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Reaction of Phosphorus Pentachloride with 2,6-Dichloro-4-phenylbenzo[*e*][1,2λ^5]oxaphosphinine 2-Oxide. Synthesis and Steric Structure of 2,2,6-Trichloro-4-phenylbenzo[*e*][1,2λ^5]oxaphosphinin-2-ylium hexachlorophosphate

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Abstract—2,2,2,6-Tetrachloro-4-phenylbenzo[*e*][1,2]oxaphosphinine and 2,2,6-trichloro-4-phenylbenzo[*e*]-[1,2λ^5]-oxaphosphinin-2-ylium hexachlorophosphate were prepared by treatment of 2,6-dichloro-4-phenylbenzo[*e*][1,2λ^5]-oxaphosphinine 2-oxide with phosphorus pentachloride. The structure of the latter compound was proved by means of X-ray diffraction analysis.

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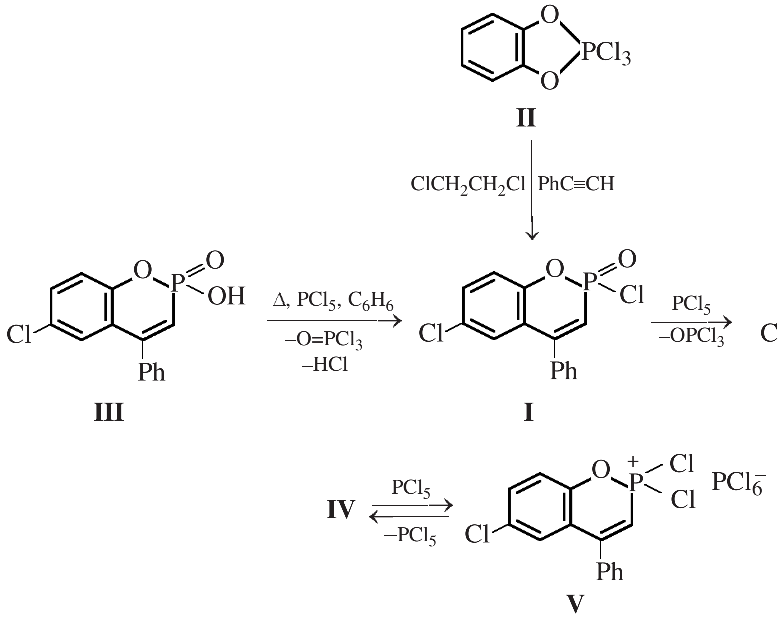
Development of mild selective methods for conversion of the phosphoryl group into a reactive pentacoordinate phosphorus fragment opens up wide perspectives for synthesis of polysubstituted functionalized organophosphorus compounds. Since the P=O group is thermodynamically very stable, there has been little work on its functionalizaton. Here we can mention reduction with lithium aluminum hydride, silicochloroform, and hydroiodic acid as an extensively used synthetic approach to P(III) derivatives. Another known process involving the phosphoryl group is the reaction with strong halogenating reagents, such as sulfur and antimony fluorides and phosphorus pentachloride [1, 2], leading to dihalophosphoranes. The use of phosphorus pentachloride allows selective substitution of one or two hydroxy or alkoxy groups in phosphorous acids or esters with preservation of the coordination number of phosphorus. Subsequent chlorination of the phosphoryl group with phosphorus pentachloride to increase the coordination number of phosphorus is also possible [2]. However, these works dealt mostly with acyclic P(IV) derivatives, whereas cyclic derivatives of phosphonic and phosphinic acids of a similar type were not involved in these reactions.

In the present work we for first time accomplished reaction of phosphorus pentachloride with 2,6-dichloro-4-phenylbenzo[*e*][1,2λ^5]-oxaphosphinine 2-oxide (**I**) synthesized earlier through the reaction of 2,2,2-trichloro-1,3,2-benzodioxaphosphole (**II**) with phenylacetylene in methylene chloride. Oxide **I** is a phosphorous analog of a widespread natural heterocycle coumarin [3]. In our work we slightly changed the method of synthesis of compound **I** and effected the reaction in boiling dichloroethane. This allowed us to exclude excess phenylacetylene and thus facilitate separation of the product from chlorostyrene formed by the side addition of HCl to phenylacetylene. Compound **I** was prepared also via chlorination of 6-chloro-2-hydroxy-4-phenylbenzo[*e*][1,2λ^5]oxaphosphosinine 2-oxide (**III**) with phosphorus pentachloride at a 1 : 1 starting reagent ratio. The structure of benzophosphinine **I** and our previous data [4, 5] point to an aromatic character of the oxaphosphinine fragment. Therefore, changing the phosphorus coordination from tetrahedral to trigonal-bipyramidal should affect significantly the properties of both the heterocyclic system and the phosphorus atom included into this heterocycle. We found that the reaction of phosphinine **I** with phosphorus pentachloride occurs in two steps differing in their rates, that allowed us to obtain intermediate phosphorane **IV** in 92–95% yield. The reaction also gave $POCl_3$ (δ_P 2.0 ppm).

The phosphorus chemical shift of compound **IV** is



characteristic of pentacoordinate phosphorus derivatives (δ_P –33.3 ppm, CH_2Cl_2) and consistent with those reported for trichlorophosphoranes (δ_P –25 to –36 ppm) [6]. Compound **IV** is relatively stable, but with excess phosphorus pentachloride its phosphorus signal shifts downfield from δ_P –20 ppm to –29.2 ppm and gets broader. In the proton-coupled spectra, this signal converts into a broadened doublet whose coupling constant $^2J_{PCH}$ varies in the range 39.1–44.6 Hz, depending on solvent (methylene chloride or benzene) and excess of phosphorus pentachloride. Thus the $^2J_{PCH}$ in benzene is 44.6 Hz. At a 1 mol excess of phosphorus pentachloride, two signals appear in the $^{31}\mathrm{P}\xi^{-\{^1\mathrm{H}\}}$ NMR spectrum (δ_{P_1} 63.8 ppm and δ_{P_2} –296.2 ppm), and, therewith, the singlet at δ_{P_1} converts into a broadened doublet with $^2J_{PCH}$ 28.3–29.0 Hz in the ^{31}P NMR spectrum. The δ_{P_1} value is also affected by the excess of phosphorus pentachloride and can achieve 44.3 ppm (C_6H_6, excess of PCl_5 110–120%). It is obvious that the signals at δ_{P_1} and δ_{P_2} belong to phosphonium salt **V**, the first belonging to the phosphonium cation and the second, to the hexachlorophosphate anion. Thus, phosphorus pentachloride was found to be a stronger Lewis acid compared to phosphorane **IV** and, therefore, it is capable of abstracting the chloride anion. It is interesting that the ^{31}P NMR spectrum of a solution of salt **V** in methylene chloride shows signals of phosphorane **IV** and phosphorus pentachloride together with the signals at δ_{P_1} and δ_{P_2}.

The structure of compounds **IV** and **V** was additionally proved by ^{13}C NMR spectroscopy. Note that the C^3 signals of compounds **IV** and **V** are shifted downfield compared to compound **I** and their $^1J_{HC}$ contants are considerably larger. The multiplicities of all other carbon signals are analogous to those in the spectrum of phosphinine **I**, providing evidence for the absence of other conversions of the benzophosphinine structure.

The structure of salt **V** was also confirmed by X-ray diffraction analysis of a crystal obtained by crystallization from methylene chloride as a stable solvate. Figure 1 depicts the geometry of this solvate in the crystal. The phosphinine heterocycle has a distorted *boat* conformation with two planar, within 0.010(3) Å, four-atomic fragments $O^1C^{8a}C^{4a}C^4$ and

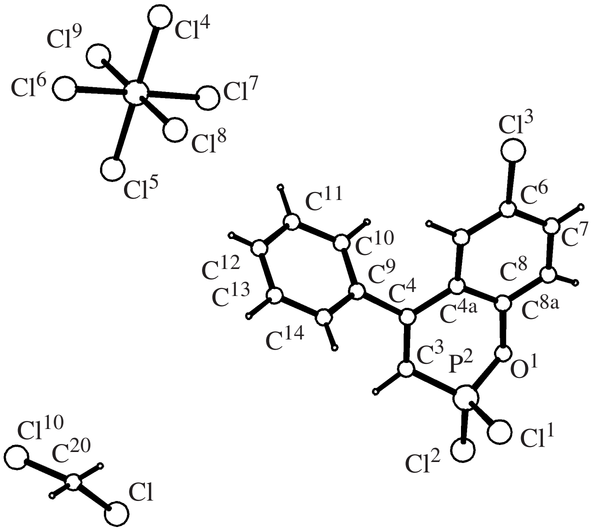


Fig. 1. Geometry of the methylene chloride solvate in the crystal of **V**.

Principal bond lengths (*d*, Å) and bond (φ, deg) and torsion angles (τ, deg) in the methylene chloride solvate of **V**

Bond	*d*	Bond	*d*	Bond	*d*
$Cl^1–P^2$	1.956(1)	$P^2–C^3$	1.731(3)	$C^6–C^7$	1.388(4)
$Cl^2–P^2$	1.935(1)	$P^2–O^1$	1.554(2)	$C^7–C^8$	1.386(5)
$Cl^3–C^6$	1.741(3)	$O^1–C^{8A}$	1.420(4)	$C^8–C^{8A}$	1.360(5)
$Cl^4–P^1$	2.151(1)	$C^3–C^4$	1.363(5)	$C^9–C^{10}$	1.392(5)
$Cl^5–P^1$	2.148(1)	$C^4–C^{4A}$	1.470(5)	$C^9–C^{14}$	1.389(5)
$Cl^6–P^1$	2.114(1)	$C^4–C^9$	1.493(4)	$C^{10}–C^{11}$	1.397(4)
$Cl^7–P^1$	2.150(1)	$C^{4A}–C^5$	1.405(5)	$C^{11}–C^{12}$	1.372(5)
$Cl^8–P^1$	2.1368(9)	$C^{4A}–C^{8A}$	1.404(4)	$C^{12}–C^{13}$	1.380(5)
$Cl^9–P^1$	2.124(1)	$C^5–C^6$	1.377(5)	$C^{13}–C^{14}$	1.393(4)
Angle	**φ**	**Angle**	**φ**	**Angle**	**φ**
$Cl^1P^2Cl^2$	106.90(5)	$Cl^5P^1Cl^8$	90.08(5)	$Cl^4P^1Cl^7$	90.34(5)
$Cl^1P^2O^1$	108.53(8)	$Cl^5P^1Cl^9$	90.24(5)	$P^2O^1C^{8A}$	119.3(2)
$Cl^1P^2C^3$	111.9(1)	$Cl^6P^1Cl^7$	90.24(4)	$P^2C^3C^4$	117.1(2)
$Cl^2P^2O^1$	105.71(9)	$Cl^6P^1Cl^8$	179.41(6)	$C^3C^4C^{4A}$	122.1(3)
$Cl^2P^2C^3$	116.0(1)	$Cl^6P^1Cl^9$	90.39(4)	$C^3C^4C^9$	118.2(3)
$O^1P^2C^3$	107.5(1)	$Cl^7P^1Cl^8$	89.91(4)	$C^{4A}C^4C^9$	119.7(3)
$Cl^4P^1Cl^8$	89.55(4)	$Cl^7P^1Cl^9$	179.32(5)	$C^4C^{4A}C^5$	121.6(3)
$Cl^4P^1Cl^9$	89.92(5)	$Cl^8P^1Cl^9$	89.46(4)	$C^4C^{4A}C^{8A}$	122.3(3)
$Cl^5P^1Cl^6$	90.49(4)	$Cl^4P^1Cl^5$	179.59(4)	$O^1C^{8A}C^{4A}$	119.9(3)
$Cl^5P^1Cl^7$	89.49(4)	$Cl^4P^1Cl^6$	89.88(5)	$O^1C^8AC^8$	116.1(3)
Angle	**τ**	**Angle**	**τ**	**Angle**	**τ**
$Cl^1P^2O^1C^{8A}$	81.8(2)	$P^2O^1C^8AC^{4A}$	27.5(4)	$C^{4A}C^4C^9C^{10}$	–49.8(4)
$C^3P^2O^1C^{8A}$	–39.4(2)	$C^3C^4C^9C^{14}$	–46.3(4)	$C^3C^4C^4AC^{8A}$	–14.5(5)

$P^2C^3C^4C^{4a}$, from which the P^2,C^3 and O^1,C^{8a} atoms deviate to the same side but by different distances [–0.6639(8), –0.254(3) and 0.633(2), 0.342(3) Å, respectively]. The Cl^1 atom occupies an axial position and deviates from the $P^2C^3C^4C^{4a}$ and $O^1C^{8a}C^{4a}C^4$ planes by –1.8222(8) and –2.5973(8) Å, respectively. The Cl^2 atom occupies an equatorial position and deviates from the $P^2C^3C^4C^{4a}$ and $O^1C^{8a}C^{4a}C^4$ planes by 1.0201(8) and –0.0272(8) Å, respectively. The axial bond P^-Cl^1 is longer then the equatorial bond $P^2–Cl^2$. The dihedral angle between the $P^2C^3C^4C^{4a}$ and $O^1C^{8a}C^{4a}C^4$ planes is 14.8(3)°. The 4-Ph substituent is appreciably turned relatively to the $P^2C^3\cdot C^4C^{4a}$ plane [torsion angle –49.8(4)°], which makes unlikely conjugation between the Ph ring and C^3=C^4 bond. The anionic part of the molecule has a regular octahedron configuration, and the P^2–Cl bond lengths span the range of 2.114–2.151(1) Å (principal bond lengths and bond and torsion angles in molecule **V** are listed in the table).

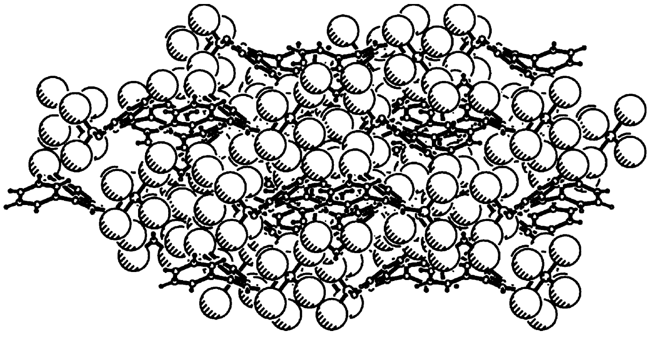


Fig. 2. Three-dimensional cylindrical domain structure in the crystal of **V**.

Analysis of intermolecular interactions revealed no classical hydrogen and C–H···O-type bonds. At the same time, a lot of short Cl···Cl and Cl···H contacts were found. These contacts form a 3*D* cylindric domain structure with prevailing "chlorine" contribution and with cavities occupied by the carbon carcass of the phosphinine molecule (Fig. 2).

In the crystal of phosphinine **V**, there are also π–π interactions between the phenylene fragments of neighboring molecules related by a center of symmetry (1 – *x*, –*y*, 2 – *z*). This short contacts have the following parameters: The distance between the ring centers is 4.184(2) Å, the dihedral angle between the planes is zero, the angle between the normal to one of the planes and the vector connecting the ring centers is 35.82°, and the interplanar spacing is 3.393 Å. There are also π–π interactions between the benzene rings of the substituents at C^4 [dimer with a center of symmetry, symmetry code (1/2 – *x*, 1/2 – *y*, 1 – *z*)]. The parameters of this interaction are as follows: The distance between the ring centers is 4.369(2) Å, the dihedral angle between the planes is zero, the angle between the normal to the one of the planes and the vector connecting the ring centers is 40.62°, and the interplanar specing is 3.316 Å. As a result, these π–π interactions form in the crystal of **V** endless chains of cations along the *a*0*c* diagonal (Fig. 3).

Thus, the reaction of phosphorus pentachloride with a cyclic phosphorus derivative, namely, 2,6-dichloro-4-phenylbenzo[*e*][1,2λ^5]-oxaphosphinine 2-oxide (**I**) leads to chlorination of the phosphoryl group, involving consecutive formation of penta- and tetracoordinate phosphorus derivatives: 2,2,2,6-tetrachloro-4-phenylbenzo[*e*][1,2]oxaphosphinine (**IV**) and 2,2,6-trichloro-4-phenylbenzo[*e*][1,2λ^5]oxaphosphinin-2-ylium hexachlorophosphate (**V**).

EXPERIMENTAL

The NMR spectra were recorded on Bruker MSL-400 (operating at 100.6 MHz for ^{13}C NMR spectra and at 162.0 MHz for ^{31}P NMR spectra), Avance-600 (^1H), and CXP-100 (36.38 MHz, ^{31}P) instruments. The mass spectra were obtained on a Finnigan MAT TRACE MS instrument at the ionizing energy 70eV and ion source temperature 200°C. The sample probe was heated from 35 to 150°C at a rate of 35 deg min^{-1}. Data treatment was performed using the Xcalibur program.

Reaction of 2,2,2-trichloro-1,3,2-benzodioxaphosphole (II) with phenylacetylene. Phenylacetylene, 4.17 g, was added to a solution of 10.5 g of phosphole **II** in 30 ml of dichloroethane heated to 80°C and stirring with a stream of dry argon fed via a capillary tube. The rate of addition was so that the mixture boiled gently. After complete addition, argon bubbling was continued until the reaction mixture cooled down to 20°C. The solvent was then removed by distillation, and the residue was dried in a vacuum of 0.1 mm Hg at 80°C and treated with a mixture of pentane and methylene chloride, 10 : 1. The crystalline precipitate that formed was filtered off and dried in a vacuum to obtain 11.0 g (83%) of compound **I**, mp 122–124°C. Mass spectrum, *m/z*: 310 ($C_{14}H_9Cl_{352}\cdot O_2P$) $[M]^+$, 275 [*M* – Cl], [*M* – POCl], 199 [*M* – Cl – PO_2]. The melting point and other spectral characteristics correspond to published data [3].

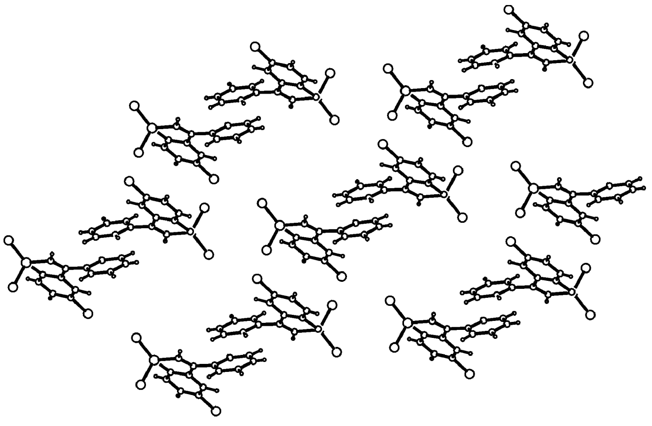


Fig. 3. Chains formed due to π–π interactions in the crystal of **V**.

Reaction of 6-chloro-2-hydroxy-4-phenyl[*e*]-[1,2λ^5]oxaphosphinine 2-oxide (III) with phosphorus pentachloride. A powdered phosphinine **III**, 4.04 g (0.0138 mol), was added to 3.15 g (0.0151 mol) of phosphorus pentachloride in 20 ml of benzene. The reaction mixture was heated under reflux for 1 h under argon. After cooling, a precipitate formed. The supernatant liquid was decanted, and the precipitate was washed with absolute pentane and dried in a vacuum of 0.1 mm Hg at 70–80°C to obtain 2.86 g (80%) of oxaphosphinine **I**.

Reaction of oxaphosphinine III with phosphorus pentachloride at a 1 : 3 ratio. A mixture of 8.31 g of phosphorus pentachloride, 20 ml of benzene, and 3.50 g of oxaphosphinine **III** was heated under reflux for 2 h. After cooling, crystals formed and were filtered off, washed with a little benzene, and dried in a vacuum to obtain 90% (5.86 g) of 2,2,6-trichloro-4-phenylbenzo[*e*][1,2λ^5]-oxaphosphinin-2-ylium hexachlorophosphate (**V**), mp 171°C. Found Cl, %: 58.21. $C_{14}H_9Cl_9OP_2$. Calculated Cl, %: 58.68. ^{31}P NMR spectrum (C_6H_6), δP, ppm: 63.8 (P^{IV}), –296.1 (P^{VI}). ^{13}C NMR spectrum ($C_6H_6 + D_2O$ in a capillary insert), δ_C, ppm (*J*, Hz) (here and hereinafter, in brackes we specify the shape of the signal in the ^{13}C–{^1H} NMR spectrum): 127.62 [d, $^1J_{HC^3}$ (not determined because of overlapping in the ^{13}C NMR spectrum] (C^3, $^1J_{PC^3}$ 153.0–154.0); 156.82 m (br.s) (C^4); 121.38 m (d) (C^{4a}, $^3J_{PCCC^{4a}}$ 20.0); 130.64 d.d.d (d) (C^5, $^1J_{HC^5}$ 160.0–163.0, $^3J_{HC^7CC^5}$ 5.5–6.0, $^4J_{PCCC^5}$ 2.0); 130.31 (s) (C^6); 134.81 d.d (d) (C^7, $^1J_{HC^7}$ 169.5, $^3J_{HC^5CC^7}$ 6.0); 121.69 d.d (d) (C^8, $^1J_{HC^8}$ 168.1, $^3J_{POCC^8}$ 8.0); 148.52 d.d.d (d)

($C^{8a,}$ $^3J_{HC^5CC^{8a}}$ 10.4, $^3J_{HC^7CC^{8a}}$ 10.4, $^2J_{POC^{8a}}$ 11.4); 135.25 m (d) (C^9, $^3J_{PCCC^9}$ 25.2); 129.17 d.d.d (s) (C^{10}, $^1J_{HC^{10}}$ 161.8, $^3J_{HC^{10'}CC^{10}}$ 7.0–7.3, $^3J_{HC^{12}CC^{10}}$ 7.0–7.3); 129.56 d.d (s) (C^{11}, $^1J_{HC^{11}}$ 163.4, $^3J_{HC^{11'}CC^{11}}$ 7.3); 131.81 d.t (s) (C^{12}, $^1J_{HC^{12}}$ 162.2, $^3J_{HC^{10}CC^{12}}$ 7.3). The melting point of the methylene chloride solvate of compound **V** is 189–190°C.

Reaction of oxaphosphinine I with phosphorus pentachloride. A mixture of 2.09 g of phosphorus pentachloride, 10 ml of benzene, and 3.11 g of oxaphosphinine **I** was heated under reflux for 2 h. After cooling, the solvent and $POCl_3$ were removed by distillation in a vacuum of 12 mm Hg. The residue was dried in a vacuum 0.1 mm Hg and characterized by spectral methods. The yield of 2,2,2,6-tetrachloro-4-phenylbenzo[*e*][1,2]oxaphosphinine (**IV**) was 92%, viscous light brown glassy substance. ^1H NMR spectrum (600 MHz, $CDCl_3$ + 20% C_6H_6, δ, ppm, *J*, Hz): 6.95 d (H^3, $^2J_{PCH}$ 44.9); 7.12 d.d (H^8, $^3J_{H^7CCH^8}$ 8.7, $^4J_{POCCH^8}$ 1.4); 7.39 d (H^5, $^4J_{H^7CCCH^5}$ 1.8); 7.47 br.d.d (H^7, $^3J_{H^8CCH^7}$ 8.7, $^4J_{H^5CCCH^7}$ 1.8); 7.52–7.57 m (C_6H_5). ^{31}P NMR spectrum (162.0 MHz, C_6H_6): δ_P –29.7 ppm (d, $^2J_{PCH}$ 44.8). ^{13}C NMR spectrum, ^{13}C–{^1H} ($CDCl_3$, δ_C, ppm, *J*, Hz): 127.73 d.d (d) (C^3, $^1J_{PC^3}$ 152.1, $^1J_{HC^3}$ 205.1); 157.87 m (d) (C^4, $^2J_{PCC^4}$ 3.0); 122.09 d.m (d) ($C^{4a,}$ $^3J_{PCCC^{4a}}$ 20.0); 129.33 br.d (d) (C^5, $^1J_{HC^5}$ 167.2, $^4J_{PCCC^5}$ 1.1); 130.41 d.d.d (s) (C^6, $^3J_{HC^8CC^6}$ 11.0, $^2J_{HC^7C^6}$ 4.0, $^2J_{HC^5C^6}$ 4.0); 132.77 d.d.d (d) (C^7, $^1J_{HC^7}$ 167.6, $^3J_{HC^5CC^7}$ 6.0, $^4J_{POCCC^7}$ 1.7); 121.54 d.d (d) (C^8, $^1J_{HC^8}$ 167.0, $^3J_{POCC^8}$ 5.9); 152.61 m (d) ($C^{8a,}$ $^2J_{POC^{8a}}$ 14.4); 136.31 m (d) (C^9, $^3J_{PCCC^9}$ 27.0); 128.27 d.m (s) (C^{10}, $^1J_{HC^{10}}$ 162.0); 128.94 d.d (s) (C^{11}, $^1J_{HC^{11}}$ 162.3, $^3J_{HC^{11'}CC^{11}}$ 6.7–7.0); 130.39 d.t (s) (C^{12}, $^1J_{HC^{12}}$ 161.8, $^3J_{HC^{10}CC^{12}}$ 7.3). Mass spectrum, *m/z*: 329 [*M* – Cl], 294 [*M* – 2Cl], 210 [*M* – 2Cl – POCl], 197 [*M* – 2Cl – POCl – CH].

X-ray diffraction analysis. Crystals of compound **V**, $C_{14}H_9Cl_3OP^- \cdot PCl_6^+ \cdot 1/2CH_2Cl_2$, monoclinic, at –150°C: $a = 30.23(1)$, $b = 10.323(2)$, $c = 16.990(5)$ Å, $\beta = 120.56(3)°$, $V = 4565(3)$ Å^3, $Z = 8$, $M = 616.67$, $d_{calc} = 1.79$ g cm^{-3}, $F(000) = 2440$, space group $C2/c$ (the solvation methylene chloride molecule is in a special position at the 2 axis). The intensities of 4989 reflections, of which 3802 with $I > 3\sigma$, were measured on an Enraf–Nonius CAD-4 diffractometer at –150°C (λ MoK_α radiation, ω/2θ scanning, $2\lambda_{max} < 52.6°$). No intensity decay of three reference reflections was observed during measurements. Empirical correction for absorption was applied (μMo 13.68 cm^{-1}). The structure was solved by the direct method using the SIR program [7] and refined first isotropically and then anisotropically. Hydrogen atoms were revealed by difference synthesis and refined isotropically at the final stage. Final divergence factors: R 0.052 and R_W 0.068 on 3802 unique reflections with $F^2 \geq 3\sigma$. All calculations were carried out by means of the MOLEN program package [8] on an AlphaStation 200 computer.

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