

the coordination found for Sr^{2+} or Ba^{2+} is eight, nine or twelve (Von der Mühl & Ravez, 1974; Von der Mühl, Daut & Ravez, 1973; Von der Mühl, Andersson & Galy, 1971; Abjean, Leblanc, De Pape & Ferey, 1985).

As could be expected, the Sr—F distances are shorter than the Ba—F ones, as shown in Table 3. The MF_{10} polyhedron is somewhat distorted, but nevertheless less so than in other fluorinated compounds (De Kozak, Leblanc, Samouël, Ferey & De Pape, 1981). In SrSbF_5 and BaSbF_5 two SbF_5 pyramids share one face [F(1), F(2), F(3)] with this polyhedron, whereas four other pyramids are linked to the polyhedron by one apex.

Every F^- ion exhibits a threefold coordination with the cations (1Sb^{3+} and 2M^{2+}), thus ensuring a strong connection in the three-dimensional network: the F(1) ions are tied to the M^{2+} ion along the z direction, whereas the F(2) and F(3) ions are bonded to this divalent cation along x and y respectively (Fig. 1*b*). Such an environment leads to the following electrostatic valence for fluorine: $e.v. = (1 \times \frac{3}{5}) + (2 \times \frac{2}{10}) = 1$. The identical result obtained for the three types of fluorine ions [F(1), F(2) and F(3)] is in good agreement with the counteraction observed in the interatomic distances: $\text{Sb—F(1)} < \text{Sb—F(2,3)}$ is correlated with $\text{M—F(1)} > \text{M—F(2,3)}$.

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Silver Dicyanamide, $\text{AgN}(\text{CN})_2$ – Orthorhombic Modification

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Abstract. $M_r = 173.9$, orthorhombic, $Pnma$, $a = 16.133$ (8), $b = 3.612$ (2), $c = 5.983$ (3) Å, $V/Z = 87.2$ (1) Å³, $Z = 4$, $D_x = 3.313$ (4) g cm⁻³, Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 456$ cm⁻¹, $F(000) = 320$, $T = 296$ (2) K, $R = 0.086$ for 331 reflections. The structure consists of infinite chains, —Ag—N≡C—N—C≡N—Ag—, running parallel to the a direction. This is similar to the arrangement found in the trigonal form previously reported, except that the 3_1 axis along the chain is replaced

here by a 2_1 axis. The interatomic distances and angles are not significantly different from those found in the trigonal form.

Introduction. The crystal structure of trigonal $\text{AgN}(\text{CN})_2$ was reported previously (Britton & Chow, 1977). At the time that that structure was determined, the first crystal examined was not identical with the trigonal crystals that comprised the bulk of the sample. The crystal was of poor quality and

was thrown out when a better (trigonal) crystal was found, but a later examination of the X-ray photographs showed that this first crystal appeared to be orthorhombic with a unit-cell volume approximately four times the volume of one formula unit occupied in the trigonal form. This strongly suggested that it was a polymorphic form. When this was realized, a search was made for more crystals of the orthorhombic form. This search was only marginally successful. The trigonal crystals grow as plates parallel to (001), which means that the orthorhombic crystals can easily be detected using a polarizing microscope. In the samples examined they occurred only as inclusions in trigonal plates and never appeared to be as much as 1% of the total sample. A number of recrystallizations were made, but the results were never better, and finally a small orthorhombic crystal was cut out of one of the trigonal plates and used for the diffraction studies. A better crystal would have been highly desirable but did not appear to be obtainable.

Experimental. A crystal 0.01 (or less) $\times 0.05 \times 0.10$ mm, mounted in air, was used for the data collection. Data were collected on an Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator. 21 reflections with $16 < \theta < 34^\circ$ were used to determine the cell parameters. Systematic extinctions ($0kl, k+l=2n+1; hk0, h=2n+1$) showed the space group possibilities to be $Pn2_1a$ (No. 33) or $Pnma$ (No. 62); the structure was successfully solved in $Pnma$. Cu radiation was used for the data collection in view of the very small size of the crystal; the greater intensity obtainable with the Cu radiation was felt to outweigh the greater absorption errors. Data were collected in the range $0 < \theta < 78^\circ$ for one quadrant (ranges: $h, -20$ to $20; k, 0$ to $4; l, 0$ to 7). 741 reflections were measured using ω - 2θ scans with 120 s scan times. The structure was solved using Patterson and Fourier syntheses, after which absorption corrections were made using the *DIFABS* program (Walker & Stuart, 1983); correction factors were 0.720 to 2.067. Equivalent reflections were combined ($R_{\text{int}} = 0.072$) to give 431 unique reflections of which the 331 with $I > \sigma(I)$ were used in the calculations. Three check reflections measured every 8000 s of exposure time showed no systematic change with time. The structure was refined with full-matrix least-squares refinement on F^2 s. All atoms were given anisotropic thermal parameters. Refinement converged with $R = 0.086, wR = 0.095$ and $S = 1.853; w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I_c) + (0.05I)^2$, where $\sigma(I_c)$ is the standard deviation in I based on counting statistics alone. The largest ratio of shift to e.s.d. in the final cycle of refinement was 0.02. The extreme variation from zero on the final difference Fourier synthesis was a peak $1.9 \text{ e } \text{\AA}^{-3}$ in height, less

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

$$B_{\text{eq}} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq} (\AA^2)
Ag	0.0865 (1)	$\frac{1}{2}$	0.1115 (3)	3.7 (1)
N1	0.210 (1)	$\frac{1}{2}$	-0.016 (4)	2 (1)
C1	0.283 (1)	$\frac{1}{2}$	-0.013 (4)	3 (1)
N0	0.361 (1)	$\frac{1}{2}$	-0.040 (4)	4 (1)
C2	0.413 (1)	$\frac{1}{2}$	0.123 (3)	1.9 (8)
N2	0.469 (1)	$\frac{1}{2}$	0.246 (4)	3 (1)

Table 2. Bond distances (\AA) and angles ($^\circ$)

	This work		Trigonal form*
	CN(1)	CN(2)	
N—C	1.28 (3)	1.29 (3)	1.29 (1)
C≡N	1.17 (3)	1.16 (3)	1.15 (1)
Ag...N	2.14 (2)	2.08 (2)	2.11 (1)
C—N—C	123 (2)		127 (2)
N—C≡N	172 (3)	170 (2)	170 (2)
Ag...N≡C	158 (2)	165 (2)	156 (2)
N...Ag...N	177 (1)		173 (1)

* Britton & Chow (1977).

than 1 \AA from the Ag position. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The computer programs used in the early stages of the calculations were from the Enraf-Nonius *Structure Determination Package* (Frenz, 1978), and in the later stages were from the *TEXRAY* package [Molecular Structure Corporation (1988)].

Discussion. The final positional parameters are given in Table 1.*

The atom labelling and thermal ellipsoids are shown in Fig. 1, the packing in Fig. 2. Bond distances and angles are given in Table 2, where they are compared with the corresponding distances and angles in the trigonal modification. There are no significant differences between the distances and angles in the two forms. The similarity goes further in that the arrangement around the Ag atoms is roughly octahedral in both cases with the four longer Ag...N distances in the trigonal form occurring at 2.91 and 3.08 \AA and in the orthorhombic form at 2.88 (2) and 2.98 (2) \AA . In the trigonal form these longer distances are all to terminal N atoms while in the orthorhombic form two are to terminal N2 atoms and two are to central N0 atoms. The essential

* Tables of anisotropic thermal parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53226 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

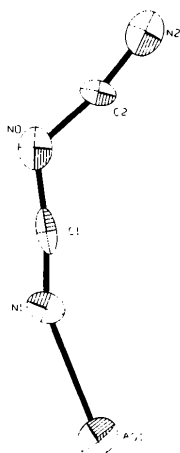


Fig. 1. Thermal ellipsoids (50% probability) for the atoms in the lower left corner of Fig. 2.

difference between the two forms is that the threefold spiral parallel to the 3_1 axis in the trigonal form is replaced by a twofold spiral parallel to the 2_1 axis in the orthorhombic form. The distance parallel to the spiral axis corresponding to one monomeric unit is $c/3$ (7.621 Å) in the trigonal form and $a/2$ (8.066 Å) in the orthorhombic form. The former is slightly shorter owing to the greater tilt of the dicyanamide group in the threefold spiral. The shortest Ag...Ag distances, a (3.601 Å) in the trigonal form and b (3.612 Å) in the orthorhombic form, are approximately equal. The molecular volumes, 87.2 (1) Å³ in the orthorhombic form vs 85.6 (1) Å³ in the trigonal form, show that in spite of similarities in the bonding, the packing is significantly more efficient in the trigonal form.

The thermal ellipsoid of C1 (Fig. 1) looks strange; this is almost certainly a consequence of the poor

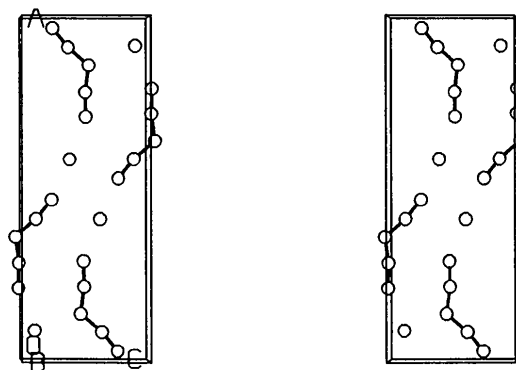


Fig. 2. The crystal structure of orthorhombic AgN(CN)₂.

quality of the crystal. If the refinement is carried out with all of the light atoms having isotropic temperature factors, convergence occurs with $R = 0.088$, $wR = 0.098$. Using Hamilton's (1965) test, $\mathcal{R}_{\text{exp}} = 1.03$ and $\mathcal{R}_{15,294,0.25} = 1.031$. Thus, the hypothesis that the temperature factors for the light atoms are isotropic can only be rejected at the 75% confidence level.

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Structure of Dipotassium Galactose 1-Phosphate Pentahydrate

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Abstract. $2\text{K}^+ \cdot \text{C}_6\text{H}_{11}\text{O}_6\text{P}^{2-} \cdot 5\text{H}_2\text{O}$, $M_r = 426.4$, monoclinic, $P2_1$, $a = 6.228$ (1), $b = 14.600$ (2), $c = 8.982$ (1) Å, $\beta = 102.01$ (1)°, $V = 798.4$ Å³, $Z = 2$, $D_m = 1.756$, $D_x = 1.773$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 7$ mm⁻¹, $F(000) = 444$, $T = 293$ K, $R = 0.033$,

0108-2701/90/122299-04\$03.00

$wR = 0.035$ for 1549 unique reflections with $I > 3\sigma(I)$. The sugar ring adopts a 4C_1 conformation. The conformation about the exocyclic C5—C6 bond is *gauche-trans*. The phosphate group exists as a dianion with charges localized on O7 and O8 atoms.

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