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Reaction of Secondary Phosphine Chalcogenides with Diallylamine

S. I. Verkhoturova, T. I. Kazantseva, S. N. Arbuzova, A. I. Albanov, N. K. Gusarova, and B. A. Trofimov

A.E. Favorskii Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia e-mail: boris_trofimov@irioch.irk.ru

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Abstract—Diphenyl- or bis(2-phenylethyl)phosphine sulfides and -phosphine selenides react with diallylamine under radical initiation (UV or AIBN) to afford the corresponding diadducts and tetrahydropyrrolylmethyl phosphine chalcogenides. The yield and the ratio of the products depend on the structure of the starting secondary phosphine chalcogenides.

Keywords: secondary phosphine chalcogenides, diallylamine, radical initiation, cyclization

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Addition of secondary phosphines and phosphine chalcogenides to polyalkenes is a convenient atomeconomic approach to the synthesis of polyphosphines and polyphosphine chalcogenides including their functional derivatives. Reactions of this type have been successfully realized under radical conditions by the example of divinyl [1], diallyl [2, 3], trivinyl [4], tetravinyl [5] ethers, divinyl chalcogenides [6–9], as well as tris(4-vinylbenzyl)phosphine oxide [10]. As a result, promising polydentate ligands for metal complexes, including metallacrown ethers [1, 11–13] have been synthesized.

At the same time, there are no data in the literature on the reaction of diallylamine with secondary phosphine chalcogenides, although substituted diallylamines react with diphenylphosphine oxide (AIBNinitiation) [2] and diphenylphosphine sulfide (microwave activation) [3] to give cyclic adducts, phosphoruscontaining pyrrolidines (Scheme 1).





In the present work we have studied for the first time the radical addition of secondary phosphine sulfides and phosphine selenides to diallylamine in order to develop a general convenient atom-economic method of the synthesis of functional tertiary phosphine chalcogenides containing an amino group.

Diphenylphosphine sulfide (I) turned out to react with diallylamine (in the ratio 2 : 1) in benzene or dioxane at UV irradiation (200W Hg arc Lamp) or in the presence of AIBN (5 wt %, 60–65°C) in 2–2.5 h to give diadduct II as the main product (the isolated yield 52%). For the equimolar ratio of the reagents, compound II is also the main product (Scheme 2).





 $R = Ph (A^1), Ph (CH_2)_2 (A^2).$

Besides, under these conditions, the formation of a small amount of side cyclic product, (4-methyltetrahydro-1*H*-pyrrol-3-yl)methyl(diphenyl)phosphine sulfide (III) was detected by 1 H, 31 P NMR and chromatomass spectrometry:



At the same time, a similar heterocycle, (4methyltetrahydro-1H-pyrrol-3-yl)methyl(diphenethyl)phosphine sulfide (**IV**), is the major product of the

radical addition (UV or AIBN-initiation, $60-65^{\circ}$ C) of bis(2-phenylethyl)phosphine sulfide (V) to diallylamine in benzene or dioxane. Under optimal conditions (UV irradiation, dioxane, 7.5 h, the ratio of the reagents 1 : 1) compound (IV) was isolated in preparative yield of 45% (Scheme 3).

The difference in chemoselectivity of the studied reaction of diallylamine phosphorylation with phosphine sulfides **I**, **V** and the formation of tertiary phosphine sulfides **II–IV** can be rationalized as follows (Scheme 4). The P-centered radical generated from secondary phosphine sulfides adds to the double bond of the diallylamine molecule to form the Ccentered radical (**A**), which can further react in two ways. In the case of diphenylphosphine sulfide **I**



radical (A^1) readily abstracts hydrogen atom from the second molecule of phosphine sulfide to give a new P-centered radical. The latter is added to the double bond of the monoadduct to give diadduct II. As distinct of that, radical (A^2) does not react with bis(2-phenylethyl)-phosphine sulfide (V) (where the P–H bond is, apparently, more strong as compared to that in the molecule of diphenylphosphine sulfide), but rather is added intramolecularly to the second double bond to form the cyclic product IV.

The reaction of secondary phosphine selenides with diallylamine under the conditions of radical initiation proceeds in a less selective manner. Thus, bis(2-phe-nylethyl)phosphine selenide reacts with diallylamine in the presence of AIBN (60–65°C, benzene) or under UV irradiation to form a mixture of organophosphorous compounds among which the main one (~60% according to the ³¹P NMR spectra) is (4-methyl-tetrahydro-1*H*-pyrrol-3-yl)methyl(diphenethyl)phosphine selenide (**VI**) (Scheme 5). Under optimal conditions (UV irradiation, dioxane, 4 h, the ratio of the reagents 1 : 1) phosphine selenide **VI** was isolated in preparative yield of 32%.

At the same time, the main products of the reaction of diphenylphosphine selenide with diallylamine under UV irradiation are diphenylphosphine **VII** and diallylammonium diphenylselenophosphinate **VIII**, whereas the expected acyclic and cyclic adducts are formed in small amounts. We failed to isolate compounds **VII**, **VIII** from the complex reaction mixture, but they were identified by ³¹P NMR spectroscopy comparing with authentic samples. To this end, diselenophosphinate **VIII** was specially first synthesized by us from diphenylphosphine selenide, diallylamine, and elemental selenium by the known reaction [14] (Scheme 6).

The formation of compounds VII, VIII can be represented by Scheme 7 including the stage of reversible disproportionation of the secondary phosphine selenide to the corresponding secondary phosphine VII and diselenophosphinic acid IX. The latter reacts with diallylamine to give diselenophosphinate VIII thus shifting the equilibrium to the right.

A similar reaction of secondary phosphine selenides with amines at room temperature without initiator was described earlier [15].





In the case of chemical initiation (AIBN, 60–65°C, dioxane), diphenylphosphine selenide reacts with diallylamine to afford, along with compounds **VII**, **VIII**, also the amide of N,N-diallyl(diphenyl)seleno-phosphinic acid (**X**) (Scheme 8).

It turned out that this reaction takes place in the absence of initiator (60–65°C, dioxane). Under these conditions, amide **X** was isolated with the yield of 24%. The latter is formed, apparently, due to thermal decomposition of diselenophosphinate **VIII** according to Scheme 9 as suggested earlier [16].

Note that secondary phosphine oxides practically do not react with diallylamine under the developed conditions (UV initiation or in the presence of AIBN). This fact is in contradiction with the aforementioned literature data [2], but it is in accordance with the commonly accepted notions of a relatively low reactivity of secondary phosphine oxides in the reactions of radical addition to the double bond [3, 17–19].

It is important that the starting bis(2-phenylethyl)phosphine sulfide and selenide are now available and can be readily prepared from elemental phosphorus, styrene, and elemental chalcogenides [20, 21].

Therefore, new fundamental data on the reactions of secondary phosphine chalcogenides with diallylamine were obtained. The course of the reaction depends both on the reaction conditions and on the structure of organic radical and the nature of the chalcogene in the molecule of the starting phosphine chalcogenide. Diorganylphosphine sulfides and bis(2phenylethyl)phosphine selenide react with diallylamine under the conditions of radical initiation (AIBN, UV irradiation) via the diaddition route and/or monocycloaddition to afford the corresponding diadducts and tetrahydropyrrolylmethylphosphine chalcogenides. An original reaction of diphenylphosphine selenide

> Scheme 9. VIII $\xrightarrow{60-65^{\circ}C} X$

with diallylamine was found leading to the mixture of organophosphorous compounds, the main products being diphenylphosphine, diallylammonium diphenyl-diselenophosphinate, and amide of *N*,*N*-diallyl(diphenyl)seleno-phosphinic acid. Secondary phosphine oxides practically do not react with diallylamine under the studied conditions.

EXPERIMENTAL

¹H, ¹³C, ³¹P, and ⁷⁷Se spectra were registered on a Bruker DPX 400 spectrometer (400.13, 100.62, 161.98, and 76.31 MHz, respectively) in CDCl₃, internal reference HMDS (¹H, ¹³C), Me₂Se (⁷⁷Se), external reference 85% H₃PO₄ (³¹P). The signals in the ¹H, ¹³C NMR spectra were assigned using 2D homoand heteronuclear NMR techniques: COSY, HSQC. Mass spectra of electron impact (70 eV) were obtained on a GCMS-QP5050A SHIMADZU instrument (quadruple mass analyzer, the range of detected masses 34–450 Da, capillary column, SPB–5 phase).

N,*N*-Bis[3-(diphenylphosphorothioyl)propyl] amine (II). The solution of diphenylphosphine sulfide I (0.093 g, 0.43 mmol) and diallylamine (0.021 g, 0.021 g)0.215 mmol) in 1.2 mL of benzene was placed in a quartz ampule flushed with argon and irradiated with UV light for 2.5 h. Benzene was removed under a reduced pressure, the residue was dried in a vacuum. The ³¹P NMR spectrum of the obtained oily product contains signals at 47.3, 42.6, 41.51 ppm belonging to *N*.*N*-bis[3-(diphenylphosphorothioyl)propyl]amine **II**, cis- and trans-isomers of (4-methyltetrahydro-1Hpyrrol-3-yl)methyl(diphenyl)phosphine sulfide (III), and non-identified signals at 57.3 and 60.7 ppm (the ratio of signals 25:8:2:1:1, respectively). The ¹H NMR spectrum, along with the signals characteristic of diadduct (II), shows the signals belonging to the cisand *trans*-isomers of adduct III: 0.96 d (Me, ${}^{3}J_{HH}$ 7.0 Hz), 0.99 d (Me, ${}^{3}J_{\text{HH}}$ 6.7 Hz) in the ratio 4 : 1, respectively. In the mass spectrum two peaks of molecular ions with m/z 315 are present. The obtained oily product was dissolved in dioxane (2 mL) and passed through Al₂O₃ layer (6 mm) (eluent dioxane). Dioxane was removed under a reduced pressure, the residue was dried in a vacuum. Yield 0.06 g (52%), oily light-yellow compound. ¹H NMR spectrum, $\delta_{\rm H}$, ppm: 1.73 m (4H, CH₂C<u>H</u>₂CH₂), 2.49 m (4H, CH₂P), 2.59 t (4H, CH₂N, ³J_{HH} 6.7 Hz), 7.42 m (12H, *m*-Ph, *p*-Ph), 7.81 m (8H, *o*-Ph, ³J_{HP} 13.0 Hz). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 22.25 (CH₂CH₂CH₂), 29.99 d (CH₂P, ¹J_{CP} 57.1 Hz), 49.21 d (CH₂N, ³J_{CP} 16.8 Hz), 128.43 d (C^m, ³J_{CP} 12.8 Hz), 130.85 d (C^o, ²J_{CP} 10.3 Hz), 131.25 (C^p), 132.53 d (C^{ipso}, ¹J_{CP} 80.2 Hz). ³¹P NMR spectrum, $\delta_{\rm P}$, ppm: 43.7. Found, %: s 67.38; H 6.40; N 2.47; P 11.28; S 11.77. C₃₀H₃₃NP₂S₂. Calculated, %: C 67.52; H 6.23; N 2.62; P 11.61; S 12.02.

(4-Methyltetrahydro-1H-pyrrol-3-yl)methyl(diphenethyl)phosphine sulfide (IV). The solution of bis-(2-phenylethyl)phosphine sulfide (V) (0.114 g, 0.42 mmol) and diallylamine (0.041 g, 0.42 mmol) in 1 mL of dioxane was placed in a quartz ampule, flushed with argon and irradiated with UV light for 7.5 h. Dioxane was removed under a reduced pressure, the residue was dissolved in benzene (2 mL) and passed through Al₂O₃ layer (5 mm) (eluent benzene, then chloroform). From the chloroform solution the solvent was removed under a reduced pressure, the residue was dried in a vacuum. Yield 0.07 g (45%) (cis- and trans-isomers in the ratio of 5 : 1), oily lightyellow compound. ¹H NMR spectrum, $\delta_{\rm H}$, ppm (*cis*isomer): 0.80 d (3H, Me, ${}^{3}J_{HH}$ 7.1 Hz), 1.65 d.d.d, 1.82 d.d.d (2H, PC<u>H</u>₂Pyr, ${}^{2}J_{HH'}$ 15.0 Hz, ${}^{2}J_{HP} = {}^{3}J_{HH} =$ 10.0 Hz, ${}^{3}J_{H'H}$ 4.0 Hz, ${}^{2}J_{H'P}$ 12.3 Hz), 2.04 m (4H, PCH₂), 2.21 m (1H, CH⁴), 2.41 m (1H, CH³), 2.59 d.d, 3.11 d.d (2H, CH_2^5 , ${}^2J_{HH}$ 11.0 Hz, ${}^3J_{H5H4}$ 4.4 Hz, ${}^3J_{H5'H4'}$ 6.6 Hz), 2.71 d.d, 3.27 d.d (2H, CH_2^2 , ${}^2J_{HH'}$ 10.7 Hz, ³*J*_H2_H3 7.2 Hz, ³*J*_H2'_H3 8.8 Hz), 2.83 m (4H, CH₂Ph), 4.01 br.s (1H, NH), 7.10 m, 7.19 m (10H, Ph). ¹H NMR spectrum, $\delta_{\rm H}$, ppm (*trans*-isomer): 0.94 d (3H, Me, ${}^{3}J_{HH}$ 6.5 Hz), 1.65–1.82 m (1H, CH⁴), the rest of signals of the trans-isomer are masked by the signals of the major *cis*-isomer. ¹³C NMR spectrum, δ , ppm (cis-isomer): 14.11 (Me), 28.62 (PhCH₂), 32.74 d (P<u>C</u>H₂Pyr, ¹*J*_{CP} 48.4 Hz), 33.39 d, 33.67 d (PhCH₂<u>C</u>H₂, ${}^{1}J_{CP}$ 48.0 and 48.3 Hz respectively), 36.46 d (C³, ${}^{2}J_{CP}$ 10.3 Hz), 37.13 d (C⁴, ${}^{3}J_{CP}$ 3.2 Hz), 50.77 d (C², ${}^{3}J_{CP}$ 4.4 Hz), 52.83 (C⁵), 126.58 (C^{*p*}), 128.21, 128.25, 128.28, 128.71 (C^o , C^m), 140.41 d, 140.43 d (C^{ipso} , ${}^{3}J_{CP}$ 13.3 and 13.5 Hz respectively). ¹³C NMR spectrum, δ , ppm (*trans*-isomer): 16.46 (Me), 28.54 d (PhCH₂, ${}^{2}J_{CP}$ 2.7 Hz), 33.35 d (P<u>C</u>H₂Pyr, ¹J_{CP} 48.1 Hz), 33.70 d, 34.24 d (PhCH₂CH₂, ${}^{1}J_{CP}$ 49.7 and 47.7 Hz respectively), 41.43 d (C³, ${}^{2}J_{CP}$ 12.5 Hz), 41.63 d (C⁴, ${}^{3}J_{CP}$

3.7 Hz), 49.85 d (C^2 , ${}^{3}J_{CP}$ 15.6 Hz), 52.70 (C^5), 126.51 (C^p), 128.24, 128.34, 128.37, 128.67 (C^o , C^m), 140.56 d, 142.16 d (C^{ipso} , ${}^{3}J_{CP}$ 13.9 and 16.9 Hz respectively). ³¹P NMR spectrum, δ_P , ppm: 49.4 (*cis*-isomer), 48.3 (*trans*-isomer). Found, %: C 70.88; H 7.98; N 3.59; P 8.12; S 8.45. C₂₂H₃₀NPS. Calculated, %: s 71.12; H 8.14; N 3.77; P 8.34; S 8.63.

(4-Methyltetrahydro-1H-pyrrol-3-yl)methyl(diphenethyl)phosphine selenide (VI). The solution of bis(2-phenylethyl)phosphine selenide (V) (0.049 g)0.15 mmol) and diallylamine (0.014 g, 0.15 mmol) in 1 mL of dioxane was placed in a quartz ampule, flushed with argon and irradiated with UV light for 4 h. Dioxane was removed under reduced pressure, the residue was dissolved in chloroform (2 mL) and passed through Al₂O₃ layer (5 mm) (eluent chloroform, 20 mL). Chloroform was removed under a reduced pressure, the residue was washed with diethyl ether $(3 \times 2 \text{ mL})$, dried in a vacuum. Yield 0.02 g (32%, purity 95 %), (cis- and trans-isomers in the ratio of 4 : 1, oily light-yellow compound. ¹H NMR spectrum, $\delta_{\rm H}$, ppm (*cis*-isomer): 0.94 d (3H, Me, {}^{3}J_{\rm HH} 7.1 Hz), 1.93 m (2H, PCH₂Pyr), 2.24 m (4H, PCH₂), 2.44 m $(1H, CH^4)$, 2.32 m, 2.96 m (2H, $CH_2^5)$, 2.51 m, 3.04 m (2H, CH₂²), 2.59 m, 3.04 m (1H, CH₂³), 2.96 m (4H, CH₂Ph), 7.20 m, 7.25 m (10H, Ph). ¹H NMR spectrum, δ, ppm (*trans*-isomer): 1.02 d (3H, Me, ${}^{3}J_{HH}$ 6.6 Hz), the rest of signals of the trans-isomer are masked by the signals of the major *cis*-isomer. ¹³C NMR spectrum, δ, ppm (*cis*-isomer): 13.41 (Me), 29.39 d (Ph<u>C</u>H₂, ${}^{2}J_{CP}$ 3.0 Hz), 33.21 d, 33.28 d (PhCH₂CH₂, ¹J_{CP} 41.8 and 41.4 Hz respectively), 35.92 d ($C^{3, 2}J_{CP}$ 9.9 Hz), 37.06 d (C⁴, ${}^{3}J_{CP}$ 2.2 Hz), 40.28 d (P<u>C</u>H₂Pyr, ${}^{1}J_{CP}$ 57.8 Hz), 48.26 d (C², ${}^{3}J_{CP}$ 4.3 Hz), 50.37 (C⁵), 126.74 (C^{*p*}), 128.30, 128.40, 128.50, 128.81 (C^o, C^m), 141.57 d, 141.74 d (C^{*ipso*}, ${}^{3}J_{CP}$ 17.3 and 16.1 Hz respectively). ${}^{31}P$ NMR spectrum, δ_P , ppm: 37.7 (+ satellites, ${}^{1}J_{PSe}$ 706.5 Hz) (cis-isomer), 36.3 (trans-isomer). ⁷⁷Se NMR spectrum, δ_{Se} , ppm (*cis*-isomer): -75.0 d (¹J_{SeP} 706.5 Hz).

Diallylammonium diphenyldiselenophosphinate (VIII). To the solution of diphenylphosphine selenide (0.117 g, 0.44 mmol) and diallylamine (0.064 g, 0.66 mmol) in 3 mL of dioxane amorphous selenium was added (0.035 g, 0.44 mmol). The mixture was stirred at 60–65°C for 3.5 h, filtered, dioxane removed under a reduced pressure, the residue was washed with diethyl ether (3 × 2 mL), dried in a vacuum. Yield 0.13 g (67 %), powder of light pink color, mp. 89–92°C. ¹H NMR spectrum, $\delta_{\rm H}$, ppm: 3.58 d (4H, CH₂N, ³*J*_{HH} 6.9 Hz), 5.28 br.d (2H_{trans}, =CH₂, ³*J*_{HH} 17.1 Hz), 5.29 br.d (2H_{cis}, =CH₂, ³*J*_{HH} 10.2 Hz), 5.81 d.d.t (2H, =CH,

1747

³*J*_{HH} 10.2 Hz, ³*J*_{HH} 17.1 Hz, ³*J*_{HH} 6.9 Hz), 7.23 m (6H, *m*-Ph, *p*-Ph), 8.10 m (4H, *o*-Ph). ¹³C NMR spectrum, $\delta_{\rm C}$, ppm: 48.25 (CH₂N), 123.95 (=CH₂), 128.29 d (C^m, ³*J*_{CP} 12.1 Hz), 129.03 (=CH), 130.49 d (C^{*p*}, ⁴*J*_{CP} 1.7 Hz), 131.88 d (C^o, ²*J*_{CP} 12.1 Hz), 140.76 d (C^{*ipso*}, ¹*J*_{CP} 78.0 Hz). ³¹P NMR spectrum, $\delta_{\rm P}$, ppm: 22.7 (+ satellites, ¹*J*_{PSe} 586 Hz). Found, %: s 48.72; H 4.98; N 3.06; P 6.76; Se 35.52. C₁₈H₂₂NPSe₂. Calculated, %: C 48.99; H 5.03; N 3.17; P 7.02; Se 35.79.

Amide of N,N-diallyl(diphenyl)selenophosphinic acid (X). The solution of diphenylphosphine selenide (0.093 g, 0.35 mmol) and diallylamine (0.034 g, 0.034 g)0.35 mmol) in 2 mL of dioxane was flushed with argon and stirred at 60-65°C for 3 h. The ³¹P NMR spectrum of the reaction mixture contains the signals at -40.0 $({}^{1}J_{PH} 210 \text{ Hz})$, 22.4 (satellites, ${}^{1}J_{PSe} 586 \text{ Hz}$) and 69.4 ppm (satellites: ${}^{1}J_{PSe}$ 754.8 Hz) from diphenylphosphine VII, diselenophosphinate VIII, and amide X, and a non-identified signal at 53.0 ppm (the ratio of signals 1 : 2 : 2 : 1 respectively). Dioxane was removed under a reduced pressure, the residue was dissolved in benzene, the solution was passed through Al₂O₃ layer (1 cm) (eluent benzene). Benzene was removed under a reduced pressure, the residue was dried in a vacuum. Yield 0.03 g (24%), light-yellow waxy product. ¹H NMR spectrum, $\delta_{\rm H}$, ppm: 3.55 d.d (4H, CH₂N, ${}^{3}J_{\rm HH}$ 6.5 Hz, ${}^{3}J_{\rm HP}$ 11.7 Hz), 5.06 d.d.t (2H_{trans}, =CH₂, ${}^{3}J_{\rm HH}$ 17.0 Hz, ${}^{2}J_{\text{HH}}$ 1.3 Hz, ${}^{4}J_{\text{HH}}$ 1.2 Hz), 5.12 br.d (2H_{cis}, =CH₂, ${}^{3}J_{\text{HH}}$ = 10.2 Hz), 5.83 d.d.t (2H, =CH, ${}^{3}J_{\text{HH}}$ 17.0 Hz, ${}^{3}J_{\text{HH}}$ 10.2 Hz, ³J_{HH} 6.5 Hz), 7.43 m (6H, *m*-Ph, *p*-Ph), 8.00 m (4H, *o*-Ph). ¹³C NMR spectrum, δ_C , ppm: 49.74 (CH₂N), 118.58 (=CH₂), 128.41 d (C^m, ³J_{CP} 13.4 Hz), 131.82 d $(=CH, {}^{3}J_{CP} 2.6 Hz), 132.52 d (C^{o}, {}^{2}J_{CP} 11.2 Hz),$ 132.86 d (C^{ipso} , ${}^{1}J_{CP}$ 114.7 Hz), 133.97 d (C^{p} , ${}^{4}J_{CP}$ 6.0 Hz). ³¹P NMR spectrum, δ_P , ppm: 69.3 (+ satellites, $^{1}J_{\text{PSe}}$ 754.8 Hz). ⁷⁷Se NMR spectrum, δ_{Se} , ppm: -267.5 d (¹J_{SeP} 754.8 Hz). Found, %: s 59.86; H 5.42; N 3.67; P 8.43; Se 21.78. C₁₈H₂₀NPSe. Calculated, %: C 60.00; H 5.60; N 3.89; P 8.60; Se 21.92.

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RUSSIAN JOURNAL OF GENERAL CHEMISTRY Vol. 84 No. 9 2014