donohue, J., Lavine, L. R. & Rollett, J. S. (1956). *Acta Cryst.* **9**, 655.

Roof, R. B., Jr. (1956). *Acta Cryst.* **9**, 781.

Sutor, D. J., Llewellyn, F. J. & Maslen, H. S. (1954). *Acta Cryst.* **7**, 145.

Traube, W. (1894). *Ber. dtsch. chem. Ges.* **27**, 1507.

Traube, W. (1895). *Ber. dtsch. chem. Ges.* **28**, 2297.

Traube, W. (1898). *Liebigs Ann.* **300**, 81.

Acta Cryst. (1959). **12**, 585

The Crystal Structures of the Dibromide and Di-iodide of 5:10-dihydro-5:10-dimethylarsanthren

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The dibromide of 5:10-dihydro-5:10-dimethylarsanthren crystallises in the orthorhombic space group $P2_12_12_1$ with unit-cell dimensions $a = 11{\cdot}27$, $b = 9{\cdot}70$, $c = 14{\cdot}87$ Å, while the di-iodide crystallises in the monoclinic space group $P2_1/c$ with $a = 10{\cdot}63$, $b = 9{\cdot}94$, $c = 17{\cdot}02$ Å, $\beta = 108{\cdot}6°$. The coordinates of the heavy atoms were determined from the $h0l$ and $0kl$ sharpened Patterson projections and refined by Fourier syntheses; coordinates for the carbon atoms of the arsanthren ring and probable coordinates for the methyl groups in the dibromide were obtained from difference syntheses. The stereochemistry is similar for the two molecules; they are folded about the As–As axis and the two halogen atoms are disposed one on each side of one arsenic atom, the line joining the halogen atoms being at right angles to the plane which contains the three As–C bonds of this arsenic atom. The valencies of the other arsenic atom are probably non-planar, as in other 3-covalent arsenic compounds, though a planar configuration cannot be definitely excluded. The As–Br and As–I bonds are significantly longer than covalent bonds and may be either ionic or intermediate between ionic and covalent.

1. Introduction

The crystal structures of the dibromide and di-iodide of 5:10-dihydro-5:10-dimethylarsanthren (see Fig. 1) have been investigated in order to determine the nature of the As–Br and As–I bonds, and if possible the stereochemistry of the molecule.

In a chemical investigation of these compounds Jones & Mann (1955) found that all the bromine in an aqueous-methanolic solution of the dibromide is ionic and hence can be estimated volumetrically. In solution, the properties of the di-iodide indicate that it may exist as an equilibrium mixture of the ionic

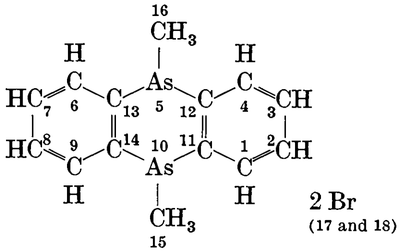

Fig. 1. The dibromide of 5:10-dihydro-5:10-dimethylarsanthren, showing the numbering system.

and covalent forms, but it was deduced, from the deep orange colour of the crystals, that in the crystalline state the di-iodide assumes solely the covalent form, for tertiary arsine di-iodides are normally orange to brown.

The stereochemistry of 3-covalent arsenic was determined by Bradley (1924) who showed that in crystalline arsenic the valencies are non-planar, the intervalency angle being 97°. The isolation of geometric isomers of an organic molecule containing 3-covalent arsenic, 5:10-di-*p*-tolyl-5:10-dihydroarsanthren,* (Chatt & Mann, 1940) demonstrated the non-planarity of the arsenic valencies in this type of compound, the intervalency angle being probably ca. 97°. In a study of the dibromide of 5:10-dihydro-5:10-dimethylarsanthren Jones & Mann (1955) have said that if in solution the bromine is ionic then the cation will exist as a resonance hybrid of the anthracene type and will be uniplanar.

2. Experimental

Crystals of the dibromide and di-iodide were supplied by Dr F. G. Mann, University Chemical Laboratory, Cambridge who suggested the problem to us. The dibromide crystals are colourless, of equal dimensions, and exhibit symmetry 222; while the di-iodide crystals are deep orange needles of hexagonal cross-section and show symmetry $2/m$.

Unit-cell dimensions and space groups obtained from rotation and Weissenberg photographs are listed in Table 1. The densities, measured by flotation using a mixture of trichlorethylene and bromoform, are 1·97±0·02 g.cm.$^{-3}$ for the dibromide and 2·31±0·02 g.cm.$^{-3}$ for the di-iodide; the values calculated for four molecules per cell are 2·00 g.cm.$^{-3}$ and 2·28 g.cm.$^{-3}$ respectively.

Table 1. *Unit-cell dimensions and space groups*

	a (Å)	b (Å)	c (Å)	β (°)	Space group
Dibromide	11·27	9·70	14·87	—	$P2_12_12_1$
Di-iodide	10·63	9·94	17·02	108·6	$P2_1/c$

For intensity data, the $h0l$ and $0kl$ layer lines of both compounds were recorded on Weissenberg photographs with Cu $K\alpha$ radiation, using the multiple-film technique. Scaling factors between films were determined from the common reflexions and the resulting intensities were converted to $|F|^2$ values using Lorentz-Polarization Factors calculated on EDSAC I. No corrections were made for absorption.

3. Determination of the structures

The coordinates of the heavy atoms in both structures were determined from the $h0l$ and $0kl$ sharpened Patterson projections. In both the $h0l$ projections, there are lines of three large peaks, suggesting that in projection three of the four heavy atoms are in a linear array. These coordinates were refined by Fourier syntheses till only small shifts were observed; at this stage, the R factors for the $h0l$ and $0kl$ projections of both compounds were all 24%. The calculations of Fourier series and structure factors were carried out on EDSAC I and EDSAC II. In the calculation of structure factors, the analytical expressions of the atomic scattering factors for the heavy atoms and later for the carbon atoms (Vand, Eiland & Pepinsky, 1957) were used on EDSAC II.

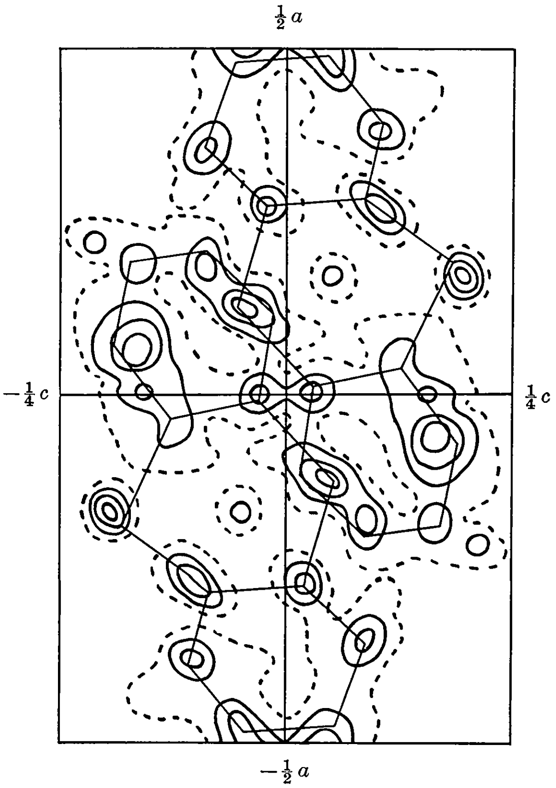

Fig. 2. First $h0l$ difference synthesis of the dibromide using the F_c values calculated from the coordinates of the heavy atoms. The positions of the carbon atoms of the arsanthren ring are readily seen. Contours are at arbitrary but equal intervals with the zero contour broken.

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* In modern nomenclature this compound would be called 5:10-dihydro-5:10-di-*p*-tolylarsanthren.

3·1. *Refinement of the dibromide structure using difference syntheses*

The first $h0l$ and $0kl$ difference syntheses of the dibromide, calculated from the F_c values for the heavy atoms, showed maxima corresponding to the carbon atoms of the arsanthren ring (see Fig. 2). The positions of the methyl groups were not so certain, but subsequent difference syntheses indicated the position of C_{16} attached to As_5 and the probable position of C_{15}, but the coordinates of this latter atom were not used in any structure factor calculations. The heavy atoms were given additional isotropic or anisotropic temperature factors, listed in Table 2, where B_X, B_Y and B_Z are the temperature factors along the axes of the vibration ellipsoid, B_Y is parallel to b^*, B_X and B_Z lie in the a^*c^* plane and ψ is the angle between B_X and a^*.

Table 2. *Additional temperature factors for the dibromide*

	B_X	B_Y	B_Z	ψ (°)
As_5	0·1	0·1	0·1	0
As_{10}	0·3	0·3	0·3	0
Br_{17}	0·8	0·3	0·3	60
Br_{18}	1·0	0·5	0·4	40

The later $h0l$ difference syntheses indicated that most of the carbon atoms in the arsanthren ring are undergoing anisotropic vibrations similar to those in anthracene (Cruickshank, 1956).

The final R factors are 11% for the $h0l$ projection and 14% for the $0kl$ projection. The smooth curves for scaling F_c to F_o cannot be represented in Gaussian

Table 3. *The fractional coordinates*

Dibromide	x	y	z
C_1	0·393	0·080	0·356
C_2	0·480	0·028	0·294
C_3	0·470	0·047	0·203
C_4	0·352	0·098	0·166
As_5	0·1227	0·2500	0·1954
C_6	−0·114	0·292	0·265
C_7	−0·198	0·297	0·338
C_8	−0·182	0·297	0·421
C_9	−0·066	0·264	0·440
As_{10}	0·1922	0·2172	0·4293
C_{11}	0·284	0·144	0·332
C_{12}	0·263	0·148	0·231
C_{13}	0·013	0·263	0·281
C_{14}	0·036	0·260	0·379
C_{15}(?)	0·227	0·180	0·563
C_{16}	0·069	0·138	0·099
Br_{17}	0·2438	0·4711	0·3922
Br_{18}	0·1344	−0·0430	0·4664
Di-iodide			
As_5	0·3773	0·8848	0·2792
As_{10}	0·2391	0·8492	0·0602
I_{17}	0·1766	0·1164	0·0844
I_{18}	0·2750	0·5634	0·0198

Table 4. *Observed and calculated structure factors for the dibromide*

These values should be doubled for $F(000) = 936$

The $00l$ and $h0l$ terms are referred to an origin displaced by $-\frac{1}{4}c$ from the origin for the $0kl$ terms

l	F_o	F_c
	$00l$	
2	16	-19
4	51	-37
6	55	57
8	94	-89
10	47	-50
12	36	-34
14	16	17
16	11	8
18	19	12
	$10l$	
1	95	83
2	105	-114
3	87	-65
4	199	199
5	97	93
6	34	35
7	53	-52
8	6	6
9	21	-23
10	12	-21
11	31	-31
12	9	3
13	7	-3
14	13	-12
15	<9	8
16	9	-14
17	11	10
18	6	-3
19	7	5
	$20l$	
0	108	-123
1	73	-73
2	68	71
3	151	151
4	90	83
5	62	61
6	73	-82
7	7	18
8	10	-4
9	41	36
10	8	-2
11	41	-43
12	23	-26
13	14	-15
14	9	-7
15	22	-27
16	<8	0
17	<7	-1
18	<5	9
19	6	-4
	$30l$	
1	95	-98
2	117	131
3	72	76
4	13	12
5	101	-110
6	20	-20
7	83	84
8	97	100
9	39	51
10	40	-44
11	<9	14
12	26	-33
13	11	7
14	20	-26
15	4	-2
16	7	2
17	20	-21
18	<5	3
	$40l$	
0	49	-43
1	89	94
2	<5	10
3	46	-40
4	111	-114
5	51	-46
6	91	94
7	78	73
8	84	76
9	32	-26
10	18	-21
11	<9	1
12	33	29
13	16	14
14	6	-4
15	14	-12
16	<7	-1
17	<6	5
18	26	-28
	$50l$	
1	24	-26
2	72	-71
3	96	-104
4	84	-89
5	13	15
6	75	71
7	60	62
8	71	-73
9	21	-11
10	9	6
11	62	66
12	37	37
13	14	-14
14	10	16
15	14	-15
16	<7	-2
	$60l$	
0	<6	-8
1	11	-14
2	29	-24
3	87	-90
4	44	54
5	7	2
6	9	-15
7	77	-82
8	64	-62
9	9	-15
10	38	37
11	33	37
12	<9	7
13	8	3
14	24	-25
15	47	41
16	<6	3
	$70l$	
1	35	17
2	11	6
3	46	50
4	36	-36
5	12	14
6	77	-77
7	67	-61
8	21	-23
9	22	25
10	42	40
11	39	-41
12	24	-24
13	<8	3
14	26	20
15	3	7
16	21	19
	$80l$	
0	90	87
1	8	-14
2	37	44
3	8	-10
4	22	19
5	31	-30
6	38	-46
7	9	15
8	22	20
9	14	14
10	40	-46
11	9	-12
12	41	-43
13	17	9
14	33	36
15	<5	-4
16	5	-6
	$90l$	
1	38	32
2	10	-12
3	26	20
4	25	31
5	<9	-3
6	35	34
7	22	-17
8	20	15
9	20	-16
10	45	-48
11	31	-28
12	25	23
13	8	5
14	9	-9
	$10,0,l$	
0	12	-7
1	12	-14
2	10	9
3	43	39
4	14	12
5	29	29
6	<9	3
7	30	24
8	26	-31
9	10	-11
10	5	11
11	14	-12
12	20	15
13	<5	1
	$11,0,l$	
1	8	-10
2	12	14
3	<9	-3
4	26	30
5	<9	3
6	<8	-4
7	15	14
8	15	13
9	9	-8
10	8	8
11	31	31
12	22	-23
	$12,0,l$	
0	29	-27
1	<8	1
2	8	-9
3	9	8
4	23	-21
5	15	11
6	19	20
7	8	-12
8	30	26
9	9	9
10	8	1
	$13,0,l$	
1	27	-21
2	20	-18
3	<7	-1
4	14	-14
5	9	-9
6	5	9
7	15	14
8	19	-17
	$14,0,l$	
0	18	-13
1	<5	3
2	<5	-5
3	26	-25
4	11	10
5	18	-13
	$01l$	
1	37	-19
2	59	59
3	85	-97
4	112	127
5	91	-94
6	47	-49
7	<7	-7
8	23	-16
9	77	79
10	42	-45
11	48	42
12	68	-75
13	<10	0
14	27	31
15	<10	-8
16	<9	-7
17	21	20
18	26	25
19	11	12
	$02l$	
0	57	-48
1	33	-28
2	90	86
3	24	26
4	33	-19
5	44	22
6	33	30
7	10	1
8	113	119
9	41	38
10	27	-10
11	40	38
12	33	-37
13	10	14
14	65	-60
15	<10	-12
16	52	-49
17	15	-6
18	19	19
19	5	-6
	$03l$	
1	18	12
2	43	-20
3	44	-44
4	120	-140
5	92	-92
6	39	-48
7	22	-17
8	47	-43
9	21	21
10	10	4
11	33	34
12	59	60
13	17	7
14	30	-38
15	23	17
16	8	11
17	14	13
18	10	-16
	$04l$	
0	185	208
1	47	20
2	18	14
3	113	120
4	<7	-6
5	61	68
6	37	-41
7	9	8
8	52	-56
9	12	5
10	51	51
11	<10	22
12	33	-36
13	<10	11
14	<10	0
15	<9	-6
16	16	19
17	<7	-1
18	14	-12
	$05l$	
1	18	-14
2	23	33
3	48	-44
4	62	69
5	14	-17
6	94	-113
7	35	37
8	64	-69
9	39	43
10	16	-10
11	<10	0
12	35	-40
13	21	-16
14	20	22
15	20	-19
16	15	-16
17	<5	4
	$06l$	
0	69	-63
1	49	47
2	41	34
3	34	38
4	28	-24
5	<9	-3
6	24	-23
7	<10	-3
8	59	58
9	45	50
10	12	-11
11	53	51
12	22	16
13	28	39
14	19	-16
15	13	-17
16	17	-16
	$07l$	
1	67	57
2	43	53
3	15	23
4	64	-67
5	42	-40
6	29	-27
7	30	-25
8	53	-60
9	12	-5
10	22	-24
11	<10	-1
12	29	37
13	8	5
14	20	-19
15	<6	7
	$08l$	
0	34	29
1	28	25
2	47	-50
3	86	91
4	10	-10
5	35	27
6	10	8
7	<10	0
8	17	-21
9	<10	-1
10	43	38
11	9	8
12	13	-12
13	<7	5
	$09l$	
1	10	3
2	<10	-6
3	<10	7
4	21	20
5	24	25
6	63	-62
7	16	24
8	15	-13
9	<9	2
10	11	10
11	7	-10
12	11	-1
13	17	-14
	$0,10,l$	
0	56	-51
1	15	13
2	9	11
3	<9	10
4	17	-18
5	<9	-12
6	18	-16
7	14	-14
8	14	9
9	13	18
10	14	-18
11	24	18
	$0,11,l$	
1	20	18
2	40	39
3	21	20
4	<8	-6
5	<7	10
6	7	11
7	<6	-5
8	25	-24
9	13	-18
	$0,12,l$	
0	9	-17
1	8	11
2	33	-29
3	16	16
4	<5	2

form. Coordinates are listed in Table 3, and structure factors in Table 4.

3·2. *Refinement of the di-iodide structure using difference syntheses*

Although peaks corresponding to most of the carbon atoms in the arsanthren ring of the di-iodide could be seen in the first $h0l$ difference projection, these carbon atoms could not be located in the $0kl$ projection. The scattering power of iodine is very much greater than that of bromine and it is unlikely that the parameters of the carbon atoms can be determined with any certainty. Refinement of this structure was therefore not taken beyond the correction of the positions of the heavy atoms for series termination effects by the use of difference syntheses, and these corrections did not greatly reduce the R factors. The coordinates of the arsenic and iodine atoms are given in Table 3.

3·3. *Bond lengths and bond angles and their accuracy*

Bond lengths and bond angles between the arsenic and carbon atoms in the dibromide are given in Fig. 3, and those corresponding to the heavy atoms in the dibromide and di-iodide in Fig. 4.

The standard deviations σx, σy and σz of the x, y and z coordinates, calculated by the method of Cruickshank (1949), are listed in Table 5.

Table 5. *Standard deviations of the coordinates*

		σx (Å)	σy (Å)	σz (Å)
Dibromide	As	0·0035	0·0052	0·0035
	Br	0·0035	0·0052	0·0035
	C	0·076	0·090	0·076
Di-iodide	As	0·0123	0·0123	0·0123
	I	0·0076	0·0076	0·0076

The standard deviations of the bond angles were calculated from the equation of Cruickshank & Robertson (1953), and are given in Table 6 together with the standard deviations of the bond lengths.

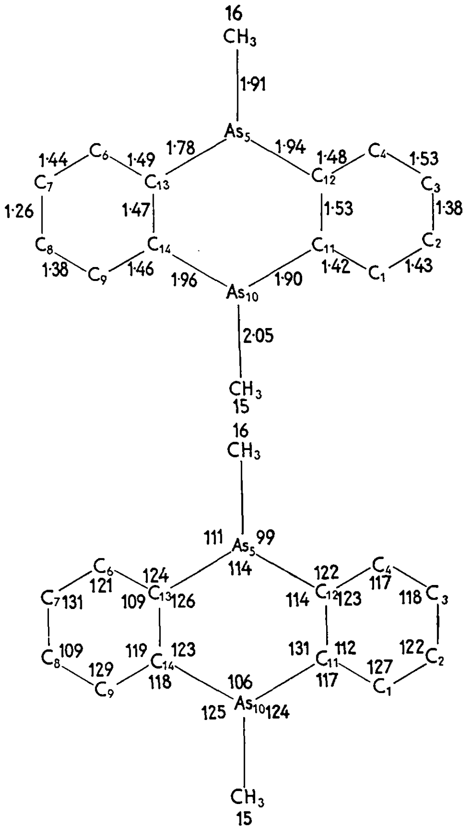

Fig. 3. Bond lengths (Å) and bond angles (°) between the arsenic and carbon atoms in the dibromide.

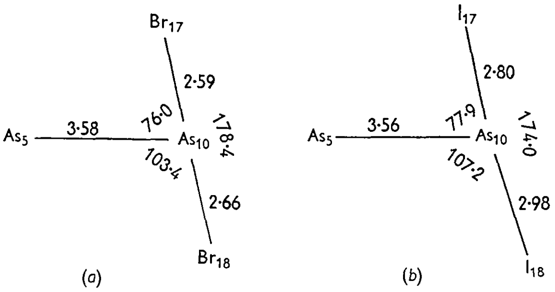

Fig. 4. Bond lengths (Å) and bond angles (°) between the heavy atoms in (a) the dibromide and (b) the di-iodide.

4. Discussion of the structures

No features of special interest are seen in the intermolecular contacts. The following discussion is therefore devoted to the stereochemistry of the molecule and the nature of the bonding of the halogens. Since the spatial arrangement of the heavy atoms is similar for the dibromide and the di-iodide, it may reasonably be assumed that the arrangement of the carbon atoms is also similar for the two compounds. Nevertheless, it should not be forgotten that the positions of the carbon atoms have been directly determined only for the dibromide.

Table 6. *Standard deviations of bond lengths and bond angles*

Bond lengths		As–X	As–As		As–C	C–C
Standard deviation (Å)	X = Br	0·006	0·006		0·08	0·14
	X = I	0·014	0·017		—	—
Bond angles		X–As–X	As–As–X	X–As–C	C–As–C	C–C–C
Standard deviation (°)	X = Br	0·3	0·3	2·4	3·4	7·0
	X = I	1·0	1·0	—	—	—

(i) The molecule is folded along the As_5–As_{10} axis. The direct evidence for this, in the dibromide, is shown in Fig. 5 which is a projection of the molecule on (100), and a simple calculation shows that the two halves of the molecule lie nearly in planes inclined at an angle of about 157°. Indirect supporting evidence is seen in the tilt (Fig. 4) of the nearly linear array of atoms Br_{17}–As_{10}–Br_{18}, with respect to the axis of folding As_5–As_{10}, by as much as 14° and in the direction to be expected since Br_{18} is *cis* with respect to the carbon rings of the folded molecule. The greater length* of the As_{10}–Br_{18} contact (2·66 Å against 2·59 Å for As_{10}–Br_{17}) is also consistent with this configuration of the molecule.

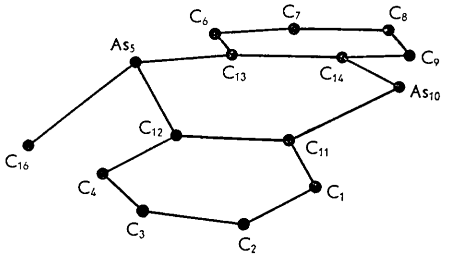

Fig. 5. Projection of the arsenic and carbon atoms down a, showing the folding along the As–As axis. (C_{15} is omitted.)

The indirect evidence for folding of the molecule, derived from the stereochemistry of the heavy atoms, is exactly comparable in the case of the di-iodide: the greater lengths of the As–I bonds correspond to the greater size of the iodine atoms.

(ii) The three As–C bonds around As_{10} are probably planar. Even in the dibromide the uncertainty in the position of C_{15} precludes a definite decision, but for this compound the indirect supporting evidence is strong. Thus the Br_{17} and Br_{18} atoms are 2·59 Å and 2·66 Å above and below As_{10} respectively; the angle Br_{17}–As_{10}–Br_{18} of 178·4° is probably not significantly different from 180°; and the C–As–Br angles quoted in Table 7 are not significantly different from 90°.

Table 7. C–As–Br *angles in the dibromide*

	Angle (°)		Angle (°)
C_{14}–As_{10}–Br_{17}	85	C_{11}–As_{10}–Br_{17}	94
C_{14}–As_{10}–Br_{18}	94	C_{11}–As_{10}–Br_{18}	86

These facts suggest strongly that C_{15} lies in the plane of $C_{11}As_{10}C_{14}$; in this case the coordination group around As_{10} is similar to that about the antimony atom in the trimethylstibnine dihalides, in which the halogen atoms are symmetrically disposed on opposite sides of a planar $Sb(CH_3)_3$ group (Wells, 1938).

Such evidence as is available suggests a similar configuration for the di-iodide.

(iii) In the group about As_{10} the As–Br and As–I bonds are probably either ionic or intermediate between ionic and covalent. The evidence lies in a comparison of the contact distances in the dibromide (2·59 and 2·66 Å) and di-iodide (2·80 and 2·98 Å) with those for the covalent bonds in $AsBr_3$ (2·31–2·36 Å) and AsI_3 (2·51–2·58 Å) (Allen & Sutton, 1950). The differences are far greater than might arise from inaccuracies in bond length determinations.

(iv) In the dibromide structure, as described, the valencies of As_5 are non-planar as in other 3-covalent arsenic compounds. As_5 is situated at the apex of a pyramid with the valencies directed along the edges; the average inter-valency angle (Fig. 3) is 108°, somewhat larger than might be expected for purely covalent bonds. It must be emphasised that this description of the bond-configuration depends upon the correct location of the single atom C_{16}, so that a planar disposition of the As_5 valencies cannot be entirely ruled out on the available evidence. No evidence on the As_5 valencies is available for the di-iodide.

(v) In the dibromide structure the limit of error in fixing the As–C and C–C distances is too great to permit any decision on whether these correspond to single or double bonds, i.e. whether the carbon rings are benzenoid or *ortho*-quinonoid.

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References

Allen, P. W. & Sutton, L. E. (1950). *Acta Cryst.* **3**, 46.
Bradley, A. J. (1924). *Phil. Mag.* **47**, 657.
Chatt, J. & Mann, F. G. (1940). *J. Chem. Soc.* p. 1184.
Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.
Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 915.
Cruickshank, D. W. J. & Robertson, A. P. (1953). *Acta Cryst.* **6**, 698.
Jones, Emrys R. H. & Mann, F. G. (1955). *J. Chem. Soc.* p. 411.
Vand, V., Eiland, P. F. & Pepinsky, R. (1957). *Acta Cryst.* **10**, 303.
Wells, A. F. (1938). *Z. Krystallogr.* **99**, 367.

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* Application of the tests of Cruickshank & Robertson (1953) shows that the difference between the two bond lengths As_{10}–Br_{17} and As_{10}–Br_{18} is significant. (Similarly for As_{10}–I_{17} and As_{10}–I_{18}).