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Reactions of Elemental Phosphorus and Phosphine with Electrophiles in Superbasic Systems: XVIII.[1] Phosphorylation of 1-(Chloromethyl)naphthalene with the Elemental Phosphorus

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Abstract — 1-Chloromethylnaphthalene reacts with white and red phosphorus, and also with the “activated red phosphorus,” the complex organophosphorus polymer of unknown structure obtained by irradiation of a solution of white phosphorus in benzene by the ^{60}Co source, in a system including KOH water solution, dioxane or benzene, and a phase transfer catalyst (22–98°C, argon), to form bis(1-naphthylmethyl)- and tris(1-naphthylmethyl)phosphine oxides, and also (1-naphthylmethyl)phosphonous- and bis(1-naphthylmethyl)phosphinic acids. The yield and the ratio of the reaction products depend on reaction conditions as well as on the nature of phosphorylating agent. It is shown that the reactivity of the “activated red phosphorus” is not worse than that of the white phosphorus and significantly exceeds the reactivity of the usual technical red phosphorus.

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Phosphorylation of organyl halides with the elemental phosphorus under the conditions of the phase transfer catalysis is one of the most convenient methods of formation of a C–P bond and prepation of organophosphorus compounds [2–4], first of all of phosphine oxides [5, 6], including the unsaturated [7, 8], functionalyzed [9, 10], and nonsymmetric ones [11]. The phosphine oxides obtained can be successfully used as the building blocks for example in the Wittig–Horner reaction [10, 12–14], and as the monomers for the prepation of new flame-resistant polymers [15]. They are also used as the effective and selective extragents of precious and rare metals [16], as antipyrenes of polyvinyl chloride plastisols [17, 18], and as the intensifying reagents in the enrichment of the copper-nickel ore [19]. Tertiary phosphine oxides attract still more and more attention as the effective ligands for creation of the metallocomplex catalysts [20-22]. In some catalytic processes they give even higher results than the corresponding tertiary phosphines [20]. Organophosphorus ligands with bulky radicals are also important. The catalysts on their basis exhibit specific activity in the reaction of substitution of chlorine in chloroarenes and formation of C–C, C–N, and C–O bonds [23]. Using of tertiary phosphine oxides containing the luminescent substituents such as, for example, naphthyl radicals [1, 24] for the design of liquid and solid scintillators with the given characteristics is promising.

In the present report with the purpose of obtaining new information about the rules of phosphorylation of electrophiles with the elementary phosphorus in the presence of bases, and the development of convenient method for preparing organophosphorus compounds with naphthyl radicals the reaction of 1-chloromethylnaphthalene with the white (P_4), red (P_n), and two modifications of the “activated red phosphorus” ($^{1*}P_n$ and $^{2*}P_n$) was studied. The samples of the “activated red phosphorus” were prepared by polymerization of white phosphorus in benzene at room temperature under the action of the ionizing radiation [26, 27].

It occured that the white, red phosphorus, and both modifications of the “activated red phosphorus” react with 1-chloro-methylnaphthalene in the KOH–H_2O–organic solvent (dioxane or benzene)–phase transfer catalyst (benzyltriethylammonium chloride) system to

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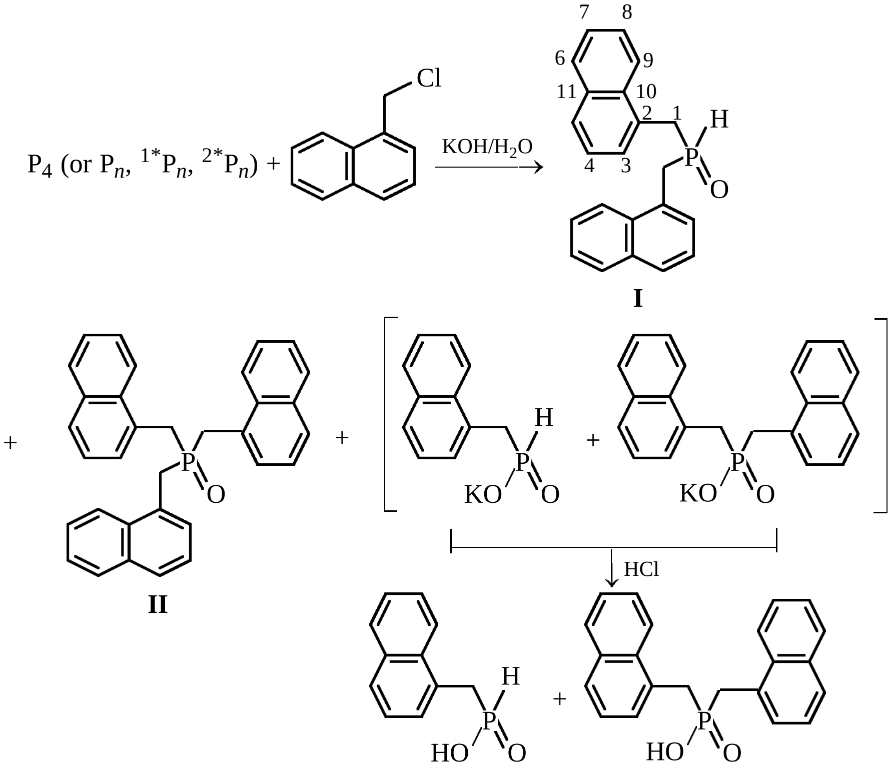
[1] For communication XVII, see [1].

give bis(1-naphthylmethyl)phosphine oxide **I**, tris(1-naphthylmethyl)phosphine oxide **II**, 1-naphthylmethylphosphonous **III** and bis(1-naphthylmethyl)phosphinic **IV** acids. Two latter compounds were isolated after treatment of the reaction mixture with the hydrochloric acid.



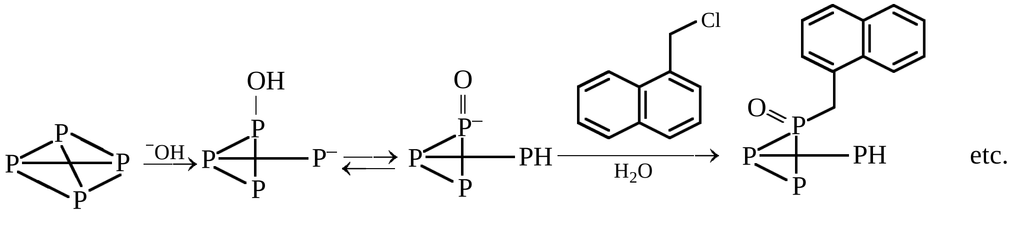
I

II

III

IV

From the table (exp. 1–4) it follows that phosphorylation of 1-chloromethylnaphthalene with the white and the ordinary red phosphorus at elevated temperature (78–95°C) proceeds practically quantitatively. In the first case the main reaction product is the secondary phosphine oxide **I** (exp. 1, 3), while in the case of red phosphorus tertiary phosphine oxide is mainly formed. Under these conditions it was obtained in 69% yield (exp. 2). The difference in behavior of white and red phosphorus in the reactions with electrophiles was previously observed on an example of phosphorylation of benzyl chloride [6], styrene, and 2-vinylnaphthalene [1] and was explained by the easier cleavage of P–P bonds in the rigid tetrahedral molecule of white phosphorus as compared to the macromolecule of red phosphorus, and hence by the greater concentration of phosphorus-centered nucleophiles which then react with 1-chloromethylnaphtalene to give mainly the products of incomplete naphthylmethylation of phosphorus **I, III**, and **IV**. At the same time no formation of corresponding organyl phosphines occured despite of performing all the stages of the experiment under the inert conditions. That means that polyphosphinite and phosphinite ions generated from the elemental phosphorus under the action of strong base exceed polyphosphide and phosphide ions in the competition for electrophile analogously to the previously presented schemes of phosphorylation of organyl halides and weakly electrophilic alkenes [1–4].



Phosphorylation of 1-chloromethylnaphthalene with elementary phosphorus under the conditions of interphase catalysis[a]

Run no.	P, mmol	(1-Chloro-methyl)-naphthlene, mmol	Tempera-ture, oC	Solvent, mol	Yield,[b] %				Conversion of phos-phorus, %
					$R_2P(O)H$ (**I**)	$R_3P=O$ (**II**)	$RP(O)H(OH)$[c] (**III**)	$R_2P(O)OH$[c] (**IV**)	
1	P_4, 33	7.0	90–95	Dioxane, 20	50	4	27	2	100
2	P_n, 33	6.8	90–95	Dioxane, 20	18	69	6	7	100
3	P_4, 33	9.0	78–80	Benzene, 40	45	29	10	15	100
4	P_n, 33	9.0	78–80	Benzene, 40	5	61	21	9	95
5	P_4, 33	6.8	22–24	Dioxane, 20	10	2	29	d	98
6	P_n, 33	6.8	22–24	Dioxane, 20	d	~1	d	~1	2
7	$^{1*}P_n$, 33	6.8	22–24	Dioxane, 20	13	8	23	2	75
8	$^{2*}P_n$, 9.7	1.9	22–24	Dioxane, 10	7	5	20	2	40

[a] In the experiments nos. 1–7 210 mmol of KOH, 5 ml of H_2O, and 0.88 mmol of benzyltriethylammonium chloride were used; in the experiment no. 8 62 mmol of KOH, 1.5 ml of H_2O, and 0.06 mmol of benzyltrimethylammonium chloride were used, heating for 3 h. [b] The yield is calculated on the involved amount of 1-chloromethylnaphthalene. [c] The acids **III** and **IV** were isolated from reaction mixture after treating of water layer with hydrochloric acid. [d] Not found.

At room temperature, red phosphorus practically does not react with 1-chloromethylnaphthalene (exp. 6). At the same time the reactivity of white phosphorus as well as of both samples of the activated red phosphorus ($^{1*}P_n$ and $^{2*}P_n$) under these conditions is much higher (the total yield of the product was 41, 46, and 34% respectively) and is comparable with one another (exp.5,7,8). Among the obtained substances **I-IV** the main product is 1-naphthylmethylphosphonous acid **III**. That means that the tendency to incomplete naphthylmethylation under these conditions also remains.

Note also the unexpected pathway of the reaction of 1-chloromethylnaphthalene with phosphine generated together with hydrogen from the red phosphorus and potassium hydroxide [28]. Bubbling of the phosphine-hydrogen mixture through the mixture of 1-chloromethylnaphthalene, KOH, and DMSO heated to 85–90°C gives 1-methylnaphthalene in 77% yield [29] instead of the expected products of phosphopylation. Hence, direct reaction of 1-chloromethylnaphthalene with elementary phosphorus in the presence of strong bases permits readily creation of a C–P bond and obtaining the promising organophosphorus compounds. Forexample tertiary phosphine oxide **II** is a luminophore. Its luminescence spectrum practically does not differ from that of naphthalene. Combination of the luminescent substituents and the phosphine oxide group capable of the selective complex formation with the rare earth elements is a principally new approach to creation of liquid and solid scintillators with given characteristics which can be used for the solving of such fundamental problem as the detecting of neutrino [25]. The acids **II** and **III** obtained are useful building blocks for the preplanned synthesis of phosphorus-containing peptide mimetics [30], of biologically active aminophosphonic acids [31–33], and of new organo-inorganic hybride materials [34].

EXPERIMENTAL

The IR spectra were recorded on a Specord IR-75 spectrometer. The ^1H, ^{13}C, and ^{31}P NMR spectra were taken on a Bruker DPX-400 spectrometer (400, 100, and 161.98 MHz respectively) in $CDCl_3$ against the internal HMDS. All the experiments were carried out under argon. The samples of the activated red phosphorus were obtained at room temperature by treating of the saturated solutions of white phosphorus in benzene with the ionizing irradiation of ^{60}Co with the dose rate 0.78 Gr s^{-1} for obtaining of $^{1*}P_n$ and 0.66 Gr s^{-1} in the presence of sulfur for obtaining of $^{2*}P_n$ until the the absorbed dose achieves 190 kGr. The samples of the activated red phosphorus obtained were washed with benzene from the unreacted white phosphorus and dried in a vacuum.

Reaction of 1-chloromethylnaphthalene with white phosphorus in dioxane at 90–95°C. Synthesis of bis(1-naphthylmethyl)phosphine oxide (I) and 1-naphthylmethylphosphonous acid (III) (exp. 1). To a mixture of 1.25 g of 1-chloromethylnaphthalene, 0.17 g of triethylbenzylammonium chloride and 1.04 g of white phosphorus in 20 ml of dioxane 11.95 g of KOH was added. After that 5 ml of water was added dropwise with stirring for 1 min. The reaction mixture was stirred for 3 h at 90–95°C under argon, cooled, diluted twice with water and extracted with chloroform. The chloroform extract was dried with potassium carbonate, chloroform was distilled off, and the residue was kept in a vacuum to give 0.76 g of oil. Its ^{31}P NMR spectrum contained following signals: –125.36 ppm ($^{1}J_{\mathrm{PH}}$ 192.7 Hz), 1-naphthylmethylphosphsphine; 35.67 ppm ($^{1}J_{\mathrm{PH}}$ 475.6 Hz), phosphine oxide **I**; 42.44 ppm, phosphine oxide **II**. The intensity ratio was 1 : 1.3 : 20 respectively. The residue was dissolved in hexane, filtered, and the hexane was removed to give 0.58 g (50%) of phosphine oxide **I**. IR spectrum, ν, cm^{-1} (KBr): 3050, 3030 ν(=CH$_{\mathrm{arom}}$), 2970, 2930, 2860 [ν(CH)], 2350 [ν(PH)], 1590, 1500, 1400, 1390 [ν(C=C$_{\mathrm{arom}}$)], 820, 790, 760 δ(PC, CH$_{\mathrm{arom}}$). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.8 d (1H, PH, $^{1}J_{\mathrm{PH}}$ 470), 3.52 d (4H, CH$_2$, $^{2}J_{\mathrm{PH}}$ 14.3), 7.35–7.53 m and 7.62–7.89 m (14H, $C_{10}H_7$). ^{31}P NMR spectrum, δ_{P}, ppm: 35.67 d, $^{1}J_{\mathrm{PH}}$ 475.6 Hz.

The water layer was treated with 30% hydrochloric acid to pH 4-5, extracted with chloroform and dried over calcium chloride. Chloroform was distilled off and the residue was kept in a vacuum to give 0.41 g of the product. Its ^{31}P NMR spectrum contained following signals: 31.88 ppm, $^{1}J_{\mathrm{PH}}$ 580 Hz, the acid **III**; 51.7O ppm, the acid **IV**. The ratio of intensities was 19.5 : 1 respectively. This mixture was five times precipitated in hexane from acetone to give 0.39 g (27%) of the acid **III**, mp 115–117°C. IR spectrum, ν, cm^{-1} (KBr): 3450 [ν(OH)], 3030 [ν(=CH$_{\mathrm{arom}}$)], 2950, 2910, 2850 [ν(CH)], 2360 [ν(PH)], 1620 [δ(OH)], 1590, 1500, 1390 [ν(C=C$_{\mathrm{arom}}$)], 1270, 1250 [δ(CH$_{\mathrm{arom}}$)], 1180 [ν(P=O)], 1170 {ν[P–O(H)]}, 970, 950, 690, 660 [δ(PC, CH$_{\mathrm{arom}}$)]. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.58 d (2H, CH$_2$P, $^{1}J_{\mathrm{PH}}$ 13.7), 7.34–7.92 m (7H, $C_{10}H_7$), 16.0 (1H, OH). ^{13}C NMR spectrum, δ_{C}, ppm (*J*, Hz): 34.22 d (C^{1}, $^{1}J_{\mathrm{PC}}$ 93.0), 123.53 (C^{9}), 125.75 (C^{8}), 125.32 (C^{4,7}), 125.75 (C^{8}), 126.30 (C^{5}), 127.96 (C^{3}), 128.35 (C^{10}), 128.59 (C^{6}), 131.77 (C^{11}), 133.71 (C^{2}). ^{31}P NMR spectrum, δ_{P}, ppm: 33.11 d, $^{1}J_{\mathrm{PH}}$ 557 Hz. Found, %: C 64.07; H 5.35; P 14.55. $C_{11}H_{11}O_2P$. Calculated, %: C 64.08; H 5.38; P 15.02.

Reaction of 1-(chloromethyl)naphthalene with red phosphorus in KOH-dioxane system at 90–95°C. Synthesis of tris(1-naphthylmethyl)phosphine oxide (II) (exp. 1). To a mixture of 1.04 g of red phosphorus, 1.2 g of 1-(chloromethyl)naphthalene, 0.17 g of triethylbenzylammonium chloride, and 11.95 g of KOH in 20 ml of dioxane 5 ml of water was added dropwise in 1 min. Reaction mixture was stirred for 3 h at 90–95°C, diluted with water, and extracted with chloroform. Chloroform extract was dried over potassium carbonate, chloroform was distilled off, and the residue was kept in a vacuum to give 0.94 g of the product. Its ^{31}P NMR spectrum contained the signals of phosphine oxides **I** and **II**, phosphonous acid **III**, and phosphinic acid **IV** in 2.5 : 8.8 : 1 : 1 ratio. Obtained mixture was precipitated in hexane from chloroform to give 0.73 g of phosphine oxide **II**, mp 260°C (from benzene). IR spectrum (KBr), ν, cm^{-1}: 3050, 3030 [ν(=CH$_{\mathrm{arom}}$)], 2950, 2910, 2850 [ν(C–H)], 1590, 1500, 1400, 1390 [ν(C=C$_{\mathrm{arom}}$)], 1170 [ν(P=O)], 820, 790, 760 [δ[PC, CH$_{\mathrm{arom}}$)]. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.49 d (6H, CH$_2$P, $^{2}J_{\mathrm{PH}}$ 13.5), 7.35–7.82 m (21H, $C_{10}H_7$). ^{13}C NMR spectrum, δ_{C}, ppm (*J*, Hz): 33.15 d (C^{1}, $^{1}J_{\mathrm{PC}}$ 60.4), 124.10 (C^{9}), 125.51 (C^{4}, C^{7}), 125.91 (C^{8}), 126.45 (C^{5}), 127.89 d (C^{3}), 128.28, 128.34 (C^{10}), 128.91 (C^{6}), 132.53 (C^{11}), 134.13 (C^{2}). ^{31}P NMR spectrum, δ_{P}, ppm: 42.44. Found, %: C 84.18; H 5.83; P 6.91. $C_{33}H_{27}OP$. Calculated, %: C 84.23; H 5.78; P 6.58.

The water layer was treated with 30% hydrochloric acid to pH 4–5, extracted with chloroform and dried over calcium chloride. Chloroform was distilled off, and the residue was dried in a vacuum to give 0.08 g of phosphonous acid **III**.

Reaction of 1-chloromethylnaphthalene with white phosphorus in benzene at 78–80°C. Synthesis of bis(1-naphthylmethyl)phosphinic acid (IV) (exp. no. 3). To a mixture of 1.04 g of white phosphorus, 11.95 g of KOH, 0.34 g of triethylbenzylammonium chloride, 5 ml of water, and 30 ml of benzene a solution of 1.63 g of 1-(chloromethyl)naphthalene in 10 ml of benzene was added dropwise for 1.5 h. Reaction mixture was stirred for 3 h at 78°C, diluted with water, and extracted with chloroform. Chloroform extract was dried with potassium carbonate, chloroform was distilled off, and the residue was kept in a vacuum to give 0.34 g of the product. Its ^{31}P NMR spectrum contained the signals of phosphine oxide **II** and the acid **IV** in 7.5 : 1 ratio. Water layer was treated with 30% hydrochloric acid to pH 4–5, extracted with chloroform, dried over calcium chloride, and the solvent was distilled off. The residue was kept in a vacuun to give 1.67 g of the product. Its ^{31}P NMR spectrum contained the signals of the acids **III** and **IV** in 5.4 : 1 ratio. Obtained mixture was washed with hot hexane, and solvent was removed to give 0.24 g (15%) of the acid **IV**, mp 93–94°C (from hexane). IR spectrum (KBr), ν, cm^{-1}: 3430 [ν(OH)], 3030 [ν(C=CH$_{\mathrm{arom}}$)], 2950, 2900, 2850 [ν(CH)], 1660 [δ(OH)], 1590, 1500, 1390 [ν(C=C$_{\mathrm{arom}}$)], 1150 [ν(P=O)], 950, 760 [δ(P–C, CH_{arom})]. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.23 d (4H, CH_2P, $^1J_{PH}$ 16.4), 7.23–7.72 m (14H, $C_{10}H_7$), 9.61 (1H, OH). ^{13}C NMR spectrum, δ_C, ppm (*J*, Hz): 32.85 d (C^1, $^1J_{PC}$ 88.7), 123.92 (C^9), 124.88 (C^4, C^7), 125.20 (C^8), 125.67 (C^5), 127.18 (C^3), 127.34, 127.45 (C^{10}), 128.11, 128.20 (C^6), 131.68 and 131.73 (C^{11}), 133.48 (C^2). ^{31}P NMR spectrum, δ_P, ppm: 51.74. Found, %: C 72.34; H 5.90; P 8.78. $C_{22}H_{19}O_2P$. Calculated, %: C 76.29; H 5.53; P 8.94.

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