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Reactions of Elemental Phosphorus and Phosphine with Electrophiles in Superbasic Systems: XV.[1] Phosphorylation of Allyl Halides with Elemental Phosphorus

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Abstract—Elemental phosphorus (red or white) reacts with allyl chloride and allyl bromide in a two-phase system aqueous KOH–organic solvent to form tertiary symmetrical and mixed phosphine oxides among which tris(prop-2-enyl)-, bis(prop-2-enyl)[(*E*)-prop-1-enyl]-, bis(prop-2-enyl)[(*Z*)-prop-1-enyl]-, (prop-2-enyl)[(*E*)-prop-1-enyl][(*Z*)-prop-1-enyl]-, bis[(*E*)-prop-1-enyl](prop-2-enyl)-, bis[(*Z*)-prop-1-enyl](prop-2-enyl)-, tris-[(*E*)-prop-1-enyl]-, and bis[(*E*)-prop-1-enyl][(*Z*)-prop-1-enyl]phosphine oxides were identified. The conditions (room temperature, 60% aqueous KOH–dioxane) allowing preparation from white phosphorus and allyl bromide of tris(prop-2-enyl)- and bis(prop-2-enyl)[(*E*)-prop-1-enyl]phosphine oxides as major products in the total yield of up to 96% were found.

Phosphorylation of electrophiles with elemental phosphorus or phosphine in the presence of strong bases is one of the most convenient methods of formation of C–P bond and preparation of phosphines and phosphine oxides [2–5] as key compounds in organophosphorus synthesis of promising ligands for the design of catalysts of new generation [6–9], extractants of transuranium elements [10], and fire retardants [11–14]. Organyl halides [15–18], electrophilic alkenes [19–27], acetylenes [28–30], oxiranes [31–34], etc., were successfully used as electrophiles.

It was briefly reported previously [15, 17] that red phosphorus reacts with allyl halides at elevated temperature under the conditions of phase-transfer catalysis to form a mixture of propenylphosphine oxides in low yield. Tris[(*E*)-prop-1-enyl]- and bis-[(*E*)-prop-1-enyl](prop-2-enyl)phosphine oxides were isolated pure, but the latter compound was erroneously identified as tris(prop-2-enyl)phosphine oxide.

In this work we thoroughly analyzed the reaction of elemental phosphorus with allyl halides with the aim of extending its synthetic potential and preparing unsaturated phosphine oxides, which are highly reactive intermediates, monomers, and comonomers [18] and can be used, for example, for preparing of new types of noncombustible polymers.

The main reaction products in phosphorylation of allyl chloride **Ia** or bromide **Ib** with red or white phosphorus in the system aqueous potassium hydroxide–dioxane are symmetrical or mixed phosphine oxides with prop-1-enyl and prop-2-enyl fragments. The yield and ratio of these compounds depend on the reactant structures and reaction conditions (reactant concentrations, temperature, heating time, etc.) (Table 1). Among the phosphine oxides formed, we identified tris(prop-2-enyl)phosphine oxide **II**, bis-(prop-2-enyl)[(*E*)-prop-1-enyl]phosphine oxide **III**, bis(prop-2-enyl)[(*Z*)-prop-1-enyl]phosphine oxide **IV**, (prop-2-enyl)[(*E*)-prop-1-enyl][(*Z*)-prop-1-enyl]phosphine oxide **V**, bis[(*E*)-prop-1-enyl](prop-2-enyl)-phosphine oxide **VI**, bis[(*Z*)-prop-1-enyl](prop-2-enyl)-phosphine oxide **VII**, tris[(*E*)-prop-1-enyl]phosphine oxide **VIII**, and bis[(*E*)-prop-1-enyl][(*Z*)-prop-1-enyl]-phosphine oxide **IX**.

Allyl bromide reacts with red phosphorus in the system comprising 60% aqueous potassium hydroxide and dioxane at room temperature to form mainly the expected kinetically controlled product, tris(prop-2-

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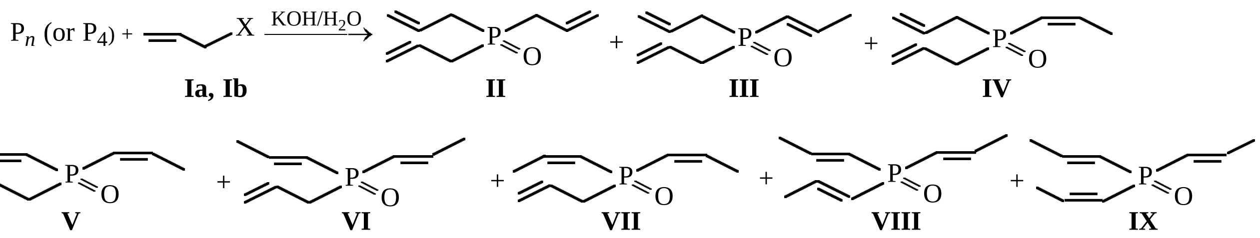
[1] For communication XIV, see [1].



X = Cl, Br.

enyl)phosphine oxide **II**, in a yield not higher than 3% (Table 1, run no. 1).

When performed at 70–75°C (molar ratio of allyl bromide and phosphorus 1.75 : 1) in the 50% KOH–dioxane system, the reaction is more efficient; its selectivity and pathways change also. The total yield of phosphine oxides **III**–**IX** is 23%. Under these conditions, the major reaction product is tris[(*E*)-prop-1-enyl]phosphine oxide **VIII**, apparently formed by thermal prototropic isomerization of tris(prop-2-enyl)-phosphine oxide (Table 1, run no. 2). Allyl chloride under the similar conditions appeared to be less active. The total yield of phosphine oxides **II**–**IX** was as low as 2% (Table 1, run nos. 3 and 2). When phosphorylation of allyl chloride with an equimolar amount of red phosphorus is performed at 60°C in the 60% KOH–dioxane system in the presence of a phase-transfer catalyst (benzyltriethylammonium chloride), the yield of phosphine oxides **III**–**VI**, **VIII**, and **IX** increases to 17%. Under these conditions, the main product is tris[(*E*)-prop-1-enyl]phosphine oxide **VIII** (Table 1, run no. 4). Further decrease in the reaction temperature is not appropriate, because below 45°C the yield of the final products decreases below 9% (Table 1, run no. 5). At the same time, under these conditions white phosphorus reacts with allyl chloride more efficiently than red phosphorus, which leads to an increase in the total yield of the phosphine oxides to 21% with a simultaneous increase in the content of the kinetically controlled product, tris(prop-2-enyl)-phosphine oxide **II**, to 24% (Table 1, run no. 6).

An unexpected result was obtained in phosphoryla-

Table 1. Reaction of elemental phosphorus with allyl halides **Ia** and **Ib**

Exp. no.	Elemental phosphorus, mmol	Allyl halide, mmol	KOH, mmol[a]	Dioxane, ml	Temperature, °C	Time, h	Percentage of indicated tris(propenyl)-phosphine oxide isomer, %[b]								Yield, %[c]
							II	**III**	**IV**	**V**	**VI**	**VII**	**VIII**	**IX**	
1[d]	P_n, 16	**Ib**, 13	85	10	20–22	4	65	21	14	[e]	[e]	[e]	[e]	[e]	3
2	P_n, 100	**Ib**, 175	45	40	70–75	5	[e]	4	2	5	27	11	47	4	23
3	P_n, 100	**Ia**, 175	45	40	70–75	5	19	7	6	11	17	6	26	8	2
4[f]	P_n, 64	**Ia**, 64	45	40	60	3.5	[e]	11	6	9	31	[e]	42	1	17
5[f]	P_n, 100	**Ia**, 100	54	50	45	3.5	14	24	[e]	[e]	36	[e]	23	3	9
6[f]	P_4, 97	**Ia**, 97	54	50	45	3.5	24	32	5	11	22	[e]	4	2	21
7	P_4, 16	**Ia**, 13	85	10	20–22	4	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]	13[g]
8	P_4, 16	**Ib**, 13	85	10	20–22	5	62	31	17	[e]	[e]	[e]	[e]	[e]	96
9[f]	P_4, 67	**Ib**, 67	45	40	45	3.5	14	20	21	11	24	[e]	7	3	40
10	P_4, 16	**Ia**, 48	23	[h]	20–22	3	49	[e]	30	12	9	[e]	[e]	[e]	12

[a] In run nos. 1, 4–10, 60% aqueous KOH, and in run nos. 2 and 3, 50% aqueous KOH was used. [b] Relative content of isomers **II**–**IX** in the reaction mixture was determined by ^{1}H and ^{31}P NMR spectroscopy (the spectra contained, along with the signals of the indicated compounds, also the signals of unidentified four-coordinate phosphorus derivatives). [c] Total yield of phosphine oxides **II**–**IX** based on the amount of allyl halide taken. [d] Conversion of red phosphorus in this experiment was 18%, and in all the other experiments the conversion of elemental phosphorus was 100%. [e] Does not form. [f] Experiments were carried out in the presence of benzyltriethylammonium chloride (1 wt % relative to allyl halide). [g] In this reaction (prop-2-enyl)phosphine is formed (identified by ^{1}H and ^{31}P NMR spectroscopy). [h] The reaction was carried out in the system DMSO (10 ml)–H_2O (0.8 ml).

tion of allyl chloride with white phosphorus in the aqueous KOH–dioxane system at room temperature. According to the ^{1}H and ^{31}P NMR spectra, under these conditions (prop-2-enyl)phosphine **X** is formed in 13% yield (Table 1, run no. 7).

Under the similar conditions, phosphorylation of allyl bromide with white phosphorus proceeds much more efficiently, with virtually quantitative formation of phosphine oxides **II**–**IV**. The major product is the kinetically controlled product, phosphine oxide **II** (Table 1, run no. 7). As the reaction temperature is increased to 45°C, the yield of the final product decreases by a factor of more than 2, obviously owing to the competing dehydrobromination of the starting allyl bromide (Table 1, run no. 9).

Our attempt to increase the efficiency of phosphorylation of allyl chloride with white phosphorus at room temperature using the KOH–DMSO system failed. The total yield of phosphine oxides **II** and **IV**–**VI** under these conditions was 12% (Table 1, run no. 10).

Phosphine, separately generated from red phosphorus and KOH in water–dioxane medium, reacts with allyl bromide at room temperature in the 60% aqueous KOH–dioxane system to give a mixture of (prop-2-enyl)phosphine **X**, bis(prop-2-enyl)phosphine **XI**, and tris(prop-2-enyl)phosphine **XII** in the total yield of up to 2%. When performed in the KOH–DMSO system, the reaction gives a mixture of phosphines **XI** and **XII** in a total yield of up to 14%. During isolation from the reaction mixture, these compounds are oxidized to the related phosphine oxides.

Formation of allylphosphines from phosphine and allyl halides proceeds via initial formation of phosphide anions from phosphine under the action of bases. At the same time, the reaction of elemental phosphorus with allyl halides, yielding phosphine oxides **II**–**IX**, involves both phosphide and phosphinite anions generated from elemental phosphorus and aqueous potassium hydroxide. Indeed, the ^{31}P NMR monitoring of phosphorylation of allyl bromide with white phosphorus at room temperature revealed simultaneous appearance of tris(prop-2-enyl)phosphine oxide **II**, bis(prop-2-enyl)phosphine **XI**, and tris(prop-2-enyl)phosphine **XII** in the reaction mixture; the phosphines are probably oxidized to give the corresponding phosphine oxides. The scheme of formation of phosphine oxides from elemental phosphorus and electrophiles, including phosphinite anions, was previously discussed with phosphorylation of organyl halides [2, 35] and of aryl- and hetarylethylenes [36] as example.

The NMR spectra of phosphine oxides **II**–**IX** are given in Table 2.

EXPERIMENTAL

The ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on a Bruker DPX-400 spectrometer in $CDCl_3$ relative to internal HMDS. The ^{31}P NMR spectra of compounds obtained in run nos. 4–8 were taken on a Jeol FX-90Q spectrometer in $CDCl_3$. Phosphine oxides **II**–**IX** were isolated by preparative GLC on a PAKhV-07 chromatograph using a 1000 × 10-mm column packed with Chezasorb (0.25-0.36 mm) containing 15% polyphenylmethylsiloxane, helium carrier gas. Phosphine was obtained in a mixture with hydrogen according to [37]. All the experiments were carried out under argon.

Reaction of allyl bromide with red phosphorus at room temperature. To a mixture of 4.77 g of potassium hydroxide, 3.2 ml of water, and 10 ml of dioxane, 0.5 g of red phosphorus was added. The mixture spontaneously warmed up to 40°C and was cooled to 20°C. After that, 1.54 g of allyl bromide was added dropwise over a period of 1 min. The resulting mixture was stirred for 4 h at room temperature. The dioxane layer was removed and dried over potassium carbonate; the solvent was distilled off at reduced pressure, and the residue was kept in a vacuum to give 0.02 g (3%) of the product containing phosphine oxides **II**, **III**, and **IV** in 65 : 21 : 14 ratio according to the ^{1}H and ^{31}P NMR spectra. Unchanged red phosphorus was filtered off, washed with water to neutral reaction, and dried in a vacuum to constant weight; 0.41 g of phosphorus was recovered (18% conversion).

Reaction of allyl bromide with white phosphorus at room temperature. To a mixture of 4.77 g of potassium hydroxide, 3.2 ml of water, and 10 ml of dioxane, 0.5 g of white phosphorus was added. The mixture spontaneously warmed up to 40°C and was cooled to 20°C. After that, 1.54 g of allyl bromide was added dropwise over a period of 1 min. The resulting mixture was stirred for an additional 5 h at room temperature. The dioxane layer was separated and dried over potassium carbonate; the solvent was removed at reduced pressure. The residue was kept in a vacuum to give 0.69 g (96%) of the product containing phosphine oxides **II**, **III**, and **IV** in 62 : 31 : 17 ratio according to the ^{1}H and ^{31}P NMR spectra.

Reaction of allyl chloride with white phosphorus at room temperature. To a mixture of 4.77 g of potassium hydroxide, 3.2 g of water, and 10 ml of dioxane, 0.5 g of white phosphorus was added. The

Table 2. ^{1}H, ^{13}C, and ^{31}P NMR spectra of phosphine oxides **II**–**IX**

Comp. no	R^1	R^2	R^3	^{1}H NMR spectrum (CDCl$_3$), δ, ppm (*J*, Hz)	^{31}P NMR spectrum, (CDCl$_3$), δ$_P$, ppm
II[a]	$C^1H_2{=}C^2HC^3H_2$		–	2.55 d.d (6H, C^{3}H, $^3J_{HH}$ 7.6 , $^2J_{PH}$ 14.8), 5.15 d.d (3H, C^{1}H, $^2J_{HH}$ 1.0, $^3J_{HH}$ 15.2), 5.19 d.d (3H, C^{1}H, $^3J_{HH}$ 10.5), 5.76 sextet (3H, C^{2}H)	41.7
III[a]	$C^1H_2{=}C^2HC^3H_2$	*E*-$C^4H_3C^5H{=}C^6H$	–	1.88–1.92 d (3H, C^{4}H, $^3J_{HH}$ 6.4), 2.59 d.d (4H, C^{3}H, $^3J_{HH}$ 7.6, $^2J_{PH}$ 14.8), 5.17 m (2H, C^{1}H, $^2J_{HH}$ 1.0, $^3J_{HH}$ 15.2), 5.25 m (2H, C^{1}H, $^3J_{HH}$ 10.4), 5.74–5.82 m (3H, C^{2,6}H), 6.67–6.77 t.q (1H, C^{5}H, $^3J_{HMe}$ 6.8, $^3J_{HH}$ 17.2, $^3J_{PH}$ 23.6)	31.57
IV	$C^1H_2{=}C^2HC^3H_2$	–	*Z*-$C^4H_3C^5H{=}C^6H$	2.10–2.18 m (3H, C^{4}H), 2.55–2.67 d.d (4H, C^{3}H, $^3J_{HH}$ 7.6, $^2J_{PH}$ 14.8), 5.10–5.23 m (4H, C^{1}H), 5.46–5.67 d.q (1H, C6H, $^3J_{HH}$ 12.90, $^2J_{PH}$ 24.4), 5.75–5.90 m (2H, C^{2}H), 6.50–6.75 m (1H, C^{5}H)	33.04
V	$C^1H_2{=}C^2HC^3H_2$	*E*-$C^4H_3C^5H{=}C^6H$	*Z*-$C^4H_3C^5H{=}C^6H$	1.92 m (3H, C^{4}H, $^3J_{HH}$ 6.4), 2.15 m (3H, C^{7}H), 2.65 d.d (2H, C^{3}H, $^2J_{HH}$ 5.0, $^2J_{PH}$ 10.0), 5.17–5.24 m (2H, C^{1}H), 5.55 d.d (1H, C^{9}H, $^3J_{HH}$ 12.9, $^2J_{PH}$ 24.4), 5.62 d.d (1H, C^{6}H, $^3J_{HH}$ 12.9, $^2J_{PH}$ 24.4), 5.78–5.87 m (1H, C^{2}H), 6.62 m (2H, C^{5,8}H)	30.69
VI[a]	$C^1H_2{=}C^2HC^3H_2$	*E*-$C^4H_3C^5H{=}C^6H$	–	1.92 m (6H, C^{4}H, $^3J_{HH}$ 6.8), 2.65 d.d (2H, C^{3}H, $^3J_{HH}$ 7.4, $^2J_{PH}$ 14.8), 5.14–5.21 m (2H, C^{1}H), 5.74–5.87 m (3H, C^{2,6}H[b], 6.54–6.68 m (2H, C^{5}H)	22.80
VII	$C^1H_2{=}C^2HC^3H_2$	–	*Z*-$C^4H_3C^5H{=}C^6H$	2.10 m (6H, C^{4}H), 2.56 d.d (2H, C3H, $^3J_{HH}$ 7.6, $^2J_{PH}$ 14.8), 5.17– 5.24 m (2H, C1H), 5.62 d.d (2H, C^{6}H, $^3J_{HH}$ 12.9, $^2J_{PH}$ 24.4), 5.78–5.87 m (1H, C^{2}H), 6.56 m (2H, C^{5}H)	22.60
VIII[a]	–	*E*-$C^4H_3C^5H{=}C^6H$	–	1.90 d (9H, C^{1}H, $^3J_{HH}$ 6.8), 5.84 d.d (3H, C^{3}H, $^3J_{HH}$ 17.6, $^2J_{PH}$ 23.6), 6.49–6.66 m (3H, C^{2}H, $^2J_{PH}$ 17.6)	15.53
IX	–	*E*-$C^4H_3C^5H{=}C^6H$	*Z*-$C^4H_3C^5H{=}C^6H$	1.92 m (6H, C^{1}H, $^3J_{HH}$ 6.8), 2.10 m (3H, C^{4}H), 5.65 d.d (1H, C^{6}H, $^3J_{HH}$ 12.9, $^2J_{PH}$ 24.4), 5.74–5.87 m (2H, C^{3}H), 6.45–6.68 m (3H, C^{2,5}H)[b]	14.11

[a] ^{13}C NMR spectrum, δ$_C$, ppm (J_{PC}, Hz) in CDCl$_3$: for **II**, 33.10 d (C^3, $^1J_{PC}$ 62.7); 120.62 d (C^1, $^3J_{PC}$ 11.2); 127.35 d (C^2, $^2J_{PC}$ 7.2). For **III**, 20.20 d (C^4, $^3J_{PC}$ 17.3); 35.00 d (C^3, $^1J_{PC}$ 66.7); 120.34 d (C^1, $^3J_{PC}$ 11.5); 121.62 d (C^6, $^1J_{PC}$ 94.9); 127.53 d (C^2, $^2J_{PC}$ 8.7); 148.05 s (C^5, $^2J_{PC}$ 0). For **VI**, 20.10 d (C^4, $^3J_{PC}$ 17.6); 37.90 d (C^3, $^1J_{PC}$ 70.1); 120.00 d (C^1, $^3J_{PC}$ 11.6); 123.76 d (C^6, $^1J_{PC}$ 99.0); 127.78 d (C^2, $^2J_{PC}$ 8.3); 148.11 s (C^5, $^2J_{PC}$ 0). For **VIII**: 20.34 d (C^1, $^3J_{PC}$ 18.3); 124.24 d (C^3, $^1J_{PC}$ 104.8); 145.72 s (C^2, $^2J_{PC}$ 0). [b] Accurate measurement of the coupling constants is impossible because of the total overlap of the two complex multiplets.

mixture spontaneously warmed up to 40°C and was cooled to 20°C. After that, 1.0 g of allyl chloride was added, and the mixture was stirred for an additional 4 h at room temperature. The primary phosphine and unchanged allyl chloride were distilled at reduced pressure into a trap cooled to –70°C. The distillate (0.45 g) contained, according to the ^{1}H and ^{31}P NMR spectra, 0.13 g (13% yield) of (prop-2-enyl)phosphine. ^{13}P NMR spectrum (CDCl$_3$), δ$_P$, ppm: –133.47 ($^1J_{PH}$ 193 Hz; published data [38]: $^1J_{PH}$ 193 Hz).

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