

On the Cubane *versus* the Stella Quadrangula Structure. The Crystal Structure of Tetrakis[iodo- (tetrahydrothiophene)silver(I)]

BERTIL NORÉN^a and ÅKE OSKARSSON^b

^a Inorganic Chemistry 1, Chemical Center, University of Lund, Box 124, S-221 00 Lund, Sweden and ^b Department of Chemistry, University College of Sundsvall/Härnösand, Box 860, S-851 24 Sundsvall, Sweden

The crystal structure of $[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\}\text{I}]_4$ has been determined from X-ray intensity data collected at 200 K with a CAD 4 diffractometer. The space group is $P\bar{1}$ with $Z=2$; $a=9.673(3)$, $b=10.684(3)$, $c=16.574(8)$ Å, $\alpha=91.73(3)$, $\beta=104.07(3)$, $\gamma=110.62(3)^\circ$. The refinement, based on 3179 reflections and 173 parameters, converged to $R=0.085$. The compound has a tetrameric structure with a concave coordination polyhedron, a stella quadrangula. Each Ag coordinates three I and one S in distorted tetrahedral geometry. The tetrahedra share edges in such a way that an empty central tetrahedron is formed. The Ag–S distances are in the range 2.53–2.56 Å, Ag–I 2.83–2.96 and Ag–Ag 2.96–3.19 Å. The shorter of the Ag–Ag distances might indicate metal-metal interactions. There are only van der Waals bonds between the tetrameric complexes. From a literature survey of other Ag-tetramers it is concluded that the iodide compounds are close to the stella quadrangula structure while the bromide and chloride compounds could be described as cubane structures.

The monovalent oxidation states of the coinage metals, copper, silver and gold, are stabilized in solvates coordinating via N or, more markedly, S.¹ In the formation of solid solvates of the type $M_mL_lX_m$, where M is the metal, L the solvate molecule and X the monovalent anion, there is a competition between the solvate molecule and the anion for the coordination sites around M. The present investigation is part of a systematic study of the coordination geometry in silver(I)-tetrahydrothiophene systems. The nature of the anion is systematically varied from one with very poor donor properties, *e.g.* BF_4^- ,² to such with good donor properties, *e.g.* Γ^- . The crystal structure of $[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\}\text{I}]_4$ is reported below. The structure turns out to have an Ag_4I_4 -core, similar to those observed in compounds with solvate molecules coordinating via N^3 or P .^{4–8} These structures are described as either cubane or cubane like. However, it will be shown below that such tetrameric structures can be divided into cubane like, with long metal-metal distances, and stella-quadrangula structures, with short metal-metal distances.

Table 1. Crystal data, $[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\text{I}\}_4]$.

Space group $P\bar{1}$	
$a = 9.673(3) \text{ \AA}$	$\alpha = 91.73(3)^\circ$
$b = 10.684(3) \text{ \AA}$	$\beta = 104.07(3)^\circ$
$c = 16.574(8) \text{ \AA}$	$\gamma = 110.62(3)^\circ$
$V = 1542 \text{ \AA}^3$	mol wt = 1291.8
$\mu = 67.26 \text{ cm}^{-1}$	ρ (calc) = 2.78 g cm^{-3}
$Z = 2$	

EXPERIMENTAL

$[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\text{I}\}_4]$ was prepared from a solution of AgI in tetrahydrothiophene (THT), saturated at room temperature. Single crystals were obtained by cooling to -15°C . In air at room temperature the initially transparent crystals gradually become white and non-transparent. Most probably THT slowly evaporates causing a decomposition of the crystals. These were analysed for all of the elements constituting the compound. Following results were obtained (calculated values are given in parenthesis): Ag, $33.36 \pm 0.04\%$ (33.40); I, $37.6 \pm 0.1\%$ (39.30); S, $9.94 \pm 0.06\%$ (9.93); C, $14.9 \pm 0.1\%$ (14.88); H, $2.35 \pm 0.03\%$ (2.50).

A single crystal with the dimensions $0.26 \times 0.38 \times 0.16 \text{ mm}^3$ was used for the data collection on an Enraf-Nonius CAD-4 diffractometer employing graphite-monochromatized $\text{MoK}\alpha$ -radiation ($\lambda = 0.7107 \text{ \AA}$). Since the crystals decompose when exposed to air at room temperature all data were collected at 200 K with the aid of a nitrogen gas blower device.⁹ Laue symmetry $\bar{1}$ give $P1$ or $P\bar{1}$ as possible choices for the space group. Cell dimensions (Table 1) were obtained by least-squares calculations of 44 θ -values determined as $\theta_{hkl} = (\omega_{hkl} - \omega_{\bar{h}\bar{k}\bar{l}})/2$. The CAD-4 diffractometer in the bisecting mode and measuring $\omega_{\bar{h}\bar{k}\bar{l}}$ at a negative θ angle, was used. The intensity of 4491 reflections in one half of the reflection sphere obeying $3^\circ < \theta < 23^\circ$ were measured with the ω - 2θ scan technique ($\Delta\omega = 1.1 + 0.5 \tan\theta$). The scan interval was extended 25% at both ends for the background measurement. The ratio $\sigma(I)/I$ requested in a scan was 0.030 and the maximum recording time was 90 s. Three standard reflections were recorded at regular intervals. No systematic variation in their intensities were obtained. I and $\sigma(I)$ were corrected for Lorentz, polarization and absorption

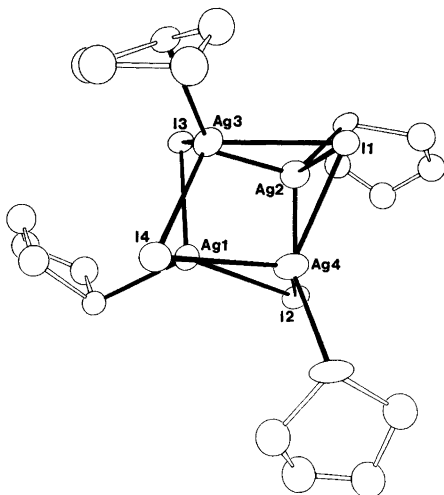


Fig. 1. A perspective view of the tetrameric unit $[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\text{I}\}_4]$. The hydrogen atoms have been omitted.

Table 2. Final positional and thermal parameters for the nonhydrogen atoms. Estimated standard deviations in the last significant digit(s) are given in parenthesis. The positions of the hydrogen atoms are not included. They were calculated assuming tetrahedral geometry around carbon, with the distance C-H=1.00 Å.

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	$U_{\text{iso}} \cdot 10^2/\text{Å}^2$	Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	$U_{\text{iso}} \cdot 10^2/\text{Å}^2$
Ag1	0.5296(2)	0.3324(2)	0.6582(1)	3.88(8) ^a	C21	-0.038(4)	-0.125(3)	0.578(2)	6.5(9)
Ag2	0.3041(2)	0.0798(2)	0.7195(1)	4.42(8) ^a	C22	-0.156(4)	-0.086(4)	0.601(2)	7.6(1.0)
Ag3	0.6395(2)	0.1710(2)	0.7951(1)	3.83(7) ^a	C23	-0.192(4)	-0.148(4)	0.677(2)	7.1(9)
Ag4	0.4716(2)	0.3665(2)	0.8291(1)	4.35(8) ^a	C24	-0.057(3)	-0.146(3)	0.739(2)	4.4(6)
I1	0.4215(2)	0.1207(2)	0.8984(1)	3.59(6) ^a	S3	0.8395(7)	0.0802(6)	0.8736(4)	3.68(23) ^a
I2	0.2364(2)	0.3151(2)	0.6693(1)	3.52(6) ^a	C31	1.021(3)	0.211(3)	0.880(2)	4.6(6)
I3	0.5096(2)	0.0560(2)	0.6241(1)	3.40(6) ^a	C32	1.085(5)	0.279(5)	0.967(3)	9.9(1.3)
I4	0.7853(2)	0.4599(2)	0.8110(1)	3.94(6) ^a	C33	0.990(7)	0.221(6)	1.016(4)	13.3(1.8)
S1	0.6643(7)	0.4609(6)	0.5548(4)	3.97(25) ^a	C34	0.856(3)	0.117(3)	0.981(2)	4.5(6)
C11	0.602(3)	0.347(3)	0.460(2)	3.8(6)	S4	0.4879(8)	0.5801(7)	0.9076(4)	4.59(27) ^a
C12	0.746(3)	0.338(2)	0.442(1)	3.3(5)	C41	0.554(4)	0.712(4)	0.851(2)	7.2(9)
C13	0.843(3)	0.337(3)	0.528(2)	5.0(7)	C42	0.424(4)	0.737(4)	0.802(2)	7.4(1.0)
C14	0.855(4)	0.447(3)	0.586(2)	6.5(8)	C43	0.305(4)	0.711(4)	0.844(2)	7.1(9)
S2	0.0803(7)	-0.1463(7)	0.6757(4)	4.02(25) ^a	C44	0.306(4)	0.590(4)	0.894(2)	6.8(9)

^a U_{iso} has been calculated from the average of the anisotropic temperature factor over all directions.

Table 3. Summary of important distances (Å) and angles (°). Estimated standard deviations in the last significant digits(s) are given in parenthesis.

A. Coordination geometry around the Ag-atoms					
Ag1-I2	2.829(2)	I2-Ag1-I3	106.47(8)	Ag3-I1	2.946(3)
Ag1-I3	2.919(3)	I2-Ag1-I4	115.75(8)	Ag3-I3	2.844(3)
Ag1-I4	2.959(3)	I3-Ag1-I4	107.23(8)	Ag3-I4	2.879(3)
Ag1-S1	2.547(7)	S1-Ag1-I2	124.35(16)	Ag3-S3	2.561(6)
		S1-Ag1-I3	104.99(16)		
		S1-Ag1-I4	96.66(16)		
Ag2-I1	2.864(3)	I1-Ag2-I2	107.68(9)	Ag4-I1	2.830(3)
Ag2-I2	2.909(3)	I1-Ag2-I3	116.48(8)	Ag4-I2	2.931(3)
Ag2-I3	2.894(3)	I2-Ag2-I3	105.01(8)	Ag4-I4	2.936(3)
Ag2-S2	2.548(7)	S2-Ag2-I1	109.03(18)	Ag4-S4	2.528(7)
		S2-Ag2-I2	116.02(16)		
		S2-Ag2-I3	102.88(17)		
B. Intracluster parameters					
Ag1-Ag2	3.189(3)	I1-I2	4.661(3)	S1-S2	7.649(4)
Ag1-Ag3	3.099(3)	I1-I3	4.897(3)	S1-S3	7.009(4)
Ag1-Ag4	3.054(3)	I1-I4	4.629(3)	S1-S4	6.683(4)
Ag2-Ag3	2.961(3)	I2-I3	4.604(2)	S2-S3	6.772(4)
Ag2-Ag4	3.169(3)	I2-I4	4.902(3)	S2-S4	7.734(4)
Ag3-Ag4	3.174(3)	I3-I4	4.732(3)	S3-S4	7.346(4)
Ag2-I1-Ag3	61.25(7)	Ag1-I3-Ag2	66.55(7)		
Ag2-I1-Ag4	67.62(8)	Ag1-I3-Ag3	65.05(7)		
Ag3-I1-Ag4	66.64(7)	Ag2-I3-Ag3	62.12(7)		
Ag1-I2-Ag2	67.52(7)	Ag1-I4-Ag3	64.10(7)		
Ag1-I2-Ag4	64.00(7)	Ag1-I4-Ag4	62.38(7)		
Ag2-I2-Ag4	65.71(7)	Ag3-I4-Ag4	66.16(7)		
C. Geometry of the tetrahydrothiophene molecules					
S1-C11	1.80(3)	S1-C11-C12	106(2)	S3-C31	1.80(3)
C11-C12	1.53(3)	C11-C12-C13	103(2)	C31-C32	1.47(5)
C12-C13	1.51(4)	C12-C13-C14	111(2)	C32-C33	1.37(7)
C13-C14	1.44(4)	C13-C14-S1	105(2)	C33-C34	1.36(7)
C14-S1	1.85(3)	C14-S1-C11	93(1)	C34-S3	1.77(3)
S2-C21	1.81(3)	S2-C21-C22	106(2)	S4-C41	1.73(4)
C21-C22	1.47(5)	C21-C22-C23	110(3)	C41-C42	1.44(5)
C22-C23	1.51(5)	C22-C23-C24	112(3)	C42-C43	1.45(5)
C23-C24	1.44(4)	C23-C24-S2	104(2)	C43-C44	1.56(5)
C24-S2	1.88(3)	C24-S2-C21	95(1)	C44-S4	1.76(3)
				S3-C31-C32	108(2)
				C31-C32-C33	111(4)
				C32-C33-C34	119(5)
				C33-C34-S3	109(3)
				C34-S3-C31	93(1)
				S4-C41-C42	108(3)
				C41-C42-C43	111(3)
				C42-C43-C44	107(3)
				C43-C44-S4	107(2)
				C44-S4-C41	95(2)

effects. The transmission factors evaluated by numerical integration varied in the interval 0.218–0.333. Only the 3179 reflections with $I > 3\sigma(I)$ were considered significantly different from the background and used in the calculations.

The structure was solved by direct¹⁰ and difference Fourier methods. The atomic parameters of the non-hydrogen atoms were refined in the space group $P\bar{1}$ using full-matrix least-squares. The positions of the H-atoms were calculated from geometrical criteria and included in the structure factor calculations. Neither positional nor temperature parameters ($U_{\text{iso}}=0.10 \text{ \AA}^2$) were refined for the H-atoms. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ with weights $w=[(\sigma_c^2/4|F_o|)^2 + (0.08|F_o|)^2 + 3.0]^{-1}$. In the final refinement, anisotropic temperature factors were applied to Ag, I and S. The total number of parameters refined was 173. Scattering factors with corrections for anomalous dispersion were taken from International Tables for X-ray Crystallography.¹¹ The final agreement indices $R=\Sigma(|F_o|-|F_c|/\Sigma|F_o|)$ and $R_w=[\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2}$ were 0.085 and 0.109, respectively. A σR plot resulted in an approximately straight line with slope 0.86 and intercept -0.11 . The largest residual electron densities; 3.4, 2.8 and $1.4 \text{ e}^-/\text{\AA}^3$, are located in the vicinities of I3, I4 and Ag2, respectively. Computer programs used are those compiled and amended by Lundgren.¹² Tables of $|F_o|$, $\sigma(|F_o|)$, $|F_c|$ are available on request from one of the authors (B. Norén). The final positional and thermal parameters are given in Table 2 and selected interatomic distances and angles in Table 3.

DESCRIPTION OF THE STRUCTURE

The crystal structure $[\text{Ag}\{\overline{\text{S}(\text{CH}_2)_3\text{CH}_2}\text{I}]_4$ is composed of discrete molecules separated by van der Waals distances. The molecular structure (Fig. 1) is closely related to other tetrameric silver halide solvates.³⁻⁸ The cage-like structure is composed of four silver- and four iodine atoms situated at alternate corners of a highly distorted cube with each silver atom further coordinating one THT-ligand. Each silver atom thus coordinates three iodine atoms and one sulfur atom in a distorted tetrahedral fashion (Table 3). The I–Ag–I angles are in the range $105.0\text{--}116.5^\circ$ (average 109.9°) and the S–Ag–I angles in the range $96.7\text{--}124.4^\circ$ (average 108.9°). The molecular structure may thus be described as composed of four AgI_3S -tetrahedra sharing edges in such a way that an empty central tetrahedron is formed (Fig. 2). The resulting star-like structure is called a *stella-quadrangula*, and it has also been observed in alloys.¹³ The symmetry of an ideal *stella-quadrangula* is T_d . However, the distortion from the T_d -symmetry is highly significant judged from the variation of the

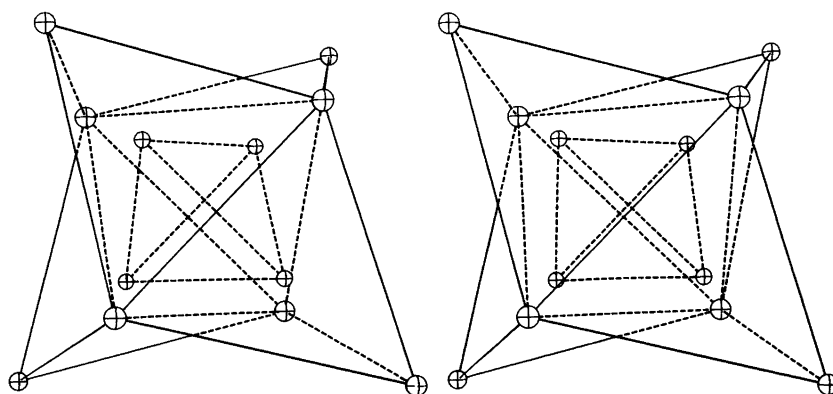


Fig. 2. A stereoscopic view of an ideal *stella quadrangula*.

bond angles around Ag as well as the variations of other intramolecular parameters such as the Ag–I–Ag angles, the Ag⋯Ag, I⋯I and the S⋯S distances (Table 3). The shortest Ag⋯Ag distance is only 0.08 Å longer than the Ag–Ag distance of 2.883 Å observed in silver metal.¹⁴ Metal-metal interactions cannot be ruled out (*vide infra*) and it is thus justified to call this polynuclear complex a cluster. The shortest intracuster I⋯I distance (4.604 Å) is 0.41 Å longer than the shortest intercluster I⋯I distance (4.192 Å), suggesting that intramolecular repulsion, I⋯I, should be of little importance in determining the geometry of the cluster.

The bond angles Ag–S–C are in the range 103–112°. Evidently the bonding around the S-atoms is close to tetrahedral, as is also found in bis(tetrahydrothiophene)silver(I)-tetrafluoroborate(III)² and in iodo(tetrahydrothiophene)gold(I).¹⁵

The bond lengths and angles of the coordinated THT molecules do not differ significantly from those found in the molecule in the gaseous phase.¹⁶

DISCUSSION

The first systematic study on star-like solids was made by Kepler.¹⁷ He called the eight-pointed star formed by eight intersecting triangles a stella octangula.^{17,18} An alternate description of a stella octangula is a central octahedron sharing its faces with eight tetrahedra. Therefore the four-pointed star composed of a central tetrahedron sharing its faces with four tetrahedra may be called a stella quadrangula (Fig. 2).

Let the length of the edges in an ideal stella quadrangula be a . Then it can be shown that the distances between two centers of gravity in the tetrahedra surrounding the central one are $2a/3$ and the distances between two top apices are $5a/3$. In a discussion of molecular geometries a stella-quadrangula may alternately be described as composed of three tetrahedra with the length of the edges being $2a/3$, a and $5a/3$, respectively, and with a common center of gravity. The smallest tetrahedron (formed by Ag) and the largest (formed by S) are positioned in a parallel fashion while the intermediate tetrahedron (formed by X) is positioned antiparallel to the other two. An expansion or contraction of these tetrahedra destroys the stella-quadrangula structure but does not violate the T_d -symmetry. If the two smallest tetrahedra are equal in size the cubane structure is obtained. Therefore we suggest that the ratio between the lengths of the edges in these tetrahedra should be used for classification, *e.g.* complexes with a ratio near $2/3$ are stella quadrangula – and those with a ratio near 1 are cubane structures.

Table 4 gives some characteristics of the coordination geometry in some tetrameric silver halide solvates. From the ratios $d_{\text{Ag-Ag}}/d_{\text{X-X}}$ it may be concluded that the bromide and chloride compounds are much closer to the cubane than to the stella quadrangula structure. The iodide compounds, with the exception of $[\text{AgIPh}_3\text{P}]_4$, are on the other hand very close to the stella quadrangula-structure. The decrease in the halide tetrahedron in the series $\text{I} > \text{Br} > \text{Cl}$ follows approximately the decrease in the ionic radius for the halides.¹⁹ This is not accompanied by a decrease of the Ag-tetrahedron. With the exception of $[\text{AgIPh}_3\text{P}]_4$, the Ag⋯Ag distances are in fact much smaller in the iodide compounds than in the other ones. This may be a consequence of a larger ionicity in the bromide and chloride compounds resulting in larger repulsive forces between the Ag-ions.

The average Ag⋯Ag distances in the iodide compounds also exhibit a certain trend (Table 4). The Ag⋯Ag distances decrease in the following sequence for the terminal donor atom $\text{P} > \text{S} > \text{N}$, *i.e.* the order of softness for these atoms.^{20,21} However, there is no trend in

Table 4. The coordination geometry in some Ag-compounds containing tetrameric units, $[\text{AgXYR}]_4$, X=Cl, Br, I; Y=S, N, P.

	Compound							
	$[\text{AgI}-(\text{C}_4\text{H}_8\text{S})]_4$	$[\text{AgI}-(\text{C}_5\text{H}_{11}\text{N})]_4$	$[\text{AgI}-(\text{C}_6\text{H}_5)_3\text{P}]_4$	$[\text{AgI}-(\text{C}_2\text{H}_5)_3\text{P}]_4$	$[\text{AgBr}-(\text{C}_6\text{H}_5)_3\text{P}]_4$	$[\text{AgBr}-(\text{C}_2\text{H}_5)_3\text{P}]_4$	$[\text{AgCl}-(\text{C}_6\text{H}_5)_3\text{P}]_4$	$[\text{AgCl}-(\text{C}_2\text{H}_5)_3\text{P}]_4$
X-Ag-X ^a								
Min	105.0	110.0	95.2	108.0	89.2	93.0	87.2	88.2
Max	116.5	112.9	115.4	109.5	100.3	104.0	101.2	99.6
Average	109.9	111.3	104.1	109.0	93.7	100.3	92.7	95.8
Y-Ag-X								
Min	96.7	99.3	104.1	109.1	106.9	110.3	109.9	112.2
Max	124.4	122.6	123.5	110.3	142.4	129.2	138.9	134.9
Average	108.9	107.1	114.1	109.9	122.3	116.6	122.4	119.7
Ag-X ^b								
Min	2.829	2.853	2.836	2.918	2.677	2.422	2.532	2.300
Max	2.959	2.942	3.037	2.919	2.962	2.897	2.760	2.821
Average	2.90	2.91	2.91	2.92	2.80	2.74	2.65	2.65
Ag...Ag								
Min	2.961	2.986	3.115	3.198	3.719	^d	3.408	^d
Max	3.189	3.095	3.768	3.229	3.930	-	3.797	-
Average	3.11	3.03	3.48	3.21	3.83	3.48	3.63	3.54
Calc. for SQ ^c	3.16	3.21	3.05	3.17	2.72	2.80	2.56	2.62
X...X								
Min	4.604	4.775	4.399	4.723	3.964	4.201	3.649	3.926
Max	4.902	4.831	4.801	4.768	4.200	4.201	4.031	3.926
Average	4.74	4.81	4.58	4.75	4.08	4.201	3.84	3.926
Ag...Ag/X...X	0.66	0.63	0.76	0.68	0.94	0.83	0.95	0.90
Ref.	This study	3	4	5	6	7	8	7

^a All angles in degrees. ^b All distances in Å. ^c SQ=Stella-quadrangula structure, see text. ^d The Ag-atoms are disordered.

Table 5. Metal-sulfur and metal-iodide distances (Å) in tetrahydrothiophene solvates of copper(I), silver(I) and gold(I) iodides.

	Compound		
	[CuI(THT) ₂] ₂	[AgI(THT)] ₄	[AuI(THT)] _∞
M-S	2.331(3) 2.318(2)	2.55 ^a	2.306(7) 2.335(6)
M-I	2.637(1) 2.639(1)	2.90 ^a	2.565(2) 2.565(2)
Ref.	22	This study	15

^a Average values, see Table 3 for individual distances.

the average Ag-I distances. It is thus the bond angles around Ag, which are affected by the thermal donor atom.

It is of interest to compare the structure of [AgI(THT)]₄ with the corresponding copper²², [CuI(THT)₂]₂, and gold¹⁵ solvates, [AuI(THT)]_∞. The copper solvate is a dimer with the two copper atoms joined by a double iodo bridge. The coordination around the copper atoms is not far from tetrahedral. The gold compound has the same stoichiometry as the silver compound but the structure is radically different. There are two differently coordinating metal atoms. One coordinates two iodides and the other two sulfur atoms. The gold atoms alternate along an infinite zig-zag chain. The Au-Au distances within the chain are quite short, 2.967 and 2.980 Å, *i.e.* only slightly longer than in metallic gold, 2.877 Å.¹⁴ This indicates fairly strong metal-metal bonds in as much as there are no ligand bridges to force the Au-atoms together. Since the metallic radii of Ag and Au are about the same, it is interesting to note that the shortest Ag-Ag distance in the Ag-compound (2.961 Å) is about the same as the shortest Au-Au distance in the gold compound. This may be a further indication of metal-metal interactions in the silver compound.

The M-I and M-S distances show a most remarkable trend in the series Cu, Ag, Au (Table 5). The increase from Cu to Ag is expected from the increase in ionic radius of the metals.¹⁹ The decrease in going from Ag to Au, 0.22 and 0.33 Å for the S respective I bond length, is so large that the Au-I and Au-S bond lengths are even shorter than the Cu-I and Cu-S bond lengths. As has been discussed elsewhere,¹⁵ several factors presumably attribute to this bond shortening. The classical interpretation is based on the lanthanoid contraction. However, the metallic radii of silver and gold are about the same¹⁴ and for the ions Ag(I) and Au(I) the gold ion is considered to be the largest.¹⁹ Therefore other factors must be taken into account such as the large increase of the relativistic contraction of the *s*- and *p*-shells, which takes place between the 5th and 6th period.²³

Acknowledgement. The idea of describing tetrameric complexes as stella quadrangula structures originates from a discussion with Dr. Staffan Hansen.

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Received May 21, 1985.