# SYNTHESIS OF HETEROCYCLIC COMPOUNDS CONTAINING

### PHOSPHORUS IN THE RING

COMMUNICATION 2. STUDY OF THE REACTION OF DIENIC HYDROCARBONS WITH ARYL- AND ALKYL-PHOSPHONOUS DICHLORIDES AND WITH ARYL AND ALKYL PHOSPHORODICHLORIDES

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As indicated in the patent literature [1], dienic hydrocarbons react by a diene-condensation process with aryland alkyl-phosphonous dichlorides with formation of 1-aryl-3-phospholene 1,1-dichlorides (I)

$$CH_2 = CH - CH = CH_2 + C_6H_5PCI_2 \longrightarrow \begin{bmatrix} CH_2 - CH = CH - CH_2 \\ I \\ C_6H_5 - P \\ \hline CI_2 \\ (I) \end{bmatrix}$$

The dichlorides (I) have not been isolated in the pure state. Treatment of the reaction mixture containing the dichlorides (I) with water, alcohol, or acetic acid leads to substituted 1-aryl-3-phospholene 1-oxides (II)

$$\begin{bmatrix} CH_2 - CH = CH - CH_2 \\ C_6H_5 - P - CI_2 \end{bmatrix} + H_2O \xrightarrow{C_6H_5 - P - CI_2} C_6H_5 - P \xrightarrow{O} (II) + 2HCI$$

As we intended to study the addition of various reagents to diene systems, we considered it of interest to investigate the reaction of dienic hydrocarbons with alkyl- and aryl-phosphonous dichlorides.

We first repeated the reaction, described in the patent [1], between butadiene and phenylphosphonous dichloride, and we obtained a compound identical to the product described. The product was distilled off at 2-4 mm, and not at 0.2-0.4 mm as stated in the patent. Under these conditions, the distillation went without decomposition, and the yield of product was not reduced.

We then carried out analogous reactions of butadiene and of piperylene with ethyl-, p-tolyl-, p-chlorophenyl-, and p-bromophenyl-phosphonous dichlorides. We obtained crystalline products or oils, which were the corresponding 1-ethyl-, 1-p-tolyl-, and 1-p-halophenyl-3-phospholene 1-oxides (III)

$$CH_2-CH=CH-CHY$$

$$XC_{e}H_4-P = 0 \quad (III)$$
where X = -CH\_3; CI; Br; Y = -H; -CH\_3.

The oxides obtained by the reaction of butadiene with the above phosphonous dichlorides are white crystalline substances melting in the range 60-100°.

The products obtained by the reaction of piperylene with the same phosphonous dichlorides were usually colorless liquids. The yields of the products were 22-30% on the phosphonous dichloride that reacted. Apart from these oxides and phosphonous dichlorides recovered unchanged, no other products were obtained in the experiments described.

The reaction of any and alkyl phosphorodichloridites with dienes was studied by us for the first time for the case of the reaction of phenyl and ethyl phosphorodichloridites with butadiene, with piperylene with isoprene, and

with 2,3-dimethyl-1,3-butadiene. The reaction was carried out at room temperature in presence of copper stearate. It was shown that phenyl phosphorodichloridite reacts with these dienes in equimolecular proportions with formation of 1-phenoxy-3-phospholene 1,1-dichlorides (IV), which were not isolated from the reaction mixture, but were immediately converted into the corresponding oxides (V)

$$CH_{2}=CH-CH=CH_{2} + C_{6}H_{5}OPCI_{2} \longrightarrow \begin{bmatrix} CH_{2}-CH=CH=CH_{2}\\ C_{6}H_{5}OPCI_{2}\\ (IV) \end{bmatrix} + H_{2}O + H_{2}$$

The oxides were either white crystalline substance melting not above 62° in the case of butadiene and isoprene, or colorless high-boiling liquids, stable to storage, in the case of piperylene and 2,3-dimethyl-1,3-butadiene. As well as products of reaction with the diene and about 50% of phenyl phosphorodichloridite recovered unchanged, two other products were isolated, and these were found to be diphenyl phosphorochloridite (21%) and triphenyl phosphite; the latter was characterized by its constants and infrared spectrum.

Hence, in the course of the reaction of phenyl phosphorodichloridite with the diene, disproportionation occurs in accordance with the scheme

$$C_6H_5OPCl_2 \rightarrow (C_6H_5O)_2PCl + (C_6H_5O)_3P + PCl_3$$
.

Some disproportionation was observed by Kabachnik and Rossiiskaya [2] in the case of bis-2-chloroethyl phosphorochloridite. By special experiments we showed that phenyl phosphorodichloridite undergoes this disproportionation on prolonged standing at room temperature both in presence of copper stearate and in its absence.

It might be expected that the diphenyl phosphorochloridite formed would also react with the diene. However, experiment showed that diphenyl phosphorochloridite does not react with butadiene, but undergoes disproportionation to phenyl phosphorodichloridite and triphenyl phosphite. The dichloridite reacts with butadiene with formation of 1-phenoxy-3-phospholene 1,1-dichloride. On treatment with water the dichloride gives the corresponding oxide. On treatment with bromine, the oxide gives the crystalline dibromo addition product, m.p. 121-122° (VI)

$$C_{6}H_{5}OP \bigcirc O$$
 (VI)

Phenyl phosphorodibromidite reacts more readily with butadiene than the dichloridite does, and the yield of 1-phenoxy-3-phospholene 1-oxide attains 70-75%.

As we showed in the case of ethyl phosphorodichloridite and 2,3-dimethyl-1,3-butadiene, alkyl phosphorodichloridites react with dienes, but the reaction takes a somewhat different course

$$C_{2}H_{5}OPCI_{2} + CH_{2} = C - C = CH_{2} \longrightarrow \begin{bmatrix} H_{3}C & CH_{3} \\ CH_{2} - C = C - CH_{2} \\ H_{3}C & CH_{3} \end{bmatrix} \xrightarrow{H_{3}C & CH_{3} \\ - CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{3} + CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{3} + CH_{2} - C = C - CH_{2} \\ - C_{2}H_{5}CI + CH_{3} + CH_{3} + CH_{3} + CH_{3} + CH_{3} + CH_{3} \\ - CH_{2} - C = C - CH_{2} + CH_{3} + CH_{3$$

It is evident that the initially formed dichloride (VII) loses ethyl chloride on distillation, and gives the acid chloride (VIII), which is a crystalline product, m.p. 79-82°, which fumes in air. On treatment with water the acid chloride gives the acid (IX), m.p. 121-123°.

## EXPERIMENTAL

<u>Reaction of Phenylphosphonous Dichloride with Butadiene.</u> A mixture of 135 g of phenylphosphonous dichloride [3], 0.6 g of copper stearate [4], and 70 ml of butadiene was shaken in a firmly stoppered bottle at room temperature for 20 days. The bottle was cooled strongly and then opened. The reaction mixture consisted of two layers. The liquid part was poured off and, after the removal of butadiene, vacuum-distilled. We obtained 55 g (40.7%) of unchanged phenylphosphonous dichloride; b.p. 90-91° (10 mm);  $n^{20}D 1.5960$ ;  $d^{20}_{4}$  1.3162. The dark-colored thick mass was washed free from traces of phenylphosphonous dichloride with petroleum ether and treated with crushed ice. The resulting solution was neutralized with 30% NaOH solution until weakly alkaline. The solution was then saturated with common salt and extracted with chloroform. After removal of chloroform the product was vacuumdistilled. We isolated a fraction of b.p. 182-183° (2.5 mm); immediately after distillation this turned into a compact white crystalline mass, m.p. 75-77°. Found: P 17.6%.  $C_{10}H_{11}PO$ . Calculated: P 17.42%. The product amounted to 25 g (29% on the phenylphosphonous dichloride that reacted). The product was 1-phenyl-3-phospholene 1oxide.

Reaction of Phenylphosphonous Dichloride with Piperylene. A mixture of 47.5 g of phenylphosphonous dichloride, 27.4 g of piperylene (cis- and trans-forms), and 0.65 g of copper stearate was left at room temperature in a closely stoppered flask for 13 weeks. The thick dark-colored mass formed was carefully separated from the liquid part of the reaction mixture and treated in the same way as in the preceding experiment. We isolated a product having the following constants: b.p. 158-159° (3.5 mm),  $n^{20}D \ 1.5768$ ;  $d^{20}_4$  1.1429; found MR 55.66; calculated for  $C_{11}H_{13}PO = 4$  MR 55.35. The value taken for the atomic refraction of phosphorus was 5.31 [5-7]. Found: P 15.73%,  $C_{11}H_{13}PO$ . Calculated: P 16.12%. The yield of product was 6.3 g (22.5% on the phenylphosphonous dichloride that reacted). The product was 2-methyl-1-phenyl-3-phospholene 1-oxide.

From the liquid part of the reaction mixture we obtained 21.5 g of unchanged phenylphosphonous dichloride; b.p.  $93-97^{\circ}$  (11 m);  $n^{20}D$  1,5952.

Reaction of p-Tolylphosphonous Dichloride with Butadiene. A mixture of p-tolylphosphonous dichloride [8], 38 ml of butadiene, and 2.2 g of copper stearate was shaken in a thick-walled bottle for 15 days at room temperature. The treatment was exactly the same as in the preceding experiment. We obtained a product with the following constants: b.p. 183-184° (3 mm);  $n^{20}D$  1.5795;  $d^{20}_{4}$  1.1590; found MR 55.10; calculated MR 55.35 for C<sub>11</sub>H<sub>13</sub>PO -Found: P 16.2% C<sub>11</sub>H<sub>13</sub>PO; calculated P 16.12%. On long standing the product solidified (m.p. 60-67°); we obtained 27 g (31% on the phosphonous dichloride). The product was 1-p-tolyl-3-phospholene 1-oxide,

Reaction of p-Tolylphosphonous Dichloride with Piperylene. A mixture of 55 g of p-tolylphosphonous dichloride, 25 g of piperylene (mixture of cis- and trans-forms), and 1 g of copper stearate was placed in a firmly stoppered bottle, which was shaken at room temperature. After 12 days a thick, dark-colored oil had formed; it was separated from the liquid part and treated as usual. We isolated a fraction having: b.p. 171-172° (2.5 mm);  $n^{20}D$  1.5702;  $d^{20}_{4}$  1.1220; found MR 60.60; calculated MR 59.96 for  $C_{12}H_{15}PO = 4$ . Found: P 14.82, 14.93%,  $C_{12}H_{15}PO$ . Calculated: P 15.02%. The yield of product was 9.5 g (27% on the p-tolylphosphonous dichloride that reacted). The product was 2-methyl-1-p-tolyl-3-phospholene 1-oxide.

The clear liquid that had been decanted from the oil was vacuum-distilled and gave a fraction of b.p. 123 to 127° (20 mm); this was unchanged p-tolylphosphonous dichloride.

Reaction of Ethylphosphonous Dichloride with Piperylene. A mixture of 55 g of ethylphosphonous dichloride [9], 34 ml (30 g) of piperylene, and 0.5 g of copper stearate was sealed in a tube, which was left at room temperature. After 35 days the liquid had turned into a thick, dark-colored oil. The oil was treated as in the preceding experiment. After several vacuum distillations we isolated a fraction of b.p. 114-115° (5 mm),  $n^{20}D$  1.5080,  $d^{20}_{4}$  1.0885; found MR 39.43; calculated for  $C_7H_{13}OP \bigsqcup$  MR 40.47. Found: P 21.96%.  $C_7H_{13}PO$ . Calculated: P 21.53%. The yield of the product was 5 g (7.45% on the ethylphosphonous dichloride). The product was 1-ethyl-2-methyl-3-phospholene 1-oxide.

<u>Reaction of p-Chlorophenylphosphonous Dichloride with Piperylene.</u> A mixture of 35.3 g of p-chlorophenylphosphonous dichloride [8], 17 ml (11.25 g) of piperylene, and 0.5 g of copper stearate was left in a sealed tube for 42 days at room temperature. After suitable treatment with water, alkali, and chloroform the reaction products were vacuum-distilled. We obtained a product of b.p. 165-166° (3.5 mm), which immediately crystallized out. The crystalline mass, which had a slightly greenish color, had m.p. 94-96°. Found: P 13.30%,  $C_{11}H_{12}$ OPCl. Calculated: P 13.68% Reaction of p-Bromophenylphosphonous Dichloride with Butadiene. A mixture of 80.5 g of p-bromophenylphosphonous dichloride [8], 40 ml of butadiene, and 0.5 g of copper stearate was left in a firmly stoppered bottle for 9 days at room temperatures. A thick, dark-colored oil containing crystals was formed. This oil was treated as in all the preceding experiments. After three vacuum distillations we isolated a fraction of b.p. 151-152° (3 mm). The product solidified immediately and had m.p. 66-71°. Found: P 12.16; 12.30%.  $C_{10}H_{10}BrOP$ . Calculated: P 12.02%. The yield of product was 10 g (14.2% on the p-bromophenylphosphonous dichloride).

Reaction of Phenyl Phosphorodichloridite with Butadiene. A mixture of 85.4 g of phenyl phosphorodichloridite [10], 36 ml of butadiene, and 0.43 g of copper stearate was shaken in a tightly closed bottle for 22 days at room temperature. The thick dark-colored oil formed was separated from the liquid part and carefully decomposed with crushed ice. The aqueous solution obtained was neutralized, with cooling, with 30% NaOH solution until weakly al-kaline; it was then saturated with sodium chloride and extracted with chloroform. The chloroform was distilled off from a water bath, and the residue was vacuum-distilled. After three successive distillations from a Favorskii flask we obtained a product having: b.p. 163-163.5° (3 mm);  $n^{20}D$  1.5628. The product rapidly solidified; m.p. 60-62°. Found: P 15.31, 15.45%. C<sub>10</sub>H<sub>11</sub>PO<sub>2</sub>. Calculated: P 15.98%. The yield of product was 5.5 g (14% on the phenyl phosphorodichloridite that reacted). The product was 1-phenoxy-3-phospholene 1-oxide.

The liquid part that was separated from the oil was vacuum-distilled and gave three fractions: Fraction I, b.p. 97-100° (12 mm);  $n^{20}D$  1.5606; 48.9 g (57.3%). This was phenyl phosphorodichloridite. Fraction II, b.p. 150 to 153° (4 mm);  $n^{20}D$  1.5735;  $d^{20}_{4}$  1.2482; found MR 66.71 for  $C_{12}H_{10}O_2PCl_{=6}$ ; calculated MR 66.70. Found: P 12.48, 12.68%.  $C_{12}H_{10}O_2PCl$ . Calculated: P 12.28%. The yield of product was 7.7 g (21.4% on the phenyl phosphorodichloridite that reacted). This fraction was diphenyl phosphorochloridite. Fraction III, b.p. 181-182° (2-2.5 mm),  $n^{20}D$ 1.5895,  $d^{20}_{4}$  1.1861; found MR 87.75 for P(OC<sub>6</sub>H<sub>5</sub>)<sub>3</sub>; calculated MR 87.59. Found P 10.78%. P(C<sub>6</sub>H<sub>5</sub>O)<sub>3</sub>. Calculated P 10.00%. The yield of triphenyl phosphite was 8.8 g (24.4% on the phenyl phosphorodichloridite that reacted).

Reaction of Diphenyl Phosphorochloridite with Butadiene. A mixture of 182 g of diphenyl phosphorochloridite, 70 ml of butadiene, and 1 g of copper stearate was shaken for 90 days. There was partial formation of a thick darkcolored oil, which was separated from the liquid part and treated as in the preceding experiments. After several distillations we isolated a product having: b.p. 169-169.5° (4 mm);  $n^{20}D \ 1.5625$ ;  $d^{20}_{4}$  1.2153; found MR 51.84 for  $C_{10}H_{11}O_2P$  a; calculated MR 51.856. Found: P 16.48%,  $C_{10}H_{11}PO_2$ . Calculated: P 15.98%. This was a thick colorless liquid, which solidified after 30 min. The white crystalline mass had m.p. 60-62°, undepressed by admixture of 1-phenoxy-3-phospholene 1-oxide.

The liquid part that had been separated from the resinous mass was vacuum-distilled and gave three fractions: Fraction I, b.p. 93-95° (12 mm);  $n^{20}D$  1.5602; yield 23 g (47.8% on the diphenyl phosphorochloridite that reacted). This was phenyl phosphorodichloridite. Fraction II, b.p. 169-172° (12 mm);  $n^{20}D$  1.5760. Yield 57.5 g (31.6%). This was unchanged diphenyl phosphorochloridite. Fraction III, b.p. 181-182° (2-2.5 mm);  $n^{20}D$  1.5901;  $d^{20}_{4}$  1.1864; found MR 88.18 for  $(C_{6}H_{5}O)_{3}P|_{=9}$ ; calculated MR 87.59. Found: P 10.26, 10.43%.  $(C_{6}H_{5}O)_{3}P$ . Calculated: P 10.00%. The yield of triphenyl phosphite was 17 g (61.5% on the diphenyl phosphorochloridite that reacted).

Addition of Bromine to 1-Phenoxy-3-phospholene 1-Oxide. A solution of 2.9 g of bromine in 15 ml of chloroform was added slowly dropwise to a solution of 3.2 g of the oxide in 25 ml of chloroform. Cooling with snow was applied during the reaction. Chloroform was vacuum-evaporated with cooling. The solid residue was carefully washed with water and dried; m.p. 119-121°. After recrystallization from water we obtained gleaming white scales, m.p. 121-122°. Found: Br 45.56, 45.55%.  $C_{10}H_{11}O_2PBr_2$ . Calculated: Br 45.19%. The product was 3,4-dibromo-1-phenoxyphospholane 1-oxide.

<u>Reaction of Phenyl Phosphorodichloridite with Piperylene.</u> A mixture of 252 g of phenyl phosphorodichloridite, 115.4 ml of piperylene (cis- and trans-), and 4.5 g of copper stearate was left in a tightly closed vessel for 20 days. Two layers were formed: a thick yellow oil, and a mobile light-yellow liquid. The oil was separated from the mobile liquid and treated in the usual way, except that the solvent was benzene instead of chloroform. Benzene was distilled off, and the residue was vacuum-distilled. Three successive distillations gave a substance having the following constants: b.p. 162-164° (3 mm);  $n^{20}D$  1.5480;  $d^{20}_4$  1.1680; found MR 56.56 for  $C_{11}H_{13}O_2P_{-4}$ ; calculated MR 56.47. The value taken for the atomic refraction of phosphorus was 4.79 in accordance with [5-7]. Found: P 14.97, 14.77%.  $C_{11}H_{13}O_2P$ . Calculated: P 14.88%. The yield of product was 9 g (9.2% on the phenyl phosphorodichloridite that reacted). The product was 2-methyl-1-phenoxy-3-phospholene 1-oxide. The liquid part that had been separated from the oil was vacuum-distilled and gave three fractions: Fraction I, b.p. 100-103° (14 mm);  $n^{20}D$  1.5600; this was unchanged phenyl phosphorodichloridite. Fraction II, b.p. 172-174° (14 mm);  $n^{20}D$  1.5753; this was diphenyl phosphorochloridite. Fraction III, b.p. 181-182° (2-2.5 mm),  $n^{20}D$  1.5900;  $d^{20}_{4}$  1.1860; found MR 88.16 for P(OC<sub>6</sub>H<sub>5</sub>)<sub>3</sub>F<sub>9</sub>; calculated MR 87.59. Found: P 9.64, 9.97%, P(OC<sub>6</sub>H<sub>5</sub>)<sub>3</sub>. Calculated: P 10.00%. The yield of triphenyl phosphite was 50 g (25% on the phenyl phosphorodichloridite that reacted).

On hydrolysis of Fraction III with hydrochloric acid at 180-200° we obtained 83.6% of phenol, calculated on the triphenyl phosphate.

Reaction of Phenyl Phosphorodichloridite with Isoprene. A mixture of 116 g of phenyl phosphorodichloridite. 40 g of isoprene, and 0.8 g of copper stearate was shaken at room temperature for 9 days. The whole mixture was converted into a thick yellow oil, which was given the usual treatment with water and alkali and extraction with chloroform. After removal of chloroform and several vacuum distillations, we isolated a product of b.p. 156-157° (2-2.5 mm). This immediately solidified; m.p. 56-58°. Found: P 15.22, 14.85%.  $C_{11}H_{13}O_2P$ . Calculated: P 14.88%. The yield of product was 80 g (51,15% on the phenyl phosphorodichloridite). The product was 3-methyl-1-phenoxy-3-phospholene 1-oxide.

Reaction of Phenyl Phosphorodichloridite with 2,3-Dimethyl-1,3-butadiene. A mixture of 130 g of phenyl phosphorodichloridite, 61 g of 2,3-dimethyl-1,3-butadiene, and 1 g of copper stearate was left in a tightly closed bottle at room temperature. After 6 days the whole liquid had been converted into a thick resinous mass, which was treated in the usual way. After three distillations from a Favorskii flask, we collected a fraction having: b.p. 146 to 147° (2.5 mm);  $n^{20}D 1.5480$ ,  $d^{20}_{4}$  1.1512; mol. wt. (cryoscopically) in benzene 220.9; calculated mol. wt. 222; found MR 61.24 for  $C_{12}H_{15}O_2P_{4}$ ; calculated MR 61.074; atomic refraction of phosphorus 4.77. Found: C 64.81, 65.03; H 6.92, 6.95; P 14.03, 13.85%  $C_{12}H_{15}O_2P$ . Calculated: C 64.85; H 6.8; P 13.94% The yield of product was 85 g (46% on the phenyl phosphorodichloridite). This was 2,3-dimethyl-1-phenoxy-3-phospholene 1-oxide.

Reaction of Phenyl Phosphorodibromidite with Butadiene. A mixture of 66.5 g of phenyl phosphorodibromidite [8], 30 ml of butadiene, and 0.5 g of copper stearate was left in a closed bottle at room temperature. In the course of 5 days the reaction was fully completed. The whole contents were converted into a dark-colored solid mass, which was treated as in previous experiments. We obtained a product of b.p. 163-164° (3 mm) and m.p. 60-62°. The yield of the product was 29 g (70.7% of the phenyl phosphorodibromidite).

<u>Reaction of Ethyl Phosphorodichloridite with 2,3-Dimethyl-1,3-butadiene.</u> A mixture of 168 g of ethyl phosphorodichloridiate [11], 77 g of 2,3-dimethyl-1,3-butadiene, and 1.2 g of copper stearate was introduced into a thick-walled bottle and left in the light at room temperature. The mixture acquired a dark yellow color, but did not thicken. After 40 days the liquid was vacuum-distilled. Fraction I came over at 19-20° (12 mm);  $n^{20}D$  1.4612; yield 55 g (31.5%). This was unchanged ethyl phosphorodichloridite. Fraction II came over at 121-122° (8 mm); a white crystalline mass, m.p. 78-82°. Found: Cl 21.41, 21.34; P 18.90%, C<sub>6</sub>H<sub>10</sub>OPCl. Calculated: Cl 21.58; P18.84%. The yield of the product was 86 g (55.2% on the ethyl phosphorodichloridite that reacted). This product was 1chloro-3,4-dimethyl-3-phospholene 1-oxide.

<u>Hydrolysis of 1-Chloro-3,4-dimethyl-3-phospholene 1-Oxide.</u> 1-Chloro-3,4-dimethyl-3-phospholene 1-oxide (20 g) was carefully treated with water. There was a rise in temperature, and hydrogen chloride was liberated. On cooling we isolated a slightly yellowish crystalline product; it was recrystallized from water in presence of activated charcoal. We obtained well-formed large crystals that refracted light strongly; m.p. 121-123°; yield 12 g (67%). Found: C 49.50, 49.39; H 7.38, 7.54; P 21.20, 21.68%. C<sub>6</sub>H<sub>11</sub>O<sub>2</sub>P. Calculated: C 49.31; H 7.53; P 21.23%. The crystals consisted of 1-hydroxy-3,4-dimethyl-3-phospholene 1-oxide.

### SUMMARY

1. Diene hydrocarbons undergo diene condensation with aryl and alkyl phosphorodihalidites with formation of five-membered heterocyclic compounds (phospholenes).

2. Aryl phosphorodibromidites react considerably more rapidly with dienes than the corresponding phosphorodichloridites.

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