SYNTHESIS AND CRYSTAL STRUCTURE OF Ag(I) \(p\)-TOLUENESULPHONATE COMPLEX WITH 5-METHYL-3-ALLYL-2 THIOHYDANTOIN

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New silver(I) toluenesulphonate coordination compound \([\text{Ag}_2(\text{amth})_4(\text{TsO})_2]\) (I) (where \(\text{amth}=3\)-allyl-5-methyl-2-thiohydantoin, \(\text{TsO}\)= \(p\)-toluenesulphonate anion) has been synthesized by direct interaction between \(\text{amth}\) ligand, \(\text{Ag}_2\text{CO}_3\) and \(\text{p-CH}_3\text{C}_6\text{H}_4\text{SO}_3\text{H}\) in \(n\)-propanol and studied by X-ray single crystal diffraction method. Complex I crystallizes in the triclinic centrosymmetric space group \(P-\overline{1}\) with one silver(I) atom, one \(\text{TsO}\)– anion and two \(\text{amth}\) molecules in the asymmetric unit, with the following cell parameters: 

\[
a=10.411(4) \ \text{Å}, \quad b=11.516(4) \ \text{Å}, \quad c=12.772(4) \ \text{Å}, \quad \alpha=109.15(3)^\circ, \quad \beta=107.75(3)^\circ, \quad \gamma=105.27(3)^\circ, \quad V=1260.3(8) \ \text{Å}^3.
\]

The structure of compound I is built of dimeric binuclear \([\text{Ag}_2(\text{amth})_4(\text{TsO})_2]\) fragments within which a weak Ag–Ag interaction was observed (Ag–Ag bond distance is equal to 3.0897(13) Å). Ag atom in I has a distorted tetrahedral environment (\(\tau=0.92\)), formed by three \(\text{amth}\) ligand’s S atoms and one O atom of \(\text{TsO}\)– anion. Both crystallographically independent \(\text{amth}\) molecules are coordinated to the metal centers only through their thiogroup’s S atoms. In order to analyze weak interactions in I, Hirshfeld surface for some structural fragments were built and discussed. Unexpected interaction between oxogroup’s O atom of one \(\text{amth}\) molecule and the ring of another one \(\text{amth}\) molecule was observed.

**Keywords:** silver(I), toluenesulphonate, coordination compound, 2-thiohydantoin, crystal structure.

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**Introduction**

Heterocyclic compounds based on 2-thiohydantoin (2-thioxoimidazolidin-4-one) fragment are well known in analytical chemistry, in particular as reagents for the determination of \(d\)-metals (such as \(\text{Pd(II)}, \text{Cu(II)}, \text{Cu(II)}, \text{Ag(I)}\) and \(\text{Hg(II)}\)) due to the strong complexes formation [1] in which ligand coordination behavior is realized thanks to a simultaneous presence of thiocarbamide >C=S, carbonyl >C=O as well as >N–H donor groups. 2-Thiohydantoin derivatives have been reported to exhibit anticancer, antimalarial, anti-diabetic treatment as well as other valuable biological properties [2]. One of the methods to increase mentioned properties is a formation of corresponding coordination compounds which are also reported to have antimicrobial activity [3]. Moreover, 2-thiohydantoin derivatives exhibit fluorescence sensing properties towards Cu\(^{2+}\) in corresponding complexes [4].

In our previous works, a series of silver complexes of 3-allyl-2-thiohydantoin in the presence of benzenesulphonate and toluenesulphonate anions was studied and unexpected coordination behavior of this organic ligand in the reaction with Ag(I) was shown [5,6]. In the course of these studies, in order to investigate the coordination features of the another 2-thiohydantoin allyl derivative, namely 3-allyl-5-methyl-2-thiohydantoin (amth) towards silver(I) \(p\)-toluensulfonate, we carried out a synthesis and X-ray diffraction analysis with further detailed structure description of new complex \([\text{Ag}_2(\text{amth})_4(\text{TsO})_2]\) (I).

**Experimental**

**General consideration**

Unless mentioned otherwise, all chemicals were obtained from commercial sources and used without further purification. The NMR experiments: \(^1\text{H}\) NMR (500 MHz), \(^{13}\text{C}\{^1\text{H}\}\) NMR (125 MHz) were recorded using a Bruker Avance 500 MHz NMR spectrometer. The chemical shifts are reported in ppm relative to the residual peak of the deuterated
Synthesis and crystal structure of Ag(I) p-toluenesulphonate complex with 5-methyl-3-allyl-2-thiohydantoin

Table 1

| Parameter | Value |
|-----------|-------|
| Empirical formula | C_{42}H_{54}Ag_{2}N_{8}O_{10}S_{6} |
| CCDC number | 1871030 |
| Formula weight (g·mol^{-1}) | 1239.03 |
| Crystal system | Triclinic |
| Space group | P–1 |
| a (Å) | 10.411(4) |
| b (Å) | 11.516(4) |
| c (Å) | 12.772(4) |
| α (°) | 109.15(3) |
| β (°) | 107.75(3) |
| γ (°) | 105.27(3) |
| V (Å^3) | 1260.3(8) |
| Z | 1 |
| μ (mm^{-1}) | 1.09 |
| F(000) | 632 |
| Calculated density (g cm^{-3}) | 1.633 |
| Crystal size (mm) | 0.33 × 0.12 × 0.06 |
| Crystal color and shape | Clear colorless prism |
| Diffractometer and detector type | Kuma KM4CCD |
| Radiation type, wavelength, λ (Å) | MoKα, 0.71073 |
| Temperature, K | 120 |
| θ range for data collection (°) | 3.0–28.8 |
| Index ranges | –13<½<10 |
| –14<½<15 |
| –17<½<16 |
| Measured reflections | 8524 |
| Independent reflections | 5816 |
| Observed refl. (I>2σ(I)) | 4582 |
| R_{int} | 0.052 |
| R[F^2>2σ(F^2)] | 0.063 |
| wR(F^2) | 0.172 |
| Goodness of fit | 1.09 |

Results and discussion

Complex I crystallizes in the triclinic centro-symmetric space group P–I with one silver(I) atom,
The structure of compound I is built of dimeric binuclear \([\text{Ag}_2(\text{amth})_4(\text{TsO})_2]\) fragments (Fig. 1). Both silver atoms are bonded among themselves through two S atoms of two \textit{amth} ligand molecules (Fig. 2). As a result, flat four-membered \{\text{Ag}_{2}\text{S}_{2}\} ring is formed (S1–Ag1–S1\(^{i}\) angle is equal to 108.28(6)\(^\circ\)). Ag–Ag\(^{i}\) distance within it is equal to 3.0897(13) Å (Table 2), that is shorter than the doubled van der Waals radii of Ag (3.44 Å) reported by Bondi and significantly shorter than the corresponding sum according to Batsanov and Alvarez [10] (4.2 and 4.06 Å, respectively) that shows availability of weak Ag–Ag interaction. Coordination environment of each silver atom also includes one exocyclic S2 atom of second \textit{amth} ligand molecule and O11 atom of TsO\(^{–}\) anion forming distorted tetrahedral arrangement (\(\tau=0.92\)) [11]. Within this coordination polyhedron two Ag–S distances between silver atom and S atoms are very close (2.5491(18) and 2.5049(16) Å) and are significantly shorter then to the third one (2.720(2) Å).

![Fig. 1](image1.png)

Fig. 1. Dimeric \([\text{Ag}_2(\text{amth})_4(\text{TsO})_2]\) fragment in the crystal structure I. Hydrogen atoms are removed for better visualization. Symmetry code: (i) 2–x, 1–y, 1–z

![Fig. 2](image2.png)

Fig. 2. Silver atoms and their closest surrounding with depicted bond length in the structure I. Symmetry code: (i) 2–x, 1–y, 1–z

Accordingly, both \textit{amth} molecules are coordinated to the metal centers only through their exocyclic S atoms. Neither carbonyl O atom nor C=C double bond of allyl group participates in coordination to the Ag(I) atom. It could be explained by larger thermodynamic stability of a complex, where, corresponding to the Pearson hard-soft acid-base theory, typical soft Lewis acid such as Ag\(^{+}\) is interconnected with a soft base as S atom in C=S group instead of O atom of C=O group. Although allylic C=C bond (as a soft base) is suitable for formation of stable Ag(I) \(\pi\)-complexes [12], thiogroup in I remains more preferable. Similar situation is also obtained with analogous \(p\)-toluene sulphonate \([\text{Ag}_2(L)_{2}(\text{CH}_3\text{C}_6\text{H}_4\text{SO}_3)_{2}]\) and benzenesulphonate \([\text{Ag}_2(L)_{2}((\text{C}_6\text{H}_5\text{SO}_3)_{2})\cdot0.5\text{C}_3\text{H}_7\text{OH}\) complexes with homologous 3-allyl-2-thiohydantoin ligand. In both these structures, there are three different coordination modes of silver atom (tetragonal pyramidal, seesaw and distorted octahedral) and four crystallographically
independent ligand molecules, all of which were coordinated through their thiogroup’s S atoms only [5,6].

In [Ag₄(amth)₄(TsO)₂] (I), dimeric fragment toluenesulphonate anion is not only bonded to the Ag atom through the O11 atom, but is also involved in a wide system of weak bonding. In order to analyze these interactions, Hirshfeld surface was built for structural {Ag₄(amth)₄}²⁺ fragment using CrystalExplorer software. Areas, where the most prominent interactions among atoms are, can be seen in the Hirshfeld surface plots as the red ones (Fig. 3). One can see a presence of two red areas corresponding to the N–H···O hydrogen bonding between amth ligand iminogroups and toluenesulphonate anion’s O atoms (Table 3). This system of hydrogen bonds stabilizes mentioned [Ag₄(amth)₄(TsO)₂] dimer. Another big red area, corresponding to the already discussed Ag1–O11 bond, is heavily painted in red, but there is another one smaller area near it slightly marked in red that responds to another one Ag1–O interaction, namely Ag1–O21 weak bonding (Ag1–O21 distance is equal to 3.015(4) Å).

In order to analyze weak interactions via [Ag₄(amth)₄(TsO)₂] dimers in I, packing Hirshfeld surface for the above dimer was built (Fig. 4). There are two main interdimeric interactions. One of them corresponds to weak C–H···O interaction, and another one, which at the Hirshfeld surface is represented as a group of three red areas, corresponds to the interaction between oxogroup’s O atom of one amth molecule and the ring of another one amth molecule. This interaction could be described as both electrostatic and VdW interaction. Since 2-thiohydantoin (2-sulfanylideneimidazolidin-4-one) ring contains thio- and oxogroup, which possess high electronegativity and pull electron density on S and O atom respectively, there is partial positive charge and lack of electron density on imidazolidin ring. In order to visualize the distribution of electron density, the 2-thiohydantoin molecule was optimized using DFT method with B3LYP functional supplemented and standard 6-31G(d) basis set using Gaussian W09 package [13] and electron density surface was built (Fig. 5). This plot shows that a negative charge is generally localized near ligand’s C=O and C=S groups (marked in yellow) at the time when the ring possesses generally positive charge (marked in blue).

Distance from O atom to the center of imidazolidin ring (Q) is equal to 2.831(8) Å and C–O–Q angle equals to 133.8(2)°. Analyzing distances from O atom to all atoms of the ring, one can notice that these distances are mainly shorter than the sum of the corresponding VdW radii, which

| Atoms involved | Symmetry | D–H···A | D–H | H···A | D···A | Angle, deg |
|----------------|----------|---------|------|-----|------|------------|
| N₂B–H₂B–O₁₁    |          | 0.88    | 1.94 | 2.7961(1) | 162 |
| N₂A–H₂A–O₂₁    | −x, 1−y, 1−z | 0.88    | 1.99 | 2.8208(1) | 156 |

**Table 3**

Geometry of selected hydrogen bonds in I

![Fig. 3. a – Hirshfeld surface analysis of structural \(\text{Ag}_4\text{amth}_4\text{TsO}_2\)²⁺ fragment in I. Hirshfeld surface is mapped with \(d_{norm}\) which highlights both donor and acceptor ability. b – Hydrogen bonds in I](image-url)
Table 4

| Atoms involved C-O-X | O--X distance, Å | Sum of VdW radii of O and X (Bondi) | Sum of VdW radii of O and X (Alvarez) | Angle, deg |
|----------------------|----------------|-------------------------------------|--------------------------------------|------------|
| C2B-O1B--N2A         | 3.159(8)       | 3.07                                | 3.16                                 | 114.1(5)   |
| C2B-O1B--C1A         | 3.083(8)       | 3.22                                | 3.27                                 | 115.9(5)   |
| C2B-O1B--N1A         | 3.027(8)       | 3.07                                | 3.16                                 | 138.8(5)   |
| C2B-O1B--C2A         | 3.026(8)       | 3.22                                | 3.27                                 | 157.5(5)   |
| C2B-O1B--C3A         | 3.082(8)       | 3.22                                | 3.27                                 | 132.9(5)   |
| C2B-O1B--H3A         | 2.63(2)        | 2.72                                | 2.70                                 | 125(2)     |

Fig. 4. a – Hirshfeld surface analysis of [Ag₆(amth)₄(TsO)₂] fragment in I. Hirshfeld surface is mapped with d_{norm} which highlights both donor and acceptor ability. b – C=O--ring interaction in I

Fig. 5. Electron density surface mapped with electrostatic potential for amth molecule
indicates the presence of interaction between O atom and the ring (Table 4). Nature of this interaction is quite debatable, but it could be treated as example of nonaromatic analog of lone pair–π interaction (lp–π), which is bonding association between a neutral electron-rich molecule and an electron-poor ring [14]. As a rule, the ring has to be aromatic one but lp–π interactions could be expanded to nonaromatic systems too [15].

Conclusions

New silver(I) toluenesulfonate complex of [Ag₂,(amth),(TsO)]) (where amth=3-allyl-5-methyl-2-thiohydantoin, TsO = p-toluenesulfonate anion) was obtained and its structure was determined and studied by X-ray single crystal diffraction. The structure of this complex is built of binuclear [Ag₂,(amth),(TsO)]) dimers in which Ag atom has a distorted tetrahedral environment (τₛ=0.92), formed by three amth ligand’s S atoms and one O atom of TsO⁻ anion. Also within this fragments, a weak Ag–Ag interaction (Ag–Ag distance is equal to 3.0897(13) Å) was observed. Both crystallographically independent amth molecules are coordinated to the metal centers through their thiogroup’s S atoms only. Unexpected interaction between oxogroup’s O atom of one amth molecule and the ring of another one amth molecule was discussed.

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СИНТЕЗ И КРИСТАЛЛИЧНА СТРУКТУРА n-ТОЛЮЕНСУЛЬФОНАТНОГО КОМПЛКЕСУ Ag(I) З 5-МЕТИЛ-3-АЛЛИЛ-2-ТИОГИДАНТОИМ

А.А. Федорчук, Ю.И. Сивка, М.Г. Масквас

Новий n-толюенсульфонатний комплекс Ag(I) складу [Ag(anth),(TsO)]₁₁ (anth=3-аліл-5-метил-2-тиогідантон) було одержано безпосередньо взаємодією між лігандом аптн, Ag₂O, та χ–СН₂,С₆Н₅SO₃Н у н-пропанолі та доданою метилгідроксиламино ноксирина – дифіксації. Комплекс I кристалізується в трицикличній центросиметричній просторовій груп P 1 і одним атомом аргенту (I), одним TsO-anіоном і двома молекулами ліганду аптн в асиметричній частині з параметрами комірки: a=10,411(4) Å, b=11,516(4) Å, c=12,772(4) Å, α=90,15(3)°, β=107,75(3)°, γ=105,27(3)°, V=2460,38(8) Å³. Структура сполук I побудована з димерних двосторонніх фрагментів [Ag(anth),(TsO)], в яких навна спілка вузлів Ag–Ag (дистанція Ag–Ag становить 3,0897(13) Å). Атом Ag в I має деформоване тетраедричне координаційне обертання (τₛ=0,92), утворене трьома атомами S ліганду аптн та одним атомом O

Synthesis and crystal structure of Ag(I) p-toluenesulfonate complex with 5-methyl-3-allyl-2-thiohydantoin
SYNTHESIS AND CRYSTAL STRUCTURE OF Ag(I) p-TOLUENE SULPHONATE COMPLEX WITH 5-METHYL-3-ALLYL-2-THIODYANTOIN

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New silver(I) toluenesulphonate coordination compound [Ag(amth)₂(TsO)₂] (I) (where amth=3-allyl-5-methyl-2-thiodyantoin, TsO=-p-toluenesulphonate anion) has been synthesized by direct interaction between amth ligand, Ag₂CO₃ and p-CH₃C₆H₄SO₂H in n-propanol and studied by X-ray single crystal diffraction method. Complex I crystallizes in the triclinic centrosymmetric space group P-1 with one silver(I) atom, one TsO- anion and two amth molecules in the asymmetric unit, with the following cell parameters: a=10.411(4) Å, b=11.516(4) Å, c=12.772(4) Å, α=109.15(3)°, β=107.75(3)°, γ=105.27(3)°, V=1260.3(8) Å³. The structure of compound I is built of digenic binuclear [Ag(amth)₂(TsO)₂] fragments within which a weak Ag–Ag interaction was observed (Ag–Ag bond distance is equal to 3.0897(13) Å). Ag atom in I has a distorted tetrahedral environment (τ=0.92), formed by three amth ligand’s S atoms and one O atom of TsO anion. Both crystallographically independent amth molecules are coordinated to the metal centers only through their thigroup’s S atoms. In order to analyze weak interactions in I, Hirshfeld surface for some structural fragments were built and discussed. Unexpected interaction between oxogroup’s O atom of one amth molecule and the ring of another one amth molecule was observed.

Keywords: silver(I); toluenesulphonate; coordination compound; 2-thiodyantoin; crystal structure.

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