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# Structure and Synthesis of Dihalodicyanoaurate Complexes $[Ph_3PR][Au(CN)_2Hal_2]$ (Hal = Cl, R = Me, $CH_2Ph$; Hal = Br, R = *cyclo*-$C_6H_{11}$; Hal = I, R = Ph)

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**Abstract**—Ionic gold(III) complexes $[Ph_3PMe][Au(CN)_2Cl_2]$ (**I**), $[Ph_3PCH_2Ph][Au(CN)_2Cl_2]$ (**II**), $[Ph_3PC_6H_{11}$-*cyclo*$][Au(CN)_2Br_2]$ (**III**), and $[Ph_4P][Au(CN)_2I_2]$ (**IV**) are synthesized by the reactions of organyltriphenylphosphonium halides with the corresponding potassium dihalodicyanoaurates in water followed by the recrystallization of the reaction products from acetonitrile or DMSO. The complexes are characterized by X-ray diffraction (XRD) (CIF files CCDC nos. 1901681 (**I**), 1912903 (**II**), 1912919 (**III**), and 2048146 (**IV**)) and IR spectroscopy. According to the XRD data, the crystals of complexes **I**–**IV** consist of centrosymmetric square-planar anions $[Au(CN)_2Hal_2]^-$ and organyltriphenylphosphonium cations with a slightly distorted tetrahedral coordination of the phosphorus atoms. The structural organization in the crystals of complexes **II**–**IV** is formed by interionic hydrogen bonds C–H···N≡C, whereas no significant interionic contacts are observed in the crystals of complex **I**.

**Keywords:** potassium dihalodicyanoaurates, organyltriphenylphosphonium halides, synthesis, structure, XRD

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## INTRODUCTION

At present, metal-organic frameworks are objects of intense studies due to a possibility of their rational design and controlling of properties [1–3]. Among numerous coordination building blocks, ionic cyanide complexes of transition metals, in particular, monovalent and trivalent gold compounds, play a significant role. The metal-organic frameworks based on these building blocks attract researchers due to a variety of physicochemical properties (magnetism [4–7], luminescence [7–11], vapochromism [12–15], birefringence [16–19], and negative thermal expansion coefficient [20, 21]), which can also be varied by a strategical choice of auxiliary ligands and counterions. For example, the birefringence ability of the cyanoaurate complexes enhances on going from $[Au(CN)_2]^-$ to $[Au(CN)_2Hal_2]^-$ due to a high polarizability of the Au–Hal bonds [16].

In this work, we described the synthesis and specific features of the earlier unknown dihalodicyanoaurate complexes of organyltriphenylphosphonium $[Ph_3PR][Au(CN)_2Cl_2]$ (R = Me (**I**), $CH_2Ph$ (**II**)), and $[Ph_3PC_6H_{11}$-*cyclo*$][Au(CN)_2Br_2]$ (**III**) and the already known diiododicyanoaurate complex $[Ph_4P][Au(CN)_2I_2]$ (**IV**), the structure of which was determined with a higher accuracy.

## EXPERIMENTAL

**Synthesis of methyltriphenylphosphonium dichlorodicyanoaurate (I).** An aqueous solution of methyltriphenylphosphonium bromide (94 mg, 0.28 mmol) was added with stirring to a solution of potassium dichlorodicyanoaurate (100 mg, 0.28 mmol) in water (10 mL). The formed bright yellow precipitate was filtered off, two timed washed with water by 5-mL portions, and dried. A weighed sample (67 mg) was recrystallized from acetonitrile. The yield of crystals of bright yellow complex **I** with $T_m$ = 154°C was 53 mg (87%).

IR (ν, $cm^{-1}$): 3063, 2992, 2916, 2218, 2168, 1587, 1487, 1439, 1398, 1339, 1194, 1163, 1117, 1028, 997, 901, 839, 787, 745, 719, 687, 505, 473, 449, 430.

For $C_{21}H_{18}N_2PCl_2Au$

| | | |
|---|---|---|
| Anal. calcd., % | C, 42.23 | H, 3.04 |
| Found, % | C, 42.19 | H, 3.07 |

Benzyltriphenylphosphonium dichlorodicyanoaurate (**II**), cyclohexyltriphenylphosphonium dibromodicyanoaurate (**III**), and tetraphenylphosphonium diiododicyanoaurate (**IV**) were synthesized using a similar procedure, and DMSO was used as the solvent for the recrystallization of complex **IV**.

Complex **II**: (57%), light yellow crystals, $T_{\mathrm{decomp}}$ = 167°C.

IR (ν, cm$^{-1}$): 3062, 3039, 2951, 2914, 2853, 2214, 2170, 1686, 1585, 1497, 1485, 1456, 1437, 1402, 1339, 1315, 1188, 1134, 1113, 1070, 1026, 997, 914, 851, 833, 785, 748, 719, 696, 581, 513, 496, 449, 424.

For $C_{27}H_{22}N_2PCl_2Au$

| | | |
|---|---|---|
| Anal. calcd., % | C, 48.16 | H, 3.30 |
| Found, % | C, 48.08 | H, 3.40 |

Complex **III**: (83%), light yellow crystals, $T_m$ = 212°C.

IR (ν, cm$^{-1}$): 2940, 2860, 2141, 1734, 1717, 1701, 1684, 1653, 1558, 1541, 1508, 1489, 1456, 1439, 1420, 1339, 1319, 1192, 1177, 1121, 1109, 997, 887, 851, 746, 723, 691, 546, 525, 517, 419.

For $C_{26}H_{26}N_2PBr_2Au$

| | | |
|---|---|---|
| Anal. calcd., % | C, 41.40 | H, 3.48 |
| Found, % | C, 41.36 | H, 3.54 |

Complex **IV**: (86%), orange crystals, $T_m$ = 252°C.

IR (ν, cm$^{-1}$): 3059, 3020, 2989, 2169, 2154, 1679, 1585, 1481, 1433, 1390, 1182, 1070, 1026, 995, 758, 748, 725, 686, 615, 526, 464, 441, 418.

For $C_{26}H_{20}N_2PI_2Au$

| | | |
|---|---|---|
| Anal. calcd., % | C, 37.08 | H, 2.40 |
| Found, % | C, 36.98 | H, 2.54 |

The IR spectra of compounds **I**–**IV** were recorded on a Shimadzu IRAffinity-1S FT-IR spectrometer for samples prepared as KBr pellets (absorption range 4000–400 cm$^{-1}$).

**XRD** of crystals of complexes **I**–**IV** was carried out on a D8 QUEST Bruker diffractometer (Mo$K_\alpha$ radiation, λ = 0.71073 Å, graphite monochromator). Data were collected and processed, unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [22]. All calculations on structure determination and refinement were performed using the SHELXL/PC [23] and OLEX2 [24] programs. The structures were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and structure refinement results are given in Table 1. Selected bond lengths and bond angles are listed in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1901681, 1912903, 1912919, and 2048146 for the structures of complexes **I**–**IV**, respectively; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

## RESULTS AND DISCUSSION

This work is a continuation of a series of studies devoted to the investigation of the structures and properties of the dihalodicyanoaurate complexes [25–29].

Complexes **I**–**IV** were synthesized by the reactions of aqueous solutions of potassium dihalodicyanoaurates with the corresponding tetraorganylphosphonium halides

$$\mathrm{K[Au(CN)_2Hal_2] + [Ph_3PR]Hal \xrightarrow{H_2O} [Ph_3PR][Au(CN)_2Hal_2] + KHal}$$

$$\mathrm{Hal = Cl{:}\ R = Me\ (\mathbf{I}),\ CH_2Ph\ (\mathbf{II});}$$

$$\mathrm{Hal = Br{:}\ R =}\ \textit{cyclo}\text{-}\mathrm{C_6H_{11}\ (\mathbf{III});}$$

$$\mathrm{Hal = I{:}\ R = Ph\ (\mathbf{IV}).}$$

Stable in air transparent light yellow crystals were prepared by the subsequent recrystallization of complexes **I**–**III** from acetonitrile. Note that only solvate dibromodicyanoaurate derivative **III**·1/2PhH, which was isolated by the recrystallization of the corresponding precipitate from a MeCN–benzene (8 : 5 vol/vol) mixture, has previously been described [25].

Orange crystals of complex **IV** were obtained after the recrystallization of the reaction product from DMSO along with a minor amount of tetraphenylphosphonium triiodide identified by XRD (dark brown crystals). The formation of the latter can be explained by the reaction of the phosphonium salt with molecular iodine evolved during the oxidation of potassium iodide. The syntheses and structures of similar triiodides, including $[Ph_4P][I_3]$, were described [30–32]. A possibility of incorporating $[I_3]^-$ anions into the lattice of the diiododicyanoaurate complex was also demonstrated [29].

The presence of cyano groups in organic and inorganic compounds can rather easily be established using IR spectroscopy: the corresponding absorption bands lie in a narrow frequency range (from 2200 to 2000 cm$^{-1}$ [33]) due to the absence of a significant influence of the environment on vibrations of these bonds. For instance, the absorption bands of stretching vibrations of C≡N bonds in compounds **I**–**IV** are low-intensity and lie at 2218 and 2168 (**I**), 2214 and 2170 (**II**), 2141 (**III**), and 2169 and 2154 (**IV**) cm$^{-1}$. The absorption bands in a range of 1450–1435 cm$^{-1}$ characteristic of similar derivatives correspond to the P–$C_{\mathrm{Ph}}$

**Table 1.** Crystallographic data and experimental and structure refinement parameters for complexes **I**–**IV**

| Parameter | Value | | | |
|---|---|---|---|---|
| | **I** | **II** | **III** | **IV** |
| *FW* | 597.21 | 673.30 | 754.24 | 842.18 |
| Crystal system | Triclinic | Triclinic | Triclinic | Monoclinic |
| Space group | $P\bar{1}$ | $P\bar{1}$ | $P\bar{1}$ | $C2/c$ |
| $a$, Å | 8.751(7) | 9.769(7) | 7.713(4) | 18.200(8) |
| $b$, Å | 8.924(11) | 11.340(10) | 10.099(5) | 7.897(5) |
| $c$, Å | 15.229(13) | 13.349(9) | 18.881(12) | 18.635(8) |
| α, deg | 102.69(4) | 69.16(3) | 90.58(2) | 90.00 |
| β, deg | 90.47(3) | 70.13(3) | 93.48(3) | 91.473(17) |
| γ, deg | 102.75(4) | 69.86(3) | 110.870(18) | 90.00 |
| $V$, Å$^3$ | 1129.6(19) | 1257.0(17) | 1370.9(13) | 2678(2) |
| $Z$ | 2 | 2 | 2 | 4 |
| $\rho_{calc}$, g/cm$^3$ | 1.756 | 1.779 | 1.827 | 2.089 |
| μ, mm$^{-1}$ | 6.827 | 6.146 | 8.354 | 7.876 |
| $F$(000) | 572.0 | 652.0 | 720.0 | 1560.0 |
| Crystal size, mm | 0.36 × 0.27 × 0.17 | 0.85 × 0.26 × 0.08 | 0.53 × 0.25 × 0.18 | 0.38 × 0.2 × 0.15 |
| Data collection range over θ, deg | 5.96–60.18 | 6.32–84.1 | 5.76–71.52 | 6.056–55.908 |
| Ranges of reflection indices | $-12 \le h \le 12$,<br>$-12 \le k \le 12$,<br>$-21 \le l \le 21$ | $-18 \le h \le 18$,<br>$-21 \le k \le 21$,<br>$-24 \le l \le 24$ | $-12 \le h \le 12$,<br>$-16 \le k \le 16$,<br>$-31 \le l \le 30$ | $-24 \le h \le 23$,<br>$-10 \le k \le 10$,<br>$-24 \le l \le 24$ |
| Measured reflections | 69945 | 106598 | 93252 | 27120 |
| Independent reflections ($R_{int}$) | 6613 (0.0375) | 17087 (0.0632) | 12698 (0.2074) | 3218 (0.0312) |
| Reflections with $I > 2\sigma(I)$ | 6613 | 17087 | 12698 | 3218 |
| Refinement variables | 248 | 301 | 292 | 158 |
| GOOF | 1.091 | 1.065 | 1.071 | 1.096 |
| $R$ factors for $F^2 > 2\sigma(F^2)$ | $R_1 = 0.0699$,<br>$wR_2 = 0.2361$ | $R_1 = 0.0642$,<br>$wR_2 = 0.1369$ | $R_1 = 0.0705$,<br>$wR_2 = 0.1649$ | $R_1 = 0.0163$,<br>$w\mathrm{R}_2 = 0.0338$ |
| $R$ factors for all reflections | $R_1 = 0.0982$,<br>$wR_2 = 0.2655$ | $R_1 = 0.1418$,<br>$wR_2 = 0.1607$ | $R_1 = 0.1164$,<br>$wR_2 = 0.1926$ | $R_1 = 0.0203$,<br>$w\mathrm{R}_2 = 0.0351$ |
| Residual electron density (max/min), e/Å$^3$ | 3.91/–2.45 | 4.03/–1.67 | 4.42/–4.01 | 0.36/–0.41 |

bond vibrations (1439 (**I**), 1437 (**II**), 1439 (**III**), and 1433 (**IV**) cm$^{-1}$) [33].

According to the XRD data, the crystals of ionic complexes **I**–**III** consist of organyltriphenylphosphonium cations with tetrahedrally coordinated phosphorus atoms and two types of crystallographically independent centrosymmetric square-planar dihalodicyanoaurate anions (Figs. 1–3). The crystal of ionic complex **IV** contains tetraphenylphosphonium cations and only one type of $[Au(CN)_2I_2]^-$ anions (Fig. 4).

The geometry of phosphonium cations in all structures is insignificantly distorted. The CPC angles vary in ranges of 108.4(4)°–110.5(4)° (**I**), 108.0(2)°–110.5(2)° (**II**), 107.5(2)°–110.5(2)° (**III**), and 105.5(4)°–111.3(2)° (**IV**). The P–C bond lengths in the complexes are close to each other and independent

**Table 2.** Bond lengths (*d*) and bond angles (ω) in the structures of complexes **I**–**IV**

| Bond | *d*, Å | Angle | ω, deg |
|---|---|---|---|
| | | **I** | |
| Au(1)–Cl(1) | 2.389(2) | Cl(1b)Au(1)Cl(1) | 180.0 |
| Au(2)–Cl(2) | 2.444(3) | C(8)Au(1)Cl(1) | 90.3(4) |
| Au(1)–C(8) | 1.941(14) | C(8a)Au(1)Cl(1) | 89.7(4) |
| Au(2)–C(9) | 2.18(2) | Cl(2b)Au(2)Cl(2) | 179.999(1) |
| P(1)–C(1) | 1.782(9) | C(9)Au(2)Cl(2) | 88.2(6) |
| P(1)–C(11) | 1.806(8) | C(9)Au(2)Cl(2b) | 91.8(6) |
| P(1)–C(21) | 1.791(8) | C(21)P(1)C(11) | 108.4(4) |
| P(1)–C(7) | 1.783(9) | C(7)P(1)C(11) | 110.5(4) |
| Symmetry transforms: (a) $1-x, -y, -z$; (b) $2-x, 2-y, 1-z$ | | | |
| | | **II** | |
| Au(1)–Cl(1) | 2.278(2) | Cl(1)Au(1)Cl(1a) | 180.0 |
| Au(2)–Cl(2) | 2.2825(19) | C(8)Au(1)Cl(1) | 89.9(2) |
| Au(1)–C(8) | 2.011(8) | C(8)Au(1)Cl(1a) | 90.1(2) |
| Au(2)–C(9) | 2.008(5) | Cl(2b)Au(2)Cl(2) | 180.0 |
| P(1)–C(21) | 1.813(5) | C(9)Au(2)Cl(2b) | 88.81(17) |
| P(1)–C(11) | 1.812(5) | C(9)Au(2)Cl(2) | 91.19(17) |
| P(1)–C(7) | 1.796(5) | C(7)P(1)C(11) | 108.0(2) |
| P(1)–C(1) | 1.788(4) | C(31)C(7)P(1) | 112.5(3) |
| C(31)–C(7) | 1.523(7) | C(31)C(7)P(1) | 112.5(3) |
| Symmetry transforms: (a) $1-x, 2-y, -z$; (b) $-x, 1-y, 1-z$ | | | |
| | | **III** | |
| Au(1)–Br(1) | 2.4214(13) | Br(1a)Au(1)Br(1) | 180.0 |
| Au(1)–C(7) | 2.009(7) | C(7)Au(1)Br(1) | 90.5(2) |
| Au(2)–Br(2) | 2.4192(13) | C(7)Au(1)Br(1a) | 89.5(2) |
| Au(2)–C(8) | 2.008(7) | Br(2b)Au(1)Br(b) | 180.0 |
| P(1)–C(31) | 1.813(5) | C(8)Au(2)Br(2b) | 90.86(18) |
| P(1)–C(21) | 1.795(5) | C(8)Au(2)Br(2) | 89.14(18) |
| P(1)–C(1) | 1.790(5) | C(21)P(1)C(11) | 107.5(2) |
| P(1)–C(11) | 1.806(6) | C(1)P(1)C(21) | 110.5(2) |
| Symmetry transforms: (a) $1-x, 2-y, 1-z$; (b) $2-x, 2-y, 2-z$ | | | |
| | | **IV** | |
| Au(1)–I(1) | 2.6035(10) | I(1a)Au(1)I(1) | 179.999(1) |
| Au(1)–C(7) | 1.998(6) | C(7a)Au(1)C(7) | 180.000(1) |
| P(1)–C(1) | 1.799(5) | C(7)Au(1)I(1) | 90.02(18) |
| P(1)–C(11) | 1.793(6) | C(7)Au(1)I(1a) | 89.98(18) |
| Symmetry transforms: | | C(11)P(1)C(1) | 111.3(2) |
| (a) $1/2-x, 3/2-y, 1-z$; (b) $1-x, y, 1/2-z$ | | C(1)P(1)C(1b) | 105.5(6) |

of the nature of the substituent (1.782(9)–1.806(8) Å (**I**), 1.788(4)–1.813(5) Å (**II**), 1.790(5)–1.813(5) Å (**III**), and 1.793(6)–1.799(5) Å (**IV**)).

The gold atoms in the $[Au(CN)_2Hal_2]^-$ anions have a nearly undistorted square-planar environment with the *trans*-CAuC angles very close to 180° and *cis*-CAuHal angles in ranges of 88.2(6)°–91.8(6)° (**I**), 88.81(17)°–91.19(17)° (**II**), 89.14(18)°–90.86(18)° (**III**), and 89.98(18)°–90.02(18)° (**IV**). The average Au–C bond lengths (2.06(2) Å (**I**), 2.010(7) Å (**II**), 2.009(7) Å (**III**), and 1.998(6) Å (**IV**)) are close to the sums of covalent radii of gold and carbon atoms (2.05 Å [34]). The average Au–Hal bond lengths

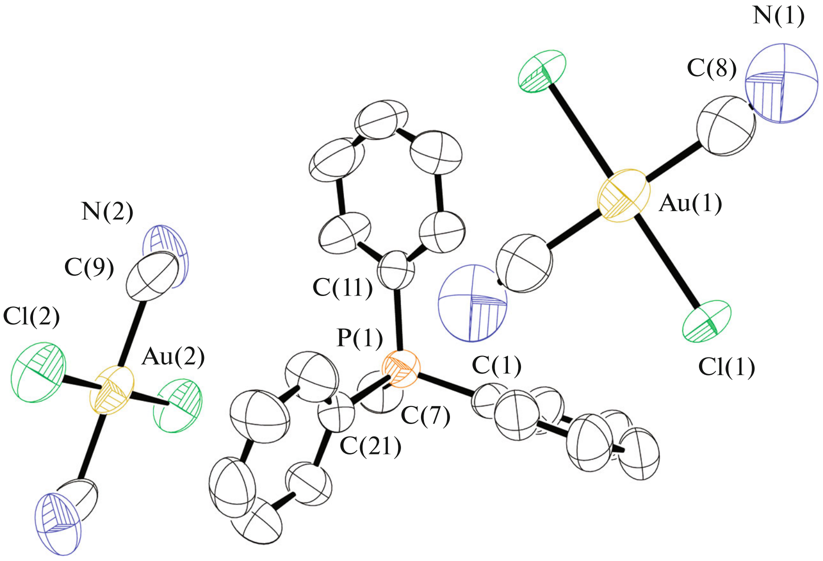

**Fig. 1.** Structure of complex $[Ph_3PMe][Au(CN)_2Cl_2]$ (**I**).

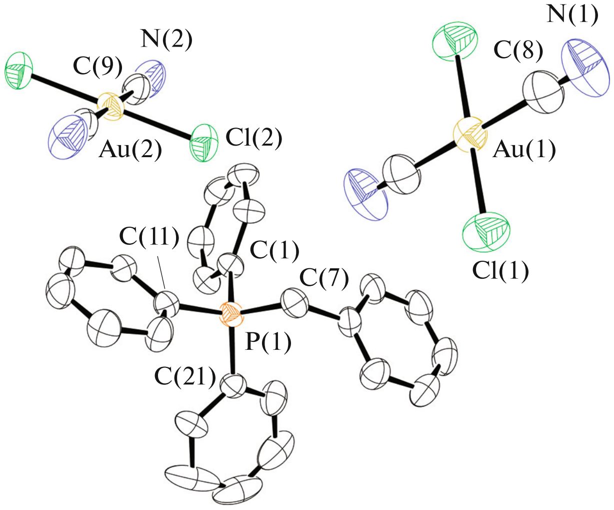

**Fig. 2.** Structure of complex $[Ph_3PCH_2Ph][Au(CN)_2Cl_2]$ (**II**).

(2.417(3) Å (**I**), 2.280(2) Å (**II**), 2.4203(13) Å (**III**), and 2.6035(10) Å (**IV**)) slightly differ from the sum of their covalent radii (Au–Cl 2.38 Å, Au–Br 2.56 Å, Au–I 2.77 Å [34]).

The steric structures of complexes **II**–**IV** are due to weak interionic hydrogen bonds C–H···N≡C ($C-H_{Ph}$···N≡C 2.56 Å (**II**); $C-H_{Ph}$···N≡C 2.43–2.59 Å, $C-H_{cyclohexyl}$···N≡C 2.47 Å (**III**), $C-H_{Ph}$···N≡C 2.63 Å (**IV**)), which are lower than the sums of van der Waals radii of hydrogen and nitrogen atoms (2.65 Å [35]). Complex **IV** also contains rather close contacts Au–I···$\pi_{Ph}$ (with the distances from the iodine atom to the plane of the Ph rings equal to 3.41 Å) forming zigzag pseudopolymer chains with the $[Ph_4P]^+$ cations. No significant interionic contacts are observed in the crystals of complex **I**.

Unlike the earlier described crystals of complex **III**·1/2PhH, the crystals of complex **III** have a higher number and a larger variety of C–H···N≡C hydrogen bonds and, as a consequence, are characterized by a higher packing density: $\rho_{calc}$ = 1.827 g/cm$^3$ (versus 1.770 g/cm$^3$ for **III**·1/2PhH).

Thus, the complexes of methyl- (**I**) and benzyltriphenylphosphonium dichlorodicyanoaurate (**II**), cyclohexyltriphenylphosphonium dibromodicyanoaurate (**III**), and tetraphenylphosphonium diiododicyanoaurate (**IV**) synthesized by the reactions of the corresponding organyltriphenylphosphonium halides and potassium dihalodicyanoaurates in water have

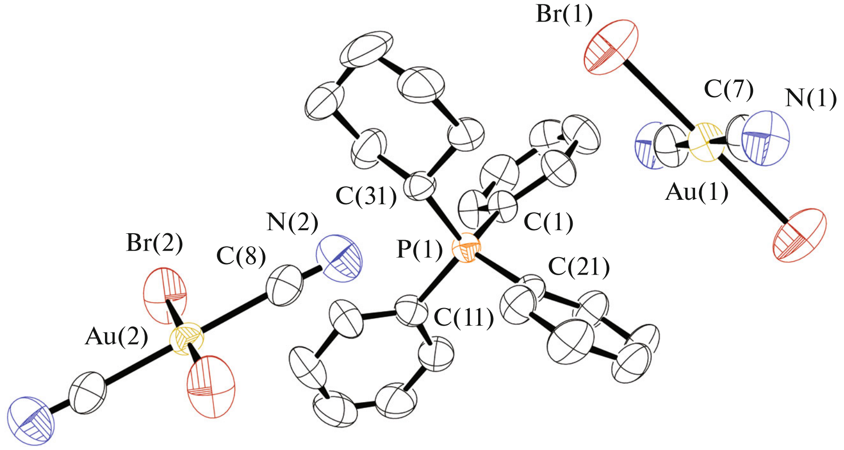

**Fig. 3.** Structure of complex $[Ph_3PC_6H_{11}\text{-}cyclo][Au(CN)_2Br_2]$ (**III**).

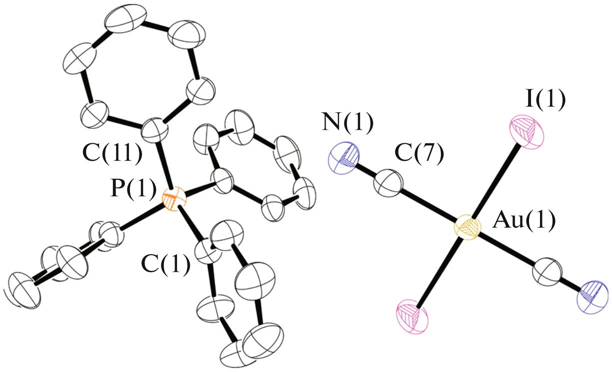

**Fig. 4.** Structure of complex $[Ph_4P][Au(CN)_2I_2]$ (**IV**).

monomeric ionic structures. The steric structures of the crystals of complexes **II–IV** are due to the interionic hydrogen bonds C–H···N≡C, whereas no significant contacts are observed in the crystals of complex **I**.

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## CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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