Refinement of the crystal structure of silver(I) thiocyanate, AgSCN

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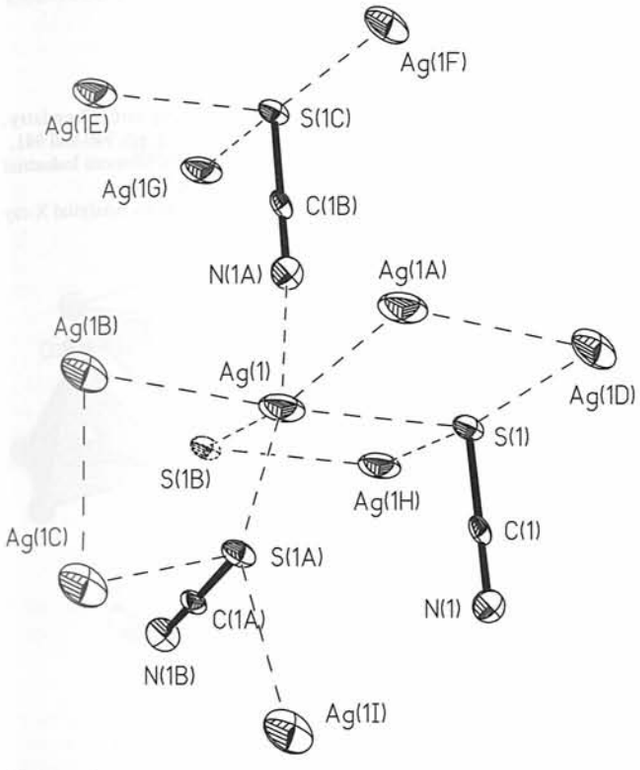

Abstract

AgCNS, monoclinic, *C*12/*c*1 (No. 15), $a = 8.792(5)$ Å, $b = 7.998(5)$ Å, $c = 8.207(5)$ Å, $\beta = 93.75(1)°$, $V = 575.9$ Å^3, $Z = 8$, $R_{gt}(F) = 0.049$, $wR_{ref}(F^2) = 0.086$, $T = 293$ K.

Source of material

All reagents and solvents were used as obtained without further purification. Crystals of AgSCN suitable for X-ray study can be prepared by two methods. In one of them, the commercial powder AgSCN (1 mmol, 166 mg) was dissolved in pyridine (5 ml), with stirring a water solution (5 ml) of excess NH_4SCN (15 mmol, 760 mg) was added. The large colorless cubic crystals crystallized at the bottom of the flask after the resulting clear solution was stood still overnight. The products were collected with filtration and washed with water, and dried in a vacuum desiccator over silica gel with the yield of 51%. Calc. for CNAgS: C, 7.24%; N, 8.44%; S, 19.32%. Found: C, 7.20%; N, 8.35%; S,19.22%.

Discussion

Many complexes containing AgSCN units have been reported [1]. Recently we prepared some complexes with the AgSCN blocks [2]. Reported in this paper is a more accurate refinement of a known structure reported by Lindqvist [3] in a different setting of the same space group *C*2/*c*.

The structure of the title complex is three-dimensional, the figure shows the coordination environment of the silver(I) atom. This structure consists of endless repeated (001) planes joined together through Ag—S bonds and weak Ag···Ag interactions (from 3.249(2) Å to 3.338(2) Å) between adjacent planes. Each Ag atom in the title complex has a coordination environment of T-shape fashion (the three angles at Ag for the T are 163.4(2), 92.0(2)° and 101.9(2)°), and is bound to two S atoms and one N atom of three thiocyanate ligands. All thiocyanates are 1,1,3-ligands [d(Ag1—S1A) = 2.474(2) Å and d(Ag1—S1) = 2.843(2) Å]. Two Ag atoms are bridged assymmetrically by the S atom of SCN ligands. The SCN^- groups are nearly linear (∠S1–C1–N1 = 179.6(5)°). Five Ag atoms, two thiocyanate ligands and four S atoms of additional SCN^- groups form the fourteen-membered rings which are recognizable as a typical fragment of the title complex. These rings share common edges to form a two-dimensional infinite network. The joining of these layers along the [100] direction leads to a three-dimensional structure clearly different from the long-recognized [4] one-dimensional zig-zag chain in AgSCN. The SCN^- ligand in the title complex acts as a μ_3 ligand, forming a covalent 3D framework. One of the weak Ag—S bonds in our structure (2.843(2) Å) is significantly shorter than that in Lindqvist's AgSCN (2.89(1) Å). The length of the other weak Ag—S bond is similar (3.001(2) Å as compared to 3.00(1) Å in [3]).

Table 1. Data collection and handling.

Crystal:	colorless cubic, size 0.5 × 0.5 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	73.95 cm^{-1}
Diffractometer, scan mode:	Siemens R3m, ω/θ
$2\theta_{max}$:	56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	740, 697
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 409
$N(param)_{refined}$:	38
Programs:	SHELXTL [5], SHELXTL-plus [6]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	8*f*	0.10606(7)	0.35198(8)	0.09516(8)	0.0667(4)	0.0571(4)	0.0647(4)	−0.0214(3)	−0.0262(3)	−0.0124(4)
S(1)	8*f*	0.2347(2)	0.4679(2)	−0.1930(2)	0.0392(9)	0.0247(8)	0.0321(9)	−0.0029(7)	−0.0132(8)	0.0006(7)
C(1)	8*f*	0.3547(7)	0.6030(7)	−0.1064(7)	0.037(3)	0.023(3)	0.018(3)	0.005(3)	−0.004(3)	0.007(3)
N(1)	8*f*	0.4404(7)	0.7007(6)	−0.0439(7)	0.055(3)	0.021(3)	0.042(3)	−0.005(3)	−0.003(3)	0.002(3)

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