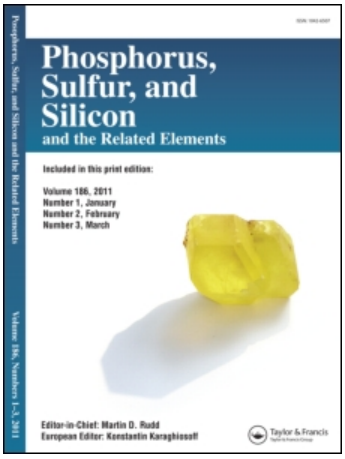

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FORMATION OF PHOSPHORUS-CONTAINING SPIRO CATIONS, STABILIZED BY THREE-CENTERED N → P ← N BOND, IN THE SERIES OF PHOSPHORYLATED α-AMINOKETONES

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FORMATION OF PHOSPHORUS-CONTAINING SPIRO CATIONS, STABILIZED BY THREE-CENTERED N → P ← N BOND, IN THE SERIES OF PHOSPHORYLATED α-AMINOKETONES

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Spiro cation $[Me_2N—CH=C(Bu\text{-}t)—O—P—O—(t\text{-}Bu)C=CH—NMe_2]^+ Cl^-$, stabilized by three-centered bond N → P ← N, is formed in the reaction of phosphorus trichloride with two molecules of t-$BuCOCH_2NMe_2$. According to X-ray analysis data, the structure of the compound obtained is close to a trigonal bipyramid with nitrogen atoms in axial positions (P-N 2.05 Å), and two oxygen atoms and lone electron pair of phosphorus in equatorial positions.

Key words: α-aminoketones, amonovinylphosphites, donor-acceptor N → P interaction, spirobi(dioxadiazonia-phospha-cyclopentene)chloride, X-ray analysis.

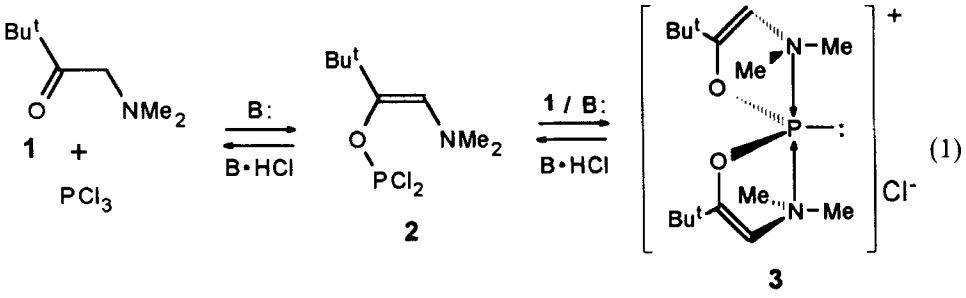
INTRODUCTION

A research on the complexation of polyhalogeno phosphorus (III) derivatives with tertiary amines dates back to Trost[1] and Holmes[2–4] as early as the late fifties and up to now it has been receiving a great deal of chemical attention as far as the nucleophilic substitution at the phosphorus atom is concerned. Studying the reaction of α-aminoketones with chlorophosphites, we have recently revealed the formation of donor-acceptor complexes just in the course of the reaction and have also elucidated the part of these intermediates in controlling the structure of final products, viz. aminovinylphosphites. In this connection, we took an interest in the reaction of aminoketones with polyhalogenophosphines so as to clarify the effect of aminoketones, potential bidentate ligands, on the reaction course, specifically, on a possibility for formation of polynuclear complexes in the systems under investigation.

RESULTS AND DISCUSSION

On the reaction of α-aminoketone **1** with PCl_3 in chloroform, run in the presence of triethylamine at room or somewhat lowered temperature, the aminoketone readily substitutes one or three chlorine atoms to give mono- and divinyl esters, **2** and **3**.[5,6]

Reaction of aminoketone **1** and phosphorus trichloride represents an equilibrium and can be presented in general case by the two-stage-equilibrium scheme:



(1)

In chloroform solution, the equilibrium is established within an hour, however, in the less polar solvents the same equilibrium is reached more slowly. Besides, divinyl ester **3** is formed even in the presence of large excess of PCl_3. By evaporation of PCl_3 from the reaction mixture the compound **2** disproportionates to give PCl_3 and product **3** till the formation of new equilibrium. For example, 12-hour boiling of mixture **2** and **3** in benzene (the starting **2** to **3** ratio of 64%:36%) leads to formation and accumulation of PCl_3 (the final PCl_3:**2**:**3** ratio is 36%:7%:55%).

Below are the most plausible schemes leading to the equilibrium concerned.

$$PCl_3 \overset{1}{\rightleftharpoons} PCl_3{\bullet}1 \overset{B:}{\underset{B:{\bullet}HCl}{\rightleftharpoons}} 2 \overset{1}{\rightleftharpoons} 2{\bullet}1 \overset{B:}{\underset{B:{\bullet}HCl}{\rightleftharpoons}} 3 \qquad (2)$$

$$PCl_3 \overset{1}{\rightleftharpoons} PCl_3{\bullet}1 \overset{1}{\rightleftharpoons} PCl_3{\bullet}(1)_2$$

$$B: \downarrow\uparrow B:{\bullet}HCl \qquad 2B: \downarrow\uparrow 2B:{\bullet}HCl \qquad (3)$$

$$\mathbf{2} \qquad\qquad \mathbf{3}$$

The first step is deduced from our information on the reaction of aminoketones with halogenophosphites[7] and implies the formation of 1:1 complexes of aminoketone with phosphorus halides followed by their conversion to products **2** and **3**. A rationale for the second step is provided by the evidence for the possible parallel formation of 1:1 and 1:2 complexes of PCl_3 and tertiary amines.[8] In this instance, products **2** and **3** should be formed from the corresponding complexes under the action of a base.

Taking into consideration thermodynamic data on the complexation of aminoketone **1** with phosphorus chlorides,[7] we suppose the former scheme to be preferable here.

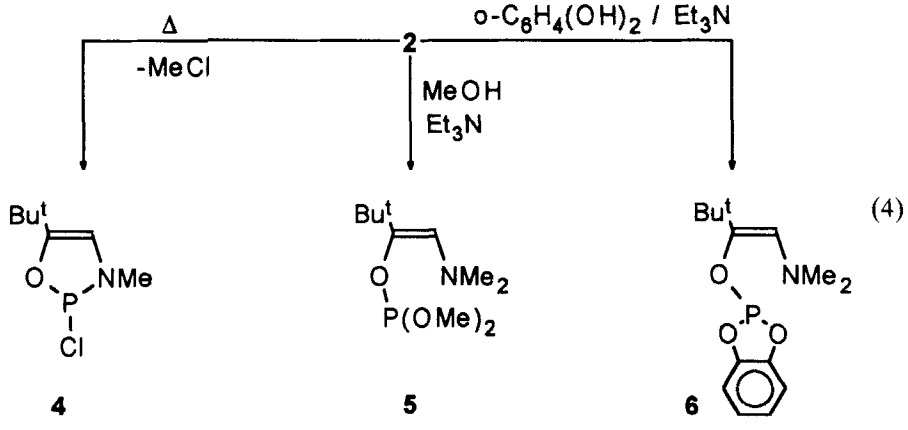
TABLE I

Dynamics of the interaction of aminoketone **1** with PCl_3 in pyridine solution at 20°C (the mole ratio aminoketone: PCl_3 = 1:1.2)

time	PCl_3	**2**	**3**
2 hours	69%	30%	1%
1 day	56%	40%	4%
8 days	26%	57%	16%
31 days	33%	49%	17%
43 days	44%	38%	18%

Knowing the regularities for the reaction of aminoketones with phosphorus chloride, we have been able to control its course in some cases. As the reaction of aminoketone **1** with PCl_3 and vinyl ester **2** runs via the N → P complex, the addition of foreign nucleophiles able of competing with the aminoketone allows the control over the formation rates for products **2** and **3**. For instance, pyridine drastically slows down the formation of compound **3** and slightly affects the first reaction stage thus allowing the isolation of vinyl ester **2**. Compound **3** being the most thermodynamically advantageous product, gradually accumulates in the reaction mixture on long standing (see Table I).

Phosphite **2** is a kinetically controlled product of the reaction of aminoketone **1** and PCl_3 and easily undergoes consequent conversions. Thus, on heating in pyridine or distillation, product **2** undergoes dealkylation and forms 2-chloro-3-methyl-5-*tert*-butyl-1,3,2-oxazaphospholene **4**.



4 **5** **6** (4)

Using reactions of dichlorophosphite **2** with methanol or catehol the vinyl ester **5** or **6** can be obtained.

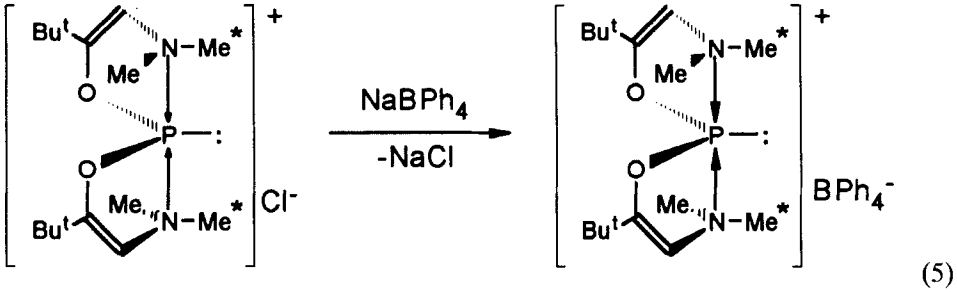
Among the products formed by the reaction of aminoketone **1** with PCl_3, the thermodynamically controlled one, viz. compound **3**, predominates regardless of the reagent ratio and the base used. This is presumably due to its advantageous chelate structure (cf. Reference 9). At aminoketone to PCl_3 ratio of 2:1, divinyl ester **3** is solely formed.

The spiro product **3** is a high melting substance, slightly soluble in non-polar solvents.[10] Its composition and structure have been determined from element analysis data, as well as IR- and NMR-spectra. N-Methyl groups show two types of signals in the NMR-spectra: singlet and doublet ones ($^3J_{PH}$ = 7.8 Hz, $^2J_{PC}$ = 38.4

Hz). This fact, as well as magnetic equivalence of other groups and the high field shift of the ^{31}P signal (δ_p 103 ppm) can be explained by the symmetric structure of the molecular fragments with Z-configuration of aminovinyloxy groups and the formation of a rigid spiro structure.

The great thermodynamic stability of the bicyclic structure is confirmed by NMR-data. On heating the solution of compound **3** in chloroform in a sealed tube up to 100°C, a weak down field shift (1.6 ppm) of the signal in the ^{31}P NMR-spectra has been detected. At the same time, there have been almost no changes in PMR-spectra.

Compound **3** is probably completely dissociated in solution with the P—Cl bond cleavage which is confirmed by the exchange of Cl^- anion by BPh_4^- anion. The spectral characteristics of the cation moiety of the molecule remain practically unchanged.



3 **7** (5)

δ_P=103 ppm
PMR:
Me*: 2.79 d, $^3J_{PH}$ 7.8 Hz
Me: 2.98 s

δ_P=103 ppm
PMR:
Me*: 2.14 d, $^3J_{PH}$ 7.8 Hz
Me: 2.35 s

Spiro compound **7** is supposed to be genetically related to a trigonal-bipyramidal structure, typical of hypervalent phosphorus compounds. In molecules of this sort, like PF_5, formed by a pentacoordinated phosphorus atom with ten electrons in its valence shell, three sp^2-hybridized valence phosphorus orbitals are involved in the formation of three normal two-centered bonds with equatorial ligands while the fourth p_z orbital participates in the two-electron three-centered bond with axial ligands. The remaining two valence electrons occupy the nonbonding orbital that involves two axial ligands.

Likewise, the trigonal-bipyramidal structure is inherent in hypervalent sulphur compounds, such as SF_4, having ten electrons in the sulphur valence shell, with the only difference that one of three sp^2-hybridized valence orbitals is occupied by a lone electron pair and forms no bond with an equatorial ligand.

As the valence shell of a tetracoordinated phosphorus atom includes ten rather than eight electrons, we should expect cation **7** to have a structure like that of SF_4, namely, with two linearly arranged axial ligands and two equatorial ones lying perpendicularly to them. This assumption is confirmed by X-ray analysis. The

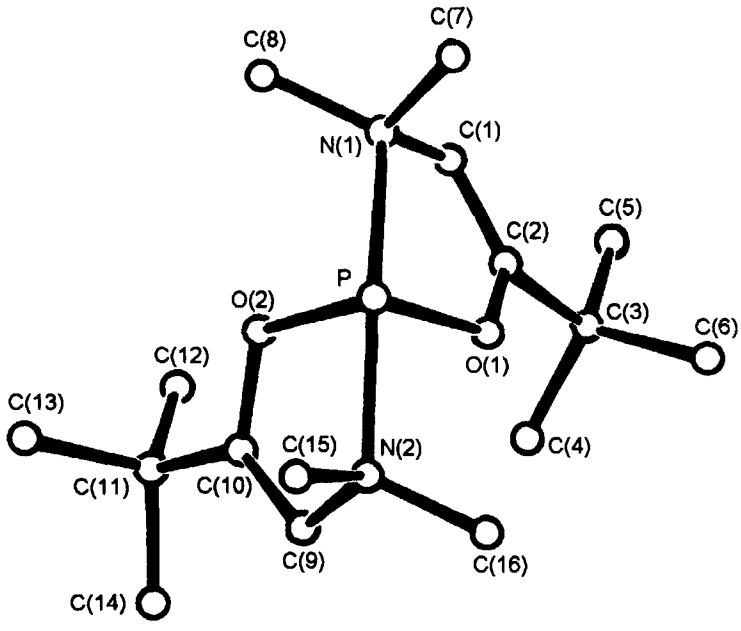


FIGURE 1 The general view of the cation **7** (H atoms omitted for clarity).

TABLE II

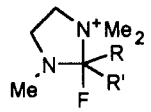
Selected structural parameters of the cation **7**

bond lengths (A°)		bond angles (°)	
P-O(1)	1.638(1),	O(1)PO(2)	102.21(7),
P-O(2)	1.641(1),	O(1)PN(1)	84.46(6),
P-N(1)	2.053(2),	O(1)PN(2)	86.89(7),
P-N(2)	2.056(2),	O(2)PN(1)	88.01(6),
O(1)-C(2)	1.396(2),	O(2)PN(2)	84.26(7),
O(2)-C(10)	1.392(2),	N(1)PN(2)	166.94(7),
N(1)-C(1)	1.434(2),	PO(1)C(2)	117.9(1),
N(2)-C(9)	1.443(3),	PO(2)C(10)	118.0(1),
C(1)-C(2)	1.311(3),	PN(1)C(1)	102.9(1),
C(9)-C(10)	1.314(3),	PN(2)C(9)	102.9(1),
B-C(17)	1.651(3),	N(1)C(1)C(2)	114.7(2),
B-C(23)	1.646(3),	O(1)C(2)C(1)	114.9(2),
B-C(29)	1.641(3),	N(2)C(9)C(10)	114.0(2),
B-C(35)	1.642(3),	O(2)C(10)C(9)	115.4(2),

general view of cation **7** is shown in Figure 1 and its basic geometrical parameters are presented in Table II.

The main structural peculiarity of the cation **7** is an unusual lengthening of the bonds P—N(1) 2.053(2) and P—N(2) 2.056(2) Å in comparison with both the range, characteristic for phosphonium cations, 1.59-1.64 Å,[11] and the length of a purely single P—N bond, 1.77 Å.[12] This is evidence for an essentially different (donor-acceptor) nature of the phosphorus-nitrogen bond in **7**. P—N Bond lengths in **7**

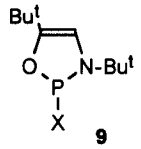
are close to the determined ones for cations **8a–c**, 1.991(3), 1.996(3), and 2.070(3) Å, having an ammonium group in axial positions of a bipyramidal structure.[13]



8

R = Ph, R' = F (a);
R = CH_2SiMe_3, R' = F (b);
R = Ph, R' =  (c)

The other structural characteristics of the 1,3,2-oxazaphospholenic fragments of compound **7** are similar to those of molecules **9a–c**.[14]



9

X = Cl (a), Br (b), I (c).

The P—O(1) and P—O(2) distances (see Table II) are in good agreement with the corresponding bond lengths for compounds **9** (1.621–1.656 Å). Similarly, the O(1)—C(2) and O(2)—C(10) distances coincide with the C—O bond lengths in **9** within experimental error limits. At the same time the bonds C(1)—C(2) (1.311(3) Å) and C(9)—C(10) (1.314(3) Å) are ~0.02 Å shorter whereas the bonds N(1)—C(1) (1.434(2) Å) and N(2)—C(9) (1.443(2) Å) are ~0.02 Å longer than the corresponding parameters in compounds **9a–c**. Evidently it can be explained by the absence of $n_N - \pi^*_{C=C}$ conjugation in compound **7** because the lone pair of the nitrogen atom is completely involved into N → P coordination. Just as in compounds **9a–c**, the five-membered heterocycles of cation **7** have an envelope conformation: the N(1)C(1)C(2)O(1) and N(2)C(9)C(10)O(2) bond systems are planar within the 0.010(2) and 0.017(2) Å, whereas the "corners" PO(1)N(1) and PO(2)N(2) form with them the dihedral angles of 20.3(2) and 20.9(2)° respectively (in the molecules (**9a–c**) these angles are equal to 12.8(7), 10.3(9) and 10.6(5)°, resp.).

To sum up, a pronounced tendency of α-aminoketones to form chelating ligands favours the stabilization of hypervalent phosphorus compounds with their trigonal-bipyramidal molecules containing a lone electron pair instead of an equatorial ligand.

EXPERIMENTAL

All experiments were conducted with exclusion of air and moisture under dry argon.

NMR: Bruker Jeminy 200, ^1H (200.13 MHz), ^{13}C (50.32 MHz), ^{31}P (81.02 MHz). Reference substances: ^1H, ^{13}C, TMS int., ^{31}P, 85% H_3PO_4 ext. All NMR spectra were recorded in $CDCl_3$ as a solvent. All chemical shift values are listed in p.p.m. The integration of all ^1H NMR spectra was consistent with the structure.

The IR spectra were measured in KBr, on a Carl Zeiss Infracord Spectrometer Model UR 10.

Crystal data for 14: $C_{40}H_{52}BN_2PO_2$, M = 634.7, monoclinic, a = 12.215(7), b = 9.976(4), c =

30.938(8) Å, β = 90.52(4)°, V = 3769.9 Å^3, Z = 4, d_c = 1.12 g/cm, spase group $P2_1/c$, μ = 8.8 cm^{-1}, F(000) = 1368.

Crystallographic measurements were made at 20°C using Enraf Nonius CAD-4 diffractometer operating in the $\omega/2\theta$ scan mode (the ratio of the scanning rates ω/θ = 1.2). The intensity data were collected within the range $2 \leq \theta \leq 56°$ using graphite monochromated Cu-K_α radiation (λ = 1.54184 Å). Intensities of 4899 unique reflections were measured. The structure was solved by direct methods and refined by full-matrix least squares techniques in the anisotropic approximation. In the refinement 4341 reflections with $I > 3\sigma(I)$ were used. About 60% of the hydrogen atoms were located in the difference Fourier maps, positions of the remaining atoms were calculated. All hydrogen atoms were included in the final refinements with the fixed positional and thermal (B_{iso} = 7 Å^2) parameters. Convergence was obtained at R = 0.049 and R_w = 0.078 (415 refined parameters; largest shift/esd

TABLE III

Fractional atomic coordinates and equivalent isotropic temperature factors B_{eq} (Å^2)

Atom	x	y	z	B_{eq}
P	0.76764(4)	0.19236(5)	0.35318(1)	4.10(1)
O(1)	0.7980(1)	0.2646(1)	0.39931(4)	4.43(3)
O(2)	0.7306(1)	0.0417(1)	0.36853(4)	4.34(3)
N(1)	0.9270(1)	0.1274(2)	0.35530(5)	4.48(3)
N(2)	0.6073(1)	0.2414(2)	0.36526(6)	5.27(4)
C(1)	0.9557(2)	0.1418(2)	0.40012(6)	5.13(5)
C(2)	0.8882(2)	0.2161(2)	0.42253(6)	4.70(4)
C(3)	0.8890(2)	0.2593(3)	0.46900(7)	6.18(5)
C(4)	0.7911(3)	0.1985(4)	0.49116(9)	10.45(9)
C(5)	0.9932(3)	0.2150(4)	0.4907(1)	11.1(1)
C(6)	0.8790(3)	0.4128(3)	0.47136(9)	8.66(8)
C(7)	0.9862(2)	0.2270(2)	0.32804(7)	5.52(5)
C(8)	0.9446(2)	-0.0101(2)	0.33816(8)	6.10(5)
C(9)	0.5723(2)	0.1350(2)	0.39354(7)	5.26(5)
C(10)	0.6370(2)	0.0297(2)	0.39357(6)	4.46(4)
C(11)	0.6230(2)	-0.1051(2)	0.41362(7)	5.29(5)
C(12)	0.7278(2)	-0.1483(3)	0.4365(1)	7.72(7)
C(13)	0.5965(3)	-0.2042(2)	0.37725(9)	7.16(6)
C(14)	0.5276(2)	-0.1005(3)	0.44556(9)	7.34(6)
C(15)	0.5523(2)	0.2308(3)	0.32226(9)	7.37(6)
C(16)	0.5902(2)	0.3778(2)	0.3830(1)	7.49(7)
C(17)	0.2630(1)	-0.4550(2)	0.38914(5)	3.90(4)
C(18)	0.3458(2)	-0.4034(2)	0.41523(6)	4.72(4)
C(19)	0.3883(2)	-0.4719(2)	0.45062(7)	5.64(5)
C(20)	0.3497(2)	-0.5954(2)	0.46110(7)	5.90(5)
C(21)	0.2688(2)	-0.6499(2)	0.43650(8)	6.28(5)
C(22)	0.2264(2)	-0.5815(2)	0.40129(7)	5.32(5)
C(23)	0.2914(2)	-0.4540(2)	0.30633(6)	4.17(4)
C(24)	0.2544(2)	-0.5624(2)	0.28187(7)	5.69(5)
C(25)	0.3211(2)	-0.6251(3)	0.25139(8)	6.91(6)
C(26)	0.4248(2)	-0.5819(3)	0.24460(7)	6.44(6)
C(27)	0.4649(2)	-0.4778(2)	0.26882(7)	5.72(5)
C(28)	0.3993(2)	-0.4158(2)	0.29878(6)	4.78(4)
C(29)	0.0869(2)	-0.3955(2)	0.33706(6)	4.40(4)
C(30)	0.0400(2)	-0.3869(3)	0.29636(7)	6.96(6)
C(31)	-0.0727(2)	-0.3877(4)	0.28953(8)	8.29(8)
C(32)	-0.1421(2)	-0.4004(3)	0.32267(9)	6.89(6)
C(33)	-0.1000(2)	-0.4084(2)	0.36328(8)	5.98(5)
C(34)	0.0128(2)	-0.4064(2)	0.37040(7)	5.00(4)
C(35)	0.2399(2)	-0.2184(2)	0.34516(6)	4.28(4)
C(36)	0.2652(2)	-0.1444(2)	0.30856(8)	5.86(5)
C(37)	0.2772(2)	-0.0032(2)	0.3099(1)	7.41(6)
C(38)	0.2667(2)	0.0645(3)	0.3485(1)	7.50(7)
C(39)	0.2397(2)	-0.0050(2)	0.38415(9)	6.82(6)
C(40)	0.2259(2)	-0.1427(2)	0.38254(7)	5.45(5)
B	0.2196(2)	-0.3810(2)	0.34444(7)	4.00(4)

after final cycle <0.35; the largest peak in the final difference map 0.51 e/Å^3). The weighting scheme $w = (\sigma^2F + 0.0016F^2)^{-1}$ was used. Corrections for the Lorentz and polarization effects but not for absorption were applied. All structural calculations were carried out with a PDP-11/23+ computer using the SDP-PLUS program package.[15] The final atomic coordinates and equivalent isotropic temperature factors are given in Table III. Full crystallographic data have been deposited at the Cambridge 1 Crystallographic Data Centre.[16]

1-Dimethylamino-2-dichlorophosphinoxy-3,3-dimethyl-1-butene (**2**): 0.12 Mol of 1-dimethylamino-3,3-dimethylbutanone-2 (**1**) was added dropwise to 150 ml of a pyridine solution, containing 0.25 mol of PCl_3. After 2 hours the reaction mixture was diluted with 300 ml of benzene, then the solution was separated from the precipitate and evaporated under reduced pressure. The viscous product was dried under oil-pump vacuum and identified using spectra analysis. Yield 87%; IR spectra (ν, cm^{-1}): 1670 (C=C); ^{1}H-NMR: 1.13 (s, Me_3C), 2.48 (s, Me_2N), 5.36 (d, HC=C, $^4J_{PH}$ 1.4 Hz); ^{31}P NMR: 160.

2,2'-Spirobi-(3,3,3',3'-tetramethyl-5,5'-ditert.-butyl-1,1'-dioxa-3,3'-diazonia-2-phospha-4-cyclopentene) chloride (**3**): 0.05 mol of PCl_3 was slowly added to the solution of 0.11 mol of aminoketone **1** and 0.11 mol of triethylamine in 300 ml of $CHCl_3$. After 12 hours, 300 ml of benzene were added. The solution was separated from the precipitate and then the solvent was evaporated under reduced pressure. The residue was dried in vacuo (0.05 mm, 40–50°C) till full evaporation of light constituents, then treated with 100 ml CH_2Cl_2. The solution was separated from the precipitate, the solvent evaporated and the product dried under oil-pump vacuum. The remaining viscous mass crystallized on standing during 10–20 days. Yield 84%, m.p. 159–160°C, IR spectra (paste in nujole, ν, cm^{-1}): 1670 (C=C); ^{1}H-NMR: 1.11 (s, Me_3C), 2.79 (d, MeN, $^3J_{PH}$ 7.8 Hz), 2.98 (s, MeN), 5.90 (d, CH=C, $^3J_{PH}$ 2.0 Hz); ^{13}C NMR: 26.7 (s, C$\underline{Me}_3$), 32.7 (s, $\underline{C}Me_3$), 43.1 (s, NMe), 47.0 d (NMe, $^2J_{PC}$ 38.4 Hz), 113.5 s (C=$\underline{CH}$), 159.8 d ($\underline{C}$=CH, $^2J_{PC}$ 15.2 Hz), ^{31}P NMR: 103.

$C_{16}H_{32}ClN_2O_2P$ (350.87)

Anal.: found: Cl 10.26, N 7.86, P 8.80%
calc.: Cl 10.11, N 7.99, P 8.83%

2-Chloro-3-methyl-5-tert. butyl-1,3,2-oxazaphospholene-4 (**4**): *Method (a).* Vinyl ester **2** was heated in a Claisen-vessel at 200°C in vacuo (10 mmHg), the distilled crude product was purified by distillation. Yield 68%. *Method (b).* The pyridine solution of vinyl ester **2** was boiled during 2 hours, then the solvent evaporated in vacuo, the residue extracted with benzene, the extract evaporated in vacuo and the remaining crude product purified by distillation. Yield 82%. The extraction residue was identified as methylpyridinium chloride. B.p. 75–76°C (10 mm Hg); IR spectra (ν, cm^{-1}): 1660 (C=C); ^{1}H-NMR: 1.14 (s, Me_3C), 3.00 (d, MeN, $^3J_{PH}$ 15.2 Hz), 5.78 (d, CH=C, $^3J_{PH}$ 5.8); ^{31}P NMR: 172.

$C_7H_{13}ClNOP$ (209.61)

Anal.: found: Cl 16.84, N 6.72, P 14.73%
calc.: Cl 16.92, N 6.68, P 14.78%

Interaction of vinyl ester **2** *with catechol:* The solution of 0.02 mol of catechol in 50 ml of CH_2Cl_2 was added to a solution of 0.02 mol of **2** and 0.05 mol of triethylamine. After 1 hour the solution was separated from the precipitate and evaporated under reduced pressure. The remaining product was purified by distillation. Yield 84%. Characteristics of this product with the one described previously[7] are identical.

1-Dimethylamino-2-(dimethoxy)phosphinoxy-3,3-dimethyl-1-butene (**5**): Interaction of vinyl ester **2** with methanol was carried out similarly to the above described reaction with catechol. Yield 78%; b.p. 68–70°C (0.1 mm Hg); IR spectra (ν, cm^{-1}): 1670 (C=C; ^{1}H-NMR: 1.09 (s, M_3C), 2.24 (s, Me_2N), 3.52 (d, MeO, $^3J_{PH}$ 8.5 Hz), 4.90 (d, CH=C, $^4J_{PH}$ 1.4 Hz); ^{31}P NMR: 123.

$C_{10}H_{22}NO_3P$ (235.26)

Anal.: found: N 5.93, P 13.10%
calc.: N 5.95, P 13.16%

2,2'-Spirobi-(3,3,3',3'-tetramethyl-5,5'-ditert.-butyl-1,1'-dioxa-3,3'-diazonia-2-phospha-4-cyclopentene) tetraphenylborate (**7**): The solution of 0.02 mol of sodium tetraphenylborate in 10 ml of CH_3CN was added to a solution of compound **3** in 10 ml of CH_3CN. Precipitation of sodium chloride was detected immediately. After 12 hours the transparent solution was separated and evaporated in the vacuum to half of the volume, which caused crystallization of the product. After 3 hours the product was filtered. The following recrystallization from acetonitrile gave crystals useful for X-ray analysis. Yield 71%, IR

spectra (ν, cm^{-1}): 1670 (C=C); ^{1}H-NMR: 1.04 (s, Me_3C), 2.14 (d, MeN, $^3J_{PH}$ 7.8 Hz), 2.35 (s, MeN), 5.22 (d, CH=C, $^3J_{PH}$ 2.0 Hz), 6.9–7.5 (m, H_{arom}); ^{31}P NMR: 103.

$C_{40}H_{52}BN_2O_2P$ (634.61)

Anal.: found: N 4.45, P 4.83%
calc.: N 4.41, P 4.88%

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