

A new two-dimensional Ag^{I} coordination polymer with $\text{Ag} \cdots \text{C}$ interactions: Thermal, fluorescence, structural and solution studies

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Abstract

A 2D polymer with $\text{Ag} \cdots \text{C}$ interactions, $[\text{Ag}(\mu_5\text{-TS})]_n$ (**1**) [TS^- = toluene-4-sulfonate], has been synthesized and characterized and its structure was determined by X-ray crystallography. In addition to coordination of the O atoms of TS^- , the Ag atoms also form weak $\eta^1 \text{Ag} \cdots \text{C}$ interactions. The thermal stability of **1** was studied by thermal gravimetric (TG) and differential thermal analyses (DTA). The ligand and compound **1** are luminescent in DMF, with emission maxima at 358 and 335, respectively. Solution studies of complex **1** were done in DMF and in CH_3CN .

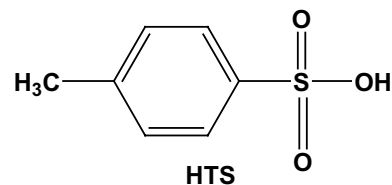
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1. Introduction

The self-assembly approach to the construction of supramolecules or extended frameworks based on coordination complexes is one of the major current research areas in inorganic and organometallic chemistry [1]. In the vast majority of such compounds, the molecular building blocks are held together by strong metal–ligand–metal bonding interactions or weaker forces such as hydrogen bonding between ligands coordinating to the different metal units. The use of bridging ligands for the controlled self-assembly of one-, two- or three-dimensional metallosupramolecular species has been the subject of enormous study in recent years [2]. In coordination polymers, the silver(I) ions give rise to an interesting array of stereochemistries and geometric configurations with the coordination numbers of two to six all occurring [3–6]. Previous investigations

have addressed that metal–heteroatom [7], metal–carbon [8] and metal–metal [9] interactions are three of the most important interactions in the construction of Ag polymeric networks in the solid state. However, although the closed d^{10} configuration of silver(I) appears to cancel any intermetallic bonding in silver(I) complexes, there are many examples of dimeric or polymeric silver(I) complexes with definite Ag/Ag attractive forces known as argentophilic interactions [9]. In this paper we describe the synthesis and coordination behaviour of TS^- ligand based on O,C-donor atoms. In addition thermal, emission and solution studies of compound **1** were also studied.



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2. Experimental

2.1. Materials and physical techniques

All chemicals were of reagent grade and were used as commercially obtained without further purification. IR spectra were recorded using Perkin–Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN–O– Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. The thermal behavior was measured with a PL–STA 1500 apparatus. The luminescent properties were investigated with a Shimadzu RF–5000 spectrofluorophotometer. All UV–Visible spectra were recorded on a computerized double-beam Shimadzu 2550 spectrophotometer, using two matched 10-mm quartz cells. Conductometric measurements were carried out with a Metrohm 712 conductometer equipped with a Julabo F12–MB circulator. Crystallographic measurements were made at 100(2) K using a Bruker SMART APEX CCD area detector. The intensity data were collected using graphite monochromated Mo K α radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and the orientation matrix were obtained from least-squares refinement. The structure has been solved by direct methods and refined by full-matrix least-squares techniques on F^2 using SHELXTL [10]. The molecular structure plots were prepared using ORTEP and MERCURY [11]. Crystallographic data and details of the data collection and structure refinements are listed in Table 1. The observed anisotropic thermal parameters, the calculated structure factors, and full lists of bond distances, bond angles and torsion angles are given in the supplementary material.

2.2. Synthesis of $[Ag(\mu_5-TS)]_n$ (**1**)

In 10 ml methanol 0.172 g (1 mmol) toluene-4-sulfonic acid (HTS) were mixed and stirred with a solution of 0.057 g (1 mmol) KOH in 3 ml H₂O to form a clear solution. Addition of 0.170 g (1 mmol) Ag(NO₃) in 5 ml H₂O produced a clear solution that was stirred and then allowed to stand in darkness at room temperature to evaporate for several days to obtain crystals suitable for single crystal X-ray diffraction. The crystals were washed with acetone and air dried, m.p. = 250 °C, Yield: 0.131 g (47%). IR (selected bands; in cm⁻¹): 535m, 559s, 687s, 806s, 843w, 947w, 1002s, 1034s, 1125vs, 1189vs, 1397w, 1437w, and 1485w. Anal. Calc. for C₇H₇AgO₃S: C, 30.10; H, 2.51; Ag, 38.34%. Found: C, 29.95; H, 2.45; Ag, 38.90%.

3. Results and discussion

3.1. Structure description

The reaction between toluene-4-sulfonate (TS⁻) and Ag(NO₃) provided a crystalline material of the general formula $[Ag(\mu_5-TS)]_n$ (**1**). Determination of the structure of **1**

Table 1
Crystal data and structure refinement for compound $[Ag(\mu_5-TS)]_n$ (**1**)

Identification code	1
Empirical formula	C ₇ H ₇ AgO ₃ S
Formula weight	279.06
Temperature (K)	100(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
<i>a</i> (Å)	15.444(6)
<i>b</i> (Å)	6.050(2)
<i>c</i> (Å)	8.587(3)
α (°)	90.00
β (°)	94.035(7)
γ (°)	90.00
Volume (Å ³)	800.4(5)
<i>Z</i>	4
Density (calculated)	2.316 g cm ⁻³
Absorption coefficient	2.736 mm ⁻¹
<i>F</i> (000)	544
Crystal size	0.46 × 0.32 × 0.08 mm ³
Theta range for data collection (°)	2.64–28.28
Index ranges	$-20 \leq h \leq 20$, $-8 \leq k \leq 8$, $-11 \leq l \leq 11$
Reflections collected	7027
Independent reflections	1969 ($R(\text{int}) = 0.0567$)
Absorption correction	Multi-scan
Maximum and minimum transmission	0.803 and 0.328
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	1969/0/110
Goodness-of-fit on F^2	1.047
Final <i>R</i> indices [$I > 2\sigma(I)$]	$R_1 = 0.0784$, $wR_2 = 0.2112$
<i>R</i> Indices (all data)	$R_1 = 0.0897$, $wR_2 = 0.2237$
Largest difference in peak and hole (e Å ⁻³)	8.358 and -3.198

by X-ray crystallography showed the complex to be a two-dimensional polymer (Figs. 1–3). The silver atoms can be considered to be five-coordinate (Fig. 2). The sulfonate group of the TS⁻ ligand acts as a bridging group where each oxygen atoms of the sulfonate group coordinates to a silver(I) ion, and two of these oxygen atoms also bridge to another two silver atoms (Scheme 1). The structure of compound **1** may also be considered as a coordination polymer consisting of two-dimensional sheets constructed from Ag(I) ions and bridging sulfonate groups, running parallel to the *bc* sheet and the individual polymeric layers are almost parallel to each other along the *a* axis, resulting in two-dimensional sheets as shown in Fig. 3.

The structure was analyzed for close Ag...Ag and Ag...C contacts. The closest Ag–Ag interaction in compound **1**, Ag1–Ag1 = 3.368 Å (Table 2), is longer than the Ag–Ag distances in similar dinuclear complexes (i.e., 2.704(2), 2.669(1), and 2.726(1) Å) [9b,12–15] and also longer than the twice the van der Waals radius of Ag (3.44 Å), [16], indicating that there are no argentophilic interactions in compound **1**. On the other hand it appears that the silver ions may be involved in Ag...C (η^1) interactions between the silver atoms and phenyl groups of TS⁻

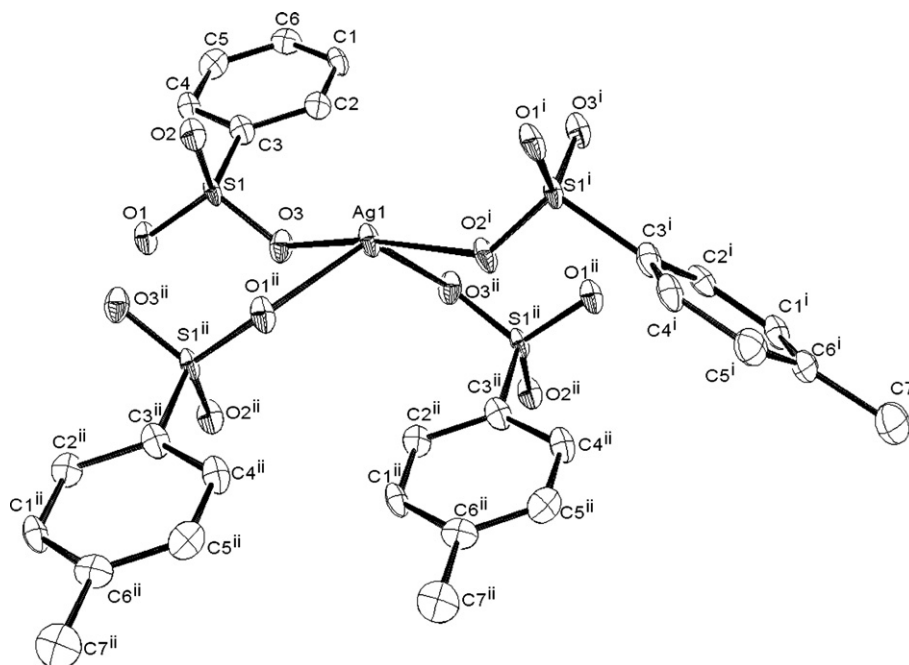


Fig. 1. ORTEP diagram of compound $[\text{Ag}(\mu_5\text{-TS})]_n$ (**1**).

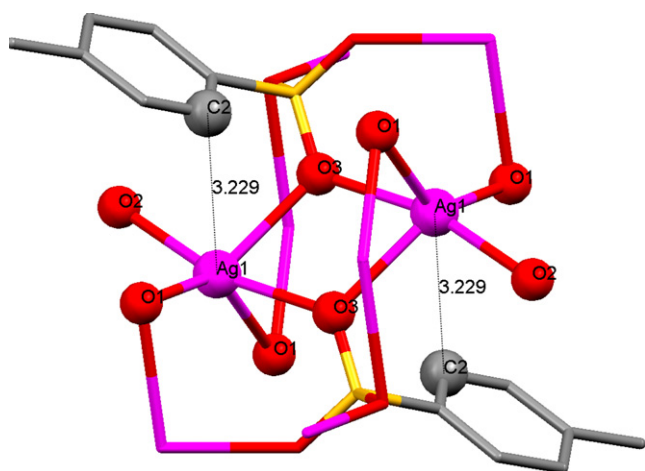


Fig. 2. Environment of the Ag-atoms in compound **1** after extending the bonding limit to show the $\text{Ag}\cdots\text{C}$ interactions.

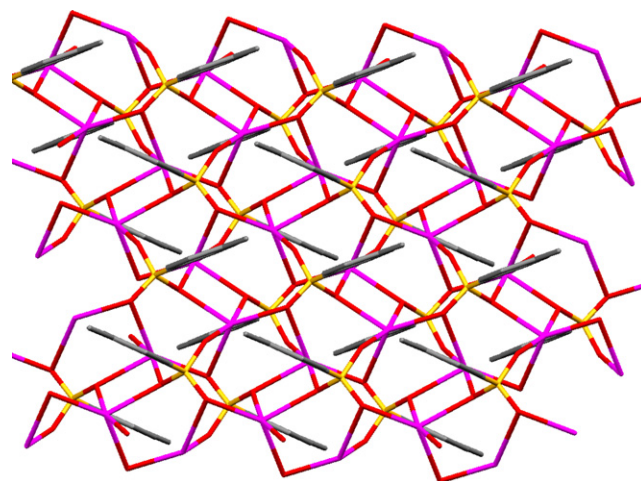
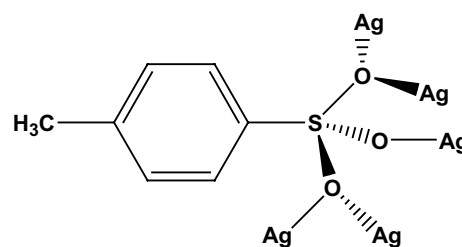


Fig. 3. A fragment of the two-dimensional polymer in compound **1**. H atoms are omitted for clarity.

(Fig. 2), with a $\text{Ag}(1)\text{-C}(2)$ distance of 3.229 Å, thus augmenting the $\text{Ag}^{\text{I}}\text{Ag1O}_5$ coordination sphere, and compound **1** can be considered to contain silver atoms with an $\text{O}_5\text{Ag}\cdots\text{C}$ environment. In compounds with similar interactions the $\text{Ag}\cdots\text{C}$ distances are in the range of 2.40–2.70 Å [17–20], in $[\text{Ag}(\text{benzene})\text{ClO}_4]$ for example they are 2.496 and 2.634 Å [14]. Some other $\text{Ag}(\text{I})$ polymeric complexes with $\text{Ag}\text{-C}(\text{sp}^2)$ bonded polycyclic aromatic ligands have been reported to have mean $\text{Ag}\cdots\text{arene}$ distances of 2.82–3.37 Å [6,21–24]. Thus, the interaction in compound **1** can be considered as a weak monohapto aromatic coordination to the Ag atoms.



Scheme 1. The coordination mode of ligand TS^- in compound **1**.

4. Thermogravimetric analysis

To examine the thermal stability of compound **1**, thermal gravimetric (TG) and differential thermal analy-

Table 2
Selected bond lengths (Å) and angles (°) for compound $[\text{Ag}(\mu_5\text{-TS})]_n$ (**1**)

Ag(1)–O(2)#1	2.392(5)	O(2)#1–Ag(1)–O(1)#2	96.54(18)
Ag(1)–O(1)#2	2.433(5)	O(2)#1–Ag(1)–O(3)	165.87(18)
Ag(1)–O(3)	2.452(5)	O(1)#2–Ag(1)–O(3)	90.43(18)
Ag(1)–O(3)#3	2.537(5)	O(2)#1–Ag(1)–O(3)#3	93.86(18)
Ag(1)–Ag(1)#4	3.3682(12)	O(1)#2–Ag(1)–O(3)#3	132.54(18)
Ag(1)–Ag(1)#1	3.3682(12)	O(3)–Ag(1)–O(3)#3	72.5(2)
		O(1)#2–Ag(1)–Ag(1)#4	52.77(13)
O(3)–Ag(1)–Ag(1)#4	80.74(13)	O(3)#3–Ag(1)–Ag(1)#4	152.33(12)
O(2)#1–Ag(1)–Ag(1)#1	72.85(13)	O(1)#2–Ag(1)–Ag(1)#1	168.89(12)
O(3)–Ag(1)–Ag(1)#1	100.65(13)	O(3)#3–Ag(1)–Ag(1)#1	53.41(13)
Ag(1)#4–Ag(1)–Ag(1)#1	127.83(4)	O(2)#1–Ag(1)–Ag(1)#4	113.24(13)

#1: $-x + 2, y - 1/2, -z - 1/2$; #2: $-x + 2, -y + 1 - z$; #3: $-x + 2, -y, -z$; #4: $-x + 2, y + 1/2, -z - 1/2$.

ses (DTA) were carried out between 30 and 700 °C in a static atmosphere of air (Fig. S1). Compound **1** melts at 250 °C with an endothermic peak at this temperature. The TG curve exhibits two decomposition stages between 325 and 475 °C with a mass loss of 61.9% (calcd 61.3%). The DTA curve displays three endothermic peaks with a maximum intensity at 250, 338 and 359 °C associated with a first decomposition state and a very sharp exothermic peak at 465 °C associated with a second decomposition state. Mass loss calculations of the end residue show that the final decomposition product is Ag_2O .

5. Luminescent properties

The UV/Vis and emission spectra of compound **1** as well as the pure ligand HTS were studied. The UV/Vis spectra of both ligand and complex in DMF display intense absorption bands with the maximum intensity at 272 nm (Fig. 4), indicating that electronic transitions are mostly of π to π^* character, originating from the aro-

matic groups of the ligand [25]. In DMF emission bands have been observed both for compound **1** and also the ligand HTS at 335 and 358 nm upon excitation at 300 nm, respectively (Fig. 4). Compared with the emission of the free ligand HTS, a blue shift of ca. 23 nm has been observed upon the coordination of the TS^- ligand to Ag(I) atoms in **1** [8m]. It should be noted that luminescent silver(I) compounds, usually exhibiting emission at low temperature, are unstable organometallic compounds containing chalcogenides and acetylides as bridging ligands [26]. Only a few monomeric [27] and polymeric [28] silver(I) complexes that exhibited luminescent properties at room temperature have been reported. However, our current complex is strongly luminescent at ambient temperature.

6. Solution studies

In the spectrophotometric analysis, 2.0 ml of the ligand solution (2.0×10^{-4} M) in DMF was deprotonated with KOH. The solution was placed in the spectrophotometer cell and the absorbance of solution was measured. Then a known amount of the concentrated solution of silver(I) nitrate in DMF (5.2×10^{-3} M) was added in a stepwise manner using a 5- μl Hamilton syringe. The absorbance spectrum of the solution was recorded after each addition. The electronic absorption spectra of the ligand toluene-4-sulfonate (TS^-) in the presence of increasing concentrations of silver(I) ions at room temperature are shown in Fig. S2. The strong absorption of the ligand at 272 nm increases with increasing concentration of the metal ion. The absorbance, measured against the $[\text{Ag}^+]/[\text{TS}^-]$ mole ratio as shown in the inset of Fig. S2, revealed no inflection point at a metal-to-ligand molar ratio of about 1. Similar spectrophotometric studies were also done in CH_3CN solution, but also no inflection point was observed at a metal-to-ligand molar ratio of about 1 (Fig. S3). The formation and stoichiometry of the $\text{Ag}^+ - \text{TS}^-$ complex in DMF and CH_3CN solution was also investigated by a conductometric analysis. 20.0 ml of a silver(I) nitrate solution (5.0×10^{-5} M) in DMF was placed in a two-wall thermostated glass cell, the temperature was adjusted to 25.00 ± 0.05 °C, and the conductance of the

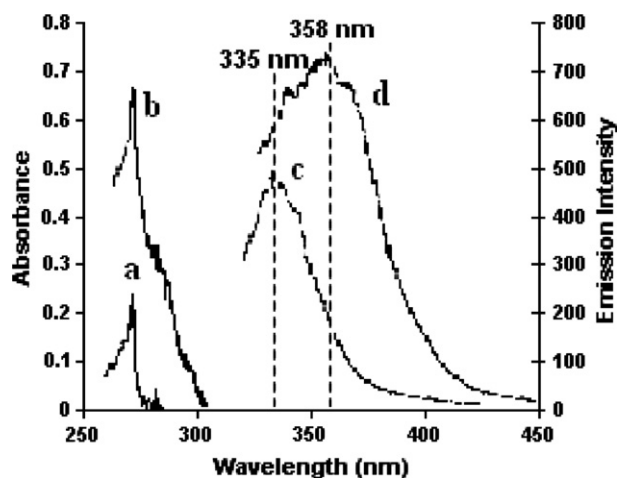


Fig. 4. Electronic absorption of ligand HTS (a), electronic absorption of compound **1** (b). Absorption: $c = 5.0 \times 10^{-2}$ mol l^{-1} , DMF, $d = 1$ cm for both ligand HTS and compound **1**. Solution-state emission spectrum for compound **1** (c). Solution-state emission spectrum for ligand HTS (d). Room temperature, $\lambda_{\text{exc}} = 300$ nm.

solution was measured. Then a known amount of the concentrated solution of ligand (TS^-) in DMF (5.0×10^{-3} M) was added in a stepwise manner using a 5- μl Hamilton syringe. The conductance of the solution was measured after each addition. The ligand solution was continually added. The conductivity of a 5.0×10^{-5} M solution of silver(I) nitrate in DMF was monitored as a function of $[\text{TS}^-]/[\text{Ag}^+]$ mole ratio at 25.00 ± 0.05 °C and the resulting plot is shown in Fig. S4. Addition of the ligand to the metal salt solution causes a linear increase in the solution conductivity with no inflection point at a molar ratio of about one. Similar conductometric studies were also done in CH_3CN solution, but also no inflection point was observed at a metal-to-ligand molar ratio of about 1 (Fig. S4).

With the absence of a visible inflection point, we could not evaluate the conditional formation constants from the spectrophotometric and conductometric methods as would be otherwise possible [29]. Emission studies of compound **1** in DMF solution show that we could consider interactions between TS^- and Ag^+ in solution state, thus we would have to conclude from the basically linear increase of the absorbance and conductance in Figs. S2–S4 that the ligand TS^- shows similar interactions with each of the varying amounts of metal ion concentrations up to a molar ratio of about 3 and 4 in both spectrophotometric and conductometric methods, respectively. These results of the stoichiometry and formation studies of complex of **1** in DMF and CH_3CN solution, however, are not supported by the solid state stoichiometry.

7. Conclusions

A new Ag^{I} two-dimensional coordination polymer of toluene-4-sulfonic acid (HTS) ligand, $[\text{Ag}(\mu_5\text{-TS})]_n$, was synthesized and characterized. One of the $\text{C}_{\text{aromatic}}$ TS^- ligand in compound **1** exhibits a weak interaction with Ag^+ ion, in addition to the normal sulfonate coordination modes. The individual two-dimensional sheets in the compound $[\text{Ag}(\mu_5\text{-TS})]_n$ are almost parallel to each other along the a axis. The results of studies of the stoichiometry and formation of $[\text{Ti}(\mu_4\text{-AB})]_n$ in DMF solution were not found to be in support of their solid state stoichiometry. Compound **1** is one of only very few stable monomeric and polymeric silver(I) complexes that exhibit luminescent properties at room temperature.

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Appendix A. Supplementary material

CCDC 659320 contains the supplementary crystallographic data for **1**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jorgchem.2007.10.057](https://doi.org/10.1016/j.jorgchem.2007.10.057).

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