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New 3D Coordination Polymer Based on the Tetrapyridyl Derivative of Thiacalix[4]arene in the 1,3-Alternate Configuration and Hexanuclear Clusters of Monovalent Silver: Synthesis and Structure

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Abstract—The reaction of the tetradentate ligand based on the tetrapyridyl derivative at the lower rim of thiacalix[4]arene (β-isomer) with silver(I) nitrate in the crystalline phase affords 3D coordination polymer in which the polynuclear clusters act as binding units. The single-crystal X-ray diffraction (XRD) study (CIF file CCDC no. 2112467) reveals that the nitrate anions as well as the S and N atoms of the thiacalixarene ligand participate in the stabilization of the formed clusters. The obtained result demonstrates a unique ability of the polydentate ligands based on thiacalix[4]arene to form polynuclear coordination polymers of high dimensionality with silver cations, which can be used for the production of new functional materials.

Keywords: thiacalix[4]arene, clusters of silver cations, coordination polymers, pyridyl derivatives, crystal engineering, molecular structure, crystal structure, X-ray diffraction

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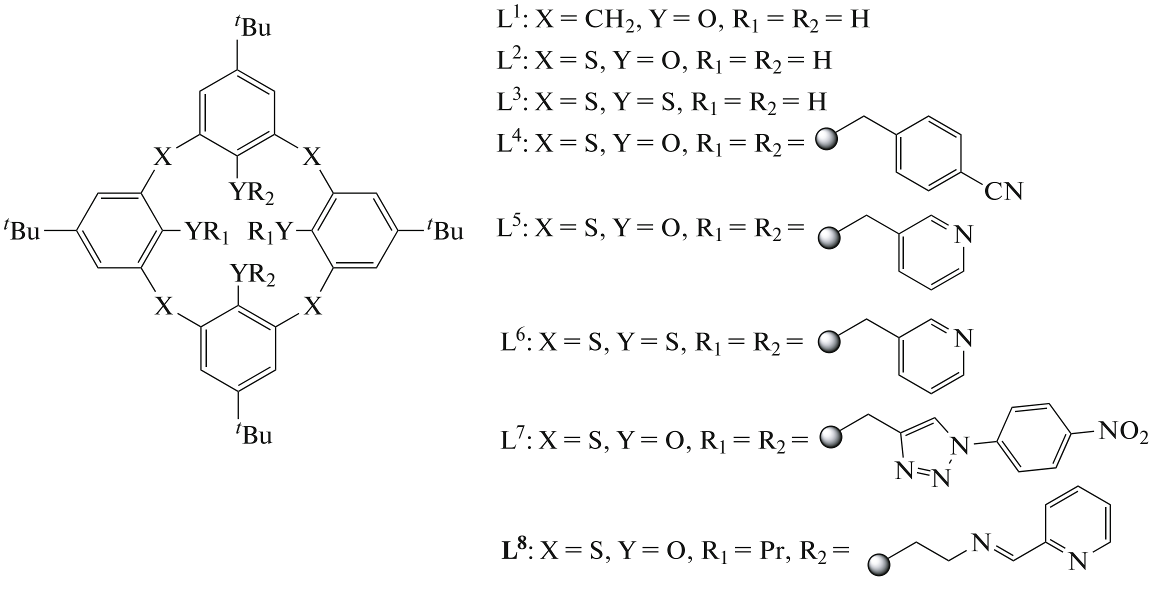
INTRODUCTION

As known, design of molecules acting as building blocks is among the key factors affecting the structure of materials prepared according to the “bottom-up” principle and their properties. *para-tert*-Butylcalix[4]arene (L^1) [1] and its structural analogs, such as *p-tert*-butyl thiacalix[4]arene (L^2) [2] and *p-tert*-butyltetramercaptothiacalix[4]arene (L^3) [3] (Scheme 1), represent macrocyclic compounds that are promising platforms for manufacturing new molecular building blocks due to an easiness of preparation, functionalization, and possibility of existing in various stereoisomeric forms. A unique ability of this family of macrocycles bearing diverse coordinating N- and O-donor centers in the structure of substituents of the lower/upper rim to form extended 1D–3D structures of coordination polymers in the crystalline phase fixed in the 1,3-alternate and cone stereoisomeric forms has previously been demonstrated [4]. It was also found that the dimension of the related coordination polymers can be controlled by the variation of the ligand shape and flexibility of the spacer and bulky substituents [5–8]. A significant role played by additional soft binding centers, as a rule, S-ether, phosphine, and pyridyl groups in the ligands, should be mentioned. These groups involved in the coordination interaction with soft metal cations, such as Hg^{2+}, Ag^+, Au^+, and Cu^+, can lead to the formation of functional metal clusters, including both discrete complexes [9–14] and coordination polymers [15–17], which can be used for manufacturing new materials with tunable optical and catalytic properties [18, 19]. The structural formulas of the following ligands are shown in Scheme 1: *p-tert*-butylcalix[4]arene (L^1), *p-tert*-butylthiacalix [4]arene (L^2), *p-tert*-butyltetramercaptothiacalix[4]arene (L^3), tetra-(4-cyano)benzyl (L^4) and tetrapyridyl (β-isomer) (L^5) derivatives, tetrapyridyl derivative (β-isomer) of *p-tert*-butyltetramercaptothiacalix[4]arene (L^6), and tetra-(4-nitro)phenylenetriazolyl (L^7) and aminopyridyl (α-isomer) (L^8) derivatives of *p-tert*-butylthiacalix[4]arene.



Scheme 1.

The 3D coordination polymer with silver(I) nitrate was prepared from the tetracyanobenzyl derivative of thiacalix[4]arene L^4 [20] in which the sulfur atoms of the macrocyclic platform as well as the nitro groups and nitrate anions participated in the formation of decanuclear clusters of silver cations with the ordered (due to the crystal packing) arrangement with a distance between the cluster centers of 25.77 Å.

The discrete polynuclear clusters [Ag_{34}] [21, 22] and [Ag_{88}] [23] based on unsubstituted thiacalix[4]arene L^2 are formed in the crystalline phase and demonstrate a surprising affinity of macrocycles of this type to silver(I) cations.

A new 3D coordination polymer $[C_{64}H_{68}N_4O_4S_4(AgNO_3)_3]_\infty$ (**I**) was synthesized in this work using the tetrapyridyl derivative of thiacalix[4]arene (L^5) in the 1,3-alternate configuration as the organic ligand and silver(I) cations as hexanuclear clusters, and its structure was studied.

EXPERIMENTAL

The starting tetrapyridyl derivative of thiacalix[4]arene (compound L^1) in the target steric configuration 1,3-alternate was synthesized using a known procedure [5]. The solvents used in the synthesis of target ligand L^5 and related coordination polymer **I** (dichloromethane, acetone, and methanol) were prepurified according to published procedures [24].

Elemental analysis was carried out on a EuroEA3028-HT-OM CHNS analyzer (Eurovector SpA, Italy).

Single crystals of coordination polymer **I** suitable for XRD were obtained by the slow diffusion of a methanol solution (1 mL) of silver(I) nitrate (0.0024 g, 0.014 mmol) into a solution of compound L^1 (0.005 g, 0.0046 mmol) in dichloromethane (1 mL) at room temperature in a crystallization tube 4 mm in diameter protected from light. After 20 days, colorless crystals of compound **I** were filtered off and washed with methanol (1 mL). The yield of **I** was 0.0045 g (62%).

For $[C_{64}H_{68}N_4O_4S_4(AgNO_3)_3]_\infty$ (**I**)

Anal. calcd., %	C, 48.19	H, 4.30	N, 6.15
Found, %	C, 49.25	H, 4.46	N, 6.50

XRD of compound **I** was carried out on a Bruker SMART APEX II diffractometer equipped with a two-dimensional CCD detector (graphite monochromator, $\lambda(\mathrm{Mo}K_\alpha) = 0.71073$ Å, ω and φ scan modes with an increment of 0.5°). The single crystals of a suitable size were glued on a glass filament in a random orientation. Preliminary unit cell parameters were determined using three runs (different positions for the φ angle) with 12 frames in each run (ω scan mode). Data were collected and edited and unit cell parameters were determined and refined using the APEX3 program package (v2018.7-2, Bruker AXS). An absorption correction and systematic errors were applied using the SADABS-2016/2 program. The structure was solved by a direct method using the SHELXT-2018/2 program and refined by full-matrix least squares for F^2 using the SHELXL-2018/3 program [25]. Non-hydrogen atoms were refined in the anisotropic approximation. The hydrogen atoms at the carbon atoms were placed in the calculated positions and included into refinement by the riding model. Strong peaks of the residual electron density appeared in the final refinement cycles. Attempts to specify them as solvate molecules were unsuccessful. It can be assumed that the crystal contains a dichloromethane

Table 1. Crystallographic data and structure refinement parameters for compound **I**

Parameter	Value
Empirical formula	$C_{64}H_{68}N_7O_{13}S_4Ag_3$
FW	1595.10
Temperature, K	150(2)
Crystal system	Triclinic
Space group	$P\bar{1}$
a, Å	12.9851(16)
b, Å	15.791(2)
c, Å	20.006(4)
α, deg	105.573(2)
β, deg	92.197(2)
γ, deg	113.320(1)
V, Å^3	3580(1)
Z and Z'	2 and 1
ρ_{calc}, g cm^{-3}	1.480
μ, mm^{-1}	0.991
F(000)	1620
Scan range over θ, deg	1.9–26.0
Index range	$-16 \le h \le 16$, $-19 \le k \le 19$, $-24 \le l \le 24$
Total number of reflections	27374
Number of independent reflections	13868
R_{int}	0.028
Data completeness over θ = 25.242°, %	99.0
T_{max}/T_{min}	0.7457/0.6526
Number of observed reflections ($I > 2\sigma(I)$)	9555
Number of reflections/number of restraints/number of parameters	13868/78/786
Goodness-of-fit	1.06
R ($I > 2\sigma(I)$)	$R_1 = 0.0580$, $wR_2 = 0.1445$
R (for all reflections)	$R_1 = 0.0835$, $wR_2 = 0.1697$
Residual electron density (max/min), e Å^{-3}	2.00/–1.22

molecule rotationally disordered over more than three positions. In addition, solvent molecules can also be in the crystal. It was decided to refine the structure with an undetermined solvate using the SQUEEZE procedure of the PLATON program [26]. A free volume of 387.2 Å^3 that can contain solvate molecules with 67 electrons (which is close to the assumed content of two dichloromethane molecules per unit cell) was found in the crystal. In addition, it was determined that two *tert*-butyl substituents were disordered over two positions by the shape of anisotropic shift ellipsoids, peaks of the residual electron density, and geometric parameters. The disordered fragments were refined in the anisotropic approximation with the refinement of populations of the positions. Structure refinement was performed using the OLEX program package [27]. The most important crystallographic characteristics of complex **I** are listed in Table 1. Since the solvent was not determined, the calculated density, linear absorption coefficient, and F(000) do not correspond to the real values.

The crystallographic data for the structure of compound **I** were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 2112467); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk.

The single-phase character of the obtained sample of compound **I** was studied by powder XRD. However, the crystals of the synthesized complex were found, unfortunately, to decompose rapidly in air at room temperature with the transition to the amorphous state, which is presumably related to the loss of volatile solvate molecules after separation from the mother liquor. Therefore, all attempts to obtain a high-quality powder XRD pattern were unsuccessful.

RESULTS AND DISCUSSION

The reaction of argenophilic compound L^5 fixed in the 1,3-alternate configuration with a threefold excess of silver(I) nitrate under the slow liquid diffusion conditions afforded single crystals, the XRD study of which showed the formation of a complex based on thiacalixarene L^5 with three formula units of silver nitrate (Table 1) that crystallizes in the centrosymmetric triclinic space group of the $P\overline{1}$ symmetry and represents 3D coordination polymer **I** in the crystalline phase.

In the structure of the coordination polymer, the thiacalix[4]arene molecules are nonsymmetrically bonded to three crystallographically nonequivalent silver(I) ions (Fig. 1a). The Ag(1) and Ag(2) atoms interact with different sulfur atoms (S(1) and S(2)) of the macrocyclic platform, whereas Ag(3) is coordinated by the N(3) nitrogen atom belonging to one of the four pyridyl substituents. The silver(I) ions are linked with each other via the bridging nitrate anions to form an S-like hexanuclear cluster $[Ag_6]$ with the Ag(1)–Ag(3) (4.977(1) Å) and Ag(2)–Ag(3) (4.924(1) and 5.380(1) Å) distances in the structure of the coordination polymer (Fig. 1b).

The Ag(1) atom is linked with the S(1) atom (2.626(2) Å) by two oxygen atoms of one nitrate anion (Ag(1)–O(71) 2.644(7) and Ag(1)–O(73) 2.559(6) Å) with approximately equal distances, two oxygen atoms of the symmetry-dependent nitrate anion Ag(1)–O(61)' 2.378(6) and Ag(1)–O(62)' 2.859(6) Å (symmetry procedure $1 + x, y, z$, translation along the x axis) with appreciably different distances, and the nitrogen atom of the adjacent molecule (Ag(1)–N(1)" 2.257(4) Å (symmetry procedure $2 - x, 2 - y, 1 - z$, molecule inverted by the symmetry center). The Ag(2) atom interacts with the S(2) atom (2.690(2) Å), oxygen atoms of three nitrate anions (Ag(2)–O(51) 2.448(4), Ag(2)–O(52) 2.628(4), and Ag(2)–O(62) 2.542(6) Å), and symmetry-dependent anion (Ag(2)–O(72)" 2.659(7) and Ag(2)–O(73)" 2.490(6) Å) (symmetry procedure $2 - x, 2 - y, 1 - z$, symmetry center). The Ag(3) atom coordinates with three nitrogen atoms of three adjacent calixarene molecules (Ag(3)–N(3) 2.320(5), Ag(3)–N(4)^3 Å (symmetry procedure $-1 + x, -1 + y, z$), 2.261(5) Å and Ag(3)–N(2)^5 (symmetry procedure $1 - x, 1 - y, -z$, symmetry center) 2.399(4) Å) and with two oxygen atoms of the symmetry-dependent nitrate anion (Ag(3)–O(51)^4 (symmetry procedure $x, -1 + y, z$, translation along the y axis) 2.604(5) Å and Ag(3)–O(53)^4 2.723(6) Å). Thus, the Ag–O distances in the structure of the synthesized coordination polymer were found to vary in wide ranges due to the formation of numerous “intermolecular” bonds of the silver atoms with the oxygen atoms of the nitrate anions and nitrogen atoms of the pyridine substituents of the calixarene platform.

At the same time, ligand L^5 exists in the conformation in which all the four nitrogen atoms enter into the *exo*-coordination with the metal atoms orienting outside relative to the macrocyclic cavity of the molecule. It should be mentioned that the nitrogen atoms from two proximal pyridyl fragments of one molecule coordinate to Ag(1) and Ag(2) belonging to the adjacent $[Ag_6]$ clusters with the formation of a binary bidentate chelate involving the sulfur atoms of the macrocycle platform, which leads to the formation of 1D chains oriented along the crystallographic 0x axis (Fig. 2). The chains are linked with each other in the crystal due to the involvement of the nitrogen atoms from the remained pyridyl groups of thiacalixarene in the coordination with the Ag(3) cations to form the final 3D structure of the coordination polymer (Fig. 3). As a result, in the observed structural motif of the synthesized coordination compound each $[Ag_6]$ cluster is bound to eight adjacent thiacalixarene molecules L^5, whereas each molecule of the organic ligand interacts with four surrounding $[Ag_6]$ species, which leads to stoichiometry $[Ag_6]/L^5 = 1/2$.

The crystal of compound **I** exhibits a free volume of 361 Å^3 per unit cell equivalent to 10% of the crystal volume, which is due to the pores along the 0x axis filled with disordered solvate molecules (presumably two dichloromethane molecules).

An analysis of the Cambridge Crystallographic Database showed four structures of the complexes of the thiacalixarene derivatives (L^4, L^6, and L^7) with silver(I) nitrate. The formation of 1D and 3D coordination polymers is observed in two of these structures (LIQTOW [28] and POXNEW [20]), whereas one of them contains the dimeric complex (UPEFAZ [29]) and the single structure of the compound (SEVJEJ [30]) in which resembling *tert*-butylthiacalix[4]arene L^8 bearing two iminopyridyl fragments formed a discrete complex with two formula units of silver nitrate. In all cases, the macrocycle exists in the 1,3-alternate conformation.

Stoichiometry $Ag/L^6 = 2/1$ is observed in the crystal of LIQTOW. It should be mentioned that the silver(I) ions have the trigonal coordination linearly binding the adjacent thiacalix[4]arene molecules due to the interactions with the nitrogen atoms of the pyridyl groups and oxygen atom of the nitrate anion, which results in the formation of the 1D coordination polymer. If taking into account rather weaker Ag(1)–O(2) contacts (2.898(3) Å) between the silver(I) atoms and oxygens of the nitrate anions from the adjacent 1D

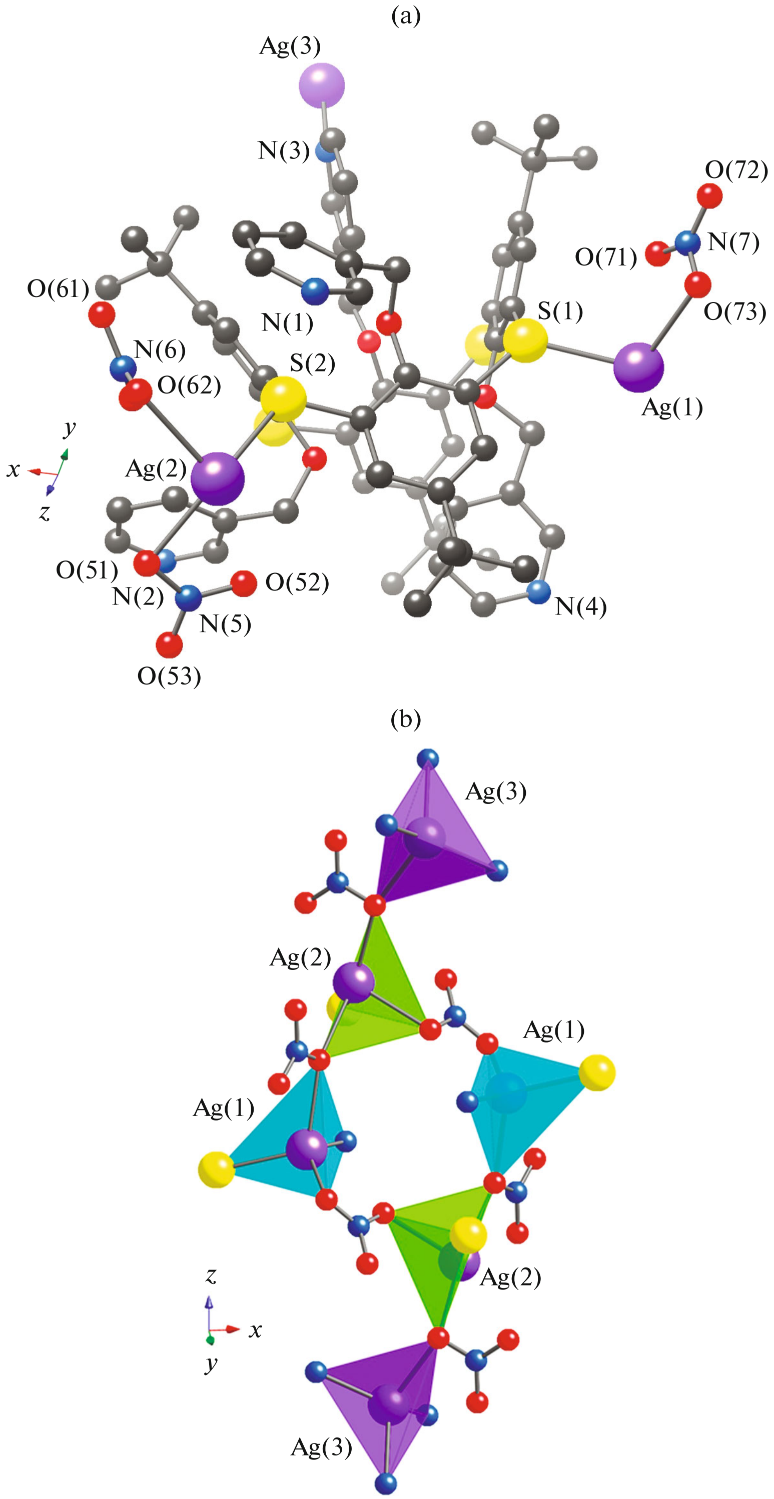


Fig. 1. (a) Geometry of the independent moiety of the crystal of compound **I** and (b) S-like [Ag_6] cluster demonstrating the coordination environment of silver(I) cations in the structure of coordination polymer **I**. In Figs. 1–3, hydrogen atoms are omitted and the main positions are shown for the disordered *tert*-butyl substituents.

chains, then the formation of dimeric [Ag_2] clusters ($d_{Ag–Ag}$ = 4.4389(4) Å) can formally be distinguished (Fig. 4a). In this case, the 1D structure of the coordination polymer can be considered as 3D.

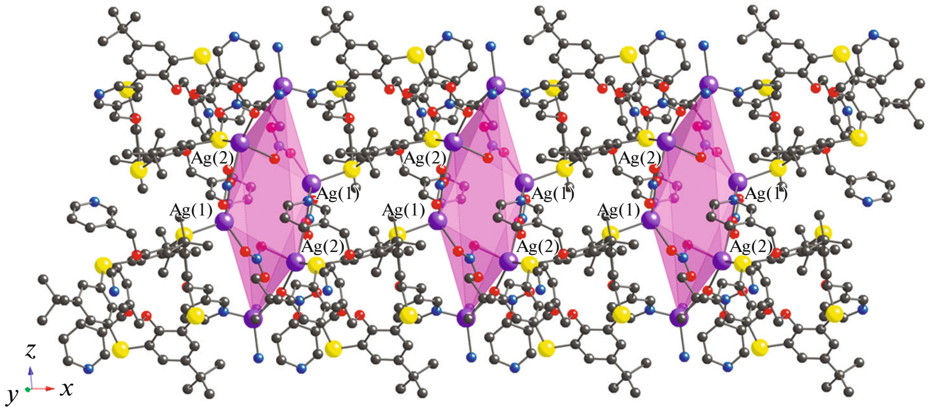


Fig. 2. Fragment of the crystal packing of compound **I** showing the formation of the 1D chain along the 0*x* axis in which the $[Ag_6]$ clusters are consecutively bonded to each other due to the calixarene molecules acting as bis(dentate) ligands.

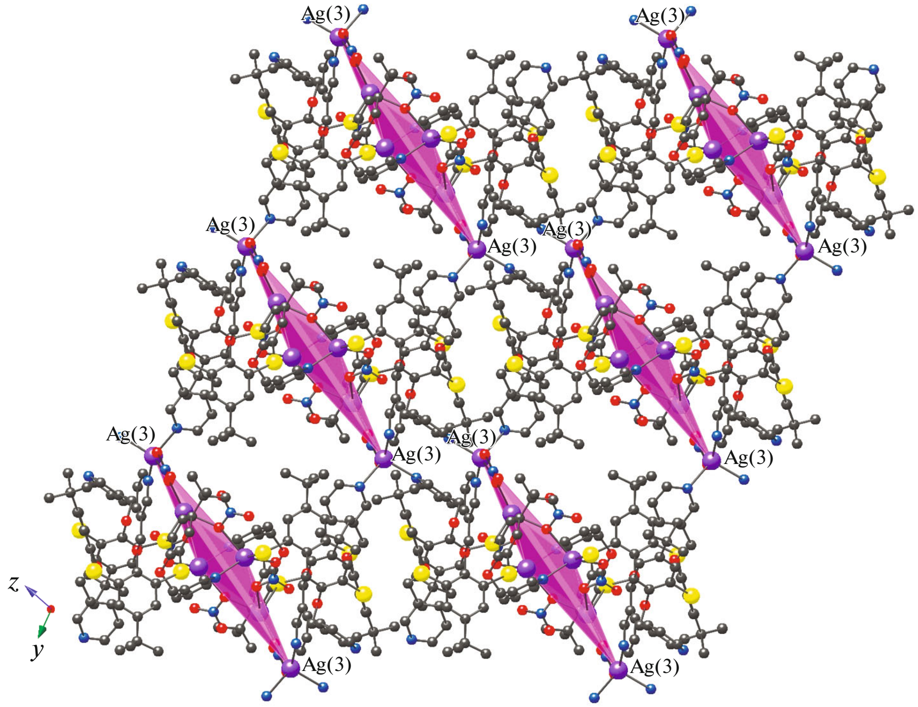


Fig. 3. Crystal packing of the hexanuclear silver clusters in compound **I**.

In the crystal of POXNEW, a 3D coordination polymer is formed due to the alternation of calixarene molecules L^4 and four-charge polynuclear complex cations $Ag_{10}(NO_3)_6^{4+}$ in which the Ag–O bonds range from 2.40(6) to 2.82(8) Å and the Ag(1)–Ag(3) and Ag(2)–Ag(3) distances are 3.85 and 5.38 Å, respectively (Fig. 4b).

In the crystal of UPEFAZ (Fig. 4c), a discrete complex is formed due to binding two binuclear clusters of silver(I) cations in which the metal atoms exist in the distorted tetrahedral (Ag(2)) and octahedral (Ag(3)) coordination environments and thiacalix[4]arene molecule L^7 with four triazolyl substituents exists in the cone steric configuration. One of two coordinated nitrate anions along with the sulfur atom of the calixarene platform plays the role of a binding bridging species. In the case of Ag(3), the nitrate anions act as bidentate ligands in which the Ag–O distances range from 2.45(1) to 2.726(8) Å. At the same time, the distance observed between one of the oxygens (O(14)) of the bridging nitrate anion with the Ag(2) atom is 3.128(8) Å, which is rather high for the Ag–O coordination bond to consider the nitrate anion to be a bidentate ligand. In the structure of the cluster,

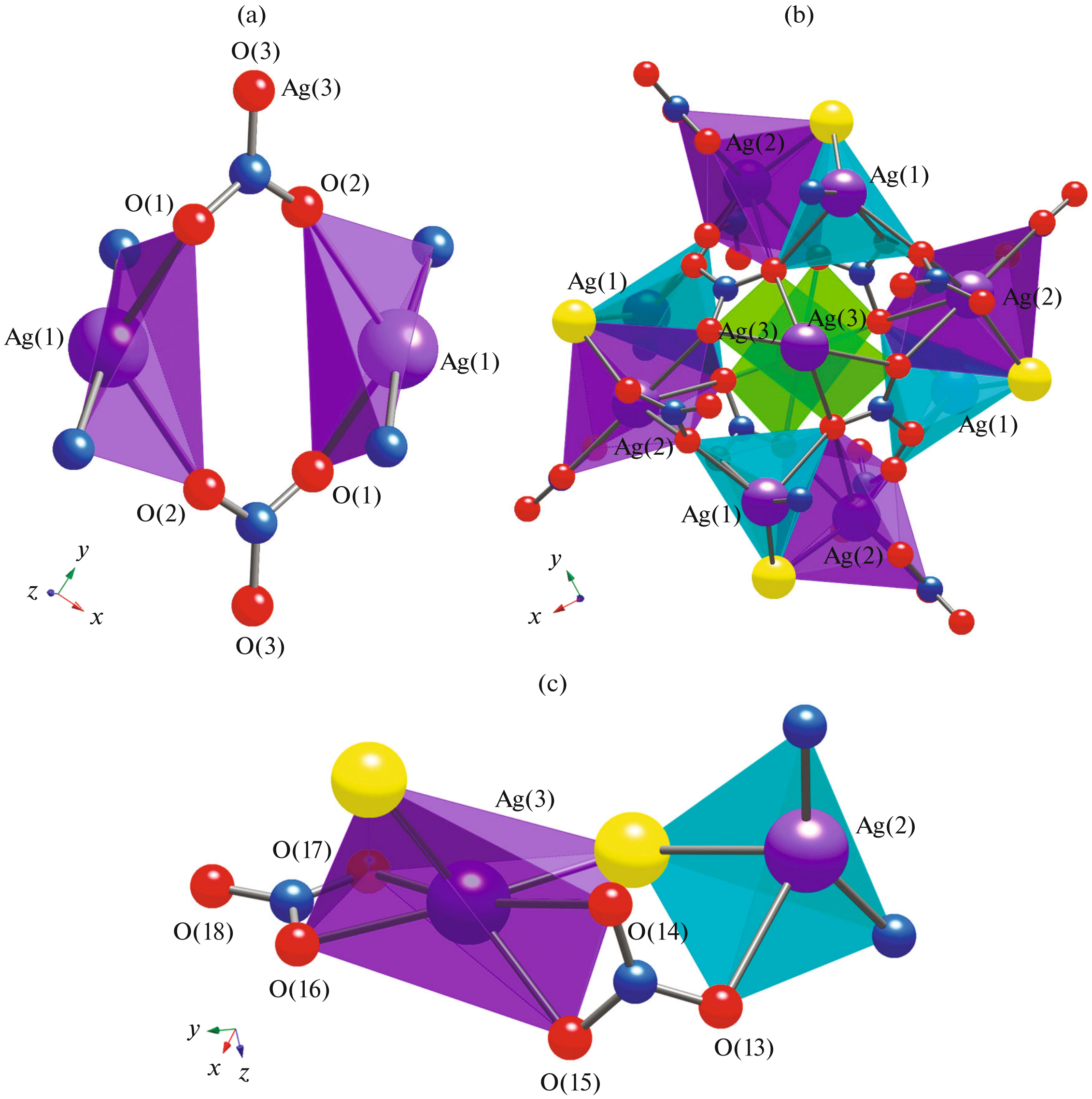


Fig. 4. Structures of the known silver clusters in the coordination compounds based on thiacalix[4]arenes tetrasubstituted at the lower rim: (a) LIQTOW [28] for L^6, (b) POXNEW [20] for L^4, and (c) UPEFAZ [29] for L^7.

the silver atoms are remote from each other at a distance of 4.716(1) Å.

Compound SEVJEJ formed by macrocycle L^8 is closest to compound **I** from the viewpoint of the nature of the coordinating group and, hence, we will compare the geometric parameters of these two molecules. The silver atoms in complex SEVJEJ are coordinated by the sulfur atom, two oxygen atoms of the nitrate anion, and two nitrogen atoms of the pyridineamino group. Unlike complex **I**, no intermolecular coordination bonds are formed in this complex, which is a discrete associate rather than coordination polymer. We would further compare the crystal structures of these compounds using averaged geometric parameters of the symmetry-dependent fragments.

The bond lengths and bond angles of the calixarene platforms in the structures of compounds **I** and SEVJEJ are equal within experimental inaccuracies and coincide with those observed in *tert*-butylthiocalixarene molecules. Therefore, they will not discussed further.

The Ag–S bonds in the crystal of compound **I** (2.626(2) and 2.690(2) Å) are much longer than those in complex SEVJEJ (average 2.494(2) Å), whereas the Ag–N bonds (from 2.257(4) to 2.399(4) Å) are shorter, on the contrary, than those in complex SEVJEJ (2.350(6) and 2.358(6) Å). The Ag–O bond lengths in the crystal of compound **I** vary in very broad ranges (from 2.378(6) to 2.859(6) Å), and the bonds in complex SEVJEJ also range from 2.349(6) to 2.617(9) Å. Two Ag–O bonds of one anion differ substantially in almost all complexes of monovalent silver with nitrate anions. Many authors do not interpret the bond in the structure if the Ag–O distance exceeds 2.6 Å; i.e., the formally bidentate NO_3 ligand is considered as monodentate. When this approach is used, the silver atoms in the complexes have an insignificantly distorted tetrahedral coordination, which was demonstrated for compound **I**.

Thus, new 3D coordination polymer **I** was synthesized, and its structure was studied in the crystalline phase by single-crystal XRD. It was shown that ligand L^5 together with nitrate anions can stabilize the formation of hexanuclear silver(I) clusters in the coordination polymer due to the involvement of the "soft" (according to Pearson) sulfur atoms of the thiacalix[4]arene platform in the coordination process. The obtained result indicates a high coordination ability of ligand L^5, which can further be used for the preparation of new coordination polymers containing macroclusters. Now we continue to study the coordination properties of ligand L^5 toward other "soft" and "hard" metal cations in the presence of various coordinating anions.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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