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Crystal Structure and Topological Interpretation of the β-Modification of Silver(I) Diethyldithiocarbamate

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The crystal structure of the β-modification of silver(I) diethyldithiocarbamate, $AgS_2CN(C_2H_5)_2$, has been determined by X-ray diffraction. The crystals are monoclinic. Space group $C2/c$; $a=18.406(3)$, $b=9.726(2)$, $c=14.305(4)$ Å, $\beta=104.79(3)°$, $Z=12$. The silver atoms and the ligands are linked in chains running along c. The silver atoms are four coordinated with Ag–S in the range 2.5 to 3.0 Å. The structure is compared to the less regular α-modification using a new type of topological maps which reveal similarities also with a large number of other sterically restricted coordination compounds.

The dialkyldithiocarbamates of the univalent coinage metals, copper, silver and gold, belong to a class of compounds where sterical restrictions strongly influence the crystal structures. Successful metal coordination requires that the ligands are arranged closely around the metal atom but the large and bulky ligands are not easily arranged in such a manner. This conflict may be solved by the formation of low polymers or chain or layer polymers. These complex structures, of which a large number have been determined at this Institute, may be difficult to grasp and to analyze. We present here, along with the crystal structure of the β-modification of silver(I) diethyldithiocarbamate, some topological tools for the interpretation and understanding of these structures.

The crystal structure of silver(I) diethyldithiocarbamate, was studied by Hesse.[1] By X-ray powder diffraction he discovered that the compound crystallized in two forms when prepared according to Åkerström.[2] However, crystals suitable for structure determination were found for only one of the forms. The structure determined by Hesse for this α-modification has been verified by Yamaguchi *et al.*[3] and again by us. There are no significant differences between the three determinations. Suitable crystals of the other form, the β-modification have now been prepared.

EXPERIMENTAL

Silver(I) diethyldithiocarbamate was obtained on mixing aqueous solutions of silver nitrate and sodium diethyldithiocarbamate trihydrate. A saturated solution of the dry precipitate in carbon disulfide was cooled to −40 °C. Crystals were obtained after three h. The pale yellow crystals appeared as six-sided plates. The cell parameters were based on 34 lines from a Guinier-Hägg powder photograph with $CrK\alpha_1$ radiation ($\lambda=2.28962$ Å) and silicon ($a=5.43054$ Å) as an internal standard. The density was measured by flotation using an aqueous solution of K_2HgI_4.

Crystal data. $AgS_2CN(C_2H_5)_2$. Monoclinic. Unit cell parameters: $a=18.406(3)$, $b=9.726(2)$, $c=14.305(4)$ Å, $\beta=104.79(3)°$. $Z=12$, $D_m=2.051$ g cm^{-3}, $D_x=2.062$ g cm^{-3}, $\mu(MoK\alpha)=27.8$ cm^{-1}. Systematic absences: hkl for $h+k=2n+1$ and $h0l$ for $l=2n+1$. Possible space groups: Cc (No. 9) and $C2/c$ (No. 15).

The intensity data were collected on a Stoe-Philips four-circle PDP 8/I computer-controlled diffractometer with graphite monochromatized $MoK\alpha$ radiation ($\lambda=0.71069$ Å) and with the $\omega-2\theta$ scan technique. The faces of the crystal were (001), (100), (110) and ($\bar{1}10$), and the corresponding approximate inter-planar distances were 0.03, 0.20, 0.15 and 0.17 mm. The count rate was reduced by a factor 2^n, $0\leq n\leq 3$ (scan time and one filter). The longest scan time was 141 s, and the background intensities were measured for 20 s on each side of a peak. Reflexions within $-21\leq h\leq 21$,

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$-11 \leq k \leq 0$, $0 \leq l \leq 17$ and $\sin\theta/\lambda \leq 0.60$ Å^{-1} were recorded. The intensities of three standard reflexions, 040, $\bar{1}0$ 0 4 and 006, measured at intervals of 40 reflexions, varied by less than ± 3.4 %. Correction were made for background, Lorentz, polarization and absorption effects. A total of 2501 intensities were reduced to 2181 independent structure amplitudes. Of these 1737 having $|F_m| \geq 2\sigma(F)$, $|F_m|/|F_c| \leq 2.0$ and $\sin\theta/\lambda > 0.060$ Å^{-1} were used in the final refinement.

The approximate positions of the silver atoms were obtained from the three-dimensional Patterson function. The sulfur, nitrogen and carbon atoms were located in difference syntheses assuming the non-centrosymmetric space group *Cc*. No attempt was made to locate the hydrogen atoms. The scale factor, the coordinates and the thermal parameters (anisotropic for silver and sulfur, isotropic for nitrogen and carbon) were refined by full-matrix least squares. The expression minimized was $\Sigma w(|F_m| - |F_c|)^2$, where $w^{-1} = \sigma^2(F) = \sigma^2_{count}(F) + (0.05|F_m|)^2$. As the structure turned out to possess center of symmetry the refinement was terminated using the space group *C*2/*c* with one of the silver, carbon and nitrogen atoms placed in the special position (e): 0 *y* 1/4, 0 $\bar{y}$ 3/4. The refinement converged at $R(F) = 0.065$ and $R_w(F) = 0.103$. The final shifts were all smaller than 0.10 of the estimated standard deviations. The fractional coordinates and the thermal parameters are presented in Tables 1 and 2. Lists of structure factors may be obtained on request from the authors. For Ag, S, N and C the scattering factors and the f' and f'' anomalous dispersion corrections were taken from *International Tables for X-Ray Crystallography*.[4] Lundgren[5] has described the computer programs. The calculations were performed on the IBM 370/155–158 and IBM 1800 computers in Uppsala.

DESCRIPTION AND DISCUSSION

General Features. The silver atoms and the diethyldithiocarbamate ligands are linked together by Ag–S bonds to form unlimited chains parallel to *c*. The only contacts between different chains are of the van der Waals type. Fig. 1 shows a part of a

Table 1. Fractional coordinates and isotropic thermal parameters B (Å^2) defined from $\exp\{-B[(\sin\theta)/\lambda]^2\}$.

	x	*y*	*z*	*B*
Ag1	0.0	−0.0552(1)	0.2500	
Ag2	−0.01838(5)	0.1253(1)	0.44914(7)	
S1	0.0749(1)	0.1802(3)	0.2259(2)	
S2	−0.1220(1)	−0.0852(3)	0.4888(2)	
S3	−0.0760(1)	0.1773(2)	0.5980(2)	
N1	0.0	0.4029(11)	0.2500	2.9(2)
N2	−0.2066(4)	0.0472(8)	0.5833(5)	2.9(1)
C1	0.0	0.2714(14)	0.2500	2.9(2)
C2	0.0631(6)	0.4882(11)	0.2319(7)	3.7(2)
C3	0.0450(6)	0.5174(12)	0.1209(8)	4.2(2)
C4	−0.1402(5)	0.0439(9)	0.5606(6)	2.8(2)
C5	−0.2603(6)	−0.0677(11)	0.5594(7)	3.6(2)
C6	−0.3159(6)	−0.0472(12)	0.4624(8)	4.0(2)
C7	−0.2343(7)	0.1704(13)	0.6276(9)	4.8(2)
C8	−0.2116(9)	0.1495(19)	0.7392(12)	7.0(4)

Table 2. Anisotropic thermal parameters b_{ij} defined from $\exp(-b_{11}h^2 - \cdots - 2b_{23}kl)$.

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Ag1	0.00339(5)	0.0091(1)	0.00643(9)	0.0	−0.00007(5)	0.0
Ag2	0.00406(4)	0.0133(1)	0.00744(7)	0.00169(5)	0.00315(4)	0.00388(7)
S1	0.00293(9)	0.0088(3)	0.0059(2)	0.0005(1)	0.00164(9)	−0.0003(2)
S2	0.00294(9)	0.0101(3)	0.0051(2)	−0.0006(1)	0.00163(9)	−0.0027(2)
S3	0.00260(8)	0.0067(3)	0.0051(1)	−0.0004(1)	0.00121(9)	−0.0004(2)

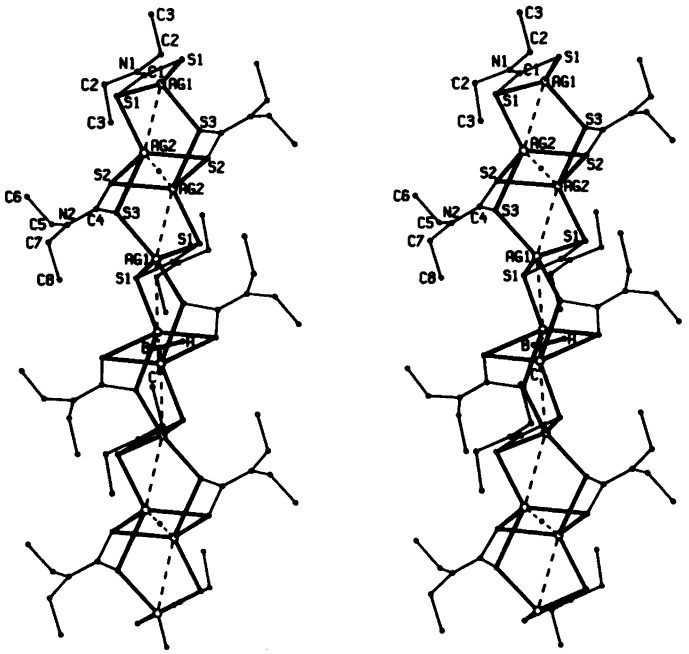

Fig. 1. The chain structure in the β-modification of silver(I) diethyldithiocarbamate.

chain which can be described as built of *monomers* of the metal-ligand unit with twofold rotational symmetry alternating with centrosymmetric *dimers* with their centres at 0,0,0 and 0,0,1/2. The silver atoms Ag1 belong to the monomers and Ag2 to the dimers. In the monomers the silver, central carbon and nitrogen atoms are situated on the twofold axes, $0,y,1/4$ and $0,\bar{y},3/4$, the carbon–nitrogen vectors pointing alternately in the $+b$ and $-b$ directions. The silver atoms form a zig-zag chain close to the plane $x=0$. One short and two long Ag···Ag distances are repeated periodically along the chain. The short distances, 2.83 Å, occur within the dimers and the long distances, 3.44 Å, between the dimers. The angles in the silver chain are 118.5° at Ag1 and 85.7° at Ag2.

The bond distances and angles of the ligands (Table 3) have usual values, and the C_2NCS_2

Table 3. Distances (Å) and angles (°) in the diethyldithiocarbamate ligands.

S1–C1	1.746(7)	S1–C1–S1	118.9(8)
C1–N1	1.278(18)	S1–C1–N1	120.6(4)
N1–C2	1.503(12)	C1–N1–C2	123.5(5)
C2–C3	1.564(15)	C2–N1–C2	113.0(11)
S2–C4	1.709(9)	N1–C2–C3	107.5(7)
S3–C4	1.744(9)	S2–C4–S3	120.6(5)
C4–N2	1.342(11)	S2–C4–N2	119.5(7)
N2–C5	1.474(13)	S3–C4–N2	119.7(7)
N2–C7	1.504(15)	C4–N2–C5	121.7(8)
C5–C6	1.512(15)	C4–N2–C7	123.3(8)
C7–C8	1.557(21)	C5–N2–C7	114.9(8)
		N2–C5–C6	111.7(9)
		N2–C7–C8	107.3(11)

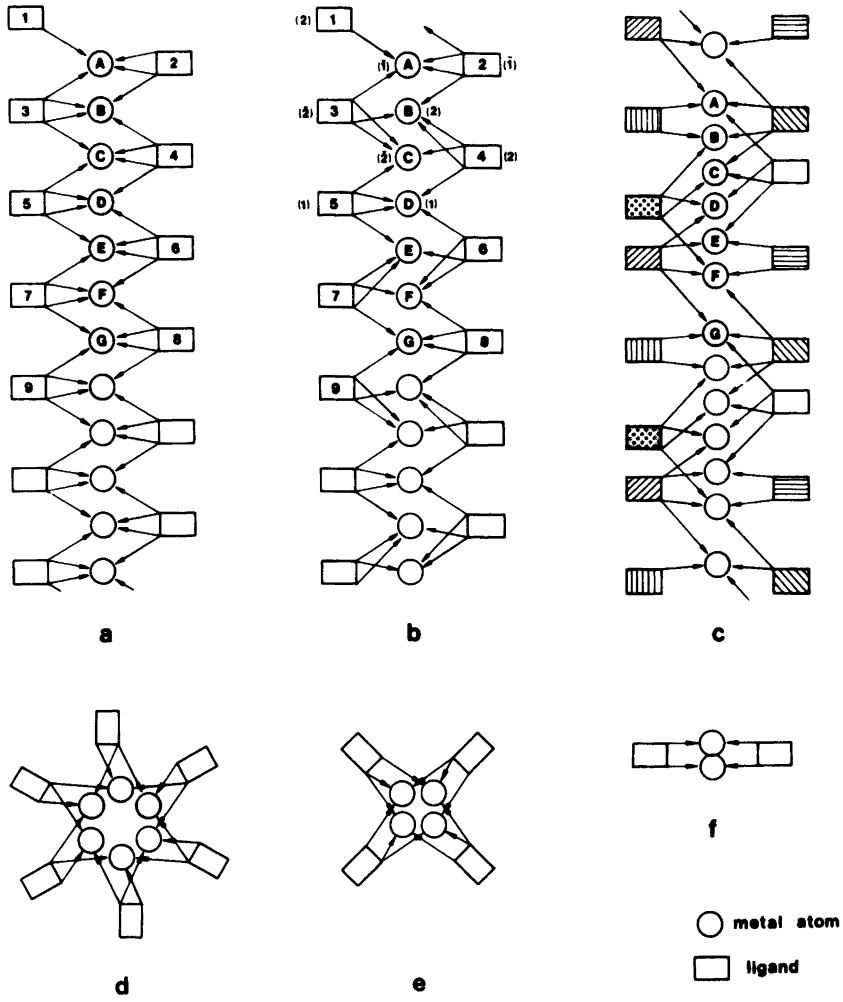

Fig. 2. Schematic representations of AX structures. The ligands are labelled 1, 2, 3, ... and the metal atoms are given alphabetical notations. In Fig. 2b the extra notations (1), ($\bar{1}$) correspond to the monomers and (2), ($\bar{2}$) to the dimers discussed in the text.

fractions of the ligand molecules are almost planar. The outer carbon atoms are in *trans*-position which is the usual case. As a contrast, two of the ligands of a hexamer in the α-modification[3] occur in *cis*-position which may be a consequence of the complicated linkage in that structure.

Coordination and geometrical dependencies. The coordination net formed by the linkage between the metal atoms and the ligands is schematically illustrated in Fig. 2b. The silver atoms are four-coordinated and the sulfur atoms two-coordinated. Each silver atom is coordinated by three ligands, one of which by chelate formation.

The silver coordination polyhedra are distorted tetrahedra. The distances of coordination vary from 2.51 to 2.95 Å (Table 4). The smallest S–Ag–S angles, 64.4 and 66.6°, are found in the chelate rings. The remaining larger angles, 102.5 to 127.6°, illustrate a tendency of the silver atoms to maintain a regular coordination environment despite the restrictions imposed by the chelate rings. The coordination angles are, however, also influenced

Table 4. Some distances (Å) and angles (°) in the chain structure.

Ag2···Ag2^i	2.831(2)	Ag1···Ag2···Ag2^i	85.7(1)
Ag1···Ag2	3.436(1)	Ag2···Ag1···Ag2^i	118.5(1)
Ag1 – S1	2.739(3)	Ag1 – S1 – Ag2	81.6(1)
Ag1 – S3	2.558(3)	Ag2 – S2 – Ag2^i	61.5(1)
Ag2 – S1	2.511(3)	Ag1 – S3 – Ag2	110.4(1)
Ag2 – S2	2.540(3)		
Ag2 – S3	2.658(3)	S1 – Ag1 – S1^i	66.6(1)
Ag2 – S2^i	2.951(3)	S1 – Ag1 – S3	117.7(1)
		S1 – Ag1 – S3^i	108.1(1)
Ag1 – S1 – C1	87.3(4)	S3 – Ag1 – S3^i	124.7(1)
Ag1 – S3 – C4	95.5(3)	S1 – Ag2 – S2	120.4(1)
Ag2 – S1 – C1	99.1(1)	S1 – Ag2 – S3	127.6(1)
Ag2 – S2 – C4	103.9(3)	S1 – Ag2 – S2^i	102.5(1)
Ag2 – S3 – C4	89.5(3)	S2 – Ag2 – S3	109.1(1)
Ag2 – S2^i – C4	80.9(3)	S2 – Ag2 – S2^i	118.5(1)
		S3 – Ag2 – S2^i	64.4(1)

by other geometrical details of the structure, *e.g.* the Ag – S – Ag and the Ag – S – C angles.

The Ag – S – Ag angles are between 61.5 and 110.4° (Table 4). The lowest angle is found in the S2 – Ag2 – $\overline{S2}$ – $\overline{Ag2}$ parallelogram of the dimer. Since the metal – sulfur – metal angles show similar strong variations in a number of other dithiocarbamates there seems to be no pronounced restrictions in these angles.

The metal – sulfur – carbon angles have a remarkable stability in a number of sulfur complexes of copper, silver and gold. In unchelated compounds their values are usually in the range 100 – 110° as pointed out by Jennische.[6] In the present compound the Ag – S – C angles have low values in the chelate rings, 80.9 – 89.5° as can be expected, whereas the unchelated angles are in the range of 95.5 – 103.9°. These angles are of considerable importance as they may influence the geometry of the coordination net by imposing sterical restrictions. They are also related to the orientations of the ligands and accordingly to the packing possibilities.

Interdepencies of the angles and distances in molecular and crystal structures have been given explicit attention by Hesse in a degree of freedom rule.[7] Consider the parallelogram S2 – Ag2 – $\overline{S2}$ – $\overline{Ag2}$ as an example. The small Ag2 – S2 – $\overline{Ag2}$ angles, 61.5°, can be considered as the geometrical consequence of the angles S2 – Ag2 – $\overline{S2}$ which have a value, 118.5°, close to the average of 116°, for the non-chelated S – Ag – S angles. The short Ag2 – $\overline{Ag2}$ distance, 2.83 Å, and the long Ag2 – S2 distance, 2.95 Å, which occur in the same parallelogram may also be interpreted by the interdependencies. A long silver – sulfur distance (Ag2 – S3, 3.57 Å), which is considered to be outside the range of coordination, may be interpreted analogously.

A more complicated coordination net is found in the α-modification of silver(I) diethyldithiocarbamate[3] (Fig. 2c) which represents a border case between high and low polymers. Irregular hexamers may be discerned and they are linked in chains by weak bonds only, Ag – S = 2.99 Å. Two of the six silver atoms have fourfold coordination, the others threefold. Two ligands coordinate to only two metal atoms, the others to four. Five short metal – metal distances occur in each of the hexamers. In the present structure, the β-modification, which is a true high polymer, all the silver atoms have fourfold coordination and all the ligands are linked to three metal atoms. There are only two short metal – metal distances per six silver atoms.

The hypothetical structure of Fig. 2a represents the most regular coordination net obtainable in a chain with chelated four-coordinated metal atoms. All the metal atoms are engaged in two double bridges and each double bridge tetragon has two edges in common with adjacent chelate rings. As in the β-modification one would then expect high silver coordination angles in the bridging tetragons and thus short metal – metal distances. In this case, however, all the metal – metal distances would be short.

Regular networks are also found in the low polymers of some coinage metal dithiocarbamates

and thiocarbamates. Hexamers (Fig. 2d) are found in copper(I) dipropylthiocarbamate,[8] silver(I) dipropylthiocarbamate[9] and silver(I) dipropyldithiocarbamate,[10] and tetramers (Fig. 2e) in copper(I) diethyldithiocarbamate.[7] Dimers (Fig. 2f) are formed in the dipropyldithiocarbamate[11] and the dibutyldithiocarbamate[12] of gold(I).

The formation of polymers in these compounds is the obvious result of the tendency of the metal atoms to obtain as high coordination numbers as possible. It is worth noticing that dimers are found only for the compounds of gold(I) which has a marked tendency for twofold linear coordination. For the copper and silver compounds the coordination number increases as the bulk of the ligand decreases. Threefold coordination is found in the tetramers and hexamers. The formation of chains or layers to attain higher coordination is probably prevented by the large size of the ligands as compared to the distances of coordination.

Fourfold coordination is found in the two modifications of the present compound and is likely to be found in the lowest homologues of the series, the dimethyldithiocarbamates of silver and copper. The structures of these compounds have not yet been determined but their high melting points and low solubilities indicate the presence of high polymers. A similar pronounced trend for the metal atom to obtain a higher coordination number in lower dialkyldithiocarbamate homologues has been found in a systematic study of the dithiocarbamates of thallium(I).[6]

Topological maps. The topological map representations given in Fig. 3 have been found to be powerful tools for recognition of linkage patterns, deriving alternatives, comparing common features and excluding sterically unreasonable networks. They have been in use for many years and were briefly mentioned by Hesse at the Sixth International Conference of Coordination Chemistry in 1961 but are published here for the first time.

Let the bonding interaction between a metal atom and a ligand be represented by a *link*. The link then corresponds to a line in an adjacency graph. There is just *one* link between the metal atom and the ligand even if there may be two or several bonds between them. In the topological map a particular link is represented by a square element of a mosaic pattern.

The networks depicted in Fig. 2 are represented by the maps of Figs. 3a–e. Since *links* are considered, the nets 2a and 2b have the same map

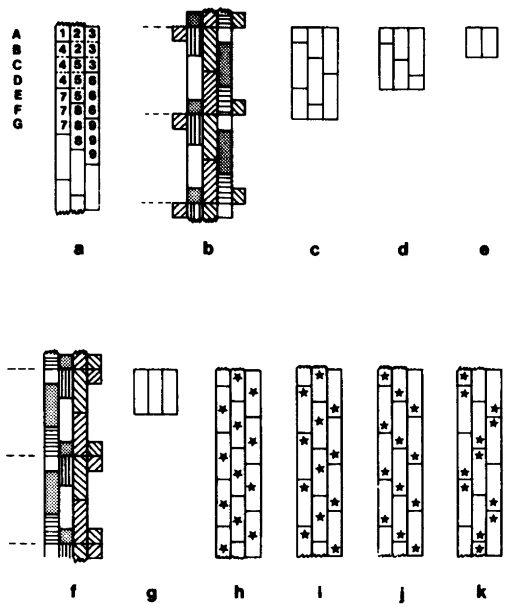

Fig. 3. Topological maps.

representation 3a. The pattern transformations from Fig. 2 to Fig. 3 can easily be followed by the letters, numbers and "colours" given in 2abc and 3 ab. Each horizontal row in Fig. 3 corresponds to a metal atom (A,B,C,,,). Each square element in a row corresponds to a link between the metal atom and a particular ligand. The ligands are labelled 1,2,3... in 3a and "coloured" in 3b as in 2abc.

Since the ligand notations are irrelevant they may be permuted arbitrarily. For the same reason the rows representing the metal atoms can be permuted at will. The order of the elements within a row can also be changed at will. Since the elements represent *links* an element with a given "colour" can only occur once in a row.

Using these permutation rules the elements can be rearranged to form easily recognizable patterns. The links belonging to the same ligand are, for instance, usually ordered to a vertical field as seen in Fig. 3a. In this way geometrically meaningful graphs can be found from the maps.

Relationships between different structures can be seen by comparing motifs in the corresponding topological maps. The structures $(LiCH_3)_4$[13,14] and $[CuS_2CN(C_2H_5)_2]_4$[7] are both represented by the map 3d which is the only way of obtaining threefold linkage of the metal atoms and ligands in a tetramer. As a starting point for bond dis-

cussions one must then ask the question: In which *other* way can the metal atoms and ligands be combined?

The hexamer, 3c, the tetramer, 3d, and the dimer, 3e, represent three possibilities of regular linkage among the low polymers. Fig. 3 shows that they may be cut from the unlimited topological chain, 3a, which is the most regular type of chain with threefold linkage in a compound with the composition AX.

By permutation of columns in 3b (the α-modification) the map 3f is obtained, still representing the same linkage pattern. On comparison of the repeat motif in 3f and 3a (the β-modification) striking similarities are seen which have been used for the construction of the graphs to be discussed in the next section.

The maps may also facilitate the exclusion of topologically possible combinations on sterical grounds. An example of a linkage scheme which is extremely unlikely to be realized in a chemical system is 3g. Three metal atoms would have to coordinate the same three ligands. A map which can be made to contain a section such as that of 3g by a series of allowed permutations must thus be discarded.

The coordination nets of the hypothetical chain, 2a, and the β-modification, 2b, are distinguished by the pattern of the chelate rings. The two structures are thus *chelate isomers*. These chelate isomers are represented by the maps 3h and 3i, where the chelate links are marked by asterisks. Two other isomers which will be discussed are given in the map 3j and 3k.

Topological maps of the type presented here define the combinatorial possibilities and restrictions of the metal–ligand linkage. The maps have been used for the derivation of a large number of theoretically possible networks for low polymers and chains in AX compounds. They can also be used for studies of AX nets in compounds with the composition A_pX_qR. The maps contain only essential information and obviate the large number of zeroes occurring in the combinatorial adjacency or bond order matrices commonly used in graph theory.[15]

The realization in space of topological schemes. Graphs corresponding to the topological maps are readily drawn. Fig. 4a shows the simple graph corresponding to the topological map of the unlimited chain, 3a. Again the notation is that of Fig. 2. The broken line illustrates the metal–metal chain.

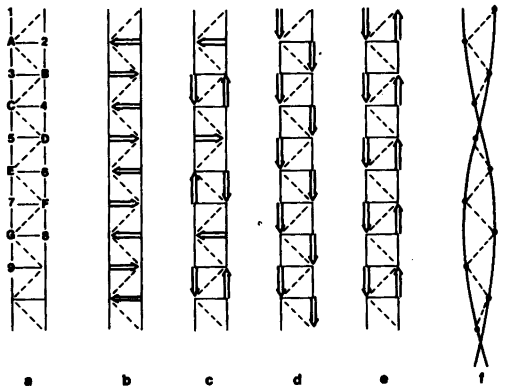

Fig. 4. Alternative chain structures.

Figs. 4b–e show the graphs of the set of chelate isomers 3h–k. The chelate links are denoted by arrows.

The distribution of arrows in Fig. 4 follows certain rules which are evident from the figure. A horizontal arrow as in 4b must be followed either by a horizontal arrow in the opposite direction, 4b, or by a pair of antiparallel vertical arrows, 4c. A vertical arrow, must be followed by a parallel arrow, 4d, or be accompanied by an antiparallel arrow, 4e. It is interesting to note that the pattern of 4d cannot be broken once it has been started. The monomers of 4b and the dimers of 4e may, however, be combined in an infinite number of sequential patterns, the pattern with the shortest repeat distance being that of 4c.

The graph 4c shows that the alternating directions of the monomers in the structure of the β-modification (Fig. 1) is a consequence of the linkage.

Despite its attractive simplicity the linkage scheme of 4b must be discarded for silver(I) diethyldithiocarbamate. Model studies indicate clearly that it cannot be realized in a planar fashion because of collisions between the ligands. If the linkage scheme is to be maintained, a twist must then be introduced so that a helix is formed, 4f. This helix is very likely to be an *irrational helix*, where the rotational repeat angle $h \times 360°$ has a value of h equal to an irrational number. Such irrational helices are not crystallizable. Theoretical aspects of rational and irrational helices and packing will be given in a separate paper.

Planar realizations are unlikely also for the linkage schemes 4d and e. The short repeat distance

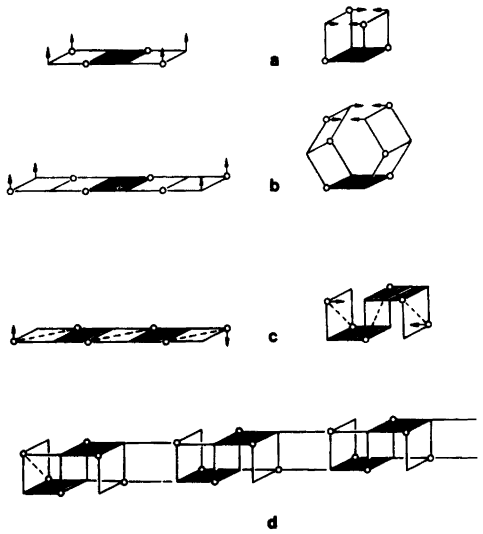

Fig. 5. Folding procedures of chain fragments (Fig. 4a) giving low polymers and the structure of the α-modification of silver(I) diethyldithiocarbamate.

make ligand–ligand collisions unavoidable. The β-modification is represented by 4c. In this scheme the repeat distance is three times as long as in the other three (4b,d,e) making it possible to form the almost planar metal–metal chain.

Instead of twisting the entire chain, which likely would produce an irrational helix, the ligand collisions may be avoided by folding sections of the chain. This is done in the low polymers and in the α-modification.

The simple foldings required to produce the tetramer and hexamer are illustrated in Figs. 5a,b. In this way it is also possible to produce even number polymers of higher polymerity. The metal atoms will always be arranged as antiprisms: linear, trigonal, tetragonal *etc.* Odd number polymers are topologically possible but very unlikely sterically as they will be realized as Möbius strips.

The irregular hexameric units of the α-modification are produced by the more complicated folding shown in Fig. 5c. The linking of the units to produce chains is shown in 5d.

This analysis makes it clear that the two apparently very different modifications of silver(I) diethyldithiocarbamate, as well as the low polymers of other coinage metals dithiocarbamates and thiocarbamates, are sterical realizations of very similar topological patterns.

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