Interaction of *E*-2-dibutylboryl-1-phenyl-1-diphenylphosphinohex-1-ene with diphenylketene and benzoyl(phenyl)diazomethane

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A new betaine, 5,6,6-tributyl-2-diphenylmethylene-3,3,4-triphenyl-1-oxa-3-phosphonia-6-boratacyclohex-4-ene, and 1-[benzoyl(phenyl)methyleneamino]-4,5,5-tributyl-2,2,3-triphenyl-2-phospha-1-azonia-5-boratacyclopenta-1,3-diene were synthesized upon interaction of *E*-2-dibutylboryl-1-phenyl-1-diphenylphosphinohex-1-ene with diphenylketene and benzoyl(phenyl)diazomethane, respectively. The structure of the latter product was established by IR spectroscopy and X-ray structural analysis.

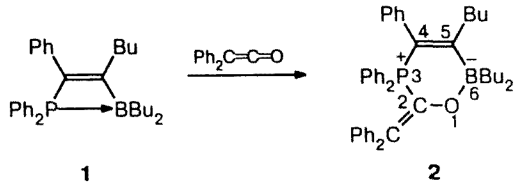
Key words: *E*-1,2-borylphosphinoalkenes, diphenylketene, benzoyl(phenyl)diazomethane, O,P,B- and N,P,B-heterocycles, X-ray structural study.

E-1,2-Borylphosphinoalkenes readily react with dienophiles and azacumulenes[1] to give cyclic P^+,B^--betaines, which are characterized by a reversible migration of the alkyl anion between the C_{sp^3} and B_{sp^3} atoms. With the aim of synthesizing new betaines of this type, we studied the interaction of *E*-2-dibutylboryl-1-phenyl-1-diphenylphosphinohex-1-ene (**1**) with diphenylketene and benzoyl(phenyl)diazomethane.

Results and Discussion

By analogy with the reactions reported previously,[1] two directions of [4+2]-cycloaddition (at the C=C or C=O bond of ketene) are the most probable for the reaction of **1** with diphenylketene, which was obtained by thermal decomposition of benzoyl(phenyl)diazomethane.[2] The reaction product is a white crystalline compound; the ^{31}P NMR spectrum of this compound shows a signal with the chemical shift of −15 ppm, which is close to the values of cyclic betaines of the 1-oxa- and 1-aza-3-phosphonia-6-boratacyclohex-4-ene types (from 0 to −10 ppm)[3,4] and differs substantially from the chemical shifts of bicyclic betaines of the 2-aza-4-phosphonia-1-boratabicyclo[3.1.0]hexane type (from 4.7 to 12 ppm).[4,5] In the mass spectrum of the compound synthesized, the peak at *m/z* 662, which corresponds to the adduct of **1** with diphenylketene, was observed; the absorption band of the carbonyl group was absent in the IR spectrum. Based on these data, it may be concluded that the compound obtained has the betaine structure, namely, 5,6,6-tributyl-2-diphenylmethylene-3,3,4-triphenyl-1-oxa-3-phosphonia-6-boratacyclohex-4-ene (**2**), *i.e.*, this compound is the product of [4+2] cycloaddition of borylphosphinohexene to diphenylketene at the C=O bond (Scheme 1).

Scheme 1



1 **2**

The 1,2-anionotropic migration of the butyl anion, which affords a bicyclic isomer, was reported for the analogs of betaine **2** (2-oxo- and 2-imino-1-aza-3-phosphonia-6-boratacyclohex-4-enes).[1,5] However, isomerization of **2** was not observed even upon prolonged boiling in benzene. No displacement of diphenylketene from **2** under the action of aldehydes and elemental sulfur occured as well.

In attempting to carry out reactions of **1** with 1,3-dipolar reagents, such as diazomethane and phenyl azide, azo compounds decomposed with the liberation of nitrogen, whereas compound **1** remained unchanged. However, in the reaction of benzoyl(phenyl)diazo-

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† Deceased.

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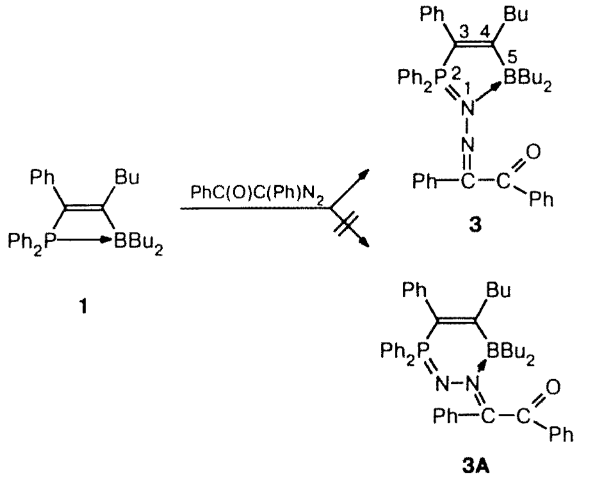
methane with **1**, the formation of **2** [2] or the products of [4+3]- or [4+2]-cycloaddition (as well as the addition at the P atom[6]) could be expected. When the reaction was carried out in benzene, a stable crystalline adduct (**3**) was isolated. In the mass spectrum of **3**, the precisely measured value of *m/z* of the $[M]^{+\cdot}$ molecular ion was 690.3892, which agrees well with the value of 690.3908 calculated based on the elemental composition, $C_{46}H_{52}BN_2OP$. In the IR spectrum, an intense absorption band of the P=N bond (1065 cm^{-1}) is somewhat shifted to the low-frequency region as compared to those of ordinary phosphaketazines (1090 cm^{-1})[7] and benzyl triphenylphosphoranediylhydrazone (1112 cm^{-1}),[8] which was obtained by the reaction of benzoyl(phenyl)diazomethane with triphenylphosphine. The ^{31}P NMR spectrum of compound **3** shows a single signal at δ 50.4. For comparison, the values of δ for aryliminotriphenylphosphoranes are in the region of about 14 ppm and higher.[9] Analogous shifts of the absorption bands in the IR spectra and the signals in the ^{31}P NMR spectra were observed previously for the derivatives of **1**, namely, for its oxide (**4**), sulfide (**5**), and selenide (**6**), compared to the corresponding derivatives of ordinary diarylalkenyl- and diarylalkylphosphines; these shifts were attributed to the formation of the coordination bond between the O, S, or Se atom and the B atom.[1] These facts altogether gave grounds to assign the structure of the compound with the intramolecular coordination B←N=P bond, namely, 1-[benzoyl(phenyl)methyleneamino]-4,5,5-tributyl-2,2,3-triphenyl-2-phospha-1-azonia-5-boratacyclopenta-1,3-diene (**3**) to the adduct obtained. However, the alternative structure **3A** could not be excluded (Scheme 2).

Scheme 2



Therefore, we studied the compound synthesized by X-ray structural analysis. Previous study covered the structures of the initial compound **1** and a number of its derivatives, which are the products of insertion at the P—B bond (2-dibutylboryl-1-phenylhex-1-enyl)diphenylphosphine oxide (**4**), (2-dibutylboryl-1-phenylhex-1-enyl)diphenylphosphine sulfide (**5**), (2-dibutylboryl-1-phenylhex-1-enyl)diphenylphosphine selenide (**6**), 5,6,6-tributyl-3,3,4-triphenyl-2-trichloromethyl-1-oxa-3-phosphonia-6-boratacyclohex-4-ene (**7**), the adduct of **1** with carbon disulfide (**8**), and 5,6,6-tributyl-2-methylthio-3,3,4-triphenyl-1-aza-3-phosphonia-6-boratacyclohexa-1,4-diene (**9**)).[10–12] The molecular structure of compound **3** is shown in Fig. 1; the bond lengths, bond angles, and torsion angles are given in Tables 1—3, respectively. As in the case of molecules **4—6**, the five-membered heterocycle of molecule **3** is planar to within 0.06 Å. Note that the twist about the double bond in molecule **3** is substantially larger than that in molecule **4** and is close to that observed in

Table 1. Selected bond lengths (*d*) in molecule **3**

Bond	*d*/Å	Bond	*d*/Å
P(1)—N(1)	1.649(3)	B(1)—C(25)	1.613(5)
P(1)—C(1)	1.758(3)	B(1)—C(29)	1.634(4)
P(1)—C(3)	1.791(3)	C(1)—C(2)	1.356(4)
P(1)—C(9)	1.801(3)	C(1)—C(15)	1.484(5)
O(1)—C(40)	1.218(4)	C(2)—C(21)	1.506(4)
N(1)—N(2)	1.386(3)	C(33)—C(40)	1.500(4)
N(1)—B(1)	1.649(4)	C(33)—C(34)	1.487(4)
N(2)—C(33)	1.279(4)	C(40)—C(41)	1.495(4)
B(1)—C(2)	1.621(5)		

Table 2. Selected bond angles (ω) in molecule **3**

Angle	ω/deg	Angle	ω/deg
N(1)P(1)C(1)	99.1(1)	C(2)B(1)C(29)	107.4(2)
N(1)P(1)C(3)	111.7(1)	C(25)B(1)C(29)	111.4(2)
C(1)P(1)C(3)	113.1(2)	P(1)C(1)C(2)	108.4(2)
N(1)P(1)C(9)	113.9(1)	P(1)C(1)C(15)	121.5(2)
C(1)P(1)C(9)	113.1(1)	C(2)C(1)C(15)	129.6(2)
C(3)P(1)C(9)	106.2(1)	B(1)C(2)C(1)	119.9(2)
P(1)N(1)N(2)	107.3(2)	B(1)C(2)C(21)	119.7(2)
P(1)N(1)B(1)	112.4(2)	C(1)C(2)C(21)	120.2(3)
N(2)N(1)B(1)	133.8(2)	N(2)C(33)C(34)	130.9(3)
N(1)N(2)C(33)	124.0(2)	N(2)C(33)C(40)	111.7(2)
N(1)B(1)C(2)	98.9(2)	C(34)C(33)C(40)	117.2(3)
N(1)B(1)C(25)	110.2(2)	O(1)C(40)C(33)	119.8(3)
N(1)B(1)C(29)	113.8(2)	O(1)C(40)C(41)	121.1(3)
C(2)B(1)C(25)	114.6(2)	C(33)C(40)C(41)	119.1(3)

Table 3. Torsion angles (τ) in molecule **3**

Angle	τ/deg	Angle	τ/deg
P(1)C(1)C(2)B(1)	−7.0	B(1)N(1)N(2)C(33)	42.1
C(1)C(2)B(1)N(1)	0.1	N(1)N(2)C(33)C(34)	10.8
C(2)B(1)N(1)P(1)	7.4	N(1)N(2)C(33)C(40)	−174.0
B(1)N(1)P(1)C(1)	−10.6	O(1)C(40)C(33)N(2)	143.4
N(1)P(1)C(1)C(2)	10.4	O(1)C(40)C(33)C(34)	−40.6
P(1)N(1)N(2)C(33)	−169.8		

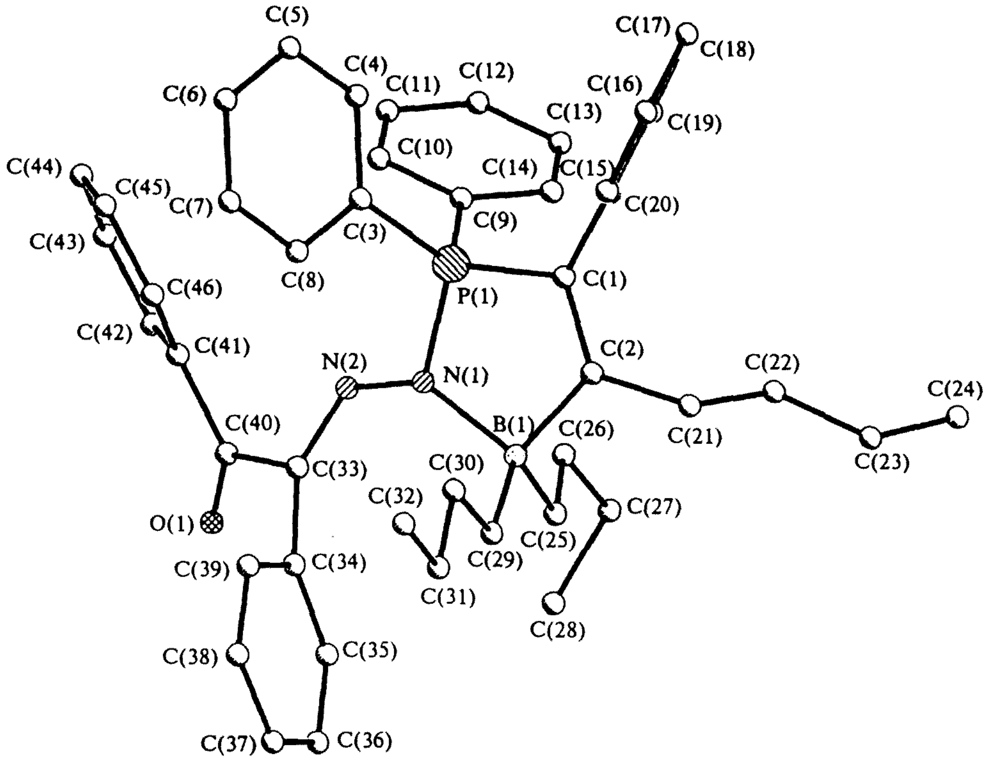


Fig. 1. Geometry of molecule 3. Hydrogen atoms are omitted.

molecule 5 (the P(1)C(1)C(2)B(1) torsion angles are −7.0°, −1.5°, and −9.3° in **3**, **4**, and **5**, respectively), which indicates that molecule **3** is sterically strained. This is also indicated by the nonzero values of other torsion angles in the boron-containing ring (see Table 3). As a whole, the conformation of the heterocycle can be described as a P envelope: the C(1)C(2)B(1)N(1) fragment is planar to within 0.001 Å, and the P(1) atom deviates from this plane by 0.02 Å. The dihedral angle between the planes of the base of the envelope and the flap is 10.4°. The P atom of molecule **3** is in a typical distorted tetrahedral coordination. The exocyclic P—C (Ph) bonds are equal in length to within the experimental error; these values agree with those observed in molecules **1** and **5**—**7**. The endocyclic P—C(1) bond in molecule **3** (1.758 Å) is somewhat shorter than those in the initial molecule **1** (1.804 Å) and in molecules **5** and **6** (1.781—1.796 Å) and is close to that found in molecule **4** (1.762 Å). The P(1)—N(1) distance (1.649 Å) fall within the upper limits of the P=N bond lengths in iminophosphoranes (1.556—1.645 Å),[13] and, on the other hand, this bond is in the range of single P—N bond lengths (1.629—1.689 Å).[13] However, a correlation was found between the P=N bond length and the P=N—C bond angle:[14] the bond shortens as the angle increases. In molecule **3**, the P(1)—N(1)—N(2) bond angle (107.3°) is smaller than the normal value of the angle at the sp^3 hybridized atom. Therefore, according to the correlation suggested,[14] it may be concluded that the P(1)—N(1) bond corresponds to the formally double P=N bond.

The N(1)—B(1) distance in molecule **3** (1.649 Å) is substantially larger than that in molecule **9** (1.555 Å) and is larger than the mean N—B bond length (1.549 Å).[15] The N(1) atom has a flattened trigonal-pyramidal configuration (the sum of the bond angles at the N atom is 353.5°). The direction of the N—B bond does not coincide with the "centroid"[16] of the N atom. Because of imperfect conditions of the overlap of the lone electron pair of the nitrogen atom and the unoccupied orbital of the boron atom, the N—B bond is elongated.

The C(1)=C(2) bond length in molecule **3** (1.356 Å) coincides with those found in molecules **1** (1.353 Å) and **5** (1.348 Å). The P—C and B—C bond lengths in molecule **3** are equal to those found in molecules **4** and **5**.

The high stability of compound **3**, which is favored by the presence of an intramolecular coordination bond, is worthy of note. Thus, compound **3** melts without decomposition (at higher temperature, resinification of **3** occurs). Isomerization of compound **3** through 1,2-sigmatropic migration of the butyl anion was not observed either.

Experimental

The ^{31}P NMR spectra were recorded on a Bruker WM-250 spectrometer (101.27 MHz, 85 % H_3PO_4 was used as the external standard); the ^1H NMR spectra were obtained on a Varian-T-60 spectrometer ($SiMe_4$ was used as the internal standard). The IR spectra were recorded on a Specord M-80 spectrometer (Nujol mulls). The mass spectra (EI) were ob-

Table 4. Atomic coordinates of nonhydrogen atoms (×10^4) and their equivalent isotropic thermal parameters (U_{eq}) in molecule **3**

Atom	x	y	z	$U_{eq}\cdot 10^3$/Å^2
P(1)	8613(1)	1084(1)	3930(1)	22(1)
O(1)	3869(2)	1754(1)	4447(2)	46(1)
N(1)	8084(2)	1342(1)	4934(2)	22(1)
N(2)	6798(2)	1439(1)	4312(2)	25(1)
B(1)	8890(3)	1267(1)	6457(3)	24(1)
C(1)	10038(3)	908(1)	5020(3)	24(1)
C(2)	10018(3)	997(1)	6236(3)	23(1)
C(3)	8958(3)	1345(1)	2659(2)	25(1)
C(4)	9120(3)	1181(1)	1559(3)	45(1)
C(5)	9446(4)	1382(1)	629(3)	60(2)
C(6)	9628(3)	1752(1)	780(3)	48(1)
C(7)	9488(3)	1917(1)	1870(3)	43(1)
C(8)	9159(3)	1715(1)	2816(3)	34(1)
C(9)	7463(3)	739(1)	3151(2)	24(1)
C(10)	6469(3)	814(1)	2031(3)	33(1)
C(11)	5564(3)	549(1)	1468(3)	40(1)
C(12)	5652(3)	209(1)	2011(3)	40(1)
C(13)	6619(3)	132(1)	3127(3)	34(1)
C(14)	7520(3)	397(1)	3700(3)	29(1)
C(15)	11100(3)	737(1)	4563(3)	26(1)
C(16)	10961(3)	398(1)	3970(3)	39(1)
C(17)	11947(4)	253(1)	3519(3)	49(1)
C(18)	13101(4)	438(1)	3661(3)	49(1)
C(19)	13255(3)	772(1)	4235(3)	47(1)
C(20)	12265(3)	921(1)	4680(3)	39(1)
C(21)	11088(3)	864(1)	7381(3)	30(1)
C(22)	10823(3)	478(1)	7775(3)	36(1)
C(23)	11779(4)	358(1)	9040(3)	52(1)
C(24)	11581(4)	−34(1)	9378(4)	73(2)
C(25)	7923(3)	1089(1)	7213(2)	27(1)
C(26)	6977(3)	795(1)	6524(3)	31(1)
C(27)	6165(3)	613(1)	7312(3)	43(1)
C(28)	5300(4)	869(1)	7800(4)	58(2)
C(29)	9635(3)	1626(1)	7208(3)	27(1)
C(30)	10581(3)	1827(1)	6620(3)	36(1)
C(31)	11282(3)	2143(1)	7423(3)	46(1)
C(32)	12233(4)	2333(1)	6848(5)	87(2)
C(33)	6146(3)	1679(1)	4737(3)	25(1)
C(34)	6576(3)	1947(1)	5799(3)	26(1)
C(35)	6162(3)	1939(1)	6913(3)	37(1)
C(36)	6557(4)	2209(1)	7828(3)	50(1)
C(37)	7328(4)	2492(1)	7618(4)	52(1)
C(38)	7703(3)	2507(1)	6507(3)	47(1)
C(39)	7330(3)	2236(1)	5599(3)	36(1)
C(40)	4741(3)	1697(1)	3932(3)	31(1)
C(41)	4434(3)	1636(1)	2507(3)	33(1)
C(42)	3241(3)	1486(1)	1864(3)	47(1)
C(43)	2930(4)	1435(1)	547(4)	64(2)
C(44)	3784(5)	1534(1)	−134(3)	68(2)
C(45)	4966(4)	1690(1)	492(3)	57(2)
C(46)	5295(3)	1743(1)	1814(3)	42(1)

tained on an MKh-1310 instrument equipped with an SM-4 computer. The energy of ionizing electrons was 70 eV; the current of the electron collector was 30 μA. The sample was introduced using a direct inlet system at 100 °C. All operations with **1** were carried out under an inert atmosphere.

5,6,6-Tributyl-2-diphenylmethylene-3,3,4-triphenyl-1-oxa-3-phosphonia-6-boratacyclohex-4-ene (2). A solution of diphenylketene (0.43 g, 2.2 mmol) in benzene (2 mL) was added to a solution of **1** (1.02 g, 2.2 mmol) in benzene (3 mL). After 24 h, the reaction mixture was concentrated *in vacuo*. The residue was crystallized from MeCN, and betaine **2** (0.85 g, 61 %) was obtained. It was purified by recrystallization from MeCN with slow cooling, m.p. 100—101 °C. Found (%): C, 83.73; H, 8.09; P, 4.41. $C_{46}H_{52}BOP$. Calculated (%): C, 83.38; H, 7.85; P, 4.68. IR, ν/cm^{-1}: 1542 (C=C). ^1H NMR (CD_2Cl_2), δ: 0.43—2.3 (m, 27 H, 3 Bu); 6.13—7.77 (m, 25 H, 5 Ph). ^{31}P NMR (C_6H_6), δ: −15. Mass spectrum, *m/z*: 662 $[M]^+$.

1-[Benzoyl(phenyl)methyleneamino]-4,5,5-tributyl-2,2,3-triphenyl-2-phospha-1-azonia-5-boratacyclopenta-1,3-diene (3). A solution of benzoyl(phenyl)diazomethane (0.24 g, 1.1 mmol) in benzene (3 mL) was added to a solution of **1** (0.5 g, 1.1 mmol) in benzene (2 mL). After 24 h, the reaction mixture was concentrated *in vacuo*. Crystallization of the residue from MeCN gave compound **3** in a yield of 0.42 g (56 %), m.p. 145—148 °C. Found (%): C, 80.40; H, 7.93; N, 4.10; P, 4.54. $C_{46}H_{52}BN_2OP$. Calculated (%): C, 80.00; H, 7.54; N, 4.06; P, 4.49. IR, ν/cm^{-1}: 1665 (C=O); 1590 (C=N); 1560 (C=C); 1065 (P=N); 860 (N—N). ^1H NMR (CD_2Cl_2), δ: 0.23—2.37 (m, 27 H, 3 Bu); 6.33—7.67 (m, 25 H, 5 Ph). ^{31}P NMR (C_6H_6), δ: 50.4.

X-ray structural study of compound 3. A single crystal of **3**, $C_{46}H_{52}BN_2OP$ (a colorless prism of dimensions 0.3×0.3×0.3 mm) was studied at 188 K on a four-circle automated Syntex $P2_1$ diffractometer (Mo-Kα radiation, graphite monochromator, θ/2θ scanning technique). The crystals are monoclinic, *a* 10.562(10), *b* 36.888(13), *c* 10.770(7) Å, β 106.04(2)°, *V* 4033(5) Å^3, space group $P2_1/n$, *Z* 4, mol. weight 690.7, d_{calc} 1.138 g cm^{-3}, μ(Mo-Kα) 0.104 mm^{-1}, *F*(000) 1480. Intensities of 4788 independent reflections were measured in the $2° < 2\theta < 50°$ range. The structure was refined by the direct method on an IBM PC/AT-386 computer using the SHELXTL PLUS program package and refined by the full-matrix least-squares method with anisotropic thermal parameters for nonhydrogen atoms (hydrogen atoms were fixed in the calculated positions throughout the refinement; their common isotropic thermal parameter was refined) to *R* 0.0403, R_w 0.0411 (3478 reflections with $I > 3\sigma(I)$ were used in the refinement). Atomic coordinates of nonhydrogen atoms are given in Table 4. The geometry of the molecule is shown in Fig. 1. The principal geometric parameters of the structure are given in Tables 1—3.

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