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Reactions of Elemental Phosphorus and Phosphine with Electrophiles in Superbasic Systems: XX.[1] Phosphorylation of 4-Vinylbenzyl Chloride with Elemental Phosphorus

S. F. Malysheva[a], **V. A. Kuimov**[a], **N. K. Gusarova**[a], **B. G. Sukhov**[a],
Yu. V. Smetannikov[b], **N. P. Tarasova**[b], and **B. A. Trofimov**[a]

[a] *Favorskii Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences,*
ul. Favorskogo 1, Irkutsk, 664033 Russia
e-mail: mal@irioch.irk.ru

[b] *Mendeleev Russian University of Chemical Technology, Moscow, Russia*

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Abstract—4-Vinylbenzyl chloride reacts with white and red phosphorus, as well as with nanostructured "activated" red phosphorus (complex organophosphorus polymer prepared from white phosphorus under ionizing radiation) in the system concentrated aqueous KOH–dioxane–phase-transfer catalyst (20–50°C, argon) to form tris(4-vinylbenzyl)phosphine oxide, along with (4-vinylbenzyl)- and bis(4-vinylbenzyl)phosphinic acids, the yield and product ratio being dependent on both the reaction conditions and the nature of the phosphorylating agent. The nanostructured "activated" red phosphorus is more reactive than ordinary commercial red phosphorus.

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Tertiary phosphine oxides are key objects in the chemistry of organophosphorus compounds. They are widely used as ligands for metal-complex catalysts [2–5], intermediates for preparation of semiconductor nanomaterials [6–8], extractants for noble, rare-earth, and transuranium elements [9–12], flame retardants for preparation of nonflammable polymers [13–15], synthons used, e.g., in the Horner–Wittig reaction [16–21] or for preparation of corresponding phosphines. Of special interest are functionalized tertiary phosphines and phosphine oxides possessing styryl moieties, on the basis of which unique polymeric catalysts of a new generation [22, 23] and new materials for nonlinear optics were created [24, 25]. One of the most convenient synthetic approaches to tertiary phosphine oxides, including functionally substituted, unsaturated and chiral, involves direct phosphorylation of electrophiles with elemental phosphorus in superbasic systems [1, 3, 26, 27]. In particular, red phosphorus was reported to react with benzyl chloride [28, 29] and styrene [30] in the presence of strong bases, like KOH–polar nonhydroxylic solvent (DMSO, HMPA) or KOH–organic solvent–phase transfer catalyst [26], to form, respectively, tribenzyl- and tris(2-phenylethyl)phosphine oxides.

We recently published a preliminary report on the chemoselective formation of tris(4-vinylbenzyl)phosphine oxide from red phosphorus and 4-vinylbenzyl chloride (bifunctional electrophile) under conditions of phase-transfer catalysis [31].

In the present work with the aim to obtain further evidence for the perspectiveness and generality of the method of synthesis of popular organophosphorus compounds, based on elemental phosphorus and super bases, we performed a detailed study of the reaction of white, ordinary (commercial) red phosphorus, and so-called "activated" red phosphorus [32, 33] with 4-vinylbenzyl chloride in the presence of a strong base.

Two samples of "activated" red phosphorus ($^{1*}P_n$ and $^{2*}P_n$) with the phosphorus content of 70% were used. The sample $^{1*}P_n$ was prepared by the action of ionizing radiation of (^{60}Co, 0.78 Gy s^{-1}) on a saturated benzene solution of white phosphorus at room temperature, whereas the sample $^{2*}P_n$ was synthesized under similar conditions in the presence of elemental sulfur at the power of absorbed dose of 0.55 Gy s^{-1}. Both samples ($^{1*}P_n$ and $^{2*}P_n$) contained carbon

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[1] For communication XIX, see [1].

Phosphorylation of 4-vinylbenzyl chloride (11.5 mmol in exp. nos. 1, 3–5 and 5.4 mmol in exp. no. 2)[a]

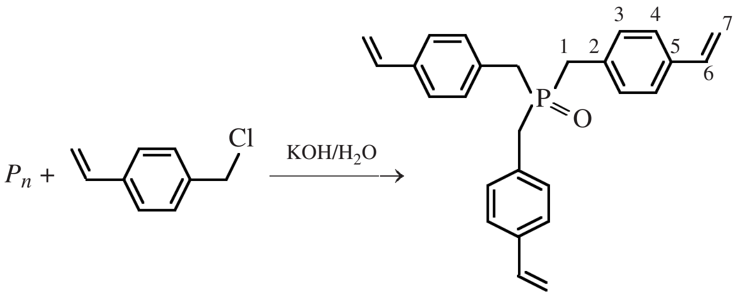
Exp. no.	P, mmol	Yield,[b] %				Conversion of 4-vinylbenzyl chloride[e], %
		$R_3P{=}O$ (**I**)	RP(O)H(OH)[c] (**II**)	$R_2P(O)OH$[c] (**III**)	Phosphorus-containing polymer[d], g	
1	P_n, 66.5	55	[f]	[f]	0.36	98
2	P_n, 66.5	49	[f]	[f]	0.17	98
3	P_4, 32.3	42	11	9	0.25	88
4	$^{1*}P_n$, 38.7	44	2	6	0.1	86
5	$^{2*}P_n$, 28	54	5	7	0.4	90

[a] Reaction temperature 45–50°C (exp. nos. 1, 2, 4, 5) and 20–50°C (exp. no. 3). Reaction time 3 h. In all experiments, 209 mmol of KOH, 0.2 g of TEBAC, 0.1 g of hydroquinone, 6 ml of H_2O and 20 ml of dioxane were used. [b] Yield per taken 4-vinylbenzyl chloride. [c] Acids **II** and **III** were isolated from the reaction mixture after treatment of its aqueous part with HCl. [d] Polymers in exp. nos. 3–5 were isolated from the organic phase and in exp. nos. 1, 2, after acidification of the reaction mixture. [e] Estimated from the ^1H NMR spectra. [f] No products formed.

(~10%) and oxygen (~17%). In addition, $^{2*}P_n$ contained sulfur (1.3%). The IR spectra of these samples contain, along with an absorption band at 500 cm^{-1} [ν(P–P)], the following absorption bands: 720 [ν(P–C)], 1021 [ν(P–O–C)], 1380 [ν(P=O)], and 1460 cm^{-1} [ν(P–OH)], implying chemical bonding of solvent (benzene) fragments with the phosphorus polymer formed [34]. In was also established that in sample $^{2*}P_n$ sulfur is incorporated into the polymeric matrix [35].

The electron microscopy data indicate that the samples of "activated" red phosphorus consist of nanosized incomplete hollow spheres (particle size ca. 250–500 nm) [35]. In this case, the developed surface of the phosphorus polymer provides a high concentration of surface defects in it, thus enhancing its chemical activity compared to ordinary commercial red phosphorus [36–38] possessing no nanostructured morphology [35].

The phosphorylation of 4-vinylbenzyl chloride with the system red phosphorus–KOH–H_2O–organic solvent (dioxane)–phase-transfer catalyst (benzyltriethylammonium chloride)–radical inhibitor (hydroquinone) proceeds under mild conditions (45–50°C, 3 h) chemoselectively to form tris(4-vinylbenzyl)phosphine oxide (**I**) in up to 55% yield (see table, exp. nos. 1, 2).

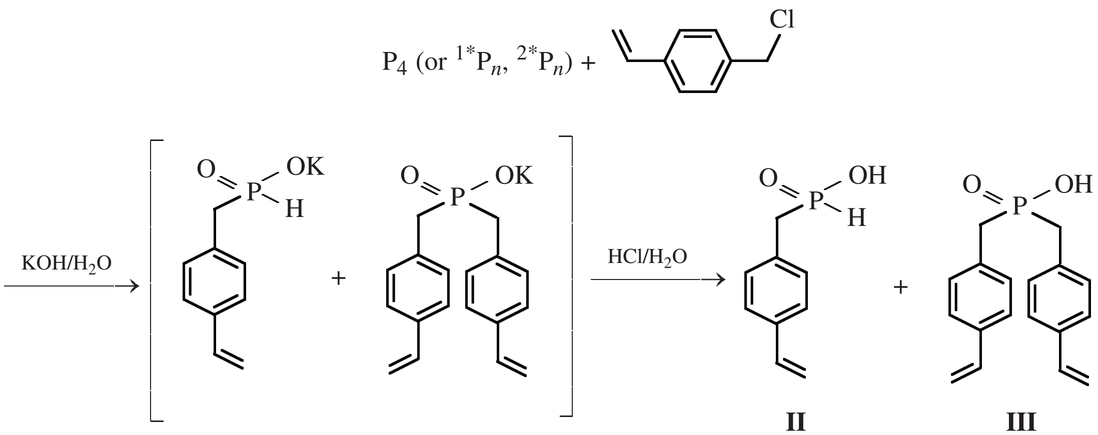


I

(1)

Under similar conditions, white and both modifications of "activated" red phosphorus react with 4-vinylbenzyl chloride to afford not only phosphine oxide **I**, but also 4-vinylbenzyl (**II**) and bis(4-vinylbenzyl)phosphinic (**III**) acids which were isolated after treatment of the reaction mixture with HCl. The total yield of compounds **I**–**III** was 52–66% (see table, exp. nos. 3–5).

Earlier on the phosphorylation of benzyl chloride [28], 4-methoxybenzyl chloride [39] and 1-(chloromethyl)naphthalene [38] with red phosphorus we observed exclusive or preferential formation of tertiary phosphine oxides, whereas with white or "activated" red phosphorus these reactions stopped on the stages of formation of secondary phosphine oxides and phosphinic acids, that is, incomplete arylmethylation



II III

(2)

products. This result is explained by increased concentration of phosphinite or polyphosphinite anions which are more readily formed upon P–P bond cleavage under the action of strong bases in the case of white phosphorus (strained tetrahedral molecule) or "activated" red phosphorus (contains biographical defects [36]) as compared to ordinary red phosphorus which is a reticulate homopolymer [35, 36].

It should be mentioned that the phosphorylation of 4-vinylbenzyl chloride with elemental phosphorus gives, along with the main products **I**–**III**, phosphorus-containing reticulate polymers insoluble in ordinary organic solvents and water (9–32% of the total weight of the compounds formed). Organophosphorus polymers become virtually the main products, when the reactions under consideration are carried out without hydroquinone. The strong tendency of compounds **I**–**III** for oligo- and polymerization is already observed at room temperature, but they are stable enough when stored in a refrigerator (0–5°C).

Since the samples of "activated" red phosphorus contained P–R bonds (where R is benzene fragment), and the phosphorus–sulfur sample $^{2*}P_n$, in addition, P–S bonds, we could expect that their phosphorylation reactions with 4-vinylbenzyl chloride would provide by-products having such fragments. Apparently, such products are actually formed, as evidenced by the presence in the ^{31}P NMR spectra of unidentified signals of low intensity at δ_P 12.41, 17.82, 24.02, and 29.64 ppm. However, we failed to isolate these compounds and to prove their structure.

Thus, the direct reaction of 4-vinylbenzyl chloride with elemental phosphorus in the presence of strong bases allows facile formation of C–P bonds and provides promising organophosphorus compounds. Thus, combining three vinyl substituents and a phosphine oxide group in the molecule of phosphine oxide **I** represents a principally new approach to the design of phosphorus-containing dendrimers [40, 41], including nanosized structures [42] widely used in coordination chemistry and catalysis [43, 44]. Furthermore, this phosphine oxide is an effective comonomer for preparing nonflammable polymer compositions [45] and promising polydentate ligands [23, 46].

The prepared phosphinic acids **II** and **III** can be used as synthons in organic synthesis [47–51], in particular, for purposeful design of phosphaisocoumarins [50, 51]. On the basis of polystyrenephosphonic acid, highly effective hemo- and enterosorption materials have been created [52], used, in particular, for extraction of radionuclides [53].

EXPERIMENTAL

Electron microscopy of the samples of "activated" red phosphorus was performed on a Tesla BS 340 scanning electron microscope. The IR spectra were recorded on a Specord IR-75 instrument. The ^1H, ^{13}C and ^{31}P NMR spectra were measured on a Bruker DPX 400 spectrometer (400, 100, and 161.98 MHz, respectively) in $CDCl_3$ solutions against internal HMDS. All experiments were performed under argon.

The samples of nanosized "activated" red phosphorus $^{1*}P_n$ and $^{2*}P_n$ were prepared at room temperature by the action of ionizing radiation of 60°C on saturated benzene solutions of white phosphorus with the power of the absorbed dose of 0.78 Gy s^{-1} ($^{1*}P_n$) and of 0.55 Gy s^{-1} ($^{2*}P_n$; in the presence of sulfur) and with the same absorbed dose of 190 kGy. The resulting samples were washed with benzene to remove unreacted white phosphorus, dried under vacuum, and analyzed. Found, %: C 10.38; H 3.09; P 69.23; O 17.3 ($^{1*}P_n$) and C 10.19; H 2.22; P 70.63; S 1.32; O 16.96 ($^{2*}P_n$).

Reaction of 4-vinylbenzyl chloride with red phosphorus: Synthesis of tris(4-vinylbenzyl)phosphine oxide (I) (exp. no. 1). To a heated (45–50°C) suspension of 2.06 g of red phosphorus, 0.21 g of benzyltriethylammonium chloride, 11.7 g of KOH, 6 ml of water, 15 ml of dioxane, and 0.1 g of hydroquinone, a solution of 1.76 g of 4-vinylbenzyl chloride in 5.5 ml of dioxane was added dropwise upon stirring over the course of 1.5 h. The reaction mixture was stirred for another 1.5 h at 45–50°C, diluted with water, extracted with benzene (3 × 30 ml), the solvent was removed under reduced pressure, and the residue was dried in a vacuum to obtain 0.85 g of a crude product whose ^{1}H NMR spectrum contained, along with signals of phosphine oxide **I**, a singlet at 4.54 ppm, related to the starting 4-vinylbenzyl chloride (conversion 98%).

Phosphine oxide **I**, 0.84 g (55%), needles, mp 136–140°C (hexane). IR spectrum, cm^{-1}: 3080, 3030 [ν(=CH, =CH$_2$)], 2990, 2950, 2850 [ν(CH, CH$_2$)], 1620 [ν(C=C)], 1600, 1500, 1400 [ν(C=C–Ph)], 1180 [ν(P=O)], 1110, 980, 900, 850 [δ(CH–Ph)]. ^{1}H NMR, δ, ppm, (*J*, Hz): 2.98 d (6H, H^1, $^2J_{PH}$ 13.7), 5.22 d (3H, H^7, $^3J_{HH}$ 10.7), 5.71 d (3H, H^7, $^3J_{HH}$ 17.6), 6.67 d.d (3H, H^6), 7.17–7.33 m (12H, C_6H_4). ^{13}C NMR, δ_C, ppm, (*J*, Hz): 35.27 d (C^1, $^1J_{PC}$ 60.6), 113.89 C^7, 126.58 C^3, 130.01 C^4, 131.17 d (C^2, $^2J_{PC}$ 7.2), 136.28 C^6, 136.38 C^5. ^{31}P NMR, δ_P 41.28 ppm. Found, %: C 80.98; H 6.87; P 8.01. $C_{27}H_{27}PO$. Calculated, %: C 81.38; H 6.83; P 7.77.

The aqueous layer was acidified with 35% HCl to pH 4, and 15 ml of $CHCl_3$ was added (no layer separation occurred). The suspension that formed was diluted with 15 ml of benzene, the solvents and water were removed under reduced pressure, and a pasty product was obtained. It was successively washed with water (to remove KCl), acetone, and ether and dried in vacuum to obtain 0.36 g of a product (30% of the total weight of the reaction products) as a beige powder insoluble in water and ordinary organic solvents and not melting up to 300°C. IR spectrum, cm^{-1}: 3032, 3018 [ν(=CH, =CH$_2$)], 2926, 2920, 2840 [ν(CH)], 2330, 1637 [ν(OH)], 1511, 1450, 1423, 1400 [ν(C=C–Ph)], 1232, 1126 [ν(P=O)], 997 [ν(OH)], 852 [δ(CH–Ph)]. Found, %: C 57.46; H 5.49; Cl 2.45; P 11.74; ash 22.74.

Under similar conditions but with 0.83 g of 4-vinylbenzyl chloride (exp. no. 2), phosphine oxide **I** was obtained in 49% yield, along with 0.17 g (32% of the total weight of the reaction products) of a phosphorus-containing reticulate polymer (insoluble in water and organic solvents) with the following absorption bands in the IR spectrum, cm^{-1}: 2930 [ν(CH)], 2323, 1638 [ν(OH)], 1512, 1423, 1400 [ν(C=C–Ph)], 1126 [ν(P=O)], 997 [ν(OH)], 850 [δ(CH–Ph)]. Found, %: C 57.05; H 6.02; Cl 1.19; P 10.78; ash 24.96.

Reaction of 4-vinylbenzyl chloride with white phosphorus: Synthesis of tris(4-vinylbenzyl)phosphine oxide (I) and 4-vinylbenzylphosphinic acid (II) (exp. no. 3). To a mixture of 1.0 g of white phosphorus, 0.1 g of hydroquinone and 0.21 g of benzyltriethylammonium chloride in 20 ml of dioxane, 11.7 g of KOH and 6 ml of water were added and then 1.76 g of 4-vinylbenzyl chloride was added dropwise at room temperature over the course of 1 h. The reaction mixture was stirred for another 1 h at room temperature and 1 h at 45–50°C, cooled, diluted with water, and extracted with benzene (3 × 30 ml). Benzene was distilled off, and the residue dried in vacuum to afford 0.90 g of product A (light yellow waxy material) whose ^{31}P NMR spectrum contained the following signals, δ_P, ppm: 47.34 (phosphinic acid **III**), 41.28 (phosphine oxide **I**), and 36.14 ($^1J_{PH}$ 458 Hz) [probably, bis(4-vinylbenzyl)phosphine oxide which under further treatment is oxidized to phosphinic acid **III**]. The intensity ratio of the above signals was 1 : 18.2 : 3, respectively. The ^{1}H NMR spectrum of product A, contained, along with the above signals, a singlet at 4.54 ppm related to the starting 4-vinylbenzyl chloride (conversion 88%). Product A was dissolved in 5 ml of benzene. Product B precipitated as a white powder and was separated by filtration [0.25 g, (22% of the total weight of the reaction products)]; the product is insoluble in water and organic solvents and does not melt up to 300°C. IR spectrum, cm^{-1}: 3080, 3020, 3000 [ν(=CH, =CH$_2$)], 2900 [ν(CH)], 2350, 1660 [ν(OH)], 1620 [ν(C=C)], 1600, 1500, 1400 [ν(C=C–Ph)], 1160 [ν(P=O)], 1115 [ν(OH)], 985, 900, 850 [δ(CH–Ph)]. Found, %: C 72.55; H 6.30; P 8.36. $C_{27}H_{27}PO$. Calcd, %: C 81.37; H 6.83; P 7.77. The benzene solution was reprecipitated into hexane to obtain 0.64 g (42%) of phosphine oxide **I**.

The aqueous layer was acidified with 35% aqueous HCl to pH 4–5, extracted with dichloromethane, and the extract was dried over $CaCl_2$. The solvent was then removed, and the residue was dried in vacuum to obtain 0.25 g of a product as a light yellow waxy material which was dissolved in chloroform and reprecipitated into ether to afford 0.23 g (11%) of phosphinic acid **II**, mp 98–100°C (dichloromethane). IR spectrum, cm^{-1}: 3080, 3040, 3000 [ν(=CH, =CH$_2$)], 2900, 2850 [ν(CH)], 2620 [ν(OH)], 2410 [ν(PH)], 2240, 2140, 1680 [ν(OH)], 1620 [ν(C=C)], 1500, 1400, 1390 [ν(C=C–Ph)], 1100 [ν(P=O)], 980 [ν(OH)], 960 [ν(P=O)], 900, 820 [δ(CH–Ph)]. ^{1}H NMR, δ, ppm, (*J*, Hz): 3.07 d (2H, H^1, $^2J_{PH}$ 18.4), 5.21 d (1H, H^7, $^3J_{HH}$ 10.8), 5.70 d (1H, H^7, $^3J_{HH}$ 17.6), 6.66 d.d (1H, H^6), 6.93 d (1H, PH), 7.14–7.34 m (4H, C_6H_4), 8.97 (1H, OH). ^{13}C NMR, δ_C, ppm, (*J*, Hz): 37.37 d (C^1, $^1J_{PC}$ 87.6), 114.07 C^7, 126.69 C^3, 128.94 C^4, 130.02 (C^2), 136.27 C^6, 136.72 C^5. ^{31}P NMR: δ_P 36.82 ppm, $^1J_{PH}$ 558.7 Hz. Found, %: C 59.78; H 6.55; P 16.80. $C_9H_{11}O_2P$. Calculated, %: C 59.34; H 6.09; P 17.00.

The yield of phosphinic acid **III**, estimated from the ^{1}H and ^{31}P NMR spectra, was 9%.

Reaction of 4-vinylbenzyl chloride with "activated" red phosphorus: Synthesis of bis(4-vinylbenzyl)phosphinic acid (III) (exp. no. 5). To a mixture 1.0 g of red phosphorus $^{2*}P_n$, 0.20 g of benzyltriethylammonium chloride, and 0.1 g of hydroquinone in 20 ml of dioxane, 11.7 g of KOH and 6 ml of water were added and then 1.76 g of 4-vinylbenzyl chloride was added dropwise at 45–50°C over the course of 1 h. The reaction mixture was stirred for another 2 h at 45–50°C, cooled, and diluted with water. A precipitate formed and was filtered off, washed on the filter with water to neutral reaction and then with acetone and ether, and dried in a vacuum to obtain 0.4 g (28% of the total weight of the reaction products) of a beige powder insoluble in water and organic solvents, and not melting up to 300°C. IR spectrum, cm^{-1}: 3080, 3020, 3000 [ν(=CH, =CH_2)], 2900, 2850 [ν(CH)], 2640, 2330, 1680 [ν(OH)], 1610, 1600, 1500, 1480, 1450, 1420, 1400 [ν(C=C–Ph)], 1110 [ν(P=O)], 980 [ν(OH)], 940 [ν(P=O)], 900, 840 [δ(CH–Ph)]. Found, %: C 55.44; H 5.72; P 12.63.

The filtrate was extracted with benzene, benzene was removed, and the residue was evacuated to give 1.02 g of a crude product whose ^{1}H NMR spectrum contained, along with signals of phosphine oxide **I**, a singlet at 4.54 ppm, related to the starting 4-vinylbenzyl chloride (conversion 90%). The yield of phosphine oxide **I** was 0.82 g (54%). The aqueous layer was acidified with 35% HCl to pH 4–5, extracted with benzene, the solvent was removed, and the residue was dried in vacuum to obtain 0.22 g of product C as a beige powder whose ^{31}P NMR spectrum contained signals at 37.17 (phosphinic acid **II**) and 51.29 ppm (phosphinic acid **III**) in a 3 : 2 ratio. Product C was placed on a filter, washed with 5 ml of methylene chloride, and dried in vacuum to obtain 0.12 g (7%) of phosphinic acid **III**, mp 180–182°C (chloroform). IR spectrum, cm^{-1}: 3080, 3030 [ν(=CH, =CH_2)], 2900, 2850 [ν(CH)], 2630, 2250, 1680 [ν(OH)], 1610, 1500, 1390 [ν(C=C, C=C–Ph)], 1100 [ν(P=O)], 980 [ν(OH)], 940 [ν(P=O)], 880, 820 [δ(CH–Ph)]. ^{1}H NMR, δ, ppm, (*J*, Hz): 2.82 d (4H, H^1, $^2J_{PH}$ 16.9), 5.17 d (2H, H^7, $^3J_{HH}$ 10.9), 5.65 d (2H, H^7, $^3J_{HH}$ 17.6), 6.62 d.d (2H, H^6), 7.12–7.30 m (8H, C_6H_4), 7.68 (1H, OH). ^{13}C NMR, δ_C, ppm, (*J*, Hz): 35.17 d (C^1, $^1J_{PC}$ 88.3), 113.75 C^7, 126.34 and 126.37 C^3, 130.15 and 130.20 C^4, 130.70 (C^2, $^2J_{PC}$ 8.1), 136.17, 136.21 C^5, 136.29 C^6. ^{31}P NMR: δ_P 51.73 ppm, $^1J_{PH}$ 558.7 Hz. Found, %: C 71.97; H 6.33; P 10.59. Calculated, %: C 72.47; H 6.42; P 10.38.

Methylene chloride was removed to obtain 0.1 g of a compound, containing, according to the ^{31}P NMR spectrum, acids **II** and **III** in a 9 : 1 ratio. The yield of acid **II** estimated from the ^{1}H and ^{31}P NMR spectra was 5%.

Under similar conditions, from red phosphorus $^{1*}P_n$ compounds **I**–**III** were obtained in 44, 2, and 6% yields, respectively, along with 0.1 g (9% of the total weight of the reaction products) of a phosphorus-containing reticulate polymer insoluble in ordinary organic solvents and water and not melting up to 300°C. IR spectrum, cm^{-1}: 3080, 3020, 3000 [ν(=CH, =CH_2)], 2900, 2850 [ν(CH)], 2600, 2300, 1680 [ν(OH)], 1620, 1600, 1500, 1400 [ν(C=C–Ph)], 1110 [ν(P=O)], 970 [ν(OH)], 950 [ν(P=O)], 890, 840 [δ(CH–Ph)]. Found, %: C 59.62; H 5.78; P 10.25.

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