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Dedicated to I.L. Eremenko on the occasion of his 70th birthday

New Gold(I) Complexes with 1,5-Diaza-3,7-Diphosphacyclooctanes: Synthesis and Structures

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Abstract—New bi- and trinuclear gold(I) complexes are synthesized by the reactions of gold(I) chloride or tetrahydrothiophenegold(I) chloride with 1,5-di(*p*-tolyl)-3,7-diphenyl-1,5,3,7-diazadiphosphacyclooctane taken in various ratios. The structures of the synthesized compounds are confirmed by NMR spectroscopy, elemental analysis, and X-ray diffraction analysis (CIF files CCDC nos. 1974032 (**I**) and 1974031 (**II**)). Unlike P-pyridyl-containing 1,5-diaza-3,7-diphosphacyclooctanes, P-phenyl-substituted ligands undergo no rearrangements with ring expansion during the formation of the trinuclear gold(I) complexes.

Keywords: complex, aminomethylphosphine, 1,5-diaza-3,7-diphosphacyclooctane, gold(I), X-ray diffraction analysis

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INTRODUCTION

The metal complexes of gold(I) with the organic ligands capable of manifesting aurophilicity (weak interactions characteristic of *d*-metal cations) are unique objects of investigation [1–5]. The polyphosphine ligands in the gold(I) complexes are structure forming. At the same time, the flexible ligands capable of tuning the structure for the formed core of the complex are used in complex formation [6–11]. The lability of the ligands in the gold(I) complexes is used for the construction of systems with a luminescence response to the presence of metal cations, organic solvents, and other substrates [6, 12]. Polyphosphine ligands with a rigid ring, having a restricted mobility, and specifying the geometry of the complex core are used much more rarely [13–15]. Medium-cycle aminomethylphosphines in which two phosphorus atoms are bound in one sufficiently rigid cyclic system can be assigned to this class of ligands. Nevertheless, it has recently been demonstrated that the rigid ring of these eight-membered heterocyclic ligands can be transformed, under complex formation conditions, to give larger heterocycles. In particular, the possibility of the rearrangement of 1,5-di(*p*-tolyl)-3,7-dipyridin-2-yl-1,5-diaza-3,7-diphosphacyclooctane on the trinuclear gold(I) template to the complex with the 16-membered ligand was shown [16].

The recently synthesized in our laboratory the gold(I) complexes based on P-pyridyl-substituted 1,5-diaza-3,7-diphosphacyclooctanes exhibit the luminescence response to the solvent molecules in both the solid phase and solutions [13, 14, 17, 18]. This phenomenon for the 1,5-diaza-3,7-diphosphacyclooctane complexes is caused by several factors, the main of which is the formation of supramolecular systems of the host–guest type. In the systems indicated above, the pyridyl fragments play the key role in the formation of frontier molecular orbitals involved in the charge transfer processes inducing the emission properties.

New gold(I) complexes with 1,5-di(*p*-tolyl)-3,7-diphenyl-1,5-diaza-3,7-diphosphacyclooctane (L), whose structures contain no chromophoric pyridyl fragment, were synthesized in this work, and their structural parameters and spectral properties were studied.

EXPERIMENTAL

All reactions and manipulations were carried out under dry argon with standard vacuum line techniques. Solvents were purified, dried, deoxygenated, and distilled before use. Ligand L was synthesized according to a described procedure [19], and Au(Tht)Cl and AuCl were commercially available.

All NMR experiments were conducted at 303 K. ^{1}H NMR (400 MHz) and ^{31}P NMR (162 MHz) spectra were recorded on a Bruker Avance-DRX 400 spectrometer. Chemical shifts are given in parts per million relative to $SiMe_4$ (1H, internal solvent), and 85% H_3PO_4 (^{31}P, external). J values are given in Hz.

Synthesis of dichloro[1,5-di(*p*-tolyl)-3,7-diphenyl-1,5-diaza-3,7-diphosphacyclooctane]digold(I) (I). Tetrahydrothiophenegold(I) chloride (0.134 g, 0.42 mmol) in CH_2Cl_2 (3 mL) was added to a solution of L (0.100 g, 0.21 mmol) in CH_2Cl_2 (2 mL), and the mixture was stirred for 3 h. The formed precipitate was filtered off, washed with CH_2Cl_2, and dried in vacuo. The yield of complex **I** was 0.17 g (92%).

The crystals of complex **I** suitable for X-ray diffraction analysis were grown by the slow evaporation of the solvent from a solution of complex **I** in CH_3CN. The crystals of complex **I** ($C_{30}H_{32}N_2P_2Cl_2Au_2$ + uncertain solvent, *FW* = 947.36) are colorless prismatic, and monoclinic.

^{31}P NMR (CD_3CN), δ, ppm: 1.87. ^{1}H NMR (CD_3CN), δ, ppm: 2.26 (s, 6H, CH_3), 4.66 (m, br, 2H, P–CH_2–N), 4.69 (m, br, 2H, P–CH_2–N), 4.86 (dd, $^2J_{HH}$ = 4.58 Hz, $^2J_{PH}$ = 15.6 Hz, 4H, P–CH_2–N), 6.98 (d, $^2J_{HH}$ = 8.2 Hz, 4H, C_6H_4), 7.06 (d, $^2J_{HH}$ = 8.24 Hz, 4H, C_6H_4), 7.67–7.71 (m, br, 6H, C_6H_5), 7.96–8.00 (m, br, 4H, C_6H_5).

For $C_{30}H_{32}N_2P_2Cl_2Au_2$

Anal. calcd., %	C, 38.00	H, 3.40	N, 2.96	P, 6.56	Cl, 7.48	Au, 41.60
Found, %	C, 39.70	H, 4.20	N, 3.30	P, 6.30	Cl, 5.60	Au, 40.90

Synthesis of dichloro[1,5-di(*p*-tolyl)-3,7-diphenyl-1,5-diaza-3,7-diphosphacyclooctane]trigold(I) chloride (II). Gold chloride (0.072 g, 0.31 mmol) in CH_2Cl_2 (3 mL) was added to a solution of L (0.100 g, 0.21 mmol) in CH_2Cl_2 (2 mL), and the mixture was stirred for 3 h. The formed precipitate was filtered off and washed with CH_2Cl_2. The yield of complex **II** was 0.065 g (17%).

The crystals of complex **II** suitable for X-ray diffraction analysis were grown from the two-phase dimethylformamide (DMF)–CH_3CN (1 : 4) system. The crystals of compound **II** ($[C_{60}H_{63}N_4P_4Cl_2Au_3]^+$-$Cl^-$ + uncertain solvent, *FW* = 1661.29) are colorless, prismatic, and monoclinic.

^{31}P NMR (DMSO-d_6), δ, ppm: –1.2. ^{1}H NMR (DMSO-d_6), δ, ppm: 1.90 (s, br, 6H, CH_3), 2.07 (s, br, 3H, CH_3 (disguised by acetone)), 2.18 (s, br, 3H, CH_3), 4.65–4.68 (m, br, 4H, P–CH_2–N), 5.16–5.20 (m, br, 4H, P–CH_2–N), 6.82–7.08 (m, br, 8H, C_6H_4), 7.65–7.77 (m, br, 10H, C_6H_5).

For $C_{60}H_{64}N_4P_4Cl_3Au_3$

Anal. calcd., %	C, 43.35	H, 3.88	N, 3.37	P, 7.45	Cl, 6.40	Au, 35.55
Found, %	C, 42.80	H, 3.40	N, 4.02	P, 6.78	Cl, 6.24	Au, 36.76

X-ray diffraction analyses (XRD) of complexes **I** and **II** were carried out on a Bruker Smart APEX II CCD diffractometer at the XRD Department of the Federal Research Center (CSF-SAC) on the basis of the Laboratory of Diffraction Methods at the Arbuzov Institute of Organic and Physical Chemistry (Kazan Research Center, Russian Academy of Sciences). The cell parameters and experimental data were obtained at T = 296(2) K (MoK_α radiation, λ = 0.71073 Å, ω scan mode). An absorption correction was applied using the SADABS program [20]. The structures were solved by a direct method using the SHELXT 2014/4 program [21] and refined by full-matrix least squares for F^2 using the SHELXL-2018/3 program [22]. All calculations were performed using the WinGX-2014.1 [23] and APEX2 [24] program packages. Non-hydrogen atoms were refined in the anisotropic approximation. Numerous intense electron density peaks were revealed in both structures, but attempts to specify them as solvate molecules were unsuccessful. Attempts to specify solvate molecules of acetonitrile, dichloromethane, acetone, and DMF were made. In all cases, it was necessary to fix the geometry of solvate molecules and to refine the fractional population of positions of the molecules. Nevertheless, these attempts resulted in unreal thermal vibration parameters. A decrease in the symmetry of the structures (transition to noncentrosymmetric space groups) did not lead to the disappearance of disordering. It was decided to refine both structures according to the SQUEEZE procedure implemented in the PLATON program [25] in order to take into account an uncertain solvent. The hydrogen atoms at the carbon atoms of the molecular complexes were placed in the calculated positions and refined by the riding model. The crystallographic data and structure refinement results are presented in Table 1.

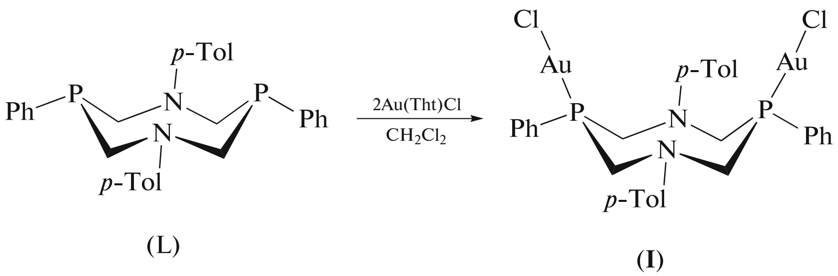
The coordinates of atoms, bond lengths, bond angles, and temperature parameters were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1974032 (**I**) and 1974031 (**II**); deposit@ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Binuclear complex **I** as a white powder is formed in a quantitative yield in the reaction of tetrahydrothiophenegold(I) chloride with 1,5-diaza-3,7-diphosphacyclooctane in a ratio of 1 : 2 (Scheme 1).

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

Parameter	Value	
	I	**II**
FW	947.36	1662.30
T, K	296	296
Space group	$C2/c$	$P2_1/n$
Z	4 (molecule in partial position on axis 2)	4
a, Å	23.04(3)	11.404(2)
b, Å	11.316(16)	26.382(5)
c, Å	16.01(2)	21.801(4)
β, deg	126.222(16)	97.160(2)
V, Å^3	3367(8)	6508(2)
ρ_{calc}, g cm^{-3}	1.869	1.697
μ, mm^{-1}	8.979	7.006
F(000)	1792	3200
$2\theta_{max}$, deg	57.2	58.0
Range of indices hkl	$-30 \le h \le 30$, $-15 \le k \le 15$, $-20 \le l \le 21$	$-14 \le h \le 15$, $-34 \le k \le 34$, $-29 \le l \le 29$
Number of measured/independent reflections	13572/4089	56618/16042
R_{int}	0.1255	0.1349
Reflections with $I > 2\sigma(I)$	1697	6474
Number of refined parameters	172	667
R_1/wR_2 ($I > 2\sigma(I)$)	0.0612/0.1340	0.0718/0.1677
R_1/wR_2 (for all reflections)	0.1572/0.1757	0.2093/0.2125
GOOF	0.852	0.995
Residual electron density (max/min), e/Å^3	−3.02/1.22	−2.01/2.19



Scheme 1.

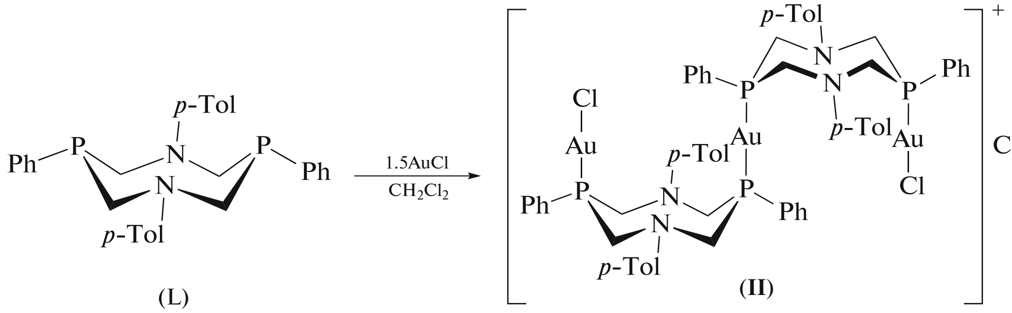
Complex **I** is stable in air in both the solid state and solution. The obtained compound is soluble in DMSO and DMF and restrictedly soluble in acetonitrile.

The ^{31}P NMR spectrum of compound **I** exhibits a narrow signal at 1.87 ppm. The ^{1}H NMR spectrum contains the well resolved set of signals for both the aromatic and heterocyclic moieties of the molecule similar to that observed for the binuclear gold(I) complex with 1,5-di(*p*-tolyl)-3,7-di(pyridyl-2-yl)-1,5,3,7-diazadiphosphacyclooctane [13]. The aromatic moiety of the *p*-tolyl fragment is presented by the AB system at 6.98 and 7.08 ppm with a spin–spin coupling constant of 8.2 Hz. The protons of the phenyl groups are detected as two multiplets at 7.7 and 7.97 ppm with the corresponding integral intensities. The protons of the heterocyclic fragment P–CH$_2$–N are detected at 4.68 and 4.88 ppm as one doublet of doublets with the spin–spin coupling constants $^2J_{\text{HH}}$ = 15.6 Hz and $^2J_{\text{PH}}$ = 4.58 Hz and one broadened doublet. The observed spectral pattern suggests that the heterocyclic moiety of the ligand exists in the C_2 symmetric conformation close to that of free ligands. The protons of the methyl groups of the *p*-tolyl fragment are detected as a narrow singlet at 2.26 ppm.

The structure of complex **I** was confirmed by the XRD method and is presented in Fig. 1a. The crystals of complex **I** suitable for the study were grown by the slow evaporation of a solution of the complex in acetonitrile. The ligand molecule in complex **I** adopts the "chair–chair" conformation with the equatorial orientation of the substituents at the phosphorus atoms and the axial orientation of the *p*-tolyl substituents at the nitrogen atoms, so that both gold-containing fragments are arranged at one side of the cycle. The intramolecular Au···Au distance is 5.834 Å. This structure of the molecule contains a small cavity formed by the P–Au–Cl fragments and the substituents at the nitrogen atoms arranged at one side of the cycle. Four axial protons of the P–CH$_2$–N fragment of the ligand molecule at another side of the heterocycle are preorganized for weak electrophilic interactions with the solvent molecule. Unfortunately, we failed to unambiguously determine the solvate molecule disordered by the symmetry axis. The localization of numerous intense peaks in the difference electron density series in the cavity of the complex indicates that the cavity contains solvate molecules (there were attempts to specify acetonitrile, dichloromethane, acetone, and DMF as solvate molecules). The structure was refined by the SQUEEZE procedure [25] in the final cycles (allowance for uncertain solvent).

An analysis of the crystal packing of compound **I** suggests that uncertain solvent molecules bind molecules of the complex into stacks along the symmetry axis of the crystal (along the *b* axis) via weak interactions according to the "head-to-tail" type. There are also interactions between the hydrogen atoms at the carbon atoms of the eight-membered heterocycle and solvate molecules at one side and the chlorine and gold atoms and *p*-tolyl substituents at another side (Fig. 2a). It can be assumed that the absence of strong directed intermolecular interactions (of the type of classical hydrogen bonds) in the crystal of complex **I** results in disordering of the solvate molecule in it.

The reaction of ligand L with gold(I) chloride in a ratio of 2 : 3 affords trinuclear complex **II** of the cationic type as a white powder in a quantitative yield (Scheme 2).



Scheme 2.

Unlike complex **I**, compound **II** is soluble only in DMSO and DMF. The NMR spectra of a solution of compound **II** in DMSO-d$_6$ exhibit signal broadening related to the possible dissociation and decomposition of the complex. The ^{31}P NMR spectrum contains a weak signal at 1.8 ppm. In the ^{1}H NMR spectrum of compound **II**, the protons of the phenyl fragment are detected as two groups of unresolved multiplets at 7.07 and 7.67 ppm, the protons of the *p*-tolyl fragment are detected as multiplets at 6.82–6.98 and 7.77–8.02 ppm partially overlapped with the signals of the protons of the phenyl fragment at the phosphorus atoms, and the protons of the heterocycle are detected as two doublets at 4.68–5.16 ppm and one strongly broadened multiplet in a range of 4.38 ppm. Thus, the nonequivalence of two moieties of the aminomethylphosphine cycle caused by different bindings of two endocyclic phosphorus atoms with the gold(I) cations is observed in complex **II** in a solution. The integral intensities and arrangement of all groups of signals are

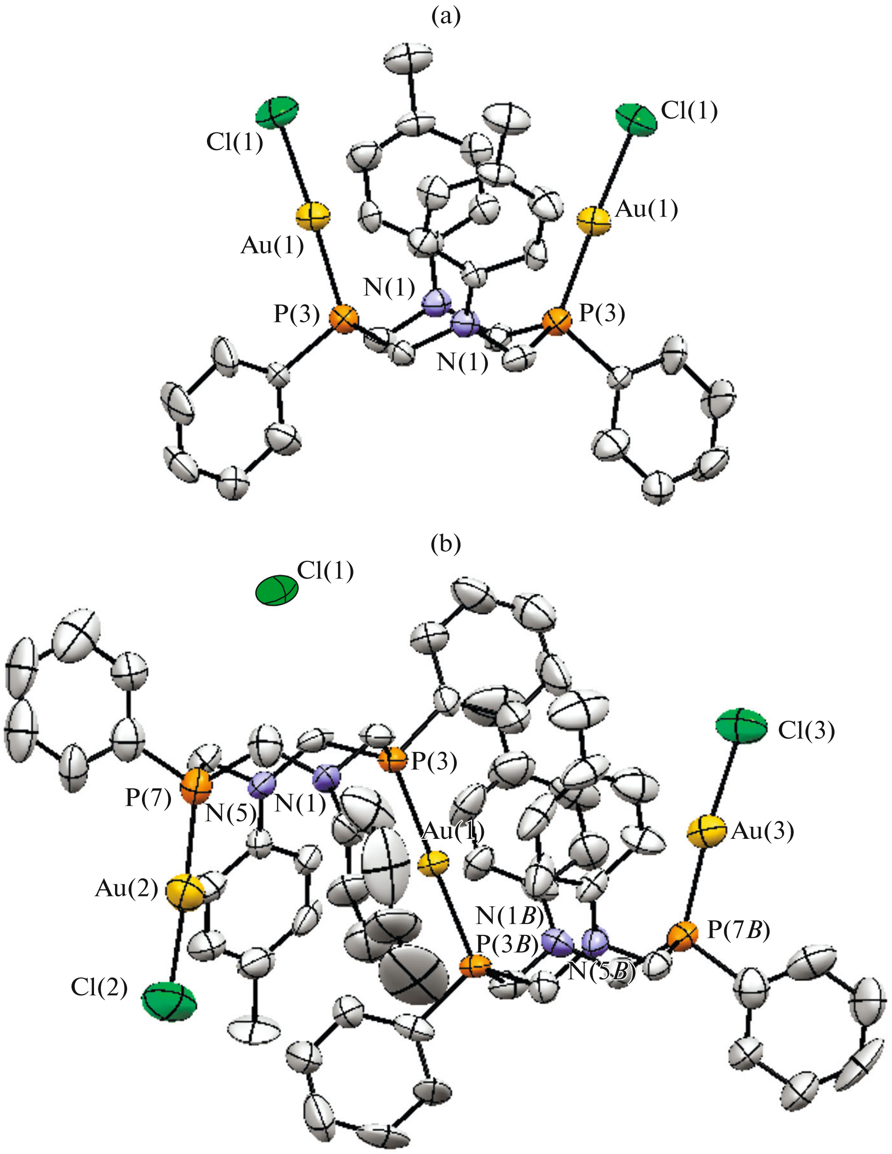


Fig. 1. Geometry of molecules of complexes (a) **I** and (b) **II** in crystal. Hydrogen atoms are omitted. Ellipsoids of 50% probability.

consistent with the assumed structure shown in Scheme 2.

The structure of complex **II** was finally determined by the XRD method. The crystals of complex **II** suitable for the study were grown from a two-phase DMF–acetonitrile mixture. Similarly to the structure of complex **I**, the type of solvent was not unambiguously determined: molecules of acetonitrile, DMF, dichloromethane, and acetone can be “collected” in the difference electron density series. For the refinement even with restraints imposed on bond lengths and thermal vibration parameters of atoms, these molecules with the fractional population give no reasonable geometry of the disordered fragments. Therefore, the structure was refined using the SQUEEZE procedure [25] (allowance for uncertain solvent).

In the crystal, complex **II** is the cation and the chloride anion is the counterion (Fig. 1b). In the trinuclear cation, one of the gold atoms is bridging between two heterocyclic ligands and has a linear coordination (bond angle P(3)Au(1)P(3*B*) 178.8(1)°). The gold atoms of the terminal groups also have an almost linear coordination (angles Cl(2)Au(2)P(7) 176.95(17)° and Cl(3)Au(3)P(7*B*) 174.18(15)°). The Au(1)–P(3) and Au(1)–P(3*B*) (2.303(3) and 2.293(3) Å) bond lengths are the same within experi-

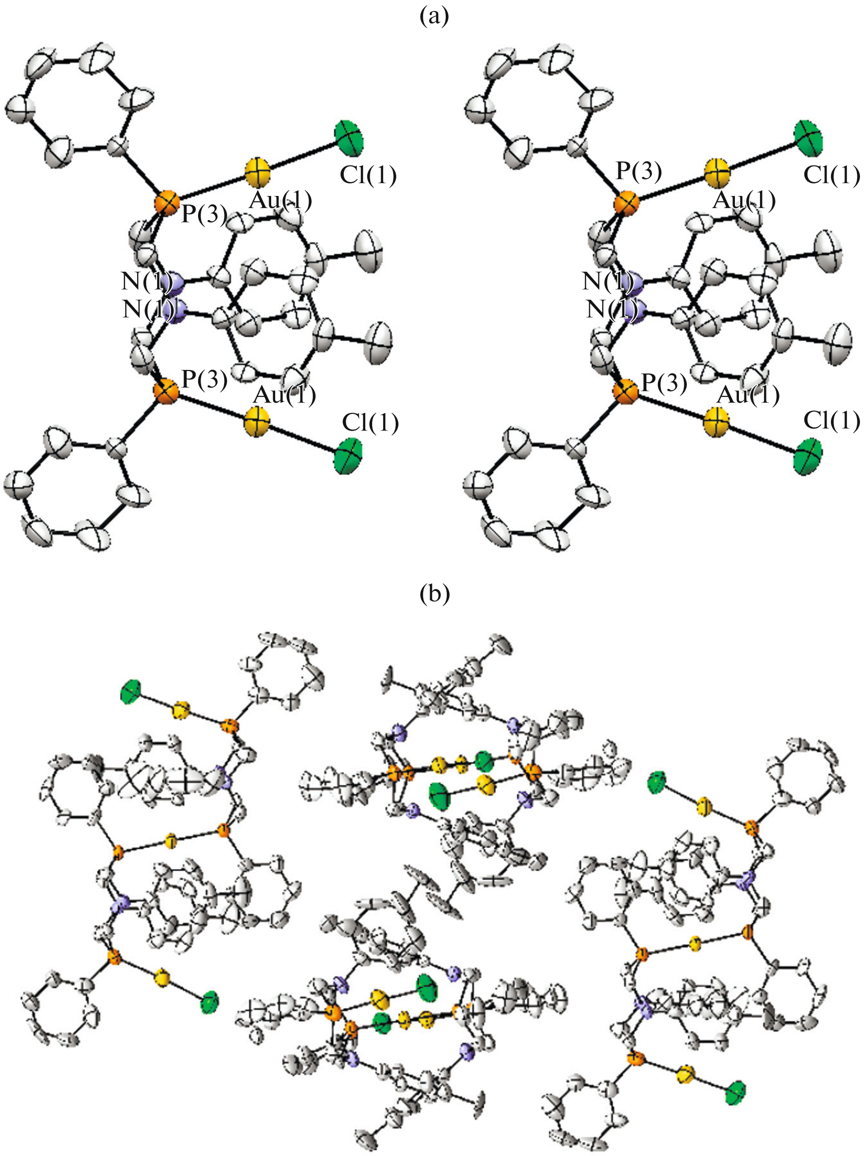


Fig. 2. Fragments of the crystal packing of compounds (a) **I** (cavities between adjacent molecules accessible for solvent molecules are shown) and (b) **II**.

mental inaccuracies (Au(2)–P(7) 2.223(4) and Au(3)–P(7*B*) 2.222(3) Å) and are slightly shorter than the lengths of the bonds of the bridging gold atom. The Au(2)–Cl(2) (2.284(5) Å) and Au(3)–Cl(3) (2.282(4) Å) bond lengths are also the same. The intramolecular distances Au(1)...Au(2) and Au(1)...Au(2) are 5.601 and 5.849 Å, respectively, which excludes any aurophilic interaction. Seemingly, the complex cation adopts the symmetric conformation and could exist in the partial position in the crystal. However, the general position of the cation in the crystal is explained by different conformations of the heterocyclic ligands. In spite of the fact that the conformations of the eight-membered heterocycles are the same ("chair–chair" as in the free ligand molecule), the phenyl substituents at the phosphorus atoms have the equatorial orientation, and the *p*-tolyl substituents at the nitrogen atoms have the axial orientation, and the ligands differ by the turn of the *p*-tolyl substituents. In ligand *B*, both nitrogen atoms adopt a nearly planar coordination of the bonds (the sum of the bond angles at the N(1*B*) and N(5*B*) atoms is 356.8(9)° and 357.1(9)°, respectively) and the planes of the aromatic rings at them are coplanar to the planes of the bonds of the nitrogen atoms (torsion angles about the N(1*B*)–C(9*B*)–N(5*B*)–C(22*B*) bonds 14(2)° and 3(2)°). In the first ligand, the N(5) atom has an almost planar coordination (the sum of bond angles at this atom is 357.7(9)°) and the N(1) atom has the trigonal pyramidal coordination (the sum of the bond angles at this atom is 348.6(9)°). The *p*-tolyl substituent at the N(5) atom lies in the plane of its bonds (torsion angle about the N(5)–C(22) bond 2(2)°), but the substituent at the N(1) atom lies in the bisector plane of the endocyclic bonds of the nitrogen atom (torsion angle about the N(1)–C(9) bond 75(2)°), which excludes the conjugation of the lone electron pair of the nitrogen atom with the aromatic ring. The absence of conjugation in the first ligand is manifested as some elongation of the N(1)–C(9) bond (1.420(16) Å) compared to the N(1*B*)–C(9*B*) bond (1.394(15) Å).

An analysis of the crystal packing of compound **II** shows that the chloride anion has short contacts with the hydrogen atoms at the carbon atoms of the eight-membered heterocycles of the adjacent molecules and uncertain solvate molecules are arranged in cavities between the Au(2) and Cl(2) atoms of the adjacent molecules (Fig. 2b) linked by the symmetry center.

It is noteworthy that the rearrangement accompanied by the expansion of the diazadiphosphacyclooctane ring to the 16-membered macrocycle occurs in the similar reaction of gold(I) chloride with P-pyridyl-substituted aminomethylphosphine [16] to form the isomeric complex. Probably, the presence of the pyridyl group causes the highest lability of the P–CH_2–N fragment of the ligand compared to the P-phenyl-substituted analogues.

Thus, new complexes of gold(I) chloride based on 1,5-diaza-3,7-diphosphacyclooctanes were synthesized, and their structures in the solution and solid phase were determined. It was shown that the so-called "open" conformation was formed in the crystal structure of the binuclear gold(I) complex. This conformation is related to the formation of the complex according to the host–guest complex accomplished due to the formation of weak interactions with the P–Au–Cl fragments, on the one hand, and P–CH_2–N fragments, on the other hand. The formation of the complex according to the host–guest type confirms the tendency of interactions of this type to occur in all similar complexes regardless of substituents at the phosphorus atoms. Unlike pyridyl-containing 1,5-diaza-3,7-diphosphacyclooctanes, P-phenyl-substituted ligands undergo no rearrangements with ring expansion during the formation of the trinuclear gold(I) complexes. The unusual bisector arrangement of the *p*-tolyl substituent in the complex inducing the escape from the conjugation of the lone electron pair of the nitrogen atom of the aminomethylphosphine cycle was found. The synthesized complexes exhibit no substantial emission in the solid phase and solution, which confirms the necessity to introduce chromophoric groups to the phosphorus atoms forming the frontier molecular orbitals involved in the charge transfer and inducing the emission properties for the design of luminescent complexes.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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