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Synthesis of Halogen-Substituted [12]Mercuracarborands-4. Crystal Structure of $\{[(9,12\text{-}I_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]Cl\}Na(H_2O)_n$

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Abstract—The reactions of the dilithium derivatives of 9,12-dihalogen-*ortho*-carboranes 1,2-Li_2-$C_2B_{10}H_8$-9,12-X_2 (X = Cl, Br, I) with mercury chloride $HgCl_2$ afford a number of complexes of the chloride ion with the halogen derivatives of [12]mercuracarborand-4: $\{[(9,12\text{-}X_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]Cl\}Na\cdot nH_2O$. The molecular crystal structure of the complex of the [12]mercuracarborand-4 octaiodine derivative with the chloride ion is determined by X-ray diffraction. The substituents at the periphery of the mercury-containing macrocycle are found to exert a substantial effect on the macrocycle geometry leading to the transition from the planar to butterfly conformation, whose geometry is predetermined by a set of intermolecular interactions in the crystal.

Keywords: *ortho*-carborane, halogen derivatives, mercury anticrowns, chloride complexes, X-ray diffraction

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INTRODUCTION

Organomercury compounds were among the first organometallic compounds synthesized 170 years ago [1–4]. Interest in using them as pharmaceuticals [5] resulted in the synthesis of diverse organomercury compounds [6–8]. Although the organomercury compounds have found for a long time and still find use in the synthesis of other organometallic compounds [9], their application in organic synthesis is very restricted because of a low chemical activity toward organic substrates, which cannot be compared with that of much more reactive organolithium and Grignard reagents [10]. Recently increasing interest in this field of organoelement chemistry is related to the enhanced Lewis acidity of the organofluorine mercury compounds capable of forming complexes with various bases and halide ions and to a wide use of X-ray diffraction (XRD) studies aimed at determining their structures [11–14]. The top of this interest was the synthesis of anticrowns: macrocyclic mercury-containing polydentate Lewis acids capable of forming stable complexes with both anions and diverse Lewis bases [11, 15–19]. The high Lewis acidity of these macrocycles is achieved due to the use of perfluorinated aromatic (–*ortho*-C_6F_4–, –*ortho*-$C_6F_4C_6F_4$–) or aliphatic (–$C(CF_3)_2$–) fragments with a strong electron-acceptor effect as binding fragments of mercury atoms. Another variety of anticrowns is presented by mercury-containing macrocycles named mercuracarborands in which *ortho*-carboranyl groups –*ortho*-$C_2B_{10}H_{10}$– act as binding fragments [20]. Unlike the mercury macrocycles based on perfluorinated organic fragments, the Lewis acidity of mercuracarborands can be controlled by changing the electron-acceptor effect of the carboranyl group due to the substitution of hydrogen atoms [21].

The purpose of this work is to synthesize complexes of the chloride ion with halogen-substituted [12]mercuracarborands-4 containing halogen atoms in the positions opposite to the carbon atoms in the carborane framework.

EXPERIMENTAL

Halogen derivatives of *ortho*-carborane 9,12-Cl_2-1,2-$C_2B_{10}H_{10}$ [22], 9,12-Br_2-1,2-$C_2B_{10}H_{10}$ [23], and 9,12-I_2-1,2-$C_2B_{10}H_{10}$ [24] were synthesized according to published procedures. Diethyl ether was distilled over metallic sodium in the presence of benzophenone [25]. The reaction course was monitored by thin-layer chromatography (TLC) on the lgel 60 F245 plates (Merck) using a 0.5% solution of palladium(II) chloride in 1% hydrochloric aqueous methanol (1 : 10) as the developer. NMR spectra (400 MHz for ^1H and 128 MHz for ^{11}B) were recorded on a Varian Inova 400 spectrometer. Chemical shifts were presented relatively to Me_4Si (for ^1H NMR spectra) and $BF_3\cdot E_{t2}O$

(for ^{11}B NMR spectra). High resolution mass spectra (HRMS) were detected on a Bruker Daltonics micrOTOF II mass spectrometer in the range from 50 to 3000 *m/z*.

Synthesis of {[(9,12-Cl_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$]Cl}-Na(H_2O)$_n$ (I). A 2.5 M solution of BuLi in hexane (1.7 mL, 4.25 mmol) was added at 0°C in an argon atmosphere to a solution of 9,12-Cl_2-1,2-$C_2B_{10}H_{10}$ (426 mg, 2.00 mmol) in anhydrous diethyl ether (10 mL), and the mixture was stirred for 2 h. Then mercury(II) chloride (543 mg, 2.00 mmol) and anhydrous diethyl ether (5 mL) were added to the reaction mixture, and the resulting solution was stirred at room temperature overnight. The reaction mixture was treated with 6% hydrochloric acid (50 mL) and stirred for 2 h. The organic fraction was separated, and the aqueous fraction was washed with ethyl acetate (3 × 30 mL). The combined organic fractions were washed with an aqueous solution (3 × 30 mL) of sodium chloride (1.00 g) and sodium carbonate (1.00 g), dried over anhydrous sodium sulfate, and evaporated. *n*-Hexane was added to the formed oily residue, and the mixture was evaporated. The procedure was repeated until the full solidification of the residue. The formed beige powder was placed on the Schott filter and washed with chlorform and *n*-hexane until the signals of the initial 9,12-dichloro-*ortho*-carborane disappeared completely from the ^{1}H NMR spectrum. The yield of compound **I** as a white powder was 340 mg (40%). ^{1}H NMR (acetone-d_6, δ, ppm): 3.5–1.0 (32H, br.m, BH_{carb}). Spectral data: ^{11}B NMR (acetone-d_6, δ, ppm): 6.8 (8B, s, *B*(9,12)-Cl), –6.5 (8B, d, *J* = 134 Hz), –10.9 (24B, m). MS (ESI HRMS): found *m/z* 1682.2400 $[M + Cl]^-$; calculated for $C_8H_{32}B_{40}Cl_9Hg_4$ 1682.2429 $[M + Cl]^-$.

Synthesis of {[(9,12-Br_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$]Cl}-Na(H_2O)$_n$ (II). A 2.5 M solution of BuLi in hexane (1.7 mL, 4.25 mmol) was added at 0°C under an argon atmosphere to a solution of 9,12-Br_2-1,2-$C_2B_{10}H_{10}$ (604 mg, 2.00 mmol) in anhydrous diethyl ether (10 mL), and the mixture was stirred for 2 h. Then mercury(II) chloride (543 mg, 2.00 mmol) and anhydrous diethyl ether (5 mL) were added to the reaction mixture, which was stirred at room temperature overnight. The reaction mixture was treated with 50 mL of 6% hydrochloric acid and stirred for 2 h. The organic fraction was separated, and the aqueous fraction was washed with ethyl acetate (3 × 30 mL). The combined organic fractions were washed with an aqueous solution (3 × 30 mL) of sodium chloride (1.00 g) and sodium carbonate (1.00 g), dried over anhydrous sodium sulfate, and evaporated. *n*-Hexane was added to the formed oily residue, and the mixture was evaporated. The procedure was repeated until the full solidification of the residue. The formed beige powder was placed on the Schott filter and washed with chlorform and *n*-hexane until the signals of the initial 9,12-dibromo-*ortho*-carborane disappeared from the ^{1}H NMR spectrum. The yield of compound **II** as a white powder was 402 mg (39%). Spectral data: ^{1}H NMR (acetone-d_6, δ, ppm): 3.5–1.5 (32H, br.m, BH_{carb}). ^{11}B NMR (acetone-d_6, δ, ppm): 0.0 (8B, s, *B*(9,12)-Br), –5.9 (8B, m), –11.2 (24B, m). MS (ESI HRMS): found *m/z* 2037.8310 $[M + Cl]^-$; calculated for $C_8H_{32}B_{40}Br_8ClHg_4$ 2037.8363 $[M + Cl]^-$.

Synthesis of {[(9,12-I_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$]Cl}-Na(H_2O)$_n$ (III). A 2.5 M solution of BuLi in hexane (1.7 mL, 4.25 mmol) was added at 0°C under an argon atmosphere to 9,12-I_2-1,2-$C_2B_{10}H_{10}$ (792 mg, 2.00 mmol) in anhydrous diethyl ether (10 mL), and the mixture was stirred for 2 h. Then mercury(II) chloride (543 mg, 2.00 mmol) and anhydrous diethyl ether (5 mL) were added to the reaction mixture, which was stirred at room temperature overnight. The reaction mixture was treated with 6% hydrochloric acid (50 mL) and stirred for 2 h. The organic fraction was separated, and the aqueous fraction was washed with ethyl acetate (3 × 30 mL). The combined organic fractions were washed with an aqueous solution (3 × 30 mL) of sodium chloride (1.00 g) and sodium carbonate (1.00 g), dried over anhydrous sodium sulfate, and evaporated. *n*-Hexane was added to the formed oily residue, and the mixture was evaporated. The procedure was repeated until the full solidification of the residue. The formed beige powder was placed on the Schott filter and washed with chlorform and *n*-hexane until the signals of the initial 9,12-diiodo-*ortho*-carborane disappeared completely from the ^{1}H NMR spectrum. The yield of compound **III** as a white powder was 538 mg (44%). Spectral data: ^{1}H NMR (acetone-d_6, δ, ppm): 4.0–1.5 (32H, br.m, BH_{carb}). ^{11}B NMR (acetone-d_6, δ, ppm): –4.6 (8B, d), –9.1 (24B, m), –14.2 (8B, s, *B*(9,12)-I). MS (ESI HRMS): found *m/z* 2414.7338 $[M + Cl]^-$; calculated for $C_8H_{32}B_{40}ClHg_4I_8$ 2414.7327 $[M + Cl]^-$.

XRD. The crystals suitable for XRD analysis were prepared by the slow evaporation of a solution of compound **III** in acetone. Intensities of 153630 reflections were measured on a SMART APEX2 CCD diffractometer (λ(MoK_α) = 0.71073 Å, graphite monochromator, ω scan mode, 2θ < 52°). The initial set of measured intensities was processed using the SAINT and SADABS programs included into the APEX2 software [26]. The structure was solved by a direct method and refined by full-matrix least squares in the anisotropic approximation for non-hydrogen atoms for F^2_{hkl}.

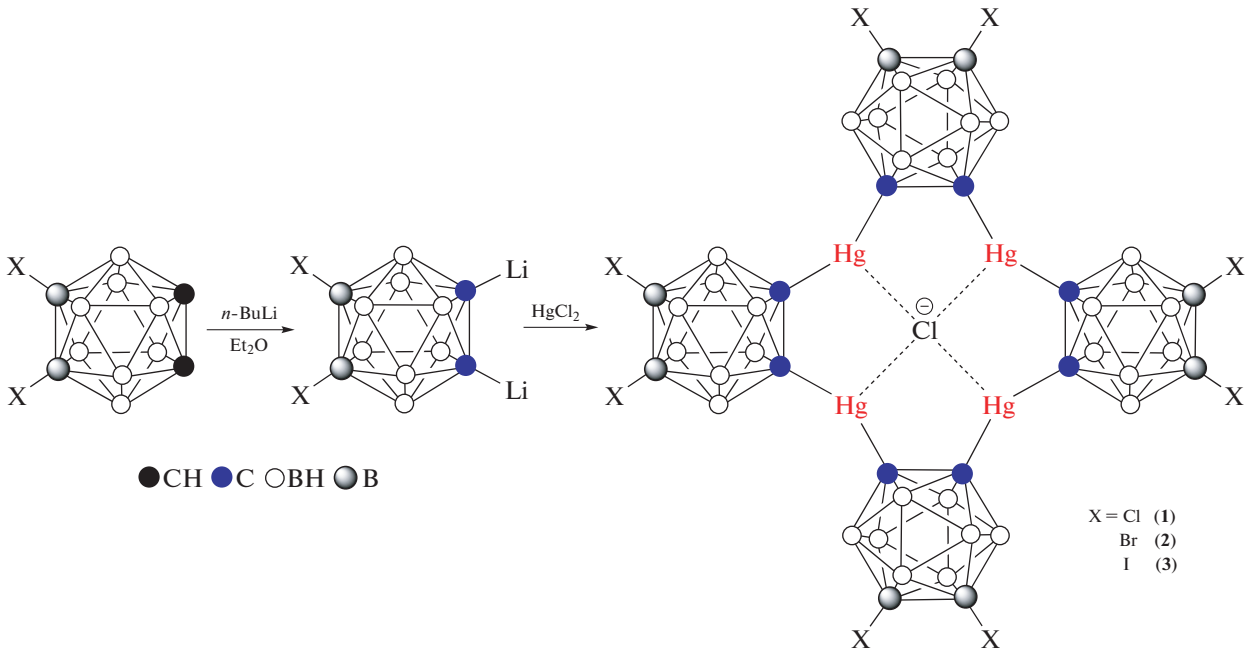
Hydrogen atoms were placed in the geometrically calculated positions and refined by the riding model using 6998 independent reflections (R_{int} = 0.0726). The number of refined parameters was 289. The refinement convergence for all independent reflections was wR_2 = 0.1058, GOOF = 1.208 (R_1 = 0.0535 for 6456 reflections with $I > 2\sigma(I)$). All calculations were performed using the SHELXTL software [27].

The crystals ($C_8H_{32}B_{40}Hg_4I_8Cl^-Na^+ \cdot 8H_2O$) at 100 K were orthorhombic with the parameters a = 18.5245(9), b = 13.8348(7), c = 27.7563(14) Å, V = 7113.5(6) Å^3, Z = 4, space group *Pbcn*, μ = 12.151 mm^{-1}, ρ_{calc} = 2.410 g/cm^{-3}.

The structure of compound **III** was deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 2310619).

RESULTS AND DISCUSSION

The [12]mercuracarborand-4 derivatives [(9,12-X_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$] (X = Cl, Br, I) were synthesized similarly to the unsubstituted tetramercury carborane macrocycle using template assembling by the reactions of the corresponding dilithium derivatives 1,2-Li_2-$C_2B_{10}H_8$-9,12-X_2 with mercury(II) chloride in diethyl ether and were isolated as complexes with the chloride ion {[(9,12-X_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$]Cl}$^-$ (X = Cl (**1**), Br (**2**), I (**3**)) (Scheme 1).



Scheme 1.

The structure of the complex of iodine-substituted [12]mercuracarborand-4 with the chloride ion {[(9,12-I_2-$C_2B_{10}H_8$-1,2'-Hg)$_4$]Cl}Na$(H_2O)_n$ (**III**) was determined by XRD.

Unlike the planar trimercury macrocycles (except for the macrocycle with perfluorinated biphenylene fragments having additional degrees of freedom because of a possibility of rotation of the aromatic rings around each other), the tetramercury macrocycles are conformationally nonrigid. For instance, a [12]mercuracarborand-4 molecule has the butterfly conformation characterized by the lowest deviations of the CHgC and CCHg angles from the ideal values (180° and 120°, respectively) and, most likely, is thermodynamically more favorable. The size of the inner cavity of the macrocycle is minimal, which favors complex formation with small anions, such as fluoride [28] or nitrate ion (coordinated via one oxygen atom) [29]. The macrocycle cavity becomes larger and more planar upon the interaction with larger anions. For example, the chloride ion is located nearly in the macrocycle plane to form a 1 : 1 complex, and larger bromide and iodide ions form 1 : 2 bipyrimidal complexes with the macrocycle. On the one hand, the influence of various substituents (I, Me, Et) in positions 9 and 12 of the *ortho*-carborane framework on the macrocycle geometry is minimal in this case [30–32]. On the

other hand, a significant distortion of the macrocycle geometry is observed upon complex formation with the chloride ion in [12]mercuracarborands-4 bearing phenyl substituents in the positions adjacent to the carbon atoms in the *ortho*-carborane framework, which is likely caused by steric interactions between the bulky substituents in the planar conformation of the macrocycle [33]. The formed conformation is stabilized by an overall influence of intermolecular interactions in the crystal. On the one hand, considered complex **III** contains no bulky substituents near the carbon atoms and, on the other hand, bears iodine atoms at the periphery capable of forming diverse intermolecular interactions, in particular, hydrogen bonds B–I···H–B and halogen bonds of I type [34–36]. The ability of the *ortho*-carborane iodine derivatives to form complexes with the tetramercury macrocycles due to B–I···Hg interactions is also known [30].

The $\{[(9,12\text{-}I_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]Cl\}^-$ complex exists in the partial position on the 2-fold symmetry axis along the crystallographic direction *b* and has the butterfly conformation with the φ angle between its "wings" equal to 109.3(8)° (Fig. 1). The C(1)C(2)Hg(2) and C(2)C(1)Hg(1) angles are 123.3(7)° and 122.0(7)°, respectively, and the C(1)Hg(1)C(1)' and C(2)Hg(2)C(2)' angles are 171.0(4)° and 169.4(4)°, respectively; i.e., the latter are rather close to ideal values, which is characteristic of the butterfly conformation. The Hg(1)–Cl(1) and Hg(2)–Cl(1) distances are 2.8468(4) and 2.7943(4) Å, respectively, which is comparable with the averaged distance in the nonplanar complex (2.824 Å) [33] and is appreciably shorter than that in the chloride complex of the parent [12]mercuracarborand-4 having the planar structure (2.944 Å) [30].

The crystal structure of complex **III** is characterized by sufficiently large cavities forming channels along the *c* axis. The cavity volume per unit cell is 1834 $Å^3$ (25%). The channels are filled with Na^+ cations and water molecules, the majority of which are strongly disordered. A part of the coordination sphere of the Na^+ cation is occupied by iodine atoms. A similar pattern is observed for the structure of $\{[(9,12\text{-}I_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]I_2\}Li_2(Me_2CO)_6{\cdot}4H_2O$ [31]. In addition to the Na···I contacts, the crystal contains very strong intermolecular B–I···Hg interactions with the I···Hg distances (3.5462(2) and 3.4154(2) Å, Fig. 2) comparable with the I···Hg distances in the complexes of [12]mercuracarborands-4 with the iodide anion [16]. Note that the formation of similar intermolecular I···M interactions is also characteristic of other types of the macrocyclic polydentate Lewis acids with halogen substituents at the heteromacrocycle periphery [37]. The crystal structure of compound **III** contains numerous B–I···H–B hydrogen bonds (I···H distance within 3.07–3.25Å) and I(12)...I(9)' halogen bond of I type (Fig. 2).

These sufficiently strong intermolecular interactions are likely responsible for the formation of a strong and stable three-dimensional framework containing cavities. It is reasonable to assume that they should predetermine, to a high extent, the conformation of the structurally nonrigid macrocycle as well.

Interestingly, unlike the complex with the chloride ion, no distortions of the plane of the mercury-containing macroheterocycle are observed in the previously described complex with the iodide ion $\{[(9,12\text{-}I_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]I_2\}^{2-}$ [31], which can be explained by the stabilizing effect of two iodide anions coordinated from the opposite sides of the macroheterocycle that do not prevent the distortion and also more efficiently neutralize its high Lewis acidity.

Thus, in this work we synthesized a series of complexes of the chloride ion with the [12]mercuracarborand-4 halogen derivatives and determined the molecular crystal structure of the complex with octaiodine [12]mercuracarborand-4 [12]. An analysis of the crystal structure shows that the substituents at the periphery of the mercury-containing macrocycle can exert a substantial effect on its geometry leading (in the case of the chloride complex) to the butterfly conformation, which is likely predetermined by a set of intermolecular interactions in the crystal.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

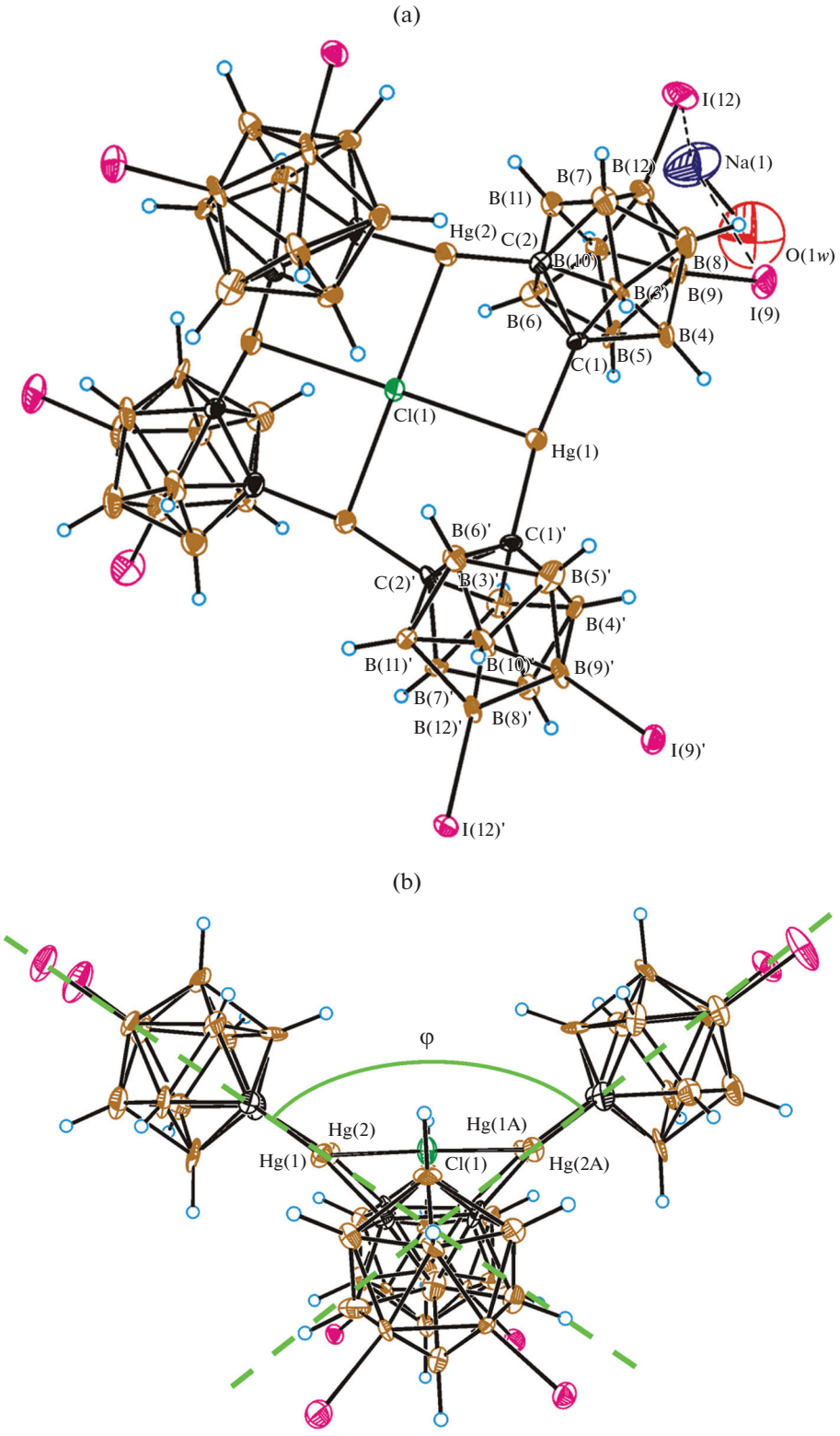

Fig. 1. General view of the $\{[(9,12\text{-}I_2\text{-}C_2B_{10}H_8\text{-}1,2'\text{-}Hg)_4]Cl\}^-$ complex in the representation of atoms by thermal vibration ellipsoids of 50% probability: (a) numeration only for the symmetrically independent part of the macrocycle and (b) side view of the macrocycle. The φ angle between the "butterfly wings" was determined as an angle between the planes of the *ortho*-carborane rings passed through the C(1), C(2), B(9), B(12), Hg(1), and Hg(2) atoms (shown by green dashed lines).

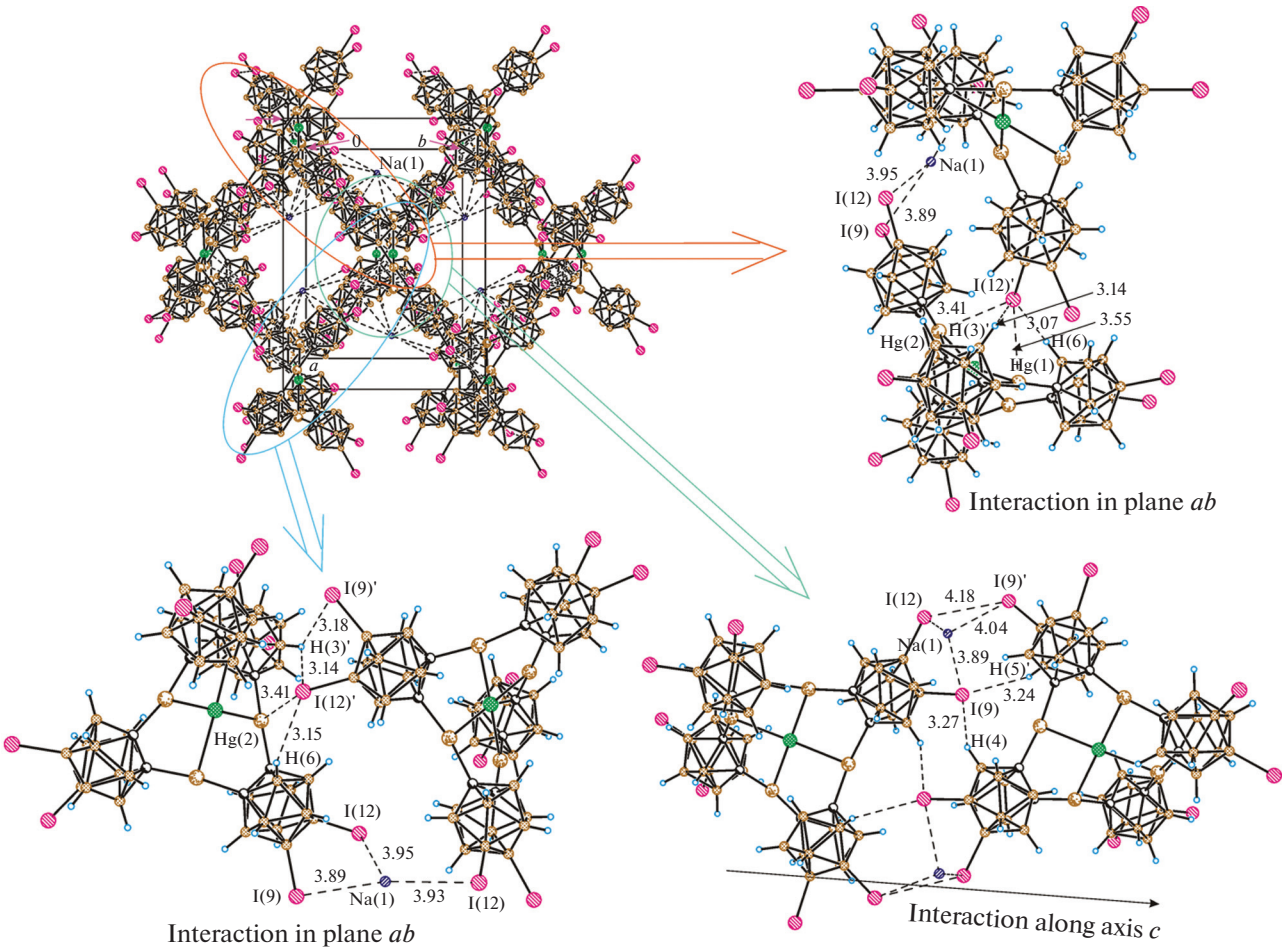

Fig. 2. At the upper left: the fragment of the crystalline packing of complex **III** (hydrogen atoms and water molecules are omitted). At the upper right and bottom: most strongly bound dimeric associates. Intermolecular contacts are shown by dash, and their distances are given in Å.

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