Synthesis of hetero-substituted derivatives of cobalt bis(1,2-dicarbollide)*

I. D. Kosenko, I. A. Lobanova, I. B. Sivaev, P. V. Petrovskii, and V. I. Bregadze

A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 ul. Vavilova, 119991 Moscow, Russian Federation. Fax: +7 (499) 135 6471. E-mail: kosenko@ineos.ac.ru

A new approach to synthesis of hetero-substituted derivatives of cobalt bis(1,2-dicarbollide) was proposed. The approach involves stepwise introduction of functional groups into different dicarbollide ligands. Halogenation of the monohydroxy derivative [8-OH-3,3′-Co-(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)]$^-$ gave the corresponding halogen hydroxy derivatives [8-OH-8′-X-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^-$ (X = Cl, Br, and I). Reactions of 8,8′-μ-iodonium-3-*commo*-cobaltbis(1,2-dicarba-*closo*-dodecaborate) [8,8′-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] with chloroform and 1,2-dibromoethane yielded the mixed halides [8-Y-8′-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^-$ (Y = Cl and Br).

Key words: cobalt bis(dicarbollide), halogen and hydroxy derivatives, 8,8′-μ-iodonium-3-*commo*-cobaltbis(1,2-dicarba-*closo*-dodecaborate), reactivity.

Derivatives of cobalt bis(1,2-dicarbollide) [3,3′-Co-(1,2-$C_2B_9H_{11}$)$_2$]$^-$ due to high stability and practically unlimited possibilities of their modification by the directed introduction of various substituents[1,2] can be used in such different areas as medicinal chemistry[3—6] and radioactive waste processing.[7,8] One promising area to use cobalt bis(1,2-dicarbollide) is preparation of new semiconducting materials on its base.[9,10]

Earlier, we have studied the effect of various substituents in dicarbollyl ligands (bromine,[11] iodine,[12—14] and hydroxy group[15]) on the structure and electroconducting properties of radical cation salts of cobalt bis(1,2-dicarbollide) with tetrathiafulvalene and its derivatives. We have found that the size of substituent and its ability to form intra- and intermolecular hydrogen bonds have determining impact on the crystal packing and electrophysical properties of the materials obtained. Thereby development of synthetic approaches to new derivatives of cobalt bis(1,2-dicarbollide) in which the carborane ligands bear different substituents becomes important.

The goal of this work was synthesis of hetero-substituted derivatives of cobalt bis(1,2-dicarbollide) [8-X-8′-Y-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^-$ containing halogen and (or) hydroxy substituents (X = Cl, Br, and I; Y = I and OH).

Results and Discussion

Because of the symmetrical structure of cobalt bis(1,2-dicarbollide), substitution reactions usually involve simultaneously both its carborane ligands; only few compounds with different substituents in the dicarbollide ligands are known. Earlier,[16—18] opening of the iodonium bridge in [8,8′-μ-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] under the action of Lewis bases in aromatic solvents has been reported to yield hetero-substituted products of the type [8-Z-8′-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^{n-}$, where Z is the Lewis base (n = 0) or aryl (n = 1). Formation of [8-Me-8′-HO-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^-$ as a by-product in cross-coupling reaction of [8,8′-I_2-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$]$^-$ was reported as well.[19]

An alternative approach involves an introduction of a second substituent into monosubstituted derivatives of cobalt bis(1,2-dicarbollide). Following this approach, here we halogenated the described earlier[20] monohydroxy derivative Cs[8-OH-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)]$^-$ (Cs[**1**]).

We found that Cs[**1**] reacts with *N*-chlorosuccinimide in refluxing THF for 6 h to give the chloro hydroxy derivative Cs[8-OH-8′-Cl-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] (Cs[**2**]) in 40% yield (Scheme 1).

Similarly, the bromination of Cs[**1**] with *N*-bromosuccinimide in boiling THF for 1 h followed by the treatment with aqueous solution of Me_3NHCl gave the bromo hydroxy derivative [Me_3NH][8-OH-8′-Br-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] ([Me_3NH]**3**) in 40% yield. The reaction of Cs[**1**] with bromine in refluxing methanol for 4 h followed by the treatment with aqueous solution of Me_4NBr produced the bromo hydroxy derivative [Me_4N]**3** in 29% yield (see Scheme 1).

The similar reaction of Cs[**1**] with iodine in refluxing methanol for 8 h gave the iodo hydroxy derivative [8-OH-8′-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] (**4**), which was isolated as

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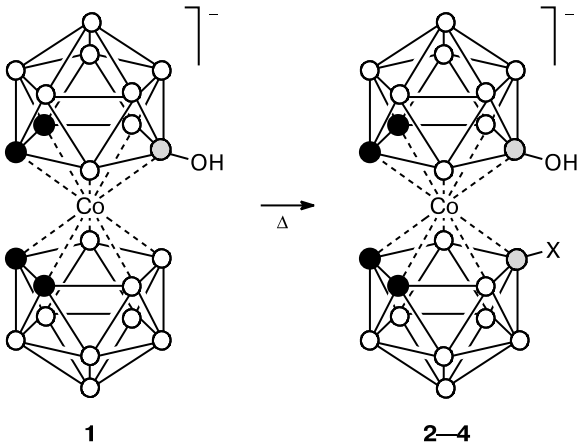
Scheme 1



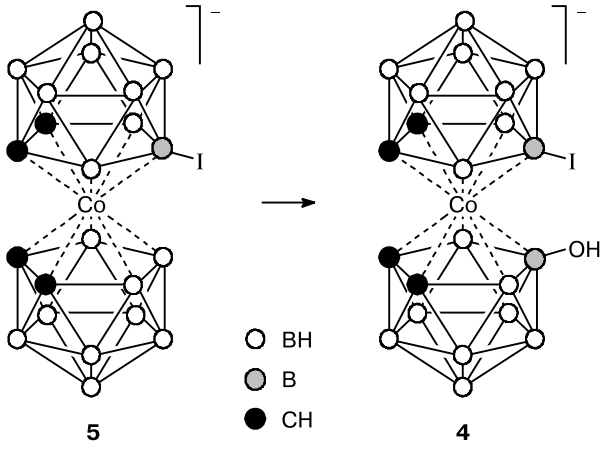
1 **2—4**

X = Cl (**2**), Br (**3**), I (**4**)

Reagents and products: *N*-chlorosuccinimide, **2**; *N*-bromosuccinimide or Br_2, **3**; I_2, **4**.

the tetramethylammonium salt $[Me_4N]$**4** in 25% yield (see Scheme 1). Alternatively, compound **4** was obtained by heating of the monoiodo derivative Cs[8-I-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (Cs[**5**])**21** with 60% H_2SO_4 at 110—120 °C for 10 h; the yield of the target product was 20% (Scheme 2). In contrast to the above mentioned reactions, in which the hydrogen atom at the B atom is replaced according to the electrophilic mechanism, this reaction follows the electrophile-induced nucleophilic substitution mechanism.[2]

Scheme 2



5 **4**

Conditions: 60% H_2SO_4, 120 °C.

The ^{1}H NMR spectra of anions **2** and **3** show two broadened signals at δ 4.15—4.31 (the CH groups of the carborane ligand) and a broad signal at δ 1.00—4.00 (B—H). The ^{11}B NMR spectrum of complex Cs[**2**] contains a set of signals consisting of two singlets for the B atoms bound to different non-hydrogen substituents and seven doublets for the B atoms bound to the hydrogen atoms. The ^{11}B NMR spectra of salts $[Me_3NH]$**3** and $[Me_4N]$**3** contain two singlets and eight doublets, respectively. The singlet at δ 22—23 corresponds to the B atom bound to the hydroxy group, while the singlets at δ 12.1 and 5.5 correspond to the B atom bound to the halogen atom (for anions **2** and **3**, respectively). The spectral characteristics of Cs[**4**] and $[Me_4N]$**4** are identical with those of the iodo hydroxy derivative Cs[8-OH-8′-I-3,3′-Co(1,2-$C_2B_9H_{10})_2$] obtained through opening of the iodonium bridge in 8,8′-μ-iodonium-3-*commo*-cobaltbis(1,2-dicarba-*closo*-dodecaborate) [8,8′-I-3,3′-Co(1,2-$C_2B_9H_{10})_2$] (**6**).[18]

Earlier, we demonstrated that reactions of complex **6** with arenes activate the C—H bonds in the latter to give the corresponding 8-aryl-8′-iodo derivatives.[18] It was supposed that these reactions involve opening of the iodonium bridge and formation of highly reactive quasiborinium cation, which is similar to the key intermediate in the electrophile-induced nucleophilic substitution mechanism.[2]

It is known that the *closo*-decaborate and *closo*-dodecaborate anions can be halogenated with neat haloalkanes in the presence of Brønsted[22—24] or Lewis acids[25] under electrophile-induced nucleophilic substitution conditions. We assumed that opening of the iodonium bridge in reactions of complex **6** with haloalkanes can also afford heterohalogen-containing products.

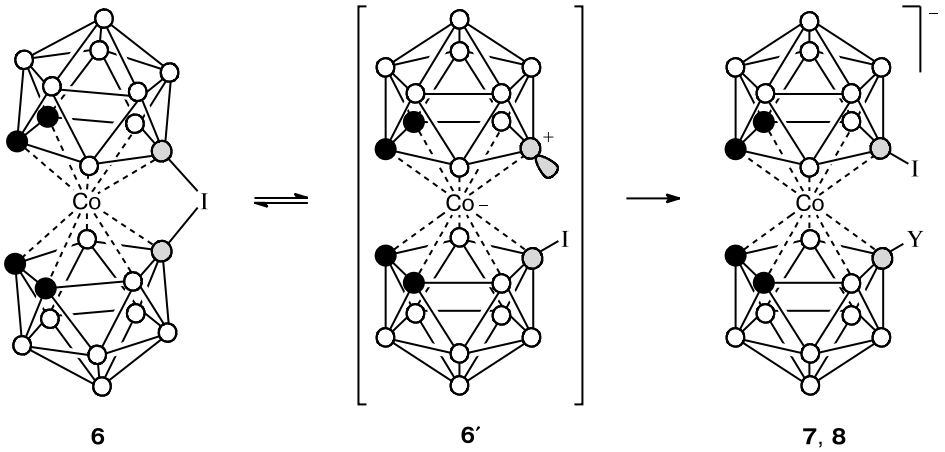
Indeed, a room-temperature reaction of complex **6** with chloroform for 30 h gave, through opening of the iodonium bridge, the chloro iodo derivative isolated as the salt Cs[8-Cl-8′-I-3,3′-Co(1,2-$C_2B_9H_{10})_2$] (Cs[**7**]) in 39% yield after the treatment with aqueous solution of CsCl (Scheme 3).

Similarly, the room-temperature reaction of complex **6** with 1,2-dibromoethane for 24 h also resulted in opening of the iodonium bridge, leading to a bromo iodo derivative, which was isolated as the cesium salt Cs[8-Br-8′-I-3,3′-Co(1,2-$C_2B_9H_{10})_2$] (Cs[**8**]) in 54% yield (see Scheme 3).

The compounds obtained are orange crystalline solids that are well soluble in Me_2CO, MeOH, EtOH, and MeCN, moderately soluble in CH_2Cl_2, $CHCl_3$, Et_2O, and benzene, and poorly soluble in water and hexane. All the compounds obtained are stable in air in the solid state but they decompose slowly in solutions.

To sum up, we developed a new synthetic approach to hetero-substituted derivatives of cobalt bis(1,2-dicarbollide) *via* stepwise introduction of functional groups into different dicarbollide ligands. Halogenation of the monohydroxy derivative of cobalt bis(1,2-dicarbollide) gave a series of its halogen hydroxy derivatives. Reactions of

Scheme 3



6 **6′** **7**, **8**

Reagent	Product	Y
$CHCl_3$	**7**	Cl
$Br(CH_2)Br$	**8**	Br

8,8′-μ-iodonium-3-*commo*-cobaltbis(1,2-dicarba-*closo*-dodecaborate) with halogen-containing solvents afforded for the first time mixed halogen derivatives of cobalt bis(1,2-dicarbollide).

Experimental

^1H and ^{11}B NMR spectra were recorded on a Bruker Avance 400 spectrometer (400.13 and 128.4 MHz) with reference to Me_4Si and $BF_3 \cdot Et_2O$, respectively. Mass spectra (ESI, negative ion detection) were measured on a micrOTOF Q II instrument (Bruker Daltonics). The course of the reactions was monitored by TLC on Kieselgel 60 F245 plates (Merck). The hydroxy derivative Cs[**1**], the iodo derivative Cs[**4**], and 8,8′-μ-iodonium-3-*commo*-cobaltbis(1,2-dicarba-*closo*-dodecaborate) were prepared according to known procedures.[20,21,16]

Cesium 8-chloro-8′-hydroxyicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate (Cs[2]). *N*-Chlorosuccinimide (0.27 g, 1.96 mmol) was added to a solution of Cs[8-OH-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (0.47 g, 0.99 mmol) in anhydrous THF (190 mL). The reaction mixture was refluxed with stirring for 6 h. On cooling, the precipitate formed was filtered off and the filtrate was evaporated to dryness. The residue was dissolved in acetone and then aqueous CsCl was added. The yellow precipitate formed was filtered off, dried in air, and chromatographed on silica gel with MeCN—CH_2Cl_2 (1 : 3) as eluent. The yield was 0.2 g (40%). ^1H NMR (acetone-d_6), δ: 4.31 (s, 2 H, CH_{carb}); 4.15 (s, 2 H, CH_{carb}). ^{11}B NMR (acetone-d_6), δ: 22.8 (s, 1 B, B(8)—OH); 12.1 (s, 1 B, B(8′)—Cl); −1.3 (d, 1 B, J = 146 Hz); −2.5 (d, 1 B, J = 148 Hz); −5.7 (m, 8 B); −19.3 (d, 2 B, J = 115 Hz); −20.1 (d, 2 B, J = 141 Hz); −25.9 (d, 1 B, J = 178 Hz); −27.7 (d, 1 B, J = 167 Hz). ESI MS (*m*/*z*): 377.4 [M], 343.4 [M − Cl].

Trimethylammonium 8-bromo-8′-hydroxyicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate ([Me_3NH][3]). *N*-Bromosuccinimide (0.59 g, 3.32 mmol) was added to a solution of Cs[8-OH-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (0.41 g, 0.86 mmol) in anhydrous THF (90 mL). The reaction mixture was refluxed for 1 h, cooled, and concentrated *in vacuo*. The oily residue was dissolved in a small amount of acetone and then aqueous Me_3NHCl was added. The resulting orange precipitate was filtered off, washed repeatedly with water, dried in air, and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 5) as an eluent. The yield was 0.19 g (40%).

Tetramethylammonium 8-bromo-8′-hydroxyicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate ([Me_4N][3]). Bromine (0.18 mL, 3.49 mmol) was carefully added dropwise to a solution of Cs[8-OH-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (0.3 g, 0.63 mmol) in anhydrous methanol (50 mL). The reaction mixture was refluxed with stirring for 4 h, cooled, and diluted with water (40 mL). The product was extracted with CH_2Cl_2 (3×15 mL). The organic layer was filtered through a short column with silica and concentrated. The oily residue was dissolved in acetone and treated with aqueous Me_4NBr. The resulting orange precipitate was filtered off and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 5) as eluent. The yield was 0.1 g (29%). ^1H NMR (acetone-d_6), δ: 4.31 (s, 2 H, CH_{carb}); 4.26 (s, 2 H, CH_{carb}); 3.44 (s, 12 H, $\underline{Me}_4N$). ^{11}B NMR (acetone-d_6), δ: 22.3 (s, 1 B, B(8)—OH); 5.5 (s, 1 B, B(8′)—Br); −0.8 (d, 1 B, J = 141 Hz); −1.9 (d, 1 B, J = 137 Hz); −5.6 (d, 4 B, J = 110 Hz); −6.2 (d, 4 B, J = 112 Hz); −18.9 (d, 2 B, J = 162 Hz); −20.2 (d, 2 B, J = 163 Hz); −25.0 (d, 1 B, J = 169 Hz); −27.7 (d, 1 B, J = 168 Hz). ESI MS (*m*/*z*): 421.4 [M].

Tetramethylammonium 8′-hydroxy-8-iodoicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate ([Me_4N][4]). Iodine (0.31 g, 1.22 mmol) was added to a solution of Cs[8-OH-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (0.34 g, 0.71 mmol) in anhydrous methanol (15 mL). The reaction mixture was refluxed with stirring for 8 h and cooled to room temperature. The precipitate formed was filtered off and the filtrate was evaporated to dryness. The residue was dissolved in acetone and treated

with aqueous $Na_2S_2O_3 \cdot 3H_2O$. The product was extracted with CH_2Cl_2 (3×30 mL) and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 3) as eluent. The eluate was concentrated, the residue was dissolved in acetone, and aqueous Me_4NBr was added. The resulting orange precipitate was filtered off and dried *in vacuo*. The yield was 0.11 g (25%).

Cesium 8′-hydroxy-8-iodoicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate (Cs[4]). The salt Cs[8-I-3,3′-Co(1,2-$C_2B_9H_{10}$)(1′,2′-$C_2B_9H_{11}$)] (0.7 g, 1.20 mmol) was added to 60% H_2SO_4 (105 mL). The reaction mixture was heated at 120 °C for 10 h, cooled, and diluted with water (100 mL). The product was extracted with CH_2Cl_2 (3×60 mL) and concentrated *in vacuo*. The oily residue was dissolved in acetone and treated with aqueous CsCl. The resulting yellow precipitate was filtered off and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 3) as eluent. The yield was 0.23 g (30%). The spectral characteristics of the complex [Cs][**4**] are identical with the literature data.[18]

Cesium 8′-chloro-8-iodoicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate (Cs[7]). A solution of [8,8′-μ-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] (0.15 g, 0.33 mmol) in chloroform (20 mL) was stirred at room temperature for 30 h. The reaction mixture was diluted with *n*-hexane (200 mL) and kept in a refrigerator for 16 h. The oily product that formed was dissolved in acetone and treated with aqueous CsCl. The resulting precipitate was filtered off and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 10) as eluent. The yield was 0.08 g (39%). ^{1}H NMR (acetone-d_6), δ: 4.42 (s, 2 H, CH_{carb}); 4.30 (s, 2 H, CH_{carb}). ^{11}B NMR (acetone-d_6), δ: 13.3 (s, 1 B, B(8′)—Cl); 2.1 (d, 1 B, J = 153 Hz); 1.2 (d, 1 B, J = 160 Hz); −3.3 (d, 2 B, J = 176 Hz); −4.1 (d, 6 B, J = 153 Hz); −5.9 (s, 1 B, B(8)—I); −17.2 (d, 2 B, J = 158 Hz); −18.5 (d, 2 B, J = 158 Hz); −22.6 (d, 1 B, J = 176 Hz); −24.8 (d, 1 B, J = 164 Hz). ESI MS (m/z): 484.1 [M$^-$].

Cesium 8′-bromo-8-iodoicosahydro-1,1′,2,2′-tetracarba-3-*commo*-cobalt-*closo*-tricosaborate (Cs[8]). A solution of [8,8′-μ-I-3,3′-Co(1,2-$C_2B_9H_{10}$)$_2$] (0.1 g, 0.22 mmol) in 1,2-dibromoethane (20 mL) was stirred at room temperature for 24 h. The reaction mixture was diluted with *n*-hexane (200 mL) and kept in a refrigerator for 16 h. The oily product that formed was dissolved in acetone and treated with aqueous CsCl. The precipitate formed was filtered off and purified by column chromatography on silica with MeCN—CH_2Cl_2 (1 : 5) as eluent. The yield was 0.08 g (54%). ^{1}H NMR (acetone-d_6), δ: 4.45 (s, 2 H, CH_{carb}); 4.34 (s, 2 H, CH_{carb}). ^{11}B NMR (acetone-d_6), δ: 7.4 (s, 1 B, B(8′)—Br); 2.4 (d, 1 B, J = 125 Hz); 1.7 (d, 1 B, J = 100 Hz); −3.9 (d, 8 B); −5.6 (s, 1 B, B(8)—I); −17.1 (d, 2 B, J = 137 Hz); −18.0 (d, 2 B, J = 135 Hz); −22.5 (d, 1 B, J = 182 Hz), −24.0 (d, 1 B, J = 180 Hz). ESI MS (m/z): 529.1 [M$^-$].

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