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Redetermination of the Structure of Zinc Disilver(I) Tetrathiocyanate (at 178 K)

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Abstract

The structure of $ZnAg_2(SCN)_4$, previously reported in *Cc*, should be described in the centrosymmetric space group *C*2/*c*. The Zn atom occupies a special position on the twofold axis $\frac{1}{2}$, *y*, $\frac{1}{4}$. Both metal atoms are tetrahedrally coordinated (zinc by thiocyanate N, silver by S), although the coordination at silver is appreciably distorted.

Comment

In the course of our structural investigations of silver complexes with sulfur-containing ligands (Roesky, Schimkowiak, Meyer-Bäse & Jones, 1986, and references therein), our attention was drawn (Herbst-Irmer, 1990) to the structure of $ZnAg_2(SCN)_4$ (Fig. 1) (Shaofang, Meiyun & Jinling, 1982). The published space group was *Cc*, but an inspection of the coordinates suggested that the true space group might be *C*2/*c*. We therefore redetermined the structure, growing crystals as described in the original paper.

The structure can indeed be successfully refined in C2/*c*, whereby the Zn atom occupies a special position on a twofold axis. The Zn—N bonds are almost equal at 1.963, 1.965 (2) Å, in contrast to the spread of 1.87–2.06 Å in the previous determination; it is well known that apparent inequalities in bond lengths arise when a centrosymmetric structure is refined in a non-centrosymmetric space group (*e.g.* Ermer & Dunitz, 1970). The Ag—S bond lengths, 2.552–2.663 (2) Å, are far from equal in *C*2/*c*, but again the spread is less than in *Cc* (2.53–2.71 Å). Coordination at Ag^I is often irregular, as can also be seen from the bond angles 99.2–135.7 (1)°.

In other respects the structure is qualitatively as described by Lu *et al.* (1982). As expected, the softer metal Ag^I is coordinated by the softer donor S atoms. Each S atom coordinates two Ag atoms, but the angles at S(1), 100.7–113.0 (1)°, are much larger than those at S(2), 92.6–103.1 (1)°. A greater *p* character in the bonding at S(2) would account for the bond-length differences noted above. The C—N—Zn angles show appreciable deviations from linearity.

The thiocyanate groups bridge Zn and Ag centres to form a three-dimensional polymer, in which the

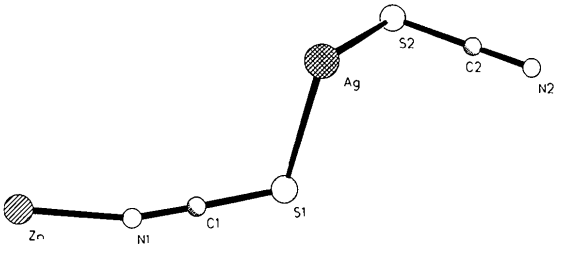

Fig. 1. The asymmetric unit of the title compound, showing the atom-numbering scheme.

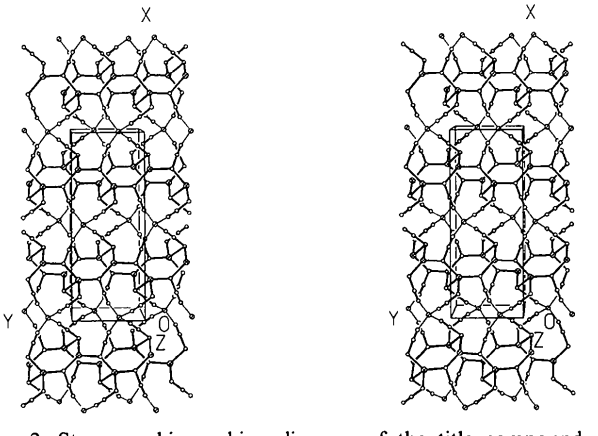

Fig. 2. Stereographic packing diagram of the title compound, viewed along the *z* axis.

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Zn atoms occupy the regions near $z = 0, 0.5, 1, \ldots$, and the Ag atoms those near $z = 0.25, 0.75, \ldots$ (Fig. 2).

Experimental

Crystal data

$ZnAg_2(SCN)_4$	$\theta = 10$–$11.5°$
$M_r = 513.4$	$V = 1170.0$ (7) Å^3
Monoclinic	$Z = 4$
$C2/c$	$D_x = 2.915$ Mg m^{-3}
$a = 19.720$ (9) Å	Mo $K\alpha$
$b = 7.703$ (3) Å	$\lambda = 0.71069$ Å
$c = 7.753$ (3) Å	$\mu = 6.0$ mm^{-1}
$\beta = 96.57$ (4)°	$T = 178$ K
Cell parameters from 49 reflections	Prism
	0.9 × 0.7 × 0.4 mm
	Colourless

Data collection

Siemens $R3$ diffractometer	$R_{int} = 0.04$
ω scans	$\theta_{max} = 27.5°$
Absorption correction: empirical ψ scans	$h = -25 \rightarrow 25$
	$k = 0 \rightarrow 10$
$T_{min} = 0.23$, $T_{max} = 0.99$	$l = 0 \rightarrow 10$
5383 measured reflections	3 standard reflections
1351 independent reflections	monitored every 147 reflections
1328 observed reflections $[F>4\sigma(F)]$	intensity variation: ±1.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.002$
Final $R = 0.026$	$\Delta\rho_{max} = 1.0$ e Å^{-3}
$wR = 0.030$	$\Delta\rho_{min} = -0.9$ e Å^{-3}
$S = 1.6$	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
1328 reflections	
70 parameters	
$w = 1/[\sigma^2(F)+0.0001F^2]$	

Extinction correction applied using $F_{corr} = F/[1+0.002xF^2/\sin 2\theta]^{1/4}$, with $x = 0.00201(9)$.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Ag	0.20526 (1)	0.09578 (2)	0.26763 (2)	0.0251 (1)
Zn	0.50000	0.16483 (4)	0.25000	0.0167 (1)
S(1)	0.29939 (3)	−0.11251 (6)	0.38966 (6)	0.0175 (1)
S(2)	0.13289 (3)	0.10224 (6)	0.53246 (6)	0.0192 (2)
N(1)	0.42556 (10)	0.01780 (22)	0.31417 (24)	0.0237 (5)
N(2)	0.04535 (10)	−0.17250 (21)	0.42132 (23)	0.0208 (5)
C(1)	0.37350 (11)	−0.03712 (22)	0.34171 (24)	0.0168 (5)
C(2)	0.08141 (11)	−0.05824 (25)	0.46642 (24)	0.0168 (5)

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

Ag—S(1)	2.552 (2)	Ag—S(2)	2.632 (2)
Ag—S(1^i)	2.553 (2)	Ag—S(2^i)	2.663 (2)
Zn—N(1)	1.963 (2)	Zn—N(2^{ii})	1.965 (2)
S(1)—C(1)	1.654 (2)	S(2)—C(2)	1.644 (2)
N(1)—C(1)	1.153 (3)	N(2)—C(2)	1.160 (3)
S(1)—Ag—S(2)	99.2 (1)	S(1)—Ag—S(1^i)	135.7 (1)
S(2)—Ag—S(1^i)	111.3 (1)	S(1)—Ag—S(2^i)	101.8 (1)
S(2)—Ag—S(2^i)	104.4 (1)	S(1^i)—Ag—S(2^i)	100.9 (1)
N(1)—Zn—N(1^{iii})	109.5 (1)	N(1)—Zn—N(2^{ii})	119.5 (1)
N(1)—Zn—N(2^i)	104.1 (1)	N(2^{ii})—Zn—N(2^i)	100.8 (1)
Ag—S(1)—C(1)	108.7 (1)	Ag—S(1)—Ag^{iv}	113.0 (1)
C(1)—S(1)—Ag^{iv}	100.7 (1)	Ag—S(2)—C(2)	96.5 (1)
Ag—S(2)—Ag^v	103.1 (1)	C(2)—S(2)—Ag^v	96.6 (1)
Zn—N(1)—C(1)	165.1 (2)	C(2)—N(2)—Zn^{vi}	154.6 (2)
S(1)—C(1)—N(1)	177.6 (2)	S(2)—C(2)—N(2)	179.3 (2)

Symmetry operators: (i) $0.5-x, 0.5+y, 0.5-z$; (ii) $0.5+x, 0.5+y, z$; (iii) $1-x, y, 0.5-z$; (iv) $0.5-x, -0.5+y, 0.5-z$; (v) $x, -y, 0.5+z$; (vi) $-0.5+x, -0.5+y, z$.

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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 55067 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1006]

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2-(*tert*-Butyl)-*cis*-5-(*p*-chlorophenylthio)-*trans*-5-methyl-1,3-dioxolan-4-one

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Abstract

The five-membered ring has a conformation intermediate between a twist form and an envelope with O1 deviating from the plane of the other four atoms. The *tert*-butyl and *p*-chlorophenylthio groups are both situated on the same side of the ring.

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