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Sodium Dicyanoargentate(I)

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Abstract. Na[Ag(CN)$_2$], $M_r = 182{\cdot}90$, monoclinic, $C2/c$, $a = 6{\cdot}572\,(1)$, $b = 3{\cdot}710\,(1)$, $c = 17{\cdot}346\,(2)$ Å, $\beta = 92{\cdot}05\,(1)^\circ$, $V = 422{\cdot}66$ Å^3, $Z = 4$, $D_x = 2{\cdot}874$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 4{\cdot}527$ mm^{-1}, $F(000) = 336$. The structure was refined to $R = 0{\cdot}072$, $wR = 0{\cdot}052$ for 293 absorption-corrected observed reflections. The Ag atoms of linear NC—Ag—CN groups are arranged in layers parallel to (001). The Na ions occupy octahedral holes between pseudohexagonal nitrogen layers with the sequence *ABAB*.

Experimental. This very stable compound is easily obtained by reaction of NaCN with AgCN in aqueous suspension. All attempts, however, to grow good-quality single crystals failed up to now, so that only powder diffraction data have been recorded in the literature. We have succeeded in growing single crystals by slow recrystallization from a 1:1 ethanol–water mixture. Unfortunately most of these crystals were also of poor quality, mainly because of intergrowth and a high degree of disorder. After inspection of a large number of crystals by microscopy and film methods a clear crystal fragment (30 × 30 × 60 μm) could be isolated, which proved suitable for a structure determination. Gandolfi and Guinier patterns showed the fragment to be the same phase as the other crystals and the bulk material.

For data collection, an Enraf–Nonius CAD-4 diffractometer was used (Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, graphite monochromator). Lattice parameters were refined from 2θ values of 25 reflections in the range 4·8–36°. Intensities measured for $2 \le \theta \le 20^\circ$; ω–2θ-scan technique (scan width $0{\cdot}85^\circ + 0{\cdot}34^\circ \tan\theta$). Three standard reflections indicated a 2·2% loss of intensity during data collection. Merging of the 1308 collected intensities [$(\sin\theta)_{\max}/\lambda = 0{\cdot}481$ Å; $-5 \le h \le +5$, $-5 \le k \le +5$, $-24 \le l \le +24$] gave 293 unique reflections ($R_{\text{int}} = 0{\cdot}031$) with $F > 3\sigma(F)$, which were considered as observed and used for all calculations (program system *SHELX76*; Sheldrick, 1976).

The structure was solved by standard direct methods. In least-squares refinement $|F|$ magnitudes were used to refine atomic coordinates and isotropic temperature factors. Convergence was obtained after a few cycles at $wR = 0{\cdot}08$. At this stage a numerical correction for absorption (program *DIFABS*; Walker & Stuart, 1983) was applied before performing the final refinement (anisotropic for Ag and Na, isotropic for C and N). Final $R = 0{\cdot}072$, $wR = 0{\cdot}052$, $w = 3{\cdot}28/\sigma^2(F)$. $(\Delta/\sigma)_{\max} < 0{\cdot}001$ in final refinement cycle, 20 parameters. Max. features in the final $\Delta\rho$ map $+2{\cdot}1$, $-1{\cdot}5$ e Å^{-3}. Atomic scattering factors and f', f'' values from *International Tables for X-ray Crystallography* (1974). Bond distances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances and important angles in Table 2.†

Table 1. *Atomic coordinates and isotropic temperature factors* (Å^2)

The equivalent isotropic U values for atoms refined anisotropically (designated by an asterisk) are defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
Ag	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{2}$	0·0496 (14)*
Na	0	−0·0389 (26)	$-\frac{1}{4}$	0·0314 (63)*
C	−0·3069 (27)	0·1077 (41)	−0·8911 (9)	0·0242 (48)
N	−0·3392 (22)	0·0322 (37)	−0·8282 (7)	0·0288 (39)

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

Ag—C	2·009 (16)	(×2)	C—Ag—C	180
—N	3·163 (12)	(×2)	N—C—Ag	178·8 (14)
—Ag	3·710	(×2)	N—Na—N	87·4 (5)
—Ag	3·773	(×4)		93·4 (4)
				179·2 (5)
C—N	1·153 (20)			95·5 (5)
				85·4 (5)
Na—N	2·532 (15)	(×2)		
—N	2·568 (14)	(×4)		

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51928 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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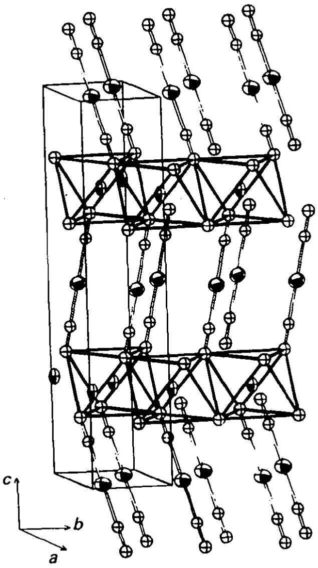

Fig. 1. *ORTEP* diagram (Johnson, 1976) of the crystal structure of $Na[Ag(CN)_2]$.

The structure (Fig. 1) comprises layers of linear NC—Ag—CN groups. These groups are canted by about 21° with respect to the *c* axis. In this way, pseudohexagonal layers of N atoms are formed with the sequence *ABAB*.... The Na ions occupy octahedral holes between the nitrogen layers.

Related literature. Surprisingly little is known about the crystal structures of dicyanoargentates. The few examples are $K_2Na[Ag(CN)_2]_3$ (Zabel, Kühnel & Range, 1989), $Ca[Ag(CN)_2]_2.2H_2O$, $Sr[Ag(CN)_2]_2.23H_2O$ (Range, Zabel, Meyer & Fischer, 1985) and, with complex cations, $[M(en)_3][Ag(CN)_2]_2$ and $[M(en)_2][Ag(CN)_2]_2$ (Kappenstein, Ouali, Guerin, Cernak & Chomic, 1988). The crystal structure of $K[Ag(CN)_2]$, proposed more than fifty years ago (Hoard, 1933), is at variance with spectroscopic evidence (Bottger, 1968) and still needs to be confirmed by modern techniques. A close relation exists between the structures of $Na[Ag(CN)_2]$ and $K[I(CN)_2]$ (Tebbe & Krauss, 1988).

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Structure of (2-Benzoxazolethiolato)(triphenylphosphine)gold(I)

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Abstract. $[Au(C_7H_4NOS)(C_{18}H_{15}P)]$, $M_r = 609.7$, monoclinic, $P2_1/n$, $a = 12.709$ (3), $b = 12.871$ (4), $c = 13.462$ (6) Å, $\beta = 94.64$ (3)°, $V = 2195$ (2) Å^3, $Z = 4$, $D_x = 1.84$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 68.7$ cm^{-1}, $F(000) = 1176$, $T = 296$ K, final $R = 0.033$ for 2823 unique observed reflections. The 2-benzoxazolethiolate ligand (Sboz), which contains three potential coordination sites (N, O and S), forms a linear complex with a P—Au—S angle of 176.43 (8)°. The Au—P and Au—S distances are 2.258 (2) and 2.299 (2) Å, respectively. This angle and these bond distances are similar to those of other phosphorous–gold–sulfur complexes such as the antiarthritic drug auranofin.

Experimental. Ph_3PAuCl (0.21 mmol) (De Stefano & Burmeister, 1971) was placed in a beaker containing 20 ml ethanol. NaOH (0.25 mmol) and then HSboz (0.26 mmol) (Fluka) were added and the solution stirred and heated. A powder formed, which was