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Refinement of the Crystal Structures of $(C_6H_5)_4AsI_3$ and CsI_3 at 20°C and at −160°C

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Three-dimensional intensity data have been collected both at room temperature and at −160°C for the triiodides CsI_3 and $(C_6H_5)_4AsI_3$. The present work confirms the conclusion of Tasman & Boswijk [*Acta Cryst.* (1955), **8**, 59] regarding the asymmetry and non-linearity of the I_3^- ion in CsI_3. The bond lengths in the symmetric I_3^- ions in $(C_6H_5)_4AsI_3$ [2·919 (1) Å] are somewhat longer than the values reported by Mooney-Slater [*Acta Cryst.* (1959), **12**, 187], but are still shorter than the average I–I bond lengths observed for $(C_2H_5)_4NI_3$(I) and $(C_2H_5)_4NI_3$(II), 2·935 and 2·936 Å respectively. The I–I bond lengths observed in different triiodides are compared, and the differences are discussed.

Introduction

The configurations of I_3^- ions in different crystals have aroused considerable interest (Brown & Nunn, 1966; Migchelsen & Vos, 1967; Wiebenga & Kracht, 1969). The bond lengths and angles known with reasonable accuracy at the beginning of the present study are given in Table 1(*a*). The Table shows that for $(C_2H_5)_4NI_7$ the I–I bond length is significantly shorter than the average I–I bond lengths observed for the compounds $(C_2H_5)_4NI_3$(I) and $(C_2H_5)_4NI_3$(II). This does not hold for $(C_6H_5)_4AsI_3$ for which the bonds had been reported to be relatively short (Mooney-Slater, 1959; Rundle, 1961). The presence of the short distances in $(C_2H_5)_4NI_7$ at −195°C cannot be attributed to the electrostatic crystal field as for all symmetric I_3^- ions the calculated bond orders are independent of this field (Wiebenga & Kracht, 1969). We have therefore tentatively assumed the presence of the short I–I bonds in $(C_2H_5)_4NI_7$ at −195°C to be due to either a shrinkage of the weak I–I bonds at low temperature or to complex formation (the I_3^- ions have strong interactions with neighbouring I_2 molecules). To get more insight in this problem we have refined the crystal structures of $(C_6H_5)_4AsI_3$, having symmetric I_3^- ions, and of CsI_3, having asymmetric I_3^- ions, both at room and at low temperature.

Discussion of the I–I bond lengths

Influence of the temperature

The I–I bond lengths and angles observed in $(C_6H_5)_4AsI_3$ and in CsI_3 at room temperature and at low temperature, are given in Table 1(*b*). We see that in both compounds the bond lengths at low temperature are equal to those at room temperature, so that it can be concluded that no shrinkage of the bonds occurs at low temperature.

Symmetric I_3^- ions

Comparison of the bond lengths in the different symmetric I_3^- ions in Table 1 shows that there are significant differences between the lengths of the bonds. Values increasing from 2·904 to 2·943 Å are observed when going from $(C_2H_5)_4NI_7$ *via* $(C_6H_5)_4AsI_3$ and $(C_2H_5)_4NI_3$(I) (ion *A*) to $(C_2H_5)_4NI_3$(I) (ion *B*). Consideration of the crystal structures shows that both for the I_3^- ion *B* in $(C_2H_5)_4NI_3$(I), having the longest I–I distance, and for the I_3^- ion in $(C_2H_5)_4NI_7$, having the shortest I–I distance, there is a relatively strong interaction with neighbouring iodine atoms. The type of interaction is different, however, for the two ions. Ion *B* is involved in intermolecular distances shorter than the van der Waals distance in such a way that a linear array I···I–I–I···I is formed [Fig. 1(*a*)], whereas for I_3^- in $(C_2H_5)_4NI_7$ the short intermolecular I···I distances are roughly perpendicular to the (I–I–I)⁻ ion [Fig. 1(*c*)]. For ion *A* in $(C_2H_5)_4NI_3$(I) the intermolecular interaction [Fig. 1(*b*)] is much smaller than for ion *B*, while for the I_3^- ion in $(C_6H_5)_4AsI_3$ no short intermolecular distances are observed ('free' I_3^- ion). This seems to suggest that the I–I bond in a free I_3^-ion lies close to 2·920 Å. The presence of a linear array as shown in Fig. 1(*a*) appears to increase the I–I bond length, whereas the configuration of Fig. 1(*c*) causes a shortening of the bonds.

The lengthening of the bonds in ion *B* has been estimated by doing a PPP calculation (Pariser & Parr, 1953; Pople, 1953) for both the ions *A* and *B* in $(C_2H_5)_4NI_3$(I). The *p* orbitals used for each of the calculations are given in Fig. 2. The intra- and intermolecular distances were taken from the crystal structure, the electrostatic crystal field was not considered as this does not change the calculated lengths for the bonds in symmetric I_3^- ions (see introduction). The calculated bond orders *P* are 0·690 for ion *B* and 0·704 for ion *A* ($P=0{\cdot}707$ for an isolated I_3^- ion), so that the bonds for *B* are expected to be longer than for *A* in agreement with experiment. From the slope $dR/dP=-1{\cdot}2$ at $P=0{\cdot}7$ of the $R(P)$ curve given by Wiebenga & Kracht (1969; Fig. 2), ΔR is calculated at 0·017 Å, which is of the right order of magnitude.

The shortening of the bonds in the I_3^- ion in $(C_2H_5)_4NI_7$ could not be made plausible from simple PPP calculations, as in this case the complete three-dimensional network consisting of I_3^- ions and I_2 molecules has to be considered. For this compound further theoretical work is required.

Asymmetric I_3^- ions

Theoretical calculations on free I_3^- ions including the (electrostatic) crystal field, predict unequal bonds for I_3^- ions having asymmetric surroundings in the crystal. As long as the asymmetry is small, the average value of the bonds is expected to be negligibly larger than the lengths observed for free symmetric I_3^- ions. For highly asymmetric ions a small increase of the average bond length is expected [see $R(P)$ curve in Wiebenga & Kracht, 1969]. Table 1 shows that for the asymmetric ions in $(C_2H_5)_4NI_3$(II) and in CsI_3 $\frac{1}{2}[R_{12}+R_{23}]$ is, within experimental error, equal to the length of the I–I bond of ion *B* in $(C_2H_5)_4NI_3$(I) and larger than the value observed for the free I_3^- ion in $(C_6H_5)_4AsI_3$.

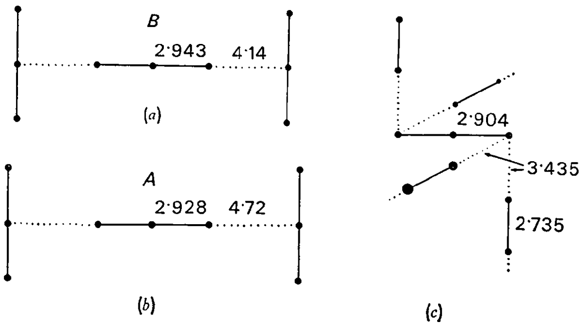

Fig. 1. Shortest intermolecular distances in the neighbourhood of (*a*) ion *B* in $(C_2H_5)_4NI_3$(I), (*b*) ion *A* in $(C_2H_5)_4NI_3$(I) and (*c*) the I_3^- ion in $(C_2H_5)_4NI_7$.

Table 1. *Distances R_{12} and R_{23}* (Å) *and angles* (°) *for I_3^- ions in different crystals*

The standard deviations in parentheses are in units of the last decimal place. Sym. = symmetry special position I_3^- ion; I···I = shortest intermolecular I···I distance, given only if smaller than van der Waals value.

(*a*) Values from older work

Compound		Sym.	Angle	R_{12}	R_{23}	$\frac{1}{2}[R_{12}+R_{23}]$	I···I	Literature
$(C_2H_5)_4NI_7$		2/*m*	180	2·910 (9)	2·910 (9)	2·910 (9)	3·47	Havinga & Wiebenga, 1958.
$(C_2H_5)_4NI_7$ (−195 °C)		2/*m*	180	2·904 (3)	2·904 (3)	2·904 (3)	3·44	Havinga & Wiebenga, 1958.
$(C_6H_5)_4AsI_3$		2	176·4	2·90 (2)	2·90 (2)	2·90 (2)		Mooney-Slater, 1959.
$(C_6H_5)_2FeI_3$		$\bar{3}$ *m*	180	2·93 (2)	2·93 (2)	2·93 (2)		Bernstein & Herbstein, 1968.
$(C_2H_5)_4NI_3$(I)	*A**	2/*m*	180	2·928 (2_5)	2·928 (2_5)	2·928 (2_5)		Migchelsen & Vos, 1967.
	B	2/*m*	180	2·943 (2_5)	2·943 (2_5)	2·943 (2_5)	4·14	Migchelsen & Vos, 1967.
$(C_2H_5)_4NI_3$(II)	*A*	*m*	177·1 (1)	2·912 (3_5)	2·961 (3_5)	2·936 (2)	4·24	Migchelsen & Vos, 1967.
	B	*m*	179·5 (1)	2·892 (3_5)	2·981 (3_5)	2·936 (2)	4·10	Migchelsen & Vos, 1967.
CsI_3		*m*	176·3 (5)	2·83 (1_5)	3·04 (1_5)	2·94 (1_5)	4·03	Tasman & Boswijk, 1955.

(*b*) Recently determined values

Compound	Sym.	Angle	R_{12}	R_{23}	$\frac{1}{2}[R_{12}+R_{23}]$	I···I	Literature
$(C_6H_5)_3AsI_3$	2	175·61 (5)	2·920 (2)	2·920 (2)	2·920 (2)		Present study.
$(C_6H_5)_4AsI_3$ (−160 °C)	2	174·69 (2)	2·919 (1)	2·919 (1)	2·919 (1)		Present study.
CsI_3	*m*	177·9 (1)	2·840 (4)	3·042 (4)	2·941 (2)	4·01	Present study.
CsI_3 (−160 °C)	*m*	178·00 (6)	2·842 (2)	3·038 (2)	2·940 (1)	3·97	Present study.
NH_4I_3	*m*	180·0 (1)	2·791 (4)	3·113 (4)	2·952 (2)	3·88	Cheesman & Finney, 1970.

* Two independent ions.

Consideration of the crystal structures shows that this lengthening can be ascribed to intermolecular interaction.

For the asymmetric ions in NH_4I_3 the value for $\frac{1}{2}[R_{12}+R_{23}]$ is slightly larger than the values observed for the remaining asymmetric I_3^- ions. A small elongation relative to these ions can be expected as: (1) the intermolecular distances in NH_4I_3 are somewhat shorter than, for instance, in the isomorphous compound CsI_3, and (2) the asymmetry of the ions is relatively high. It must further be mentioned that accurate work on NH_4I_3 is hampered by its rapid decomposition, so that it cannot be excluded that the standard deviations reported by Cheesman & Finney (1970) are lower estimates of the errors in the structure.

Structure refinements

Crystals of CsI_3 were obtained by adding iodine to an equivalent amount of CsI dissolved in ethanol and slow evaporation of the solute. Crystals of $(C_6H_5)_4AsI_3$ were grown by diffusion from solutions of $(C_6H_5)_4AsI$ and I_2 in ethanol, the solutions being put in the vertical parts of a U-shaped tube having pure ethanol between glass filters in its horizontal part. Large crystals measuring approximately 0·2 × 0·3 × 0·4 mm were obtained after some days. Details of the experimental work are given in Table 2. For the low temperature work the crystals were cooled down to −160 °C by a stream of cold nitrogen gas (for apparatus used, see van Bolhuis, 1971).

Table 2. *Details of the experimental work on* $(C_6H_5)_4AsI_3$ *and* CsI_3

The compounds are indicated as C(1) and C(2) in the Table.

Crystallographic data (Table 3)	Zero layer line Weissenberg photographs calibrated with NaCl reflexions; λ(Cu $K\alpha_1$) = 1·54050, λ(Cu $K\alpha_2$) = 1·54434, a(NaCl) = 5·64006 Å.
Method used for intensity measurements	3-circle Nonius diffractometer, θ–2θ scan, Zr-filtered Mo radiation.
Crystal used	Approximately 0·2 × 0·3 × 0·4 mm [C(1)] and 0·11 × 0·14 × 0·14 mm [C(2)].
θ region and number of reflexions with I(net) > 0	$\theta \leq 28°$, 2827 [C(1), 20 °C]; $\theta \leq 38°$, 6257 [C(1), −160 °C] $\theta \leq 25°$, 747 [C(2), 20 °C]; $\theta \leq 35°$, 1630 [C(2), −160 °C]
Calculation of $\|F\|$	Corrections for Lp and for absorption according to Busing & Levy (1957).
Starting coordinates	C(1): Mooney-Slater, 1959. C(2): Tasman & Boswijk, 1955.
Refinement	Anisotropic block-diagonal least-squares (Cruickshank, 1961), TR4 computer, hydrogen atoms fixed with reasonable parameters.
Weighting scheme*	$w=[\{\sigma_c(F_o)\}^2+PF_o^2]^{-1}$ $P=0{\cdot}25\times10^{-4}$ [C(1), 20 °C]; $P=0{\cdot}6\times10^{-4}$ [C(1), −160 °C] $w=1$ [C(2), 20 °C]; $P=1\times10^{-4}$ [C(2), −160 °C]
Extinction	Zachariasen (1968).
Scattering factors	f_o (Doyle & Turner, 1968; Stewart, Davidson & Simpson, 1965). $\Delta f'$ and $\Delta f''$ (*International Tables for X-ray Crystallography*, 1962)
$R_w=\frac{\sum w\|\Delta F\|}{\sum w\|F_o\|}$	0·061 [C(1), 20 °C]; 0·044 [C(1), −160 °C] 0·066 [C(2), 20 °C]; 0·067 [C(2), −160 °C]
Final parameters†	Tables 4, 5 [C(1), −160 °C], Table 6 [C(2), −160 °C]
Bond lengths and angles	Table 7
$\|F_o\|$ and $\|F_c\|$	Available on request.

* $\sigma_c(F_o)$ is the standard deviation in F_o based on counting statistics.
† Final parameters are given for the low temperature studies only.

Table 3. *Crystallographic data*

Compound	$(C_6H_5)_4AsI_3$		CsI_3	
Temperature	−160 °C	20 °C	−160 °C	20 °C
a	15·365 (5) Å	15·421 (4) Å	6·751 (5) Å	6·853 (3) Å
b	7·622 (3)	7·773 (3)	9·963 (5)	10·028 (8)
c	10·298 (4)	10·422 (3)	10·997 (7)	11·082 (5)
β	93·58 (2)°	93·03 (1)°		
Space group	$P2/n$		$Pmcn$	
Z	2		4	
μ(Mo)	53·6 cm^{-1}	52·0 cm^{-1}	176·0 cm^{-1}	171·0 cm^{-1}

Table 4. $(C_6H_5)_4AsI_3$ *at* −160°C. *Final coordinates with standard deviations as calculated by the least-squares program in units of the last decimal place in parentheses.*

For numbering of atoms see Fig. 3.

	x	y	z
I(1)*	0·25000 (0)	0·14500 (3)	0·75000 (0)
I(2)	0·06093 (1)	0·16273 (3)	0·70221 (2)
As*	0·25000 (0)	−0·01473 (5)	0·25000 (0)
C(1)	0·2319 (2)	−0·1663 (3)	0·3925 (3)
C(2)	0·1594 (2)	−0·1455 (3)	0·4655 (3)
C(3)	0·1449 (2)	−0·2653 (4)	0·5633 (3)
C(4)	0·2021 (2)	−0·4036 (4)	0·5856 (3)
C(5)	0·2743 (2)	−0·4243 (4)	0·5138 (3)
C(6)	0·2903 (2)	−0·3060 (4)	0·4155 (3)
C(7)	0·3472 (2)	0·1367 (3)	0·2844 (3)
C(8)	0·3963 (2)	0·1281 (4)	0·4022 (3)
C(9)	0·4647 (2)	0·2465 (4)	0·4244 (3)
C(10)	0·4825 (2)	0·3700 (4)	0·3300 (3)
C(11)	0·4325 (2)	0·3778 (4)	0·2144 (3)
C(12)	0·3640 (2)	0·2614 (4)	0·1892 (3)

* Special position with symmetry 2.

Details of the structures

The bond lengths and angles in $(C_6H_5)_4AsI_3$ and CsI_3 at −160°C are listed in Table 7.

The [010] projection of the structure of $(C_6H_5)_4AsI_3$ is given in Fig. 3, both the I_3^- ion and the $(C_6H_5)_4As^+$ group lie at special positions with symmetry 2. It is seen that the I_3^- ions have no short intermolecular distances to neighbouring atoms. The As atom is approximately tetrahedrally surrounded by benzene rings, which are planar within experimental error. The suggestion given in the literature (Brown & Nunn,

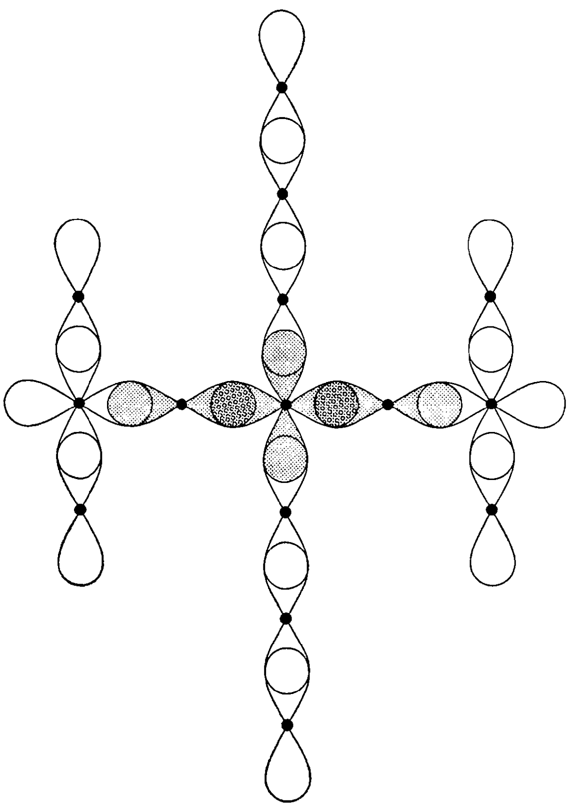

Fig. 2. The p-orbitals used in the bond order calculations for the I_3^- ions in $(C_2H_5)_4NI_3$(I). The orbitals of the 'central' ion (A or B) are dotted. The structure is idealized in such a way [compare with Fig. 1(a) in Migchelsen & Vos, 1967], that only angles of 90° and 180° are considered. Only part of a two-dimensional layer is taken into account.

Table 5. $(C_6H_5)_4AsI_3$ *at* −160°C. *Thermal parameters* × 10^4 (Å^2) *of the temperature factor* $\exp[-2\pi^2(h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots)]$

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
I(1)	258 (1)	168 (1)	238 (1)	0	0	−25 (2)
I(2)	237 (1)	304 (1)	329 (1)	77 (1)	−80 (2)	19 (1)
As	158 (1)	156 (2)	170 (2)	0	0	21 (2)
C(1)	193 (10)	170 (10)	172 (10)	25 (17)	35 (17)	4 (16)
C(2)	211 (11)	198 (11)	221 (12)	−26 (19)	5 (19)	47 (18)
C(3)	315 (14)	255 (13)	253 (14)	−67 (22)	84 (22)	124 (22)
C(4)	394 (16)	225 (13)	250 (14)	−152 (23)	147 (22)	−70 (24)
C(5)	340 (15)	220 (13)	319 (15)	43 (22)	147 (23)	−96 (24)
C(6)	253 (13)	220 (12)	295 (14)	104 (20)	65 (22)	− 8 (21)
C(7)	178 (10)	173 (11)	205 (11)	−15 (17)	−27 (18)	40 (17)
C(8)	207 (11)	237 (12)	213 (12)	10 (18)	−15 (19)	12 (18)
C(9)	242 (12)	296 (14)	271 (14)	−56 (21)	−115 (23)	−79 (21)
C(10)	267 (13)	219 (13)	344 (15)	−125 (21)	−101 (23)	51 (23)
C(11)	272 (13)	203 (12)	369 (17)	−100 (20)	− 5 (23)	128 (24)
C(12)	246 (12)	210 (12)	224 (12)	−46 (19)	5 (19)	8 (19)

Table 6. CsI_3 *at* −160°C. *Positional and thermal parameters* (Å^2)

All atoms lie on the mirror plane $x=0·25$. For numbering of atoms see Fig. 4.

	y	z	U_{11}	U_{22}	U_{33}	$2U_{23}$
I(1)	0·35763 (9)	0·16272 (8)	0·0226 (4)	0·0142 (3)	0·0154 (3)	−0·0008 (5)
I(2)	0·54967 (8)	0·37731 (8)	0·0177 (4)	0·0142 (3)	0·0140 (3)	0·0024 (5)
I(3)	0·73692 (9)	0·57225 (8)	0·0238 (4)	0·0146 (3)	0·0159 (3)	0·0019 (5)
Cs	0·46639 (9)	0·82872 (8)	0·0202 (4)	0·0185 (3)	0·0164 (3)	−0·0029 (5)

Table 7. *Distances and angles in* $(C_6H_5)_4AsI_3$ (*a*) *and in* CsI_3 (*b*) *at* −160°C

The standard deviations in the Table are obtained from those given in Tables 4 and 6 after multiplication by 1·5, as the standard deviations from the least-squares program are lower estimates of the errors in the structure (Visser & Vos, 1971).

(*a*)			
I(1)—I(2)	2·919 (1) Å	I(2)—I(1)—I(2′)	174·69 (2)°
C(1)—C(2)	1·392 (6)	C(6)—C(1)—C(2)	121·4 (4)
C(2)—C(3)	1·388 (6)	C(1)—C(2)—C(3)	119·2 (4)
C(3)—C(4)	1·383 (7)	C(2)—C(3)—C(4)	119·5 (4)
C(4)—C(5)	1·379 (7)	C(3)—C(4)—C(5)	121·5 (4)
C(5)—C(6)	1·389 (7)	C(4)—C(5)—C(6)	120·1 (4)
C(6)—C(1)	1·403 (6)	C(5)—C(6)—C(1)	118·3 (4)
		C(12)–C(7)—C(8)	121·9 (4)
C(7)—C(8)	1·389 (6)	C(7)—C(8)—C(9)	118·4 (4)
C(8)—C(9)	1·393 (6)	C(8)—C(9)—C(10)	120·1 (4)
C(9)—C(10)	1·393 (7)	C(9)—C(10)–C(11)	120·6 (4)
C(10)–C(11)	1·377 (7)	C(10)–C(11)–C(12)	120·7 (4)
C(11)–C(12)	1·388 (6)	C(11)–C(12)–C(7)	118·2 (4)
C(12)–C(7)	1·401 (6)	C(1)—As —C(7)	112·1 (2)
		C(1′) –As —C(7)	111·2 (2)
As —C(1)	1·902 (4)	C(1)—As —C(1′)	105·2 (2)
As —C(7)	1·903 (4)	C(7)—As —C(7′)	105·3 (2)
(*b*)			
I(1)—I(2)	3·038 (2) Å	I(1)–I(2) –I(3)	178·00 (6)°
I(2)—I(3)	2·842 (2)	I(2)–I(3) –I(2′)	171·34 (6)
I(3)···I(2′)	3·972 (2)	I(3)–I(2′)–I(1′)	73·40 (6)
I(3)···I(1′)	4·160 (2)		

1966; Bernstein & Herbstein, 1968), that the twofold symmetry of the I_3^- ion is not genuine but due to disorder, is not supported by the thermal parameters of the present low temperature study.

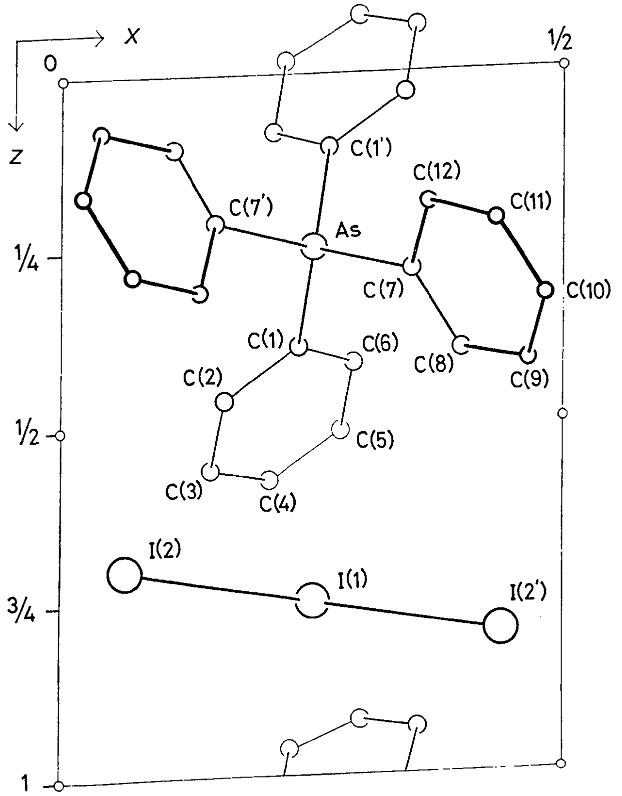

Fig. 3. The structure of $(C_6H_5)_4AsI_3$ seen along the [010] axis. As and I(1) lie on the twofold axes $[\frac{1}{4},y,\frac{1}{4}]$ and $[\frac{1}{4},y,\frac{3}{4}]$ respectively.

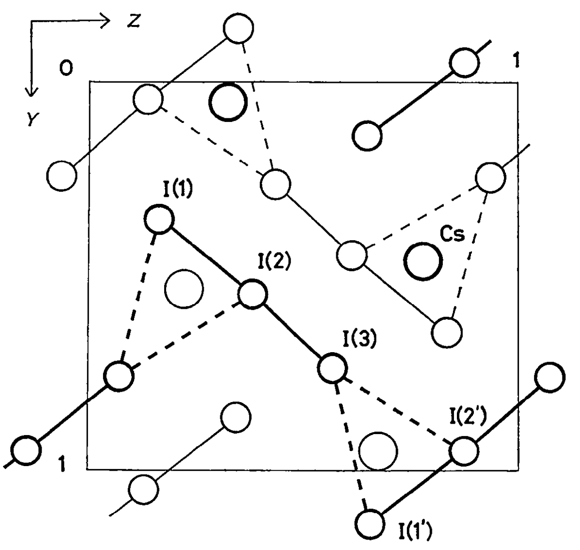

Fig. 4. The structure of CsI_3 seen along the [010] axis.

In CsI_3 all atoms lie in the mirror plane $x=0·25$, and in symmetrically related planes. Fig. 4 clearly shows that the surroundings of the I_3^- ion are strongly asymmetric, and that there are approximately linear arrays of type I–I–I···I.

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