

COORDINATION
COMPOUNDS

Behavior of Dodecahydro-*closo*-dodecaborate Anion $B_{12}H_{12}^{2-}$ in Reaction with $Au(Ph_3P)Cl$

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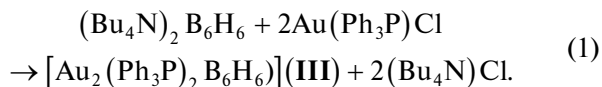
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Abstract—The reaction of the anion $B_{12}H_{12}^{2-}$ with $[Au(Ph_3P)Cl]$ in acetonitrile was studied. The nature of the initial cation of the *closo*-dodecaborate and the reagent ratio was shown to affect complexing. The X-ray structures of compounds $Au(Ph_3P)_2[AgB_{12}H_{12}]$ (**I**) and $[Au_9(Ph_3P)_8]B_{24}H_{23}$ (**II**) were determined. Complex **I** is the first example where $B_{12}H_{12}^{2-}$ is coordinated to silver(I) atoms in the face coordination mode. In complex **II**, $B_{12}H_{12}^{2-}$ is oxidized to the dimeric trianion $B_{24}H_{23}^{3-}$, where the two icosahedra are linked by a B–H–B' bridge.

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The documentation concerning the coordination chemistry of cluster boron anions $B_nH_n^{2-}$ (where $n = 10, 12$) implies that not only these electron-deficient compounds having low donating capacities [1] are coordinated to metal atoms, but they also show a great variety of types of bonding to the metal in coordination compounds. In copper(I) and silver(I) complexes, for example, the *closo*-dodecaborate anion was shown to be capable of forming metal complexes where $B_{12}H_{12}^{2-}$ is involved in the inner coordination sphere via forming $3c2e$ (MHB) bonds with metal atoms [2–5] in the same manner as the *closo*-decaborate anion is [6].

Only one gold compound with *closo*-hexaborate anion $B_6H_6^{2-}$, having the composition $[Au_2(Ph_3P)_2(B_6H_6)]$ was described [7, 8]; this compound was prepared by the reaction



Each Au(I) atom in compound **III** is linked to the triangular face B_3 of the *closo*-hexaborate anion and the phosphorus atom of triphenylphosphine. The terminal hydrogen atoms are presumably uninvolved in gold coordination [7, 8]. The IR spectrum of compound **III** features an absorption band at 2473 cm^{-1} associated with the bending vibrations of pendant B–H bonds in $B_6H_6^{2-}$.

Our preliminary studies of reactions of $B_{10}H_{10}^{2-}$ and $B_{12}H_{12}^{2-}$ with $Au(Ph_3P)Cl$ allowed us to isolate the complex $[1-(Au_3(Ph_3P)_3)B_{10}H_9]$ and complex **II**, respec-

tively [9]. Here, we study the reaction of *closo*-dodecaborate anion $B_{12}H_{12}^{2-}$ with $Au(Ph_3P)Cl$ in acetonitrile, study the effect of the nature of the cation on the initial *closo*-dodecaborate and the reagent ratio on the product composition, and solve the structure of the products.

EXPERIMENTAL

Syntheses

Dodecahydro-*closo*-dodecaboratosilver(I) $[Ag_2B_{12}H_{12}]$ was prepared according to [10].

Chloro(triphenylphosphine)gold(I) $Au(Ph_3P)Cl$ was prepared by reacting $[HAuCl_4]$ with Ph_3P as described elsewhere [11].

Bis(triphenylphosphine)gold(I) catena-(μ^2 -dodecahydro-*closo*-dodecaborate $\{(Au(Ph_3P)_2)(AgB_{12}H_{12})\}_n$ (I**).** To a solution containing 0.1 mol of $Au(Ph_3P)Cl$ in acetonitrile + benzene (20 mL), added was a solution of 0.1 mole of $[Ag_2B_{12}H_{12}]$ in 20 mL of acetonitrile. The Au^+ to $B_{12}H_{12}^{2-}$ ratio in the reaction mixture was 1 : 1. The resulting clear reaction solution was allowed to stand in the dark for 12 h. Crystals of prismatic habit were forming upon isothermal evaporation of solvents. Yield of compound **I** was about 80%.

Octakis(triphenylphosphine)nonagold(I) tricosahydro-tetracosaborate $[Au_9(Ph_3P)_8]B_{24}H_{23}$ (II**).** To a solution of 0.1 mole of $[Ag_2B_{12}H_{12}]$ in 20 mL of acetonitrile, added was a solution of $Au(Ph_3P)Cl$, taken in an excess, in acetonitrile + benzene (20 mL). After the reagents were combined, a white precipitate ($AgCl$) was gradually forming. The reaction solution was allowed to stand in the dark for 5 h. Light-orange crys-

tals of prismatic habit coated with a reduced finely divided silver layer were isolated from the reaction mixture. The yield of compound **II** did not exceed 50%.

Elemental Analyses

Analysis for carbon and hydrogen was carried out on a CHINS-3 FA 1108 Elemental Analyzer (Carlo Erba). Boron was determined by atomic absorption spectroscopy on PerkinElmer spectrophotometers model 2100 equipped with an HGA-700 electrothermal atomizer [12]. Silver and gold were determined on AAS-303 in acetylene–air flame.

For $C_{36}H_{48}AgAuB_{12}P_2$ anal. calcd., %: H, 4.95; B, 13.27; C, 44.24; Ag, 11.04; Au, 20.15.

For compound **I** found, %: H 4.78; B 12.58; C 44.12; Ag, 10.69; Au, 20.08.

For $C_{144}H_{167}Au_9B_{24}P_8$ anal. calcd., %: H, 4.03; B, 6.21; C, 41.40; Au, 42.43.

For compound **II** found, %: H, 3.91; B, 6.12; C, 41.10; Au, 42.24.

IR Spectra

IR spectra for compounds **I** and **II** were recorded as Nujol mulls on an Infracum FT-02 (Lumex) spectrophotometer in the range 600–4000 cm^{-1} with resolution of 1 cm^{-1} .

For compound **I**: $\nu(BH)$ 2475; $\nu(BH)_{MHB}$ 2342, 2150; $\delta(BBH)$ 1070 cm^{-1} .

For compound **II**: $\nu(BH)$ 2515; $\nu(BH)_{BHB}$ 1875; $\delta(BBH)$ 1054 cm^{-1} .

^{11}B NMR Spectra

^{11}B NMR spectra of solutions of complexes **I** and **II** in acetonitrile were recorded on a Bruker AC 200 spectrometer at a frequency of 64.297 MHz with internal deuterium stabilization relative to external boron trifluoride etherate. ^{11}B NMR data for complex **II** are found in [9].

X-ray Crystallography

Diffraction intensity arrays were obtained from crystals of complexes **I** and **II** on Bruker AXS P4 and Bruker AXS SMART 1000 automated diffractometers (λMoK_{α} , graphite monochromator, ω scans) at 297(2) and 120(2) K, respectively. Program packages XSCANS [13] (for complex **I**) and SMART and SAINT [14] (for complex **II**) were used in collecting and processing $I(hkl)$ data sets. The correction for absorption was applied using the MULTISCAN method [15, 16]. The structures were solved by a direct method (SHELXS97 [17]). Non-hydrogen atoms were refined in the anisotropic approximation. The positions of the H atoms of polyhedral anions were found from difference syntheses and then idealized.

Table 1. Selected crystallographic data, details of experiment, and refinement factors for compounds **I** and **II**

| Parameter | I | II |
|--|-------------------------|-------------------------|
| Formula weight | 971.19 | 2070.19 |
| Crystal system | Monoclinic | Monoclinic |
| Space group, Z | $P2_1/c$, 4 | $C2/c$, 8 |
| a , Å | 12.711(3) | 30.556(3) |
| b , Å | 23.256(4) | 16.1407(16) |
| c , Å | 14.759(4) | 32.958(3) |
| β , deg | 104.99(2) | 116.646(2) |
| V , Å ³ | 4214.4(17) | 14528(2) |
| ρ_{calc} , g/cm ³ | 1.531 | 1.893 |
| μ_{Mo} , mm ⁻¹ | 4.043 | 9.187 |
| Crystal size, mm | 0.45 × 0.30 × 0.22 | 0.30 × 0.25 × 0.10 |
| θ range, deg | 1.75 < θ < 27.00 | 1.76 < θ < 24.00 |
| Number of reflections | | |
| measured | 11311 | 53017 |
| unique (N) [R_{int}] | 9190 [0.0347] | 11310 [0.1687] |
| observed ($I > 2\sigma(I)$) (N_o) | 6527 | 3889 |
| GOOF | 1.016 | 0.774 |
| $R1$, $wR2$ for N_o | 0.0617, 0.1570 | 0.0461, 0.0663 |
| $R1$, $wR2$ for N | 0.0918, 0.1833 | 0.1719, 0.0843 |
| $\Delta\rho_{max}/\Delta\rho_{min}$, e/Å ³ | 1.522/–2.715 | 1.432/–1.065 |

The coordinates of H atoms of Ph_3P molecules were calculated. All H atoms were refined using the rider model with isotropic thermal parameters as $U_j(H) = 1.2U_{eq}$ of the relevant non-hydrogen atom. The least-squares fits were carried out using software SHELXL97 [17]. Selected crystallographic data for compounds **I** and **II**, the parameters of the X-ray diffraction experiment, and structure refinement parameters are listed in Table 1; some bond lengths and bond angles are found in Table 2.

The crystallographic data for compound **I** have been deposited with the Cambridge Structural Database (CCDC No. 723647). Data for compound **II** are available from the Cambridge Structural Database (Refcode KIYZAU).

RESULTS AND DISCUSSION

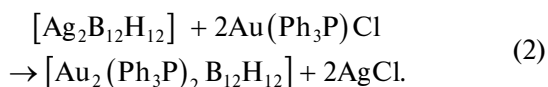
A method for preparing a gold(I) complex of the *closo*-hexaborate anion $[Au_2(Ph_3P)_2(B_6H_6)]$ by reac-

Table 2. Selected bond lengths (d) and bond angles (ω) in the structure of complex **I**

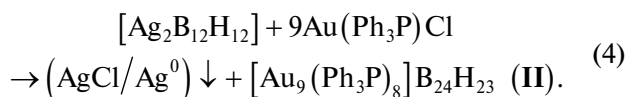
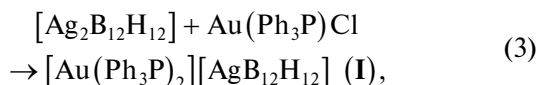
| Bond | d , Å |
|-------------------|----------------|
| Au(1)–P(1) | 2.338(2) |
| Au(1)–P(2) | 2.3405(19) |
| Ag(1)–H(12)#1 | 2.09 |
| Ag(1)–H(10)#1 | 2.29 |
| Ag(1)–H(9)#1 | 2.49 |
| Ag(1)–B(1) | 2.498(11) |
| Ag(1)–B(12)#1 | 2.528(11) |
| Ag(1)–B(10)#1 | 2.614(11) |
| Ag(1)–B(2) | 2.630(13) |
| Ag(1)–B(9)#1 | 2.738(11) |
| Ag(1)–H(1) | 1.95 |
| Ag(1)–H(2) | 2.21 |
| Angle | ω , deg |
| B(1)H(1)Ag(1) | 107 |
| B(2)H(2)Ag(1) | 100 |
| B(9)H(9)Ag(1)#2 | 91 |
| B(10)H(10)Ag(1)#2 | 94 |
| B(12)H(12)Ag(1)#2 | 100 |

Note: Atoms related to the initial atoms via symmetry transformations: #1 $x, -y + 3/2, z + 1/2$; #2 $x, -y + 3/2, z - 1/2$.

tion(1) using $(\text{Bu}_4\text{N})_2\text{B}_6\text{H}_6$ and $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ as the reagents is described in [6]. Our studies have shown that it is impossible to prepare a gold(I) complex of $\text{B}_{12}\text{H}_{12}^{2-}$ by a similar reaction of $(\text{Bu}_4\text{N})_2\text{B}_{12}\text{H}_{12}$ with $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ in acetonitrile, since an isothermal evaporation of the reaction mixture leads to the isolation of the least soluble compound ($\text{Au}(\text{Ph}_3\text{P})\text{Cl}$) in colorless crystals. Therefore, we chose $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ and $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ for preparing gold complexes of the *closo*-dodecaborate anion. The reaction scheme was presumed to be as follows:



AgCl formation was expected to remove silver from the reaction system and to shift the reaction toward a suggested gold(I) complex of the anion $\text{B}_{12}\text{H}_{12}^{2-}$. However, it appeared that reaction (2) did not occur, and depending on the reagent ratio, the reaction of $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ with $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ may be described as follows:



For the equimolar reagent ratio, AgCl was not formed, and the combination of physicochemical methods allowed us to identify the reaction product with compound **I** (Scheme 3). Elemental analysis showed both silver(I) and gold(I) atoms in complex **I**. In the IR spectrum of compound **I**, in the region of the boron–hydrogen stretches there are a strong band $\nu(\text{BH})$ due to “free” BH groups at 2475 cm^{-1} and two broadened medium-intensity bands (~ 2342 and 2150 cm^{-1}), which are associated with the stretching vibrations $\nu(\text{BH})_{\text{MHB}}$. A similar pattern is observed in the IR spectra of $\text{Cat}[\text{AgB}_{12}\text{H}_{12}]$ anionic complexes [4]. In the region where the vibrations of phenyl rings of triphenylphosphine molecules appear ($600\text{--}1600 \text{ cm}^{-1}$), the IR spectrum of complex **I** is an analogue of the spectrum of the precursor $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$.

Crystals of compound **I** were isolated from the reaction solution, and their structure was solved by X-ray crystallography. Complex **I** represents a coordination polymer built of $[\text{AgB}_{12}\text{H}_{12}]_n^{n-}$, anionic chains which are extended along axis c (Fig. 1a) and $[\text{Au}(\text{Ph}_3\text{P})_2]^+$ complex cations (Fig. 1b). The atom $\text{Ag}(1)$ is coordinated by the edge $\text{B}(1)\text{--}\text{B}(2)$ of one $\text{B}_{12}\text{H}_{12}^{2-}$ anion and the face $\text{B}(9)\text{B}(10)\text{B}(12)$ of the other anion. $\text{Ag}\text{--}\text{B}(\text{H})$ bond lengths with $\text{B}_{12}\text{H}_{12}^{2-}$ polyhedra are nonequivalent: $\text{Ag}(1)\text{--}\text{B}(1)$ ($2.498(11) \text{ \AA}$) is far shorter than $\text{Ag}(1)\text{--}\text{B}(2)$ bond lengths ($2.630(13) \text{ \AA}$), and $\text{Ag}(1)\text{--}\text{B}(12)$ ($2.528(11) \text{ \AA}$) is shorter than $\text{Ag}(1)\text{--}\text{B}(10)$ ($2.614(11) \text{ \AA}$) and $\text{Ag}(1)\text{--}\text{B}(9)$ ($2.738(11) \text{ \AA}$). The longest bond $\text{Ag}(1)\text{--}\text{B}(9)$ in Fig. 1a is indicated by a dashed line.

In the complex cation $[\text{Au}(\text{Ph}_3\text{P})_2]^+$, the atom $\text{Au}(1)$ has a nearly linear coordination: angle $\text{P}(1)\text{Au}(1)\text{P}(2)$ is $170.91(7)^\circ$; $\text{Au}(1)\text{--}\text{P}$ bond lengths are $2.338(2)$ and $2.341(2) \text{ \AA}$. Molecular stacking in a crystal in the projection along axis c is shown in Fig. 1c.

closo-Hydroborate anions, by virtue of their geometric specificity, can be coordinated to metal atoms by various edges and/or faces of their boron cages, giving rise to a great many position isomers, specifically mirror isomers [18]. In compound **I**, two enantiomers cocrystallize in a polymer chain: 1-2, 8-9-12 and 1-2, 9-10-12 isomers (Fig. 2). Compound **I** may be classified with $\text{Cat}[\text{AgB}_{12}\text{H}_{12}]$ complexes where $\text{Cat} = \text{Au}(\text{Ph}_3\text{P})_2^+$. As distinct from the $\text{Cat}[\text{AgB}_{12}\text{H}_{12}]$ complexes described in [3–5] (where $\text{Cat} = [(\text{NaphCH}_2)\text{Ph}_3\text{P}]^+$, NBzlEt_3^+), compound **I** is the first example where the anion $\text{B}_{12}\text{H}_{12}^{2-}$ is coordinated simultaneously by an edge to one silver(I) atom and by a face to another silver(I) atom.

The X-ray crystallography and IR spectroscopic data taken together imply that the coordination of

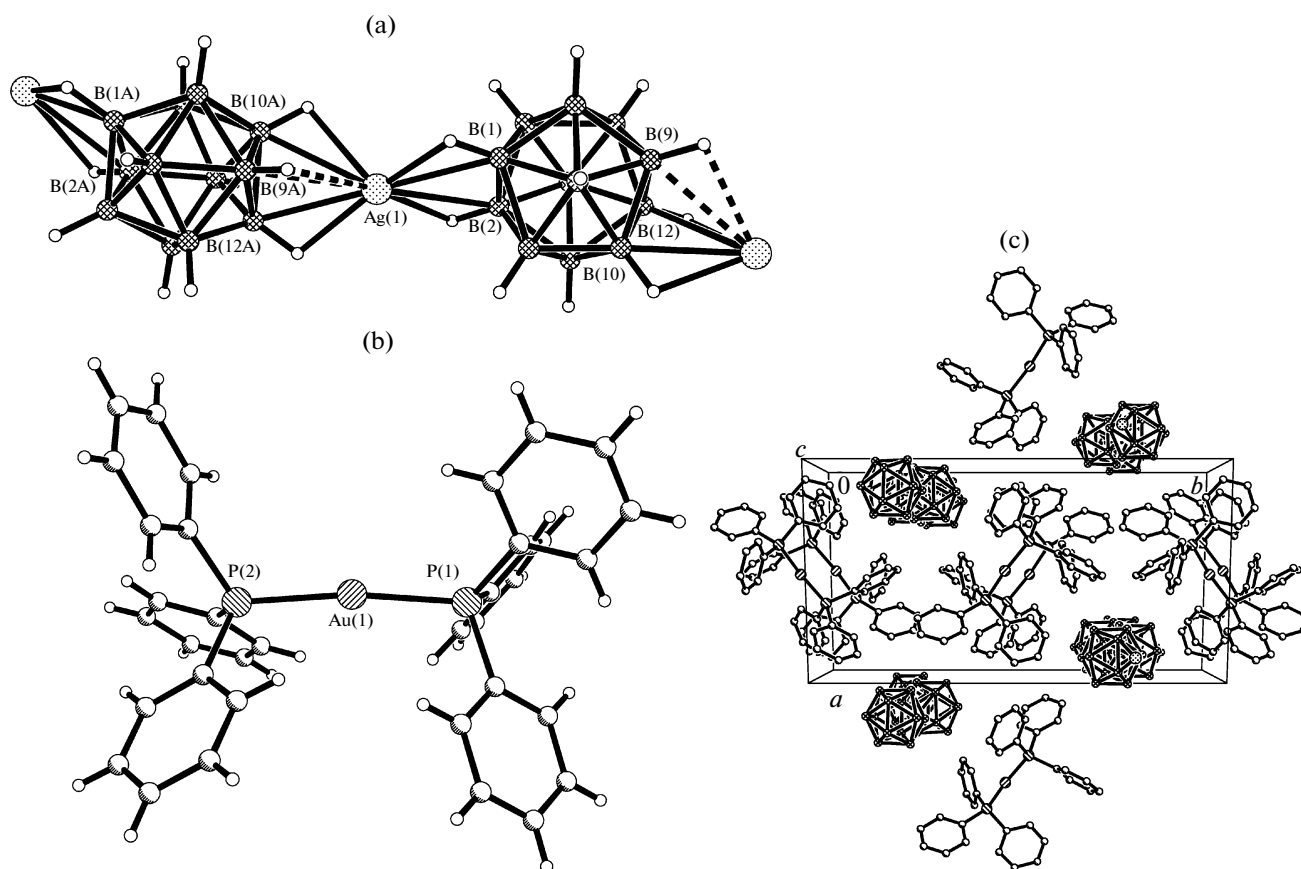


Fig. 1. Crystal structure of compound **I**: (a) a fragment of an $[\text{AgB}_{12}\text{H}_{12}]_n^-$ chain (the anionic part), (b) $[\text{Au}(\text{Ph}_3\text{P})_2]^+$ (the cationic part), and (c) projection along axis c (H atoms are omitted).

$\text{B}_{12}\text{H}_{12}^{2-}$ anions to Ag(I) atoms is via long and short three-center two-electron bonds (MHB). This is evidenced by the values of Ag–H(B) (1.95–2.49 Å) and Ag–B(H) (2.498(11)–2.738(11) Å) bond lengths and AgHB bond angles (90.8°–106.8°) (Table 2) and by the appearance in the IR spectrum of two bands arising from $\nu(\text{BH})_{\text{MBH}}$ and having peaks at 2342 cm^{-1} ($\Delta\nu = 133 \text{ cm}^{-1}$) and 2150 cm^{-1} ($\Delta\nu = 325 \text{ cm}^{-1}$), which refer to longer and shorter Ag–H(B) contacts, respectively.

The study of the behavior of the *closo*-dodecaborate anion in a greater excess of $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ with respect to $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ (where the ratio of $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ to $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ was 9 : 1) produced a radically different complex $[\text{Au}_9(\text{Ph}_3\text{P})_8]\text{B}_{24}\text{H}_{23}$ (**II**). In this case, precisely as implied by reaction (2), AgCl is produced to then enter a redox reaction where Ag^+ is reduced to Ag^0 and Au^+ to $\text{Au}^{0.33+}$ and where $\text{B}_{12}\text{H}_{12}^{2-}$ is oxidized to $\text{B}_{24}\text{H}_{23}^{3-}$ (Scheme 4). Among other reasons, the formation of degradation products of $\text{B}_{12}\text{H}_{12}^{2-}$ is responsible for the low yield of compound **II**.

Complex **II** contains a nonanuclear gold cluster (Fig. 3b), which constitutes the cationic part of the complex. The counterion (Fig. 3a) is the dimeric centrosymmetrical trianion $\text{B}_{24}\text{H}_{23}^{3-}$. B–H and B···B' distances in the B–H–B' bridge are 1.08 and 2.154 Å,

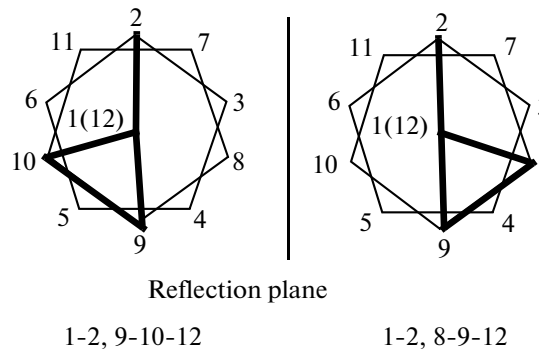


Fig. 2. Schematics of enantiomers 1-2, 8-9-12 and 1-2, 9-10-12 that cocrystallize in the crystal of complex **I** (top view).

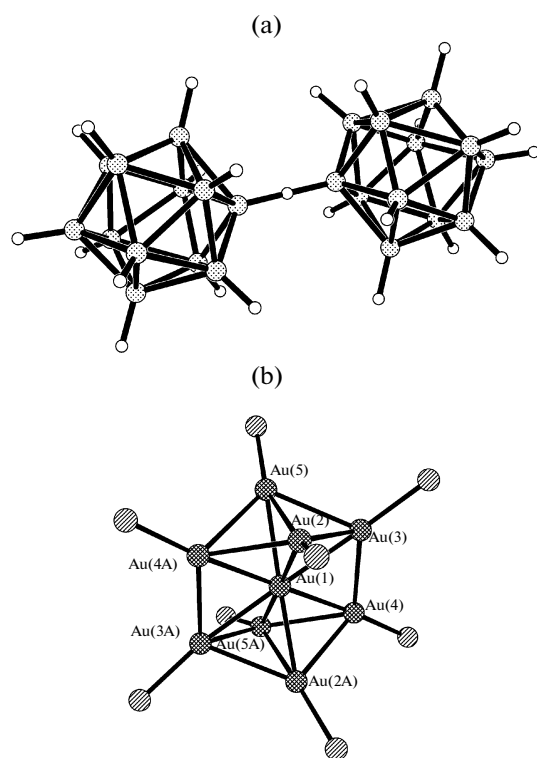


Fig. 3. Crystal structure of compound **II**: (a) $[\text{B}_{24}\text{H}_{23}]^{3-}$ (anionic part) and (b) $[\text{Au}_9(\text{Ph}_3\text{P})_8]^{3+}$ (cationic part).

respectively in match with theoretical calculations [19]. The existence of the trianion $\text{B}_{24}\text{H}_{23}^{3-}$ in complex **II** is unambiguously identified by ^{11}B NMR and IR spectroscopy [9].

Compounds of the cation $[\text{Au}_9(\text{Ph}_3\text{P})_8]^{3+}$ and anion $[\text{B}_{24}\text{H}_{23}]^{3-}$ with other counterions are known in the literature. The oxidation of $\text{B}_{12}\text{H}_{12}^{2-}$ to yield trianion $[\text{B}_{24}\text{H}_{23}]^{3-}$ was described as early as in 1969 [20]; $\text{Na}_3[\text{B}_{24}\text{H}_{23}]$ was obtained by electrolysis of a solution of $\text{Na}_2\text{B}_{12}\text{H}_{12}$ in acetonitrile at 1.5 V:



Based on the data of elemental analysis and IR and NMR spectroscopy, a link by a bridging hydrogen atom between the two $\text{B}_{12}\text{H}_{12}^{2-}$ icosahedra in $\text{B}_{24}\text{H}_{23}^{3-}$ was suggested [20].

Later [21], the X-ray diffraction structure of the trianion $[\text{B}_{24}\text{H}_{23}]^{3-}$ was determined. The angle BHB' between the icosahedra was 128.2° , whereas in complex **II**, the $\text{B}-\text{H}-\text{B}'$ bridge is strictly linear (in terms of symmetry).

Polynuclear gold clusters with triphenylphosphine and its derivatives are described in [22]. Nonanuclear gold clusters are mostly prepared by the reduction of mononuclear gold(I) complexes. Specifically, the reduction of $\text{Au}(\text{Ph}_3\text{P})\text{NO}_3$ by NaBH_4 produced the

complex $[\text{Au}_9(\text{Ph}_3\text{P})_8](\text{NO}_3)_3$ [23]. Other reducing agents are also useful for preparing $[\text{Au}_9(\text{Ph}_3\text{P})_8]^{3+}$, for example, the titanium complex $[\text{Ti}(\eta\text{-C}_5\text{H}_5\text{Me})_2]$ [24]. In our case, the reducing agent is likely to be the initial *closo*-dodecaborate anion $\text{B}_{12}\text{H}_{12}^{2-}$.

To summarize, we may say that having studied the behavior of the *closo*-dodecaborate anion $\text{B}_{12}\text{H}_{12}^{2-}$ in reaction with $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$, we found a number of features of the complexing process. We have found that $\text{Cat}_2\text{B}_{12}\text{H}_{12}$ salts of organic cations ($\text{Cat}^+ = \text{Ph}_4\text{P}^+$, Bu_4N^+) do not react with $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ in acetonitrile, whereas the use of $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ as the precursor *closo*-dodecaborate and variation of the reagent ratio produce fundamentally different products. The equimolar ratio of $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ to $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ yields a silver(I) complex that is the first example where the *closo*-dodecaborate anion is coordinated to silver(I) atoms in the face coordination. The reaction of $[\text{Ag}_2\text{B}_{12}\text{H}_{12}]$ with an excess of $\text{Au}(\text{Ph}_3\text{P})\text{Cl}$ generates ideal conditions for preparing $[\text{B}_{24}\text{H}_{23}]^{3-}$ and $[\text{Au}_9(\text{Ph}_3\text{P})_8]^{3+}$, whereas the preparation of the aforementioned cation and anion individually requires more severe conditions. Both complexes contain gold atoms, which are however not linked to the cluster boron anion.

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