

## Oxidation of Phosphine with Quinone and Quinoid Redox Polymers in Alcohol Solutions of Copper

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**Abstract**—New quinoid redox polymers were obtained by chemical modification of commercial weakly basic anion exchangers with quinone and its derivatives. The redox properties of quinone and quinoid redoxites with respect to phosphine were studied in alcohol solutions of copper complexes.

**Keywords:** quinones, redox polymers, weakly basic anion exchangers, phosphine, oxidation, butanol, copper carboxylates

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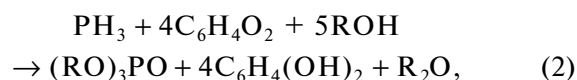
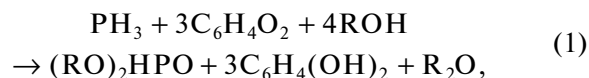
Redox polymers is one of the popular classes of high-molecular compounds due to their ability to be involved in both redox and ion exchange or complexation reactions [1, 2]. Quinoid redoxites are most promising [3, 4] in view of the wide spread of quinones in nature and living organisms. Their biological activity is primarily determined by their ability to undergo the reversible redox process, due to which they are multifunctional and have diverse properties. Many redox processes occurring in the living nature are based on the transformations of quinones. The introduction of electron-accepting substituents into quinones increases their oxidative ability.

The methods based on the reduction of the number of stages in the synthesis of redoxites are promising from the viewpoint of green chemistry [5–7]. This is achieved by using industrial weakly basic anion exchangers containing primary and/or secondary amino groups as the starting matrices for modification. Earlier, metal-containing electron ion exchangers (EIEs) were obtained from commercial anion or cation exchange resins by depositing variable-valence metals for effective removal of dissolved oxygen from desalinated water [8–10]. Previously, we obtained condensation redox polymers by chemical modification of commercial AN-31 anionite with benzoquinone (BQ) and quinones containing electron-accepting substituents under nearly green-chemistry conditions. The good solubility and high degree of swelling of the starting anionites in the majority of organic solvents provide high conversion of the primary and secondary amino groups of anionites. The synthesis was

performed using environmentally friendly solvents (ethanol, water, or their mixtures) [11, 12].

Previously, the oxidative alkoxylation of phosphine (PH<sub>3</sub>) in solutions of molecular iodine, *p*-benzoquinone, and redox polymers based on monoethanolamine vinyl ether (MEAVE), 1,4-benzoquinone,  $\alpha$ - and  $\beta$ -naphthoquinone, and chlorine- and CN-substituted quinines, forming valuable phosphoric acid ethers, namely, dialkylphosphites (RO)<sub>2</sub>HPO **1** and trialkylphosphates (RO)<sub>3</sub>PO **2** was found. The key stages of the process were assumed to be the oxidation of PH<sub>3</sub> with iodine to organophosphorus compounds (OPCs) and reoxidation of the iodide ion with quinone [13–16].

The present paper describes the kinetic regularities of the oxidative alkoxylation of phosphine into phosphoric acid ethers in the presence of a polyfunctional catalytic system (PFCS) containing quinone or condensation redoxites and CuX<sub>2</sub> (X = Cl, Br, CH<sub>3</sub>CO<sub>2</sub>, C<sub>3</sub>H<sub>7</sub>CO<sub>2</sub>, and C<sub>17</sub>H<sub>35</sub>CO<sub>2</sub>):



R = BuOH.

The reaction was studied by the kinetics method, redox potentiometry, gas chromatography, and quantitative analysis.

## EXPERIMENTAL

The kinetic regularities of the oxidation of  $\text{PH}_3$  in alcohol solutions of PFCS were studied based on phosphine absorption in a flow-through unit with a vigorously agitated isothermal reactor to provide the kinetic mode and gradientless conditions. The experimental procedure was described in detail in [14, 16]. During the experiments, the experiment time (min), redox potential (V),  $\text{PH}_3$  absorption rate ( $W$ , mol/(L min)), and amount of absorbed  $\text{PH}_3$  ( $Q$ , M) were continuously measured, and the composition of the liquid and gas phases was periodically analyzed. The rate of reactions (1) and (2) was assessed from the consumption of  $\text{PH}_3$ , quinone, and accumulation of organophosphorus products. The test solutions were regenerated after phosphine absorption with air oxygen. The quantitative analysis of the organophosphorus products, quinone, and hydroquinone relative to the standard samples was performed on a GC-2010 Plus chromatograph equipped with a flame ionization detector and Supelco SMS (30 m  $\times$  0.25 mm) (Shimadzu) capillary columns. Hydrogen was used as the carrier gas. The initial and final temperatures were 120 and 220°C; the initial and end time 0–7 min. The heating rate was 25 K/min; the detector temperature 300°C.

Quinoid redox ionites synthesized at the Institute of Chemical Sciences were used; the commercial weakly basic anionite AN-31 was used as the starting matrix. The redox capacity of the samples based on the 0.1 N solution of  $\text{Fe}_2(\text{SO}_4)_3$  was 1.3–2.9 mg-eq/g; the anion exchange capacity 1.7–6.3 mg-eq/g; the redox potential 0.69 V. The IR spectra of the synthesized redoxites have intense characteristic absorption bands related to the stretching vibrations of the C=O (1653  $\text{cm}^{-1}$ ) and phenol –C–O– (1210  $\text{cm}^{-1}$ ) bonds of the quinoid ring, as well as the  $>\text{C}=\text{C}<$  (1501  $\text{cm}^{-1}$ ), =NH– (1580  $\text{cm}^{-1}$ ), and C–N (1340  $\text{cm}^{-1}$ ) bonds, which confirms the presence of aminoquinoid polymers.

## RESULTS AND DISCUSSION

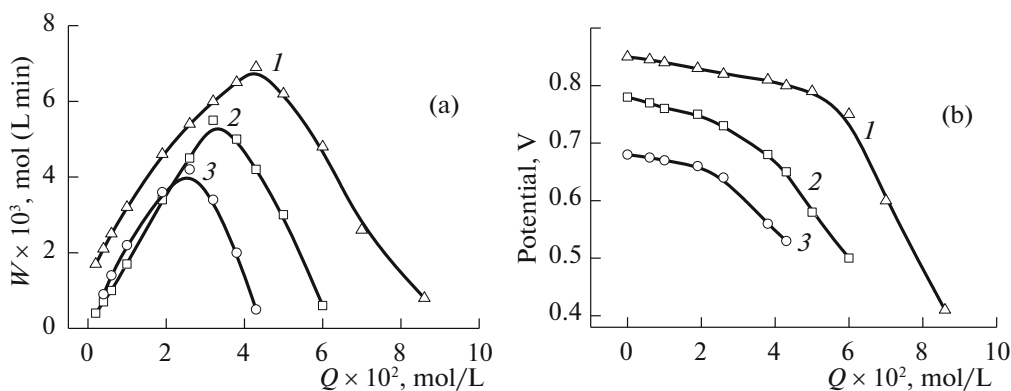
The quinone–hydroquinone system serves as a classic example of an organic redox system and is often used as an oxidizing agent and a component of the catalytic system in liquid-phase processes. The main chemical function of quinones in chemical and biochemical processes is the electron transfer. Quinones are single- and two-electron oxidizers of medium strength; hydroquinones are relatively easily oxidizable molecules. Therefore, quinones can serve as both oxidants and catalysts. In addition, quinones occasionally act as ligands in metal complex catalysis [17–19].

The products of the reactions of benzoquinone with P(III) derivatives ( $\text{PH}_3$ ,  $\text{PhH}_2\text{P}$ ,  $\text{Ph}_2\text{HP}$ ,  $\text{Ph}_3\text{P}$ ,  $\text{R}_3\text{P}$ ,  $\text{Ph}_2\text{HPO}$ ,  $(\text{HO})\text{H}_2\text{PO}$ ,  $(\text{RO})_3\text{P}$ , and  $(\text{RO})_2\text{HPO}$ ) are determined by the high redox potential due to the

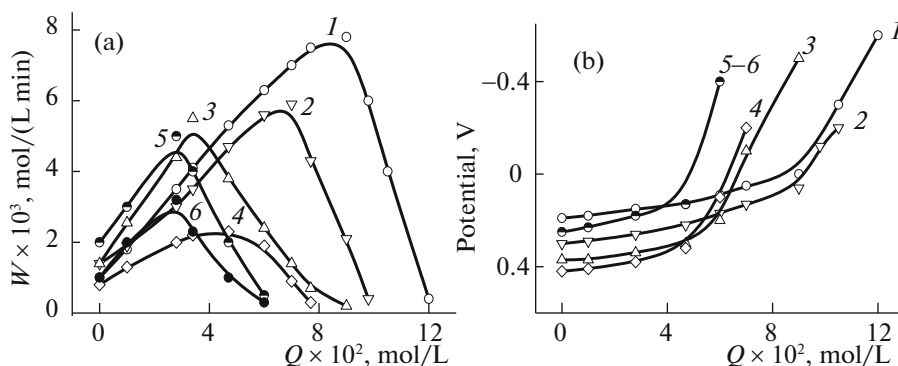
conversion of the quinone structure into the energetically more favorable semiquinone or aromatic system and the presence of two electrophilic (carbonyl and unsaturated carbon atoms) and nucleophilic (carbonyl oxygen atom) centers. Depending on the basicity, phosphines react with either the carbonyl atom or the unsaturated carbon atom of quinone, giving the P–C coupling products. Unlike secondary and tertiary phosphines, primary phosphines do not attach to benzoquinones, but reduce them to hydroquinone, while transforming into diphosphines. The behavior of  $\text{PH}_3$  in reactions with quinones, unlike that of other P(III) compounds with a P–H group, was not studied [19].

Phosphine is not oxidized by benzoquinone in alkaline, neutral, and acidic media. The alcohol solutions of benzoquinone do not absorb phosphine, and the redox potential of the test solution does not change. In mixed quinone– $\text{CuBr}_2$ – $\text{BuOH}$  systems at 70°C, the phosphine oxidation rate increases, and the amount of absorbed  $\text{PH}_3$  corresponds to the total stoichiometry of phosphine oxidation with copper(II) and *p*-benzoquinone (Fig. 1a). Three cycles were performed with addition of a portion of quinone (curves 1–3). The potential changed from 0.8 to 0.47 V during the experiment (Fig. 1b), and the color of the solution changed from dark brown to light yellow.

The amount of absorbed  $\text{PH}_3$  and the change in the potential during the experiment show that copper(II) is reduced to copper(I) in solution, and quinone is reduced to hydroquinone. After regeneration with air oxygen, the potential and the color of the  $\text{CuBr}_2$ –quinone solution returns to initial, but the reaction rate decreases, and the amount of absorbed phosphine corresponds only to the stoichiometry of the oxidation of  $\text{PH}_3$  with copper. After three sequential cycles, the yield of **2** was 86% (experiment 1, Table 1). The copper(II) carboxylate complexes, namely, butyrates, stearates, and acetates  $\text{Cu}(\text{C}_3\text{H}_7\text{CO}_2)_2$ ,  $\text{Cu}(\text{C}_{17}\text{H}_{35}\text{CO}_2)_2$ , and  $\text{Cu}(\text{CH}_3\text{CO}_2)_2$  are well soluble in alcohol. The use of carboxylates as catalysts increases the reaction rate and the amount of absorbed phosphine. The potential of the solution changes from 0.4 to –0.05 V during the experiment, and the color of the solution changes from blue–green to dark brown. The amount of absorbed  $\text{PH}_3$  and the change in the potential during the test indicate that copper(II) is reduced to copper(0) in solution, and quinone is reduced to hydroquinone (Figs. 2a and 2b). Regeneration of the waste solutions in the system involving copper carboxylates restores their activity without further quinone additions. The rate and amount of absorbed  $\text{PH}_3$  decrease, but are higher than in the individual copper solutions. In the stearate solutions, compound **2** formed after six cycles with a yield of 89% (experiments 2 and 3, Figs. 2a and 2b). After two cycles, the yield was 40% for **1** and 33% for **2** in the presence of copper butyrate and 52 and 32%, respectively, in the acetate system (experiments 4–7).



**Fig. 1.** (a) Conversion and (b) potentiometric curves of  $\text{PH}_3$  absorption with an alcohol solution of quinone in the presence of  $\text{CuBr}_2$  at  $70^\circ\text{C}$ ;  $[\text{CuBr}_2] = 0.22$ ,  $[\text{C}_6\text{H}_4\text{O}_2] = 0.45$ ,  $[\text{BuOH}] = 5.45$ , and  $[\text{C}_4\text{H}_8\text{O}_2] = 5.85$  M,  $[\text{PH}_3] = 1.2$  vol %.



**Fig. 2.** (a) Conversion and (b) potentiometric curves of  $\text{PH}_3$  absorption with an alcohol solution of quinone in the presence of  $\text{CuSt}_2$  at  $70^\circ\text{C}$ ;  $[\text{CuSt}_2] = 0.15$ ,  $[\text{C}_6\text{H}_4\text{O}_2] = 0.46$ ,  $[\text{BuOH}] = 5.45$ , and  $[\text{C}_4\text{H}_8\text{O}_2] = 5.85$  M,  $[\text{PH}_3] = 1.5$  vol %.

The  $\text{CuCl}_2$ – $\text{NaOAc}$ –quinone system exhibits the properties characteristic of acetate solutions; after four cycles, the yield of **2** was 89% (experiment 8). A distinction of the reaction of  $\text{PH}_3$  with  $\text{Cu(II)}$  complexes is its autocatalytic character [20].

During the experiments in the absence of quinone, the maximum on the conversion curves is reached at a concentration of  $\text{Cu(I)}$  approximately equal to that of  $\text{Cu(II)}$ ; in the presence of quinone, the maximum shifts toward the end of the experiment, which indicates that  $\text{Cu(I)}$  was oxidized with quinone. The decrease in the  $\text{PH}_3$  absorption rate after the maximum is due to the decrease in the quinone concentration. The reaction rate increased with the alcohol and phosphine concentration and temperature.

According to our experimental data, the kinetic regularities of the oxidative alkoxylation of phosphine in quinone and copper(II) solutions are described by the equation

$$W = k[\text{PH}_3]_2[\text{Cu(II)}]_2[\text{C}_6\text{H}_4\text{O}_2]_2, \quad (3)$$

where  $k$  is the reaction rate constant,  $\text{L}^2/(\text{mol}^2 \text{min})$ ;  $[\text{PH}_3]$ ,  $[\text{Cu}]$ , and  $[\text{C}_6\text{H}_4\text{O}_2]$  are the concentrations of

phosphine, quinone, and copper, respectively,  $M$ . The activation and thermodynamic parameters of the reaction were calculated from the temperature dependence:  $k = 4.5 \times 10^2 \text{ L}^2/(\text{mol}^2 \text{min})$ ,  $E^\ddagger = 62.0 \text{ kJ/mol}$ , and  $\Delta S^\ddagger = -28.4 \text{ J}/(\text{mol K})$  at  $70^\circ\text{C}$ .

In the absence of copper salts, the alcohol solutions of quinoid redoxites do not absorb  $\text{PH}_3$  at a noticeable rate, and the potential does not change for a long time. The following polymers were used as redox polymers based on AN-31 for experiments: P1 containing 1,4-benzoquinone; P2 2,5-dihydroxy- and 3,6-dichlorobenzoquinone; P3 tetrachloro-1,4-benzoquinone; P4  $\alpha$ -naphthoquinone; and redoxites based on allylamine: P5 allylamine-1,4-benzoquinone; and P6 bis-allylamine-1,4-benzoquinone. In the presence of copper salts, the phosphine oxidation rate and the amount of absorbed  $\text{PH}_3$  increase. During the experiment, the potential of the mixed  $\text{Cu(II)}$ –polymer solution shifts from 0.55 to 0.05 V to the cathode region. The  $\text{PH}_3$  oxidation rate and the amount of absorbed phosphine depend on the nature and concentration of copper and polymer (Figs. 3a and 3b). The alcohol solutions of the polymers containing

**Table 1.** Oxidative alkoxylation of  $\text{PH}_3$  in alcohol solutions of quinone and quinoid monomers, polymers, and copper(II) salts

Experiment no.	Composition of solution, M		$\text{PH}_3$ , vol %	$T$ , °C	Cycles	$Q_{\text{PH}_3} \times 10^2$ , M	Yield, %	
	quinone	$\text{CuX}_2$					1	2
$\text{CuBr}_2$								
1	0.45	0.22	1.2	70	1	8.6		27
	0.45				2	6.0		60
	0.45				3	4.3		86
$\text{CuSt}_2$								
2	0.45	0.15	1.4	75	6	53.5		89
3	0.9	0.15	1.6	75	3	35.6		66
$\text{CuBut}_2$								
4	0.45	0.23	1.4	75	2	26.4	40	33
$\text{Cu(OAc)}_2$								
5	0.18	0.01	0.5	60	1	3.0	22	3
6	0.18	0.1	0.5	70	2	6.0	52	32
7	0.45	0.16	0.6	60	2	21.0		65
$\text{CuCl}_2$								
8*	0.45	0.15	1.6	75	4	41.2		89
9	$\text{P}_2$	0.03	1.6	50	1	1.0		50
$\text{Cu(OAc)}_2$								
10	$\text{P}_2$	0.1	0.6	60	2	6.0	52	33
11	$\text{P}'_2$	0.025	1.4	60	2	5.0	60	40
12	$\text{P}_4$	0.025	1.4	60	2	4.0	33	33
13	$\text{P}_5$	0.1	0.6	60	2	0.8	69	7
14	$\text{P}_6$	0.1	0.5	60	2	3.5	17	9
15	$\text{P}'_1$	0.05	1.4	60	2	5.0	20	10

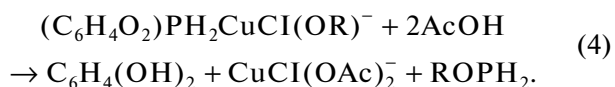
\*  $\text{NaOAc}$  0.3 M; polymers 0.2 g;  $\text{BuOH}$  5.5 M (experiments 1–8) and 10.9 M (experiments 9–15);  $\text{P}'_2$  is the monomer of the  $\text{P}_2$  polymer;  $\text{P}'_1$  is the monomer of the  $\text{P}_1$  polymer;  $\text{But} = \text{C}_3\text{H}_7\text{CO}_2^-$ ;  $\text{St} = \text{C}_{17}\text{H}_{35}\text{CO}_2^-$ ;  $\text{Ac} = \text{CH}_3\text{CO}_2^-$ ; **1** is dibutyl phosphite  $(\text{BuO})_2\text{HPO}$ , and **2** is tributyl phosphate  $(\text{BuO})_3\text{PO}$ .

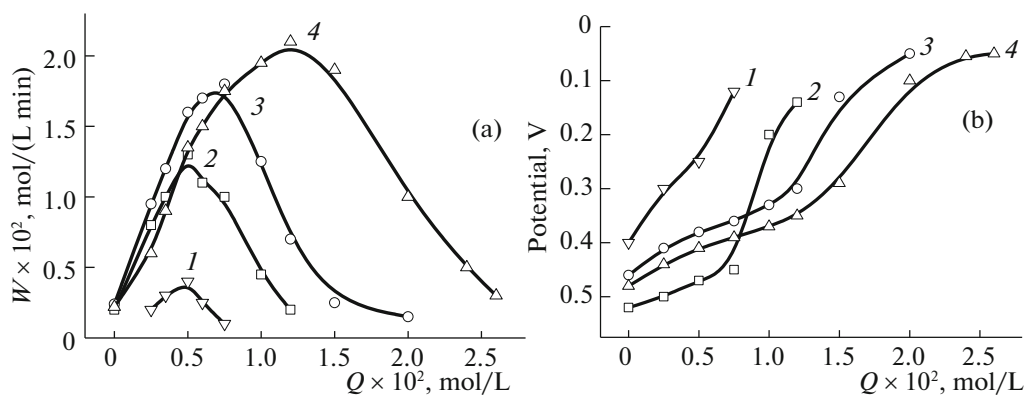
$\text{Cu(OAc)}_2$  absorb a twice larger amount of  $\text{PH}_3$  than in the presence of  $\text{CuCl}_2$  and are faster regenerated with air oxygen. After regeneration, the potential of the system acquires the initial value, the original color of the solution is recovered, and the activity of the solution is restored, which allows two or three cycles (experiments 9–15). In the alcohol solutions of  $\text{CuCl}_2$  and  $\text{P}_2$  polymer, phosphine is oxidized to **2** with a 50% yield; in the presence of  $\text{Cu(OAc)}_2$ , **1** (52%) and **2** (33%) are formed.

The kinetic regularities of the oxidation of  $\text{PH}_3$  in the alcohol solutions of copper and quinoid redoxites agree well with the regularities obtained for the alcohol solutions of quinone and copper salts.

The set of potentiometric, kinetic, and analytical data and the review of the literature on the reactions involving the P(III), quinone, and copper derivatives [17–22] allow us to suggest the following main stages of the oxidative alkoxylation of phosphine (**1**) in the presence of a PFCS containing quinone and copper

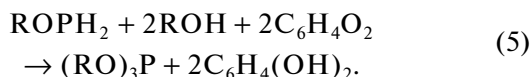
salts. The catalytic cycle starts with the formation of an intermediate copper(I) phosphide  $\text{CuX}_2\text{PH}_2^{2-}$ . The copper(I) ions activate  $\text{PH}_3$ , which forms copper(II) alkoxyphosphide with Cu(II) alkoxy ions. According to our quantum-chemical calculations [23], the negative charge of the coordinated phosphide ion is slightly lower than that of the free  $\text{P}^{3-}$  ion, but much higher than that of the phosphorus atom of  $\text{PH}_3$ . Hence it follows that  $\text{CuX(OR)PH}_2^-$  is a strong nucleophile and is attached to the unsaturated carbon atom of  $\text{C}_6\text{H}_4\text{O}_2$  to form a  $\pi$ -complex similar to the intermediate appearing in the electrophilic aromatic substitution [19]. The resulting phosphide alkoxide complex containing quinone and Cu(II) undergoes an intraspheric redox decomposition, forming phosphinite  $\text{ROPH}_2$  and hydroquinone:





**Fig. 3.** (a) Conversion and (b) potentiometric curves of  $\text{PH}_3$  absorption with an alcohol solution of quinoid polymers in the presence of  $\text{Cu}(\text{OAc})_2$  at  $60^\circ\text{C}$ ;  $[\text{BuOH}] = 10.9 \text{ M}$ ,  $[\text{PH}_3] = 1.2 \text{ vol } \%$ ; (1)  $\text{P}_5$ ,  $\text{Cu}(\text{OAc})_2$  0.1 M; (2)  $\text{P}_4$ , 0.2 g,  $\text{Cu}(\text{OAc})_2$  0.025 M; (3)  $\text{P}_2$ , 0.15 g,  $\text{Cu}(\text{OAc})_2$  0.1 M; and (4)  $\text{P}'_2$ ,  $\text{Cu}(\text{OAc})_2$  0.025 M.

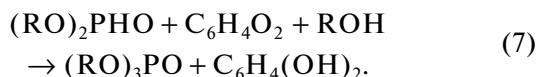
The  $\text{Cu}(\text{II})$  complexes catalyze the fast oxidation of phosphinite with quinone into trialkylphosphite  $(\text{RO})_3\text{P}$ :



Trialkylphosphites are dealkylated to dialkylphosphite  $(\text{RO})_2\text{PHO}$  in an acid medium:



which is further oxidized with quinone to trialkylphosphate  $(\text{RO})_3\text{PO}$ :



The  $\text{Cu}(\text{II})$  complexes also catalyze the oxidation of hydroquinone with oxygen to quinone, which facilitates several cycles of oxidative alkoxylation of phosphine. The use of oxygen for the oxidation of  $\text{PH}_3$  is extremely promising. However, despite its high redox potential, the oxidation of  $\text{PH}_3$  is very slow.

The second and third components of PFCS studied in different oxidations are often oxidizing agents, but in the presence of other additional reagents ( $\text{O}_2$ ,  $\text{H}_2\text{O}_2$ ), they become process catalysts that perform specific chemical and kinetic functions [17]. This suggests high potential of PFCS as a tool in industrial and synthetic chemistry.

Thus, a new catalytic system  $\text{C}_6\text{H}_4\text{O}_2\text{—Cu}(\text{I, II})$  was found for the oxidation of  $\text{PH}_3$  with oxygen to valuable phosphoric acid ethers in alcohol. Due to the distribution of the redox functions between the components of the mixed system, a synergistic effect is observed, and the reaction proceeds by a route that is thermodynamically more favorable. The oxidative alkoxylation of  $\text{PH}_3$  occurring in the coordination sphere of copper(II) complexes and quinone studied for the first time make an important contribution to the molecular organic chemistry of phosphine.

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