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One-Pot Synthesis of Borolanes by Reaction of Aluminacyclopentanes with $BF_3 \cdot Et_2O$

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Abstract—A selective procedure was developed for the synthesis of substituted borolanes via transmetalation with $BF_3 \cdot Et_2O$ of 3-alkyl-1-ethylaluminacyclopentanes obtained from the corresponding terminal olefins and $AlEt_3$ in the presence of Cp_2ZrCl_2 as a catalyst. 3-Substituted 1-fluoroborolanes were isolated and identified as 1:1 complexes with $EtBF_2$.

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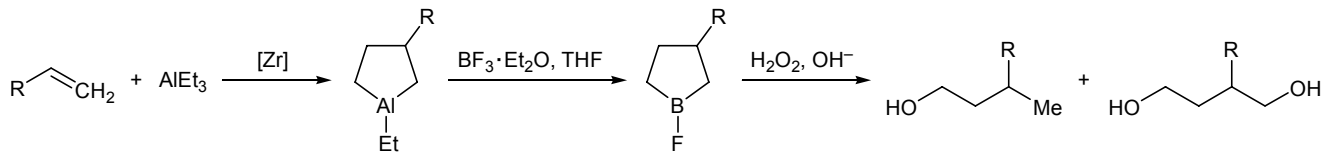
An efficient procedure for the synthesis of cyclic organoboron compounds is based on the reaction of cyclic or α,ω-bis-metalated organometallic compounds containing both transition and non-transition metals (Li, Mg, Sn, Hg, Zr, Ti) with an equimolar amount of boron halide [1–11]. Prior to our studies we have found no published data on the synthesis of boracycloalkanes with the use of organoaluminum compounds. On the other hand, successful exchange reactions between trialkylalanes [$AlEt_3$, $Al(i\text{-}Bu)_3$] or alkylhaloalanes ($AlEt_2Cl$, $AlEtCl_2$) and $BF_3 \cdot Et_2O$ or BCl_3 were reported [12, 13].

With a view to develop an efficient procedure for the synthesis of borolanes (boracyclopentanes) and elucidate the possibility for transmetalation of five-membered cyclic organoaluminum compounds with boron halides we examined reactions of 1,3-substituted aluminacyclopentanes with $BF_3 \cdot Et_2O$ as transmetalating agent [12]. We presumed that 3-substituted 1-ethylaluminacyclopentanes **I** obtained by catalytic cycloalumination of olefins with $AlEt_3$ in the presence of Cp_2ZrCl_2 [14, 15] could react *in situ* with $BF_3 \cdot Et_2O$ to produce the corresponding 3-alkyl-1-fluoroborolanes **II** (Scheme 1). As model reaction we selected the reaction of 1-ethyl-3-hexylaluminacyclopentane (**Ib**) generated *in situ* from oct-1-ene and $AlEt_3$ in hexane in the presence of 5 mol % of Cp_2ZrCl_2 [14] with an equimolar amount (with respect to $AlEt_3$) of $BF_3 \cdot Et_2O$. After removal of the solvent from the reaction mixture in a stream of argon, the residue was distilled under reduced pressure to isolate a colorless liquid fuming on exposure to air but stable in an inert atmosphere, bp 98°C (16 mm). The product was identified by NMR spectroscopy. Comparison of the ^1H and ^{13}C NMR spectra of initial aluminacyclopentane **Ib** [16] and compound **IIb** revealed considerable differences only in the chemical shifts of CH_2 groups directly linked to Al or B.

Compound **Ib** characteristically showed in the ^1H and ^{13}C NMR spectra upfield signals from protons and carbon atoms directly attached to Al: δ_{C^2} 13.6 ppm, δ(2-H) –0.98, 0.00 ppm; δ_{C^5} 5.7 ppm, δ(5-H) –0.49, 0.20 ppm [4]. In the HSQC NMR spectrum of **IIb** (Fig. 1) we observed signals from three couples of dia-

Scheme 1.



Ia–Ic **IIa–IIc** **IIIa–IIIc** **IVa–IVc**

R = Bu (**a**), C_6H_{13} (**b**), C_8H_{17} (**c**).

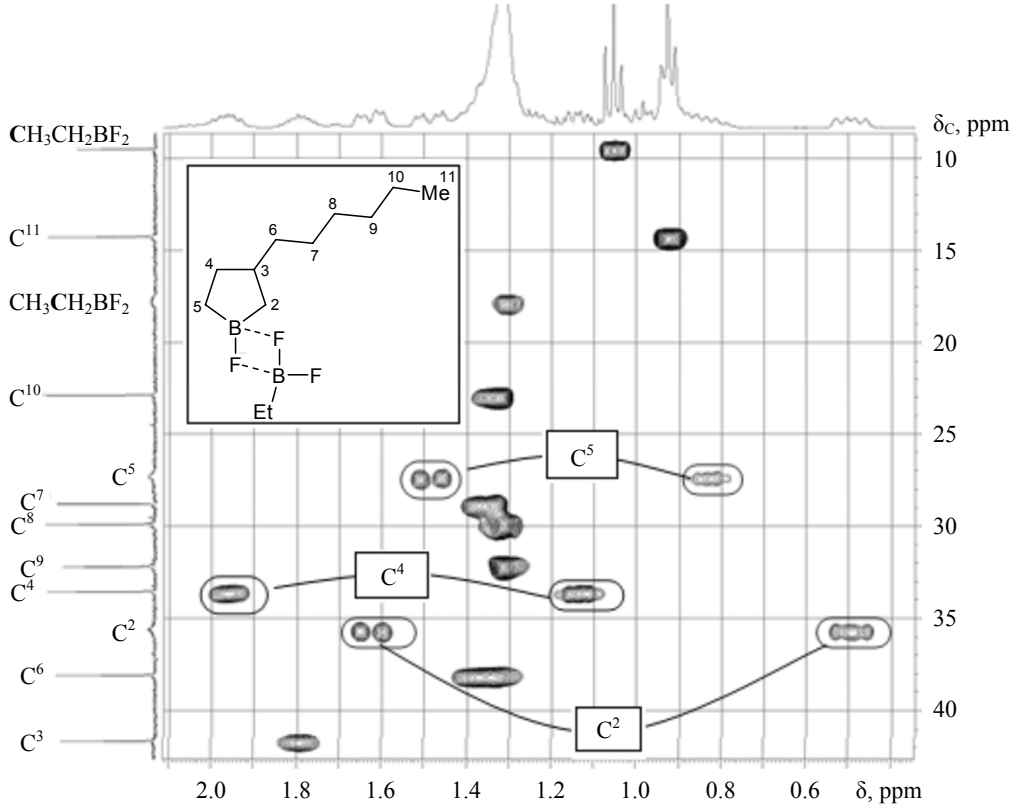


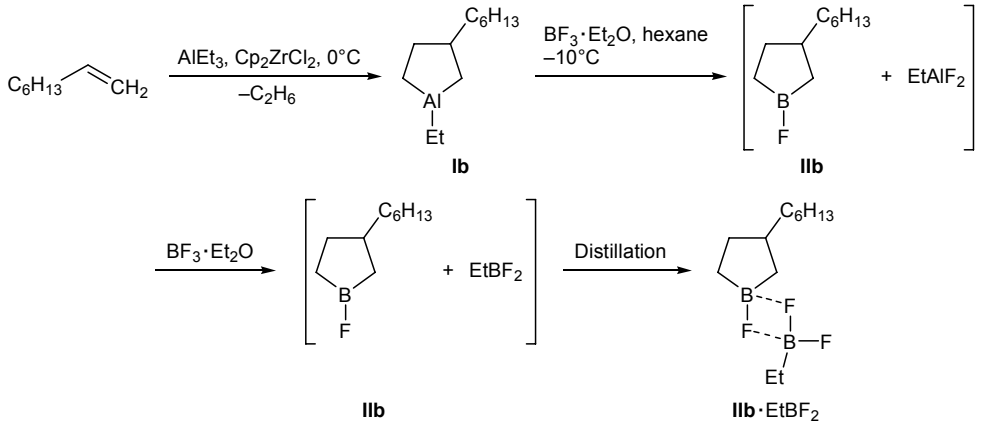
Fig. 1. HSQC spectrum of 1-ethyl-3-hexylborolane (**IIb**) in $CDCl_3$.

stereotopic protons on C^2, C^4, and C^5 in the boracyclopentane ring: δ_{C^2} 35.59 ppm, δ(2-H) 0.49, 1.63 ppm; δ_{C^5} 27.33 ppm, δ(5-H) 0.83, 1.49 ppm. In addition, we observed signals at δ_C 9.48 and 17.83 ppm (the latter was broadened) which displayed couplings with protons resonating at δ 1.05 and 1.30 ppm, respectively. Signals in the ^{13}C NMR spectrum of **IIb** were broadened due to coupling with boron. The position of the C^3 signal (δ_C 43.10 ppm) was almost the same as in the spectrum of **Ib**. Thus compound **IIb** was identified as 1-fluoro-3-hexylborolane. The ^{11}B NMR spectrum of the isolated compound contained two signals, one of which (δ_B 93.2 ppm) was assigned to the endocyclic boron atom, while the second (δ_B 34.6 ppm) was typical of $EtBF_2$ [17]. Therefore, we presumed that the product is a molecular complex of 1-fluoro-3-hexylborolane (**IIb**) with $EtBF_2$.

Presumably, $EtBF_2$ is formed as a result of reaction of $EtAlF_2$ with $BF_3 \cdot Et_2O$, whereas $EtAlF_2$ is the transmetalation product of aluminacyclopentane with $BF_3 \cdot Et_2O$. Thus transmetalation of 1-ethyl-3-hexylaluminacyclopentane (**IIb**) to 1-fluoro-3-hexylboracyclopentane by the action of $BF_3 \cdot Et_2O$ is accompanied by formation of an equimolar amount of $EtBF_2$. Obviously, the above noted additional NMR signals at δ 1.05, 1.30 ppm, δ_C 9.48, 17.83 ppm, and δ_B 34.60 ppm should be assigned to ethyldifluoroborane. The molecular weight of the product, determined by cryoscopy, was 259 (calcd. 248) which corresponds to **IIb**·$BEtF_2$. Thus the data of ^1H, ^{13}C, and ^{11}B NMR spectroscopy, including two-dimensional COSY, HMBC, and HSQC experiments, as well as molecular weight determination, allowed us to conclude that the reaction of 1-ethyl-3-hexylaluminacyclopentane (**Ib**) with $BF_3 \cdot Et_2O$ yields a stable molecular complex of 1-fluoro-3-hexylborolane (**IIb**) with $EtBF_2$ (Scheme 2).

Quantum-chemical simulation (PBE/3z, PRIRODA 6.0 program [18]) of the potential energy surface for complex **IIb**·$BEtF_2$ revealed two energy minima. The corresponding optimized structures are shown in Fig. 2. The calculated distances between the boron and fluorine atoms in complex **A** are about 3.0 Å, whereas the bridging B···F bond length in structure **B** is almost equal to covalent B–F bond length (1.6 Å), so that the configuration of bonds at the boron atom approaches tetrahedral. As a result, the energy of complex **B** is higher by 20.6 kcal/mol than that of **A**. The energy minima are related to each other through transition state TS (Fig. 3), the energy barrier being 20.8 kcal/mol, and the imaginary frequency 179*i* corresponds to

Scheme 2.



vibrations of just bridging B–F bonds. We believe that the experimental NMR spectra of the complexes match structure **A** as thermodynamically more favorable; in addition, the chemical shifts of the boron atom (δ_B ~93 ppm) imply trigonal bond configuration at the boron atom [19]. On the other hand, the B···F distance equal to ~3.0 Å and the calculated enthalpy of the reaction **IIb** + $EtBF_2$ → **IIb**·$EBtF_2$ (ΔH = –1.65 kcal× mol^{-1}; ΔG_{298} = 5.3 kcal/mol) indicate that complex **A** is fairly weak; therefore, it should be stable only in nonpolar medium.

Analogous results were obtained by transmetalation with $BF_3 \cdot Et_2O$ of 3-butyl- and 3-octylaluminacyclopentanes **Ia** and **Ic** which were prepared by cycloalumination of hex-1-ene and dec-1-ene, respectively, according to [14] (Scheme 1).

We made an attempt to extend the developed procedure to other terminal olefins containing cyclic and aromatic substituents. For this purpose, 1,3-substituted aluminacyclopentanes prepared from 4-vinylcyclohexene and allylbenzene were brought into reaction with $BF_3 \cdot Et_2O$ under analogous conditions. 3-(Cyclohex-3-en-1-yl)-1-ethylaluminacyclopentane (**V**) and 3-benzyl-1-ethylaluminacyclopentane (**IX**) reacted with $BF_3 \cdot Et_2O$ to produce the corresponding borolanes **VI** and **X** as 1:1 complexes with $EtBF_2$ (Scheme 3).

Oxidation of substituted borolanes **IIa**–**IIc**, **VI**, and **X** with hydrogen peroxide in alkaline medium [12, 20, 21] gave mixtures of 1,4-diols **IVa**–**IVc**, **VIII**, and **XII** and monools **IIIa**–**IIIc**, **VII**, and **XI** at a ratio of 3:1 (Schemes 1, 3). The formation of monools **IIIa**–**IIIc**, **VII**, and **XI** may be rationalized in terms of specificity of oxidation of fluorine-containing borolanes as complexes with $EtBF_2$. As might be expected, the ^{13}C NMR spectra of compounds **VI**–**VIII** having a cyclohexenyl substituent displayed double sets of signals due to the presence of a chiral center in the six-membered ring.

To conclude, we have found that substituted aluminacyclopentanes undergo transmetalation by the action of $BF_3 \cdot Et_2O$ with formation of the corresponding 1,3-substituted borolanes which are stabilized as 1:1 complexes with $EtBF_2$. The proposed procedure ensures a simple and efficient one-pot synthesis of

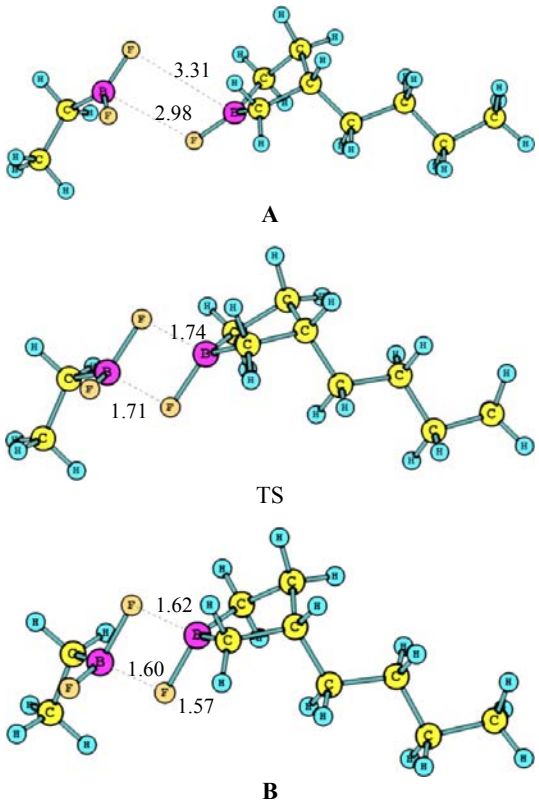
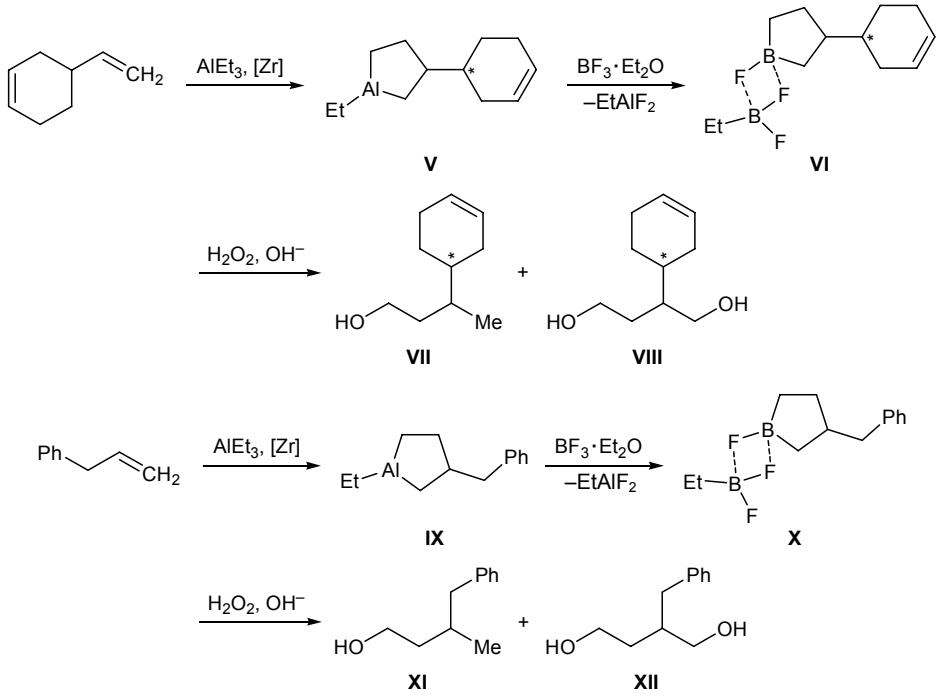


Fig. 2. Optimized structures **A** and **B** of complex **IIb**·$EtBF_2$ at the energy minima and transition state TS according to DFT–PBE/3z calculations (PRIRODA 06).*

* The geometric parameters of the 1:1 complex of 3-butyl-1-fluoroborolane with ethyldifluoroborane, optimized at the DFT–MP2 level of theory, were fairly similar.

Scheme 3.



five-membered cyclic organoboron compounds starting from olefins, $AlEt_3$, and $BF_3 \cdot Et_2O$.

EXPERIMENTAL

All reactions with organometallic reagents were carried out under argon. Commercially available 98% $AlEt_3$ and 48% $BF_3 \cdot Et_2O$ were used without additional purification. GC–MS analysis was performed on a Shimadzu GCMS-QP2010 Plus instrument [SLB-5ms glass capillary column, 60000 mm × 0.25 mm × 0.25 μm (Supelco, USA); carrier gas helium; oven temperature programming from 40 to 280°C at a rate of 5 deg/min; injector temperature 280°C; ion source temperature 200°C; electron impact, 70 eV]. The IR spectra were recorded on a Bruker Vertex 70 spectrometer from liquid films. The ^1H, ^{13}C, and ^{11}B NMR spectra and two-dimensional homo- (COSY) and heteronuclear (HSQC, HMBC) correlation spectra were recorded from solutions in $CDCl_3$ on a Bruker Avance 400 spectrometer operating at 400.13 (^1H), 100.62 (^{13}C), or 128.33 MHz (^{11}B). The chemical shifts were determined relative to tetramethylsilane (^1H, ^{13}C; internal reference) or $BF_3 \cdot Et_2O$ (^{11}B). The molecular weights of organoboron compounds were determined by cryoscopy in benzene under argon according to [22] with the use of a three-necked glass cell equipped with a Beckmann thermometer (accuracy ±0.005°C). The elemental analyses were obtained using a Carlo Erba 1106 analyzer.

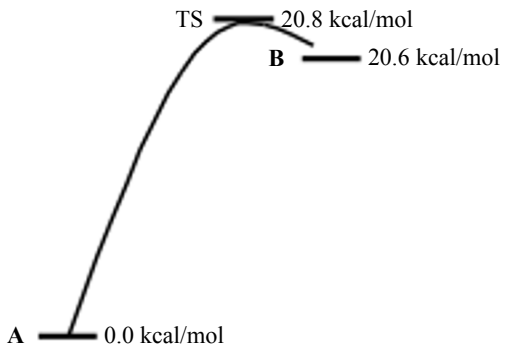


Fig. 3. Calculated (DFT–PBE/3z; PRIRODA 06) relative Gibbs energy diagram; TS is the transition state calculated for model complex **IIb**·$EtBF_2$ (gas phase).

3-Substituted 1-fluoroborolanes IIa–IIc, VI, and X (*general procedure*). A 50-ml glass reactor was charged under dry argon at 0°C with 0.5 mmol of Cp_2ZrCl_2; 10 mmol of the corresponding alkene and 12 mmol of $AlEt_3$ were added in succession under stirring, and the mixture was stirred for 8 h at 20–22°C. Hexane, 10 ml, was then added, the mixture was cooled to –10°C, 24 mmol of $BF_3 \cdot Et_2O$ was added dropwise, and the mixture was allowed to warm up to room temperature and stirred for 0.5 h. The solvent was evaporated, and the residue was distilled under reduced pressure in a stream of argon.

3-Butyl-1-fluoroborolane–ethyldifluoroborane (1/1) (IIa). Yield 1.01 g (46%), colorless liquid,

bp 72°C (16 mm). IR spectrum, ν, cm^{-1}: 2975, 2926, 2858, 1460, 1375, 1206, 1079, 774, 728. ^{1}H NMR spectrum, δ, ppm: 0.48 d.d (1H, 2-H, J = 17.6, 10.0 Hz), 0.82–0.98 m (4H, 5-H, CH_3), 1.04 t (3H, C**H**$_3$CH$_2$BF$_2$, J = 7.6 Hz), 1.12 m (1H, 4-H), 1.22–1.35 m (6H, CH_2), 1.30** q (2H, CH_3C**H**$_2$BF$_2$, J = 7.6 Hz), 1.47 br.d (1H, 5-H, J = 18.6 Hz), 1.61 br.d (1H, 2-H, J = 18.0 Hz), 1.78 m (1H, 3-H), 1.94 m (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 9.47 (**C**H$_3$CH$_2$-BF$_2$), 14.29, 17.39 (CH$_3$**C**H$_2$BF$_2$), 23.17, 27.31 (C^5), 31.04, 33.51 (C^4), 35.52 (C^2), 37.70, 41.58 (C^3). ^{11}B NMR spectrum, δ_B, ppm: 34.6 (BEtF$_2$, $W_{1/2}$ = 1.6 kHz), 92.9 ($W_{1/2}$ = 1.2 kHz). Found, %: C 54.39; H 9.52. M 229. $C_{10}H_{21}B_2F_3$. Calculated, %: C 54.62; H 9.63. M 219.89.

1-Fluoro-3-hexylborolane–ethyldifluoroborane (1/1) (IIb). Yield 1.19 g (48%), colorless liquid, bp 98°C (16 mm). IR spectrum, ν, cm^{-1}: 2963, 2950, 2856, 1462, 1371, 1206, 1076, 769, 730. ^{1}H NMR spectrum, δ, ppm: 0.49 d.d (1H, 2-H, J = 17.8, 10.4 Hz), 0.78–0.90 m (1H, 5-H), 0.92 t (3H, CH_3, J = 7.2 Hz), 1.05 t (3H, C**H**$_3$CH$_2$BF$_2$, J = 7.6 Hz), 1.11–1.18 m (1H, 4-H), 1.27–1.40 m (10H, CH_2), 1.30** q (2H, CH_3C**H**$_2$BF$_2$, J = 7.6 Hz), 1.49 d.d (1H, 5-H, J = 18.2, 6.8 Hz), 1.63 d.d (1H, 2-H, J = 18.0, 6.4 Hz), 1.79 m (1H, 3-H), 1.95 m (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 9.48 (**C**H$_3$CH$_2$BF$_2$), 14.25, 17.83 (CH$_3$**C**H$_2$BF$_2$), 22.90, 27.33 (C^5), 28.79, 29.81, 32.21, 33.54 (C^4), 35.59 (C^2), 38.07, 41.65 (C^3). ^{11}B NMR spectrum, δ_B, ppm: 34.6 (EtBF$_2$, $W_{1/2}$ = 2.8 kHz), 93.2 ($W_{1/2}$ = 1.6 kHz). Found, %: C 57.92; H 10.08. M 259. $C_{12}H_{25}B_2F_3$. Calculated, %: C 58.13; H 10.16. M 247.94.

1-Fluoro-3-octylborolane–ethyldifluoroborane (1/1) (IIc). Yield 1.30 g (47%), colorless liquid, bp 117°C (9 mm). IR spectrum, ν, cm^{-1}: 2961, 2952, 2853, 1487, 1458, 1419, 1374, 1337, 1220, 1053, 765, 724. ^{1}H NMR spectrum, δ, ppm: 0.49 d.d (1H, 2-H, J = 17.6, 10.4 Hz), 0.76 m (1H, 5-H), 0.89 t (3H, CH_3, J = 7.2 Hz), 0.98 t (3H, C**H**$_3$CH$_2$BF$_2$, J = 7.6 Hz), 1.10–1.17 m (1H, 4-H), 1.18 q (2H, CH_3C**H**$_2$BF$_2$, J = 7.6 Hz), 1.23–1.40 m (15H, 5-H, CH_2), 1.48 d.d (1H, 2-H, J = 17.2, 6.0 Hz), 1.73 m (1H, 3-H), 1.89 m (1H, 4-H). ^{13}C NMR spectrum, δ_C, ppm: 9.46 (**C**H$_3$CH$_2$-BF$_2$), 14.56, 17.80 (CH$_3$**C**H$_2$BF$_2$), 23.61, 27.95 (C^5), 29.73, 30.42, 30.77, 31.09, 32.98, 33.61 (C^4), 35.48 (C^2), 39.09, 41.70 (C^3). ^{11}B NMR spectrum, δ_B, ppm: 34.7 (EtBF$_2$, $W_{1/2}$ = 1.8 kHz), 93.1 ($W_{1/2}$ = 1.6 kHz). Found, %: C 60.69; H 10.47. M 267. $C_{14}H_{29}B_2F_3$. Calculated, %: C 60.92; H 10.59. M 275.99.

** The signal was obscured by those of alkyl protons.

3-(Cyclohex-3-en-1-yl)-1-fluoroborolane–ethyldifluoroborane (1/1) (VI). Yield 1.12 g (46%), colorless liquid, bp 99°C (16 mm). IR spectrum, ν, cm^{-1}: 3022, 2921, 2661, 1653, 1619, 1457, 1372, 912, 773, 728, 655. ^{1}H NMR spectrum, δ, ppm: 0.47 m (1H, 2-H), 0.73–0.85 m (2H, 2-H, 5-H), 0.90–0.96 m (3H, C**H**$_3$CH$_2$BF$_2$), 1.20–1.40 m (2H, 5-H, 1′-H), 1.25 m (2H, CH_3C**H**$_2$BF$_2$), 1.50–1.78 m (5H, 2′-H, 4-H, 6′-H), 1.58 m (1H, 3-H), 2.00–2.18 m (3H, 4-H, 5′-H), 5.60 br.s (2H, 3′-H, 4′-H). ^{13}C NMR spectrum, δ_C, ppm: 8.00, 9.20 (**C**H$_3$CH$_2$BF$_2$), 18.85, 19.50 (CH$_3$**C**H$_2$-BF$_2$), 25.68, 25.72, 26.75, 26.79 (C$^{5'}$, C$^{6'}$), 26.32, 26.40 (C^5), 30.33, 30.44, 30.91, 30.98 (C^4, C$^{2'}$), 32.57, 33.10 (C^2), 40.95, 41.09 (C$^{1'}$), 47.25, 47.32 (C^3), 127.07, 127.11 (C$^{3'}$, C$^{4'}$). ^{11}B NMR spectrum, δ_B, ppm: 35.63 (EtBF$_2$, $W_{1/2}$ = 2.8 kHz), 87.10 ($W_{1/2}$ = 1.8 kHz). Found, %: C 58.90; H 8.55. M 258. $C_{12}H_{21}B_2F_3$. Calculated, %: C 59.09; H 8.68. M 243.91.

3-Benzyl-1-fluoroborolane–ethyldifluoroborane (1/1) (X). Yield 1.19 g (47%), colorless liquid, bp 103°C (17 mm). IR spectrum, ν, cm^{-1}: 3084, 3062, 3026, 2959, 2925, 2873, 1602, 1494, 1375, 1205, 1113, 742, 699. ^{1}H NMR spectrum, δ, ppm: 0.73 d.d (1H, 2-H, J = 17.7, 10.3 Hz), 0.99 m (1H, 5-H), 1.15 t (3H, C**H**$_3$CH$_2$BF$_2$, J = 7.5 Hz), 1.34 m (1H, 4-H), 1.40 q (2H, CH_3C**H**$_2$BF$_2$, J = 7.6 Hz), 1.56–1.75 m (2H, 5-H, 2-H), 2.02 m (1H, 4-H), 2.23 m (1H, 3-H), 2.77 d.d and 2.83 d.d (1H each, PhC**H**$_2$, J = 12.8, 7.2 Hz), 7.10–7.50 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 9.48 (**C**H$_3$CH$_2$BF$_2$), 17.75 (CH$_3$**C**H$_2$BF$_2$), 27.08 (C^5), 33.71 (C^4), 35.24 (C^2), 43.41 (C^3), 43.96 (C^6), 125.58, 128.38, 129.06, 142.49. ^{11}B NMR spectrum, δ_B, ppm: 36.06 (EtBF$_2$, $W_{1/2}$ = 2.4 kHz), 92.99 ($W_{1/2}$ = 1.4 kHz). Found, %: C 61.28; H 7.42. M 252. $C_{13}H_{19}B_2F_3$. Calculated, %: C 61.49; H 7.54. M 253.90.

Oxidation of borolanes IIa–IIc, VI, and X with hydrogen peroxide in alkaline medium (*general procedure*). A solution of 5 mmol of borolane **IIa**–**IIc**, **VI**, or **X** in 10 ml of hexane was cooled to 0°C, 6 ml of 20% aqueous sodium hydroxide was added, and 2 ml of 30% hydrogen peroxide was then slowly added dropwise, and the mixture was stirred for 6 h. The organic layer was separated, the aqueous layer was extracted with diethyl ether (2×30 ml), the extracts were combined with the organic phase, dried over $CaCl_2$, and concentrated under reduced pressure, and the residue was separated by column chromatography on silica gel (40–100 mesh, 30 cm× 12 mm; ethyl acetate–hexane, 1:50). The spectral parameters and physical constants of compounds **IIIa**, **IIIc**, **IVa**, **IVb**, **XI**, and **XII** were reported previously [14, 15, 23–26].

3-Methylnonan-1-ol (IIIb). Yield 0.44 g (31%), oily substance, R_f 0.66 (ethyl acetate–hexane, 1:1). ^{1}H NMR spectrum, δ, ppm: 0.92 m (6H, CH_3), 1.13–1.71 m (13H, CH, CH_2), 3.57–3.82 m (2H, C**H**$_2$OH). ^{13}C NMR spectrum, δ_C, ppm: 14.10, 19.64, 22.64, 26.92, 29.60, 29.61, 31.91, 37.17, 40.00, 61.28. Mass spectrum: *m*/*z* 158 $[M]^+$. Found, %: C 75.79; H 13.95. $C_{10}H_{22}O$. Calculated, %: C 75.88; H 14.01. *M* 158.28.

2-Octylbutane-1,4-diol (IVc). Yield 1.25 g (62%), oily substance, R_f 0.17 (ethyl acetate–hexane, 1:1). ^{1}H NMR spectrum, δ, ppm: 0.86 t (3H, CH_3, J = 7.0 Hz), 1.08–1.75 m (17H, CH, CH_2), 3.25–3.75 m (4H, C**H**$_2$OH). ^{13}C NMR spectrum, δ_C, ppm: 14.30, 22.84, 28.31, 28.63, 28.82, 29.61, 30.20, 32.51, 35.90, 36.72, 60.72, 65.91. Mass spectrum: *m*/*z* 202 $[M]^+$. Found, %: C 71.01; H 12.88. $C_{12}H_{26}O_2$. Calculated, %: C 71.23; H 12.95. *M* 202.23.

3-(Cyclohex-3-en-1-yl)butan-1-ol (VII). Yield 0.42 g (27%), oily substance, R_f 0.49 (ethyl acetate–hexane, 1:1). ^{1}H NMR spectrum, δ, ppm: 0.94–0.97 m (3H, CH_3), 1.32–2.07 m (10H, CH, CH_2), 3.63–3.81 m (2H, C**H**$_2$OH), 5.68 m (2H, CH=CH). ^{13}C NMR spectrum, δ_C, ppm: 14.08 (14.47), 20.51 (20.58), 26.03 (26.10), 26.15 (26.21), 28.69 (29.12), 34.13 (34.17), 47.00 (47.05), 62.94 (63.14), 126.89 (127.00), 127.07 (126.14). Mass spectrum: *m*/*z* 154 $[M]^+$. Found, %: C 77.73; H 11.69. $C_{10}H_{18}O$. Calculated, %: C 77.86; H 11.76. *M* 154.25.

2-(Cyclohex-3-en-1-yl)butane-1,4-diol (VIII). Yield 1.09 g (64%), oily substance, R_f 0.14 (ethyl acetate–hexane, 1:1). ^{1}H NMR spectrum, δ, ppm: 1.67–2.02 m (10H, CH, CH_2), 3.49–3.73 m (4H, C**H**$_2$OH), 4.48 s (2H, OH), 5.62 m (2H, CH=CH). ^{13}C NMR spectrum, δ_C, ppm: 25.84, 26.04 (26.09), 28.59 (28.99), 32.81 (33.02), 35.61 (35.51), 43.99 (44.05), 61.29 (61.36), 64.17 (64.38), 126.54 (126.62), 126.99. Mass spectrum: *m*/*z* 170 $[M]^+$. Found, %: C 69.45; H 10.58. $C_{10}H_{18}O_2$. Calculated, %: C 70.55; H 10.66. *M* 170.25.

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