The Synthesis of Organophosphorus Compounds from Phosphine and Alcohols in the Presence of Quinones and Redox Polymers on their Basis

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Abstract

This article presents the synthesis of phosphoric acid esters from phosphoric hydrogen PH₃ and aliphatic alcohols. The process is based on the oxidation of phosphine by quinones and redox polymers on the basis of mono- and disubstituted quinoid derivatives of monoethanolamine vinyl ether. Molecular iodine is used as a catalyst. Two-, three-, four- and multicomp onent systems are studied in order to determine optimal conditions of the oxidation of phosphine quinones and repoxpolymers on their basis. The rate and selectivity of reaction were monitored by the absorption of PH₃. As alcohols used aliphatic alcohols: BuOH, PrOH, EtOH, MeOH. Organophosphorus compounds were analyzed by a chromatographic method. It was established that alcohol solutions of individual components of reactionary system (quinones, redox ionites or iodine) are characterized by a low activity in relation to phosphine. Organophosphorus compounds are formed in insignificant quantities. In the mixed alcohol solution of benzoquinone takes place PH₃ oxidation forming trialkylphosphates. Conversion of phosphine constitutes 80-100%. Increasing the concentration of reagents of catalytic system has a positive effect on the process as a whole. Similar patterns were obtained when redox monomers and polymers on the base of quinones in the presence of iodine were used as oxidants. Esters of phosphoric acid - dialkylphosphites and esters of phosphorous acid – trialkylphosphates were identified as organophosphorus compounds. By selecting a redox agent in a zone of the catalysis it is possible to direct process in the desirable direction. The most activity in the oxidation of phosphine by iodine-alcohol solutions of quinoid monomers and polymers, is exhibited by 2-[N-(2-vinyloxy) ethyl]amino-NQ and polymer on their basis. Results of our experiments and literature data on oxidation-reduction processes with participation of iodine and quinones in organic solutions allowed to propose the separate oxidation-reduction mechanism of formation of organophosphorus compounds. In investigated multicomponent systems, the synergetic effect is manifested which is reached at the expense of distribution of oxidation-reduction functions among iodine, quinones and its derivatives. In this report, for the first time, it is established reactionary ability of quinones and redox ionites on their basis in an oxidizing alkoxylation of phosphine to valuable esters of acids of phosphorus. Reaction can be used for purification of exhaust and technological gases from phosphine and its utilization.

Introduction

Phosphin (PH₃) is formed as toxic impurity during electro-thermal production of yellow

phosphorus in processes with participation of phosphides of metals and when acetylene is obtained. Maximally admissible concentration of PH₃ in a working zone of production rooms is 0.1 mg/m³. In this regard the problem of purification of

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exhaust and technological gases from toxic phosphine is a topical issue from the ecological point of view.

At the same time phosphine is a universal obtaining raw material for valuable organophosphorus compounds (OPC) in а laboratory and industrial practice. Traditional methods for obtaining important classes of the OPC, including organic phosphines, phosphinhalkogenides, ethers acids of of phosphorus are based on aggressive and sensitive to moisture halogenides of phosphorus. Realization of these methods is accompanied by formation of significant amounts of a difficult utilized dangerous waste [1]. Therefore currently the increasing attention is given to the development of "non chlorine" methods of synthesis of OPC directly from the elemental phosphorus, phosphine and phosphides of transient metals as alternative phosphorylated agents [2, 3].

Generally, phosphin is considered very active in radical-chain of reactions and less active in heterolytic organic transformations [1]. The considerable number of publications are devoted to the chemistry of functional replaced phosphines and their derivatives – alkyl- and arylphosphines. The area of their application is extremely various [4-6]. However use of PH₃ for formation of P-O, P-C - bonds and synthesis organic and inorganic phosphines is very limited in the absence of effective technological methods of its obtaining.

Known organic reactions of phosphine proceed in hard conditions: under the influence of high pressures, temperatures, the superacids, the superbases, and in the presence of initiators of free radicals. Synthesis, on the basis of PH₃, is used very rarely and it is not realized in the industry because there are no effective technological methods of its Trofimov with colleagues [7-14] obtaining. developed in recent years convenient methods of synthesis of organophosphorus compounds on the basis of phosphine, generated in the water and organic medium from red phosphorus in the presence of the superstrong bases. Phosphin can be used for obtaining various OPhC which are important supplementary reagents, construction

blocks, semi-products, and also ligands for metalcomplex catalysts, extragents and antipyrenes. High reactionary ability of phosphine is the objective precondition of its wider involvement in phosphor organic synthesis, despite many obvious advantages in comparison with phosphorus chlorides. In spite of the fact that hydride of phosphorus is quite strong reducing agent, in the absence of catalysts it does not oxidize with appreciable speed by any of known acceptors of electrons.

The purpose of this research is a development of new ecologically safe catalytic synthesis of esters of phosphorus and phosphoric acids from PH_3 with the use of an oxidizer of quinones and redoxionites on their basis.

Ethers of phosphoric acid - dialkylphosphites are widely applied in organic synthesis for obtaining medicines and biologically active substances. Ethers of phosphoric acid trialkylphosphates are used as extragents of rare and radioactive elements from mineral raw materials and wastes of nuclear power, solvents of varnishes and paints in printing industry, additives to combustive-lubricating materials, softeners, antipyrenes, etc. World production of OPhC composes tens of thousands tons per year [1].

Experimental

Syntheses based on PH₃ was carried out in a thermostated glass reactor type catalyst "duck" with a potentiometric device under vigorous stirring. The gas mix of Ar-PH3 was obtained by acid decomposition of Zn₃P₂, then it was dried up by granulated NaOH. Alcohols were cleared by distillation. The analysis of gases on the content of PH₃ carried out iodometric and colorimetric methods. The rate of reaction was monitored by the absorption of PH₃. Chromatographic analysis for alcohols, acetals, dioxane, quinone, hydroquinone, and OPhC was performed using a flame ionization detector and steel columns (300×0.3 cm). The columns were filled with unsilaneted chromaton soaked with apiezon L (5%) in the chromatograph model 3700 and Chrom-5. For oxidative

alkoxylation of PH_3 aliphatic alcohols (butyl – BuOH, propyl – PrOH, ethyl – EtOH, methyl – MeOH) in a mixture with dioxane, toluene or DMFA were used. Benzoquinone (BQ), 1.2naphthoquinone (NQ), its mono- and disubstituted derivatives of monoethanolamine vinyl ether (VEMA) and polymers on their basis next structure were used as oxidants (Table 1) [15, 16]:

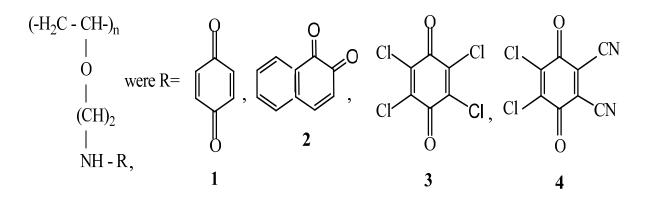


 Table 1

 Mono- and disubstituted quinoid derivatives of VEMA

N	Monomer	Brutto-formula	Polymer
1	2,5-bis [N-(2-vinyloxy) ethyl]amino-BQ - M ₁	$C_{14}H_{18}N_2O_4$	RP_1
2	2-[N-(2-vinyloxy) ethyl]amino-(NQ) - M_2	C ₁₄ H ₁₃ NO ₃	RP ₂
3	2,5-bis[N-(2-vinyloxy)ethyl]amino-3,6-dichloro-BQ - M ₃	$C_{14}H_{16}CI_2N_2O_4$	RP ₃
4	2-[N-(2-vinyloxy)ethyl]amino-3-chloro-5,6-dicyan-BQ - M ₄	C ₁₂ H ₈ ClN ₃ O ₃	RP ₄
5	1,2-naphthoquinone - M ₅	$C_{10}H_6O_2$	-

The experimental procedure is described in detail in publications [3]. Products of catalytic reactions of oxidation of PH₃ in butanolic solutions of benzoquinone, redox-polymers on its basis and iodine obtained with vacuum distillation at pressure of 2-5 mm mercury column. Obtained OPC identified GL chromatography methods, NMR³¹P-, IR-spectroscopy, and also at temperatures of boiling and refraction indicators. IR-spectra obtained OPC registered on the Specord M80/M85, spectra NMR³¹P - on "Bruker" AHM-400 and WP-80 spectrometer in relation to 75% H₃PO₄, indicators of refraction (n_D²⁰) are established on the IRF-454 refractometer.

Results and Discussion

By the methods of chemical modeling with use of two - three - and four-componential systems

were studied the main stages of catalytic synthesis and influence of structure of the reactionary medium on speed and selectivity of process.

The system of $PH_3 - BQ - ROH - C_4H_8O_2$. Character of products of reaction of derivatives of P (III) with the benzoquinone depend on its high redox-potential, presence of the electrophilic and nucleophilic centers. Phosphites join carbonyl oxygen, tertiary phosphines depending on basicity react either with carbonyl, or with nonsaturated carbon. Secondary phosphines form oxides tertiary phosphines. Primary phosphines - are oxidized to diphosphines.

The behavior of PH_3 in contrast to other compounds P (III), including P-H group, in reactions with quinones has not been studied [1, 17]. We found that the dioxane-alcoholic solution of BQ (3.7 mmol) at 80 °C shows low activity and absorbs PH_3 (10³ Pa) at low velocity. Potential of the system slightly shifted to the cathode side. OPC in appreciable amounts were not detected (Table 2, experiment 1).

The system of $PH_3 - I_2 - ROH - C_4H_8O_2$. Dioxane-alcoholic solution of I_2 (0.2-3.0 mmol) is quite stable at 50 °C. For a long period of time the red-brown color of the solution does not change, the oxidation products of alcohol do not form. The introduction of PH₃ in the test solution is accompanied by absorption of PH₃, by sharp shift in the cathodic potential direction and bleaching of the initial solution. Increasing the concentration of I₂ has a positive effect on the reaction rate (W, M/min) and the amount of absorbed phosphine (Q, M). This system produces mainly inorganic phosphites and alkyl iodides [18]. An ester of phosphoric acid trialkylphosphate (RO)₃PO with 10-15% of yield (experiments 2-4) is identified as a OPhC.

N⁰	I ₂ ,	$C_6H_4O_2$,	ROH,	PH ₃ ·10 ⁻³ ,	T°,C	W $\cdot 10^3$,	$Q \cdot 10^2$,	(RO) ₃ PO,
	mole	mmole	ml	Ра		M/min	М	%
			BuOH					
1	-	3.7	10	1.7	80	0.5	0.2	-
2	0.2	-	10	1.7	50	1.0	0.5	10
3	1.5	-	10	1.7	50	5.2	3.8	12
4	3.0	-	10	1.7	50	6.8	5.9	15
5	0.4	7.4	10	1.7	80	10.2	6.4	100
6	0.6	7.4	10	1.7	80	11.5	9.0	100
7	1.2	7.4	10	1.0	80	14.0	10.1	84
8	0.6	3.7	10	1.7	80	10.9	6.0	80
9	0.6	14.8	10	1.7	80	14.0	8.9	87
10	1.2	7.4	10	0.7	80	10.5	10.8	90
11	0.6	7.4	10	1.7	60	12.8	12.0	80
12	0.6	7.4	10	1.7	40	11.5	11.0	80
13	0.6	7.4	5	1.7	80	12.8	10.2	100
14	0.6	7.4	2	1.7	80	11,5	8.6	90
			PrOH					
15	0.6	3.7	2	1.0	60	9.6	11.6	84
16	0.6	7.4	2	1.0	60	11.6	13.7	94
17	0.6	11.0	2	1.5	60	13.7	18.1	94
			EtOH					
18	0.6	7.4	2	0.9	50	7.7	11.3	80
19	1.2	7.4	2	0.9	50	9.3	11.7	86
			МеОН					
20	0.4	7.4	2	0.9	40	6.2	7.3	85
21	0.6	7.4	2	0.9	40	7.5	13.5	100
22	1.2	7.4	2	0.9	40	9.3	12.0	82
23	0.6	3.7	2	0.9	40	5.6	6.5	80

Table 2The oxidation of PH_3 in dioxane-alcohol solutions of iodine and BQ

The system of $PH_3 - BQ - I_2 - ROH - C_4H_8O_2$. In the presence of a mixed system of I_2 (0.4-1.2 mmol) and BQ (3.7-14.8 mmol), the growth rate of the reaction, the amount of absorbed PH_3 and yield of OPhC are observed at 40-80 °C. The oxidation of PH_3 by BQ takes place in the studied conditions. This is accompanied by preferential formation of trialkyl phosphate (1) with a yield of 80-100% (experiment 5-23)

$$4C_6H_4O_2 + PH_3 + 5ROH \longrightarrow (RO)_3PO + 4C_6H_4(OH)_2 + R_2O$$
 (1)

With increasing concentration of iodine, BQ, BuOH, the partial pressure of PH_3 and the temperature, the reaction rate and the amount of absorbed phosphine increase. The quantity absorbed phosphine depends on concentration of an oxidizer. The high selectivity of the process is preserved (experiment 5-14). Similar patterns were obtained at using dioxane-alcohol solutions PrOH, EtOH and MeOH (experiment 5-23).

The system of PH_3-I_2-BuOH -redox monomers (M_2 , M_5) and redox polymers (RP_{1-4}) In these systems, the amount of introduced monomer was 0.2 g (0,72-1,26 mmol), and DMFA and C_7H_8 were used as the solvent. In the absence of iodine butanol solution (21,8 mmol) of redox polymers RP_2 oxidized PH_3 at 50°C at low speed. The formation of OPhC was not observed (Table 3, experiment 1).

N⁰	I ₂ ,	BuOH,	PH ₃ ·10 ⁻³ ,	$W_{PH_3} \cdot 10^3$,	$Q_{PH3} \cdot 10^2$,	(RO) ₂ HPO,	(RO) ₃ PO,
	mmole-RP _{1-4,} $M_{2,}M_{5}$	ml	Ра	M/min	М	%	%
1	RP ₂	2	1.0	0.2	0.2	-	-
2	$0.2 - RP_1$	2	1.0	1.9	1.1	17	7
3	$0.2 - RP_2$	2	1.0	1.5	2.1	32	6
4	$0.2 - RP_3$	2	1.0	1.1	1.2	-	-
5	$0.2 - RP_4$	2	1.0	0.8	0.8	-	-
6	$0.2 - M_5$	2	1,0	1.3	2.1	26	9
7	$0.2 - M_2$	2	1.0	2.3	2.3	20	14
^a 8	$0.2 - M_2$	6	1.3	2.8	2.7	10	52
^a 9	$0.2 - M_2$	8	1.3	2.7	3.5	21	42
^a 10	$0.2 - M_2$	10	1.3	3.0	1.8	62	38
^a 11	$0.2 - RP_2$	10	1.3	3.0	2.3	64	36
12	0.2 - M ₂	10	1.7	1.9	2.0	49	51
13	0.4 - M ₂	10	1.7	2.5	3.2	39	51
14	0.7- M ₂	10	1.7	4.5	3.8	40	60
15	$1.5 - M_2$	10	1.7	5.1	4.1	30	70

Table 3The oxidation of PH_3 in alcoholic solutions* of quinoid M and RP^{**} and iodine

* Reaction conditions: 50°C, RP₁₋₄, M₂, M₅ 0.2 g, solvent DMFA, C₇H₈

**RP₁0.72; RP₂0.82; RP₃0.68; RP₄0.72; M₂0.82; M₅1.26 mmole; ^a at 70°C

The insignificant additions of iodine (0.2)mmole) in the test solution increased the reaction rate, the amount of absorbed PH₃ and yield of the OPhC. Unlike the iodine-alcohol solution BQ, in the presence of RP₂, dialkyl phosphytes (RO)₂HPO and trialkyl phosphates (RO)₃PO with total yield of 38% (experiment 2) were formed. In butanol solutions of RP₃ and RP₄ at 50 °C, the formation of the OPhC in appreciable amounts is practically not observed (experiments 3 and 4), whereas at use of RP₁, RP₂, M₅ or M₂ in iodine-alcohol solution the yield of OPhC rather increases and reached 24-38% (experiments 2. 5-7). The highest activity showed a redox monomer M₂ and its polymer RP₂. Increasing the concentration of BuOH to 109 mmol, the temperature to 70 °C in iodine alcohol solutions RP₂ and M₂ contributed to complete conversion of PH₃ forming 10-64% dialkylphosphite and 36-52%

trialkylphosphate (experiments 8-11).

With the increase in iodine concentration to 1.5 mmol in butanol solutions M_2 at 50°C, the reaction rate and the amount of absorbed PH₃ increased. A high yield of OPhC was preserved. Under such conditions, the yield of tributyl phosphate increased to 70%, the content of dibutylphosphite decreased to 30% (experiments 12-15). Thus, it is possible to direct process in the desirable direction by the corresponding selection a redox agent in a zone of catalysis.

Fig. 1. presents typical conversion (a) and potentiometric (b) curves of PH_3 absorption by iodine-butanol solutions of investigated redox systems. Insignificant at the beginning of experiment, the speed of reaction reaches its maximum in the process of PH_3 absorption, and then gradually falls.

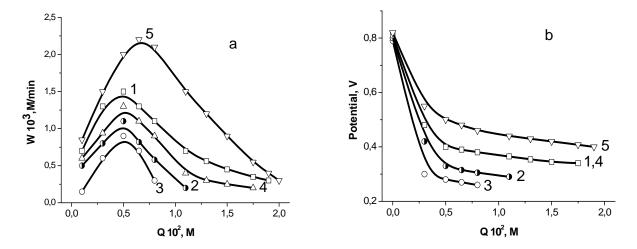


Fig. 1. Conversion (a) and potentiometric (b) curves of PH₃ absorption by iodine-alcohol solutions of redox polymer at 50 °C (PH₃ 1.0×10³ Pa, BuOH 21.8, DMFA 10.3, I₂ 0.2 mmol/l): 1 - RP₂, 2 - RP₃, 3 - RP₄, 4 - M₅, 5 - M₂ (0.2 g).

At the same time shift of redox-potential in cathodic area (b) is observed. This evidence of reduction of components of reactionary solution (iodine and redox polymers) by PH₃. The highest speed shows redox systems on the basis of M_2 monomer (monosubstituted derivative VEMA on the basis of NQ) (a curve 5) and RP₂ polymer (a curve 1).

In the presence of NQ (a curve 4) the speed of absorption of PH_3 is much lower. Low speeds of

oxidation of PH_3 are observed for polymers on the basis of chlorosubstituted quinones (curves 2 and 3).

The quantity of the absorbed PH_3 with the iodine-alcohol solutions, containing quinone and polymers on their basis (Tables 2, 3; Fig. 1), corresponds to stoichiometric coefficient and evidence for the participation of quinone and its derivatives in reaction of an oxidizing alkoxylation of PH_3 to esters of acids of phosphorus. The obtained products of reaction of oxidation of PH_3

identified at boiling temperatures, refraction indicators, at IR- and NMR³¹P spectra. In IR- spectra absorption bands of valency oscillations of P=O groups (1260-1350 cm⁻¹), P-O-C (1032-1140 cm⁻¹) and P-H (2430 cm⁻¹) are observed. Spectra NMR³¹P contain characteristic shifts for P(O)H(OBu)₂ in the field of -1-1 ppm., for P(O)(OBu)₃ in the field of 2.8-8 ppm. Boiling temperatures, refraction indicators for obtained R(O)H(OBu)₂ make 100-108 °C/2-3 mm mercury column, $n_D^{20}=1,4254$; for $P(O)(OBu)_3 - 108-120^{\circ}C/2-3 \text{ mm mercury column},$ $n_D^{20}=1,4250$. The obtained physical and chemical dialkylphosphites characteristics of and

thryalkylphosphates corresponds to reference manual data.

Results of our experiments and literature data on oxidation-reduction processes with participation of iodine, quinones and redox-ionites on their basis [15-17] in organic solutions allowed to propose the separate oxidation-reduction mechanism of formation of organophosphorus compounds. Reaction of an oxidizing alkoxylation of PH₃ proceeds on the following key stages: reduction of I₂ with phosphine to dialkylphosphite (2) and trialkylphosphite (3):

$$PH_3 + 3I_2 + 3ROH \longrightarrow (RO)_2 HPO + RI + 5HI$$
(2)

$$PH_3 + 4I_2 + 4ROH \longrightarrow (RO)_3PO + RI + 7HI$$
(3)

and the oxidation of HI by BQ, quinoid monomers or redoxites (4):

$$C_6H_4O_2 + 2HI \longrightarrow I_2 + C_6H_4(OH)_2$$
(4)

In investigated multicomponent systems, the synergetic effect is manifested which is reached at the expense of distribution of oxidation-reduction functions among iodine, quinones and redox polymers on their basis.

Thus, in this research possibility of obtaining valuable ethers of acids of phosphorus from phosphine and aliphatic alcohols at presence of quinones and redox ionites on their basis is shown. Reaction can be used for purification of exhaust and technological gases from phosphine and its utilization in industrially important products.

Conclusion

Thus, on the basis of the carried out studies it was established that:

1) the individual components of the alcohol reaction solution: iodine, quinones and redox polymers on their basis in interaction with PH_3 in appreciable amounts do not form the organophosphorus compounds;

2) in the mixed iodine-alcohol solutions of quinones and its derivatives proceeds the reaction of oxidizing alkoxylation of phosphine to dialkylphosphite and trialkylphosphate with a total yield of 100 %;

3) the most activity, in the oxidation of phosphine by iodine-alcohol solutions of quinoid monomers and polymers, is exhibited by 2-[N-(2-vinyloxy) ethyl]amino-NQ and polymer on their basis;

4) key oxidation-reduction stages of oxidizing alkoxylation of PH₃ in iodine-alcoholic solutions of quinones, quinoid derivatives of vinyl ether of monoethanolamine and quinoid redox polymers to organophosphorus compounds are proposed.

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