SHORT STRUCTURAL PAPERS

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Silver Iodide–Hexamethylethylenediamine

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Abstract. $Ag_6I_8C_8H_{22}N_2$, triclinic, $P\bar{1}$, $a=10{\cdot}47$ (1), $b=9{\cdot}58$ (1), $c=7{\cdot}86$ (1) Å, $\alpha=101{\cdot}4$ (4), $\beta=101{\cdot}8$ (4), $\gamma=95{\cdot}5$ (4)°, $Z=1$, $D_x=4{\cdot}01$ g cm^{-3}. Final $R=0{\cdot}076$. The Ag are tetrahedrally surrounded by I atoms with all the tetrahedra doubly edge-shared.

Introduction. $Ag_6I_8C_8H_{22}N_2$ was synthesized by reacting stoichiometric quantities of AgI and hexamethylethylenediamine diiodide at 130°C. Colourless crystals of the material were isolated from the solid reaction mixture. A spherical crystal with diameter 0·04 cm was selected for examination. The cell parameters and intensities for 1831 independent reflexions were measured on a Philips PW1100 four-circle automatic diffractometer. Data were collected in the range $3° \le \theta \le 22°$ with graphite-monochromated Mo $K\alpha$ radiation ($\lambda=0{\cdot}7107$ Å, ω–2θ scan, scan width 1·2°, scan speed 0·04° s^{-1}). 1379 reflexions with $I/\sigma(I) > 1{\cdot}65$, where $\sigma(I)=[(0{\cdot}02S)^2+S+B]^{1/2}$, S = scan count and B = total background count, were considered to be observed. The background was counted for half the total scanning time on each side of the reflexion.

Table 1. *Refined atomic parameters* ($\times 10^4$)

Thermal parameters are of the form $T=\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)]$. Standard deviations are given in parentheses.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	588 (4)	−874 (5)	6869 (7)	502 (27)	660 (32)	885 (36)	−8 (23)	−31 (24)	214 (27)
Ag(2)	2181 (5)	1159 (5)	2349 (6)	633 (29)	573 (29)	748 (33)	131 (23)	184 (24)	163 (25)
Ag(3)	3492 (4)	−267 (5)	5649 (6)	595 (28)	595 (29)	622 (29)	−7 (22)	39 (22)	149 (23)
I(1)	753 (3)	3443 (3)	2794 (5)	413 (19)	423 (20)	491 (20)	69 (16)	11 (15)	153 (16)
I(2)	1035 (3)	−1445 (3)	3187 (4)	340 (18)	315 (19)	433 (20)	−23 (14)	19 (14)	23 (15)
I(3)	3061 (3)	180 (3)	9175 (4)	418 (19)	397 (20)	344 (18)	30 (15)	9 (14)	45 (15)
I(4)	4702 (3)	2280 (3)	4907 (4)	371 (18)	351 (18)	355 (17)	−35 (14)	31 (14)	10 (14)
C(1)	4369 (38)	4431 (44)	−315 (60)	128 (214)	224 (241)	436 (280)	29 (181)	−55 (195)	85 (209)
C(2)	2068 (41)	3903 (44)	−1757 (57)	274 (244)	190 (242)	344 (262)	−226 (198)	−55 (198)	16 (201)
C(3)	2828 (50)	6209 (52)	532 (60)	513 (331)	435 (309)	177 (272)	132 (258)	152 (235)	−119 (230)
C(4)	3393 (49)	5886 (52)	−2501 (56)	566 (313)	474 (306)	214 (244)	−193 (250)	87 (222)	192 (224)
N(1)	3177 (34)	5074 (40)	−1031 (45)	330 (216)	441 (242)	195 (207)	9 (180)	−10 (164)	103 (185)

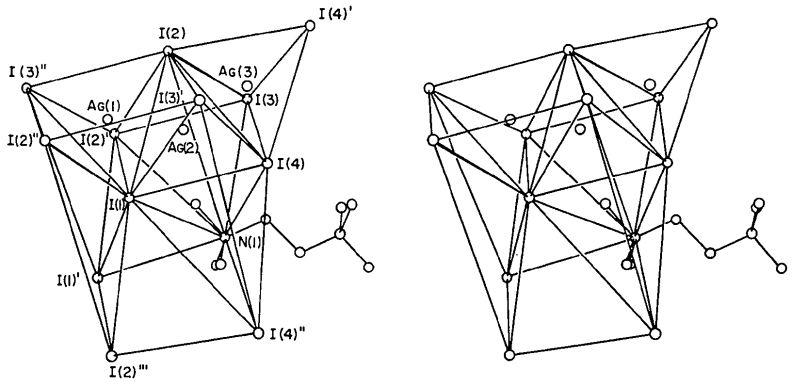

Fig. 1. Stereoscopic drawing showing the atomic arrangement in $Ag_6I_8C_8H_{22}N_2$.

No correction for absorption was made. Lorentz and polarization factors were applied.

The symbolic addition procedure for centrosymmetric crystals (Karle & Karle, 1966) was followed for solving the structure, with the program *PHASE* from the X-RAY 72 set of programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). All other computations were also made with these programs.

The positional and anisotropic thermal parameters were refined by full-matrix least-squares calculations with unit weight for all reflexions. This led to a final R of 0·076. The scattering factors were those of Cromer & Mann (1968).

The atomic parameters are listed in Table 1, and selected interatomic distances and angles in Table 2.*

Discussion. The structural features of $Ag_6I_8C_8H_{22}N_2$ are shown in Fig. 1 (*ORTEP*: Johnson, 1965). The AgI lattice is characterized by layers of Ag atoms on (042) planes, sandwiched between layers of I atoms on (021) planes. Each Ag is tetrahedrally surrounded by I with all the tetrahedra doubly edge-shared. The average Ag···I and I···I interatomic distances in a tetrahedron are 2·87 and 4·68 Å, respectively. A set of four tetrahedra is stacked in such a way that each contributes a single face to form a square pyramid of I atoms. This is illustrated in Fig. 1 by I(1), I(2), I(2″), I(3′) and I(3″).

If the N atoms of the diamine molecule are included in the AgI lattice, we find that both N's lie approximately in the plane described by the I atoms. It is further seen that a combination of I and N atoms gives rise to a quasi body-centred lattice [*e.g.* I(1), I(1′), I(2), I(2‴), I(2″), I(3′), I(3‴), I(4″) and N(1) in Fig. 1].

Two types of tetrahedra are created as a result of this lattice. One consists only of I atoms [*e.g.* I(1), I(2), I(2′), and I(3″)] which enclose an Ag atom, while the other is formed by one N and three I [*e.g.* I(1), I(1′), I(2′) and N(1)].

The asymmetric unit contains only half a formula unit, *viz* $Ag_3I_4 . \frac{1}{2}(C_8H_{22}N_2)$ with the $C_8H_{22}N_2$ molecule lying on the symmetry centre at $\frac{1}{2},\frac{1}{2},0$.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31029 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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

Standard deviations are in parentheses.

The primed atoms are related to the unprimed by a centre of symmetry.

Ag(1)–I(1′)	2·80 (1)	I(1)–I(2)	4·79 (2)
Ag(1)–I(2)	2·98 (1)	I(1′)–I(2′)	4·82 (2)
Ag(1)–I(3)	2·81 (1)	I(1′)–I(2)	4·61 (1)
Ag(1)–I(2′)	2·92 (1)	I(1)–I(3′)	4·94 (2)
Ag(2)–I(1)	2·77 (1)	I(1′)–I(3)	4·80 (2)
Ag(2)–I(2)	2·92 (1)	I(1)–I(4)	4·46 (2)
Ag(2)–I(3′)	2·85 (1)	I(2)–I(3)	4·63 (2)
Ag(2)–I(4)	2·93 (1)	I(2)–I(3′)	4·57 (1)
Ag(3)–I(2)	2·86 (1)	I(2′)–I(3)	4·68 (2)
Ag(3)–I(3)	2·85 (1)	I(2)–I(2′)	4·58 (1)
Ag(3)–I(4)	2·86 (1)	I(2)–I(4)	4·76 (2)
Ag(3)–I(4′)	2·86 (1)	I(2)–I(4′)	4·61 (2)
		I(3)–I(4)	4·76 (1)
N(1)–C(1)	1·50 (2)	I(3)–I(4′)	4·74 (1)
N(1)–C(2)	1·47 (2)	I(3′)–I(4)	4·57 (1)
N(1)–C(3)	1·59 (2)	I(4)–I(4′)	4·50 (2)
N(1)–C(4)	1·56 (2)		
C(1)–C(1′)	1·56 (2)		
I(1)–Ag(1)–I(2)	105·9 (1)	I(2)–Ag(3)–I(3)	108·3 (1)
I(1)–Ag(1)–I(2′)	113·4 (1)	I(2)–Ag(3)–I(4)	112·6 (1)
I(1)–Ag(1)–I(3′)	117·9 (1)	I(2)–Ag(3)–I(4′)	107·3 (1)
I(2)–Ag(1)–I(2′)	101·9 (1)	I(3)–Ag(3)–I(4)	112·7 (1)
I(2)–Ag(1)–I(3′)	106·4 (1)	I(3)–Ag(3)–I(4′)	112·0 (1)
I(2′)–Ag(1)–I(3′)	109·7 (1)	I(4)–Ag(3)–I(4′)	103·7 (1)
I(1)–Ag(2)–I(2)	114·6 (1)		
I(1)–Ag(2)–I(3)	123·1 (1)	C(1)–N(1)–C(2)	107·9 (2)
I(1)–Ag(2)–I(4)	102·9 (1)	C(1)–N(1)–C(3)	110·2 (2)
I(2)–Ag(2)–I(3)	104·9 (1)	C(1)–N(1)–C(4)	112·3 (1)
I(2)–Ag(2)–I(4)	108·9 (1)	C(2)–N(1)–C(3)	109·1 (1)
I(3)–Ag(2)–I(4)	100·9 (1)	C(2)–N(1)–C(4)	110·0 (1)
		C(3)–N(1)–C(4)	107·2 (1)
		N(1)–C(1)–C(1′)	111·8 (1)

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