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ORGANOBORON COMPOUNDS

CDIII *. IODINATION OF ATE COMPLEXES OF 1-BORAADAMANTANE

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Summary

The complex of 1-boraadamantane with tetrahydrofuran (I) reacts with iodine in the presence of lithium methylate or methyllithium to produce 3-methoxy- (III) and 3-methyl-7-iodomethyl-3-borabicyclo[3.3.1]nonane (X), respectively. Further iodination of III in the presence of lithium methylate proceeds in a complicated manner due to replacement of iodine by the methoxy group in the starting compound. The high reactivity of the halogen in III permitted a one-pot synthesis of 3-methoxy-7-methoxymethyl-3-borabicyclo[3.3.1]nonane from I. Iodination of lithium 1-isopropenyl-1-boraadamantanate leads, after methanolysis, to 3-methoxy-7-isobutenyl-3-borabicyclo[3.3.1]nonane. The latter compound was converted to 7-isobutenyl-3-methyl-3-borabicyclo[3.3.1]nonane on treatment with MeMgI.

Results and discussion

Trialkylboranes are known to be unreactive towards iodine. Thus, tripropylborane reacts with iodine only at temperature above 140°C to form propyl iodide and iodo(di-n-propyl)borane [1]. The reactivity of trialkylboranes with respect to iodine increases in the presence of bases. Thus, reaction of primary trialkylboranes with iodine in the presence of NaOH proceeds at a high rate under mild conditions (25°C), with two boron–carbon bonds being cleaved [2].

$$R_3B \xrightarrow{2\ I_2/2\ NaOH} 2\ RI + RB(OH)_2$$

In the case of secondary trialkylboranes, only one of the three B–C bonds is cleaved, the reaction occurring considerably more slowly [2]. Similar results are observed in iodination of trialkylboranes with ICl in the presence of sodium acetate

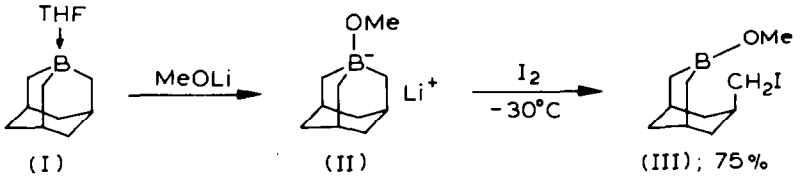
* For part CDII see ref. 18.

[3]. The latter method may be applied to iodination of boron compounds with functional groups sensitive to strong bases. The use of sodium methylate as a base leads to a significant increase in the iodide yields. Under these conditions, two B–C bonds are cleaved in the secondary trialkylboranes, while the third B–C bond is cleaved partially in the primary trialkylboranes [4]. An important feature of the iodination of trialkylboranes is the inversion of the configuration of the carbon atom attached to boron [5], since retention of the configuration is more characteristic for reactions of organoboranes (with the exception of bromination).

In the iodination reaction, the role of the base is to coordinate with the boron atom, which results in the formation of a borate anion, in which polarization of the B–C bond is increased (an increase in carbanionic character of the alkyl group) thus facilitating the heterolytic rupture of the bond. Hence it follows that the actual reacting species in the iodination is the ate complex $\overset{+}{M}R_3\bar{B}OR$ (R = Alk, H) rather than the trialkylborane. The alkyl groups in tetraalkylborates $R_4\bar{B}$ are the most carbanionic in character; therefore, their iodination proceeds especially readily [6]. In the course of the investigation of the reactivity of ate complexes of the 1-boraadamantane series [7], we have undertaken a study on their reactions with iodine. The ate complexes studied were not isolated and were used in reactions as their solutions. Formation of the borates in the solutions was observed with the aid of ^{11}B NMR spectra.

As can be seen in Table 1, the chemical shifts of ate complexes of the 1-boraadamantane series and those of 7-substituted 3-borabicyclo[3.3.1]nonanes are, characteristic of these series of compounds. The compounds from which the organoborates were obtained, tetrahydrofuran-1-boraadamantane and 3-methoxy-7-methyl-3-borabicyclo[3.3.1]nonane, have ^{11}B chemical shifts of −8 [8] and 53 ppm [9], respectively. For comparison, the values of the chemical shifts of some alicyclic ate complexes are as follows: $[Me_4B]^-Li^+$ (−20.7 ppm) [10], $[Me_3BOMe]^-K^+$ (−1.0 ppm) [11], $[Me_2B(OMe)_2]^-K^+$ (14.4 ppm) [11].

The ate complex II, obtained by the action of MeOLi on I in MeOH, reacts with iodine at −30°C to form 3-methoxy-7-iodomethyl-3-borabicyclo[3.3.1]nonane (III) in 75% yield.

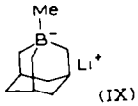
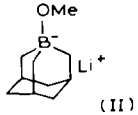
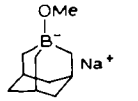
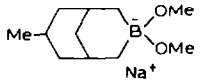
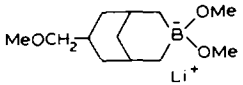
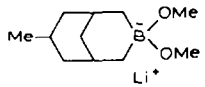


(I) (II) (III); 75%

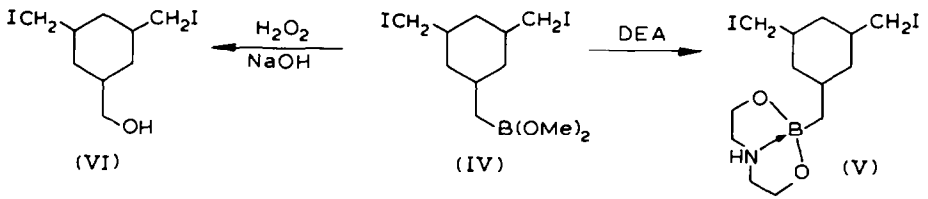
The reaction of the compound III with I_2 in the presence of MeOLi has been studied further, while III, obtained by monoiodination of II, was later iodinated without isolation. It turned out that a complex mixture of products is formed in this case. The desired dimethoxy[3,5-di(iodomethyl)cyclohex-1-ylmethyl]borane (IV) was isolated in 30% yield by fractional distillation under high vacuum. The ^1H NMR spectrum of IV (in C_6H_6) contains a singlet at 3.38 ($B(OMe)_2$) and a doublet at 2.75 ppm (CH_2I). Because of partial decomposition upon distillation, the diiodide IV contained some impurities, so it was characterized as its diethanolamine derivative (V).

TABLE I

^{11}B NMR CHEMICAL SHIFTS OF ATE COMPLEXES OF 1-BORAADAMANTANE AND 7-SUBSTITUTED 3-METHOXY-3-BORABICYCLO[3.3.1]NONANES (RELATIVE TO $BF_3 \cdot Et_2O$) [a]

Compound	Solvent	Chemical shift (ppm)
 (IX)	Et_2O/THF (1/1)	−20.3
 (II)	MeOH/THF (2/3)	−3.1
	THF	−3.0
	THF	6.0
	MeOH/THF (2/3)	14.3
	MeOH/THF (2/3)	15.5

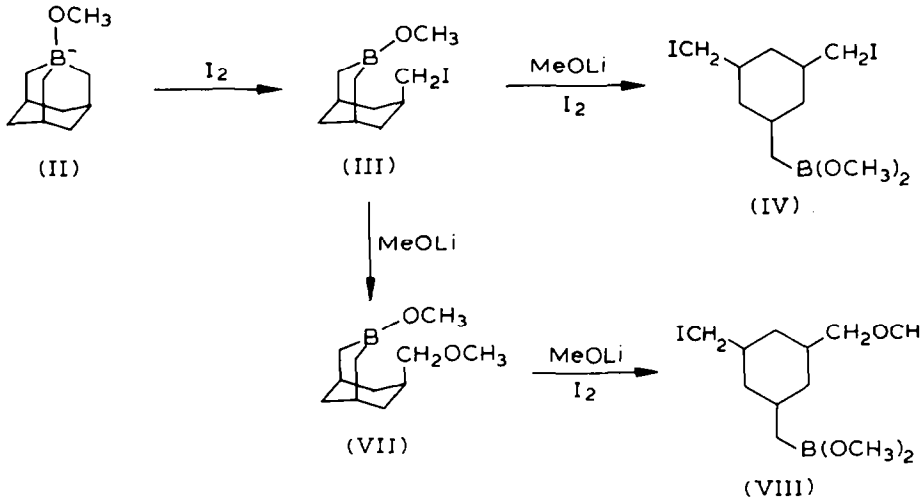
[a] Signals downfield relative to the etherate are positive.



Oxidation of IV with alkaline hydrogen peroxide afforded 1,3-di(iodomethyl)-5-hydroxymethylcyclohexane (VI). Both the composition and approximate ratio of products of the diiodination reaction of II was determined by comparing ^1H NMR spectra of authentic compounds with that of the mixture. The most volatile fraction obtained by distillation of the reaction products (up to 80°C at 8×10^{-3} mmHg) consisted mainly (70%) of 3-methoxy-7-methoxymethyl-3-borabicyclo[3.3.1]nonane (VII) (a singlet at 3.43 ($BOCH_3$), a singlet at 3.18 (OCH_3), a doublet at 3.12 ppm

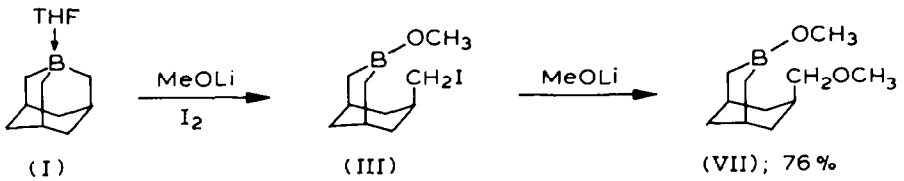
(CH_2O)). In addition, the ^1H NMR spectrum indicates the presence in the mixture of the bicyclic compound III formed by the monoiodination of II (a singlet at 3.43 ppm ($BOCH_3$) and a doublet at 3.03 ppm (CH_2I)). The remainder doublet at 2.85 ppm was assigned to dimethoxy(3-iodomethyl-5-methoxymethylcyclohex-1-ylmethyl)borane (VIII). The yields obtained were as follows: ~ 30% (IV), ~ 25% (VII), ~ 15% (III), ~ 5% (VIII).

Formation of the bicyclic compound VII is by a side reaction, namely the replacement of iodine by an OMe group in the product of the monoiodination of III. Iodination of VII produces one more byproduct (VIII).



Reversing the order of introducing the reagents in the second stage did not cause any significant change in the composition of the products (the yield of IV was 35%).

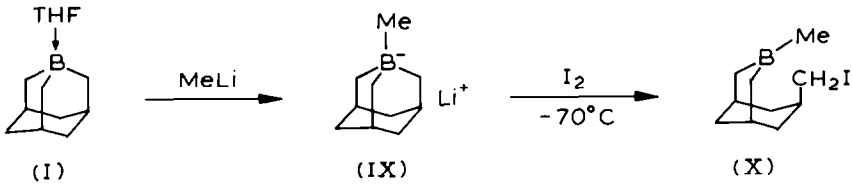
The high reactivity of the iodine in compound III was used to develop a method for the one-pot synthesis of 3-methoxy-7-methoxymethyl-3-borabicyclo[3.3.1]nonane (VII) from I (see [9,12]). The compound I was treated with one mole of iodine in the presence of two moles of MeOLi. The initially formed iodide III smoothly forms VII under the action of the second mole of the methylate at 20°C.



It was shown previously that bromine in 3-methoxy-7-bromomethyl-3-borabicyclo[3.3.1]nonane can be replaced by a methoxy group upon heating (50°C) with sodium methylate in methanol [9].

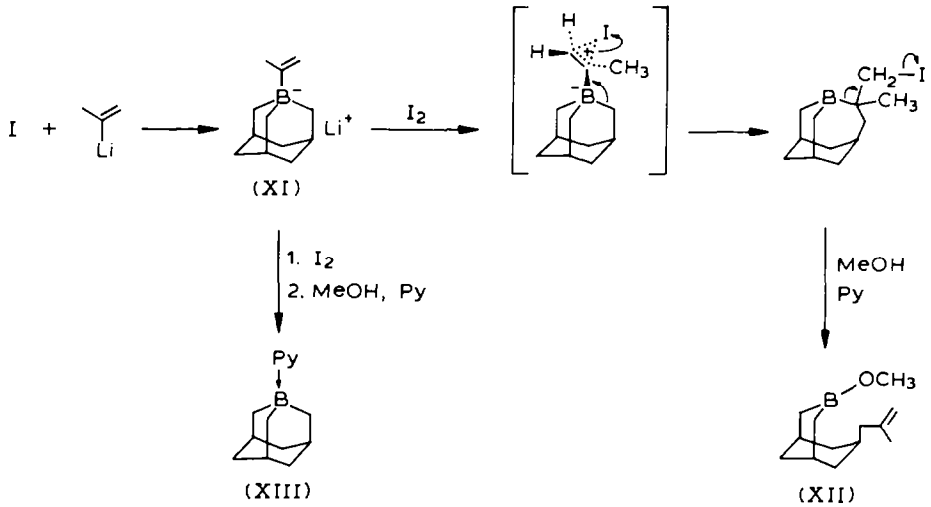
There are two possible directions for the iodination of the ate complex IX (obtained from I and MeLi): cleavage of the B–Me bond with retention of the 1-boraadamantane structure, or a cleavage of the cyclic B–C bond to form a derivative of 3-borabicyclo[3.3.1]nonane. The reactivity of IX is considerably higher than that of the ate complex II, which enables lower temperature to be used for the

halogenation (−70°C). As a result of the reaction, 7-iodomethyl-3-methyl-3-borabicyclo[3.3.1]nonane is obtained in 65% yield, i.e. 65% of the iodination occurs by the second way.



(I) (IX) (X)

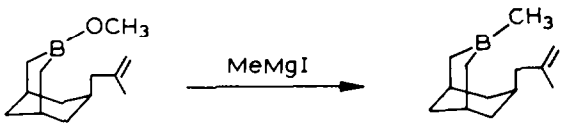
The reaction of iodine with alkenyldialkylboranes in the presence of bases produces olefins rather than alkyliodides. It was proposed that, coordination between the base and the boron atom takes place to form an ate complex. Further electrophilic addition of iodine to the double bond results in formation of a cyclic iodonium ion, followed by migration of an alkyl group from the boron to the α-carbon atom to produce a β-iodoborane. The reaction is completed by β-elimination of a boron-containing group and iodine [13]. The ate complexes obtained from trialkylboranes and 1-alkenyllithium compounds react with iodine in an analogous manner [14,15]. It was of great interest to investigate iodination of the alkenyl ate complexes of 1-boraadamantane. Compound I reacts with isopropenyllithium to form the borate XI, sequential treatment of which with iodine at −70°C and then with a pyridine-methanol mixture furnishes 3-methoxy-7-isobutenyl-3-borabicyclo[3.3.1]nonane (XII) in 45% yield and pyridine-1-boraadamantane (XIII) (35%).



(XI) (XIII) (XII)

According to the above scheme, formation of the bicycle XII takes place via an addition-elimination. The moderate yield of XII is apparently explained by a parallel reaction taking place: rupture of the boron–isopropenyl bond by iodine.

7-Isobutenyl-3-methyl-3-borabicyclo[3.3.1]nonane (XIV) was synthesized by reaction of XII with MeMgI. The ^{13}C NMR spectra of the compounds prepared are presented in Table 2. The chemical shifts of the C(9) atoms in the bicycles III, X,



(XII) (XIV)

XII, and XIII are close to those previously determined for the compounds with the preferred double chair conformation [16].

Experimental

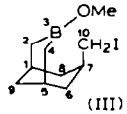
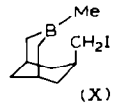
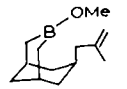
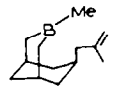
All organoboron compounds were worked up under dry argon. IR spectra were recorded on a UR-20 spectrometer. ^{1}H NMR spectra were obtained on a Tesla BS-497 instrument (100 MHz), ^{13}C NMR spectra on Bruker WP-60 (15.08 MHz for carbon) and on Bruker WM-250 (68.69 MHz for carbon) spectrometers. The off-resonance method was used for assignment of the spectral lines. Tetrahydrofuran-1-boraadamantane was prepared according by a reported method [17].

3-Methoxy-7-iodomethyl-3-borabicyclo[3.3.1]nonane (III)

To 24 g (116.4 mmol) of I in 80 ml of THF was added a solution of 4.32 g (116.4 mmol) of LiOMe in 50 ml of MeOH. To the mixture at −30°C were added dropwise

TABLE 2

^{13}C NMR CHEMICAL SHIFTS OF 3,7-DISUBSTITUTED 3-BORABICYCLO[3.3.1]NONANES (NEAT, δ, ppm)

Compound	C(1,5)	C(2,4)	C(6,8)	C(7)	C(9)	C(10)	Others
 (III)	26.7	26.2	37.15	34.7	34.2	16.2	52.6 (OCH_3)
 (X)	26.1	36.0	36.6	35.0	34.7	15.9	12.45 (BCH_3)
 (XII)	27.6	26.9	36.8	28.7	35.1	46.75	21.8 (CH_3) 52.45 (OCH_3) 111.95 (CH_2=C) 144.7 (C=CH_2)
 (XIII)	27.1	36.0	36.4	29.0	35.6	46.2	12.2 (BCH_3) 21.7 (CH_3) 111.6 (CH_2=C) 144.2 (C=CH_2)

29.8 g (116.5 mmol) of I_2 in 80 ml of THF. After stirring for 1 h at 0°C, the mixture was heated to 20°C. The solvent was removed, and to the residue 20 ml of MeOH were added, followed by extraction with hexane (3 × 40 ml). The combined hexane extracts were evaporated to 60 ml of volume and treated with a small amount of solid $Na_2S_2O_3 \cdot 5H_2O$. After the solvent had been removed, the residue was distilled to yield 25.2 g (74%) of III, b.p. 82–84°C (10^{-2} mmHg), n_D^{20}1.5463. Found: C, 41.46; H, 6.32; B, 3.37; I, 42.76. $C_{10}H_{18}OBI$ calcd.: C, 41.13; H, 6.22; B, 3.72; I, 43.47%. ^1H NMR ($CHCl_3$, δ, ppm): 3.57 s (3 H, CH_3O), 3.10 d (2 H, CH_2I, $J = 7.2$ Hz), 2.40–0.72 (signals from bicycle protons).

Dimethoxy[3,5-di(iodomethyl)cyclohex-1-ylmethyl]borane (IV)

a) To the ate complex II (from 10.93 g (53 mmol) of I in 50 ml of THF and 2.06 g (53 mmol) of MeOLi in 50 ml of MeOH) was added dropwise at −30°C a solution of 13.45 g (53 mmol) of I_2 in 50 ml of THF. The mixture was heated to 20°C, then cooled to −30°C, whereupon were subsequently added 2.06 g (53 mmol) of MeOLi and 13.45 g (53 mmol) of I_2 in 50 ml of THF. The mixture was heated to 20°C, solvent was removed, and 12 ml of MeOH were added with subsequent extraction with hexane (3 × 40 ml). The combined hexane extracts were treated with a small amount of $Na_2S_2O_3 \cdot 5H_2O$. Evaporation of solvent and distillation of the residue gave three fractions:

1) 3.84 g, b.p. 60–80°C (8×10^{-3} mmHg), ^1H NMR shows the fraction to consist of VII (70%), III (20%) and VIII (10%);

2) 1.72 g, b.p. 80–145°C (8×10^{-3} mmHg), contains III (60%), VII (15%), IV (15%) and VIII (10%);

3) 6.89 g (30%) of IV, b.p. 149–152°C (8×10^{-3} mmHg), n_D^{20} 1.5572. Found: C, 30.70; H, 4.91; B, 2.44; I, 54.89. $C_{11}H_{21}BI_2O$ calcd.: C, 29.36; H, 4.70; B, 2.40; I, 56.42%. ^1H NMR ($CHCl_3$, δ, ppm): 3.46 s (6 H, CH_3O), 3.05 d (4 H, CH_2I, $J = 6.0$ Hz), 2.06–0.39 (signals from aliphatic protons), 0.67 d (2 H, CH_2B).

b) To a solution of sodium 1-methoxy-1-boraadamantanate, obtained from 13.8 g (67 mmol) of I in 50 ml of THF and 50 ml of methanolic MeONa (1.36 *M*), was added dropwise at −30°C a solution of 17.1 g (67 mmol) of I_2 in 80 ml of THF, and the mixture was stirred for 30 min at −30°C and then for 1 h at 20°C. After standing overnight, the mixture was treated at −30°C with 17.1 g (67 mmol) of I_2 in 80 ml of THF and then with 50 ml of methanolic MeONa (1.36 *M*). After removing the solvent, the residue was extracted with hexane (3 × 50 ml) and treated with solid $Na_2S_2O_3 \cdot 5H_2O$. Hexane was removed, and the residue was vacuum distilled to give following fractions:

1) 2.98 g, b.p. 60–100°C (8×10^{-3} mm Hg), this consists of VII (85%) and III (10%) (^1H NMR);

2) 1.80 g, b.p. 100–145°C (8×10^{-3} mm Hg), contains III (60%), VII (20%), IV (10%) and VIII (10%);

3) 10.92 g (36%) of IV, b.p. 150–152°C (8×10^{-3} mmHg), n_D^{20} 1.5576.

Diethanolamino[3,5-di(iodomethyl)cyclohex-1-ylmethyl]borane (V)

To 3.44 g (7.6 mmol) of IV was added a solution of 0.80 g (7.6 mmol) of diethanolamine in 10 ml of THF. The precipitate formed was crystallized from THF to yield 2.75 g (65%) of V, m.p. 151–154°C (decomp.). Found: C, 32.30; H, 5.02; B, 2.30; I, 50.82. $C_{13}H_{24}BIO_2$ calcd.: C, 31.79; H, 4.94; B, 2.21: I, 51.64%.

1,3,-Di(iodomethyl)-5-hydroxymethylcyclohexane (VI)

To 5 g (11.2 mmol) of IV in 15 ml of ether at 0°C were added 0.45 g (11.3 mmol) of NaOH in 8 ml of H_2O and then 7 ml of a 30% solution of H_2O_2. After stirring for 1 h, the mixture was extracted with ether (3 × 20 ml). The combined ethereal extracts were dried over Na_2SO_4, the solvent was removed, and the residue was crystallized from ether. The product thus obtained was purified on silica to afford 2.67 g (60%) of the carbinol VI, m.p 65–67°C. Found: C, 27.80; H, 4.18; I, 64.44. $C_9H_{16}I_2O$ calcd.: C, 27.43; H, 4.09; I, 64.42%. ^{1}H NMR ($CHCl_3$, δ, ppm): 3.53 d (2 H, CH_2O, J = 6.0 Hz), 3.15 d (4 H, CH_2I, J = 5.1 Hz), 2.17 s (1 H, OH), 2.10–0.62 (signals from cycle protons).

3-Methoxy-7-methoxymethyl-3-borabicyclo[3.3.1]nonane (VII)

To 24.94 g (121 mmol) of I in 70 ml of THF were added 9.2 g (242 mmol) of MeOLi in 30 ml of MeOH. The mixture was cooled to −30°C, and afterwards was added dropwise a solution of 30.73 g (121 mmol) of I_2 in THF. After heating to 20°C, the mixture was allowed to stand overnight. After removing the solvent, the residue was treated with 40 ml of MeOH and extracted with hexane (3 × 50 ml). Removal of solvent and vacuum distillation gave 18 g (76%) of VII, b.p. 66–68°C (1 mmHg), n_D^{20} 1.4805 (lit.: [12]). ^{1}H NMR ($CHCl_3$, δ, ppm): 3.53 s (3 H, CH_3OB), 3.22 s (3 H, CH_3OC), 3.07 d (2 H, CH_2O, J = 7.0 Hz), 2.55–0.50 (bicyclic protons).

7-Iodomethyl-3-methyl-3-borabicyclo[3.3.1]nonane (X)

To a solution of the complex IX, obtained from 10.75 g (52.5 mmol) of I in 100 ml of THF and 37.5 ml of ethereal MeLi (1.4 M) were added at −70°C 13.5 g (52.5 mmol) of I_2 in 60 ml of THF. The mixture was stirred for 1 h at −70°C, and then it was heated to 20°C. After removing solvent, the residue was extracted with hexane (3 × 50 ml). After hexane was evaporated, the residue was vacuum distilled to yield 9.5 g (65%) of X, b.p. 65–68°C (10^{-2} mmHg), n_D^{20} 1.5510. Found: C, 43.75; H, 6.56; B, 3.89; I, 45.69. $C_{10}H_{18}BI$ calcd.: C, 43.52; H, 6.54; B, 3.95; I, 45.99%. ^{1}H NMR ($CHCl_3$, δ, ppm): 2.93 d (2 H, CH_2I, J = 7.3 Hz), 2.28–0.58 (bicyclic protons), 0.61 s (3 H, CH_3B).

3-Methoxy-7-isobutenyl-3-borabicyclo[3.3.1]nonane (XII)

To a solution of the ate complex XI, obtained from 5.95 g (29 mmol) of I in 40 ml of THF and 40 ml of ethereal isopropenyllithium (0.73 M), were added at −70°C 7.5 g (29 mmol) of I_2 in 70 ml of THF. The mixture was stirred for 1 h at −70°C, heated to 20°C, and treated with 3.5 ml of pyridine and 5 ml of MeOH. After distilling off solvent, 12 ml of MeOH were added to the residue, followed by extraction with hexane (3 × 25 ml). The combined hexane extracts were treated with a small amount of $Na_2S_2O_3 \cdot 5H_2O$. Removal of solvent and subsequent vacuum distillation gave 2.48 g (42%) of XII, b.p. 81–83°C (1.5 mmHg), n_D^{20} 1.4914. Found: C, 75.36; H, 11.13; B, 5.10. $C_{13}H_{23}BO$ calcd.: C, 75.74; H, 11.32; B, 5.25%. ^{1}H NMR ($CHCl_3$, δ, ppm): 4.66, 4.58 (2 H, CH_2=), 3.60 s (3 H, CH_3O), 2.30–0.68 (aliphatic protons), 1.87 (2 H, CH_2–C=), 1.60 (3 H, CH_3–C=). IR (ν, cm^{-1}): 890 (δ, CH_2=), 1652 (CH_2=C), 3079 (CH_2=).

The residue and mother liquor remaining after the extraction, were treated with $CHCl_3$. The solvent was distilled off, and the residue was crystallized from ethanol to give 2.89 g (35%) of pyridine-1-boraadamantane (XIII), m.p. 163–165°C (lit.: [17]).

7-Isobutenyl-3-methyl-3-borabicyclo[3.3.1]nonane (XIV)

To 14.38 g (70 mmol) of XII in 40 ml of pentane was added an ethereal solution of MeMgI (from 1.78 g (74 mmol) of Mg and 12.5 g (74 mmol) of MeI). After refluxing for 2 h, the solvent was removed and the residue was extracted with pentane (3 × 25 ml). Removal of pentane and vacuum distillation yielded 9.52 g (72%) of XIV, b.p. 66–68°C (1.5 mm Hg), n_D^{20} 1.4910. Found: C, 81. 80; H, 12.28; B, 5.46. $C_{13}H_{23}B$ calcd.: C, 82.12; H, 12.28; B, 5.60%. ^1H NMR ($CHCl_3$, δ, ppm): 4.92, 4.82 (2 H, CH_2=), 2.35–0.55 (aliphatic protons), 1.70 (2 H, CH_2–C=), 1.58 (3 H, CH_3C=), 0.63 s (3 H, CH_3B). IR (ν, cm^{-1}): 829 (δ, CH_2=), 1649 (CH_2=C), 3079 (CH_2=).

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