Interaction of *Z*-1,2-borylphosphinoalkene with ketenimine

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N-Phenyl-*C*,*C*-diphenylketenimine adds to *Z*-1-butyl-1-dibutylboryl-2-diphenylphosphino-2-phenylethene to give 2,2,3-tributyl-6-diphenylmethylideno-1,4,5,5-tetraphenyl-1-aza-5-phosphonia-2-boratacyclohex-3-ene, which rearranges into 4,6-dibutyl-2-diphenylphosphino-1,1,3,5-tetraphenyl-3-aza-4-boradeca-1,5-diene upon heating. Its crystal structure has been established by X-ray analysis.

Key words: *Z*-1,2-borylphosphinoalkene; *N*-phenyl-*C*,*C*-diphenylketenimine; 4,6-dibutyl-2-diphenylphosphino-1,1,3,5-tetraphenyl-3-aza-4-boradeca-1,5-diene, X-ray analysis.

In continuation of our series of studies on the interaction of *Z*-1,2-borylphosphinoalkenes with heterocumulenes[1–3] we have investigated the reaction of 1-butyl-1-dibutylboryl-2-diphenylphosphino-2-phenylethene (**1**) with *N*-phenyl-*C*,*C*-diphenylketenimine. On the basis of data previously obtained,[1–3] one could expect ketenimine to enter into the [4+2]-cycloaddition reaction at the C=N bond, while the [4+2]-cycloaddition product should undergo 1,2-anionotropic migration of the butyl group from the B atom to the α-C atom with the formation of a bicycle.[3]

Indeed, borylphosphinoethene **1** easily reacts with ketenimine in benzene at 20 °C. An orange crystalline substance **2** is isolated from the reaction mixture. According to elemental analysis it is a 1 : 1 adduct. It is also evidenced by the ratio of the integral intensities of the phenyl and butyl proton signals in the ^{1}H NMR spectrum of compound **2**. Its mass spectra contain a peak with *m/z* 737, due to the $[M]^+$ ion of the molecule that is a 1 : 1 adduct of borylphosphinoethene and ketenimine. This assignment is proved by a precisely measured value of *m/z* for the peak in question (737.4330), which is in good agreement with that calculated for its empirical formula $C_{52}H_{57}BNP$ (737.4321). The elemental composition was calculated for the molecular ion containing the most widespread isotopes. The main directions of decomposition of this ion are the elimination of the C_4H_8 molecule or that of the C_4H_9 radical and elimination of the Ph_2C_2NPh molecule, yielding ions with *m/z* 412 $[M-C_4H_8-Ph_2C_2NPh]^+$ and 411 $[M-C_4H_9-Ph_2C_2NPh]^+$. The subsequent fragmentation of the above ions with elimination of the C_4H_8 molecule or C_4H_9 radical results in ions with *m/z* 355 and 354; an intense peak of the $[Ph_2C_2NPh]^+$ ion with *m/z* 269 is also present.

Bands corresponding to vibrations of the P=O and C=N bonds are absent in the IR spectrum of compound **2**. There is a band of middle intensity at 1534 cm^{-1}, which might correspond to vibrations of the endocyclic C=C bond, which is characteristic of 1-hetero-5-phosphonia-2-boratacyclohex-3-enes.[2,3] However, only one signal at 30.6 ppm is observed in the ^{31}P NMR spectrum of adduct **2**, which is not typical of adducts of borylphosphinoalkenes with heterocumulenes of both 1-hetero-5-phosphonia-2-boratacyclohex-3-ene (−4 to −15 ppm) and 2-aza-4-phosphonia-1-boratabicyclo[3.1.0]hexane (12—4 ppm) series.[1–3] For adduct **2** the structure of six-membered cyclic betaine seems to be more likely.

Adduct **2** is thermally unstable: it transforms into a white crystalline substance **3** upon heating its solution in benzene with small quantities of acetonitrile during 1 h. This transformation proceeds much more slowly in pure benzene and is completed in 8 h. As follows from the elemental analysis and ^{1}H NMR spectroscopy data, compound **3** is a structural isomer of **2**; its IR spectrum is similar to the spectrum of **2**, however the band at 1534 cm^{-1}, corresponding to vibrations of the C=C bond in the P—C=C—B fragment, is absent; several differences are also observed in 600—1600 cm^{-1} region. In the ^{31}P NMR spectrum of **3** a signal with 5.34 ppm is observed. According to X-ray analysis data, compound **3** is 4,6-dibutyl-2-diphenylphosphino-1,1,3,5-tetraphenyl-3-aza-4-boradeca-1,5-diene. It is likely that its

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[†] Deceased in 1995.

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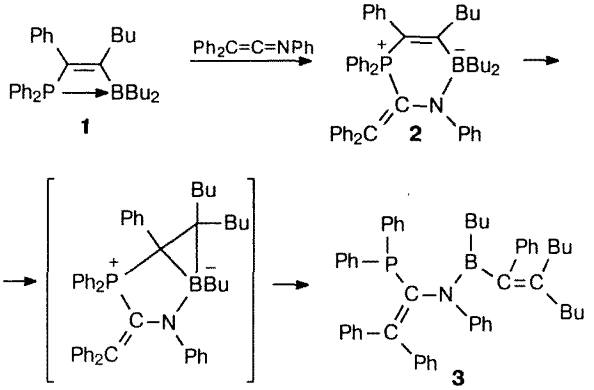
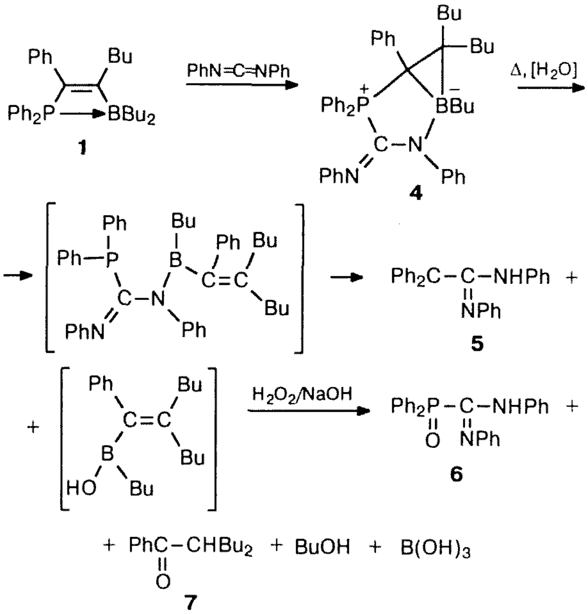
formation includes the [4+2]-cycloaddition of ketenimine to borylphosphinoethene **1**, the rearrangement of the monocyclic betaine **2** into a bicyclic one and decomposition of the phosphonium salt with the opening of the three-membered cycle by cleavage of the B—CBu$_2$ bond (Scheme 1).

Scheme 1



The suggested scheme of formation of diene **3** is confirmed by thermolysis of 1,6,6-tributyl-2,4,4,5-tetraphenyl-6-phenylimino-2-aza-4-phosphonia-1-boratabicyclo[3.1.0]hexane (**4**) — the product of interaction between compound **1** and diphenylcarbodiimide.[2] The thermolysis of compound **4** proceeds under more severe conditions than the transformation of betaine **2**. Thus, diphenyl(*N*,*N'*-diphenylformamidino)phosphine (**5**) was isolated from the reaction mixture upon heating compound **4** in xylene to 130—140 °C for 7 h; its constants and spectral parameters coincide with those reported in literature.[4] Upon thermolysis of compound **4** with subsequent oxidation of the reaction mixture by hydrogen peroxide in an alkaline medium phosphine oxide **6** was isolated, while the residue, according to the mass-spectroscopy measurements, was 2-butyl-1-phenylhexene-1-one (**7**). These decomposition products indicate to the same way of three-membered ring opening in compound **4** as during the formation of the diene **3** (Scheme 2). It is likely that borylaminomethynephosphine, formed as an intermediate in this case, is less stable towards hydrolysis than compound **3**.

Scheme 2



Diene **3** is a new representative of a poorly understood class of compounds containing the P—C—N—B fragment.[5] Despite the presence of three-coordinated P and B atoms, compound **3** in crystalline state is stable in the air and slowly oxidized in solutions. This might be due to its somewhat unusual spatial structure. According to the X-ray data (Fig. 1) the N and B atoms in molecule **3** have a trigonal planar coordination, which is characteristic of aminoboranes. The sums of bond angles are equal to 360° and 359.9°, respectively; the torsion angles C(31)—N(3)—B(4)—C(5), C(2)—N(3)—B(4)—C(5), and C(2)—N(3)—B(4)—C(41) are equal to 9.6°, 9.02°, and −3.0°, respectively; the N—B and B—C bond lengths are usual for compounds of such type. The planes of C(2)=C(1) and C(5)=C(6) bonds are rotated with respect to the plane of the aminoborane fragment by 67.9° and 73.3°, respectively. The diphenylphosphino group has a usual geometry; the P(1)—C(2) bond is somewhat longer than analogous bonds in the dimethyl ester of 2,3-bis(diphenylphosphino)fumaric acid[6] (1.861 and 1.842 Å, respectively). The molecular conformation is of interest because of the presence of two pairs of "eclipsed" phenyl cycles: the Ph group at the N atom and one of the Ph substituents at the P atom, as well as the second Ph substituent at the P atom and one of the Ph substituents of the methylidene fragment. Short contacts are observed between these phenyl cycles; the distances C(111)—C(31), C(112)—C(31), and C(112)—C(36) are 3.16, 3.35, and 3.31 Å, and C(121)—C(11) and C(126)—C(16) are 3.10 and 3.31 Å, respectively. The phenyl rings are somewhat shifted with respect to each other; for instance, C(13) atom is almost above the center of the opposite cycle. An analogous position of the approached planar fragments was observed for 5,6-benzo-4-diphenylphosphino-2-phenyl-1,3,2-dioxaborinane[7] and was explained by a dispersive interaction. In this case two stacking-interactions are realizes in one molecule, which might cause a rapid transformation of the intermediate bicyclic betaine and chemical stability of compound **3**.

Thus, as was suggested, the *Z*-adduct of borylphosphinoethene with ketenimine, owing to the nucleophilic N atom, adjacent to boron, undergoes 1,2-anionotropic migration of the butyl substituent from the B to the C atom. However the rearrangement is not completed at this point and an easy opening of the

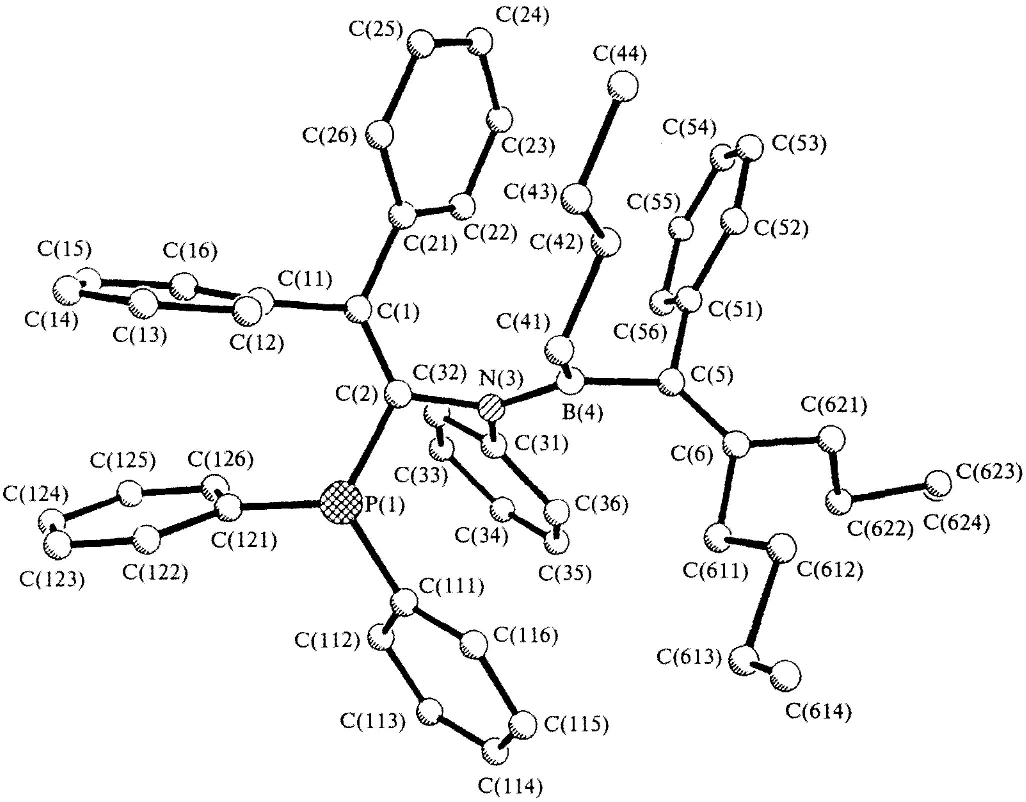


Fig. 1. Molecular structure of 4,6-dibutyl-2-diphenylphosphino-1,1,3,5-tetraphenyl-3-aza-4-boradeca-1,5-diene.

three-membered ring results in α-borylaminoalkenylphosphine. This is another of possible directions of the reaction of trialkylboranes with unsaturated compounds,[8] followed by the cleavage of the B—C bond formed at the first stage.

Experimental

The ^{31}P NMR spectra were recorded on a Bruker MSL-400 spectrometer (161 MHz). The ^{1}H NMR spectra (in DMSO-d_6) were obtained on a Varian T-60 at 34.5 °C ($SiMe_4$ as an external standard). IR spectra (suspensions in Vaseline oil) were recorded on a Specord M-80 spectrometer. Mass spectra (EI) were obtained on an MKh-1310 instrument with an SM-4 computer (energy of ionizing electrons — 70 eV, electron emission current — 30 μA, a direct inlet system was used). Precise measurements of the molecular ions' masses were carried out automatically using reference peaks of perfluorokerosine.

Compound **4** was obtained using the method described previously.[2]

2,2,3-Tributyl-6-diphenylmethylideno-1,4,5,5-tetraphenyl-1-aza-5-phosphonia-2-boratacyclohex-3-ene (2). In a solution of 0.5 g (1.07 mmol) of **1** in 2 mL of benzene 0.29 g (1.07 mmol) of *N*-phenyl-*C*,*C*-diphenylketenimine was dissolved with stirring. After ~24 h the solution was evaporated, and the crystals formed were recrystallized from acetonitrile. The yield of compound **2** was 0.55 g (70 %), m.p. 138 °C. Found (%): C, 84.05; H, 8.18; N, 1.97; P, 4.17. $C_{52}H_{57}BNP$. Calculated (%): C, 84.67; H, 7.73; N, 1.90; P, 4.21. IR, ν/cm^{-1}: 1534 (C=C). ^{1}H NMR, δ: 0.37—1.83 (m, 27 H, Bu); 6.30—8.00 (m, 30 H, Ph). ^{31}P NMR (C_6H_6), δ: 30.66. MS, *m/z* (I_{rel} (%)): 737 $[M]^+$ (0.58), 681 $[M-C_4H_8]^+$ (0.68), 680 $[M-C_4H_9]^+$ (0.58), 522 $[M-B(C_4H_9)(C_4H_8)NPh]^+$ (1.13), 468 $[M-Ph_2C_2NPh]^+$ (1.13), 412 $[M-C_4H_8-Ph_2C_2NPh]^+$ (32.29), 411 $[M-C_4H_9-Ph_2C_2NPh]^+$ (35.37), 355 $[M-C_4H_8-Ph_2C_2NPh-C_4H_9]^+$ (100), 354 $[M-2C_4H_9-Ph_2C_2NPh]^+$ (11.13), 269 $[Ph_2C_2NPh]^+$ (93.36), 186 $[Ph_2PH]^+$ (0.59), 185 $[Ph_2P]^+$ (2.90), 166 $[Ph_2C]^+$ (11.56), 108 $[PhP]^+$ (1.63), 91 $[PhCH_2]^+$ (0.93), 77 $[Ph]^+$ (1.70).

4,6-Dibutyl-2-diphenylphosphino-1,1,3,5-tetraphenyl-3-aza-4-boradeca-1,5-diene (3). A solution of 0.4 g (0.54 mmol) of compound **2** in 10 mL benzene was boiled for 8 h. The benzene was evaporated, and the crystals formed were recrystallized from an acetonitrile—DMFA (1 : 2) mixture. The yield of compound **3** was 0.11 g (27 %), m.p. 139—144 °C. Found (%): C, 84.06; H, 7.99; N, 1.90; P, 3.95. $C_{52}H_{57}BNP$. Calculated (%): C, 84.67; H, 7.73; N, 1.90; P, 4.21. ^{1}H NMR, δ: 0.49—1.70 (m, 27 H, Bu); 6.17—7.51 (m, 30 H, Ph). ^{31}P NMR (C_6H_6), δ: 5.35.

X-ray study of compound 3. The parameters of the unit cells and diffraction intensities of crystal **3** were measured on a Siemens P3/PC four-circle automatic diffractometer (λ(Mo-Kα), graphite monochromator, θ/2θ-scanning, $2\theta_{max} = 60°$) at 298 K. Crystals of compound **3** are colorless prisms, triclinic, $a = 1.674(4)$ Å, $b = 12.952(6)$ Å, $c = 15.169(5)$ Å, $\alpha = 92.93(2)°$, $\beta = 99.16(2)°$, $\gamma = 93.57°$, $V = 72255.4(16)$ Å^3, space group $P1$, $Z = 2$, $d_{calc} = 1.086$ g cm^{-3}.

The structure was solved by direct methods and refined anisotropically for non-hydrogen atoms. The final values of the

Table 1. Coordinates of non-hydrogen atoms ($\times 10^4$) and their thermal parameters ($U_{\mathrm{iso}}^{\mathrm{eq}} \times 10^3$) in structure **3**

Atom	x	y	z	$U_{\mathrm{iso}}^{\mathrm{eq}}$/Å^2
P(1)	2394(1)	2054(1)	5173(1)	42(1)
N(3)	2912(3)	2692(3)	7007(3)	37(1)
B(4)	2943(5)	1922(5)	7655(4)	42(1)
C(2)	2009(4)	2630(4)	6227(3)	37(1)
C(5)	3805(5)	2054(4)	8580(3)	43(1)
C(6)	4752(5)	1530(4)	8781(4)	47(1)
C(1)	871(5)	2760(4)	6277(4)	43(1)
C(111)	3987(4)	2225(4)	5310(3)	40(1)
C(112)	4648(5)	3119(5)	5095(4)	54(1)
C(113)	5846(6)	3096(6)	5165(5)	78(1)
C(114)	6407(6)	2238(6)	5459(5)	86(1)
C(115)	5796(6)	1384(6)	5657(5)	87(1)
C(116)	4590(6)	1371(5)	5588(4)	68(1)
C(121)	1959(4)	2957(4)	4324(3)	42(1)
C(122)	1441(5)	2485(5)	3488(4)	57(1)
C(123)	1089(6)	3106(6)	2769(4)	73(1)
C(124)	1213(6)	4130(6)	2888(5)	73(1)
C(125)	1690(5)	4617(5)	3705(5)	64(1)
C(126)	2060(5)	4031(5)	4408(4)	52(1)
C(31)	3752(4)	3570(4)	7059(3)	35(1)
C(32)	3377(5)	4525(4)	6855(4)	50(1)
C(33)	4185(6)	5382(5)	6901(5)	63(1)
C(34)	5369(5)	5262(5)	7175(5)	65(1)
C(35)	5735(5)	4311(5)	7355(4)	56(1)
C(36)	4947(4)	3458(4)	7302(3)	41(1)
C(41)	2041(5)	921(5)	7461(4)	61(1)
C(42)	1300(7)	692(6)	8172(5)	83(1)
C(43)	321(7)	−123(7)	7897(6)	107(1)
C(44)	−404(8)	−274(8)	8592(7)	150(1)
C(51)	3437(5)	2758(4)	9292(4)	47(1)
C(52)	2781(6)	2352(5)	9925(4)	69(1)
C(53)	2444(6)	2997(6)	10572(5)	87(1)
C(54)	2739(6)	4015(6)	10625(5)	91(1)
C(55)	3376(6)	4445(6)	10032(5)	89(1)
C(56)	3720(6)	3794(5)	9359(4)	66(1)
C(611)	5176(6)	837(5)	8062(4)	65(1)
C(612)	5182(8)	−264(6)	8228(6)	114(1)
C(613)	5483(9)	−959(8)	7392(8)	190(1)
C(614)	5409(9)	−1826(9)	7364(9)	292(1)
C(621)	5520(5)	1590(5)	9682(4)	60(1)
C(622)	6664(7)	2241(6)	9740(6)	103(1)
C(623)	7323(9)	2332(8)	10715(8)	187(1)
C(624)	7926(10)	3028(9)	11065(9)	335(1)
C(11)	−9(4)	2565(4)	5441(4)	43(1)
C(12)	−410(5)	1556(4)	5125(4)	52(1)
C(13)	−1214(5)	1364(5)	4350(5)	68(1)
C(14)	−1643(6)	2162(6)	3878(5)	76(1)
C(15)	−1271(5)	3176(5)	4192(5)	69(1)
C(16)	−490(5)	3350(5)	4978(4)	54(1)
C(21)	376(5)	3042(4)	7113(4)	44(1)
C(22)	956(5)	3686(4)	7831(4)	53(1)
C(23)	446(5)	3881(5)	8586(4)	65(1)
C(24)	−654(6)	3456(5)	8620(5)	75(1)
C(25)	−1248(6)	2839(5)	7932(5)	73(1)
C(26)	−750(5)	2635(5)	7167(4)	59(1)

Table 2. Major bond lengths (d) in structure **3**

Bond	d/Å
P(1)—C(2)	1.861(6)
P(1)—C(111)	1.836(5)
P(1)—C(121)	1.818(6)
N(3)—B(4)	1.434(8)
N(3)—C(2)	1.450(6)
N(3)—C(31)	1.445(6)
B(4)—C(5)	1.586(7)
B(4)—C(41)	1.602(8)
C(2)—C(1)	1.362(8)
C(5)—C(6)	1.336(8)
C(5)—C(51)	1.509(8)
C(6)—C(611)	1.539(9)
C(6)—C(621)	1.506(8)
C(1)—C(11)	1.499(7)
C(1)—C(21)	1.513(8)
C(33)—C(34)	1.399(9)
C(34)—C(35)	1.356(9)
C(35)—C(36)	1.383(8)
C(41)—C(42)	1.516(11)
C(42)—C(43)	1.499(10)
C(43)—C(44)	1.465(14)
C(51)—C(52)	1.421(9)
C(51)—C(56)	1.357(8)
C(52)—C(53)	1.377(11)
C(53)—C(54)	1.337(11)
C(54)—C(55)	1.374(12)
C(55)—C(56)	1.414(11)
C(611)—C(612)	1.461(10)
C(612)—C(613)	1.615(15)
C(613)—C(614)	1.119(15)
C(111)—C(112)	1.432(8)
C(111)—C(116)	1.395(9)
C(112)—C(113)	1.388(9)
C(113)—C(114)	1.382(11)
C(114)—C(115)	1.352(11)
C(115)—C(116)	1.395(10)
C(121)—C(122)	1.406(8)
C(122)—C(123)	1.412(10)
C(123)—C(124)	1.325(10)
C(124)—C(125)	1.378(9)
C(125)—C(126)	1.371(9)
C(31)—C(32)	1.370(8)
C(31)—C(36)	1.404(7)
C(32)—C(33)	1.403(8)
C(621)—C(622)	1.521(10)
C(622)—C(623)	1.550(14)
C(623)—C(624)	1.159(15)
C(11)—C(12)	1.394(8)
C(11)—C(16)	1.365(8)
C(12)—C(13)	1.384(8)
C(13)—C(14)	1.364(10)
C(15)—C(16)	1.381(8)
C(21)—C(22)	1.394(7)
C(21)—C(26)	1.401(8)
C(22)—C(23)	1.390(9)
C(23)—C(24)	1.375(9)
C(24)—C(25)	1.351(9)
C(25)—C(26)	1.399(10)

reliability factors: R = 7.56 % and R_w = 8.46 % for 3830 reflections with $F^2 \geq 6\sigma(F)$. All calculations were carried out on an IBM PC/AT computer using SHELXTL PLUS software.

The coordinates of non-hydrogen atoms are listed in Table 1, the main bond lengths and angles are given in Tables 2 and 3, respectively. The structure of compound **3** is shown in Fig. 1.

Thermolysis of 1,6,6-tributyl-2,4,4,5-tetraphenyl-3-phenylimino-2-aza-4-phosphonia-1-boratabicyclo[3.1.0]hexane (4). A solution of 1.5 g (2.2 mmol) of compound **4** in 3 mL of *p*-xylene was heated in a sealed ampule at 130—140 °C for 7 h. The reaction mixture was evaporated *in vacuo* at 100 °C, the residue was crystallized from acetonitrile, and the crystals were washed with acetonitrilebenzene mixture (4 : 1). The yield of compound **5** was 0.3 g (35 %), m.p. 131—134 °C. IR, ν/cm^{-1}: 3400 (N—H); 1608 (C=N). ^{31}P NMR (C_6H_6), δ: −11.7 (*cf.* Ref. 4).

A solution of 0.6 g of compound **4** in 1.5 mL of *p*-xylene was heated for 7 h in a sealed ampule at 130—140 °C. The

Table 3. Major bond angles (ω) in structure **3**

Angle	ω/deg	Angle	ω/deg
C(2)—P(1)—C(111)	105.3(2)	C(32)—C(31)—C(36)	119.5(5)
C(2)—P(1)—C(121)	105.6(3)	C(31)—C(32)—C(33)	120.0(5)
C(111)—P(1)—C(121)	101.8(3)	C(32)—C(33)—C(34)	119.8(6)
B(4)—N(3)—C(2)	121.0(4)	C(33)—C(34)—C(35)	119.9(6)
B(4)—N(3)—C(31)	124.4(4)	C(34)—C(35)—C(36)	120.8(5)
C(2)—N(3)—C(31)	114.6(4)	C(31)—C(36)—C(35)	120.1(5)
N(3)—B(4)—C(5)	121.9(5)	B(4)—C(41)—C(42)	116.9(5)
N(3)—B(4)—C(41)	119.2(4)	C(41)—C(42)—C(43)	115.2(6)
C(5)—B(4)—C(41)	118.8(5)	C(42)—C(43)—C(44)	112.9(7)
P(1)—C(2)—N(3)	116.3(4)	C(5)—C(51)—C(52)	120.8(5)
P(1)—C(2)—C(1)	119.2(4)	C(5)—C(51)—C(56)	121.8(6)
N(3)—C(2)—C(1)	123.0(5)	C(52)—C(51)—C(56)	117.4(6)
B(4)—C(5)—C(6)	124.7(5)	C(51)—C(52)—C(53)	120.6(6)
B(4)—C(5)—C(51)	115.6(5)	C(52)—C(53)—C(54)	120.7(8)
C(6)—C(5)—C(51)	119.4(5)	C(53)—C(54)—C(55)	121.0(8)
C(5)—C(6)—C(611)	120.7(5)	C(54)—C(55)—C(56)	119.0(7)
C(5)—C(6)—C(621)	124.9(5)	C(51)—C(56)—C(55)	121.3(7)
C(611)—C(6)—C(621)	114.4(5)	C(6)—C(611)—C(612)	115.4(6)
C(2)—C(1)—C(11)	118.4(5)	C(611)—C(612)—C(613)	112.6(8)
C(2)—C(1)—C(21)	126.5(5)	C(612)—C(613)—C(614)	122.3(12)
C(11)—C(1)—C(21)	115.1(5)	C(6)—C(621)—C(622)	115.0(6)
P(1)—C(111)—C(112)	125.9(4)	C(621)—C(622)—C(623)	110.3(7)
P(1)—C(111)—C(116)	116.1(4)	C(622)—C(623)—C(624)	127.2(11)
C(112)—C(111)—C(116)	117.8(5)	C(1)—C(11)—C(12)	120.6(5)
C(111)—C(112)—C(113)	119.0(6)	C(1)—C(11)—C(16)	122.3(5)
C(112)—C(113)—C(114)	121.1(7)	C(12)—C(11)—C(16)	116.9(5)
C(113)—C(114)—C(115)	120.7(7)	C(11)—C(12)—C(13)	121.3(6)
C(114)—C(115)—C(116)	120.0(7)	C(12)—C(13)—C(14)	120.5(6)
C(111)—C(116)—C(115)	121.4(6)	C(13)—C(14)—C(15)	119.1(6)
P(1)—C(121)—C(122)	114.5(4)	C(14)—C(15)—C(16)	119.3(6)
P(1)—C(121)—C(126)	128.1(4)	C(11)—C(16)—C(15)	122.6(6)
C(122)—C(121)—C(126)	117.4(5)	C(1)—C(21)—C(22)	124.6(5)
C(121)—C(122)—C(123)	119.7(6)	C(1)—C(21)—C(26)	117.8(5)
C(122)—C(123)—C(124)	120.2(6)	C(22)—C(21)—C(26)	117.6(5)
C(123)—C(124)—C(125)	121.5(7)	C(21)—C(22)—C(23)	120.8(5)
C(124)—C(125)—C(126)	119.4(6)	C(22)—C(23)—C(24)	120.0(6)
C(121)—C(126)—C(125)	121.7(5)	C(23)—C(24)—C(25)	120.9(7)
N(3)—C(31)—C(32)	119.5(4)	C(24)—C(25)—C(26)	119.9(6)
N(3)—C(31)—C(36)	121.0(5)	C(21)—C(26)—C(25)	120.8(5)

reaction mixture was evaporated *in vacuo*, and the residue was dissolved in 3 mL of THF. To the obtained solution 1 mL of 3 *N* NaOH solution and 1 mL of 30 % solution of H_2O_2 were added at 0 °C. The mixture was stirred for 3 h at 20 °C and 15 min at 50 °C; then it was extracted with a hexane—benzene (1 : 1) mixture. The organic layer was washed with water and dried with magnesium sulfate. Crystals were separated in the course of evaporation of the organic layer. They were washed with a hexane—acetonitrile mixture (5 : 1). Yield of compound **6** was 0.12 g (33 %), m.p. 137—140 °C. Found (%): C, 75.90; H, 5.67; N, 7.33; P, 7.10. $C_{25}H_{21}BN_2O$. Calculated (%): C, 75.75; H, 5.30; N, 7.07; P, 7.82. IR, ν/cm^{-1}: 3220 (N—H); 1634 (C=N); 1180 (P=O). ^{31}P NMR (C_6H_6), δ: 16.36.

After removing compound **6** the filtrate was evaporated, and a viscous yellow oil was obtained as a residue. The yield of compound **7** was 0.1 g (47 %). MS, *m/z* (I_{rel} (%)): 232 $[M]^+$ (47), 176 $[M-C_4H_8]^+$ (50.7), 133 $[M-C_4H_8-C_3H_7]^+$ (22.8), 105 $[M-PhCO]^+$ (100), 77 $[Ph]^+$ (26.2).

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