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# Synthesis and Deborination of Polyhalo-Substituted *ortho*-Carboranes

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**Abstract**—Tetraiodo-1,2-dicarba-*closo*-dodecaborane and 9-bromo-8,10,12-triiodo-1,2-dicarba-*closo*-dodecaborane were synthesized by oxidative iodination of 1,2-dicarba-*closo*-dodecaborane and 9-bromo-1,2-dicarba-*closo*-dodecaborane, respectively, in AcOH using a mixture of nitric and sulfuric acid as an oxidant of iodine. The intermediates in the *ortho*-carborane iodination were identified. By the action of elemental bromine on 9-iodo-1,2-dicarba-*closo*-dodecaborane in the presence of aluminum chloride catalyst 9-iodo-8,10,12-tribromo-1,2-dicarba-*closo*-dodecaborane was obtained. Deborination of the synthesized substances with an alcohol solution of KOH led to formation of 1,5,6,10-tetraiodo-5-bromo-1,6,10-triiodo- and 5-iodo-1,6,10-tribromo-7,8-dicarbaundecaborates methylammonium salts.

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Iodine derivatives of carboranes are valuable compounds in the reactions of cross-coupling with organomagnesium and organozinc compounds at the catalysis with palladium and nickel metallocomplexes [1]. This situation promotes research and development of new convenient methods of synthesis of the *ortho*-carborane iodo-derivatives.

The known methods of obtaining the majority of the *ortho*-carborane iodo-derivatives are the reactions of iodination in the presence of aluminum chloride [2–8], as well as deborination of *ortho*-carborane with subsequent addition of boron iodides [9]. Recent studies of the synthesis of the *ortho*-carborane iodo-derivatives belong to the synthesis of polyiodized compounds [10–17].

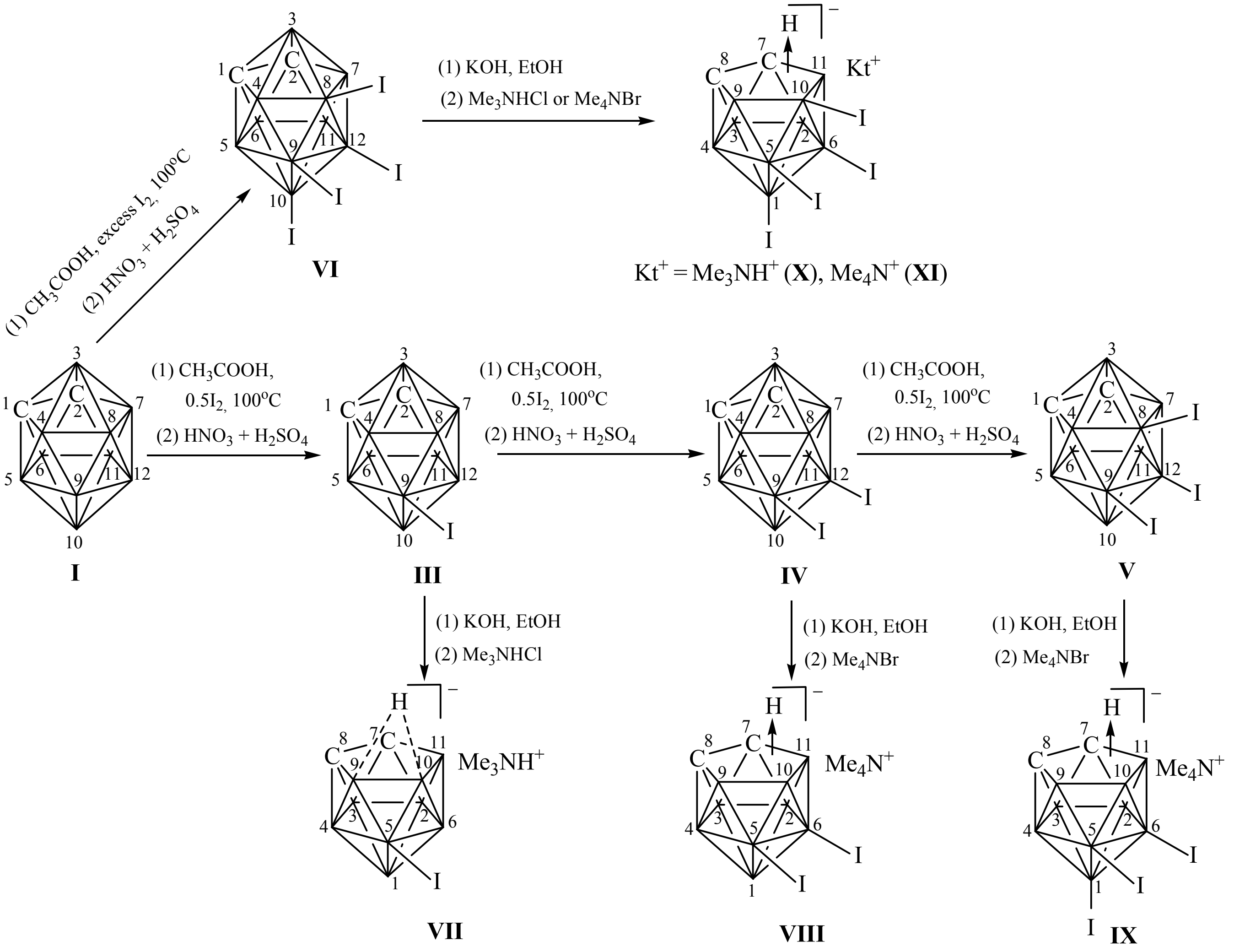
The purpose of this study was to develop an effective and convenient method of preparation of tetraiodo-, bromotriiodo- and iodotribromcarboranes and the products of their deborination, the potential reagents for cross-coupling reactions.

For iodination of *ortho*-carborane 1,2-$C_2B_{10}H_{12}$ (**I**) and 9-bromo-*ortho*-carborane 9-Br-1,2-$C_2B_{10}H_{11}$ (**II**) we chose the method of oxidative iodination that had been used previously for 9,11-diiodo-1,2-diphenyl-*ortho*-carborane 1,2-$Ph_2$-9,11-$I_2$-1,2-$C_2B_{10}H_8$ [18]. The universal solvent for this reaction is glacial acetic acid. It dissolves carborane and 9-bromocarborane, iodine, and also is mixing with mineral acids and has a sufficiently high boiling point. As an iodine oxidant a mixture of concentrated sulfuric and nitric acids was used. The process was carried out at 100°C, while without heating the iodination did not occur.

We found that the oxidative iodination of *ortho*-carborane can be carried out in stages, as a successive replacement of the 1, 2, 3 and 4 hydrogen atoms in BH fragments. Thus, under the action of one equivalent of iodine on the *ortho*-carborane **I** a monoiodo-derivative, 9-I-1,2-$C_2B_{10}H_{11}$ (**III**) is formed in 67% yield. The reaction of the obtained 9-iodcarborane **III** with iodine under similar conditions leads to a diiodo-derivative, 9,12-$I_2$-1,2-$C_2B_{10}H_{10}$ (**IV**), in 63% yield. The main product of the reaction of the latter compound with iodine is a triiodo-substituted carborane, 8,9,12-$I_3$-1,2-$C_2B_{10}H_9$ (**V**) (yield 64%). Using the methods of gas chromatography–mass spectrometry and NMR spectroscopy, we found that along with the main products **III** and **IV** formed the corresponding regioizomers: 8-I-1,2-$C_2B_{10}H_{11}$ (**IIIa**) (yield 10%) and 8,9-$I_2$-1,2-$C_2B_{10}H_{10}$ (**IVa**) (yield 18%). The synthesis of the triiodo-derivative is accompanied with the formation of diiodo-substituted carborane 9,12-$I_2$-1,2-$C_2B_{10}H_{10}$ (**IV**) (yield

9%) and teteraiodcarborane 8,9,10,12-$I_4$-1,2-$C_2B_{10}H_9$ (**VI**) (yield 11%). The synthesis of compounds **IV** and **V** directly from the *ortho*-carborane is hardly appropriate as a preparative method since a complex mixture of iodo-derivatives is formed, as follows from the data of gas chromatography–mass spectrometry. Thus, iodination of *ortho*-carborane **I** with 1 mole of iodine leads to a mixture consisting of monoiodized product **III** (26%) and two diiodized regioizomers, **IVa** (23%) and **IV** (51%), and the use of 1.5 mol of iodine under similar conditions gives a mixture of two diiodo- (5% of **IVa** + 62% of **IV**) and one triiodo-derivative (17% of **V**). The final product of the iodination of *ortho*-carborane with an excess of iodine is the individual tetraiodocarborane 8,9,10,12-$I_4$-1,2-$C_2B_{10}H_8$ (**VI**), formed in 91% yield.

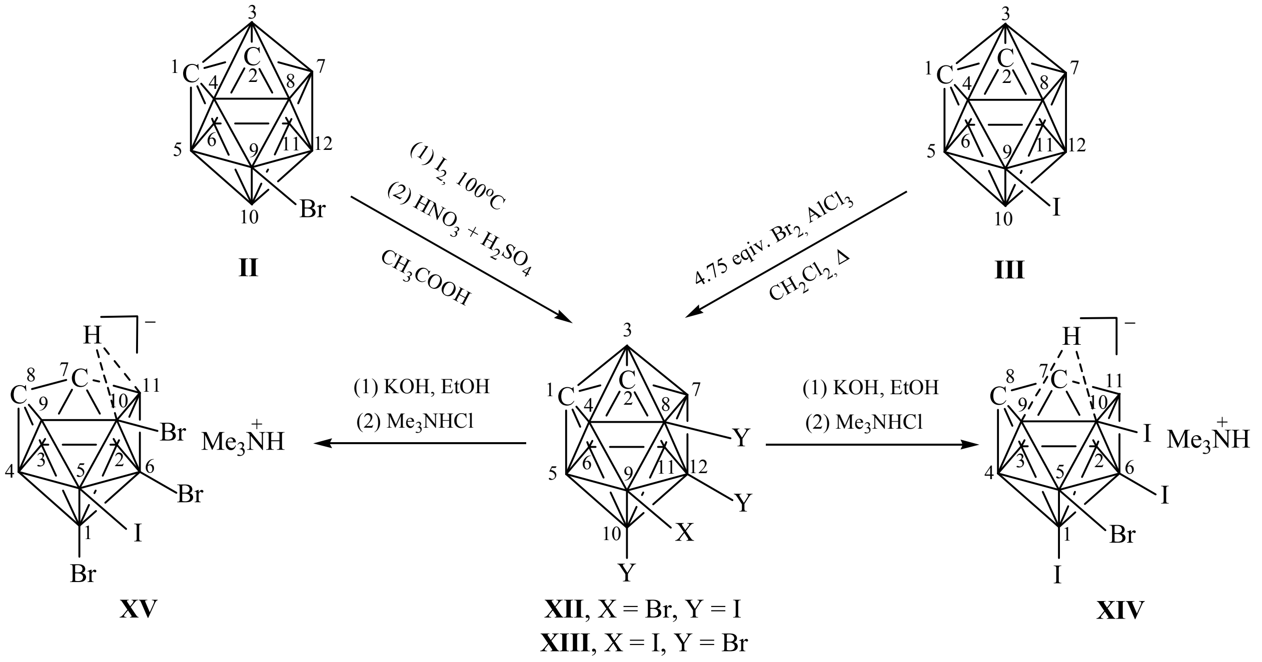
Deborination of the carborane iodo-derivatives **III**–**VI** with **a** solution of alkali in alcohol leads to the corresponding iodo-substituted dicarbaundecaborates which were isolated as the methylammonium salts $Me_3NH^+$ [5-I-1,2-$C_2B_{10}H_{11}$]$^-$ (**VII**) (yield 68%), $Me_4N^+$ [5,6-$I_2$-1,2-$C_2B_{10}H_{10}$]$^-$ (**VIII**) (yield 62%), $Me_4N^+$ [1,5,6-$I_3$-1,2-$C_2B_{10}H_9$]$^-$ (**IX**) (yield 96%) and $Kt^+$ [1,5,6,10-$I_4$-1,2-$C_2B_{10}H_8$]$^-$ [$Kt^+$ = $Me_3NH^+$ (**X**) (yield 74%), $Kt^+$ = $Me_4N^+$ (**XI**)]. In the case of 8,9,12-triiodo-1,2-dicarba-*closo*-dodecaborane (**V**) the deborination proceeds regioselectively at the atom $B^6$ that is the most distant from the core $B^8$–I. We found that the salts of tetraiodo-substituted dicarbaundecaborates **X** and **XI** that differ by the type of cation (trimethylammonium and tetramethylammonium, respectively) differ significantly in solubility, which can be used for their purification and isolation. Thus, compound **XI** is soluble in DMSO and insoluble in acetone, whereas substance **X** is readily soluble in both acetone and dimethylsulfoxide.



Compounds **III, IV, VI–VIII, X,** and **XI** were identified by comparing their physicochemical characteristics and IR and $^{11}B$ NMR spectra with the published data [5, 11, 12]. The structure of products **V** and **IX** was established on the basis of IR spectra, $^{1}H$, $^{11}B$, and $^{11}B–\{^{1}H\}$ NMR spectra, and $^{11}B–^{11}B$ COSY experiments. We found that the $^{11}B$ NMR spectrum of compound **X** dissolved in acetone-$d_6$ differs significantly from the spectrum of compound **XI** in DMSO-$d_6$, although both compounds formally differ only by the type of cation. Apparently, 7,8-dicarbaundecaborate exists in acetone-$d_6$ as a singly charged anion, while in DMSO-$d_6$ it is doubly charged, which is confirmed by the published data concerning related substances [11, 12, 19].

The $^{11}B$ NMR spectra of compounds **III–X** contain singlet signals of the molecular fragment B–I. In the $^{1}H$ NMR spectra of compounds **VII–X** the signals of terminal hydrogen groups $B^{10}–H$ are shifted consecutively downfield, which indicates an increase in electron-acceptor effect of iodine atoms with the increase in their number in the cluster, and decrease in the electron density in the open $C_2B_3$ pentagonal plane.

Under the same conditions as the iodination of *ortho*-carborane, we performed the iodination of 9-bromo-*ortho*-carborane, 9-Br-1,2-$C_2B_{10}H_{11}$ (**II**), and obtained a mixed bromotriiodo-derivative 9-Br-8,10,12-$I_3$-1,2-$C_2B_{10}H_8$ (**XII**), in 83% yield. By the action of bromine excess on 9-I-1,2-$C_2B_{10}H_{11}$ (**III**) in methylene chloride in the presence of aluminum chloride iodotribromocarborane 9-I-8,10,12-$Br_3$-1,2-$C_2B_{10}H_8$ (**XIII**) was synthesized in 92% yield.



The structure of compounds **XII** and **XIII** was established from IR, NMR, $^{11}B$, $^{11}B–\{^{1}H\}$, and $^{11}B–^{11}B$ COSY-spectra and gas chromatography–mass spectrometry. The mass spectra of the bromotriiodo- and iodotribromo-derivatives **XII** and **XIII** contained peaks of the corresponding molecular ions $[BrI_3C_2B_{10}H_8]^+$ ($m/z$ = 601) and $[IBr_3C_2B_{10}H_8]^+$ ($m/z$ = 507), with maximum intensity in the respective spectra.

We performed deborination of compounds **XII** and **XIII** by the action of excess alkaline alcohol, and isolated respective trimethylammonium salts of the singly charged clusters, $Me_3NH^+$ [5-Br-1,6,10-$I_3$-7,8-$C_2B_9H_8]^-$ (**XIV**) (yield 72%) and $Me_3NH^+$ [5-I-1,6,10-$Br_3$-7,8-$C_2B_9H_8]^-$ (**XV**) (yield 95%). The composition and structure of compounds **XIV** and **XV** was confirmed by the data of elemental analysis, IR, $^{1}H$ NMR, $^{11}B$, $^{11}B–\{^{1}H\}$, and $^{11}B–^{11}B$ COSY spectra.

In the $^{1}H$ NMR spectra of compounds **XIV** and **XV** a characteristic broadening is seen of the quadruplets at –0.12 and 0.16 ppm, respectively, of B(10)–H protons located over the open pentagonal plane of the $C_2B_3$ core. In the IR spectra of the tetrahalo-substituted compounds **X, XIV**, and **XV** a significant decrease occurs in the intensity of the band corresponding to the B–H stretching vibrations at 2500–2600 cm$^{-1}$ in comparison with the spectra of mono-, di-, and trihalo-substituted carboranes **III–V**. In the spectrum of compound **XI** two bands are observed at once, at 2562 and 2437 cm$^{-1}$. In the IR spectra of compounds **XIV** and **XV**, as in the spectra of undecaborates **X** and **XI**, there are the absorption bands in the region of 900–800 cm$^{-1}$ corresponding to vibrations of B–Br and B–I bonds.

The method of the oxidative iodination of *ortho*-carborane and 9-bromo-*ortho*-carborane we have developed is preferable compared to the known approaches to the iodination using elementary iodine in the presence of Lewis acids [2–8] and iodination in a sealed tube [12], because it can be easily scaled, reduces the process duration, and does not require an excess of iodine, which is especially important at the introducing iodine isotopes in the synthesis of carboranes for radiation diagnostics.

## EXPERIMENTAL

IR spectra were recorded on a Fourier spectrometer Protege-460, the samples were prepared as tablets with KBr. The $^{11}$B and $^{1}$H NMR spectra were recorded on an AVANCE-500 spectrometer from solutions of substances in acetone-*d*$_6$, for compound **XI** in DMSO-*d*$_6$. Chemical shifts are given relative to $Me_4Si$ for $^{1}$H signals and $Et_2O\cdot BF_3$ for $^{11}$B signals.

Chromato-mass spectra were obtained on an Agilent 6890N/5975 Inert instrument equipped with a capillary column HP-5MS (30 m×0.25 mm×0.25 mm). Carrier gas helium (0.8 ml min$^{-1}$), evaporator temperature 250°C. The program of the evaporator oven: 50°C (2 min), then raising the temperature to 300°C at a rate 20°C min$^{-1}$. Ionizing energy 70 eV.

**Synthesis of 9-I-1,2-$C_2B_{10}H_{11}$ (III)**. A solution of 0.5 g (3.47 mmol) of *ortho*-carborane 1,2-$C_2B_{10}H_{12}$ (**I**) and 0.44 g (1.74 mmol) of iodine in 90 ml of acetic acid was heated to 100°C, a mixture of concentrated sulfuric acid (20 ml) and nitric acid (20 ml) was added dropwise within 20–25 min at vigorous stirring, and the mixture was stirred until the disappearance of iodine color. Then the solution was poured into the ice water, the precipitate was filtered off, dissolved in acetone, reprecipitated by adding water, and dried in a vacuum over $CaCl_2$. A mixture (0.74 g) was obtained, containing, according to $^{1}$H NMR and $^{11}$B spectroscopy and gas chromatography–mass spectrometry, 9-I-1,2-$C_2B_{10}H_{11}$ (**III**) (85%) and 8-I-1,2-$C_2B_{10}H_{11}$ (**IIIa**) (13%). The total yield 79%. The $^{1}$H and $^{11}$B NMR spectra of the resulting products **III** and **IIIa** conform to the published data [11].

**Synthesis of 9,12-$I_2$-1,2-$C_2B_{10}H_{10}$ (IV)** was carried out in a similar way using a mixture of regioizomers of monoiodo-derivativas **III** and **IIIa**. A mixture of 8,9-$I_2$-1,2-$C_2B_{10}H_{10}$ (22%) (**IVa**) and 9,12-$I_2$-1,2-$C_2B_{10}H_{10}$ (**IV**) (78% ) was obtained. The total yield 81%. The $^{1}$H and $^{11}$B NMR spectra of the products **IV** and **IVa** coincided with the published data [5, 11, 12].

**Synthesis of 8,9,12-$I_3$-1,2-$C_2B_{10}H_9$ (V)** was performed in the same way as the iodination of the mixture of diiodo-*ortho*-carboranes **IV** and **IVa** obtained by the method described above. A mixture containing 9,12-$I_2$-1,2-$C_2B_{10}H_{10}$ (**IV**) (10%), 8,9,12-$I_3$-1,2-$C_2B_{10}H_9$ (**V**) (75% ), and 8,9,10,12-$I_4$-1,2-$C_2B_{10}H_9$ (**VI**) (13%) was obtained. The total yield 80%. The main product, 8,9,12-triiodo-*ortho*-carborane (**V**) was isolated and purified by fractional crystallization from a hexane–chloroform mixture, 5:2.

The IR spectrum of compound **V**, ν, cm$^{-1}$: 3040 s, 2924 w, 2852 w, 2617 m, 1196 s, 1130 s, 1075 w, 1056 w, 1025 w, 973 s, 947 w, 928 s, 889 s, 870 s, 850 vs, 826 w, 799 s, 767 s, 742 s, 626 m.

$^{1}$H NMR spectrum, δ, ppm: 5.33 s (2H, $2CH_{carbs}$), 3.7–2.1 m (7H, 7BH). $^{11}$B NMR, δ, ppm: –6.0 d (1B, B-10, $J_{BH}$ 156 Hz), –11.2 d (2B, B-4, B-7, $J_{BH}$ 180 Hz), –11.7 s (2B, B-9, B-12), –12.4 d (2B, B-5, C-11, $J_{BH}$ 188 Hz), –13.6 d (1B, B-3, $J_{BH}$ 182 Hz), –15.3 d (1B, B-6, $J_{BH}$ 201 Hz), –17.1 s (1B, B-8).

**Synthesis of 8,9,10,12-$I_4$-1,2-$C_2B_{10}H_8$ (VI)**. To a solution containing 1.0 g (6.9 mmol) of *ortho*-carborane 1,2-$C_2B_{10}H_{12}$ (**I**) and 3.5 g (13.8 mmol) of iodine in 90 ml of glacial acetic acid heated to 100°C was added dropwise at vigorous stirring within 30–45 min a mixture of concentrated sulfuric (45 ml) and nitric (45 ml) acids. The reaction mixture was stirred for 10 min and poured into ice water, the precipitate was filtered off, dissolved in acetone, excess iodine was neutralized with aqueous solution of sodium sulfite. The solution was evaporated to 1/4 of the original volume, the precipitate was separated, washed with water (3×15 ml), dissolved in 90 ml of glacial acetic acid and 1.5 g (5.9 mmol) of iodine was added to it, and the resulting mixture was heated to 100°C. Next, at vigorous stirring a mixture of concentrated sulfuric (45 ml) and nitric (45 ml) acids was added dropwise in 30–45 min, and then the solution was stirred for another 10 min and poured into ice water. The precipitate was filtered off, dissolved in acetone, excess iodine was neutralized with aqueous solution of sodium sulfite. The solution was evaporated to 1/4 of the original volume, the precipitate was filtered off, washed with water (3×15 ml), and dried over $CaCl_2$. 4.07 g (6.28 mmol) of 8,9,10,12-$I_4$-1,2-$C_2B_{10}H_8$ was obtained, yield 91%. The $^1H$ and $^{11}B$ NMR spectral data of the obtained compound **VI** coincided with the published ones [12].

**Synthesis of 9-Br-8,10,12-$I_3$-1,2-$C_2B_{10}H_8$ (XII).** To a solution containing 0.63 g (2.83 mmol) of 9-Br-1,2-$C_2B_{10}H_{11}$ and 3.0 g (11.81 mmol) of iodine in 90 ml of glacial acetic acid, heated to 100°C, at stirring a mixture of concentrated sulfuric (45 ml) and nitric (45 ml) acids was added dropwise over 40 min. The reaction mixture was stirred for 10 min and poured into ice water. The precipitate was filtered off, dissolved in acetone, excess iodine was neutralized with aqueous solution of sodium sulfite. The solution was evaporated to 1/4 of the original volume, the precipitate was filtered off and washed with water (3×15 ml). The resulting product was purified by reprecipitation with water from acetone and then by hexane from ether. 1.42 g (2.36 mmol) of 9-Br-8,10,12-$I_3$-1,2-$C_2B_{10}H_8$ (**XII**) was obtained, yield 83%.

IR spectrum, ν, $cm^{-1}$: 3031 vs, 2970 w, 2925 w, 2839 w, 2624 s, 1382 m, 1199 s, 1132 m, 1103 m 1076 m 1019 m, 981 mr, 962 s, 943 s, 918 w, 882 vs, 821 w, 842 vs, 821 w, 795 s, 763 m, 742 s, 632 m, 616 m.

$^1H$ NMR spectrum, δ, ppm: 5.48 s (1H, $C_{carb}H$), 5.29 (1H, $C_{carb}H$), 3.31–2.24 m (6H, 6BH).

$^{11}B$ NMR spectrum, δ, ppm: 2.1 s (1B, B-9), –9.5 s (1B, B-12), –12.0 d (4B, B-4, B-5, B-7, B-11, $J_{BH}$ 165 Hz), –15.8 d (2B, B-3, B-6, $J_{BH}$ 187 Hz), –18.9 s (2B, B-8, B-10).

**Synthesis of 9-I-8,10,12-$Br_3$-1,2-$C_2B_{10}H_8$ (XIII).** To a solution of 1.65 g (6.1 mmol) of 9-I-1,2-$C_2B_{10}H_{11}$ in 30 ml of methylene chloride containing 0.5 g $AlCl_3$ was added 1.5 ml (4.65 g, 29.0 mmol) of bromine, and the mixture was refluxed for 3.5 h. Then the solution was evaporated to dryness, to the residue water was added to neutralize $AlCl_3$, the product was extracted with methylene chloride and dried over $CaCl_2$. After removal of the solvent 2.85 g (5.62 mmol) of compound 9-I-8,10,12-$Br_3$-1,2-$C_2B_{10}H_8$ (**XIII**) was obtained, yield 92%.

IR spectrum, ν, $cm^{-1}$: 3040 s, 2924 w, 2852 w, 2617 m, 1196 s, 1130 s, 1075 w, 1056 w, 1025 w, 973 s, 947 w, 928 s, 889 s, 870 s, 850 vs, 826 w, 799 s, 767 s, 742 s, 626 m.

$^1H$ NMR spectrum, δ, ppm: 5.22 s (1H, $CH_{carb}$), 5.08 s (1H, $CH_{carb}$), 3.8–1.8 m (6H, 6BH).

$^{11}B$ NMR spectrum, δ, ppm: 1.1 (1B, B-12-Br), –5.5 s (2B, B-8-Br, B-10-Br), –10.9 s (1B, B-9-I), –13.7 d (4B, B-4, B-5, B-7, B-11, $J_{BH}$ 166 Hz), –18.9 d (2B, B-3, B-6, $J_{BH}$ 184 Hz).

**Synthesis of $Me_4N^+$ [1,5,6-$I_3$-7,8-$C_2B_9H_9$]$^-$ (IX).** To a solution of 1.56 g (3.0 mmol) of 8,9,12-$I_3$-1,2-$C_2B_{10}H_9$ **V** in 10 ml of anhydrous ethanol was added 0.35 g (6.0 mmol) of potassium hydroxide and the mixture was refluxed while stirring for 3.5 h, after which the reaction mixture was evaporated to dryness and to it water was added. Unreacted triiodide (**IX**) was filtered off. To the filtrate was added 0.62 g (4 mmol) of tetramethylammonium bromide in 10 ml of water. The precipitate was filtered off, dissolved in acetone, the solution was filtered, the filtrate was evaporated to dryness, washed with water (3 × 10 ml) and dried over $CaCl_2$. 1.26 g of $Me_4N^+$[1,5,6-$I_3$-7,8-$C_2B_9H_9$]$^-$ (**IX**) was isolated. Yield 96%.

IR spectrum, ν, $cm^{-1}$: 3049 w, 3023 w, 2947 w, 2913 w, 1479 OS, 1447 w, 1415 w, 1287 w, 1253 w, 1172 m, 1082 s, 1018, 984 w, 948 a, 927 s, 858 s, 825 s, 812 s, 760 s, 712 w, 635 w, 603 w.

$^1H$ NMR spectrum, δ, ppm: 3.43 s (12H, $Me_4N$), 3.0–0.8 m (8H, 8B-H), 2.31 s (2H, $2CH_{carb}$), –1.62 d (1H, B-10-H, $J_{BH}$ 62 Hz).

$^{11}B$ NMR spectrum, δ, ppm: –8.9 d (2B, B-9, B-11, $J_{BH}$ 148 Hz), –15.3 d (1B, B-3, $J_{BH}$ 181 Hz), –18.5 d (2B, B-2, B-4, $J_{BH}$ 164 Hz), –20.7 s (2B, B-5, B-6), d.d –26.8 (1B, B-10, $J_{BH}$ 139, $J_{BH}$ 32 Hz), –35.1 s (1B, B-1).

Found, %: C 12.66; H 4.11; B 16.76; I 65.03; N 2.57. $C_6H_{21}B_9I_3N$. Calculated, %: C 12.31; H 3.62; B 16.63; I 65.05; N 2.39

**Synthesis of $Me_3NH^+$[5-I-7,8-$C_2B_9H_{11}$]$^-$ (VII), $Me_4N^+$ [5,6-$I_2$-7,8-$C_2B_9H_{10}$]$^-$ (VIII), $Me_3NH^+$ [1,5,6,10-$I_4$-7,8-$C_2B_9H_8$]$^-$ (X), $Me_4N^+$ [1,5,6,10-$I_4$-7,8-$C_2B_9H_8$]$^-$ (XI), (XIV) and (XV)** was carried out similarly to carboranes **III, IV, VI, XII,** and **XIII**, respectively.

**Compound XIV.** IR spectrum, ν, $cm^{-1}$: 3122 s, 3042 w, 2957 w, 2923 w, 2853 w, 2731 w, 2561 s, 1475 s, 1462 vs, 1448 s, 1411 m, 1372 m, 1247 m, 1168 w, 1079 m, 1042 w, 1013 m, 971 s, 926 s, 897 w, 879 w, 860 m, 818 s, 757 m, 642 w, 621 w, 579 w.

$^1$H NMR spectrum, δ, ppm: 3.14 s (12H, $Me_4N$), 2.44 s (1H, $CH_{carb}$), 2.31 s (1H, $CH_{carb}$), 3.0–1.3 m (5H, 5BH), –0.12 br.q (1B, B-10-H, $J_{BH}$ 75 Hz, 179 Hz).

$^{11}$B NMR spectrum, δ, ppm: –7.0 s (1B, B-5-Br), –9.7 d (2B, B-9, B-11, $J_{BH}$ 146 Hz), –17.0 d (1B, B-3, $J_{BH}$ 193 Hz), –18.4 d (1B, B-2, $J_{BH}$ 193 Hz), –19.5 q+s (2B, B-4, B-6-I, $J_{BH}$ 159 Hz), –36.0 q+s (1B, B-10-H + B-10-I, $J_{BH}$ 89 Hz), –36.5 s (1B, B-1).

Found, %: C 9.69; H 2.95; B 14.57; Hlg 70.55; N 2.31. $C_5H_{18}B_9I_3BrN$. Calculated, %: C 9.24; H 2.79; B 14.97; Hlg 70.85; N 2.15.

**Compound XV.** IR spectrum, ν, cm$^{-1}$: 3136 s, 3045 w, 2959 w, 2924 w, 2853 w, 2786 w, 2752 w, 2541 s, 1477 vs, 1465 a, 1451 vs, 1414 m 1372 m 1252 m, 1174 w, 1086 s, 1045 w, 1019 s, 973 vs, 953 w, 930 s, 900 w, 874 s, 824 vs, 759 m, 650 w, 630 w, 609 w, 579 w, 533 w.

$^1$H NMR spectrum, δ, ppm: 3.16 s (5H, $Me_3NH$), 3.15 s (4H, $Me_3NH$), 2.35 s (1H, $CH_{carb}$), 2.26 s (1H, $CH_{carb}$), 2.8–0.6 m (5H, 5BH), 0.16 br.q (1H, B-10-H, $J_{BH}$ 74 Hz, 178 Hz).

$^{11}$B NMR spectrum, δ, ppm: –7.4 s (1B, B-6-Br), –10.7 d (2B, B-9, B-11, $J_{BH}$ 147 Hz), –20.0 d + s (3B, B-3, B-4 + B-5-I, $J_{BH}$ 155 Hz), –21.0 d (1B, B-2, $J_{BH}$ 143 Hz), –23.7 s (1B, B-1-Br ), –24.3 q + s (1B, B-10-H + B-10-Br, $J_{BH}$ 51 Hz).

Found, %: C 11.26; H 3.03; B 17.62; Hlg 65.50; N 2.51. $C_5H_{18}B_9Br_3IN$. Calculated, %: C 10.80; H 3.26; B 17.50; Hlg 65.92; N 2.52.

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