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# Toward absolute asymmetric synthesis of coordination polymers with bidentate sulfide ligands

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ABSTRACT

In search for sulfide-containing coordination polymers that crystallize as conglomerates, five new copper(I) complexes with prochiral sulfide ligands have been prepared and characterized by single crystal X-ray structure determination. Three unsymmetrical sulfides have been used: phenyl propargyl sulfide (Sprop), allyl methyl sulfide (Sally), and 2,5-dithiahexane (SS). In $[CuCl(Sprop)]_n$ (**1**), layers are formed via π-coordination of propargyl groups to copper(I). In $[Cu_2Br_2(Sprop)_4]$ (**2**), discrete dimers form with non-coordinating propargyl groups. In $[CuCl(Sally)]_n$ (**3**), layers are formed via π-coordination of allyl groups to copper(I), but disordered Sally ligands are also found. The mesitylcopper complex $[Cu_4(Mes)_4(Sally)_2]$ (**4**) is chiral but discrete. In $[Cu_4(Mes)_4(SS)]_n$ (**5**), racemic chains are formed by the SS ligand. Three out of five complexes prepared thus form coordination polymers, and all of the five complexes (**1**–**5**) exhibit terminal sulfide ligands that could be oxidized selectively when incorporated in an enantiopure polymer. Unfortunately none of **1**–**5** crystallized as a conglomerate, but whether this reflects an inherent tendency in this system is too early to say.



## 1. Introduction

Homochirality of essential molecules, such as sugars and amino acids, is one of the fundamental aspects of Life. The origin of such biomolecular homochirality, and at what stage during evolution homochirality became dominant, are matters that are still open for speculation [1]. Although many stereoselective reactions, under the name of asymmetric synthesis, have become powerful tools in the quest for enantiopure molecules and materials, they usually depend on pre-formed optical activity in some form (*e.g.* in the substrate, reagent or catalyst) and are thus not very informative concerning the genesis of molecular optical activity. On the other hand, the discovery of new spontaneous symmetry-breaking reactions may provide insight into the origin of biomolecular homochirality. Reactions that can form enantiomerically enriched products from achiral precursors, without the intervention of pre-formed optical activity, constitute examples of absolute asymmetric synthesis (AAS) [1c,2]. There are only a few genuine examples of absolute asymmetric syntheses known today, and they often depend on the transformation (usually photo-chemically) of an achiral substrate in a chiral crystal [3]. Although AAS using achiral substrates in chiral crystals can give high enantiomeric excesses, they are limited to special cases. However, if a general reagent could be obtained as a homochiral batch of enantiopure crystals by total spontaneous resolution (TSR) [4] a wide range of reactions giving products from AAS would be feasible [5]. Or, alternatively, if a general prochiral substrate would form a stereochemically labile chiral metal complex, then single-colony crystallization could lead to a homochiral batch of enantiopure crystals. The substrate in such a homochiral crystal batch could then be transformed to several different enantiopure products by solid-state reactions with achiral reagents [6].

We are especially interested in crystallizing chiral polymers and networks containing prochiral substrates [7]. If such crystals could be induced to crystallize as a conglomerate, it would be possible to obtain homochiral crystal batches on a large scale by single-colony crystallization. In addition to the relevance in connection to the origin of biomolecular homochirality, there would be several potential applications for such enantiopure materials, for example as heterogenous catalysts, as chiral selector phases in separation and chromatography, or as bio-materials or -sensors. We would like to induce chiral polymer formation by using unsymmetrical sulfide ligands; such ligands could be oxidized enantioselectively in a chiral crystal (Scheme 1) [8]. Such chiral coordination polymers should be unsoluble in non-coordinating solvents and could thus be subjected to heterogenous oxidation at elevated temperatures, if

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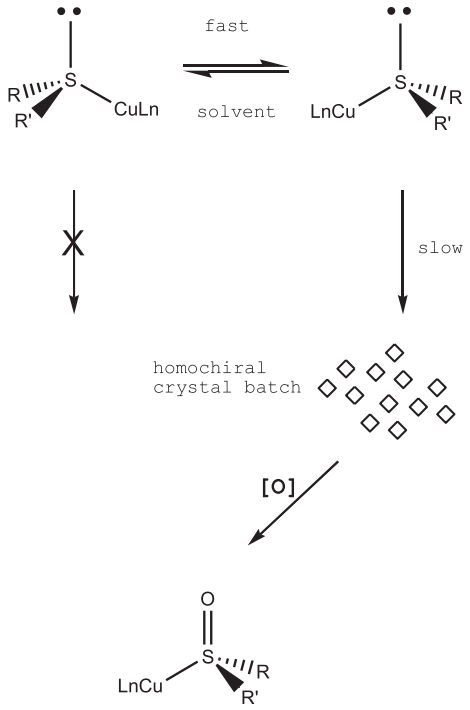



**Scheme 1.** Enantioselective sulfide oxidation by AAS.

necessary. In this work we set out to investigate if using bidentate sulfides could aid in polymer formation by bridging.

## 2. Experimental

### 2.1. General

All operations were carried out under nitrogen using Schlenk or low temperature [9] techniques. Solvents were distilled from sodium/benzophenone shortly prior to use. Copper(I)chloride and copper(I)bromide were purified according to literature procedures [10]. Phenyl propargyl sulfide (Sprop) and allyl methyl sulfide (Sally) were dried with molecular sieves and deoxygenated. Mesitylcopper [11] and 2,5-dithiahexane [12] (SS) were prepared as previously described.

### 2.2. Preparation of $[CuCl(Sprop)]_n$ (**1**)

Copper(I) chloride (0.14 g, 1.4 mmol) was dissolved in 1.0 mL phenyl propargyl sulfide at ambient temperature. Colorless needles were obtained after a week at −15 °C in 60% yield.

### 2.3. Preparation of $[Cu_2Br_2(Sprop)_4]$ (**2**)

Copper(I) bromide (0.30 g, 2.1 mmol) was dissolved in 1.0 mL phenyl propargyl sulfide at ambient temperature. Colorless crystals were obtained after a few days at −25 °C in 65% yield.

### 2.4. Preparation of $[CuCl(Sally)]_n$ (**3**)

Copper(I) chloride (0.14 g, 1.4 mmol) was dissolved in 1.0 mL allyl methyl sulfide at ambient temperature. Colorless crystals were obtained after a week at −80 °C in 75% yield.

### 2.5. Preparation of $[Cu_4(Mes)_4(Sally)_2]$ (**4**)

A solution of mesitylcopper in THF [11] (0.8 mmol, 0.4 M, 2.0 mL) was stripped of solvent *in vacuo* and dissolved in 0.10 mL allyl methyl sulfide with gentle heating. Needle-shaped yellow crystals were deposited after a few hours at ambient temperature in 31% yield.

### 2.6. Preparation of $[Cu_4(Mes)_4(SS)]_n$ (**5**)

A solution of mesitylcopper in THF [11] (0.8 mmol, 0.4 M, 2.0 mL) was stripped of solvent *in vacuo* and dissolved in 1.0 mL toluene wherafter 0.10 mL 2,5-dithiahexane was added. A light yellow precipitate was formed immediately and the mixture was heated to give a clear yellow solution from which yellow crystals were deposited after a few hours in 27% yield.

### 2.7. X-ray crystallography

Crystal and experimental data are summarized in Table 1. All crystals were selected and mounted under nitrogen in a glass capillary at low temperature [9] and transferred in liquid nitrogen to a Rigaku R-AXIS IIc image plate system. Diffracted intensities were measured using graphite-monochromated Mo Kα ($\lambda = 0.71073$ Å) radiation from a RU-H3R rotating anode operated at 50 kV and 90 mA. Using the R-AXIS IIc detector, 90 oscillation photos with a rotation angle of 2° were collected and processed using the CrystalClear software package. An empirical absorption correction was applied using the REQAB program under CrystalClear. Crystal and refinement data for compounds **1**–**5** are summarized in Table 1. All structures were solved by direct methods (SIR 97) [13] and refined using full-matrix least-squares calculations on $F^2$ (SHELXL-97) [14] operating in the WinGX program package [15]. Anisotropic thermal displacement parameters were refined for all the non-hydrogen atoms. Hydrogen atoms were included in calculated positions and refined using a riding model. Figs. 1–5 have been drawn with ORTEP-3 for Windows [15] under WinGX.

## 3. Results and discussion

We reasoned that a soft metal center, such as copper(I), would be suitable to coordinate the soft sulfide ligands. Halide ligands could bridge two, three, or even four metal centers and thus assist in polymer formation [16]. Mesityl ligands can also bridge and have proved useful in organocopper chemistry [11]. In order to obtain enantiopure crystals we need to identify conglomerate [17] phases. However, finding a conglomerate could be a time-consuming enterprise since we essentially are left with trial-and-error strategies. It is estimated that less than 10% of all chiral compounds form conglomerates [18], but it must be kept in mind that the statistics is largely based on organic compounds, and the numbers could be very different for stereochemically labile coordination compounds. In this work we have prepared and structurally characterized five new copper(I) halide and mesityl (Mes) complexes using phenyl propargyl (Sprop), allyl methyl sulfide (Sally), and 2,5-dithiahexane (SS) as ligands (Scheme 2 and Table 2). While many metal complexes having symmetrical $SR_2$ ligands have been previously reported [19], complexes with unsymmetrical (and thus prochiral) RSR' ligands are much rarer. A polymeric copper(I) chloride complex with 2,5-dithiahexane that exhibits a centrosymmetric crystal structure is one example [20]. A search in the Cambridge Structural Database (CSD) reveals only two metal complexes with the Sally ligand: a ruthenium(II) chloride complex [21] with a σ-bonded Sally and a rhodium(I) complex [22] with σ,π-bonded Sally.

**Table 1**
Crystal and refinement data for **1**–**5**.

| Complex | **1** | **2** | **3** | **4** | **5** |
|---|---|---|---|---|---|
| Formula | $C_9H_8CuClS$ | $C_{18}H_{16}CuBrS_2$ | $C_8H_{16}Cu_2Cl_2S_2$ | $C_{44}H_{60}Cu_4S_2$ | $C_{47}H_{62}Cu_4S_2$ |
| Formula weight | 247.20 | 439.88 | 374.31 | 907.20 | 945.25 |
| Crystal system | Orthorhombic | Monoclinic | Monoclinic | Monoclinic | Monoclinic |
| Space group | *Pbca* | $P2_1/n$ | $P2_1/c$ | $C2/c$ | $C2/c$ |
| *a* [Å] | 8.8918(15) | 8.966(2) | 6.3196(9) | 25.662(6) | 45.392(7) |
| *b* [Å] | 10.188(2) | 20.530(4) | 21.046(4) | 8.697(2) | 8.581(1) |
| *c* [Å] | 21.641(4) | 9.825(2) | 10.7575(17) | 23.505(6) | 24.489(4) |
| $\beta$ [°] | 90 | 95.930(9) | 111.43(1) | 125.320(6) | 109.881(5) |
| *V* [Å$^3$] | 1960.5() | 1798.9(6) | 1331.9(4) | 4280(2) | 8970(2) |
| *Z* | 8 | 4 | 4 | 4 | 8 |
| $\delta_{calcd}$ [g cm$^{-3}$] | 1.675 | 1.624 | 1.867 | 1.408 | 1.400 |
| $\mu$ [mm$^{-1}$] | 2.652 | 3.660 | 3.869 | 2.087 | 1.995 |
| $\theta$ range [°] | 3.58–26.00 | 3.03–26.00 | 3.45–26.00 | 3.18–25.50 | 2.52–25.00 |
| Refls collected | 12,602 | 12,081 | 9123 | 13,344 | 27,823 |
| Indep refls | 1867 | 3374 | 2586 | 3830 | 7521 |
| Parameters | 113 | 207 | 124 | 233 | 493 |
| *R*1 [$I > 2\sigma(I)$] | 0.044 | 0.030 | 0.055 | 0.047 | 0.050 |
| *wR*2 [all data] | 0.083 | 0.076 | 0.130 | 0.106 | 0.089 |
| Max peak [e Å$^{-3}$] | 0.34 | 0.89 | 1.03 | 0.78 | 0.44 |
| Max hole [e Å$^{-3}$] | −0.43 | −0.62 | −1.01 | −0.50 | −0.61 |

Interestingly, there are no metal complexes with the Sprop ligand in the CSD.

Crystals of $[CuCl(Sprop)]_n$ (**1**) could be prepared by using neat phenyl propargyl sulfide as solvent; slow cooling proved efficient. A crystal structure determination (Fig. 1) reveals formation of layers, where bridging is afforded both via the anionic and neutral ligands. The Sprop ligand is bridging by side-on coordination of the C8–C9 triple bond to Cu1. This is reflected in the ligand geometry: the

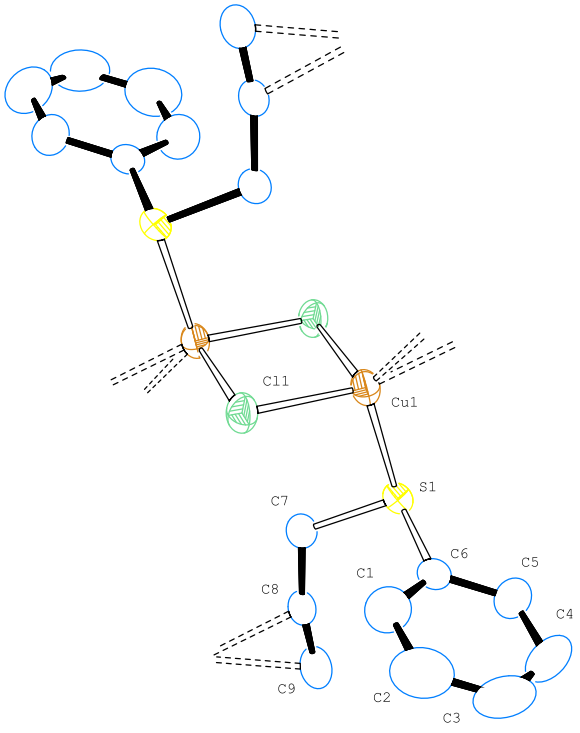



**Fig. 1.** ORTEP drawing showing part of a layer in **1**. The interaction between Cu1 and the C8–C9 triple bond is instrumental in building the polymer. Selected bond distances (Å) and angles (°): C8–C9 1.190(5), Cu1–C8 2.053(3), Cu1–C9 2.054(4), Cu1–S1 2.4065(10), Cu1–Cl1 2.2614(9), Cu1–Cl1* 2.5845(10), C7–C8–C9 168.1(4), Cu1–Cl1–Cu1* 83.58(3), Cl1–Cu1–Cl1* 96.42(3), Cl1–Cu1–S1 107.44(4).

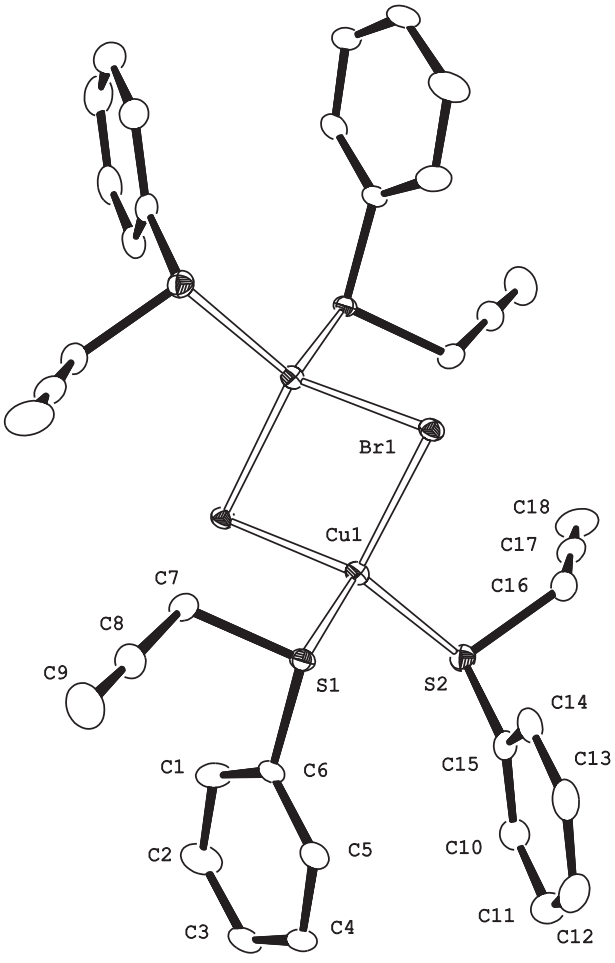



**Fig. 2.** ORTEP drawing of the dimer in **2**; the alkyne groups do not bridge. Selected bond distances (Å) and angles (°): C8–C9 1.174(4), C17–C18 1.179(4), Cu1–S1 2.3159(8), Cu1–S2 2.3085(7), Cu1–Br1 2.4756(6), Cu1–Br1* 2.4939(5), Cu1–Cu1* 3.0062(7), C7–C8–C9 179.1(3), C16–C17–C18 179.8(4), S1–Cu1–Br1 107.15(2), Br1–Cu1–Br1* 105.553(13), Cu1–Br1–Cu1* 74.447(13).

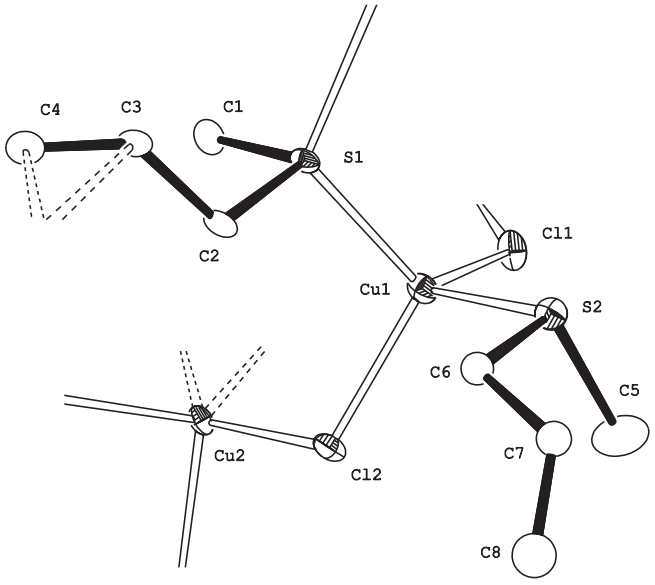



**Fig. 3.** ORTEP drawing of the asymmetric unit in **3**. Layers extend in the *ac* plane, via sulfide and chloride bridging. Selected bond distances (Å) and angles (°): C3–C4 1.343(9), Cu2–C3* 2.119(6), Cu2–C4* 2.118(6), C7–C8 1.35(2), Cu1–Cl1 2.3307(16), Cu2–Cl2 2.2930(16), Cu1–Cl2 2.3629(16), Cu1–S1 2.2733(16).

triple bond is somewhat lengthened to 1.190(5) Å and the C7–C8–C9 bond angle of 168.1(4)° deviates from the 180° required by sp hybridization. These changes in ligand geometry upon coordination to Cu1 are quite small and indicate that π back donation is weak, as expected in copper(I) complexes [15]. That the S atom in itself is terminal means that it has a free electron pair, which could be used in a subsequent enantioselective oxygen transfer reaction to form the corresponding chiral sulfoxide. Such a reaction requires that an enantiopure crystal, *i.e.* a conglomerate, can be prepared. Crystals of **1** are however racemic; they belong to the centrosymmetric space group *Pbcn*.

While trying to investigate if the CuBr analog of **1** crystallized as a conglomerate, we instead obtained $[Cu_2Br_2(Sprop)_4]$ (**2**). This complex is a discrete dimer (Fig. 2) where none of the triple bonds in the Sprop ligand are coordinated; the C8–C9 bond distance is 1.174(4) Å, while the C7–C8–C9 bond angle is 179.1(3)°. That CuBr is less prone to coordinate alkenes and alkynes than CuCl should perhaps not come as a surprise; this tendency has been reported previously [16]. All sulfur atoms in **2** are chirogenic, this is an effect of coordination to Cu1. The dimer itself is not chiral because two of the sulfur atoms have the *R* configuration and two have the *S* configuration.

Encouraged by the formation of layers via π-complexation in **1**, we decided to use CuCl with another functionalized sulfide ligand: allyl methyl sulfide (Sally). This sulfide is extra interesting since the allyl group is prochiral in itself, which opens up the possibility of selective attack on one side of the double bond in a chiral crystal. By using the same simple preparative method as for **1** and **2** (the neat ligand is used as solvent), we could isolate crystals of $[CuCl(Sally)]_n$ (**3**). As indicated in Fig. 3, a coordination polymer is indeed formed. Unfortunately, half of the sulfide ligands (C5–S2–C6–C7–C8) are disordered, possibly because the allyl group is not coordinated by copper. The other Sally ligand bridges three copper atoms; the double bond is coordinated and both electron pairs on S1 are engaged in bonding to copper. While this is good in the sense that it assists in formation of a layer, it is unfortunate because S1 is no longer accessible for oxidation. The low precision in the geometrical data for the disordered Sally ligand precludes a meaningful comparison between the coordinated and free ligands. Although there are elements of chirality in the layers in **3** (such as the coordinated double bond and the S atom in the disordered sulfide), each layer as well as the whole crystal is centrosymmetric.

Mesitylcopper (CuMes) is a very useful starting material for a variety of organocopper and -cuprate complexes [11]. The main reasons are that CuMes is soluble in many organic solvents (most other homoleptic organocopper complexes form insoluble polymers) and that CuMes is reasonably easy and safe to handle (alkylcopper complexes may be explosive when isolated and dried). Mesitylcopper is known to aggregate, and in benzene an equilibrium between tetramers and pentamers exists [11]. We decided to see if Sally could break or bridge such aggregates. As previously, we used the neat sulfide ligand as solvent and could isolate crystals of $[Cu_4(Mes)_4(Sally)_2]$ (**4**). A tetrameric $Cu_4Mes_4$ core is retained (Fig. 4) despite complexation of two Sally ligands by each tetramer. The allyl groups are not coordinated and it seems like π-complexation is not sufficient to break up the tetramer. Interestingly, both chirogenic sulfur atoms, in each molecule of **4**, have the same configuration. In Fig. 1 both sulfur atoms have the *R* configuration; the whole tetranuclear complex is thus chiral. Unfortunately, $[Cu_4(Mes)_4(Sally)_2]$ crystallizes in the centrosymmetric *C*2/*c* space group, which means that crystals of **4** are racemic.

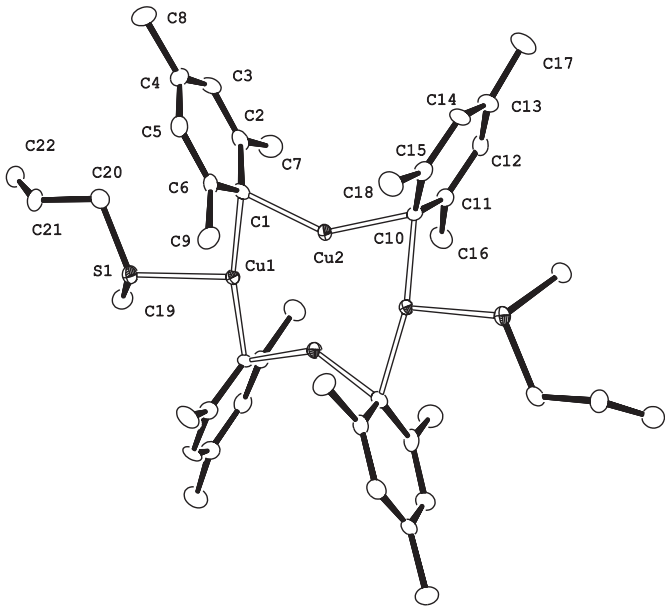



**Fig. 4.** ORTEP drawing of a discrete tetranuclear aggregate in **4**. Selected bond distances (Å) and angles (°): C21–C22 1.314(7), Cu1–C1 2.069(4), Cu2–C1 2.020(4), Cu2–C10 2.007(4), Cu1–S1 2.3455(12), Cu1–Cu2 2.4220(8), Cu2–Cu2* 2.6253(12), C20–C21–C22 124.0(5), C1–Cu1–S1 99.52(12), Cu1–C1–Cu2 72.63(15), C19–S1–Cu1 109.70(17).

All molecules in **4** are chiral; if one could link such molecules together it should be possible to construct homochiral helices. We thus decided to replace the weakly coordinating allyl group in Sally for a second sulfide group, resulting in the 2,5-dithiahexane ligand (SS). Addition of this ligand to a toluene solution of CuMes resulted in precipitation of a yellow powder, which could be recrystallized to give $[Cu_4(Mes)_4(SS)]_n$ (**5**). A crystal structure determination (Fig. 5) revealed that we indeed had been successful in linking tetrameric $Cu_4Mes_4$ units by the SS ligand to form polymeric chains. Similar stacking of $Cu_4Ar_4$ units has been previously reported [23]. Unfortunately, the two sulfur atoms in **5** have different configurations so that racemic chains are formed instead of the desired helices.

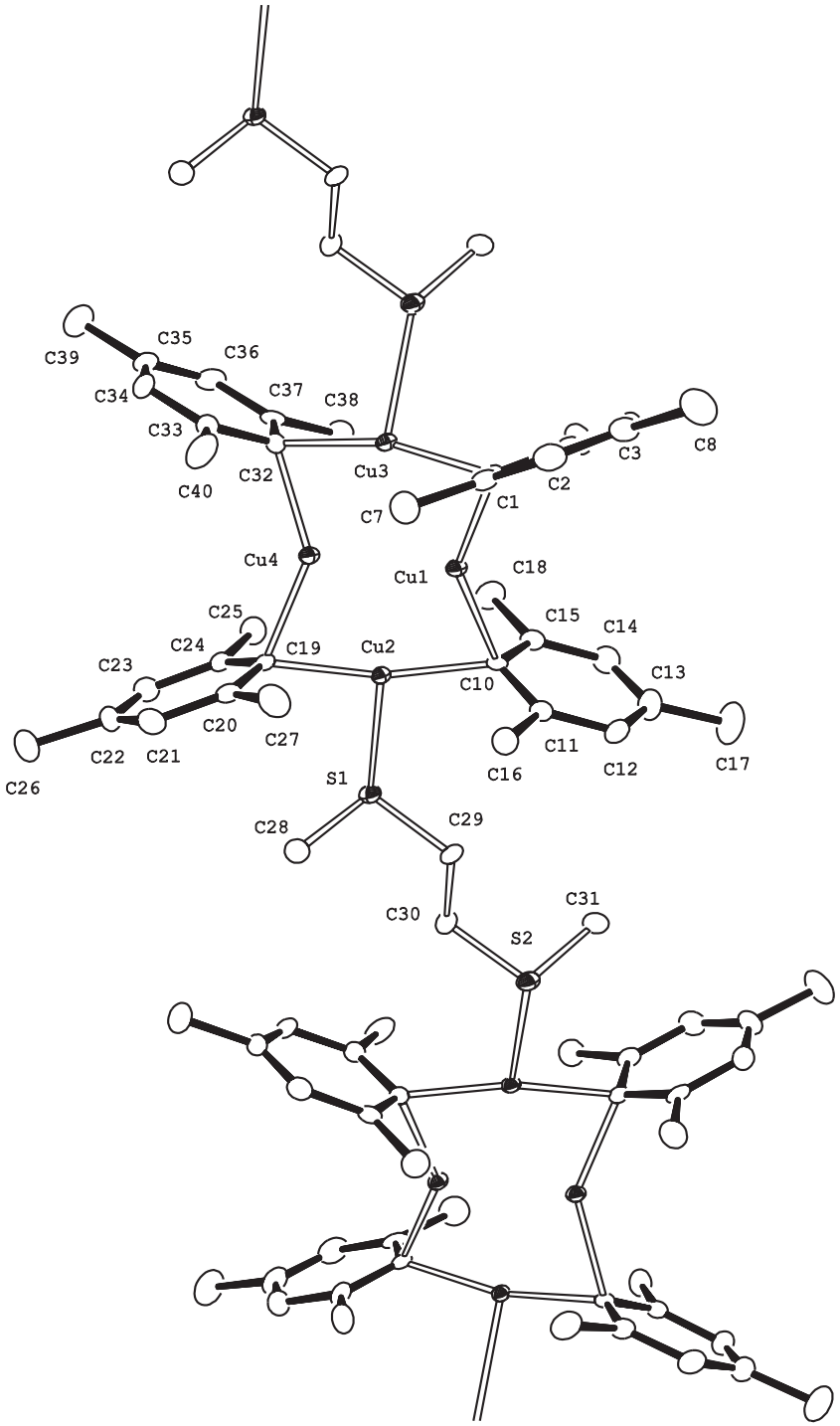
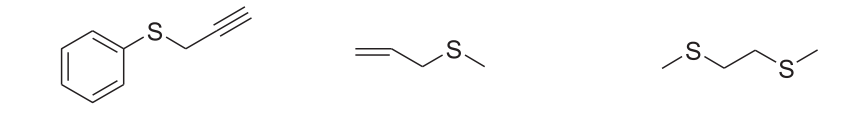



**Fig. 5.** ORTEP drawing showing how the bridging SS-ligand form chains in **5**. Selected bond distances (Å) and angles (°): Cu2–S1 2.3459(9), Cu3–S2 2.3565(9), Cu1–Cu2 2.4526(7), Cu3–Cu4 2.4581(7), Cu1–Cu4 2.5773(6); C10–Cu1–C1 141.28(13); C19–Cu2–C10 163.76(13).




phenyl propargyl sulfide (Sprop) allyl methyl sulfide (Sally) 2,5-dithiahexane (SS)

**Scheme 2.** Sulfide ligands used in **1**–**5**.

**Table 2**
Aggregation and coordination mode in **1**–**5**.

| Complex | Aggregate | CuX:L | L[a] |
|---|---|---|---|
| (**1**) $[CuCl(Sprop)]_n$ | Layer | 1:1 | 1:0 |
| (**2**) $[Cu_2Br_2(Sprop)_4]$ | Dimer | 1:2 | 1:0 |
| (**3**) $[CuCl(Sally)]_n$ | Layer | 1:1 | 1:1 |
| (**4**) $[Cu_4Mes_4(Sally)_2]$ | Tetramer | 2:1 | 2:0 |
| (**5**) $[Cu_4Mes_4(SS)]_n$ | Chain | 4:1 | 1:0 |

[a] Terminal:bridging sulfide ligands.

## 4. Conclusions

Functionalized ligands like allyl methyl sulfide and phenyl propargyl sulfide are suitable to use with copper(I) chloride to form coordination polymers. Copper(I) bromide frameworks may be less well suited, due to their lower tendency to form π-complexes. The 2,5-dithiahexane ligand can be used to link mesitylcopper aggregates to form chains or potentially helices. All five complexes (**1**–**5**) exhibit terminal sulfide ligands that may be oxidized selectively when incorporated in an enantiopure polymer. Unfortunately none of **1**–**5** crystallized as a conglomerate, but whether this reflects an inherent tendency in this system is too early to say. Complexes **1**–**5** are highly sensitive to air, which could prove to be valuable in oxidation reactions [24] once a conglomerate has been isolated.

## Acknowledgments

Financial support from the Swedish Research (VR) is gratefully acknowledged.

## Appendix. ASupplementary material

CCDC 818801, 818803, 818802, 818800 and 818799 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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