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Disilver Tetraiodide–Hexamethylethylenediamine

By M. M. Thackeray and J. Coetzer

National Physical Research Laboratory, CSIR, P.O. Box 395, Pretoria 0001, *South Africa*

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Abstract. $Ag_2I_4C_8H_{22}N_2$, orthorhombic, *Pnnm*, $a = 14{\cdot}19$ (2), $b = 9{\cdot}72$ (1), $c = 7{\cdot}13$ (1) Å, $D_c = 2{\cdot}94$ g cm^{-3}, $Z = 2$, $R = 0{\cdot}058$ for 908 reflexions. The Ag^+ ions are situated at the centres of edge-sharing I^- tetrahedra. The tetrahedra are arranged in chains parallel to **c**.

Introduction. Crystals of $Ag_2I_4[(CH_3)_3$–N–$(CH_2)_2$–N–$(CH_3)_3]$ were obtained as well faceted needles. A $0{\cdot}06 \times 0{\cdot}06 \times 0{\cdot}11$ mm crystal was selected for data collection. The intensities of 951 reflexions were measured in the range $3° \leq \theta \leq 25°$ on a Philips PW1100 diffractometer with graphite-monochromated Mo $K\alpha$ ($\lambda = 0{\cdot}7107$ Å) radiation. The ω–2θ scan technique was employed, the scan width being $1{\cdot}2°\theta$ and scan speed $0{\cdot}04°\theta$ s^{-1}. 43 reflexions were considered to be unobserved according to the criterion $I(\mathrm{rel}) < 1{\cdot}65\sigma(I)$, where $\sigma(I) = [(0{\cdot}02S)^2 + S + B]^{1/2}$, S = scan count and B = background count. The intensities were corrected for Lorentz and polarization effects but not for absorption ($\mu = 82{\cdot}8$ cm^{-1}). Systematic absences $0kl$ for $k + l \neq 2n$ and $h0l$ for $h + l \neq 2n$ indicated space groups *Pnn*2 or *Pnnm*. The space group was assigned as *Pnnm* following the final structural analysis. The I^- and Ag^+ ion positions were obtained from a Patterson map. The non-hydrogen atoms were located from a difference map. After several cycles of isotropic and anisotropic refinement, R converged to 0·058. The shift/error values at this stage were $< 0{\cdot}1$ for all parameters. Unit weights were applied. Scattering factors for Ag^+, I^-, C and N were those of Cromer & Mann (1968). All computations were carried out with X-RAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Final atomic and thermal parameters are listed in Table 1. Bond lengths and angles are given in Table 2. The labelling scheme is shown in Fig. 1.*

Discussion. Numerous solid compounds (Geller & Owens, 1972; Owens, 1970; Owens & Argue, 1967) containing AgI have been reported, several of which

Table 1. *Final positional* (× 10⁴) *and thermal* (× 10³) *parameters*

Anisotropic temperature factors are of the form:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\right].$$

	Position	x	y	z	U_{11}	U_{22}	U_{23}	U_{12}	U_{13}	U_{23}
I(2)	4(*g*)	443 (2)	2305 (2)	0	101 (2)	36 (1)	30 (1)	−14 (1)	0	0
I(2)	4(*g*)	1443 (1)	−592 (2)	0	43 (1)	40 (1)	44 (1)	0 (1)	0	0
Ag	4(*e*)	0	0	2326 (3)	71 (1)	50 (1)	46 (1)	−4 (1)	0	0
N	4(*g*)	1338 (15)	4750 (19)	5000 (0)	46 (12)	18 (10)	89 (18)	7 (9)	0	0
C(1)	4(*g*)	306 (16)	4346 (23)	5000 (0)	26 (11)	25 (12)	98 (22)	4 (10)	0	0
C(2)	4(*h*)	1597 (14)	5564 (18)	3264 (23)	72 (12)	50 (10)	27 (9)	2 (9)	19 (9)	6 (8)
C(3)	4(*g*)	1926 (24)	3415 (29)	5000 (0)	56 (20)	23 (14)	303 (70)	24 (14)	0	0

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

Symmetry code	*a*	*b*	*c*	*d*
Rotation matrix with translation vector in 12ths	$\begin{matrix}-1 & 0 & 0\\ 0 & -1 & 0\\ 0 & 0 & -1\end{matrix}\begin{bmatrix}0\\0\\0\end{bmatrix}$	$\begin{matrix}-1 & 0 & 0\\ 0 & -1 & 0\\ 0 & 0 & -1\end{matrix}\begin{bmatrix}0\\0\\12\end{bmatrix}$	$\begin{matrix}-1 & 0 & 0\\ 0 & -1 & 0\\ 0 & 0 & -1\end{matrix}\begin{bmatrix}0\\12\\12\end{bmatrix}$	$\begin{matrix}1 & 0 & 0\\ 0 & 1 & 0\\ 0 & 0 & -1\end{matrix}\begin{bmatrix}0\\0\\12\end{bmatrix}$

(*a*) Iodide tetrahedron

I(1)—I(1)*a*	4·655 (5)	Ag—I(1)*a*	2·857 (3)
I(1)—I(2)	4·759 (4)	Ag—I(2)*b*	2·856 (3)
I(1)—I(2)*b*	4·759 (5)	I(1)—Ag–I(1)*a*	109·1 (1)
I(2)—I(1)*a*	4·759 (5)	I(1)—Ag–I(2)	112·8 (1)
I(2)—I(2)*b*	4·254 (6)	I(1)—Ag–I(2)*b*	112·8 (1)
I(1)*a*–I(2)*b*	4·759 (4)	I(2)—Ag–I(1)*a*	112·8 (1)
Ag—I(1)	2·857 (3)	I(2)—Ag–I(2)*b*	96·3 (1)
Ag—I(2)	2·856 (3)	I(1)*a*–Ag–I(2)*b*	112·8 (1)

(*b*) Amine chain

N—C(1)	1·52 (3)	C(1)—N—C(2)	111·7 (12)
N—C(2)	1·51 (2)	C(1)—N—C(2)*d*	111·7 (12)
N—C(3)	1·54 (4)	C(1)—N—C(3)	107·7 (18)
C(1)–C(1)*c*	1·54 (3)	C(2)—N—C(2)*d*	109·6 (15)
		C(2)—N—C(3)	108·0 (13)
		C(2)*d*–N—C(3)	108·0 (13)
		N—C(1)–C(1)*c*	109·3 (17)

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31047 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

have conductivities approaching those of liquid electrolytes. When AgI is reacted with hexamethylethylenediamine diiodide, several compounds are formed according to the reaction (Coetzer & Thackeray, 1975)

$$n\mathrm{AgI} + \mathrm{C_8H_{22}N_2I_2} \rightarrow \mathrm{Ag}_n\mathrm{I}_{n+2}\mathrm{C_8H_{22}N_2}\,.$$

An investigation into the conductive properties and crystal structures of these compounds is in progress at this laboratory. Here we report the structure of the compound $Ag_2I_4C_8H_{22}N_2$ in which the AgI concentration is 67 mol.%.

The structural features of the compound are illustrated in Fig. 1. The I^- ions form tetrahedra, each of which contains an Ag^+ ion at its centre. The mean Ag–I distance within each tetrahedron is 2·857 (3) Å. The tetrahedral bond angles are listed in Table 2.

Each I^- tetrahedron shares two of its edges to form chains parallel to **c**. The Ag^+ ions at the centres of the tetrahedra are separated by 3·81 Å.

The amine chain lies along **a** on a mirror plane at $z=\frac{1}{2}$. It is also situated about a symmetry centre at $(0,\frac{1}{2},\frac{1}{2})$. The bond lengths and angles within the amine chain are normal (Table 2).

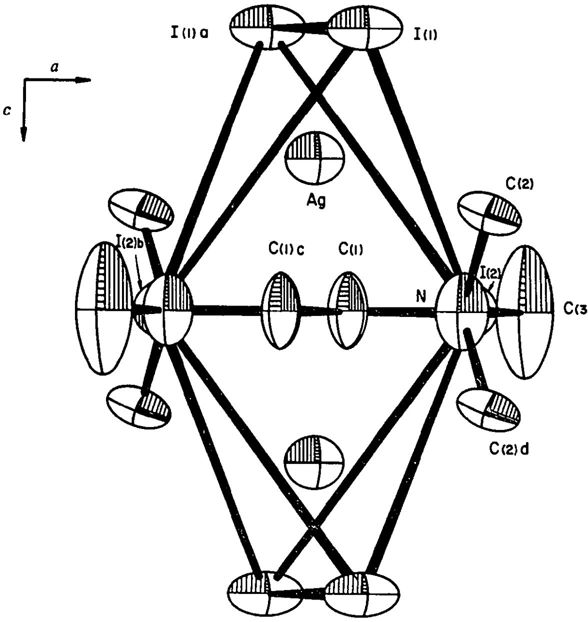

Fig. 1. [010] projection of the structure. The symmetry code for the atoms is given in Table 2.

References

Coetzer, J. & Thackeray, M. M. (1975). Results to be published elsewhere.

Cromer, D. T. & Mann, J. (1968). *Acta Cryst.* **A24**, 321–324.

Geller, S. & Owens, B. B. (1972). *J. Phys. Chem. Solids*, **33**, 1241–1250.

Owens, B. B. (1970). *J. Electrochem. Soc.* **117**, 1536–1539.

Owens, B. B. & Argue, G. R. (1967). *Science*, **157**, 308–309.

Stewart, J. M., Kruger, G. J., Ammon, H., Dickinson, C. H. & Hall, S. R. (1972). Univ. of Maryland Comput. Sci. Tech. Rep. TR-192.

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2-(Nitromethylene)piperidine

By J. J. de Boer and D. Bright

Koninklijke/Shell Laboratorium Amsterdam (Shell Research B.V.), Badhuisweg 3,
Amsterdam-Noord, The Netherlands

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Abstract. $C_6H_{10}O_2N_2$, M.W. 142·2, orthorhombic, $Pbca$, $a=6{\cdot}963(1)$, $b=10{\cdot}459(3)$, $c=19{\cdot}717(7)$ Å, $Z=8$, $D_x=1{\cdot}30$ g cm^{-3}, $\mu(\mathrm{Cu}\ K\alpha)=0{\cdot}845$ mm^{-1}. The pale-yellow transparent crystals are elongated along [001]. The nitro group is *cis* to the ring N atom.

Introduction. Crystals were supplied by Dr C. Boyce. Preliminary precession photographs of two crystals in different orientations showed the extinction rules: $0kl$: $k=2n$, $h0l$: $l=2n$, $hk0$: $h=2n$, unambiguously defining the space group as $Pbca$. A crystal with approximate dimensions 0·20 × 0·25 × 0·18 mm was glued to a Lindemann glass capillary and mounted on a Nonius automatic three-circle diffractometer equipped with scintillation counter and pulse-height discriminator. The c axis coincided with the φ axis of the diffractometer. Cell dimensions were obtained from a least-squares fit on θ, $-\theta$ values of 13 reflexions with $\theta \le 15°$, measured with Cu $K\alpha$ radiation ($\lambda=1{\cdot}54182$ Å). In the range $0 \le \sin\theta/\lambda \le 0{\cdot}497$ Å^{-1}, 564 unique reflexions, out of a possible 905, were observed using Ni-filtered Cu $K\alpha$ radiation and a $\theta, 2\theta$ scan. Attenuation filters were automatically inserted if the intensity of a reflexion exceeded 2000 c.p.s. A reflexion was considered to be observed when $\frac{1}{2}I>\sigma(I)=[C+B_1+B_2+(0{\cdot}05I)^2]^{1/2}$, in which $\sigma(I)$ is the standard deviation of