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Synthesis and Structure of Gold Complexes $[Ph_3PR][Au(CN)_2Cl_2]$ (R = $CH_2CH{=}CHCH_3$, CH_2CN) and $Ph_3PC(H)(CN)Au(CN)_2Cl$

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Abstract—The ionic complexes $[Ph_3PR][Au(CN)_2Cl_2]$ (R = $CH_2CH{=}CHCH_3$ (**I**), CH_2CN (**II**)) were prepared by the reactions of organyltriphenylphosphonium chlorides with potassium dichlorodicyanoaurate in water followed by recrystallization from acetonitrile. Apart from the major product **II**, the crystals of the molecular complex $Ph_3PC(H)(CN)Au(CN)_2Cl$ (**III**) were isolated. The products were characterized by X-ray diffraction (CIF files CCDC nos. 1957185 (**I**), 2060227 (**II**), 2066549 (**III**)) and NMR and IR spectroscopy. According to X-ray diffraction data, complexes **I** and **II** consisted of organyltriphenylphosphonium cations with a slightly distorted tetrahedral geometry of phosphorus atoms and the centrosymmetric square anions $[Au(CN)_2Cl_2]^-$, which, in the case of complex **II**, form coordination pseudopolymer chains via the Au···Cl interanion contacts (3.40 Å). In complex **III**, the phosphorus and gold atoms are also coordinated in the tetrahedral and square environments; the ylide carbon atom is located at the gold atom in the *trans*-position relative to chlorine.

Keywords: dichlorodicyanoaurate, organyltriphenylphosphonium, triphenylphosphanylideneacetonitrile, synthesis, structure, X-ray diffraction

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INTRODUCTION

In the development of new, potentially useful compounds, an important issue is the possibility for molecules and other structural units to be involved in noncovalent interactions, which play an important role in determining the physicochemical properties and biological activities of these compounds [1–6]. In addition, considerable attention of researchers is currently attracted by metal-organic coordination polymers (MOCPs) [7–12].

Among the abundant building blocks for MOCPs, an important position belongs to cyanide complexes, in particular, monovalent and trivalent gold compounds possessing properties such as luminescence [13–16], birefringence [17–19], vapochromism [20–22], negative thermal expansion coefficient [23, 24], and magnetism [13, 25–27]. The strategic choice of auxiliary ligands and counter-ions makes it possible to modify these properties.

In order to discover new cyanoaurate MOCPs and as a continuation of a series of studies devoted to the structures and properties of dihalodicyanoaurate complexes [28–33], we synthesized the complexes $[Ph_3PCH_2CH{=}CHCH_3][Au(CN)_2Cl_2]$ (**I**) and $[Ph_3PCH_2CN][Au(CN)_2Cl_2]$ (**II**) (as a mixture with the $Ph_3PC(H)(CN)Au(CN)_2Cl$ (**III**) by-product) and described characteristic features of their structure.

EXPERIMENTAL

The synthesis was carried out in air. Commercially available (but-2-enyl)triphenylphosphonium chloride (97%, Alfa Aesar), cyanomethyltriphenylphosphonium chloride (98%, Alfa Aesar), and acetonitrile (special purity grade, Kriochrom) were used as received. Potassium dichlorodicyanoaurate and triphenylphosphonium cyanomethylide were prepared by procedures described previously [17, 34].

Synthesis of (but-2-enyl)triphenylphosphonium dichlorodicyanoaurate (I). An aqueous solution of (but-2-enyl)triphenylphosphonium chloride (0.094 g, 0.28 mmol) was added with stirring to a solution of potassium dichlorodicyanoaurate (0.100 g, 0.28 mmol) in water (10 mL). The yellow precipitate that formed was collected on a filter, washed with water, and dried. Recrystallization from acetonitrile gave light yellow crystals. The yield of complex **I** was 0.160 g (90%). T_m = 146°C.

IR (ν, cm^{-1}): 3065, 3053, 3030, 2990, 2945, 2905, 2851, 2216, 1638, 1616, 1585, 1485, 1435, 1398, 1377, 1339, 1315, 1180, 1159, 1111, 1055, 997, 970, 922, 839, 795, 746, 737, 723, 689, 615, 540, 503, 486, 451, 426.

For $C_{24}H_{22}N_2PCl_2Au$

Anal. calcd., %	C, 45.23	H, 3.49
Found, %	C, 45.05	H, 3.57

Compound **II**, cyanomethyltriphenylphosphonium dichlorodicyanoaurate, was synthesized by a procedure similar to that used for **I**. The product was formed as light yellow crystals in 71% yield. T_m = 121°C.

IR (ν, cm^{-1}): 3061, 3024, 2930, 2866, 2766, 2259, 1587, 1483, 1441, 1393, 1319, 1252, 1186, 1113, 995, 843, 756, 743, 723, 689, 550, 503, 453, 428.

For $C_{44}H_{34}N_6P_2Cl_4Au_2$

Anal. calcd., %	C, 42.46	H, 2.76
Found, %	C, 42.34	H, 2.79

Molecular complex **III** was formed as a by-product (6%) upon the synthesis of complex **II** and was prepared by an alternative route according to the procedure given below.

Synthesis of (cyanomethyltriphenylphosphonium)-chlorodicyanogold (III). Triphenylphosphonium cyanomethylide (0.084 g, 0.28 mmol) was added with stirring to a solution of potassium dichlorodicyanoaurate (0.100 g, 0.28 mmol) in acetonitrile (10 mL). After evaporation of the solvent, colorless crystals were isolated from the filtrate. The yield of **III** was 0.134 g (82%). T_m = 112°C.

IR (ν, cm^{-1}): 3059, 3024, 2930, 2866, 2766, 2257, 2156, 2143, 1587, 1483, 1439, 1395, 1339, 1317, 1250, 1186, 1113, 997, 843, 756, 743, 723, 689, 547, 500, 428.

For $C_{22}H_{16}N_3PClAu$

Anal. calcd., %	C, 45.11	H, 2.76
Found, %	C, 45.09	H, 2.81

The IR spectra of compounds **I–III** were measured on a Shimadzu IRAffinity-1S FTIR spectrometer; the samples were prepared as KBr pellets (4000–400 cm^{-1} absorption range).

Single crystal X-ray diffraction study of **I–III** was carried out on a D8 QUEST Bruker diffractometer (MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator). The SMART and SAINT-Plus software programs were used to collect and edit data, to refine the unit cell parameters, and to apply absorption corrections [35]. All calculations for structure solution and refinement were performed using the SHELXL/PC [36] and OLEX2 [37] software. The structures were solved by direct methods and refined by the least squares method in the anisotropic approximation for non-hydrogen atoms. The key crystallographic data and structure refinement details are summarized in Table 1; selected bond lengths and bond angles are given in Table 2.

Full tables of atom coordinates, bond lengths and bond angles were deposited with the Cambridge Crystallographic Data Centre (nos. 1957185 (**I**), 2060227 (**II**), 2066549 (**III**) for structures **I–III**, respectively, deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

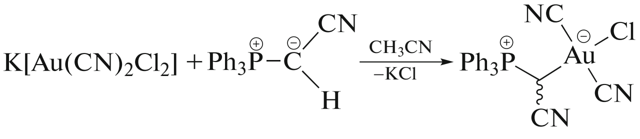
RESULTS AND DISCUSSION

Complexes **I** and **II** were obtained by reactions of aqueous solutions of potassium dichlorodicyanoaurate with organyltriphenylphosphonium chlorides (1 : 1 mol/mol) followed by recrystallization from acetonitrile.

$$K[Au(CN)_2Cl_2] + [Ph_3PR]Cl \xrightarrow{\substack{1.\,H_2O\\ 2.\,CH_3CN}} [Ph_3PR][Au(CN)_2Cl_2] + KCl,$$
$$R = CH_2CH{=}CHCH_3\ (\mathbf{I}),\ CH_2CN\ (\mathbf{II})$$

Apart from the light yellow-colored ionic complex **II**, formed as the major product, a minor product was isolated as colorless crystals of cyanomethyltriphenylphosphonium chlorodicyanogold (**III**) in 6% yield. Apparently, triphenylphosphonium cyanomethylide $Ph_3PC(H)CN$ is formed in the reaction mixture under conditions of synthesis; this compound attacks the $[Au(CN)_2Cl_2]^-$ anions to give complex **III**.

We carried out an alternative synthesis of **III** by the reaction of potassium dichlorodicyanoaurate with triphenylphosphonium cyanomethylide, which was specially prepared using a reported procedure [34]. In this case, complex **III** was the major reaction product isolated in 82% yield.



(III)

It is noteworthy that, owing to high reactivity of methylene hydrogen in cyanomethyltriphenylphosphonium chloride, the ylide complex can also be formed via direct C–H auration; however, this process is more typical for gold(I) compounds and usually takes place in the presence of bases [38–40].

The IR spectra of compounds **I–III** exhibit low-intensity C≡N stretching bands at 2216 (**I**), 2259 (**II**), 2257, 2156, and 2143 (**III**) cm^{-1}. The P–C_{Ph} bond vibrations are responsible for the absorption bands at

Table 1. Crystallographic data and X-ray experiment and structure refinement details for compounds **I**–**III**

Parameter	Value		
	I	**II**	**III**
M	637.27	1244.44	585.76
System	Monoclinic	Monoclinic	Monoclinic
Space group	$P2/c$	$P2_1$	$P2_1/c$
a, Å	16.983(8)	8.710(5)	8.370(5)
b, Å	8.483(3)	14.036(6)	14.984(8)
c, Å	17.082(7)	19.376(9)	17.232(13)
α, deg	90.00	90.00	90
β, deg	91.18(2)	90.10(3)	92.63(3)
γ, deg	90.00	90.00	90
V, Å^3	2460.4(18)	2368.7(19)	2159(2)
Z	4	2	4
ρ(calcd.), g/cm^3	1.720	1.745	1.802
μ, mm^{-1}	6.275	6.517	7.024
F(000)	1232.0	1192.0	1120.0
Crystal size, mm	0.34 × 0.27 × 0.11	0.44 × 0.31 × 0.22	0.65 × 0.5 × 0.46
Data collection range of θ, deg	5.86–65.66	5.9–54.28	5.93–56.996
Ranges of reflection indices	$-25 \leq h \leq 25$, $-12 \leq k \leq 12$, $-25 \leq l \leq 25$	$-11 \leq h \leq 11$, $-18 \leq k \leq 18$, $-24 \leq l \leq 24$	$-11 \leq h \leq 11$, $-20 \leq k \leq 20$, $-23 \leq l \leq 23$
Number of measured reflections	75175	36915	43689
Number of unique reflections (R_{int})	8951 (0.0425)	10456 (0.0313)	5463 (0.0611)
Number of reflections with $I > 2\sigma(I)$	8951	10456	5463
Number of refinement parameters	274	523	254
GOOF	1.074	1.077	1.060
R-factors on $F^2 > 2\sigma(F^2)$	$R_1 = 0.0357$, $wR_2 = 0.0681$	$R_1 = 0.0395$, $wR_2 = 0.0959$	$R_1 = 0.0460$, $wR_2 = 0.1149$
R-factors for all reflections	$R_1 = 0.0593$, $wR_2 = 0.0775$	$R_1 = 0.0438$, $wR_2 = 0.0990$	$R_1 = 0.0508$, $wR_2 = 0.1192$
Residual electron density (max/min), e/Å^3	1.42/–1.72	1.31/–2.86	2.71/–3.69

1450–1435 and 1005–995 cm^{-1} (1435, 997 (**I**), 1441, 995 (**II**), 1439, 997 (**III**) cm^{-1}). Also, the IR spectrum of **I** exhibits absorption bands that can be assigned to ν(C=C) (1683 cm^{-1}) and δ(HC=CH) (970 cm^{-1}) vibrations of the but-2-enyl moiety [41].

According to X-ray diffraction data, the crystals of **I** and **II** consist of organyltriphenylphosphonium cations and centrosymmretric square dichlorodicyanoaurate anions (Figs. 1 and 2, respectively). The coordination of phosphorus atoms is slightly distorted: the CPC angles vary in the ranges of 108.18(15)°–111.17(16)° (**I**) and 105.8(3)°–112.3(3)° (**II**). The but-2-enyl substituent in complex **II** occurs as the *trans*-isomer, in which the C(27)C(28)C(29) and C(28)C(29)C(30) angles are 123.5(4)° and 123.8(5)°, respectively. The gold atoms in the $[Au(CN)_2Cl_2]^-$ anions have almost undistorted square geometry, with the CAuC *trans*-angles and CAuCl *cis*-angles being close to 180° and 90°.

The P–C$_{Alk}$ bonds (1.812(3) Å (**I**), 1.826(7) and 1.827(7) Å (**II**)) are longer than the P–C$_{Ph}$ bonds (1.793(3)–1.797(3) Å (**I**) and 1.765(7)–1.797(6) Å (**II**)). The Au–C distances (2.002(4), 2.015(4) Å (**I**) and 1.955(12)–2.054(12) Å (**II**)) are shorter than the sum of the covalent radii of gold and *sp*-hybridized carbon atoms (2.05 Å [42]). The Au–Cl bond lengths (2.2808(12), 2.2947(13) Å (**I**) and 2.275(2)–2.286(2) Å (**II**)) are also shorter than the sum of the covalent radii of gold and chlorine atoms (2.38 Å [42]).

Table 2. Bond lengths (*d*) and bond angles (ω) in compounds **I**–**III**

Bond	*d*, Å	Angle	ω, deg
I			
Au(1)–Cl(1)	2.2947(13)	Cl(1)Au(1)Cl(1a)	177.81(7)
Au(2)–Cl(2)	2.2808(12)	C(7)Au(1)C(7a)	179.0(2)
Au(1)–C(7)	2.002(4)	C(7a)Au(1)Cl(1a)	90.45(12)
Au(2)–C(8)	2.015(4)	C(7)Au(1)Cl(1a)	89.57(12)
P(1)–C(1)	1.793(3)	Cl(2)Au(2)Cl(2b)	180.0
P(1)–C(11)	1.797(3)	C(8)Au(2)C(8b)	179.999(1)
P(1)–C(21)	1.793(3)	C(8b)Au(2)Cl(2b)	89.65(12)
P(1)–C(27)	1.812(3)	C(8)Au(2)Cl(2b)	90.35(12)
Symmetry codes: (a) $-x, y, 3/2 - z$; (b) $1 - x, 2 - y, 1 - z$		C(1)P(1)C(11)	108.18(15)
		C(1)P(1)C(27)	111.17(16)
II			
Au(1)–Cl(1)	2.281(2)	Cl(1)Au(1)Cl(2)	178.28(10)
Au(1)–Cl(2)	2.286(2)	C(9)Au(1)C(10)	178.9(5)
Au(1)–C(9)	1.955(12)	C(9)Au(1)Cl(1)	89.6(3)
Au(1)–C(10)	2.054(12)	C(9)Au(1)Cl(2)	89.8(3)
Au(2)–Cl(3)	2.275(2)	C(10)Au(1)Cl(1)	90.0(3)
Au(2)–Cl(4)	2.280(2)	C(10)Au(1)Cl(2)	90.7(3)
Au(2)–C(39)	2.023(13)	Cl(3)Au(2)Cl(4)	178.49(10)
Au(2)–C(40)	2.021(12)	C(39)Au(2)C(40)	178.7(4)
P(1)–C(1)	1.792(7)	C(39)Au(2)Cl(3)	90.8(3)
P(1)–C(11)	1.786(7)	C(39)Au(2)Cl(4)	89.9(3)
P(1)–C(21)	1.792(7)	C(40)Au(2)Cl(3)	90.5(3)
P(1)–C(7)	1.827(7)	C(40)Au(2)Cl(4)	88.9(3)
P(2)–C(31)	1.797(6)	C(1)P(1)C(21)	111.7(3)
P(2)–C(41)	1.765(7)	C(11)P(1)C(7)	106.1(3)
P(2)–C(51)	1.789(7)	C(41)P(2)C(31)	112.3(3)
P(2)–C(37)	1.826(7)	C(41)P(2)C(37)	105.8(3)
III			
Au(1)–Cl(1)	2.318(2)	C(7)Au(1)Cl(1)	177.76(15)
Au(1)–C(9)	1.997(8)	C(9)Au(1)C(10)	176.3(3)
Au(1)–C(10)	2.006(7)	C(9)Au(1)C(7)	93.7(2)
Au(1)–C(7)	2.121(5)	C(9)Au(1)Cl(1)	88.5(2)
C(7)–C(8)	1.427(9)	C(10)Au(1)C(7)	88.2(2)
P(1)–C(1)	1.786(6)	C(10)Au(1)Cl(1)	89.7(2)
P(1)–C(11)	1.801(7)	P(1)C(7)Au(1)	113.8(3)
P(1)–C(21)	1.791(7)	C(8)C(7)Au(1)	111.1(4)
P(1)–C(7)	1.851(6)	C(8)C(7)P(1)	112.7(4)
C(8)–N(1)	1.137(10)	C(11)P(1)C(7)	105.4(3)
C(9)–N(2)	1.124(11)	C(21)P(1)C(7)	113.9(3)

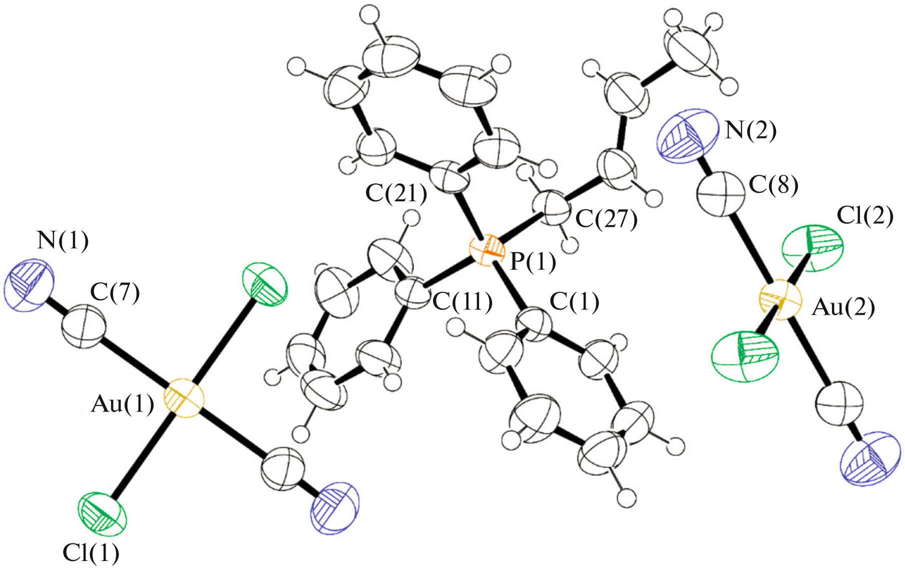


Fig. 1. General view of complex **I** (the thermal ellipsoids are drawn at 50% probability level).

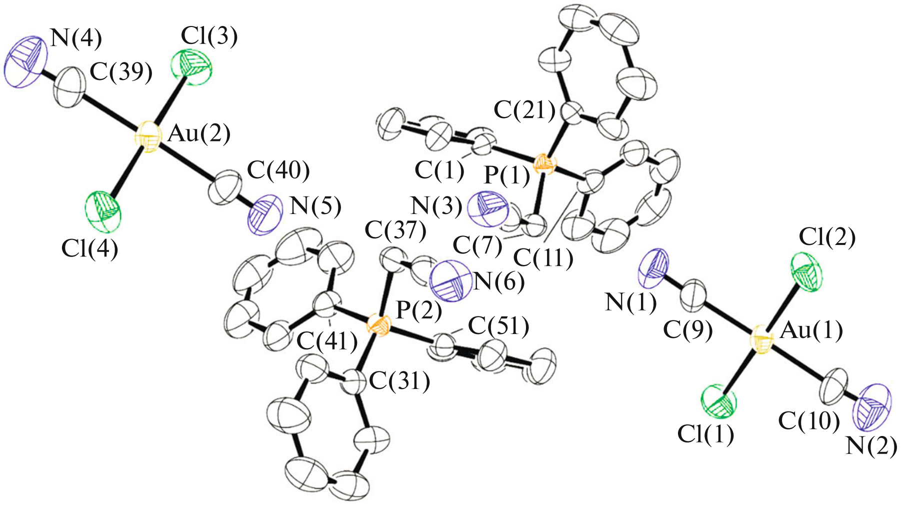


Fig. 2. General view of complex **II** (the thermal ellipsoids are drawn at 50% probability level; the hydrogen atoms are not shown).

In complex **III**, the phosphorus and gold atoms have a tetrahedral and square coordinations, respectively; the *trans*-position of gold relative to chlorine is occupied by the ylide carbon atom (Fig. 3). The CPC angles (105.4(3)°–113.9(3)°) differ little from those for **II** and **III**; the P(1)C(7)Au(1), C(8)C(7)Au(1), and C(8)C(7)P(1) angles are 113.8(3)°, 111.1(4)°, and 112.7(4)°, respectively. The P(1)–C_{Ph} bonds are shorter than P(1)–C(7) (1.851(6) Å) and vary in the range of 1.786(6)–1.801(7) Å.

The sizes of the CAuC *trans*-angles (176.3(3)° and 177.76(15)°) indicate a minor distortion of the square geometry of the gold atom; the CAuC and CAuCl *cis*-angles are 93.7(2)°, 88.2(2)° and 88.5(2)°, 89.7(2)°, respectively. The Au(1)–C_{CN} distances (1.997(8), 2.006(7) Å) virtually do not differ from those in the ionic complexes; the Au(1)–C(7) distance is 2.121(5) Å, which coincides with the sum of the covalent radii of gold and sp^3-hybridized carbon atoms (2.12 Å [42]). The Au(1)–Cl(1) bond (2.318(2) Å) is 0.035 Å longer than the average Au–Cl bond in **I** and **II** (2.283 Å); this is caused by the *trans*-effect of the ylide ligand. It is noteworthy that in a similar, structurally characterized $Ph_3PC(H)(CN)AuCl_3$ complex, analogous bond lengths between the gold atom and carbon and chlorine atoms are shorter (Au–C 2.083(4), Au–Cl 2.3095(13) Å) [43].

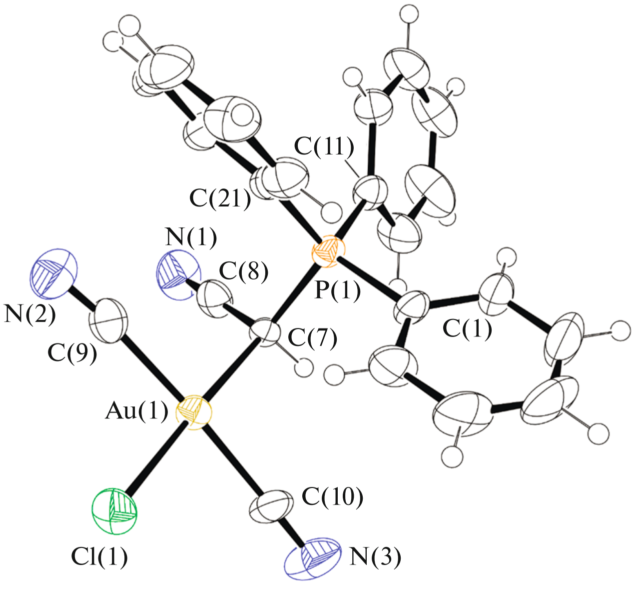


Fig. 3. General view of complex **III** (the thermal ellipsoids are drawn at 50% probability level).

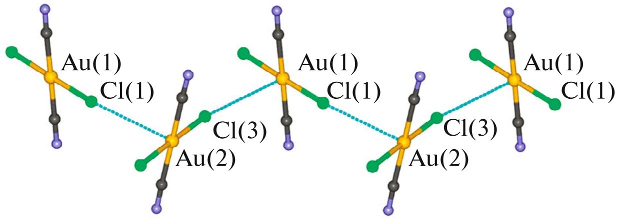


Fig. 4. Fragment of the coordination pseudopolymer chain in the crystals of **II**.

The structure of the crystals of **I**–**III** is formed by the C–H···N≡C (2.58, 2.64 Å (**I**); 2.28–2.43 Å (**II**); 2.40–2.55 Å (**III**)) and C–H···Cl–Au hydrogen bonds (2.83 Å (**I**); 2.81, 2.84 Å (**II**); 2.81 Å (**III**)). In the crystal of **II**, the $[Au(CN)_2Cl_2]^-$ anions form the coordination pseudopolymer chains arranged along the crystallographic *a* axis (Fig. 4) and held together via Au(1)···Cl(3) and Au(2)···Cl(1) contacts (3.40 Å), which are shorter than the sum of the van der Waals radii of the gold and chlorine atoms (3.41 Å [44]).

Thus, the reaction of (but-2-enyl)triphenylphosphonium chloride with potassium dichlorodicyanoaurate gave the monomeric ionic complex, (but-2-enyl)triphenylphosphonium dichlorodicyanoaurate. A similar reaction involving cyanomethyltriphenylphosphonium chloride resulted in the formation of cyanomethyltriphenylphosphonium dichlorodicyanoaurate, which is composed of pseudopolymer chains of the $[Au(CN)_2Cl_2]^-$ anions held by the Au···Cl contacts, and a minor reaction product, the (cyanomethyltriphenylphosphonium)chlorodicyanogold ylide complex.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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