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ORGANOBORON COMPOUNDS

CDI *. CHLORO-ALLYLIC REARRANGEMENT OF 7-CHLOROMETHYL-3-ALLYL-3-BORABICYCLO[3.3.1]NON-6-ENE TO 6-CHLORO-7-METHYLENE-3-ALLYL-3-BORABICYCLO-[3.3.1]NONANE. SYNTHESIS OF 4-CHLORO-1-BORAADAMANTANE COMPLEXES

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Summary

7-Chloromethyl-3-allyl-3-borabicyclo[3.3.1]non-6-ene (VI) was found to isomerise at 120–130°C to 6-chloro-7-methylene-3-allyl-3-borabicyclo[3.3.1]nonane (VII), the ratio between VI and VII in an equilibrium mixture thereby formed being 2 : 3. Hydroboration of compound VI with $BH_3 \cdot THF$ leads to 1-boraadamantane, while hydroboration of the equilibrium mixture of VI and VII with Et_2BH in THF produces 1-boraadamantane together with 4-chloro-1-boraadamantane. Complexes of the latter compound with THF, Et_3N, Et_2O, and C_5H_5N were synthesized and their ^1H and ^{13}C NMR spectra were investigated.

Results and discussion

Like other acetylene compounds [1], propargyl chloride (I) is capable of reacting with triallylborane (II) [2–4]. With an equimolar ratio of I and II, these compounds slowly react at 20°C to form (2-chloromethyl-1,4-pentadien-1-yl)-diallylborane (III) which then undergoes cyclization to 3-chloromethyl-5-allyl-1-boracyclohex-2-ene (IV). The latter compound is isomerised reversibly to 2-chloro-3-methylene-5-allyl-1-boracyclohexane (V) and is partially cyclized to 7-chloromethyl-3-allyl-3-borabicyclo[3.3.1]non-6-ene (VI).

When the starting compounds have been consumed (after 20–25 days at 20°C), the reaction mixture contains the compounds IV, V, and VI in a ratio of 80 : 15 : 5.

* For part CD see ref. 19.

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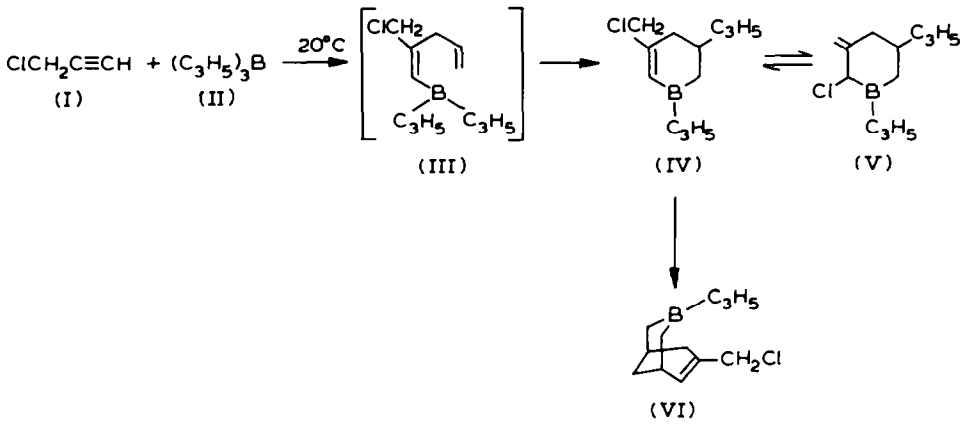
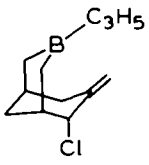


When heated at 120–130°C for 45 min, the mixture forms the bicyclic compound VI along with 3–5% of the isomeric 6-chloro-7-methylene-3-allyl-3-borabicyclo[3.3.1]nonane (VII) [5].



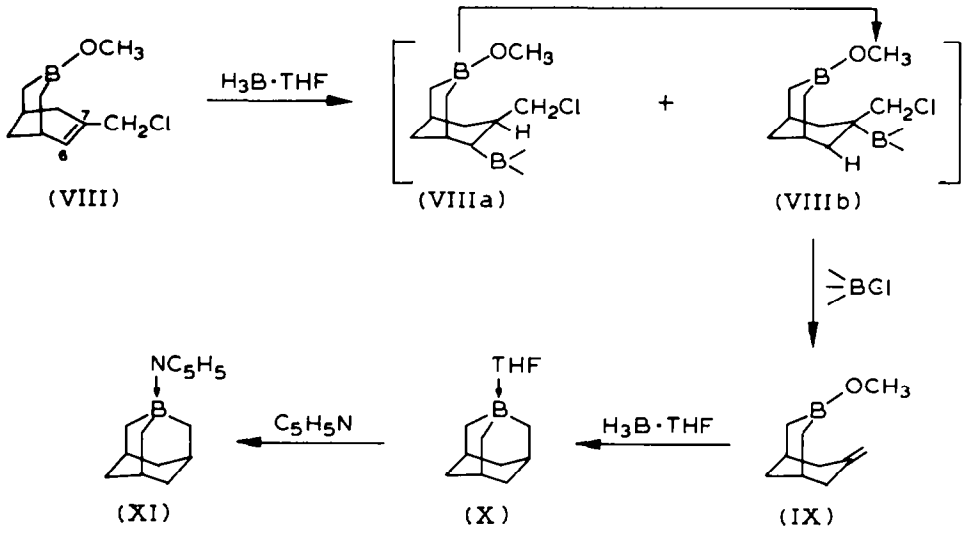
(VII)

Compounds I and II were also reacted by another method by adding propargyl chloride to triallylborane heated to 125–130°C. Like the first reaction method, in this case a mixture of the bicyclic compounds VI and VII was obtained, however, the content of VII increased from 3–5% to 20–25% [2–4].

It is not obvious why the ratio between VI and VII should depend on the reaction conditions. If VII is formed as a result of the cyclization of V, the amount of VII, formed in a stepwise reaction, should correspond with the content of V in the initial mixture. However, the actual content of compound VII under these conditions is only 3–5% and does not change with a change in an amount of V in the initial mixture. This fact might be explained by shift of an IV ⇄ V equilibrium to the left upon heating. However, in such a case both the first and the second reaction methods would provide formation of the compound VII in similar quantities, i.e. about 3–5%. This contradiction may, in our opinion, be explained if the compound VI be assumed to isomerise to VII at 120–130°C. Experiments substantiated this suggestion. It turned out that chloro-allylic rearrangement proceeds at 120–130°C with a content of VII in the equilibrium mixture of 55–65% (by ^{1}H NMR spectroscopy). The chloro-allylic rearrangement may be catalyzed by Lewis acids which are present in the reaction mixture (X–B$\langle^{R_1}_{R_2}$) [6].

Using mixtures with various ratios of the bicyclic allylboranes VI and VII, we have investigated hydroboration of the corresponding methoxy derivatives (VIII and XII), as a result of which the compounds of the 1-boraadamantane series are obtained. 3-Methoxy-7-chloromethyl-3-borabicyclo[3.3.1]non-6-ene (VIII), like its analogues with the methoxymethyl [7] or tetrahydropyranyl group [8] at the 7

position, is capable of adding the boron atom in the 6 or 7 position to give the diboron compounds VIIIa and VIIIb, respectively.



In the presence of an excess of $>$B–H, the compound VIIIa isomerises to VIIIb, which forms 3-methoxy-7-methylene-3-borabicyclo[3.3.1]nonane (IX) as a result of β-elimination. Hydroboration of IX with the complex $BH_3 \cdot THF$ leads to the THF complex of 1-boraadamantane (X) [9]. The rates of these reactions may be judged by the amount of the hydride consumed. Data on the hydroboration of VIII with $BH_3 \cdot THF$ in tetrahydrofurane solution are presented in Table 1. As can be seen in the Table, two equivalents of hydride are consumed in the hydroboration of VIII; the reaction proceeds at a high rate and even at 0°C is virtually complete after 4 h. The 1-boraadamantane thus formed was isolated as a complex with pyridine (XI) in a yield of about 70%.

A mixture of compounds VIII and XII was hydroborated with an equimolar quantity of Et_2BH in tetrahydrofuran. In this process, 6-chloro-3-methoxy-7-methylene-3-borabicyclo[3.3.1]nonane (XII) forms the THF complex of 4-chloro-1-boraadamantane (XIII), which was isolated in 50–60% yield (90–95% based on the isomer XII) by freezing off from a pentane solution. After separation of XIII, the

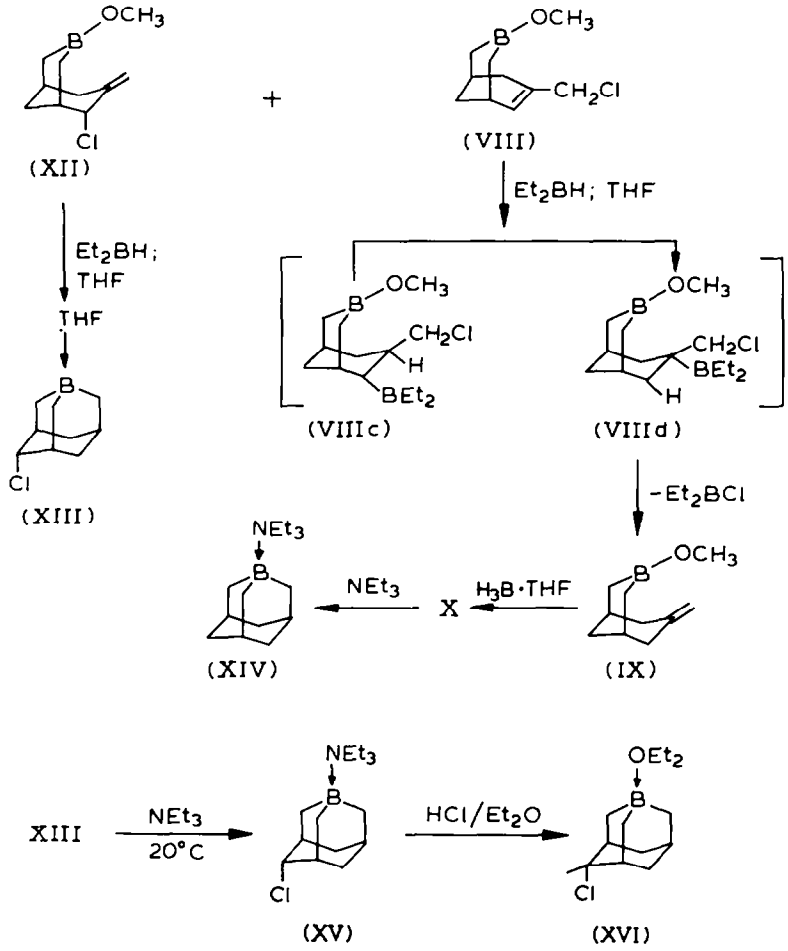
TABLE 1

HYDRIDE UPTAKE (EQUIVALENTS) DURING THE HYDROBORATION OF VIII [a] (B–H : VIII = 3)

Temp. (°C)	Time						
	5 min	30 min	1 h	2 h	3 h	4 h	24 h
0	0.80	1.27	1.52	1.72	1.83	1.90	1.97
20	1.69	1.89	1.98				

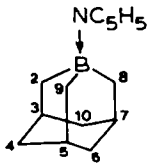
[a] With an admixture of isomeric XII (~ 3%).

residue contained two products from the addition of the boron atom in the 6 and 7 positions of VIII: compounds VIIIc and VIIId. To isomerise VIIIc to VIIId and to hydroborate IX, formed from VIIId by β-elimination of Et_2BCl, a solution of $BH_3 \cdot THF$ in THF was added to the solution of the mixture of VIIIc and VIIId. As a result of these sequential reactions, 1-boraadamantane was obtained which was isolated as a complex with triethylamine (XIV) in 60% yield (based on VIII). The THF complex of 4-chloro-1-boraadamantane was converted subsequently to its triethylamine complex XV and the etherate XVI.



The ^{13}C NMR spectra of complexes XIII, XV and 4-chloro-1-boraadamantane pyridinate (XVII), which had been prepared by us previously [5], were measured. An inspection of these spectra made it possible to determine the location of the chlorine atom in the compounds under question. The ^{13}C NMR spectrum of 4-chloro-1-boraadamantane pyridinate (XVII), recorded with complete suppression of coupling with protons, contains 8 signals: 31.8, 32.8 (broad signal), 33.1, 39.9, 73.3, 125.2, 139.1, 144.3 ppm. The signals centered at 125.2, 139.1, and 144.3 ppm are assigned to the pyridine carbon atoms, while that at 32.8 ppm is a broad signal related to the

carbon atoms neighbouring the boron atom. Assignment of all the signals was carried out on the basis of the spectra obtained with the aid of partial suppression of a C–H coupling (off-resonance), with the calculations of ^{13}C chemical shifts being accomplished according an additive scheme. Providing that the chlorine atom is sufficiently remote from the boron in compound XVII and that the geometry of the molecule of adamantane is similar to that of pyridine-1-boraadamantane [10,11], it is possible, in this case, to use values of the increments α, β, γ_{syn}, γ_{anti}, δ_{syn}, δ_{anti} as well of ε-effects quoted [12,13] which describe the alteration of chemical shifts in progressing from adamantane to 2-chloroadamantane (a similar calculation scheme was previously applied with success to 4,6-dimethyl-1-boraadamantane complexes [14]). The following values of the chemical shifts of the 1-boraadamantane nucleus carbon in 1-boraadamantane pyridinate (XI) were obtained: 33.1 ($B-CH_2$, broad signal), 33.1 (C–H), 40.4 (CH_2) (assignments were carried out with the aid of off-resonance).

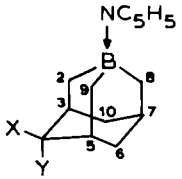


(XI)

The value of the chemical shift of any carbon atom in XVII is the result of an addition of the chemical shift value of corresponding carbon atom in the non-substituted 1-boraadamantane complex (XI) to the corresponding increment value which is conditioned by the presence of the chlorine atom at C(4). Results of these

TABLE 2

CALCULATED AND EXPERIMENTAL VALUES OF THE ^{13}C NMR CHEMICAL SHIFTS IN THE COMPOUND XVII



(XVIIa, X = H, Y = Cl;
XVIIb, X = Cl, Y = H)

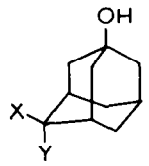
Chemical shift (ppm)	C(2,9)	C(3,5)	C(4)	C(6,10)	C(7)	C(8)
XVIIa (calcd.)	33.6	40.6	70.7	33.6	31.6	33.2
XVIIb (calcd.)	26.4	40.6	70.7	40.9	32.2	33.2
XVII (experimental) [a]	32.8	39.9	73.2	33.1	31.8	32.8

[a] In $CDCl_3$ solution, relative to TMS.

calculations are represented in Table 2. Hence it follows that XVII exists in the *trans*-isomer form XVIIa *.

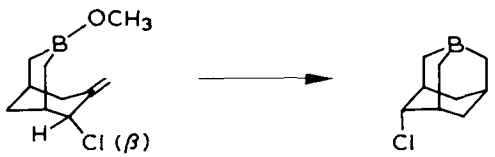
Similar calculations with the ^{13}C NMR chemical shifts of tetrahydrofuran-4-chloro-1-boraadamantane (XIII) ** and triethylamine-4-chloro-1-boraadamantane (XV) *** indicate these compounds to be also *trans* isomers.

The ^{1}H NMR spectrum of 4-chloro-1-hydroxyadamantane (XVIII) (previously prepared by us [5]), recorded on a 'Bruker WH-360' instrument, contains two multiplets from a proton at C(4) (α-proton) centered at 4.27 (0.9 H) and 4.19 ppm (0.1 H). Analogously to 1,4-dichloroadamantanes [16], it may be assumed that the downfield multiplet pertains to the α-proton in *trans*-4-chloro-1-hydroxyadamantane (XVIIIa), whereas the upfield signal corresponds to the α-proton in the *cis* isomer (XVIIIb). Hence, the mixture under study contains mainly the *trans* isomer XVIIIa.



(XVIIIa, X = H, Y = Cl;
XVIIIb, X = Cl, Y = H)

We made use of the above data on the ^{1}H and ^{13}C NMR spectra in the investigation of the reaction of triallylborane with propargyl chloride. Hydroboration of compound XII produces 4-chloro-1-boraadamantane (XIX). The C(6) atom remains unaffected in this transformation, consequently, the chlorine in compound XIX can be in the *trans* (4*e*) position (*e* with respect to the boron-containing cycle) only in that case when it occupies the 6β (*exo*) position. Direct conversion of the



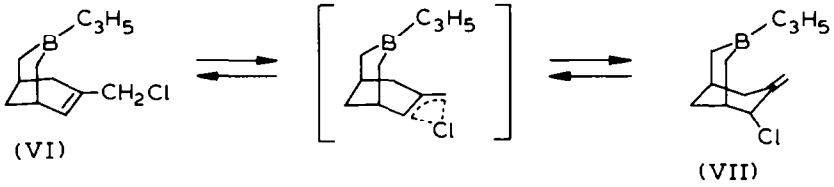
(XII) (XIX)

compound V to VII, previously assumed [2–4], is not a stereospecific process, as was shown by the inspection of molecular models. In other words, the formation of both possible stereoisomers of compounds VII, XII, and XVII can take place with the same probability. Alternatively, the formation of the *exo* isomer VII in the course of chloro-allylic rearrangement is predominant.

* In the chemical literature 1,4-disubstituted adamantanes are usually named either *cis*(*trans*) or 1,4*a*(4*e*) (*a* and *e* relative to the most substituted cycle) [15].

** Data for the 1-boraadamantane tetrahydrofuran complex are taken from the literature [14].

*** The ^{13}C NMR chemical shifts of triethylamine-1-boraadamantane (XIV) are as follows: 30.6 (broadened s, B–CH_2), 33.7 (d, C–H), 40.4 (t, CH_2) and signals of Et_3N, 10.0 (q, CH_3), 48.5 (t, N–CH_2).



(VI) (VII)

Thus, the NMR spectroscopy data substantiate an assumption that in the reaction of triallylborane with propargyl chloride the compound VII is formed by the isomerisation of VI rather than by the cyclization of V.

Experimental

All manipulations with organoboron compounds were carried out under dry argon atmosphere. IR spectra were recorded on a UR-20 spectrometer. ^{1}H NMR spectra were obtained on Varian DA 60-IL, Tesla BS-497 (100), Bruker WM-250, and Bruker WH-360 instruments (with TMS and HMDS as internal standards). ^{13}C NMR spectra were recorded on Bruker Physik WP-60 (15.08 MHz) and Bruker WM-250 (62.89 MHz) spectrometers. All chemical shifts are presented relative to TMS in ppm.

7-Chloromethyl-3-allyl-3-borabicyclo[3.3.1]non-6-ene (VI)

40.8 g (0.3 mol) of triallylborane (II) (with traces of phenothiazine) were placed in a flask fitted with a dropping funnel, thermometer and a condenser, whereupon were added at 20°C 22.7 g (0.3 mol) of propargyl chloride (I). After 25 days, 8.6 g of the reaction mixture (containing 17% of V by ^{1}H NMR) were heated to 120°C during 10 min and, for an additional 45 min, from 120 to 130°C. Distillation gave 7.4 g (86%) of VI, b.p. 79–79.5°C (2 mm Hg) with an admixture of VII (~ 3%, ^{1}H NMR).

Isomerisation of VI to 6-chloro-7-methylene-3-allyl-3-borabicyclo[3.3.1]nonane (VII)

a) 6.9 g of freshly distilled VI were heated for 2 h at 120–128°C (oil bath). Distillation afforded 6.2 g (90%) of a mixture containing ~ 18% of VII.

b) 6.3 g of freshly distilled VI were heated for 7 h at 125–130°C. Distillation yielded 5.3 g (84%) of a mixture of VI and VII with a content of VII of ~ 58%.

7-Chloromethyl-3-allyl-3-borabicyclo[3.3.1]non-6-ene (VI) and 6-chloro-7-methylene-3-allyl-3-borabicyclo[3.3.1]nonane (VII)

In a flask fitted with a stirrer, dropping funnel, thermometer and a condenser were placed 41.5 g (0.31 mol) of triallylborane (II), and then at 125–130°C (oil bath) were added during 2 h 23.2 g (0.31 mol) of propargyl chloride (I). The reaction mixture was heated at the same temperature for a further 1 h. Distillation gave 49 g of a mixture of VI and VII with a content of the latter compound of ~ 25%, b.p. 89–93°C (3 mm Hg), n_D^{20} 1.5160 (lit.data: [2,4]).

3-Methoxy-7-chloromethyl-3-borabicyclo[3.3.1]non-6-ene (VIII)

The title compound and its mixture with 3-methoxy-6-chloro-7-methylene-3-borabicyclo[3.3.1]nonane (XII) were obtained according to a reported method [4] from the bicyclic compounds VI and VII.

Hydroboration of VIII with $BH_3 \cdot THF$ in THF with a (VIII): B–H ratio of 1 : 3

a) At 0°C. To a solution of 5.3 mmol of BH_3 in 24 ml of THF was added 1 ml (1.055 g, 5.3 mmol) of VIII. To determine a content of $\rangle$BH in the reaction mixture, aliquot samples were periodically introduced with the aid of a syringe via a septum inlet into the hydrolyzing mixture (H_2O : glycerol : THF = 1 : 1 : 1) which was placed in a two-necked flask equipped with a magnetic stirring bar. Hydrogen evolved was collected in a gas burette.

b) At 20°C. Experiments were carried out as above. The results are presented in Table 1. Previous data on the hydroboration of VIII [5] were based on an incorrect method for the determination of the extent of hydroboration.

Pyridine-1-boraadamantane (XI)

a) 5.3 g (5 ml, 26.6 mmol) of the ether VIII were added at 20°C to a tetrahydrofuran solution of 26.6 mmol BH_3. The next day, low-boiling substances were distilled off at 20°C (1 mmHg), and then to the residue were added sequentially 20 ml of abs. hexane, 4.2 ml of abs. pyridine, and 15 ml of ethanol to decompose excess $\rangle$B–H and to recrystallize the complex XI. 2.8 g (50% of pyridine-1-boraadamantane (XI) were obtained, m.p. 161–164°C (lit.data: [17]). Evaporation of the filtrate and two-fold crystallization from ethanol gave an additional 0.9 g of XI, m.p. 162–165°C. The total yield of XI is equal to 3.7 g (65%).

In the filtrate was detected Cl^- (11 mmol). Apparently, BCl_3 formed interacts with pyridine to produce the complex $BCl_3 \cdot C_5H_5N$ which forms $Py \cdot HCl$ and $(EtO)_3B$ under the action of C_2H_5OH.

b) In a flask fitted with a stirrer, dropping funnel, thermometer, and a condenser were placed 10 ml of abs. decane, to which was added a mixture of VIII (5 g, 25.2 mmol), $Et_3N \cdot BH_3$ (2.9 g, 25.2 mmol), and Et_3N (0.42 g, 4.2 mmol) at 130–138°C during 30 min. During the addition, the temperature rose spontaneously to 147°C. The mixture was kept for 10 min at 130°C and then cooled to 20°C. After adding to the mixture at 20°C 2 g (25.2 mmol) of abs. pyridine, the precipitate formed was twice crystallized from abs. ethanol to yield 3.7 g (68%) of XI, m.p. 162–165°C.

Hydroboration of a mixture of VIII and XII

To 6.15 g (31 mmol) of a mixture of VIII and XII (2 : 3) in 10 ml of abs. THF were added 2.2 g (32 mmol) of $Et_4B_2H_2$ in 5 ml of abs. THF. The reaction temperature increased spontaneously to 56°C. The reaction mixture was kept at 40–50°C, while stirring, for 2 h. After cooling to 20°C, low-boiling substances was distilled off in vacuum (1 mmHg), and to the residue 8 ml of abs. pentane were added. The mixture was then cooled to −78°C; the precipitate formed was washed with pentane (4 × 2.5 ml) and maintained under 1 mmHg vacuum to afford 4.1 g of XIII (54% or, based on the isomer XII, 90%), m.p. 46–49°C (lit.data: [5]). ^{13}C NMR spectrum ($CDCl_3$): 72.29 doublet (C(4)), 40.71 doublet (C(3,5)), 33.05 triplet (C(6,10)), 28.7 broadened signal ($B–CH_2$), 32.81 doublet (C(7)) and signals of THF, 68.88 ($CH_2–O$), 24.88 triplet (CH_2).

After distilling off the volatile substances in vacuum, to the filtrate were added 5 ml of abs. THF and 11.5 ml of a solution of $BH_3 \cdot THF$ in THF (14 mmol of BH_3). After boiling for 5 h and subsequent distilling off volatile substances, 2.8 g (28 mmol) of Et_3N were added to the residue. Distillation yielded 1.6 g of XIV (23%, or 60% based on the isomer VIII), b.p. 101–105°C (2 mmHg) (lit.data: [9,18]). The structure of XIV was confirmed by its ^1H NMR spectrum.

Triethylamine-4-chloro-1-boraadamantane (XV)

To a solution of 2.7 g (11.1 mmol) of XIII in 6 ml of abs. ether, 1.13 g (11.1 mmol) of Et_3N were added; the reaction temperature increasing from 20 to 35°C. To the mixture were then added 5 ml of abs. ether and the mixture was allowed to stand overnight at −78°C. The ethereal layer was decanted, and the crystals formed were washed on a filter with ether (2 ml) and abs. hexane (15 ml). Keeping the crystals in oil pump vacuum gave 1.6 g (54%) of XV, m.p. 74–77°C (fast heating in a sealed capillary). An additional 1 g of XV (m.p. 73–77°C) was obtained from the filtrate by evaporation with subsequent washing with hexane. The total yield of XV is equal 87%. Found: C, 66.47; H, 10.75, Cl, 13.20; B, 4.14. $C_{15}H_{29}BClN$ calcd.: C, 66.80; H, 10.84; Cl, 13.15; B, 4.01%. ^1H NMR ($CDCl_3$, ppm): multiplets of adamantane protons: 0.73 and 0.84 ($B-CH_2$), 1.28, 1.36, 2.06, 2.12, 2.28, 4.34 (CHCl); 1.17 triplet ($J = 7$ Hz, $N-CH_2-CH_3$), 2.74 quartet ($J = 7$ Hz, $N-CH_2$) ^{13}C NMR ($CDCl_3$, ppm): 73.41 doublet (C(4)), 40.60 doublet (C(3,5)), 33.37 triplet (C(6,10)), 32.58 doublet (C(7)), 30.28 broadened signal ($B-CH_2$) and signals of carbon atoms of Et_3N: 48.94 triplet ($N-CH_2$), 9.96 quartet (CH_3).

4-Chloro-1-boraadamantane etherate (XVI)

To a solution of 4.2 g (15.6 mmol) of XV in 20 ml of abs. ether at 10–15°C were added 7.9 ml of an ethereal solution of HCl (15.6 mmol). After 45 min, 10 ml of pentane were added, the precipitate was filtered off and washed with abs. pentane. Removal of volatile substantces by vacuum distillation gave 3.2 g (85%) of XVI as a viscous yellowish substance. Found: C, 64.60; H, 9.89; B, 5.19; Cl, 13.44. $C_{13}H_{24}BClO$ calcd.: C, 64.35; H, 9.97, B, 4.46, Cl, 14.61%. ^1H NMR ($CHCl_3$, ppm): signals of the 1-boraadamantane nucleus protons (0.55–2.2) with multiplets centered at 0.82 ($B-CH_2$), 1.95, 2.22, 4.23 (CHCl); signals of C_2H_5O group: 3.77 quartet ($J = 7.5$ Hz, CH_2O), 1.19 triplet ($J = 7.5$ Hz, CH_3).

^1H NMR spectrum of 1-hydroxy-4-chloroadamantane (XVIII)

4.19 (0.1 H) and 4.27 (0.9 H) multiplets (α-proton at C(4), J = 1.5 Hz), 1.24 singlet (OH); signals of the remaining adamantane protons: 1.41 and 1.44 two quintets, 1.57, 1.65 and 1.70 broadened signals, 1.76 and 1.83 doublets ($J = 3$ Hz); 2.13, 2.18, 2.21, and 2.31 ppm broadened signals.

^1H NMR spectrum of pyridine-4-chloro-1-boraadamantane (XVII)

This spectrum was recorded on a Bruker WM-250 spectrometer (in $CDCl_3$): signals of adamantane protons at 0.75, 0.85 ($B-CH_2$), 1.40, 1.45, 2.17, 2.22, 2.39, multiplet at 4.42 (CHCl), and signals of pyridine protons.

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